Supporting Information

Synthesis of Indenes via Brønsted Acid-Catalyzed Cyclization of Diaryl- and Alkyl Aryl-1,3-dienes

Dahan Eom, Sangjune Park, Youngchul Park, Taekyu Ryu, and Phil Ho Lee*

Department of Chemistry, Kangwon National University, Chuncheon 200-701, Republic of Korea

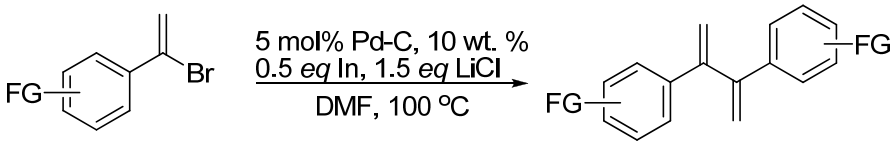
phlee@kangwon.ac.kr

Table of Contents

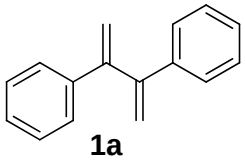
Experimental Section

General: A variety of chemical reagents were commercially purchased and used without further purification. Dichloromethane (CH_2Cl_2) was freshly distilled from calcium hydride. All reaction mixtures were stirred magnetically and were monitored by thin-layer chromatography using silica gel precoated glass plates, which were visualized with UV light and then developed using a solution of *p*-anisaldehyde. Flash column chromatography was carried out using silica gel (0.040-0.063 mm, 230-400 mesh). ^{1}H and ^{13}C NMR spectra were measured at 298 K on 400 Fourier Transform NMR spectrometer. Chemical shifts were reported in δ (ppm), relative to the internal standard of TMS. The signals observed were described as: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplets). The number of protons (n) for a given resonance was indicated as nH. Coupling constants are reported as *J* value in Hz. ^{13}C NMR are reported as δ (ppm) in downfield from TMS and relative to the signal of chloroform-*d* (δ 77.00, triplet). Mass spectrometry was performed on GC/HRMS spectrometer (KBSI) under electron impact (EI) ionization technique (magnetic sector - electric sector double focusing mass analyzer). Infrared spectra were recorded on FT-IR spectrometer as a thin film pressed between two sodium chloride plates. Melting points were determined in open capillary tube.

Typical experimental procedures for synthesis of symmetric 2,3-diaryl-1,3-butadienes:

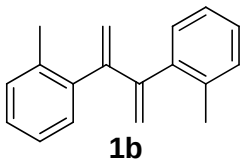


To a solution of Pd-C, 10 wt. % (26.6 mg, 0.025 mmol, 5 mol %), indium (57.4 mg, 0.5 mmol), and lithium chloride (63.5 mg, 1.5 mmol) in DMF (2 mL) were added α-bromostyrene (204.0 mg, 1.0 mmol) at room temperature. The dark blue mixture was stirred at 100 ^{o}C for 2 h. The reaction mixture was quenched with sat-$NaHCO_3$ (20 mL). The aqueous layer was extracted with diethyl ether (3 x 20 mL), and the organic layer was washed with brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. Silica gel fresh column chromatography (hexane) gave buta-1,3-diene-2,3-diyldibenzene (95.9 mg, 0.93 mmol, 93%) as white solid.[1]



1a

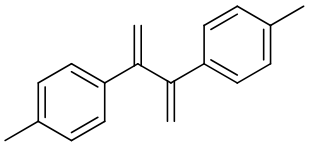
Buta-1,3-diene-2,3-diyldibenzene (1a) : R_f = 0.4 (hexane); White solid. m.p. 45-47 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38-7.40 (m, 4H), 7.19-7.28 (m, 6H), 5.54 (d, *J* = 1.6 Hz, 2H), 5.31 (d, J = 1.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.9, 140.23, 128.2, 127.6, 116.4; IR (film): 3032, 1608, 1574, 1493, 1443, 1098, 1071, 1029, 902, 774, 705 cm^{-1}. HRMS (EI): *m/z* calcd. for $C_{16}H_{14}$: 206.1096; found: 206.1093



1b

To a solution of Pd-C, 10 wt. % (26.6 mg, 0.025 mmol, 5 mol %), indium (57.4 mg, 0.5 mmol), and lithium chloride (63.5 mg, 1.5 mmol) in DMF (2 mL) were added 1-(1-bromovinyl)-2-methylbenzene (197.1 mg, 1.0 mmol) at room temperature. The dark blue mixture was stirred at 100 ^{o}C for 2 h. The reaction mixture was quenched with sat-$NaHCO_3$ (20 mL). The aqueous layer was extracted with diethyl ether (3 x 20 mL), and the organic layer was washed with brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. Silica gel fresh column chromatography (hexane) gave 2,2'-(buta-1,3-diene-2,3-diyl)bis(methylbenzene (190 mg, 0.81 mmol, 81%) as white solid.[1]

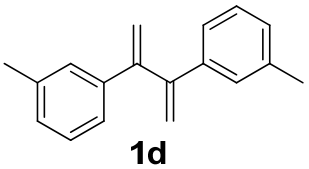
2,2'-(Buta-1,3-diene-2,3-diyl)bis(methylbenzene) (1b)[1] : R_f= 0.4 (hexane); White solid. m.p. 56-57 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ 7.13-7.17 (m, 8H), 4.97 (s, 2H), 4.80 (s, 2H), 2.28 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.4, 141.3, 136.5, 130.2, 130.1, 127.6, 125.9, 118.7, 20.1; IR (film): 3060, 3017, 2952, 2924, 1762, 1588, 1487, 1455, 1380, 1041, 908 cm^{-1}; HRMS (EI): *m/z* calcd. for $C_{18}H_{18}$: 234.1409; found 234.1406



1c

To a solution of Pd-C, 10 wt. % (26.6 mg, 0.025 mmol, 5 mol %), indium (57.4 mg, 0.5 mmol), and lithium chloride (63.5 mg, 1.5 mmol) in DMF (2 mL) were added 1-(1-bromovinyl)-4-methylbenzene (197.1 mg, 1.0 mmol) at room temperature. The dark blue mixture was stirred at 100 ^oC for 2 h. The reaction mixture was quenched with sat-$NaHCO_3$ (20 mL). The aqueous layer was extracted with diethyl ether (3 x 20 mL), and the organic layer was washed with brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. Silica gel fresh column chromatography (hexane) gave 4,4'-(buta-1,3-diene-2,3-diyl)bis(methylbenzene) (199 mg, 0.85 mmol, 85%) as white solid.[1]

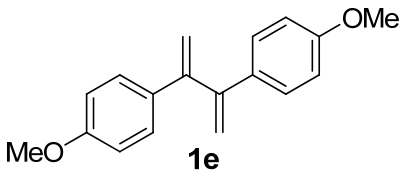
4,4'-(Buta-1,3-diene-2,3-diyl)bis(methylbenzene) (1c)[1,2] : R_f= 0.4 (hexane); White solid. m.p. 56-58 ^oC; ^1H NMR (200 MHz, $CDCl_3$) δ 7.28 (d, *J* = 8.1 Hz, 4H), 7.07 (d, *J* = 7.9 Hz, 4H), 5.51 (d, *J* = 1.7 Hz, 2H), 5.30 (d, *J* = 1.6 Hz, 2H), 2.30 (s, 6H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 149.7, 137.3, 137.2, 128.9, 127.3, 115.4, 21.2; IR (film): 3023, 2919, 2862, 1511, 1447, 1183, 1118, 1019, 898, 824 cm^{-1}; MS (EI): *m/z* (%)=235 $[M+1]^+$ (32), 205 $[M-1]^+$ (94), 218 (100)



1d

To a solution of Pd-C, 10 wt. % (26.6 mg, 0.025 mmol, 5 mol %), indium (57.4 mg, 0.5 mmol), and lithium chloride (63.5 mg, 1.5 mmol) in DMF (2 mL) were added 1-(1-bromovinyl)-3-methylbenzene (197.1 mg, 1.0 mmol) at room temperature. The dark blue mixture was stirred at 100 ^oC for 3 h. The reaction mixture was quenched with sat-$NaHCO_3$ (20 mL). The aqueous layer was extracted with diethyl ether (3 x 20 mL), and the organic layer was washed with brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. Silica gel fresh column chromatography (hexane) gave 3,3'-(buta-1,3-diene-2,3-diyl)bis(methylbenzene) (195 mg, 0.83 mmol, 83%) as white solid.[1]

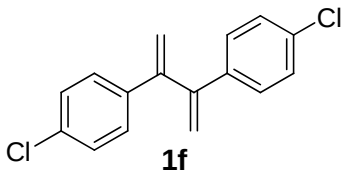
3,3'-(Buta-1,3-diene-2,3-diyl)bis(methylbenzene) (1d)[1] : R_f = 0.4 (hexane); White solid. m.p. 49-51 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ 7.06-7.15 (m, 6H), 6.96-6.98 (m, 2H), 5.43 (d, J = 1.7 Hz, 2H), 5.18 (d, J = 1.7 Hz, 2H), 2.26 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.4, 140.8, 138.0, 128.7, 128.6, 128.4, 125.1, 116.6, 21.9; IR (film): 3037, 2949, 2922, 1754, 1681, 1603, 1486, 1344, 1260, 998, 790, 737 cm^{-1}; HRMS (EI): *m/z* calcd. for $C_{18}H_{18}$: 234.1409; found 234.1407



1e

To a solution of Pd-C, 10 wt. % (26.6 mg, 0.025 mmol, 5 mol %), indium (57.4 mg, 0.5 mmol), and lithium chloride (63.5 mg, 1.5 mmol) in DMF (2 mL) were added 1-(1-bromovinyl)-4-methoxybenzene (213.1 mg, 1.0 mmol) at room temperature. The dark blue mixture was stirred at 100 ^oC for 2 h. The reaction mixture was quenched with sat-$NaHCO_3$ (20 mL). The aqueous layer was extracted with diethyl ether (3 x 20 mL), and the organic layer was washed with brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. Silica gel fresh column chromatography (hexane) gave 4,4'-(buta-1,3-diene-2,3-diyl)bis(methoxybenzene) (232 mg, 0.87 mmol, 87%) as white solid.[1]

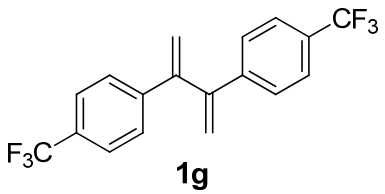
4,4'-(Buta-1,3-diene-2,3-diyl)bis(methoxybenzene) (1e)[1,2] : R_f = 0.3 (hexane); white solid. m.p. 108-109 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ 7.30-7.33 (m, 4H), 6.77-6.81 (m, 4H), 5.47 (d, J = 1.8 Hz, 2H), 5.23 (d, J = 1.8 Hz, 2H), 3.76 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.4, 147.5, 131.6, 131.3, 128.9, 120.7, 117.5, 111.4, 56.2; ; IR (film): 3084, 3008, 2957, 2929, 2835, 1589, 1490, 1247, 1024, 754 cm^{-1}; MS (EI): *m/z* (%)=276 $[M+1]^+$ (42), 266 $[M]^+$ (100), 265 $[M-1]^+$ (47), 251 (49), 234 (77), 121 (52)



1f

To a solution of Pd-C, 10 wt. % (26.6 mg, 0.025 mmol, 5 mol %), indium (57.4 mg, 0.5 mmol), and lithium chloride (63.5 mg, 1.5 mmol) in DMF (2 mL) were added 1-(1-bromovinyl)-4-chlorobenzene (217.5 mg, 1.0 mmol) at room temperature. The dark blue mixture was stirred at 100 ^oC for 2 h. The reaction mixture was quenched with sat-$NaHCO_3$ (20 mL). The aqueous layer was extracted with diethyl ether (3 x 20 mL), and the organic layer was washed with brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. Silica gel fresh column chromatography (hexane) gave 4,4'-(buta-1,3-diene-2,3-diyl)bis(chlorobenzene) (245 mg, 0.89 mmol, 89%) as white solid.[1]

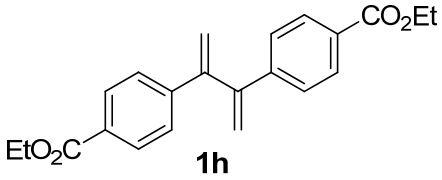
4,4'-(Buta-1,3-diene-2,3-diyl)bis(chlorobenzene) (1f)[1] : R_f = 0.5 (hexane); white solid. m.p. 64-69 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ 7.26-7.29 (m, 2H), 7.21-7.24 (m, 2H), 5.53 (d, *J* = 2.0 Hz, 2H), 5.33 (d, *J* = 2.1 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.8, 138.6, 133.9, 129.1, 128.9, 117.4; IR (film): 2921, 2849, 1753, 1489, 1398, 1092, 1013 cm^{-1}; HRMS (EI): *m/z* calcd. for $C_{16}H_{12}Cl_2$: 274.0316; found 274.0319



1g

To a solution of Pd-C, 10 wt. % (26.6 mg, 0.025 mmol, 5 mol %), indium (57.4 mg, 0.5 mmol), and lithium chloride (63.5 mg, 1.5 mmol) in DMF (2 mL) were added 1-(1-bromovinyl)-4-(trifluoromethyl)benzene (251.0 mg, 1.0 mmol) at room temperature. The dark blue mixture was stirred at 100 ^oC for 2 h. The reaction mixture was quenched with sat-$NaHCO_3$ (20 mL). The aqueous layer was extracted with diethyl ether (3 x 20 mL), and the organic layer was washed with brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. Silica gel fresh column chromatography (hexane) gave 4,4'-(buta-1,3-diene-2,3-diyl)bis((trifluoromethyl)benzene) (301 mg, 0.88 mmol, 88%) as white solid.[1]

4,4'-(Buta-1,3-diene-2,3-diyl)bis((trifluoromethyl)benzene) (1g)[1,2] : R_f = 0.4 (hexane); white solid. m.p. 103-106 ^oC; ^1H NMR (200 MHz, $CDCl_3$) δ 7.55 (d, *J* = 8.1 Hz, 4H), 7.48 (d, *J* = 8.1 Hz, 4H), 5.64 (d, *J* = 1.1 Hz, 2H), 5.43 (d, *J* = 1.1 Hz, 2H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 148.1, 143.2, 129.8 (q, *J* = 32.3 Hz), 127.7, 125.3 (q, *J* = 3.3 Hz), 124.0 (q, *J* = 27.1 Hz), 118.6; IR (film): 3423, 3056, 2927, 2855, 1760, 1410, 1325, 1167, 1124, 1068, 849 cm^{-1}; MS (EI): *m/z* (%)=343 $[M+1]^+$ (3), 342 $[M]^+$ (13), 341 $[M-1]^+$ (2), 253 (100)

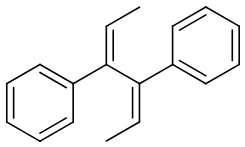


1h

To a solution of $PdCl_2$(dppf) (22.0 mg, 3 mol %) and potassium phosphoxide (636.8 mg, 3.0 mmol) in DMF (4.0 mL) were added ethyl 4-(1-(trifluoromethylsulfonyloxy)vinyl)benzoate (324.3 mg, 1.0 mmol) and ethyl 4-(1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)vinyl)benzoate (332.4 mg, 1.1 mmol) at room temperature. The mixture was stirred at 60 ^oC for 3.5 h. The reaction mixture was quenched with H_2O (20 mL). The aqueous layer was extracted with diethyl ether (3 x 20 mL), and the organic layer was washed with brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. Silica gel fresh column

chromatography (EtOAc:hexane = 1:10) gave diethyl 4,4'-(buta-1,3-diene-2,3-diyl)dibenzoate (249 mg, 0.71 mmol, 71%) as colorless oil. [4]

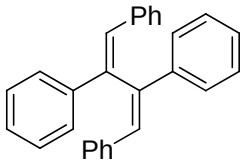
Diethyl 4,4'-(buta-1,3-diene-2,3-diyl)dibenzoate (1h)[4] : R_f = 0.3 (EtOAc:hexane = 1:10); Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92-7.95 (m, 4H), 7.40-7.43 (m, 4H), 5.65 (d, *J* = 1.2 Hz, 2H), 5.45 (d, *J* = 1.2 Hz, 2H), 4.34 (q, *J* = 7.1 Hz, 4H), 1.37 (t, *J* = 7.1 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.3, 148.6, 144.2, 129.63, 129.60, 127.4, 118.3, 60.9, 14.3; IR (film): 3091, 3052, 2981, 2937, 2905, 1714, 1607, 1406, 1277, 1178, 1107, 1020, 863, 721 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{22}H_{22}O_4$: 350.1518; found 350.1515



1i

To a solution of Pd-C, 10 wt. % (26.6 mg, 0.025 mmol, 5 mol %), indium (57.4 mg, 0.5 mmol), and lithium chloride (63.5 mg, 1.5 mmol) in DMF (2 mL) were added 1-bromoprop-1-enylbenzene (197.1 mg, 1.0 mmol) at room temperature. The dark blue mixture was stirred at 100 ^{o}C for 6 h. The reaction mixture was quenched with sat-$NaHCO_3$ (20 mL). The aqueous layer was extracted with diethyl ether (3 x 20 mL), and the organic layer was washed with brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. Silica gel fresh column chromatography (hexane) gave hexa-2,4-diene-3,4-diyldibenzene (166 mg, 0.71 mmol, 71%) as colorless oil.[1]

Hexa-2,4-diene-3,4-diyldibenzene (1i)[1] : R_f = 0.5 (hexane); Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37-7.40 (m, 4H), 7.27-7.31 (m, 2H), 7.17-7.20 (m, 4H), 5.24 (q, *J* = 6.54 Hz, 2H), 1.44 (d, *J* = 6.45 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.5, 140.4, 130.3, 128.4, 126.9, 125.8, 15.6; IR (film): 3025, 2909, 2853, 1522, 1445, 1189, 1113, 1014, 889 cm^{-1}; HRMS (EI): *m/z* calcd. for $C_{16}H_{14}$: 234.1409; found 234.1410

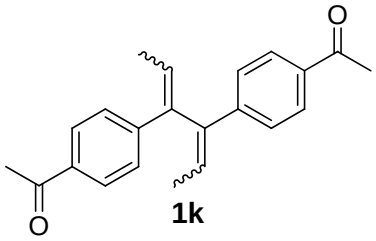


1j

To a solution of Pd-C, 10 wt. % (26.6 mg, 0.025 mmol, 5 mol %), indium (57.4 mg, 0.5 mmol), and lithium chloride (63.5 mg, 1.5 mmol) in DMF (2 mL) were added (1-bromoethene-1,2-diyl)dibenzene (259.1 mg, 1.0 mmol) at room temperature. The dark blue mixture was stirred at 100 ^{o}C for 12 h. The

reaction mixture was quenched with sat-$NaHCO_3$ (20 mL). The aqueous layer was extracted with diethyl ether (3 x 20 mL), and the organic layer was washed with brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. Silica gel fresh column chromatography (hexane) gave buta-1,3-diene-1,2,3,4-tetrayltetrabenzene (251 mg, 0.70 mmol, 70%) as colorless oil.[1]

Buta-1,3-diene-1,2,3,4-tetrayltetrabenzene (1j)[3] : R_f = 0.3 (hexane); Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.25-7.44 (m, 10H), 7.00-7.03 (m, 6H), 6.72-6.75 (m, 4H), 6.29 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.7, 137.4, 136.8, 136.6, 131.3, 130.1, 129.5, 129.4, 127.7, 126.4, ; IR (film): 3079, 3050, 3027, 2923, 1597, 1510, 1490, 1444 cm^{-1}; MS (70 eV) *m/z* (%): 358 (M^+, 52), 267 (100)

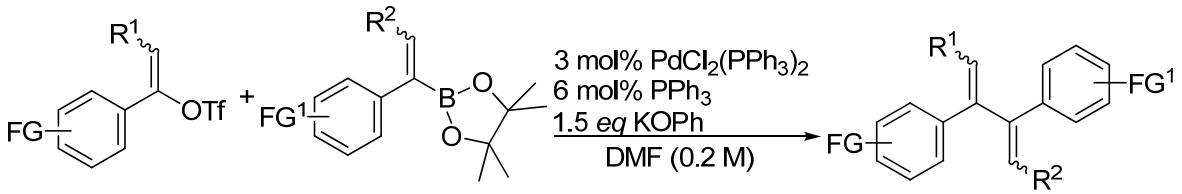


1k

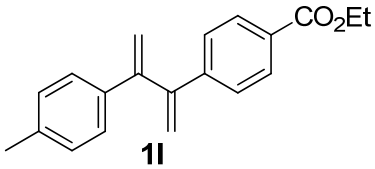
To a solution of $PdCl_2$(dppf) (22.0 mg, 3 mol %) and potassium phosphoxide (636.8 mg, 3.0 mmol) in DMF (4.0 mL) were added 1-(4-acetylphenyl)prop-1-enyltrifluoromethanesulfonate (308.3 mg, 1.0 mmol) and 1-(4-(1-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)prop-1-enyl)phenyl)ethanone (314.8 mg, 1.1 mmol) at room temperature. The mixture was stirred at room temperature for 5.5 h. The reaction mixture was quenched with H_2O (20 mL). The aqueous layer was extracted with diethyl ether (3 x 20 mL), and the organic layer was washed with brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. Silica gel fresh column chromatography (EtOAc:hexane = 1:10) gave 1,1'-(4,4'-(hexa-2,4-diene-3,4-diyl)bis(4,1-phenylene))diethanone (239 mg, 0.75 mmol, 75%) as colorless oil. [4]

1,1'-(4,4'-(Hexa-2,4-diene-3,4-diyl)bis(4,1-phenylene))diethanone (1k)[4] : R_f = 0.3 (EtOAc:hexane = 1:10); Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83-7.86 (m, 4H), 7.28-7.32 (m, 4H), 6.24 (q, *J* = 8.0 Hz, 2H), 2.56 (s, 6H), 1.96 (d, *J* = 7.1 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.0, 146.5, 144.3, 135.9, 130.4, 128.6, 127.8, 127.2, 26.9, 15.7; Data for the minor isomer 1: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78-7.81 (m, 4H), 7.39-7.41 (m, 4H), 5.89 (q, *J* = 7.1 Hz, 2H), 2.53 (s, 6H), 1.91 (d, *J* = 7.0 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.0, 143.7, 137.7, 135.8, 130.6, 129.6, 128.8, 128.7, 26.9, 16.4; Data for the minor isomer 2: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97-8.00 (m, 4H), 7.28-7.32 (m, 4H), 5.28 (q, *J* = 7.3 Hz, 2H), 2.63 (s, 6H), 1.46 (d, *J* = 6.6 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.3, 145.3, 144.2, 136.1, 129.1, 126.8, 126.3, 27.0, 15.9, 15.6; IR (film): 2912, 2854, 1682, 1601, 1403, 1357, 1267, 957 cm^{-1}; HRMS (EI): *m/z* calcd. for $C_{22}H_{22}O_2$: 318.1620, found 318.1616

Typical experimental procedures for synthesis of unsymmetric 2,3-diaryl-1,3-butadienes:

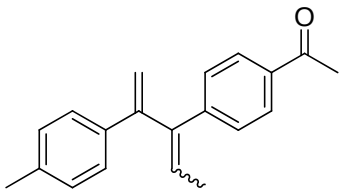


To a solution of $PdCl_2(dppf)_2$ (22.0 mg, 3 mol %) and potassium phosphoxide (636.8 mg, 3.0 mmol) in DMF (4.0 mL) were added ethyl 4-(1-(trifluoromethylsulfonyloxy)vinyl)benzoate (324.3 mg, 1.0 mmol), 4,4,5,5-tetramethyl-2-(1-p-tolylvinyl)-1,3,2-dioxaborolane (268.5 mg, 1.1 mmol) at room temperature. The mixture was stirred at 60 ^oC for 5 h. The reaction mixture was quenched with H_2O (20 mL). The aqueous layer was extracted with diethyl ether (3 x 20 mL), and the organic layer was washed with brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. Silica gel fresh column chromatography (hexane) gave ethyl 4-(3-*p*-tolylbuta-1,3-dien-2-yl)benzoate (222 mg, 0.76 mmol, 76%) as yellow oil.[4]



1l

Ethyl 4-(3-*p*-tolylbuta-1,3-dien-2-yl)benzoate (1l) : R_f = 0.4 (EtOAc:hexane = 1:10); Yellow oil. ^1H NMR (400 MHz, $CDCl_3$) δ 7.92-7.94 (m, 2H), 7.42-7.44 (m, 2H), 7.24-7.26 (m, 2H), 7.06 (d, *J* = 8.1 Hz, 2H), 5.61 (d, *J* = 1.5 Hz, 1H), 5.54 (d, *J* = 1.5 Hz, 1H), 5.42 (d, *J* = 1.5 Hz, 1H), 5.28 (d, *J* = 1.6 Hz, 1H), 4.34 (q, *J* = 7.1 Hz, 2H), 2.29 (s, 3H), 1.36 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.8, 149.7, 149.6, 145.1, 137.9, 137.2, 129.9, 129.8, 129.4, 127.8, 127.7, 118.1, 116.3, 61.3, 21.5, 14.7; IR (film): 3088, 3025, 2980, 2924, 2871, 1717, 1607, 1276, 1106, 1020, 908, 863 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{20}H_{20}O_2$: 292.1463; found: 292.1466

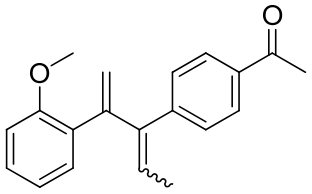


dr (1 : 2.3) **1m**

To a solution of $PdCl_2(dppf)_2$ (22.0 mg, 3 mol %) and potassium phosphoxide (636.8 mg, 3.0 mmol) in DMF (4.0 mL) were added 1-(4-acetylphenyl)prop-1-enyl trifluoromethanesulfonate (308.3 mg, 1.0 mmol), 4,4,5,5-tetramethyl-2-(1-p-tolylvinyl)-1,3,2-dioxaborolane (268.5 mg, 1.1 mmol) at room

temperature. The mixture was stirred at 60 ^{o}C for 6 h. The reaction mixture was quenched with H_2O (20 mL). The aqueous layer was extracted with diethyl ether (3 x 20 mL), and the organic layer was washed with brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. Silica gel fresh column chromatography (hexane) gave 1-(4-(2-p-tolylpenta-1,3-dien-3-yl)phenyl)ethanone (122 mg, 0.44 mmol, 44%) as brown oil.[4]

1-(4-(2-*p*-Tolylpenta-1,3-dien-3-yl)phenyl)ethanone (1m) : R_f = 0.48 (EtOAc:hexane = 1:5); Brown oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93-7.95 (m, 2H), 7.32-7.35 (m, 2H), 7.22-7.26 (m, 2H), 7.10-7.13 (m, 2H), 5.83 (q, *J* = 7.1 Hz, 1H), 5.17 (d, *J* = 1.3 Hz, 1H), 4.84 (d, *J* = 1.1 Hz, 1H), 2.60 (s, 3H), 2.34 (s, 3H), 1.64 (d, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.9, 151.2, 144.8, 142.5, 138.3, 137.1, 135.6, 130.0, 128.8, 128.30, 128.27, 128.2, 115.6, 26.6, 21.2, 15.4; Data for the minor isomer: R_f = 0.49, (EtOAc:hexane = 1:5); Brown solid. m.p. 64.0-65.2 ^{o}C.; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80-7.83 (m, 2H), 7.46-7.48 (m, 2H), 7.29-7.32 (m, 2H), 7.05-7.08 (m, 2H), 6.41 (q, *J* = 6.9 Hz, 1H), 5.92 (d, *J* = 1.4 Hz, 1H), 5.16 (d, *J* = 1.4 Hz, 1H), 2.53 (s, 3H), 2.29 (s, 3H), 1.85 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.7, 145.6, 144.9, 141.2, 137.7, 136.0, 135.4, 129.2, 128.7, 128.5, 127.5, 126.2, 126.1, 26.5, 21.1, 15.8; IR (film): 3084, 3024, 2919, 2857, 1684, 1604, 1510, 1401, 1356, 1265, 826, 602 cm^{-1}; HRMS (EI): *m/z* calcd. for $C_{20}H_{20}O$: 276.1514; found 276.1513

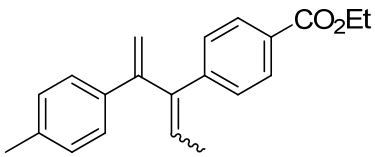


dr (1 : 1.3) **1n**

To a solution of $PdCl_2(dppf)_2$ (22.0 mg, 3 mol %) and potassium phosphoxide (636.8 mg, 3.0 mmol) in DMF (4.0 mL) were added 1-(4-acetylphenyl)prop-1-enyl trifluoromethanesulfonate (308.3 mg, 1.0 mmol), 2-(1-(2-methoxyphenyl)vinyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (286.1 mg, 1.1 mmol) at room temperature. The mixture was stirred at 80 ^{o}C for 12 h. The reaction mixture was quenched with H_2O (20 mL). The aqueous layer was extracted with diethyl ether (3 x 20 mL), and the organic layer was washed with brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. Silica gel fresh column chromatography (hexane) gave (E,Z)-1-(4-(2-(2-methoxyphenyl)penta-1,3-dien-3-yl)phenyl) ethanone (137 mg, 0.47 mmol, 47%) as colorless oil.[4]

(E,Z)-1-(4-(2-(2-Methoxyphenyl)penta-1,3-dien-3-yl)phenyl)ethanone (1n) : R_f = 0.1 (EtOAc:hexane = 1:10); Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.79-7.82 (m, 2H), 7.46-7.49 (m, 2H), 7.16-7.27 (m, 4H), 6.22 (q, *J* = 7.0 Hz, 1H), 5.96 (d, *J* = 2.3 Hz, 1H), 5.37 (d, *J* = 2.3 Hz, 1H), 3.79 (s, 3H), 2.54 (s, 3H),

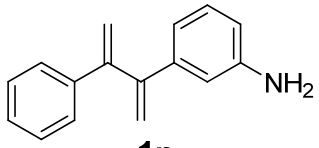
1.88 (d, J = 7.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 198.0, 157.4, 155.6, 146.6, 142.3, 131.0, 130.3, 129.7, 128.3, 128.2, 126.8, 120.7, 120.5, 115.3, 111.3, 55.8, 26.6, 15.9; Data for the minor isomer: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.79-7.82 (m, 2H), 7.46-7.49 (m, 2H), 7.16-7.27 (m, 4H), 5.48 (q, J = 7.1 Hz, 1H), 5.04 (s, 1H), 4.78 (s, 1H), 3.82 (s, 3H), 2.64 (s, 3H), 1.50 (d, J = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.9, 135.2, 129.7, 129.1, 128.7, 128.6, 126.5, 121.2, 116.7, 111.0, 55.4, 26.7, 15.3; IR (film): 3001, 2934, 2909, 2835, 1682, 1601, 1490, 1463, 1245, 1027, 754 cm^{-1}; HRMS (EI): *m/z* calcd. for $C_{20}H_{20}O_2$: 292.1463; found 292.1462



dr (1 : 1.1) **1o**

To a solution of $PdCl_2(dppf)_2$ (22.0 mg, 3 mol %) and potassium phosphoxide (636.8 mg, 3.0 mmol) in DMF (4.0 mL) were added ethyl 4-(1-(trifluoromethylsulfonyloxy)prop-1-enyl)benzoate (338.3 mg, 1.0 mmol), 4,4,5,5-tetramethyl-2-(1-p-tolylvinyl)-1,3,2-dioxaborolane (268.5 mg, 1.1 mmol) at room temperature. The mixture was stirred at 60 ^{o}C for 12 h. The reaction mixture was quenched with H_2O (20 mL). The aqueous layer was extracted with diethyl ether (3 x 20 mL), and the organic layer was washed with brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. Silica gel fresh column chromatography (EtOAc:hexane = 1:30) gave (E,Z)-ethyl 4-(2-p-tolylpenta-1,3-dien-3-yl)benzoate (178 mg, 0.58 mmol, 58%) as colorless oil.[4]

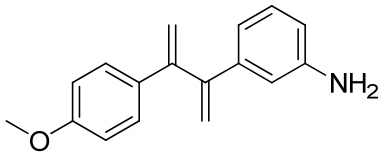
(E,Z)-Ethyl 4-(2-p-tolylpenta-1,3-dien-3-yl)benzoate (1o) : R_f = 0.4 (EtOAc:hexane = 1:30); Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01-8.03 (m, 2H), 7.28-7.32 (m, 2H), 7.22-7.24 (m, 2H), 7.11 (d, J = 7.9 Hz, 2H), 5.81 (q, J = 7.2 Hz, 1H), 5.15-5.16 (m, 1H), 4.83 (d, J = 1.3 Hz, 1H), 4.37 (q, J = 7.2 Hz, 2H), 2.33 (s, 3H), 1.70 (d, J = 7.1 Hz, 3H), 1.39 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.0, 151.7, 143.0, 138.7, 137.4, 130.2, 130.0, 129.8, 129.0, 128.6, 128.7, 126.51, 115.94, 61.3, 21.54, 15.7, 14.8; Data for the minor isomer: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87-7.90 (m, 2H), 7.43-7.46 (m, 2H), 7.28-7.32 (m, 2H), 7.06 (d, J = 8.1 Hz, 2H), 6.38 (q, J = 6.9 Hz, 1H), 5.90 (d, J = 1.5 Hz, 1H), 5.15-5.16 (m, 1H), 4.32 (q, J = 7.2 Hz, 2H), 2.29 (s, 3H), 1.78 (d, J = 6.9 Hz, 3H), 1.35 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.9, 145.8, 145.4, 144.8, 141.8, 138.0, 136.5, 129.2, 129.0, 128.5, 127.6, 126.48, 115.90, 61.1, 21.50, 16.2, 14.7; IR (film): 3086, 3024, 2979, 2921, 2870, 1716, 1606, 1510, 1274, 1103, 826, 775cm^{-1}; HRMS (EI): *m/z* calcd. for $C_{21}H_{22}O_2$: 306.1620; found 306.1619



1p

To a solution of $PdCl_2(dppf)_2$ (22.0 mg, 3 mol %) and potassium phosphoxide (636.8 mg, 3.0 mmol) in DMF (4.0 mL) were added 3-(1-bromovinyl)aniline (198.1 mg, 1.0 mmol), 4,4,5,5-tetramethyl-2-(1-phenylvinyl)-1,3,2-dioxaborolane (253.1 mg, 1.1 mmol) at room temperature. The mixture was stirred at 60 ^oC for 18 h. The reaction mixture was quenched with H_2O (20 mL). The aqueous layer was extracted with diethyl ether (3 x 20 mL), and the organic layer was washed with brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. Silica gel fresh column chromatography (EtOAc:hexane = 1:5) gave 3-(3-phenylbuta-1,3-dien-2-yl)aniline (73 mg, 0.33 mmol, 33%) as brown oil.[4]

3-(3-Phenylbuta-1,3-dien-2-yl)aniline (1p) : R_f = 0.4 (EtOAc:hexane = 1:5); Brown oil. ^1H NMR (400 MHz, $CDCl_3$) δ 7.40-7.37 (m, 2H), 7.29-7.20 (m, 3H), 7.06 (t, *J* = 7.1 Hz, 1H), 6.81 (td, *J* = 1.4, 7.7 Hz, 1H), 6.73 (t, *J* = 2.0 Hz, 1H), 6.57 (ddd, *J* = 0.8, 1.5, 7.9 Hz, 1H), 5.53 (d, *J* = 1.7 Hz, 1H), 5.51 (d, *J* = 1.7 Hz, 1H), 5.30 (d, *J* = 1.7 Hz, 1H), 5.26 (d, *J* = 1.7 Hz, 1H) ; ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.9, 149.8, 146.0, 141.4, 140.2, 129.0, 128.2, 127.5, 127.4, 118.2, 116.2, 116.1, 114.5, 114.4; IR (film): 3447, 3374, 3214, 3082, 3025, 2924, 2852, 1618, 1492, 1444, 904 cm^{-1}; HRMS (EI): *m/z* calcd. for $C_{16}H_{15}N$: 221.1204; found 221.1202

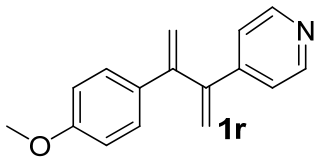


1q

To a solution of $PdCl_2(dppf)_2$ (22.0 mg, 3 mol %) and potassium phosphoxide (636.8 mg, 3.0 mmol) in DMF (4.0 mL) were added 3-(1-bromovinyl)aniline (198.1 mg, 1.0 mmol), 2-(1-(4-methoxyphenyl) vinyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (286.1 mg, 1.1 mmol) at room temperature. The mixture was stirred at 60 ^oC for 18 h. The reaction mixture was quenched with H_2O (20 mL). The aqueous layer was extracted with diethyl ether (3 x 20 mL), and the organic layer was washed with brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. Silica gel fresh column chromatography (EtOAc:hexane = 1:2) gave 3-(3-(4-methoxyphenyl)buta-1,3-dien-2-yl)aniline (78 mg, 0.31 mmol, 31%) as brown oil.[4]

3-(3-(4-Methoxyphenyl)buta-1,3-dien-2-yl)aniline (1q) : R_f = 0.5 (EtOAc:hexane = 1:2); Brown oil. ^1H NMR (400 MHz, $CDCl_3$) δ 7.32 (dt, *J* = 8.9, 2.2 Hz, 2H), 7.05 (t, *J* = 7.8 Hz, 1H), 6.82-6.78 (m, 3H), 6.72 (t, *J* = 2.0 Hz, 1H), 6.56 (ddd, 7.9, 2.3, 0.9 Hz, 1H), 5.51 (d, *J* = 1.8 Hz, 1H), 5.46 (d, *J* = 1.8 Hz,

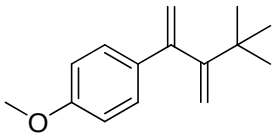
1H), 5.27 (d, J = 1.8 Hz, 1H), 5.22 (d, J = 1.8 Hz, 1H), 3.77 (s, 3H), 3.59 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.1, 150.1, 149.1, 146.1, 141.4, 132.7, 129.1, 128.5, 118.0, 115.8, 114.5, 114.4, 114.2, 1137, 113.5, 55.2; IR (film): 3459, 3370, 3222, 3087, 3005, 2958, 2933, 2836, 1604, 1510, 1249, 1177, 1031, 903, 837, 791 cm^{-1}; HRMS (EI): *m/z* calcd. for $C_{17}H_{17}NO$: 251.1310; found 251.1308



1r

To a solution of $PdCl_2(dppf)_2$ (22.0 mg, 3 mol %) and potassium phosphoxide (636.8 mg, 3.0 mmol) in DMF (4.0 mL) were added 4-(1-bromovinyl)pyridine (184 mg, 1.0 mmol), 2-(1-(4-methoxyphenyl) vinyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (286.1 mg, 1.1 mmol) at room temperature. The mixture was stirred at 60 ^{o}C for 18 h. The reaction mixture was quenched with H_2O (20 mL). The aqueous layer was extracted with diethyl ether (3 x 20 mL), and the organic layer was washed with brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. Silica gel fresh column chromatography (EtOAc:hexane = 1:1) gave 4-(3-(4-Methoxyphenyl)buta-1,3-dien-2-yl)pyridine (145 mg, 0.61 mmol, 61%) as white solid.[4]

4-(3-(4-Methoxyphenyl)buta-1,3-dien-2-yl)pyridine (1r) : R_f = 0.4 (EtOAc:hexane = 1:1); white solid. m.p. 48-51 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (d, J = 6.0 Hz, 2H), 7.29-7.25 (m, 4H), 6.80 (d, J = 8.8 Hz, 2H), 5.71 (d, J = 1.3 Hz, 1H), 5.53 (d, J = 1.4 Hz, 1H), 5.50 (d, J = 1.3 Hz, 1H), 5.25 (d, J = 1.4 Hz, 1H), 3.77 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.4, 149.9, 147.98, 147.9, 147.5, 131.8, 128.5, 122.0, 118.8, 115.4, 113.7, 55.2; IR (film): 3090, 3035, 2836, 1606, 1595, 1510, 1251, 1178, 1033, 835 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{16}H_{15}NO$: 237.1154; found: 237.1151



1s

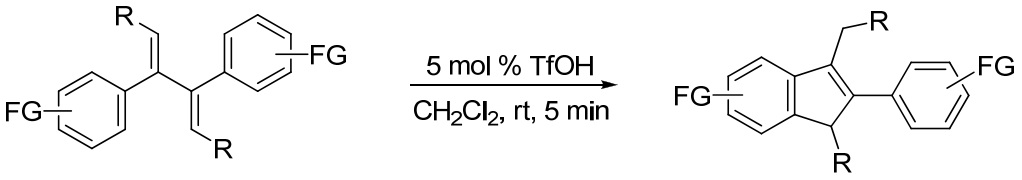
To a solution of $PdCl_2(dppf)_2$ (22.0 mg, 3 mol %) and potassium phosphoxide (636.8 mg, 3.0 mmol) in DMF (4.0 mL) were added 3,3-dimethylbut-1-en-2-yl trifluoromethanesulfonate (232.2 mg, 1.0 mmol), 2-(1-(4-methoxyphenyl)vinyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (286.1 mg, 1.1 mmol) at room temperature. The mixture was stirred at 60 ^{o}C for 18 h. The reaction mixture was quenched with H_2O (20 mL). The aqueous layer was extracted with diethyl ether (3 x 20 mL), and the organic layer was washed with brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. Silica gel fresh column

chromatography (hexane) gave 1-(4,4-Dimethyl-3-methylenepent-1-en-2-yl)-4-methoxybenzene (71 mg, 0.33 mmol, 33%) as colorless oil.[4]

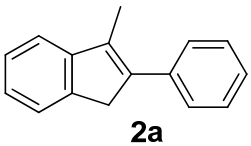
1-(4,4-Dimethyl-3-methylenepent-1-en-2-yl)-4-methoxybenzene (1s) : R_f = 0.3 (hexane); Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (dt, J = 8.89, 2.96 Hz, 2H), 6.83 (dt, J = 8.88, 2.96 Hz, 2H), 5.27 (d, J = 2.08 Hz, 1H), 5.20 (d, J = 2.00 Hz, 1H), 4.91 (d, J = 2.04 Hz, 2H), 3.81 (s, 3H), 0.99 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.1, 158.9, 150.7, 133.9, 127.8, 113.4, 113.0, 112.3, 55.2, 35.8, 30.4; IR (film): 3086, 2964, 2904, 2868, 2835, 1606, 1509, 1463, 1289, 1249, 1178, 1037, 902, 837 cm^{-1}; HRMS (EI): *m/z* calcd. for $C_{15}H_{20}O$: 216.1514; found 216.1511

Typical experimental procedures for synthesis of indenes via Brønsted acid-catalyzed cyclization of 2,3-diaryl-1,3-dienes:

1) TfOH-catalyzed cyclization of symmetric 2,3-diaryl-1,3-dienes:

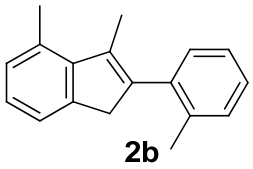


To a solution of buta-1,3-diene-2,3-diyldibenzene (103.1 mg, 0.5 mmol) in CH_2Cl_2 (5 mL) was added trifluoromethanesulfonic acid (2 μL, 0.025 mmol, 5 mol %) at room temperature. The mixture was stirred at room temperature for 5 min. The solvent was removed under reduced pressure. The crude product was purified by silica gel fresh column chromatography (hexane) to give 3-methyl-2-phenyl-1*H*-indene (94.9 mg, 0.46 mmol, 92%).



2a

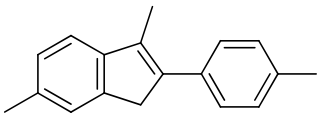
3-Methyl-2-phenyl-1*H*-indene (2a)[5] : R_f = 0.2 (hexane); Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46-7.50 (m, 3H), 7.37-7.43 (m, 3H), 7.33 (t, J = 7.3 Hz, 1H), 7.28 (tt, J = 7.0 Hz, 1.4 Hz, 1H), 7.21 (td, J = 7.4 Hz, 1.4 Hz, 1H), 3.73 (d, J = 2.0 Hz, 2H), 2.30 (t, J = 2.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.9, 142.9, 140.7, 138.0, 135.1, 128.8, 128.7, 127.1, 126.9, 125.2, 123.8, 119.6, 41.4, 12.4; IR (film): 3052, 3020, 2925, 2856, 1599, 1493, 1466, 1390, 1208, 760, 699 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{16}H_{14}$: 206.1096; found: 206.1096



2b

To a solution of 2,2'-(buta-1,3-diene-2,3-diyl)bis(methylbenzene) (117.2 mg, 0.5 mmol) in CH_2Cl_2 (5 mL) was added trifluoromethanesulfonic acid (2 μL, 0.025 mmol, 5 mol %) at room temperature. The mixture was stirred at room temperature for 5 min. The solvent was removed under reduced pressure. The crude product was purified by silica gel fresh column chromatography (hexane) to give 3,4-dimethyl-2-*o*-tolyl-1*H*-indene (103.1 mg, 0.44 mmol, 88%).

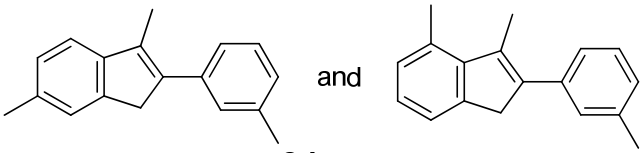
3,4-Dimethyl-2-*o*-tolyl-1*H*-indene (2b) : R_f = 0.3 (hexane); White solid. m.p. 61-63 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ 7.29 (d, J = 6.58 Hz, 1H), 7.18-7.27 (m, 3H), 7.11-7.14 (m, 1H), 7.05-7.09 (m, 2H), 3.54 (d, J = 2.0 Hz, 2H), 2.64 (s, 3H), 2.21 (s, 3H), 2.12 (t, J = 2.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.5, 144.2, 142.5, 138.4, 137.1, 136.9, 131.5, 130.4, 129.9, 129.6, 127.5, 125.9, 124.9, 121.8, 42.9, 20.5, 20.4, 15.8; IR (film): 3040, 3013, 2958, 2922, 1489, 1453, 1390, 1262, 1059, 908, 760, 728 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{18}H_{18}$:234.1409; found: 234.1407



2c

To a solution of 4,4'-(buta-1,3-diene-2,3-diyl)bis(methylbenzene) (117.2 mg, 0.5 mmol) in CH_2Cl_2 (5 mL) was added trifluoromethanesulfonic acid (2 μL, 0.025 mmol, 5 mol %) at room temperature. The mixture was stirred at room temperature for 5 min. The solvent was removed under reduced pressure. The crude product was purified by silica gel fresh column chromatography (hexane) to give 3,6-dimethyl-2-*p*-tolyl-1*H*-indene (102 mg, 0.44 mmol, 87%).

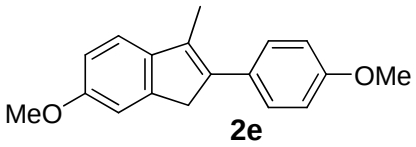
3,6-Dimethyl-2-*p*-tolyl-1*H*-indene (2c)[6] : R_f = 0.2 (hexane); Colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 7.36-7.38 (m, 2H), 7.27 (s, 1H), 7.20-7.23 (m, 3H), 7.13 (d, J = 7.4 Hz, 1H), 3.67 (d, J = 1.9 Hz, 2H), 2.41 (s, 1H), 2.38 (s, 1H), 2.27 (t, J = 2.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.5, 143.1, 139.6, 136.6, 135.3, 134.7, 134.4, 129.5, 128.5, 127.5, 124.7, 119.1, 41.2, 21.9, 21.6, 12.4; IR (film): 3019, 2920, 2858, 1644, 1447, 1391, 908, 867, 817, 733, 545 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{18}H_{18}$: 234.1409; found: 234.1406



2d (1:1.4)

To a solution of 3,3'-(buta-1,3-diene-2,3-diyl)bis(methylbenzene) (117.2 mg, 0.5 mmol) in CH_2Cl_2 (5 mL) was added trifluoromethanesulfonic acid (2 μL, 0.025 mmol, 5 mol %) at room temperature. The mixture was stirred at room temperature for 5 min. The solvent was removed under reduced pressure. The crude product was purified by silica gel fresh column chromatography (hexane) to give 3,5-dimethyl-2-*m*-tolyl-1*H*-indene and 3,7-dimethyl-2-*m*-tolyl-1*H*-indene (100 mg, 0.43 mmol, 85%).

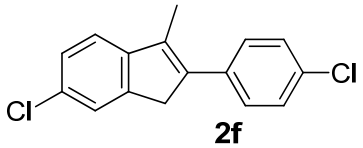
Mixture of 3,5-dimethyl-2-*m*-tolyl-1*H*-indene and 3,7-dimethyl-2-*m*-tolyl-1*H*-indene (2d) : R_f = 0.3 (hexane); Colorless oil. Data for the major isomer; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.92-7.24 (m, 7H), 3.57 (d, J = 1.3 Hz, 2H), 2.33 (s, 3H), 2.30 (s, 3H), 2.18 (t, J = 2.0, 3H); Data for the minor isomer; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.92-7.24 (m, 7H), 3.51 (d, J = 1.9 Hz, 2H), 2.31 (s, 3H), 2.29 (s, 3H), 2.20 (t, J = 2.0 Hz, 3H); Mixture of product; ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.3, 147.7, 141.5, 141.2, 140.6, 140.0, 138.4, 138.2, 136.4, 135.3, 134.9, 133.0, 129.40, 129.39, 128.7, 127.9, 127.3, 126.5, 126.0, 125.90, 125.87, 123.5, 120.3, 117.3, 41.1, 40.5, 22.09, 22.07, 19.0, 12.6, 12.4; IR (film): 3038, 2919, 2859, 1689, 1603, 1474, 1379, 1038, 910, 782, 703 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{18}H_{18}$: 234.1409; found: 234.1409



2e

To a solution of 4,4'-(buta-1,3-diene-2,3-diyl)bis(methoxybenzene) (133.2 mg, 0.5 mmol) in CH_2Cl_2 (5 mL) was added trifluoromethanesulfonic acid (2 μL, 0.025 mmol, 5 mol %) at room temperature. The mixture was stirred at room temperature for 5 min. The solvent was removed under reduced pressure. The crude product was purified by silica gel fresh column chromatography (hexane) to give 6-methoxy-2-(4-methoxyphenyl)-3-methyl-1*H*-indene (117 mg, 0.44 mmol, 88%).

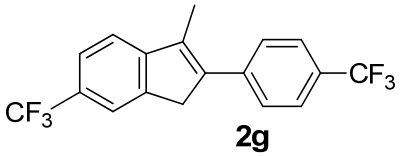
6-Methoxy-2-(4-methoxyphenyl)-3-methyl-1*H*-indene (2e)[7] : R_f = 0.3 (hexane); White solid. m.p. 89-92 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38-7.41 (m, 2H), 7.23 (d, J = 8.3 Hz, 1H), 7.05-7.06 (m, 1H), 6.93-6.96 (m, 2H), 6.86-6.90 (m, 1H), 3.841 (s, 3H), 3.837 (s, 3H), 3.66 (d, J = 1.8 Hz, 2H), 2.26 (t, J = 2.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.6, 158.3, 144.4, 141.4, 138.2, 133.4, 130.8, 129.5, 119.6, 114.3, 112.2, 110.4, 56.0, 56.7, 41.3, 12.5; IR (film): 2998, 2935, 2835, 1707, 1606, 1510, 1481, 1284, 1250, 1180, 1033, 831 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{18}H_{18}O_2$: 266.1307; found: 266.1307



2f

To a solution of 4,4'-(buta-1,3-diene-2,3-diyl)bis(chlorobenzene) (137.6 mg, 0.5 mmol) in CH_2Cl_2 (5 mL) was added trifluoromethanesulfonic acid (2 μL, 0.025 mmol, 5 mol %) at room temperature. The mixture was stirred at room temperature for 5 min. The solvent was removed under reduced pressure. The crude product was purified by silica gel fresh column chromatography (hexane) to give 6-chloro-2-(4-chlorophenyl)-3-methyl-1*H*-indene (110 mg, 0.4 mmol, 80%).

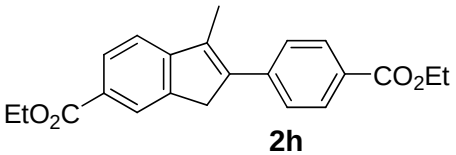
6-Chloro-2-(4-chlorophenyl)-3-methyl-1*H*-indene (2f)[6] : R_f = 0.3 (hexane); White solid. m.p. 111-114 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ 7.41 (s, 1H), 7.37 (s, 4H), 7.30 (dd, J = 8.1 Hz, 1.6 Hz, 1H), 7.24-7.26 (m, 1H), 3.66 (d, J = 1.6 Hz, 2H), 3.38 (t, J = 2.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ146.2, 144.2, 139.8, 135.9, 135.1, 133.1, 131.4, 129.8, 129.1, 127.1, 124.2, 120.4, 41.1, 12.4; IR (film): 2923, 2853, 1568, 1491, 1398, 1179, 1092, 890, 856, 816 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{16}H_{12}Cl_2$: 274.0316; found: 274.0317



2g

To a solution of 4,4'-(buta-1,3-diene-2,3-diyl)bis((trifluoromethyl)benzene) (171.1 mg, 0.5 mmol) in CH_2Cl_2 (5 mL) was added trifluoromethanesulfonic acid (2 μL, 0.025 mmol, 5 mol %) at room temperature. The mixture was stirred at room temperature for 5 min. The solvent was removed under reduced pressure. The crude product was purified by silica gel fresh column chromatography (hexane) to give 3-methyl-6-(trifluoromethyl)-2-(4-(trifluoromethyl)phenyl)-1*H*-indene (145 mg, 0.425 mmol, 85%).

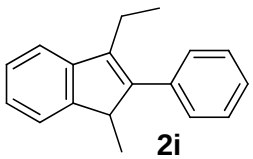
3-Methyl-6-(trifluoromethyl)-2-(4-(trifluoromethyl)phenyl)-1*H*-indene (2g) : R_f = 0.3 (hexane); White solid. m.p. 62-64 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ 7.71 (d, J = 9.5 Hz, 2H), 7.68 (s, 1H), 7.63 (d, J = 8.1 Hz, 1H), 7.58 (d, J = 8.2 Hz, 2H), 7.47 (d, J = 8.0 Hz, 1H), 3.80 (d, J = 1.9 Hz, 2H), 2.34 (t, J = 2.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.6, 142.9, 142.1, 140.7, 136.5, 128.9, 125.9 (q, J = 0.04 Hz), 124.4 (q, J = 0.04 Hz), 120.7 (q, J = 0.04 Hz), 119.9, 41.3, 12.4; IR (film): 3055, 2926, 2864, 1716, 1616, 1436, 1324, 1167, 1117, 1070, 901, 836 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{18}H_{12}F_6$: 342.0843; found: 342.0840



2h

To a solution of diethyl 4,4'-(buta-1,3-diene-2,3-diyl)dibenzoate (175.2 mg, 0.5 mmol) in CH_2Cl_2 (5 mL) was added trifluoromethanesulfonic acid (2 μL, 0.025 mmol, 5 mol %) at room temperature. The mixture was stirred at 70 ^oC for 30 min. The solvent was removed under reduced pressure. The crude product was purified by silica gel fresh column chromatography (EtOAc: hexane = 1:10) to give ethyl 2-(4-(ethoxycarbonyl)phenyl)-3-methyl-1*H*-indene-6-carboxylate (170 mg, 0.485 mmol, 97%).

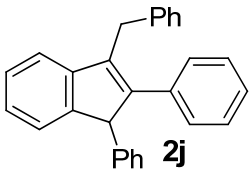
Ethyl 2-(4-(ethoxycarbonyl)phenyl)-3-methyl-1*H*-indene-6-carboxylate (2h) : R_f = 0.2 (EtOAc: hexane = 1:10); Yellow solid. m.p. 72-76 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, J = 0.6 Hz, 1H), 8.07-8.11 (m, 3H), 7.54-7.57 (m, 2H), 7.43 (d, J = 8.0 Hz, 1H), 4.41 (q, J = 7.1 Hz, 2H), 4.40 (q, J = 7.1 Hz, 2H), 3.81 (d, J = 1.8 Hz, 2H), 2.35 (t, J = 2.0 Hz, 3H), 1.422 (t, J = 7.2 Hz, 3H), 1.417 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.6, 166.8, 152.0, 143.2, 142.6, 141.8, 136.9, 136.9, 130.1, 129.3, 129.1, 128.5, 127.7, 124.7, 119.5, 61.4, 61.2, 41.2, 14.82, 14.77, 12.6; IR (film): 3056, 2981, 2937, 2906, 1714, 1606, 1366, 1275, 1178, 1105, 1022, 772 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{22}H_{22}O_4$: 350.1518; found: 350.1516



2i

To a solution of (2Z,4Z)-hexa-2,4-diene-3,4-diyldibenzene (117.2 mg, 0.5 mmol) in CH_2Cl_2 (5 mL) was added trifluoromethanesulfonic acid (2 μL, 0.025 mmol, 5 mol %) at room temperature. The mixture was stirred at room temperature for 5 min. The solvent was removed under reduced pressure. The crude product was purified by silica gel fresh column chromatography (hexane) to give 3-ethyl-1-methyl-2-phenyl-1*H*-indene (111 mg, 0.475 mmol, 95%).

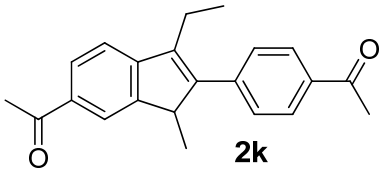
3-Ethyl-1-methyl-2-phenyl-1*H*-indene (2i)[8] : R_f = 0.3 (hexane); Colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 7.40-7.47 (m, 4H), 7.28-7.38 (m, 4H), 7.22-7.25 (m, 1H), 3.85 (q, J = 7.5 Hz, 1H), 2.68 (q, J = 7.6 Hz, 2H), 1.28 (t, J = 7.5 Hz, 3H), 1.20 (d, J = 7.5 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.1, 146.9, 145.2, 139.7, 137.1, 129.3, 128.7, 127.1, 126.9, 125.1, 123.1, 119.9, 46.4, 19.6, 16.7, 14.4; IR (film): 3018, 2965, 2928, 2871, 1493, 1465, 743, 698 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{18}H_{18}$: 234.1409; found: 234.1405



2j

To a solution of (1E,3E)-buta-1,3-diene-1,2,3,4-tetrayltetrabenzene (179.2 mg, 0.5 mmol) in CH_2Cl_2 (5 mL) was added trifluoromethanesulfonic acid (10 μL, 0.125 mmol, 25 mol %) at room temperature. The mixture was stirred at room temperature for 20 min. The solvent was removed under reduced pressure. The crude product was purified by silica gel fresh column chromatography (hexane) to give 3-benzyl-1,2-diphenyl-1*H*-indene (165 mg, 0.46 mmol, 92%).

3-Benzyl-1,2-diphenyl-1*H*-indene (2j)[9] : R_f= 0.2 (hexane); White solid. m.p. 117-119 ^oC; ^1H NMR (300 MHz, $CDCl_3$) δ 7.09-7.32 (m, 19H), 5.05 (s, 1H), 4.12 (d, J = 16.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.2, 147.5, 144.9, 139.7, 139.5, 137.4, 135.9, 129.4, 128.71, 128.65, 128.6, 128.3, 128.2, 127.8, 127.0, 126.9, 126.6, 126.1, 125.4, 123.7, 120.4, 58.2, 32.2; IR (film): 3060, 3025, 2923, 1601, 1494, 1467, 1452, 1442 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{28}H_{22}$: 358.1721; found: 358.1694

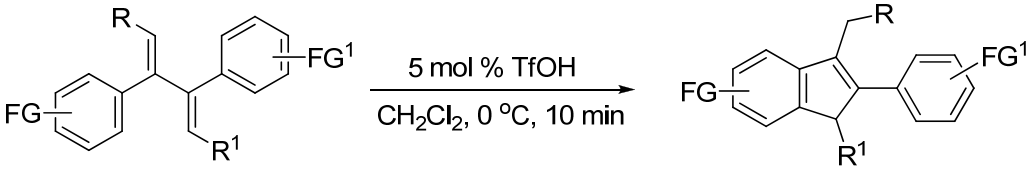


2k

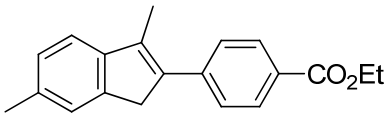
To a solution of 1,1'-(4,4'-(hexa-2,4-diene-3,4-diyl)bis(4,1-phenylene))diethanone (159.2 mg, 0.5 mmol) in CH_2Cl_2 (5 mL) was added trifluoromethanesulfonic acid (10 μL, 0.125 mmol, 25 mol %) at room temperature. The mixture was stirred at 70 ^oC for 16 h. The solvent was removed under reduced pressure. The crude product was purified by silica gel fresh column chromatography (EtOAc:hexane = 1:5) to give 1-(4-(6-acetyl-3-ethyl-1-methyl-1*H*-inden-2-yl)phenyl)ethanone (119 mg, 0.375 mmol, 75%).

1-(4-(6-Acetyl-3-ethyl-1-methyl-1*H*-inden-2-yl)phenyl)ethanone (2k) : R_f= 0.1 (EtOAc:hexane = 1:5); light yellow solid. m.p. 105-111 ^oC; ^1H NMR (300 MHz, $CDCl_3$) δ 8.068-8.072 (m, 1H), 8.03-8.06 (m, 2H), 7.99 (dd, J = 1.3 Hz, 8.0 Hz, 1H), 7.44-7.49 (m, 3H), 3.95 (q, J =7.6 Hz, 1H), 2.72 (q, J = 7.7 Hz, 2H), 2.66 (s, 3H), 2.65 (S, 3H), 1.30 (t, J = 7.6 Hz, 3H), 1.23 (d, J = 7.5 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 198.2, 197.6, 149.3, 148.7, 141.0, 135.7, 134.4, 128.9, 128.7, 128.6, 128.1, 125.9, 122.4, 119.5, 46.1, 26.8, 26.6, 19.3, 16.0, 13.8; IR (film): 3054, 2967, 2929, 2873, 1680, 1603, 1426, 1358, 1267, 1250, 957, 829 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{22}H_{22}O_2$: 318.1620; found: 318.1621

2) TfOH-catalyzed cyclization of unsymmetric 2,3-diaryl-1,3-dienes:

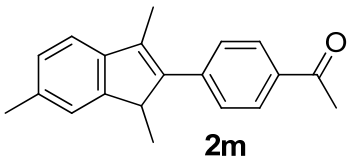


To a solution of ethyl 4-(3-*p*-tolylbuta-1,3-dien-2-yl)benzoate (146.1 mg, 0.5 mmol) in CH_2Cl_2 (5 mL) was added trifluoromethanesulfonic acid (2 μL, 0.025 mmol, 5 mol%) at 0 ^oC. The mixture was stirred at 0 ^oC for 10 min. The solvent was removed under reduced pressure. The crude product was purified by silica gel fresh column chromatography (EtOAc:hexane = 1:10) to give ethyl 4-(3,6-dimethyl-1*H*-inden-2-yl)benzoate (141.7 mg, 0.49 mmol, 97%).



2l

Ethyl 4-(3,6-dimethyl-1*H*-inden-2-yl)benzoate (2l) : R_f = 0.2 (EtOAc:hexane = 1:10); Light yellow solid. m.p. 53-58 ^oC; ^1H NMR (300 MHz, $CDCl_3$) δ 8.05-8.09 (m, 2H), 7.52-7.54 (m, 2H), 7.25-7.30 (m, 2H), 7.16 (d, *J* = 7.2 Hz, 1H), 4.39 (q, *J* = 7.1 Hz, 2H), 3.72 (d, *J* = 2.0 Hz, 2H), 2.42 (s, 3H), 2.32 (t, *J* = 2.1 Hz, 3H), 1.41 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.6, 144.7, 142.8, 142.8, 142.3, 138.1, 136.9, 135.2, 129.6, 128.1, 127.9, 127.3, 124.4, 119.2, 60.9, 40.5, 21.6, 14.4, 12.3; IR (film): 2979, 2917, 2864, 1713, 1605, 1446, 1366, 1274, 1183, 1107, 1021, 772 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{20}H_{20}O_2$: 292.1463; found: 292.1465

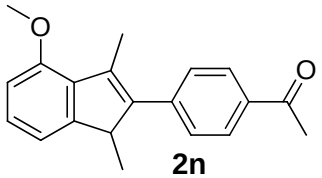


2m

To a solution of 1-(4-(2-p-tolylpenta-1,3-dien-3-yl)phenyl)ethanone (138.2 mg, 0.5 mmol) in CH_2Cl_2 (5 mL) was added trifluoromethanesulfonic acid (2 μL, 0.025 mmol, 5 mol%) at 0 ^oC. The mixture was stirred at 0 ^oC for 10 min. The solvent was removed under reduced pressure. The crude product was purified by silica gel fresh column chromatography (EtOAc:hexane = 1:5) to give 1-(4-(1,3,6-trimethyl-1*H*-inden-2-yl)phenyl)ethanone (126 mg, 0.455 mmol, 91%).

1-(4-(1,3,6-Trimethyl-1*H*-inden-2-yl)phenyl)ethanone (2m) : R_f = 0.4 (EtOAc:hexane = 1:5); Yellow solid. m.p. 98-100 ^oC; ^1H NMR (300 MHz, $CDCl_3$) δ 8.02 (d, *J* = 8.3 Hz, 2H), 7.46 (d, *J* = 8.3 Hz, 2H), 7.27-7.29 (m, 2H), 7.17 (d, *J* = 7.8 Hz, 1H), 3.89 (qd, *J* = 7.4, 1.7 Hz, 1H), 2.64 (s, 3H), 2.44 (s, 3H), 2.27 (d, *J* = 2.0 Hz, 3H), 1.20 (d, *J* = 7.5 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.7, 148.6, 144.5, 142.9,

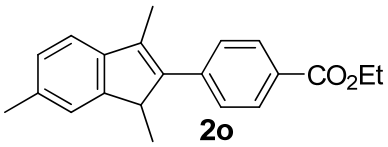
141.8, 135.5, 135.4, 134.9, 129.0, 128.4, 127.5, 123.5, 119.2, 45.3, 26.6, 21.6, 16.4, 11.9; IR (film): 3003, 2964, 2925, 2864, 1681, 1601, 1405, 1357, 1268, 956, 816, 596 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{20}H_{20}O$: 276.1514; found: 276.1518



2n

To a solution of 1-(4-(2-(2-methoxyphenyl)penta-1,3-dien-3-yl)phenyl)ethanone (146.2 mg, 0.5 mmol) in CH_2Cl_2 (5 mL) was added trifluoromethanesulfonic acid (2 μL, 0.025 mmol, 5 mol%) at 0 ^oC. The mixture was stirred at 0 ^oC for 30 min. The solvent was removed under reduced pressure. The crude product was purified by silica gel fresh column chromatography (EtOAc:hexane = 1:10) to give 1-(4-(4-methoxy-1,3-dimethyl-1*H*-inden-2-yl)phenyl)ethanone (130 mg, 0.445 mmol, 89%).

1-(4-(4-Methoxy-1,3-dimethyl-1*H*-inden-2-yl)phenyl)ethanone (2n) : R_f = 0.2 (EtOAc:hexane = 1:10); Colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 7.99-8.02 (m, 2H), 7.41-7.44 (m, 2H), 7.19-7.25 (m, 1H), 7.08 (d, *J* = 7.5 Hz, 1H), 6.83 (d, *J* = 8.1 Hz, 1H), 3.88 (m, 4H), 2.63 (s, 3H), 2.44 (d, *J* = 2.0 Hz, 3H), 1.15 (d, *J* = 7.5 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.2, 155.6, 151.3, 144.5, 142.3, 136.5, 135.3, 133.2, 130.0, 129.7, 128.8, 127.1, 116.1, 115.7, 109.8, 55.8, 46.3, 27.0, 16.9, 15.4; IR (film): 3060, 3001, 2962, 2927, 2868, 2835, 1680, 1600, 1479, 1259, 1103, 743 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{20}H_{20}O_2$: 292.1463; found: 292.1465

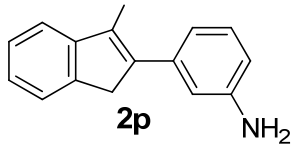


2o

To a solution of ethyl 4-(2-*p*-tolylpenta-1,3-dien-3-yl)benzoate (153.2 mg, 0.5 mmol) in CH_2Cl_2 (5 mL) was added trifluoromethanesulfonic acid (2 μL, 0.025 mmol, 5 mol%) at 0 ^oC. The mixture was stirred at 0 ^oC for 30 min. The solvent was removed under reduced pressure. The crude product was purified by silica gel fresh column chromatography (EtOAc:hexane = 1:10) to give ethyl 4-(1,3,6-trimethyl-1*H*-inden-2-yl)benzoate (150 mg, 0.49 mmol, 98%).

Ethyl 4-(1,3,6-trimethyl-1*H*-inden-2-yl)benzoate (2o) : R_f = 0.3 (EtOAc:hexane = 1:10); Colorless oil. ^1H NMR (300 MHz, $CDCl_3$) δ 8.08-8.10 (m, 2H), 7.41-7.44 (m, 2H), 7.26-7.29 (m, 2H), 7.16 (d, *J* = 7.8 Hz, 1H), 4.40 (q, *J* = 7.1 Hz, 2H), 3.88 (qd, *J* = 7.4, 1.8 Hz, 1H), 2.44 (s, 3H), 2.26 (d, *J* = 2.0 Hz, 3H), 1.41 (t, *J* = 7.1 Hz, 3H), 1.19 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.6, 148.6, 144.7, 143.0,

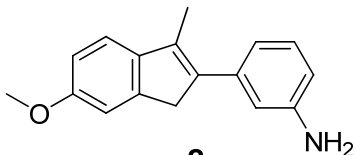
141.0, 135.3, 135.2, 129.5, 128.8, 128.2, 127.5, 123.5, 119.2, 60.9, 45.4, 21.6, 16.4, 14.4, 11.9; IR (film): 2975, 2927, 2806, 1715, 1602, 1276, 1179, 1108, 1022, 859, 815, 777 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{21}H_{22}O_2$: 306.1620; found: 306.1624



2p

To a solution of 3-(3-phenylbuta-1,3-dien-2-yl)aniline (110.7 mg, 0.5 mmol) in CH_2Cl_2 (5 mL) was added trifluoromethanesulfonic acid (10 μL, 0.125 mmol, 25 mol%) at room temperature. The mixture was stirred at room temperature for 10 min. The reaction mixture was quenched with sat-$NaHCO_3$ (20 mL). The aqueous layer was extracted with diethyl ether (3 x 20 mL), and the organic layer was washed with brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. The crude product was purified by silica gel fresh column chromatography (EtOAc:hexane = 1:5) to give 3-(3-methyl-1H-inden-2-yl)aniline (108.4 mg, 0.49 mmol, 98%).

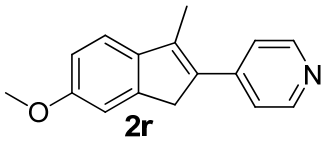
3-(3-Methyl-1H-inden-2-yl)aniline (2p) : R_f = 0.5 (EtOAc:hexane = 1:5); Brown oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (d, *J* = 7.3 Hz, 1H), 7.38-7.36 (m, 1H), 7.212 (td, *J* = 7.2, 1.5 Hz, 1H), 7.207 (t, *J* = 7.8 Hz, 1H), 6.89 (dt, 7.6, 1.0 Hz, 1H), 6.80 (t, *J* = 2.0 Hz, 1H), 6.63 (ddd, *J* = 8.0, 2.4, 0.9 Hz, 1H), 3.70 (d, *J* = 2.1 Hz, 2H), 3.70 (NH_2, 2H), 2.30 (t, *J* = 2.0 Hz, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.0, 146.3, 144.2, 140.9, 138.8, 138.3, 134.2, 129.3, 119.4, 118.8, 114.7, 113.4, 111.9, 110.0, 55.6, 40.9, 12.1; IR (film): 3458, 3375, 3214, 3045, 2961, 2924, 2853, 1603, 1456, 1261, 1094 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{16}H_{15}N$: 221.1204; found: 221.1205



2q

To a solution of 3-(3-(4-methoxyphenyl)buta-1,3-dien-2-yl)aniline (125.7 mg, 0.5 mmol) in CH_2Cl_2 (5 mL) was added trifluoromethanesulfonic acid (10 μL, 0.125 mmol, 25 mol%) at room temperature. The mixture was stirred at room temperature for 10 min. The reaction mixture was quenched with sat-$NaHCO_3$ (20 mL). The aqueous layer was extracted with diethyl ether (3 x 20 mL), and the organic layer was washed with brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. The crude product was purified by silica gel fresh column chromatography (EtOAc:hexane = 1:2) to give 3-(6-methoxy-3-methyl-1*H*-inden-2-yl)aniline (121.9 mg, 0.49 mmol, 97%).

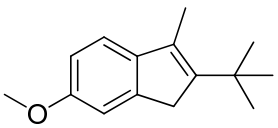
3-(6-Methoxy-3-methyl-1*H*-inden-2-yl)aniline (2q) : R_f = 0.3 (EtOAc:hexane = 1:2); Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.12 (t, *J* = 7.8 Hz, 2H), 7.19 (t, *J* = 7.8 Hz, 1H), 7.06 (d, *J* = 2.2 Hz, 1H), 6.90-6.86 (m , 2H), 6.78 (t, *J* = 6,8 Hz, 1H), 6.61 (ddd, *J* = 7.9, 2.3, 0.9 Hz, 1H), 3.85 (s, 3H), 3.70 (s, 2H), 3.67 (d, *J* = 1.8 Hz, 2H), 2.27 (t, *J* = 2.1 Hz, 3H) ; ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.0, 146.3, 144.2, 140.9, 138.8, 138.3, 134.2, 129.3, 119.4, 118.8, 114.7, 113.4, 111.9, 110.0, 55.6, 40.9, 12.1; IR (film): 3447, 3368, 3220, 2933, 2854, 2833, 1602, 1480, 1288, 1244, 1027 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{17}H_{17}NO$: 251.1310; found: 251.1312



2r

To a solution of 4-(3-(4-methoxyphenyl)buta-1,3-dien-2-yl)pyridine (118.6 mg, 0.5 mmol) in CH_2Cl_2 (5 mL) was added trifluoromethanesulfonic acid (52 μL, 0.65 mmol, 1.3 equiv.) at room temperature. The mixture was stirred at room temperature for 30 min. The reaction mixture was quenched with sat-$NaHCO_3$ (20 mL). The aqueous layer was extracted with diethyl ether (3 x 20 mL), and the organic layer was washed with brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. The crude product was purified by silica gel fresh column chromatography (EtOAc:hexane = 1:1) to give 4-(6-methoxy-3-methyl-1*H*-inden-2-yl)pyridine (106.7 mg, 0.45 mmol, 90%).

4-(6-Methoxy-3-methyl-1*H*-inden-2-yl)pyridine (2r): R_f = 0.3 (EtOAc:hexane = 1:1); Yellow solid. m.p. 89-91 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.60 (d, *J* = 5.6 Hz, 2H), 7.35-7.33 (m, 2H), 7.32 (d, *J* = 8.3 Hz, 1H), 7.10 (s, 1H), 6.91 (dd, *J* = 8.3 Hz, 2.4 Hz, 1H), 3.86 (s, 3H), 3.70 (d, *J* = 1.8 Hz, 2H), 2.35 (t, *J* = 2.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.8, 149.9, 144.9, 144.3, 140.0, 138.8, 134.9, 122.3, 120.3, 112.4, 110.0, 55.6, 40.0, 12.4; IR (film): 3033, 2937, 2834, 1594, 1483, 1288, 1235, 815 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{16}H_{15}NO$: 237.1154; found: 237.1152



2s

To a solution of 1-(4,4-dimethyl-3-methylenepent-1-en-2-yl)-4-methoxybenzene (108.2 mg, 0.5 mmol) in CH_2Cl_2 (5 mL) was added trifluoromethanesulfonic acid (2 μL, 0.025 mmol, 5 mol%) at 0 ^{o}C. The mixture was stirred at 0 ^{o}C for 10 min. The solvent was removed under reduced pressure. The crude product was purified by silica gel fresh column chromatography (EtOAc:hexane = 1:30) to give 2-*tert*-butyl-6-methoxy-3-methyl-1*H*-indene (107.1 mg, 0.495 mmol, 99%).

2-*tert*-Butyl-6-methoxy-3-methyl-1*H*-indene (2s) : R_f = 0.3 (EtOAc:hexane = 1:30); White solid. m.p. 52-54 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.12 (d, *J* = 8.3 Hz, 1H), 6.99 (t, *J* = 1.0 Hz, 1H), 6.83 (dd, *J* = 8.3, 2.4 Hz, 1H), 3.83 (s, 3H), 3.35 (d, *J* = 1.9 Hz, 2H), 2.18 (t, *J* = 2.0 Hz, 3H), 1.30 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 157.3, 147.8, 143.3, 141.8, 130.3, 118.1, 111.5, 109.8, 55.7, 39.6, 33.7, 30.7, 12.4; IR (film): 2951, 2856, 2829, 1461, 1395, 1271, 1148, 1027, 846, 814 cm^{-1}; HRMS (EI): *m/z* calcd for $C_{15}H_{20}O$: 216.1514; found: 216.1516

References

1. Lee, P. H.; Seomoon, D.; Lee, K. *Org. Lett.* **2005***, 7*, 343.

2. Shimizu, M.; Kurahashi, T.; Shimono, K.; Tanaka, K.; Nagao, I.; Kiyomoto, S.-I.; Hiyama, T. *Chem. Asian J*. **2007**, *2*, 1400.

3. Sun, X.; Izumi, K. -J.; Hu, C. -Q.; Lin, G. -Q. *Chin. J. Chem.* **2006**, *24*, 430.

4. Takagi, J.; Takahashi, K.; Ishiyama, T.; Miyaura, N. *J. Am. Chem. Soc.* **2002**, *124*, 8001.

5. Usanov, D. L.; Yamamoto, H. *Org. Lett*. **2012**, *14*, 414.

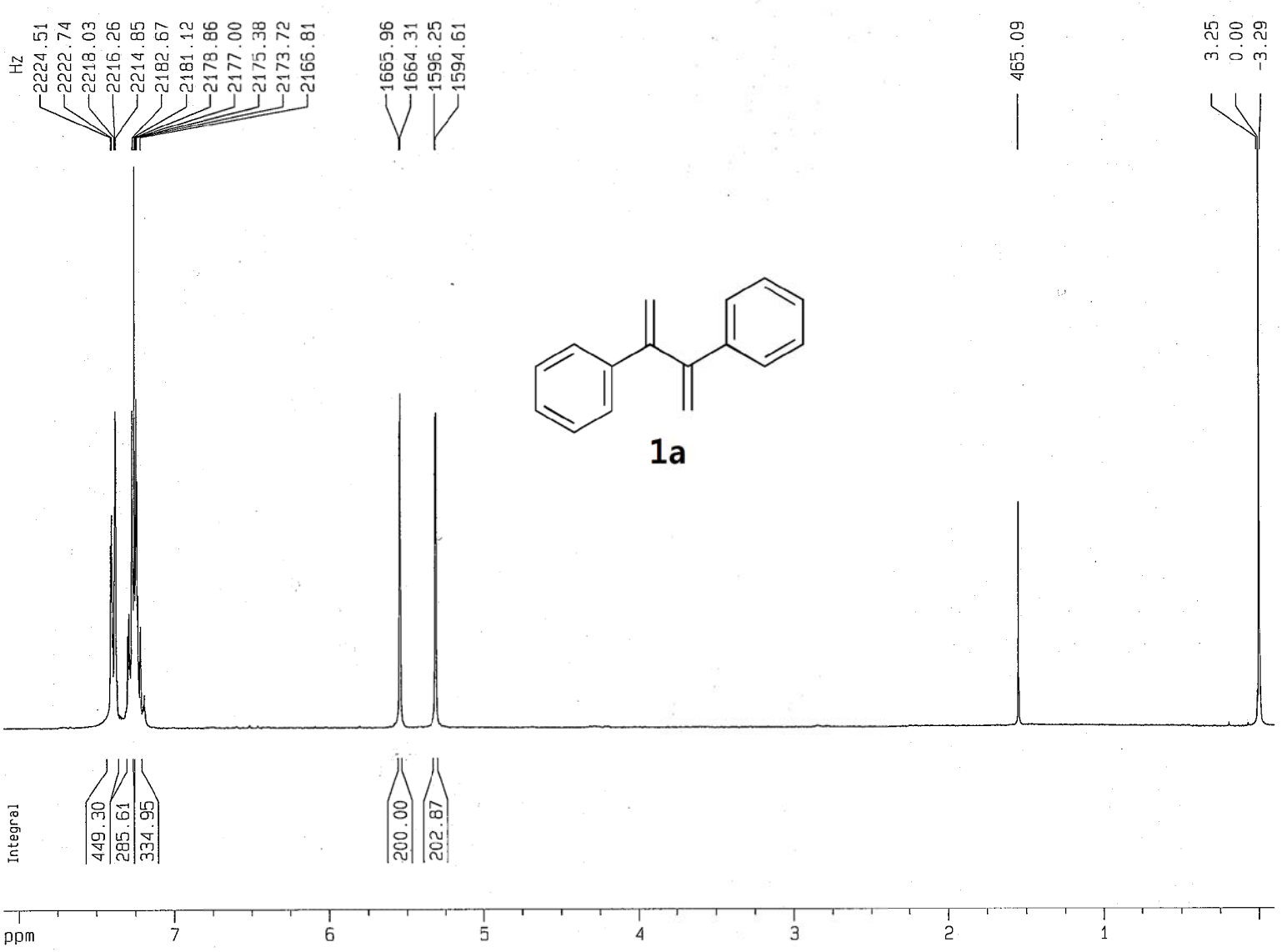
6. Heindel, N. D.; Lemke, S. M.; Mosher, W. A. *J. Org. Chem.* **1966**, *31*, 2680.

7. Ogata, Y.; Kawasaki, A.; Haba, M.; Tsujino, T. *J. Org. Chem*. **1977**, *42*, 2423.

8. Dinulescu, I. G.; Staicu, S.; Dumitru, A.; Ghenciulescu, A.; Chiraleu, F.; Avram, M. *Revue Roumaine de Chimie* **1985**, *30*, 585.

9. Xi, Z.; Guo, R.; Mito, S.; Yan, H.; Kanno, K.-i.; Nakajima, K.; Takahashi, T. *J. Org. Chem.* **2003**, *68*, 1252.

EDH-699



1a

Current Data Parameters

NAME	feb-11
EXPNO	103
PROCNO	1

F2 - Acquisition Parameters

Date_	20110213
Time	14.44
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	1149.4
DW	81.000 usec
DE	6.00 usec
TE	0.0 K
D1	1.00000000 sec

======== CHANNEL f1 ========

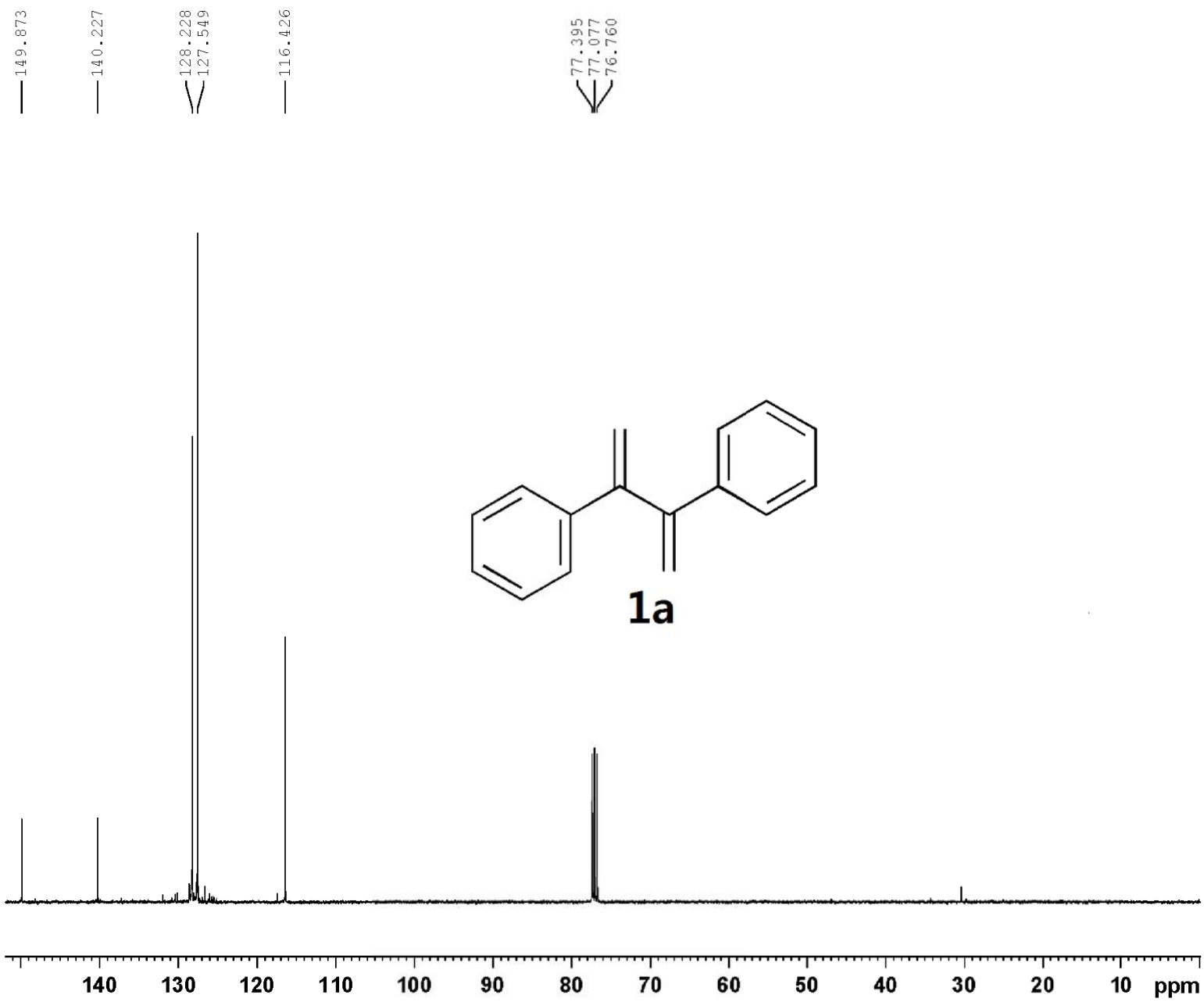
NUC1	1H
P1	10.20 usec
PL1	0.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

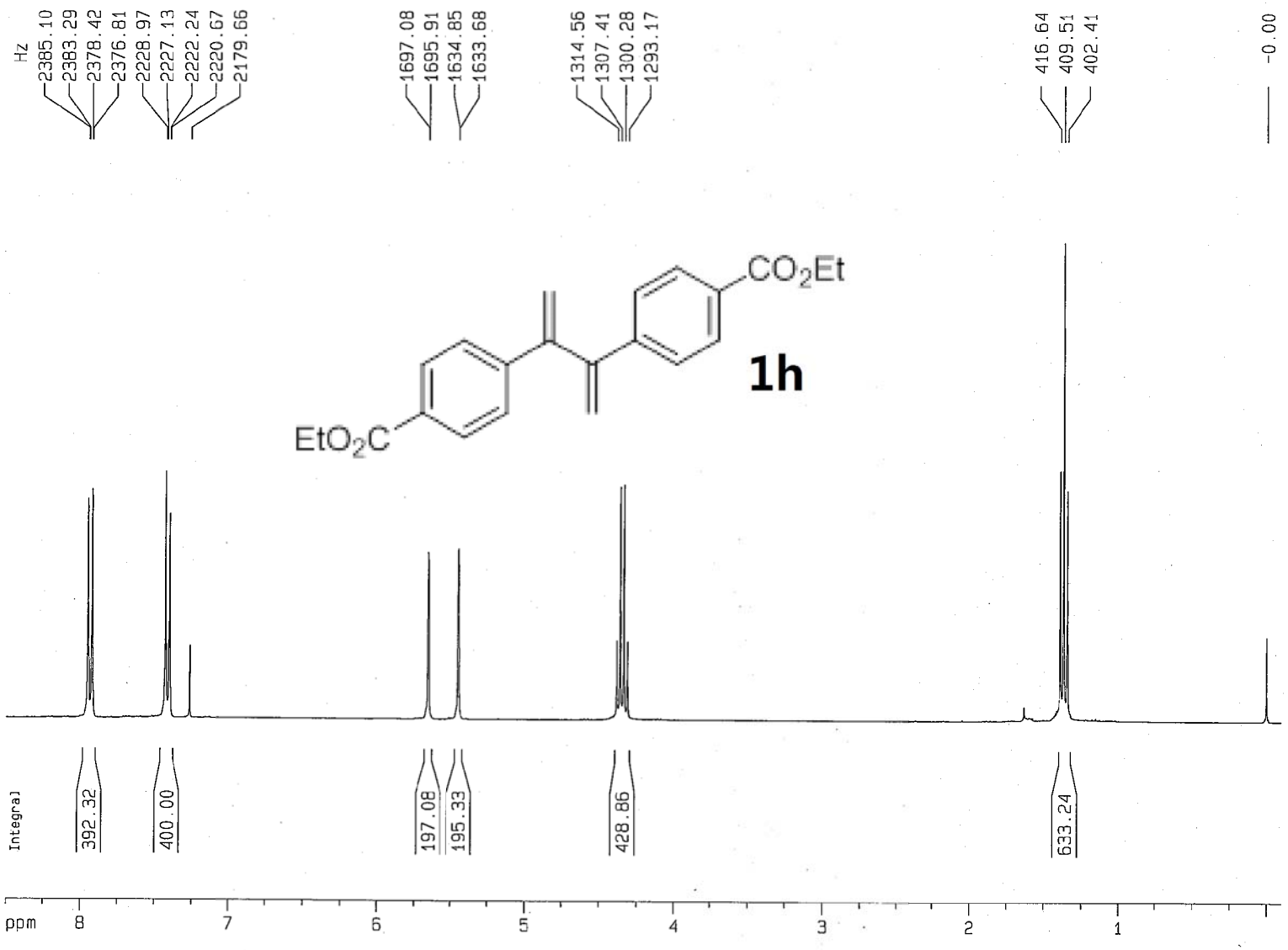
SI	32768
SF	300.1300067 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	21.00 cm
CY	16.00 cm
F1P	8.100 ppm
F1	2431.05 Hz
F2P	-0.100 ppm
F2	-30.01 Hz
PPMCM	0.39048 ppm/cm
HZCM	117.19363 Hz/cm


149.873
140.227
128.228
127.549
116.426
77.395
77.077
76.760
Current Data Parameters
NAME KYR-052
EXPNO 2
PROCNO 1
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1a
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

PSJ-536



Current Data Parameters

NAME	oct-11
EXPNO	137
PROCNO	1

F2 - Acquisition Parameters

Date_	20111020
Time	22.47
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	362
DW	81.000 usec
DE	6.00 usec
TE	0.0 K
D1	1.00000000 sec

======== CHANNEL f1 ========

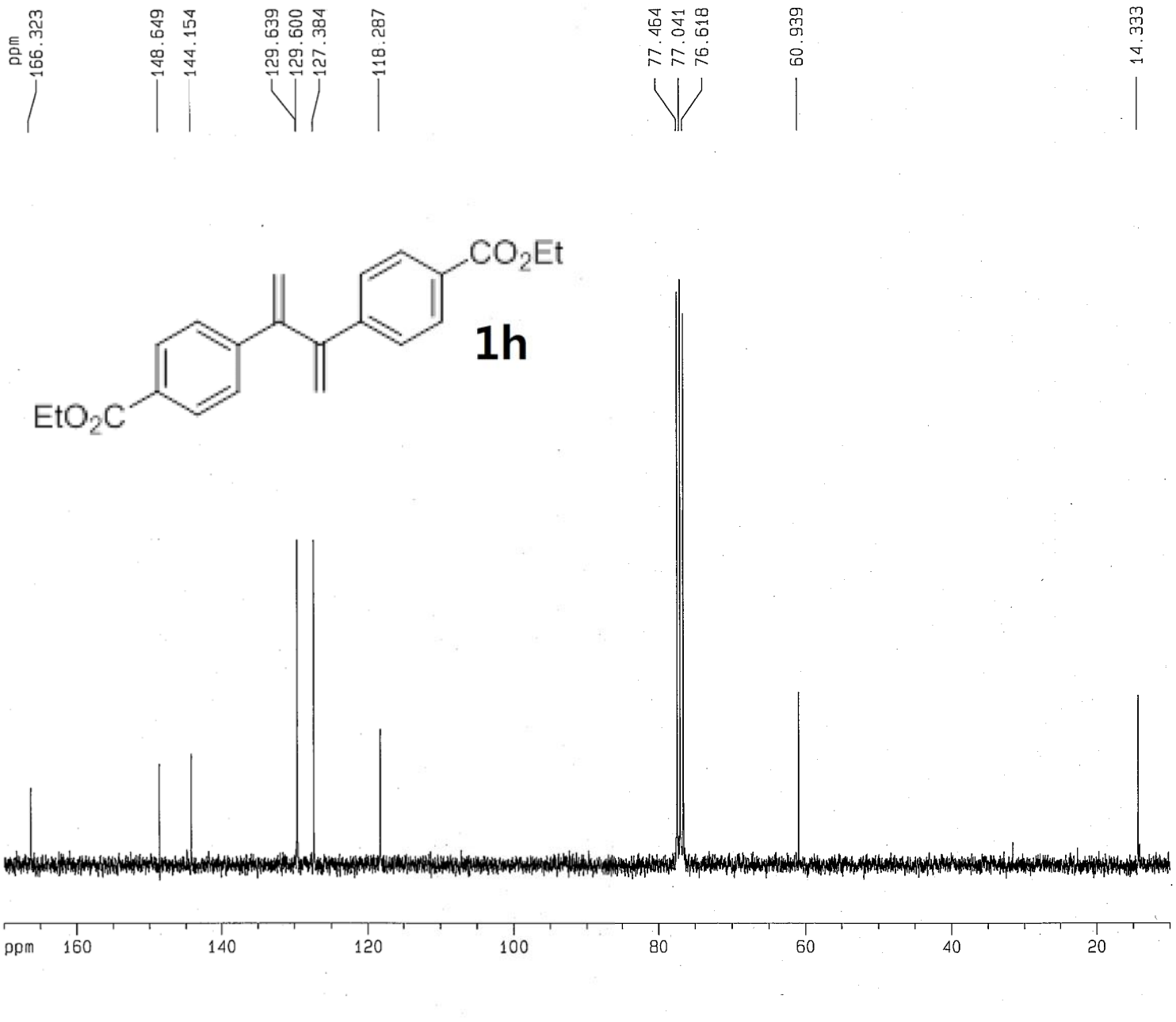
NUC1	1H
P1	10.20 usec
PL1	0.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	32768
SF	300.1300057 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

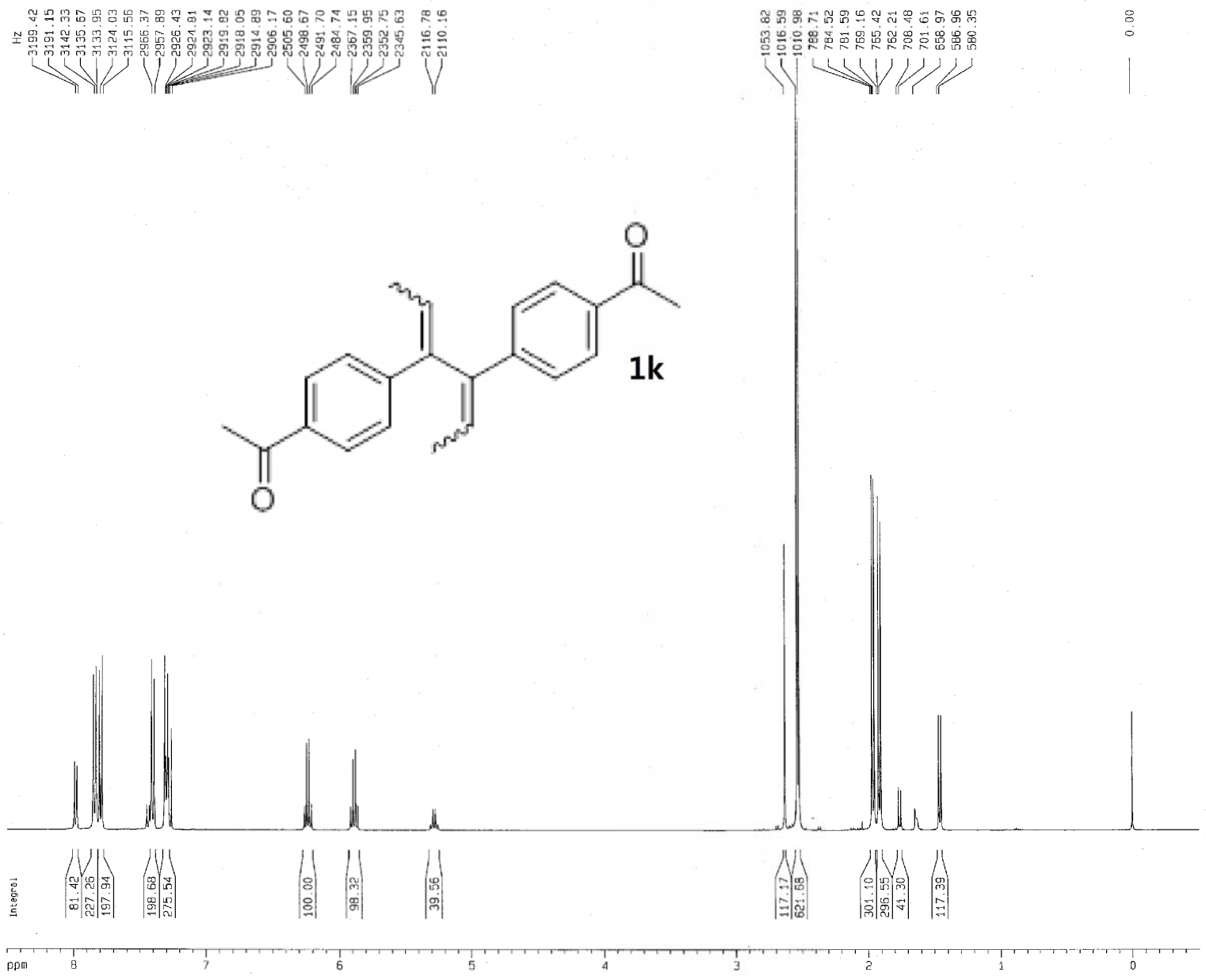
1D NMR plot parameters

CX	21.00 cm
CY	8.00 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.100 ppm
F2	-30.01 Hz
PPMCM	0.40952 ppm/cm
HZCM	122.91039 Hz/cm




```
Current Data Parameters
NAME                  oct-11
EXPNO                    144
PROCNO                     1

F2 - Acquisition Parameters
Date_              20111021
Time                  13.10
INSTRUM               spect
PROBHD   5 mm QNP  1H/1
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      177
DS                        0
SWH               22675.736 Hz
FIDRES            0.346004 Hz
AQ                1.4451188 sec
RG                  10321.3
DW                   22.050 usec
DE                    6.00 usec
TE                     0.0 K
D1              2.00000000 sec
d11             0.03000000 sec
d12             0.00002000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                     8.00 usec
PL1                    2.00 dB
SFO1            75.4760505 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                100.00 usec
PL2                    0.00 dB
PL12                  20.00 dB
PL13                  22.00 dB
SFO2           300.1312005 MHz

F2 - Processing parameters
SI                      32768
SF              75.4677485 MHz
WDW                        EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                     20.00 cm
CY                     10.00 cm
F1P                  170.000 ppm
F1                 12829.52 Hz
F2P                   10.000 ppm
F2                   754.68 Hz
PPMCM                8.00000 ppm/cm
HZCM               603.74200 Hz/cm
```

PSJ-494



Current Data Parameters

NAME	sep-11
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date_	20110905
Time	17.05
INSTRUM	spect
PROBHD	5 mm Dual 13C/
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	0
SWH	8223.685 Hz
FIDRES	0.250957 Hz
AQ	1.9923444 sec
RG	256
DW	60.800 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	11.00 usec
PL1	-2.00 dB
SFO1	400.1324710 MHz

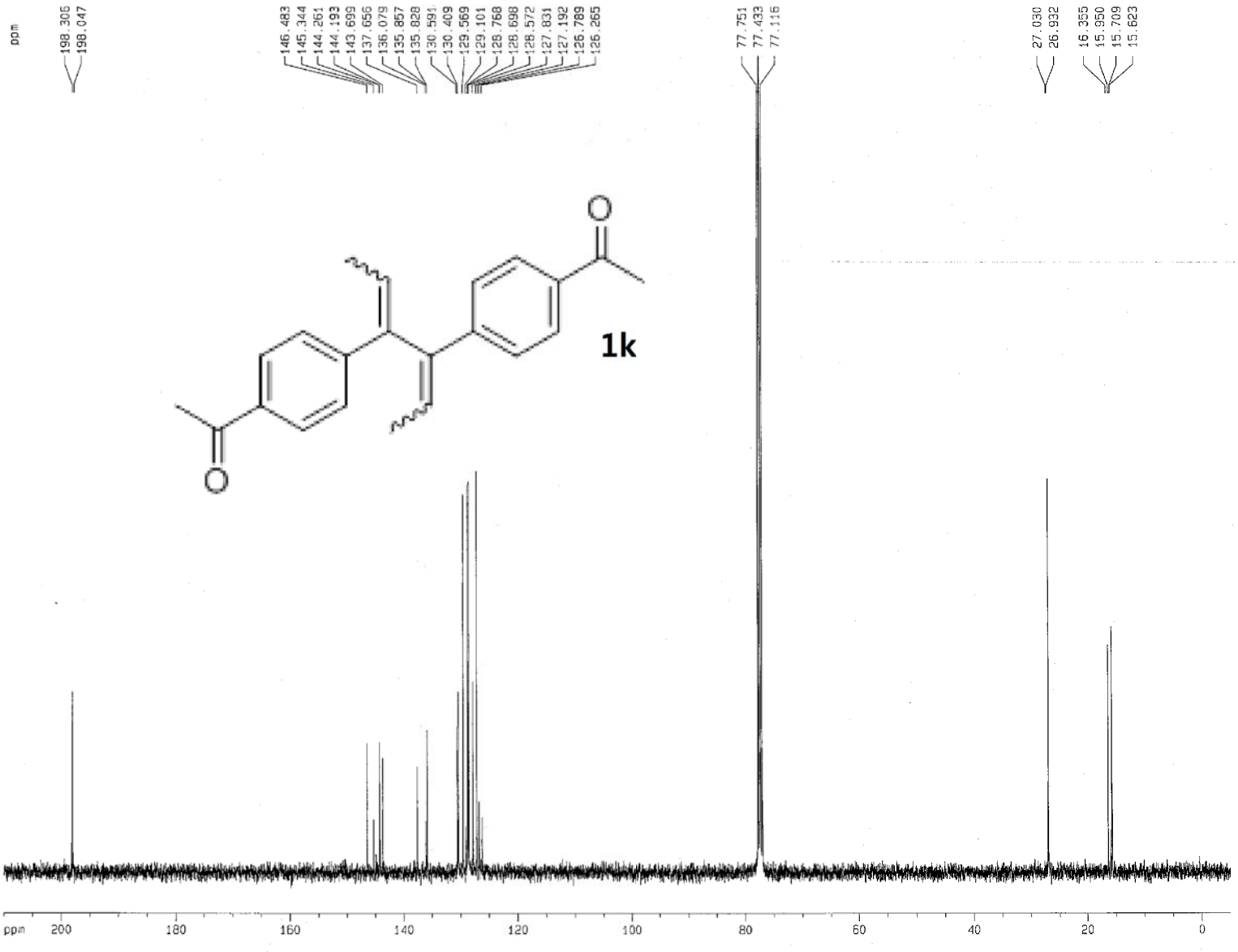
F2 - Processing parameters

SI	16384
SF	400.1300084 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	30.00 cm
CY	20.00 cm
F1P	8.500 ppm
F1	3401.10 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.30000 ppm/cm
HZCM	120.03900 Hz/cm

PSJ-494



Current Data Parameters

NAME	sep-11
EXPNO	4
PROCNO	1

F2 - Acquisition Parameters

Date_	20110905
Time	17.21
INSTRUM	spect
PROBHD	5 mm Dual 13C/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	800
DS	2
SWH	31847.133 Hz
FIDRES	0.485949 Hz
AQ	1.0289652 sec
RG	3649.1
DW	15.700 usec
DE	6.00 usec
TE	298.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	10.50 usec
PL1	-1.00 dB
SFO1	100.6254358 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	105.00 usec
PL2	-2.00 dB
PL12	18.00 dB
PL13	20.00 dB
SFO2	400.1324708 MHz

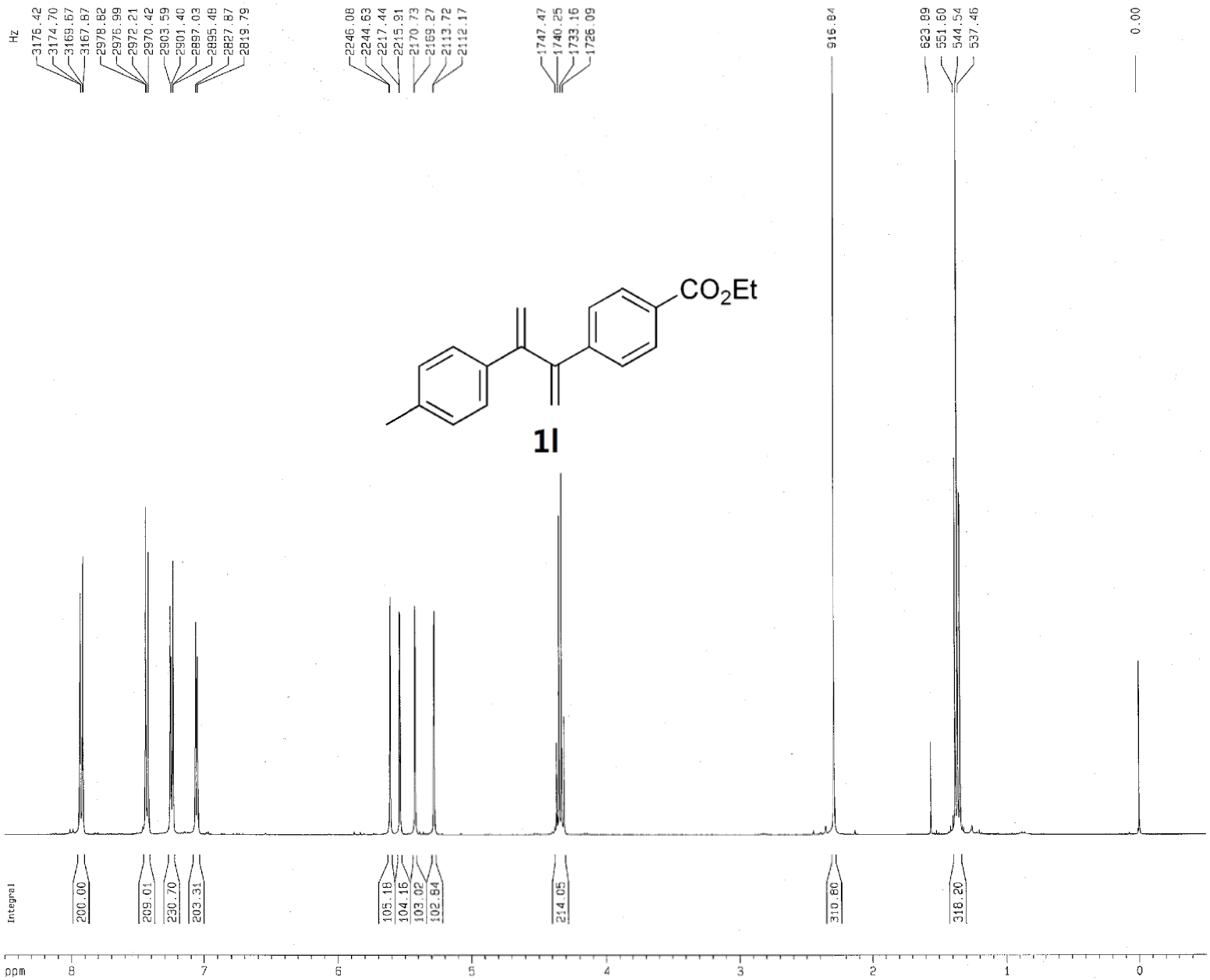
F2 - Processing parameters

SI	32768
SF	100.6127290 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	30.00 cm
CY	30.00 cm
F1P	210.000 ppm
F1	21128.67 Hz
F2P	-5.000 ppm
F2	-503.06 Hz
PPMCM	7.16667 ppm/cm
HZCM	721.05786 Hz/cm

PSJ-537



Current Data Parameters

NAME	sep-11
EXPNO	49
PROCNO	1

F2 - Acquisition Parameters

Date_	20111024
Time	11.37
INSTRUM	spect
PROBHD	5 mm Dual 13C/
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	0
SWH	8223.685 Hz
FIDRES	0.250967 Hz
AQ	1.9923444 sec
RG	203.2
DW	60.800 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	11.00 usec
PL1	-2.00 dB
SFO1	400.1324710 MHz

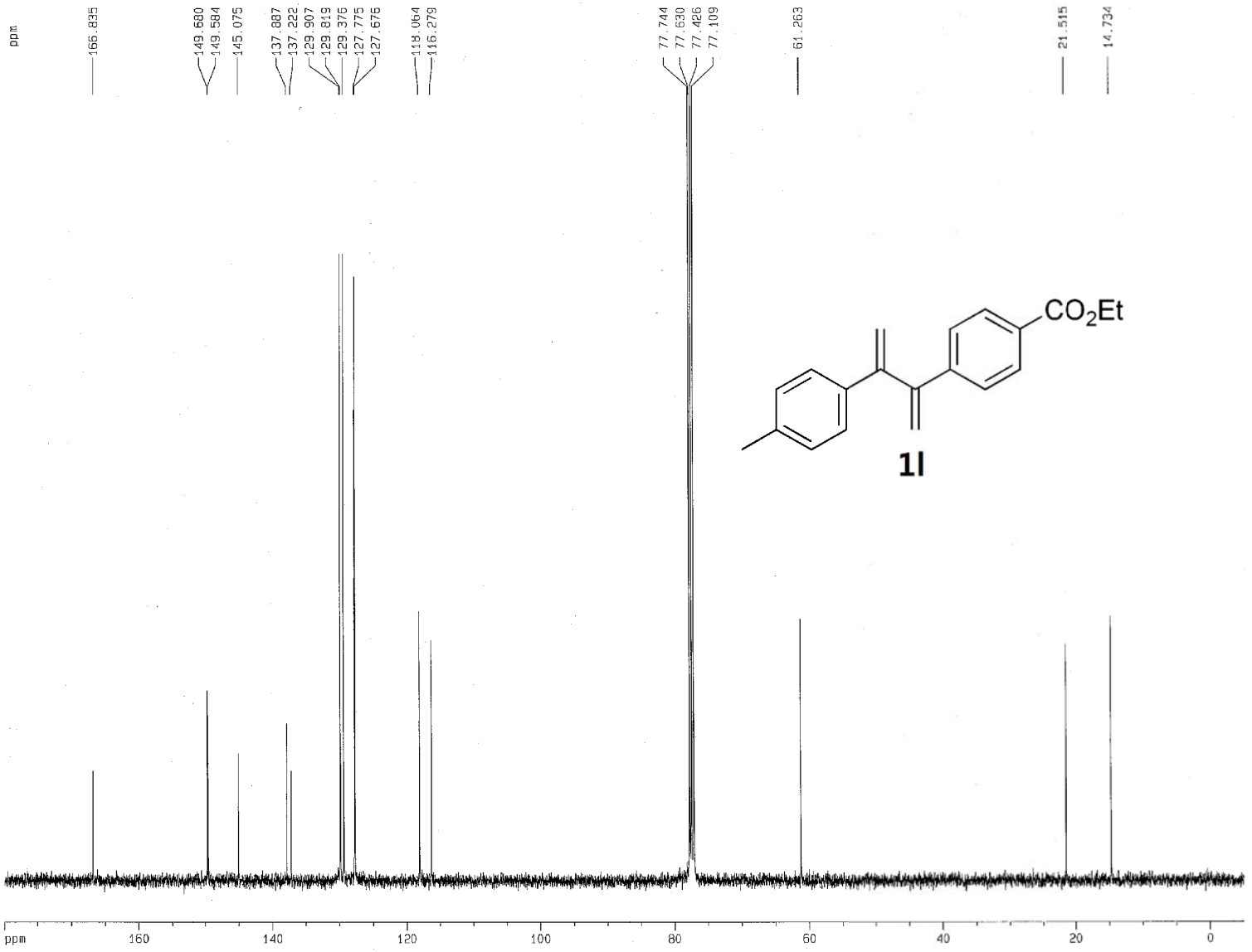
F2 - Processing parameters

SI	16384
SF	400.1300130 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

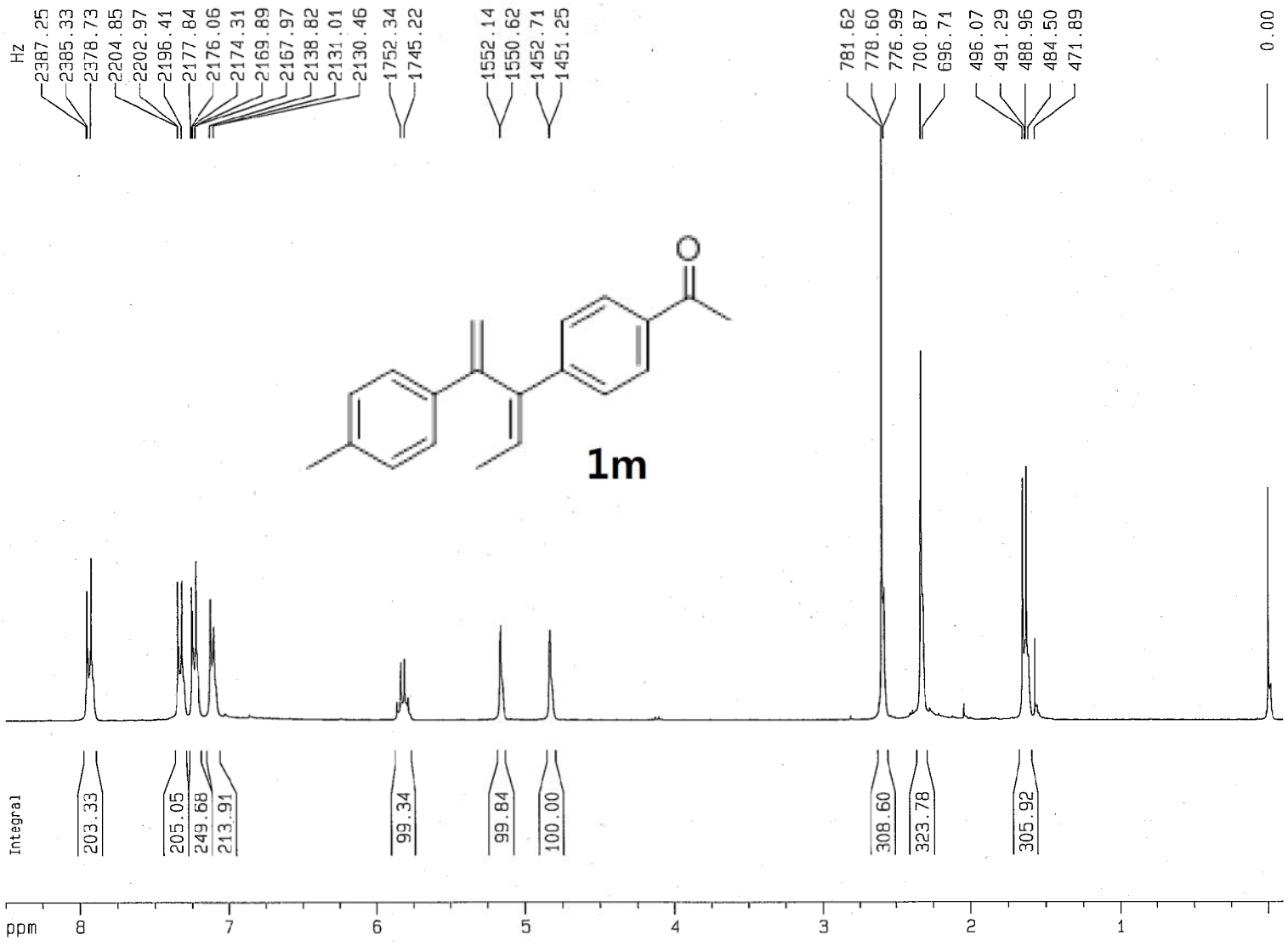
CX	30.00 cm
CY	20.00 cm
F1P	8.500 ppm
F1	3401.10 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.30000 ppm/cm
HZCM	120.03900 Hz/cm

PSJ-537




```
Current Data Parameters
NAME                  sep-11
EXPNO                     50
PROCNO                     1

F2 - Acquisition Parameters
Date_              20111024
Time                  12.31
INSTRUM               spect
PROBHD    5 mm Dual 13C/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1024
DS                       2
SWH              31847.133 Hz
FIDRES           0.485949 Hz
AQ               1.0289652 sec
RG                 3649.1
DW                15.700 usec
DE                 6.00 usec
TE                298.2 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.50 usec
PL1                -1.00 dB
SFO1         100.6254358 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             105.00 usec
PL2                -2.00 dB
PL12               18.00 dB
PL13               20.00 dB
SFO2         400.1324708 MHz

F2 - Processing parameters
SI                   32768
SF           100.6127290 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                30.00 cm
CY                30.00 cm
F1P              180.000 ppm
F1             18110.29 Hz
F2P               -5.000 ppm
F2              -503.06 Hz
PPMCM          6.16667 ppm/cm
HZCM         620.44519 Hz/cm
```

PSJ-514-1



Current Data Parameters

NAME	sep-11
EXPNO	188
PROCNO	1

F2 - Acquisition Parameters

Date_	20110929
Time	17.18
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	574.7
DW	81.000 usec
DE	6.00 usec
TE	0.0 K
D1	1.00000000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	10.20 usec
PL1	0.00 dB
SFO1	300.1318534 MHz

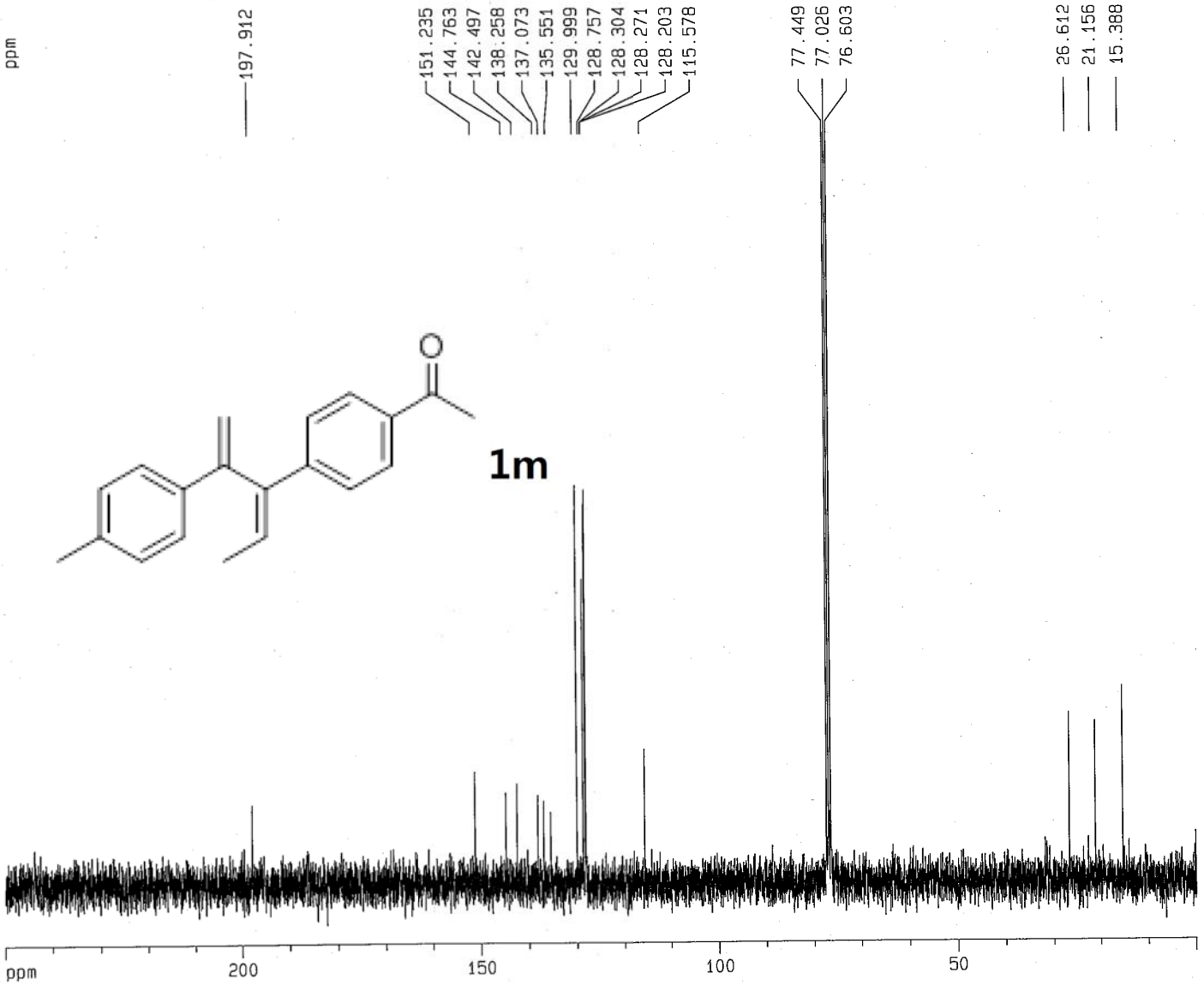
F2 - Processing parameters

SI	32768
SF	300.1300082 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	21.00 cm
CY	12.00 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.100 ppm
F2	-30.01 Hz
PPMCM	0.40952 ppm/cm
HZCM	122.91039 Hz/cm

PSJ-514-1



Current Data Parameters

NAME	sep-11
EXPNO	191
PROCNO	1

F2 - Acquisition Parameters

Date_	20110929
Time	19.20
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	299
DS	0
SWH	22675.736 Hz
FIDRES	0.346004 Hz
AQ	1.4451188 sec
RG	9195.2
DW	22.050 usec
DE	6.00 usec
TE	0.0 K
D1	2.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	8.00 usec
PL1	2.00 dB
SFO1	75.4760505 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	0.00 dB
PL12	20.00 dB
PL13	22.00 dB
SFO2	300.1312005 MHz

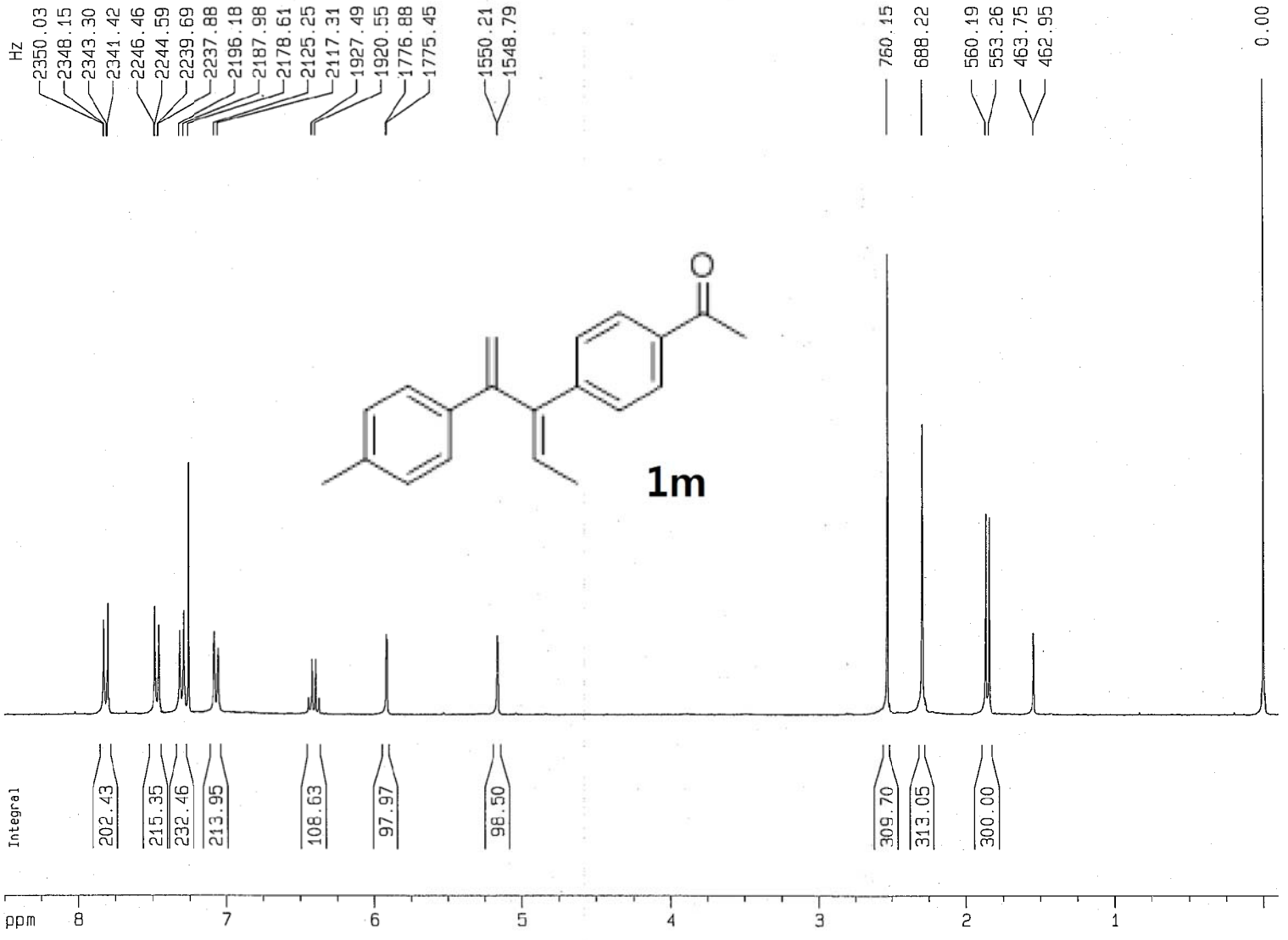
F2 - Processing parameters

SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	25.00 cm
F1P	250.000 ppm
F1	18866.94 Hz
F2P	-0.100 ppm
F2	-7.55 Hz
PPMCM	12.50500 ppm/cm
HZCM	943.72424 Hz/cm

PSJ-516-2



Current Data Parameters

NAME	sep-11
EXPNO	206
PROCNO	1

F2 - Acquisition Parameters

Date_	20110930
Time	16.56
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	1149.4
DW	81.000 usec
DE	6.00 usec
TE	0.0 K
D1	1.00000000 sec

======== CHANNEL f1 ========

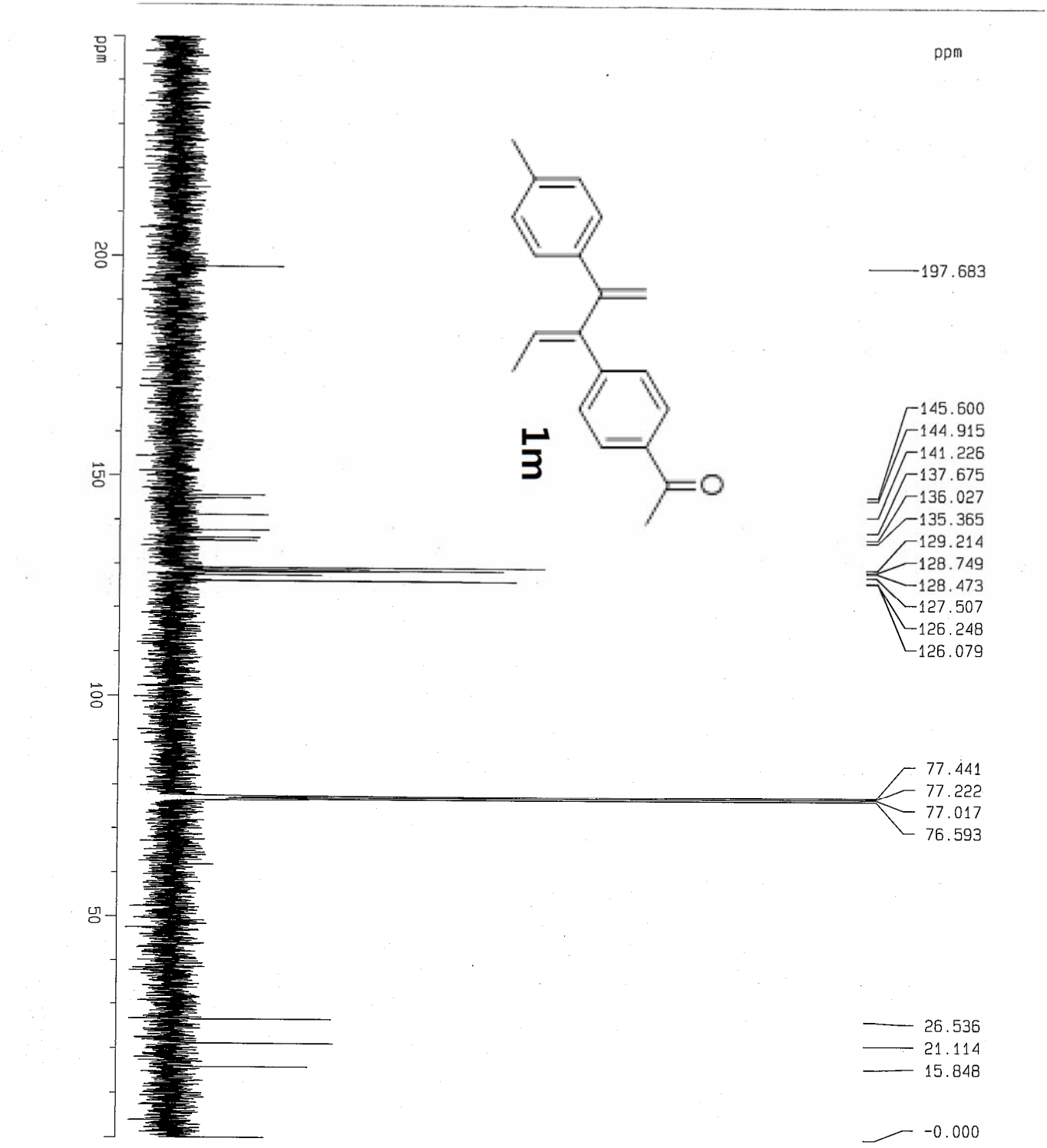
NUC1	1H
P1	10.20 usec
PL1	0.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	32768
SF	300.1300071 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

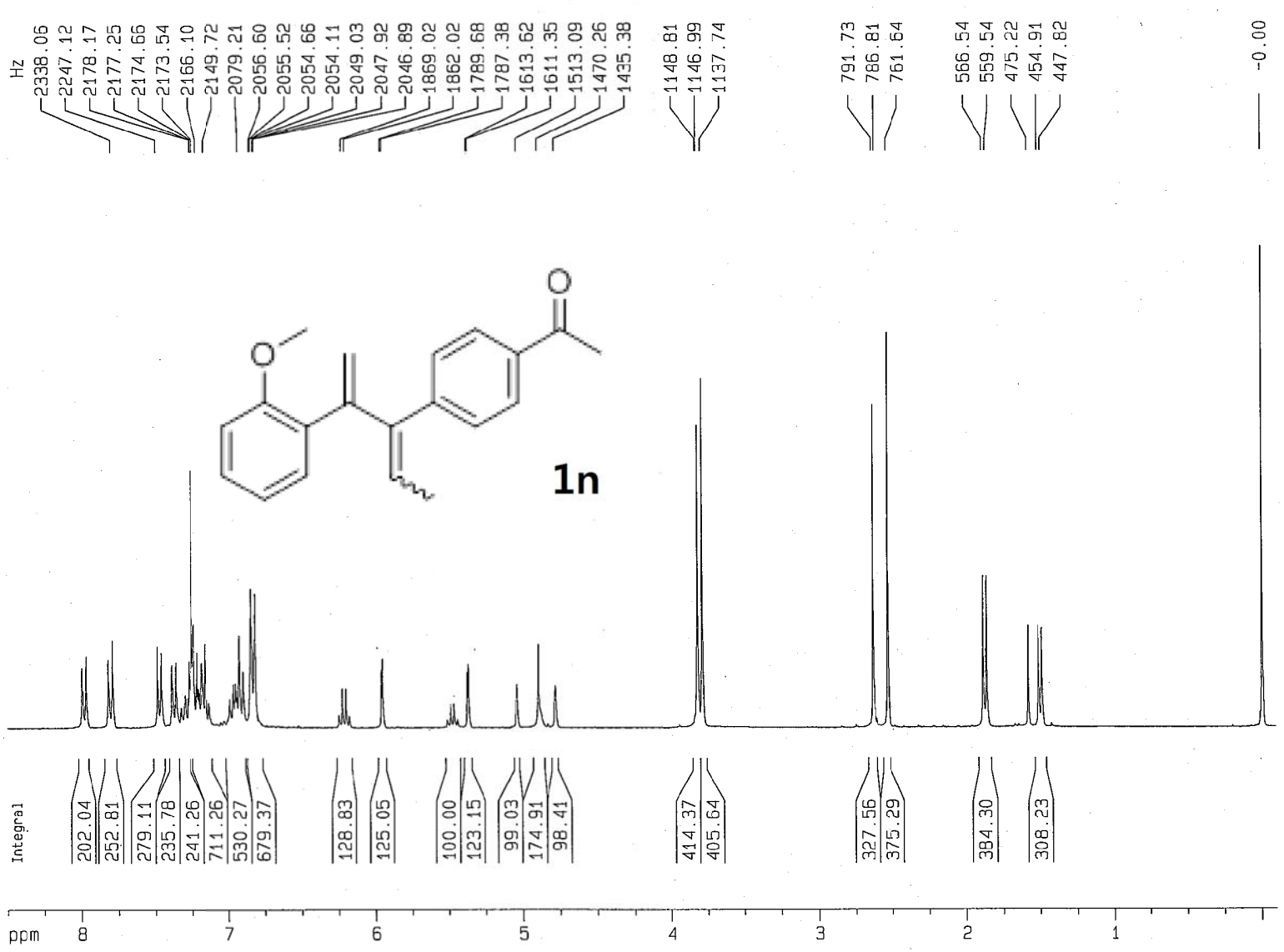
1D NMR plot parameters

CX	21.00 cm
CY	10.00 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.100 ppm
F2	-30.01 Hz
PPMCM	0.40952 ppm/cm
HZCM	122.91039 Hz/cm




```
Current Data Parameters
NAME                  sep-11
EXPNO                    190
PROCNO                     1

F2 - Acquisition Parameters
Date_              20110929
Time                   18.56
INSTRUM                spect
PROBHD   5 mm QNP  1H/1
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1304
DS                      0
SWH              22675.736 Hz
FIDRES               0.346004 Hz
AQ                1.4451188 sec
RG                   2298.8
DW                  22.050 usec
DE                   6.00 usec
TE                      0.0 K
D1                2.00000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                    8.00 usec
PL1                   2.00 dB
SFO1           75.4760505 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2               100.00 usec
PL2                   0.00 dB
PL12                 20.00 dB
PL13                 22.00 dB
SFO2           300.1312005 MHz

F2 - Processing parameters
SI                      32768
SF             75.4677494 MHz
WDW                        EM
SSB                         0
LB                   1.00 Hz
GB                          0
PC                      1.40

1D NMR plot parameters
CX                   20.00 cm
CY                   70.00 cm
F1P                250.000 ppm
F1                 18866.94 Hz
F2P                 -0.100 ppm
F2                   -7.55 Hz
PPMCM          12.50500 ppm/cm
HZCM           943.72424 Hz/cm
```

PSJ-518-3



Current Data Parameters

NAME	sep-11
EXPNO	236
PROCNO	1

F2 - Acquisition Parameters

Date_	20111004
Time	19.42
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	812.7
DW	81.000 usec
DE	6.00 usec
TE	0.0 K
D1	1.00000000 sec

======== CHANNEL f1 ========

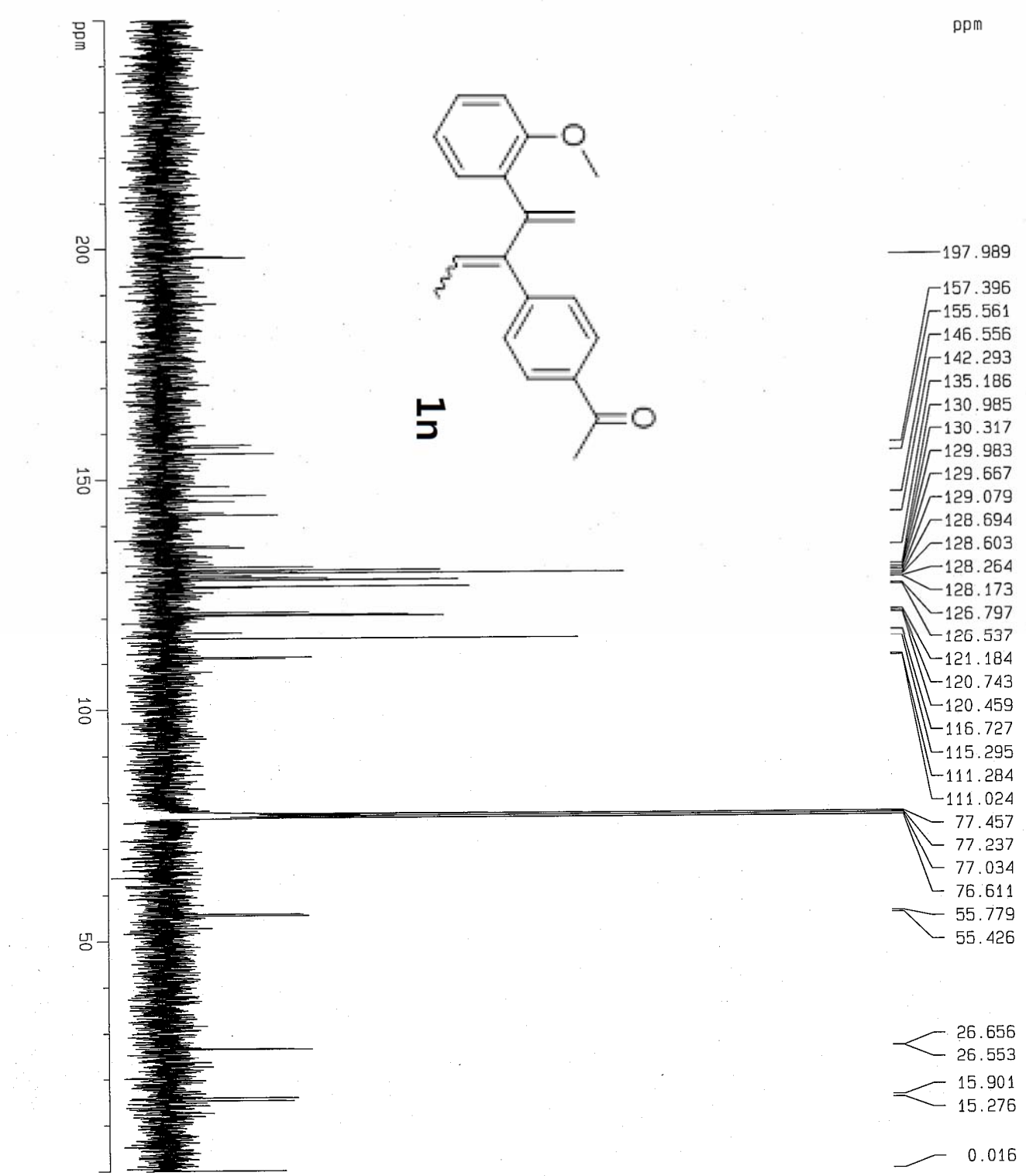
NUC1	1H
P1	10.20 usec
PL1	0.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	32768
SF	300.1300079 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

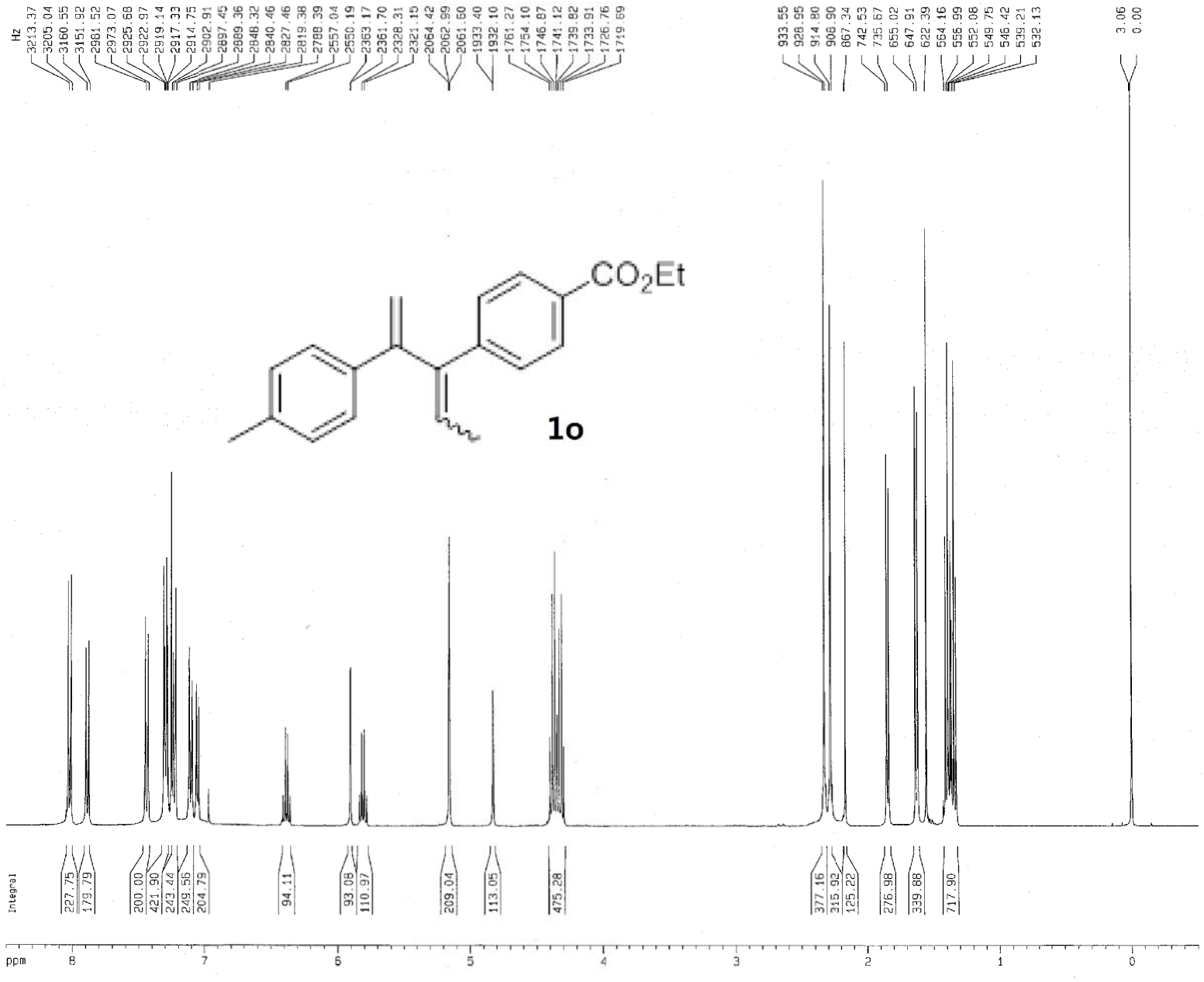
1D NMR plot parameters

CX	21.00 cm
CY	8.00 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.100 ppm
F2	-30.01 Hz
PPMCM	0.40952 ppm/cm
HZCM	122.91039 Hz/cm




```
Current Data Parameters
NAME                  sep-11
EXPNO                    269
PROCNO                     1

F2 - Acquisition Parameters
Date_              20111005
Time                   22.18
INSTRUM                spect
PROBHD  5 mm QNP  1H/1
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1159
DS                       0
SWH              22675.736 Hz
FIDRES           0.346004 Hz
AQ               1.4451188 sec
RG                   20642.5
DW                  22.050 usec
DE                   6.00 usec
TE                      0.0 K
D1                2.00000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                     8.00 usec
PL1                    2.00 dB
SFO1             75.4760505 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                100.00 usec
PL2                    0.00 dB
PL12                  20.00 dB
PL13                  22.00 dB
SFO2             300.1312005 MHz

F2 - Processing parameters
SI                       32768
SF               75.4677485 MHz
WDW                         EM
SSB                          0
LB                    1.00 Hz
GB                           0
PC                        1.40

1D NMR plot parameters
CX                    20.00 cm
CY                    80.00 cm
F1P                 250.000 ppm
F1                 18866.94 Hz
F2P                 -0.100 ppm
F2                   -7.55 Hz
PPMCM              12.50500 ppm/cm
HZCM              943.72424 Hz/cm
```

PSJ-523



Current Data Parameters
NAME sep-11
EXPNO 37
PROCNO 1

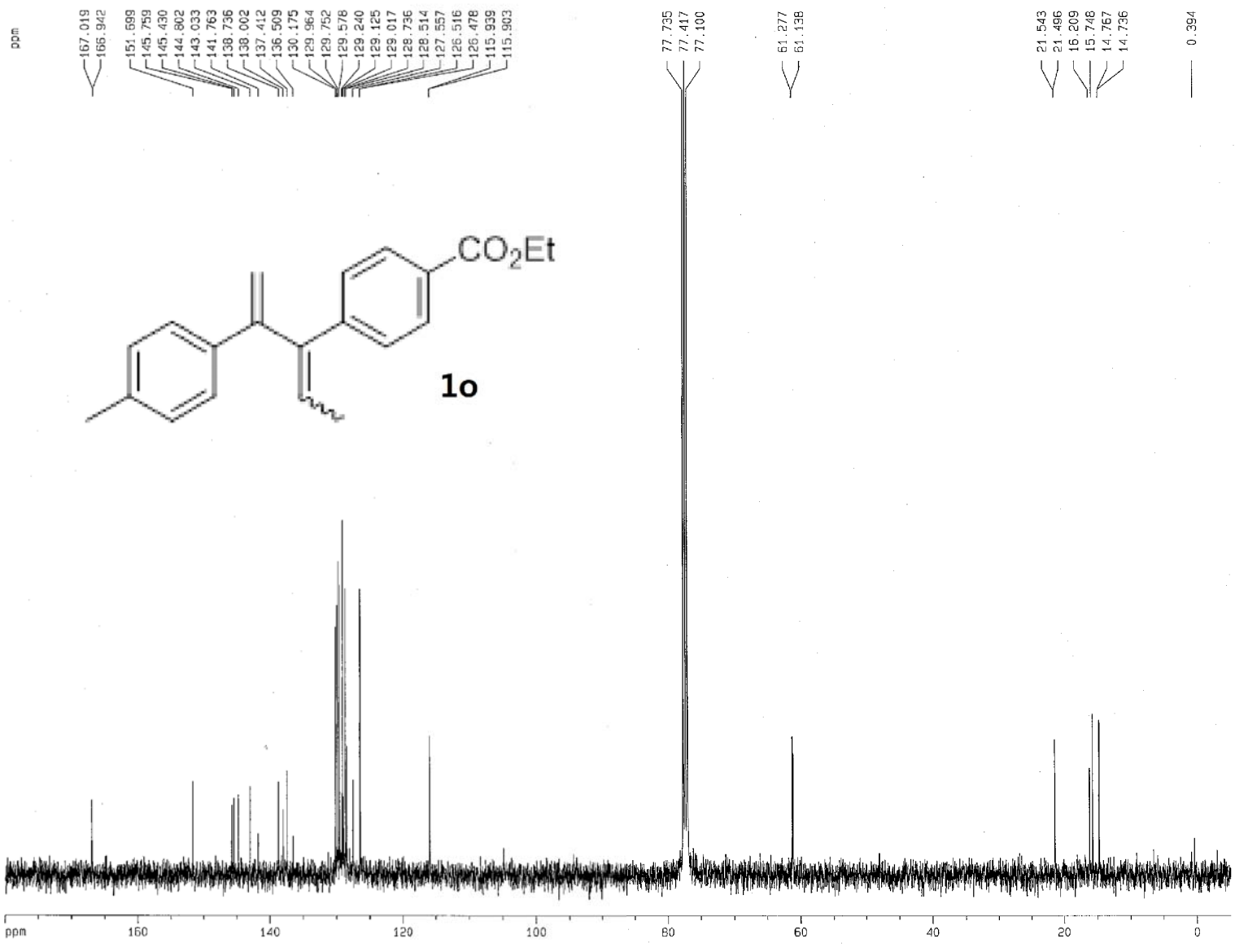
F2 - Acquisition Parameters
Date_ 20111013
Time 16.03
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 287.4
DW 60.800 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 16384
SF 400.1300114 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 30.00 cm
CY 20.00 cm
F1P 8.500 ppm
F1 3401.10 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.30000 ppm/cm
HZCM 120.03900 Hz/cm

PSJ-523



Current Data Parameters

NAME	sep-11
EXPNO	38
PROCNO	1

F2 - Acquisition Parameters

Date_	20111013
Time	16.16
INSTRUM	spect
PROBHD	5 mm Dual 13C/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	2
SWH	31847.133 Hz
FIDRES	0.485949 Hz
AQ	1.0289652 sec
RG	3649.1
DW	15.700 usec
DE	6.00 usec
TE	298.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	10.50 usec
PL1	-1.00 dB
SFO1	100.6254358 MHz

======== CHANNEL f2 ========

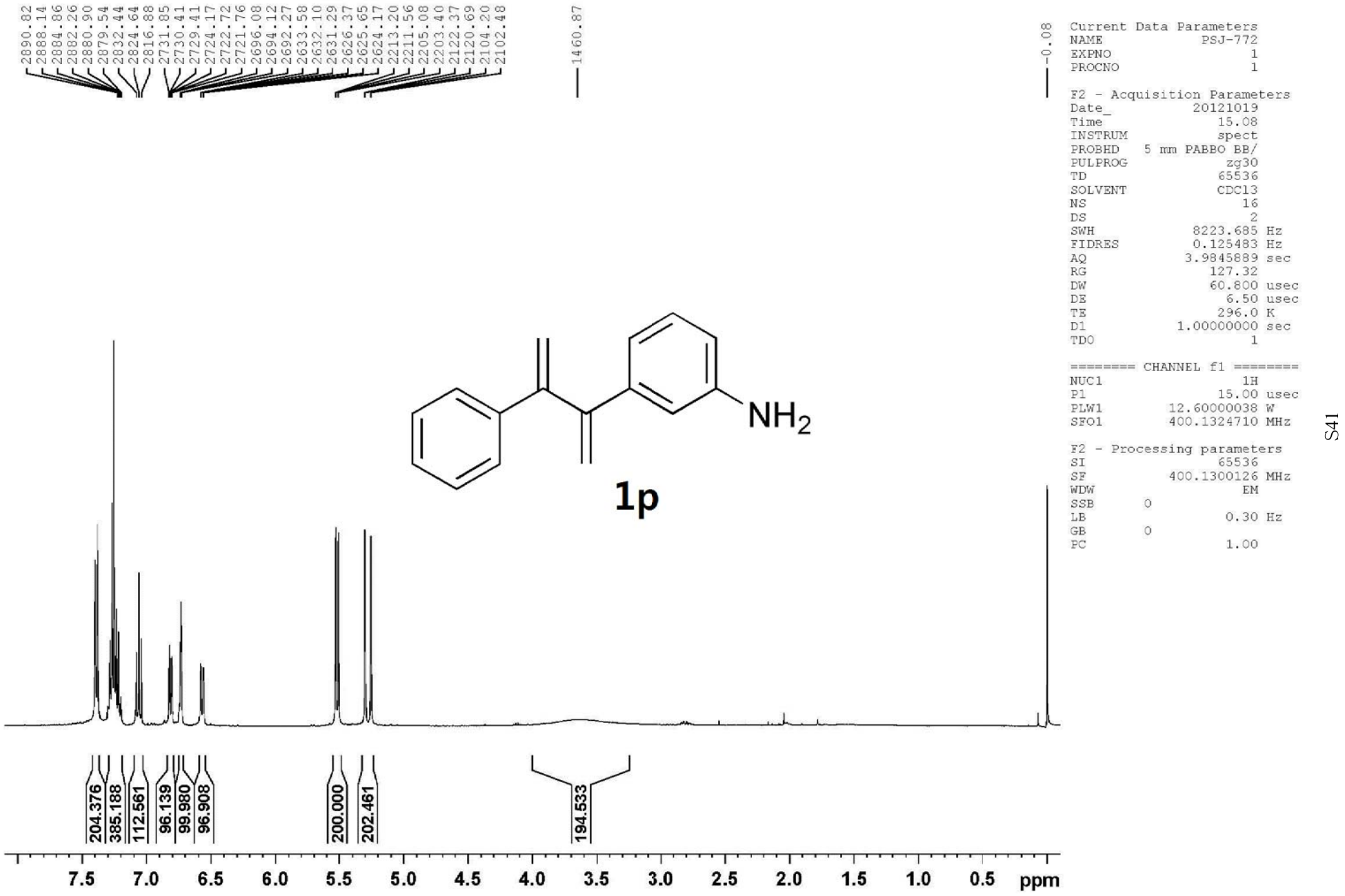
CPDPRG2	waltz16
NUC2	1H
PCPD2	105.00 usec
PL2	-2.00 dB
PL12	18.00 dB
PL13	20.00 dB
SFO2	400.1324708 MHz

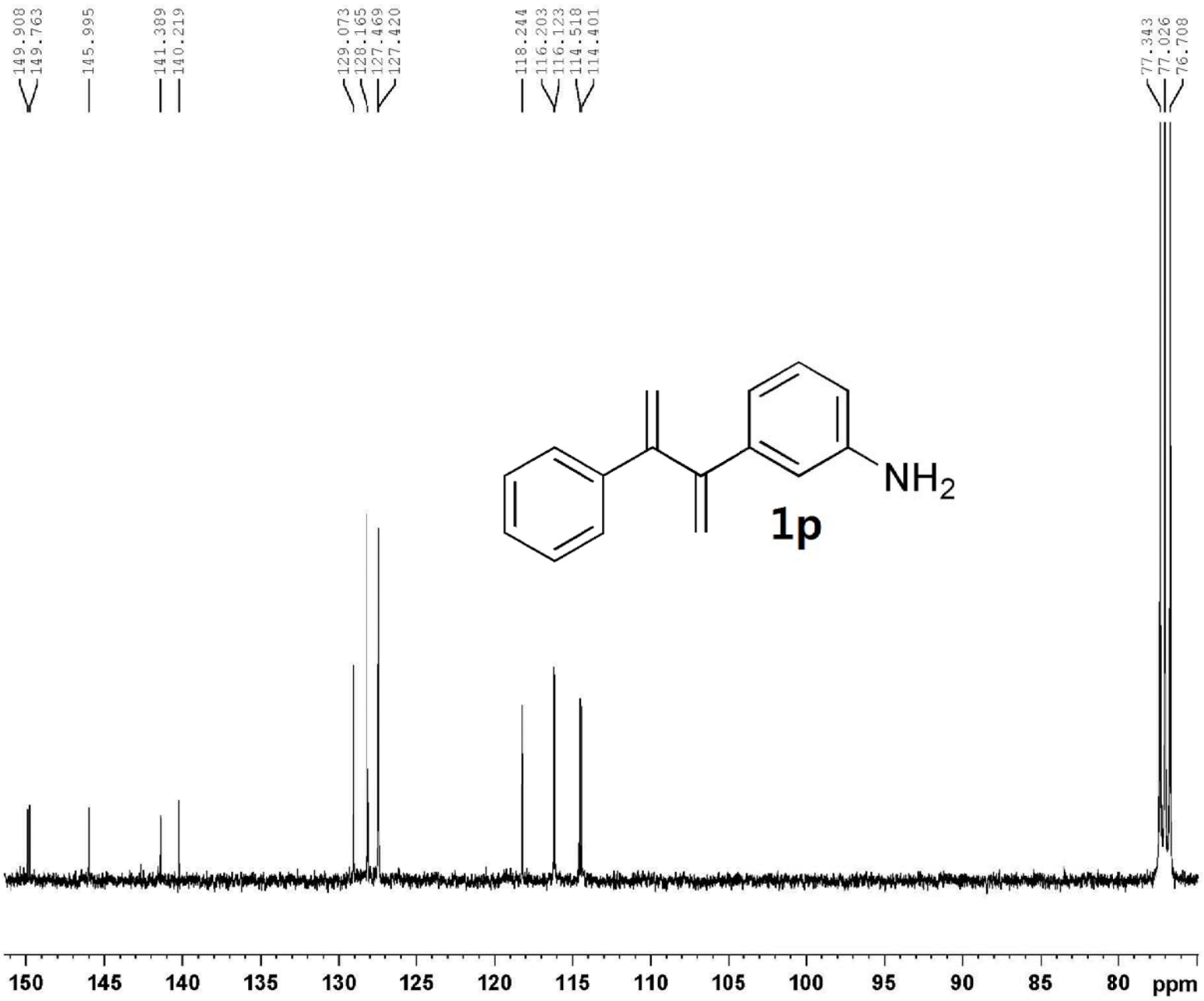
F2 - Processing parameters

SI	32768
SF	100.6127290 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

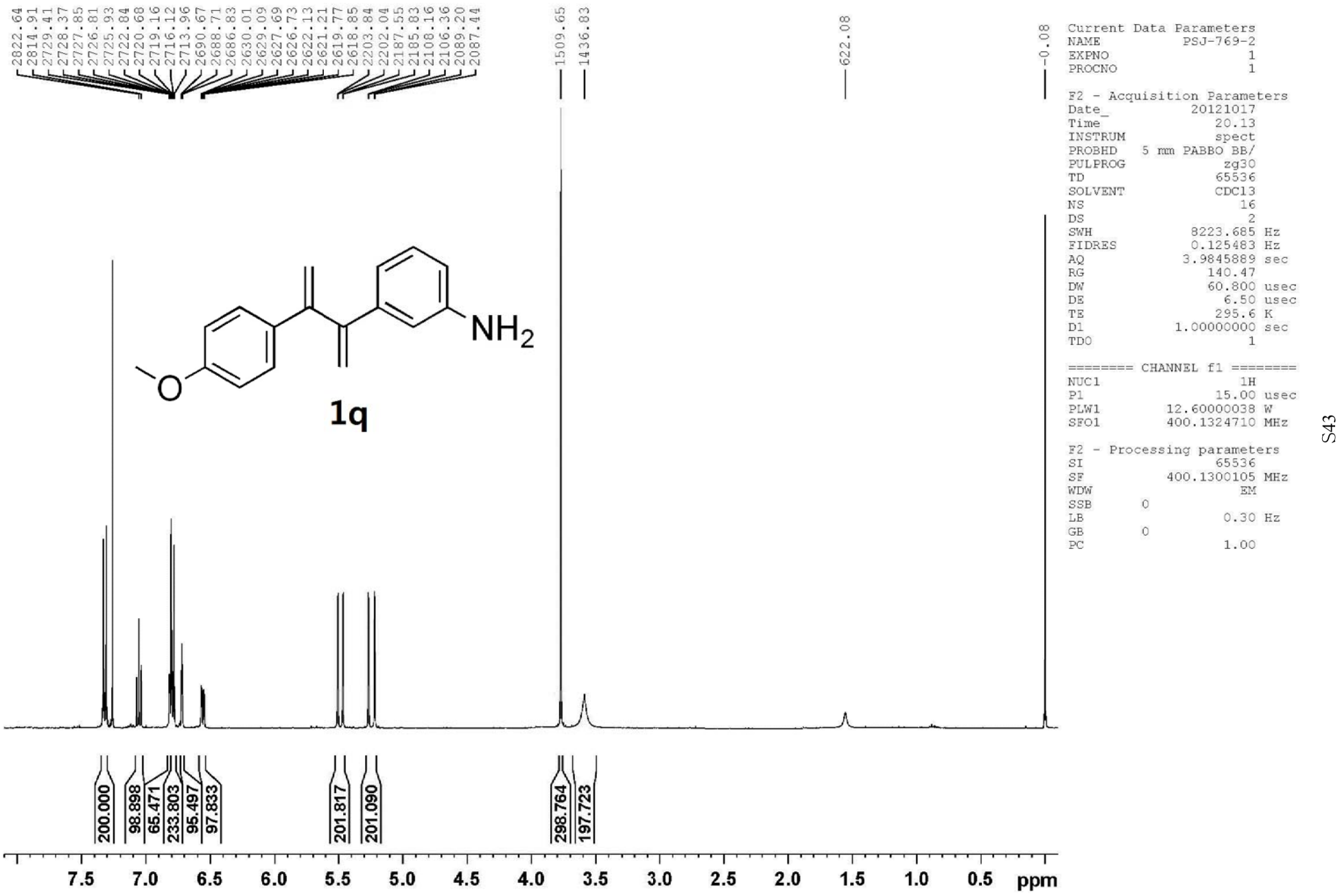
1D NMR plot parameters

CX	30.00 cm
CY	60.00 cm
F1P	180.000 ppm
F1	18110.29 Hz
F2P	-5.000 ppm
F2	-503.06 Hz
PPMCM	6.16667 ppm/cm
HZCM	620.44519 Hz/cm


2890.82
2888.14
2884.86
2882.26
2880.90
2879.54
2832.44
2824.64
2816.88
2731.85
2730.41
2729.41
2724.17
2722.72
2721.76
2696.08
2694.12
2692.27
2633.58
2632.10
2631.29
2626.37
2625.65
2624.17
2213.20
2211.56
2205.08
2203.40
2122.37
2120.69
2104.20
2102.48
1460.87
-0.08
Current Data Parameters
NAME PSJ-772
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121019
Time 15.08
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 127.32
DW 60.800 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.60000038 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300126 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NH2
1p
204.376
385.188
112.561
96.139
99.980
96.908
200.000
202.461
194.533
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




```
Current Data Parameters
NAME                  PSJ-772
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20121019
Time                  18.02
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1024
DS                       4
SWH              24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631488 sec
RG                  186.53
DW                20.800 usec
DE                 6.50 usec
TE                 297.5 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                      1
SFO1           100.6228293 MHz
NUC1                   13C
P1                  9.98 usec
PLW1           52.09999847 W
SFO2           400.1316005 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2              90.00 usec
PLW2           12.60000038 W
PLW12           0.34999999 W
PLW13           0.28349999 W

F2 - Processing parameters
SI                   32768
SF             100.6127690 MHz
WDW                     EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

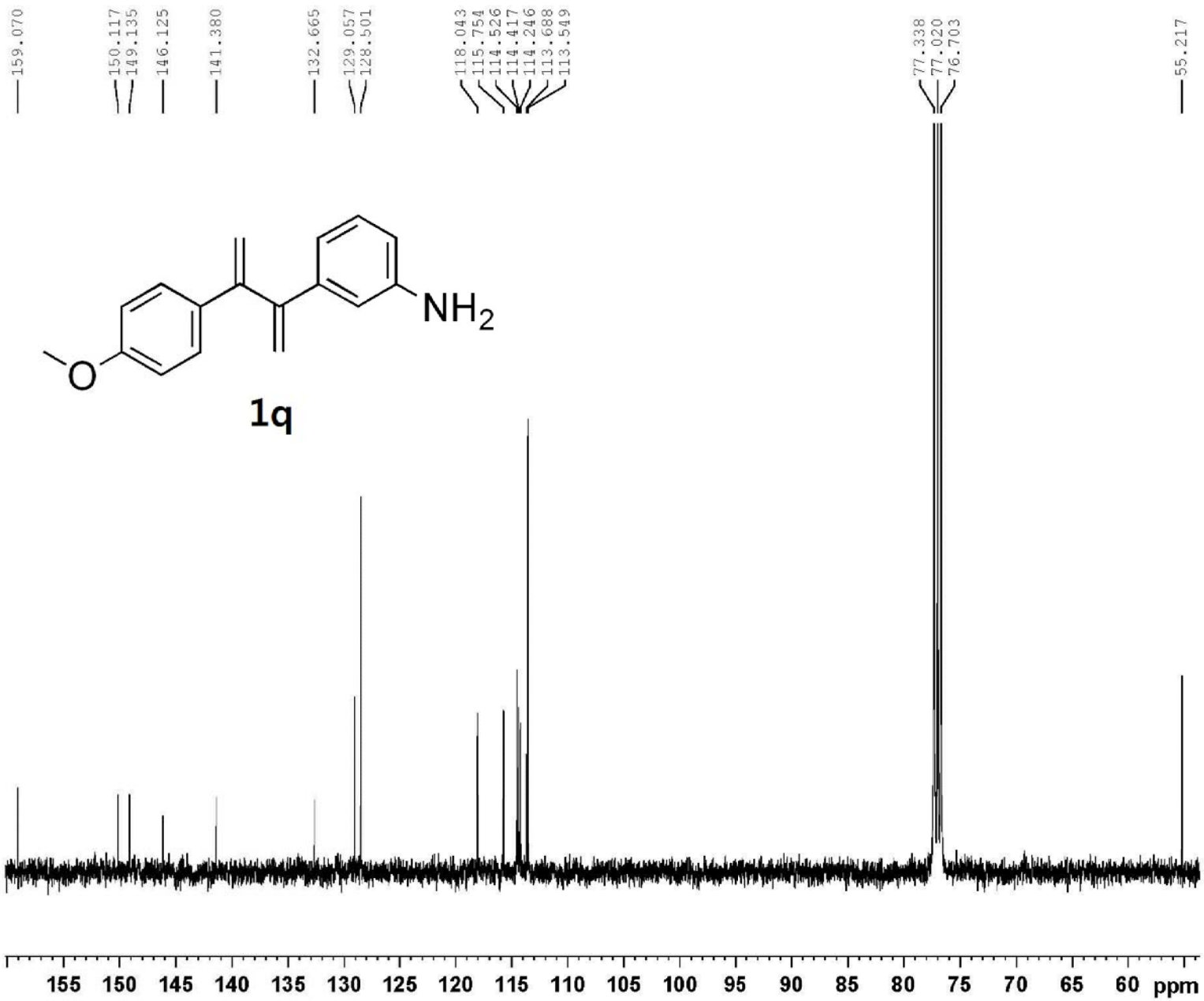


Current Data Parameters
NAME PSJ-769-2
EXPNO 1
PROCNO 1

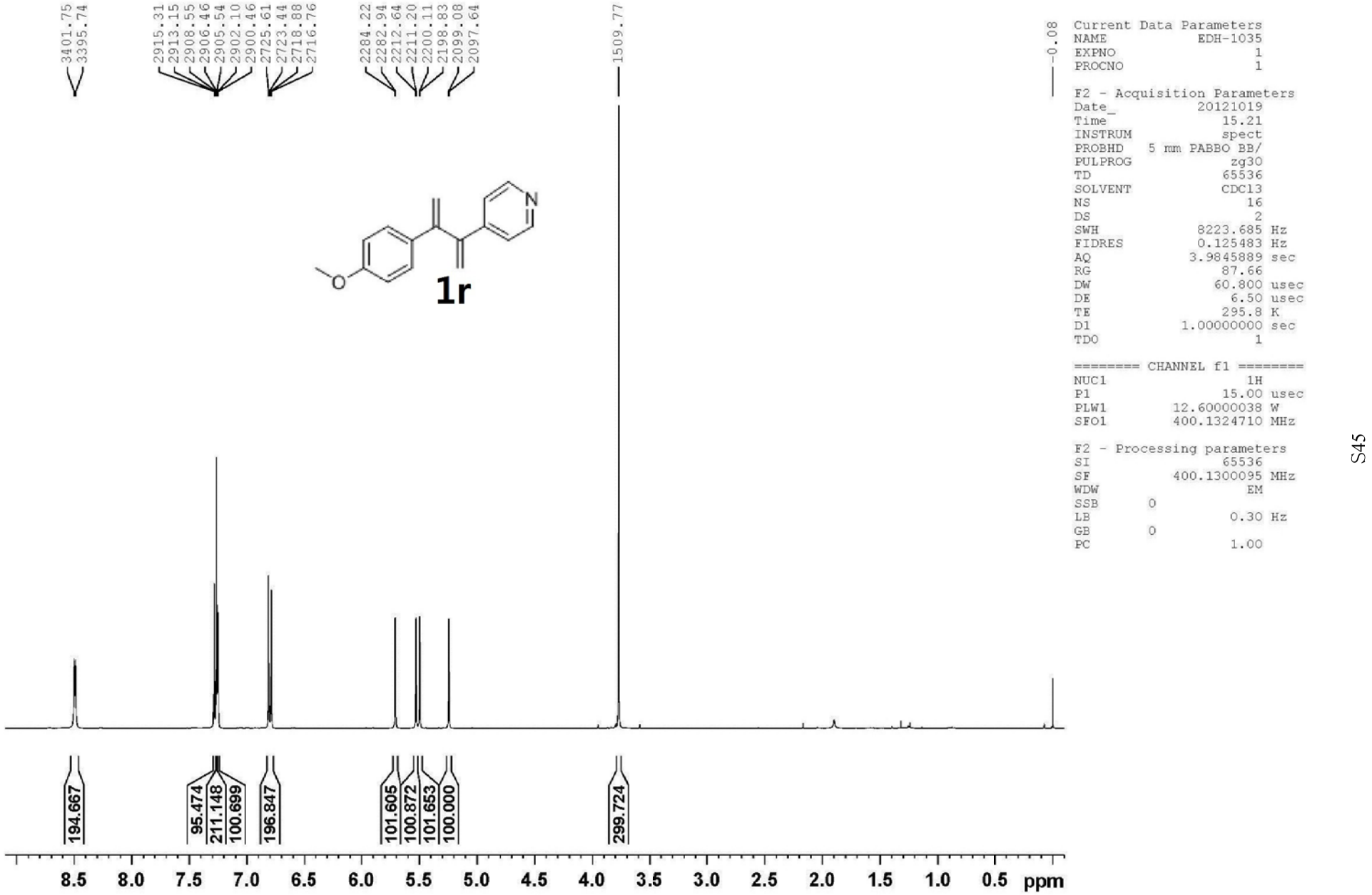
F2 - Acquisition Parameters
Date_ 20121017
Time 20.13
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 140.47
DW 60.800 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1

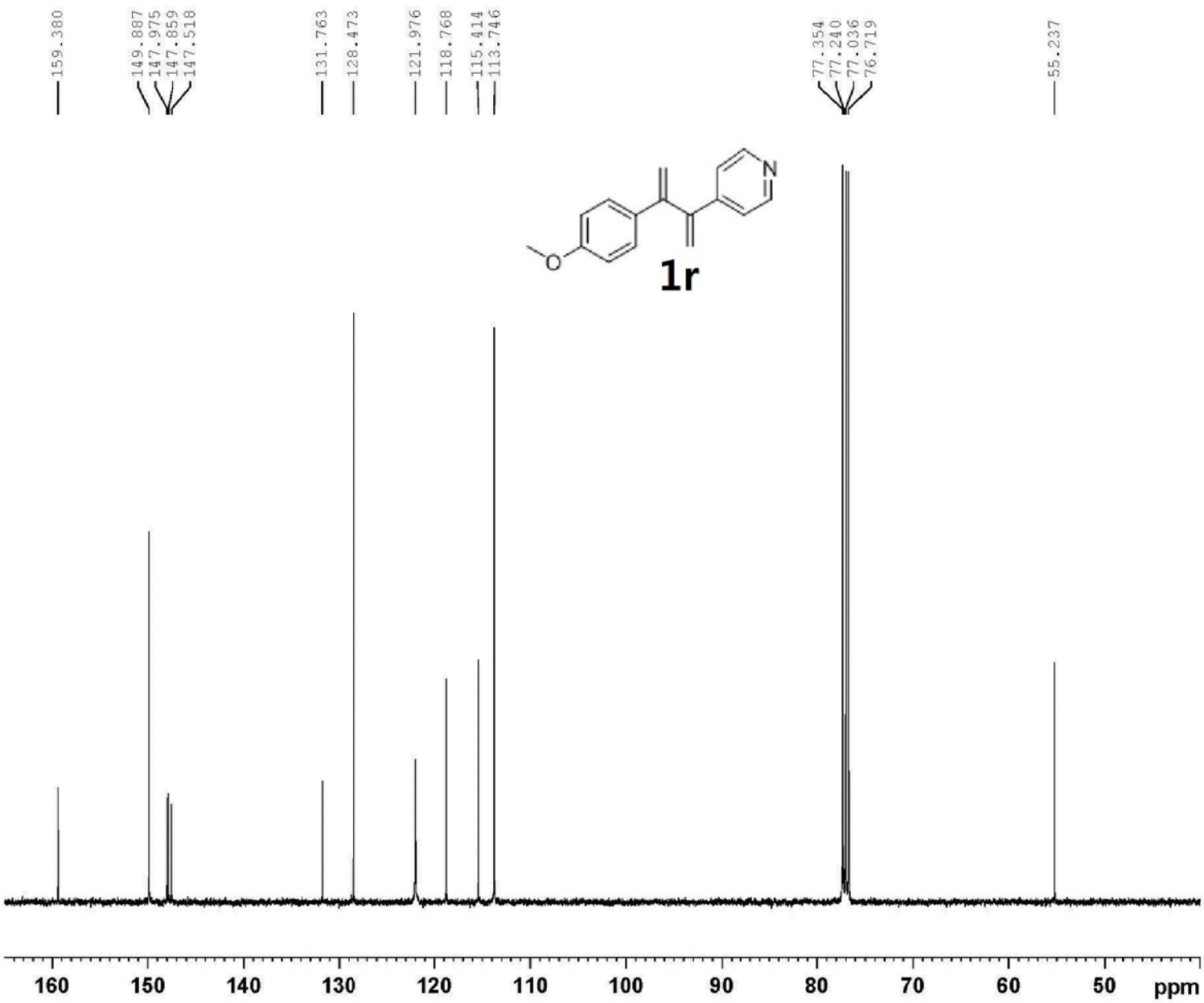
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.60000038 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300105 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

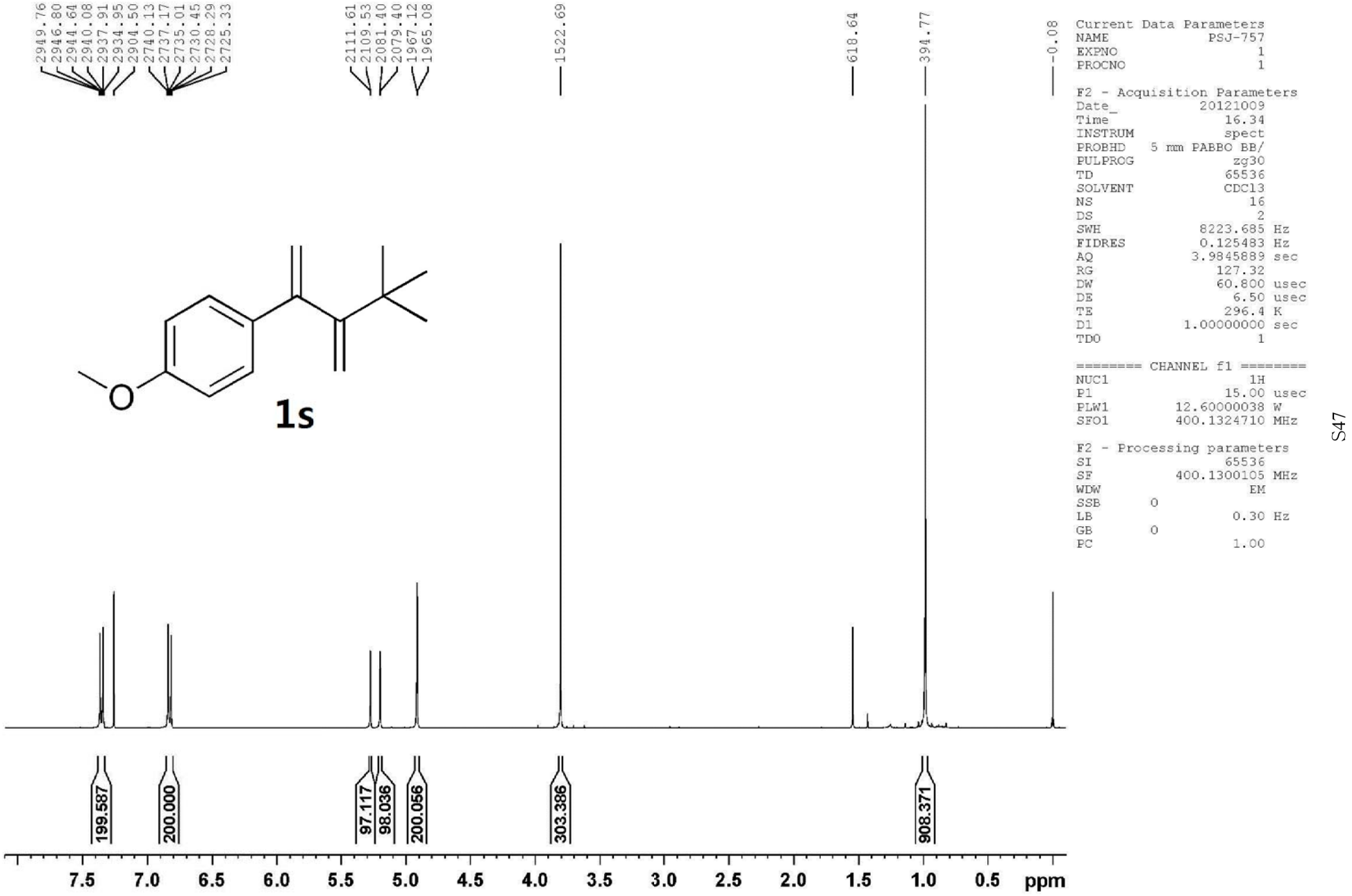



```
Current Data Parameters
NAME                  PSJ-769-2
EXPNO                         3
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20121017
Time                      21.21
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         980
DS                           4
SWH                  24038.461 Hz
FIDRES                0.366798 Hz
AQ                    1.3631488 sec
RG                       186.53
DW                       20.800 usec
DE                        6.50 usec
TE                       297.1 K
D1                    2.00000000 sec
d11                   0.03000000 sec
DELTA                 1.89999998 sec
TD0                           1
SFO1                100.6228293 MHz
NUC1                        13C
P1                        9.98 usec
PLW1                52.09999847 W
SFO2                400.1316005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2                12.60000038 W
PLW12                0.34999999 W
PLW13                0.28349999 W

F2 - Processing parameters
SI                        32768
SF                  100.6127690 MHz
WDW                          EM
SSB       0
LB                        1.00 Hz
GB        0
PC                        1.40
```

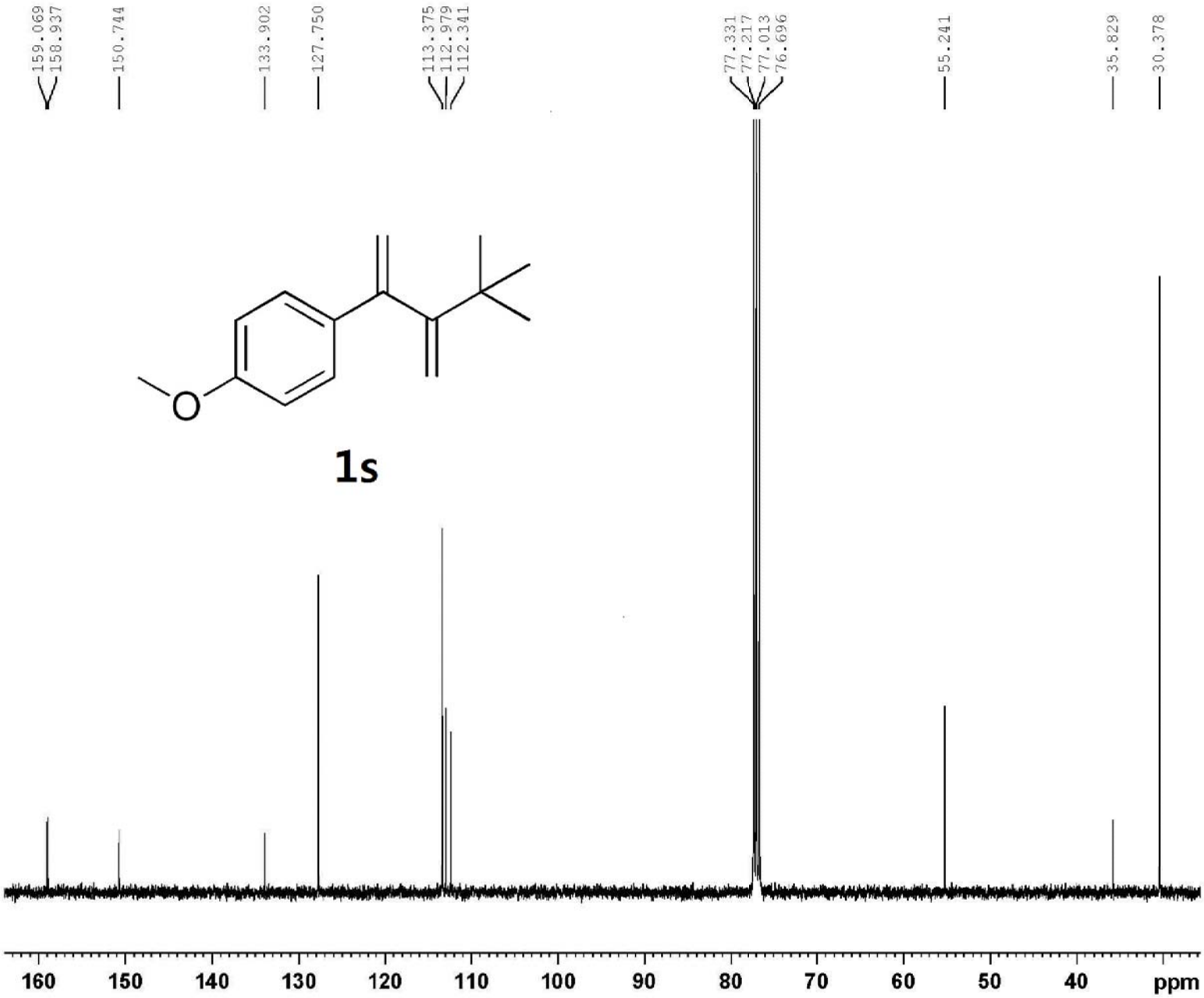

3401.75
3395.74
2915.31
2913.15
2908.55
2906.46
2905.54
2902.10
2900.46
2725.61
2723.44
2718.88
2716.76
2284.22
2282.94
2212.64
2211.20
2200.11
2198.83
2099.08
2097.64
1509.77
-0.08
N
O
1r
Current Data Parameters
NAME EDH-1035
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121019
Time 15.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 87.66
DW 60.800 usec
DE 6.50 usec
TE 295.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.60000038 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
194.667
95.474
211.148
100.699
196.847
101.605
100.872
101.653
100.000
299.724
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




```
Current Data Parameters
NAME                  EDH-1035
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20121019
Time                     16.51
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1024
DS                          4
SWH                 24038.461 Hz
FIDRES               0.366798 Hz
AQ                   1.3631488 sec
RG                      186.53
DW                      20.800 usec
DE                       6.50 usec
TE                      297.3 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
TD0                         1
SFO1             100.6228293 MHz
NUC1                       13C
P1                       9.98 usec
PLW1             52.09999847 W
SFO2             400.1316005 MHz
NUC2                       1H
CPDPRG[2              waltz16
PCPD2                   90.00 usec
PLW2             12.60000038 W
PLW12             0.34999999 W
PLW13             0.28349999 W

F2 - Processing parameters
SI                       32768
SF               100.6127690 MHz
WDW                         EM
SSB          0
LB                       1.00 Hz
GB           0
PC                       1.40
```

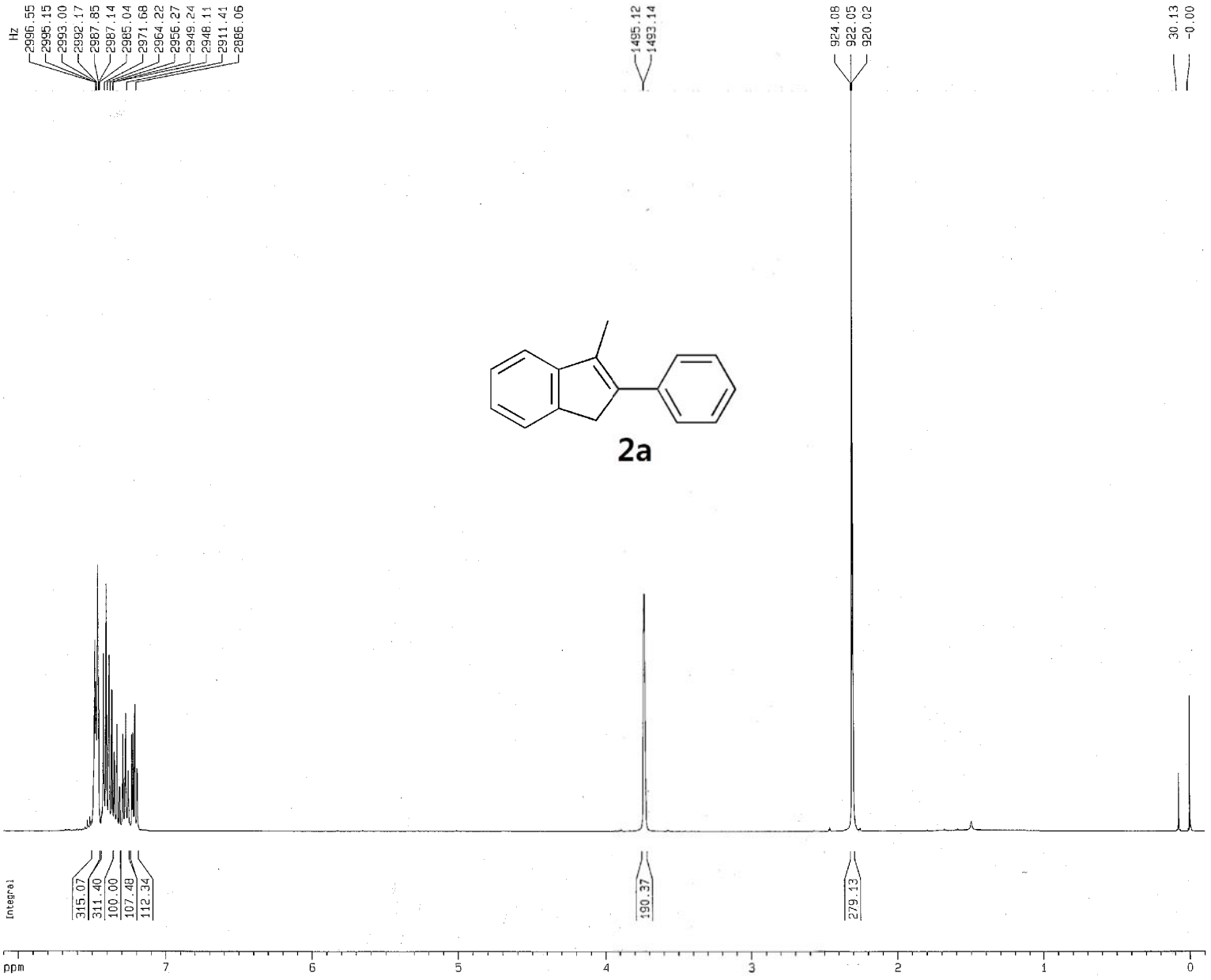



```
Current Data Parameters
NAME                  PSJ-757
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20121009
Time                  16.34
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9845889 sec
RG                   127.32
DW                   60.800 usec
DE                    6.50 usec
TE                   296.4 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    15.00 usec
PLW1           12.60000038 W
SFO1           400.1324710 MHz

F2 - Processing parameters
SI                    65536
SF             400.1300105 MHz
WDW                      EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```




```
Current Data Parameters
NAME                  PSJ-757
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20121009
Time                  18.51
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     834
DS                       4
SWH             24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                  186.53
DW                20.800 usec
DE                 6.50 usec
TE                297.8 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                     1
SFO1           100.6228293 MHz
NUC1                  13C
P1                  9.98 usec
PLW1           52.09999847 W
SFO2           400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2           12.60000038 W
PLW12           0.34999999 W
PLW13           0.28349999 W

F2 - Processing parameters
SI                   32768
SF             100.6127690 MHz
WDW                     EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

EDY
~~SU~~ 709



Current Data Parameters
NAME mar-11
EXPNO 76
PROCNO 1

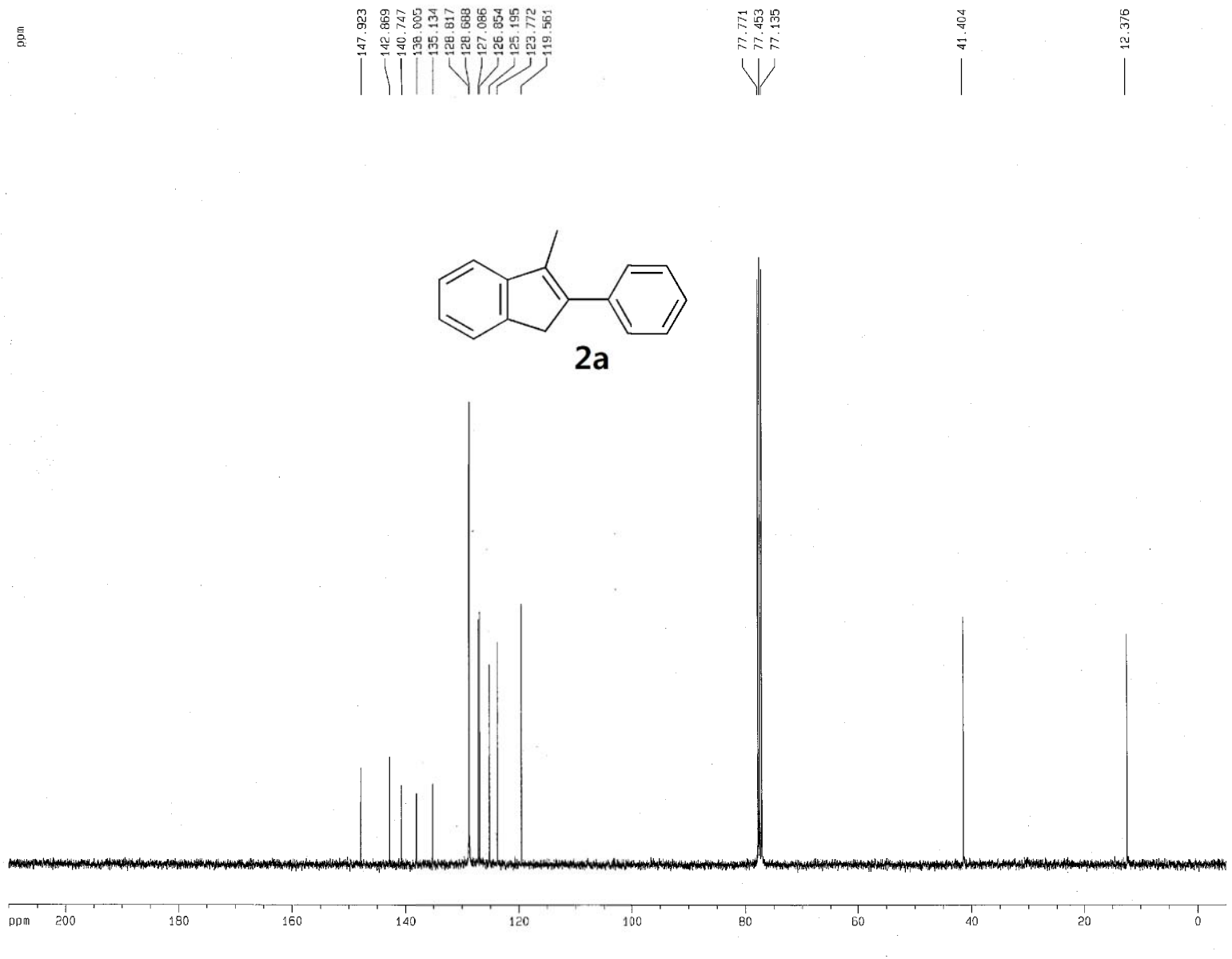
F2 - Acquisition Parameters
Date_ 20110405
Time 16.47
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 181
DW 60.800 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 16384
SF 400.1300235 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

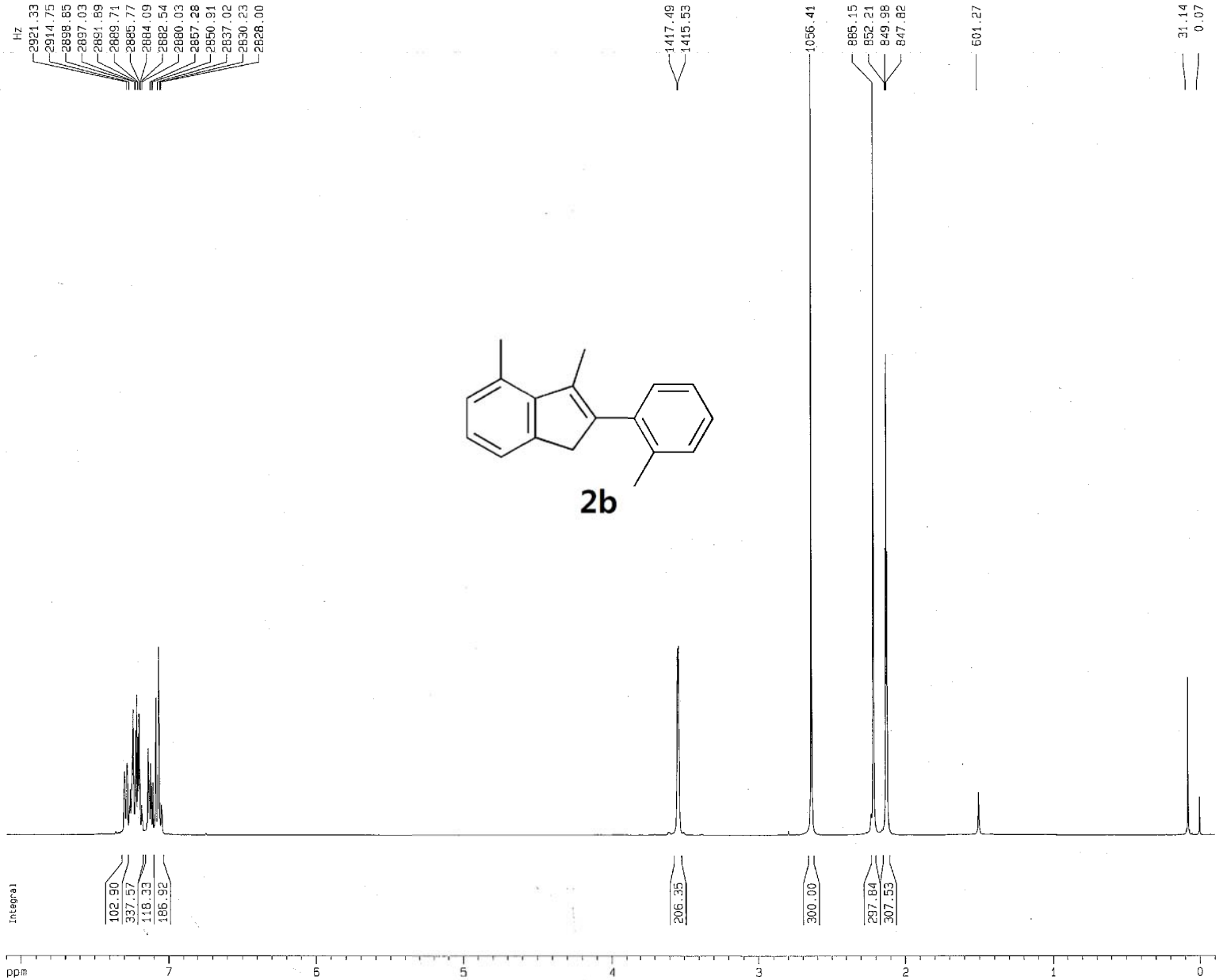
1D NMR plot parameters
CX 30.00 cm
CY 20.00 cm
F1P 8.100 ppm
F1 3241.05 Hz
F2P -0.100 ppm
F2 -40.01 Hz
PPMCM 0.27333 ppm/cm
HZCM 109.36888 Hz/cm

SJM-709




```
Current Data Parameters
NAME                  mar-11
EXPNO                     77
PROCNO                     1

F2 - Acquisition Parameters
Date_              20110405
Time                  16.56
INSTRUM               spect
PROBHD   5 mm Dual 13C/
PULPROG               zgpg30
TD                     65536
SOLVENT               CDCl3
NS                       604
DS                         2
SWH               31847.133 Hz
FIDRES            0.485949 Hz
AQ                1.0289652 sec
RG                     322.5
DW                  15.700 usec
DE                   6.00 usec
TE                   298.2 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                   10.50 usec
PL1                  -1.00 dB
SFO1           100.6254358 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2               105.00 usec
PL2                  -2.00 dB
PL12                 18.00 dB
PL13                 20.00 dB
SFO2           400.1324708 MHz

F2 - Processing parameters
SI                     32768
SF            100.6127290 MHz
WDW                       EM
SSB                        0
LB                    1.00 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                    30.00 cm
CY                    15.00 cm
F1P                 210.000 ppm
F1                 21128.67 Hz
F2P                  -5.000 ppm
F2                 -503.06 Hz
PPMCM                7.16667 ppm/cm
HZCM               721.05786 Hz/cm
```

EDH-755



Current Data Parameters

NAME	may-11
EXPNO	7
PROCNO	1

F2 - Acquisition Parameters

Date_	20110502
Time	18.09
INSTRUM	spect
PROBHD	5 mm Dual 13C/
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	0
SWH	8223.685 Hz
FIDRES	0.250967 Hz
AQ	1.9923444 sec
RG	114
DW	60.800 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	11.00 usec
PL1	-2.00 dB
SFO1	400.1324710 MHz

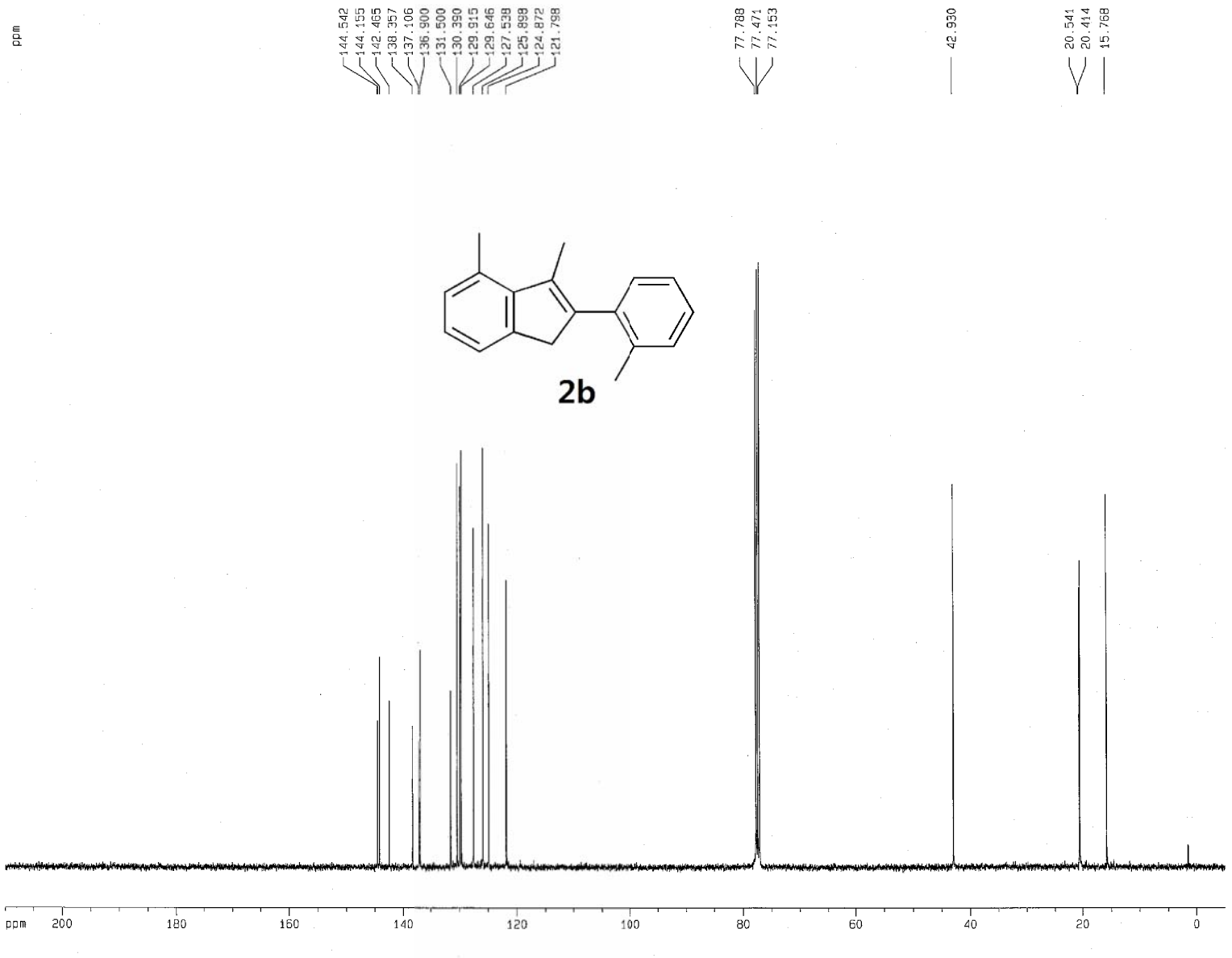
F2 - Processing parameters

SI	16384
SF	400.1300301 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	30.00 cm
CY	20.00 cm
F1P	8.100 ppm
F1	3241.05 Hz
F2P	-0.100 ppm
F2	-40.01 Hz
PPMCM	0.27333 ppm/cm
HZCM	109.36888 Hz/cm

EDH-755



Current Data Parameters

NAME	may-11
EXPNO	8
PROCNO	1

F2 - Acquisition Parameters

Date_	20110502
Time	19.23
INSTRUM	spect
PROBHD	5 mm Dual 13C/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1417
DS	2
SWH	31847.133 Hz
FIDRES	0.485949 Hz
AQ	1.0289652 sec
RG	3649.1
DW	15.700 usec
DE	6.00 usec
TE	298.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	10.50 usec
PL1	-1.00 dB
SFO1	100.6254358 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	105.00 usec
PL2	-2.00 dB
PL12	18.00 dB
PL13	20.00 dB
SFO2	400.1324708 MHz

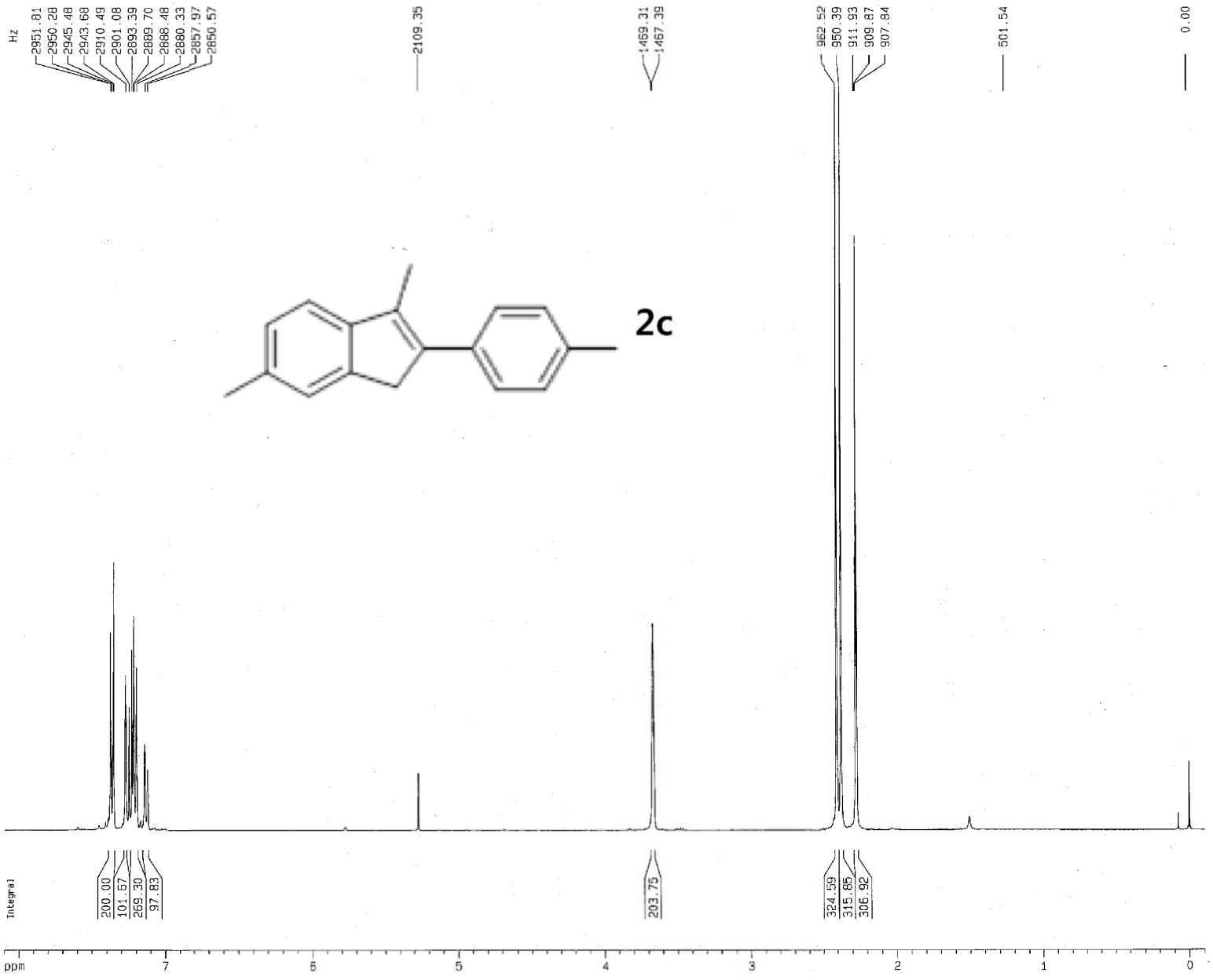
F2 - Processing parameters

SI	32768
SF	100.6127290 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	30.00 cm
CY	15.00 cm
F1P	210.000 ppm
F1	21128.67 Hz
F2P	-5.000 ppm
F2	-503.06 Hz
PPMCM	7.16667 ppm/cm
HZCM	721.05786 Hz/cm

EDH-734



Current Data Parameters

NAME	mar-11
EXPNO	74
PROCNO	1

F2 - Acquisition Parameters

Date_	20110405
Time	16.12
INSTRUM	spect
PROBHD	5 mm Dual 13C/
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	0
SWH	8223.685 Hz
FIDRES	0.250967 Hz
AQ	1.9923444 sec
RG	161.3
DW	60.800 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	11.00 usec
PL1	-2.00 dB
SFO1	400.1324710 MHz

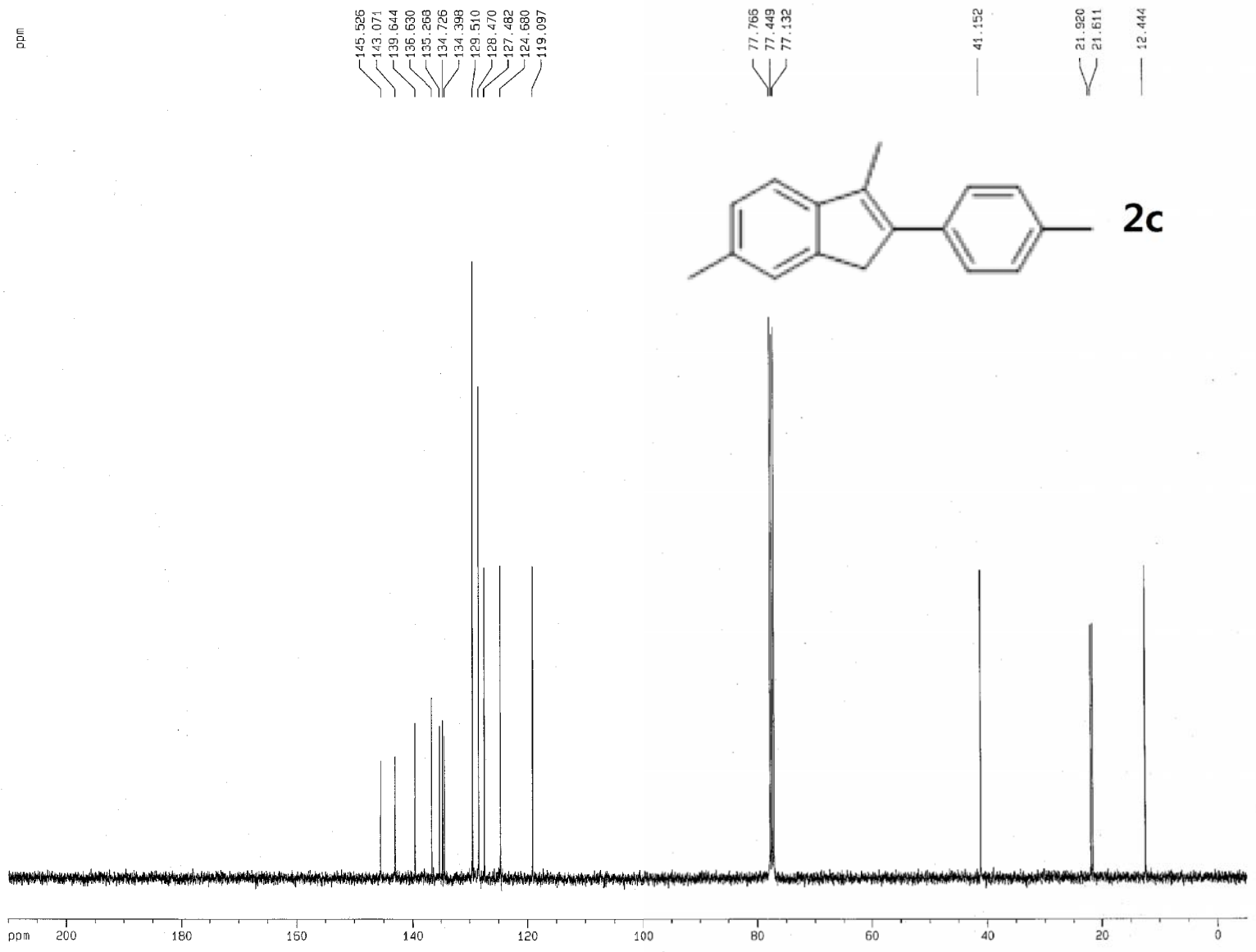
F2 - Processing parameters

SI	16384
SF	400.1300243 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	30.00 cm
CY	20.00 cm
F1P	8.100 ppm
F1	3241.05 Hz
F2P	-0.100 ppm
F2	-40.01 Hz
PPMCM	0.27333 ppm/cm
HZCM	109.36888 Hz/cm

EDH-734



Current Data Parameters

NAME	mar-11
EXPNO	75
PROCNO	1

F2 - Acquisition Parameters

Date_	20110405
Time	16.26
INSTRUM	spect
PROBHD	5 mm Dual 13C/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	518
DS	2
SWH	31847.133 Hz
FIDRES	0.485949 Hz
AQ	1.0289652 sec
RG	143.7
DW	15.700 usec
DE	6.00 usec
TE	298.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	10.50 usec
PL1	-1.00 dB
SFO1	100.6254358 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	105.00 usec
PL2	-2.00 dB
PL12	18.00 dB
PL13	20.00 dB
SFO2	400.1324708 MHz

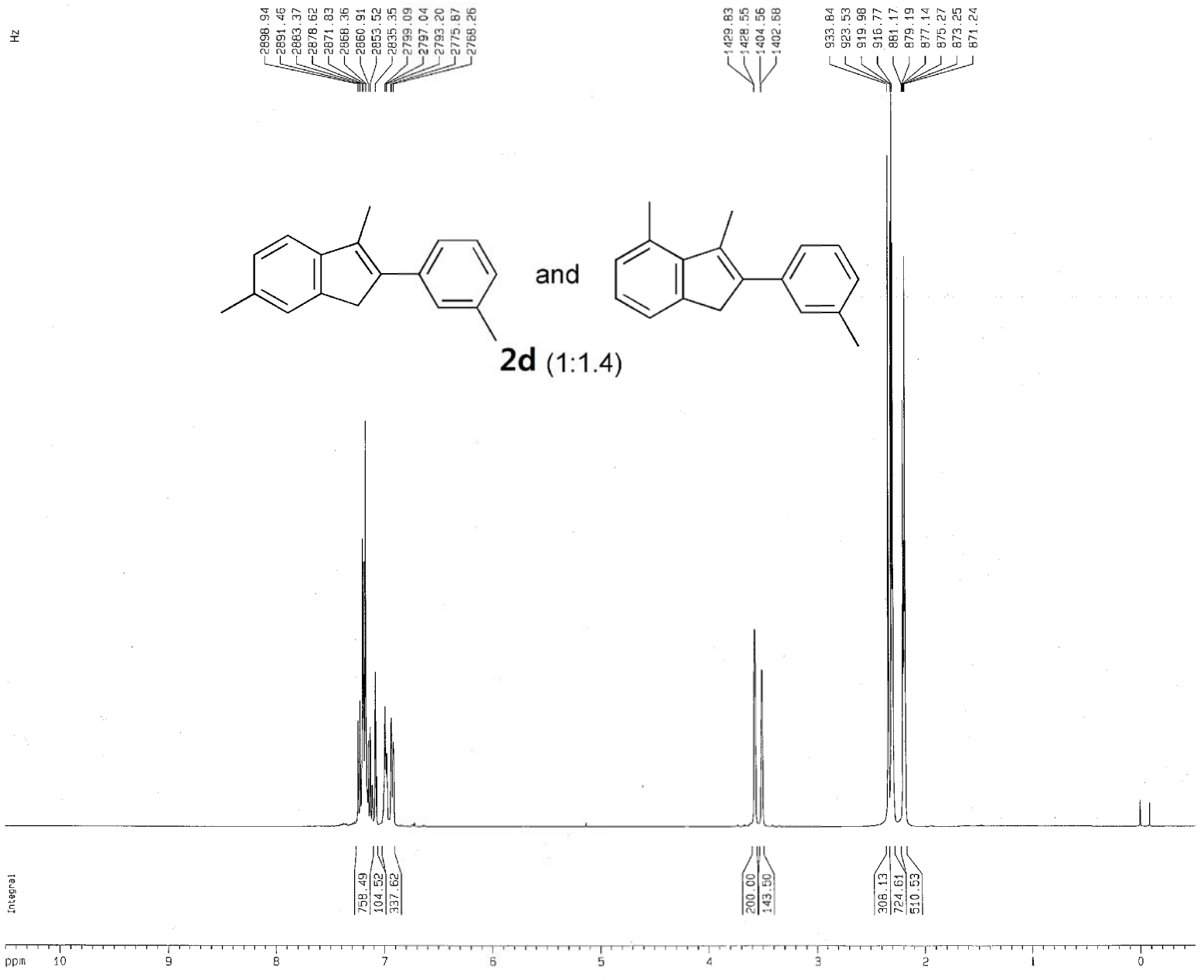
F2 - Processing parameters

SI	32768
SF	100.6127290 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	30.00 cm
CY	15.00 cm
F1P	210.000 ppm
F1	21128.67 Hz
F2P	-5.000 ppm
F2	-503.06 Hz
PPMCM	7.16667 ppm/cm
HZCM	721.05786 Hz/cm

EDH-742



Current Data Parameters

NAME	mar-11
EXPNO	114
PROCNO	1

F2 - Acquisition Parameters

Date_	20110426
Time	13.13
INSTRUM	spect
PROBHD	5 mm Dual 13C/
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	64
DS	0
SWH	8223.685 Hz
FIDRES	0.250967 Hz
AQ	1.9923444 sec
RG	64
DW	60.800 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	11.00 usec
PL1	-2.00 dB
SFO1	400.1324710 MHz

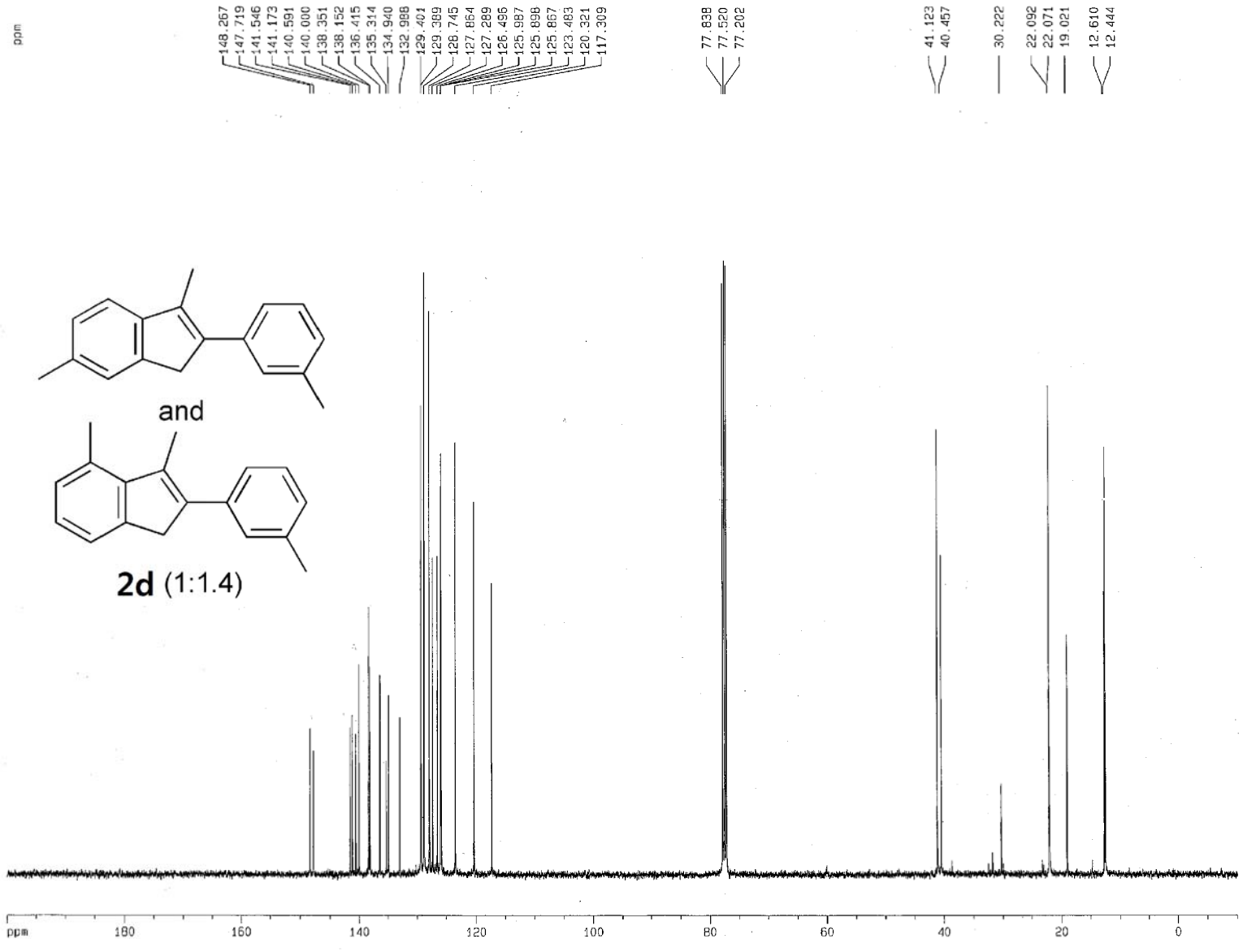
F2 - Processing parameters

SI	16384
SF	400.1300845 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

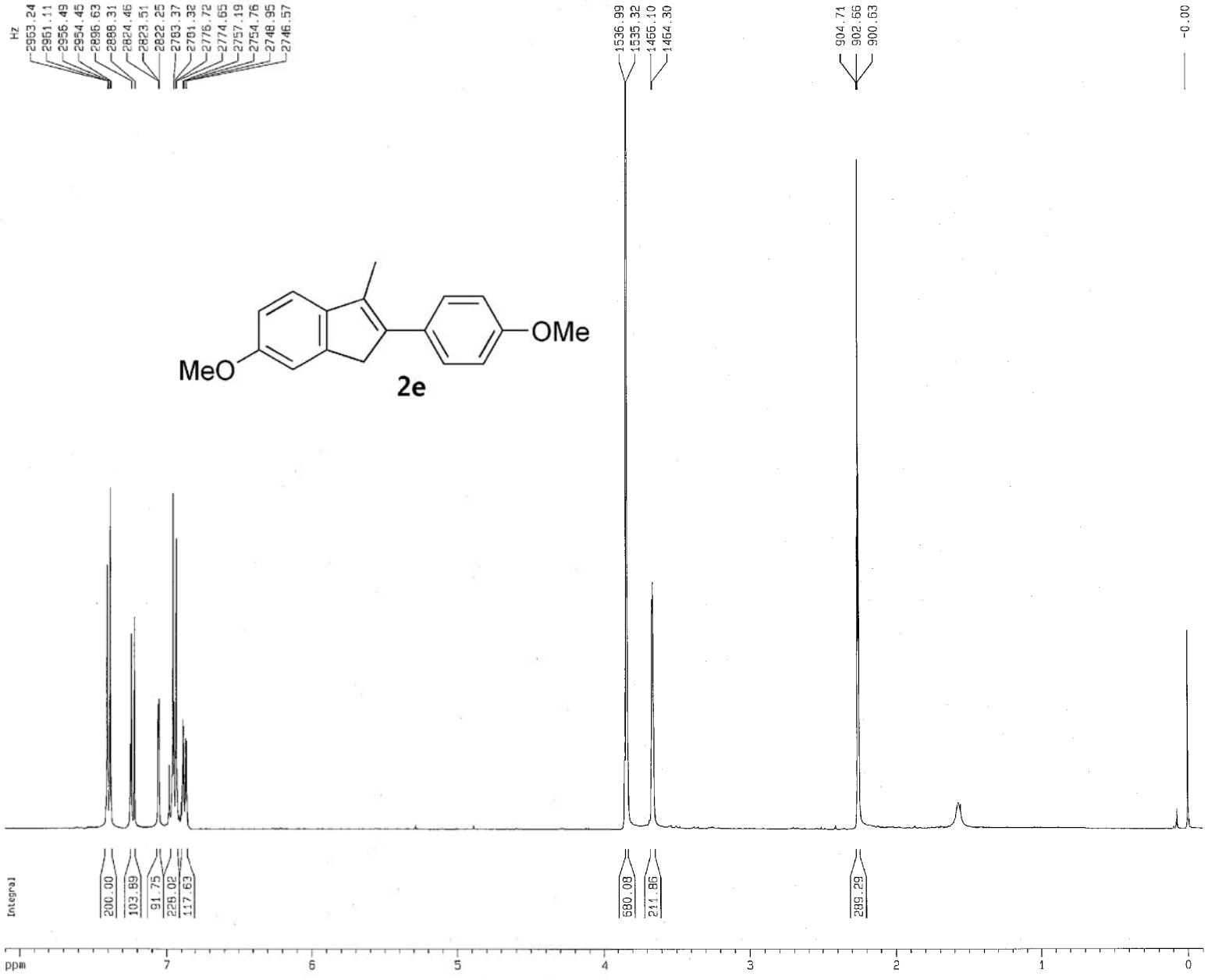
CX	30.00 cm
CY	20.00 cm
F1P	10.500 ppm
F1	4201.37 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.36667 ppm/cm
HZCM	146.71437 Hz/cm

EDH-742




```
Current Data Parameters
NAME                  mar-11
EXPNO                    115
PROCNO                     1

F2 - Acquisition Parameters
Date_              20110426
Time                  15.03
INSTRUM               spect
PROBHD   5 mm Dual 13C/
PULPROG               zgpg30
TD                     65536
SOLVENT                CDCl3
NS                      2048
DS                         2
SWH               31847.133 Hz
FIDRES            0.485949 Hz
AQ                1.0289652 sec
RG                   3649.1
DW                   15.700 usec
DE                    6.00 usec
TE                   298.2 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                    10.50 usec
PL1                   -1.00 dB
SFO1          100.6254358 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                105.00 usec
PL2                   -2.00 dB
PL12                  16.00 dB
PL13                  20.00 dB
SFO2          400.1324708 MHz

F2 - Processing parameters
SI                       32768
SF            100.6127290 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                    30.00 cm
CY                    15.00 cm
F1P                  200.000 ppm
F1                 20122.54 Hz
F2P                  -10.000 ppm
F2                 -1006.13 Hz
PPMCM                7.00000 ppm/cm
HZCM               704.28906 Hz/cm
```

EDH-752



Current Data Parameters

NAME	may-11
EXPNO	5
PROCNO	1

F2 - Acquisition Parameters

Date_	20110502
Time	16.37
INSTRUM	spect
PROBHD	5 mm Dual 13C/
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	0
SWH	8223.685 Hz
FIDRES	0.250967 Hz
AQ	1.9923444 sec
RG	228.1
DW	60.800 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	11.00 usec
PL1	-2.00 dB
SFO1	400.1324710 MHz

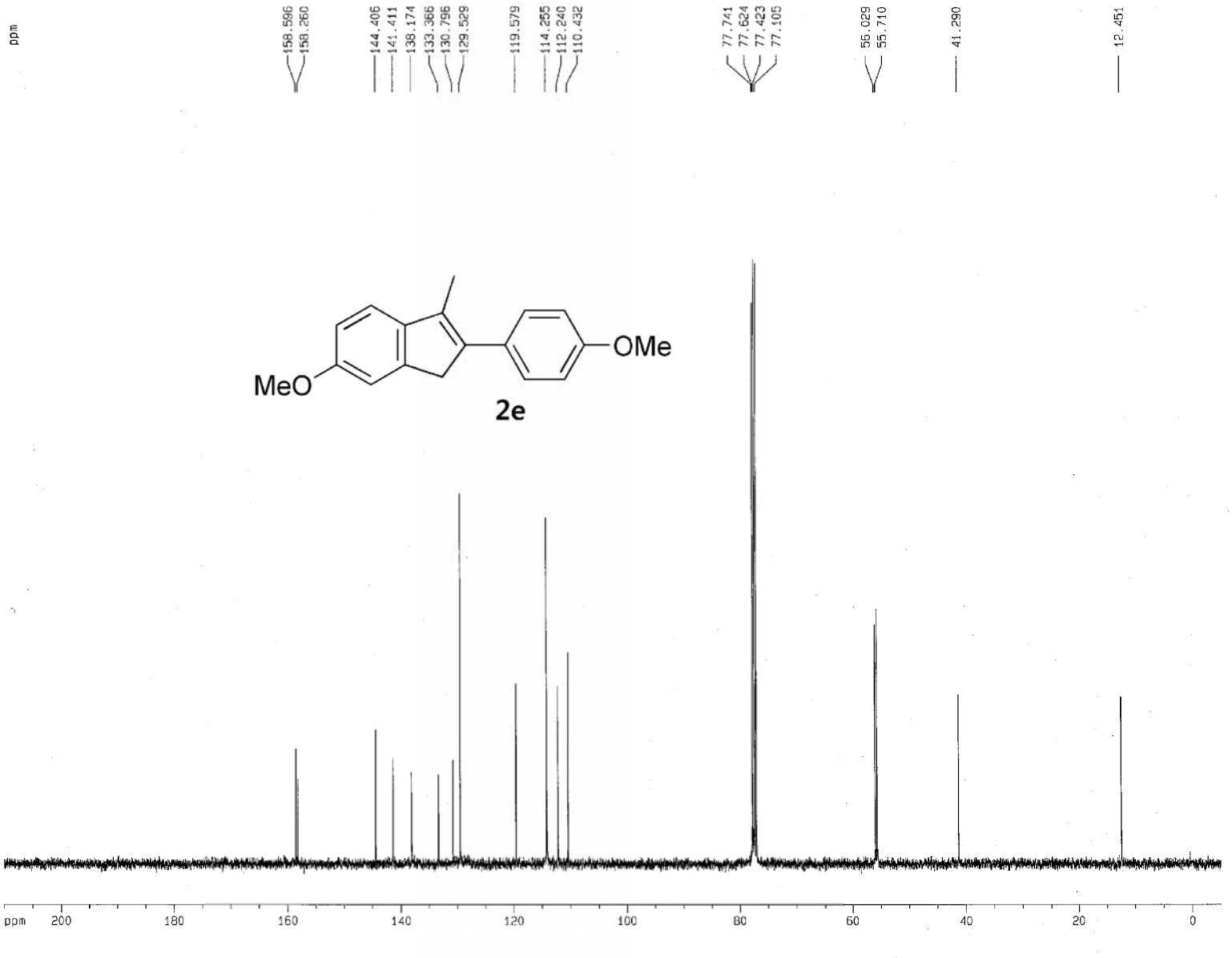
F2 - Processing parameters

SI	16384
SF	400.1300144 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	30.00 cm
CY	45.00 cm
F1P	8.100 ppm
F1	3241.05 Hz
F2P	-0.100 ppm
F2	-40.01 Hz
PPMCM	0.27333 ppm/cm
HZCM	109.36887 Hz/cm

EDH-752



Current Data Parameters

NAME	may-11
EXPNO	6
PROCNO	1

F2 - Acquisition Parameters

Date_	20110502
Time	16.50
INSTRUM	spect
PROBHD	5 mm Dual 13C/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1256
DS	2
SWH	31847.133 Hz
FIDRES	0.485949 Hz
AQ	1.0289652 sec
RG	3649.1
DW	15.700 usec
DE	6.00 usec
TE	298.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	10.50 usec
PL1	-1.00 dB
SFO1	100.6254358 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	105.00 usec
PL2	-2.00 dB
PL12	18.00 dB
PL13	20.00 dB
SFO2	400.1324708 MHz

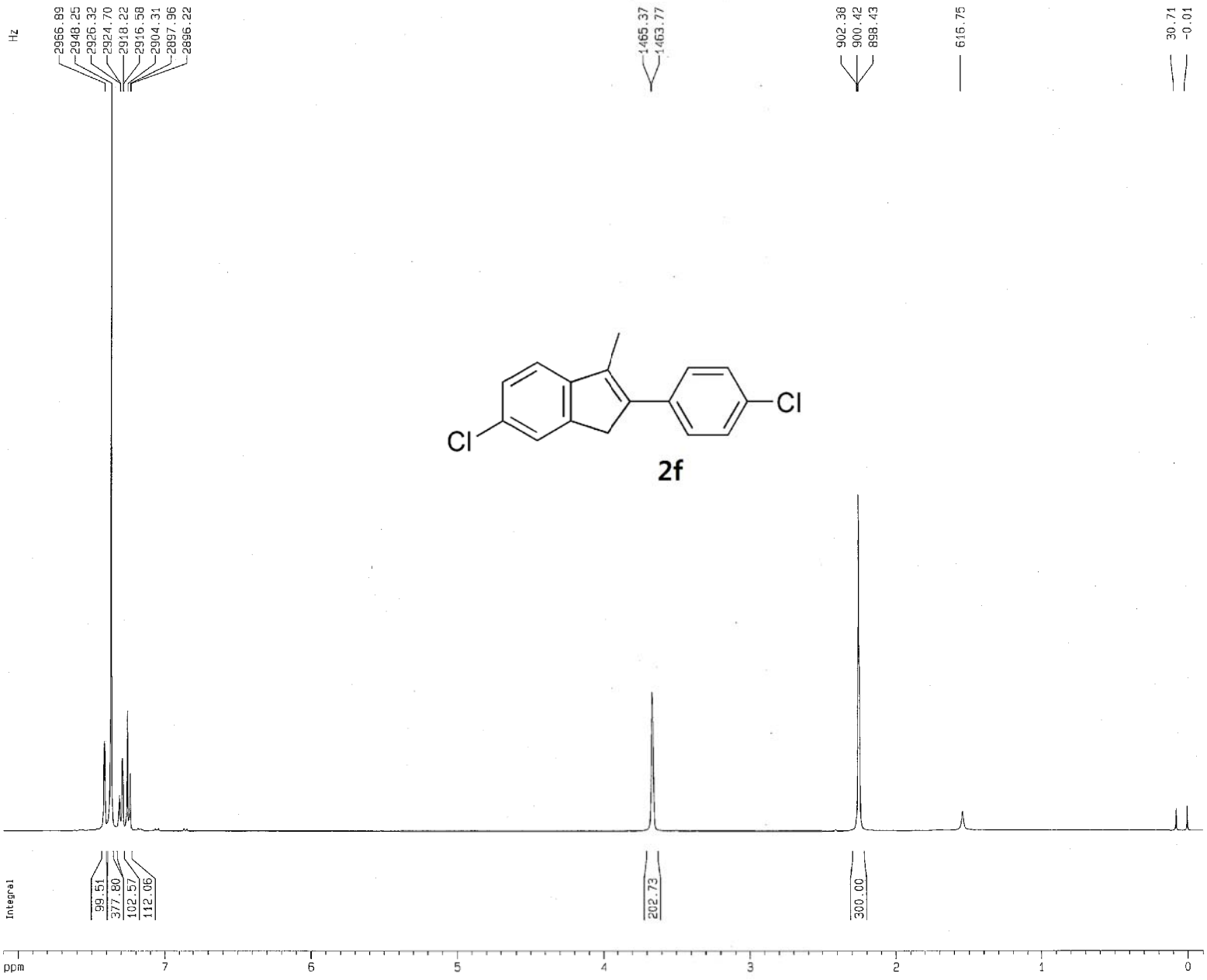
F2 - Processing parameters

SI	32768
SF	100.6127290 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	30.00 cm
CY	15.00 cm
F1P	210.000 ppm
F1	21128.67 Hz
F2P	-5.000 ppm
F2	-503.06 Hz
PPMCM	7.16667 ppm/cm
HZCM	721.05786 Hz/cm

EDH-756



Current Data Parameters

NAME	may-11
EXPNO	9
PROCNO	1

F2 - Acquisition Parameters

Date_	20110502
Time	19.32
INSTRUM	spect
PROBHD	5 mm Dual 13C/
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	0
SWH	8223.685 Hz
FIDRES	0.250967 Hz
AQ	1.9923444 sec
RG	161.3
DW	60.800 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	11.00 usec
PL1	-2.00 dB
SFO1	400.1324710 MHz

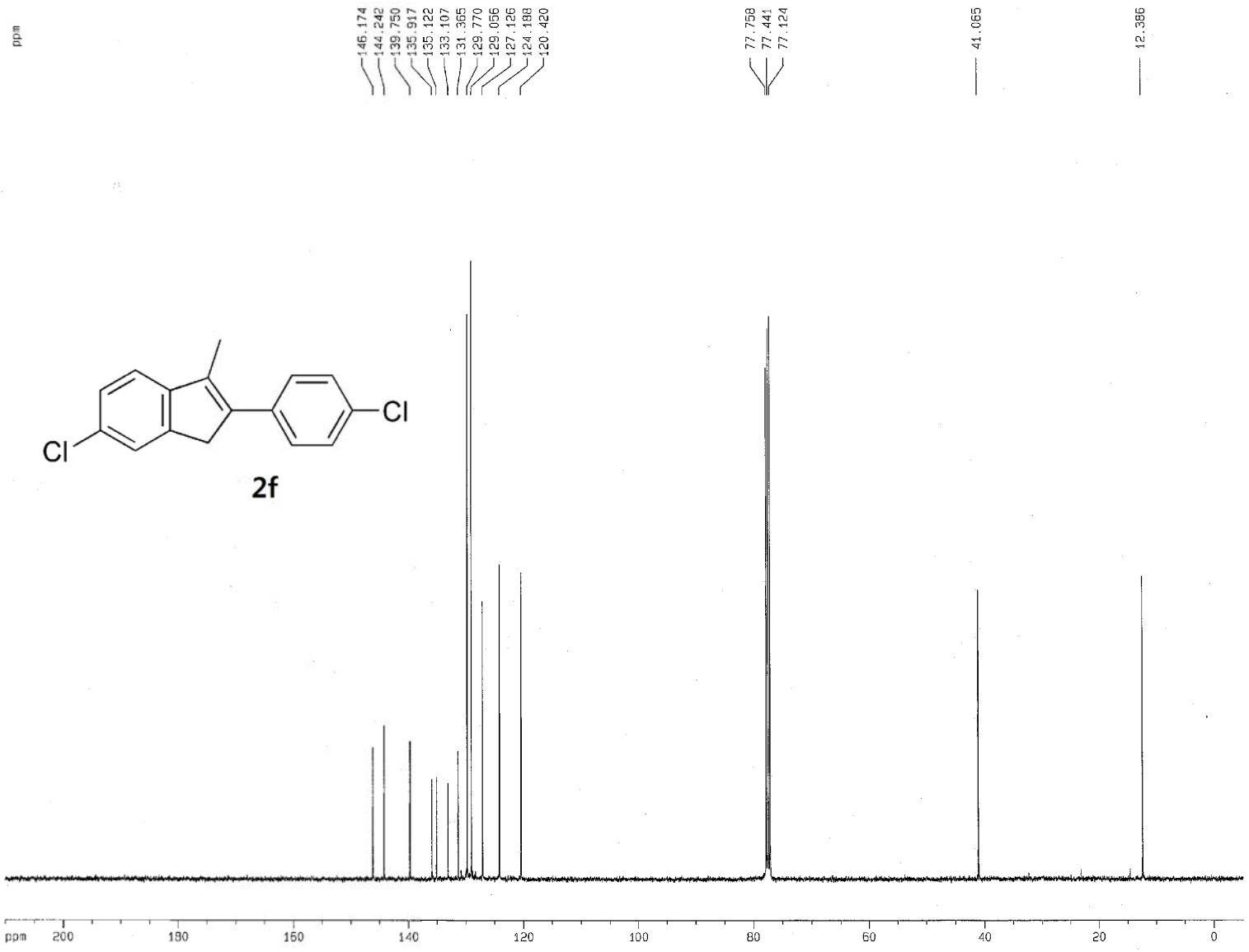
F2 - Processing parameters

SI	16384
SF	400.1300161 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	30.00 cm
CY	20.00 cm
F1P	8.100 ppm
F1	3241.05 Hz
F2P	-0.100 ppm
F2	-40.01 Hz
PPMCM	0.27333 ppm/cm
HZCM	109.36887 Hz/cm

EDH-756



Current Data Parameters

NAME	may-11
EXPNO	10
PROCNO	1

F2 - Acquisition Parameters

Date_	20110502
Time	19.38
INSTRUM	spect
PROBHD	5 mm Dual 13C/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	2048
DS	2
SWH	31847.133 Hz
FIDRES	0.485949 Hz
AQ	1.0289652 sec
RG	3649.1
DW	15.700 usec
DE	6.00 usec
TE	298.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	10.50 usec
PL1	-1.00 dB
SFO1	100.6254358 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	105.00 usec
PL2	-2.00 dB
PL12	18.00 dB
PL13	20.00 dB
SFO2	400.1324708 MHz

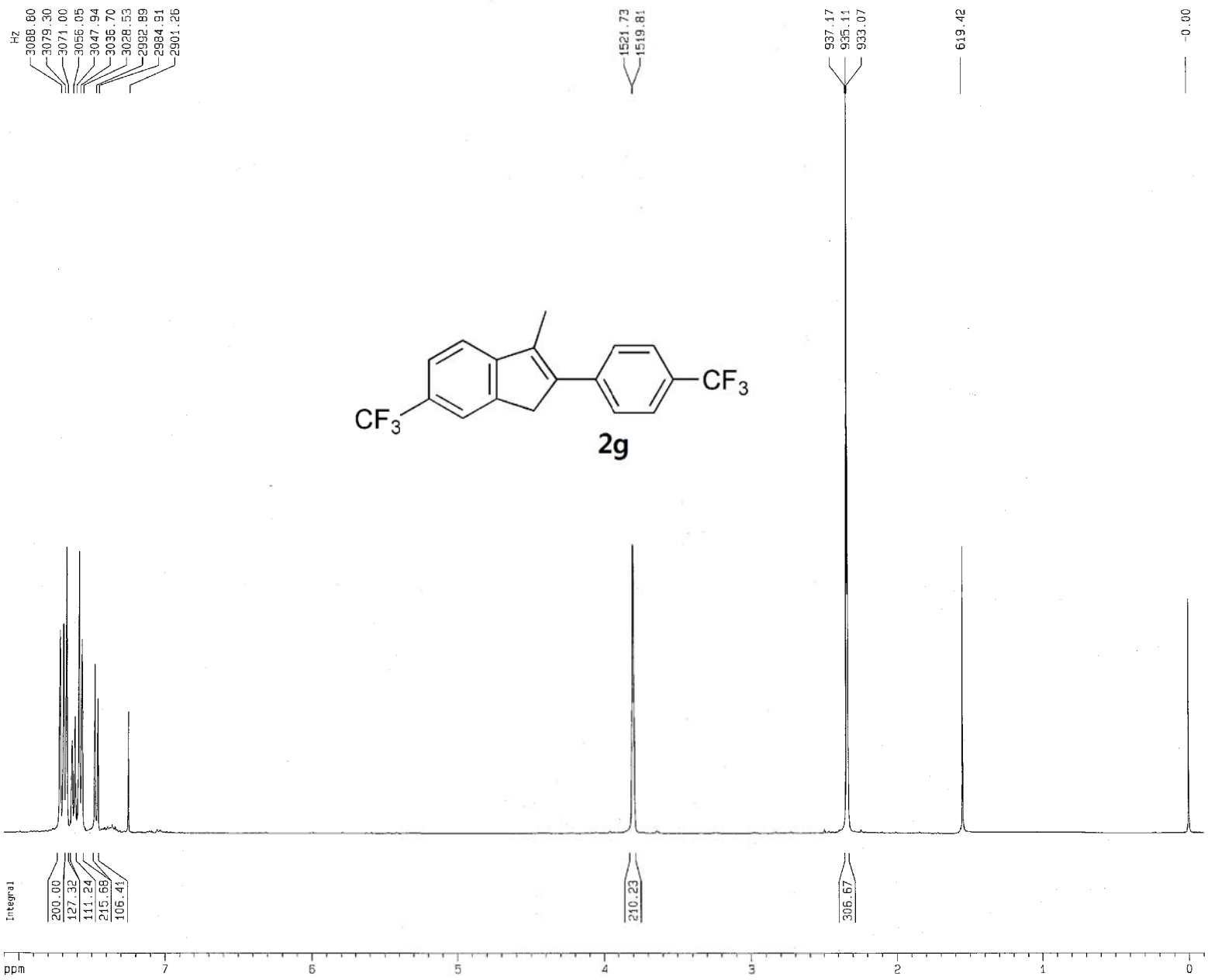
F2 - Processing parameters

SI	32768
SF	100.6127290 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	30.00 cm
CY	15.00 cm
F1P	210.000 ppm
F1	21128.67 Hz
F2P	-5.000 ppm
F2	-503.06 Hz
PPMCM	7.16667 ppm/cm
HZCM	721.05786 Hz/cm

EDH-728



Current Data Parameters

NAME	mar-11
EXPNO	72
PROCNO	1

F2 - Acquisition Parameters

Date_	20110405
Time	15.10
INSTRUM	spect
PROBHD	5 mm Dual 13C/
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	0
SWH	8223.685 Hz
FIDRES	0.250967 Hz
AQ	1.9923444 sec
RG	228.1
DW	60.800 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	11.00 usec
PL1	-2.00 dB
SFO1	400.1324710 MHz

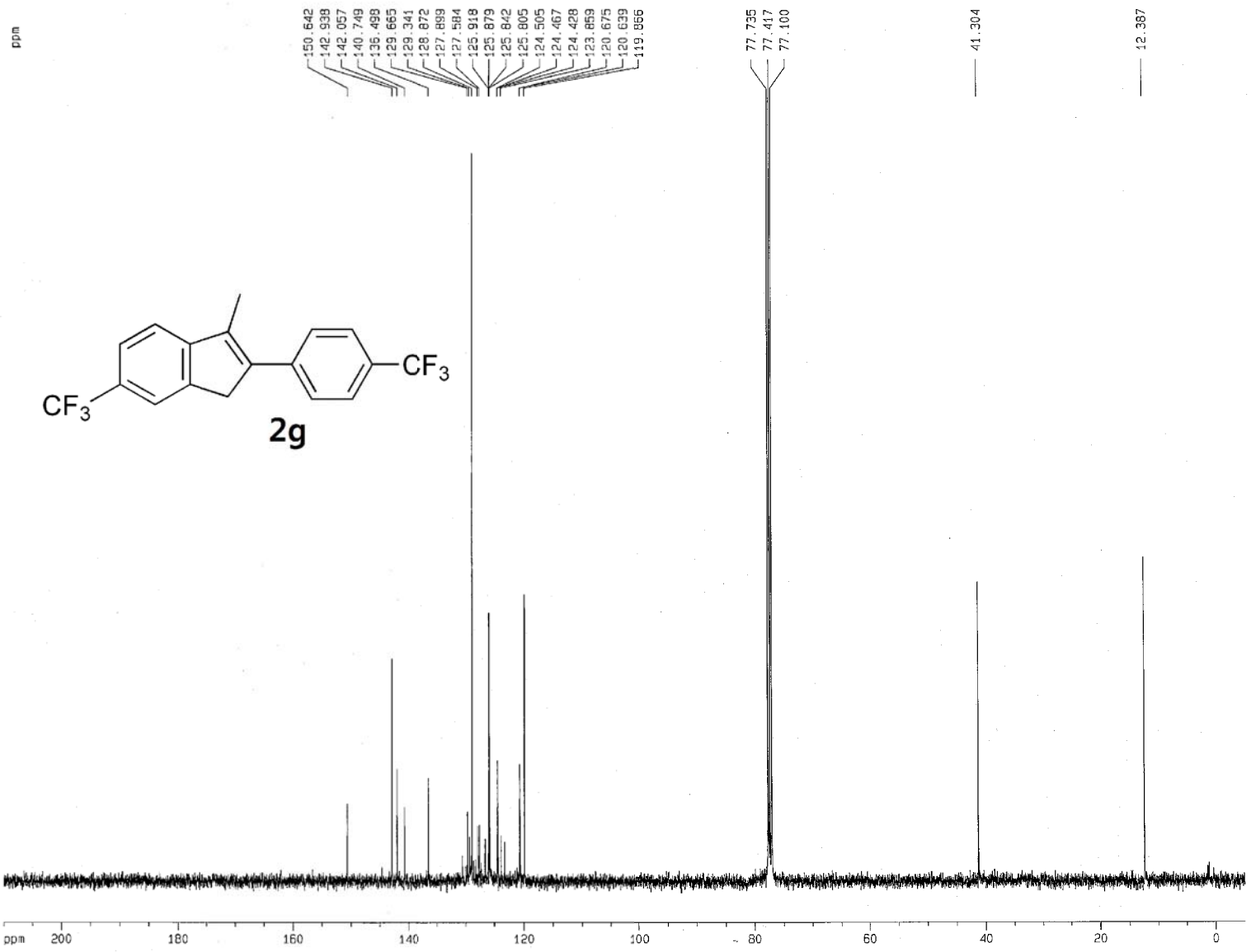
F2 - Processing parameters

SI	16384
SF	400.1300131 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	30.00 cm
CY	20.00 cm
F1P	8.100 ppm
F1	3241.05 Hz
F2P	-0.100 ppm
F2	-40.01 Hz
PPMCM	0.27333 ppm/cm
HZCM	109.36887 Hz/cm

EDH-728



Current Data Parameters
NAME mar-11
EXPNO 73
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110405
Time 15.15
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1005
DS 2
SWH 31847.133 Hz
FIDRES 0.485949 Hz
AQ 1.0289652 sec
RG 1149.4
DW 15.700 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

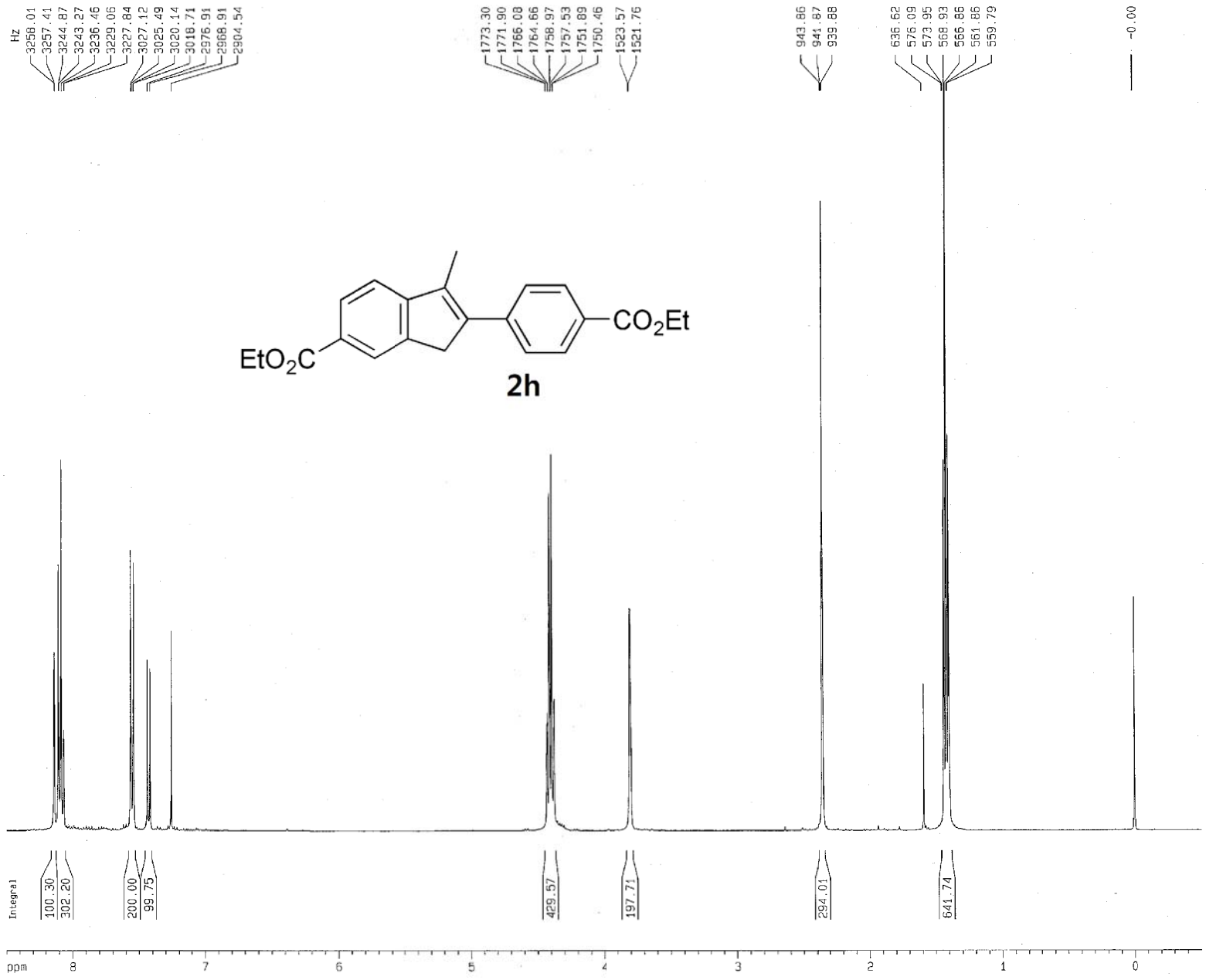
======== CHANNEL f1 ========
NUC1 13C
P1 10.50 usec
PL1 -1.00 dB
SFO1 100.6254358 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 105.00 usec
PL2 -2.00 dB
PL12 18.00 dB
PL13 20.00 dB
SFO2 400.1324708 MHz

F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 30.00 cm
CY 30.00 cm
F1P 210.000 ppm
F1 21128.57 Hz
F2P -5.000 ppm
F2 -503.06 Hz
PPMCM 7.16667 ppm/cm
HZCM 721.05786 Hz/cm

PSJ-538



Current Data Parameters

NAME	sep-11
EXPNO	51
PROCNO	1

F2 - Acquisition Parameters

Date_	20111024
Time	12.39
INSTRUM	spect
PROBHD	5 mm Dual 13C/
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	0
SWH	8223.685 Hz
FIDRES	0.250967 Hz
AQ	1.9923444 sec
RG	256
DW	60.800 usec
DE	6.00 usec
TE	298.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	11.00 usec
PL1	-2.00 dB
SFO1	400.1324710 MHz

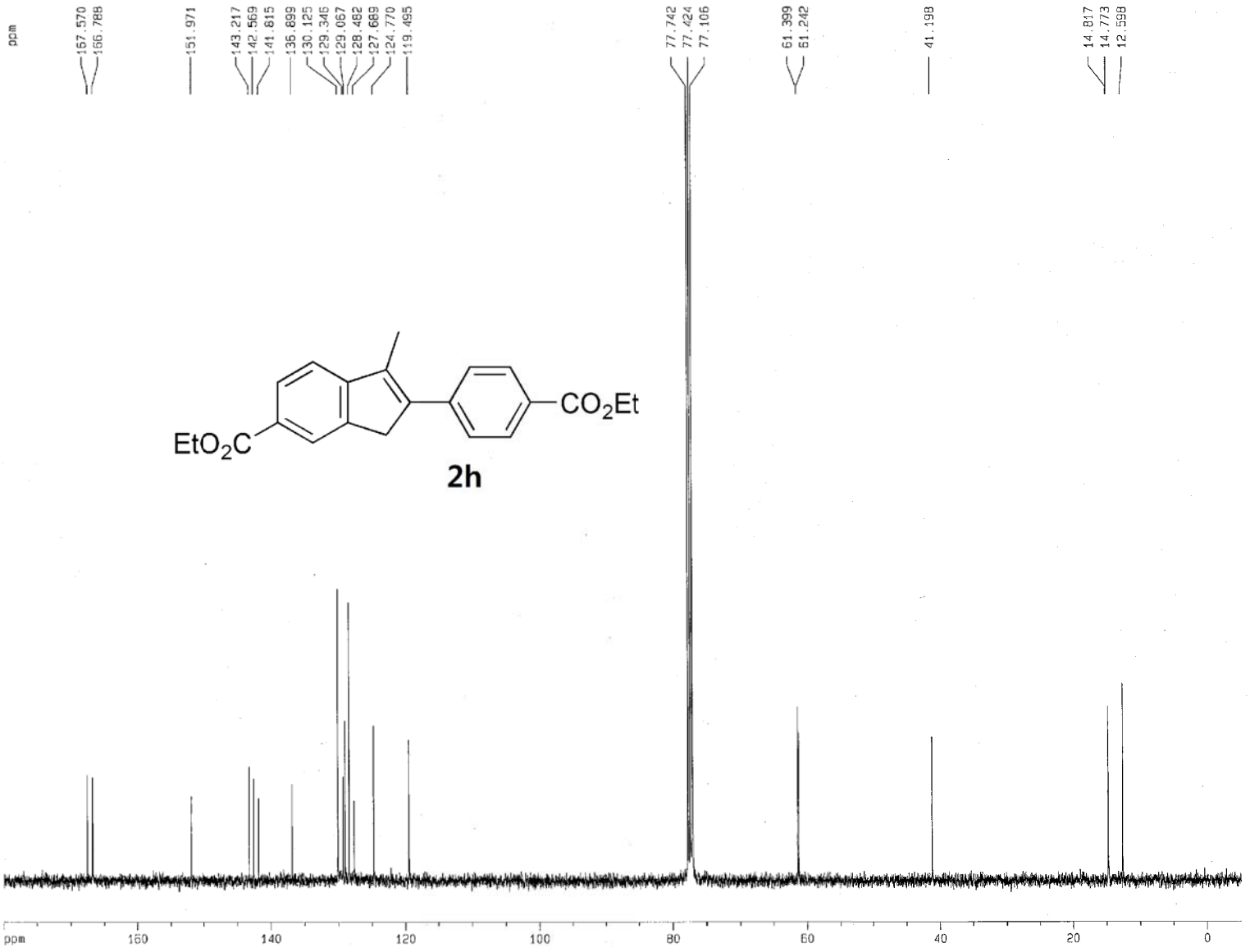
F2 - Processing parameters

SI	16384
SF	400.1300098 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

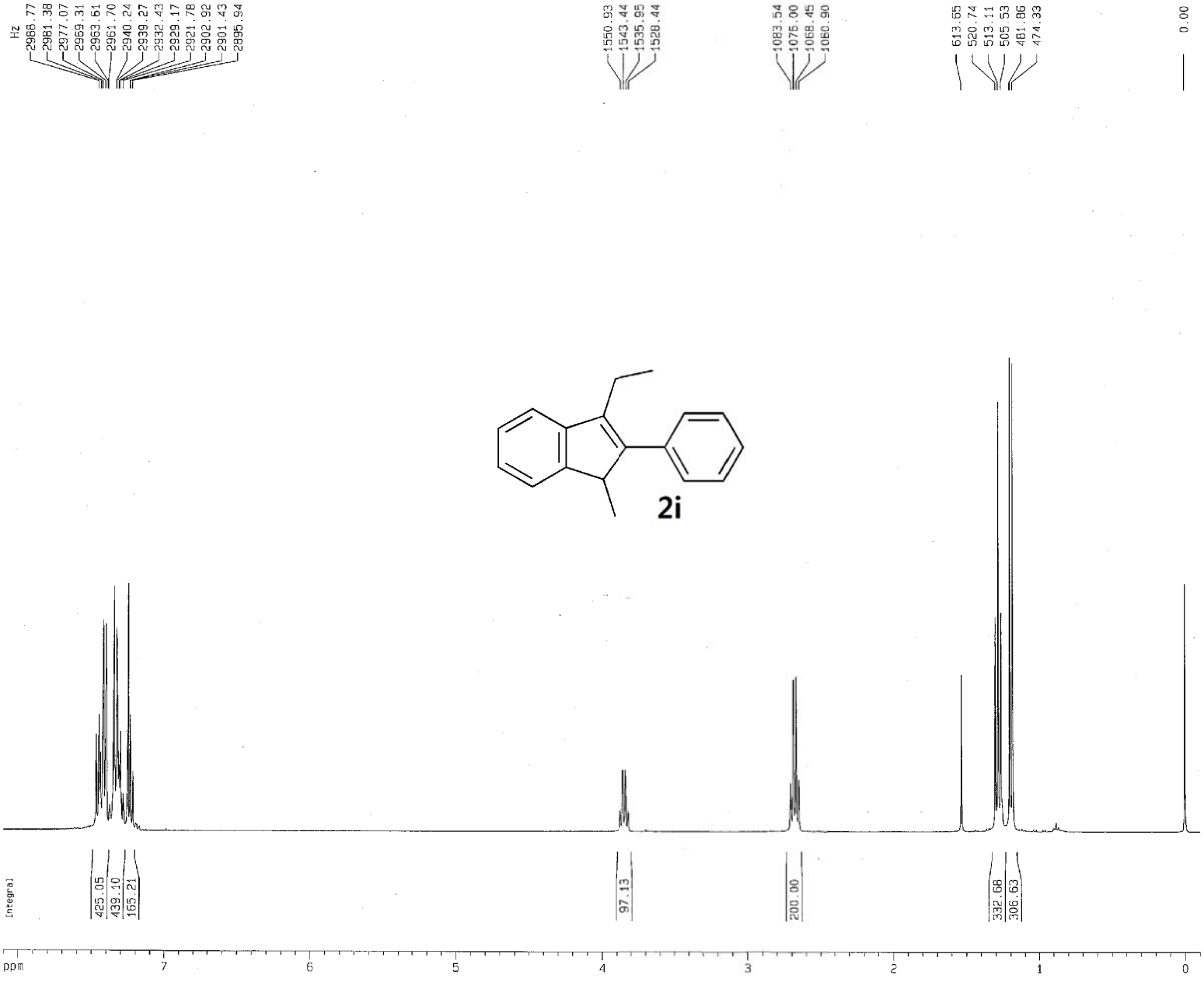
CX	30.00 cm
CY	20.00 cm
F1P	8.500 ppm
F1	3401.10 Hz
F2P	-0.500 ppm
F2	-200.07 Hz
PPMCM	0.30000 ppm/cm
HZCM	120.03900 Hz/cm

PSJ-538




```
Current Data Parameters
NAME                  sep-11
EXPNO                     52
PROCNO                     1

F2 - Acquisition Parameters
Date_               20111024
Time                   13.33
INSTRUM                spect
PROBHD   5 mm Dual 13C/
PULPROG                zgpg30
TD                      65536
SOLVENT                CDCl3
NS                      1024
DS                         2
SWH                31847.133 Hz
FIDRES             0.485949 Hz
AQ                 1.0289652 sec
RG                     574.7
DW                    15.700 usec
DE                     6.00 usec
TE                     298.2 K
D1                 2.00000000 sec
d11                0.03000000 sec
DELTA              1.89999998 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                      10.50 usec
PL1                     -1.00 dB
SFO1              100.6254358 MHz

======== CHANNEL f2 ========
CPDPRG2               waltz16
NUC2                      1H
PCPD2                  105.00 usec
PL2                     -2.00 dB
PL12                    18.00 dB
PL13                    20.00 dB
SFO2              400.1324708 MHz

F2 - Processing parameters
SI                        32768
SF                100.6127290 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                        30.00 cm
CY                        30.00 cm
F1P                      180.000 ppm
F1                     18110.29 Hz
F2P                       -5.000 ppm
F2                      -503.06 Hz
PPMCM                   6.16667 ppm/cm
HZCM                  620.44519 Hz/cm
```

PSJ-446



Current Data Parameters

NAME	may-11
EXPNO	109
PROCNO	1

F2 - Acquisition Parameters

Date_	20110630
Time	14.43
INSTRUM	spect
PROBHD	5 mm Dual 13C/
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	0
SWH	8223.685 Hz
FIDRES	0.250967 Hz
AQ	1.9923444 sec
RG	322.5
DW	60.800 usec
DE	6.00 usec
TE	296.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	11.00 usec
PL1	-2.00 dB
SFO1	400.1324710 MHz

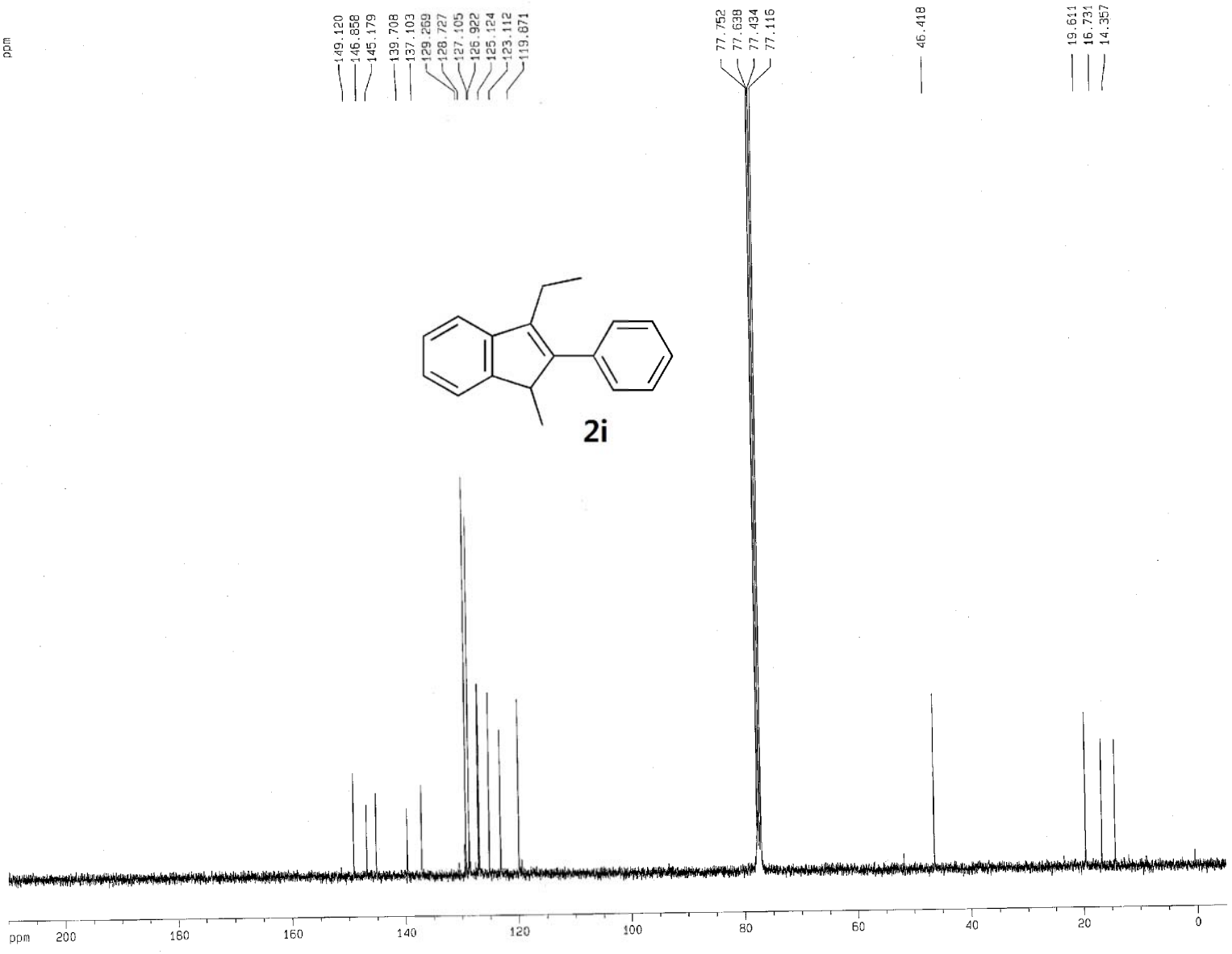
F2 - Processing parameters

SI	16384
SF	400.1300131 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	30.00 cm
CY	12.00 cm
F1P	8.100 ppm
F1	3241.05 Hz
F2P	-0.100 ppm
F2	-40.01 Hz
PPMCM	0.27333 ppm/cm
HZCM	109.36887 Hz/cm

PSJ-446



Current Data Parameters

NAME	may-11
EXPNO	110
PROCNO	1

F2 - Acquisition Parameters

Date_	20110630
Time	15.03
INSTRUM	spect
PROBHD	5 mm Dual 13C/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	2048
DS	2
SWH	31647.133 Hz
FIDRES	0.485949 Hz
AQ	1.0289652 sec
RG	1448.2
DW	15.700 usec
DE	6.00 usec
TE	297.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	10.50 usec
PL1	-1.00 dB
SFO1	100.6254358 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	105.00 usec
PL2	-2.00 dB
PL12	18.00 dB
PL13	20.00 dB
SFO2	400.1324708 MHz

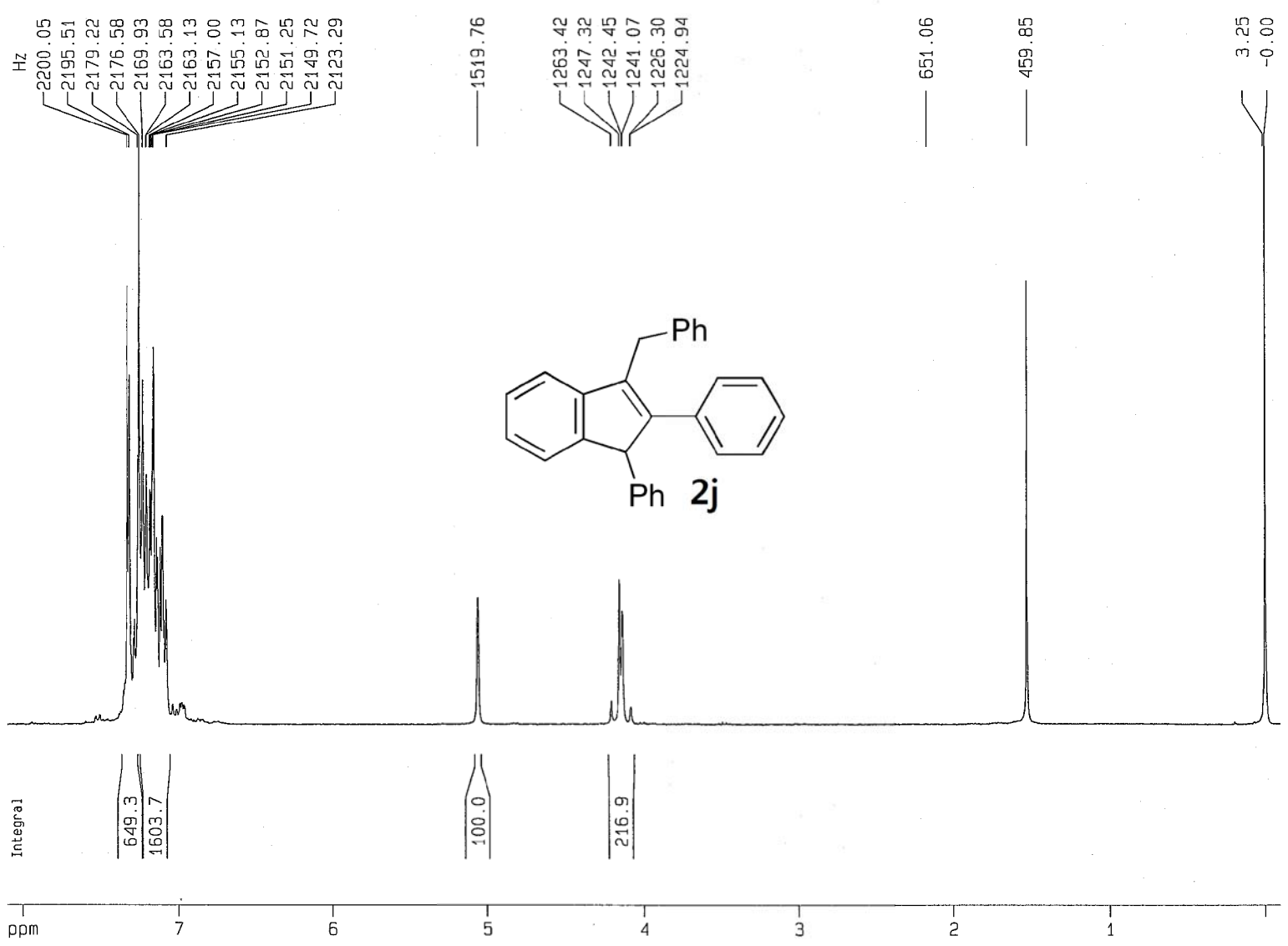
F2 - Processing parameters

SI	32768
SF	100.6127290 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	30.00 cm
CY	34.00 cm
F1P	210.000 ppm
F1	21126.67 Hz
F2P	-5.000 ppm
F2	-503.06 Hz
PPMCM	7.16667 ppm/cm
HZCM	721.05786 Hz/cm

PSJ-465



Current Data Parameters

NAME	aug-11
EXPNO	47
PROCNO	1

F2 - Acquisition Parameters

Date_	20110803
Time	11.37
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	645.1
DW	81.000 usec
DE	6.00 usec
TE	0.0 K
D1	1.00000000 sec

======== CHANNEL f1 ========

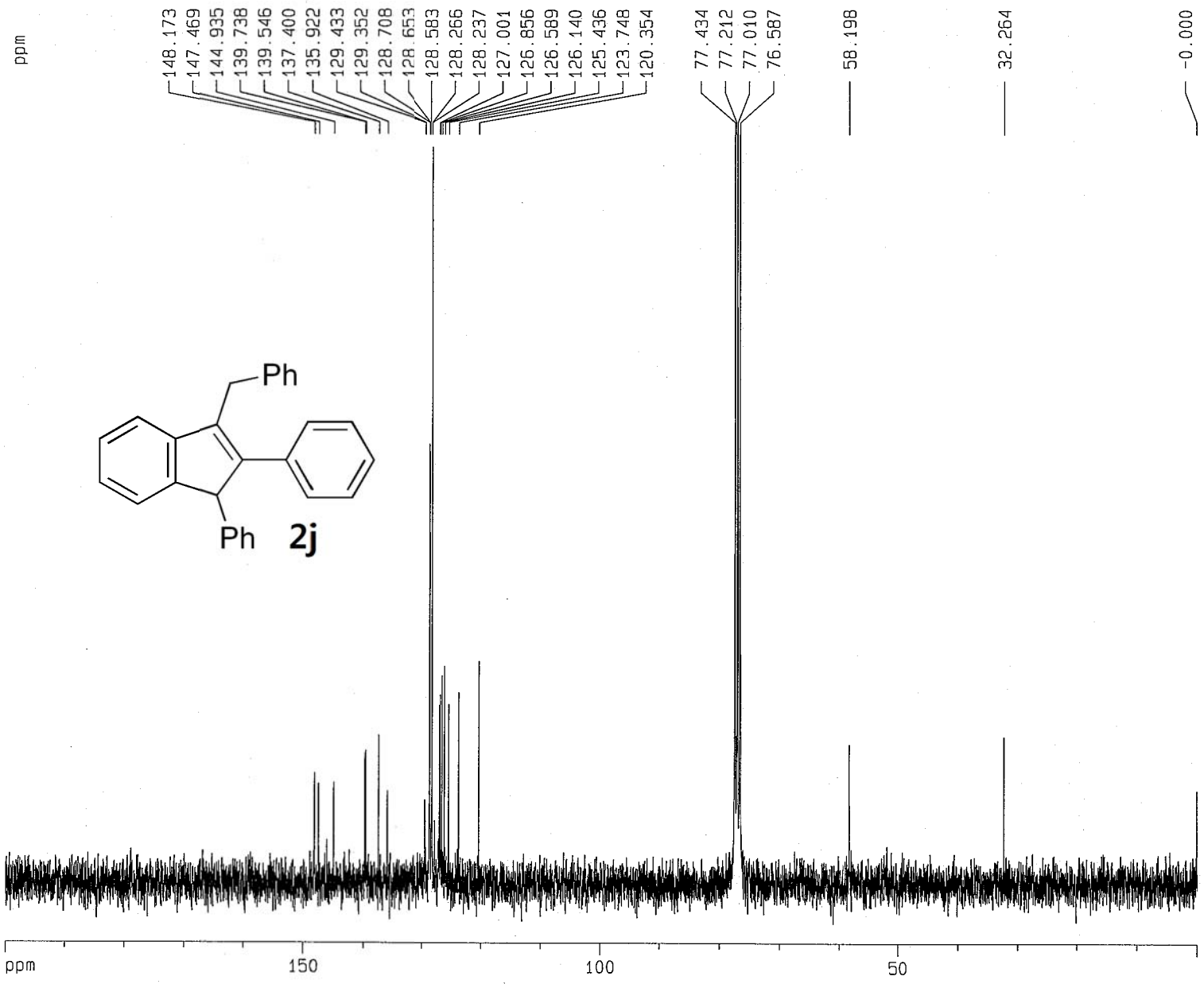
NUC1	1H
P1	10.20 usec
PL1	0.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	32768
SF	300.1300091 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	21.00 cm
CY	12.00 cm
F1P	8.100 ppm
F1	2431.05 Hz
F2P	-0.100 ppm
F2	-30.01 Hz
PPMCM	0.39048 ppm/cm
HZCM	117.19363 Hz/cm



Current Data Parameters

NAME	aug-11
EXPNO	49
PROCNO	1

F2 - Acquisition Parameters

Date_	20110803
Time	12.42
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1501
DS	0
SWH	22675.736 Hz
FIDRES	0.346004 Hz
AQ	1.4451188 sec
RG	1625.5
DW	22.050 usec
DE	6.00 usec
TE	0.0 K
D1	2.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	8.00 usec
PL1	2.00 dB
SFO1	75.4760505 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	0.00 dB
PL12	20.00 dB
PL13	22.00 dB
SFO2	300.1312005 MHz

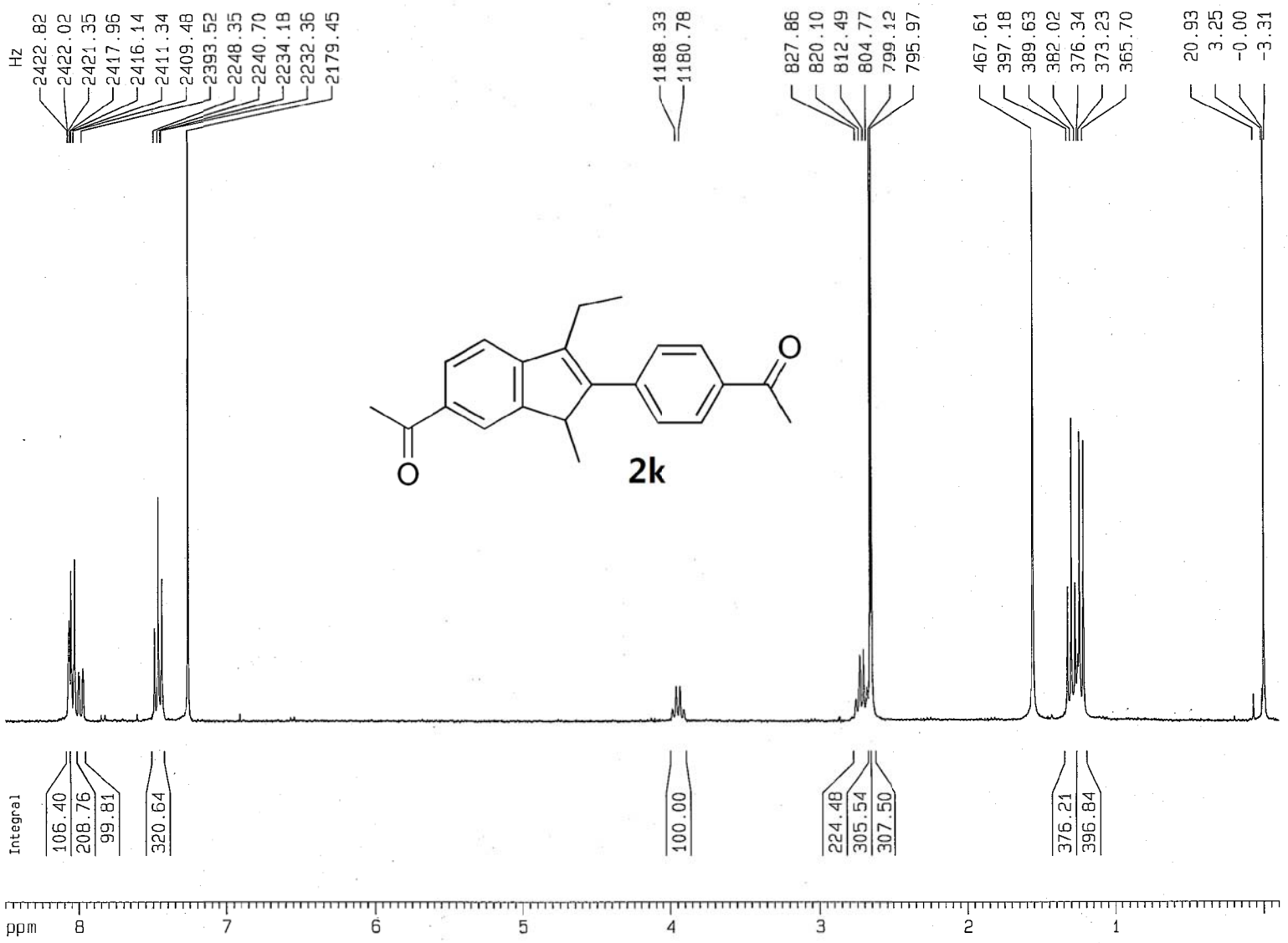
F2 - Processing parameters

SI	32768
SF	75.4677503 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	60.00 cm
F1P	200.000 ppm
F1	15093.55 Hz
F2P	-0.100 ppm
F2	-7.55 Hz
PPMCM	10.00500 ppm/cm
HZCM	755.05487 Hz/cm

PSJ-504-1



Current Data Parameters

NAME	sep-11
EXPNO	111
PROCNO	1

F2 - Acquisition Parameters

Date_	20110920
Time	10.52
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	1824.6
DW	81.000 usec
DE	6.00 usec
TE	0.0 K
D1	1.00000000 sec

======== CHANNEL f1 ========

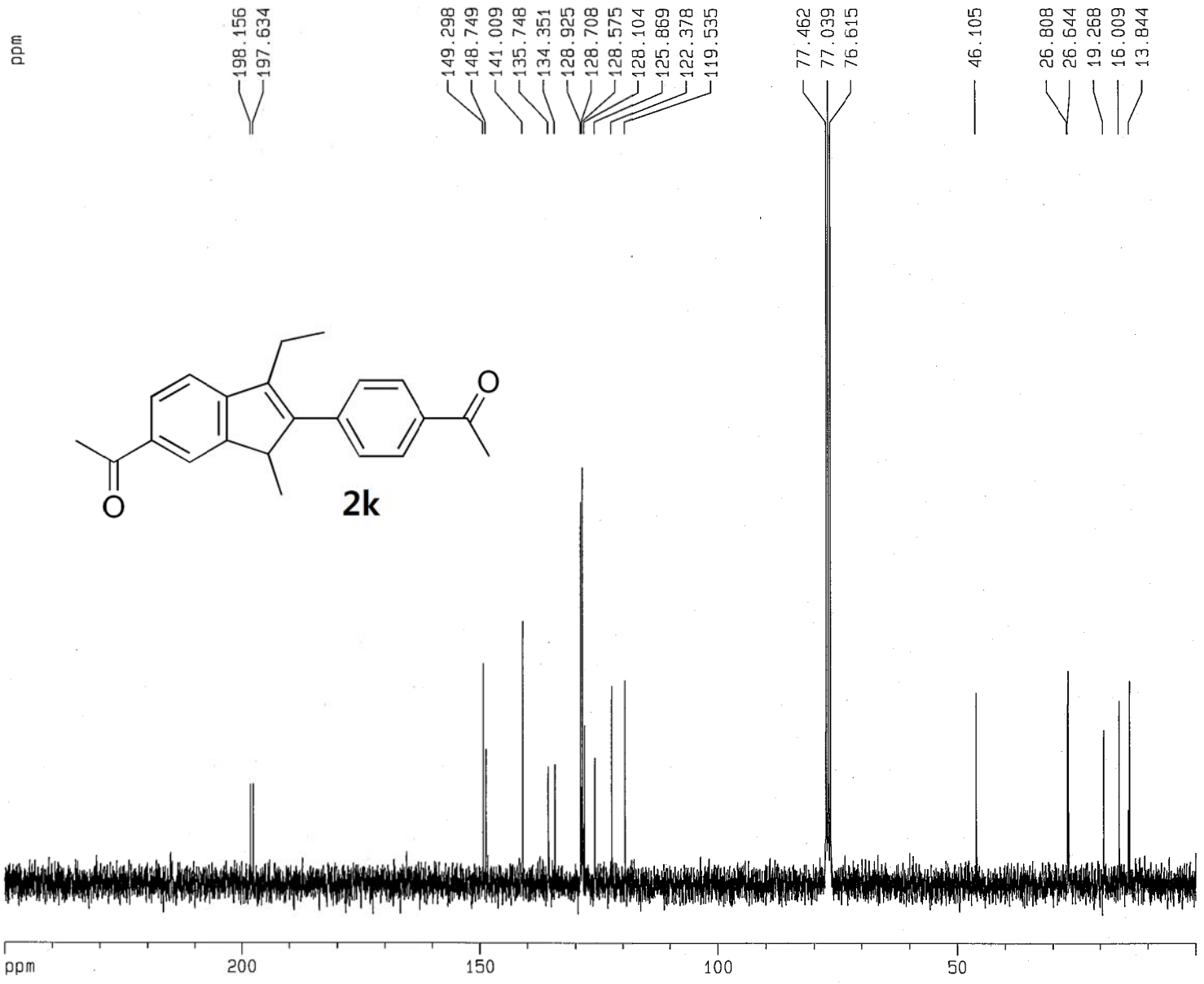
NUC1	1H
P1	10.20 usec
PL1	0.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	32768
SF	300.1300061 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	21.00 cm
CY	20.00 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.100 ppm
F2	-30.01 Hz
PPMCM	0.40952 ppm/cm
HZCM	122.91039 Hz/cm



Current Data Parameters

NAME	sep-11
EXPNO	143
PROCNO	1

F2 - Acquisition Parameters

Date_	20110922
Time	14.45
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	621
DS	0
SWH	22675.736 Hz
FIDRES	0.346004 Hz
AQ	1.4451188 sec
RG	11585.2
DW	22.050 usec
DE	6.00 usec
TE	0.0 K
D1	2.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	8.00 usec
PL1	2.00 dB
SF01	75.4760505 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	0.00 dB
PL12	20.00 dB
PL13	22.00 dB
SF02	300.1312005 MHz

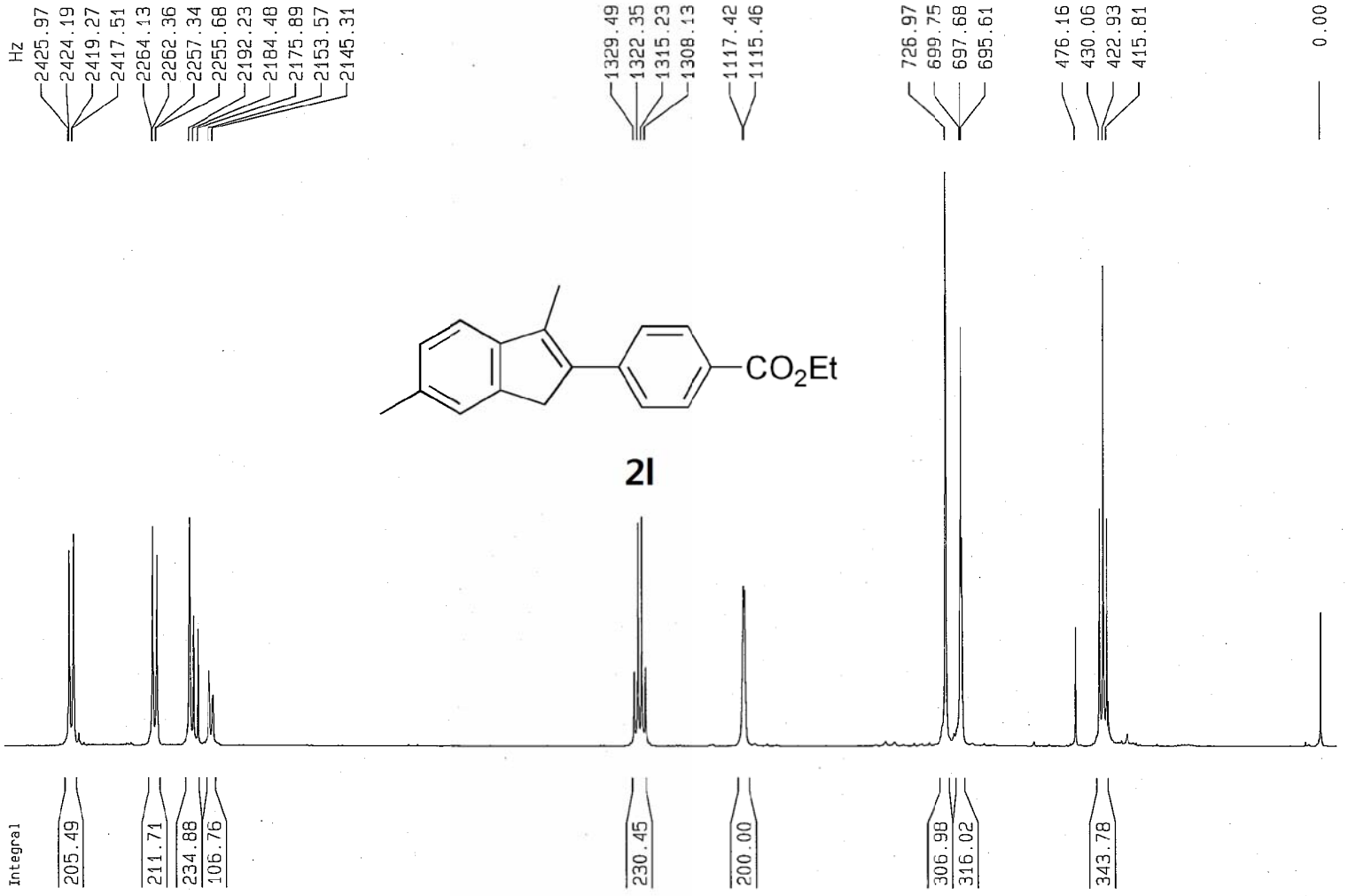
F2 - Processing parameters

SI	32768
SF	75.4677485 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	30.00 cm
F1P	250.000 ppm
F1	18866.94 Hz
F2P	-0.100 ppm
F2	-7.55 Hz
PPMCM	12.50500 ppm/cm
HZCM	943.72424 Hz/cm

PSJ-539



Current Data Parameters

NAME	oct-11
EXPNO	171
PROCNO	1

F2 - Acquisition Parameters

Date_	20111024
Time	11.26
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	287.4
DW	81.000 usec
DE	6.00 usec
TE	0.0 K
D1	1.00000000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	10.20 usec
PL1	0.00 dB
SFO1	300.1318534 MHz

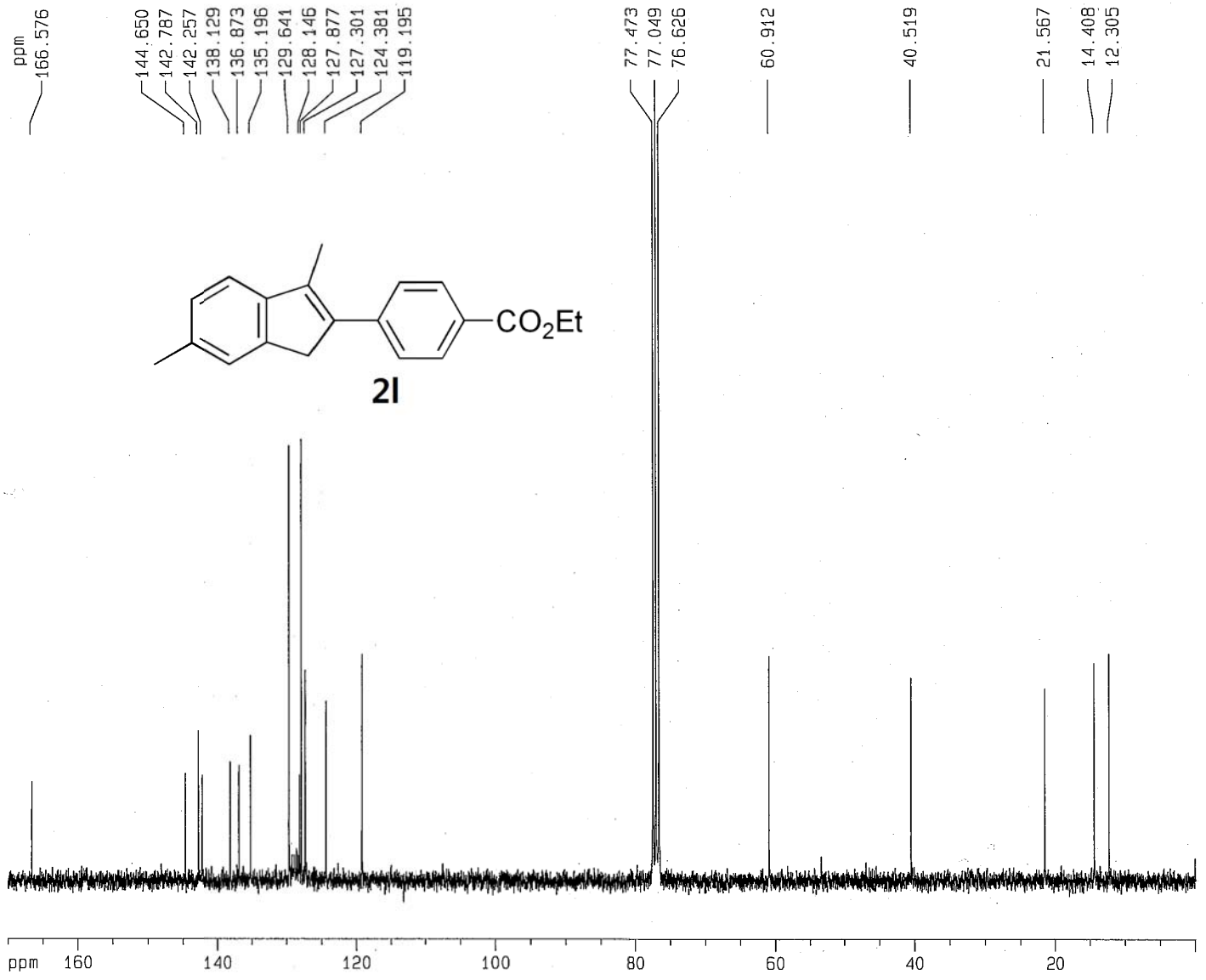
F2 - Processing parameters

SI	32768
SF	300.1300095 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	21.00 cm
CY	9.00 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.100 ppm
F2	-30.01 Hz
PPMCM	0.40952 ppm/cm
HZCM	122.91039 Hz/cm

PSJ-539-C



Current Data Parameters

NAME	oct-11
EXPNO	173
PROCNO	1

F2 - Acquisition Parameters

Date_	20111024
Time	12.39
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	745
DS	0
SWH	22675.736 Hz
FIDRES	0.346004 Hz
AQ	1.4451188 sec
RG	11585.2
DW	22.050 usec
DE	6.00 usec
TE	0.0 K
D1	2.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	8.00 usec
PL1	2.00 dB
SFO1	75.4760505 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	0.00 dB
PL12	20.00 dB
PL13	22.00 dB
SFO2	300.1312005 MHz

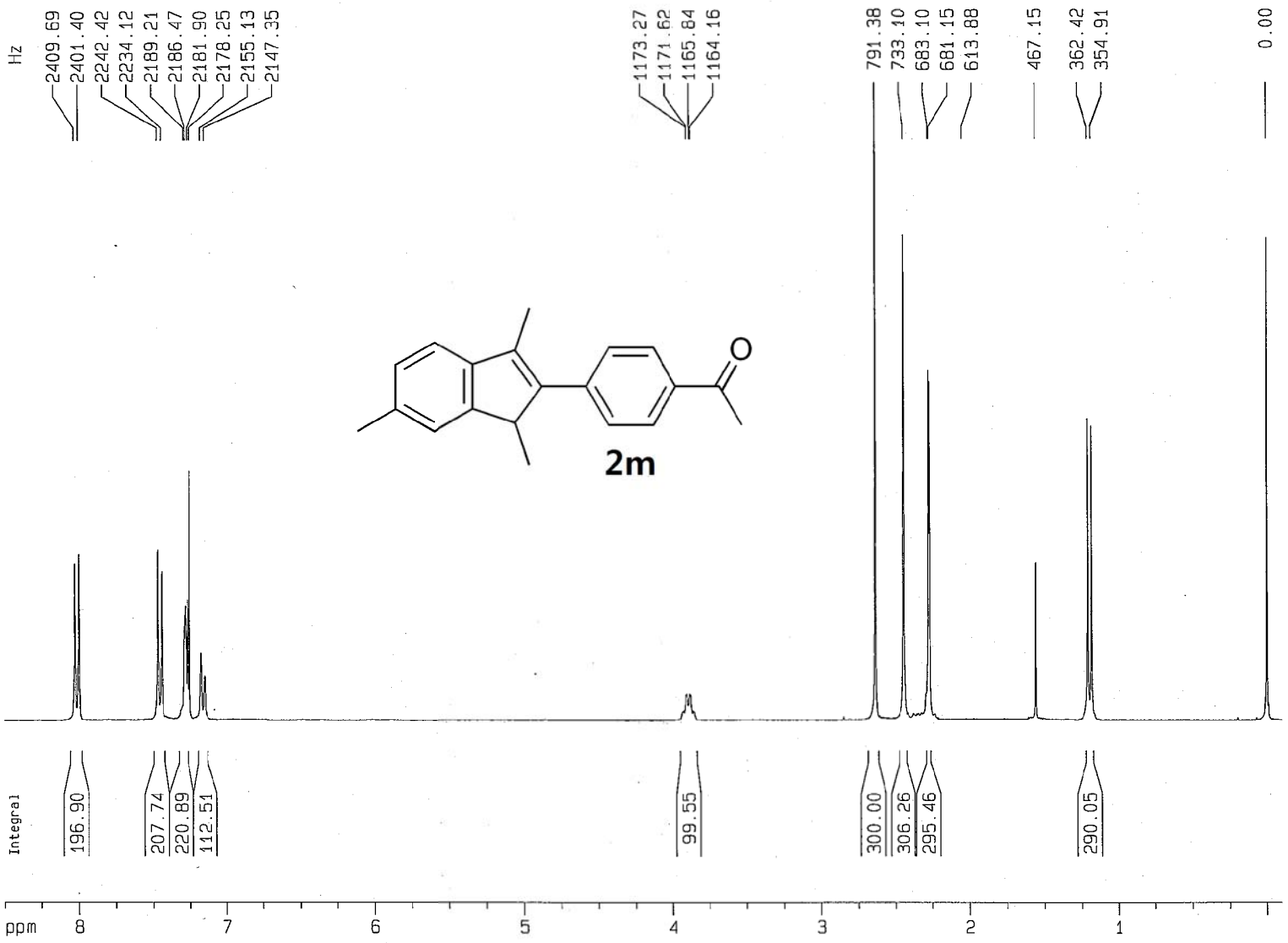
F2 - Processing parameters

SI	32768
SF	75.4677485 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	20.00 cm
F1P	170.000 ppm
F1	12829.52 Hz
F2P	-0.100 ppm
F2	-7.55 Hz
PPMCM	8.50500 ppm/cm
HZCM	641.85315 Hz/cm

PSJ-516-re-2



Current Data Parameters

NAME	sep-11
EXPNO	212
PROCNO	1

F2 - Acquisition Parameters

Date_	20110930
Time	21.07
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	724.1
DW	81.000 usec
DE	6.00 usec
TE	0.0 K
D1	1.00000000 sec

======== CHANNEL f1 ========

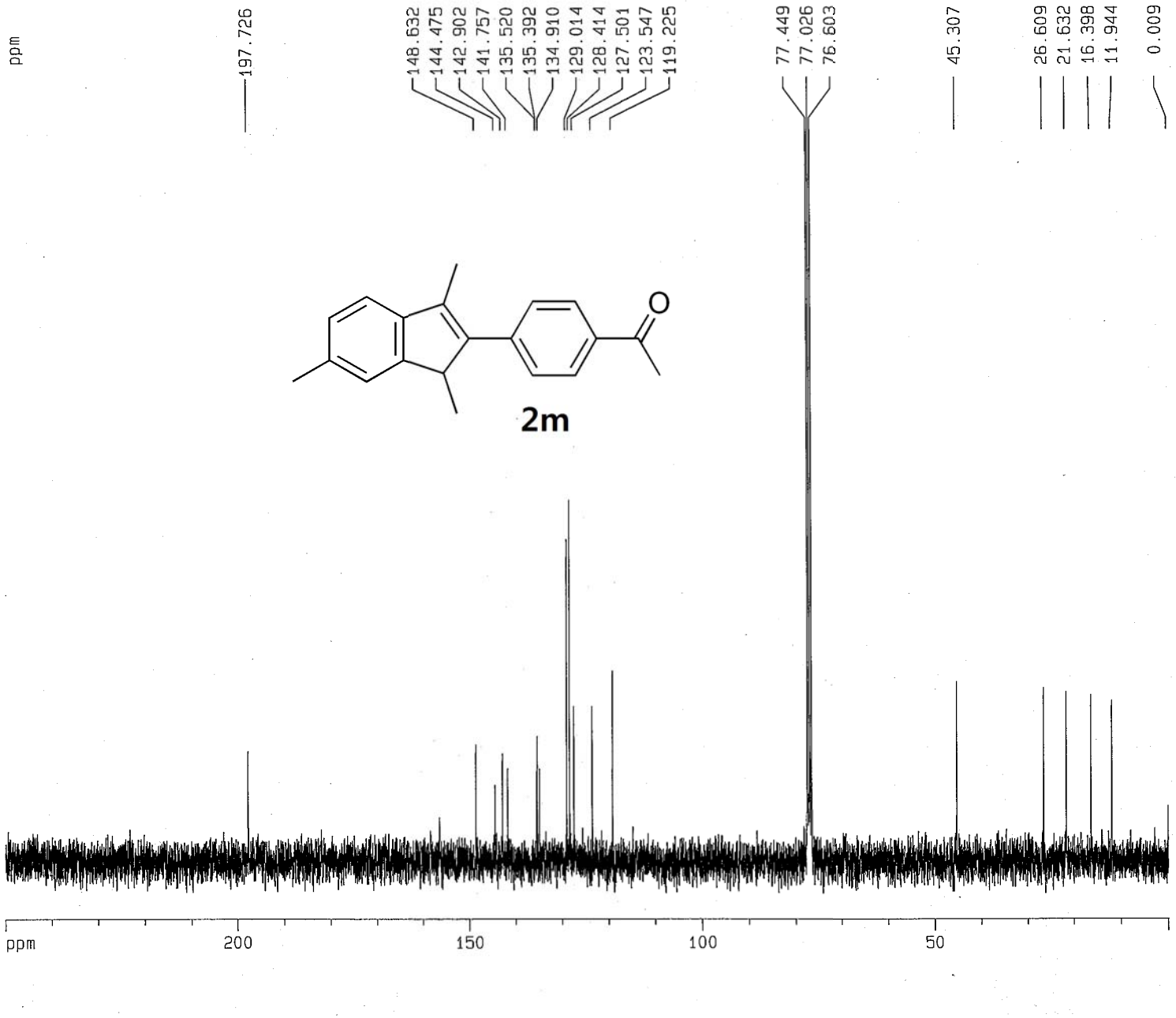
NUC1	1H
P1	10.20 usec
PL1	0.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	32768
SF	300.1300071 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	21.00 cm
CY	12.00 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.100 ppm
F2	-30.01 Hz
PPMCM	0.40952 ppm/cm
HZCM	122.91039 Hz/cm



Current Data Parameters

NAME	sep-11
EXPNO	213
PROCNO	1

F2 - Acquisition Parameters

Date_	20111001
Time	19.19
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	581
DS	0
SWH	22675.736 Hz
FIDRES	0.346004 Hz
AQ	1.4451188 sec
RG	13004
DW	22.050 usec
DE	6.00 usec
TE	0.0 K
D1	2.00000000 sec
d11	0.03000000 sec
d12	0.00002000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	8.00 usec
PL1	2.00 dB
SFO1	75.4760505 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	0.00 dB
PL12	20.00 dB
PL13	22.00 dB
SFO2	300.1312005 MHz

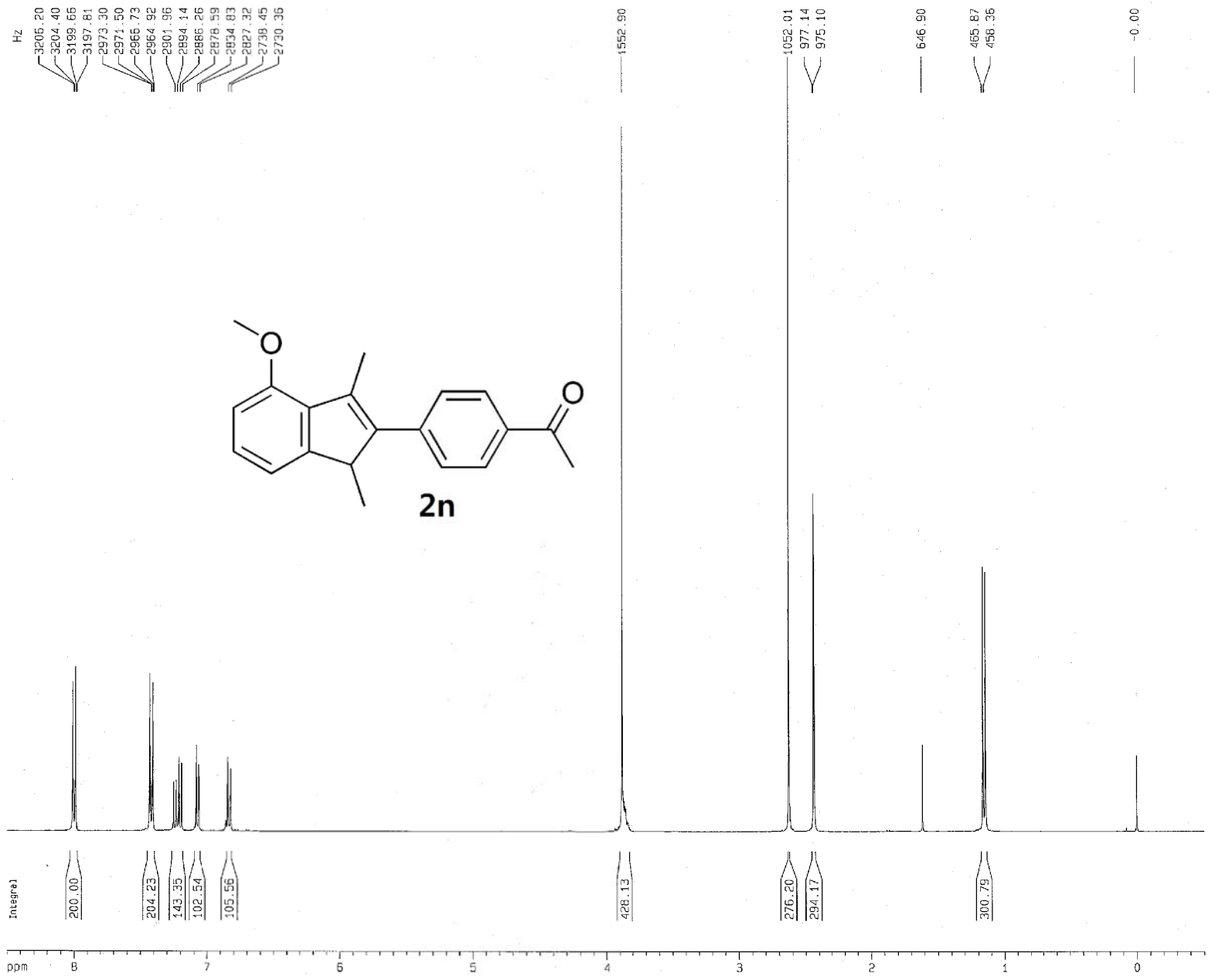
F2 - Processing parameters

SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	40.00 cm
F1P	250.000 ppm
F1	18866.94 Hz
F2P	-0.100 ppm
F2	-7.55 Hz
PPMCM	12.50500 ppm/cm
HZCM	943.72424 Hz/cm

PSJ-519



Current Data Parameters
NAME sep-11
EXPNO 21
PROCNO 1

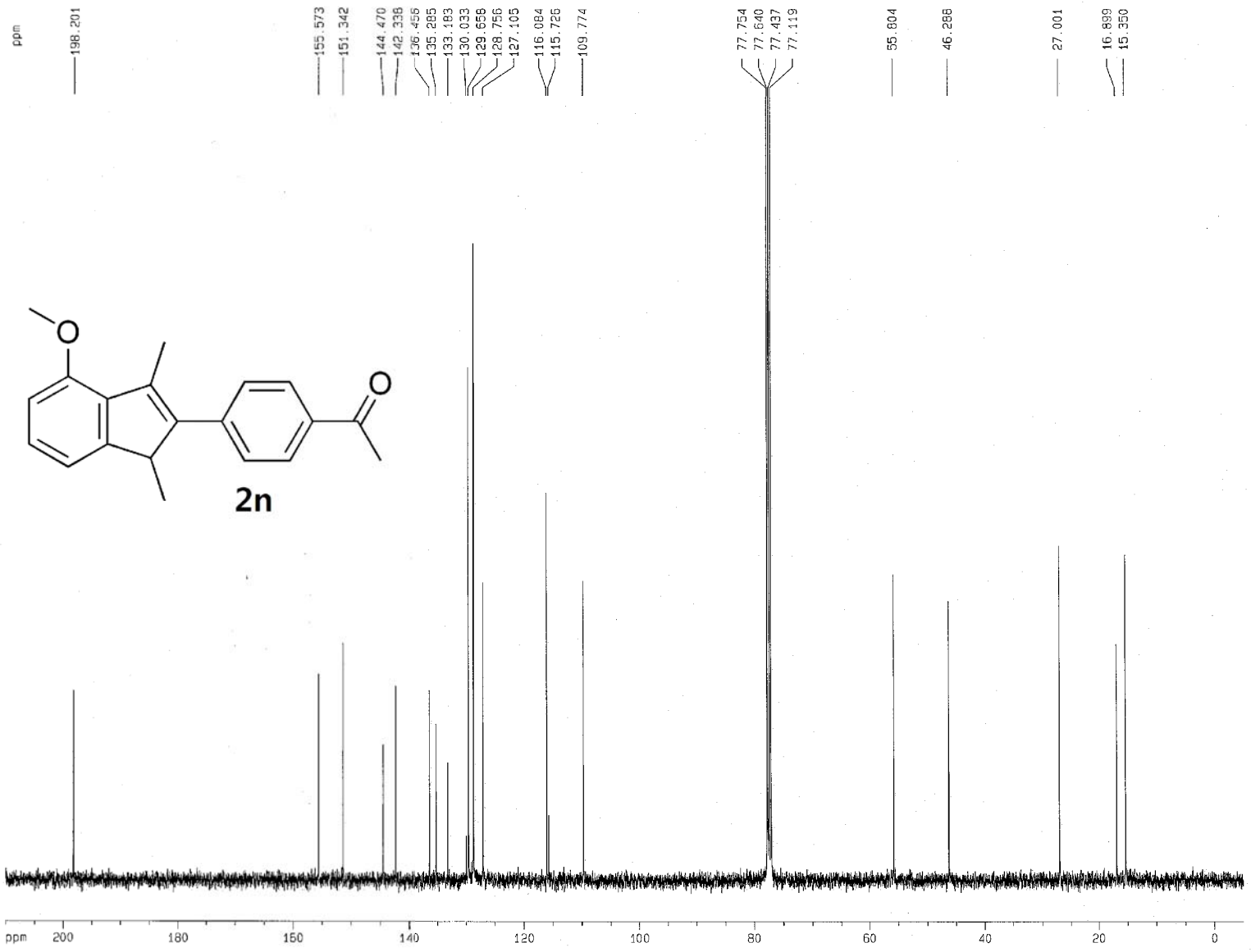
F2 - Acquisition Parameters
Date_ 20111005
Time 11.05
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 181
DW 60.800 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 16384
SF 400.1300126 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 30.00 cm
CY 20.00 cm
F1P 8.500 ppm
F1 3401.10 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.30000 ppm/cm
HZCM 120.03900 Hz/cm

PSJ-519



Current Data Parameters

NAME	sep-11
EXPNO	22
PROCNO	1

F2 - Acquisition Parameters

Date_	20111005
Time	11.15
INSTRUM	spect
PROBHD	5 mm Dual 13C/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	2
SWH	31847.133 Hz
FIDRES	0.485949 Hz
AQ	1.0289652 sec
RG	7298.2
DW	15.700 usec
DE	6.00 usec
TE	298.2 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	10.50 usec
PL1	-1.00 dB
SFO1	100.6254358 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	105.00 usec
PL2	-2.00 dB
PL12	18.00 dB
PL13	20.00 dB
SFO2	400.1324708 MHz

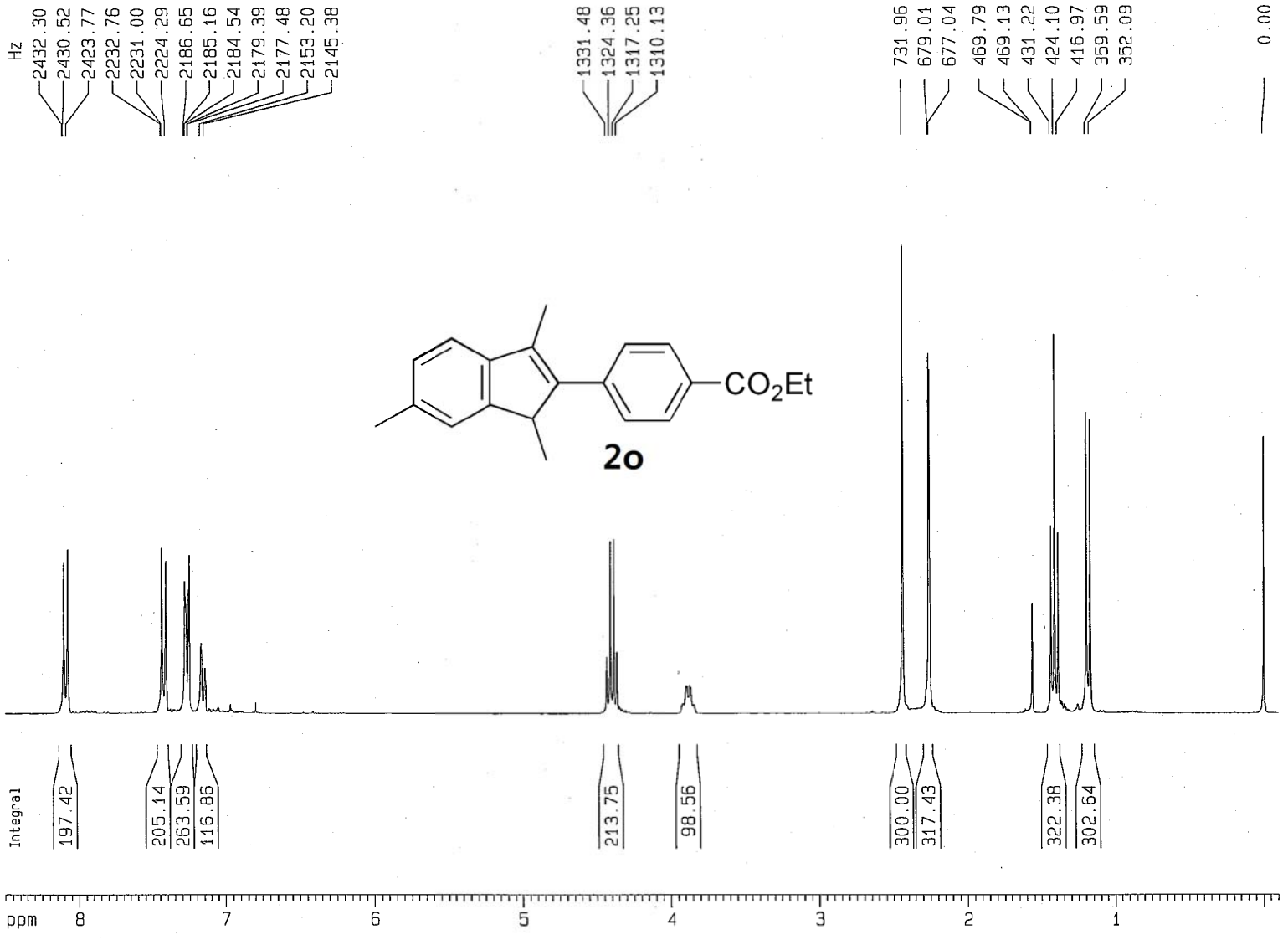
F2 - Processing parameters

SI	32768
SF	100.6127290 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	30.00 cm
CY	30.00 cm
F1P	210.000 ppm
F1	21128.67 Hz
F2P	-5.000 ppm
F2	-503.06 Hz
PPMCM	7.16667 ppm/cm
HZCM	721.05786 Hz/cm

PSJ-528



Current Data Parameters

NAME	oct-11
EXPNO	49
PROCNO	1

F2 - Acquisition Parameters

Date_	20111013
Time	13.37
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	0
SWH	6172.839 Hz
FIDRES	0.188380 Hz
AQ	2.6542580 sec
RG	574.7
DW	81.000 usec
DE	6.00 usec
TE	0.0 K
D1	1.00000000 sec

======== CHANNEL f1 ========

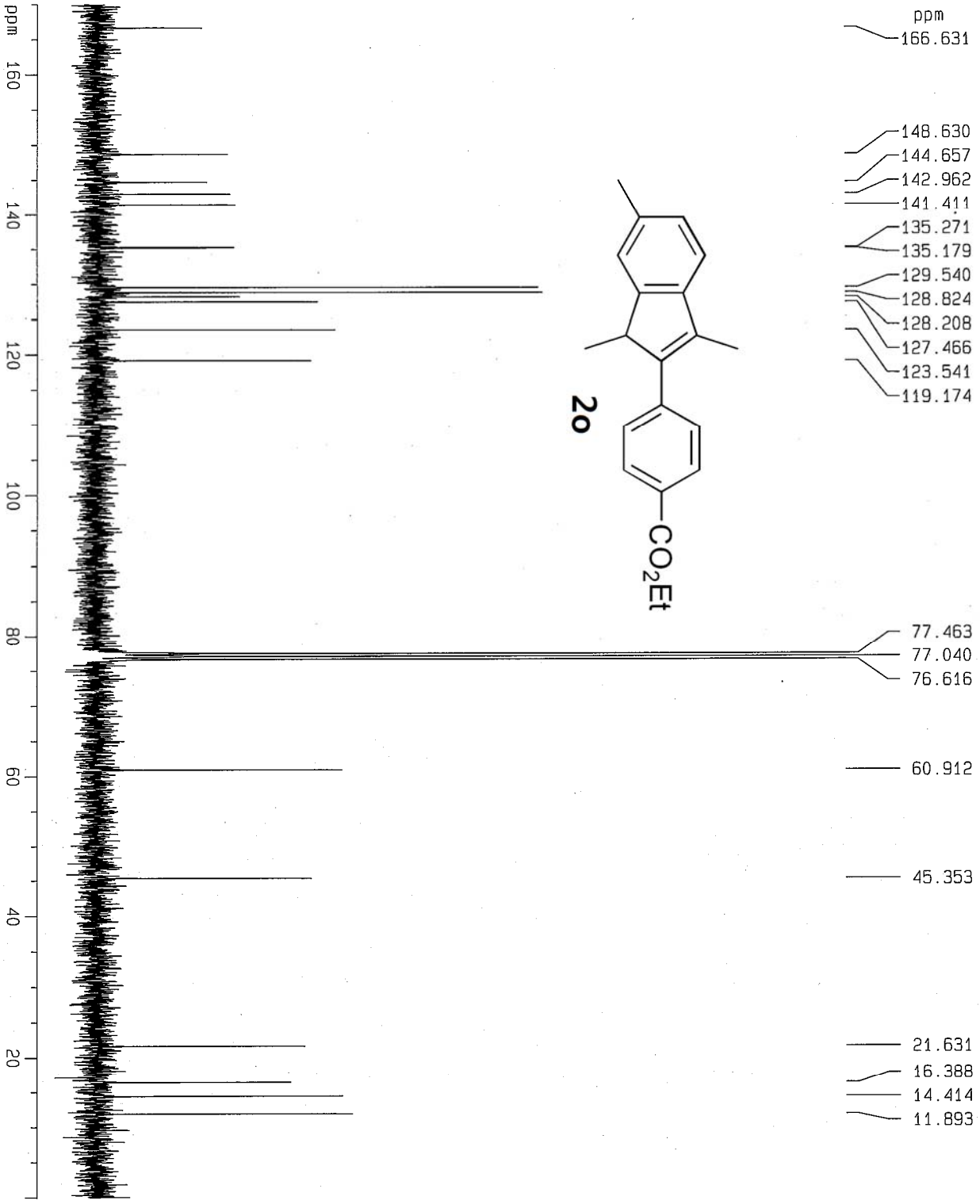
NUC1	1H
P1	10.20 usec
PL1	0.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

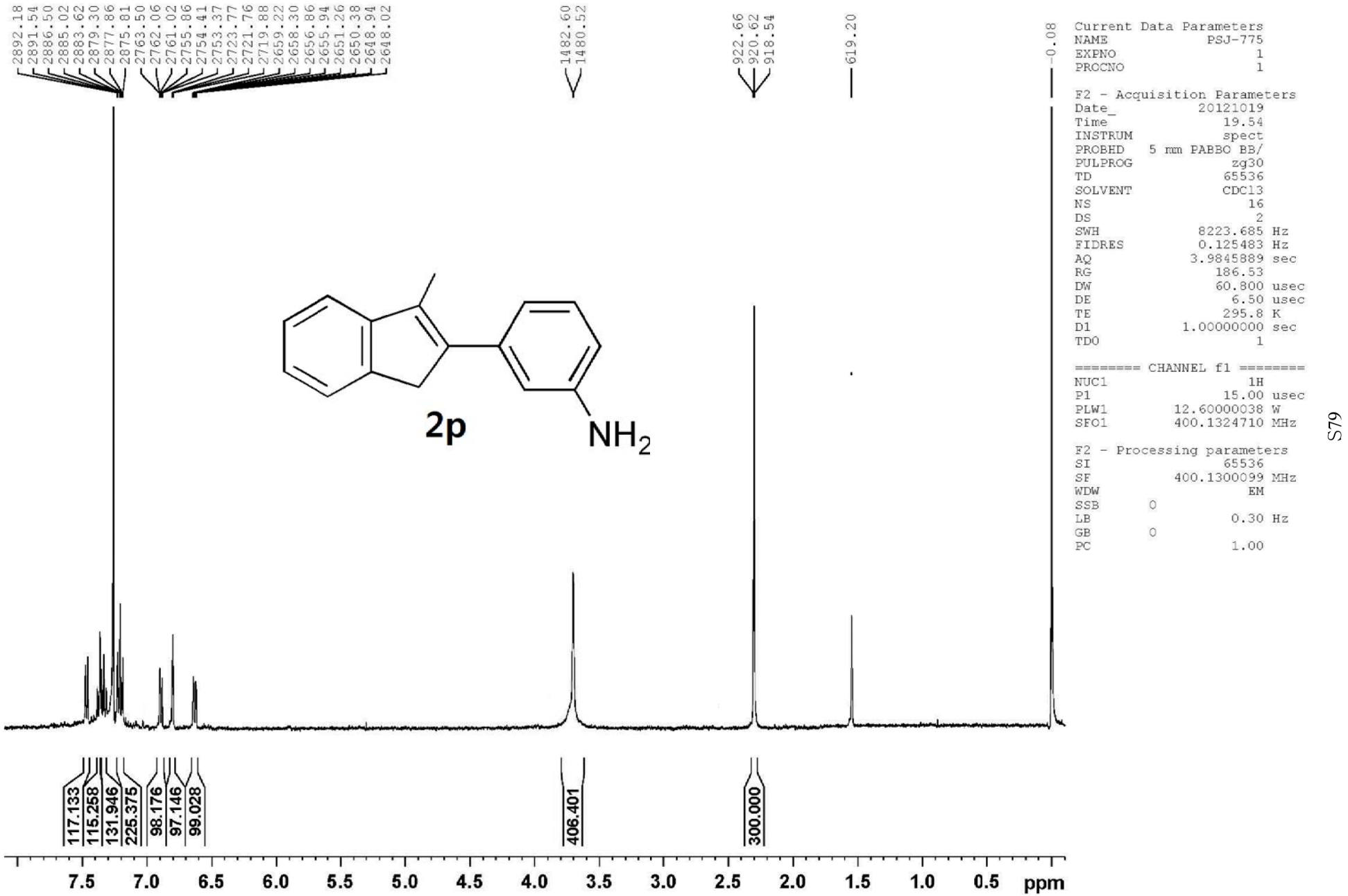
SI	32768
SF	300.1300081 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

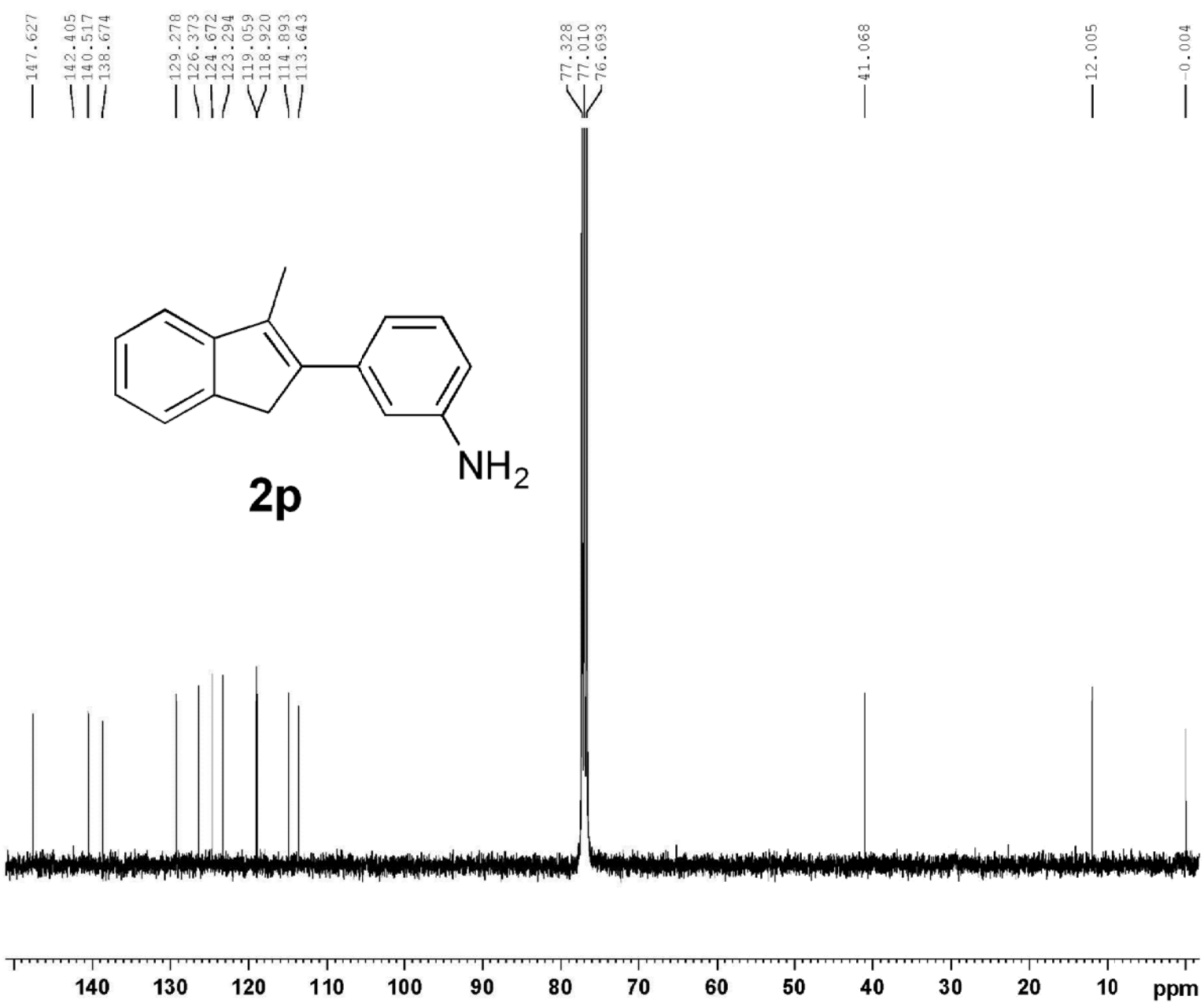
CX	21.00 cm
CY	10.00 cm
F1P	8.500 ppm
F1	2551.10 Hz
F2P	-0.100 ppm
F2	-30.01 Hz
PPMCM	0.40952 ppm/cm
HZCM	122.91039 Hz/cm



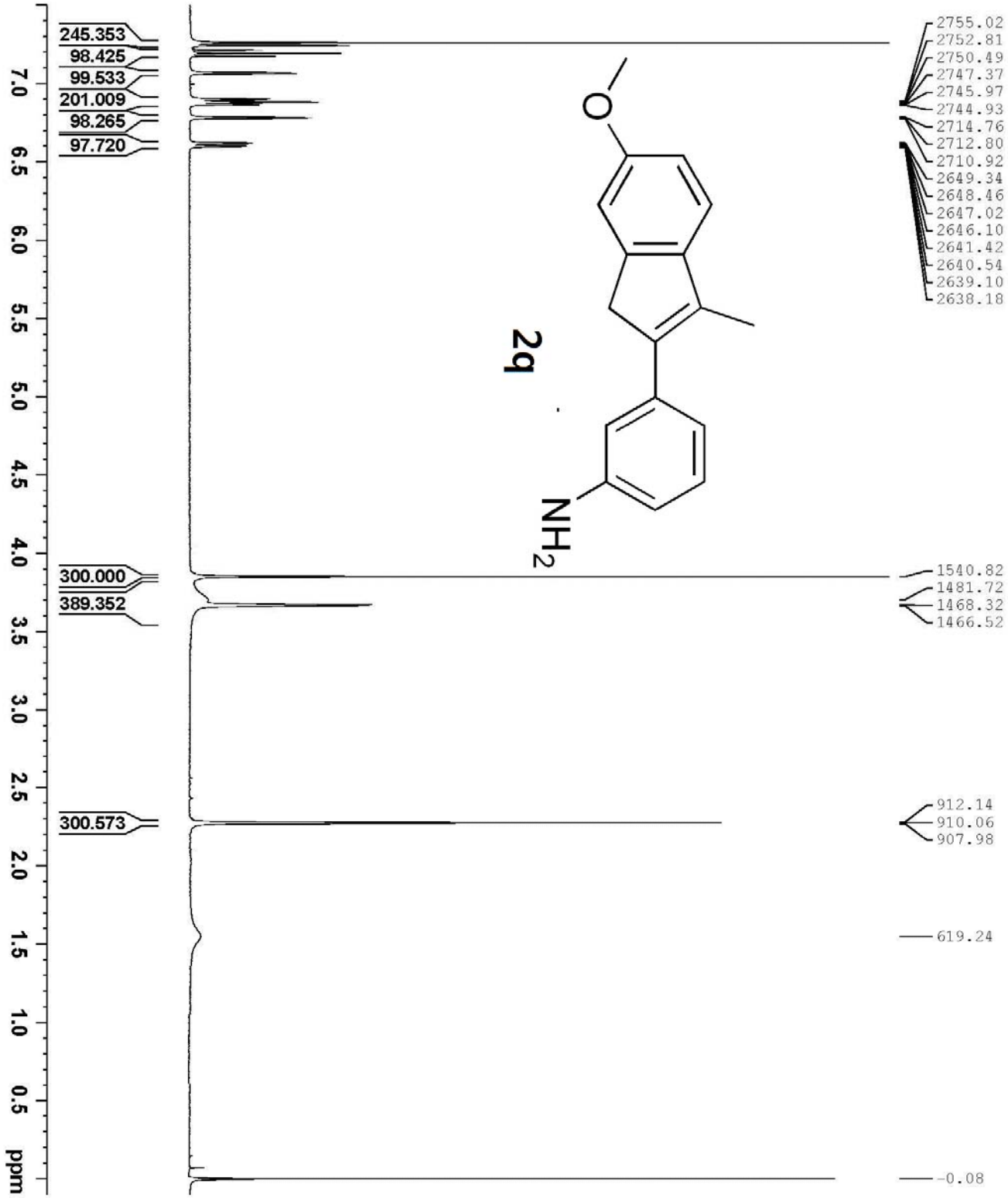

```
Current Data Parameters
NAME                  oct-11
EXPNO                     52
PROCNO                     1

F2 - Acquisition Parameters
Date_               20111013
Time                   18.24
INSTRUM                spect
PROBHD  5 mm QNP  1H/1
PULPROG                zgpg30
TD                      65536
SOLVENT                CDCl3
NS                       513
DS                         0
SWH                22675.736 Hz
FIDRES                 0.346004 Hz
AQ                  1.4451188 sec
RG                      1625.5
DW                     22.050 usec
DE                      6.00 usec
TE                       0.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
d12                 0.00002000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                       8.00 usec
PL1                      2.00 dB
SF01               75.4760505 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                  100.00 usec
PL2                      0.00 dB
PL12                    20.00 dB
PL13                    22.00 dB
SF02               300.1312005 MHz

F2 - Processing parameters
SI                       32768
SF                 75.4677485 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40

1D NMR plot parameters
CX                      20.00 cm
CY                      30.00 cm
F1P                   170.000 ppm
F1                   12829.52 Hz
F2P                    -0.100 ppm
F2                      -7.55 Hz
PPMCM                8.50500 ppm/cm
HZCM               641.85315 Hz/cm
```

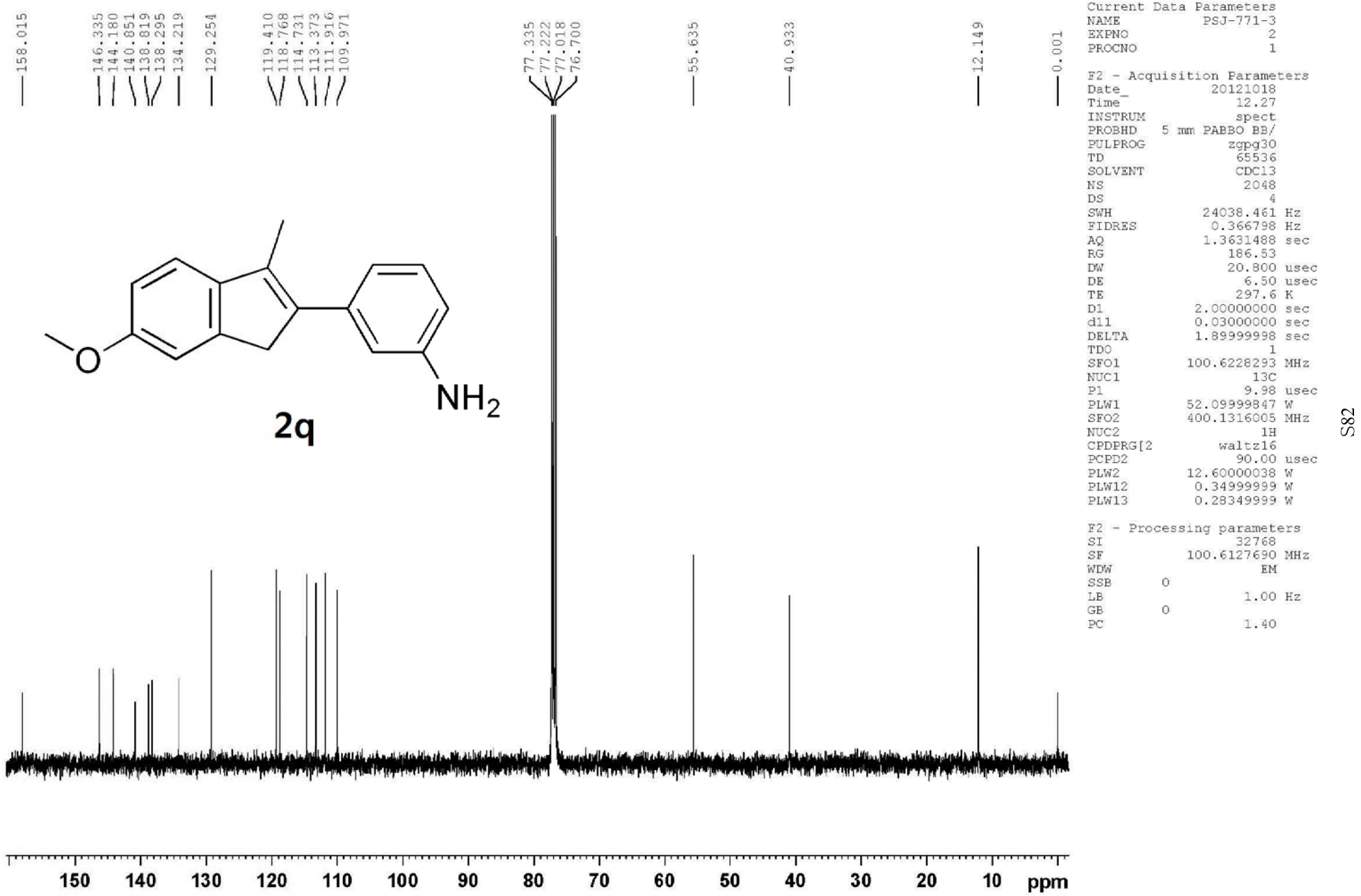



```
Current Data Parameters
NAME                  PSJ-775
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20121019
Time                  19.54
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9845889 sec
RG                   186.53
DW                   60.800 usec
DE                    6.50 usec
TE                   295.8 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                    15.00 usec
PLW1            12.60000038 W
SFO1            400.1324710 MHz

F2 - Processing parameters
SI                    65536
SF              400.1300099 MHz
WDW                      EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```

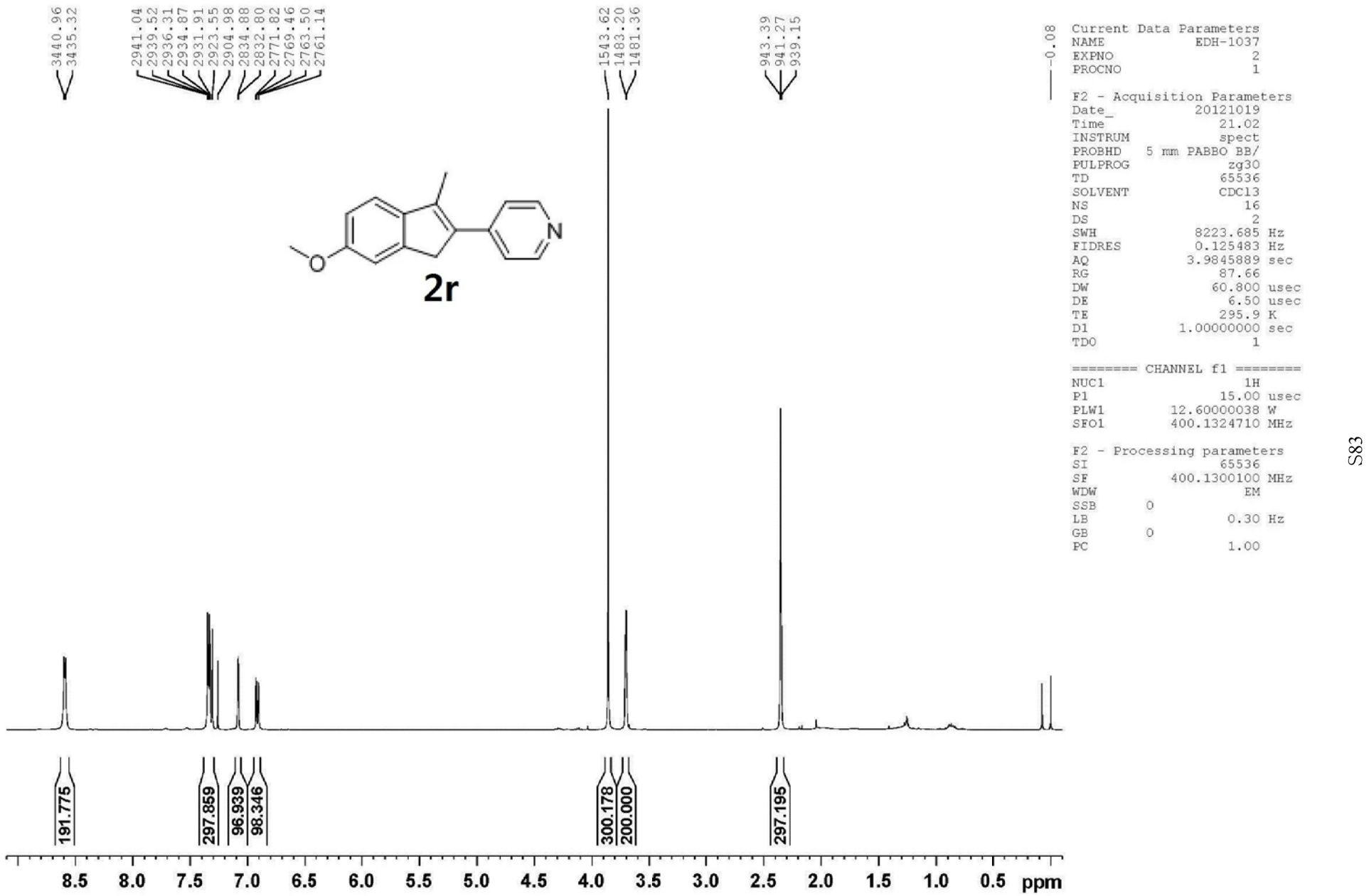



```
Current Data Parameters
NAME                  PSJ-775
EXPNO                       4
PROCNO                      1

F2 - Acquisition Parameters
Date_              20121020
Time                    8.48
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       9216
DS                          4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631488 sec
RG                     186.53
DW                     20.800 usec
DE                      6.50 usec
TE                     297.3 K
D1                 2.00000000 sec
d11                0.03000000 sec
DELTA              1.89999998 sec
TD0                         1
SFO1              100.6228293 MHz
NUC1                      13C
P1                      9.98 usec
PLW1              52.09999847 W
SFO2              400.1316005 MHz
NUC2                      1H
CPDPRG[2             waltz16
PCPD2                  90.00 usec
PLW2              12.60000038 W
PLW12              0.34999999 W
PLW13              0.28349999 W

F2 - Processing parameters
SI                      32768
SF                100.6127693 MHz
WDW                        EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```

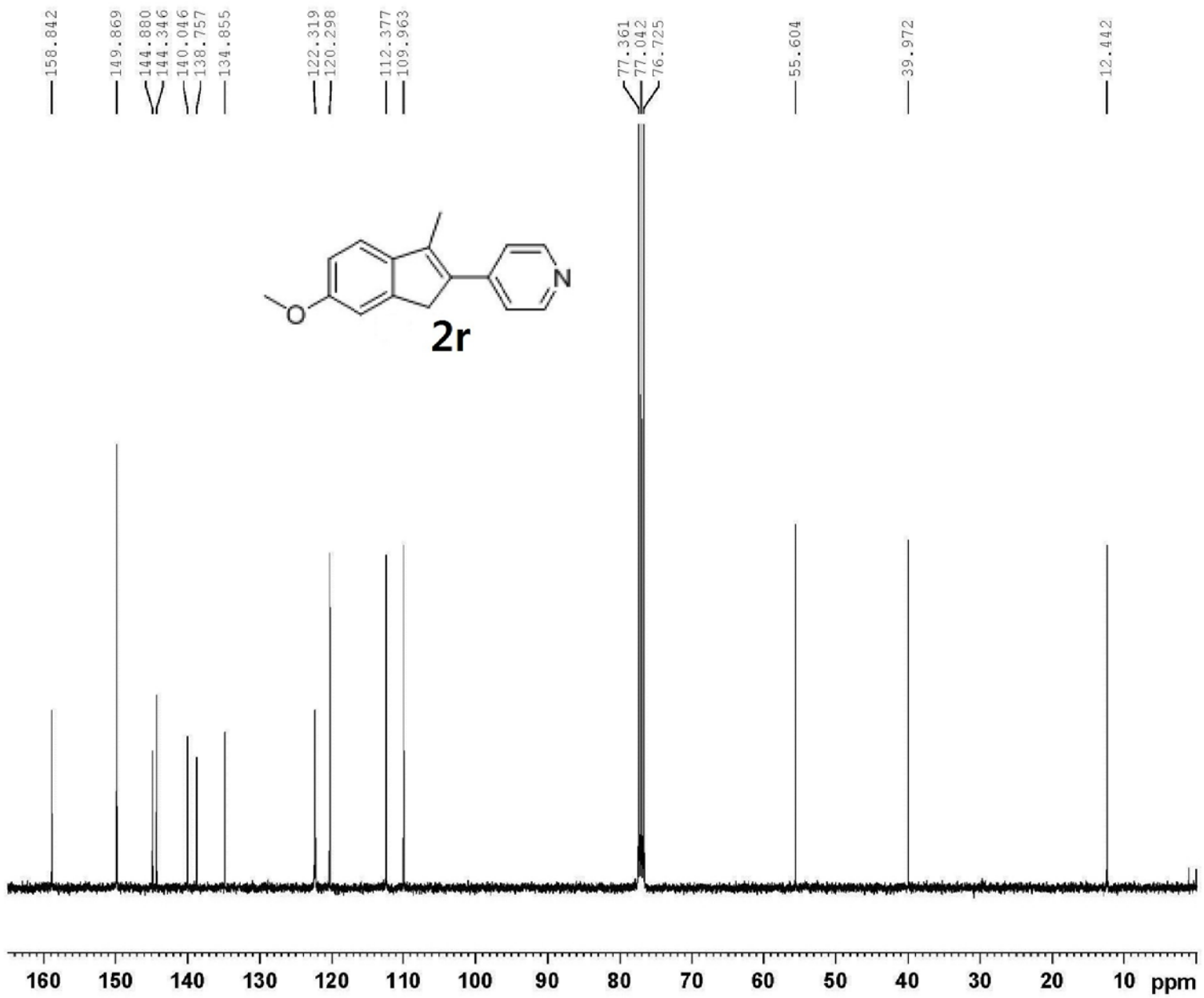

245.353
98.425
99.533
201.009
98.265
97.720
300.000
389.352
300.573
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2755.02
2752.81
2750.49
2747.37
2745.97
2744.93
2714.76
2712.80
2710.92
2649.34
2648.46
2647.02
2646.10
2641.42
2640.54
2639.10
2638.18
1540.82
1481.72
1468.32
1466.52
912.14
910.06
907.98
619.24
-0.08
O
NH2
2q
Current Data Parameters
NAME PSJ-771-3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121018
Time_ 10.23
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 167.01
DW 60.800 usec
DE 6.50 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.60000038 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300109 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



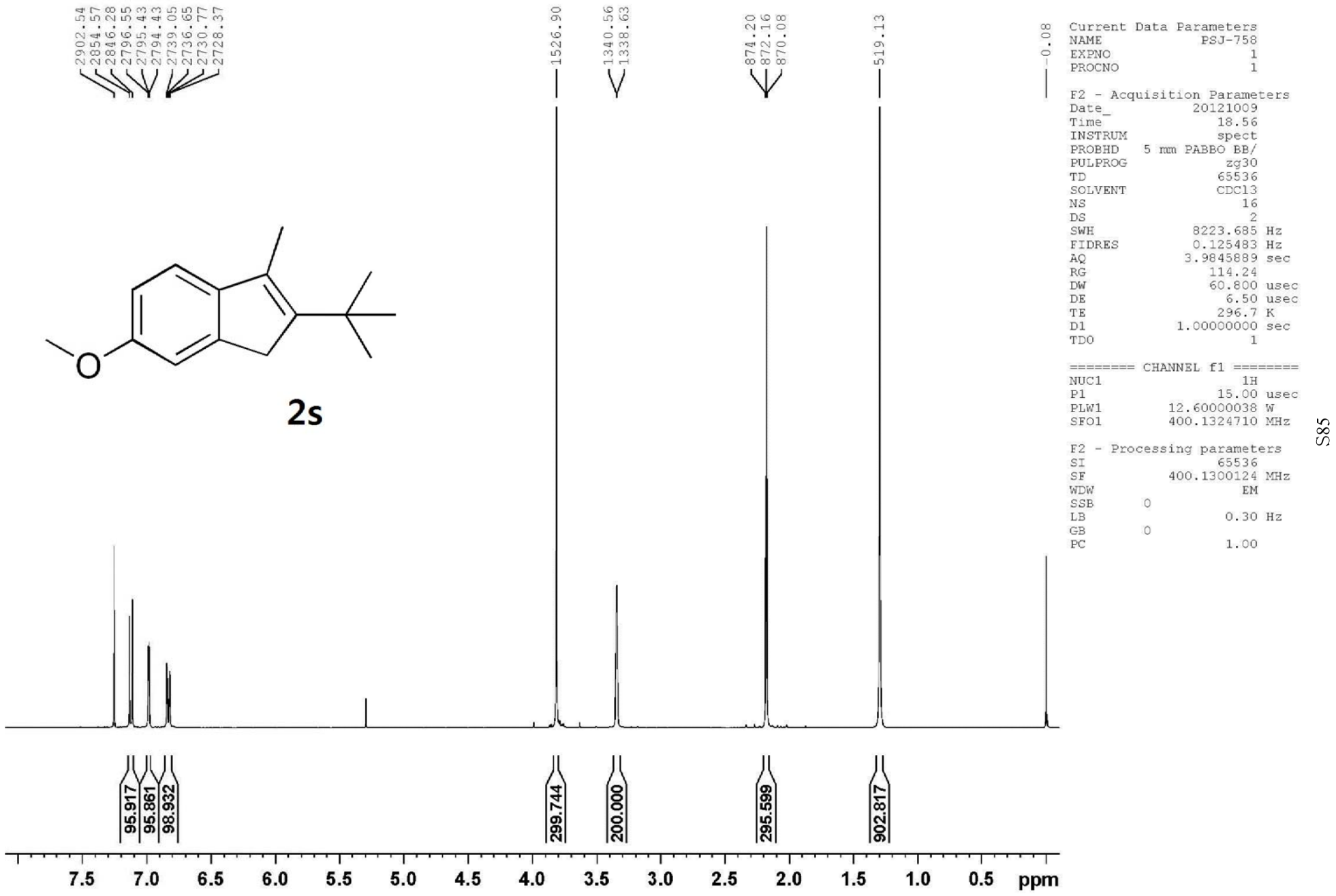

```
Current Data Parameters
NAME                  PSJ-771-3
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20121018
Time                      12.27
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        2048
DS                           4
SWH                  24038.461 Hz
FIDRES                0.366798 Hz
AQ                    1.3631488 sec
RG                       186.53
DW                       20.800 usec
DE                        6.50 usec
TE                       297.6 K
D1                    2.00000000 sec
d11                   0.03000000 sec
DELTA                 1.89999998 sec
TD0                           1
SFO1                100.6228293 MHz
NUC1                        13C
P1                        9.98 usec
PLW1                52.09999847 W
SFO2                400.1316005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2                12.60000038 W
PLW12                0.34999999 W
PLW13                0.28349999 W

F2 - Processing parameters
SI                        32768
SF                  100.6127690 MHz
WDW                          EM
SSB                   0
LB                        1.00 Hz
GB                    0
PC                        1.40
```

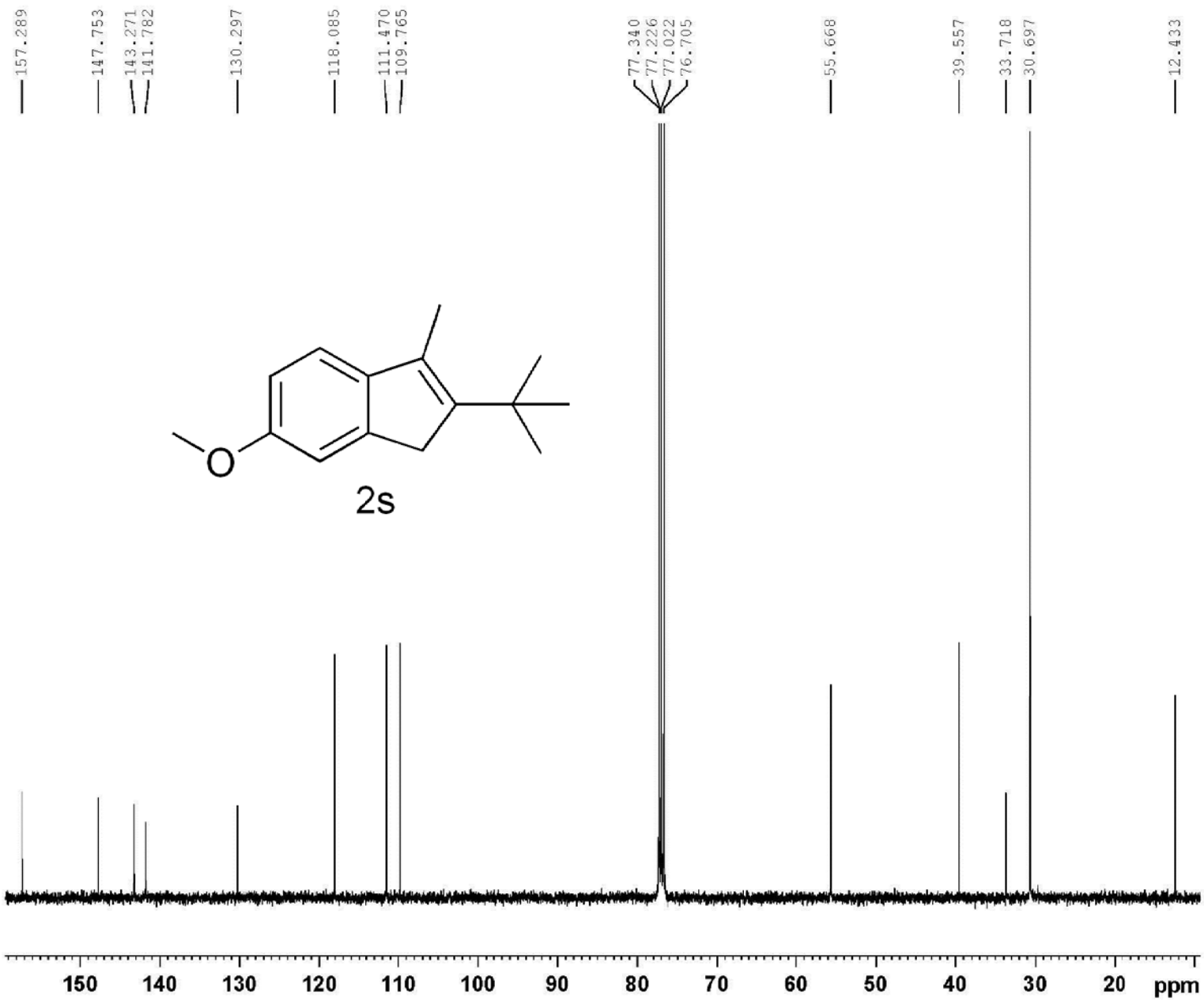



```
Current Data Parameters
NAME                  EDH-1037
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20121019
Time                     21.02
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                  8223.685 Hz
FIDRES               0.125483 Hz
AQ                   3.9845889 sec
RG                       87.66
DW                      60.800 usec
DE                       6.50 usec
TE                      295.9 K
D1                   1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                       15.00 usec
PLW1                12.60000038 W
SFO1               400.1324710 MHz

F2 - Processing parameters
SI                      65536
SF                 400.1300100 MHz
WDW                        EM
SSB                 0
LB                       0.30 Hz
GB                  0
PC                       1.00
```




```
Current Data Parameters
NAME                  EDH-1037
EXPNO                        3
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20121019
Time                     23.27
INSTRUM                  spect
PROBHD     5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                       1024
DS                          4
SWH                 24038.461 Hz
FIDRES               0.366798 Hz
AQ                   1.3631488 sec
RG                      186.53
DW                      20.800 usec
DE                       6.50 usec
TE                      297.2 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
TD0                         1
SFO1             100.6228293 MHz
NUC1                       13C
P1                       9.98 usec
PLW1             52.09999847 W
SFO2             400.1316005 MHz
NUC2                       1H
CPDPRG[2              waltz16
PCPD2                   90.00 usec
PLW2             12.60000038 W
PLW12             0.34999999 W
PLW13             0.28349999 W

F2 - Processing parameters
SI                       32768
SF               100.6127690 MHz
WDW                         EM
SSB                  0
LB                       1.00 Hz
GB                   0
PC                       1.40
```


2902.54
2854.57
2846.28
2796.55
2795.43
2794.43
2739.05
2736.65
2730.77
2728.37
1526.90
1340.56
1338.63
874.20
872.16
870.08
519.13
0.08
O
2s
Current Data Parameters
NAME PSJ-758
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121009
Time 18.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9845889 sec
RG 114.24
DW 60.800 usec
DE 6.50 usec
TE 296.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PLW1 12.60000038 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300124 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
95.917
95.861
98.932
299.744
200.000
295.599
902.817
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




```
Current Data Parameters
NAME                  PSJ-758
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20121010
Time                   0.01
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1024
DS                       4
SWH              24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631488 sec
RG                  186.53
DW                  20.800 usec
DE                   6.50 usec
TE                  296.6 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                      1
SFO1           100.6228293 MHz
NUC1                    13C
P1                    9.98 usec
PLW1           52.09999847 W
SFO2           400.1316005 MHz
NUC2                    1H
CPDPRG[2           waltz16
PCPD2                90.00 usec
PLW2           12.60000038 W
PLW12           0.34999999 W
PLW13           0.28349999 W

F2 - Processing parameters
SI                   32768
SF             100.6127690 MHz
WDW                     EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```