Electronic Supplementary Information for

Refining boron-iodane exchange to access versatile arylation reagents

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1. General Considerations

Materials and methods: Commercially available reagents and solvents were used without further purification. All other materials were prepared as described in detail below. Reactions performed above ambient room temperature were done so in an oil bath or aluminium block heated externally. Crude reaction mixtures were analyzed by ^{1}H-NMR spectroscopy in presence of internal standard. ^{1}H, ^{13}C{^{1}H}, ^{19}F{^{1}H} NMR spectra were obtained at 298 K in DMSO-d_6 on Bruker Avance 400 MHz or Bruker Avance 600 MHz spectrometer and referenced to residual solvent peak (2.5 ppm) or tetramethylsilane when applicable. Residual water from the solvent (DMSO-d_6) is observed in all ^{1}H-NMR spectra at 3.33 ppm. The following notation is used while analyzing NMR data: s – singlet, d – doublet, dd – doublet of doublets, ddd – doublet of doublet of doublets, t – triplet, q – quartet, n – nonet, br – broad signal. High resolution mass spectrometry (HRMS) data were recorded on Thermo Scientific Q-exactive mass spectrometer by electrospray ionization with an Orbitrap mass-analyzer (ESIOrbitrap). FTIR spectra were recorded on Thermo Scientific Nicolet iS5 Infra-red spectrometer. Melting points(°C) were obtained on the Stuart SMP10 melting point apparatus or the Mel-Temp Electrothermal melting point apparatus are uncorrected.

%Purity of the synthesized aryl(Mes)iodonium salts was calculated by quantitative NMR according to the protocol as described on the by *Organic Syntheses* author instruction guidelines (http://www.orgsyn.org/instructions.aspx). The procedure is described as below:

Accurately weigh 10-20 mg of the desired compound and approximately equimolar amount of the internal standard (at least 10 mg) in a clean glass container. This mixture is dissolved in approximately 1 mL of DMSO-d_6. Proton NMR is obtained for the mixture with a minimum relaxation delay of 30s between the scans. The weight percent purity is calculated using the following equation:

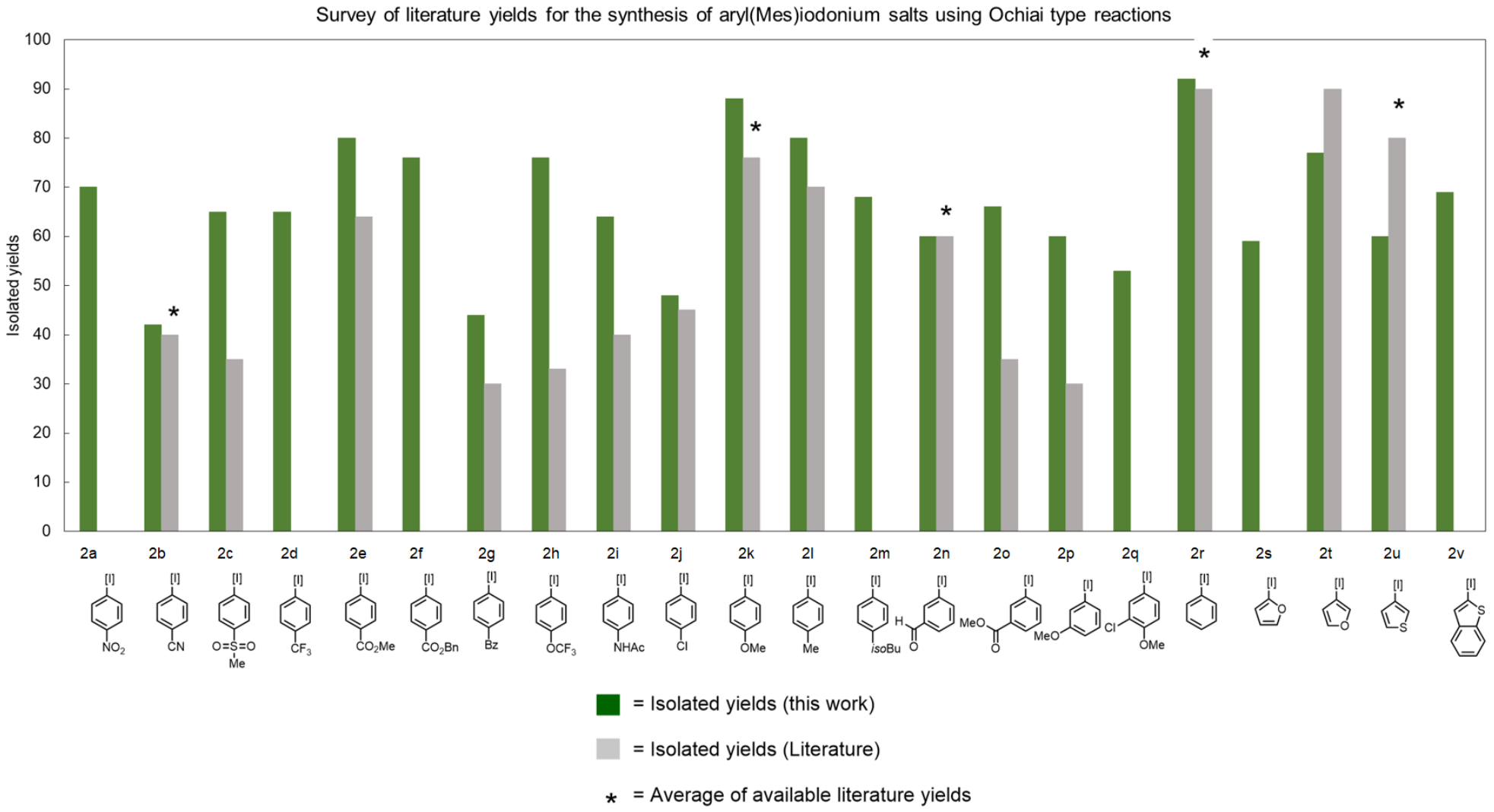
$$\text{Molar ratio} = \frac{\left[\frac{I_{cpd}}{nH_{cpd}}\right]}{\left[\frac{I_{std}}{nH_{std}}\right]}$$

$$\text{wt\%} = \frac{mg_{std} \times MW_{cpd} \times \text{Molar ratio} \times P_{std}}{mg_{cpd} \times MW_{std}} \times 100$$

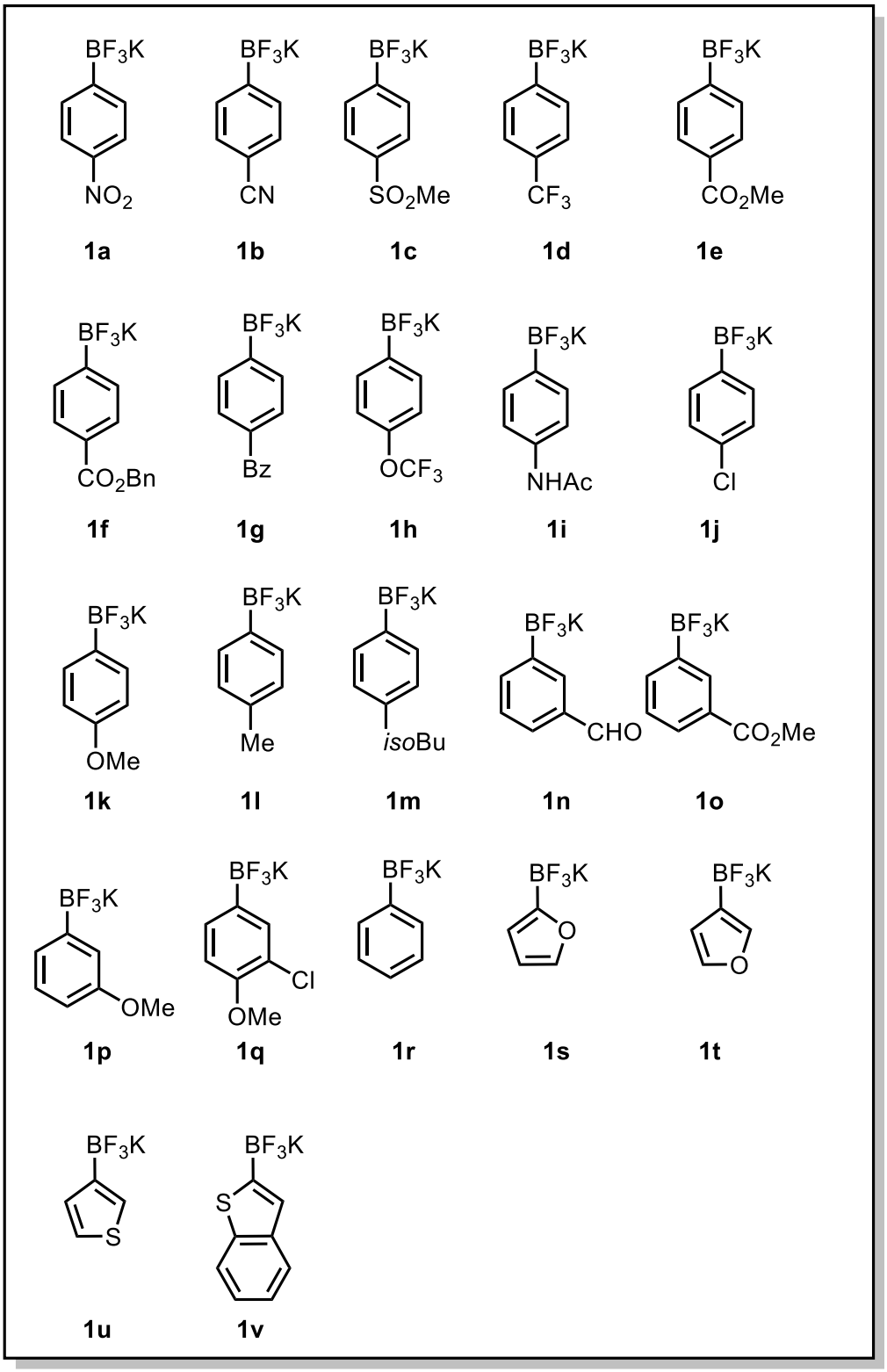
Where,

wt% = Purity of the sample
I_{cpd} = Proton integral area of a known peak on the compound being analyzed
nH_{cpd} = Number of hydrogens associated with the compound NMR peak
I_{std} = Proton integral area of a known peak on the internal standard
nH_{std} = Number of hydrogens associated with the internal standrd NMR peak
mg_{std} = Amount of intenral standard used for analysis (in milligrams)
mg_{cpd} = Amount of compound used for analysis (in milligrams)
MW_{std} = Molecular weight of the the internal standard
MW_{cpd} = Molecular weight of the compound being analyzed
P_{std} = wt% purity of the internal standard expressed as decimal value (usually 1.00)

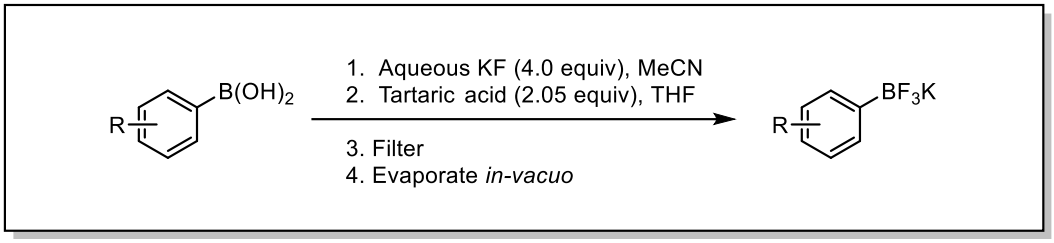
2. Comparison of yields from current work with literature yields



3. List of the potassium aryltrifluoroborate salts used in this work



4. Synthesis of potassium aryltrifluoroborate salts from arylboronic acids (General procedure A) and their characterization data.

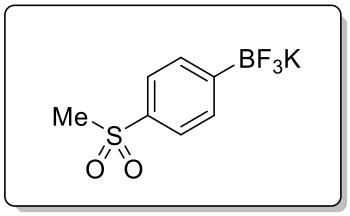


Synthesis of potassium aryltrifluoroborate salts was carried out using the protocol reported by Lennox et al (*Angew. Chem. Int. Ed. 2012, 51, 9385 –9388*).[1]

Arylboronic acid (1 mmol) was suspended in acetonitrile (4 mL). To this suspension potassium fluoride (4 equiv., 4 mmol, 232 mg) dissolved in H_2O (0.4 mL) was added at room temperature. In most cases, the solution becomes clear after adding KF within 2-5 mins (1-2 mL MeOH was added if clear solution doesn't appear). To this rapidly stirring biphasic mixture was added tartaric acid (2.05 equiv., 2.05 mmol, 308 mg) dissolved in THF over 1 minute (the solution was warmed to dissolve tartaric acid in THF). White precipitate forms as tartaric acid solution is added to the mixture. The reaction was allowed to stir for 10 mins at room temperature after which the mixture was diluted with 5 mL acetonitrile and filtered. The filter cake was rinsed with acetonitrile (3 x 5 mL). The filtrate was concentrated *in-vacuo* to give the crude solid which was co-evaporated using toluene (2 x 5mL) to remove residual water to give the corresponding potassium aryltrifluoroborate salt.

Compounds **1a**,**1b**, and **1l** were prepared according to the literature procedure above and the analytical data is in agreement with that previously reported.[1]

Compound 1c



Prepared from 4-(methanesulfonyl)phenylboronic acid using the general procedure A and isolated as a white crystalline solid in 54% yield.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.65 (d, *J* = 7.6 Hz, 2H), 7.56 (d, *J* = 7.7 Hz), 3.10 (s, 3H) ppm.

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 138.1, 132.3, 125.2, 44.4 ppm, C-B signal not observed due to quadrupolar relaxation.

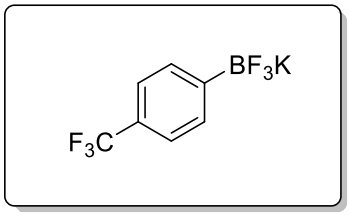
^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -140.1 ppm

HRMS: Calculated for $C_7H_7BF_3O_2S$ $[M\text{-}K]^-$: 223.02119, found 223.02112

FT-IR: 3019, 1385, 1323, 1291, 1205, 1146, 1086, 1035, 976, 944(broad), 929, 859, 830, 733, 632 cm^{-1}

Melting point: 310-315 °C

Compound 1d



Prepared from 4-trifluomethylphenylboronic acid using the general procedure A and isolated as a while solid in 80% yield.

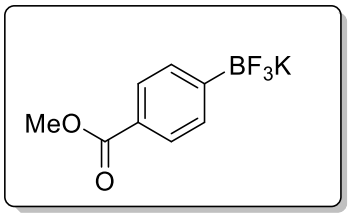
^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.53 (d, *J* = 7.6 Hz), 7.42 (d, *J* = 7.6 Hz).

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 130.6, 124.7 (q, $^{2}J_{C\text{-}F}$ = 40 Hz), 123.9 (q, $^{1}J_{C\text{-}F}$ = 272 Hz), 121.6, C-B signal not observed due to quadrupolar relaxation.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -60.4, -139.9

The analytical data are in accordance with the previously reported literature.[2]

Compound 1e



Prepared from 4-methoxycarbonylphenylboronic acid using the general procedure A and isolated as a while solid in 47% yield.

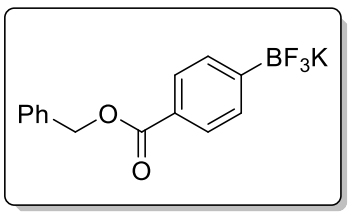
^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.73 (d, *J* = 7.6 Hz), 7.44 (d, *J* = 7.6 Hz), 3.80 (s, 3H) ppm

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 167.6, 131.9, 127.7, 126.9, 52.1 ppm; C-B signal not observed due to quadrupolar relaxation.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -139.9 ppm

The analytical data are in accordance with the previously reported literature.[3]

Compound 1f



Prepared from 4-((benzyloxy)carbonyl)phenylboronic acid using the general procedure A and isolated as a white solid in 86% yield.

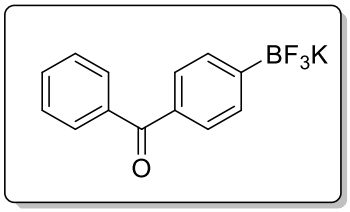
^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.76 (d, *J* = 7.6 Hz, 2H), 7.50-7.31 (m, 7H), 5.31 (s, 2H) ppm.

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 166.9, 137.0, 132.0, 129.0, 128.5, 128.3, 127.8, 126.8, 66.0 ppm. C-B signal not observed due to quadrupolar relaxation.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -139.8 ppm.

The analytical data are in accordance with the previously reported literature.[4]

Compound 1g



Prepared from 4-Benzylphenylboronic acid using the general procedure A and isolated as a fluffy white solid in 80% yield.

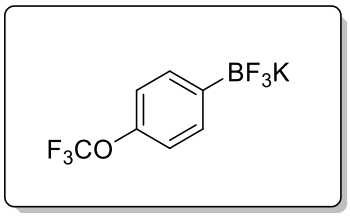
^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.71 (d, *J* = 7.7 Hz, 2H), 7.64 (t, *J* = 7.7 Hz, 1H), 7.58-7.48 (m, 6H), ppm.

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 194.7, 136.3, 132.3, 130.4, 129.6, 127.7, 126.7, 126.4, ppm. C-B signal not observed due to quadrupolar relaxation.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -139.8 ppm.

The analytical data are in accordance with the previously reported literature.[5]

Compound 1h



Prepared from 4-trifluoromethoxyphenylboronic acid using the general procedure A and isolated as a white solid in 98% yield.

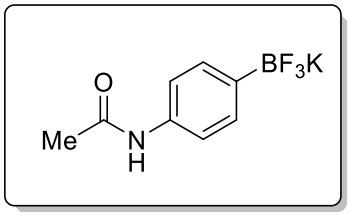
^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.41 (d, *J* = 7.84 Hz, 2H), 7.04 (d, *J* = 7.80 Hz, 2H), ppm.

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 145.0, 131.1, 118.6 (q, $^{1}J_{C\text{-}F}$ = 255 Hz), 117.1, ppm. C-B signal not observed due to quadrupolar relaxation.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -56.6, -139.3 ppm.

The analytical data are in accordance with the previously reported literature.[6]

Compound 1i



Prepared from 4-acetamidophenylboronic acid using the general procedure A and isolated as a white solid in 80% yield.

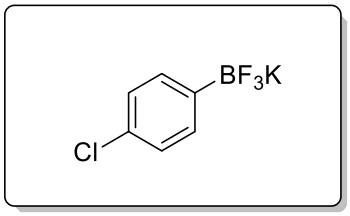
^{1}H NMR (400 MHz, [D6] DMSO): δ = 9.58 (s, 1H), 7.28 (d, *J* = 7.8 Hz, 2H), 7.21(d, *J* =7.8 Hz, 2H), 2.95 (s, 3H) ppm.

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 166.0, 134.8, 129.7, 115.9, 22.3, ppm. C-B signal not observed due to quadrupolar relaxation.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -138.6 ppm.

The analytical data are in accordance with the previously reported literature.[7]

Compound 1j



Prepared from 4-chlorophenylboronic acid using the general procedure A and isolated as a white solid in 98% yield.

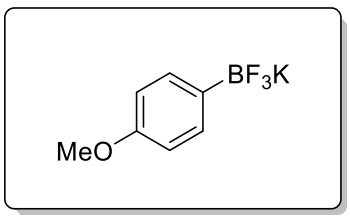
^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.32 (d, *J* = 7.7 Hz, 2H), 7.11 (d, *J* = 7.7 Hz, 2H) ppm.

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 133.6, 130.2, 126.6 ppm.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -139.3 ppm.

The analytical data are in accordance with the previously reported literature.[8]

Compound 1k



Prepared from 4-methoxyphenylboronic acid using the general procedure A and isolated as shiny crystalline solid in 84% yield.

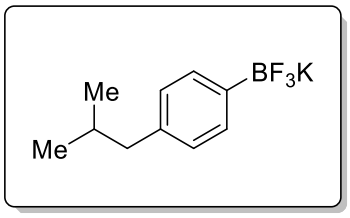
^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.22 (d, *J* = 8.0 Hz, 2H), 6.66 (d, *J* = 8.0 Hz, 2H), 3.66 (s, 3H) ppm.

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 157.7, 132.7, 112.3, 55.0, ppm. C-B signal not observed due to quadrupolar relaxation.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -138.2 ppm.

The analytical data are in accordance with the previously reported literature.[5]

Compound 1m



Prepared from 4-isobutylphenylboronic acid using the general procedure A and isolated as a white solid in 88% yield.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.22 (d, *J* = 7.3 Hz, 2H), 6.86 (d, *J* = 7.4 Hz, 2H), 2.22 (d, *J* = 7.0 Hz, 2H), 1.77 (m, 1H), 0.84 (d, *J* = 6.5 Hz, 6H) ppm.

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 137.6, 131.6, 127.4, 45.3, 30.3, 22.7, ppm. C-B signal not observed due to quadrupolar relaxation.

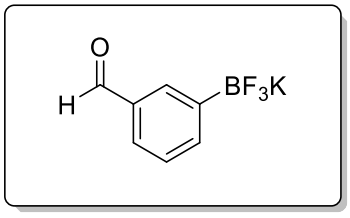
^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -138.6 ppm.

HRMS = Calculated for $C_{10}H_{13}BF_3^-$ [M-K]$^-$: 201.10624, found: 201.10605

FT-IR: 3009, 2951, 2922, 2866, 2843, 1601, 1465, 1396, 1381, 1223, 1194, 1053, 949(broad), 880, 790 cm^{-1}

Melting point: 287-300 °C

Compound 1n



Prepared from 3-formylphenylboronic acid using the general procedure A and isolated as an off-white solid in 95% yield.

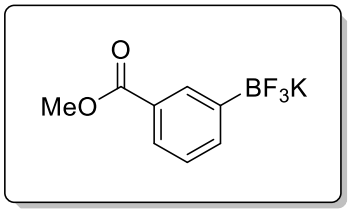
^{1}H NMR (400 MHz, [D6] DMSO): δ = 9.95 (s, 1H), 7.88 (s, 1H), 7.67 (d, *J* = 7.0 Hz, 1H), 7.60 (d, *J* = 7.5 Hz, 1H), 7.33 (t, *J* = 7.3 Hz, 1H) ppm.

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 194.7, 138.4, 135.2, 133.8, 127.6, 126.7 ppm. C-B signal not observed due to quadrupolar relaxation.

$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): δ = -139.7 ppm.

The analytical data are in accordance with the previously reported literature.[8]

Compound 1o



Prepared from 3-methoxycarbonylphenylboronic acid using the general procedure A and isolated as a white solid in 76% yield.

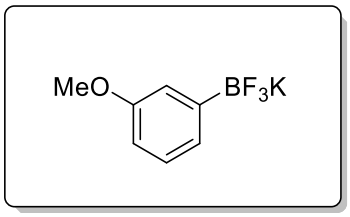
^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.99 (s, 1H), 7.67 (d, *J* = 7.6 Hz, 1H), 7.6 (d, *J* = 7.2 Hz, 1H), 7.23 (t, *J* = 7.4 H, 1H), 3.81 (s, 3H) ppm.

$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): δ = 167.9, 136.8, 132.8, 128.0, 127.0, 126.6, 52.1 ppm. C-B signal not observed due to quadrupolar relaxation.

$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): δ = -139.6 ppm.

The analytical data are in accordance with the previously reported literature.[2]

Compound 1p



Prepared from 3-methoxyphenylboronic acid using the general procedure A and isolated as a white solid in 86% yield.

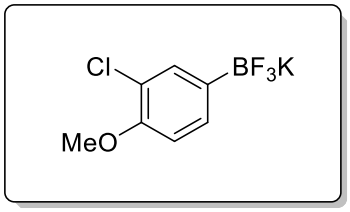
^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.01 (t, 7.4 Hz, 1H), 6.94-6.82 (m, 2H), 6.58 (d, *J* = 3.8 Hz, 1H) ppm.

$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): δ = 158.5, 127.6, 124.3, 116.7, 111.1, 54.9 ppm. C-B signal not observed due to quadrupolar relaxation.

$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): δ = -139.1 ppm.

The analytical data are in accordance with the previously reported literature.[8]

Compound 1q



Prepared from 3-chloro-4-methoxyphenylboronic acid using the general procedure A and isolated as a white solid in 90% yield.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.22 (s, 1H), 7.20 (d, *J* = 8.0 Hz, 1Hz), 6.87 (d, *J* = 7.9 Hz, 1H), 3.76 (s, 3H) ppm.

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 150.6, 130.9, 129.2, 117.8, 116.4, 109.7, 54.9 ppm. C-B signal not observed due to quadrupolar relaxation.

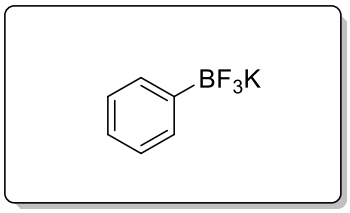
^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -138.9 ppm.

HRMS = Calculated for $C_7H_6BClF_3O^-$ [M-K]$^{-:}$ 209.01523; found 209.01520

Melting point: 237-241 °C

The analytical data are in accordance with the previously reported literature.[9]

Compound 1r



Prepared from phenylboronic acid using the general procedure A and isolated as a crystalline solid in 92% yield.

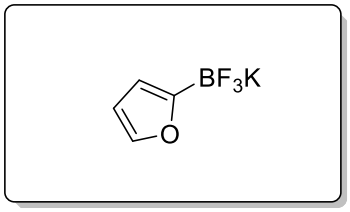
^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.32 (d, *J* = 7.0 Hz, 2H), 7.11-6.96 (m, 3H) ppm.

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 131.8, 126.7, 125.4 ppm. C-B signal not observed due to quadrupolar relaxation.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -139.1 ppm.

The analytical data are in accordance with the previously reported literature.[10]

Compound 1s



Prepared from Furan-2-ylboronic acid using the general procedure A and isolated as a crystalline solid in 84% yield

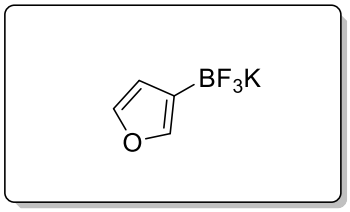
^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.39 (m, 1H), 6.15 (m, 1H), 5.94 (m, 1H) ppm.

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 141.5, 110.6, 108.9 ppm.C-B signal not observed due to quadrupolar relaxation.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -138.5 ppm.

The analytical data are in accordance with the previously reported literature.[11]

Compound 1t



Prepared from furan-3-ylboronic acid using the general procedure A and isolated as a crystalline solid in 87% yield.

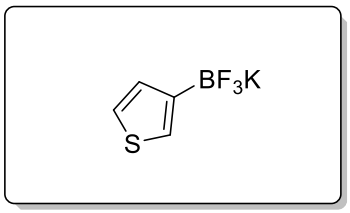
^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.35 (m, 1H), 7.07 (m, 1H), 6.18 (m, 1H) ppm.

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 143.1, 130.9, 114.2 ppm. C-B signal not observed due to quadrupolar relaxation.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -134.5 ppm.

The analytical data are in accordance with the previously reported literature.[3]

Compound 1u



Prepared from Thiophen-3-ylboronic acid using the general procedure A and isolated as a crystalline solid in 81% yield.

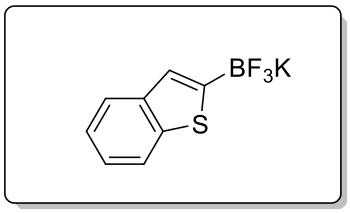
^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.17 (m, 1H), 7.03-7.93 (m, 2H) ppm.

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 132.3, 124.8, 123.0 ppm. C-B signal not observed due to quadrupolar relaxation.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -135.5 ppm.

The analytical data are in accordance with the previously reported literature.[3]

Compound 1v



Prepared from Benzo[b]thiophen-2-ylboronic acid using the general procedure A and isolated as a crystalline solid in 78% yield.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.79 (d, *J* = 7.8 Hz, 1H), 7.67(d, *J* =7.8 Hz, 1H), 7.21 (t, *J* = 7.2 Hz, 1H), 7.14 (t, *J* = 7.2 Hz, 1H), 7.09 (s, 1H) ppm.

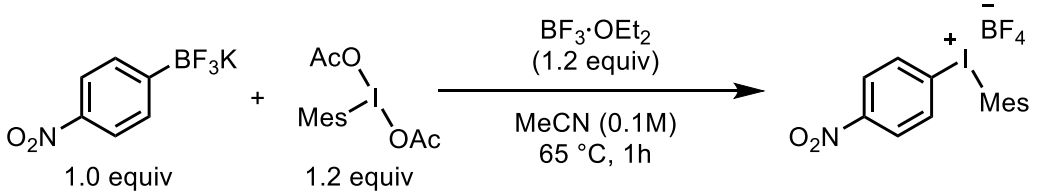
^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 141.9, 141.5, 123.8, 123.4, 122.7, 122.5, 122.3 ppm. C-B signal not observed due to quadrupolar relaxation.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -134.9 ppm.

The analytical data are in accordance with the previously reported literature.[11]

5. Optimization of Aryl (Mes)iodonium salts synthesis from potassium aryltrifluoroborates and Iodomesitylene diacetate

To an oven dried 4 mL vial equipped with a stir bar was charged with potassium aryltrifluoroborate salt (1.0 equiv, 0.1 mmol) and iodomesitylene diacetate (1.2 equiv, 0.12 mmol). This mixture was suspended in MeCN (1 mL, 0.1 M) and $BF_3.OEt_2$ (0.12 equiv, 0.12 mmol) was added dropwise. The vial was sealed and transferred to a pre-heated aluminium block at 65 ºC and stirred at that temperature for 1hour. The crude reaction mixture was concentrated under-vacuum and internal standard (ethylene carbonate was added) to the mixture. This mixture was dissolved in DMSO-d_6 and analyzed by ^{1}H-NMR spectroscopy.

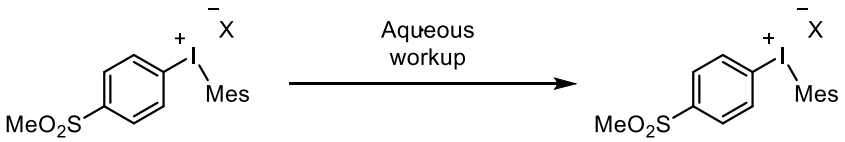


a = NMR yields; b = isolated yields

Entry	***Deviations from conditions***	***%Yield[a]***
1	None	85%
2	-$B(OH)_2$ and DCM, RT, 24h	21%[b]
3	-$B(OH)_2$ and DCM, RT	11%[b]
4	-BPin and DCM, RT	< 5%[b]
5	DCM, RT	56%[b]
6	MeCN, RT	52%
7	TMS-OTf	70%
8	TES-OTf	71%
9	TIPS-OTf	67%
10	TBDMS-OTf	75%
11	$Yb(OTf)_3$	*trace*
12	$Ti(OEt)_4$	*trace*
13	TsOH	35%
14	TfOH	53%

6. Recovery studies from liquid-liquid extraction

To test the water-solubility of iodonium salts during liquid-liquid extraction as a function of counter anion, we synthesized three distinct iodonium salts where counter anions were BF_4, OTs and OTf. 0.5 mmol of pure salt was taken up in a separating funnel and 25 mL of water was added. The salt was extracted with DCM (3x15 mL). The organic layer was dried with Na_2SO_4 and concentrated *in-vacuo* to afford a crude oil which was triturated with Et_2O. The white precipitates were filtered, and recovery was calculated.

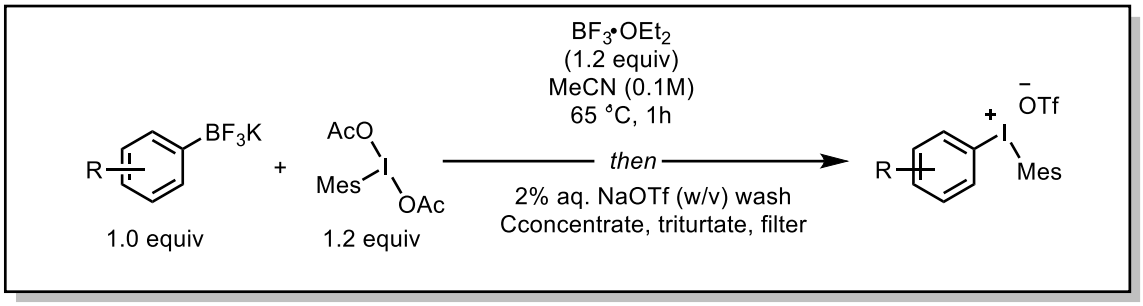


X = BF_4, OTs, OTf

Entry	*Counteranion (X)*	*%Recovery*[a]
1	BF_4	20%
2	OTs	67%
3	OTf	82%

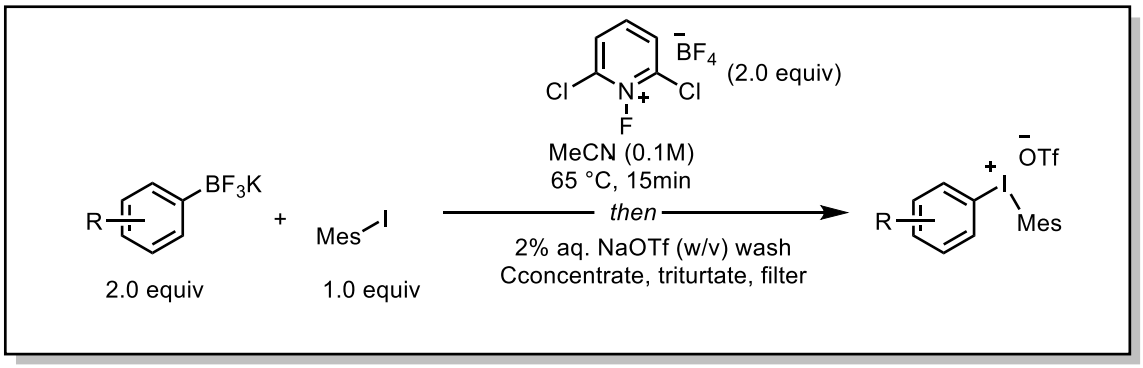
7. Syntheses of aryl (Mes)iodonium salts

7.1. Synthesis of aryl(Mes)iodonium salts from potassium aryltrifluoroborates and iodomesitylene diacetate **(General procedure B)**



An Oven-dried 12 mL glass vial with stir bar was charged with potassium aryltrifluoroborate salt (0.5 mmol, 1.0 equiv) and iodomesitylene diacetate (0.6 mmol, 1.2 equiv) and acetonitrile (5 mL, 0.1 M). To this mixture, BF_3•OEt_2 (0.075 mL, 1.2 equiv) was added dropwise under stirring. The solution shows intermittent blue-green colouration and turns yellow over a period of one minute. The vial was transferred to a preheated block at 65 °C, and the mixture was stirred at the same temperature for 1 hour. The solvent was evaporated in-vacuo and the crude residue was transferred to a separatory funnel using DCM (10 mL x 3). The organic layer was washed with 2% (w/v) aqueous sodium trifluoromethanesulfonate solution (10 mL x 3). The organic phase was dried over sodium sulfate and concentrated in-vacuo. The crude was dissolved in minimum amount of DCM and was triturated in diethyl ether and the resulting solid was filtered and weighed.

7.2. Synthesis of aryl(Mes)iodonium salts from potassium aryltrifluoroborates and 2-iodomesitylene **(General procedure C)**

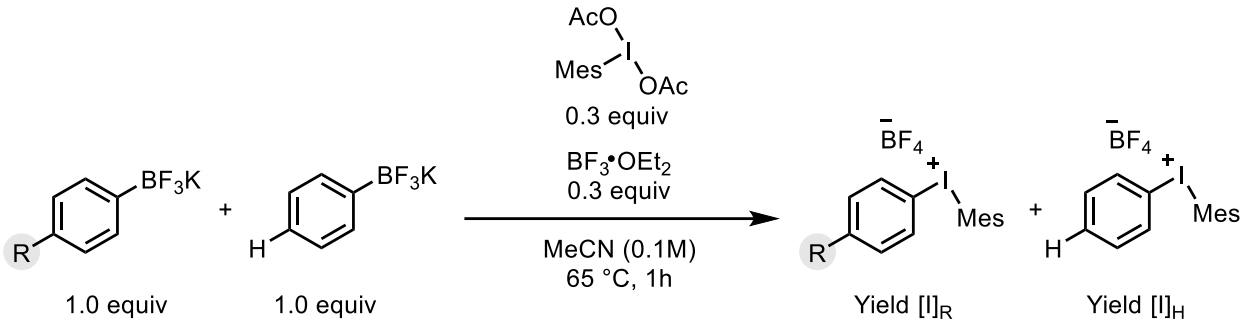


An oven-dried 12 mL glass vial with stir bar was charged with potassium aryltrifluoroborate salt (1.0 mmol, 2.0 equiv), iodomesitylene (0.5 mmol, 1.0 equiv) and acetonitrile (5 mL, 0.1 M). To this mixture, 2,6-dichloropyridinium tetrafluoroborate (1.0 mmol, 2.0 equiv) was added in portions under stirring. The vial was transferred to a preheated block at 65 °C and heated at the same temperature for 15 minutes. The solvent was evaporated in-vacuo and the crude residue was transferred to a separatory funnel using DCM (10 mL x 3). The organic layer was washed with 2% (w/v) aqueous sodium trifluoromethanesulfonate solution (10 mL x 3). The organic phase was dried over sodium sulfate and concentrated in-vacuo. The crude was dissolved in minimum amount of DCM and was triturated in diethyl ether and the resulting solid was filtered and weighed.

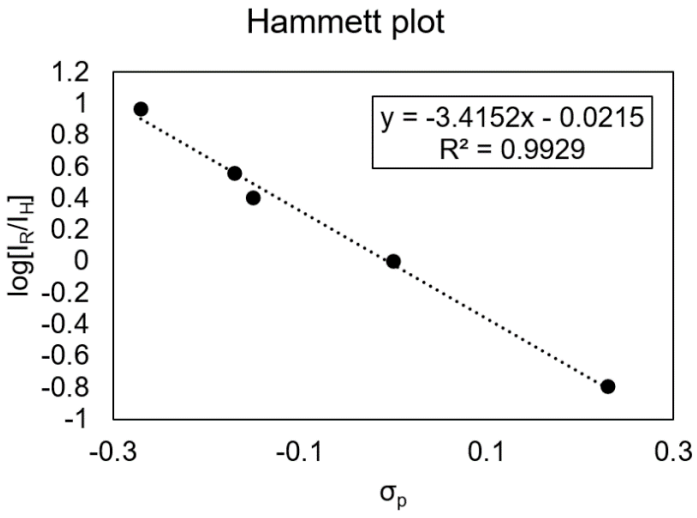
8. **Mechanistic Studies:** Boron-Iodane exchange Hammett plot using potassium aryltrifluoroborates and Iodomesitylene diacetate

Evaluating the electronic effects on the aryl ring using Hammett plot.

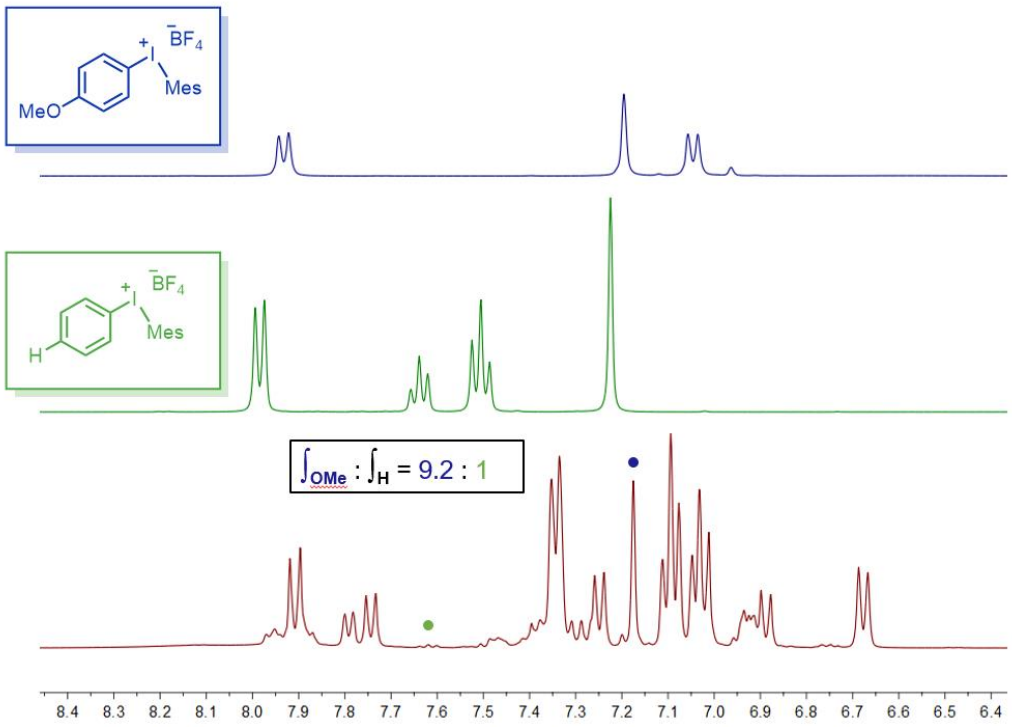
In a flame dried 5 mL vial equipped with stir bar was charged with potassium phenyltrifluoroborate (0.1 mmol, 1.0 equiv), 4-substituted potassium aryltrifluoroborate (0.1 mmol, 1.0 equiv), and iodomesiylene diacetate (0.03 mmol, 0.3 equiv). The components were suspended in acetonitrile (1 ml, 0.1 M). To this stirred suspension, BF_3•OEt_2 (0.03 mmol, 0.3 equiv) was added under stirring at room temperature. The reaction was sealed with screw cap and was placed in a preheated heating block at 65 °C for 1 hour while stirring. After one hour, the solvent was evaporated in-vacuo and the crude was dissolved in DMSO-d_6 and analyzed by ^{1}H NMR spectroscopy.



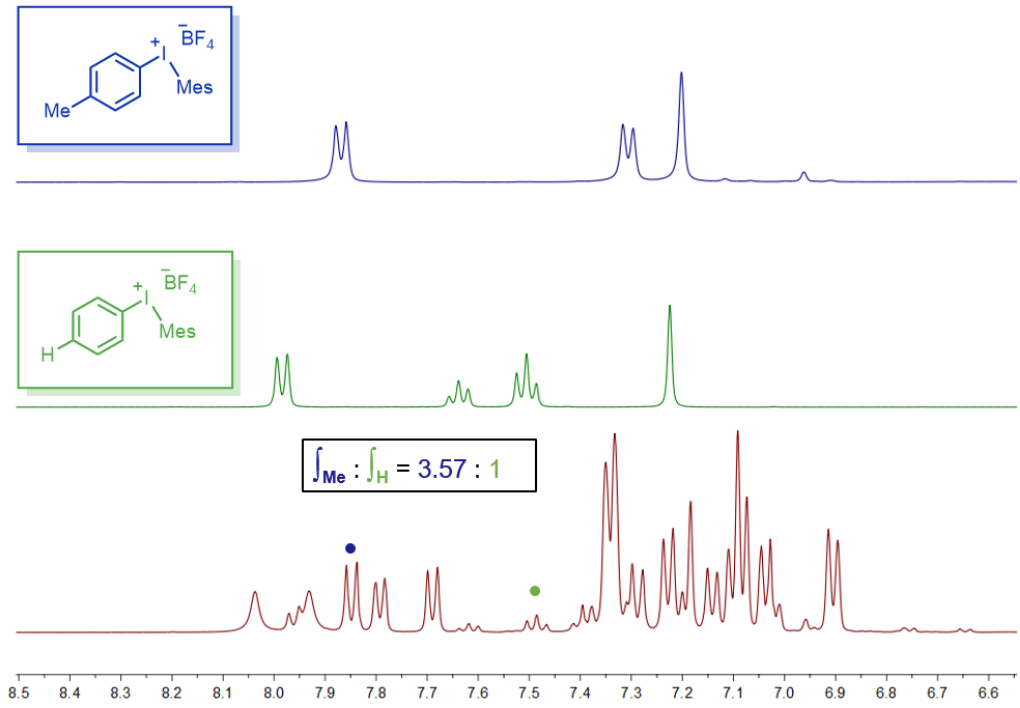
Entry	R	σ_p	Yield $[I]_R$	Yield $[I]_H$	$\log\left[\frac{[I]_R}{[I]_H}\right]$
1	-OMe	-0.27	9.20	1.00	0.963788
2	-Me	-0.17	3.57	1.00	0.552668
3	-*iso*Bu	-0.15	2.52	1.00	0.401401
4	-H	0.00	1.00	1.00	0
5	-Cl	0.24	0.16	1.00	-0.795880



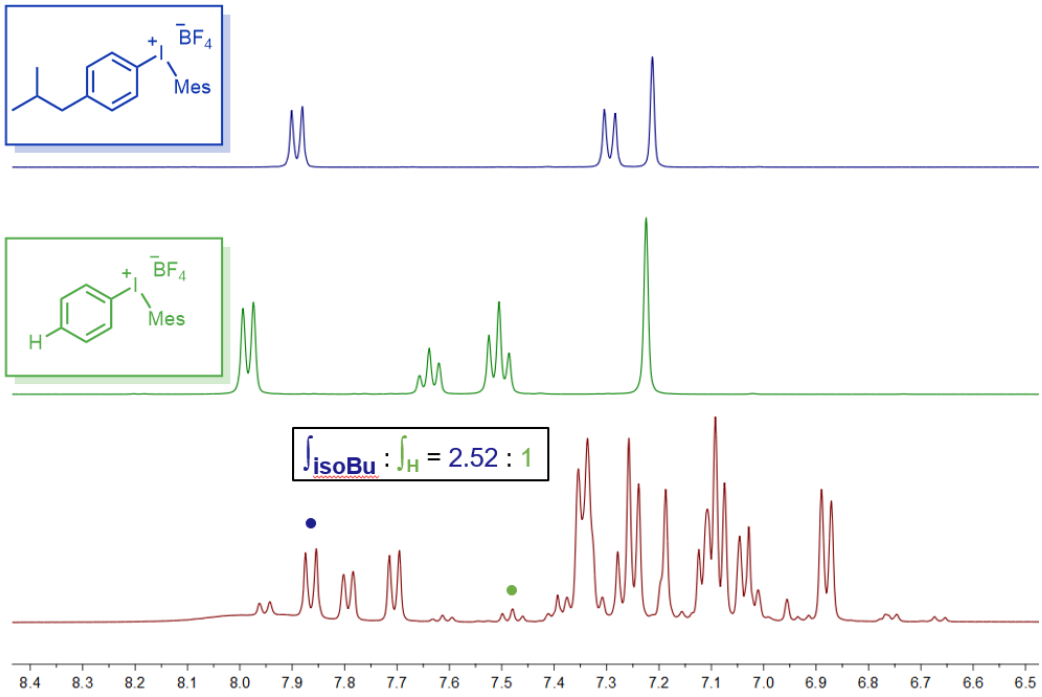
Experiment 1 (OMe vs H)



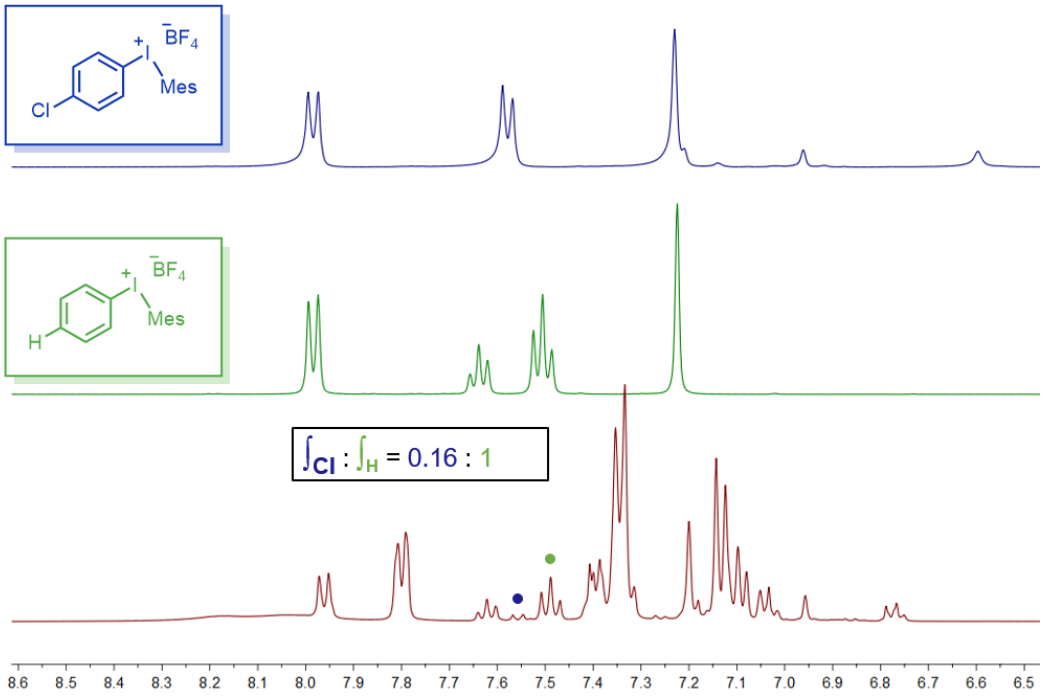
Experiment 2 (Me vs H)



Experiment 3 (*iso*Bu vs H)

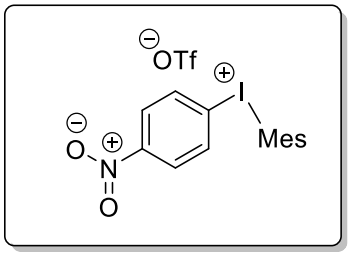


Experiment 4 (Cl vs H)



9. Characterization of aryl (Mes)iodonium salts

Compound 2a



Synthesized using the general procedure B on 0.5 mmol scale and isolated as a pale-yellow solid in 70% yield (0.1815 g, 0.35 mmol). The NMR yield for corresponding reaction that generates the tetrafluoroborate salt was 85%.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 8.26 (d, *J* = 8.8 Hz, 2H), 8.16 (d, *J* = 8.8 Hz, 2H), 7.25 (s, 2H), 2.60 (s, 6H), 2.31 (s, 3H) ppm.

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 149.7, 143.9, 142.1, 135.9, 130.4, 126.6, 123.8, 122.1, 121.2 (q, $^{1}J_{C\text{-}F}$ = 323 Hz), 26.8, 21.00 ppm.

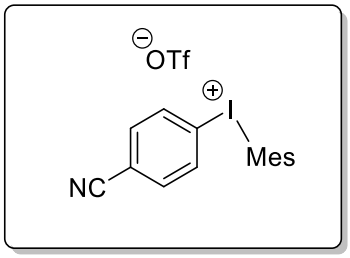
^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -77.7 (s, 3F) ppm.

%Purity (QNMR) = 95%

The analytical data are in accordance with the previously known literature.[12]

Compound 2b

Synthesized using the general procedure B on 0.5 mmol scale and isolated as a white solid in 42% yield (0.104 g, 0.21 mmol). The NMR yield for corresponding reaction that generates the tetrafluoroborate salt was 82%.



^{1}H NMR (400 MHz, [D6] DMSO): δ = 8.10 (d, *J* = 8.3 Hz, 2H), 7.9 (d, *J* = 8.3 Hz, 2H), 7.24(s, 2H), 2.58 (s, 6H), 2.31 (s, 3H) ppm.

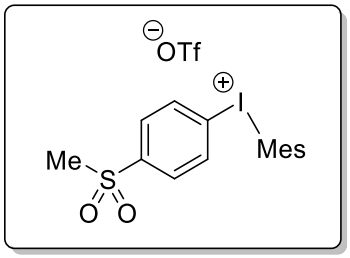
^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 143.9, 142.1, 135.5, 135.3, 130.4, 123.6, 121.2 (q, $^{1}J_{C\text{-}F}$ = 323Hz, -CF_3), 120.6, 118.0, 114.7, 26.8, 21.1 ppm.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -77.7 (s, 3F) ppm.

%Purity (QNMR) = >99%

The analytical data are in accordance with the previously known literature.[13]

Compound 2c



Synthesized using the general procedure B on 0.5 mmol scale and isolated as a white solid in 65% yield (0.176 g, 0.32 mmol). The NMR yield for corresponding reaction that generates the tetrafluoroborate salt was 85%.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 8.13 (d, *J* = 8.16 Hz, 2H), 7.95 (d, *J* = 8.16 Hz), 7.21 (s, 2H), 3.26 (s, 3H), 2.59 (s, 6H), 2.29 (s, 3H) ppm.

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 143.61, 143.34, 141.73, 135.22, 130.20, 130.06, 125.36, 123.34, 121.15 (q, $^{1}J_{C\text{-}F}$ = 323Hz, -CF_3) 43.51, 26.76, 20.99 ppm.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -77.7 (s, 3F) ppm.

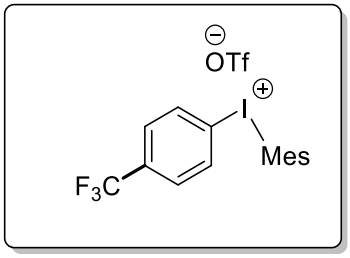
%Purity (QNMR) = 93%

HRMS: Calculated for $C_{16}H_{18}IO_2S^+$ $[M\text{-}OTf]^+$: 401.0067; found 401.00599

FT-IR: 3680, 2980, 2922, 2863, 2843, 1563, 1453, 1387, 1320, 1300, 1268, 1244, 1149, 1024, 771, 633 cm^{-1}

Melting Point: 194-197 °C

Compound 2d



Synthesized using the general procedure B on 0.5 mmol scale and isolated as a white solid in 65% yield (0.173 g, 0.32 mmol). The NMR yield for corresponding reaction that generates the tetrafluoroborate salt was 85%.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 8.13 (d, *J* = 8.2 Hz, 2H), 7.86 (d, *J* = 8.2 Hz, 2H), 7.25 (s, 2H), 2.59 (s, 6H), 2.31 (s, 3H) ppm.

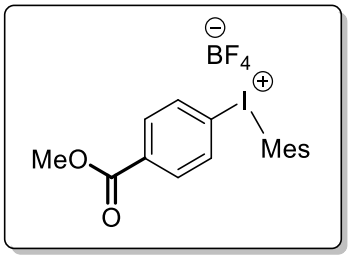
^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 143.90, 142.17, 135.48, 132.08 (q,$^{1}J_{C\text{-}F}$ = 32.3 Hz), 130.39, 128.85 (q, $^{2}J_{C\text{-}F}$ = 3.78 Hz), 125.20, 123.28, 121.1 ($^{1}J_{C\text{-}F}$ = 296 Hz), 119.47, 26.77, 29.99 ppm.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -61.7 (s, 3F), -77.7 (s, 3F) ppm.

%Purity (QNMR) = 98%

The analytical data corresponds to the previously reported literature.[14]

Compound 2e



Synthesized using the general procedure B employing aq. $NaBF_4$ wash instead of aq. NaOTf on 0.5 mmol scale and isolated as a white solid in 76% yield (0.178 g, 0.38 mmol). The NMR yield for corresponding reaction was 99%.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 8.06 (d, *J* = 8.4 Hz, 2H), 7.96 (d, *J* = 8.4 Hz, 2H), 7.22 (s, 2H), 3.85 (s, 3H), 2.59 (s, 6H), 2.30 (s, 3H) ppm.

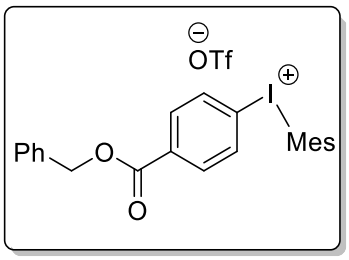
^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 165.6, 143.6, 142.0, 135.0, 132.7, 132.4, 130.3, 123.9, 120.9, 53.1, 26.7, 21.0 ppm.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -148.2, -148.2 (1:3) ppm.

%Purity (QNMR) = 99%

The analytical data corresponds to the previously reported literature.[15]

Compound 2f



Synthesized using the general procedure B on 0.5 mmol scale and isolated as a white solid in 76% yield (0.23 g, 0.38 mmol). The NMR yield for the corresponding reaction was 93%.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 8.08 (d, *J* = 8.3 Hz, 2H), 8.02 (d, *J* = 8.4 Hz, 2H), 7.31-7.51 (m, 5H), 7.23 (s, 2H), 5.34 (s, 2H), 2.58 (s, 6H), 2.30 (s, 3H) ppm.

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 164.9, 143.8, 142.1, 136.2, 135.1, 132.8, 132.5, 130.3, 129.0, 128.7, 128.5, 123.4, 121.2 (q, $^1J_{C\text{-}F}$ = 324 Hz, CF_3), 121.3, 67.3, 26.8, 21.1 ppm.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -77.7 (s, 3F) ppm.

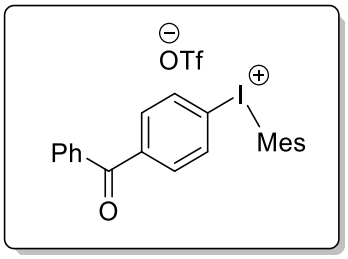
%Purity (QNMR) = 92%

HRMS: Calculated for $C_{23}H_{22}IO_2^+$ [M-OTf]$^+$: 457.06590; found 457.06490

FT-IR: 3076**,** 3680, 2980, 3005, 2980, 2966, 2922, 2863, 2843, 1725, 1583, 1454, 1263, 1244, 1171, 1032, 752, 679, 631 cm^{-1}

Melting point: 154-159 °C

Compound 2g



Synthesized using the general procedure B on 0.5 mmol scale and isolated as a white solid in 44% yield (0.126 g, 0.22 mmol). The NMR yield for corresponding reaction that generates the tetrafluoroborate salt was 75%.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 8.10 (d, *J* = 8.0 Hz, 2H), 7.78 (d, *J* = 8.0 Hz, 2H), 7.65-7.75 (m, 3H), 7.57 (t, *J* = 7.7 Hz,2H), 7.26 (s, 2H), 2.63 (s, 6H), 2.31 (s, 3H) ppm.

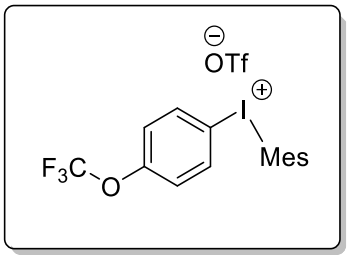
^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 195.1, 143.8, 142.2, 140.1, 136.4, 134.8, 133.9, 132.7, 130.4, 130.3, 129.2, 123.3, 121.2 (q, $^{1}J_{C\text{-}F}$ = 324 Hz, CF_3), 119.1, 26.8, 21.0 ppm.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -77.7 (s, 3F) ppm.

%Purity (QNMR) = 99%

The analytical data corresponds to the previously reported literature.[16]

Compound 2h



Synthesized using the general procedure B on 0.5 mmol scale and isolated as a white solid in 76% yield (0.211 g, 0.38 mmol). The NMR yield for corresponding reaction that generates the tetrafluoroborate salt was 88%.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 8.07 (d, *J* = 9 Hz, 2H), 7.51 (d, *J* = 8.32 Hz, 2H), 7.23 (s, 2H), 2.60 (s, 6H), 2.30 (s, 3H) ppm.

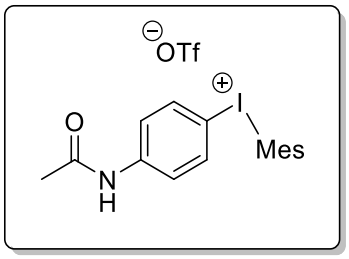
^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 150.8, 150.8, 143.8, 142.0, 137.2, 130.3, 124.5, 121.2 (q, $^{1}J_{C\text{-}F}$ = 324 Hz, -CF_3), 120.3 (q, $^{1}J_{C\text{-}F}$ = 258 Hz, -OCF_3), 112.9, 26.7, 21.0 ppm.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -56.9 (s, 3F), -77.8 (s, 3F) ppm.

%Purity (QNMR) = >99%

The analytical data are in accordance with the previously reported literature.[17]

Compound 2i



Synthesized using the general procedure B on 0.5 mmol scale and isolated as a white solid in 64% yield (0.169 g, 0.32 mmol). The NMR yield for corresponding reaction that generates the tetrafluoroborate salt was 88%.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 10.29 (s, 1H), 7.92 (d, *J* = 8.8 Hz, 2H), 7.66 (d, *J* = 8.8 Hz), 7.19 (s, 2H), 2.60 (s, 6H), 2.28 (s, 3H), 2.05 (s, 3H) ppm.

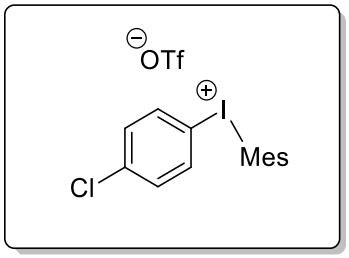
^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 169.5, 142.2, 142.7, 141.7, 136.1, 130.1, 123.9, 122.8, 121.9, 121.2(q, $^{1}J_{C\text{-}F}$ = 323 Hz), 107.0, 26.7, 24.6, 21.0 ppm.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -77.7 (s, 3F) ppm.

%Purity (QNMR) = 95%

The analytical data are in accordance with the previously reported literature.[14]

Compound 2j



Synthesized using the general procedure B on 0.5 mmol scale and isolated as a white solid in 48% yield (0.125 g, 0.24 mmol). The NMR yield for corresponding reaction that generates the tetrafluoroborate salt was 97%.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.95 (d, *J* = 8.4 Hz, 2H), 7.75 (d, *J* = 8.4 Hz, 2H), 7.22 (s, 2H), 2.59 (s, 6H), 2.30 (s, 3H) ppm.

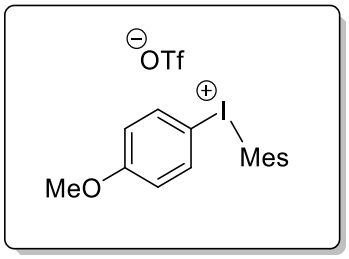
^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 143.6, 142.0, 137.4, 136.6, 132.2, 130.3, 123.5, 121.2 (q, $^{1}J_{C\text{-}F}$ = 324 Hz, -CF_3), 113.2, 26.7, 21.0 ppm.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -77.7 (s, 3F) ppm.

%Purity (QNMR) = 91%

The analytical data are in accordance with the previously known literature.[12]

Compound 2k



Synthesized using the general procedure B on 0.5 mmol scale and isolated as a white solid in 89% yield (0.23g, 0.46 mmol). The NMR yield for corresponding reaction was 98%.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.92 (d, *J* = 8.8 Hz, 2H), 7.19 (s, 2H), 7.03 (d, *J* = 8.8 Hz, 2H), 3.78 (s, 3H), 2.60 (s, 6H), 2.28 (s, 3H) ppm.

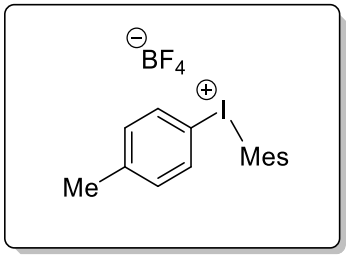
^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 162.2, 143.3, 141.8, 137.0, 130.1, 123.6, 122.8, 121.2 (q, $^{1}J_{C\text{-}F}$ = 323 Hz, -CF_3), 117.9, 104.0, 56.1, 26.7, 21.0 ppm.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -77.7 (s, 3F) ppm.

%Purity (QNMR) = 94%

The analytical data are in accordance with the previously known literature.[12]

Compound 2l



Synthesized using the general procedure B employing aq. $NaBF_4$ wash instead of aq. NaOTf, on 0.5 mmol scale and isolated as a white solid in 80% yield (0.176 g, 0.4 mmol). The NMR yield for corresponding reaction that generates the tetrafluoroborate salt was 97%.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.83 (d, *J* = 7.9 Hz, 2H), 7.29 (d, *J* = 7.8 Hz, 2H), 7.18 (s, 2H), 2.58 (s, 6H), 2.31 (s, 3H), 2.28 (s, 3H) ppm.

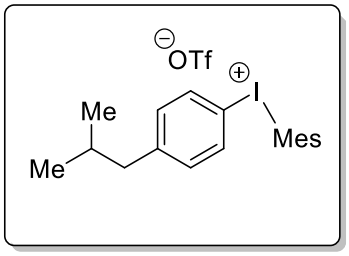
^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): 143.2, 142.4, 141.8, 134.8, 132.8, 130.1, 124.0, 123.3, 26.7, 21.2, 21.0 ppm

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): -148.2 ppm.

%Purity (QNMR) = 99%

The analytical data are in accordance with the previously known literature.[12]

Compound 2m



Synthesized using the general procedure B on 0.5 mmol scale and isolated as white solid in 68% yield (0.182 g, 0.34 mmol). The NMR yield for the corresponding reaction that generates the tetrafluoroborate salt was 83%. **Melting point** = 161-167 ºC

^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.88 (d, *J* = 8.0 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 7.21 (s, 2H), 2.59 (s, 6H), 2.47 (d, *J* = 7.2 Hz, 2H), 2.29 (s, 3H), 1.81 (m, 1H), 0.83 (d, *J* = 6.5 Hz, 6H) ppm.

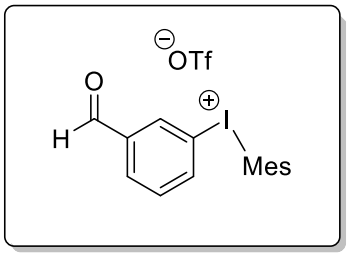
^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 146.0, 143.4, 141.9, 134.9, 132.9, 130.2, 123.3, 121.1 (q, $^{1}J_{C\text{-}F}$ = 323 Hz), 112.0, 44.3, 29.9, 26.7, 22.4, 21.0 ppm.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -77.7 (s, 3F) ppm.

%Purity (QNMR) = >99%

The analytical data are in accordance with the previously reported literature.[18]

Compound 2n



Synthesized using the general procedure B on 0.5 mmol scale and isolated as a pale white solid in 60% yield (0.154 g, 0.3 mmol). The NMR yield for the corresponding reaction that generates the tetrafluoroborate salt was 96%.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 9.99 (s, 1H), 8.44 (s, 1H), 8.19 (d, *J* = 7.8 Hz, 1H), 8.13 (d, *J* = 7.5 Hz, 1H), 7.71 (t, *J* = 7.8 Hz, 1H), 7.23 (s, 2H), 2.61 (s, 6H), 2.30 (s, 3H) ppm.

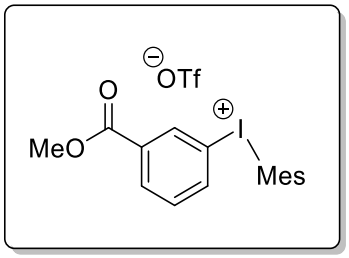
^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 192.1, 143.8, 142.1, 139.9, 138.9, 134.8, 133.0, 130.3, 123.3, 121.2 (q, $^{1}J_{C\text{-}F}$ = 324 Hz), 116.0, 26.8, 21.0 ppm.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -77.7 (s, 3F) ppm.

%Purity (QNMR) = 94%

The analytical data are in accordance with the previously reported literature.[19]

Compound 2o



Synthesized using the general procedure B on 0.5 mmol scale as while power in 66% isolated yield (0.180 g, 0.33 mmol). The NMR yield of the corresponding reaction that generates the tetrafluoroborate salt was 92%.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 8.49 (s, 1H), 8.14 (d, *J* = 7.72 Hz, 1H), 8.07 (d, *J* = 8.0 Hz, 1H), 7.63 (t, *J* = 7.9 Hz, 2H), 7.24 (s, 2H), 3.88 (s, 3H), 2.59 (s, 6H), 2.31 (s, 3H) ppm.

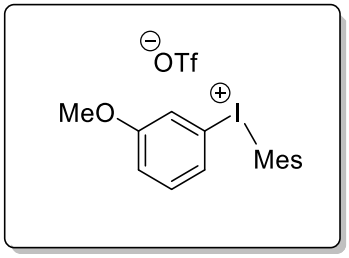
^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 164.9, 143.8, 142.1, 138.7, 134.9, 132.8, 132.7, 132.4, 130.3, 123.2, 122.7, 121.2 (q, $^{1}J_{C\text{-}F}$ = 323 Hz), 115.3, 53.3, 26.8, 21.0 ppm.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -77.7 (s, 3F) ppm.

%Purity (QNMR) = 96%

The analytical data are in accordance with the previously reported literature.[17]

Compound 2p



Synthesized using the general procedure B on 0.5 mmol scale and isolated as a sticky solid in 60% yield (0.155 g, 0.3 mmol). NMR yield of the corresponding reaction that generates the tetrafluoroborate salt was 84%.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.56 (s, 1H), 7.36-7.47 (m, 2H), 7.17-7.26 (m, 3H), 3.78 (s, 3H), 2.60 (s, 6H), 2.30 (s, 3H) ppm.

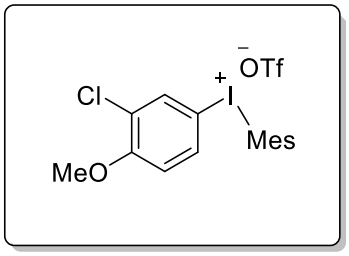
^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 161.0, 143.6, 142.1, 133.1, 130.2, 126.5, 123.0, 122.8, 122.0 (q, $^{1}J_{C\text{-}F}$ = 323 Hz), 120.5, 117.7, 114.8, 56.3, 26.8, 21.0 ppm.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -77.7 (s, 3F) ppm.

%Purity (QNMR) = 99%

The analytical data are in accordance with the previously reported literature.[20]

Compound 2q



Synthesized using the general procedure B on 0.5 mmol scale and isolated as white solid in 53% yield (0.143 g, 0.26 mmol). rThe NMR yield for the corresponding reaction that generates the tetrafluoroborate salt was 84%.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 8.13 (s, 1H), 7.86 (d, *J* = 7.8 Hz), 7.29-7.09 (m, 3H), 3.88 (s, 3H), 2.60 (s, 6H), 2.29 (s, 3H) ppm.

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 157.6, 143.3, 141.8, 135.6, 135.5, 130.2, 124.3, 123.7, 121.1 (q, $^{1}J_{C\text{-}F}$ = 323 Hz), 116.1, 104.5, 57.1, 26.7, 20.9 ppm.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -77.7 (s, 3F) ppm.

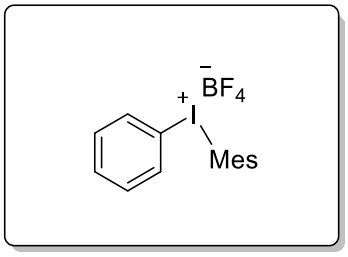
HRMS: Calculated for $C_{16}H_{17}ICIO^{+}$ [M-OTf]$^{+}$: 387.00071; found 386.9999

FT-IR: 3705, 3005, 2951, 2865, 2843, 1610, 1570, 1454, 1380, 1298, 1239, 1221, 1195, 1168, 1057, 1014, 826, 790, 630 cm^{-1}

Melting point: 176-182 °C

% Purity (QNMR) = 99%

Compound 2r



Synthesized using the general procedure B with aq. $NaBF_4$ was instead of NaOTf and isolated as a pale white solid in 92% yield (0.187g, 0.46 mmol). The NMR yield for the corresponding reaction was 99%.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 7.95 (d, *J* = 7.8 Hz, 2H), 7.62 (t, *J* = 7.3 Hz, 1H), 7.5 (t, *J* = 7.7 Hz, 2H), 7.21 (s, 2H), 2.59 (s, 6H), 2.29 (s, 3H) ppm.

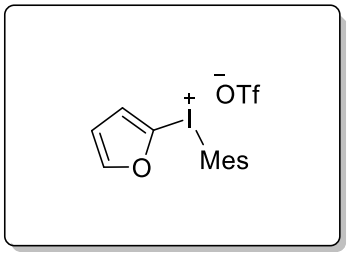
^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 143.4, 142.0, 134.8, 132.3, 132.1, 130.2, 123.6, 115.7, 26.7, 21.0 ppm.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = - 148.2, -148.3 (1:3) ppm.

% Purity (QNMR) = 97%

The analytical data are in accordance with the previously known literature.[12]

Compound 2s



Synthesized using the general procedure B on 0.5 mmol scale and isolated as a white solid in 59% yield (0.136 g, 0.29 mmol). The NMR yield for the corresponding reaction that generates the tetrafluoroborate salt was 87%.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 8.0 (d, *J* = 1.32 Hz, 1H), 7.48 (d, *J* = 3.4, 1H), 7.20 (s, 2H), 6.68-6.64 (m, 1H), 2.66 (s, 6H), 2.28 (s, 3H) ppm.

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 151.3, 143.4, 141.1, 130.1, 126.8, 124.4, 114.8, 121.1 (q, $^{1}J_{C\text{-}F}$ = 323 Hz), 113.9, 26.7, 21.0 ppm.

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -77.7 (s, 3F) ppm.

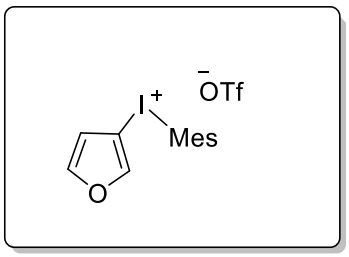
% Purity (QNMR) = 93%

HRMS: Calculated for $C_{13}H_{14}IO^{+}$ $[M\text{-}OTf]^{+}$: 313.00838; found 313.00780

FT-IR: 3712, 3680, 3132, 2980, 2864, 1589, 1449, 1379, 1268, 1244, 1160, 1031, 893, 851, 759, 628 cm^{-1}

Melting point: 136-139 °C

Compound 2t



Synthesized using the general procedure B on 0.5 mmol scale and isolated as a white solid in 77% yield (0.179 g, 0.38 mmol). The NMR yield for the corresponding reaction that generates the tetrafluoroborate salt was 93%.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 8.52 (s, 1H), 7.91 (m, 1H), 7.30 (s, 2H), 7.05 (d, *J* = 1.4 Hz), 2.64 (s, 6H), 2.29 (s, 3H) ppm.

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 150.4, 146.7, 143.4, 141.5, 130.0, 124.2, 121.1 (q, $^{1}J_{C\text{-}F}$ = 323 Hz), 114.1, 89.0, 26.8, 21.0 ppm

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -77.7 (s, 3F) ppm

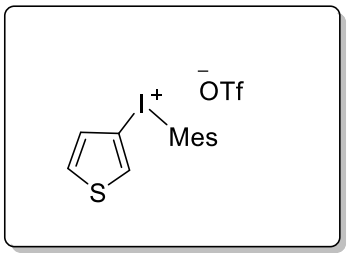
% Purity (QNMR) = 98%

HRMS: Calculated for $C_{13}H_{14}IO^+$ $[M\text{-}OTf]^+$: 313.00838; found 313.00786

FT-IR: 3680, 3162, 3134, 2986, 2936, 1478, 1451, 1272, 1221, 1237, 1161, 1019, 903, 868, 851, 792, 628 cm^{-1}

Melting point: 168-170 °C

Compound 2u



Synthesized using the general procedure B on 0.5 mmol scale and isolated as a white solid in 60% yield (0.145 g, 0.3 mmol). The NMR yield for the corresponding reaction that generates the tetrafluoroborate salt was 79%.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 8.49 (m, 1H), 7.84-7.43 (m, 1H), 7.53 (d, *J* = 5 Hz, 1H), 7.20 (s, 2H), 2.63 (s, 6H), 2.28 (s, 3H) ppm

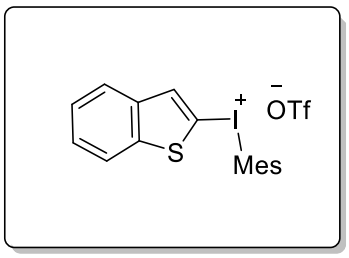
^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 143.4, 141.6, 135.5, 131.7, 131.1, 130.1, 124.3, 121.2 (q, $^1J_{C\text{-}F}$ = 324 Hz), 99.8, 26.8, 21.0 ppm

^{19}F{^{1}H} NMR (376 MHz, [D6] DMSO): δ = -77.7 (s, 3F) ppm.

% Purity (QNMR) = 98%

The analytical data are in accordance with the previously known literature.[21]

Compound 2v



Synthesized using the GP-A on 0.5 mmol scale and isolated as a white solid in 69% yield (0.179 g, 0.33 mmol). The NMR yield for the corresponding reaction that generates the tetrafluoroborate salt was 78%.

^{1}H NMR (400 MHz, [D6] DMSO): δ = 8.29 (s, 1H), 8.12 - 7.89 (m, 2H), 7.54 - 7.38 (m, 2H), 7.23 (s, 2H), 2.70 (s, 6H), 2.29 (s, 3H) ppm.

^{13}C{^{1}H} NMR (101 MHz, [D6] DMSO): δ = 143.7, 143.2, 141.5, 139.2, 136.0, 130.2, 127.2, 126.2, 126.0, 125.1, 123.0, 121.2 (q, $^1J_{C\text{-}F}$ = 324 Hz), 105.0, 26.8, 21.0 ppm.

$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): δ = -77.7 (s, 3F) ppm.

% Purity (QNMR) = >99%

HRMS: Calculated for $C_{17}H_{16}IS^{+}$ $[M\text{-}OTf]^{+}$:379.00119; found 379.00305

FT-IR: 3672, 3054, 2978, 1738, 1590, 1455, 1377, 1221, 1157, 1058, 1023, 958, 945, 848, 829, 750, 705, 634 cm^{-1}

Melting point: Decomposition starting at 146-151 °C

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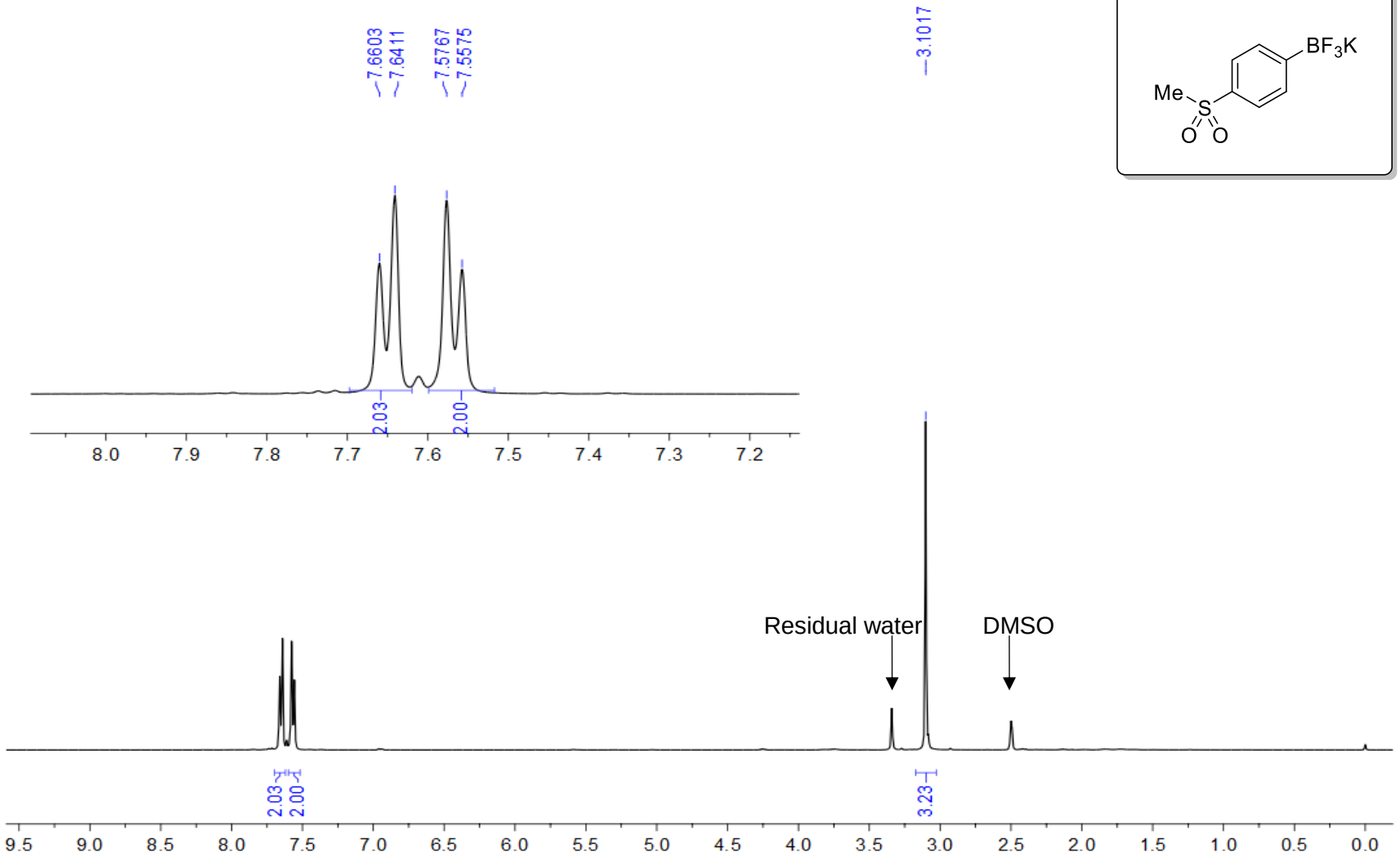
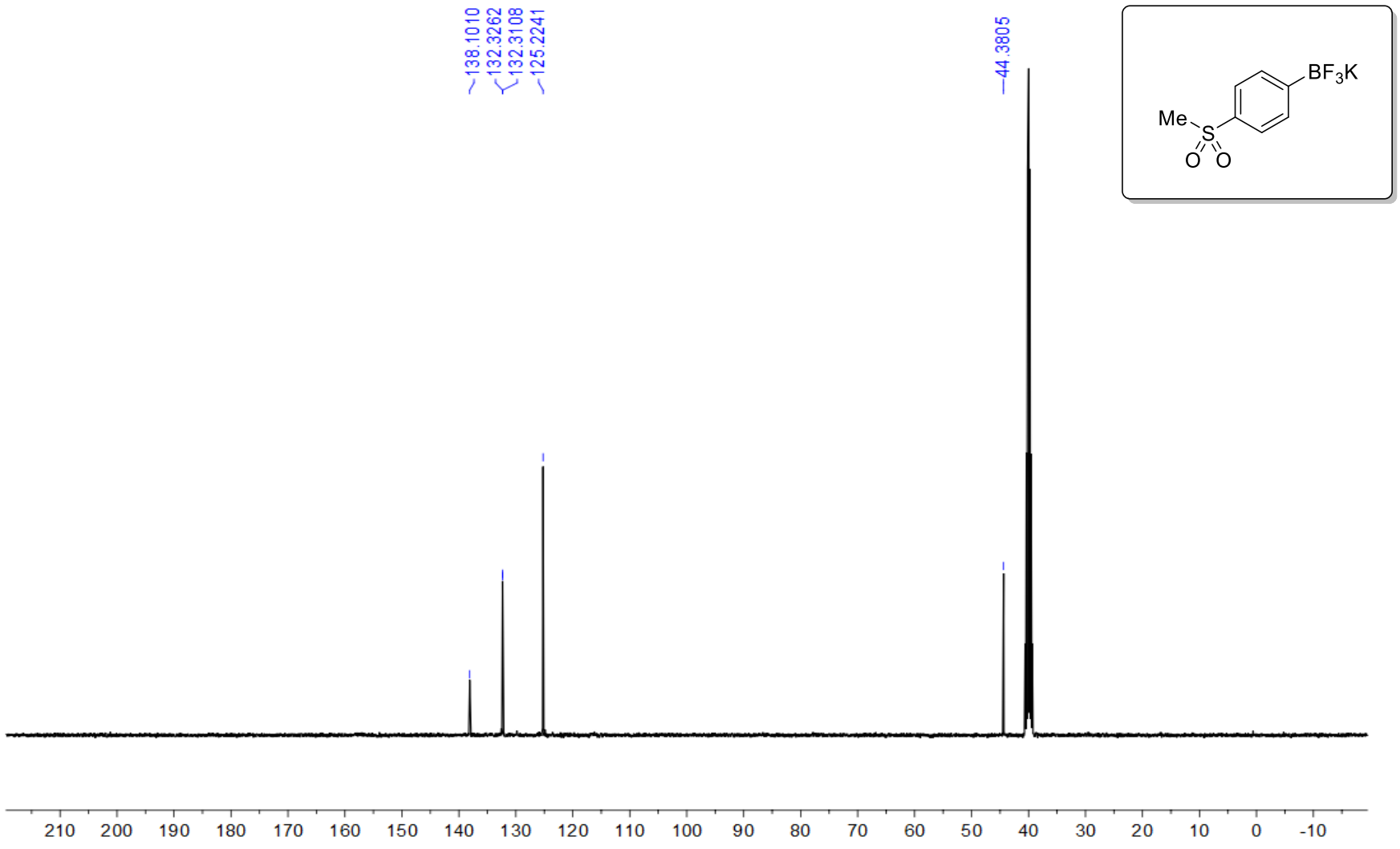
10. <u>Spectral data for potassium aryltrifluoroborate salts</u>

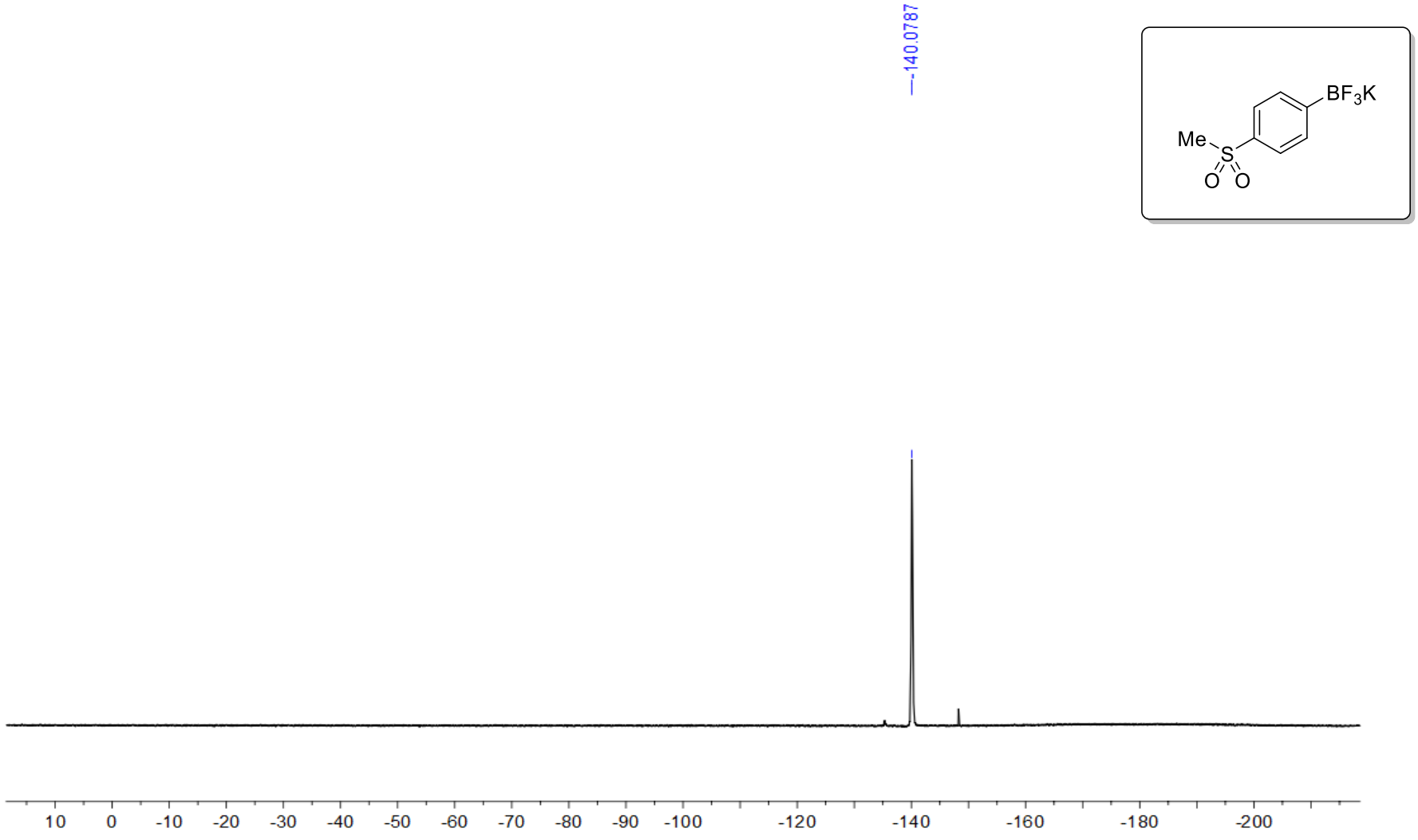
^{1}H NMR (400 MHz, [D6] DMSO): Compound 1c



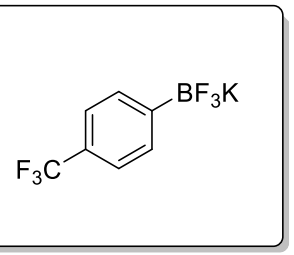
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound 1c



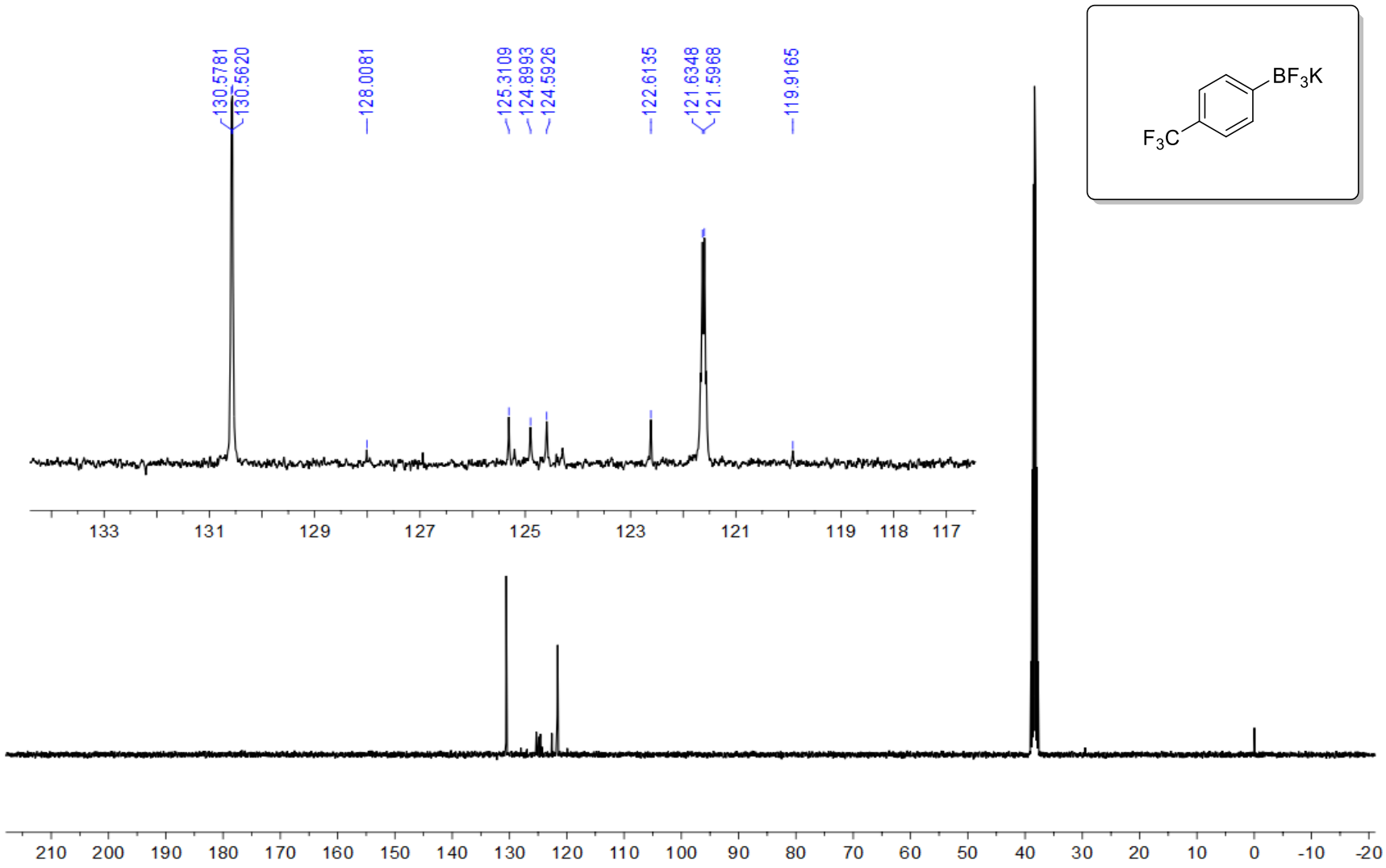
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound 1c



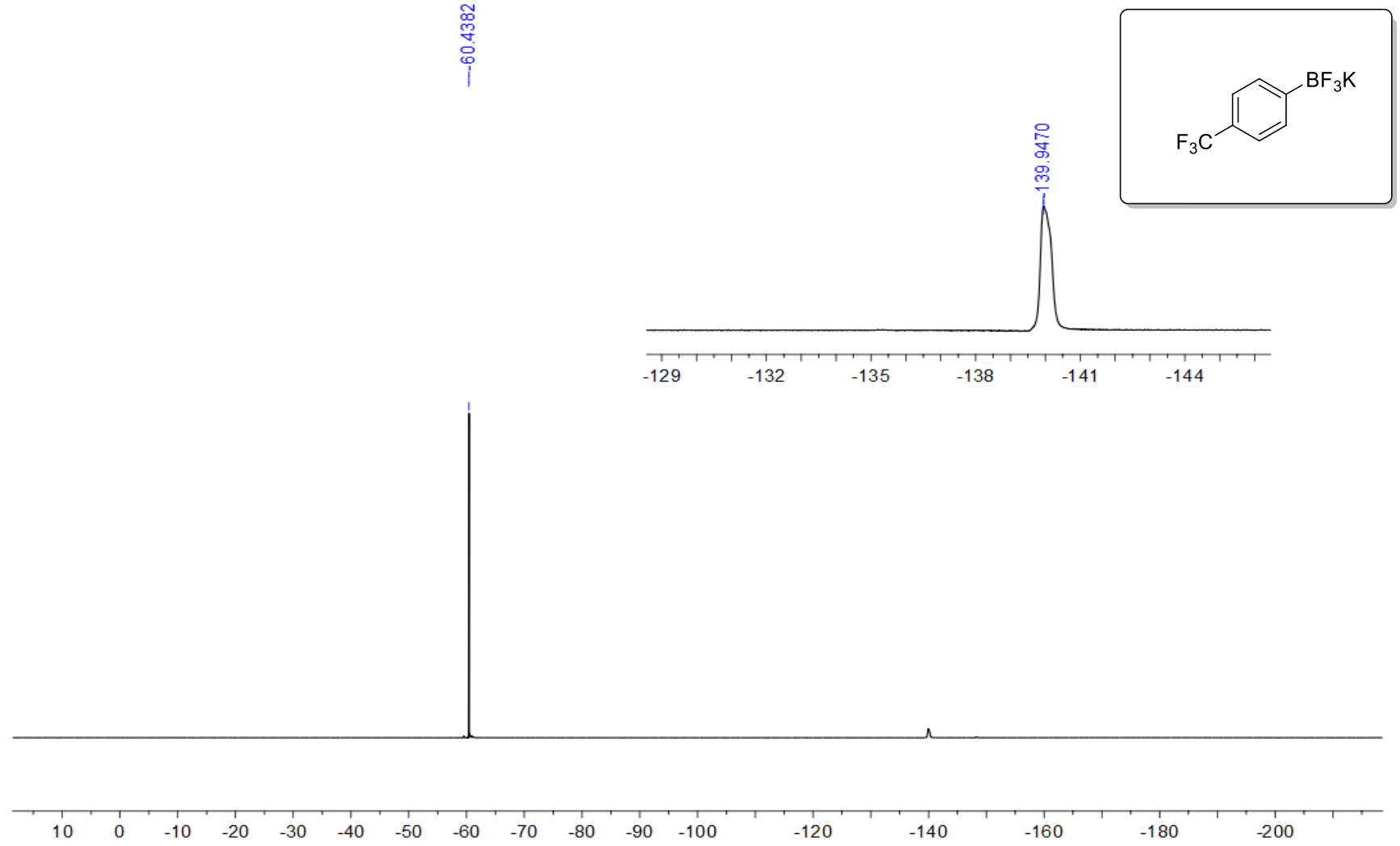
^{1}H NMR (400 MHz, [D6] DMSO): Compound 1d



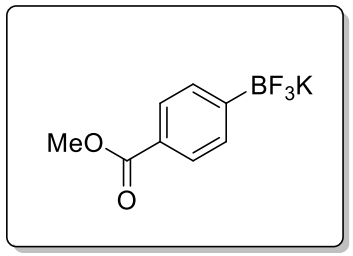
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound 1d



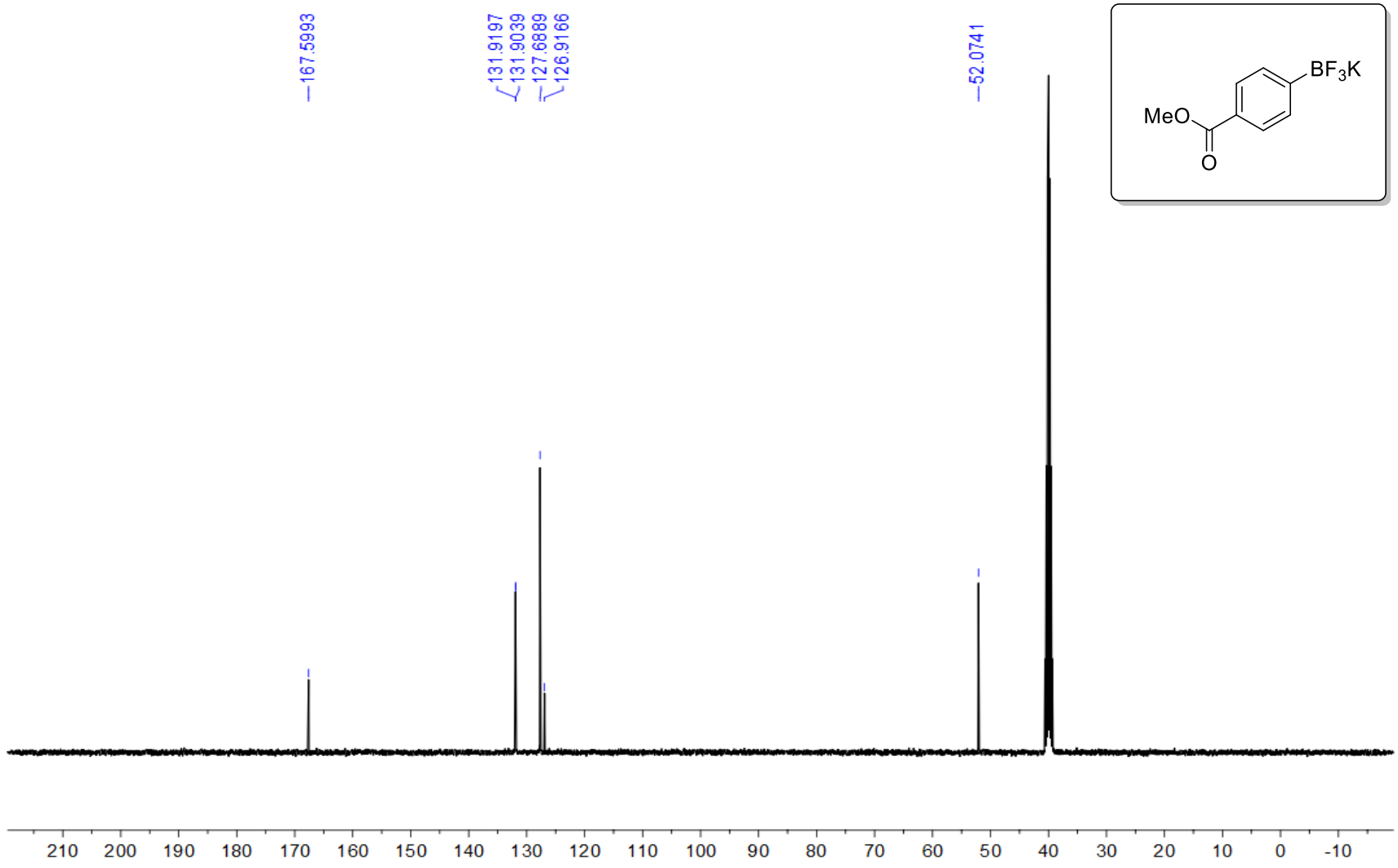
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound 1d



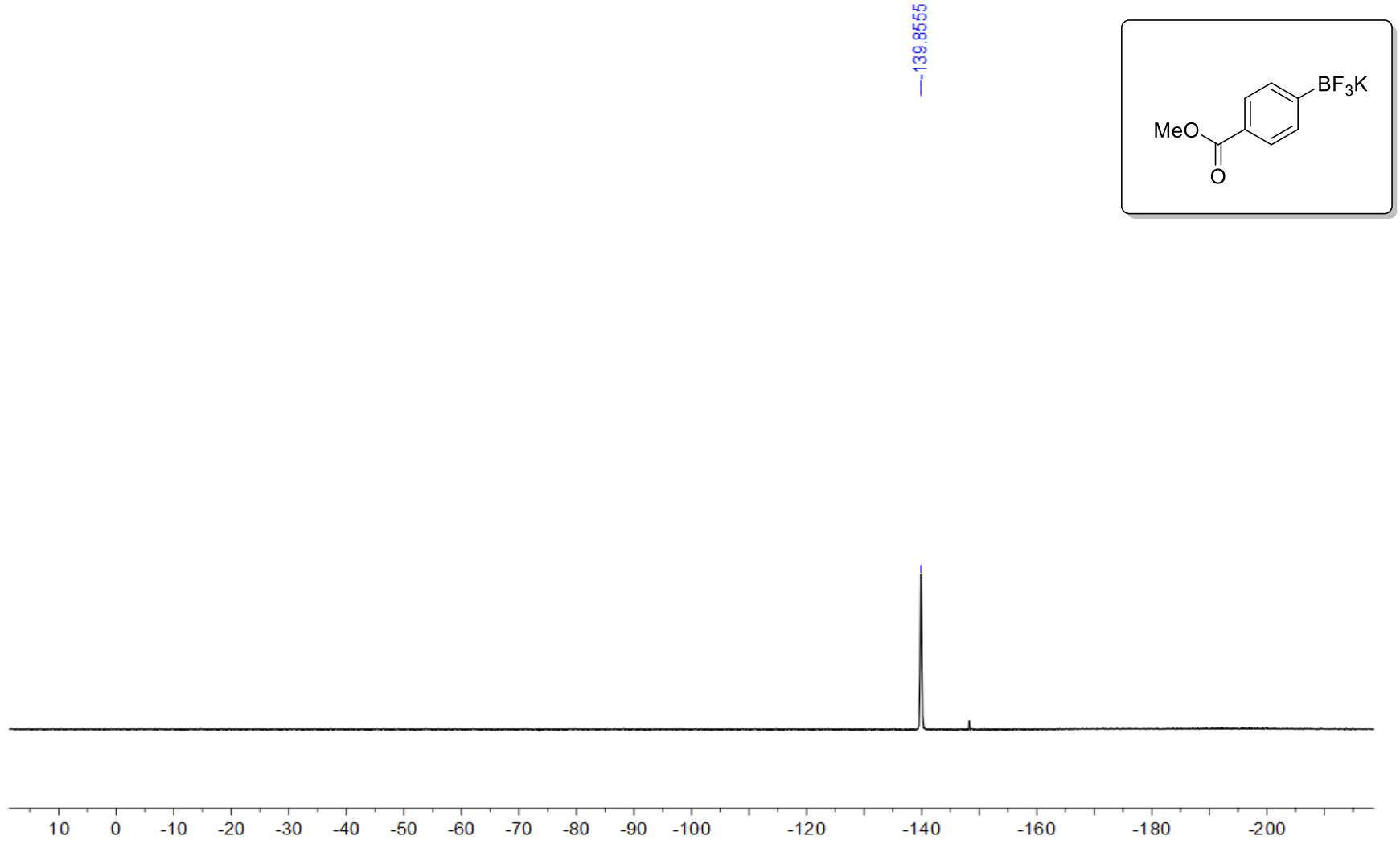
^{1}H NMR (400 MHz, [D6] DMSO): Compound 1e



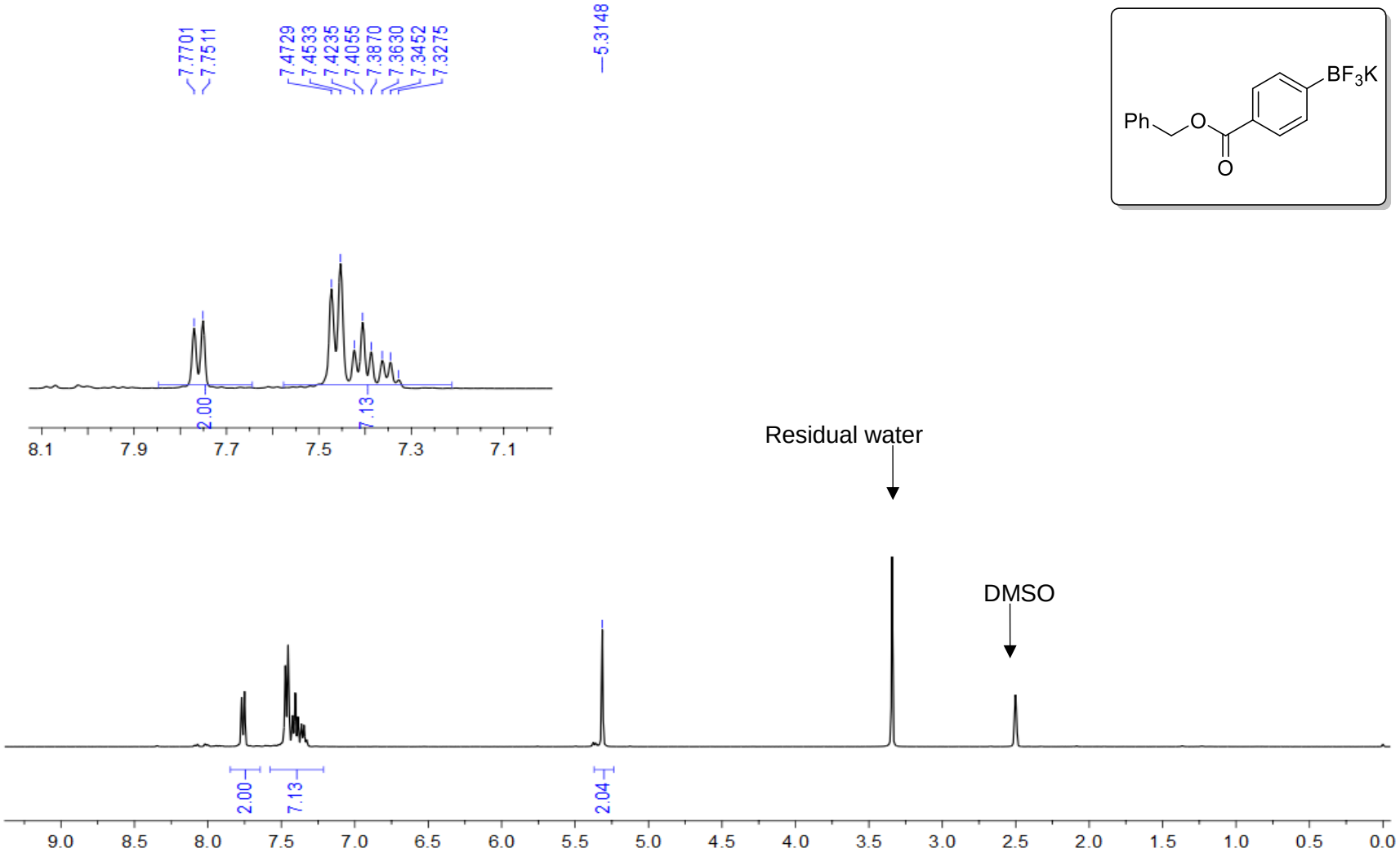
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound 1e



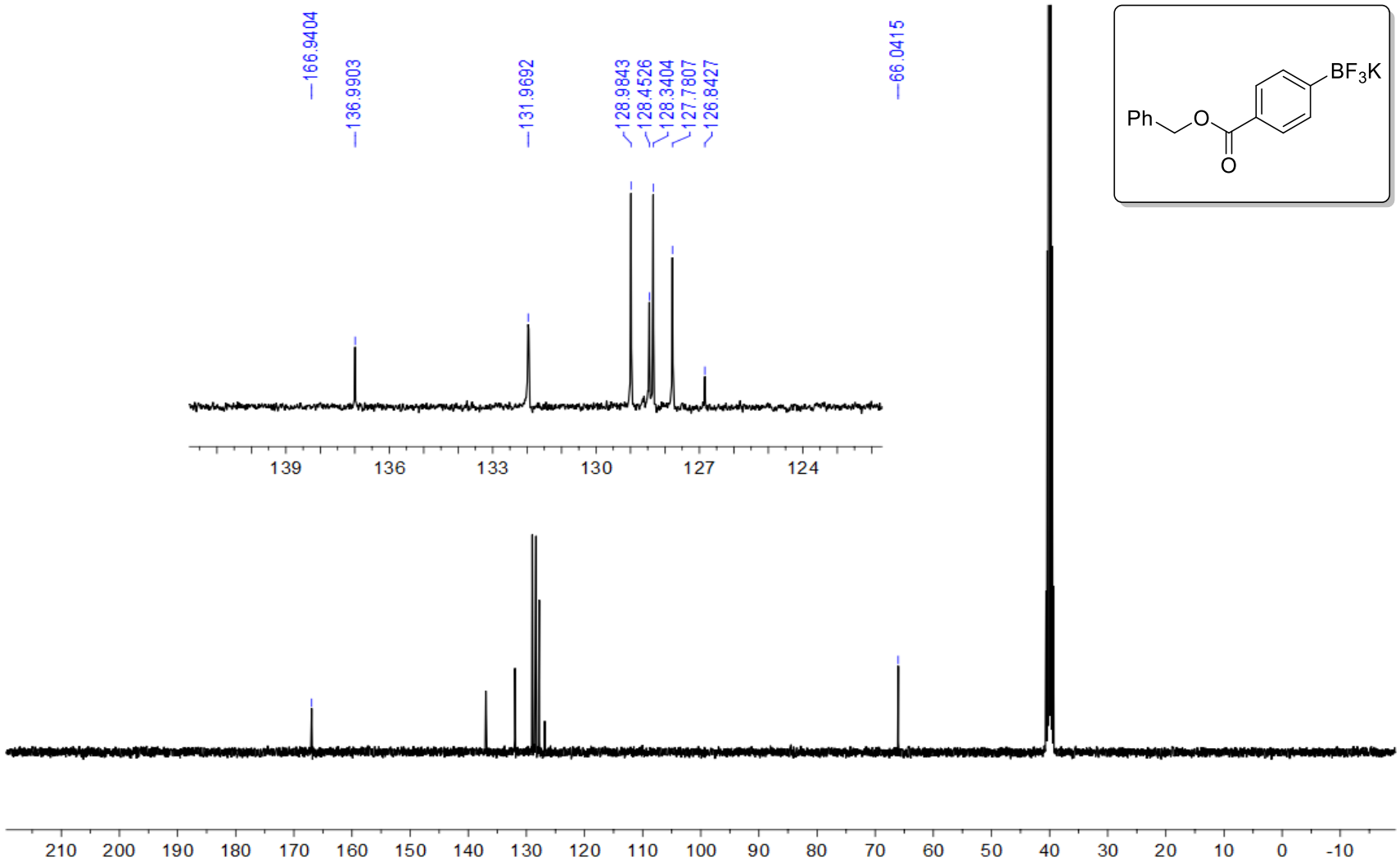
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound 1e



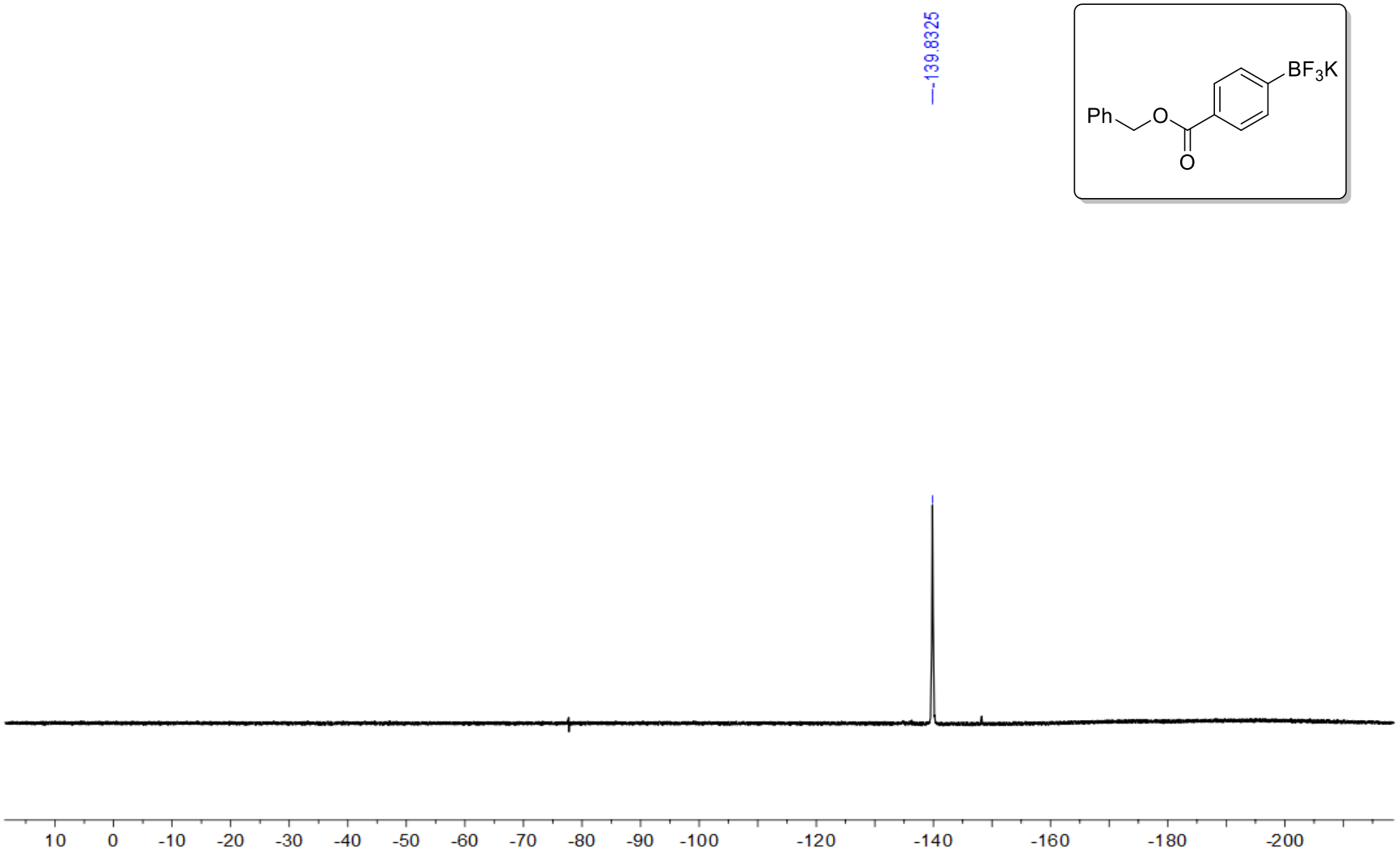
^{1}H NMR (400 MHz, [D6] DMSO): Compound 1f



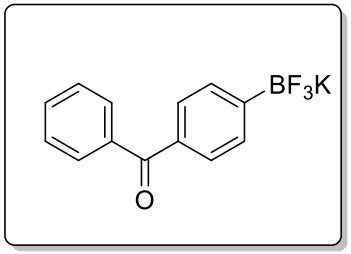
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound 1f



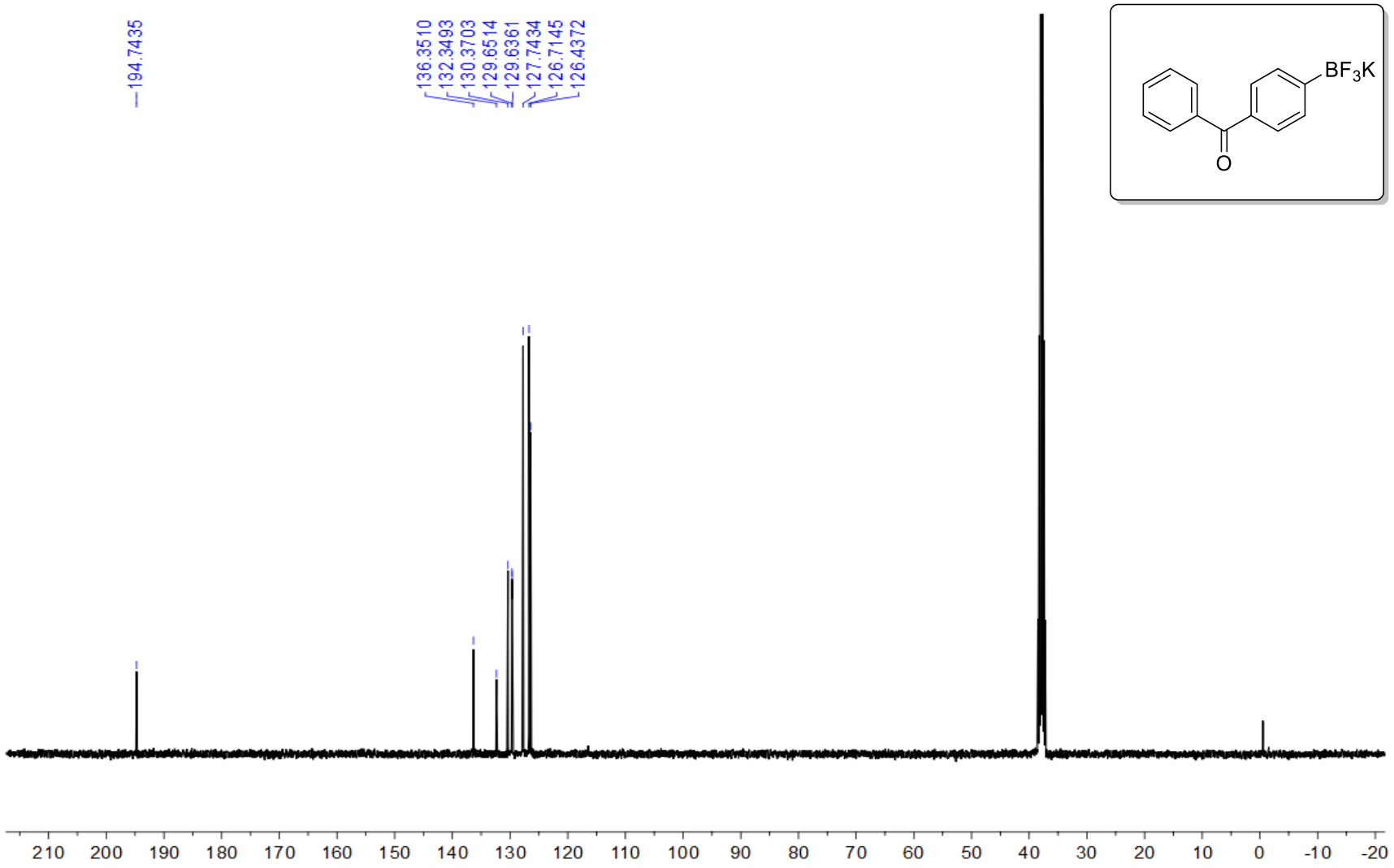
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound 1f



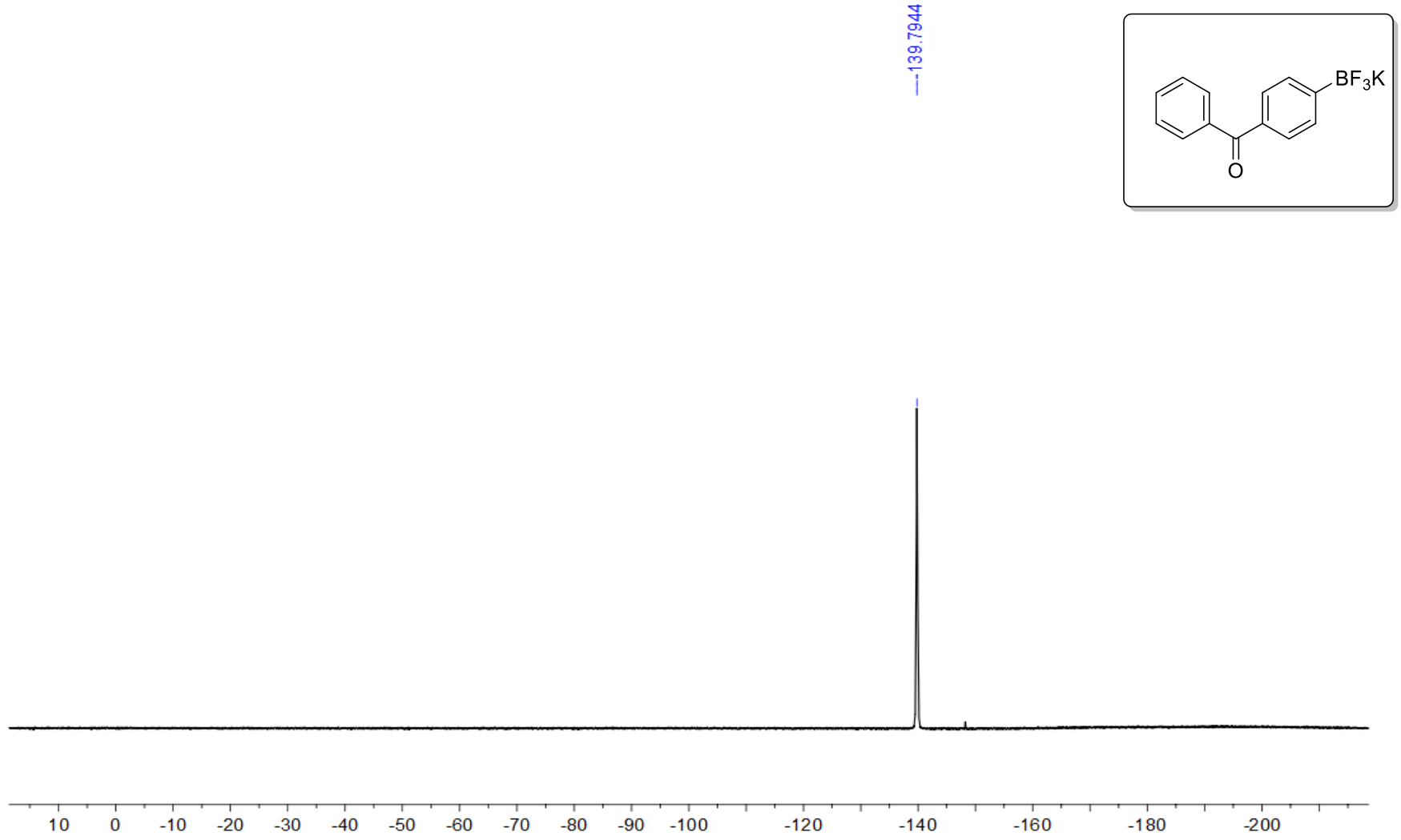
^{1}H NMR (400 MHz, [D6] DMSO): Compound **1g**



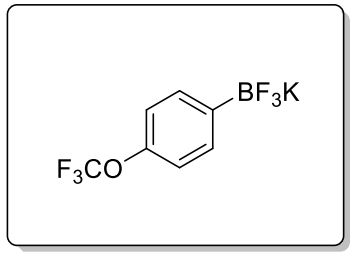
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **1g**

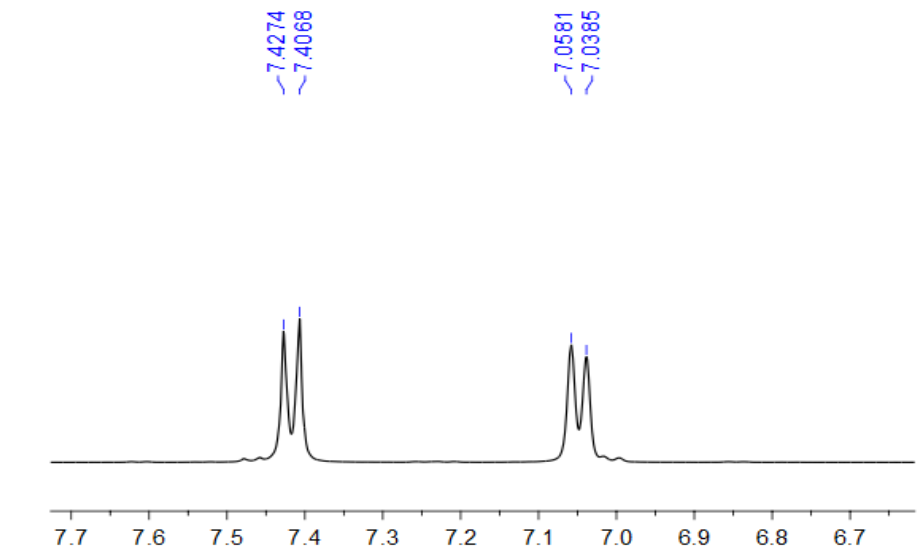


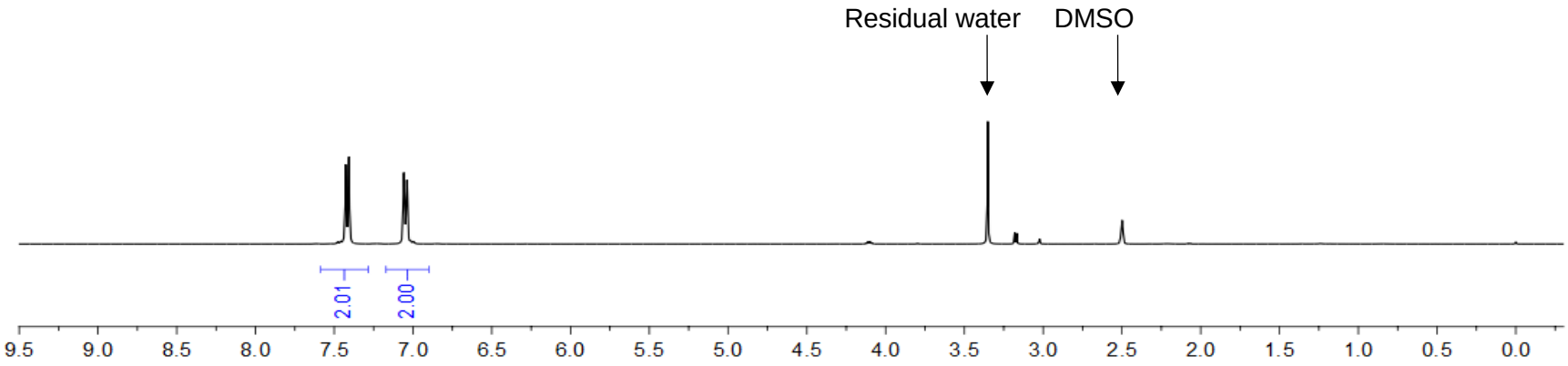
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **1g**



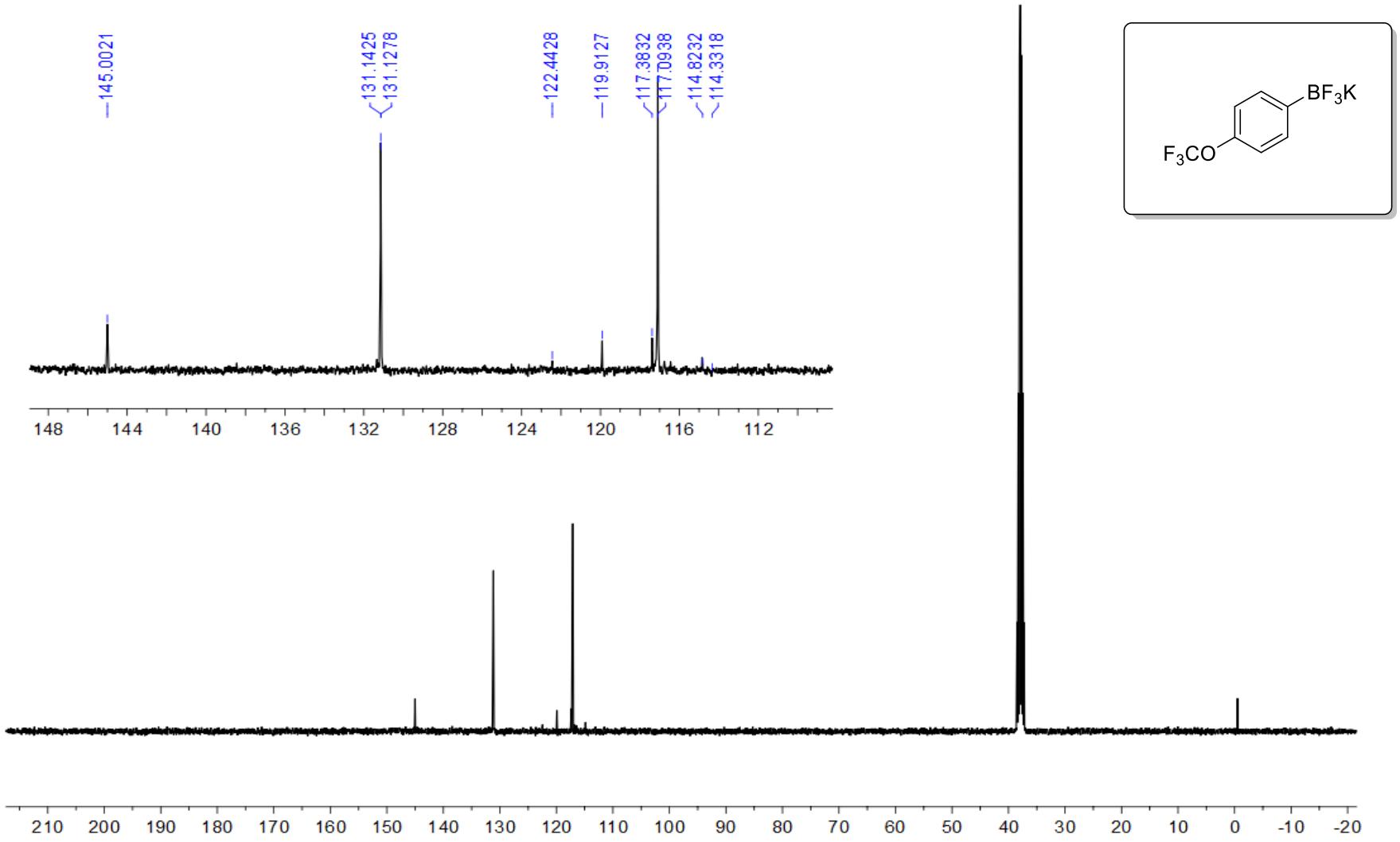
^{1}H NMR (400 MHz, [D6] DMSO): Compound **1h**



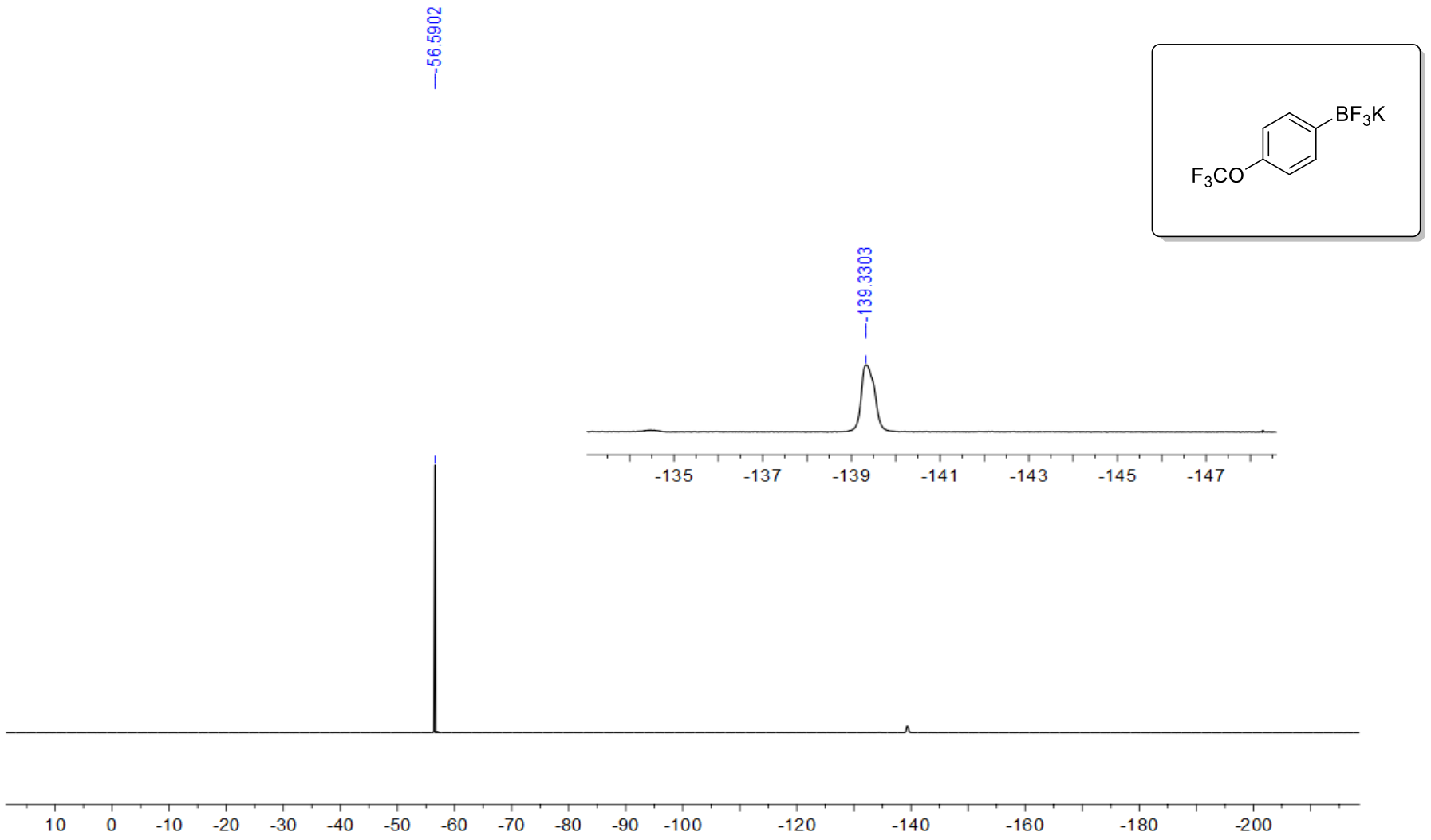




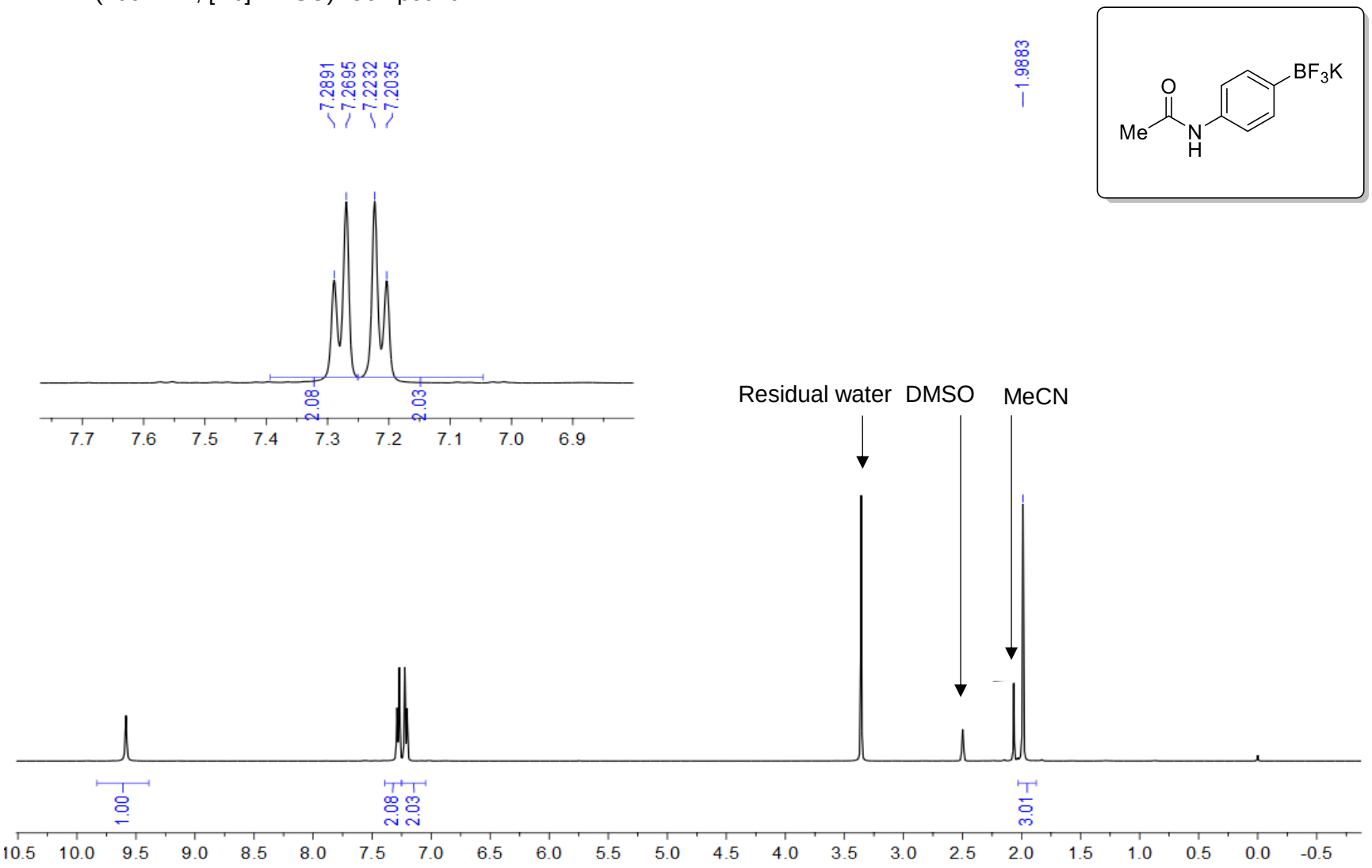
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **1h**



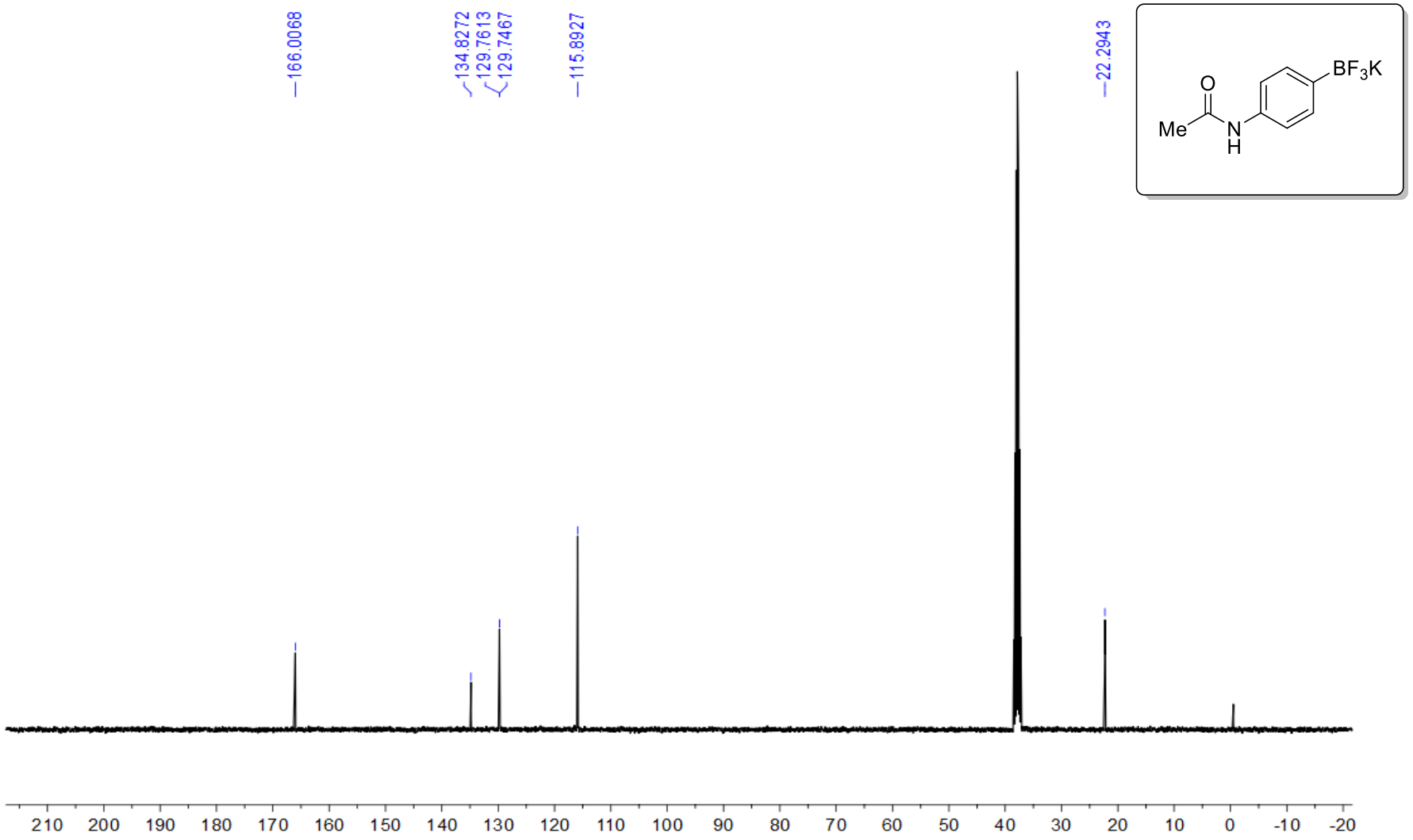
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **1h**



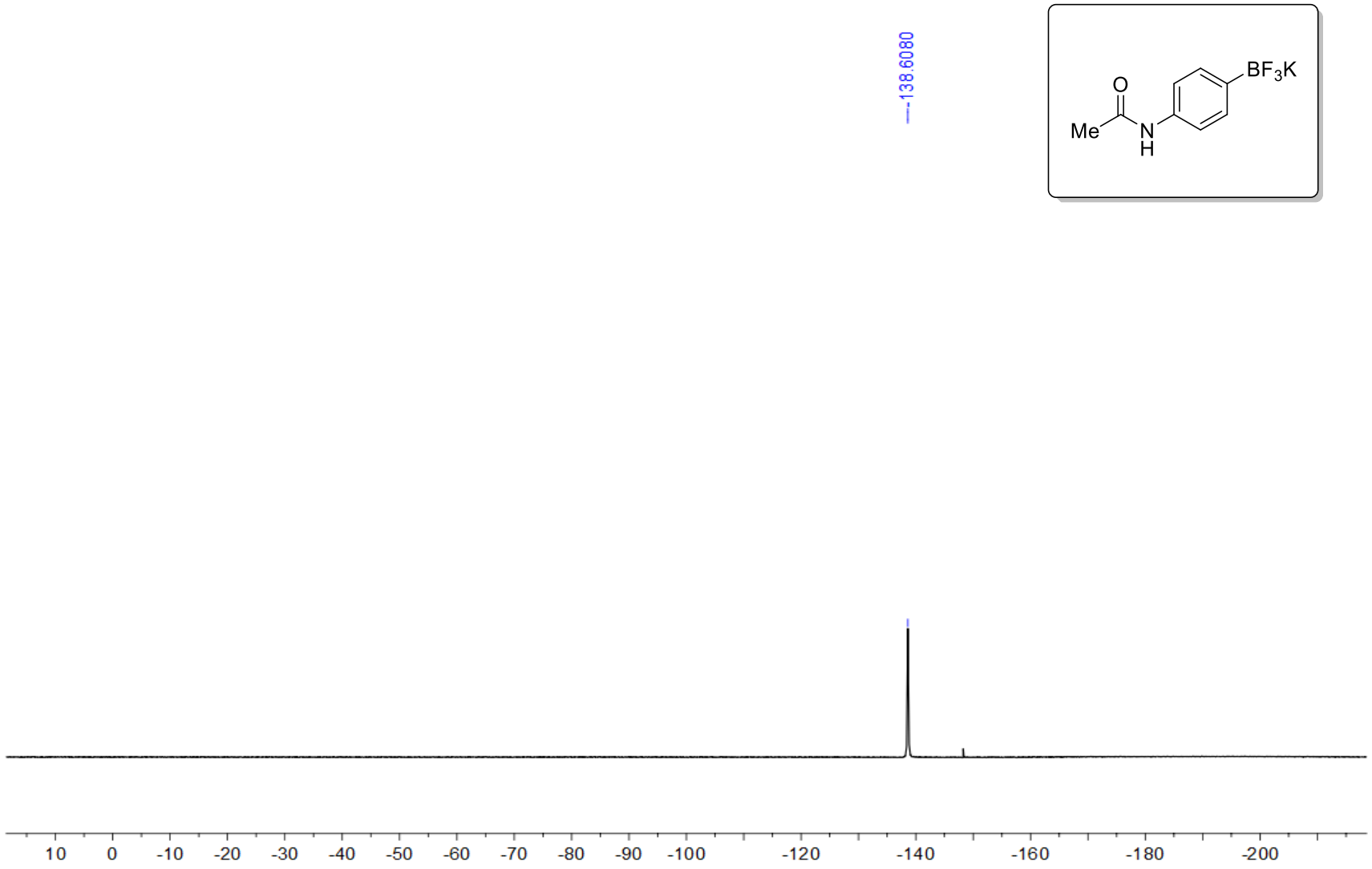
^{1}H NMR (400 MHz, [D6] DMSO): Compound **1i**



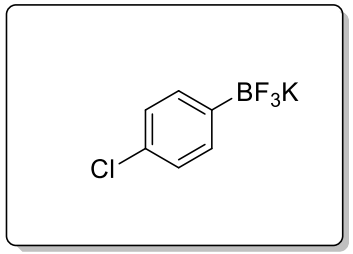
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **1i**



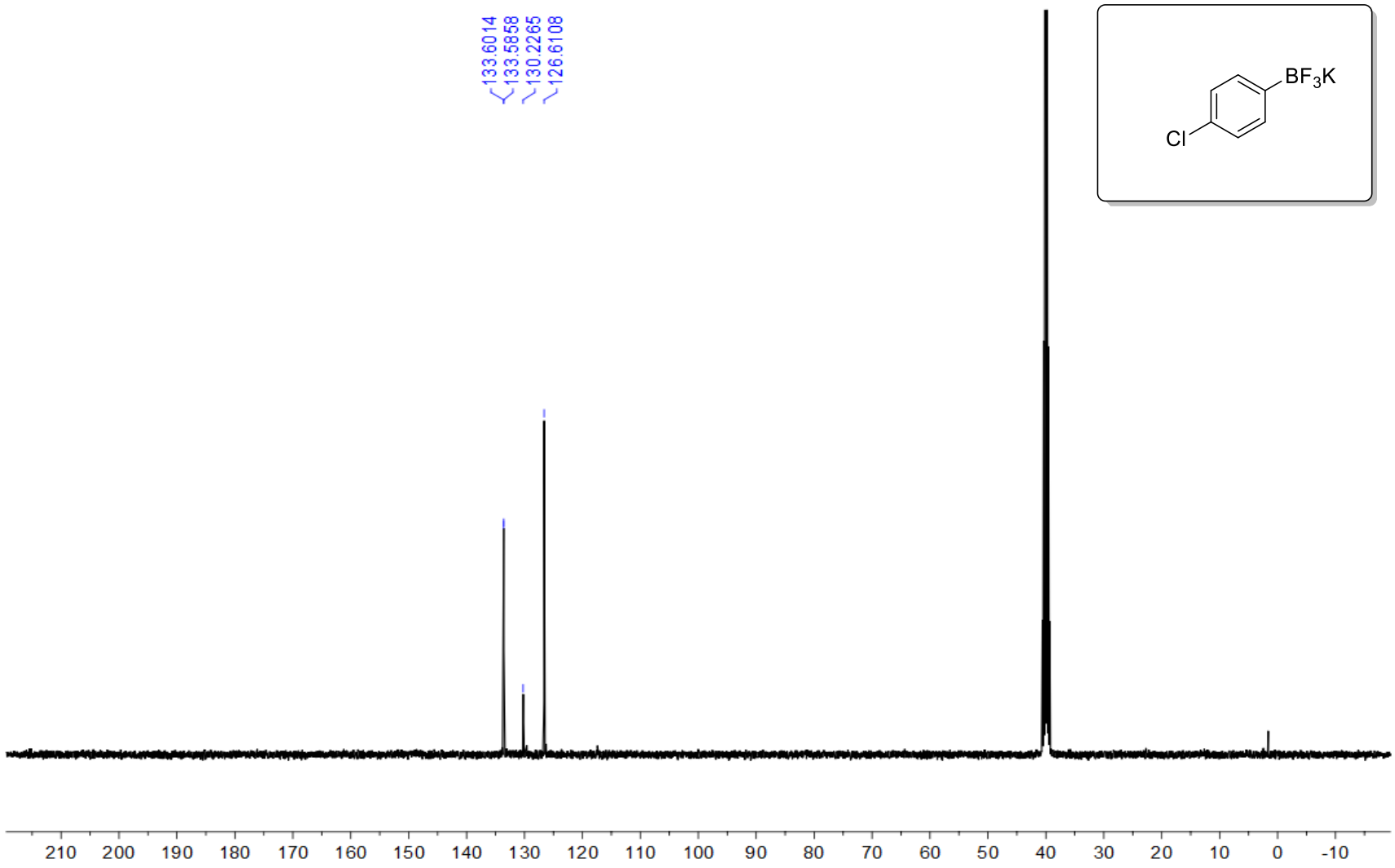
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **1i**



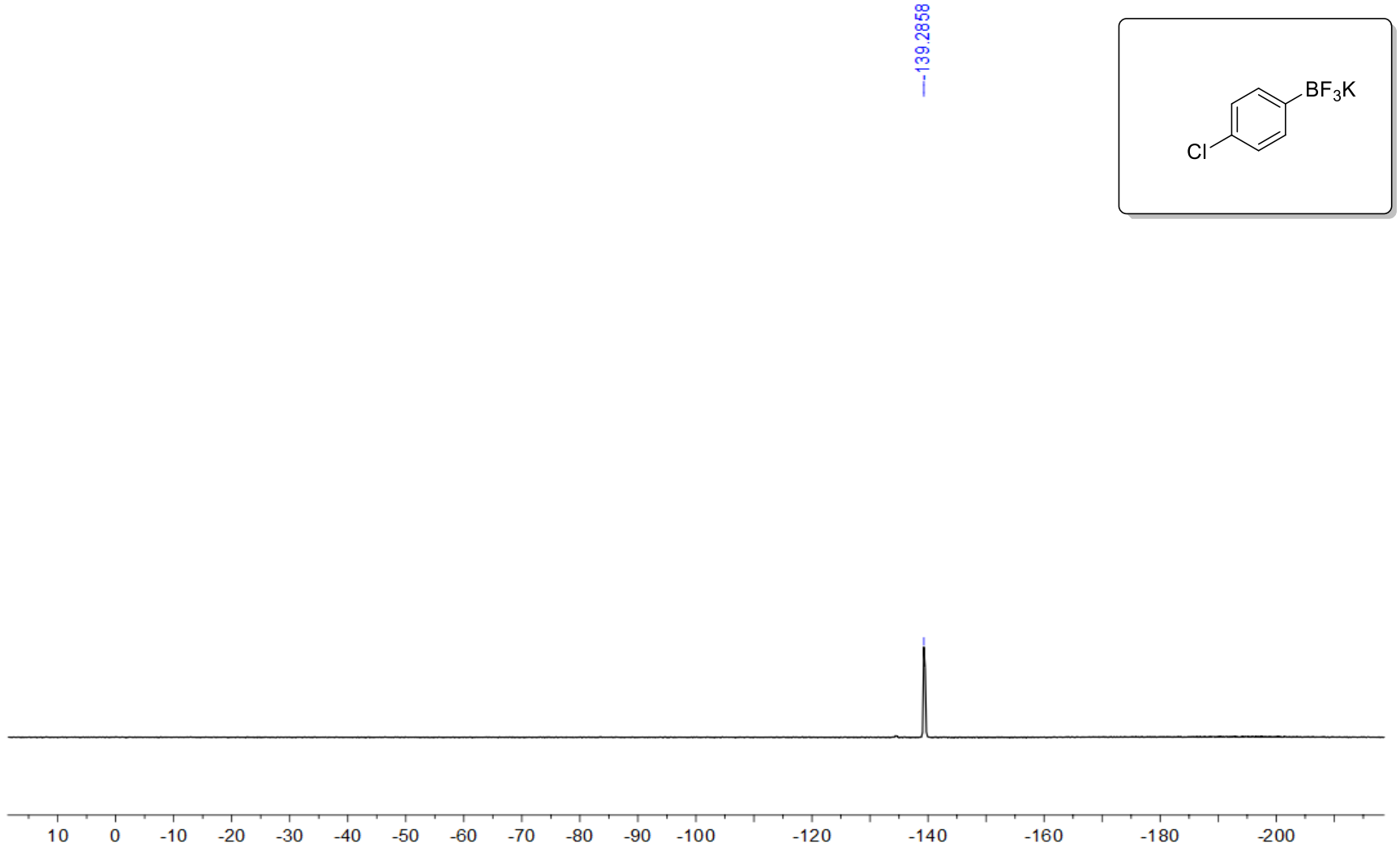
^{1}H NMR (400 MHz, [D6] DMSO): Compound **1j**



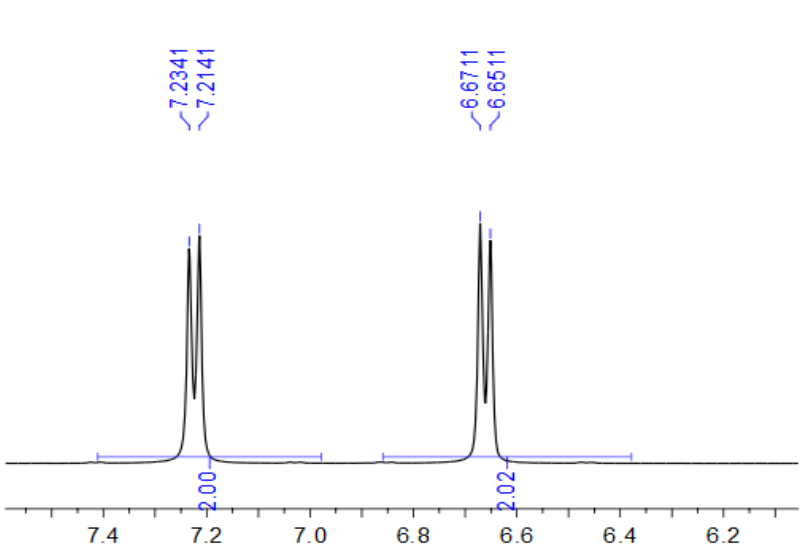
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **1j**

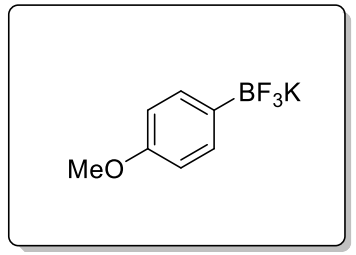


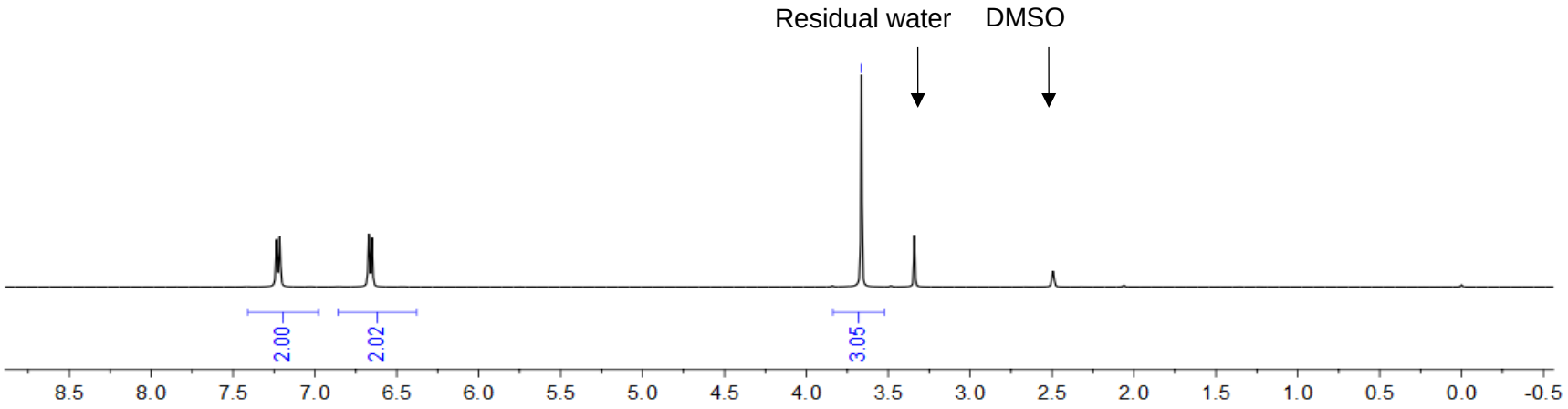
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **1j**



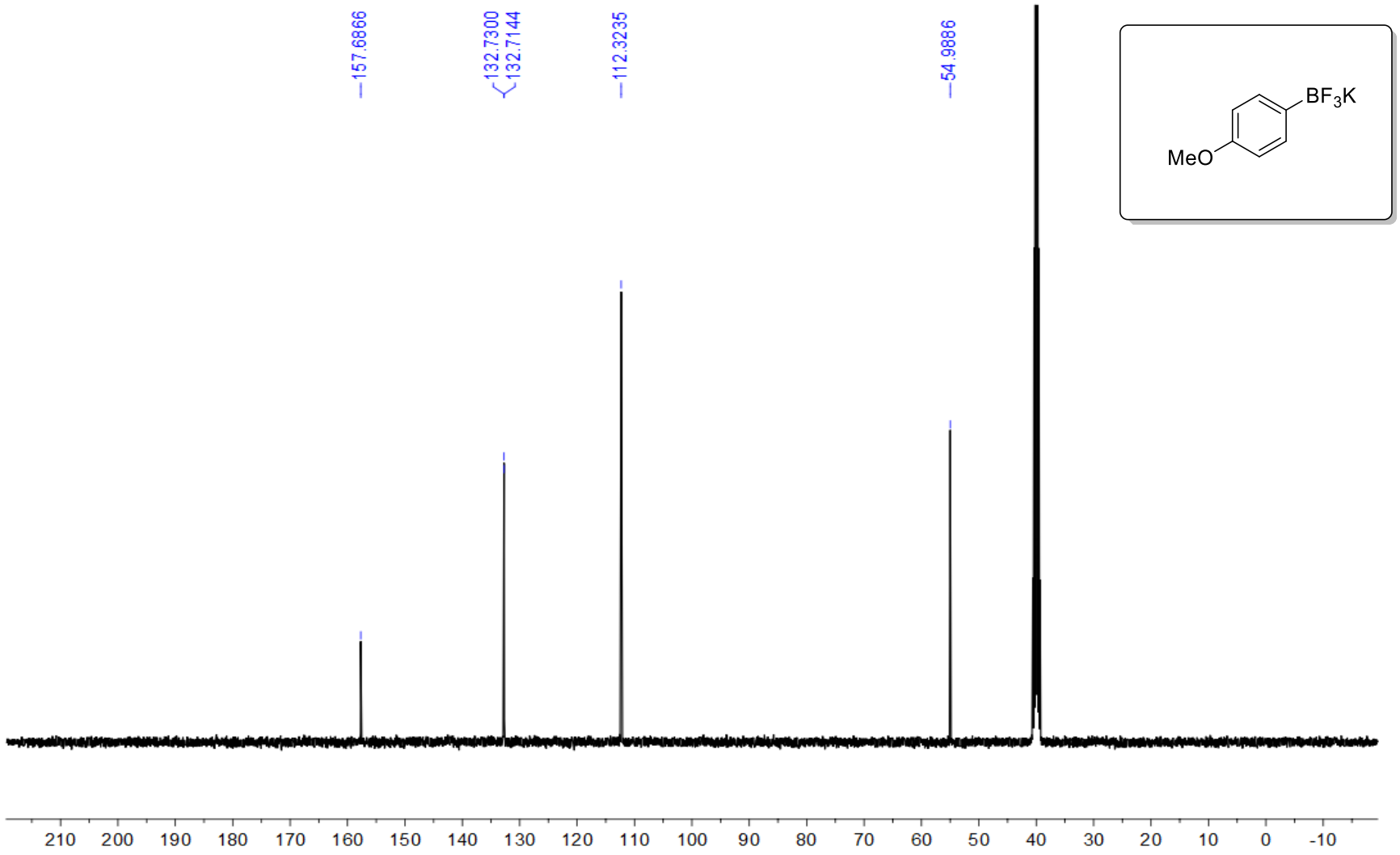
^{1}H NMR (400 MHz, [D6] DMSO): Compound **1k**



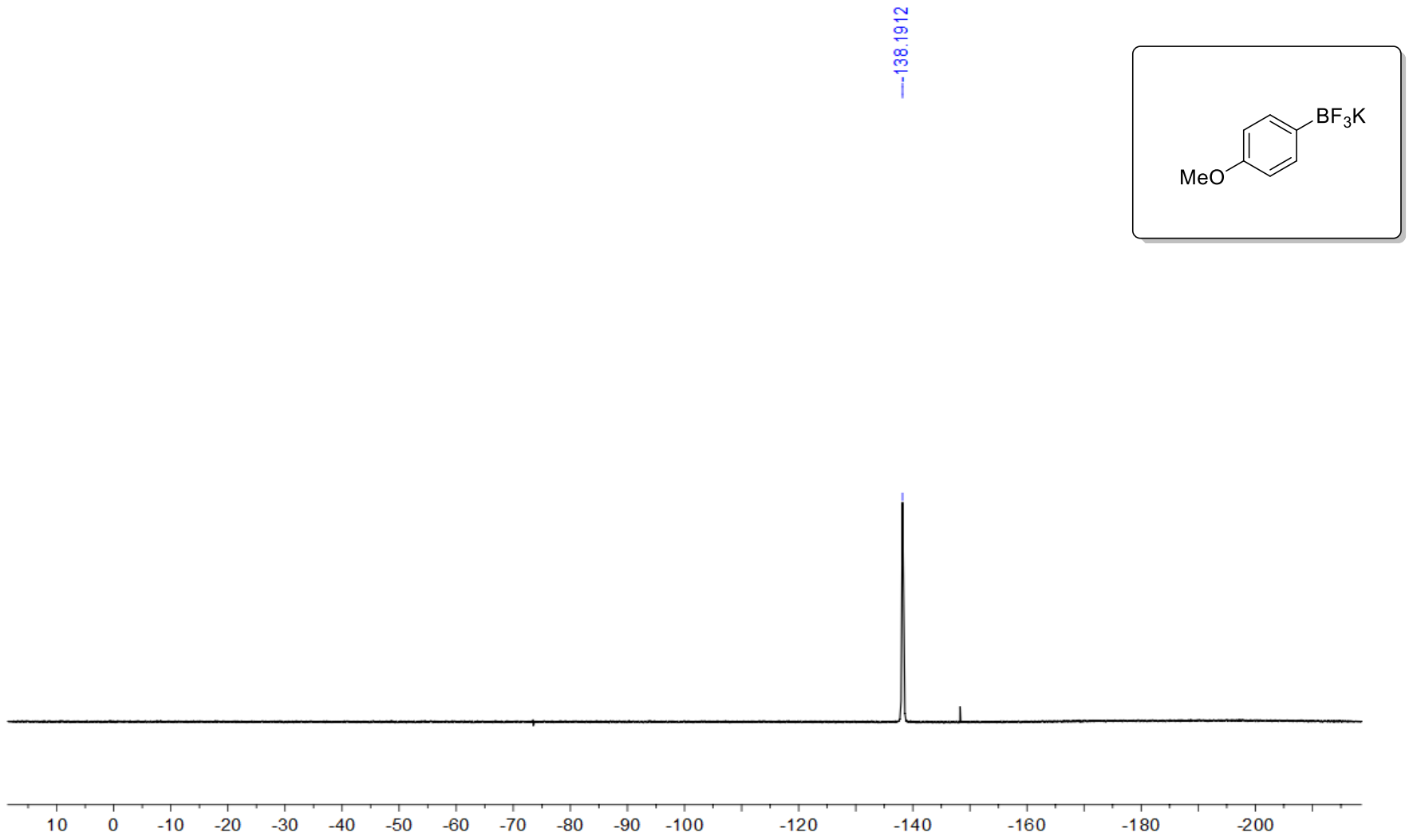




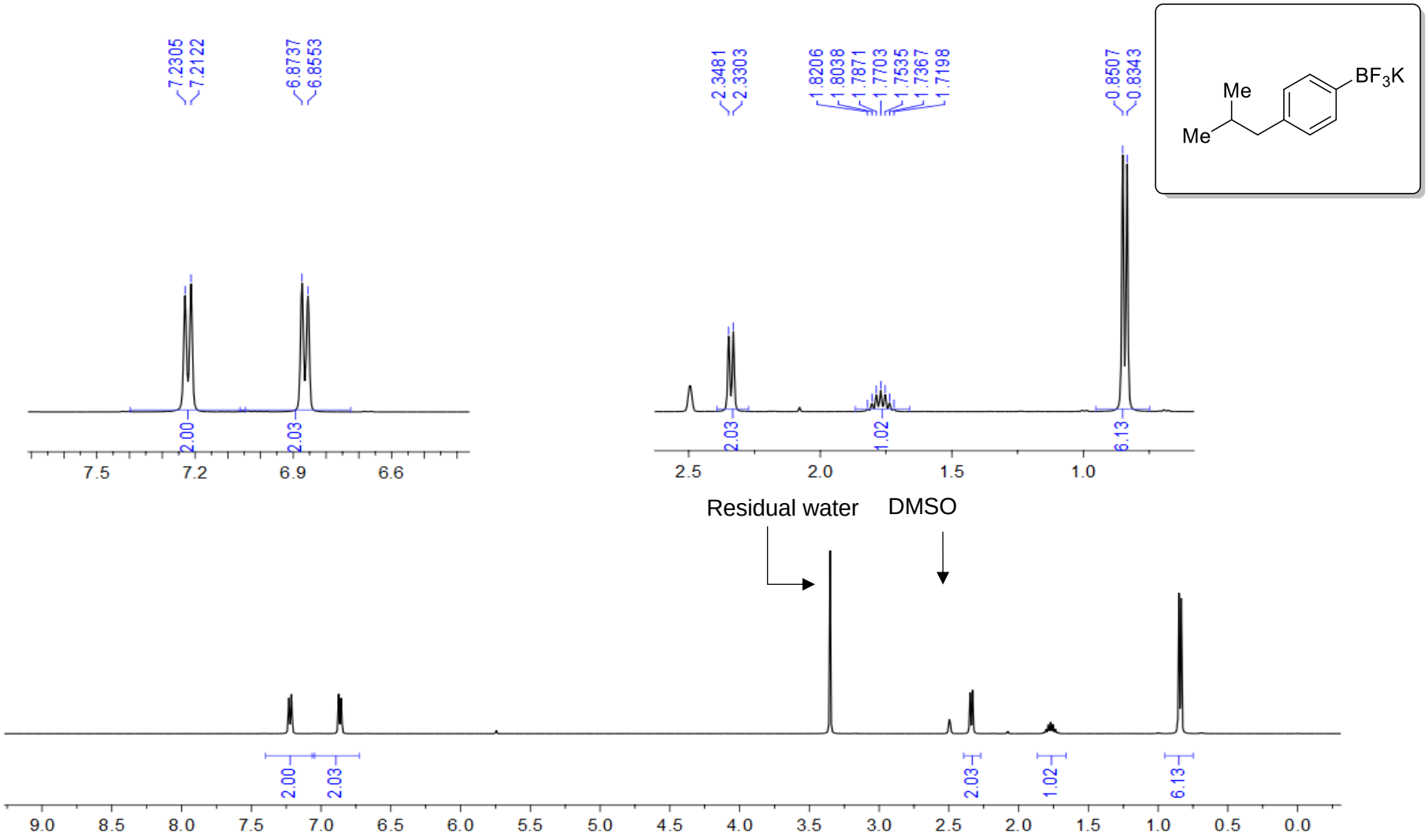
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **1k**



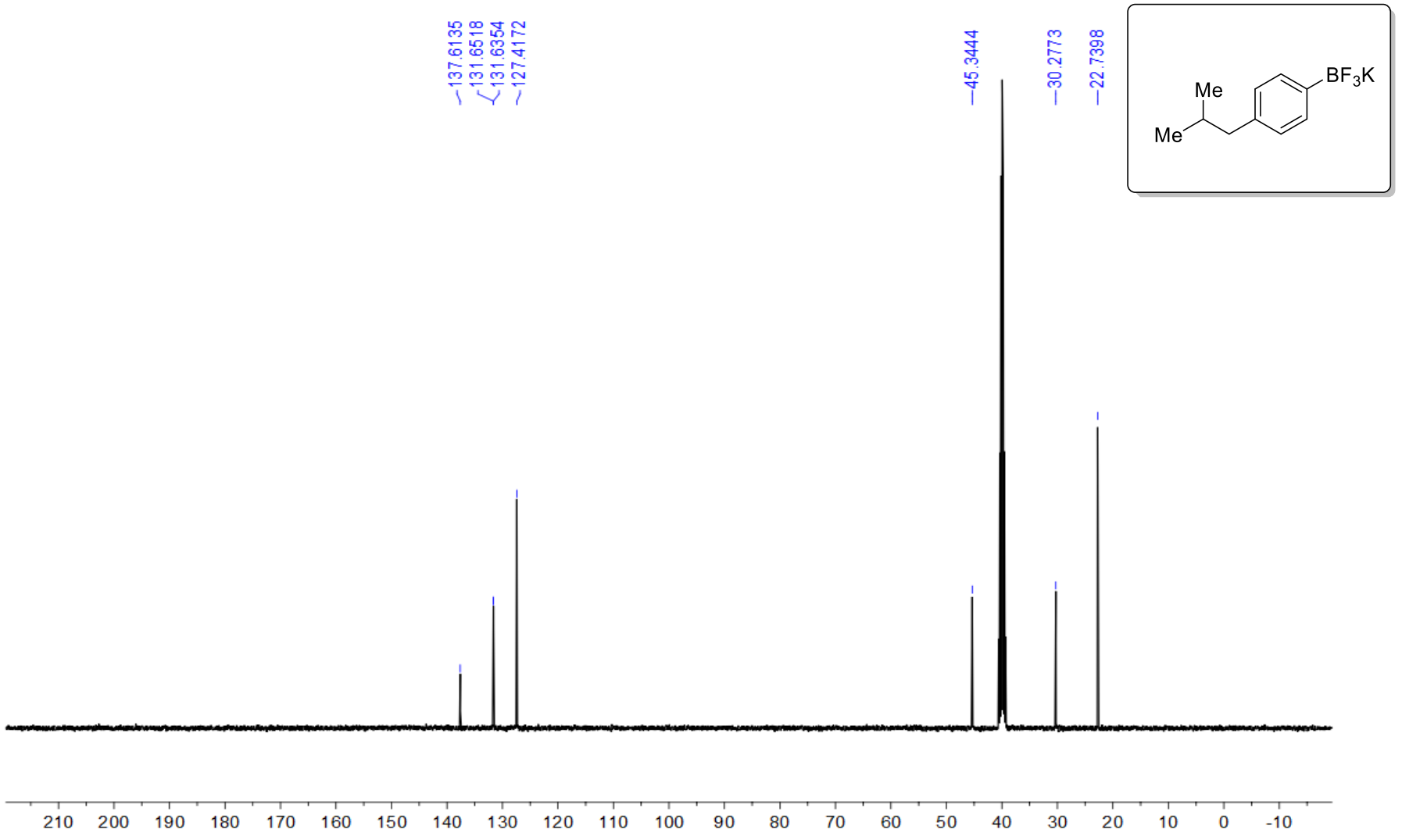
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **1k**



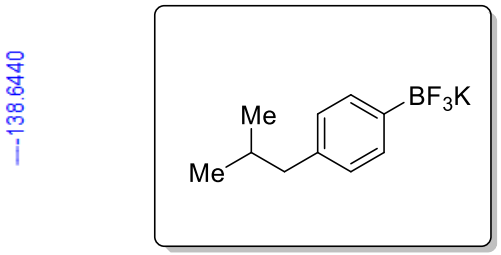
^{1}H NMR (400 MHz, [D6] DMSO): Compound **1m**

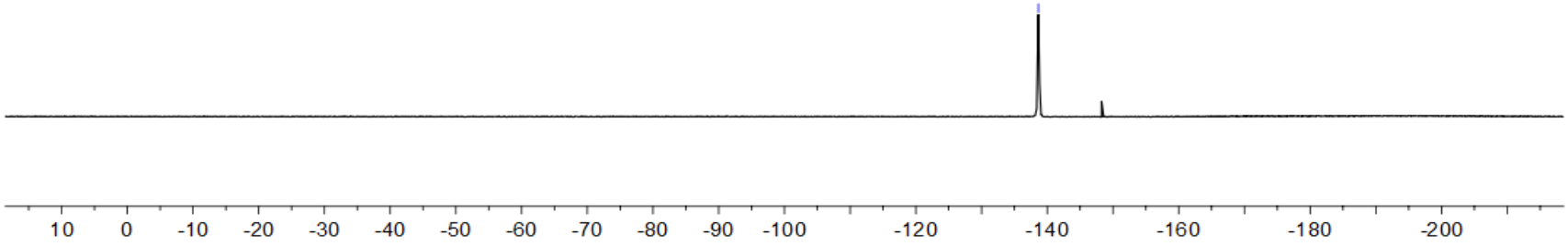


$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **1m**

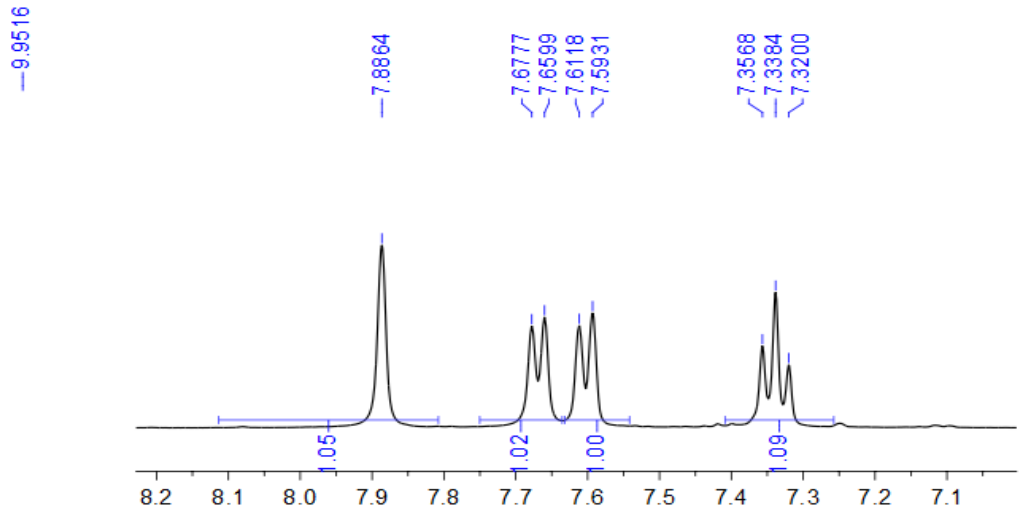


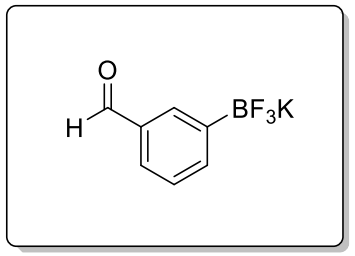
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **1m**

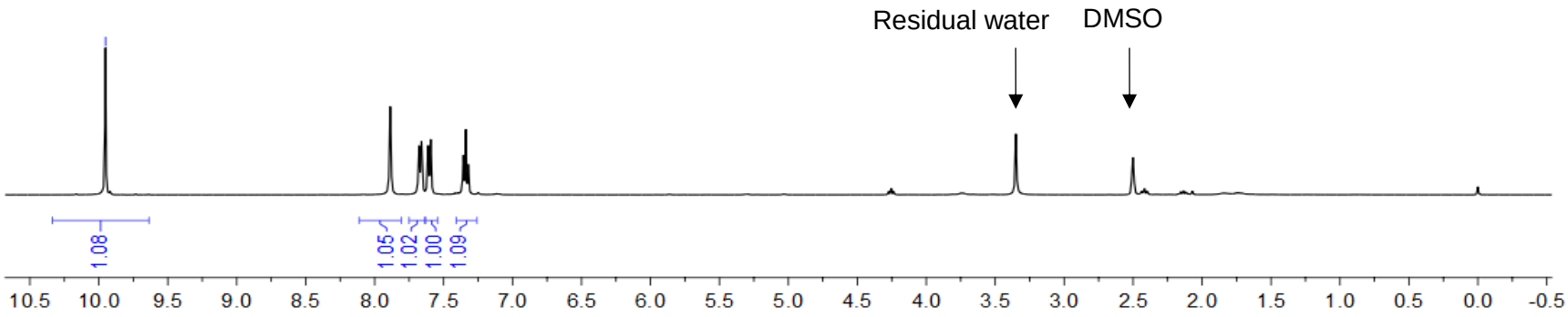




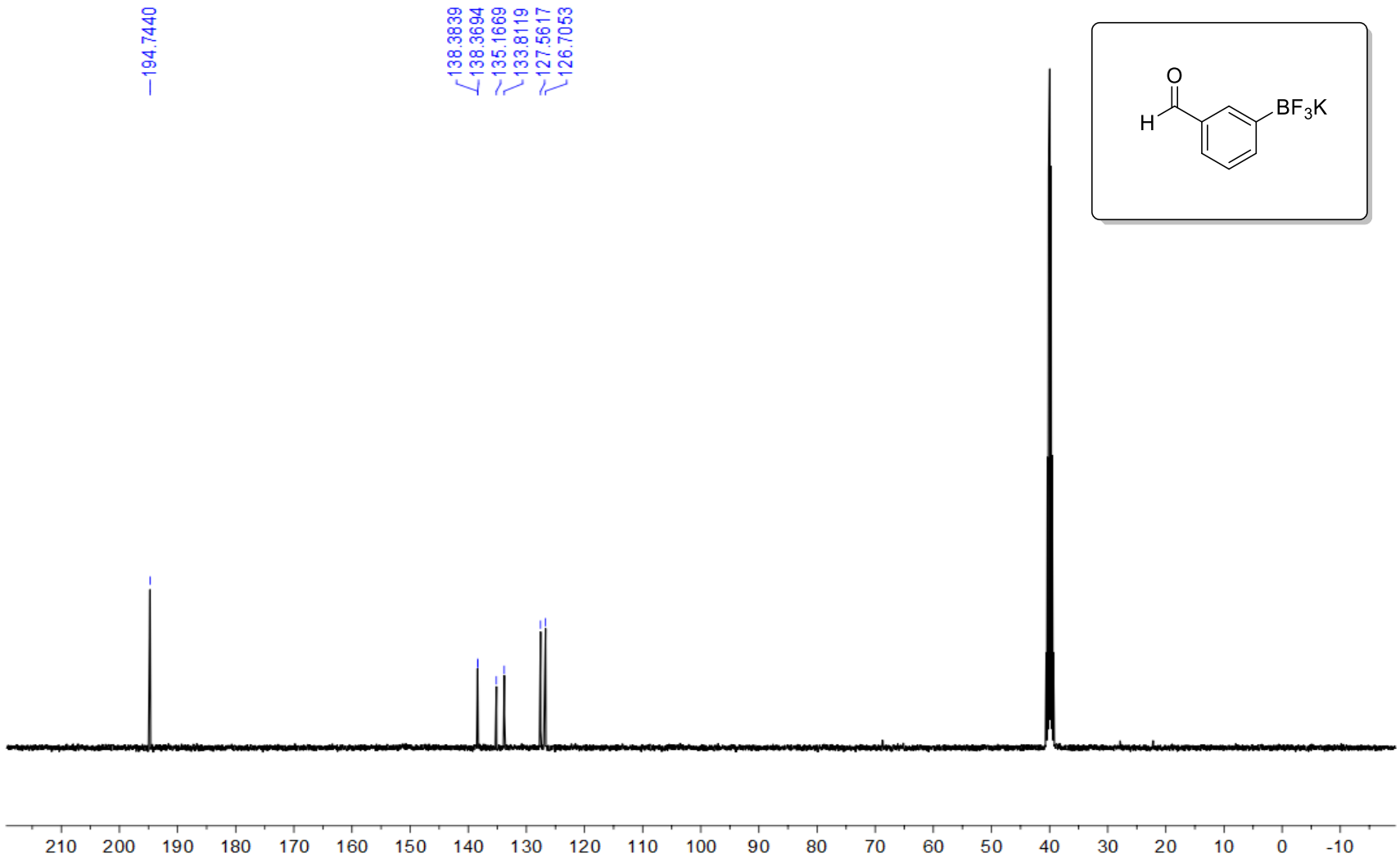
^{1}H NMR (400 MHz, [D6] DMSO): Compound **1n**



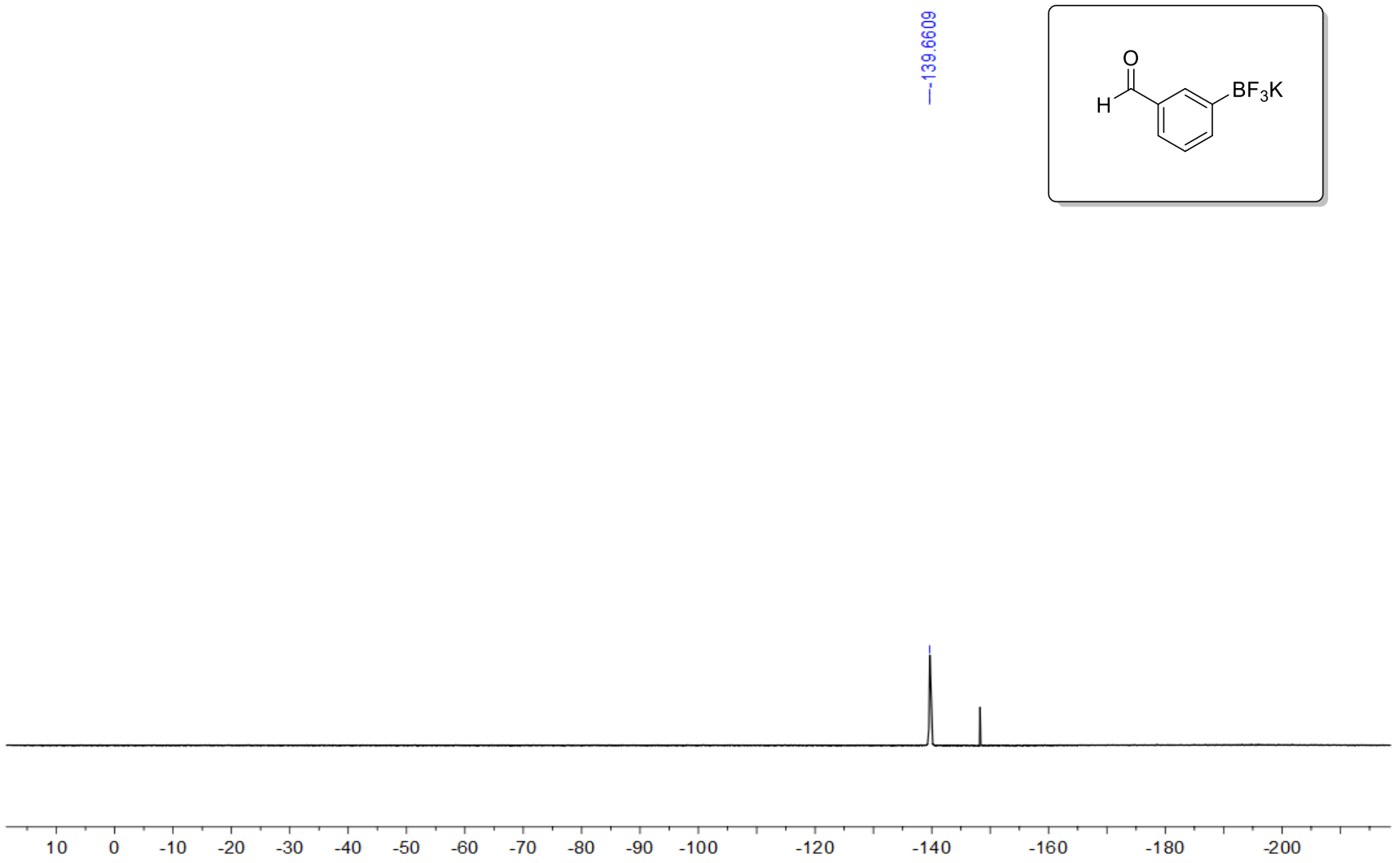




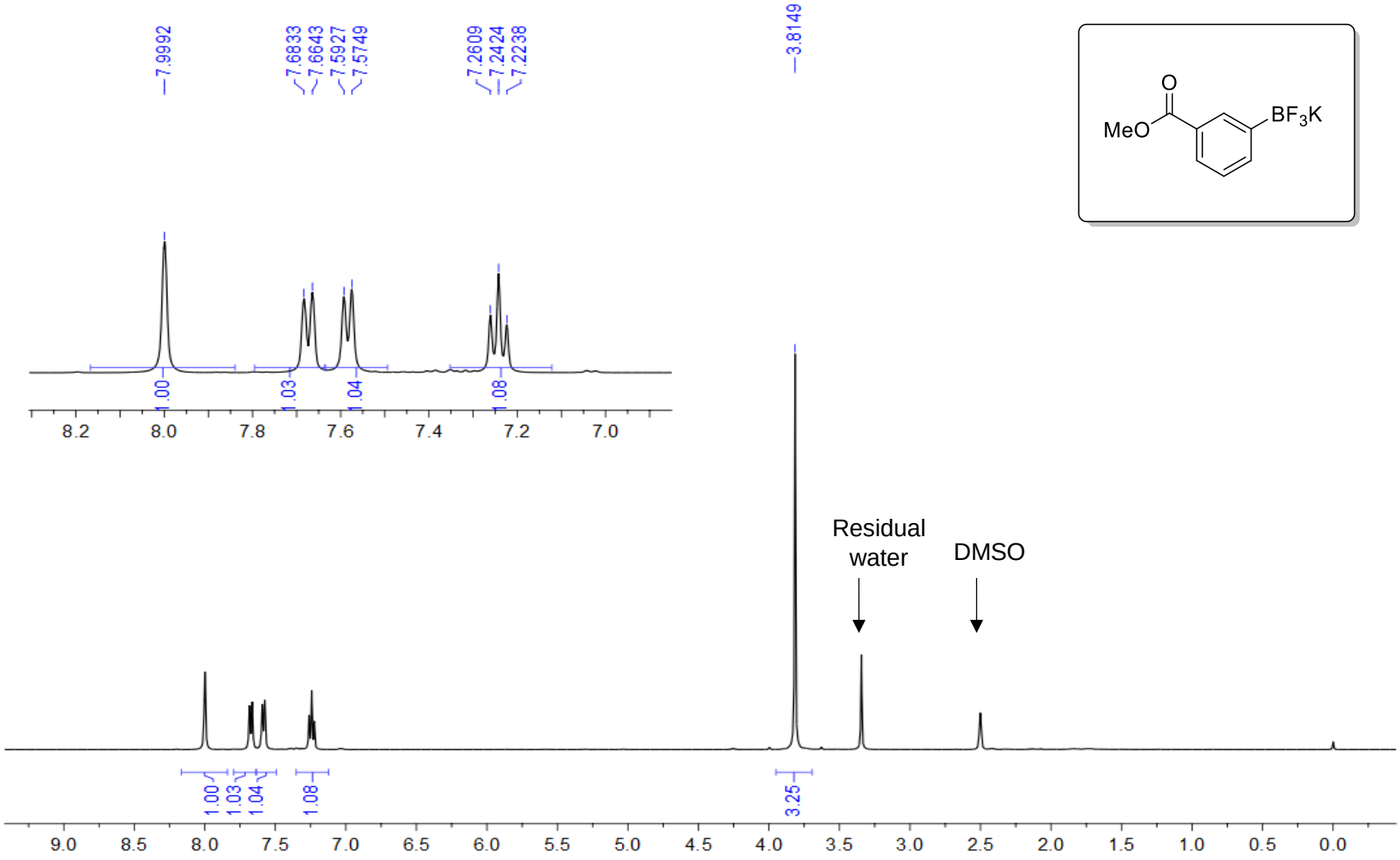
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **1n**



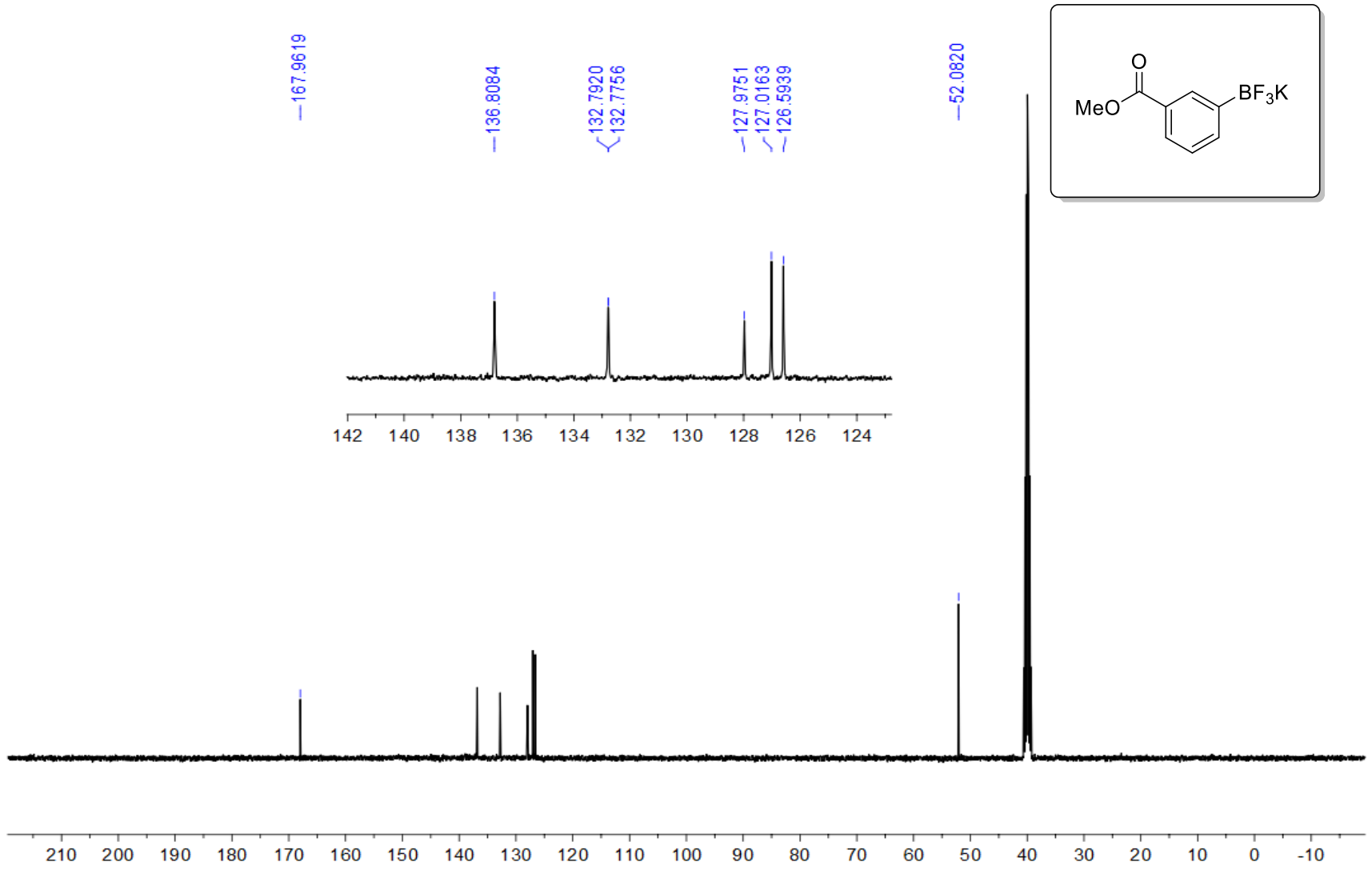
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **1n**



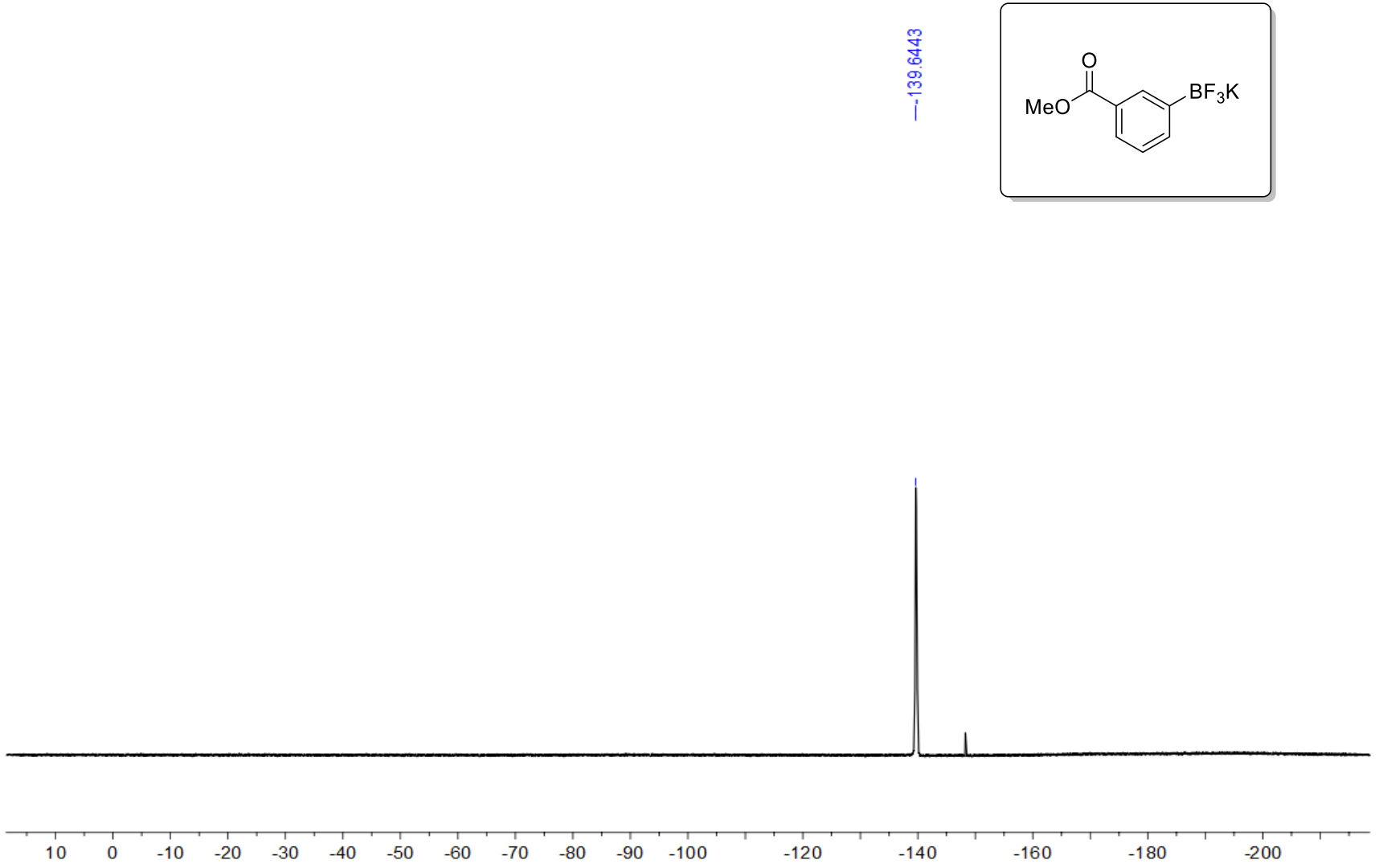
^{1}H NMR (400 MHz, [D6] DMSO): Compound **1o**



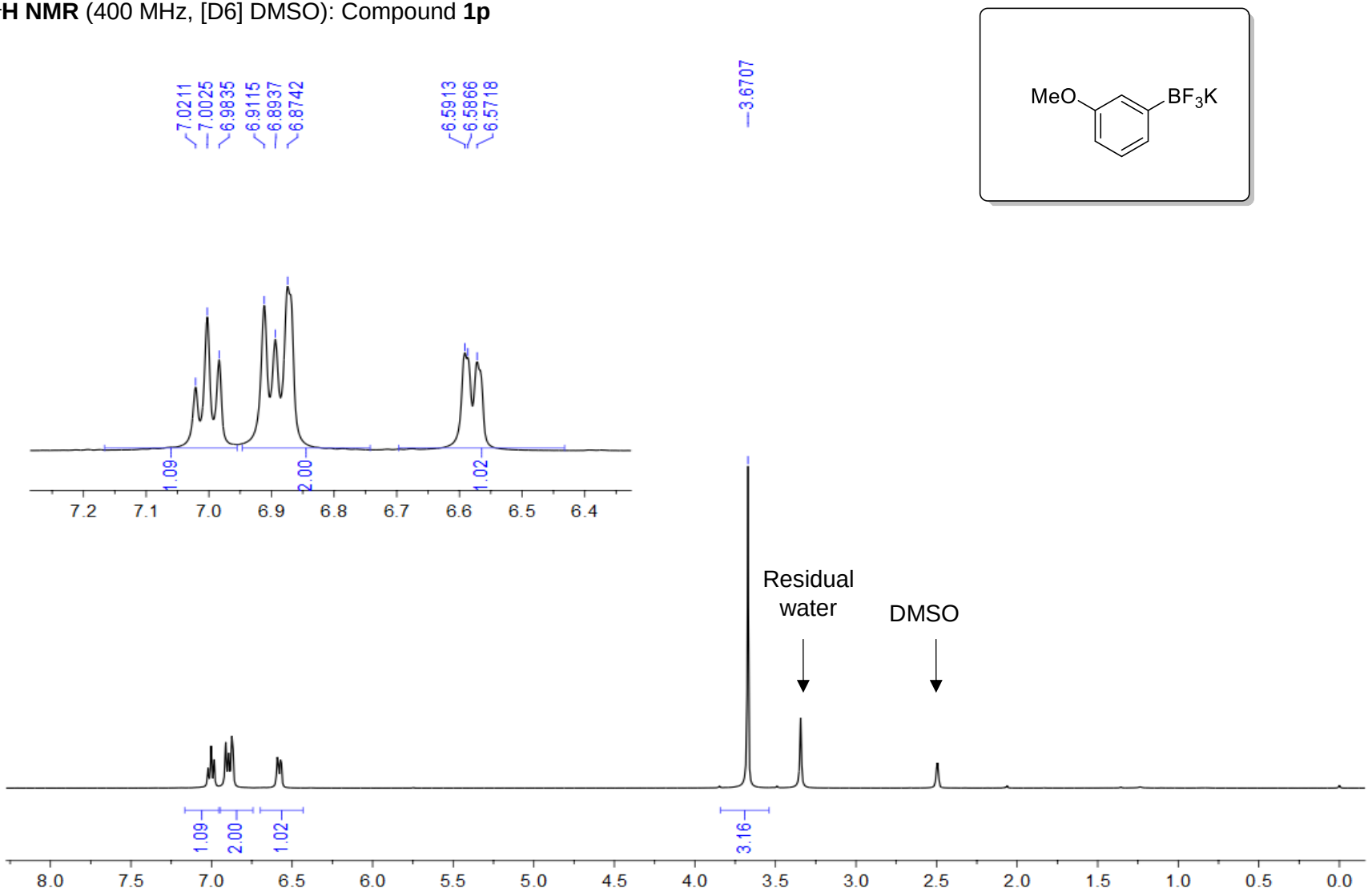
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **1o**



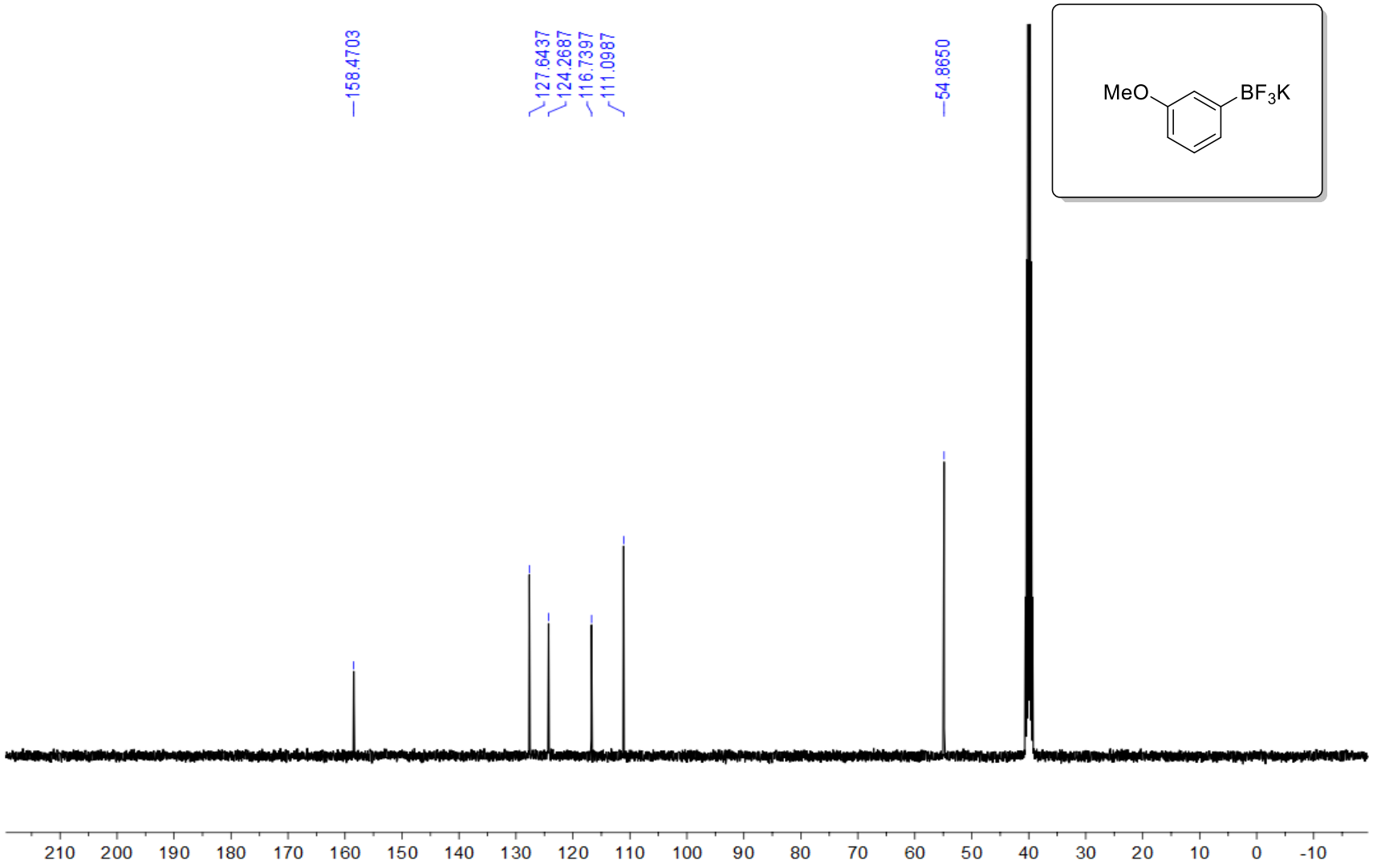
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **1o**



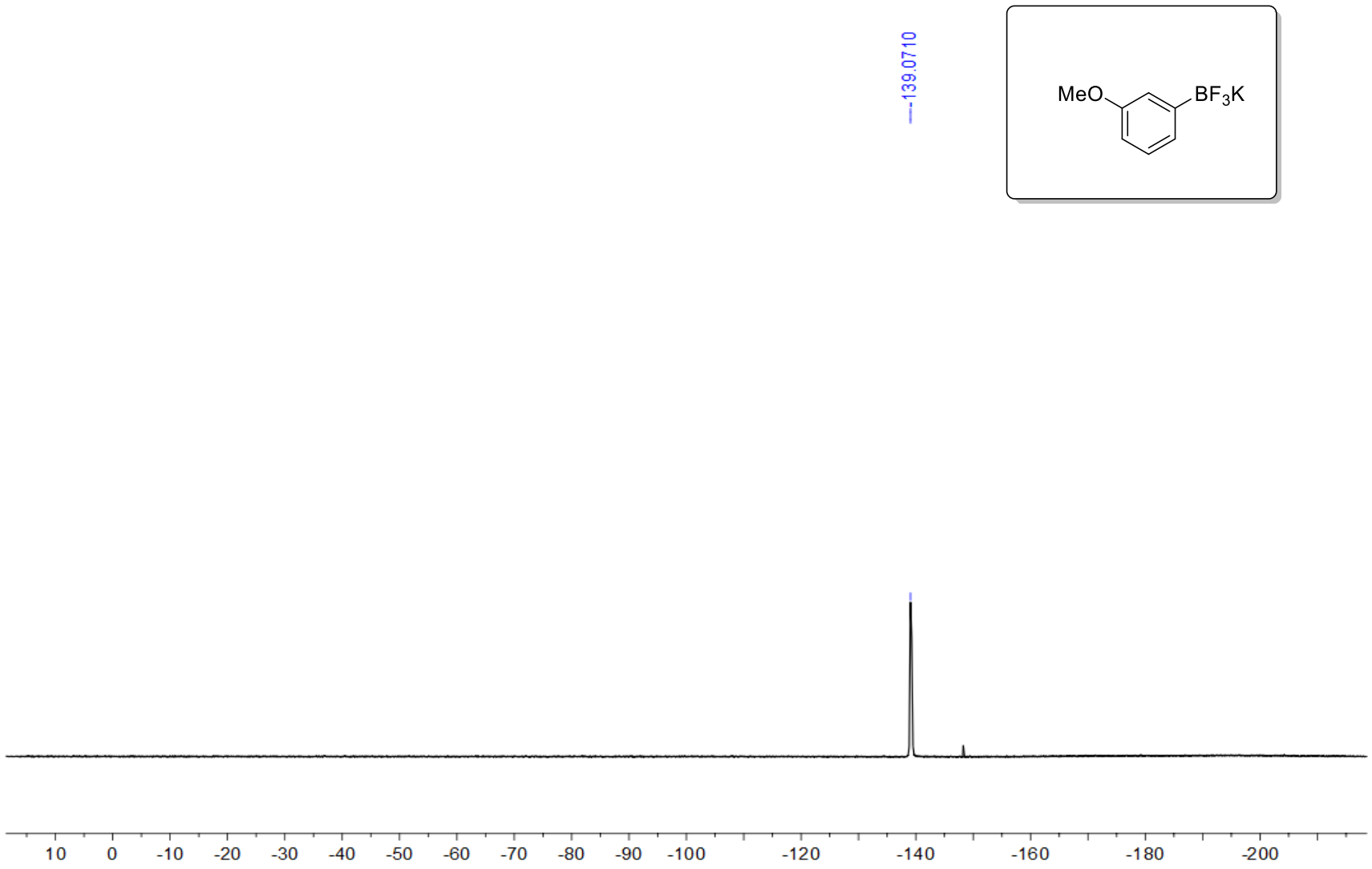
^{1}H NMR (400 MHz, [D6] DMSO): Compound **1p**



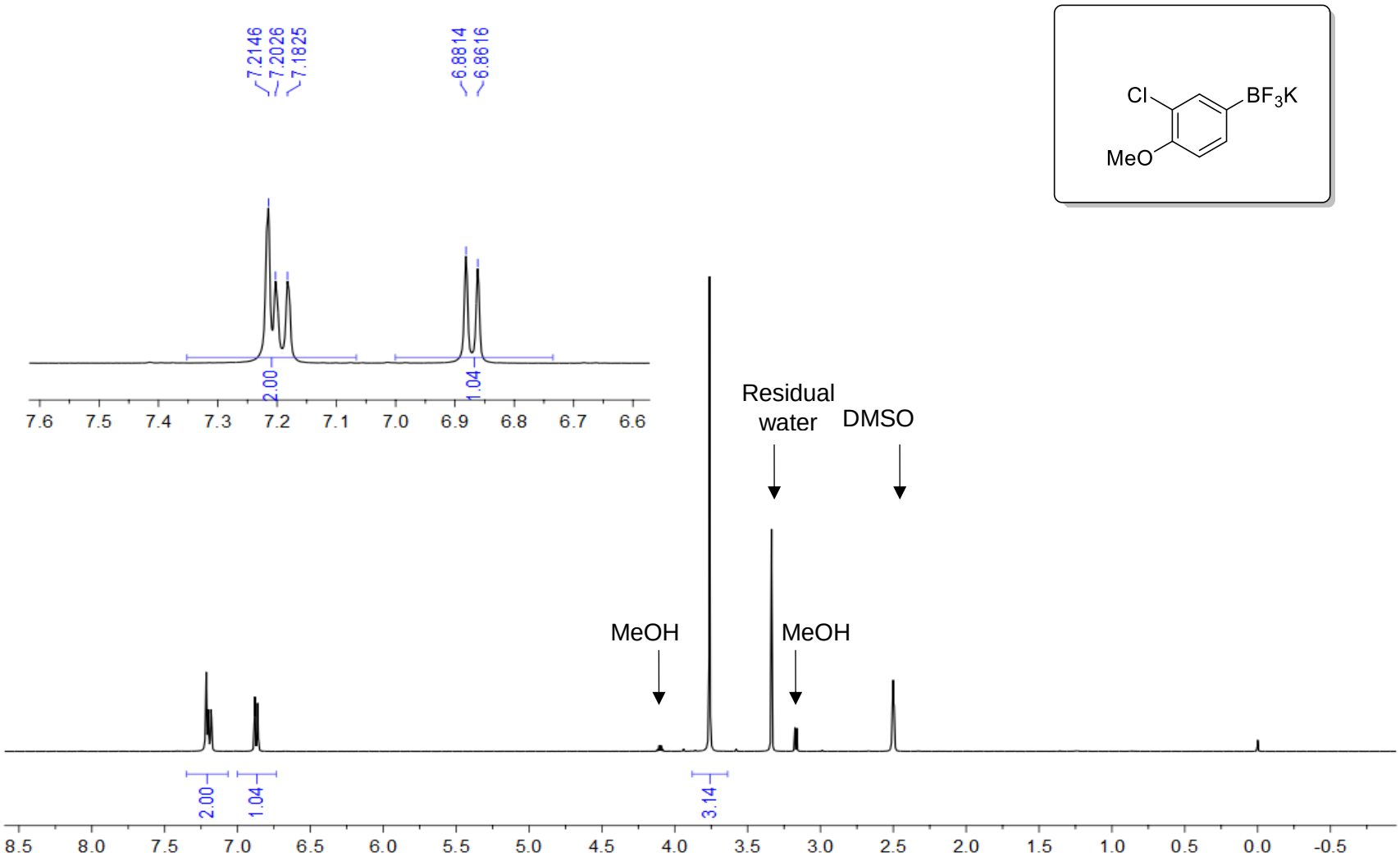
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **1p**



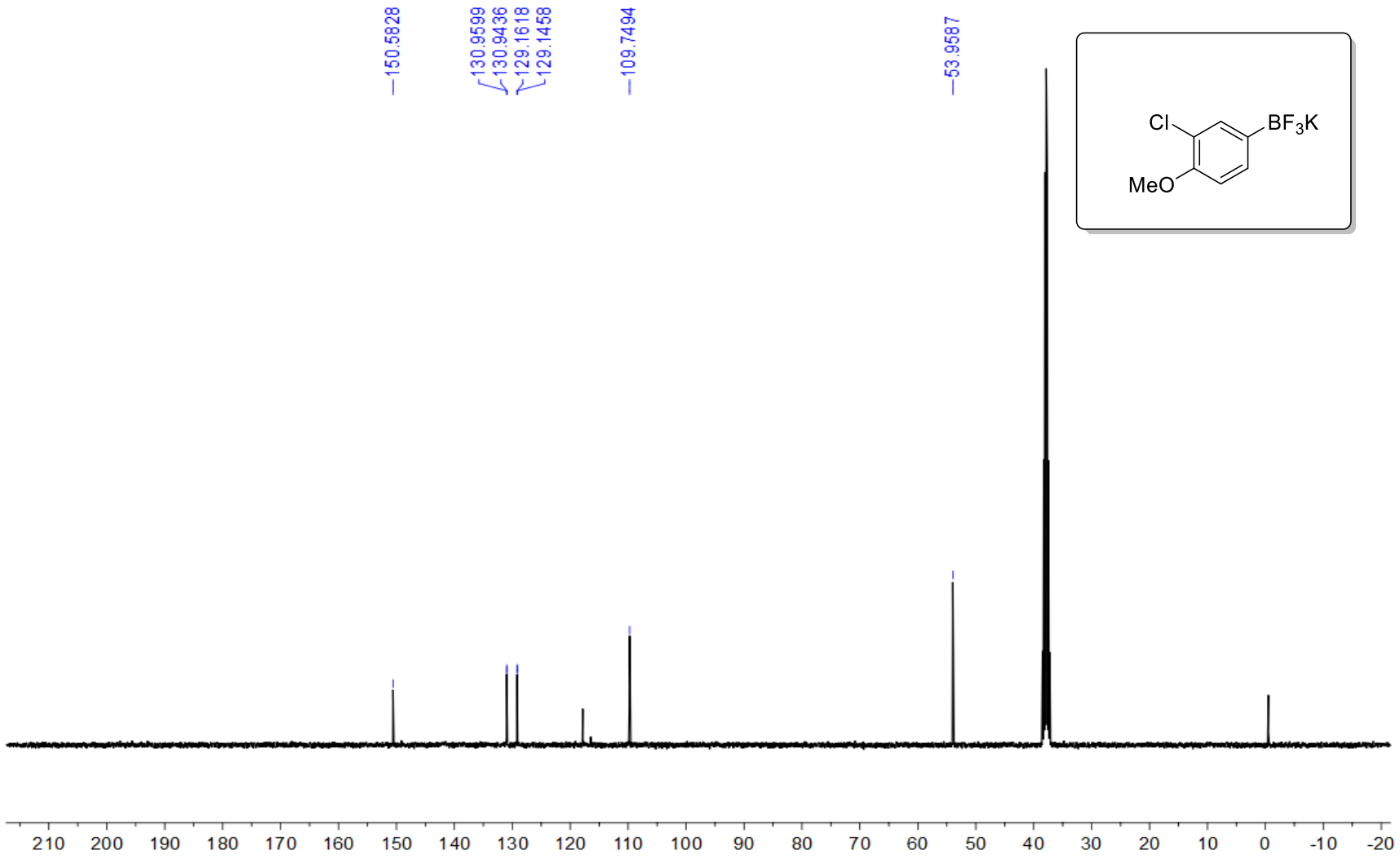
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **1p**



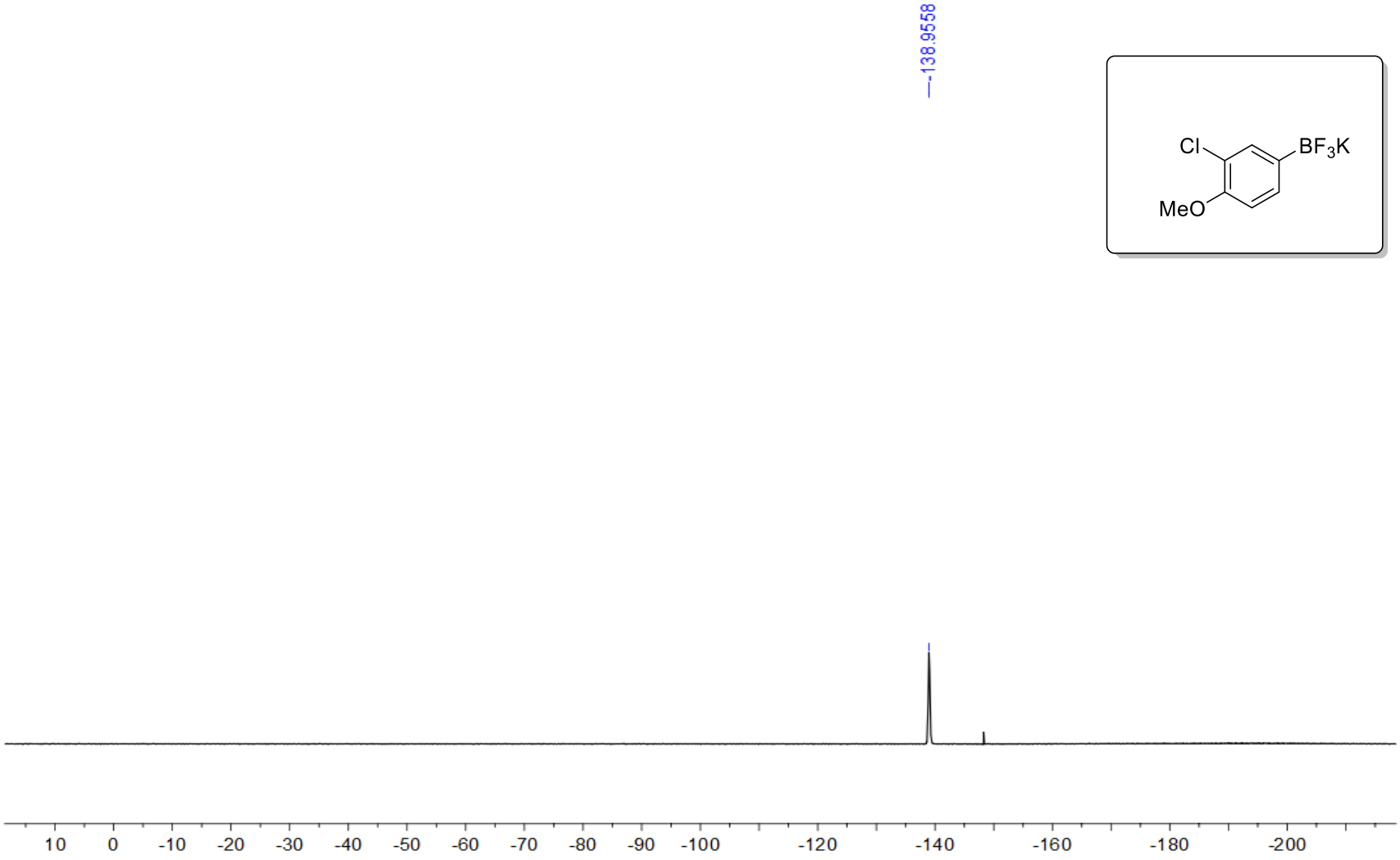
^{1}H NMR (400 MHz, [D6] DMSO): Compound **1q**



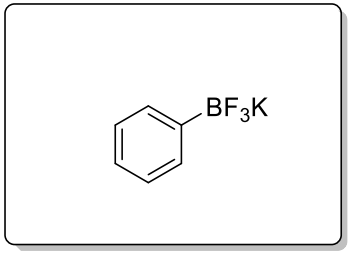
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **1q**



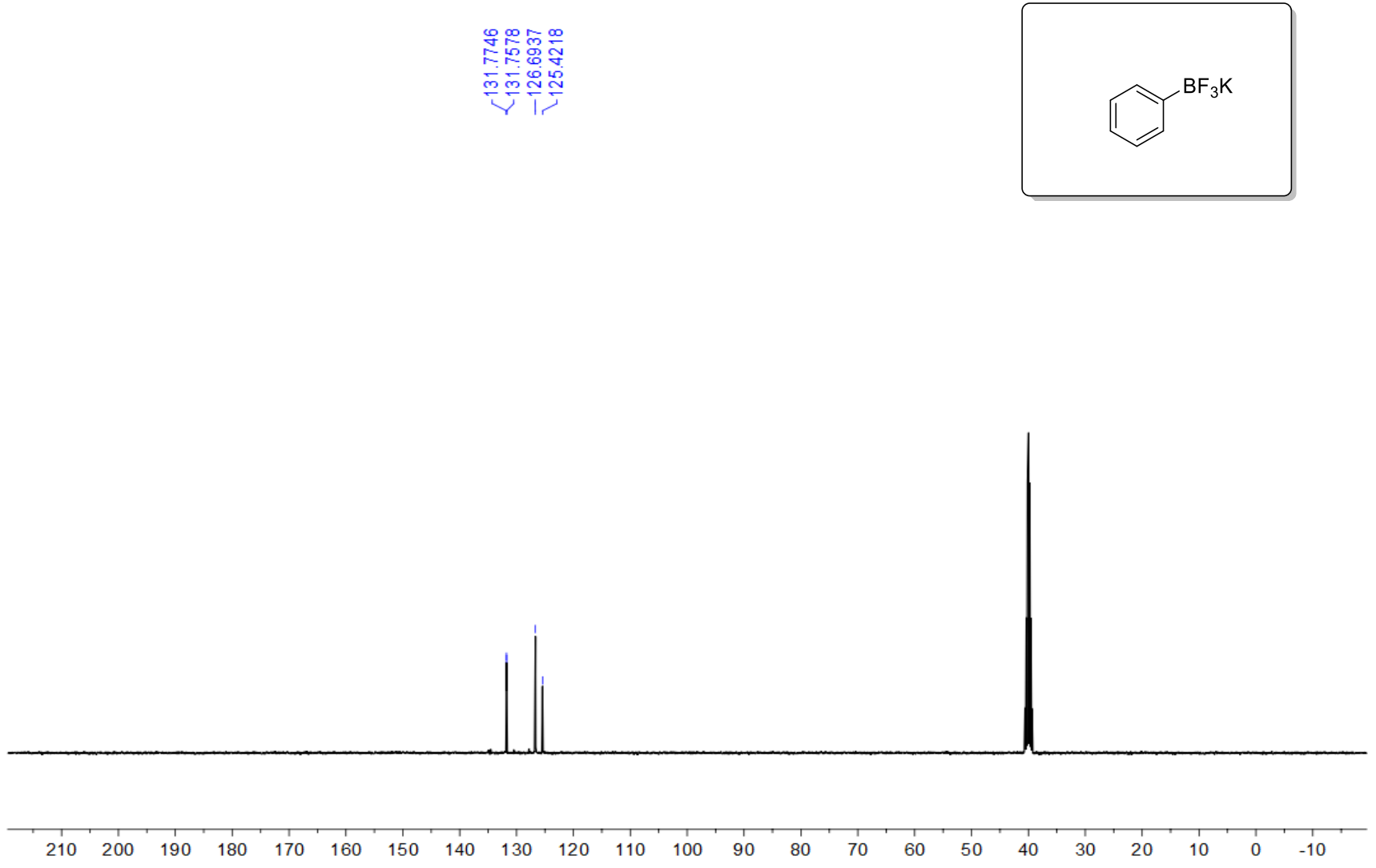
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **1q**



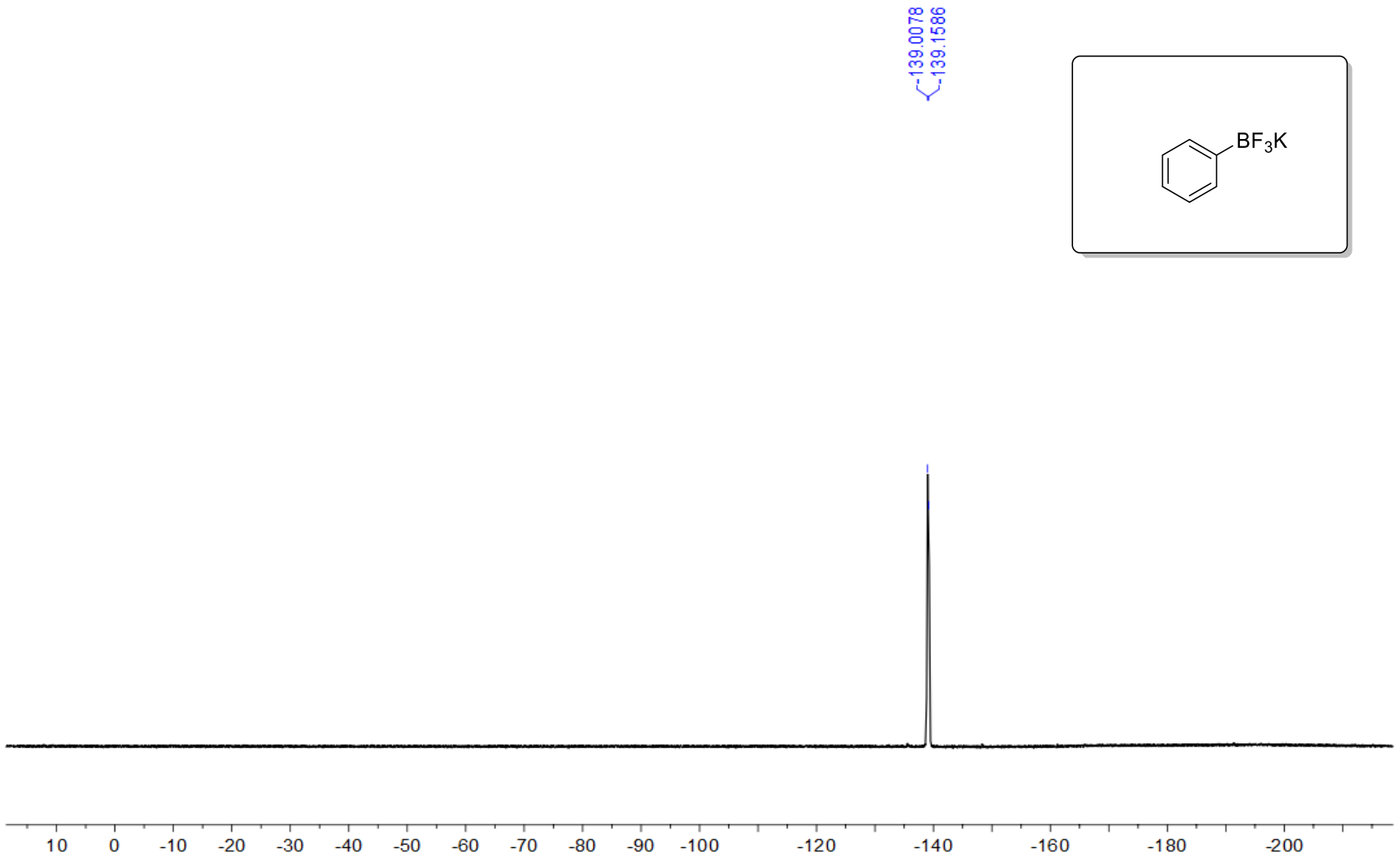
^{1}H NMR (400 MHz, [D6] DMSO): Compound **1r**



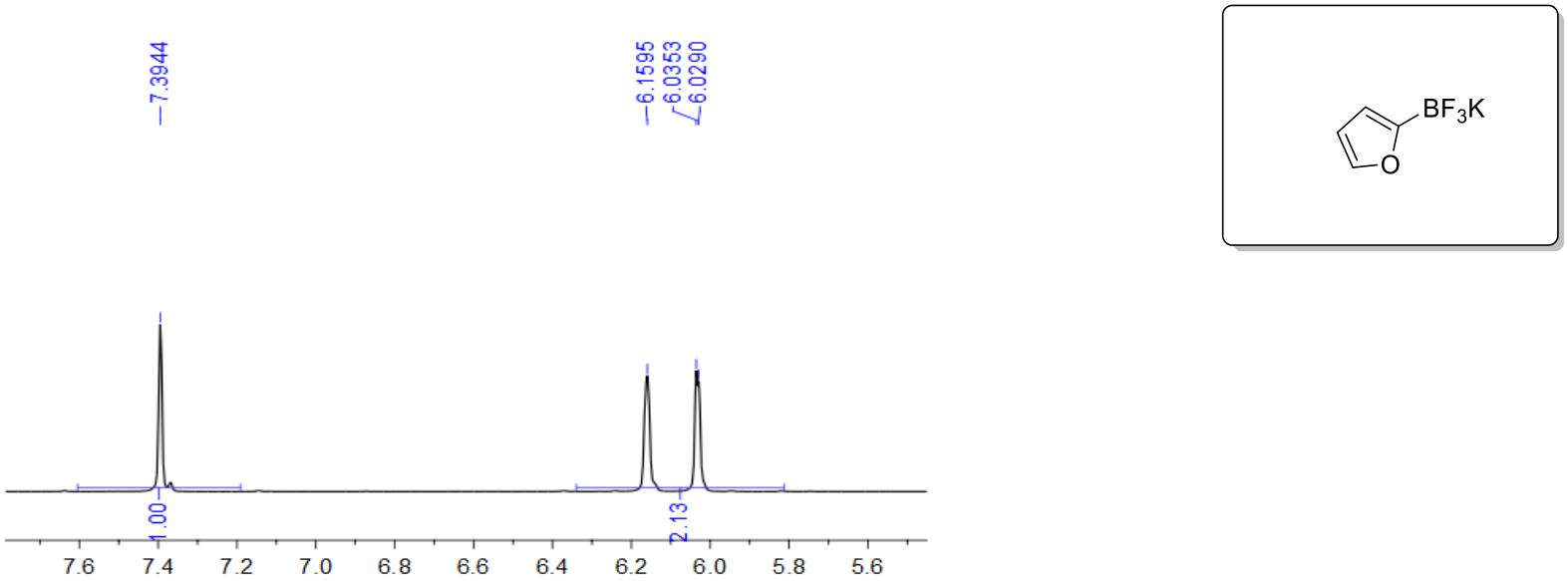
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **1r**

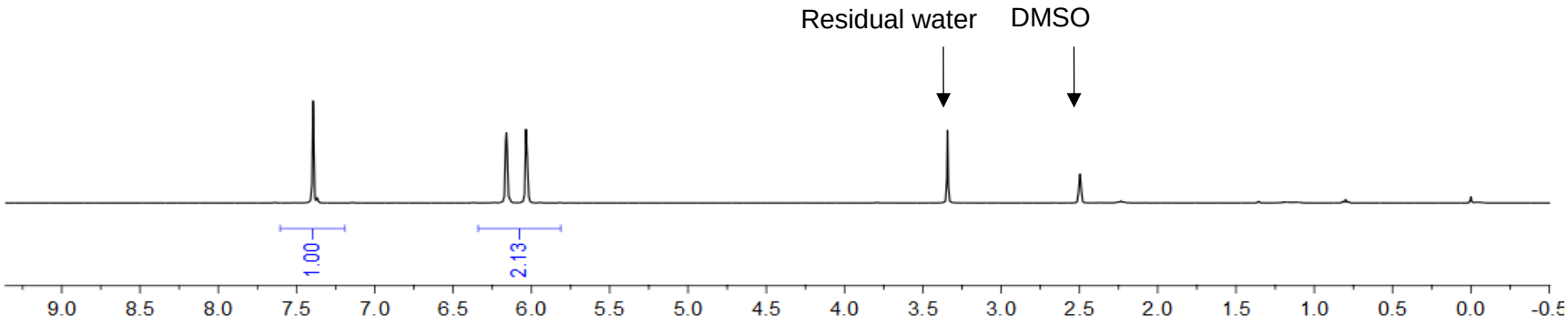


$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **1r**

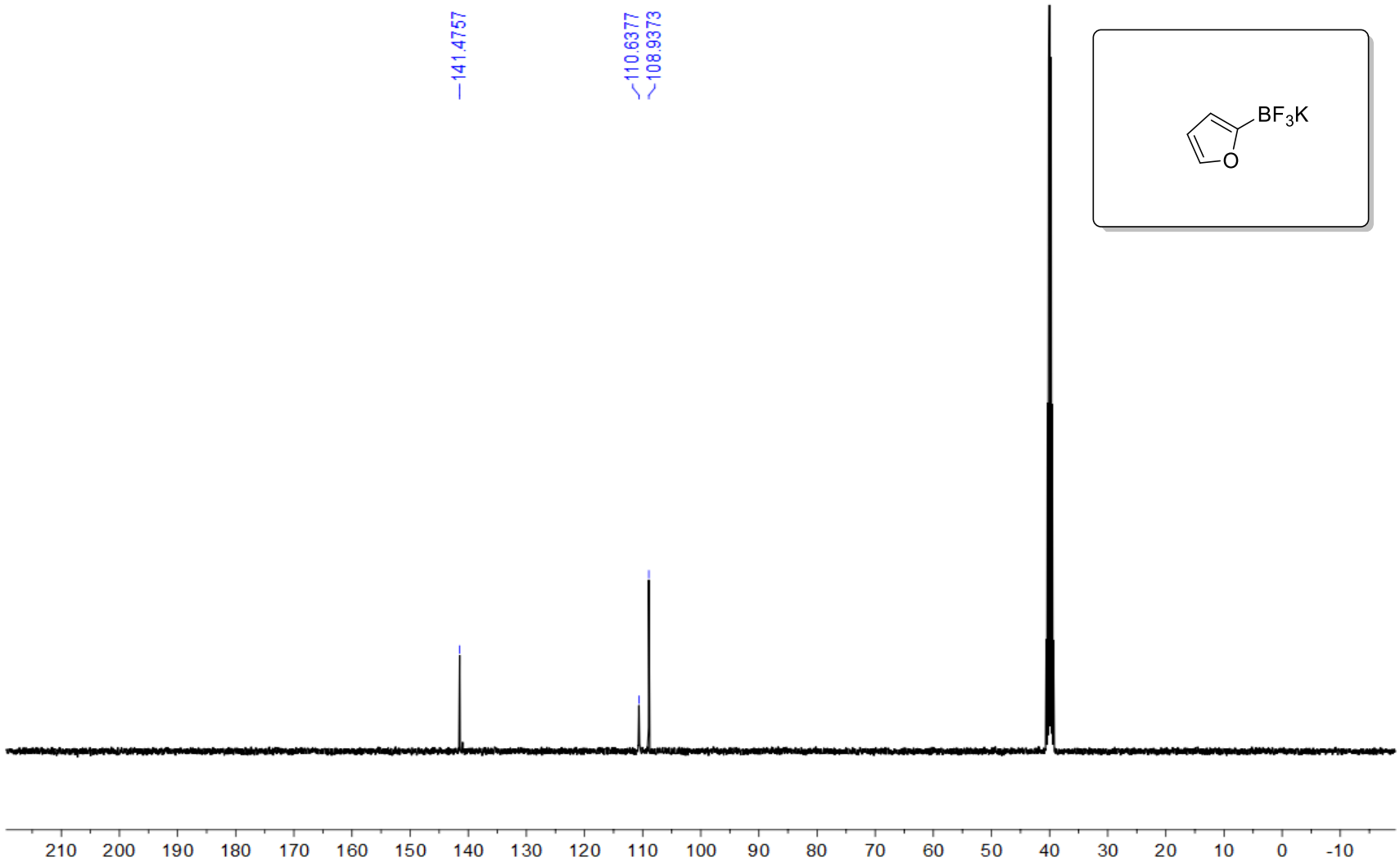


^{1}H NMR (400 MHz, [D6] DMSO): Compound **1s**

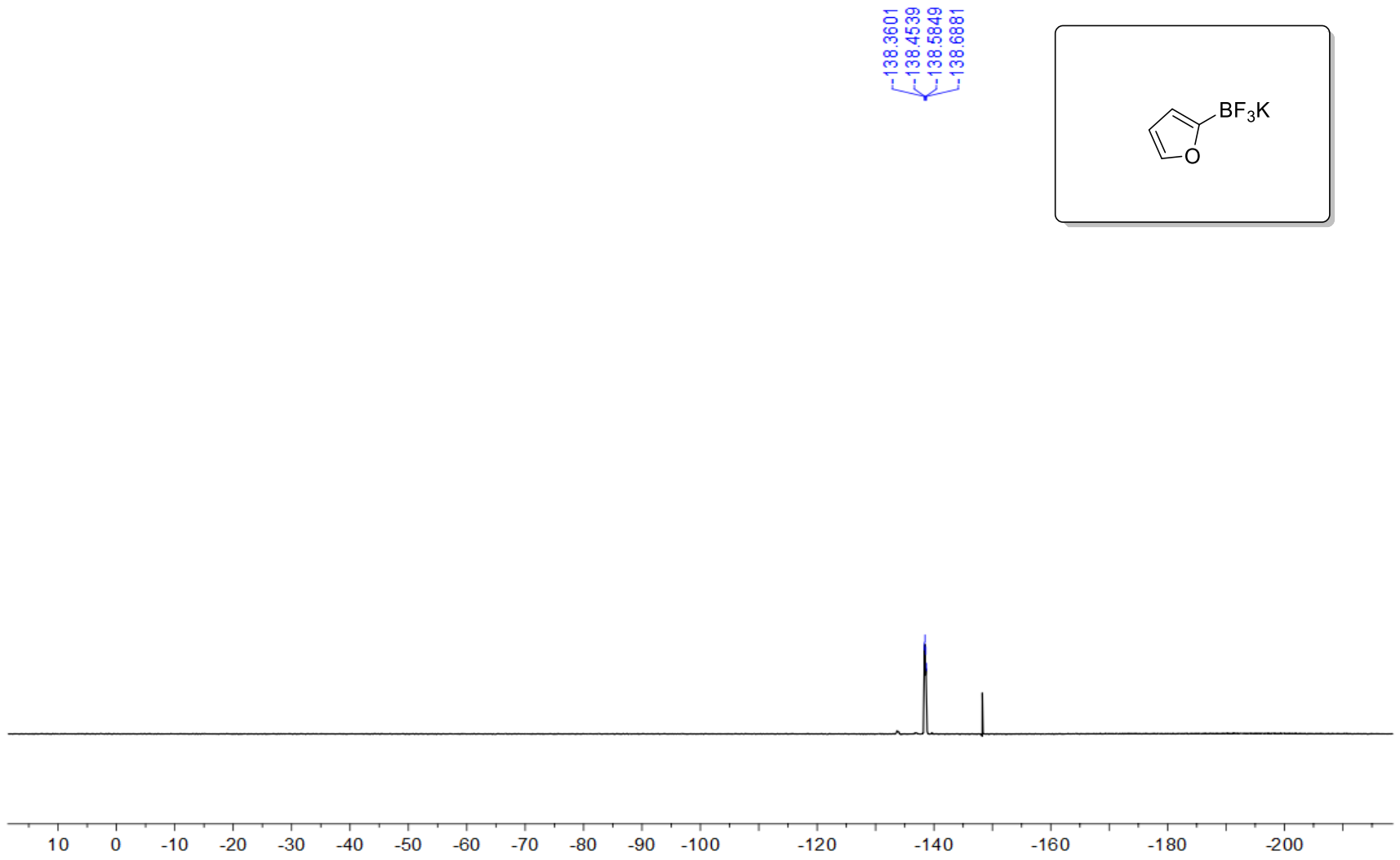




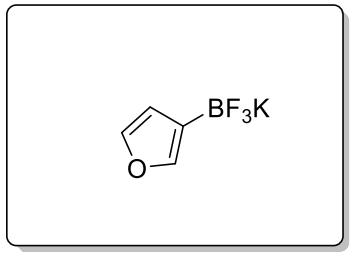
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **1s**



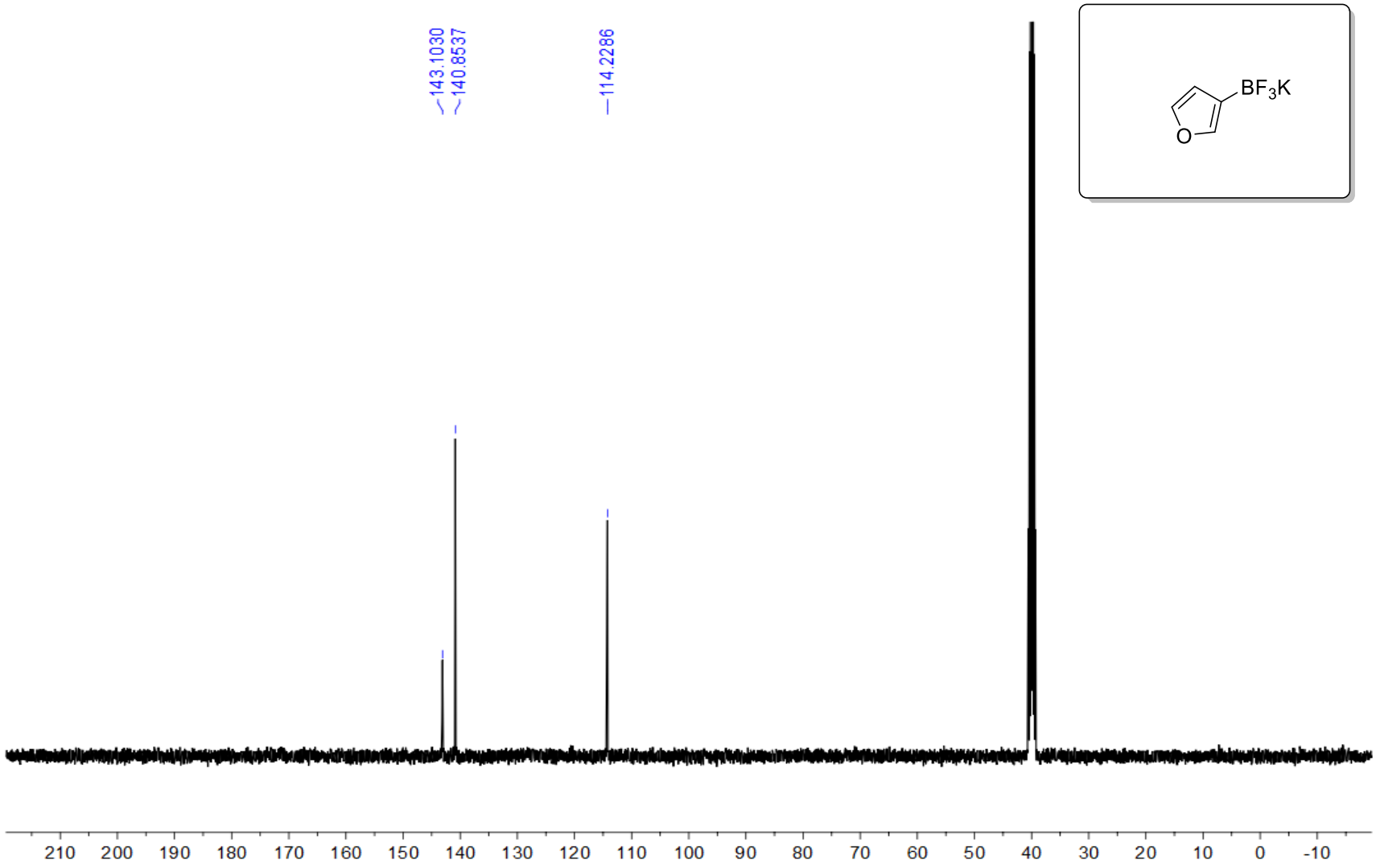
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **1s**



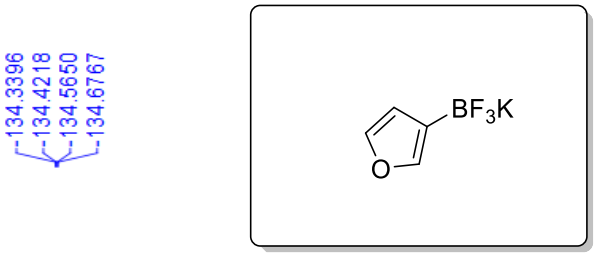
^{1}H NMR (400 MHz, [D6] DMSO): Compound **1t**

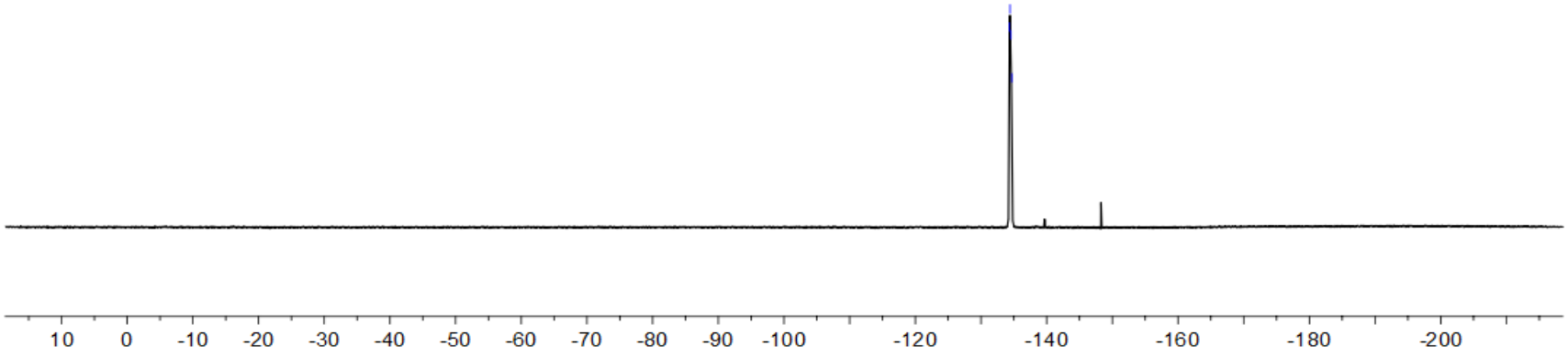


$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **1t**

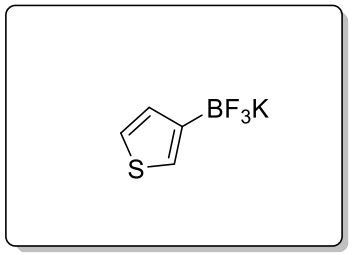


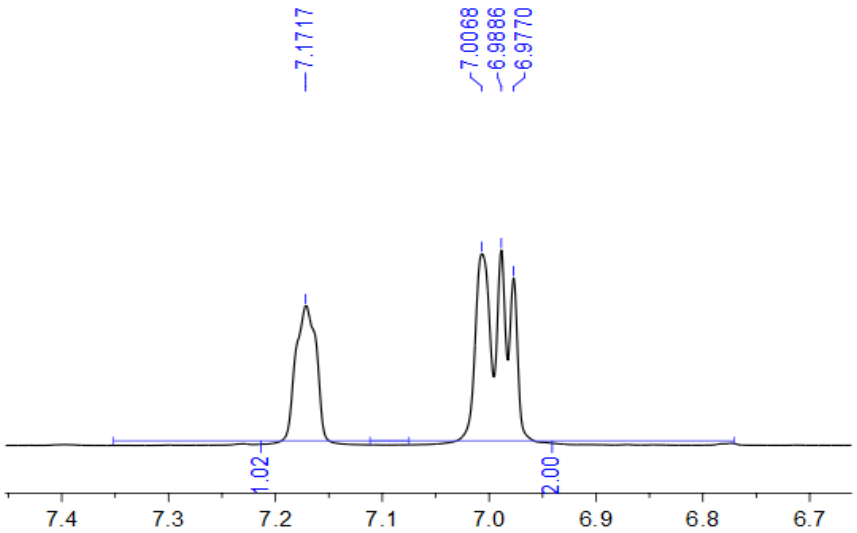
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **1t**

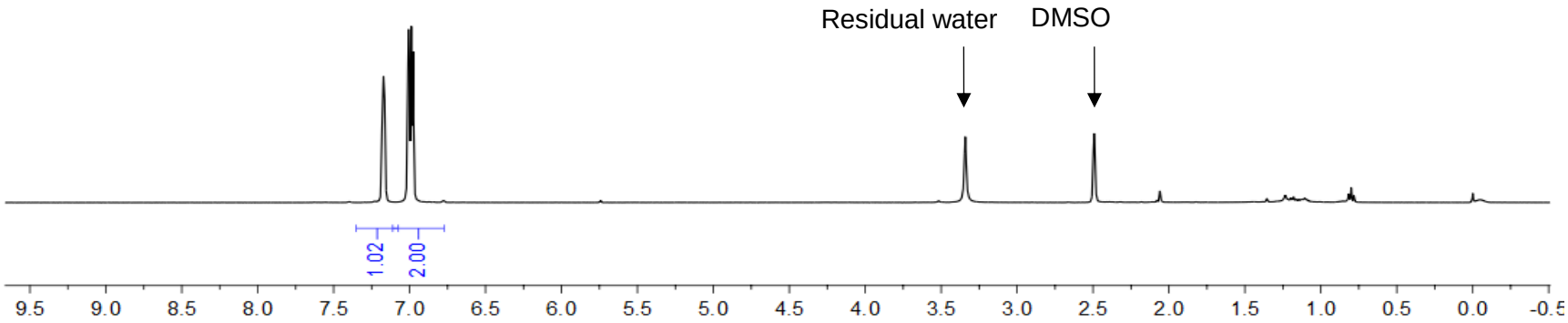




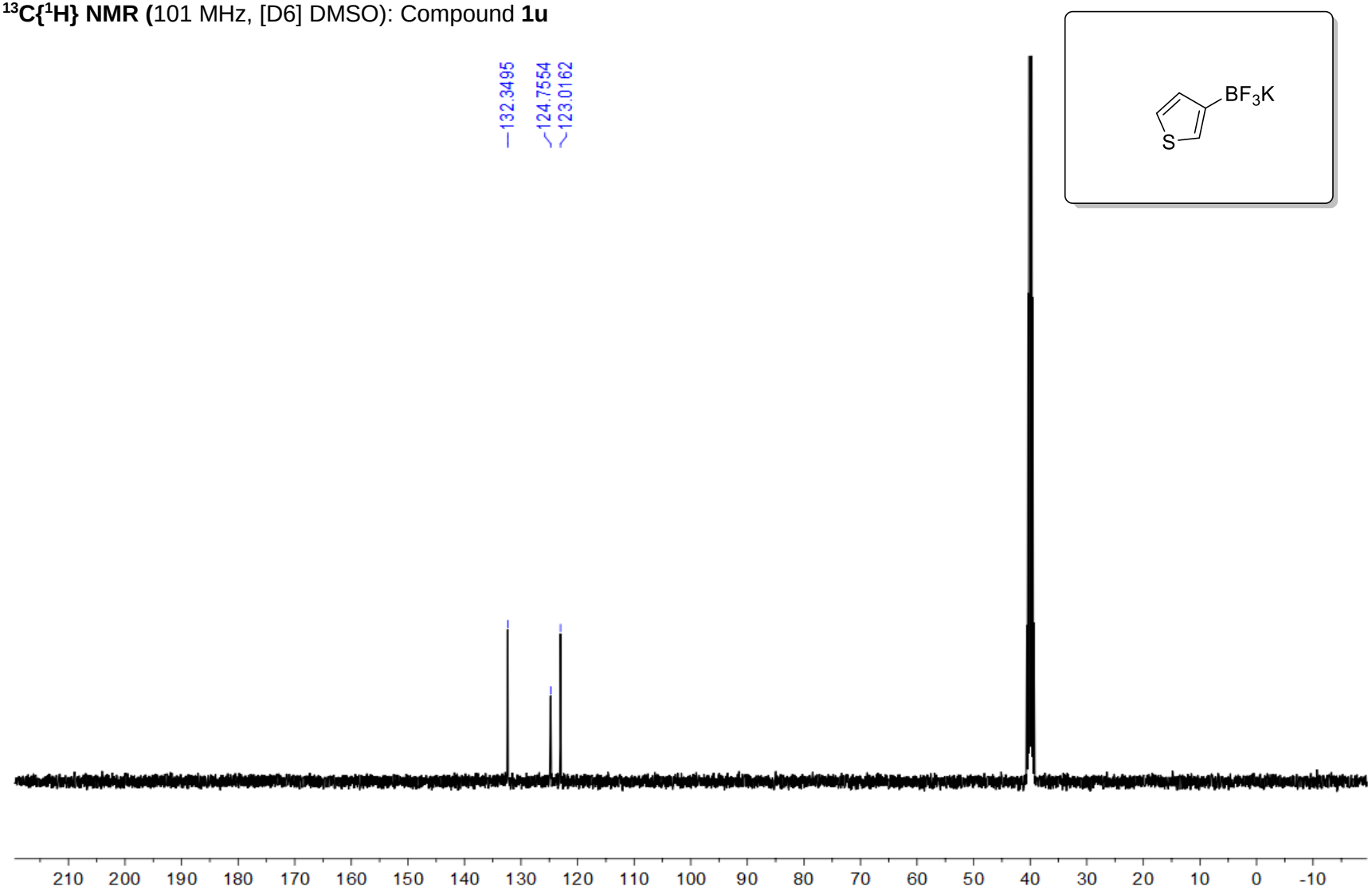
^{1}H NMR (400 MHz, [D6] DMSO): Compound **1u**



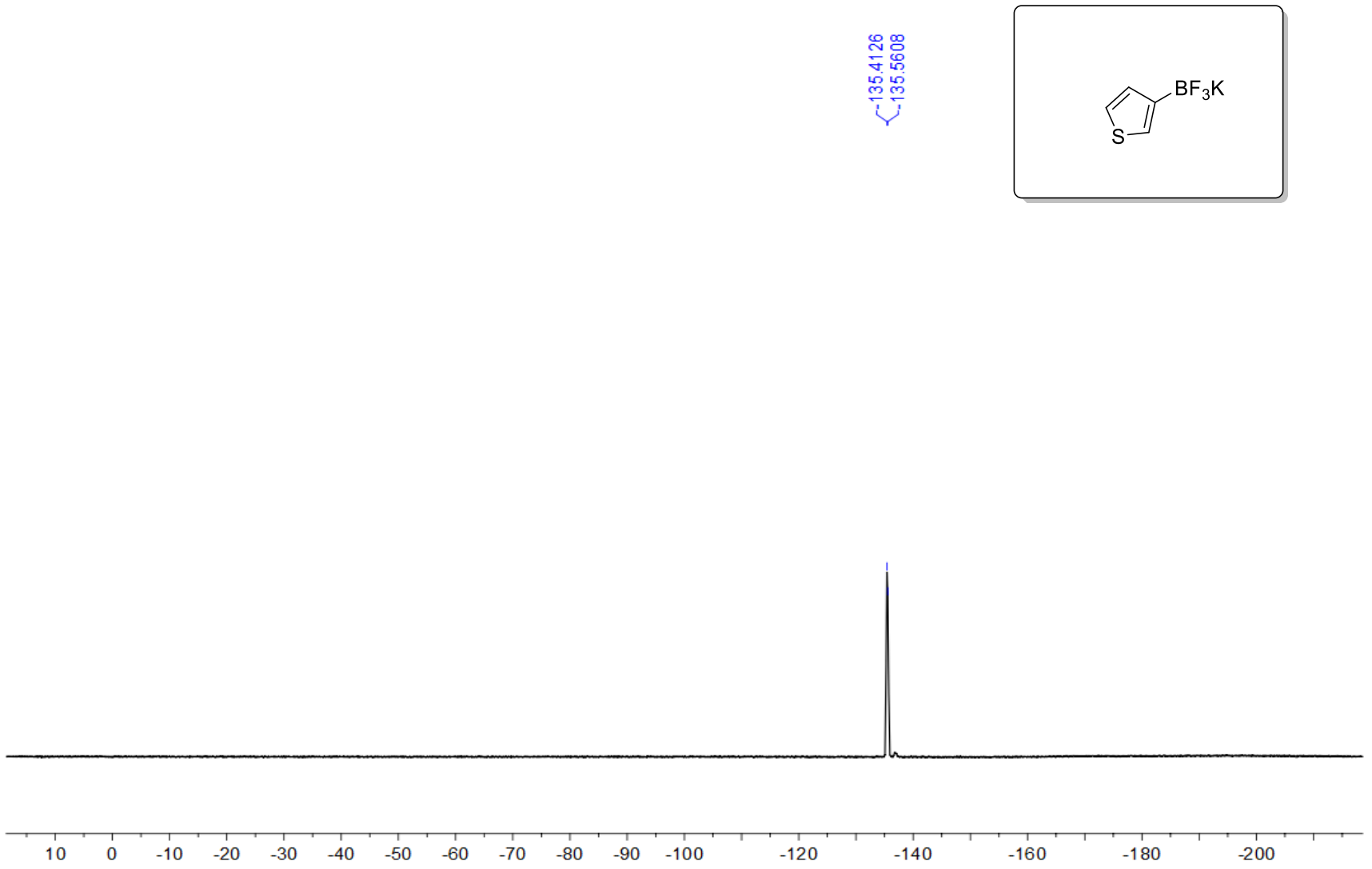




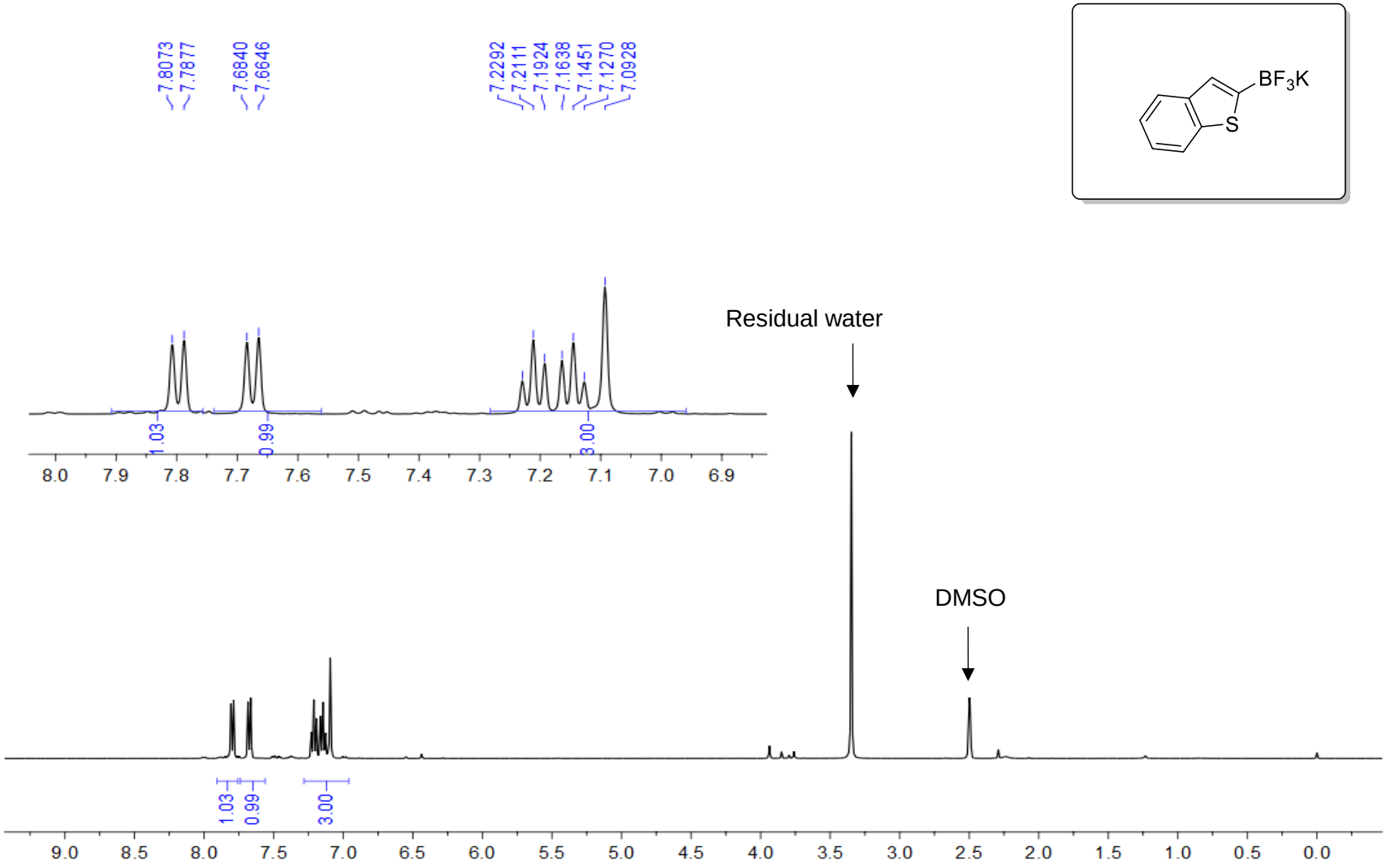
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **1u**



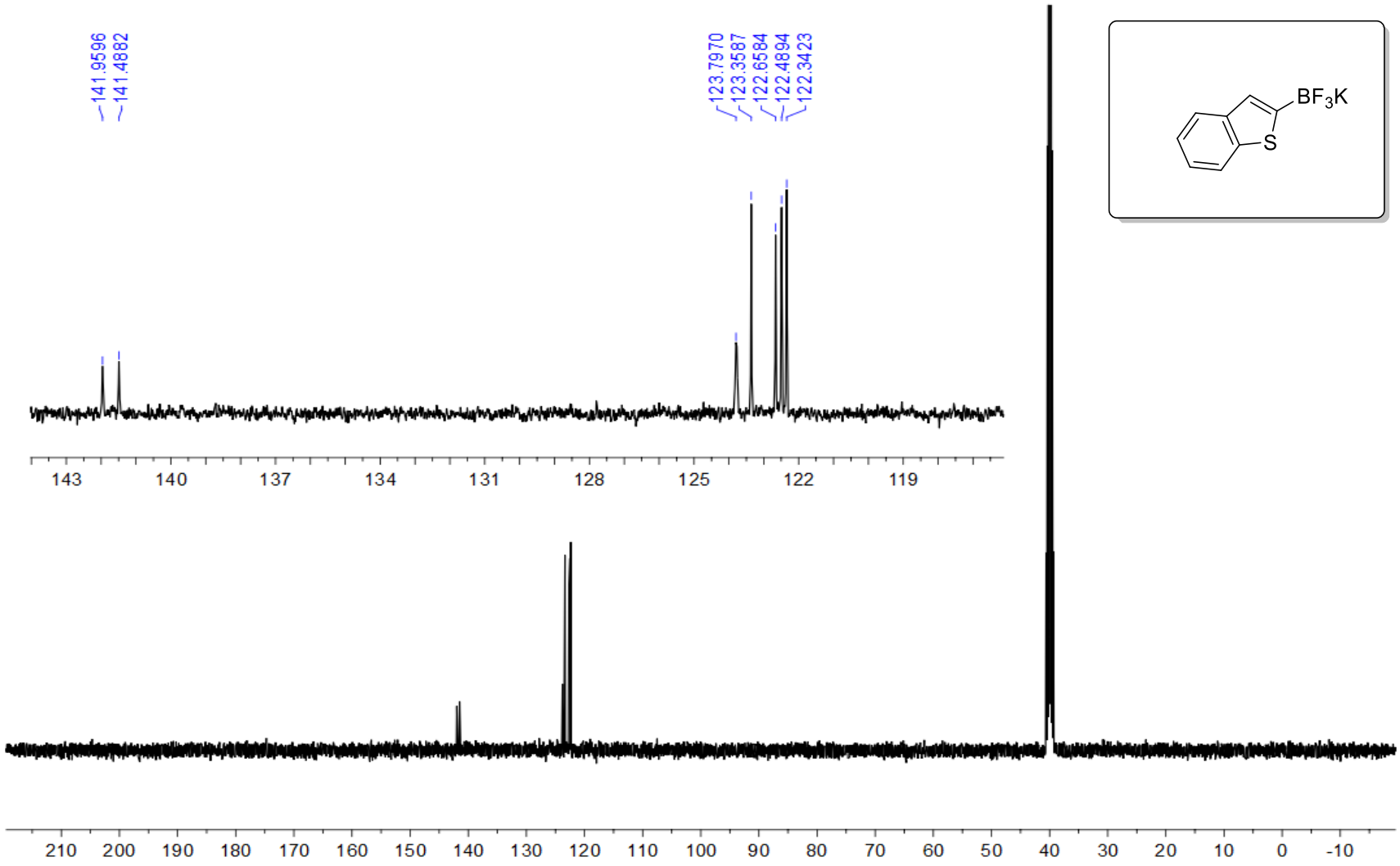
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **1u**



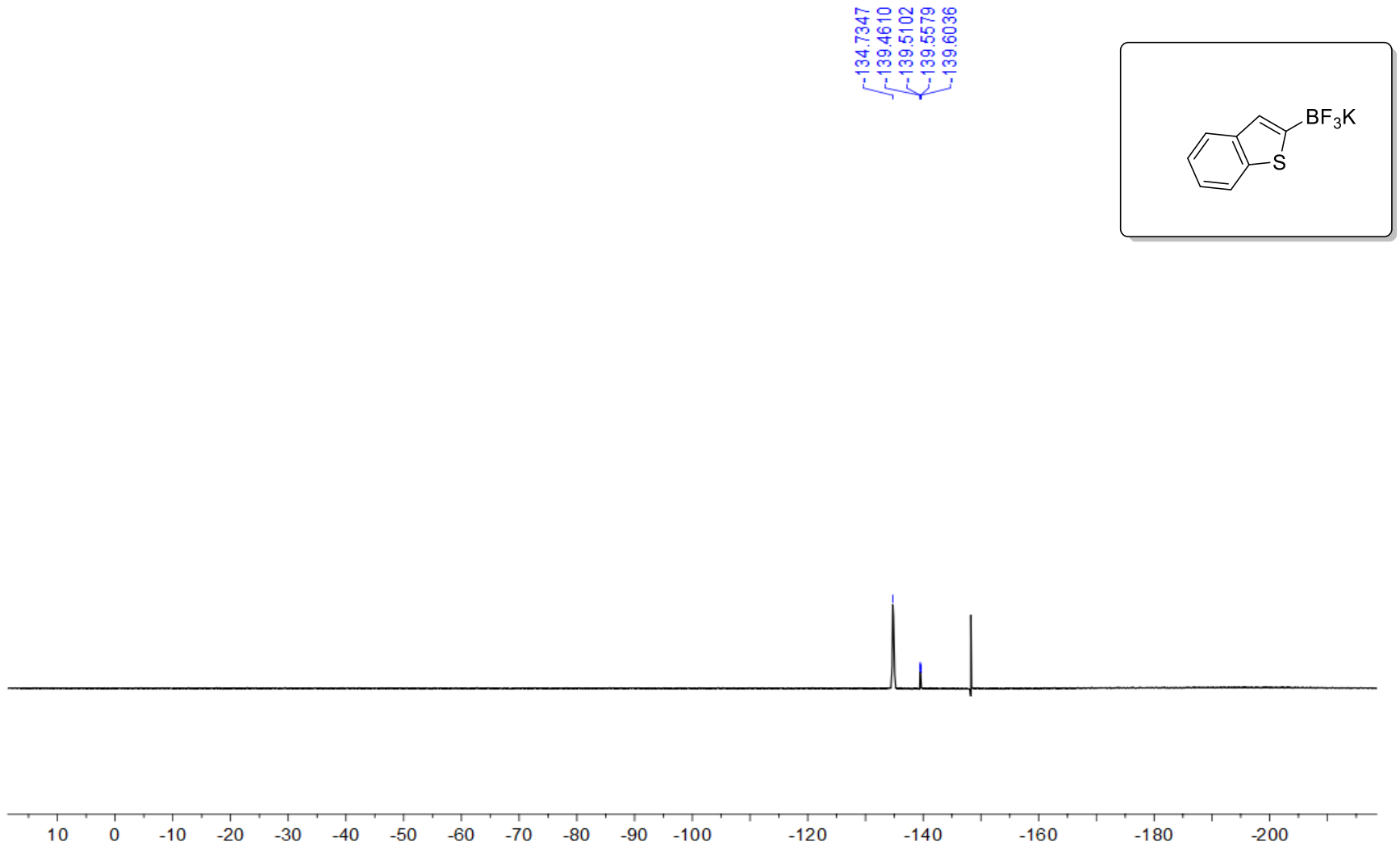
^{1}H NMR (400 MHz, [D6] DMSO): Compound **1v**



$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **1v**

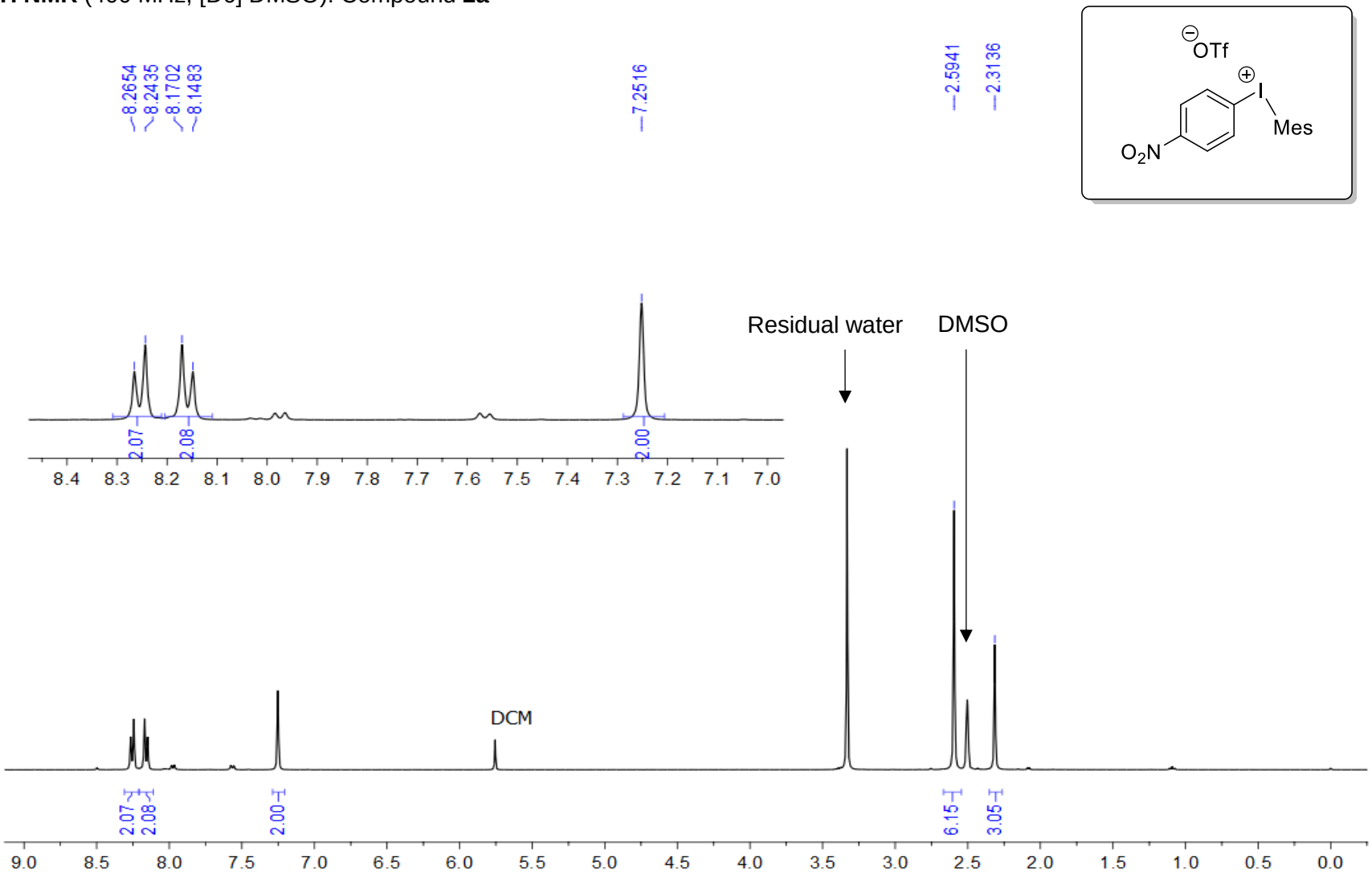


$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **1v**

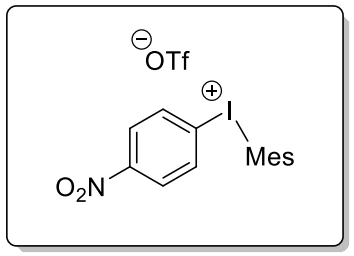


Spectral data for Aryl (Mes)iodonium salts

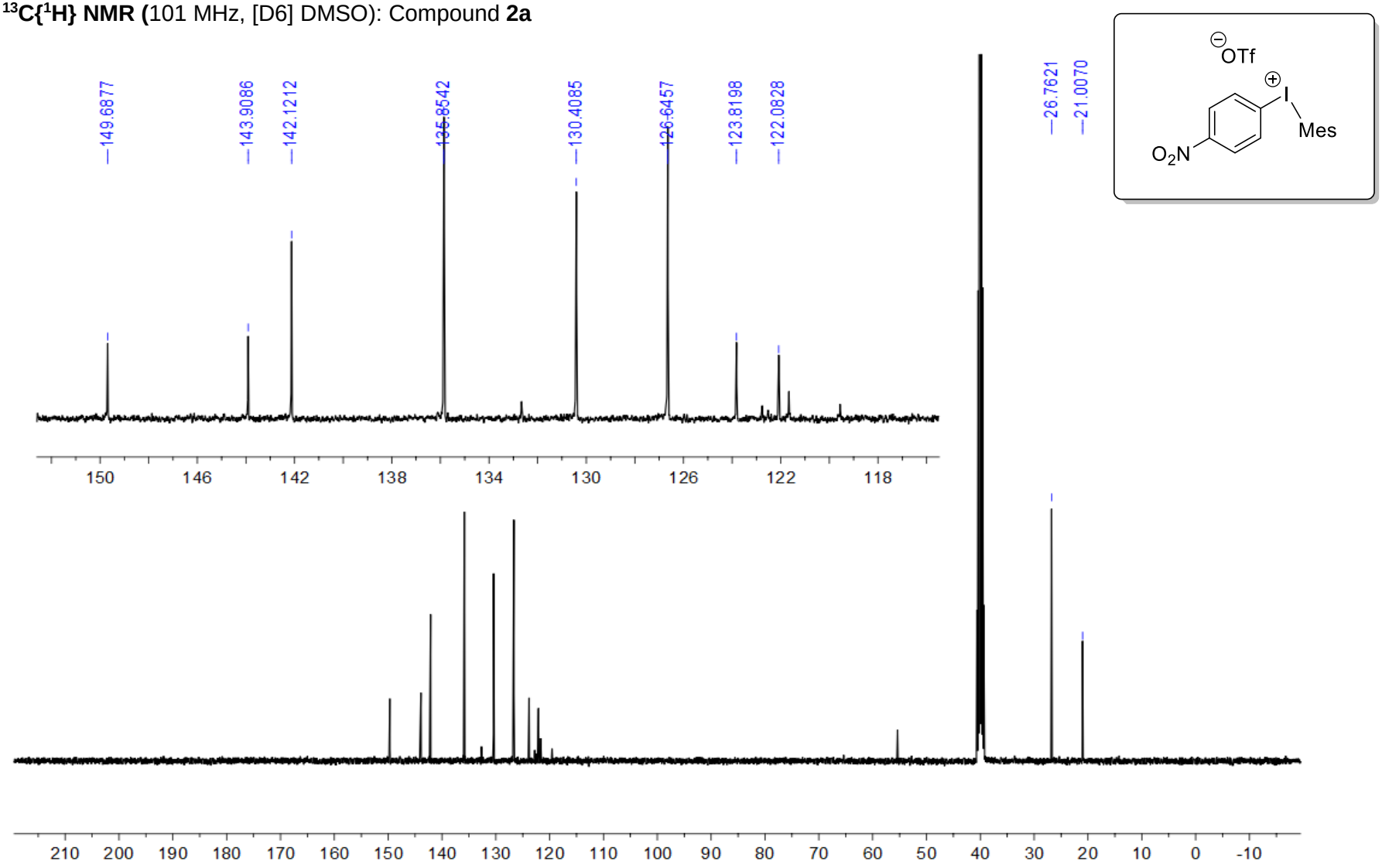
^{1}H NMR (400 MHz, [D6] DMSO): Compound **2a**



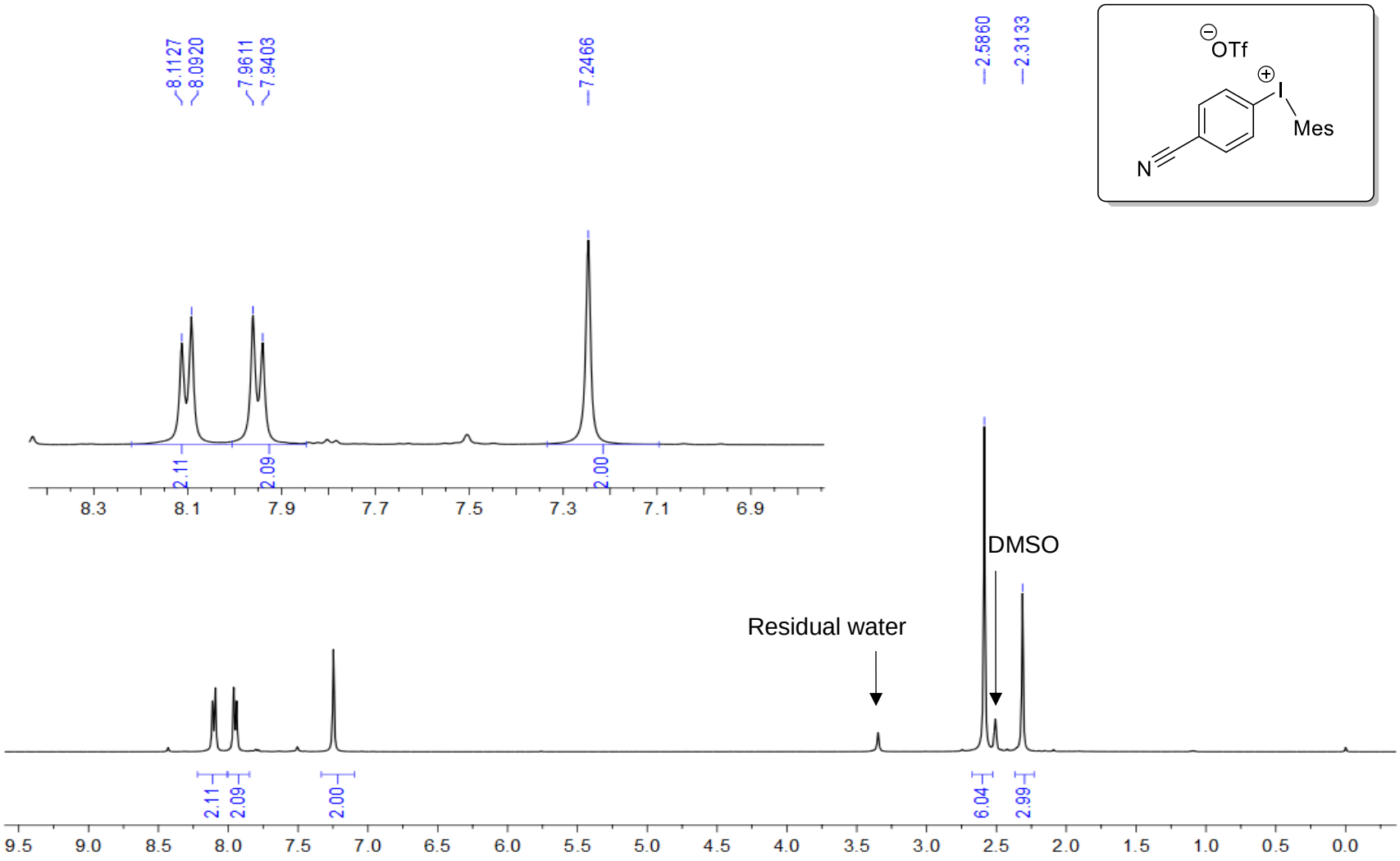
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **2a**



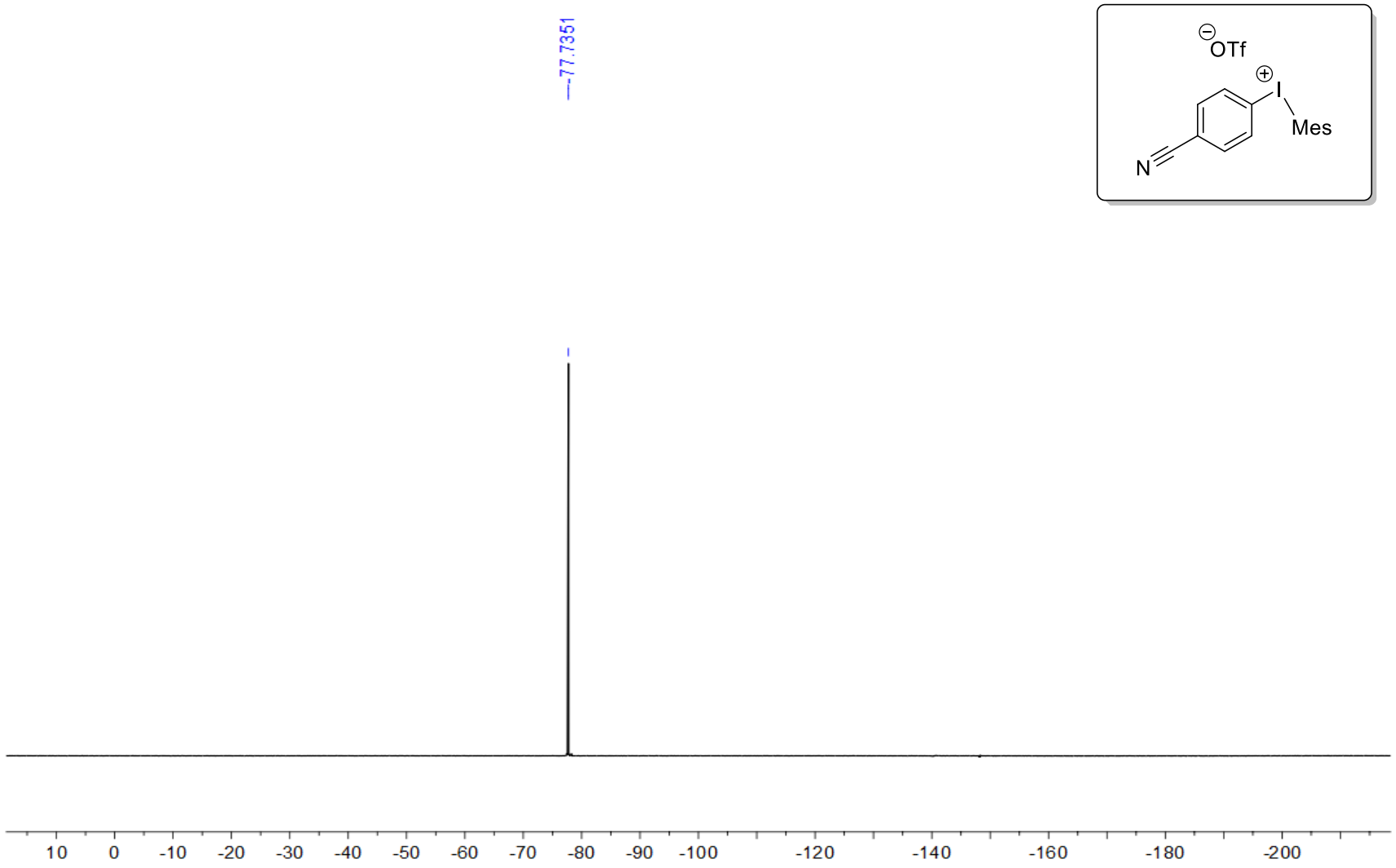
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **2a**



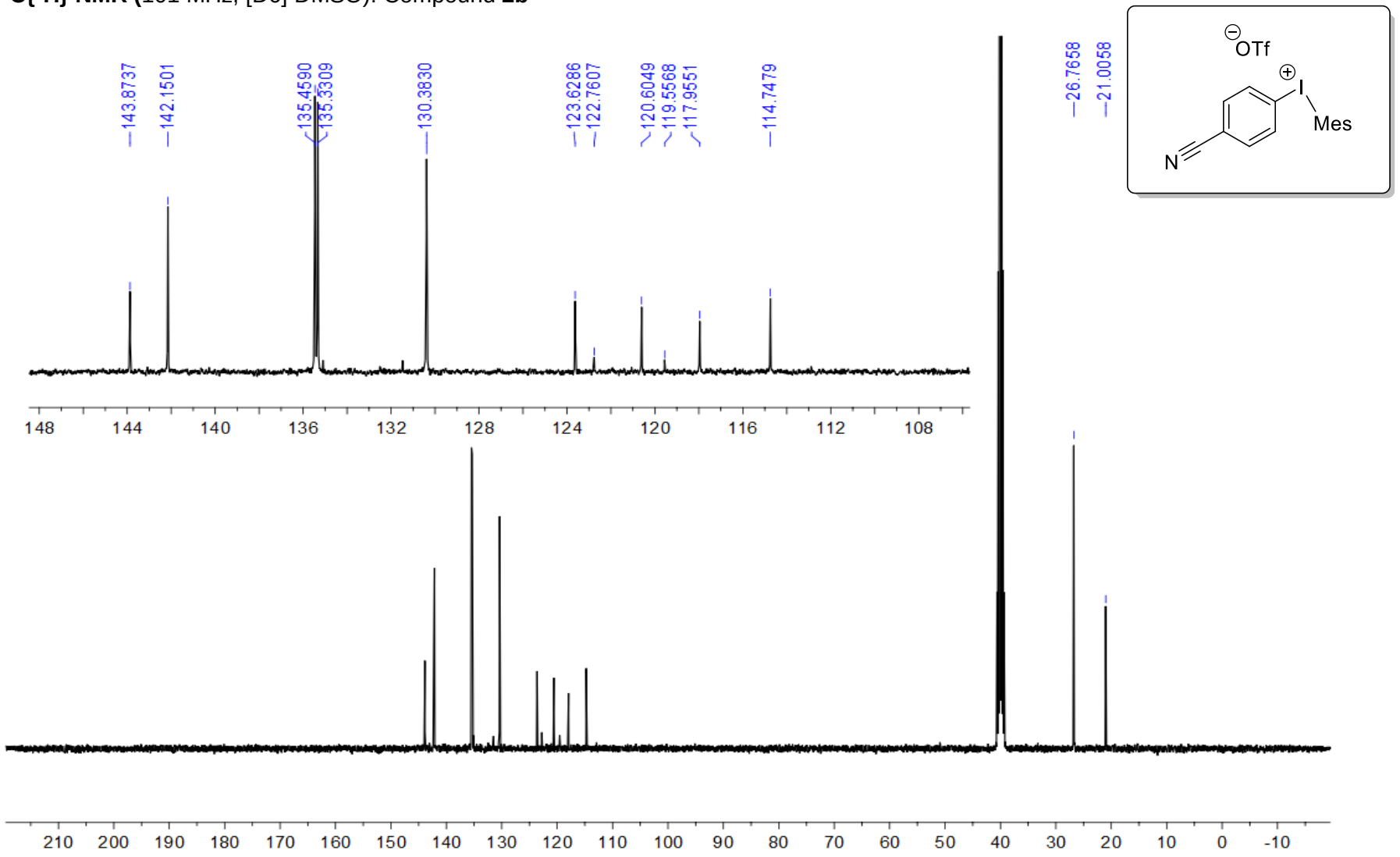
^{1}H NMR (400 MHz, [D6] DMSO): Compound **2b**



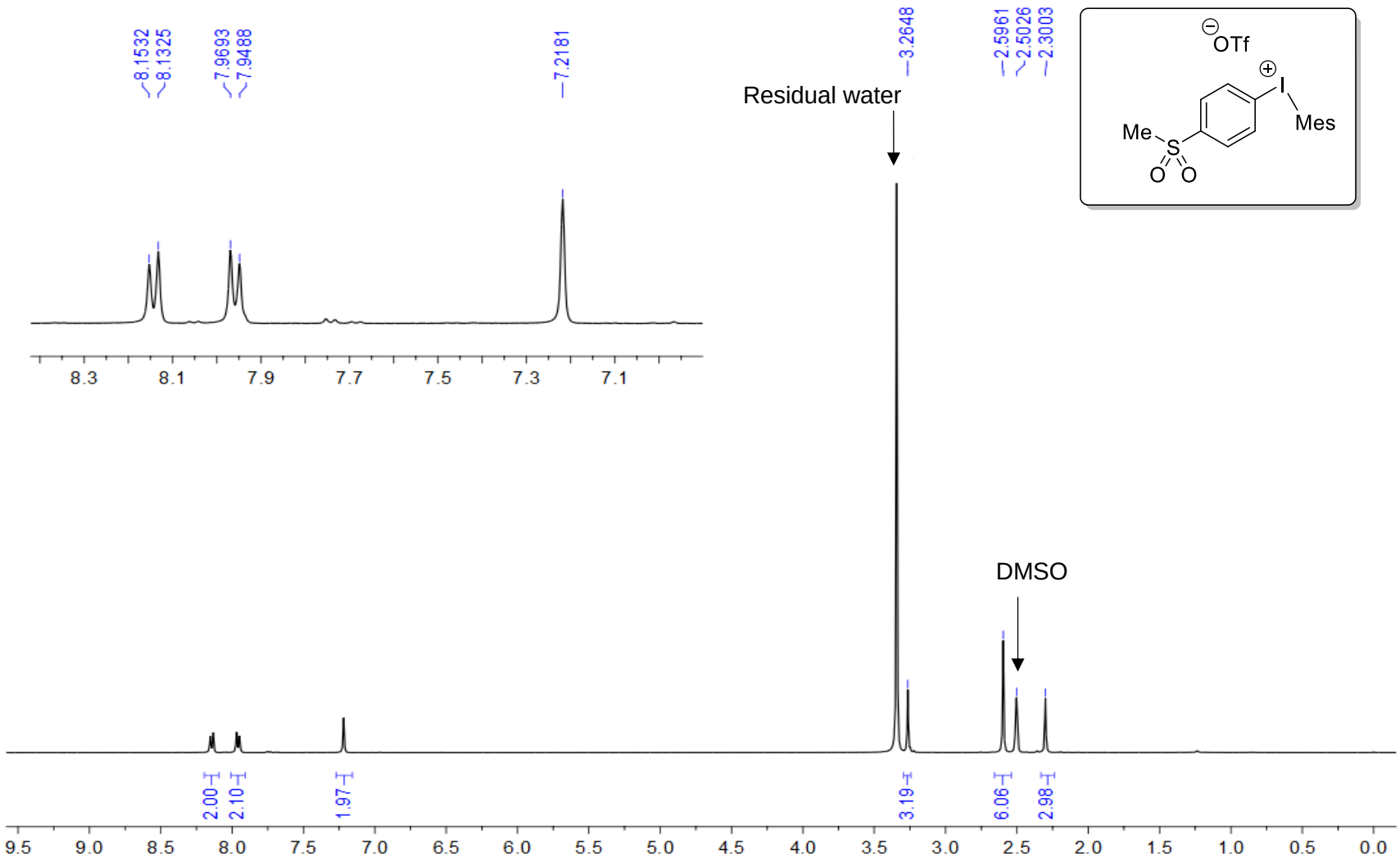
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **1b**



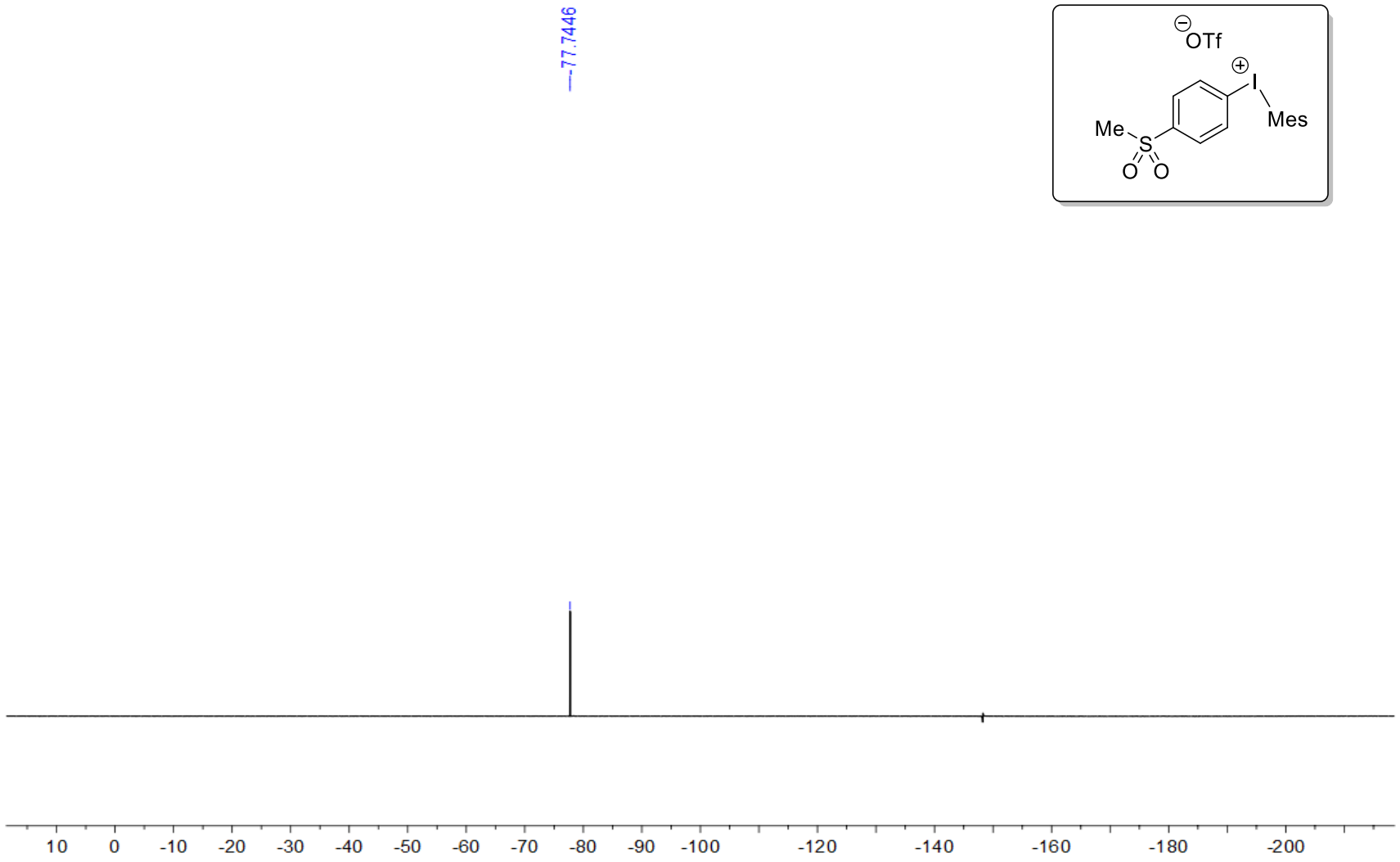
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **2b**



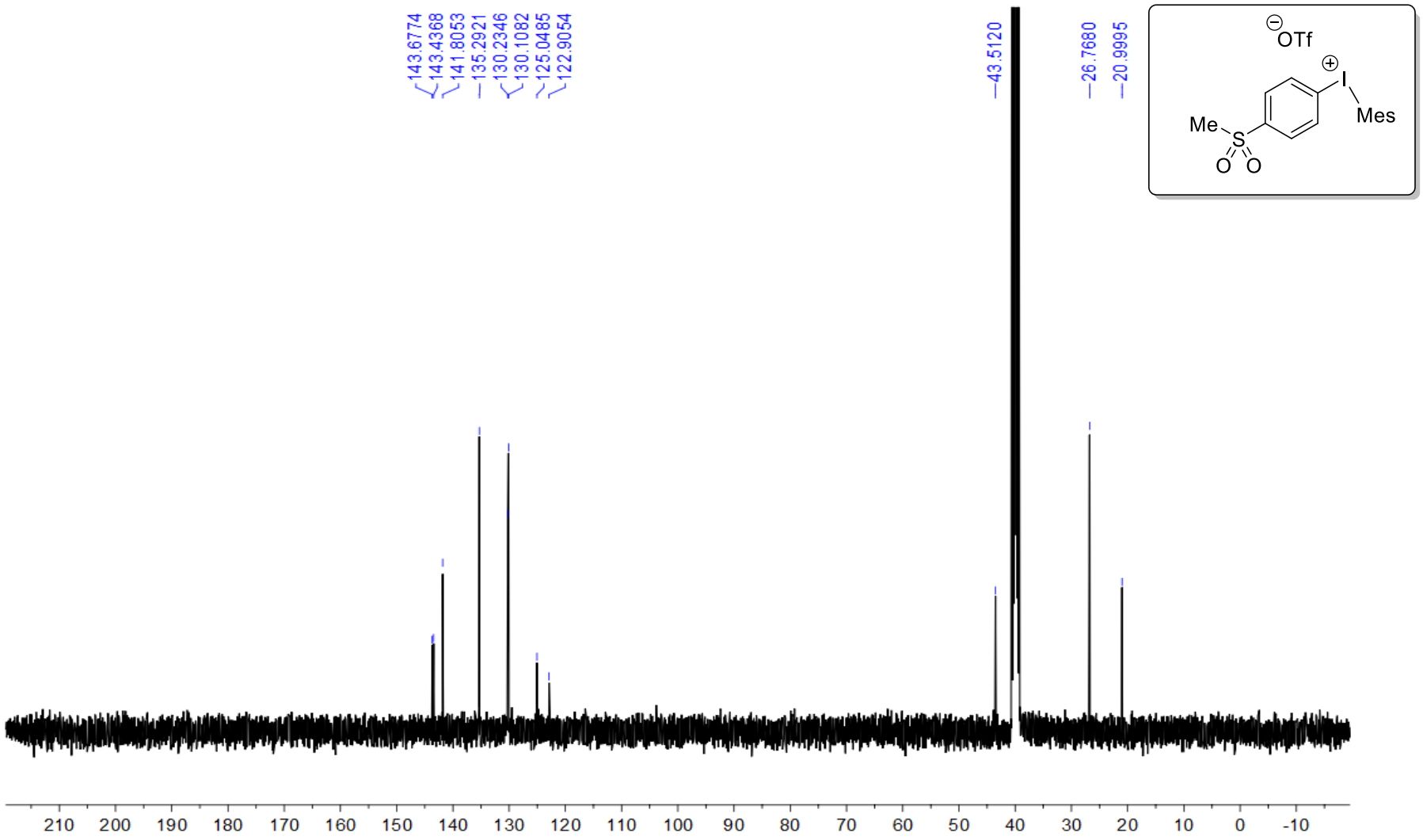
^{1}H NMR (400 MHz, [D6] DMSO): Compound **2c**



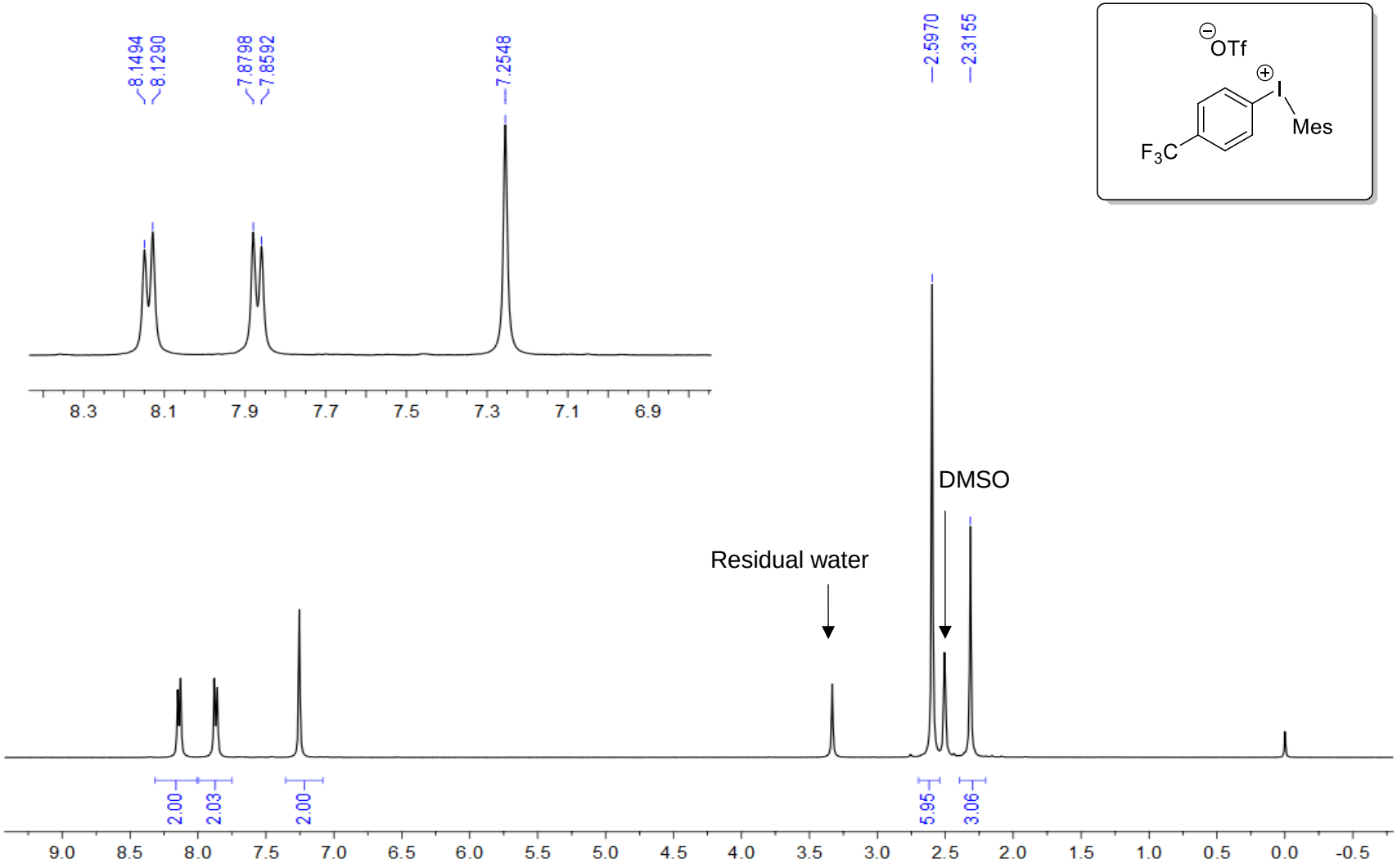
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **2c**



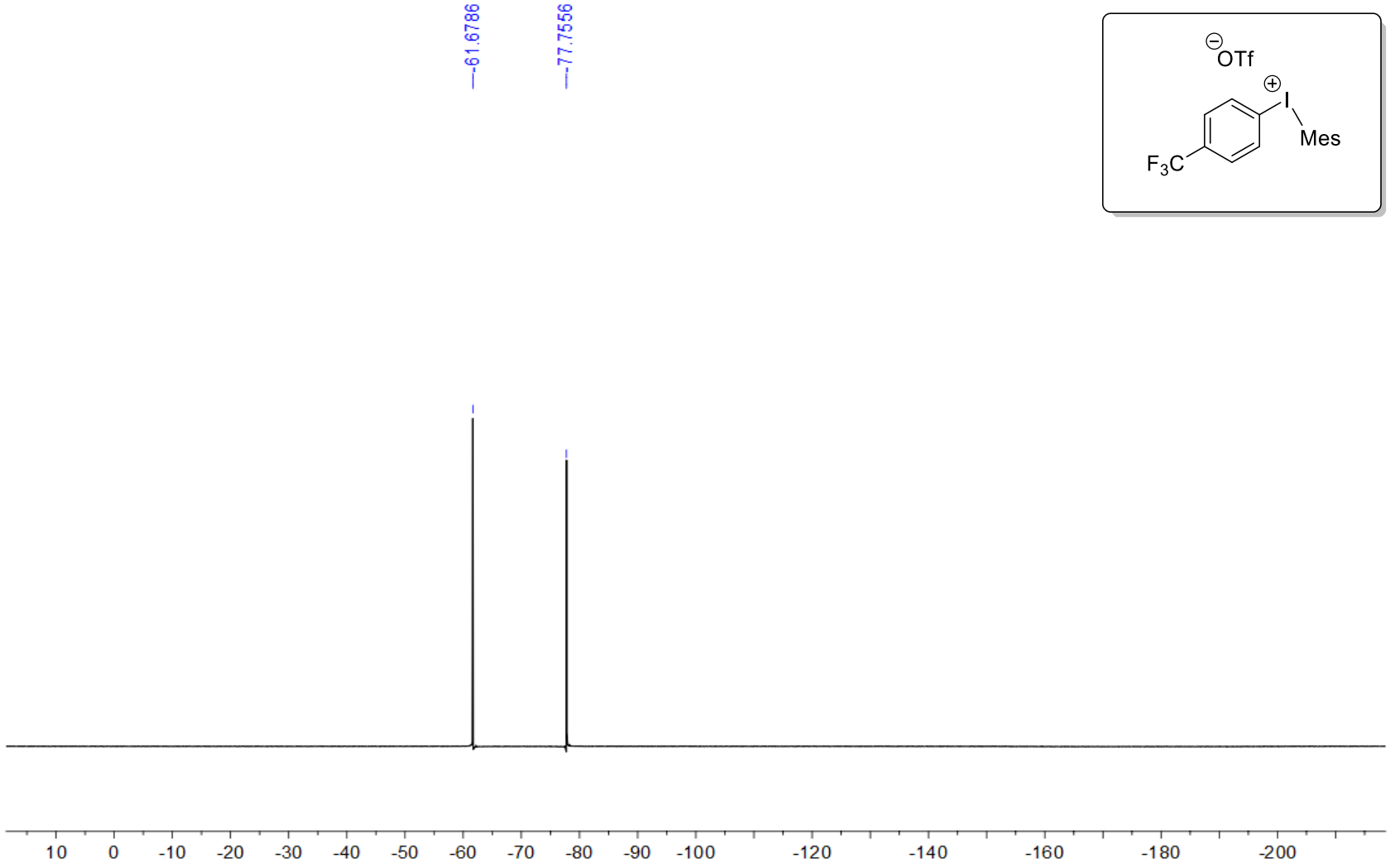
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **2c**



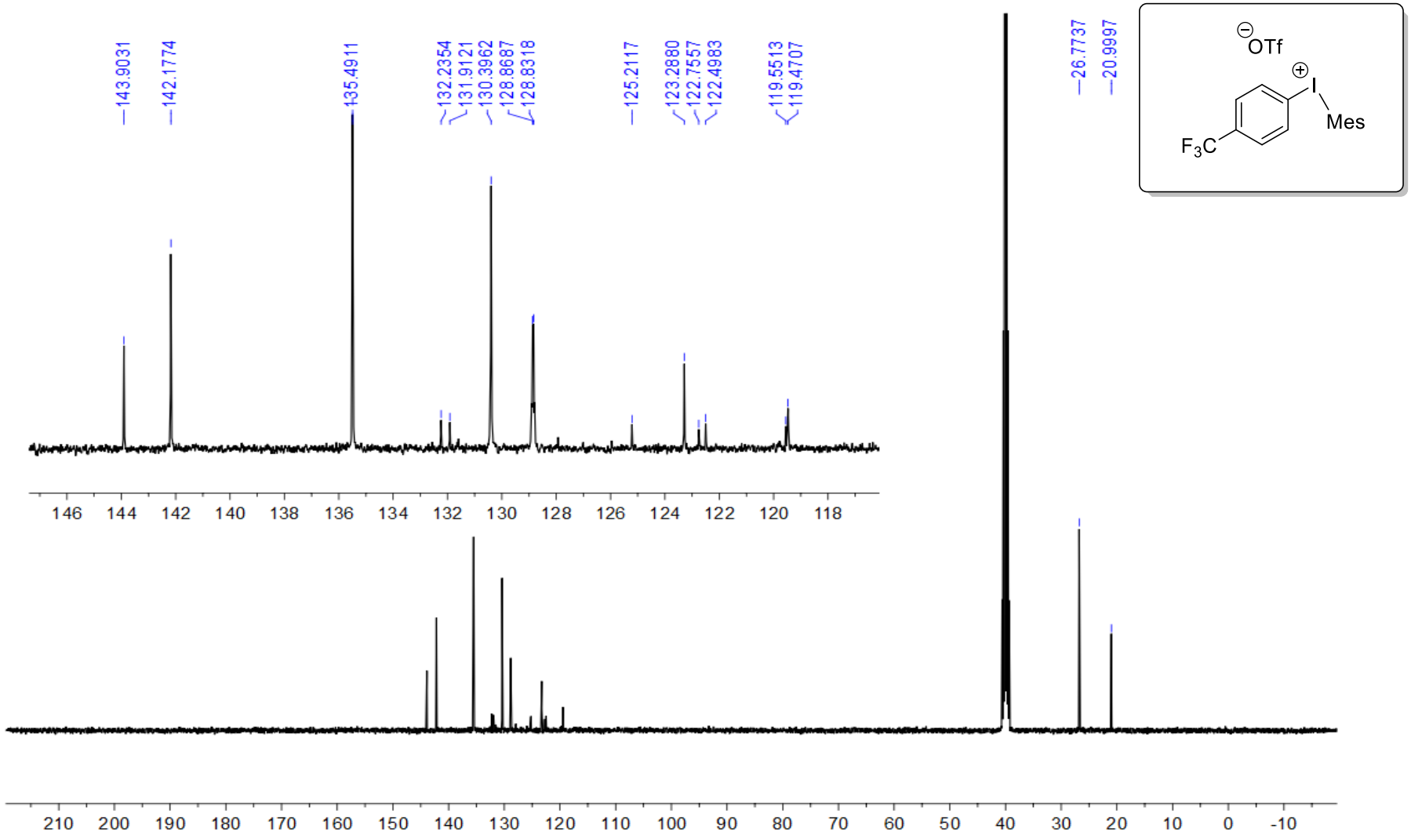
^{1}H NMR (400 MHz, [D6] DMSO): Compound **2d**



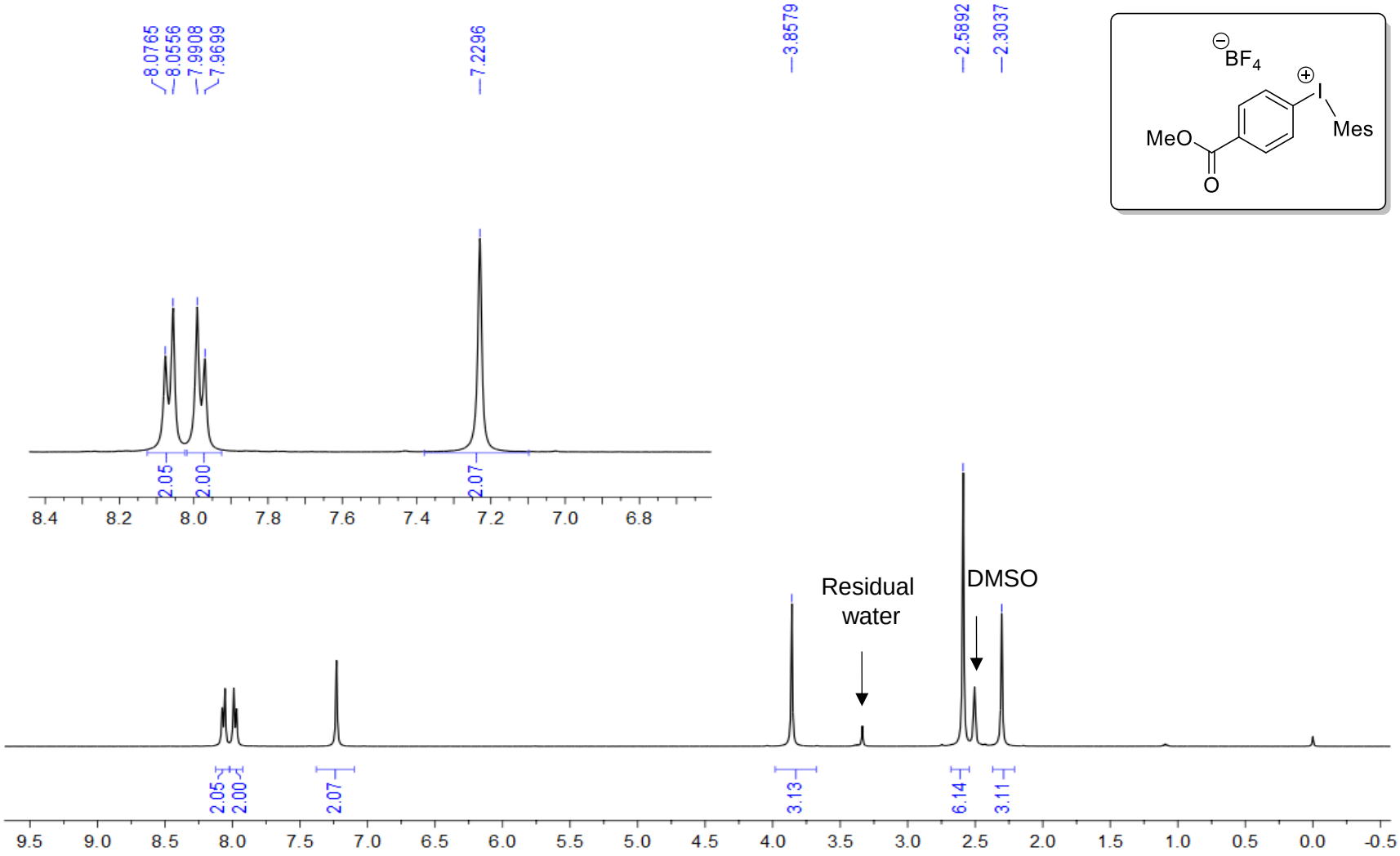
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **2d**



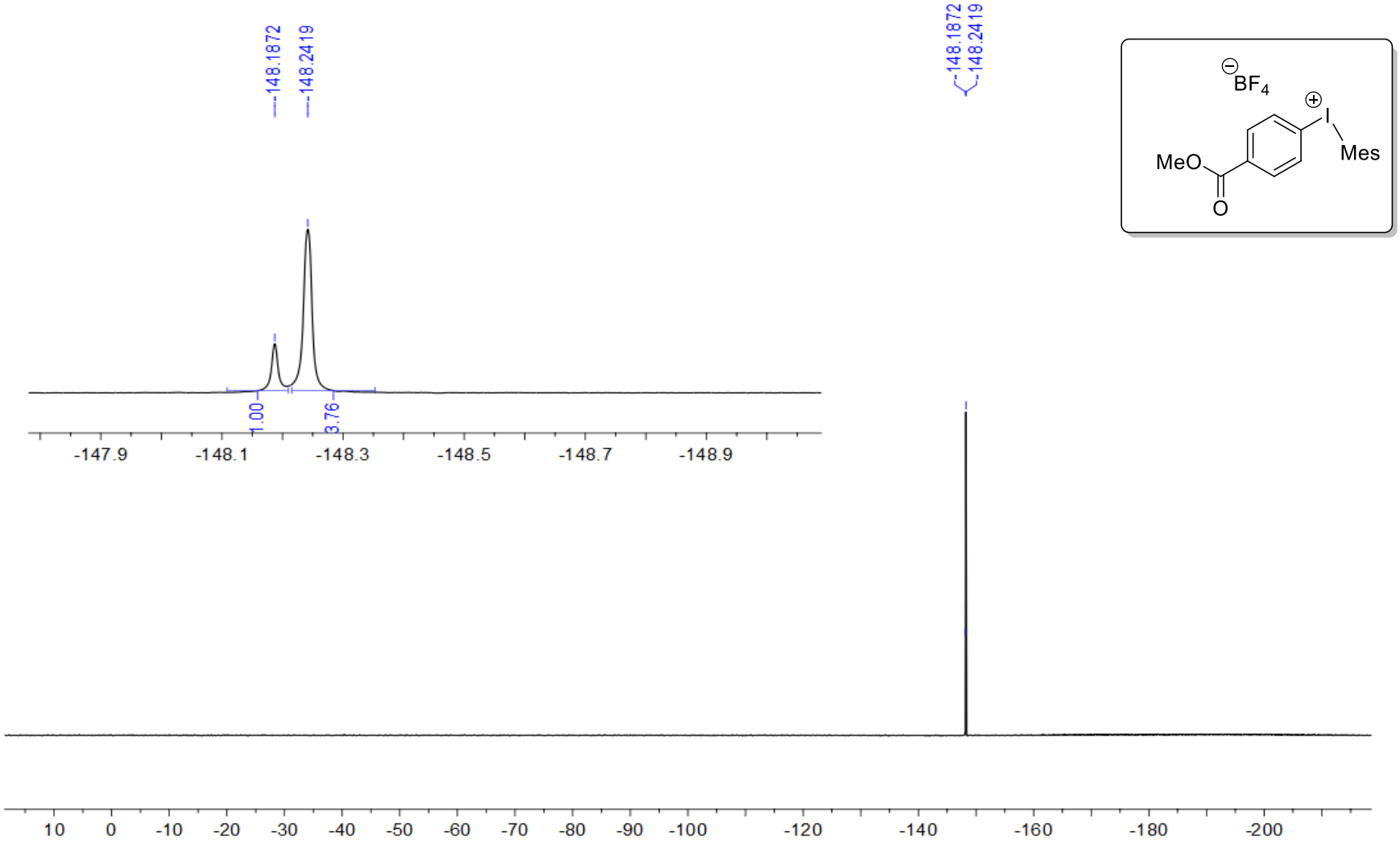
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **2d**



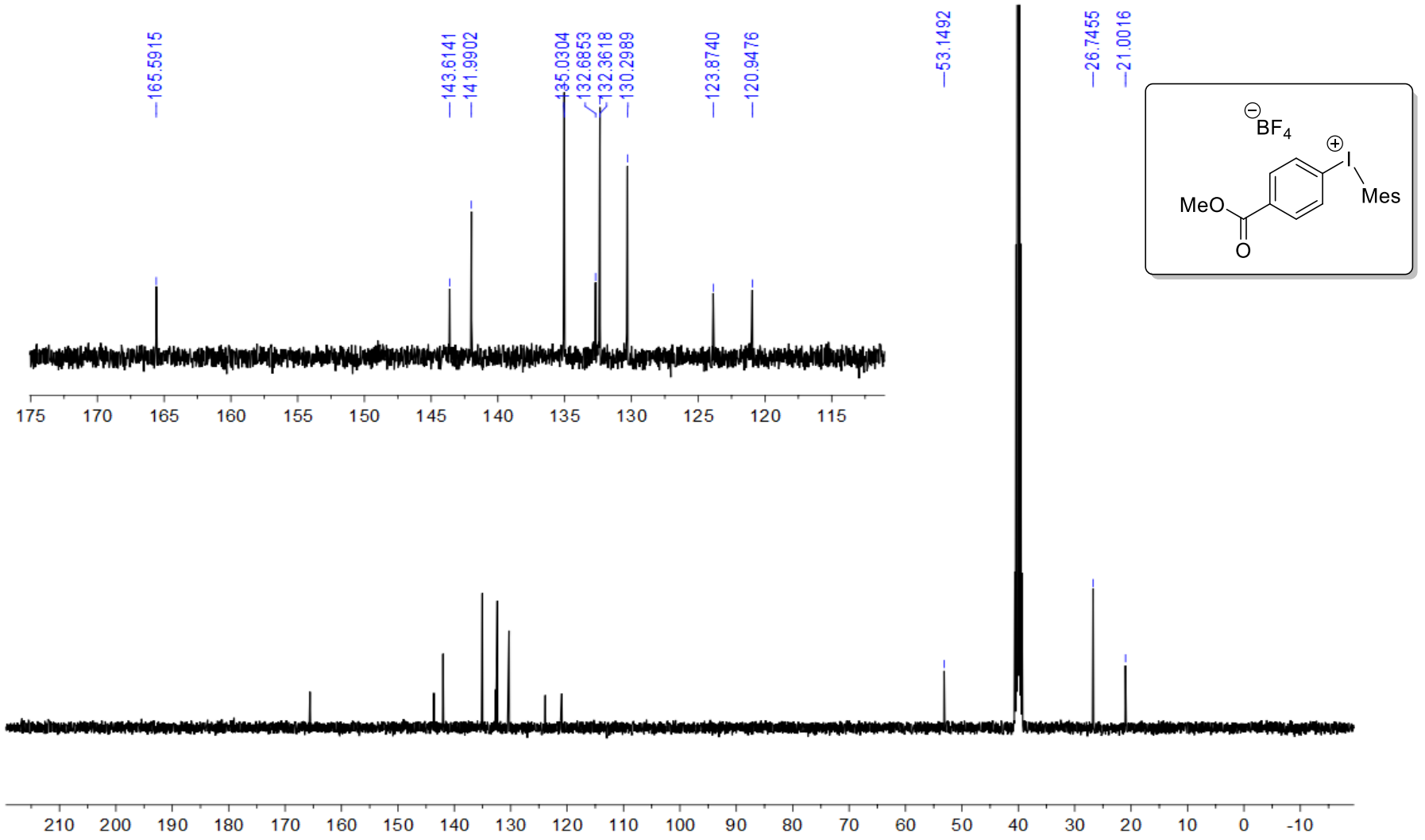
^{1}H NMR (400 MHz, [D6] DMSO): Compound **2e**



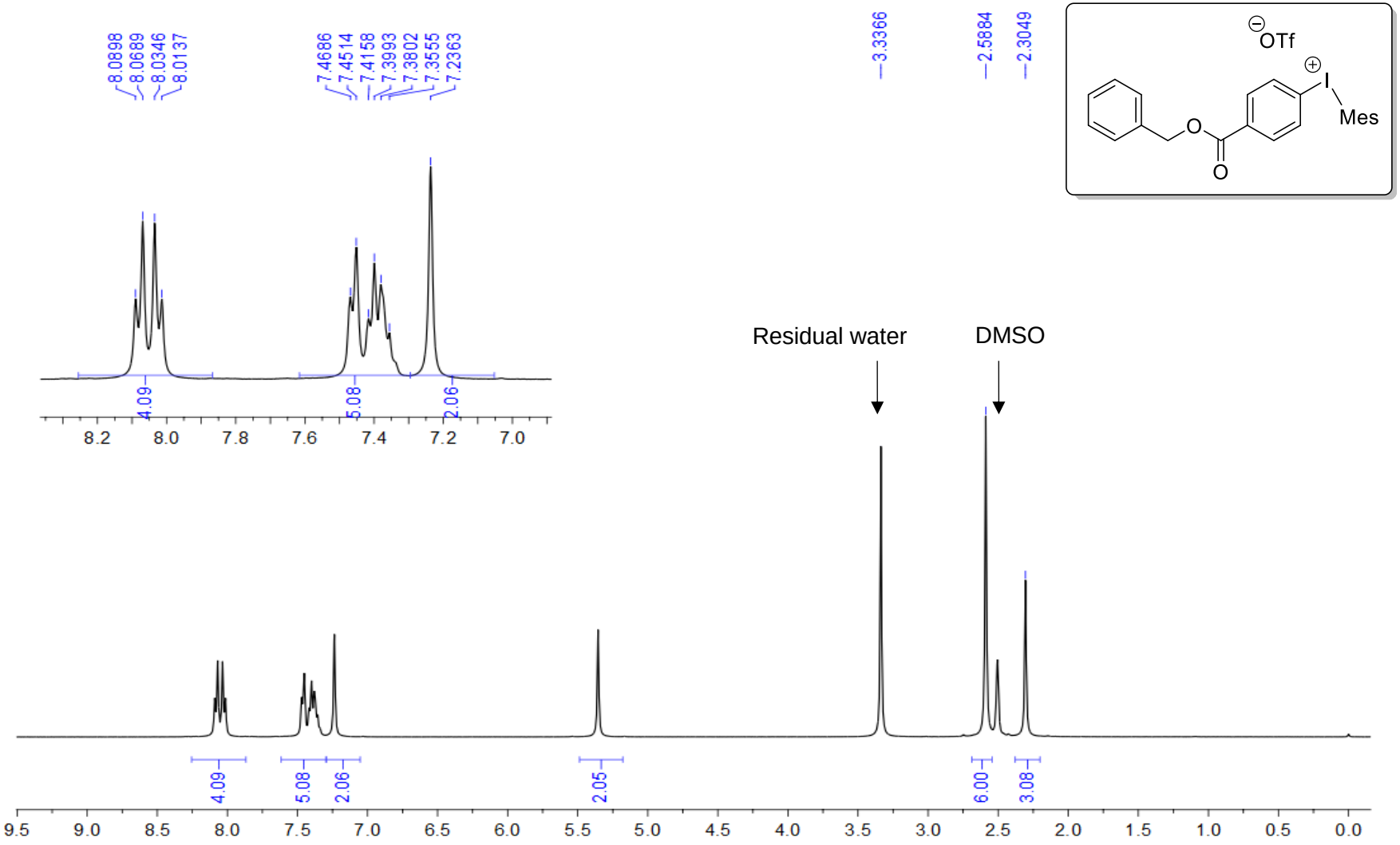
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **2e**



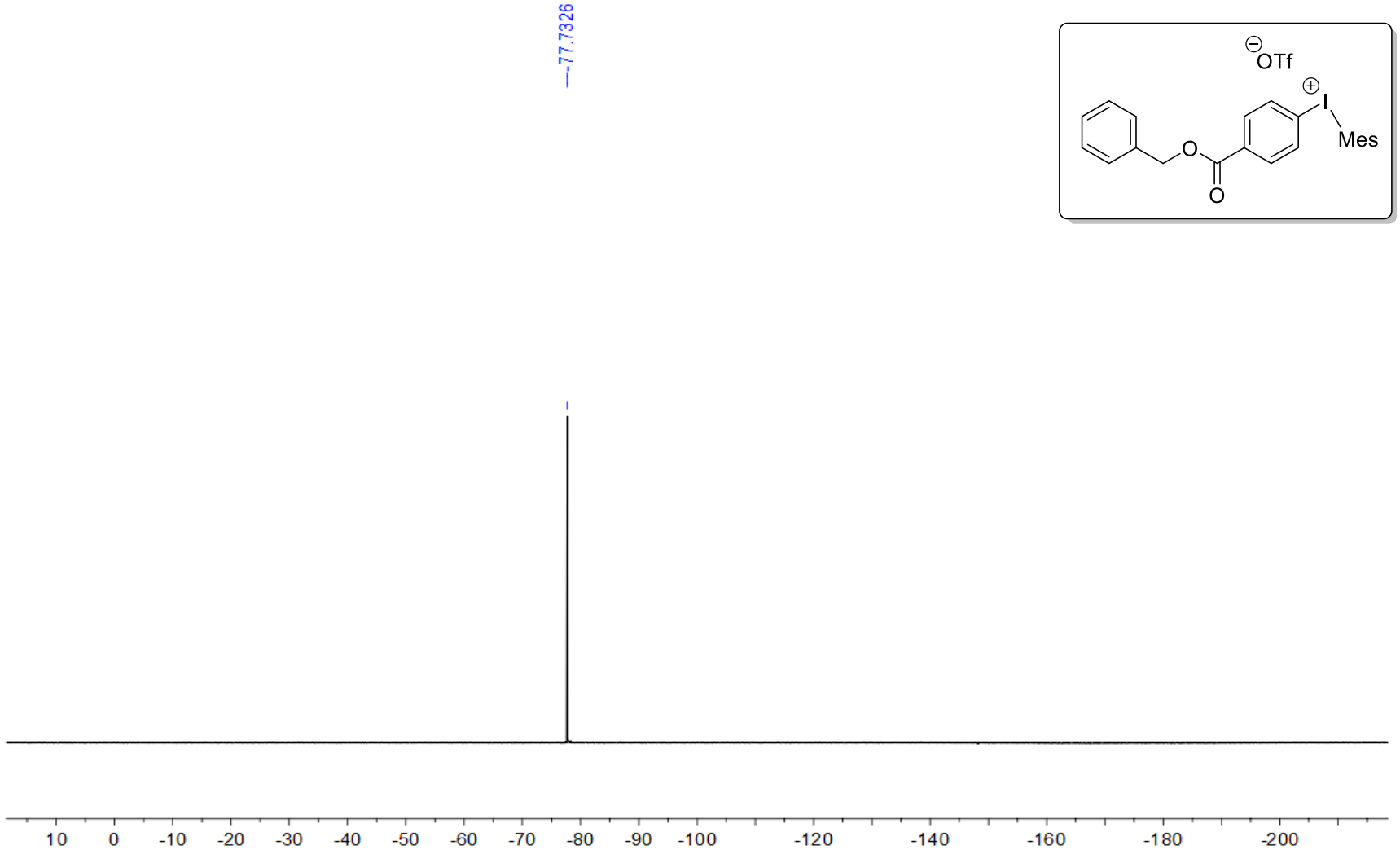
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **2e**



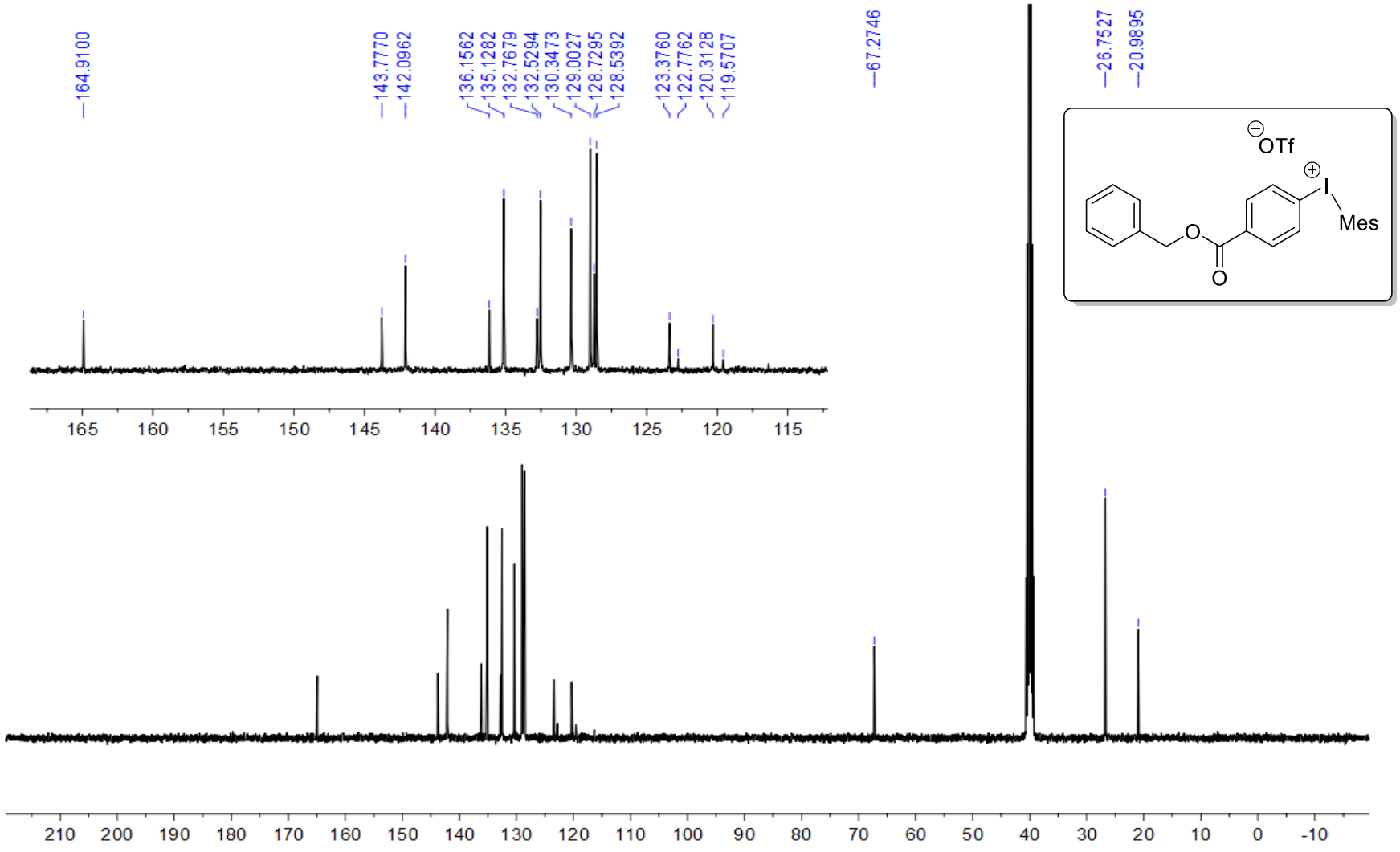
^{1}H NMR (400 MHz, [D6] DMSO): Compound **2f**



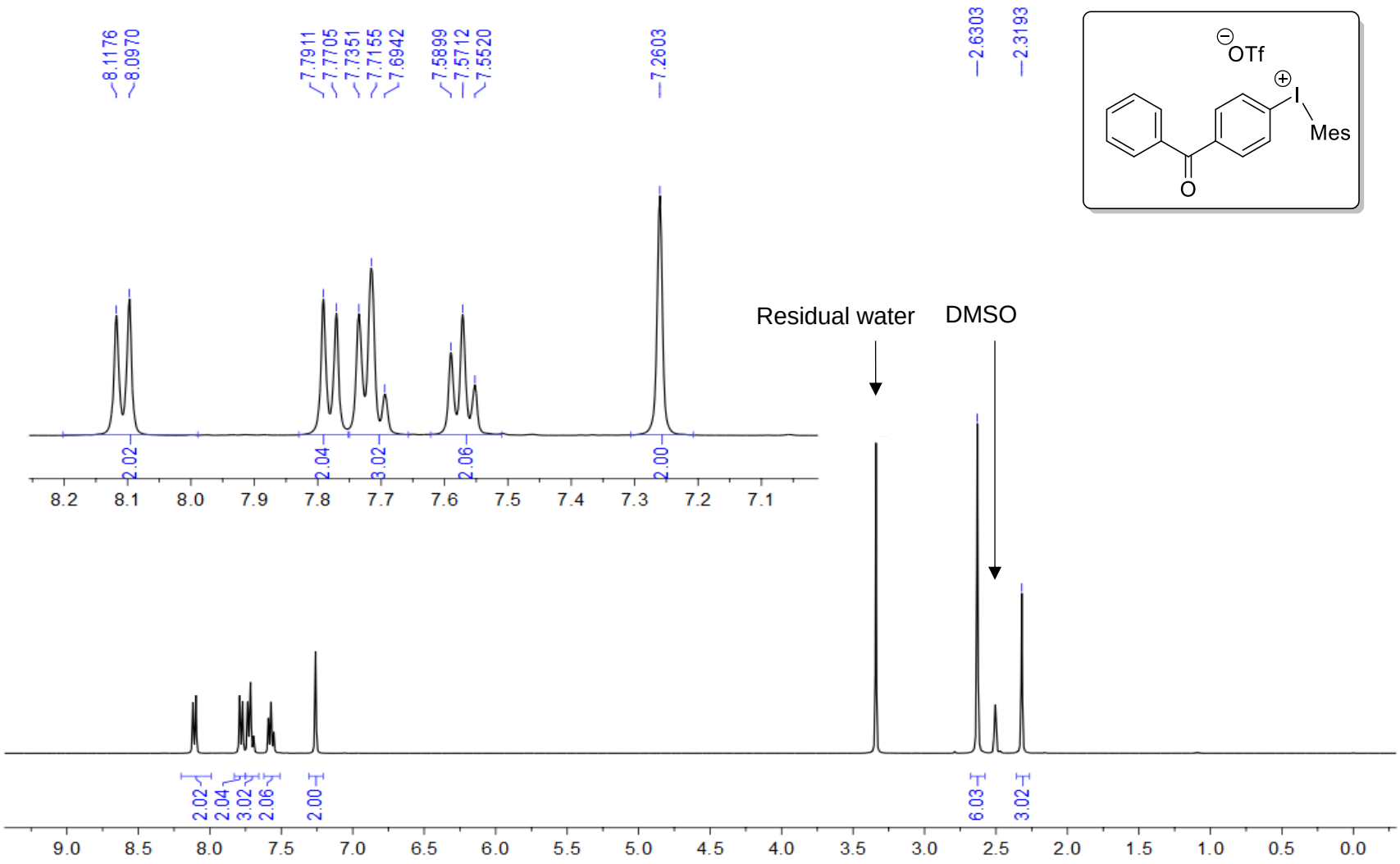
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **2f**



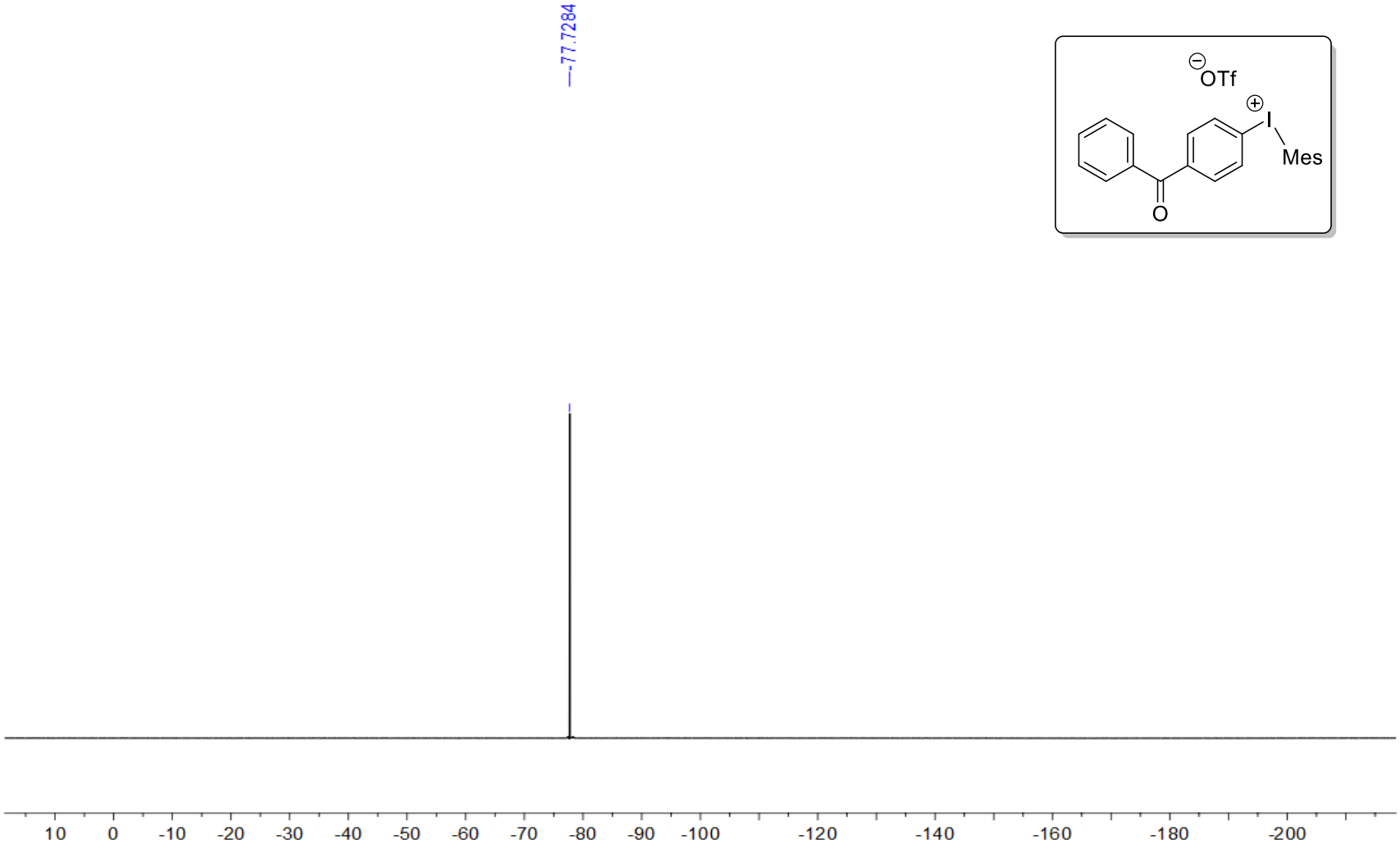
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **2f**



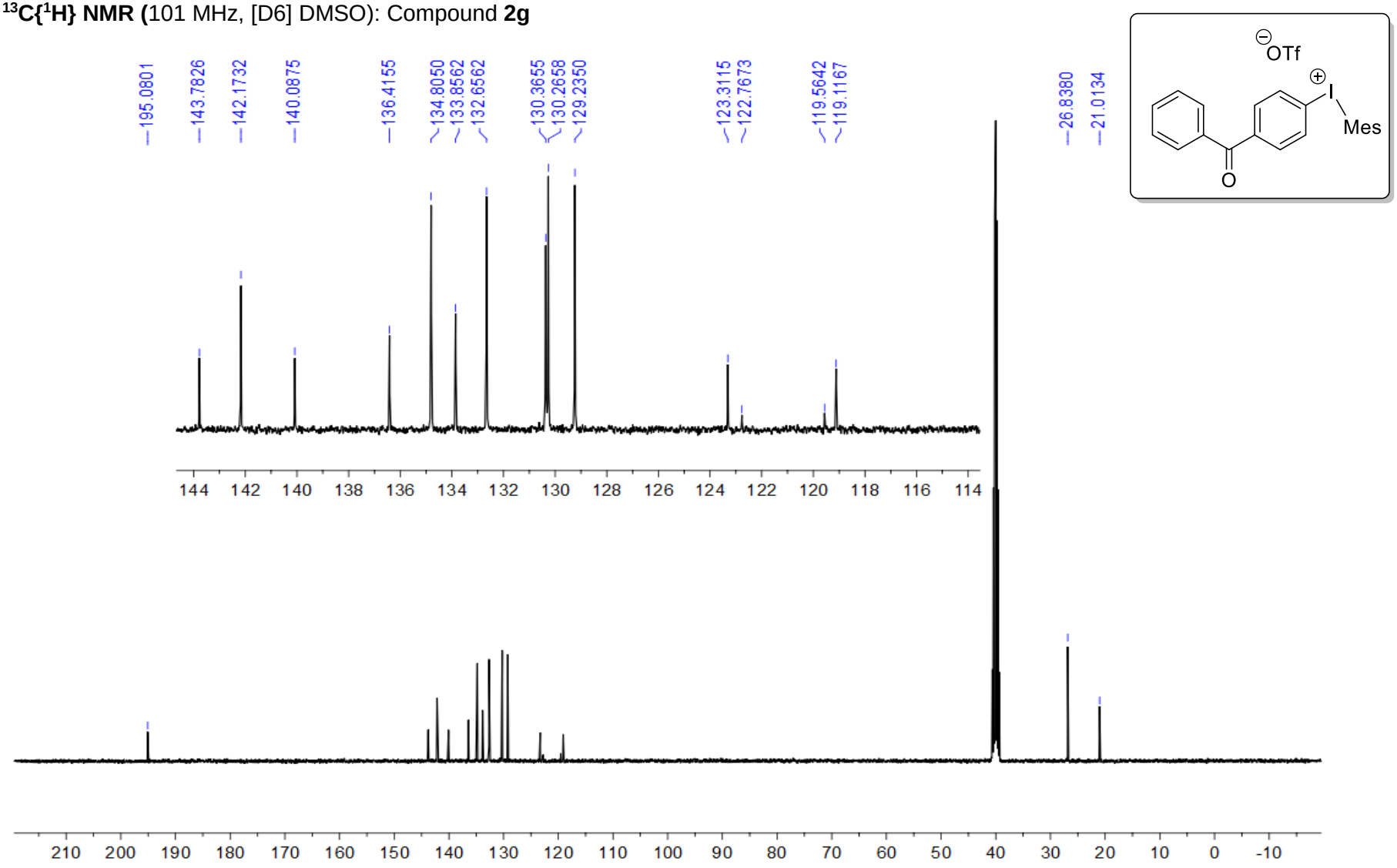
^{1}H NMR (400 MHz, [D6] DMSO): Compound **2g**



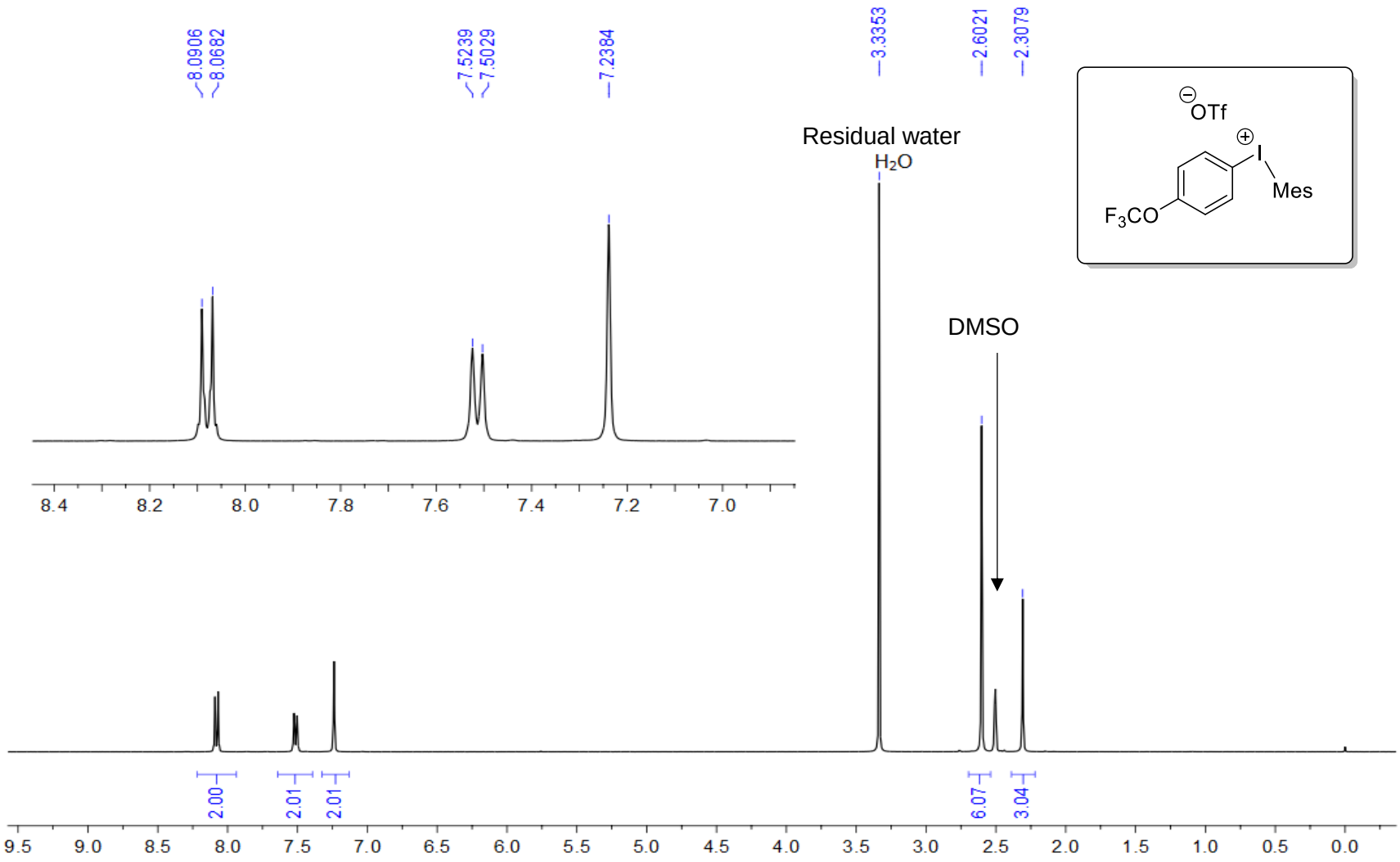
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **2g**



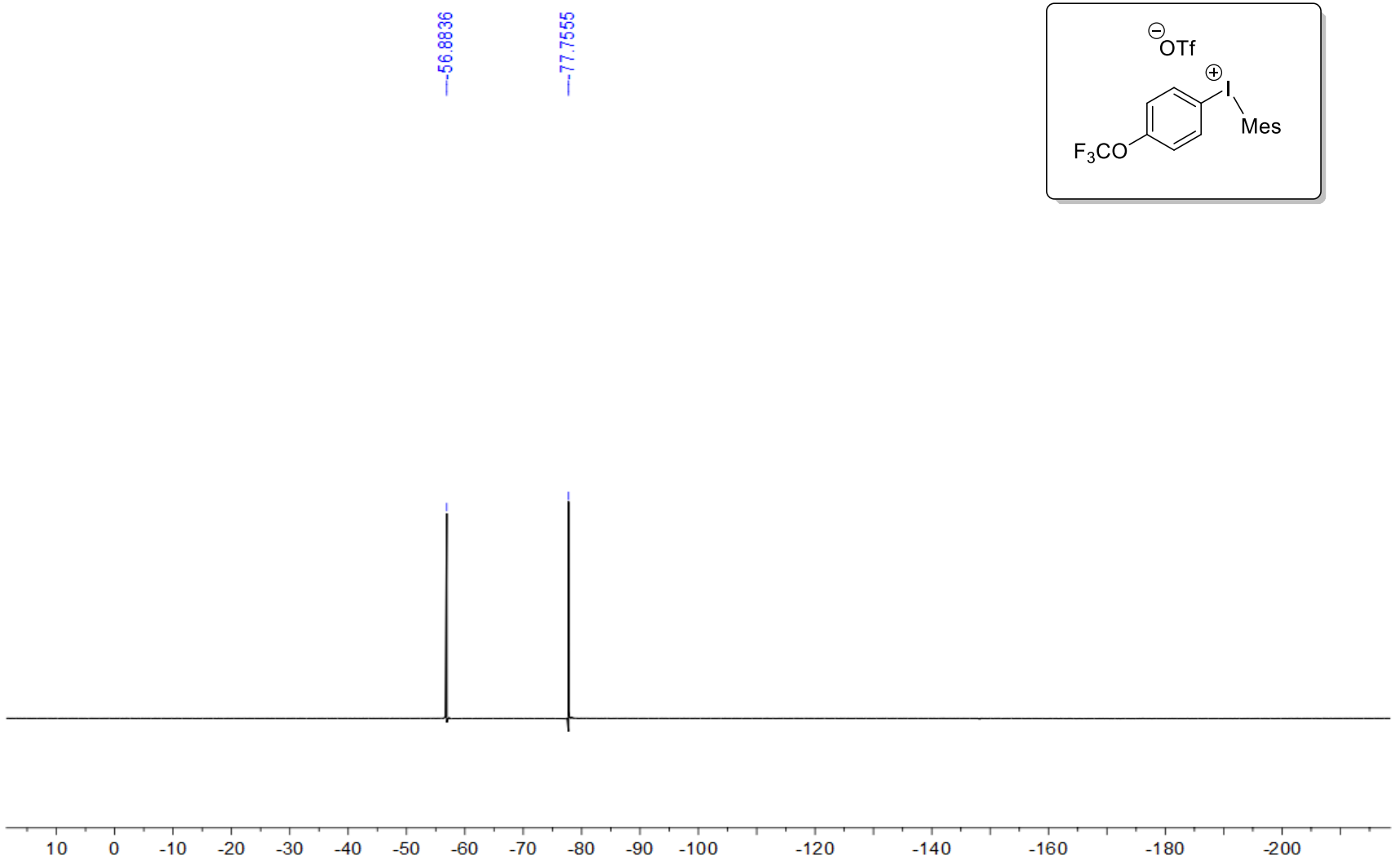
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **2g**



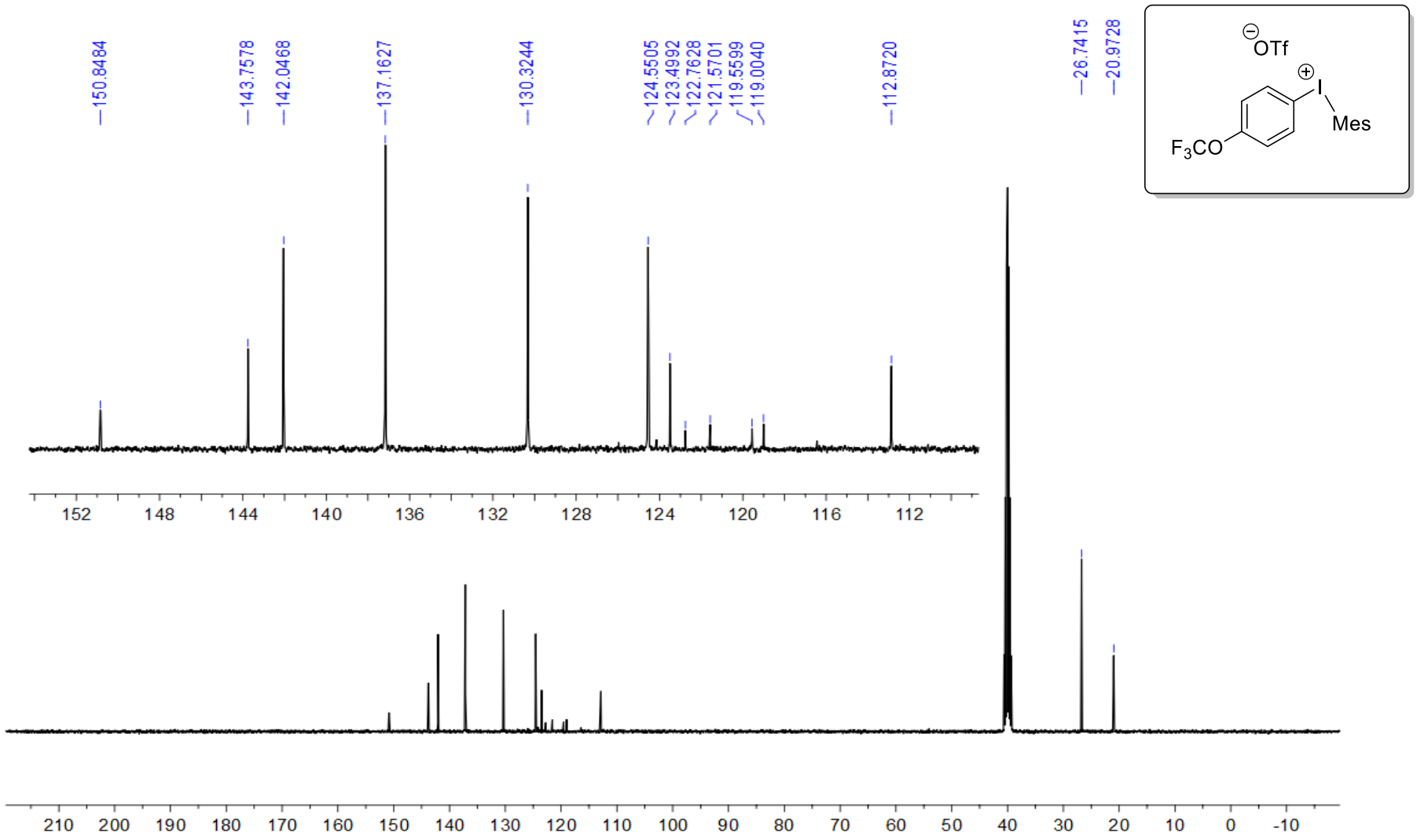
^{1}H NMR (400 MHz, [D6] DMSO): Compound **2h**



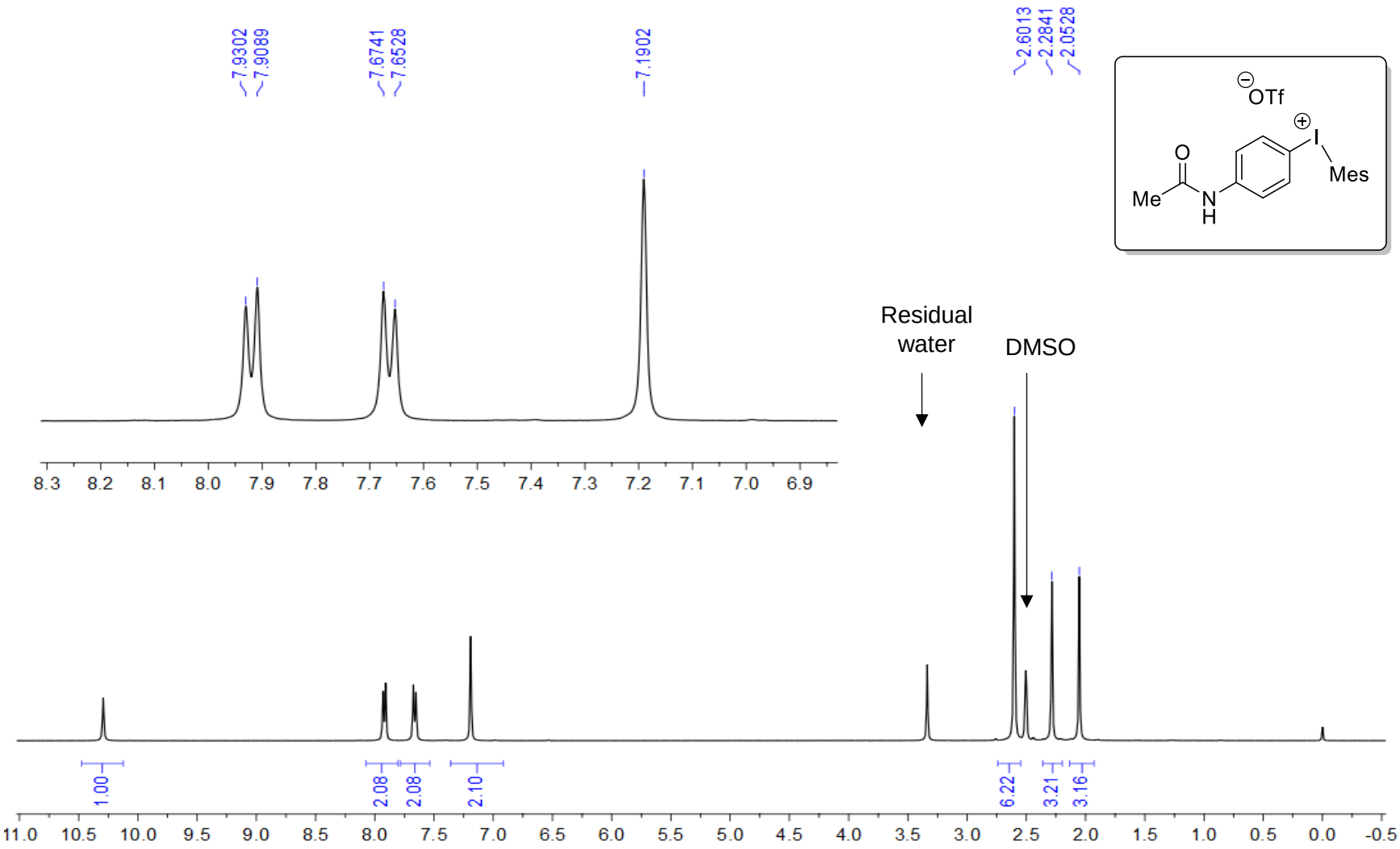
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **2h**



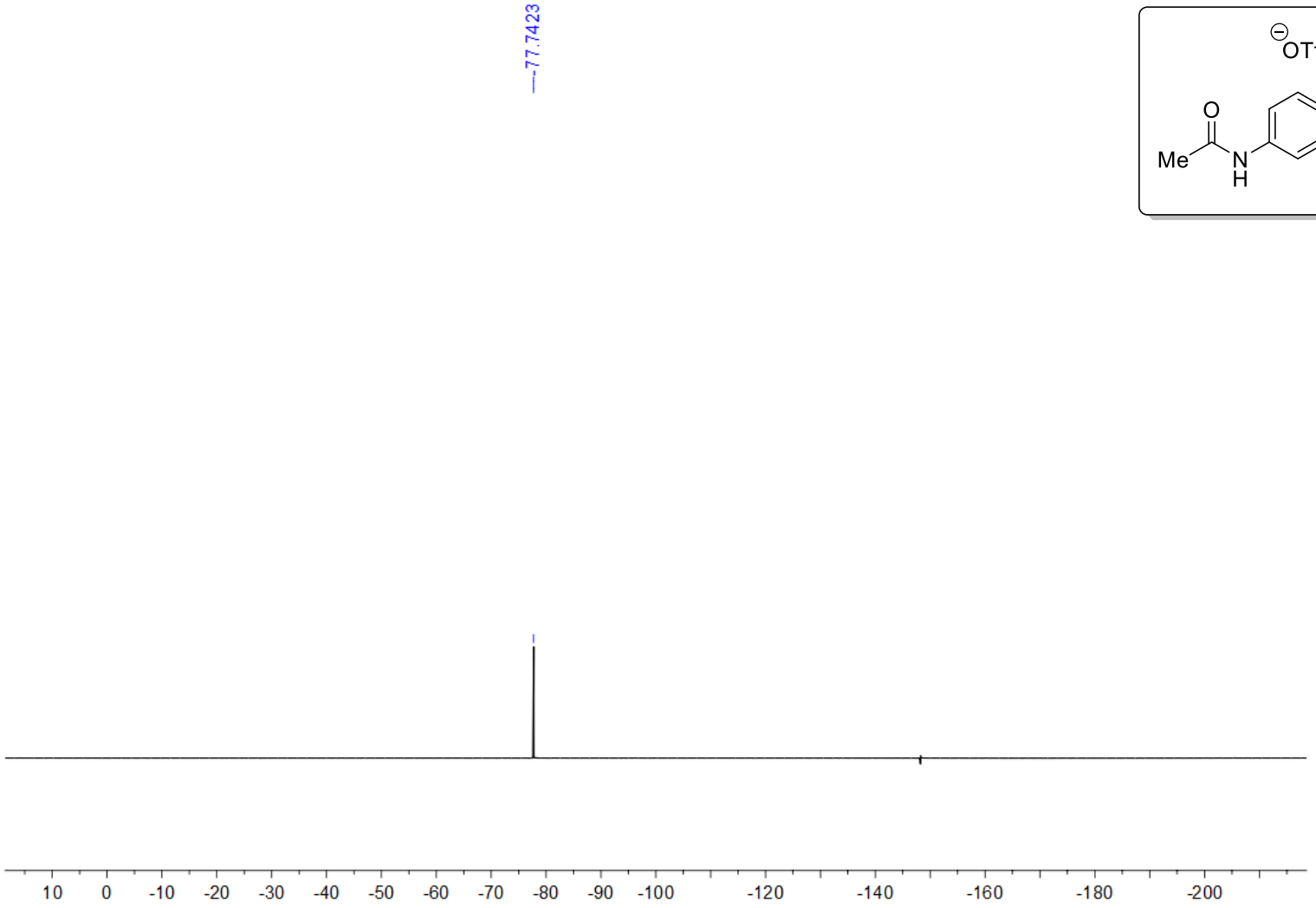
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **2h**



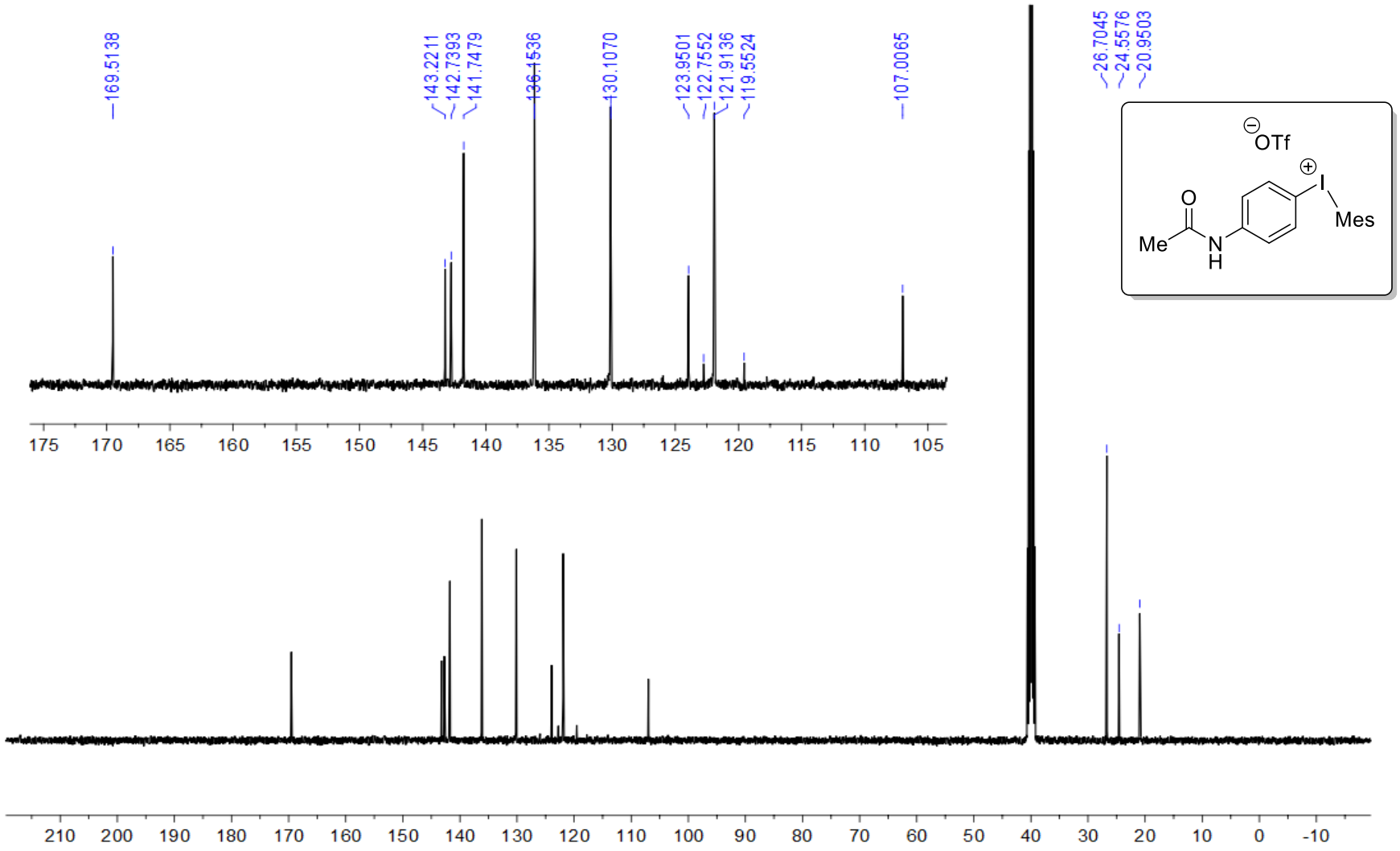
^{1}H NMR (400 MHz, [D6] DMSO): Compound **2i**



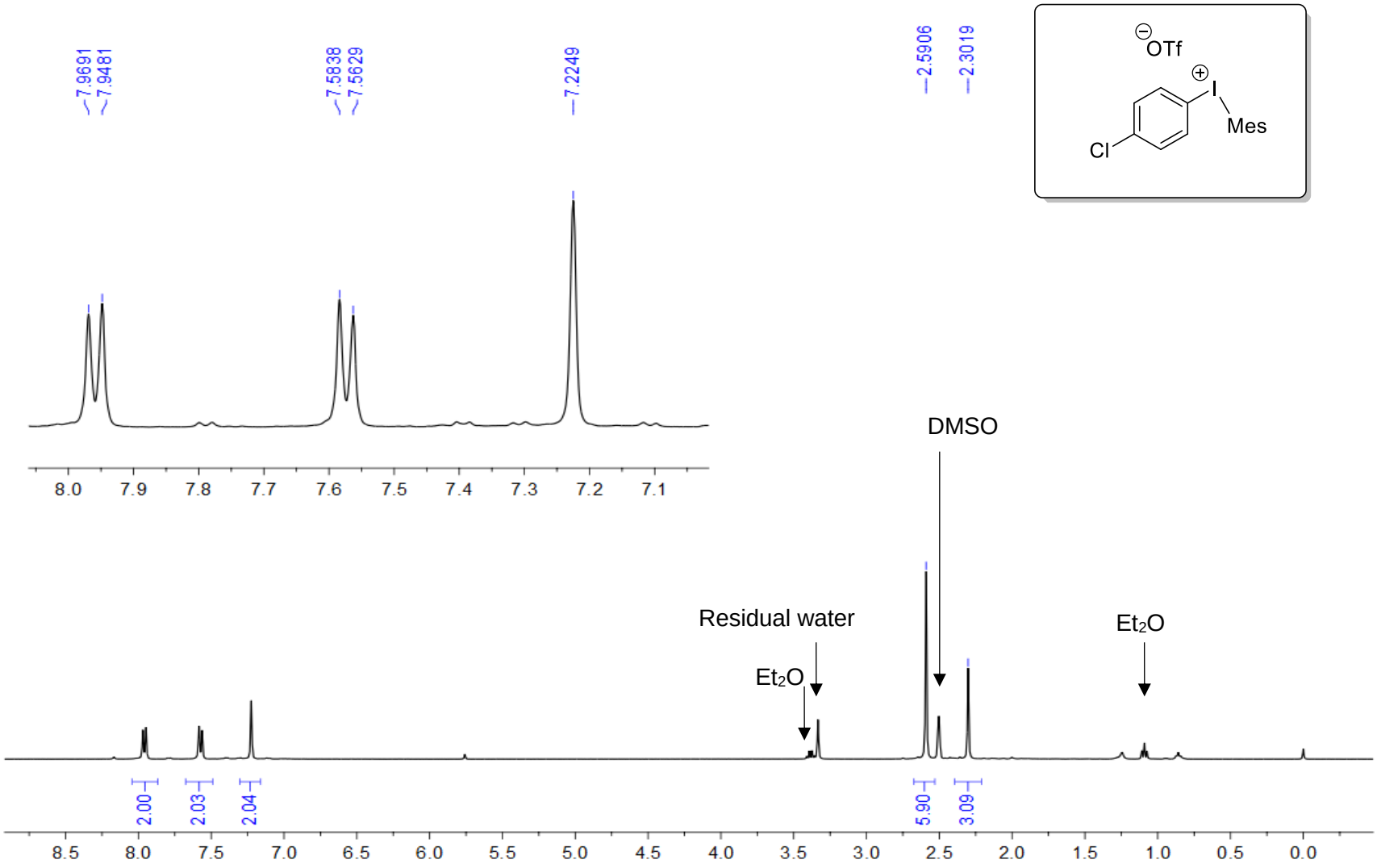
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **2i**



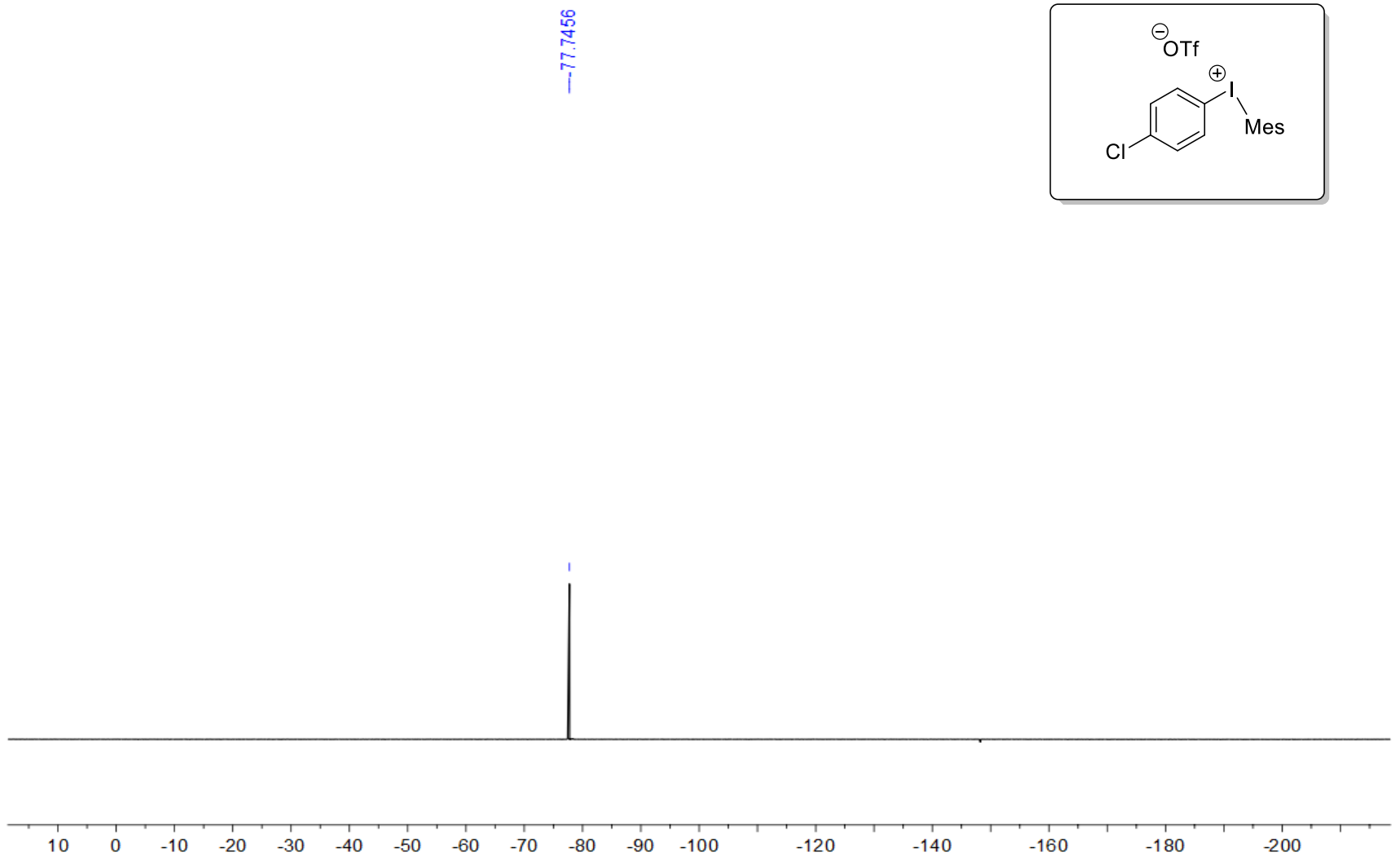
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **2i**



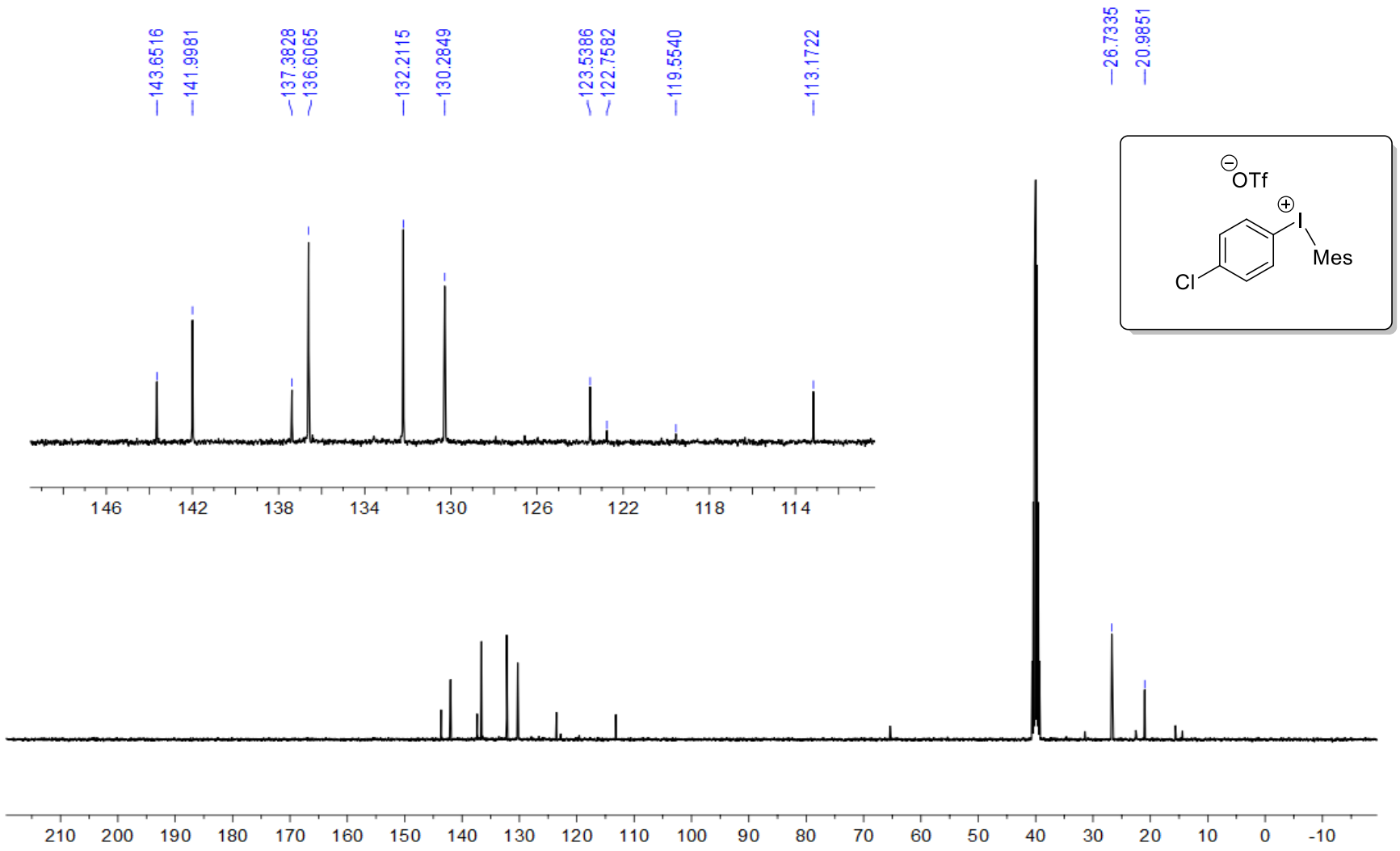
^{1}H NMR (400 MHz, [D6] DMSO): Compound **2j**



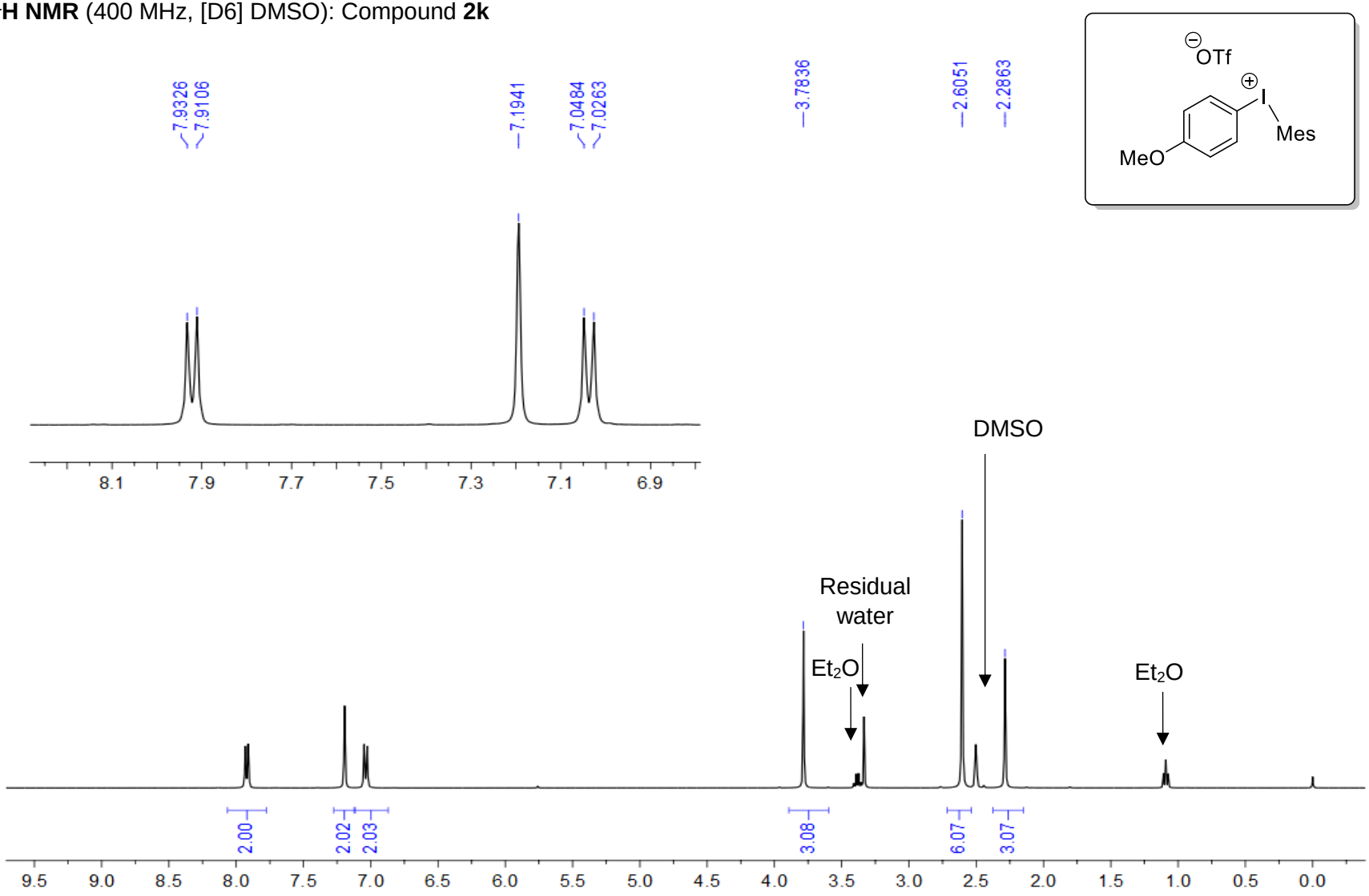
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **2j**



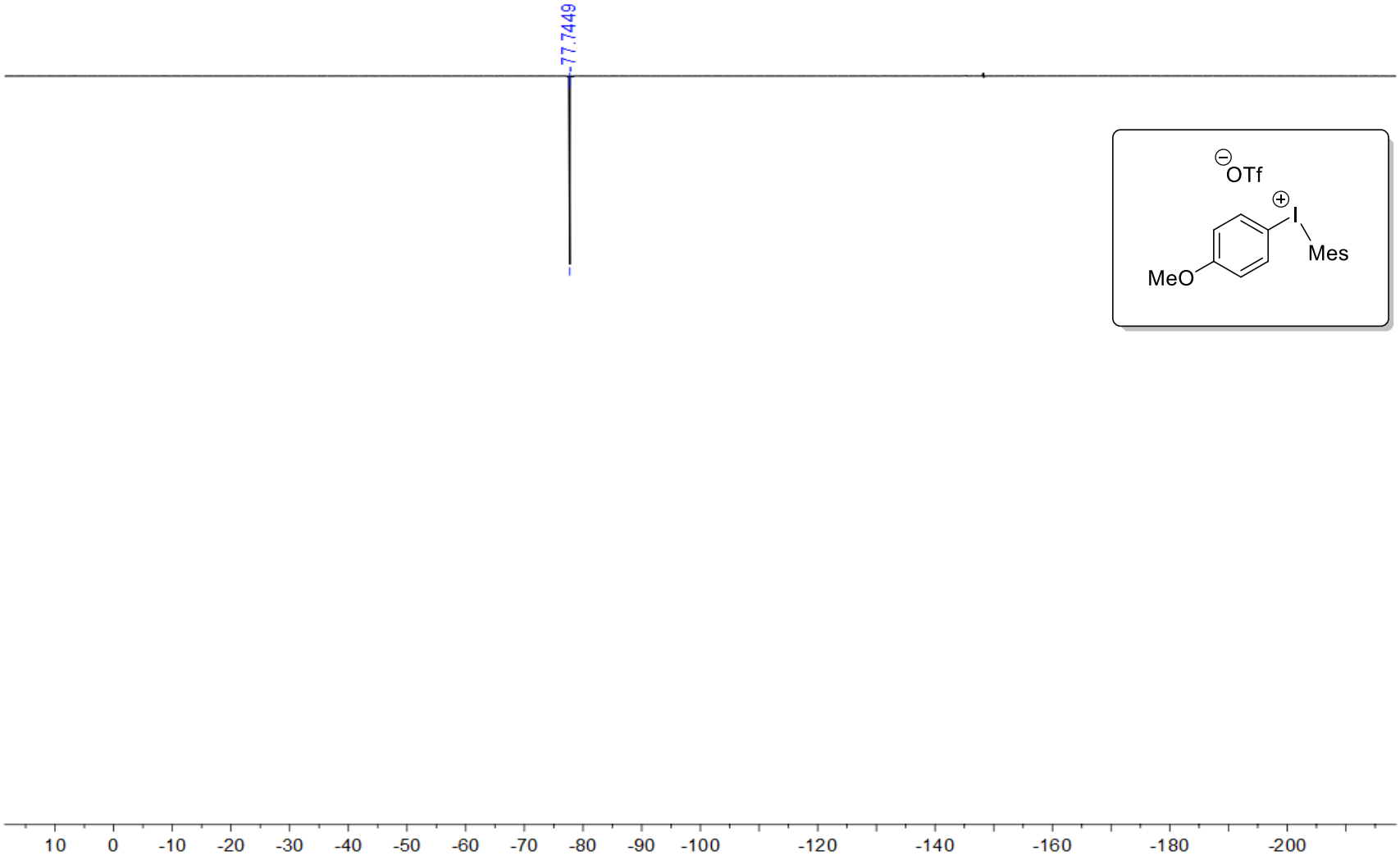
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **2j**



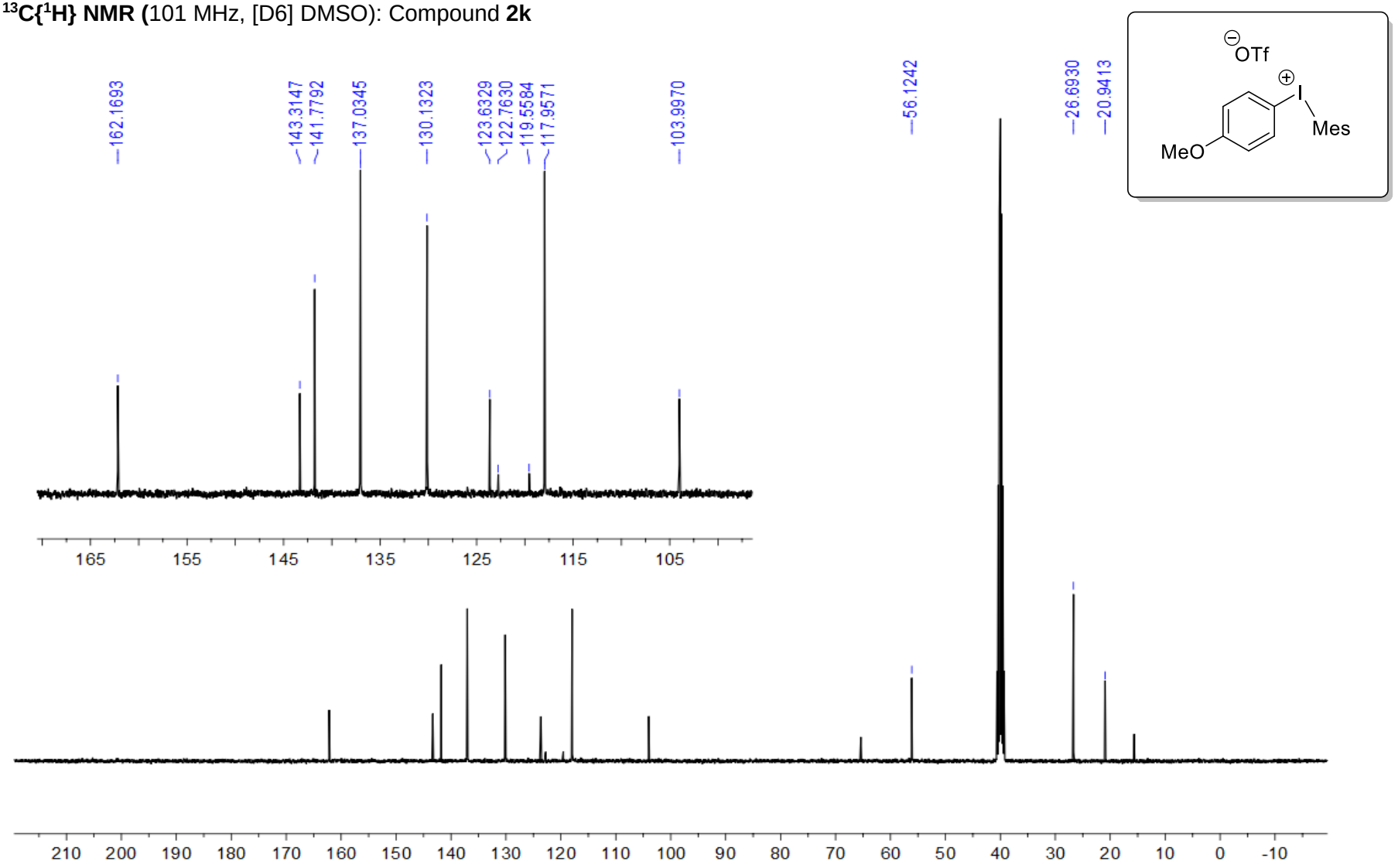
^{1}H NMR (400 MHz, [D6] DMSO): Compound **2k**



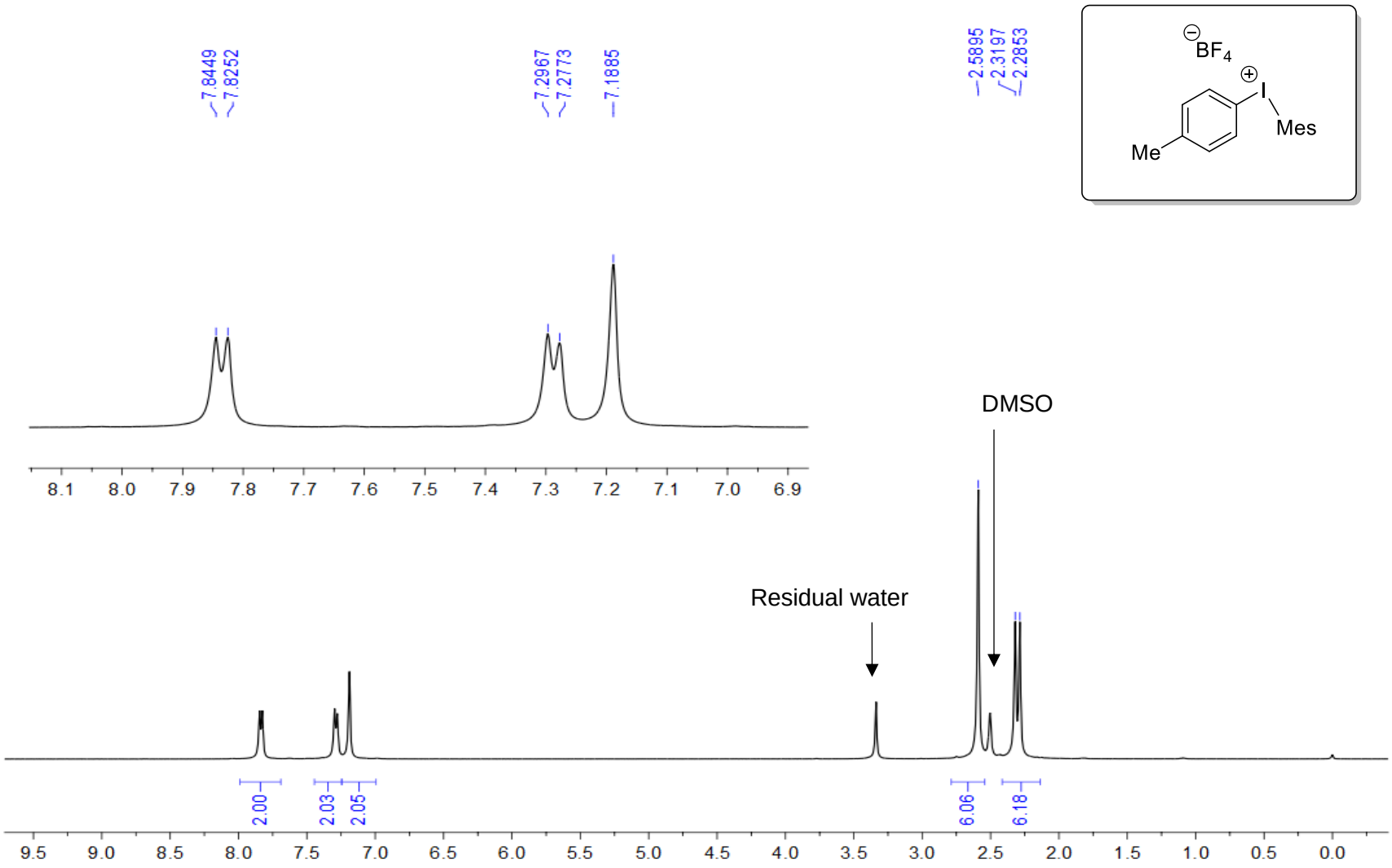
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **2k**



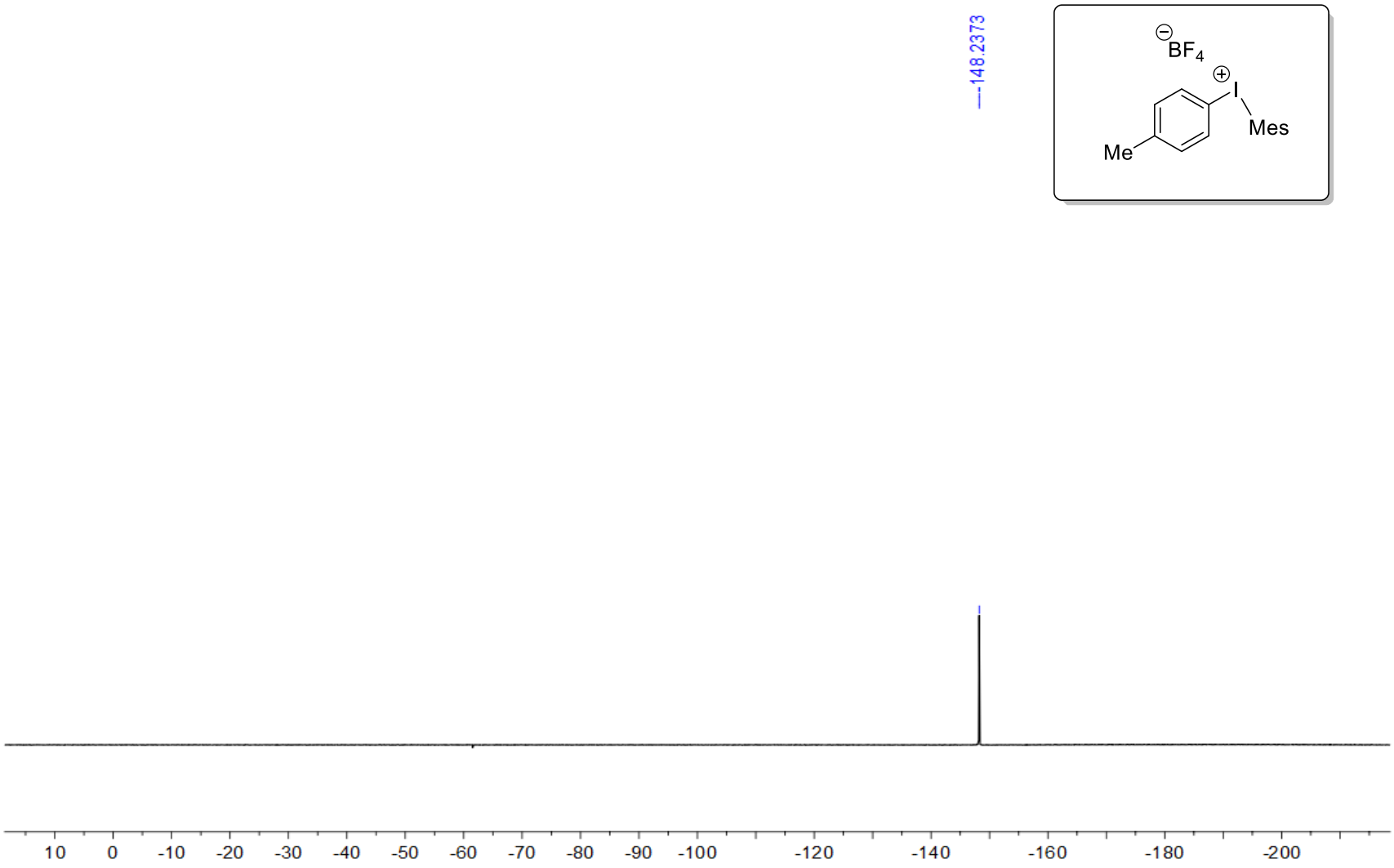
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **2k**



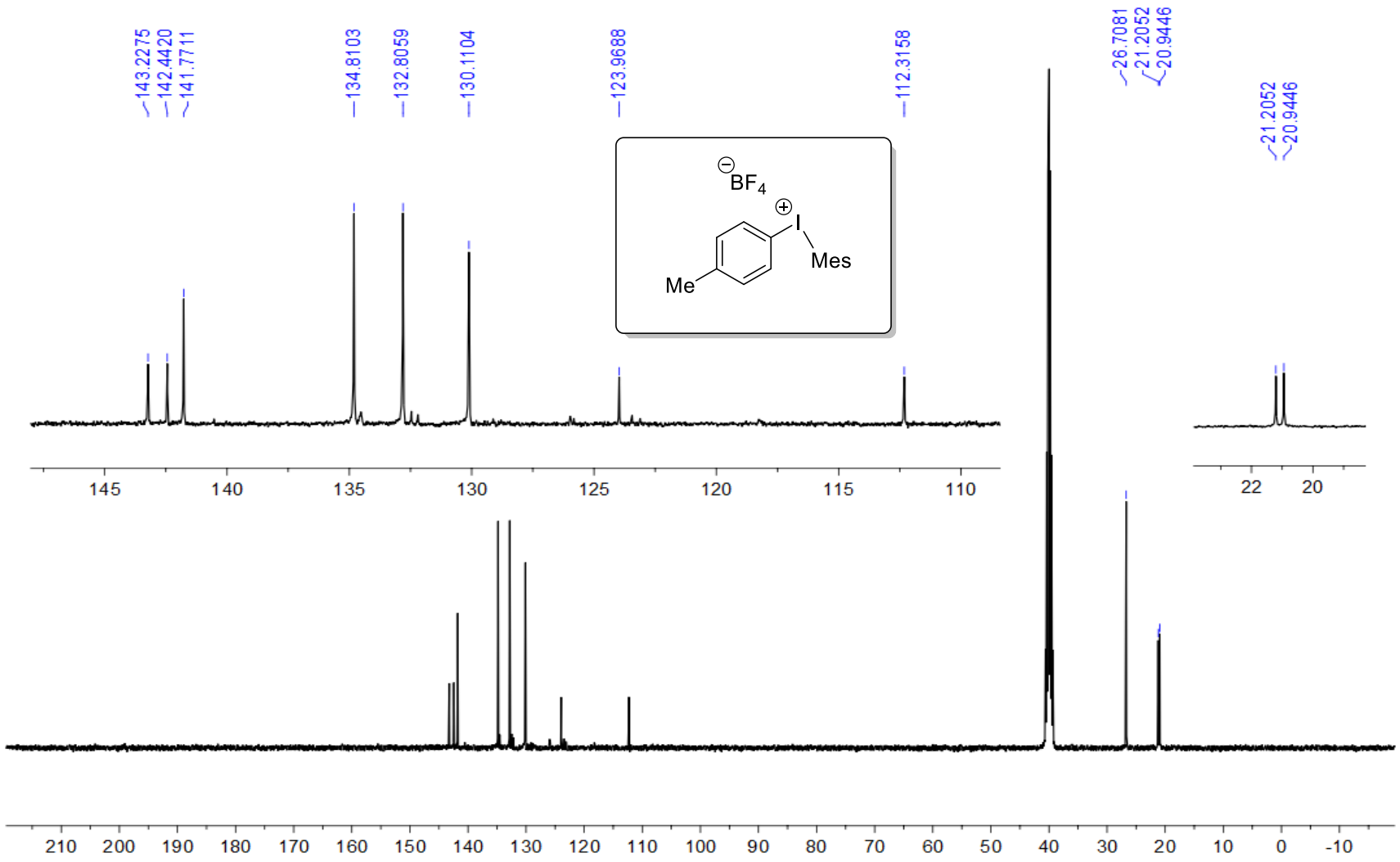
^{1}H NMR (400 MHz, [D6] DMSO): Compound **2l**



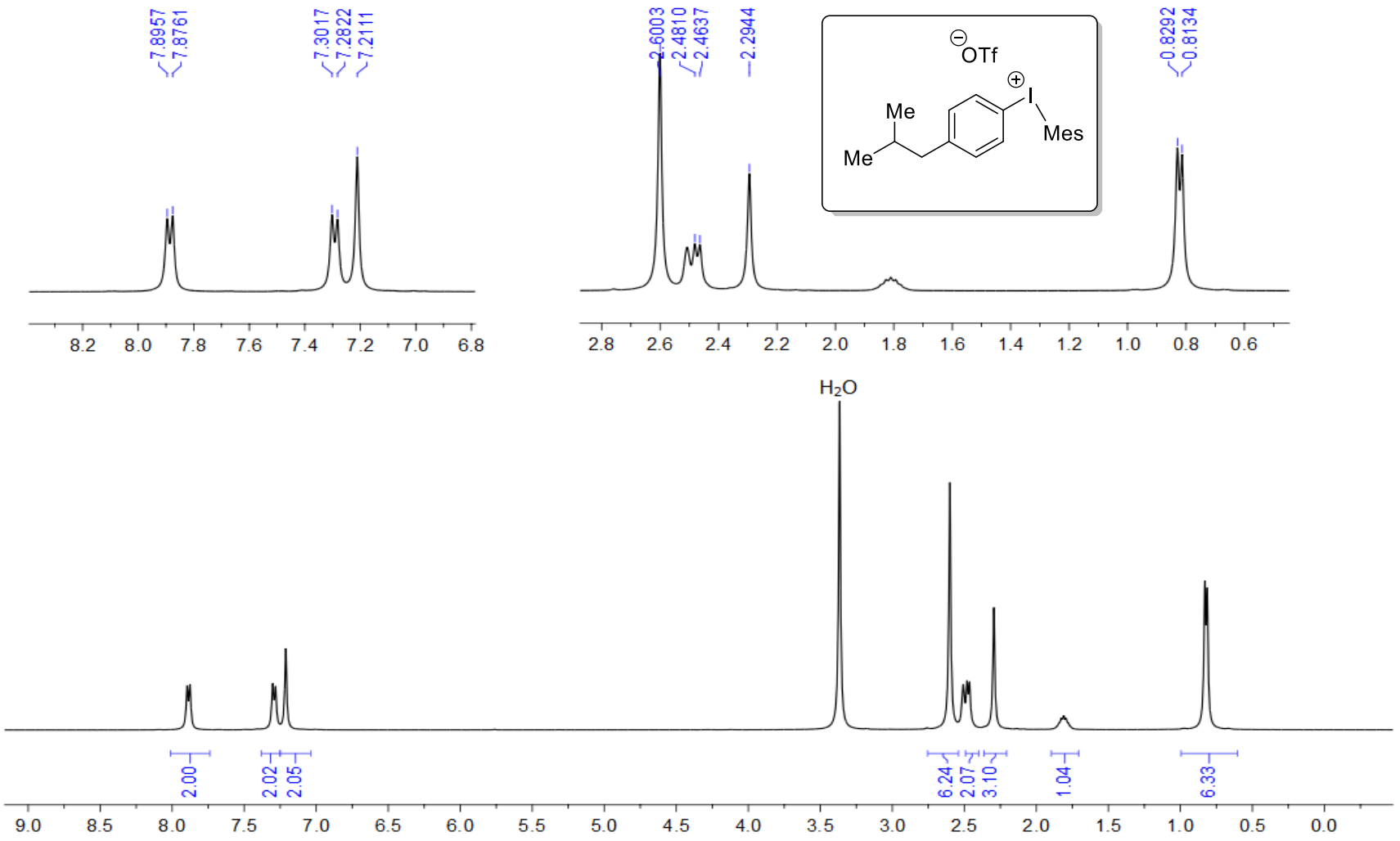
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **2l**



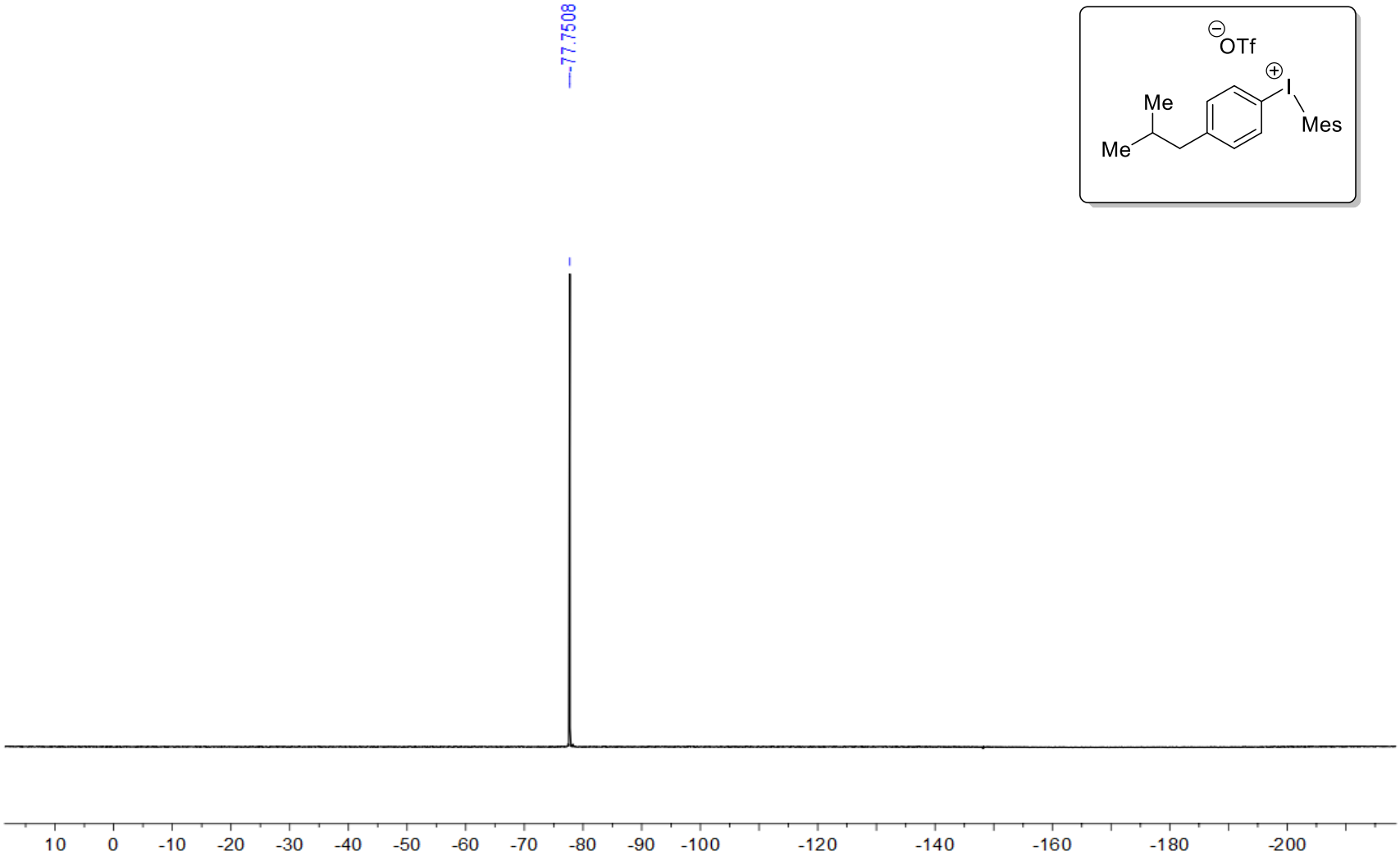
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **2l**



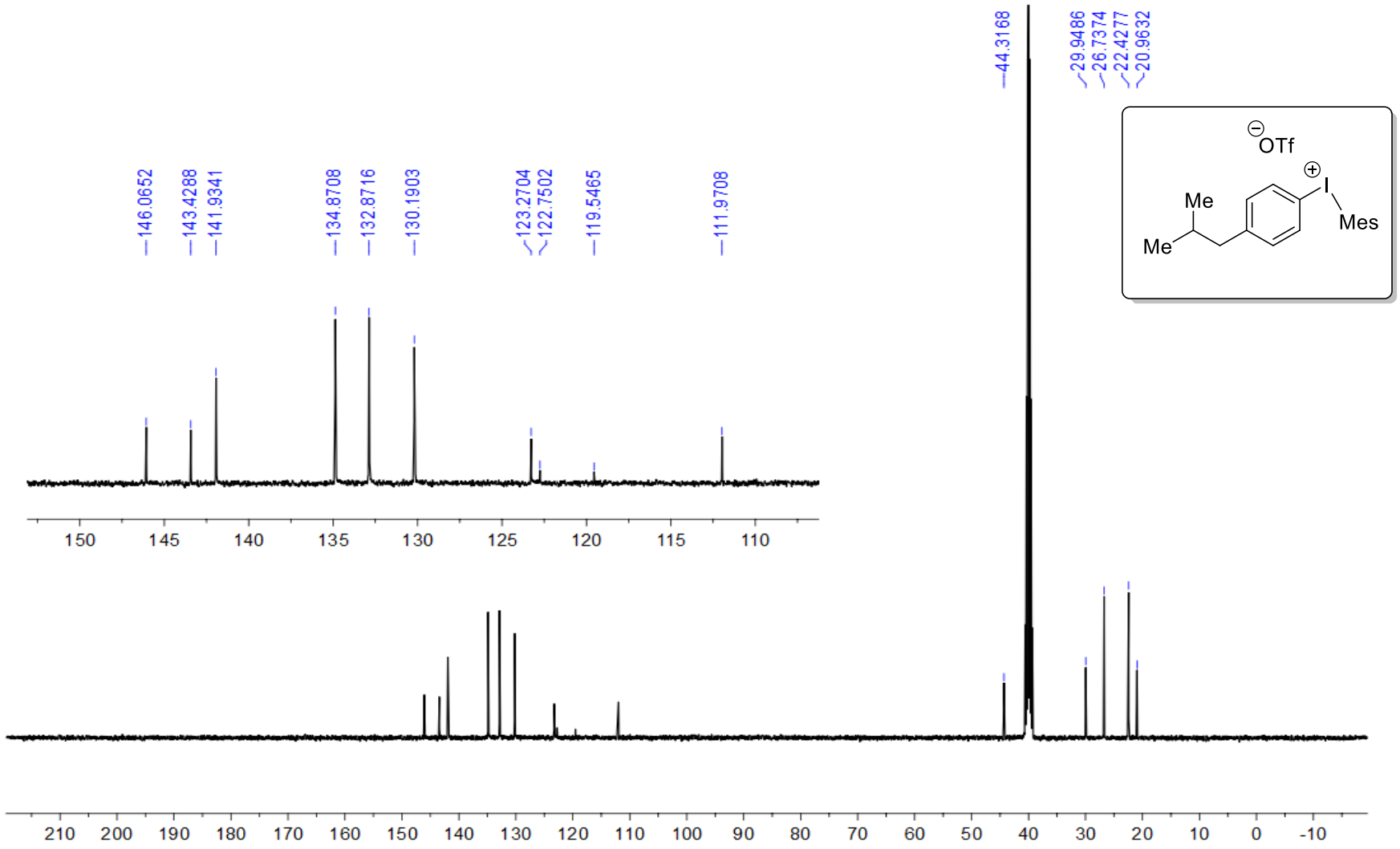
^{1}H NMR (400 MHz, [D6] DMSO): Compound **2m**



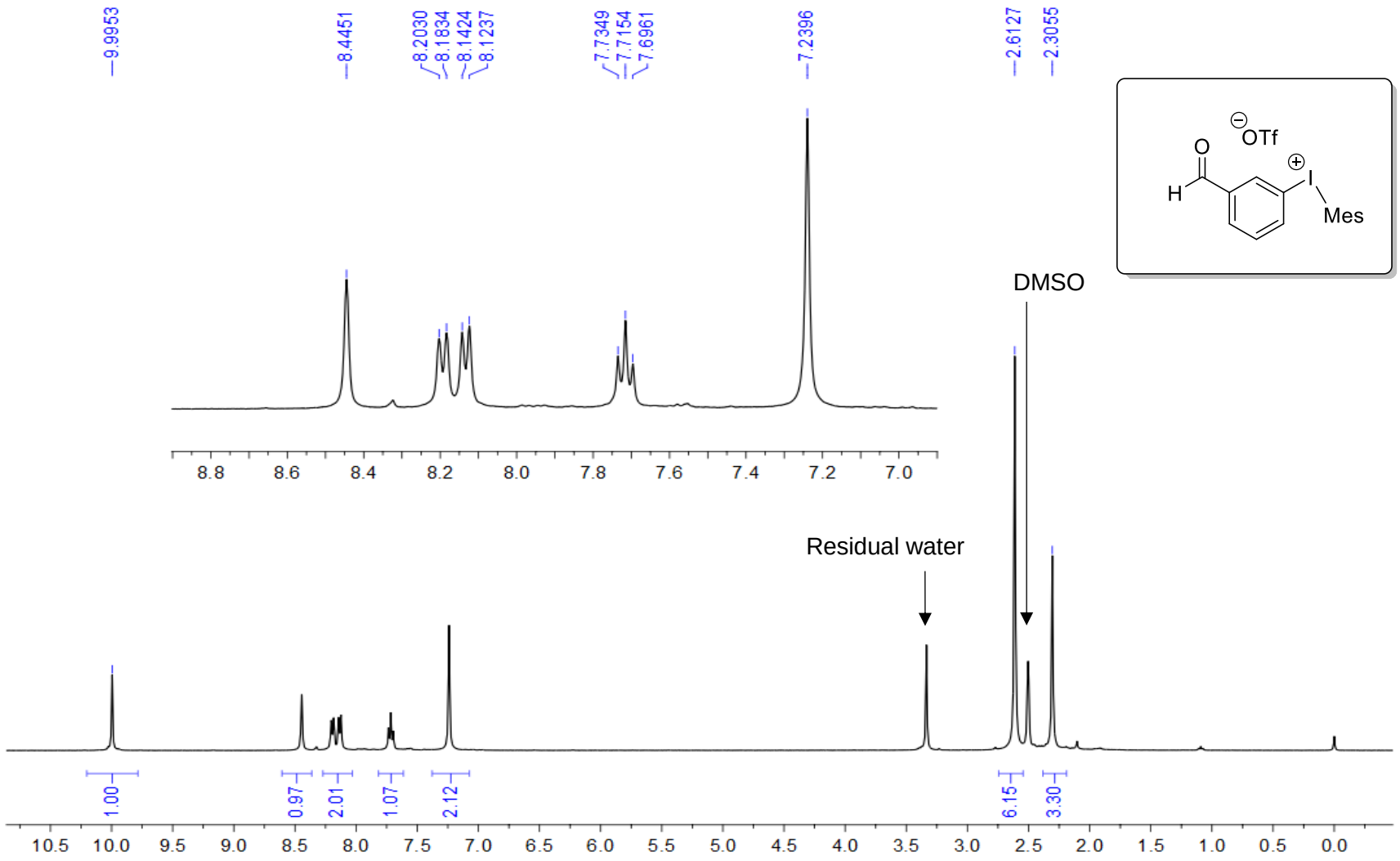
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **2m**



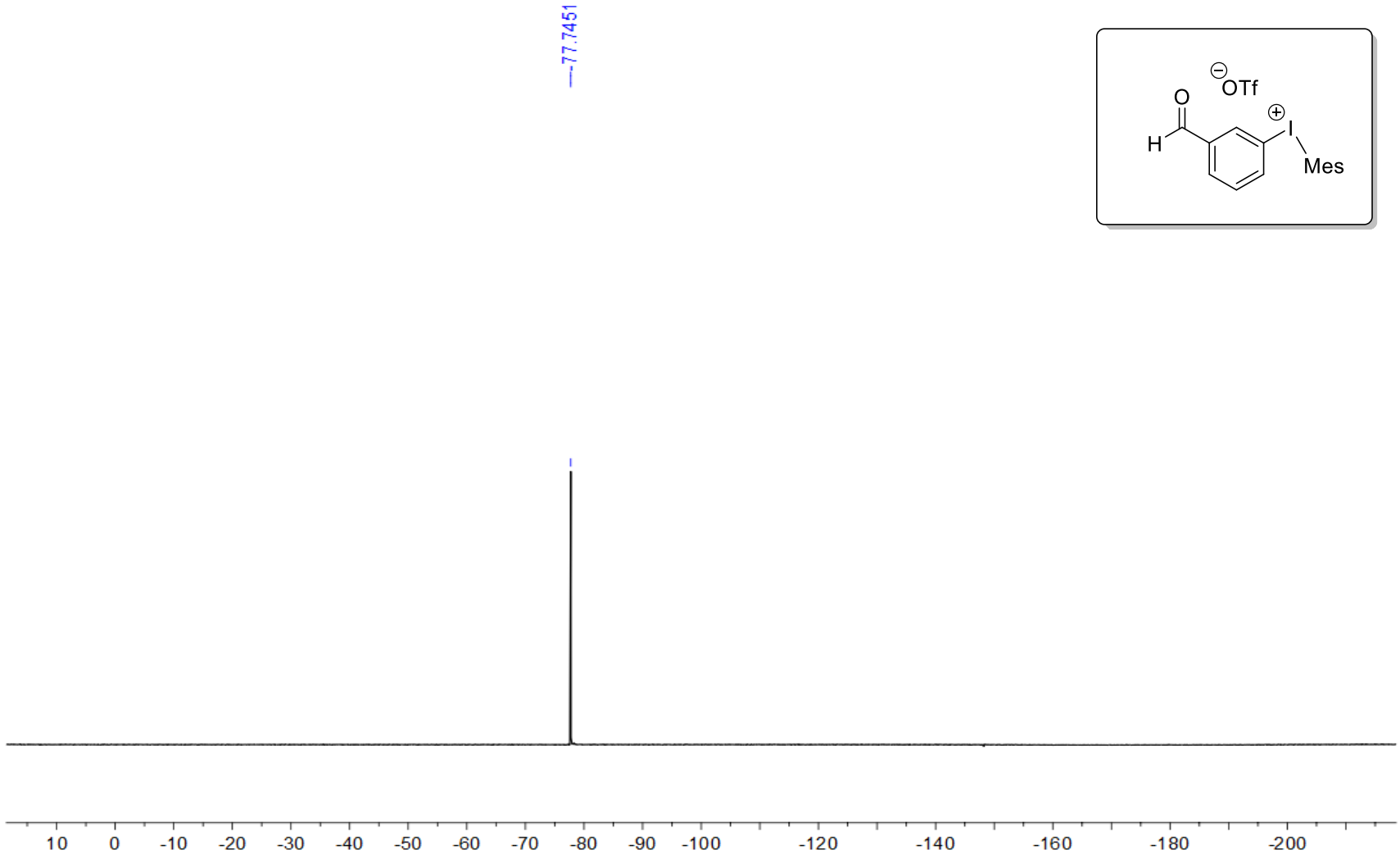
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **2m**



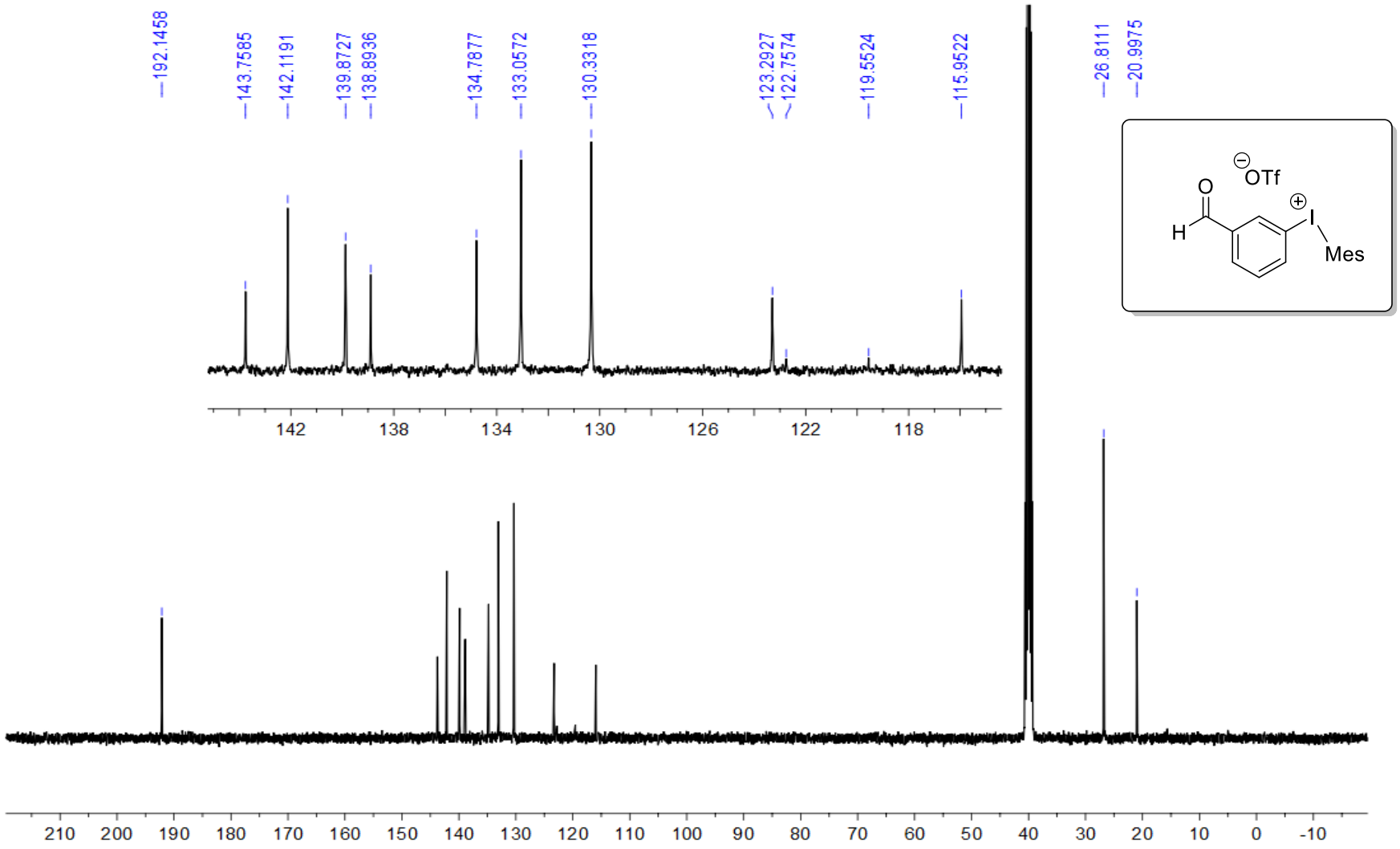
^{1}H NMR (400 MHz, [D6] DMSO): Compound **2n**



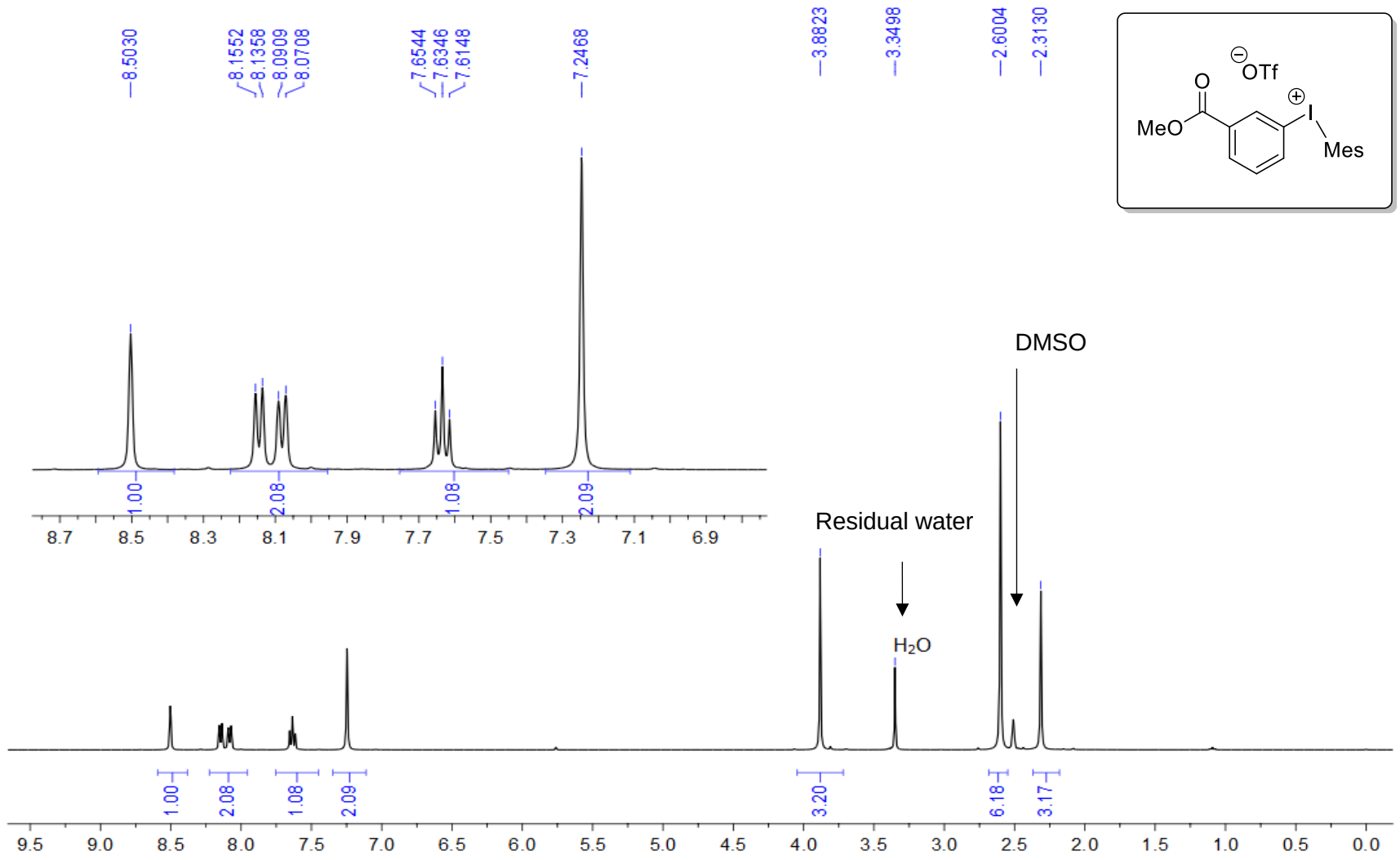
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **2n**



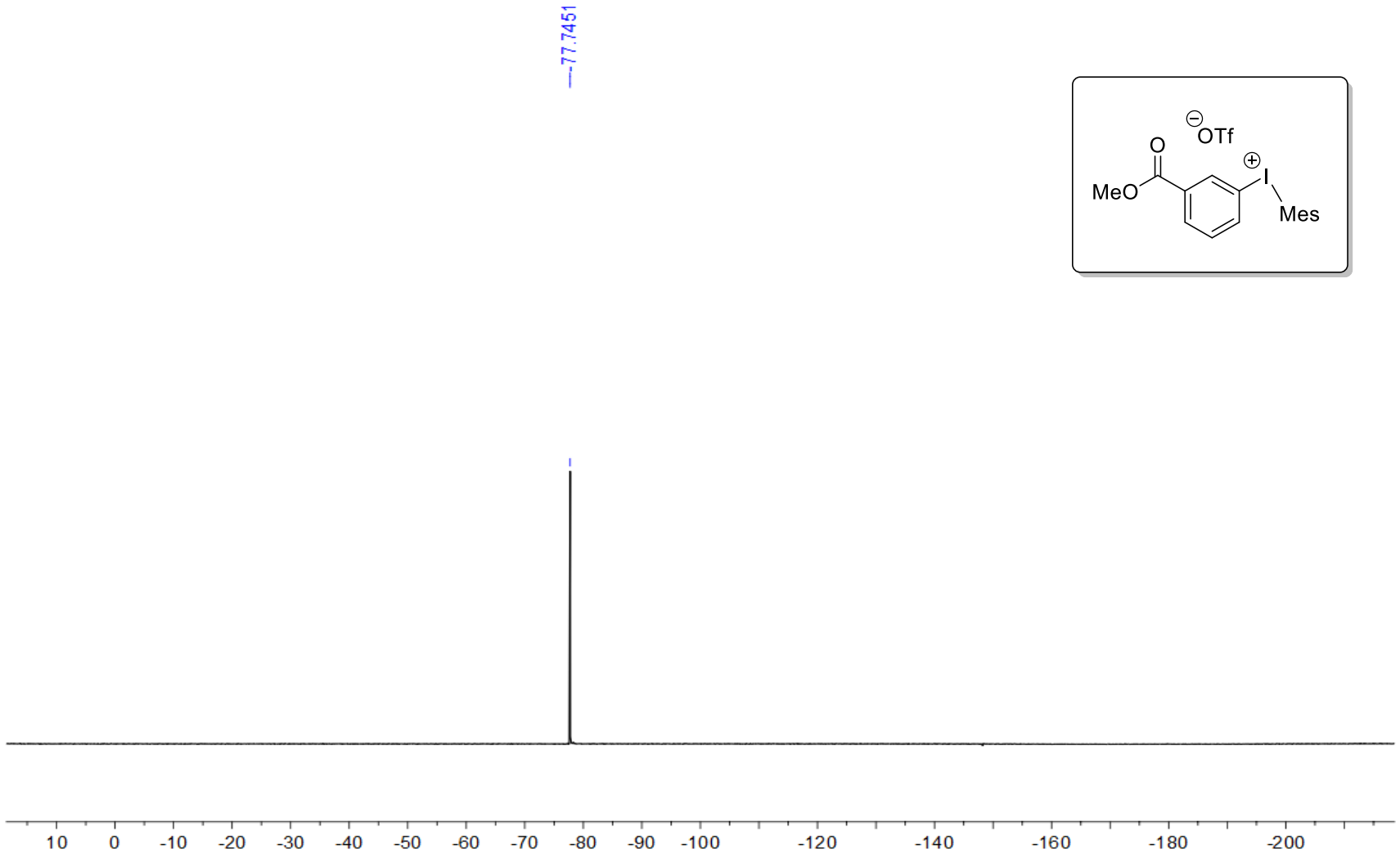
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **2n**



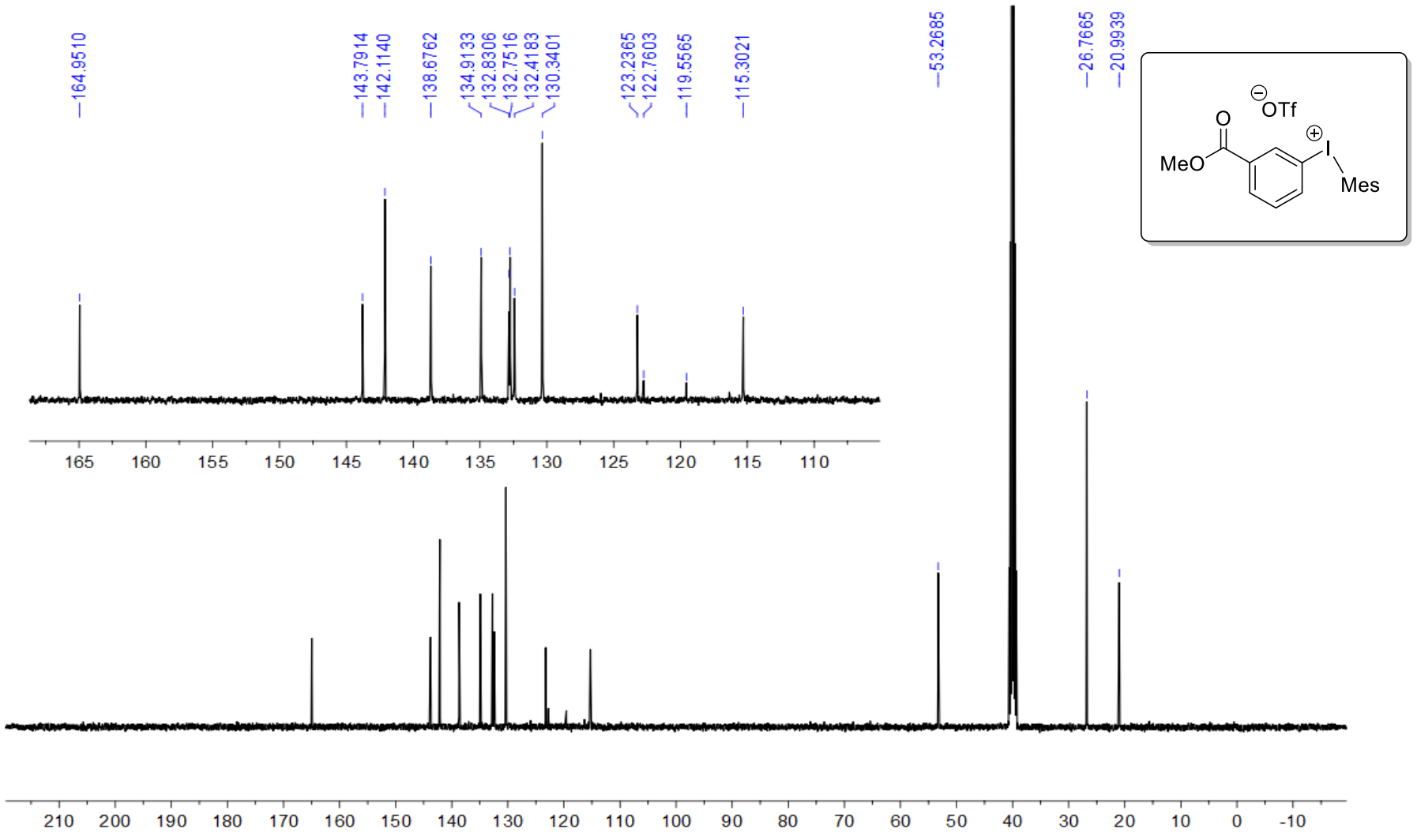
^{1}H NMR (400 MHz, [D6] DMSO): Compound **2o**



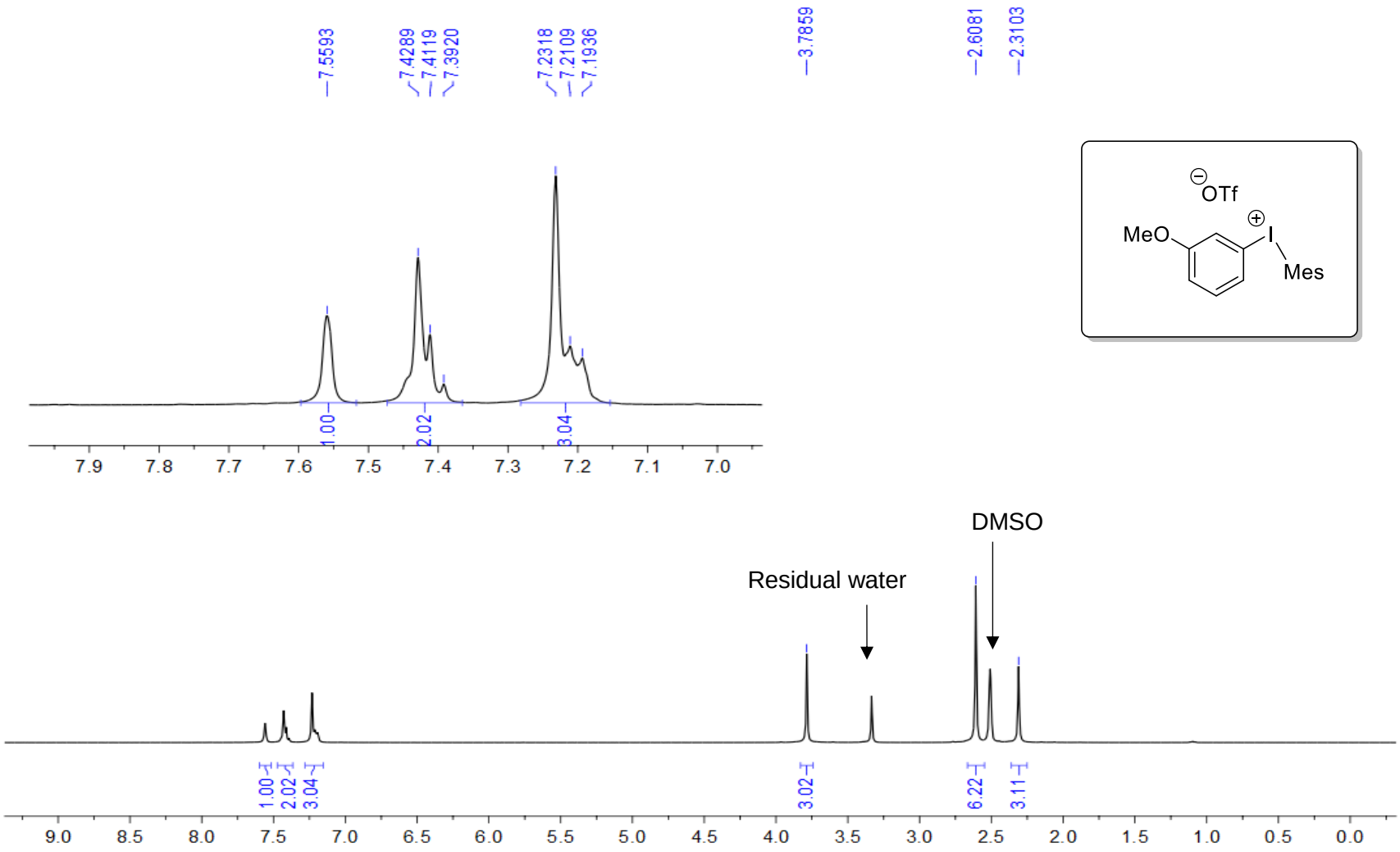
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **2o**



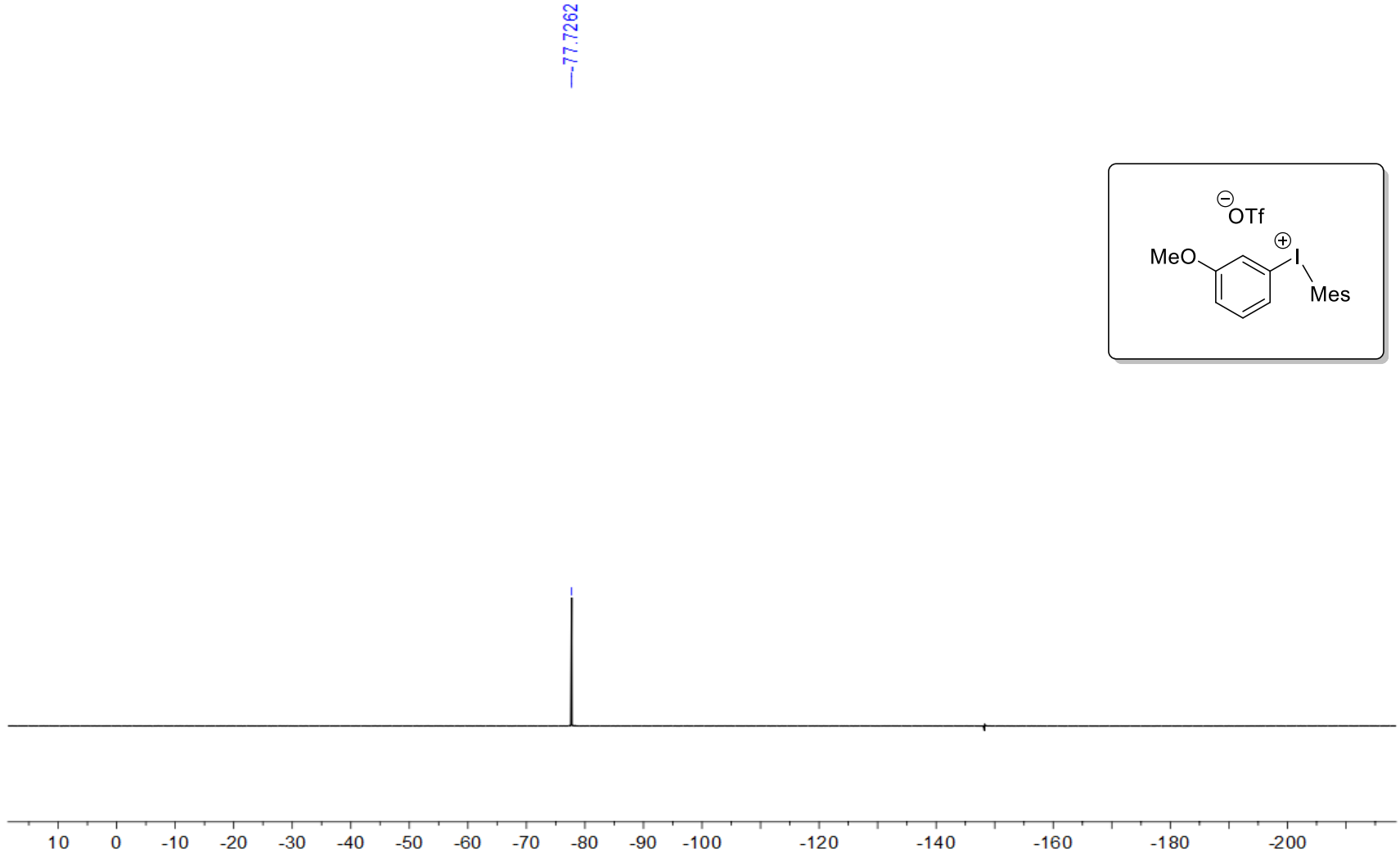
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **2o**



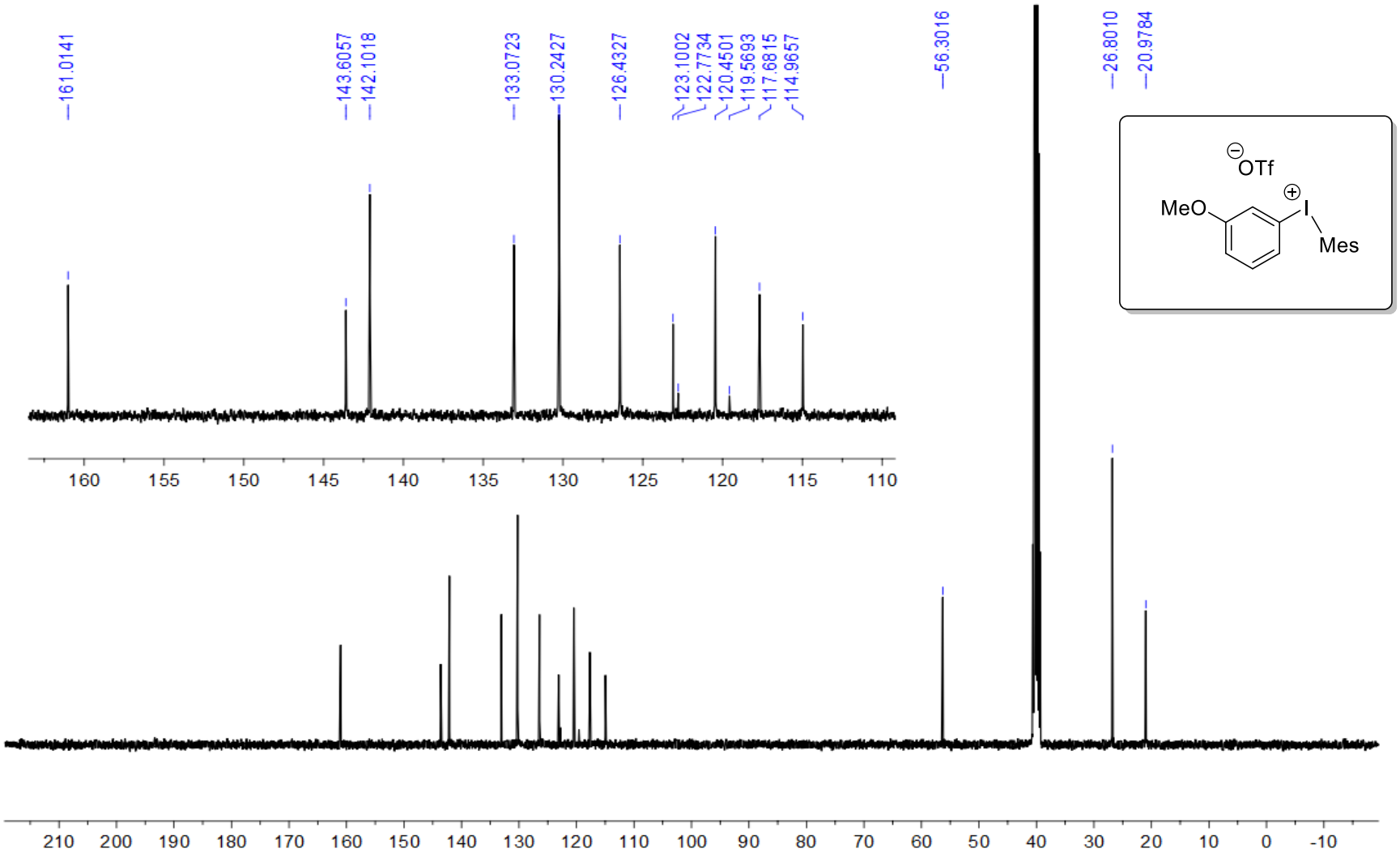
^{1}H NMR (400 MHz, [D6] DMSO): Compound **2p**



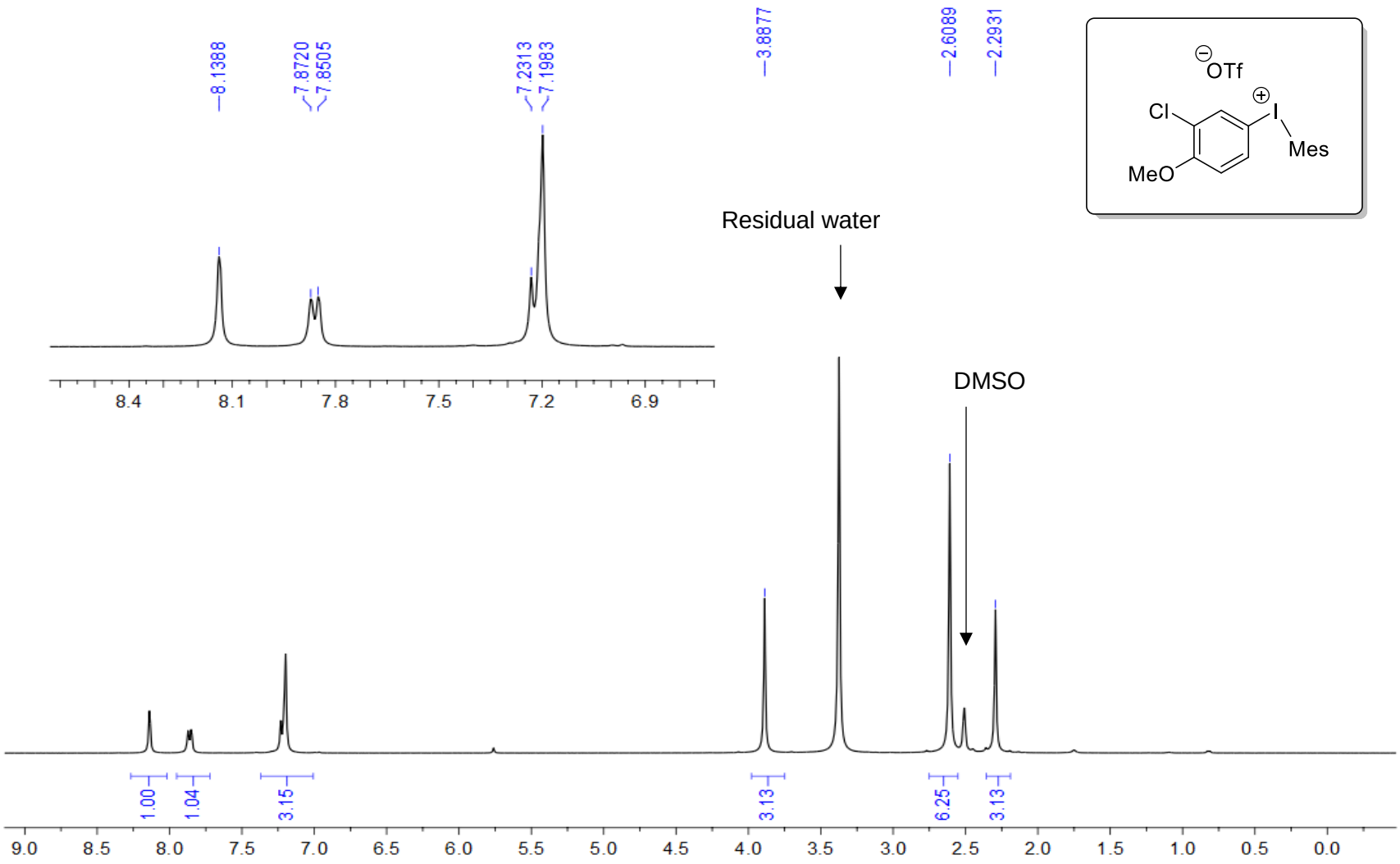
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **2p**



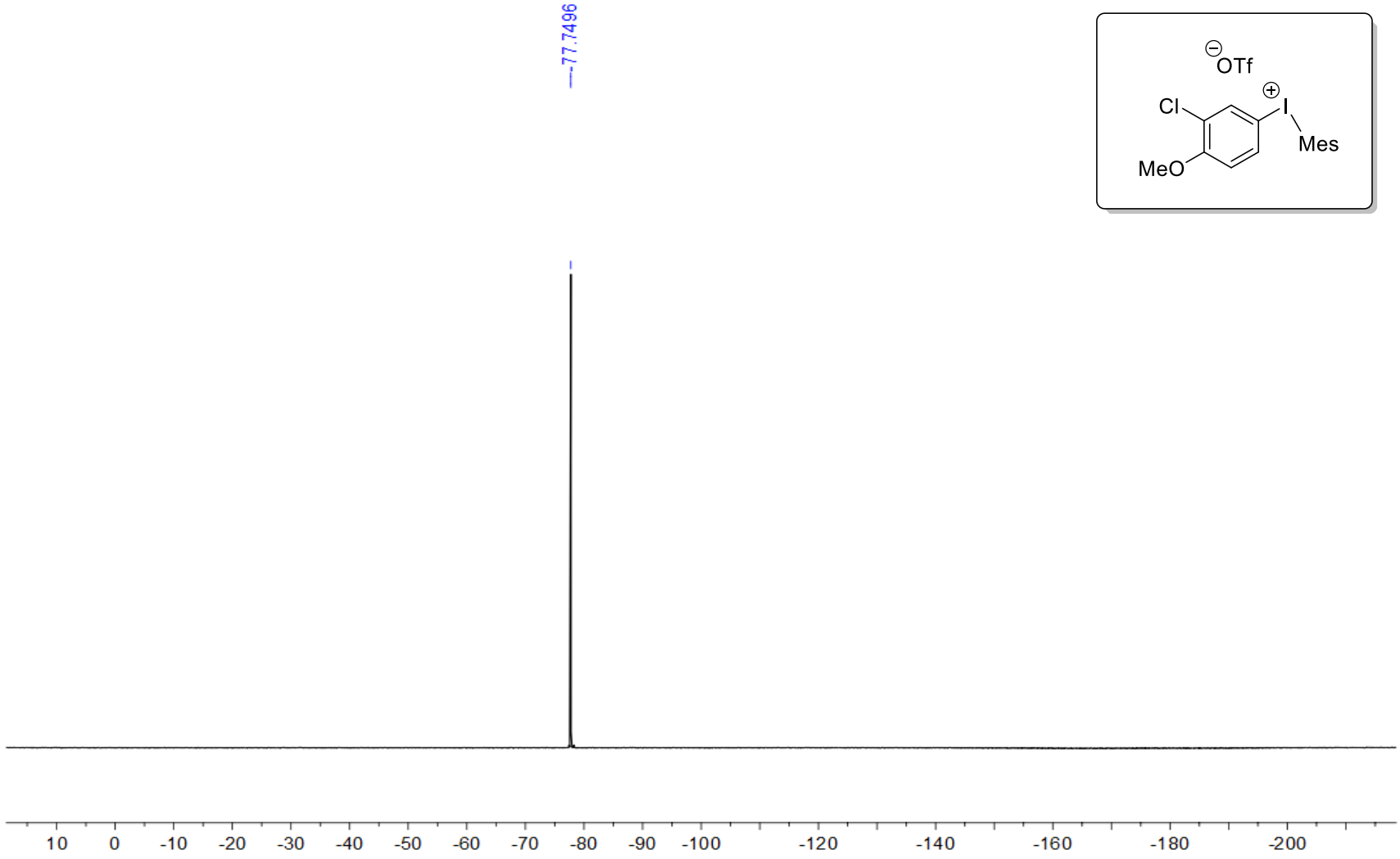
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **2p**



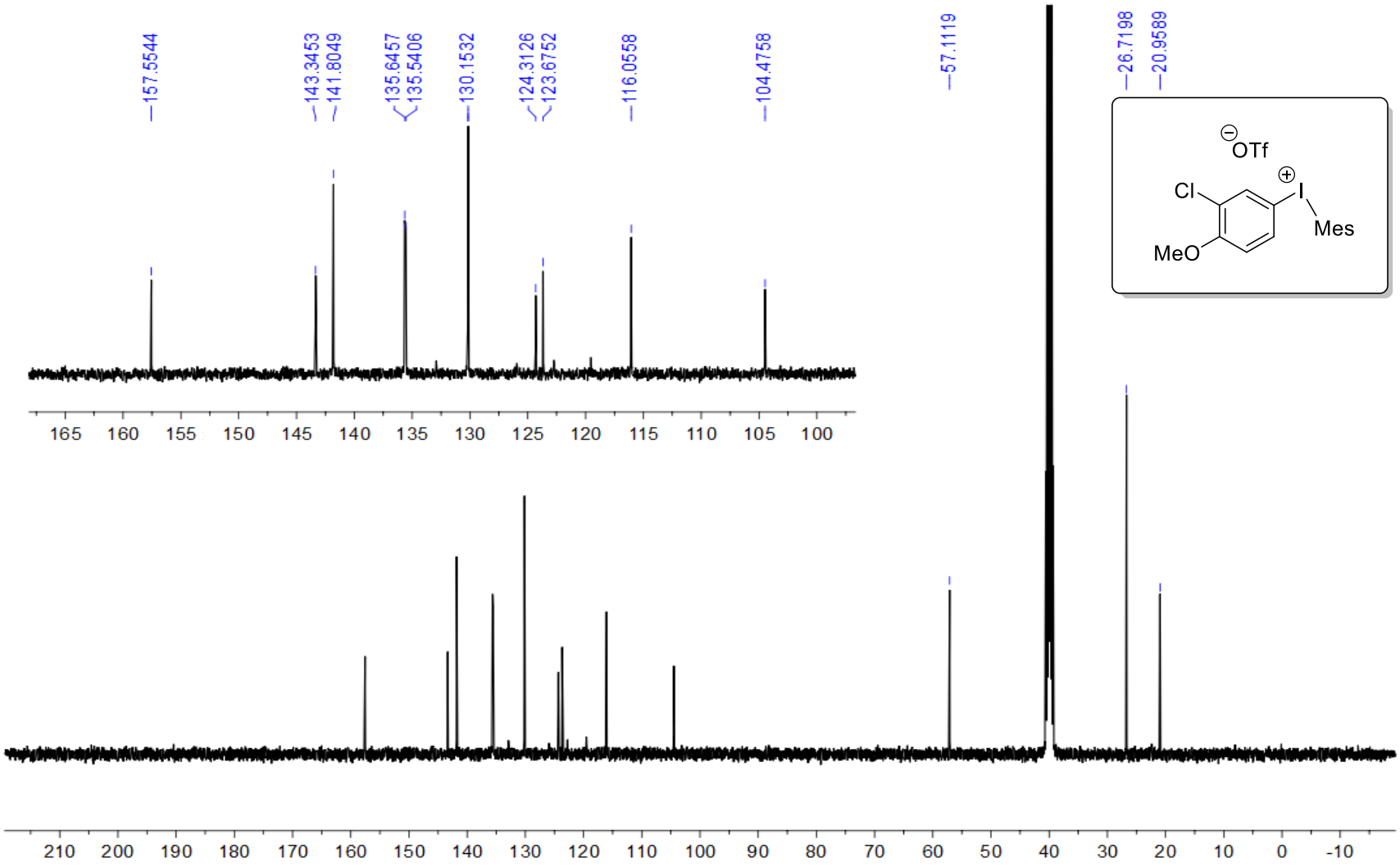
^{1}H NMR (400 MHz, [D6] DMSO): Compound **2q**



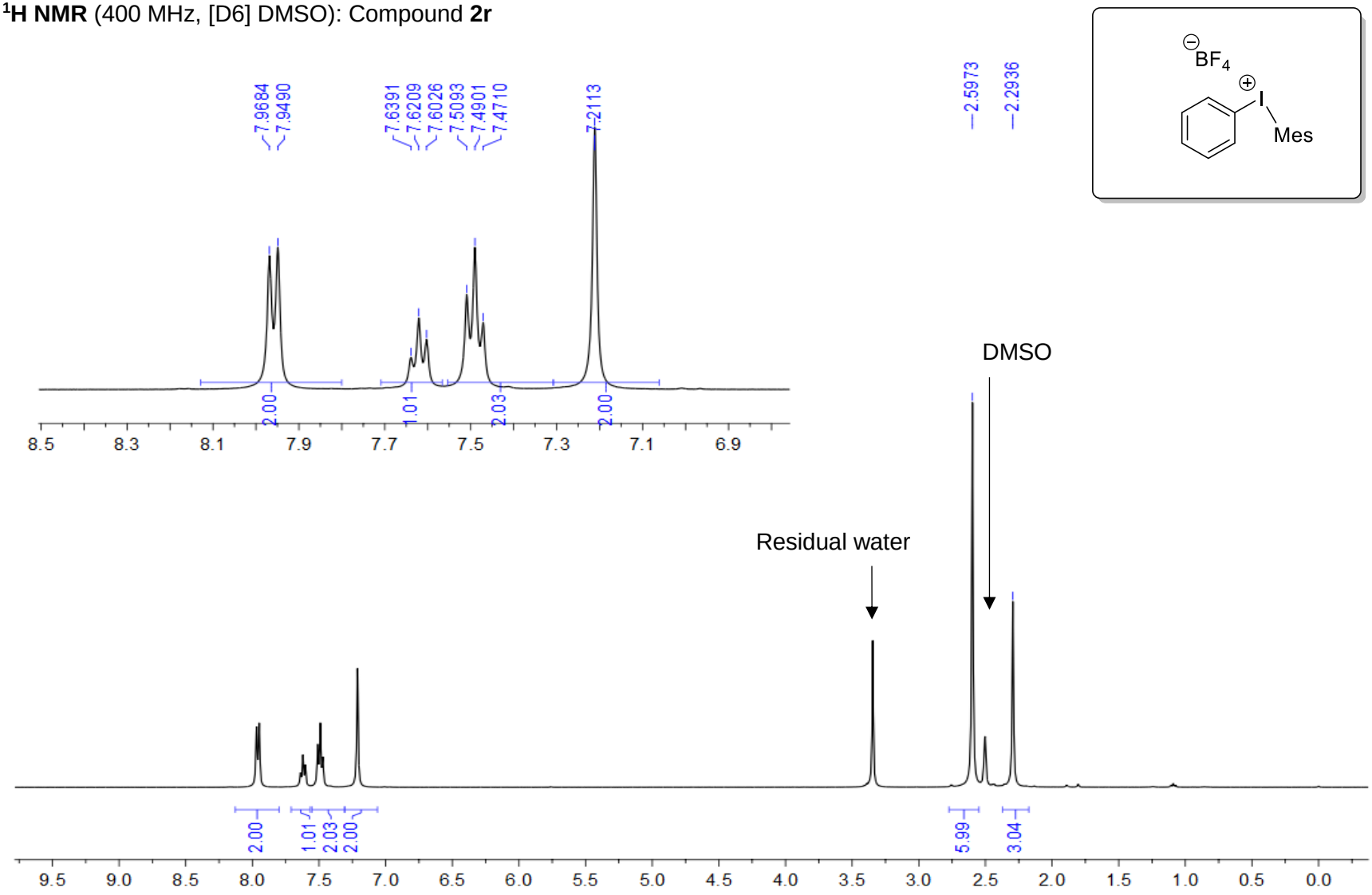
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **2q**



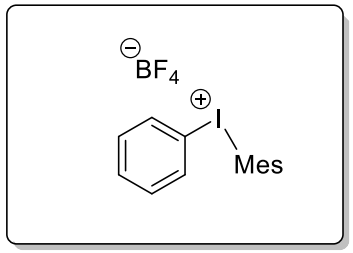
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **2q**



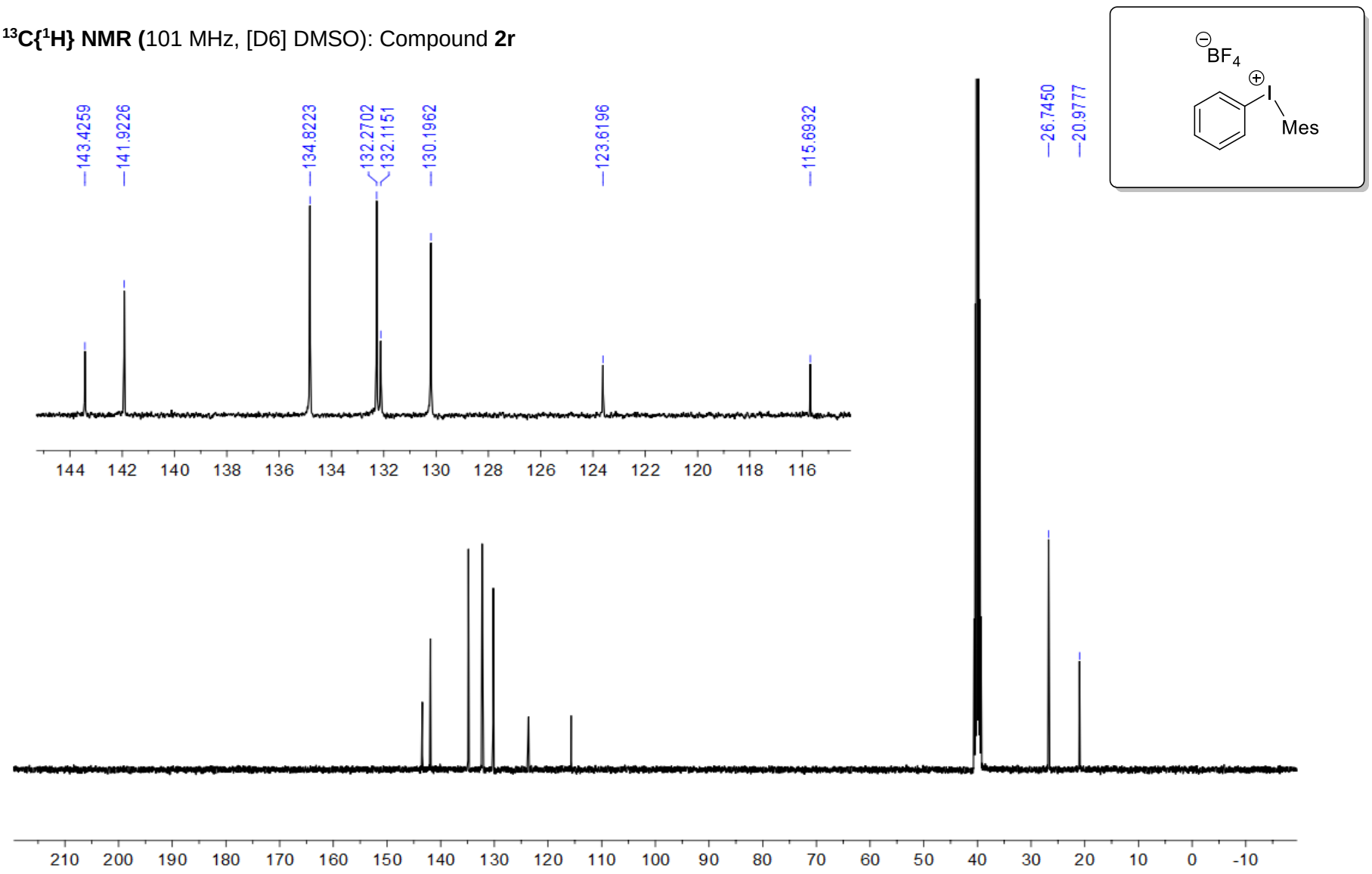
^{1}H NMR (400 MHz, [D6] DMSO): Compound **2r**



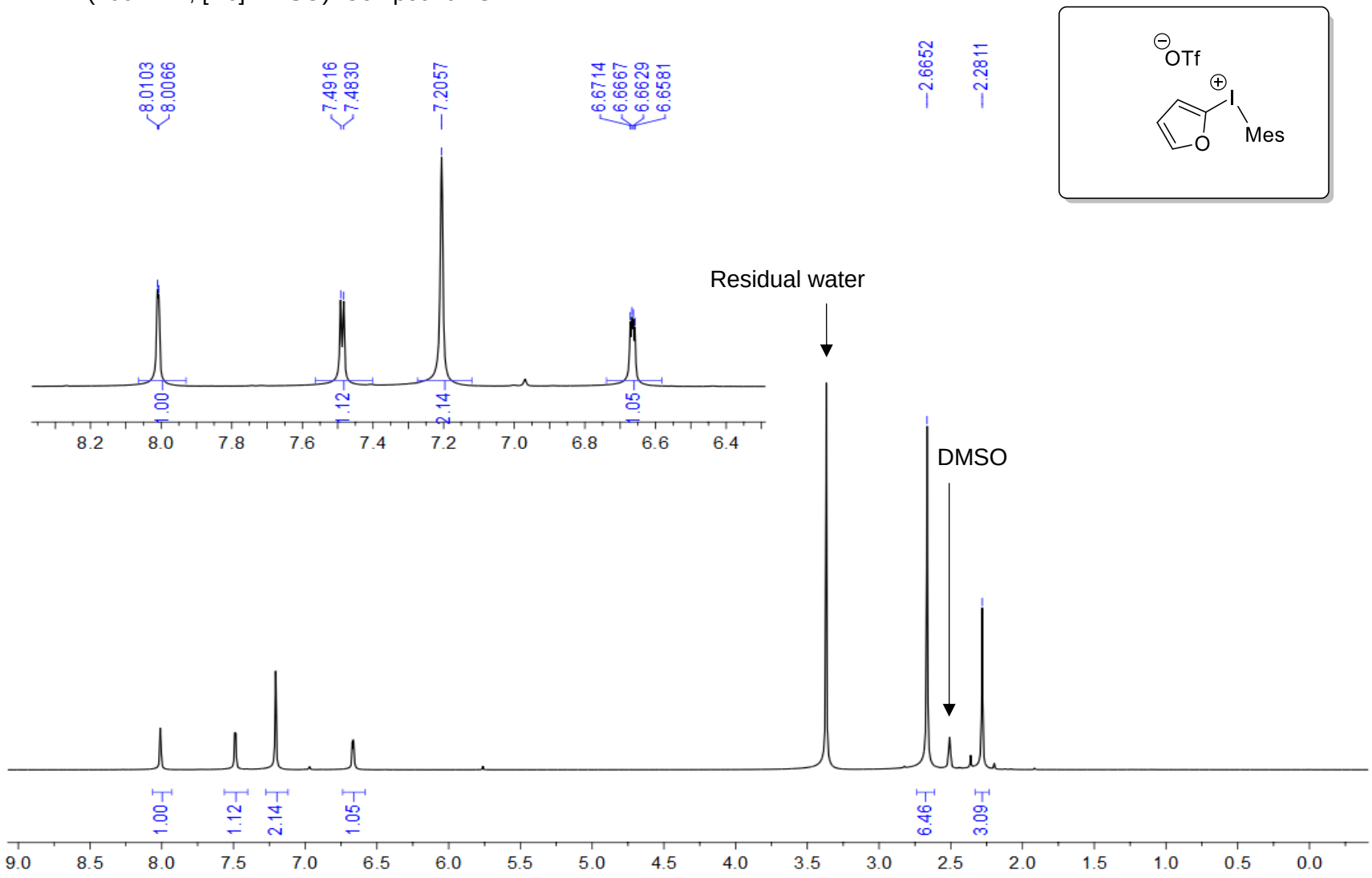
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **2r**



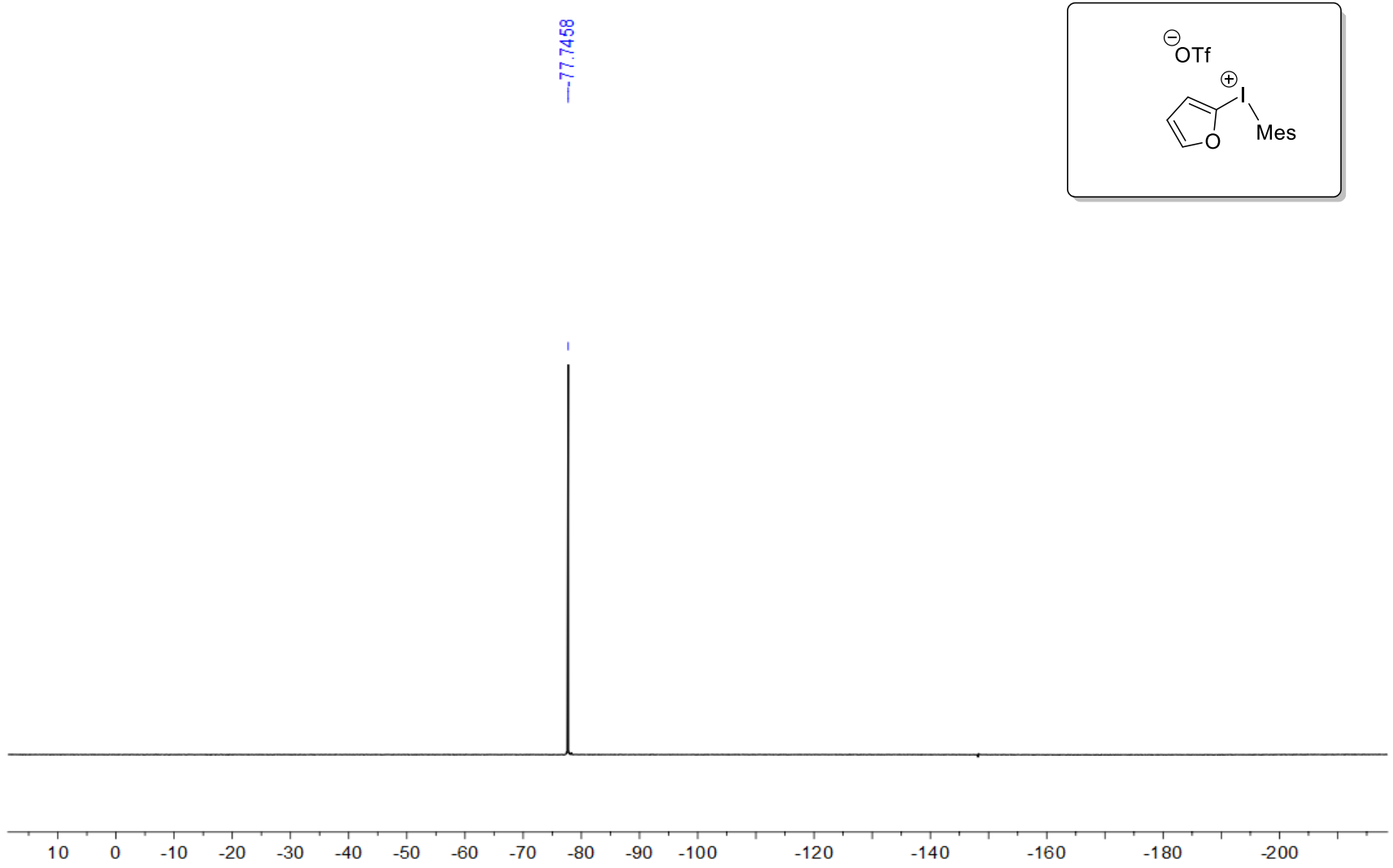
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **2r**



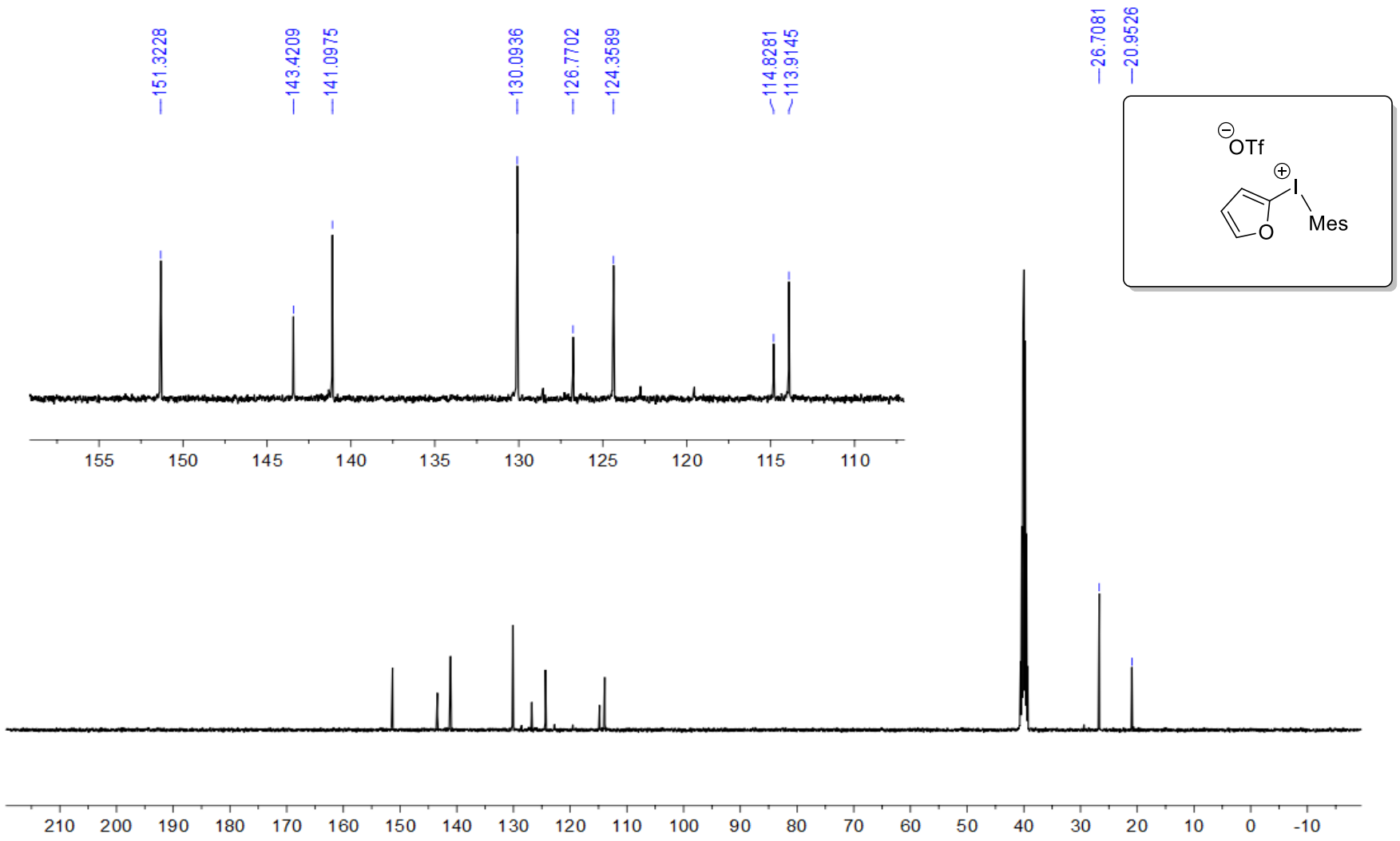
^{1}H NMR (400 MHz, [D6] DMSO): Compound **2s**



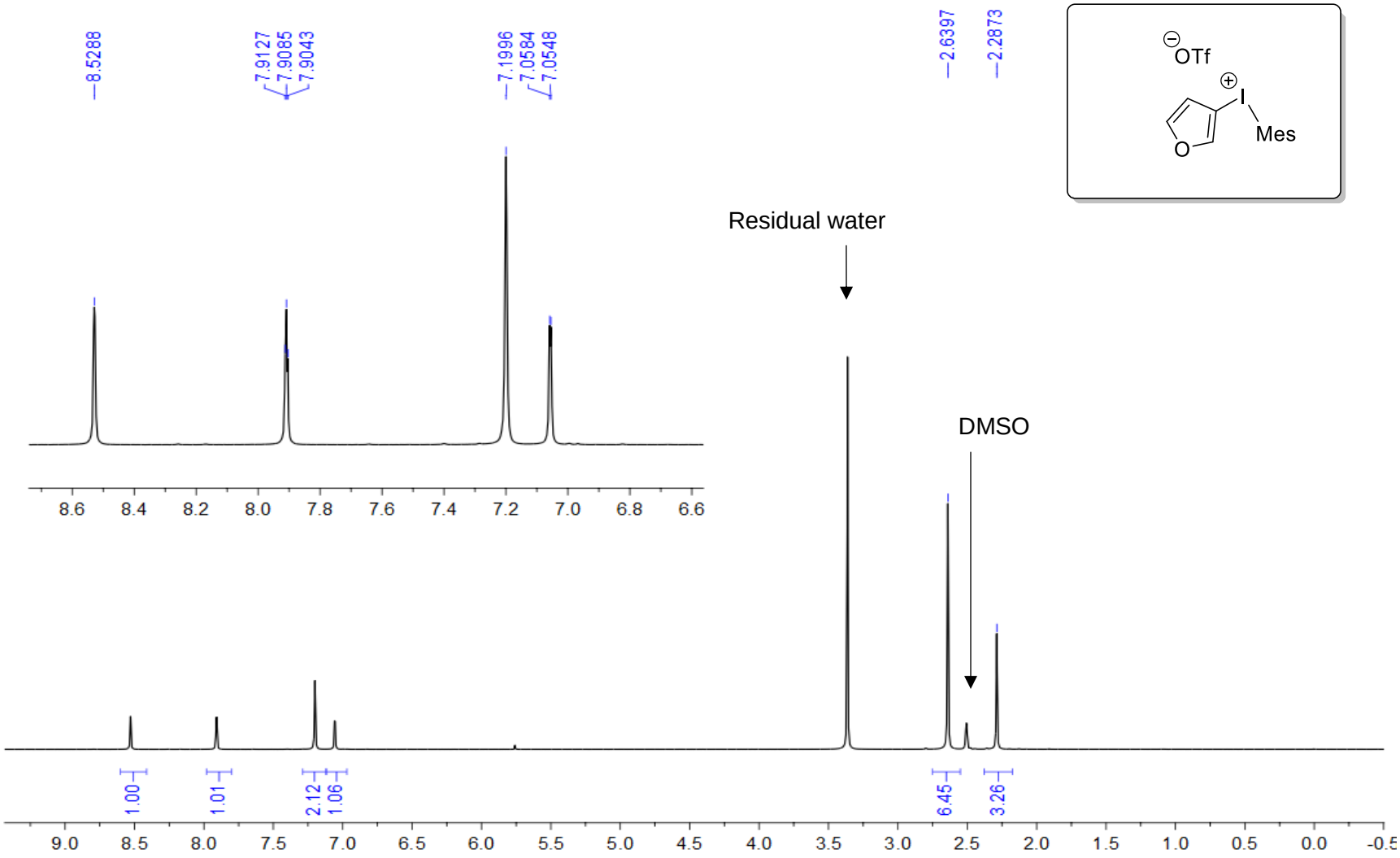
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **2s**



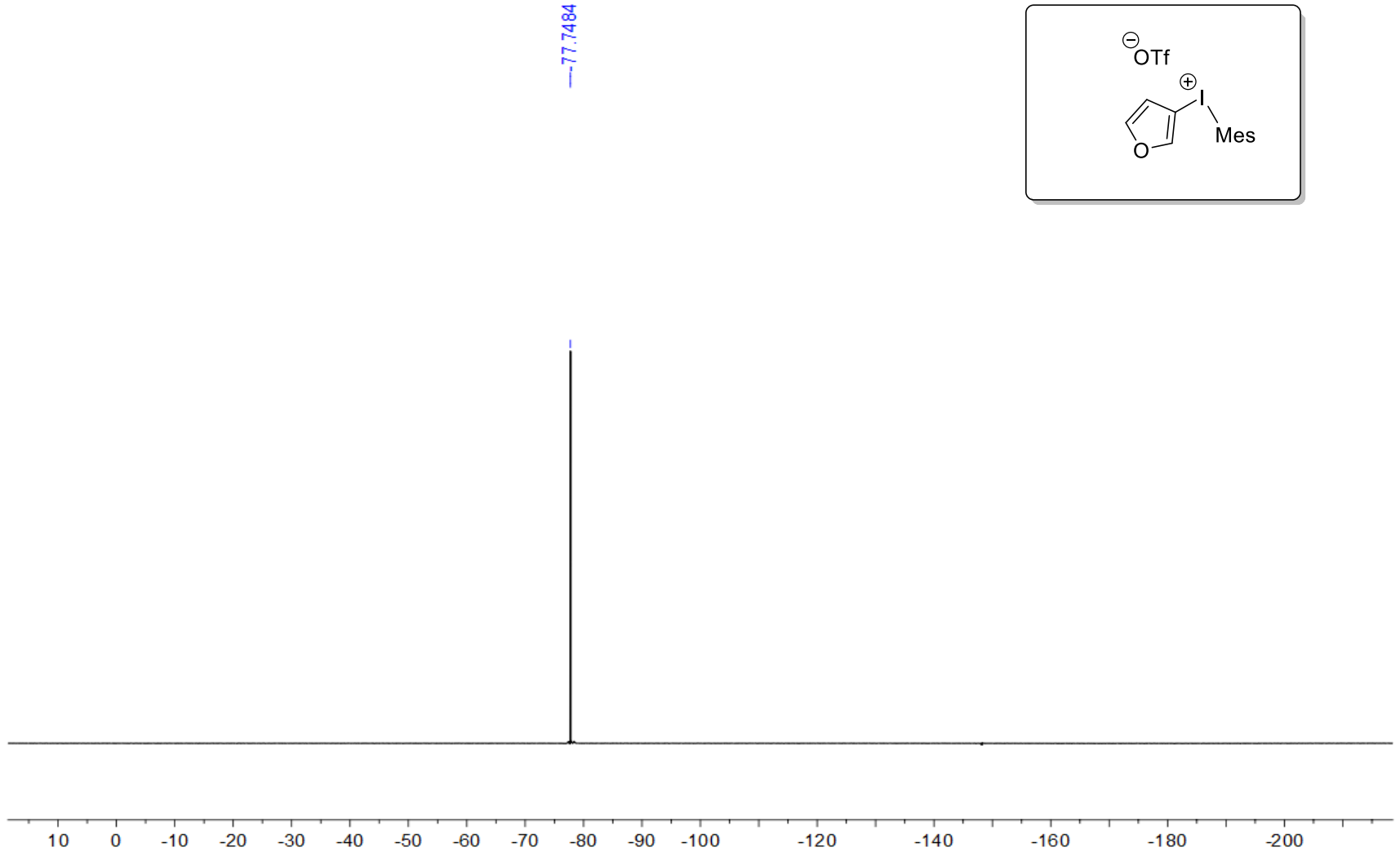
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **2s**



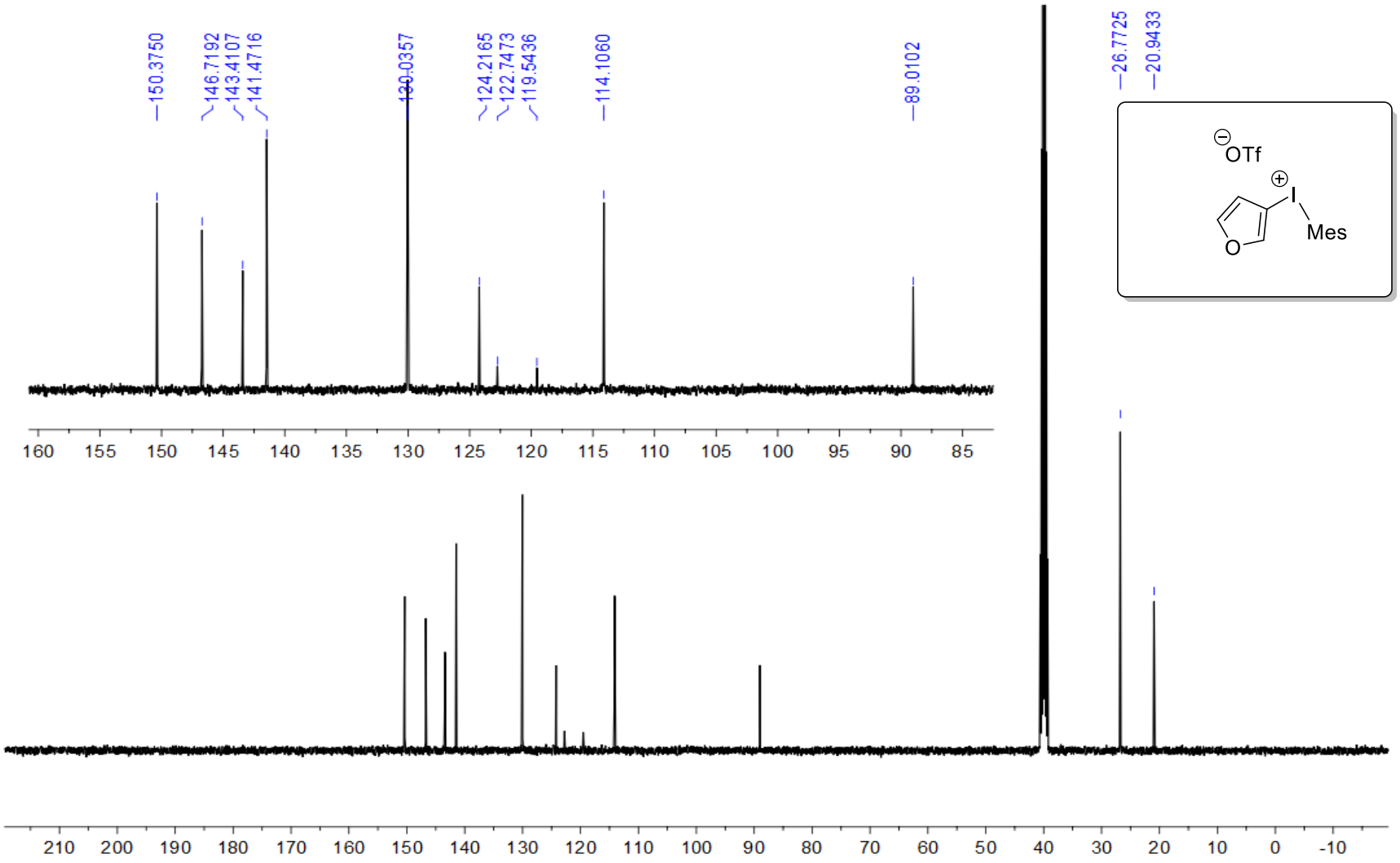
^{1}H NMR (400 MHz, [D6] DMSO): Compound **2t**



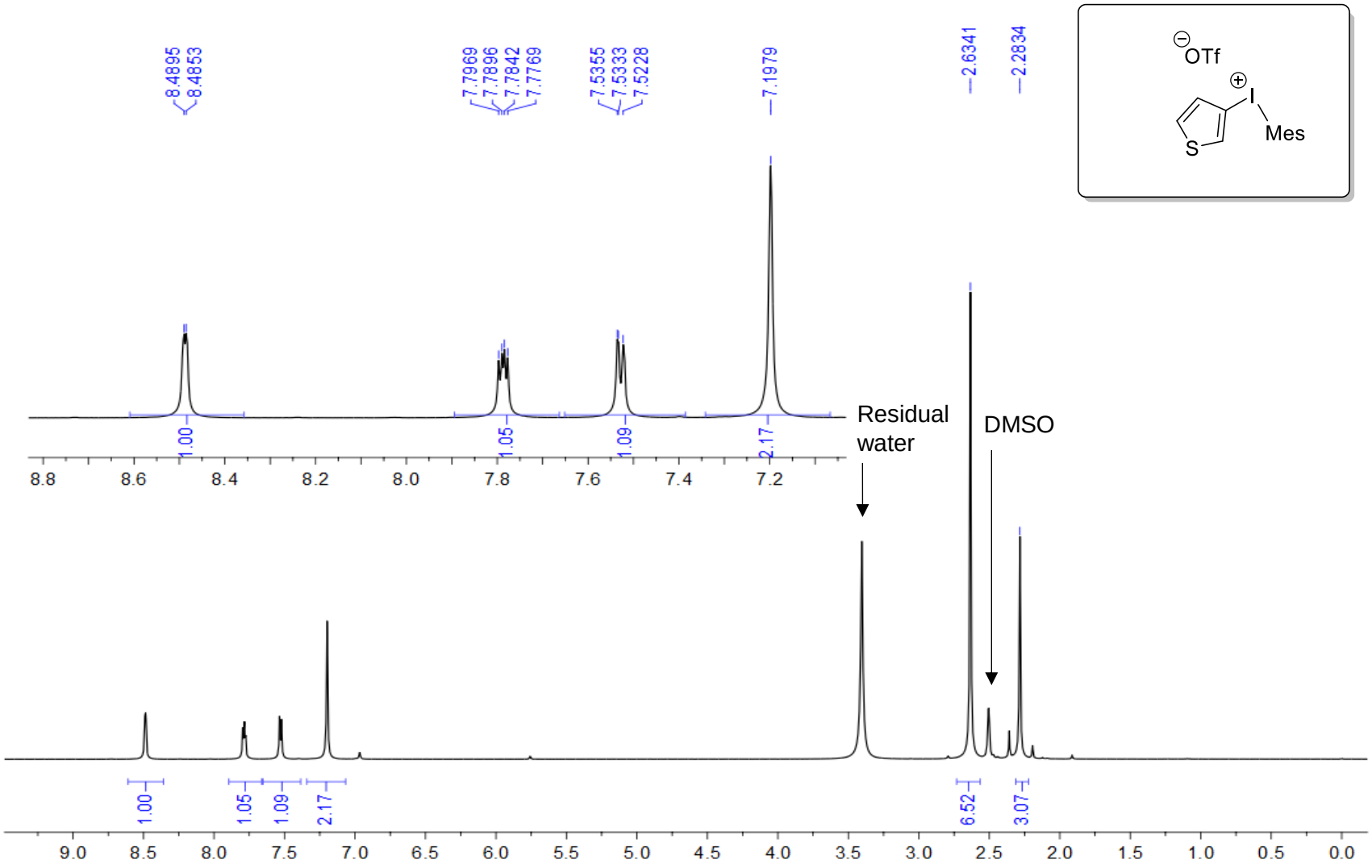
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **2t**



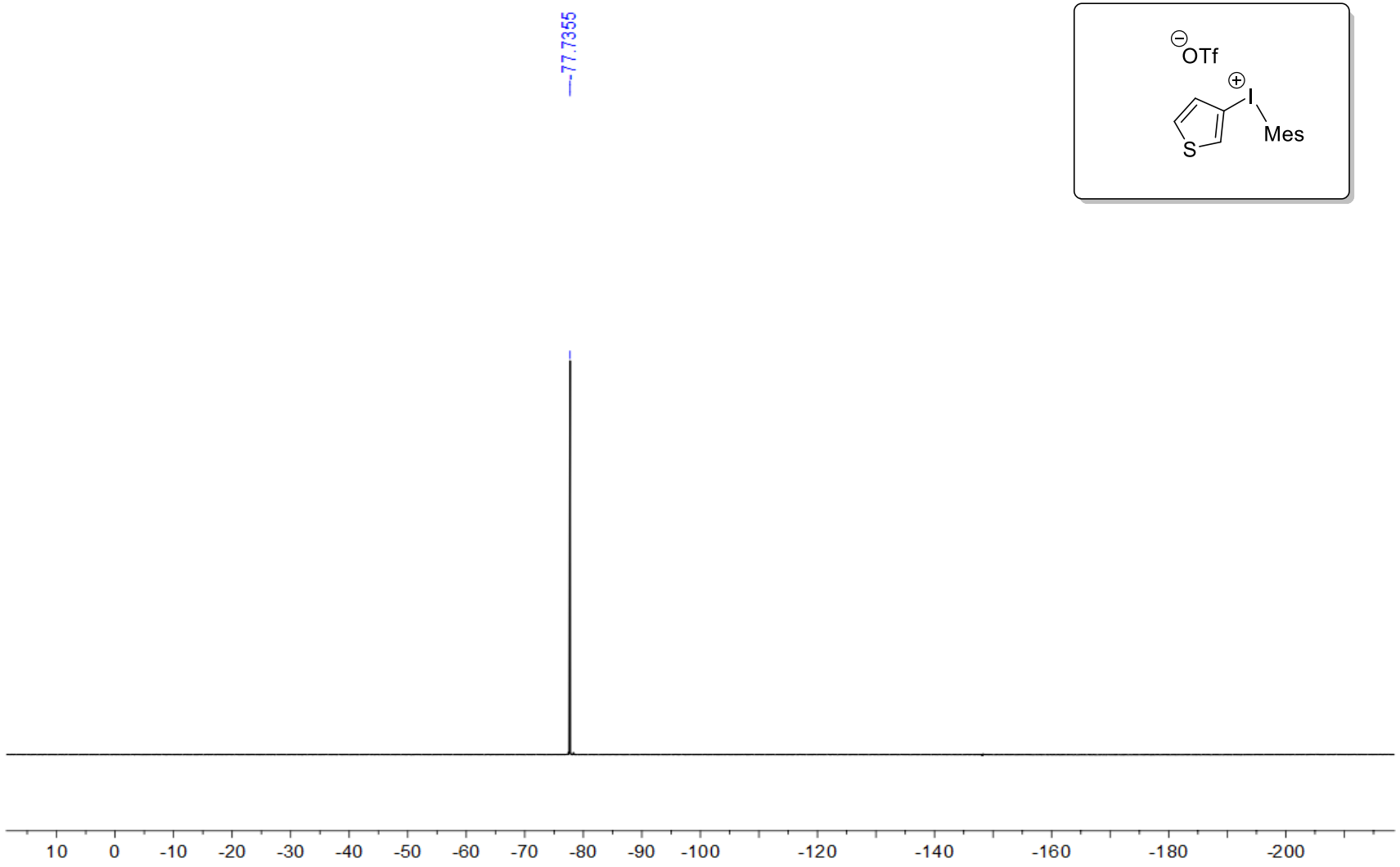
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **2t**



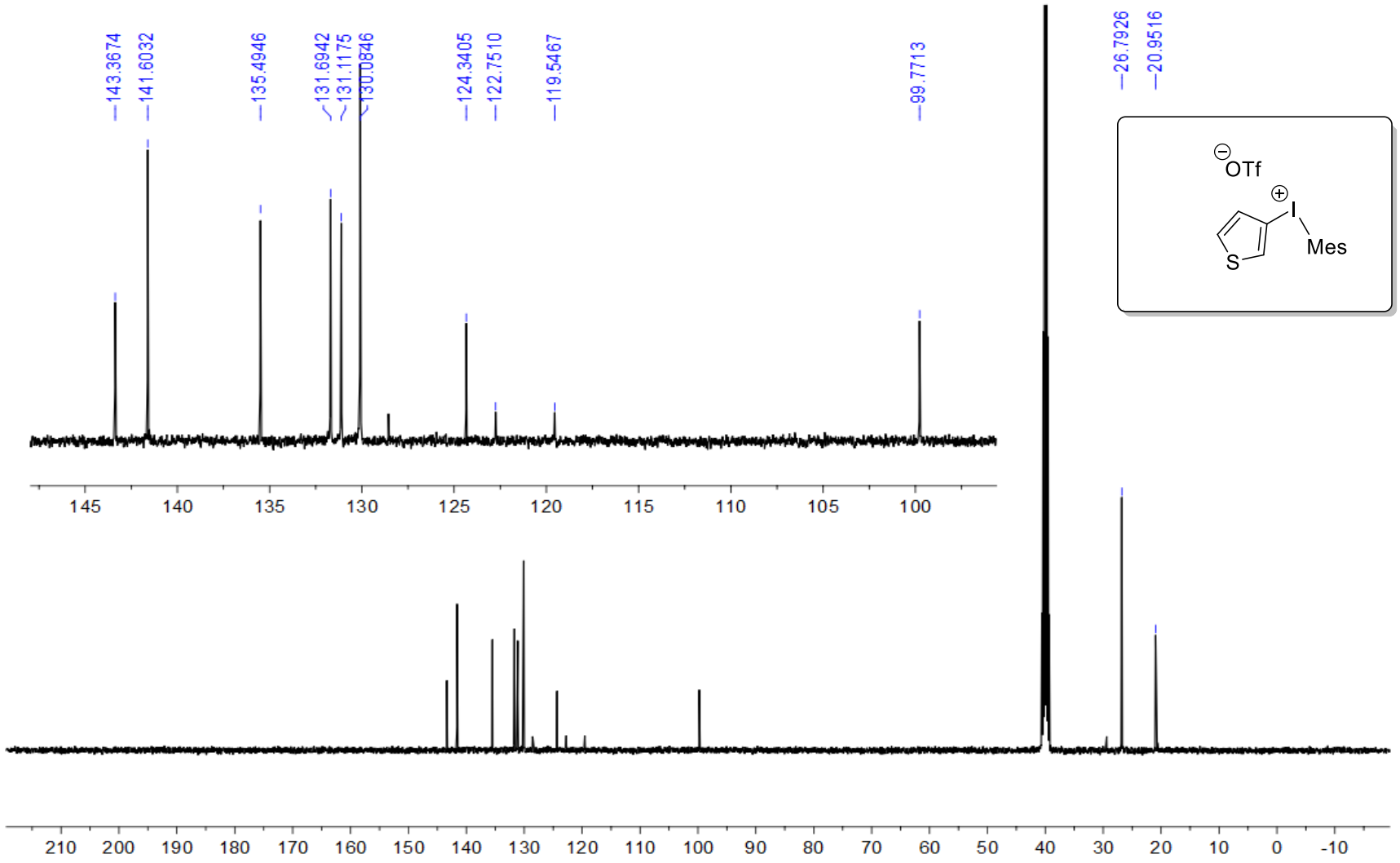
^{1}H NMR (400 MHz, [D6] DMSO): Compound **2u**



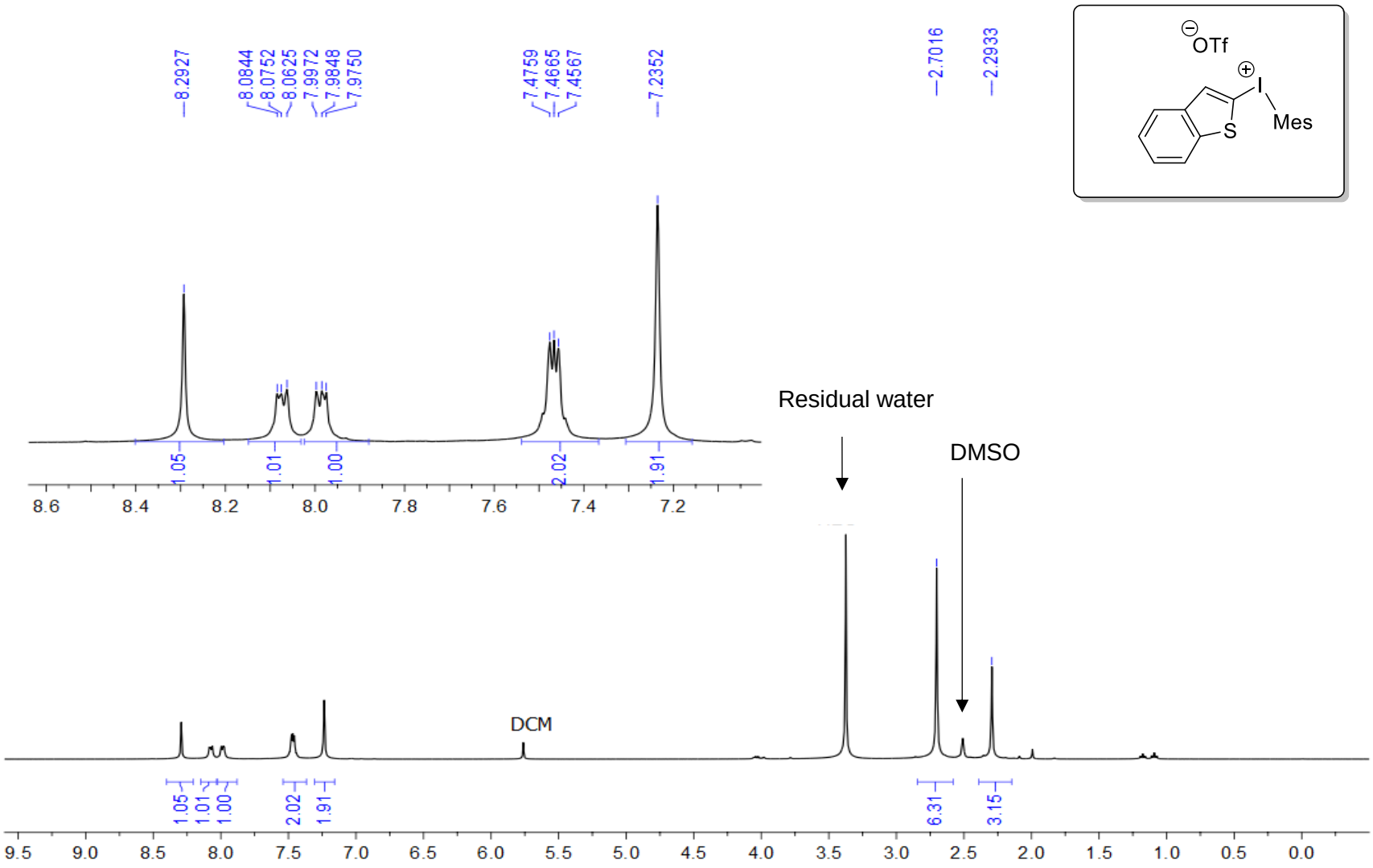
$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **2u**



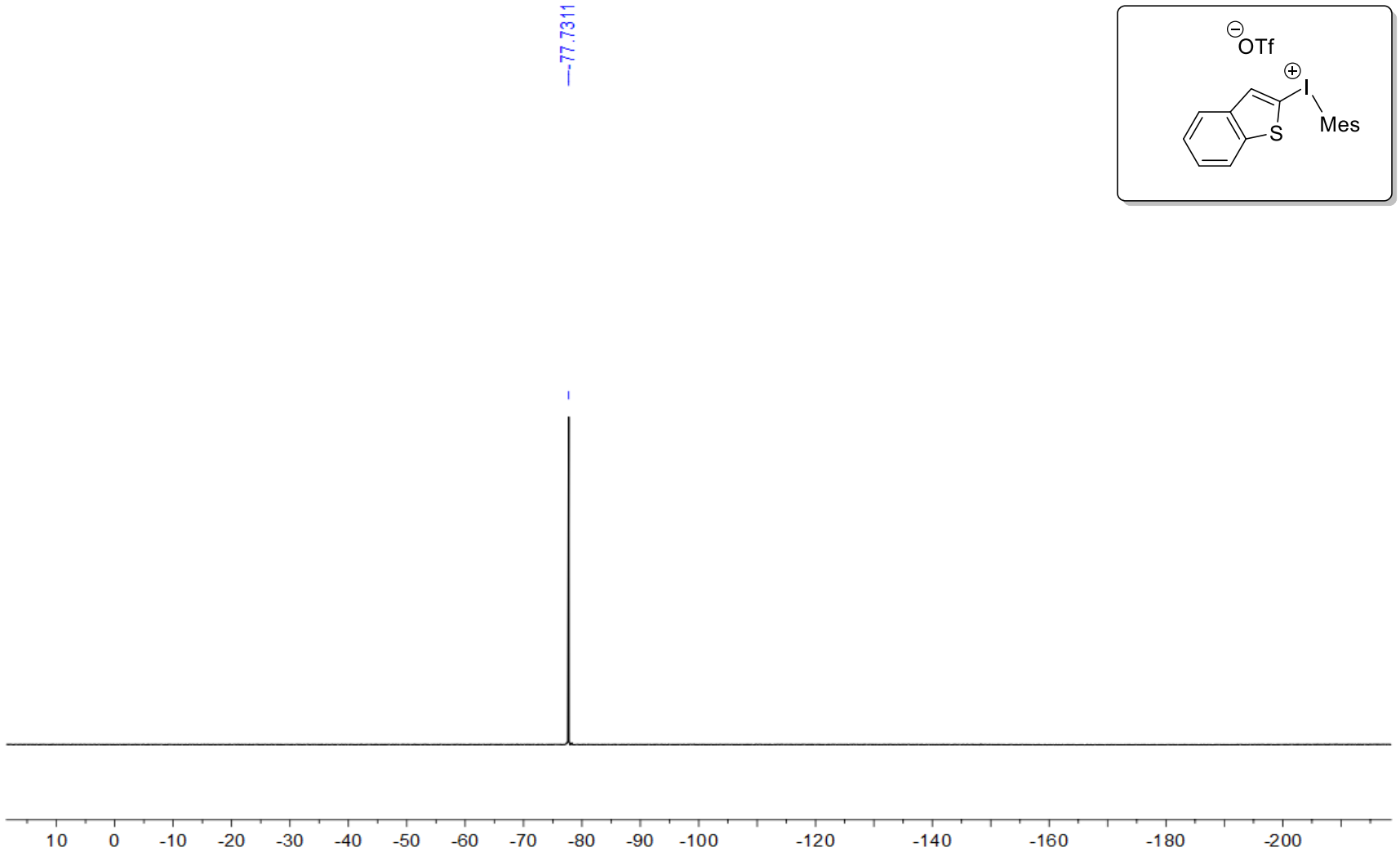
$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **2u**



^{1}H NMR (400 MHz, [D6] DMSO): Compound **2v**



$^{19}F\{^{1}H\}$ NMR (376 MHz, [D6] DMSO): Compound **2v**



$^{13}C\{^{1}H\}$ NMR (101 MHz, [D6] DMSO): Compound **2v**