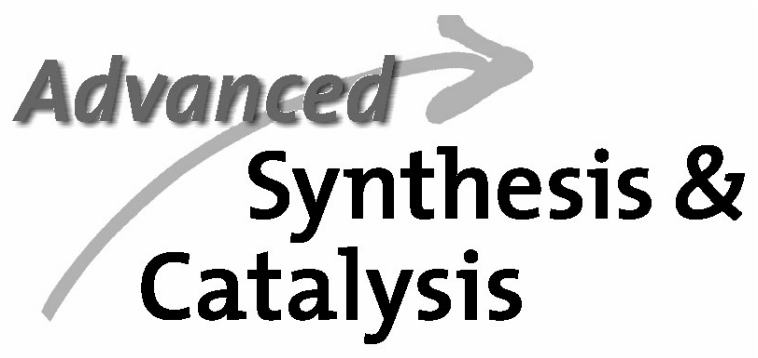

Supporting Information

Efficient and General One-Pot Synthesis of Diaryliodonium Triflates: Optimization, Scope and Limitations

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SUPPORTING INFORMATION

1 Optimization studies

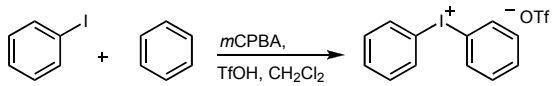
1.1 The use of mCPBA and TfOH to synthesize 3a.

Stoichiometric amounts of all components resulted in a sluggish reaction (Table S1, entry 1). No iodobenzene could be recovered, which is indicative of a working oxidation process but inefficient EAS. In order to increase the reaction rate of the EAS step, 2 equiv TfOH was employed, which indeed resulted in a much faster formation of **3a** (entries 2, 3). An increase of *m*CPBA to 2 equivalents instead resulted in decreased yield (entry 4), which could be due to over-oxidation to iodine(V). The amount of benzene (**2a**) also had an impact on the reaction outcome, as depicted in entries 5, 6. Increasing the temperature considerably reduced the reaction time, delivering **3a** in good yields after 3 h at 40 °C (entry 7) or 10 min at 80 °C (entry 8). Pleasingly, the use of 3 equiv. of TfOH delivered **3a** in 89% yield (entry 9).

A subsequent temperature study with the optimized conditions revealed that the reaction is much faster than we had anticipated (Table S2). Thus, the reaction was complete within 10 min at temperatures down to -50 ºC, and 0 ºC or rt was deemed to be the most convenient temperature for further reactions. Control experiments with 2 equiv. TfOH showed that the reactivity is considerably decreased with less acid (Table S1, entries 10, 11).

Changing the solvent to Et_2O, $CHCl_3$ or CH_3CN resulted in decreased yields (entries 12-14), whereas changes in concentration were less important (entries 15-17). We subsequently investigated whether TfOH was needed in the oxidation or only mediated the EAS step. Thus, iodobenzene, *m*CPBA and benzene were reacted in the absence of TfOH, which indeed gave a slow oxidation but no salt formation (entry 18). Purification by flash chromatography in CH_2Cl_2/MeOH instead of precipitation gave **3a** in slightly reduced yield (entry 19).

Table S1: Optimization of reaction conditions for synthesis of salt **3a**.[a]



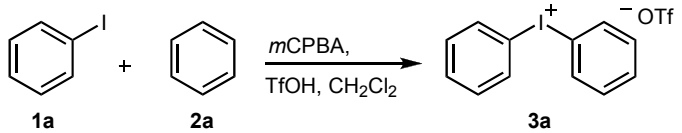
1a 2a 3a

Entry	2a (equiv.)	mCPBA (equiv.)	TfOH (equiv.)	T (ºC)	Time	Yield (%)[b]
1	1.1	1.1	1.1	rt	18 h	5
2	1.1	1.1	1.5	rt	2.5 h	61
3	1.1	1.1	2	rt	18 h	68
4	1.1	2	2	rt	18 h	60
5	2	1.1	2	rt	18 h	75
6	5	1.1	2	rt	21 h	82
7	1.1	1.1	2	40	3 h	83
8	1.1	1.1	2	80	10 min	73
9	1.1	1.1	3	80	10 min	89
10	1.1	1.1	2	rt	10 min	58
11	1.1	1.1	2	0	10 min	56
12[c]	1.1	1.1	3	0	10 min	5
13[d]	1.1	1.1	3	0	10 min	66
14[e]	1.1	1.1	3	0	10 min	-[i]
15[f]	1.1	1.1	3	0	10 min	84
16[g]	1.1	1.1	3	0	10 min	91
17[h]	1.1	1.1	3	0	10 min	52
18	1.1	1.1	0	0	10 min	-[j]
19	1.1	1.1	3	0	10 min	79[k]

[a] Reaction conditions: **1a** (1.0 equiv., 0.23 mmol), **2a** and mCPBA were dissolved in CH_2Cl_2 (1 mL), TfOH was added dropwise at 0 ºC (or tabulated temp. if lower) and the reaction was stirred at the indicated temperature and

time. [b] Isolated yield. [c] In Et_2O. [d] In $CHCl_3$. [e] In CH_3CN. [f] In CH_2Cl_2 (2 mL). [g] In CH_2Cl_2 (0.5 mL). [h] No solvent was used. [i] No precipitation. [j] To see if the oxidation took place, see text. [k] Isolated by flash chromatography.

Table S2: Temperature influence on yield of **3a** with optimized stoichiometry.[a]



1a **2a** **3a**

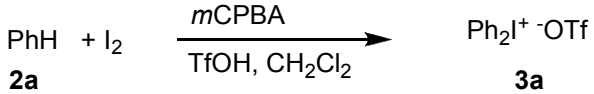
Entry	T (°C)	Yield (%)[b]
1	-78	38
2	-50	87
3	-25	87
4	0	91
5	rt	92
6	40	91
7	50	90
8	60	90
9	70	90
10	80	89
11	90	89

[a] Reaction conditions: *m*CPBA (65% active oxidant, 66 mg, 0.25 mmol) and iodobenzene (**1a**, 0.23 mmol) were dissolved in CH_2Cl_2 (1 mL) in a sealed tube. Benzene (**2a**, 0.25 mmol) was added and the solution was cooled to 0 °C for reactions run at/over 0 °C; in the others at the tabulated temperature, followed by dropwise addition of TfOH (62 μL, 0.69 mmol). The reaction was stirred at the indicated temperature for 10 min. [b] Isolated yield.

1.2 Synthesis of diaryliodonium salts directly from arenes and iodine.

Direct application of the previously optimized reaction conditions but with 3 equiv. mCPBA[1] indeed resulted in formation of salt **3a** (Table S3, entry 1). The use of 4 equivalents of *m*CPBA improved the conversion, and resulted in a clean formation of salt **3a** in 60 % yield (entry 6). The yield could be improved by prolonged reaction time or using an excess of benzene, as depicted in entries 7, 10. When the temperature was increased to 80 °C, a reaction time of 10 min was sufficient to give **3a** in 93% yield (entry 13). The use of substoichiometric amount of mCPBA, with the aim of using only one iodine atom in the reaction, proved inferior and delivered **3a** in only 33% yield (entry 21).[2]

Table S3. Synthesis of diaryliodonium salts directly from arenes.[a]



2a **3a**

Entry	2a (equiv.)	*m*CPBA (equiv)	TfOH (equiv.)	T (°C)	Time	Yield (%)[b]
1	4.1	3	4	rt	10 min	45
2	4.1	3	4	rt	12 h	58
3	4.1	3	4	rt	20 h	61
4	4.1	4	3	rt	16 h	13
5	4.1	3	6	rt	10 min	92
6	4.1	4	4	rt	2 h	60
7	4.1	4	4	rt	21 h	72
8	4.1	4	4	rt	10 min	41
9	10	4	4	rt	10 min	64
10	10	4	4	rt	22 h	81
11	10	4	4	40	10 min	75
12	4.1	3	4	60	10 min	92 (87)

13	4.1	3	4	80	10 min	93 (80)
14	4.1	3	3	80	10 min	46
15	10	3	4	80	10 min	85 (78)
16	4.1	4	4	80	10 min	78
17	10	4	4	80	10 min	76
18[c]	10	3	3	80	10 min	(51)
19[c]	10	4	4	80	10 min	(66)
20[c]	10	6	6	80	10 min	(72)
21	4.1	2.1	3	rt	22 h	33

[a] Reaction conditions: *m*CPBA (65% active oxidant) and iodine were dissolved in CH_2Cl_2 in a sealed tube. Benzene (**2a**) was added and the solution was cooled to 0 °C for reactions run at/over 0 °C; in the others at the tabulated temperature, followed by dropwise addition of TfOH. [b] Isolated yield. ( ) are isolated by flash chromatography.

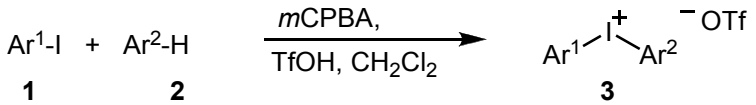
2 Reaction conditions for the synthesis of salts 3

General experimental conditions

NMR spectra were recorded using DMSO-d_6, $CDCl_3$ or MeOD-d_4 as solvent. DMSO-d_6 was used in most cases, as all iodonium salts were soluble in this solvent. Chemical shifts are given in ppm relative to TMS for $CDCl_3$, the residual peak of DMSO (^{1}H NMR δ 2.50, ^{13}C NMR 39.52) or MeOD (^{1}H NMR δ 3.31, ^{13}C NMR 49.00) as internal standard, with multiplicity (s=singlet, d=doublet, t=triplet, q=quartet, m=multiplet, br=broad, app=apparent), integration and coupling constants (Hz). In the IR spectra, only the strongest/structurally most important peaks are listed. The reactions were carried out in sealed tubes to allow for reaction temperatures above the boiling point of CH_2Cl_2. The reactions were run without precaution to avoid moisture or air, *i.e.* without protective gas or dried solvent. TfOH (≥99%) was stored under argon atmosphere. The percentage of active oxidizing agent in *m*CPBA was determined by iodometric titration.[3] All other chemicals were used as received without further purification. Analytical details of all novel salts are given in the supporting information.

General synthetic procedures are given in the Experimental part. Tables S4 and S5 give details on reaction times and temperatures, which have not been optimized for each product but roughly anticipated from the electronic properties of the substrates. The reactions were generally performed with 2 equiv. triflic acid, unless this resulted in byproduct formation. Flash chromatography was performed with a CH_2Cl_2/MeOH gradient from 50:1 to 10:1.

Table S4: Reaction conditions for the synthesis of salts **3** from aryl iodides and arenes.

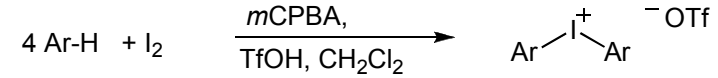


Entry	Ar^1I	Ar^2H	TfOH (equiv)	T (°C)	Time	Product	Yield (%)[a]
1	**1a**	**2a**	3	rt	10 min	**3a**	92
2	**1a**	**2b**	2	80	2 h	**3b**	85
3	**1a**	**2c**	3	rt	22 h	**3c**	71[b]
4	**1a**	**2d**	3	rt	17 h	**3d**	57[b]
5	**1a**	**2e**	3	rt	1 h	**3e**	92[b]
6	**1a**	**2f**	2	0	10 min	**3f**	85
7	**1a**	**2g**	2	0→rt	1 h	**3g**	86
8	**1a**	**2h**	2	rt	3 h	**3h**	66

9	**1a**	**2i**	3	0	1 h	**3i**	80
10	**1a**	**2j**	2	rt	1 h	**3j**	78
11	**1a**	**2k**	2	0→rt	1 h	**3k':3k''** 1.2:1	94
12	**1a**	**2l**	2	rt	1 h	**3l**	83
13	**1a**	**2m**	2	rt / -78	10 + 10 min	**3m**	87
14	**1a**	**2n**	2	rt / 0	10 + 15 min	**3n**	82
15	**1b**	**2a**	3	0	1 h	**3c**	78
16	**1b**	**2c**	3	0	1 h	**3o**	91
17	**1b**	**2m**	2	0 / -20	40 + 10 min	**3p**	58
18	**1c**	**2a**	3	rt	1 h	**3d**	65
19	**1c**	**2d**	3	rt	19 h	**3q**	83
20	**1c**	**2m**	2	rt / -20	60 + 10 min	**3r**	57
21	**1d**	**2a**	2	rt	10 min	**3s**	85
22	**1d**	**2e**	2	rt	24 h	**3t**	82
23	**1d**	**2f**	2	rt	10 min	**3u**	90
24	**1d**	**2g**	2	rt	1 h	**3v**	89
25	**1d**	**2h**	2	80	15 min	**3w**	62
26	**1d**	**2i**	2	0	1 h	**3x**	51[c]
27	**1d**	**2j**	2	rt	1 h	**3y**	84
28	**1d**	**2m**	3	-20	10 min	**3z**	56[c]
29	**1e**	**2a**	3	0	45 min	**3f**	71
30	**1e**	**2f**	2	rt	10 min	**3aa**	52
31	**1f**	**2a**	2	rt	1 h	**3g**	66[c]
32	**1g**	**2a**	2	80	15 h	**3ab**	85
33	**1h**	**2a**	2	80	15 h	**3ac**	59
34	**1i**	**2a**	2	80	15 h	**3ad**	63
35	**1j**	**2a**	2	0	1 h	**3ae**	73
36	**1k**	**2a**	3	80	2 h	**3af**	60
37	**1k**	**2m**	3	0	15 min	**3ag**	53

[a] Isolated yield. [b] About 5% *ortho*-isomer detectable by NMR. [c] Isolated by flash chromatography.

Table S5. Reaction conditions for the reaction of arenes **2** with iodine.



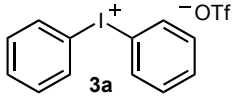
2 **3**

Entry	ArH / (equiv)	T (˚C)	Time	Product	Yield (%)[a]
1[b]	**2a** / 4.1	80	10 min	**3a**	93
2	**2a** / 10	rt	22 h	**3a**	81
3	**2c** / 10	rt	20 min	**3o**	64
4	**2d** / 10	rt	21 h	**3q**	57
5	**2e** / 10	rt	12 h	**3ah**	71
6	**2f** / 10	rt	2 h	**3u:3aa** 3:1	52
7	**2f** / 4.1	0	1 h	**3u**	31
8	**2g** / 10	rt	10 min	**3ai**	78
9	**2h** / 10	rt	12 h	**3aj**	47
10[b]	**2j** / 4.1	rt	1 h	**3ak**	52
11	**2o** / 10	rt	19 h	**3al**	24

[a] Isolated yield. [b] 3 equiv. *m*CPBA was used.

3 Analytical data of diaryliodonium salts 3

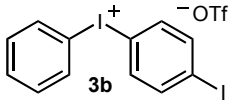
Diphenyliodonium triflate (3a):[4-6]



3a

Synthesized from PhI + PhH in 92% yield or from PhH + I_2 in 81% yield. Analytical data were in agreement with previous reports.

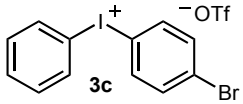
(4-Iodophenyl)(phenyl)iodonium triflate (3b):[5, 6]



3b

Synthesized from PhI + PhI in 85% yield as a white solid. Analytical data were in agreement with previous reports.

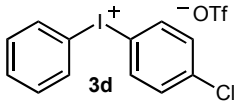
(4-Bromophenyl)(phenyl)iodonium triflate (3c):[6]



3c

Synthesized from 4-BrPhI + PhH in 78% yield or from PhI + PhBr in 71% as a white solid. Analytical data were in agreement with previous reports.

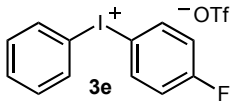
(4-Chlorophenyl)(phenyl)iodonium triflate (3d):[6]



3d

Synthesized from 4-ClPhI + PhH in 65% yield or from PhI + PhCl in 57% as a white solid. Analytical data were in agreement with previous reports.

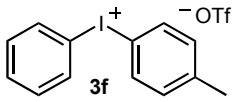
(4-Fluorophenyl)(phenyl)iodonium triflate (3e):[6]



3e

Synthesized from PhI + PhF in 92% yield as a white solid. Analytical data were in agreement with previous reports.

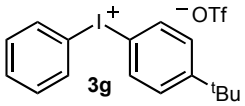
(4-Methylphenyl)(phenyl)iodonium triflate (3f):[5, 7]



3f

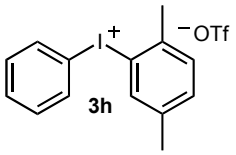
Synthesized from PhI + PhMe in 85% yield or from 4-MePhI + PhH in 71% yield as an off-white solid. The ^{13}C NMR given in reference 7 is in accordance with our data, whereas the ^{1}H NMR differs. mp: 120-121 °C (lit[7]: 122-125 °C); ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.21 (d, J = 7.6 Hz, 2H), 8.12 (d, J = 8.2 Hz, 2H), 7.66 (t, J = 7.6 Hz, 1H), 7.52 (t, J = 7.6 Hz, 2H), 7.34 (d, J = 8.2 Hz, 2H), 2.34 (s, 3H); IR (film): 3082, 3060, 2922, 1258, 1170, 1026 cm^{-1}.

(4-*tert*-Butylphenyl(phenyl)iodonium triflate (3g):[6]



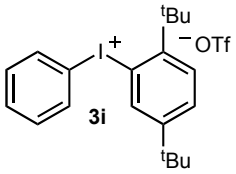
Synthesized from PhI + Ph^{t}Bu in 86% yield or from 4-^{t}BuPhI + PhH in 66% as a white solid. Analytical data were in agreement with previous reports.

(2,5-Dimethylphenyl)(phenyl)iodonium triflate (3h):[8]



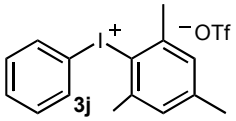
Synthesized from PhI + *p*-xylene in 66% yield as a off-white solid. Analytical data were in agreement with previous reports.

(2,5-Di-*tert*-butylphenyl)(phenyl)iodonium triflate (3i):



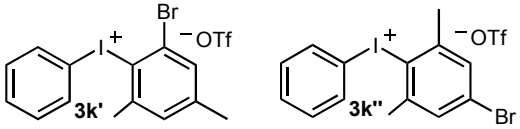
Synthesized from PhI and 1,4-^{t}Bu$_2$Ph in 80% yield as a white solid. mp: 160-161 °C; ^{1}H NMR (400 MHz, MeOD-d_4): δ 8.40 (s, 1H), 8.01 (d, J = 8.42 Hz, 2H), 7.73 (s, 2H), 7.68 (t, 7.44 Hz, 1H), 7.55 (app. t, J = 7.80 Hz, 2H), 1.53 (s, 9H), 1.34 (s, 9H); ^{13}C NMR (100 MHz, MeOD-d_4): δ 155.2, 149.2, 140.0, 135.1, 133.6, 133.3, 131.2, 130.7, 121.8 (q, J = 316.4 Hz, $CF_3SO_3^-$), 117.0, 116.0, 38.1, 35.8, 31.9, 31.2; IR (film): 2922, 1263, 1173, 1028 cm^{-1}; HRMS (ESI): calcd for $C_{20}H_{26}I$ ([M – TfO$^-$]$^+$): 393.1074; found 393.1066.

(2,4,6-Trimethylphenyl)(phenyl)iodonium triflate (3j):[7]



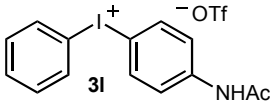
Synthesized from PhI + mesitylene in 78% yield as a white solid. Analytical data were in agreement with previous reports.

(1-Bromo-4,6-dimethylphenyl)(phenyl)iodonium triflate (3k') and (2,6-dimethyl-4-bromophenyl)(phenyl)iodonium triflate (3k''):



Synthesized from PhI + 3,5-Me$_2$PhBr in 94% yield as a regioisomeric mixture. Ratio **3k':3k''** = 1.2:1. ^{1}H NMR (400 MHz, MeOD-d_4): **3k'**: 8.08 (d, J = 7.6 Hz, 2H), 7.71-7.65 (m, 1H), 7.63 (s, 1 H), 7.57-7.51 (m, 2 H), 7.38 (s, 1 H), 2.78 (s, 3 H), 2.37 (s, 3 H). **3k''**: δ 7.96 (d, J = 7.6 Hz, 2 H), 7.71-7.65 (m, 1H), 7.63 (s, 2 H), 7.57-7.51 (m, 2 H), 2.70 (s, 6 H).

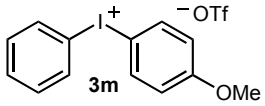
(4-Acetoaminophenyl)(phenyl)iodonium triflate (3l):[8]



Synthesized from PhI + acetanilide in 83% yield as a white solid. mp: 175-177 °C; ^{1}H NMR (400 MHz, DMSO-d$_6$): δ 10.30 (br, 1 H), 8.18 (dd, J = 8.2, 1.0 Hz, 2 H), 8.16 (d, J = 9.0 Hz,

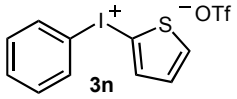
2 H), 7.69 (d, J = 9.0 Hz, 2 H), 7.65 (t, J = 7.6 Hz, 1 H), 7.52 (app t, J = 7.7 Hz, 2 H), 2.06 (s, 3 H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 169.1, 142.6, 136.3, 134.8, 131.9, 131.7, 121.4, 116.8, 107.7, 24.1; ^{19}F NMR (376 MHz, DMSO-d_6): δ -77.7; IR ($CHCl_3$): 3316.7, 3183.1, 1681.1, 1530.5, 1216.8, 1182.7, 1029.3 cm^{-1}; HRMS (ESI): calcd for $C_{14}H_{13}INO$ ([M – TfO^-]$^+$): 338.0036; found 338.0041.

(4-Methoxyphenyl)(phenyl)iodonium triflate (3m):[4, 5, 7]



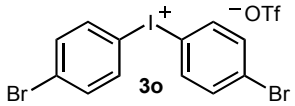
Synthesized from PhI + PhOMe in 87% yield. Analytical data were in agreement with previous reports.

(2-Thienyl)(phenyl)iodonium triflate (3n):[5, 9]



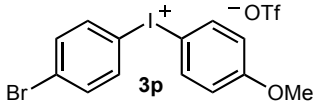
Synthesized from PhI + thiophene in 82% yield. Analytical data were in agreement with previous reports.

Bis(4-bromophenyl)iodonium triflate (3o):[10-12]



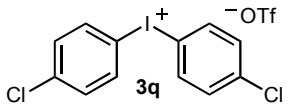
Synthesized from 4-BrPhI + PhBr in 91% yield or from PhBr + I_2 in 64% yield. Our analytical data match reference 11 but ^{1}H NMR is not in agreement with reference 10. mp: 203 °C (decomposed); ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.17 (d, J = 8.6 Hz, 4 H), 7.77 (d, J = 8.6 Hz, 4 H); ^{13}C NMR (100 MHz, CD_3OD): δ 138.1, 136.3, 128.8, 121.8 (q, J = 318.5 Hz, $CF_3SO_3^-$), 114.5; IR (film): 3084, 1471, 1260, 1173, 1028 cm^{-1}; HRMS (ESI): calcd for $C_{12}H_8Br_2I$ ([M – TfO^-]$^+$): 436.8032; found 436.8041.

(4-Bromophenyl)(4-methoxyphenyl)iodonium triflate (3p):



Synthesized from 4-BrPhI + PhOMe in 58% yield as a grey solid. mp: 143-144 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.17 (d, J = 9.0 Hz, 2H), 8.12 (d, J = 8.6 Hz, 2H), 7.74 (d, J = 8.6 Hz, 2H), 7.08 (d, J = 9.0 Hz, 2H), 3.80 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.1, 137.3, 136.7, 134.5, 126.0, 120.7 (q, J = 322.1 Hz, $CF_3SO_3^-$), 117.5, 115.5, 105.6, 55.7; IR (film): 3084, 1572, 1488, 1259, 1175, 1028 cm^{-1}; HRMS (ESI): calcd for $C_{13}H_{11}BrOI$ ([M – TfO^-]$^+$): 388.9032; found 388.9022.

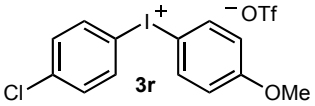
Bis(4-chlorophenyl)iodonium triflate (3q):[5, 10]



Synthesized from 4-ClPhI + PhCl in 83% yield or from PhCl + I_2 in 57% yield as a single regioisomer. Our analytical data are not in agreement with reference 10. mp: 181-183 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.26 (d, J = 8.6 Hz, 4H), 7.63 (d, J = 8.6 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 139.8, 136.8, 132.5, 120.0 (q, J = 318 Hz, $CF_3SO_3^-$), 111.2; IR (film):

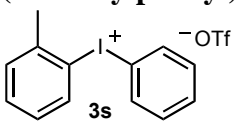
3086, 1260, 1172, 1026 cm^{-1}; HRMS (ESI): calcd for $C_{12}H_8Cl_2I$ ([M – TfO^-]$^+$): 348.9042; found 348.9041.

(4-Chlorophenyl)(4-methoxyphenyl)iodonium triflate (3r):



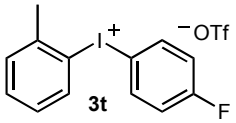
Synthesized from 4-ClPhI + PhOMe in 57% yield as a grey solid. mp: 129-130 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.19 (app. t, J = 8.9 Hz, 4H), 7.61 (d, J = 8.6 Hz, 2H), 7.08 (d, J = 8.9 Hz, 2H), 3.80 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.1, 137.2, 137.1, 136.6, 131.6, 120.7 (q, J = 322.4 Hz), 117.5, 114.8, 105.6, 55.7; IR (film):): 3087, 1572, 1488, 1258, 1175, 1028 cm^{-1}; HRMS (ESI): calcd for $C_{13}H_{11}ClOI$ ([M – TfO^-]$^+$): 344.9538; found 344.9534.

(2-Methylphenyl)(phenyl)iodonium triflate (3s):[5, 13]



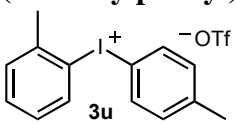
Synthesized from 2-MePhI + PhH in 85% yield as an off-white solid. mp: 162-164°C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.39 (d, J = 7.8 Hz, 1H), 8.20 (d, J = 7.7 Hz, 2H), 7.66 (t, J = 7.4 Hz, 1H), 7.57 (m, 2H),7.51 (t, J = 7.7 Hz, 2H), 7.32 (t, J = 6.2 Hz, 1H), 2.61 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): 140.6, 137.1, 135.0, 132.9, 132.0, 131.8, 131.4, 129.3, 121.4, 120.7 (q, J = 320 Hz, $CF_3SO_3^-$), 115.9, 25.0; HRMS (ESI): calcd for $C_{13}H_{12}I$ ([M – TfO^-]$^+$): 294.9978; found 294.9985.

(2-Methylphenyl)(4-fluorophenyl)iodonium triflate (3t):



Synthesized from 2-MePhI + PhF in 82% yield. mp: 161-162 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.40 (d, J = 8.0 Hz, 1 H), 8.27-8.30 (m, 2 H), 7.55-7.61 (m, 2 H), 7.40 (t, J = 8.7 Hz, 2 H), 7.32 (t, J = 7.1 Hz, 1 H), 2.62 (s, 3 H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 163.9 (d, J = 351.7 Hz), 140.54, 137.9 (d, J = 8.8 Hz), 137.0, 132.9, 131.5, 129.3, 121.8, 120.7 (q, J = 322.7 Hz, CF_3SO_3), 119.2 (d, J = 22.8 Hz), 110.0 (d, J = 3.1 Hz), 25.0; ^{19}F NMR (376 MHz, DMSO-d_6): δ -77.7, -106.8; IR (film): 3092.7, 3055.4, 1634.1, 1574.9, 1482.1, 1268.5, 1253.9, 1236.5, 1163.4, 1026.2 cm^{-1}; HRMS (ESI): calcd for $C_{13}H_{11}FI$ ([M – TfO^-]$^+$): 312.9884; found 312.9888.

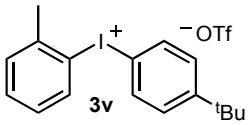
(2-Methylphenyl)(4-methylphenyl)iodonium triflate (3u):[5, 14]



Synthesized from 2-MePhI + PhMe in 87% yield; from PhMe + I_2 as a regioisomeric mixture with **3aa** (ratio **3u**:**3aa** 3:1) in 52% combined yield or as the single regioisomer in 31% yield. Analytical data were in agreement with previous reports. ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.36 (d, J = 8.6 Hz, 1H), 7.32 (d, J = 8.6 Hz, 2H), 7.57 (m, 2H), 7.32 (m, 3H) 2.60 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 142.5, 140.5, 137.0, 135.0, 132.8, 132.4,

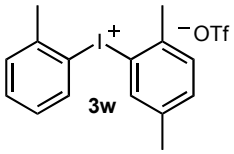
131.4, 129.3, 121.5, 120.7 (q, J = 321 Hz, $CF_3SO_3^-$), 112.2, 25.0, 20.8; HRMS (ESI): calcd for $C_{14}H_{14}I$ ([M – TfO^-]$^+$): 309.0135; found 309.0140.

(2-Methylphenyl)(4-*tert*-butylphenyl)iodonium triflate (3v):[14]



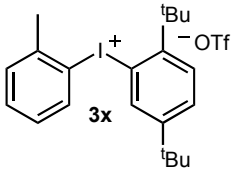
Synthesized from 2-MePhI + Ph^{t}Bu in 89% yield as a white solid. mp: 168-170 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.40 (d, J = 8.0 Hz, 1 H), 8.11 (d, J = 8.4 Hz, 2H), 7.60-7.52 (m, 4 H), 7.31 (app. t, 1 H), 2.62 (s, 3 H), 1.25 (9 H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 155.2, 140.5, 137.1, 134.8, 132.8, 131.4, 129.3, 128.9, 121.3, 120.7 (q, J = 320.3 Hz, $CF_3SO_3^-$), 119.1, 115.9, 112.3, 34.9, 30.7, 25.0; IR ($CHCl_3$): 2971, 2907, 1259, 1173, 1028 cm^{-1}; HRMS (ESI): calcd for $C_{17}H_{20}I$ ([M – TfO^-]$^+$): 351.0604; found 351.0601.

(2-Methylphenyl)(2,5-dimethylphenyl)iodonium triflate (3w):



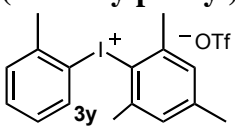
Synthesized from 2-MePhI + *p*-xylene in 62% yield as a white solid. mp: 140-141 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.28 (d, J = 8.0 Hz, 1H), 8.16 (s, 1H), 7.60-7.55 (m, 2H), 7.43 (d, J = 7.8 Hz, 1H), 7.39 (d, J = 7.8 Hz, 1 H), 7.32-7.28 (m, 1 H), 2.61 (s, 3H), 2.55 (s, 3H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 140.6, 139.1, 137.4, 137.1, 133.4, 132.7, 131.6, 131.2, 129.3, 120.7 (q, J = 320.3 Hz, $CF_3SO_3^-$), 120.3, 120.2, 25.0, 24.4, 20.0; IR ($CHCl_3$): 2925, 12578, 1173, 1036, 1027 cm^{-1}; HRMS (ESI): calcd for $C_{15}H_{16}I$ ([M – TfO^-]$^+$): 323.0291; found 323.0301.

(2-Methylphenyl)(2,5-bis(*tert*-butyl)phenyl)iodonium triflate (3x):



Synthesized from 2-MePhI + 1,4-^{t}Bu$_2$Ph in 51% yield as a white solid. mp: 102-103 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03 (dd, J = 8.1, 1.1 Hz, 1H), 7.65 (td, J = 7.6, 1.1 Hz, 1H), 7.58 (d, J = 8.4 Hz, 1H), 7.57 (m, 1H), 7.46 (dd, J = 8.4, 2.0 Hz, 1H), 7.35 (t, J = 7.7 Hz, 1H), 7.27 (d, J = 2.0 Hz, 1 H); 2.59 (s, 3 H), 1.55 (s, 9 H), 1.07 (s, 9 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.7, 147.4, 141.5, 137.9, 134.0, 132.5, 131.5, 130.0, 129.7, 128.8, 120.4 (q, J = 320.7 Hz, $CF_3SO_3^-$), 120.0, 112.3, 36.3, 34.6, 31.2, 30.6, 25.7; IR (film): 3085, 2965, 2873, 1596, 1480, 1460, 1260, 1157, 1028 cm^{-1}; HRMS (ESI): calcd for $C_{21}H_{28}I$ ([M – TfO^-]$^+$): 407.1230; found 407.1238.

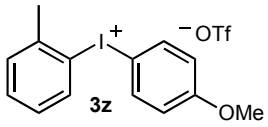
(2-Methylphenyl)(2,4,6-trimethylphenyl)iodonium triflate (3y):



Synthesized from 2-MePhI + mesitylene in 84% yield as a white solid. mp: 164-166 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.95 (d, J = 8 Hz), 7.58-7.55 (m, 2 H), 7.28-7.24 (m, 1 H), 7.21 (s, 2 H), 2.57-2.56 (m, 9 H), 2.29 (s, 3 H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 143.0, 141.6, 140.7, 136.7, 132.4, 131.9, 129.9, 129.3, 121.8, 120.7 (q, J = 320.6 Hz, $CF_3SO_3^-$),

118.6, 26.1, 24.4, 20.4; IR ($CHCl_3$): 2922, 1262, 1173, 1037, 1028 cm^{-1}; HRMS (ESI): calcd for $C_{16}H_{18}I$ ($[M - TfO^-]^+$): 337.0448; found 337.0453.

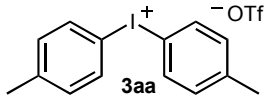
(2-Methylphenyl)(4-methoxyphenyl)iodonium triflate (3z):



3z

Synthesized from 2-MePhI + anisole in 56% yield as a white solid. mp: 171-172 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.36 (dd, J = 7.9, 1.0 Hz, 1 H), 8.13 (dt, J = 9.2 Hz, 2 H), 7.52-7.58 (m, 2 H), 7.28-7.32 (m, 1 H), 7.05 (dt, J = 9.2 Hz, 2 H), 3.79 (s, 3 H), 2.61 (s, 3 H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 161.9, 140.3, 137.1, 136.8, 132.7, 131.3, 129.2, 121.9, 120.7 (q, J = 322.4 Hz), 117.5, 104.8, 55.7, 24.9; ^{19}F NMR (376 MHz, DMSO-d_6): δ -77.7; IR (film): 3089.5, 2924.0, 1572.9, 11488.1, 1258.8, 1175.5, 1026.5 cm^{-1}; HRMS (ESI): calcd for $C_{14}H_{14}IO$ ($[M - TfO^-]^+$): 325.0084; found 325.0092.

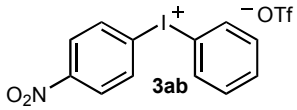
Bis(4-methylphenyl)iodonium triflate (3aa):[5, 15]



3aa

Synthesized from 4-MePhI + toluene in 52% yield as an off-white solid. The ^{13}C NMR given in reference 15 is in accordance with our data, whereas the ^{1}H NMR differs. ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.08 (d, J = 8.0 Hz, 4H), 7.32 (d, J = 8.0 Hz, 4H), 2.34 (s, 6H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 142.5, 135.0, 132.3, 120.7 (q, J = 320 Hz, $CF_3SO_3^-$), 113.0, 20.8; HRMS (ESI): calcd for $C_{14}H_{14}I$ ($[M - TfO^-]^+$): 309.0135; found 309.0131.

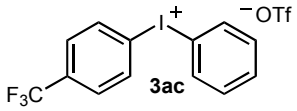
(4-Nitrophenyl)(phenyl)iodonium triflate (3ab):[5, 6]



3ab

Synthesized from 4-NO_2PhI + PhH in 85% yield as a white solid. Analytical data were in agreement with previous reports.

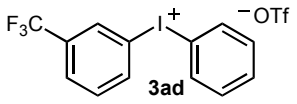
(4-Trifluoromethylphenyl)(phenyl)iodonium triflate (3ac):



3ac

Synthesized from 4-CF_3PhI + PhH in 59% yield as a white solid. mp: 120-122 °C; ^{1}H NMR (400 MHz, MeOD-d_4): δ 8.37 (d, J = 8.4 Hz, 2H), 8.24 (d, J = 8.3 Hz, 2H), 7.83 (d, J = 8.4 Hz, 2H), 7.73 (t, J = 7.6 Hz, 1H), 7.57 (app. t, J = 7.8 Hz, 2H); ^{13}C NMR (100 MHz, MeOD-d_4): δ 135.7, 135.3, 133.7 (q, J = 33.0 Hz), 132.6, 132.0, 128.3 (q, J = 3.7 Hz), 123.3 (q, J = 270.6 Hz), 120.4 (q, J = 316.0 Hz), 118.4, 114.8; IR (film): 3091, 1595, 1259, 1173, 1028 cm^{-1}; HRMS (ESI): calcd for $C_{13}H_9F_3I$ ($[M - TfO^-]^+$): 348.9696; found 348.9681.

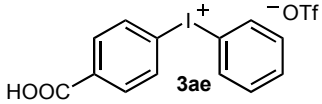
(3-Trifluoromethylphenyl)(phenyl)iodonium triflate (3ad):[6]



3ad

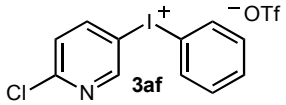
Synthesized from 3-CF_3PhI + PhH in 63% yield as a white solid. Analytical data were in agreement with previous reports.

(4-Carboxyphenyl)(phenyl)iodonium triflate (3ae):



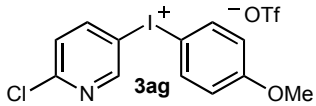
Synthesized from 4-(COOH)PhI + PhH in 73% yield as a off-white solid. mp: 175-176 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 13.50 (s, 1H), 8.34 (d, J = 8.6 Hz, 2H), 8.27 (d, J = 7.2 Hz, 2H), 8.00 (d, J = 8.6 Hz, 2H), 7.69 (t, J = 7.6 Hz, 1H), 7.55 (app. t, J = 7.8 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 166.1, 135.4, 133.9, 132.2, 132.1, 131.9, 120.8, 120.7 (q, J = 320.1 Hz), 116.6; IR ($CHCl_3$): 3020 (br), 2823, 1715, 1582, 1259, 1173, 1037 cm^{-1}; HRMS (ESI): calcd for $C_{13}H_{10}IO_2$ ([M – TfO–]$^+$): 324.9720; found 324.9710.

(6-Chloro-pyridin-3-yl)(phenyl)iodonium triflate (3af):[5, 16]



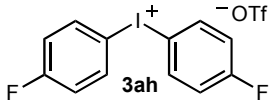
Synthesized from 2-chloro-5-iodopyridine + PhH in 60% yield as a white solid. mp: 153-155 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.17 (d, J = 2.3 Hz, 1H), 8.70 (dd, J = 8.5, 2.3 Hz, 1H), 8.28 (d, J = 7.6 Hz, 2H), 7.75 (d, J = 8.5 Hz, 1H), 7.70 (t, J = 7.6 Hz, 1H), 7.56 (t, J = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 154.3, 153.4, 145.5, 135.2, 132.4, 132.0, 127.6, 120.7 (q, J = 322 Hz, CF$_3$SO$_3^-$), 116.9, 114.2; IR (film): 3084, 3055, 1554, 1445, 1260, 1172, 1027 cm^{-1}; HRMS (ESI): calcd for $C_{11}H_8ClIN$ ([M – TfO$^-$]$^+$): 315.9384; found 315.9393.

(6-chloro-pyridin-3-yl)(4-methoxyphenyl)iodonium triflate (3ag):[5]



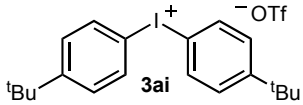
Synthesized from 2-chloro-5-iodopyridine + PhOMe in 53% yield. mp: 101-103 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.12 (d, J = 2.1 Hz, 1H), 8.64 (dd, J = 8.5, 2.1 Hz, 1H), 8.20 (d, J = 8.9 Hz, 2H), 7.72 (d, J = 8.5 Hz, 1H), 7.10 (d, J = 8.9 Hz, 2H) 3.80 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 162.2, 154.0, 153.2, 145.3, 137.3, 127.5, 120.7 (q, J = 321 Hz, CF$_3$SO$_3^-$), 117.7, 114.6, 105.7, 55.8; IR (film): 3090, 3047, 1572, 1443, 1258, 1172, 1026 cm^{-1}; HRMS (ESI): calcd for $C_{12}H_{10}ClINO$ ([M – TfO$^-$]$^+$): 345.9490; found 345.9501.

Bis(4-fluorophenyl)iodonium triflate (3ah):[10]



Synthesized from PhF + I_2 in 71% yield. The ^{13}C NMR given in reference 10 is in accordance with our data, whereas the ^{1}H NMR differs. mp: 101 °C (decomposed); ^{1}H NMR (400 MHz, CD_3OD): δ 8.26-8.21 (m, 4 H), 7.34-7.27 (m, 4 H); ^{13}C NMR (100 MHz, CD_3OD): δ 166.5 (d, J = 255.5 Hz), 139.3 (d, J = 8.9 Hz), 121.8 (q, J = 318.6 Hz, CF$_3$SO$_3^-$), 120.6 (d, J = 23.1 Hz), 110.2 (J = 2.3 Hz); IR ($CHCl_3$): 2977, 2920, 1636, 1260, 1174, 1036 cm^{-1}; HRMS (ESI): calcd for $C_{12}H_8F_2I$ ([M – TfO$^-$]$^+$): 316.9633; found 316.9632.

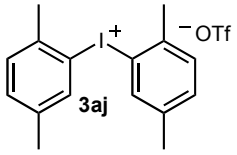
Bis(4-*tert*-butylphenyl)iodonium triflate (3ai):[4, 10]



Synthesized from *t*-butylbenzene + I_2 in 78% yield. The NMR data given in reference 10 are not accordance with our data. mp: 153-155 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (dt, J =

8.9, 2.5 Hz, 4H), 7.45 (dt, *J* = 8.9, 2.5 Hz, 4H), 1.29 (s, 18H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.5, 134.9, 129.6, 120.2 (q, *J* = 320.5 Hz, *C*$F_3SO_3^-$), 109.4, 35.2, 30.9; IR (film): 2966, 2907, 2871, 1643, 1254, 1163, 1030 cm^{-1}; HRMS (ESI): calcd for $C_{20}H_{26}I$ ([M – TfO^-]$^+$): 393.1074; found 393.1066.

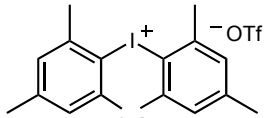
Bis(2,5-dimethylphenyl)iodonium triflate (3aj):



3aj

Synthesized from p-xylene + I_2 in 43% yield. mp: 173-175 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74 (m, 2 H), 7.33 (m, 4 H), 2.56 (s, 6 H), 2.34 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.3, 138.0, 136.8, 134.2, 131.8, 120.3 (q, *J* = 320.9 Hz, *C*$F_3SO_3^-$), 116.9, 24.9, 20.6; ^{19}F NMR (376 MHz, $CDCl_3$): δ -78.4; IR (film): 2924, 1645, 1271, 1159, 1030 cm^{-1}; HRMS (ESI): calcd for $C_{16}H_{18}I$ ([M – TfO^-]$^+$): 337.0448; found 337.0444.

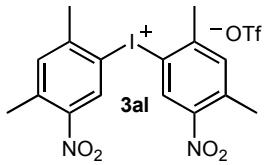
Bis(2,4,6-trimethylphenyl)iodonium triflate (3ak):[12, 14]



3ak

Synthesized from mesitylene + I_2 in 24% yield. mp: 187-188 °C (decomposed); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.04 (s, 4 H), 2.50 (s, 12 H), 2.32 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.80, 142.2, 130.9, 120.4 (q, *J* = 320.3 Hz, *C*$F_3SO_3^-$), 117.2, 26.1, 20.9; IR ($CHCl_3$): 2925, 1636, 1453, 1256, 1166, 1030 cm^{-1}; HRMS (ESI): calcd for $C_{18}H_{22}I$ ([M – TfO^-]$^+$): 365.0761; found 365.0757.

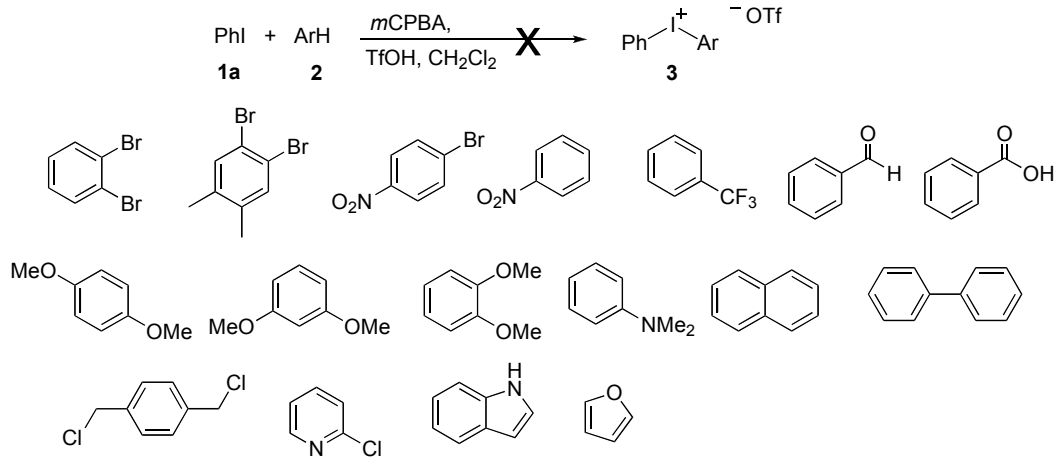
Bis(2,4-dimethyl-5-nitrophenyl)iodonium triflate (3al):[5]



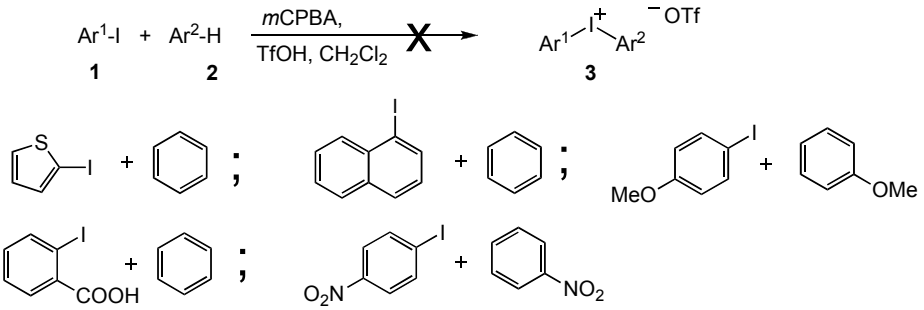
3al

Synthesized from 2,4-Me_2-nitrobenzene + I_2 in 24% yield as a single regioisomer. mp: 200-202 °C; ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.10 (s, 2H), 7.70 (s, 2H), 2.65 (s, 6H), 2.53 (s, 6H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 147.5, 146.4, 137.9, 135.1, 132.9, 120.7 (q, *J* = 320 Hz, *C*$F_3SO_3^-$), 117.4, 24.7, 19.6; IR (film): 3095, 1523, 1342, 1261, 1173, 1024 cm^{-1}; HRMS (ESI): calcd for $C_{16}H_{16}IN_2O_4$ ([M – TfO^-]$^+$): 427.0149; found 427.0145.

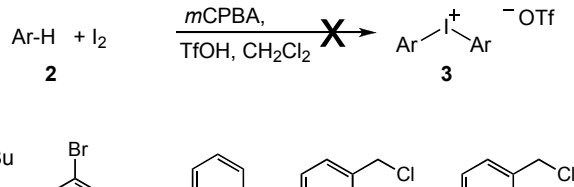
4 Limitations to the developed protocol



Scheme 1. Arenes that gave no or poor yield of the desired iodonium salts with iodobenzene.



Scheme 2. Aryl iodide and arene combinations that gave no or poor yield of the desired iodonium salts.



Scheme 3: Substrates that gave no or poor yield in reactions with iodine.

References

[1] Complete transformation of iodine to two iodine(III) molecules would require 3 equivalents of mCPBA.

[2] Iodobenzene was detected in reactions with short reaction time, supporting the assumption that iodobenzene is an intermediate in the reaction.

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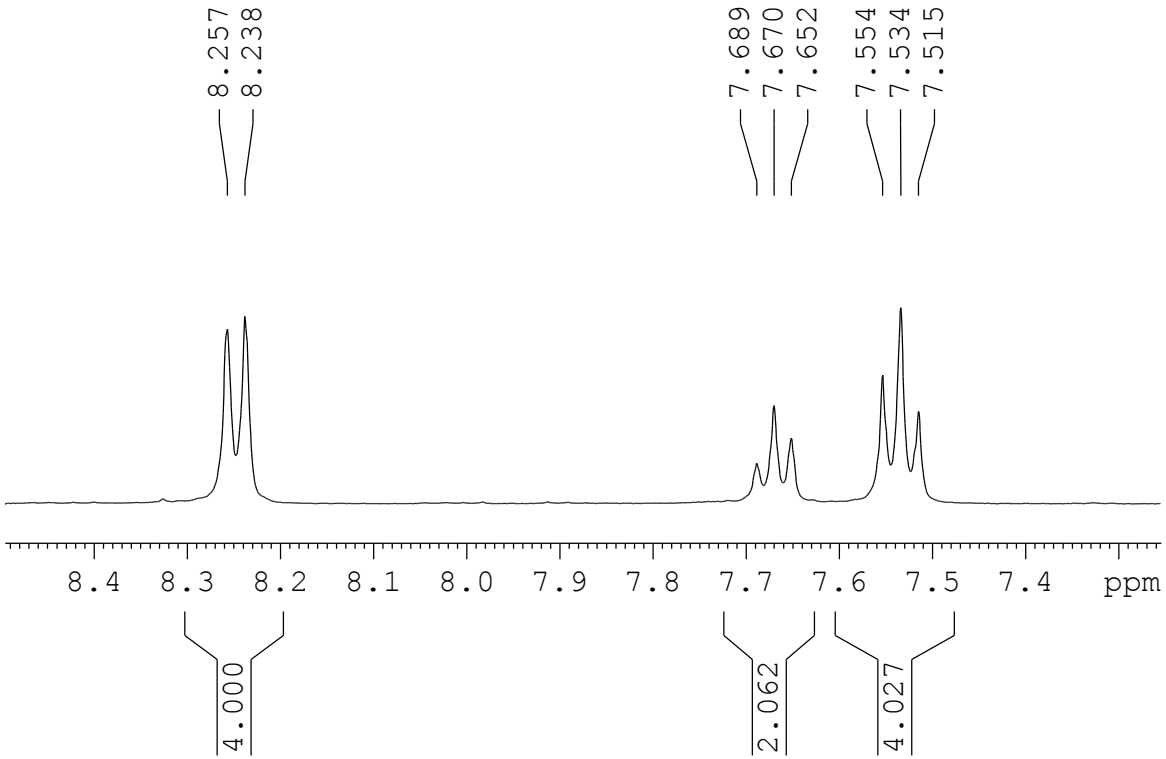
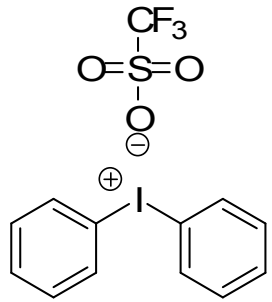
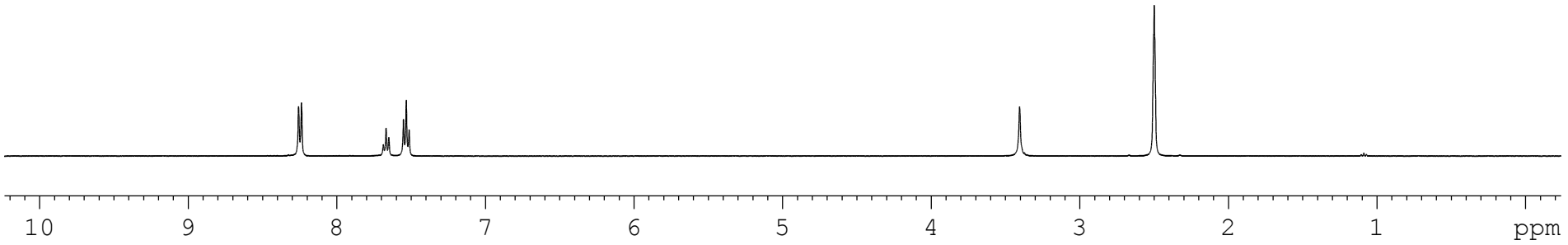
5 NMR spectra of diaryliodonium salts 3

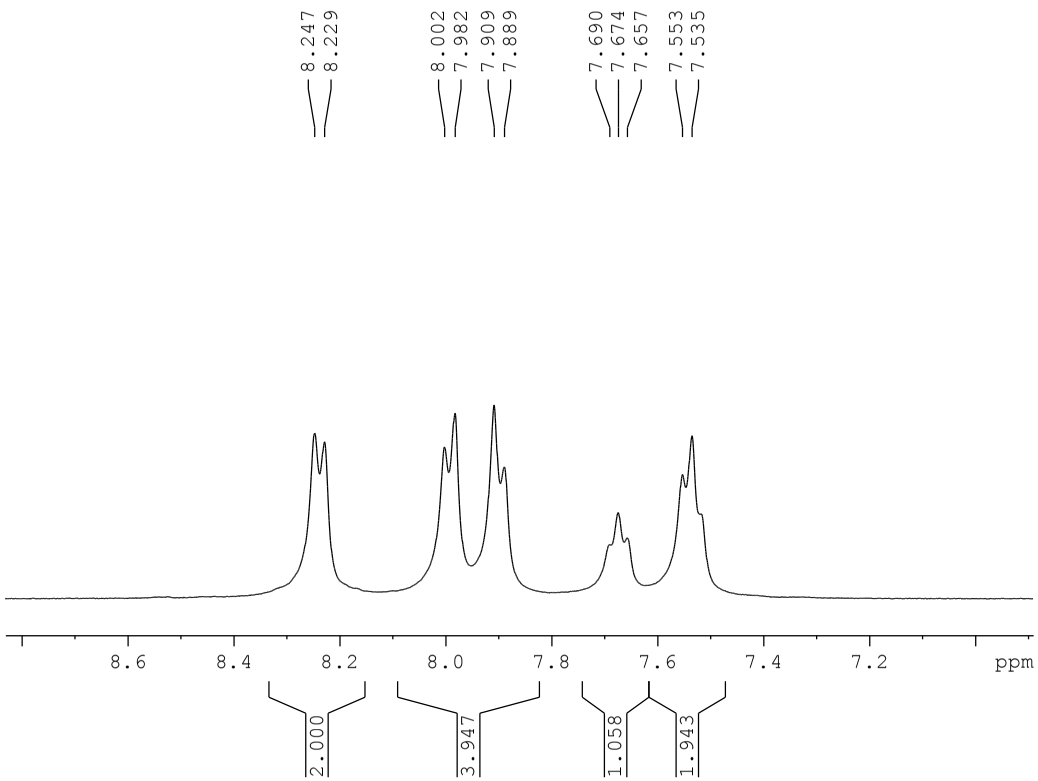


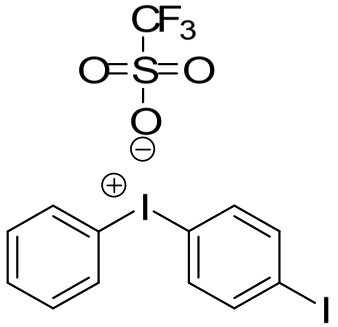


400 MHz
DMSO

3a

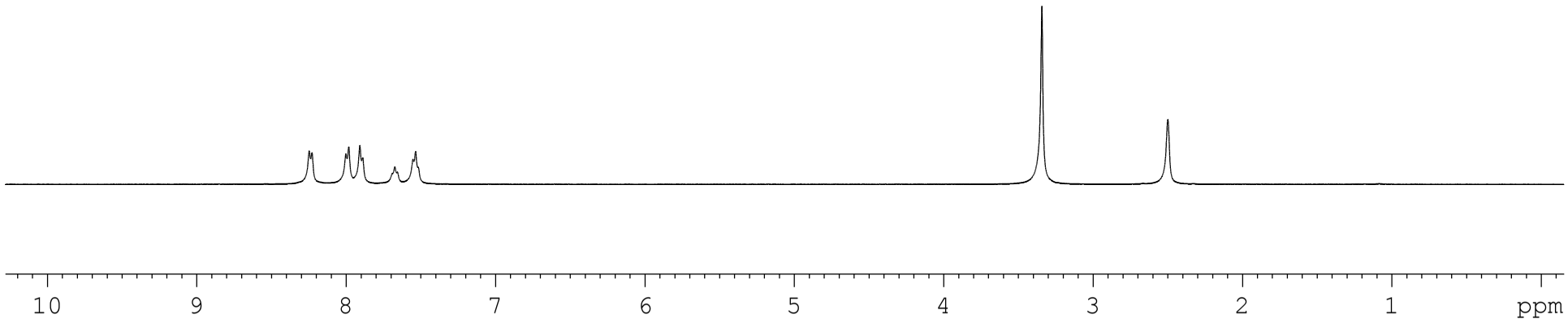


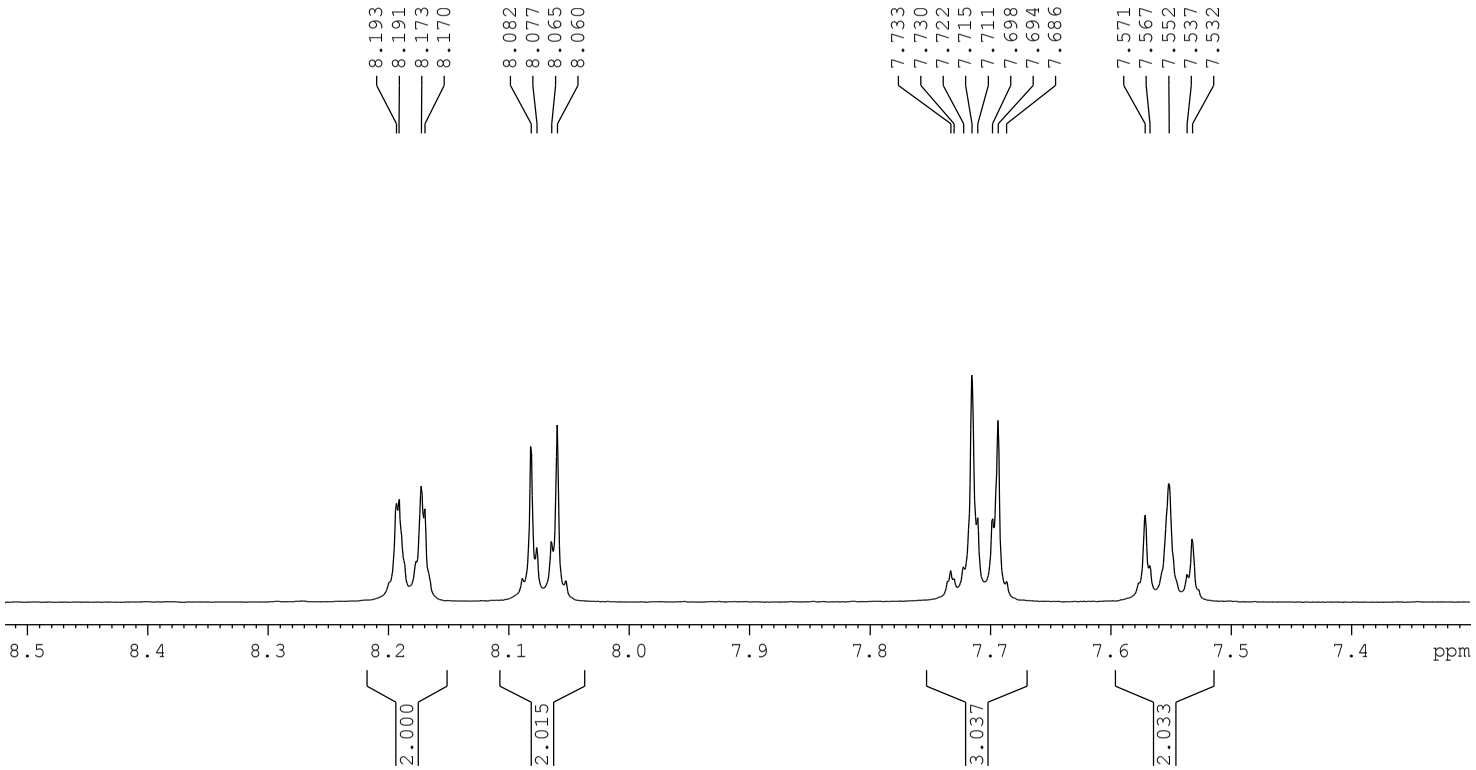


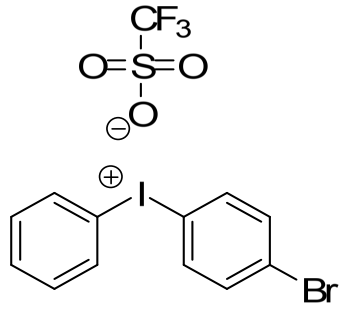


400 MHz
DMSO

3b

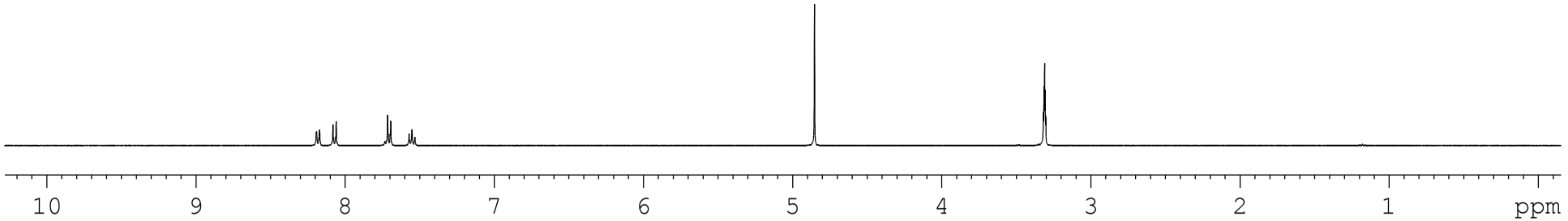


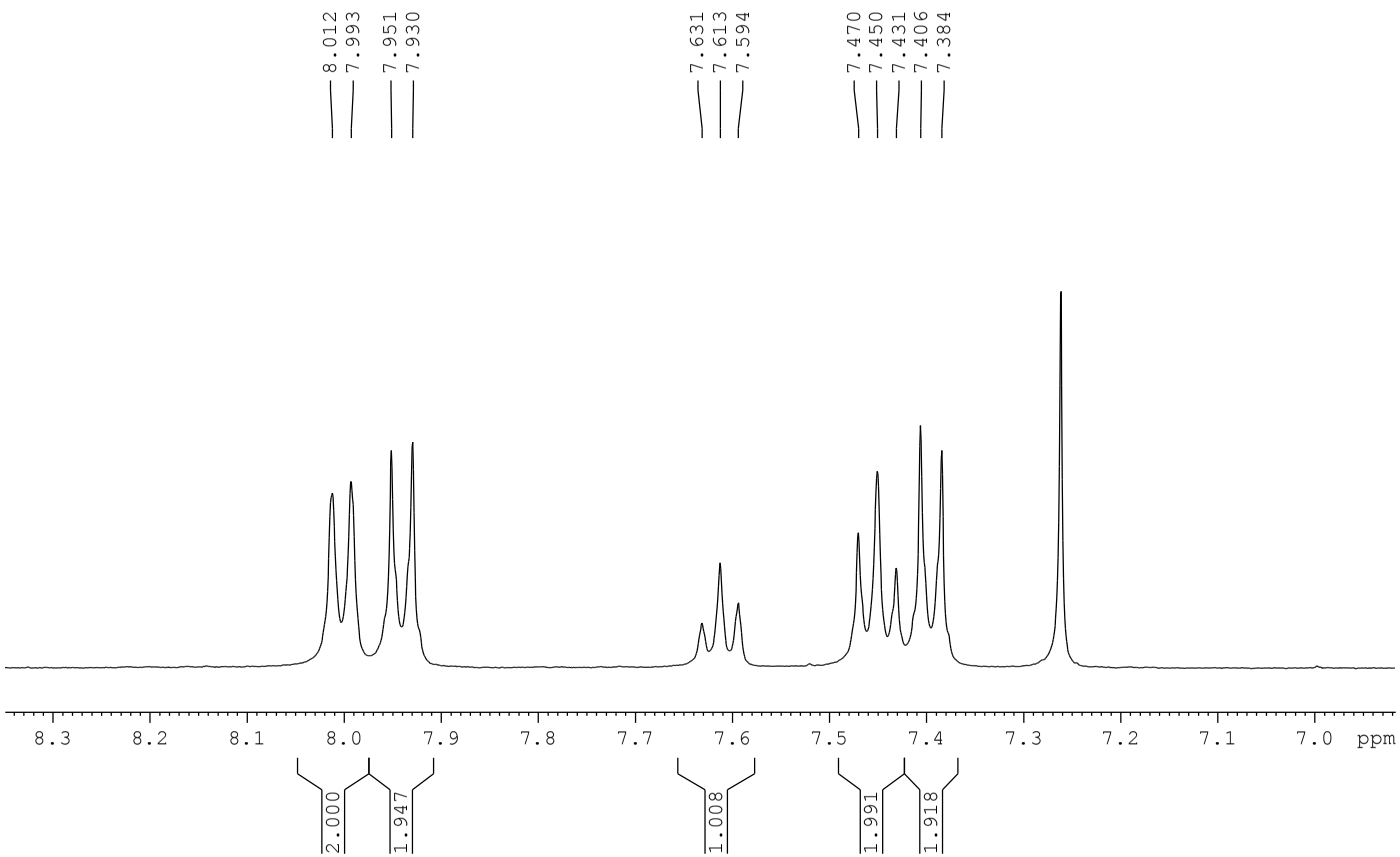


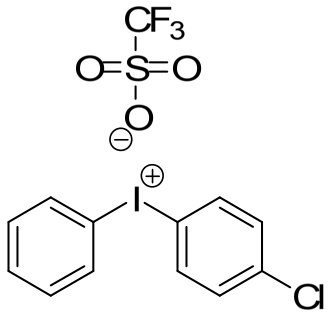


400 MHz
MeOD

3c

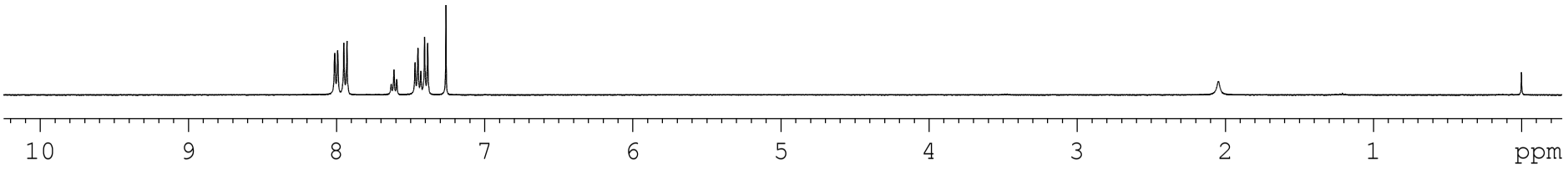


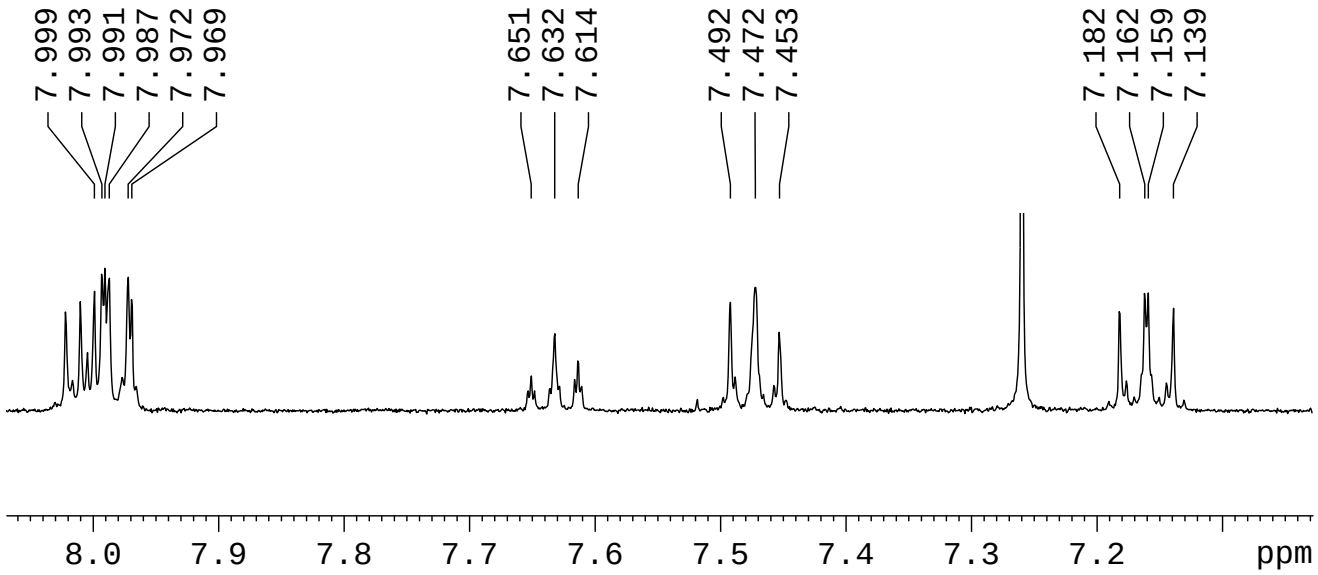




400 MHz
CDCl3

3d

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7.991
7.987
7.972
7.969
7.651
7.632
7.614
7.492
7.472
7.453
7.182
7.162
7.159
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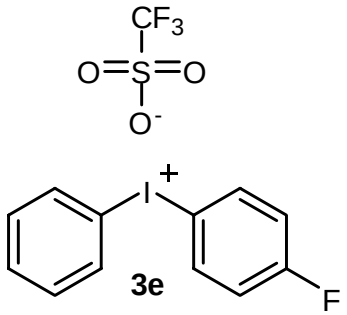
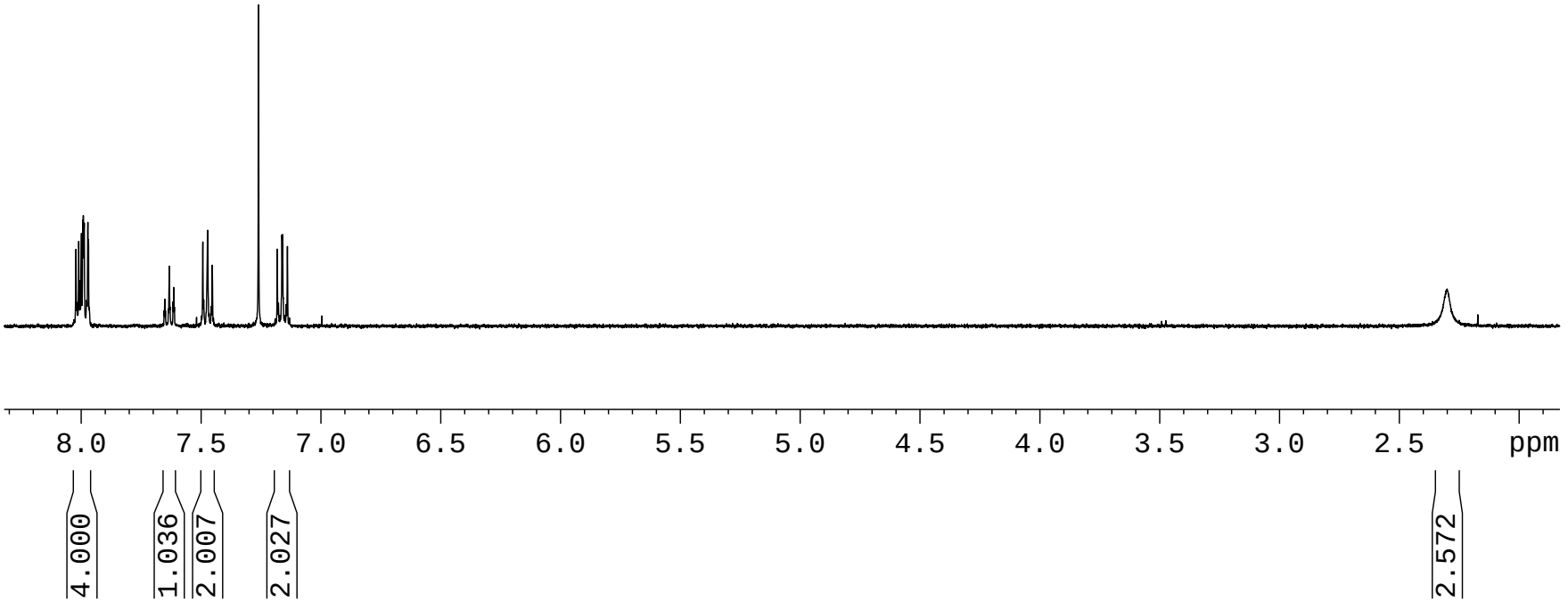
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
ppm


CF3
O
S
O
O
O-
I+
3e
F

400 Hz
$CDCl_3$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6.0
5.5
5.0
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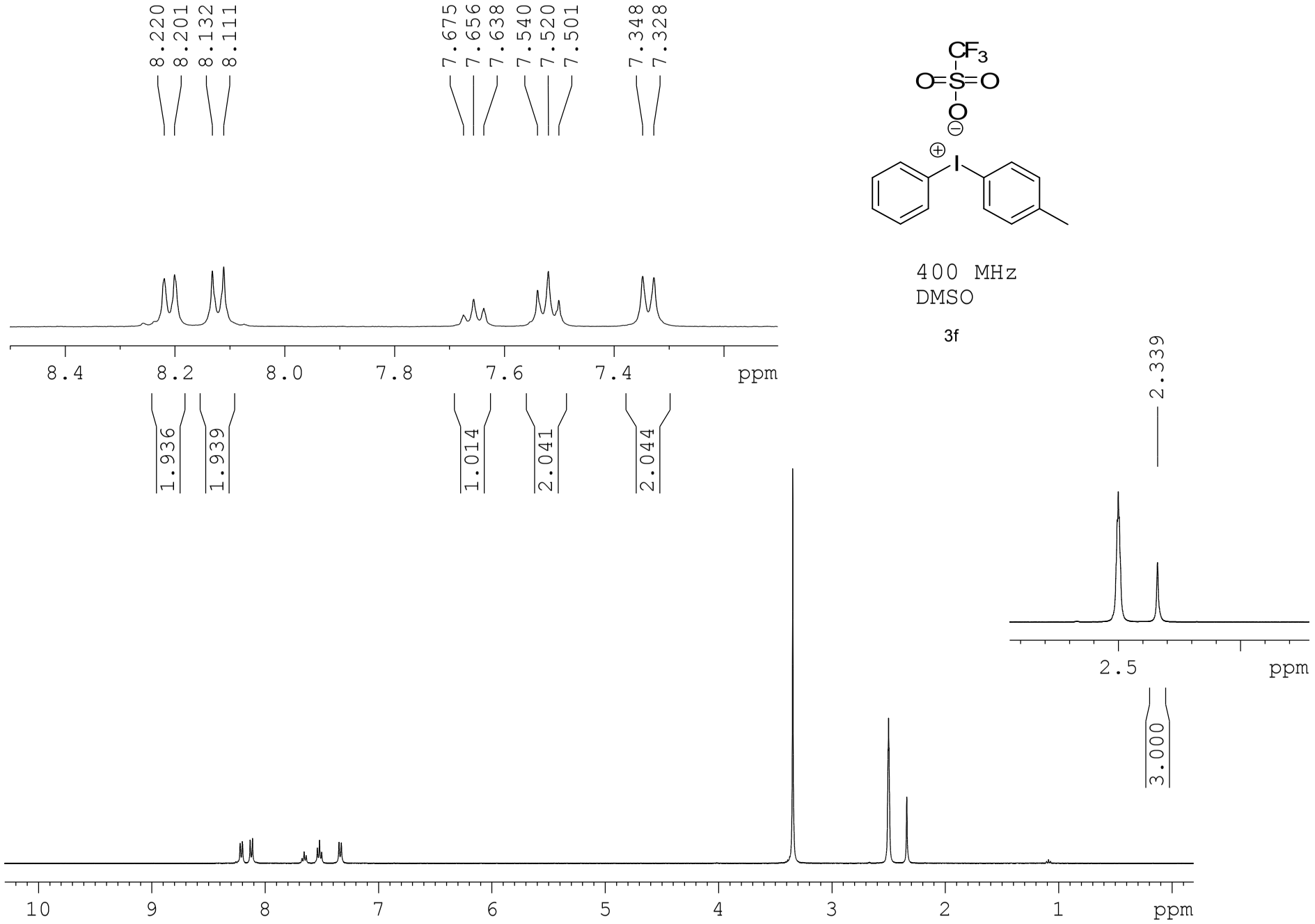
3.5
3.0
2.5
ppm
4.000
1.036
2.007
2.027
2.572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7.540
7.520
7.501
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400 MHz
DMSO
3f
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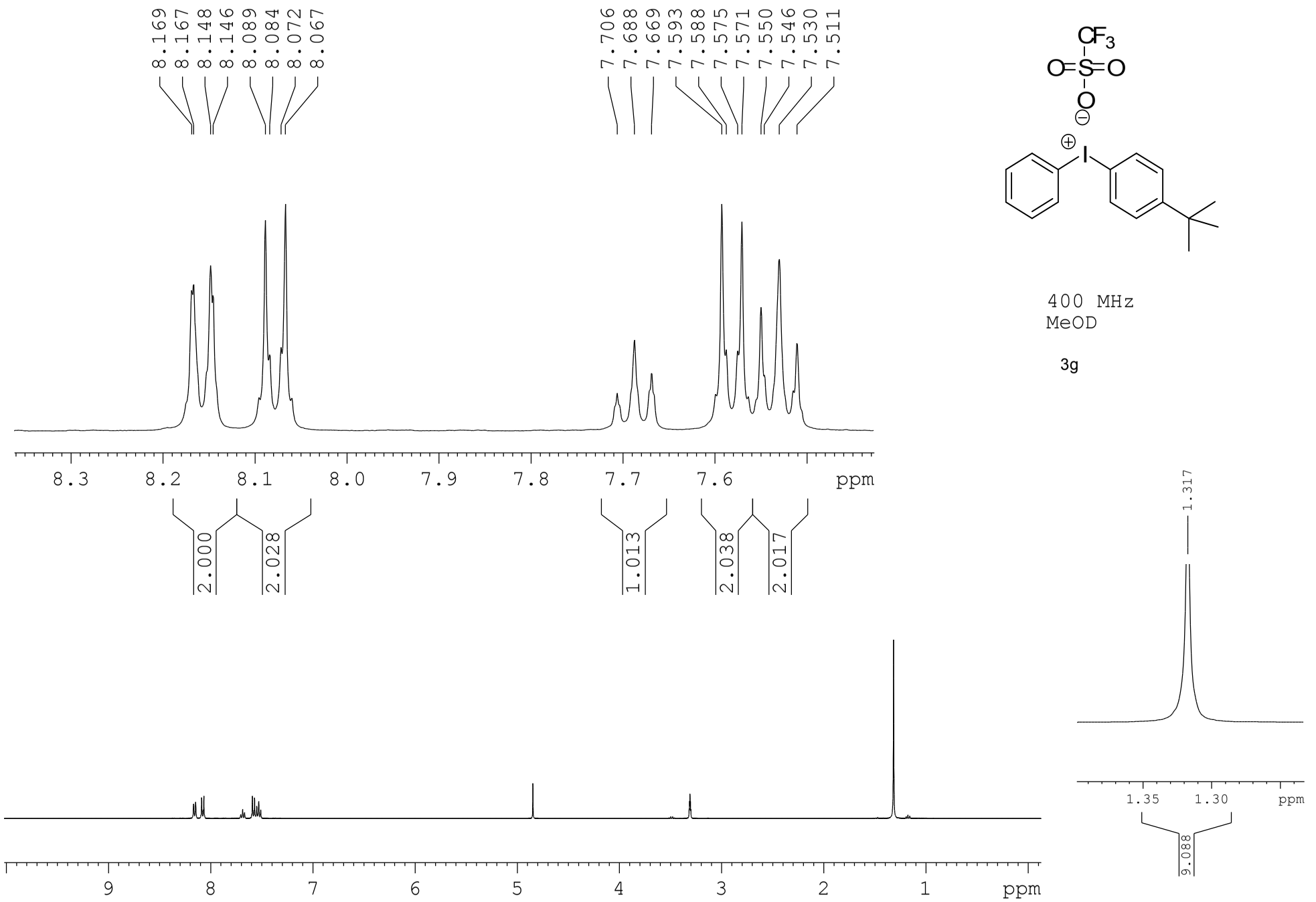
2.044
2.339
3.000
ppm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7.669
7.593
7.588
7.575
7.571
7.550
7.546
7.530
7.511
CF3
O=S=O
O
I
400 MHz
MeOD
3g
8.3
8.2
8.1
8.0
7.9
7.8
7.7
7.6
ppm
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2.028
1.013
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ppm
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7
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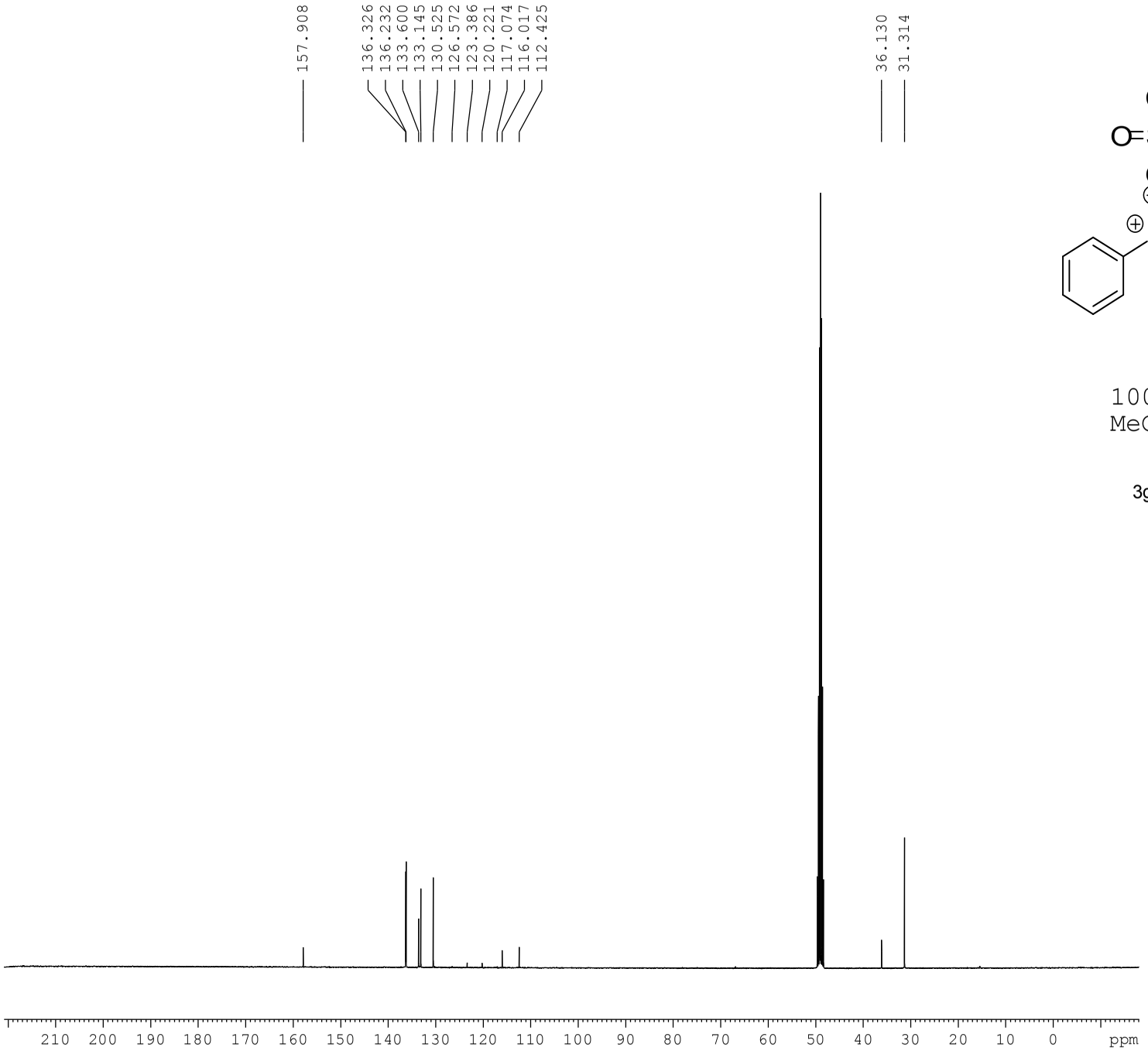
3
2
1
ppm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O
I
100 MHz
MeOD
3g
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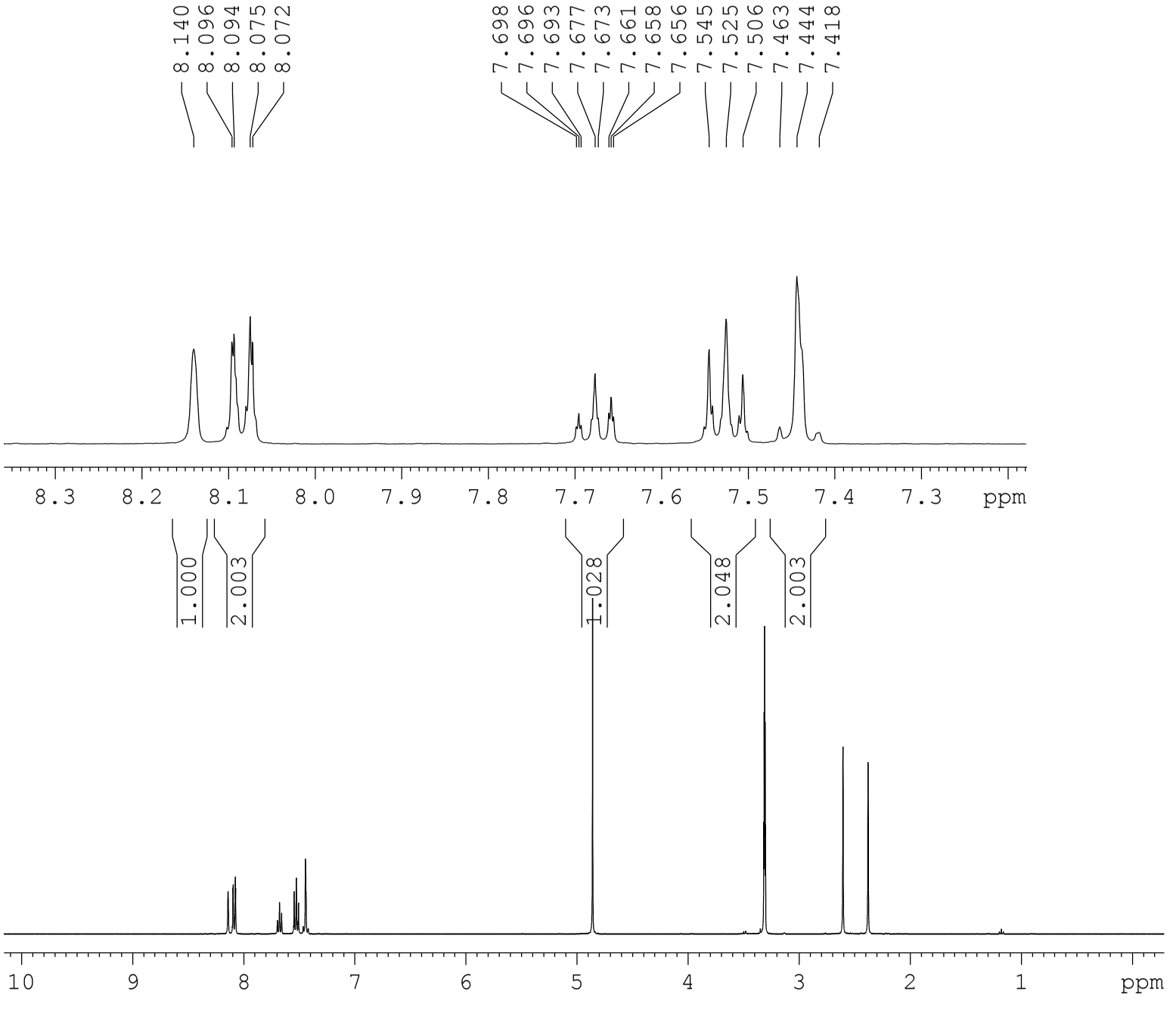
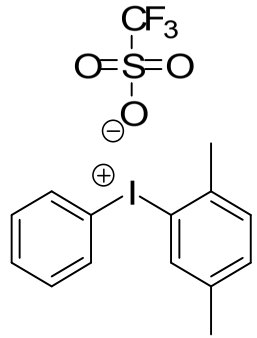
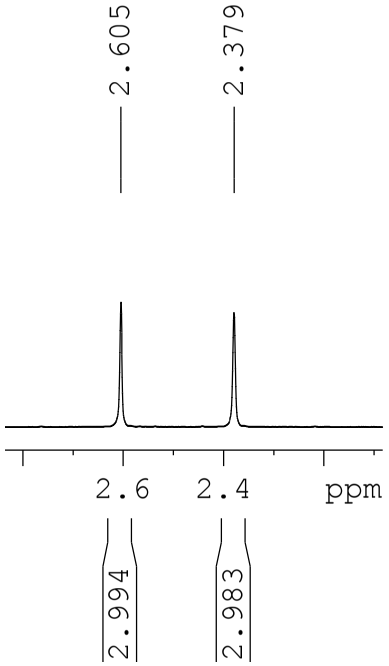
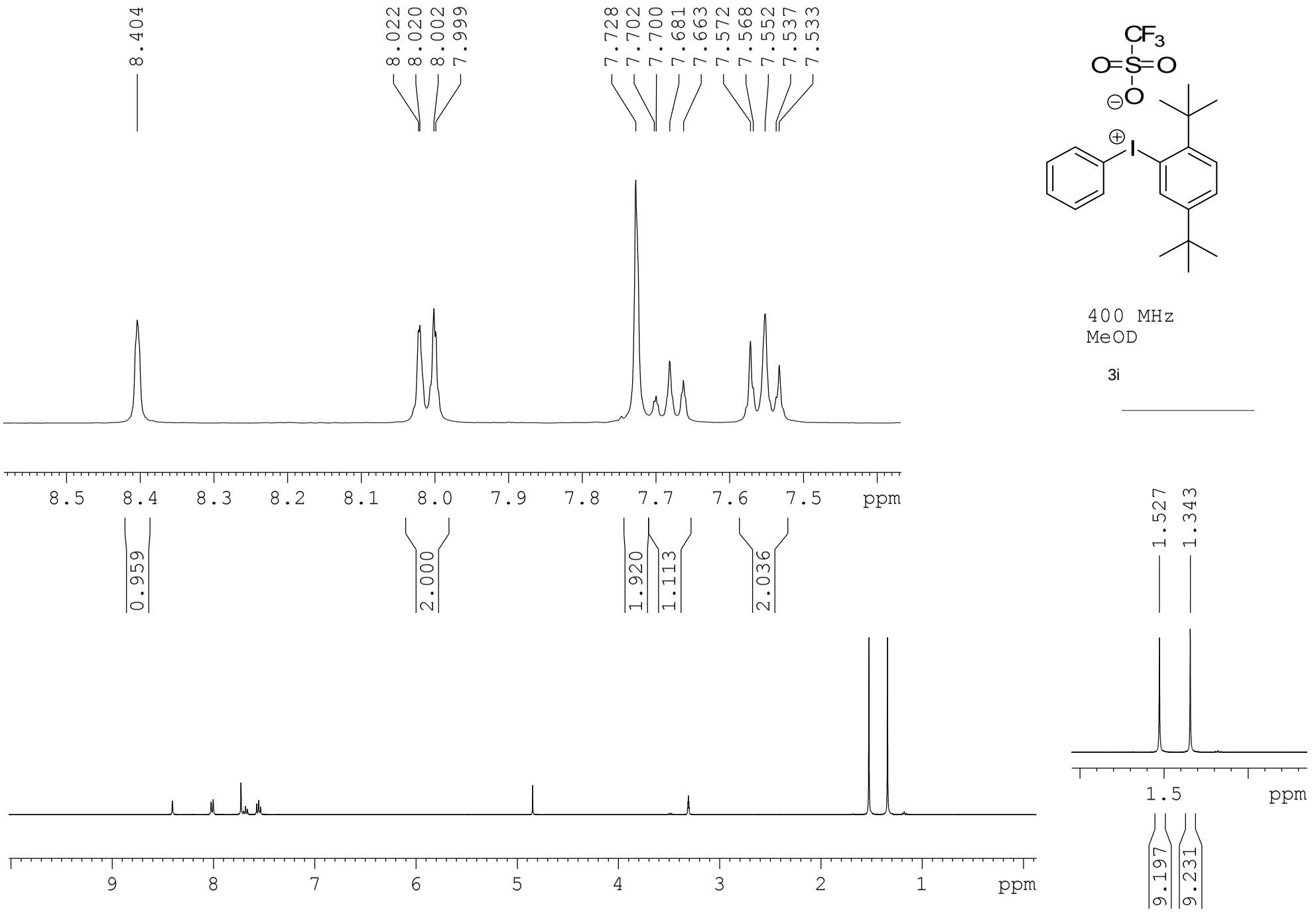




400 MHz
MeOD

3h

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8.3
8.2
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7.7
7.6
7.5
ppm
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9
8
7
6
5
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MeOD
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ppm
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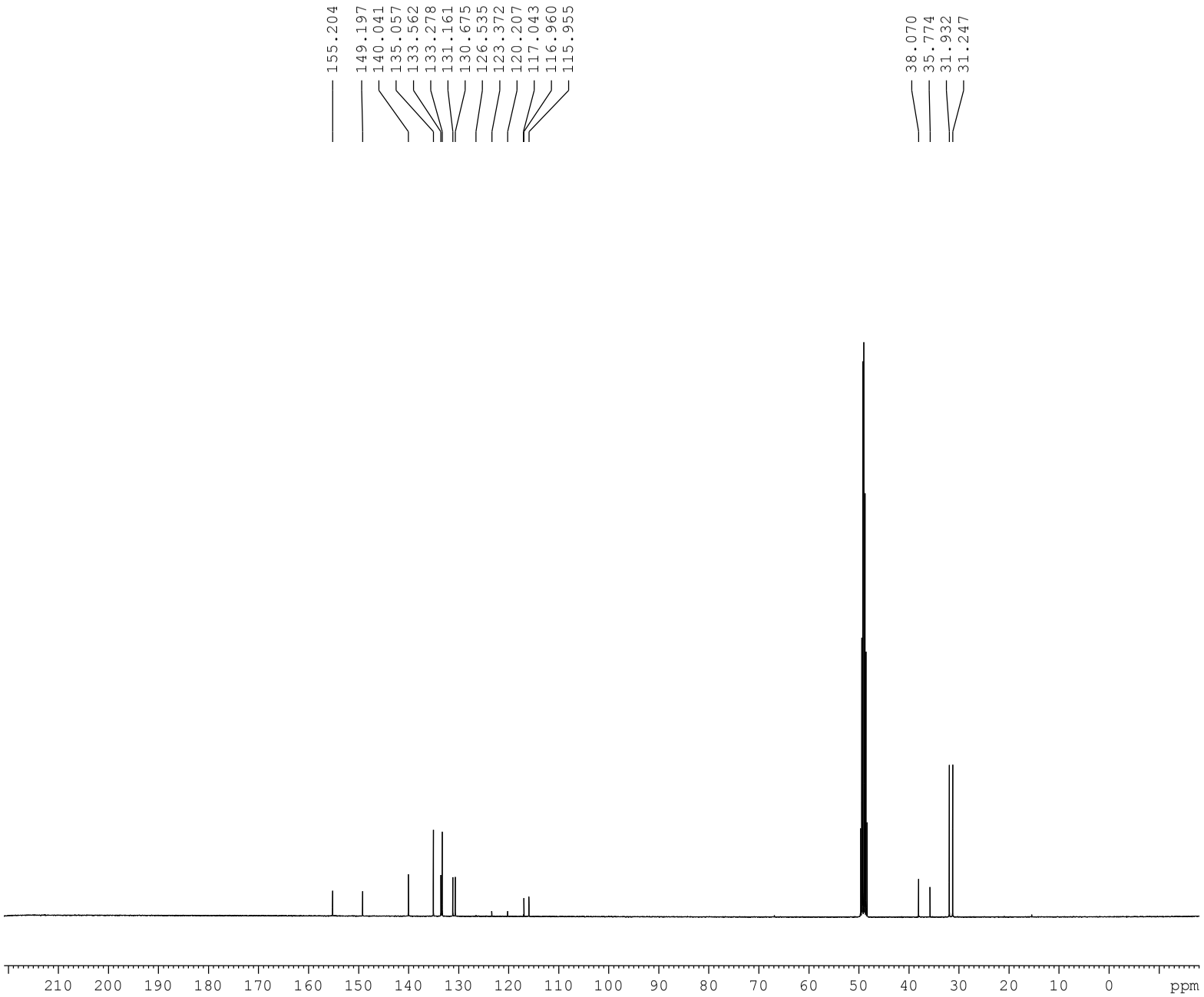
9.231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115.955
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35.774
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100 MHz
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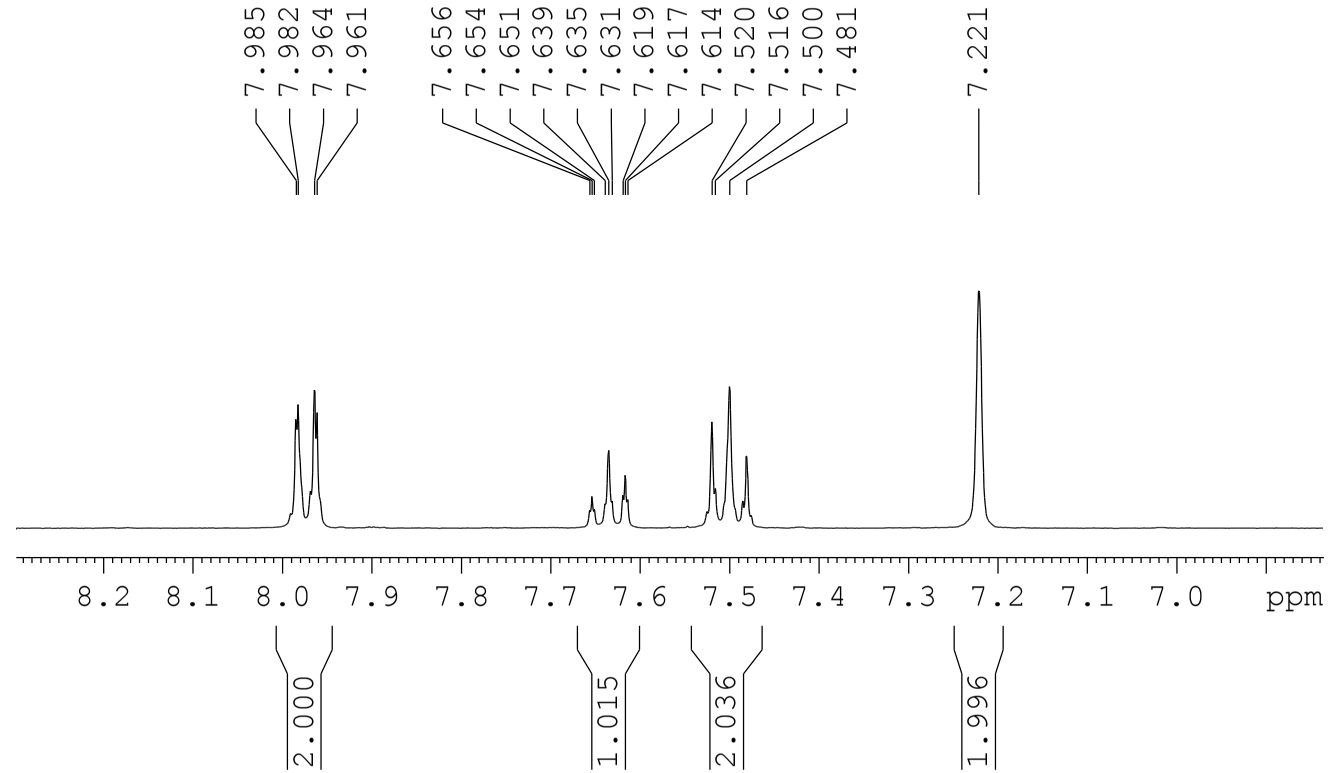
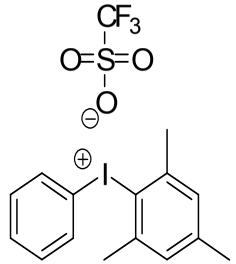
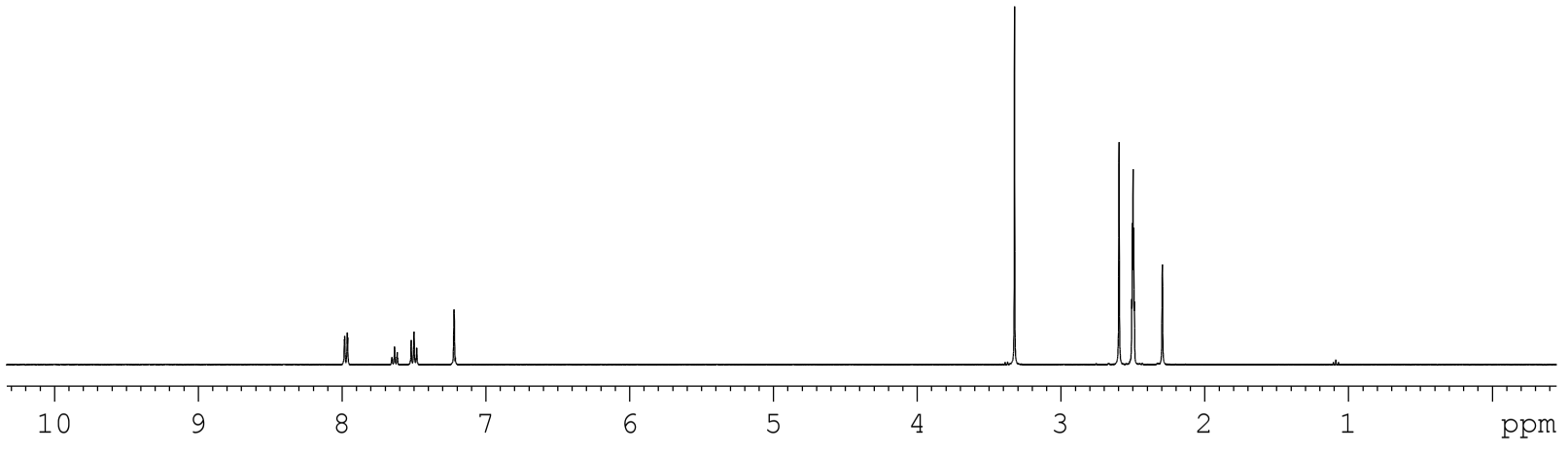
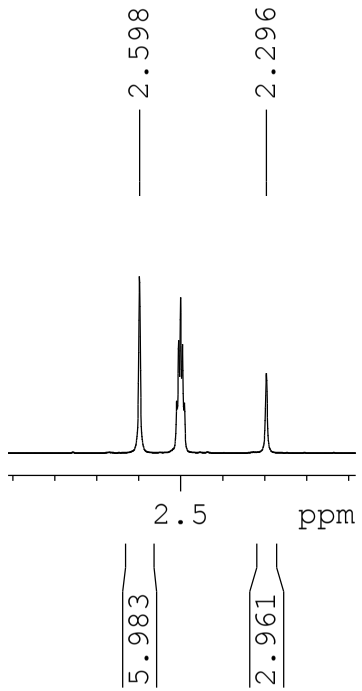


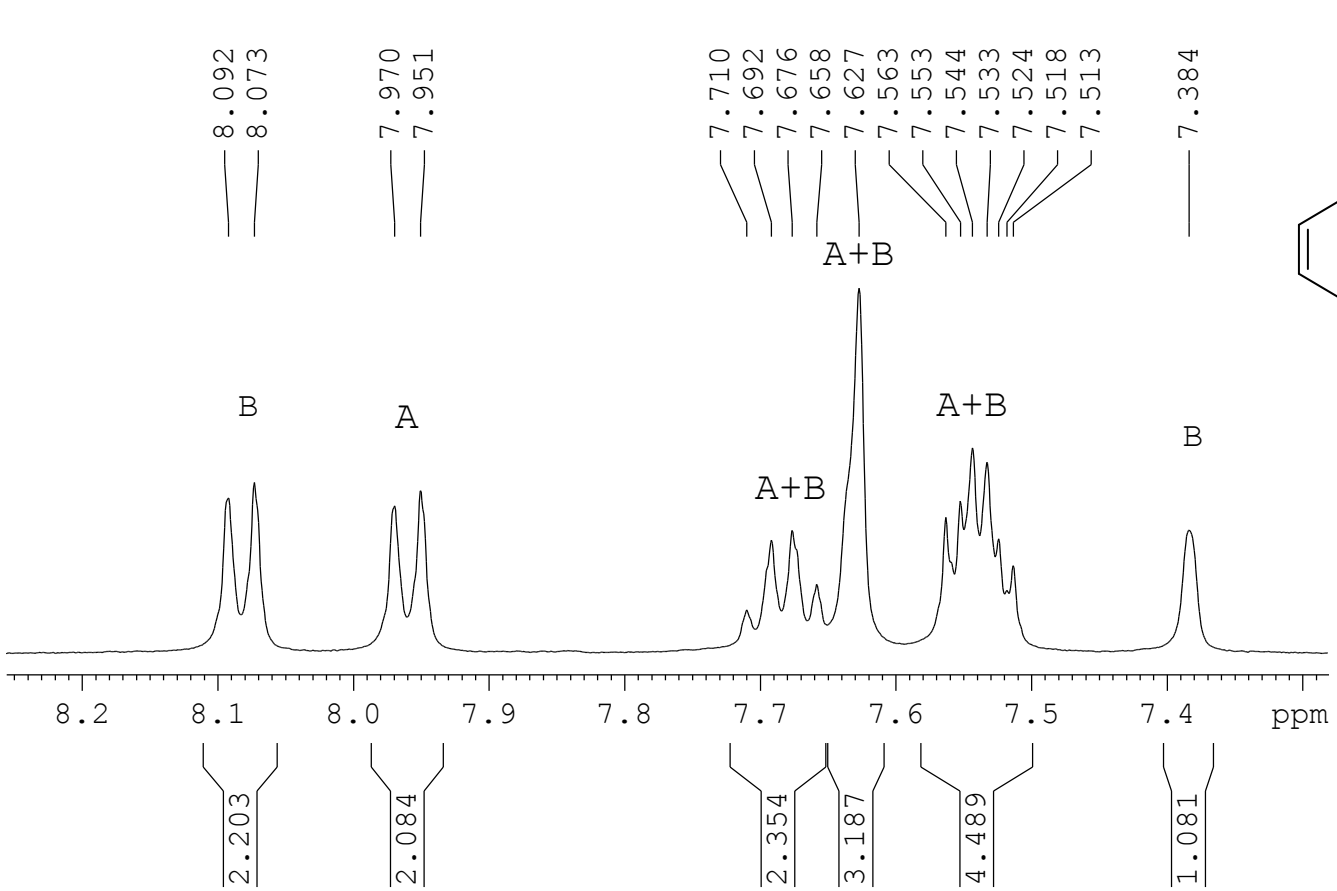


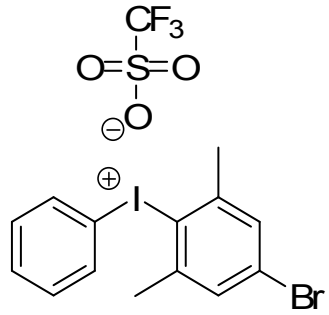
400 MHz
DMSO

3j

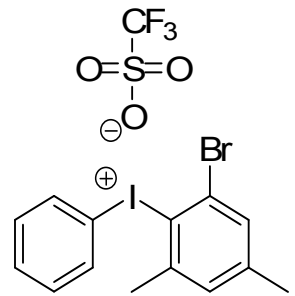








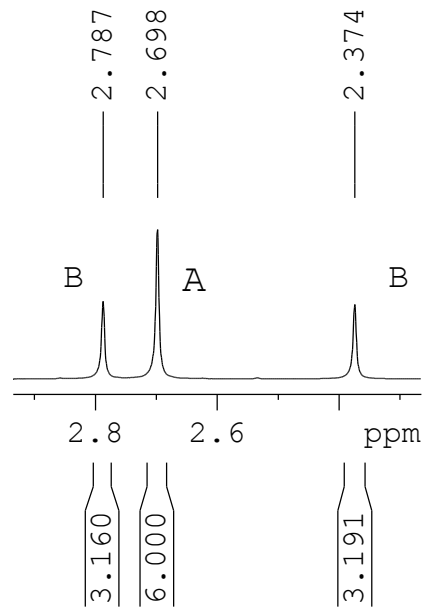
A 3k"

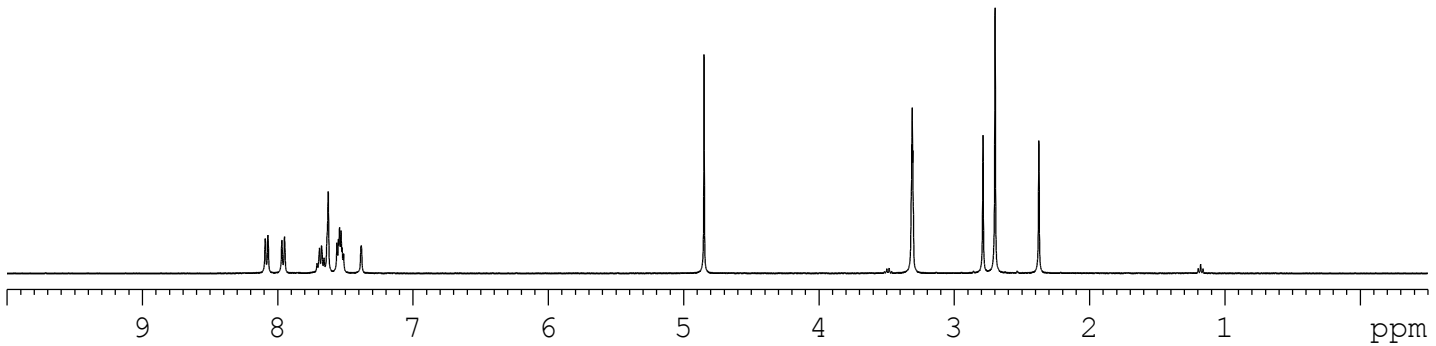


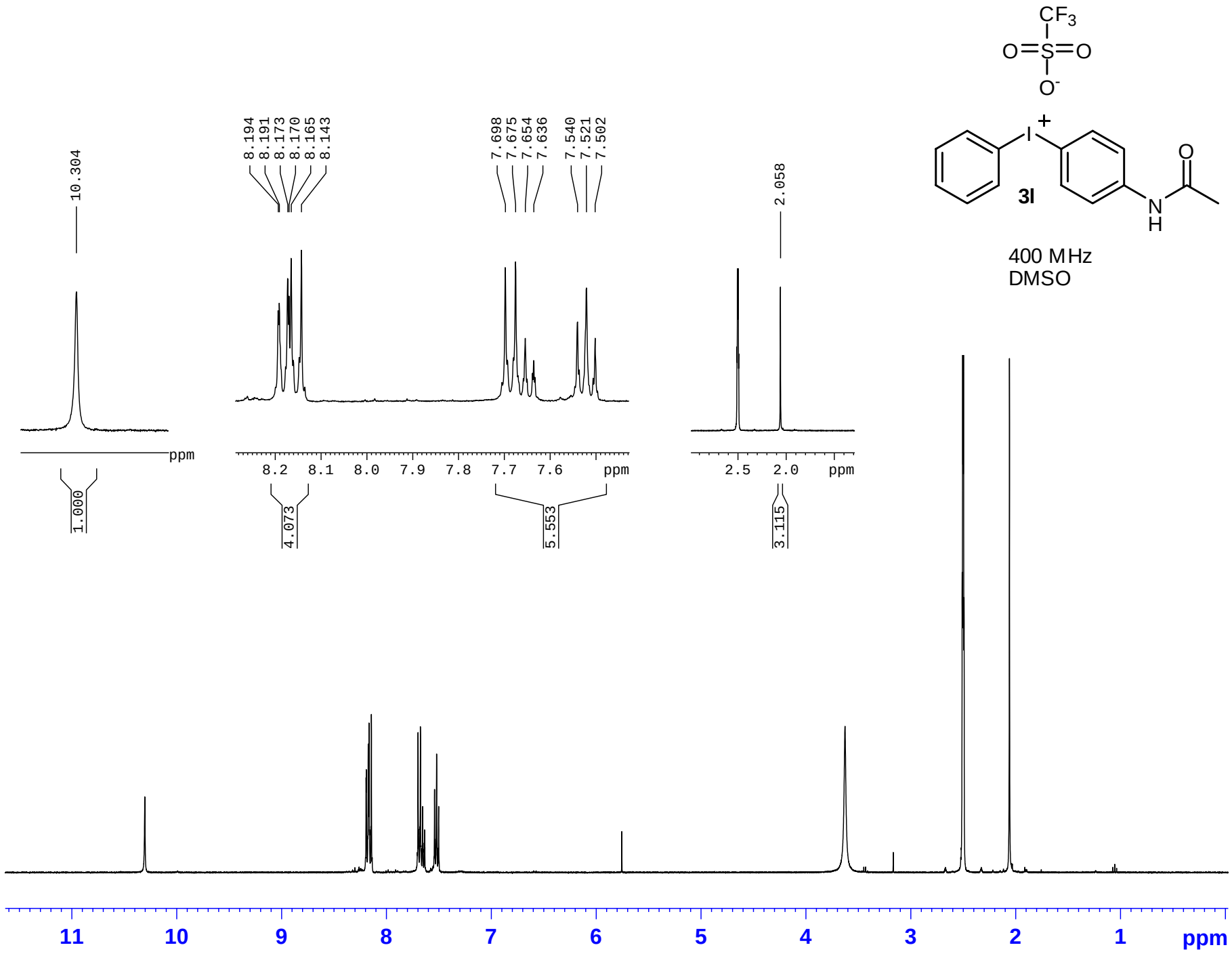
B 3k'

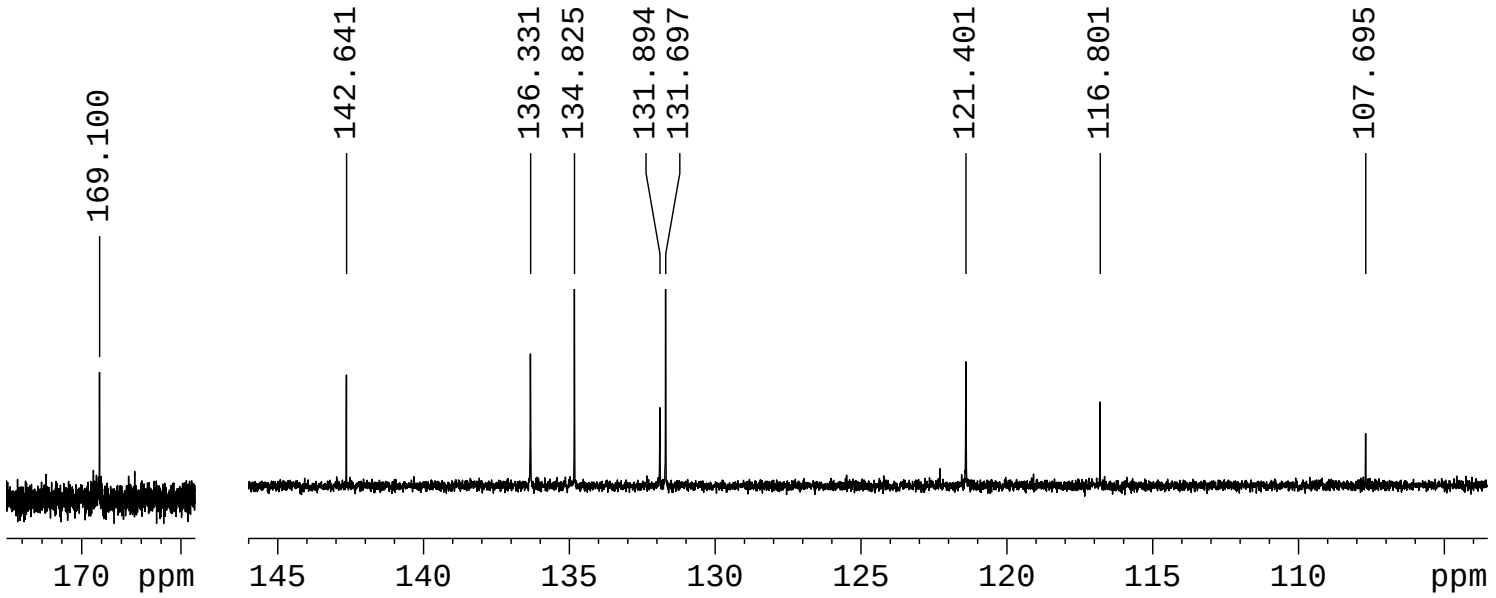
Regioisomeric Mixture
A : B ~ 1 : 1.2

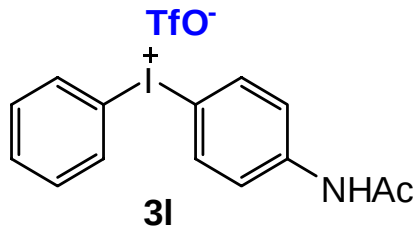
400 MHz
MeOD





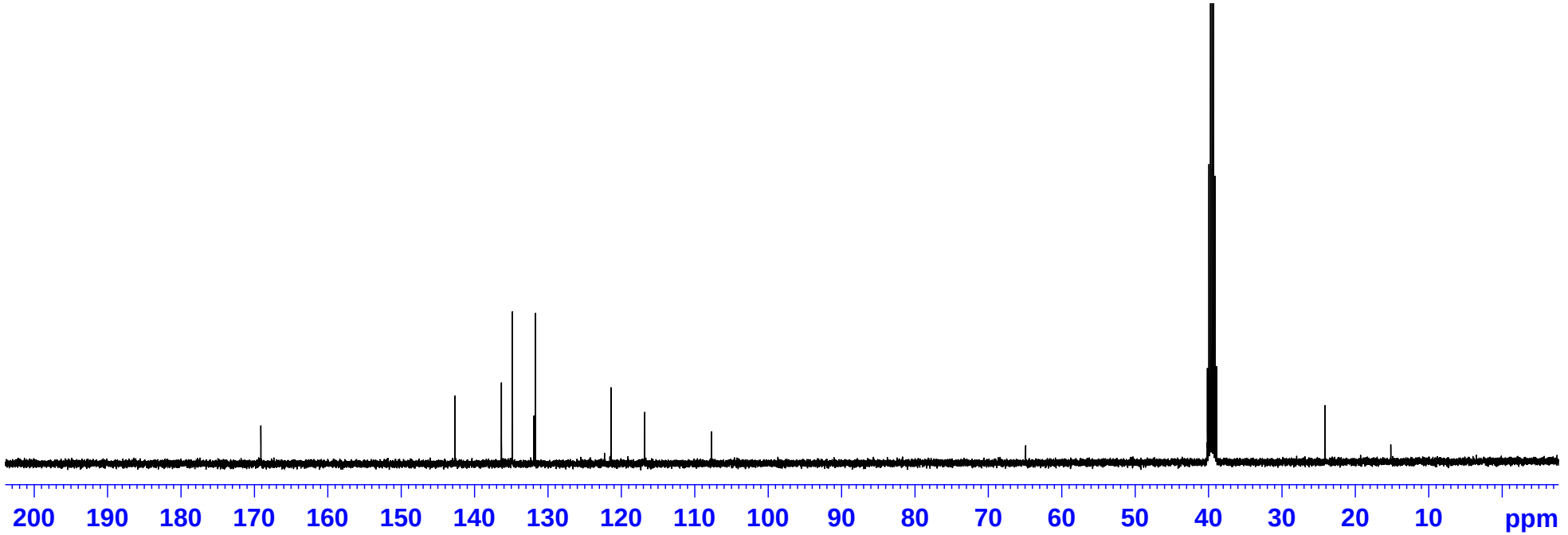

10.304
8.194
8.191
8.173
8.170
8.165
8.143
7.698
7.675
7.654
7.636
7.540
7.521
7.502
2.058
ppm
8.2
8.1
8.0
7.9
7.8
7.7
7.6
2.5
2.0
1.000
4.073
5.553
3.115
CF3
O=S=O
O-
I+
O
N
H
3l
400 MHz
DMSO
11
10
9
8
7
6
5
4
3
2
1
ppm

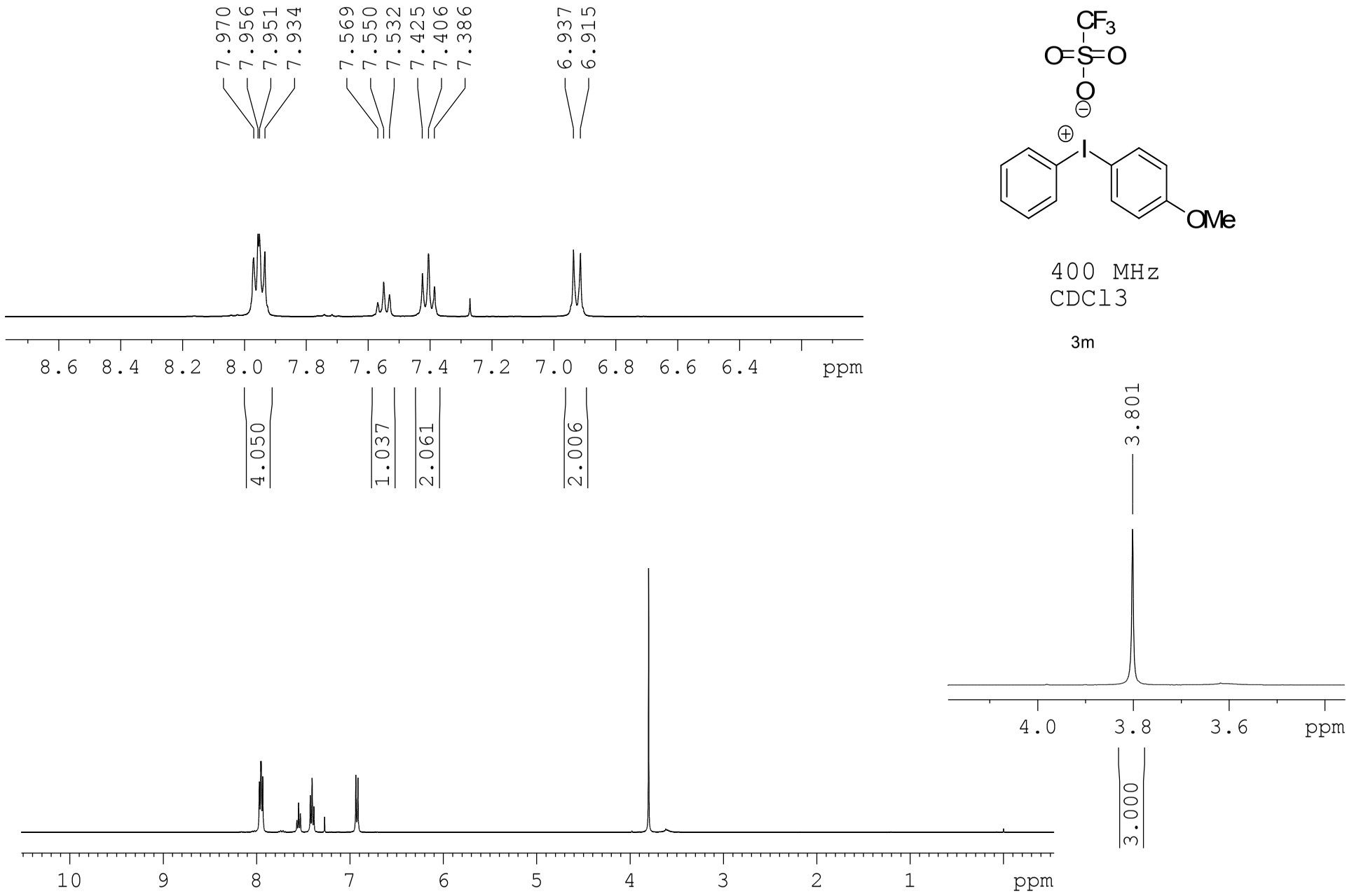


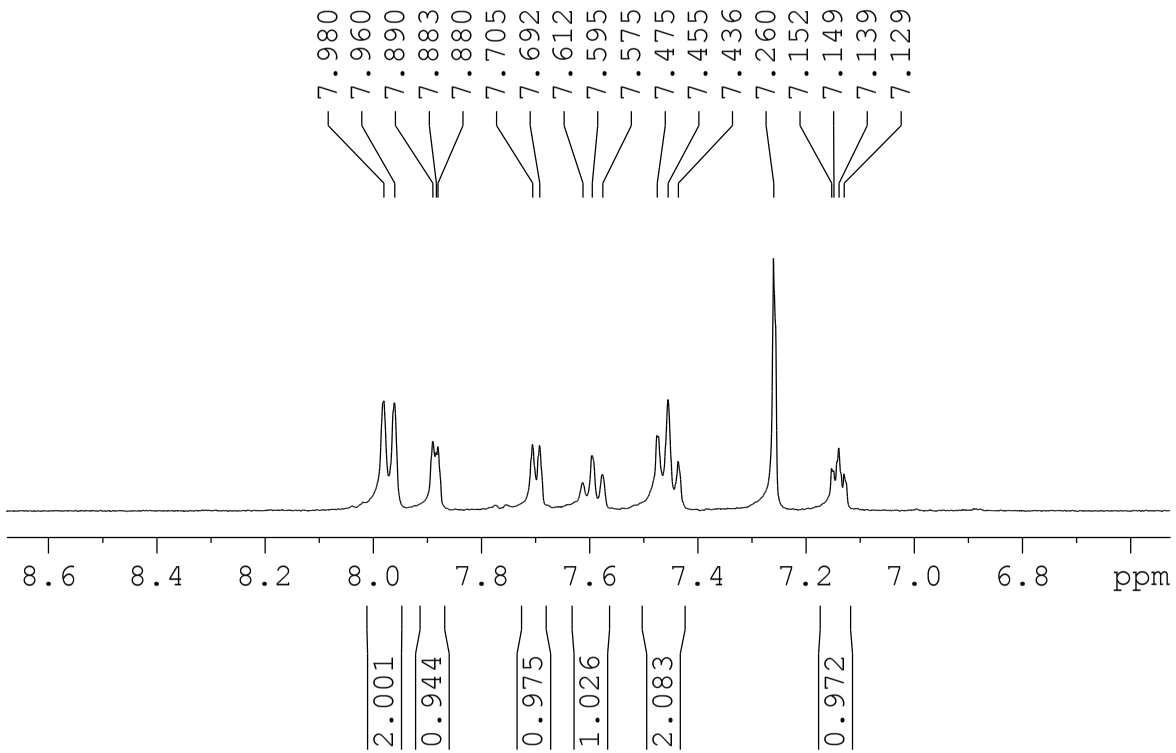


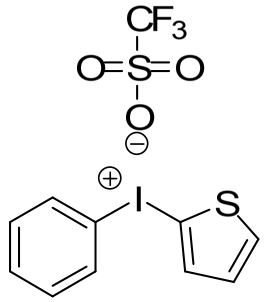
3l

100 MHz
DMSO-d_6



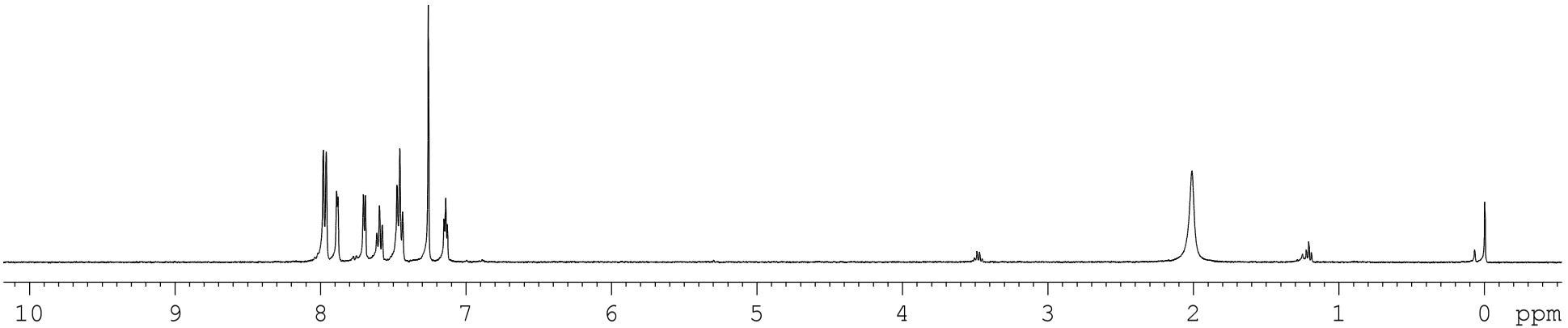

7.970
7.956
7.951
7.934
7.569
7.550
7.532
7.425
7.406
7.386
6.937
6.915
8.6 8.4 8.2 8.0 7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4 ppm
4.050
1.037
2.061
2.006
CF3
O=S=O
O
I
OMe
400 MHz
CDCl3
3m
3.801
4.0 3.8 3.6 ppm
3.000
10 9 8 7 6 5 4 3 2 1 ppm

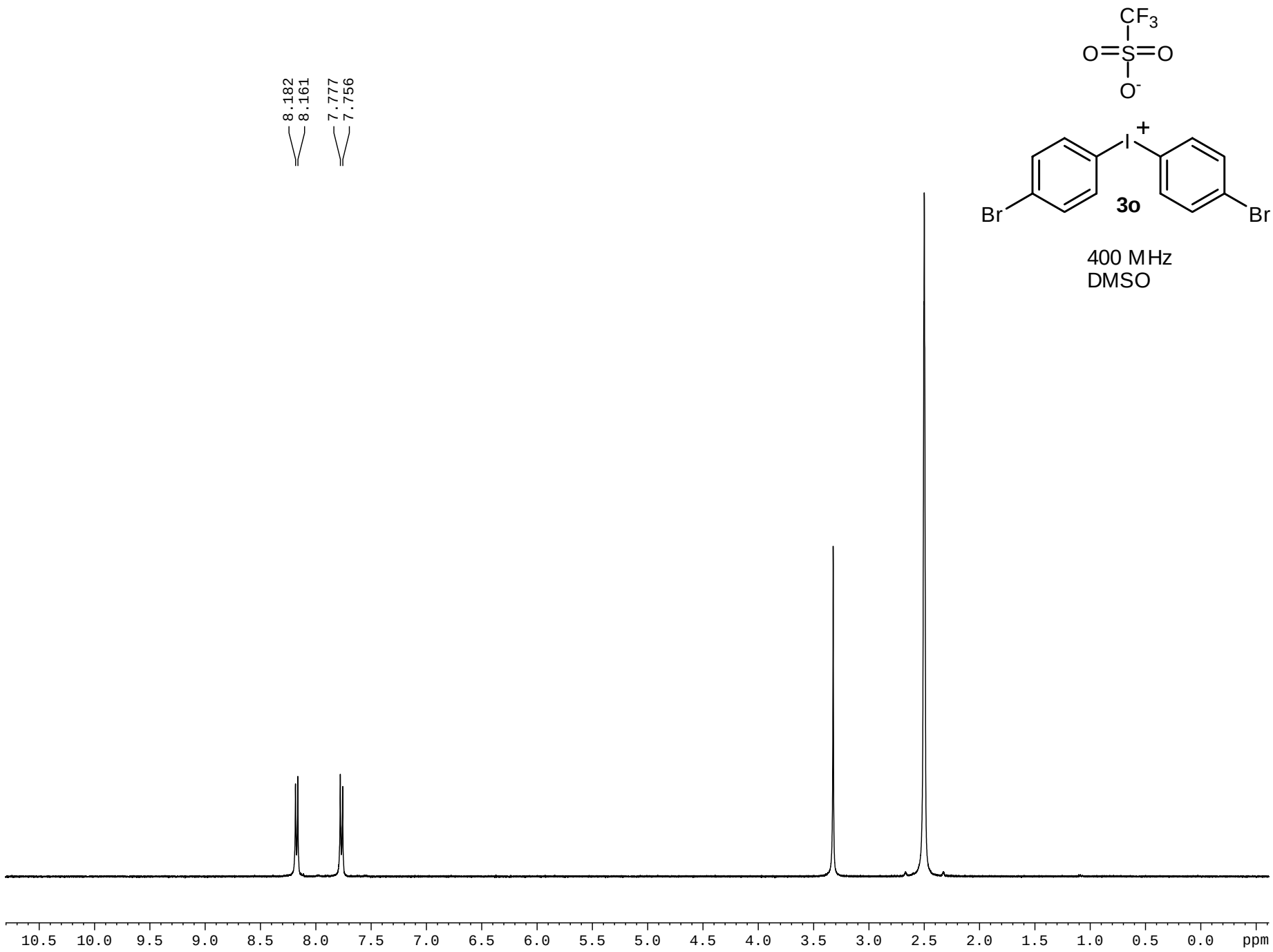


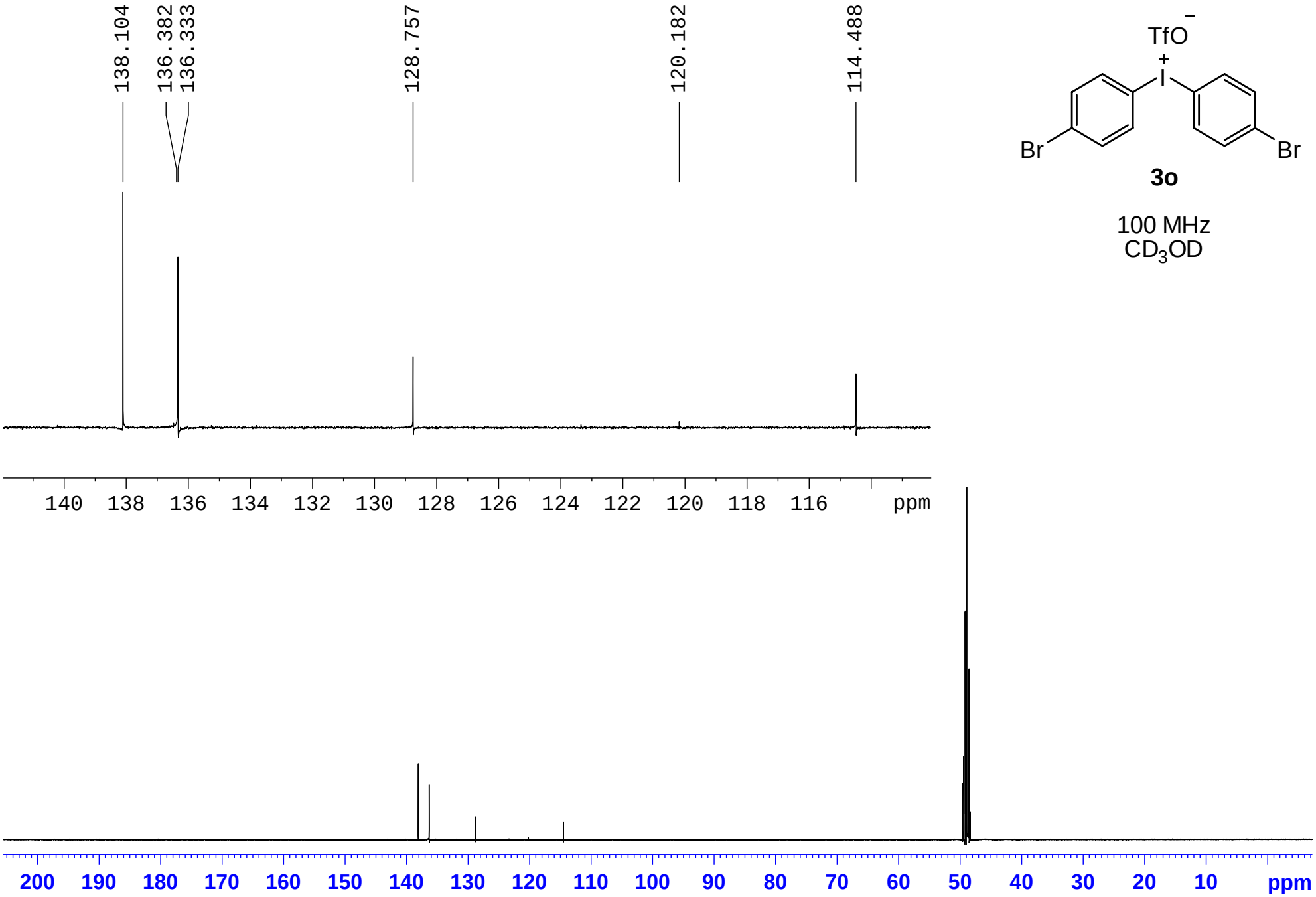


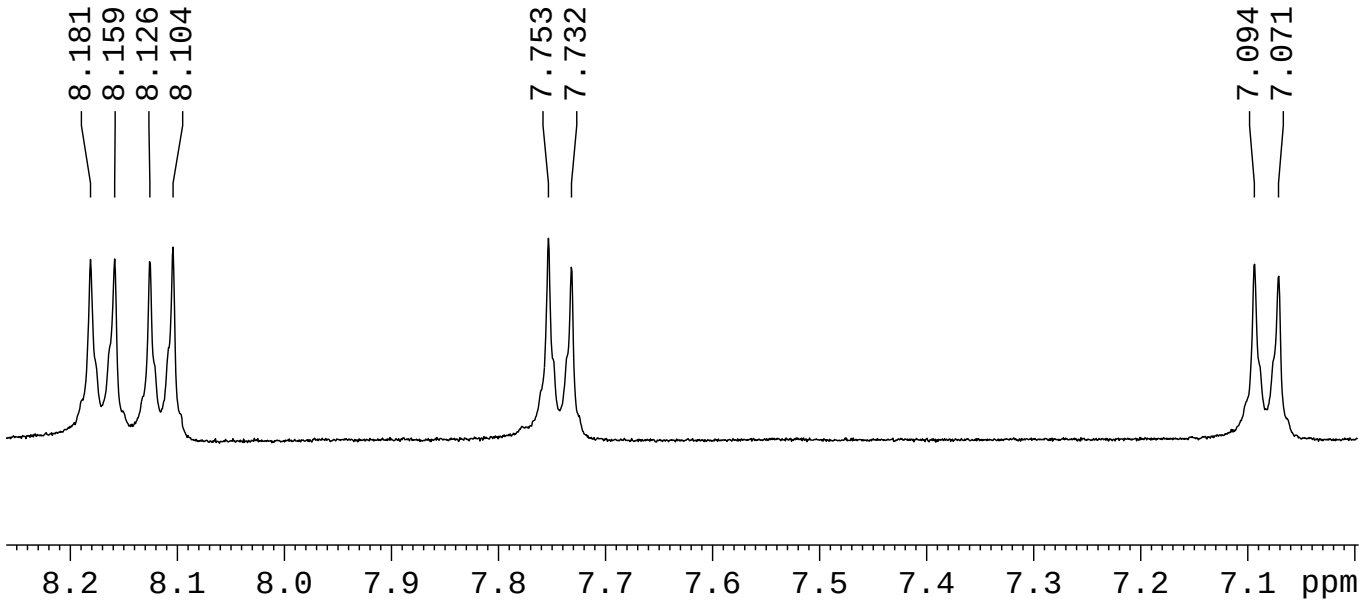
400 MHz
CDCl3

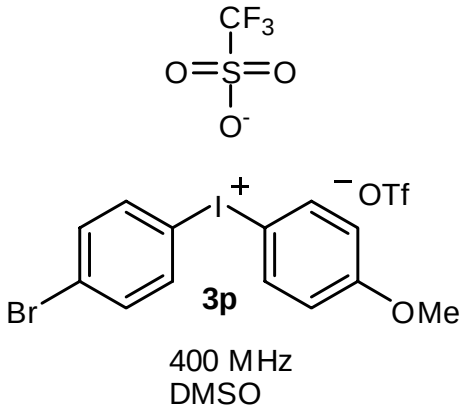
3n



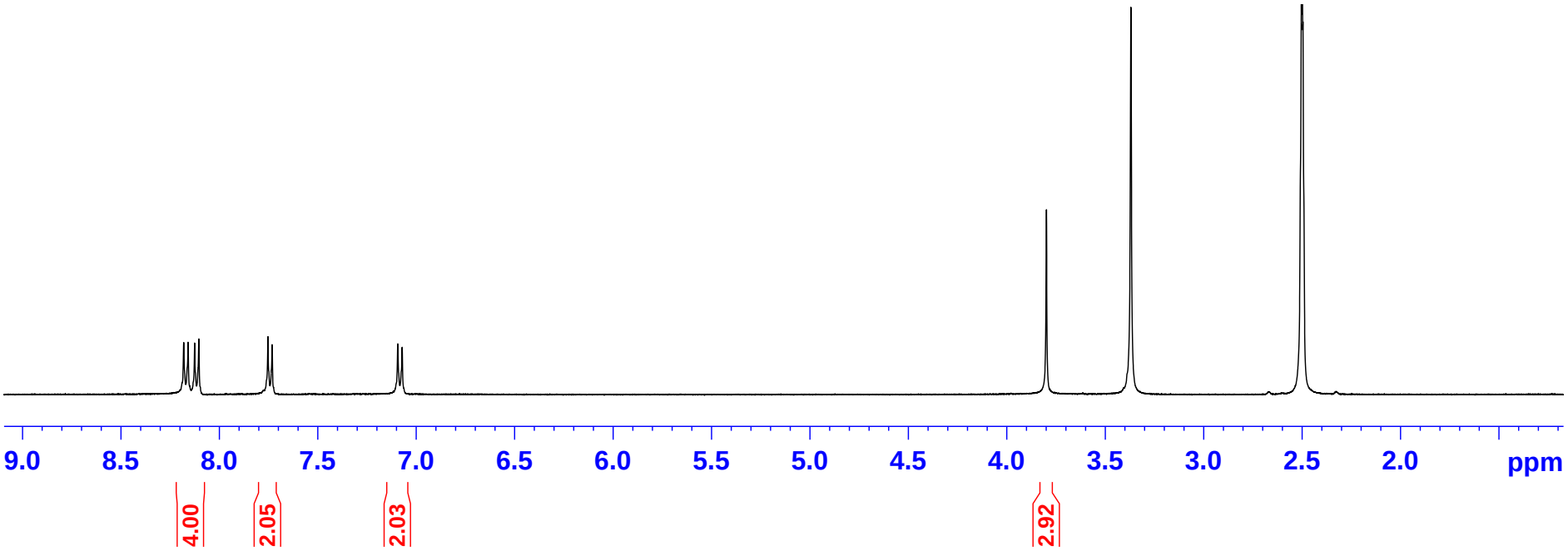

8.182
8.161
7.777
7.756
CF3
O=S=O
O-
I+
Br
Br
3o
400 MHz
DMSO
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm

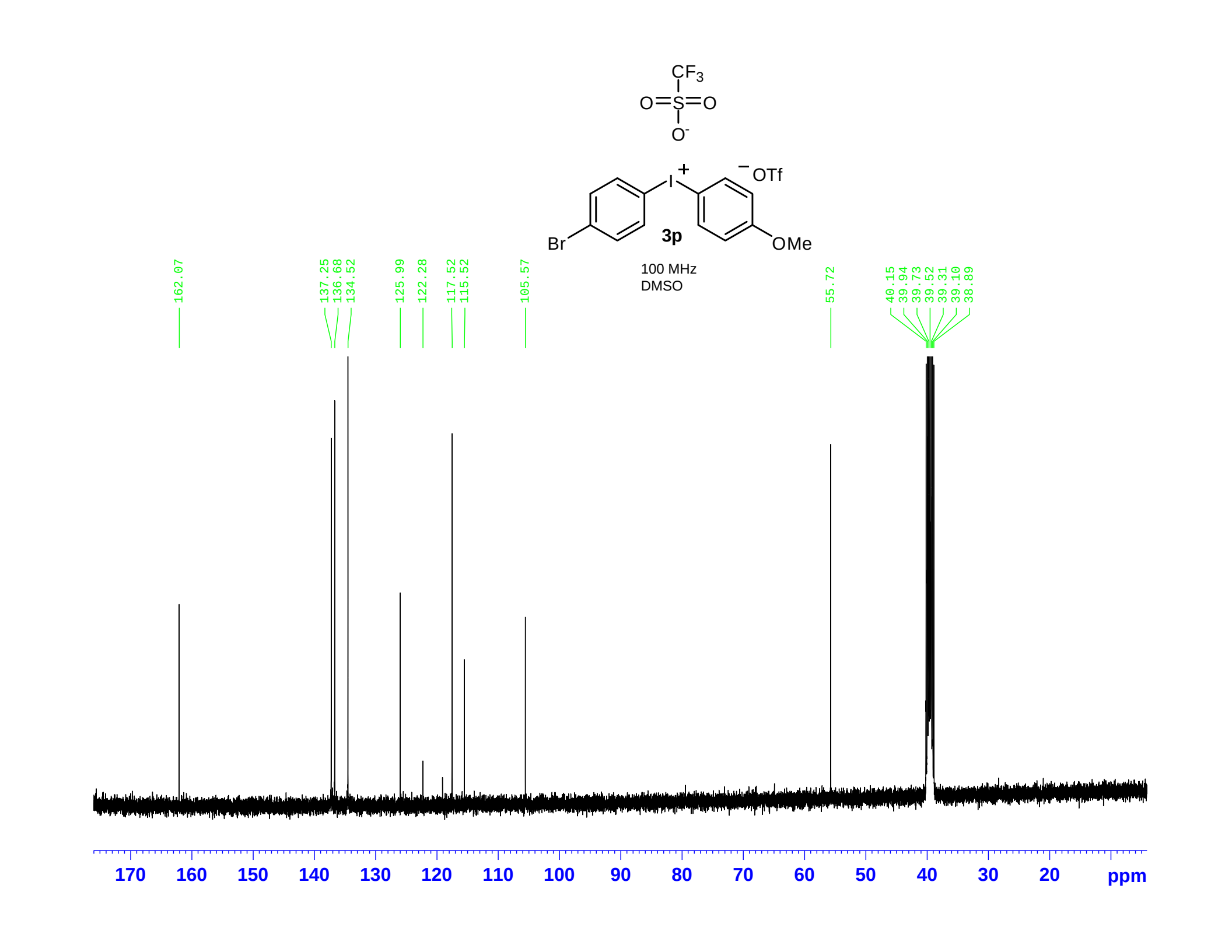

138.104
136.382
136.333
128.757
120.182
114.488
TfO
Br
Br
3o
100 MHz
CD3OD
140 138 136 134 132 130 128 126 124 122 120 118 116 ppm
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

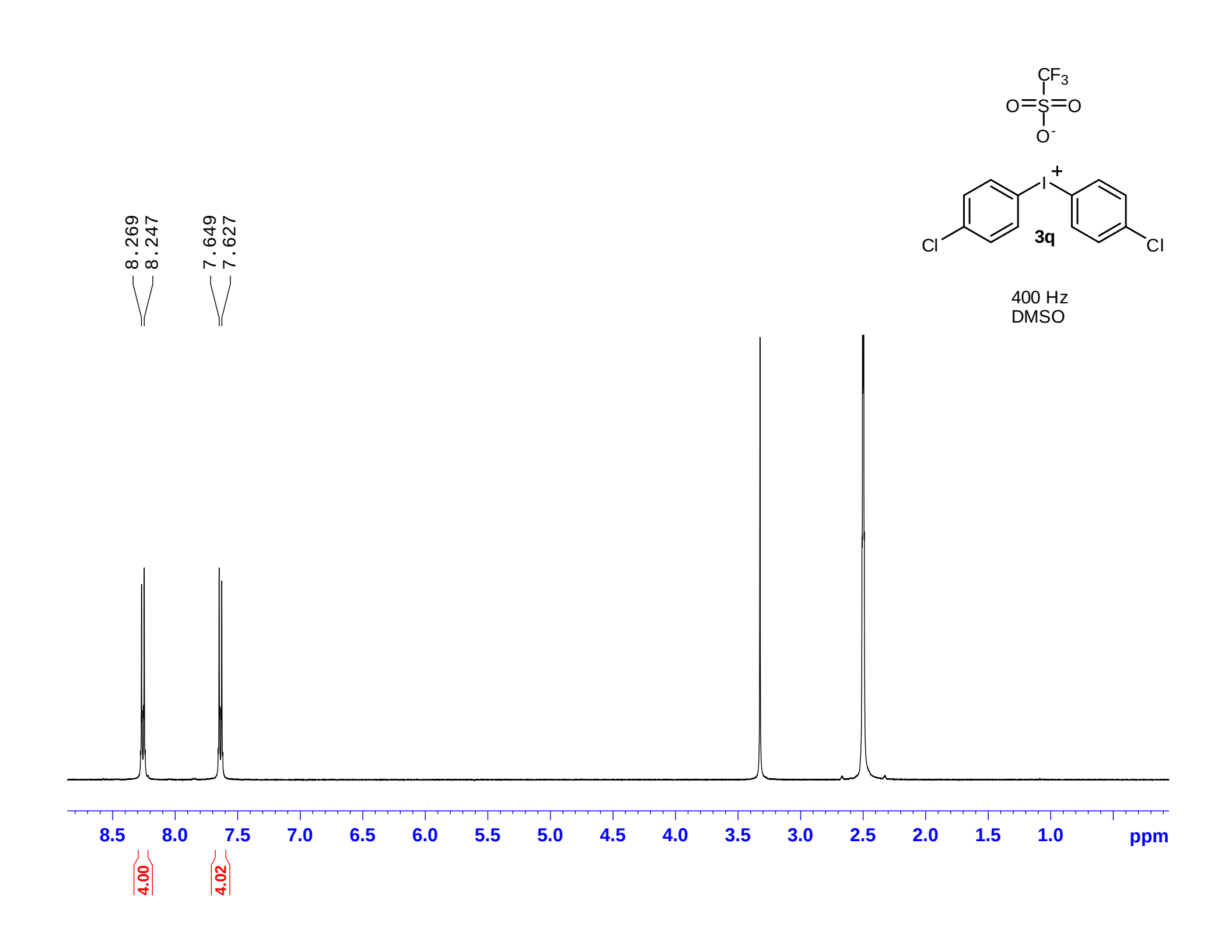

8.181
8.159
8.126
8.104
7.753
7.732
7.094
7.071
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
ppm

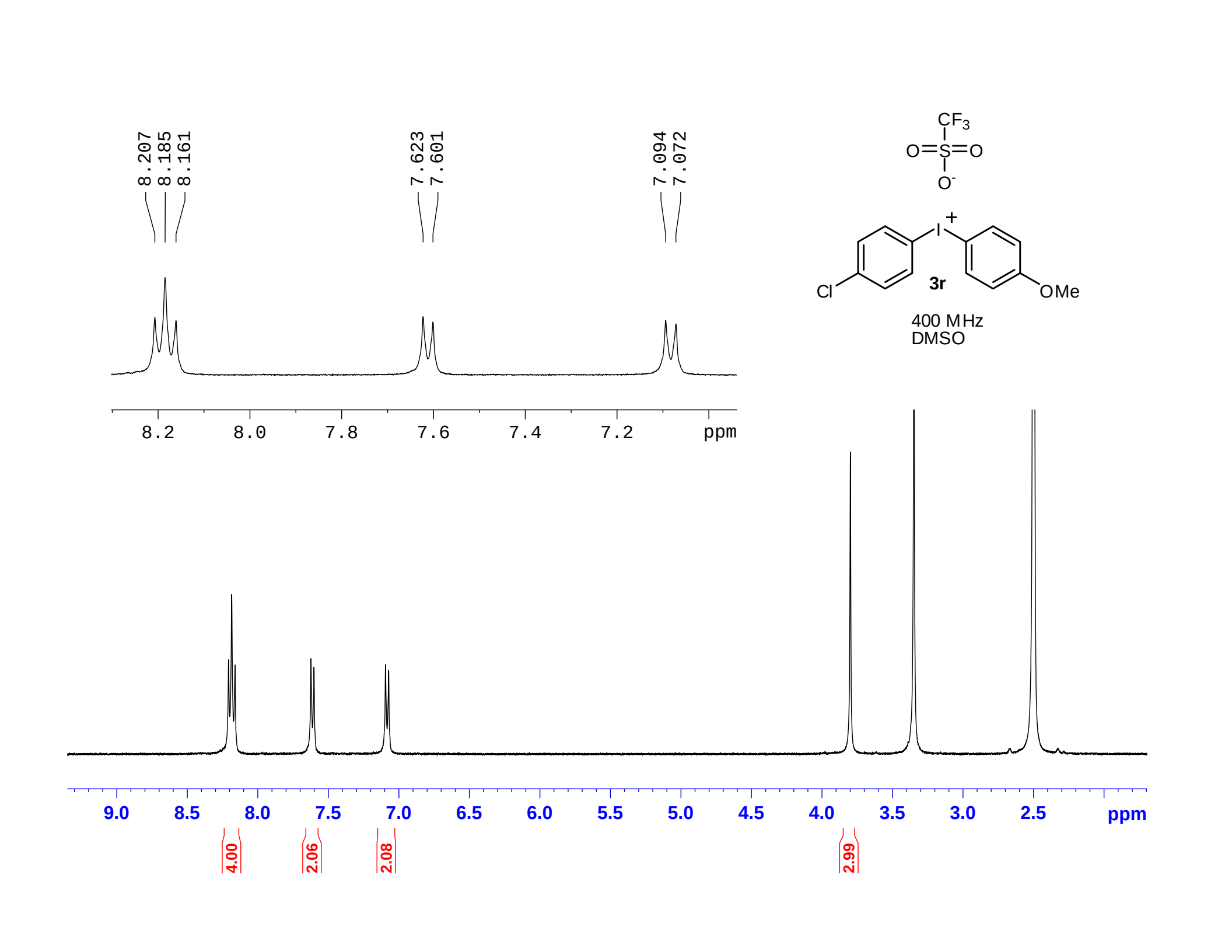

CF3
O=S=O
O-
I+
OTf
Br
OMe
3p

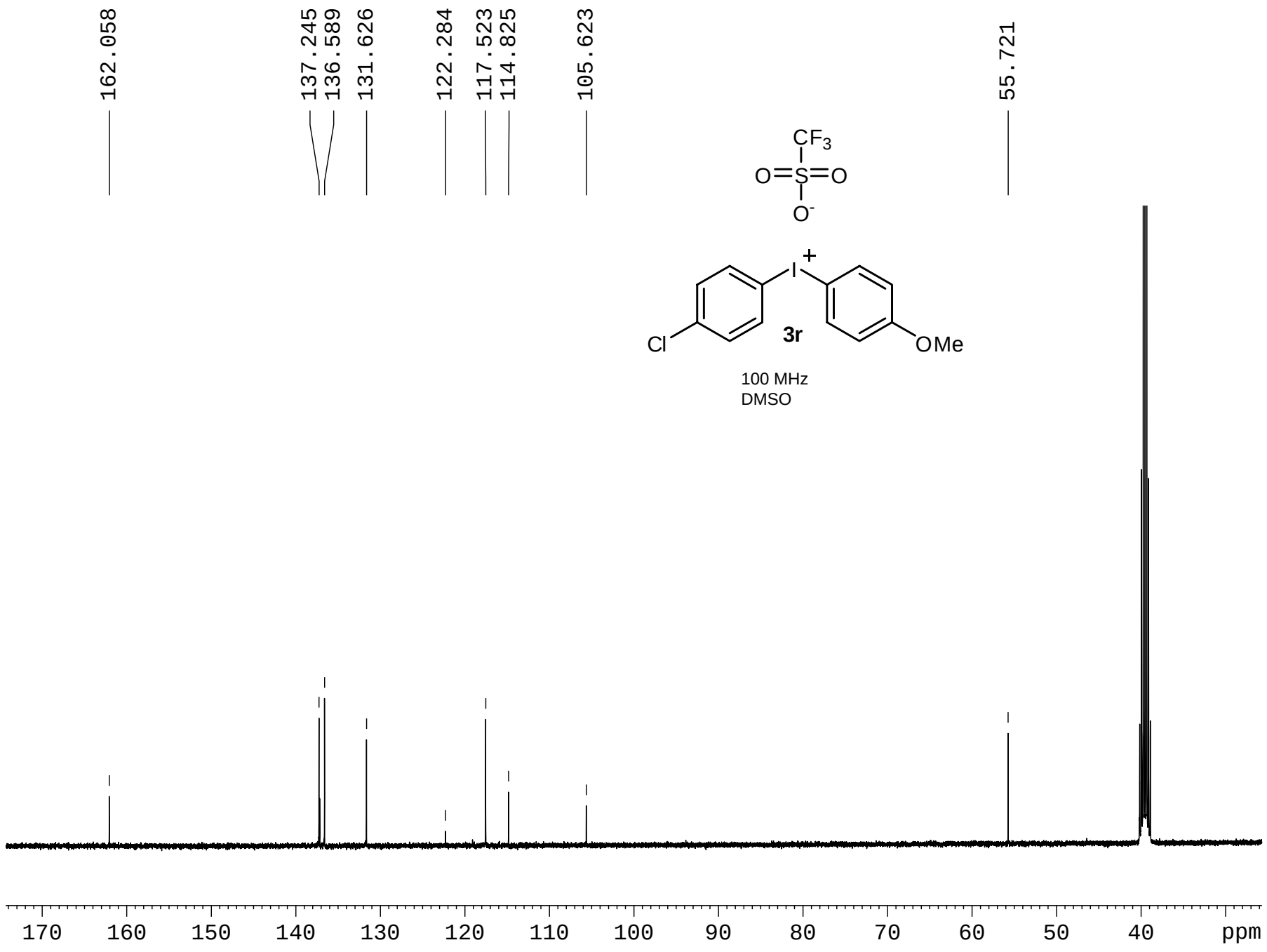
400 MHz
DMSO

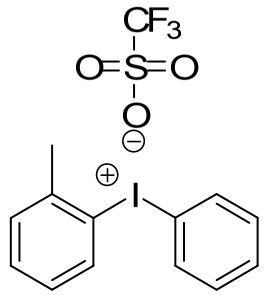

9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm
4.00
2.05
2.03
2.92


CF3
O=S=O
O-
I+
−OTf
Br
OMe
3p
100 MHz
DMSO
162.07
137.25
136.68
134.52
125.99
122.28
117.52
115.52
105.57
55.72
40.15
39.94
39.73
39.52
39.31
39.10
38.89
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


8.269
8.247
7.649
7.627
CF3
O=S=O
O-
I+
Cl
Cl
3q
400 Hz
DMSO
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
4.00
4.02

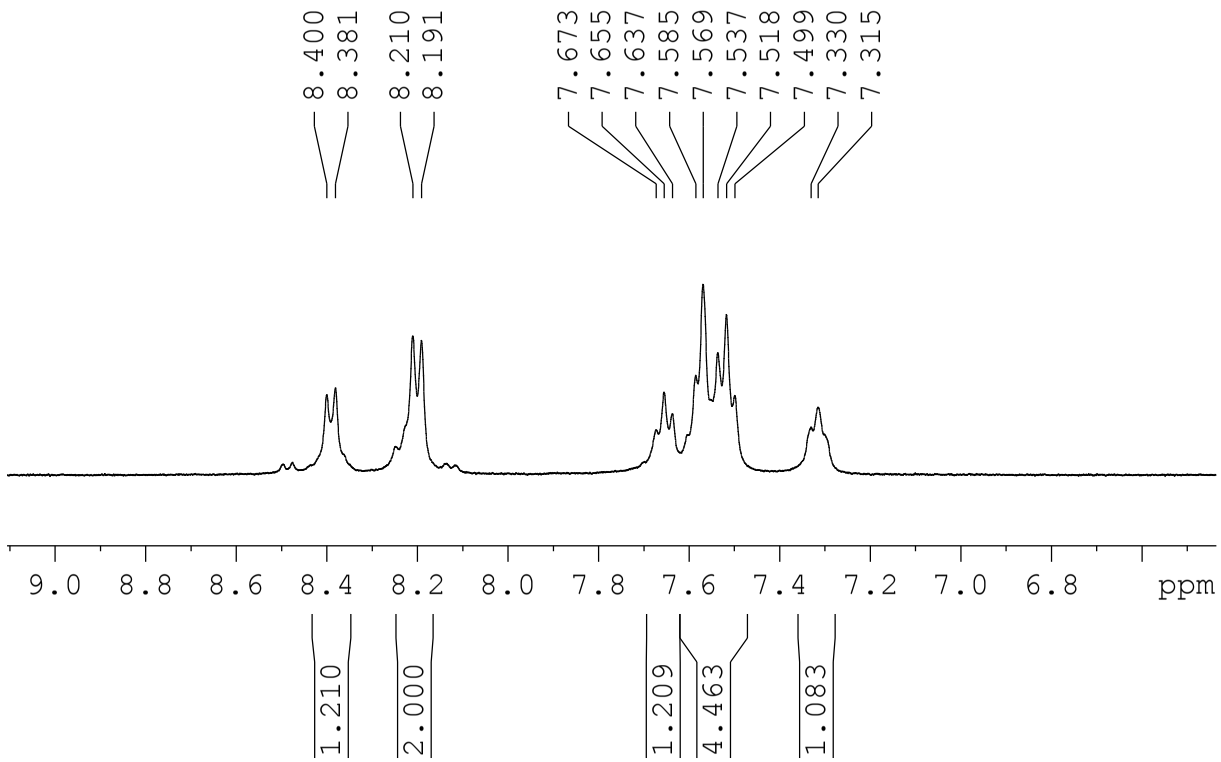

8.207
8.185
8.161
7.623
7.601
7.094
7.072
8.2
8.0
7.8
7.6
7.4
7.2
ppm
CF3
O=S=O
O-
I+
Cl
OMe
3r
400 MHz
DMSO
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
4.00
2.06
2.08
2.99

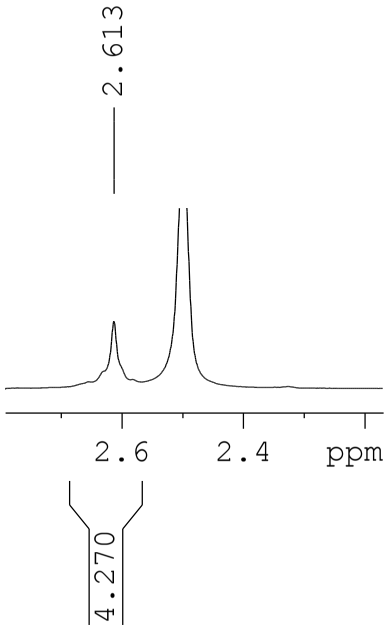

162.058
137.245
136.589
131.626
122.284
117.523
114.825
105.623
55.721
CF3
O
S
O
O-
I+
Cl
OMe
3r
100 MHz
DMSO
170
160
150
140
130
120
110
100
90
80
70
60
50
40
ppm

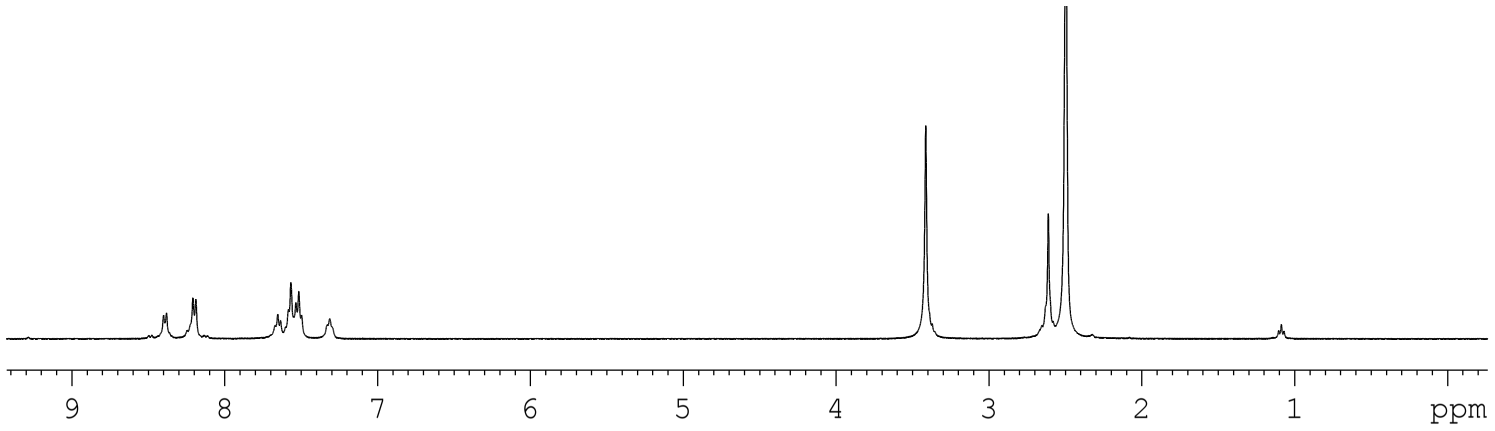


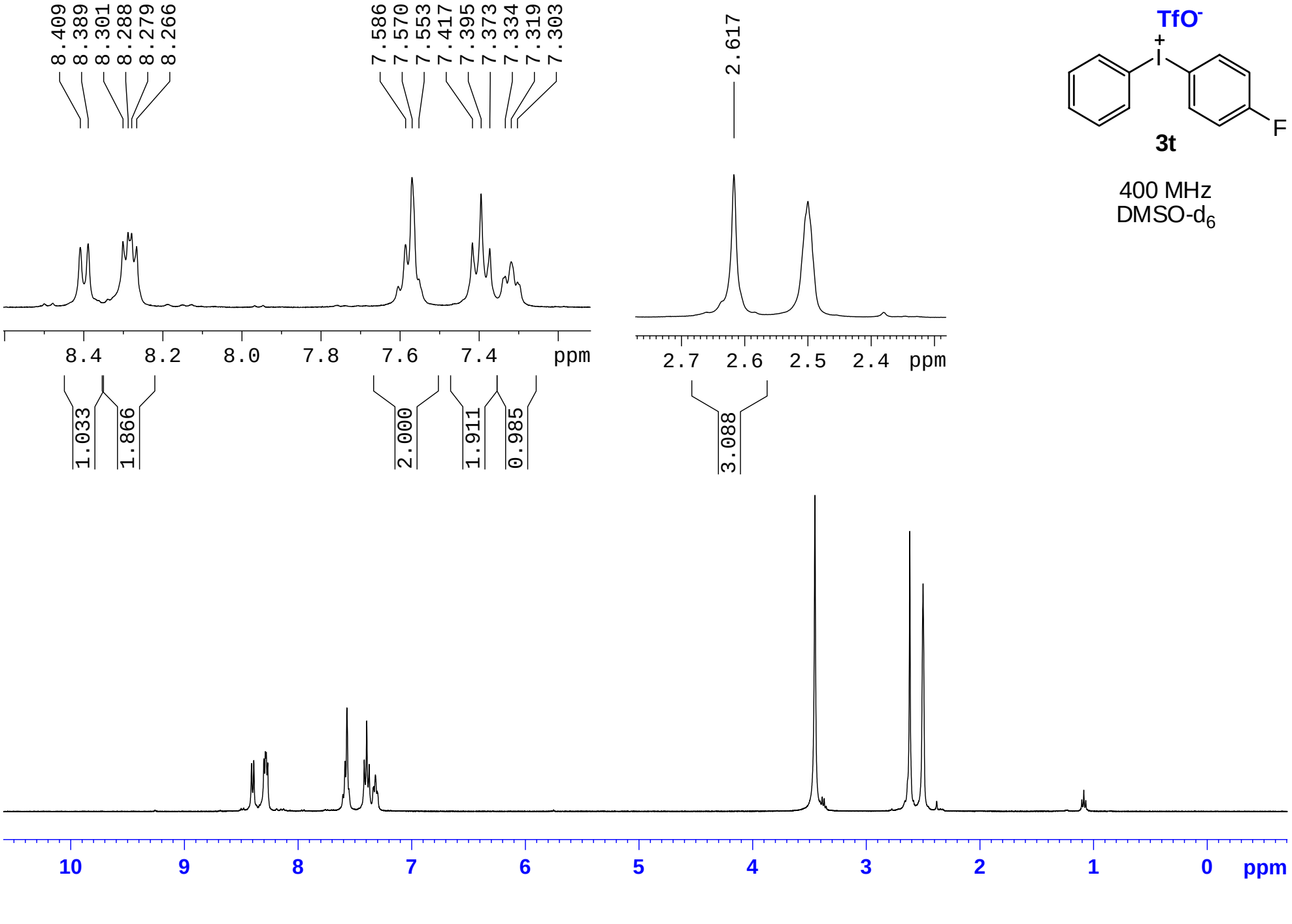
400 MHz
DMSO

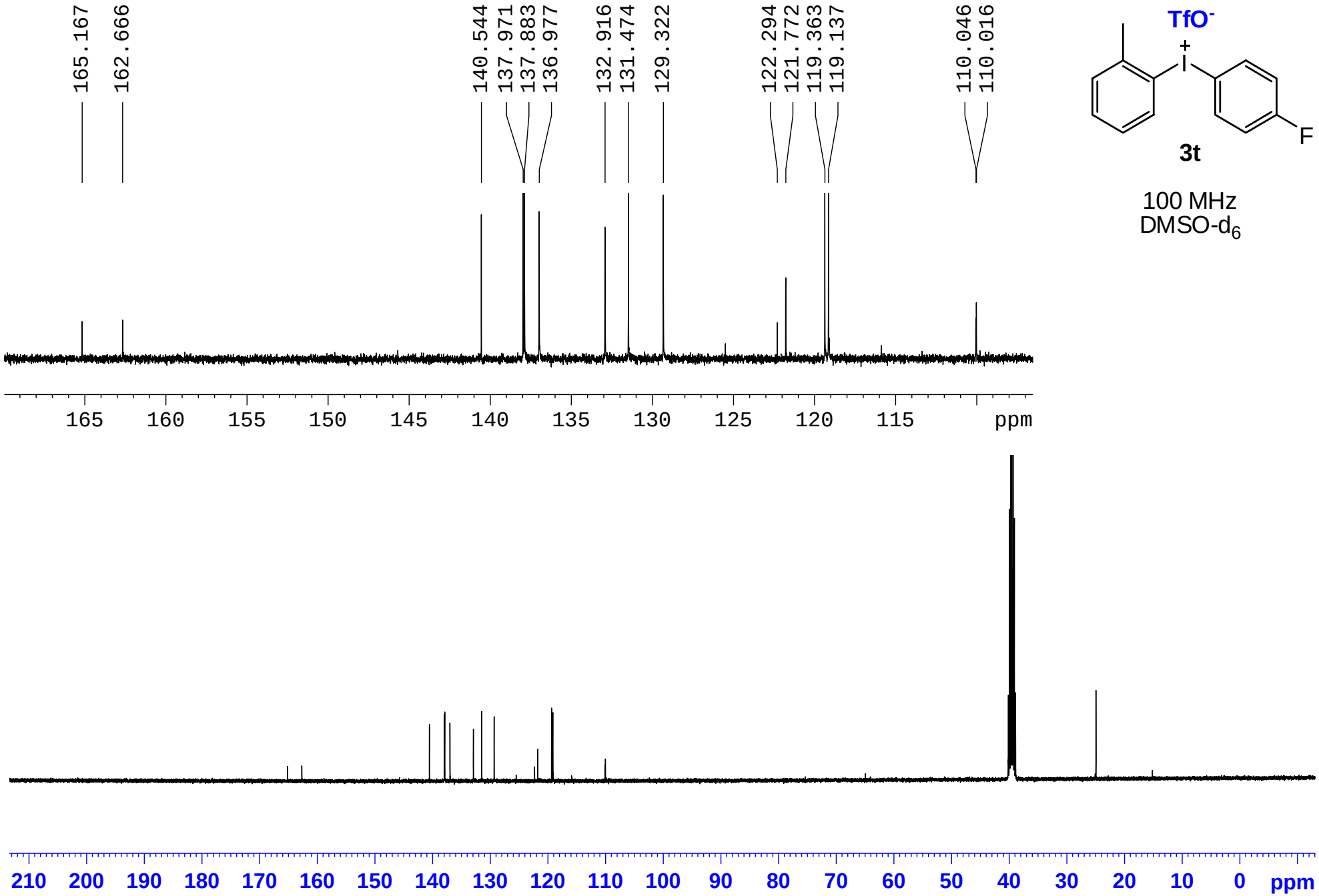
3s

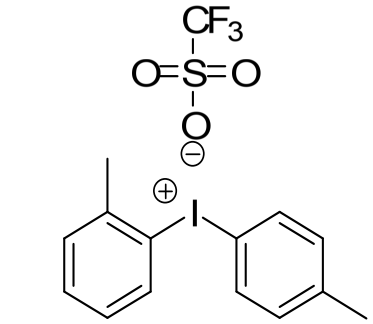






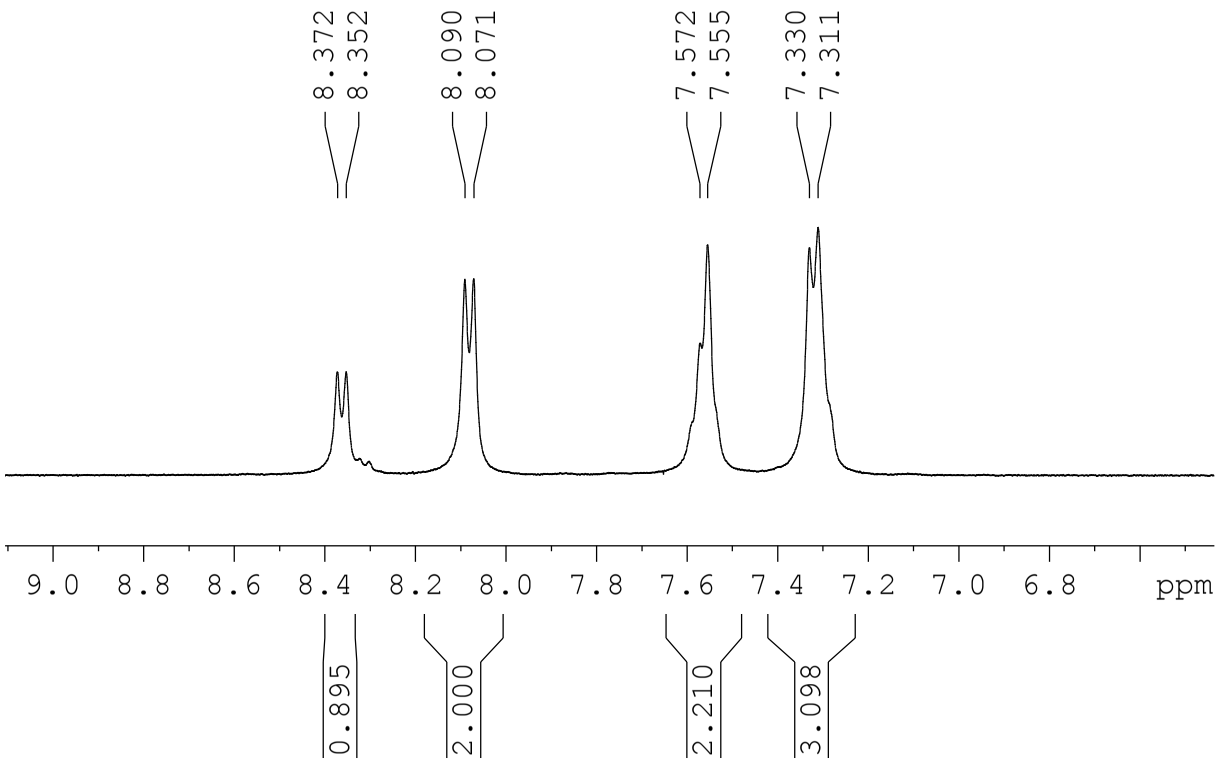

8.409
8.389
8.301
8.288
8.279
8.266
7.586
7.570
7.553
7.417
7.395
7.373
7.334
7.319
7.303
2.617
8.4
8.2
8.0
7.8
7.6
7.4
ppm
2.7
2.6
2.5
2.4
ppm
1.033
1.866
2.000
1.911
0.985
3.088
TfO-
F
3t
400 MHz
DMSO-d6
10
9
8
7
6
5
4
3
2
1
0
ppm

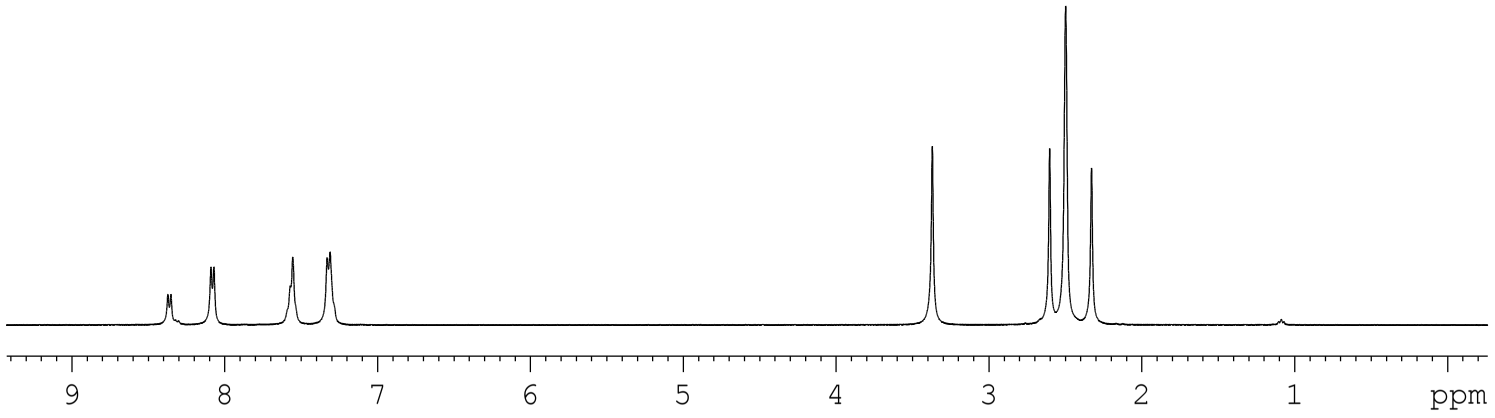

165.167
162.666
140.544
137.971
137.883
136.977
132.916
131.474
129.322
122.294
121.772
119.363
119.137
110.046
110.016
TfO-
F
3t
100 MHz
DMSO-d6
165 160 155 150 145 140 135 130 125 120 115 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

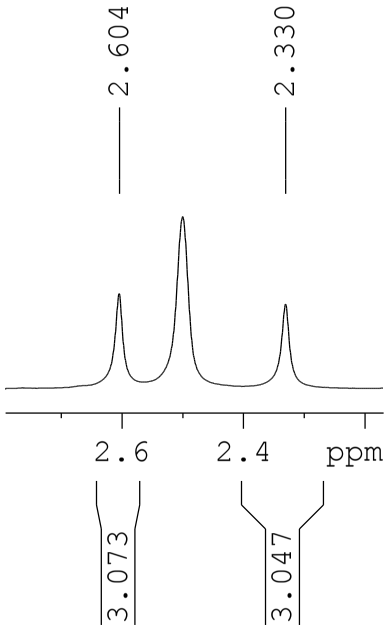


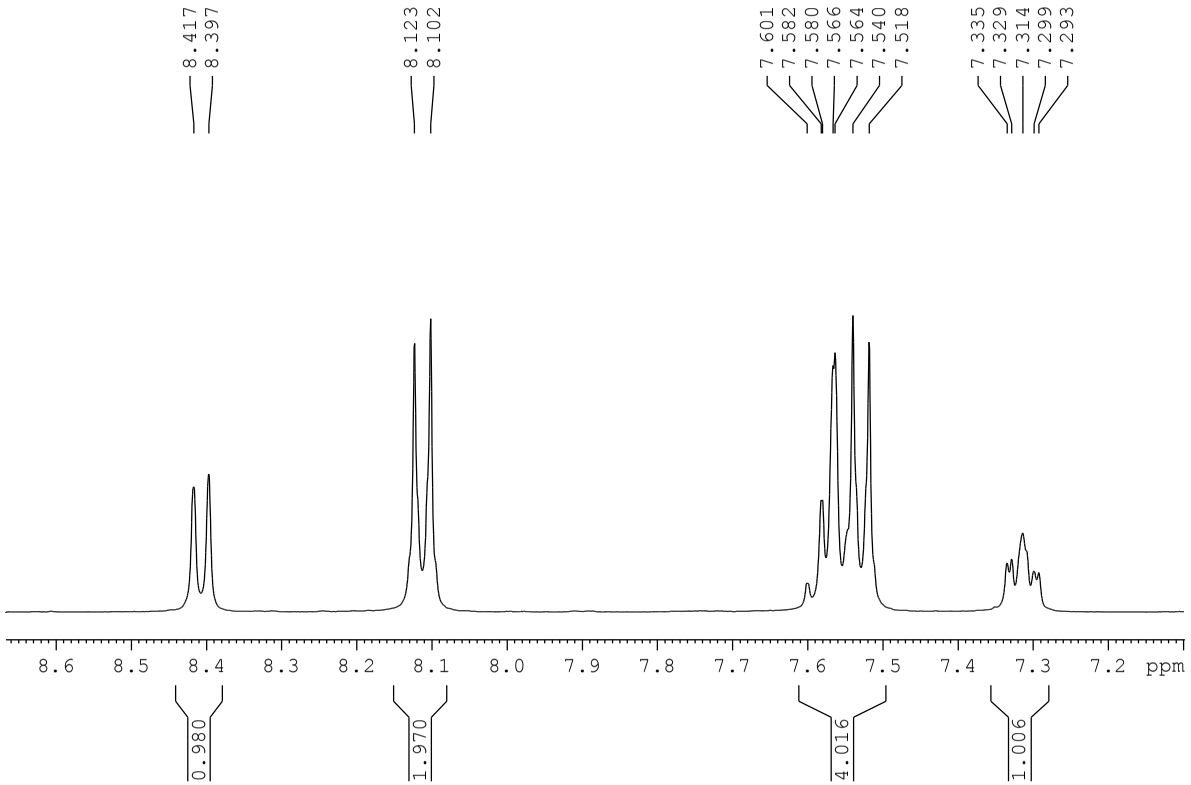
400 MHz
DMSO

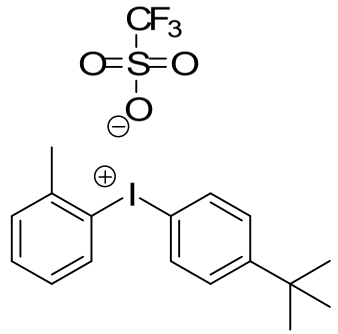
3u





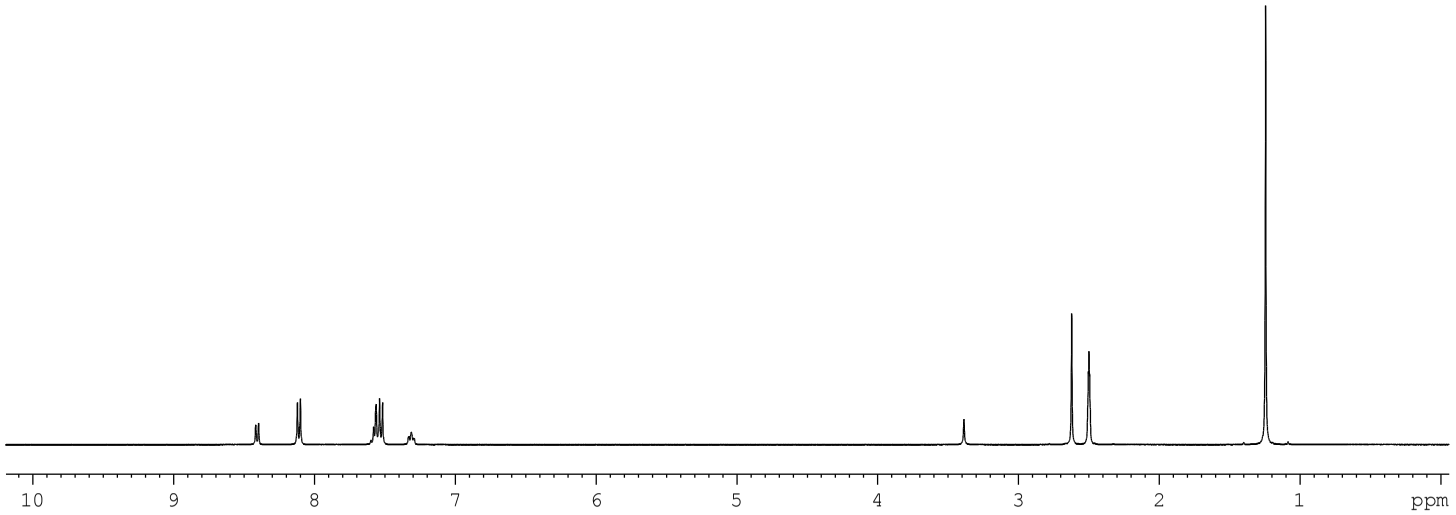


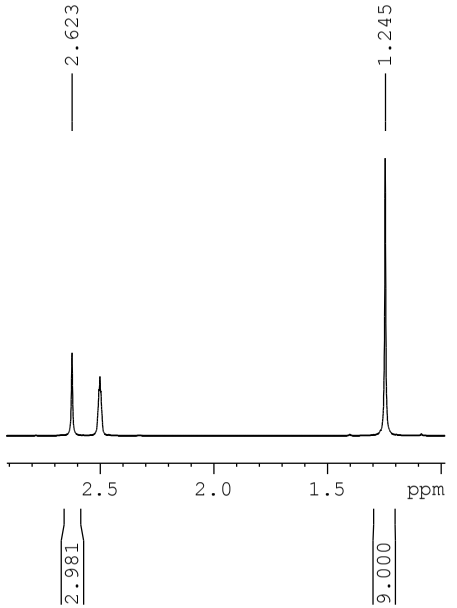


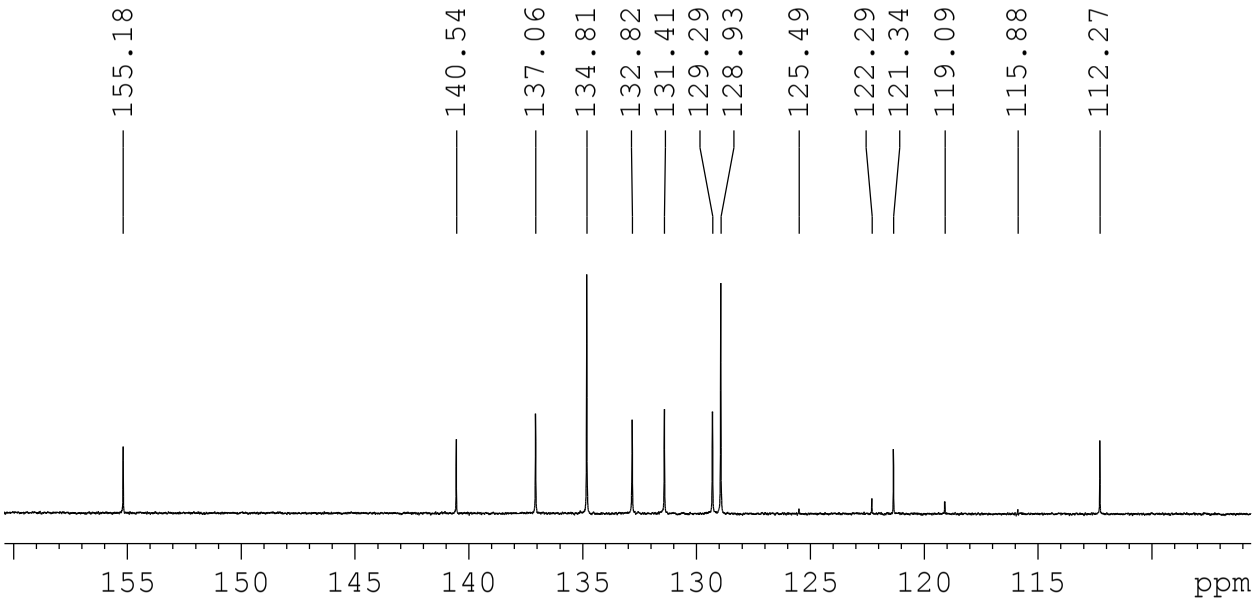


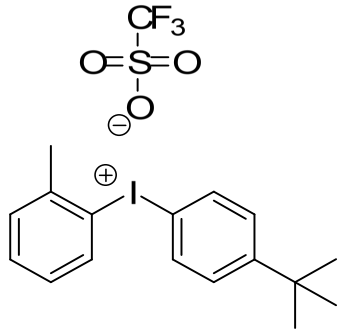
400 MHz
DMSO

3v



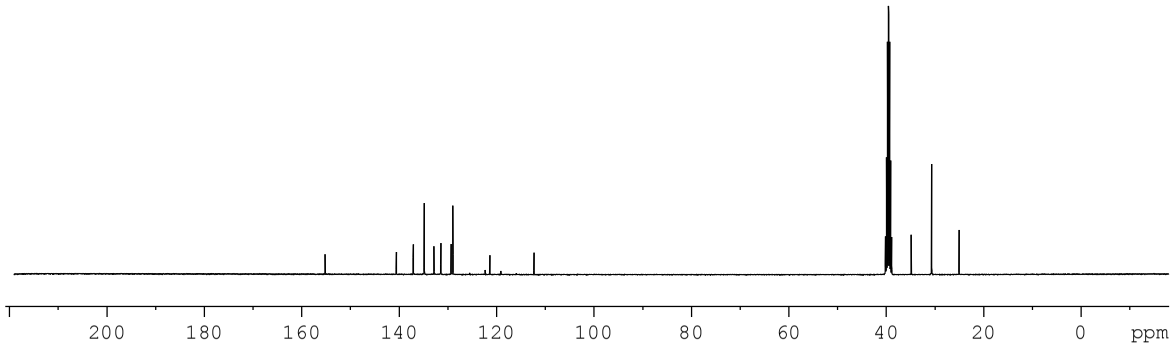


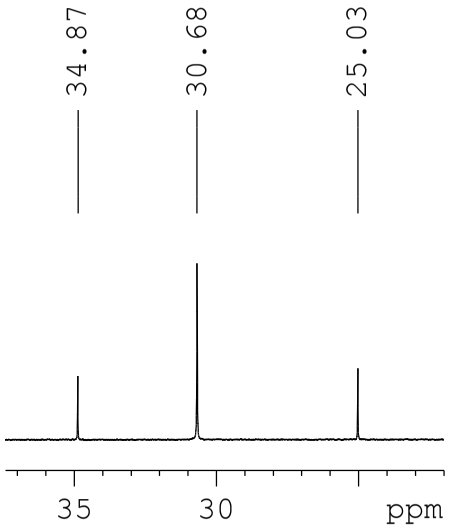


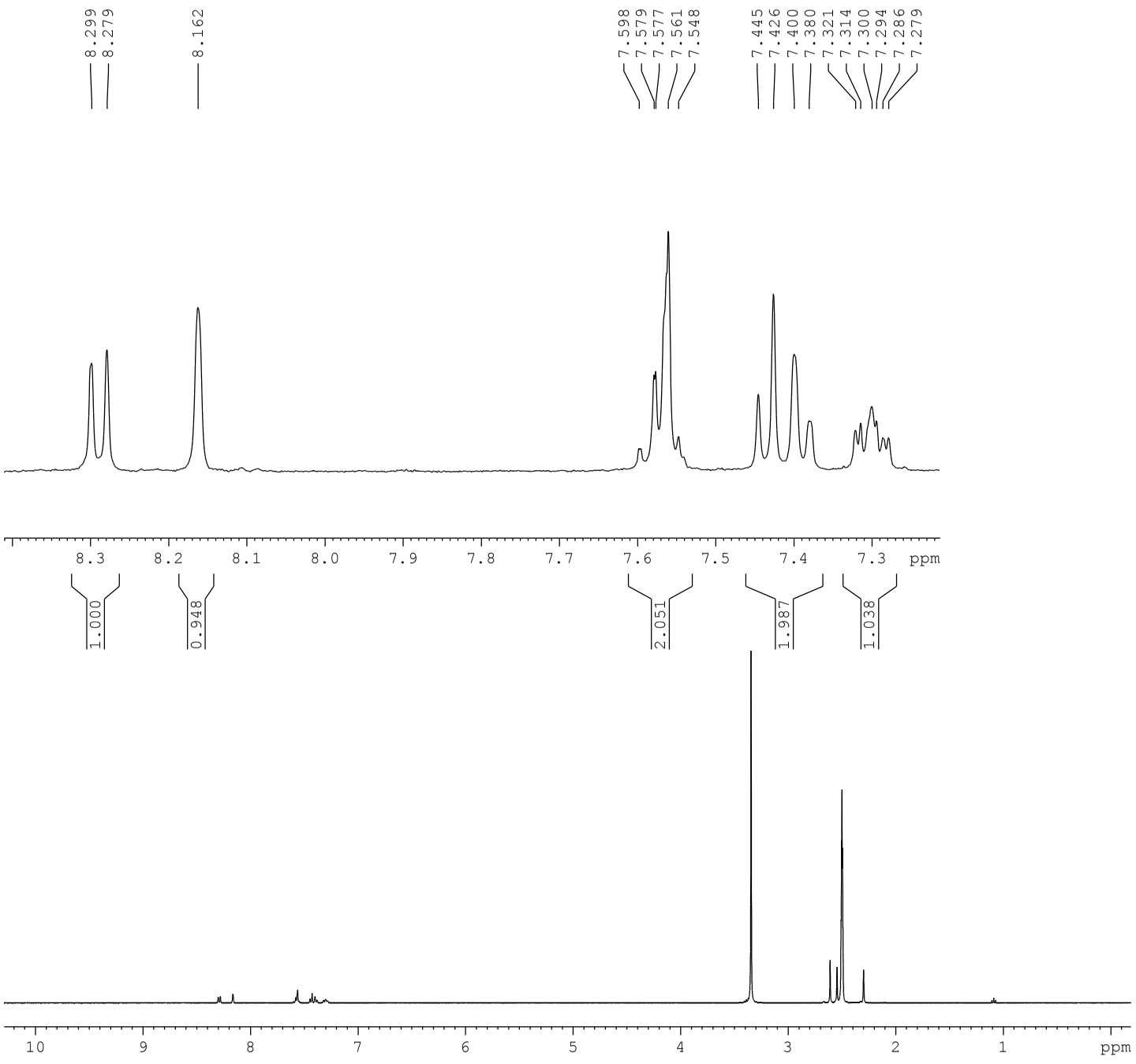


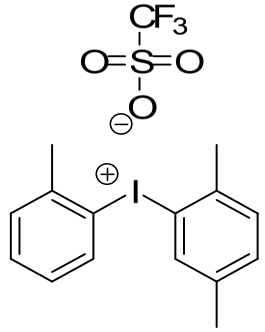
100 MHz
DMSO

3v



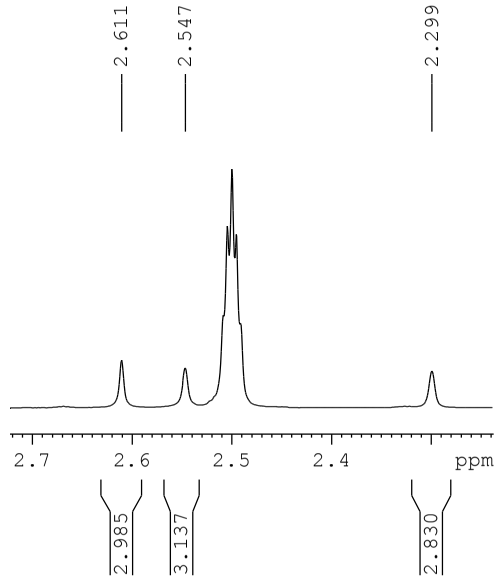


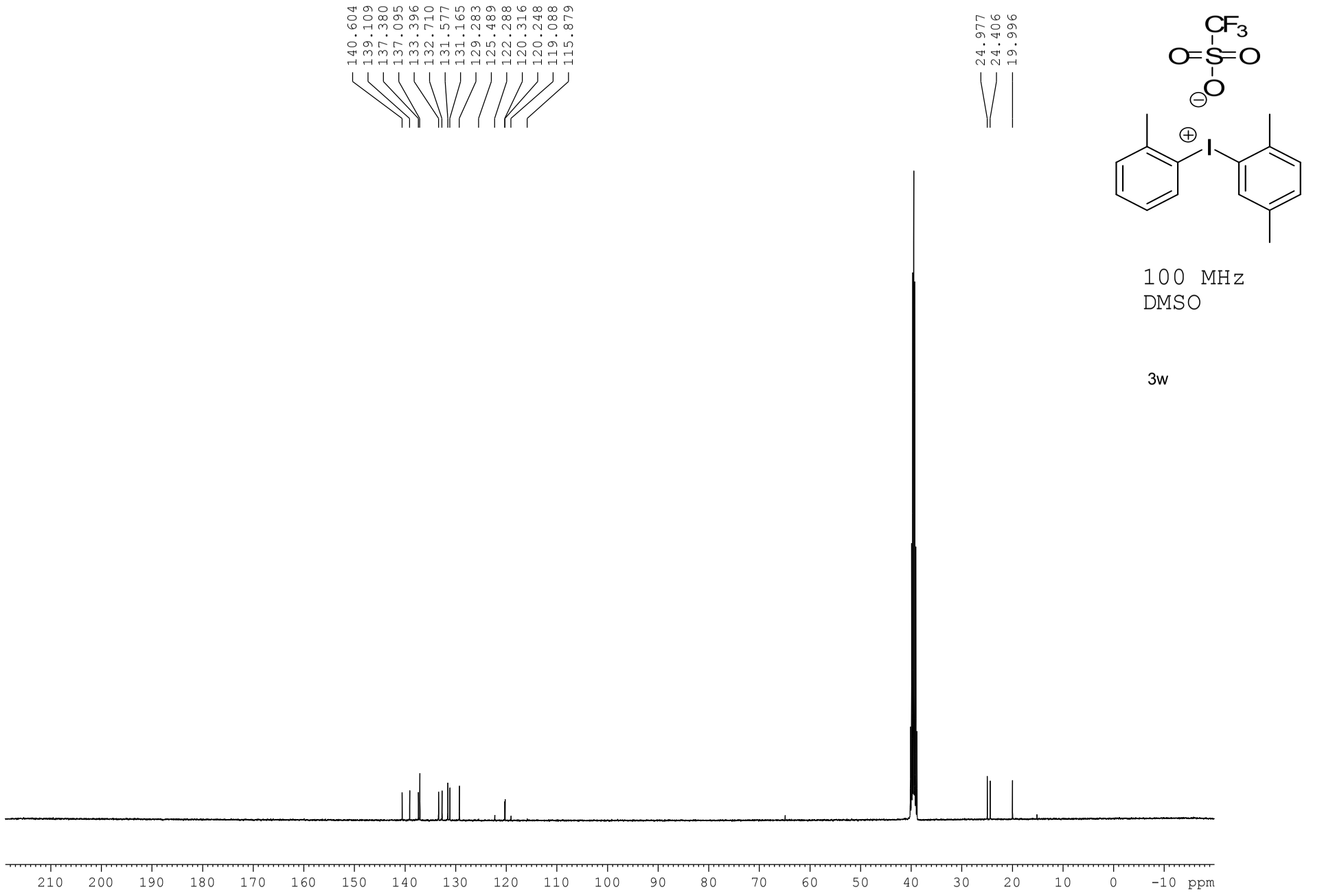


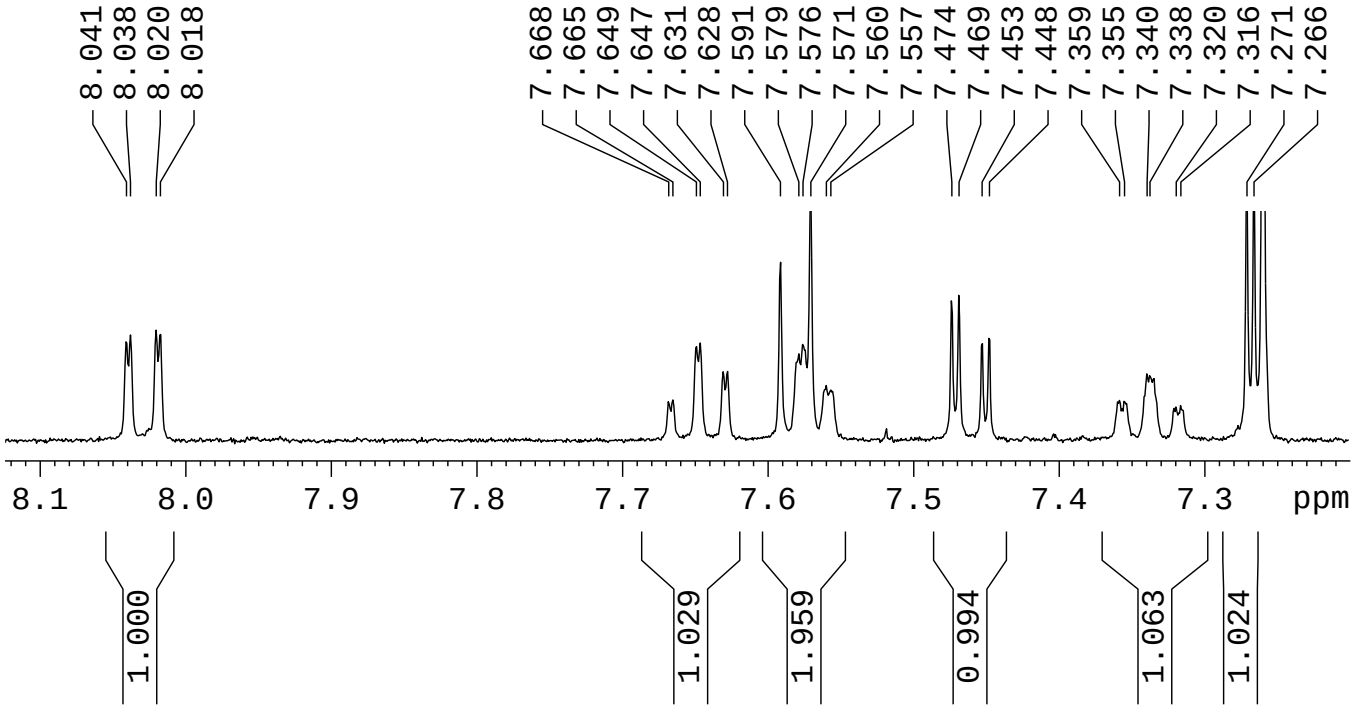


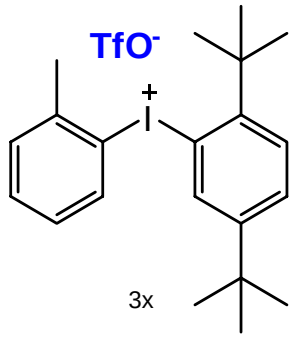
400 MHz
DMSO

3w

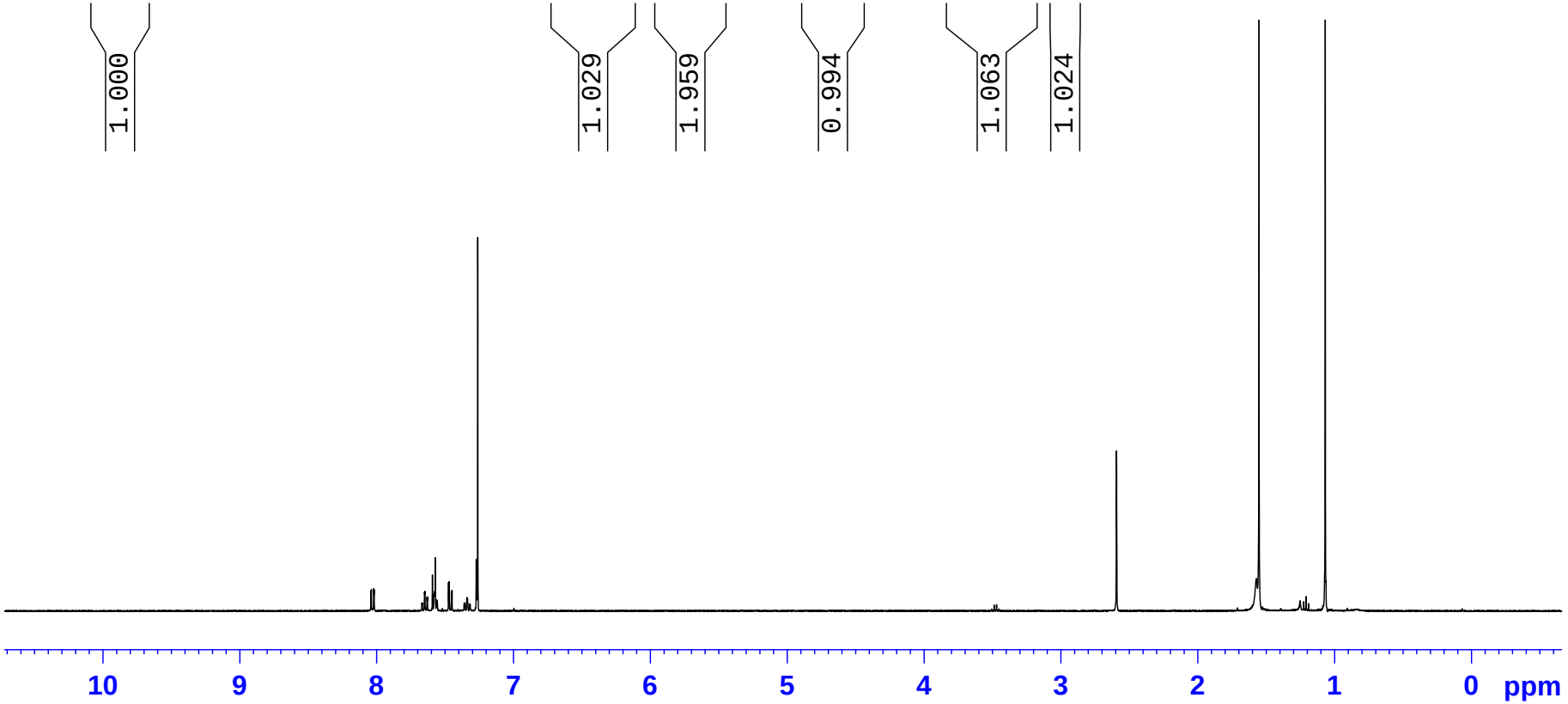


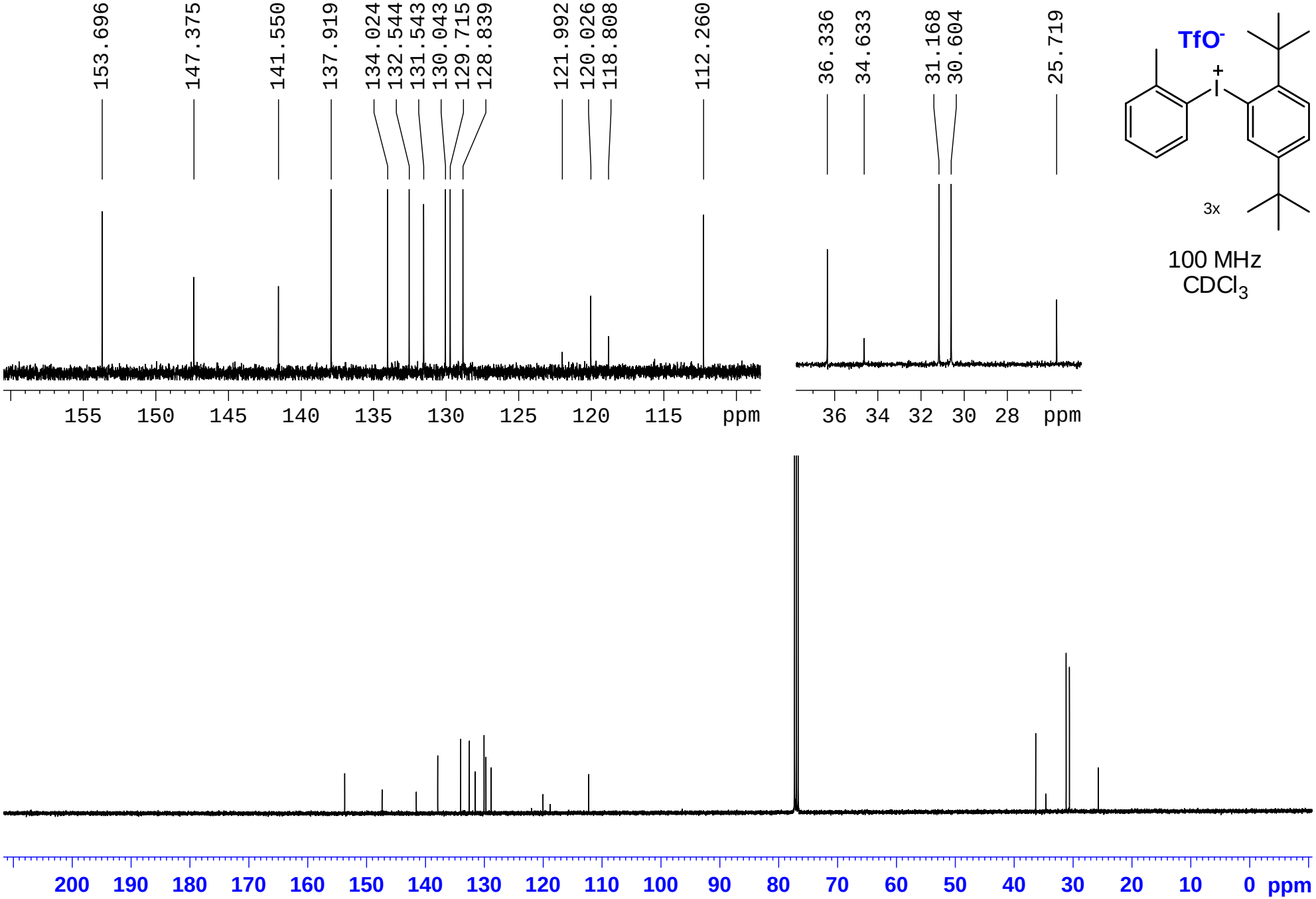

140.604
139.109
137.380
137.095
133.396
132.710
131.577
131.165
129.283
125.489
122.288
120.316
120.248
119.088
115.879
24.977
24.406
19.996
100 MHz
DMSO
3w
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10 ppm

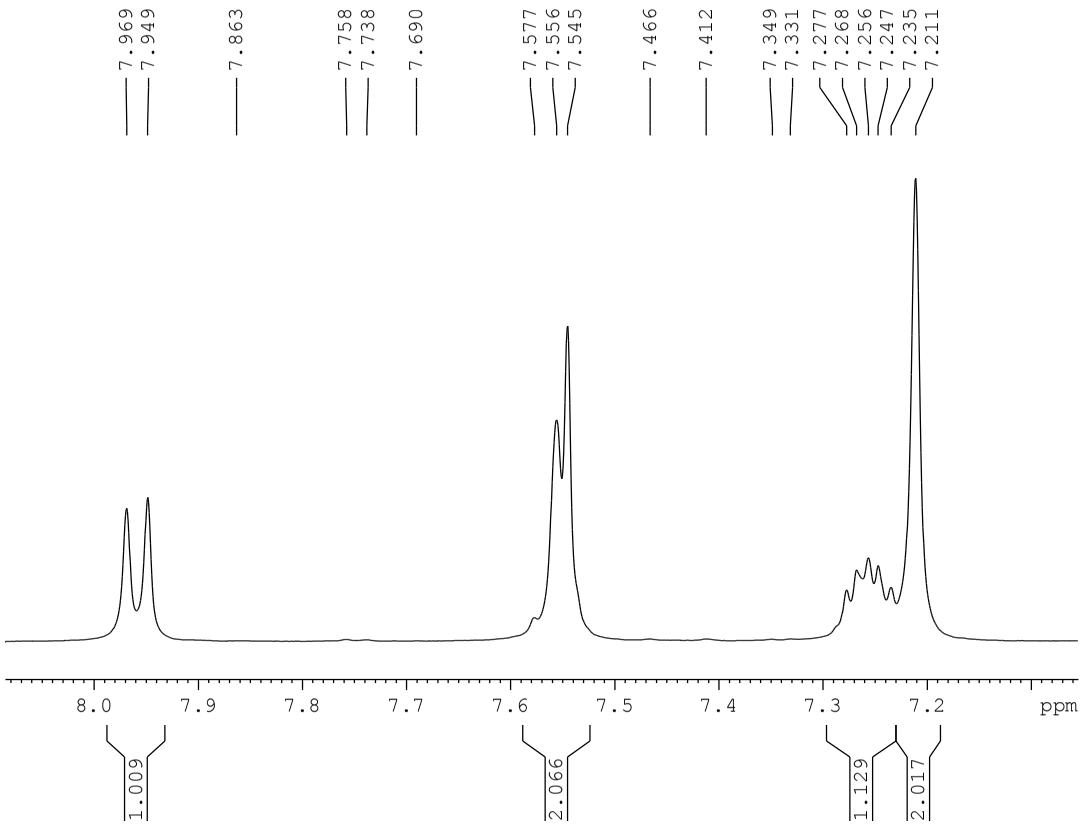


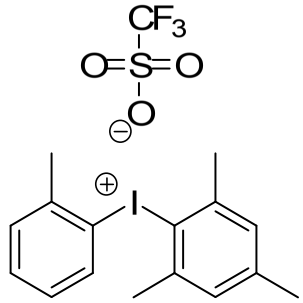


400 MHz
$CDCl_3$



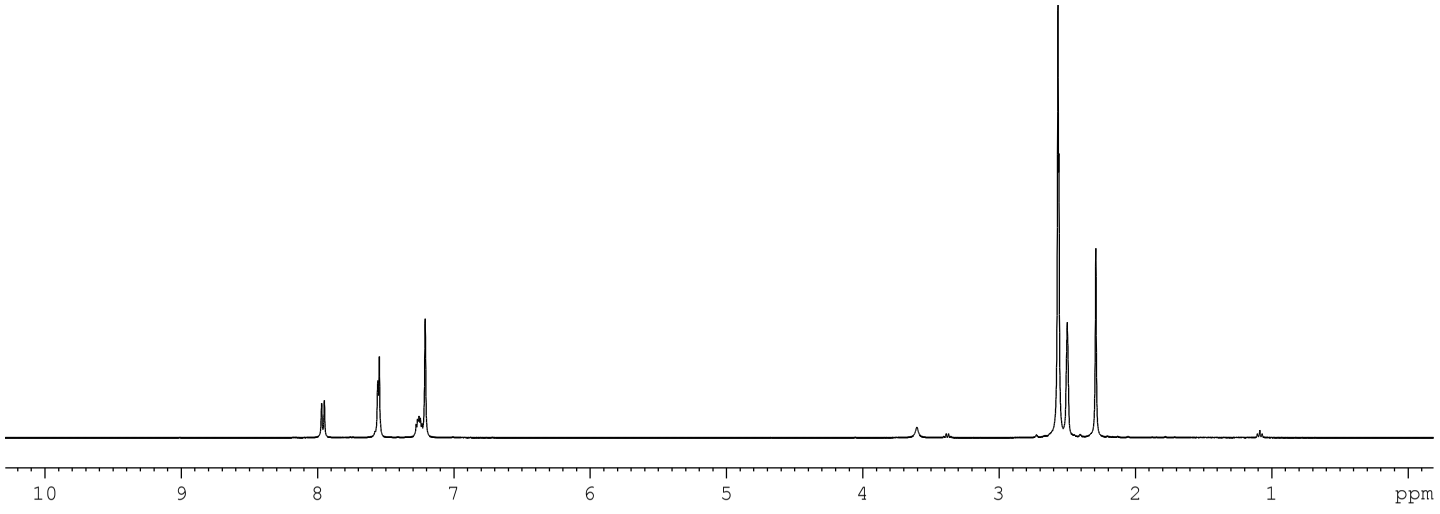

153.696
147.375
141.550
137.919
134.024
132.544
131.543
130.043
129.715
128.839
121.992
120.026
118.808
112.260
36.336
34.633
31.168
30.604
25.719
155 150 145 140 135 130 125 120 115 ppm
36 34 32 30 28 ppm
TfO-
3x
100 MHz
CDCl3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

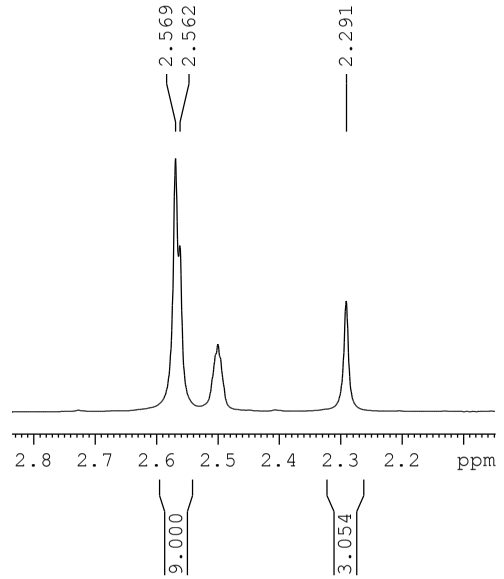


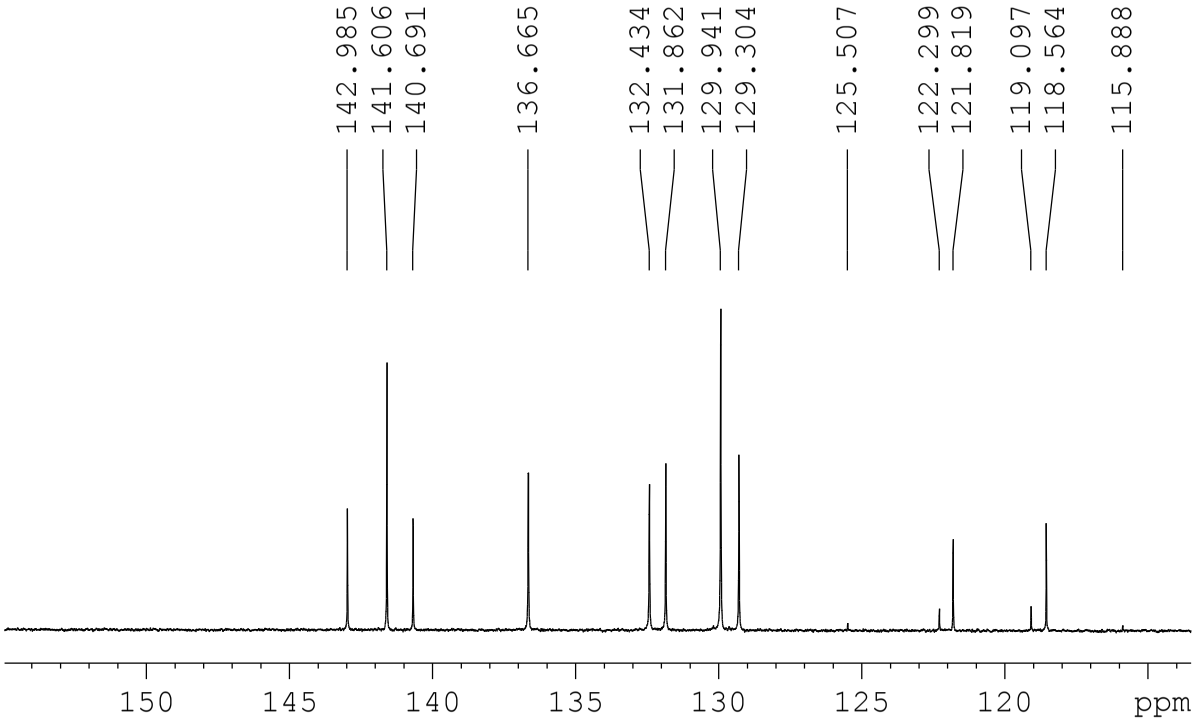


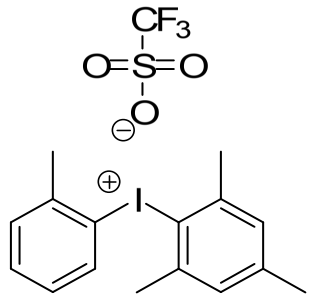
400 MHz
DMSO

3y



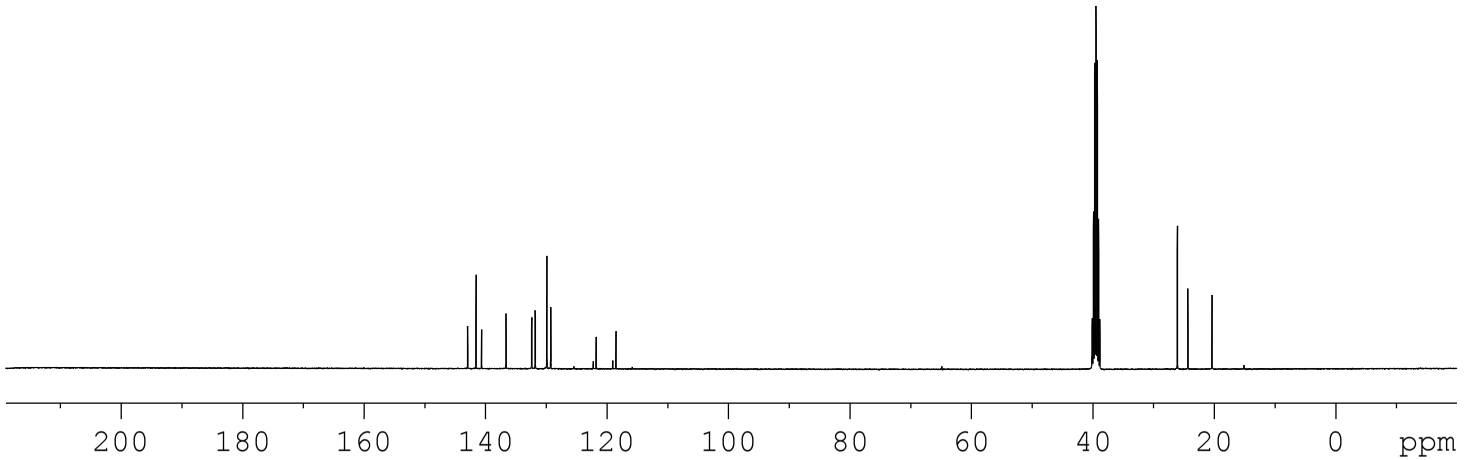


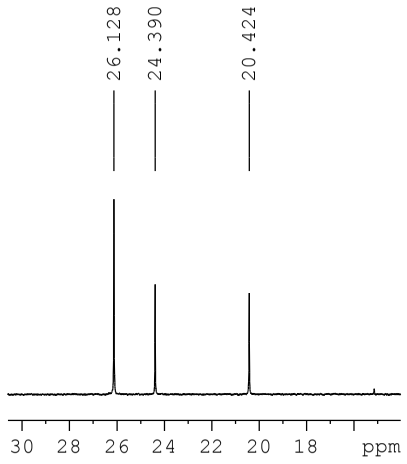


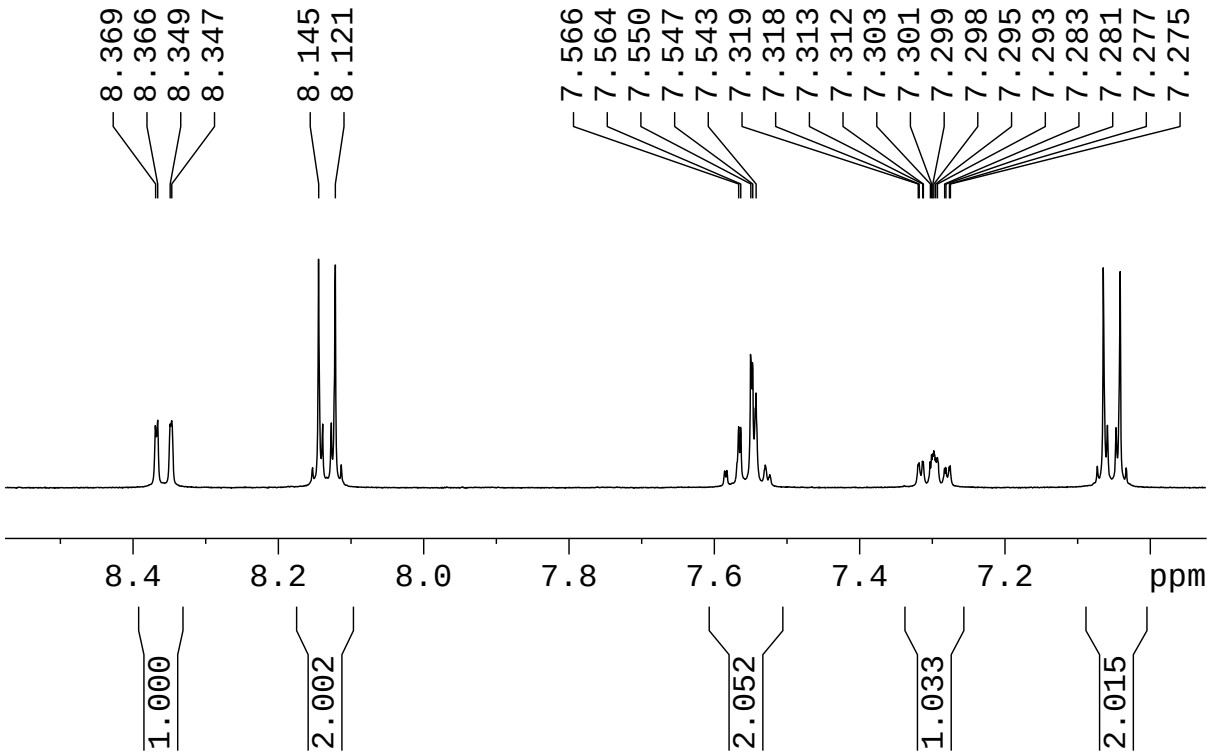


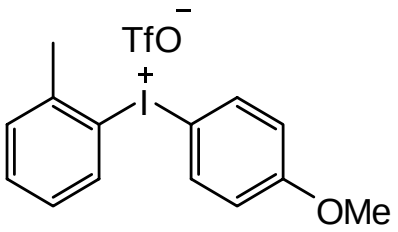
100 MHz
DMSO

3y



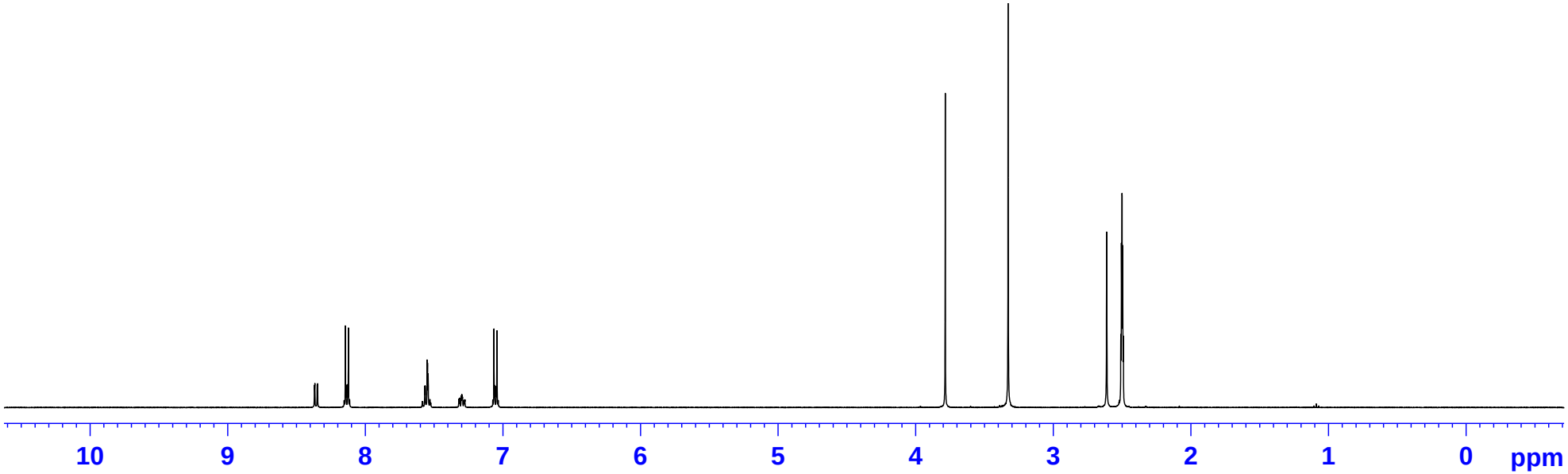


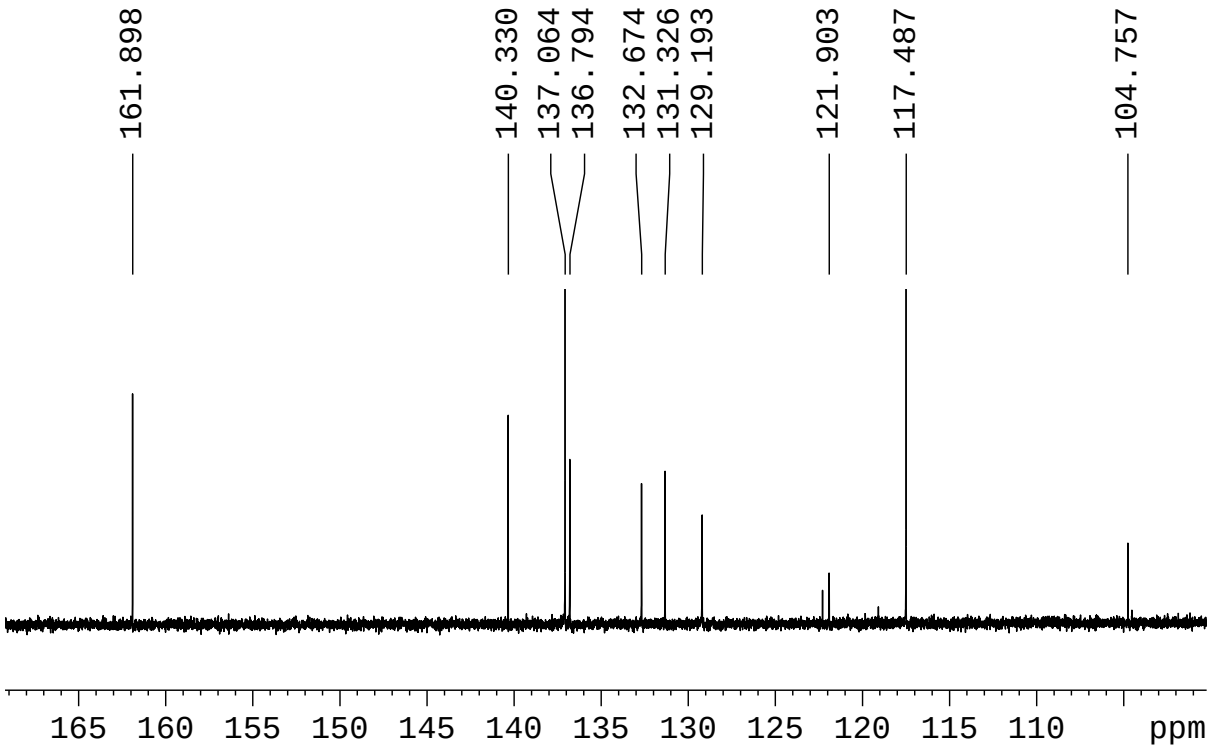


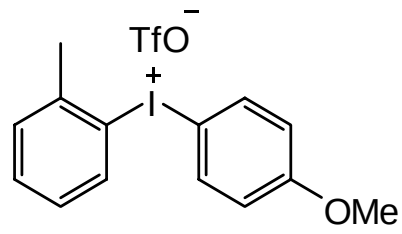


3z

400 MHz
DMSO-d_6

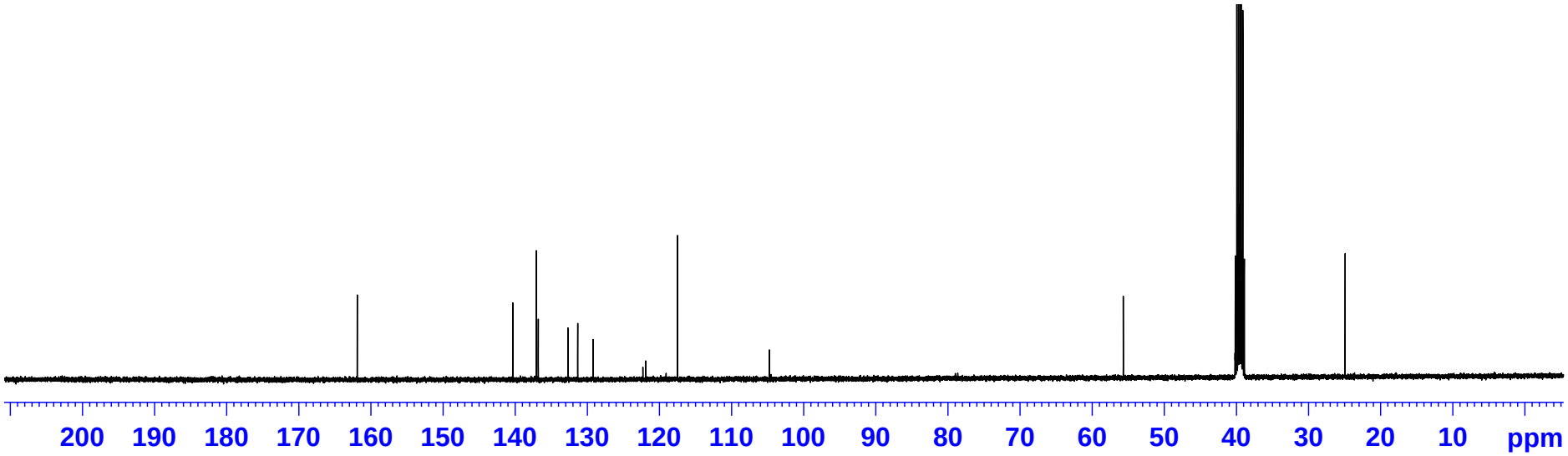


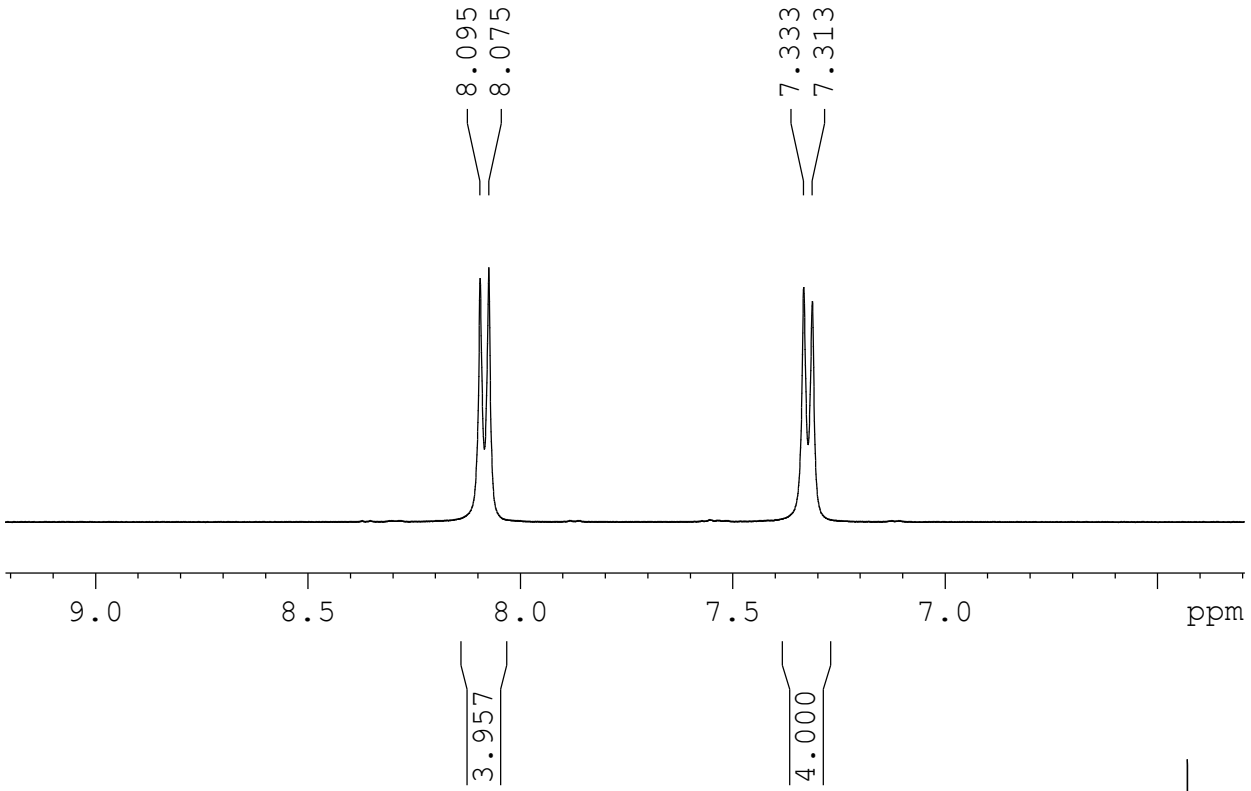


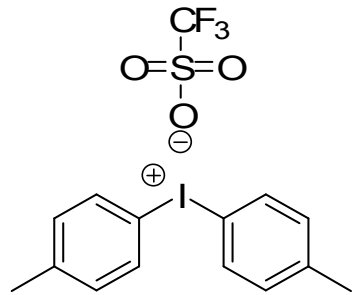


3z

100 MHz
DMSO-d_6

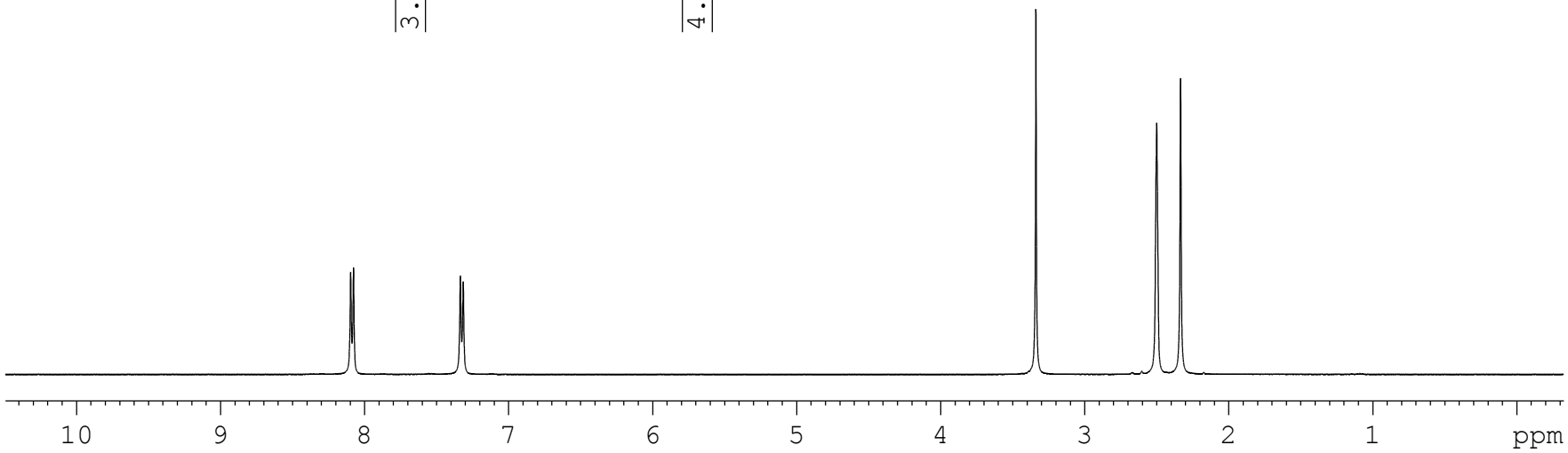


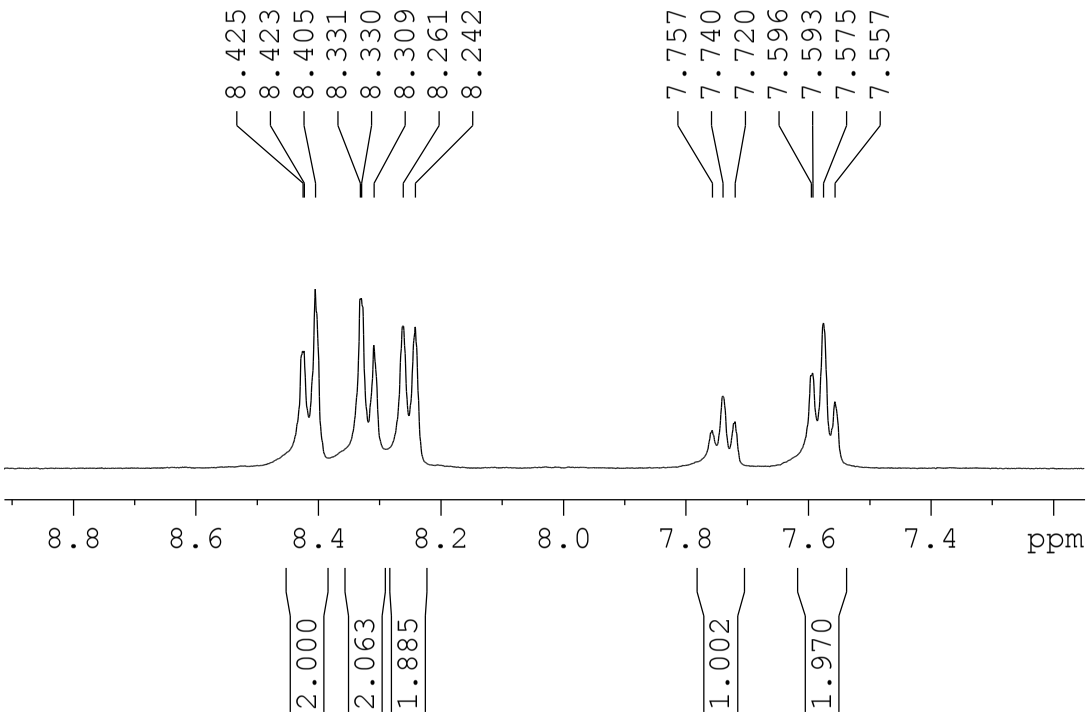


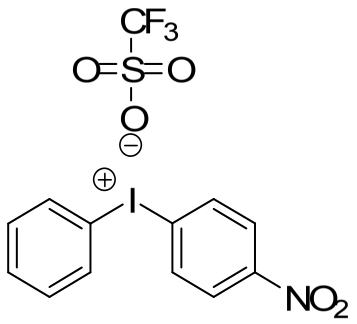


400 MHz
DMSO

3aa

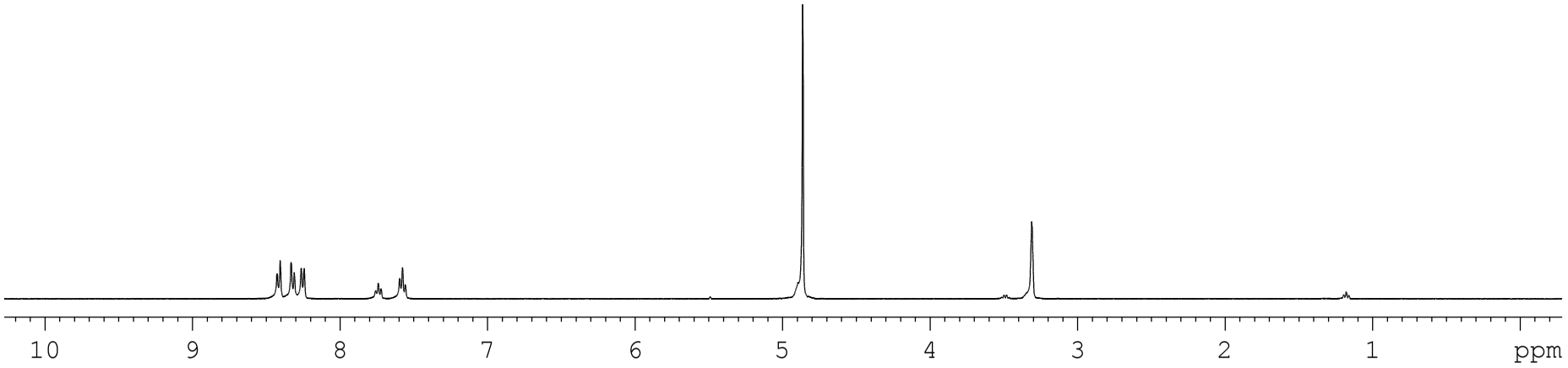


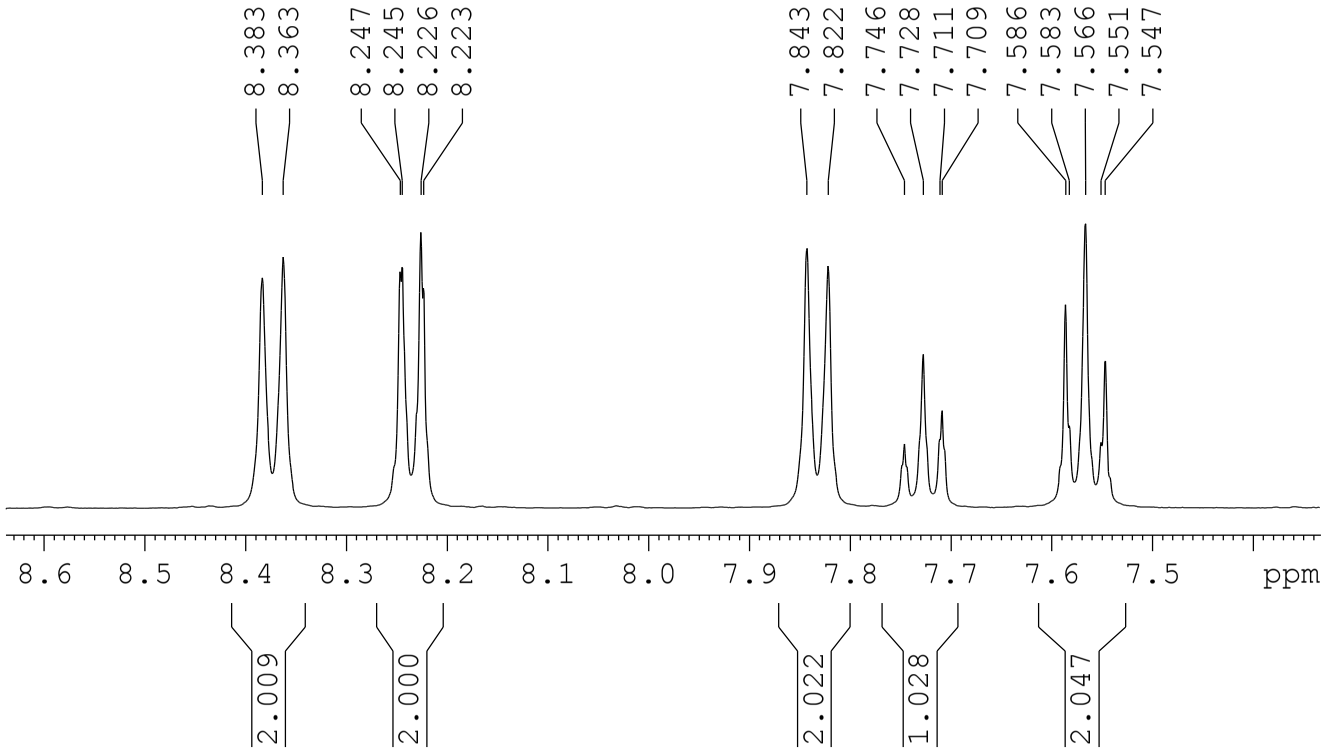


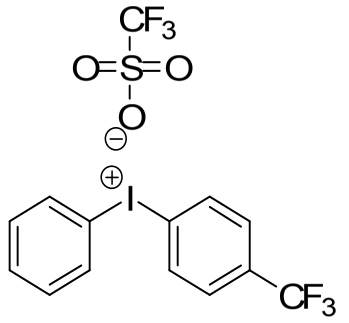


400 MHz
MeOD

3ab

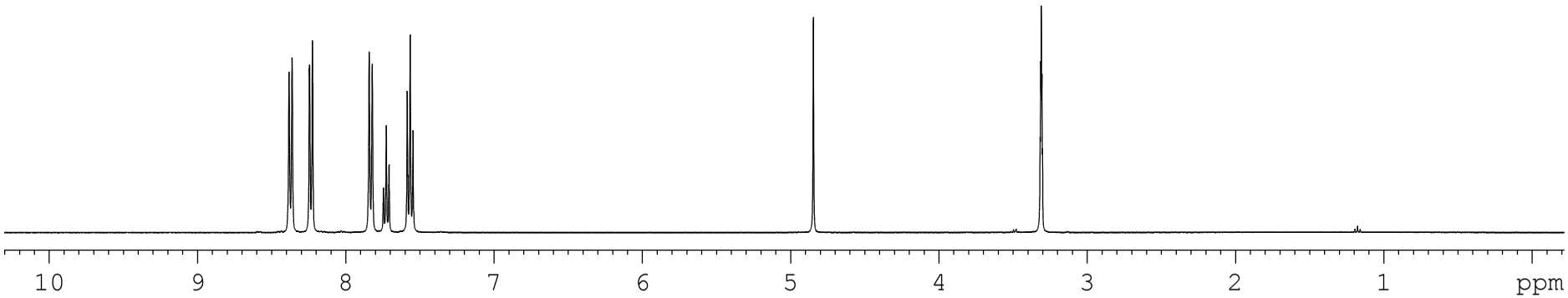


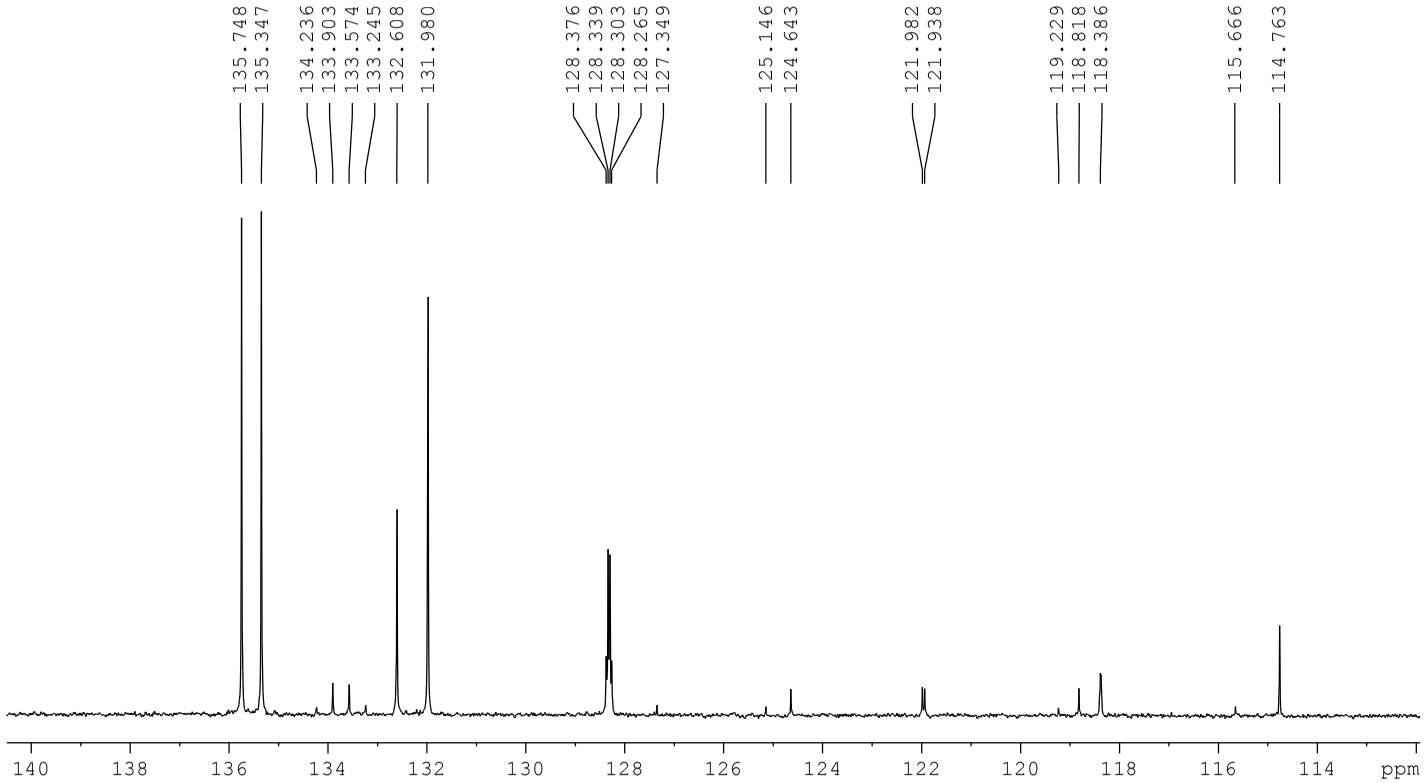


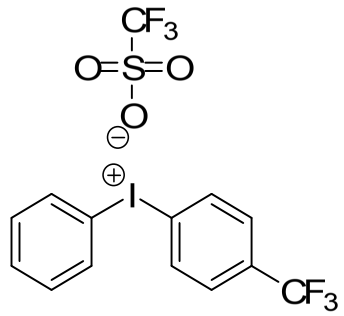


400 MHz
MeOD

3ac

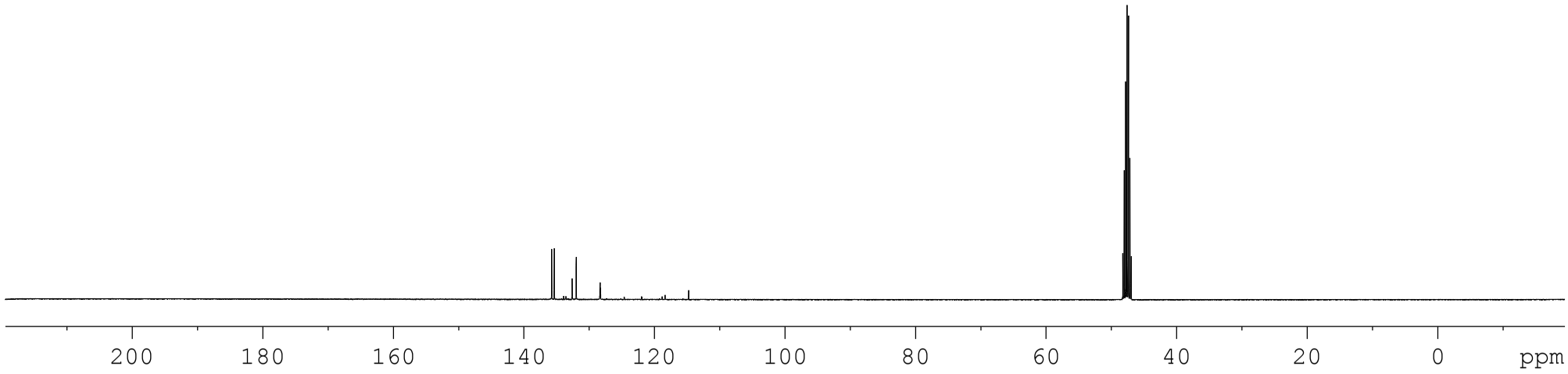


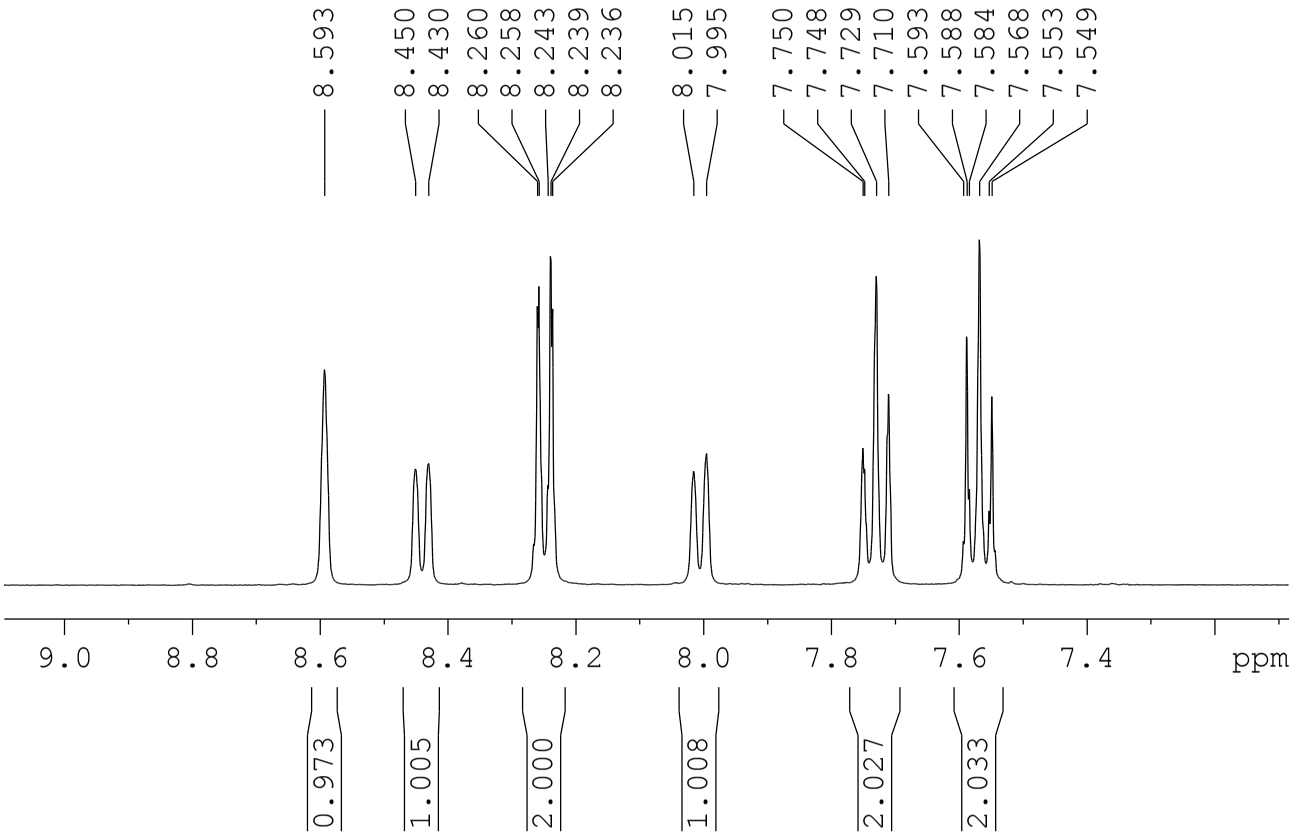


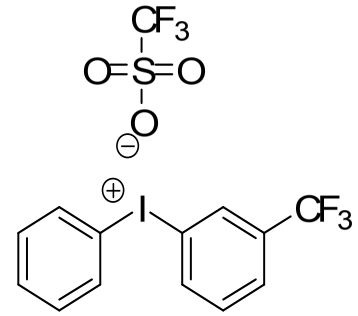


100 MHz
MeOD

3ac

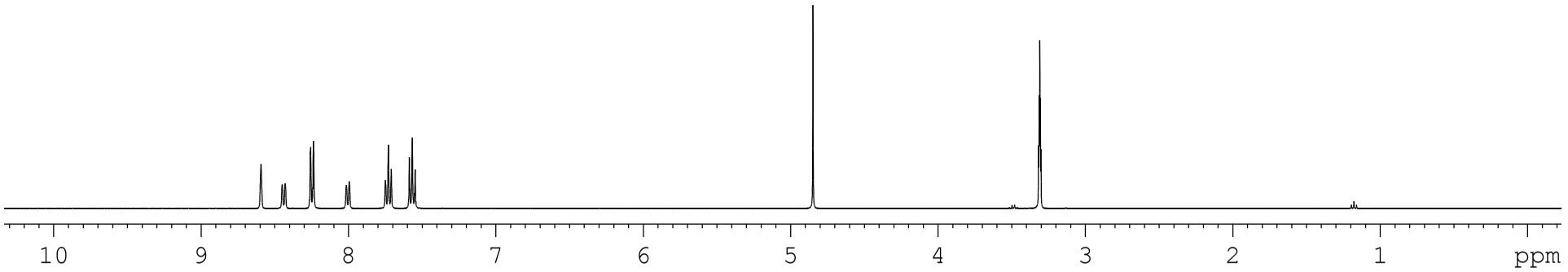


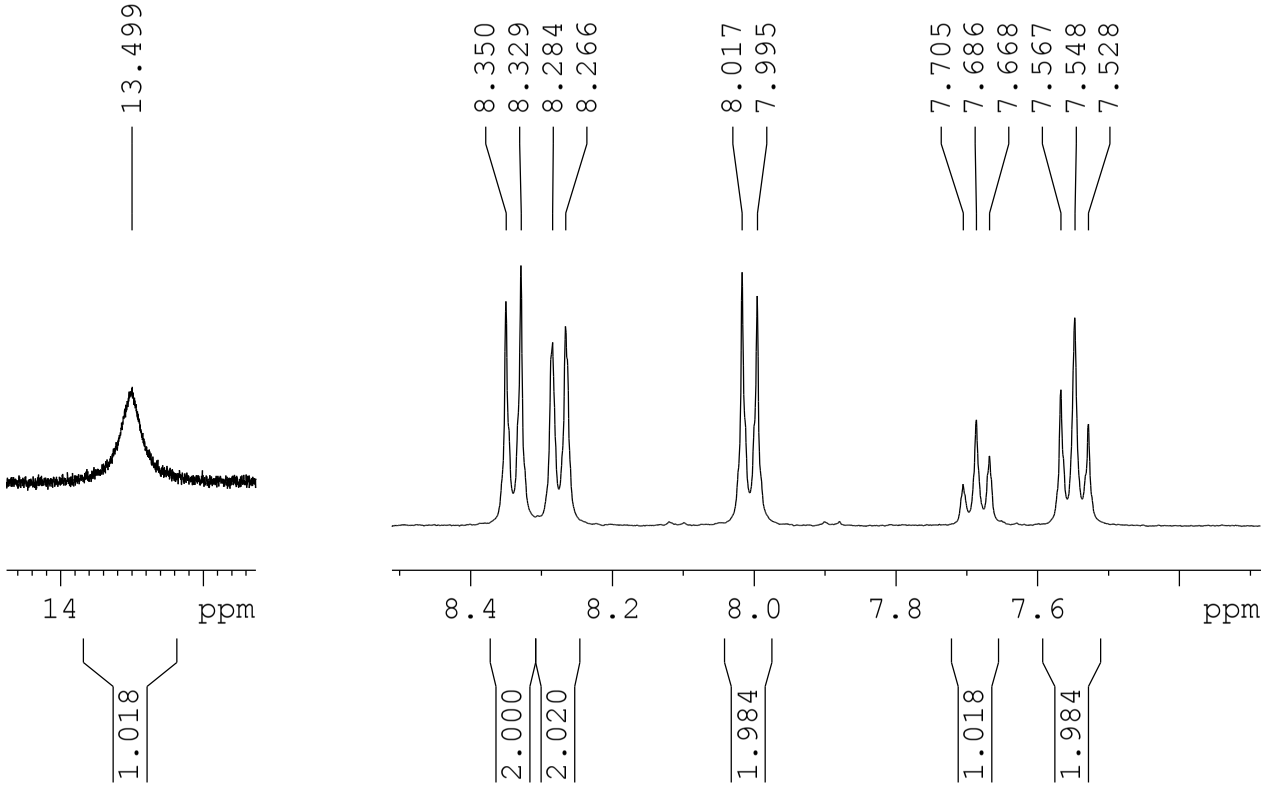


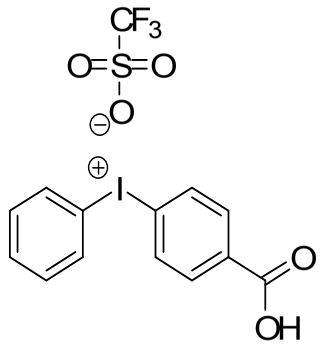


400 MHz
MeOD

3ad

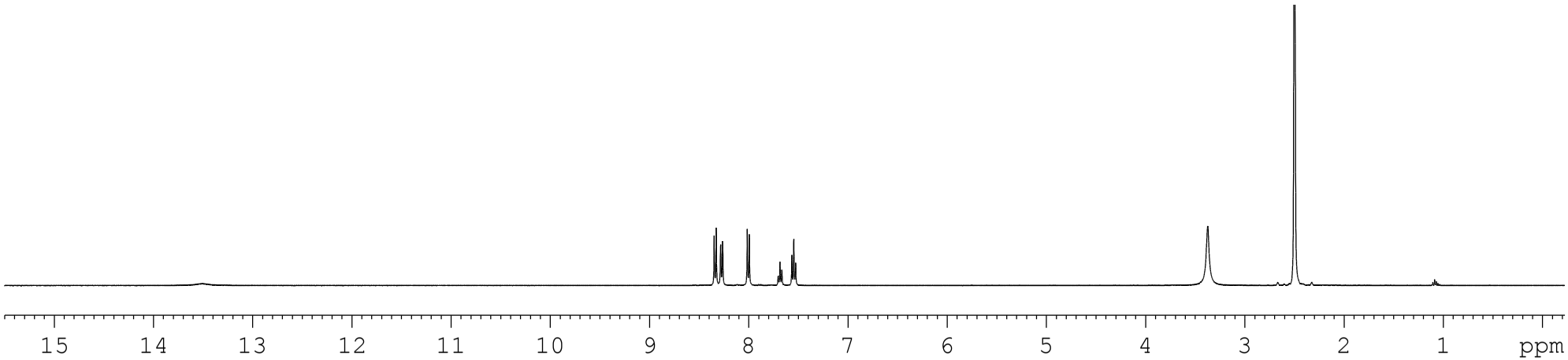


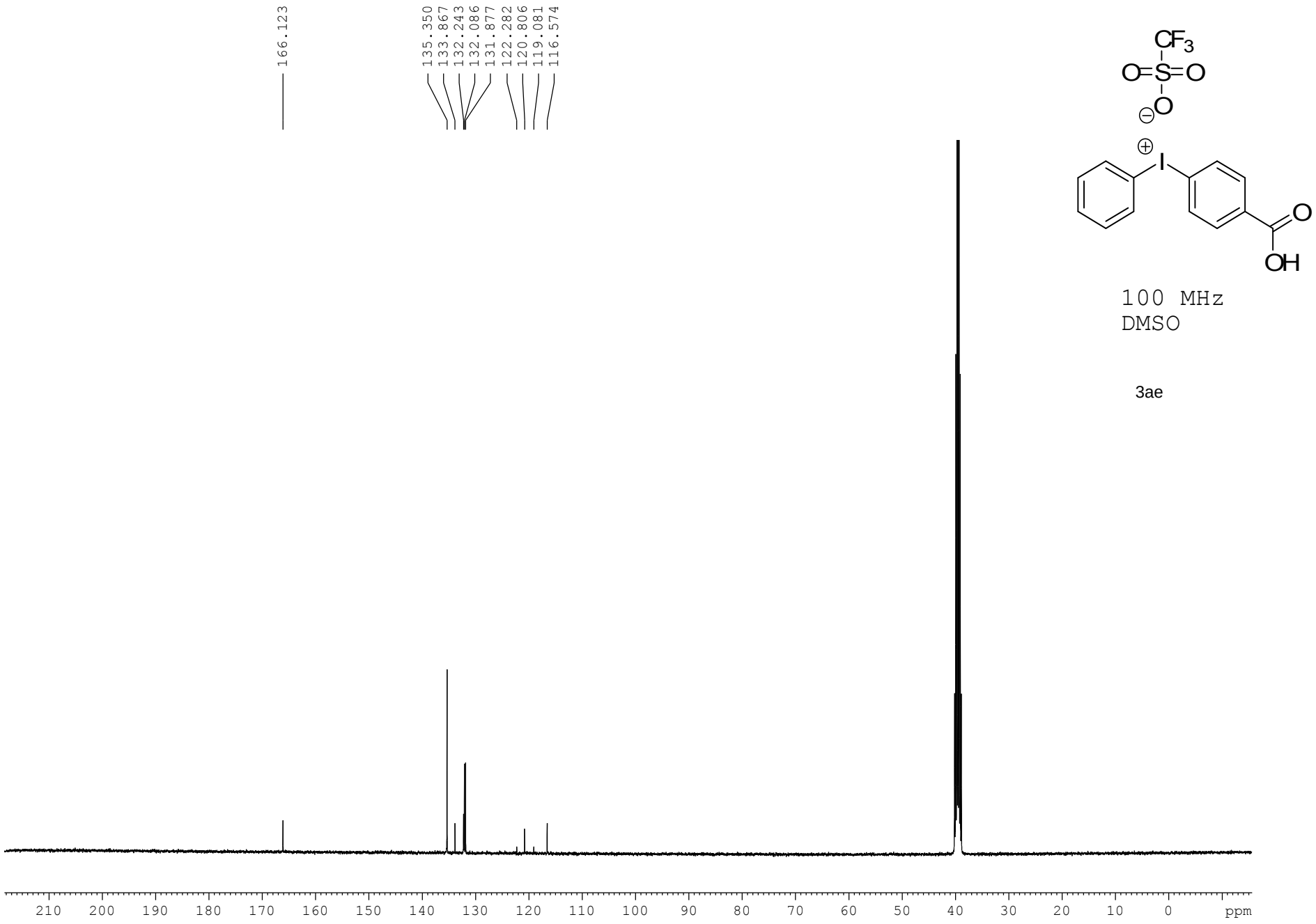


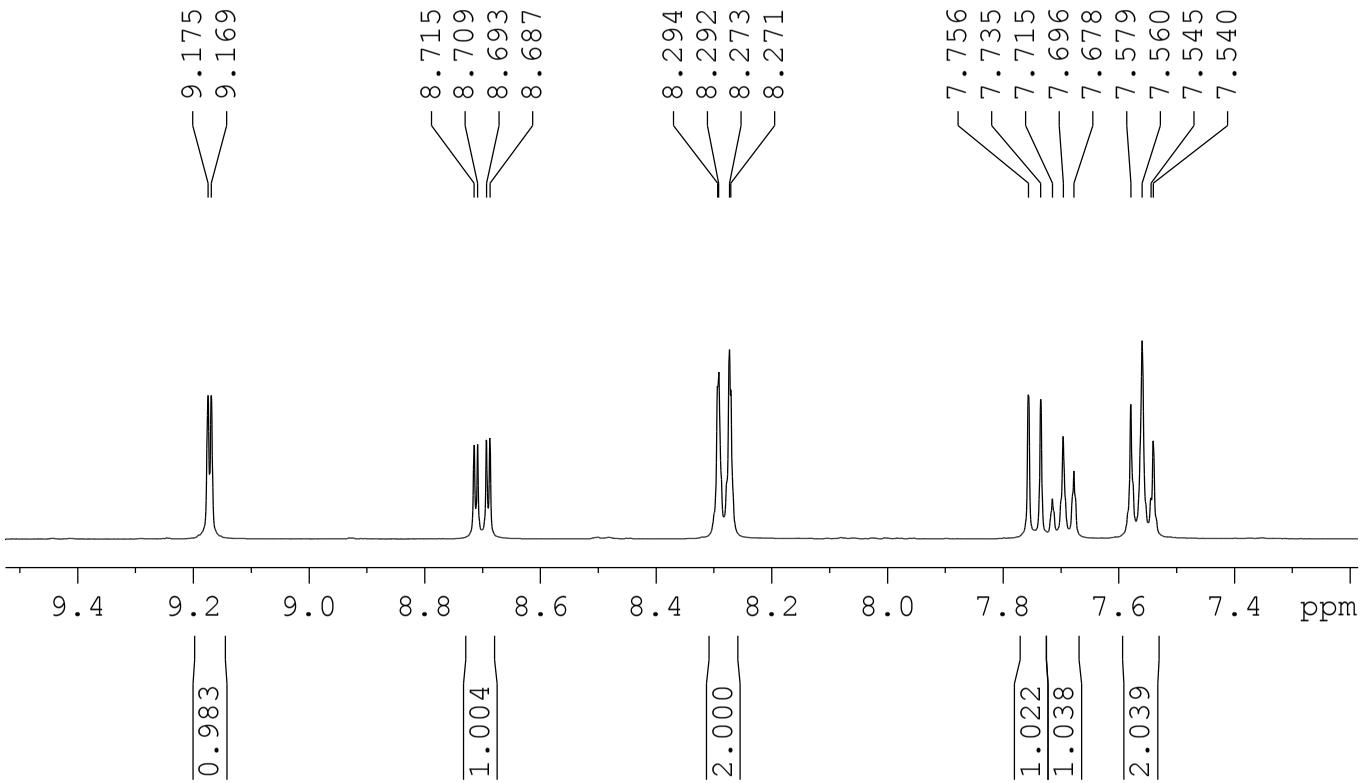


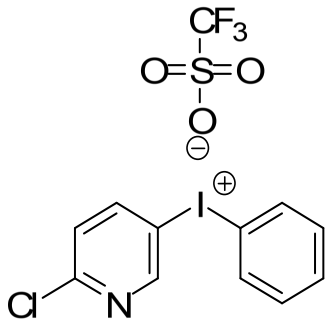
400 MHz
DMSO

3ae



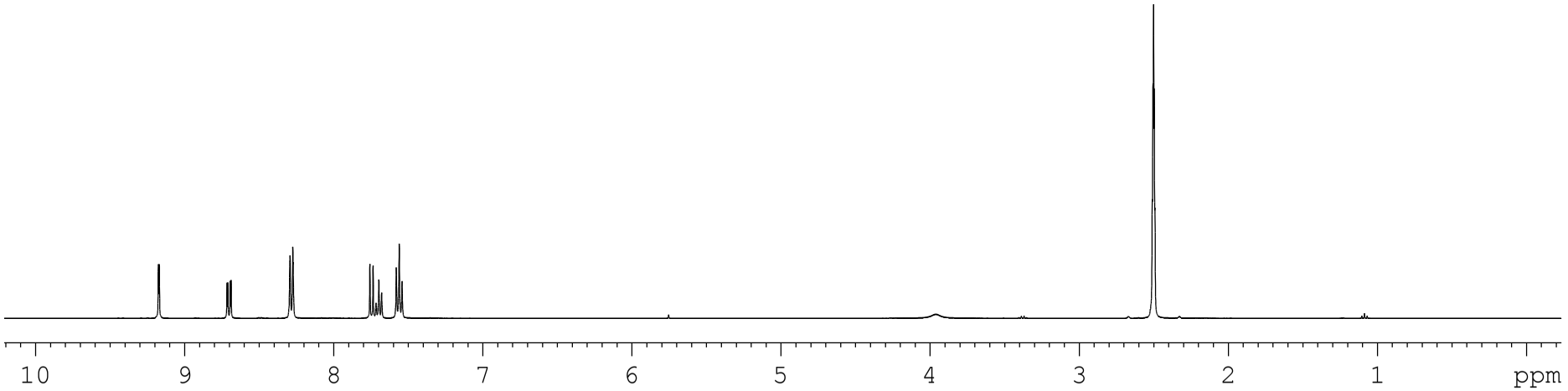

166.123
135.350
133.867
132.243
132.086
131.877
122.282
120.806
119.081
116.574
100 MHz
DMSO
3ae
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

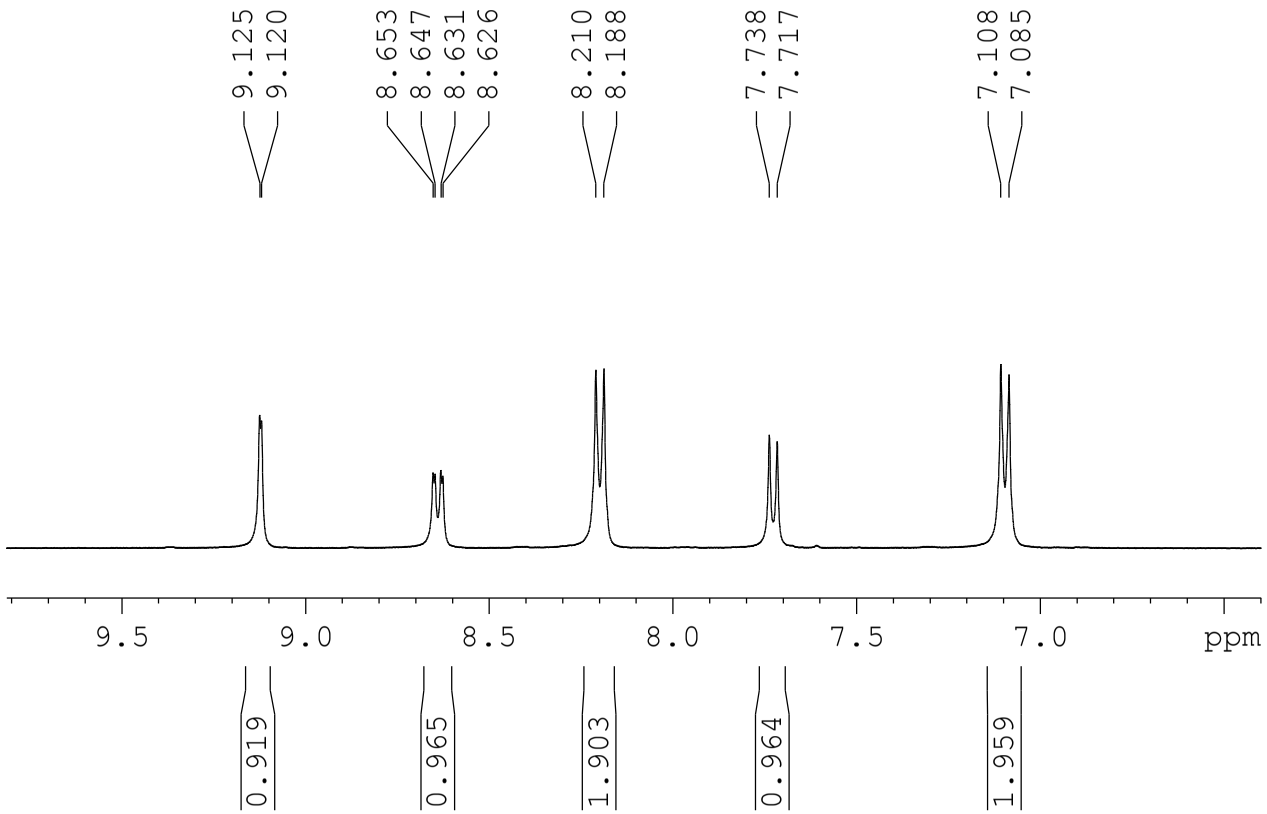


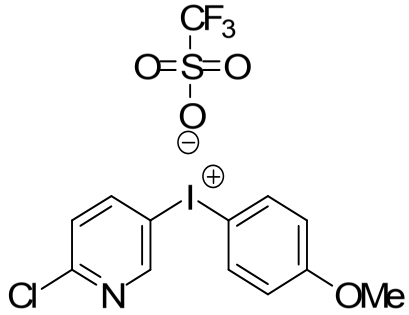


400 MHz
MeOD

3af

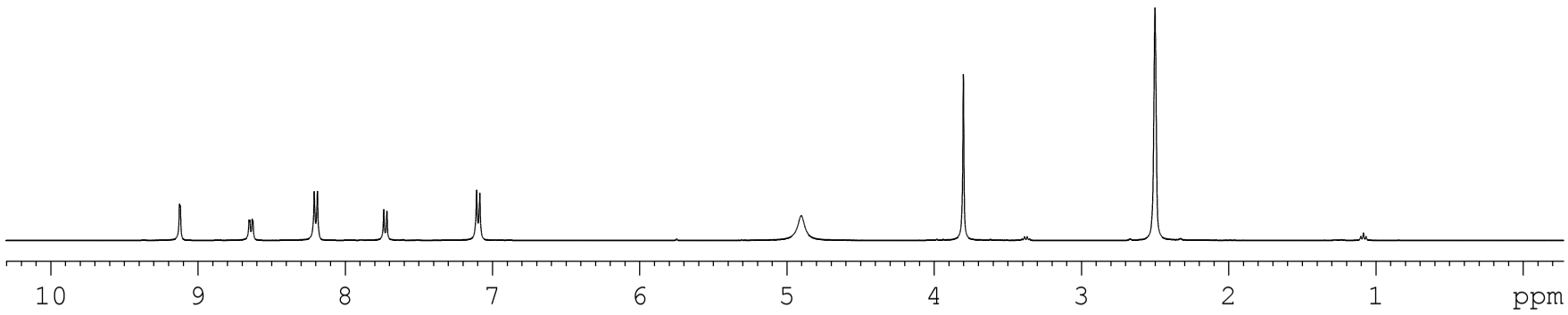


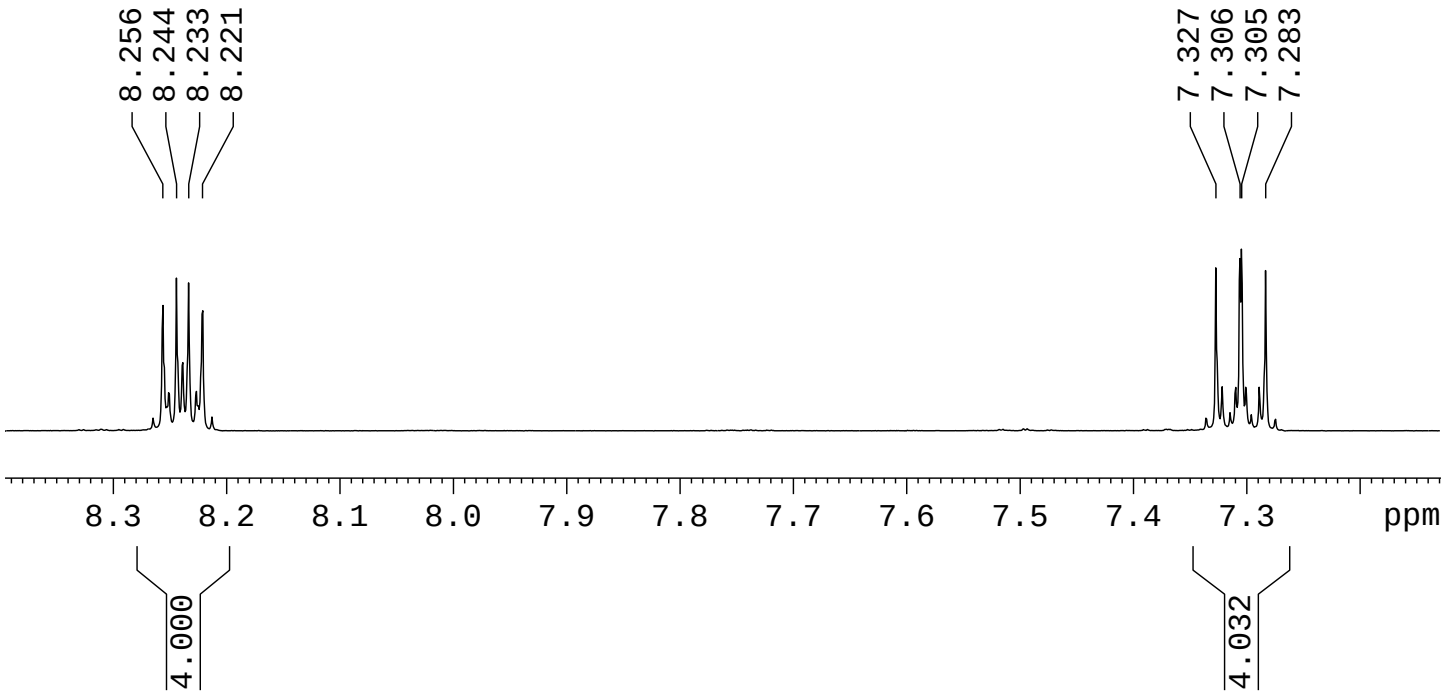


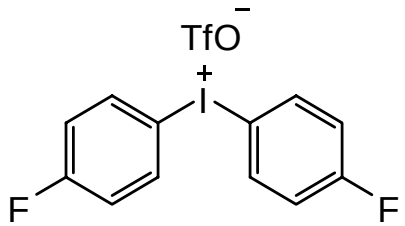


400 MHz
DMSO

3ag

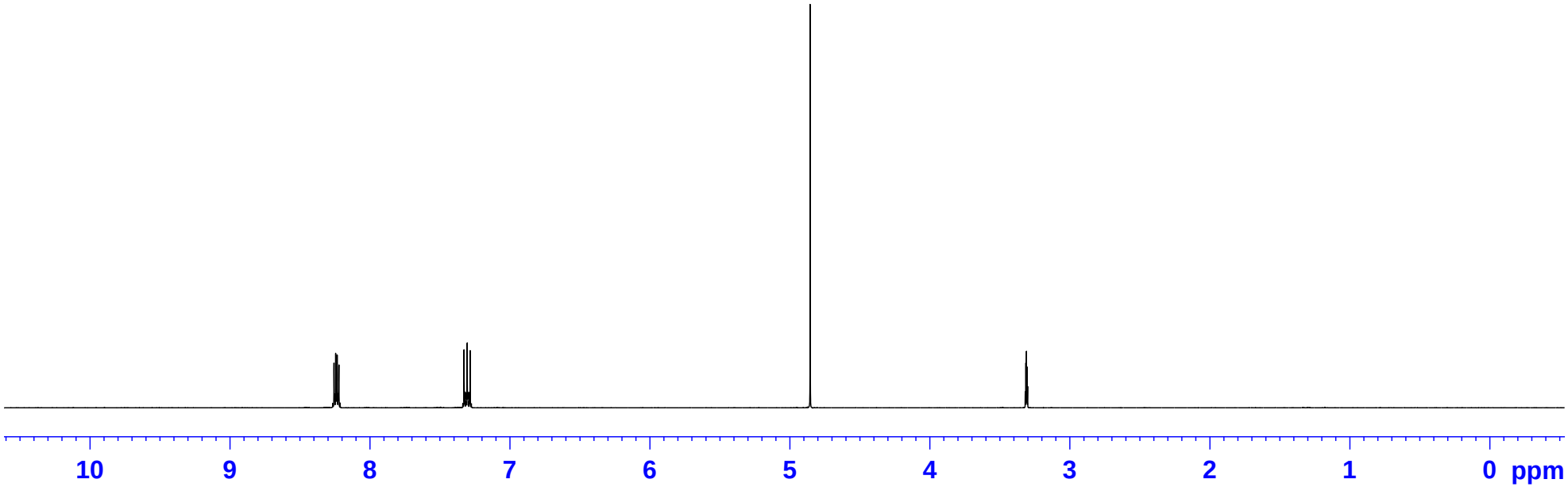


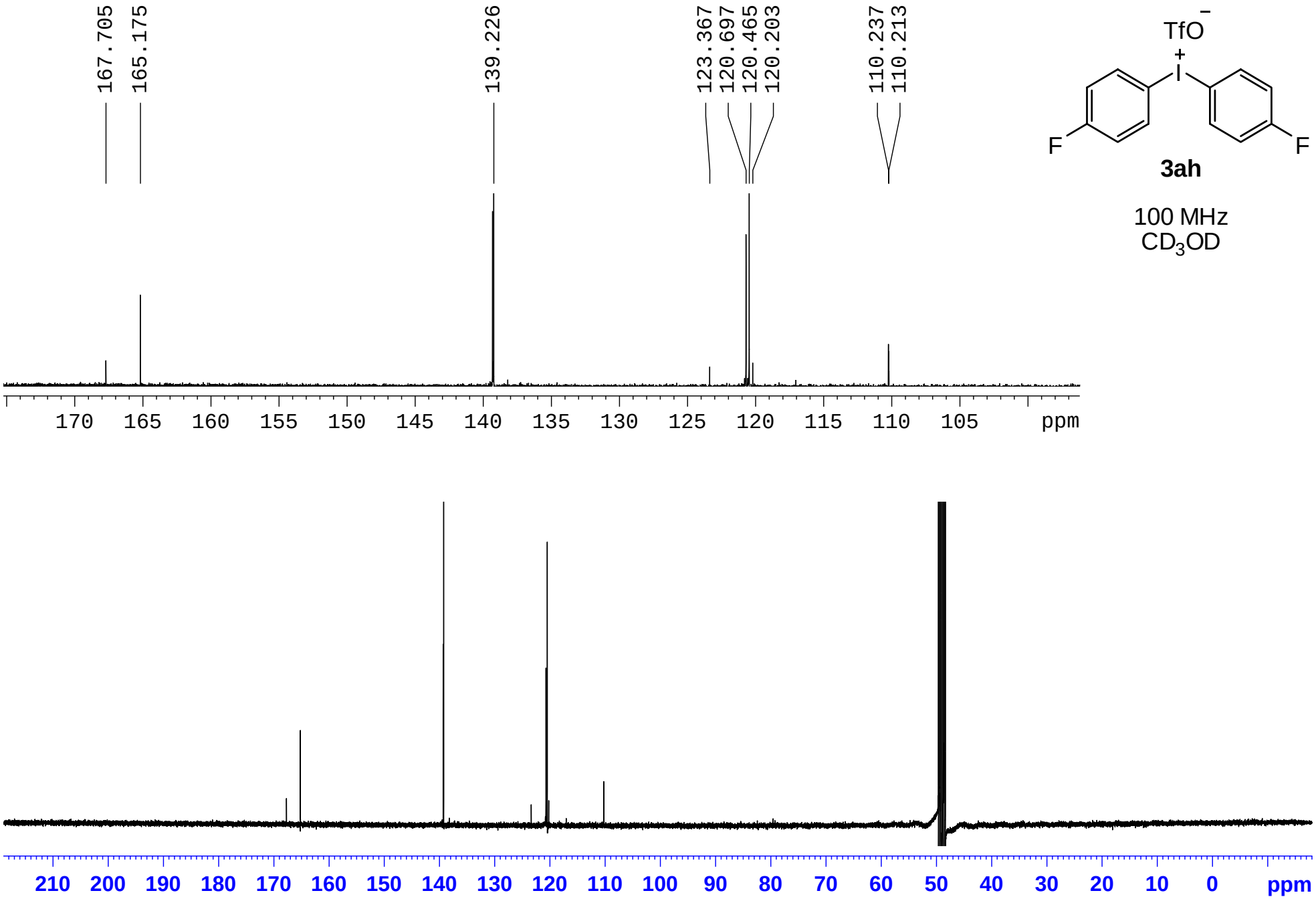


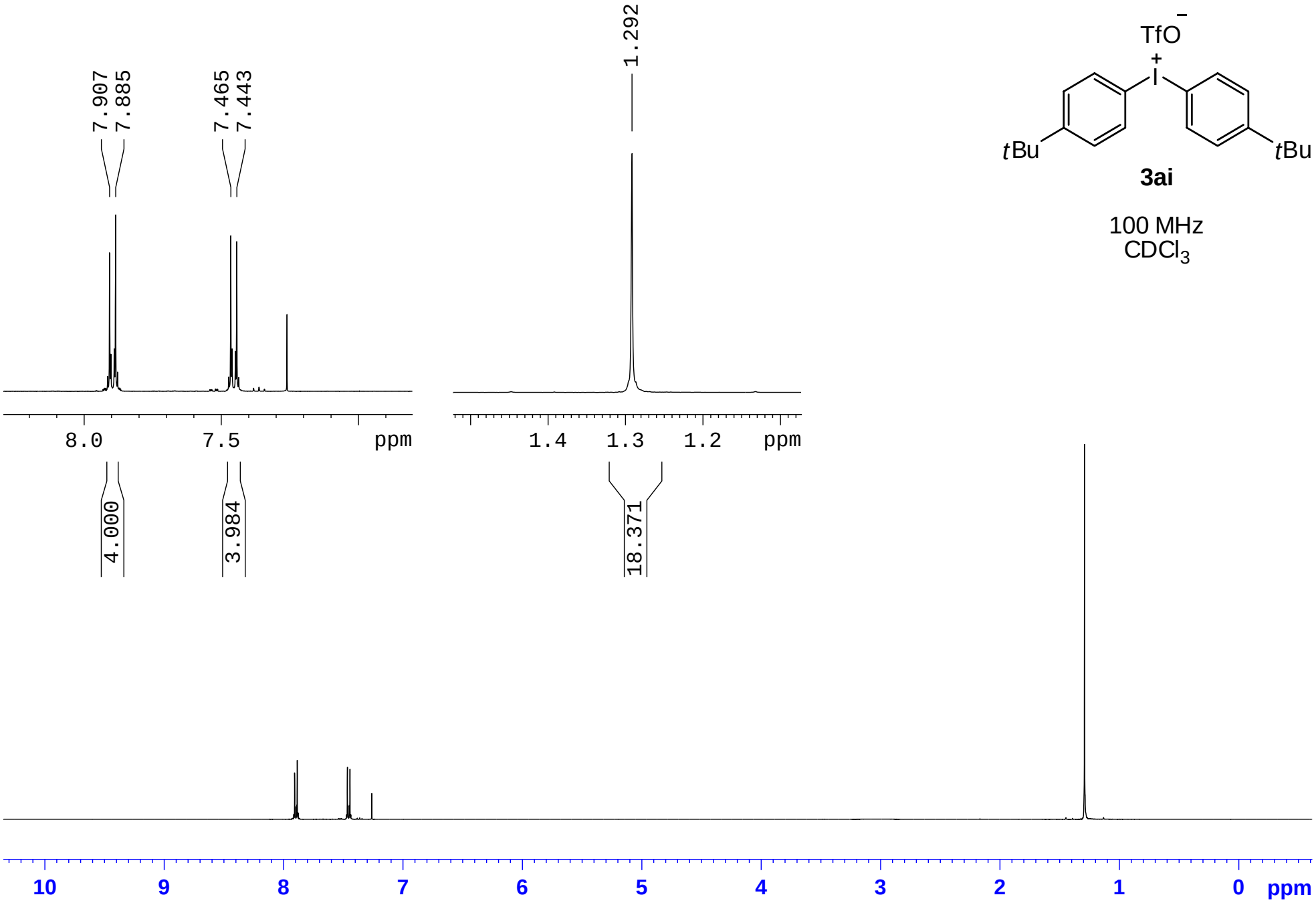


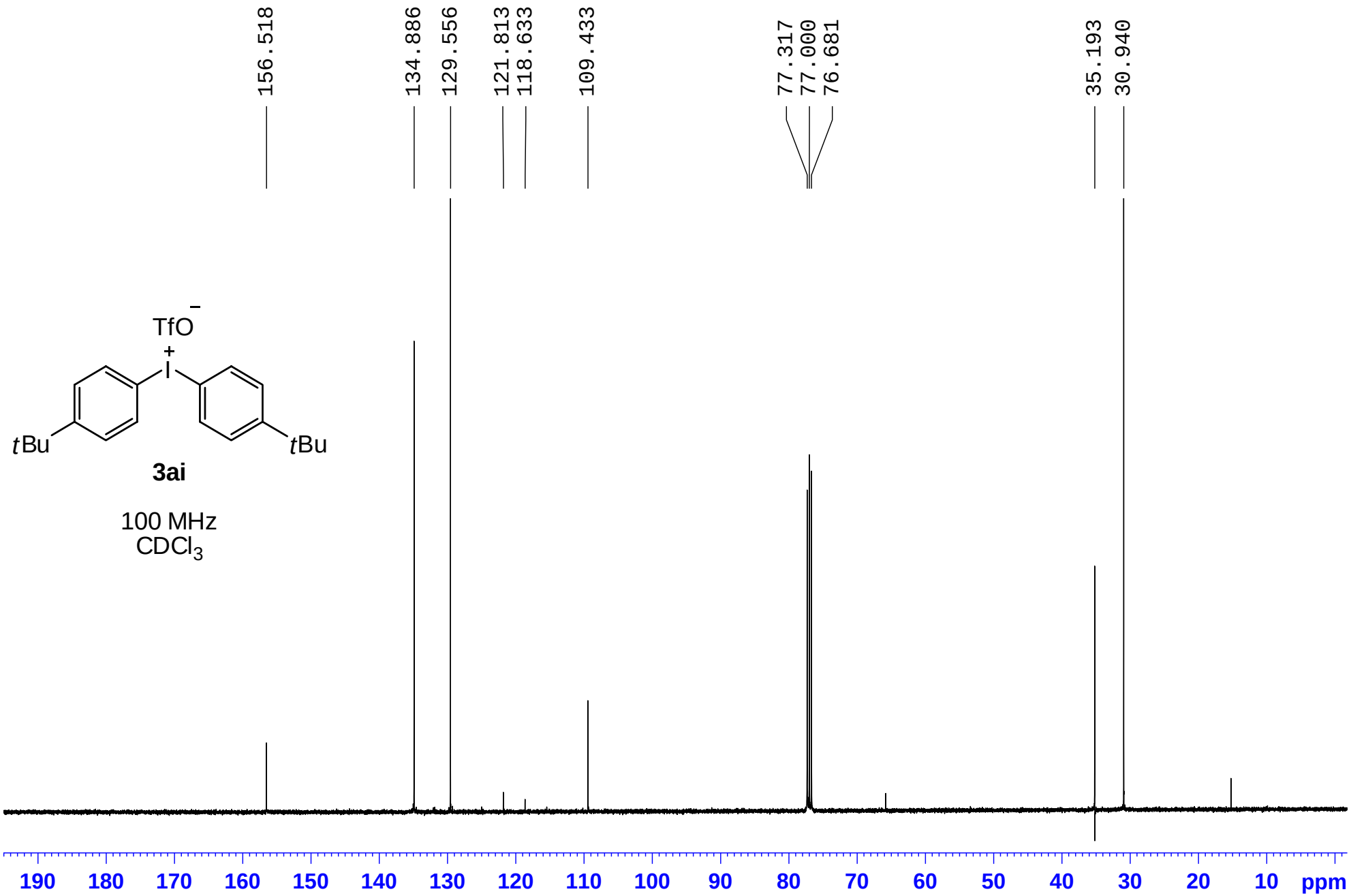
3ah

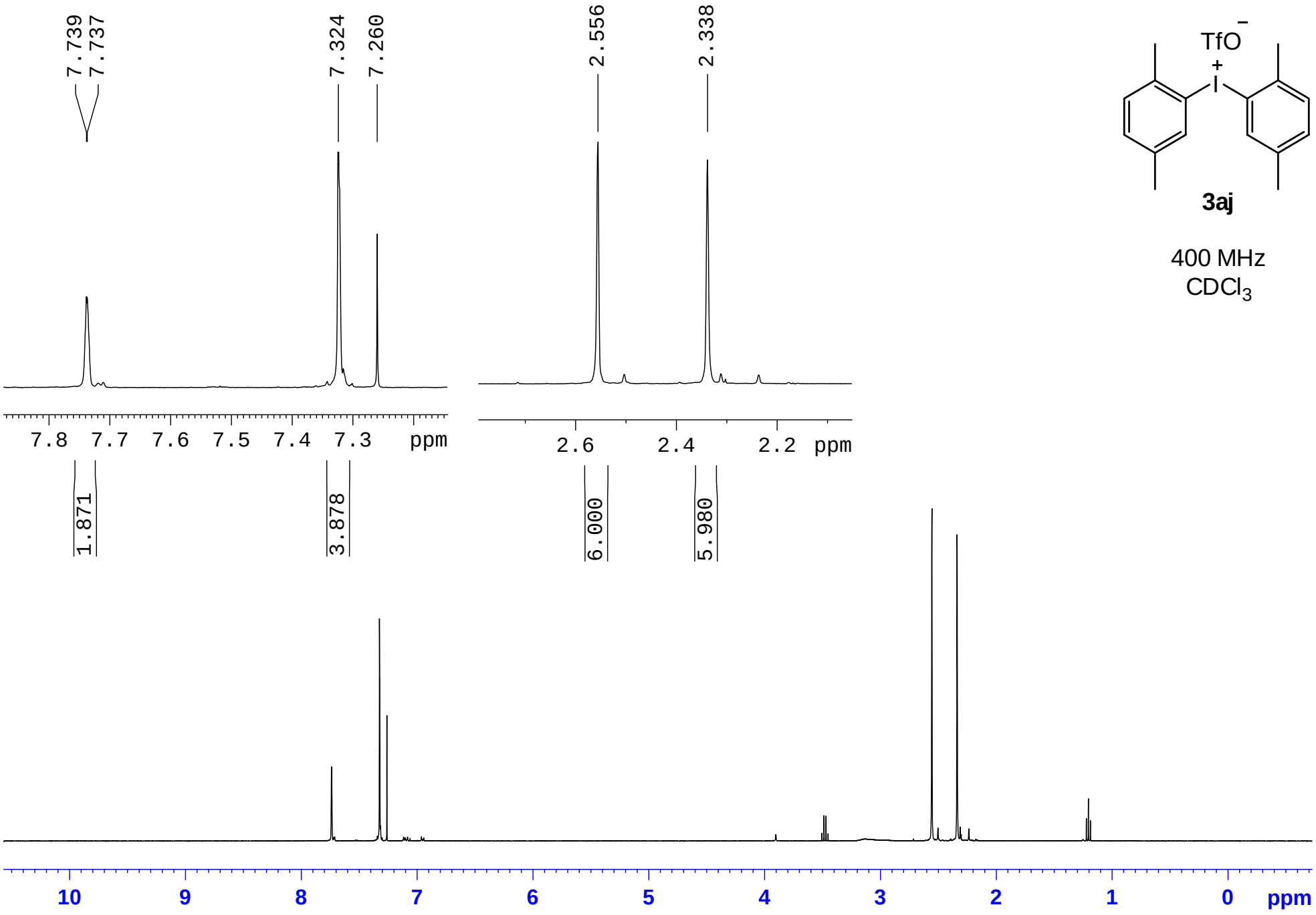
400 MHz
CD_3OD

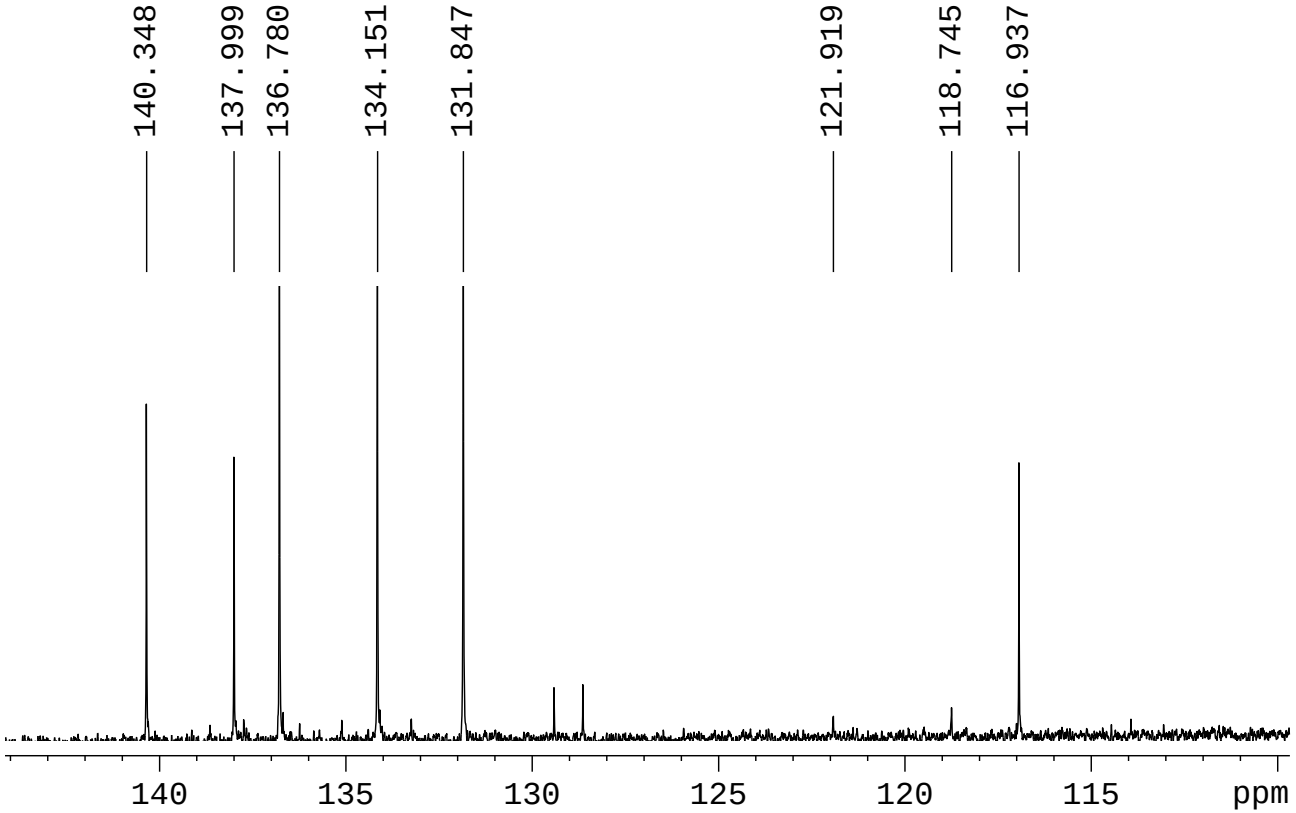


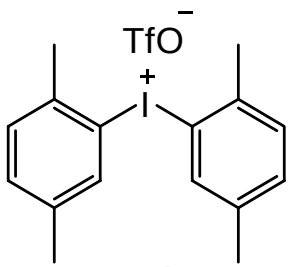

167.705
165.175
139.226
123.367
120.697
120.465
120.203
110.237
110.213
170
165
160
155
150
145
140
135
130
125
120
115
110
105
ppm
TfO
I
F
F
3ah
100 MHz
CD3OD
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


7.907
7.885
7.465
7.443
1.292
8.0
7.5
ppm
1.4
1.3
1.2
ppm
4.000
3.984
18.371
TfO
I
tBu
tBu
3ai
100 MHz
CDCl3
10
9
8
7
6
5
4
3
2
1
0
ppm


156.518
134.886
129.556
121.813
118.633
109.433
77.317
77.000
76.681
35.193
30.940
TfO
I
tBu
tBu
3ai
100 MHz
CDCl3
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

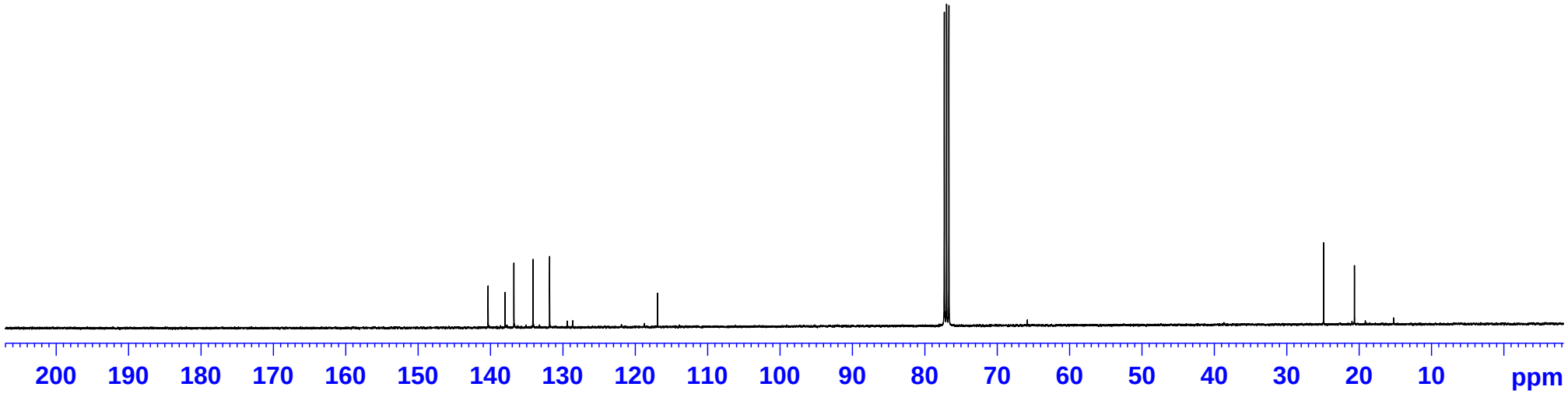

7.739
7.737
7.324
7.260
2.556
2.338
7.8
7.7
7.6
7.5
7.4
7.3
ppm
2.6
2.4
2.2
ppm
1.871
3.878
6.000
5.980
TfO
I
3aj
400 MHz
CDCl3
10
9
8
7
6
5
4
3
2
1
0
ppm

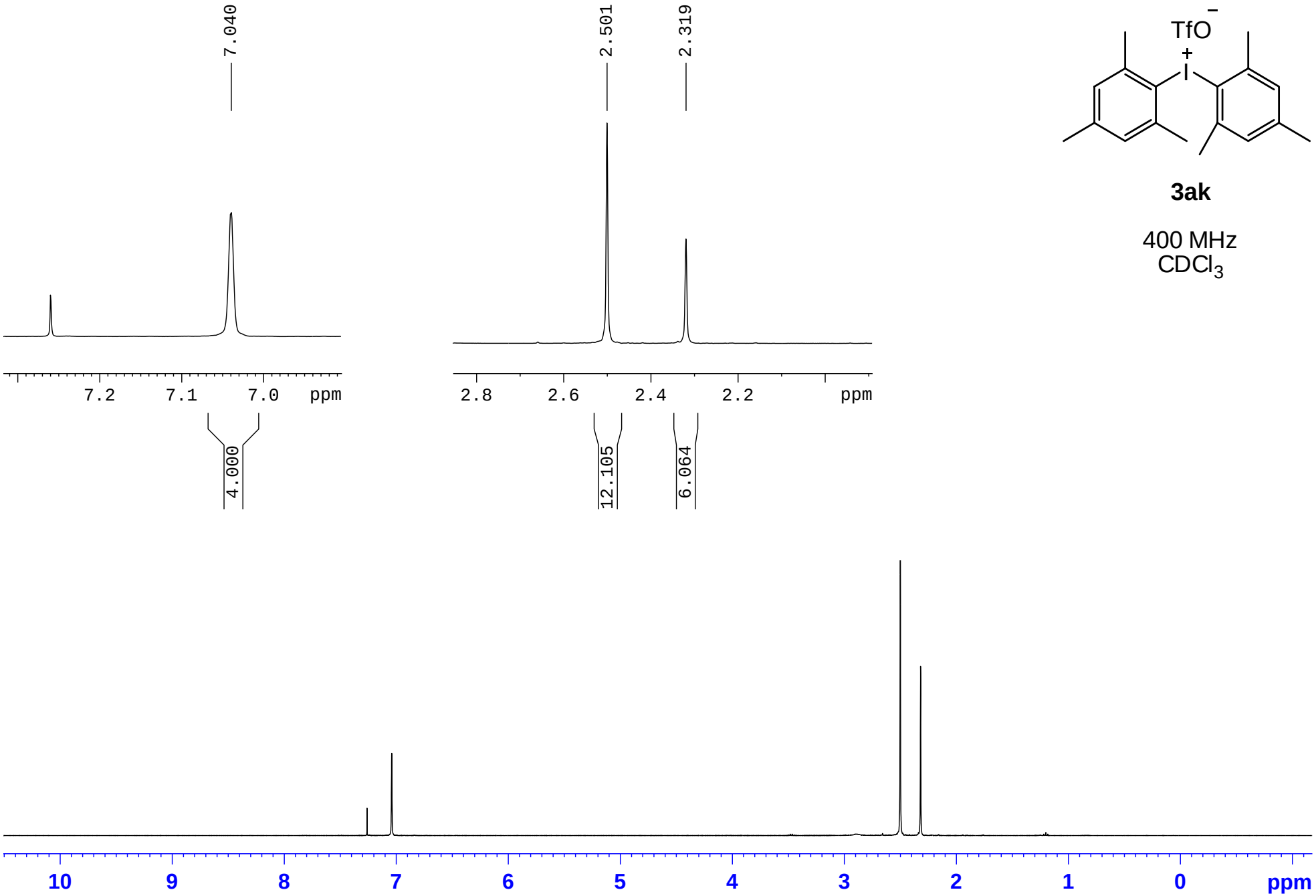


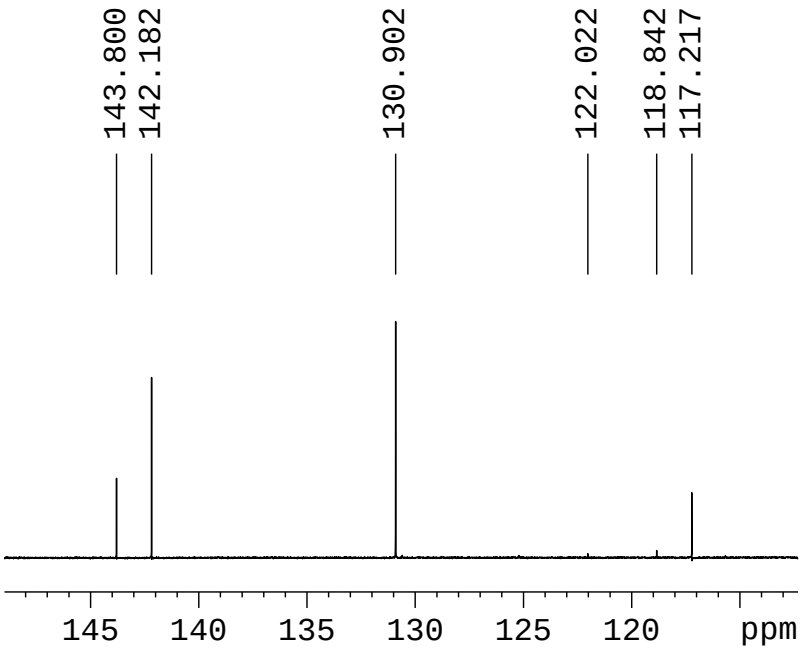


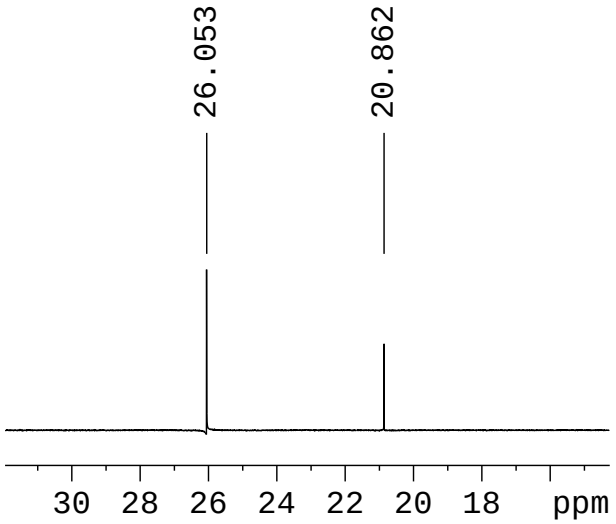
3aj

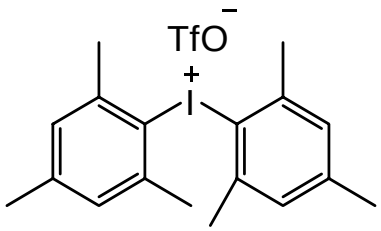
100 MHz
$CDCl_3$




7.040
2.501
2.319
TfO
I
3ak
400 MHz
CDCl3
7.2
7.1
7.0
ppm
2.8
2.6
2.4
2.2
ppm
4.000
12.105
6.064
10
9
8
7
6
5
4
3
2
1
0
ppm

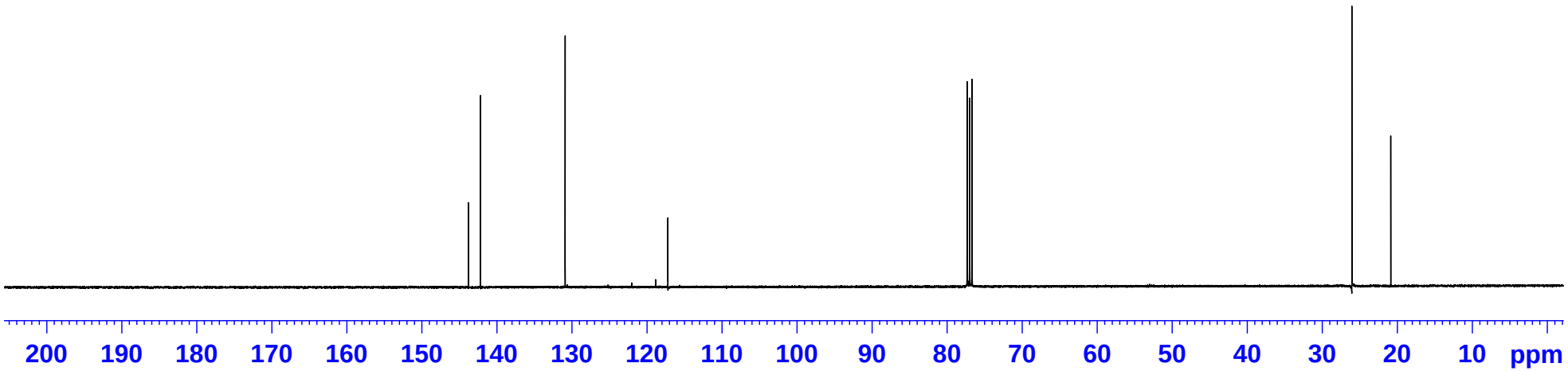


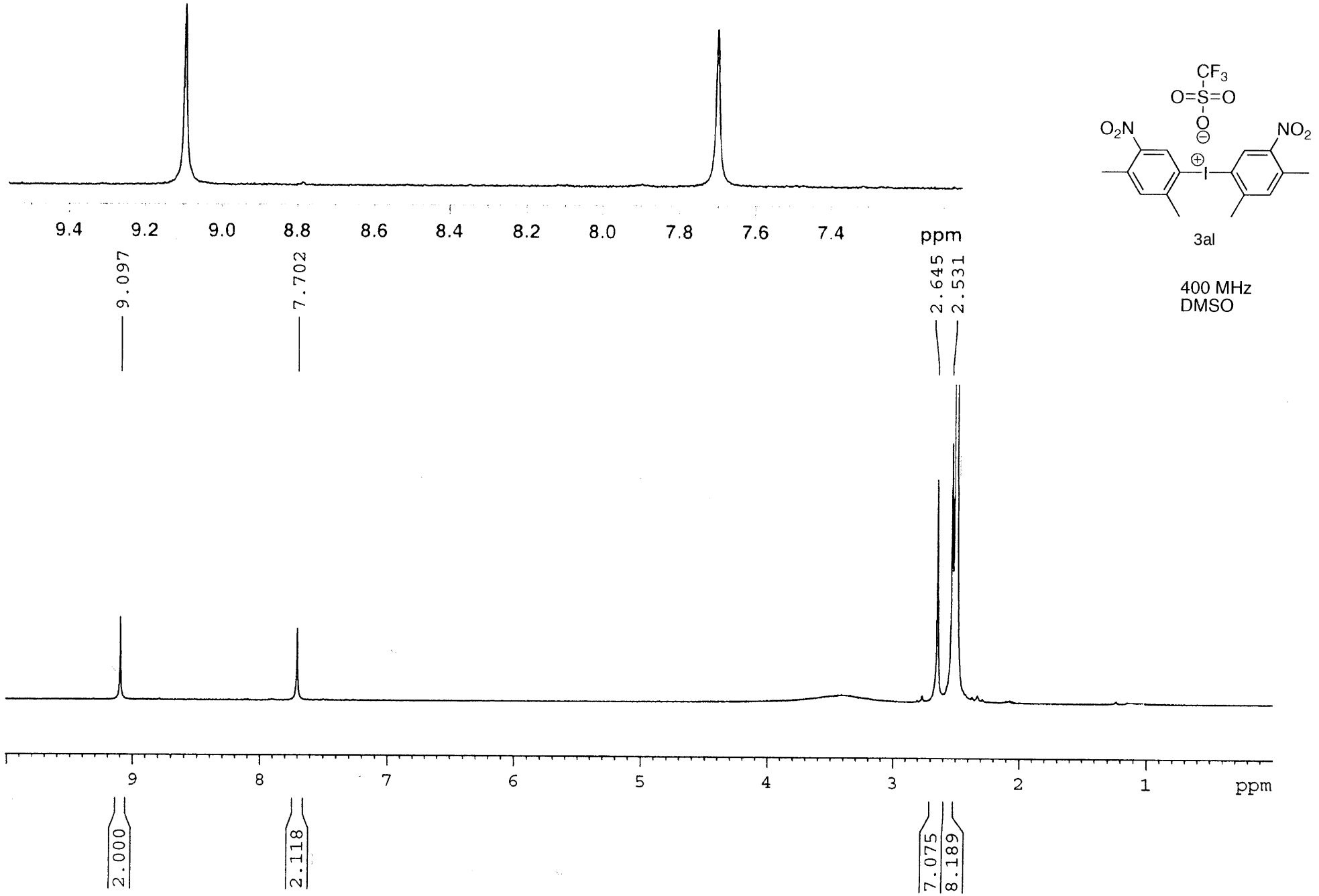




3ak

100 MHz
$CDCl_3$





3al

400 MHz
DMSO