Iodonium Metathesis Reactions

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Supporting Information

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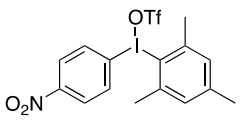
1. Experimental Protocols

Unless otherwise stated, ^{1}H and ^{13}C NMR spectra were recorded on Bruker model AVANCE II+ 300 (300 MHz for ^{1}H and 75.5 MHz for ^{13}C) spectrometer using deuteroacetone (C_3D_6O) as the solvent. Chemical shifts are reported in parts per million (ppm) on the ™ scale and coupling constants, *J*, are in hertz (Hz). Multiplicities are reported as "s" (singlet), "d" (doublet), "t" (triplet), "q" (quartet), "dd" (doublet of doublets), "ddd" (doublet of doublets of doublets), "m" (multiplet), "app" (apparent) and "br" (broad). Low–resolution mass spectra (m/z) were obtained in the electrospray (ESI) mode on a Waters Micromass ZQ mass spectrometer. High–resolution mass spectra (m/z) were recorded in the electrospray (ESI) mode on a Micromass LCT mass spectrometer by the UBC Mass Spectrometry laboratory. Low– and high–resolution mass spectra obtained in electron impact (EI) mode were recorded on MASPEC II System mass spectrometer by the UBC Mass Spectrometry laboratory. Melting points (uncorrected) were measured on a Mel–Temp apparatus. All reagents and solvents were commercial products and used without further purification except *m*CPBA, which was dried *in vacuo* (5g of *m*CPBA was dried for 1h under high-vacuum) and active content was determined by iodometric titration. Flash chromatography was performed on Silicycle 230-400 mesh silica gel. Analytic TLC was carried out with Merck silica gel 60 plates with fluorescent indicator. Spots were visualized with UV light. All reactions were performed under dry argon in flame– or oven–dried flasks equipped with Teflon™ stirbars. All flasks were fitted with rubber septa for the introduction of substrates, reagents and solvents via syringe. Solvents, pure liquid reagents or reagents in solution, and solids were added in one portion, unless otherwise stated.

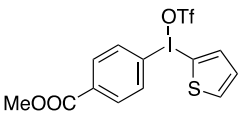
2. Preparation of Iodonium Triflates

The following compounds were prepared as described in the literature and purified by dissolving in the minimal volume of hot MeCN, followed by addition of excess cold Et_2O to induce crystallization: diphenyliodonium triflate,[1] 4-carbomethoxyphenyl-phenyliodonium triflate,[1] 4-nitrophenyl-phenyliodonium triflate,[1] 4-anisyl-4-nitrophenyiodonium triflate,[1] bis(4-anisyl)iodonium triflate.[2] Substantially the same procedures afforded the following previously unreported diaryliodonium triflates:



a. Mesityl-4-nitrophenyliodonium triflate. Solid *m*CPBA (90 % active oxidant, 242 mg, 1.25 mmol, 1.25 equiv) was added in one portion at RT to a CH_2Cl_2 (2 mL) solution of 4-iodonitrobenzene (249 mg, 1.0 mmol), followed by dropwise addition of triflic acid (0.2 mL, 2.0 mmol, 2.0 equiv). The mixture was stirred for 10 min, then it was cooled to 0 °C. A CH_2Cl_2 (1 mL) solution of mesitylene (0.15 mL, 1.1 mmol, 1.1 equiv) was added dropwise over 5 min and the mixture was stirred for 30 min while warming to RT. The solution was then concentrated under reduced pressure and the residue was taken up in Et_2O and stirred for 30 min to allow full precipitation of the product, which was recovered by vacuum filtration (510 mg, 97%). This crude material was recrystallized by dissolving in the minimal volume of hot MeCN, followed by addition of excess cold Et_2O. Mesityl-4-nitrophenyliodonium triflate was thus obtained as a white solid (450 mg, 87%), mp: 199-201°C (dec.).

^{1}H: 8.34 (ABq, 4 H, $\Delta\nu_{AB}$ = 0.02, J_{AB} = 9.2 Hz), 7.34 (s, 2 H), 2.73 (s, 6 H), 2.39 (s, 3 H). **^{13}C**: 150.11, 144.94, 142.82, 135.36, 130.47, 126.52, 121.14, 118.20, 26.23, 20.14. **HRMS** (ESI): calcd for $C_{15}H_{15}NO_2I^+$ ($[M - TfO^-]^+$): 368.0148; found 368.0143.



b. 4-Carbomethoxyphenyl-2-thienyliodonium triflate. Solid *m*-CPBA (70% active oxidant, 670 mg, 2.71 mmol, 1.0 equiv) was added in one portion at RT to a CH_2Cl_2 (15 mL) solution of methyl-4-iodobenzoate (755 mg, 2.82 mmol, 1 equiv) followed by dropwise addition of trifluoromethanesulfonic acid (0.5 mL, 5.65 mmol, 2.0 equiv.). The mixture was stirred for at RT for 5 min and cooled to 0 °C. A solution of thiophene (0.25 mL, 3.12 mmol, 1.1 equiv.) in CH_2Cl_2 (2 mL) was added dropwise over 5 min resulting in a dark green/blue mixture. The reaction was stirred at 0 ^{o}C for 5 min, then at r.t. for 3 h, and concentrated under reduced pressure to give a dark colored solid residue. This material was suspended in 10% acetone/Et_2O (20 mL). The suspended solid was recovered by vacuum filtration and rinsed with Et_2O to obtain crude 4-carbomethoxyphenyl-2-

[1] Bielawski, M.; Zhu, M.; Olofsson, B. *Adv. Synth. Catal.* **2007**, *349*, 2610 – 2618.
[2] Zhu, M.; Jalalian, N.; Olofsson, B. *Synlett*, **2008**, *4*, 592-596.

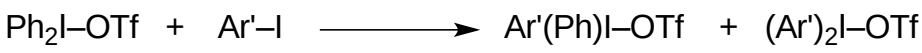
thienyliodonium triflate as a brown solid. The compound was dissolved in acetone (15 mL), suspended particles were removed by gravity filtration, and the filtrate was concentrated under reduced pressure. The solid residue of was recrystallized by dissolving in the minimal volume of hot MeCN, followed by addition of excess cold Et_2O, to afford the title compound (576 mg, 41%) as a light brown, shiny solid, mp: 134-135°C (dec.).

^{1}H: 8.45 (app d, *J* = 8.7 Hz, 2 H), 8.27 (dd, *J* = 3.9, 1.3 Hz, 1 H), 8.12 (app d, *J* = 8.7 Hz, 2 H), 8.06 (dd, *J* = 5.5, 1.3 Hz, 1 H), 7.29 (dd, *J* = 5.4, 3.9 Hz, 1 H), 3.92 (s, 3H). **^{13}C**: 165.8, 143.1, 139.5, 135.8, 134.7, 133.3, 131.0, 122.6, 97.1, 53.0. **HRMS** (ESI): calcd for $C_{15}H_{15}NO_2I^+$ ([M – TfO^-]$^+$): 344.9446; found 344.9449.

3. General Procedure for Melt Metathesis Reactions.

The aryl iodide (0.5 mmol, 5 equiv.) was combined with diphenyliodonium triflate (0.1 mmol) in a 0.2 mL microwave vial, which was then sealed with a crimped cap. The mixture was heated at 120 °C (oil bath temperature) for 24h, then it was cooled to r.t. The crude material was purified by flash chromatography (gradient 10 → 40% acetone/ CH_2Cl_2).

Results of melt metathesis reactions of Ph_2IOTf

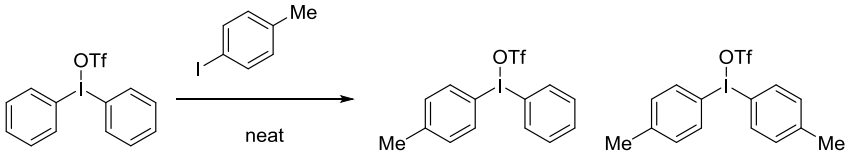


A **B** **C**

a. Ar'–I = 4-Me-C_6H_4: ratio **B** : **C** : **A** = 1.0:2.3:1.5 (60% yield)

b. Ar'–I = 4-MeO-C_6H_4 ratio **B** : **C** : **A** = 3.5:1.3:1.0 (82% yield)

a. Melt metathesis between Ph_2IOTf and 4-iodotoluene



A **B** **C**

4-iodotoluene (109 mg, 0.5 mmol) and Ph_2IOTf (43 mg, 0.1 mmol) combined to afford 60% (27 mg) of a mixture of **A**, **B**, and **C**.

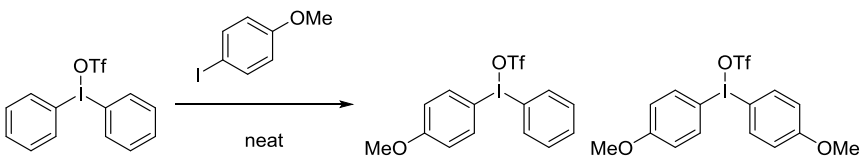
^{1}H: diphenyliodonium triflate (**A**): 8.34 (app dd, *J* = 8.6, 1.0 Hz, 4 H), 7.74 (app tt, *J* = 7.5, 1.7 Hz, 2 H), 7.59 (app t, *J* = 7.9 Hz, 4 H).

4-tolyl-phenyliodonium triflate (**B**): 8.34-8.28 (m, 2 H), 8.24-8.19 (m, 2 H), 7.76-7.70 (m, 1 H), 7.64-7.60 (m, 2 H), 7.43-7.37 (m, 2 H), 2.40 (s, 3 H).

bis-(4-tolyl)iodonium triflate (**C**): 8.18 (app d, *J* = 8.5 Hz, 4 H), 7.39 (app d, *J* = 8.0 Hz, 4 H), 2.40 (s, 6 H).

^{13}C: 144.6, 144.5, 136.51, 136.48, 136.32, 136.29, 133.7, 133.6, 133.5, 133.4, 133.00, 132.95, 124.2, 120.0, 115.3, 111.8, 21.3.

b. Melt metathesis between Ph_2IOTf and 4-iodoanisole (Entry 1b)



A **B** **C**

4-iodoanisole (117 mg, 0.5 mmol) and Ph_2IOTf (43 mg, 0.1 mmol) combined to afford 82% (38 mg) of a mixture of **A**, **B**, and **C**.

^{1}H: diphenyliodonium triflate (**A**): 8.34 (m, 4 H), 7.77-7.70 (m, 2 H), 7.63-7.53 (m, 4 H).

4-anisyl-phenyliodonium triflate (**B**): 8.31-8.24 (m, 4 H), 7.72 (app tt, app *J* = 7.3, 1.5 Hz, 1 H), 7.61-7.53 (m, 2 H), 7.11 (app d, *J* = 9.3Hz, 2 H), 3.87 (s, 3 H).

bis-(4-anisyl)iodonium triflate (**C**): 8.22 (app d, *J* = 9.2 Hz, 4 H), 7.10 (app d, *J* = 9.2 Hz, 4 H), 3.86 (s, 6 H).

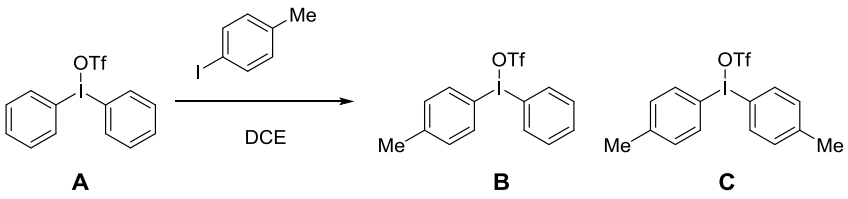
^{13}C: 164.0, 163.8, 138.7, 138.2, 136.5, 136.0, 133.5, 133.3, 133.0, 132.9, 124.2, 120.0, 118.7, 118.6, 115.3, 115.2, 104.3, 103.5, 56.3.

4. General Procedure for Solution Metathesis Reactions

A 15 mL heavy walled pressure tube equipped with a screw-cap seal was charged with the aryl iodide (5 equiv), the iodonium triflate in 0.5 mL 1,2-dichloroethane (0.2M in iodonium triflate). The tube was sealed with a screw cap and immersed in an oil bath maintained at 125 °C. Heating was continued for the specified time, then the mixture was cooled to RT and concentrated under reduced pressure. The crude product was purified by silica gel flash chromatography (gradient 10 → 40% acetone/ CH_2Cl_2).

> **Note**: in the procedures that follow, compounds are numbered as Entry 1a, 2c, 3f, etc., to indicate that, in the main body of the paper, the substance in question is found in Table 1, entry a; or Table 2, entry c; or Table 3, entry f; etc.

a. Solution metathesis between Ph_2IOTf and 4-iodotoluene (Entry 1a)



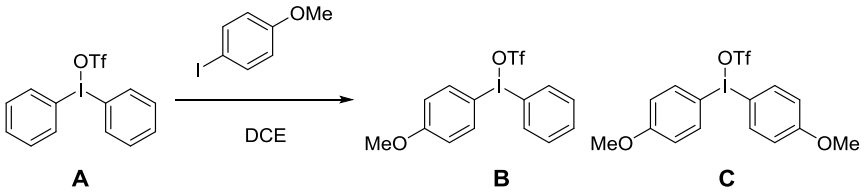
4-iodotoluene (109 mg, 0.5 mmol) and Ph_2IOTf (43 mg, 0.1 mmol) combined and stirred for 24h to afford 48% (22 mg) of a mixture of products.

^{1}H: phenyl-4-tolyliodonium triflate (**B**): 8.31 (app d, *J* = 7.8 Hz, 2 H), 8.24-8.19 (m, 2 H), 7.74 (app t, *J* = 7.5 Hz, 1 H), 7.59 (app t, *J* = 7.9 Hz, 2 H), 7.44-7.40 (m, 2 H), 2.41 (s, 3 H).

bis-(4-tolyl)iodonium triflate (**C**): 8.18 (app d, *J* = 8.4 Hz, 4 H), 7.40 (app d, *J* = 8.3 Hz, 4 H), 2.41 (s, 6 H).

^{13}C: 144.51, 144.47, 136.5, 136.31, 136.28, 133.7, 133.6, 133.4, 133.0, 111.88, 111.85, 21.3.

b. Solution metathesis between Ph_2IOTf and 4-iodoanisole (Entry 1b)



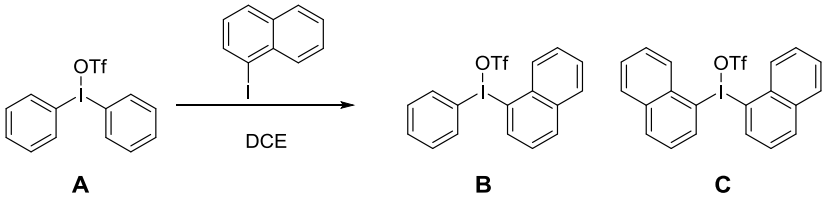
4-iodoanisole (117 mg, 0.5 mmol) and Ph_2IOTf (43 mg, 0.1 mmol) combined and stirred for 25h to afford 80% (37 mg) of a mixture of products.

^{1}H: 4-anisyl-phenyliodonium triflate (**B**): 8.32-8.24 (m, 4 H), 7.74 (app t, *J* = 7.5 Hz, 1 H), 7.59 (app t, *J* = 7.9 Hz, 2 H), 7.16-7.11 (m, 2 H), 3.88 (s, 3 H).

bis-(4-anisyl)iodonium triflate (**C**): 8.23 (app d, *J* = 9.2 Hz, 4 H), 7.11 (app d, *J* = 9.2 Hz, 4 H), 3.88 (s, 6 H).

^{13}C: 163.9, 163.8, 138.7, 138.2, 136.5, 136.0, 133.5, 133.2, 133.0, 132.9, 124.2, 119.9, 118.7, 118.5, 115.9, 104.4, 103.6, 56.2.

c. Solution metathesis between Ph_2IOTf and 1-iodonaphthalene (Entry 1c)



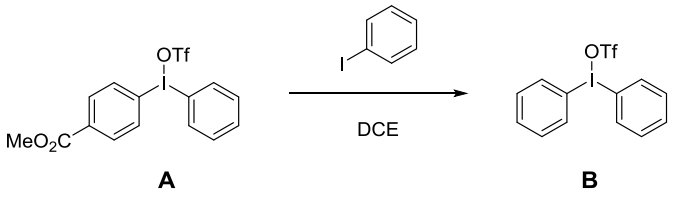
1-iodonaphthalene (73 µL, 0.5 mmol) and Ph_2IOTf (43 mg, 0.1 mmol) combined and stirred for 26h to afford 14% (7 mg) of a mixture of products.

^{1}H: 1-naphthyl-phenyliodonium triflate (**B**): 8.88 (d, *J* = 7.5 Hz, 1 H), 8.41-8.31 (m, 4 H), 8.10 (d, *J* = 8.1 Hz, 1 H), 7.85 (app t, *J* = 8.2 Hz, 1 H), 7.80-7.57 (m, 3 H), 7.53 (app t, *J* = 7.6 Hz, 2 H).

bis-(1-naphthyl)iodonium triflate (**C**): 8.93 (d, *J* = 7.5 Hz, 2 H), 8.53 (d, *J* = 8.5 Hz, 2 H), 8.28 (d, *J* = 8.2 Hz, 2 H), 8.04 (d, *J* = 8.3 Hz, 2 H), 7.85 (app t, *J* = 8.2 Hz, 2 H), 7.75-7.58 (m, 4 H).

^{13}C: 139.3, 139.0, 138.0, 136.5, 136.0, 135.9, 135.3, 135.0, 133.5, 133.3, 133.1, 133.0, 132.5, 131.0, 130.9, 130.61, 130.55, 129.7, 129.6, 129.15, 129.07, 128.6, 128.5, 124.2, 118.2, 115.2.

d. Solution metathesis between 4-carbomethoxyphenyl-phenyliodonium triflate and iodobenzene (Entry 2a)

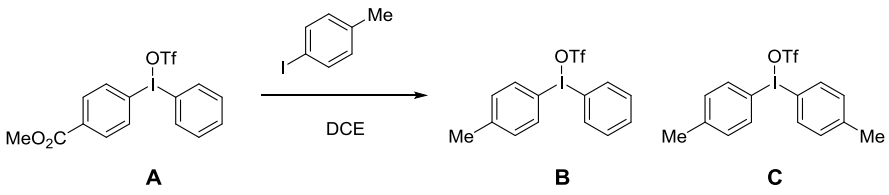


Iodobenzene (56 µL, 0.5 mmol) and 4-carbomethoxyphenyl-phenyliodonium triflate (49 mg, 0.1 mmol) combined and stirred for 15h to afford 51% (22 mg) of a mixture of products.

^{1}H: 4-carbomethoxyphenyl -phenyliodonium triflate (**A**): 8.46 (app d, *J* = 8.7 Hz, 2 H), 8.11 (app d, *J* = 8.7 Hz, 2 H), 3.91 (s, 3 H).

diphenyliodonium triflate (**B**): 8.38-8.31 (m, 4 H), 7.75 (tt, *J* = 7.5, 1.1 Hz, 2 H), 7.60 (app t, *J* = 7.9 Hz, 4 H). **^{13}C:** 136.5, 133.5, 133.0, 115.3.

e. Solution metathesis between 4-carbomethoxyphenyl-phenyliodonium triflate and 4-iodotoluene (Entry 2b)



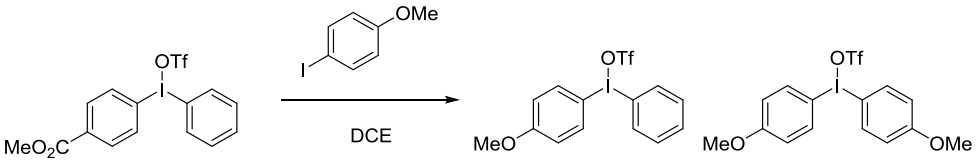
4-iodotoluene (109 mg, 0.5 mmol) and 4-carbomethoxyphenyl-phenyliodonium triflate (49 mg, 0.1 mmol) combined and stirred for 15h to afford 59% (27 mg) of a mixture of products.

^{1}H: phenyl-4-tolyliodonium triflate (**B**): 8.31 (app dd, *J* = 8.4, 1.0 Hz, 2 H), 8.24-8.18 (m, 2 H), 7.74 (app tt, *J* = 7.4, 1.1 Hz, 1 H), 7.59 (app t, *J* = 7.9 Hz, 2 H), 7.43-7.38 (m, 2 H), 2.40 (s, 3 H).

bis-(4-tolyl)iodonium triflate (**C**): 8.18 (app d, *J* = 8.5 Hz, 4 H), 7.39 (app d, *J* = 8.5 Hz, 4 H), 2.40 (s, 6 H).

^{13}C: 144.6, 144.5, 136.5, 136.3, 133.71, 133.65, 133.4, 133.0, 128.5, 124.2, 120.0, 115.4, 111.7, 111.6, 21.3.

f. Solution metathesis between 4-carbomethoxyphenyl-phenyliodonium triflate and 4-iodoanisole (Entry 2c)



A **B** **C**

4-iodoanisole (117 mg, 0.5 mmol) and 4-carbomethoxyphenyl-phenyliodonium triflate (49 mg, 0.1 mmol) combined and stirred for 15h to afford 80% (38 mg) of a mixture of products.

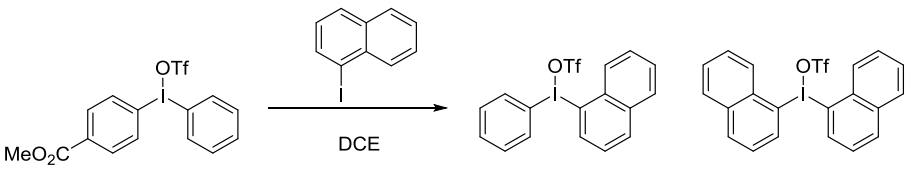
^{1}H: 4-carbomethoxyphenyl-phenyliodonium triflate (**A**): 8.40 (app d, *J* = 8.7 Hz, 2 H), 8.33-8.26 (m, 2H), 7.76-7.71 (m, 1 H), 7.63-7.58 (m, 2 H), 8.09 (app d, *J* = 8.7 Hz, 2 H), 3.91 (s, 3 H).

4-anisyl-phenyliodonium triflate (**B**): 8.34-8.22 (m, 4 H), 7.72 (tt, *J* = 7.4, 1.1 Hz, 1 H), 7.58 (app t, *J* = 7.9 Hz, 2 H), 7.16-7.10 (m, 2 H), 3.88 (s, 3 H).

bis-(4-anisyl)iodonium triflate (**C**): 8.22 (app d, *J* = 9.2 Hz, 4 H), 7.10 (app d, *J* = 9.3 Hz, 4 H), 3.87 (s, 6 H).

^{13}C: 165.9, 164.0, 163.8, 134.0, 138.7, 138.2, 136.1, 136.0, 133.3, 133.1, 132.9, 124.2, 120.3, 119.9, 118.8, 118.7, 118.6, 115.8, 104.3, 103.5, 56.3, 53.0.

g. Solution metathesis between 4-carbomethoxyphenyl-phenyliodonium triflate and 1-iodonaphthalene (Entry 2d)



A **B** **C**

1-iodonaphthalene (73 µL, 0.5 mmol) and 4-carbomethoxyphenyl-phenyliodonium triflate (49 mg, 0.1 mmol) combined and stirred for 24h to afford 35% (38 mg) of a mixture of products.

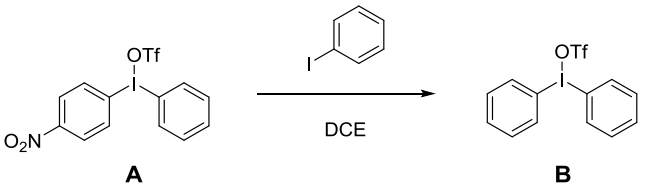
^{1}H: 4-carbomethoxyphenyl-phenyliodonium triflate (**A**): 8.44 (app d, *J* = 8.7 Hz, 2 H), 8.41-8.35 (m, 2 H), 8.05-7.99 (m, 2 H), 7.74-7.67 (m, 1 H), 7.51 (app t, *J* = 7.9 Hz, 2 H).

1-naphthyl-phenyliodonium triflate (**B**): 8.88 (dd, *J* = 7.6, 0.8 Hz, 1 H), 8.41-8.30 (m, 4 H), 8.09 (d, *J* = 8.1 Hz, 1 H), 7.85 (ddd, *J* = 8.2 7.2 1.1 Hz, 1 H), 7.78-7.57 (m, 3 H), 7.51 (app t, *J* = 7.9 Hz, 2 H).

bis-(1-naphthyl)iodonium triflate (**C**): 8.93 (dd, *J* = 7.5, 0.8 Hz, 2 H), 8.53 (d, *J* = 8.5 Hz, 2 H), 8.26 (d, *J* = 8.2 Hz, 2 H), 8.02 (app d, *J* = 7.8 Hz, 2 H), 7.85 (ddd, *J* = 8.2, 7.2, 1.1 Hz, 2 H), 7.75-7.58 (m, 4 H).

^{13}C: 165.8, 139.3, 139.0, 136.0, 135.9, 135.3, 135.0, 133.3, 133.0, 132.4, 131.0, 130.9, 130.6, 130.5, 129.58, 129.56, 129.1, 129.0, 128.6, 128.5, 124.3, 120.0, 118.0, 115.0.

h. Solution metathesis between 4-nitrophenyl-phenyliodonium triflate and iodobenzene (Entry 3a)



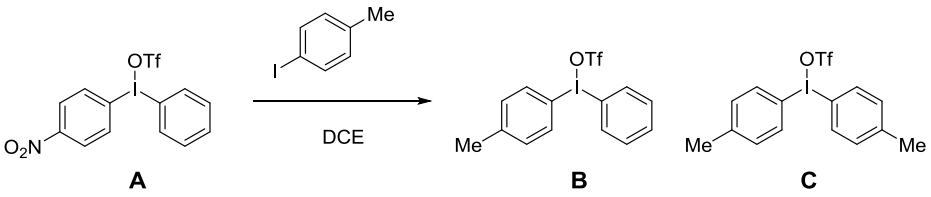
Iodobenzene (56 µL, 0.5 mmol) and 4-nitrophenyl-phenyliodonium triflate (48 mg, 0.1 mmol) combined and stirred for 12h to afford 65% (28 mg) of a mixture of products.

^{1}H: 4-nitrophenyl-phenyliodonium triflate (**A**): 8.61 (app d, *J* = 9.1 Hz, 2 H), 8.38-8.33 (m, 4 H), 7.82-7.72 (m, 1 H), 7.67-7.57 (m, 2 H).

diphenyliodonium triflate (**B**): 8.34 (app dd, *J* = 8.3, 1.1 Hz, 4 H), 7.75 (tt, *J* = 7.4, 1.0 Hz, 2 H), 7.59 (app t, *J* = 7.9 Hz, 4 H).

^{13}C: 137.8, 136.9, 136.1, 133.8, 133.3, 133.2, 128.5, 127.2, 124.2, 119.9, 115.9, 115.3.

i. Solution metathesis between 4-nitrophenyl-phenyliodonium triflate and 4-iodotoluene (Entry 3b)



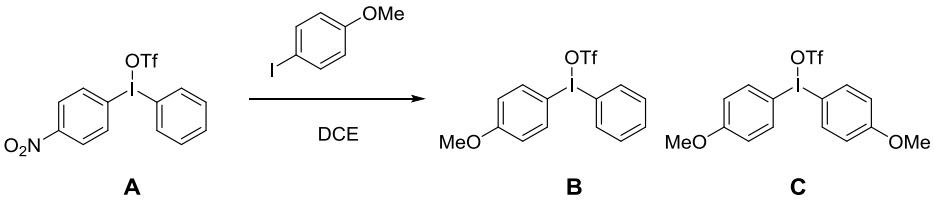
4-iodotoluene (109 mg, 0.5 mmol) and 4-nitrophenyl-phenyliodonium triflate (48 mg, 0.1 mmol) combined and stirred for 12h to afford 73% (33 mg) of a mixture of products.

^{1}H: phenyl-4-tolyliodonium triflate (**B**): 8.34-8.27 (m, 2 H), 8.24-8.18 (m, 2 H), 7.73 (tt, *J* = 7.5, 1.0 Hz, 1 H), 7.58 (app t, *J* = 7.9 Hz, 2 H), 7.43-7.37 (m, 2 H), 2.40 (s, 3 H).

bis-(4-tolyl)iodonium triflate (**C**): 8.18 (app d, *J* = 8.5 Hz, 4 H), 7.39 (app d, *J* = 8.3 Hz, 4 H), 2.40 (s, 6 H).

^{13}C: 144.6, 144.5, 136.5, 136.32, 136.29, 133.7, 133.6, 133.4, 133.0, 124.2, 112.0, 115.4, 111.7, 111.5, 21.6.

j. Solution metathesis between 4-nitrophenyl-phenyliodonium triflate and 4-iodoanisole (Entry 3c)



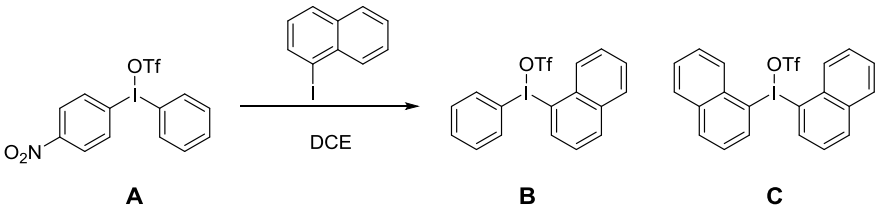
4-iodoanisole (117 mg) and 4-nitrophenyl-phenyliodonium triflate (48 mg, 0.1 mmol) combined and stirred for 12h to afford 76% (36 mg) of a mixture of products.

^{1}H: 4-anisyl-phenyliodonium triflate (**B**): 8.31-8.23 (m, 4 H), 7.72 (tt, *J* = 7.4, 1.1 Hz, 1 H), 7.76 (app t, *J* = 7.9 Hz, 2 H), 7.12 (app d, *J* = 9.2 Hz, 2 H), 3.88 (s, 3 H).

bis-(4-anisyl)iodonium triflate (**C**): 8.22 (app d, *J* = 9.2 Hz, 4 H), 7.09 (app d, *J* = 9.2 Hz, 4 H), 3.87 (s, 6 H).

^{13}C: 164.0, 163.8, 138.7, 138.2, 136.0, 133.3, 132.9, 124.2, 112.0, 118.7, 118.6, 115.8, 104.3, 103.5, 56.3.

k. Solution metathesis between 4-nitrophenyl-phenyliodonium triflate and 1-iodonaphthalene (Entry 3d)



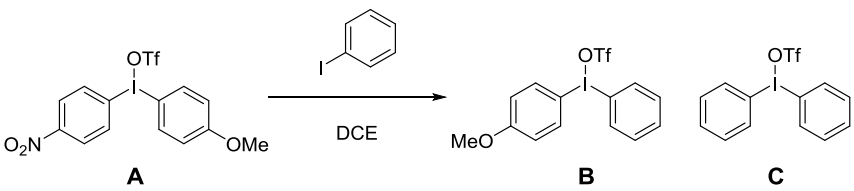
1-iodonaphthalene (73 µL, 0.5 mmol) and 4-nitrophenyl-phenyl-iodonium triflate (48 mg, 0.1 mmol) combined and stirred for. 12h to afford 59% (29 mg) of a mixture of products

^{1}H: 1-naphthyl-phenyliodonium triflate (**B**): 8.88 (dd, *J* = 7.5, 1.0 Hz, 1 H), 8.40-8.30 (m, 4 H), 8.08 (app dd, *J* = 8.2, 1.1 Hz, 1 H), 7.84 (ddd, *J* = 8.3, 7.0, 1.3 Hz, 1 H), 7.78-7.57 (m, 3 H), 7.51 (app t, *J* = 7.8 Hz, 2 H).

bis-(1-naphthyl)iodonium triflate (**C**): 8.94 (dd, *J* = 7.6, 1.0 Hz, 2 H), 8.52 (app dd, *J* = 8.5, 0.6 Hz, 2 H), 8.25 (d, *J* = 8.2, 2 H), 8.01 (d, *J* = 8.2 Hz, 2 H), 7.84 (ddd, *J* = 8.3, 7.0, 1.3 Hz, 2 H), 7.75-7.58 (m, 4 H).

^{13}C: 139.3, 139.0, 137.2, 136.5, 136.0, 135.9, 135.3, 135.0, 133.5, 133.3, 133.0, 132.9, 132.4, 131.0, 130.9, 130.6, 130.5, 129.6, 129.5, 129.1, 129.0, 128.6. 128.5, 124.3, 120.0, 118.0, 115.04, 115.00.

l. Solution metathesis between 4-anisyl-4-nitrophenyliodonium triflate and iodobenzene (Entry 3e)



Iodobenzene (56 µL, 0.5 mmol) and 4-anisyl-4-nitrophenyliodonium triflate (50 mg, 0.1 mmol) combined and stirred for 12h to afford 60% (27

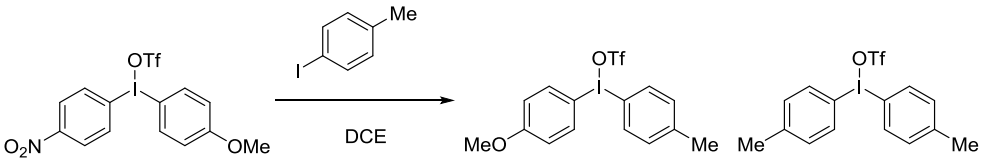
mg) of a mixture of products.

^{1}H: 4-anisyl-phenyliodonium triflate (**A**): 8.29 (app dd, *J* = 8.4, 1.0 Hz, 2 H), 8.27 (app d, *J* = 9.2 Hz, 2 H), 7.72 (tt, *J* = 7.1, 1.2 Hz, 1 H), 7.58 (app t, *J* = 7.4 Hz, 2 H), 7.12 (app d, *J* = 9.3 Hz, 2 H), 3.88 (s, 3 H).

diphenyliodonium triflate (**C**): 8.34 (app dd, *J* = 8.5, 1.0 Hz, 4 H), 7.75 (tt, *J* = 7.3, 1.1 Hz, 2 H), 7.60 (app t, *J* = 7.8 Hz, 4 H).

^{13}C: 164.0, 139.2, 138.7, 138.2, 137.2, 136.5, 136.0, 133.5, 133.3, 133.0, 132.9, 128.5, 127.1, 124.2, 120.0, 118.9, 118.7, 118.6, 115.9, 115.3, 104.3, 103.5, 56.3.

m. Solution metathesis between 4-anisyl-4-nitrophenyliodonium triflate and 4-iodotoluene (Entry 3f)



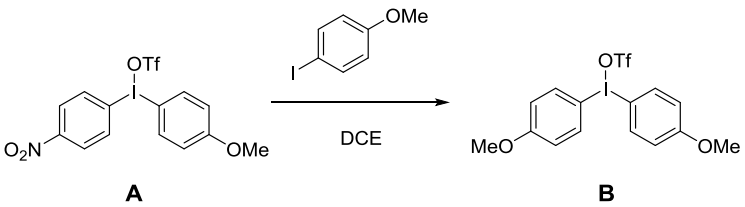
4-iodotoluene (109 mg, 0.5 mmol) and 4-anisyl-4-nitrophenyliodonium triflate (50 mg, 0.1 mmol) combined and stirred for 12h to afford 79% (37 mg) of a mixture of products.

^{1}H: 4-anisyl-4-tolyliodonium triflate (**B**): 8.24(app d, *J* = 9.2 Hz, 2 H), 8.16 (app d, *J* = 8.5 Hz, 2 H), 7.38 (app d, *J* = 8.7 Hz, 2 H), 7.11 (app d, *J* = 9.2 Hz, 2 H), 3.87 (s, 3 H).

bis-(4-tolyl)iodonium triflate (**C**): 8.18 (app d, *J* = 8.4 Hz, 4 H), 7.39 (app d, *J* = 8.6 Hz, 4 H), 3.87 (s, 6 H).

^{13}C: 163.9, 144.5, 144.4, 138.5, 138.2, 136.3, 136.0, 133.64, 133.57, 124.2, 120.0, 118.64, 118.56, 112.2, 111.8, 103.8, 56.3, 21.3.

n. Solution metathesis between 4-anisyl-4-nitrophenyliodonium triflate and 4-iodoanisole (Entry 3g)



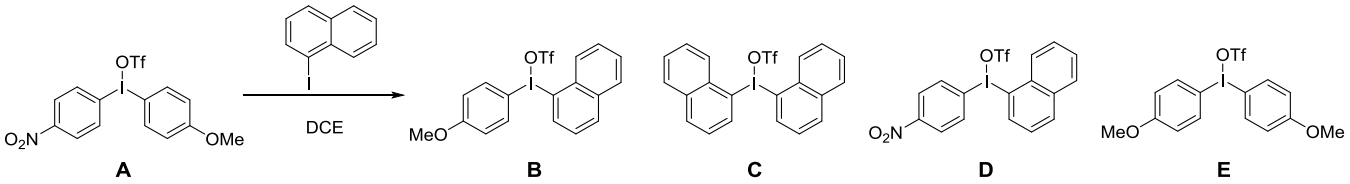
4-iodoanisole (117 mg, 0.5 mmol) and 4-anisyl-4-nitrophenyliodonium triflate (50 mg, 0.1 mmol) combined and stirred for 12h to afford 89% (44 mg) of a mixture of products.

^{1}H: 4-anisyl-4-nitrophenyliodonium triflate (**A**): 8.55 (app d, *J* = 9.2 Hz, 2 H), 8.33 (app dd, *J* = 9.2, 1.5 Hz, 4 H), 7.17-7.08 (m, 2 H), 3.88 (s, 3 H).

bis-(4-anisyl)iodonium triflate (**B**): 8.22 (app d, *J* = 9.2 Hz, 4 H), 7.09 (app d, *J* = 9.2 Hz, 4 H), 3.86 (s, 6 H).

^{13}C: 164.2, 163.8, 150.9, 139.1, 138.61, 138.58, 137.83, 137.79, 137.2, 130.3, 128.4, 127.1, 124.2, 121.8, 119.9, 119.0, 118.9, 118.13, 118.07, 115.7, 104.0, 56.3, 55.5.

o. Solution metathesis between 4-anisyl-4-nitrophenyliodonium triflate and 1-iodonaphthalene (Entry 3h)



1-iodonaphthalene (73 µL, 0.5 mmol) and 4-anisyl-4-nitrophenyliodonium triflate (50 mg, 0.1 mmol) combined and stirred for 12h to afford 64% (29 mg) of a mixture of products.

^{1}H: 4-anisyl-4-nitrophenyliodonium triflate (**A**): 8.54 (app d, *J* = 9.1 Hz, 2 H), 8.39-8.33 (m, 4 H), 7.14 (app d, *J* = 9.2 Hz, 2 H), 3.88 (s, 3 H).

4-anisyl-1-naphthyliodonium triflate (**B**): 8.84 (dd, *J* = 7.5, 0.9 Hz, 1 H), 8.39 (app dd, *J* = 8.5, 0.7 Hz, 1 H), 8.32 (app d, *J* = 8.0 Hz, 1 H), 8.28 (app d, *J* = 9.2 Hz, 2 H), 8.07 (d, *J* = 8.1 Hz, 1 H), 7.85 (ddd, *J* = 8.3, 7.0, 1.3 Hz, 1 H), 7.73 (ddd, *J* = 8.1, 7.0, 1.0 Hz, 1 H), 7.66 (app t, *J* = 7.9, 1 H), 7.02 (app d, *J* = 9.2 Hz, 2 H), 3.79 (s, 3 H).

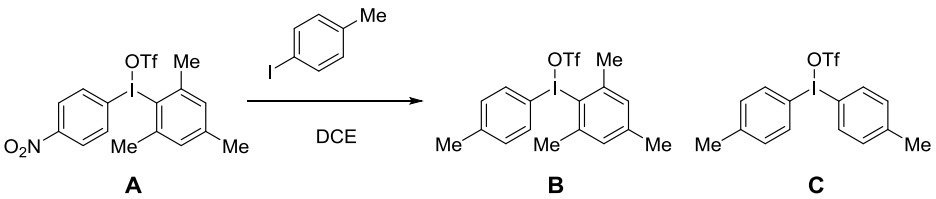
bis-(1-naphthyl)iodonium triflate (**C**): 8.94 (dd, *J* = 7.6, 1.0 Hz, 2 H), 8.52 (app d, *J* = 8.3 Hz, 2 H), 8.31-8.26 (m, 2 H), 8.01 (d, *J* = 8.3 Hz, 2 H), 7.85 (ddd, *J* = 8.3, 7.0, 1.3 Hz, 2 H), 7.76-7.59 (m, 4 H).

bis-(4-anisyl)iodonium triflate (**D**): 8.22 (app d, *J* = 9.3 Hz, 4 H), 7.09 (app d, *J* = 9.2 Hz, 4 H), 3.86 (s, 6 H).

1-naphthyl-4-nitrophenyliodonium triflate (**E**): 8.94 (dd, *J* = 7.5, 1.0 Hz, 1 H), 8.58 (app d, *J* = 9.2 Hz, 2 H), 8.42-8.25 (m, 4 H), 8.12-8.07 (m, 1 H), 7.89-7.63 (m, 3 H).

^{13}C: 164.2, 163.8, 150.9, 139.8, 139.1, 139.0, 138.8, 137.2, 136.0, 135.9, 135.8, 135.6, 135.04, 135.01, 132.4, 132.2, 131.2, 130.9, 130.6, 130.54, 130.50, 129.6, 129.5, 129.2, 129.0, 129.0, 128.7, 128.51, 128.47, 127.14, 127.12, 124.2, 121.8, 120.0, 118.9, 118.7, 118.0, 115.8, 104.3, 103.9, 103.4, 56.3, 56.2.

p. Solution metathesis between mesityl-4-nitrophenyliodonium triflate and 4-iodotoluene (Entry 3i)



4-iodotoluene (109 mg, 0.5 mmol) and mesityl-4-nitrophenyliodonium triflate (52 mg, 0.1 mmol) combined

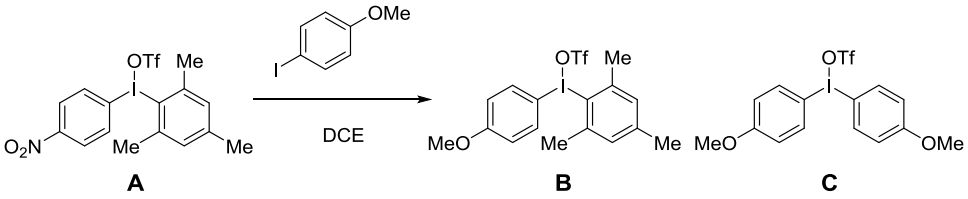
and stirred for 12h to afford 26% (12 mg) of a mixture of products.

^{1}H: mesityl-4-tolyliodonium triflate (**B**): 7.96 (app d, *J* = 8.5 Hz, 2 H), 7.43-7.36 (m, 2 H), 7.26 (s, 2 H), 2.71 (s, 6 H), 2.41 (s, 3 H), 2.35 (s, 3 H).

bis-(4-tolyl)iodonium triflate (**C**): 8.18 (app d, *J* = 8.5 Hz, 4 H), 7.40 (app d, *J* = 8.4 Hz, 4 H), 2.41 (s, 6 H).

^{13}C: 144.6, 143.3, 136.3, 135.3, 133.8, 133.7, 131.1, 124.3, 120.0, 111.8, 27.1, 21.3.

q. Solution metathesis between mesityl-4-nitrophenyliodonium triflate and 4-iodoanisole (Entry 3j)



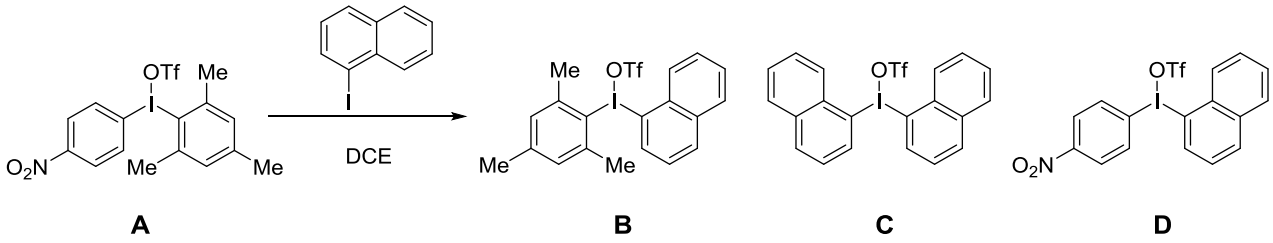
4-iodoanisole (117 mg, 0.5 mmol) and mesityl-4-nitrophenyliodonium triflate (52 mg, 0.1 mmol) combined and stirred for 12h to afford 37% (18 mg) of a mixture of products.

^{1}H: 4-anisyl-mesityl-iodonium triflate (B): 8.03 (app d, *J* = 9.2 Hz, 2 H), 7.25 (s, 2 H), 7.16-7.08 (m, 2 H), 3.87 (s, 3 H), 2.72 (s, 6 H), 2.34 (s, 3 H).

bis-(4-anisyl)iodonium triflate: 8.22 (app d, *J* = 9.2 Hz, 4 H), 7.10 (app d, *J* = 9.1 Hz, 4 H), 3.87 (s, 6 H).

^{13}C: 162.8, 142.1, 137.2, 136.6, 130.0, 123.2, 119.0, 117.7, 117.6, 103.4, 55.3, 26.0, 19.9.

r. Solution metathesis between mesityl-4-nitrophenyliodonium triflate and 1-iodonaphthalene (Entry 3k)



1-iodonaphthalene (73 µL, 0.5 mmol) and mesityl-4-nitrophenyliodonium triflate (52 mg, 0.1 mmol) combined and stirred for 12h to afford 27% (14 mg) of a mixture of products.

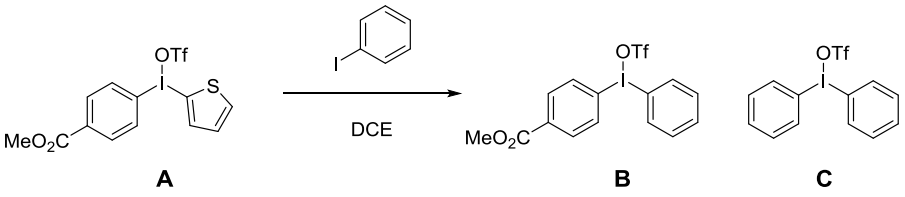
^{1}H: mesityl-4-nitrophenyliodonium triflate (**A**): 8.37-8.29 (m, 4 H), 7.32 (s, 2 H), 2.71 (s, 6 H), 2.38 (s, 3 H).

bis-(1-naphthyl)iodonium triflate (**C**): 8.93 (dd, *J* = 7.5, 0.8 Hz, 2 H), 8.53 (d, *J* = 8.3 Hz, 2 H), 8.31-8.26 (m, 2 H), 8.04 (d, *J* = 8.1 Hz, 2 H), 7.85 (ddd, *J* = 8.3, 7.1, 1.2 Hz, 2 H), 7.72 (app ddd, *J* = 8.0, 7.3, 0.7 Hz, 2 H), 7.62 (t, *J* = 7.8, 2 H).

mesityl-1-naphyliodonium triflate (**B**) and 4-nitrophenyl-1-naphthyliodonium triflate (**D**) were only detected through LC-MS and peak assignments for ^{1}H NMR was not possible.

^{13}C: 145.6, 143.6, 143.5, 138.9, 136.16, 136.14, 135.9, 135.0, 132.5, 131.3, 130.9, 130.5, 129.6, 129.1, 128.5, 127.3, 124.2, 122.6, 119.9. 119.8, 118.1, 55.5, 27.1, 21.0.

s. Solution metathesis between 4-carbomethoxyphenyl-2-thienyliodonium triflate and iodobenzene (Entry 4a)



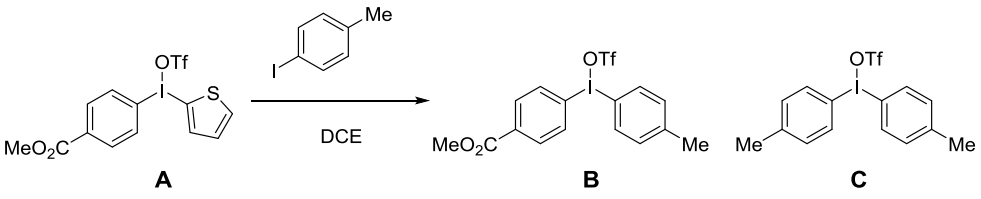
Iodobenzene (67 µL, 0.6 mmol) and 4-carbomethoxyphenyl-2-thienyliodonium triflate (60 mg, 0.12 mmol) combined and stirred for 12h to afford 40% (21 mg) of a mixture of products.

^{1}H: 4-carbomethoxyphenyl-phenyliodonium triflate (**B**): 8.46 (app d, *J* = 8.7 Hz, 2 H), 8.40-8.34 (m, 2 H), 8.10 (app d, *J* = 8.6 Hz, 2 H), 7.81-7.74 (m, 1 H), 7.64-7.58 (m, 2 H).

diphenyliodonium triflate (**C**): 8.34 (app dd, *J* = 8.6, 1.1 Hz, 4 H), 7.75 (tt, *J* = 7.4, 1.1 Hz, 2 H), 7.60 (app t, *J* = 7.9 Hz, 4 H).

^{13}C: 136.5, 133.5, 133.0, 124.3, 120.0, 115.3.

t. Solution metathesis between 4-carbomethoxyphenyl-2-thienyliodonium triflate and 4-iodotoluene (Entry 4b)



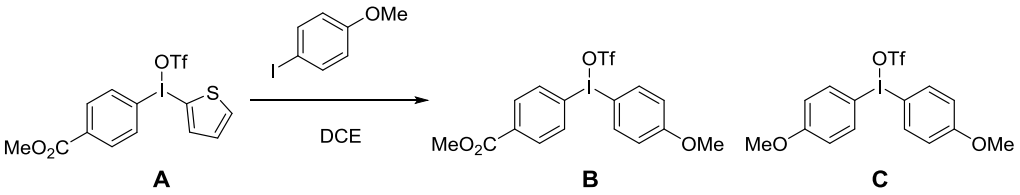
4-iodotoluene (132 mg, 0.60 mmol) and 4-carbomethoxyphenyl-2-thienyliodonium triflate (60 mg, 0.12 mmol) combined and stirred for 12h to afford 66% (37 mg) of a mixture of products.

^{1}H: 4-carbomethoxyphenyl-4-tolyliodonium triflate (**B**): 8.42 (app d, *J* = 8.7 Hz, 2 H), 8.23 (app d, *J* = 8.6 Hz, 2 H), 8.08 (app d, *J* = 8.6 Hz, 2 H), 7.43-7.37 (m, 2 H), 3.90 (s, 3 H).

bis-(4-tolyl)iodonium triflate (**C**): 8.18 (app d, *J* = 8.5 Hz, 4 H), 7.38 (app d, *J* = 8.3 Hz, 4 H), 2.39 (s, 6 H).

^{13}C: 136.7, 136.4, 136.3, 133.8, 133.6, 133.1, 124.2, 119.9, 111.8, 53.0, 21.3.

u. Solution metathesis between 4-carbomethoxyphenyl-2-thienyliodonium triflate and 4-iodoanisole (Entry 4c)

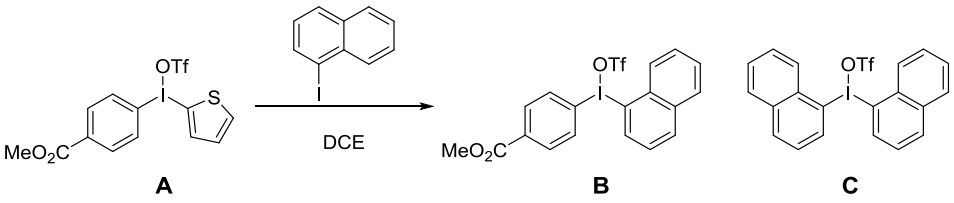


4-iodoanisole (140 mg, 0.6 mmol) and 4-carbomethoxyphenyl-2-thienyliodonium triflate (60 mg, 0.12 mmol) combined and stirred for 12h to afford 91% (55 mg) of a mixture of products.

^{1}H: 4-carbomethoxyphenyl-4-anisyliodonium triflate (**B**): 8.39 (app d, *J* = 8.7 Hz, 2 H), 8.29 (app d, *J* = 9.2 Hz, 2 H), 8.07 (app d, *J* = 8.7 Hz, 2 H), 7.12 (app d, *J* = 9.3 Hz, 2 H), 3.90 (s, 3 H), 3.87 (s, 3 H).
bis-(4-anisyl)iodonium triflate (**C**): 8.21 (app d, *J* = 9.2 Hz, 4 H), 7.09 (app d, *J* = 9.2 Hz, 4 H), 3.86 (s, 6 H).

^{13}C: 165.9, 164.0, 163.8, 142.8, 141.8, 139.1, 138.9, 138.4, 138.2, 138.0, 136.1, 135.8, 134.3, 133.1, 130.8, 130.6, 128.3, 124.1, 120.5, 119.8, 118.8, 118.53, 118.48, 115.6, 104.4, 103.7, 98.7, 56.3, 56.2, 53.0.

v. Solution metathesis between 4-carbomethoxyphenyl-2-thienyliodonium triflate and 1-iodonaphthalene (Entry 4d)



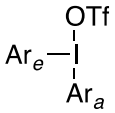
1-iodonaphthalene (88 µL, 0.6 mmol) and 4-carbomethoxyphenyl-2-thienyliodonium triflate (60 mg, 0.12 mmol) combined and stirred for 12h to afford 38% (24 mg) of a mixture of products.

^{1}H: 4-carbomethoxyphenyl-1-naphthyliodonium triflate (**B**): 8.94-8.90 (m, 1 H), 8.45 (app d, *J* = 8.7 Hz, 2 H), 8.38 (d, *J* = 8.3 Hz, 1 H), 8.37 (d, *J* = 8.4 Hz, 1 H), 8.10 (d, *J* = 8.4 Hz, 1 H), 8.03 (app d, *J* = 8.6 Hz, 2 H), 7.85 (ddd, *J* = 8.3, 7.0, 1.2 Hz, 1 H), 7.79-7.60 (m, 2 H), 3.86 (s, 3H).
bis-(1-naphthyl)iodonium triflate (**C**): 8.94 (dd, *J* = 7.6, 0.9 Hz, 2 H), 8.53 (app d, *J* = 8.5 Hz, 2 H), 8.27 (d, *J* = 8.2 Hz, 2 H), 8.02 (app d, *J* = 8.3 Hz, 2 H), 7.85 (ddd, *J* = 8.3, 7.0, 1.2 Hz, 2 H), 7.71 (ddd, *J* = 8.1, 7.2, 0.8 Hz, 2 H), 7.61 (t, *J* = 7.9 Hz, 2 H).

^{13}C: 139.6, 139.0, 136.1, 135.9, 135.5, 135.1, 133.2, 132.53, 132.45, 131.1, 130.9, 130.63, 130.55, 129.55, 129.2, 129.1, 128.7, 128.5, 127.8, 124.2, 120.0, 119.4, 117.9, 53.0.

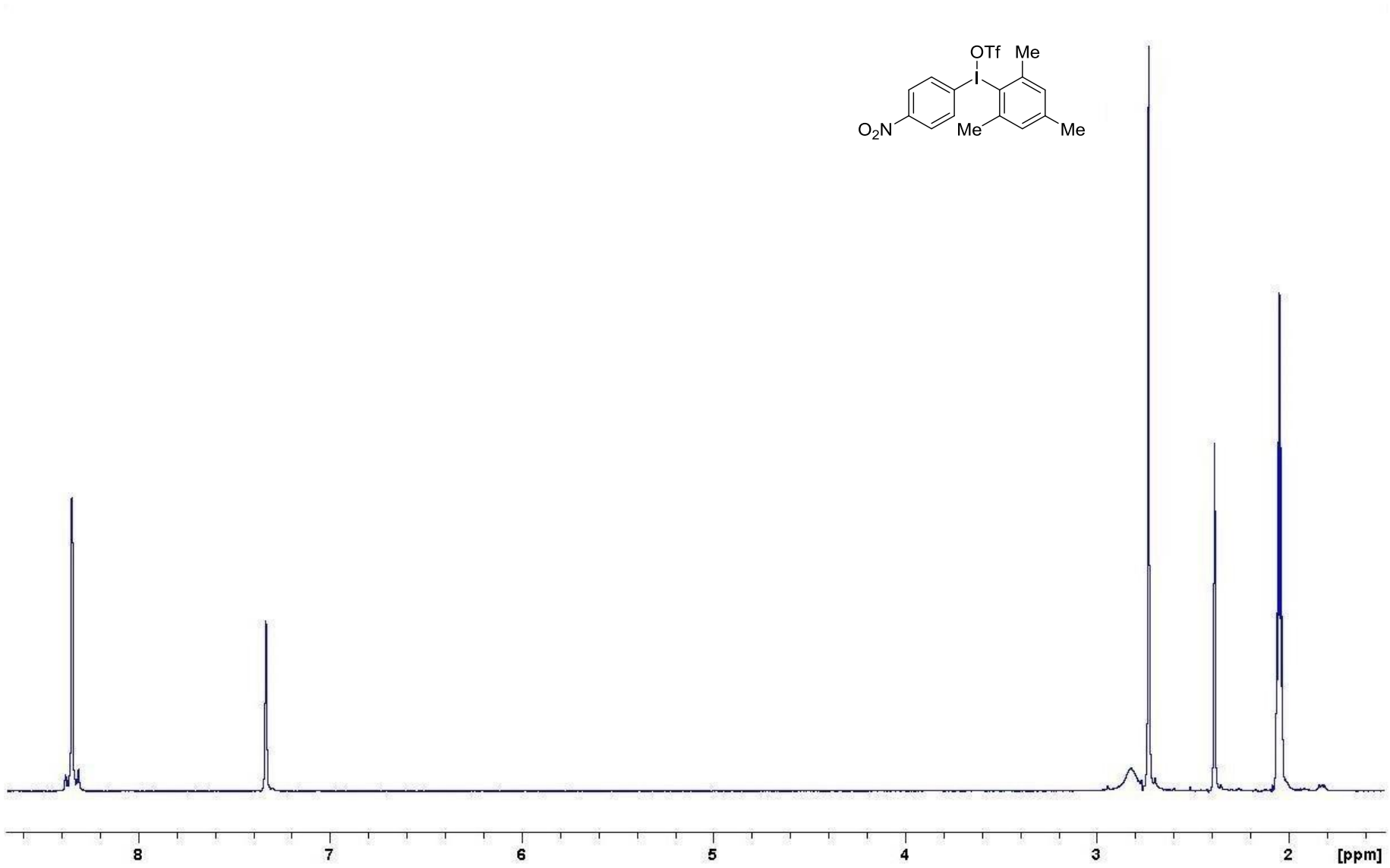
5. Table of calculated charges on the I-atom of selected diaryliodonium triflates

Note: charge values are reported only for the more thermodynamically favorable stereoisomer of the diaryliodonium triflate, which in all cases, and in accord with ref. 11b in the manuscript, was predicted to be the one with the more electron-deficient aryl ligand at an axial position (cf. Ar_a), and the more electron-rich one, at an equatorial position (cf. Ar_e). As stated in the manuscript (ref. 17), semiempirical calculations tend to be imprecise for hypervalent halogen compounds (cf. J. J. P. Stewart, *J. Comput. Chem.* **1989**, *10*, 221). Accordingly, the data below should be interpreted only as reflecting a trend.

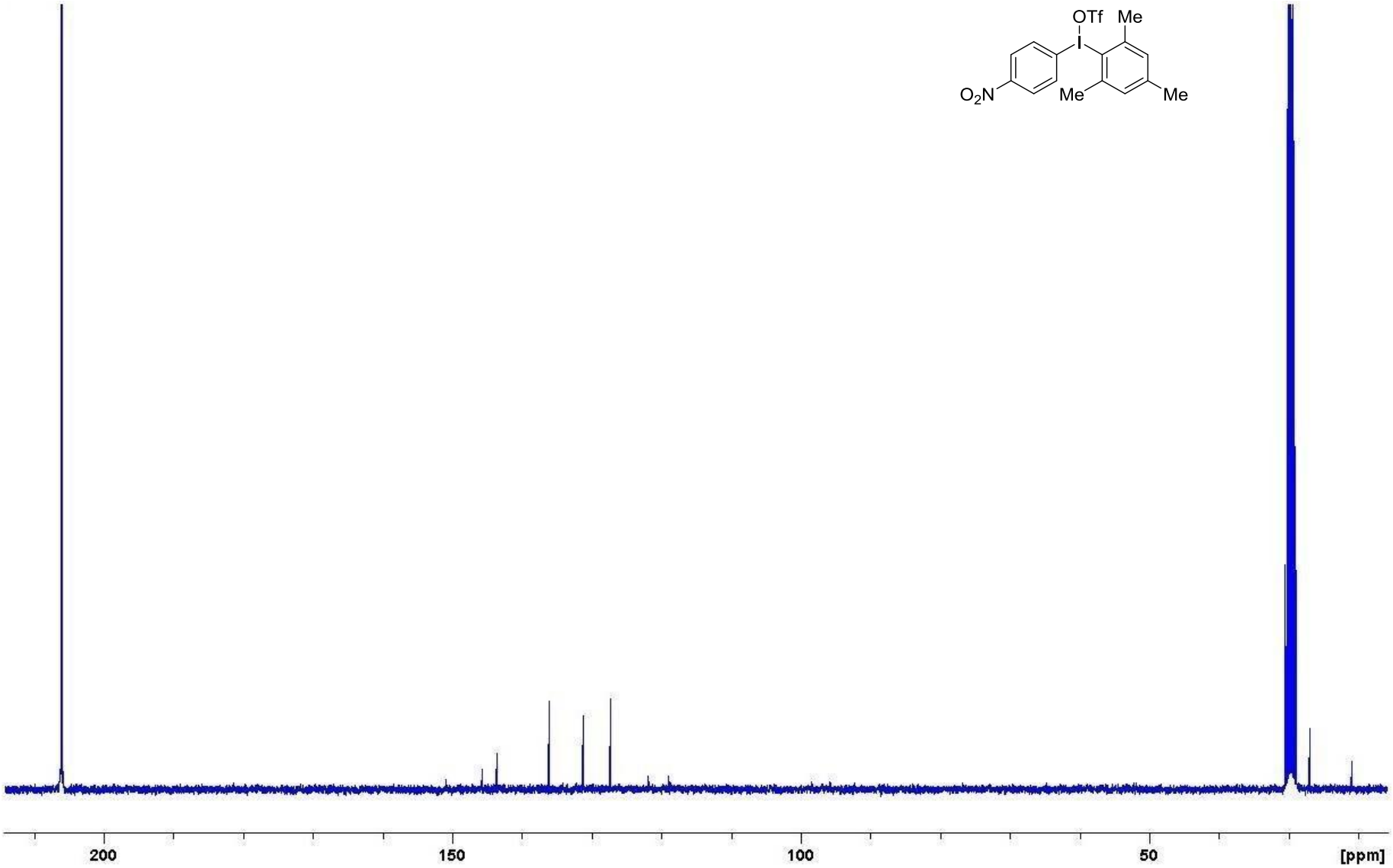
structure			charge on the I atom[a]	
	Ar_e	**Ar_a**	**MNDO**	**MNDO-d**
	1-Naphthyl	Ph	+ 0.859	+ 1.366
	Ph	Ph	+ 0.875	+ 1.379
	4-Me-C_6H_4	4-Me-C_6H_4	+ 0.876	+ 1.381
	Ph	4-MeOOC-C_6H_4	+ 0.882	+ 1.381
	Ph	4-O_2N-C_6H_4	+ 0.896	+ 1.401
	4-MeOOC-C_6H_4	2-Thienyl	+ 0.947	+ 1.453

[a] Values are in units of electron charge, *e*

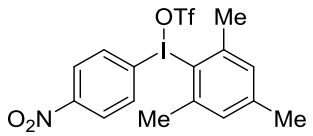
5. Proton and ^{13}C NMR Spectra



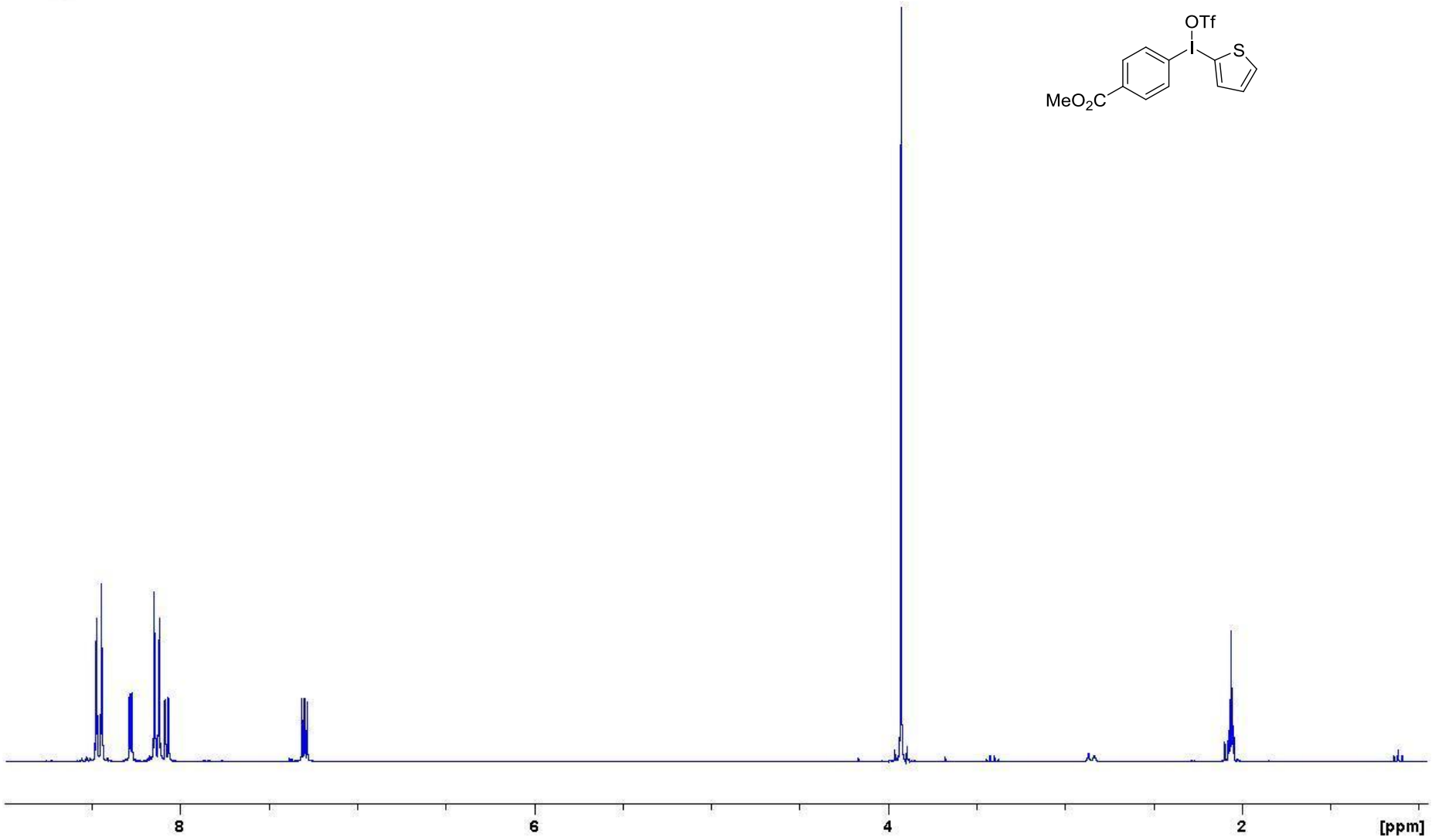
^{1}H NMR spectrum of mesityl-4-nitrophenyliodonium triflate (acetone-d_6)



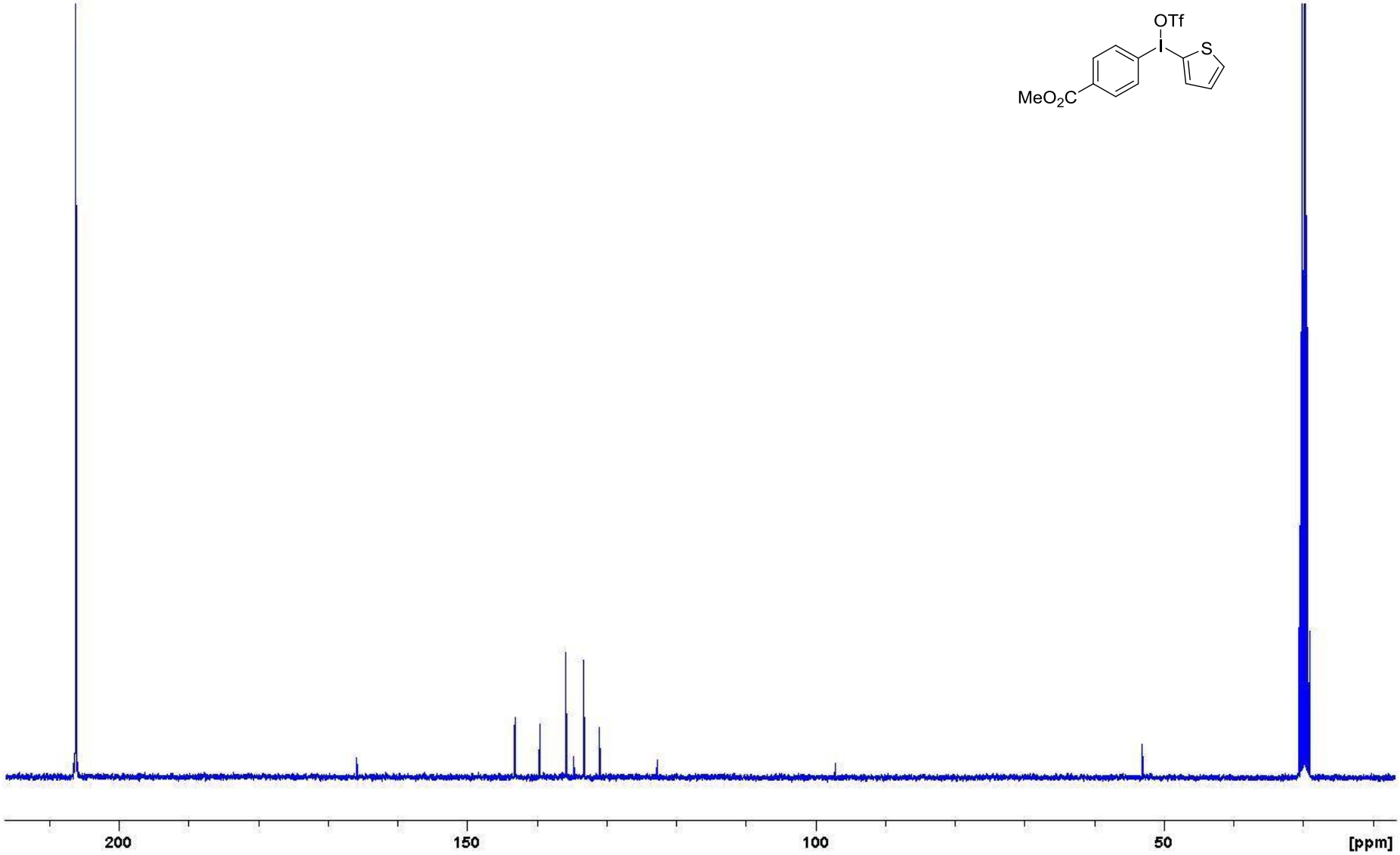
^{13}C NMR spectrum of mesityl-4-nitrophenyliodonium iodonium triflate (acetone-d_6)



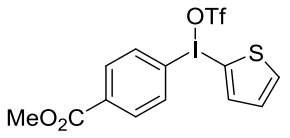
Expanded ^{13}C NMR spectrum of mesityl-4-nitrophenyliodonium iodonium triflate (acetone-d_6)

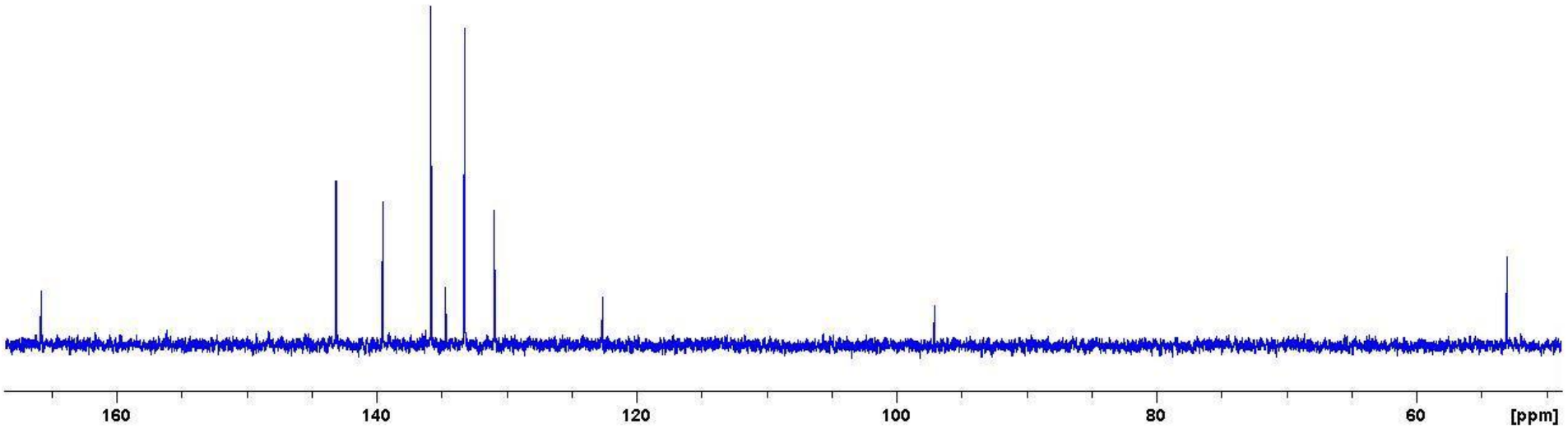


^{1}H NMR spectrum of 4-carbomethoxyphenyl-2-thienyliodonium triflate (acetone-d_6)

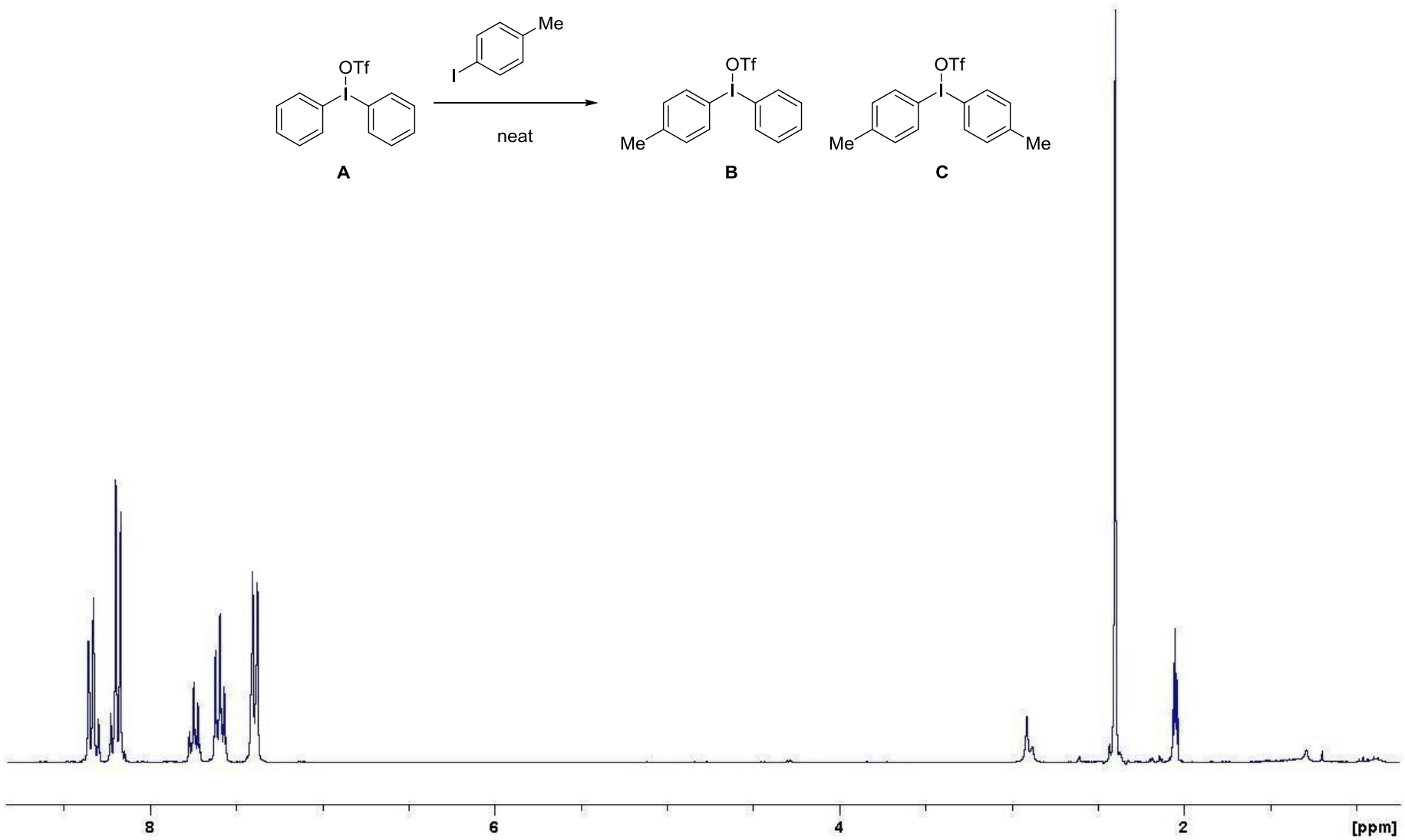


^{13}C NMR spectrum of 4-carbomethoxyphenyl-2-thienyliodonium triflate (acetone-d_6)

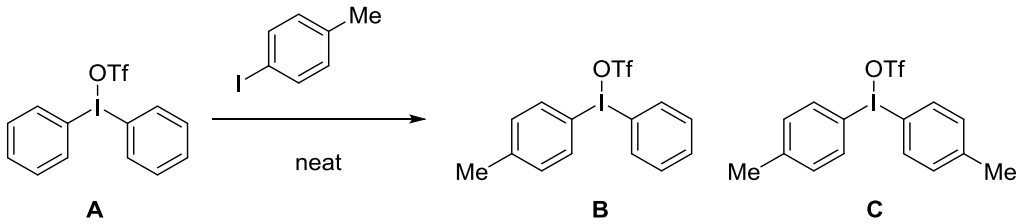


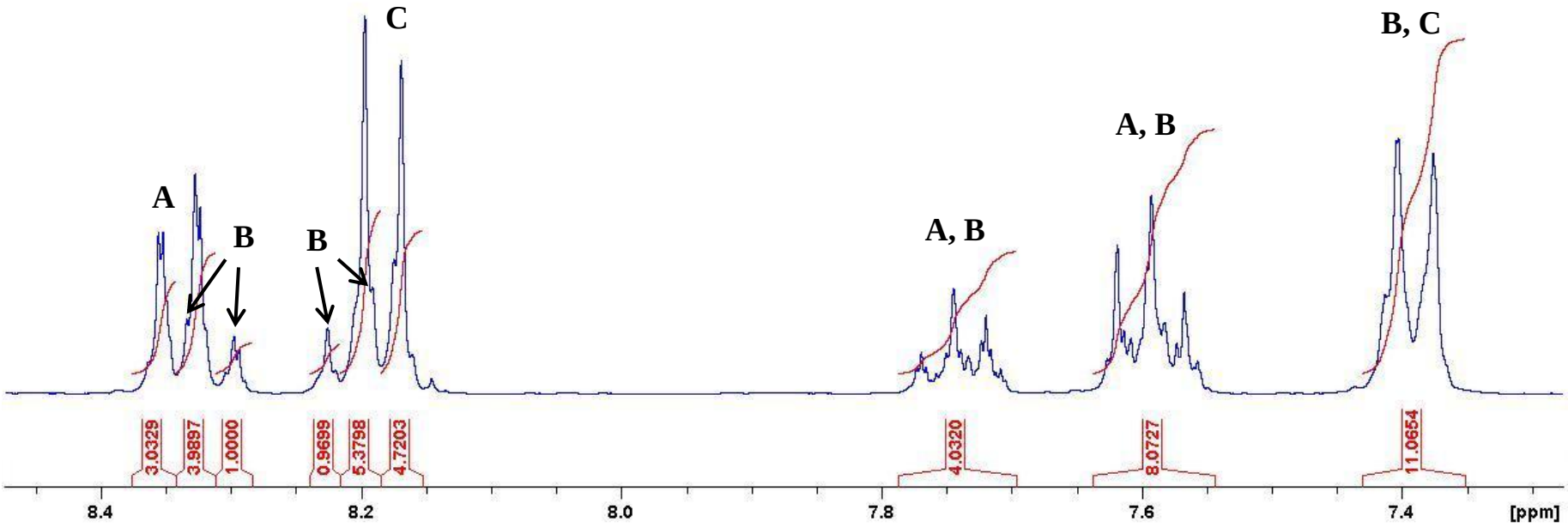


Expanded ^{13}C NMR spectrum of 4-carbomethoxyphenyl-2-thienyliodonium triflate (acetone-d_6)

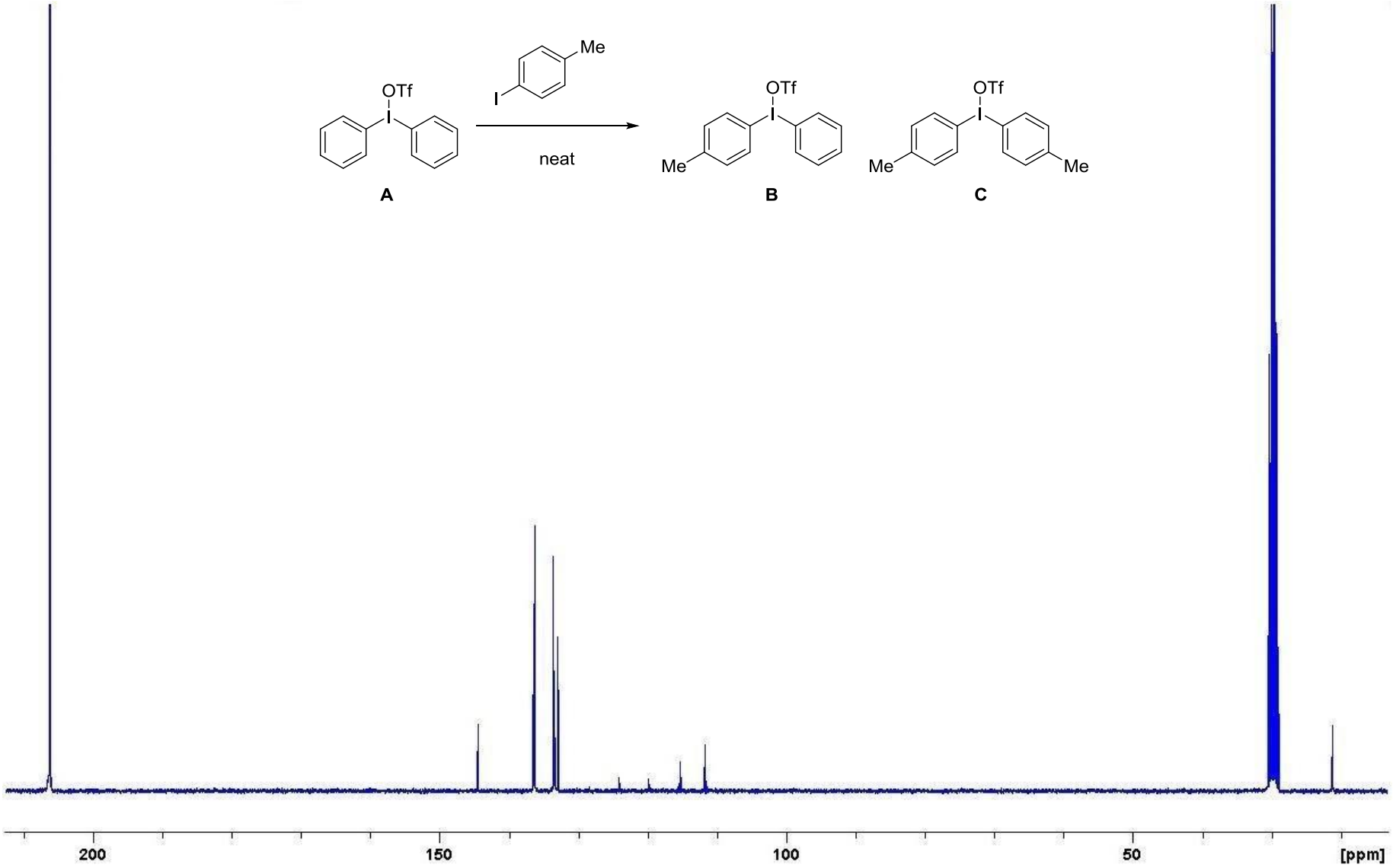


^{1}H NMR spectrum of the product mixture obtained upon melt metathesis of Ph_2IOTf and 4-iodotoluene (acetone-d_6)

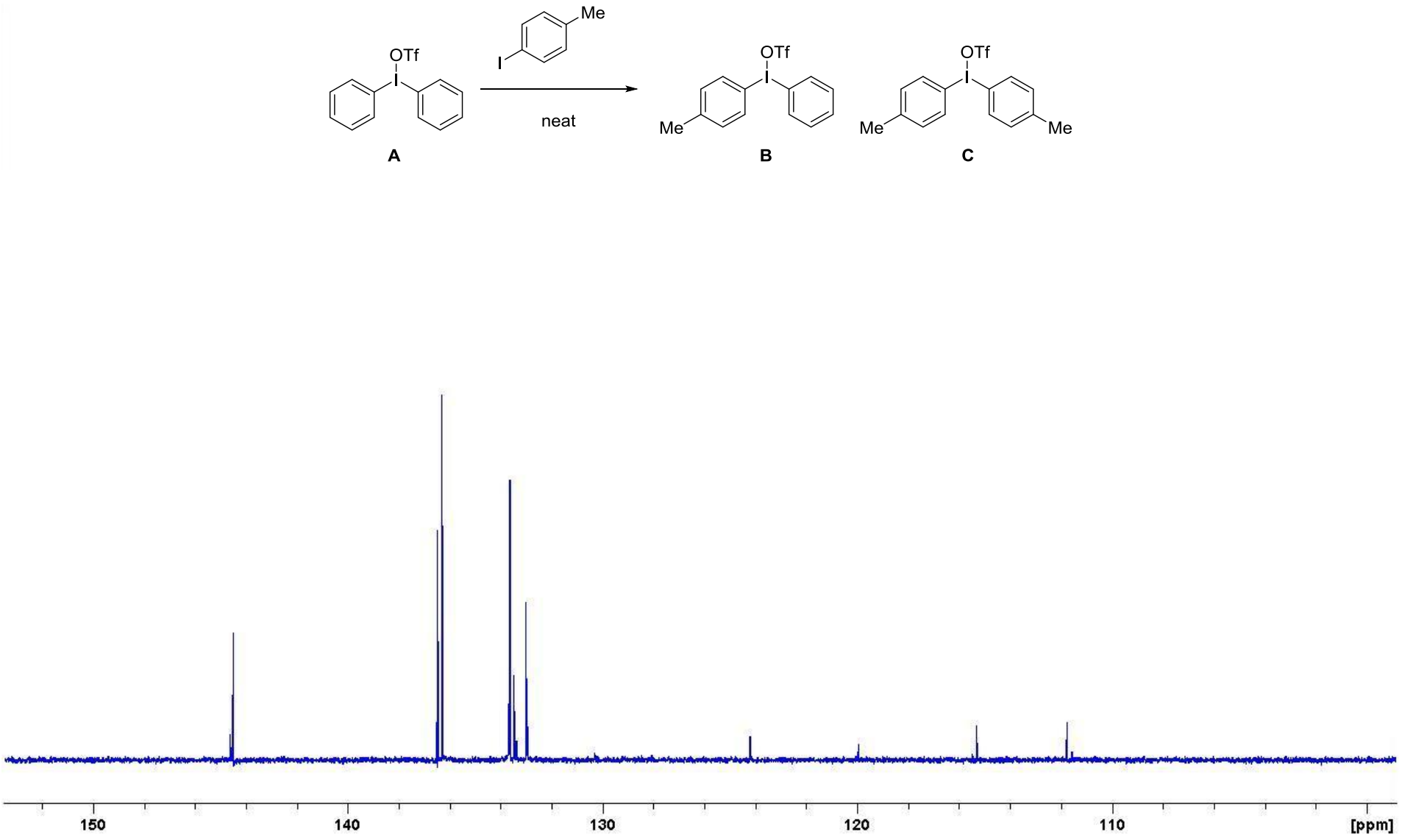




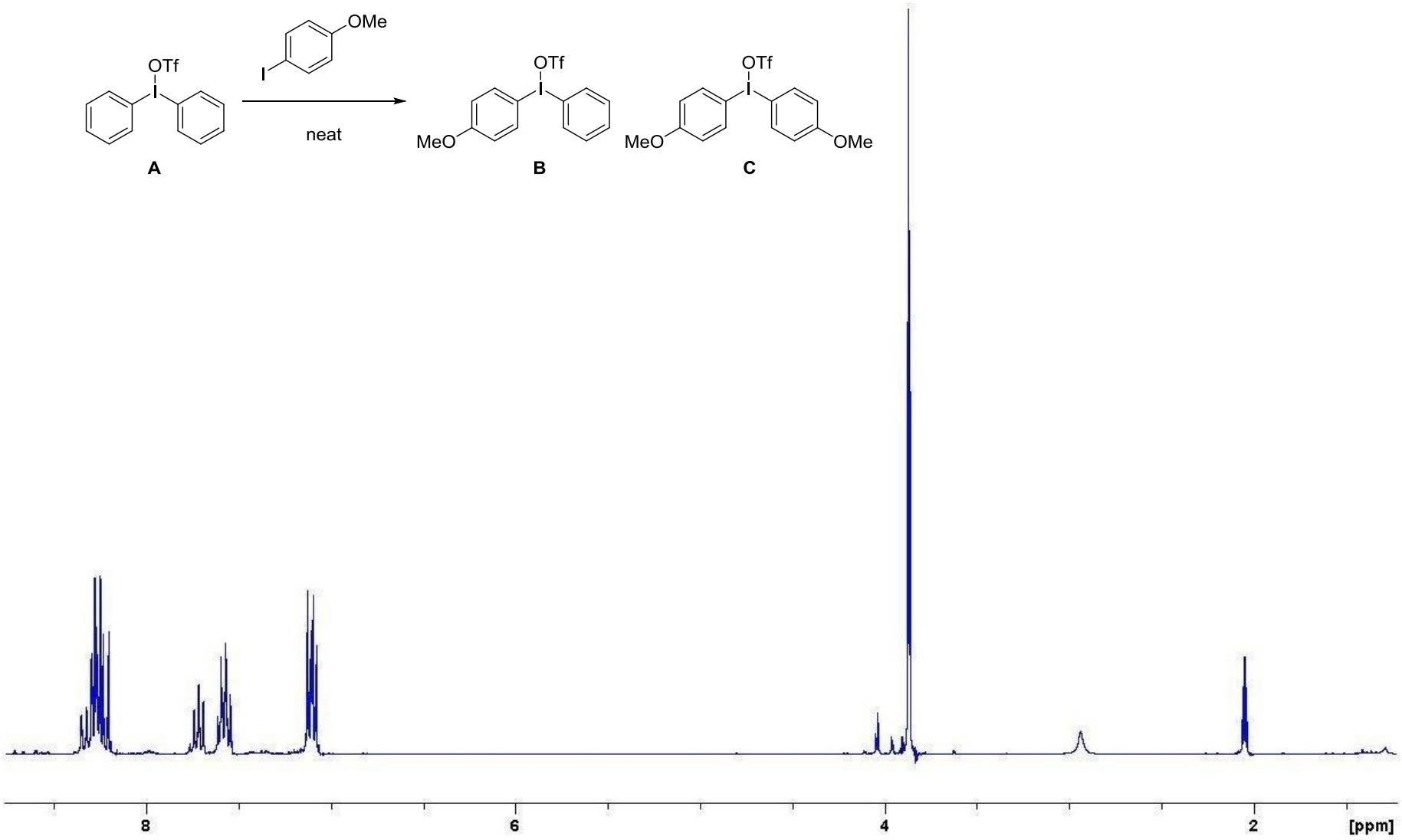
Expanded ^{1}H NMR spectrum of the product mixture obtained upon melt metathesis of Ph_2IOTf and 4-iodotoluene (acetone-d_6)



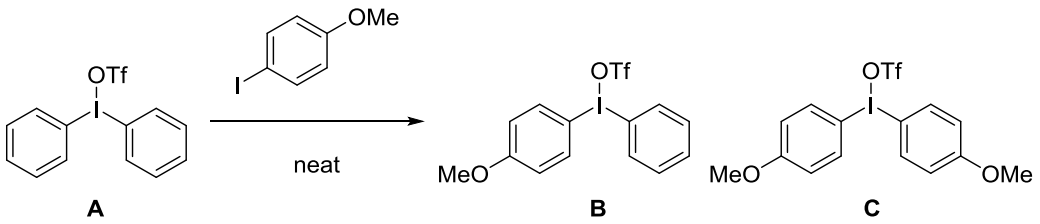
^{13}C NMR spectrum of the product mixture obtained upon melt metathesis of Ph_2IOTf and 4-iodotoluene (acetone-d_6)

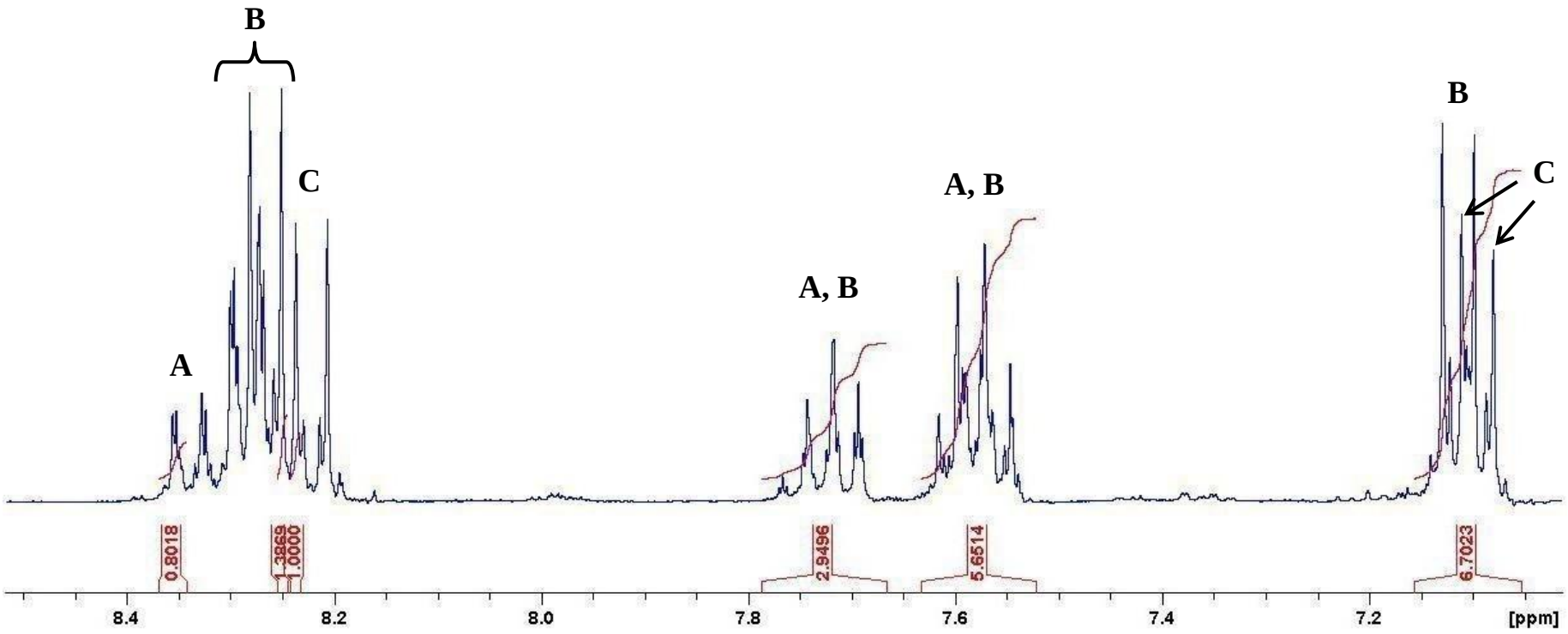


Expanded ^{13}C NMR spectrum of the product mixture obtained upon melt metathesis of Ph_2IOTf and 4-iodotoluene (acetone-d_6)

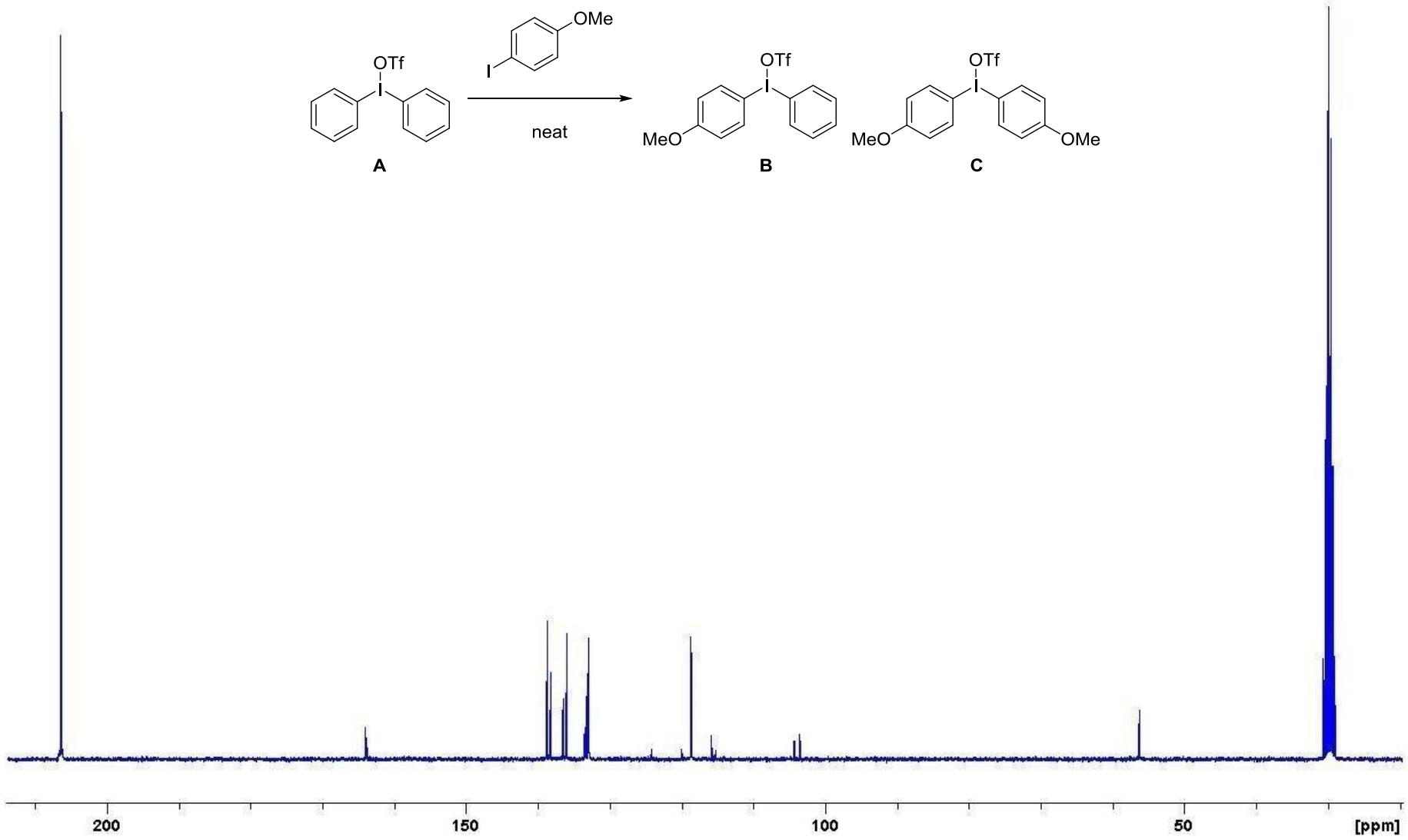


^{1}H NMR spectrum of the product mixture obtained upon melt metathesis of Ph_2IOTf and 4-iodoanisole (acetone-d_6)

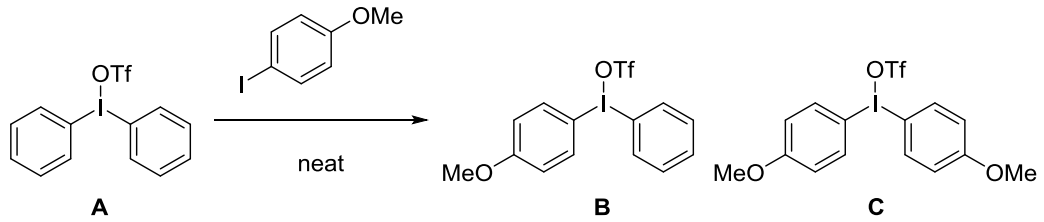


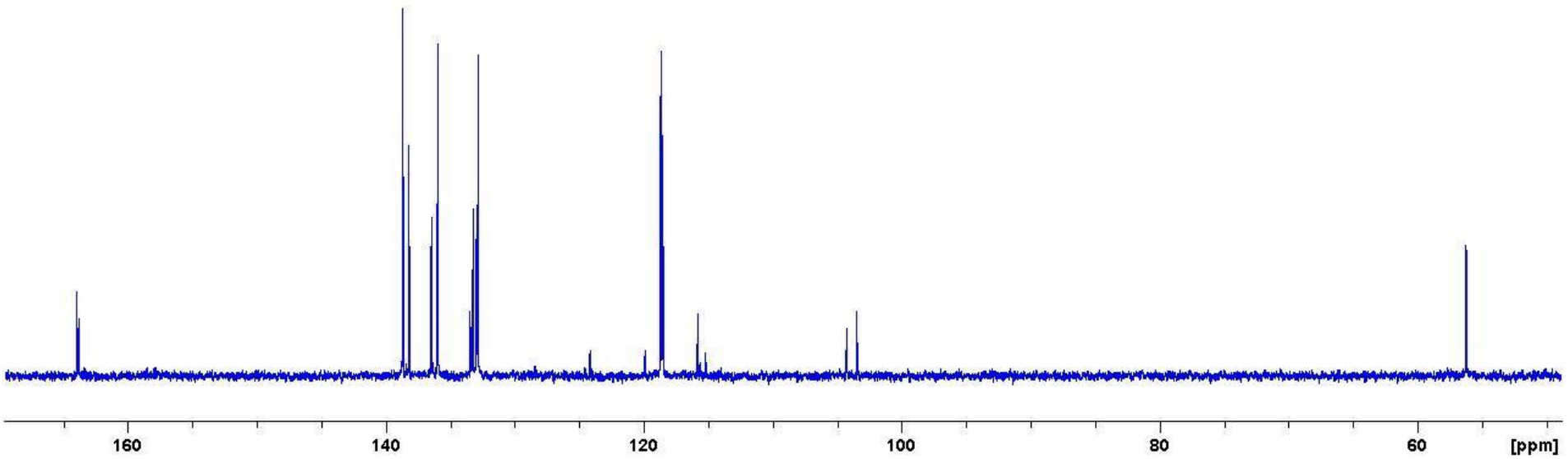


Expanded ^{1}H NMR spectrum of the product mixture obtained upon melt metathesis of Ph_2IOTf and 4-iodoanisole (acetone-d_6)

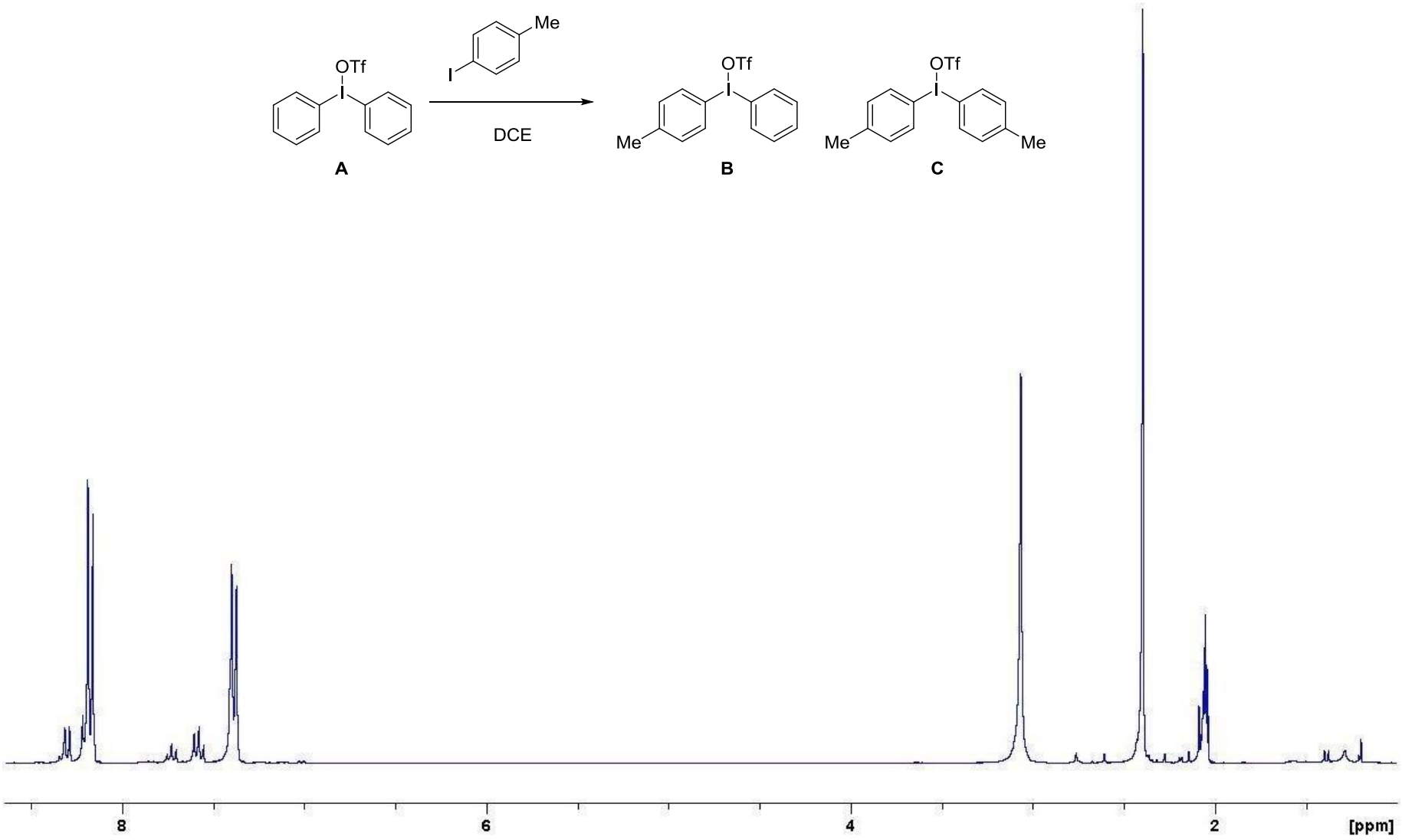


^{13}C NMR spectrum of the product mixture obtained upon melt metathesis of Ph_2IOTf and 4-iodoanisole (acetone-d_6)

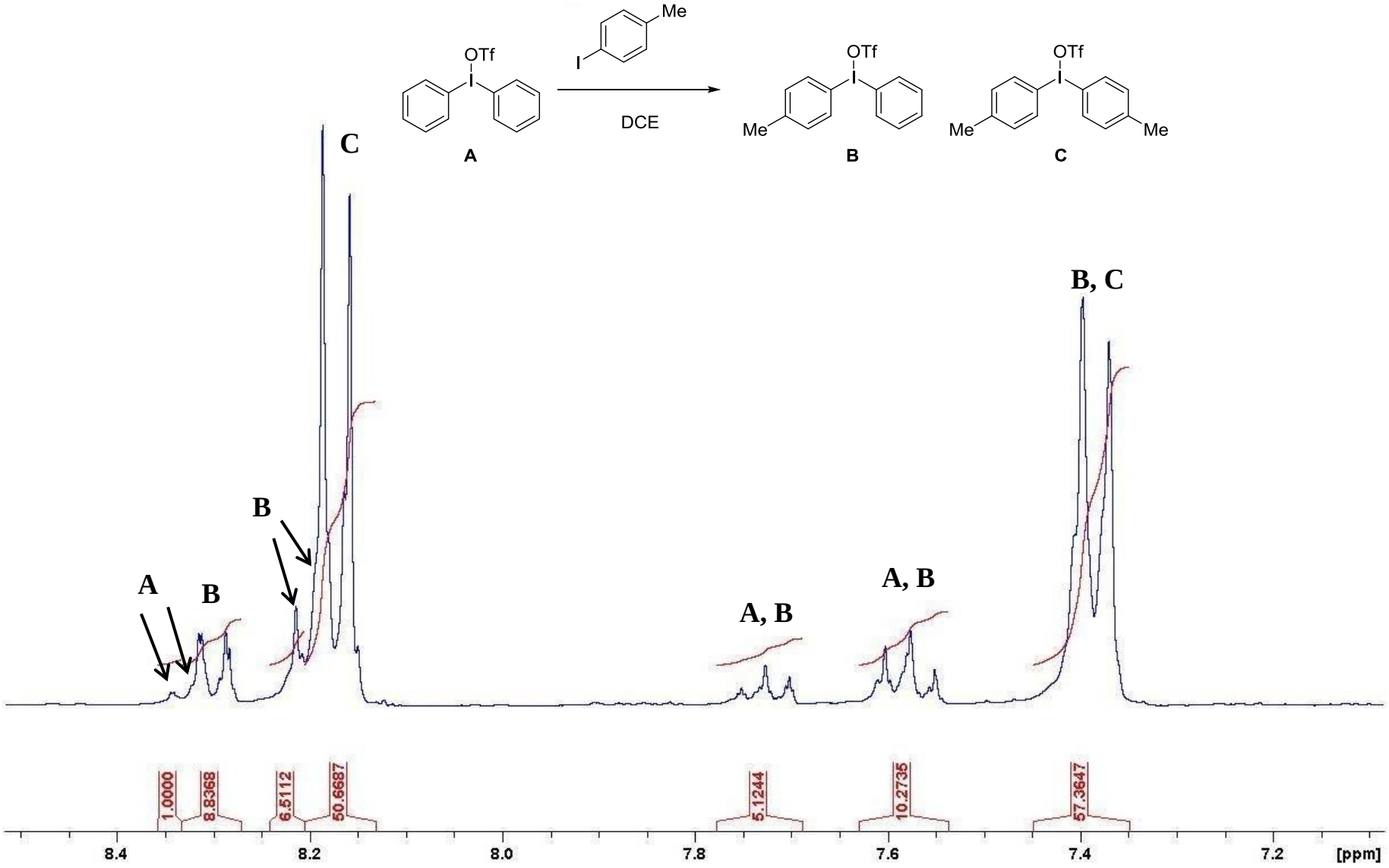




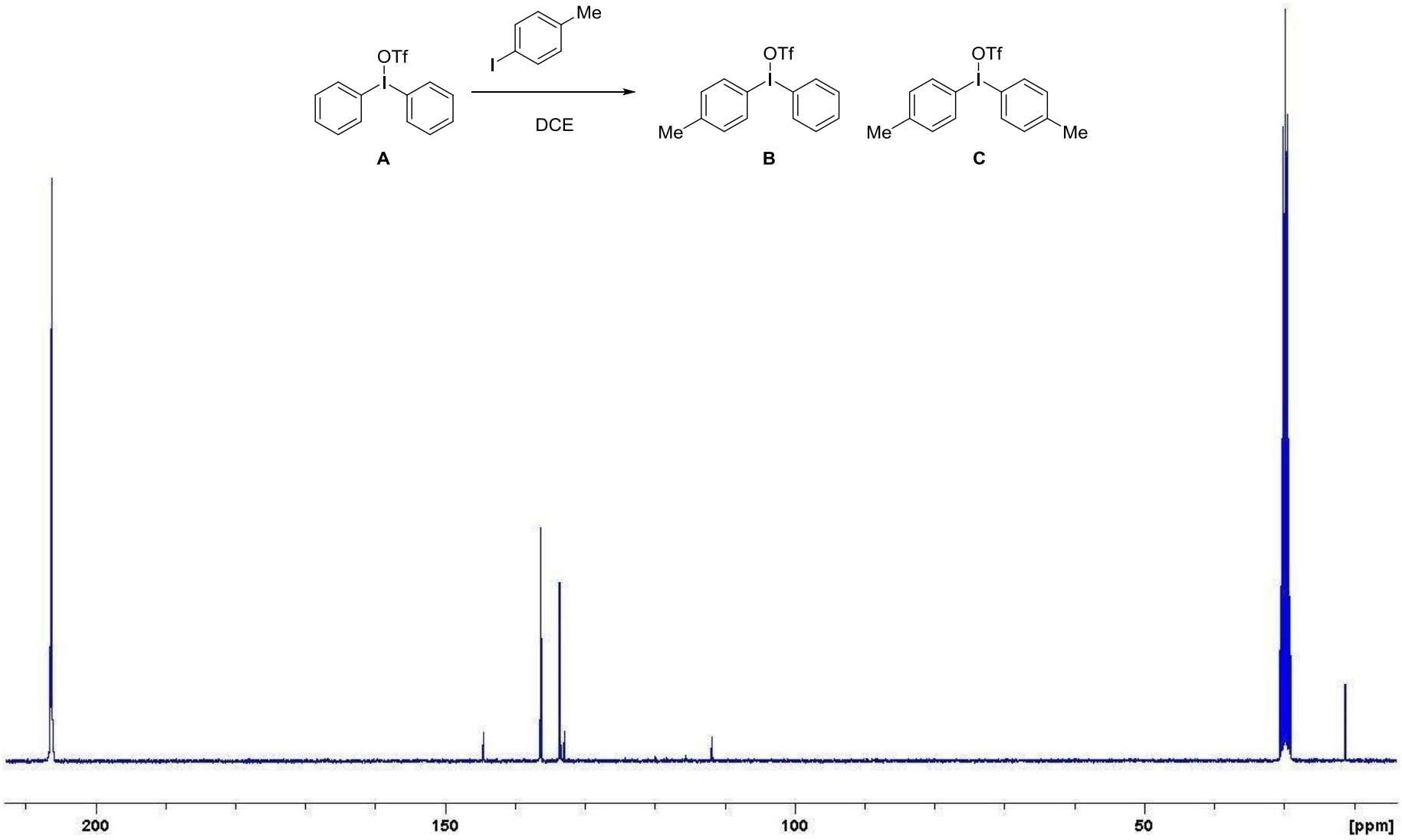
Expanded ^{13}C NMR spectrum of the product mixture obtained upon melt metathesis of Ph_2IOTf and 4-iodoanisole (acetone-d_6)



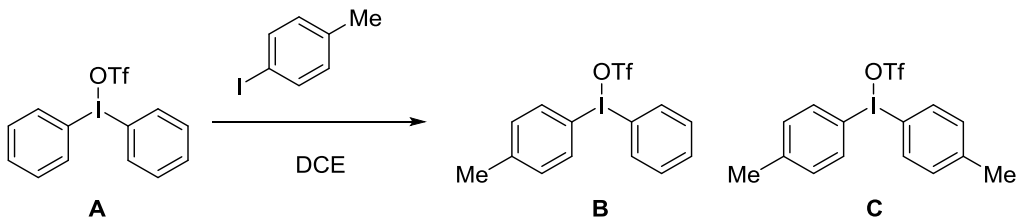
^{1}H NMR spectrum of the product mixture obtained in entry a of Table 1 (acetone-d_6)

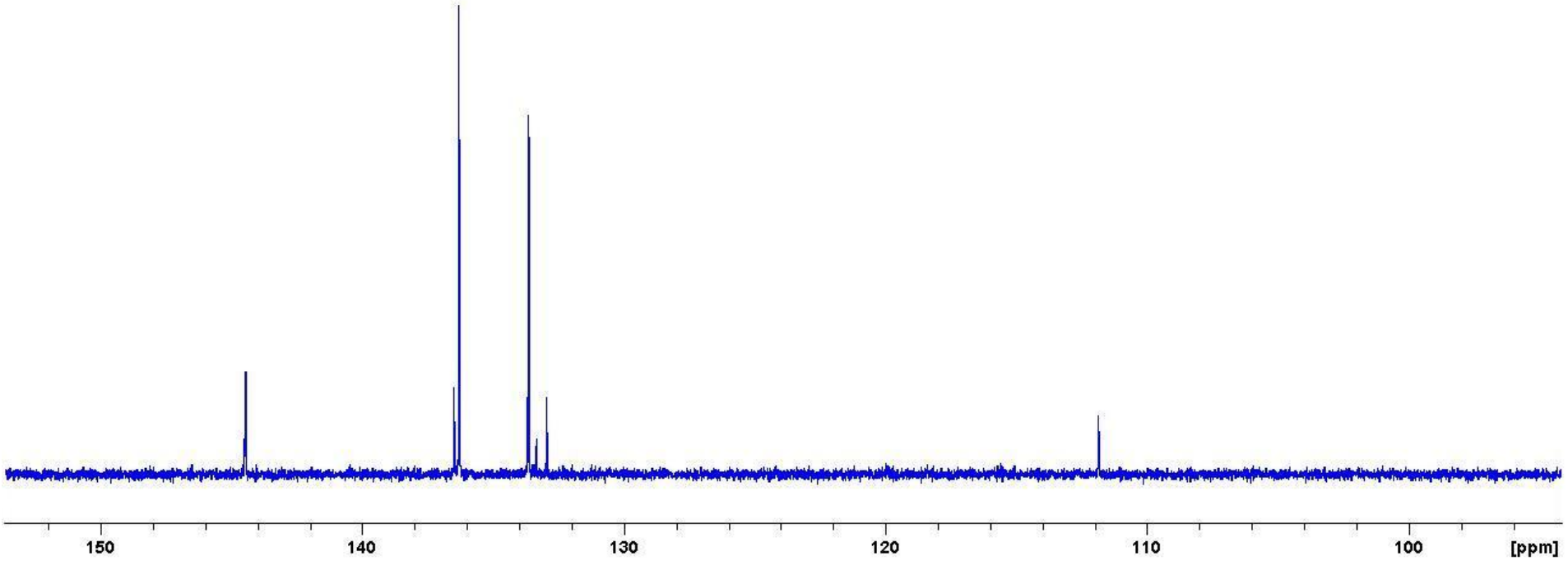


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry a of Table 1 (acetone-d_6)

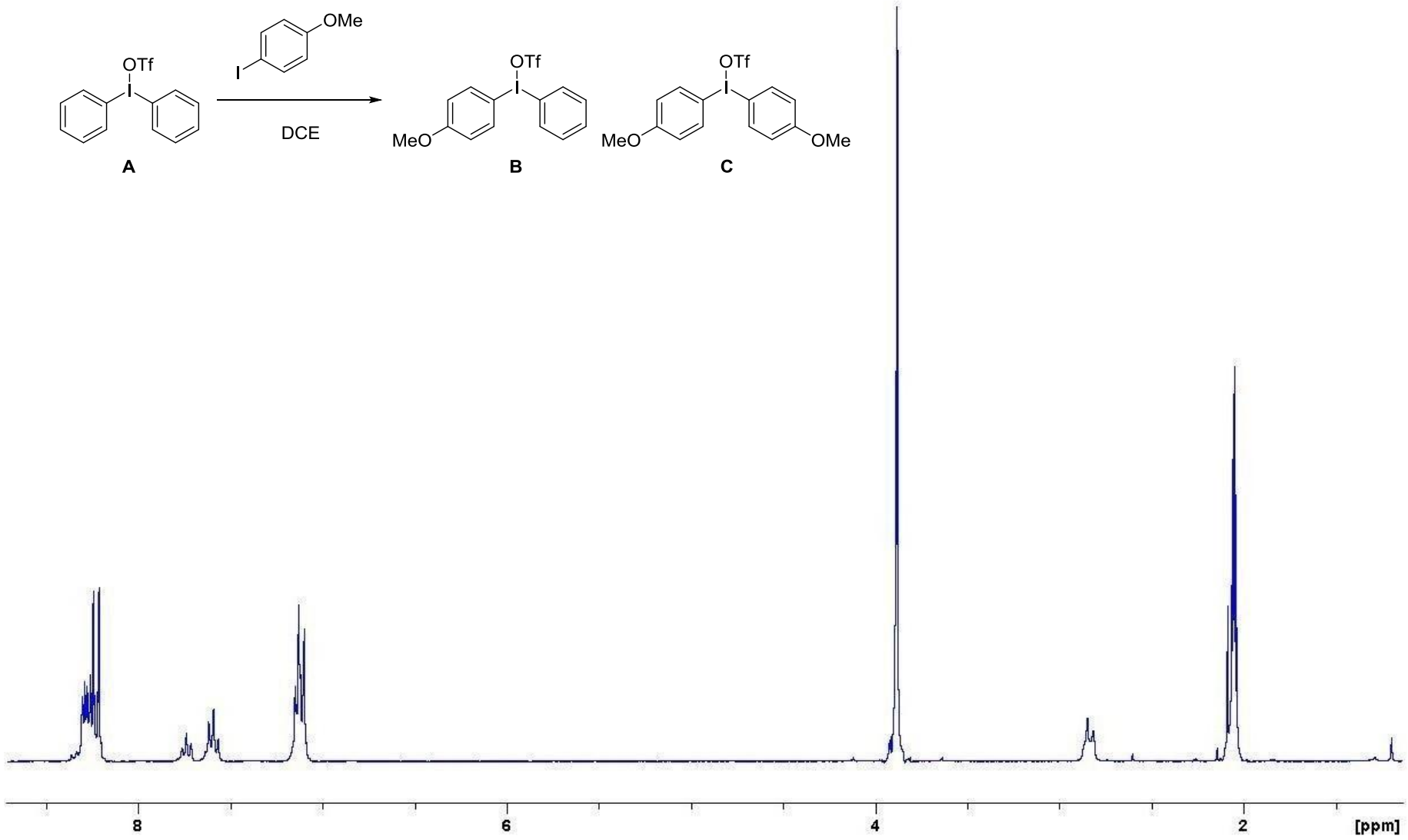


^{13}C NMR spectrum of the product mixture obtained in entry a of Table 1 (acetone-d_6)

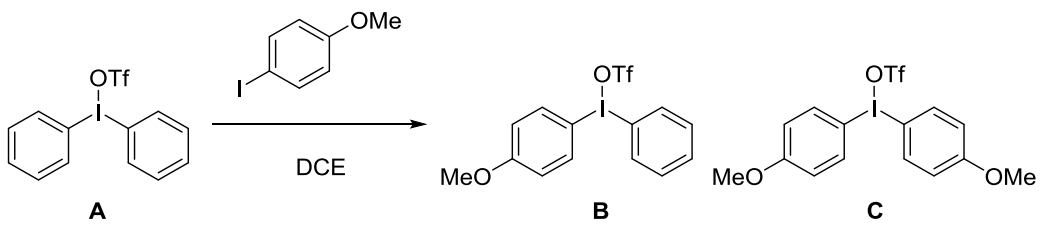


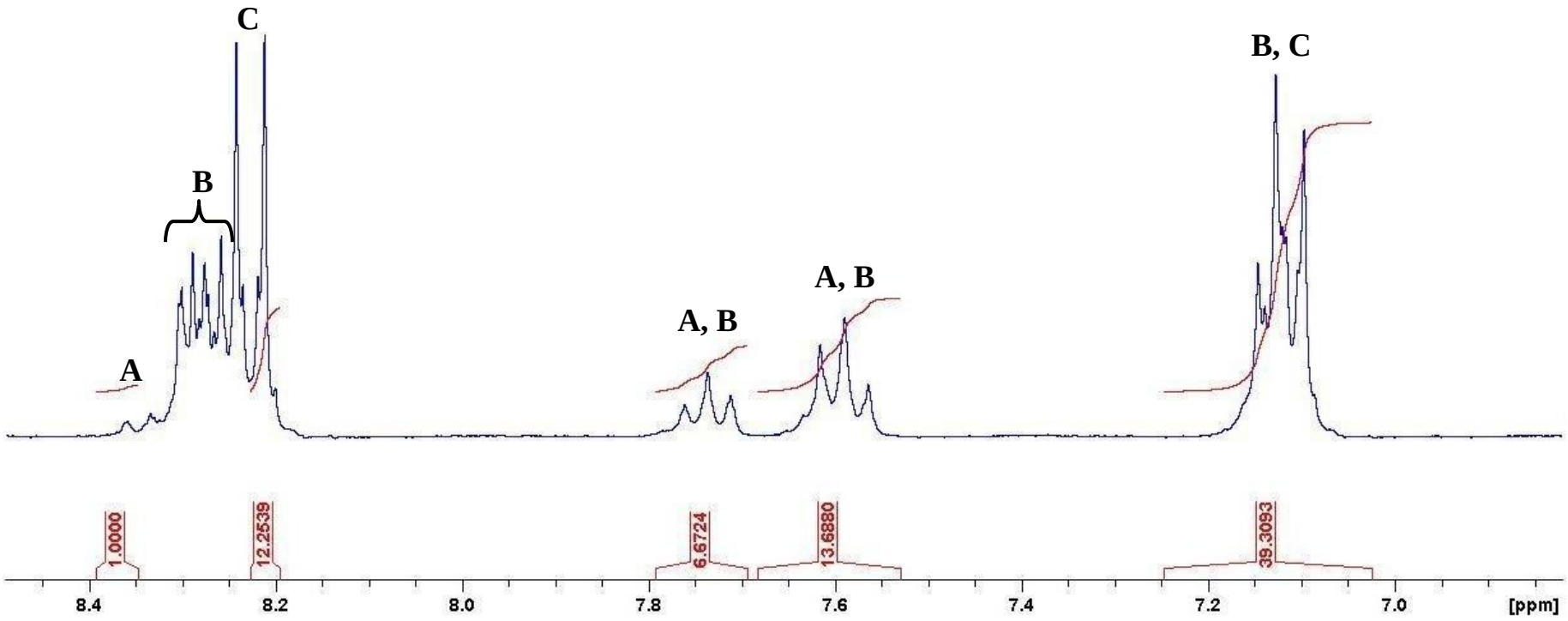


Expanded ^{13}C NMR spectrum of the product mixture obtained in entry a of Table 1 (acetone-d_6)

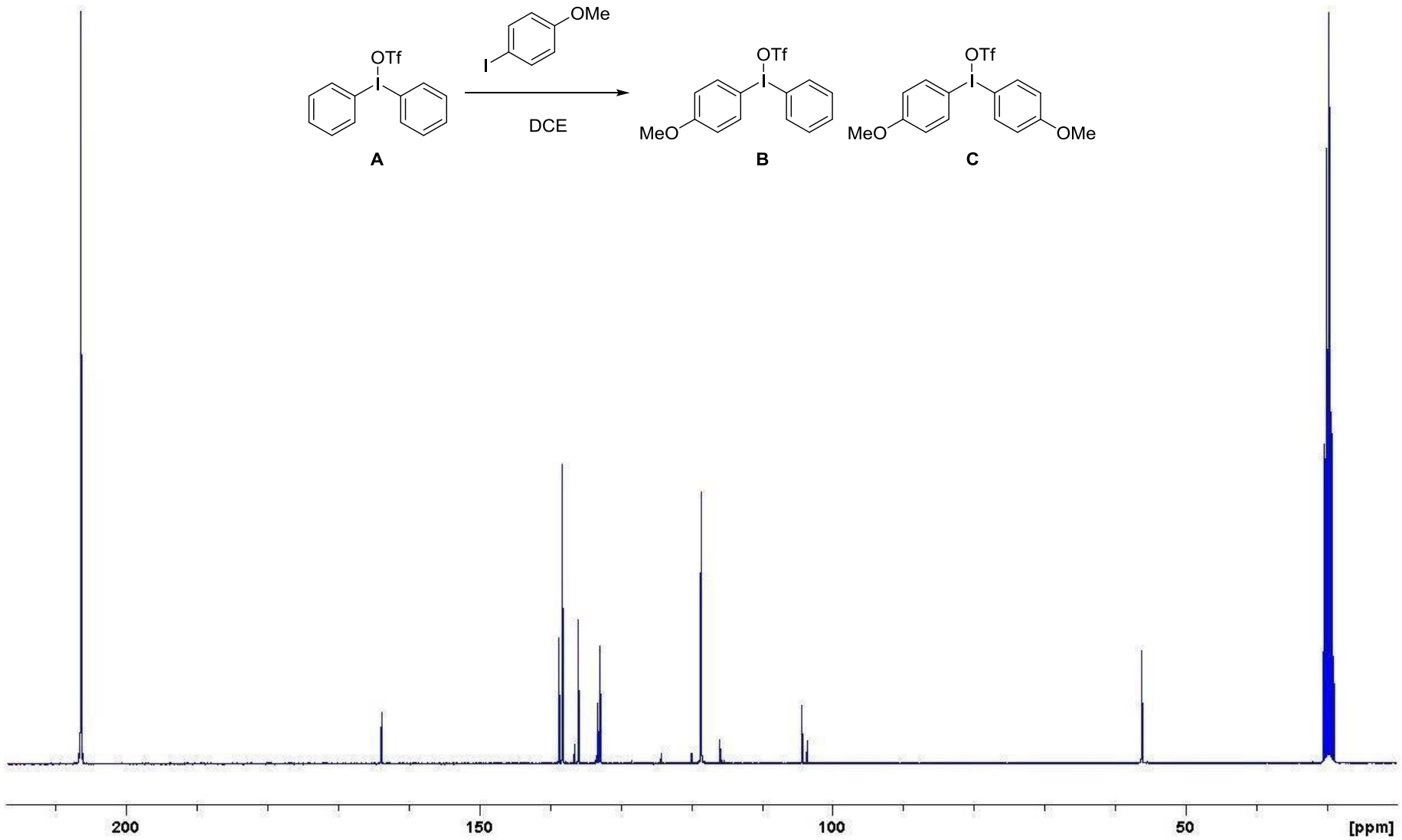


^{1}H NMR spectrum of the product mixture obtained in entry b of Table 1 (acetone-d_6)

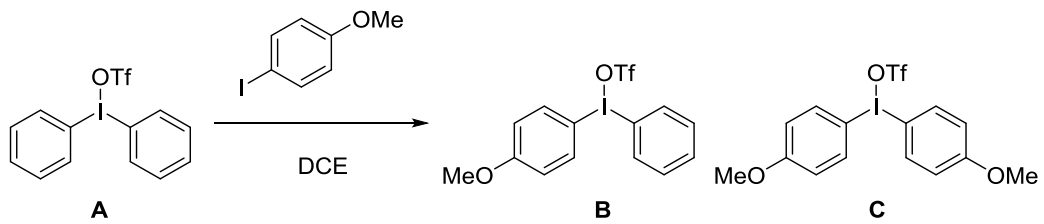


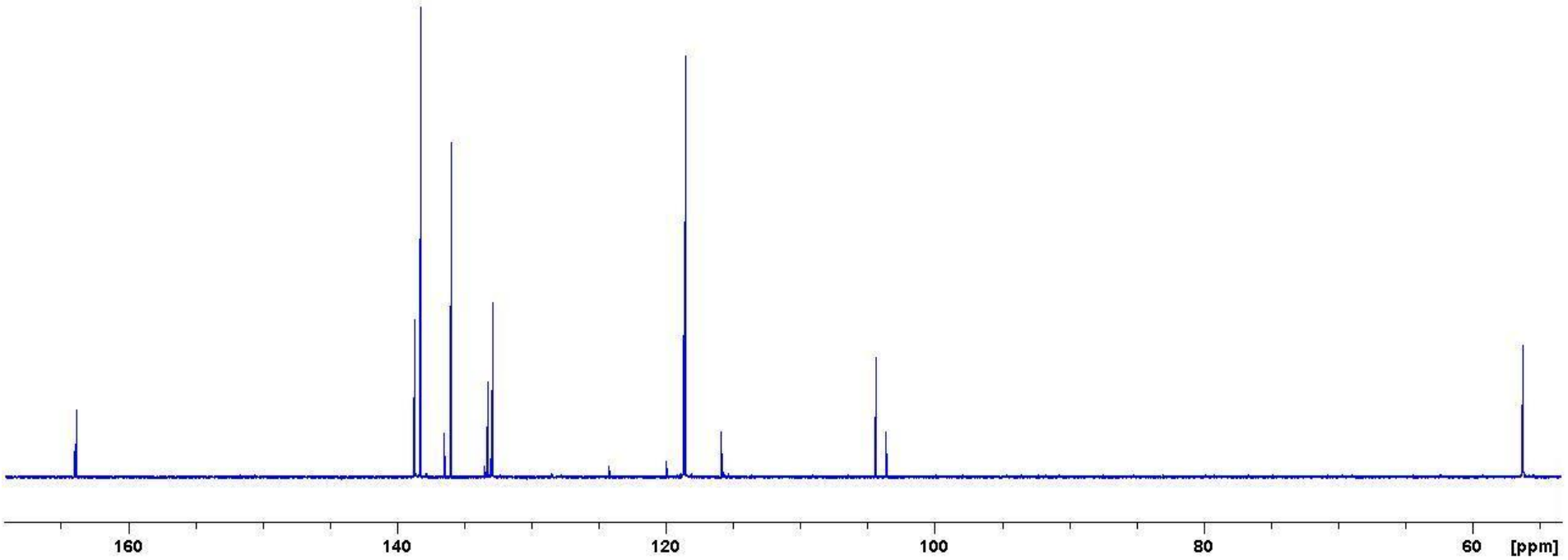


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry b of Table 1 (acetone-d_6)

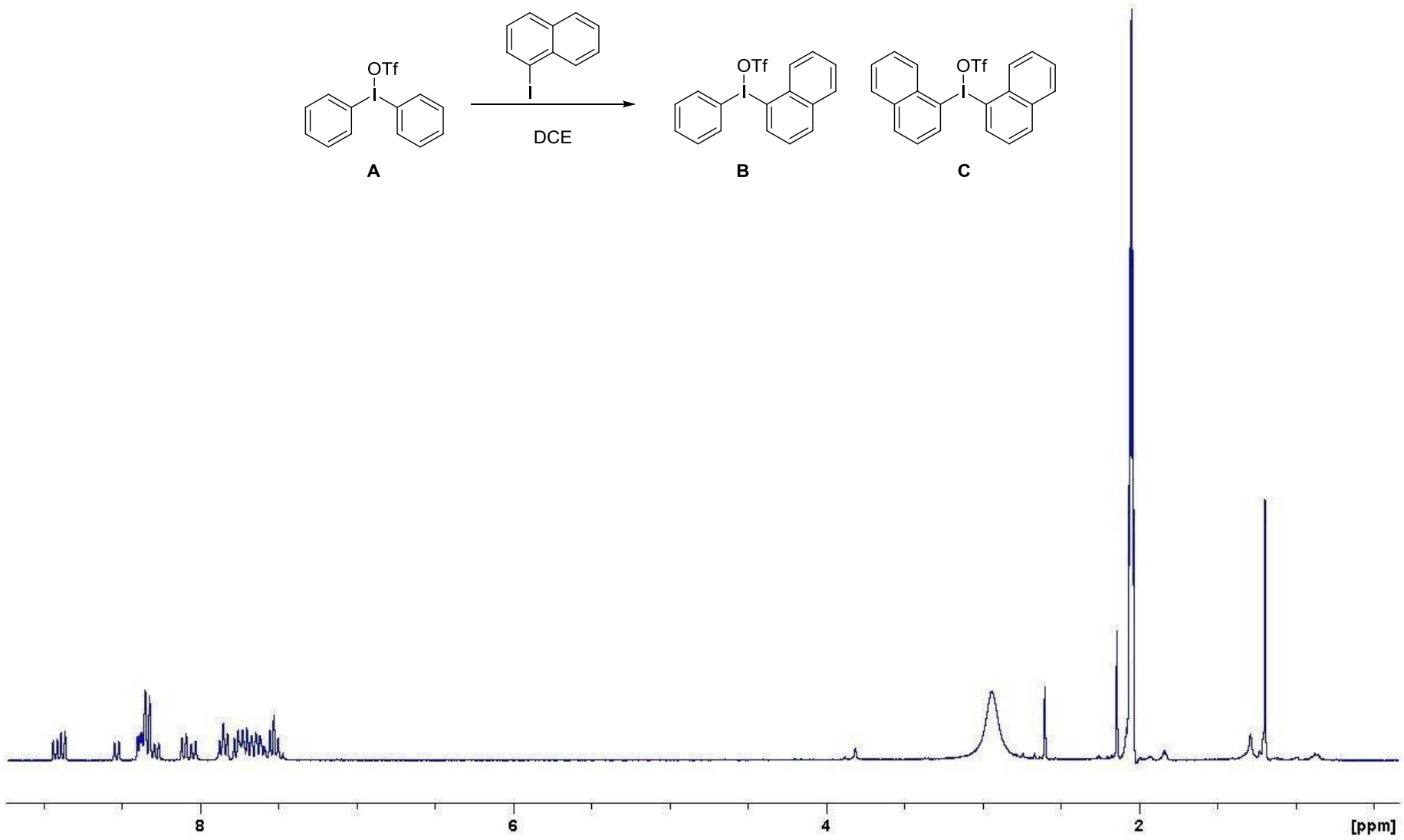


^{13}C NMR spectrum of the product mixture obtained in entry b of Table 1 (acetone-d_6)

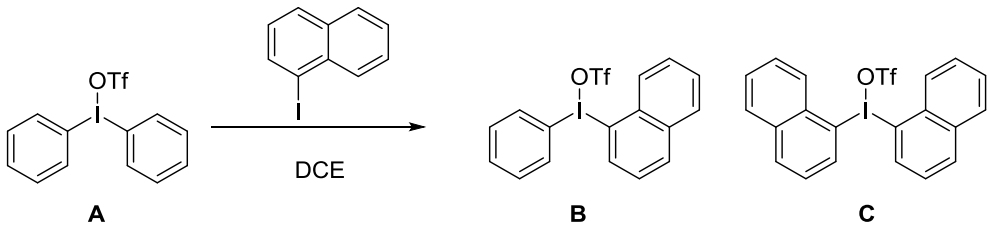


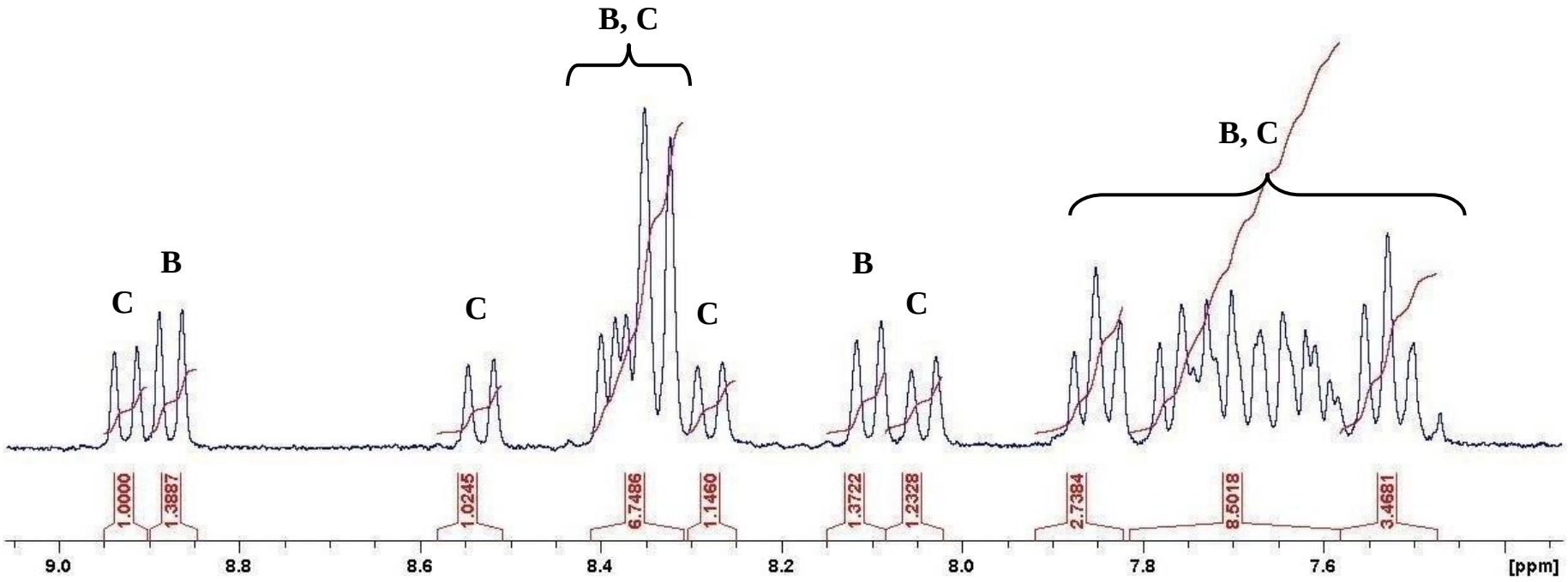


Expanded ^{13}C NMR spectrum of the product mixture obtained in entry b of Table 1 (acetone-d_6)

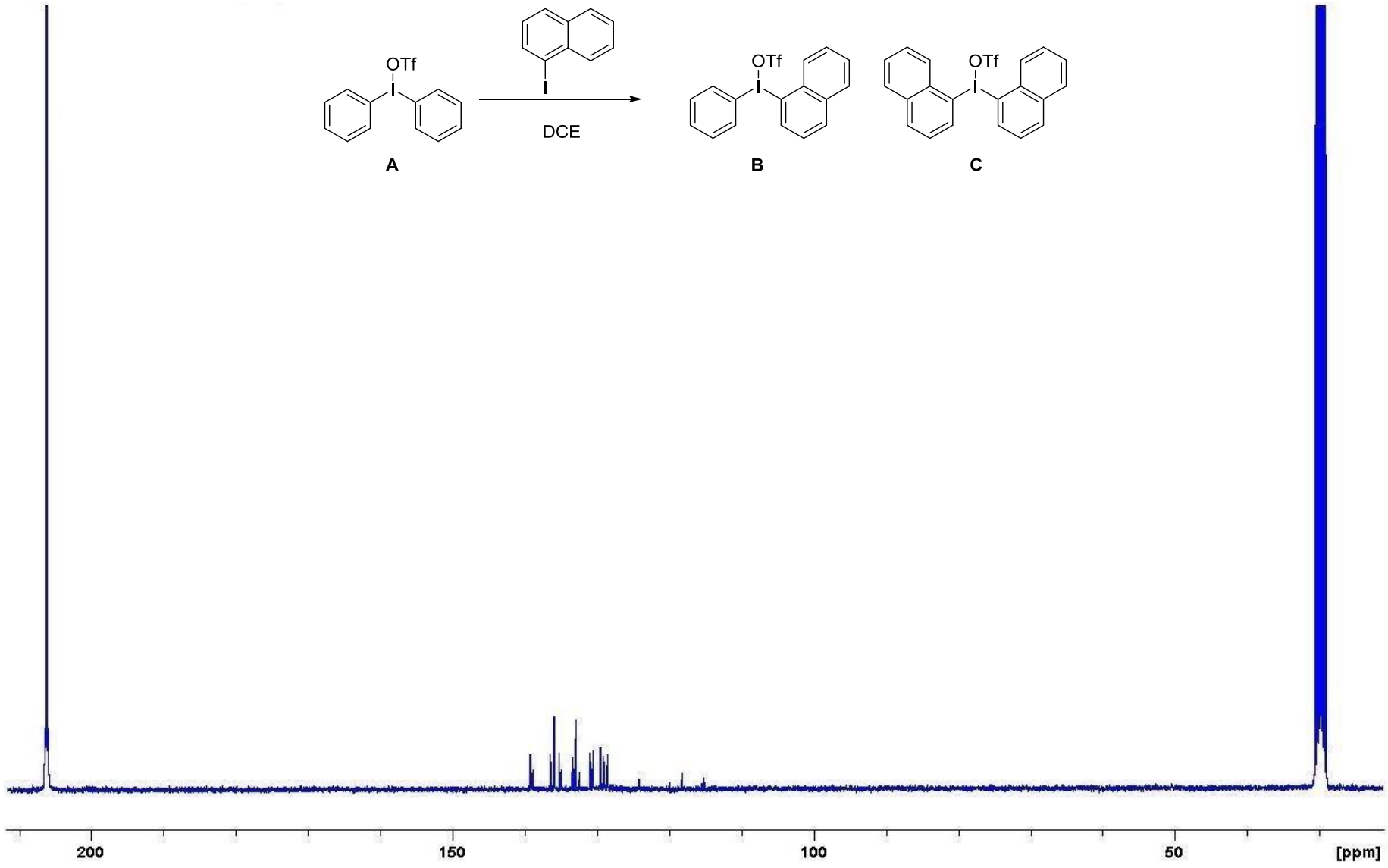


^{1}H NMR spectrum of the product mixture obtained in entry c of Table 1 (acetone-d_6)

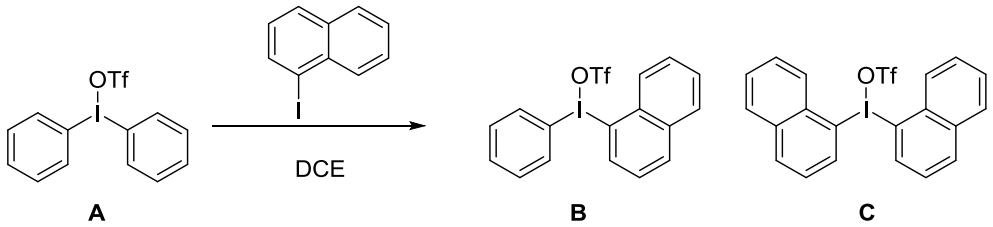


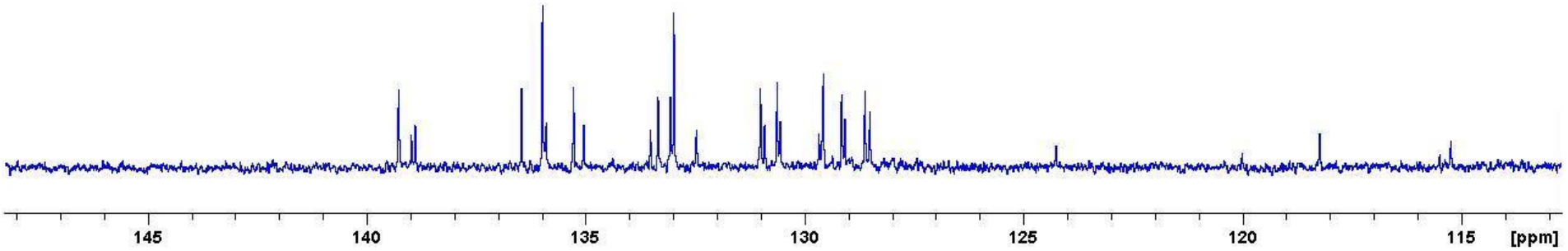


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry c of Table 1 (acetone-d_6)

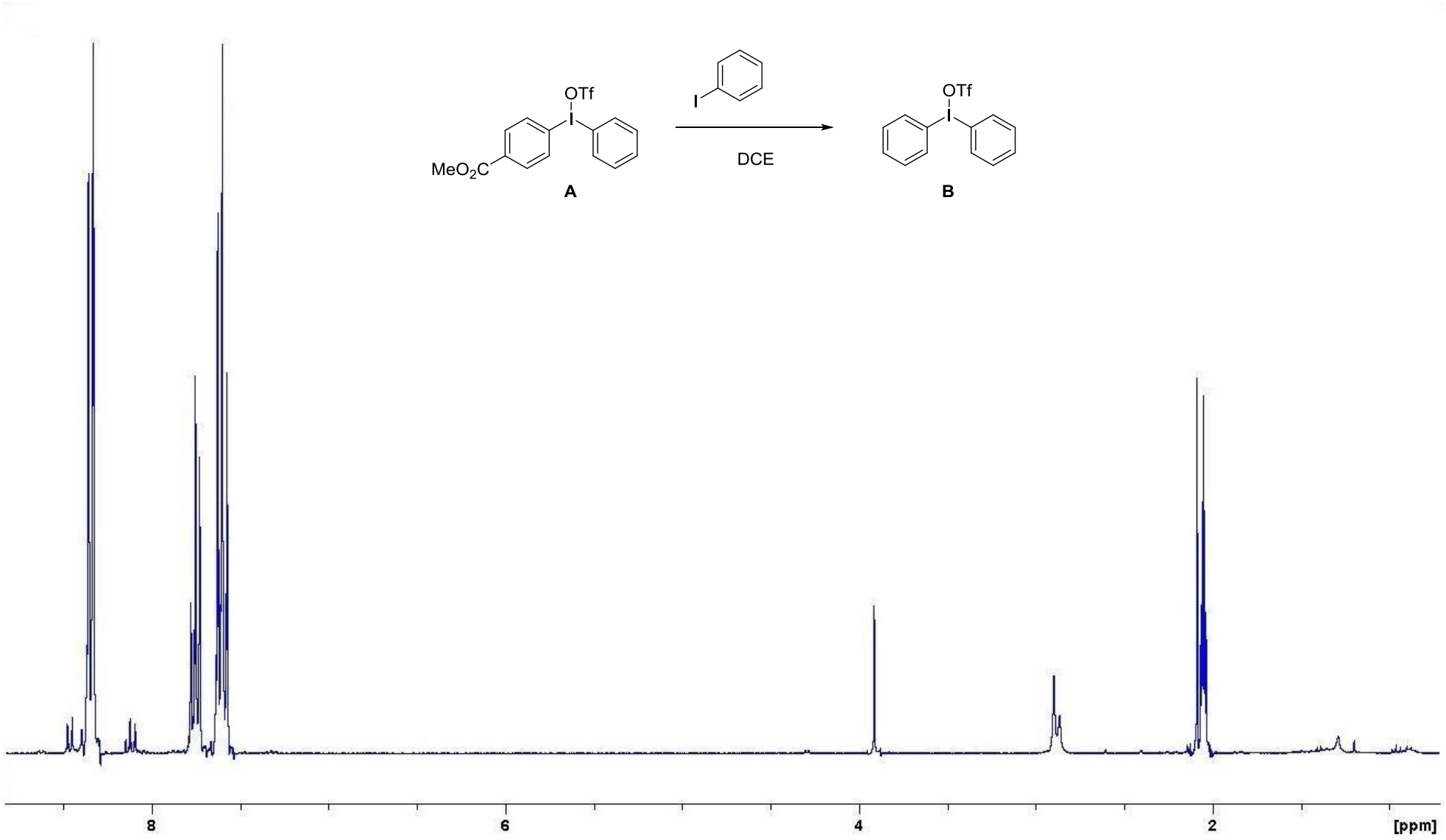


^{13}C NMR spectrum of the product mixture obtained in entry c of Table 1 (acetone-d_6)

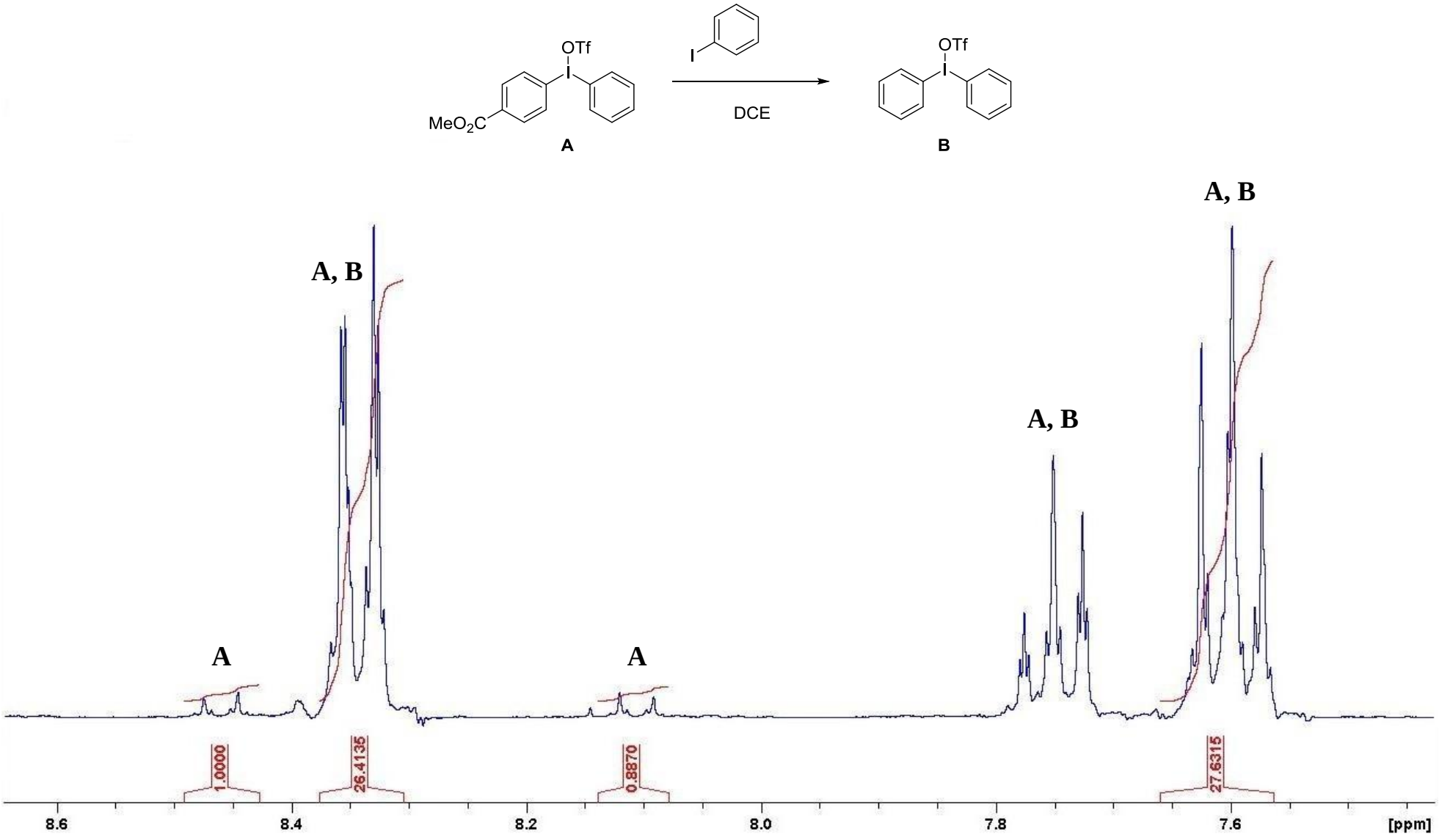




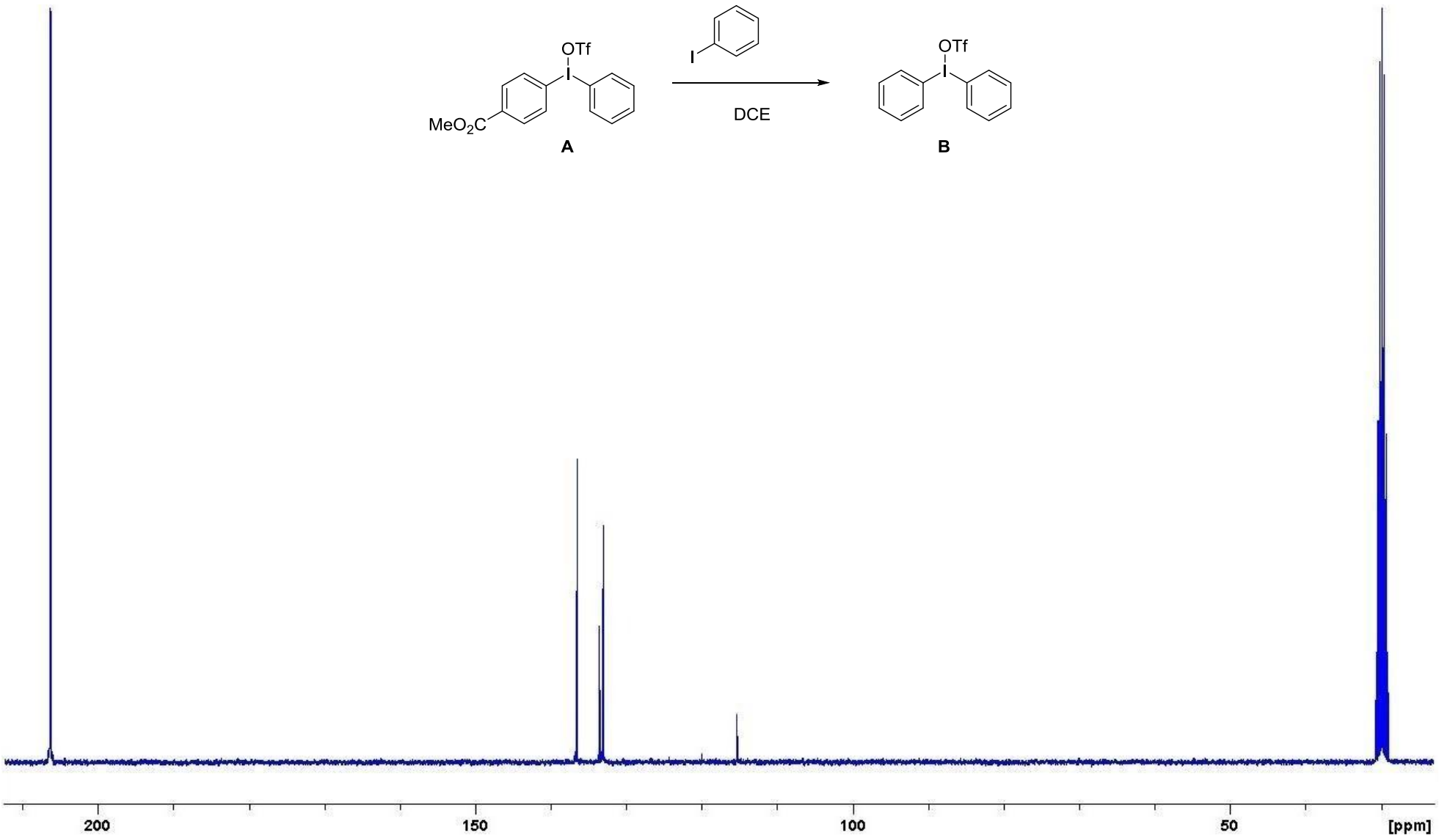
Expanded ^{13}C NMR spectrum of the product mixture obtained in entry c of Table 1 (acetone-d_6)



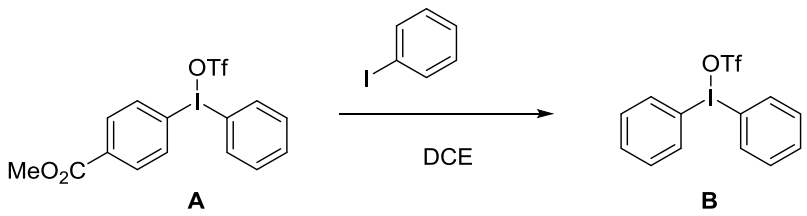
^{1}H NMR spectrum of the product mixture obtained in entry a of Table 2 (acetone-d_6)

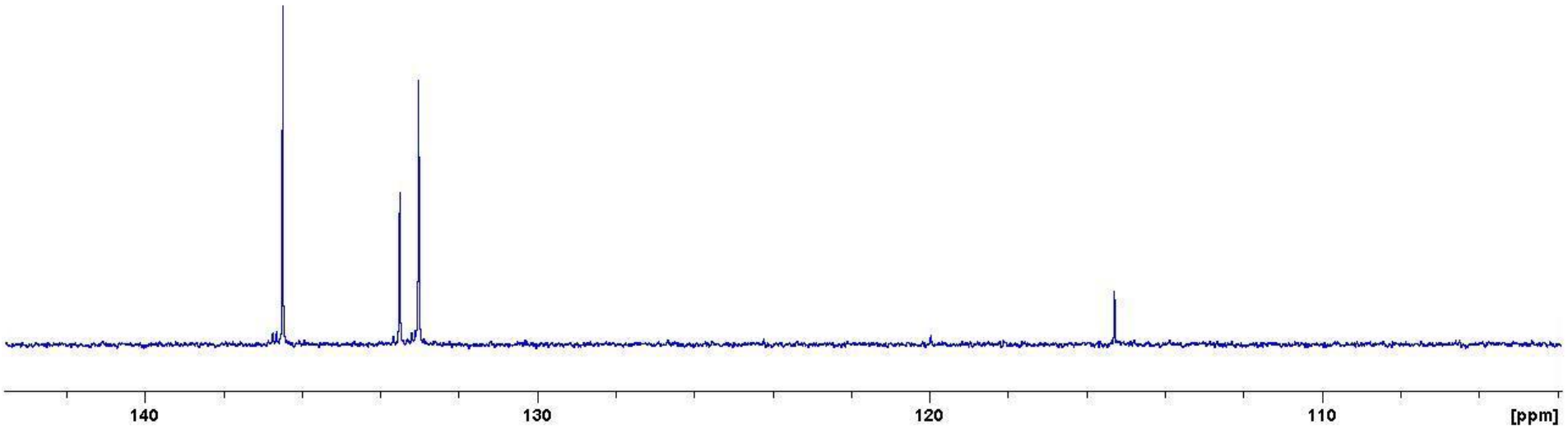


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry a of Table 2 (acetone-d_6)

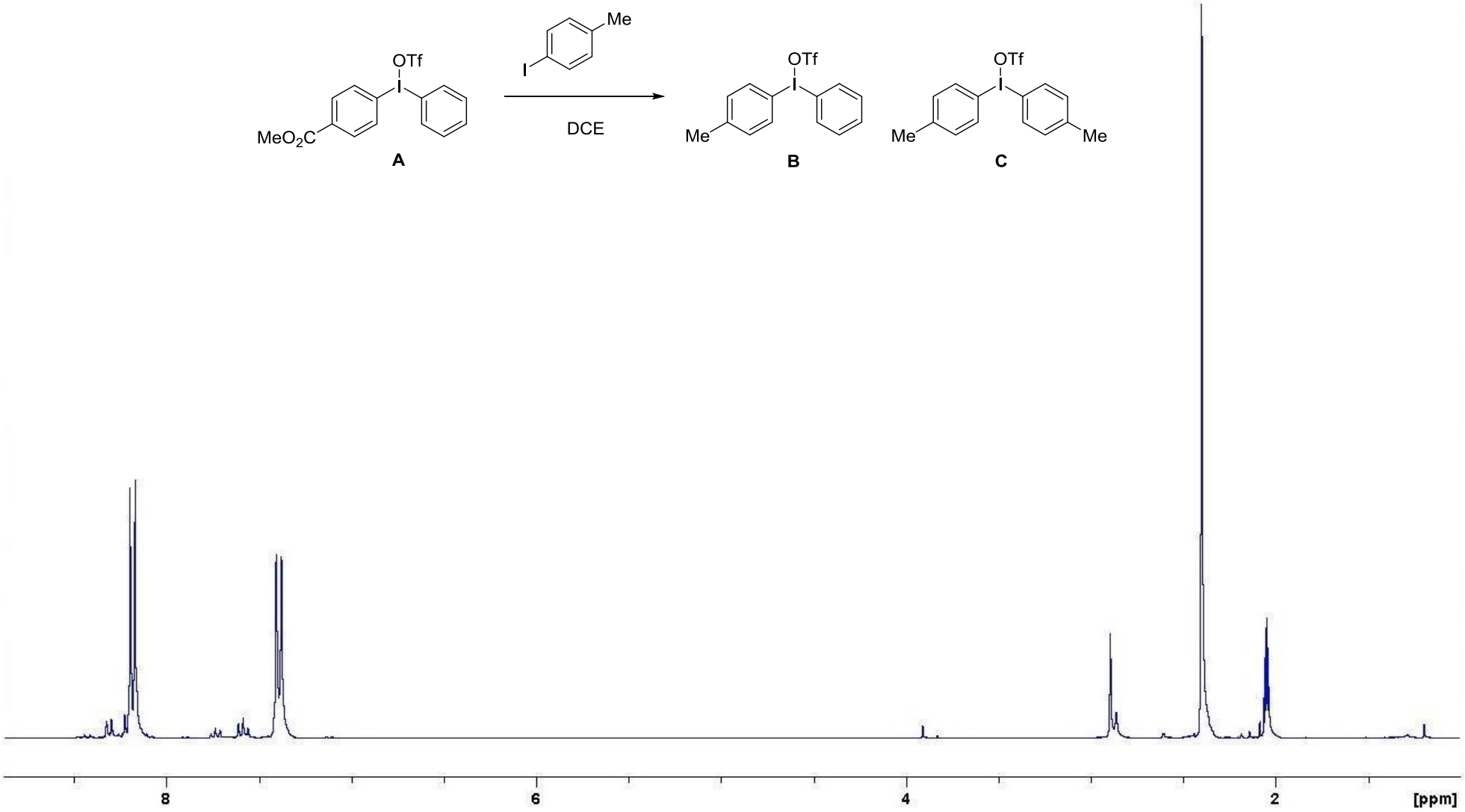


^{13}C NMR spectrum of the product mixture obtained in entry a of Table 2 (acetone-d_6)

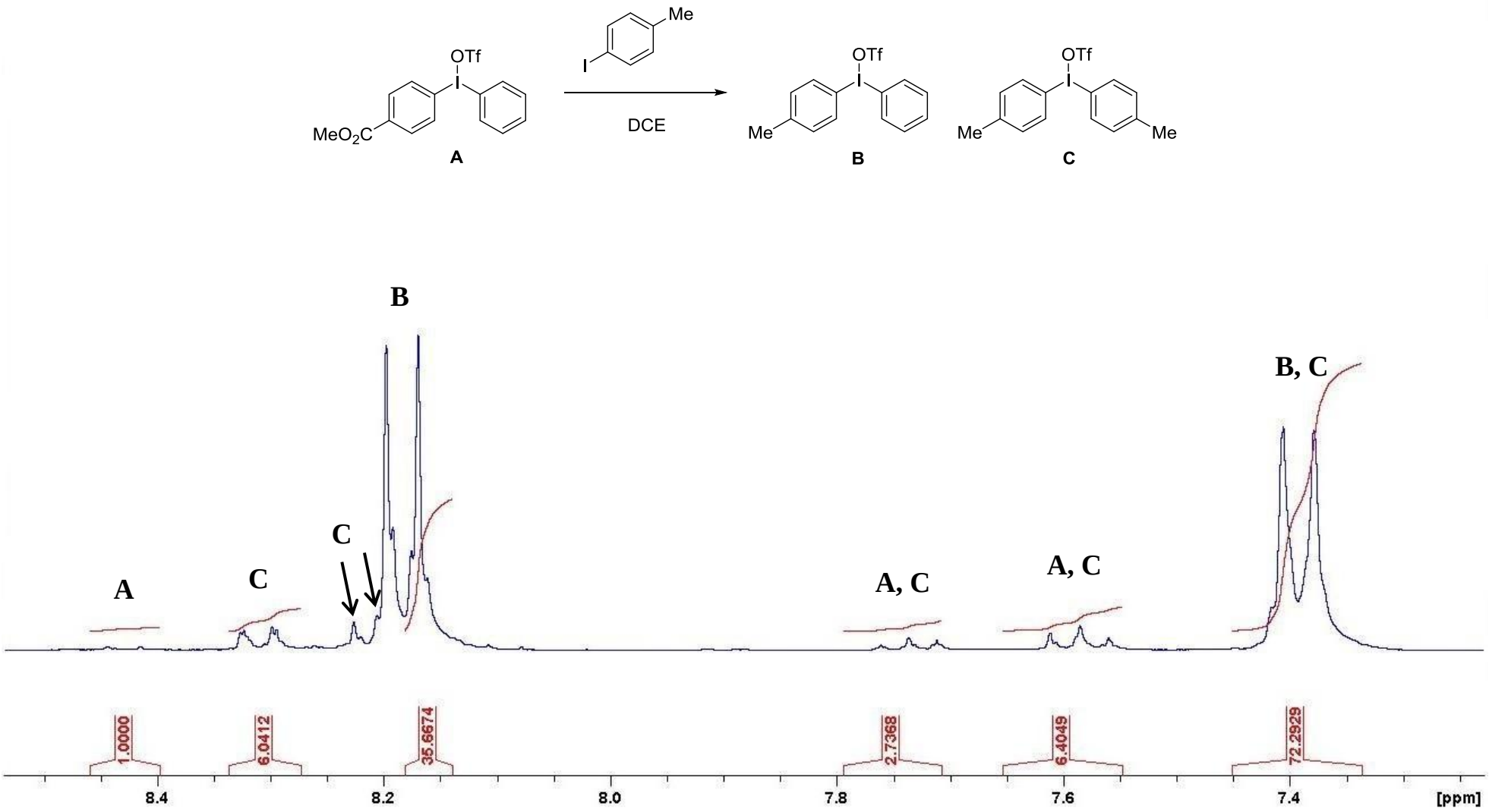




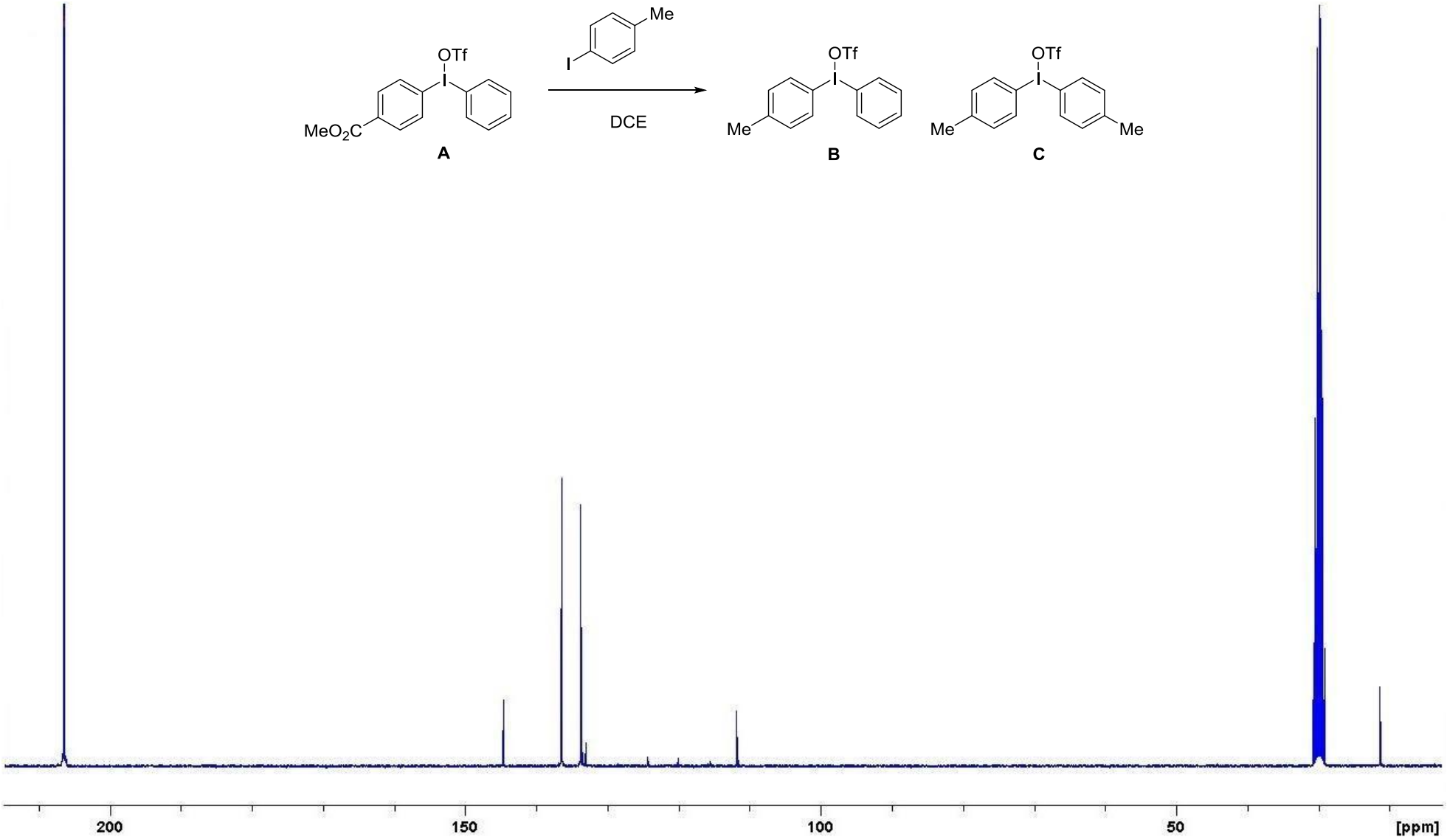
Expanded ^{13}C NMR spectrum of the product mixture obtained in entry a of Table 2 (acetone-d_6)



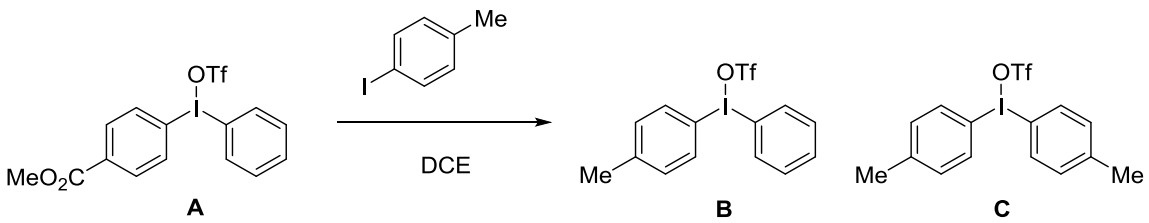
^{1}H NMR spectrum of the product mixture obtained in entry b of Table 2 (acetone-d_6)

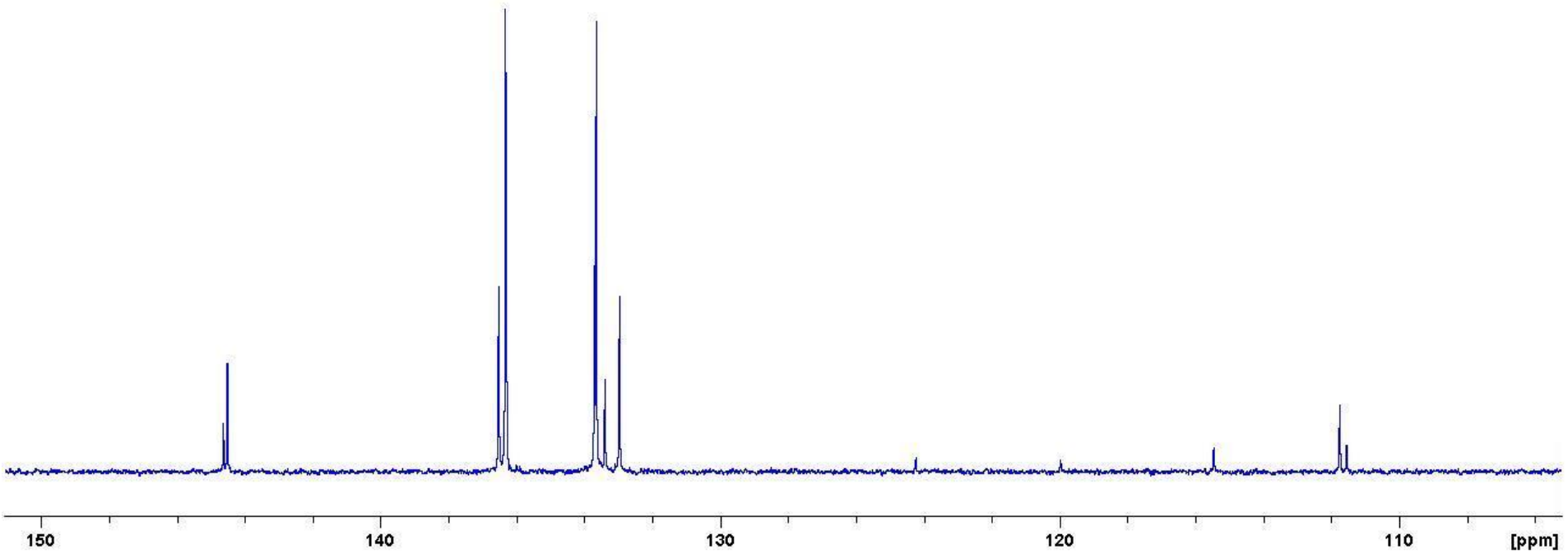


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry b of Table 2 (acetone-d_6)

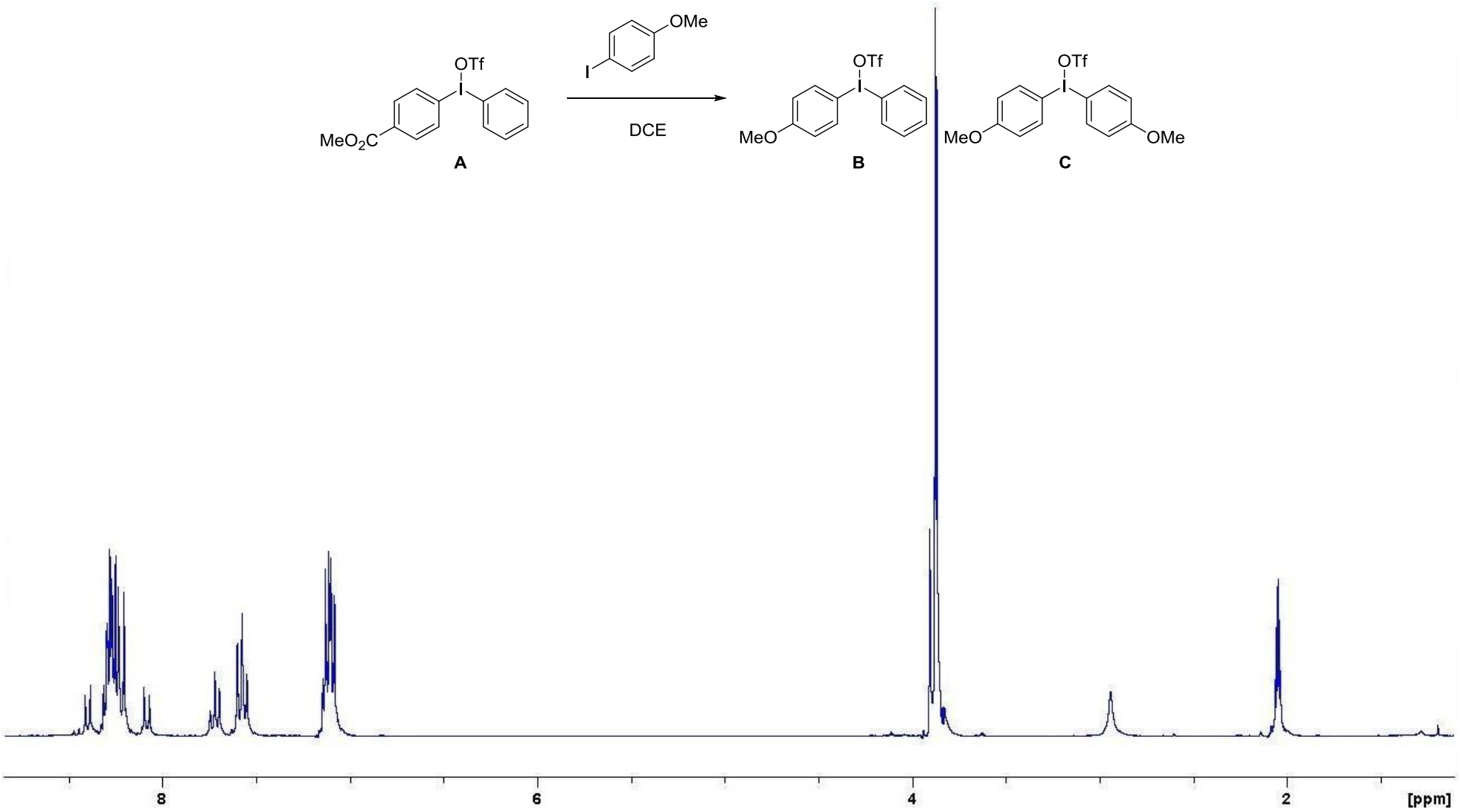


^{13}C NMR spectrum of the product mixture obtained in entry b of Table 2 (acetone-d_6)

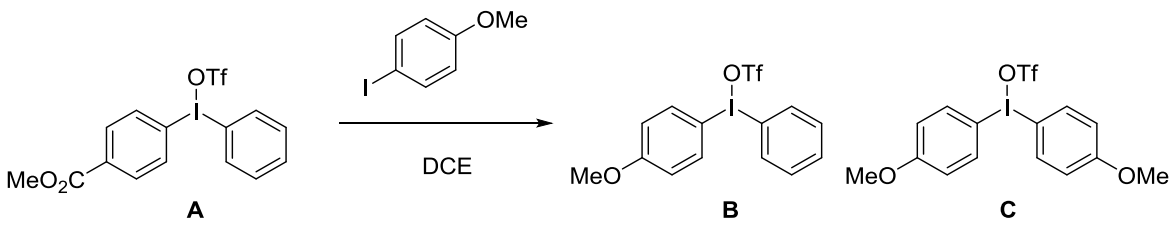


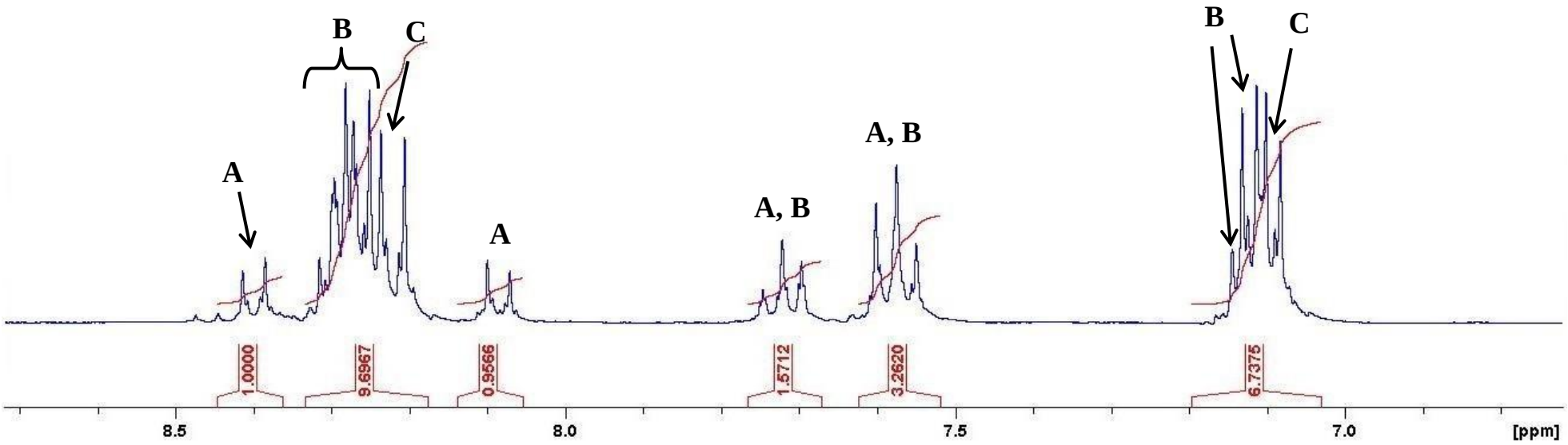


Expanded ^{13}C NMR spectrum of the product mixture obtained in entry b of Table 2 (acetone-d_6)

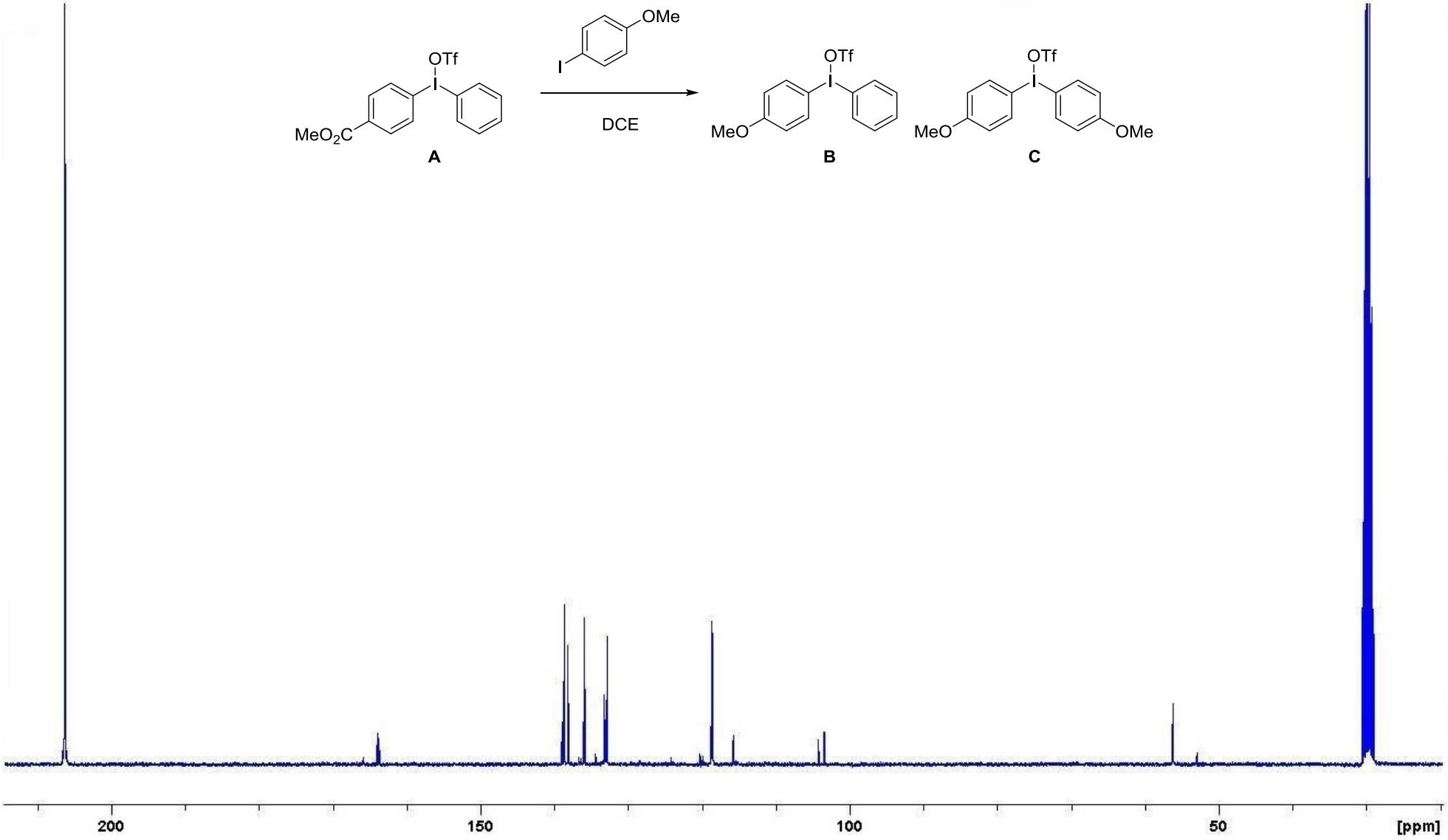


^{1}H NMR spectrum of the product mixture obtained in entry c of Table 2 (acetone-d_6)

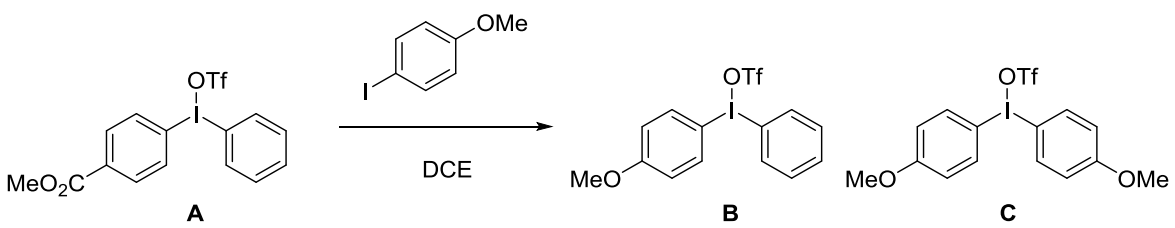


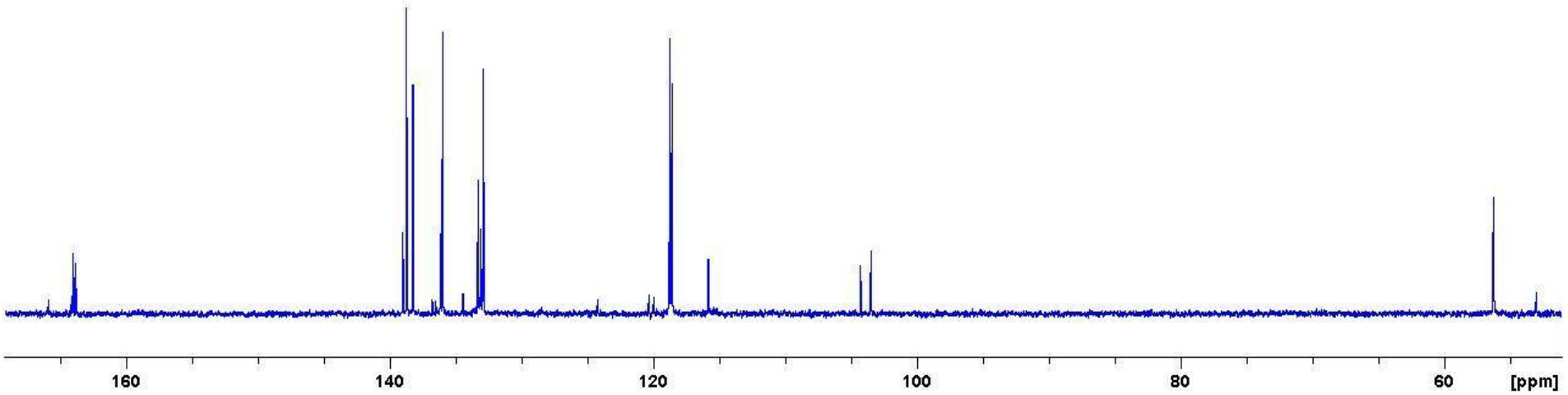


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry c of Table 2 (acetone-d_6)

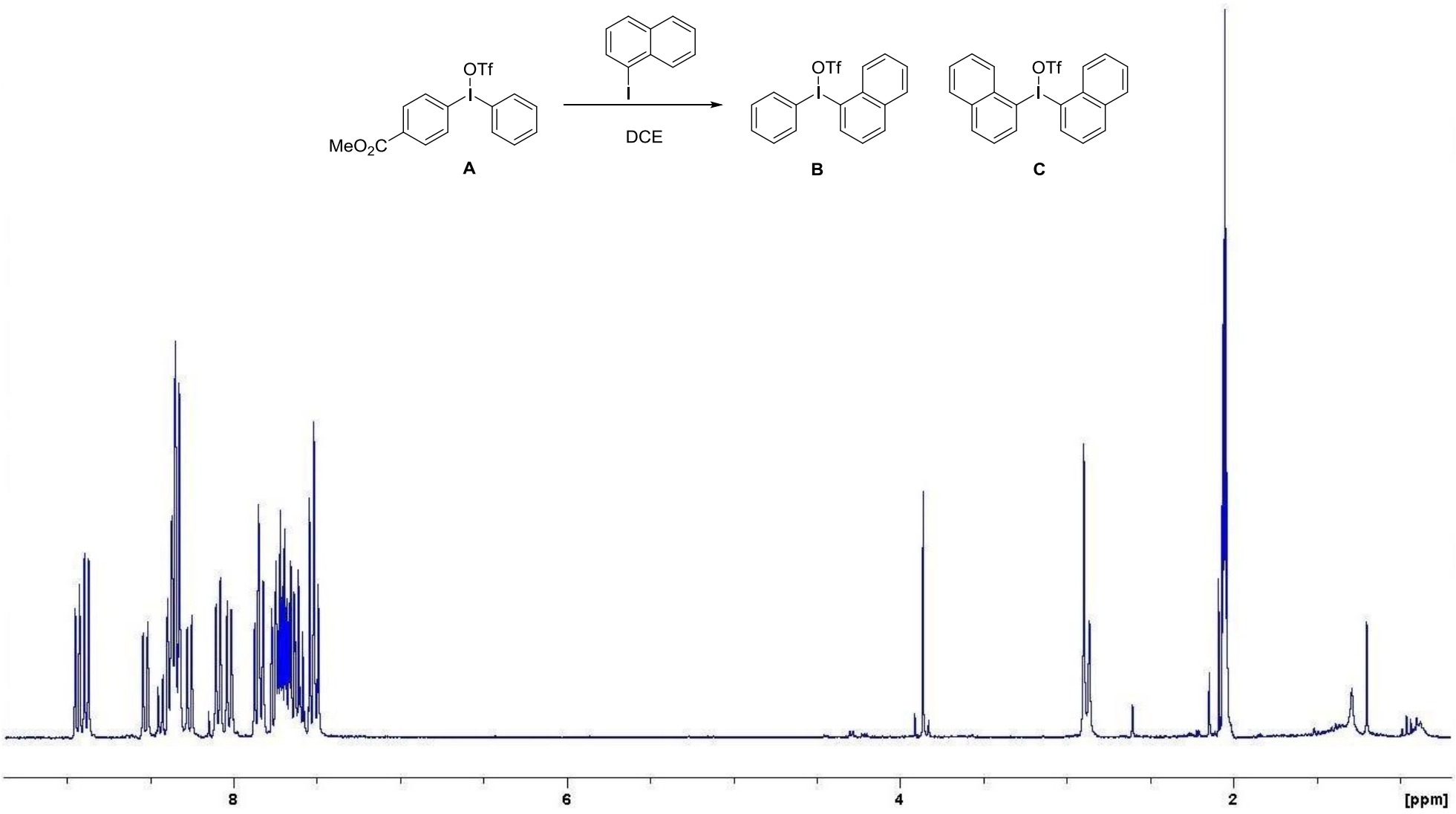


^{13}C NMR spectrum of the product mixture obtained in entry c of Table 2 (acetone-d_6)

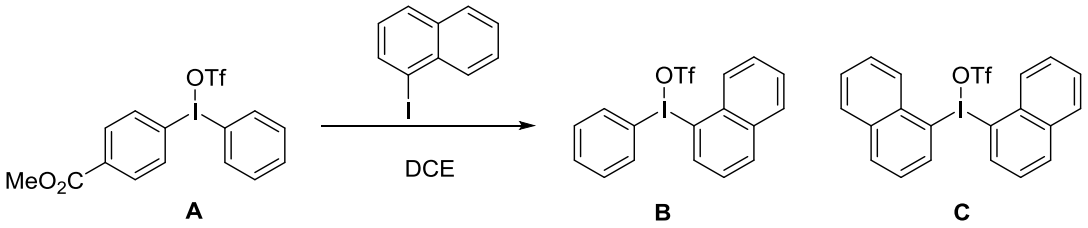


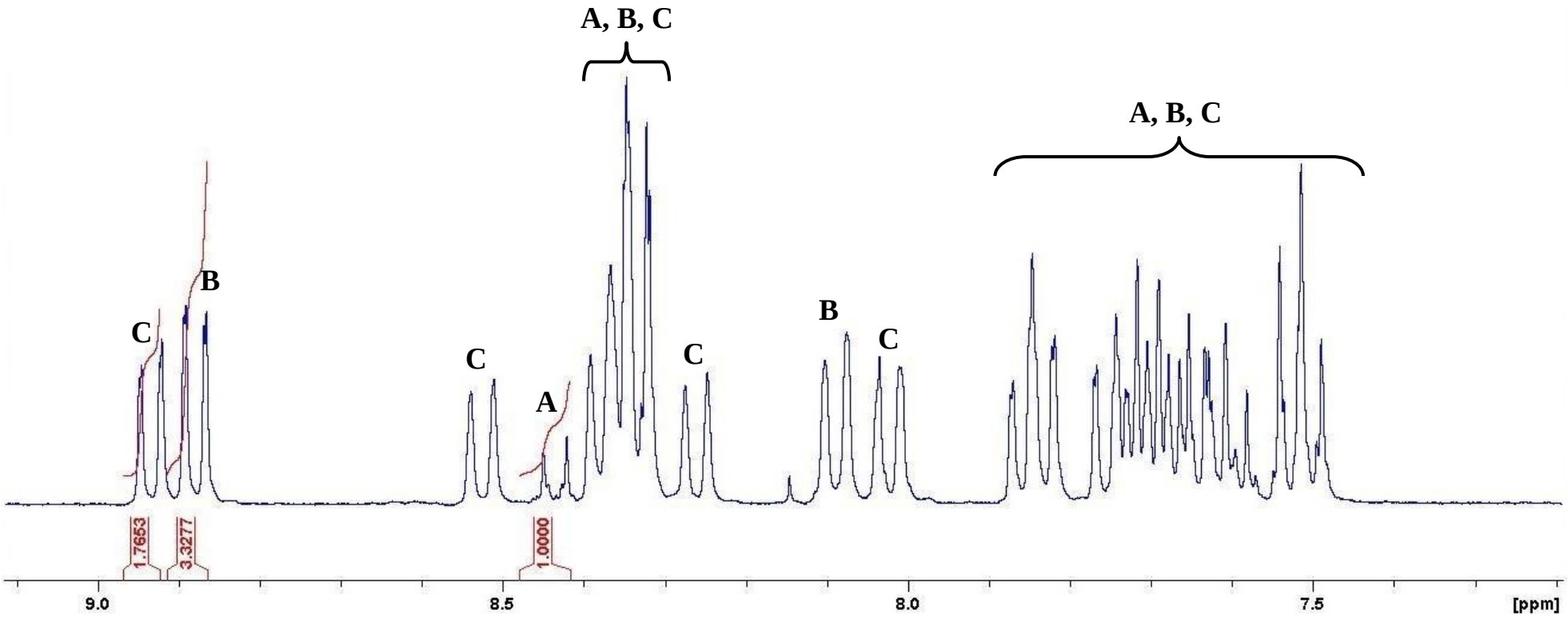


Expanded ^{13}C NMR spectrum of the product mixture obtained in entry c of Table 2 (acetone-d_6)

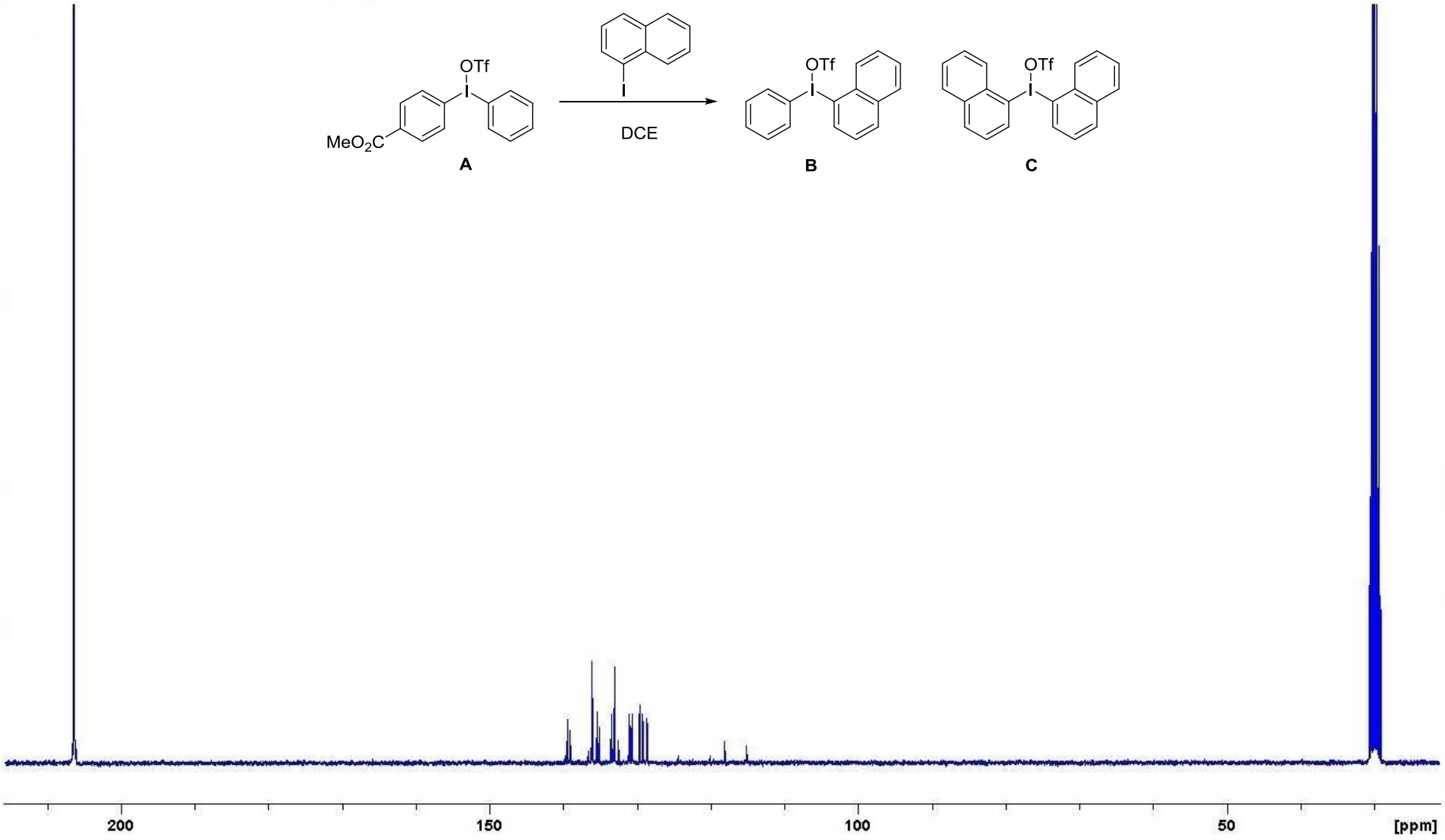


^{1}H NMR spectrum of the product mixture obtained in entry d of Table 2 (acetone-d_6)

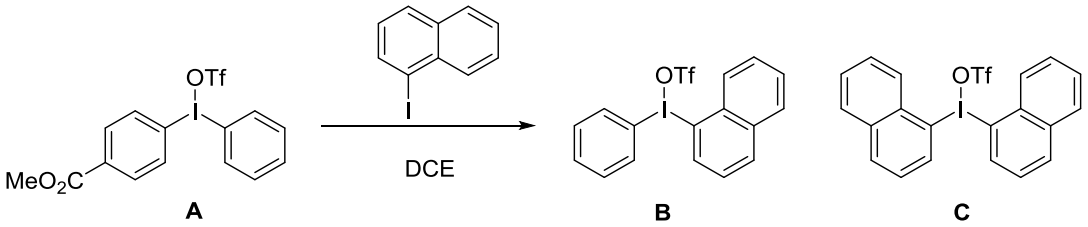


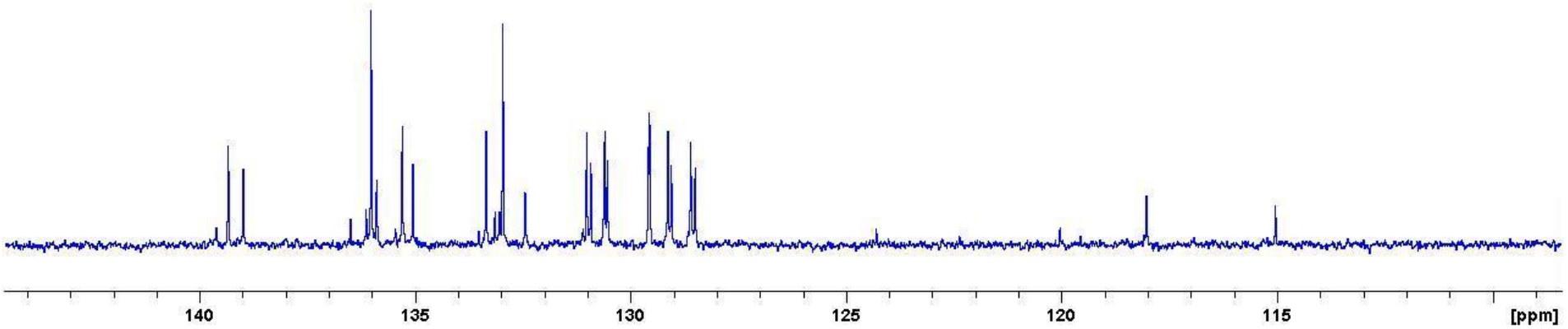


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry d of Table 2 (acetone-d_6)

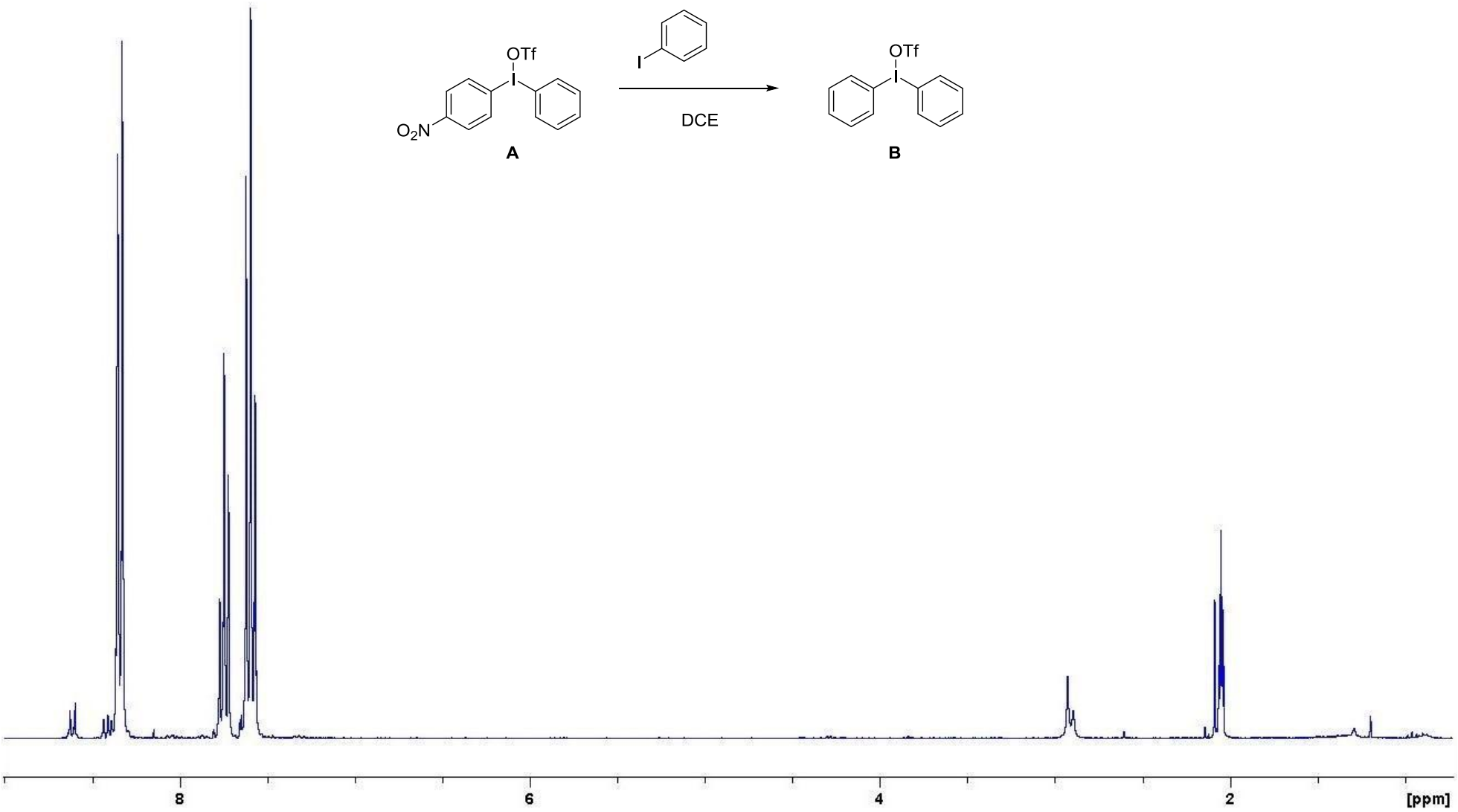


^{13}C NMR spectrum of the product mixture obtained in entry d of Table 2 (acetone-d_6)

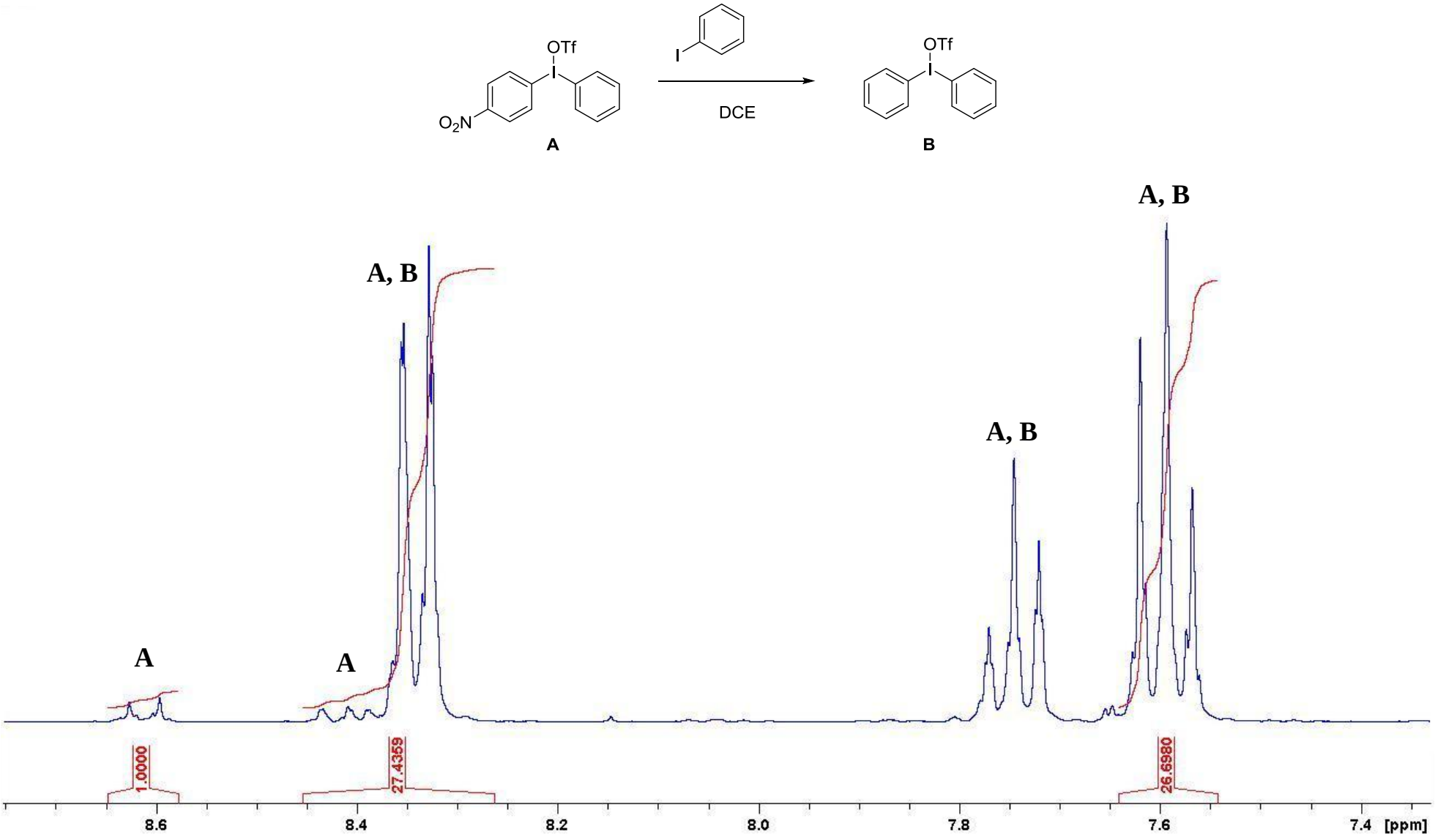




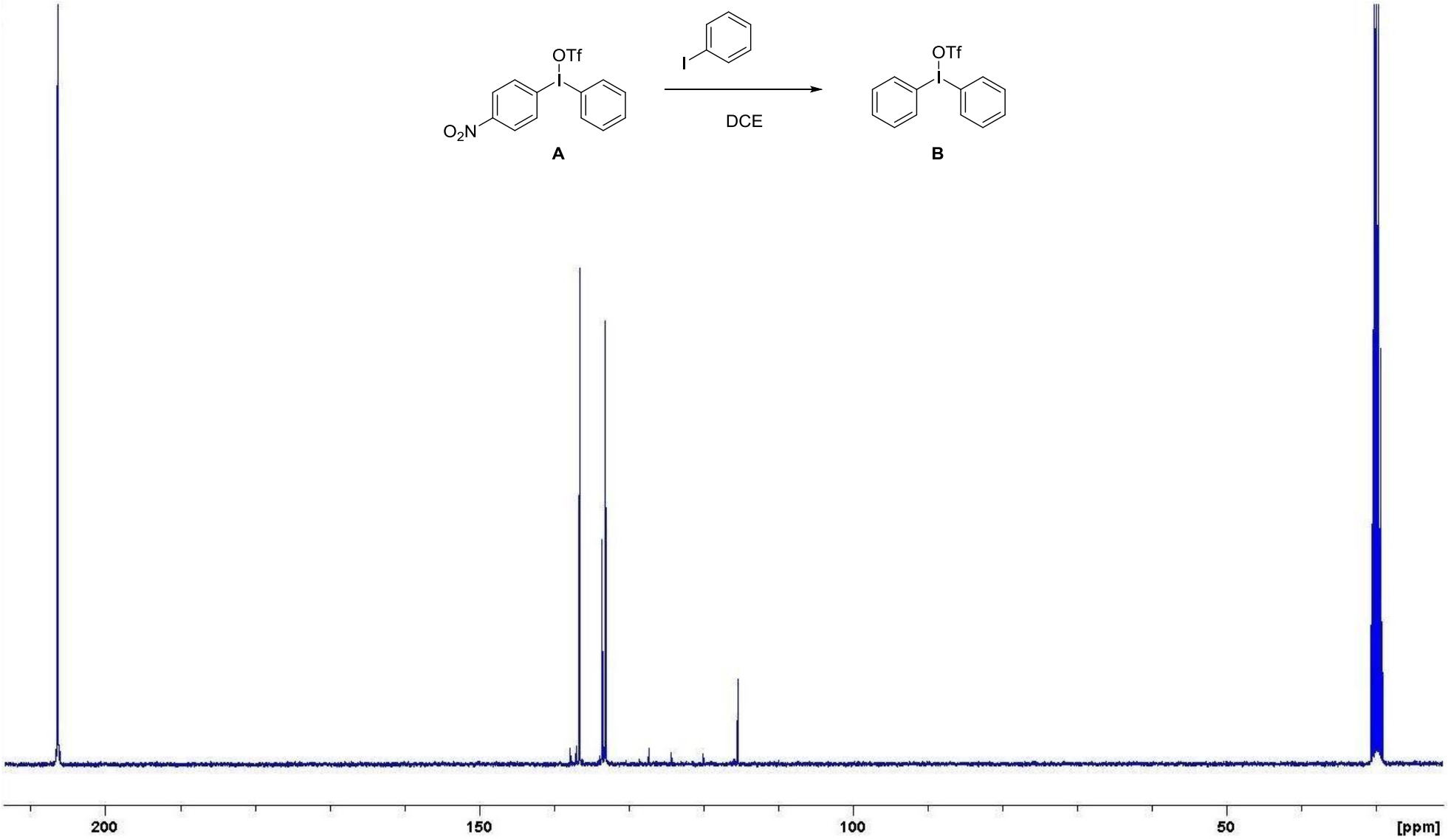
Expanded ^{13}C NMR spectrum of the product mixture obtained in entry d of Table 2 (acetone-d_6)



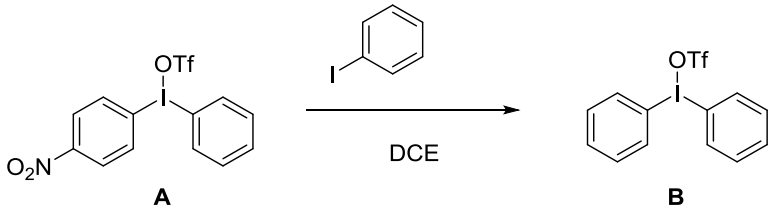
^{1}H NMR spectrum of the product mixture obtained in entry a of Table 3 (acetone-d_6)

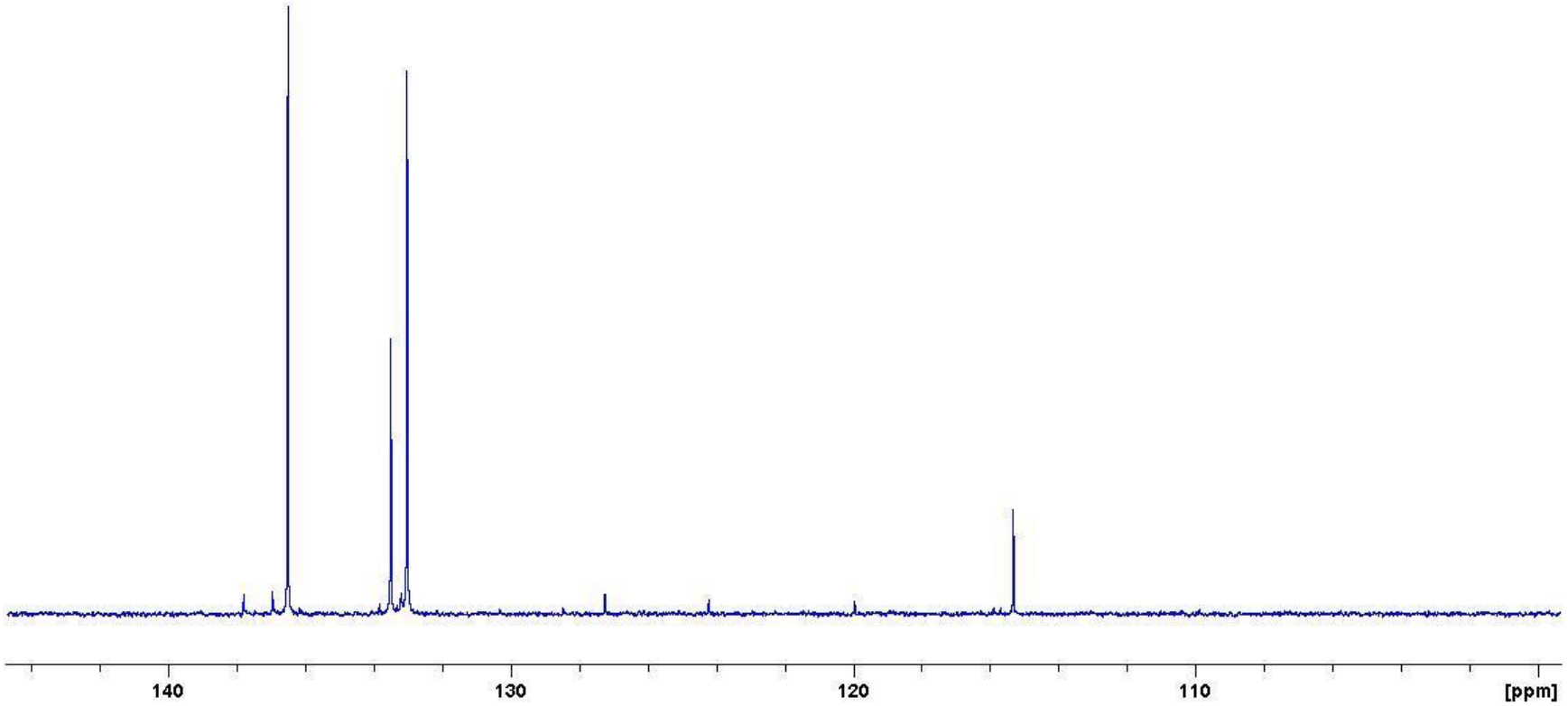


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry a of Table 3 (acetone-d_6)

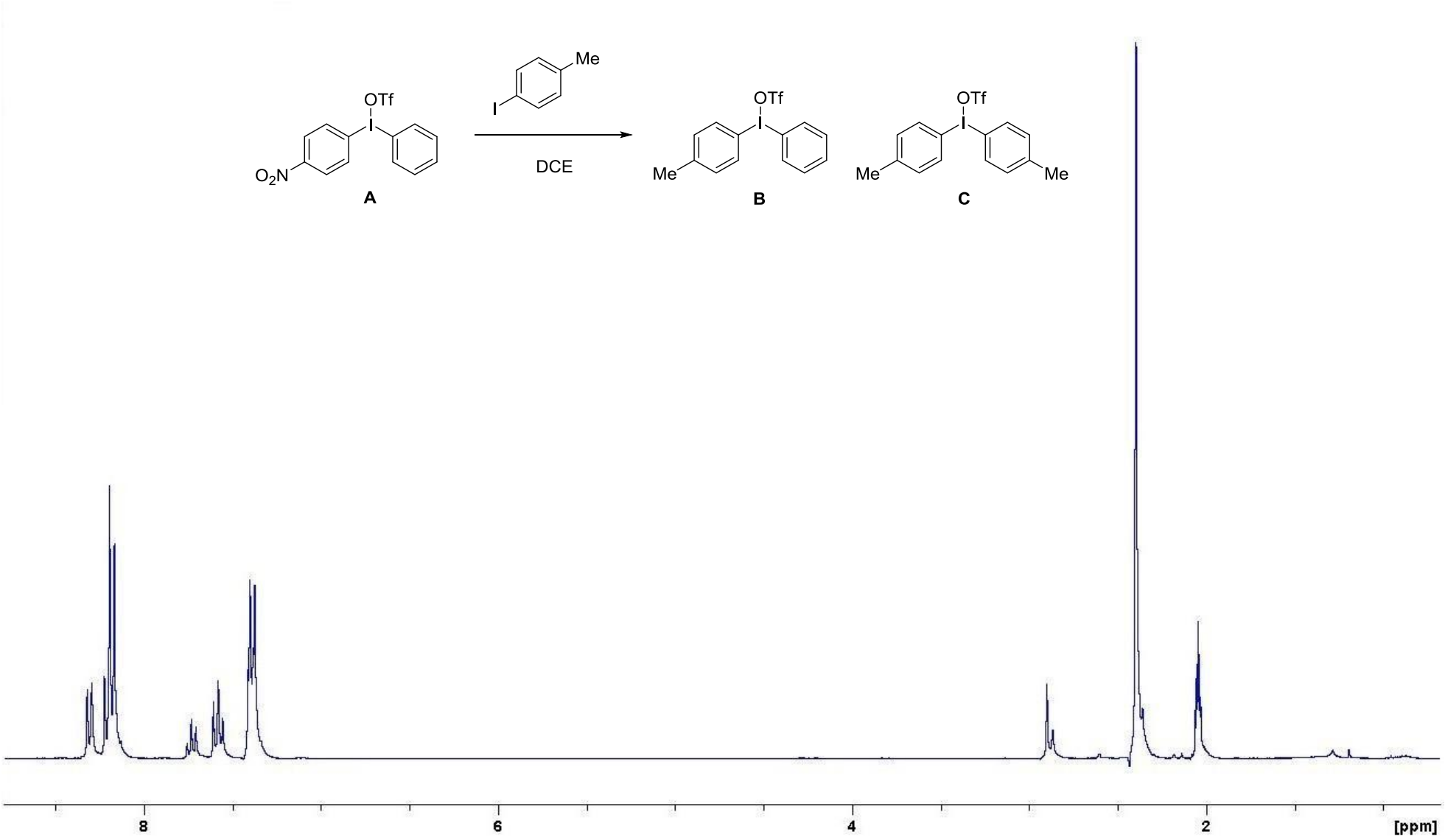


^{13}C NMR spectrum of the product mixture obtained in entry a of Table 3 (acetone-d_6)

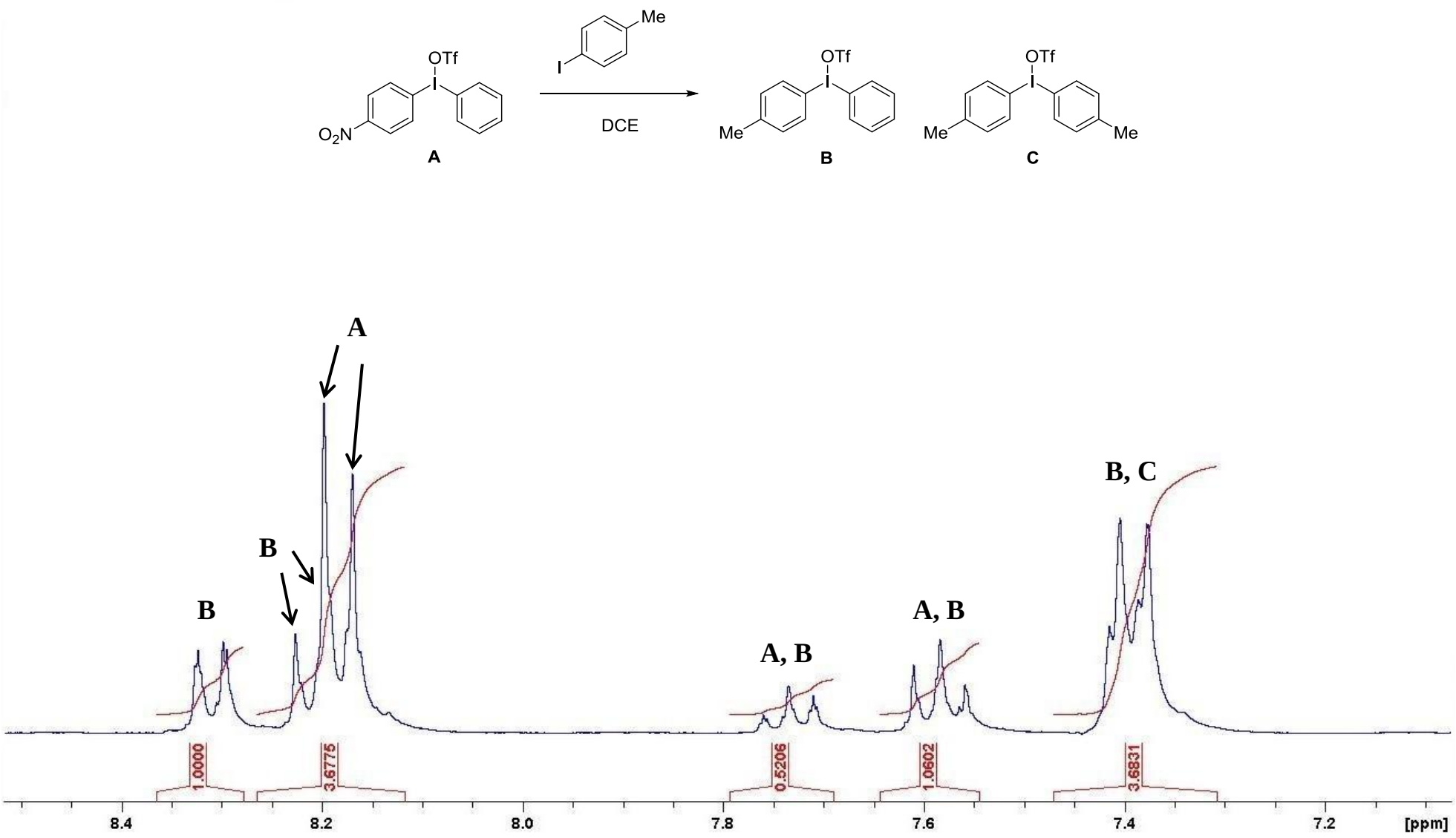




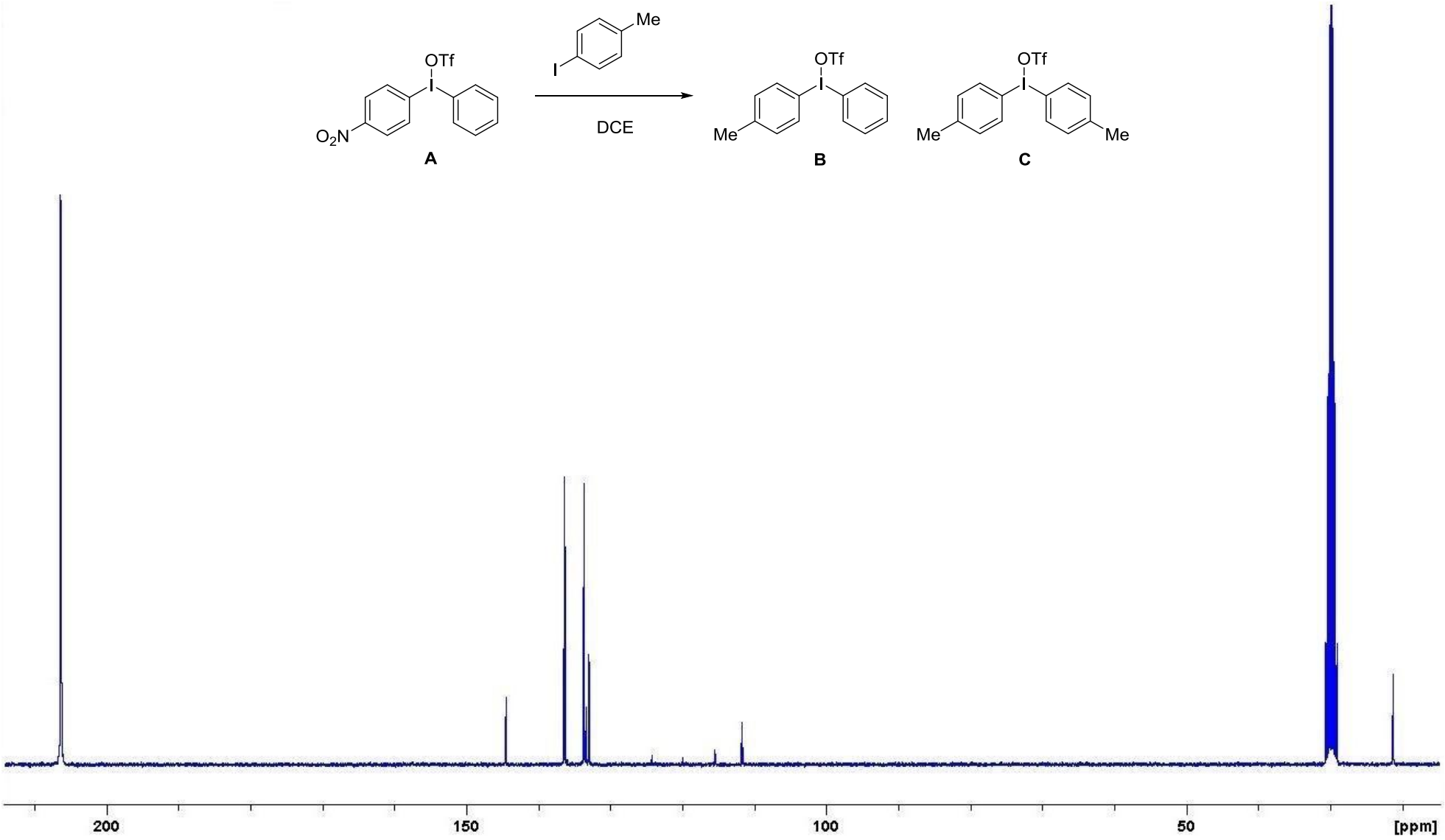
Expanded ^{13}C NMR spectrum of the product mixture obtained in entry a of Table 3 (acetone-d_6)



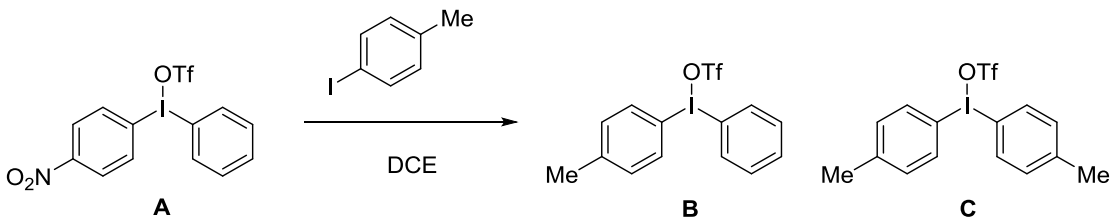
^{1}H NMR spectrum of the product mixture obtained in entry b of Table 3 (acetone-d_6)

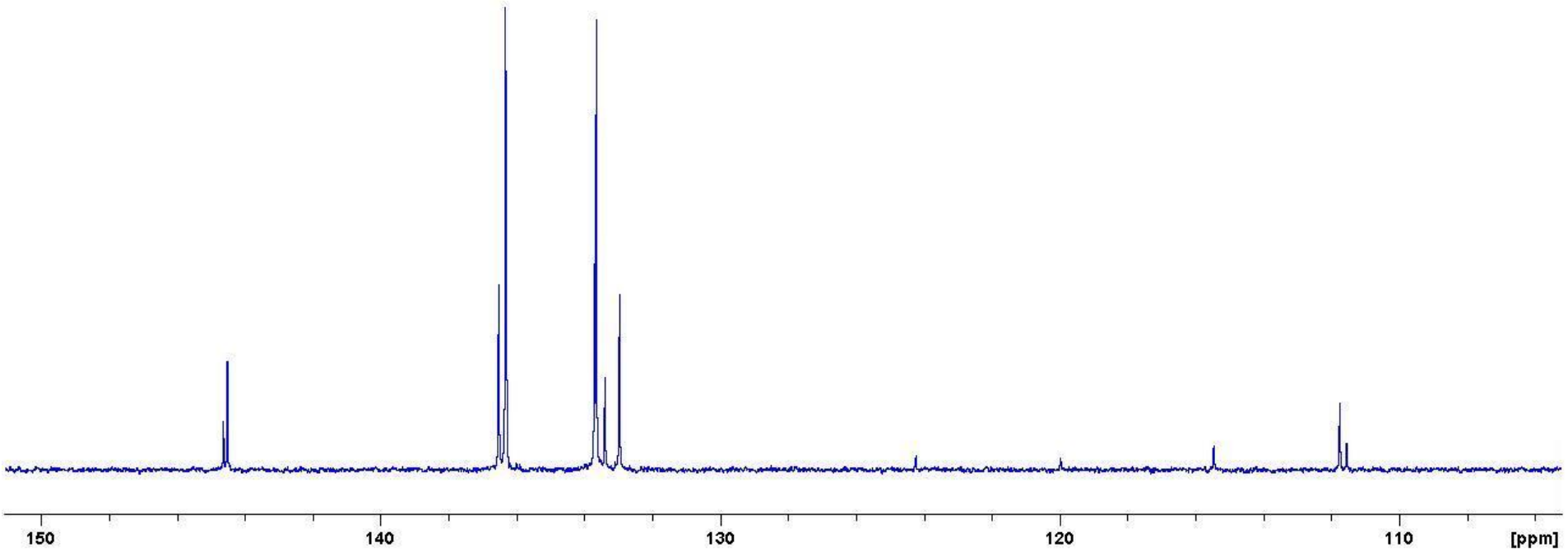


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry b of Table 3 (acetone-d_6)

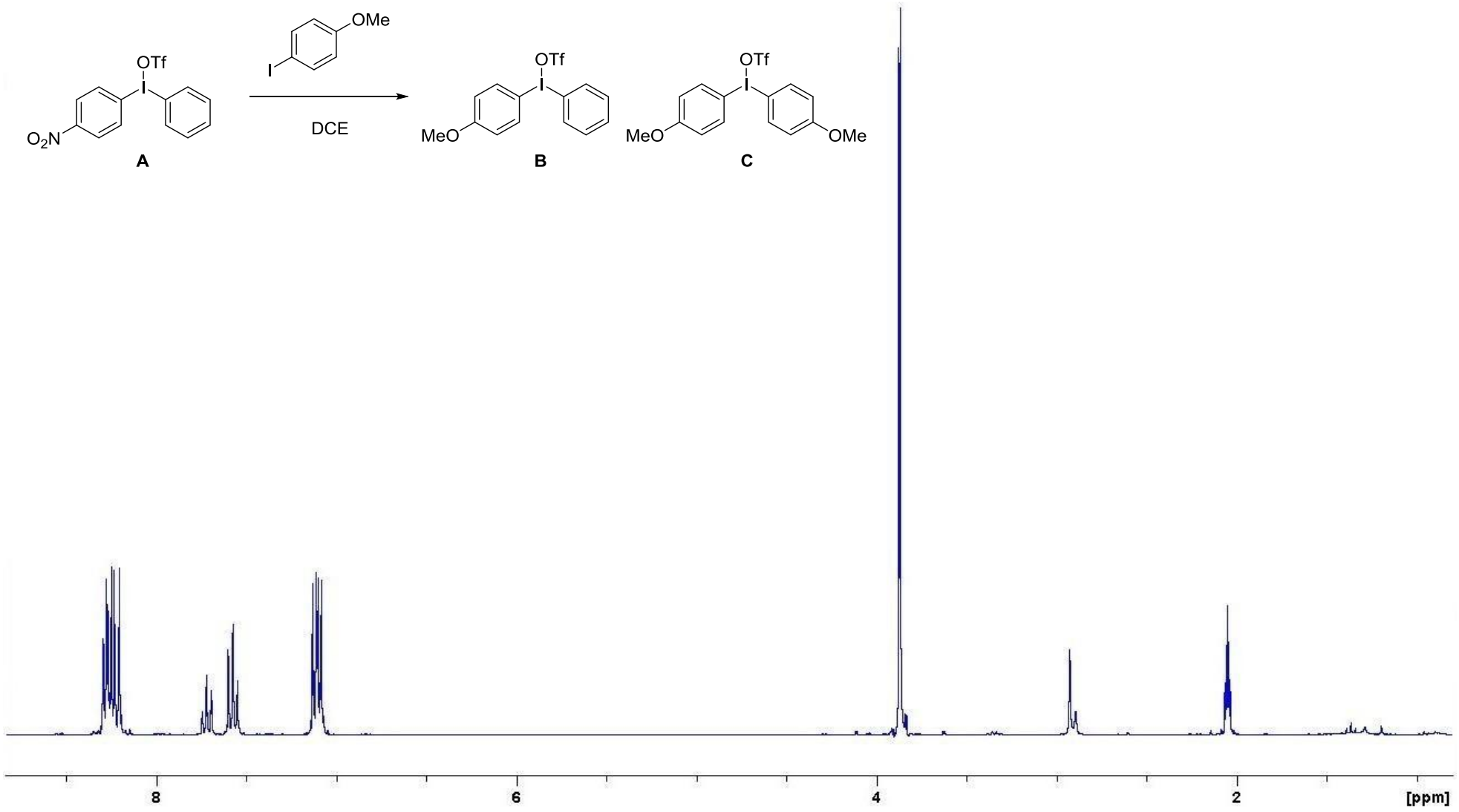


^{13}C NMR spectrum of the product mixture obtained in entry b of Table 3 (acetone-d_6)

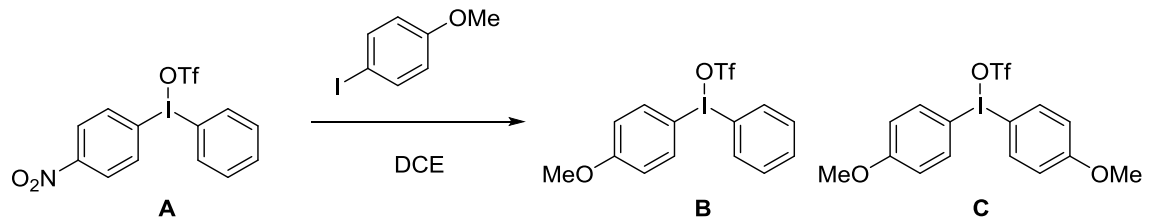


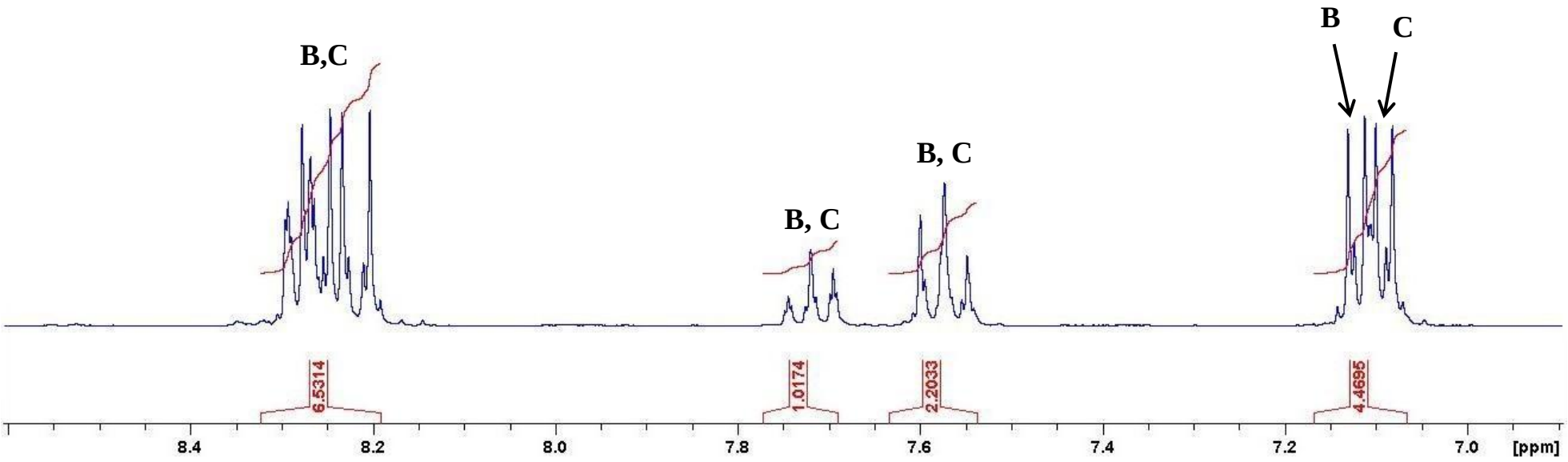


Expanded ^{13}C NMR spectrum of the product mixture obtained in entry b of Table 3 (acetone-d_6)

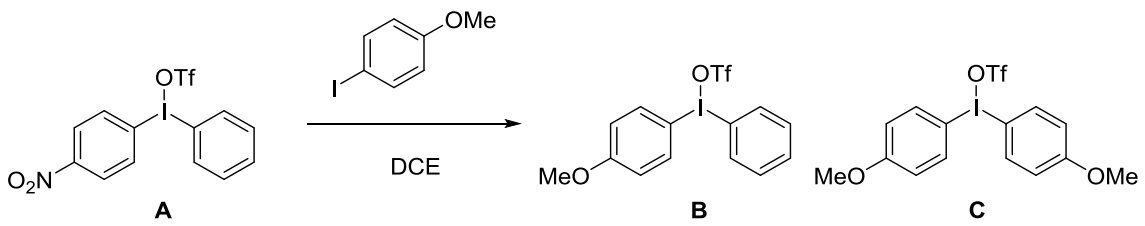


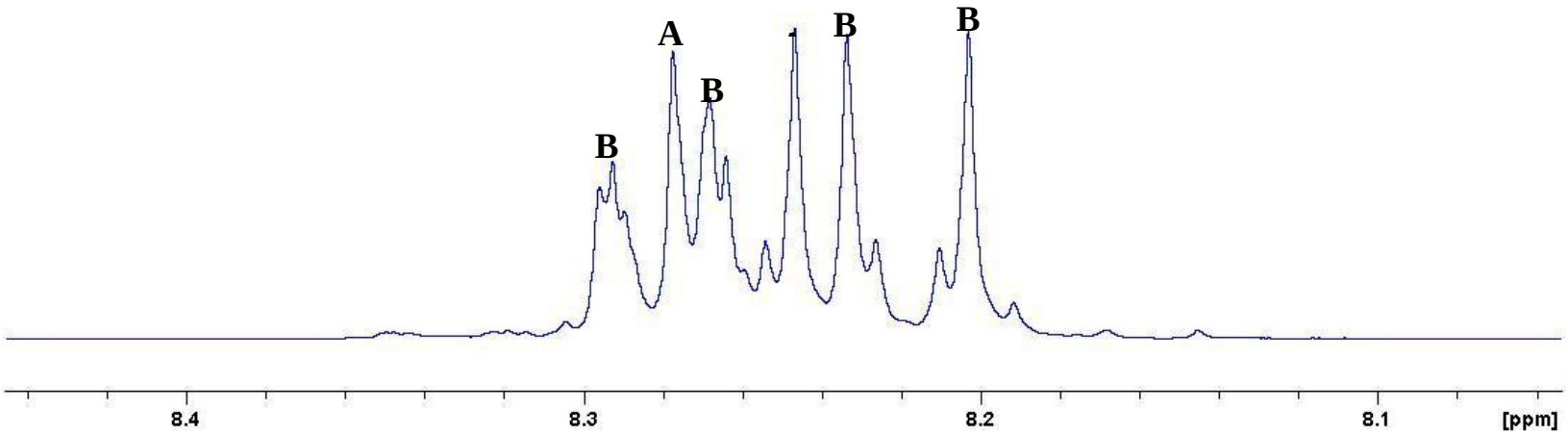
^{1}H NMR spectrum of the product mixture obtained in entry c of Table 3 (acetone-d_6)



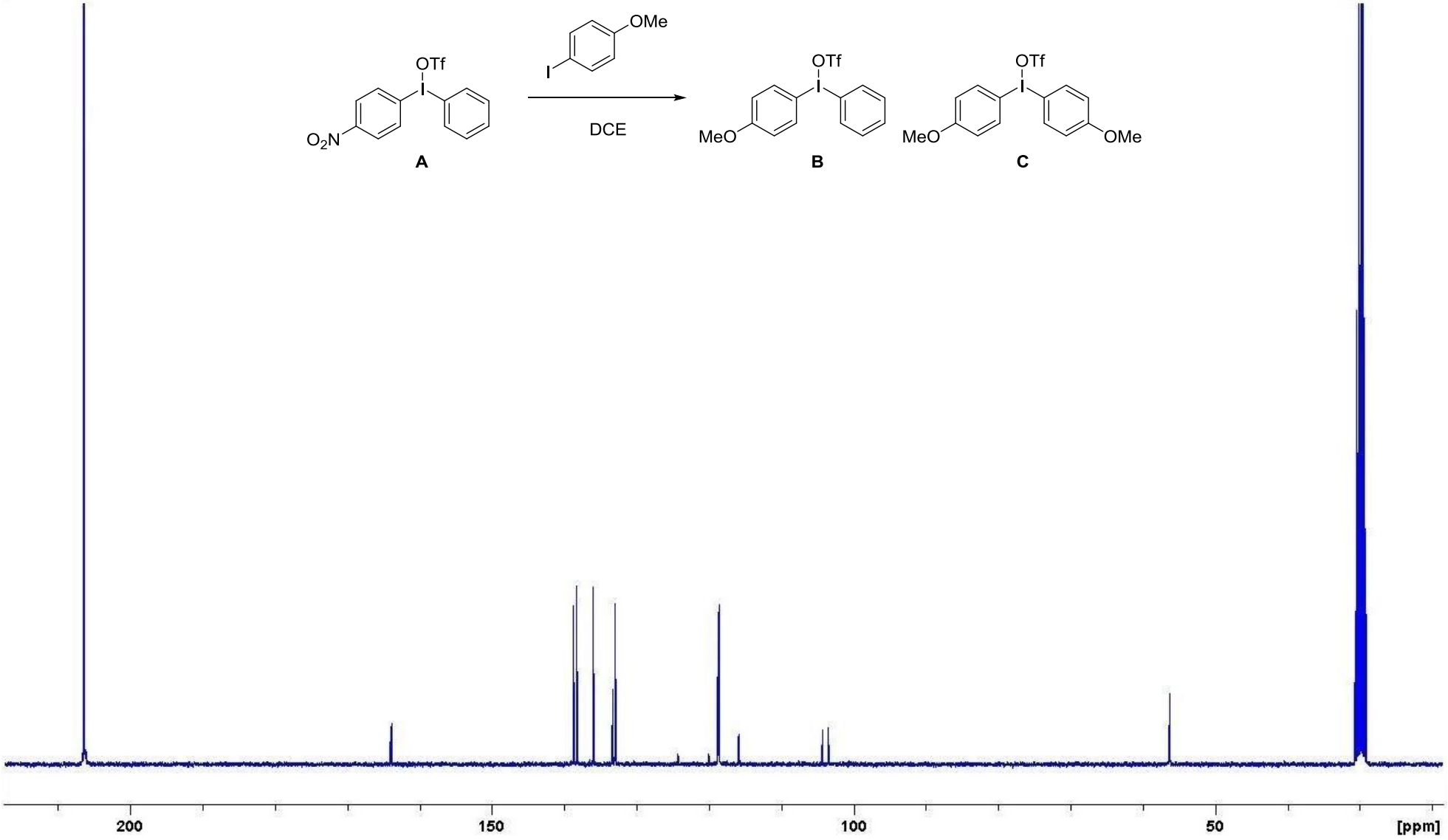


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry c of Table 3 (acetone-d_6)

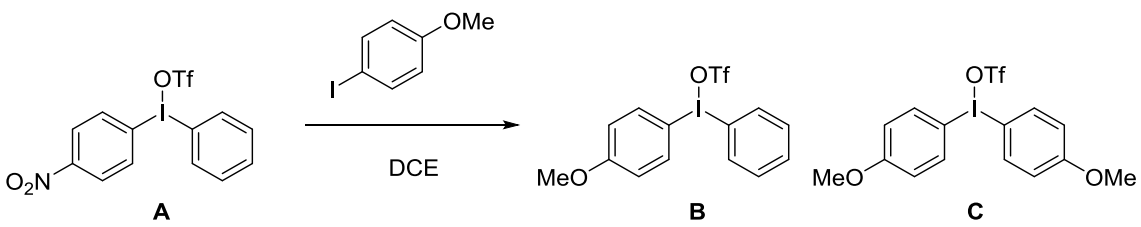


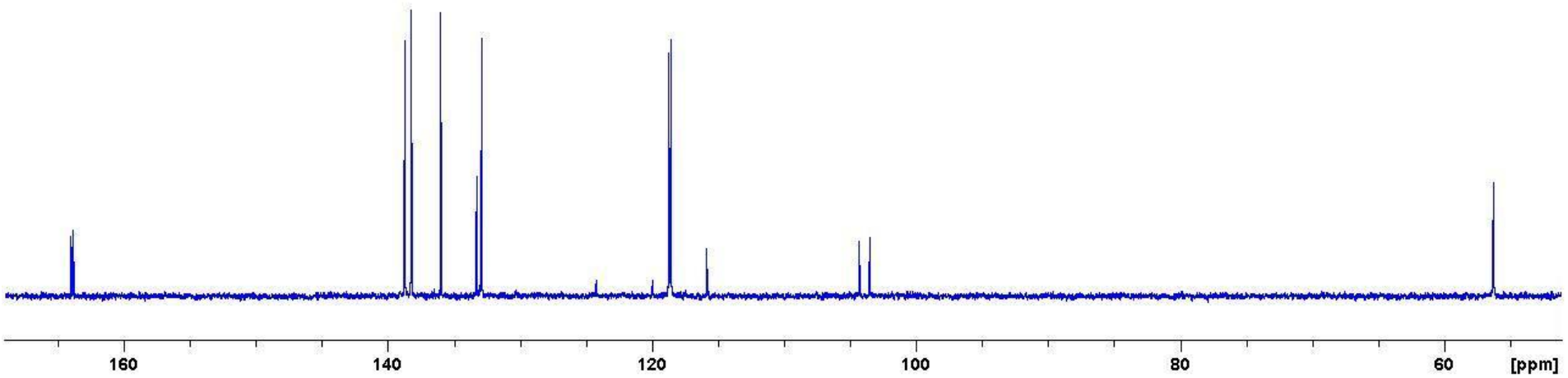


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry c of Table 3 (acetone-d_6)

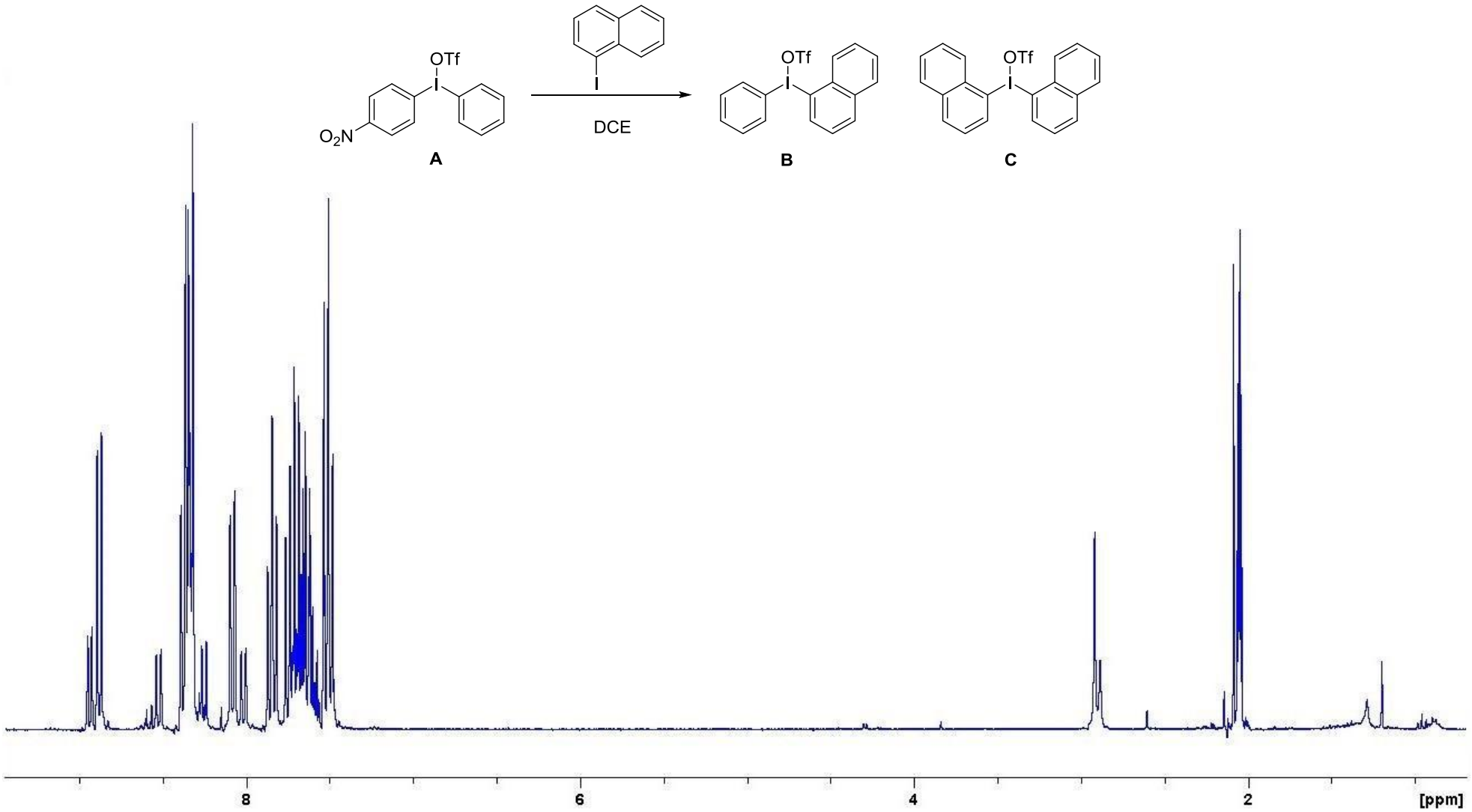


^{13}C NMR spectrum of the product mixture obtained in entry c of Table 3 (acetone-d_6)

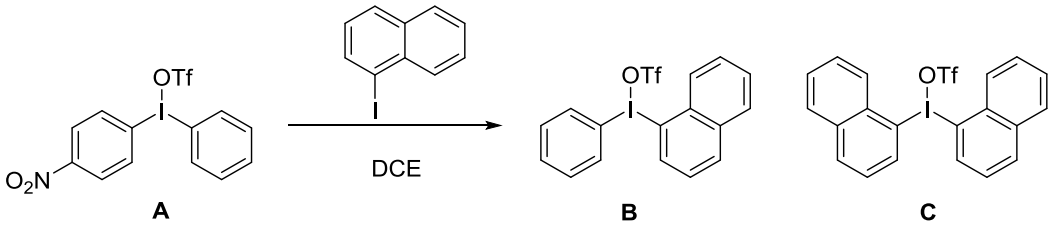


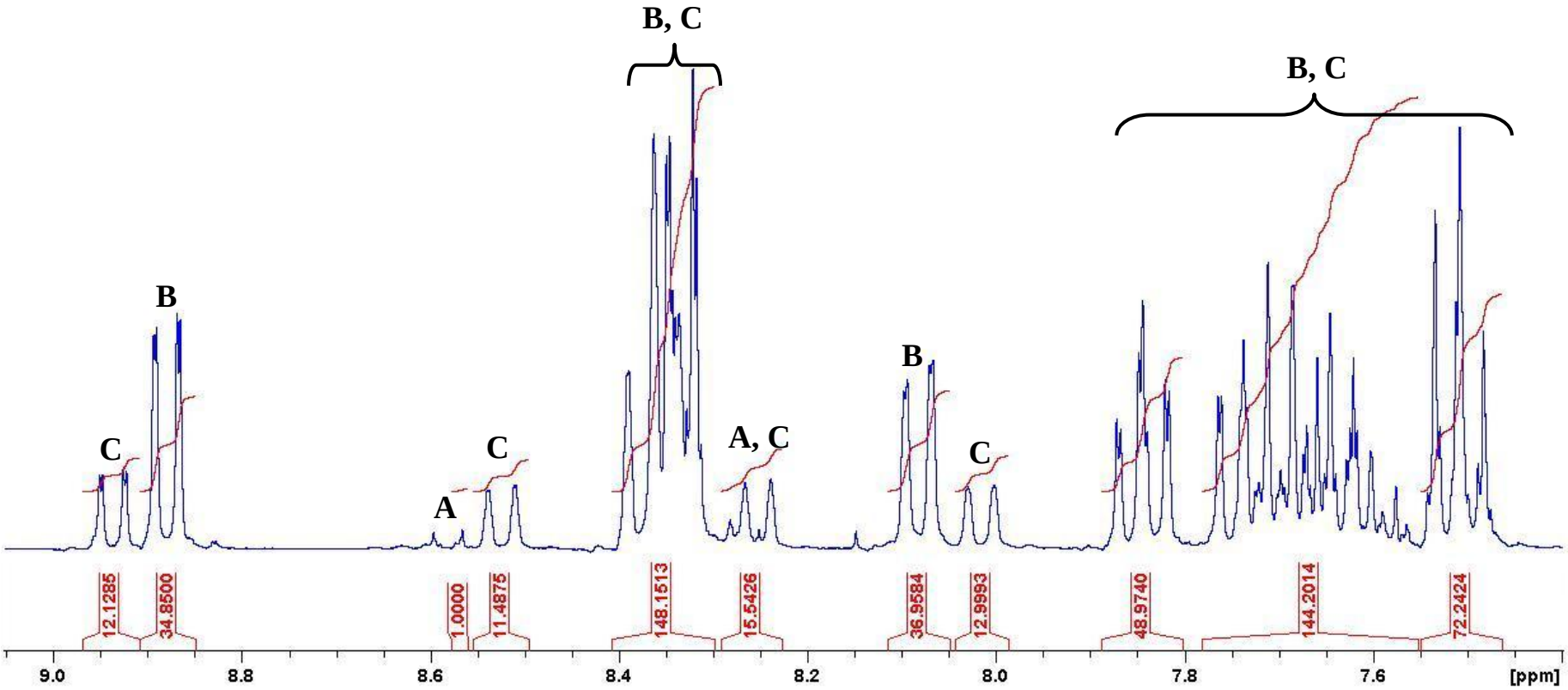


Expanded ^{13}C NMR spectrum of the product mixture obtained in entry c of Table 3 (acetone-d_6)

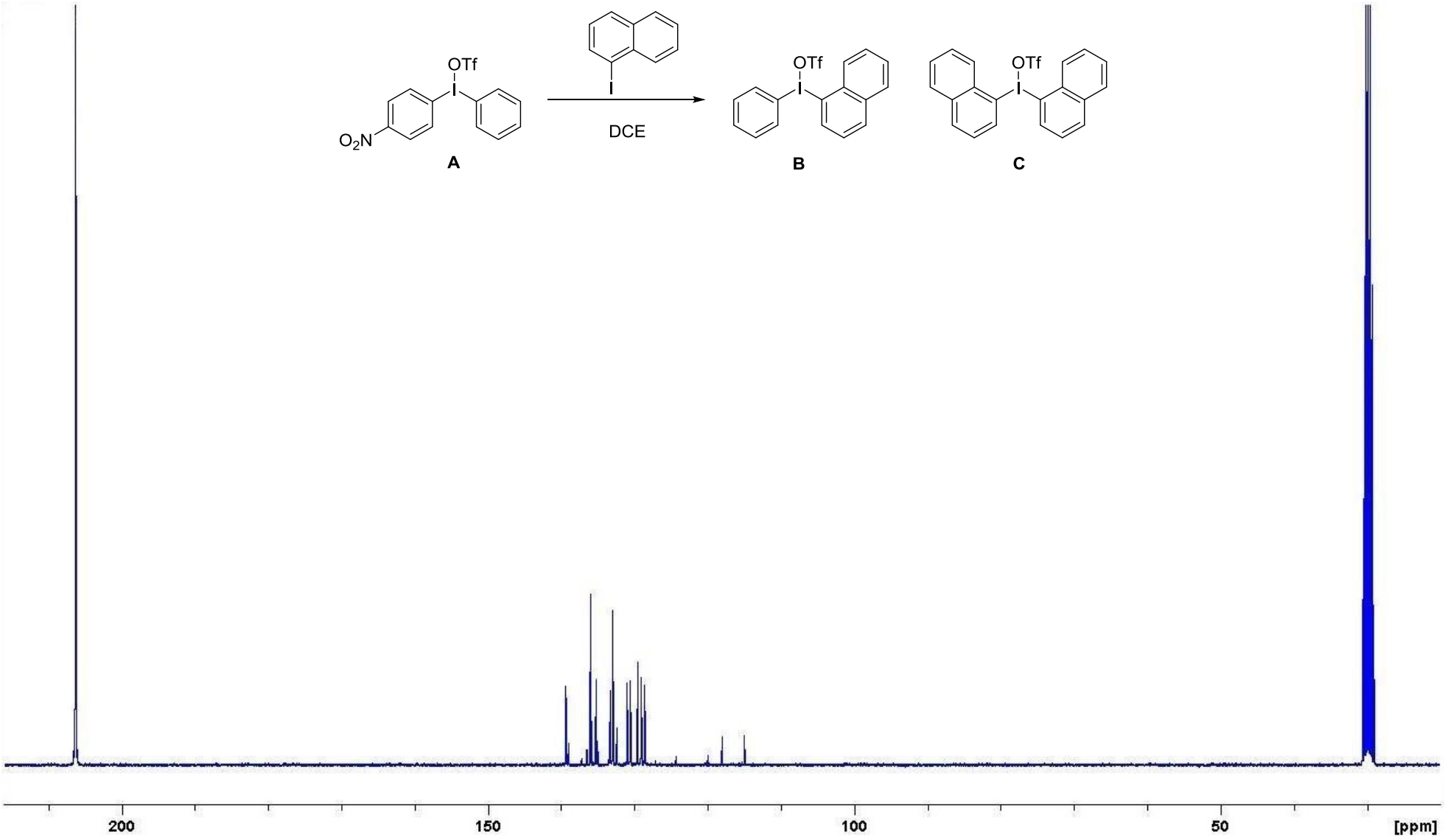


^{1}H NMR spectrum of the product mixture obtained in entry d of Table 3 (acetone-d_6)

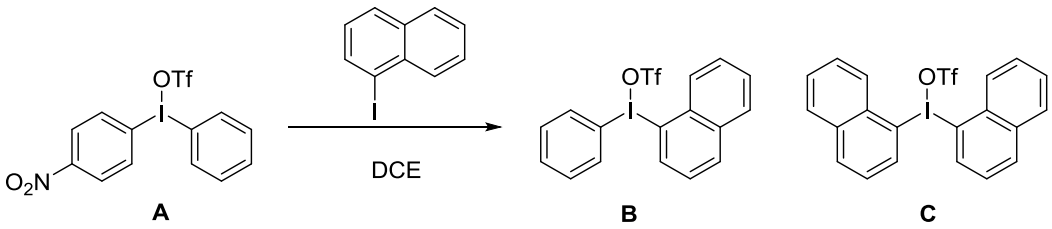


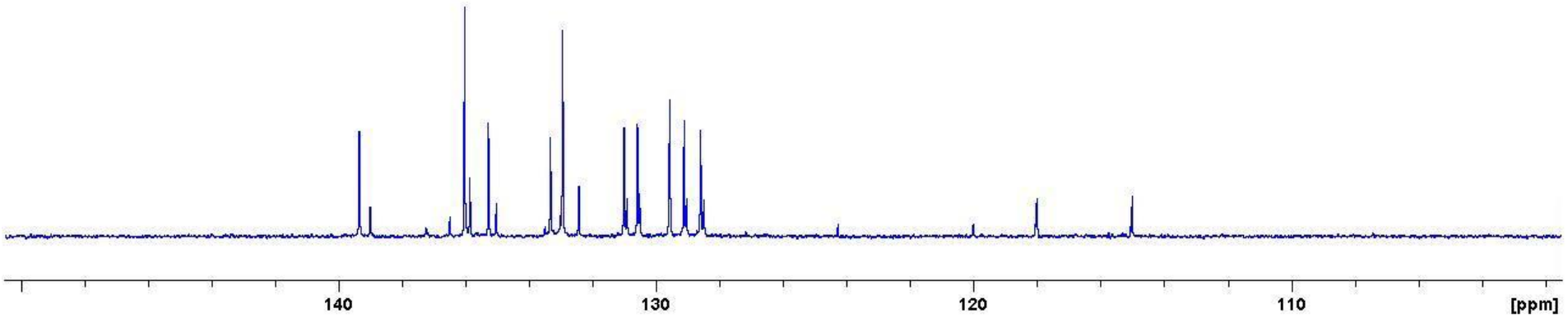


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry d of Table 3 (acetone-d_6)

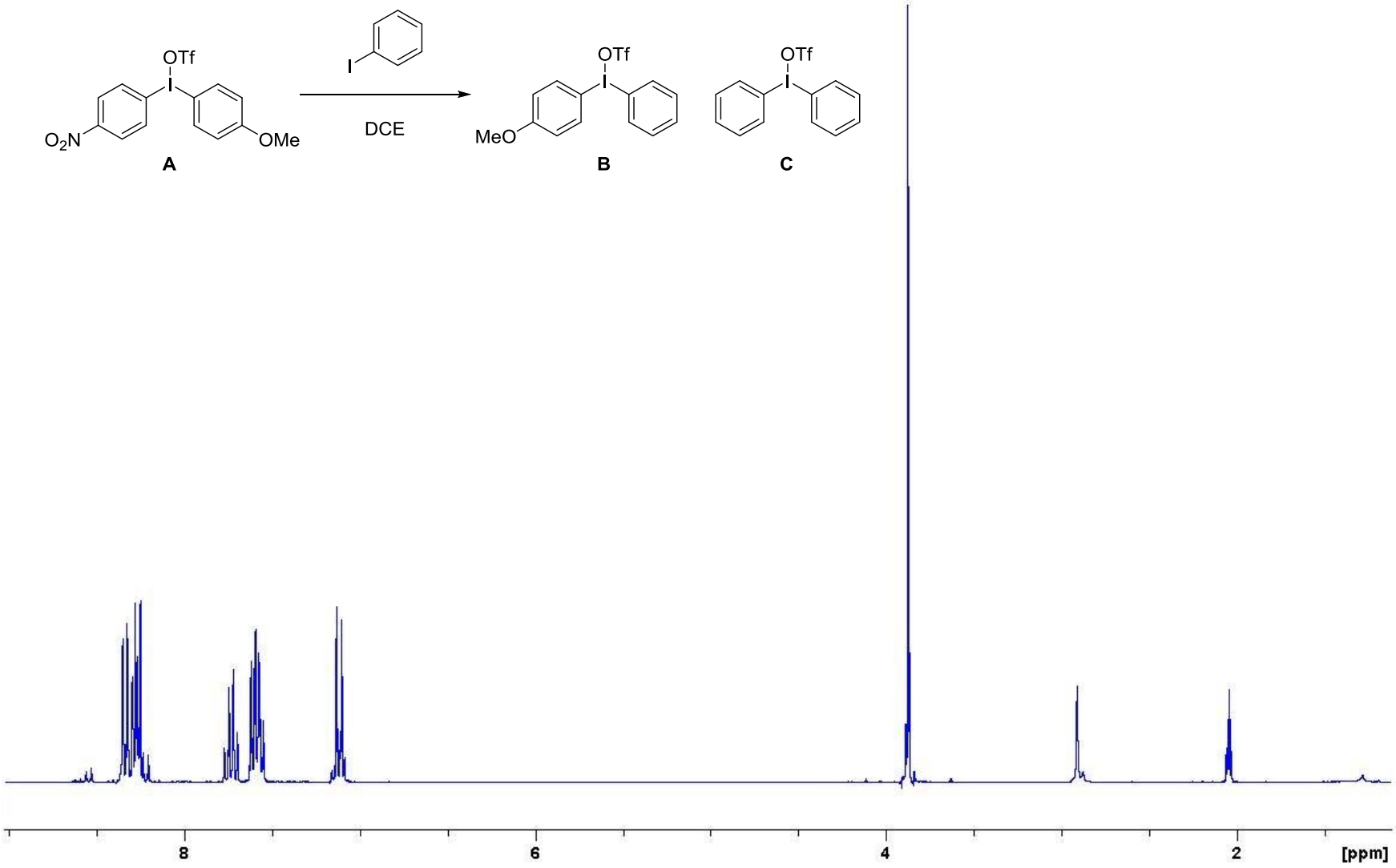


^{13}C NMR spectrum of the product mixture obtained in entry d of Table 3 (acetone-d_6)

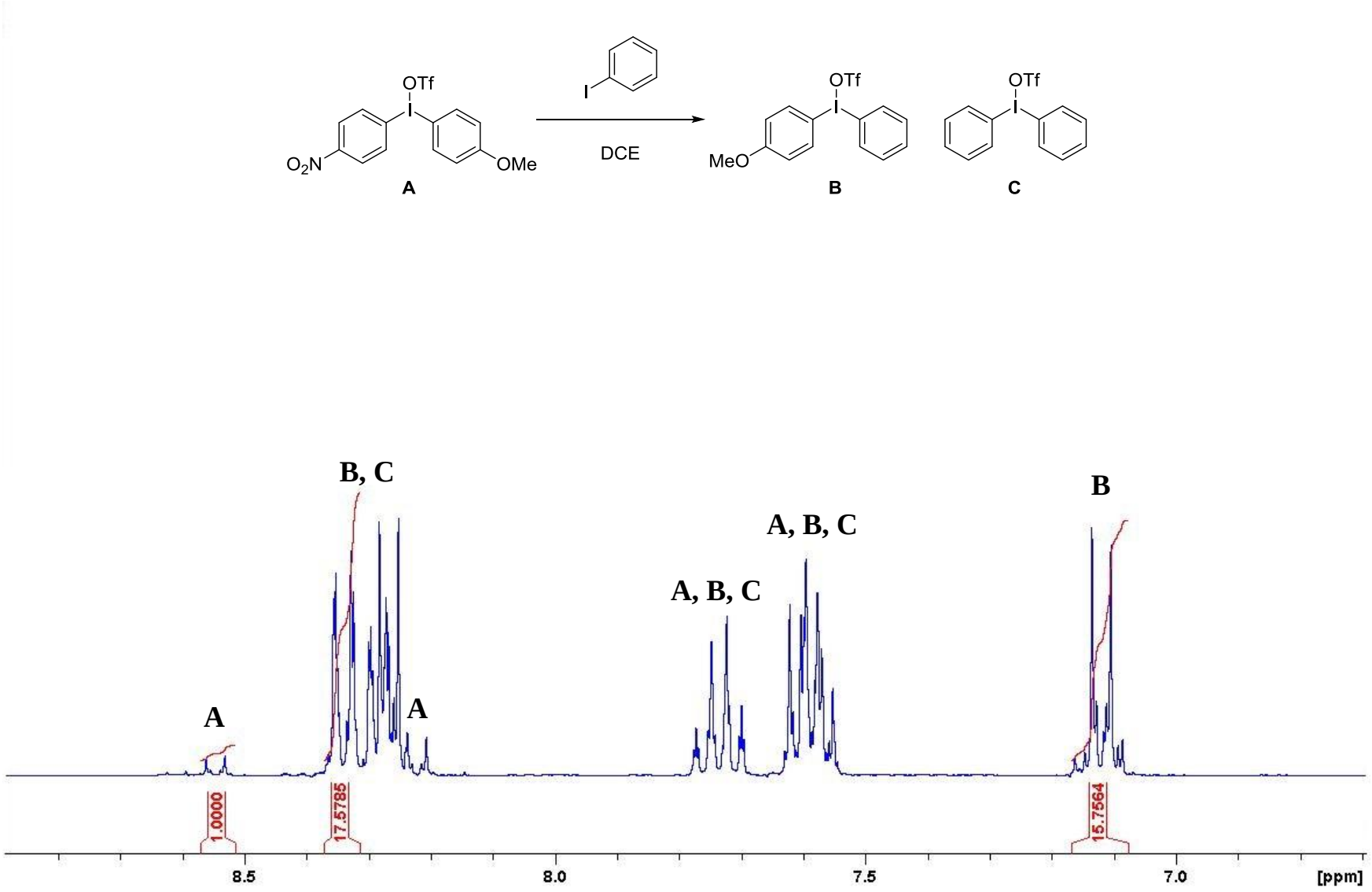




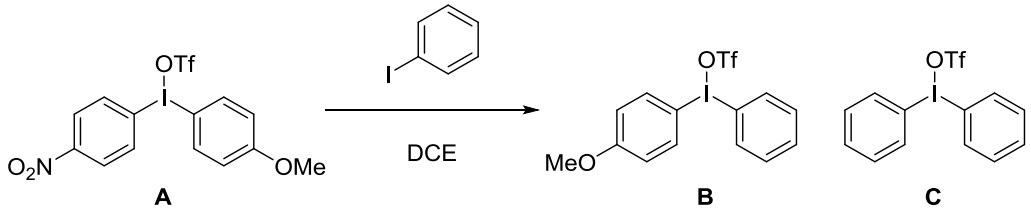
Expanded ^{13}C NMR spectrum of the product mixture obtained in entry d of Table 3 (acetone-d_6)

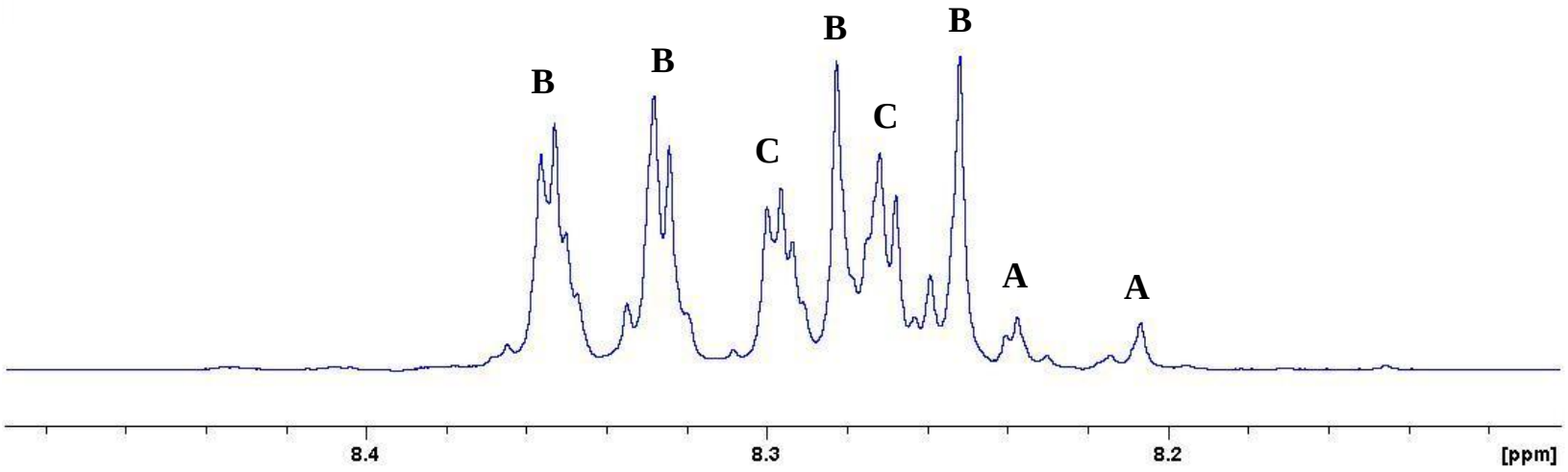


^{1}H NMR spectrum of the product mixture obtained in entry e of Table 3 (acetone-d_6)

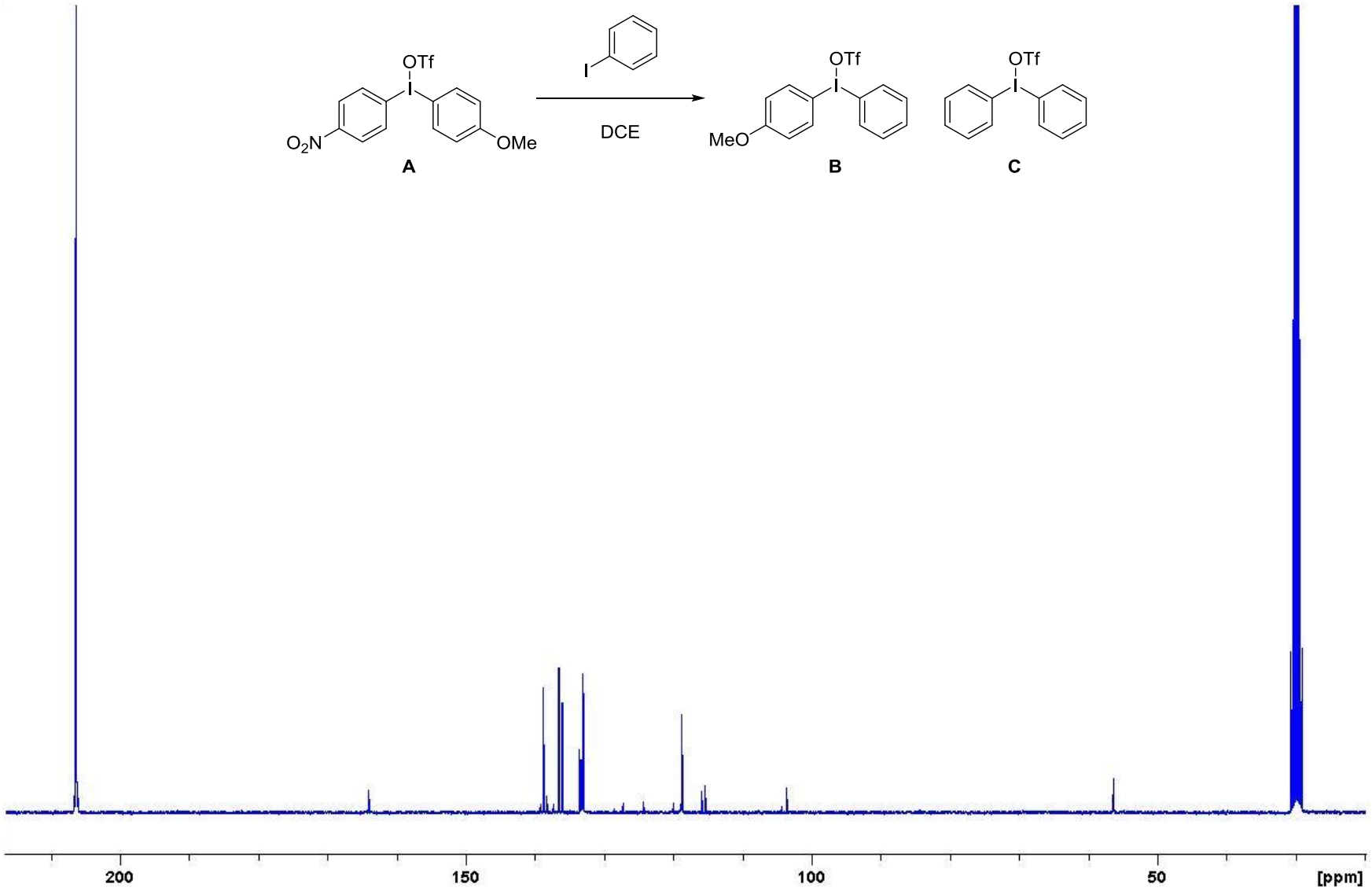


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry e of Table 3 (acetone-d_6)

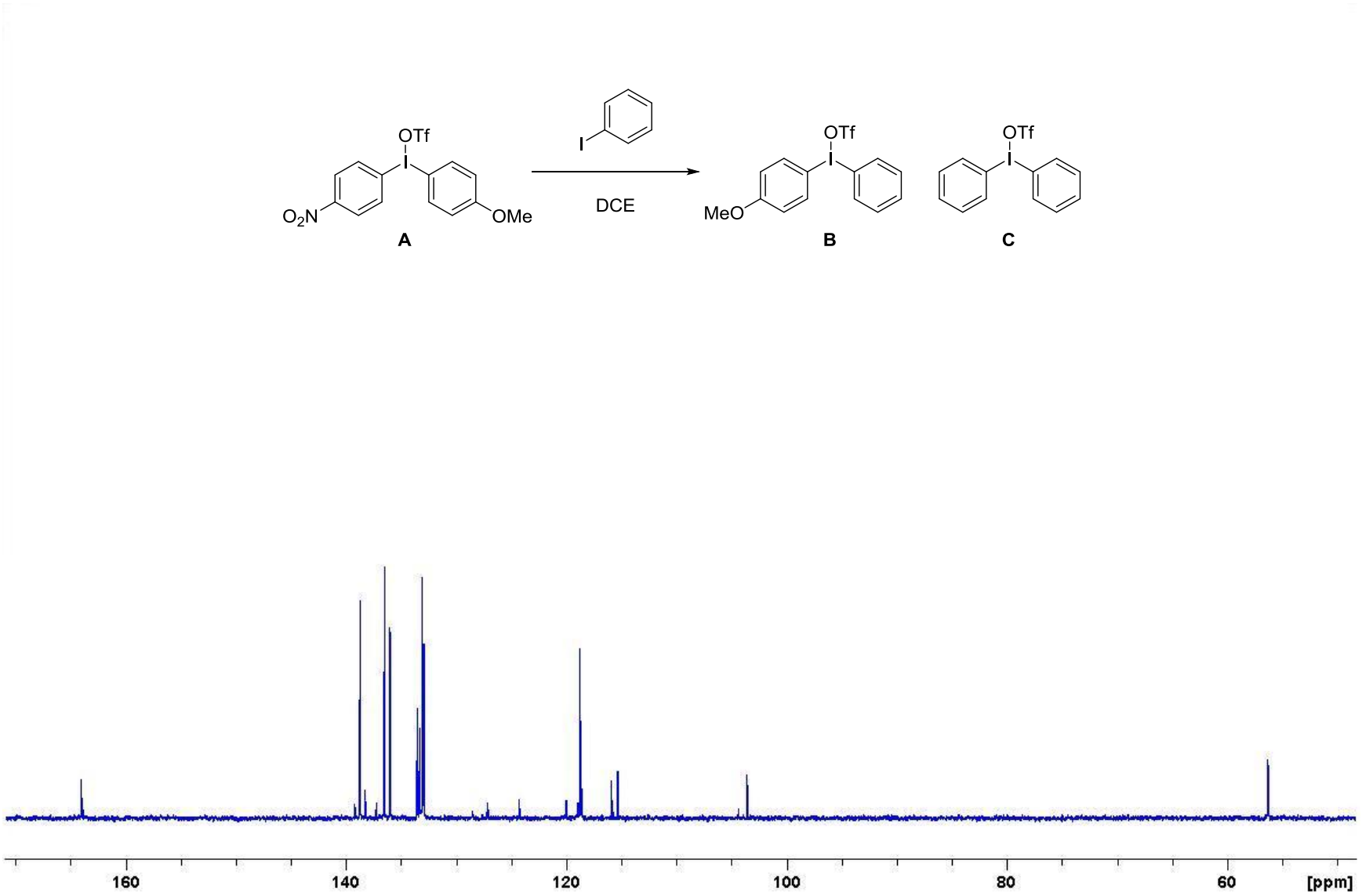




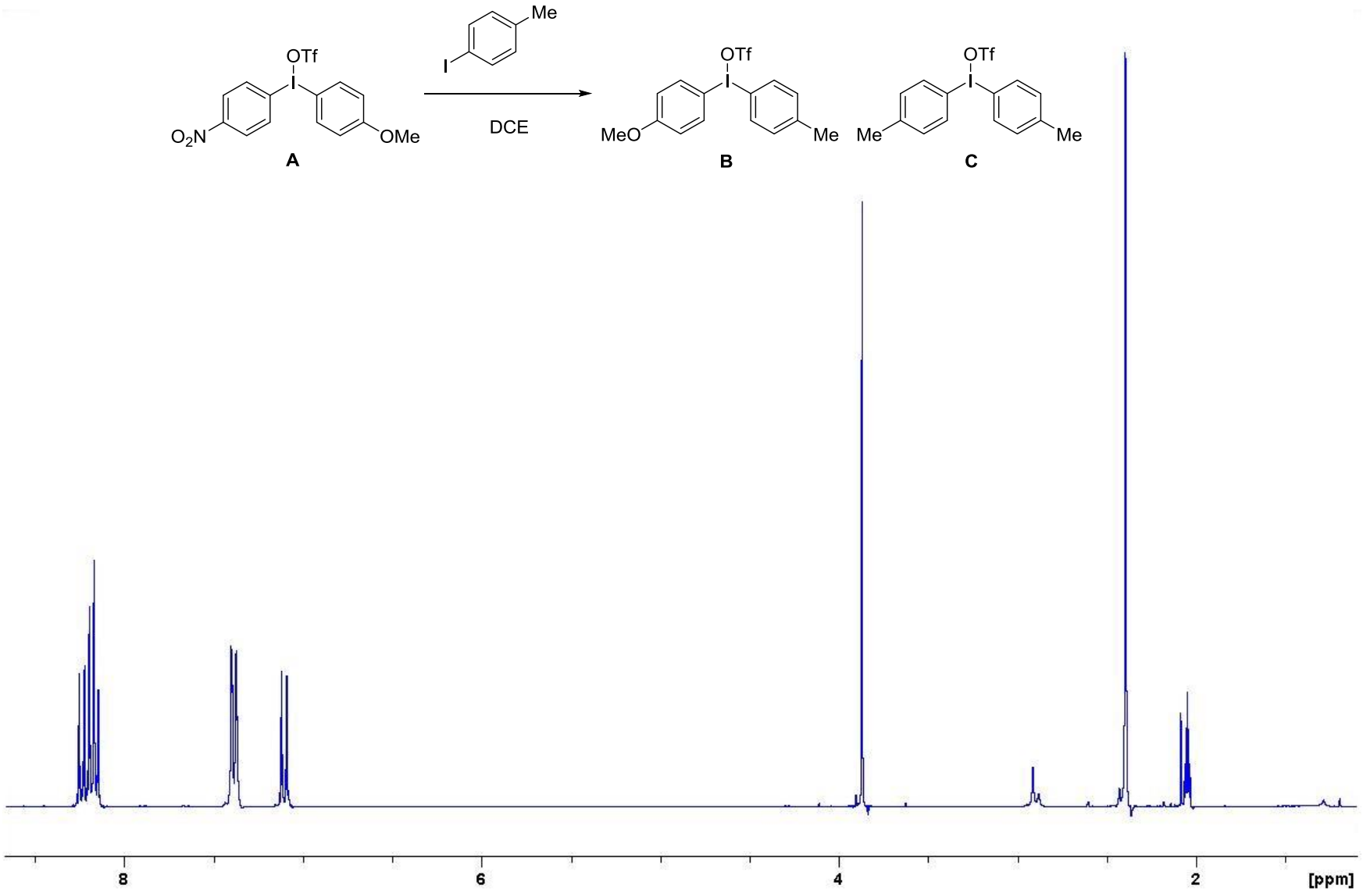
Expanded ^{1}H NMR spectrum of the product mixture obtained in entry e of Table 3 (acetone-d_6)



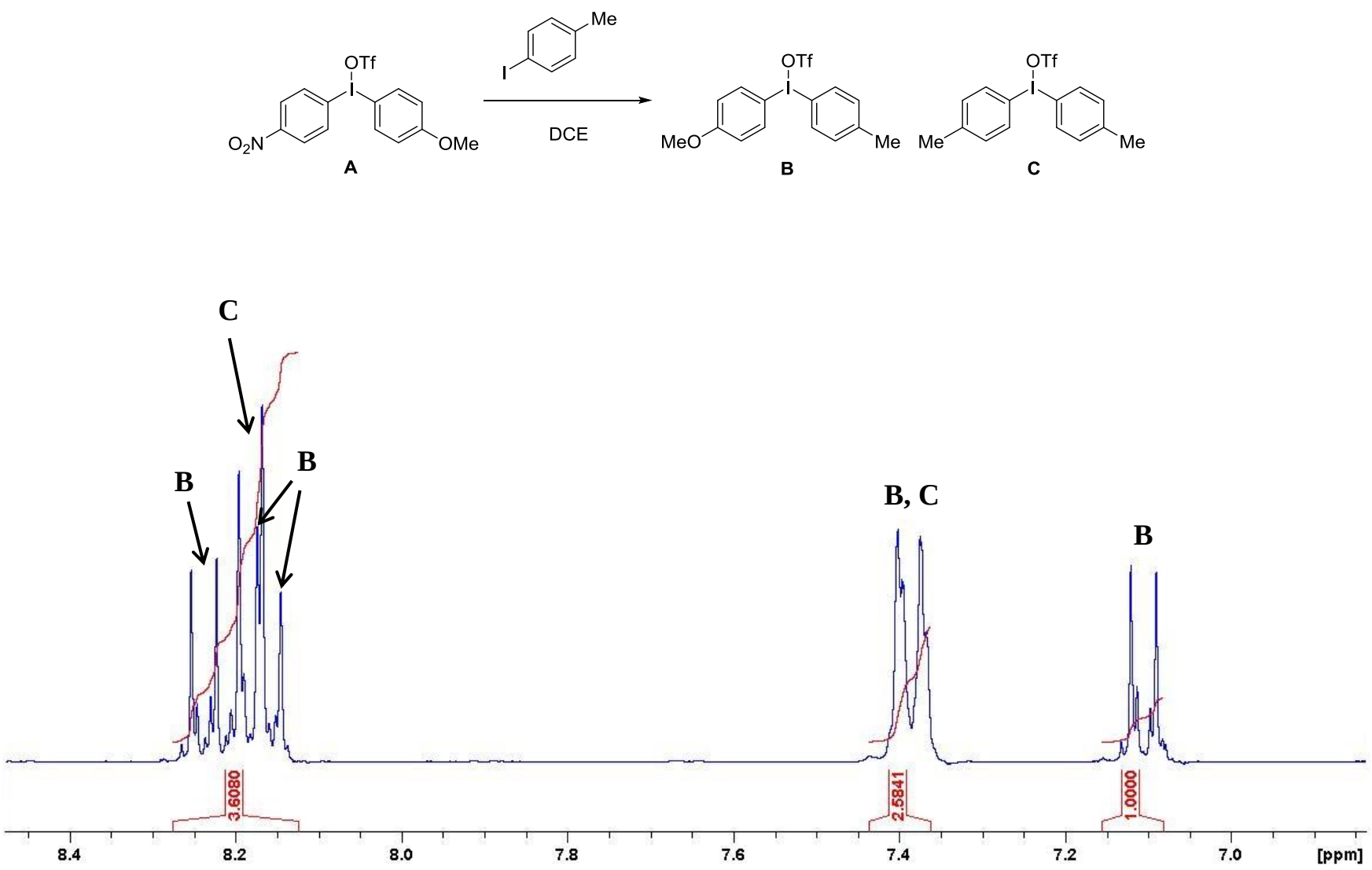
^{13}C NMR spectrum of the product mixture obtained in entry e of Table 3 (acetone-d_6)



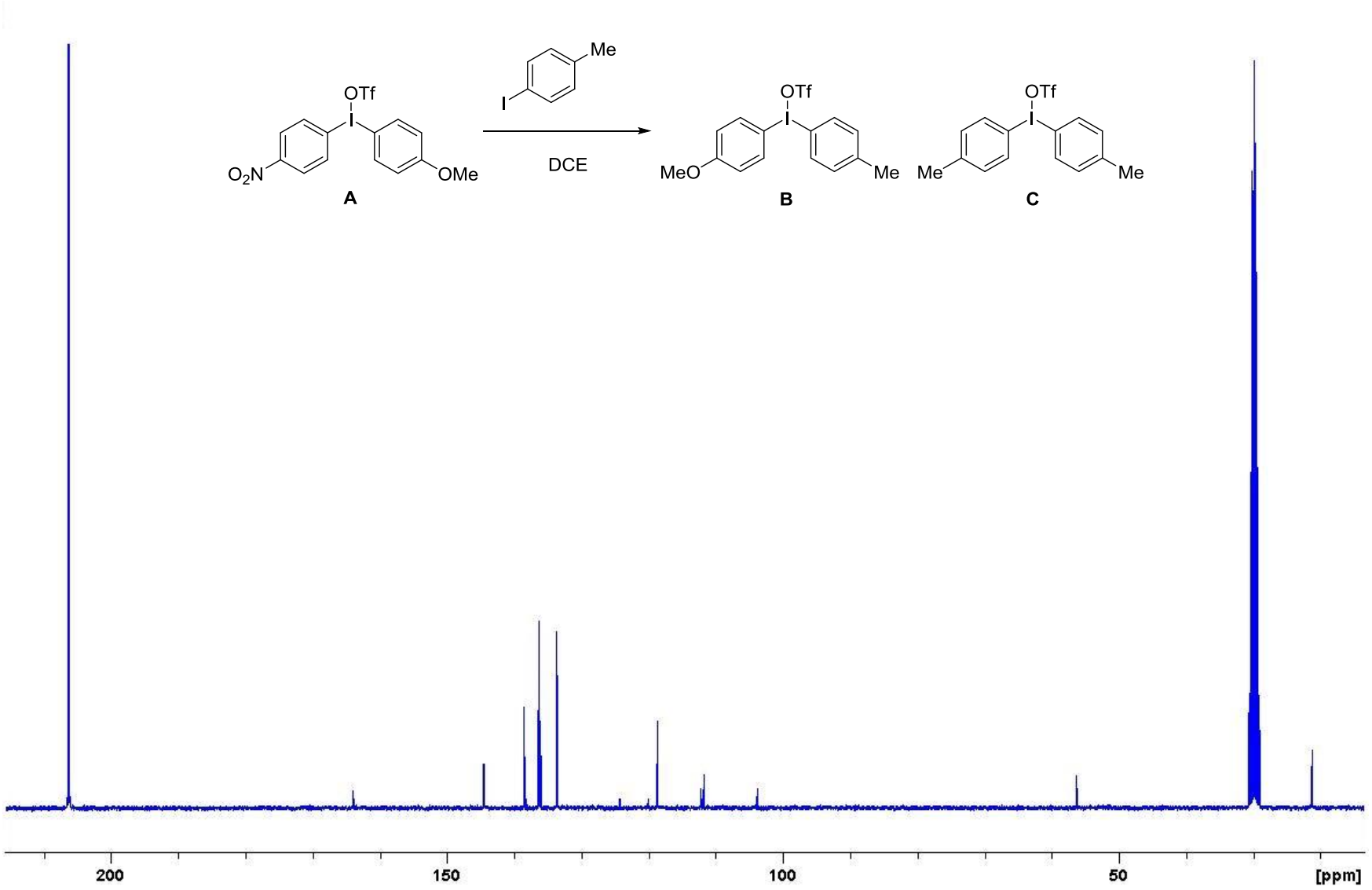
Expanded ^{13}C NMR spectrum of the product mixture obtained in entry e of Table 3 (acetone-d_6)



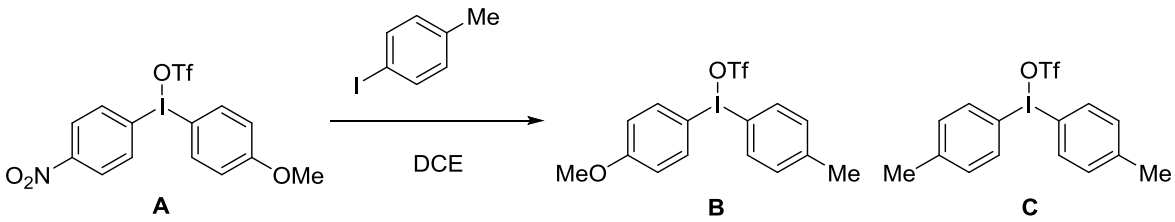
^{1}H NMR spectrum of the product mixture obtained in entry f of Table 3 (acetone-d_6)

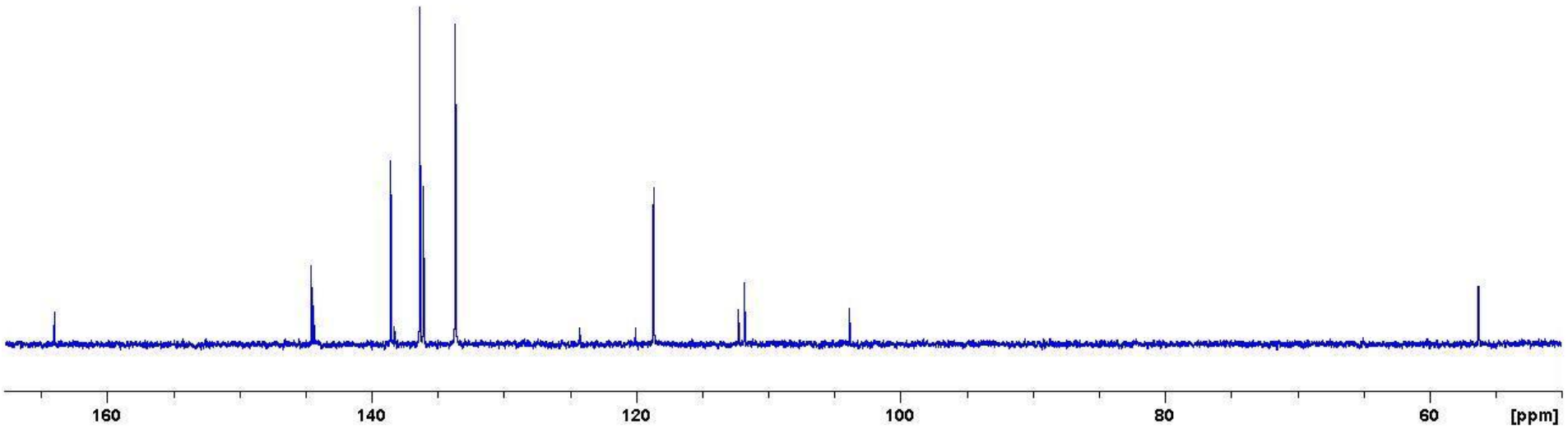


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry f of Table 3 (acetone-d_6)

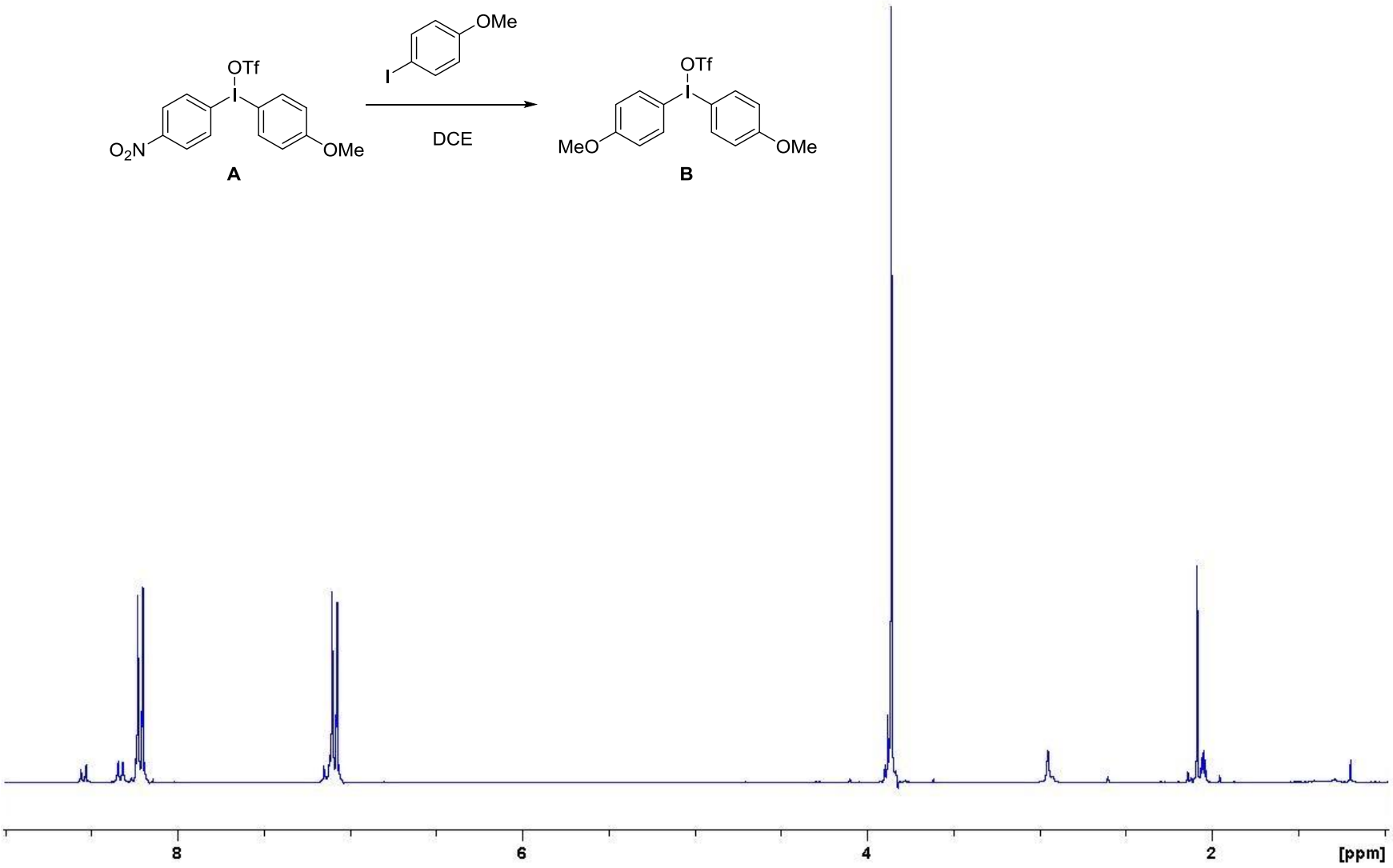


^{13}C NMR spectrum of the product mixture obtained in entry f of Table 3 (acetone-d_6)

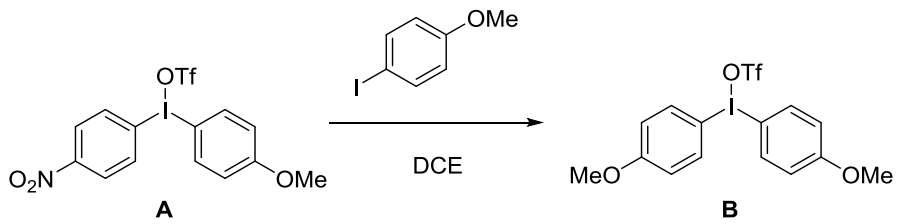


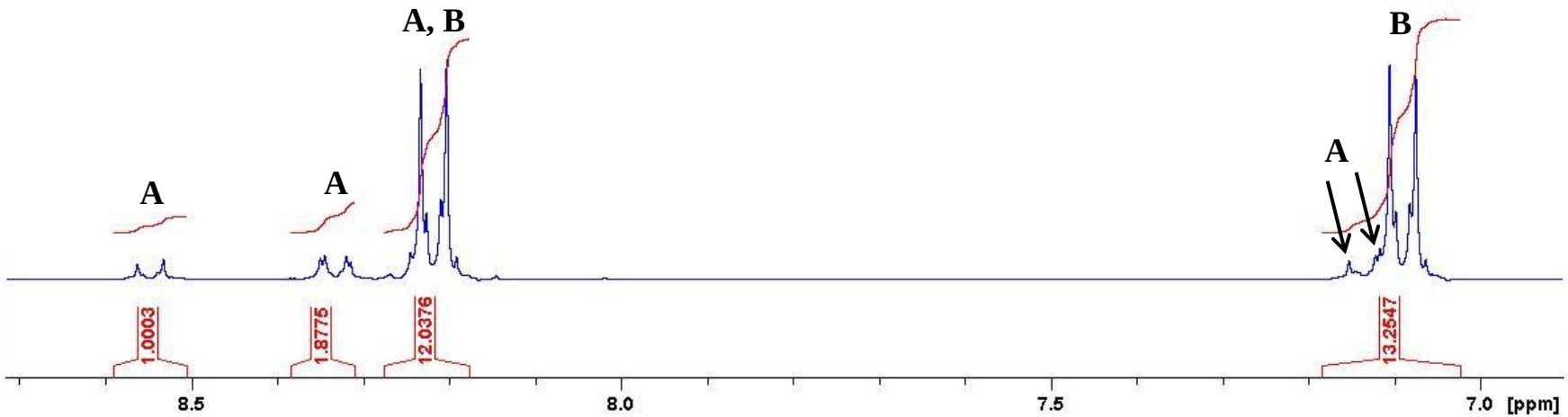


Expanded ^{13}C NMR spectrum of the product mixture obtained in entry f of Table 3 (acetone-d_6)

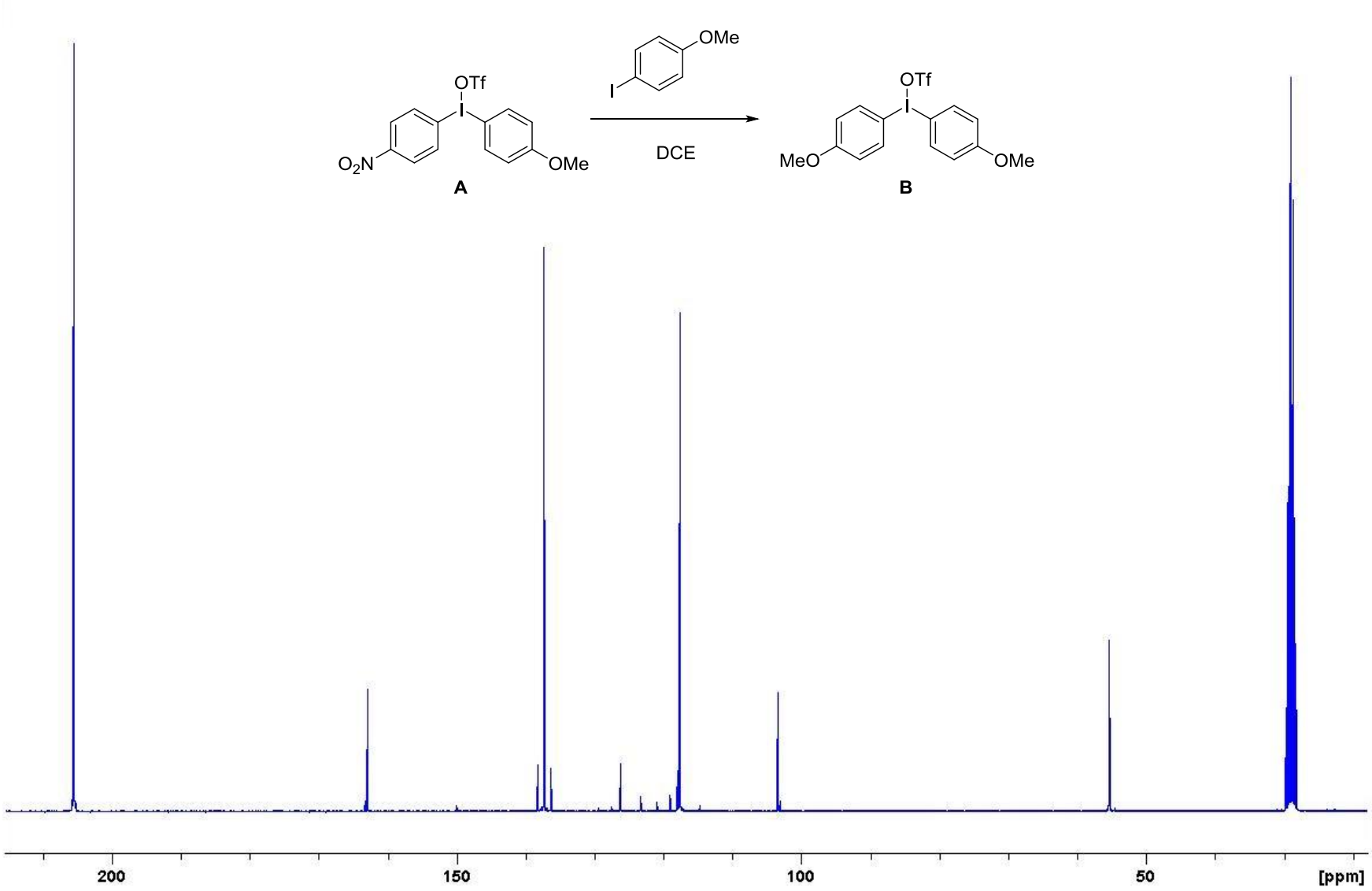


^{1}H NMR spectrum of the product mixture obtained in entry g of Table 3 (acetone-d_6)

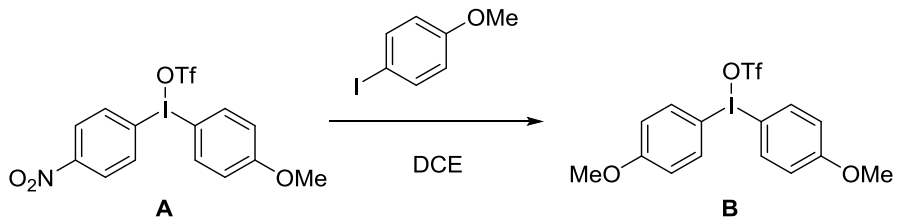


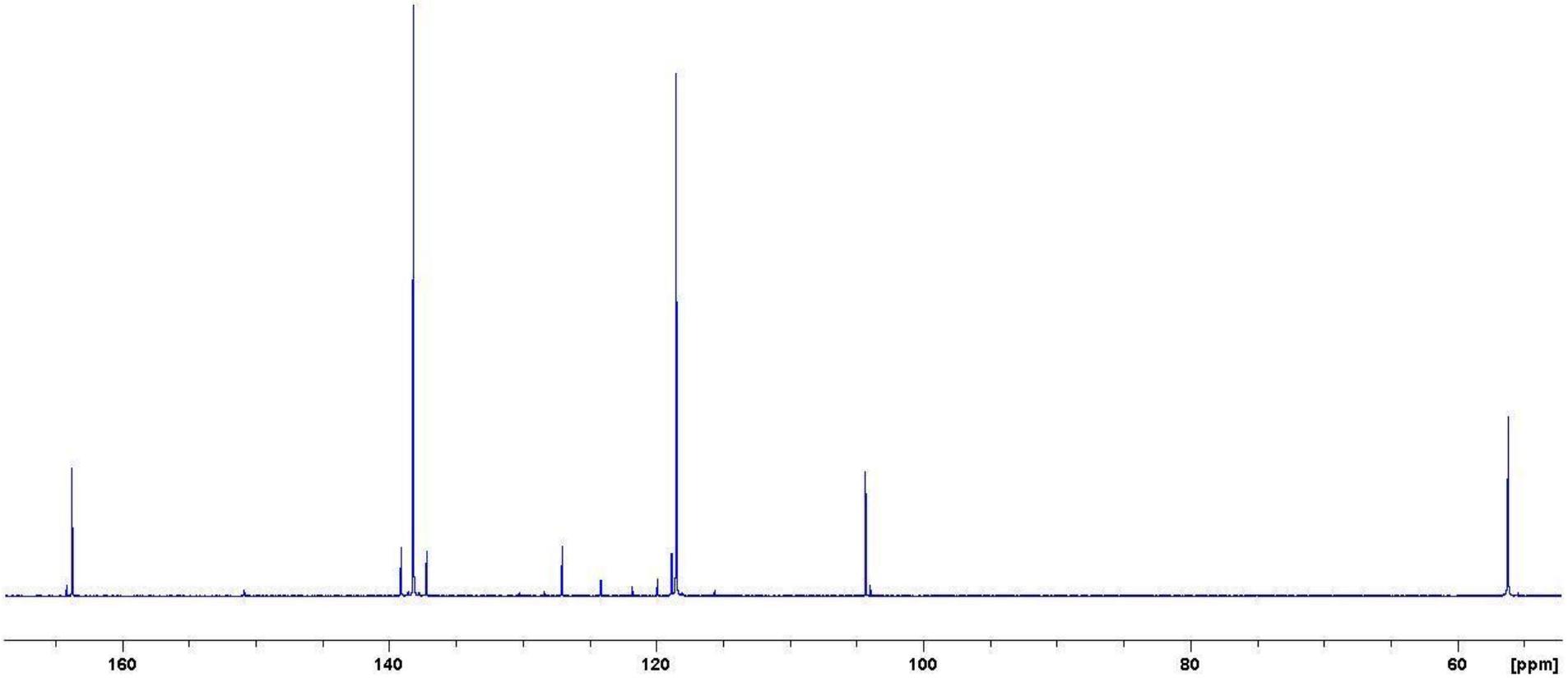


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry g of Table 3 (acetone-d_6)

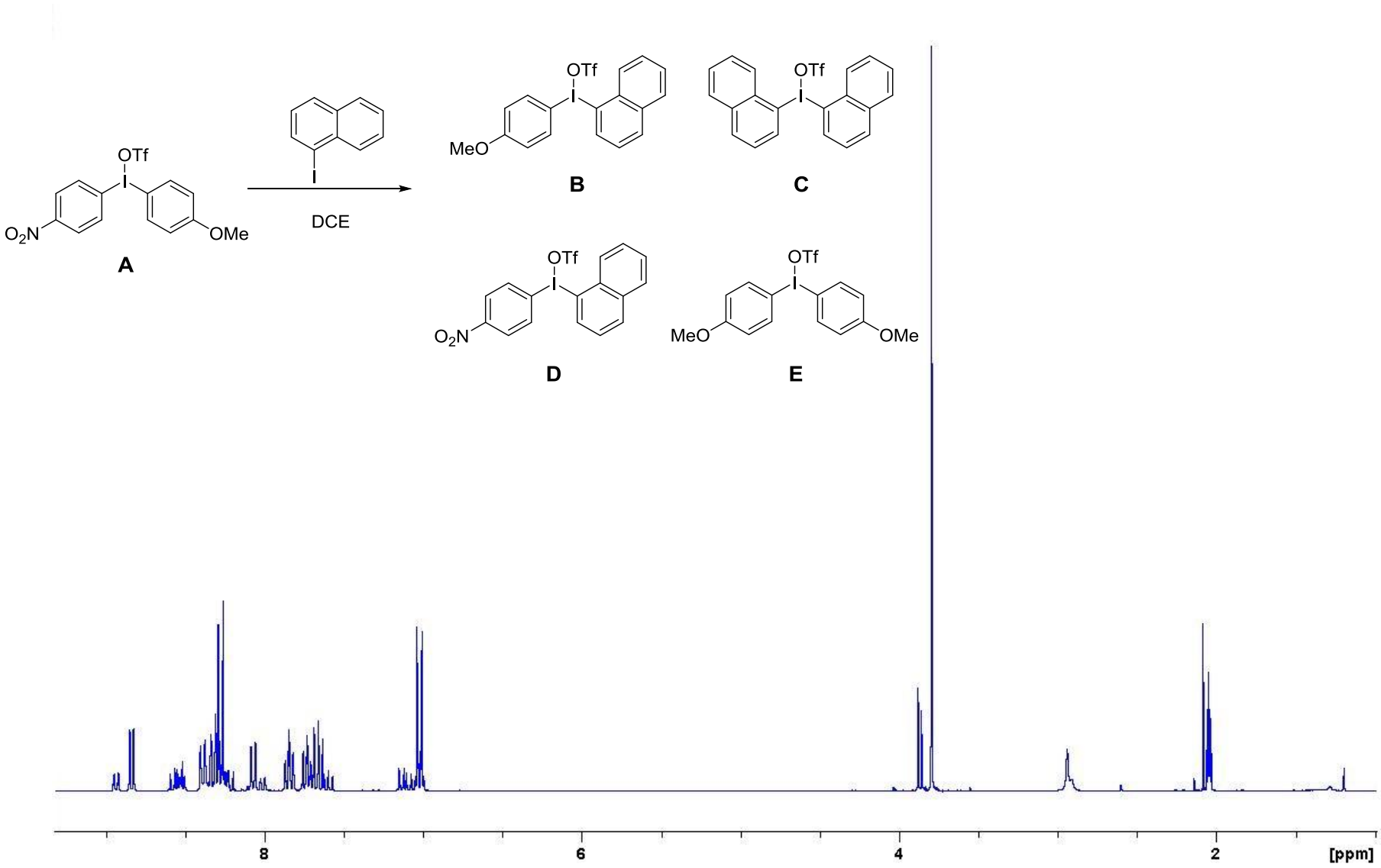


^{13}C NMR spectrum of the product mixture obtained in entry g of Table 3 (acetone-d_6)

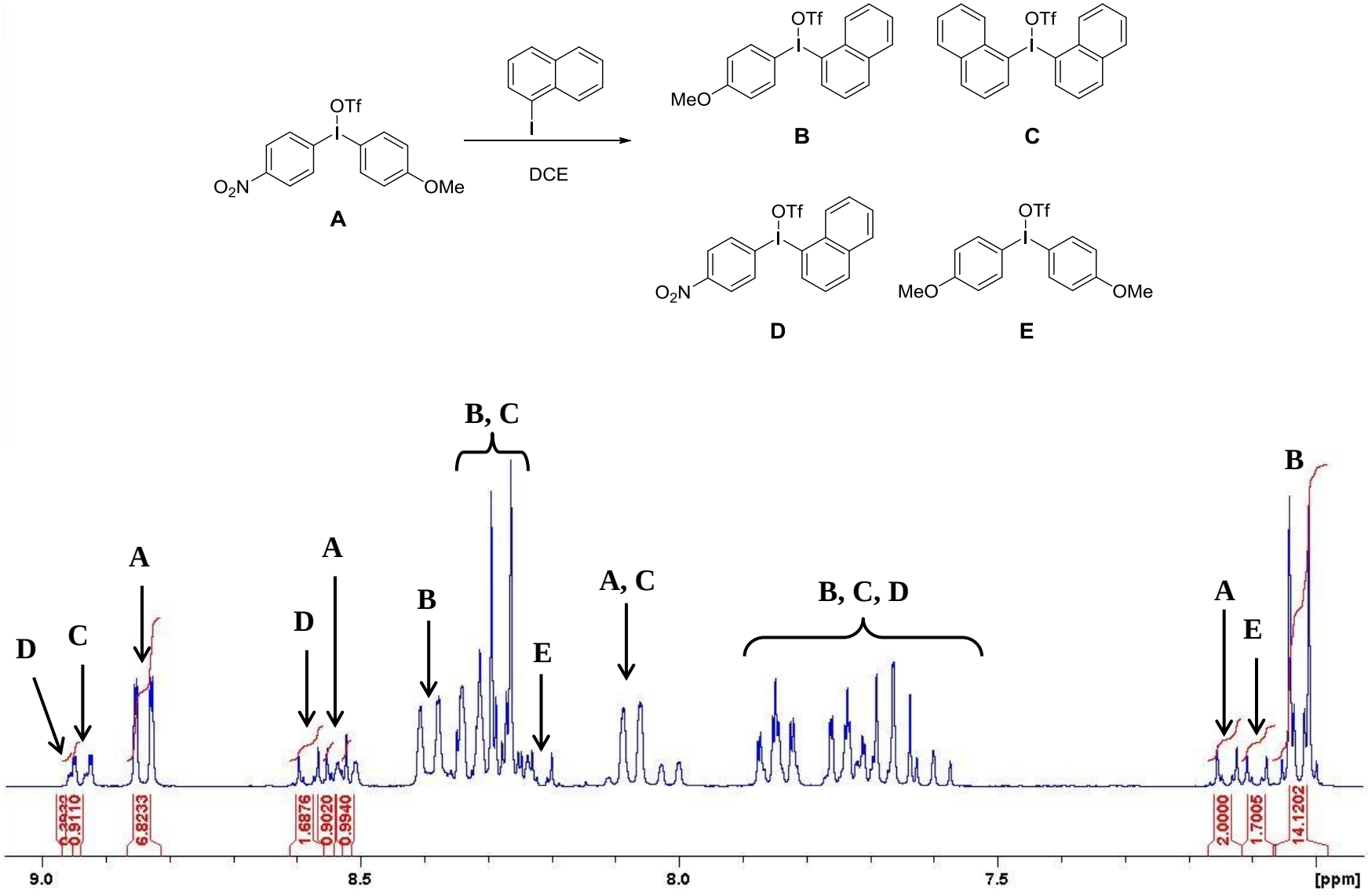




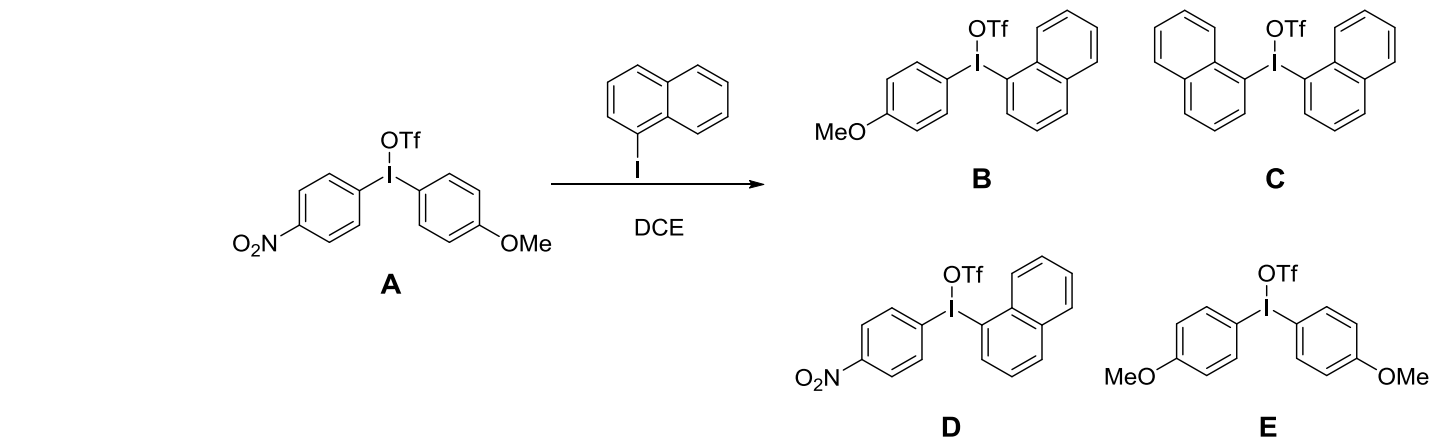
Expanded ^{13}C NMR spectrum of the product mixture obtained in entry g of Table 3 (acetone-d_6)

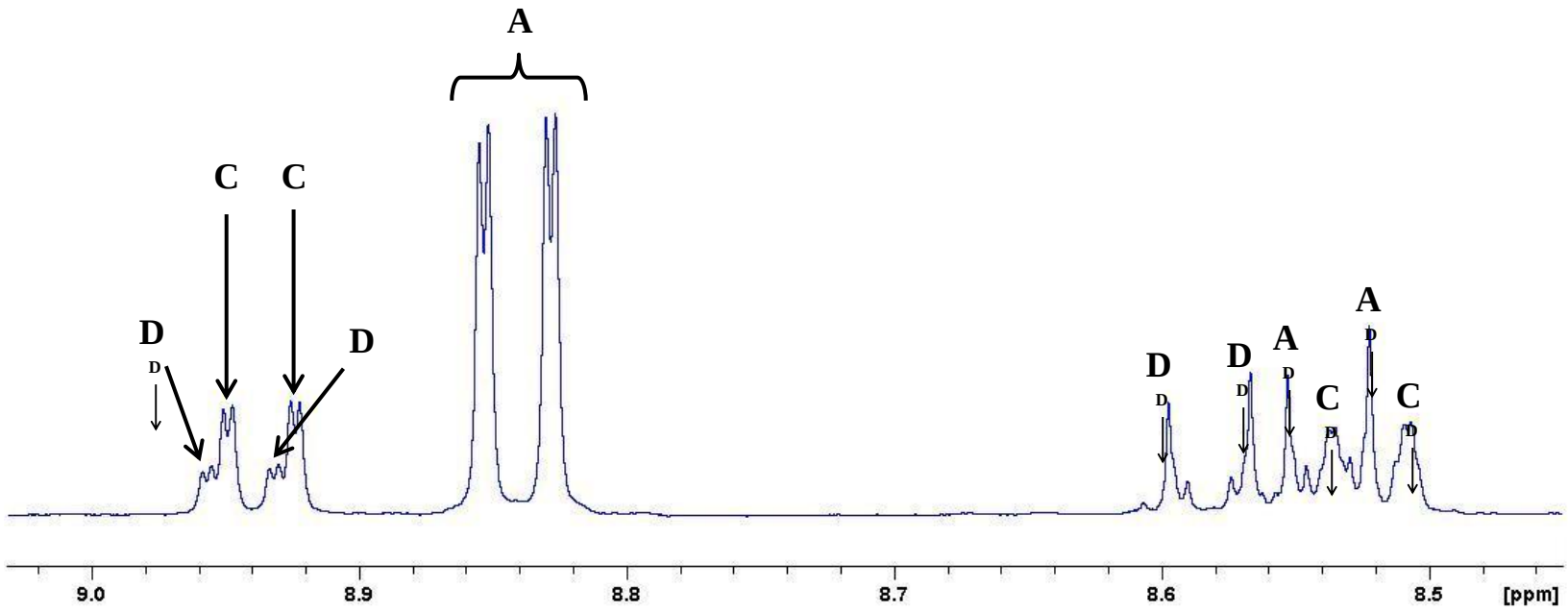


^{1}H NMR spectrum of the product mixture obtained in entry h of Table 3 (acetone-d_6)

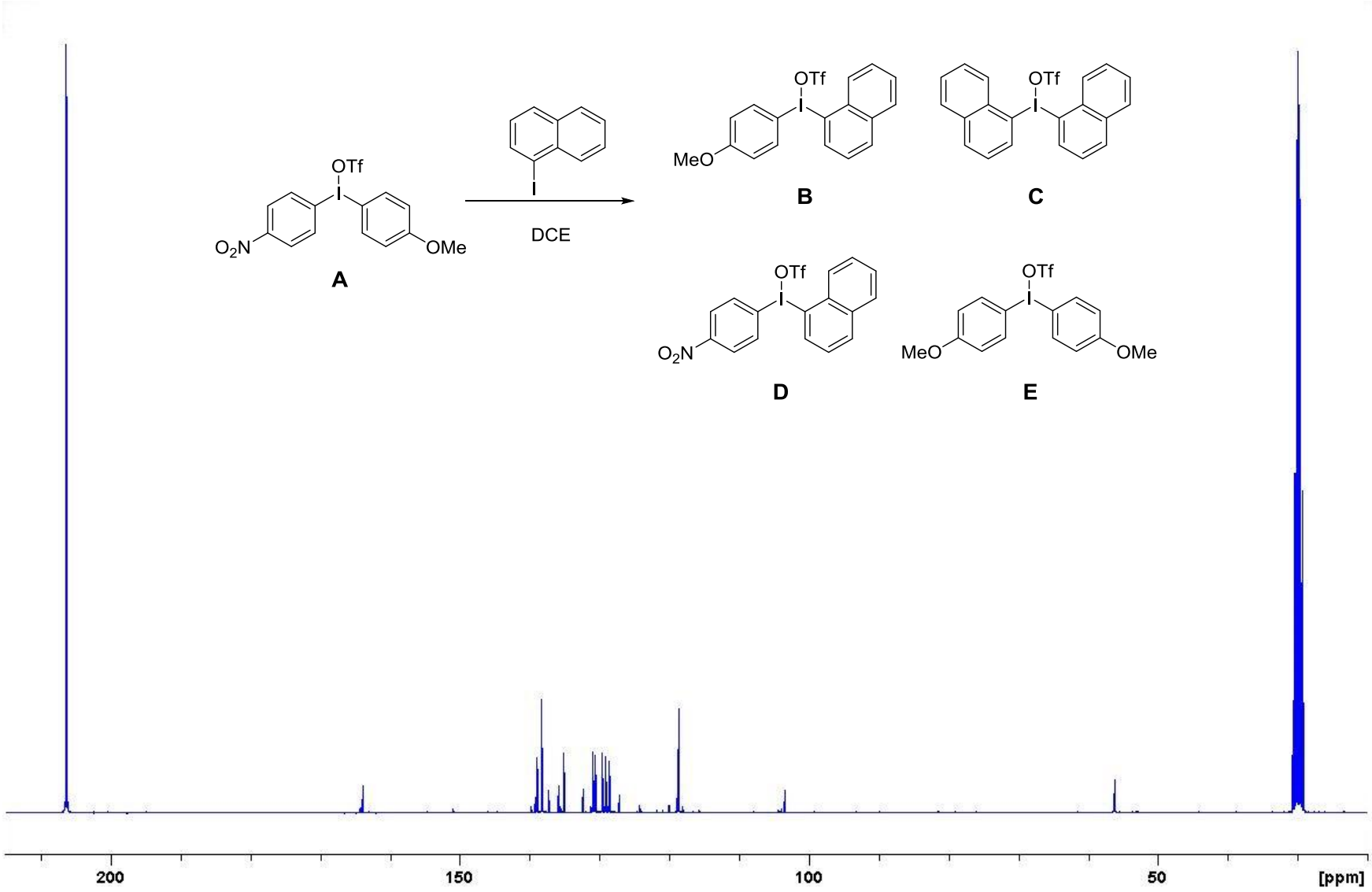


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry h of Table 3 (acetone-d_6)

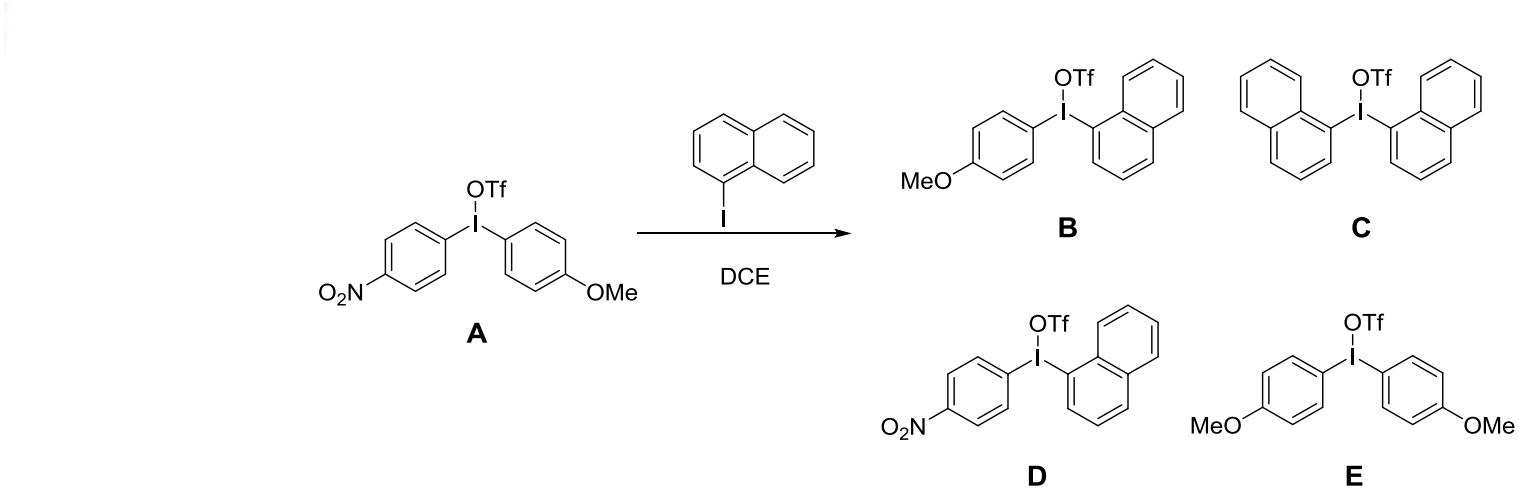


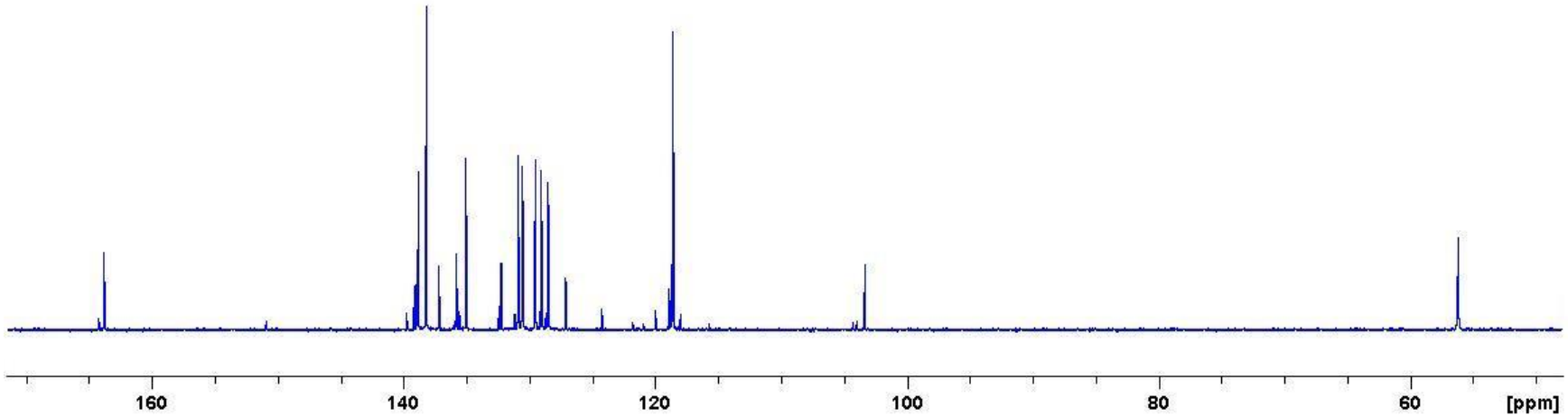


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry h of Table 3 (acetone-d_6)

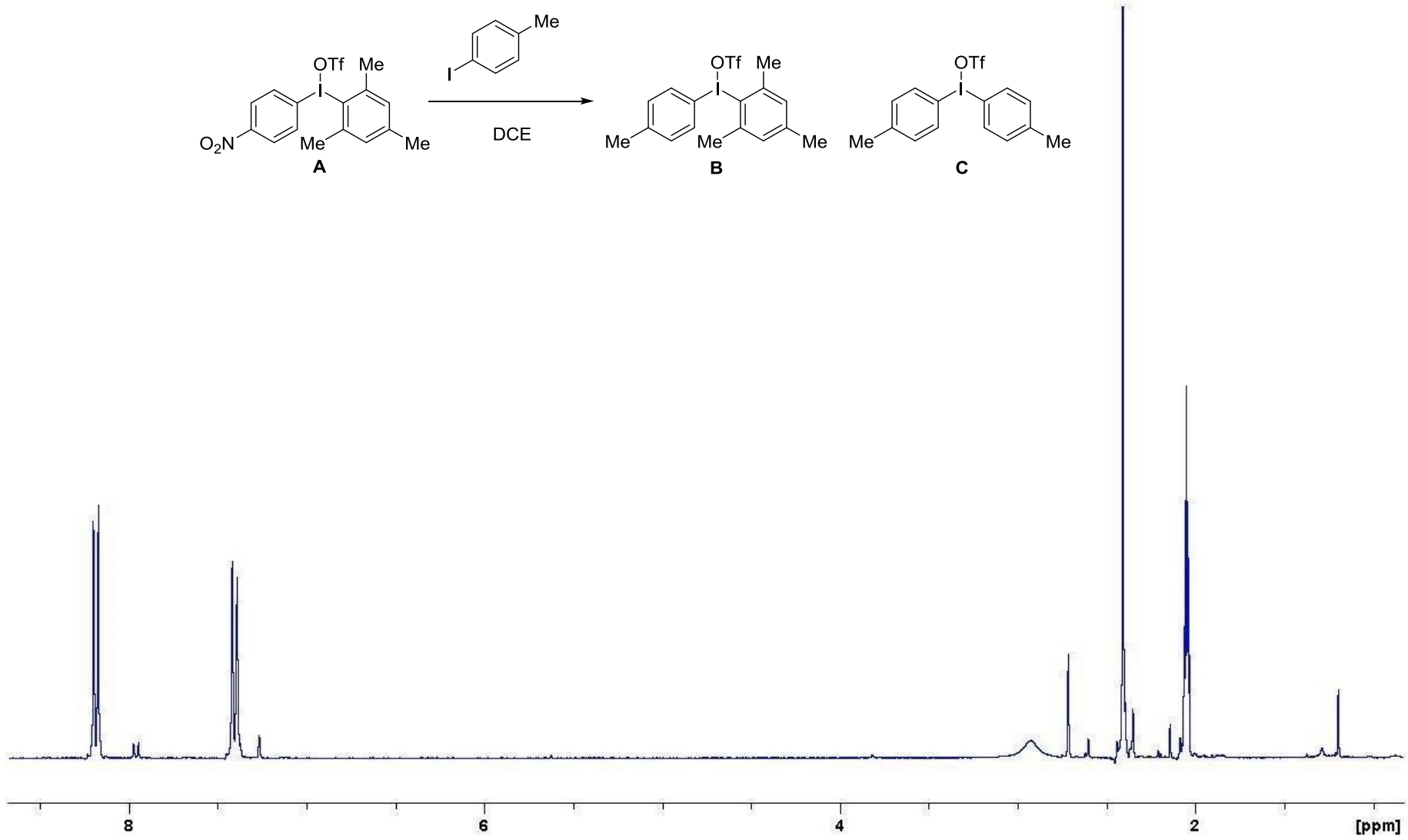


^{13}C NMR spectrum of the product mixture obtained in entry h of Table 3 (acetone-d_6)

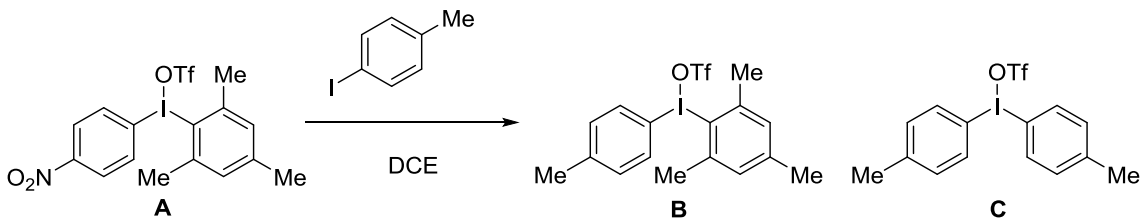


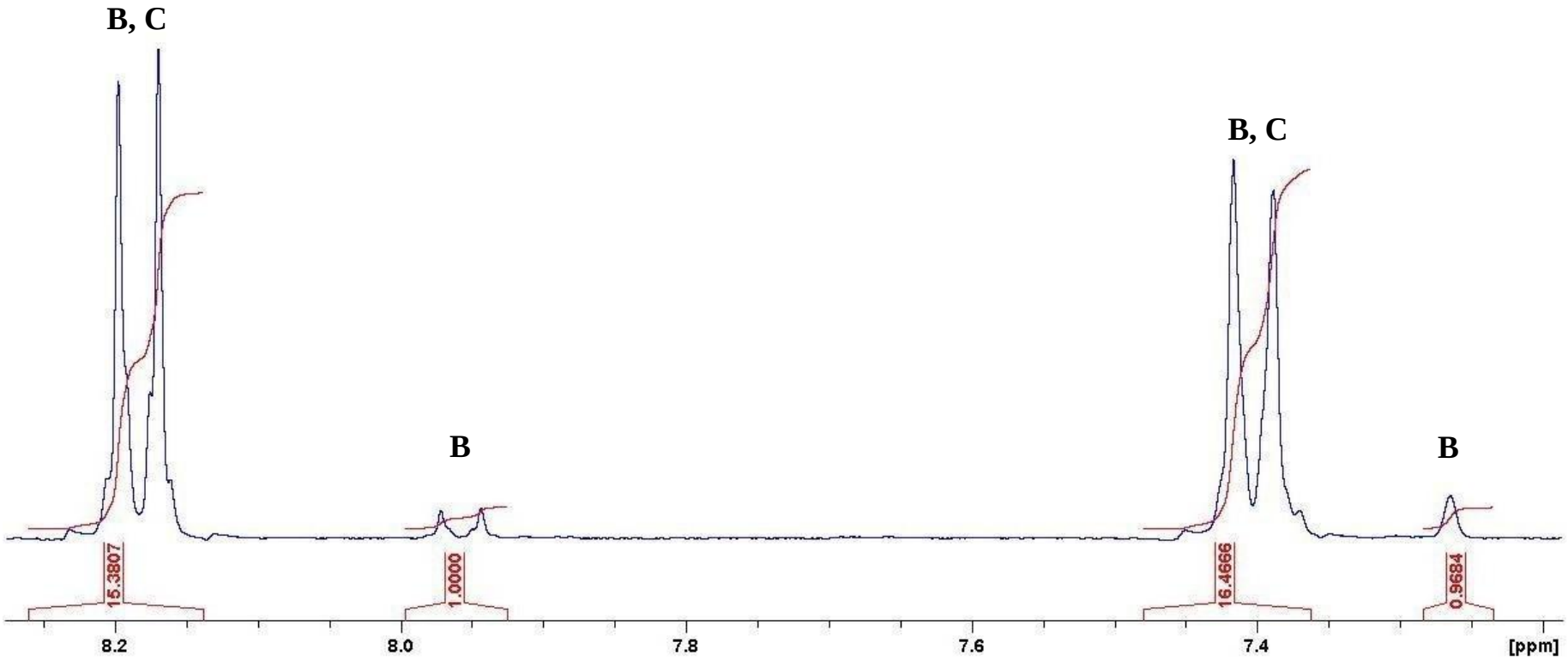


Expanded ^{13}C NMR spectrum of the product mixture obtained in entry h of Table 3 (acetone-d_6)

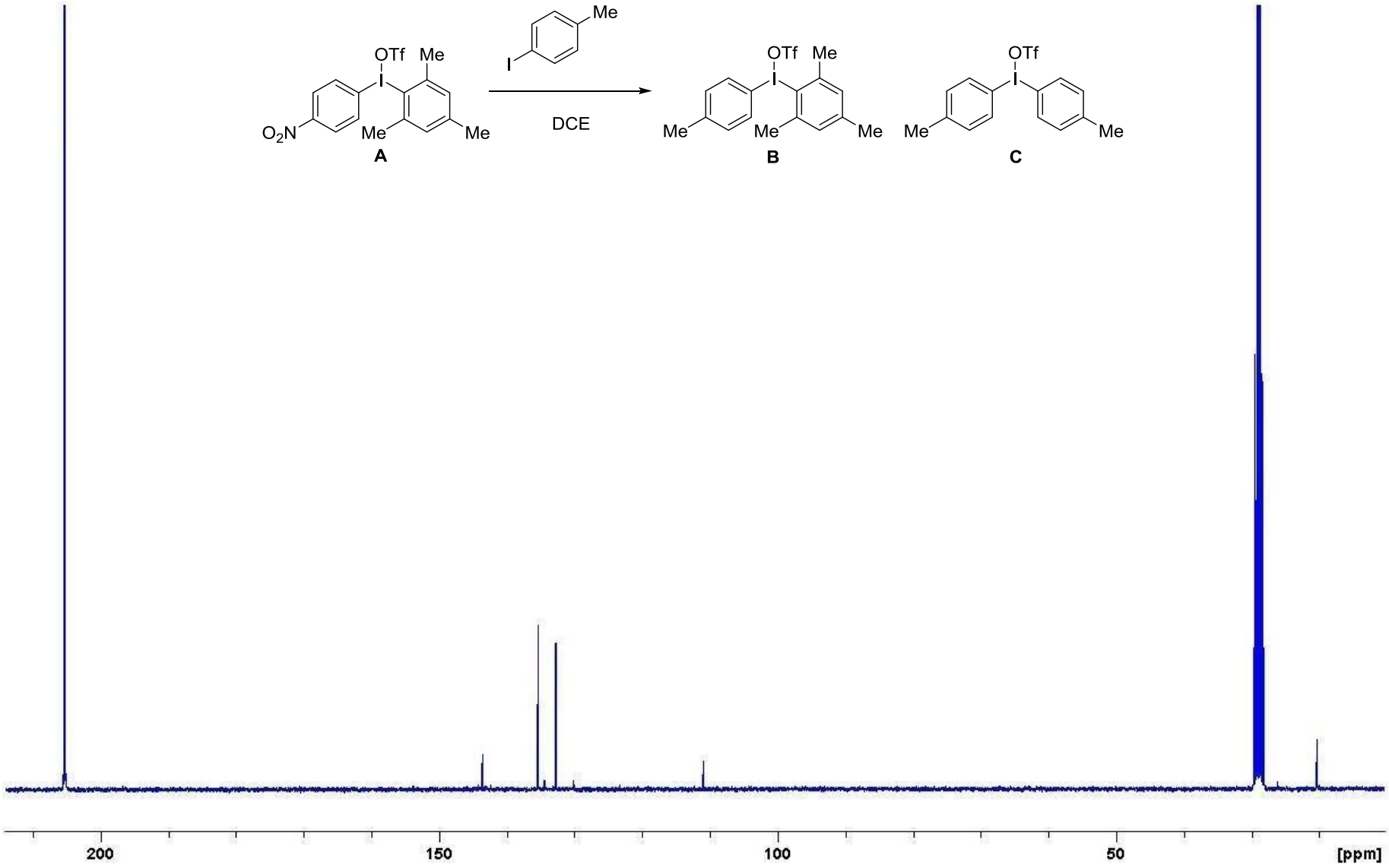


^{1}H NMR spectrum of the product mixture obtained in entry i of Table 3 (acetone-d_6)

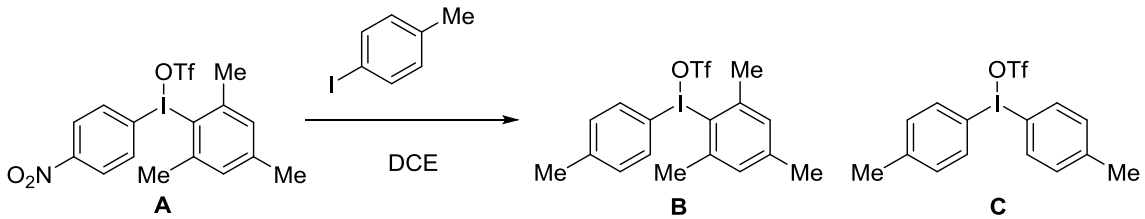


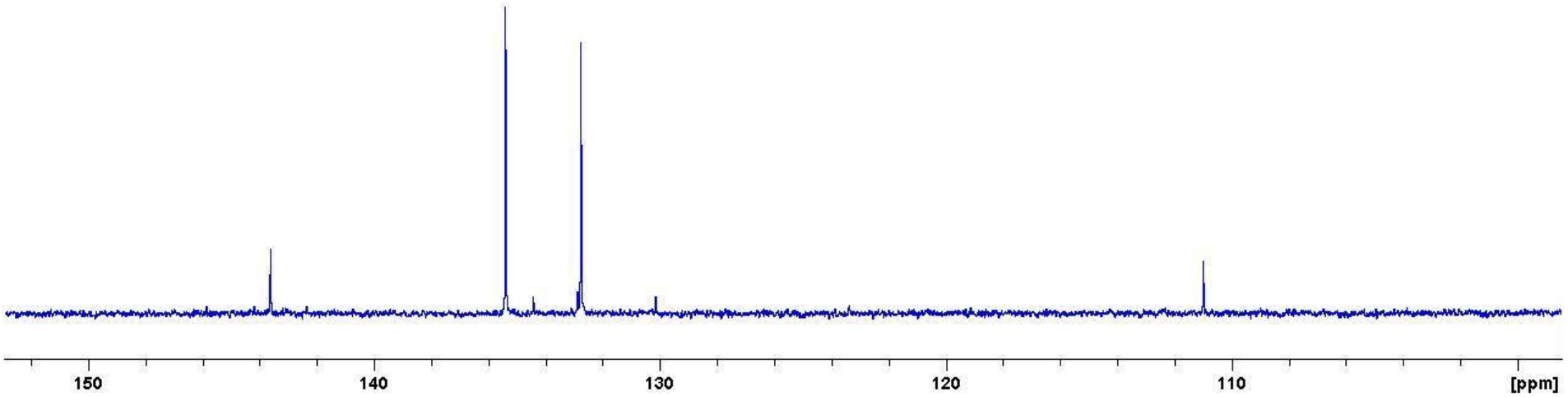


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry i of Table 3 (acetone-d_6)

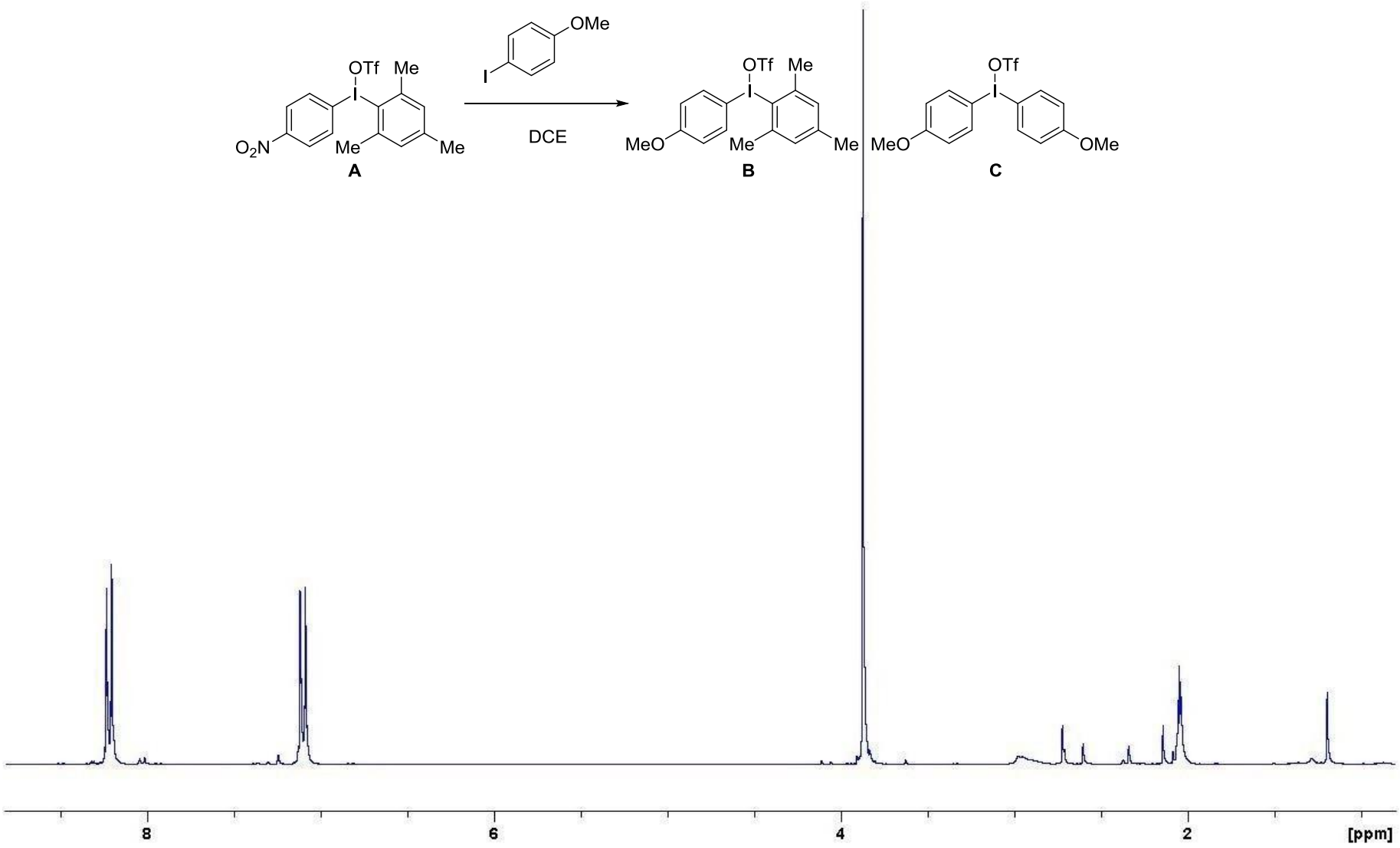


^{13}C NMR spectrum of the product mixture obtained in entry i of Table 3 (acetone-d_6)

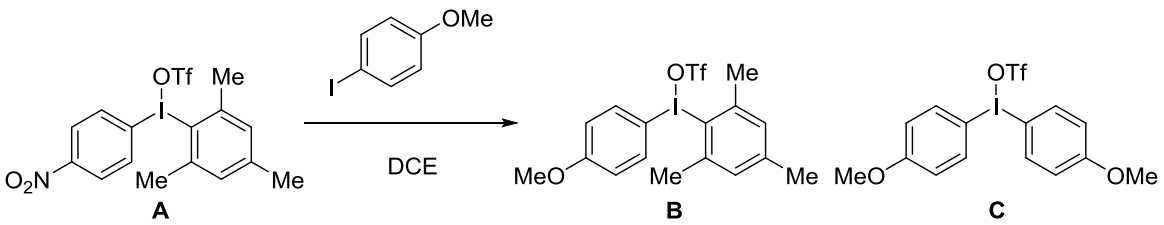


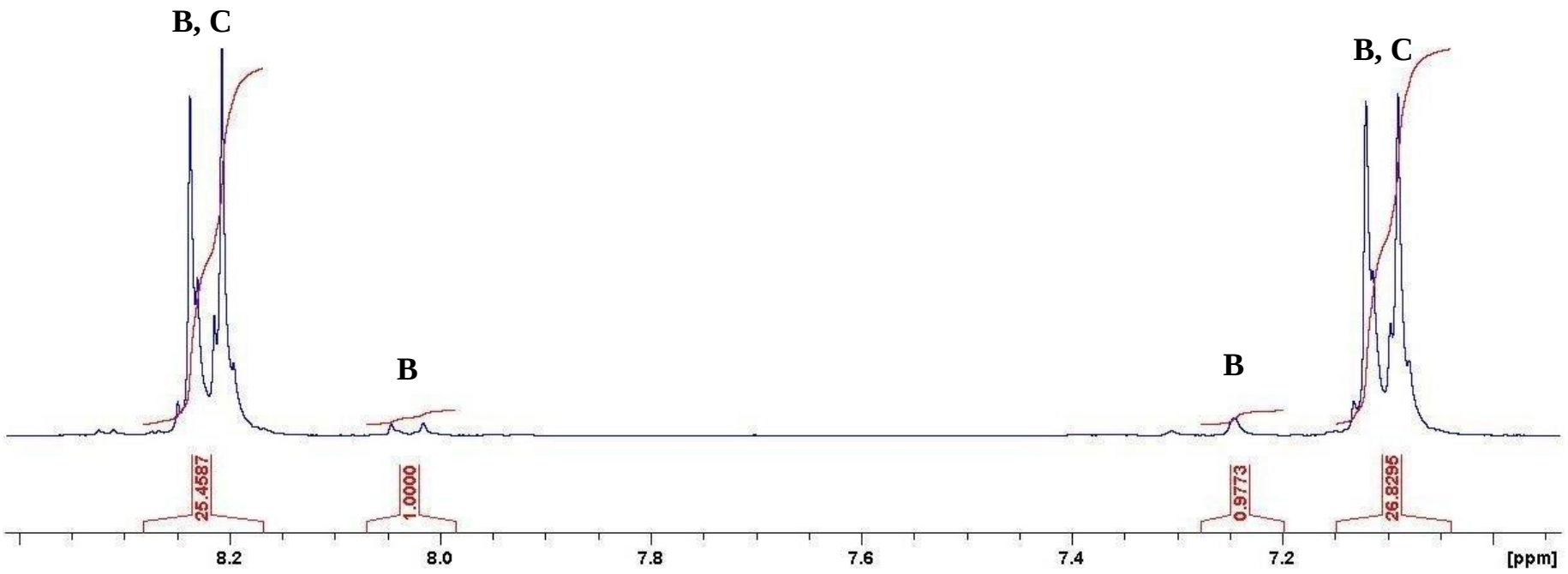


Expanded ^{13}C NMR spectrum of the product mixture obtained in entry i of Table 3 (acetone-d_6)

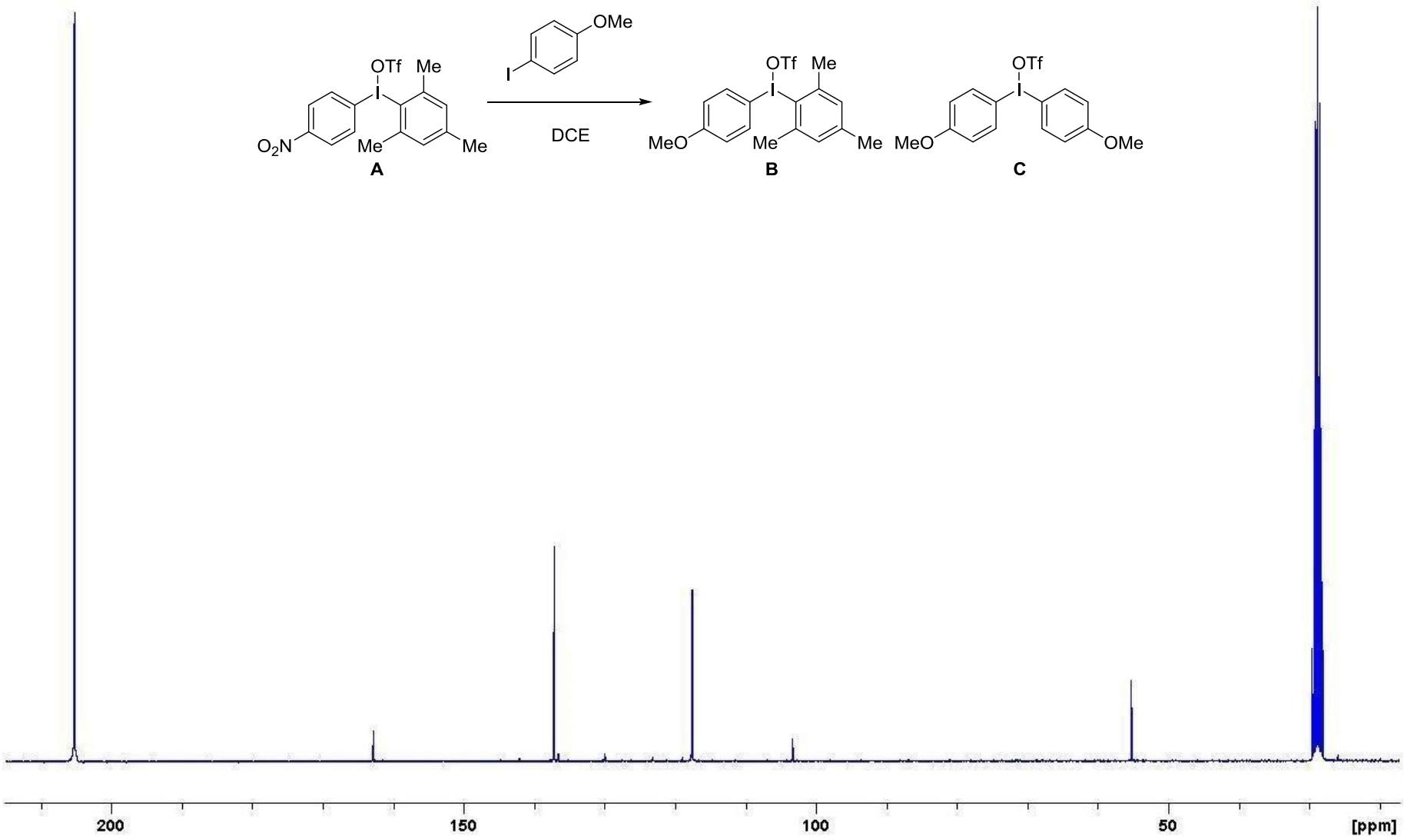


^{1}H NMR spectrum of the product mixture obtained in entry j of Table 3 (acetone-d_6)

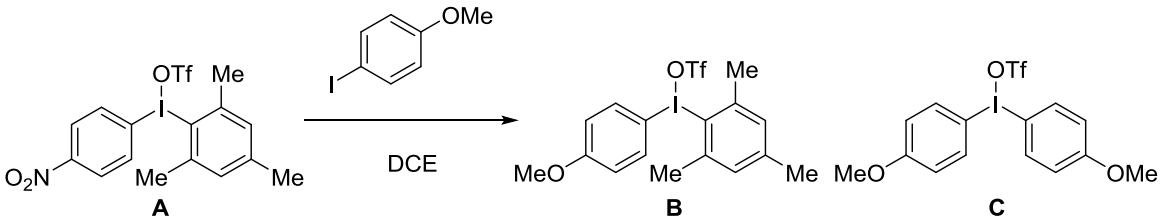


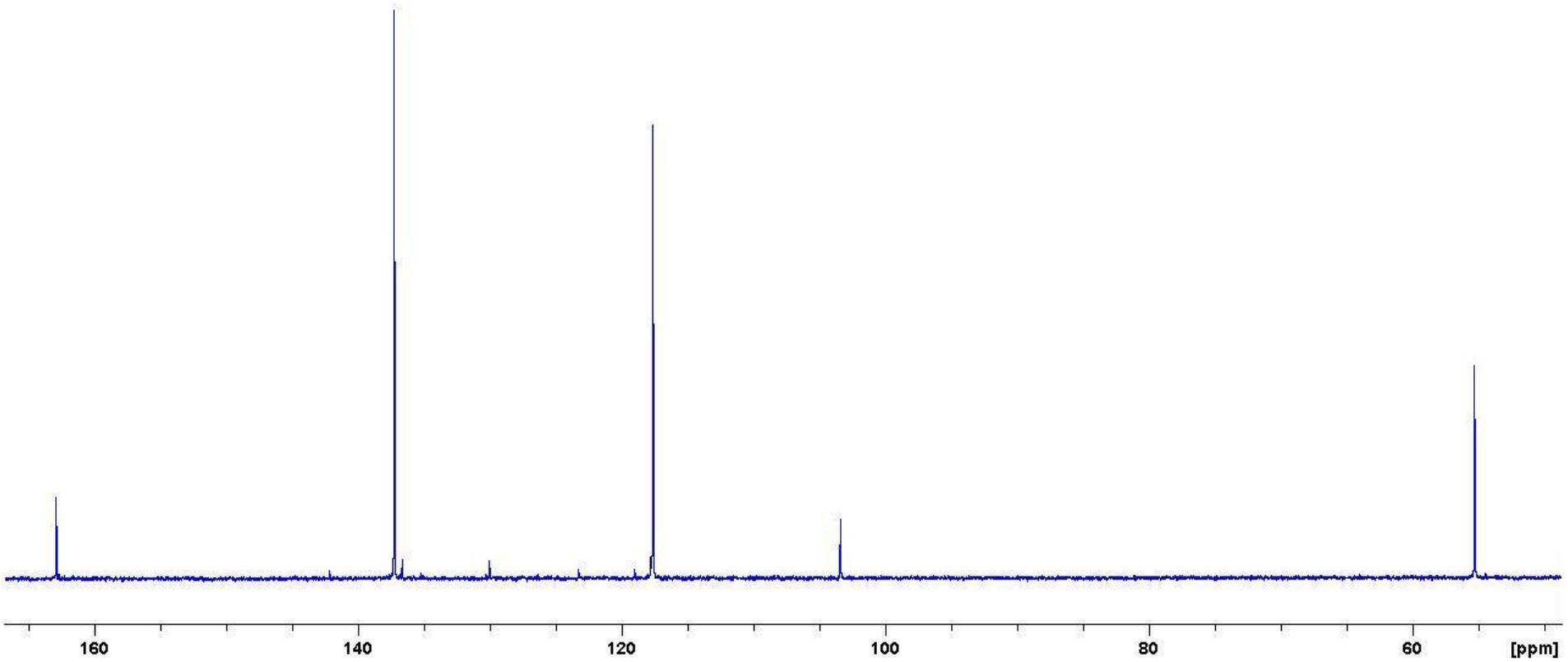


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry j of Table 3 (acetone-d_6)

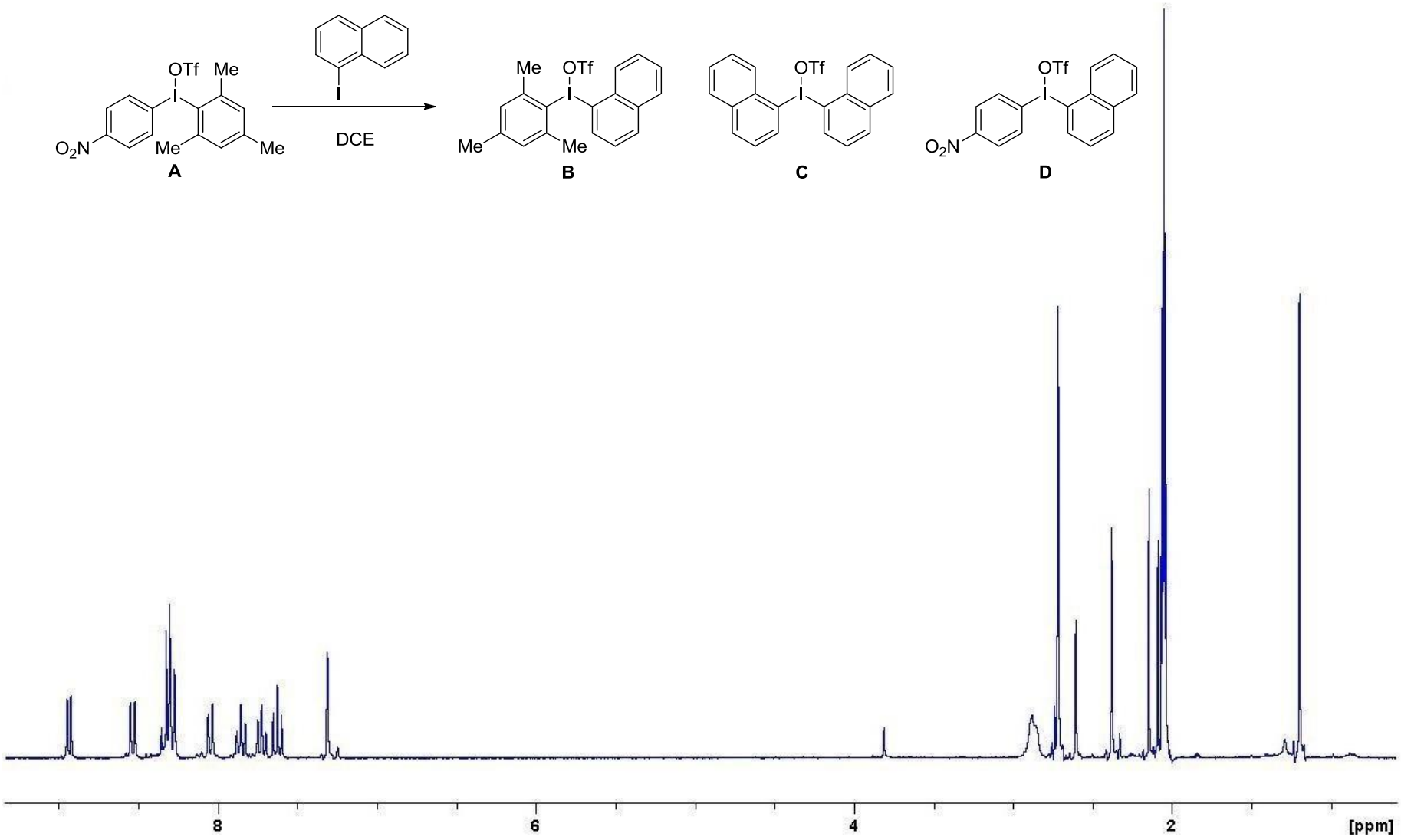


^{13}C NMR spectrum of the product mixture obtained in entry j of Table 3 (acetone-d_6)

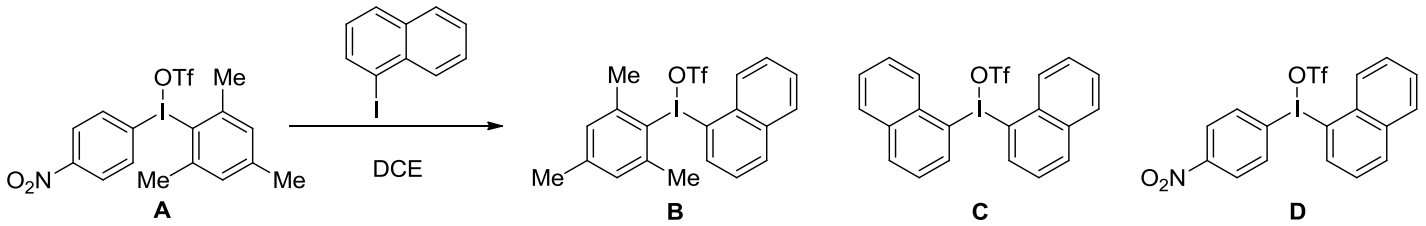


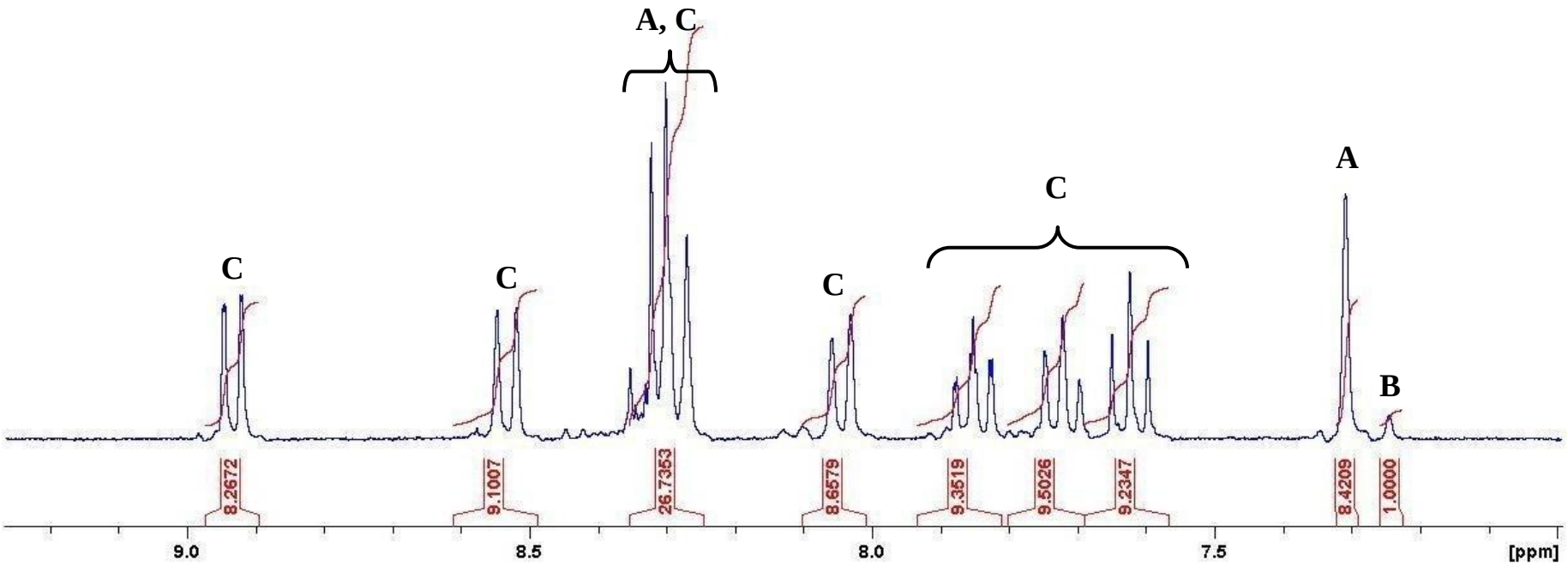


Expanded ^{13}C NMR spectrum of the product mixture obtained in entry j of Table 3 (acetone-d_6)

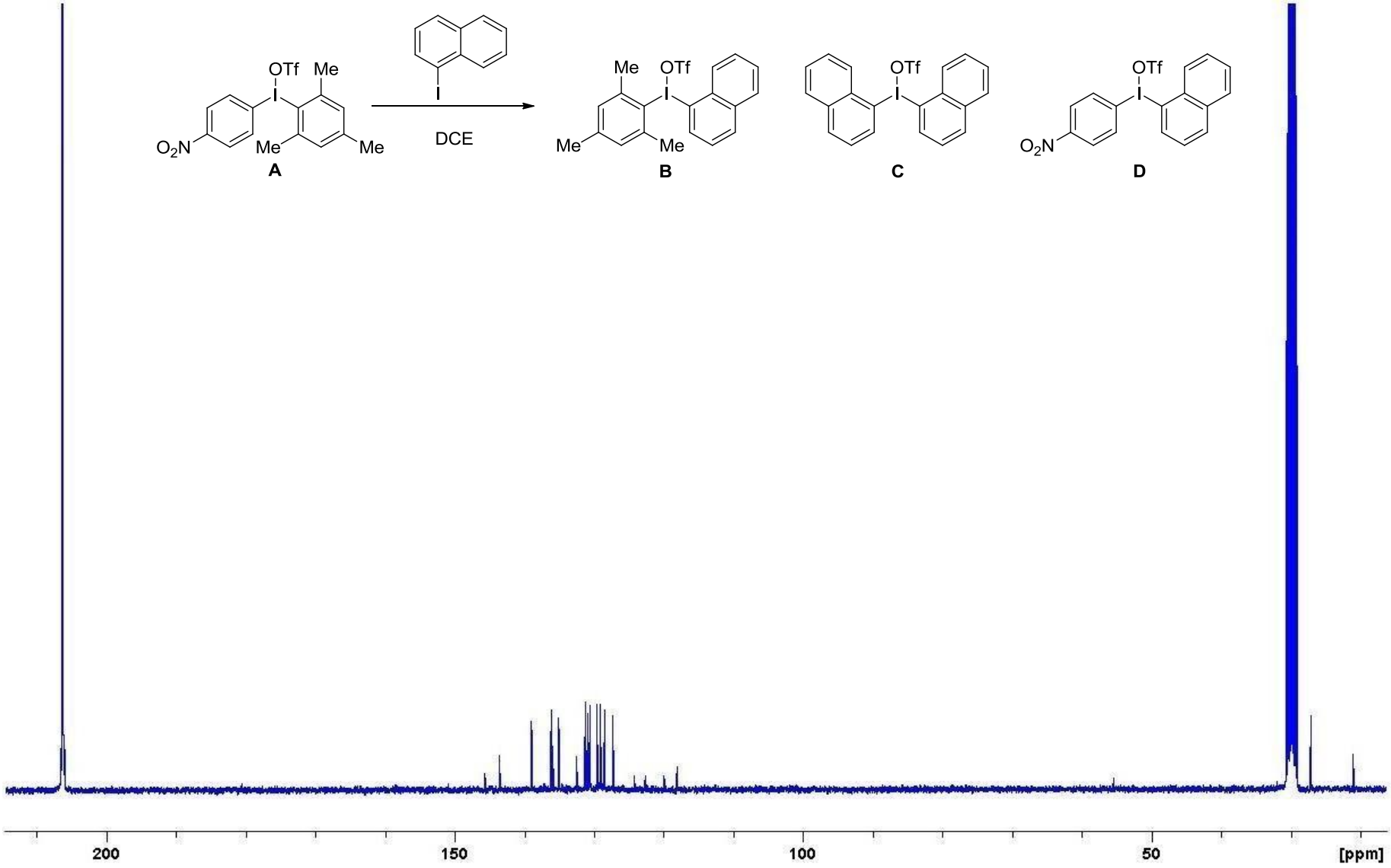


^{1}H NMR spectrum of the product mixture obtained in entry k of Table 3 (acetone-d_6)

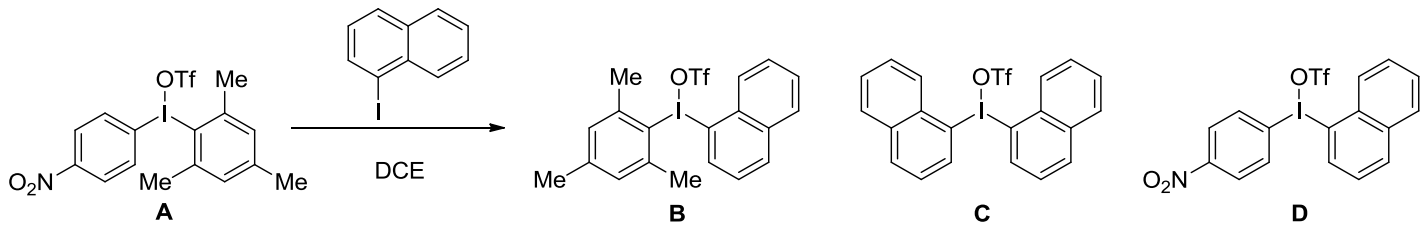


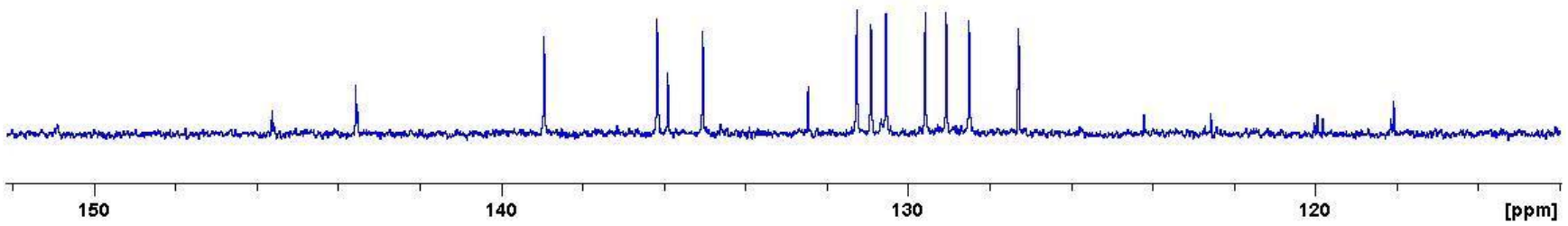


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry k of Table 3 (acetone-d_6)

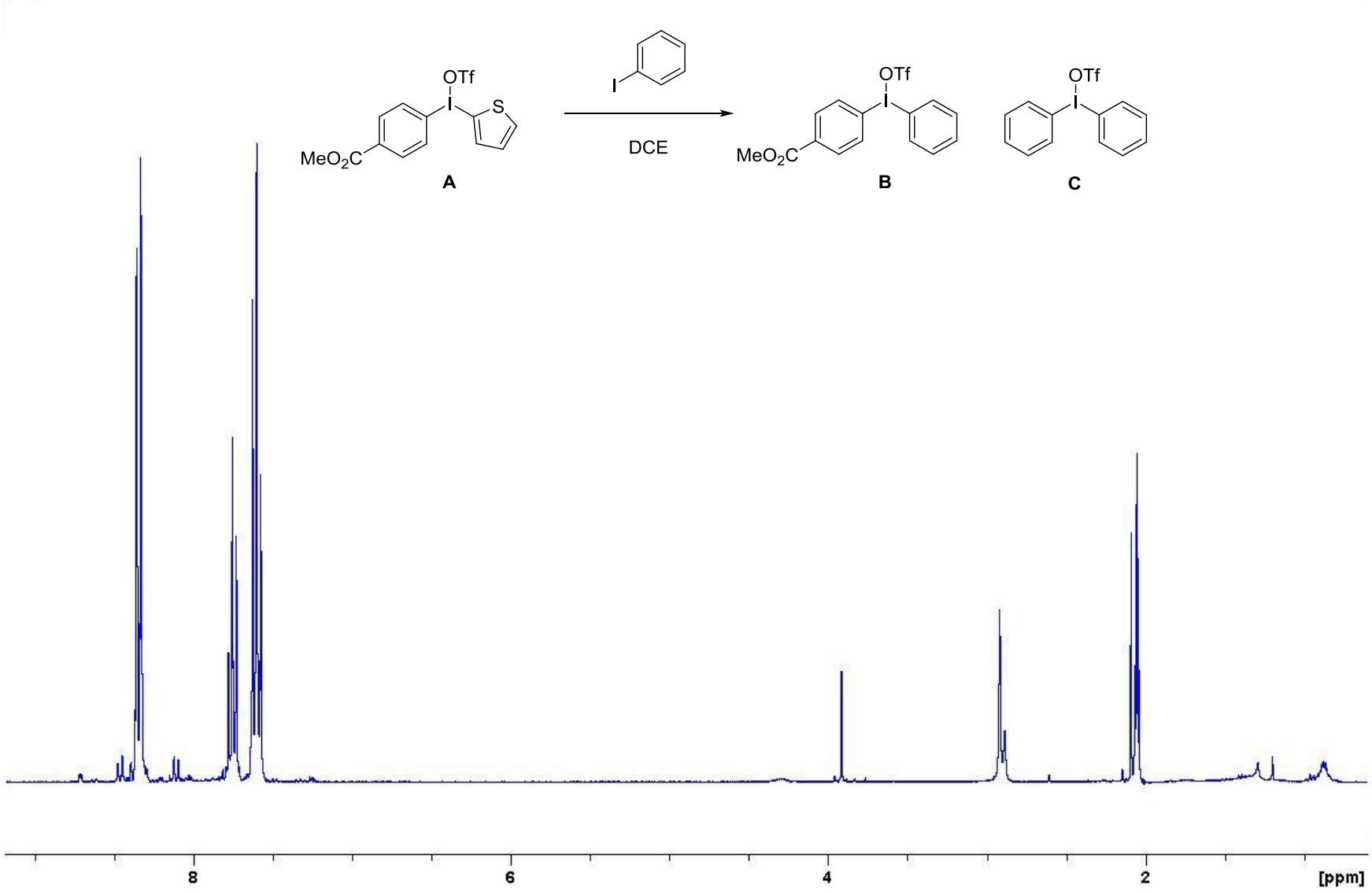


^{13}C NMR spectrum of the product mixture obtained in entry k of Table 3 (acetone-d_6)

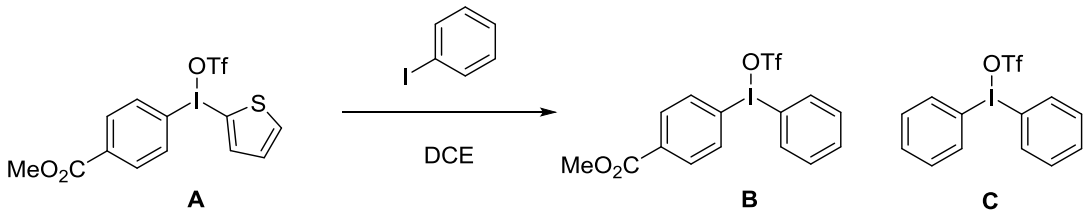


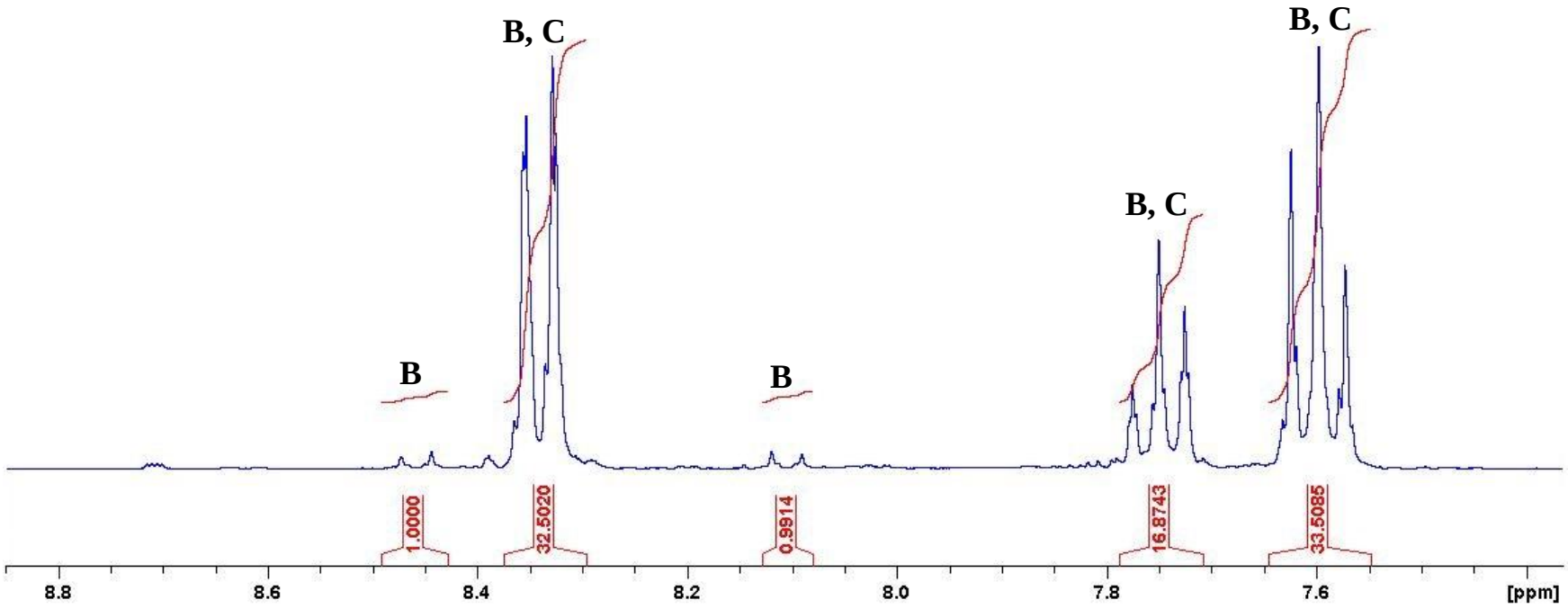


Expanded ^{13}C NMR spectrum of the product mixture obtained in entry k of Table 3 (acetone-d_6)

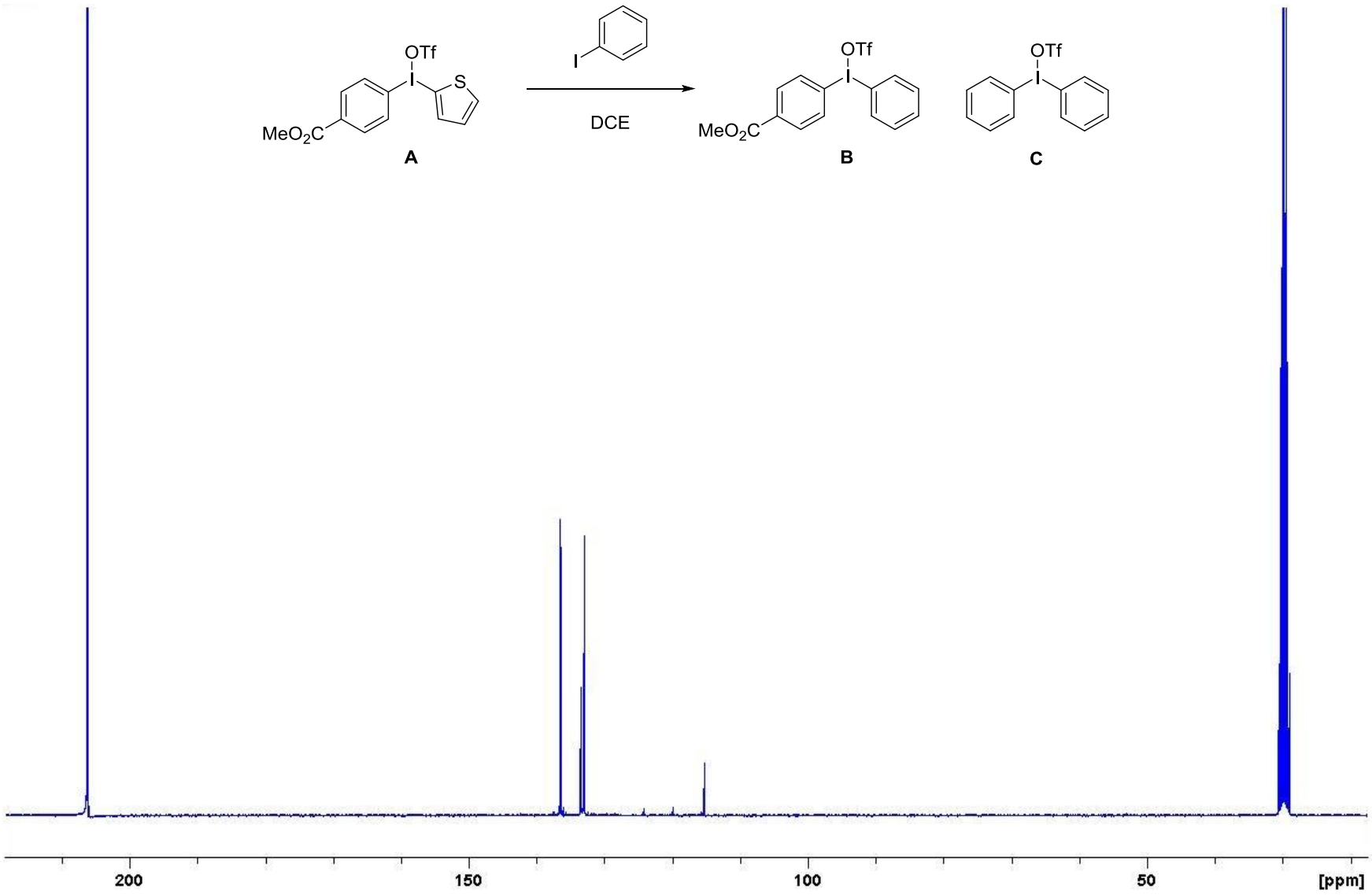


^{1}H NMR spectrum of the product mixture obtained in entry a of Table 4 (acetone-d_6)

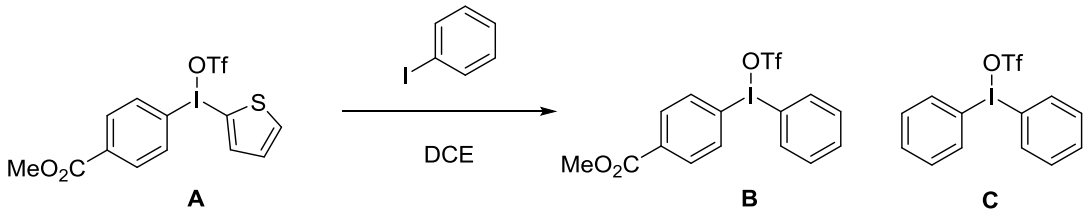


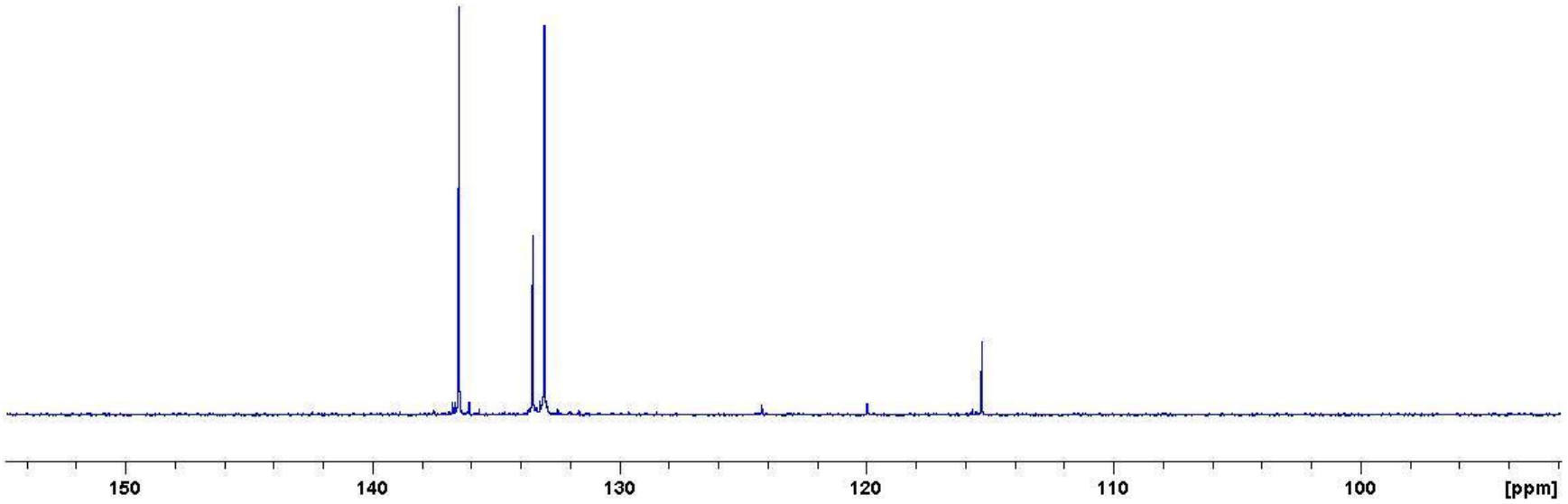


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry a of Table 4 (acetone-d_6)

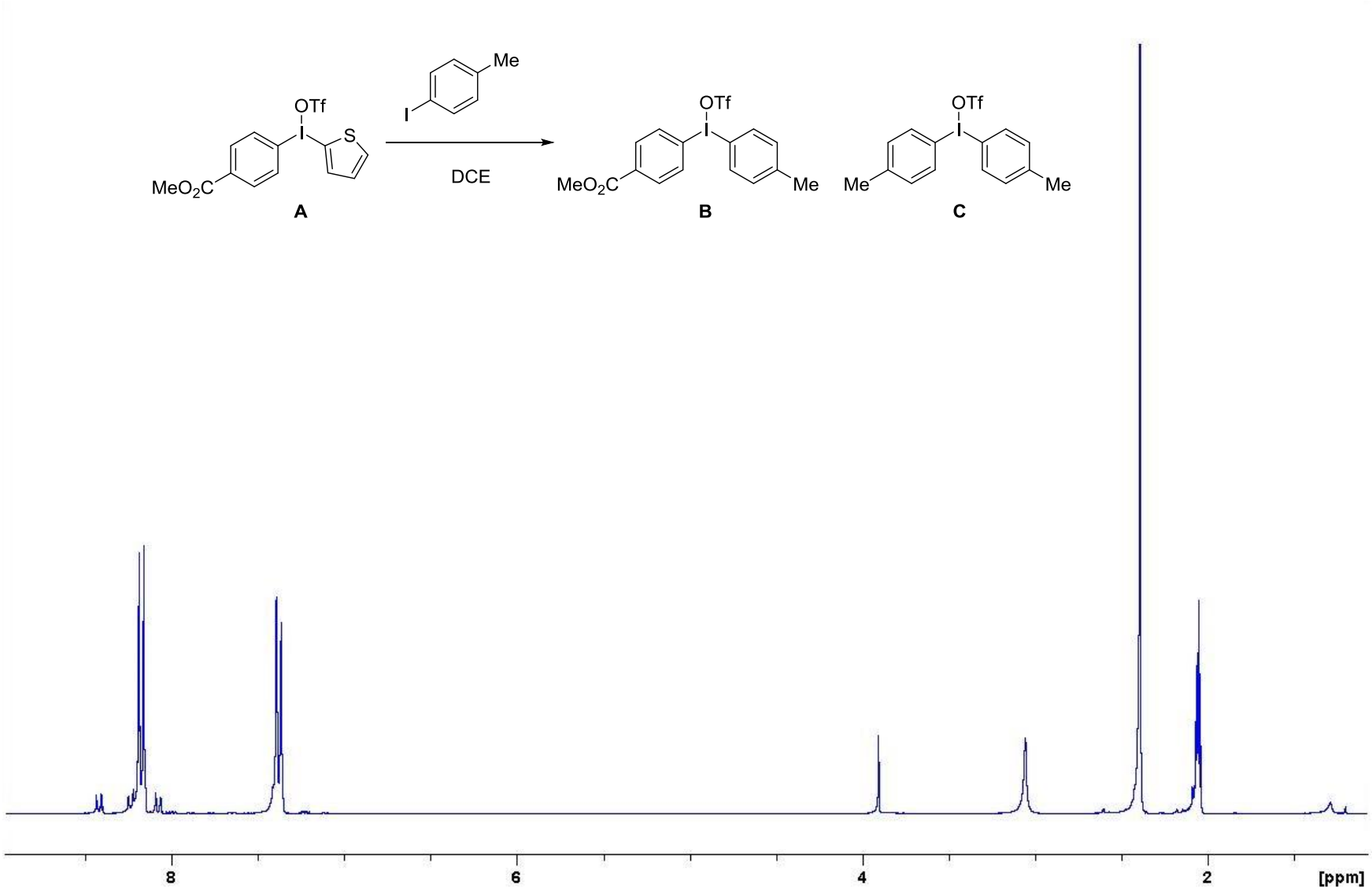


^{13}C NMR spectrum of the product mixture obtained in entry a of Table 4 (acetone-d_6)

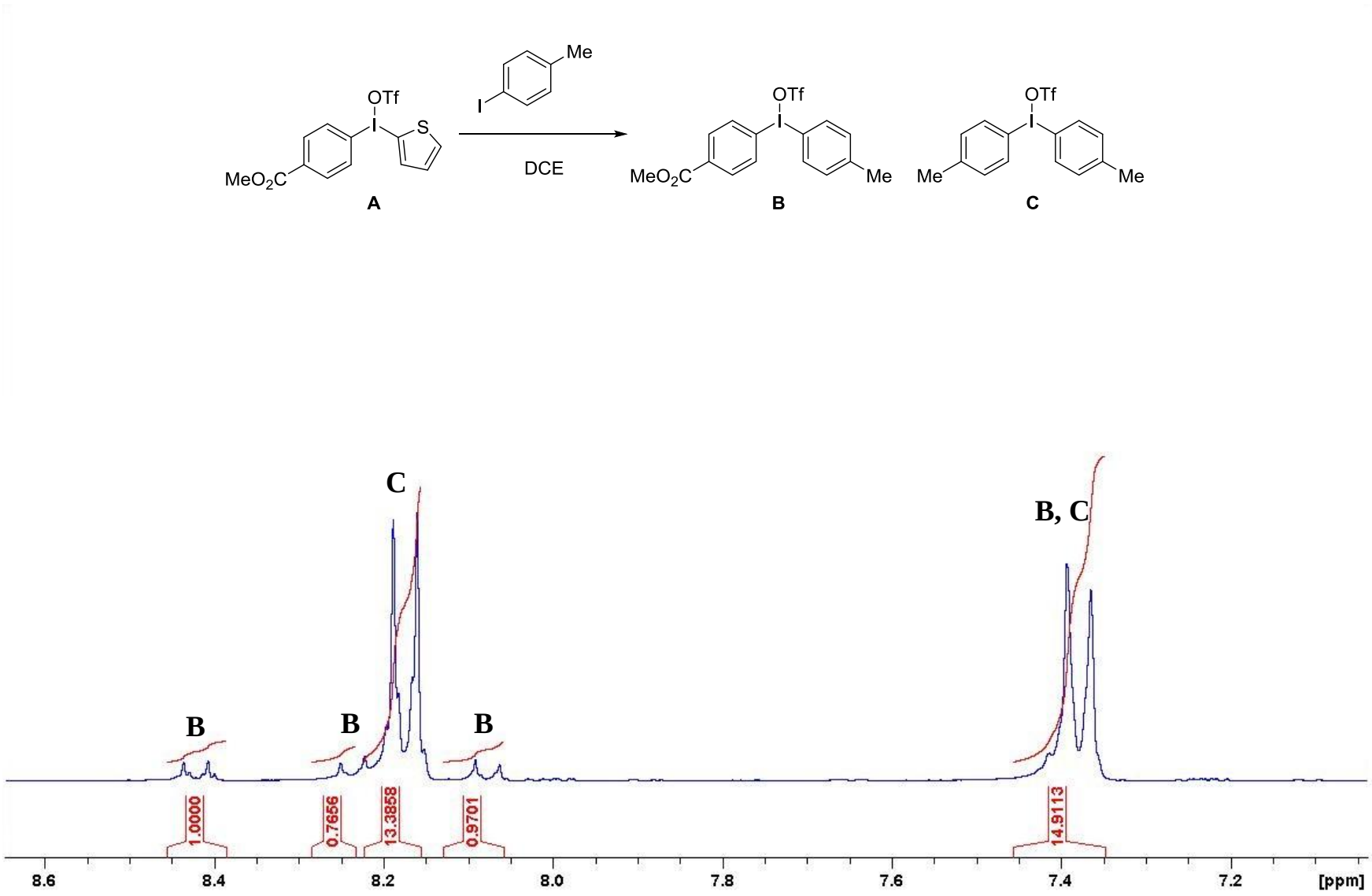




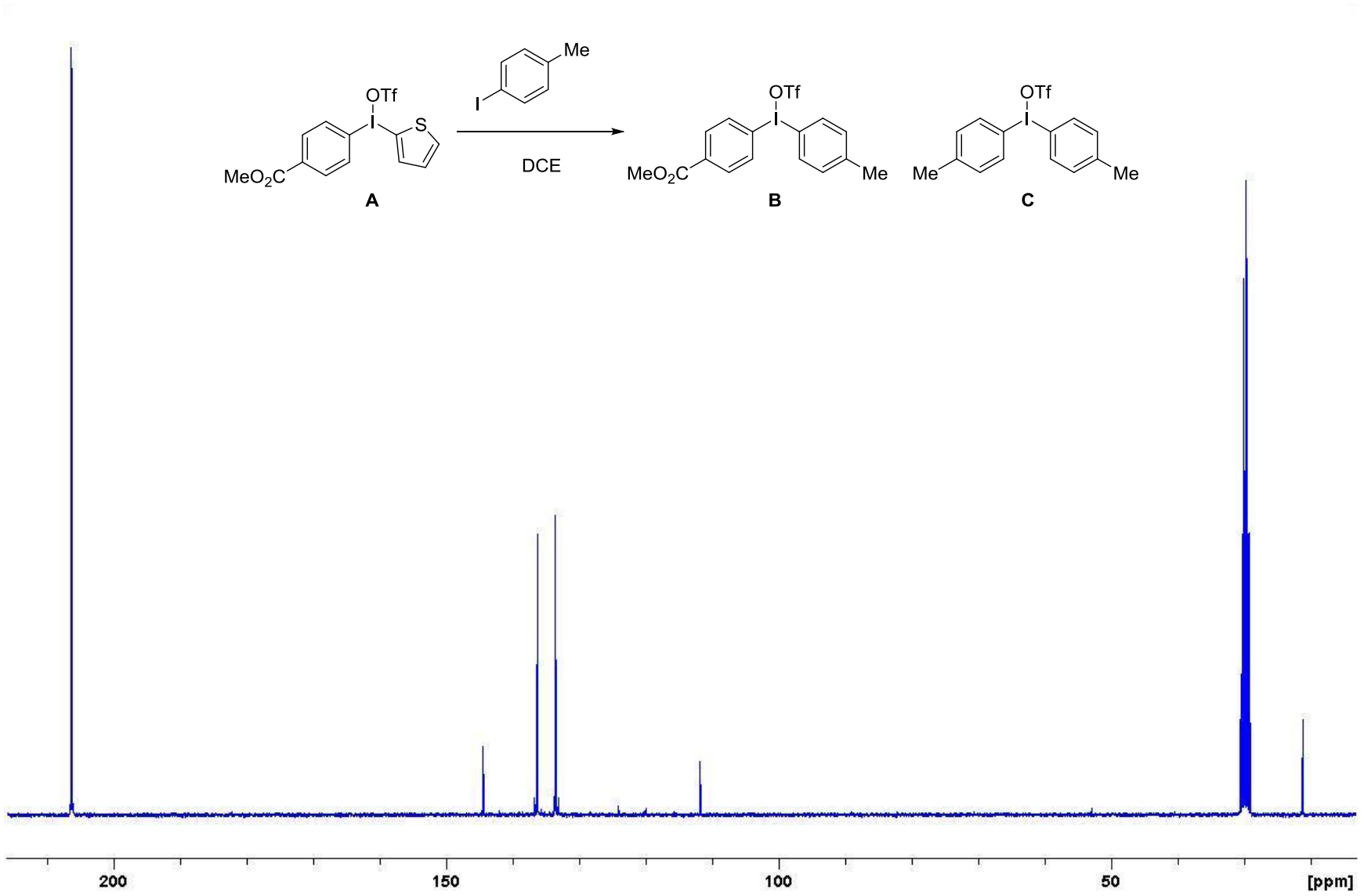
Expanded ^{13}C NMR spectrum of the product mixture obtained in entry a of Table 4 (acetone-d_6)



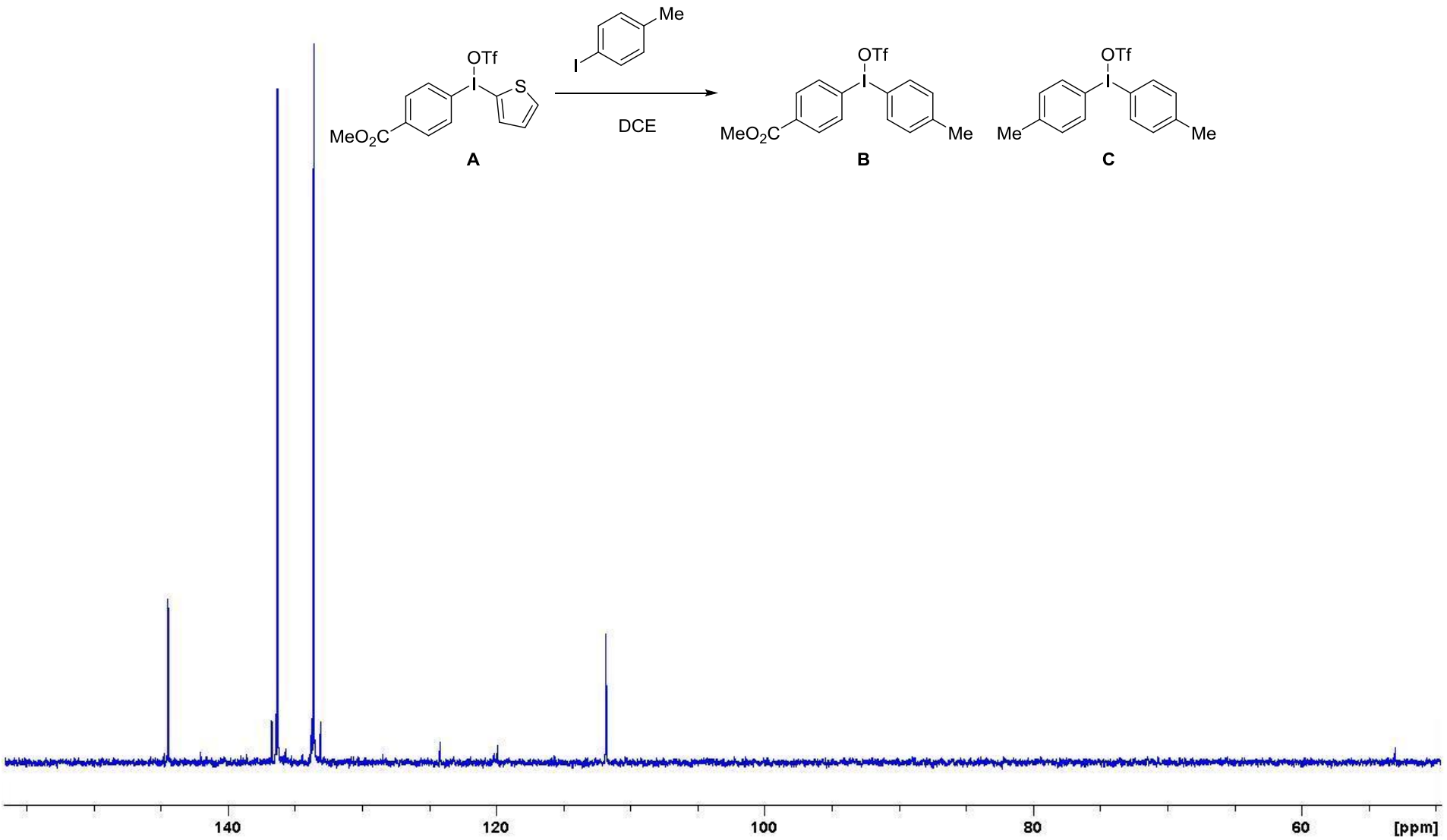
^{1}H NMR spectrum of the product mixture obtained in entry b of Table 4 (acetone-d_6)



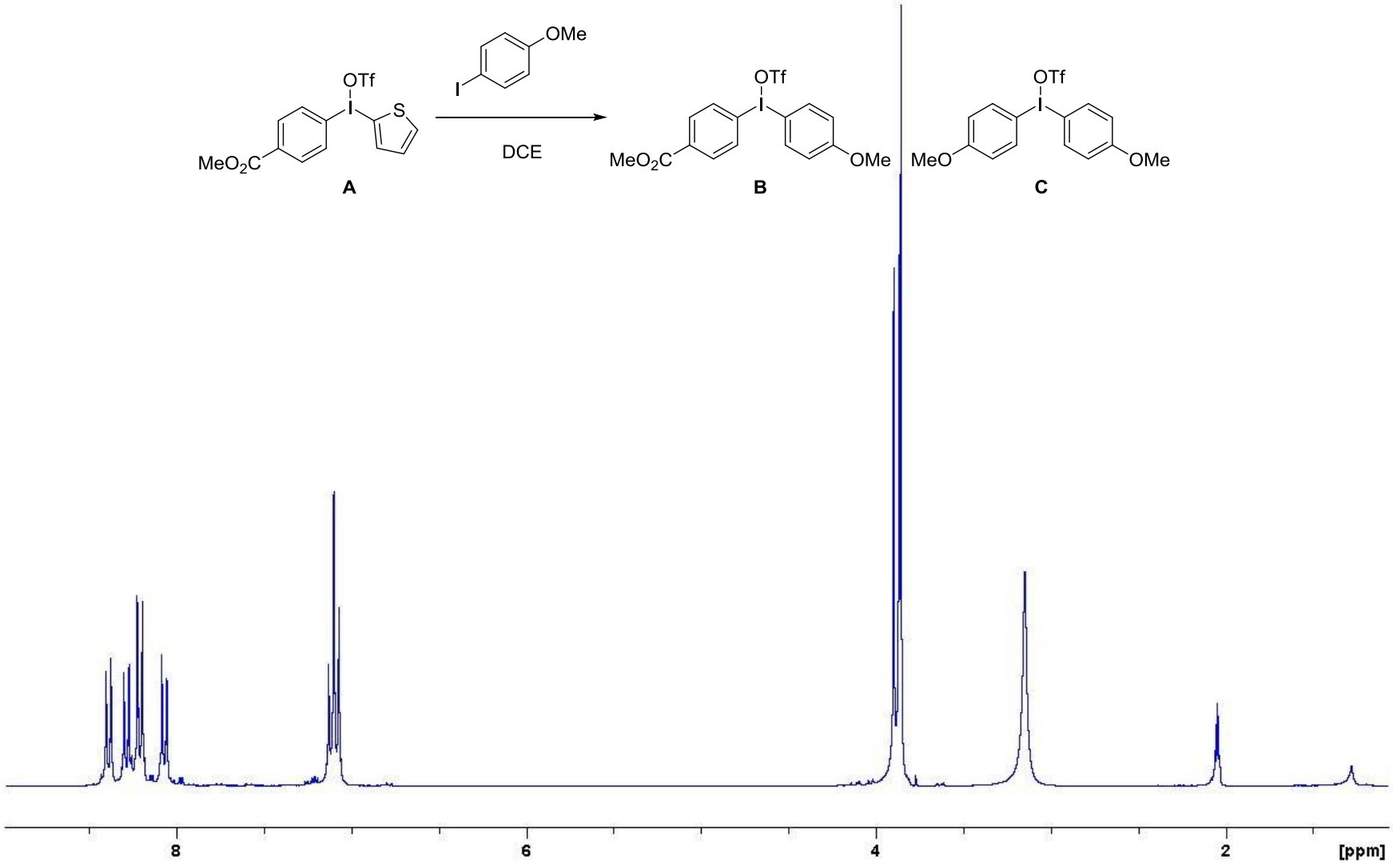
Expanded ^{1}H NMR spectrum of the product mixture obtained in entry b of Table 4 (acetone-d_6)



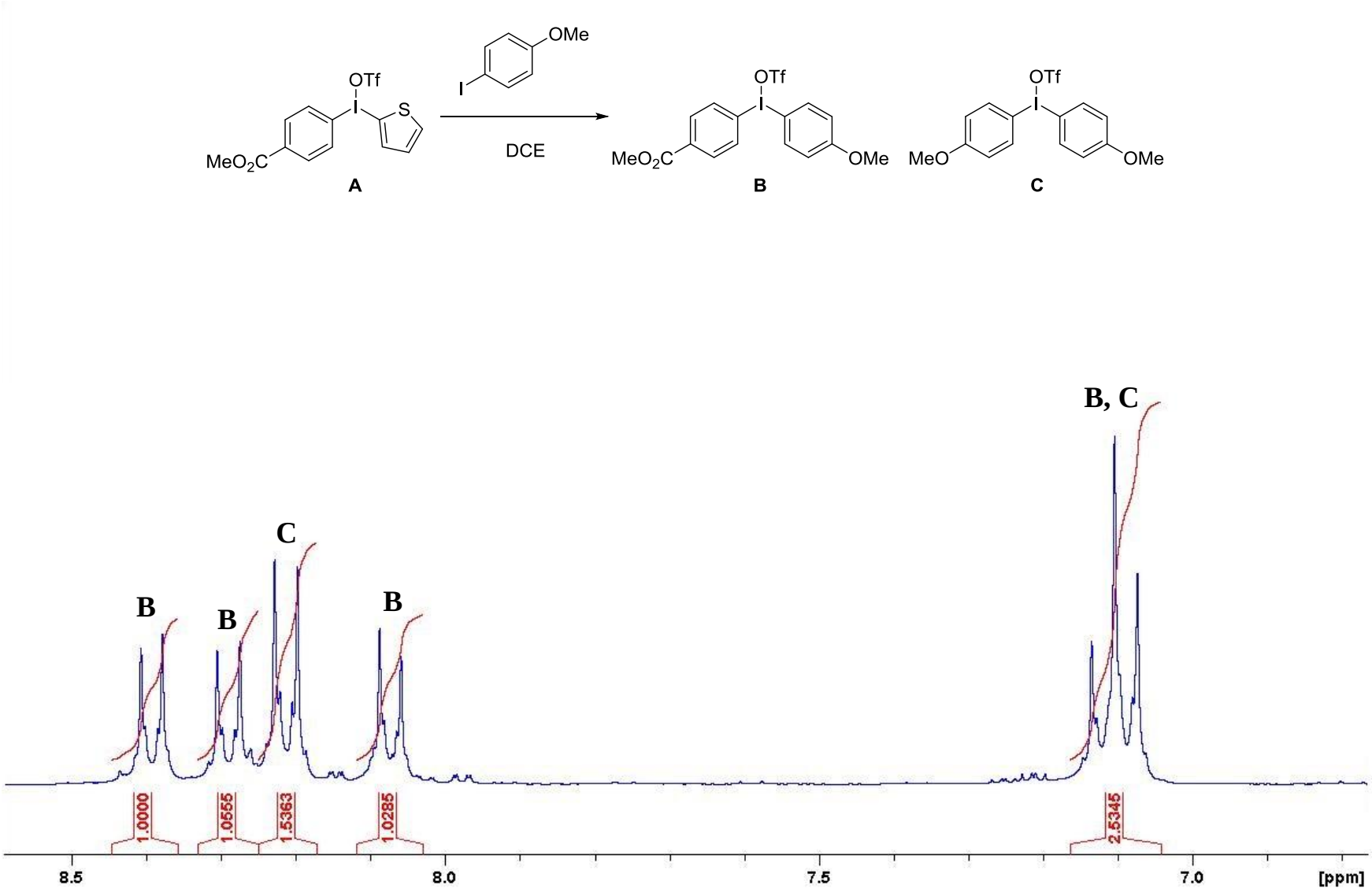
^{13}C NMR spectrum of the product mixture obtained in entry b of Table 4 (acetone-d_6)



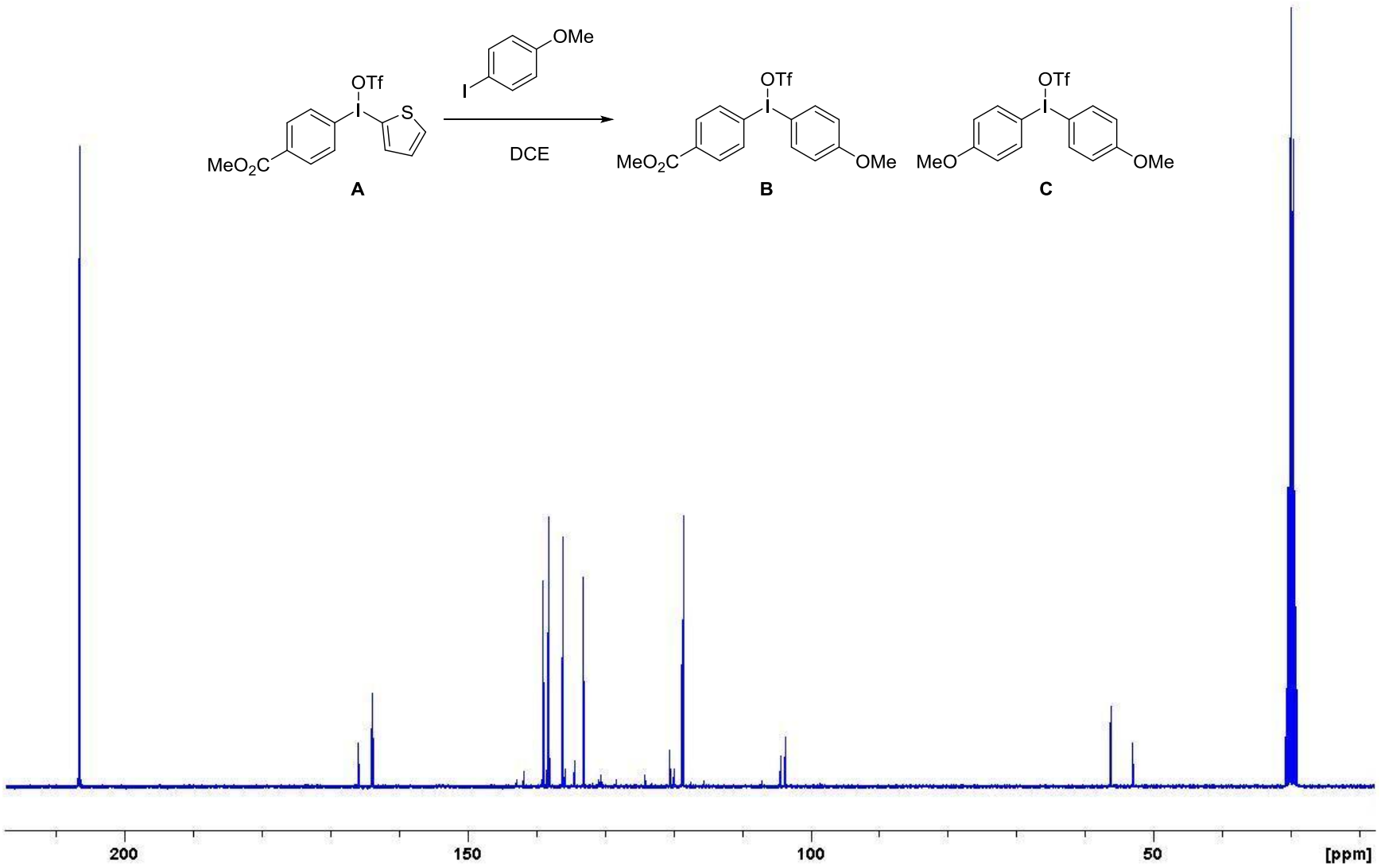
Expanded ^{13}C NMR spectrum of the product mixture obtained in entry b of Table 4 (acetone-d_6)



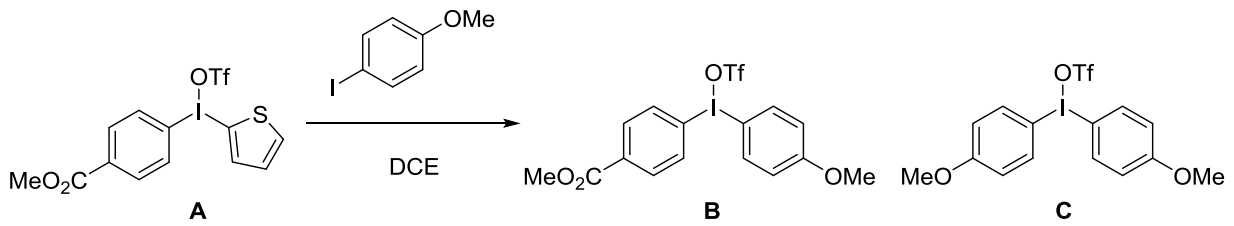
^{1}H NMR spectrum of the product mixture obtained in entry c of Table 4 (acetone-d_6)

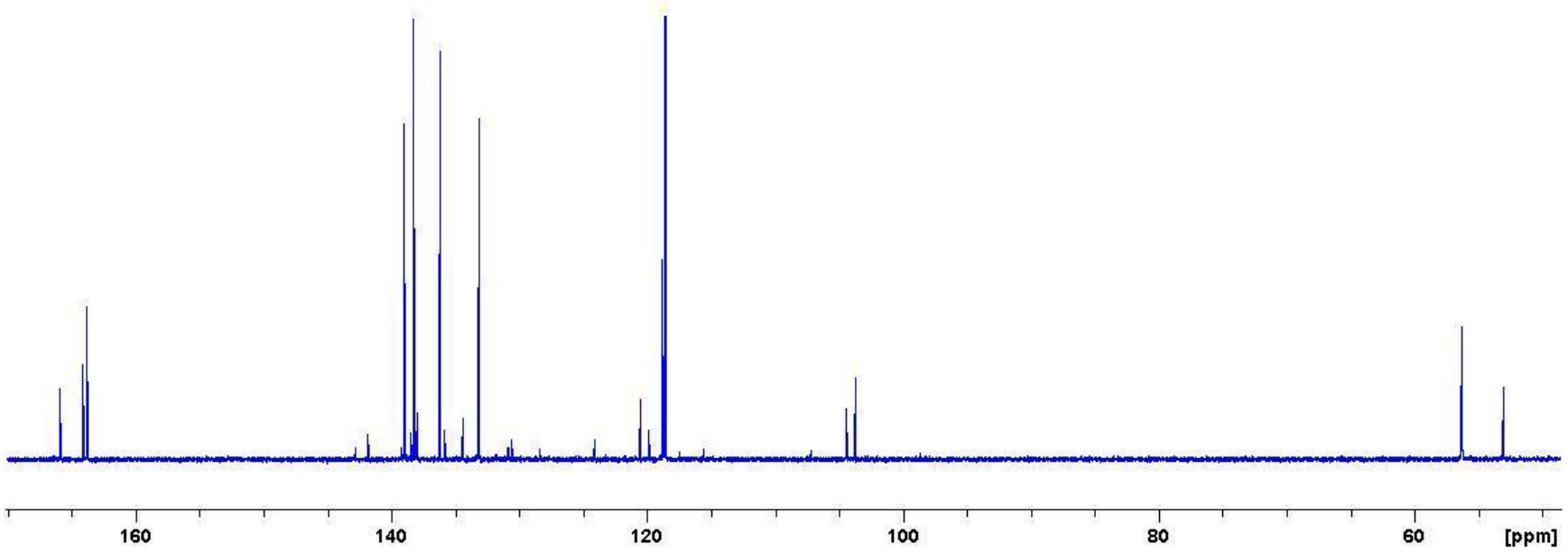


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry c of Table 4 (acetone-d_6)

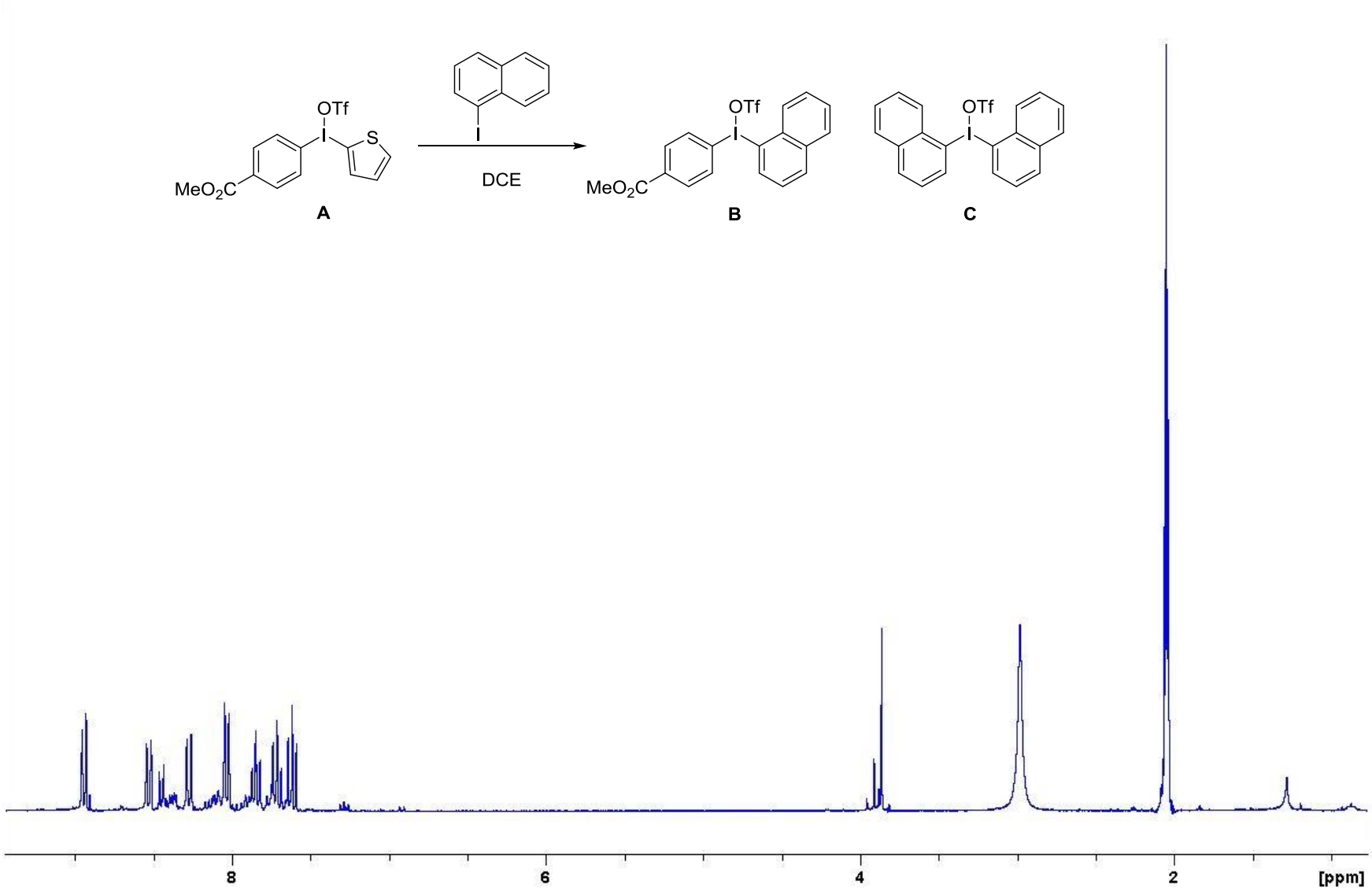


^{13}C NMR spectrum of the product mixture obtained in entry c of Table 4 (acetone-d_6)

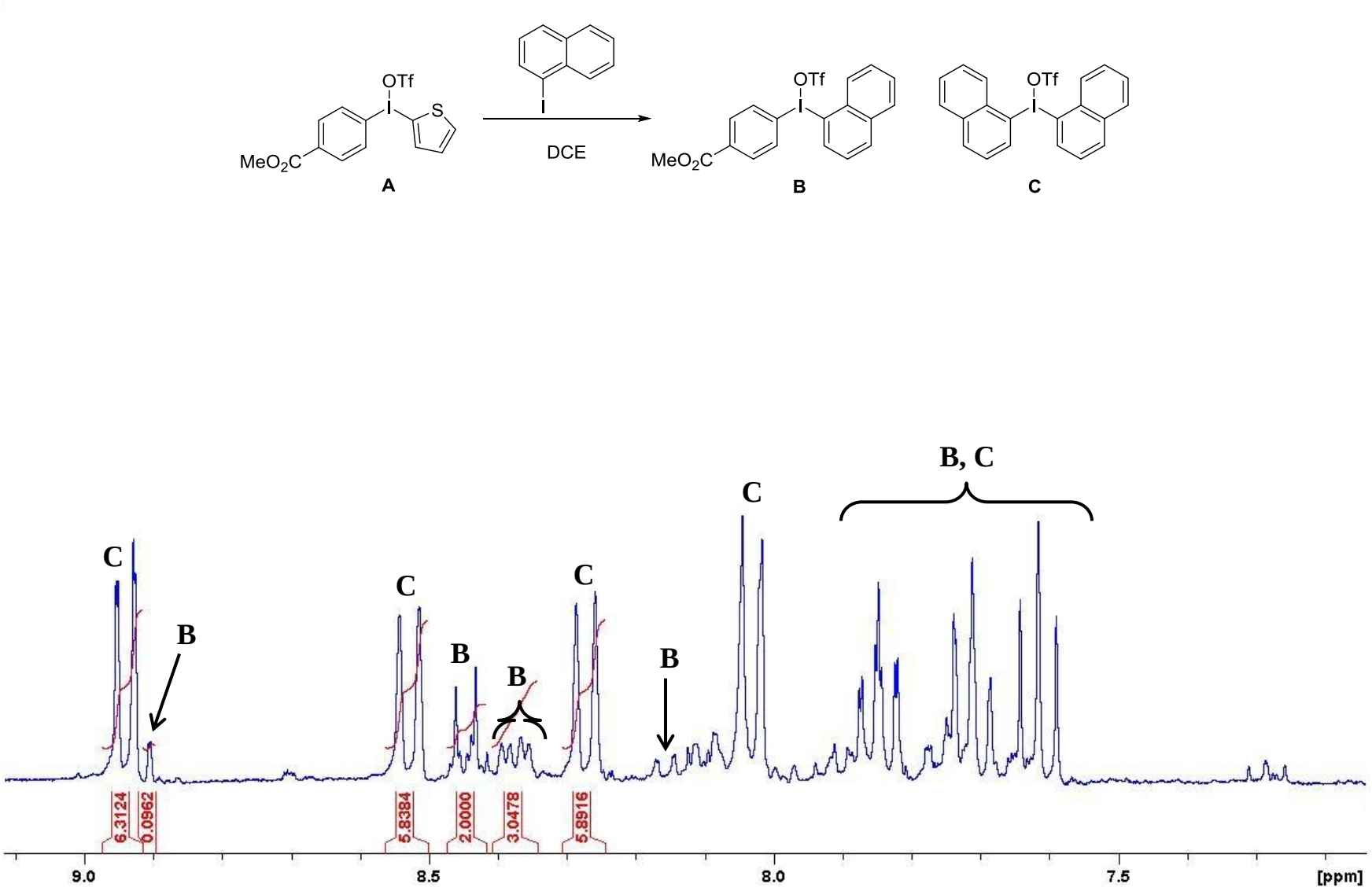




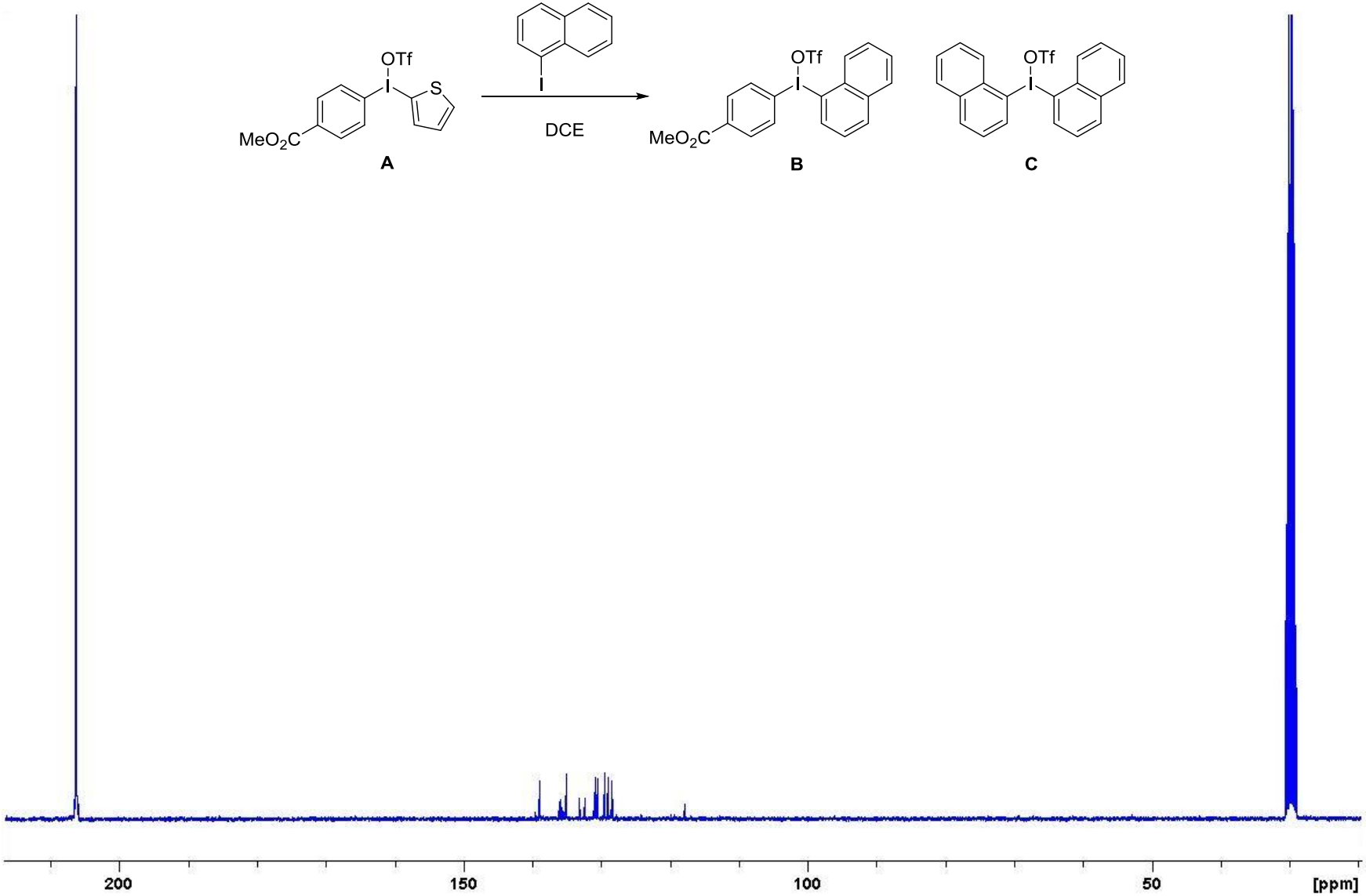
Expanded ^{13}C NMR spectrum of the product mixture obtained in entry c of Table 4 (acetone-d_6)



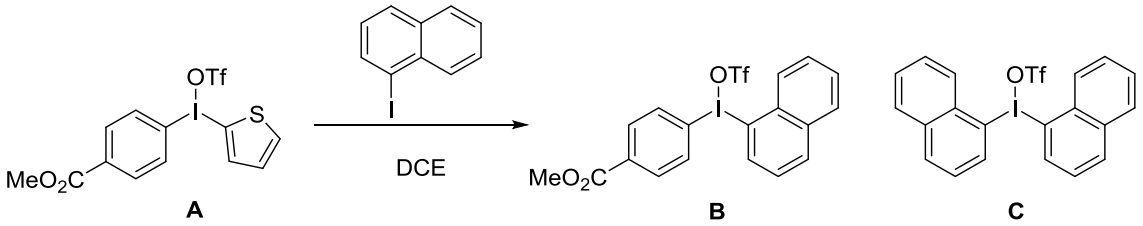
^{1}H NMR spectrum of the product mixture obtained in entry d of Table 4 (acetone-d_6)

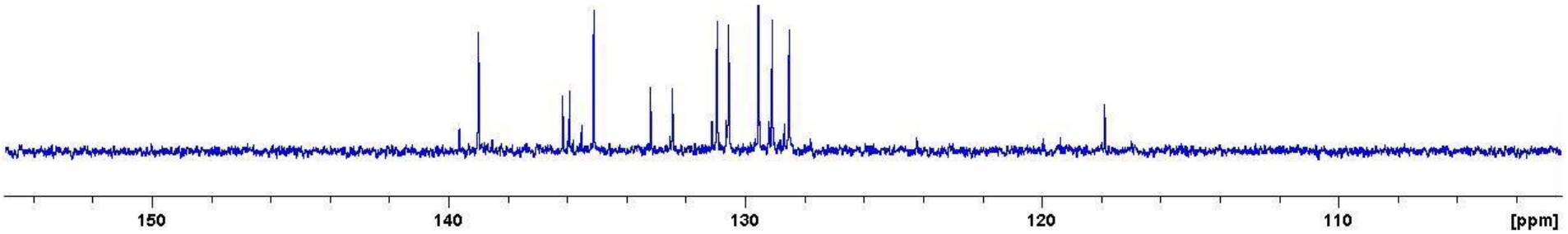


Expanded ^{1}H NMR spectrum of the product mixture obtained in entry d of Table 4 (acetone-d_6)



^{13}C NMR spectrum of the product mixture obtained in entry d of Table 4 (acetone-d_6)





Expanded ^{13}C NMR spectrum of the product mixture obtained in entry d of Table 4 (acetone-d_6)