

Supporting Information

Synthesis of di/tri-substituted carbazoles involving Pd-Mediated Sonogashira coupling of indolyltriflates with aryl acetylenes

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025, Tamil Nadu, India,

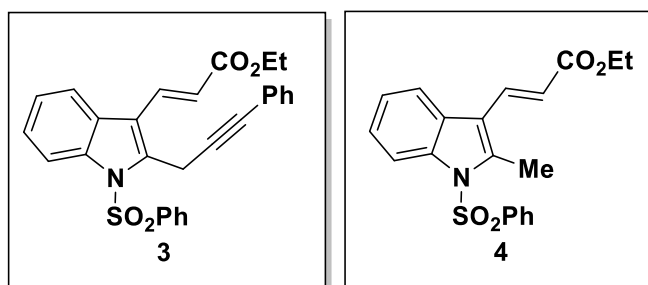
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Experimental Procedure

Attempted synthesis of ethyl (*E*)-3-(2-(3-phenylprop-2-yn-1-yl)-1-(phenylsulfonyl)-1*H*-indol-3-yl)acrylate **3** from 2-bromomethyl-3-vinylindole **1**



Using Pd(PPh₃)₄ condition

To a solution of 2-bromomethylindole¹ **1** (0.25 g, 0.45 mmol) in dry THF (10 mL), phenylacetylene (91 mg, 0.89 mmol), Pd(PPh₃)₄ (52 mg, 0.04 mmol), and K₂CO₃ (62 mg, 0.45 mmol) were added and refluxed for 8 h. Then, the reaction mixture was dissolved in EA (20 mL). It was then diluted with water (30 mL), and the combined organic layer was separated. The aqueous layer was extracted with EA (2 x 10 mL), and the combined organic extract was dried (Na₂SO₄). The solvent was removed under *vacuo* and triturated with MeOH (5 mL), to give the de-brominated indole **4** (165 mg, 80%).

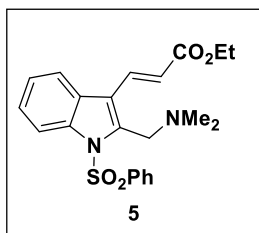
Using LHMDS & CuI condition

To a solution of 2-bromomethylindole¹ **1** (0.2 g, 0.56 mmol) in dry toluene (5 mL), phenylacetylene (0.17 g, 1.67 mmol) copper iodide (11 mg, 0.05 mmol) and LHMDS (0.11 g, 0.67 mmol) were added, the reaction mixture was stirred at 90 °C for 8 h. The reaction mixture was then filtered by celite pad and washed with EA (20 mL). The filtrate was quenched with crushed ice (100 g) containing NH₄Cl (2 g), and the combined organic layer was separated. The aqueous layer was extracted with EA (2 x 10 mL), and the combined organic extract was dried (Na₂SO₄). The solvent was removed under *vacuo* and triturated with MeOH (5 mL), to give the de-brominated indole **4** (157 mg, 95%).

Ethyl (*E*)-3-(2-methyl-1-(phenylsulfonyl)-1*H*-indol-3-yl)acrylate **4**¹

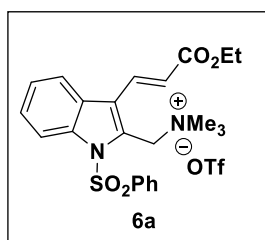
¹H NMR (300 MHz, CDCl₃): δ 8.29 (d, *J* = 8.7 Hz, 1 H), 7.80-7.87 (m, 4 H), 7.31- 7.60 (m, 5 H), 6.53 (d, *J* = 16.2 Hz, 1 H), 4.29 (q, *J* = 7.2 Hz, 2 H), 2.74 (s, 3 H), 1.36 (t, *J* = 7.2 Hz, 3 H) ppm; ¹³C NMR (75 MHz, CDCl₃): δ 167.3, 138.9, 136.7, 135.4, 134.1, 129.5, 127.5, 126.4, 124.9, 119.9, 118.7, 116.1, 114.7, 60.5, 14.4, 13.2 ppm.

Ethyl (*E*)-3-(2-((dimethylamino)methyl)-1-(phenylsulfonyl)-1*H*-indol-3-yl)acrylate **5**



To a solution of 2-bromomethyl-3-vinylindole¹ **1** (6 g, 11.13.38 mmol) in dry THF (40 mL), dimethylamine (0.72 g, 16.06 mmol) and K₂CO₃ (2.21 g, 16.06 mmol) were added. The reaction mixture was stirred at room temperature for 3 h. After completion of the reaction (monitored by TLC), the solvent was removed under *vacuo*, and the reaction mixture was dissolved in DCM (50 mL). It was then diluted with water (30 mL), and the combined organic layer was separated. The aqueous layer was extracted with DCM (10 mL), and the combined organic extracts were dried (Na₂SO₄). The solvent was removed under vacuum, and the product was triturated with cold MeOH to afford 2-((dimethylamino)methyl)-3-vinyl indole **5** (5.02 g, 91%) as a white crystalline solid. mp: 113-115 °C; ¹H NMR (300 MHz, CDCl₃): δ 8.16 (d, *J* = 7.8 Hz, 1 H), 7.97 (d, *J* = 7.2 Hz, 2 H), 7.86 (d, *J* = 15.9 Hz, 1 H), 7.74 (d, *J* = 7.5 Hz, 1 H), 7.42 (t, *J* = 7.5 Hz, 1 H), 7.33-7.20 (m, 4 H), 6.47 (d, *J* = 16.2 Hz, 1 H), 4.2 (q, *J* = 6.9 Hz, 2 H), 3.89 (s, 2 H), 2.07 (s, 6 H), 1.26 (t, *J* = 6.9 Hz, 3H) ppm; ¹³C NMR (75 MHz, CDCl₃): δ 167.1, 140.0, 137.2, 135.5, 133.4, 128.6, 127.0, 126.8, 125.4, 124.0, 120.4, 119.9, 118.1, 115.1, 60.5, 52.1, 44.4, 14.3 ppm; DEPT-135 NMR (75 MHz, CDCl₃): δ 135.4, 133.4, 128.6, 127.0, 125.3, 124.0, 120.3, 119.8, 115.0, 60.4, 52.0, 44.4, 14.3 ppm.

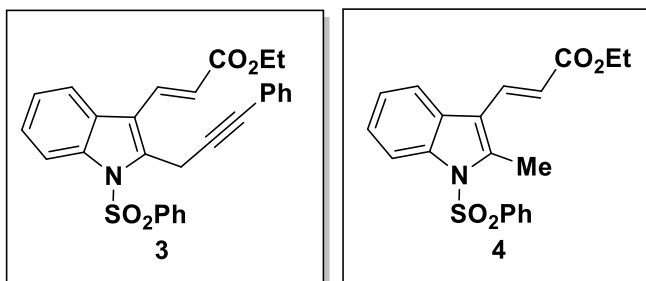
(*E*)-1-(3-(3-Ethoxy-3-oxoprop-1-en-1-yl)-1-(phenylsulfonyl)-1*H*-indol-2-yl)-*N,N,N*-trimethylmethanaminium trifluoromethanesulfonate **6a**



To a solution of amine compound **5** (5.0 g, 12.12 mmol) in dry THF (40 mL), MeOTf (2.19 g, 13.33 mmol) was added dropwise at 0 °C, then allowed to stir at rt for 20 minutes. Removal of solvent followed by the trituration of the crude product with diethyl ether (60 mL) afforded amine salt **6a** (6.63 g, 95%) as colorless solid. The amine salt **6a** was stored at 0 °C under inert atmosphere. mp: 184-186 °C; ¹H NMR (300 MHz, DMSO-*d*₆): δ 8.21 (d, *J* = 8.1 Hz, 1 H), 7.98-7.90 (m, 2 H), 7.81 (d, *J* = 7.8 Hz, 2 H), 7.65 (t, *J* = 6.9 Hz, 1 H), 7.60-7.44 (m, 4 H),

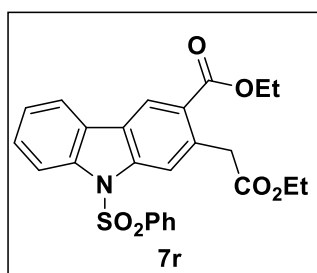
6.74 (d, $J = 16.2$ Hz, 1 H), 5.37 (s, 2 H), 4.25 (q, $J = 6.6$ Hz, 2 H), 3.20 (s, 9 H), 1.29 (t, $J = 6.9$ Hz, 3 H) ppm; ^{13}C NMR (75 MHz, DMSO- d_6): δ 165.5, 137.5, 135.3, 135.1, 133.3, 129.6, 128.2, 127.6, 127.1, 126.9, 126.5, 125.6, 124.6, 121.5, 116.0, 60.4, 58.1, 53.2, 14.0 ppm.

Attempted synthesis of ethyl (*E*)-3-(2-(3-phenylprop-2-yn-1-yl)-1-(phenylsulfonyl)-1*H*-indol-3-yl)acrylate **3 from amine salt **6a****



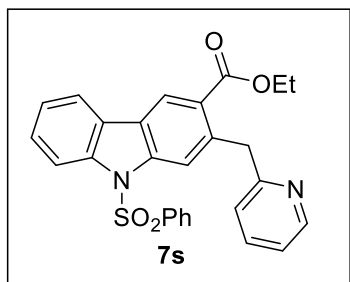
The reaction of amine salt **6a** (0.3 g, 0.52 mmol) with phenylacetylene (80 mg, 0.78 mmol) in the presence of Pd(PPh₃)₄ (60 mg, 0.05 mmol), K₂CO₃ (72 mg, 0.52 mmol) in a dry toluene (5 mL) at 100 °C for 8 h. After the reaction was completion (monitored by TLC), the reaction mixture was cooled to room temperature, the reaction mixture was quenched by water and extracted with ethyl acetate (2 x 20 mL). The combined organic layer was dried (Na₂SO₄) and evaporated under *vacuo* followed by column chromatographic purification on silica gel (230-420 mesh, n-hexane/ethyl acetate, 19:1) afforded 2-methyl-3-vinylindole **4** (48 mg, 25%) as a colorless solid; mp: 203-205 °C. Further elution of the column (n-hexane/ethyl acetate, 9:1) afforded carbazole **7a** (0.103 g, 42%) as a colorless solid; mp: 145-1148 °C.

Attempted synthesis of ethyl 2-(2-ethoxy-2-oxoethyl)-9-(phenylsulfonyl)-9*H*-carbazole-3-carboxylate **7r**



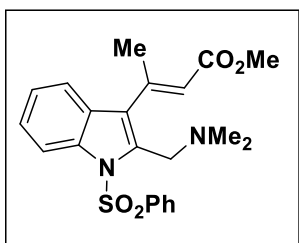
The reaction of amine salt **6a** (0.3 g, 0.52 mmol) with ethyl propiolate (77 mg, 0.78 mmol) in the presence of Pd(PPh₃)₄ (60 mg, 0.05 mmol), K₂CO₃ (36 mg, 0.26 mmol) in a mixture of DMSO (0.8 mL) and dry toluene (4.8 mL) at 100 °C for 8 h followed by workup using the above mentioned procedure failed to give the expected carbazole **7r**, instead 2-methyl-3-vinylindole **4** (198 mg, 80%) was isolated.

Attempted synthesis of ethyl 9-(phenylsulfonyl)-2-(pyridin-2-ylmethyl)-9H-carbazole-3-carboxylate **7s**



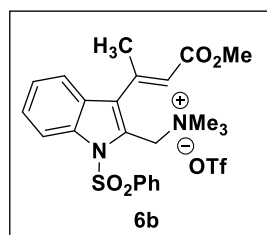
The reaction of amine salt **6a** (0.3 g, 0.52 mmol) with 2-ethynylpyridine (80 mg, 0.78 mmol) in the presence of Pd(PPh₃)₄ (60 mg, 0.05 mmol), K₂CO₃ (36 mg, 0.26 mmol) in a mixture of DMSO (0.8 mL) and dry toluene (4.8 mL) at 100 °C for 8 h followed by workup using the above mentioned procedure failed to give the expected carbazole **7s**.

Methyl (*E*)-3-(2-((dimethylamino)methyl)-1-(phenylsulfonyl)-1*H*-indol-3-yl)but-2-enoate



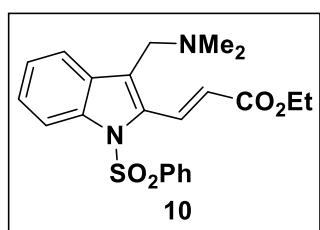
A mixture of methyl (*E*)-3-(2-(bromomethyl)-1-(phenylsulfonyl)-1*H*-indol-3-yl)but-2-enoate² (5.2 g, 11.59 mmol) in dry THF, dimethylamine (0.63 g, 13.92 mmol) and K₂CO₃ (1.92 g, 13.92 mmol) were added, the reaction mixture was stirred at room temperature for 3 h. Then the THF solvent was removed under *vacuo* and the reaction mixture dissolved in DCM. It was then diluted with water (10 mL) and the combined organic layer was separated. The aqueous layer was extracted with DCM (10 mL) and the combined organic extracts were dried (Na₂SO₄). The crude product methyl (*E*)-3-(2-((dimethylamino)methyl)-1-(phenylsulfonyl)-1*H*-indol-3-yl)but-2-enoate was used as such for next step without any further purification.

((Trifluoromethyl)sulfonyl)-11-oxidane, (*E*)-1-(3-(4-methoxy-4-oxobut-2-en-2-yl)-1-(phenylsulfonyl)-1*H*-indol-2-yl)-*N,N,N*-trimethylmethanaminium salt **6b**



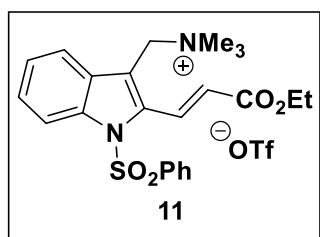
A mixture of methyl (*E*)-3-(2-((dimethylamino)methyl)-1-(phenylsulfonyl)-1*H*-indol-3-yl)but-2-enoate (4 g, 9.70 mmol) in dry THF (40 mL), MeOTF (1.75 g, 10.67 mmol) was added dropwise at 0 °C, then allowed to stir at rt for 20 minutes. Removal of solvent followed by trituration of the crude product with diethyl ether (60 mL) afforded amine salt **6b** (5.14 g, 92%) as pale yellow solid. The amine salt **6b** was stored at 0 °C under inert atmosphere. mp: 150-152 °C; ¹H NMR (300 MHz, DMSO-*d*₆): δ 8.20 (d, *J* = 8.7 Hz, 1 H), 7.81 (d, *J* = 7.5 Hz, 2 H), 7.68 (t, *J* = 7.2 Hz, 1 H), 7.61-7.50 (m, 4 H), 7.42 (d, *J* = 7.8 Hz, 2 H), 6.02 (s, 1 H), 5.15 (broad s, 2 H), 3.77 (s, 3 H), 3.22 (m, 8 H), 2.50 (s, 3 H) ppm; ¹³C NMR (75 MHz, DMSO-*d*₆): δ 164.0, 144.5, 136.2, 136.0, 133.7, 133.5, 128.2, 127.3, 126.4, 125.3, 124.2, 122.5, 122.1, 119.9, 115.1, 57.9, 51.9, 49.8, 18.6 ppm.

Ethyl (*E*)-3-(3-((dimethylamino)methyl)-1-(phenylsulfonyl)-1*H*-indol-2-yl)acrylate **10**



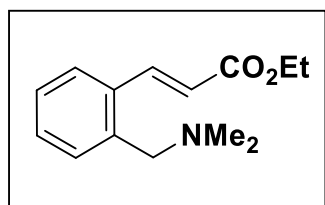
To a solution of ethyl (*E*)-3-(3-(bromomethyl)-1-(phenylsulfonyl)-1*H*-indol-2-yl)acrylate¹ (1 g, 2.24 mmol) in dry THF (20 mL), dimethylamine (0.121 g, 2.68 mmol) and K₂CO₃ (0.37 g, 2.68 mmol) were added, the reaction mixture was stirred at room temperature for 3 h. Then the THF solvent was removed under *vacuo* and the reaction mixture dissolved in DCM (20 mL). It was then diluted with water (10 mL) and the combined organic layer was separated. The aqueous layer was extracted with DCM (2 x 10 mL) and the combined organic extract was dried (Na₂SO₄). The solvent was removed under *vacuo* and trituated with cold MeOH (10 mL) to afford 3-((dimethylamino)methyl)-2-vinyl indole **10** (0.84 g, 91%) as a colorless solid. mp: 112-114 °C; ¹H NMR (300 MHz, CDCl₃): δ 8.15-8.07 (m, 2 H), 7.58 (d, *J* = 7.2 Hz, 2 H), 7.38 (t, *J* = 7.2 Hz, 1 H), 7.31-7.15 (m, 4 H), 6.55 (d, *J* = 15.9 Hz, 1 H), 4.24 (q, *J* = 7.2 Hz, 2 H), 3.34 (s, 2 H), 2.06 (s, 6 H), 1.30 (t, *J* = 7.2 Hz, 3 H) ppm; ¹³C NMR (75 MHz, CDCl₃): δ 166.5, 137.8, 137.3, 134.8, 133.7, 133.6, 131.4, 128.9, 126.6, 126.3, 125.0, 124.6, 124.3, 120.4, 115.6, 60.7, 53.3, 45.0, 14.3 ppm; DEPT-135 NMR (75 MHz, CDCl₃): δ 133.7, 133.5, 128.9, 126.6, 126.2, 124.6, 124.2, 120.4, 115.5, 60.6, 53.3, 45.0, 14.3 ppm.

(E)-1-(2-(3-ethoxy-3-oxoprop-1-en-1-yl)-1-(phenylsulfonyl)-1H-indol-3-yl)-N,N,N-trimethylmethanaminium trifluoromethanesulfonate 11



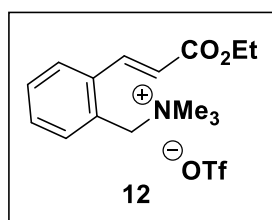
A mixture of 3-((dimethylamino)methyl)-2-vinyl indole **10** (0.5 g, 1.21 mmol) in dry THF (10 mL) and MeOTf (0.22 g, 1.33 mmol) was added dropwise at 0°C, and then allowed to stir for 20 minutes at room temperature. Removal of solvent afforded amine salt as a thick liquid. The ¹HNMR spectrum of crude product failed to confirm the formation of salt **11**.

Ethyl (E)-3-(2-((dimethylamino)methyl)phenyl)acrylate



To a solution of ethyl (E)-3-(2-(bromomethyl)phenyl)acrylate³ (2 g, 7.43 mmol) in dry THF (20 mL), dimethylamine (0.4 g, 8.92 mmol) and K₂CO₃ (1.23 g, 8.92 mmol) were added, the reaction mixture was stirred at room temperature for 3 h. Then, the THF solvent was removed under *vacuo* and the reaction mixture dissolved in DCM. It was then diluted with water (10 mL) and the combined organic layer was separated. The aqueous layer was extracted with DCM (2 x 10 mL) and the combined organic extract was dried (Na₂SO₄). The crude product ethyl (E)-3-(2-((dimethylamino)methyl)phenyl)acrylate was used as such for next step without any further purification.

(E)-1-(2-(3-Ethoxy-3-oxoprop-1-en-1-yl)phenyl)-N,N,N-trimethylmethanaminium trifluoromethanesulfonate 12

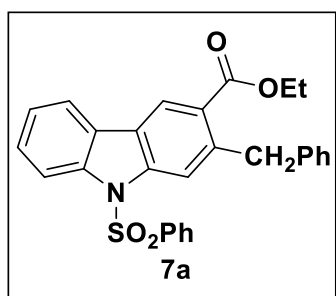


A mixture of ethyl (E)-3-(2-((dimethylamino)methyl)phenyl)acrylate (1.5 g, 6.43 mmol) in dry THF (20 mL), MeOTf (1.16 g, 7.07 mmol) was added dropwise at 0 °C, and allowed to stir for

30 minutes at rt. Subsequent removal of solvent followed by trituration of the crude product with diethyl ether afforded (*E*)-1-(2-(3-ethoxy-3-oxoprop-1-en-1-yl)phenyl)-*N,N,N*-trimethylmethanaminium trifluoromethanesulfonate **12** (1.68 g, 99%) as colorless solid. mp : 110-112 °C; ¹H NMR (300 MHz, DMSO-*d*₆): δ 8.09 (d, *J* = 15.6 Hz, 1 H), 7.96 (d, *J* = 7.5 Hz, 1 H), 7.63-7.57 (m, 3 H), 6.67 (d, *J* = 15.6 Hz, 1 H), 4.78 (s, 2 H), 4.78 (q, *J* = 7.2 Hz, 2 H), 3.01 (s, 9 H), 1.28 (t, *J* = 6.9 Hz, 1 H) ppm; ¹³C NMR (75 MHz, DMSO-*d*₆): δ 165.8, 140.9, 136.7, 135.2, 131.0, 130.2, 128.1, 127.5, 122.1, 64.3, 60.3, 52.1, 14.1 ppm; DEPT-135 NMR (75 MHz, DMSO-*d*₆): δ 141.4, 135.7, 131.5, 130.7, 128.6, 122.6, 64.8, 60.8, 52.6, 14.6 ppm.

Representative Gram Scale synthesis

Ethyl 2-benzyl-9-(phenylsulfonyl)-9*H*-carbazole-3-carboxylate **7a**

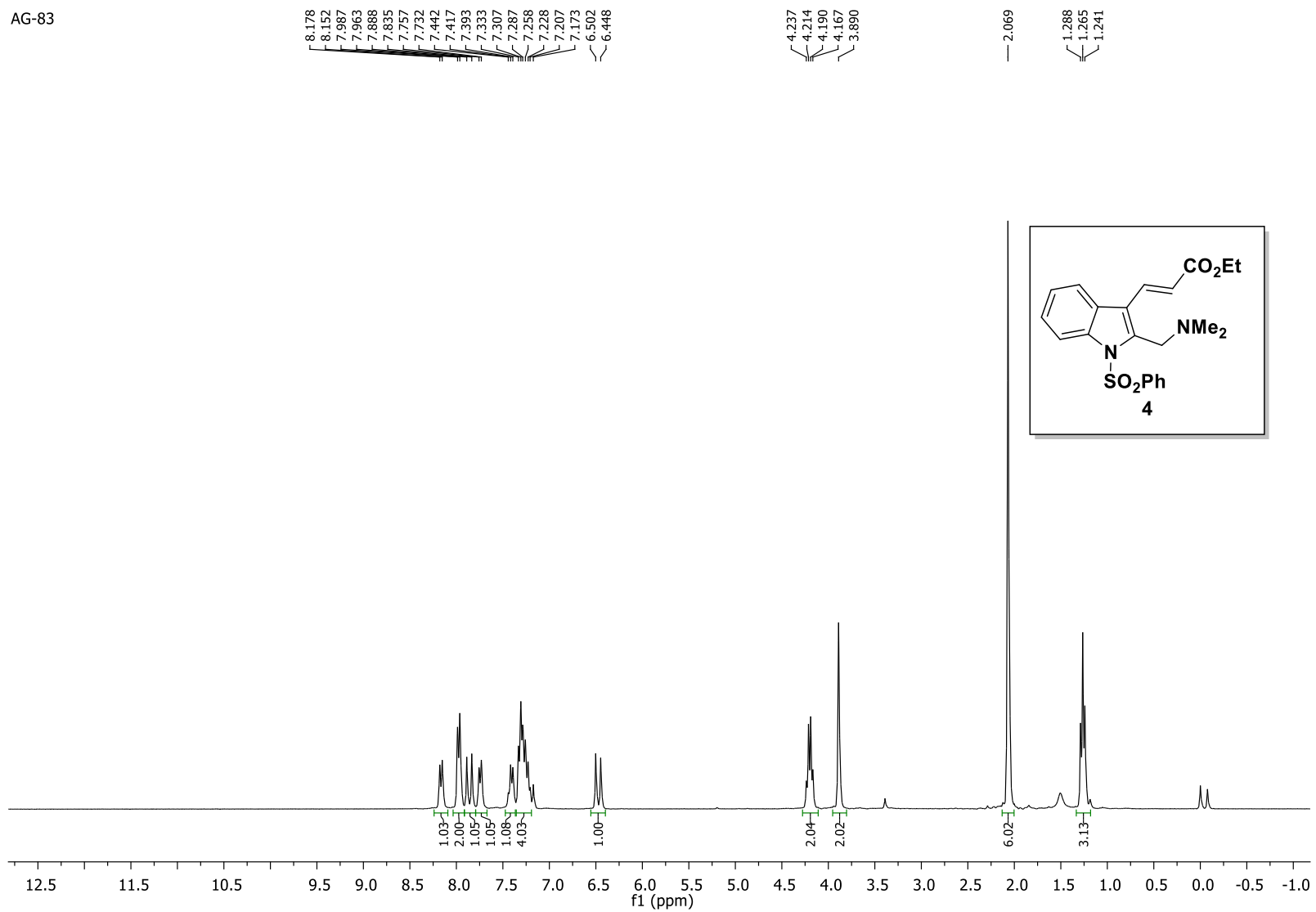


The reaction of amine salt **6a** (0.3 g, 0.52 mmol) with phenylacetylene (80 mg, 0.78 mmol) in the presence of Pd(PPh₃)₄ (60 mg, 0.05 mmol), K₂CO₃ (36 mg, 0.26 mmol) in a mixture of DMSO (0.8 mL) and dry toluene (4.8 mL) at 100 °C for 8 h followed by workup using the above mentioned procedure gave carbazole **7a** (0.218 g, 89%) as a colorless solid.

References

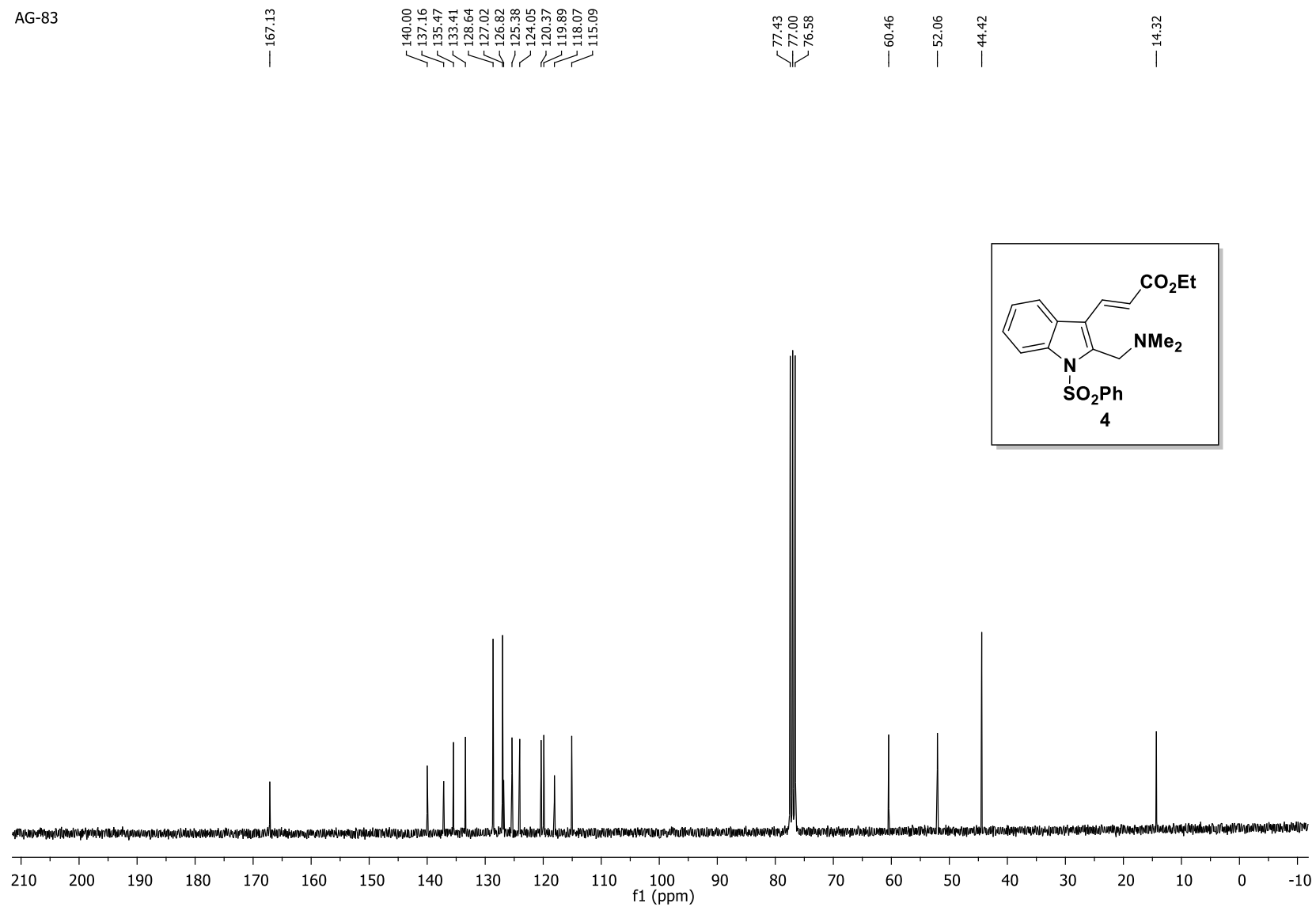
1. A. K. Mohanakrishnan and P. C. Srinivasan, *J. Org. Chem.*, 1995, **60**, 1939-1946.
2. P. Raju, G. Gobi Rajeshwaran and A. K. Mohanakrishnan, *Eur. J. Org. Chem.*, 2015, 7173-7145.
3. M. Grübel, C. Jandl and T. Bach, *Synlett*, 2019, **30**, 1825-1829.

AG-83



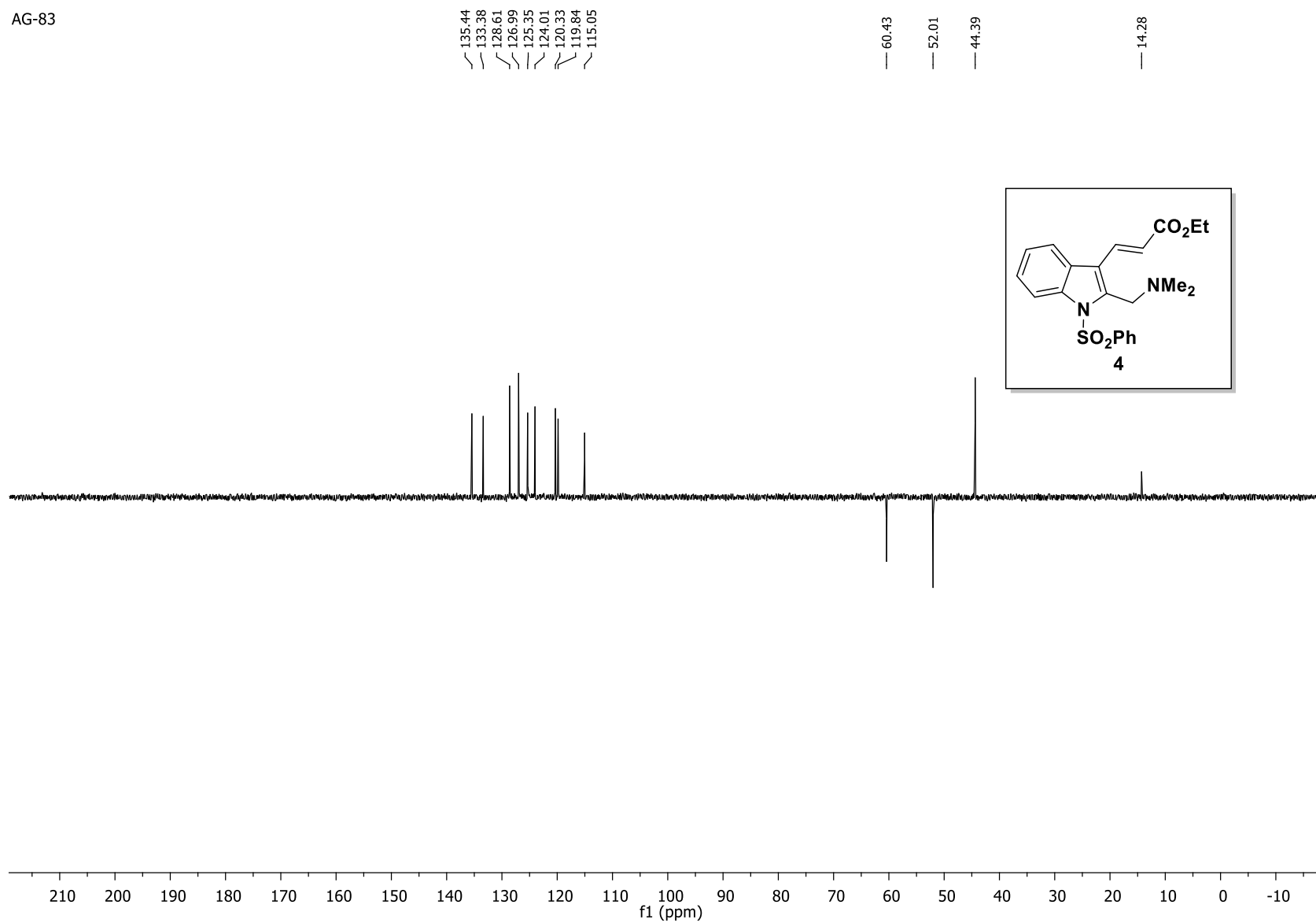
^1H NMR (300 MHz, CDCl_3) spectrum of compound 4

AG-83



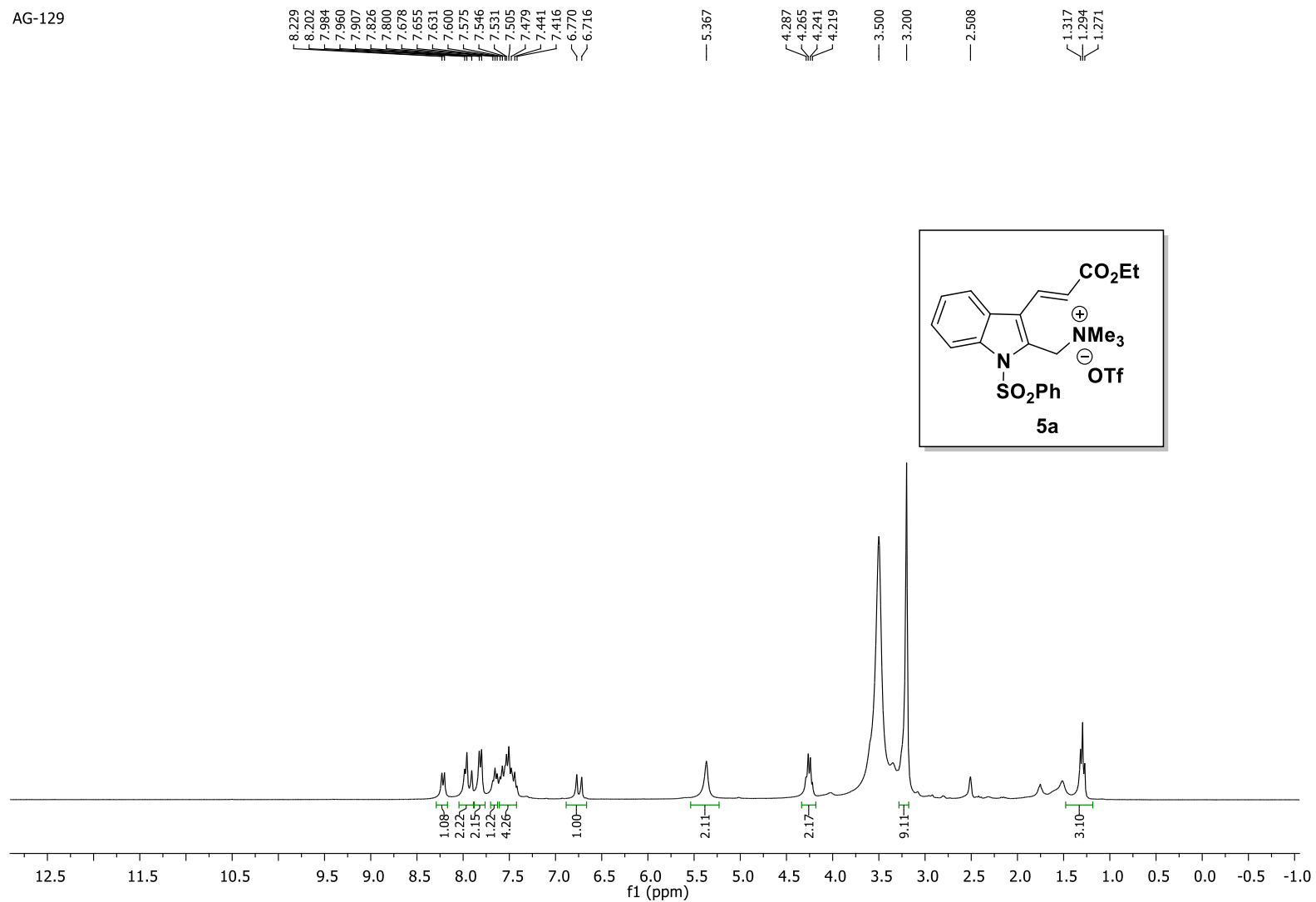
¹³C {¹H} NMR (75 MHz, CDCl₃) spectrum of compound **4**

AG-83



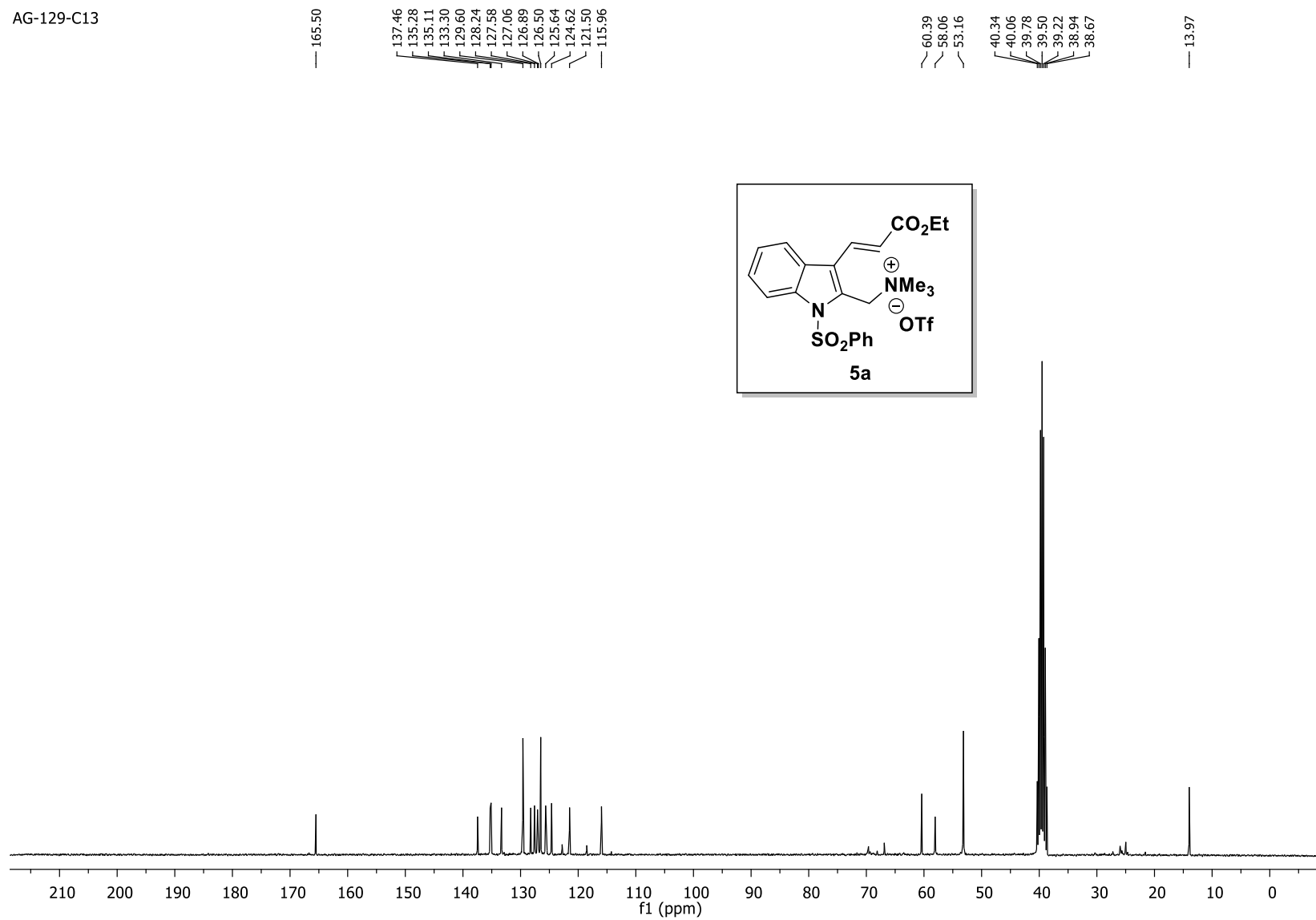
DEPT-135 NMR (75 MHz, CDCl₃) spectrum of compound 4

AG-129



^1H NMR (300 MHz, $\text{DMSO}-d_6$) spectrum of compound **5a**

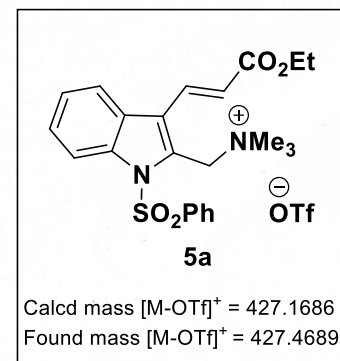
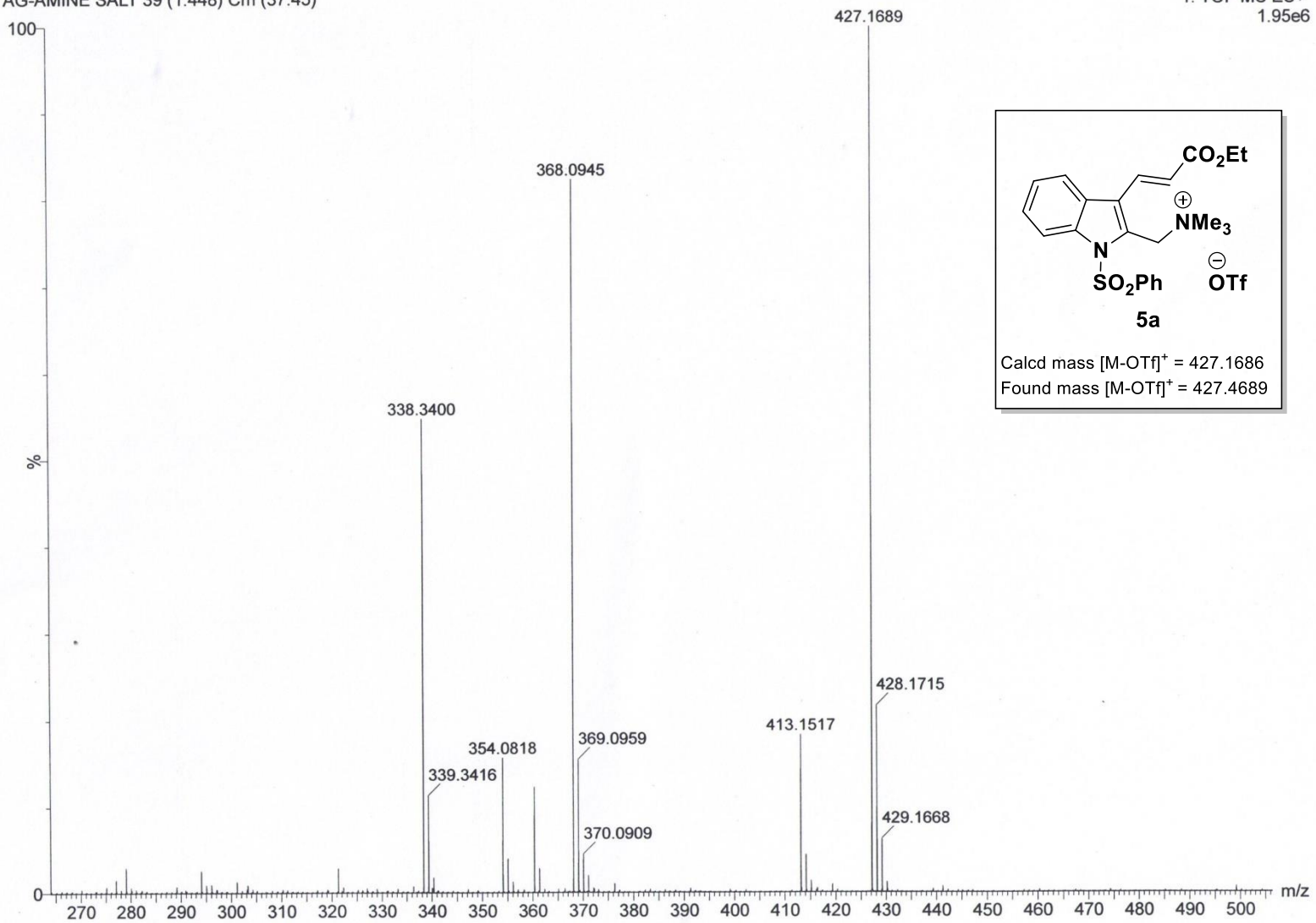
AG-129-C13



$^{13}\text{C} \{^1\text{H}\}$ NMR (75 MHz, $\text{DMSO-}d_6$) spectrum of compound **5a**

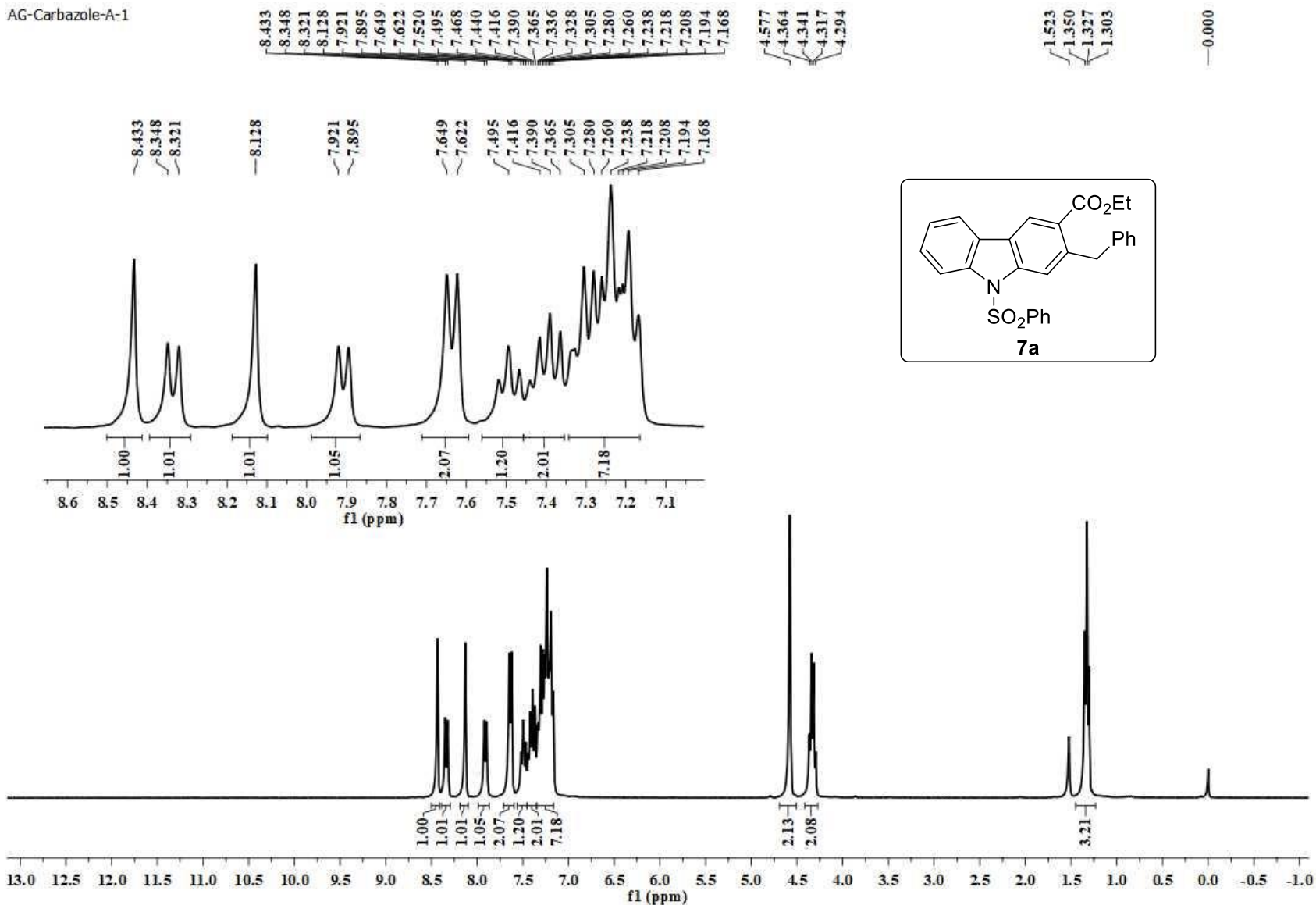
DR.AKM
AG-AMINE SALT 39 (1.448) Cm (37:45)

1: TOF MS ES+
1.95e6



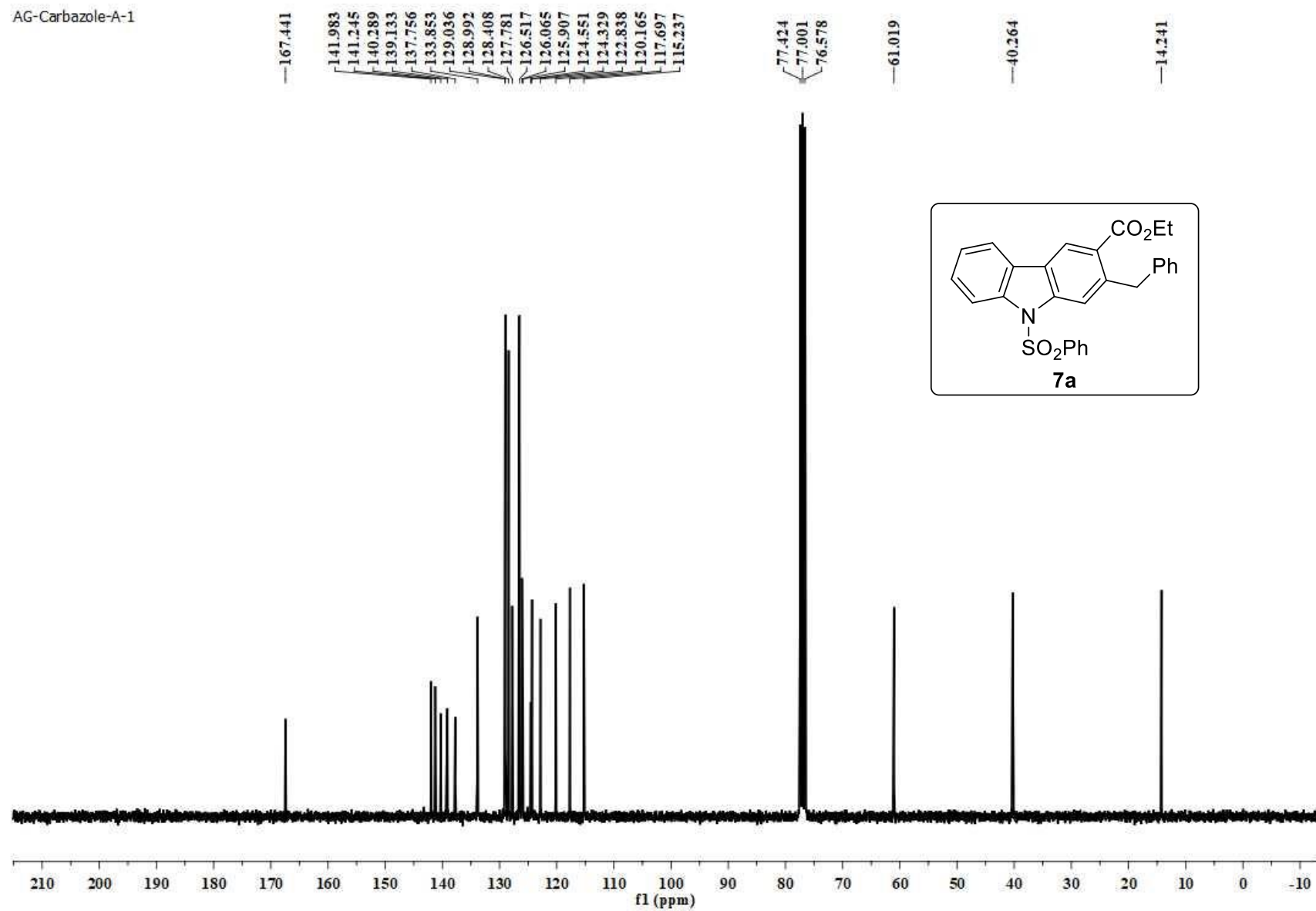
HRMS Spectrum of compound 5a

AG-Carbazole-A-1



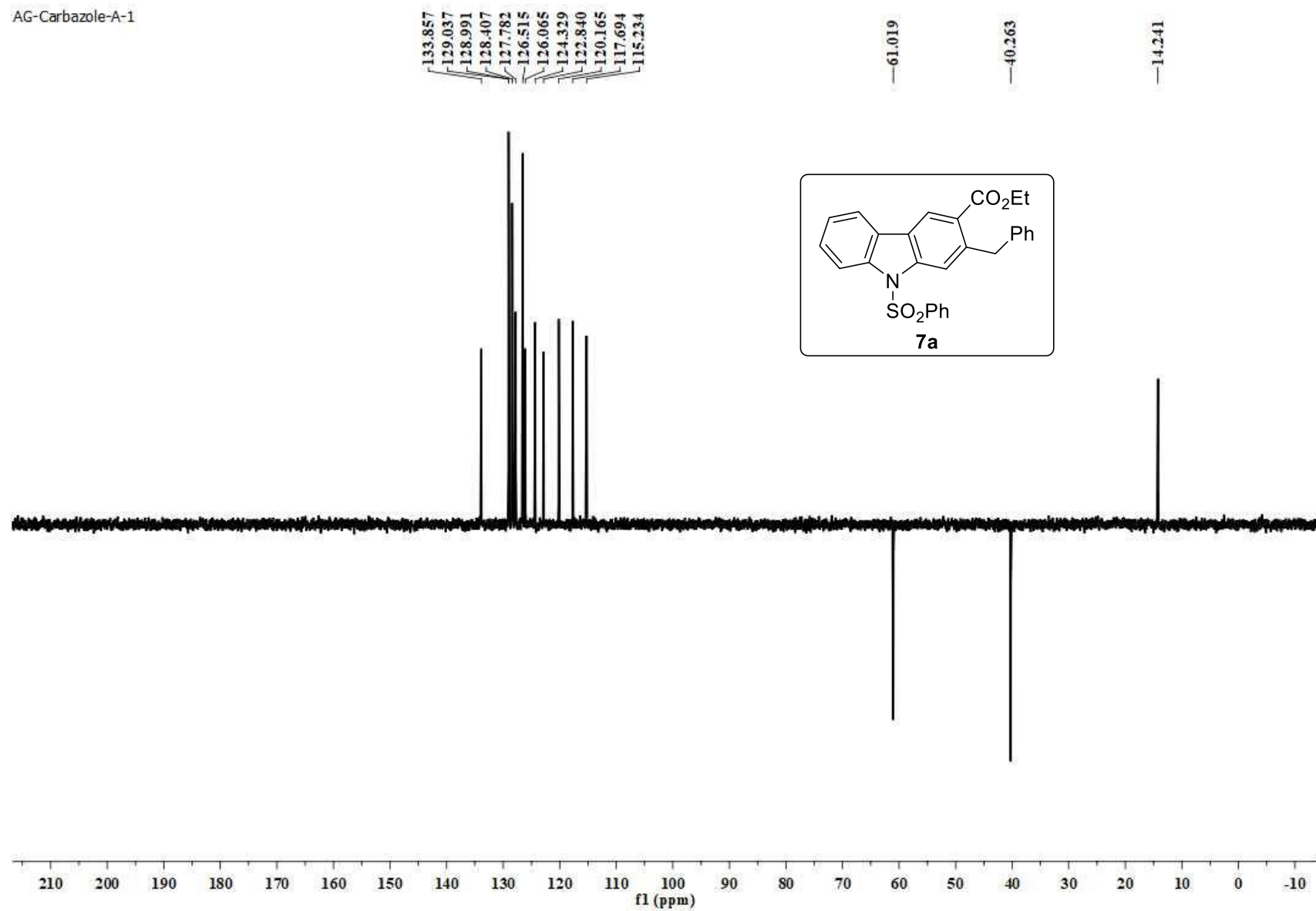
¹H NMR (300 MHz, CDCl₃) spectrum of compound **7a**

AG-Carbazole-A-1



$^{13}\text{C} \{^1\text{H}\}$ NMR (75 MHz, CDCl_3) spectrum of compound **7a**

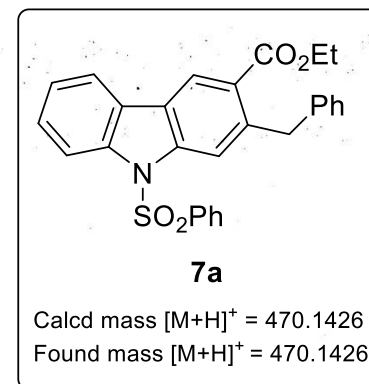
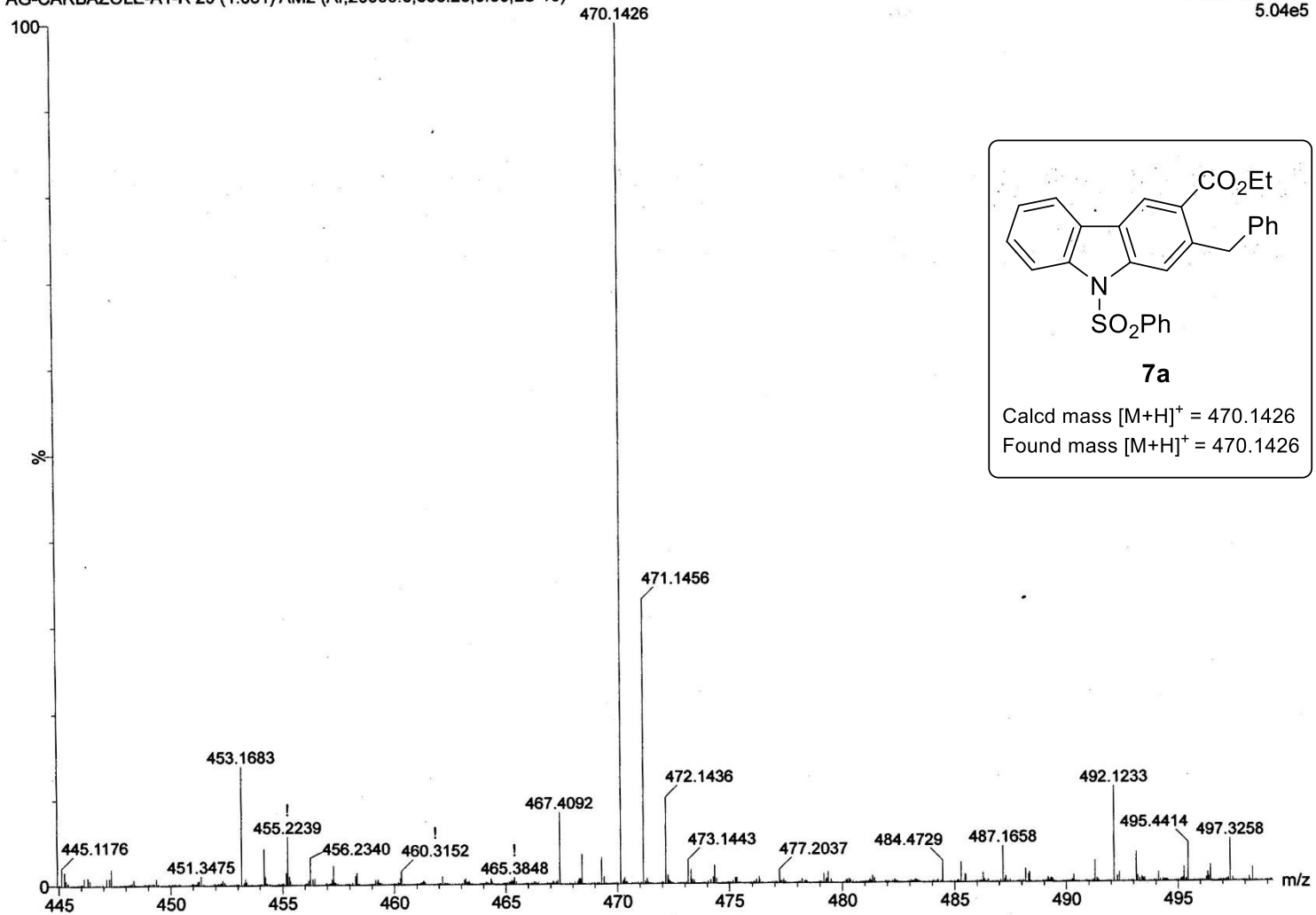
AG-Carbazole-A-1



DEPT-135 NMR (75 MHz, CDCl₃) spectrum of compound **7a**

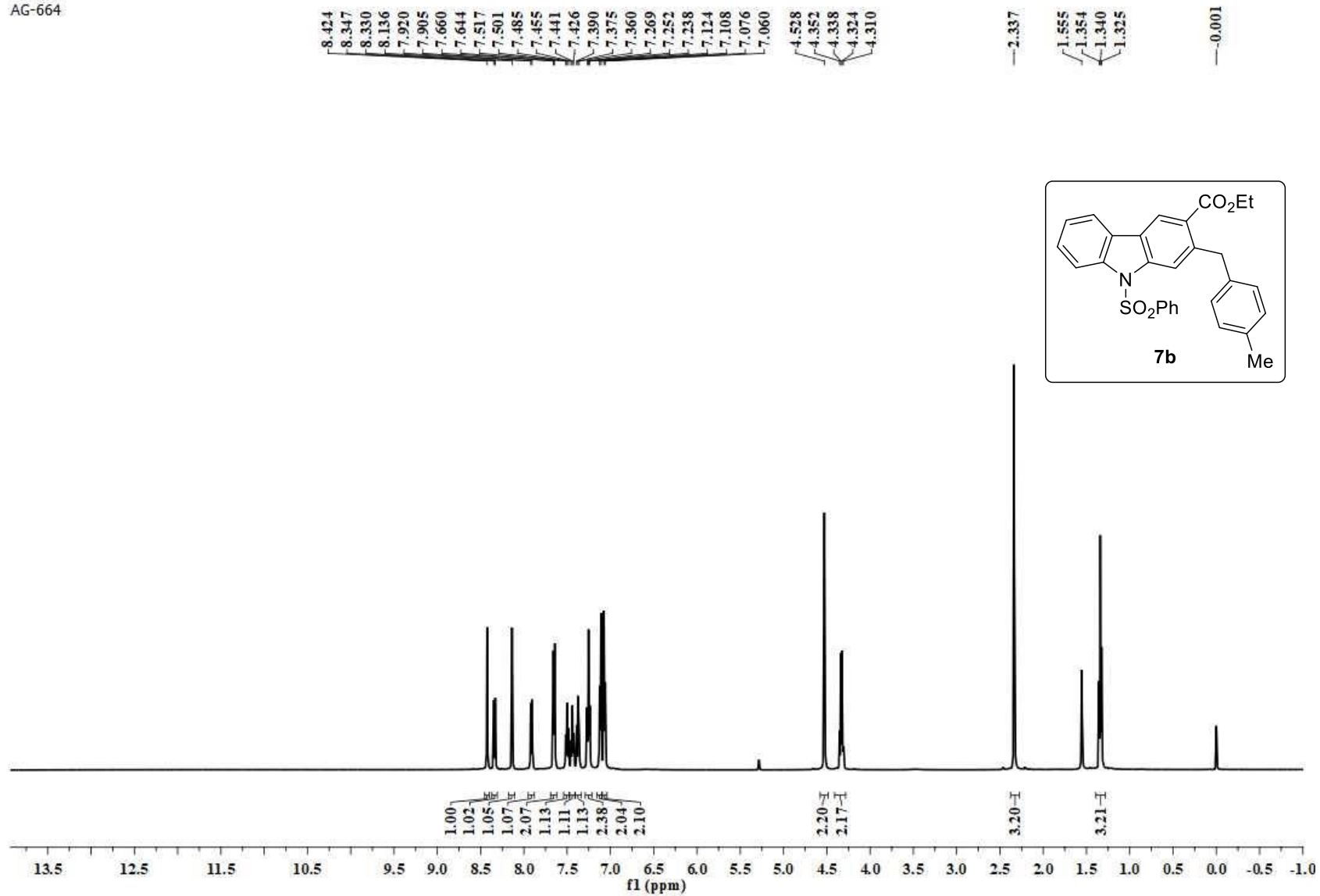
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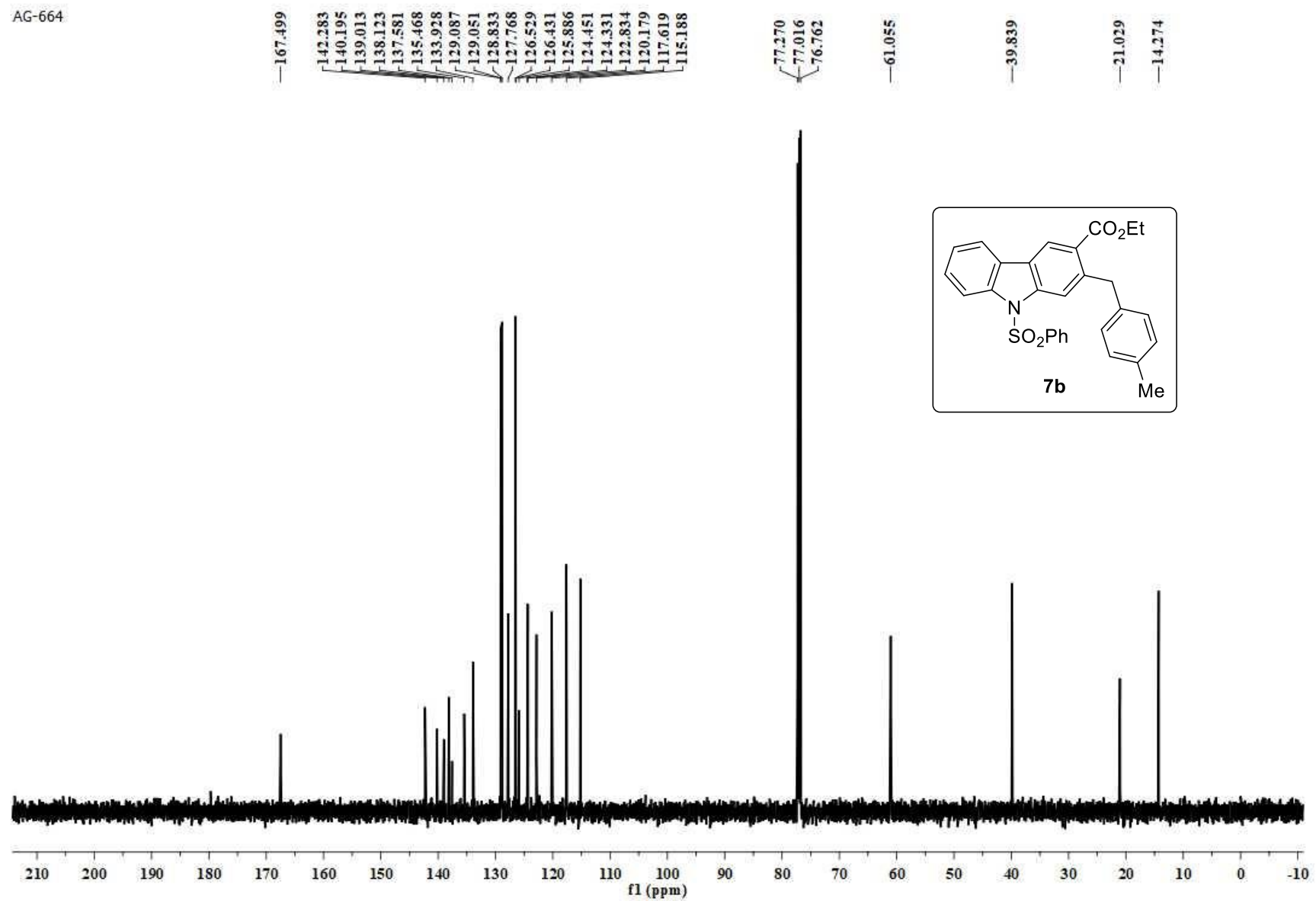
HRMS Spectrum of compound 7a

AG-664



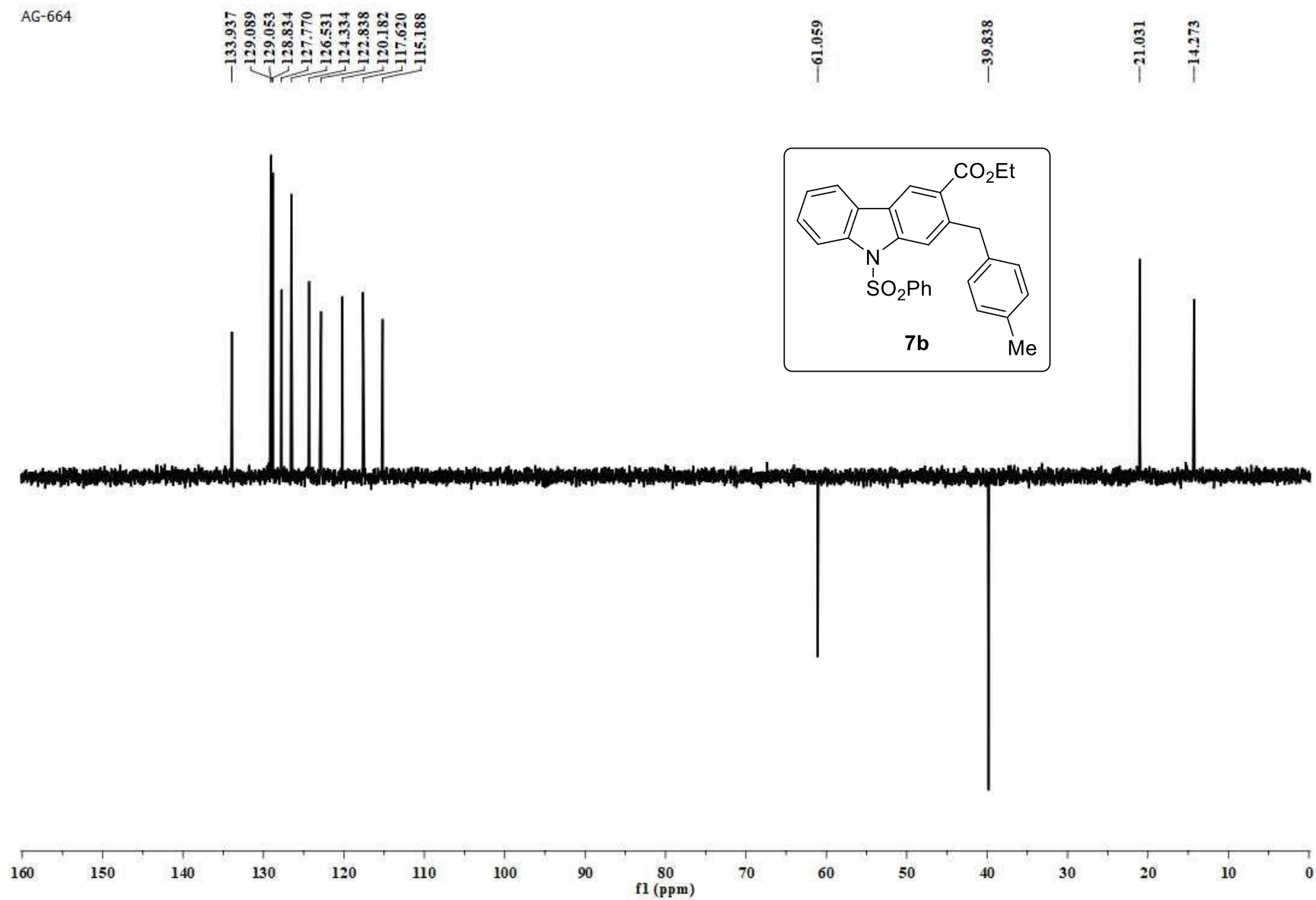
¹H NMR (500 MHz, CDCl₃) spectrum of compound 7b

AG-664

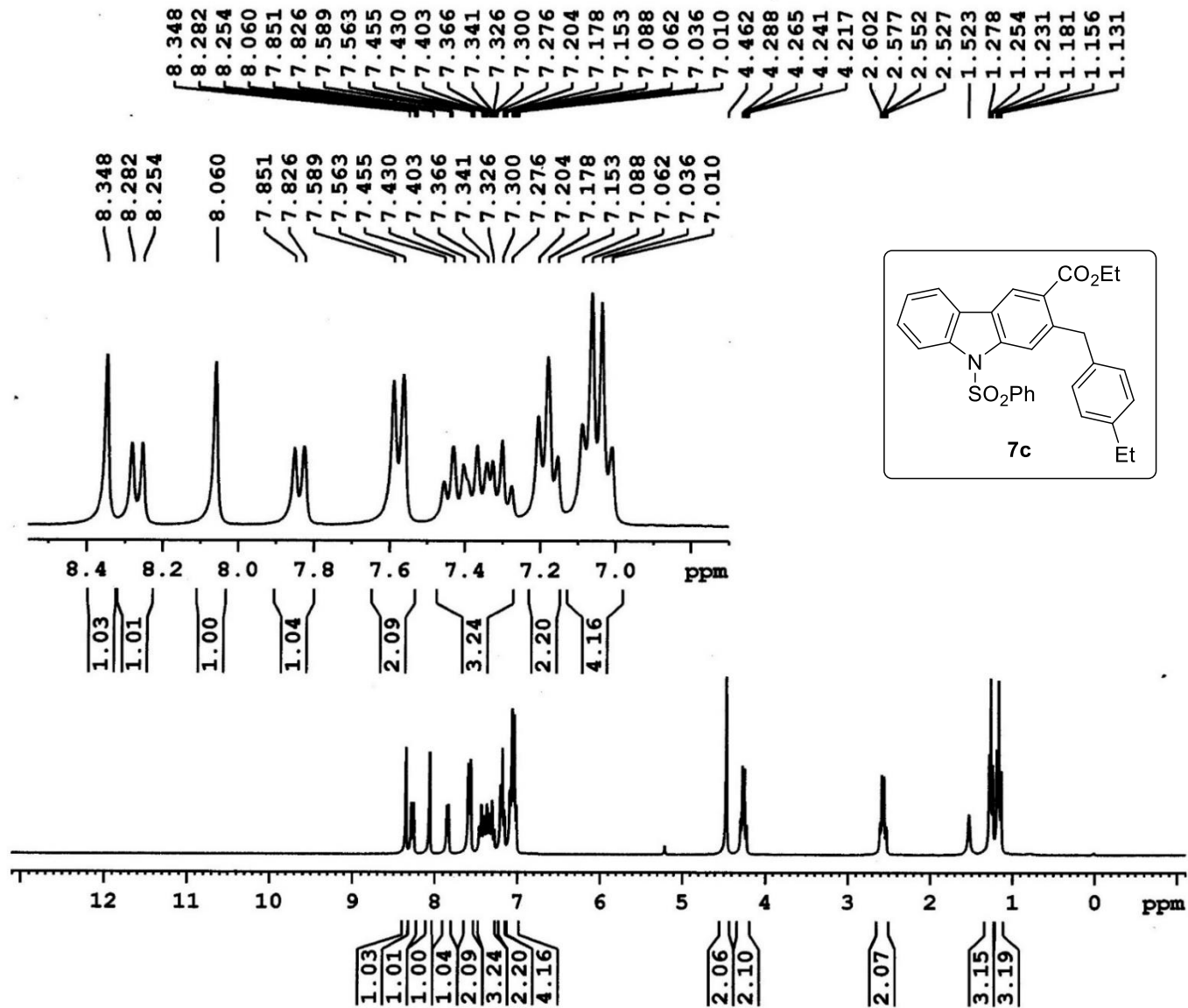


$^{13}\text{C} \{^1\text{H}\}$ NMR (125 MHz, CDCl_3) spectrum of compound **7b**

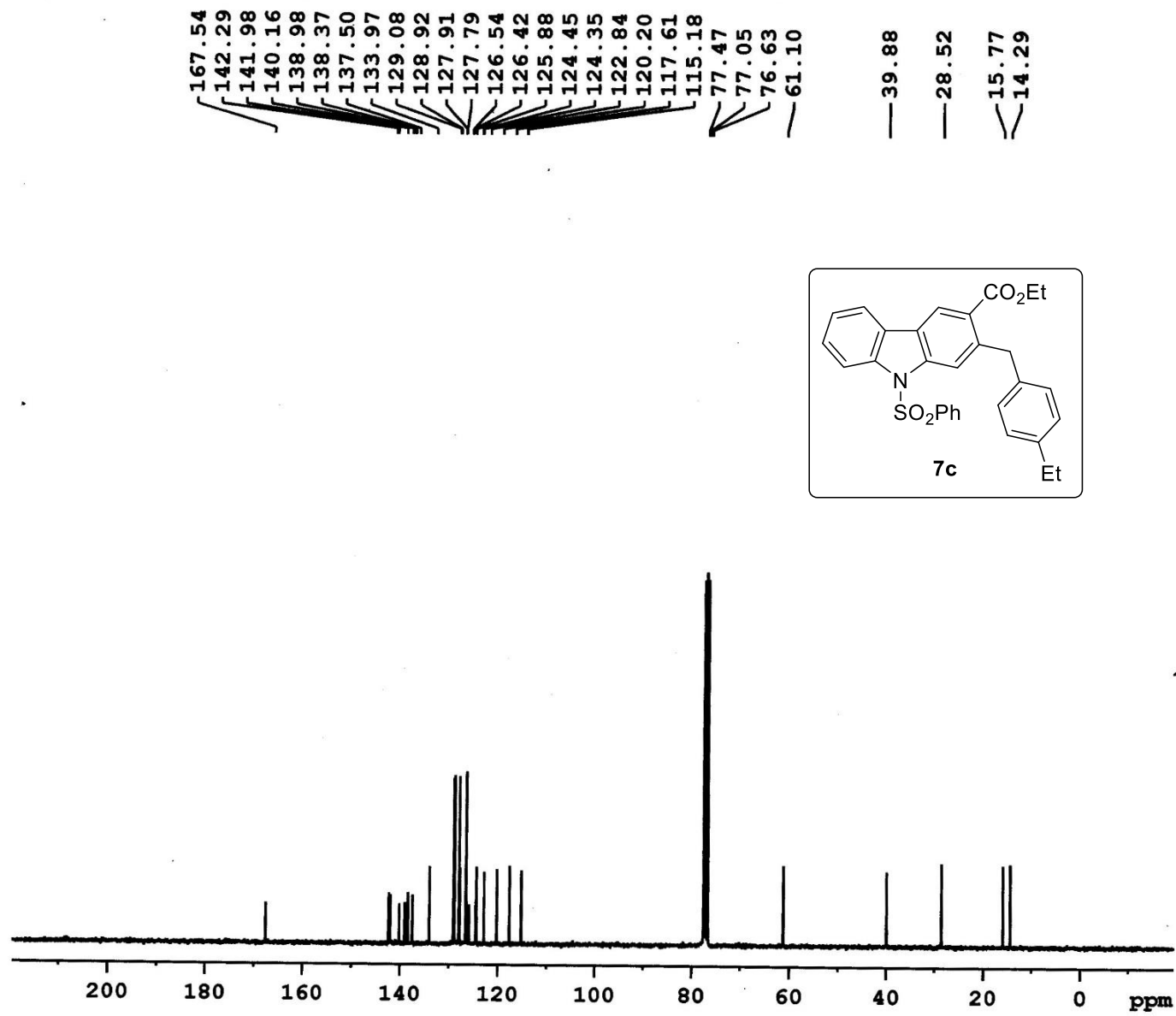
AG-664



DEPT-135 NMR (125 MHz, CDCl₃) spectrum of compound **7b**



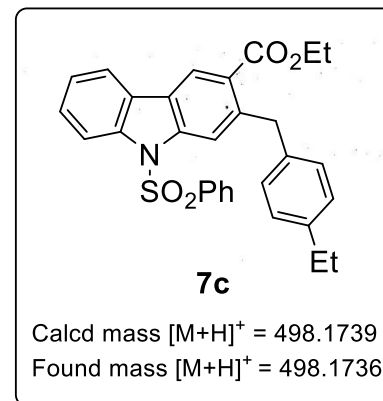
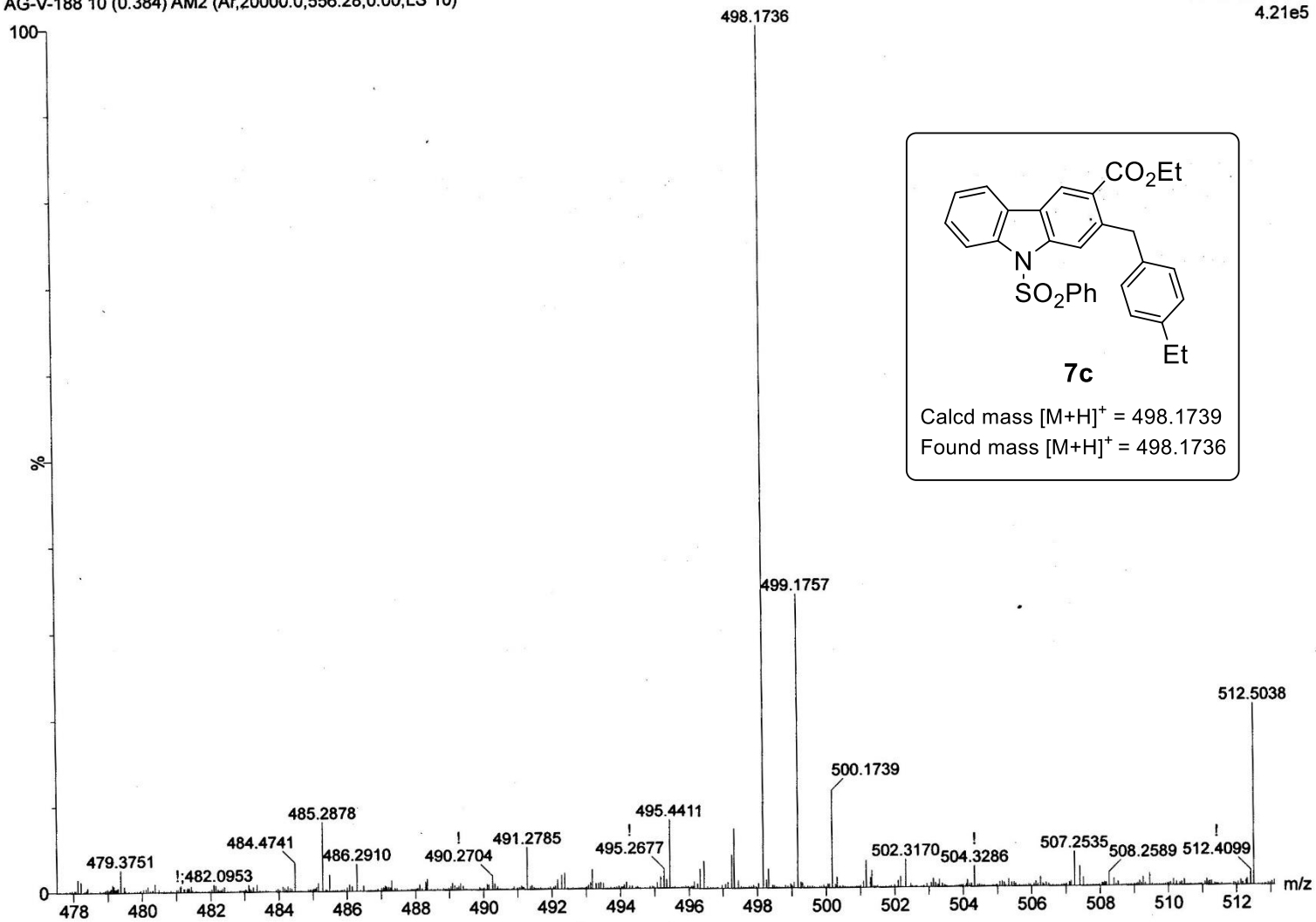
¹H NMR (300 MHz, CDCl₃) spectrum of compound 7c



$^{13}\text{C} \{^1\text{H}\}$ NMR (75 MHz, CDCl_3) spectrum of compound **7c**

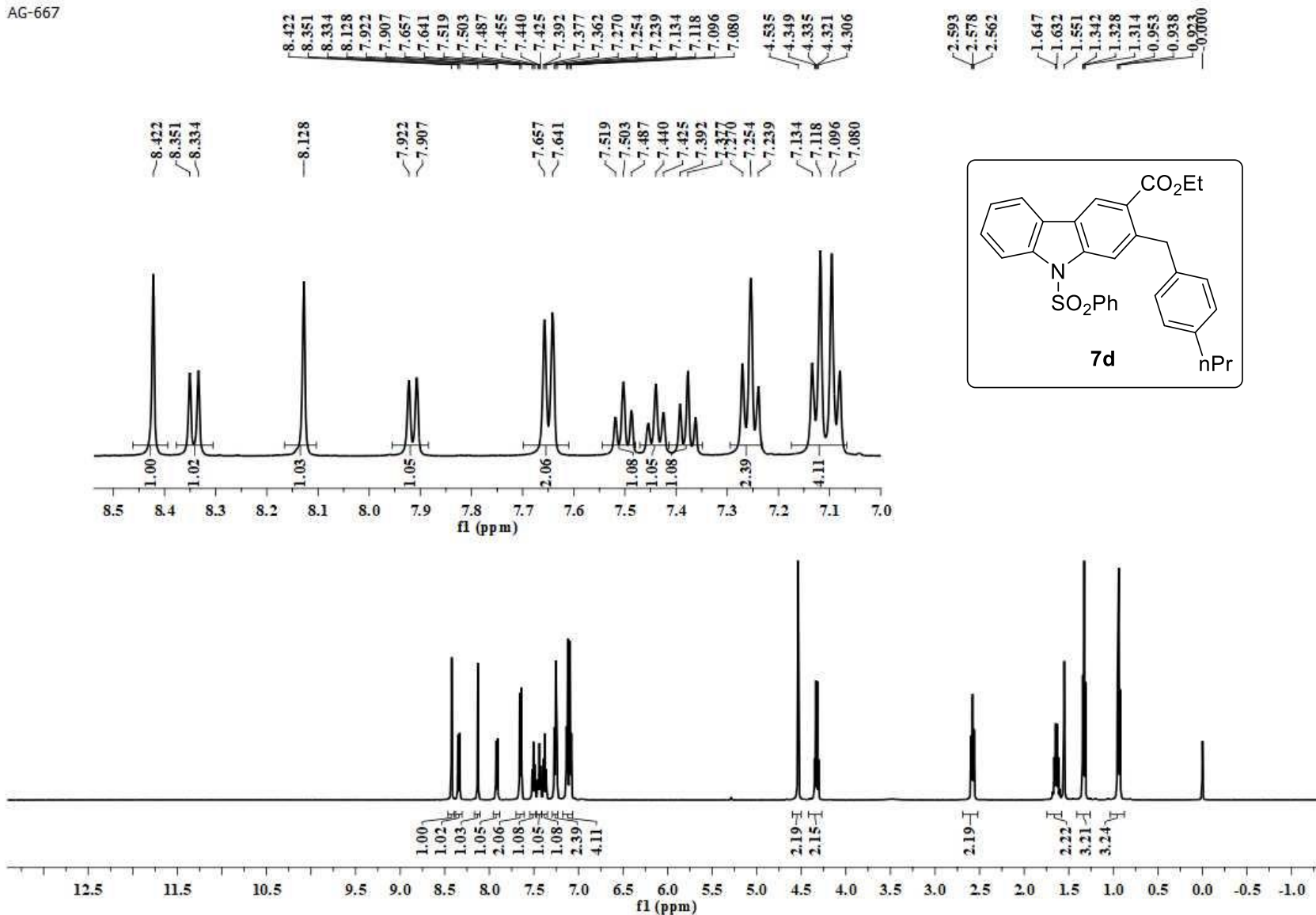
DRAKM
AG-V-188 10 (0.384) AM2 (Ar,20000.0,556.28,0.00,LS 10)

1: TOF MS ES+
4.21e5



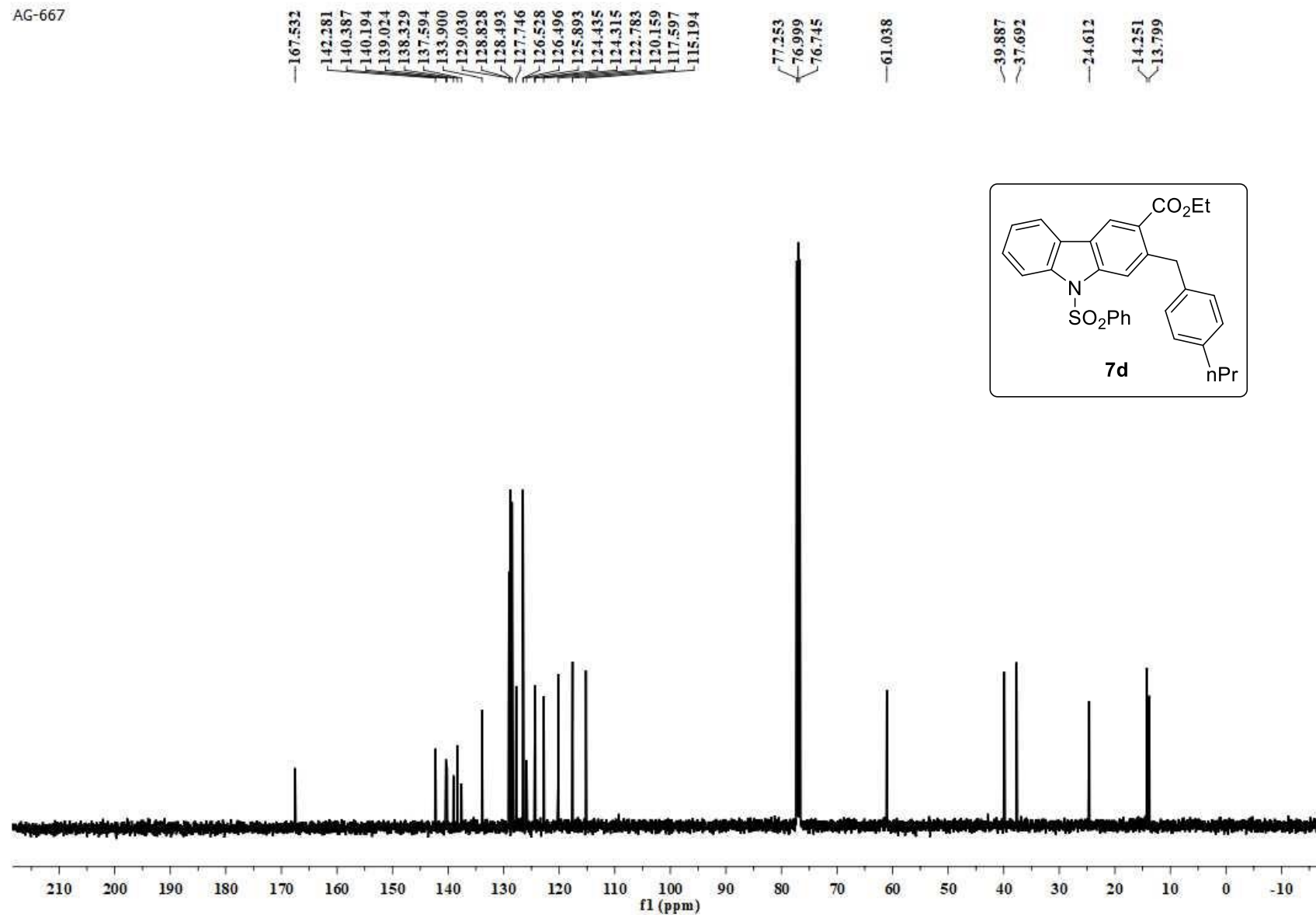
HRMS Spectrum of compound **7c**

AG-667



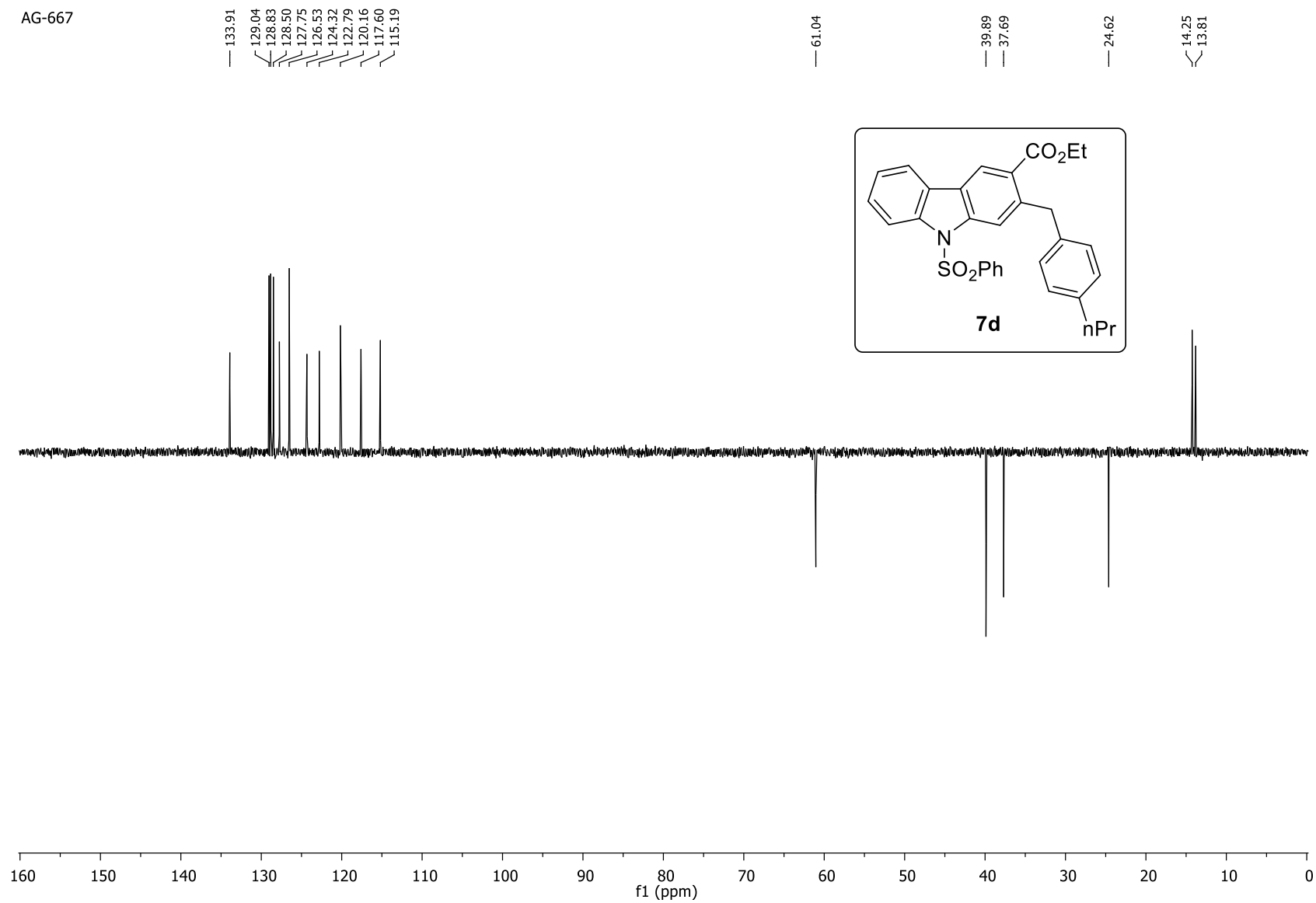
¹H NMR (500 MHz, CDCl₃) spectrum of compound **7d**

AG-667



^{13}C $\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) spectrum of compound 7d

AG-667



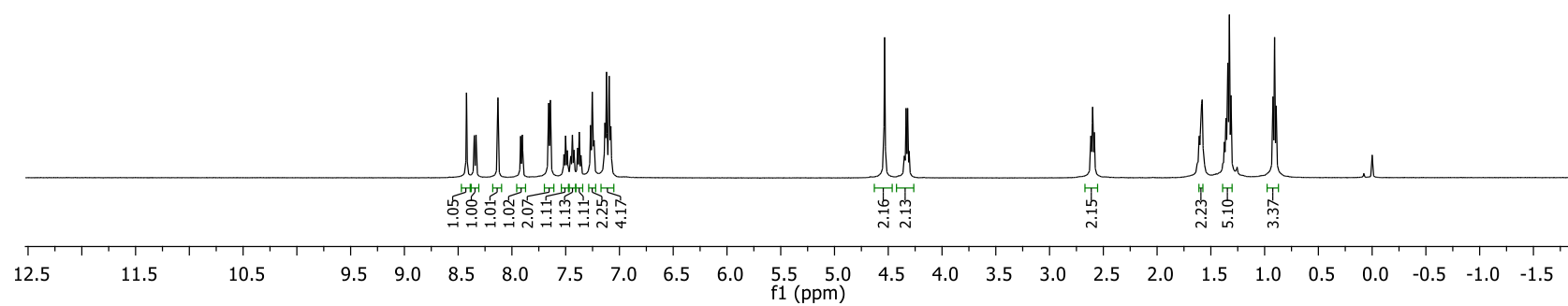
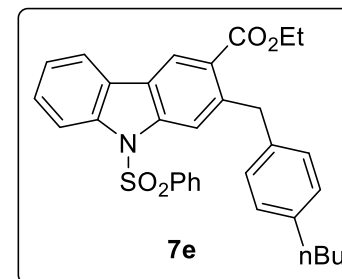
DEPT-135 NMR (125 MHz, CDCl₃) spectrum of compound **7d**

AG-821

8.422
8.350
8.333
8.130
7.919
7.903
7.658
7.642
7.516
7.501
7.485
7.452
7.438
7.423
7.389
7.374
7.359
7.269
7.253
7.238
7.135
7.120
7.096
7.080

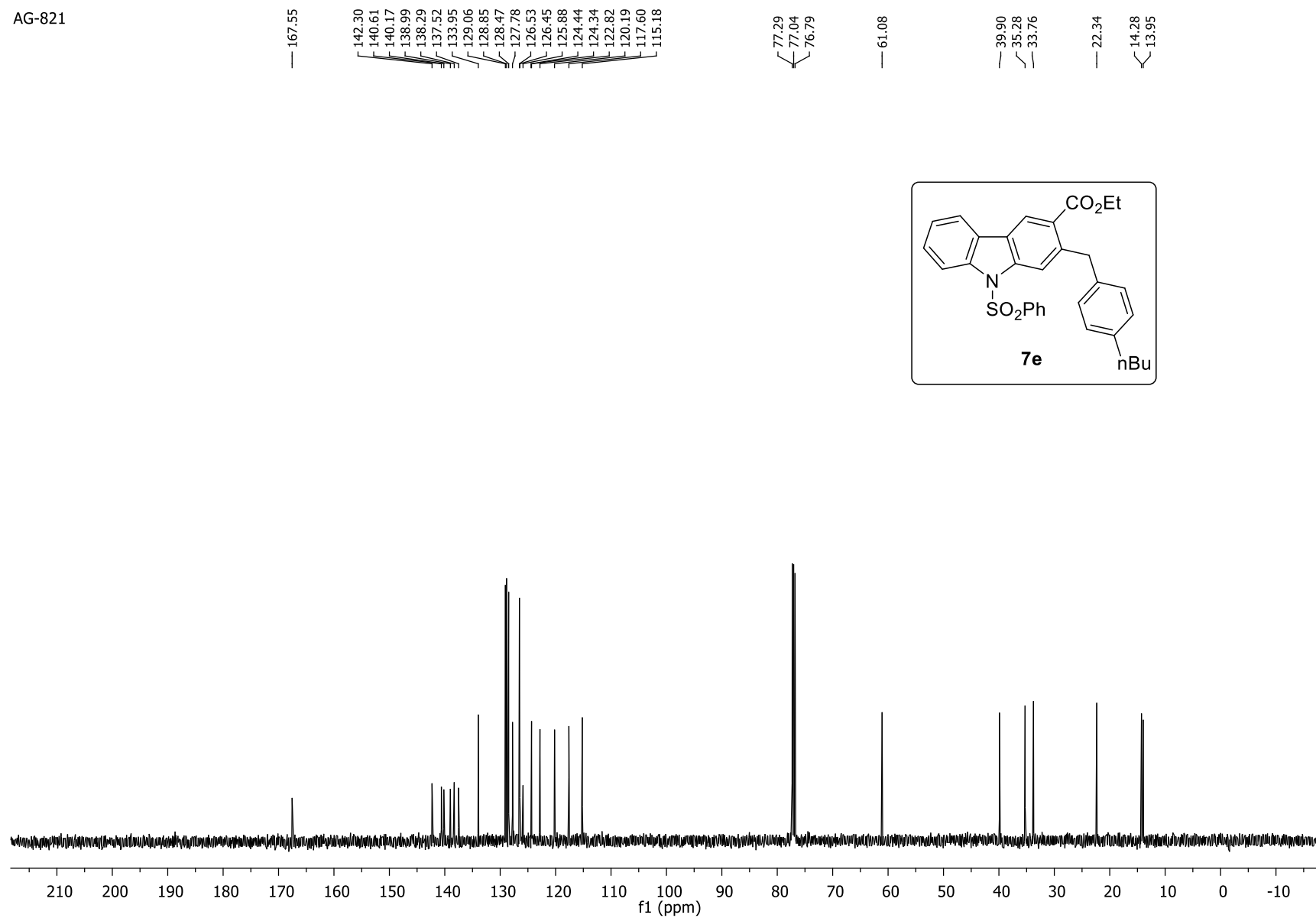
4.534
4.348
4.334
4.320
4.306

2.615
2.599
2.584
1.608
1.594
1.580
1.360
1.342
1.327
0.912
0.907
0.893



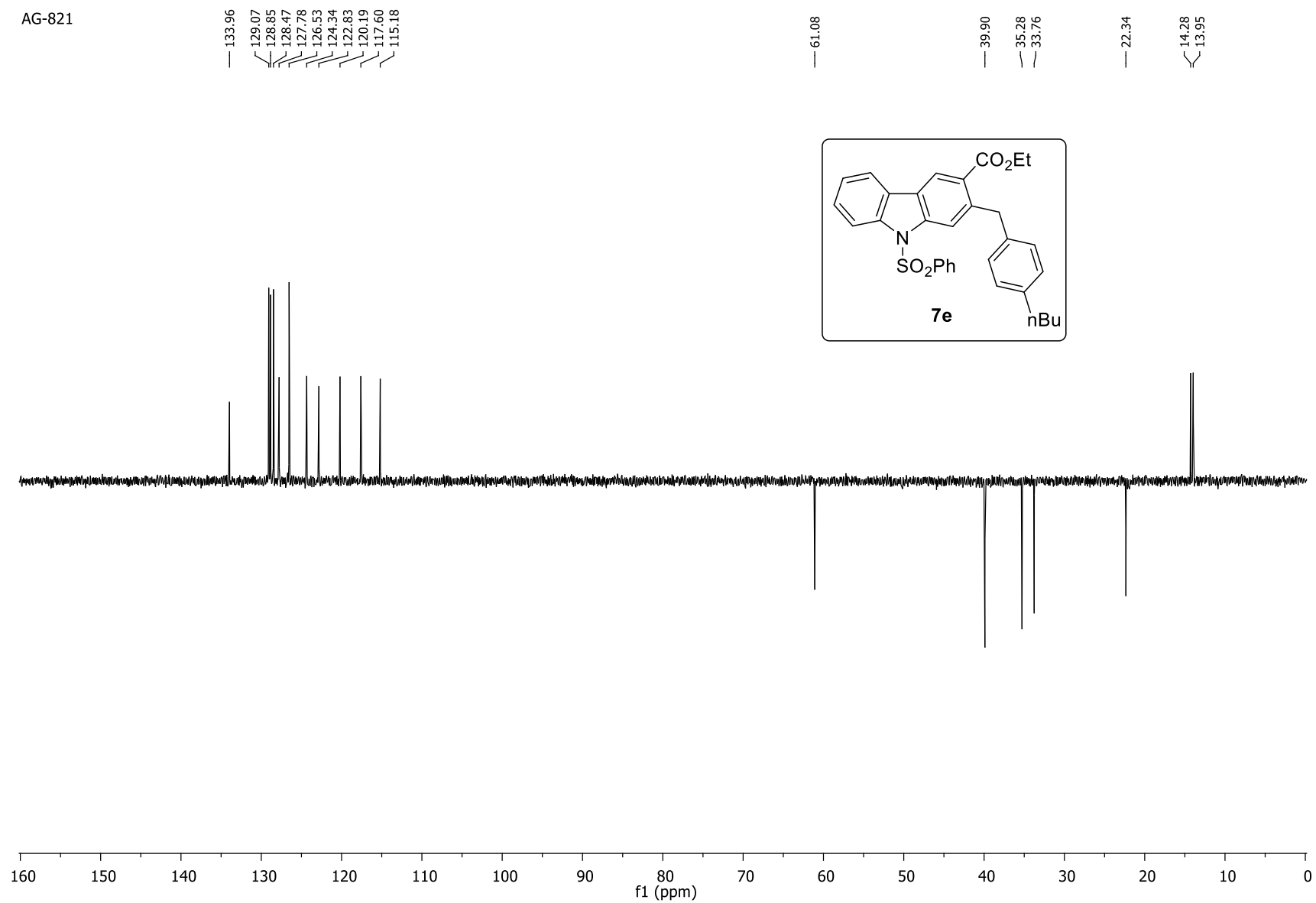
¹H NMR (500 MHz, CDCl₃) spectrum of compound **7e**

AG-821



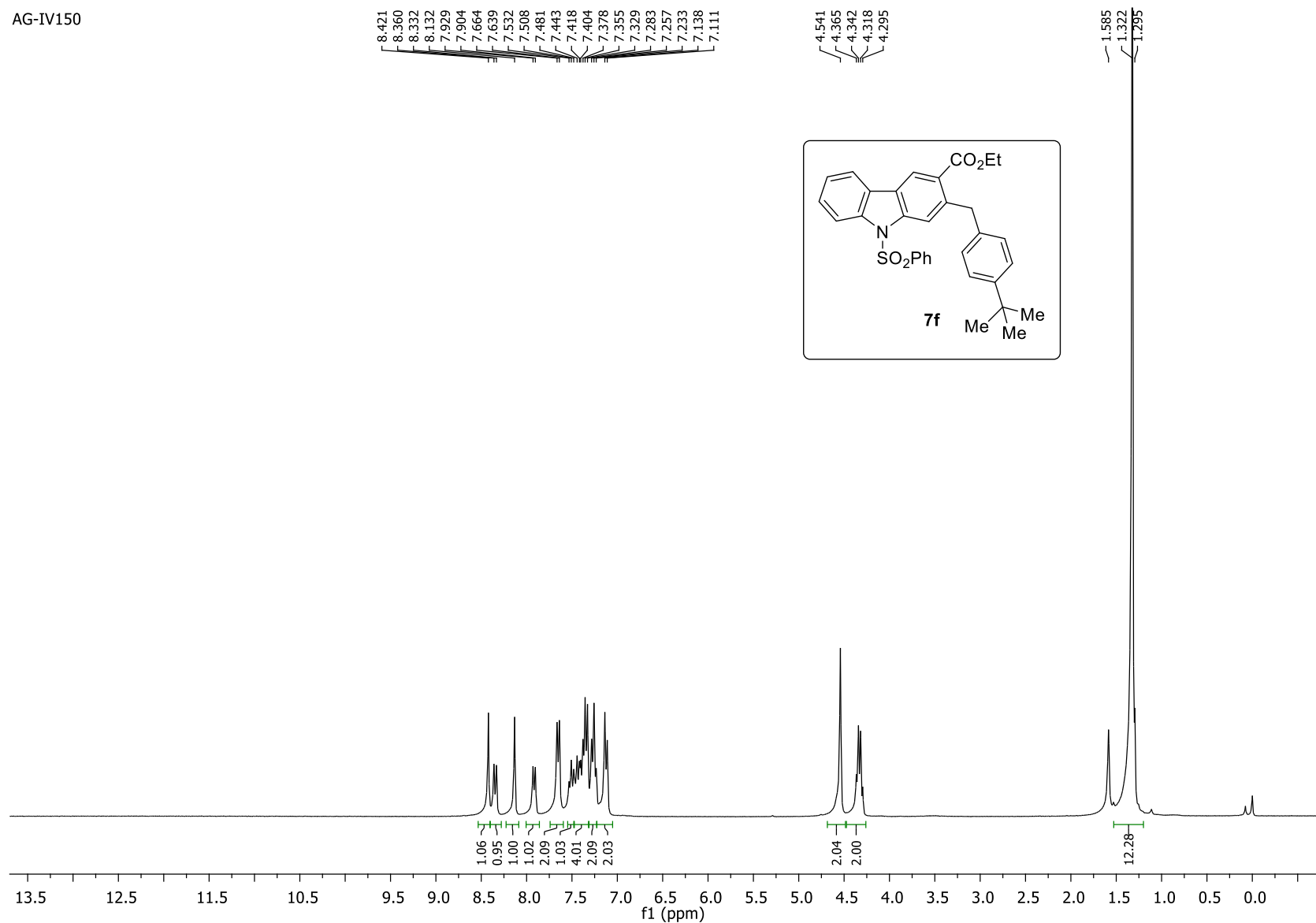
$^{13}\text{C} \{^1\text{H}\}$ NMR (125 MHz, CDCl_3) spectrum of compound **7e**

AG-821



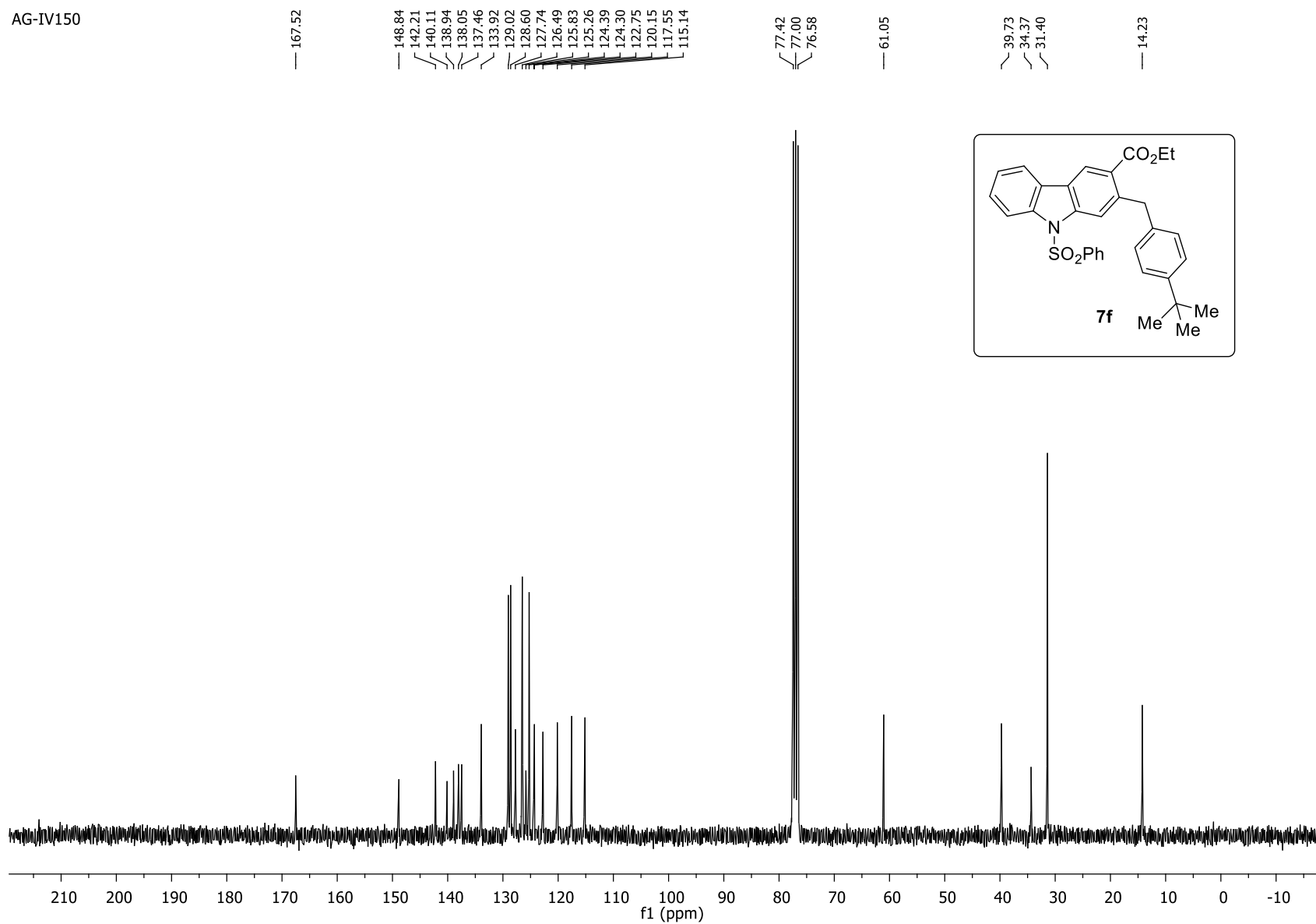
DEPT-135 NMR (125 MHz, CDCl_3) spectrum of compound **7e**

AG-IV150



^1H NMR (300 MHz, CDCl_3) spectrum of compound **7f**

AG-IV150



$^{13}\text{C} \{^1\text{H}\}$ NMR (75 MHz, CDCl_3) spectrum of compound **7f**

AG-IV150

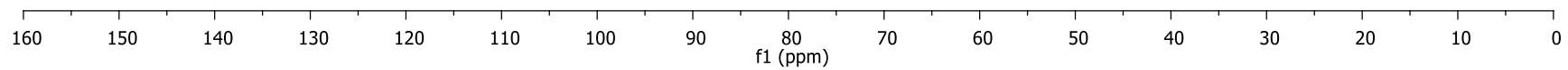
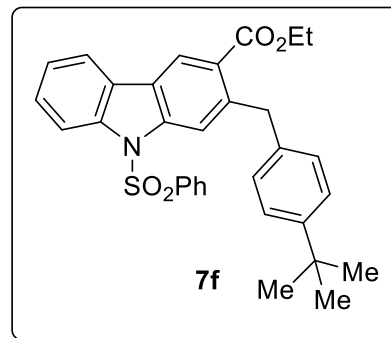
133.97
129.07
128.65
127.79
126.54
125.30
124.35
122.80
120.20
117.60
115.18

61.10

39.78

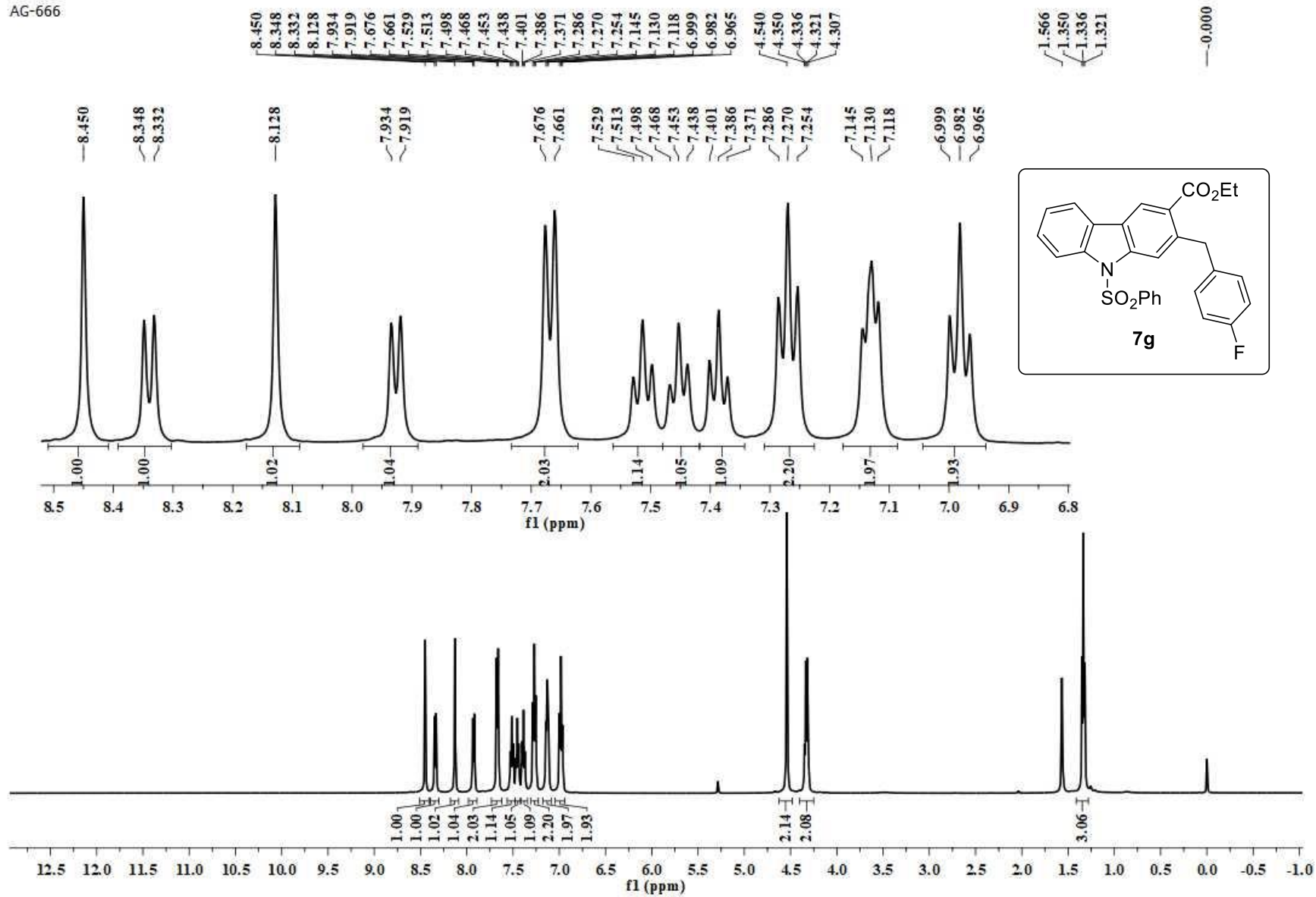
31.45

14.28



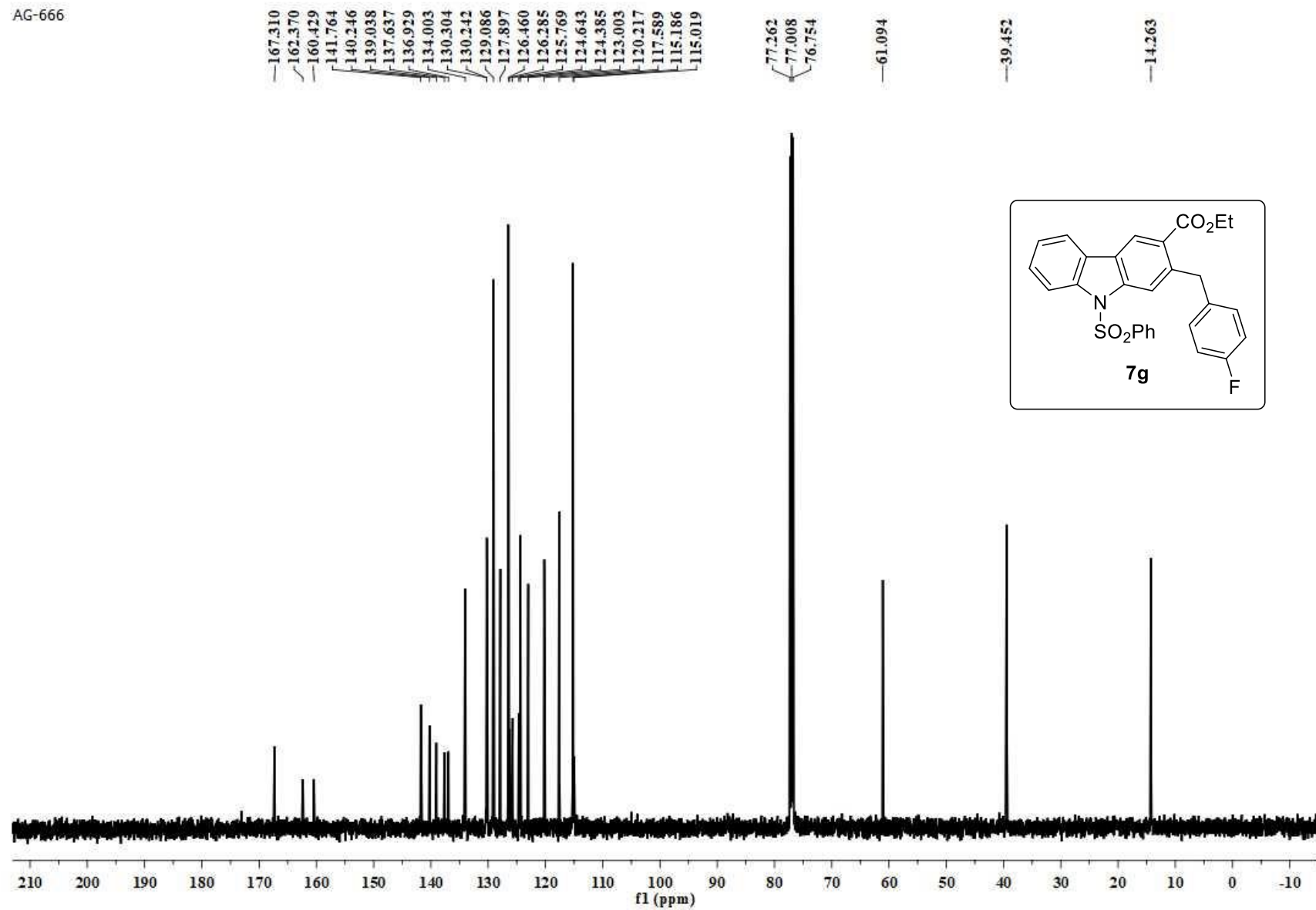
DEPT-135 NMR (75 MHz, CDCl_3) spectrum of compound **7f**

AG-666



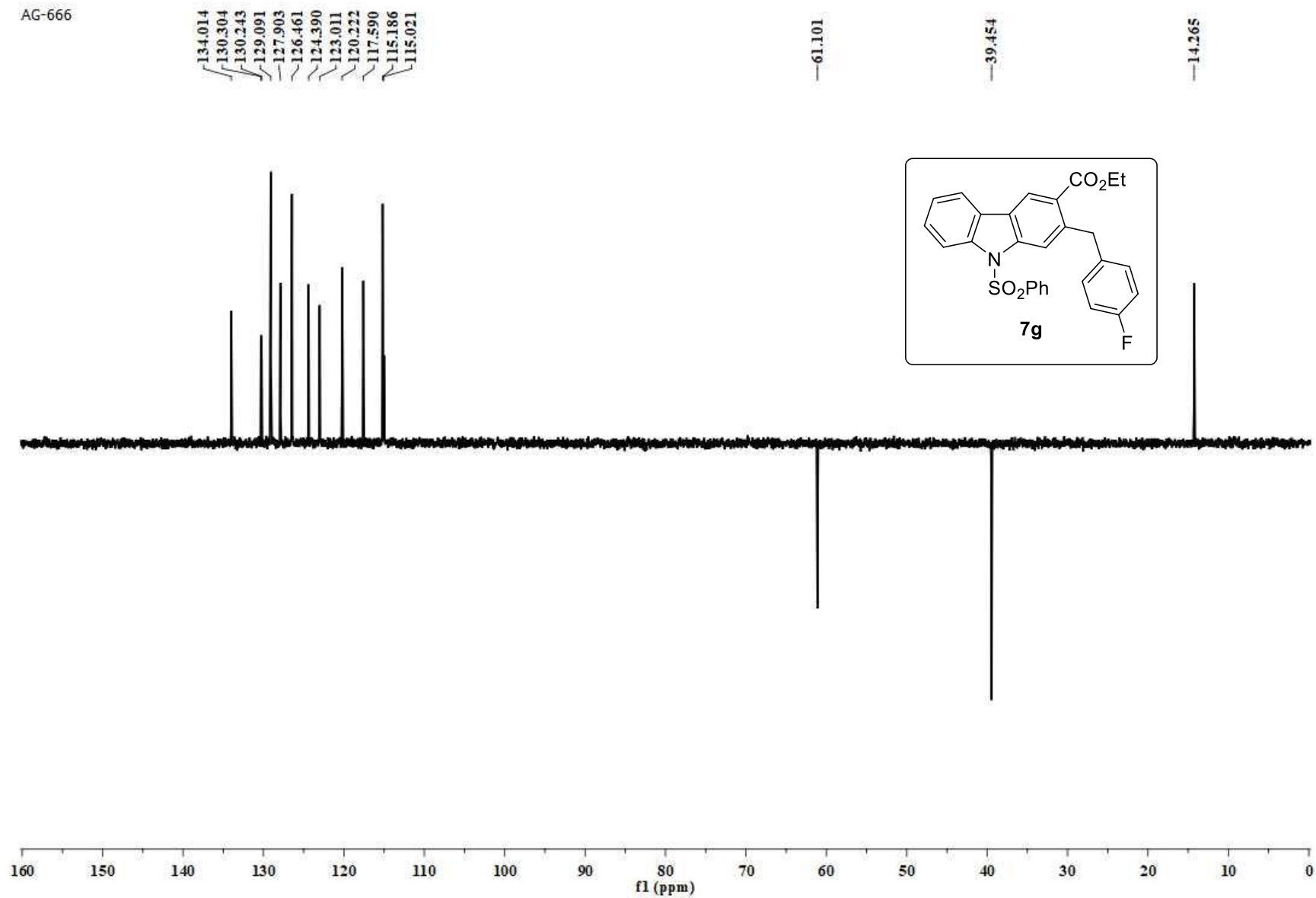
¹H NMR (500 MHz, CDCl₃) spectrum of compound 7g

AG-666



$^{13}\text{C} \{^1\text{H}\}$ NMR (125 MHz, CDCl_3) spectrum of compound **7g**

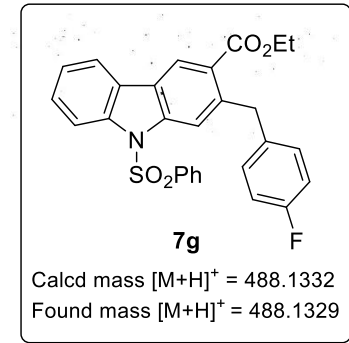
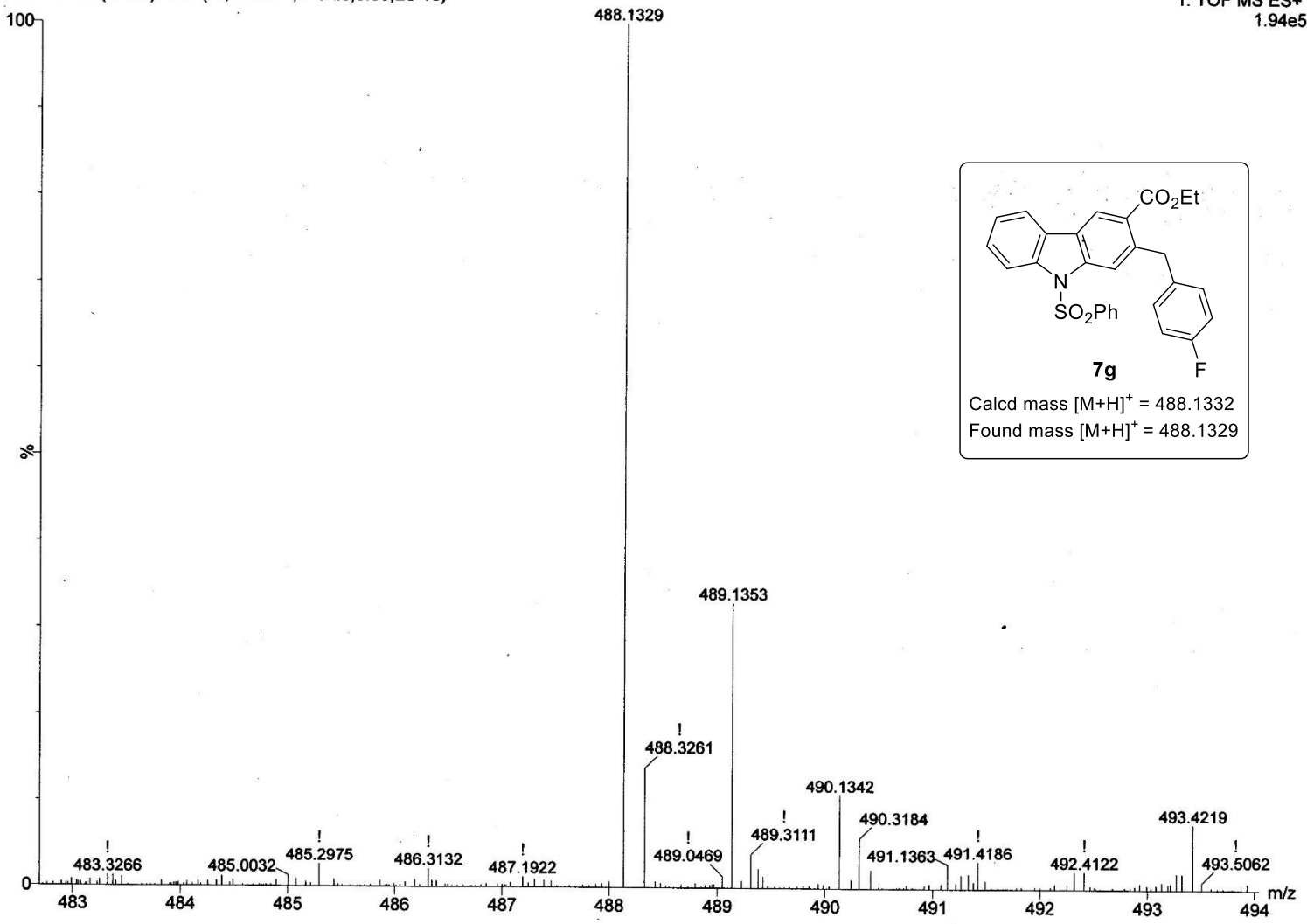
AG-666



DEPT-135 NMR (125 MHz, CDCl₃) spectrum of compound **7g**

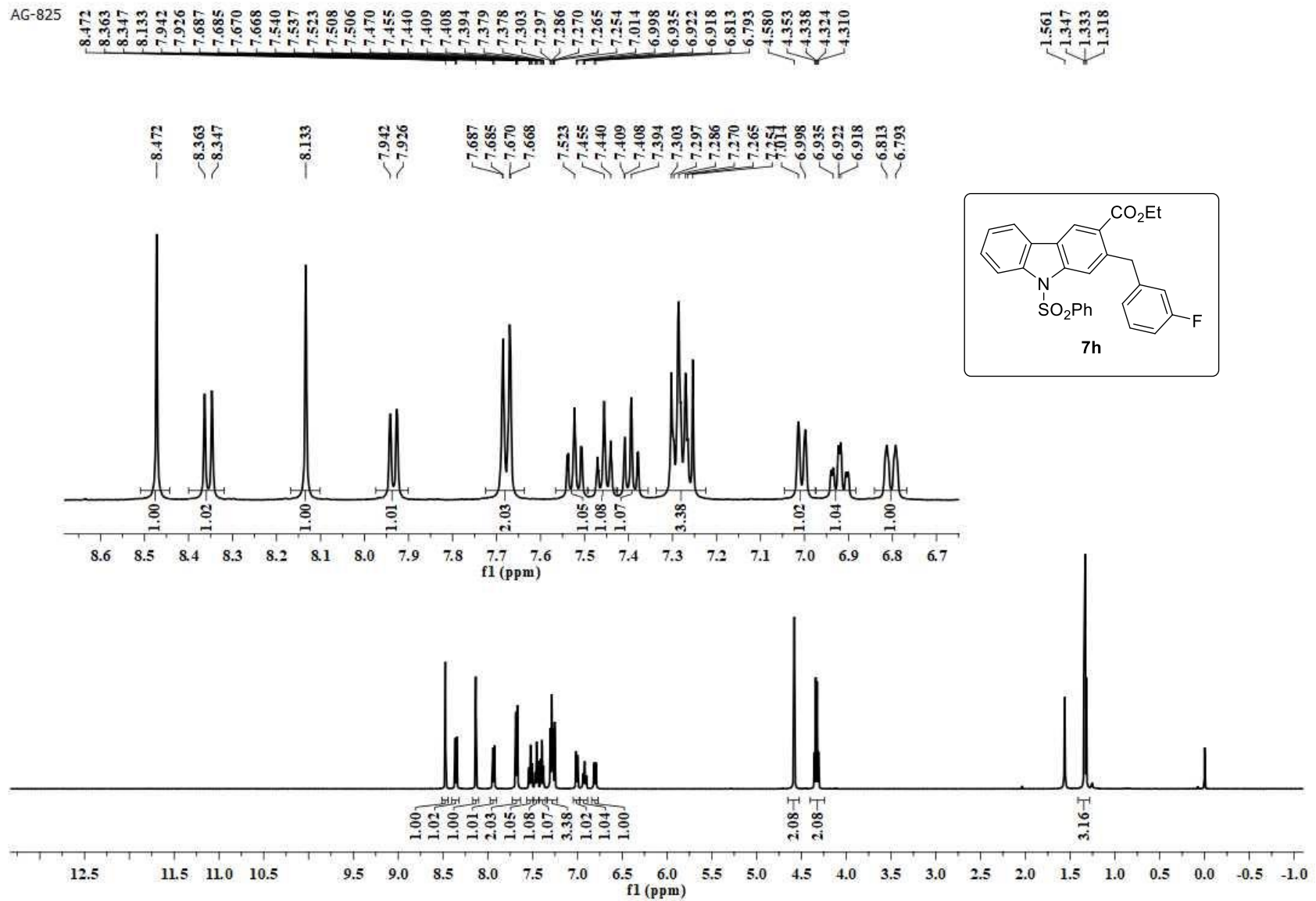
DRAKM
AG-666-R 3 (0.127) AM2 (Ar,20000.0,556.28,0.00,LS 10)

1: TOF MS ES+
1.94e5



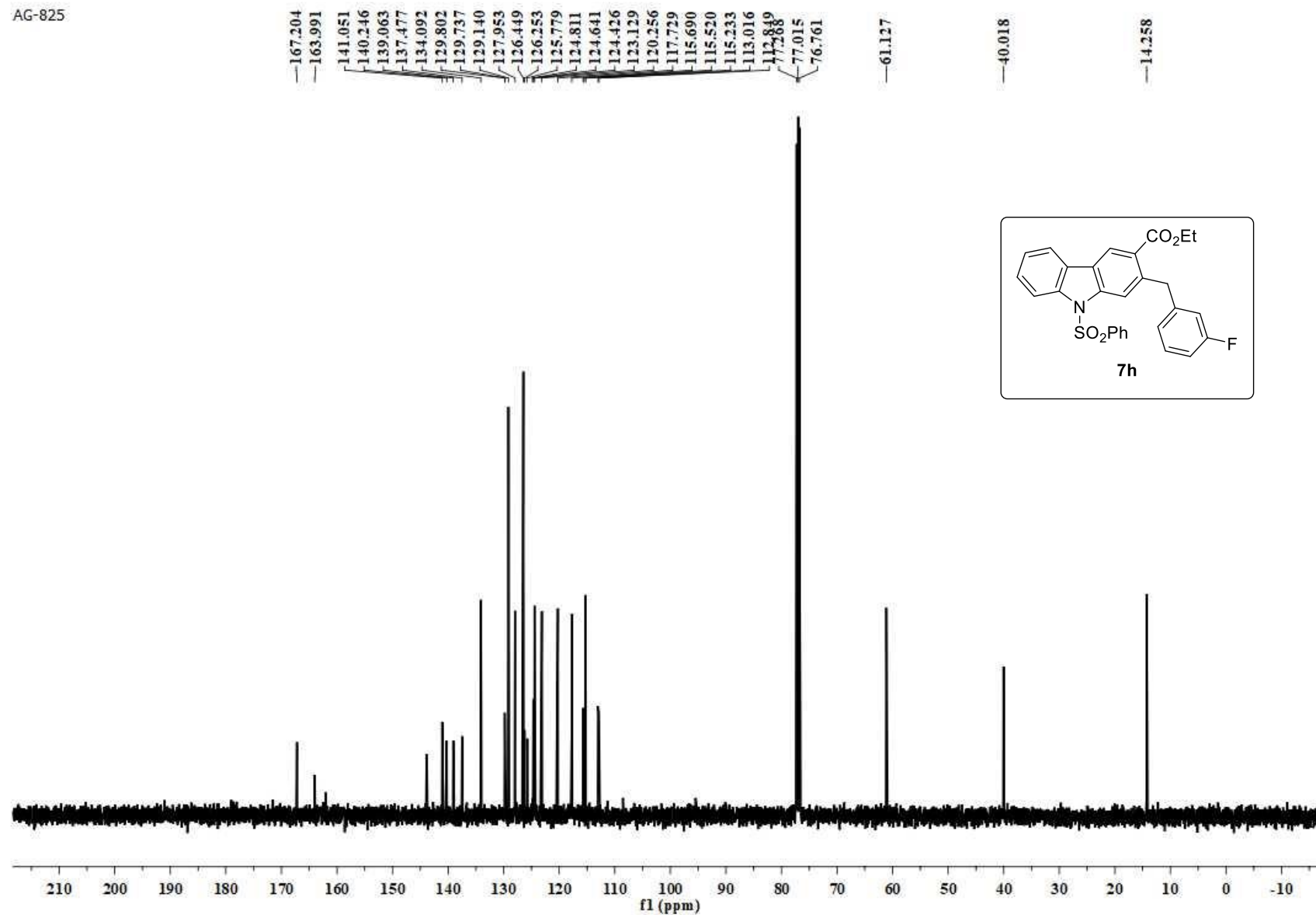
HRMS Spectrum of compound 7g

AG-825



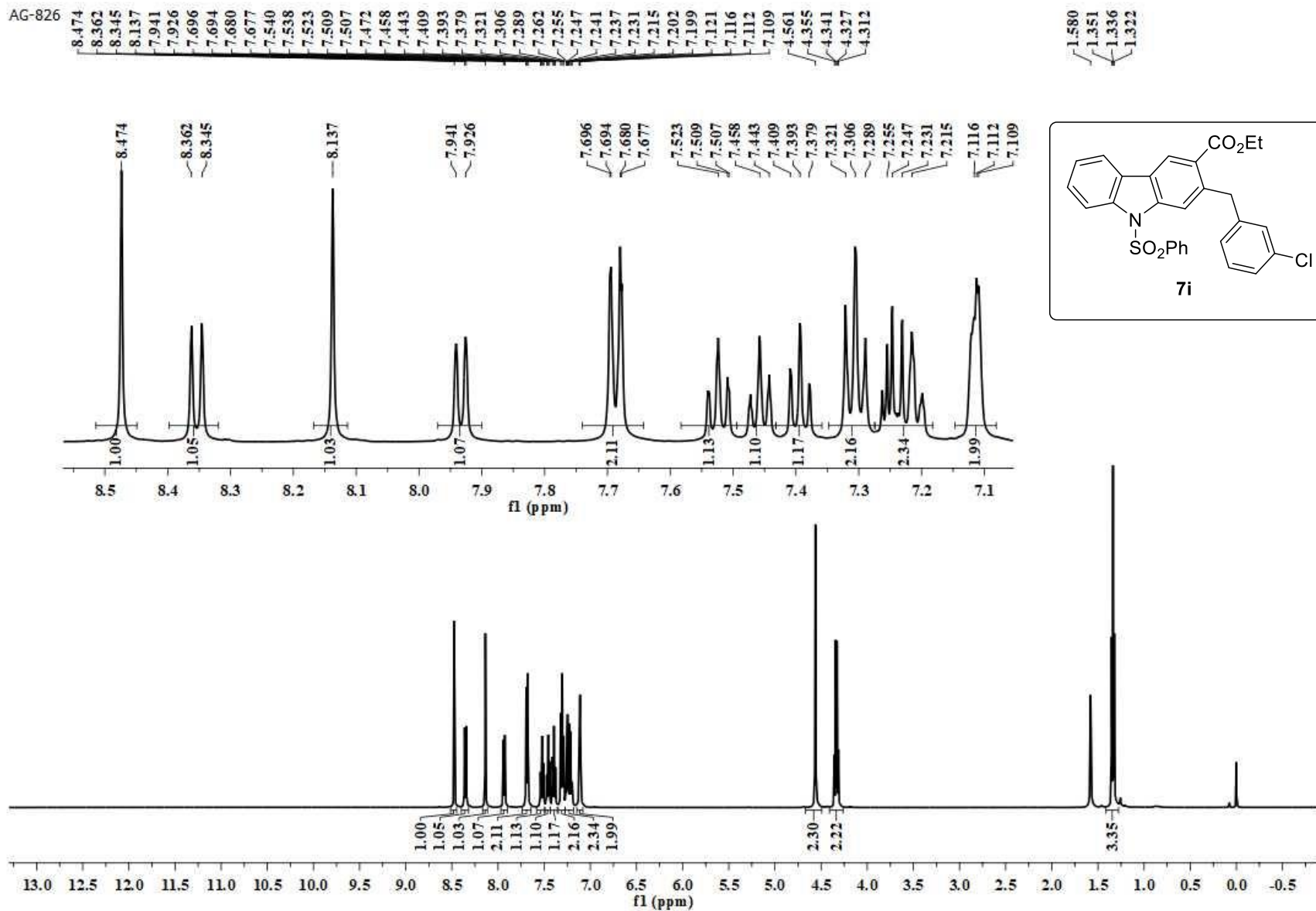
¹H NMR (300 MHz, CDCl₃) spectrum of compound **7h**

AG-825



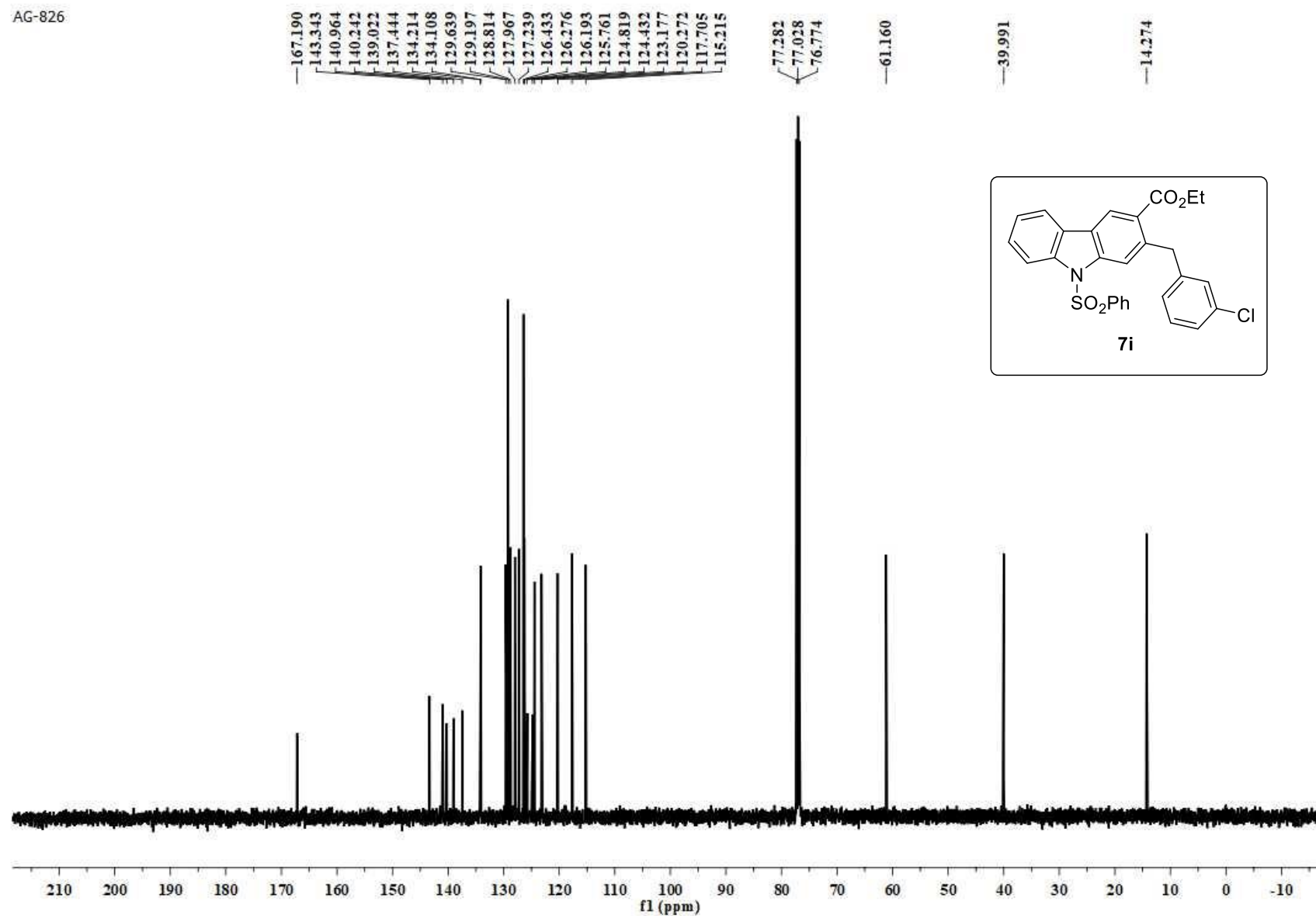
^{13}C $\{^1\text{H}\}$ NMR (75 MHz, CDCl_3) spectrum of compound **7h**

AG-826

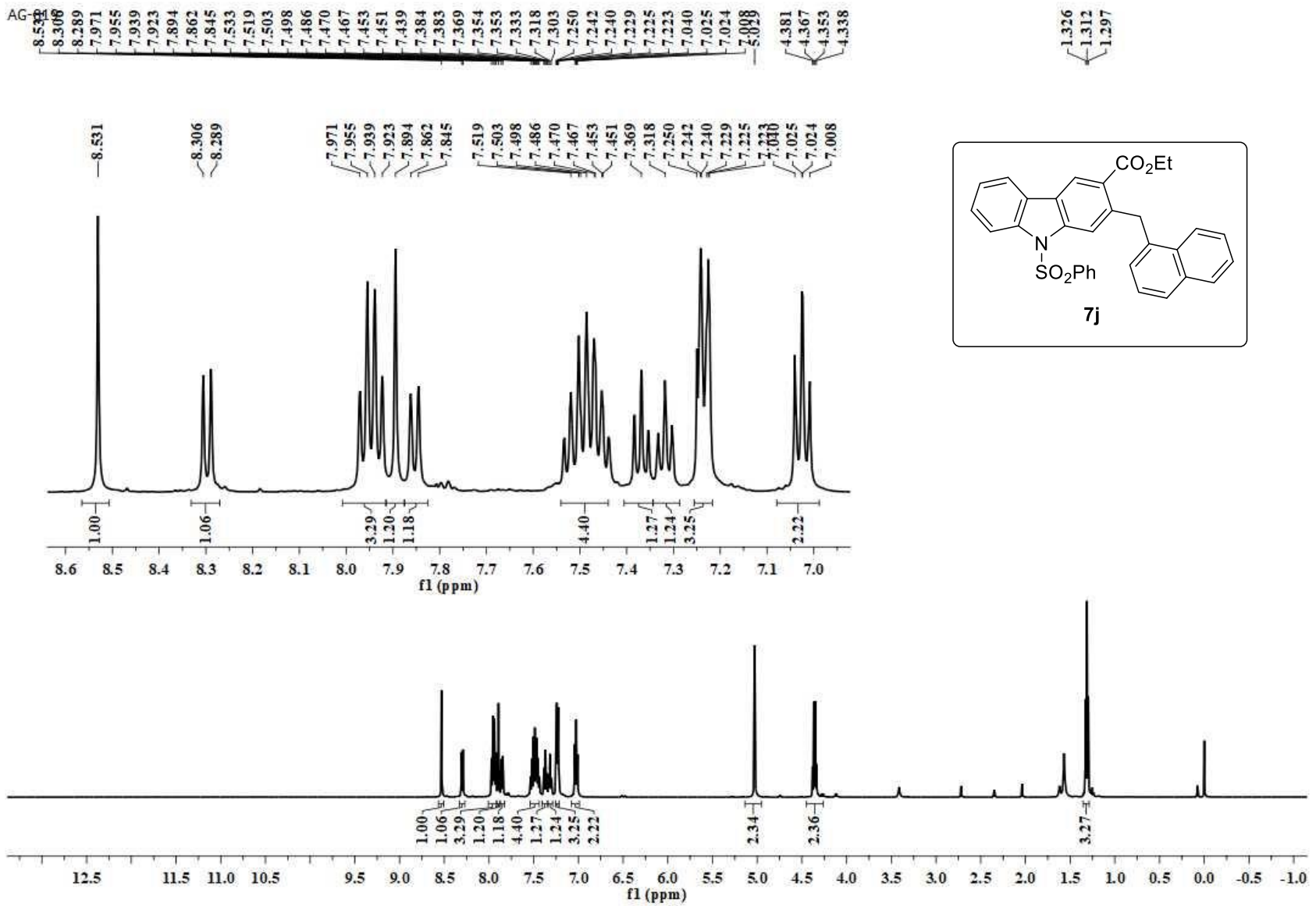


^1H NMR (300 MHz, CDCl_3) spectrum of compound **7i**

AG-826

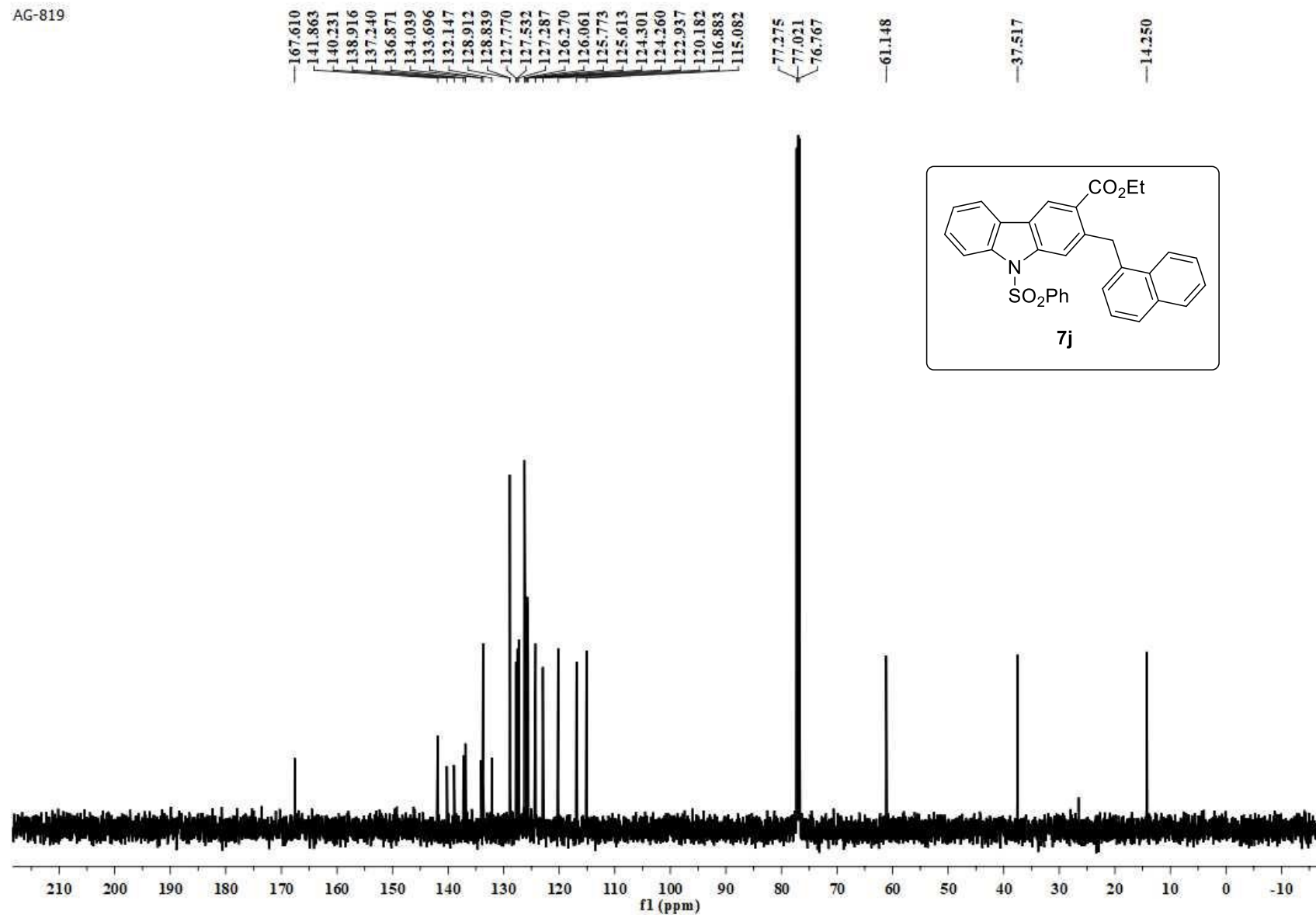


^{13}C $\{^1\text{H}\}$ NMR (75 MHz, CDCl_3) spectrum of compound **7i**



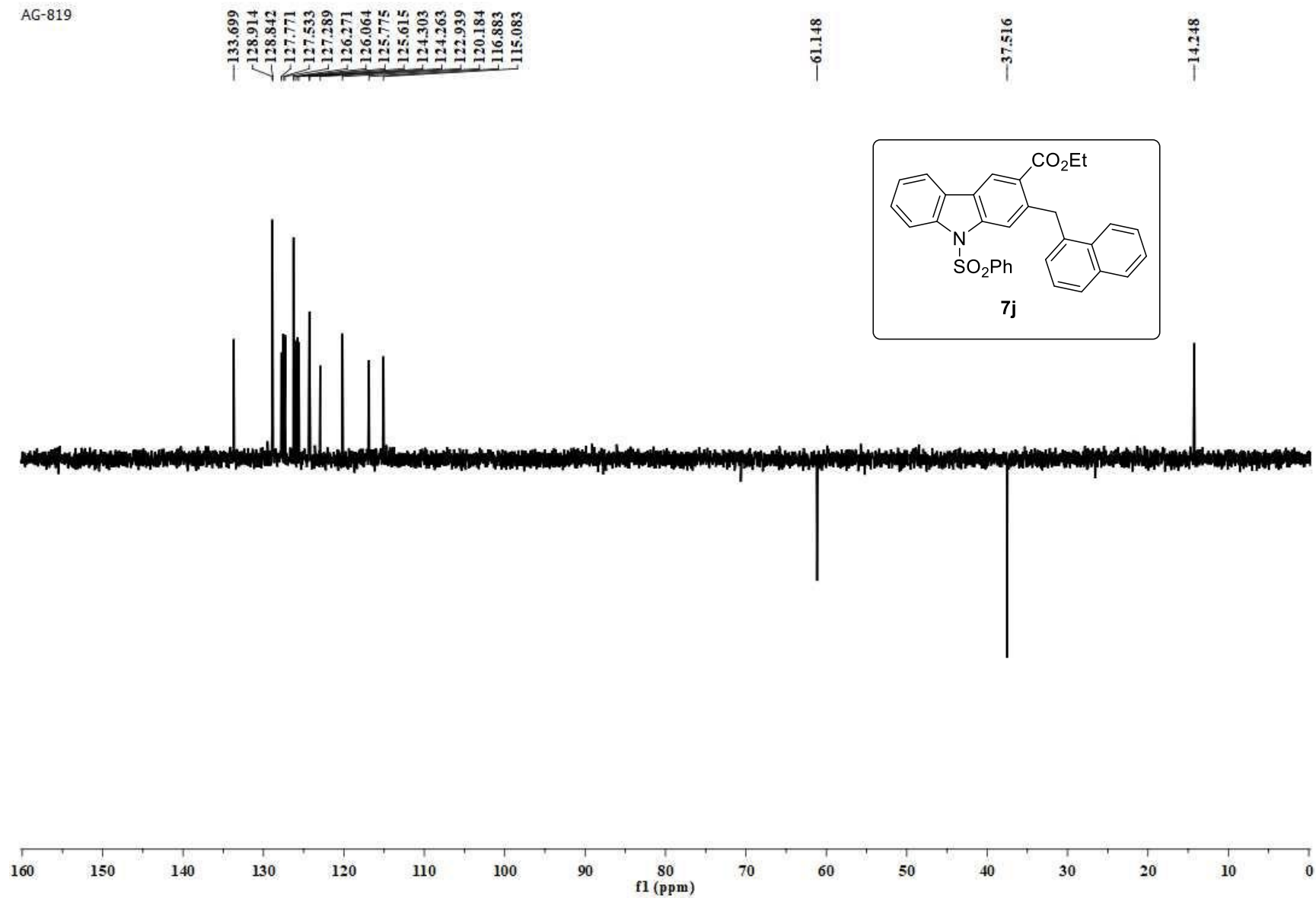
^1H NMR (500 MHz, CDCl_3) spectrum of compound 7j

AG-819



^{13}C $\{^1\text{H}\}$ NMR (125 MHz, CDCl_3) spectrum of compound **7j**

AG-819



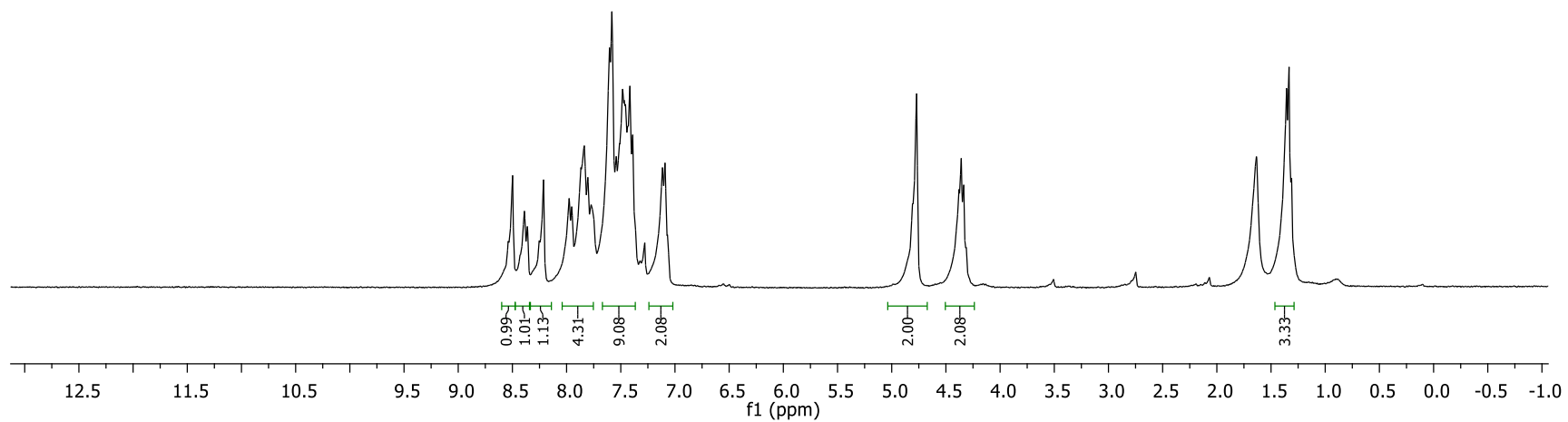
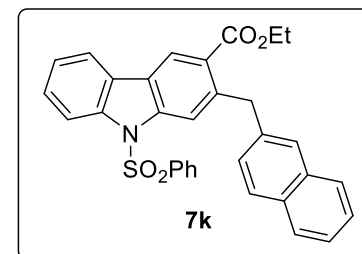
DEPT-135 NMR (125 MHz, CDCl_3) spectrum of compound **7j**

AG-V-106

8.498
8.389
8.362
8.213
7.976
7.952
7.836
7.804
7.773
7.604
7.582
7.542
7.484
7.469
7.459
7.416
7.390
7.279
7.115
7.091

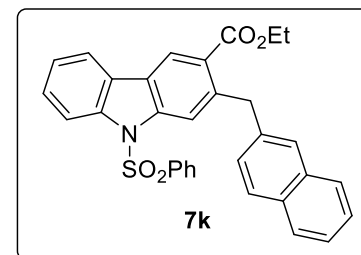
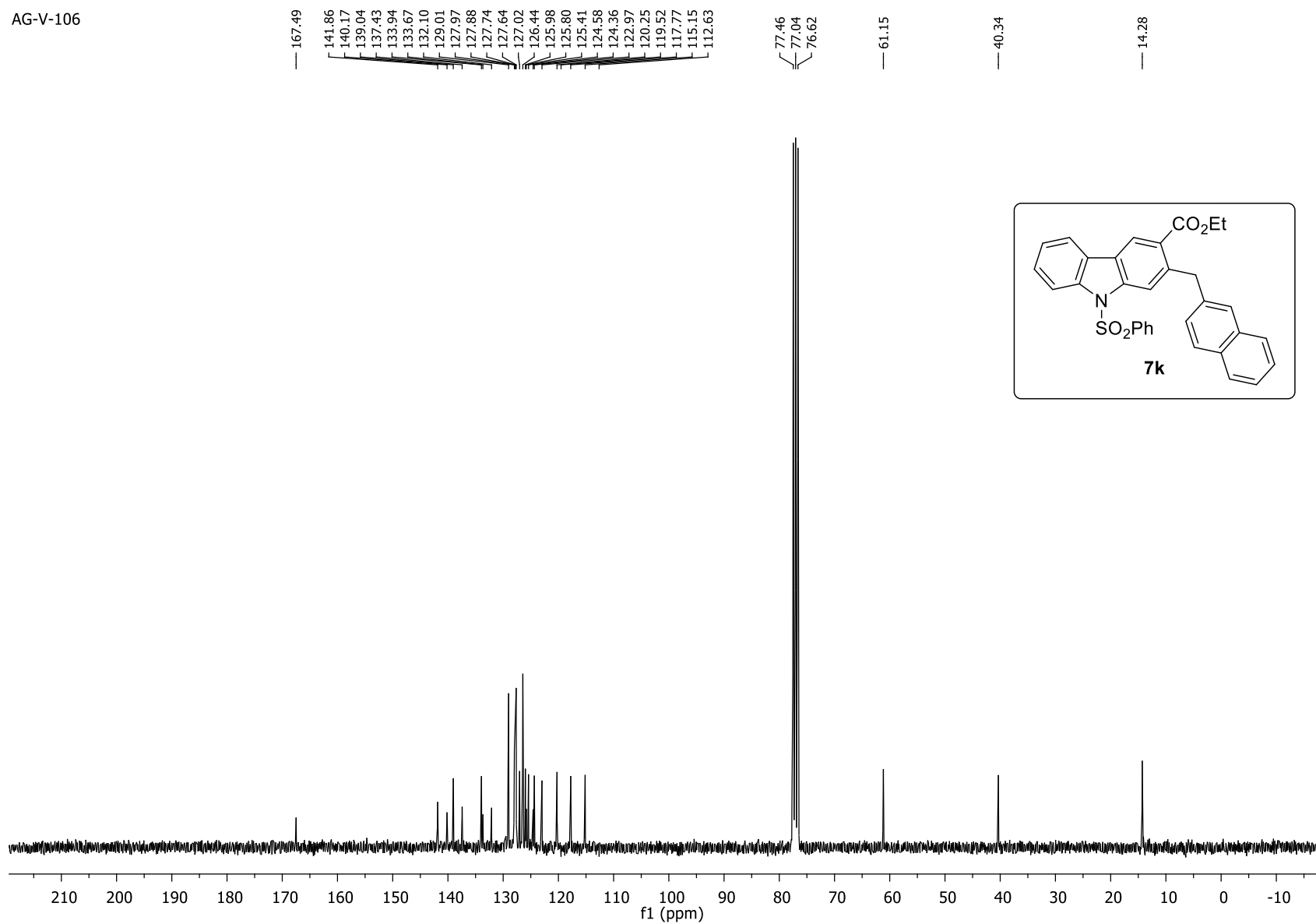
4.770
4.379
4.358
4.335
4.311

1.635
1.356
1.334
1.310



^1H NMR (300 MHz, CDCl_3) spectrum of compound **7k**

AG-V-106



$^{13}\text{C} \{^1\text{H}\}$ NMR (75 MHz, CDCl_3) spectrum of compound 7k

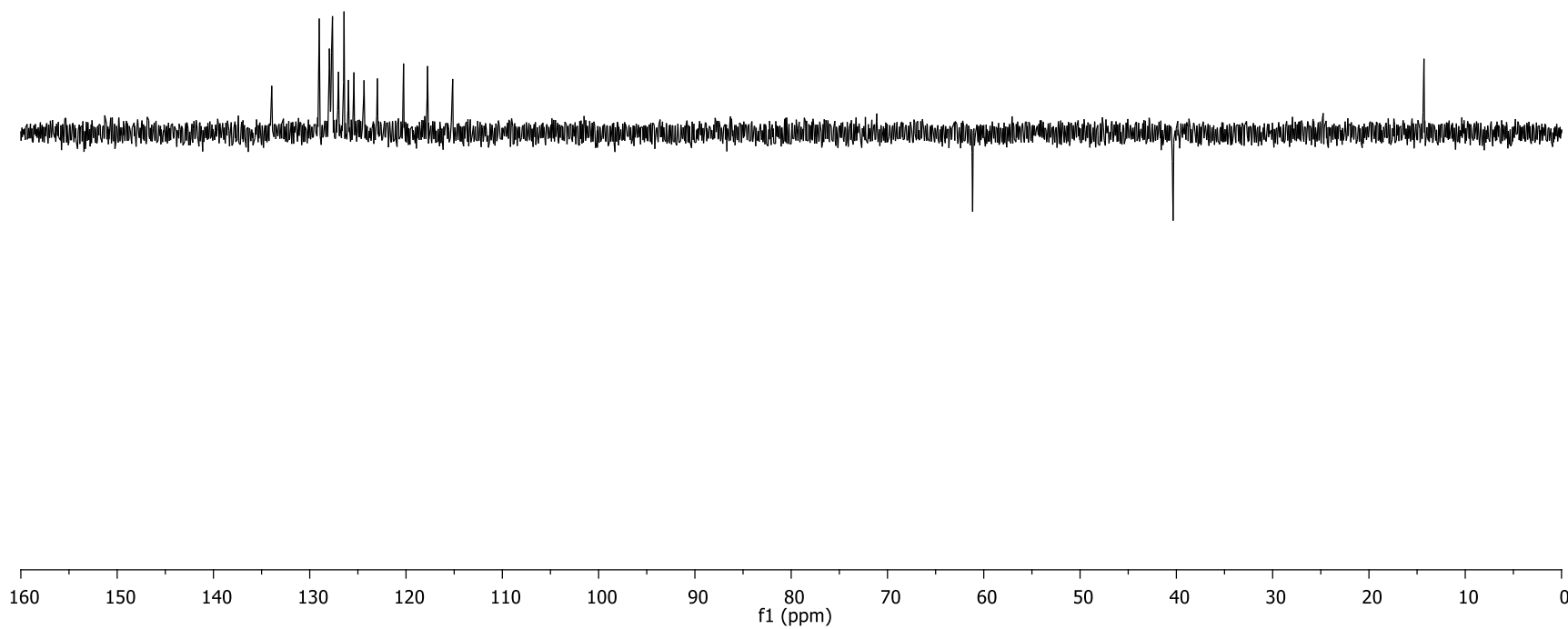
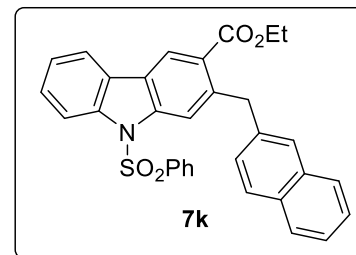
AG-V-106

133.95
129.02
127.97
127.88
127.73
127.64
127.02
126.44
125.98
125.41
124.36
122.98
120.26
117.76
115.15

61.16

40.34

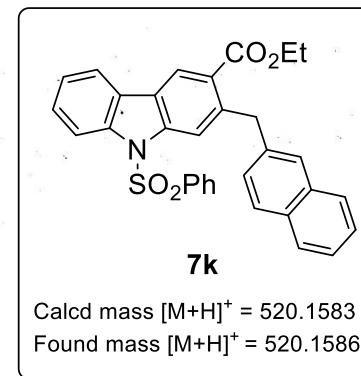
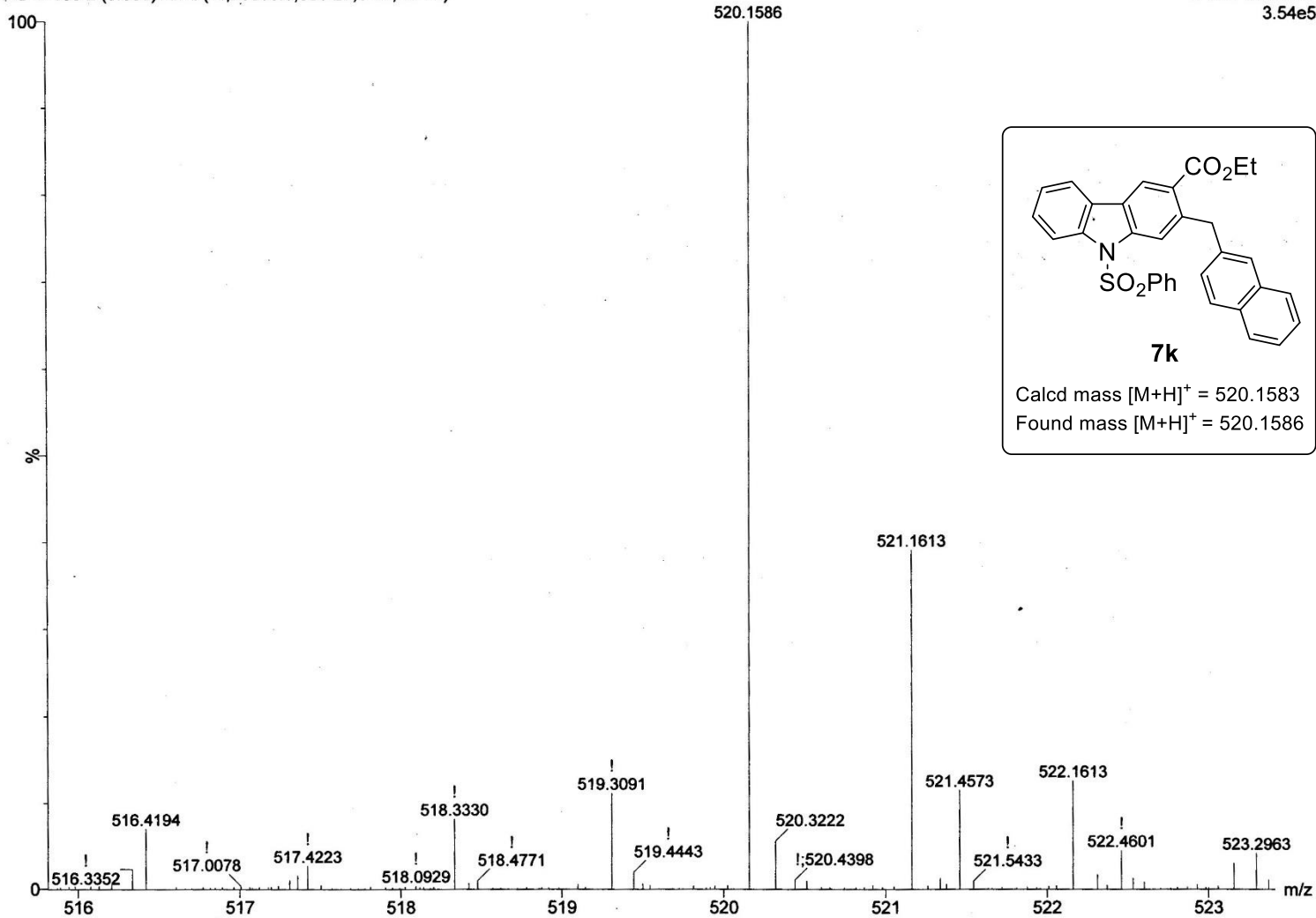
14.29



DEPT-135 NMR (75 MHz, CDCl₃) spectrum of compound **7k**

DRAKM
AG-V-106 2 (0.090) AM2 (Ar,20000.0,556.28,0.00,LS 10)

1: TOF MS ES+
3.54e5



HRMS Spectrum of compound **7k**

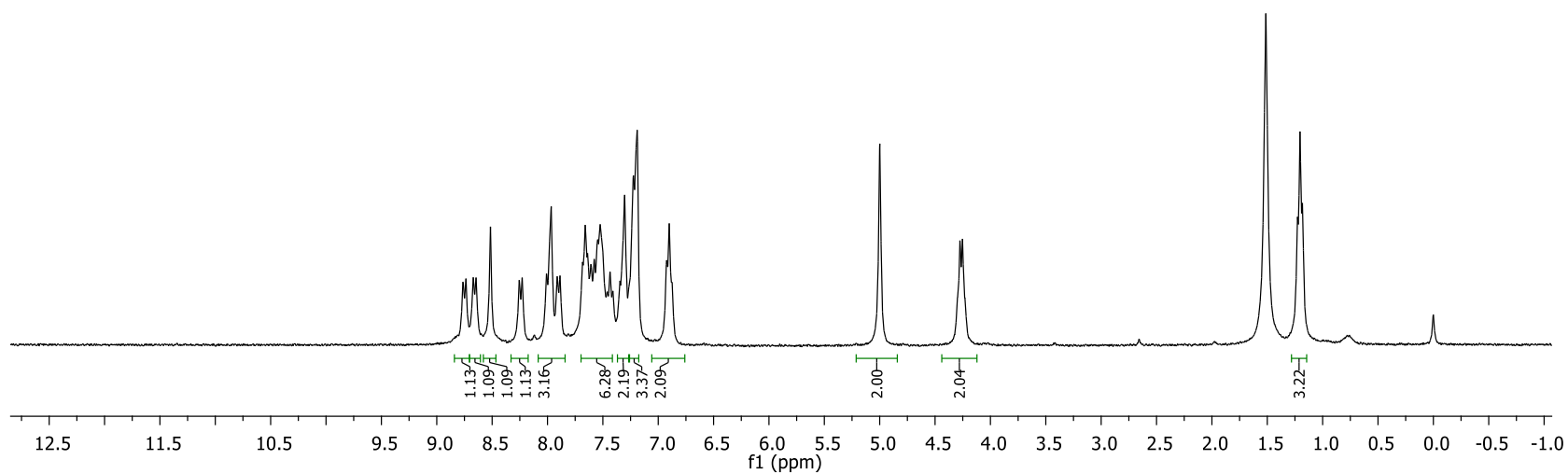
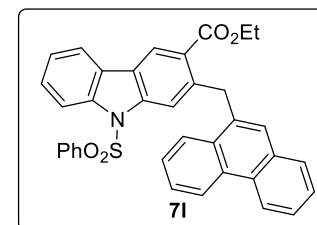
AG-V-108R3

8.764
8.736
8.670
8.645
8.515
8.255
8.228
8.009
7.966
7.912
7.887
7.660
7.638
7.608
7.578
7.546
7.524
7.460
7.436
7.410
7.345
7.303
7.224
7.190
6.924
6.900
6.873

4.999

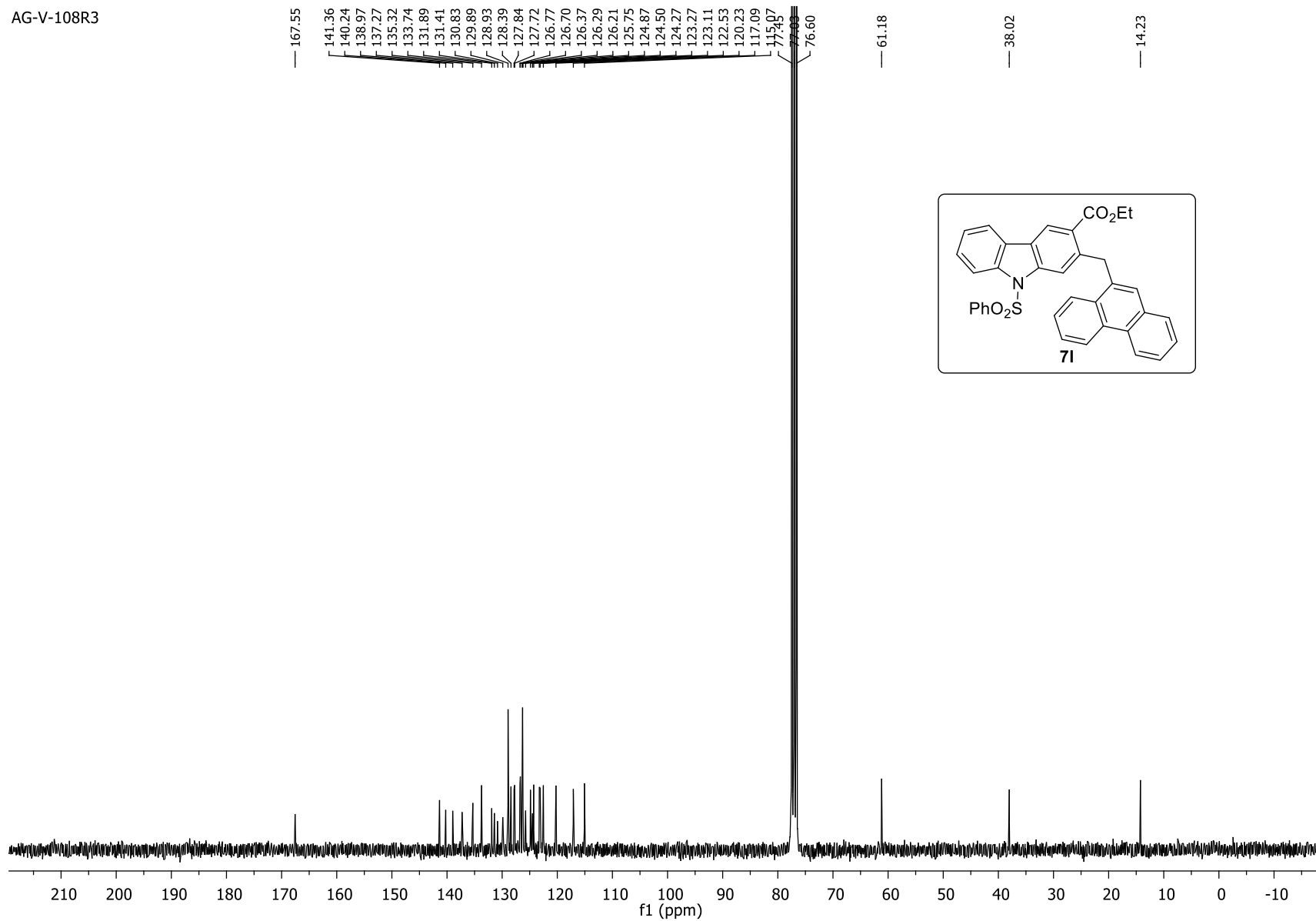
4.299
4.275
4.252
4.230

1.513
1.227
1.204
1.182



¹H NMR (300 MHz, CDCl₃) spectrum of compound **7I**

AG-V-108R3



¹³C {¹H} NMR (75 MHz, CDCl₃) spectrum of compound **71**

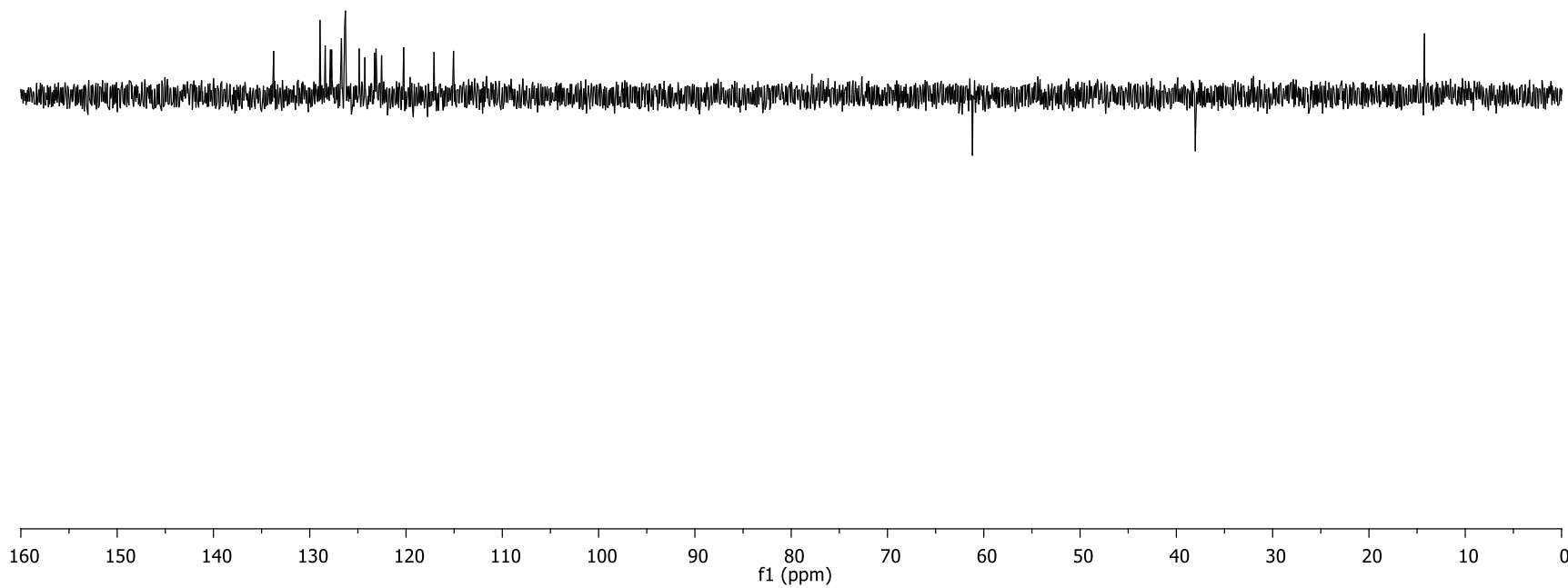
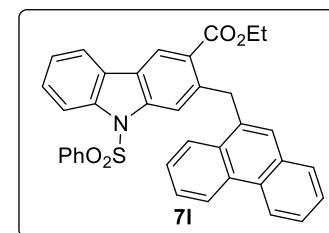
AG-V-108R3

133.75
128.93
128.39
127.84
127.72
126.71
126.37
126.30
124.87
124.28
123.27
123.11
122.54
120.24
117.09
115.07

61.18

38.03

14.24



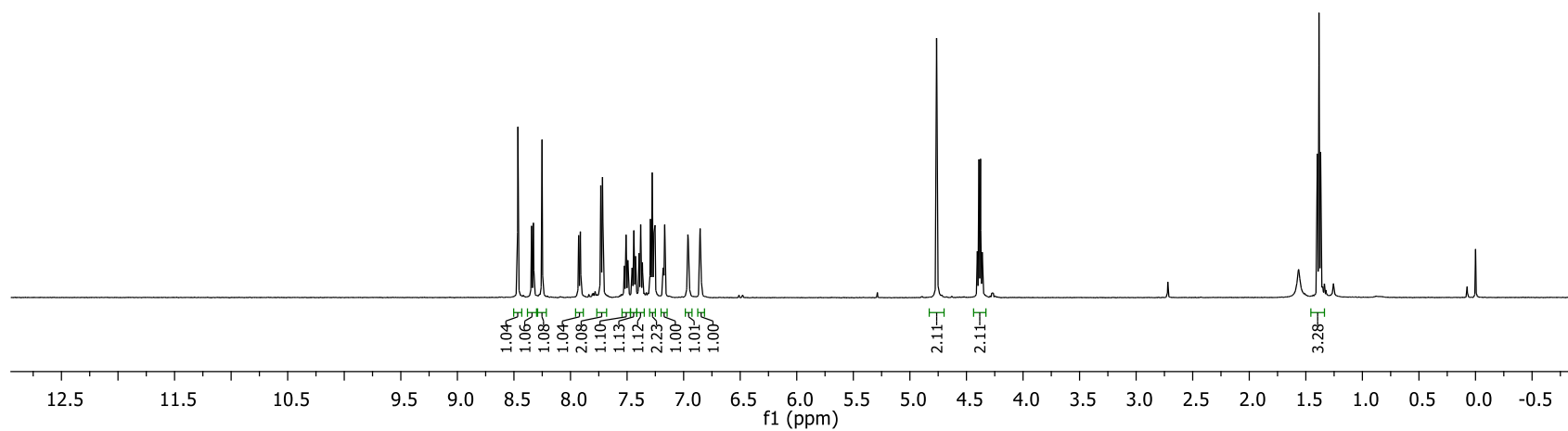
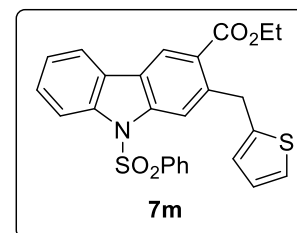
DEPT-135 NMR (75 MHz, CDCl₃) spectrum of compound **71**

AG-829

8.465
8.344
8.328
8.252
7.928
7.913
7.732
7.717
7.509
7.440
7.425
7.394
7.379
7.294
7.278
7.262
7.253
7.178
7.169
7.167
6.969
6.962
6.959
6.952
6.854
6.849

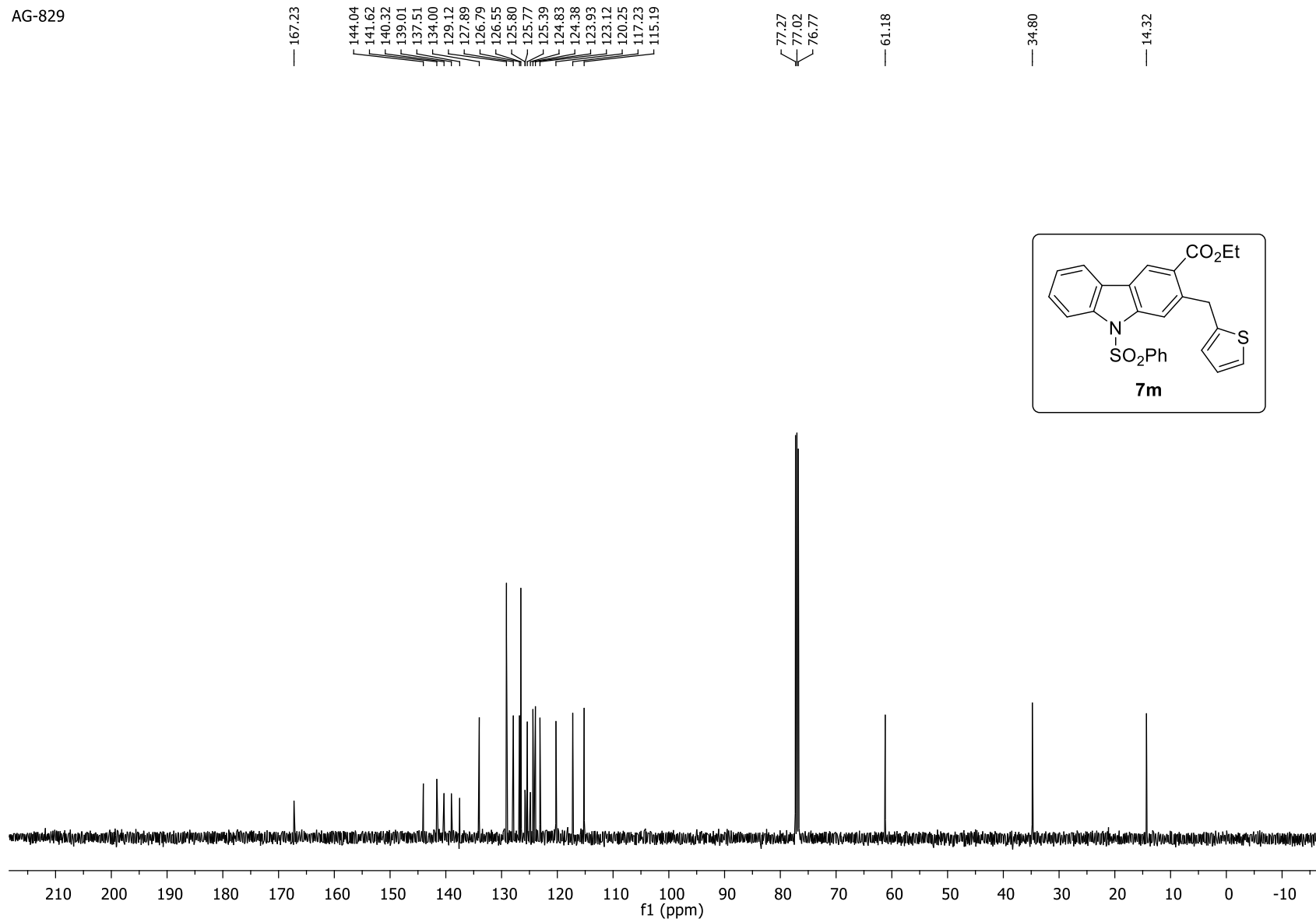
4.764
4.404
4.390
4.375
4.361

1.398
1.384
1.369



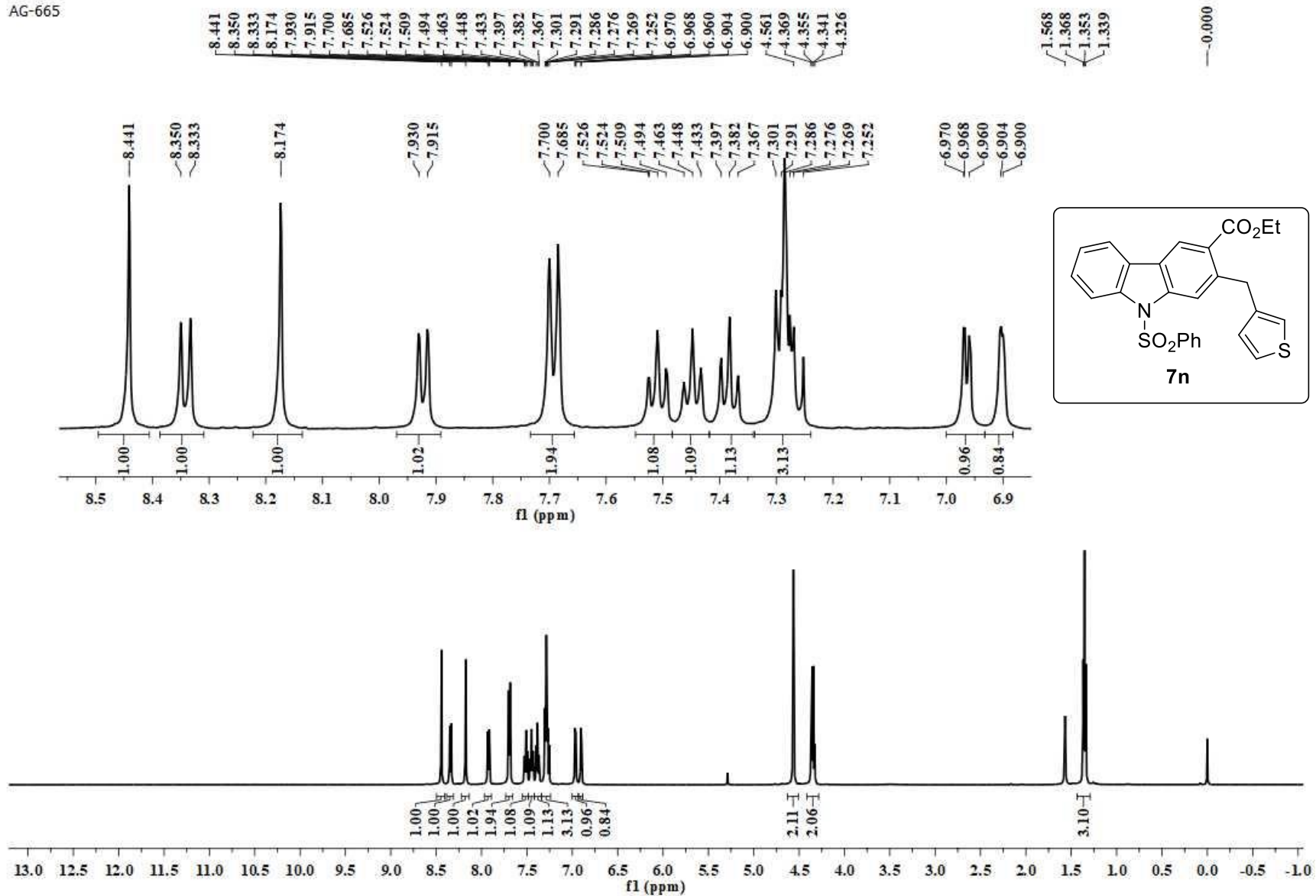
¹H NMR (500 MHz, CDCl₃) spectrum of compound **7m**

AG-829



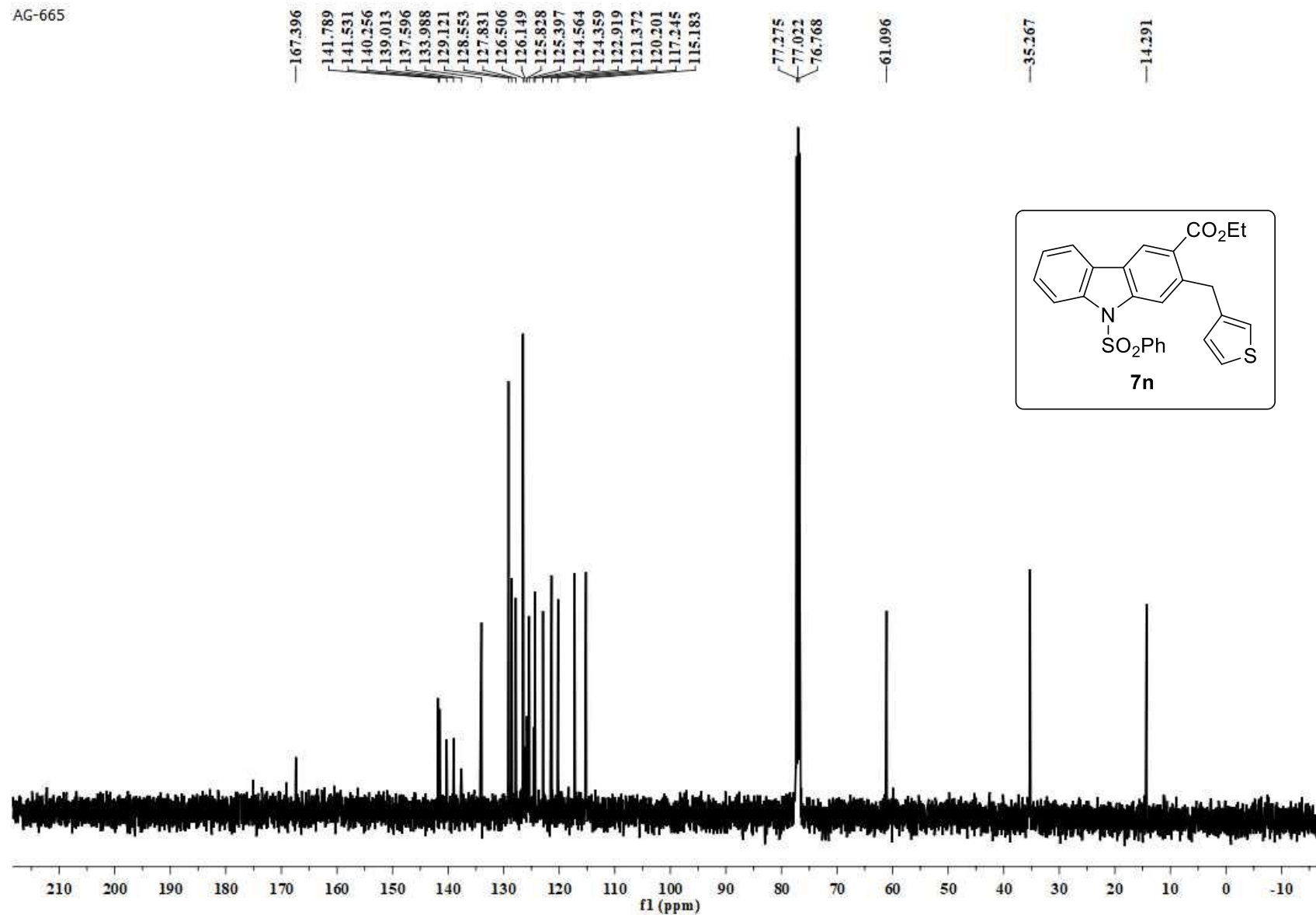
^{13}C { ^1H } NMR (125 MHz, CDCl_3) spectrum of compound **7m**

AG-665



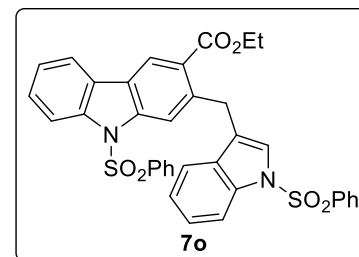
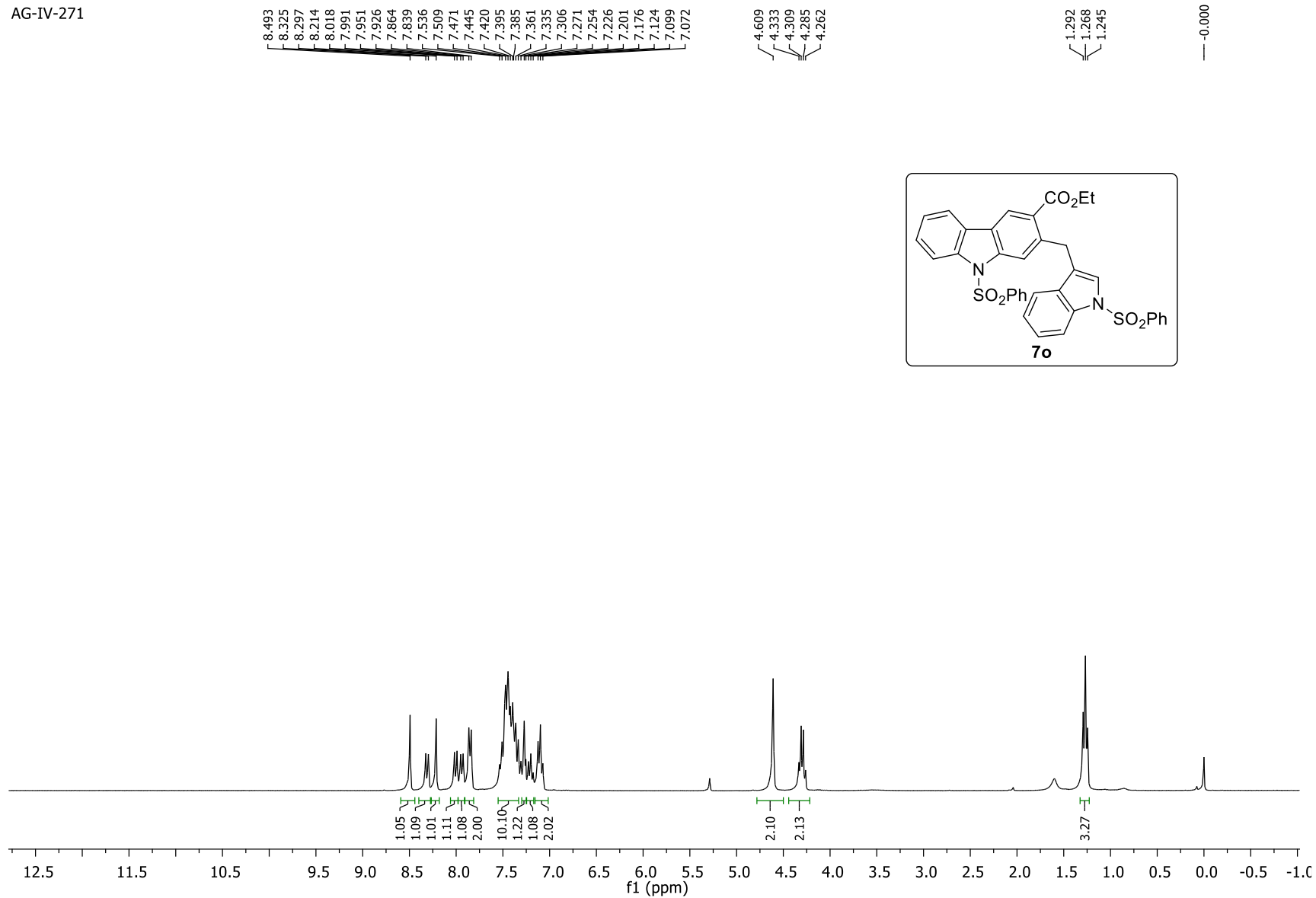
¹H NMR (500 MHz, CDCl₃) spectrum of compound 7n

AG-665



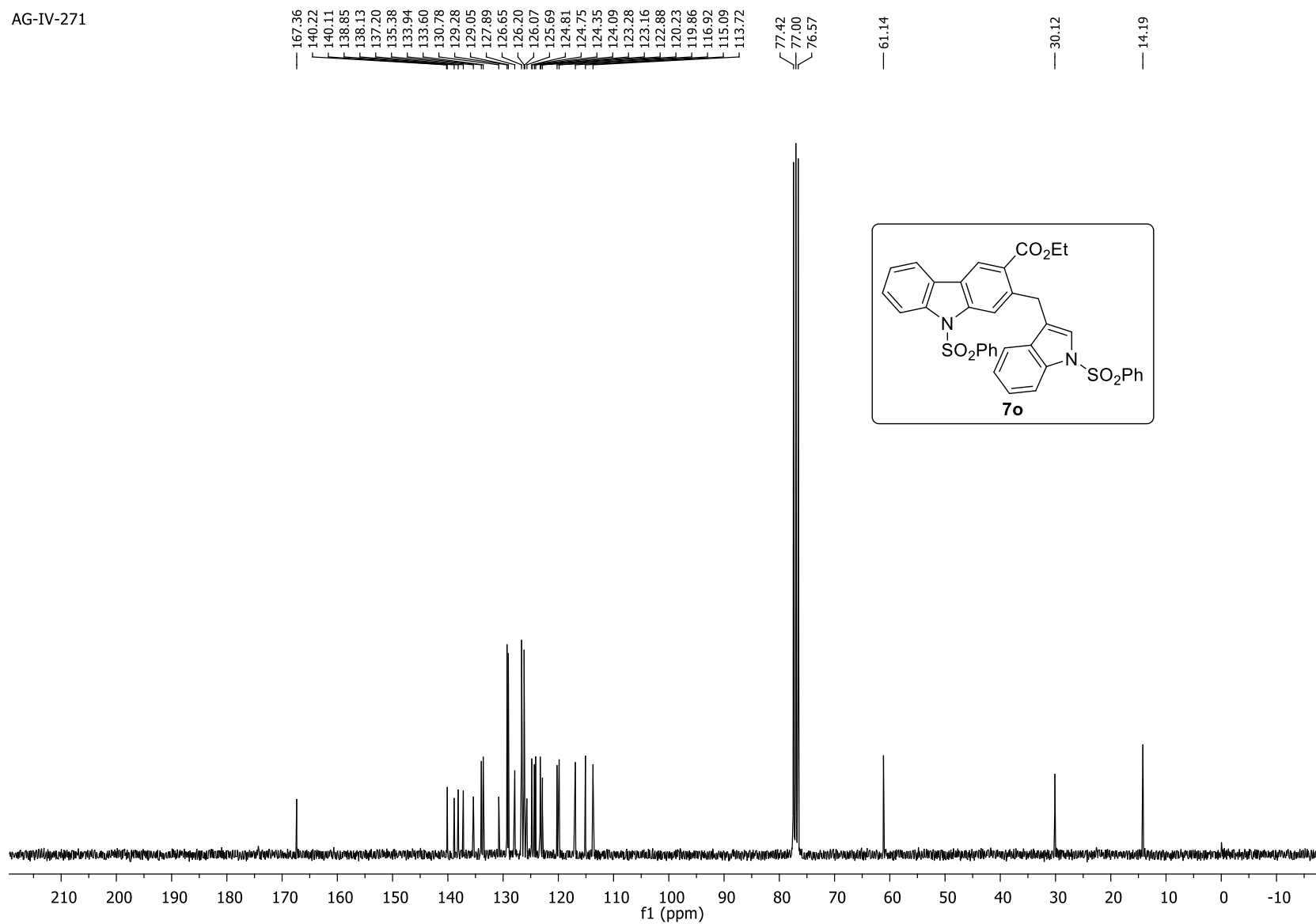
$^{13}\text{C} \{^1\text{H}\}$ NMR (125 MHz, CDCl_3) spectrum of compound **7n**

AG-IV-271



¹H NMR (300 MHz, CDCl₃) spectrum of compound **7o**

AG-IV-271



$^{13}\text{C} \{^1\text{H}\}$ NMR (75 MHz, CDCl_3) spectrum of compound **7o**

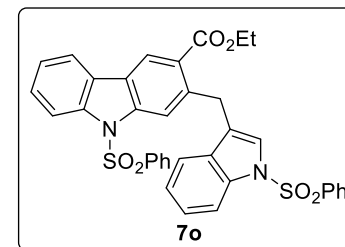
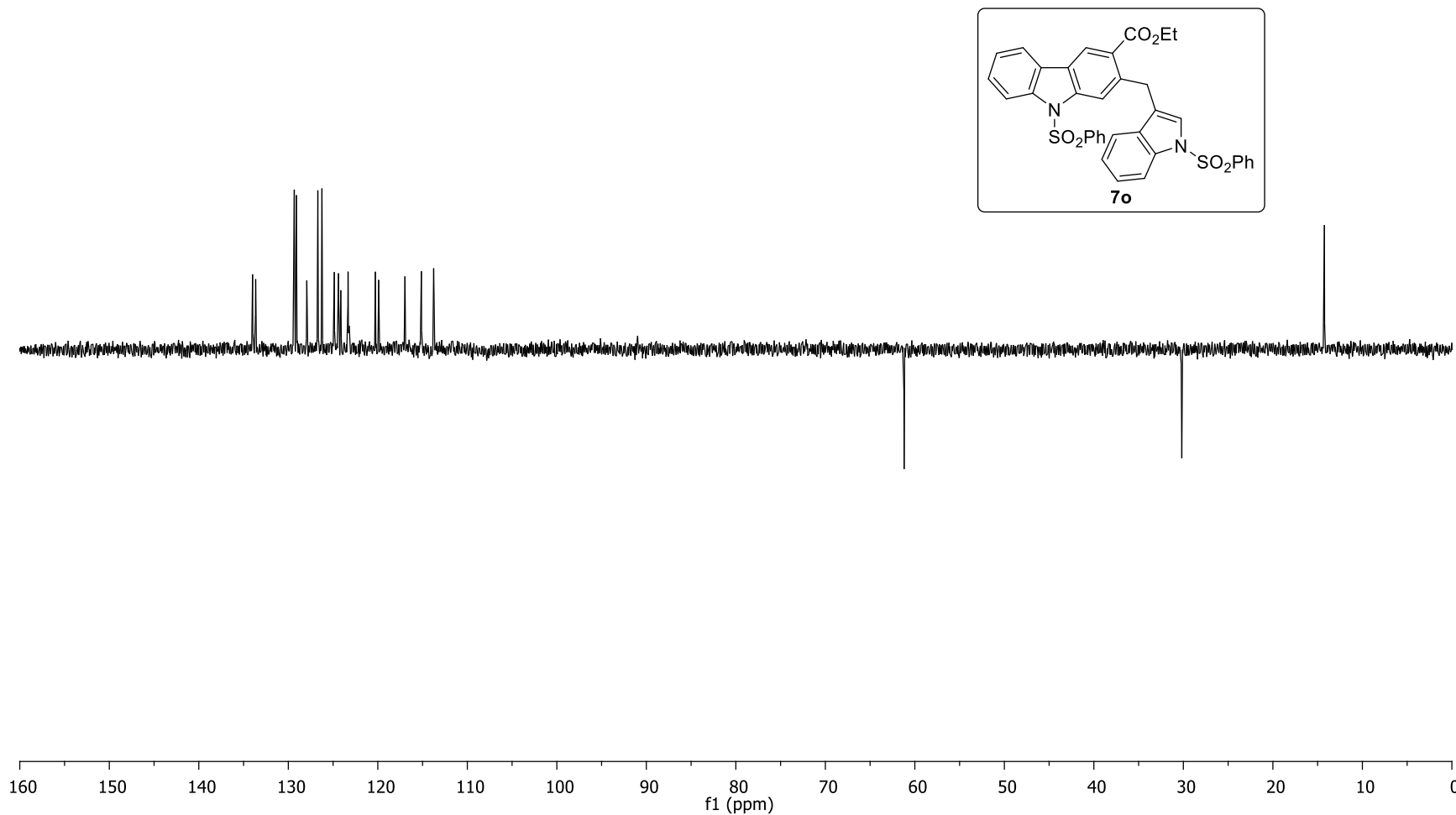
AG-IV-271

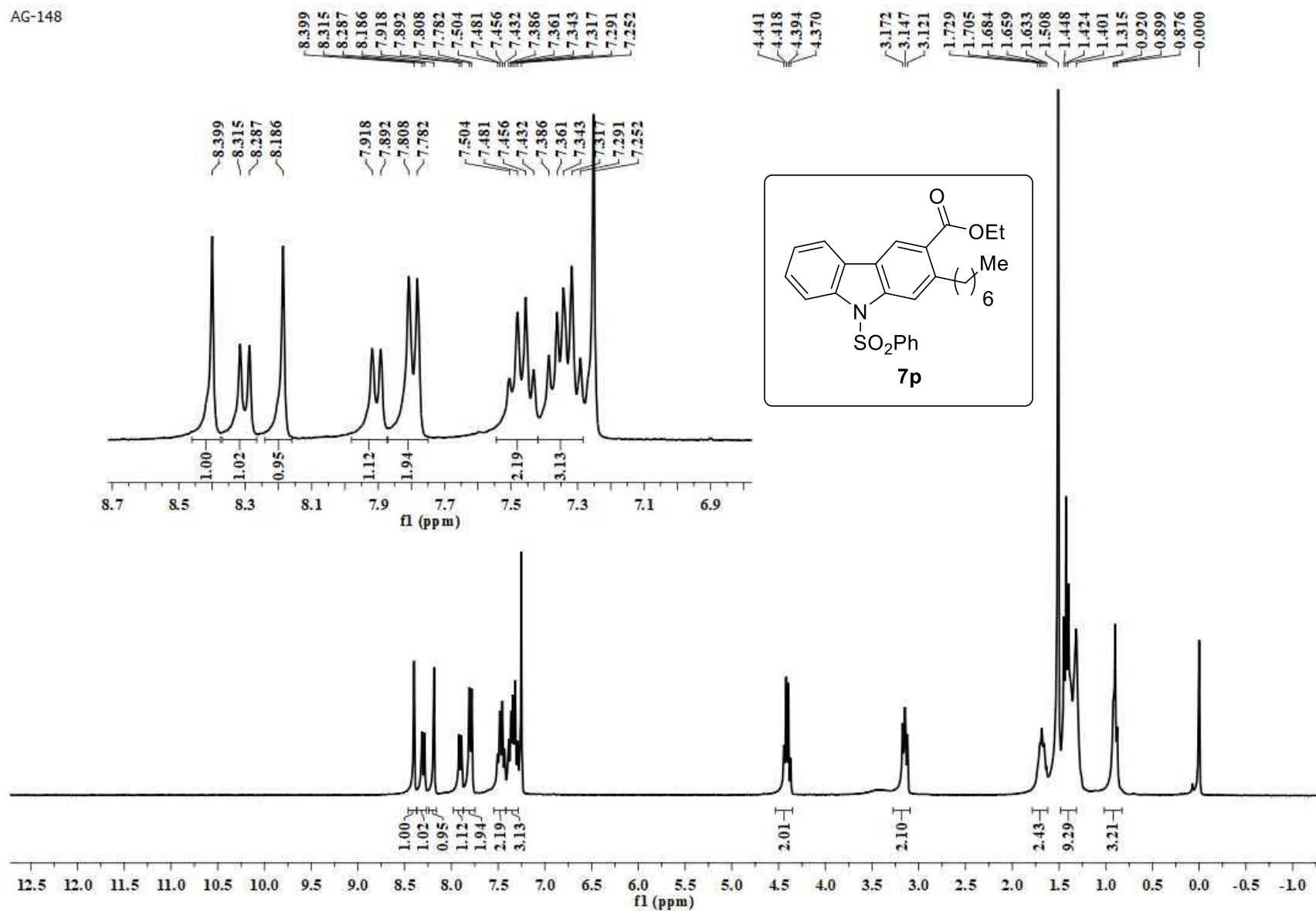
133.99
133.65
129.33
129.10
127.93
126.70
126.24
124.86
124.40
124.14
123.32
123.21
120.28
119.90
116.97
115.13
113.76

61.20

30.17

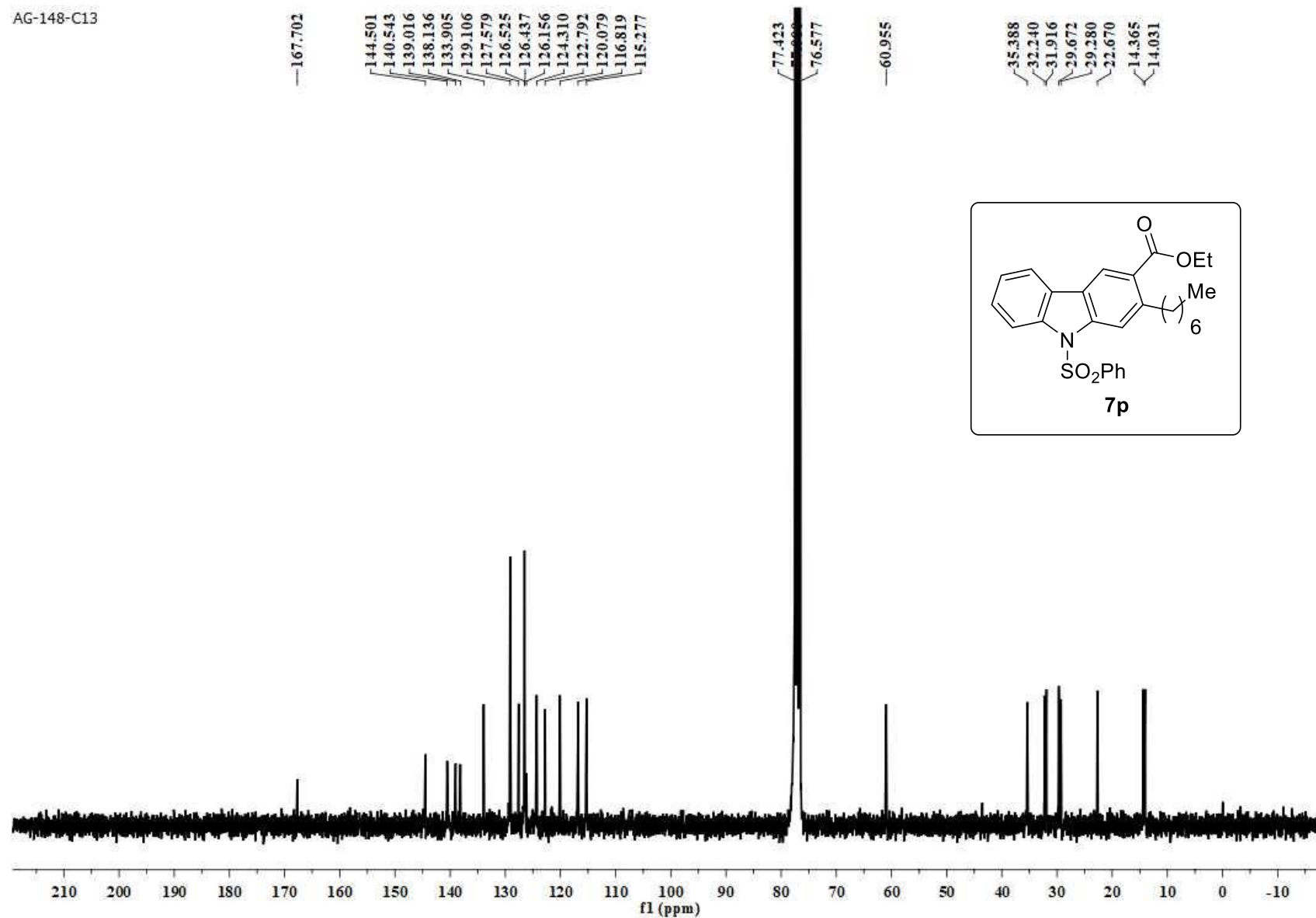
14.24





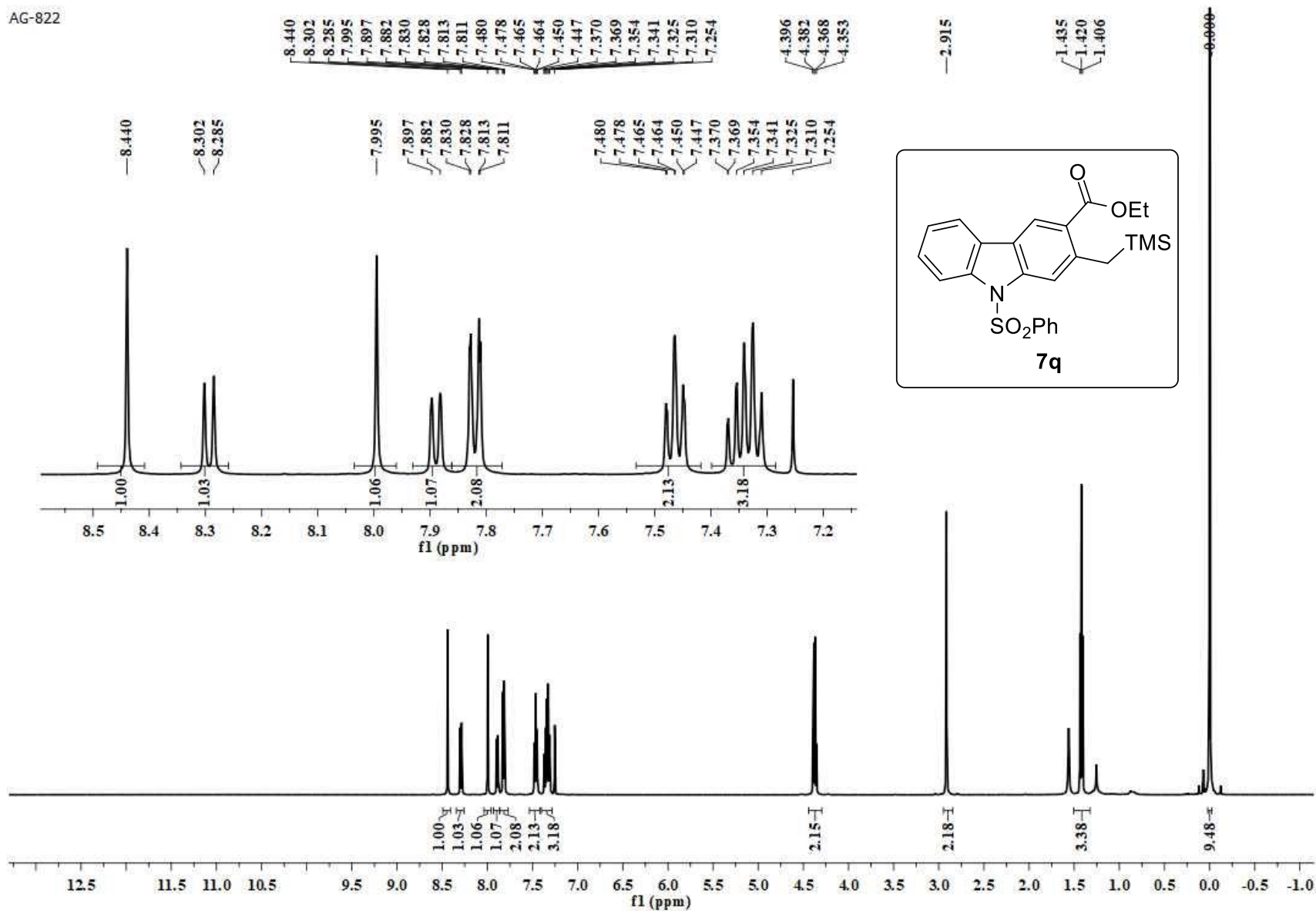
¹H NMR (300 MHz, CDCl₃) spectrum of compound 7p

AG-148-C13



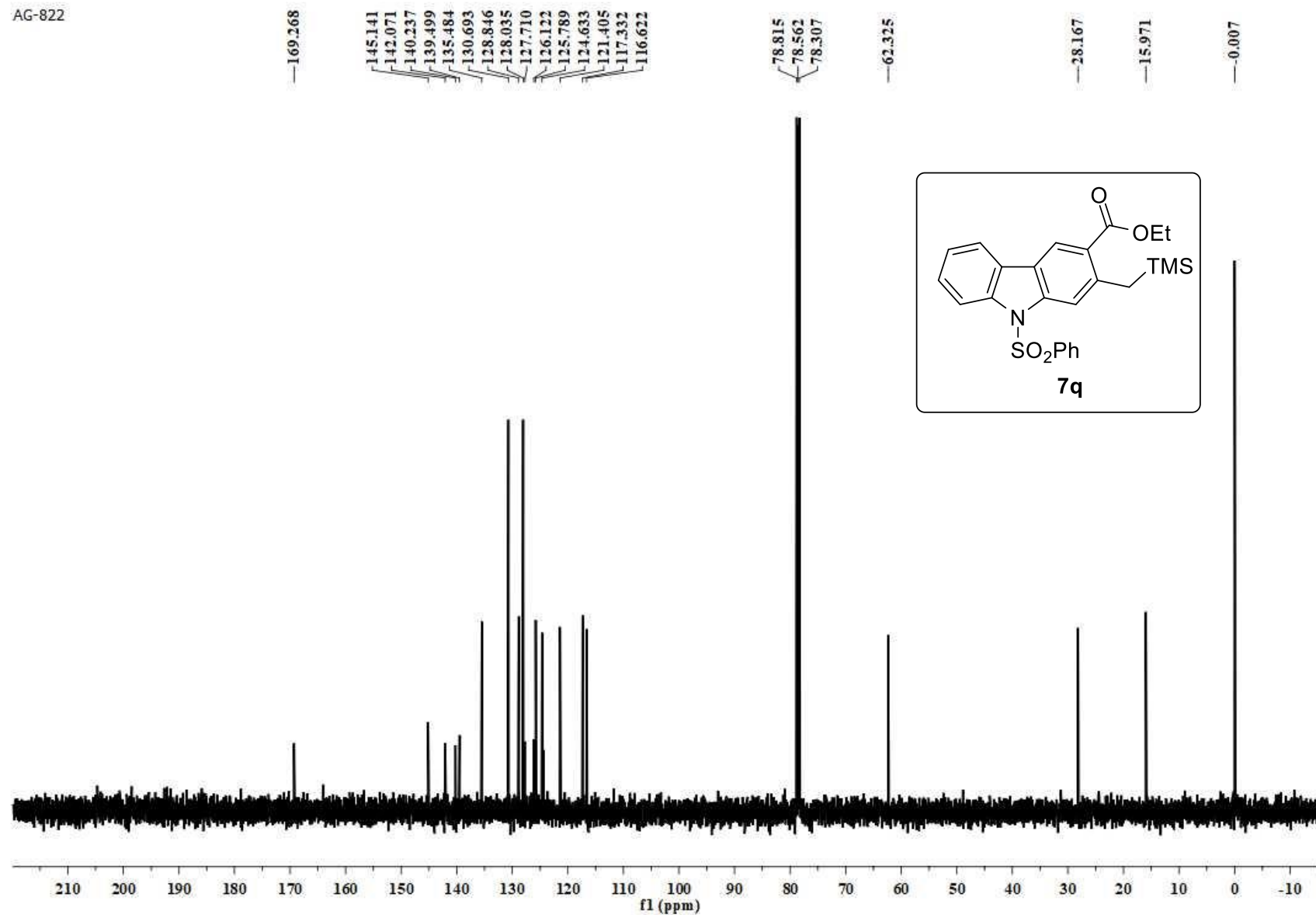
^{13}C $\{^1\text{H}\}$ NMR (75 MHz, CDCl_3) spectrum of compound **7p**

AG-822



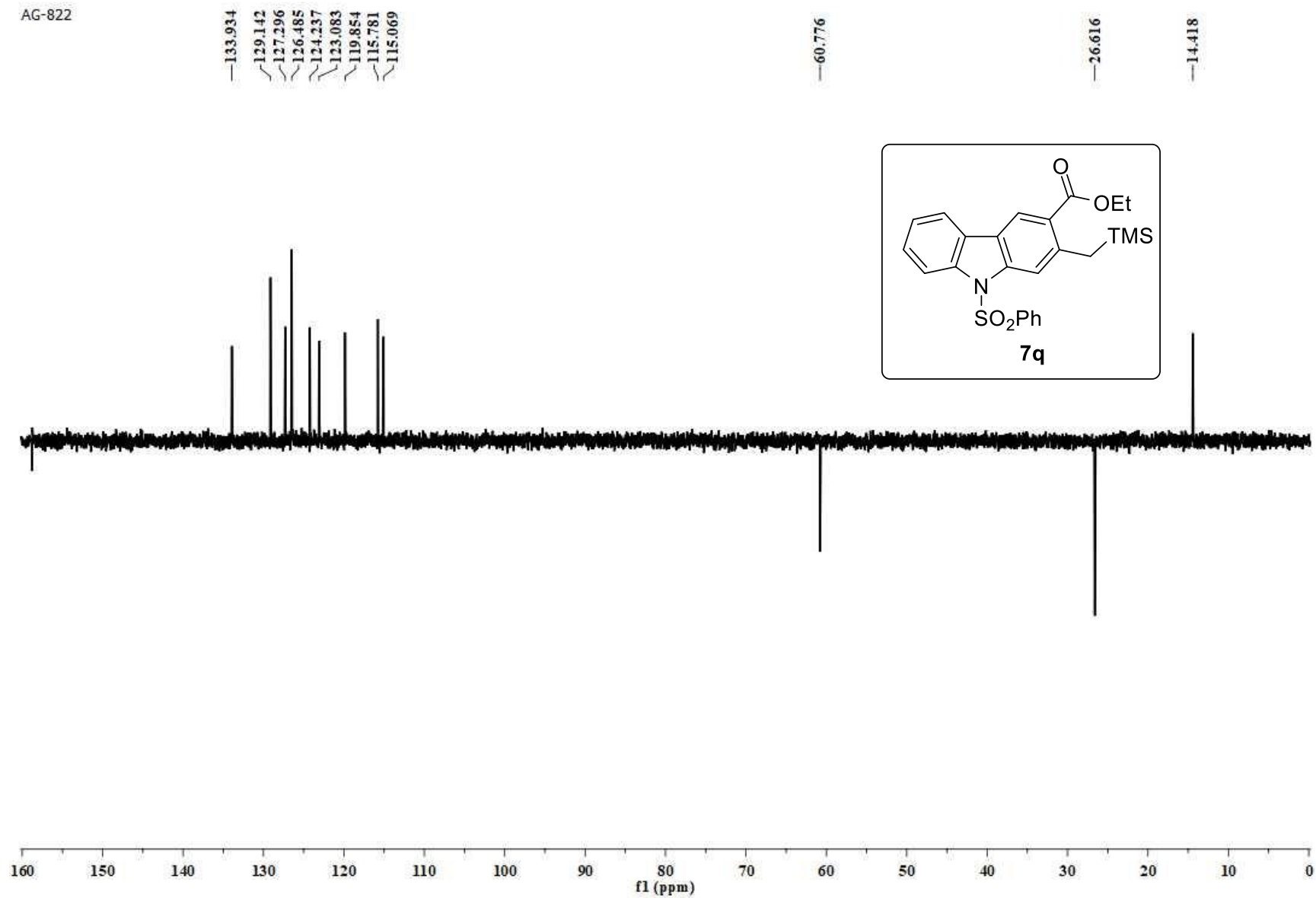
¹H NMR (500 MHz, CDCl₃) spectrum of compound 7q

AG-822



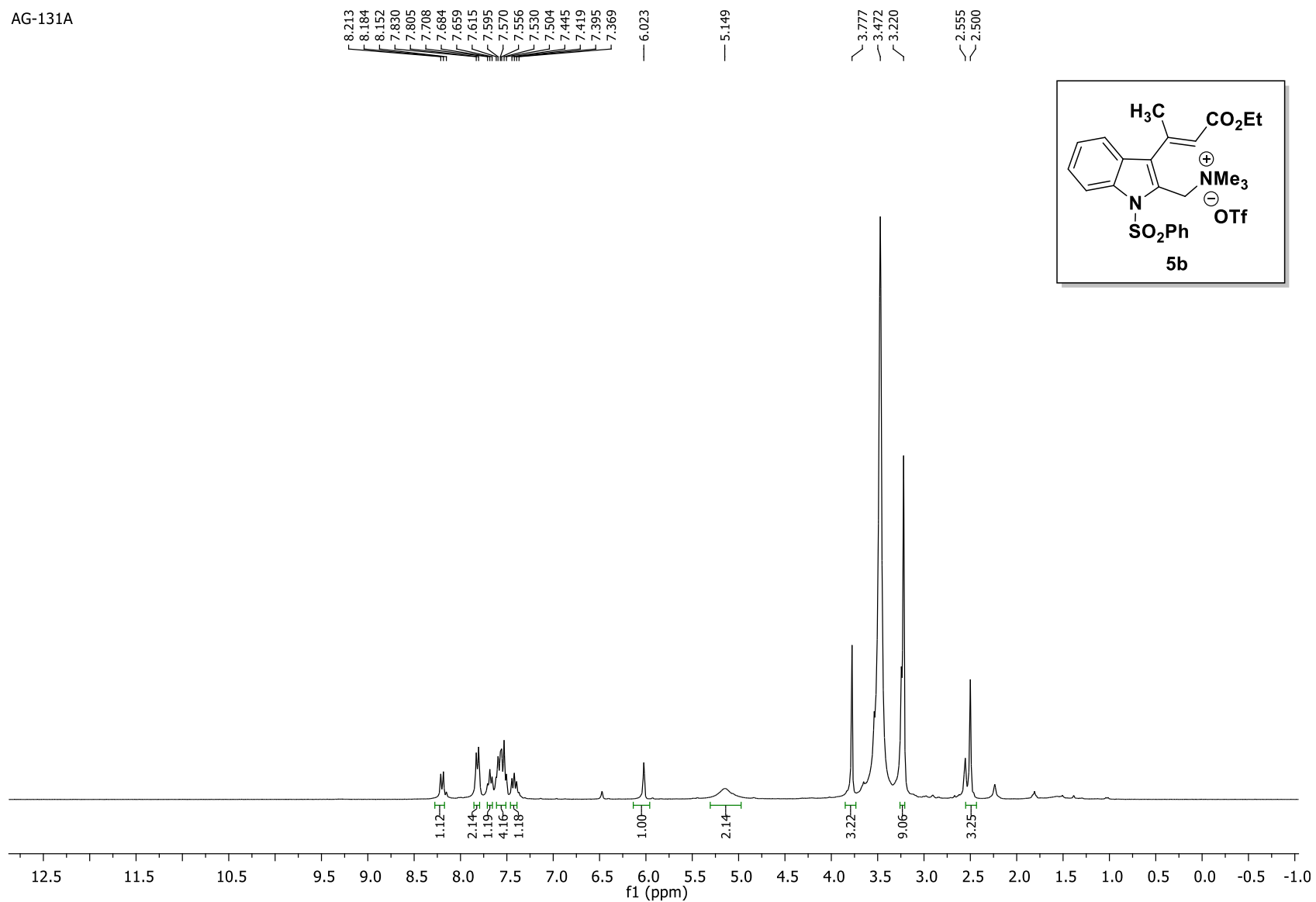
$^{13}\text{C} \{^1\text{H}\}$ NMR (125 MHz, CDCl_3) spectrum of compound **7q**

AG-822



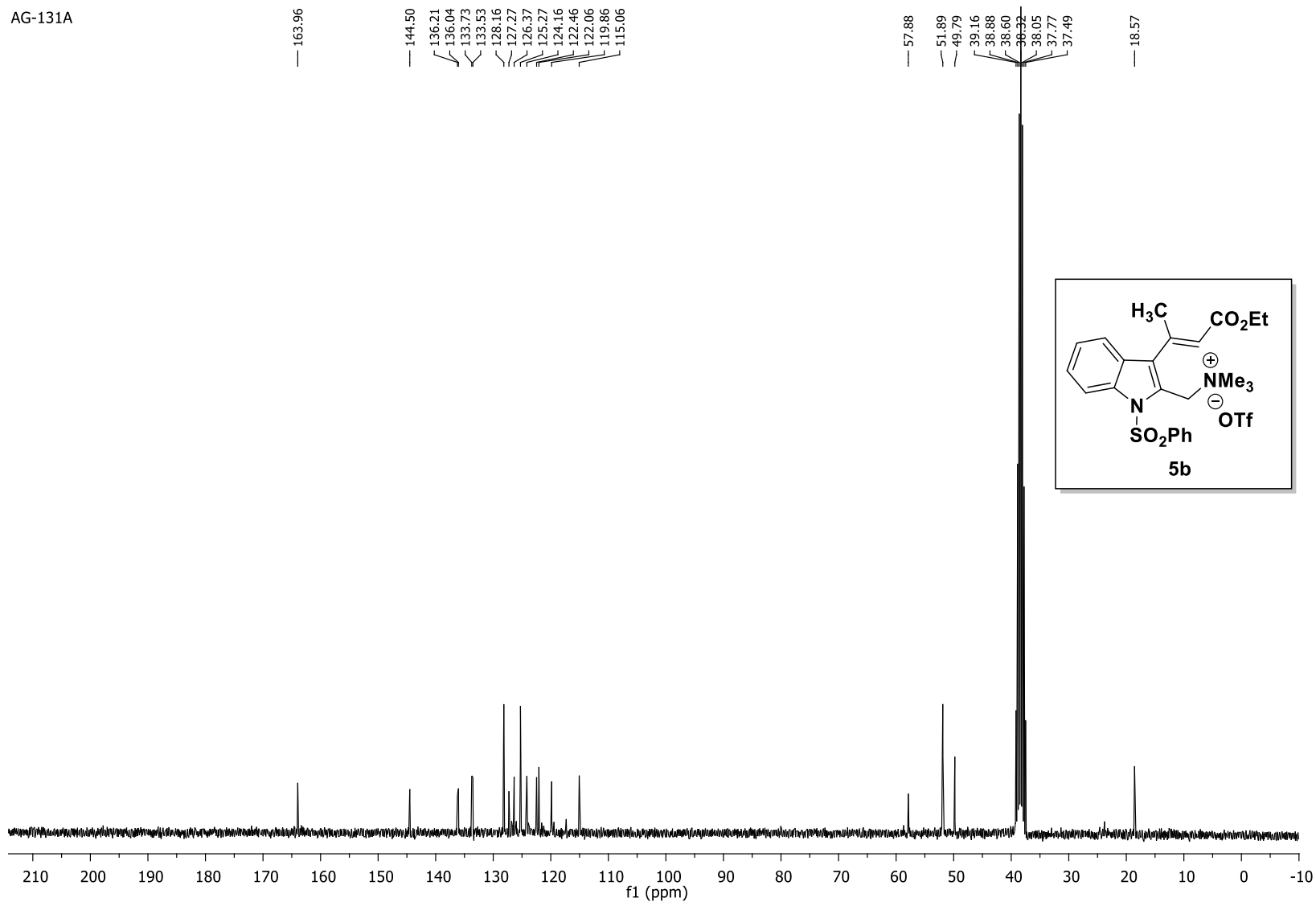
DEPT-135 NMR (125 MHz, CDCl₃) spectrum of compound **7q**

AG-131A

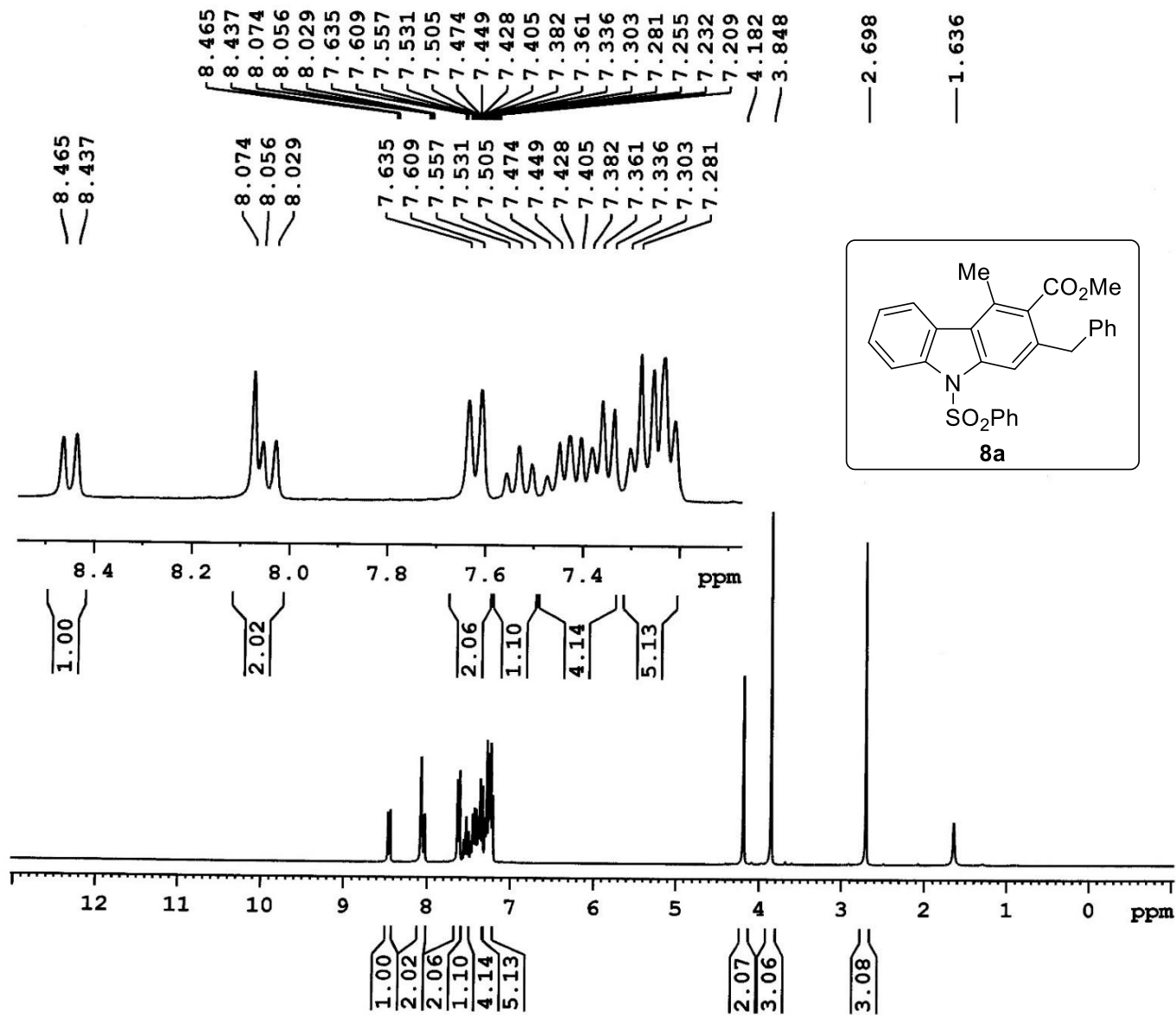


^1H NMR (300 MHz, $\text{DMSO}-d_6$) spectrum of compound **5b**

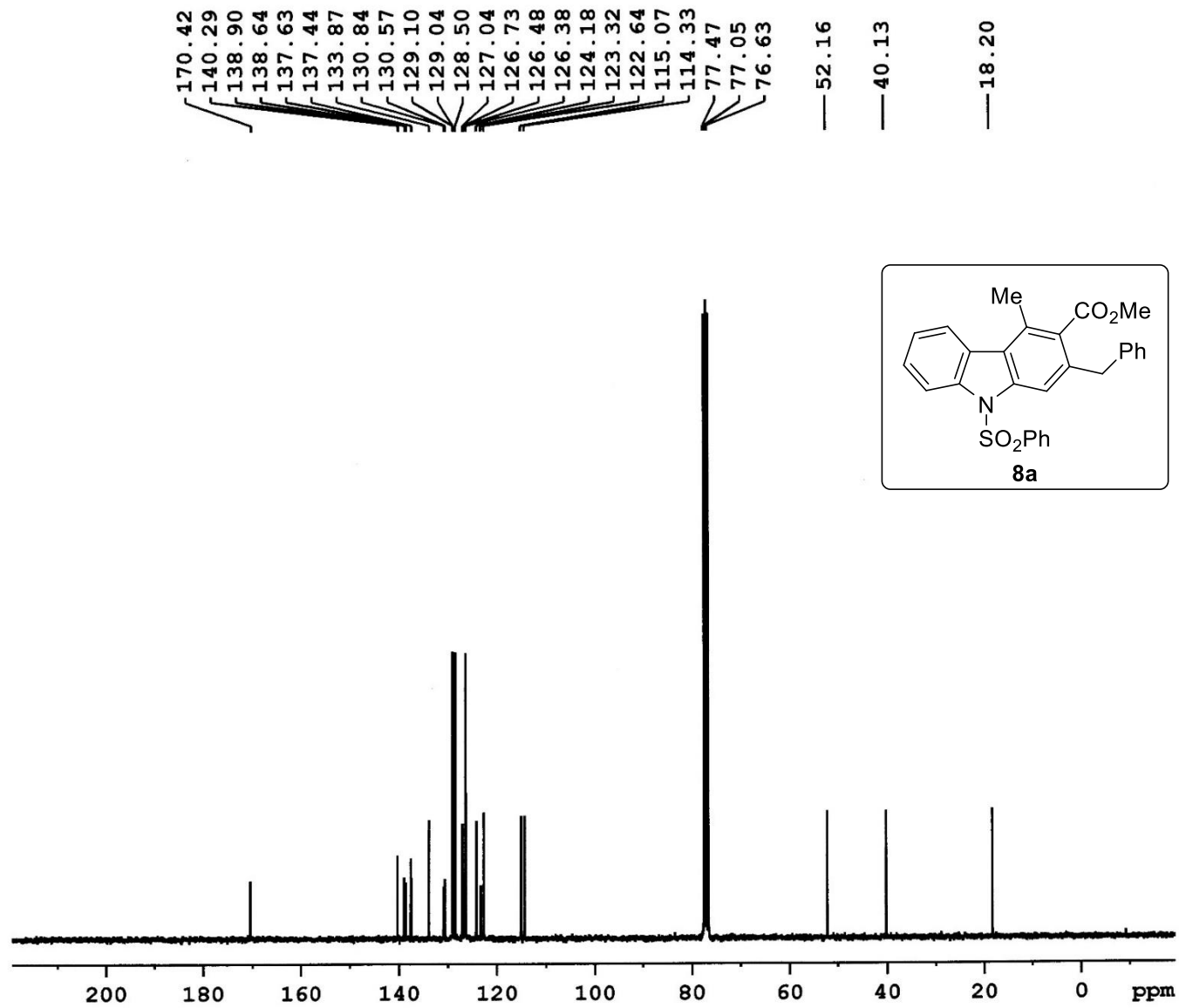
AG-131A



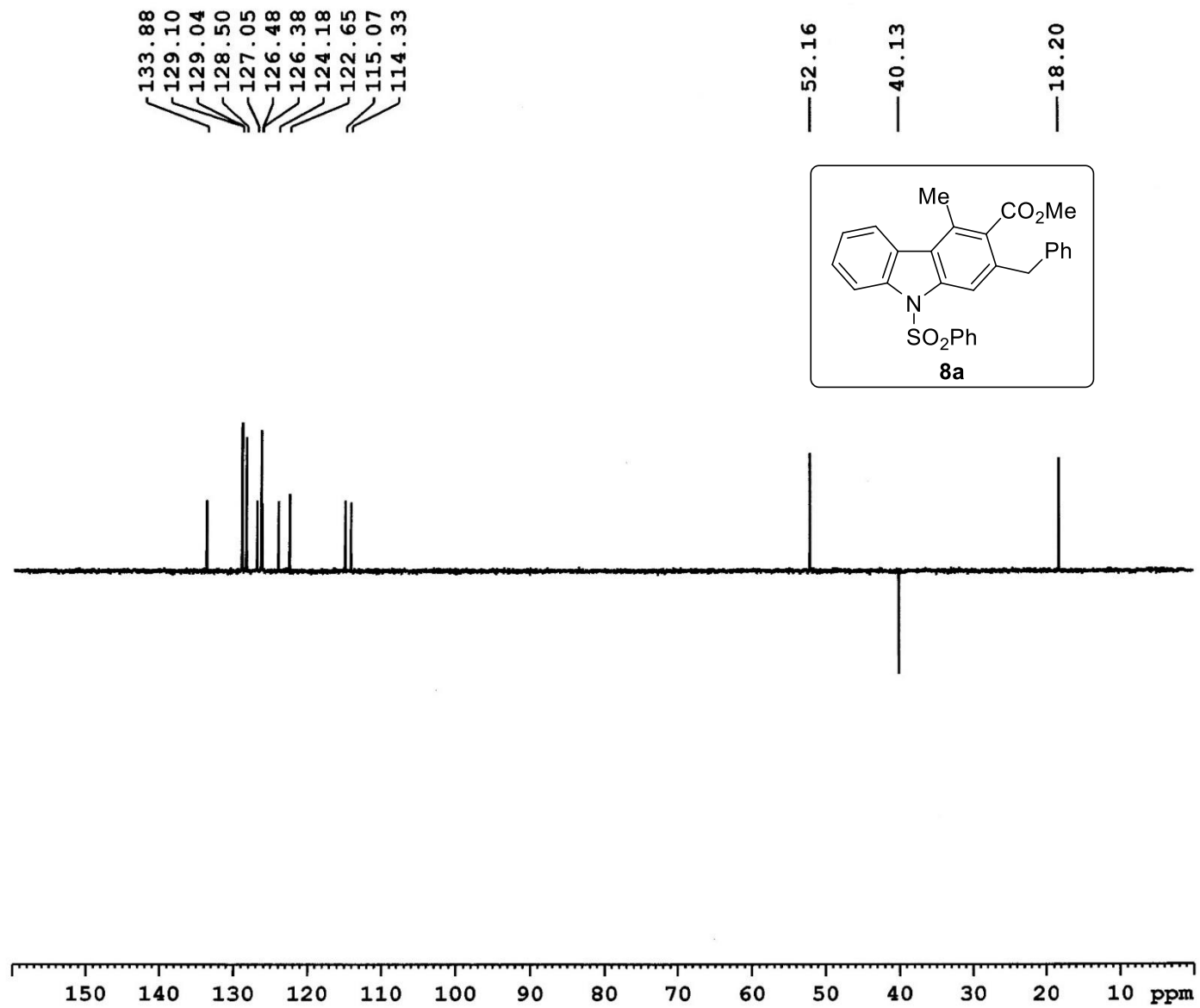
$^{13}\text{C} \{^1\text{H}\}$ NMR (75 MHz, $\text{DMSO-}d_6$) spectrum of compound **5b**



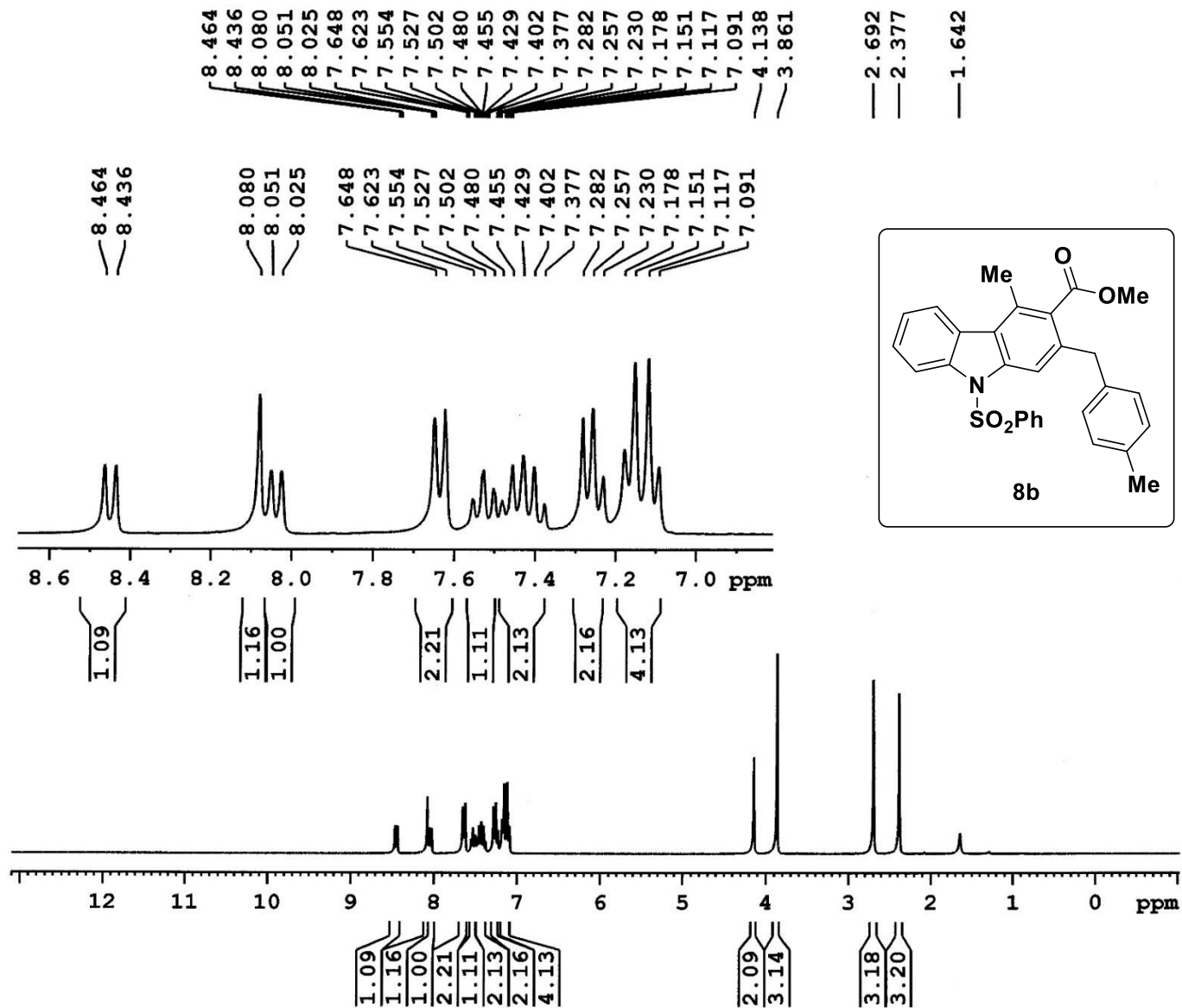
¹H NMR (300 MHz, CDCl₃) spectrum of compound **8a**



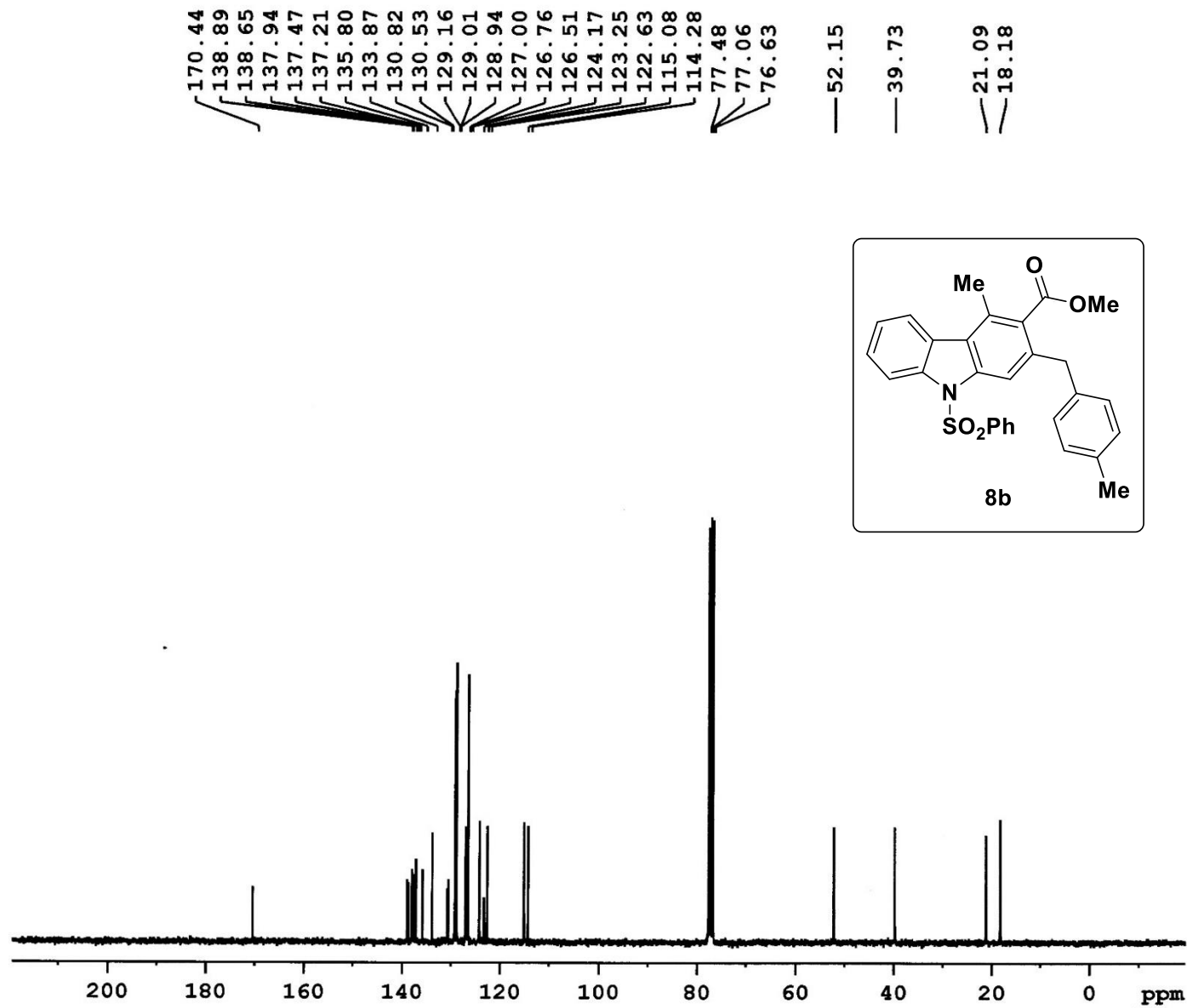
¹³C {¹H} NMR (75 MHz, CDCl₃) spectrum of compound **8a**



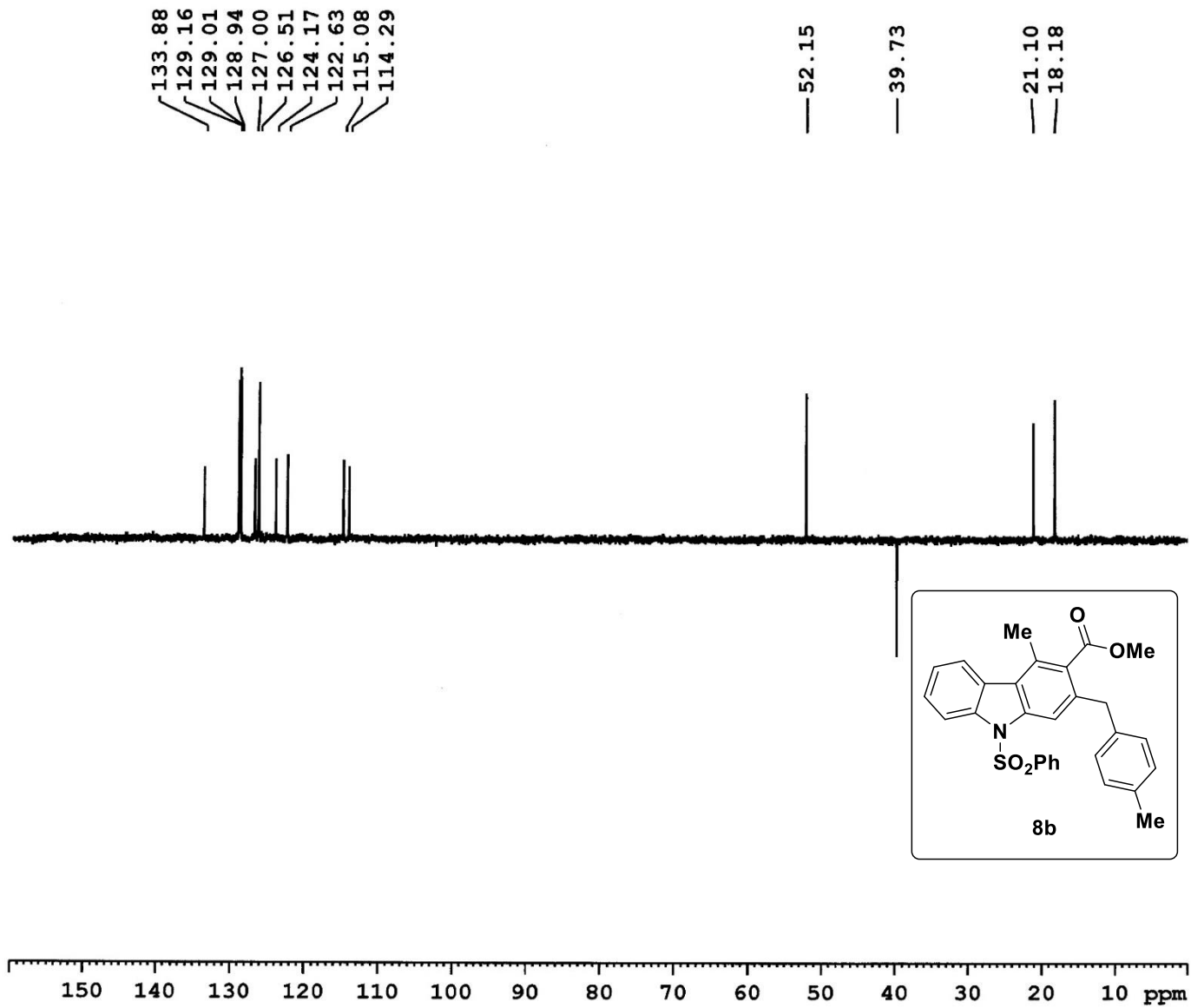
DEPT-135 NMR (75 MHz, CDCl₃) spectrum of compound **8a**



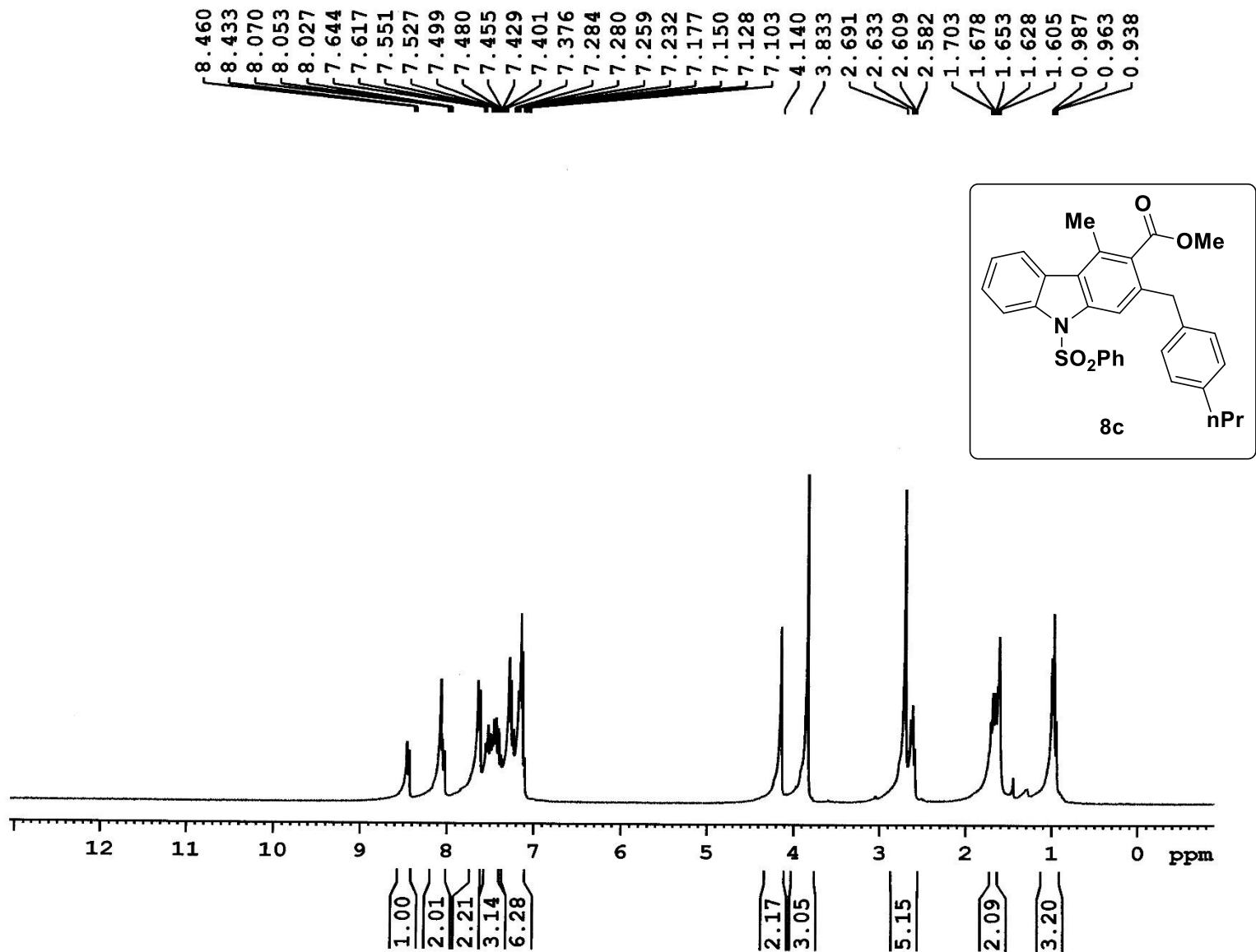
¹H NMR (300 MHz, CDCl₃) spectrum of compound **8b**



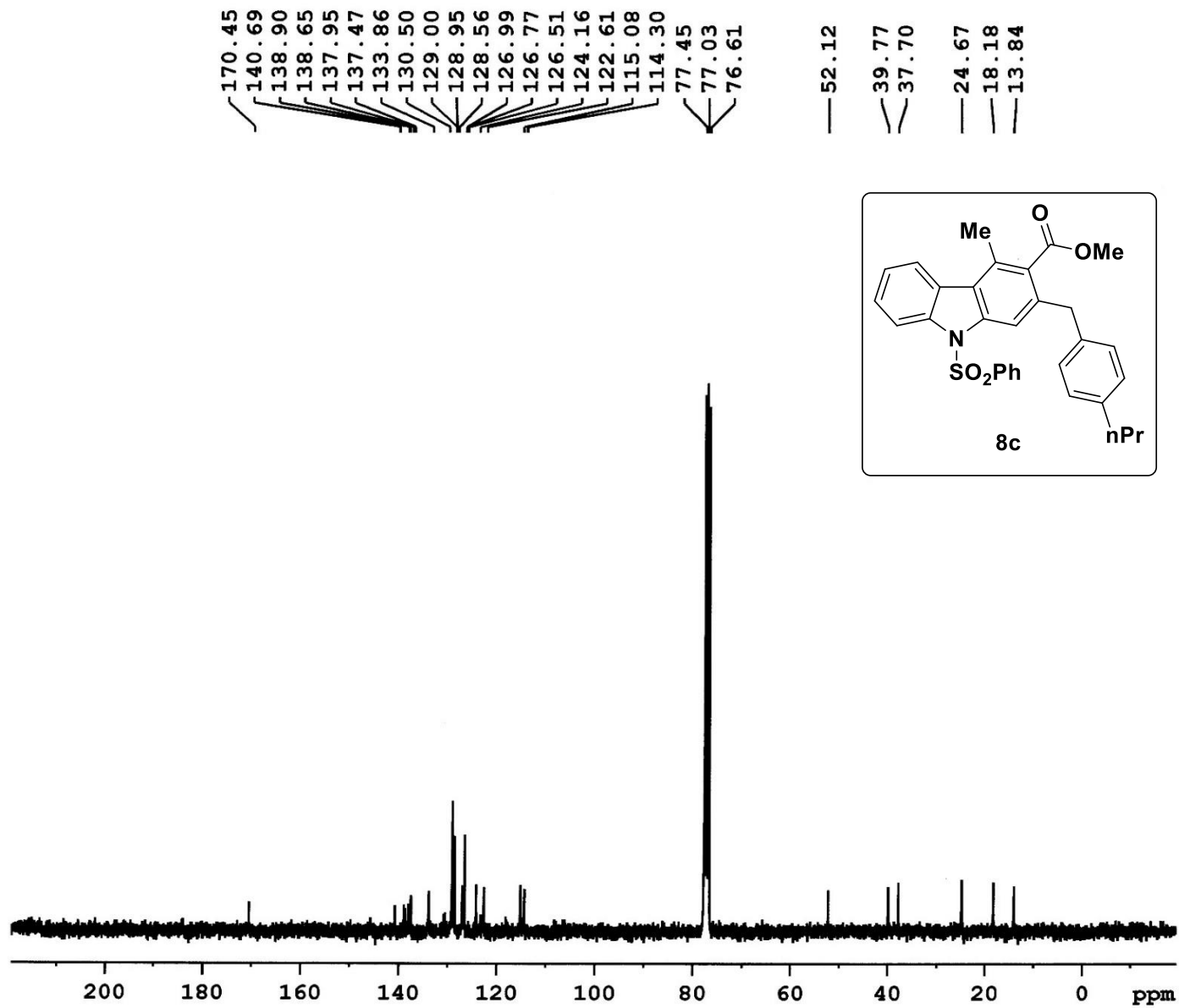
^{13}C $\{^1\text{H}\}$ NMR (75 MHz, CDCl_3) spectrum of compound **8b**



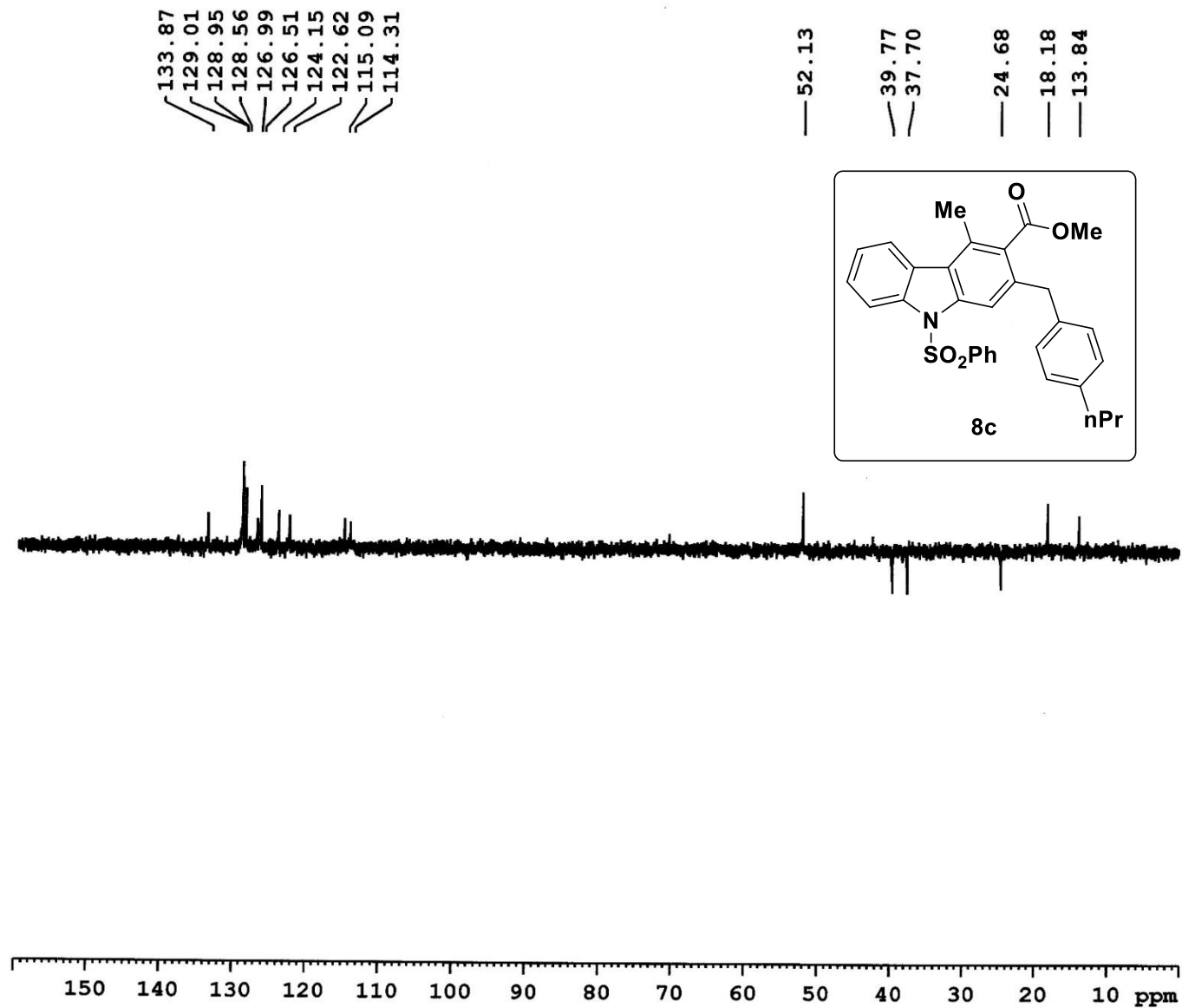
DEPT-135 NMR (75 MHz, CDCl₃) spectrum of compound **8b**



¹H NMR (300 MHz, CDCl₃) spectrum of compound **8c**



$^{13}\text{C} \{^1\text{H}\}$ NMR (75 MHz, CDCl_3) spectrum of compound **8c**

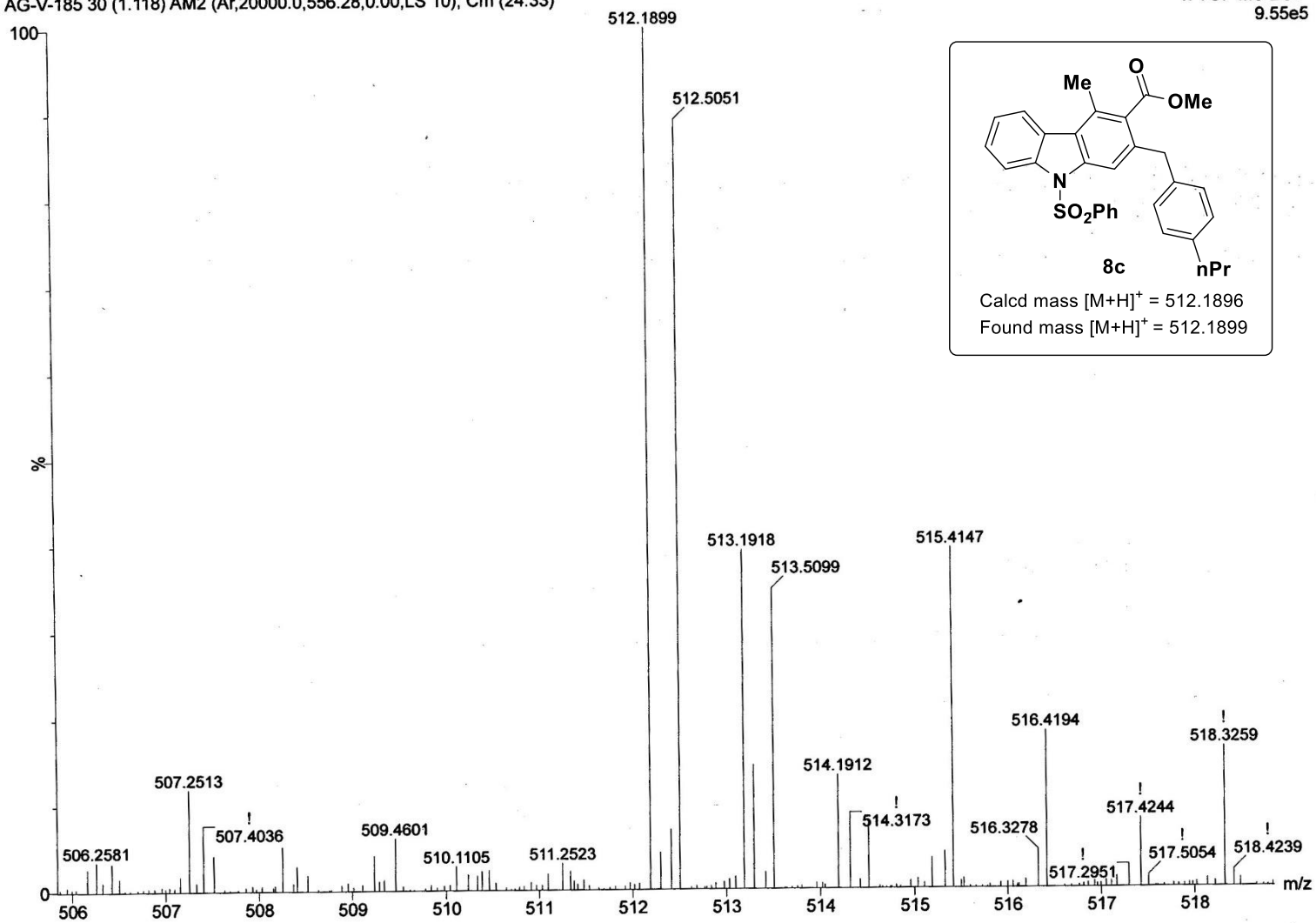


DEPT-135 NMR (75 MHz, CDCl₃) spectrum of compound **8c**

DRAKM

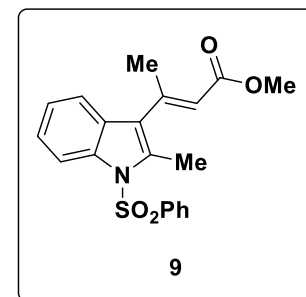
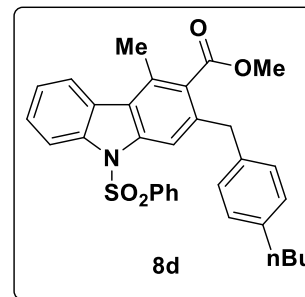
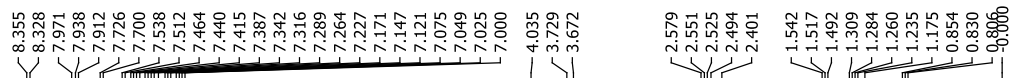
AG-V-185 30 (1.118) AM2 (Ar,20000.0,556.28,0.00,LS 10); Cm (24:33)

1: TOF MS ES+
9.55e5

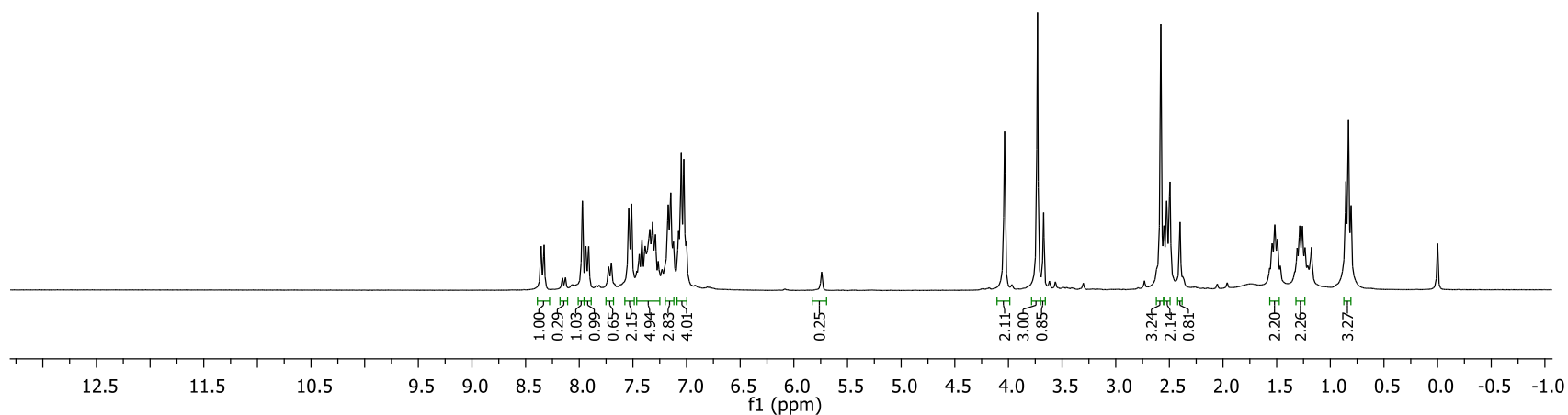


HRMS Spectrum of compound **8c**

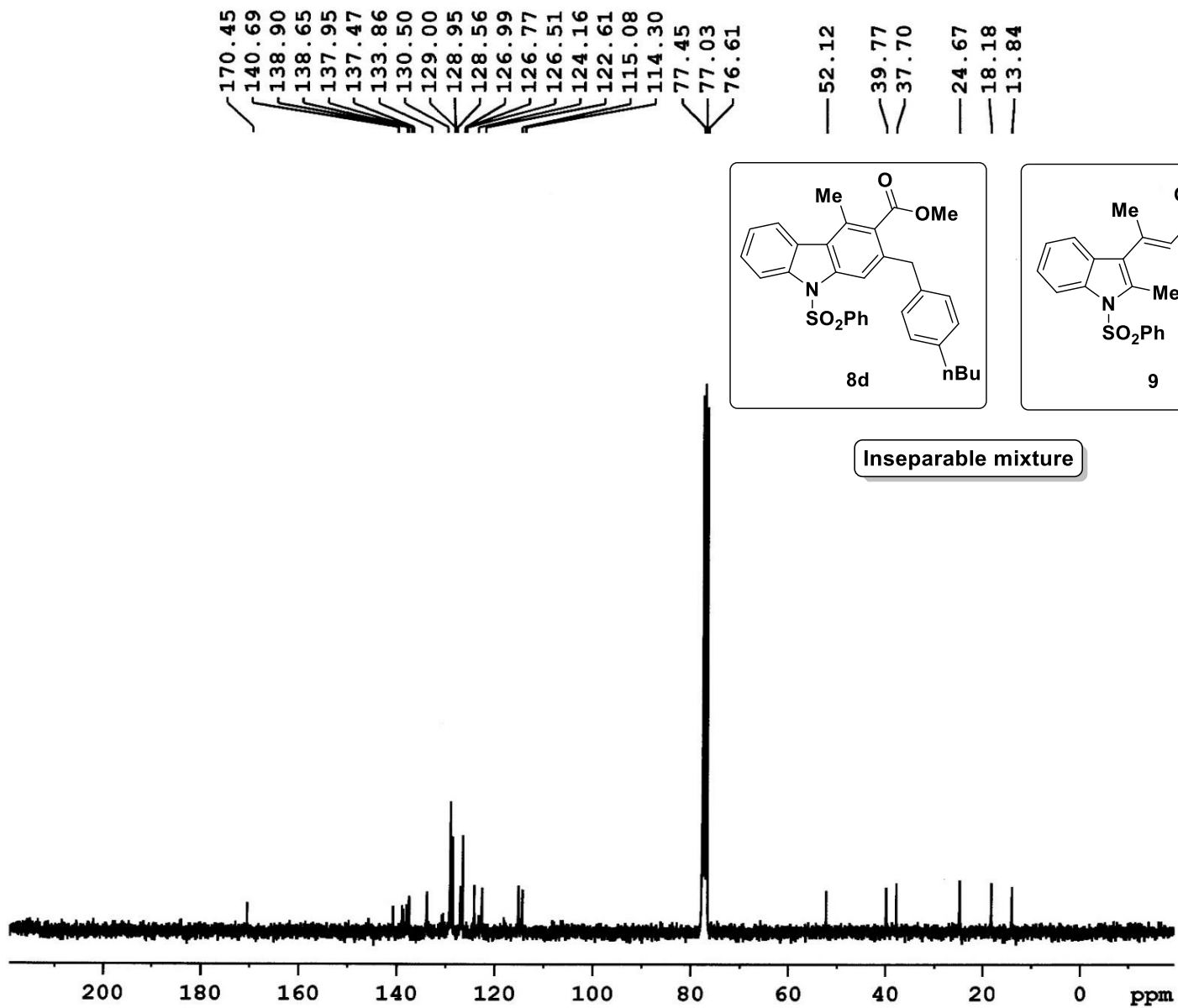
AG-V-187



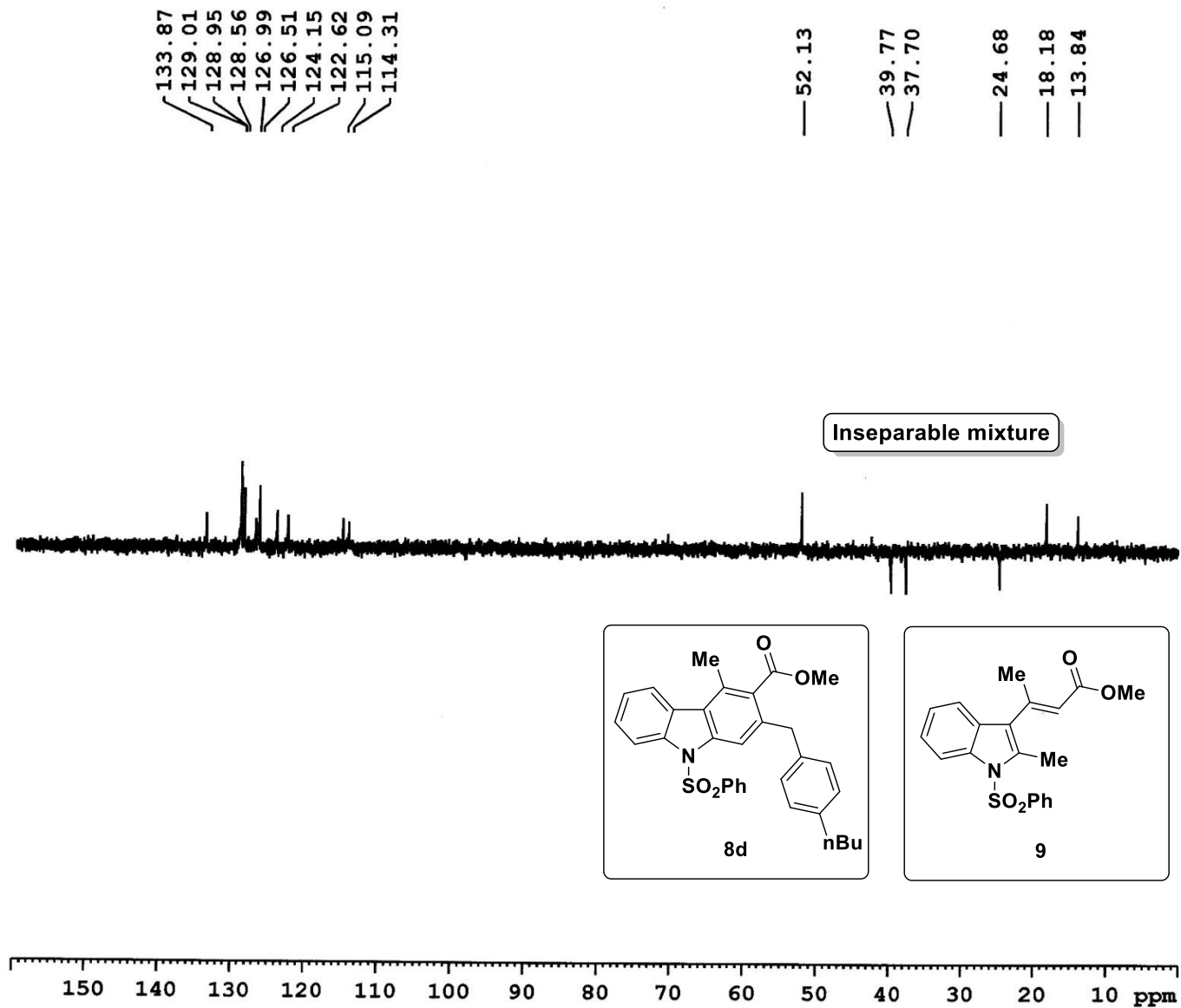
Inseparable mixture



¹H NMR (CDCl₃, 300 MHz) spectrum of compounds **8d** & **9**



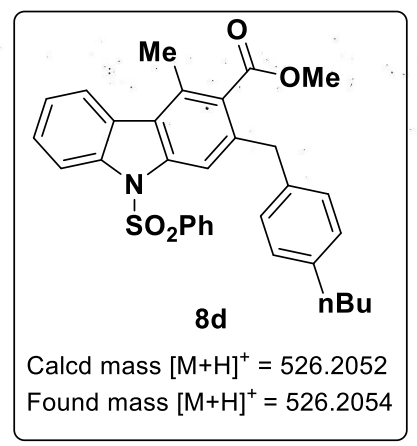
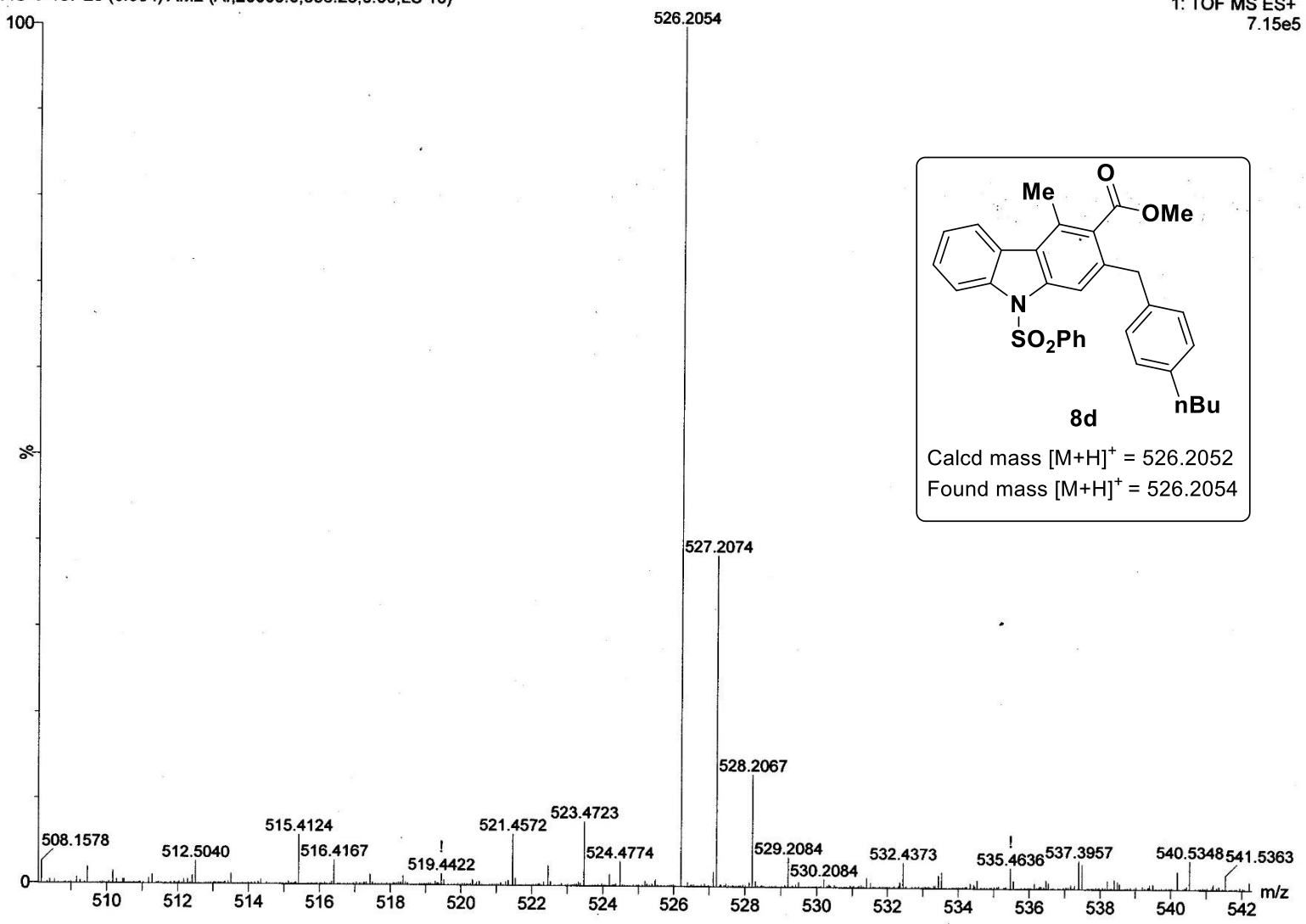
$^{13}\text{C} \{^1\text{H}\}$ NMR (75 MHz, CDCl_3) spectrum of compounds **8d** & **9**



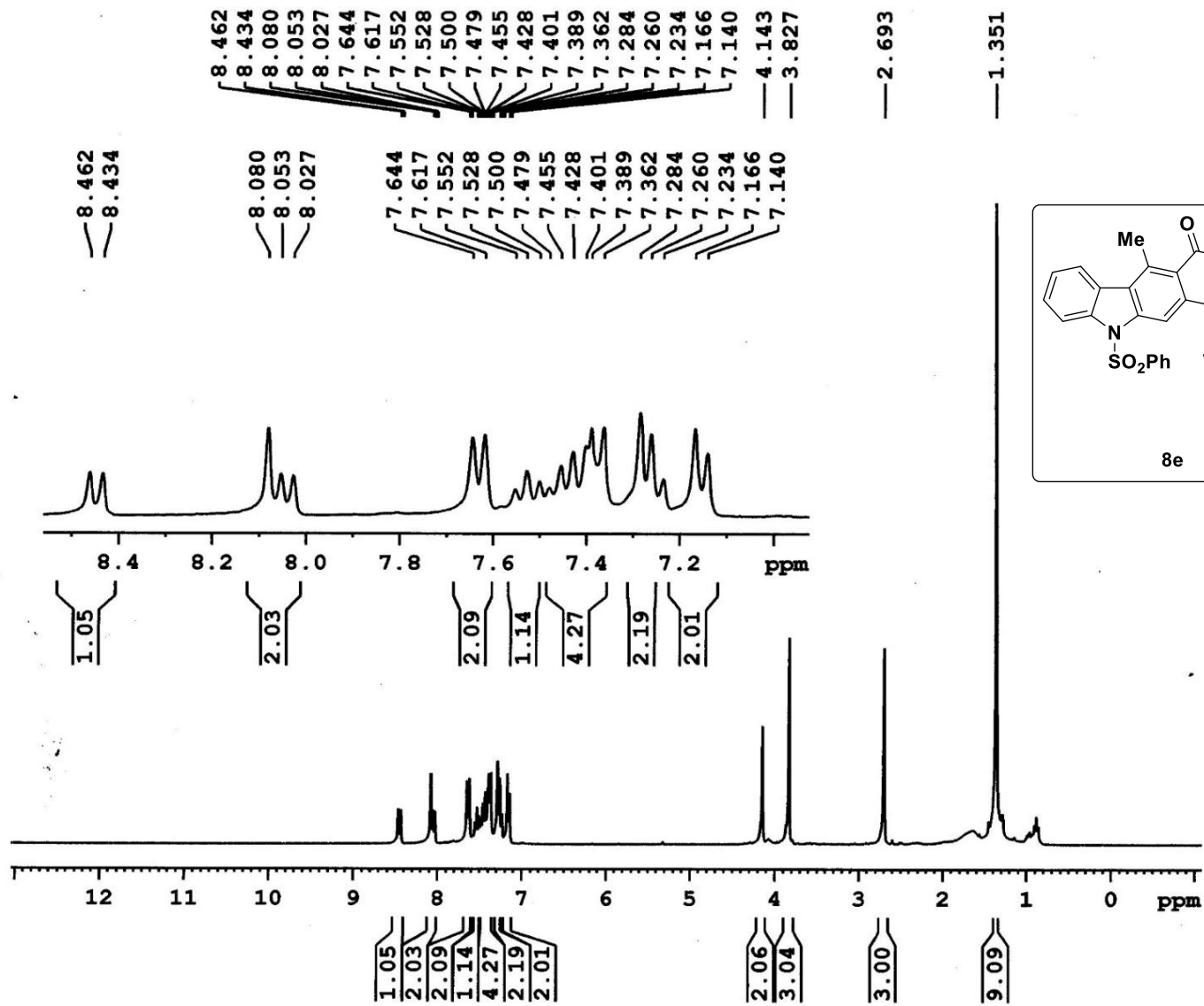
DEPT-135 NMR (75 MHz, CDCl₃) spectrum of compounds **8d** & **9**

DRAKM
AG-V-187 25 (0.934) AM2 (Ar,20000.0,556.28,0.00,LS 10)

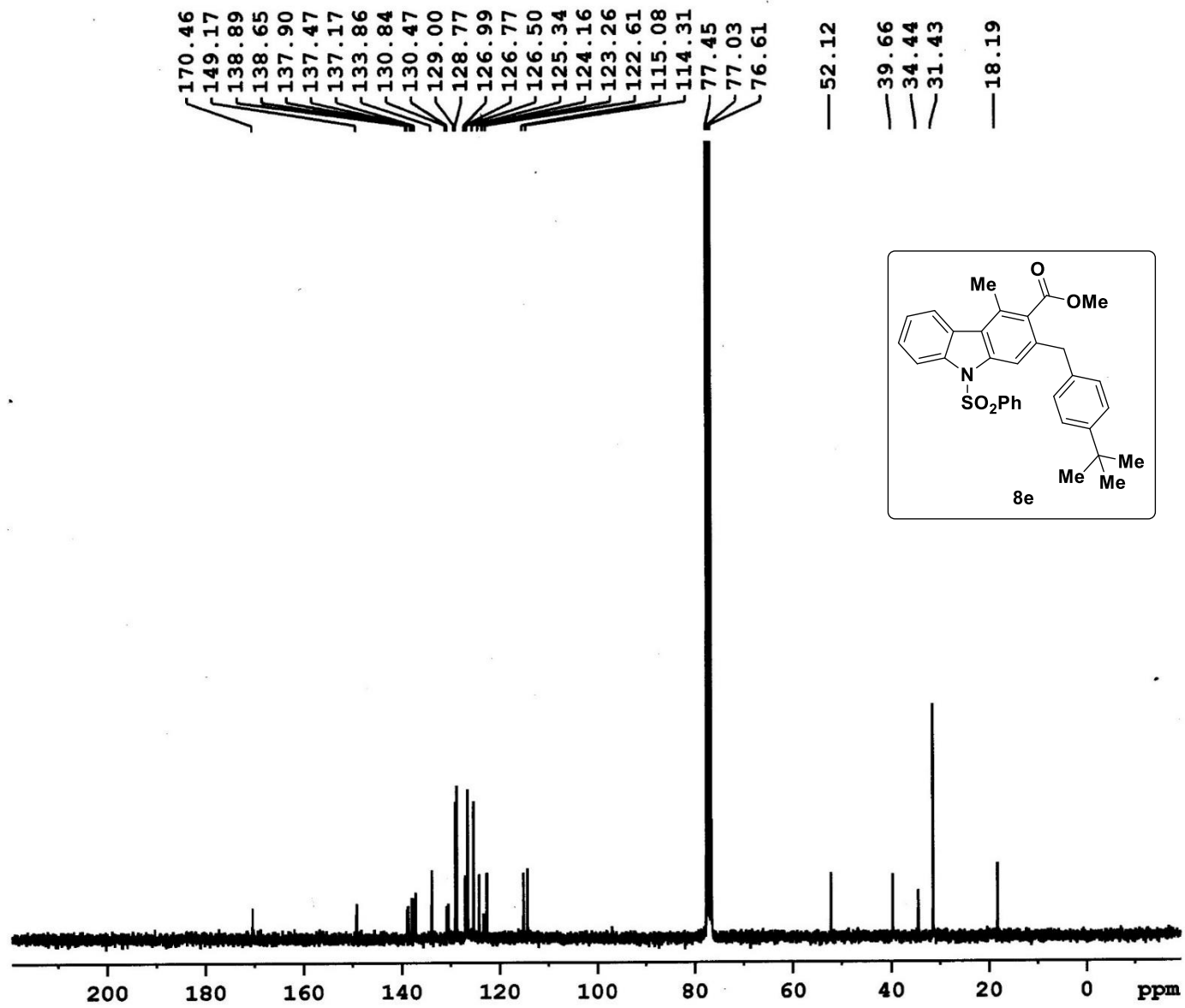
1: TOF MS ES+
7.15e5



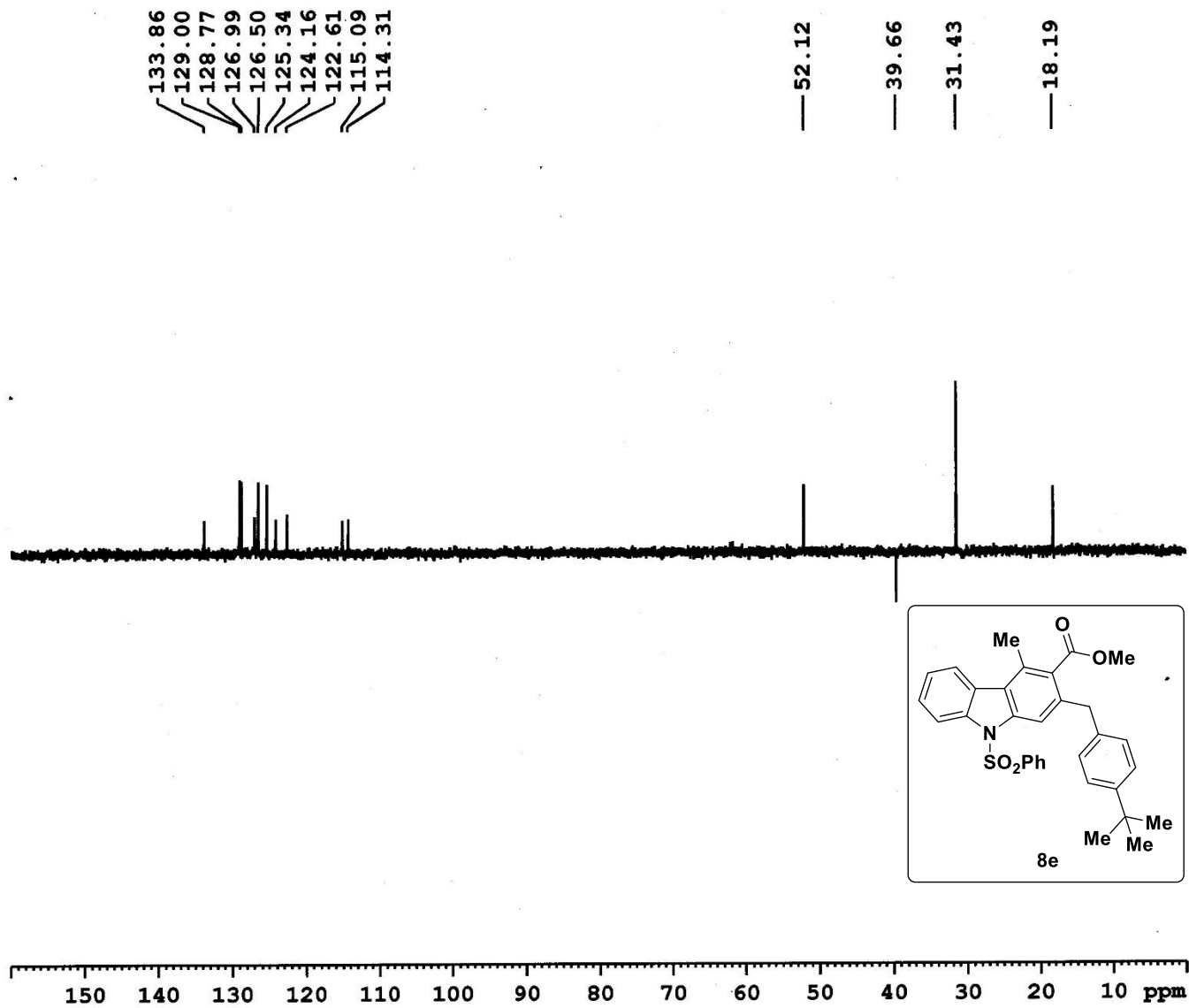
HRMS Spectrum of compound **8d**



¹H NMR (300 MHz, CDCl₃) spectrum of compound 8e



¹³C {¹H} NMR (75 MHz, CDCl₃) spectrum of compound **8e**

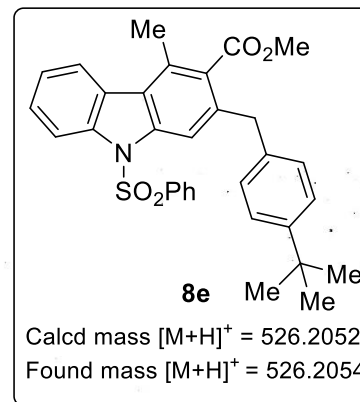
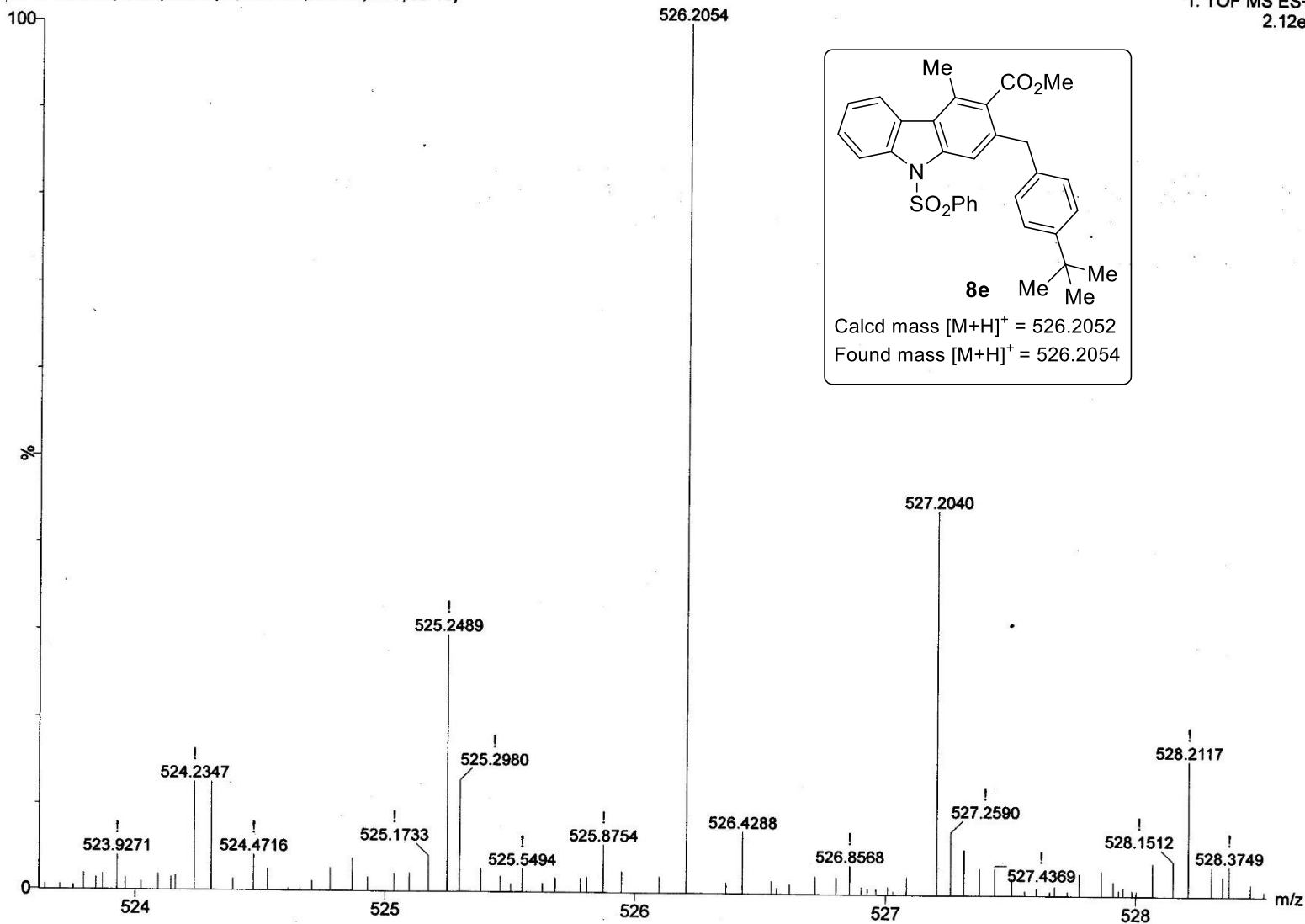


DEPT-135 NMR (75 MHz, CDCl₃) spectrum of compound **8e**

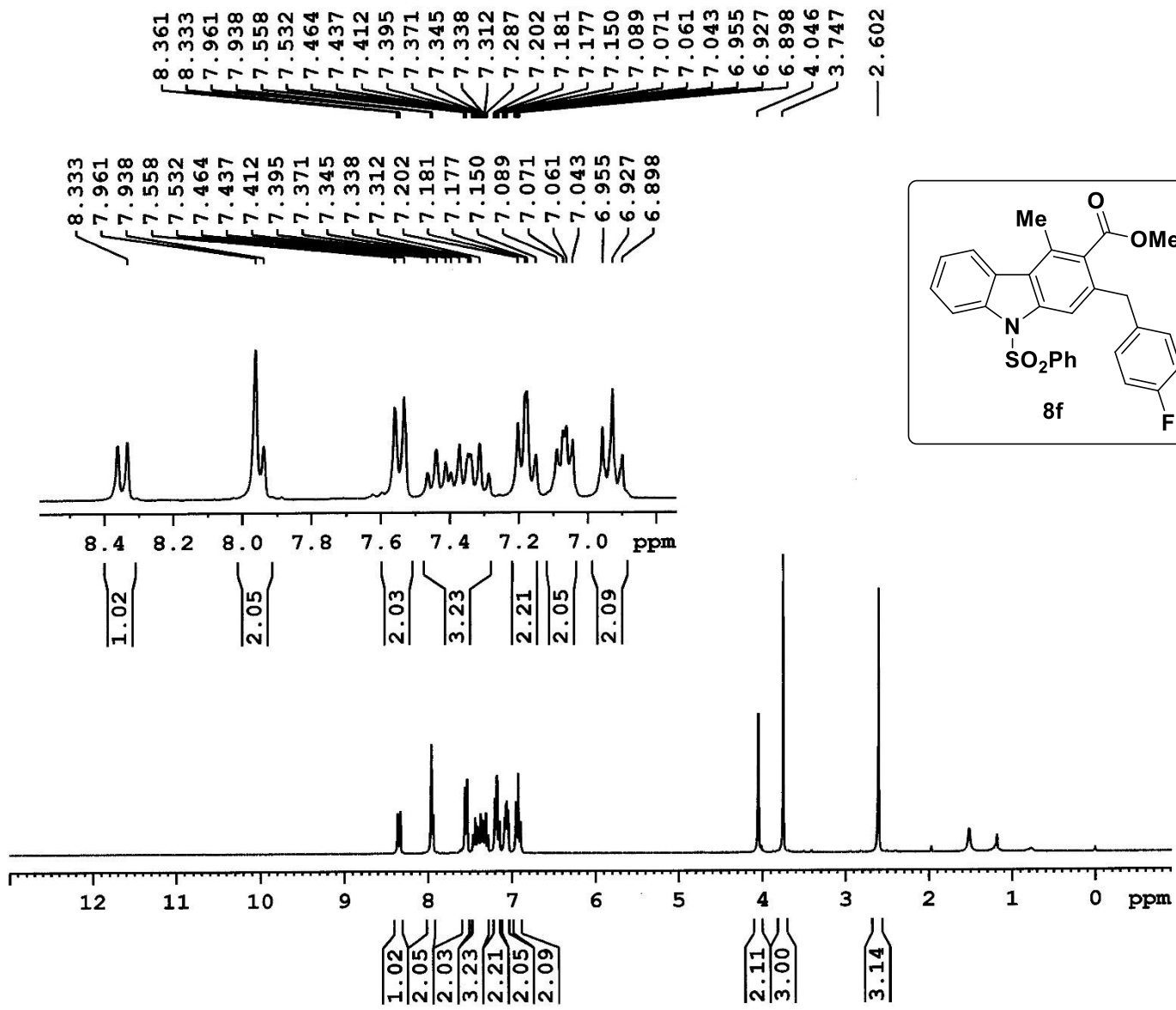
DRAKM

AG-V-184 24 (0.898) AM2 (Ar,20000.0,556.28,0.00,LS 10)

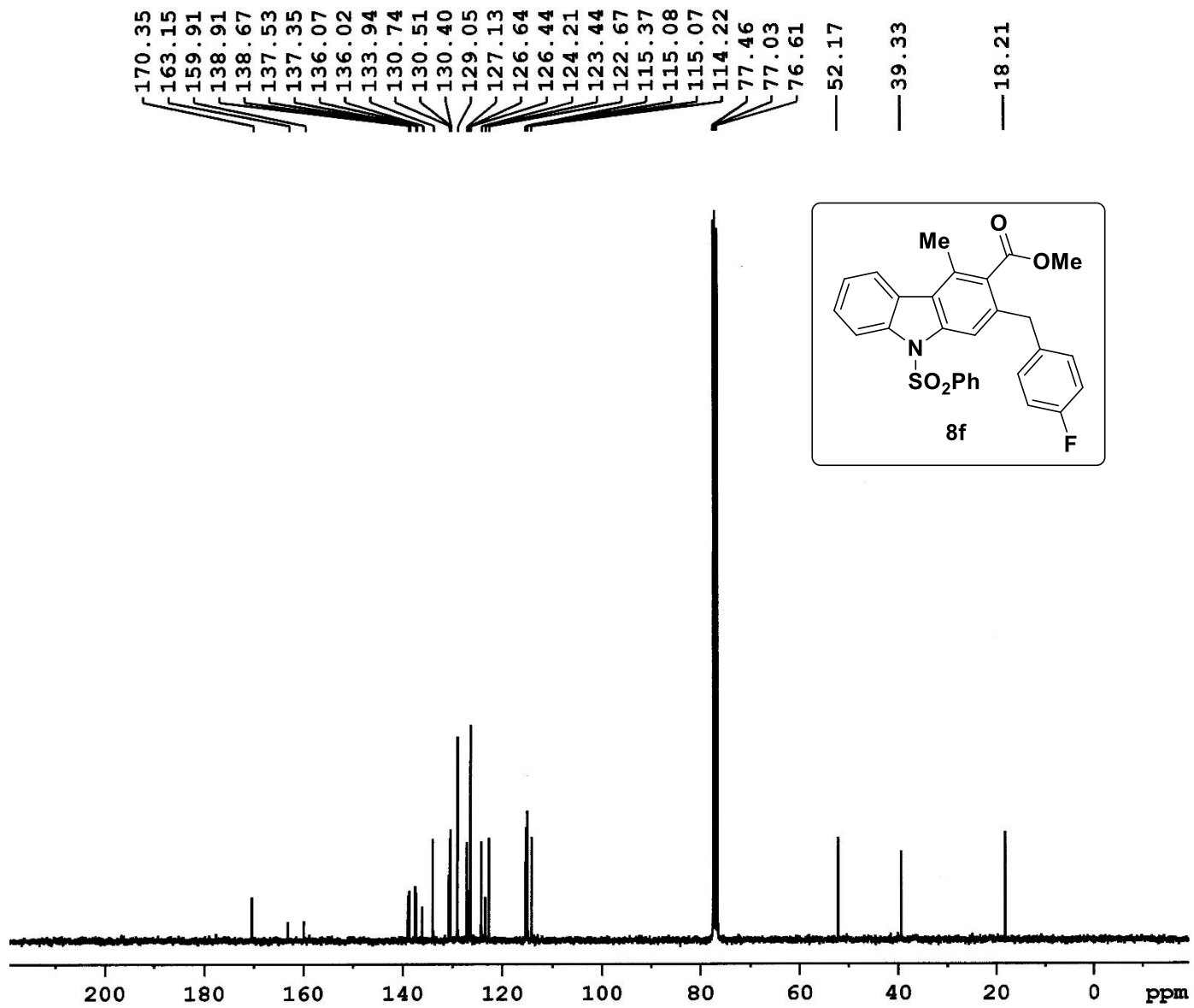
1: TOF MS ES+
2.12e4



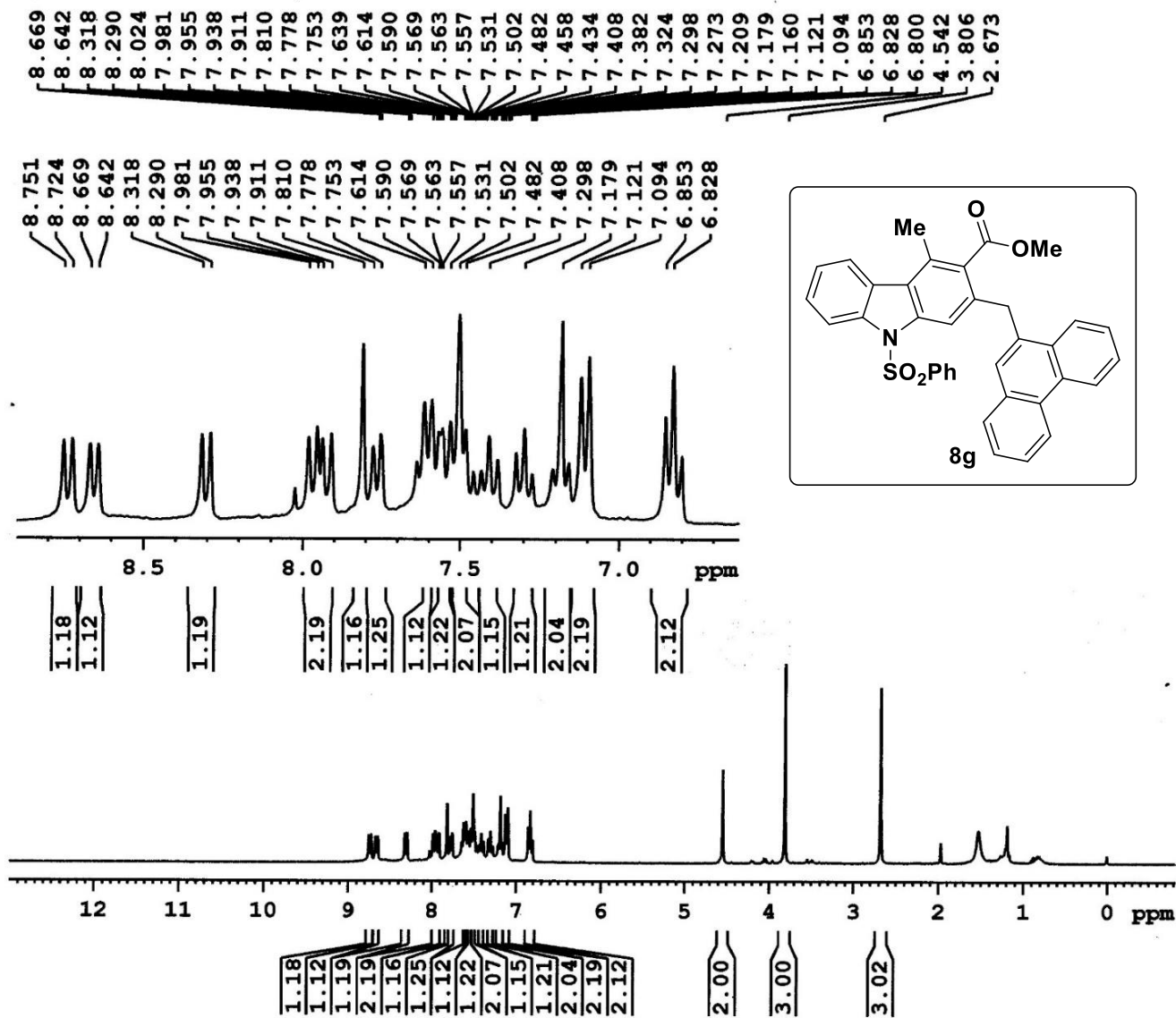
HRMS Spectrum of compound **8e**



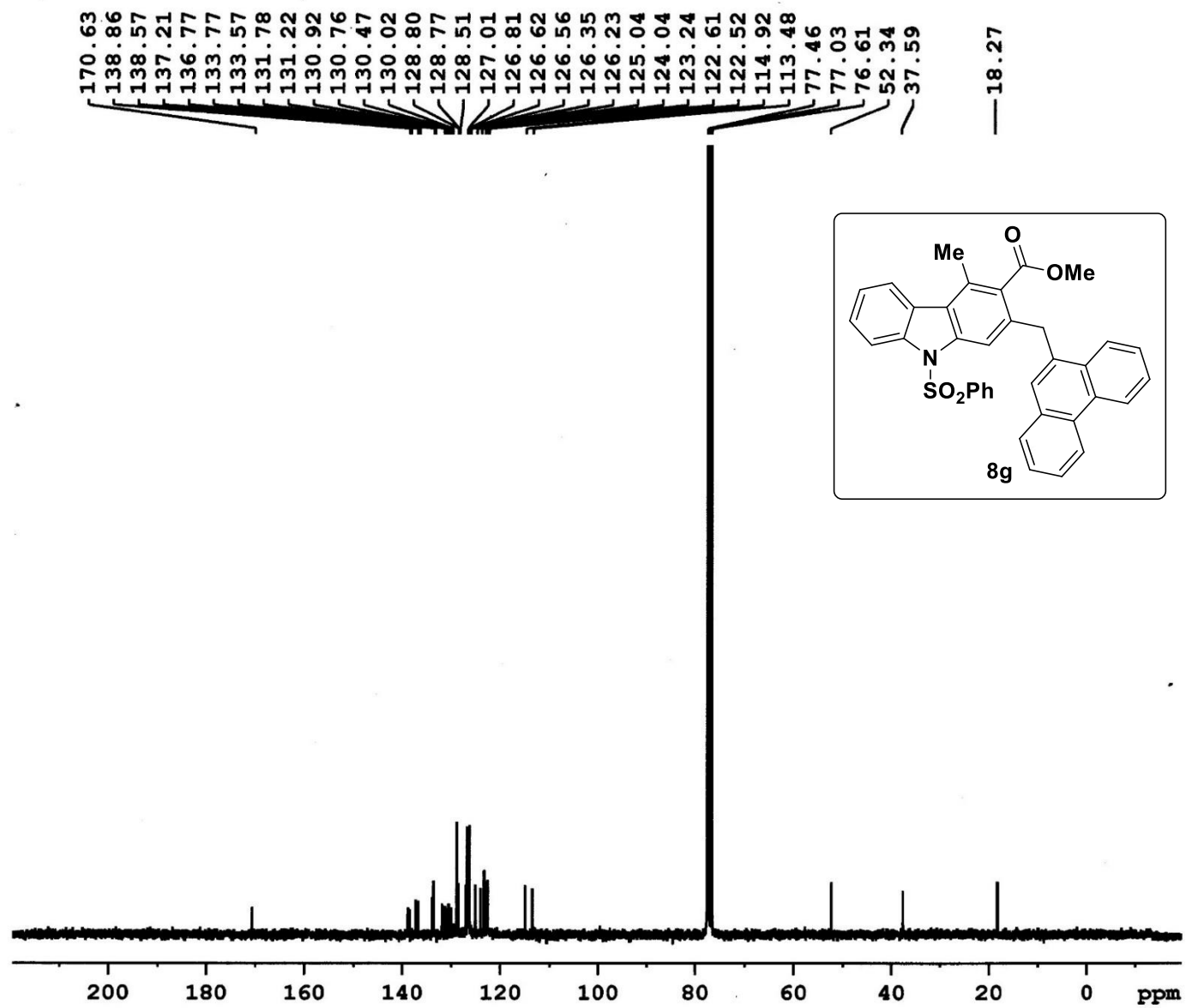
¹H NMR (300 MHz, CDCl₃) spectrum of compound **8f**



¹³C {¹H} NMR (75 MHz, CDCl₃) spectrum of compound **8f**



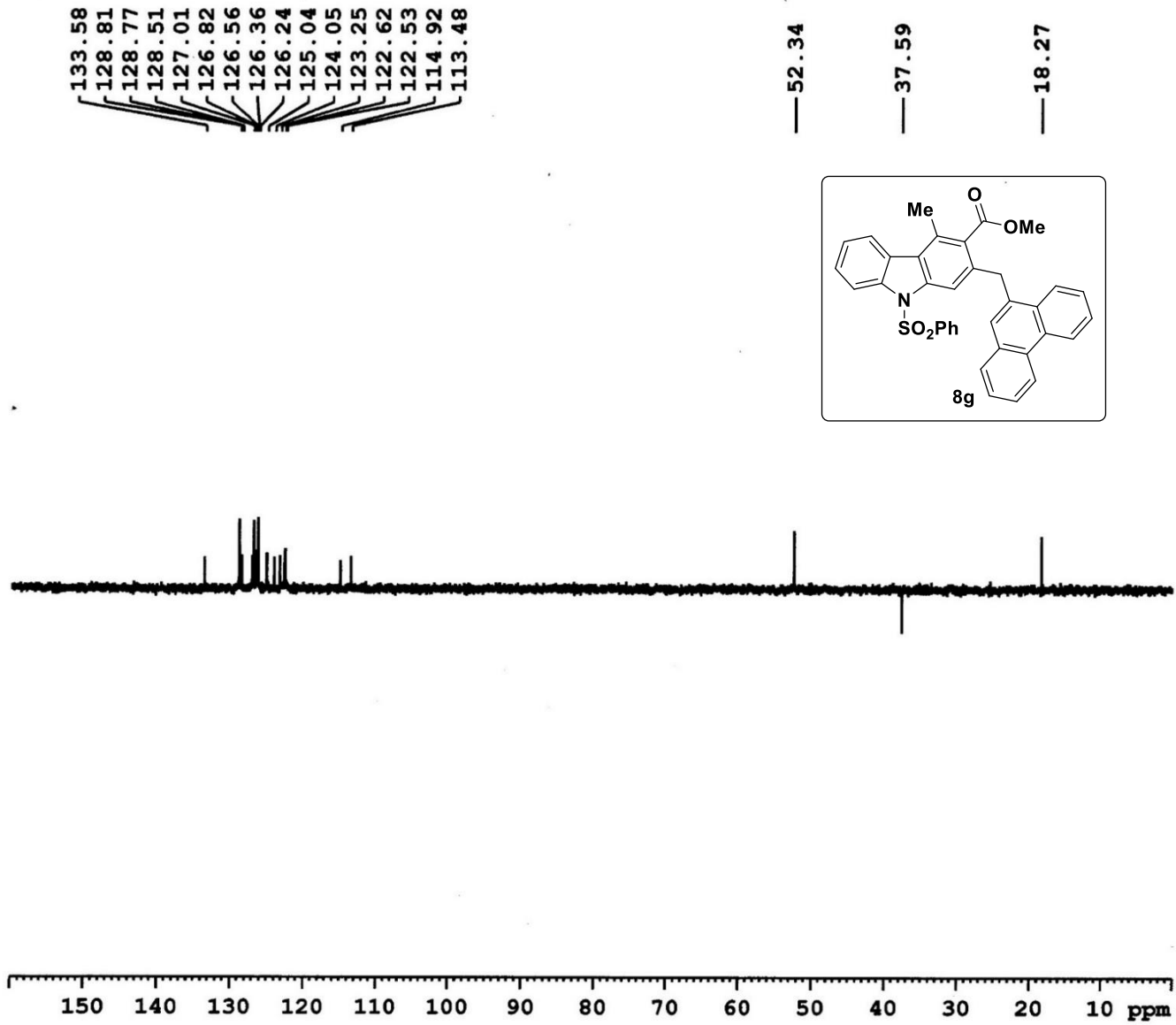
¹H NMR (300 MHz, CDCl₃) spectrum of compound **8g**



170.63
138.86
138.57
137.21
136.77
133.77
133.57
131.78
131.22
130.92
130.76
130.47
130.02
128.80
128.77
128.51
127.01
126.81
126.62
126.56
126.35
126.23
125.04
124.04
123.24
122.61
122.52
114.92
113.48
77.46
77.03
76.61
52.34
37.59

— 18.27

13C {1H} NMR (75 MHz, CDCl₃) spectrum of compound 8g

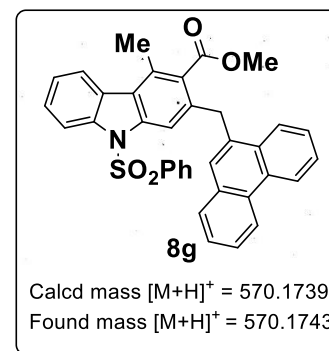
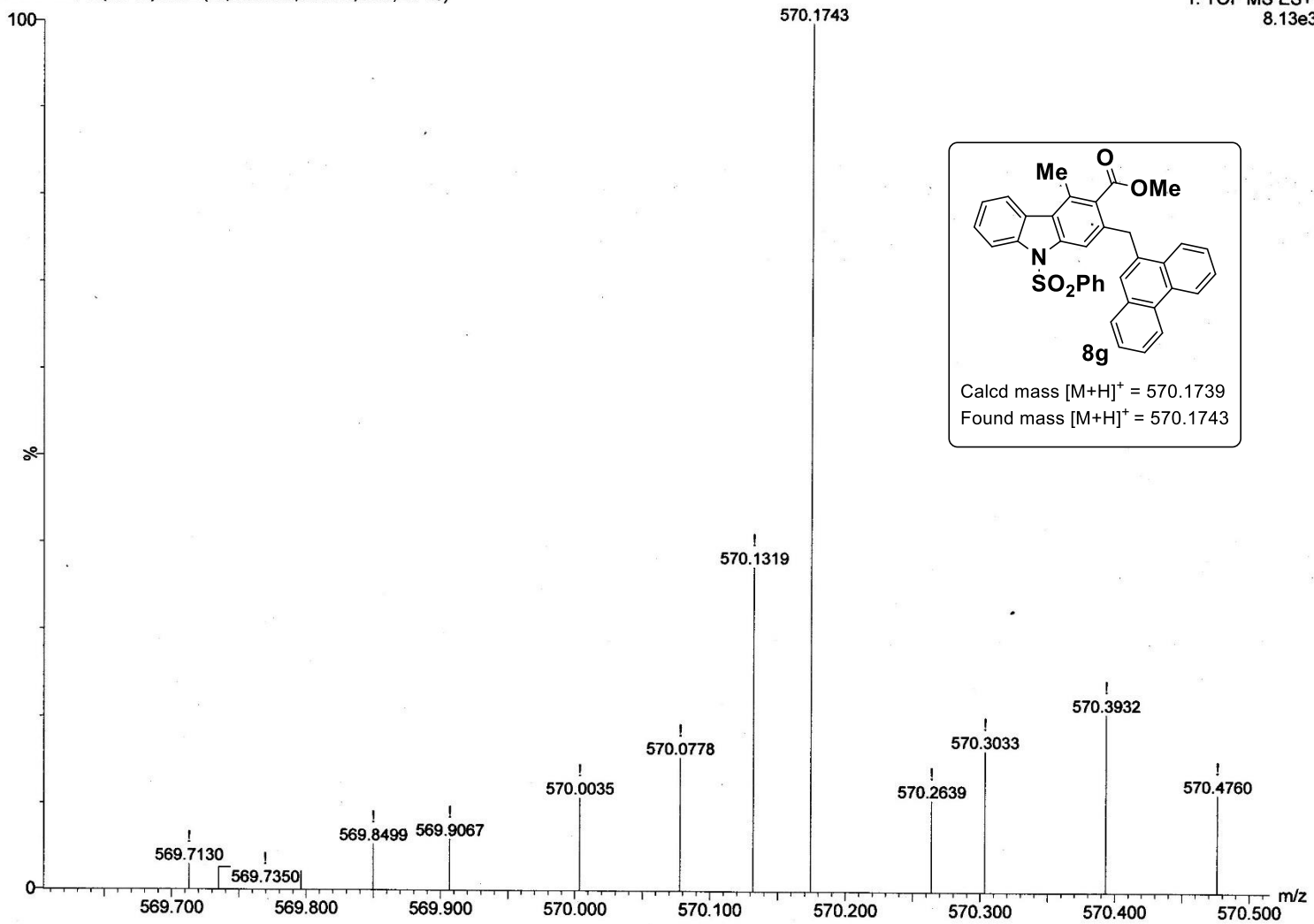


DEPT-135 NMR (75 MHz, CDCl₃) spectrum of compound **8g**

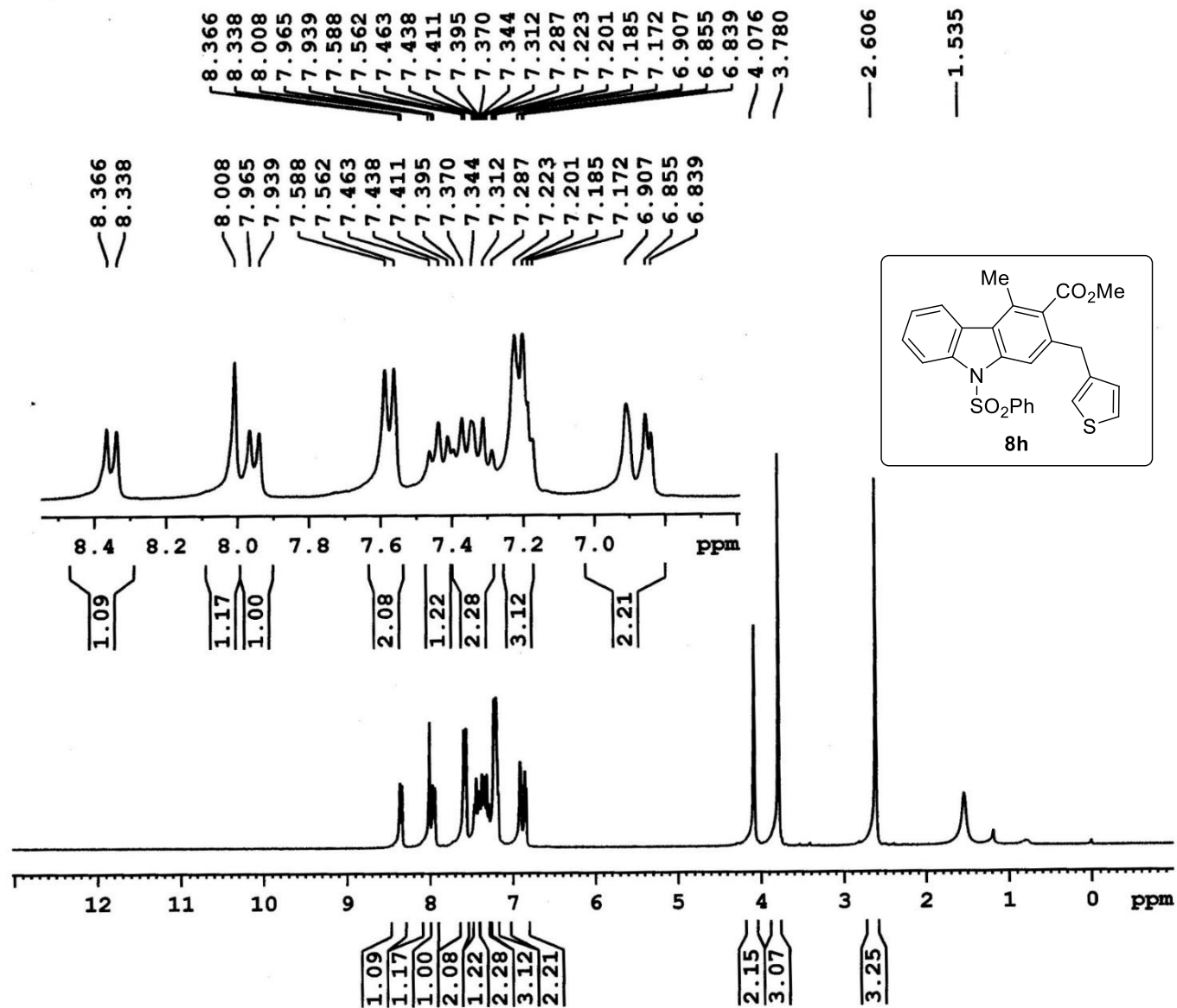
DRAKM

AG-V-186 9 (0.347) AM2 (Ar,20000.0,556.28,0.00,LS 10)

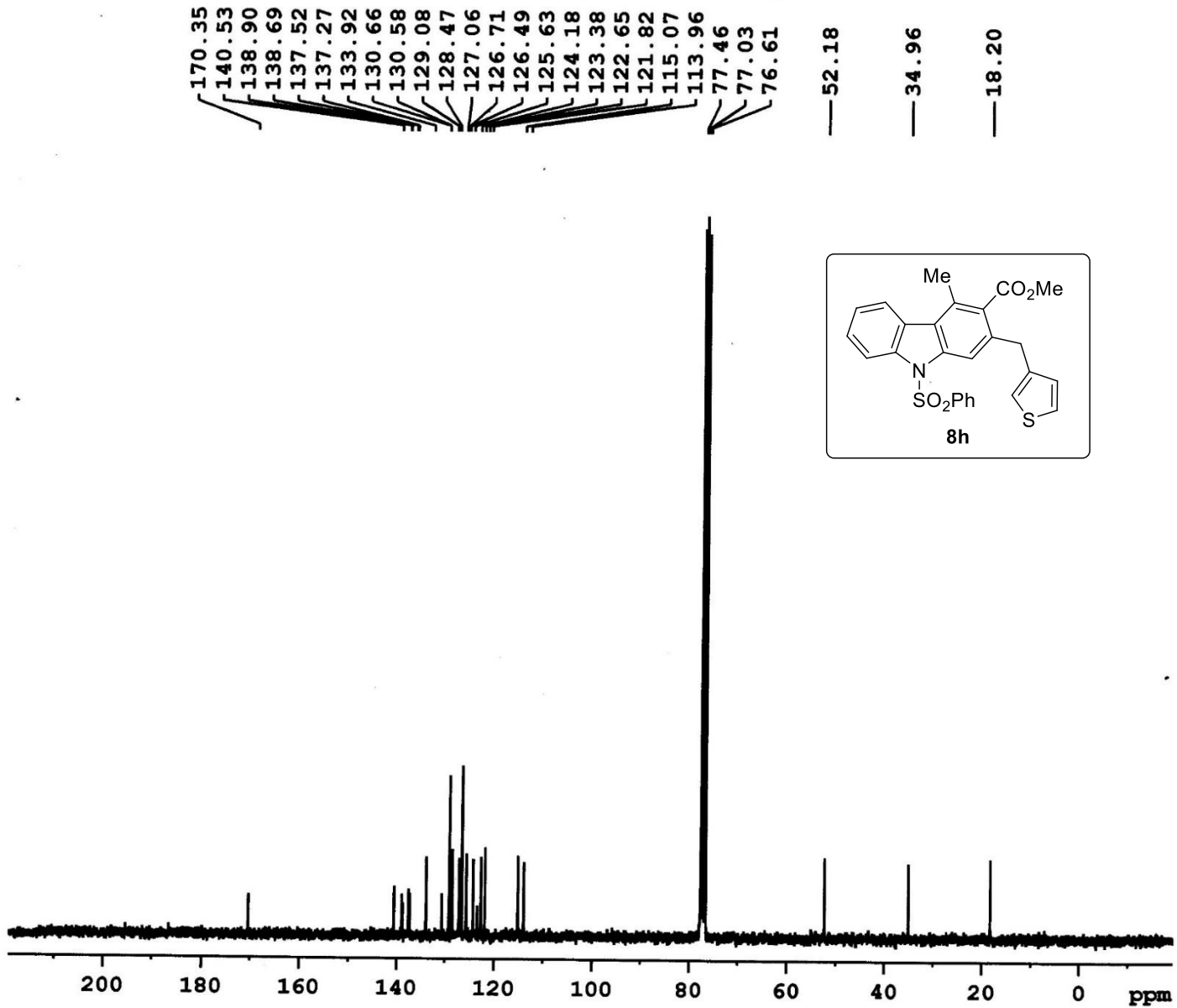
1: TOF MS ES+
8.13e3



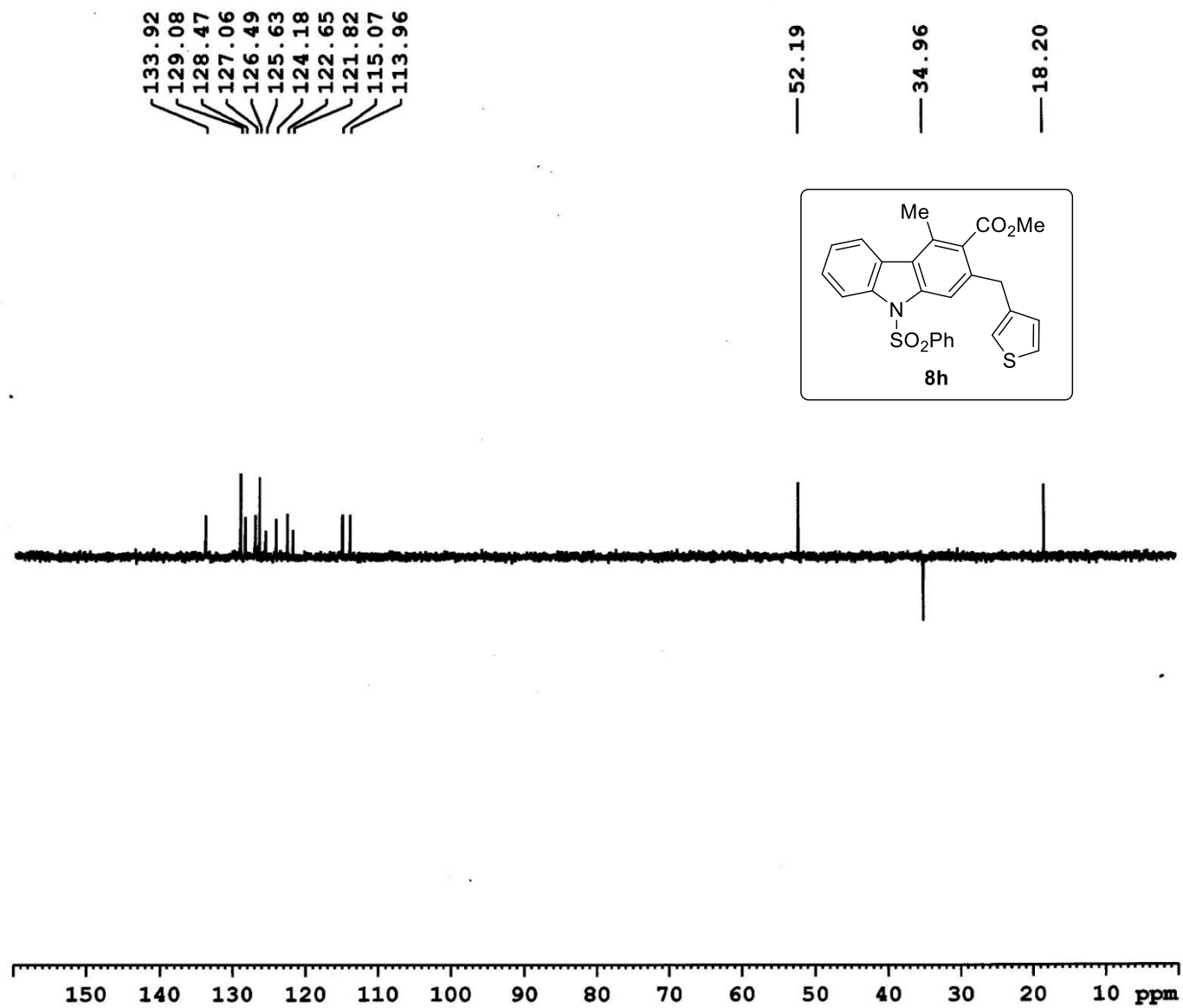
HRMS Spectrum of compound **8g**



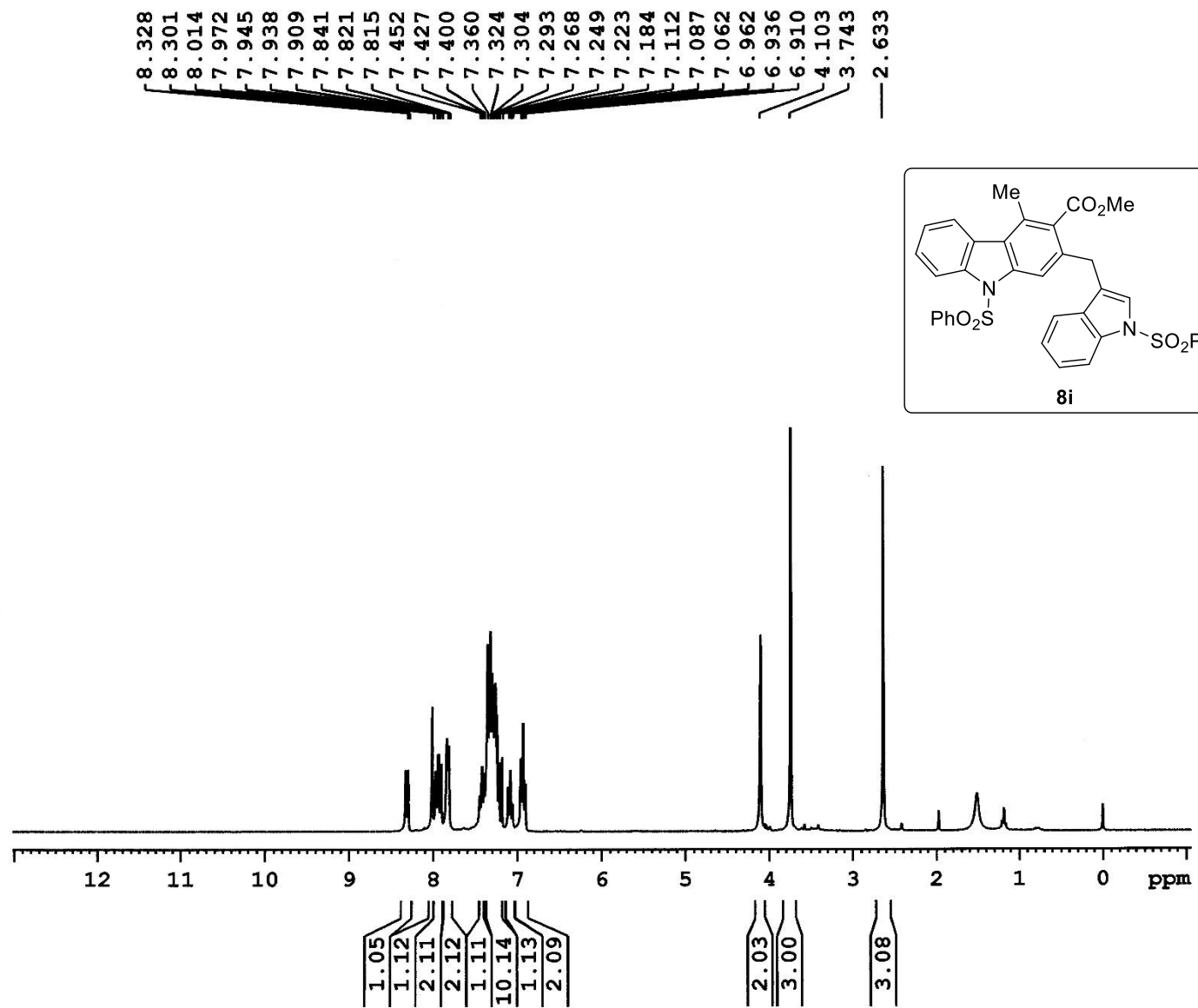
¹H NMR (300 MHz, CDCl₃) spectrum of compound 8h



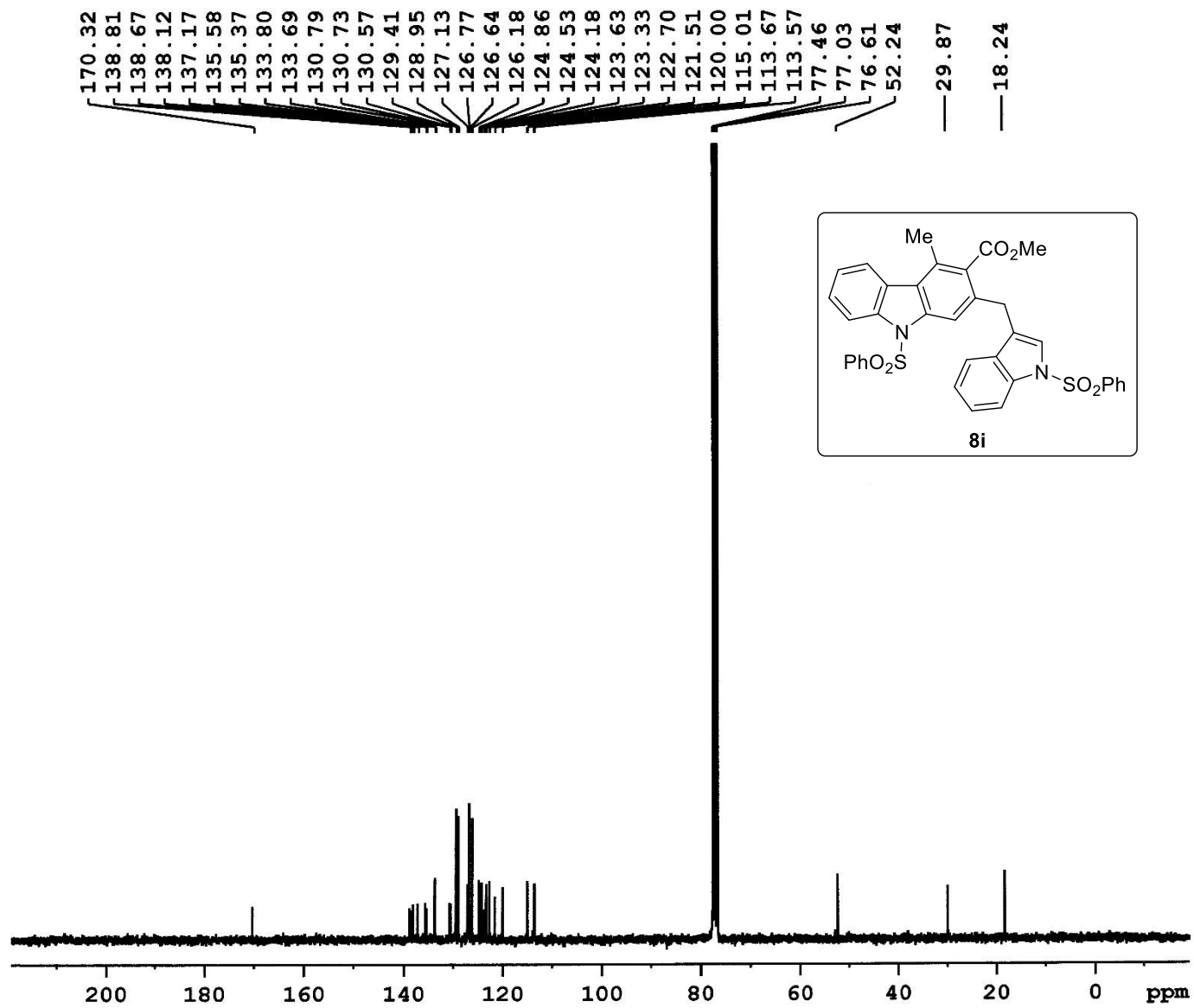
¹³C {¹H} NMR (CDCl₃, 75 MHz) spectrum of compound **8h**



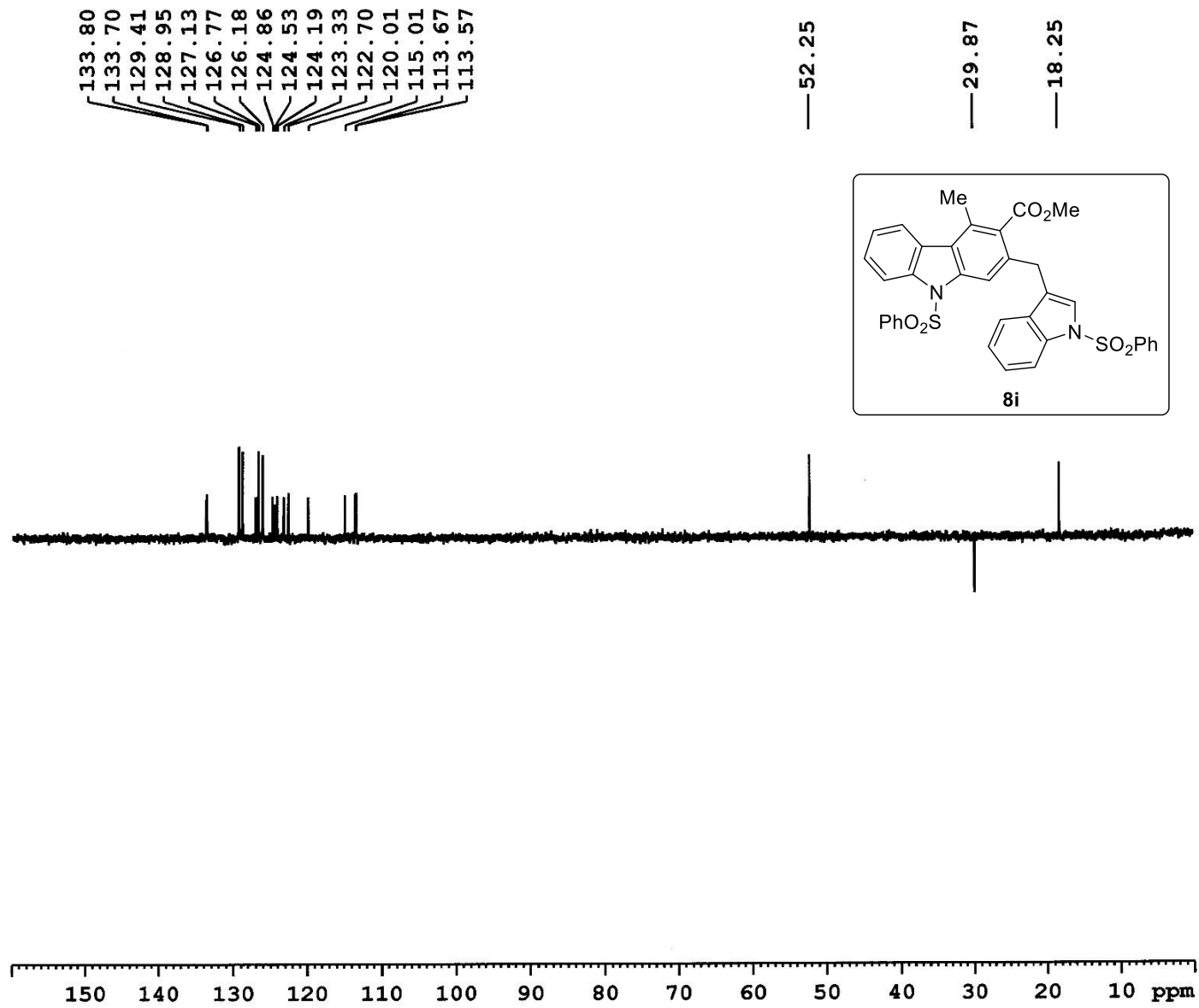
DEPT-135 NMR (75 MHz, CDCl₃) spectrum of compound **8h**



¹H NMR (300 MHz, CDCl₃) spectrum of compound **8i**

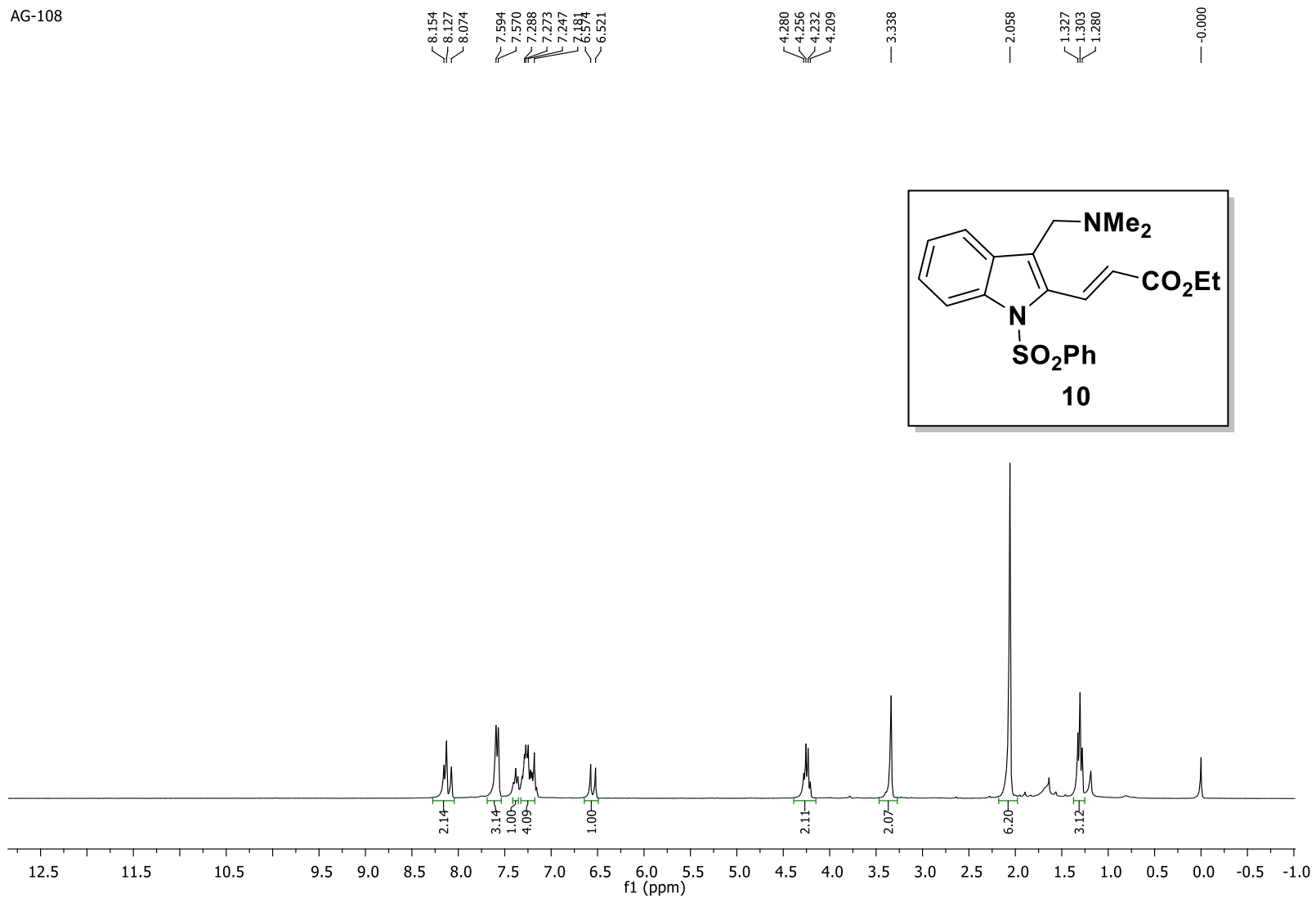


¹³C {¹H} NMR (CDCl₃, 75 MHz) spectrum of compound **8i**



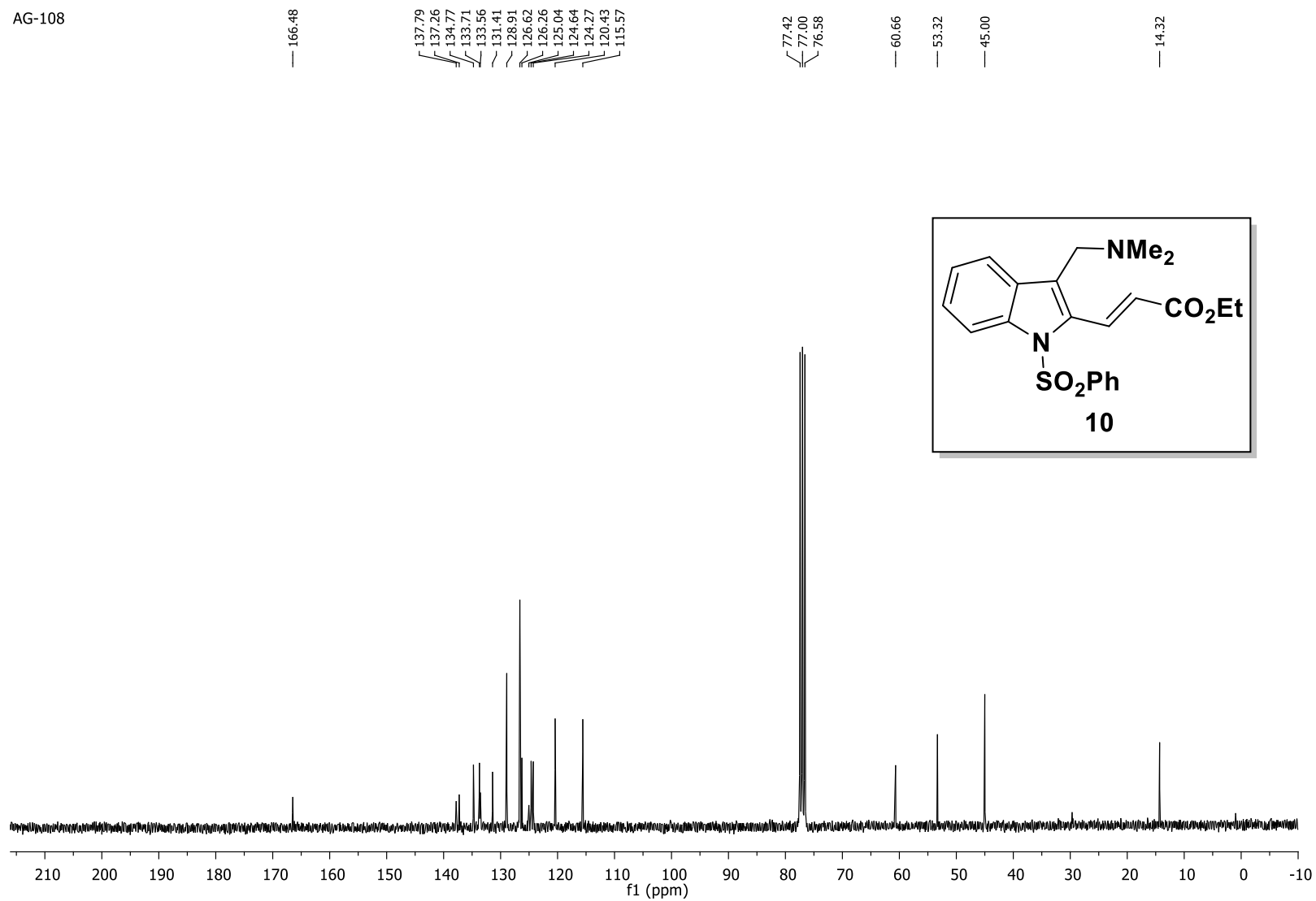
DEPT-135 NMR (75 MHz, CDCl₃) spectrum of compound **8i**

AG-108



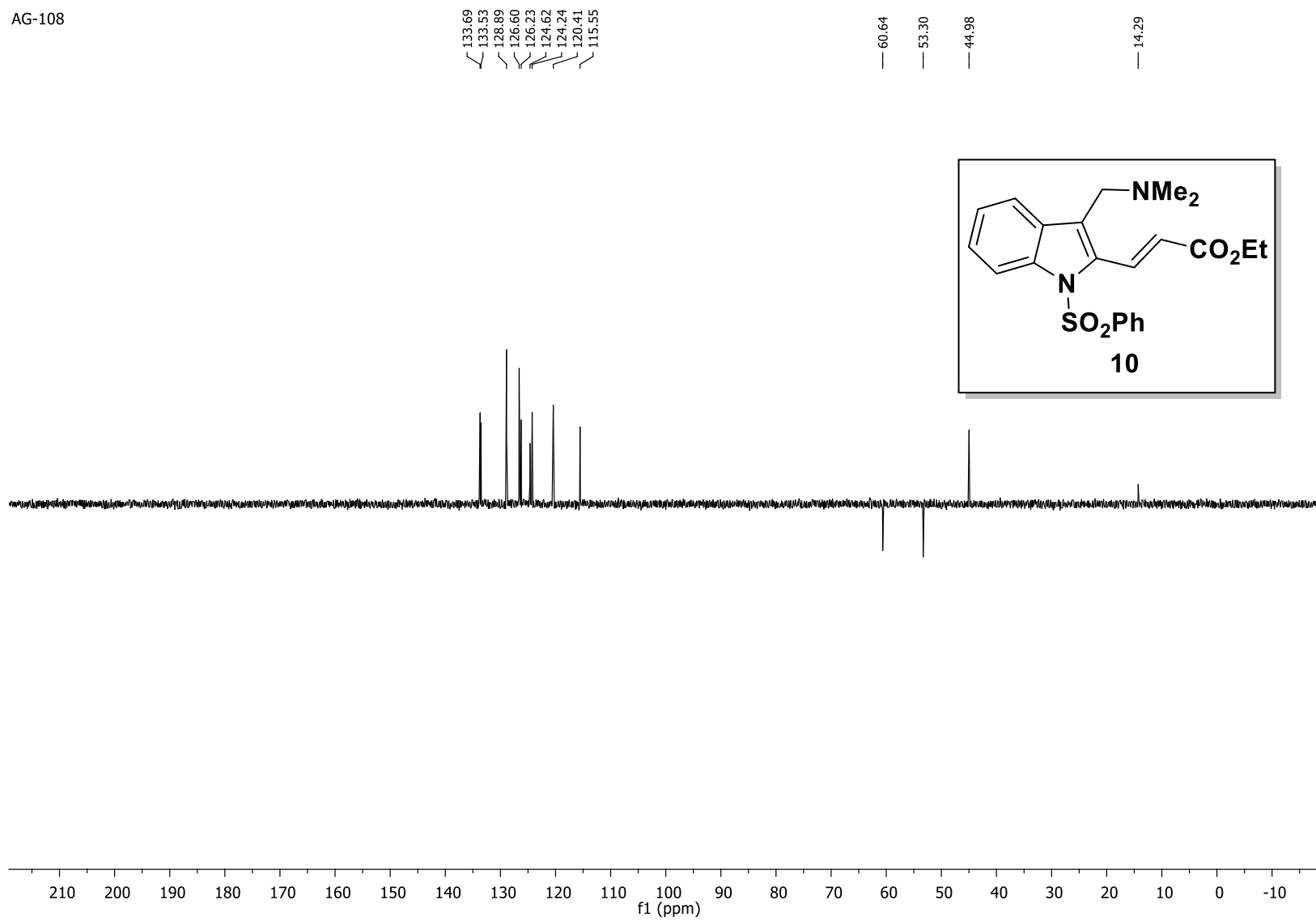
¹H NMR (300 MHz, CDCl₃) spectrum of compound **10**

AG-108



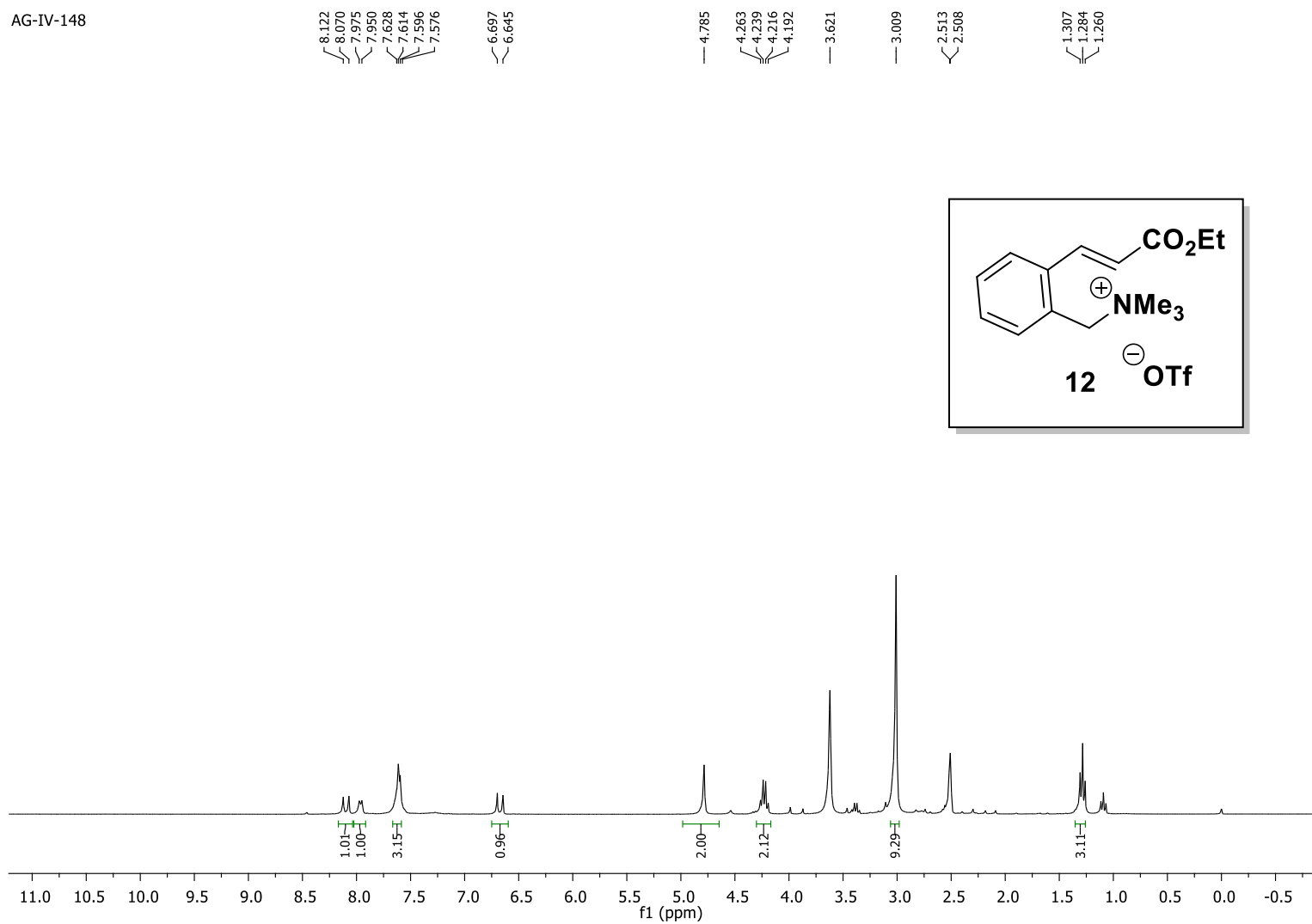
^{13}C { ^1H } NMR (CDCl_3 , 75 MHz) spectrum of compound **10**

AG-108



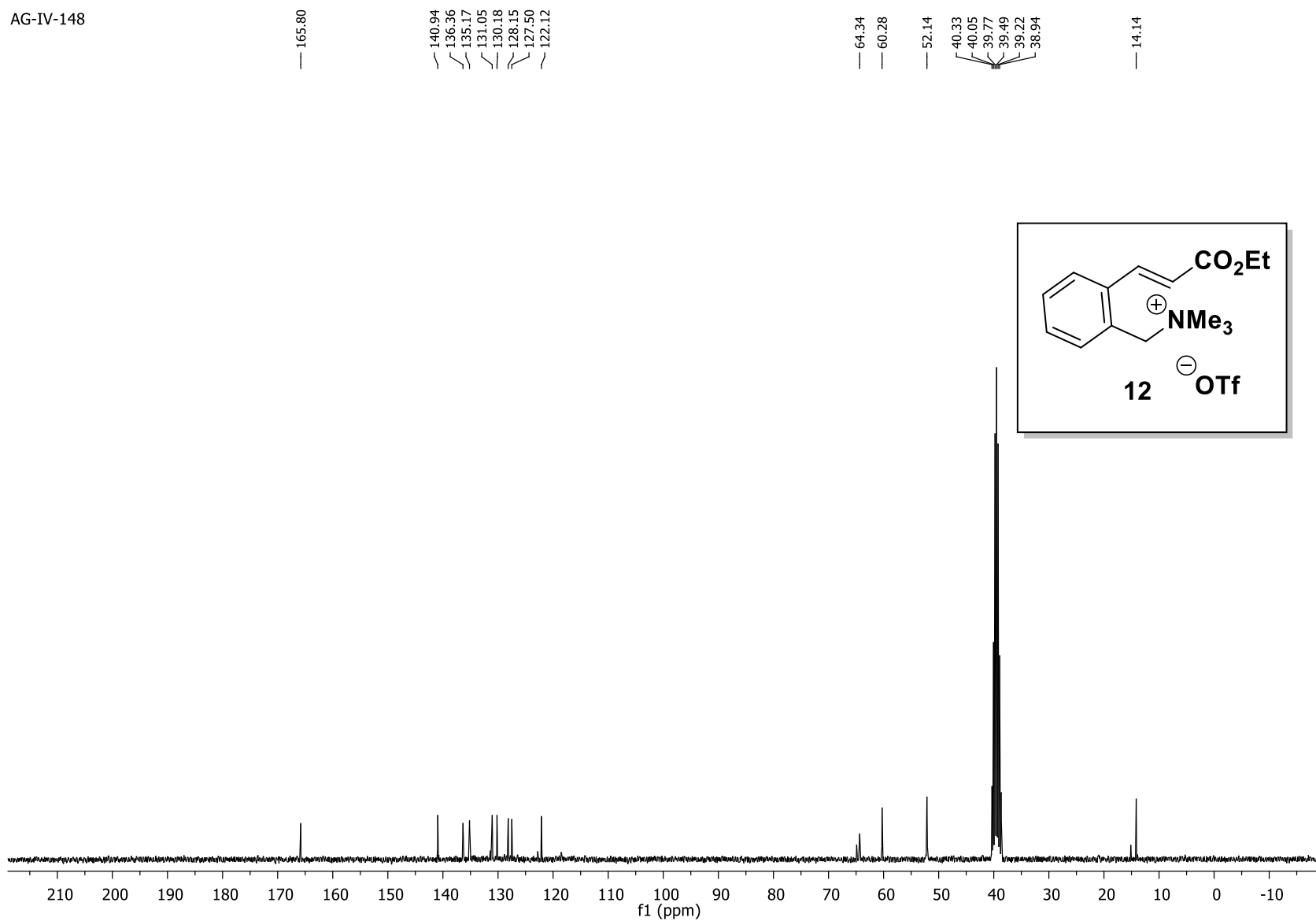
DEPT-135 NMR (75 MHz, CDCl₃) spectrum of compound **10**

AG-IV-148



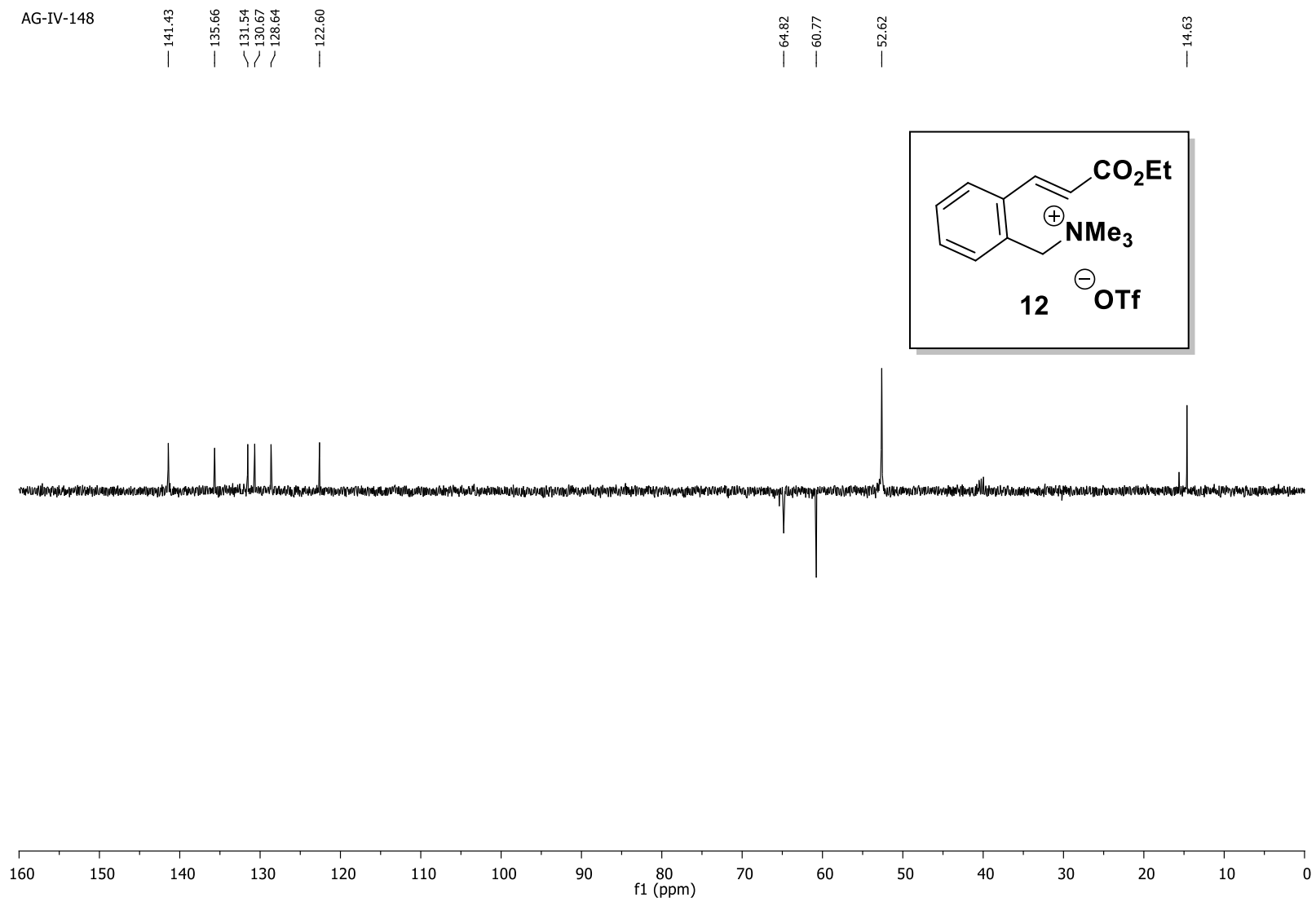
¹H NMR (300 MHz, DMSO-*d*₆) spectrum of compound 12

AG-IV-148



¹³C {¹H} NMR (75 MHz, DMSO-*d*₆) spectrum of compound **12**

AG-IV-148



DEPT-135 NMR (75 MHz, DMSO- d_6) spectrum of compound **12**