Photoinduced C-C Bond Cleavge for the Synthesis of 2,4-Disubstituted-1-Naphthols from Indenone Derivatives and Sulfoxonium Ylides

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Supporting Information

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■ EXPERIMENTAL SECTION

General Procedure. 1 Commercial grade reagents and solvents were used without further purification, unless otherwise specified. All solvents used were purified by the solvent purification system. The oven-dried glassware (110 °C at least for 2 h) was used for all reactions. Crude reaction mixtures were concentrated under reduced pressure by removing organic solvent with the rotary evaporator. Column chromatography was performed using silica gel 60 (particle size 0.06–0.2 mm; 70–230 mesh ASTM). Analytical thin layer chromatography (TLC) was performed with silica gel 60 F254 aluminum sheets. The nuclear magnetic resonance (NMR) spectra were recorded in deuterochloroform ($CDCl_3$) with 300 MHz spectrometer. ^1H NMR and $^{13}C\{1H\}$ NMR chemical shifts (δ) were reported in ppm (part per million). The chemical shift of ^1H NMR spectra is reported with reference to tetramethylsilane (TMS, 0.00 ppm) as the internal reference while chemical shift of $^{13}C\{1H\}$ NMR is reported with reference to $CDCl_3$ at 77.0 ppm. Chemical shift of ^{19}F NMR is reported with reference to hexafluorobenzene as the internal reference at –164.9 ppm. Coupling constants (*J*) were reported in hertz (Hz). Infrared spectra were measured using an FT-IR spectrometer and were reported in cm^{-1}. High resolution mass spectra (HRMS) were obtained using time-of-flight (TOF) and orbitrap.

The commercial grade chemicals were used without further purification, unless otherwise specified. All solvents used were purified by the solvent purification system. The oven-dried glassware (110 ºC at least for two hours) was used for all reactions. Crude reaction mixtures were concentrated under reduced pressure by removing organic solvent with the rotary evaporator. Column chromatography was performed using silica gel 60 (particle size 0.06-0.2 mm; 70-230 mesh ASTM). Analytical thin layer chromatography (TLC) was performed with silica gel 60 F_{254} aluminum sheets. The nuclear magnetic resonance (NMR) spectra were recorded in deuterochloroform ($CDCl_3$) with 300 and 600 MHz spectrometers. Chemical shifts for ^1H NMR and ^{13}C NMR spectra were reported in part per million (ppm, δ), relative to tetramethylsilane (TMS) as the internal reference. Coupling constants (*J*) were reported in Hertz (Hz). Infrared spectra were measured using FT-IR spectrometer and were reported in cm^{-1}. High resolution mass spectra (HRMS) were obtained using time-of-flight (TOF).

General procedure for the synthesis of *ortho*-ketophenylacetylenol (1a): Indeonone **3a** (118.7 mg, 0.4204 mmol, 1.0 equiv) was dissolved in dry THF (2.0 mL) in vial with screw cap. This mixture was then added trimethylsulfoxonium chloride (59.3 mg, 0.4624 mmol, 1.1 equiv) and NaH (18.5 mg, 0.4624 mmol, 1.1 equiv) at room temperature and stirred for overnight at room temperature. The reaction was diluted with sat. NH_4Cl and extracted with EtOAc. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure to provide the crude product which was purified on silica gel (EtOAc/hexane: 3:7) to yield the corresponding product **1a** 106.6 mg (86%, white solid); mp 168.3-170.0 °C; IR (neat) ν_{max}: 2923, 1700, 1096, 697 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.80 (d, 1H, *J* = 7.5 Hz), 7.48 (t, 1H, *J* = 7.5 Hz), 7.34 (t, 1H, *J* = 7.5 Hz), 7.23-7.11 (m, 11H), 2.72 (d, 1H, *J* = 4.8 Hz), 2.19 (d, 1H, *J* = 4.5 Hz); $^{13}C\{1H\}$ NMR (75 MHz, $CDCl_3$) δ 202.3, 156.7, 134.24, 134.16, 133.5, 133.0, 130.0, 129.5, 128.3, 128.2, 127.4, 127.3, 127.2, 124.9, 124.2,

49.6, 45.0, 39.8; HRMS (ESI-TOF) m/z: calcd for $C_{22}H_{17}O$ $[M+Na]^+$ 297.1274; found 297.1268.

General procedure for the synthesis of *ortho*-ketophenylacetylenol (1f): Indeonone **3f** (95.3 mg, 0.3216 mmol, 1.0 equiv) was dissolved in dry THF (2.0 mL) in vial with screw cap. This mixture was then added trimethylsulfoxonium chloride (45.1 mg, 0.3537 mmol, 1.1 equiv), TEMPO (50.3 mg, 0.3216 mmol, 1.0 equiv) and NaH (14.0 mg, 0.3537 mmol, 1.1 equiv) at room temperature and stirred for overnight at room temperature. The reaction was diluted with sat. NH_4Cl and extracted with EtOAc. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure to provide the crude product which was purified on silica gel (EtOAc/hexane: 3:7) to yield the corresponding product **1f** 43.2 mg (43%, white solid); mp 154.1-155.5 °C; IR (neat) ν_{max}: 2920, 1707, 947, 698 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.71 (d, 1H, *J* = 7.5 Hz), 7.46 (t, 1H, *J* = 7.5, 0.9 Hz), 7.25 (t, 1H, *J* = 7.1 Hz), 7.17-7.10 (m, 6H), 6.98 (t, 1H, *J* = 7.1 Hz), 6.88-6.85 (m, 2H), 6.81 (d, 1H, *J* = 7.6 Hz), 2.62 (d, 1H, *J* = 4.7 Hz), 2.13 (s, 3H), 2.09 (d, 1H, *J* = 4.7 Hz); $^{13}C\{1H\}$ NMR (100 MHz, $CDCl_3$) δ 202.1, 156.4, 137.2, 133.9, 133.7, 133.1, 132.4, 130.6, 129.2, 127.9, 127.64, 127.60, 127.0, 126.8, 126.6, 124.5, 123.8, 49.2, 44.5, 39.4, 20.9; HRMS (ESI-TOF) m/z: calcd for $C_{23}H_{19}O$ $[M+H]^+$ 311.1430; found 311.1428.

General procedure for the synthesis of *ortho*-ketophenylacetylenol (4): Indeonone **3a** (107.2 mg, 0.3797 mmol, 1.0 equiv) was dissolved in dry THF (2.0 mL) in vial with screw cap. This mixture was then added trimethylsulfoxonium chloride (53.5 mg, 0.0.4176 mmol, 1.1 equiv) and NaH (16.7 mg, 0.4176 mmol, 1.1 equiv) at room temperature and stirred under photoirradiation (390-nm UV-LED light (40 Watt) from Kessil PR160-390nm) for overnight at room temperature. The reaction was diluted with sat. NH_4Cl and extracted with EtOAc. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered and concentrated under reduced pressure to provide the crude product which was purified on silica gel (EtOAc/hexane: 1:9) to yield the corresponding product **4a** 90.8 mg (81%, pale brown solid); mp 103.6-104.8 °C; IR (neat) ν_{max}: 3536, 1372, 1211, 759 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.39 (d, 1H, *J* = 8.0 Hz), 7.90 (d, 1H, *J* = 8.1 Hz), 7.57-7.34 (m, 13H), 5.89 (s, 1H); $^{13}C\{1H\}$ NMR (75 MHz, $CDCl_3$) δ 147.3, 140.6, 137.2, 132.8, 132.1, 130.3, 129.6, 129.4, 128.5, 128.2, 128.0, 127.0, 126.6, 125.8, 125.5, 124.5, 122.7, 120.8; HRMS (ESI-TOF) m/z: calcd for $C_{22}H_{17}O$ $[M+H]^+$ 297.1274; found 297.1260.

*2-phenyl-4-(p-tolyl)naphthalen-1-ol (**4b**)*. Yield 84.5 mg (79%, yellow oil); IR (neat) ν_{max}: 3546, 2923, 1662, 1265, 736 cm^{-1}; ^1H NMR (400 MHz, Acetone-d_6) δ 8.48 (d, 1H, *J* = 8.0 Hz), 8.13 (s, 1H), 7.90 (d, 1H, *J* = 8.0 Hz), 7.64 (d, 2H, *J* = 7.6 Hz), 7.56-7.28 (m, 10H), 2.40 (s, 3H); $^{13}C\{1H\}$ NMR (100 MHz, Acetone-d_6) δ 149.1, 139.4, 138.6, 137.3, 133.3, 133.0, 130.9, 130.6, 130.0, 129.8, 129.5, 128.0, 127.9, 127.5, 127.1, 126.8, 126.5, 126.0, 123.6, 123.1, 21.2; HRMS (ESI-TOF) m/z: calcd for $C_{23}H_{17}O$ $[M-H]^-$ 309.1285; found 309.1282.

*4-(4-methoxyphenyl)-2-phenylnaphthalen-1-ol (**4c**)*. Yield 85.1 mg (80%, brown solid); mp 114.1-115.8 °C; IR (neat) ν_{max}: 3537, 2907, 1243, 765 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.39 (d, 1H, *J* = 7.8 Hz), 7.93 (d, 1H, *J* = 8.1 Hz), 7.62-7.44 (m, 9H), 7.34 (s, 1H), 7.04 (d, 2H, *J* = 8.4 Hz), 5.89 (s, 1H), 3.91 (s, 3H); $^{13}C\{1H\}$ NMR (75 MHz, $CDCl_3$) δ 158.7, 147.1, 137.3, 132.9, 132.4, 132.3, 131.2, 129.6, 129.3, 128.4, 127.9, 126.4, 125.8, 125.4, 124.5, 122.6, 120.8, 113.7, 55.3; HRMS (ESI-TOF) m/z: calcd for $C_{23}H_{17}O_2$ $[M-H]^-$ 325.1234; found 325.1226.

*4-(4-chlorophenyl)-2-phenylnaphthalen-1-ol (**4d**)*. Yield 78.5 mg (74%, yellow oil); IR (neat) ν_{max}: 3536, 1490, 1371, 1210, 757 cm^{-1}; ^{1}H NMR (400 MHz, Acetone-d_6) δ 8.48 (d, 1H, *J* = 8.0 Hz), 8.25 (br s, 1H), 7.83 (d, 1H, *J* = 8.4 Hz), 7.63 (d, 2H, *J* = 7.2 Hz), 7.57-7.44 (m, 8H), 7.37-7.33 (m, 2H); $^{13}C\{1H\}$ NMR (100 MHz, Acetone-d_6) δ 149.5, 140.2, 139.1, 133.3, 132.63, 132.57, 131.7, 130.7, 130.3, 129.5, 129.2, 128.1, 127.4, 126.8, 126.2, 126.0, 123.7, 123.1; HRMS (ESI-TOF) m/z: calcd for $C_{22}H_{14}ClO$ (Cl-35) $[M-H]^-$ 329.0739; found 329.0741.

*4-phenyl-2-(p-tolyl)naphthalen-1-ol (**4e**)*. Yield 99.6 mg (79%, yellow oil); IR (neat) ν_{max}: 3527, 2921, 1514, 1210, 772 cm^{-1}; ^{1}H NMR (400 MHz, Acetone-d_6) δ 8.46 (d, 1H, *J* = 7.2 Hz), 8.09 (s, 1H), 7.87 (d, 1H, *J* = 8.0 Hz), 7.53-7.27 (m, 12H), 2.37 (s, 3H); $^{13}C\{1H\}$ NMR (100 MHz, Acetone-d_6) δ 149.2, 141.6, 137.5, 136.2, 133.2, 132.8, 131.0, 130.5, 130.2, 129.2, 127.8, 127.1, 126.8, 126.3, 126.0, 123.6, 123.0, 21.2; HRMS (ESI-TOF) m/z: calcd for $C_{23}H_{17}O$ $[M-H]^-$ 309.1285; found 309.1281.

*4-phenyl-2-(m-tolyl)naphthalen-1-ol (**4f**)*. Yield 73.5 mg (56%, brown oil); IR (neat) ν_{max}: 3534, 2920, 1600, 1370, 701 cm^{-1}; ^{1}H NMR (400 MHz, Acetone-d_6) δ 8.45 (d, 1H, *J* = 8.4 Hz), 8.10 (s, 1H), 7.86 (d, 1H, *J* = 8.4 Hz), 7.57-7.32 (m, 11H), 7.19 (d, 1H, *J* = 7.2 Hz), 2.40 (s, 3H); $^{13}C\{1H\}$ NMR (100 MHz, Acetone-d_6) δ 149.2, 141.6, 139.11, 139.06, 133.1, 132.8, 131.2, 131.0, 130.1, 129.5, 129.3, 128.7, 127.8, 127.6, 127.1, 126.7, 126.3, 126.0, 123.6, 123.0, 21.5; HRMS (ESI-TOF) m/z: calcd for $C_{23}H_{17}O$ $[M-H]^-$ 309.1285; found 309.1285.

*2-(4-hexylphenyl)-4-phenylnaphthalen-1-ol (**4g**)*. Yield 66.4 mg (58%, brown oil); IR (neat) ν_{max}: 3544, 2927, 1372, 1048, 702 cm^{-1}; ^{1}H NMR (400 MHz, Acetone-d_6) δ 8.46 (d, 1H, *J* = 8.4 Hz), 8.09 (s, 1H), 7.87 (d, 1H, *J* = 8.4 Hz), 7.56-7.40 (m, 9H), 7.33-7.28 (m, 3H), 2.66 (t, 1H, *J* = 7.6 Hz), 1.66 (quint, 2H, *J* = 7.2 Hz), 1.41-1.28 (m, 6H), 0.89 (t, 3H, *J* = 6.8 Hz); $^{13}C\{1H\}$ NMR (100 MHz, Acetone-d_6) δ 149.2, 142.6, 141.6, 136.5, 133.2, 132.7, 131.0, 130.4, 130.2, 129.6, 129.1, 127.7, 127.0, 126.8, 126.3, 126.0, 123.6, 123.0, 36.2, 32.4, 32.3, 29.7, 23.3, 14.4; HRMS (ESI-TOF) m/z: calcd for $C_{28}H_{27}O$ $[M-H]^-$ 379.2067; found 379.2060.

*2-(2-fluorophenyl)-4-phenylnaphthalen-1-ol (**4h**)*. Yield 81.7 mg (77%, yellow oil); IR (neat) ν_{max}: 3545, 1572, 1492, 1211, 758 cm^{-1}; ^{1}H NMR (400 MHz, Acetone-d_6) δ 8.47 (d, 1H, *J* = 7.2 Hz), 7.89 (d, 1H, *J* = 8.0 Hz), 7.58-7.39 (m, 10H), 7.30-7.21 (m, 3H); $^{13}C\{1H\}$ NMR (100 MHz, Acetone-d_6) δ 161.5 (d, J_{C-F} = 244 Hz), 150.5, 150.4, 141.6, 133.6 (d, J_{C-F} = 4 Hz), 133.4, 133.1, 131.2, 130.7 (d, J_{C-F} = 8 Hz), 130.5, 129.4, 128.1, 127.7, 126.9, 126.6, 126.3, 125.6 (d, J_{C-F} = 4 Hz), 123.9, 117.4, 116.8 (d, J_{C-F} = 22 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ –116.6 (S), HRMS (ESI-TOF) m/z: calcd for $C_{22}H_{14}FO$ $[M-H]^-$ 313.1034; found 313.1030.

*2-(3-fluorophenyl)-4-phenylnaphthalen-1-ol (**4i**)*. Yield 96.0 mg (77%, brown oil); IR (neat) ν_{max}: 3545, 1584, 1212, 771 cm^{-1}; ^{1}H NMR (400 MHz, Acetone-d_6) δ 8.49 (d, 1H, *J* = 8.4 Hz), 7.88 (d, 1H, *J* = 8.0 Hz), 7.57-7.36 (m, 10H), 7.12 (br s, 1H); $^{13}C\{1H\}$ NMR (100 MHz, Acetone-d_6) δ 163.7 (d, J_{C-F} = 242 Hz), 149.4, 141.8 (d, J_{C-F} = 8 Hz), 141.4, 133.4, 133.1, 131.2 (d, J_{C-F} = 8 Hz), 131.0, 129.7, 129.6 (d, J_{C-F} = 5 Hz), 129.2, 127.8, 127.4, 126.9, 126.6, 126.3 (d, J_{C-F} = 18 Hz), 123.6, 122.1, 117.4 (d, J_{C-F} = 21 Hz), 114.6 (d, J_{C-F} = 22 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ –119.9 (S), HRMS (ESI-TOF) m/z: calcd for $C_{22}H_{14}FO$ $[M-H]^-$ 313.1034; found 313.1033.

2-(4-fluorophenyl)-4-phenylnaphthalen-1-ol ***(4j)***. Yield 39.6 mg (67%, yellow oil); mp 69.8-70.2 °C; IR (neat) ν_{max}: 3420, 2928, 1514, 1247, 760 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$+MeOH-d_4) δ 8.37 (d, 1H, *J* = 8.4 Hz), 7.90 (d, 1H, *J* = 8.4 Hz), 7.58-7.40 (m, 9H), 7.29-7.19 (m, 3H), 4.84 (br s, 1H); $^{13}C\{1H\}$ NMR (100 MHz, $CDCl_3$+MeOH-d_4) δ 162.3 (d, J_{C-F} = 246 Hz), 147.4, 140.4, 133.4 (d, J_{C-F} = 2 Hz), 132.8, 132.0, 131.2 (d, J_{C-F} = 7 Hz), 130.2, 128.6, 128.2, 127.0, 126.5, 125.8, 125.5, 124.7, 122.5, 120.1, 116.2 (d, J_{C-F} = 21 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ –114.2 (S), HRMS (ESI-TOF) m/z: calcd for $C_{22}H_{14}FO$ $[M-H]^-$ 313.1034; found 313.1031.

2-(3-chlorophenyl)-4-phenylnaphthalen-1-ol ***(4k)***. Yield 76.1 mg (70%, yellow oil); IR (neat) ν_{max}: 3539, 1594, 1372, 1210, 700 cm^{-1}; ^{1}H NMR (400 MHz, Acetone-d_6) δ 8.57 (br s, 1H), 8.46 (d, 1H, *J* = 8.0 Hz), 7.87 (d, 1H, *J* = 8.4 Hz), 7.70 (s, 1H), 7.67-7.33 (m, 11H); $^{13}C\{1H\}$ NMR (100 MHz, Acetone-d_6) δ 148.6, 140.8, 140.5, 133.8, 132.6, 132.2, 130.2, 130.1, 129.7, 128.9, 128.4, 127.1, 127.0, 126.6, 126.1, 125.6, 125.4, 122.7, 121.1; HRMS (ESI-TOF) m/z: calcd for $C_{22}H_{14}ClO$ (Cl-35) $[M-H]^-$ 329.0739; found 329.0734.

2-(4-chlorophenyl)-4-phenylnaphthalen-1-ol ***(4l)*** Yield 86.7 mg (79%, yellow oil); IR (neat) ν_{max}: 3541, 1491, 1210, 1046, 700 cm^{-1}; ^{1}H NMR (400 MHz, Acetone-d_6) δ 8.45 (d, 1H, *J* = 8.1 Hz), 8.37 (br s, 1H), 7.87 (d, 1H, *J* = 8.4 Hz), 7.69-7.64 (m, 2H), 7.58-7.54 (m, 1H), 7.52-7.48 (m, 6H), 7.44-7.42 (m, 1H); $^{13}C\{1H\}$ NMR (100 MHz, Acetone-d_6) δ 149.4, 141.4, 138.2, 133.5, 133.4, 133.1, 132.4, 131.0, 129.8, 129.5, 129.2, 127.9, 127.4, 126.9, 126.5, 126.3, 123.6, 122.1; HRMS (ESI-TOF) m/z: calcd for $C_{22}H_{14}ClO$ (Cl-35) $[M-H]^-$ 329.0739; found 329.0733.

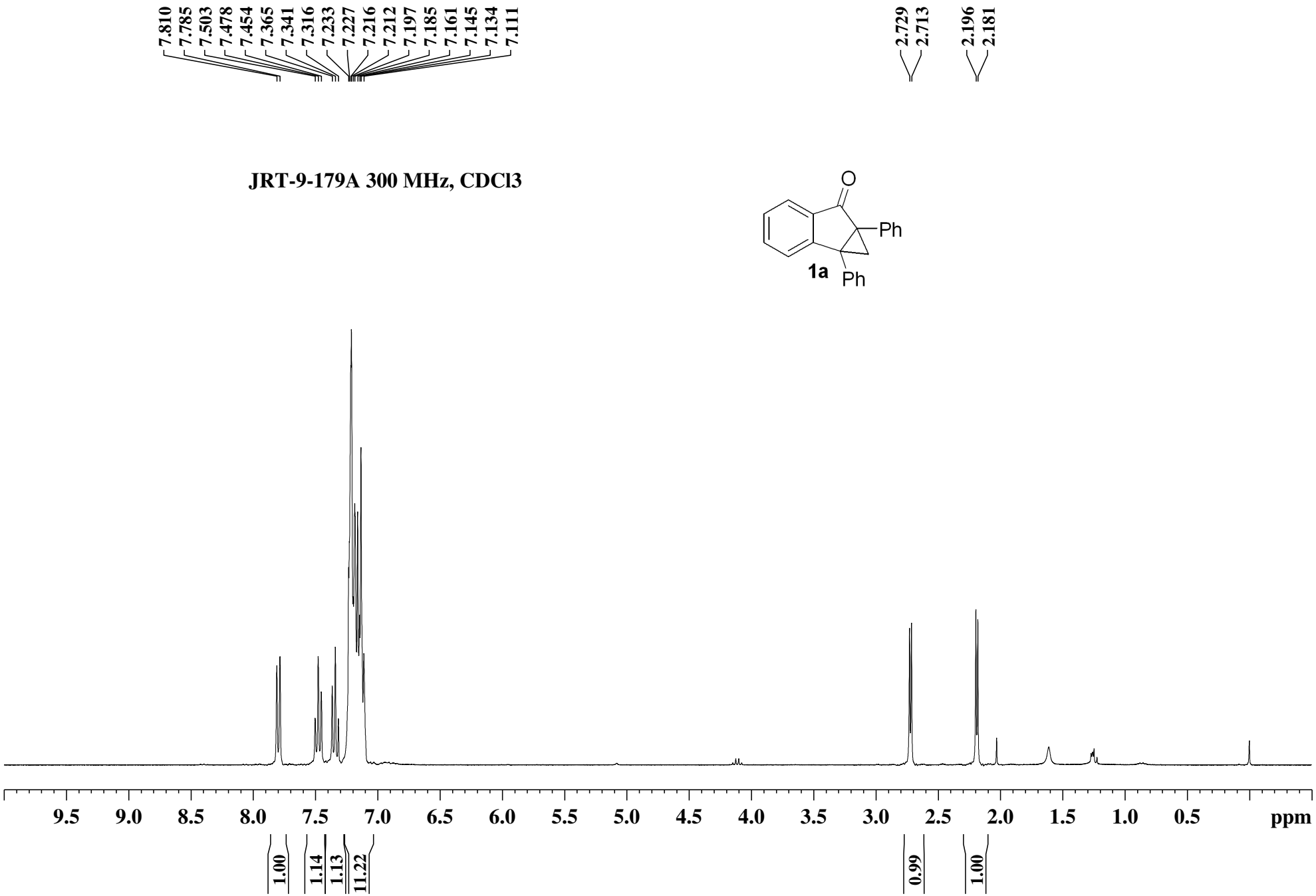
2-(3-methoxyphenyl)-4-phenylnaphthalen-1-ol ***(4m)***. Yield 89.6 mg (78%, brown oil); IR (neat) ν_{max}: 3525, 2937, 1596, 1215, 701 cm^{-1}; ^{1}H NMR (400 MHz, Acetone-d_6) δ 8.48 (d, 1H, *J* = 8.4 Hz), 8.14 (s, 1H), 7.88 (d, 1H, *J* = 8.4 Hz), 7.57-7.47 (m, 6H), 7.42-7.36 (m, 3H), 7.22-7.19 (m, 2H), 6.93 (d, 1H, *J* = 6.4 Hz), 3.84 (s, 3H); $^{13}C\{1H\}$ NMR (100 MHz, Acetone-d_6) δ 160.9, 149.2, 141.5, 133.1, 132.9, 131.0, 130.7, 130.0, 129.2, 127.8, 127.2, 126.7, 126.3, 126.1, 123.7, 122.9, 122.8, 116.0, 113.9, 55.6; HRMS (ESI-TOF) m/z: calcd for $C_{23}H_{17}O_2$ $[M-H]^-$ 325.1234; found 325.1227.

2-butyl-4-(4-fluorophenyl)naphthalen-1-ol ***(4n)***. Yield 13.0 mg (20%, brown solid); mp 109.9-111.7 °C; IR (neat) ν_{max}: 3422, 2930, 1515, 1174, 700 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.86-7.80 (m, 2H), 7.50-7.40 (m, 4H), 7.19-7.11 (m, 2H), 5.14 (s, 1H), 2.76 (t, 2H, *J* = 7.8 Hz), 1.75-1.65 (m, 2H), 1.58 (s, 1H), 1.51-1.39 (m, 2H), 0.96 (t, 3H, *J* = 7.2 Hz); $^{13}C\{1H\}$ NMR (75 MHz, $CDCl_3$) δ 160.5 (d, J_{C-F} = 244 Hz), 147.2 (d, J_{C-F} = 5 Hz), 140.6, 133.0, 130.2, 129.8, 128.6 (d, J_{C-F} = 5 Hz), 128.32, 128.28, 127.1, 125.6 (d, J_{C-F} = 9 Hz), 115.7 (d, J_{C-F} = 25 Hz), 105.4 (d, J_{C-F} = 5 Hz), 32.2, 29.7, 22.7. 14.0. ^{19}F NMR (376 MHz, $CDCl_3$) δ –118.5 (S), HRMS (ESI-TOF) m/z: calcd for $C_{20}H_{18}FO$ $[M-H]^-$ 293.1347; found 293.1350.

2-butyl-4-(4-methoxyphenyl)naphthalen-1-ol ***(4o)***. Yield 21.6 mg (36%, yellow solid); IR (neat) ν_{max}: 3543, 2924, 1509, 1224, 702 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$+MeOH-d_4) δ 8.20 (d, 1H, *J* = 8.4 Hz), 7.85 (d, 1H, *J* = 8.4 Hz), 7.47 (t, 1H, *J* = 7.2 Hz), 7.40-7.36 (m, 3H), 7.18 (s, 1H), 7.01 (d, 2H, *J* = 8.4 Hz), 5.26 (d, 1H, *J* = 3.6 Hz), 3.88 (s, 3H), 2.77 (t, 2H, *J* = 7.6 Hz), 1.73-1.65 (m, 2H), 1.48-1.39 (m, 2H), 0.95 (t, 3H, *J* = 7.2 Hz); $^{13}C\{1H\}$ NMR (100 MHz, $CDCl_3$+MeOH-d_4) δ 158.6, 147.4, 133.2, 132.4, 131.4, 131.2, 129.2, 125.9, 125.4, 125.1,

124.6, 121.3, 120.8, 113.6, 55.3, 32.3, 29.7, 22.7, 14.0; HRMS (ESI-TOF) m/z: calcd for $C_{21}H_{21}O_2$ $[M-H]^-$ 305.1547; found 305.1548.

*6-methoxy-4-(3-methoxyphenyl)-2-phenylnaphthalen-1-ol (**4p**)*. Yield 75.1 mg (71%, brown oil); IR (neat) ν_{max}: 3536, 2936, 1599, 1436, 1221, 702 cm^{-1}; ^1H NMR (400 MHz, Acetone-d_6) δ 8.05 (s, 1H), 7.84-7.78 (m, 2H), 7.65 (d, 2H, *J* = 6.4 Hz), 7.47-7.37 (m, 4H), 7.20-6.97 (m, 5H), 3.95 (s, 3H), 3.84 (s, 3H); $^{13}C\{1H\}$ NMR (100 MHz, Acetone-d_6) δ 160.7, 158.5, 148.3, 143.1, 139.5, 133.2, 130.6, 130.1, 129.9, 129.5, 128.8, 128.2, 128.0, 127.6, 123.7, 123.3, 119.5, 116.5, 113.4, 102.0, 55.6, 55.5; HRMS (ESI-TOF) m/z: calcd for $C_{24}H_{19}O_3$ $[M-H]^-$ 355.1340; found 355.1332.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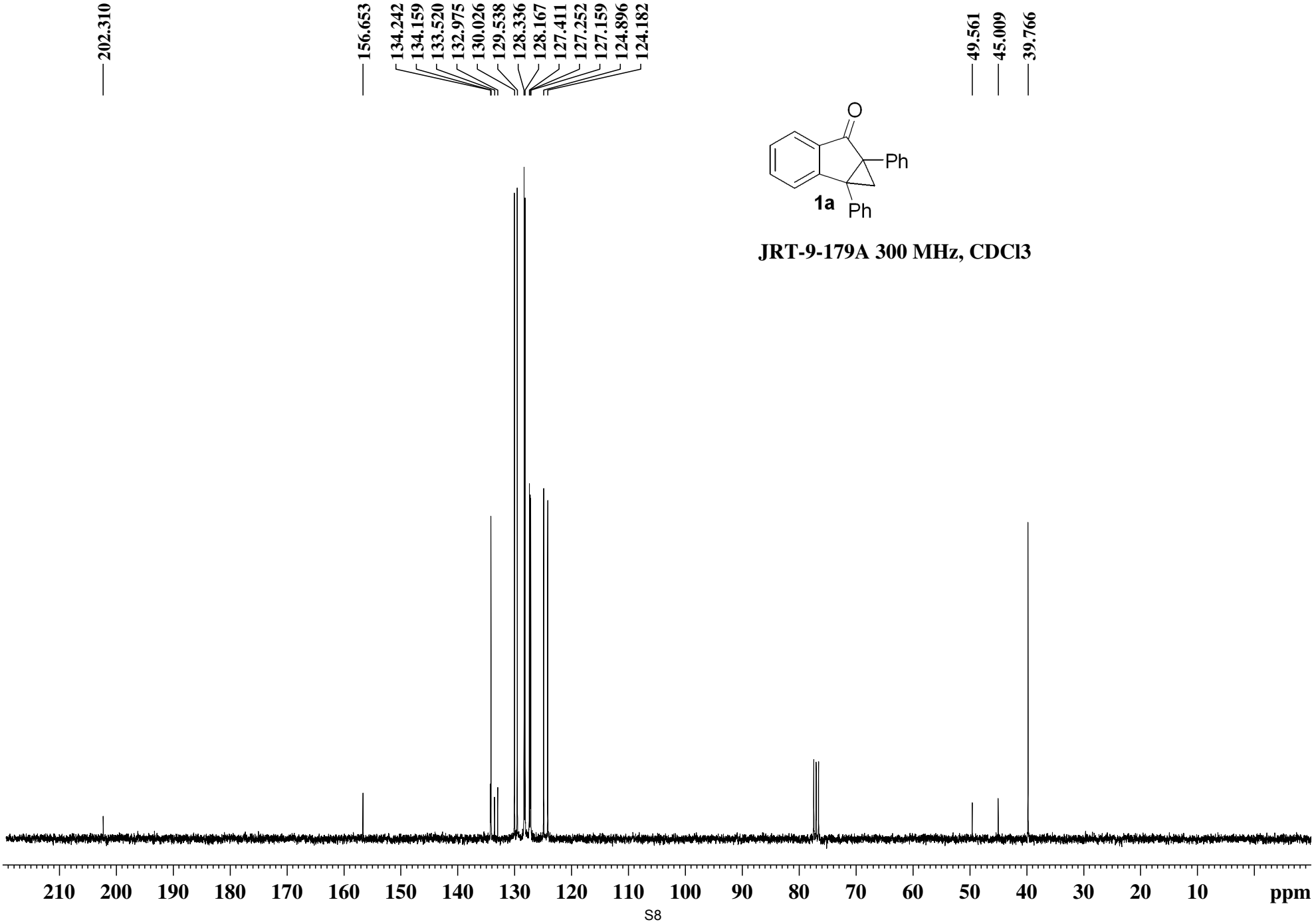
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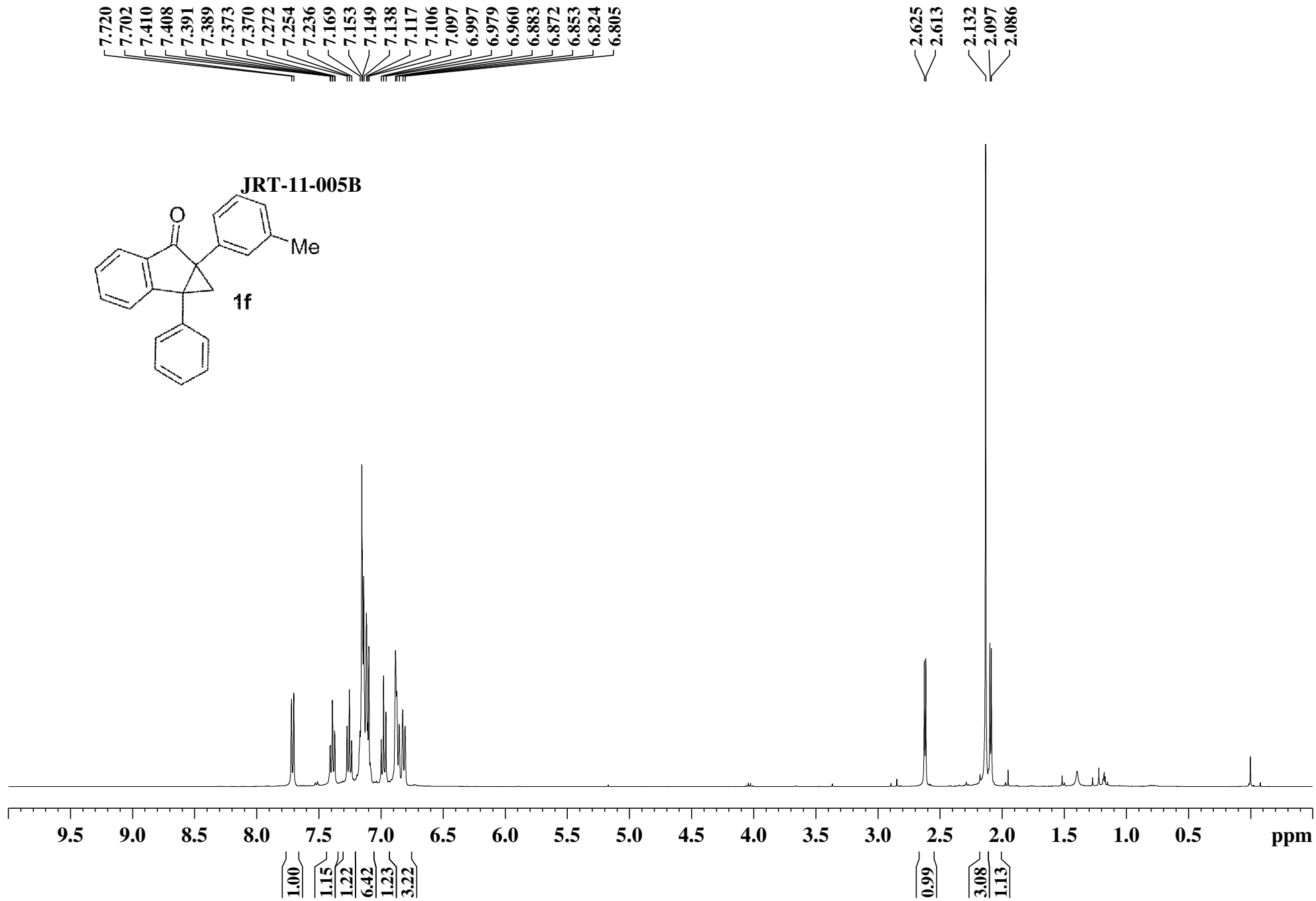
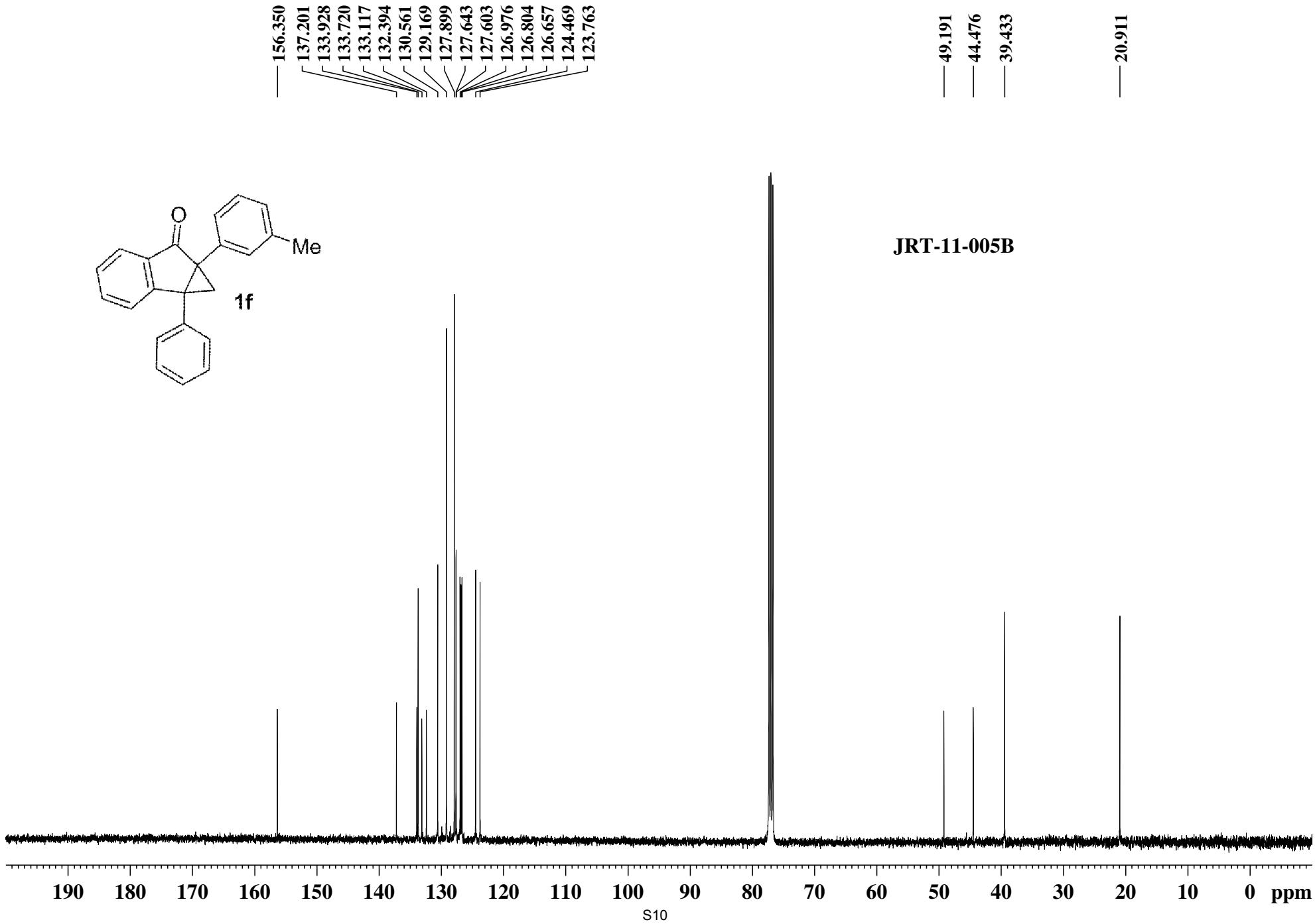
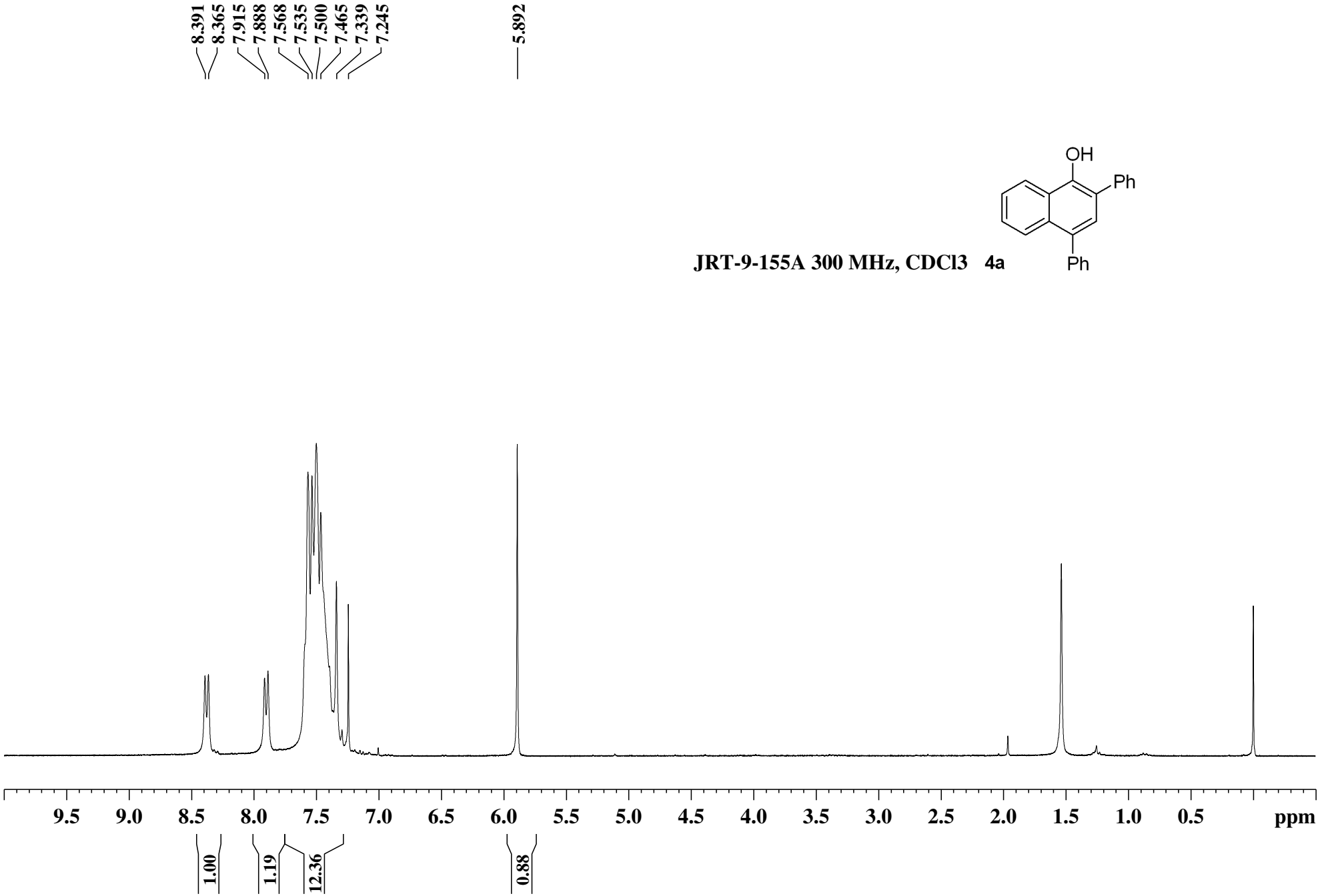
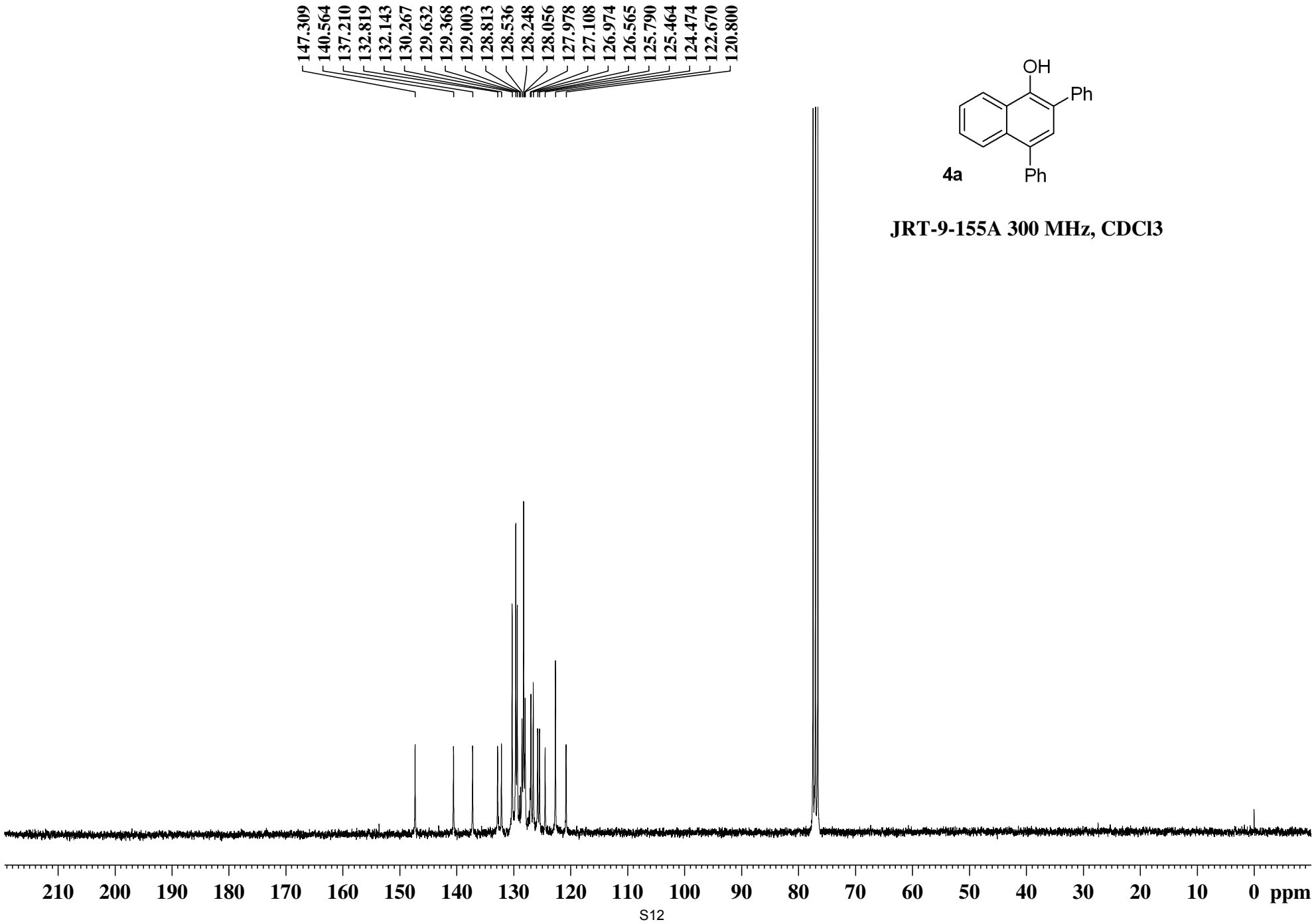
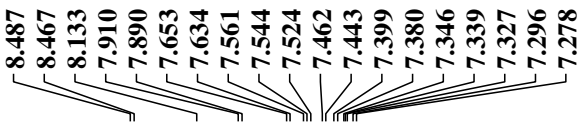
134.242
134.159
133.520
132.975
130.026
129.538
128.336
128.167
127.411
127.252
127.159
124.896
124.182
49.561
45.009
39.766
O
Ph
Ph
1a
JRT-9-179A 300 MHz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

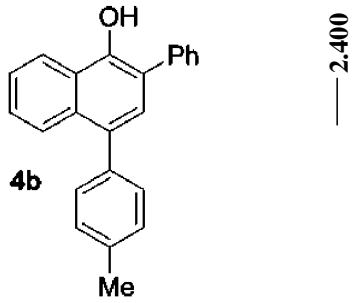

7.720
7.702
7.410
7.408
7.391
7.389
7.373
7.370
7.272
7.254
7.236
7.169
7.153
7.149
7.138
7.117
7.106
7.097
6.997
6.979
6.960
6.883
6.872
6.853
6.824
6.805
2.625
2.613
2.132
2.097
2.086
JRT-11-005B
O
Me
1f
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.00
1.15
1.22
6.42
1.23
3.22
0.99
3.08
1.13


156.350
137.201
133.928
133.720
133.117
132.394
130.561
129.169
127.899
127.643
127.603
126.976
126.804
126.657
124.469
123.763
49.191
44.476
39.433
20.911
O
Me
1f
JRT-11-005B
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

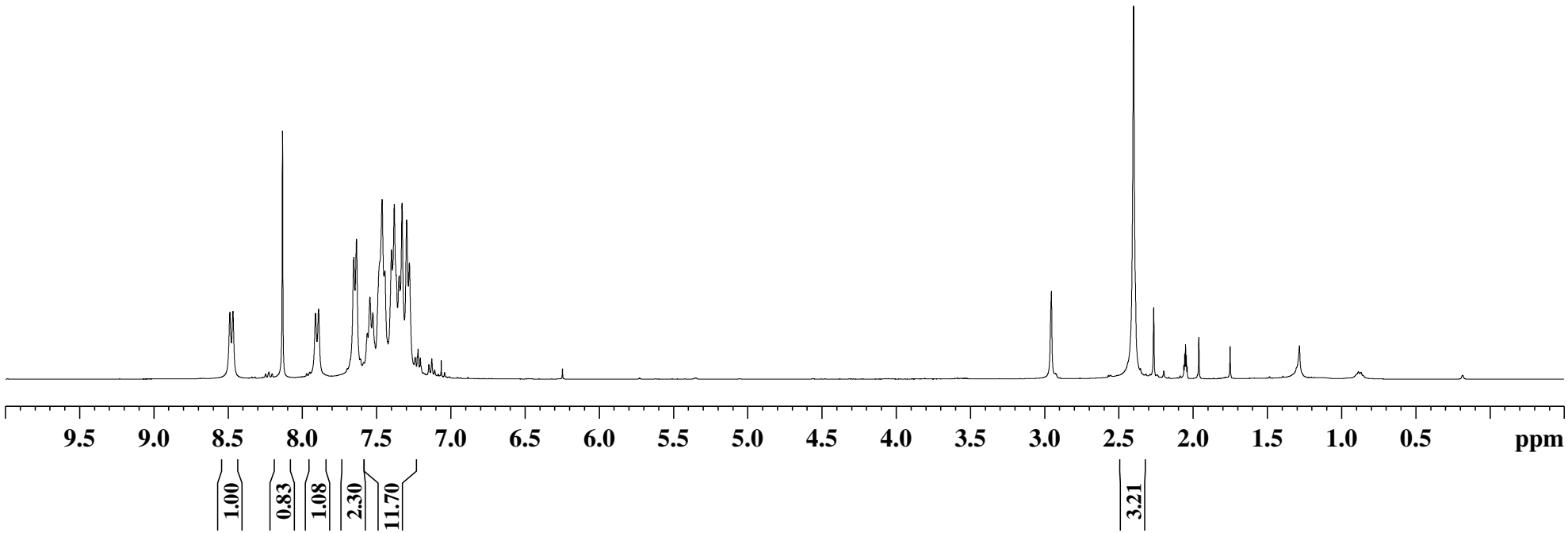

8.391
8.365
7.915
7.888
7.568
7.535
7.500
7.465
7.339
7.245
5.892
OH
Ph
Ph
JRT-9-155A 300 MHz, CDCl3 4a
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.00
1.19
12.36
0.88

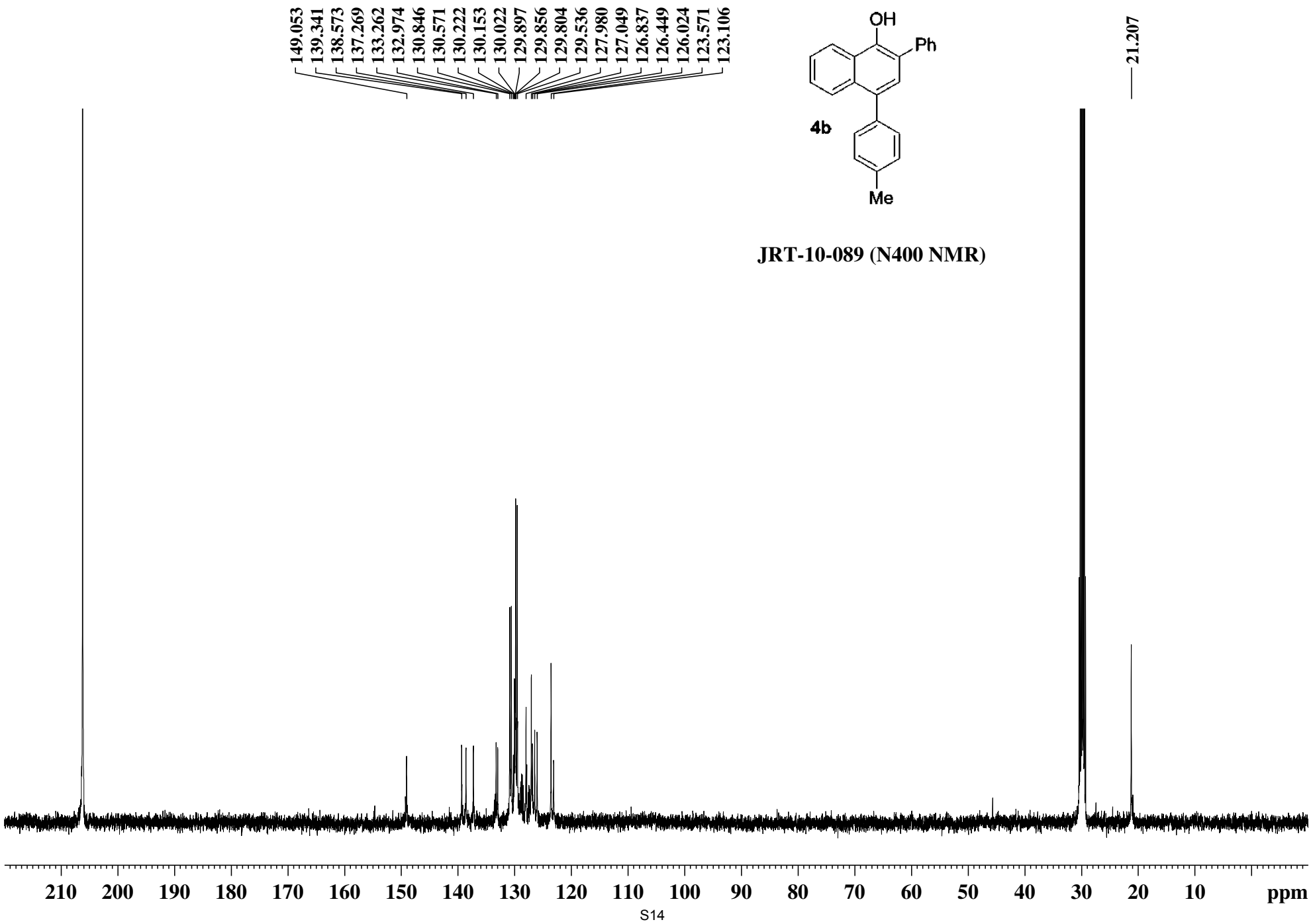

147.309
140.564
137.210
132.819
132.143
130.267
129.632
129.368
129.003
128.813
128.536
128.248
128.056
127.978
127.108
126.974
126.565
125.790
125.464
124.474
122.670
120.800
OH
Ph
Ph
4a
JRT-9-155A 300 MHz, CDCl3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

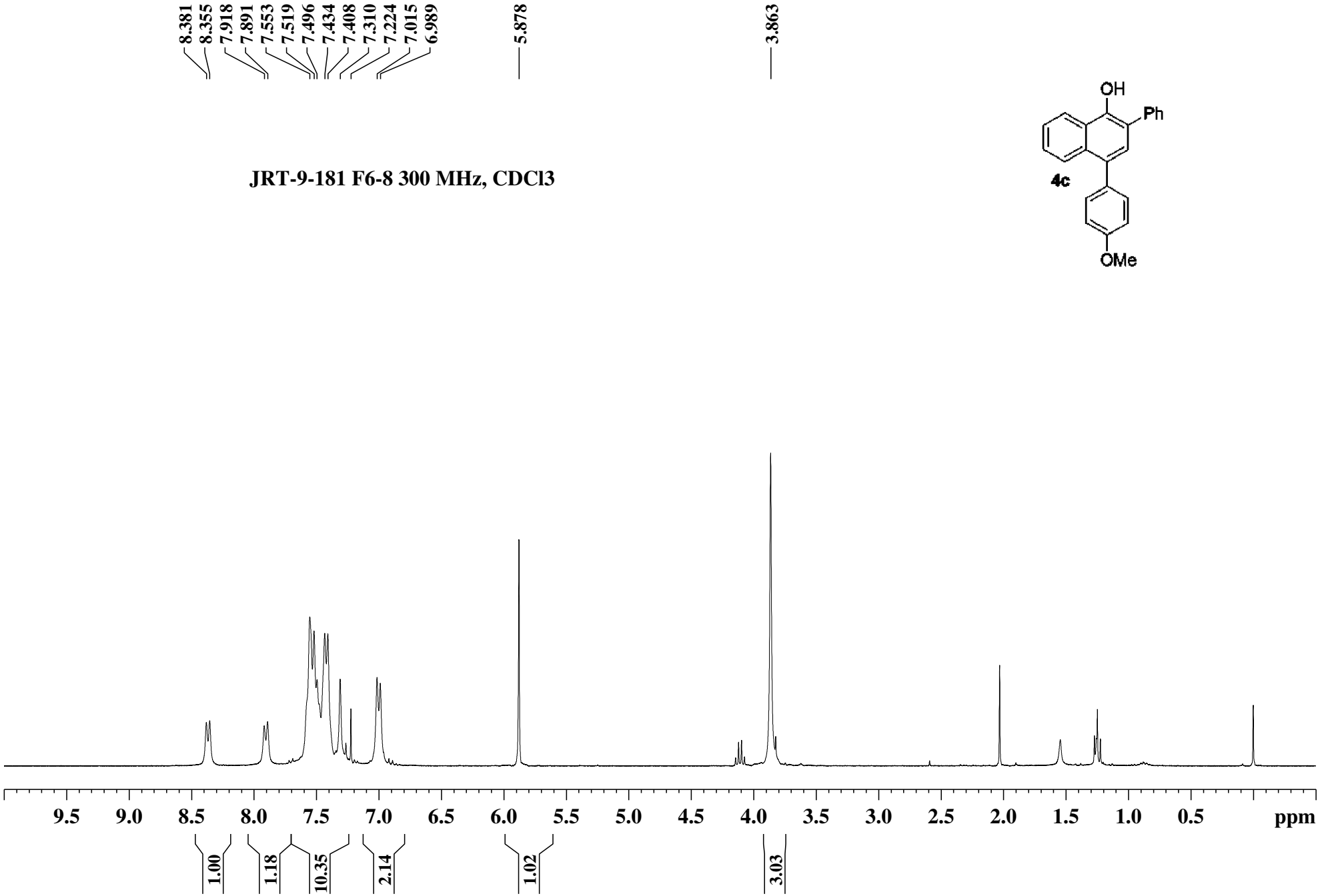


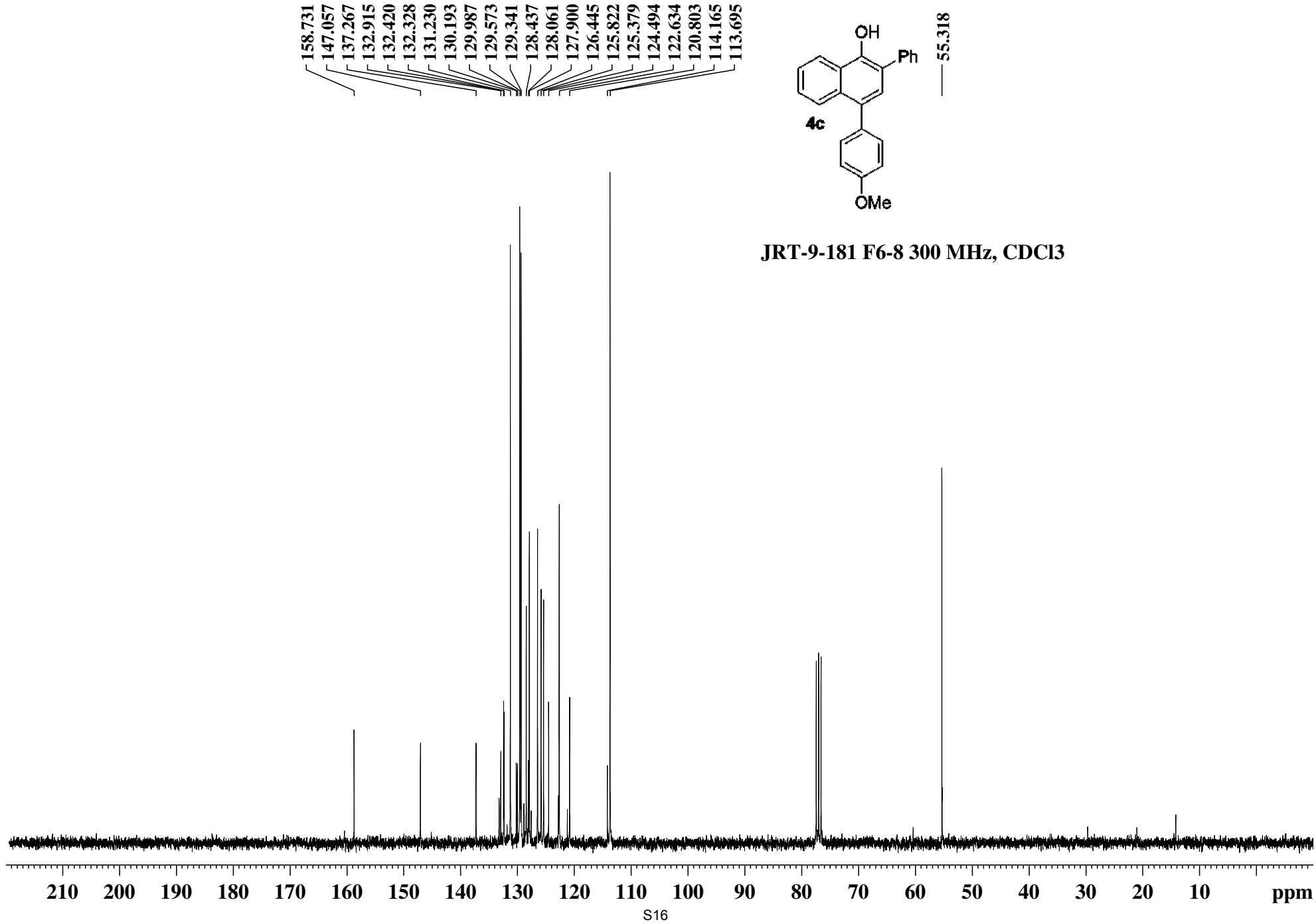


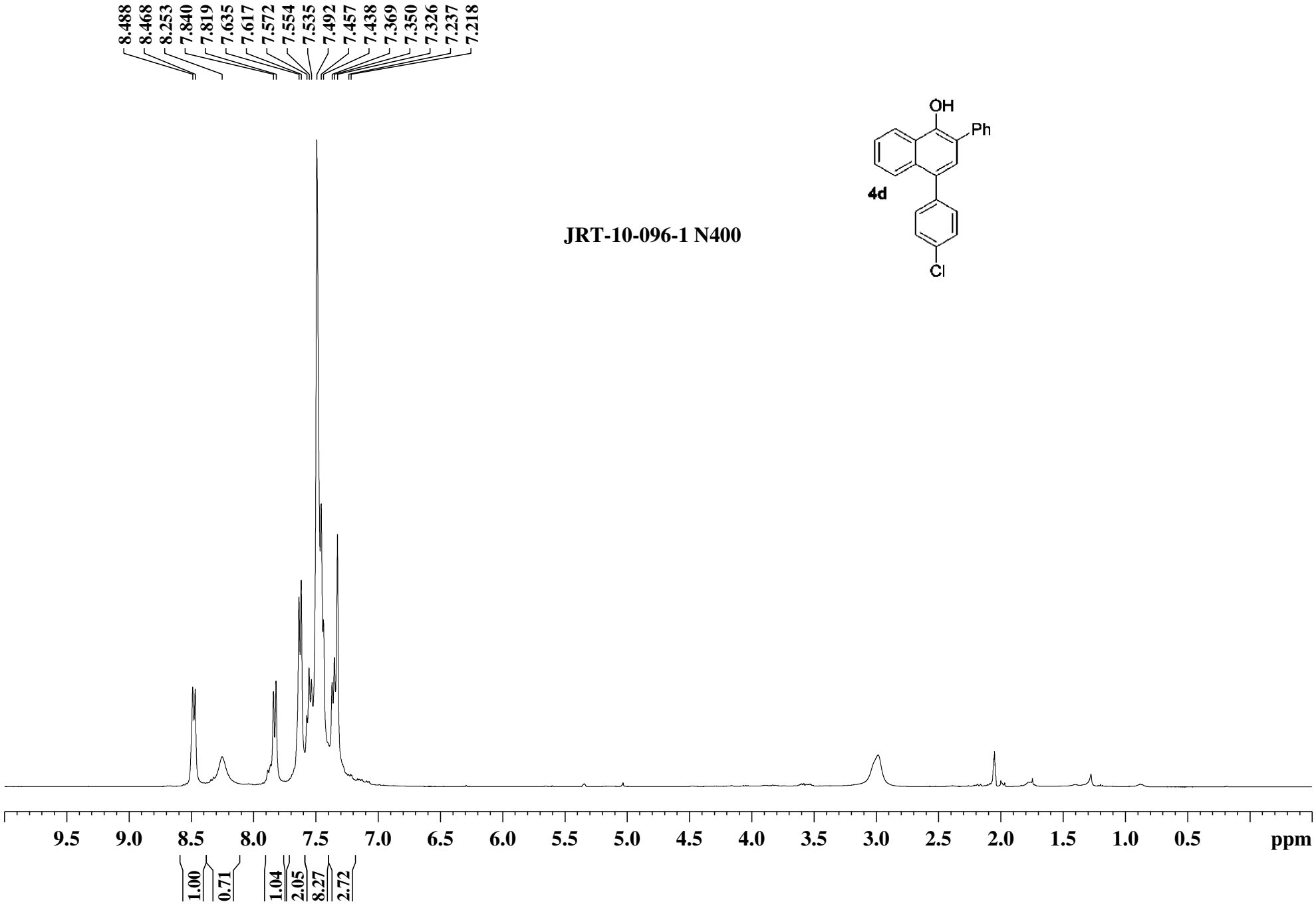
JRT-10-089 (N400)

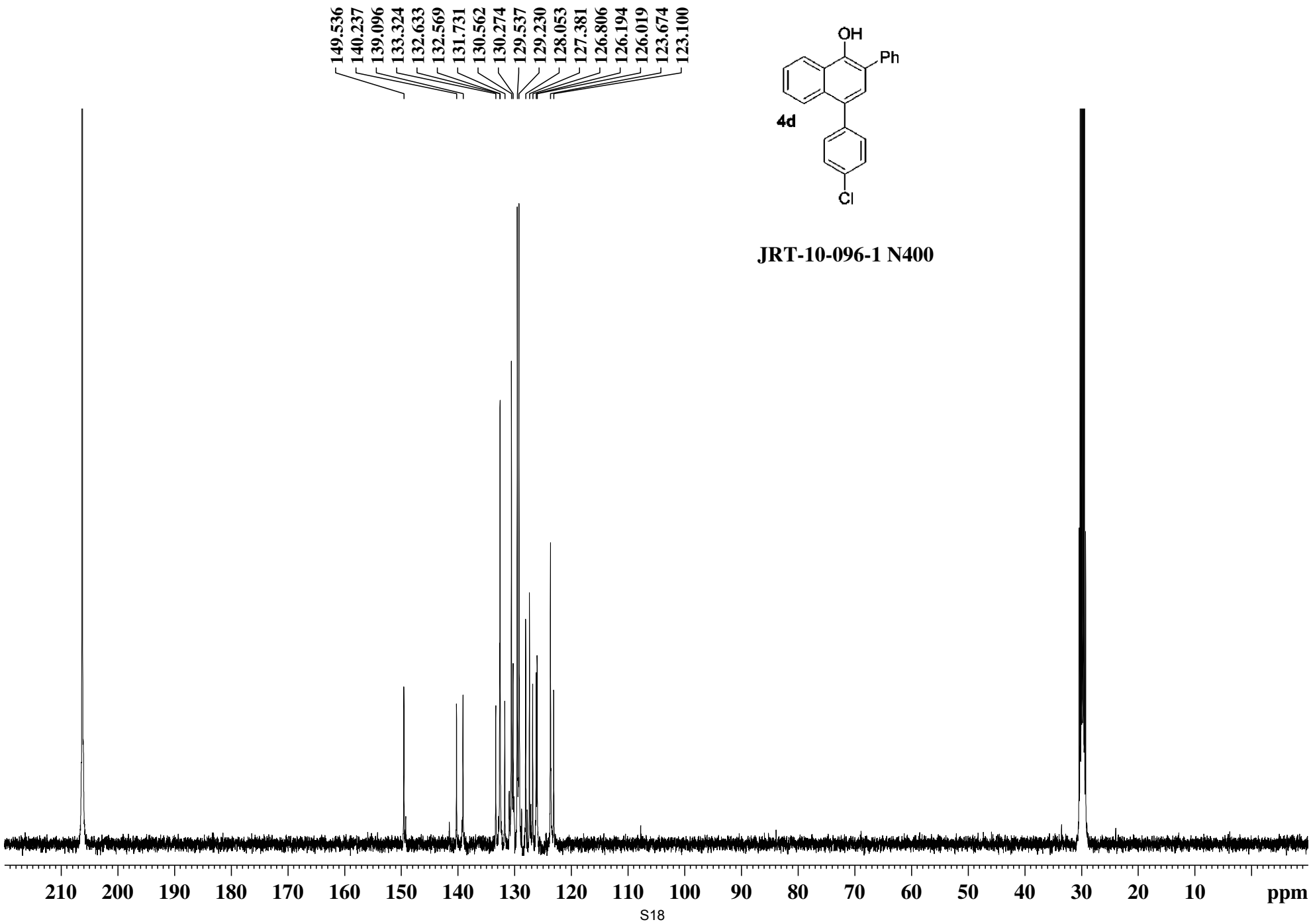


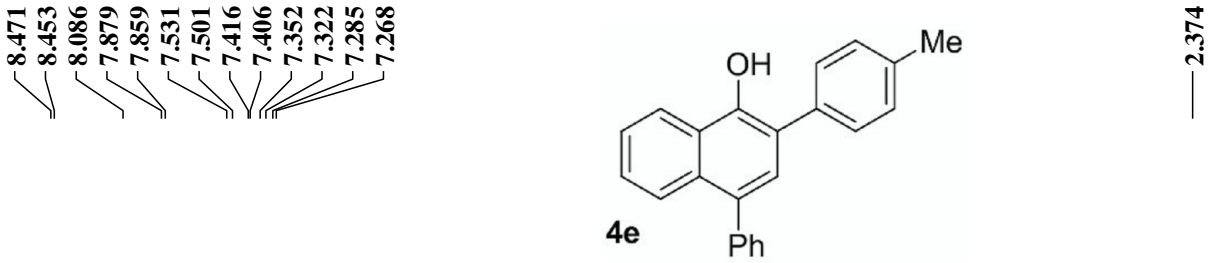

149.053
139.341
138.573
137.269
133.262
132.974
130.846
130.571
130.222
130.153
130.022
129.897
129.856
129.804
129.536
127.980
127.049
126.837
126.449
126.024
123.571
123.106
21.207
OH
Ph
Me
4b
JRT-10-089 (N400 NMR)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


8.381
8.355
7.918
7.891
7.553
7.519
7.496
7.434
7.408
7.310
7.224
7.015
6.989
5.878
3.863
JRT-9-181 F6-8 300 MHz, CDCl3
OH
Ph
OMe
4c
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.00
1.18
10.35
2.14
1.02
3.03

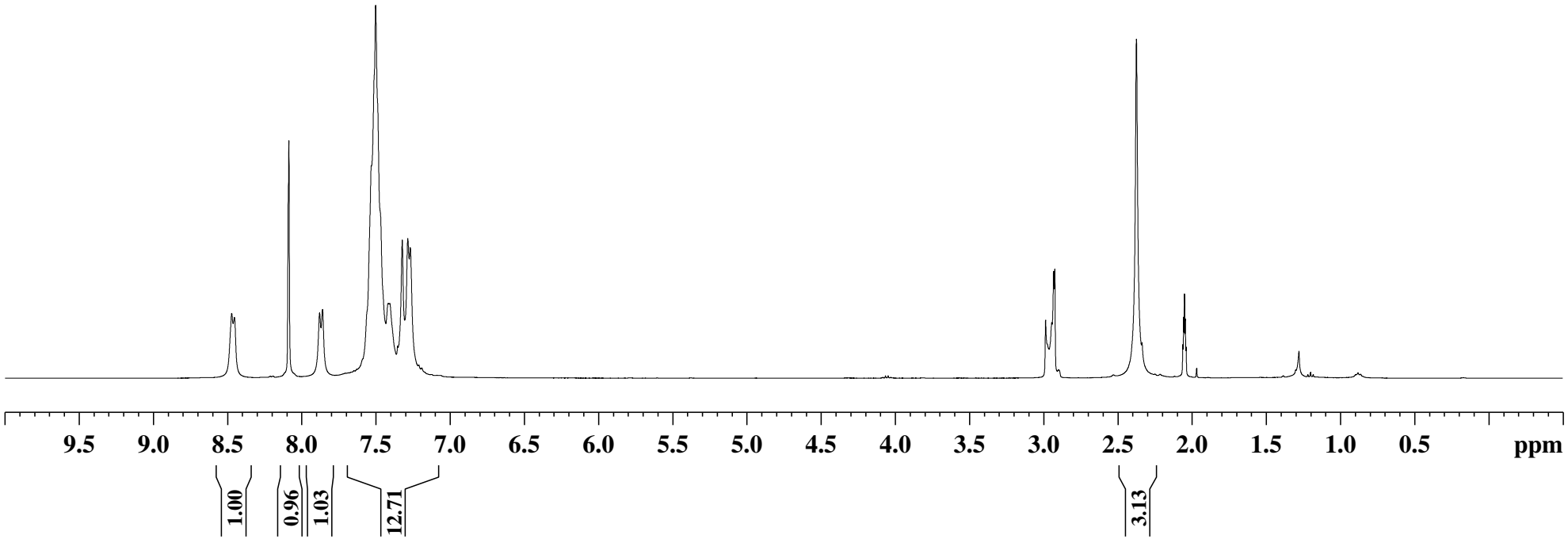

158.731
147.057
137.267
132.915
132.420
132.328
131.230
130.193
129.987
129.573
129.341
128.437
128.061
127.900
126.445
125.822
125.379
124.494
122.634
120.803
114.165
113.695
55.318
OH
Ph
OMe
4c
JRT-9-181 F6-8 300 MHz, CDCl3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

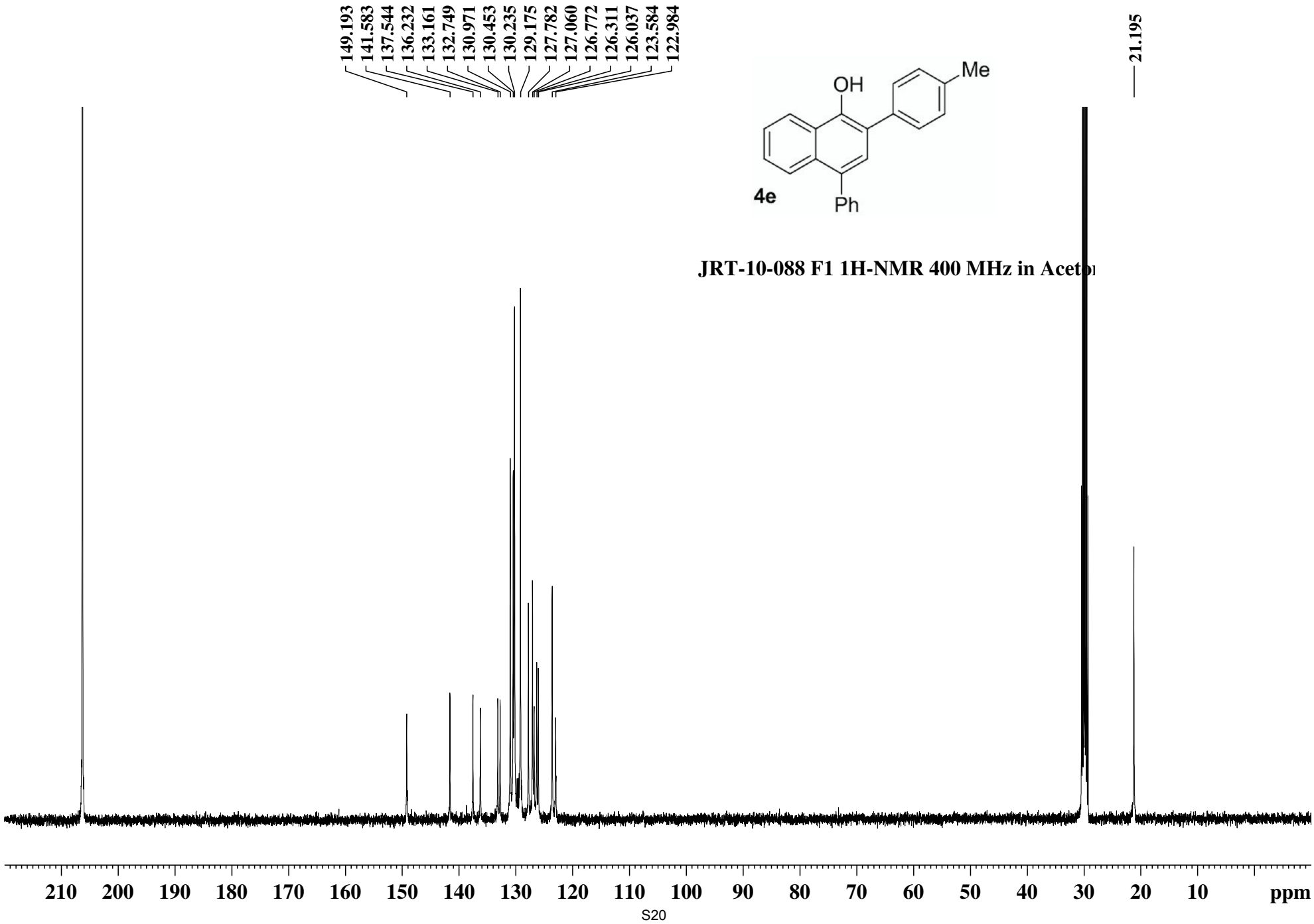

8.488
8.468
8.253
7.840
7.819
7.635
7.617
7.572
7.554
7.535
7.492
7.457
7.438
7.369
7.350
7.326
7.237
7.218
JRT-10-096-1 N400
OH
Ph
4d
Cl
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.00
0.71
1.04
2.05
8.27
2.72

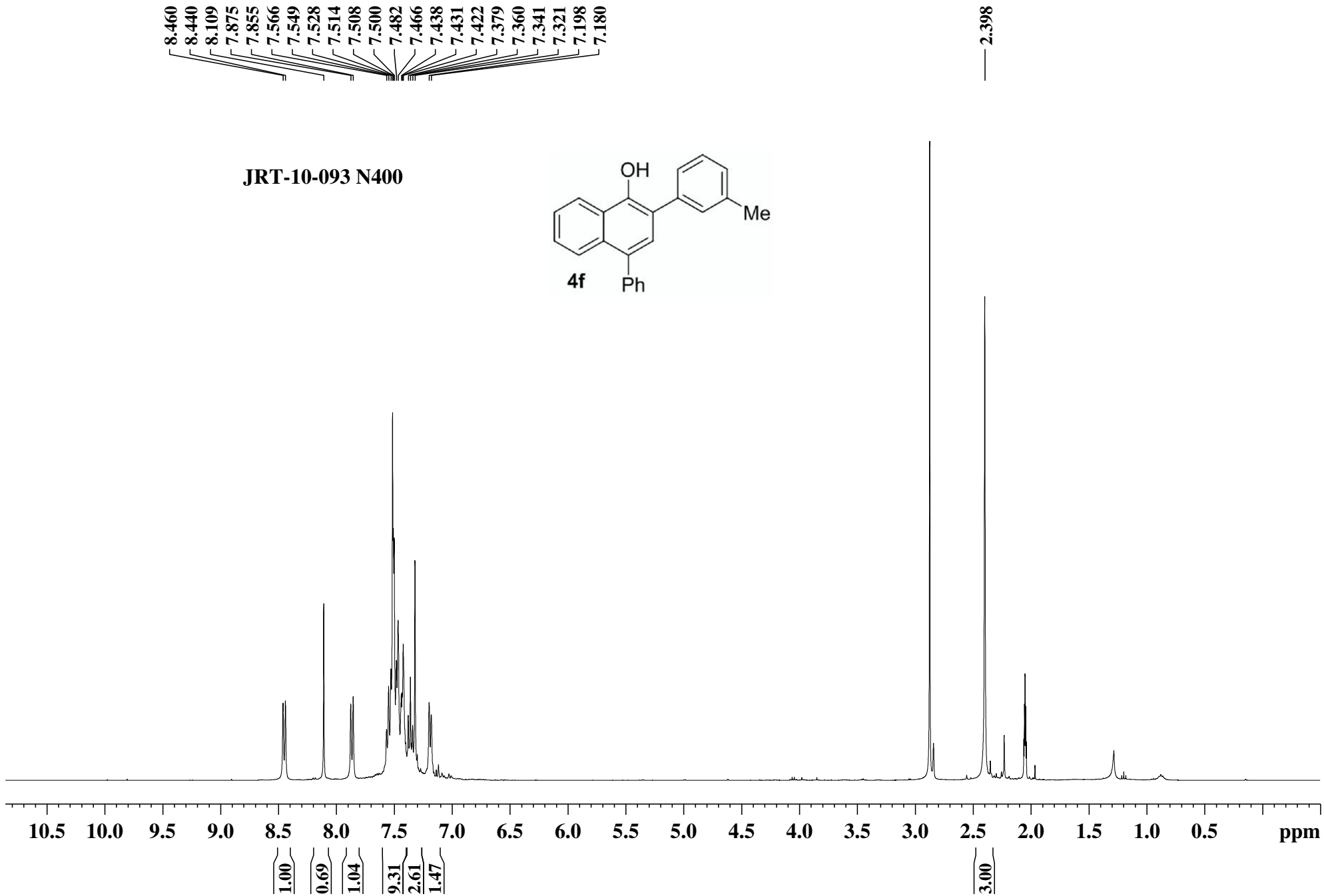

149.536
140.237
139.096
133.324
132.633
132.569
131.731
130.562
130.274
129.537
129.230
128.053
127.381
126.806
126.194
126.019
123.674
123.100
OH
Ph
4d
Cl
JRT-10-096-1 N400
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

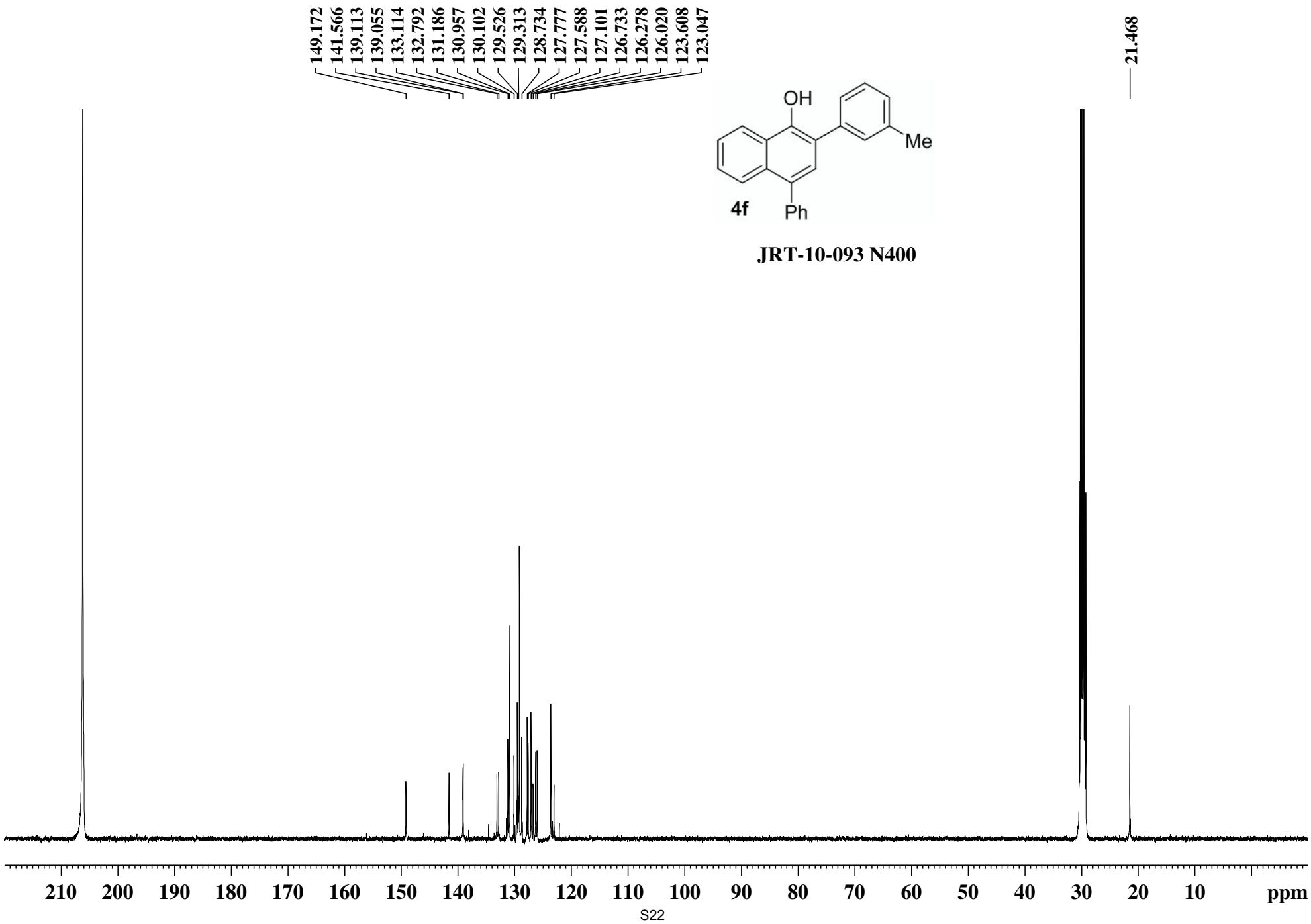


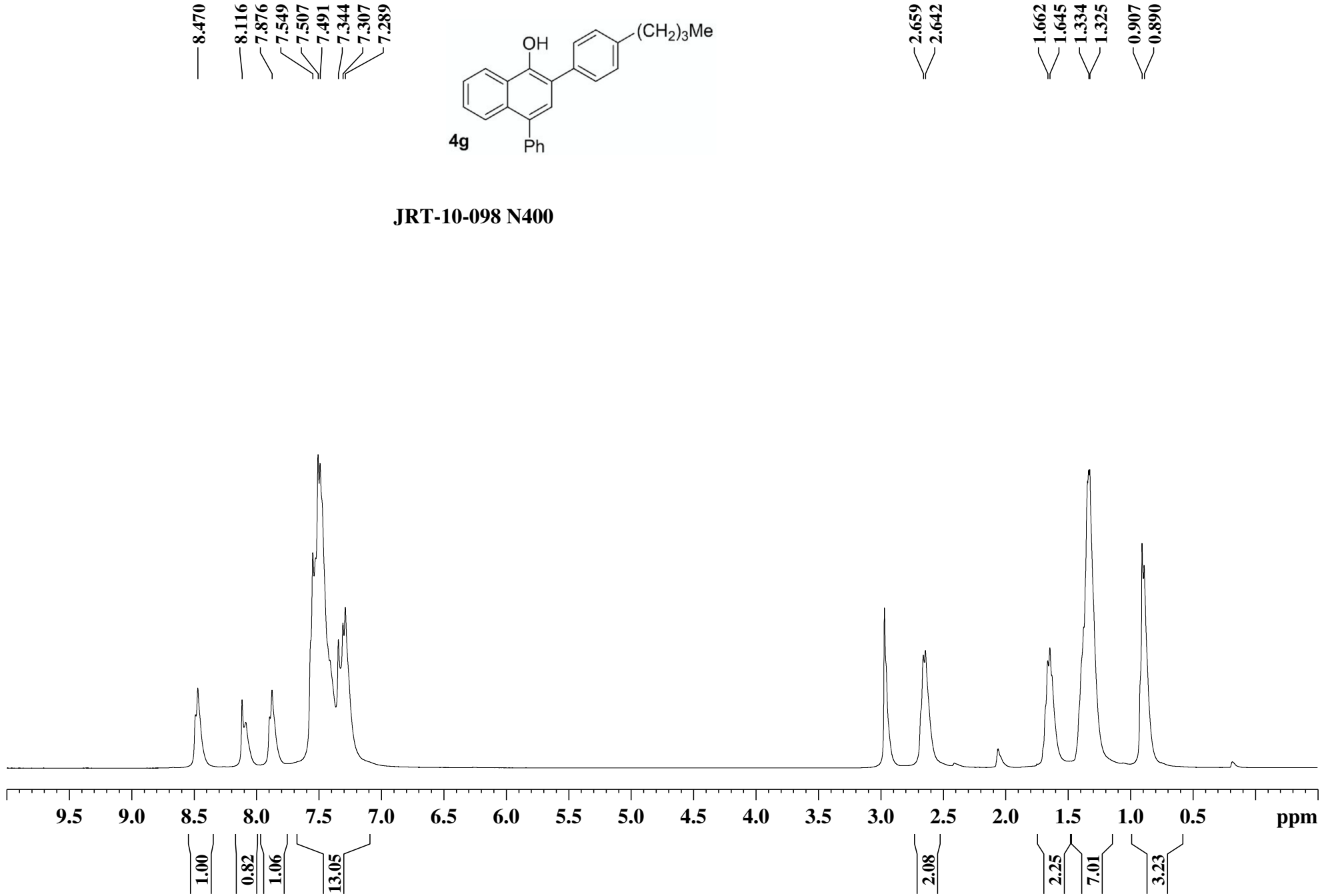
JRT-10-088 F1 1H-NMR 400 MHz in Ac



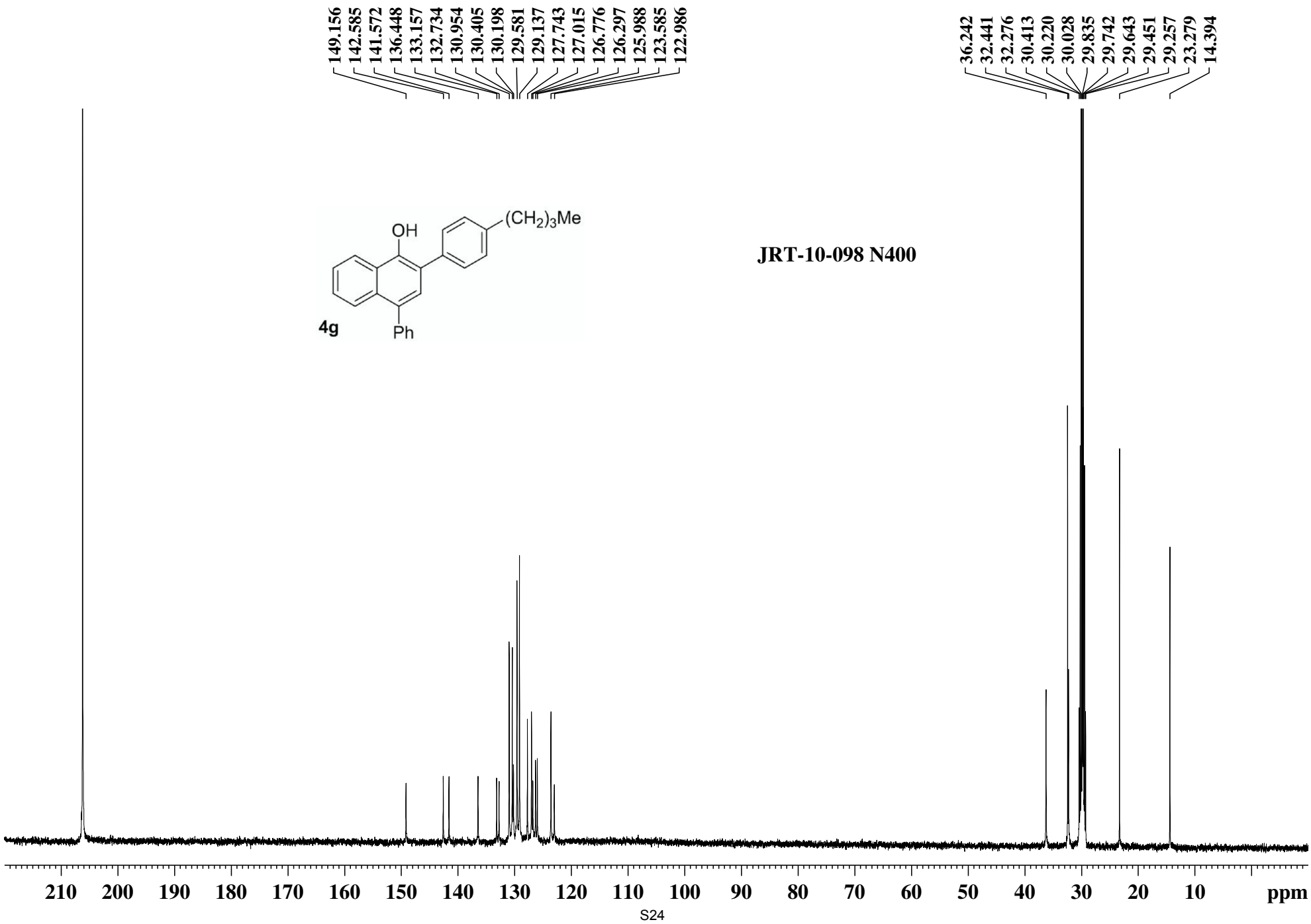

149.193
141.583
137.544
136.232
133.161
132.749
130.971
130.453
130.235
129.175
127.782
127.060
126.772
126.311
126.037
123.584
122.984
21.195
OH
Me
Ph
4e
JRT-10-088 F1 1H-NMR 400 MHz in Aceto
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

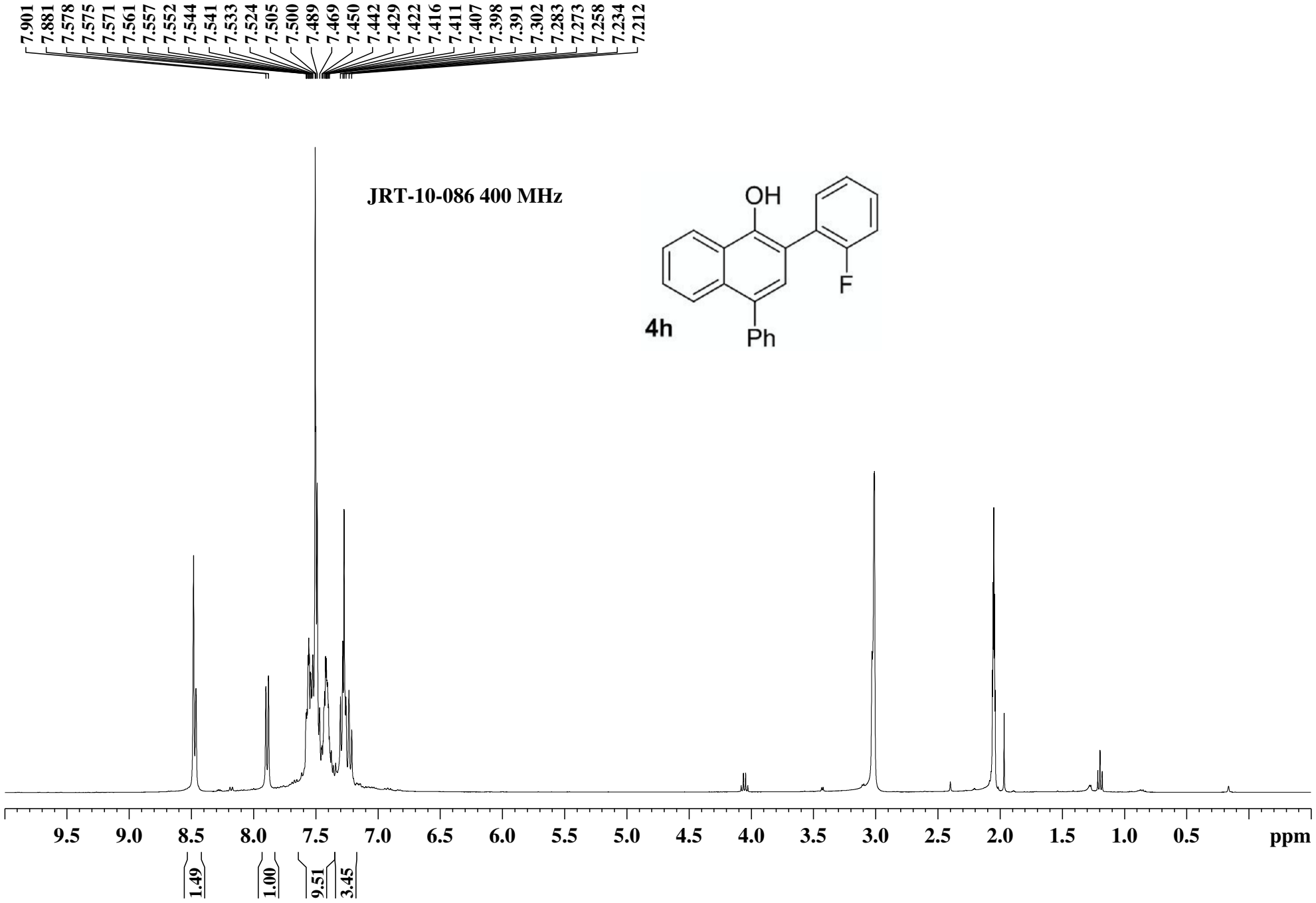

8.460
8.440
8.109
7.875
7.855
7.566
7.549
7.528
7.514
7.508
7.500
7.482
7.466
7.438
7.431
7.422
7.379
7.360
7.341
7.321
7.198
7.180
2.398
JRT-10-093 N400
OH
Me
4f
Ph
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
0.69
1.04
9.31
2.61
1.47
3.00

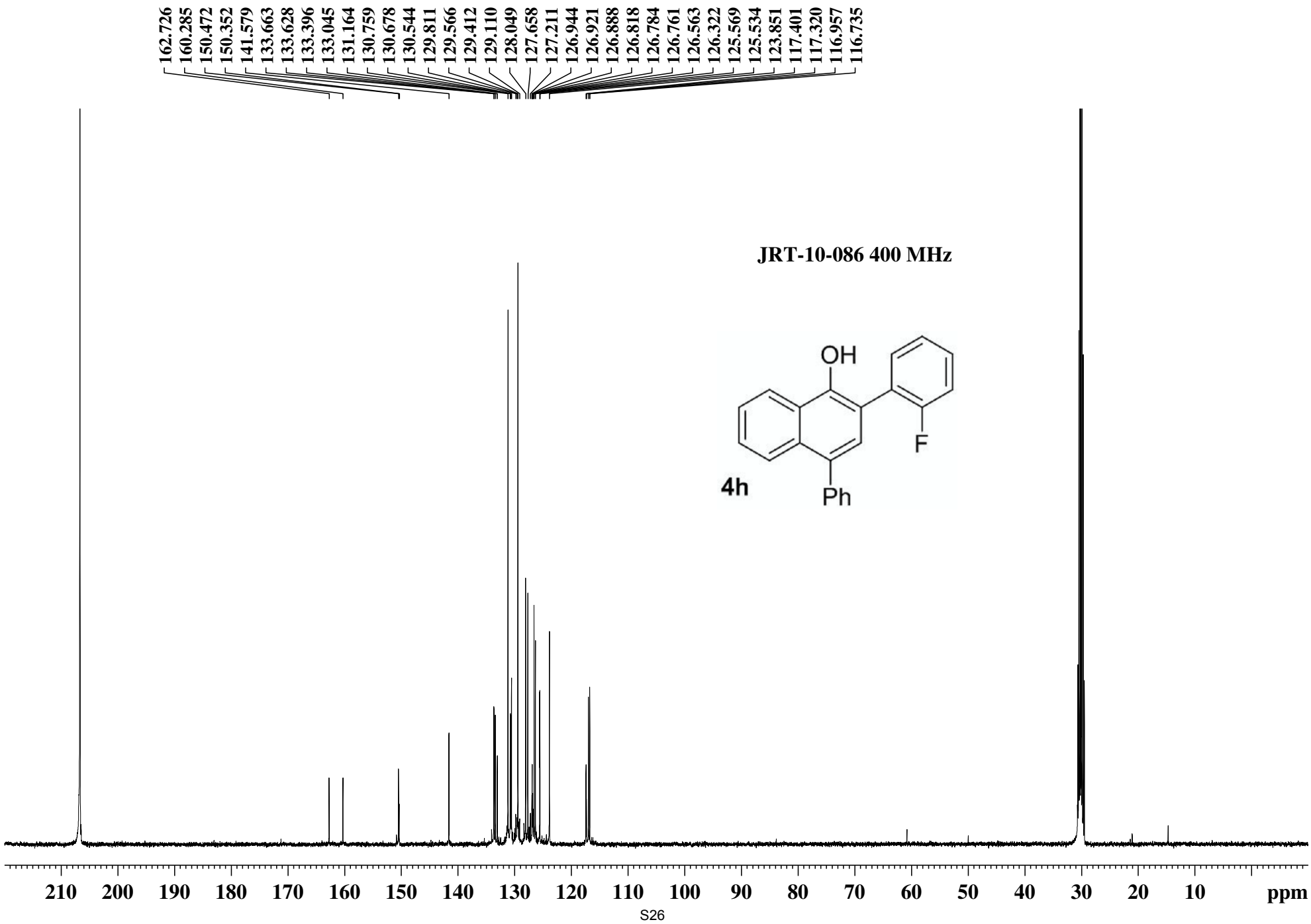

149.172
141.566
139.113
139.055
133.114
132.792
131.186
130.957
130.102
129.526
129.313
128.734
127.777
127.588
127.101
126.733
126.278
126.020
123.608
123.047
21.468
OH
Me
4f
Ph
JRT-10-093 N400
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

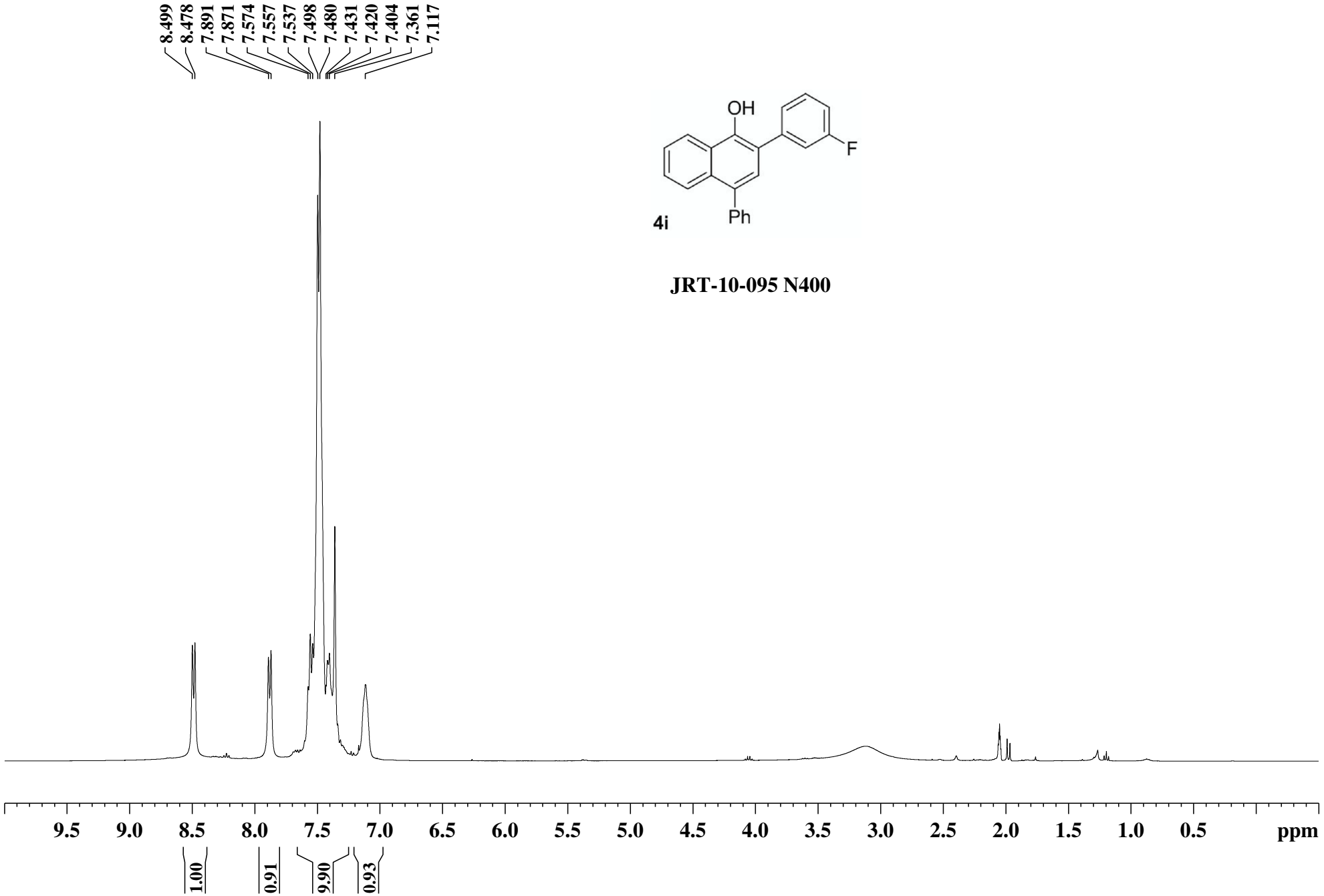


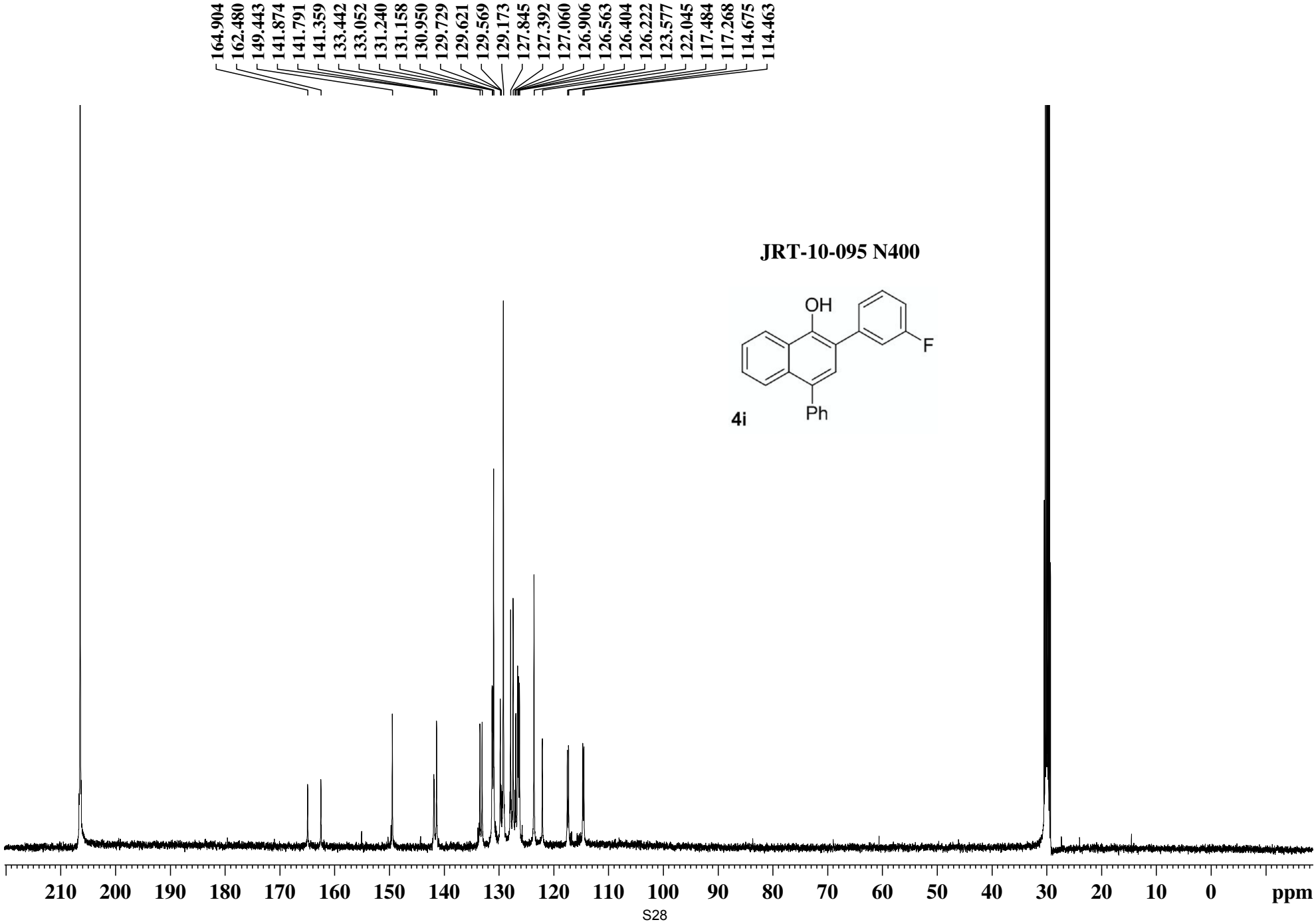
JRT-10-098 N400

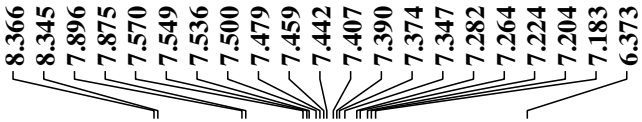

149.156
142.585
141.572
136.448
133.157
132.734
130.954
130.405
130.198
129.581
129.137
127.743
127.015
126.776
126.297
125.988
123.585
122.986
36.242
32.441
32.276
30.413
30.220
30.028
29.835
29.742
29.643
29.451
29.257
23.279
14.394
OH
(CH2)3Me
Ph
4g
JRT-10-098 N400
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

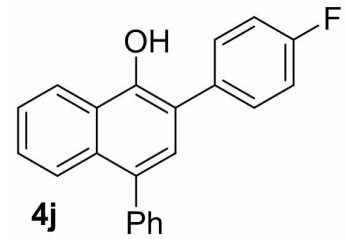

7.901
7.881
7.578
7.575
7.571
7.561
7.557
7.552
7.544
7.541
7.533
7.524
7.505
7.500
7.489
7.469
7.450
7.442
7.429
7.422
7.416
7.411
7.407
7.398
7.391
7.302
7.283
7.273
7.258
7.234
7.212
JRT-10-086 400 MHz
OH
F
4h
Ph
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.49
1.00
9.51
3.45


162.726
160.285
150.472
150.352
141.579
133.663
133.628
133.396
133.045
131.164
130.759
130.678
130.544
129.811
129.566
129.412
129.110
128.049
127.658
127.211
126.944
126.921
126.888
126.818
126.784
126.761
126.563
126.322
125.569
125.534
123.851
117.401
117.320
116.957
116.735
JRT-10-086 400 MHz
OH
F
Ph
4h
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

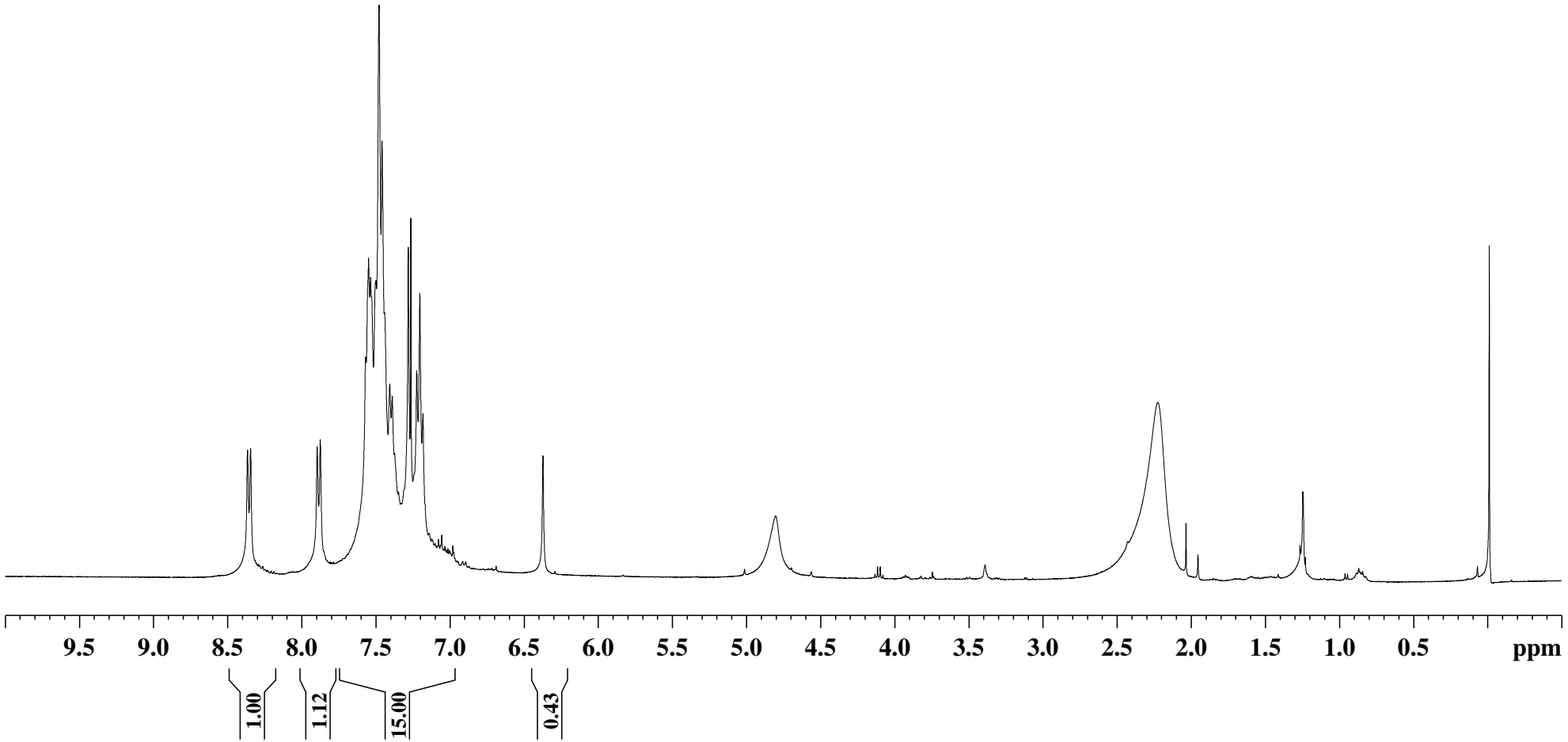

8.499
8.478
7.891
7.871
7.574
7.557
7.537
7.498
7.480
7.431
7.420
7.404
7.361
7.117
OH
F
Ph
4i
JRT-10-095 N400
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.00
0.91
9.90
0.93

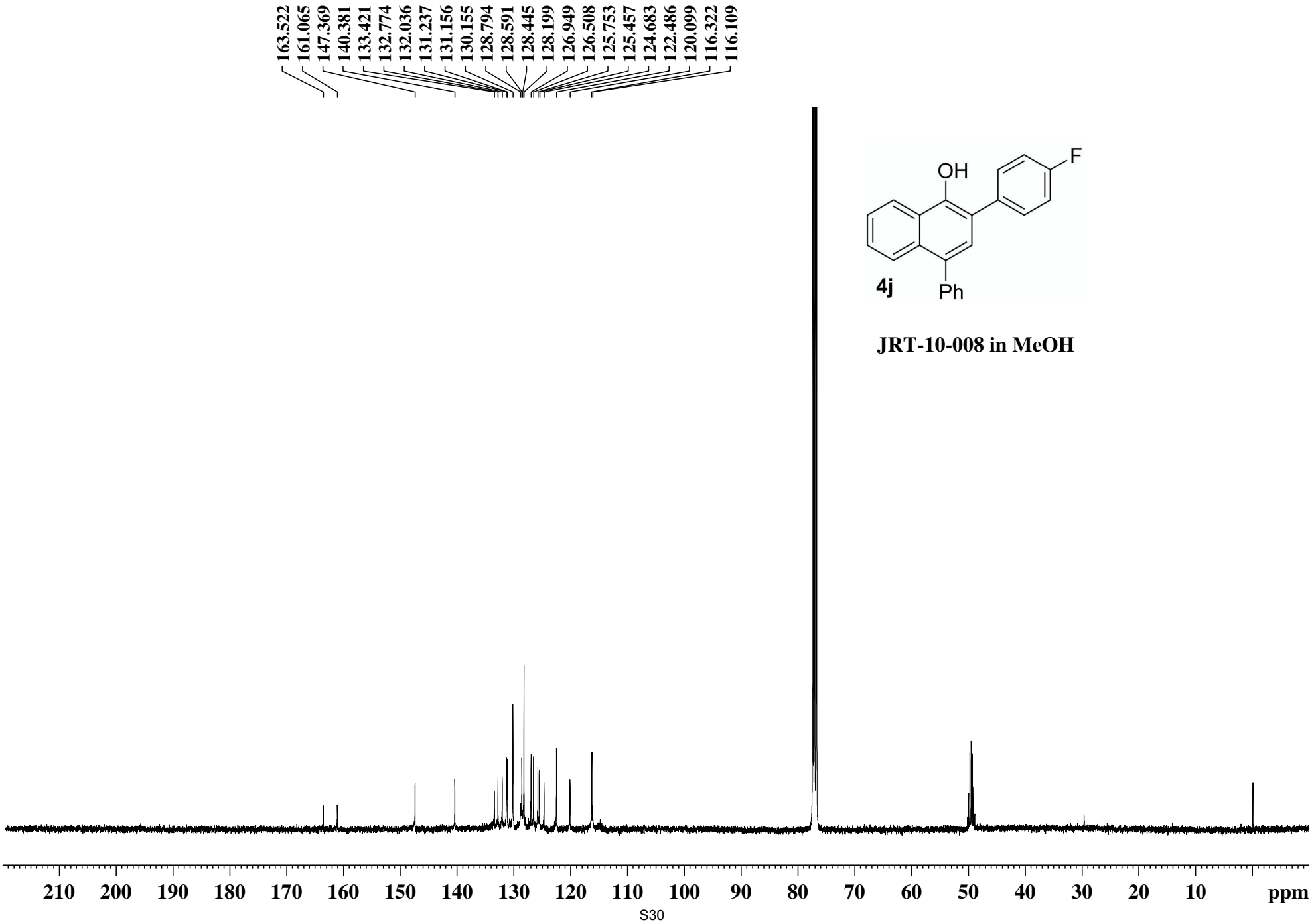

164.904
162.480
149.443
141.874
141.791
141.359
133.442
133.052
131.240
131.158
130.950
129.729
129.621
129.569
129.173
127.845
127.392
127.060
126.906
126.563
126.404
126.222
123.577
122.045
117.484
117.268
114.675
114.463
JRT-10-095 N400
OH
F
Ph
4i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

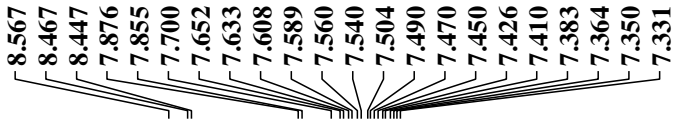




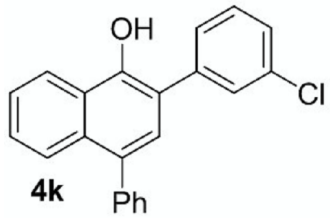
JRT-10-008 in MeOH



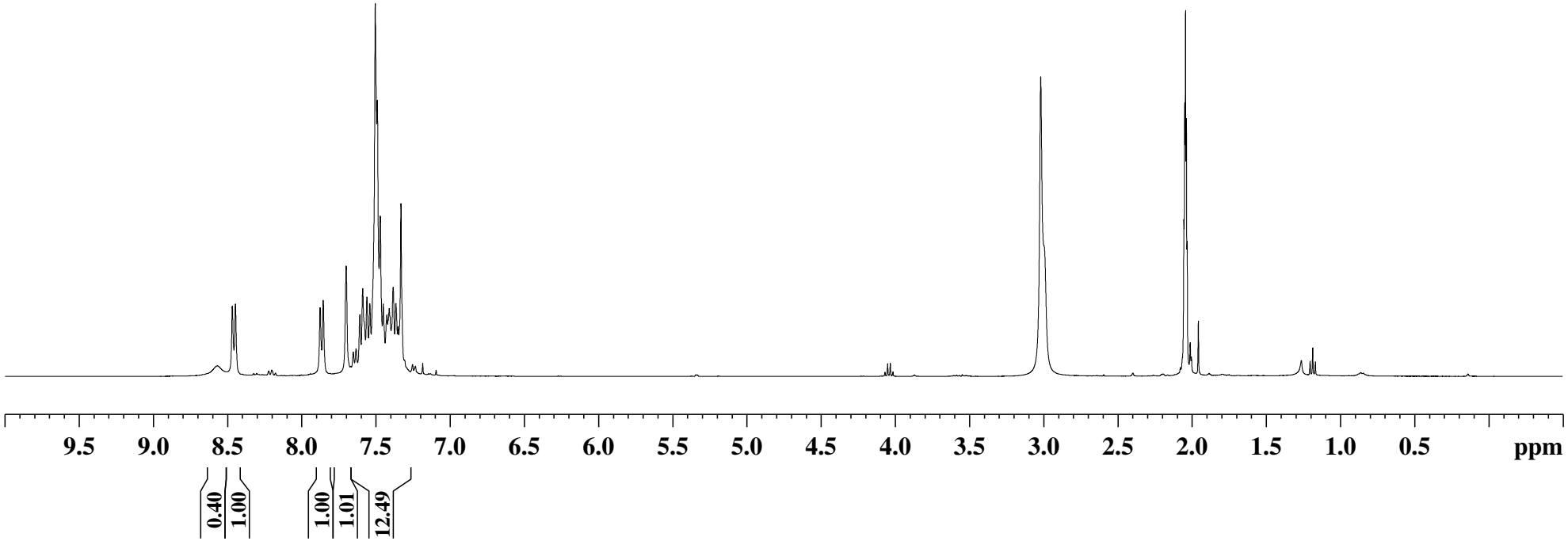

163.522
161.065
147.369
140.381
133.421
132.774
132.036
131.237
131.156
130.155
128.794
128.591
128.445
128.199
126.949
126.508
125.753
125.457
124.683
122.486
120.099
116.322
116.109
OH
F
Ph
4j
JRT-10-008 in MeOH
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

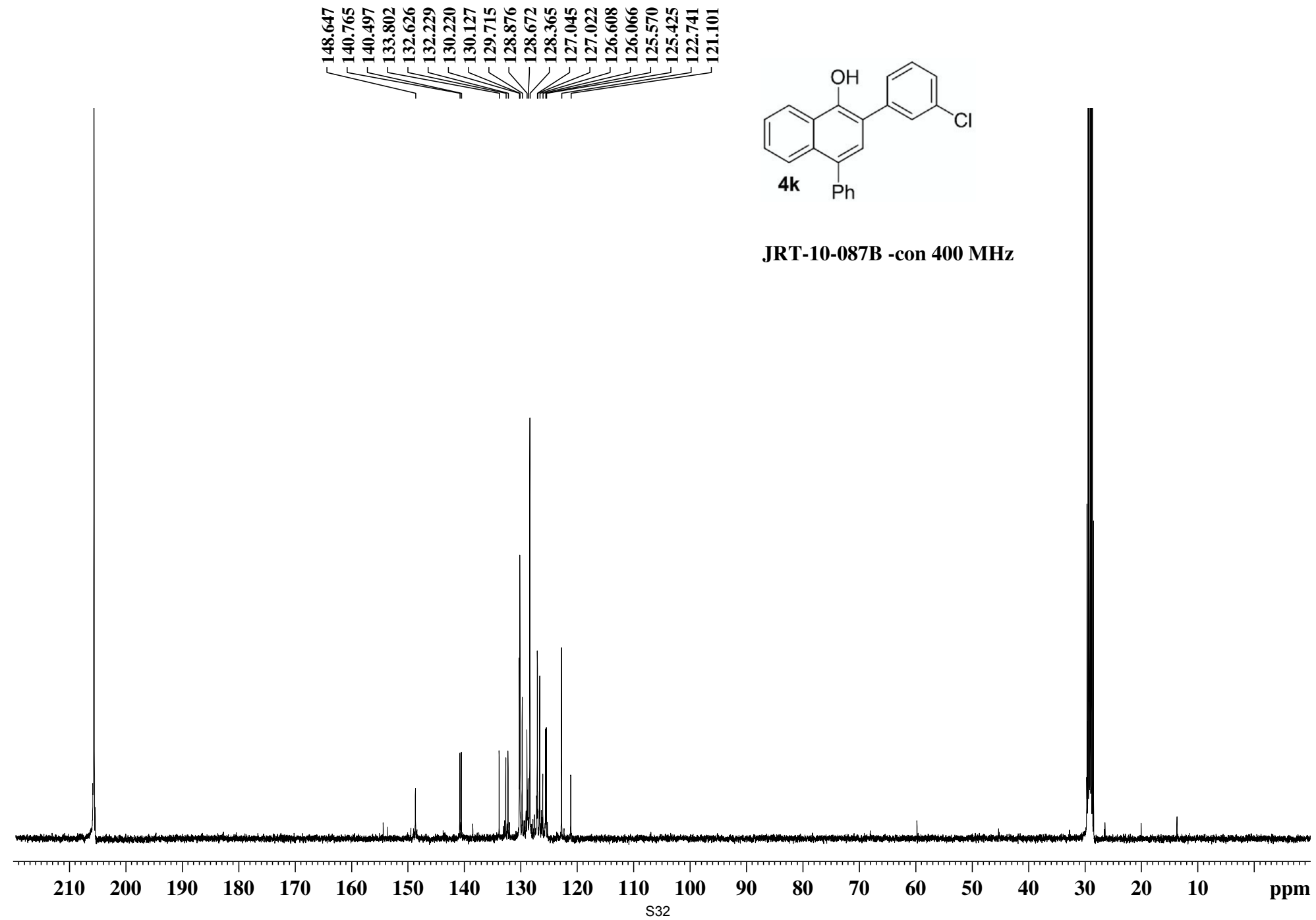


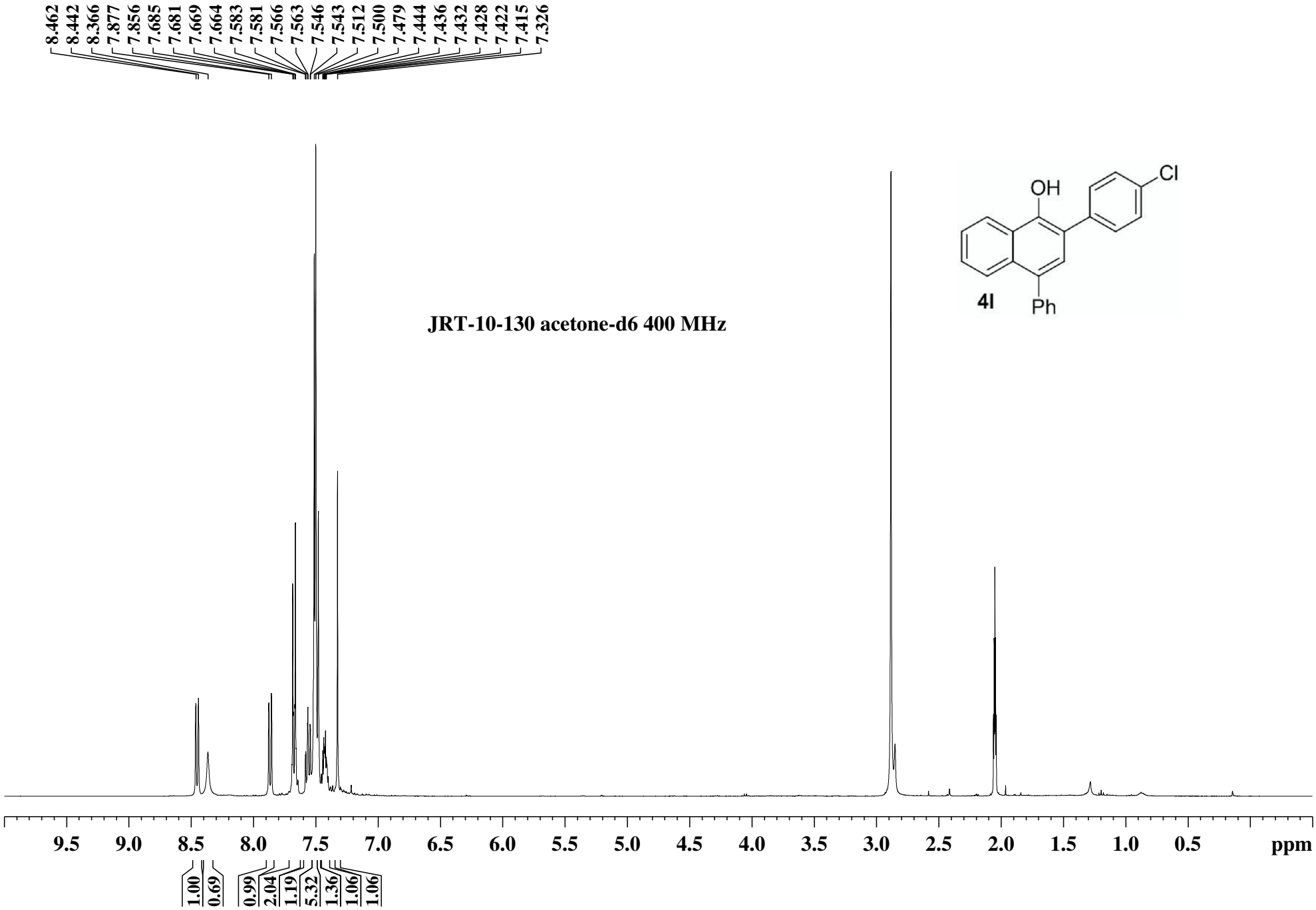
JRT-10-087B -con 400 MHz

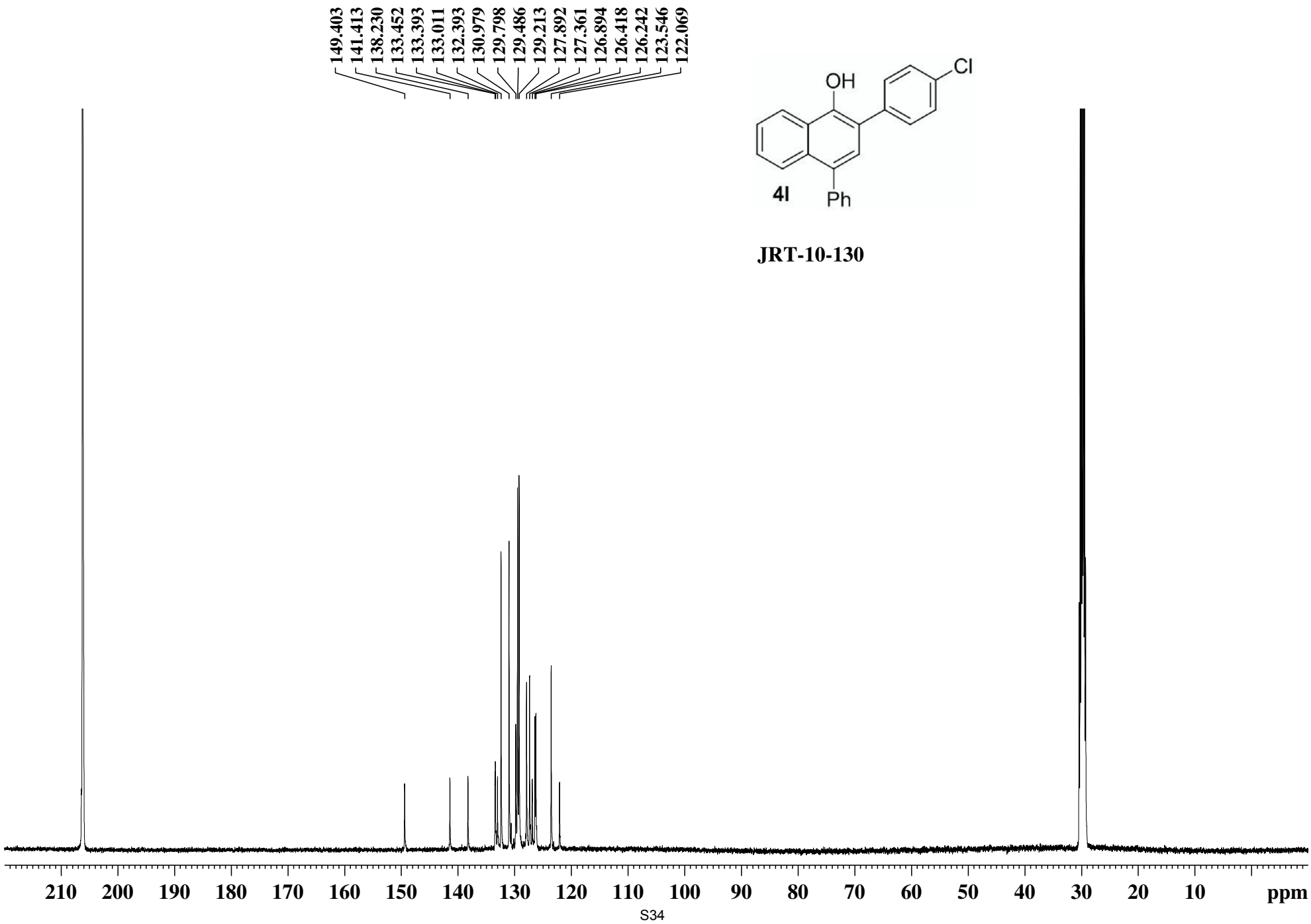


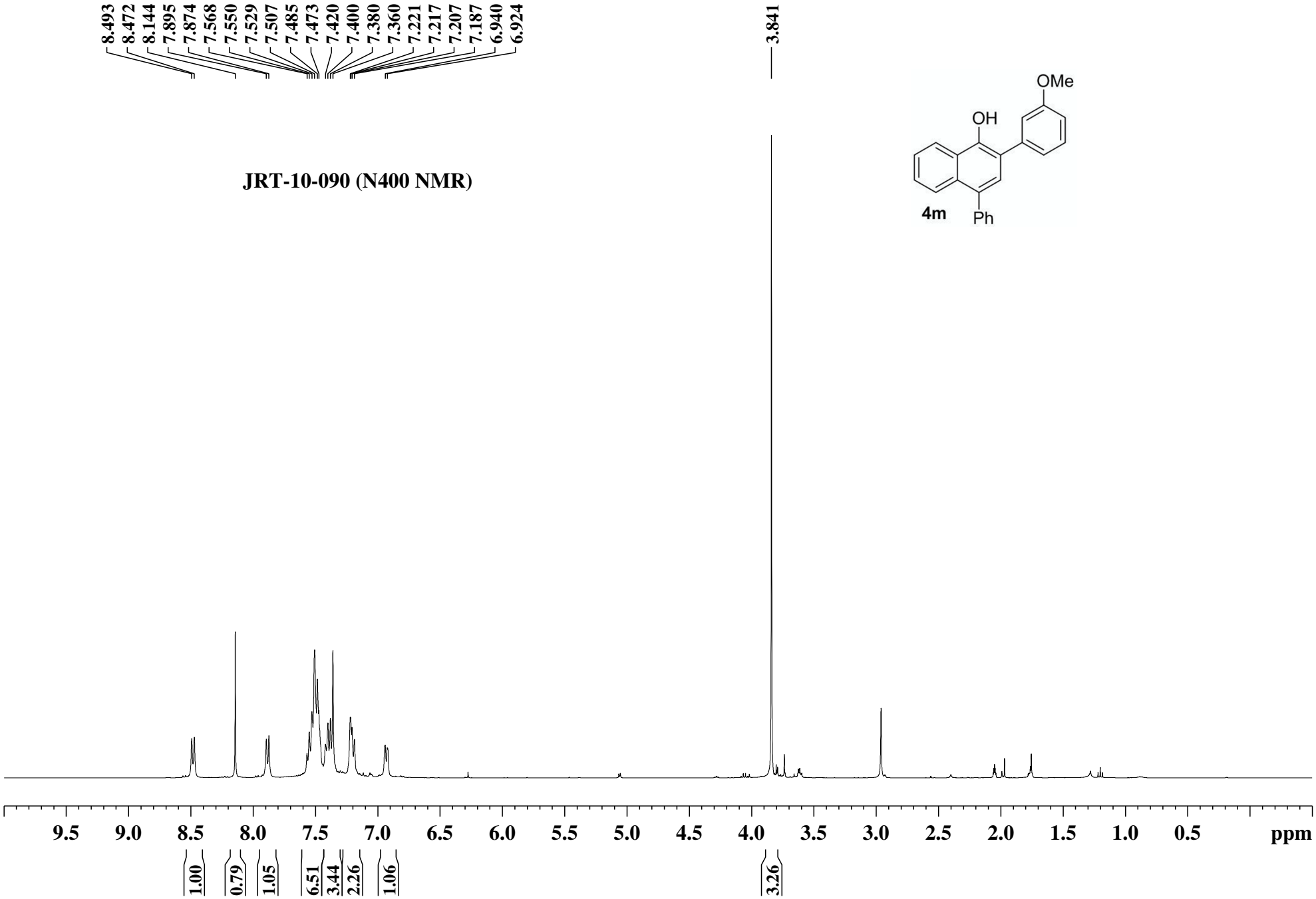
4k

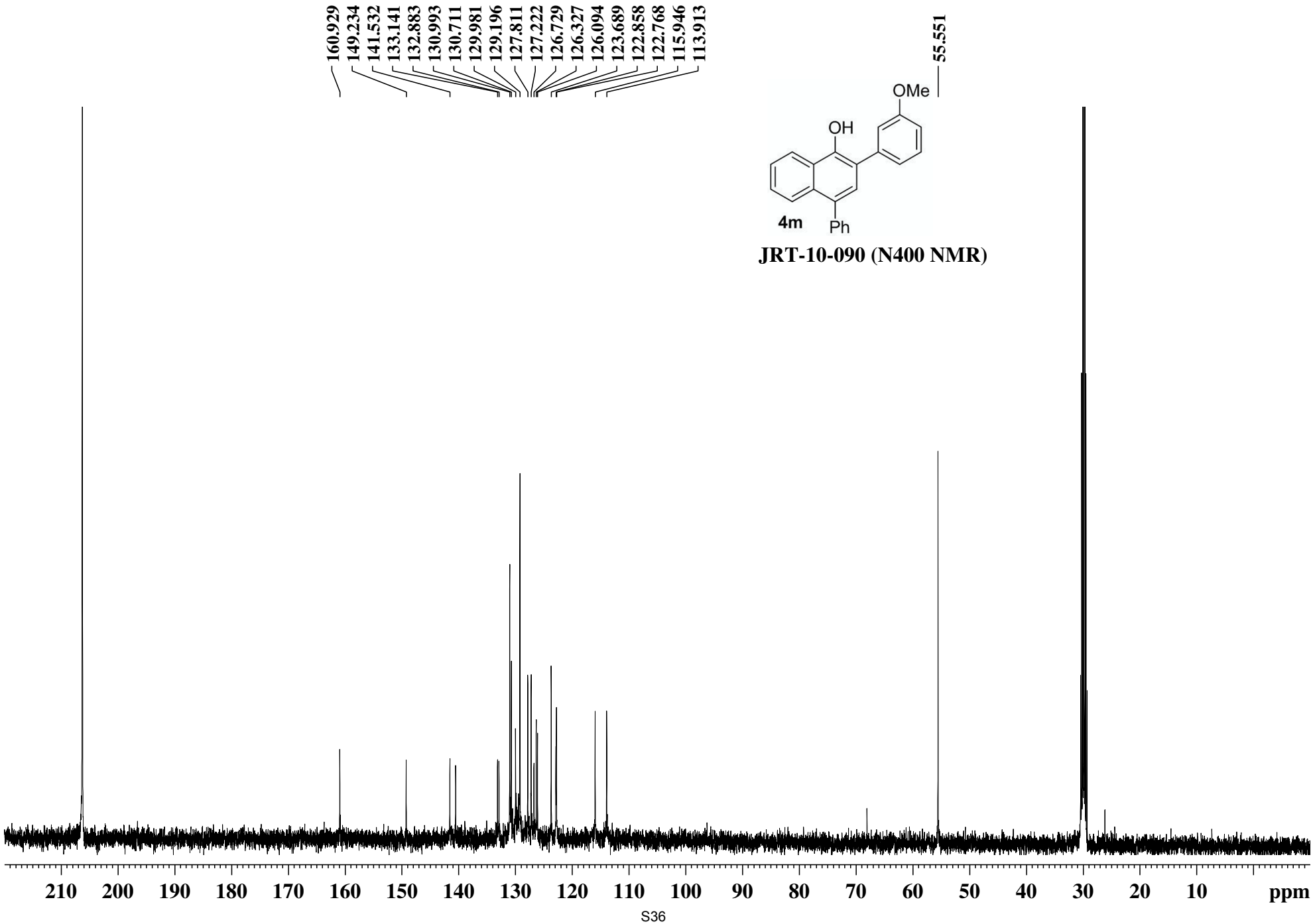


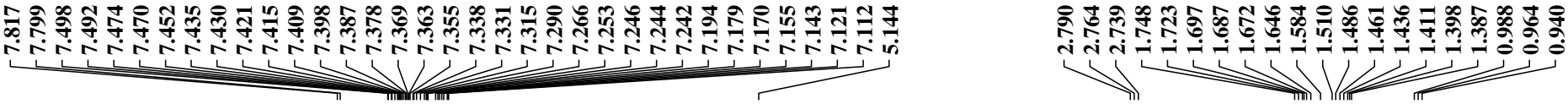

148.647
140.765
140.497
133.802
132.626
132.229
130.220
130.127
129.715
128.876
128.672
128.365
127.045
127.022
126.608
126.066
125.570
125.425
122.741
121.101
OH
Cl
4k
Ph
JRT-10-087B -con 400 MHz
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

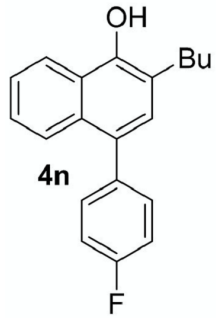

8.462
8.442
8.366
7.877
7.856
7.685
7.681
7.669
7.664
7.583
7.581
7.566
7.563
7.546
7.543
7.512
7.500
7.479
7.444
7.436
7.432
7.428
7.422
7.415
7.326
JRT-10-130 acetone-d6 400 MHz
OH
Cl
Ph
4l
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.00
0.69
0.99
2.04
1.19
5.32
1.36
1.06
1.06


149.403
141.413
138.230
133.452
133.393
133.011
132.393
130.979
129.798
129.486
129.213
127.892
127.361
126.894
126.418
126.242
123.546
122.069
OH
Cl
Ph
4l
JRT-10-130
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


8.493
8.472
8.144
7.895
7.874
7.568
7.550
7.529
7.507
7.485
7.473
7.420
7.400
7.380
7.360
7.221
7.217
7.207
7.187
6.940
6.924
3.841
JRT-10-090 (N400 NMR)
OMe
OH
4m
Ph
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.00
0.79
1.05
6.51
3.44
2.26
1.06
3.26

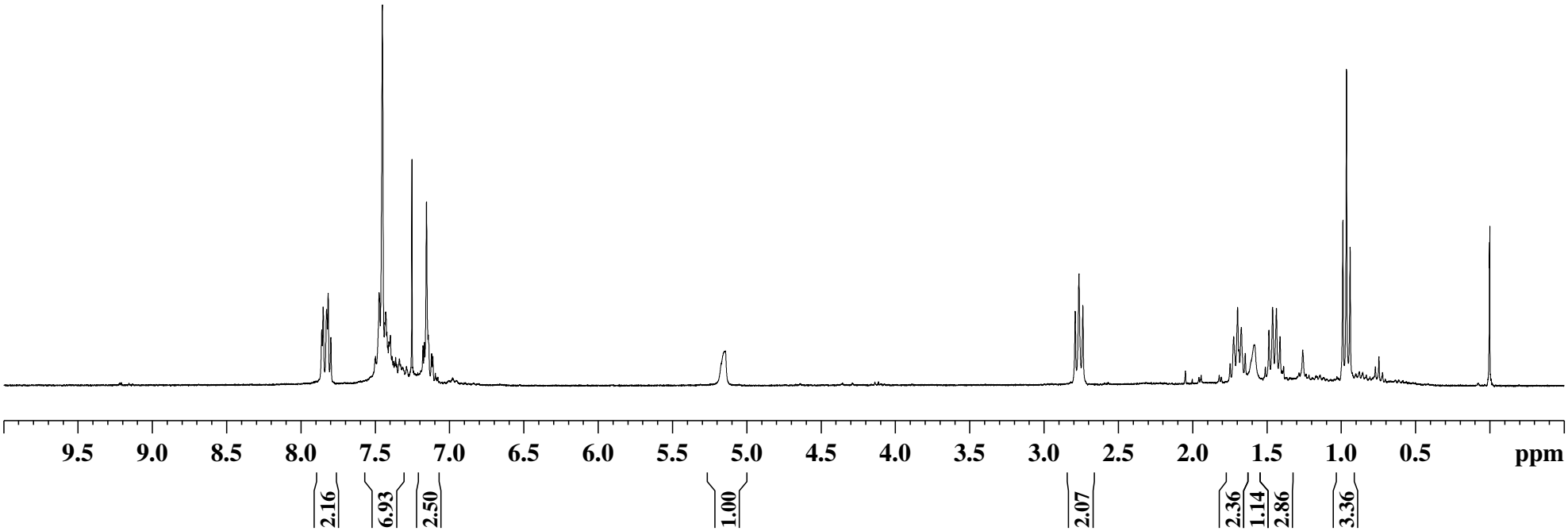

160.929
149.234
141.532
133.141
132.883
130.993
130.711
129.981
129.196
127.811
127.222
126.729
126.327
126.094
123.689
122.858
122.768
115.946
113.913
55.551
OMe
OH
4m
Ph
JRT-10-090 (N400 NMR)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

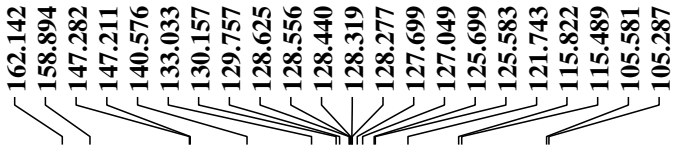


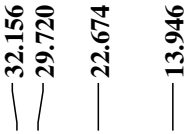


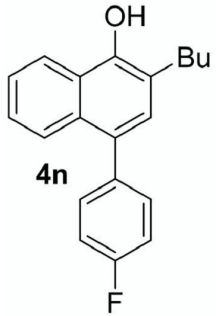
4n

JRT-9-182 F2 300 MHz, CDCl3

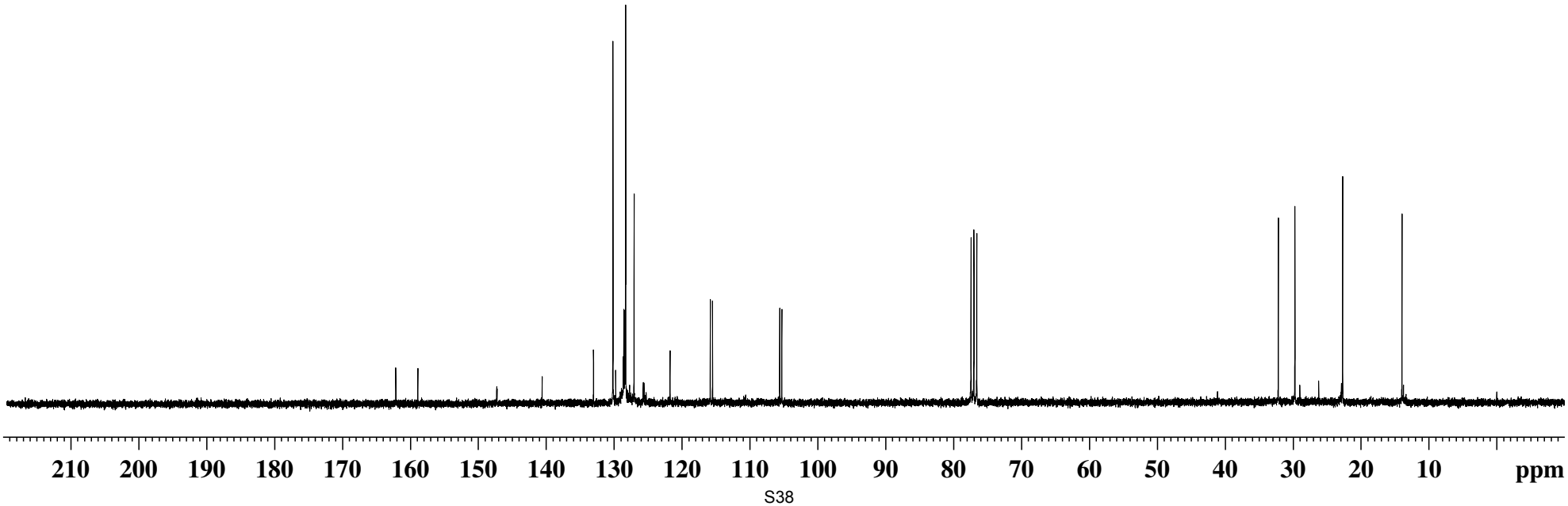


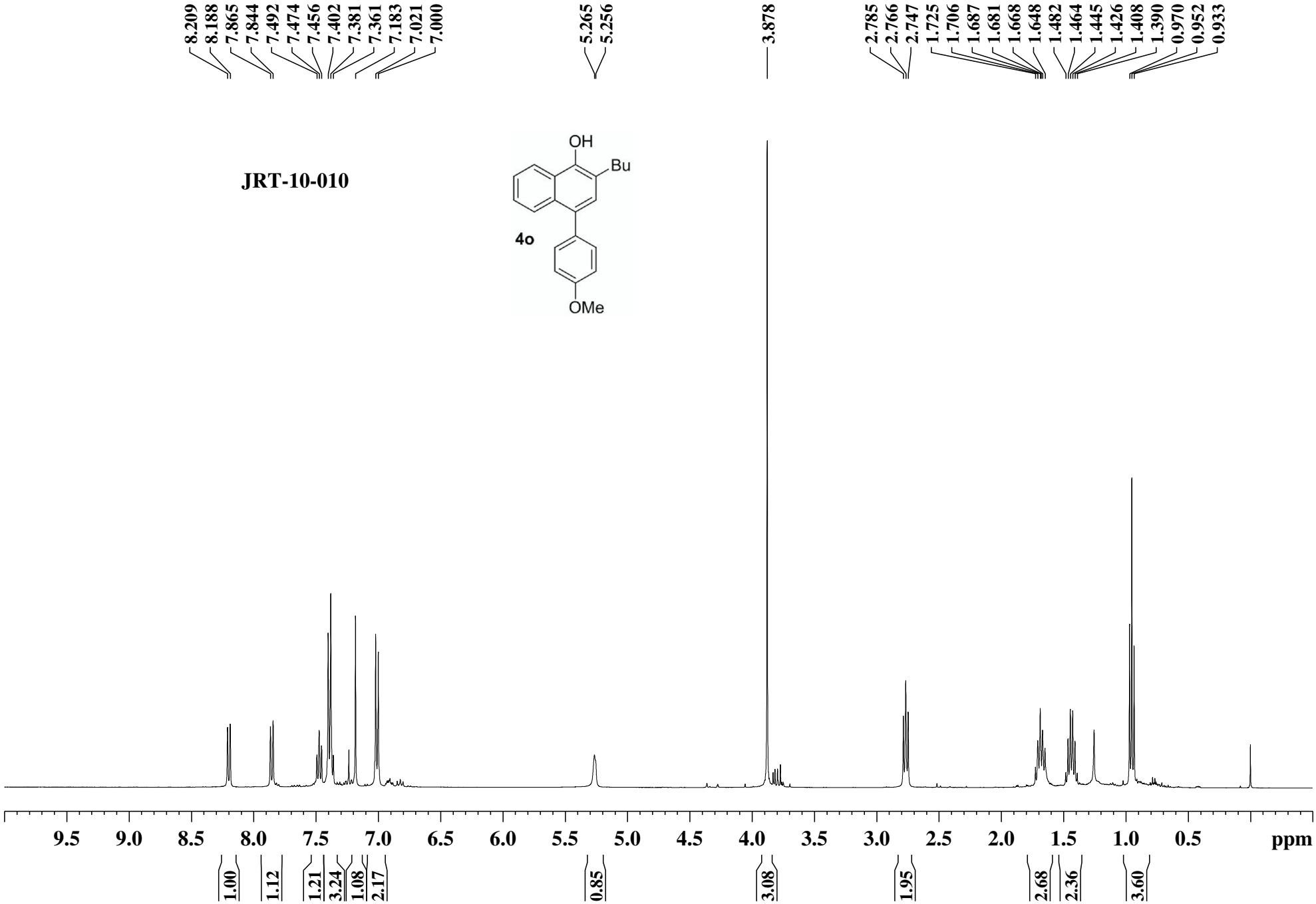


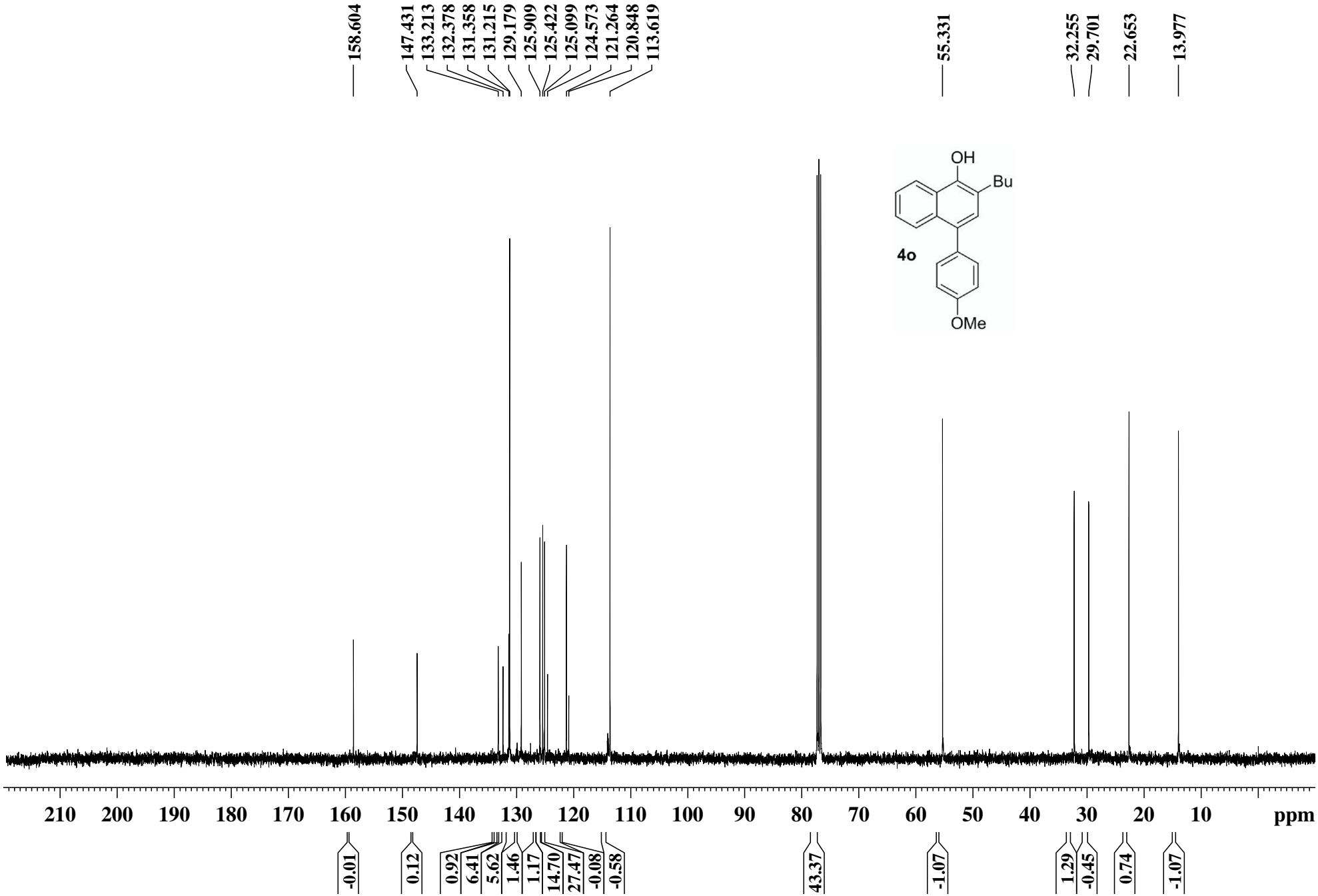


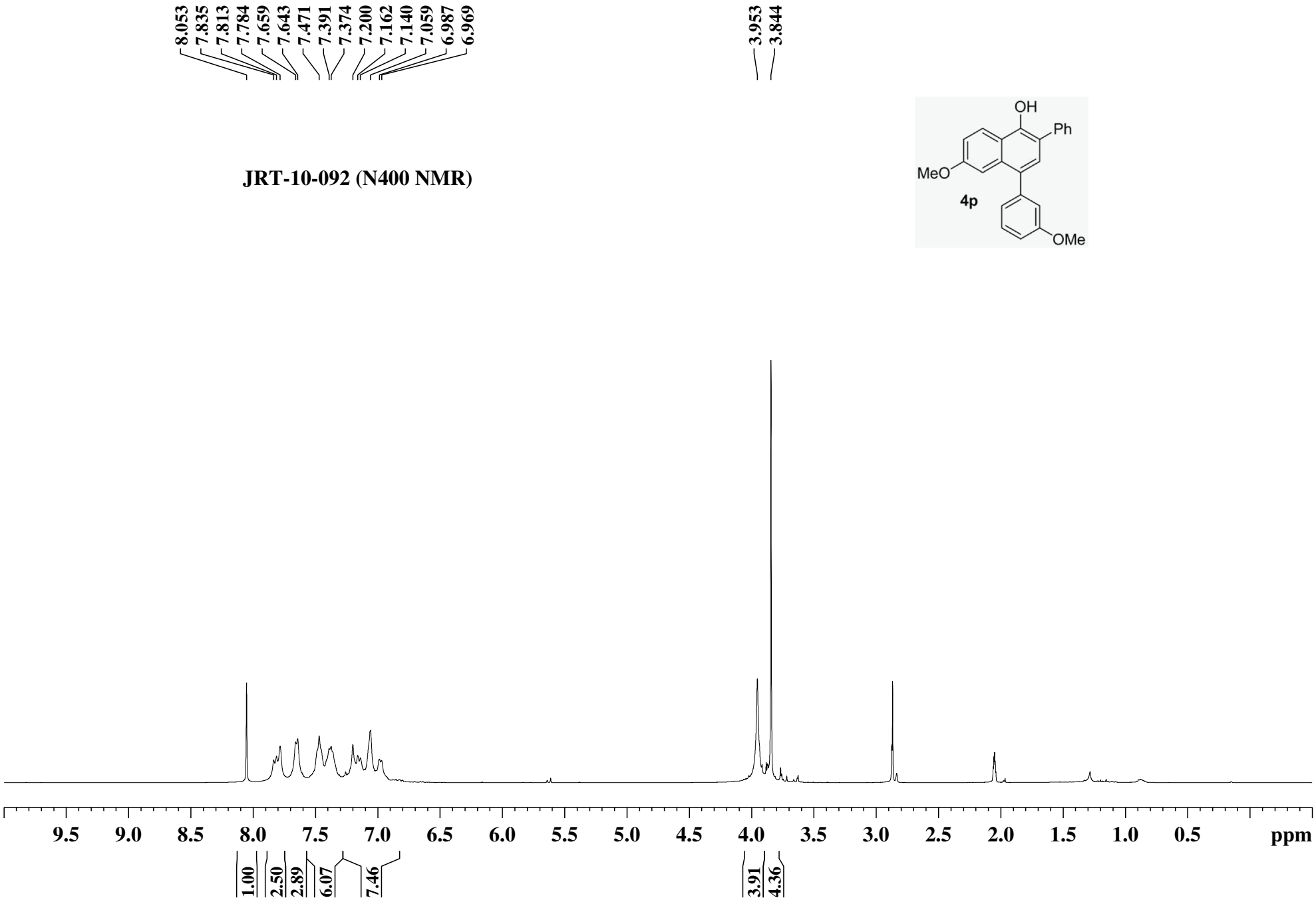


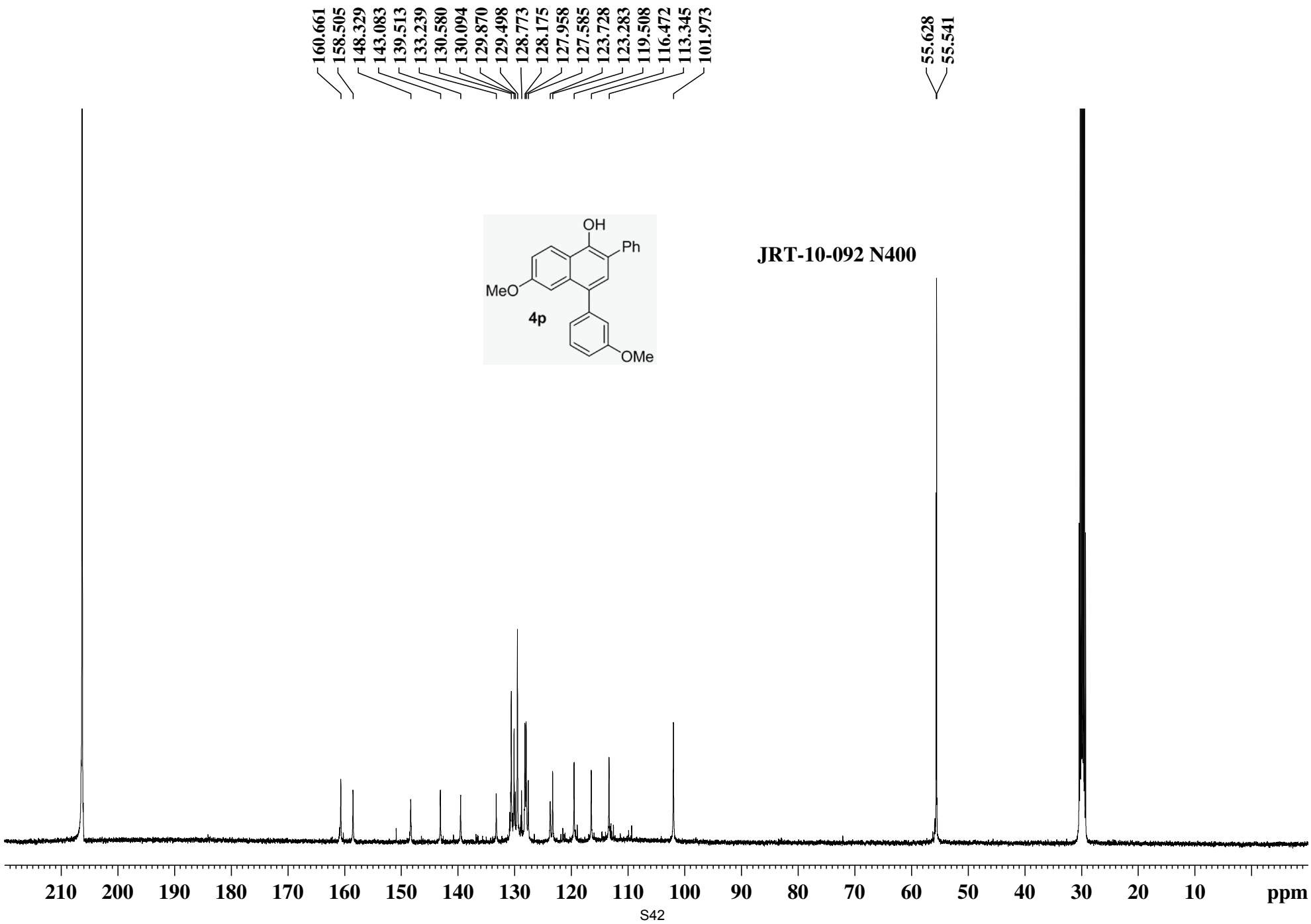
JRT-9-182 F2 300 MHz, CDCl3

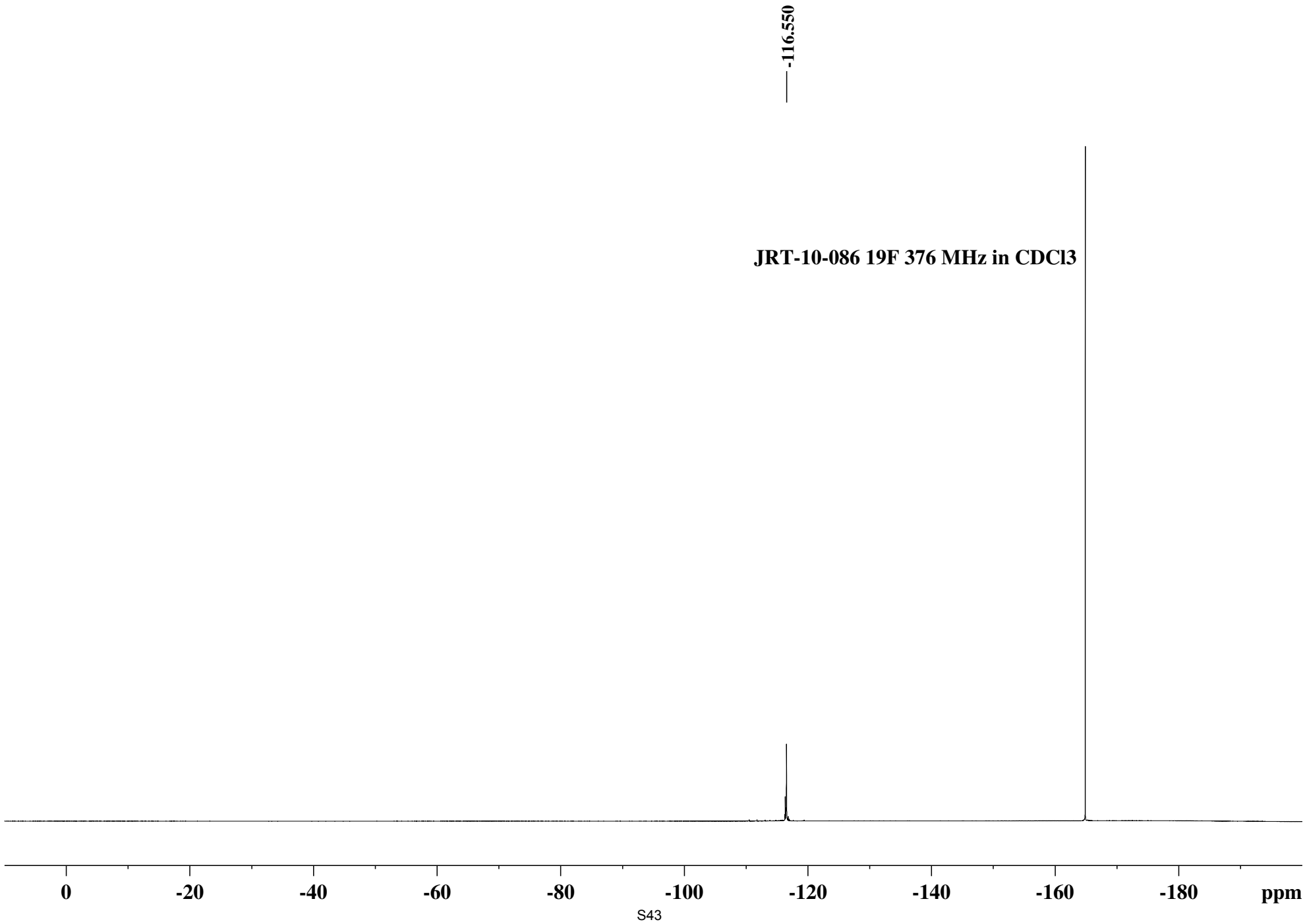


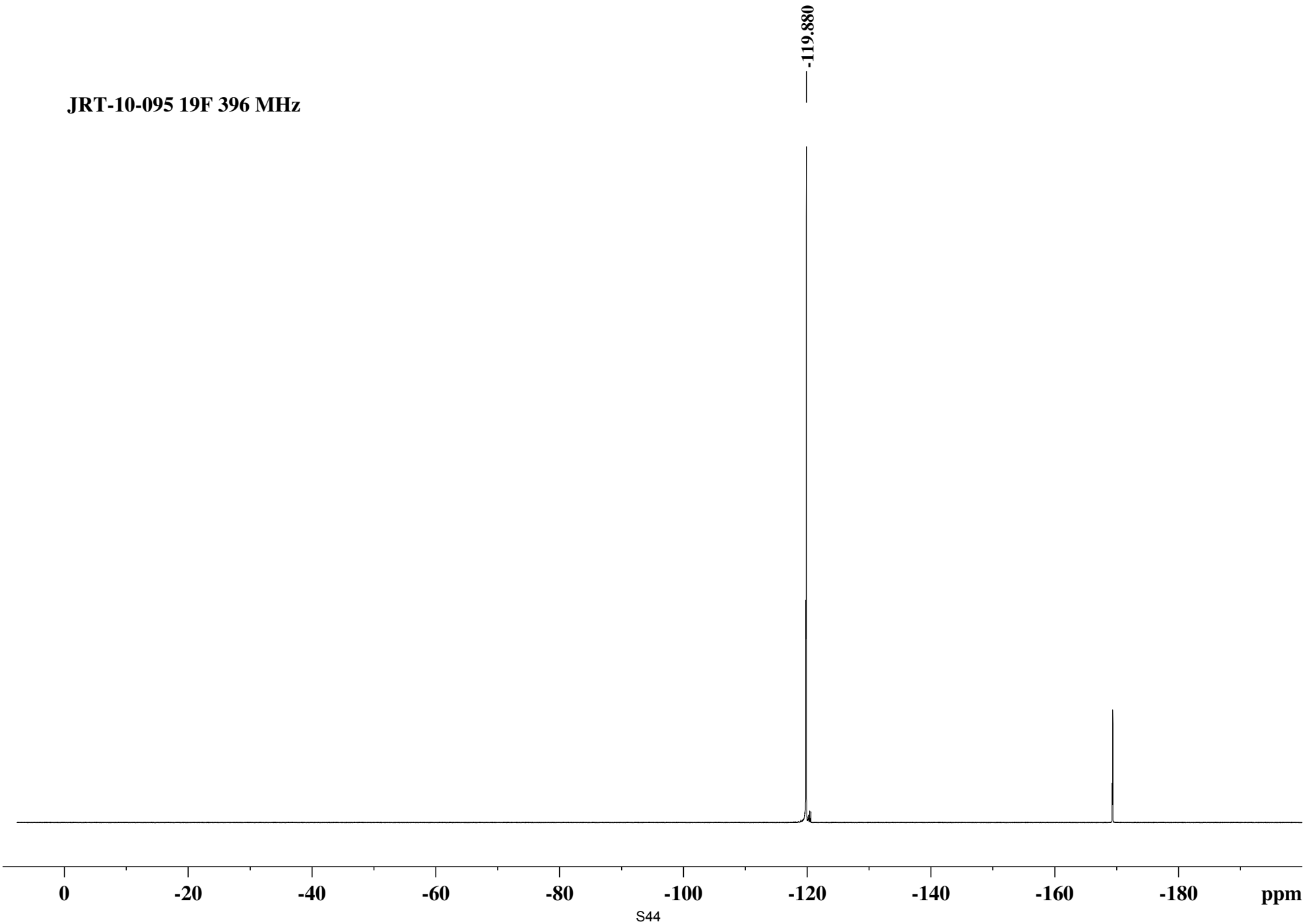

8.209
8.188
7.865
7.844
7.492
7.474
7.456
7.402
7.381
7.361
7.183
7.021
7.000
5.265
5.256
3.878
2.785
2.766
2.747
1.725
1.706
1.687
1.681
1.668
1.648
1.482
1.464
1.445
1.426
1.408
1.390
0.970
0.952
0.933
JRT-10-010
OH
Bu
4o
OMe
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.00
1.12
1.21
3.24
1.08
2.17
0.85
3.08
1.95
2.68
2.36
3.60

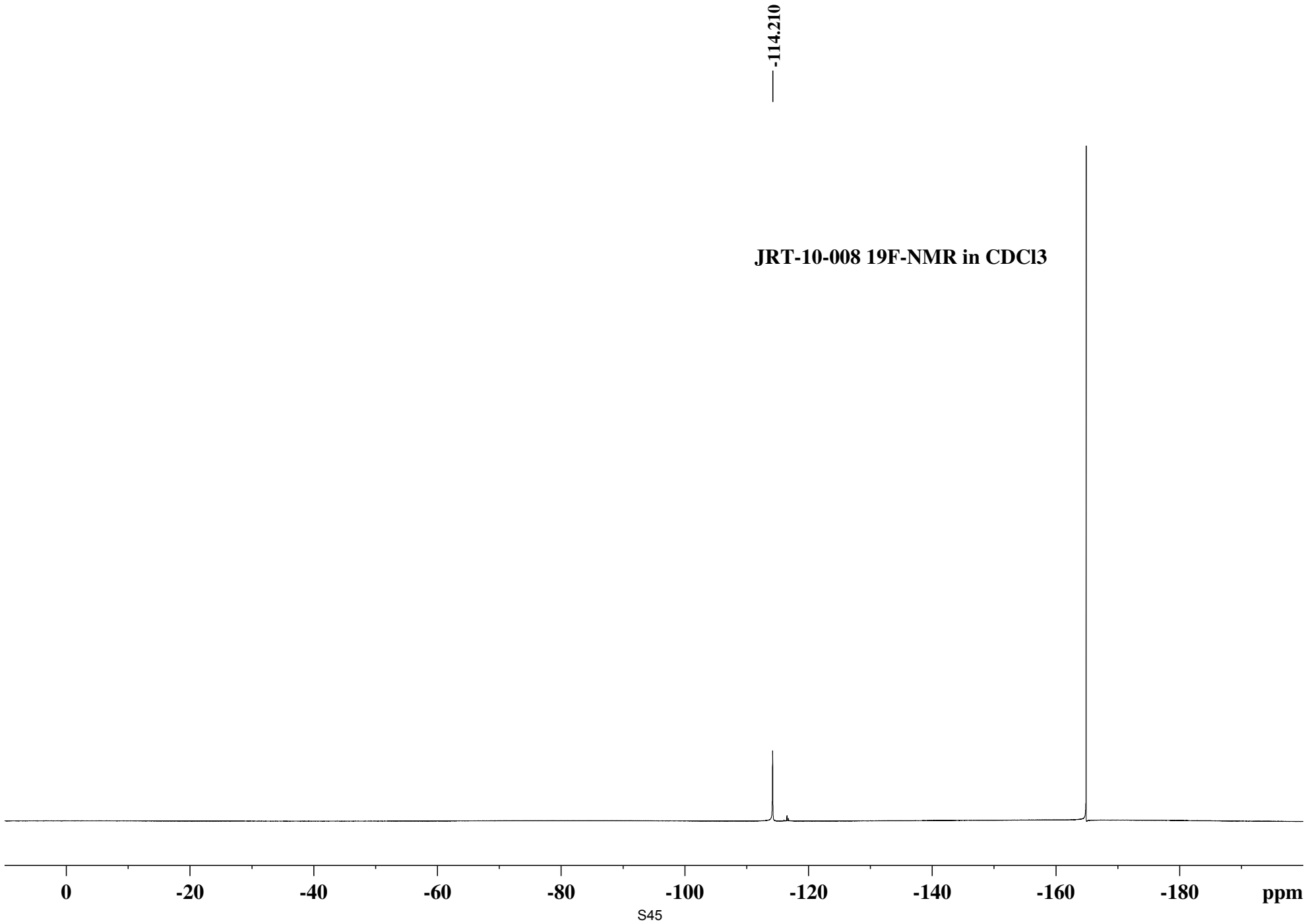

158.604
147.431
133.213
132.378
131.358
131.215
129.179
125.909
125.422
125.099
124.573
121.264
120.848
113.619
55.331
32.255
29.701
22.653
13.977
OH
Bu
4o
OMe
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
-0.01
0.12
0.92
6.41
5.62
1.46
1.17
14.70
27.47
-0.08
-0.58
43.37
-1.07
1.29
-0.45
0.74
-1.07

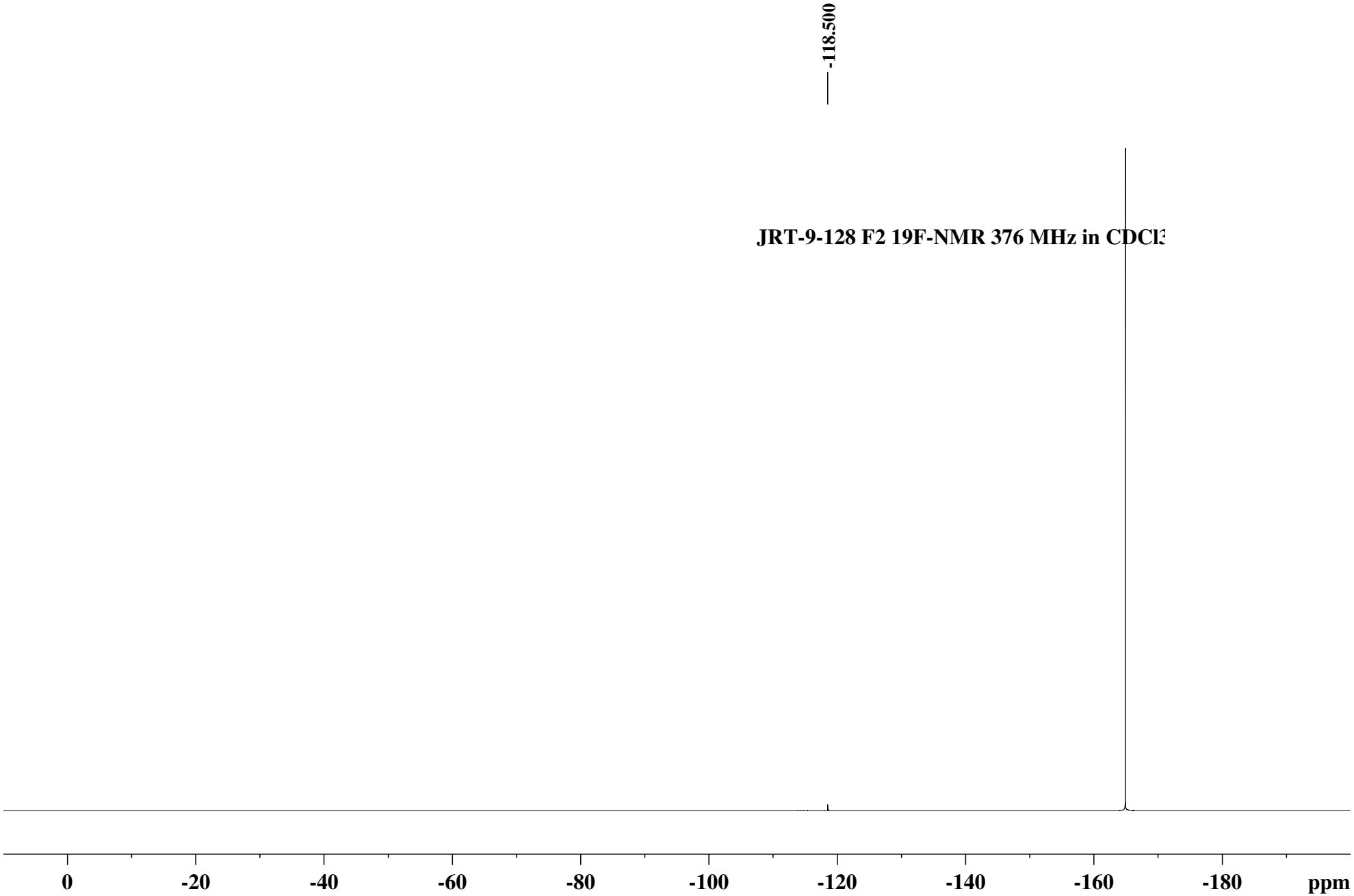

8.053
7.835
7.813
7.784
7.659
7.643
7.471
7.391
7.374
7.200
7.162
7.140
7.059
6.987
6.969
3.953
3.844
JRT-10-092 (N400 NMR)
OH
Ph
MeO
4p
OMe
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.00
2.50
2.89
6.07
7.46
3.91
4.36


160.661
158.505
148.329
143.083
139.513
133.239
130.580
130.094
129.870
129.498
128.773
128.175
127.958
127.585
123.728
123.283
119.508
116.472
113.345
101.973
55.628
55.541
OH
Ph
MeO
4p
OMe
JRT-10-092 N400
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


-116.550
JRT-10-086 19F 376 MHz in CDCl3
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
ppm


JRT-10-095 19F 396 MHz
-119.880
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
ppm

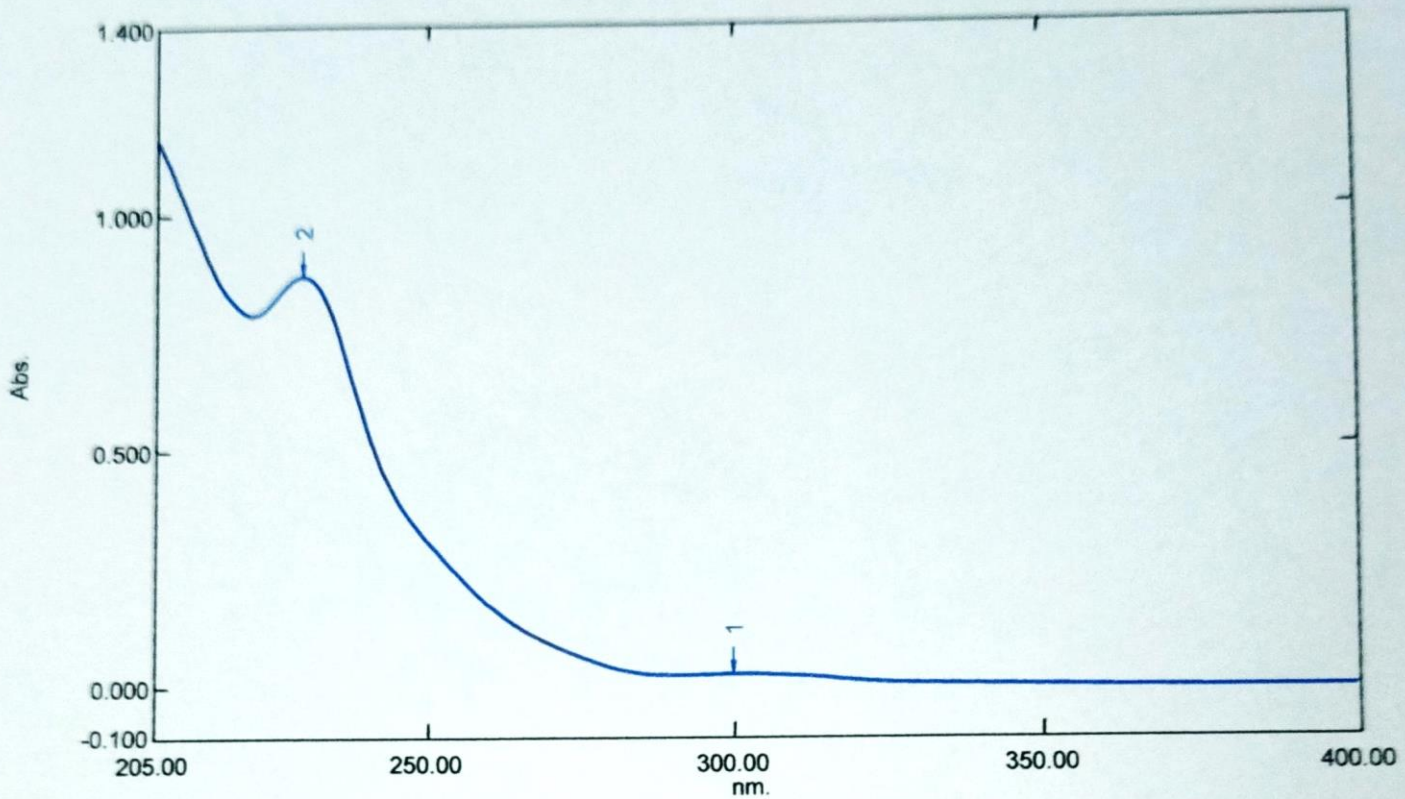

-114.210
JRT-10-008 19F-NMR in CDCl3
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
ppm


-118.500
JRT-9-128 F2 19F-NMR 376 MHz in CDCl3
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
ppm

Spectrum Peak Pick Report

07/06/2022 19:40:42

Data Set: D:\User Kittiporn\Dr.Chansak\CT- JRT.spc - RawData



No.	P/V	Wavelength	Abs.	Description
1		300.00	0.028	
2		229.50	0.871	

[Measurement Properties]

Wavelength Range (nm.):	205.00 to 700.00
Scan Speed:	Fast
Sampling Interval:	0.5
Auto Sampling Interval:	Enabled
Scan Mode:	Single

[Instrument Properties]

Instrument Type:	UV-1700 Series
Measuring Mode:	Absorbance
Slit Width:	1.0 nm
Light Source Change Wavelength:	340.8 nm
S/R Exchange:	Normal

[Attachment Properties]

Attachment:	None

[Operation]

Threshold:	0.0010000
Points:	4
InterPolate:	Disabled
Average:	Disabled

[Sample Preparation Properties]

Weight:	2.8 mg
Volume:	3 ml
Dilution:	150
Path Length:	1 cm
Additional Information:	DR.Chansak---CT-JRT --- Solvent MeOH --UV Cut off 205