Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

Fe-Catalyzed Synthesis of Substituted N-Aryl Oxazolidines

*Siva Murru, Charles Seth Lott, Brandon McGough, Dakota M. Bernard and Radhey S. Srivastava**

Department of Chemistry, University of Louisiana at Lafayette, LA 70504.

rss1805@louisiana.edu

List of Contents

General information:

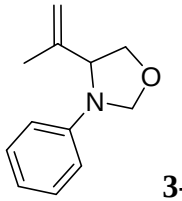
All the reagents were commercial grade and purified according to the established procedures. Organic extracts were dried over anhydrous magnesium sulphate. Solvents were removed in a rotary evaporator under reduced pressure. Silica gel (60-120 mesh size) was used for the column chromatography. Reactions were monitored by TLC on silica gel 60 F_{254} (0.25mm). NMR spectra were recorded in $CDCl_3$ with tetramethylsilane (TMS) as the internal standard for ^{1}H NMR (400 MHz) and for ^{13}C NMR (100 MHz). The

NMR data was collected on Varian 400 MHz and Bruker 400 MHz AVIII HD spectrometers. IR spectra were recorded on NICOLET IS-10 FT-IR spectrometer. GC-MS analysis carried out on an Agilent GC-MS (7890A – 5975C VL MSD) system.

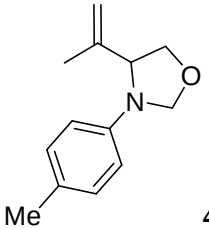
General Procedure for the Preparation of 4-Propenyl-3-Aryl Oxazolidines: To the solution of anh. $FeCl_2$ (0.05 mmol), allyl alcohol (1.5 mmol) and para-formaldehyde (0.6 mmol) in anh. THF (5 mL), the aryl hydroxylamine (0.5 mmol) solution was added slowly in THF (5 mL) using a syringe pump over 4 hours at 60 ^{o}C while keeping the flask under inert atmosphere using nitrogen balloon. Reactions were allowed to continue for two more hours for the complete consumption of aryl hydroxylamine. Then the mixture was filtered through celite and the filtrate was concentrated to dryness. The crude product was purified over a short column of silica gel (hexane/ethyl acetate eluents) to obtain the pure oxazolidine which was then directly analyzed by GC-MS, ESI-MS, NMR and FT-IR.

General Procedure for the Synthesis of Allyl Amino Alcohols: To the propenyl oxazolidine (obtained from the previous step) in a RB flask, 2 mL of conc. HCl was added and stirred for 2 hours. Once the reaction is completed (monitored by TLC and GC-MS), the mixture was filtered through celite and the filtrate was concentrated to dryness. The crude product was purified over a short column of silica gel (hexane/ethyl acetate eluents) and the isolated pure N-aryl amino alcohol which was then directly analyzed by GC-MS, ESI-MS, NMR and FT-IR.

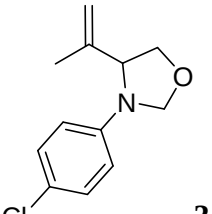
Spectral data



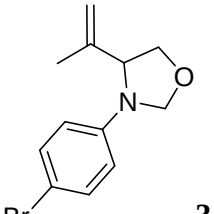
3-phenyl-4-(prop-1-en-2-yl)oxazolidine (1a): ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.72 (s, 3H), 3.91 (dd, 1H, J^1 = 8.0Hz, J^2 = 3.6Hz), 4.07-4.10 (m, 1H), 4.16 (t, 1H, *J* =7.6 Hz), 4.86 (d, 1H, *J* = 2.4 Hz), 4.92 (s, 1H), 5.03 (s, 1H), 5.09 (d, 1H, *J* = 2.0 Hz), 6.51 (d, 2H, *J* = 8.0 Hz), 6.74 (t, 1H, *J* = 7.6 Hz), 7.20 (t, 2H, *J* = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 18.4, 63.3, 72.8, 82.4, 112.5, 112.6, 117.6, 125.7, 129.4, 144.5; GC-MS : 189.0 (M^+); IR (KBr): 2918, 2847, 1621, 1542, 1162, 1092, 811 cm^{-1}.



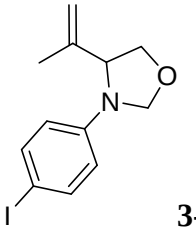
4-(prop-1-en-2-yl)-3-p-tolyloxazolidine (1b): ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.73 (s, 3H), 2.43 (s, 3H), 3.90 (dd, 1H, J^1= 8.4Hz, J^2= 4.0 Hz), 4.04-4.07 (m, 1H), 4.17 (t, 1H, *J* = 8.0 Hz), 4.84 (d, 1H, *J* = 2.0 Hz), 4.93 (s, 1H), 5.04 (s, 1H), 5.09 (d, 1H, *J* = 2.4 Hz), 6.46 (d, 2H, *J* = 8.4 Hz), 7.02 (d, 2H, *J* = 8.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 18.4, 20.5, 63.5, 72.7, 82.7, 112.4, 112.6, 122.3, 125.9, 129.9, 144.6; GC-MS : 203.0 (M^+); IR (KBr): 2920, 2855, 1620, 1521, 1499, 1164, 1095, 947, 899, 801 cm^{-1}.



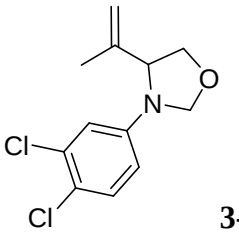
3-(4-chlorophenyl)-4-(prop-1-en-2-yl)oxazolidine (1c): ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.72 (s, 3H), 3.94 (dd, 1H, J^1= 8.4 Hz, J^2 = 4.4 Hz), 4.06-4.09 (m, 1H), 4.21 (t, 1H, *J* = 7.2 Hz), 4.85 (d, 1H, *J* = 2.0 Hz), 4.95 (brs, 1H), 5.03 (brs, 1H), 5.08 (brs, 1H), 6.44 (d, 2H, *J* = 6.8 Hz), 7.15 (d, 2H, *J* = 6.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 18.3, 63.3, 72.8, 82.4, 112.9, 113.5, 122.5, 129.2, 143.8, 144.0; HRMS (ESI) calcd for C12H15ClNO ($M + H^+$) 224.0837, found 224.0844; IR (KBr): 2881, 2826, 1599, 1498, 1488, 1389, 1368, 1174, 1090, 942, 900, 809 cm^{-1}.



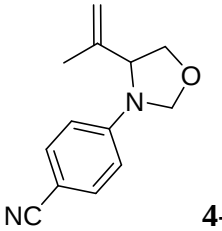
3-(4-brorophenyl)-4-(prop-1-en-2-yl)oxazolidine (1d):^{1}H NMR (400 MHz, $CDCl_3$): δ 1.69 (s, 3H), 3.90 (dd, 1H, J^1= 8.4 Hz, J^2= 4.0 Hz), 4.03-4.06 (m, 1H), 4.82 (d, 1H, *J* = 2.0 Hz), 4.93 (s, 1H), 5.00 (s, 1H), 5.03 (d, 1H, *J* = 2.0 Hz), 6.37 (d, 2H, *J* = 9.2 Hz), 7.26 (d, 2H, *J* = 8.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 18.3, 63.2, 72.8, 82.2, 109.6, 112.9, 114.0, 132.1, 143.9, 144.2; HRMS (ESI) calcd for C12 H15 Br N O ($M + H^+$) 268.0332; found, 268.0331; IR (KBr): 2972, 2914, 2859, 1593, 1490, 1358, 1146, 1094, 899, 807 cm^{-1}.



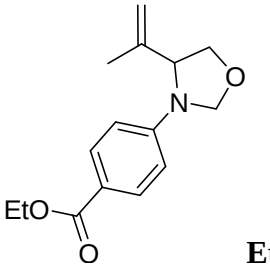
3-(4-iodophenyl)-4-(prop-1-en-2-yl)oxazolidine (1e): ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.69 (s, 3H), 3.91 (dd, 1H, J^1 = 8.4 Hz, J^2 = 4.0 Hz), 4.03-4.06 (m, 1H), 4.17 (t, 1H, J = 7.6 Hz), 4.81 (s, 1H), 4.93 (s, 1H), 4.99 (s, 1H), 5.03 (s, 1H), 6.27 (d, 2H, J = 8.4 Hz), 7.44 (d, 2H, J = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 18.3, 29.9, 63.1, 72.8, 82.1, 112.9, 114.7, 124.2, 137.9, 143.8; GC-MS : 314.9 (M^+); IR (KBr): 2989, 2889, 1595, 1488, 1352, 1134, 1096, 1004, 949, 893, 805 cm^{-1}.



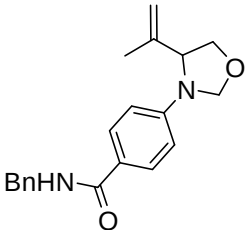
3-(3,4-dichlorophenyl)-4-(prop-1-en-2-yl)oxazolidine (1f): ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.70 (s, 3H), 3.94 (dd, 1H, J^1 = 8.4 Hz, J^2 = 4.0 Hz), 4.05-4.08 (m, 1H), 4.20 (t, 1H, J = 8.0 Hz), 4.83 (d, 1H, J = 2.4 Hz), 4.97 (s, 1H), 5.02 (s, 1H), 5.04 (d, 1H, J = 2.4 Hz), 6.33 (dd, 1H, J^1 = 8.8 Hz, J^2 = 2.8 Hz), 6.57 (d, 1H, J = 2.8 Hz), 7.22 (d, 1H, J = 8.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 18.3, 63.2, 72.8, 82.1, 112.0, 113.3, 113.8, 120.4, 130.8, 133.1, 143.4, 144.5; HRMS (ESI) calcd for C12 H14 Cl2 N O ($M + H^+$) 258.0447; found, 258.0445; IR (KBr): 2970, 2917, 2853, 1597, 1483, 1360, 1019, 795 cm^{-1}.



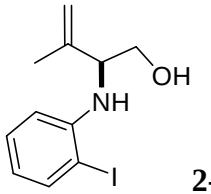
4-(4-(prop-1-en-2-yl)oxazolidin-3-yl)benzonitrile (1g): ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.72 (s, 3H), 3.98 (dd, 1H, J^1 = 8.0Hz, J^2 = 3.2Hz), 4.18-4.23 (m, 2H), 4.92 (d, 1H, *J* = 2.0 Hz), 4.99 (s, 2H), 5.09 (d, 1H, *J* = 2.0 Hz), 6.48 (d, 2H, *J* = 8.4 Hz), 7.46 (d, 2H, *J* = 8.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 18.3, 62.8, 72.8, 81.5, 105.2, 112.3, 113.6, 120.4, 133.8, 142.9, 147.5; HRMS (ESI) calcd for C13 H15 N2 O ($M + H^+$) 215.1179; found, 215.1178; IR (KBr): 2915, 2881, 2848, 2213, 1651, 1521, 1497, 1389, 1371, 1174, 1092, 1062, 942, 900, 810 cm^{-1}.



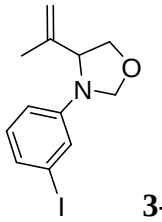
Ethyl 4-(4-(prop-1-en-2-yl)oxazolidin-3-yl)benzoate (1h): ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.33 (t, 3H, *J* = 7.2 Hz), 1.70 (s, 3H), 4.18 (brs, 2H), 4.30 (q, 2H, *J* = 7.2 Hz), 4.93 (d, 2H, *J* = 8.4 Hz), 4.98 (s, 1H), 5.09 (s, 1H), 6.45 (d, 2H, *J* = 8.0 Hz), 7.88 (d, 2H, *J* = 8.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.6, 18.4, 60.5, 62.8, 72.8, 81.7, 111.5, 113.2, 131.4, 131.5, 143.4, 148.1, 167.0; HRMS (ESI) calcd for C15 H20 N O3 ($M + H^+$) 262.1438; found, 262.1440; IR (KBr): 2978, 2927, 2871, 1699, 1604, 1522, 1364, 1270, 1178, 1101, 767 cm^{-1}.



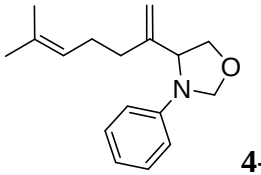
N-benzyl-4-(4-(prop-1-en-2-yl)oxazolidin-3-yl)benzamide (1i): ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.23 (t, 1H, *J* = 6.8 Hz), 1.70 (s, 3H), 3.95 (d, 1H, *J* = 8.0 Hz), 4.14-4.20 (m, 2H), 4.60 (d, 2H, *J* = 5.6 Hz), 4.90 (s, 1H), 4.95 (s, 1H), 4.99 (s, 1H), 5.08 (s, 1H), 6.42 (brs, 1H), 6.47 (d, 2H, *J* = 8.8 Hz), 7.26 (d, 1H, *J* = 5.2 Hz), 7.31 (d, 3H, *J* = 4.0 Hz), 7.68 (d, 2H, *J* = 8.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 18.3, 44.1, 62.8, 72.7, 81.8, 111.6, 113.1, 122.7, 127.6, 128.0, 128.7, 128.8, 138.9, 143.5, 147.3, 167.3; HRMS (ESI) calcd for C20 H23 N2 O2 ($M + H^+$) 323.1754; found, 323.1763; IR (KBr): 3317, 2912, 2868, 1612, 1518, 1359, 1303, 1201, 1169, 945, 828, 764, 729 cm^{-1}.



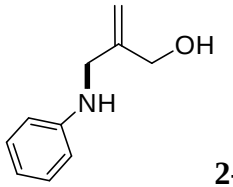
2-(2-iodophenylamino)-3-methylbut-3-en-1-ol (1k): ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.77 (s, 3H), 1.94 (brs, 1H), 3.72-3.76 (m, 1H), 3.81 (brs, 1H), 3.91-3.94 (m, 1H), 4.69 (brs, 1H), 5.01 (s, 2H), 6.42-6.49 (m, 2H), 7.15 (t, 1H, *J* = 7.6 Hz), 7.65 (d, 1H, *J* = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 19.6, 61.3, 63.9, 86.1, 112.1, 113.9, 119.3, 129.4, 139.2, 142.2, 146.5; HRMS (ESI) calcd for C11 H15 I N O ($M + H^+$) 304.0193; found, 304.0201; IR (KBr): 3376, 2923, 2856, 1587, 1501, 1451, 1313, 1008, 899, 748 cm^{-1}.



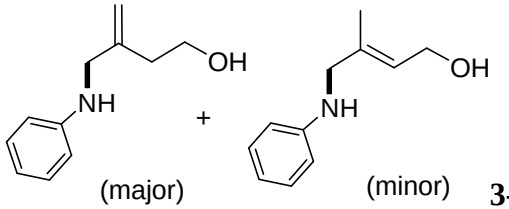
3-(3-iodophenyl)-4-(prop-1-en-2-yl)oxazolidine (1l): ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.71 (s, 3H), 3.91-3.94 (m, 1H), 4.05-4.08 (m, 1H), 4.17 (t, 1H, *J* = 7.6 Hz), 4.84 (s, 1H), 4.95 (s, 1H), 5.03 (d, 2H, *J* = 8.0 Hz), 6.47 (d, 1H, *J* = 8.8 Hz), 6.82 (s, 1H), 6.90 (t, 1H, *J* = 8.0 Hz), 7.06 (d, 1H, *J* = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 18.3, 63.0, 72.7, 82.0, 95.5, 111.7, 113.0, 121.1, 126.5, 130.8, 139.2, 143.7, 146.3; HRMS (ESI) calcd for C12 H15 I N O ($M + H^+$) 316.0193; found, 316.0190; IR (KBr): 3071, 2922, 2865, 1591, 1551, 1479, 1388, 1353, 1170, 1093, 980, 947, 758 cm^{-1}.



4-(6-methylhepta-1,5-dien-2-yl)-3-phenyloxazolidine (2a): ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.60 (s, 3H), 1.68 (s, 3H), 2.07 (t, 2H, *J* = 7.6 Hz), 2.18 (t, 2H, *J* = 7.6 Hz), 3.90-3.93 (m, 1H), 4.09-4.11 (m, 1H), 4.17 (t, 1H, *J* = 7.2 Hz), 4.87 (s, 1H), 4.94 (s, 1H), 5.10 (d, 2H, *J* = 8.0 Hz), 6.50 (d, 2H, *J* = 8.0 Hz), 6.74 (t, 1H, *J* = 7.6 Hz), 7.22 (t, 2H, *J* = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 17.9, 25.9, 26.4, 32.0, 63.1, 73.1, 82.4, 111.0, 112.6, 117.5, 124.1, 129.3, 132.2, 145.4, 147.9; HRMS (ESI) calcd for C17 H24 N O ($M + H^+$) 258.1852; found, 258.1859; IR (KBr): 2966, 2912, 2857, 1604, 1510, 1391, 1345, 1170, 1095, 949, 752 cm^{-1}.

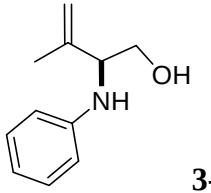


2-Phenylaminomethyl-prop-2-en-1-ol (3a): ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.33-1.36 (m, 1H), 1.61 (brs, 1H), 3.88 (s, 2H), 4.23 (s, 2H), 5.20 (d, 2H, *J* = 4.0 Hz), 6.67 (d, 2H, *J* = 8.0 Hz), 6.75 (t, 1H, *J* = 8.0 Hz), 7.20 (t, 2H, *J* = 8.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 47.0, 65.0, 112.3, 113.1, 117.8, 129.2, 145.9, 148.2; GC-MS : 163.0 (M^+); IR (KBr): 3392, 3051, 2924, 1607, 1507, 1315, 1261, 1070, 1048, 754 cm^{-1}.



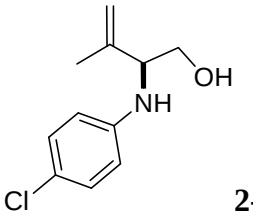
3-Phenylaminomethyl-but-3-en-1-ol and 3-Methyl-4-phenylamino-but-2-en-1-ol (4a+4a'):

Inseparable mixture of two isomers were obtained after column chromatography which was confirmed by NMR and GC-MS analysis as shown on pages: S49, S50 and S51. GC-MS : 177.0 (M^+).

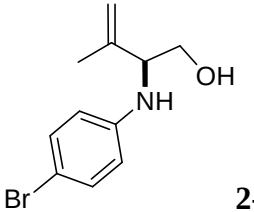


3-methyl-2-(phenylamino)but-3-en-1-ol (1a'): ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.76 (s, 3H), 1.92 (brs, 1H), 3.63-3.67 (m, 1H), 3.75-3.78 (m, 1H), 3.86-3.88 (m, 1H), 4.15 (brs, 1H), 5.00 (s, 1H), 5.03 (s, 1H), 6.59 (d, 2H, *J* = 7.6 Hz), 6.70 (t, 1H, *J* = 7.2 Hz), 7.15 (t, 2H, *J* = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 19.7, 60.9, 64.1, 113.5, 113.8, 117.9, 129.3, 142.9, 147.5;

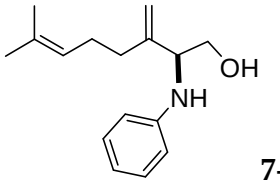
HRMS (ESI) calcd for C11 H16 N O ($M + H^+$) 178.1226; found, 178.1229; IR (KBr): 3392, 3051, 2924, 1607, 1507, 1315, 1261, 1070, 1048, 754 cm^{-1}.



2-(4-chlorophenylamino)-3-methylbut-3-en-1-ol (1c'): ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.63 (brs, 1H), 1.76 (s, 3H), 3.66-3.70 (m, 1H), 3.76-3.84 (m, 2H), 4.21 (brs, 1H), 5.02 (s, 2H), 6.51 (d, 2H, *J* = 7.6 Hz), 7.10 (d, 2H, *J* = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 19.7, 60.9, 63.9, 113.8, 114.8, 122.4, 129.2, 142.5, 146.0; GC-MS : 211.0 (M^+); IR (KBr): 3400, 2935, 1655, 1599, 1604, 1315, 1091, 1050, 902, 816 cm^{-1}.



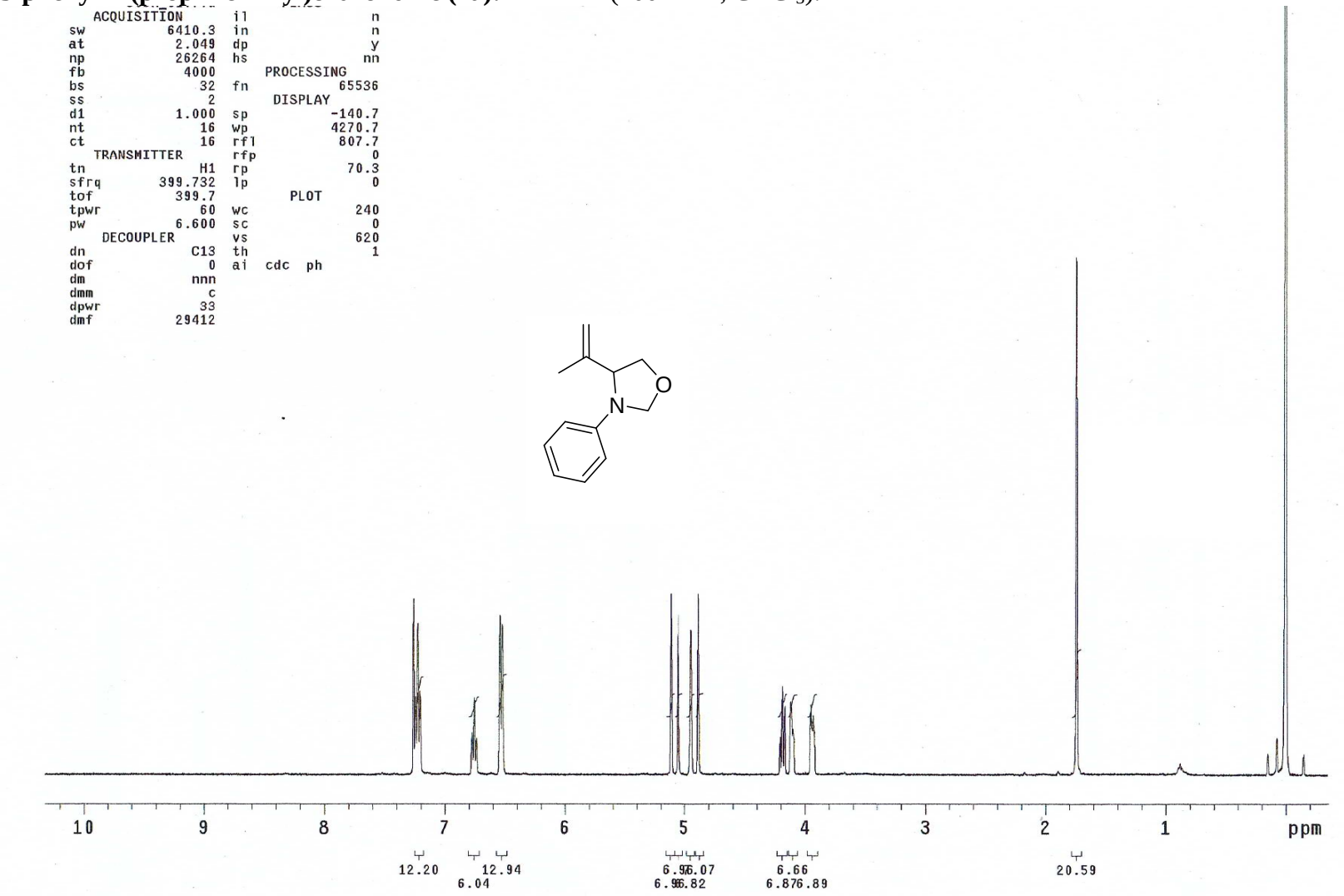
2-(4-bromophenylamino)-3-methylbut-3-en-1-ol (1d'): ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.63 (brs, 1H), 1.77 (s, 3H), 3.69-3.70 (m, 1H), 3.78-3.83 (m, 2H), 4.24 (brs, 1H), 5.02 (s, 2H), 6.47 (d, 2H, *J* = 7.6 Hz), 7.22 (d, 2H, *J* = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 19.7, 60.9, 63.9, 113.8, 115.3, 125.2, 132.1, 142.4, 146.5; GC-MS : 256.9 (M^+); IR (KBr): 3403, 2929, 1594, 1502, 1314, 1073, 902, 813 cm^{-1}.



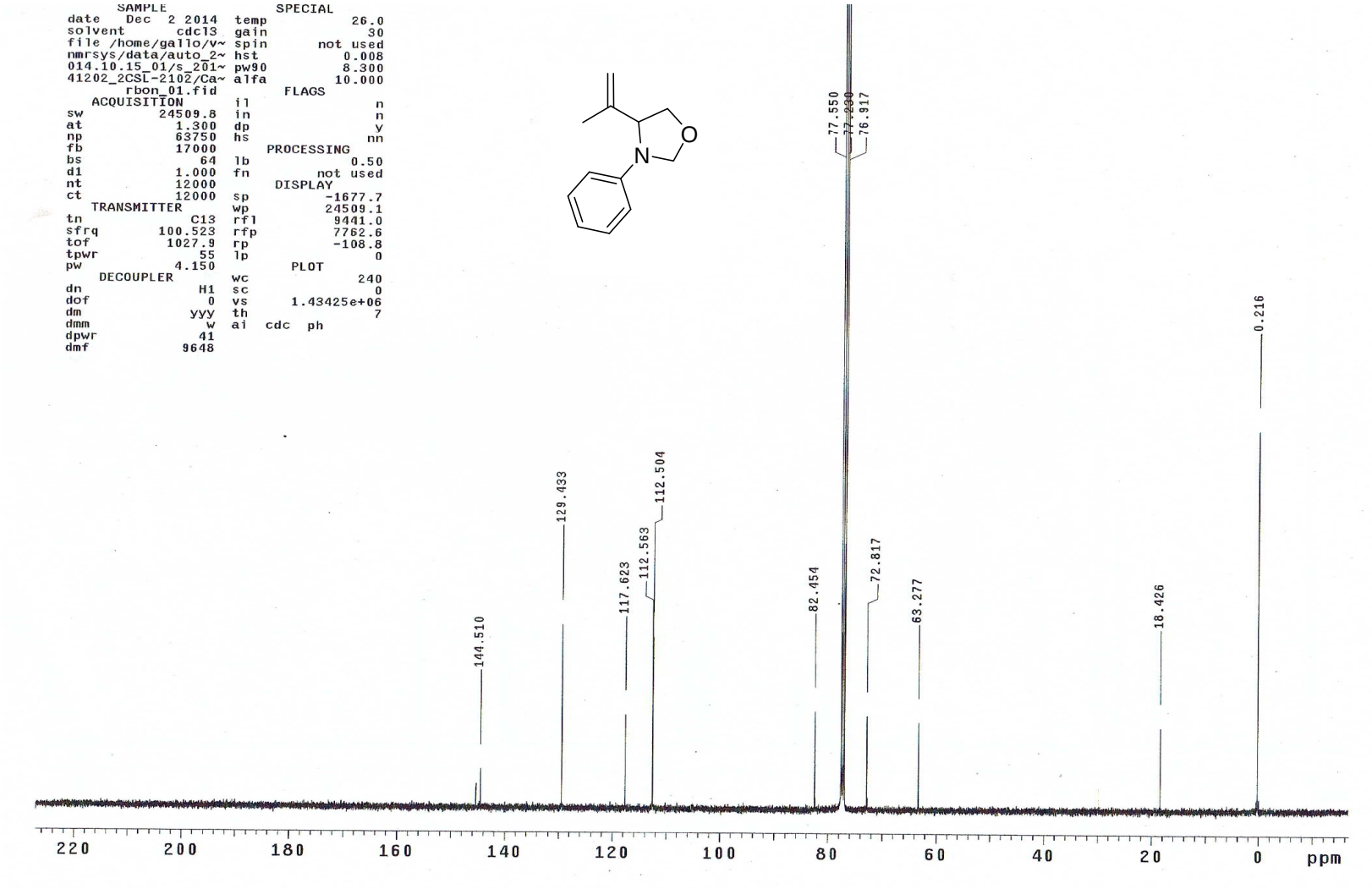
7-methyl-3-methylene-2-(phenylamino)oct-6-en-1-ol (2a'): ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.61 (brs, 1H), 1.67 (s, 3H), 1.69 (s, 3H), 1.74-1.77 (m, 2H), 2.08-2.14 (m, 2H), 3.72 (t, 2H, *J* = 6.4 Hz), 4.14 (d, 2H, *J* = 6.8 Hz), 4.91 (s, 1H), 4.97 (s, 1H), 5.42 (t, 1H, *J* = 6.8 Hz), 6.56 (d, 2H, *J* = 8.0 Hz), 6.66 (t, 1H, *J* = 7.2 Hz), 7.14 (t, 2H, *J* = 7.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 16.6, 18.0, 32,5, 36.3, 59.3, 59.5, 105.2, 112.5, 113.4, 117.3, 124.1, 129.3, 139.1, 145.7, 147.8; HRMS (ESI) calcd for C16 H24 N O ($M + H^+$) 246.1852; found, 246.1855; IR (KBr): 3360, 3052, 2922, 2859, 1607, 1509, 1317, 1258, 993, 896, 748 cm^{-1}.

SPECTRA

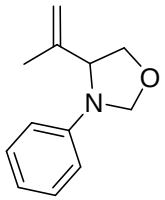
3-phenyl-4-(prop-1-en-2-yl)oxazolidine (1a): ^{1}H NMR (400 MHz, $CDCl_3$):

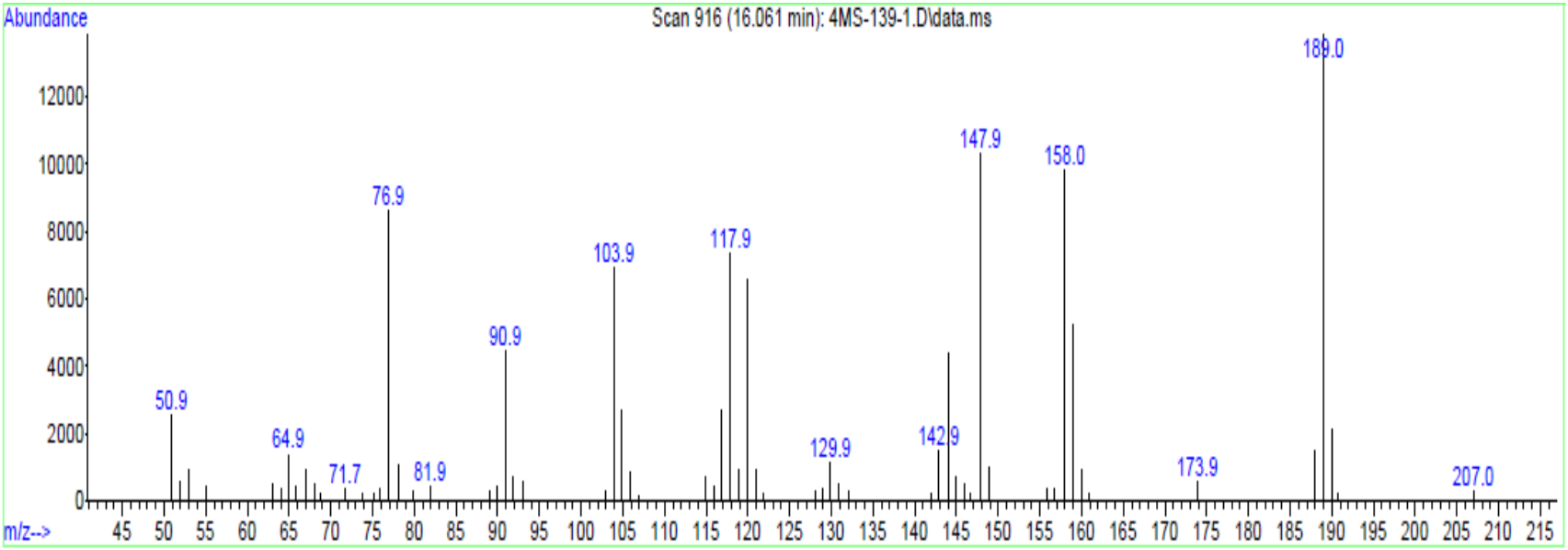


3-phenyl-4-(prop-1-en-2-yl)oxazolidine (1a): ^{13}C NMR (100 MHz, $CDCl_3$):

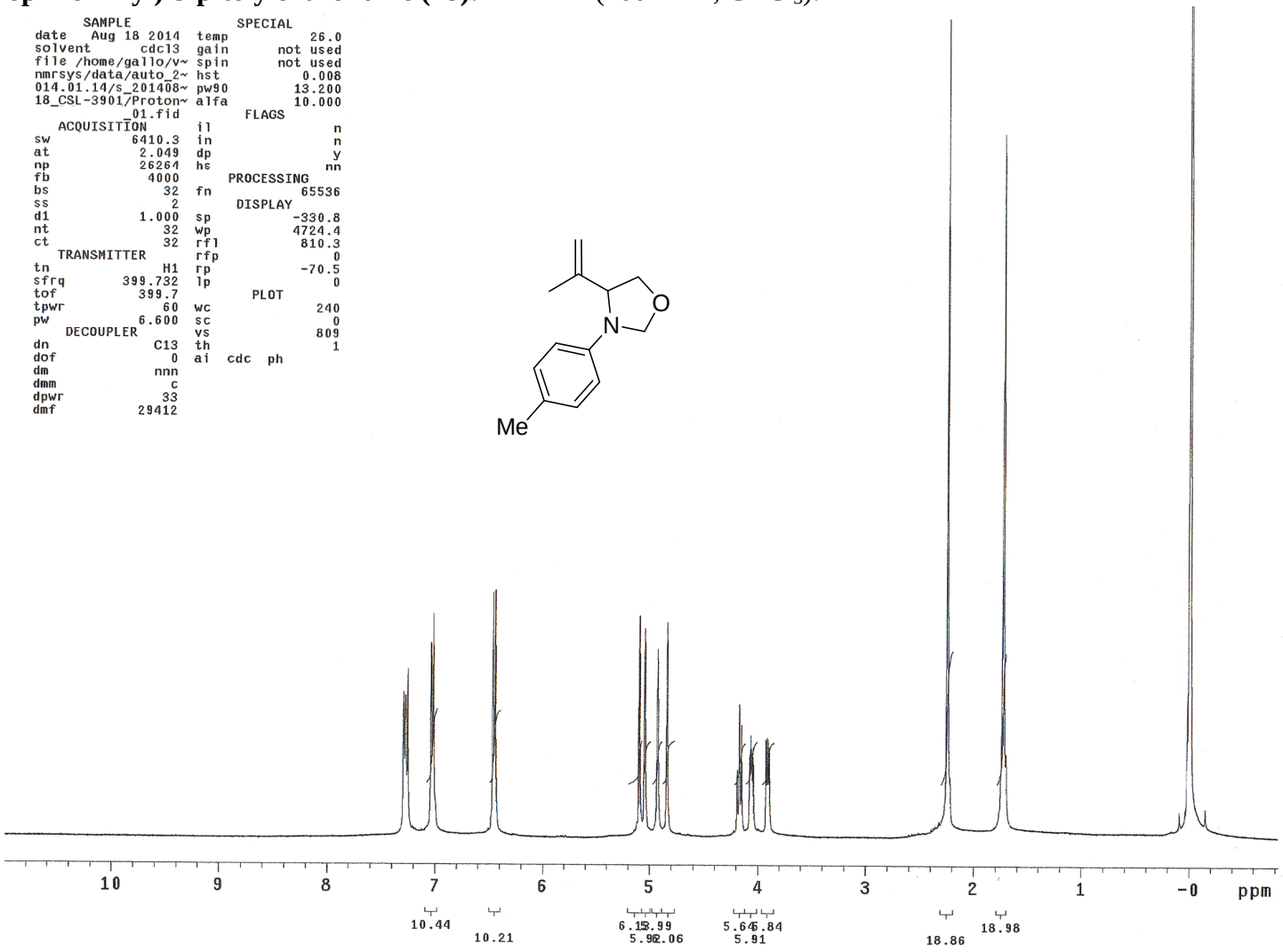


3-phenyl-4-(prop-1-en-2-yl)oxazolidine (1a): GC-MS analysis

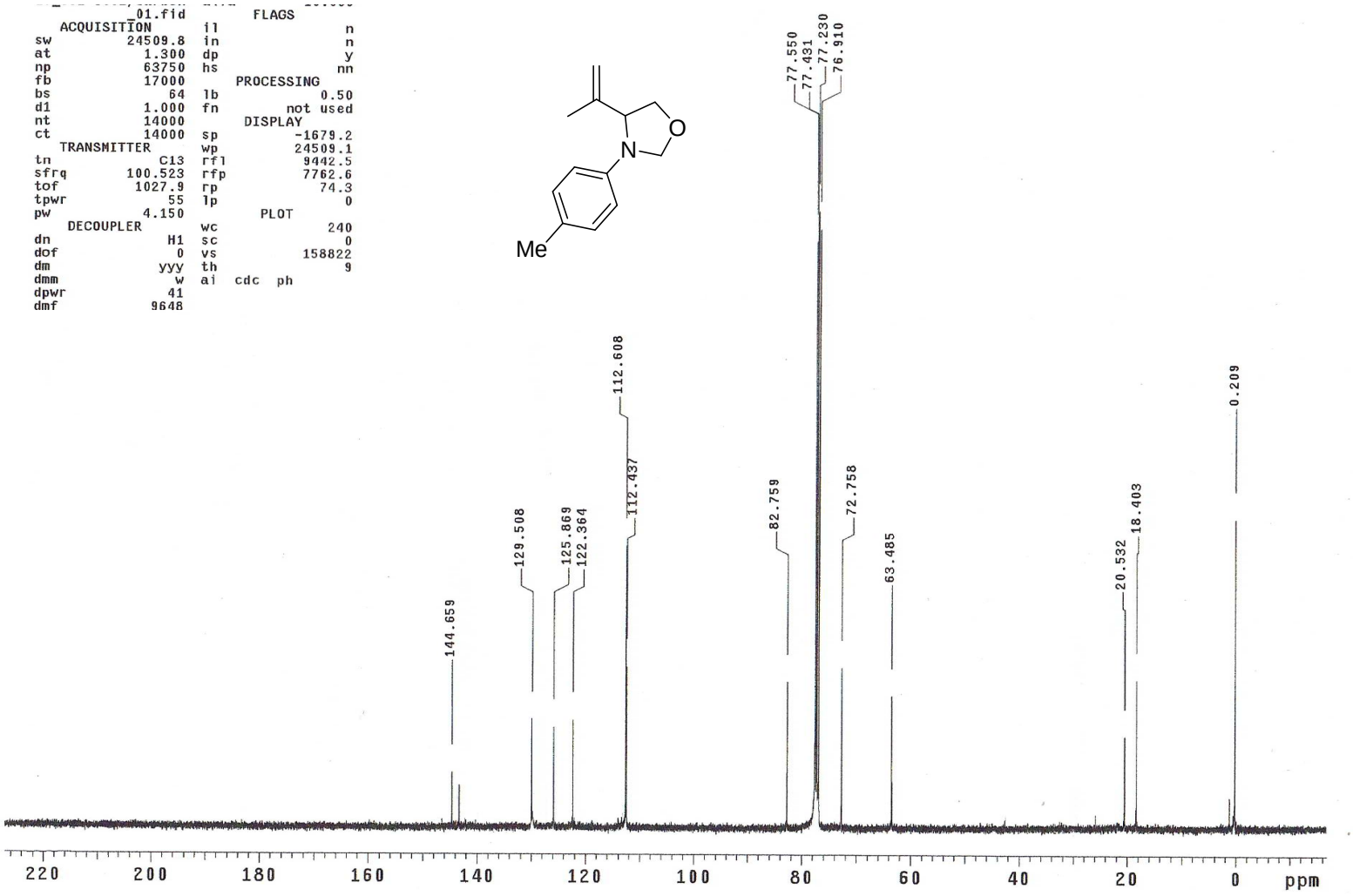




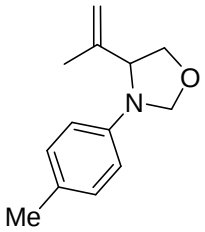
4-(prop-1-en-2-yl)-3-p-tolyloxazolidine (1b): ^{1}H NMR (400 MHz, $CDCl_3$):

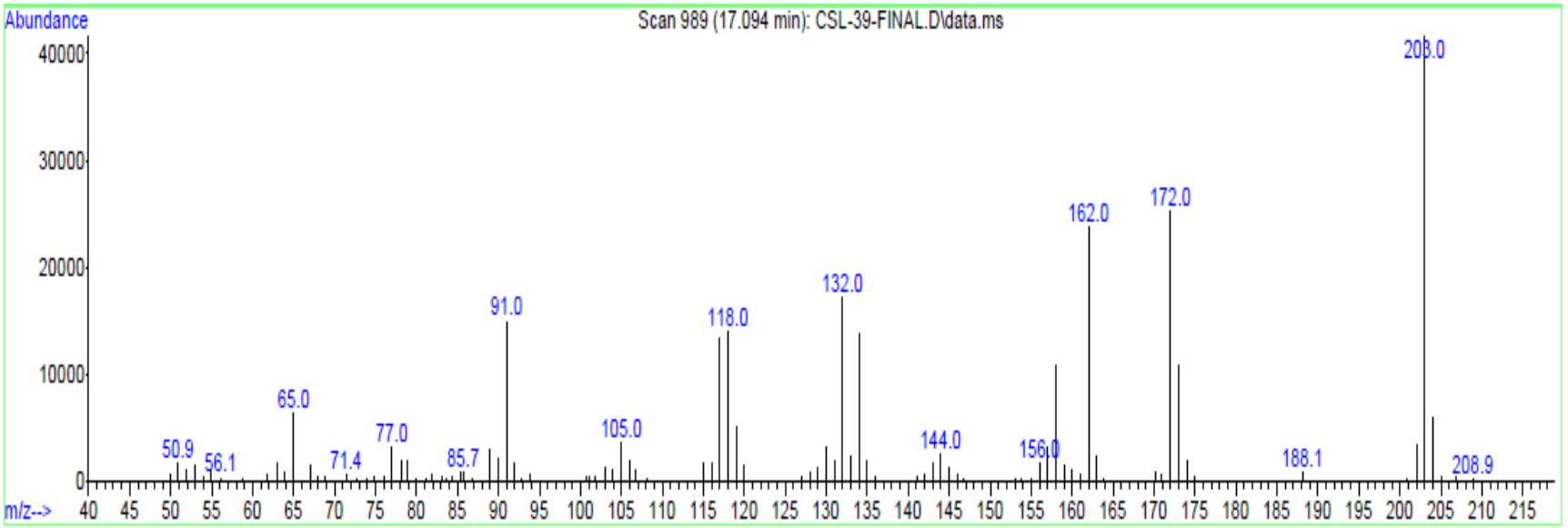


4-(prop-1-en-2-yl)-3-p-tolyloxazolidine (1b): ^{13}C NMR (100 MHz, $CDCl_3$):

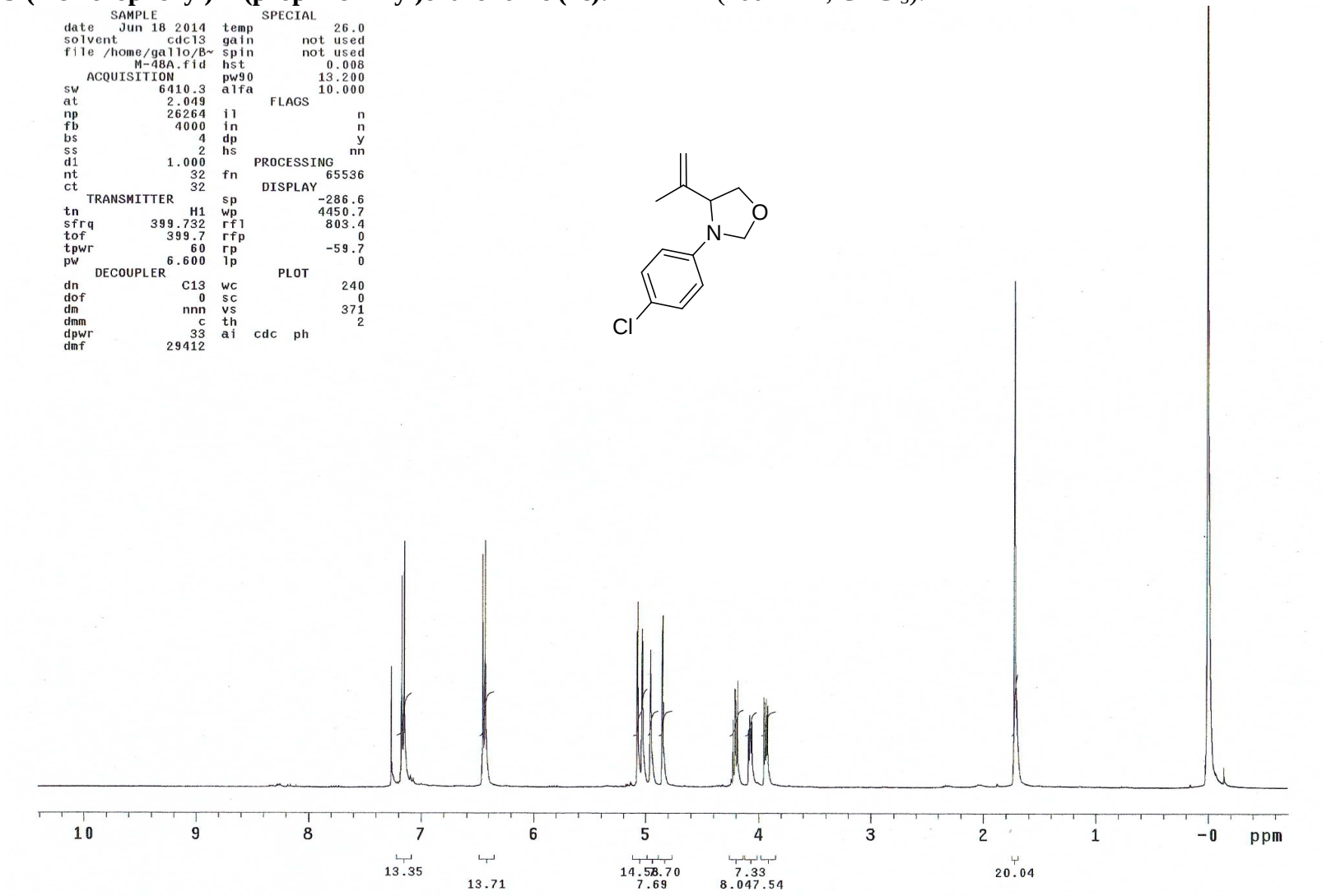


4-(prop-1-en-2-yl)-3-p-tolyloxazolidine (1b): GC-MS analysis

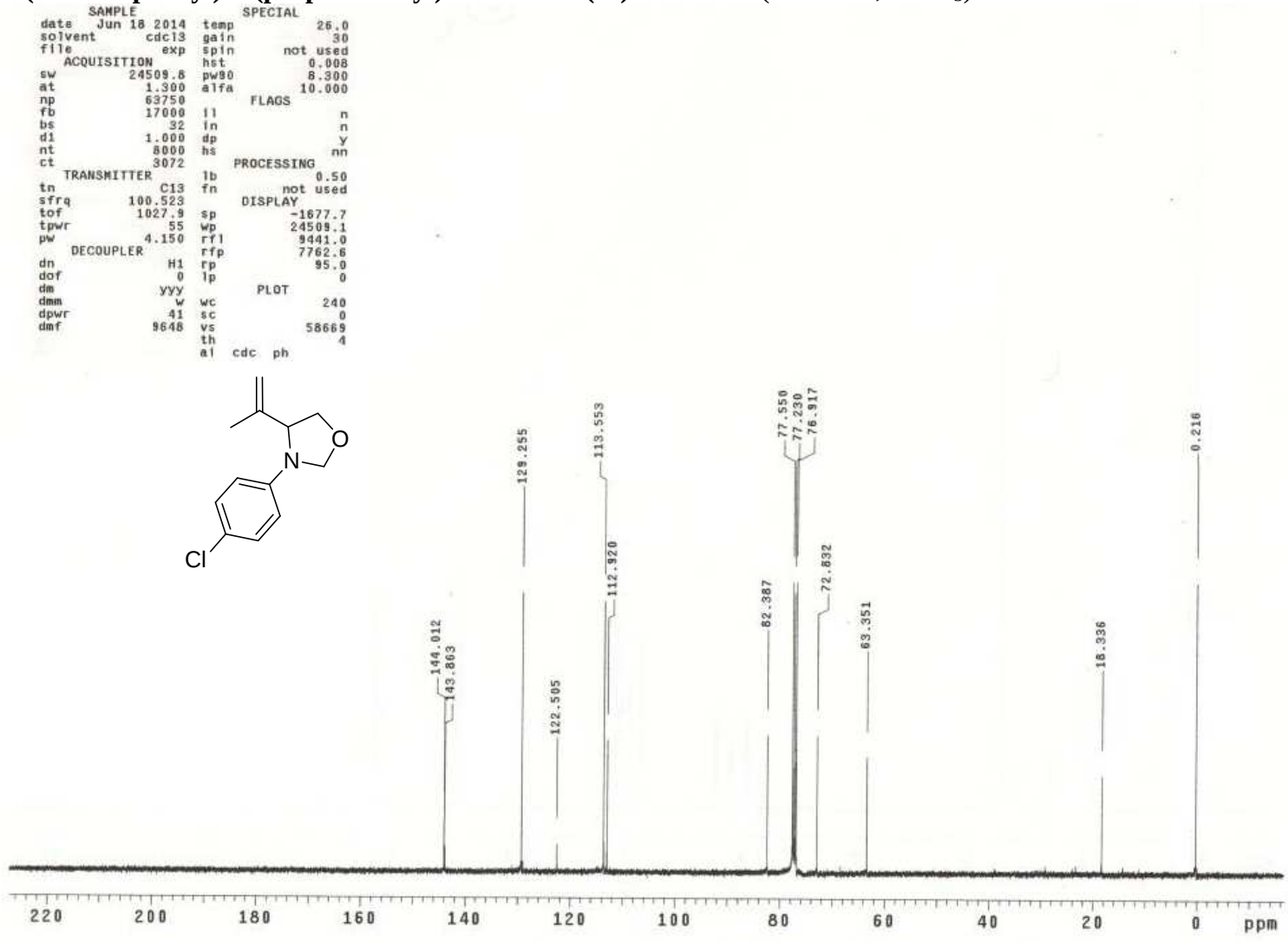




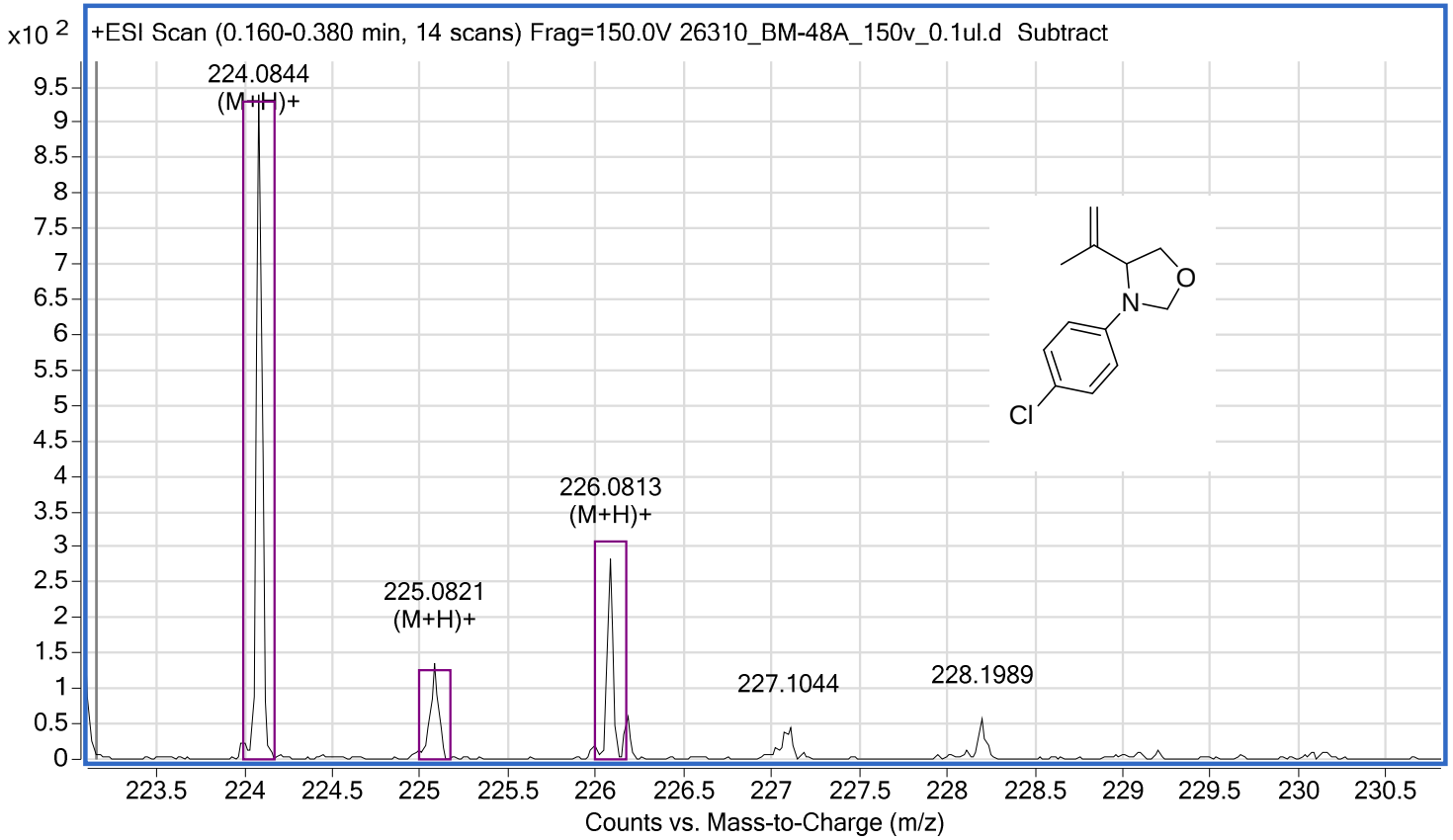
3-(4-chlorophenyl)-4-(prop-1-en-2-yl)oxazolidine (1c): ^{1}H NMR (400 MHz, $CDCl_3$):



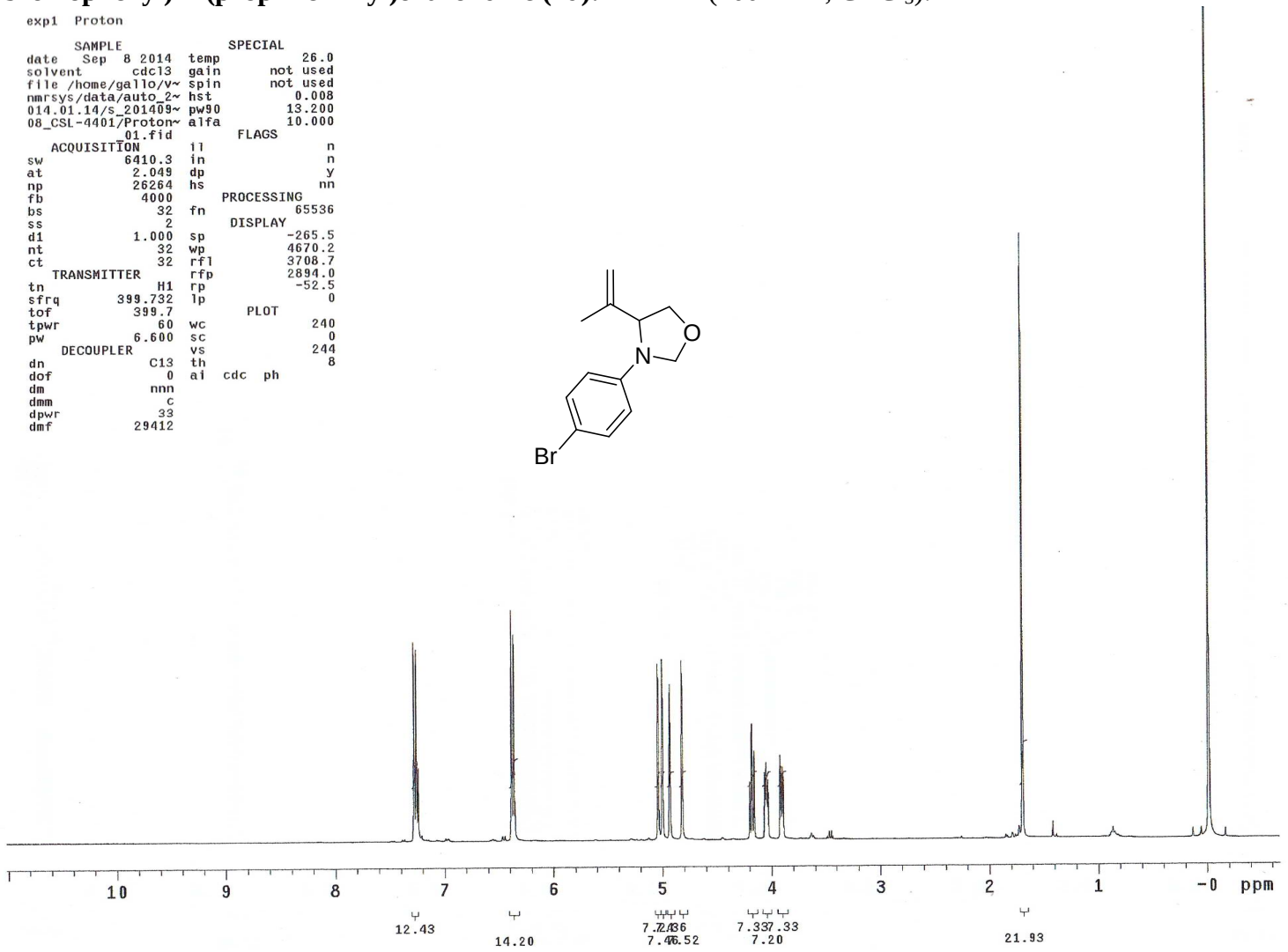
3-(4-chlorophenyl)-4-(prop-1-en-2-yl)oxazolidine (1c): ^{13}C NMR (100 MHz, $CDCl_3$):



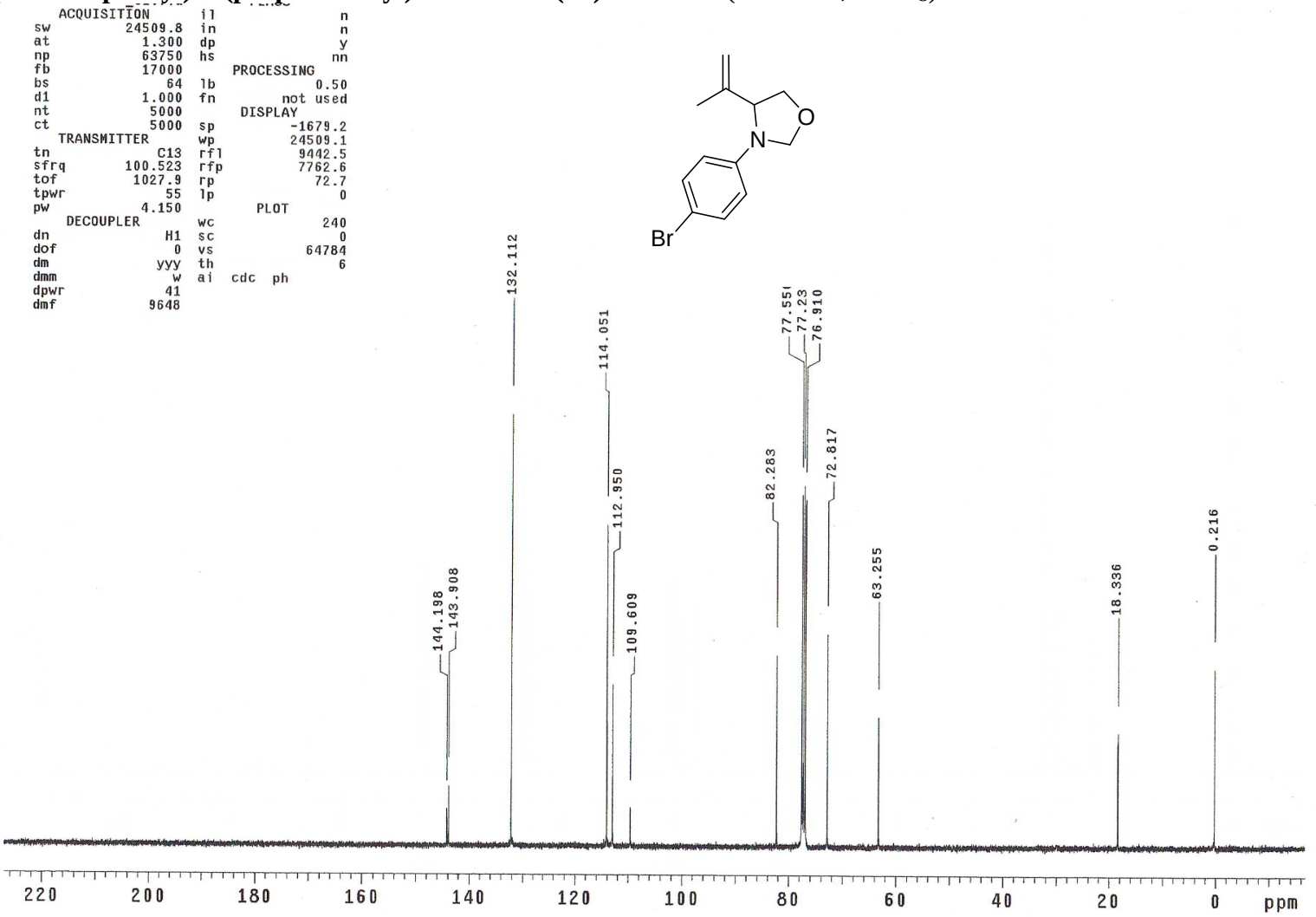
3-(4-chlorophenyl)-4-(prop-1-en-2-yl)oxazolidine (1c): HR-MS analysis



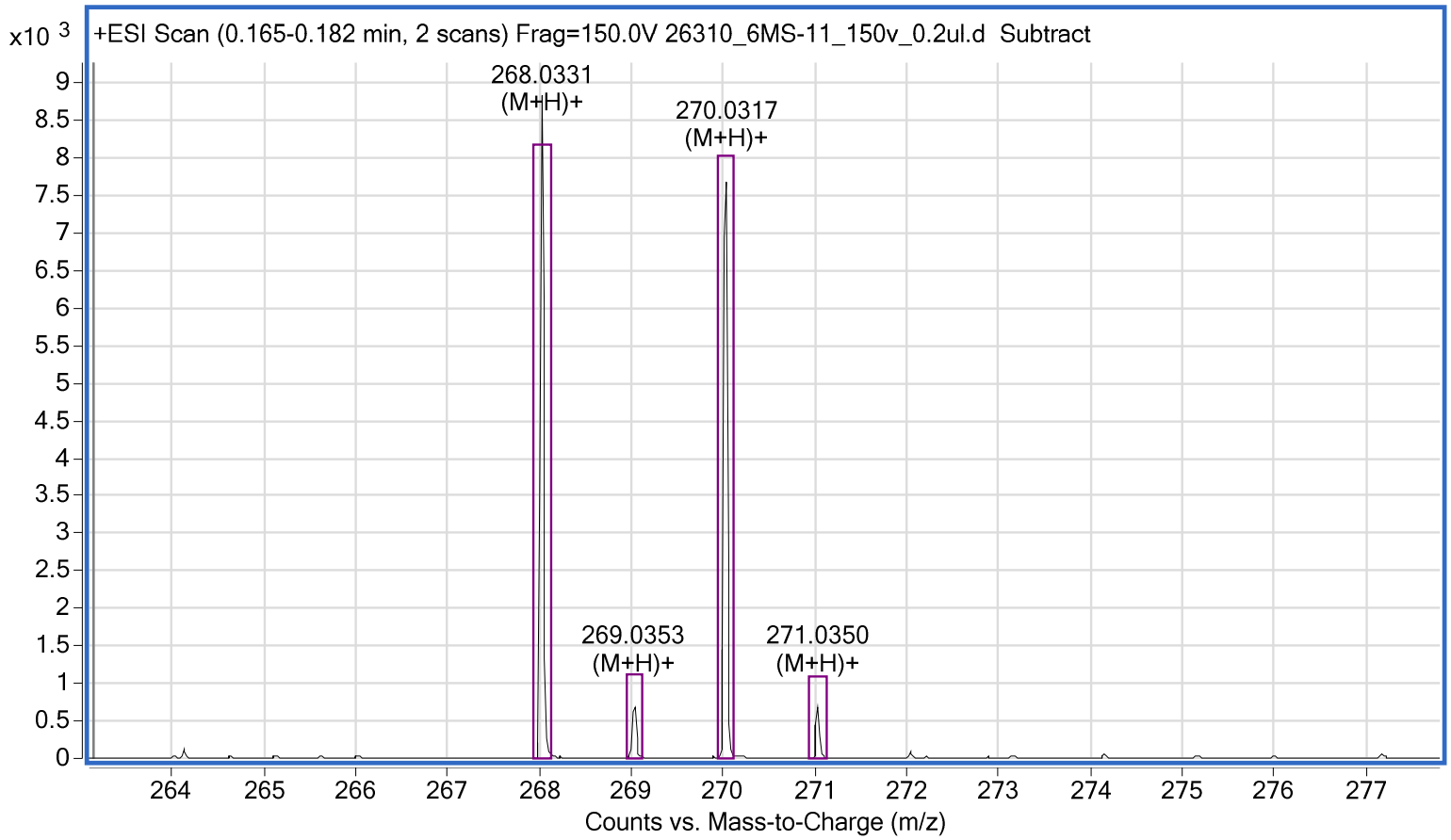
3-(4-bromophenyl)-4-(prop-1-en-2-yl)oxazolidine (1d): ^{1}H NMR (400 MHz, $CDCl_3$):



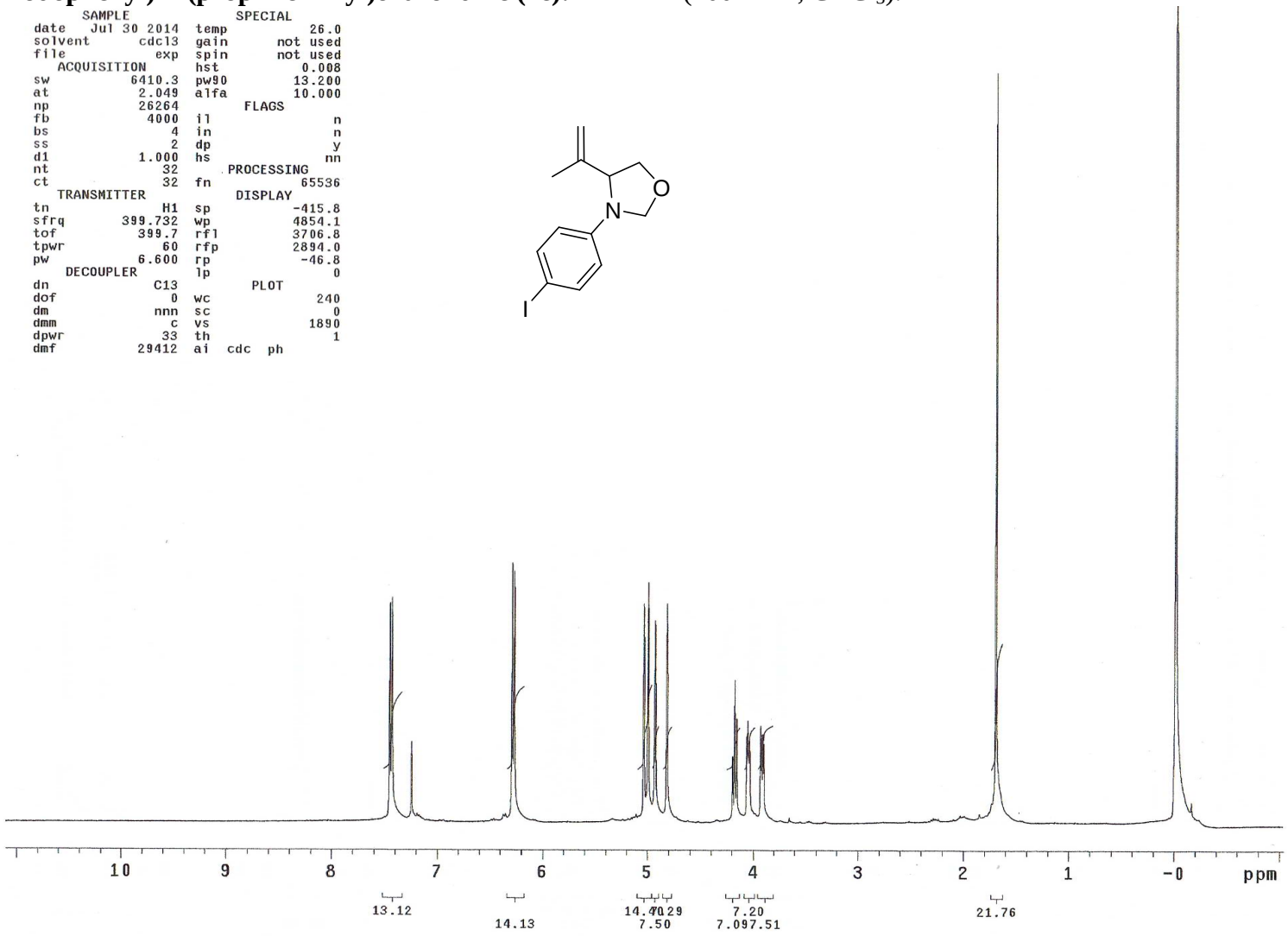
3-(4-bromophenyl)-4-(prop-1-en-2-yl)oxazolidine (1d): ^{13}C NMR (100 MHz, $CDCl_3$):



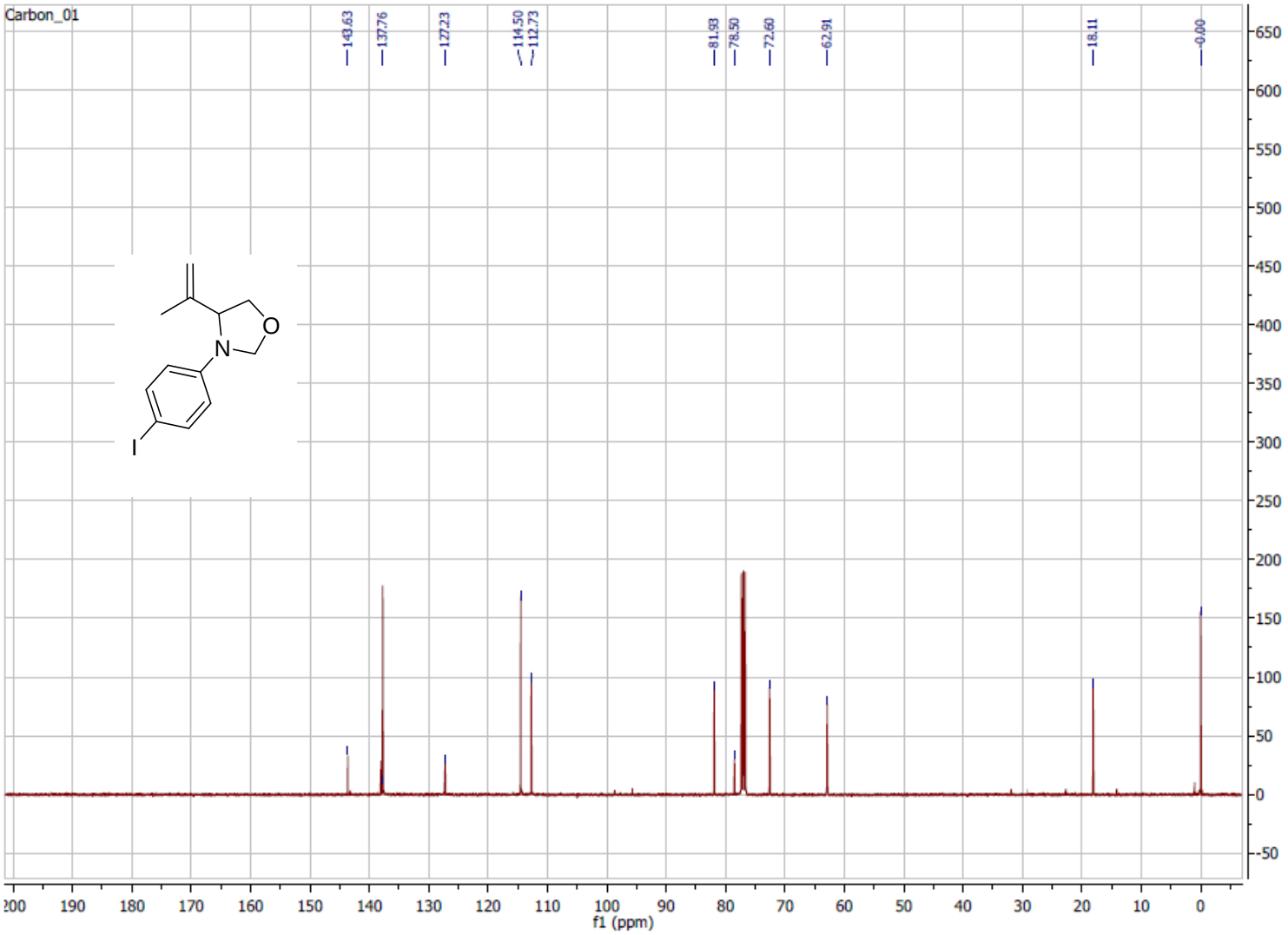
3-(4-bromophenyl)-4-(prop-1-en-2-yl)oxazolidine (1d): HR-MS analysis



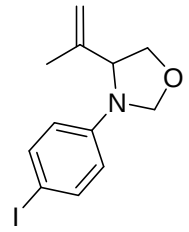
3-(4-iodophenyl)-4-(prop-1-en-2-yl)oxazolidine (1e): ^{1}H NMR (400 MHz, $CDCl_3$):

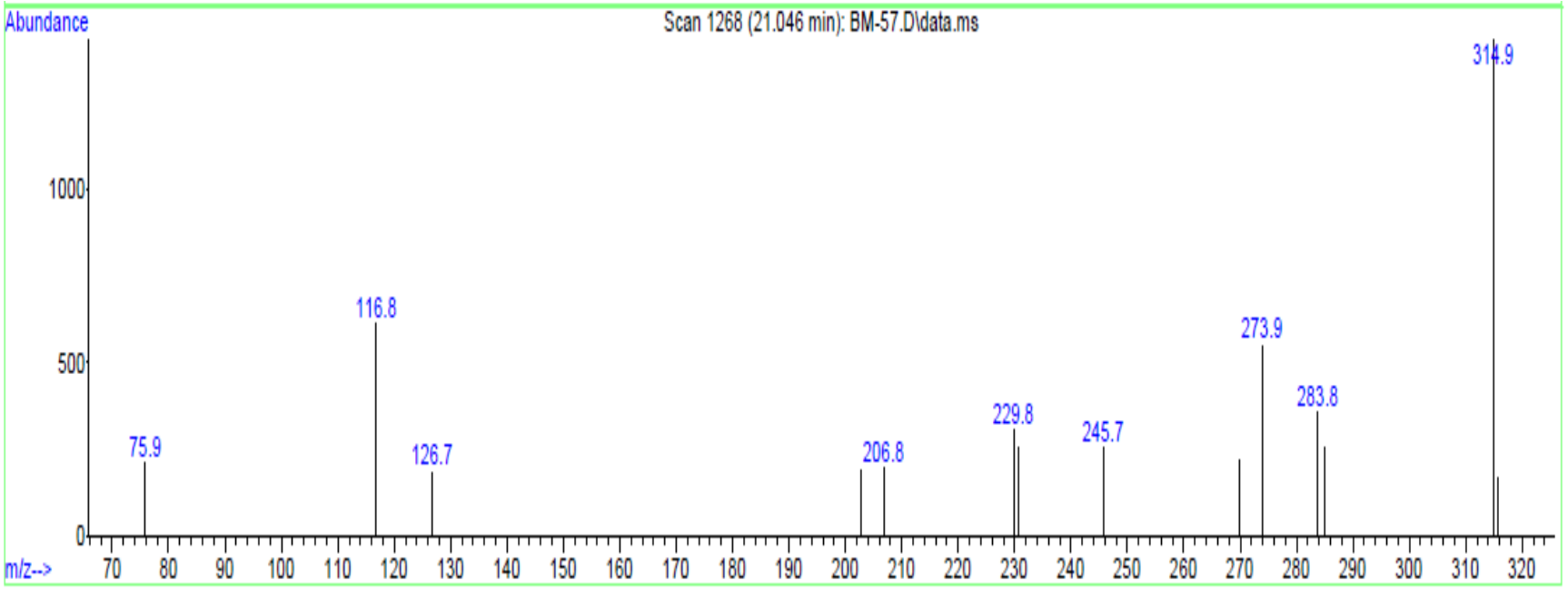


3-(4-iodophenyl)-4-(prop-1-en-2-yl)oxazolidine (1e): ^{13}C NMR (100 MHz, $CDCl_3$):

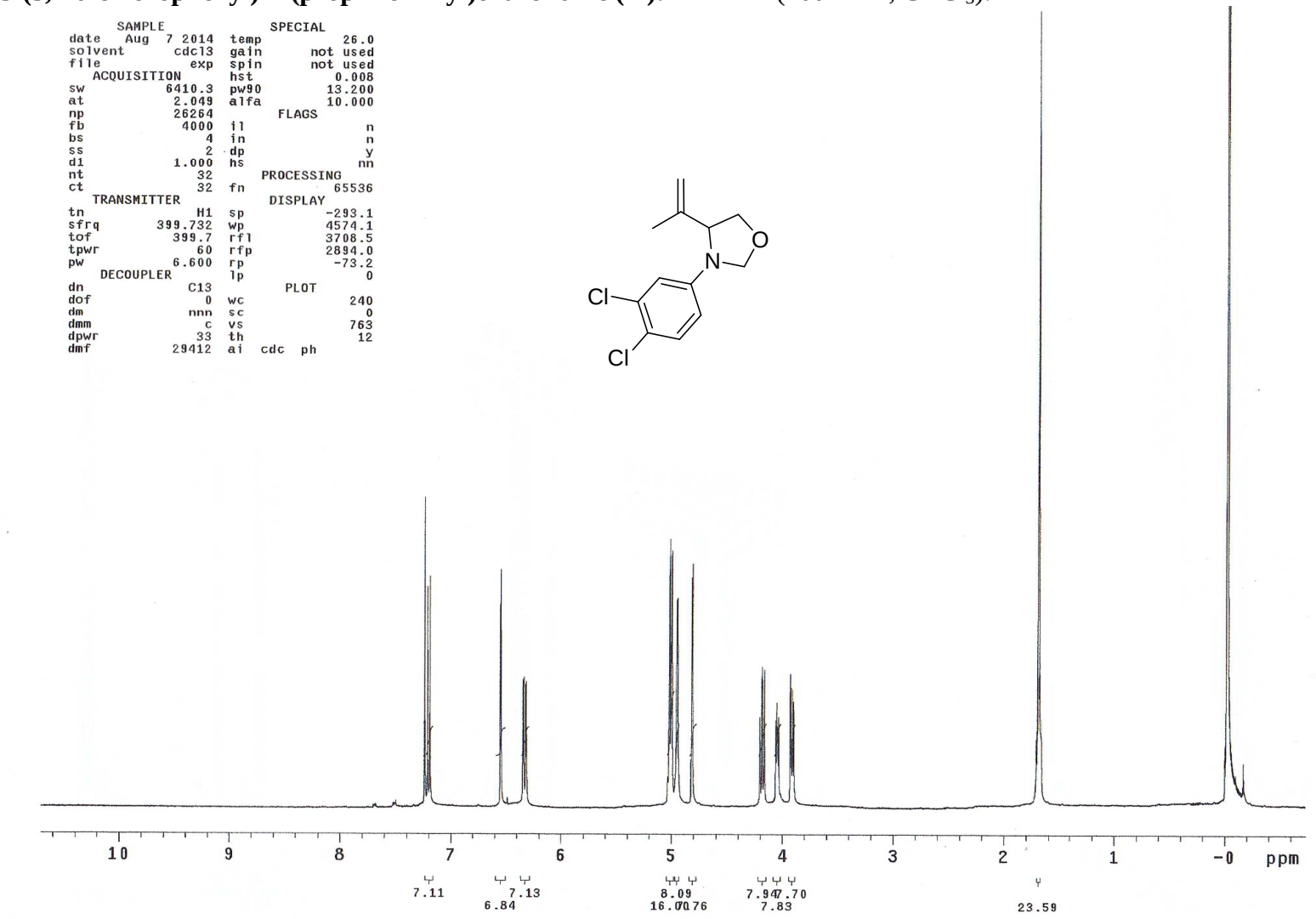


3-(4-iodophenyl)-4-(prop-1-en-2-yl)oxazolidine (1e): GC-MS analysis

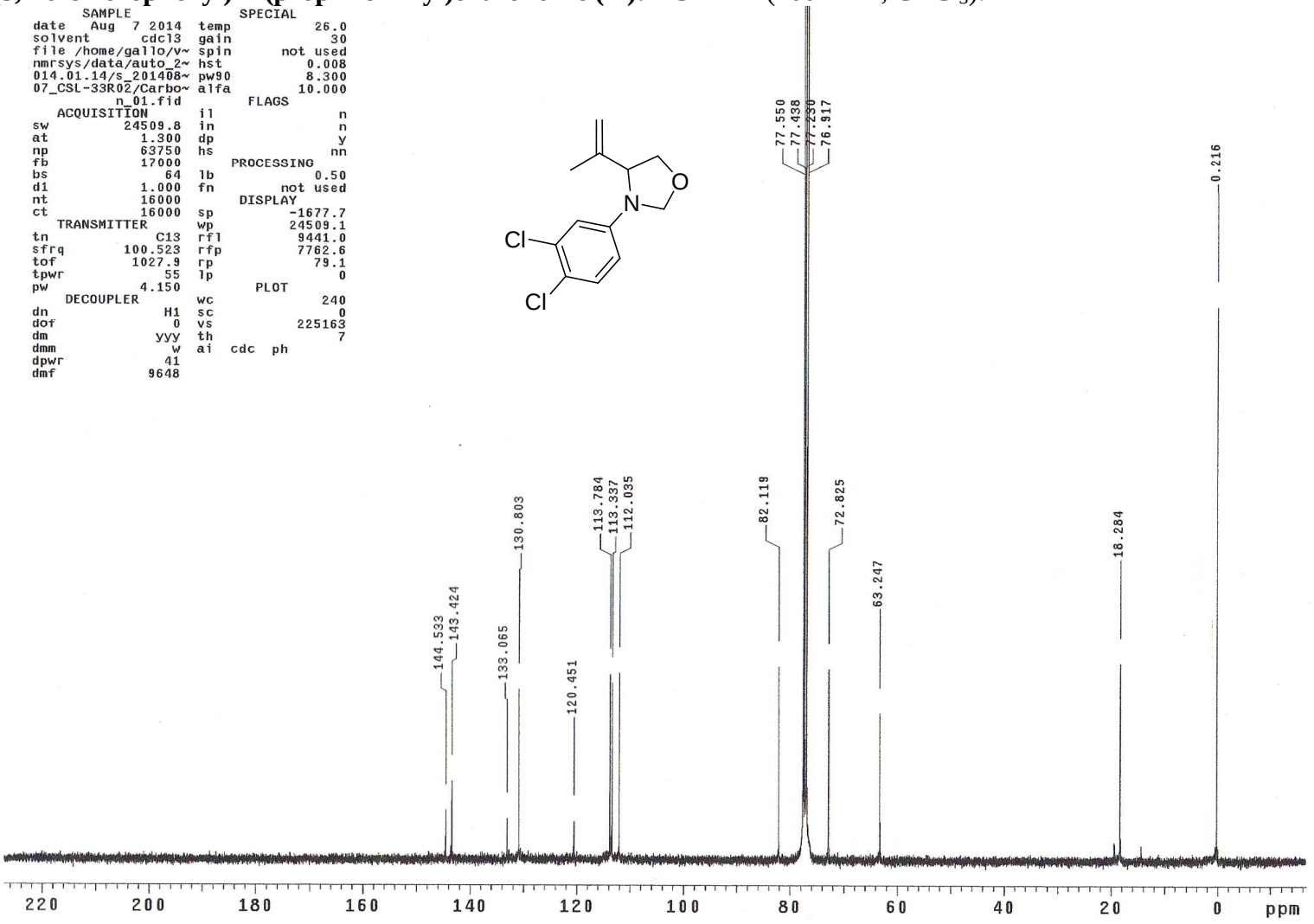




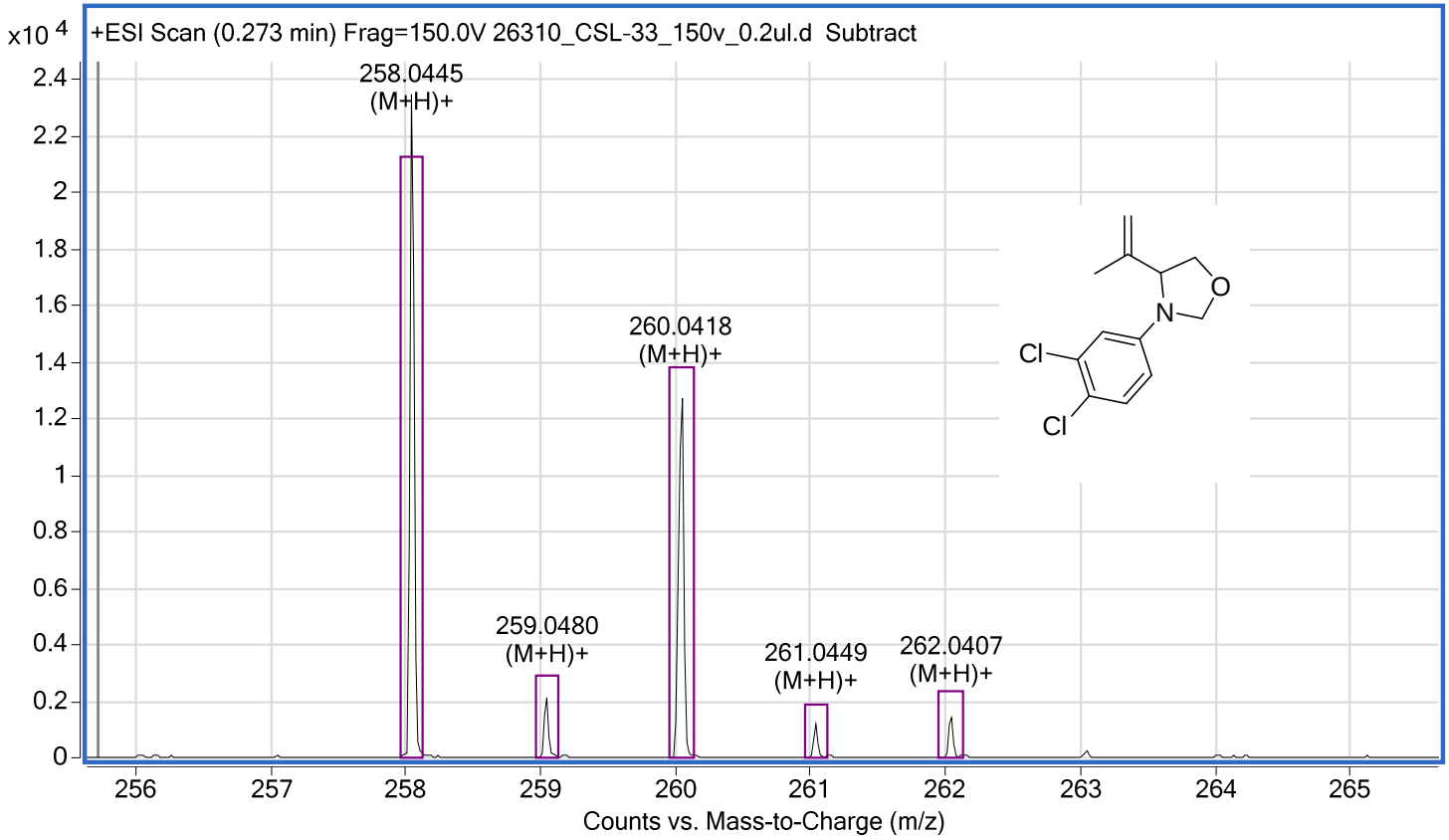
3-(3,4-dichlorophenyl)-4-(prop-1-en-2-yl)oxazolidine (1f): ^{1}H NMR (400 MHz, $CDCl_3$):



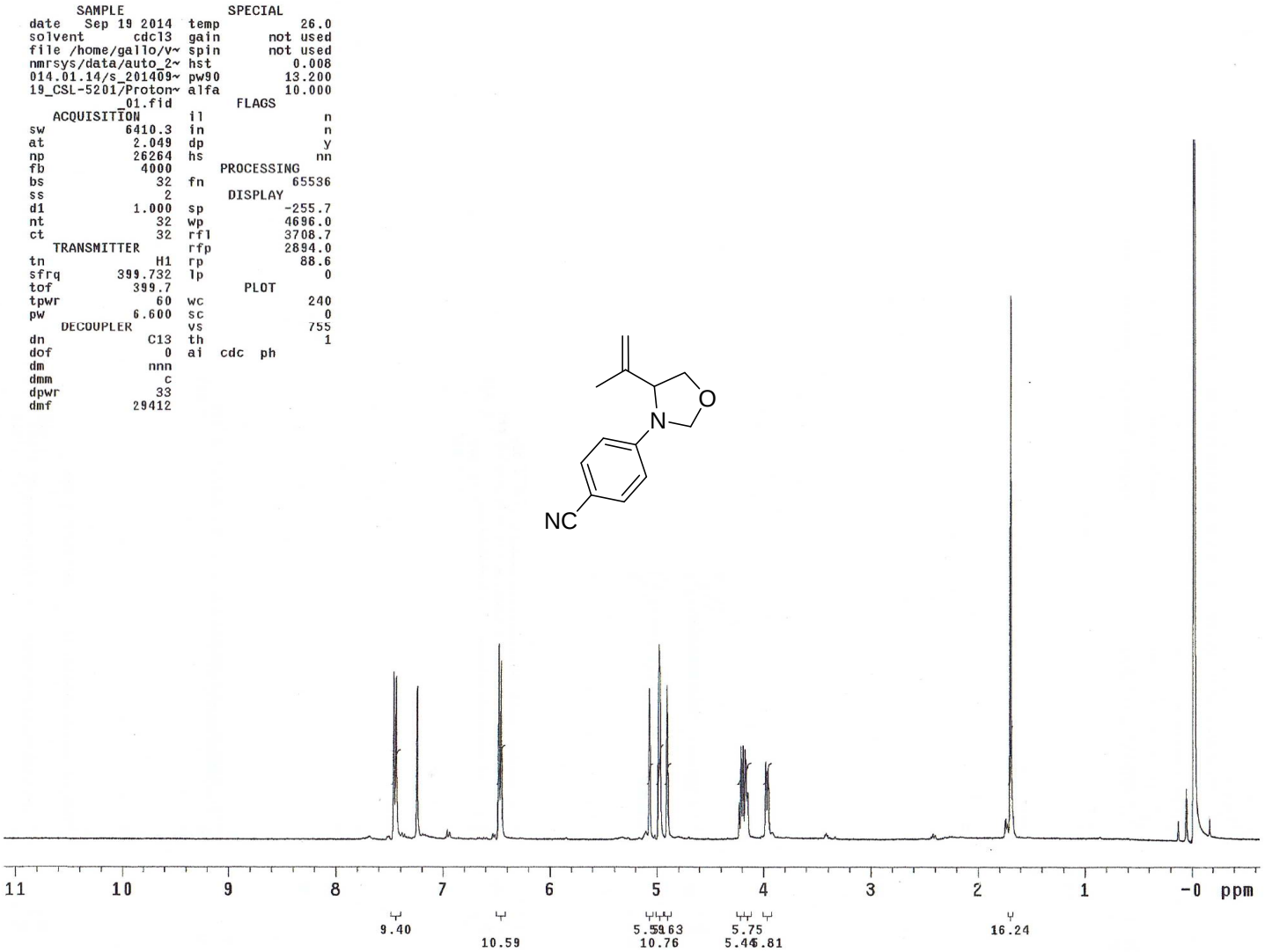
3-(3,4-dichlorophenyl)-4-(prop-1-en-2-yl)oxazolidine (1f): ^{13}C NMR (100 MHz, $CDCl_3$):



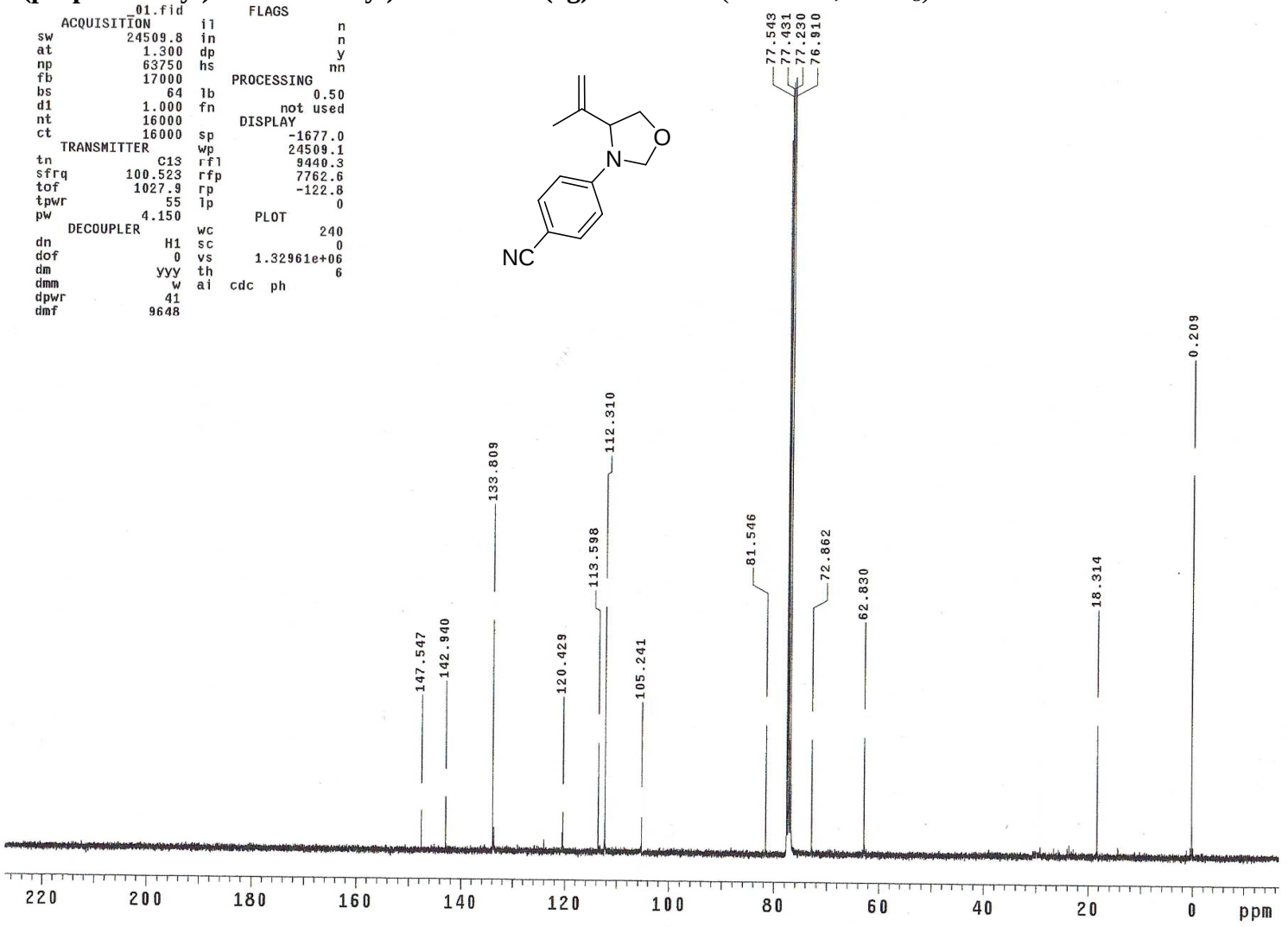
3-(3,4-dichlorophenyl)-4-(prop-1-en-2-yl)oxazolidine (1f): HR-MS analysis



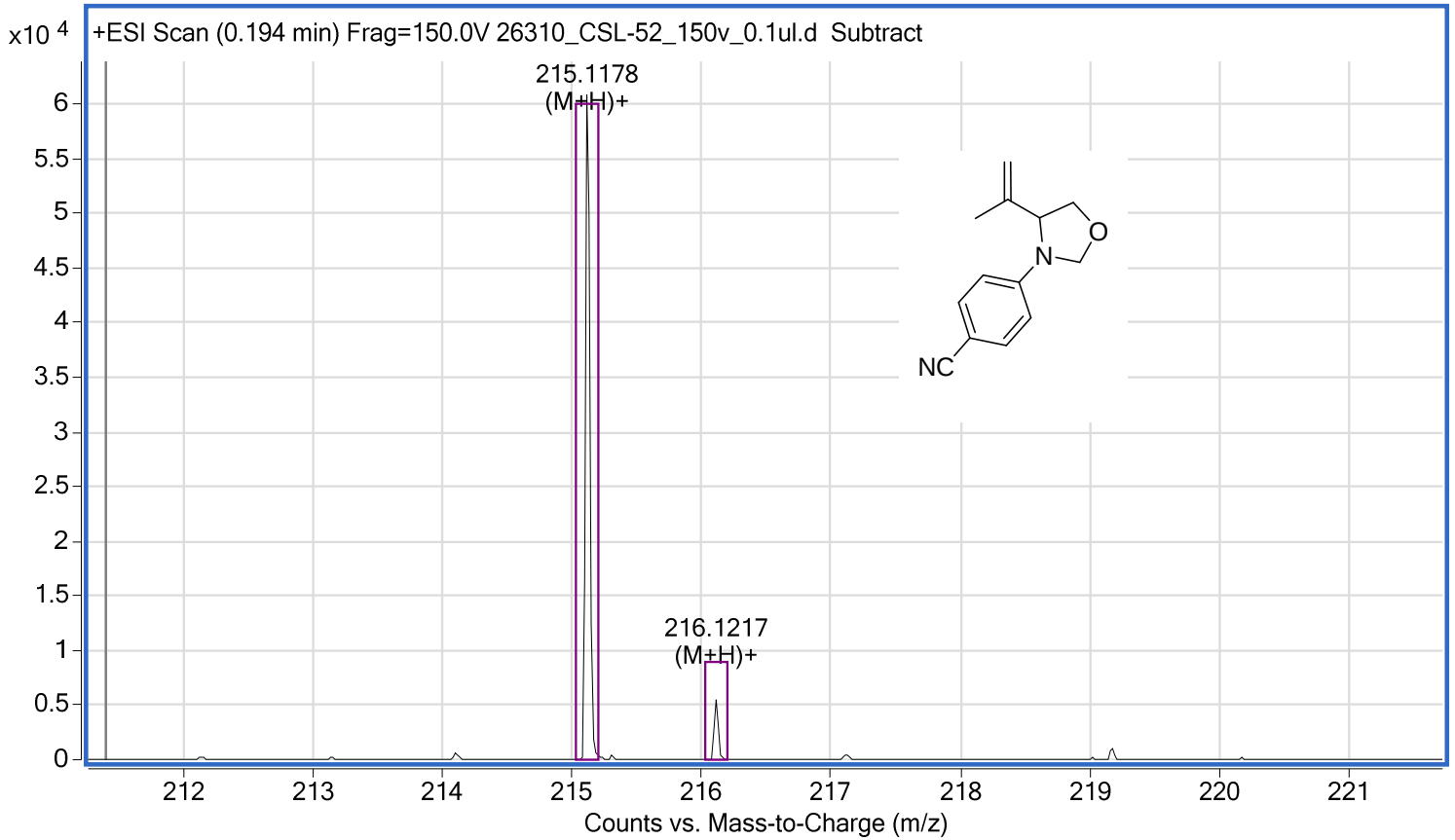
4-(4-(prop-1-en-2-yl)oxazolidin-3-yl)benzonitrile (1g): ^{1}H NMR (400 MHz, $CDCl_3$):



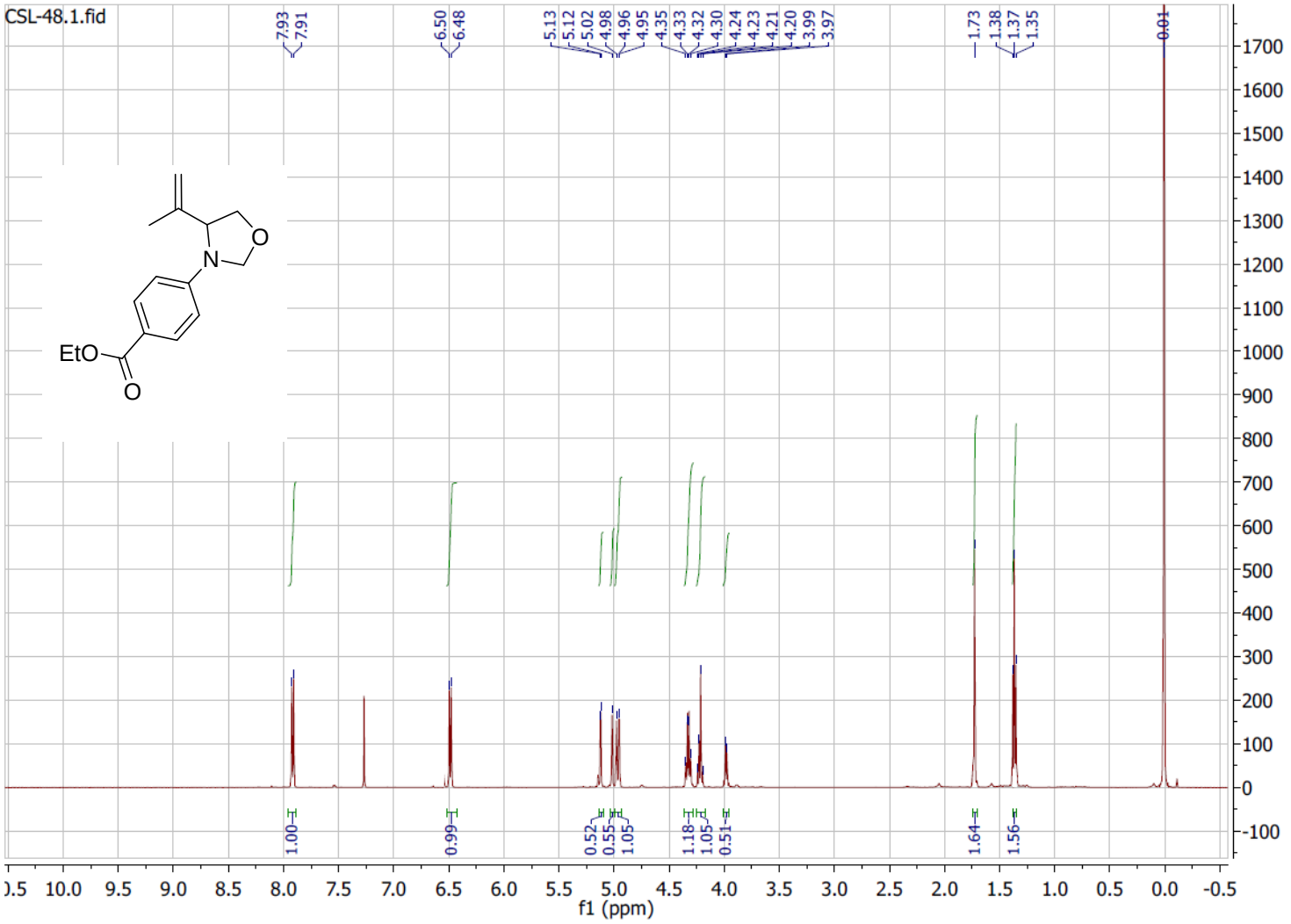
4-(4-(prop-1-en-2-yl)oxazolidin-3-yl)benzonitrile (1g): ^{13}C NMR (100 MHz, $CDCl_3$):



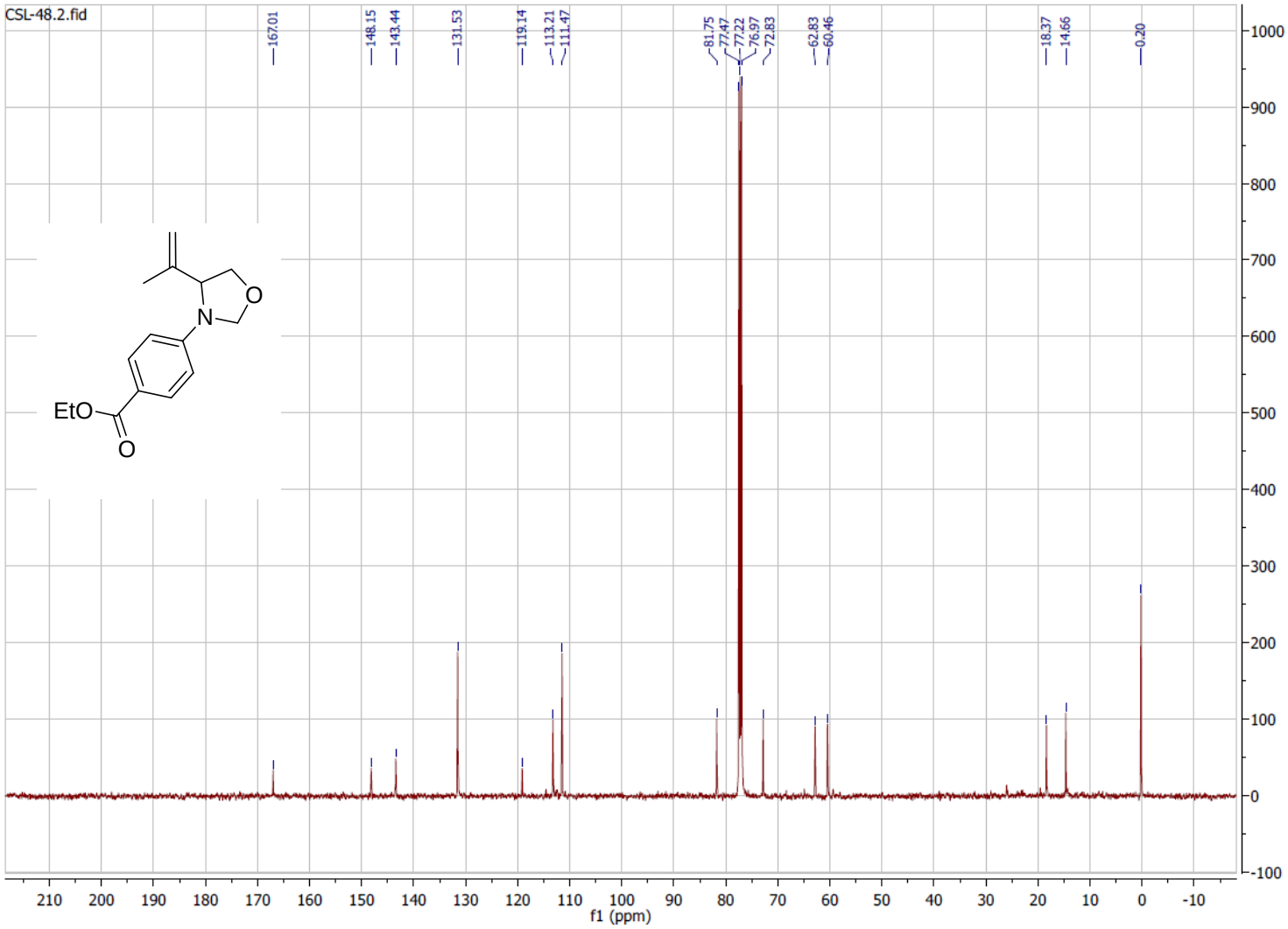
4-(4-(prop-1-en-2-yl)oxazolidin-3-yl)benzonitrile (1g): HR-MS analysis



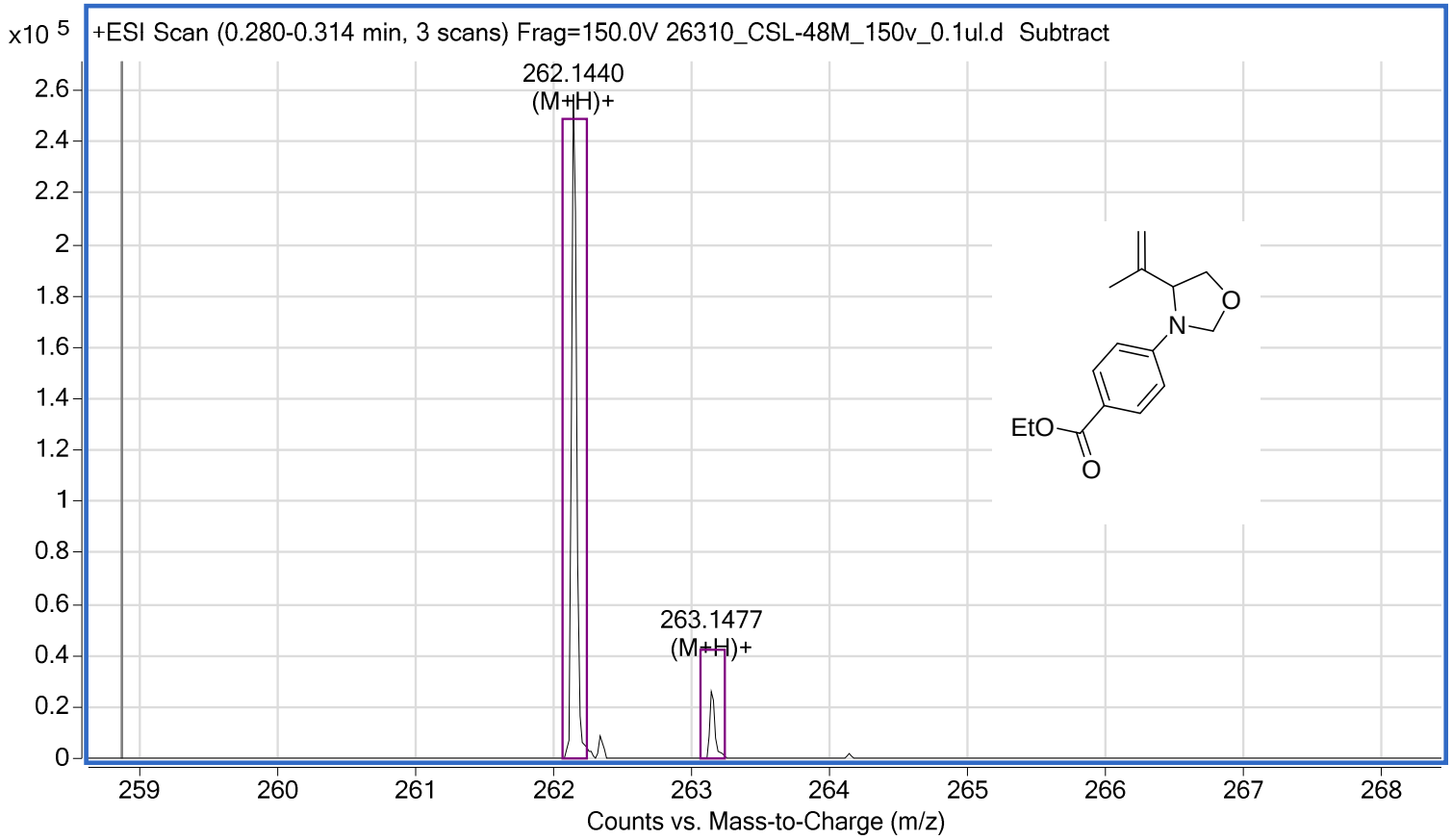
Ethyl 4-(4-(prop-1-en-2-yl)oxazolidin-3-yl)benzoate (1h): ^{1}H NMR (400 MHz, $CDCl_3$)



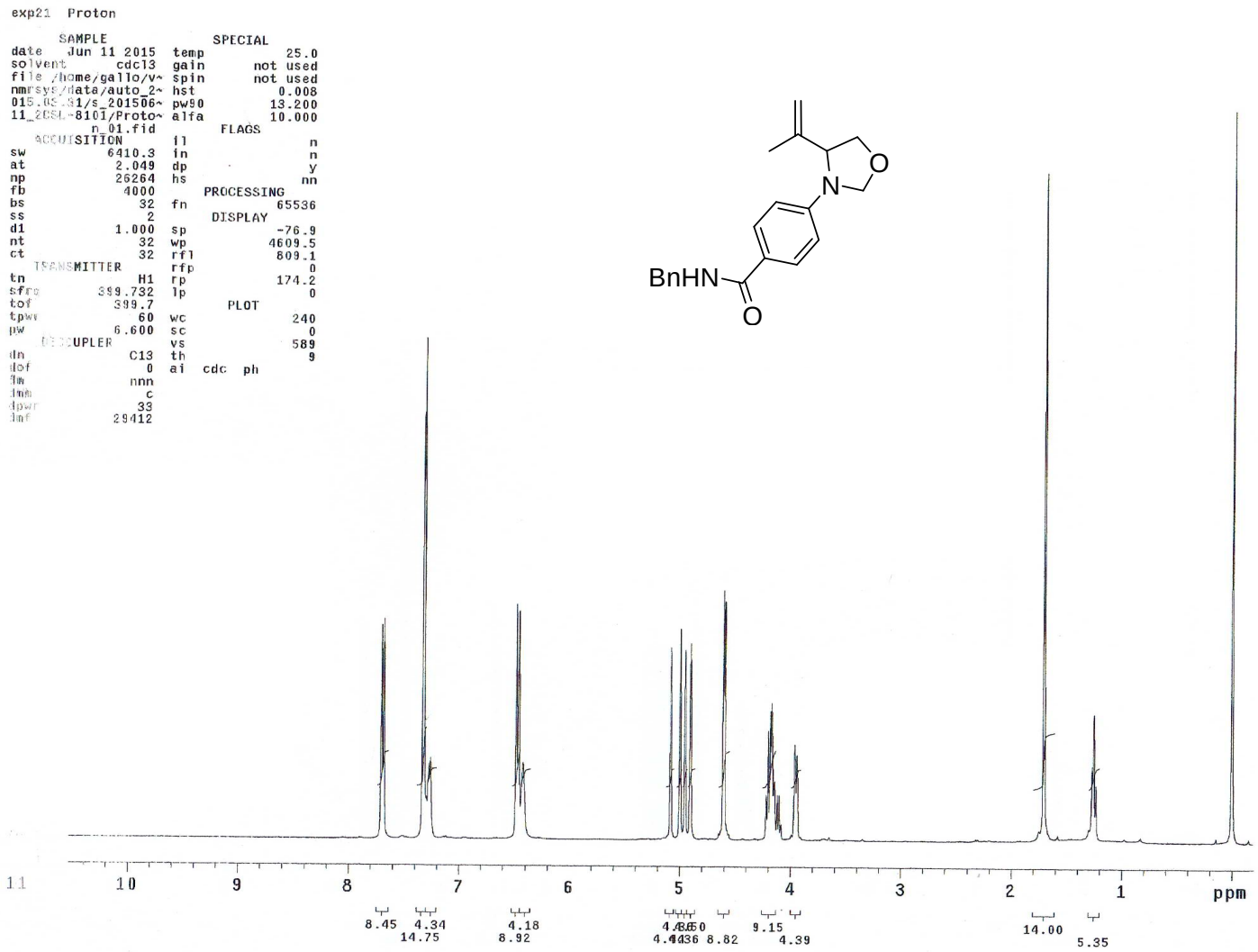
Ethyl 4-(4-(prop-1-en-2-yl)oxazolidin-3-yl)benzoate (1h): ^{13}C NMR (100 MHz, $CDCl_3$)



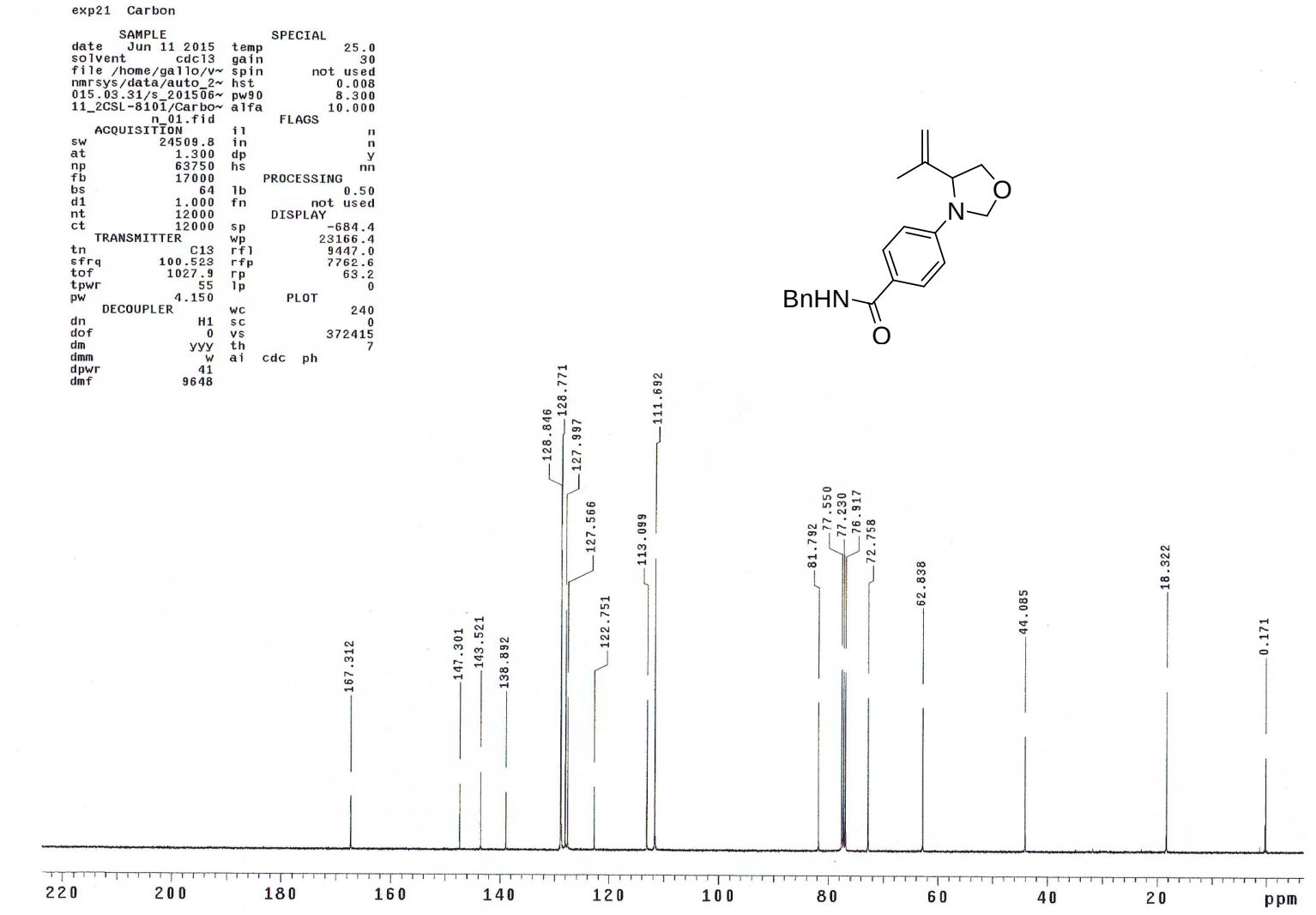
Ethyl 4-(4-(prop-1-en-2-yl)oxazolidin-3-yl)benzoate (1h): HR-MS analysis



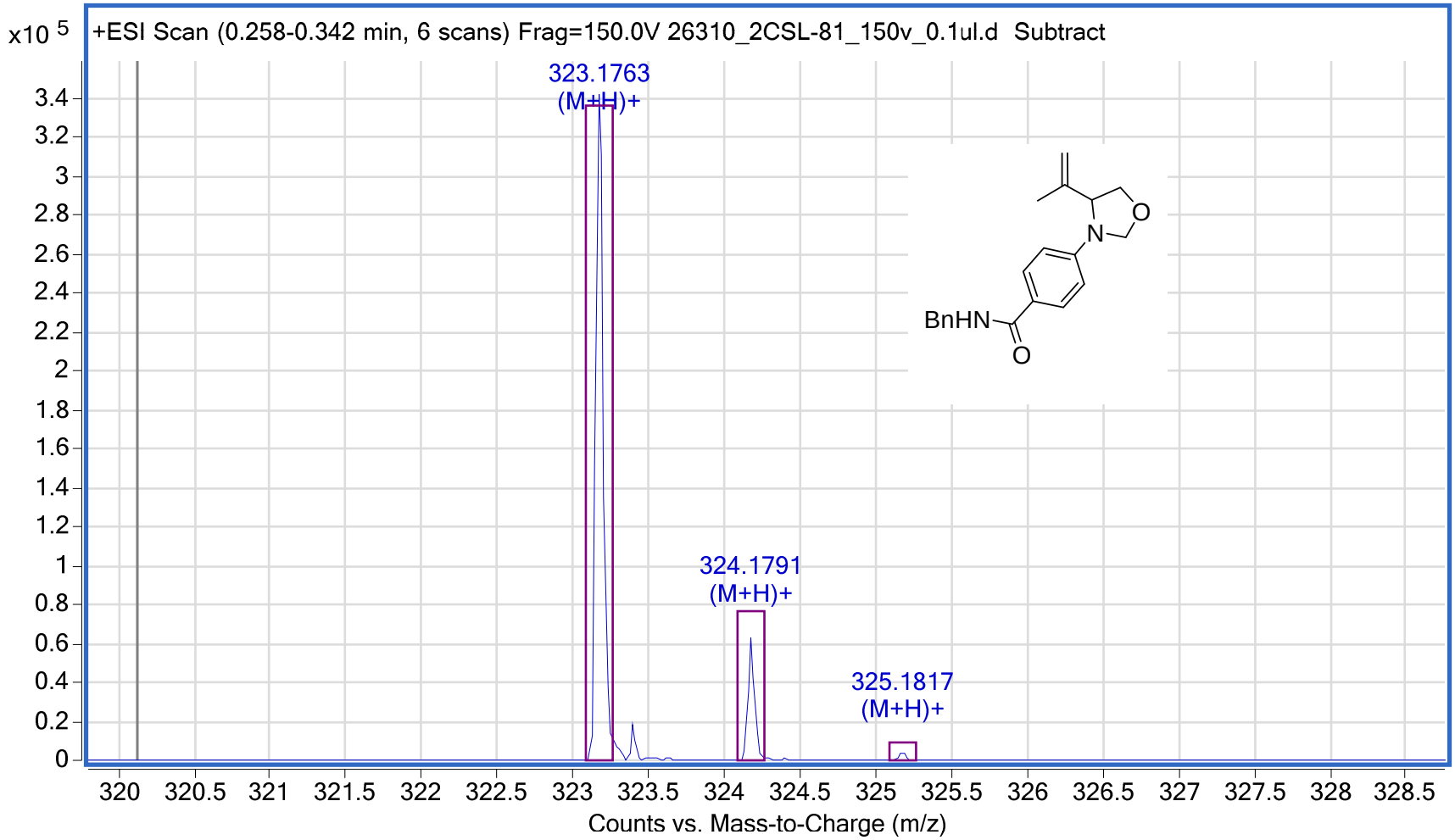
N-benzyl-4-(4-(prop-1-en-2-yl)oxazolidin-3-yl)benzamide (1i): ^{1}H NMR (400 MHz, $CDCl_3$)



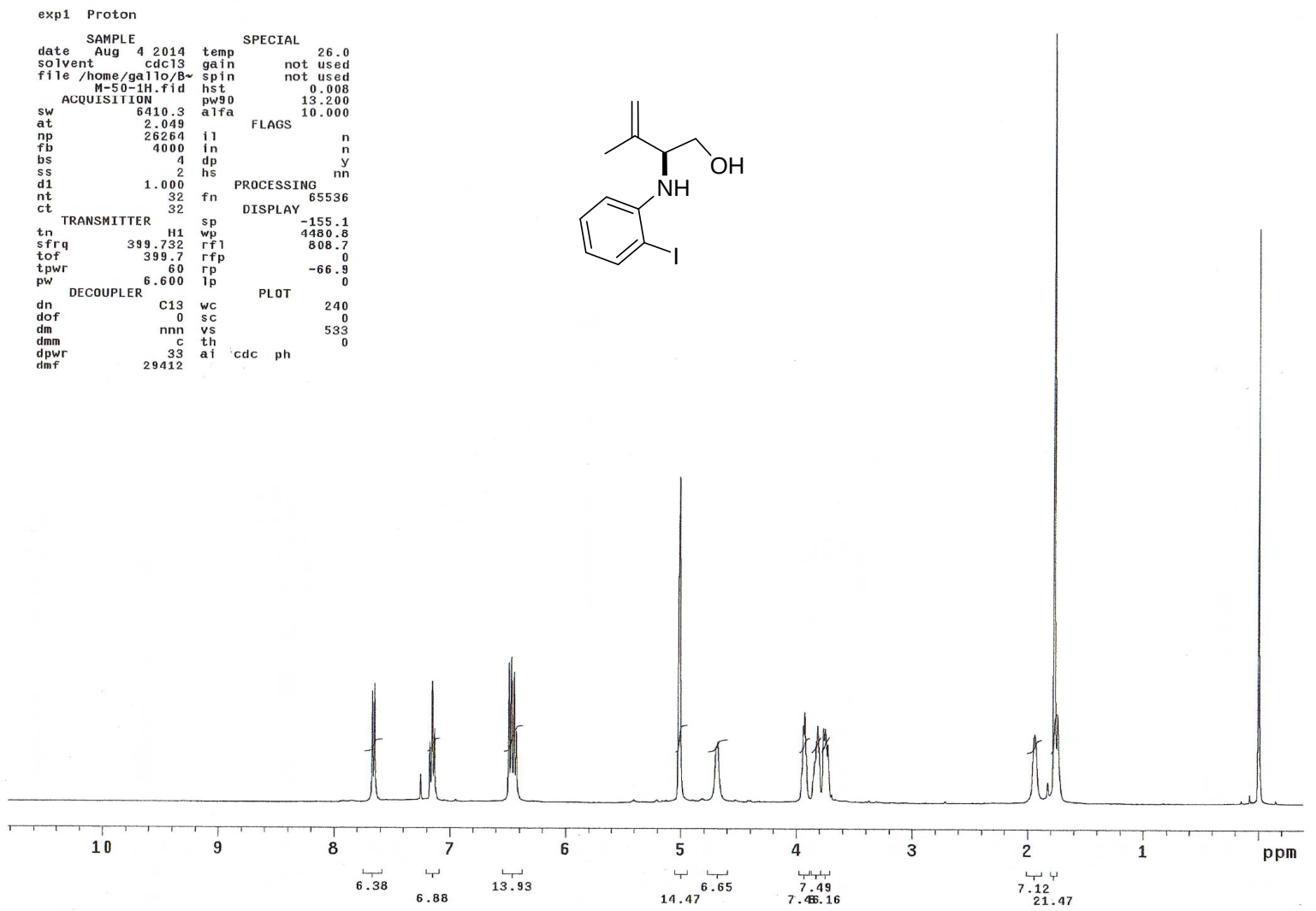
N-benzyl-4-(4-(prop-1-en-2-yl)oxazolidin-3-yl)benzamide (1i): ^{13}C NMR (100 MHz, $CDCl_3$)



N-benzyl-4-(4-(prop-1-en-2-yl)oxazolidin-3-yl)benzamide (1i): HR-MS analysis



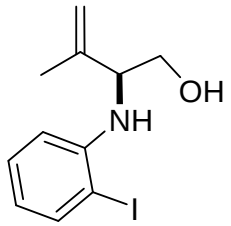
2-(2-iodophenylamino)-3-methylbut-3-en-1-ol (1k): ^{1}H NMR (400 MHz, $CDCl_3$)

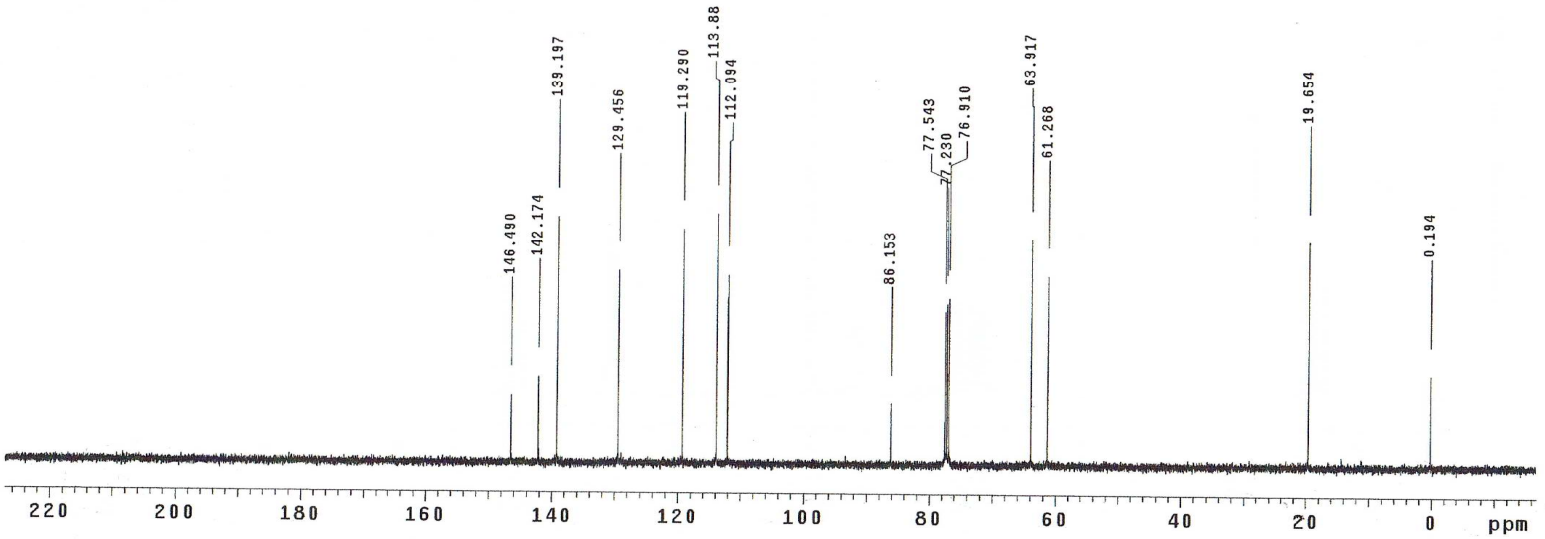


2-(2-iodophenylamino)-3-methylbut-3-en-1-ol (1k): ^{13}C NMR (100 MHz, $CDCl_3$)

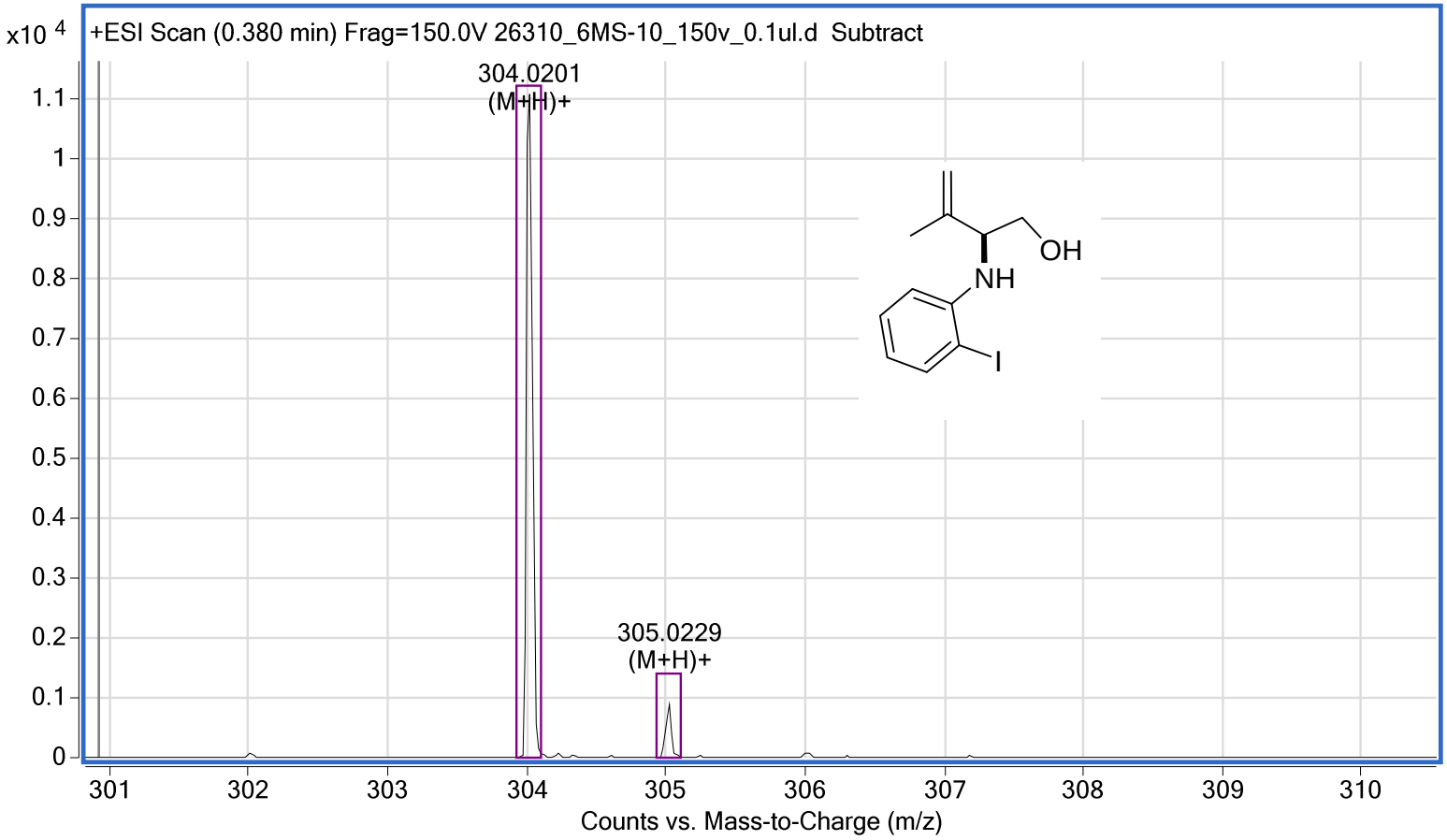
```
exp1  Carbon

      SAMPLE                SPECIAL
date    Aug  4 2014  temp            26.0
solvent       cdcl3  gain              30
file            exp  spin        not used
   ACQUISITION        hst             0.008
sw           24509.8  pw90            8.300
at             1.300  alfa           10.000
np             63750        FLAGS
fb             17000  il                 n
bs                16  in                 n
d1             1.000  dp                 y
nt              4000  hs                nn
ct               304      PROCESSING
   TRANSMITTER        lb              0.50
tn               C13  fn          not used
sfrq         100.523        DISPLAY
tof           1027.9  sp           -1683.7
tpwr              55  wp           24509.1
pw             4.150  rfl           9447.0
    DECOUPLER        rfp           7762.6
dn                H1  rp              77.0
dof                0  lp                 0
dm               yyy          PLOT
dmm                w  wc               240
dpwr              41  sc                 0
dmf             9648  vs             23995
                      th                 9
                      ai  cdc  ph
```

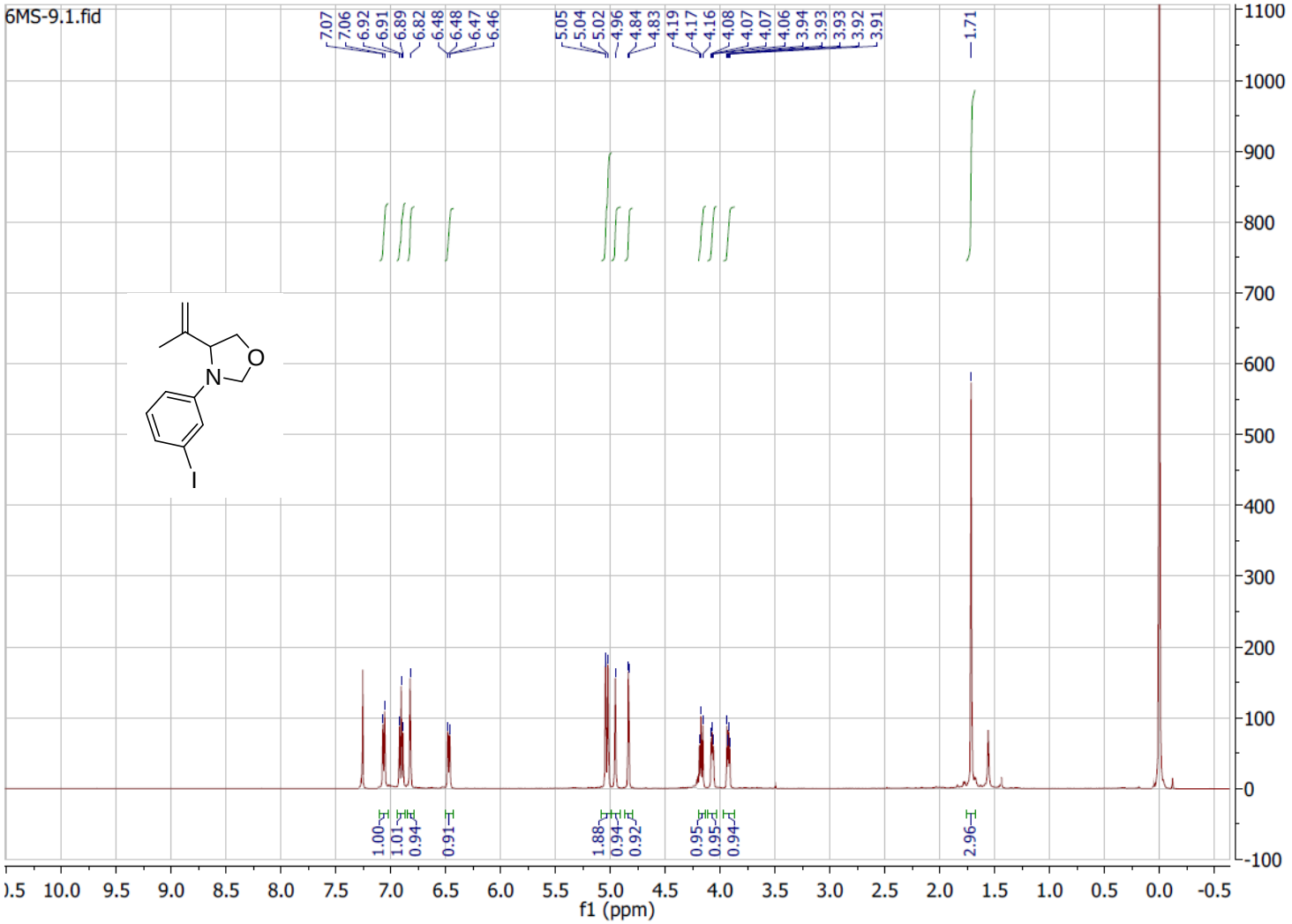




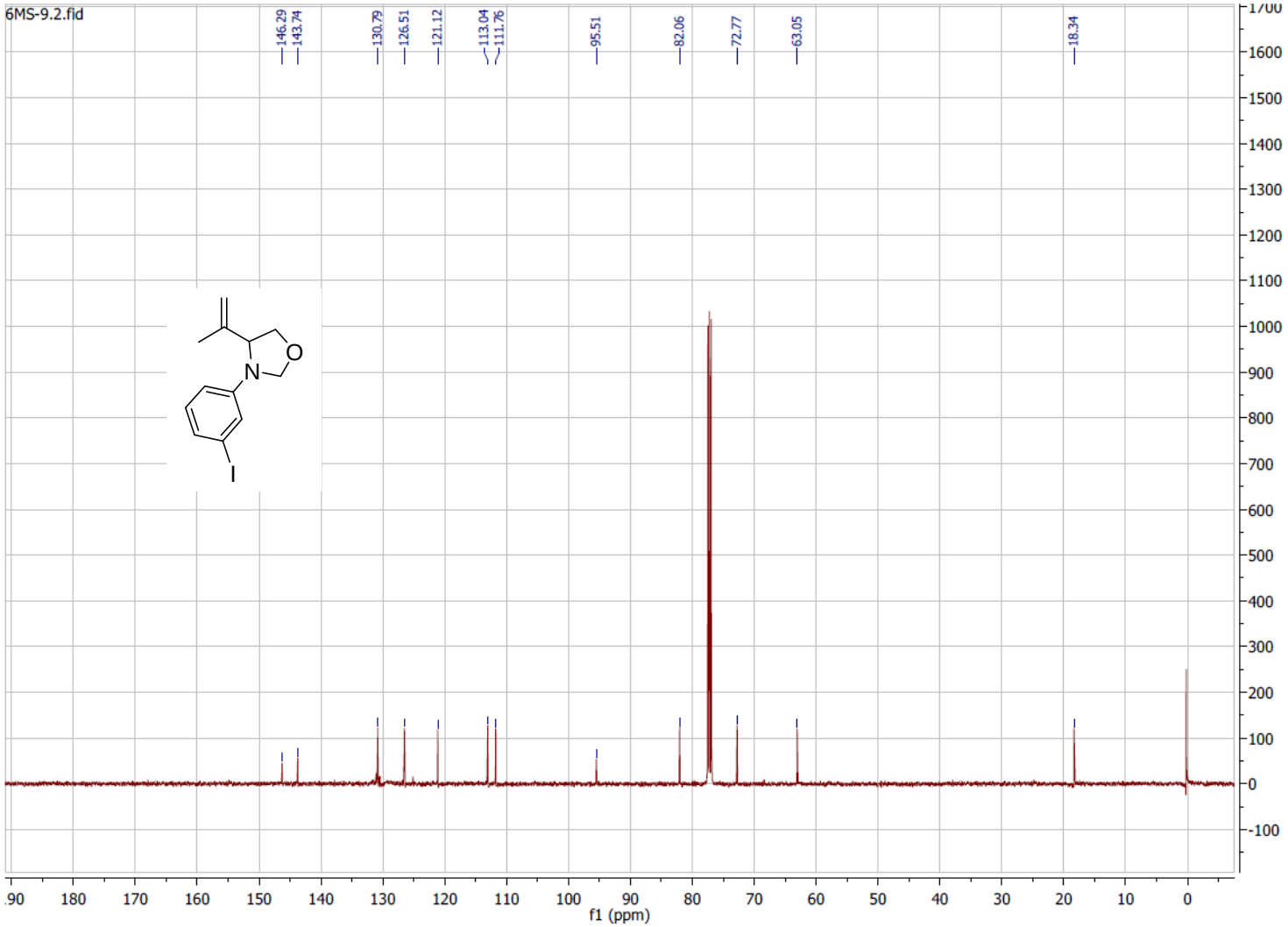
2-(2-iodophenylamino)-3-methylbut-3-en-1-ol (1k): HR-MS analysis



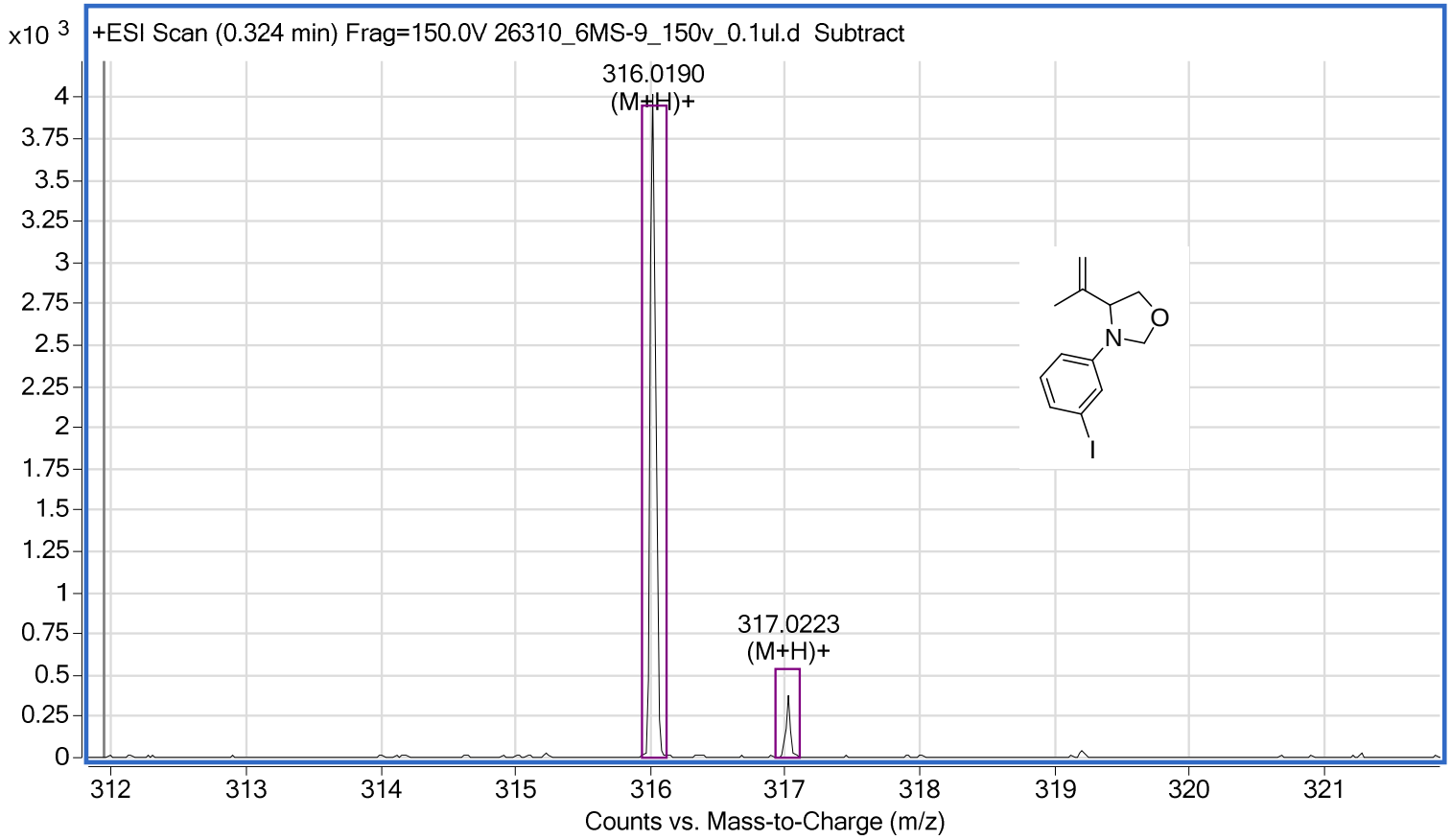
3-(3-iodophenyl)-4-(prop-1-en-2-yl)oxazolidine (1l): ^{1}H NMR (400 MHz, $CDCl_3$)



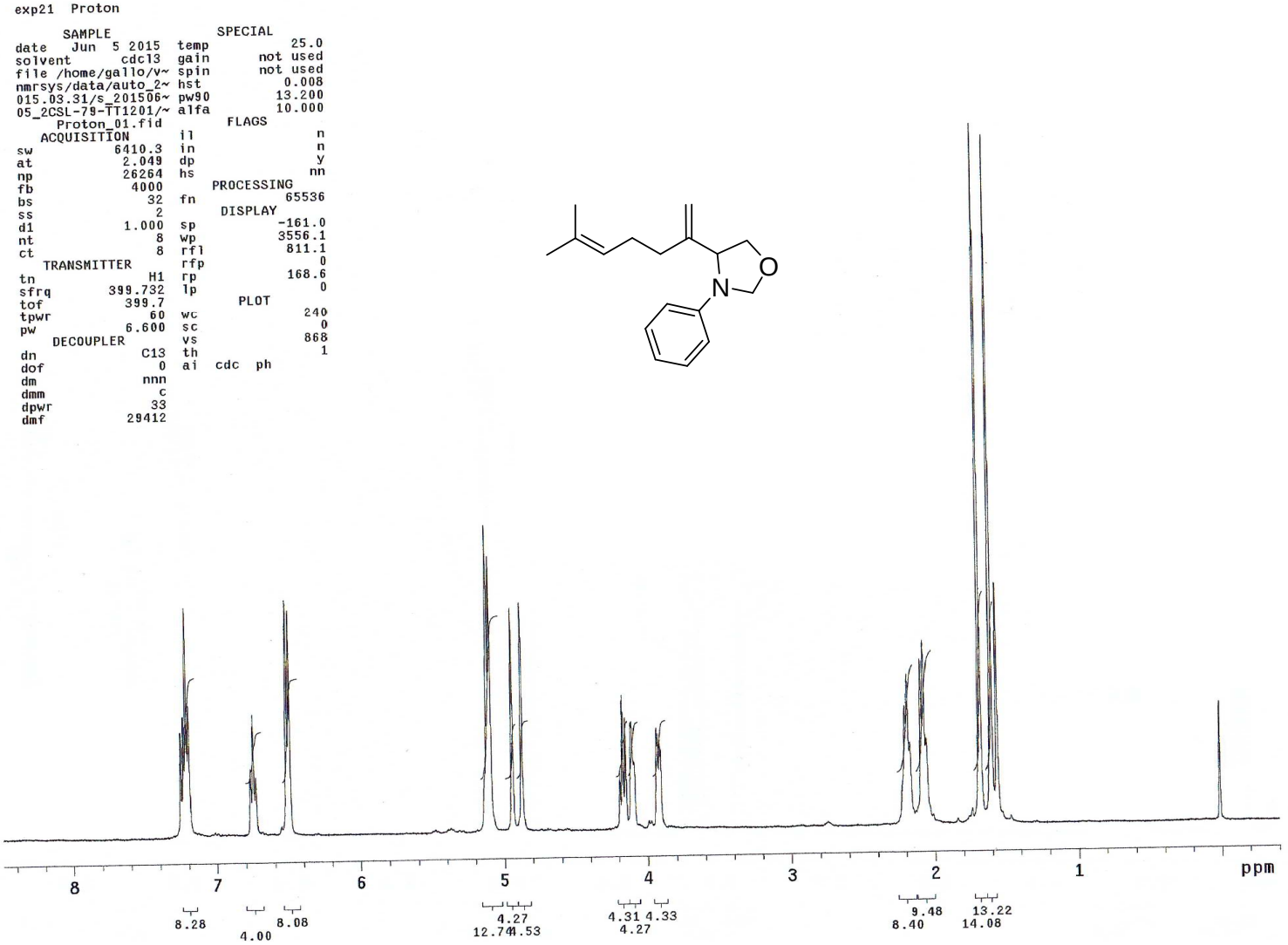
3-(3-iodophenyl)-4-(prop-1-en-2-yl)oxazolidine (1l): ^{13}C NMR (100 MHz, $CDCl_3$)



3-(3-iodophenyl)-4-(prop-1-en-2-yl)oxazolidine (1l): HR-MS analysis



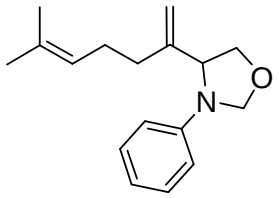
4-(6-methylhepta-1,5-dien-2-yl)-3-phenyloxazolidine (2a): ^{1}H NMR (400 MHz, $CDCl_3$)

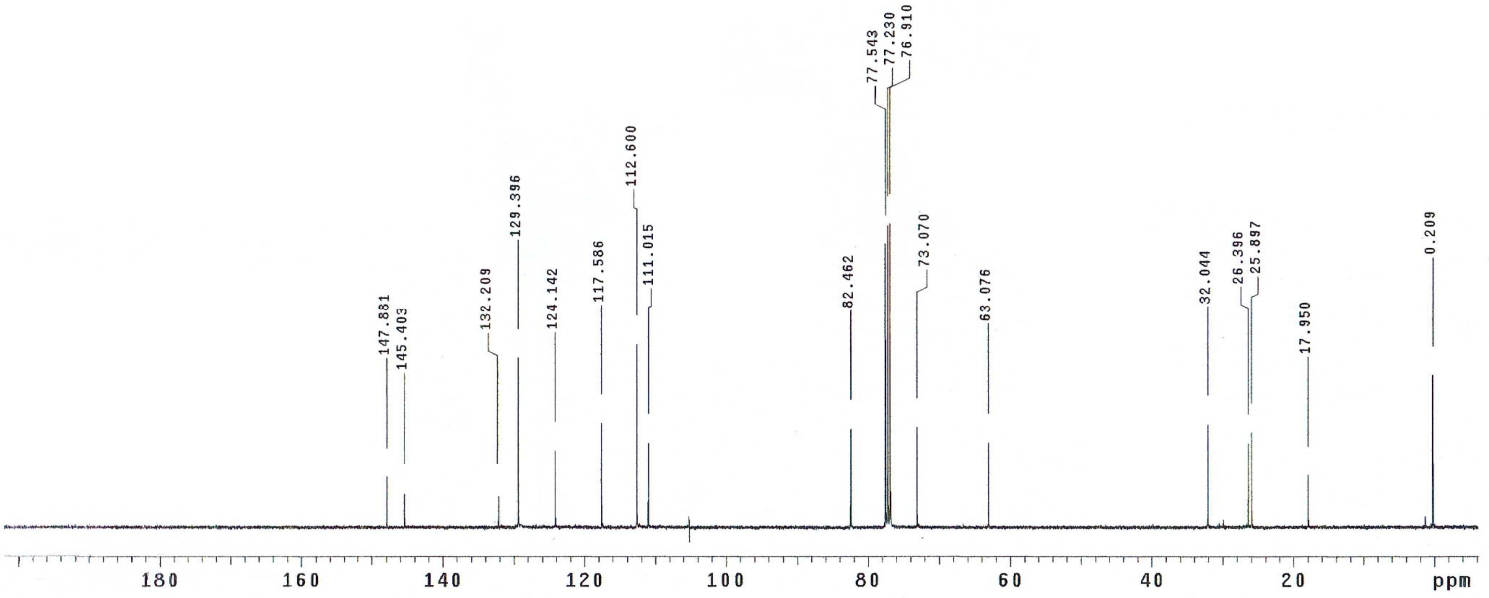


4-(6-methylhepta-1,5-dien-2-yl)-3-phenyloxazolidine (2a): ^{13}C NMR (100 MHz, $CDCl_3$)

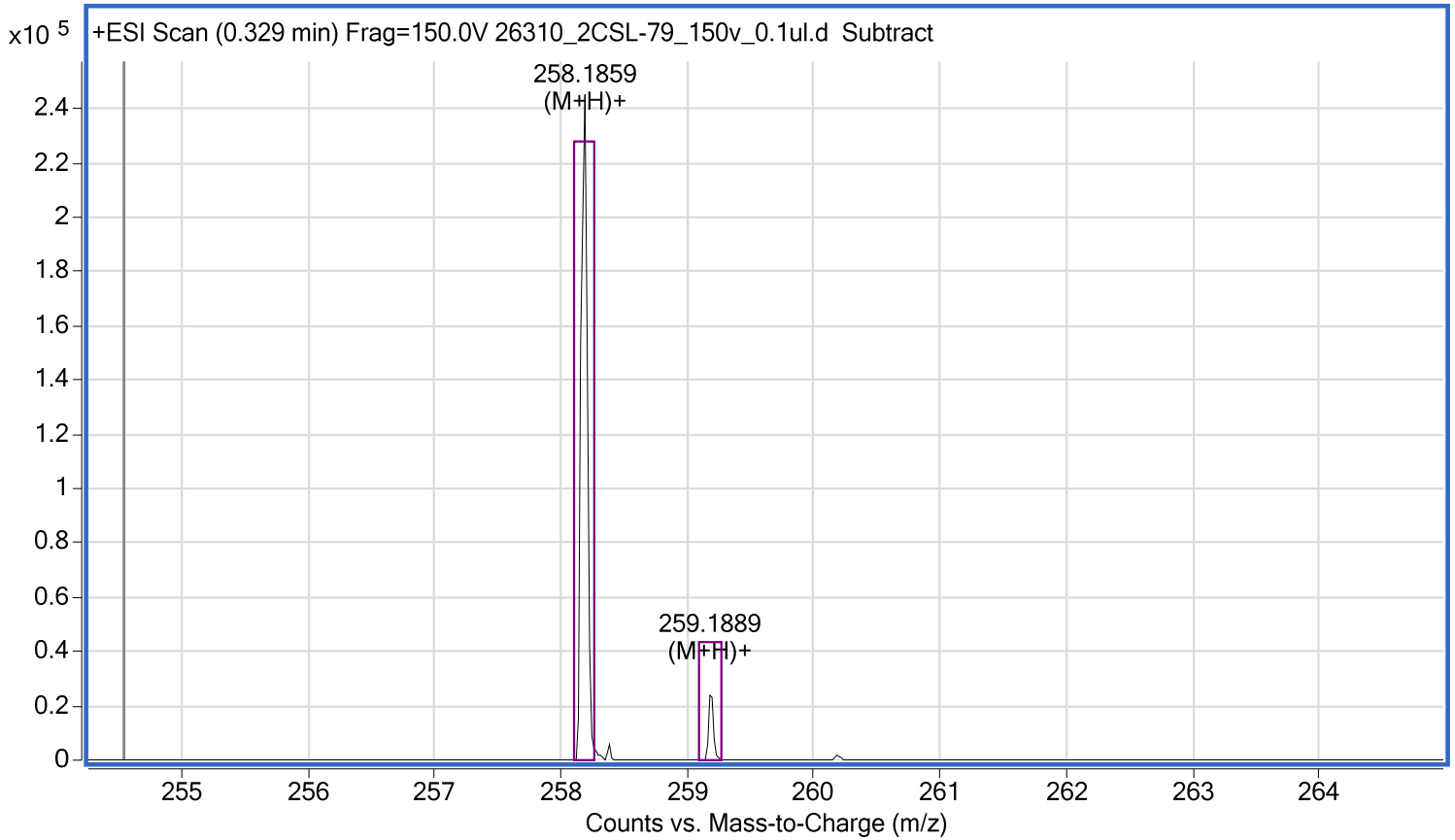
exp21 Carbon

SAMPLE		SPECIAL	
date	Jun 5 2015	temp	25.0
solvent	cdcl3	gain	30
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	24509.8	pw90	8.300
at	1.300	alfa	10.000
np	63750	FLAGS	
fb	17000	il	n
bs	32	in	n
d1	1.000	dp	y
nt	10000	hs	nn
ct	10000	PROCESSING	
TRANSMITTER		lb	0.50
tn	C13	fn	not used
sfrq	100.523	DISPLAY	
tof	1027.9	sp	-622.3
tpwr	55	wp	20938.2
pw	4.150	rfl	9442.5
DECOUPLER		rfp	7762.6
dn	H1	rp	60.5
dof	0	lp	0
dm	yyy	PLOT	
dmm	w	wc	240
dpwr	41	sc	0
dmf	9648	vs	461966
		th	8
		ai	cdc ph

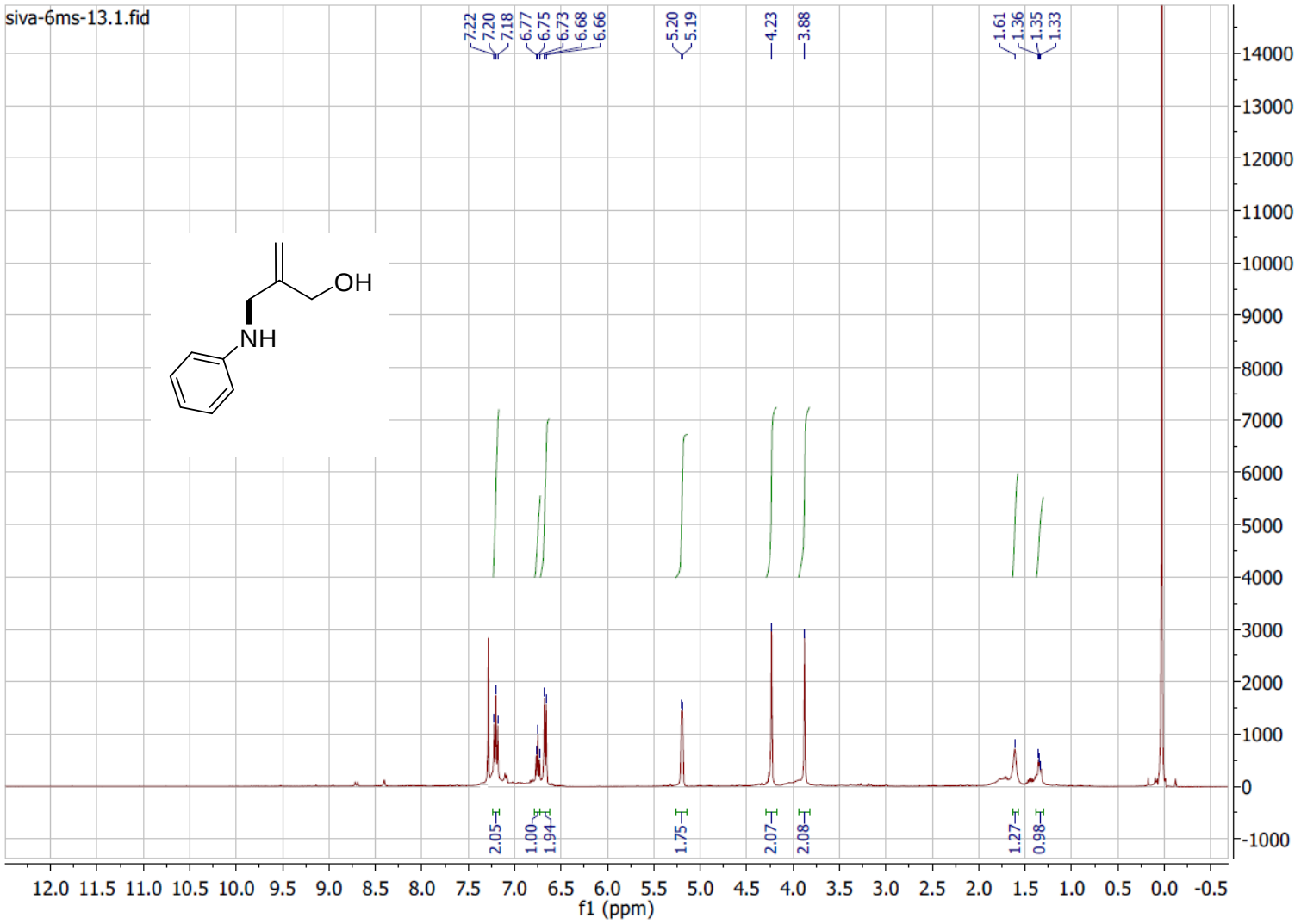




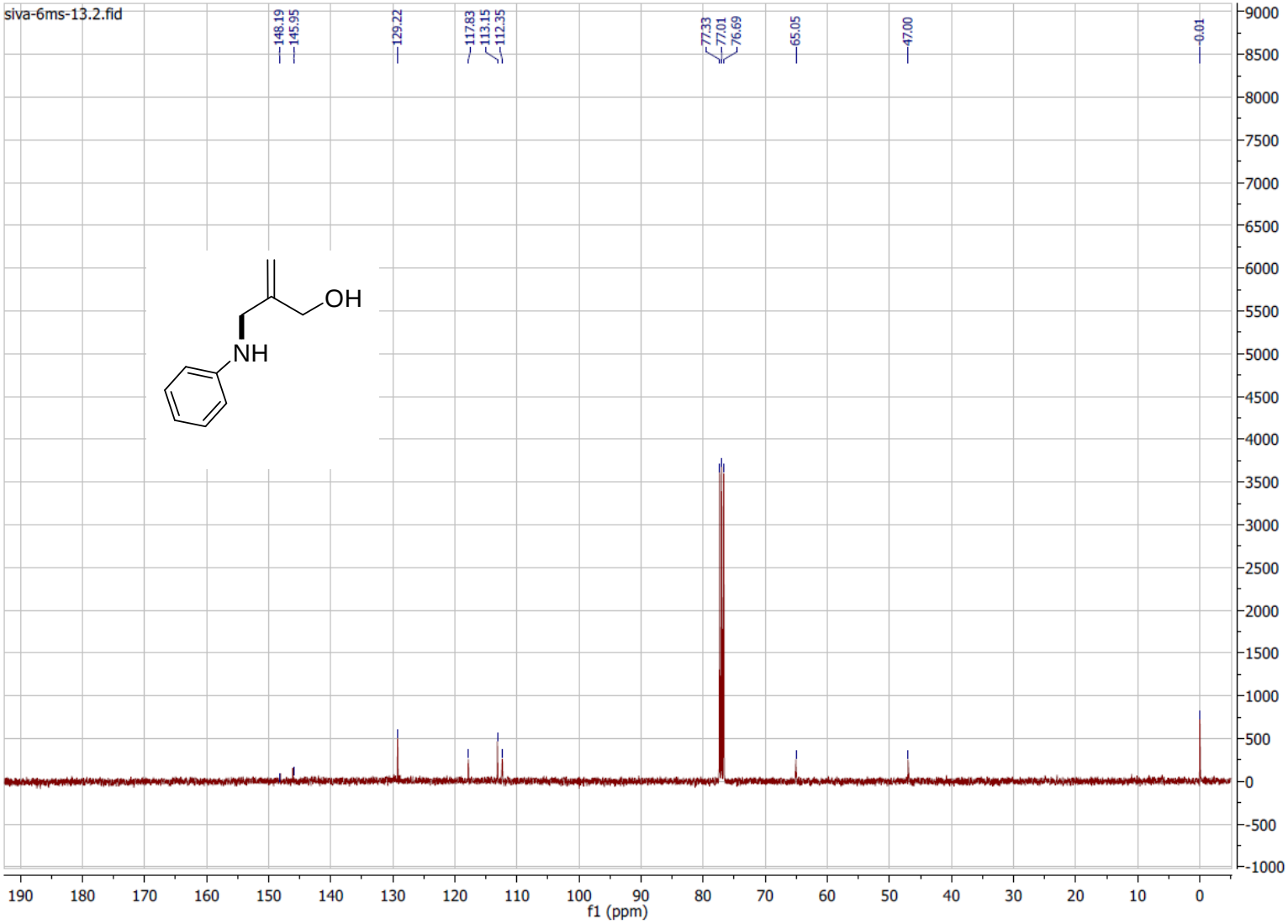
4-(6-methylhepta-1,5-dien-2-yl)-3-phenyloxazolidine (2a): HR-MS analysis



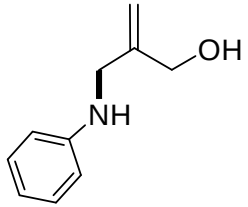
2-Phenylaminomethyl-prop-2-en-1-ol (3a): ^{1}H NMR (400 MHz, $CDCl_3$)

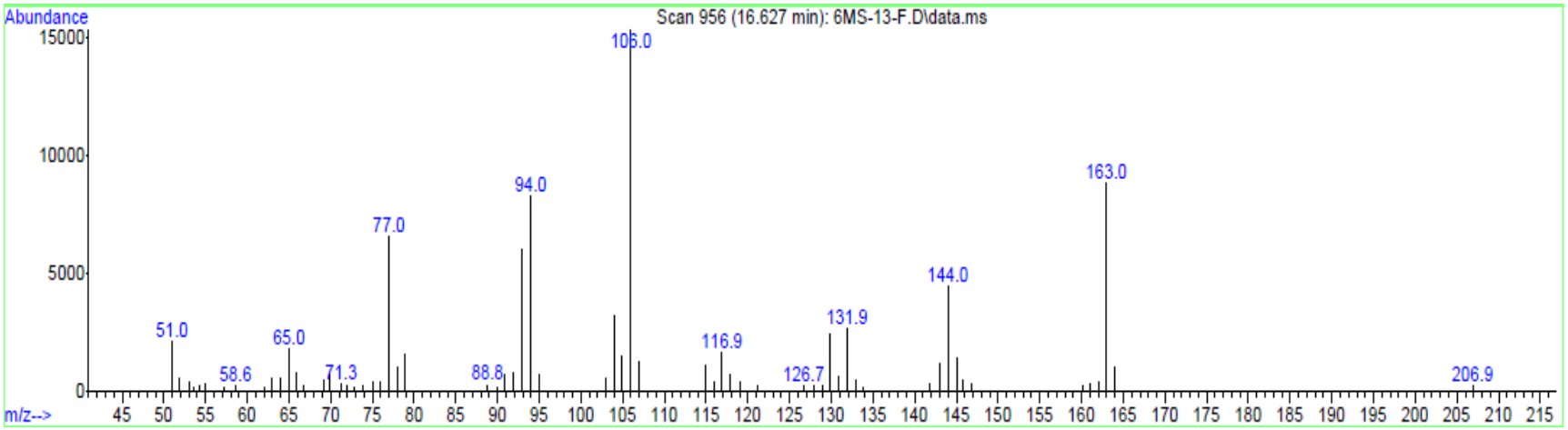


2-Phenylaminomethyl-prop-2-en-1-ol (3a): ^{13}C NMR (100 MHz, $CDCl_3$)

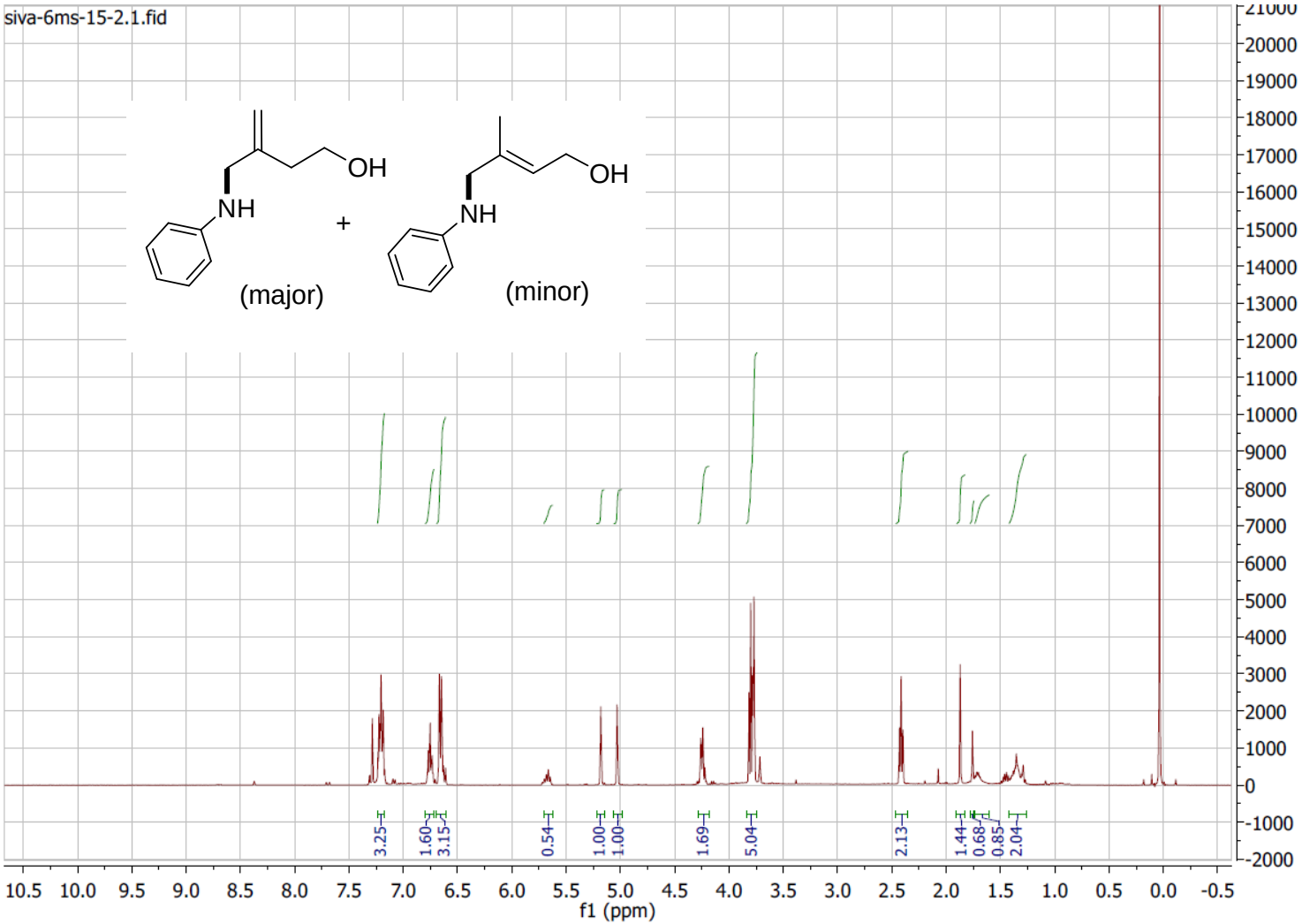


2-Phenylaminomethyl-prop-2-en-1-ol (3a): GC-MS analysis

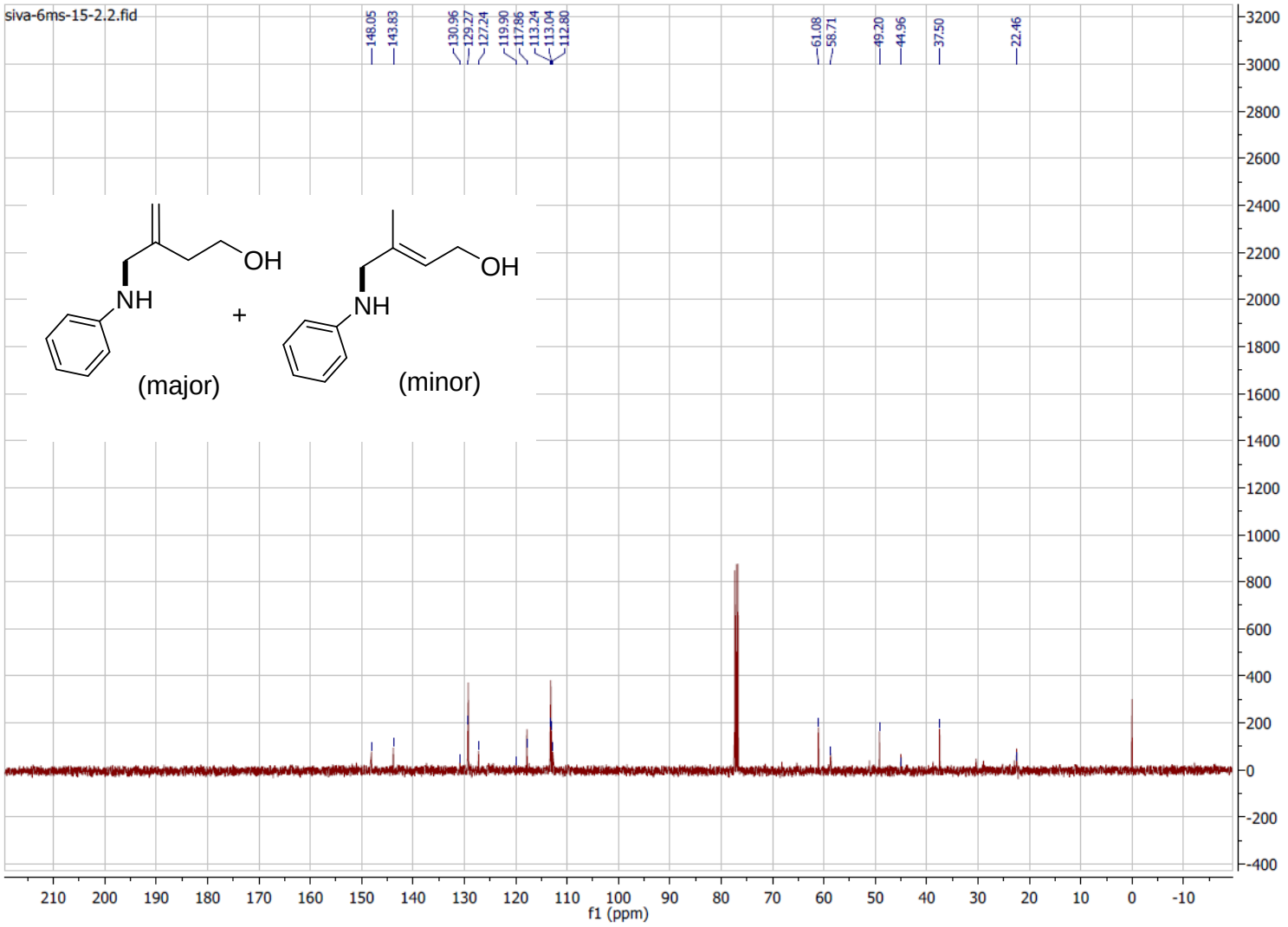




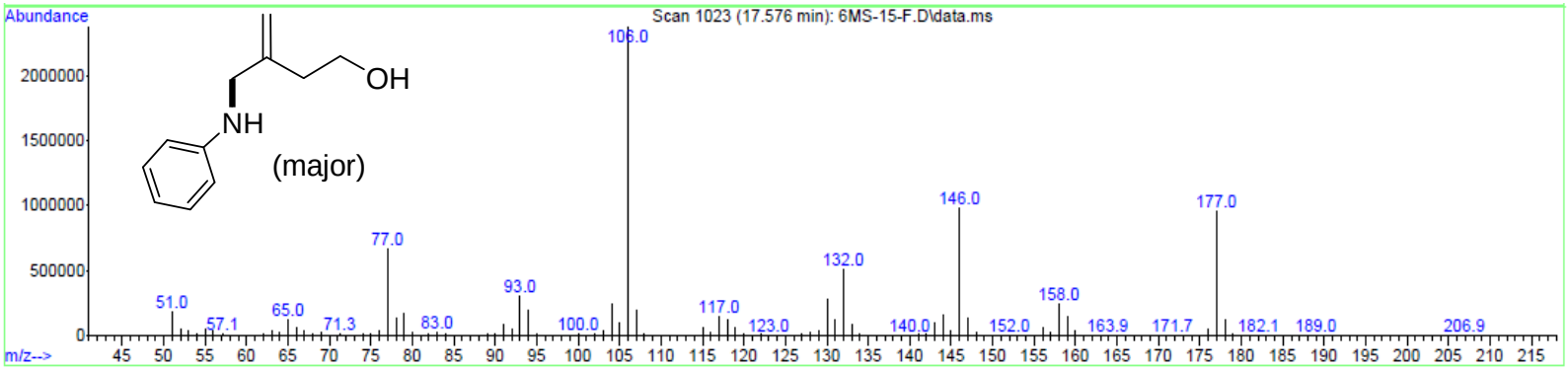
3-Phenylaminomethyl-but-3-en-1-ol and 3-Methyl-4-phenylamino-but-2-en-1-ol (4a+4a'): ^{1}H NMR (400 MHz, $CDCl_3$)

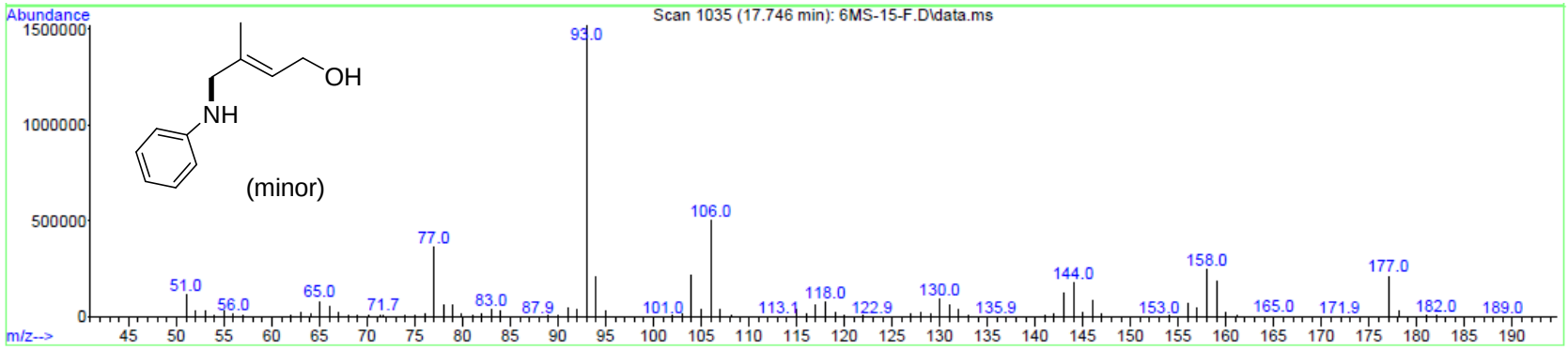


3-Phenylaminomethyl-but-3-en-1-ol and 3-Methyl-4-phenylamino-but-2-en-1-ol (4a+4a'): ^{13}C NMR (100 MHz, $CDCl_3$)

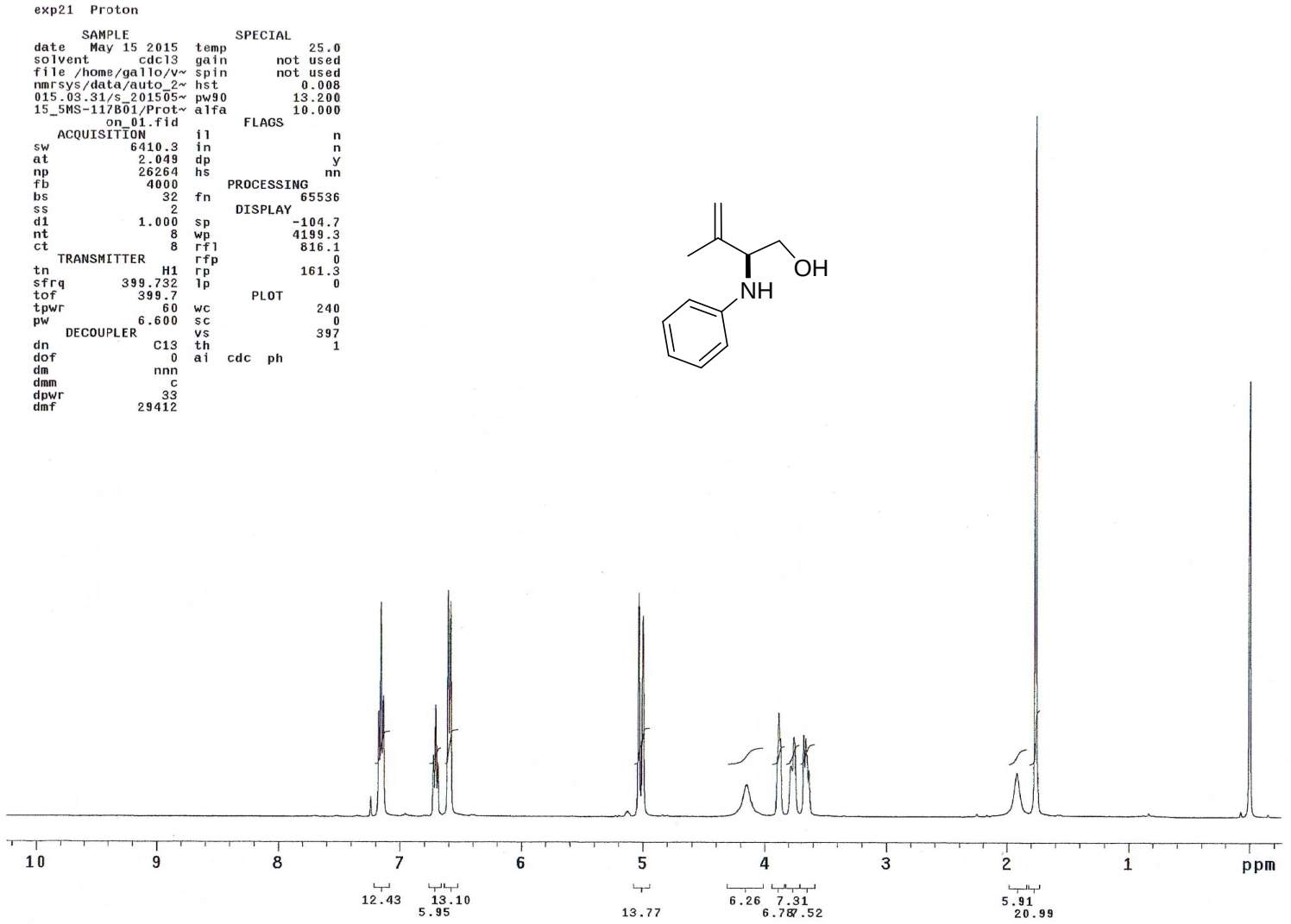


3-Phenylaminomethyl-but-3-en-1-ol and 3-Methyl-4-phenylamino-but-2-en-1-ol (4a+4a'): GC-MS analysis

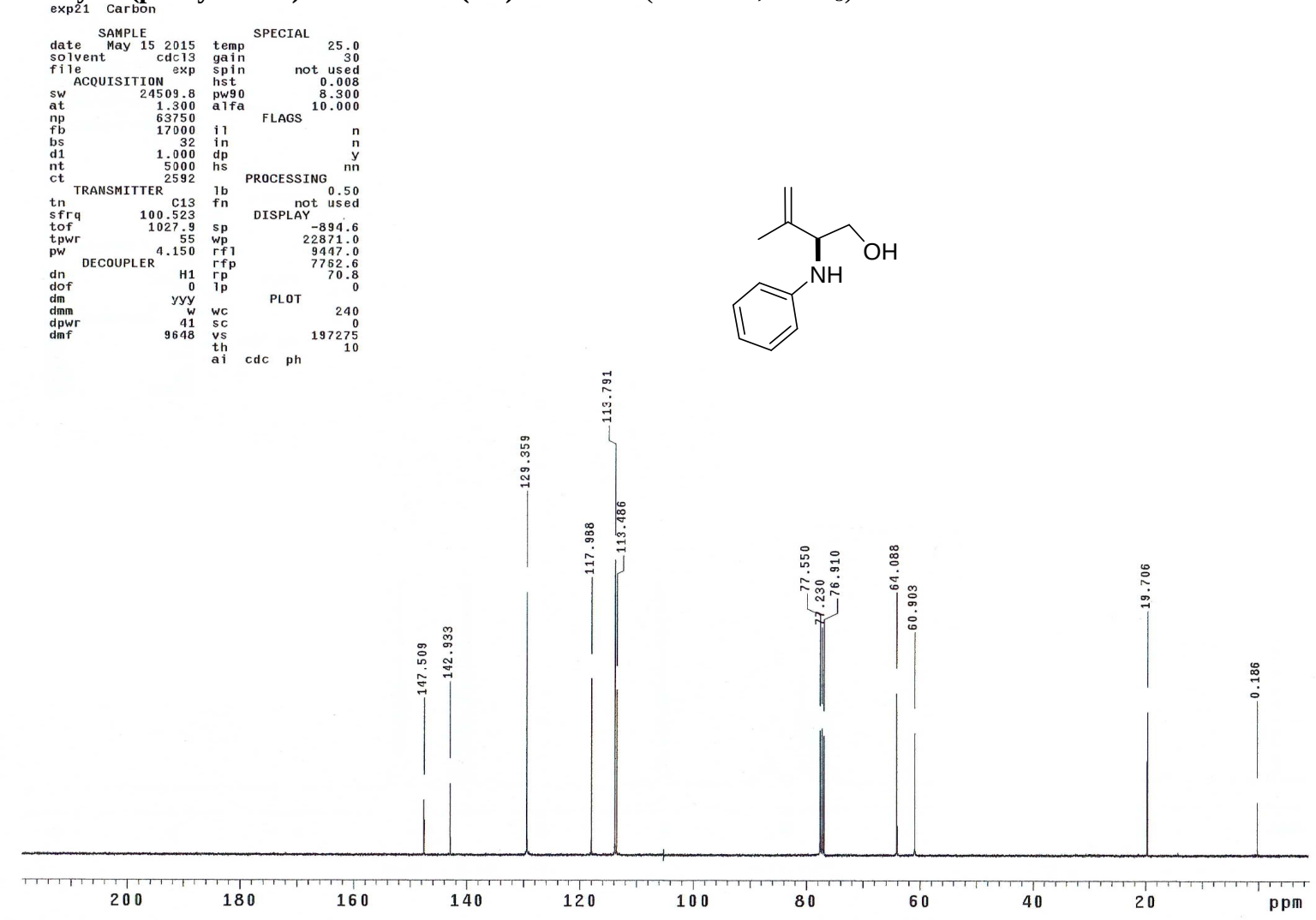




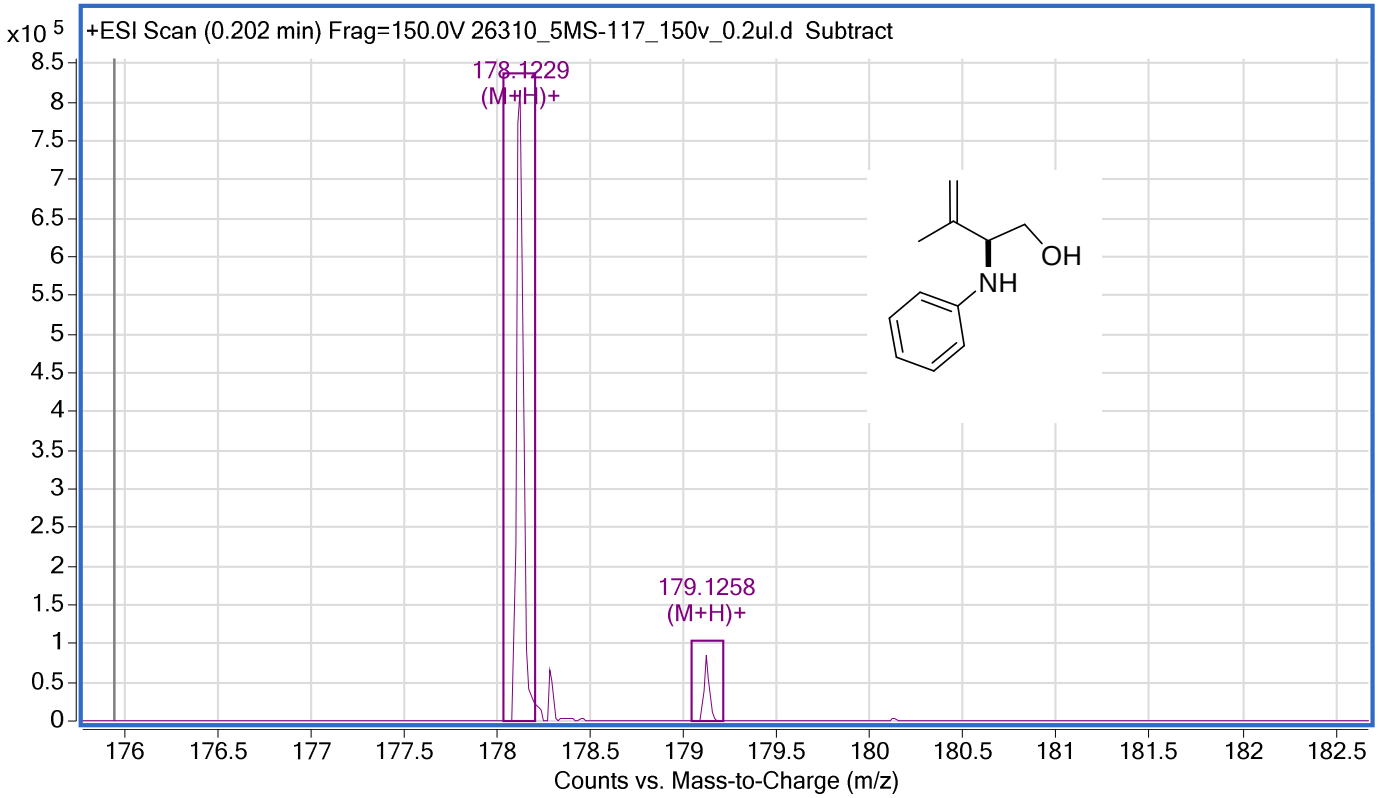
3-methyl-2-(phenylamino)but-3-en-1-ol (1a'): ^{1}H NMR (400 MHz, $CDCl_3$)



3-methyl-2-(phenylamino)but-3-en-1-ol (1a'): ^{13}C NMR (100 MHz, $CDCl_3$)

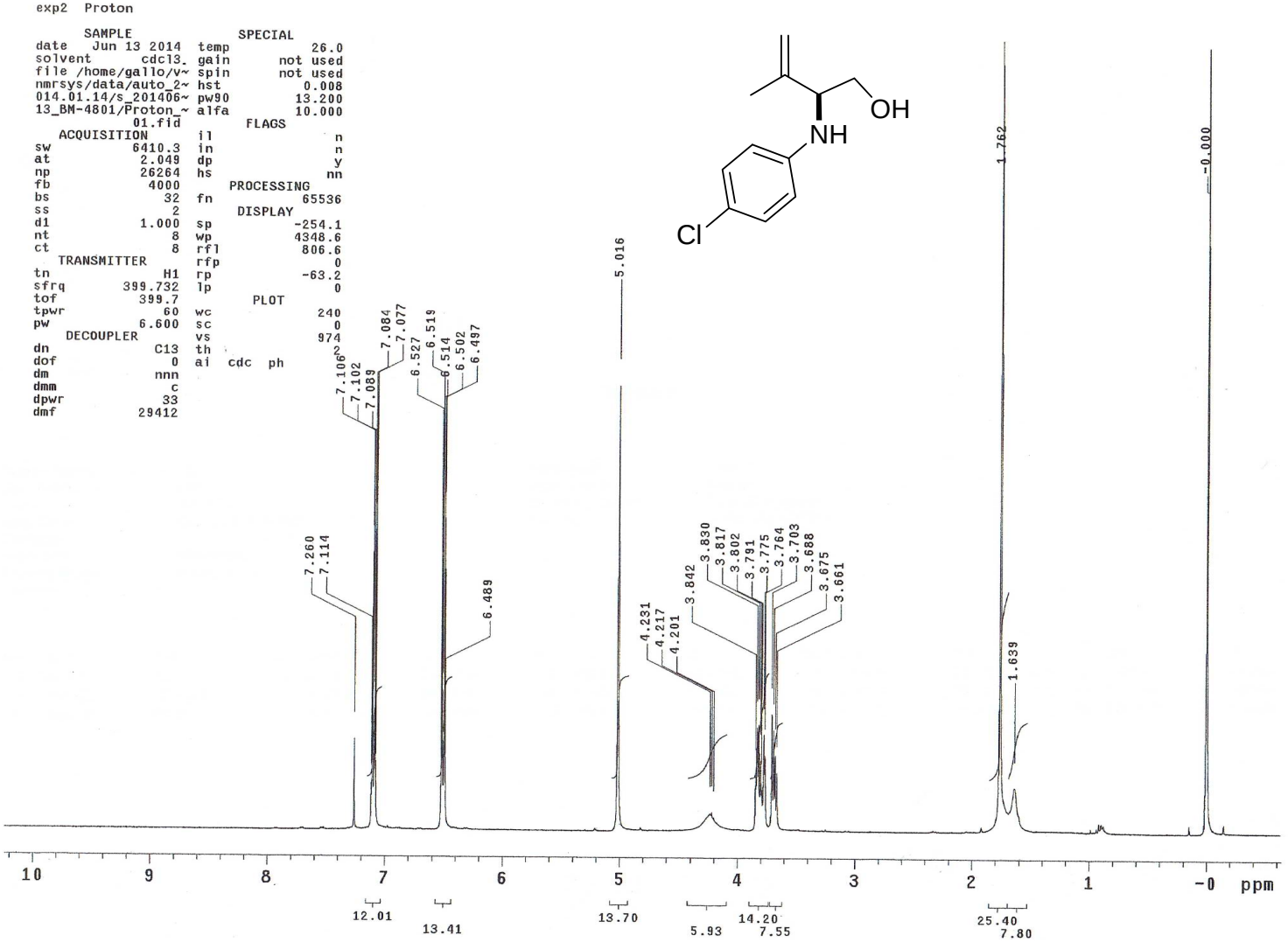


3-methyl-2-(phenylamino)but-3-en-1-ol (1a'): HR-MS analysis

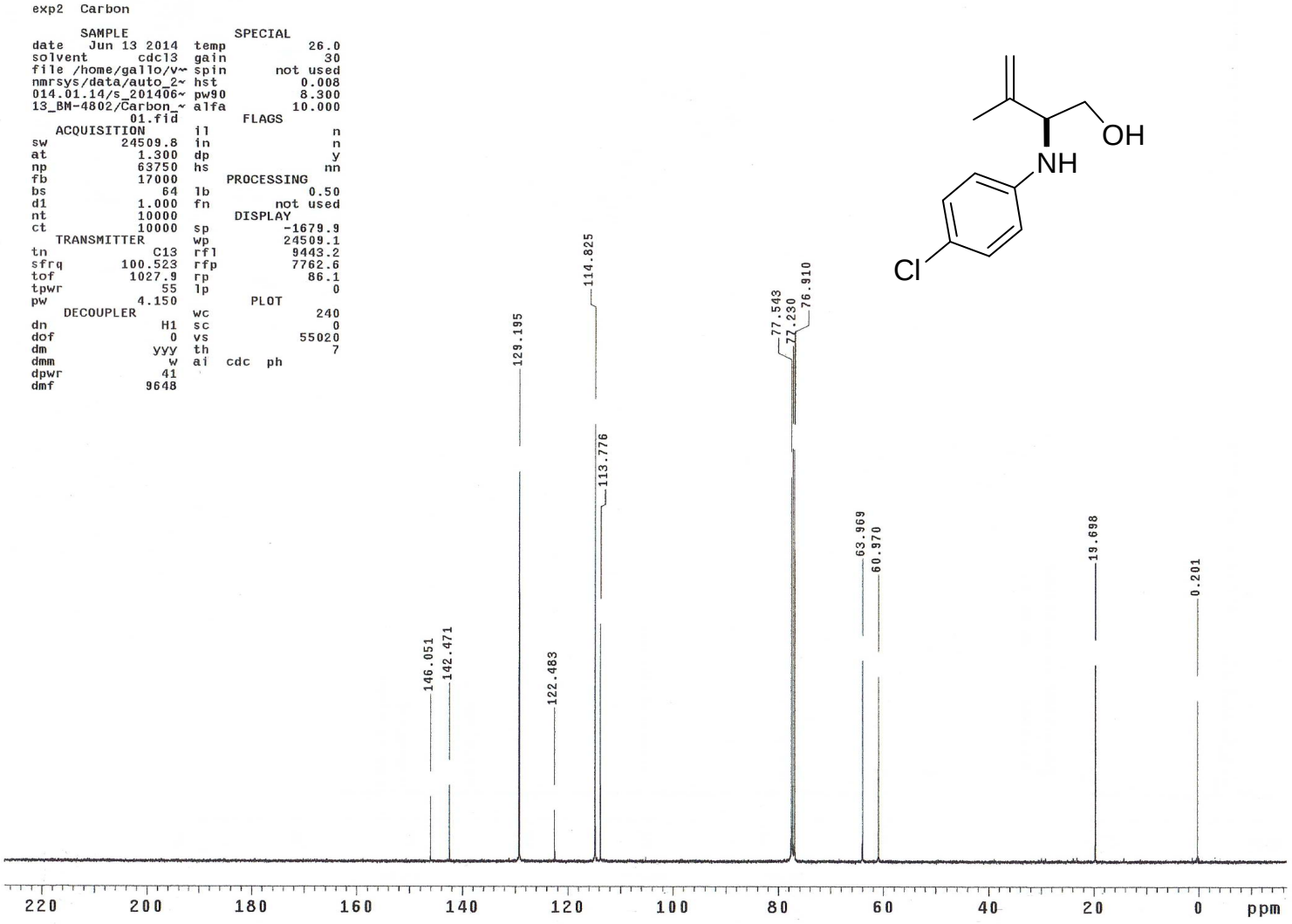


Formula (M),	Ion Formula,	Mass,	Calc Mass,	Calc m/z,	Diff (ppm),	Mass Match
C11 H15 N O,	C11 H16 N O,	177.1156,	177.1154,	178.1226,	1.4,	99.321

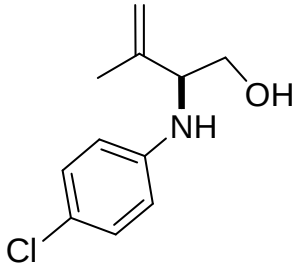
2-(4-chlorophenylamino)-3-methylbut-3-en-1-ol (1c'): ^{1}H NMR (400 MHz, $CDCl_3$)

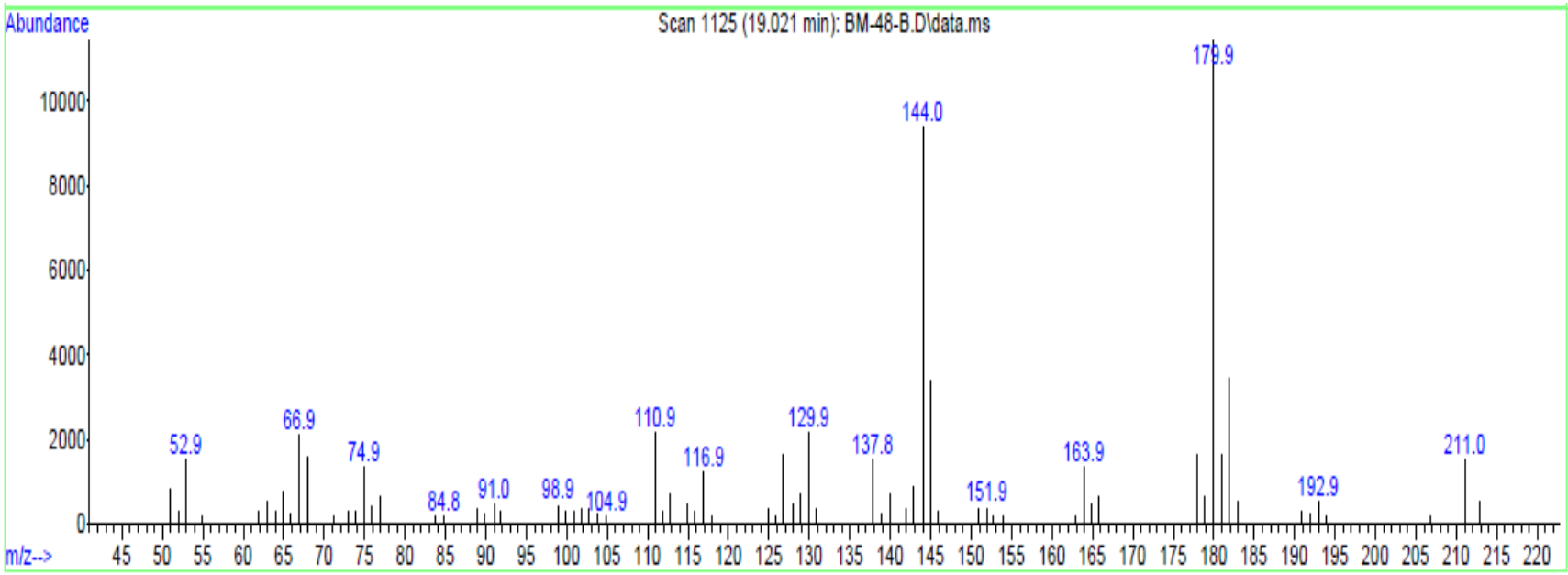


2-(4-chlorophenylamino)-3-methylbut-3-en-1-ol (1c'): ^{13}C NMR (100 MHz, $CDCl_3$)

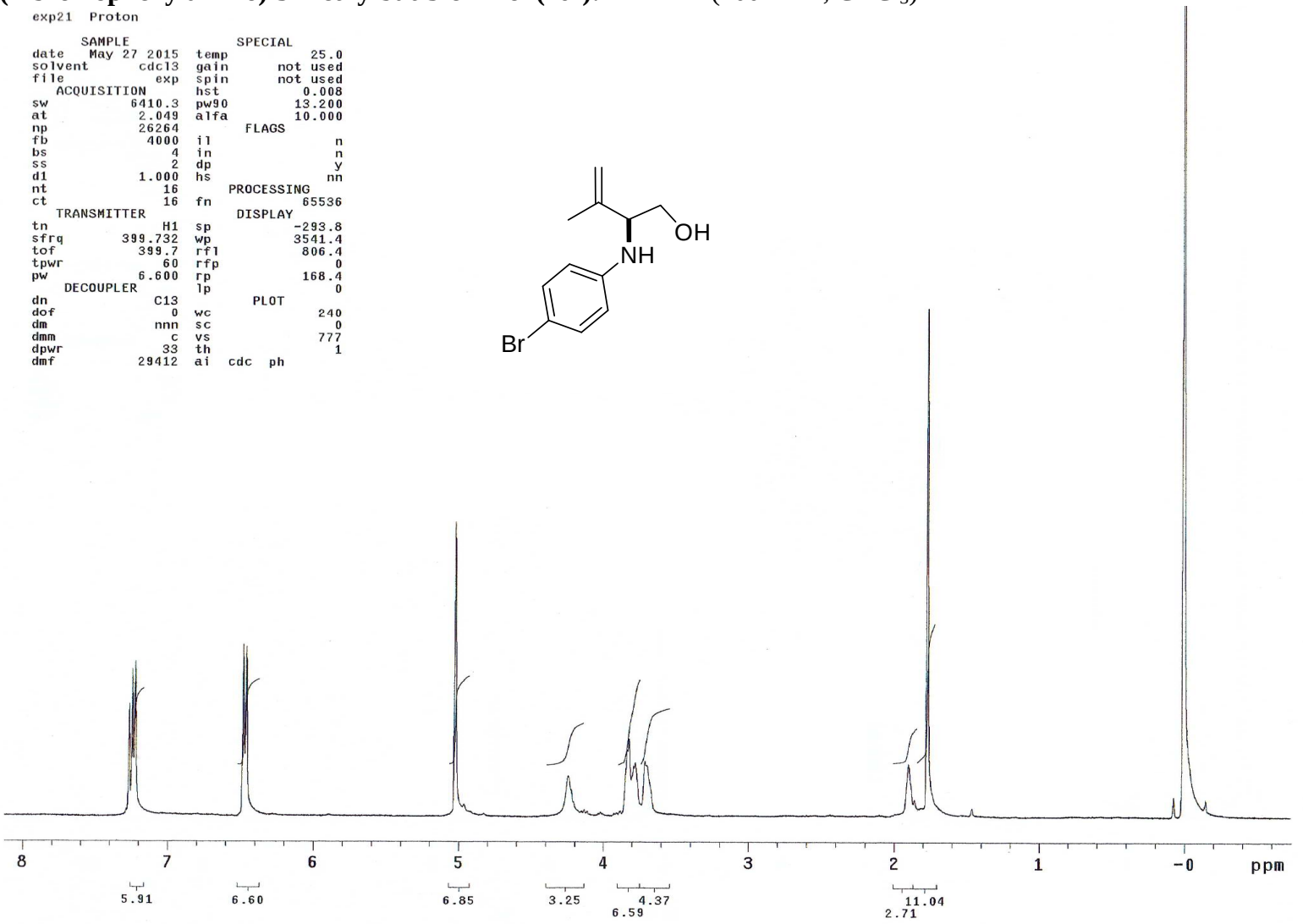


2-(4-chlorophenylamino)-3-methylbut-3-en-1-ol (1c'): GC-MS analysis

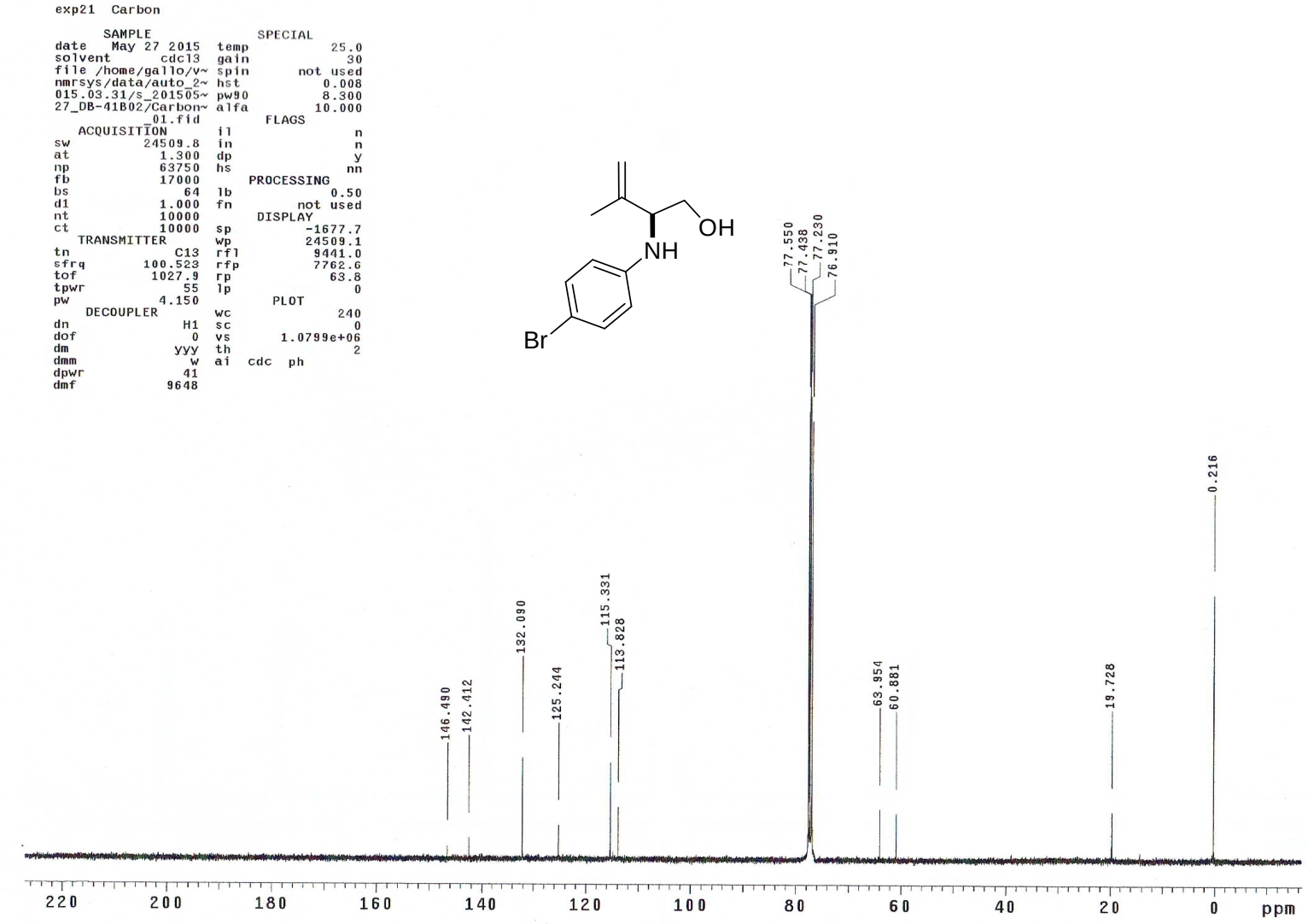




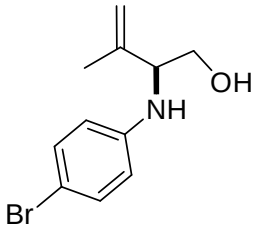
2-(4-bromophenylamino)-3-methylbut-3-en-1-ol (1d'): ^{1}H NMR (400 MHz, $CDCl_3$)

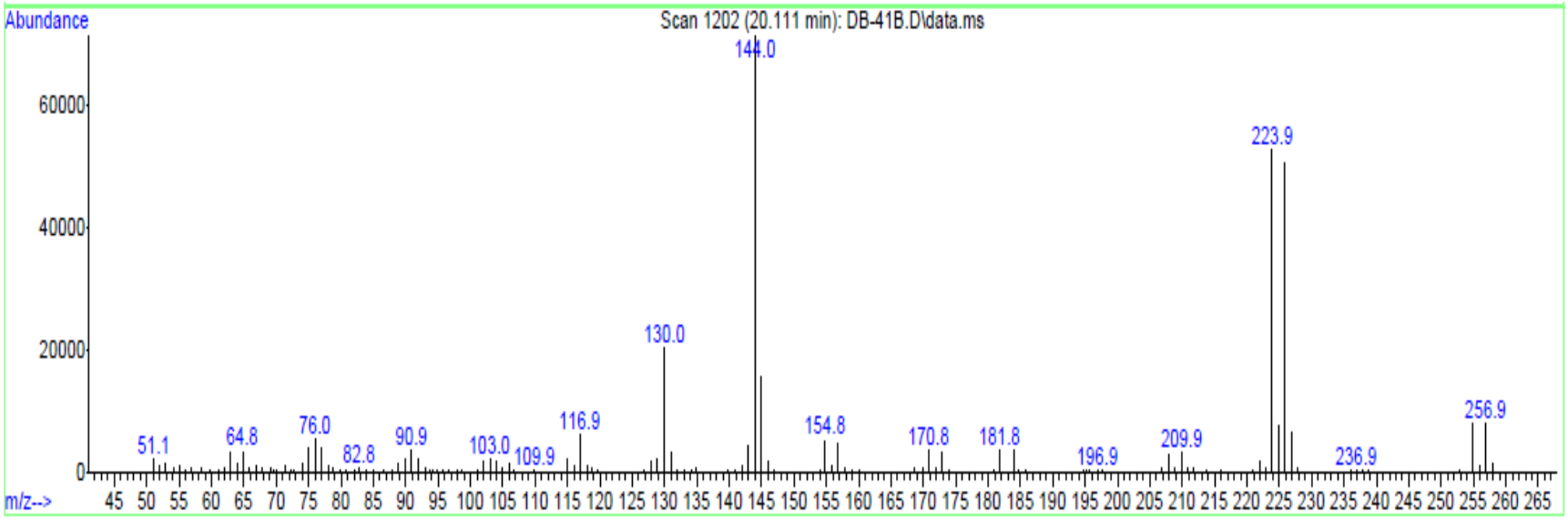


2-(4-bromophenylamino)-3-methylbut-3-en-1-ol (1d'): ^{13}C NMR (100 MHz, $CDCl_3$)

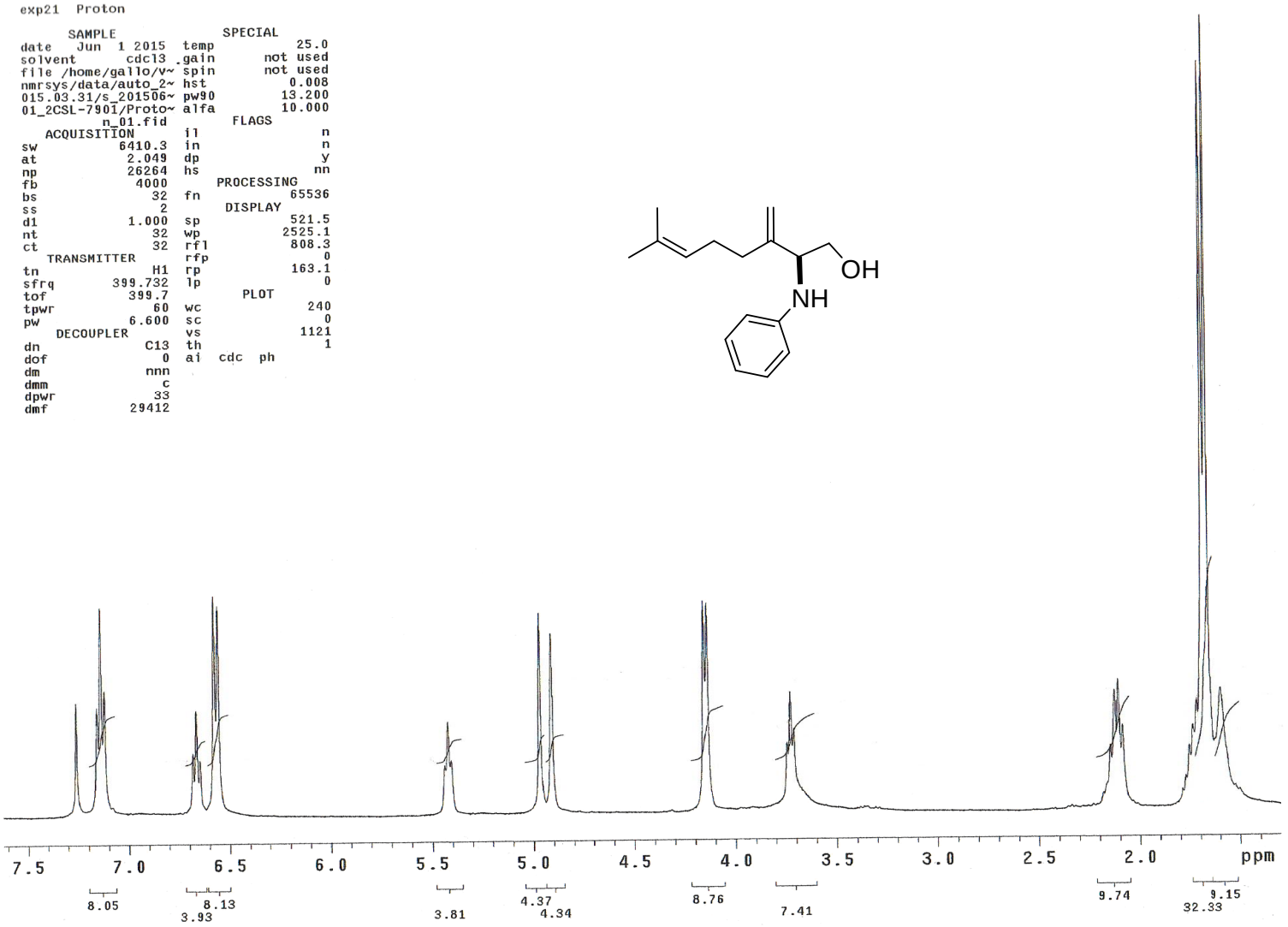


2-(4-bromophenylamino)-3-methylbut-3-en-1-ol (1d'): GC-MS analysis

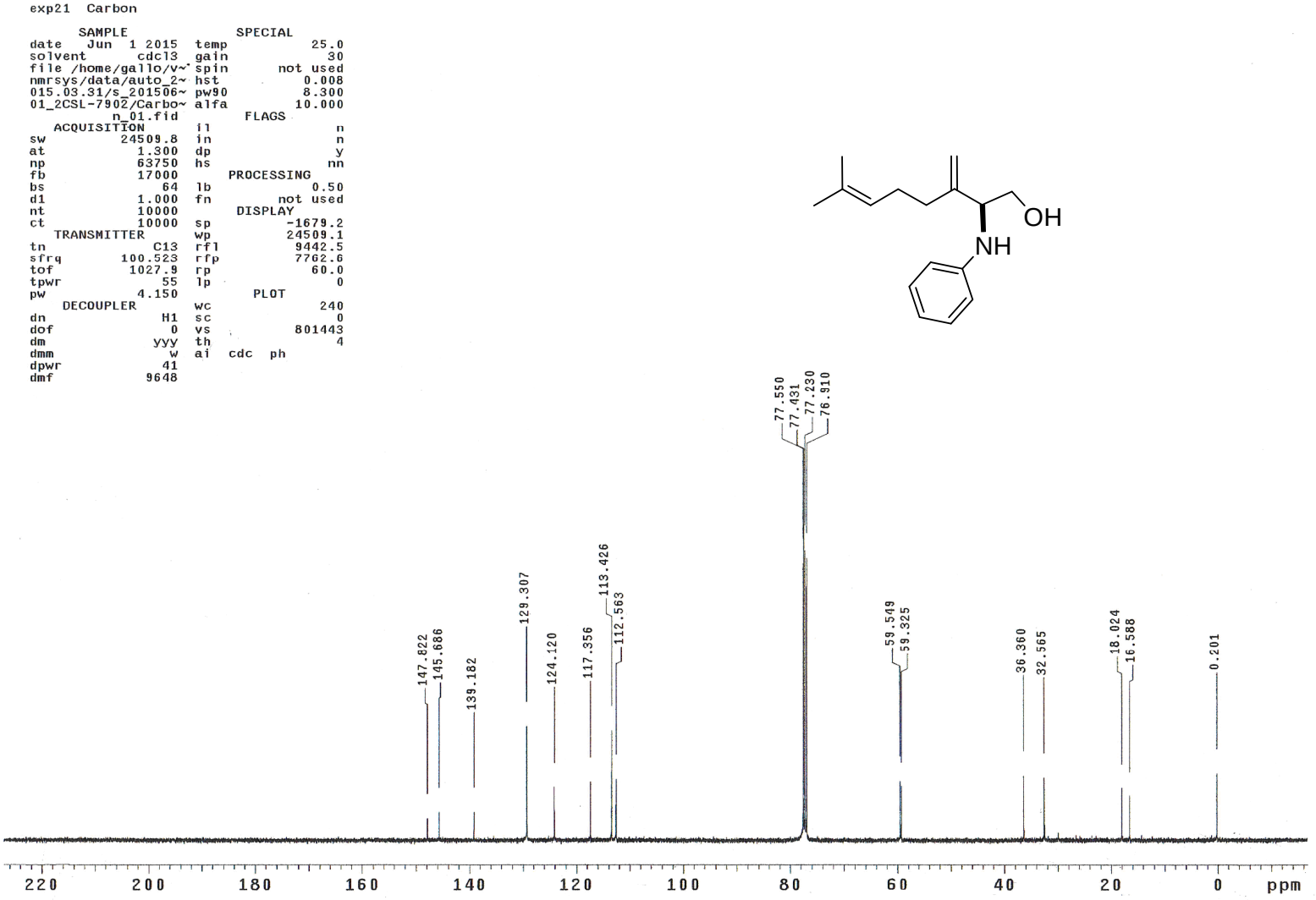




7-methyl-3-methylene-2-(phenylamino)oct-6-en-1-ol (2a'): ^{1}H NMR (400 MHz, $CDCl_3$)



7-methyl-3-methylene-2-(phenylamino)oct-6-en-1-ol (2a'): ^{13}C NMR (100 MHz, $CDCl_3$)



7-methyl-3-methylene-2-(phenylamino)oct-6-en-1-ol (2a'): HR-MS analysis