Electronic Supplementary Material (ESI) for Chemical Communications

Supporting Information:

Gold-catalyzed Diastereoselective [2+2+2]-Cycloaddition of 1,7-Enynes with Carbonyl Compounds

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Contents:

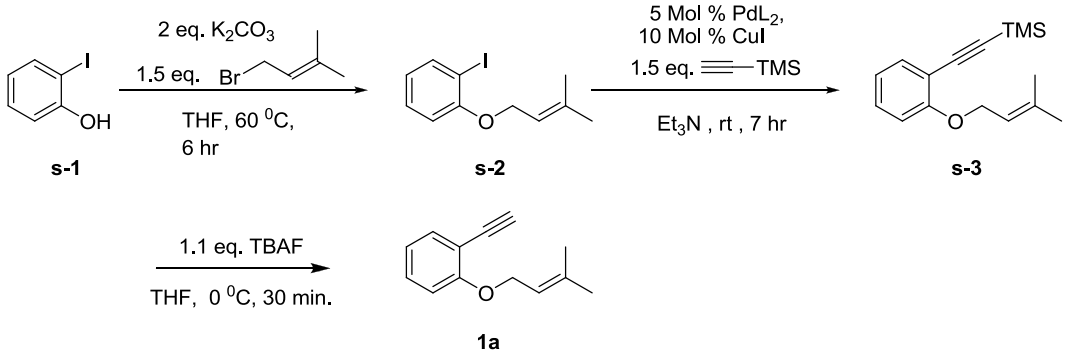
(I) Representative Synthetic Procedures:

(a) General procedure:

Unless otherwise noted, all the reactions for the preparation of the substrates were performed in oven-dried glassware under nitrogen atmosphere with freshly distilled solvents. The catalytic reactions were performed under argon atmosphere. DCM were distilled from CaH_2 under nitrogen. THF was distilled from Na metal under nitrogen. All other commercial reagents were used without further purification, unless otherwise indicated. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Varian 400 MHz, Bruker 400 and 600 MHz Spectrometers using chloroform-d as the internal standards. Aldehydes derivatives & 1-bromo-2-iodobenzene and 2-iodo-phenol was purchased from Sigma Aldrich, Alfa Aesar company.

b) Procedure for the preparation of 1-ethynyl-2-((3-methylbut-2-en-1-yl)oxy)benzene (1a) :

Scheme-s1



(b-1) Synthesis of 1-iodo-2-((3-methylbut-2-en-1-yl)oxy)benzene (s-2):

To a THF solution (20 mL) of 2-iodo-phenol (2.00 g, 9.09 mmol) was added K_2CO_3 (2.50 g, 18.18 mmol) and 1-bromo-3-methylbut-2-ene (1.62 g, 10.9 mmol); the reaction mixture was heated at 60 ^{0}C for 6 h. After cooling to room temperature, the mixture was filtered through a celite bed and extracted by ethyl acetate. The organic layer was washed with water, dried over anhydrous MgSO4 and concentrated under reduced pressure. The residue was chromatographed on a silica column with hexane as the eluent to afford 1-iodo-2-((3-methylbut-2-en-1-yl)oxy)benzene **(s-2)** (2.52 g, 96%) as a colourless liquid.

(b-2) Synthesis of trimethyl((2-((3-methylbut-2-en-1-yl)oxy)phenyl)ethynyl)silane (s-3) :

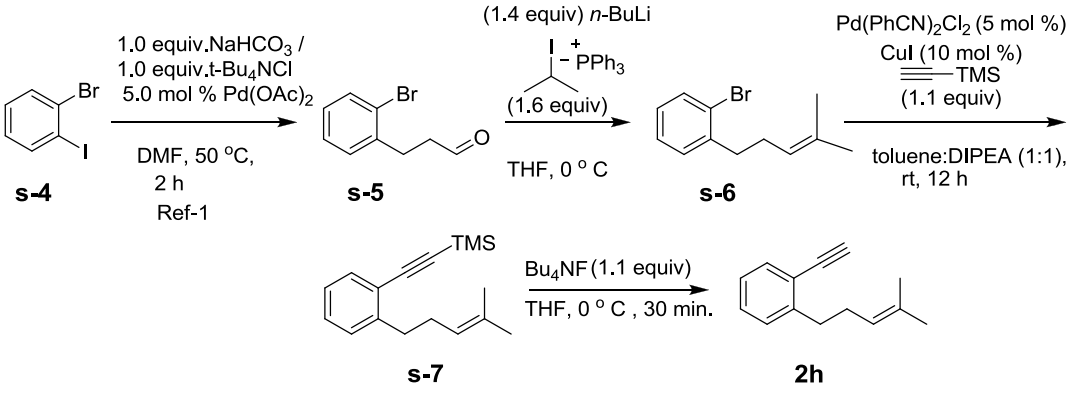
To a Et_3N (40 mL) solution of $PdCl_2(PPh_3)_2$ (243.0 mg, 0.34 mmol), CuI (132.0 mg, 10.7 mmol) and 1-iodo-2-((3-methylbut-2-en-1-yl)oxy)benzene **(s-2)** (2.00 g, 6.94 mmol) was added ethynyltrimethylsilane (1.02 g, 10.14 mmol) slowly; the reaction mixture was stirred at room temperature for 7 h. Ethyl acetate was added to this solution. The mixture was washed with brine, extracted with ethyl acetate, dried by anhydrous Na_2SO_4, and evaporated under vacuum. The residue was chromatographed on a silica column with hexane as an eluent to afford trimethyl((2-((3-methylbut-2-en-1-yl)oxy)phenyl)ethynyl)silane **(s-3)** (1.60 g, 90%) as a colourless oil.

(b-3) Synthesis of 1-ethynyl-2-((3-methylbut-2-en-1-yl)oxy)benzene (1a) :

To a THF solution of trimethyl((2-((3-methylbut-2-en-1-yl)oxy)phenyl)ethynyl)silane compound (**s-3**) (1.60 g, 6.20 mmol); was added Bu_4NF(1.0 M THF, 6.82 mL, 6.82 mmol) at 0 ^oC and the mixture was stirred at 0 °C for 30 min before the addition of water (10 mL). The solution was concentrated, extracted with ethyl acetate, and chromatographed on a silica column to give 1,7 enyne **(1a)** (1.05 g, 87%) as a pale yellow oil.

(c) Procedure for the preparation of 1-ethynyl-2-(4-methylpent-3-en-1-yl)benzene (2h) :

Scheme-s2



(c-1) Synthesis of 3-(2-bromophenyl)propanal (s-5) :

To a suspension, in DMF (20 ml) was added $NaHCO_3$ (1.48 g, 17.6 mmol) and additive tetra-Bu_4NCl (2.28 g, 7.0 mmol) was well stirred for about 20 min. Then 1-bromo-2-iodobenzene (2.00 g, 7.0 mmol) and prop-2-en-1-ol (1.0 ml, 14.0 mmol) was then added. The suspension stirred another 15 min before addition of $Pd(OAc)_2$ (80.0 mg, 7.0 mmol). The reaction mixture was heated at 50 ^oC for 2 h, then cool it to room temperature, filtered through celite bed, concentrated and purified by silica coloumn, to yield the desired product **s-5** (1.20 g, 80%) as colorless oil.

(c-2) Synthesis of 1-bromo-2-(4-methylpent-3-enyl)benzene (s-6):

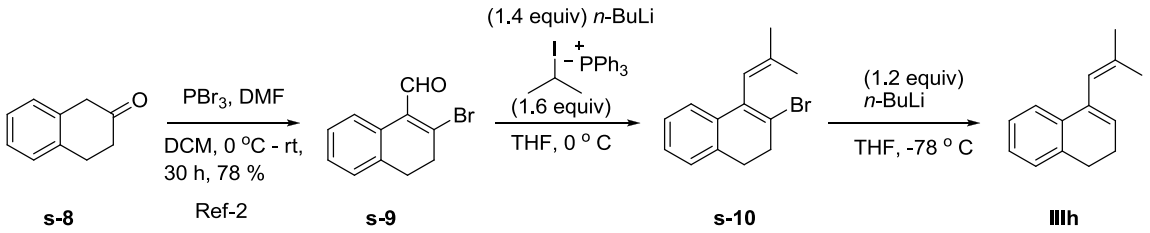
To a dry THF suspension (40 mL) of isopropylphosphonium iodide (9.70 g, 22.50 mmol) under nitrogen atmosphere, was added *n*-BuLi (5.60 ml, 14.0 mmol) slowly at 0 ^oC; the reaction mixture was stirred at 0 ^0C for 30 min before the slow addition of 3-(2-bromophenyl)propanal(**s-3**) (3.00 g, 14.07 mmol). After completion of reaction, the reaction was quenched by water, extracted with ethyl acetate, dry over $MgSO_4$, concentrated and chromatographed on a silica column to give 1,7-enyne **(s-6)** (1.05 g, 87%) as a pale yellow oil.

c-3) Synthesis of trimethyl((2-(4-methylpent-3-enyl)phenyl)ethynyl)silane (s-7) :

To a toluene suspension (10 mL) of $Pd(PhCN)_2Cl_2$ (80.0mg, 0.20 mmol) and CuI (79.0mg, 0.41 mmol) was added P(*t*-Bu)$_3$ (10 % in xylene, 0.41 mmol), HN(*i*-Pr)$_2$ (10 ml), bromo compound **s-6** (1.0g, 4.18 mmol), and trimethylsilyl acetylene (0.61g, 6.21 mmol); the resulting mixtures were stirred at room temperature for 12 h. The resulting black mixture was filtered through a small celite pad, concentrated, and purified by flash chromatographed, to yield the desired product **s-7** (0.81 g, 84%) as colorless oil. Desilylation of **s-7** follow the similar procedure of **b-3** to afford 1-ethynyl-2-(4-methylpent-3-enyl)benzene **2h,** (0.50 g, 89 %) as colourless oil.

d) Procedure for the preparation of 4-(2-methylprop-1-enyl)-1,2-dihydronaphthalene (IIIh) :

Scheme-s3

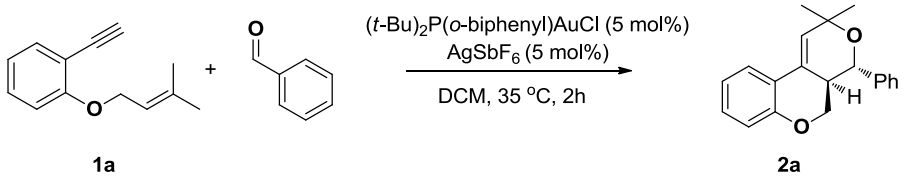


To a dichloromethane solution (60 mL) of DMF (7.5 mL, 102.6 mmol) was added PBr_3 (8.77 mL, 92.34 mmol) at 0 ^oC slowly; the resulting solution was stirred for 45 min before the addition of β-tetralone **s-8** (5.0 g, 34.20 mmol). The reaction mixture was stirred for 20 h at room temperature. To this solution was added cold water, saturated $NaHCO_3$, and then extracted with dichloromethane; the extracts were washed with saturated NaCl solution, dried over anhydrous $MgSO_4$, and concentrated under reduced pressure. The residue was purified by elution through a short silica column to afford 2-bromo-3,4-dihydronaphthalene-1-carbaldehyde **s-9** (6.50 g, 80 %). The synthesis of **s-10** follow similar procedure of compounds **c-2 to** afford 3-bromo-4-(2-methylprop-1-enyl)-1,2-dihydronaphthalene **s-10,** (2.40 g, 80 %).

To a THF solution (30 ml) of compound **s-10** (2.0 g, 10.85 mmol) was added *n*-BuLi (5.20 ml, 13.02 mmol) at -78 ^oC; the reaction mixture was stirred at 0 ^oC for 30 min before it was quenched by water. The organic layer was extracted with ethyl acetate. The ether extracts were washed with saturated NaCl solution, dried over anhydrous $MgSO_4$, and concentrated under reduced pressure. The residues were chromatographed on a short silica column to afford 4-(2-methylprop-1-enyl)-1,2-dihydronaphthalene (**IIIh**) (1.20 g, 86 % as pale yellow oil.

(II) General Procedure for Gold-Catalyzed Diastereoselective [2+2+2]-Cycloaddition of 1,7-Enynes with Corbonyl Compound.

(a) Standard Procedure for catalytic operations



1a **2a**

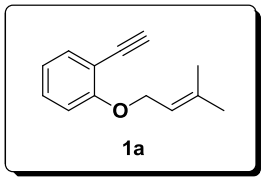
Under argon atmosphere, A two-necked flask was charged with AuCl(*t*-Bu)$_2$P(*o*-biphenyl) (14.0 mg, 0.02 mmol) and silver hexafluoroantimonate (9.0 mg, 0.02 mmol), and to this mixture was added dry dichloromethane (1.0 ml). The resulting solution was stirred at room temperature for 5 min before it was added slowly to a dry dichloromethane solution (2 mL) of compound **1a** (100.0 mg, 0.53 mmol) and benzaldehyde (113.0 mg, 1.07 mmol) through a syringe pump over a 2 h period. At the end of addition, the resulting mixture was filtered over a short silica bed, concentrated under reduce pressure, and eluted through a silica column to give the desired (4S,4aR)-2,2-dimethyl-4-phenyl-2,4,4a,5-tetrahydropyrano[3,4-c]chromene (**2a)** (133.0 mg, 85%) as white crystal solid.

References.

1. T. Jeffery, *Tetrahedron,* 1996, **52,** 10113-10130
2. S. Bhunia, S.Ghorpade, D. B. Huple, R.S. Liu, *Angew.Chem, Int. ed, 2012,* **51,** 2939 - 2942

(III) Spectral data:

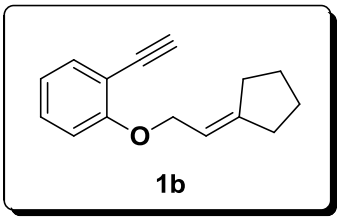
Spectral data for 1-ethynyl-2-((3-methylbut-2-en-1-yl)oxy)benzene (1a) :



1a

Pale yellow oil, IR (neat, cm^{-1}): 3060 (m), 2952 (s), 2160 (s), 1625 (s), 1386 (m), 1271 (s), 1170 (s), 685 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41 (d, *J* = 7.6 Hz, 1 H), 7.26~ 7.22 (m, 1 H), 6.87~ 6.82 (m, 2 H), 5.47 ~ 5.44 (m, 1 H), 4.58 (d, *J* = 6.4 Hz, 2 H), 3.24 (s, 1 H), 1.73 (s, 3 H), 1.70 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.8, 137.3, 134.0, 129.9, 120.2, 119.6, 112.1, 111.5, 81.0, 80.0, 65.5, 25.6, 18.1; HRMS calcd. for Chemical Formula: $C_{13}H_{14}O$, Exact Mass: 186.1045, found: 186.1031.

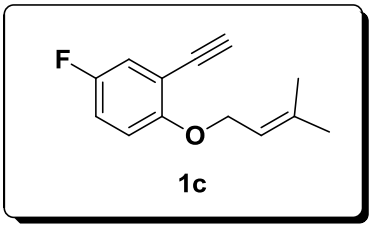
Spectral data for 1-(2-cyclopentylideneethoxy)-2-ethynylbenzene (1b) :



1b

Colourless oil, IR (neat, cm^{-1}): 3064 (m), 2960 (s), 2145 (s), 1655 (s), 1310 (m), 1275 (s), 1170 (s), 694 (s); ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.44 (dd, *J* = 7.5, 1.7 Hz, 1 H), 7.28~ 7.25 (m, 1 H), 6.88~ 6.85 (m, 2 H), 5.61 ~ 5.58 (m, 1 H), 4.58 (d, *J* = 6.4 Hz, 2 H), 3.26 (s, 1 H), 2.31 ~ 2.28 (m, 4 H), 1.70 ~ 1.60 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.0, 148.8, 134.1, 130.0, 120.3, 115.1, 112.4, 112.1, 81.0, 80.2, 67.1, 33.8, 29.0, 26.0 (2X CH_2); HRMS calcd. for Chemical Formula: $C_{15}H_{16}O$, Exact Mass: 212.1201, found: 212.1202.

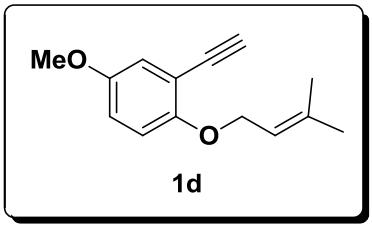
Spectral data for 2-ethynyl-4-fluoro-1-((3-methylbut-2-en-1-yl)oxy)benzene (1c) :



1c

Brown oil, IR (neat, cm^{-1}): 3055 (m), 2959 (s), 2155 (s), 1665 (s), 1320 (m), 1280 (s), 1161 (s), 689 (s); ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.11 (dd, *J* = 8.5, 3.2 Hz, 1 H), 6.96 ~ 6.92 (m, 1 H), 6.77 (dd, *J* = 9.1, 4.5 Hz, 1 H), 5.46~ 5.43 (m, 1 H), 4.55 (d, *J* = 6.6 Hz, 2 H), 3.28 (s, 1 H), 1.74 (s, 3 H), 1.70 (s, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 156.6 (d, J_{CF} = 386.4 Hz), 155.3, 137.8, 120.7 (d, J_{CF} = 24.1 Hz), 119.5, 119.4, 116.5 (d, J_{CF} = 22.8 Hz), 113.9 (d, J_{CF} = 8.0 Hz), 81.9, 79.0, 66.6, 25.6, 18.8; HRMS calcd. for Chemical Formula: $C_{13}H_{13}OF$, Exact Mass: 204.0950, found: 204.0944.

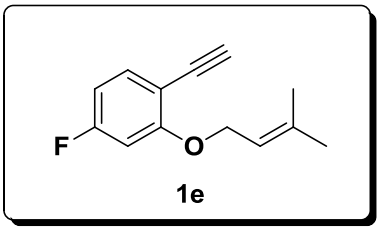
Spectral data for 2-ethynyl-4-methoxy-1-((3-methylbut-2-en-1-yl)oxy)benzene (1d) :



1d

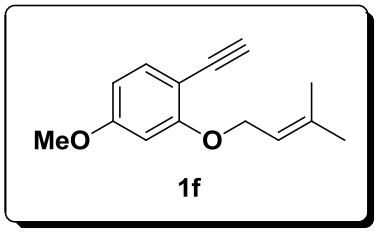
Pale Yellow oil, IR (neat, cm^{-1}): 3061 (m), 2955 (s), 2152 (s), 1661 (s), 1316 (m), 1270 (s), 1162 (s), 693 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.97 (d, *J* = 2.8 Hz, 1 H), 6.83 ~ 6.78 (m, 2 H), 5.48~ 5.44 (m, 1 H), 4.54 (d, *J* = 6.5 Hz, 2 H), 3.72 (s, 3 H), 3.25 (s, 1 H), 1.74 (s, 3 H), 1.70 (s, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 154.3, 153.1, 137.3, 120.0, 118.5, 116.1, 114.5, 112.6, 81.0, 80.0, 66.7, 55.6, 25.7, 18.10; HRMS calcd. for Chemical Formula: $C_{14}H_{16}O_2$, Exact Mass: 216.1150, found: 216.1141.

Spectral data for 1-ethynyl-4-fluoro-2-((3-methylbut-2-en-1-yl)oxy)benzene (1e) :



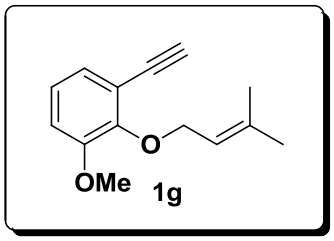
Brown oil, IR (neat, cm^{-1}): 3045 (m), 2945 (s), 2180 (s), 1650 (s), 1324 (m), 1160 (s), 710 (s); ^1H NMR (400 MHz, $CDCl_3$): δ7.40 ~ 738 (m, 1 H), 6.68 ~ 6.56 (m, 2 H), 5.47 ~ 5.44 (m, 1 H), 4.51 (d, J = 6.5 Hz, 2 H), 3.22 (s, 1 H), 1.77 (s, 3 H), 1.72 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.5 (d, J_{CF} = 247.3 Hz), 161.2 (d, J_{CF} = 10.3 Hz), 138.4, 135.0 (d, J_{CF} = 10.2 Hz), 119.2, 119.0, 107.3 (d, J_{CF} = 8.9 Hz), 100.5 (d, J_{CF} = 26.0 Hz), 80.6, 79.2, 65.9, 25.6, 18.2; HRMS calcd. for Chemical Formula: $C_{13}H_{13}OF$, Exact Mass: 204.0950, found: 204.0951.

Spectral data for 1-ethynyl-4-methoxy-2-((3-methylbut-2-en-1-yl)oxy)benzene (1f) :



Pale Yellow oil, IR (neat, cm^{-1}): 3065 (m), 2956 (s), 2154 (s), 1656 (s), 1312 (m), 1272 (s), 1164 (s), 691 (s); ^1H NMR (600 MHz, $CDCl_3$): δ 7.35 ~ 7.34 (m, 1 H), 6.42 ~ 6.40 (m, 2 H), 5.48~ 5.45(m, 1 H), 4.57 (d, J = 6.4 Hz, 2 H), 3.79 (s, 3 H), 3.18 (s, 1 H), 1.75 (s, 3 H), 1.72 (s, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 161.2, 161.1, 137.4, 134.8, 119.6, 104.9, 104.2, 99.7, 80.2, 79.5, 65.8, 55.3, 25.7, 18.2; HRMS calcd. for Chemical Formula: $C_{14}H_{16}O_2$, Exact Mass: 216.1150, found: 216.1141.

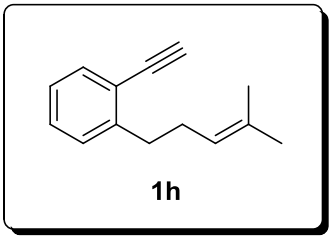
Spectral data for 1-ethynyl-3-methoxy-2-((3-methylbut-2-en-1-yl)oxy)benzene (1g):



1g

Colourless oil, IR (neat, cm^{-1}): 3059 (m), 2953 (s), 2149 (s), 1658 (s), 1314 (m), 1265 (s), 1160 (s), 694 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.00 (dd, *J* = 6.4, 1.5 Hz, 1 H), 6.95 ~ 6.91 (m, 1 H), 6.87~ 6.84 (m, 1 H), 5.60 ~ 5.55 (m, 1 H), 4.58 (d, *J* = 7.1 Hz, 2 H), 3.81 (s, 3 H), 3.21 (s, 1 H), 1.72 (s, 3 H), 1.65 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 152.9, 149.7, 138.1, 125.2, 123.6, 120.3, 117.4, 113.1, 80.8, 79.9, 69.7, 55.7, 25.6, 17.7; HRMS calcd. for Chemical Formula: $C_{14}H_{16}O_2$, Exact Mass: 216.1150, found: 216.1133.

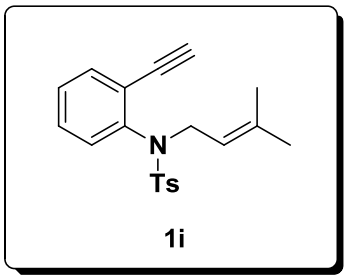
Spectral data for 1-ethynyl-2-(4-methylpent-3-en-1-yl)benzene (1h) :



1h

Colourless oil, IR (neat, cm^{-1}): 3040 (m), 2930 (s), 2145 (s), 1625 (s), 1320 (m), 1270 (s), 1160 (s), 690 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.45 (d, *J* = 7.5 Hz, 1 H), 7.29 ~ 7.25 (m, 1 H), 7.21~ 7.813 (m, 2 H), 5.24 ~ 5.21 (m, 1 H), 3.25 (s, 1 H), 2.83 (t, *J* = 7.4 Hz, 2H), 2.34 (q, *J* = 7.4 Hz, 2 H), 1.71 (s, 3 H), 1.58 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.9, 132.8, 132.3, 128.8, 128.7, 125.6, 123.6, 121.5, 82.4, 80.5, 34.8, 29.2, 25.7, 17.5; HRMS calcd. for Chemical Formula: $C_{14}H_{16,}$ Exact Mass: 184.1252, found: 184.1250.

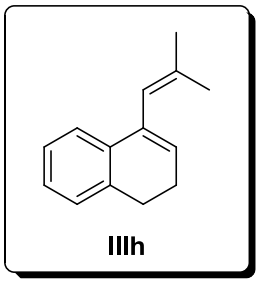
Spectral data for N-(2-ethynylphenyl)-4-methyl-N-(3-methylbut-2-en-1-yl)benzenesulfonamide (1i) :



1i

Yellow oil, IR (neat, cm^{-1}): 3035 (m), 2934 (s), 2146 (s), 1628 (s), 1322 (m), 1268 (s), 1162 (s), 686 (s); ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.61 (d, *J* = 8.2 Hz, 2 H), 7.42 ~ 7.41 (m, 1 H), 7.26~ 7.21 (m, 4 H), 7.10 ~ 7.00 (m, 1 H), 5.15 ~ 5.12 (m, 1 H), 4.23 (d, *J* = 5.9 Hz, 2 H), 2.96 (s, 1 H), 2.38 (s, 3 H), 1.54 (s, 3 H), 1.35 (s, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 143.1, 140.6, 137.2, 137.0, 133.9, 131.4, 129.3, 129.0, 127.8, 127.7, 123.4, 118.7, 81.6, 80.2, 48.2, 25.5, 21.4, 17.4; HRMS calcd. for Chemical Formula: $C_{20}H_{21}NO_2S$, Exact Mass: 339.1293,found: 339.1277.

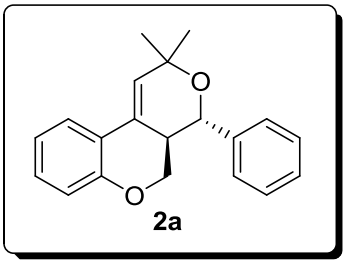
Spectral data for 4-(2-methylprop-1-en-1-yl)-1,2-dihydronaphthalene (IIIh):



IIIh

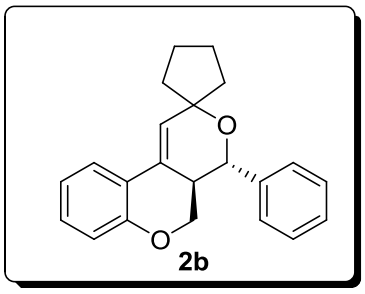
Pale yellow oil, IR (neat, cm^{-1}): 3035 (m), 2934 (s), 1628 (s), 1322 (m), 1268 (s), 686 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.27 ~ 7.19 (m, 4 H), 6.03 ~ 6.02 (m, 1 H), 5.94 ~ 5.92 (m, 1 H), 2.85 (t, *J* = 7.6 Hz, 2 H), 2.44 ~ 2.38 (m, 2 H), 1.97 (s, 3 H), 1.81 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 136.1, 136.0, 135.3, 134.9, 127.3, 127.0, 126.6, 126.2, 124.1, 123.0, 28.1, 25.9, 23.1, 19.5; HRMS calcd. for Chemical Formula: $C_{14}H_{16,}$ Exact Mass: 184.1252, found: 184.1255.

Spectral data for (4S,4aR)-2,2-dimethyl-4-phenyl-2,4,4a,5-tetrahydropyrano[3,4-c]chromene (2a) :



White Solid, m. p. 116.0-117.0 ^{o}C IR (KBr, cm^{-1}): 3022 (m), 2910 (s), 1625 (s), 1268 (s), 1162 (s), 901 (s), 686 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52 (dd, *J* = 7.8, 1.2 Hz, 1 H), 7.38 ~ 7.30 (m, 5 H), 7.14~ 7.10 (m, 1 H), 6.93 ~ 6.89 (m, 1 H), 6.79 (d, *J* = 8.2 Hz, 1 H), 6.19 (d, *J* = 2.2 Hz, 1 H), 4.26 (d, *J* = 9.4 Hz, 1 H), 3.92 (dd, *J* = 10.5, 4.8 Hz, 1 H), 3.65 (dd, *J* = 12.2, 10.5 Hz, 1 H), 2.88 ~ 2.81 (m, 1 H), 1.42 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.4, 140.1, 128.9, 128.7, 128.4, 127.2, 124.3, 123.6, 120.9, 119.7, 117.5, 74.4, 73.3, 66.8, 38.8, 29.8, 26.1(one peak merged with others). Conformed by X-ray data.

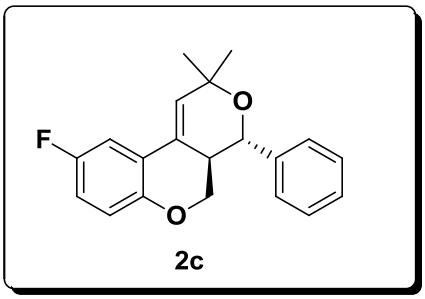
Spectral data for (4'S,4a'R)-4'-phenyl-4a',5'-dihydro-4'H-spiro[cyclopentane-1,2'-pyrano[3,4-c]chromene] (2b):



Colourless oil, IR (neat, cm^{-1}): 3022 (m), 2915 (s), 1627 (s), 1270 (s), 1159 (s), 910 (s), 690 (s); ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.52 (dd, *J* = 7.8, 1.5 Hz, 1 H), 7.37 ~ 7.31 (m, 5 H), 7.12~ 7.09 (m, 1 H), 6.91 ~ 6.88 (m, 1 H), 6.78 (dd, *J* = 8.2, 1.2 Hz, 1 H), 6.15 (d, *J* = 2.1 Hz, 1 H), 4.15 (d, *J* = 9.6 Hz, 1 H), 3.95 (dd, *J* = 10.6, 4.8 Hz, 1 H), 3.66 (dd, *J* = 12.3, 10.6

Hz, 1 H), 2.89 ~ 2.84 (m, 1 H), 2.00 ~ 1.67 (m, 8 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 154.3, 140.4, 128.7, 128.5, 128.2, 127.6, 127.2, 123.7, 123.5, 120.9, 119.9, 117.4, 85.5, 74.2, 67.0, 40.5, 38.8, 37.7, 24.4(2X CH2); HRMS calcd. for Chemical Formula: $C_{22}H_{22}O_2$, Exact Mass: 318.1620, found: 318.1617.

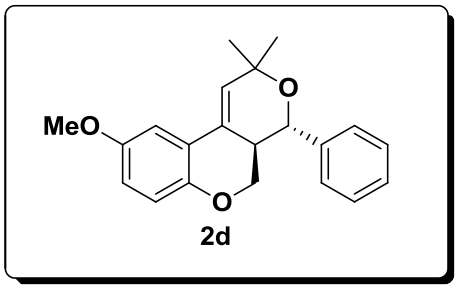
Spectral data for (4S,4aR)-9-fluoro-2,2-dimethyl-4-phenyl-2,4,4a,5-tetrahydropyrano-[3,4-c]chromene (2c):



2c

Colourless oil, IR (neat, cm^{-1}): 3028 (m), 2918 (s), 1630 (s), 1275 (s), 1162 (s), 912 (s), 695 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.45 ~ 7.30 (m, 5 H), 7.17 (dd, *J* = 10.2, 2.8 Hz, 1 H), 6.85~ 6.80 (m, 1 H), 6.73 (dd, *J* = 9.2, 5.2 Hz, 1 H), 6.13 (d, *J* = 2.0 Hz, 1 H), 4.24 (d, *J* = 10.0 Hz, 1 H), 3.89 (dd, *J* = 10.8, 4.8 Hz, 1 H), 3.60 (dd, *J* = 12.4, 10.8 Hz, 1 H), 2.85 ~ 2.79 (m, 1 H), 1.42 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.7 (J_{CF} = 218.1 Hz), 154.5, 139.9, 128.7, 128.4, 127.2, 1271, 125.6, 120.6, 118.5 (J_{CF} = 8.4 Hz), 115.8 (J_{CF} = 13.5 Hz), 109.3 (J_{CF} = 23.5 Hz), 74.4, 73.3, 66.9, 38.6, 29.5, 26.1; HRMS calcd. for Chemical Formula: $C_{20}H_{19}FO_2$, Exact Mass: 310.1369, found: 310.1373.

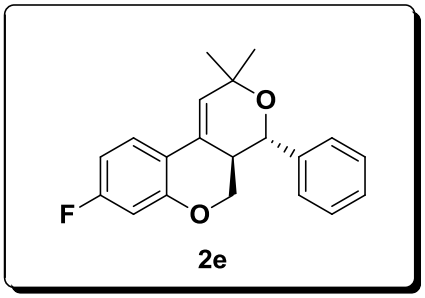
Spectral data for (4S,4aR)-9-methoxy-2,2-dimethyl-4-phenyl-2,4,4a,5-tetrahydropyrano [3,4-c]chromene (2d):



2d

Pale yellow solid, m. p. 123.8- 124.5 ^{o}C, IR (KBr, cm^{-1}): 3033 (m), 2924 (s), 1620 (s), 1265 (s), 1156 (s), 914 (s), 685 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38 ~ 7.30 (m, 5 H), 7.00 ~ 6.99 (m, 1 H), 6.73~ 6.72 (m, 2 H), 6.14 (d, *J* = 2.0 Hz, 1 H), 4.25 (d, *J* = 9.6 Hz, 1 H), 3.87 (dd, *J* = 10.4, 4.8 Hz, 1 H), 3.78 (s, 3 H), 3.59 (dd, *J* = 12.0, 10.4 Hz, 1 H), 2.86 ~ 2.79 (m, 1 H), 1.43 (s, 3 H), 1.42 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.9, 148.8, 140.2, 128.7, 128.4, 127.5, 127.2, 124.6, 120.0, 118.3, 115.9, 107.4, 74.4, 73.4, 67.0, 55.8, 38.9, 29.7, 26.2; HRMS calcd. for Chemical Formula: $C_{21}H_{22}O_3$, Exact Mass: 322.1569, found: 322.1567.

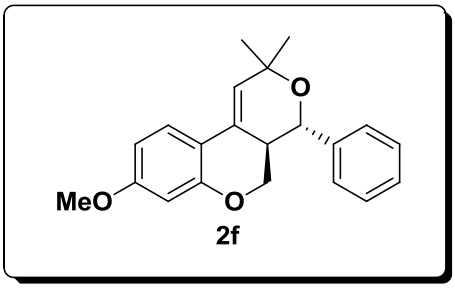
Spectral data for (4S,4aR)-8-fluoro-2,2-dimethyl-4-phenyl-2,4,4a,5-tetrahydropyrano [3,4-c]chromene (2e):



2e

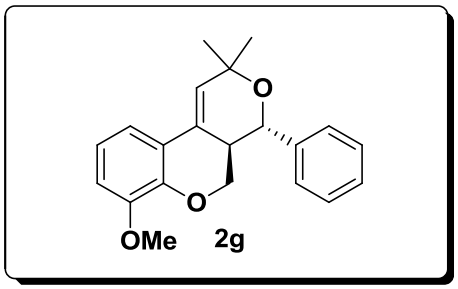
Off white solid, m.p. 125.0-126.0 ^{o}C, IR (KBr, cm^{-1}): 3029 (m), 2925 (s), 1625 (s), 1275 (s), 1160 (s), 915 (s), 690 (s); ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.46 (dd, *J* = 8.8, 6.4 Hz, 1 H), 7.38 ~ 7.30 (m, 5 H), 6.63 (dt, *J* = 8.8, 2.6 Hz, 1 H), 6.50 (dd, *J* = 9.9, 2.6 Hz, 1 H), 6.08 (d, *J* = 2.2 Hz, 1 H), 4.25 (d, *J* = 9.5 Hz, 1 H), 3.92 (dd, *J* = 10.6, 4.8 Hz, 1 H), 3.64 (dd, *J* = 12.3, 10.6 Hz, 1 H), 2.84 ~ 2.79 (m, 1 H), 1.41 (s, 6 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 162.9 (J_{CF} = 246.0 Hz), 155.4 (J_{CF} = 12.3 Hz), 140.1, 128.7, 128.4, 127.2, 126.5, 125.0 (J_{CF} = 9.6 Hz), 123.9, 116.1, 108.5 (J_{CF} = 22.0 Hz), 104.3 (J_{CF} = 24.0 Hz), 74.4, 73.3, 67.1, 38.6, 29.7, 26.1; HRMS calcd. for Chemical Formula: $C_{20}H_{19}FO_2$, Exact Mass: 310.1369, found: 310.1375.

Spectral data for (4S,4aR)-8-methoxy-2,2-dimethyl-4-phenyl-2,4,4a,5-tetrahydropyrano [3,4-c]chromene (2f) :



Colourless oil, IR (neat, cm^{-1}): 3026 (m), 2920 (s), 1620 (s), 1265 (s), 1155 (s), 910 (s), 675 (s); ^1H NMR (600 MHz, $CDCl_3$): δ 7.41 (d, *J* = 8.8 Hz, 1 H), 7.38 ~ 7.32 (m, 5 H), 6.51 (dd, *J* = 8.8, 2.6 Hz, 1 H), 6.31 (d, *J* = 2.6 Hz, 1 H), 6.01 (d, *J* = 2.4 Hz, 1 H), 4.25 (d, *J* = 9.6 Hz, 1 H), 3.89 (dd, *J* = 10.5, 4.8 Hz, 1 H), 3.74 (s, 3 H), 3.64 (dd, *J* = 12.3, 10.5 Hz, 1 H), 2.84 ~ 2.79 (m, 1 H), 1.41 (s, 6 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 160.4, 155.5, 140.3, 128.7, 128.3, 127.2, 126.9, 124.6, 122.1, 112.7, 108.7, 101.4, 74.4, 73.4, 67.1, 55.3, 38.8, 29.7, 26.3; HRMS calcd. for Chemical Formula: $C_{21}H_{22}O_3$, Exact Mass: 322.1569, found: 322.1571.

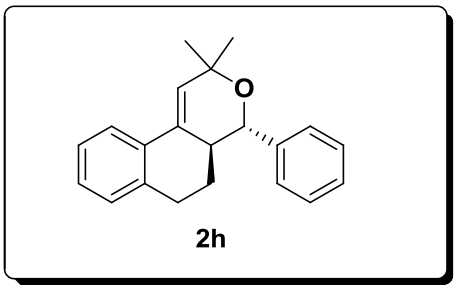
Spectral data for (4S,4aR)-7-methoxy-2,2-dimethyl-4-phenyl-2,4,4a,5-tetrahydropyrano [3,4-c]chromene (2g) :



Colourless oil, IR (neat, cm^{-1}): 3032 (m), 2920 (s), 1624 (s), 1262 (s), 1150 (s), 923 (s), 680 (s); ^1H NMR (600 MHz, $CDCl_3$): δ 7.37 ~ 7.31 (m, 5 H), 7.14 (dd, *J* = 8.0, 1.2 Hz, 1 H), 6.86 (dd, *J* = 8.0, 8.0 Hz, 1 H), 6.74 (dd, *J* = 8.0, 1.2 Hz, 1 H), 6.18 (d, *J* = 2.2 Hz, 1 H), 4.27

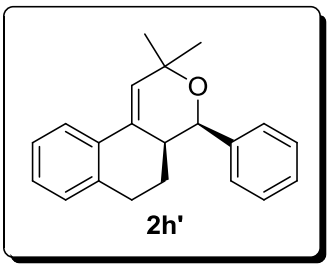
(d, *J* = 9.6 Hz, 1 H), 4.03 (dd, *J* = 10.5, 4.8 Hz, 1 H), 3.82 (s, 3 H), 3.68 (dd, *J* = 12.3, 10.5 Hz, 1 H), 2.88 ~ 2.86 (m, 1 H), 1.42 (s, 6 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 148.8, 144.1, 139.9, 128.7, 128.4, 127.2, 127.1, 124.9, 120.4, 120.3, 115.5, 110.4, 74.4, 73.2, 67.2, 55.9, 38.4, 29.6, 26.1; HRMS calcd. for HRMS calcd. for Chemical Formula: $C_{21}H_{22}O_3$, Exact Mass: 322.1569, found: 322.1565.

Spectral data for (4S,4aS)-2,2-dimethyl-4-phenyl-4,4a,5,6-tetrahydro-2H-benzo[f]iso-chromene (2h) :



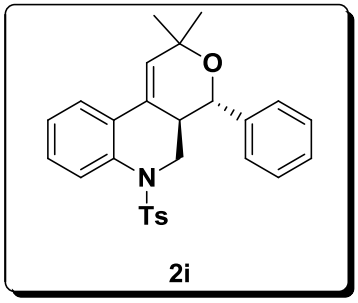
Colourless oil, IR (neat, cm^{-1}): 3020 (m), 2915 (s), 1620 (s), 1262 (s), 910 (s), 665 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.66 ~ 7.64 (m, 1 H), 7.44 ~ 7.29 (m, 5 H), 7.19 ~ 7.05 (m, 3 H), 6.29 (d, *J* = 2.1 Hz, 1 H), 4.32 (d, *J* = 9.3 Hz, 1 H), 2.74 ~ 2.70 (m, 2 H), 2.50 ~ 2.43 (m, 1 H), 1.67 ~ 1.55 (m,1 H), 1.44 ~ 1.25 (m, 7 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.4, 136.7, 132.7, 132.2, 129.3, 128.4, 127.9, 127.7, 127.1, 126.7, 126.0, 123.2, 77.7, 74.1, 41.2, 29.8, 29.1, 26.2, 25.3; HRMS calcd. for Chemical Formula: $C_{21}H_{22}O$, Exact Mass: 290.1671, found: 290.1669.

Spectral data for (4R,4aS)-2,2-dimethyl-4-phenyl-4,4a,5,6-tetrahydro-2H benzo[f]iso-chromene (2h') :



Colourless oil, IR (neat, cm^{-1}): 3021 (m), 2915 (s), 1622 (s), 1260 (s), 911 (s), 660 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38 ~ 7.25 (m, 6 H), 7.15 ~ 7.07 (m, 3 H), 5.93 (d, *J* = 1.2 Hz, 1 H), 5.12 (d, *J* = 4.4 Hz, 1 H), 2.83 ~ 2.71 (m, 2 H), 2.37 ~ 2.32 (m, 1 H), 1.60 ~ 1.24 (m, 8 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.2, 137.4, 136.2, 136.1, 129.0, 128.2, 128.1, 127.0, 126.8, 126.1, 125.8, 123.3, 73.3, 72.3, 39.7, 30.2, 29.4, 25.0, 24.5; HRMS calcd. for Chemical Formula: $C_{21}H_{22}O$, Exact Mass: 290.1671, found: 290.1672.

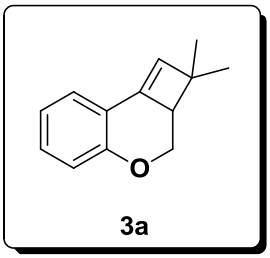
Spectral data for (4S,4aR)-2,2-dimethyl-4-phenyl-6-tosyl-4,4a,5,6-tetrahydro-2H-pyrano[3,4-c]quinoline (2i) :



2i

Pale yellow viscous oil, IR (neat, cm^{-1}): 3030 (m), 2924 (s), 1630 (s), 1360 (s), 1268 (s), 1155 (s), 925 (s), 670 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81 (dd, *J* = 8.0, 1.4 Hz, 1 H), 7.56 (dd, *J* = 8.0, 1.4 Hz, 1 H), 7.42~ 7.10 (m, 11 H), 6.14 (d, *J* = 2.3 Hz, 1 H), 4.04 (d, *J* = 9.3 Hz, 1 H), 3.82 (dd, *J* = 14.4, 4.4 Hz, 1 H), 3.05 (dd, *J* = 14.4, 12.5 Hz, 1 H), 2.40 (s, 3 H), 2.03 ~ 1.95 (m, 1 H), 1.31 (s, 3 H), 1.29 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.6, 139.6, 136.6, 135.6, 129.7, 128.7, 128.6, 128.0, 127.3, 127.1, 125.9, 125.8, 125.3, 123.2, 74.4, 74.3, 46.8, 36.9, 29.4, 26.2, 21.6 (Two peaks merged with others). HRMS calcd. for Chemical Formula: $C_{27}H_{27N}O3S$, Exact Mass: 445.1712, found: 445.0809.

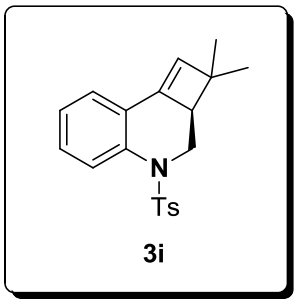
Spectral data for 2,2-dimethyl-2a,3-dihydro-2H-cyclobuta[c]chromene (3a) :



3a

Colourless oil, IR (neat, cm^{-1}): 3045 (m), 2915 (s), 1645 (s), 1286 (m), 690 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.25 (dd, *J* = 7.6, 1.6 Hz, 1 H), 7.13 ~ 7.10 (m, 1 H), 6.87~ 6.83 (m, 2 H), 6.10 (s, 1 H), 4.55 (dd, *J* = 9.8, 5.8 Hz, 1 H), 4.08 (dd, *J* = 12.3, 9.8 Hz, 1 H), 2.64 (dd, *J* = 12.3, 5.8 Hz, 1 H), 1.32 (s, 3 H), 1.15 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.5, 137.2, 133.2, 129.0, 124.2, 120.5, 119.5, 117.1, 70.4, 46.0, 44.1, 27.6, 20.3; HRMS calcd. for Chemical Formula: $C_{13}H_{14}O$, Exact Mass: 186.1045, found: 186.1046.

Spectral data for (R)-2,2-dimethyl-4-tosyl-2,2a,3,4-tetrahydrocyclobuta[c]quinoline (3i) :

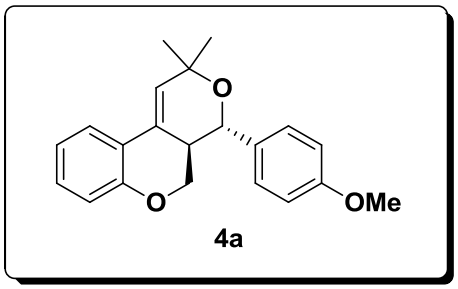


3i

Yellow oil, IR (neat, cm^{-1}): 3030 (m), 2924 (s), 1630 (s), 1360 (s), 1268 (s), 925 (s), 670 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (d, *J* = 2.0 Hz, 1 H), 7.57 ~ 7.54 (m, 2 H), 7.32~ 7.09 (m, 5 H), 6.08 (s, 1 H), 4.60 (dd, *J* = 12.8, 5.2 Hz, 1 H), 3.33 (t, *J* = 12.8 Hz, 1 H), 2.39 (s, 3 H), 2.1 (dd, *J* = 12.8, 5.6 Hz, 1 H), 1.15 (s, 3 H), 1.09 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.5, 139.0, 137.5, 136.6, 135.8, 129.5, 127.9, 127.0, 125.3, 125.2, 124.4, 123.9, 50.2, 45.4, 43.6, 27.1, 21.4, 20.1; HRMS calcd. for Chemical Formula: $C_{20}H_{21}NO_2S$, Exact Mass: 339.1293, found: 339.1277.

Spectral data for (4S,4aR)-2,2-dimethyl-4-phenyl-2,4,4a,5-tetrahydropyrano[3,4-c]-

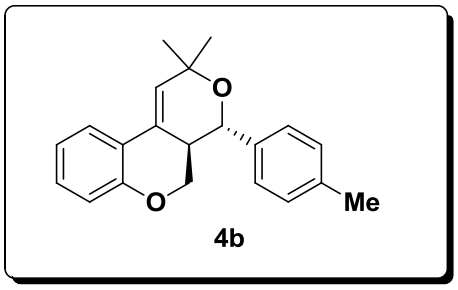
chromene (4a) :



4a

Off white Solid, m. p. 151.2-151.9 ^{o}C (KBr, cm^{-1}): 3030 (m), 2924 (s), 1630 (s), 1360 (s), 1268 (s), 1155 (s), 925 (s), 670 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52 (d, *J* = 8.0 Hz, 1 H), 7.31 (d, *J* = 8.8 Hz, 2 H), 7.14 ~ 7.10 (m, 1 H), 6.92 ~ 6.89 (m, 3 H), 6.79 (d, *J* = 8.0 Hz, 1 H), 6.16 (d, *J* = 2.0 Hz, 1 H), 4.21 (d, *J* = 9.6 Hz, 1 H), 3.92 (dd, *J* = 10.4, 4.8 Hz, 1 H), 3.80 (s, 3 H), 3.62 (dd, *J* = 12.0, 10.4 Hz, 1 H), 2.88 ~ 2.81 (m, 1 H), 1.41 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.6, 154.4, 132.2, 128.8, 128.4, 127.3, 124.3, 123.6, 120.9, 119.8, 117.5, 114.1, 74.4, 72.9, 66.9, 55.3, 38.3, 29.7, 26.2; HRMS calcd. for Chemical Formula: $C_{21}H_{22}O_3$, Exact Mass: 322.1569, found: 322.1519.

Spectral data for (4S,4aR)-2,2-dimethyl-4-p-tolyl-2,4,4a,5-tetrahydropyrano[3,4-c]chromene (4b) :

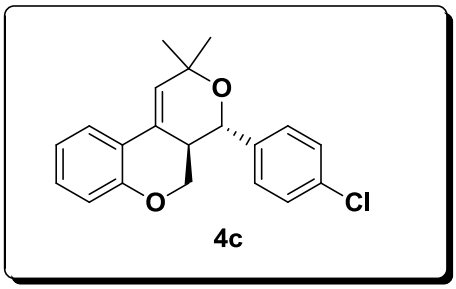


4b

Yellow solid, m. p. 136.5-137.3 ^{o}C IR (KBr, cm^{-1}): 3032 (m), 2915 (s), 1615 (s), 1269 (s), 1150 (s), 910 (s), 685 (s); ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.51 (dd, *J* = 8.0, 1.5 Hz, 1 H), 7.27 ~ 7.17 (m, 4 H), 7.18 ~ 7.11 (m, 1 H), 6.91 ~ 6.89 (m, 1 H), 6.79 (dd, *J* = 8.0, 1.0 Hz, 1 H), 6.16 (d, *J* = 2.2 Hz, 1 H), 4.22 (d, *J* = 9.6 Hz, 1 H), 3.93 (dd, *J* = 10.5, 4.8 Hz, 1 H), 3.63

(dd, *J* = 12.3, 10.5 Hz, 1 H), 2.87 ~ 2.82 (m, 1 H), 2.34 (s, 3 H), 1.41 (s, 6 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 154.4, 138.1, 137.1, 129.3, 128.8, 127.3, 127.1, 124.3, 123.6, 120.9, 119.8, 117.5, 74.4, 73.1, 66.9, 38.6, 29.7, 26.1, 21.2; HRMS calcd. for Chemical Formula: $C_{21}H_{22}O_2$, Exact Mass: 306.1620, found: 306.1625.

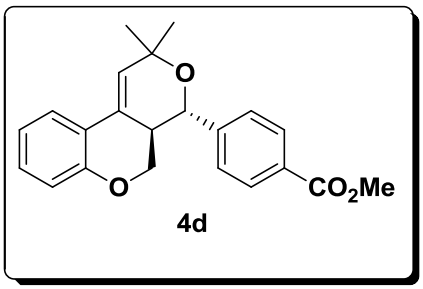
Spectral data for (4S,4aR)-4-(4-chlorophenyl)-2,2-dimethyl-2,4,4a,5-tetrahydropyrano [3,4-c]chromene (4c) :



4c

Colourless oil, IR (neat, cm^{-1}): 3030 (m), 2924 (s), 1630 (s), 1360 (s), 1268 (s), 1155 (s), 925 (s), 670 (s); ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.51 (dd, *J* = 8.0, 1.5 Hz, 1 H), 7.36 ~ 7.31 (m, 4 H), 7.13 ~ 7.11 (m, 1 H), 6.92 ~ 6.89 (m, 1 H), 6.79 (dd, *J* = 8.0, 1.1 Hz, 1 H), 6.17 (d, *J* = 2.3 Hz, 1 H), 4.23 (d, *J* = 9.6 Hz, 1 H), 3.90 (dd, *J* = 10.5, 4.8 Hz, 1 H), 3.64 (dd, *J* = 12.3, 10.5 Hz, 1 H), 2.81 ~ 2.76 (m, 1 H), 1.42 (s, 6 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 154.3, 138.7, 134.1, 129.0, 128.9, 128.6, 127.1, 124.2, 123.6, 121.0, 119.6, 117.5, 74.6, 72.7, 66.7, 38.9, 29.6, 26.2; HRMS calcd. for Chemical Formula: $C_{20}H_{19}ClO_2$, Exact Mass: 326.1074, found: 326.1066.

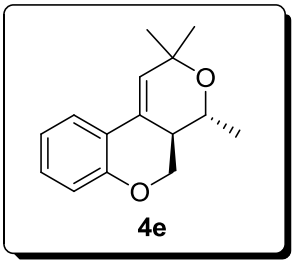
Spectral data for methyl 4-((4S,4aR)-2,2-dimethyl-2,4,4a,5-tetrahydropyrano[3,4-c]chromen-4-yl)benzoate (4d) :



4d

Yellow Solid, m. p. 156.6-157.4 ^{o}C, IR (KBr, cm^{-1}): 3025 (m), 2912 (s), 1735 (s), 1622 (s), 1340 (s), 1265 (s), 1159 (s), 920 (s), 685 (s); ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.05 (dd, *J* = 6.6, 1.6 Hz, 2 H), 7.51 (dd, *J* = 8.0, 1.5 Hz, 1 H), 7.46 (dd, *J* = 6.6, 1.5 Hz, 2 H), 7.13 ~ 7.10 (m, 1 H), 6.92 ~ 6.89 (m, 1 H), 6.79 (dd, *J* = 8.0, 1.1 Hz, 1 H), 6.18 (d, *J* = 2.3 Hz, 1 H), 4.32 (d, *J* = 9.6 Hz, 1 H), 3.90 ~ 3.89 (m, 4 H), 3.67 (dd, *J* = 12.3, 10.6 Hz, 1 H), 2.83 ~ 2.78 (m, 1 H), 1.43 (s, 3 H), 1.42 (s, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 166.8, 154.3, 145.2, 130.2, 130.0, 129.0, 127.2, 127.0, 124.2, 123.6, 121.0, 119.6, 117.5, 74.6, 73.0, 66.6, 52.1, 38.9, 29.6, 26.2; HRMS calcd. for Chemical Formula: $C_{22}H_{22}O_4$, Exact Mass: 350.1518, found: 350.1518.

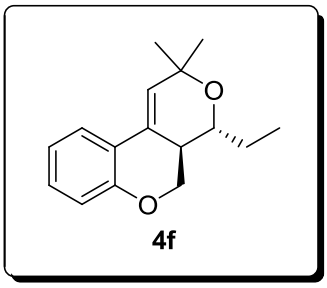
Spectral data for (4R,4aR)-2,2,4-trimethyl-2,4,4a,5-tetrahydropyrano[3,4-c]chromene (4e) :



4e

Colourless oil, IR (neat, cm^{-1}): 3015 (m), 2920 (s), 1630 (s), 1350 (s), 1261 (s), 970 (s), 655 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.45 (dd, *J* = 8.0, 1.5 Hz, 1 H), 7.14 ~ 6.79 (m, 3 H), 6.07 (d, *J* = 2.1 Hz, 1 H), 4.29 (dd, *J* = 10.4, 4.8 Hz, 1 H), 3.62 (dd, *J* = 12.3, 10.4 Hz, 1 H), 3.51 ~ 3.44 (m, 1 H), 2.50 ~ 2.43 (m, 1 H), 1.33 ~ 131 (m, 9 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.1, 128.8, 126.9, 124.5, 123.6, 120.9, 117.3, 115.6, 73.1, 67.5, 65.7, 38.9, 29.6, 26.0, 19.7; HRMS calcd. for Chemical Formula: $C_{15}H_{18}O_2$, Exact Mass: 230.1307, found: 230.1309.

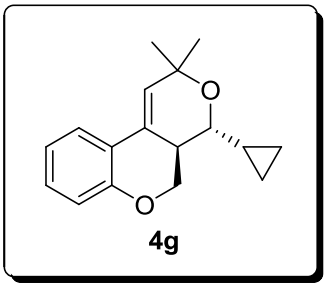
Spectral data for (4R,4aR)-4-ethyl-2,2-dimethyl-2,4,4a,5-tetrahydropyrano[3,4-c]chromene (4f) :



4f

Colourless oil, IR (neat, cm^{-1}): 3020 (m), 2916 (s), 1625 (s), 1355 (s), 1260 (s), 974 (s), 660 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46 (dd, *J* = 8.2, 1.5 Hz, 1 H), 7.14 ~ 6.86 (m, 2 H), 6.81 (dd, *J* = 8.2, 1.1 Hz, 1 H), 6.07 (d, *J* = 2.1 Hz, 1 H), 4.30 (dd, *J* = 10.5, 4.8 Hz, 1 H), 3.61 (dd, *J* = 12.3, 10.5 Hz, 1 H), 3.67 ~ 3.22 (m, 1 H), 2.57 ~ 2.50 (m, 1 H), 1.73 ~ 1.54 (m, 2 H), 1.33 (s, 3 H), 1.30 (s, 3 H), 1.04 (t, *J* = 7.4 Hz, 3 H),; ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.1, 128.7, 127.7, 124.7, 123.7, 120.8, 117.2, 115.6, 72.9, 70.8, 67.5, 36.9, 29.6, 26.1, 25.7, 9.8; HRMS calcd. for Chemical Formula: $C_{16}H_{20}O_2$, Exact Mass: 244.1463, found: 244.1469.

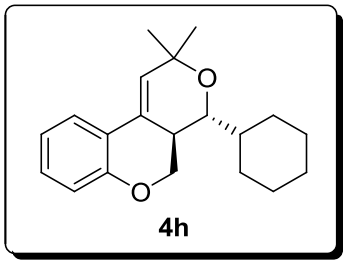
Spectral data for (4R,4aR)-4-cyclopropyl-2,2-dimethyl-2,4,4a,5-tetrahydropyrano[3,4-c]chromene (4g) :



4g

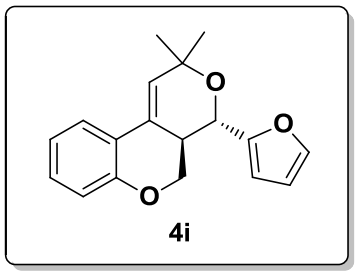
Yellow Solid, m.p. 82.0-83.0 ^{o}C, IR (KBr, cm^{-1}): 3020 (m), 2918 (s), 1625 (s), 1352 (w), 1270 (s), 971 (s), 665 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46 (dd, *J* = 7.9, 1.5 Hz, 1 H), 7.13 ~ 6.79 (m, 3 H), 6.05 (d, *J* = 2.0 Hz, 1 H), 4.59 (dd, *J* = 10.5, 5.0 Hz, 1 H), 3.60 (dd, *J* = 12.4, 10.5 Hz, 1 H), 2.83 ~ 2.76 (m, 1 H), 2.50 (t, *J* = 9.0 Hz, 1 H), 1.36 (s, 3 H), 1.26 (s, 3 H), 1.06 ~ 0.96 (m, 1 H), 0.68 ~ 0.55 (m, 2 H), 0.38 ~ 0.18 (m, 2 H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.2, 128.7, 127.0, 124.3, 123.6, 120.8, 119.8, 117.4, 75.2, 73.4, 67.2, 38.9, 29.7, 25.8, 14.3, 3.5, 2.0; HRMS calcd. for Chemical Formula: $C_{17}H_{20}O_2$, Exact Mass: 256.1463, found: 256.1465.

Spectral data for (4R,4aR)-4-cyclohexyl-2,2-dimethyl-2,4,4a,5-tetrahydropyrano[3,4-c]chromene (4h) :



Colourless oil, IR (neat, cm^{-1}): 3020 (m), 2918 (s), 1625 (s), 1390 (w), 1265 (s), 975 (s), 680 (s); ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.44 (dd, *J* = 7.8, 1.4 Hz, 1 H), 7.12 ~ 7.09 (m, 1 H), 6.89 ~ 6.85 (m, 1 H), 6.80 (dd, *J* = 8.2, 1.1 Hz, 1 H), 6.05 (d, *J* = 2.1 Hz, 1 H), 4.29 (dd, *J* = 10.5, 4.8 Hz, 1 H), 3.58 (dd, *J* = 12.3, 10.5 Hz, 1 H), 3.18 ~ 3.16 (dd, *J* = 9.6, 2.1 Hz, 1 H), 2.74 ~ 2.70 (m, 1 H), 1.77 ~ 1.17 (m, 17 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 154.1, 128.6, 127.7, 124.9, 123.7, 120.8, 120.3, 117.2, 73.4, 72.9, 67.4, 39.4, 33.4, 30.4, 29.6, 26.7, 26.5, 26.4, 25.6, 25.5; HRMS calcd. for Chemical Formula: $C_{20}H_{26}O_2$, Exact Mass: 298.1933, found: 298.1926.

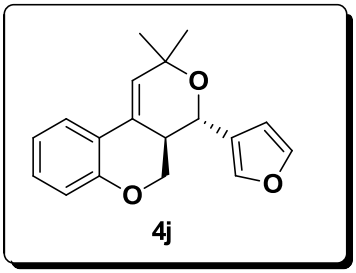
Spectral data for (4S,4aR)-4-(furan-2-yl)-2,2-dimethyl-2,4,4a,5-tetrahydropyrano[3,4-c]chromene (4i) :



Yellow Solid, m.p. 114.5-115.5 ^{o}C, IR (KBr, cm^{-1}): 3018 (m), 2920 (s), 1640 (s), 1354 (s), 1255 (s), 971 (s), 685 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50 (dd, *J* = 7.9, 1.4 Hz, 1 H), 7.43 ~ 7.42 (m, 1 H), 7.16 ~ 7.11 (m, 1 H), 6.93 ~ 6.89 (m, 1 H), 6.82 (dd, *J* = 8.2, 1.0 Hz, 1 H), 6.41 ~ 6.36 (m, 2 H), 6.14 (d, *J* = 2.1 Hz, 1 H), 4.40 (d, *J* = 9.9 Hz, 1 H), 4.08 (dd, *J* =

10.5, 4.7 Hz, 1 H), 3.62 (dd, *J* = 12.2, 10.6 Hz, 1 H), 3.18 ~ 3.11 (m, 1 H), 1.43 (s, 3 H), 1.40 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.3, 152.6, 142.8, 129.0, 127.1, 124.2, 123.7, 121.0, 119.6, 117.5, 110.3, 108.3, 74.6, 67.0, 66.2, 36.0, 29.5, 26.0; HRMS calcd. for Chemical Formula: $C_{18}H_{18}O_3$, Exact Mass: 282.1256, found: 282.1263.

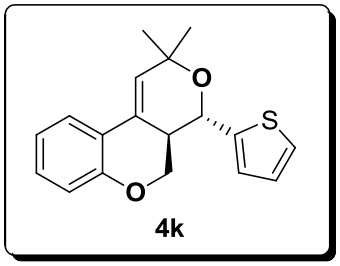
Spectral data for (4S,4aR)-4-(furan-3-yl)-2,2-dimethyl-2,4,4a,5-tetrahydropyrano[3,4-c]chromene (4j) :



4j

Yellow Solid, m.p. 87.0-88.0 ^{o}C, IR (KBr, cm^{-1}): 3020 (m), 2925 (s), 1642 (s), 1352 (s), 1250 (s), 974 (s), 680 (s); ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.50 (dd, *J* = 7.9, 1.5 Hz, 1 H), 7.49 (s, 1 H), 7.44 ~ 7.43 (m, 1 H), 7.15 ~ 7.12 (m, 1 H), 6.92 ~ 6.89 (m, 1 H), 6.82 (dd, *J* = 8.2, 1.1 Hz, 1 H), 6.50 ~ 6.49 (m, 1 H), 6.14 (d, *J* = 2.2 Hz, 1 H), 4.32 (d, *J* = 9.7 Hz, 1 H), 4.01 (dd, *J* = 10.5, 4.8 Hz, 1 H), 3.60 (dd, *J* = 12.3, 10.5 Hz, 1 H), 2.88 ~ 2.83 (m, 1 H), 1.41 (s, 3 H), 1.40 (s, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 154.3, 143.7, 140.3, 128.9, 127.1, 124.8, 124.3, 123.6, 121.0, 119.7, 117.4, 108.9, 74.2, 67.1, 65.1, 37.7, 29.6, 26.1; HRMS calcd. for Chemical Formula: $C_{18}H_{18}O_3$, Exact Mass: 282.1256, found: 282.1251.

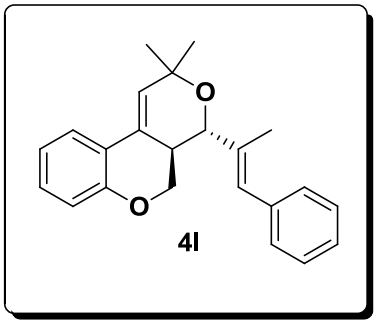
Spectral data for (4S,4aR)-2,2-dimethyl-4-(thiophen-2-yl)-2,4,4a,5-tetrahydropyrano [3,4-c]chromene (2k) :



4k

Pale Yellow Solid, m.p. 119.0-120.0 ^{o}C, IR (KBr, cm^{-1}): 3020 (m), 2915 (s), 1625 (s), 1350 (s), 1258 (s), 965 (s), 690 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51 (dd, *J* = 7.8, 1.5 Hz, 1 H), 7.32 ~ 7.31 (m, 1 H), 7.16 ~ 6.89 (m, 4 H), 6.80 (dd, *J* = 8.2, 1.1 Hz, 1 H), 6.16 (d, *J* = 2.3 Hz, 1 H), 4.60 (d, *J* = 9.6 Hz, 1 H), 4.07 (dd, *J* = 10.5, 5.0 Hz, 1 H), 3.64 (dd, *J* = 12.2, 10.5 Hz, 1 H), 2.97 ~ 2.89 (m, 1 H), 1.43 (s, 3 H), 1.42 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.3, 143.2, 128.9, 127.0, 126.5, 125.7, 125.4, 124.2, 123.6, 121.0, 119.5, 117.5, 74.9, 68.7, 67.0, 39.5, 29.6, 26.2; HRMS calcd. for Chemical Formula: $C_{18}H_{18}O_2S$, Exact Mass: 298.1028, found: 298.1032.

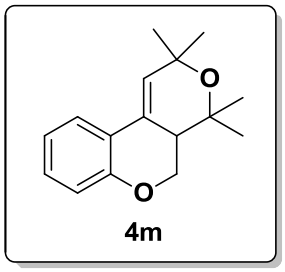
Spectral data for (4S,4aR)-2,2-dimethyl-4-((E)-1-phenylprop-1-en-2-yl)-2,4,4a,5-tetrahydropyrano[3,4-c]chromene (4l) :



4l

Off white solid, m.p. 93.5-94.5, IR (KBr, cm^{-1}): 3025 (m), 2930 (s), 1615 (s), 1650 (s), 1263 (s), 960 (s), 685 (s); ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.50 (dd, *J* = 7.9, 1.5 Hz, 1 H), 7.35 ~ 7.24 (m, 5 H), 7.15 ~ 7.12 (m, 1 H), 6.92 ~ 6.89 (m, 1 H), 6.83 (dd, *J* = 8.2, 1.2 Hz, 1 H), 6.49 (s, 1 H), 6.12 (d, *J* = 2.3 Hz, 1 H), 4.23 (dd, *J* = 10.9, 4.9 Hz, 1 H), 3.88 (d, *J* = 9.6 Hz, 1 H), 3.63 (dd, *J* = 12.4, 10.9 Hz, 1 H), 2.86 ~ 2.81 (m, 1 H), 1.99 (s, 3 H), 1.40 (s, 6 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 154.4, 137.0, 136.2, 129.1, 128.9 (2X CH), 128.1, 127.0, 126.7, 124.2, 123.6, 120.9, 119.9, 117.5, 77.2, 73.7, 67.2, 34.2, 29.6, 26.2, 13.2; HRMS calcd. for Chemical Formula: $C_{23}H_{24}O_2$, Exact Mass: 332.1776, found: 332.1775.

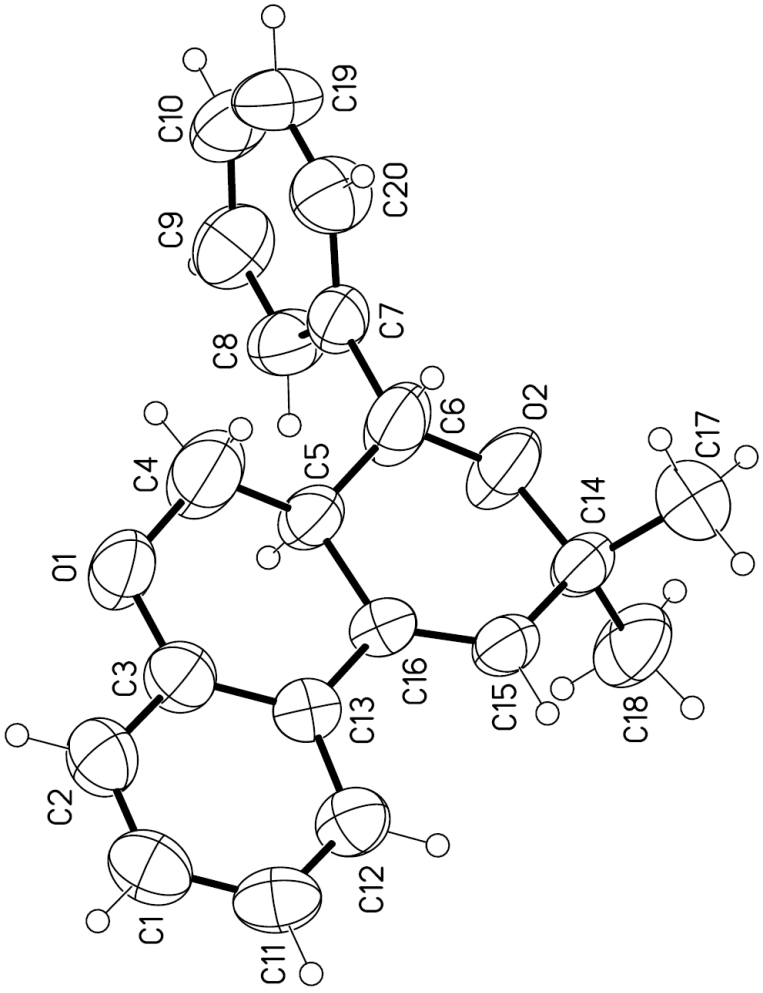
Spectral data for 2,2,4,4-tetramethyl-2,4,4a,5-tetrahydropyrano[3,4-c]chromene (4m) :



4m

Colourless oil, IR (neat, cm^{-1}): 3010 (m), 2936 (s), 1640 (s), 1255 (s), 956 (s), 690 (s); ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.58 (dd, *J* = 7.9 , 1.5 Hz, 1 H), 7.12 ~ 7.10 (m, 1 H), 6.92 ~ 6.91 (m, 1 H), 6.85 (dd, *J* = 8.2, 1.2 Hz, 1 H), 6.20 (d, *J* = 2.2 Hz, 1 H), 4.24 (dd, *J* = 10.5, 4.9 Hz, 1 H), 3.63 (dd, *J* = 12.5, 10.5 Hz, 1 H), 2.69 ~ 2.66 (m, 1 H), 1.36 (s, 3 H), 1.33 (s, 3 H), 1.30 (s, 3 H), 1.27 (s, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 155.4, 128.4, 124.3, 123.2, 123.1, 121.0, 120.7, 117.8, 72.0, 71.2, 67.2, 41.7, 34.7, 29.7, 29.4, 23.3; HRMS calcd. for Chemical Formula: $C_{16}H_{20}O_{2,}$ Exact Mass: 244.3288, found: 244.3286.

X-ray crystallographic structure and data
Compound (2a)



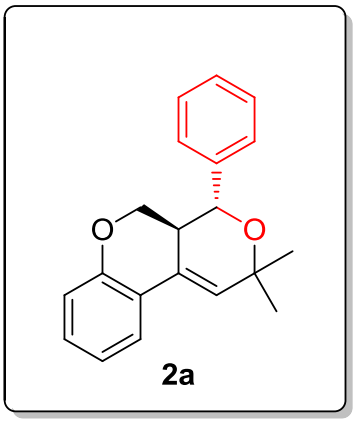


Table 1. Crystal data and structure refinement for 120240.

Identification code	120240	
Empirical formula	C20 H20 O2	
Formula weight	292.36	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P b c a	
Unit cell dimensions	a = 8.9306(4) Å	α= 90°.
	b = 17.9709(7) Å	β= 90°.
	c = 19.8097(8) Å	γ = 90°.
Volume	3179.3(2) $Å^3$	
Z	8	
Density (calculated)	1.222 Mg/m^3	
Absorption coefficient	0.077 mm^{-1}	
F(000)	1248	
Crystal size	0.10 x 0.07 x 0.03 mm^3	
Theta range for data collection	2.06 to 26.40°.	
Index ranges	-11<=h<=11, -22<=k<=22, -20<=l<=24	
Reflections collected	24248	
Independent reflections	3258 [R(int) = 0.0874]	
Completeness to theta = 26.40°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9486 and 0.8565	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3258 / 0 / 202	
Goodness-of-fit on F^2	1.018	
Final R indices [I>2sigma(I)]	R1 = 0.0731, wR2 = 0.1961	
R indices (all data)	R1 = 0.1806, wR2 = 0.2528	
Extinction coefficient	0.0053(17)	
Largest diff. peak and hole	0.744 and -0.395 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 120240. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	558(3)	-224(2)	671(1)	74(1)
O(2)	5281(3)	1028(2)	1052(2)	81(1)
C(1)	149(5)	-1874(2)	1686(2)	69(1)
C(2)	-105(5)	-1328(2)	1224(2)	66(1)
C(3)	903(4)	-736(2)	1157(2)	58(1)
C(4)	1334(6)	455(3)	716(3)	108(2)
C(5)	2925(4)	409(2)	842(2)	53(1)
C(6)	3842(5)	1083(3)	848(2)	79(1)
C(7)	3746(4)	1589(2)	247(2)	53(1)
C(8)	4397(5)	1406(2)	-363(2)	65(1)
C(9)	4313(5)	1867(3)	-910(2)	74(1)
C(10)	3571(5)	2523(3)	-858(2)	74(1)
C(11)	1397(5)	-1840(2)	2099(2)	66(1)
C(12)	2398(5)	-1265(2)	2022(2)	59(1)
C(13)	2201(4)	-705(2)	1548(2)	49(1)
C(14)	5658(4)	607(2)	1649(2)	56(1)
C(15)	4523(4)	11(2)	1782(2)	52(1)
C(16)	3282(4)	-98(2)	1428(2)	46(1)
C(17)	5783(6)	1130(3)	2245(2)	82(1)
C(18)	7174(4)	268(3)	1492(2)	77(1)
C(19)	2894(6)	2715(3)	-268(3)	80(1)
C(20)	2974(5)	2255(3)	280(2)	71(1)

Table 3. Bond lengths [Å] and angles [°] for 120240.

O(1)-C(3)	1.368(5)
O(1)-C(4)	1.406(5)
O(2)-C(6)	1.352(5)

O(2)-C(14)	1.443(4)
C(1)-C(2)	1.361(6)
C(1)-C(11)	1.383(6)
C(1)-H(1)	0.9300
C(2)-C(3)	1.400(5)
C(2)-H(2)	0.9300
C(3)-C(13)	1.395(5)
C(4)-C(5)	1.445(6)
C(4)-H(4A)	0.9700
C(4)-H(4B)	0.9700
C(5)-C(6)	1.463(5)
C(5)-C(16)	1.509(5)
C(5)-H(5)	0.9800
C(6)-C(7)	1.499(5)
C(6)-H(6)	0.9800
C(7)-C(8)	1.381(5)
C(7)-C(20)	1.384(6)
C(8)-C(9)	1.366(6)
C(8)-H(8)	0.9300
C(9)-C(10)	1.356(6)
C(9)-H(9)	0.9300
C(10)-C(19)	1.360(6)
C(10)-H(10)	0.9300
C(11)-C(12)	1.375(6)
C(11)-H(11)	0.9300
C(12)-C(13)	1.388(5)
C(12)-H(12)	0.9300
C(13)-C(16)	1.475(5)
C(14)-C(15)	1.498(5)
C(14)-C(17)	1.514(6)
C(14)-C(18)	1.517(5)
C(15)-C(16)	1.327(5)
C(15)-H(15)	0.9300
C(17)-H(17A)	0.9600
C(17)-H(17B)	0.9600
C(17)-H(17C)	0.9600

C(18)-H(18A)	0.9600
C(18)-H(18B)	0.9600
C(18)-H(18C)	0.9600
C(19)-C(20)	1.367(6)
C(19)-H(19)	0.9300
C(20)-H(20)	0.9300

C(3)-O(1)-C(4)	115.3(3)
C(6)-O(2)-C(14)	120.4(3)
C(2)-C(1)-C(11)	120.1(4)
C(2)-C(1)-H(1)	120.0
C(11)-C(1)-H(1)	120.0
C(1)-C(2)-C(3)	120.3(4)
C(1)-C(2)-H(2)	119.9
C(3)-C(2)-H(2)	119.9
O(1)-C(3)-C(13)	123.4(3)
O(1)-C(3)-C(2)	115.7(4)
C(13)-C(3)-C(2)	120.8(4)
O(1)-C(4)-C(5)	116.5(4)
O(1)-C(4)-H(4A)	108.2
C(5)-C(4)-H(4A)	108.2
O(1)-C(4)-H(4B)	108.2
C(5)-C(4)-H(4B)	108.2
H(4A)-C(4)-H(4B)	107.3
C(4)-C(5)-C(6)	120.2(4)
C(4)-C(5)-C(16)	112.0(3)
C(6)-C(5)-C(16)	112.1(3)
C(4)-C(5)-H(5)	103.4
C(6)-C(5)-H(5)	103.4
C(16)-C(5)-H(5)	103.4
O(2)-C(6)-C(5)	118.2(4)
O(2)-C(6)-C(7)	109.7(3)
C(5)-C(6)-C(7)	117.7(3)
O(2)-C(6)-H(6)	102.8
C(5)-C(6)-H(6)	102.8
C(7)-C(6)-H(6)	102.8

C(8)-C(7)-C(20)	117.2(3)
C(8)-C(7)-C(6)	121.8(4)
C(20)-C(7)-C(6)	121.0(4)
C(9)-C(8)-C(7)	121.8(4)
C(9)-C(8)-H(8)	119.1
C(7)-C(8)-H(8)	119.1
C(10)-C(9)-C(8)	119.6(4)
C(10)-C(9)-H(9)	120.2
C(8)-C(9)-H(9)	120.2
C(9)-C(10)-C(19)	120.1(4)
C(9)-C(10)-H(10)	119.9
C(19)-C(10)-H(10)	119.9
C(12)-C(11)-C(1)	119.4(4)
C(12)-C(11)-H(11)	120.3
C(1)-C(11)-H(11)	120.3
C(11)-C(12)-C(13)	122.6(4)
C(11)-C(12)-H(12)	118.7
C(13)-C(12)-H(12)	118.7
C(12)-C(13)-C(3)	116.8(4)
C(12)-C(13)-C(16)	124.2(3)
C(3)-C(13)-C(16)	119.0(3)
O(2)-C(14)-C(15)	111.2(3)
O(2)-C(14)-C(17)	109.4(3)
C(15)-C(14)-C(17)	110.9(3)
O(2)-C(14)-C(18)	104.5(3)
C(15)-C(14)-C(18)	110.6(3)
C(17)-C(14)-C(18)	110.1(3)
C(16)-C(15)-C(14)	125.3(3)
C(16)-C(15)-H(15)	117.4
C(14)-C(15)-H(15)	117.4
C(15)-C(16)-C(13)	124.8(3)
C(15)-C(16)-C(5)	119.6(3)
C(13)-C(16)-C(5)	115.6(3)
C(14)-C(17)-H(17A)	109.5
C(14)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5

C(14)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
C(14)-C(18)-H(18A)	109.5
C(14)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5
C(14)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5
C(10)-C(19)-C(20)	120.4(4)
C(10)-C(19)-H(19)	119.8
C(20)-C(19)-H(19)	119.8
C(19)-C(20)-C(7)	120.7(4)
C(19)-C(20)-H(20)	119.6
C(7)-C(20)-H(20)	119.6

Symmetry transformations used to generate equivalent atoms:

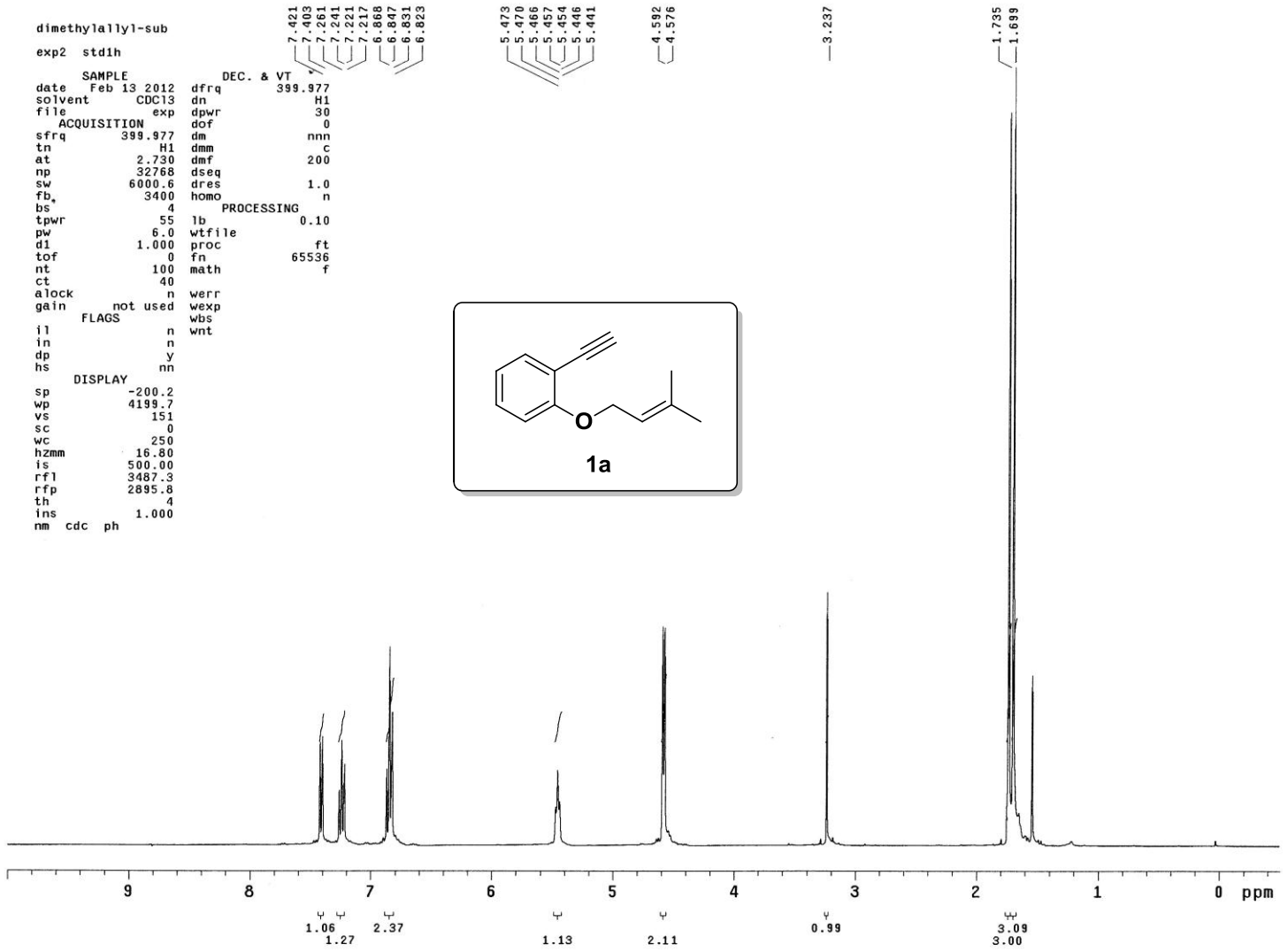
Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 120240. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

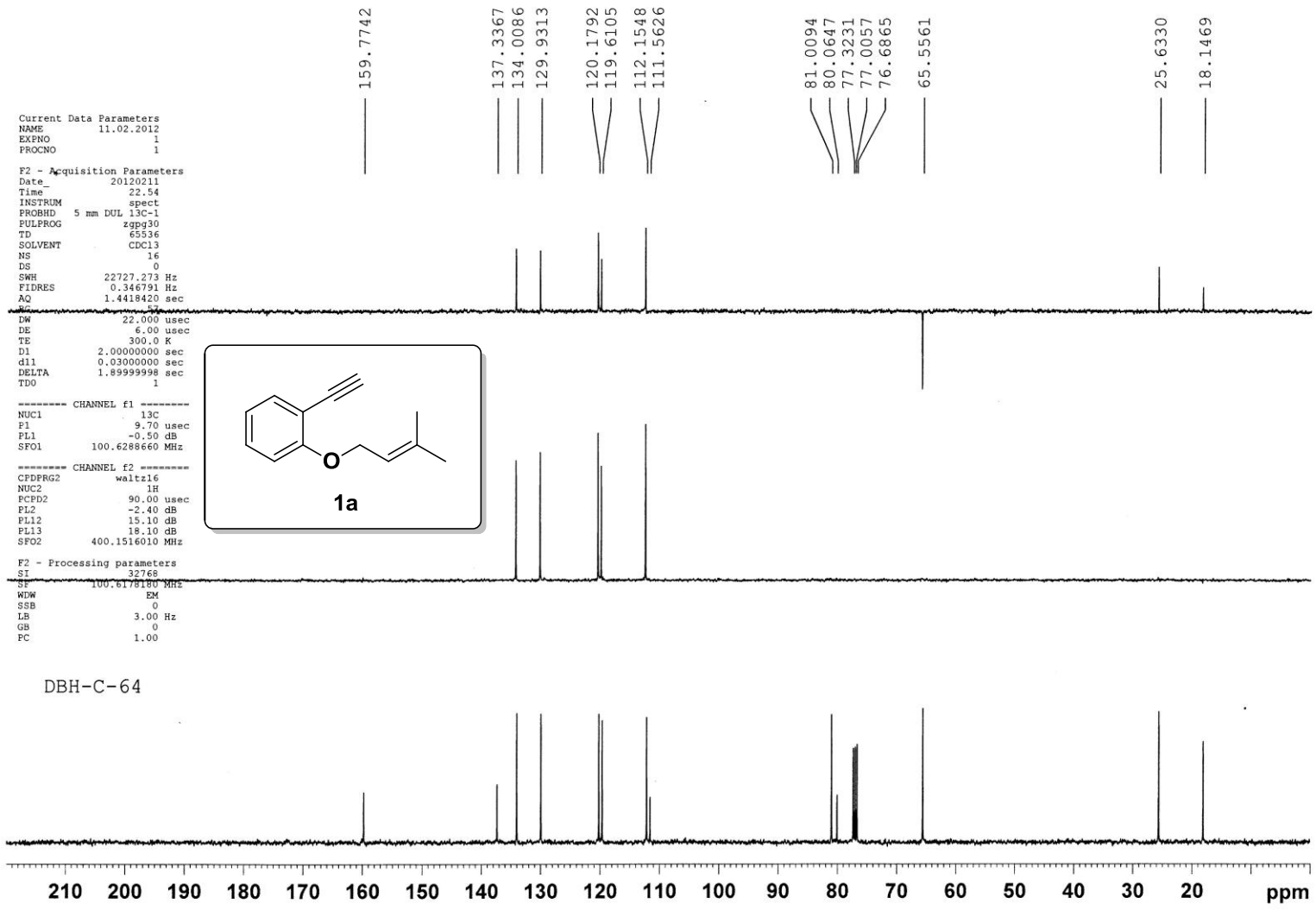
	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	66(2)	75(2)	81(2)	29(2)	-28(1)	-23(2)
O(2)	53(2)	89(2)	102(2)	50(2)	-30(2)	-19(2)
C(1)	69(3)	58(3)	79(3)	4(3)	13(2)	-14(2)
C(2)	57(3)	67(3)	75(3)	2(2)	-4(2)	-13(2)
C(3)	57(3)	58(3)	60(2)	4(2)	1(2)	-4(2)
C(4)	93(4)	96(4)	135(4)	50(3)	-56(4)	-31(3)
C(5)	43(2)	53(2)	64(2)	13(2)	-10(2)	0(2)
C(6)	73(3)	84(3)	79(3)	35(2)	-33(2)	-25(3)
C(7)	53(2)	54(3)	53(2)	11(2)	-11(2)	-10(2)
C(8)	67(3)	57(3)	71(3)	3(2)	-3(2)	14(2)
C(9)	72(3)	88(4)	61(3)	7(3)	3(2)	5(3)
C(10)	79(3)	71(3)	70(3)	27(2)	-13(3)	-1(3)
C(11)	73(3)	54(3)	72(3)	18(2)	12(2)	3(2)
C(12)	58(3)	58(3)	61(2)	8(2)	0(2)	2(2)

C(13)	50(2)	48(2)	49(2)	2(2)	2(2)	2(2)
C(14)	53(2)	53(2)	62(2)	16(2)	-18(2)	0(2)
C(15)	54(2)	49(2)	52(2)	11(2)	-7(2)	4(2)
C(16)	47(2)	44(2)	48(2)	7(2)	-1(2)	5(2)
C(17)	90(3)	67(3)	88(3)	-7(3)	-20(3)	-9(3)
C(18)	44(2)	91(4)	98(3)	17(3)	-12(2)	5(2)
C(19)	89(3)	51(3)	101(4)	8(3)	-7(3)	18(3)
C(20)	80(3)	70(3)	62(3)	-4(2)	5(2)	10(3)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 120240.

	x	y	z	U(eq)
H(1)	-517	-2269	1725	82
H(2)	-953	-1349	952	79
H(4A)	884	747	1075	130
H(4B)	1184	726	298	130
H(5)	3304	134	451	64
H(6)	3381	1381	1207	95
H(8)	4907	957	-403	78
H(9)	4762	1732	-1315	89
H(10)	3525	2843	-1227	88
H(11)	1558	-2204	2425	80
H(12)	3239	-1250	2298	71
H(15)	4704	-309	2142	62
H(17A)	6591	1473	2170	123
H(17B)	5975	848	2648	123
H(17C)	4864	1401	2296	123
H(18A)	7075	-77	1125	116
H(18B)	7543	12	1883	116
H(18C)	7862	655	1367	116
H(19)	2374	3162	-238	97
H(20)	2504	2392	680	85





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PROCNO                  1

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TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            8389.262 Hz
FIDRES         0.256020 Hz
AQ             1.9530228 sec
RG                  128
DW               59.600 usec
DE                6.50 usec
TE                297.6 K
D1            2.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                9.80 usec
PL1               0.00 dB
SFO1        598.9029945 MHz

F2 - Processing parameters
SI                32768
SF          598.9000256 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                15.00 cm
F1P              10.000 ppm
F1                5989.00 Hz
F2P               -0.500 ppm
F2                -299.45 Hz
PPMCM             0.52500 ppm/cm
HZCM            314.42252 Hz/cm
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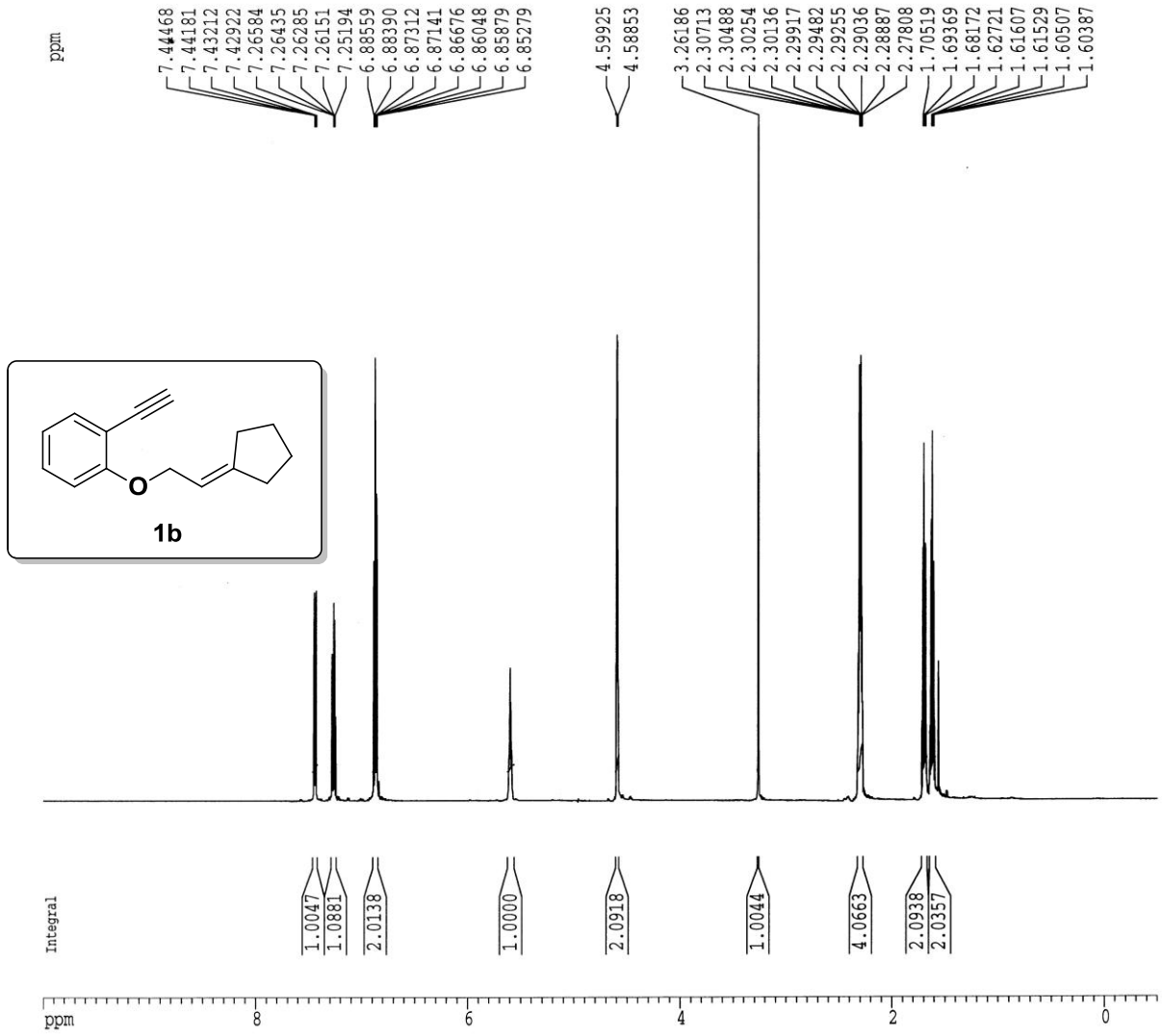

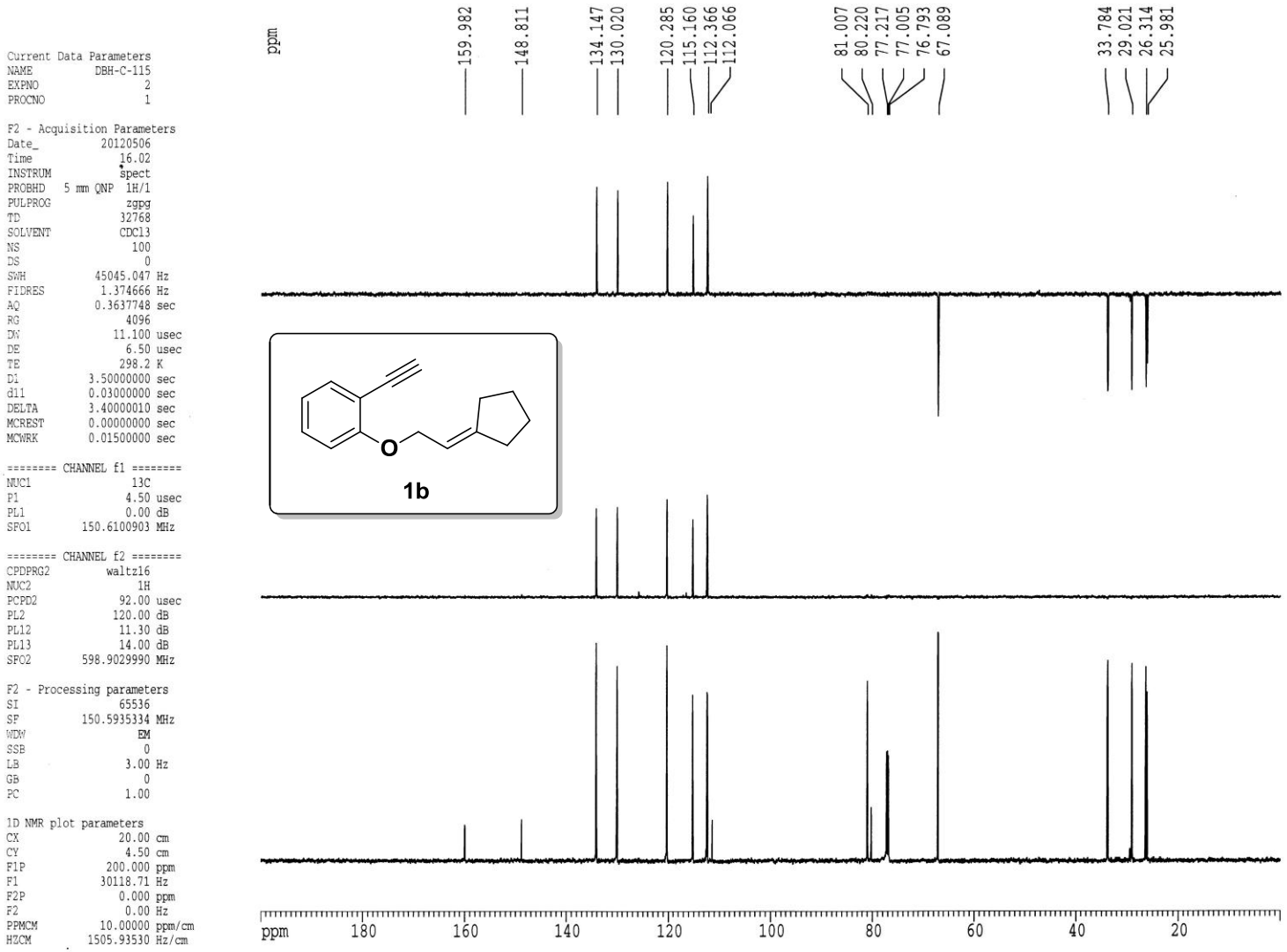

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EXPNO                        1
PROCNO                       1

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Time                   18.03
INSTRUM                spect
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PULPROG                   zg
TD                       16384
SOLVENT                CDCl3
NS                          16
DS                           0
SWH                 8389.262 Hz
FIDRES              0.512040 Hz
AQ                  0.9765364 sec
RG                         128
DW                     59.600 usec
DE                      6.50 usec
TE                      295.8 K
D1                  3.50000000 sec
MCREST              0.00000000 sec
MCWRK               0.01500000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                      8.20 usec
PL1                     3.00 dB
SFO1               598.9025066 MHz

F2 - Processing parameters
SI                       16384
SF                 598.9000257 MHz
WDW                         no
SSB                          0
LB                        0.00 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       20.00 cm
CY                       12.00 cm
F1P                     10.000 ppm
F1                     5989.00 Hz
F2P                     -0.500 ppm
F2                    -299.45 Hz
PPMCM                 0.52500 ppm/cm
HZCM                314.42252 Hz/cm
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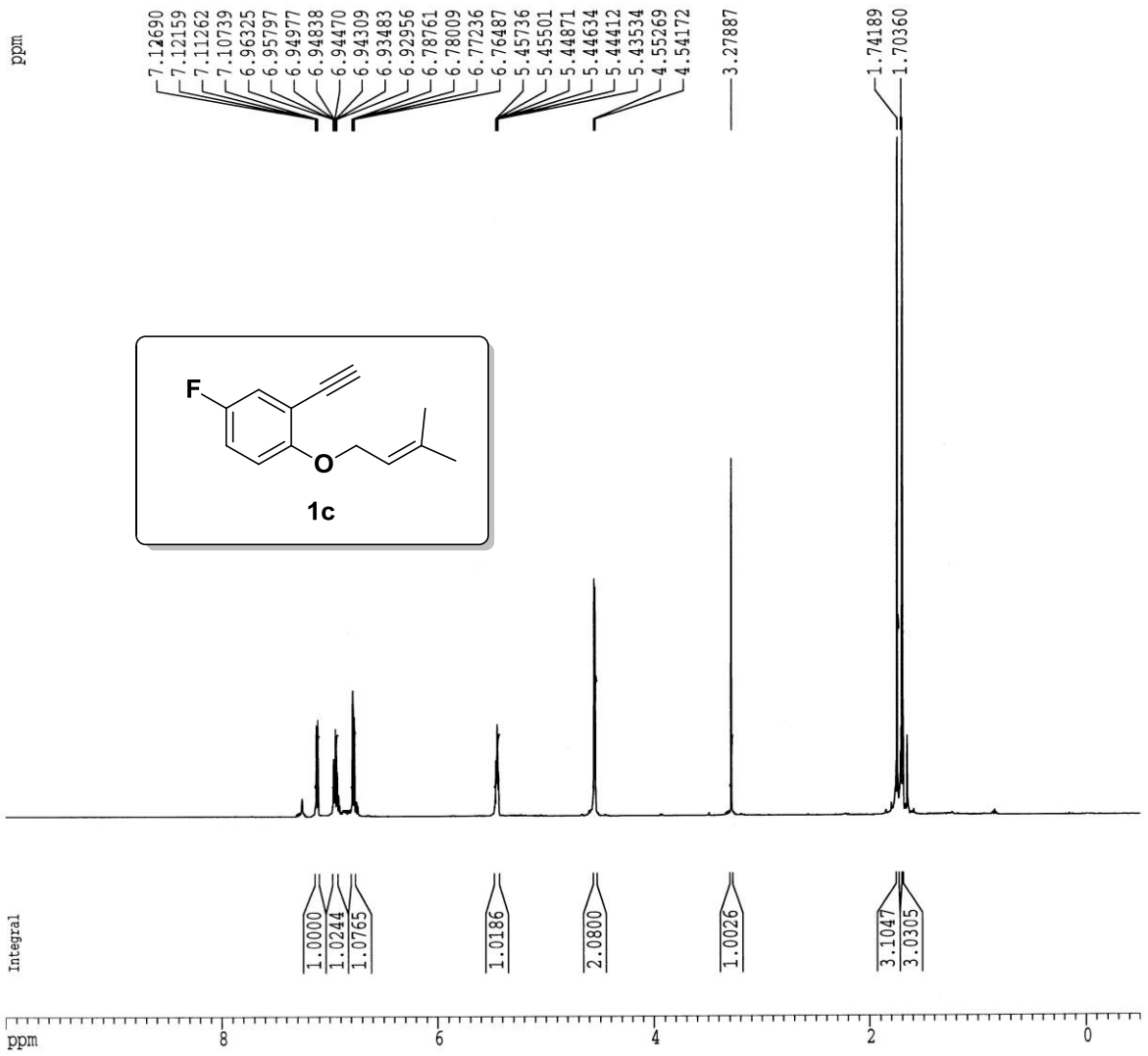

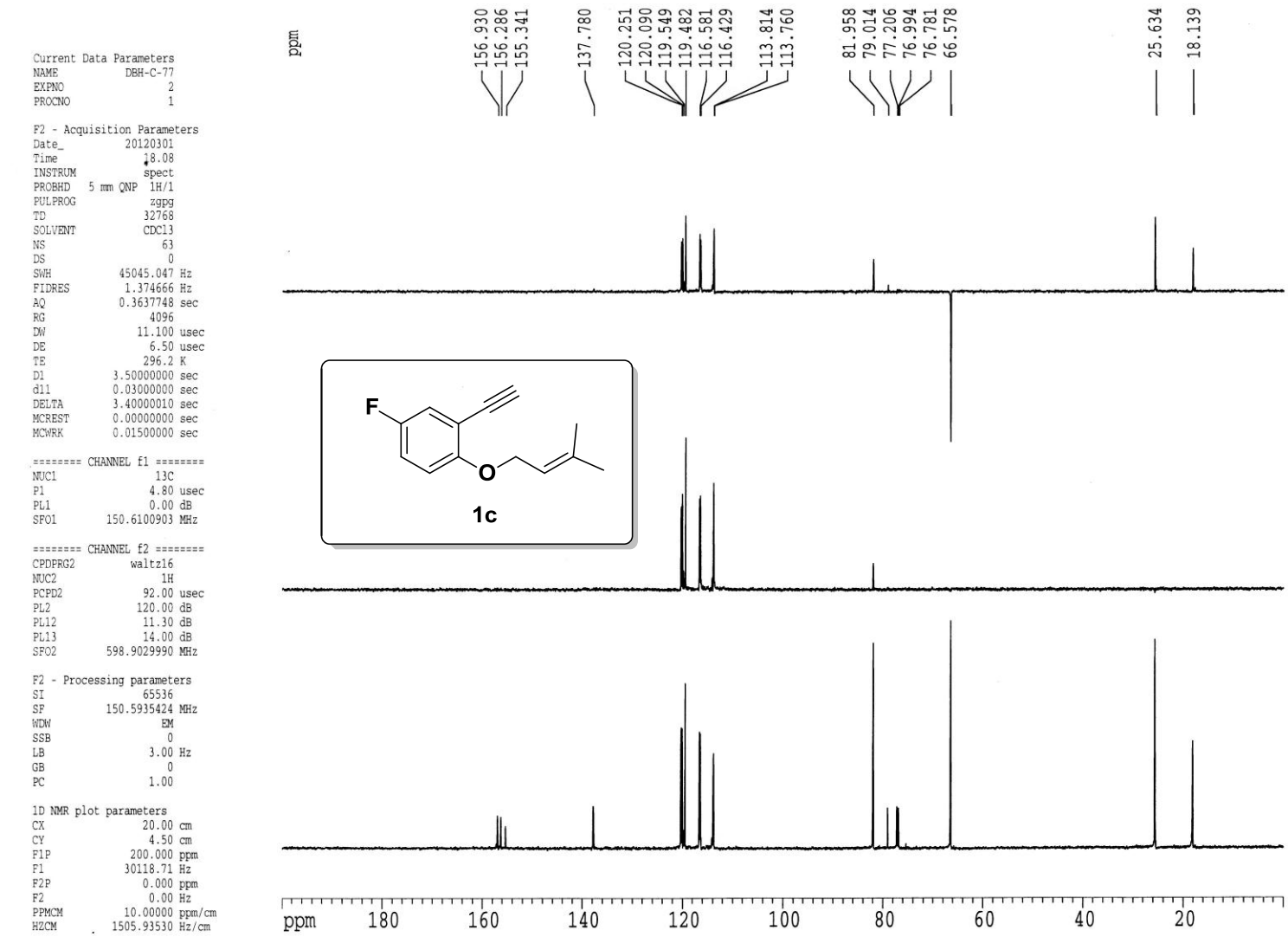

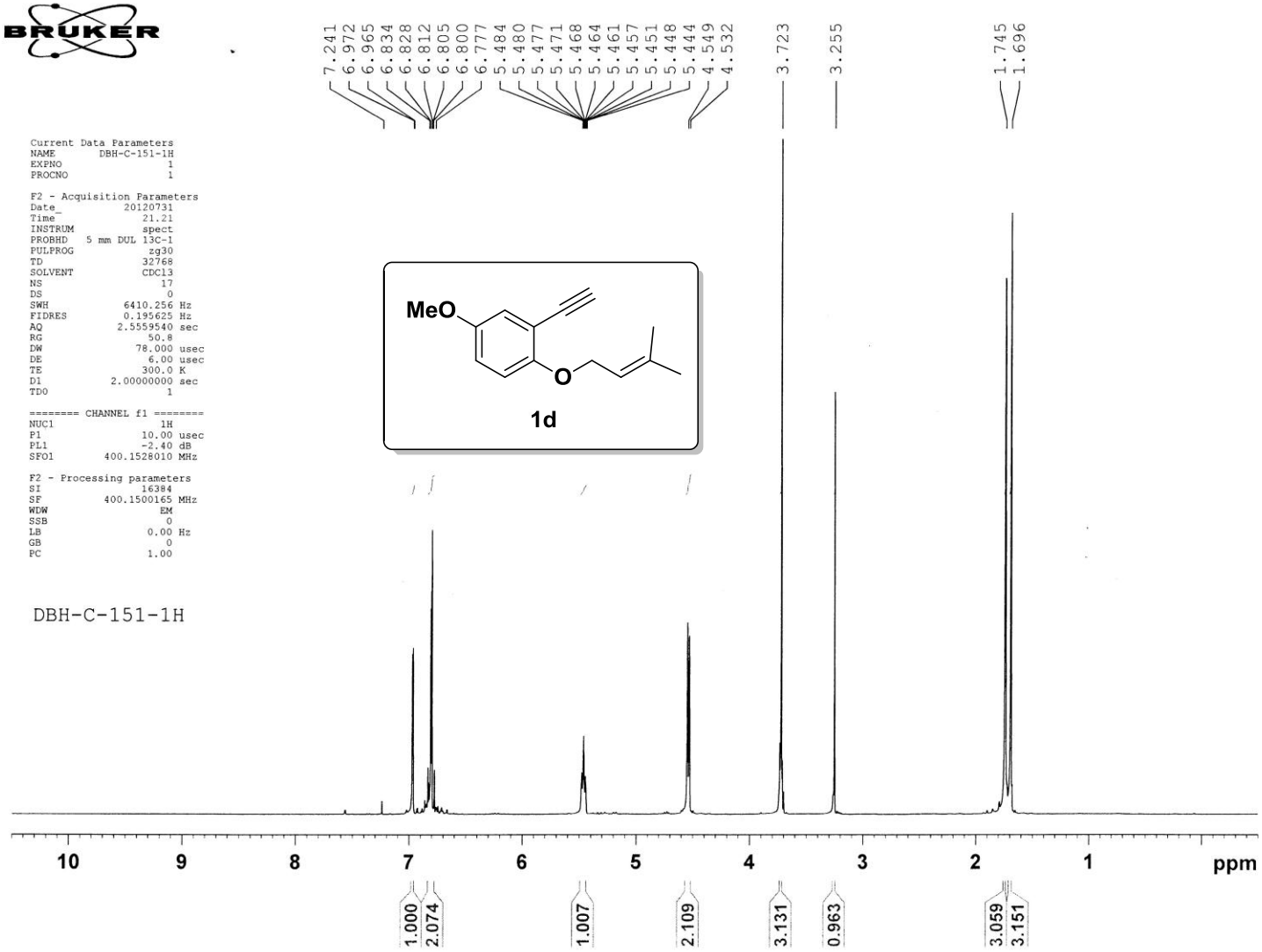

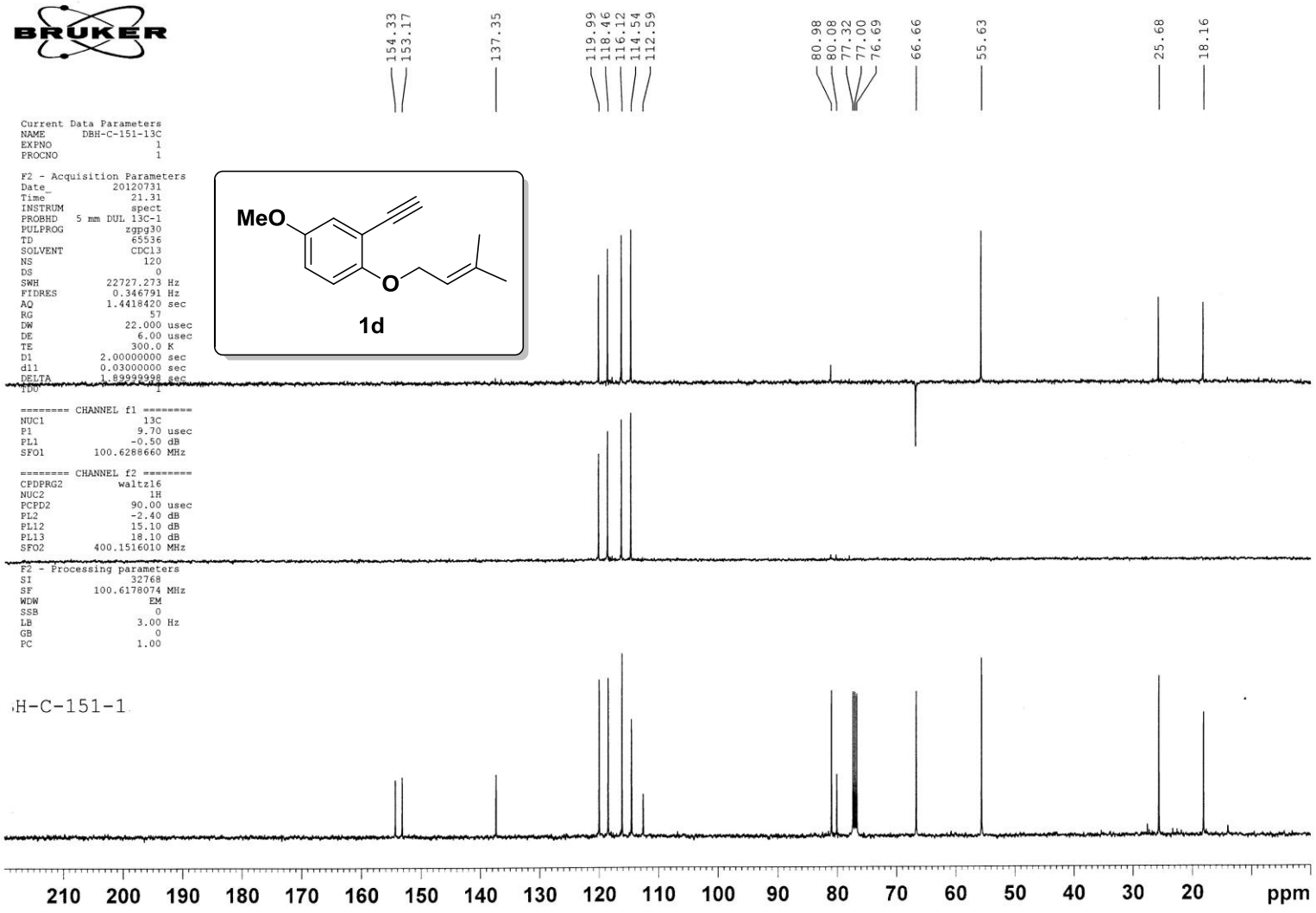

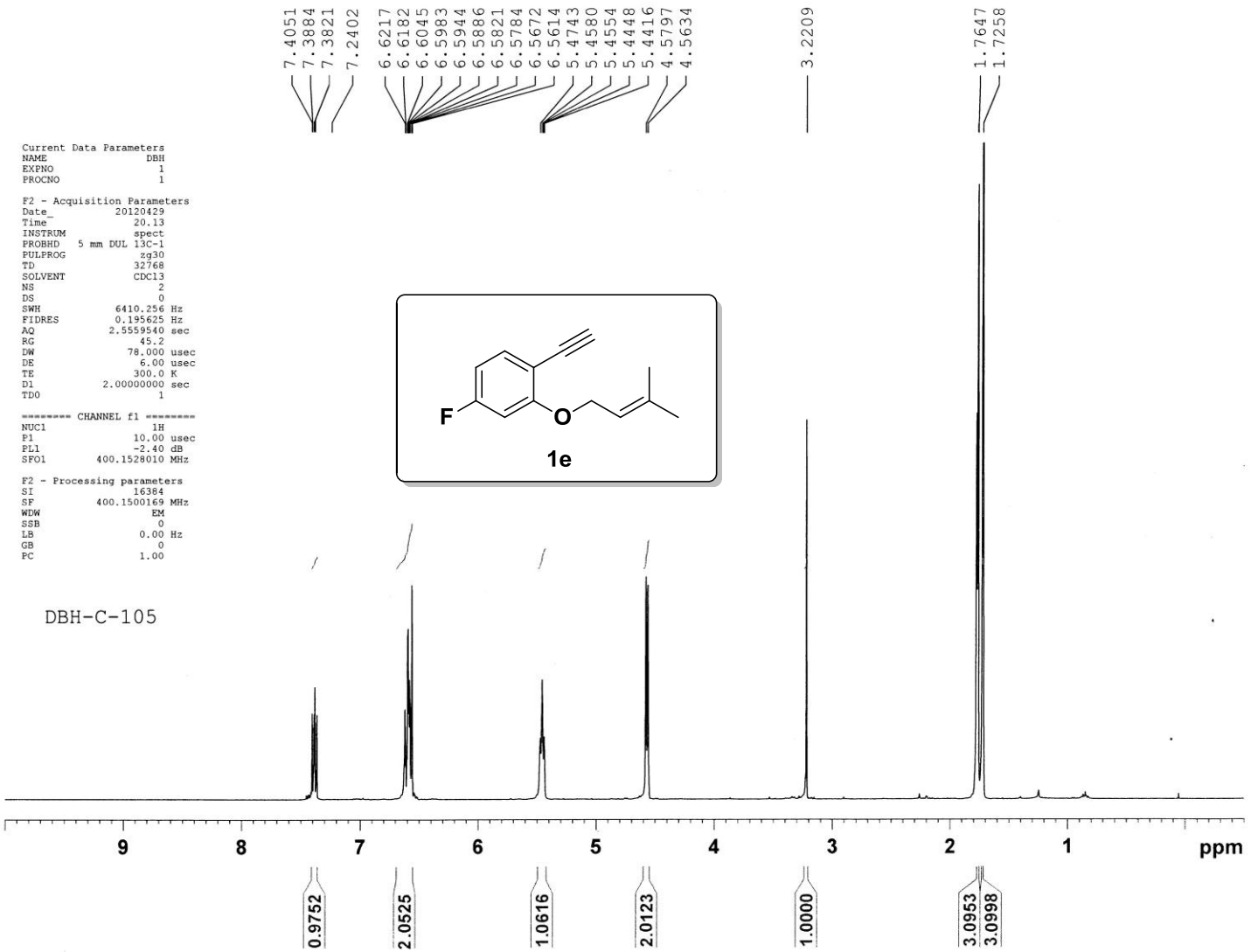

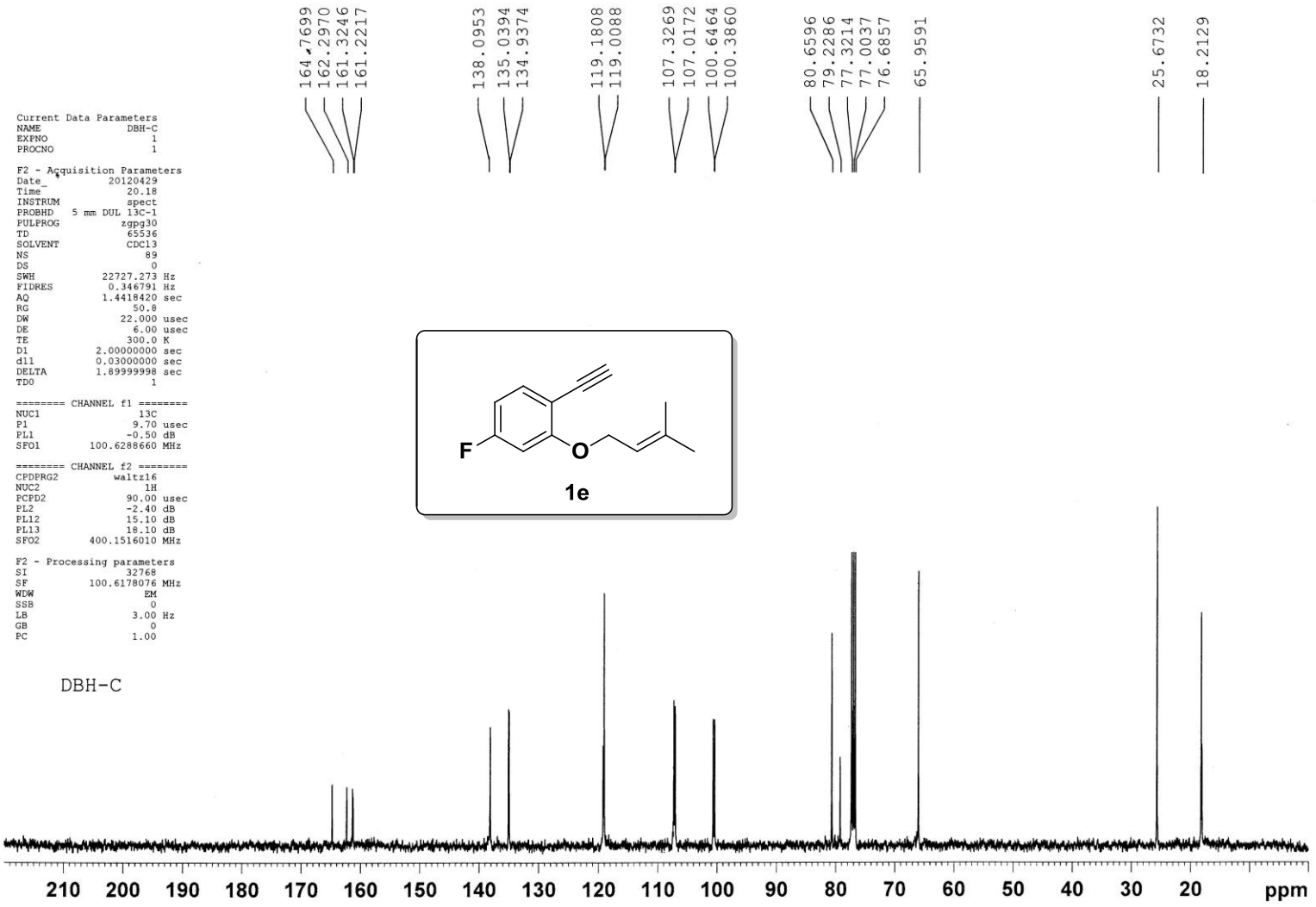

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PROCNO                  1

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PULPROG                zg
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              8389.262 Hz
FIDRES           0.256020 Hz
AQ               1.9530228 sec
RG                     32
DW                59.600 usec
DE                 6.50 usec
TE                296.9 K
D1             2.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  9.80 usec
PL1                 0.00 dB
SFO1          598.9029945 MHz

F2 - Processing parameters
SI                  32768
SF            598.9000256 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  20.00 cm
CY                  20.00 cm
F1P                10.000 ppm
F1                 5989.00 Hz
F2P                -0.500 ppm
F2                 -299.45 Hz
PPMCM              0.52500 ppm/cm
HZCM             314.42252 Hz/cm
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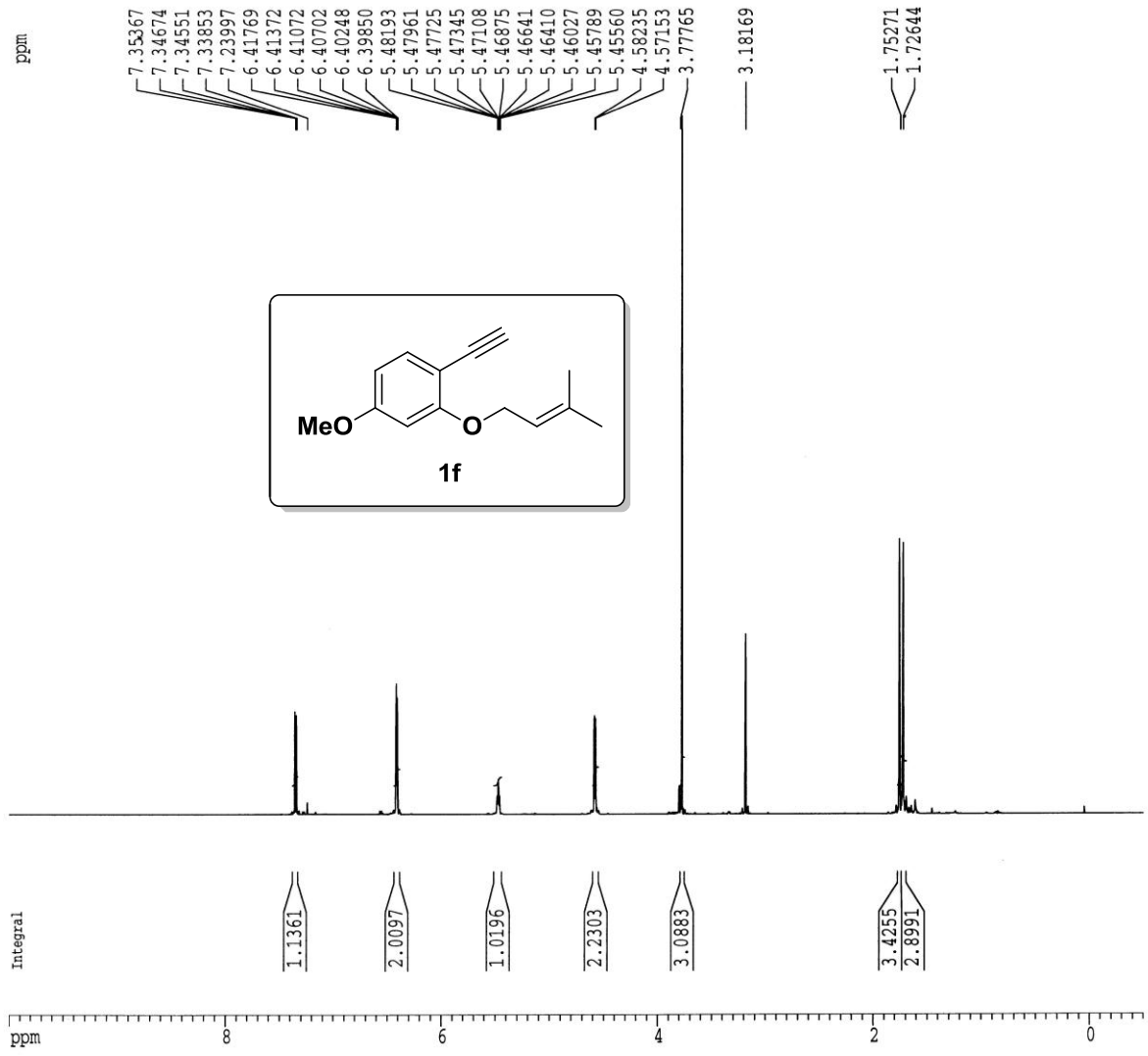

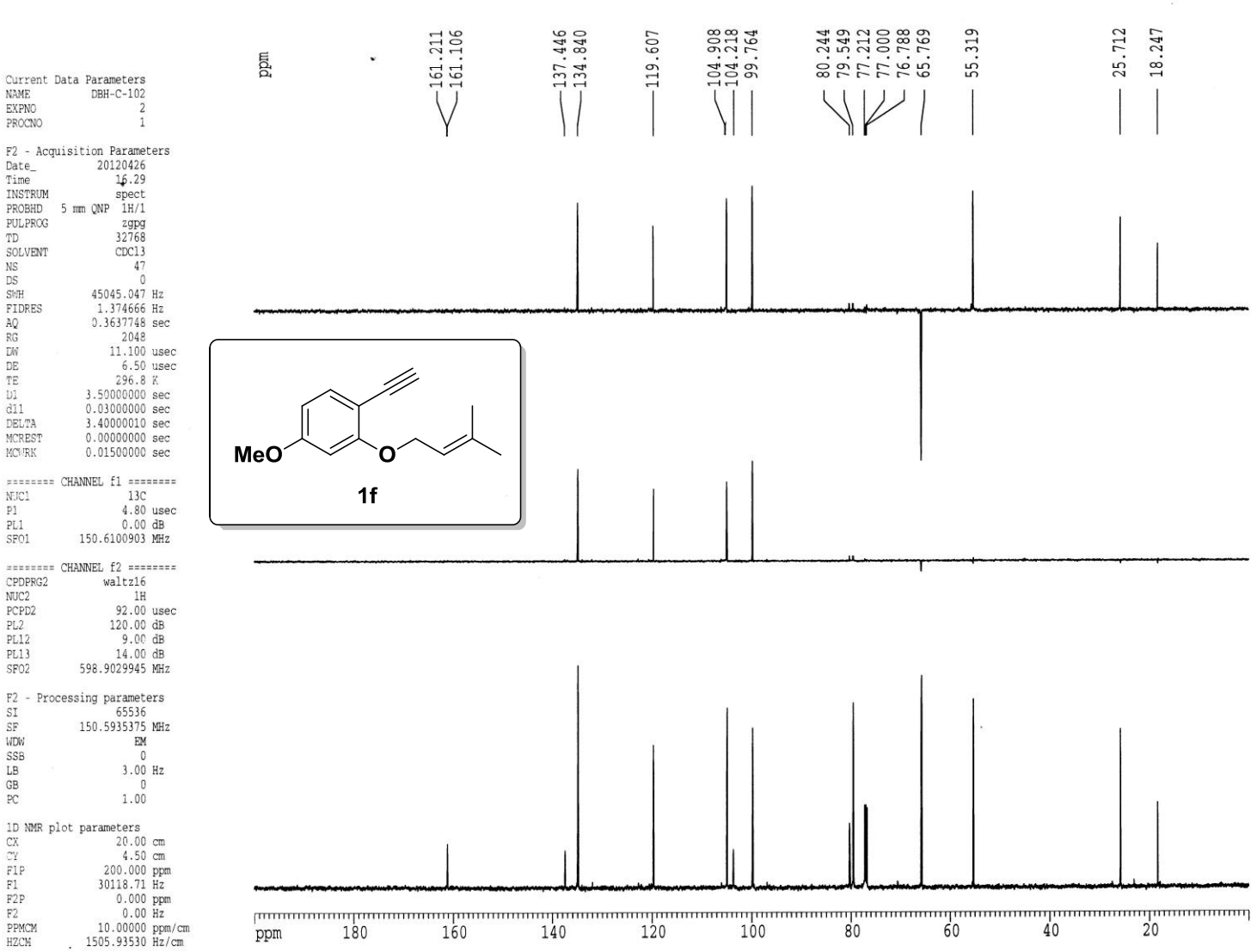

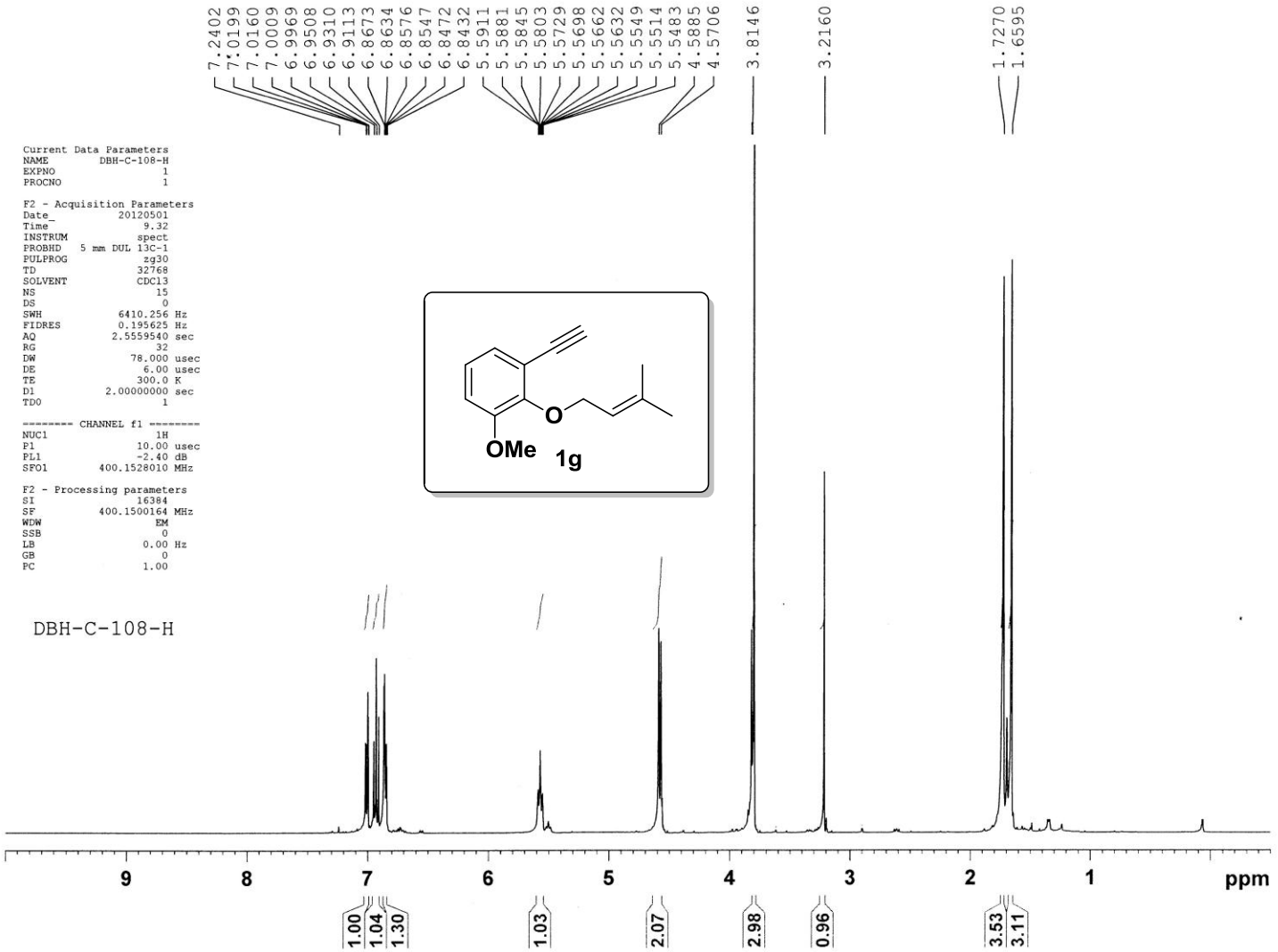

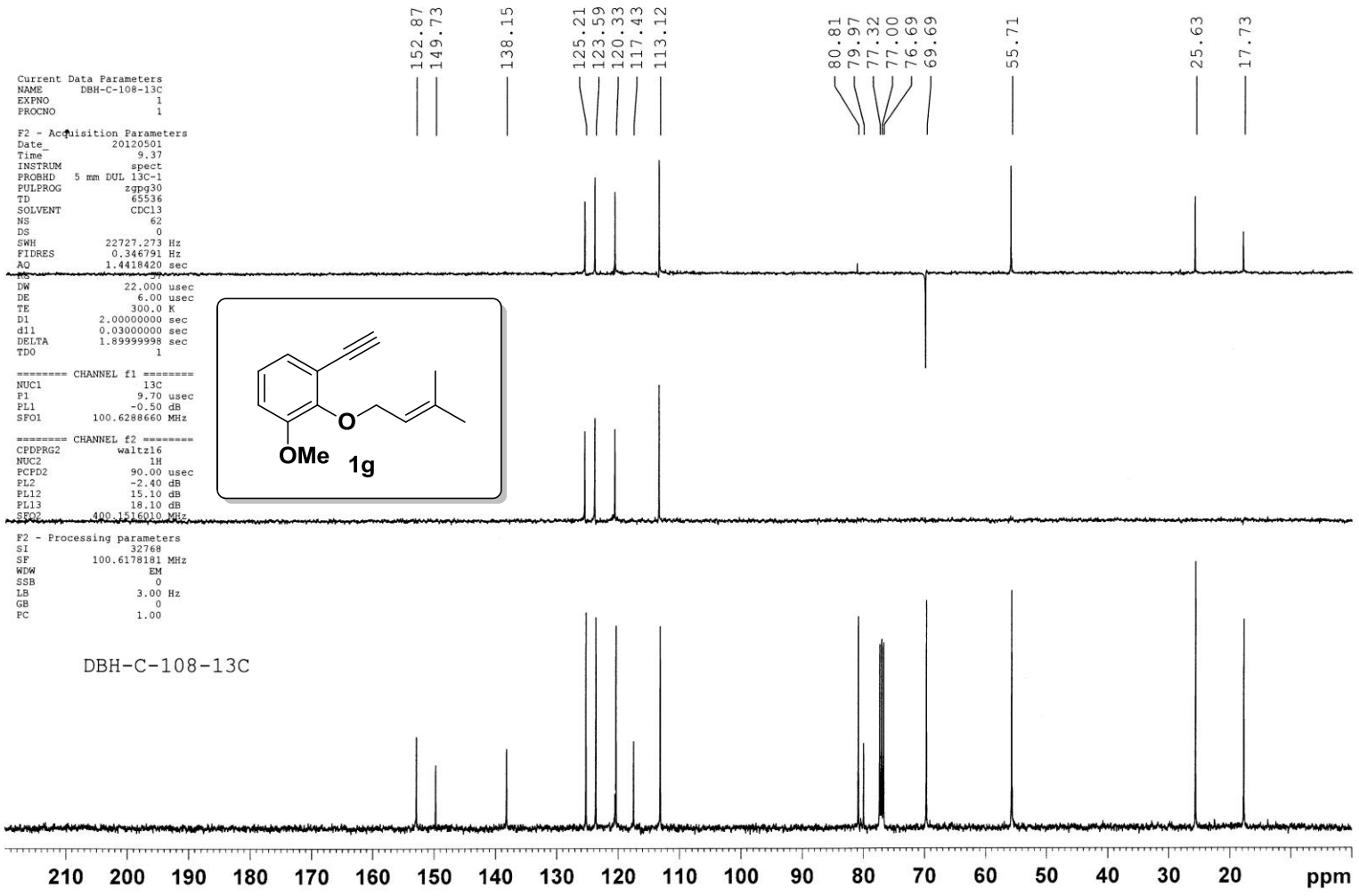

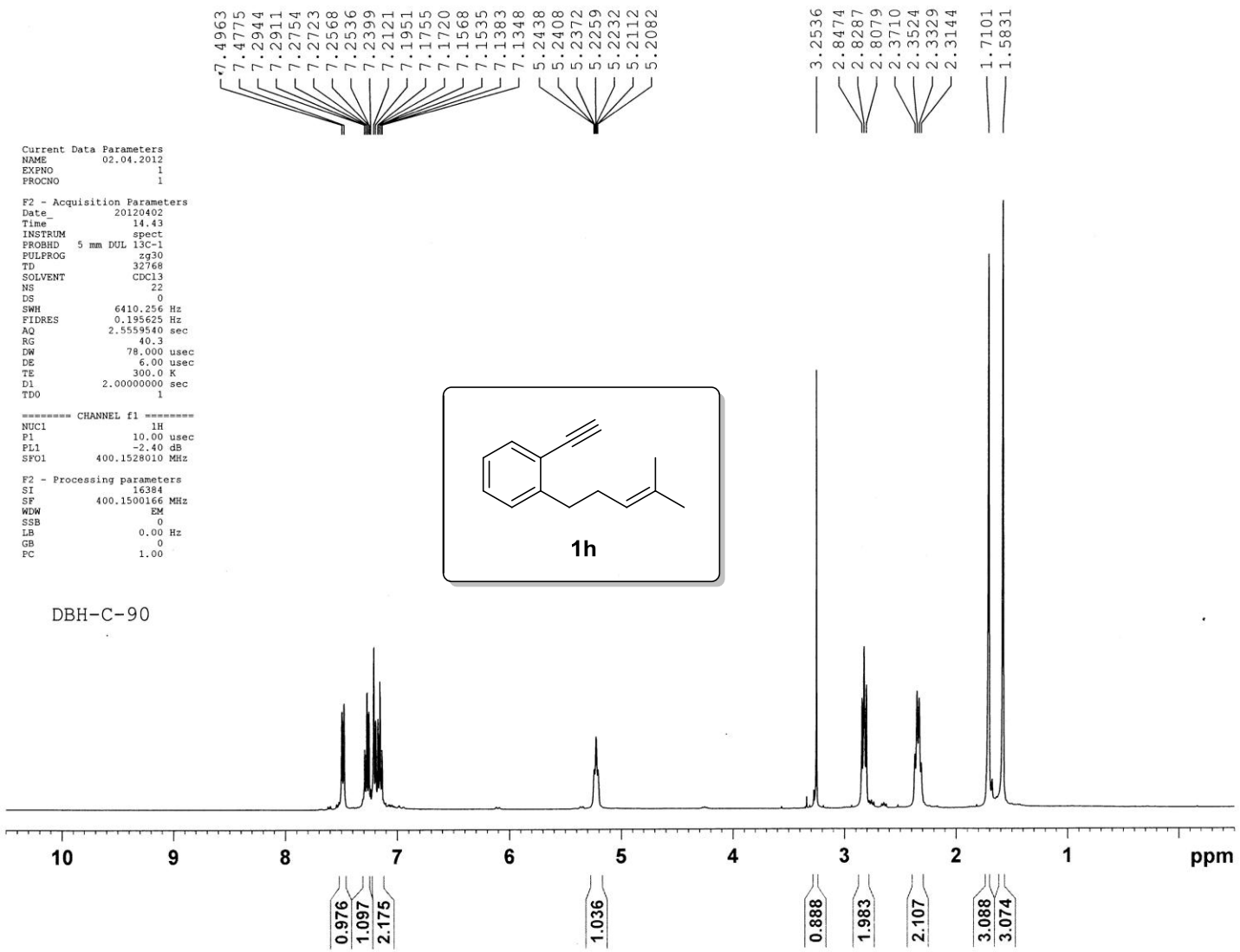

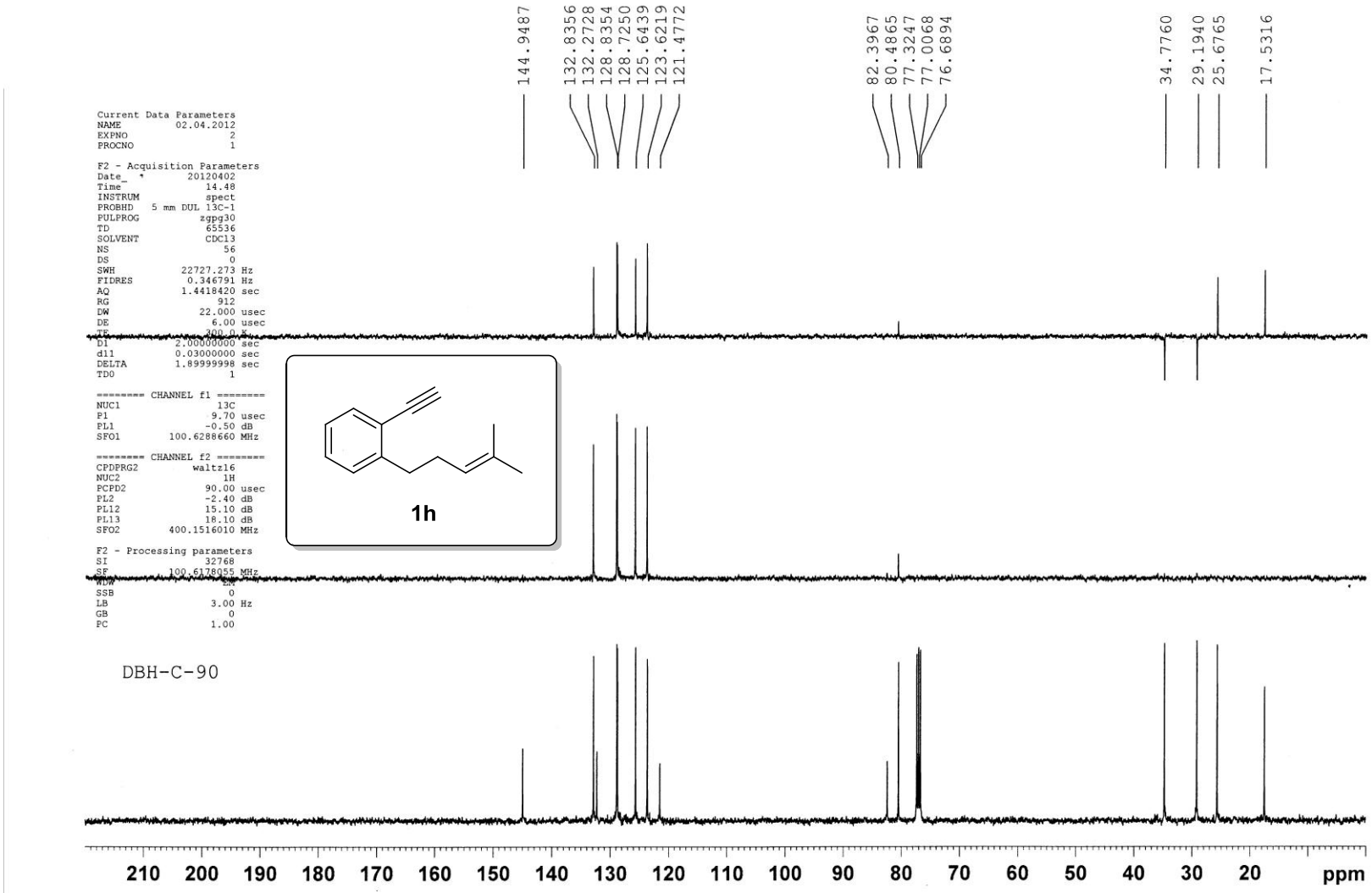

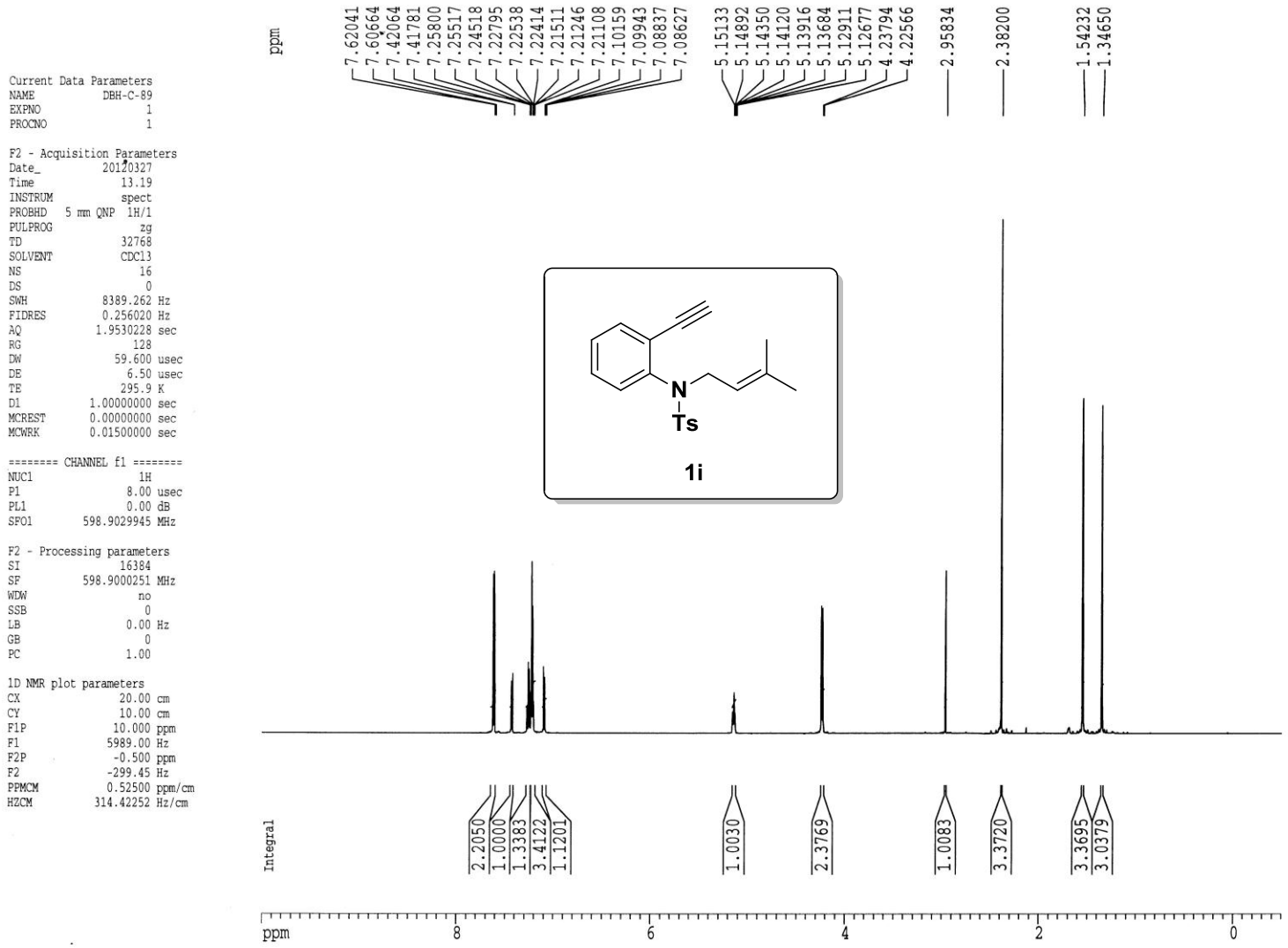

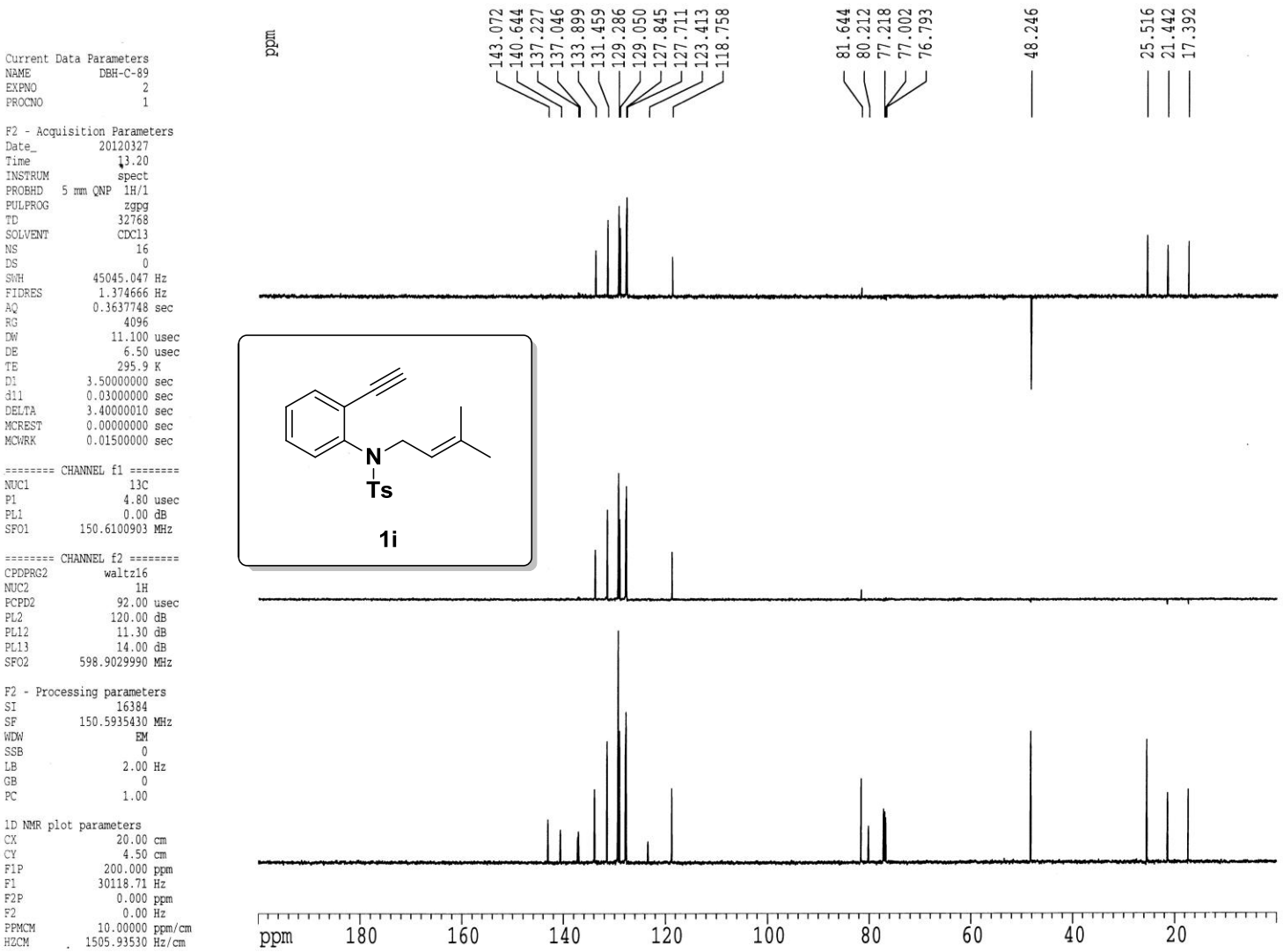

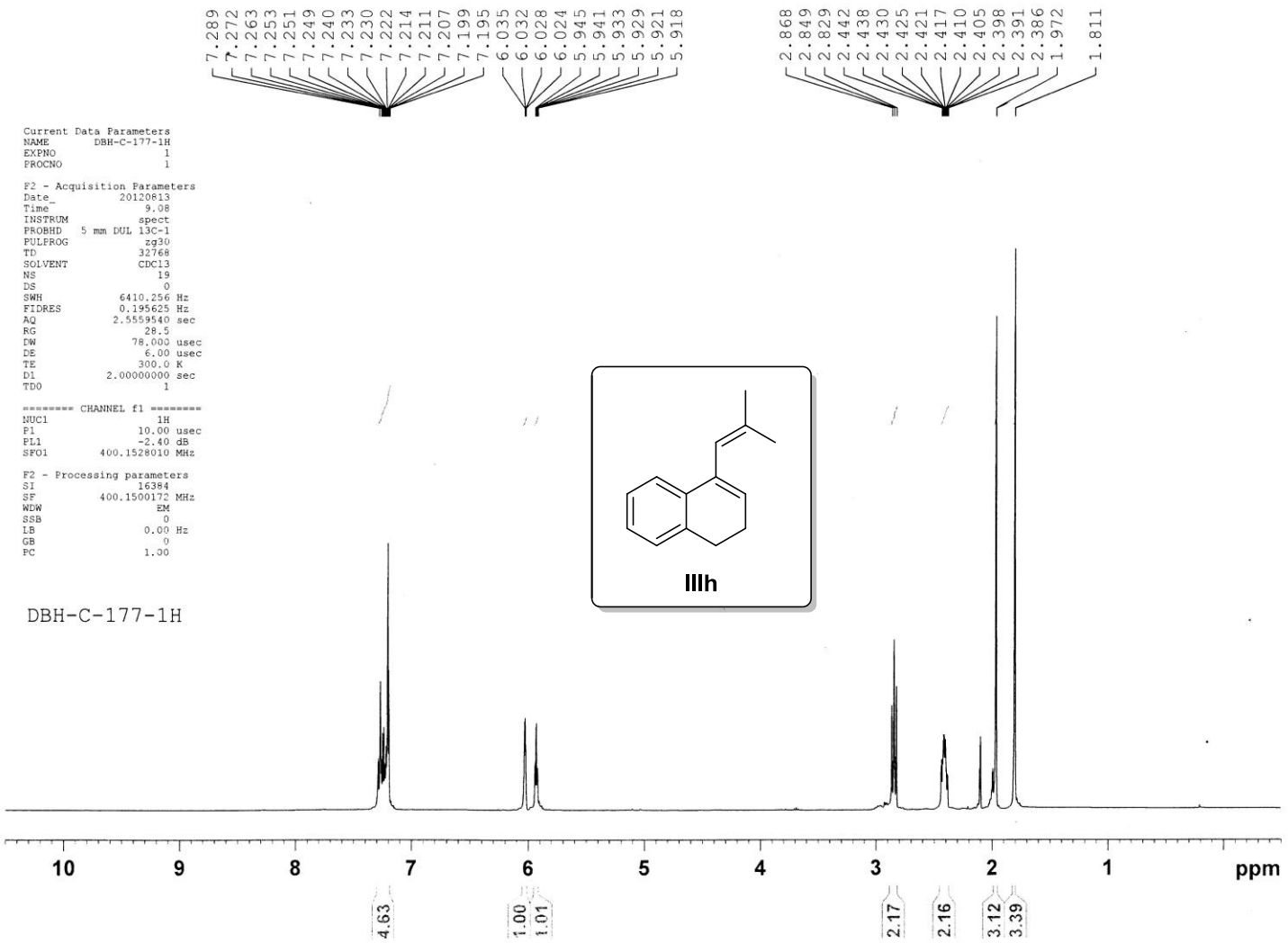

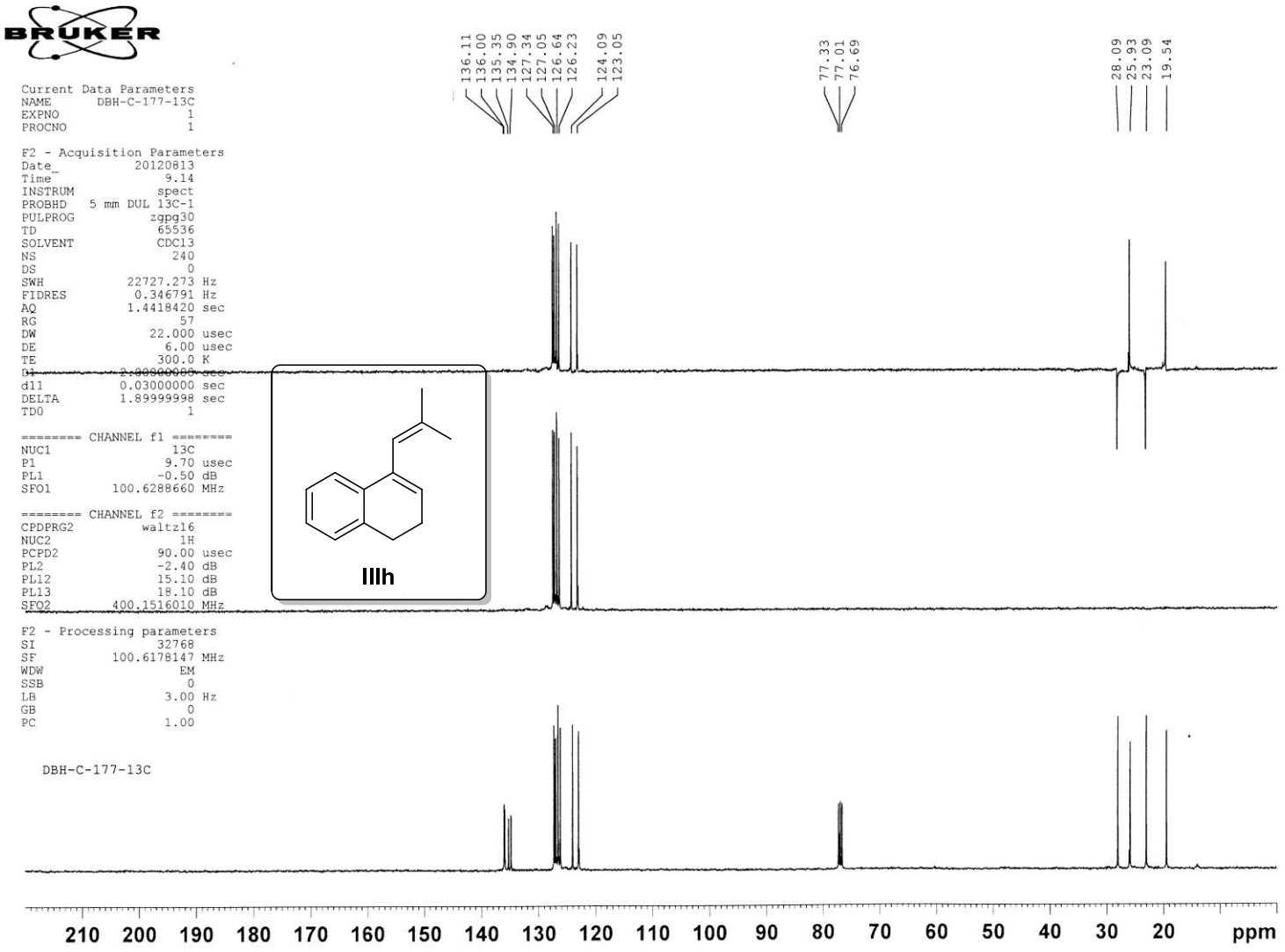

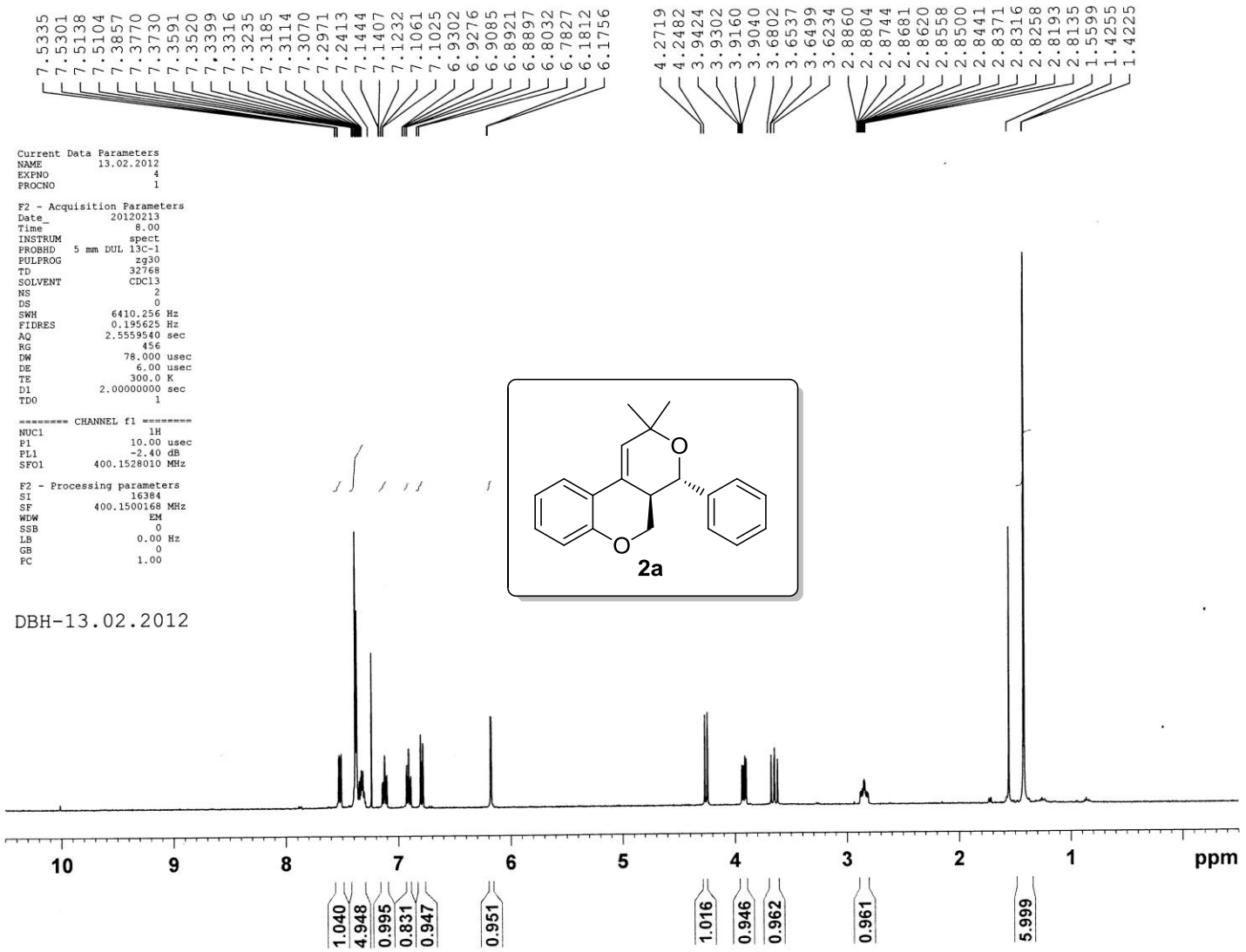

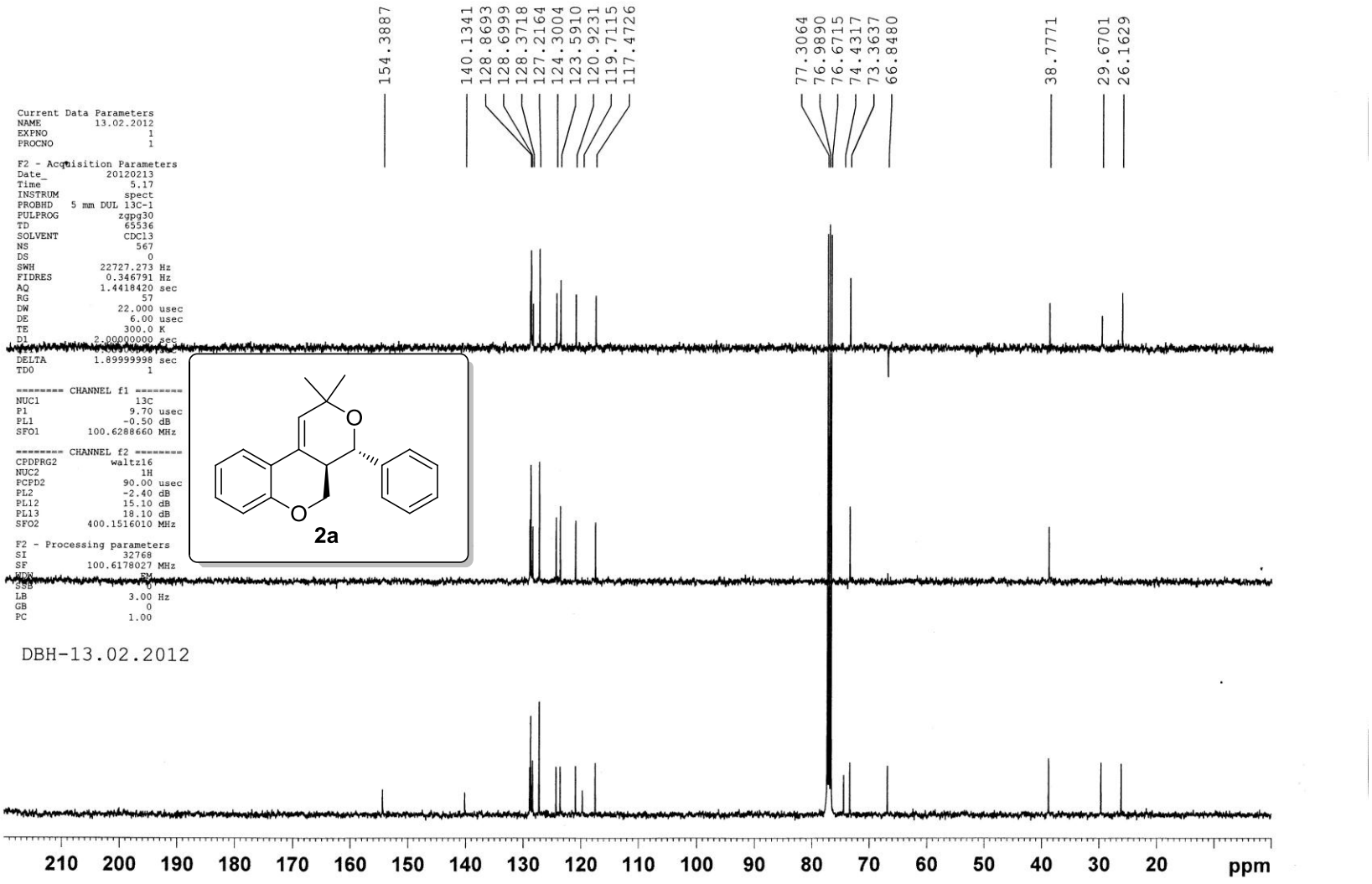

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TD                   32768
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SWH               8389.262 Hz
FIDRES            0.256020 Hz
AQ                1.9530228 sec
RG                     512
DW                  59.600 usec
DE                   6.50 usec
TE                  298.4 K
D1             2.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.80 usec
PL1                  0.00 dB
SFO1          598.9028168 MHz

F2 - Processing parameters
SI                   32768
SF            598.9000259 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                   20.00 cm
CY                   30.00 cm
F1P                 10.000 ppm
F1                 5989.00 Hz
F2P                 -0.500 ppm
F2                 -299.45 Hz
PPMCM               0.52500 ppm/cm
HZCM              314.42252 Hz/cm
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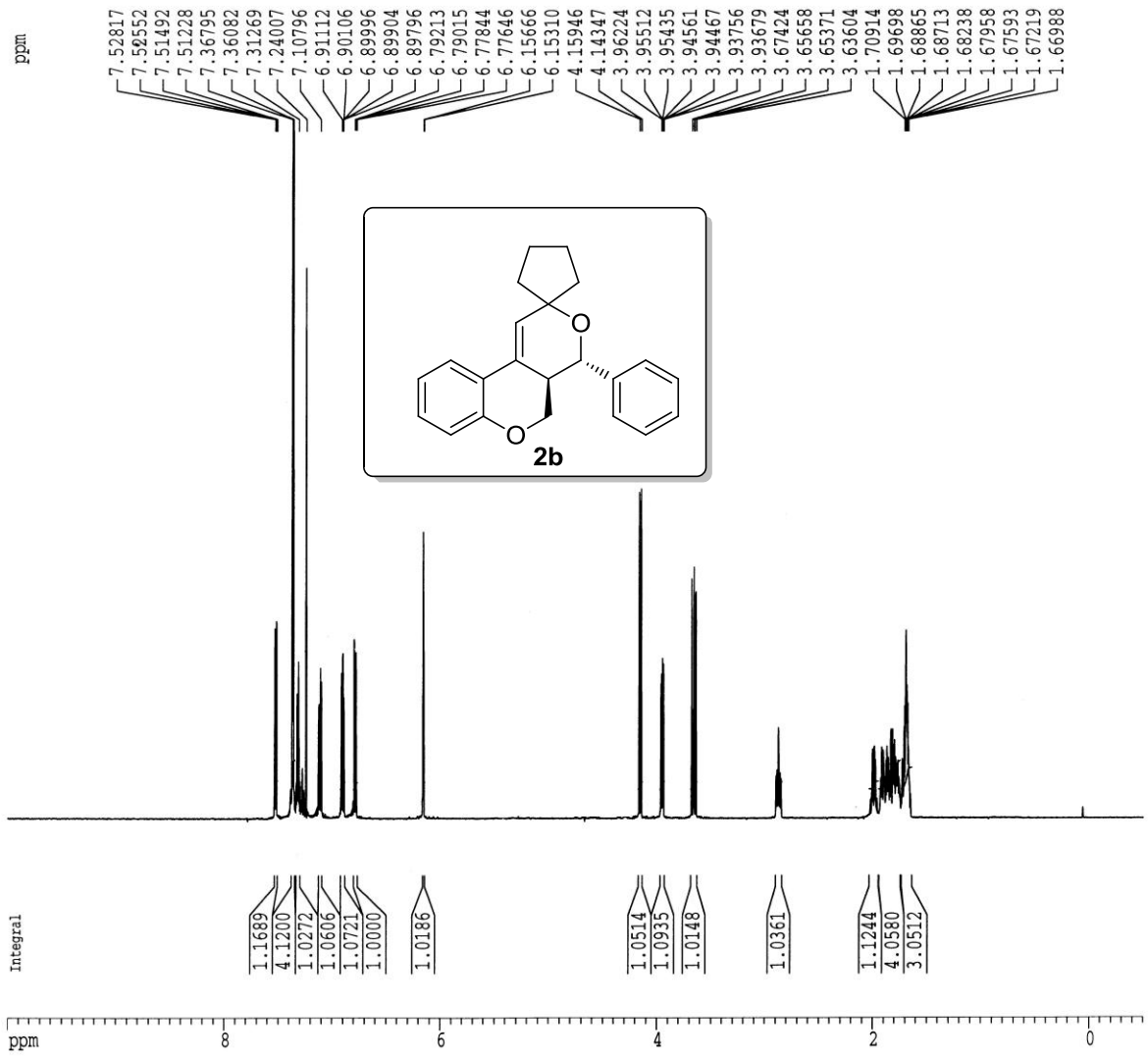
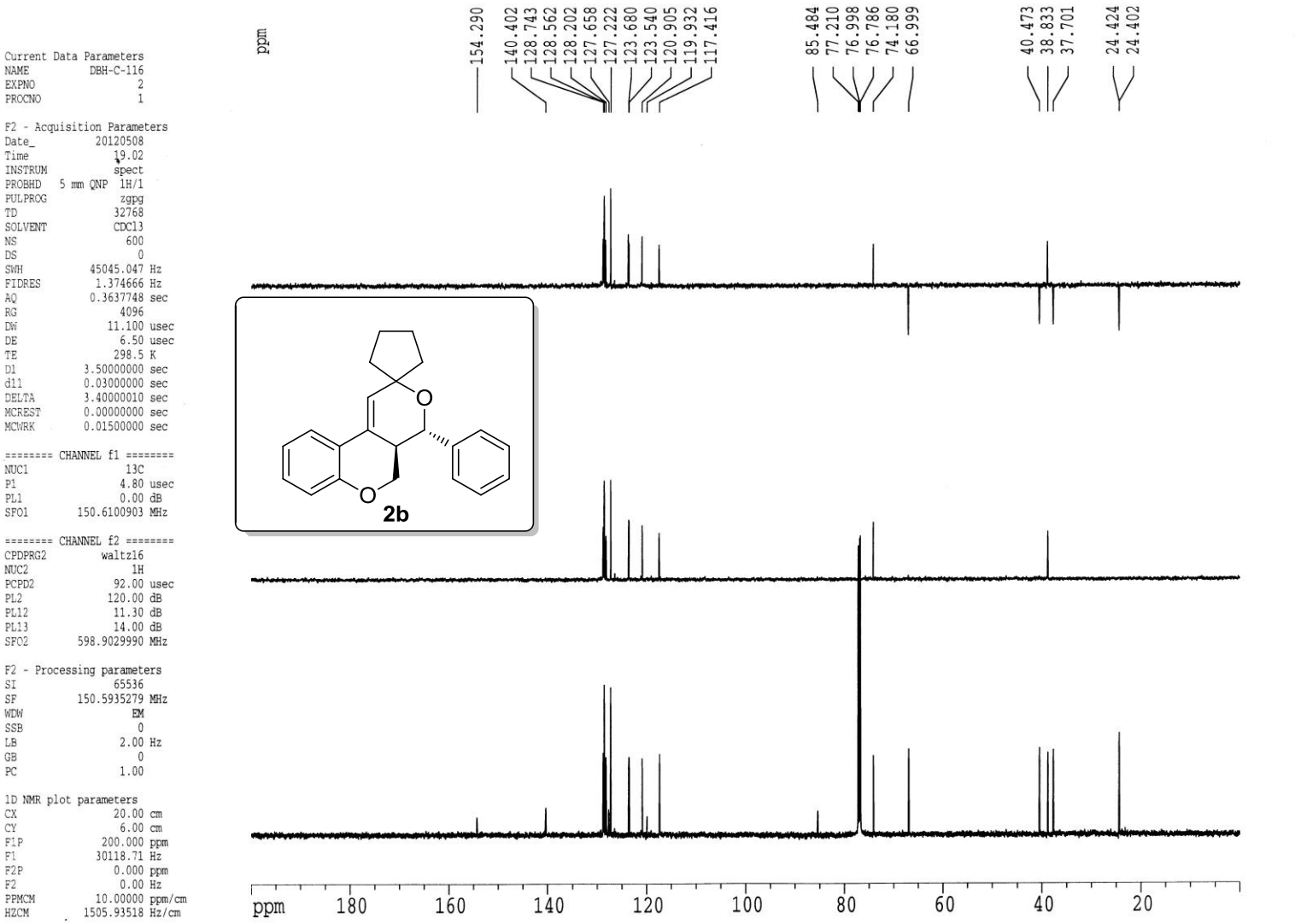
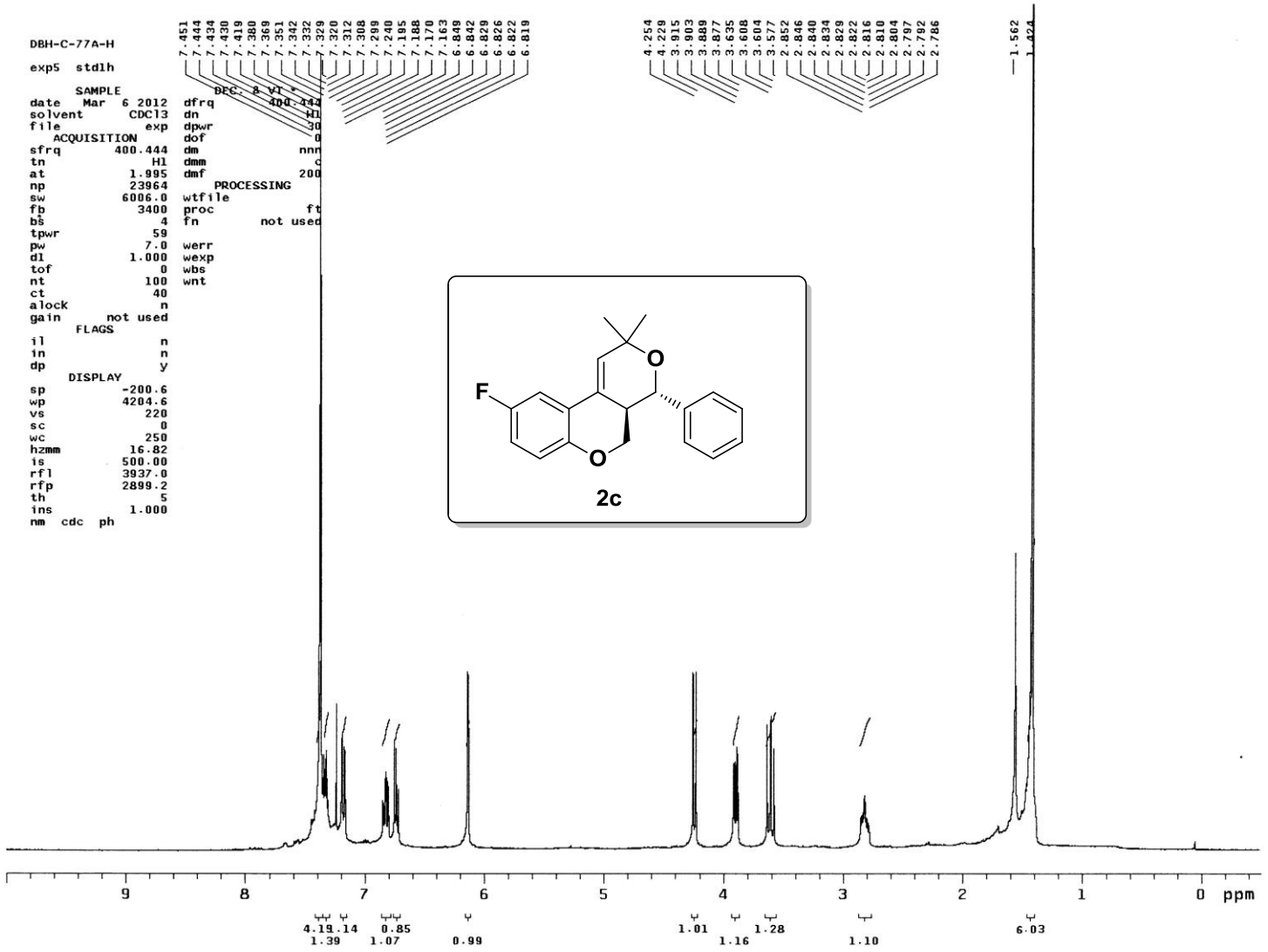
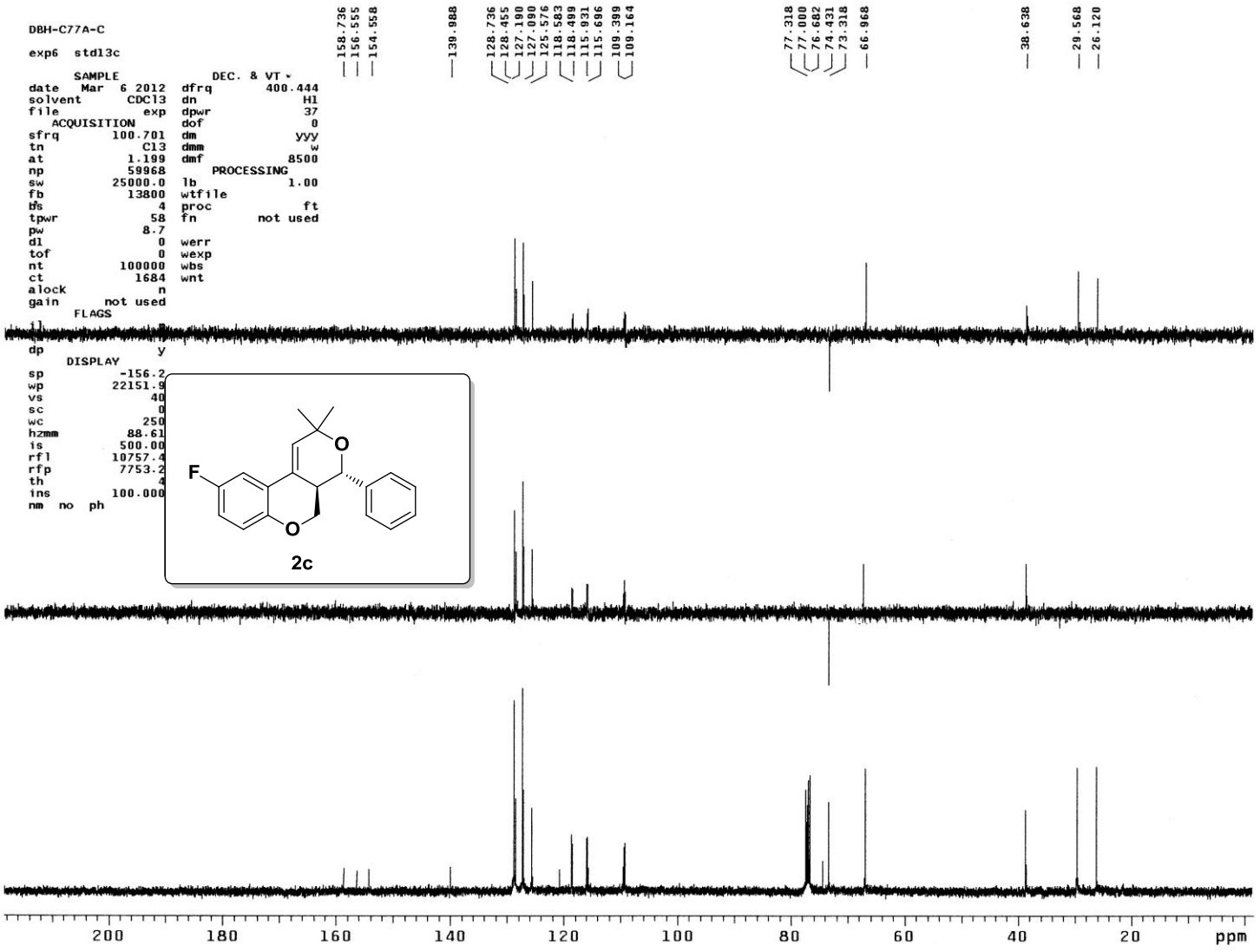
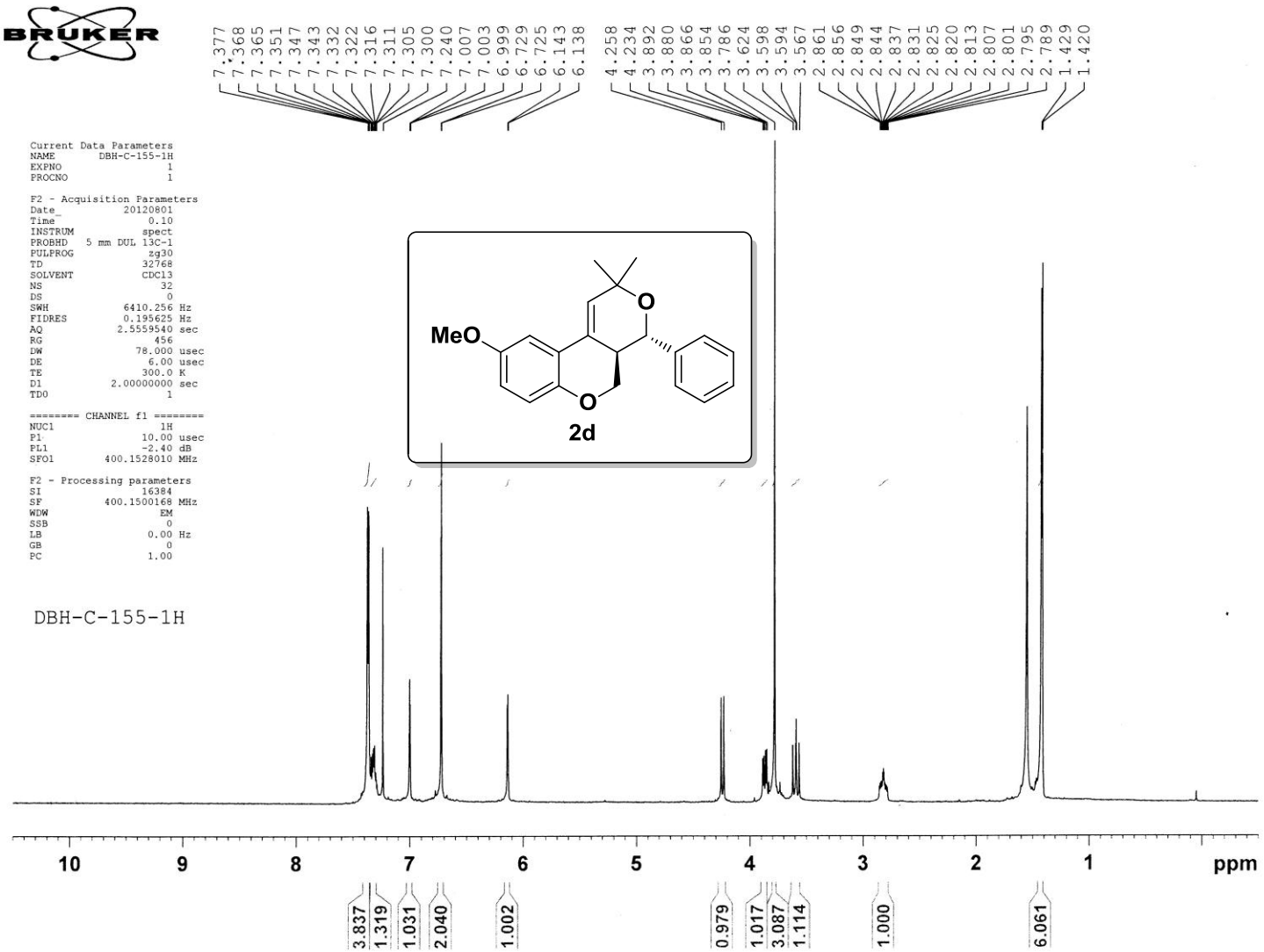
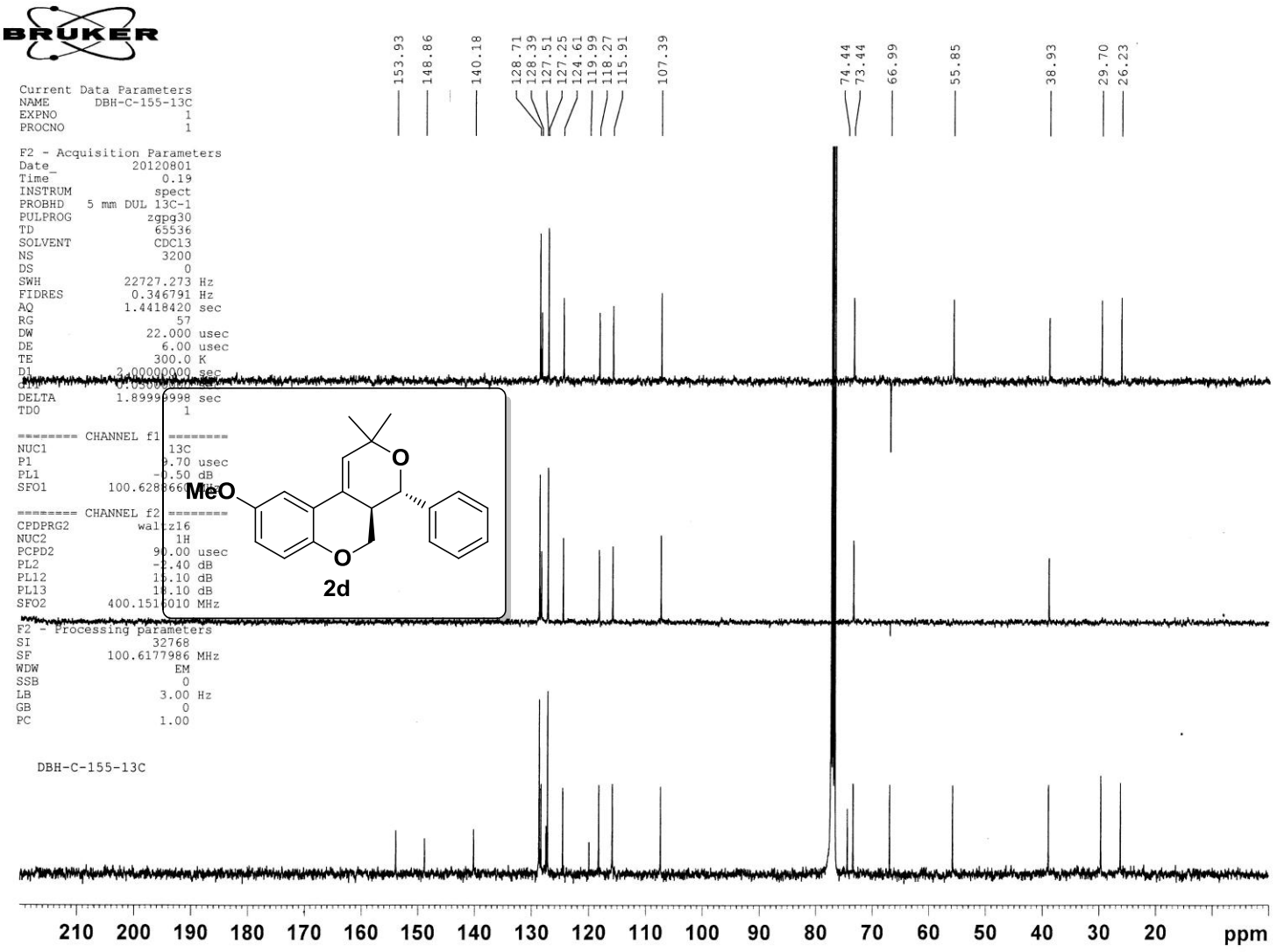




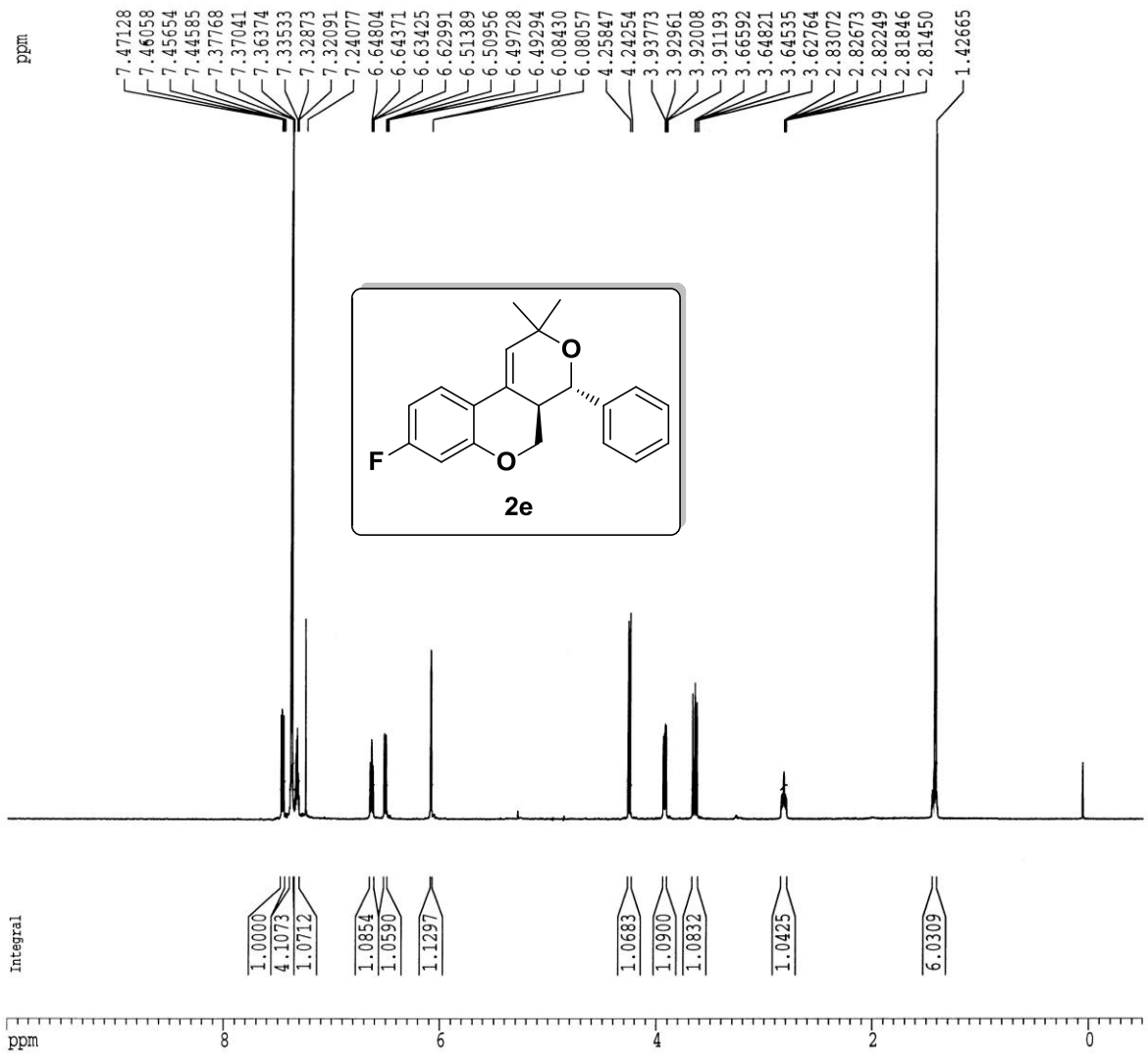




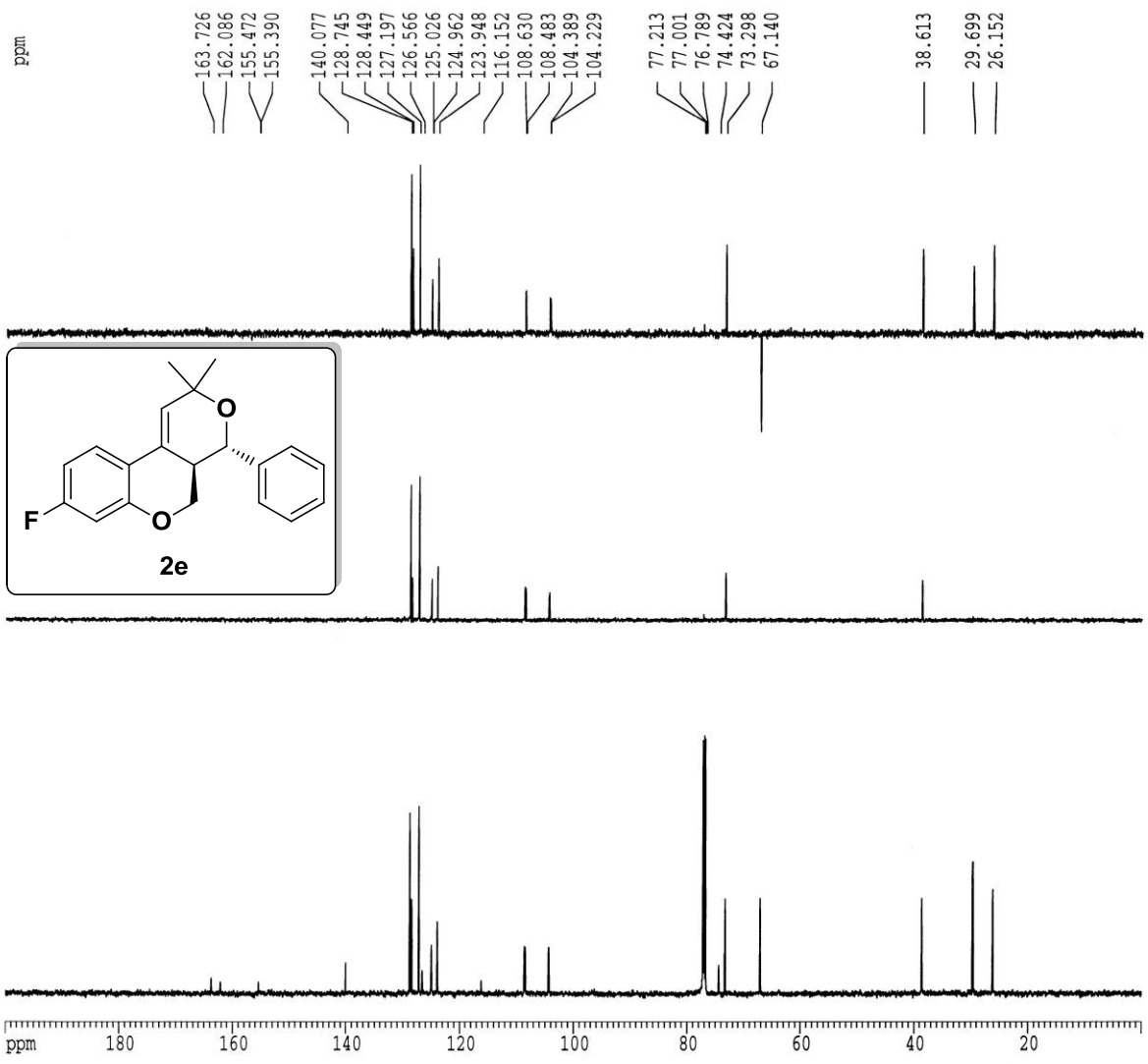


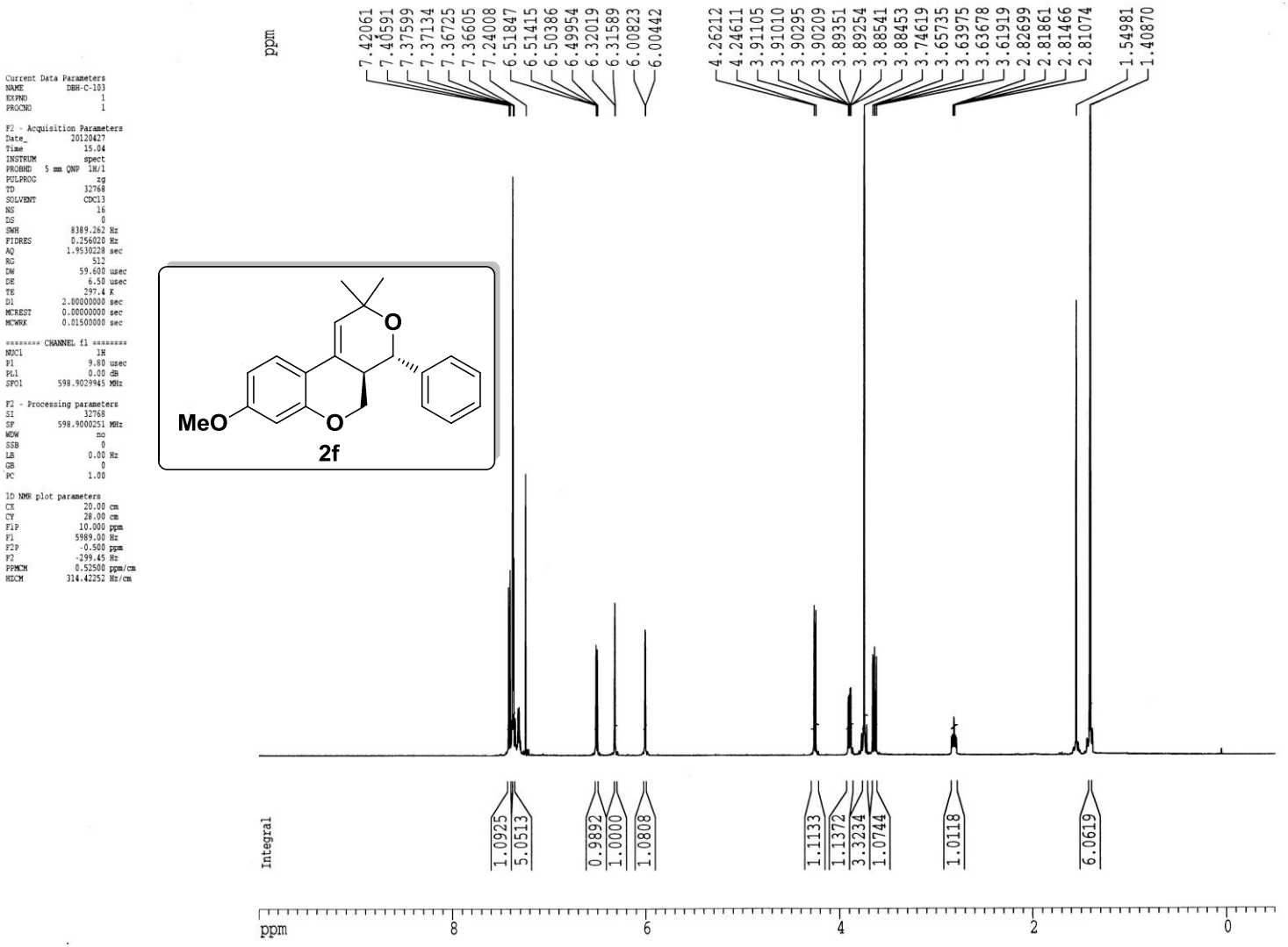


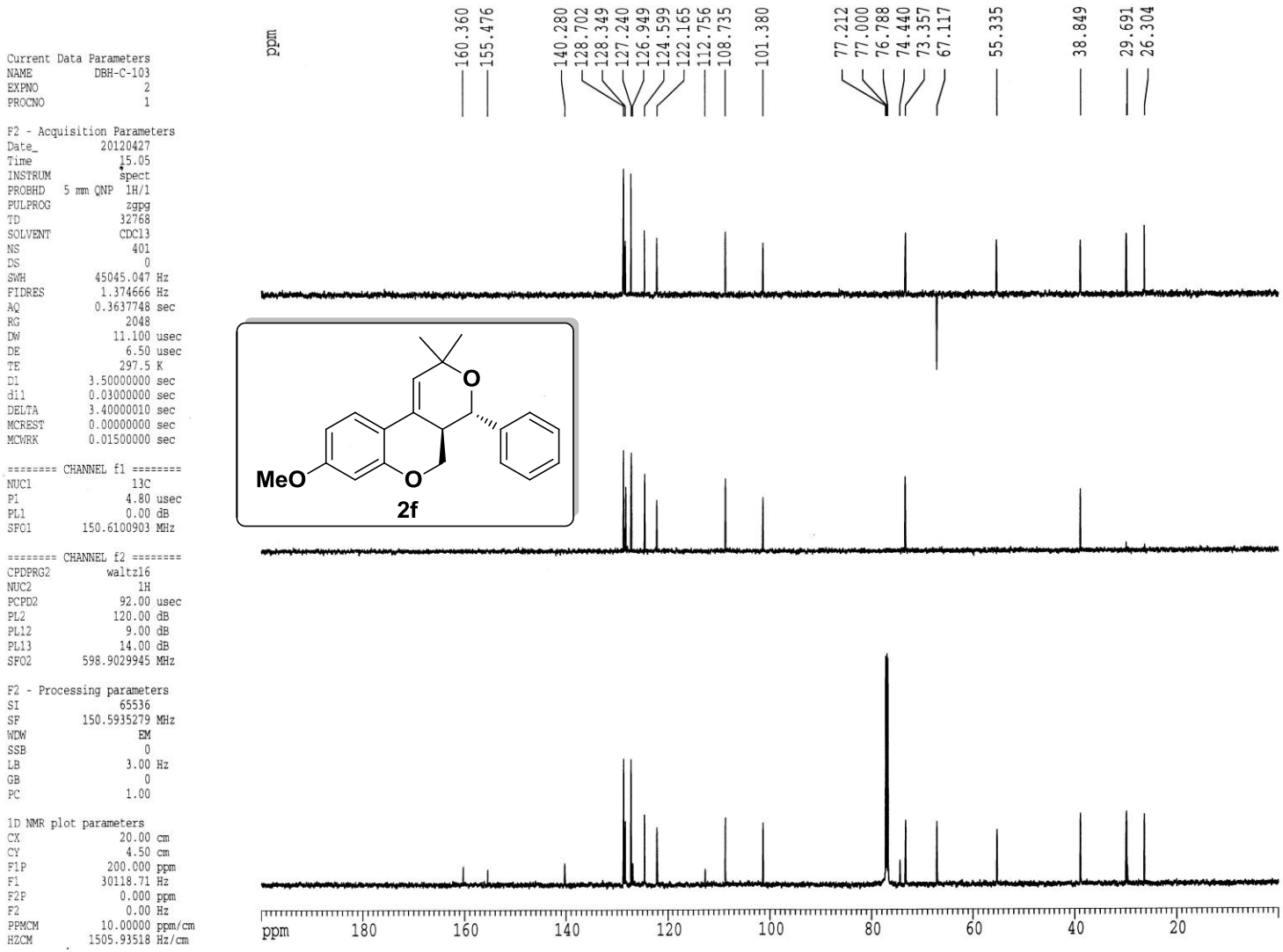
```
Current Data Parameters
NAME                 DBH-C-107
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_               20120429
Time                    16.57
INSTRUM                 spect
PROBHD   5 mm QNP  1H/1
PULPROG                 zg
TD                      32768
SOLVENT                 CDCl3
NS                         16
DS                          0
SWH                8389.262 Hz
FIDRES                0.256020 Hz
AQ                 1.9530238 sec
RG                         32
DW                    59.600 usec
DE                     6.50 usec
TE                     297.8 K
D1                 2.00000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                      9.80 usec
PL1                     0.00 dB
SFO1               598.9029945 MHz

F2 - Processing parameters
SI                      32768
SF                 598.9000246 MHz
WDW                        no
SSB                         0
LB                      0.00 Hz
GB                          0
PC                      1.00

1D NMR plot parameters
CX                     20.00 cm
CY                     20.00 cm
F1P                   10.000 ppm
F1                   5989.00 Hz
F2P                   -0.500 ppm
F2                   -299.45 Hz
PPMCM                 0.52500 ppm/cm
HZCM                314.42252 Hz/cm
```



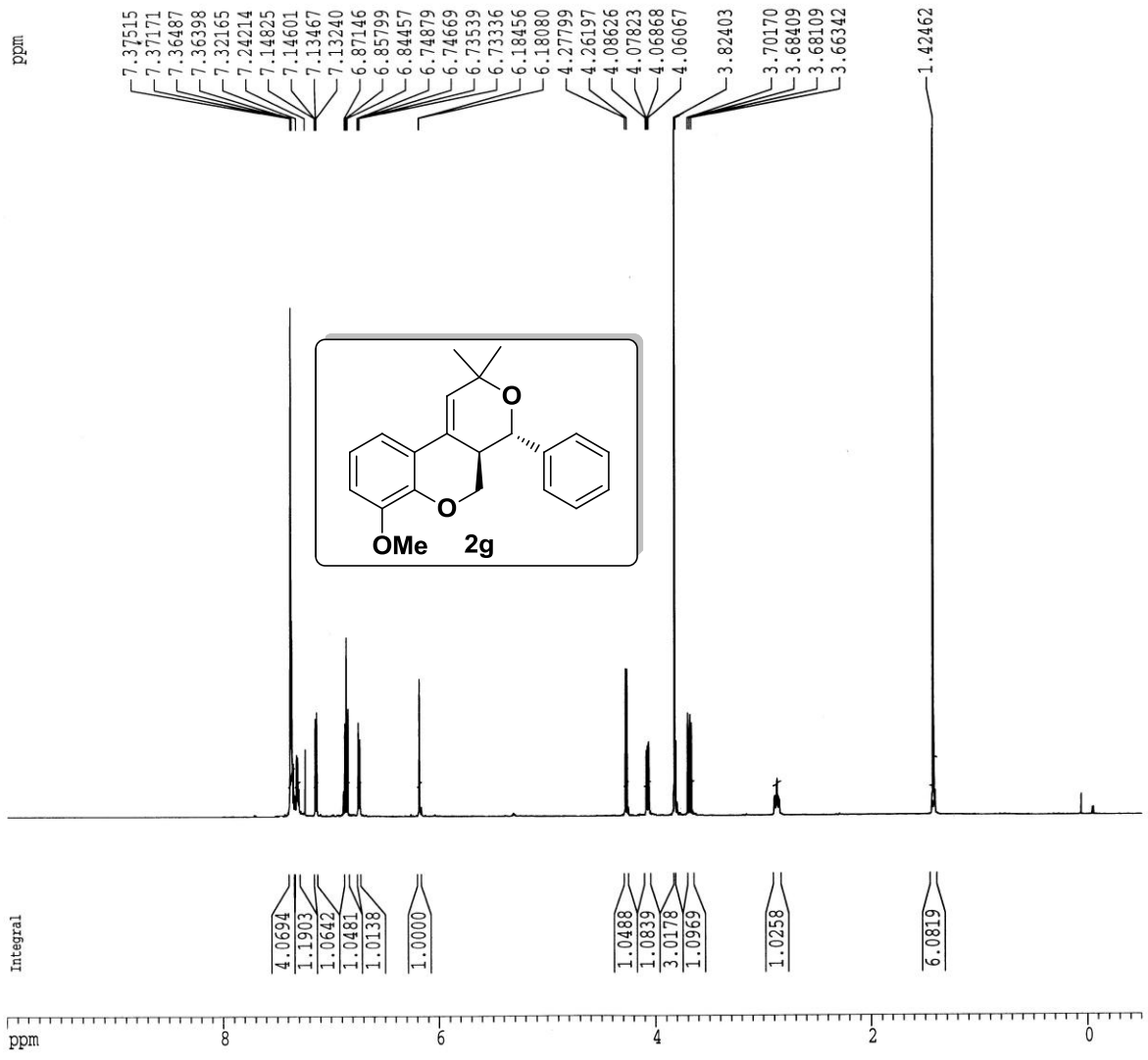
```
Current Data Parameters
NAME          DBH-C-107
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20120429
Time              17.25
INSTRUM           spect
PROBHD   5 mm QNP  1H/1
PULPROG           zgpg
TD                32768
SOLVENT           CDCl3
NS                  446
DS                    0
SWH           45045.047 Hz
FIDRES         1.374666 Hz
AQ            0.3637748 sec
RG                 2048
DW               11.100 usec
DE                6.50 usec
TE               298.9 K
D1           3.50000000 sec
d11          0.03000000 sec
DELTA        3.40000010 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                4.80 usec
PL1               0.00 dB
SFO1        150.6100903 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             92.00 usec
PL2              120.00 dB
PL12               9.00 dB
PL13              14.00 dB
SFO2        598.9029945 MHz

F2 - Processing parameters
SI                65536
SF          150.5935272 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                 4.50 cm
F1P              200.000 ppm
F1             30118.71 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM          10.00000 ppm/cm
HZCM         1505.93518 Hz/cm
```



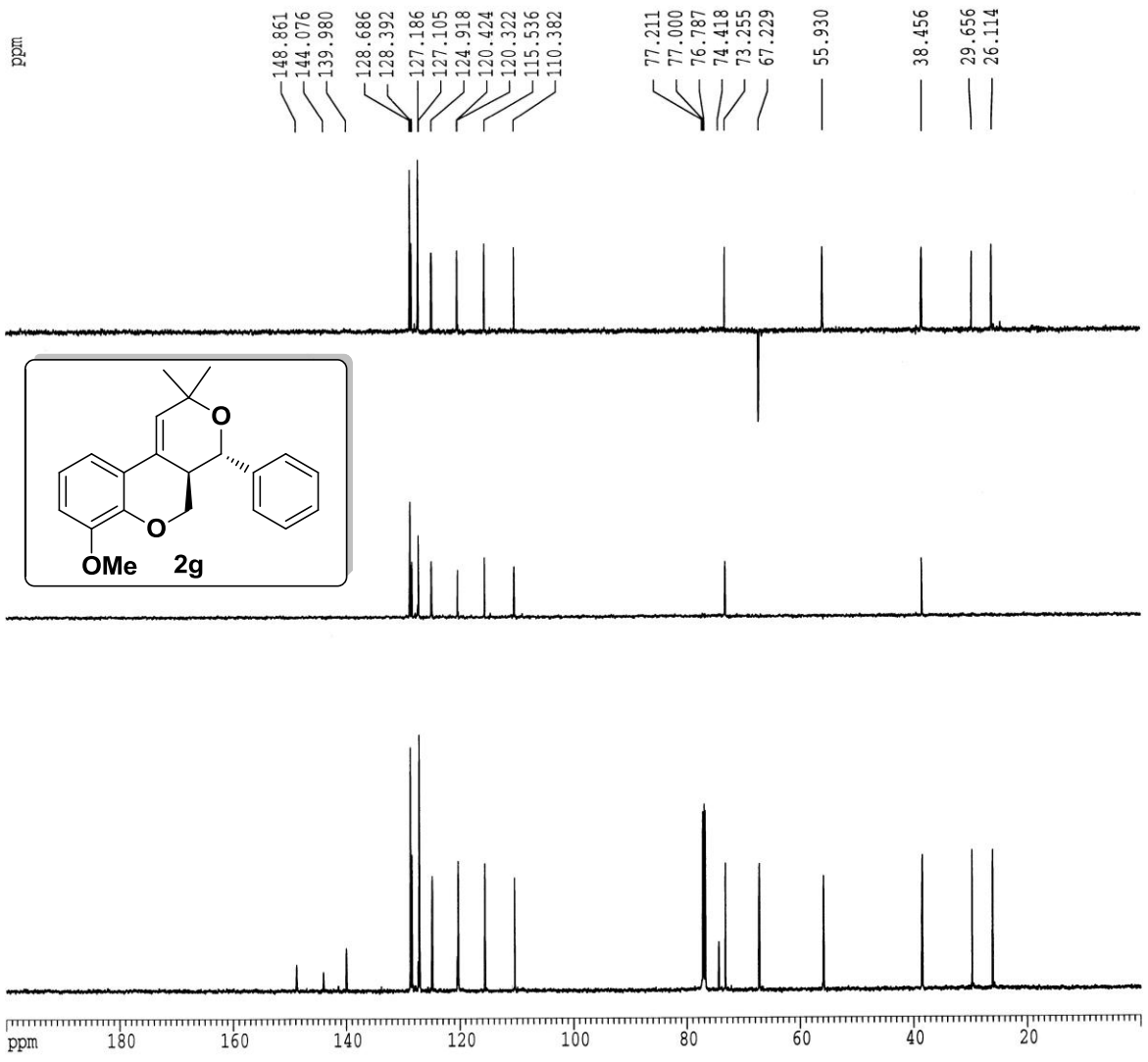


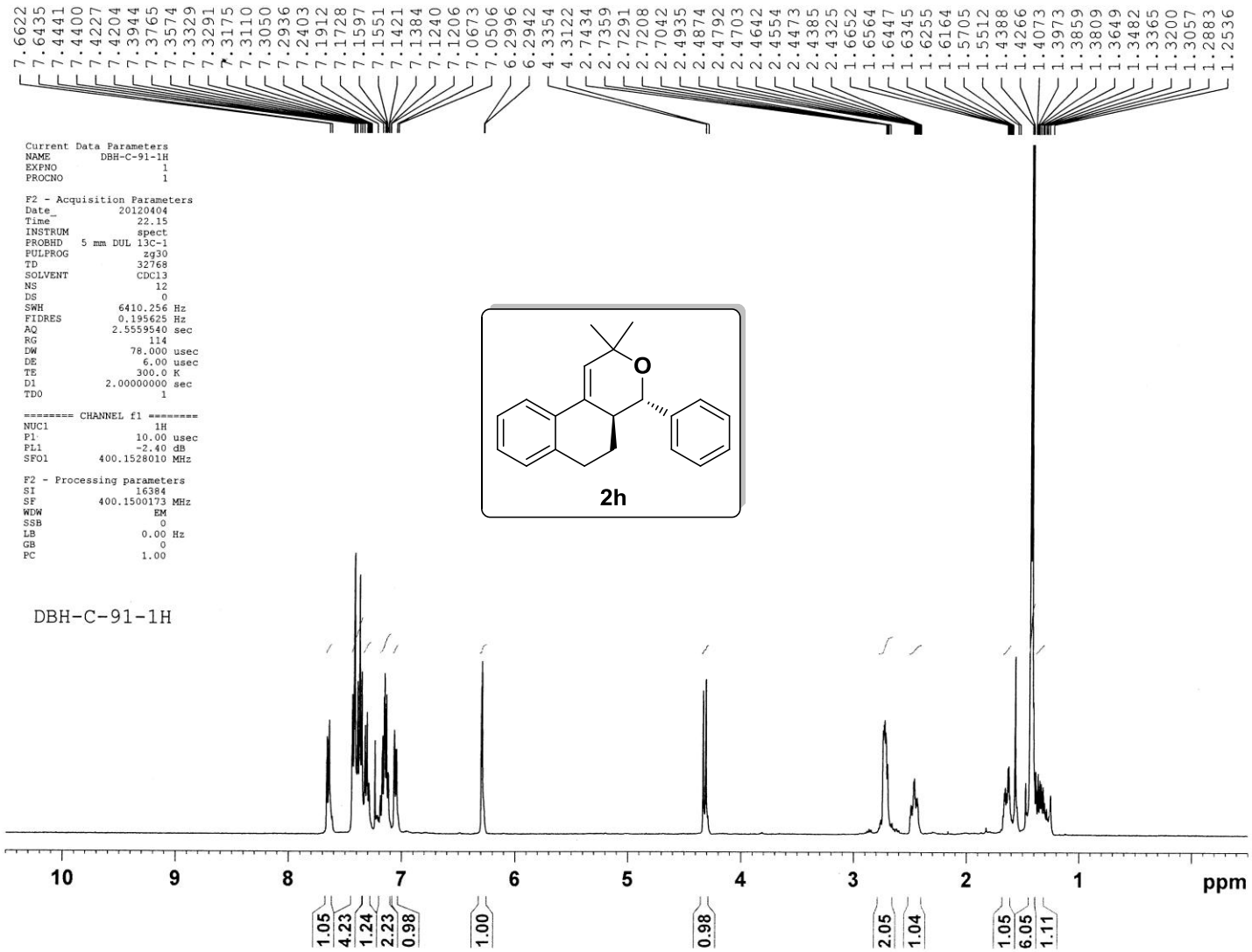


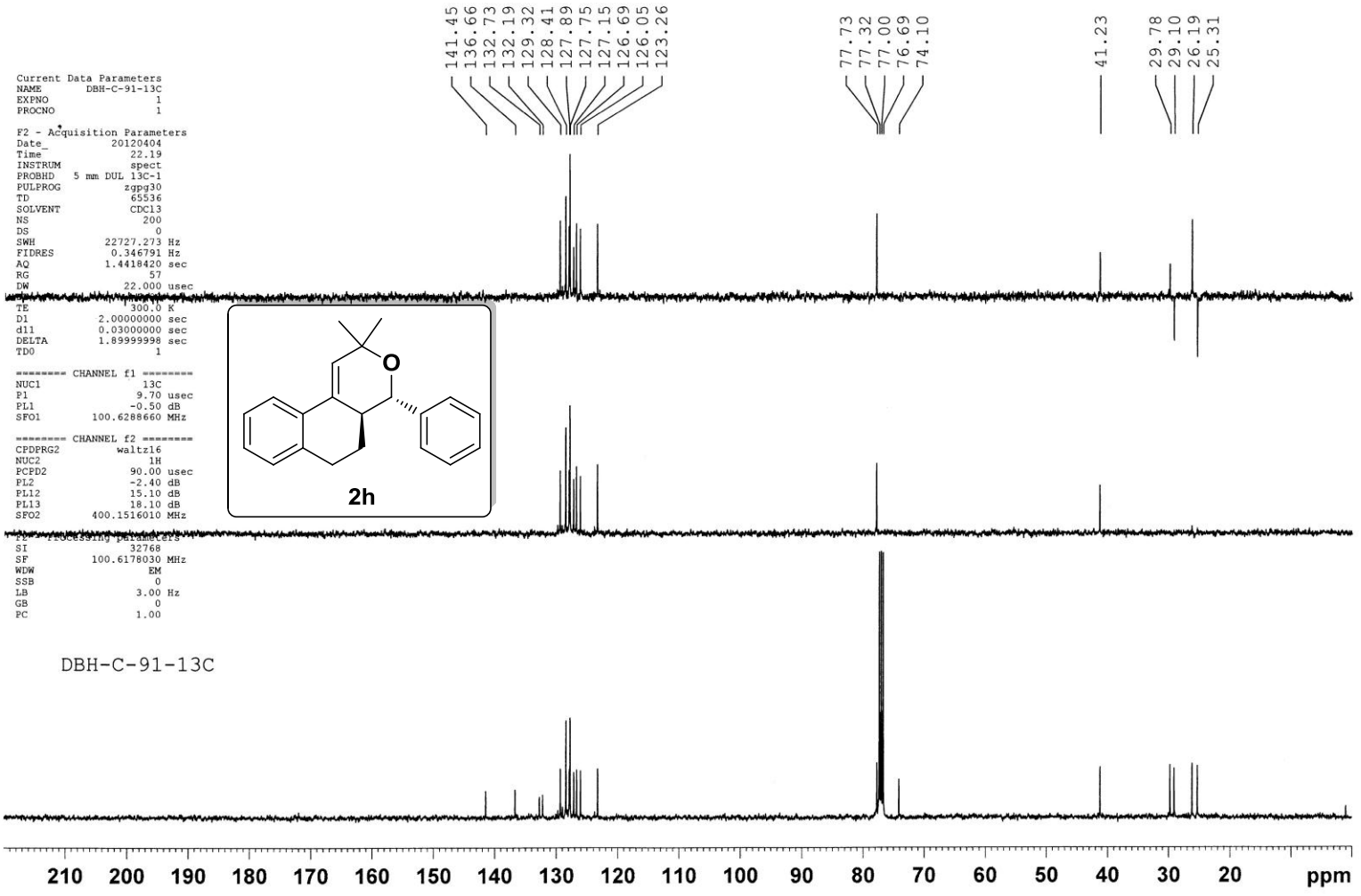
```
Current Data Parameters
NAME            DBH-C-109
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_          20120430
Time               15.00
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG              zg
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            8389.262 Hz
FIDRES         0.256020 Hz
AQ             1.9530228 sec
RG                  128
DW                59.600 usec
DE                 6.50 usec
TE                297.8 K
D1            2.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.80 usec
PL1                0.00 dB
SFO1         598.9029945 MHz

F2 - Processing parameters
SI                32768
SF           598.9000243 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                20.00 cm
F1P               10.000 ppm
F1                5989.00 Hz
F2P               -0.500 ppm
F2              -299.45 Hz
PPMCM          0.52500 ppm/cm
HZCM          314.42252 Hz/cm
```

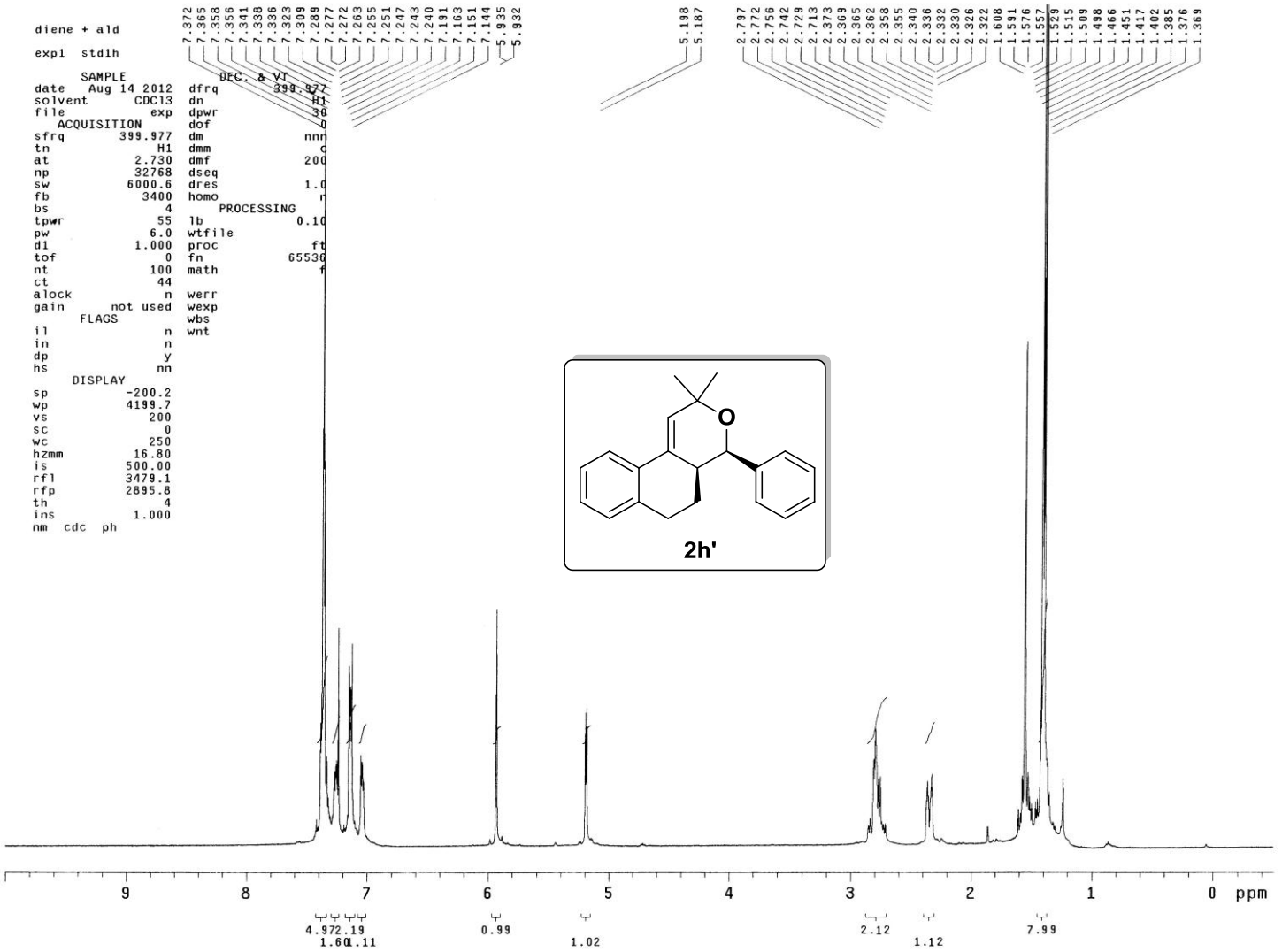


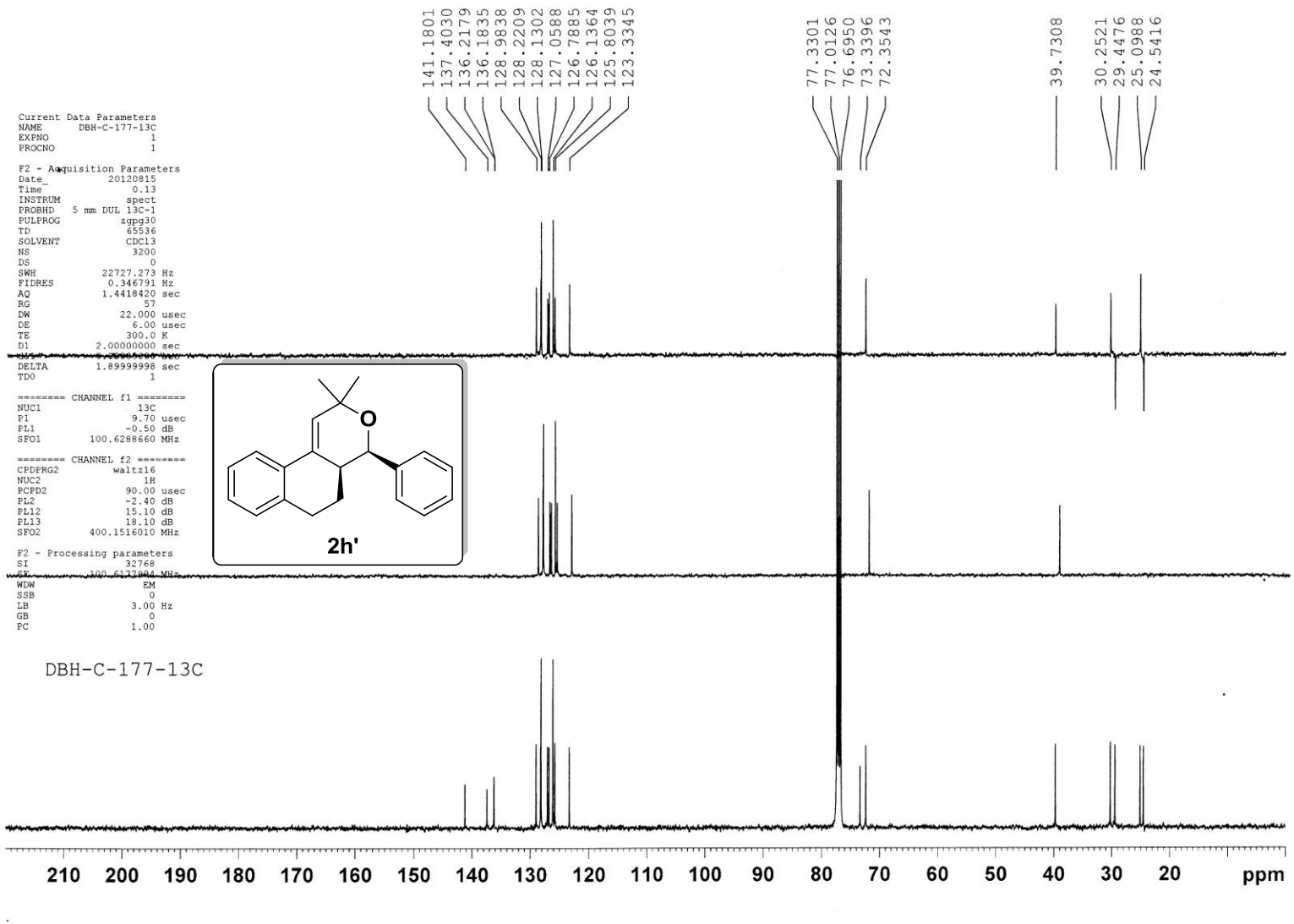
```
Current Data Parameters
NAME          DBH-C-109
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20120430
Time              15.33
INSTRUM           spect
PROBHD   5 mm QNP 1H/1
PULPROG           zgpg
TD                32768
SOLVENT           CDCl3
NS                  500
DS                    0
SWH           45045.047 Hz
FIDRES         1.374666 Hz
AQ             0.3637748 sec
RG                 2048
DW                11.100 usec
DE                 6.50 usec
TE                298.9 K
D1            3.50000000 sec
d11           0.03000000 sec
DELTA         3.40000010 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  4.80 usec
PL1                 0.00 dB
SFO1         150.6100903 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              92.00 usec
PL2               120.00 dB
PL12               9.00 dB
PL13              14.00 dB
SFO2         598.9029945 MHz

F2 - Processing parameters
SI                  65536
SF           150.5935307 MHz
WDW                    EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 20.00 cm
CY                  4.50 cm
F1P               200.000 ppm
F1              30118.71 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM           10.00000 ppm/cm
HZCM          1505.93530 Hz/cm
```

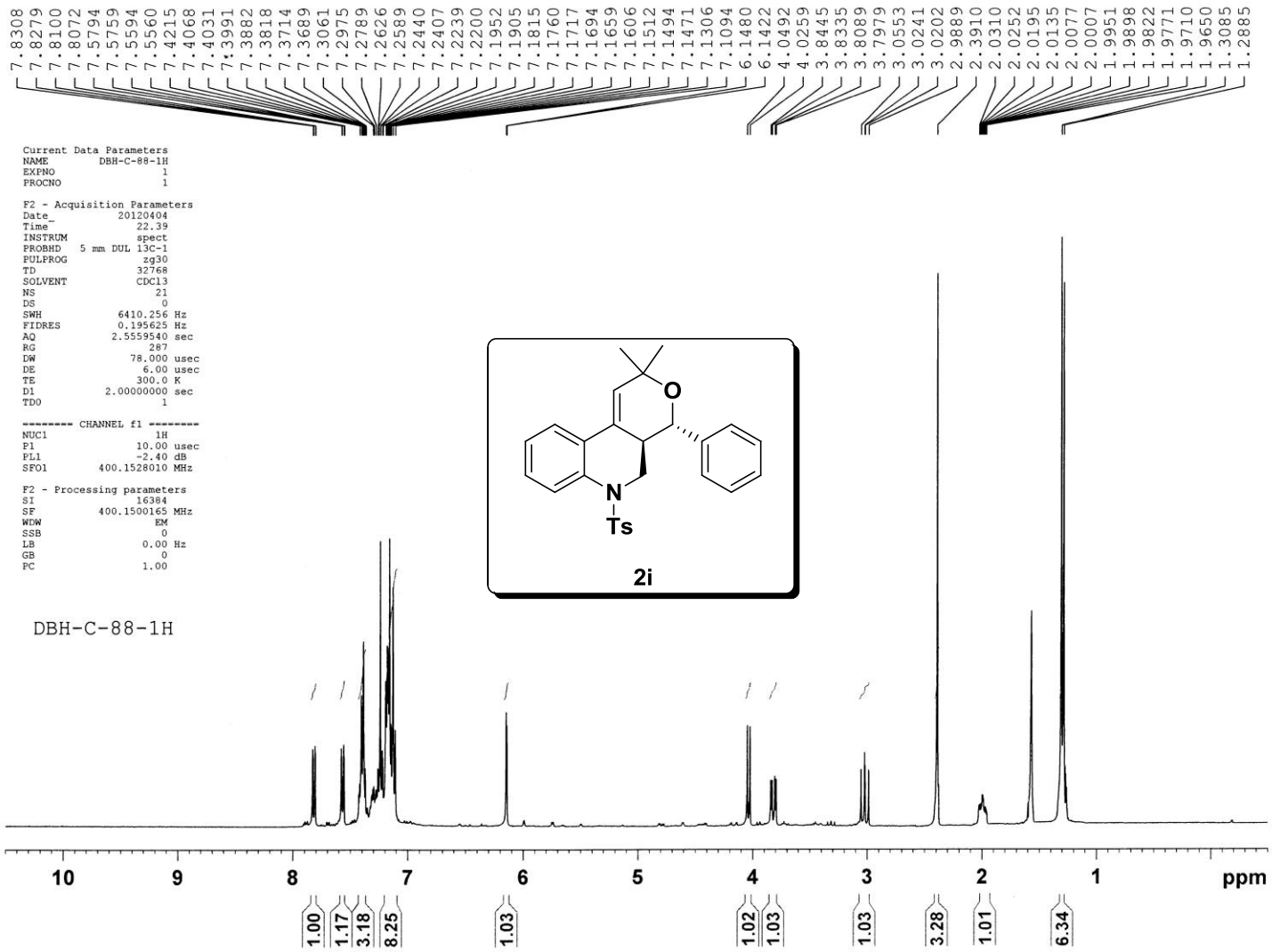


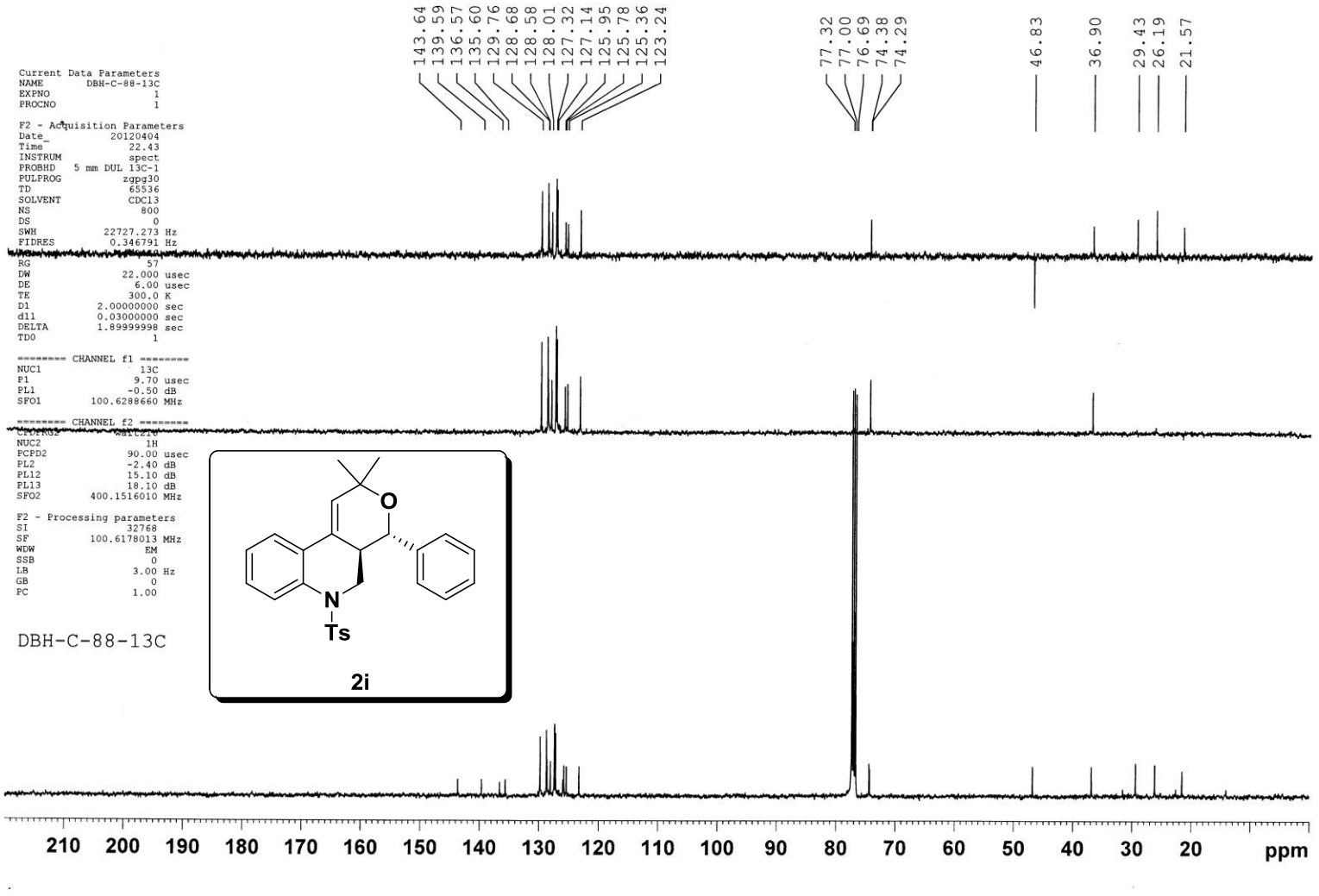


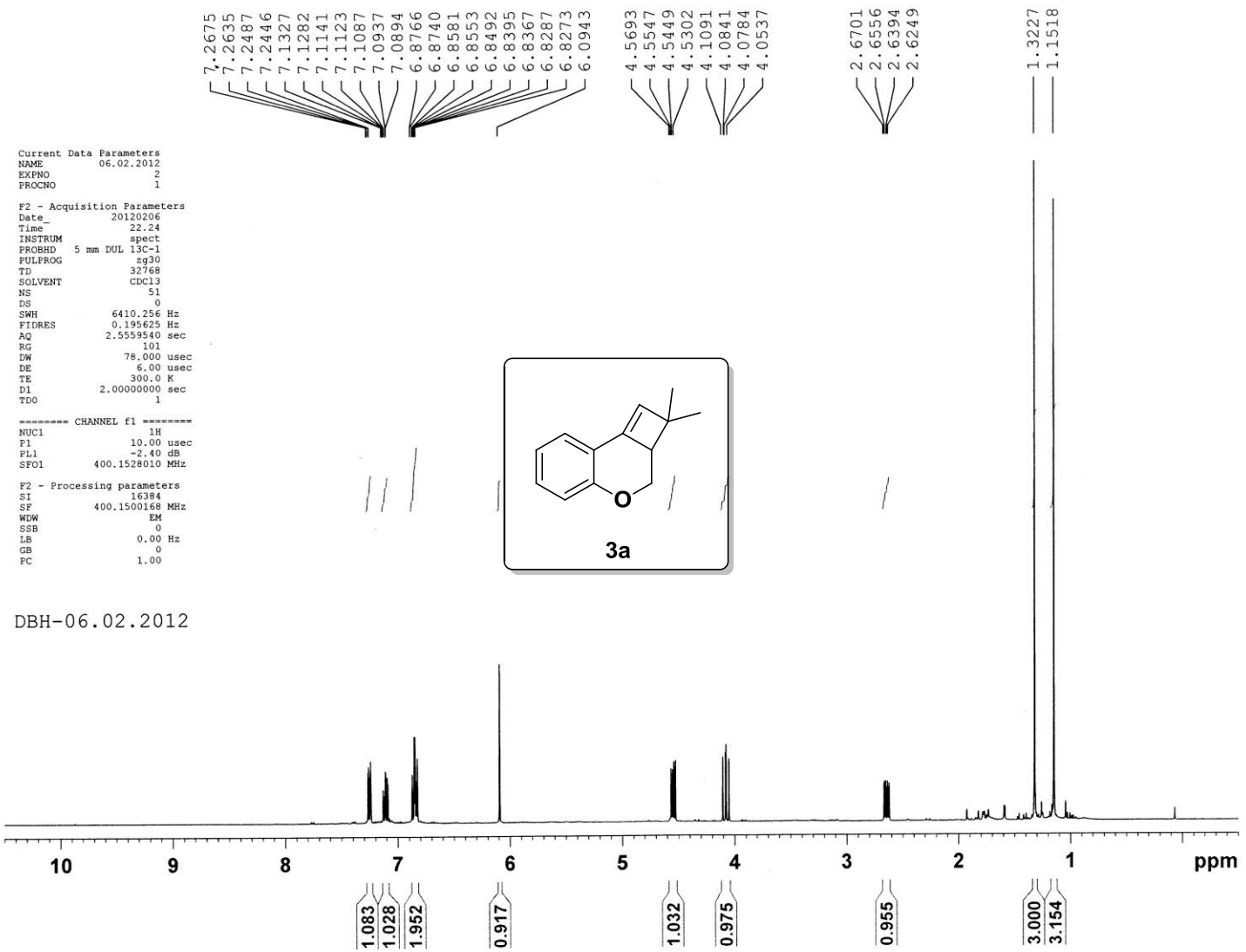


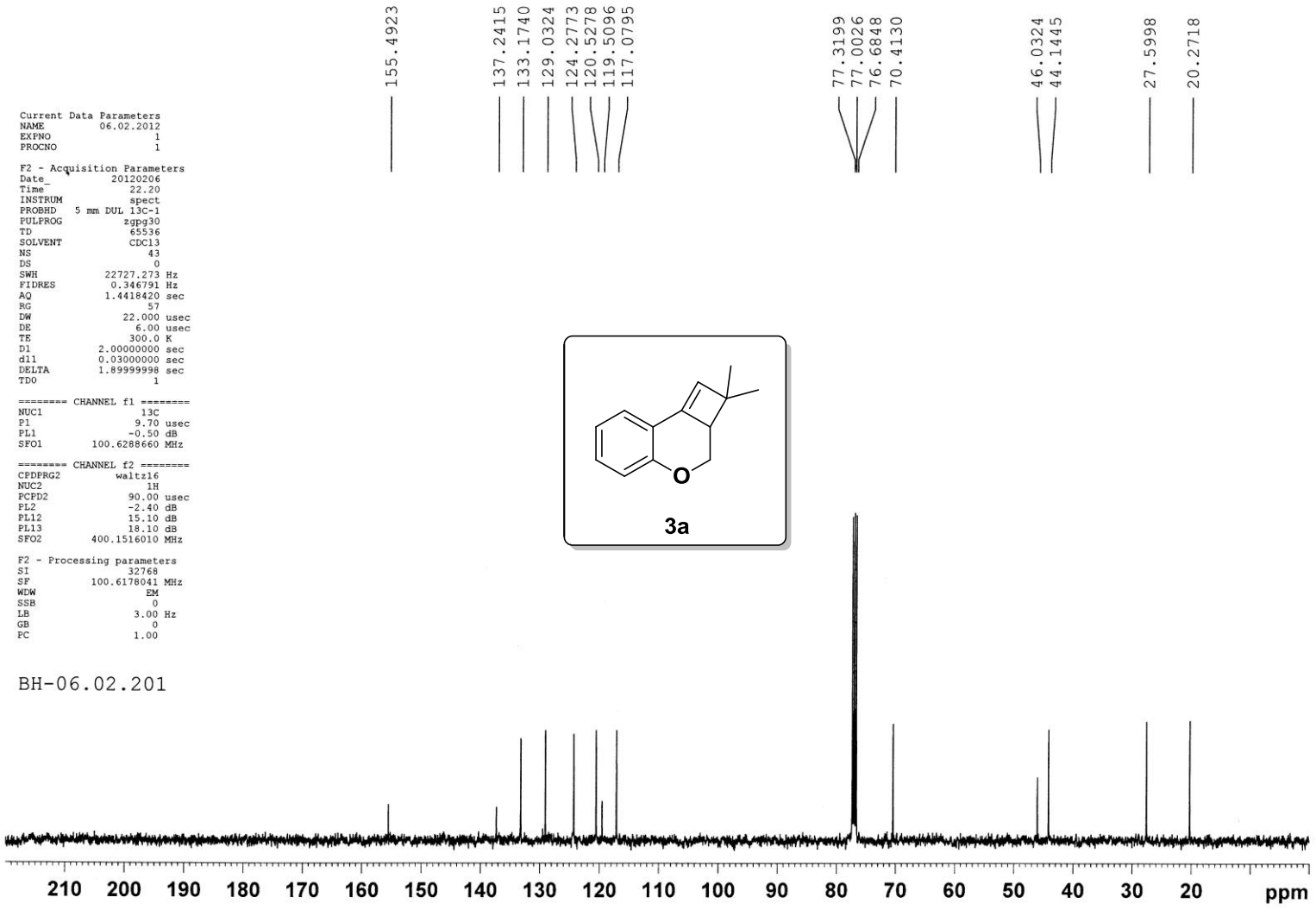


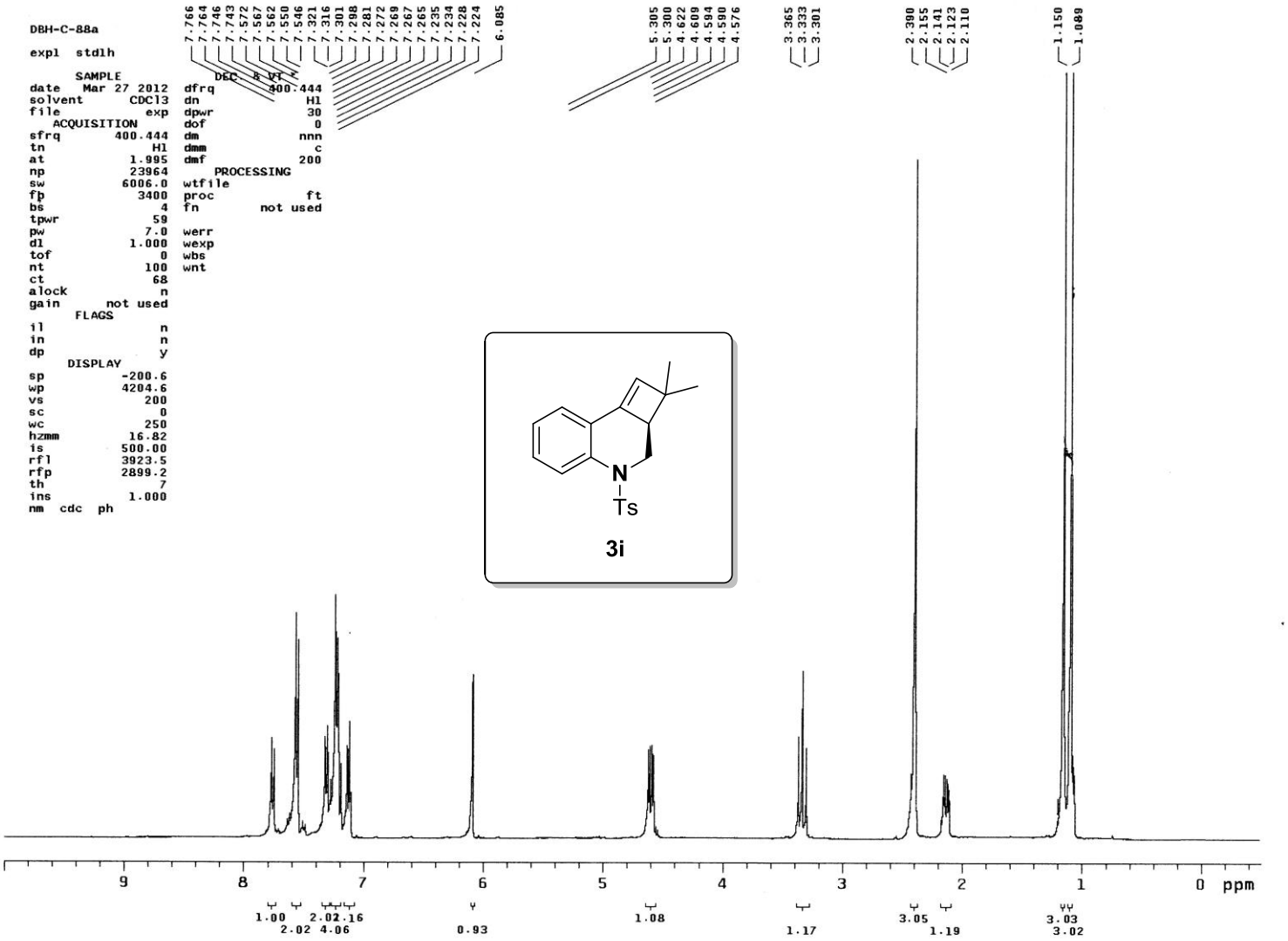


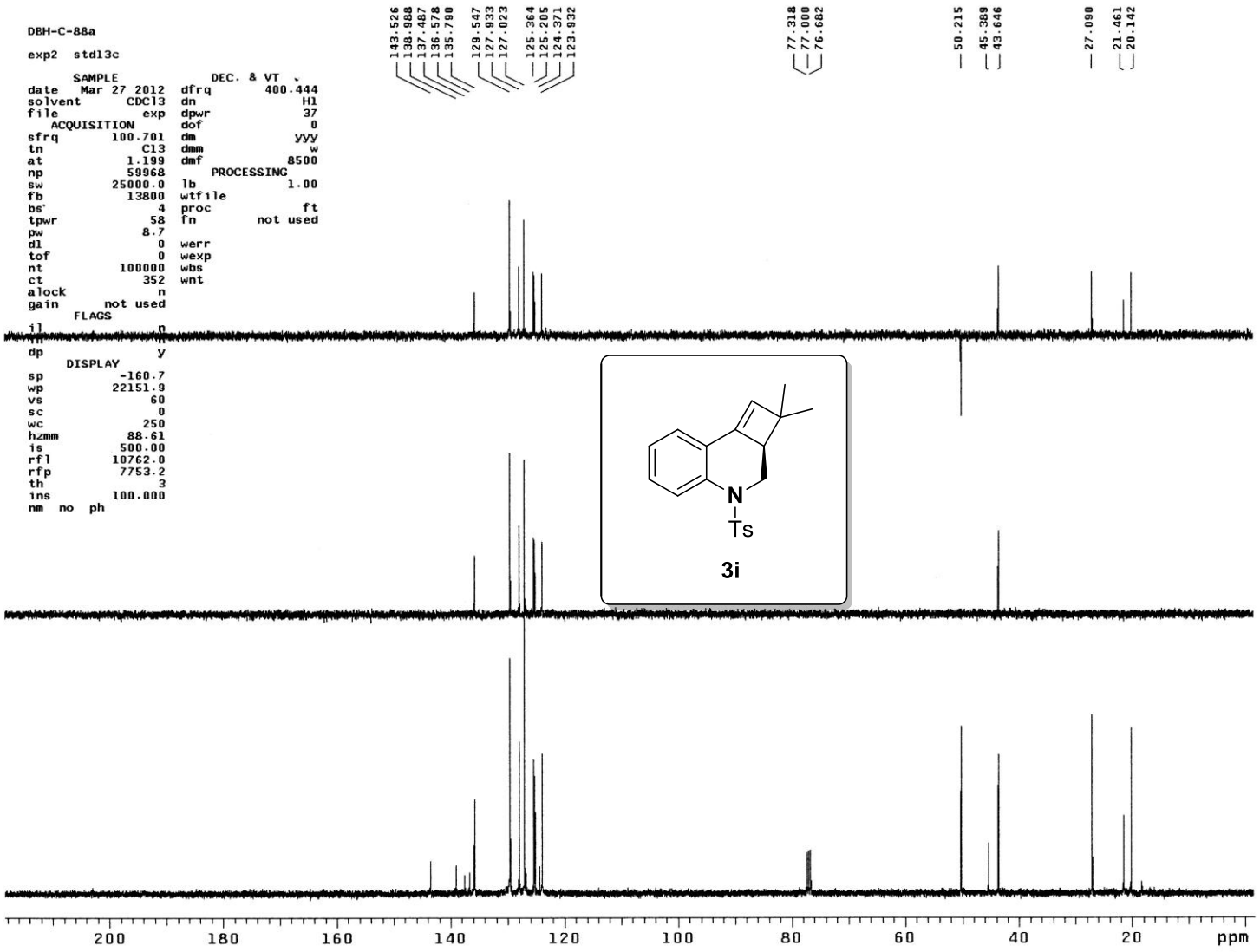


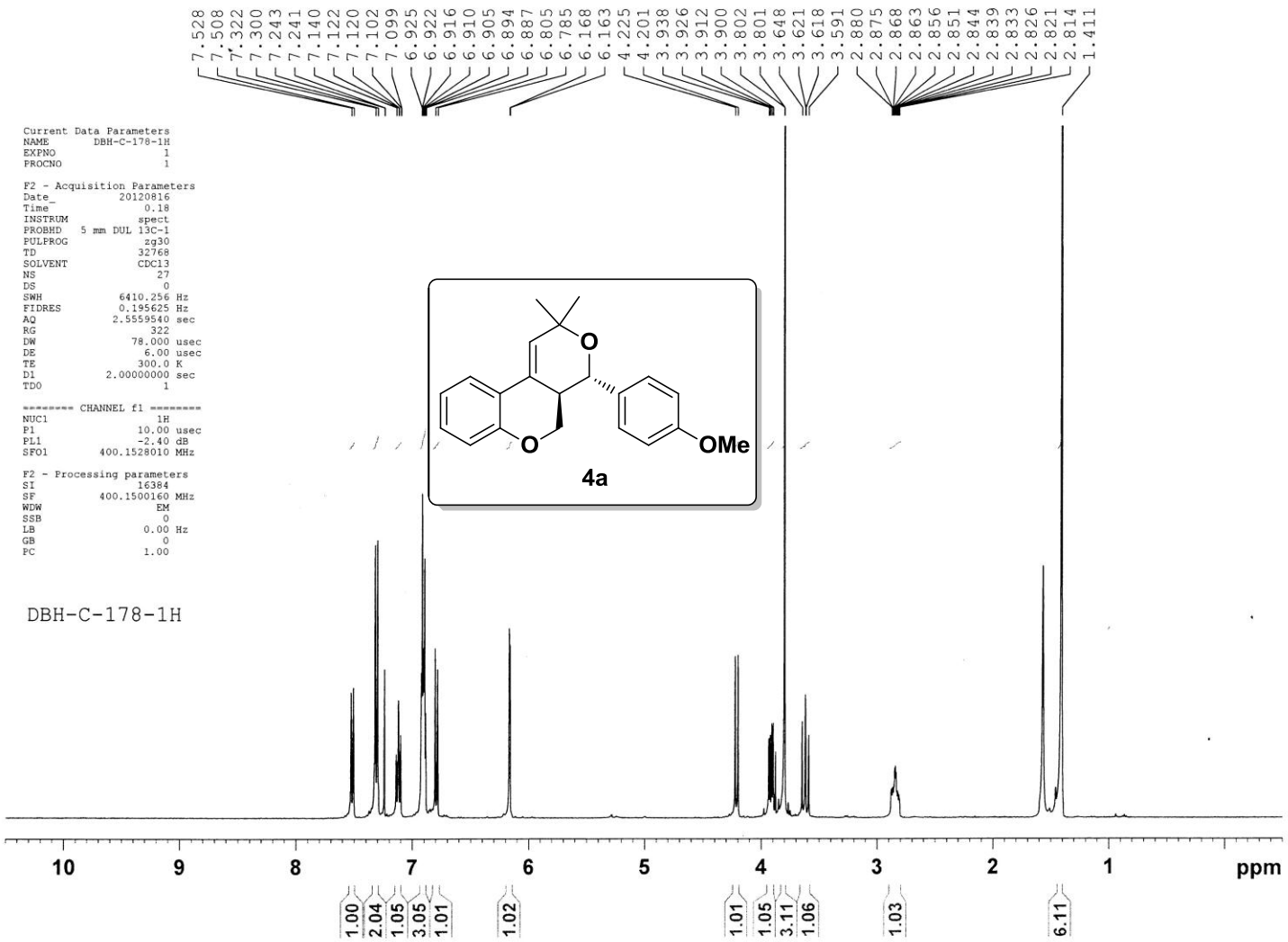


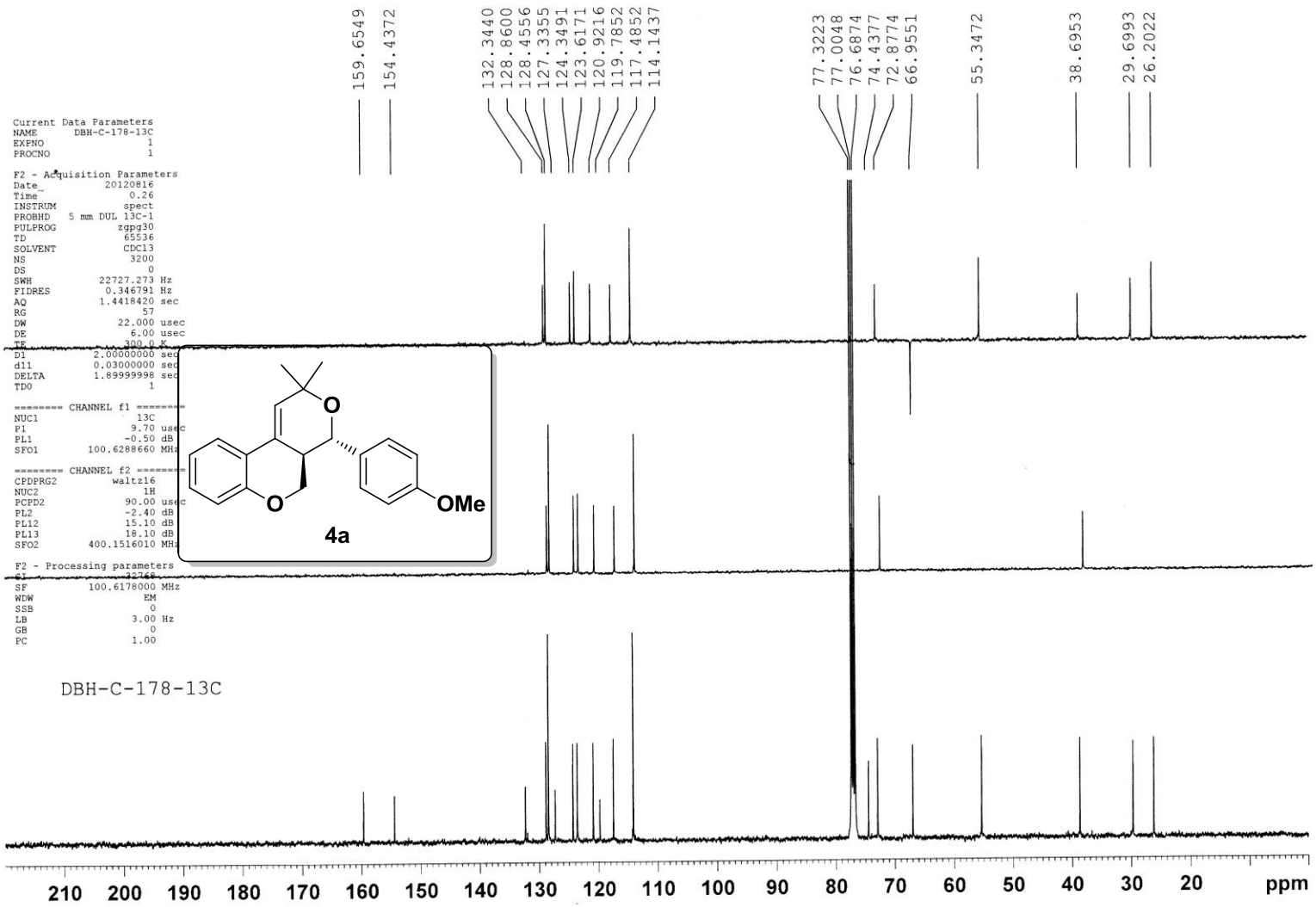




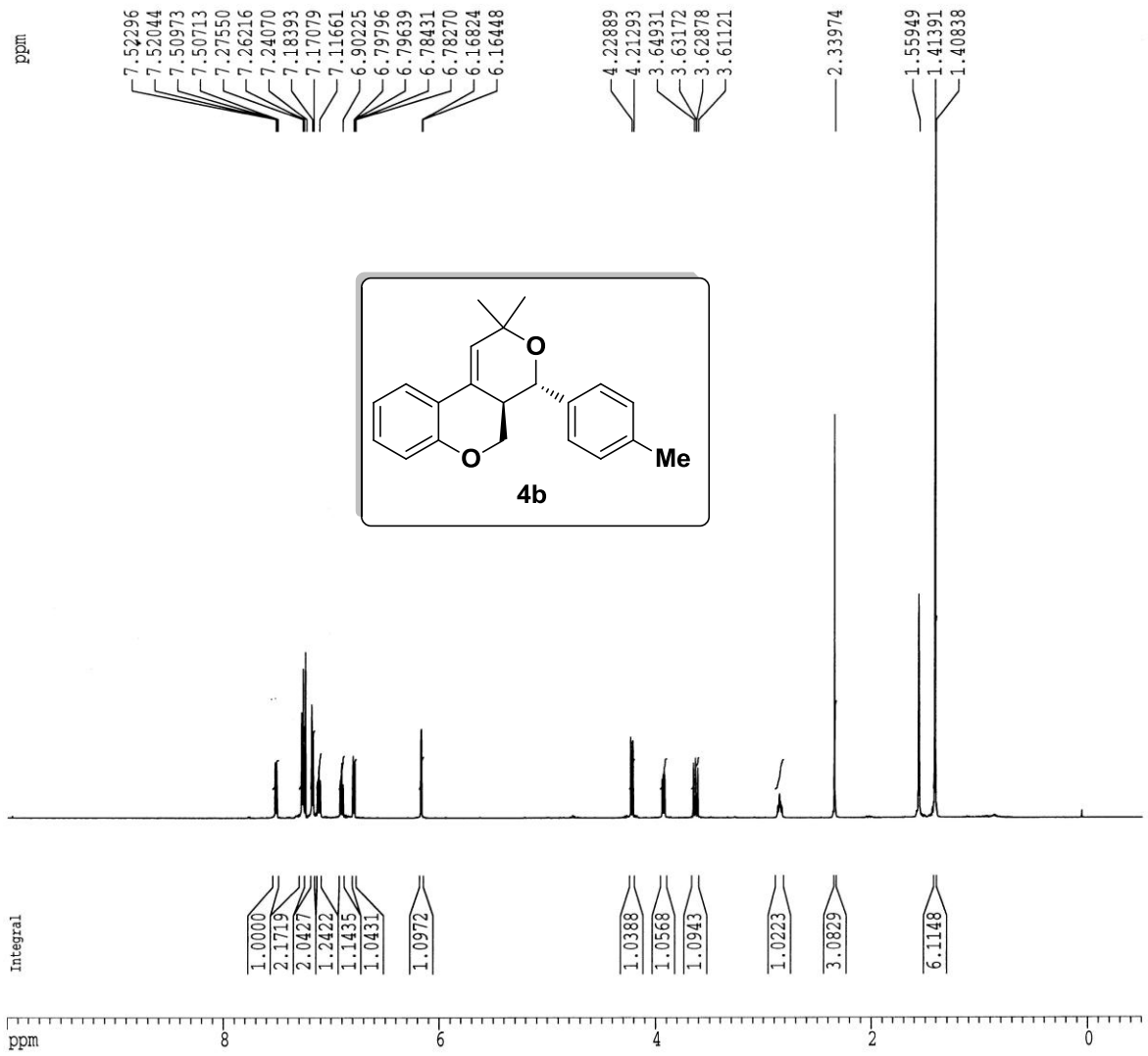




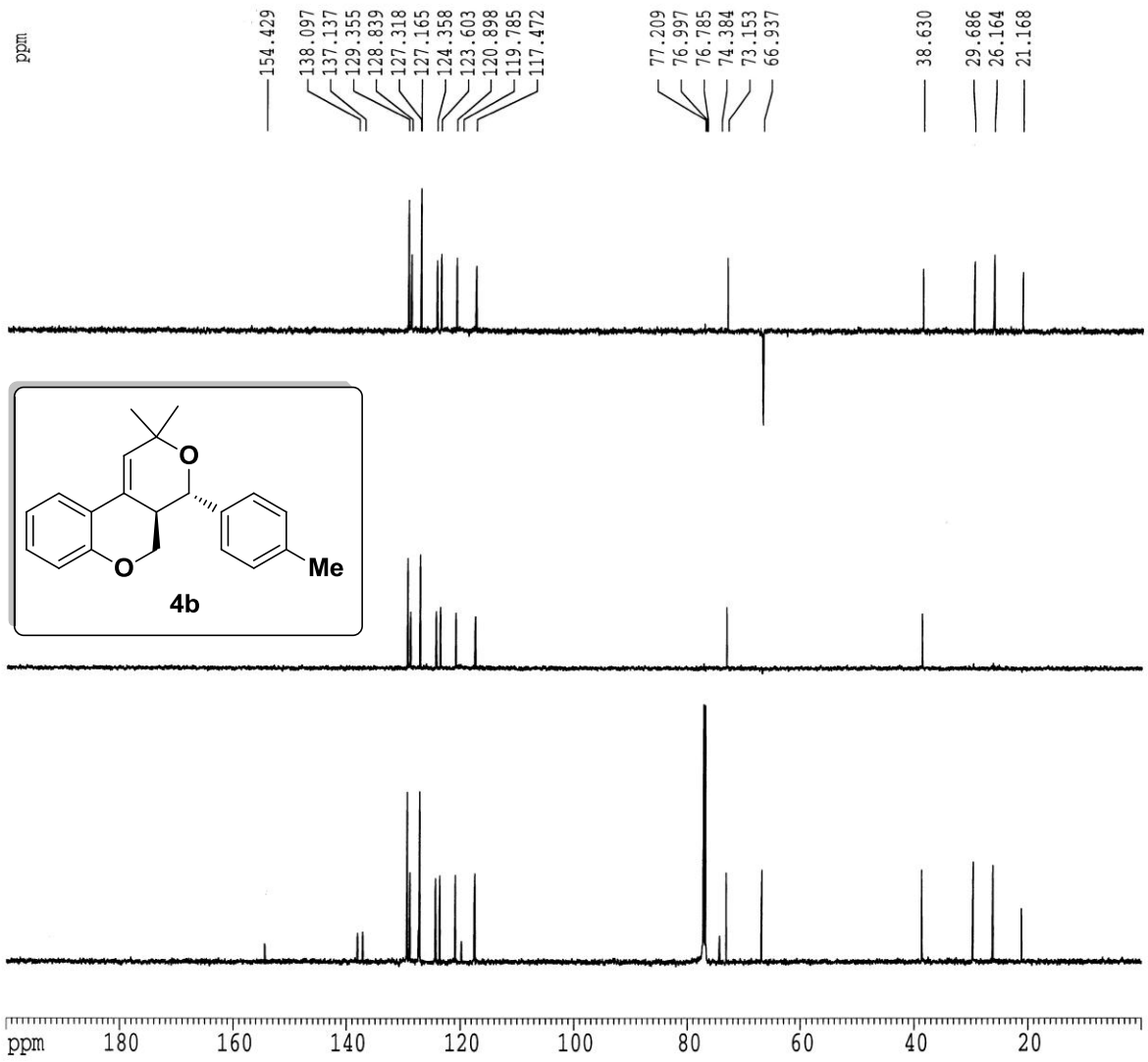




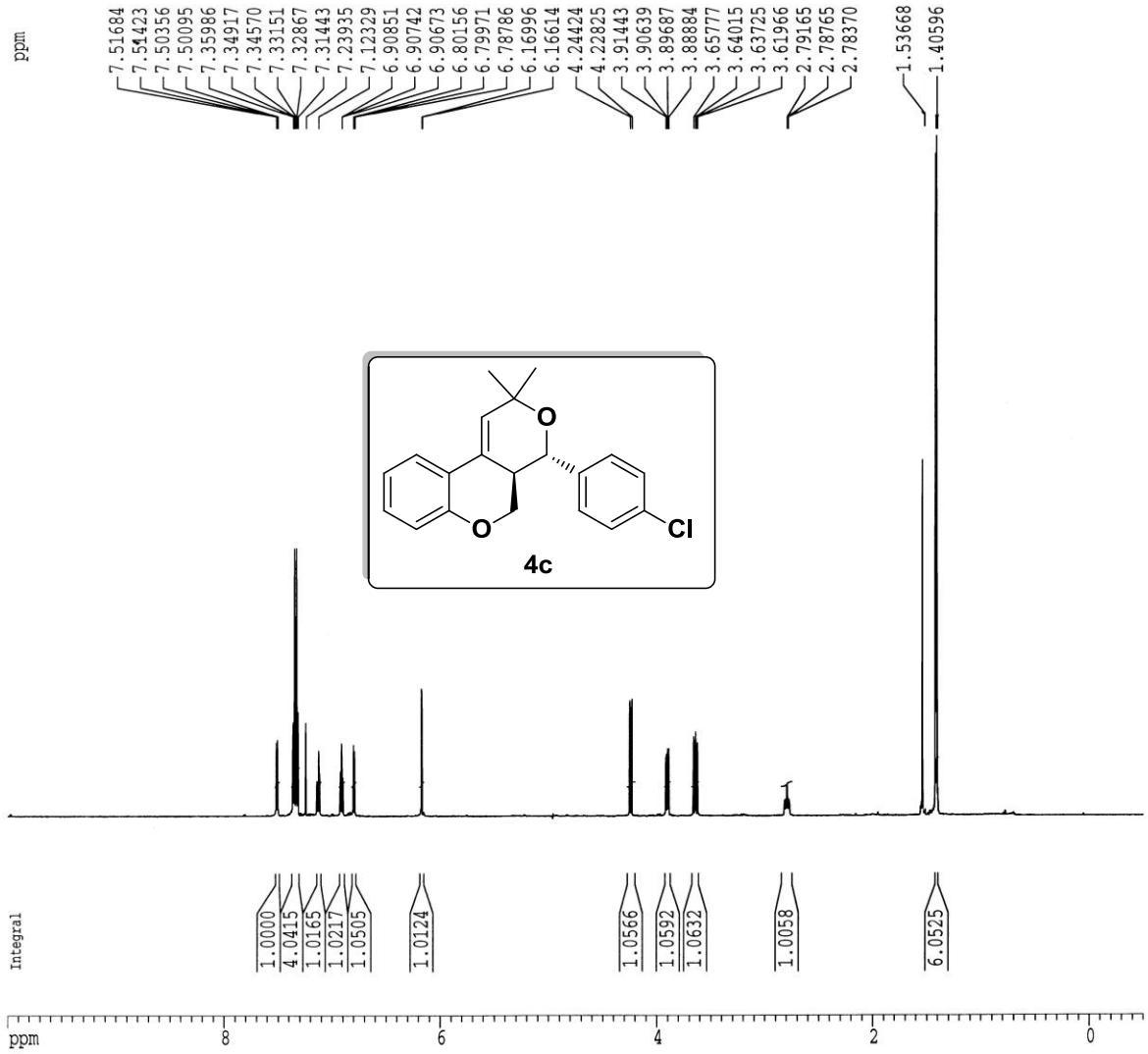
```
Current Data Parameters
NAME                  DBH-C-66
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_              20120215
Time                   14.08
INSTRUM                spect
PROBHD   5 mm QNP  1H/1
PULPROG                   zg
TD                     16384
SOLVENT                CDCl3
NS                        16
DS                         0
SWH                8389.262 Hz
FIDRES             0.512040 Hz
AQ                 0.9765364 sec
RG                       256
DW                  59.600 usec
DE                   6.50 usec
TE                   295.0 K
D1                3.50000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                    8.20 usec
PL1                   3.00 dB
SFO1            598.9029945 MHz

F2 - Processing parameters
SI                     16384
SF              598.9000241 MHz
WDW                       no
SSB                        0
LB                      0.00 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                    20.00 cm
CY                    20.00 cm
F1P                  10.000 ppm
F1                 5989.00 Hz
F2P                  -0.500 ppm
F2                 -299.45 Hz
PPMCM               0.52500 ppm/cm
HZCM              314.42252 Hz/cm
```



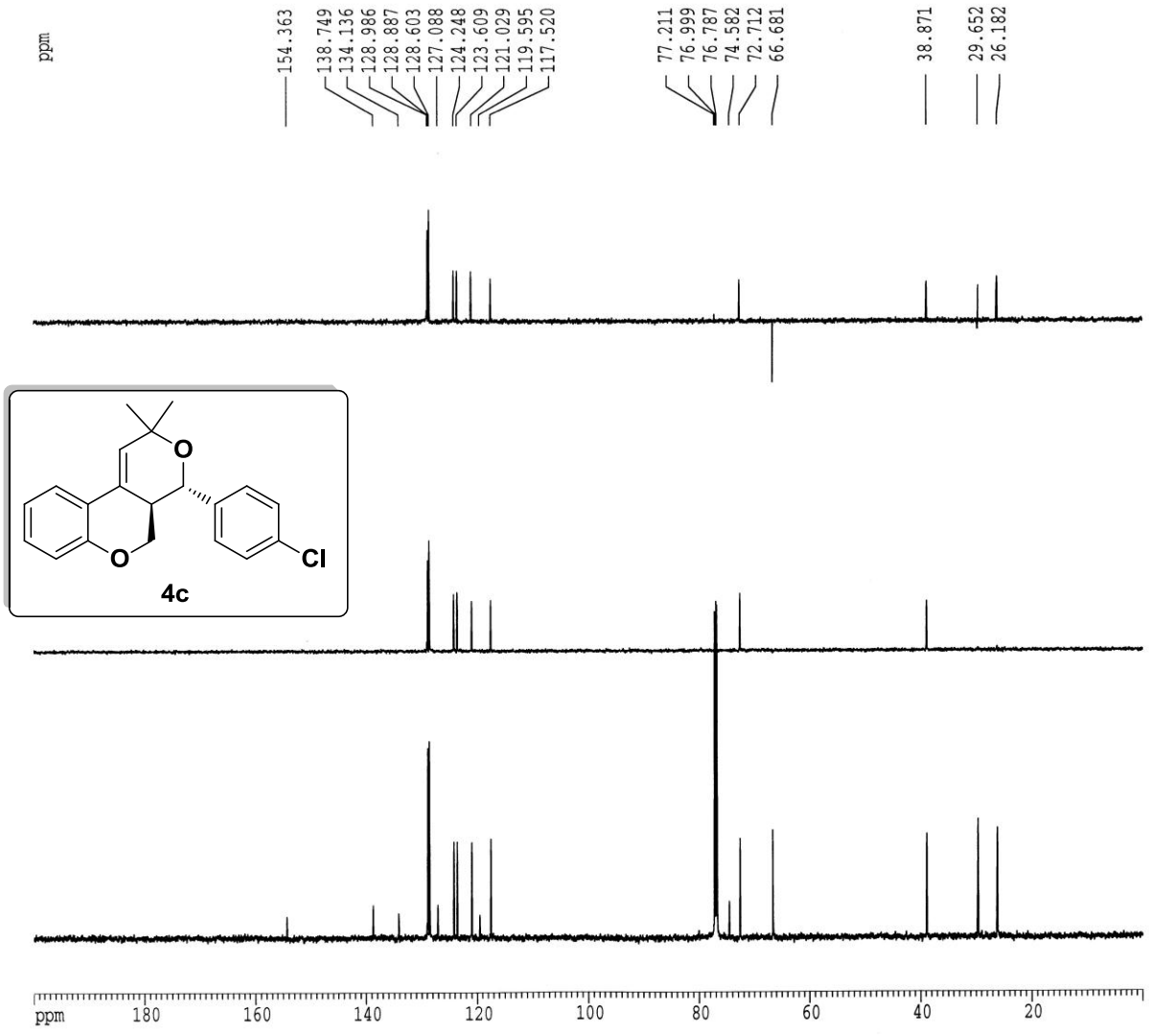
```
Current Data Parameters
NAME            DBH-C-66
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120215
Time               14.11
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG            zgpg
TD                 32768
SOLVENT            CDCl3
NS                   514
DS                     0
SWH            45045.047 Hz
FIDRES          1.374666 Hz
AQ              0.3637748 sec
RG                  4096
DW                11.100 usec
DE                 6.50 usec
TE                295.4 K
D1            3.50000000 sec
d11           0.03000000 sec
DELTA         3.40000010 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 4.80 usec
PL1                0.00 dB
SFO1         150.6100903 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             92.00 usec
PL2              120.00 dB
PL12              11.30 dB
PL13              14.00 dB
SFO2         598.9029990 MHz

F2 - Processing parameters
SI                 65536
SF           150.5935300 MHz
WDW                   EM
SSB                    0
LB                 3.00 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                20.00 cm
CY                 4.50 cm
F1P              200.000 ppm
F1             30118.71 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM          10.00000 ppm/cm
HZCM         1505.93530 Hz/cm
```



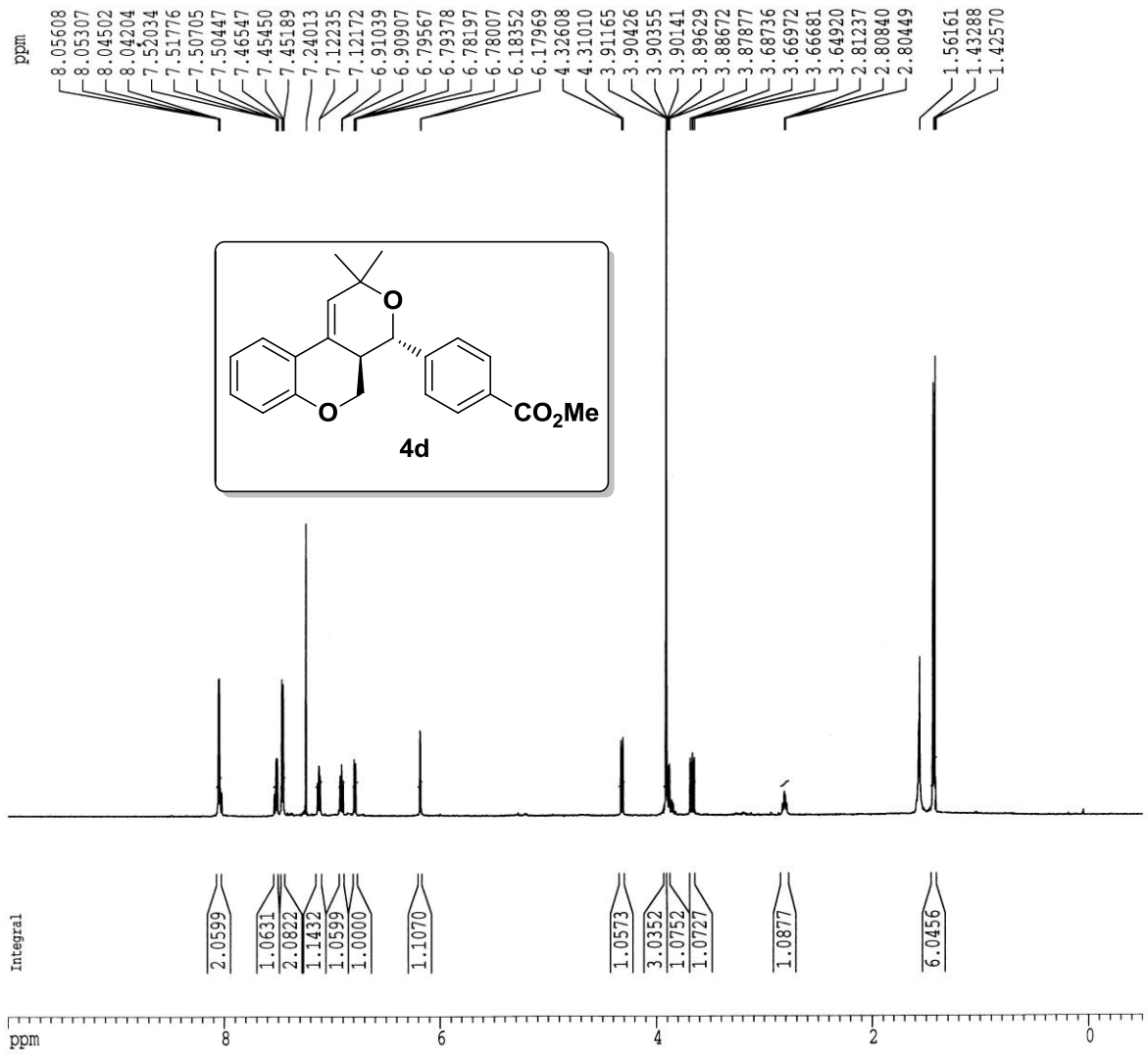
```
Current Data Parameters
NAME            DBH-C-113
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20120506
Time                17.35
INSTRUM             spect
PROBHD   5 mm QNP  1H/1  *
PULPROG                zg
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              8389.262 Hz
FIDRES           0.256020 Hz
AQ               1.9530228 sec
RG                    128
DW                59.600 usec
DE                 6.50 usec
TE                297.8 K
D1            2.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  9.80 usec
PL1                 0.00 dB
SFO1         598.9029945 MHz

F2 - Processing parameters
SI                  32768
SF           598.9000259 MHz
WDW                    no
SSB                     0
LB                  0.00 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  20.00 cm
CY                  12.00 cm
F1P                10.000 ppm
F1                 5989.00 Hz
F2P                -0.500 ppm
F2                 -299.45 Hz
PPMCM               0.52500 ppm/cm
HZCM              314.42252 Hz/cm
```

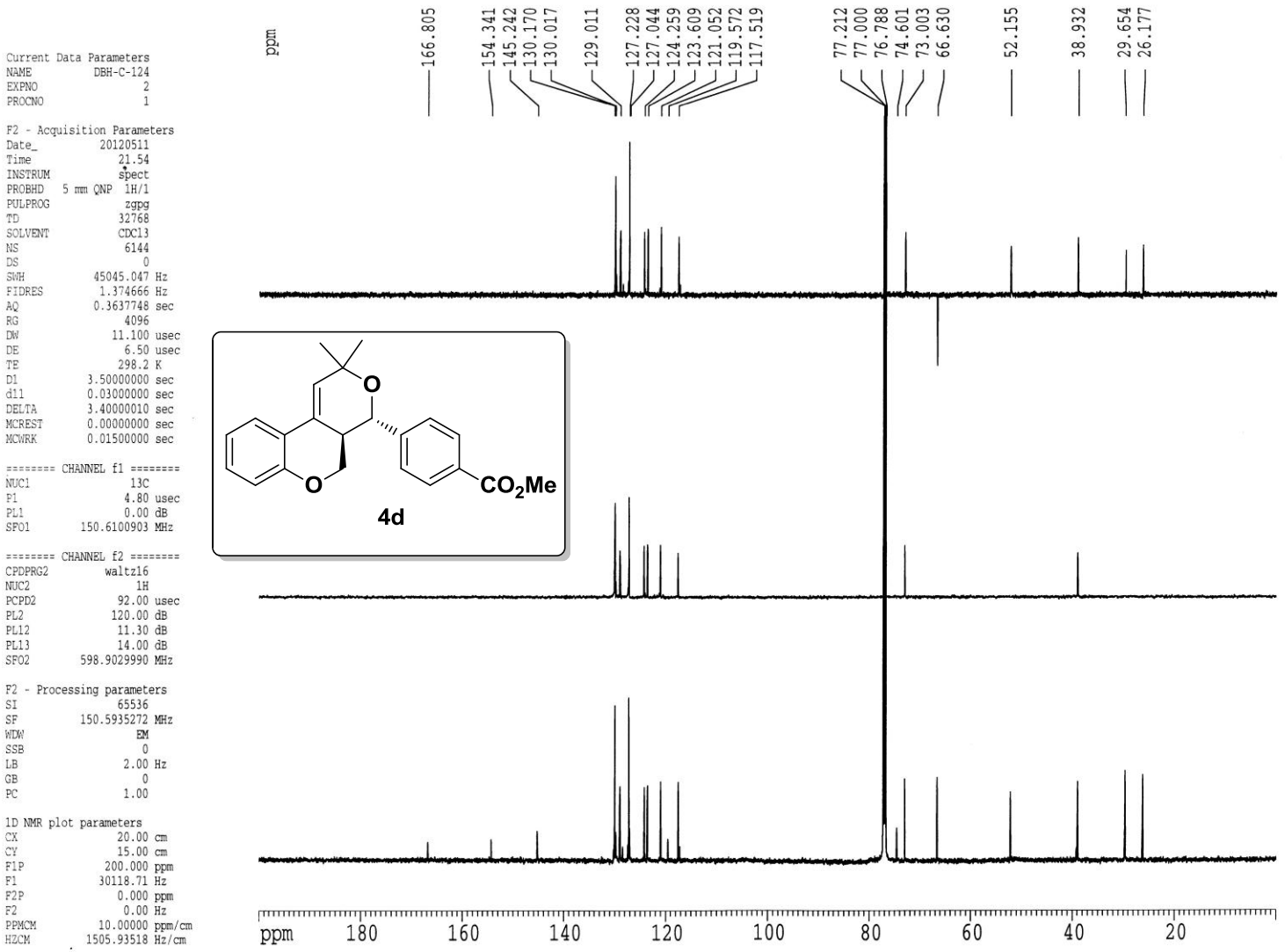


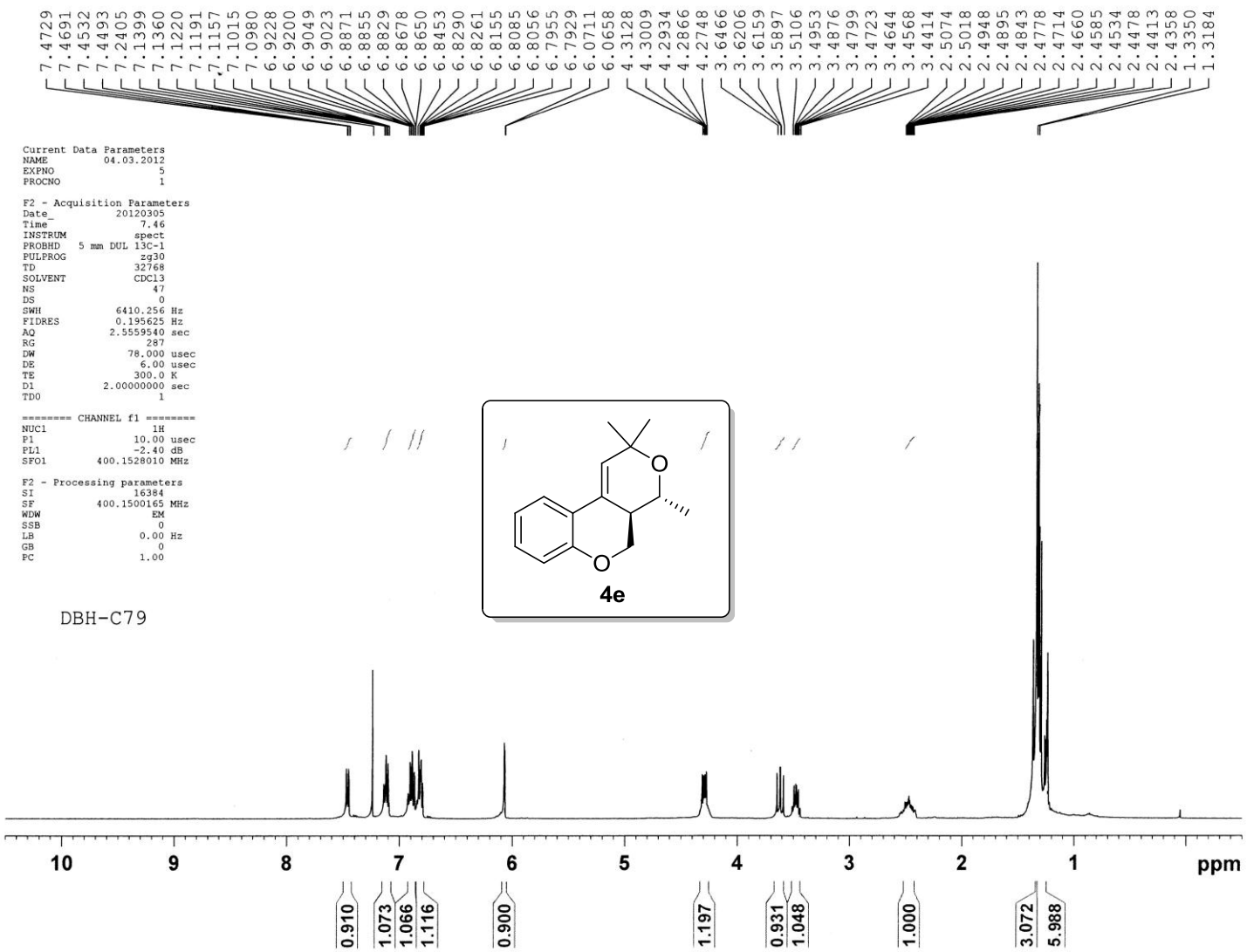
```
Current Data Parameters
NAME          DBH-C-113
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20120506
Time              18.08
INSTRUM           spect
PROBHD   5 mm QNP  1H/1
PULPROG           zgpg
TD               32768
SOLVENT           CDCl3
NS                  500
DS                    0
SWH           45045.047 Hz
FIDRES         1.374666 Hz
AQ             0.3637748 sec
RG                 4096
DW               11.100 usec
DE                6.50 usec
TE                298.4 K
D1           3.50000000 sec
d11          0.03000000 sec
DELTA        3.40000010 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1               13C
P1                4.50 usec
PL1               0.00 dB
SFO1        150.6100903 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2            92.00 usec
PL2              120.00 dB
PL12              11.30 dB
PL13              14.00 dB
SFO2        598.9029990 MHz

F2 - Processing parameters
SI                65536
SF          150.5935279 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                 6.00 cm
F1P              200.000 ppm
F1             30118.71 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM         10.00000 ppm/cm
HZCM        1505.93518 Hz/cm
```

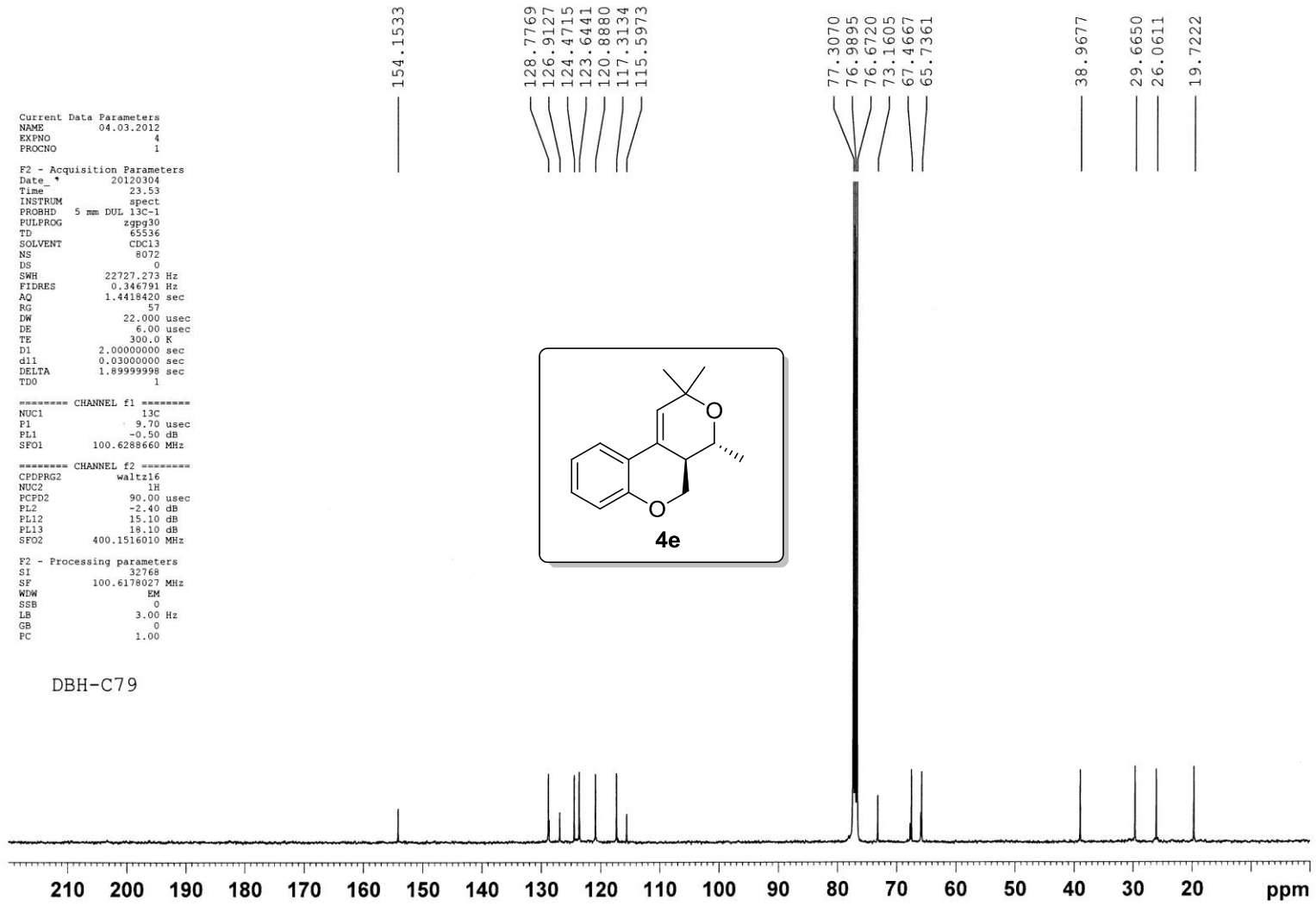


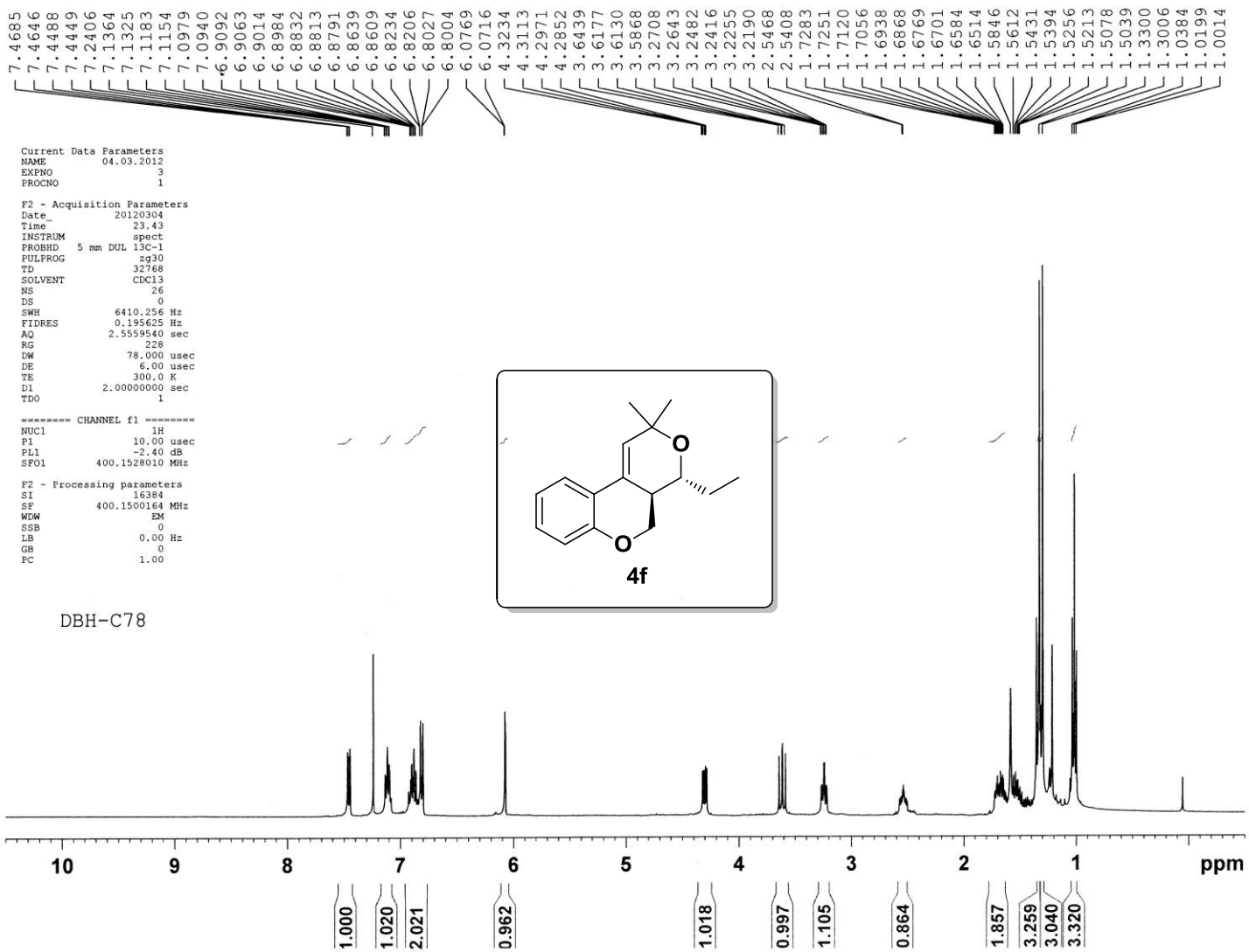
```
Current Data Parameters
NAME            DBH-C-124
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20120511
Time                21.50
INSTRUM             spect
PROBHD   5 mm QNP  1H/1
PULPROG                 zg
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       0
SWH              8389.262 Hz
FIDRES           0.256020 Hz
AQ               1.9530228 sec
RG                     128
DW                59.600 usec
DE                 6.50 usec
TE                 297.8 K
D1            2.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.80 usec
PL1                  0.00 dB
SFO1          598.9029945 MHz

F2 - Processing parameters
SI                   32768
SF            598.9000254 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                   20.00 cm
CY                   15.00 cm
F1P                 10.000 ppm
F1                 5989.00 Hz
F2P                 -0.500 ppm
F2                 -299.45 Hz
PPMCM               0.52500 ppm/cm
HZCM              314.42252 Hz/cm
```

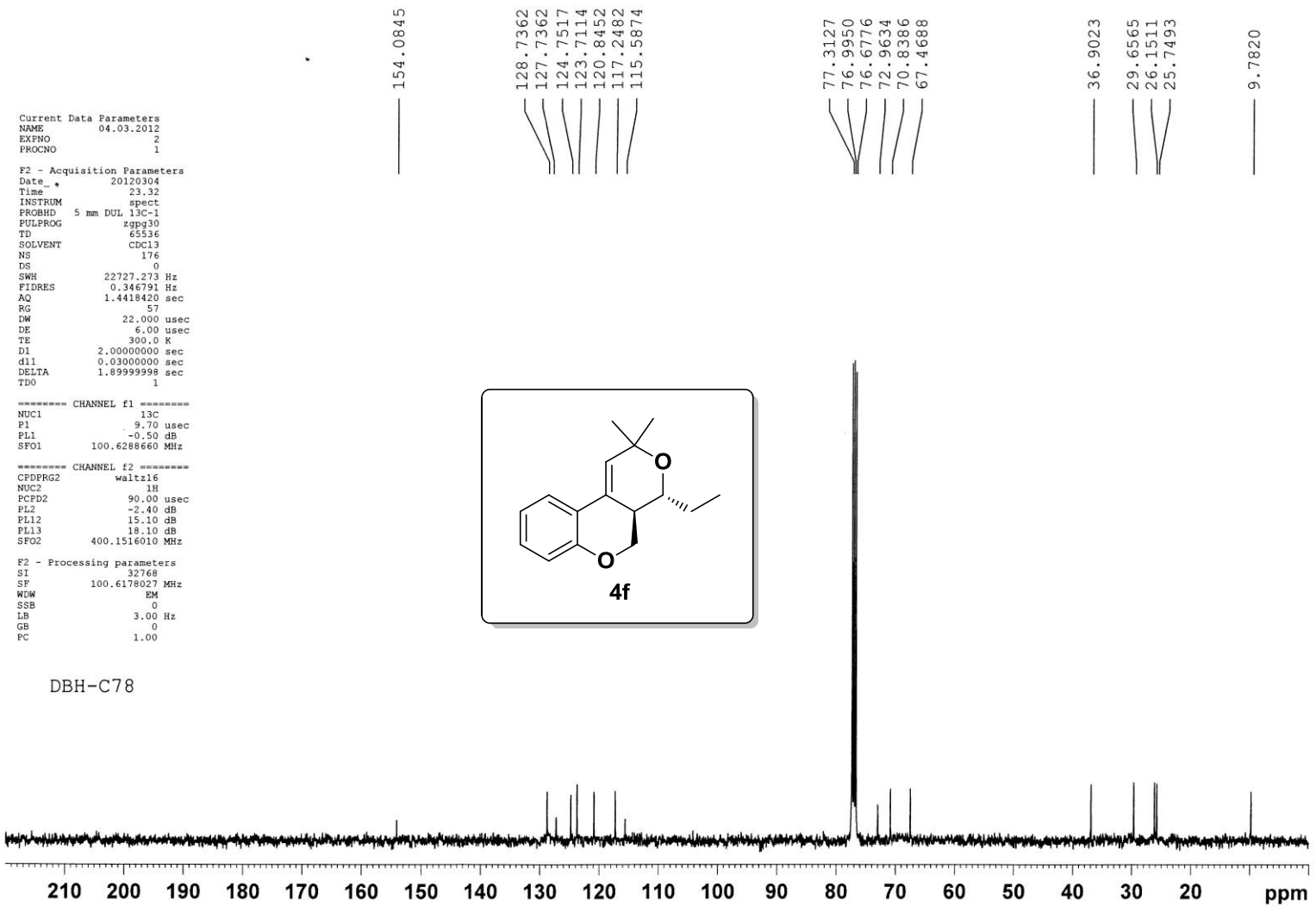


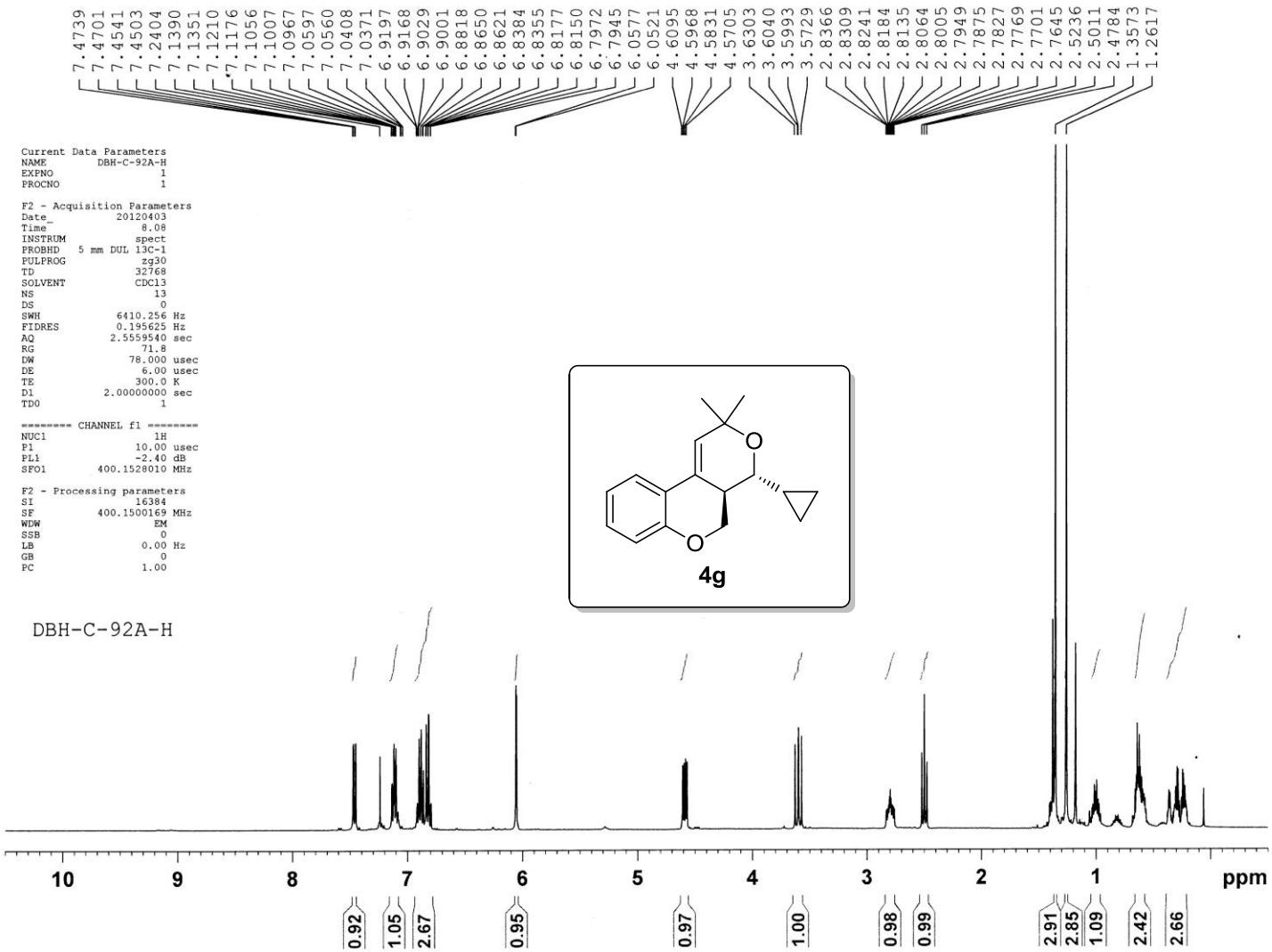


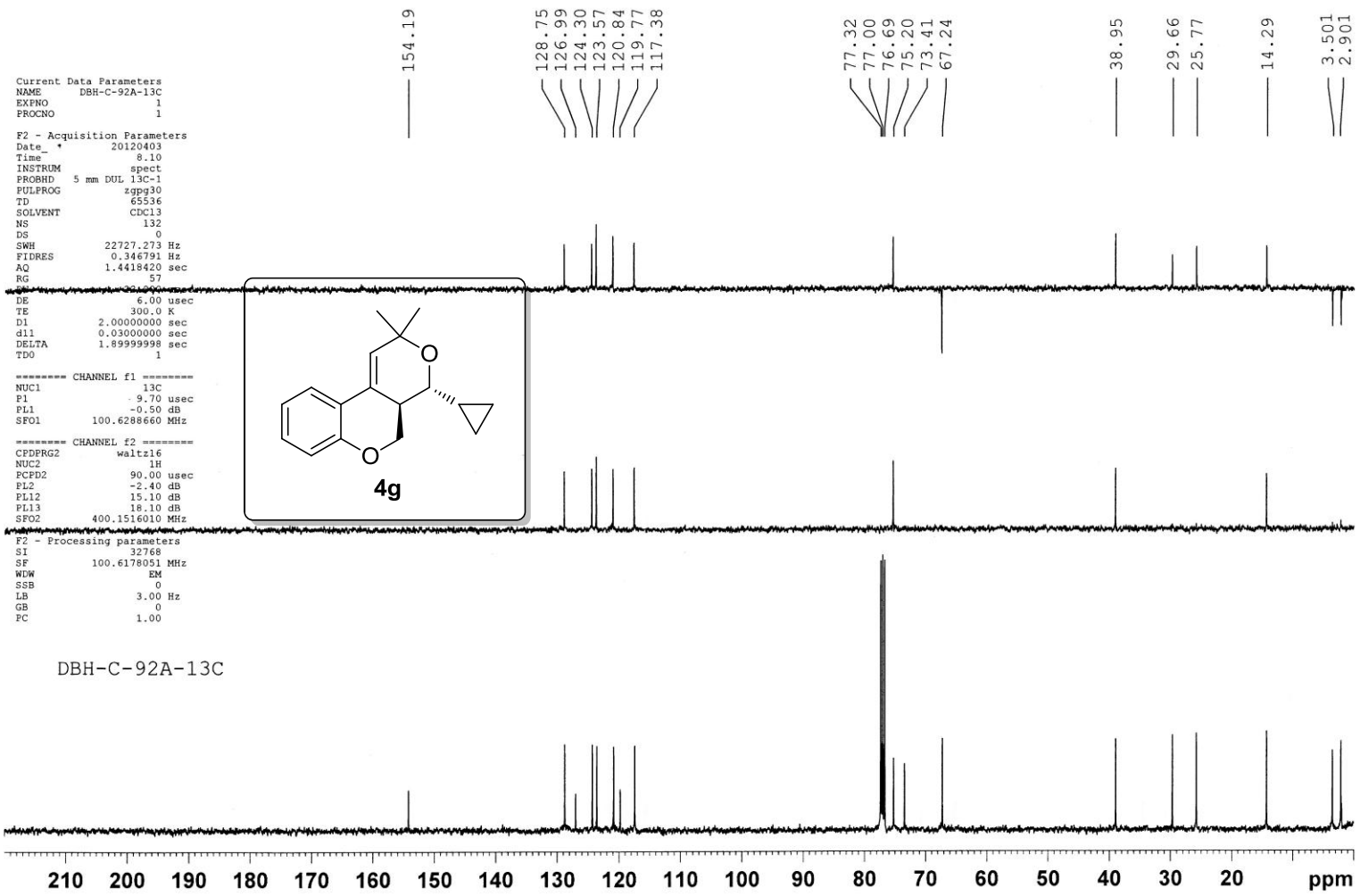




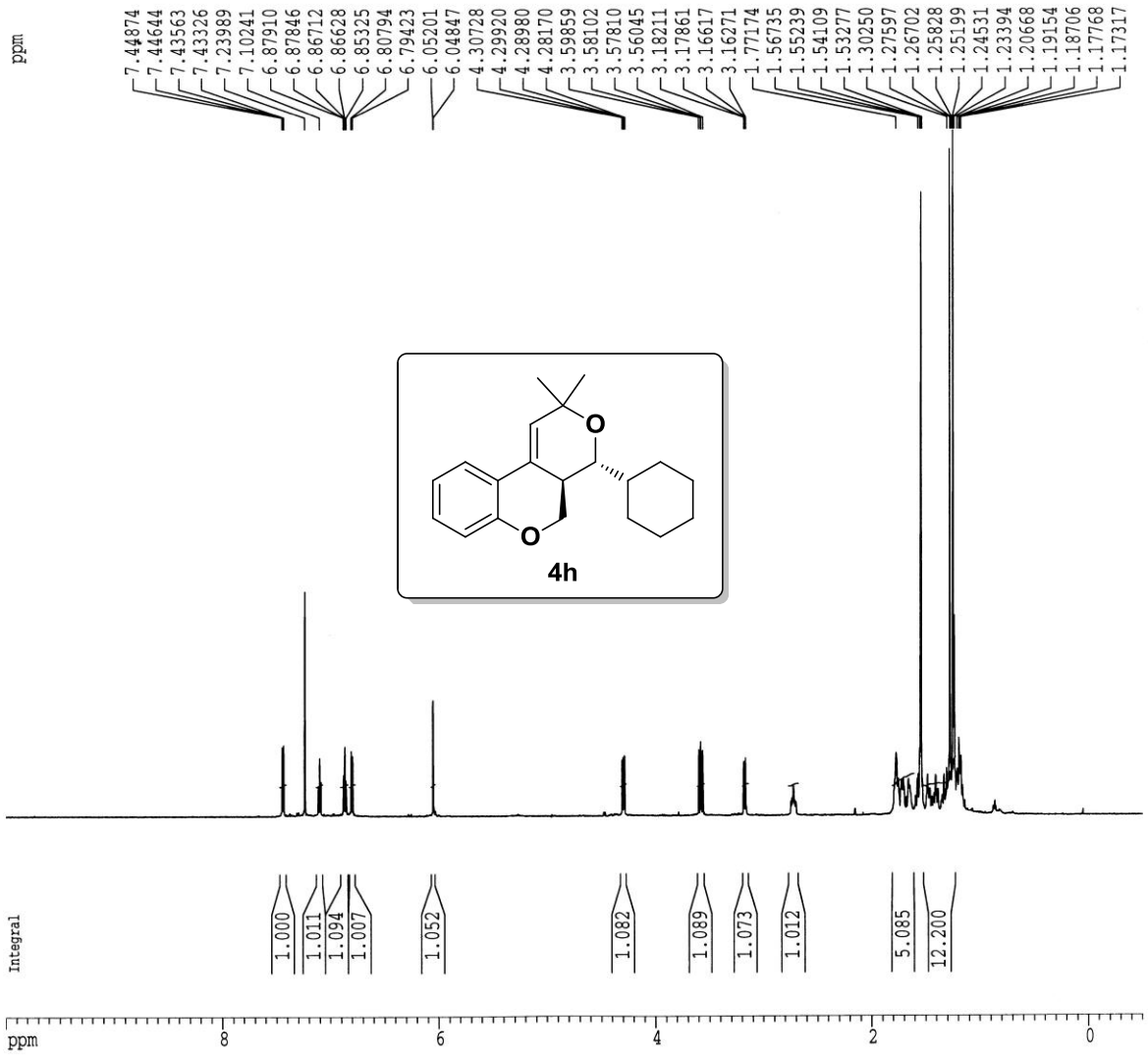




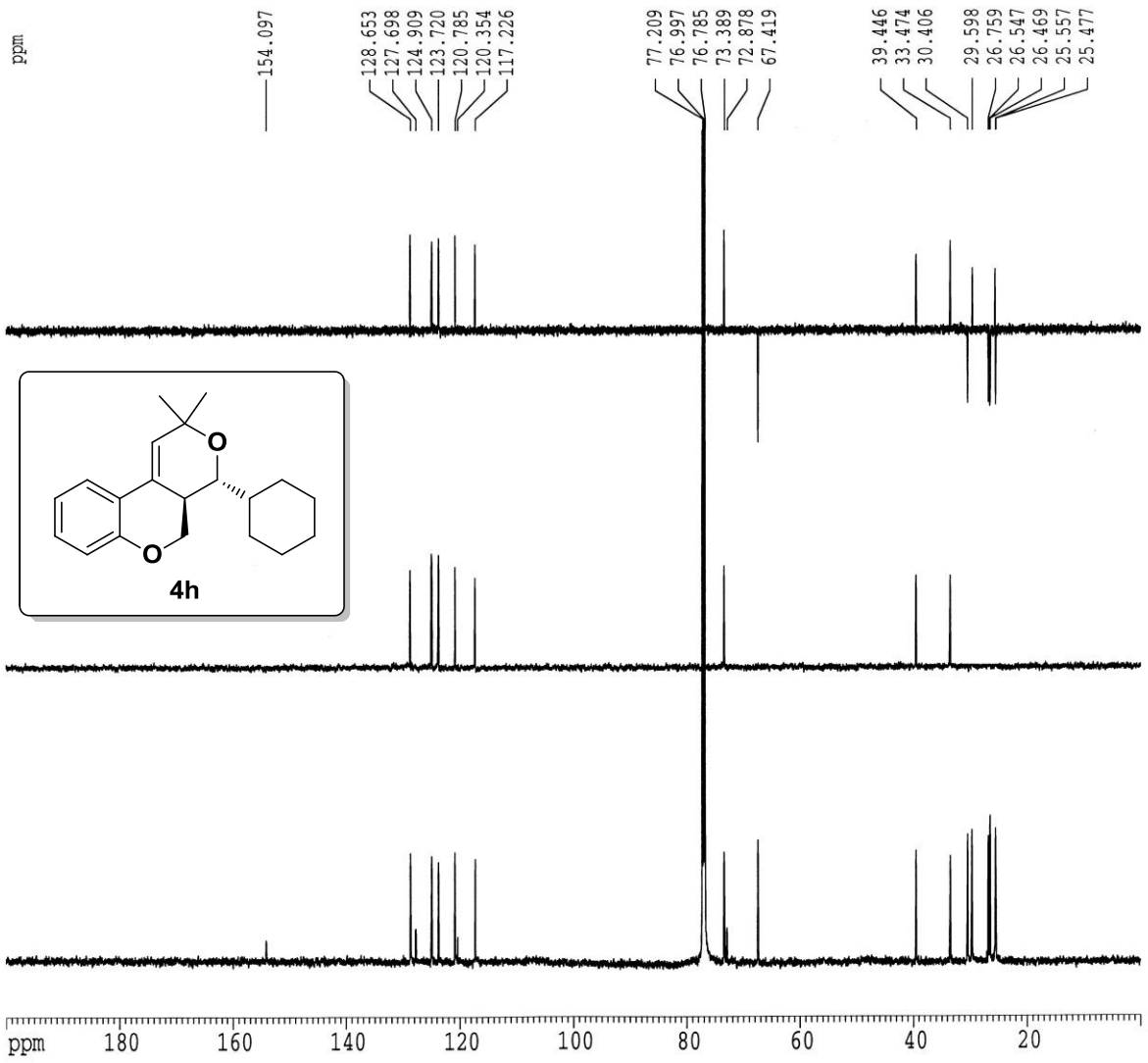


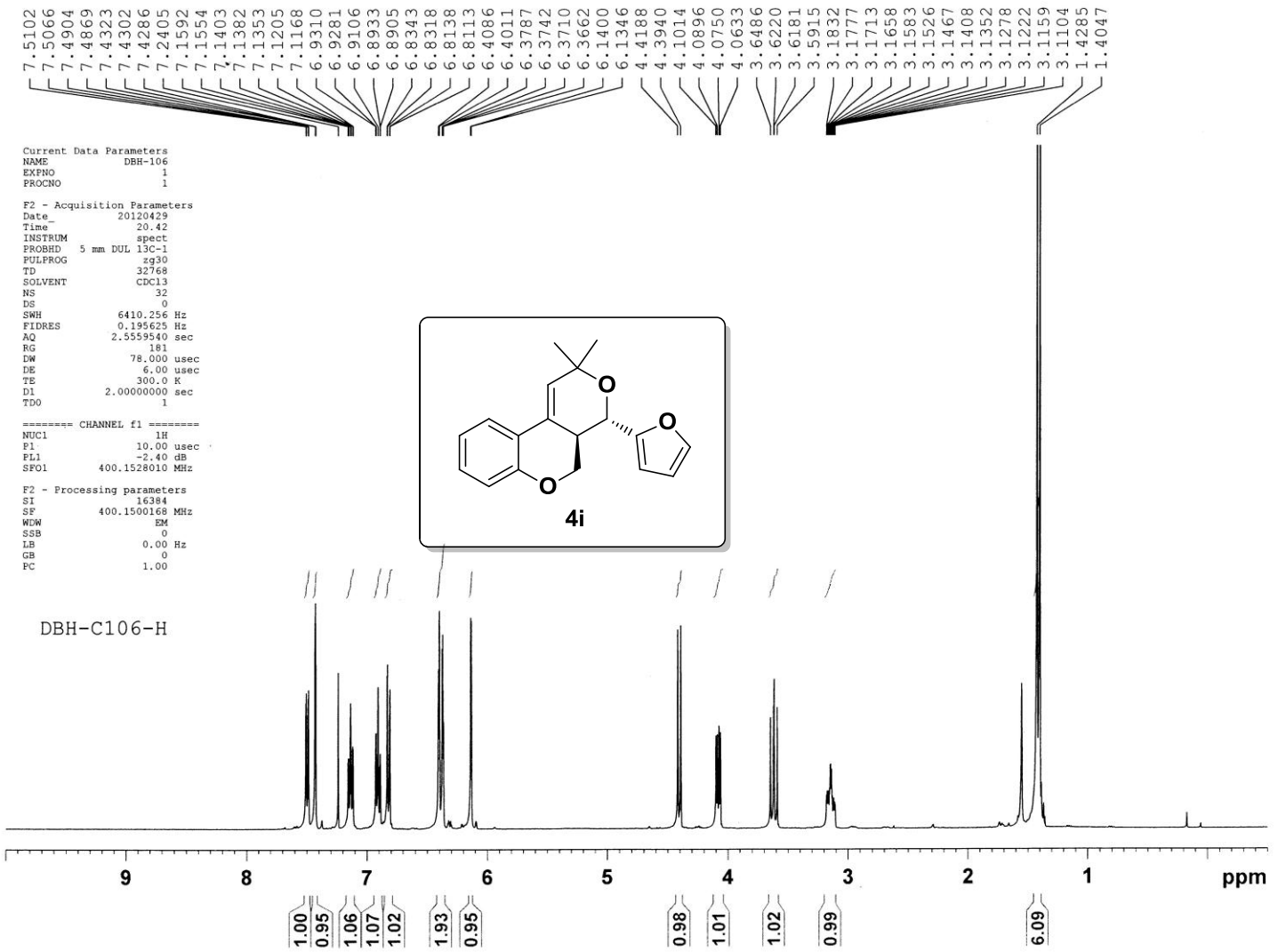


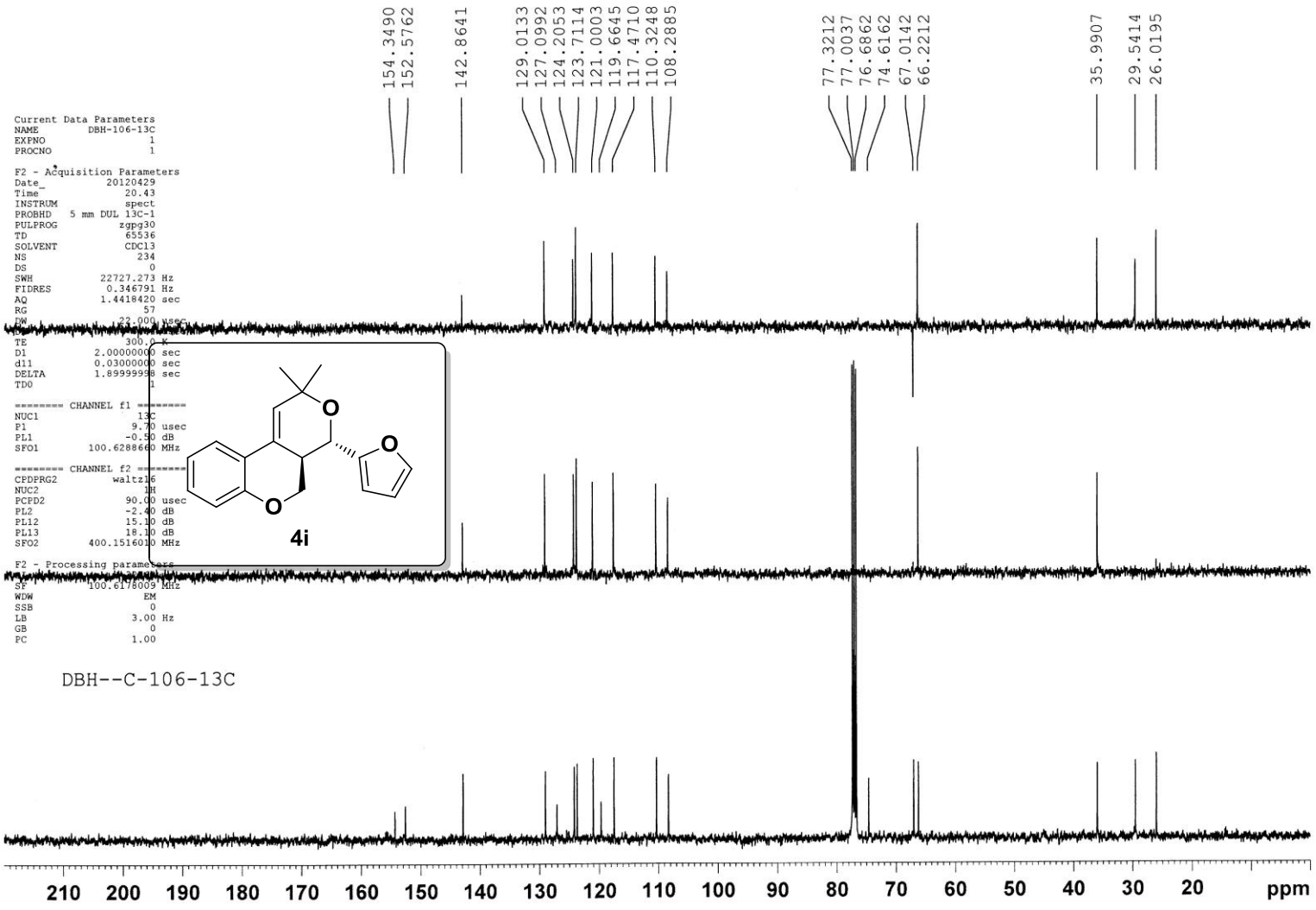
```
Current Data Parameters
NAME          DBH-C-123
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20120513
Time              21.39
INSTRUM           spect
PROBHD   5 mm QNP 1H/1
PULPROG             zg
TD                32768
SOLVENT           CDCl3
NS                   32
DS                    0
SWH            8389.262 Hz
FIDRES         0.256020 Hz
AQ             1.9530228 sec
RG                  512
DW               59.600 usec
DE                6.50 usec
TE                298.6 K
D1           2.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                9.80 usec
PL1               0.00 dB
SFO1        598.9029945 MHz

F2 - Processing parameters
SI                32768
SF          598.9000259 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                12.00 cm
F1P              10.000 ppm
F1                5989.00 Hz
F2P              -0.500 ppm
F2               -299.45 Hz
PPMCM             0.52500 ppm/cm
HZCM          314.42252 Hz/cm
```



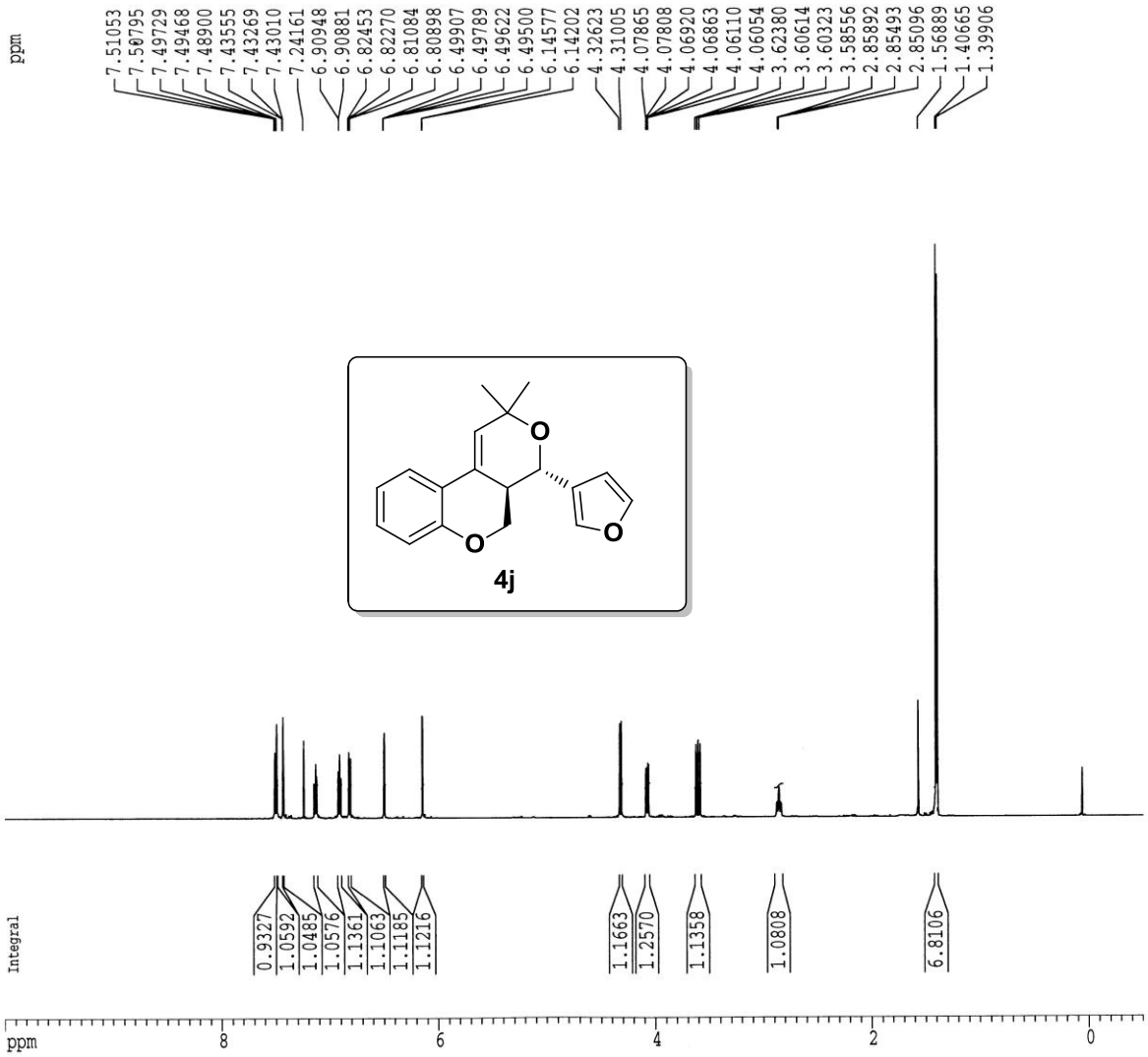
```
Current Data Parameters
NAME          DBH-C-123
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20120516
Time               5.37
INSTRUM           spect
PROBHD   5 mm QNP  1H/1
PULPROG           zgpg
TD               32768
SOLVENT          CDCl3
NS                6144
DS                   0
SWH          45045.047 Hz
FIDRES        1.374666 Hz
AQ            0.3637748 sec
RG                4096
DW              11.100 usec
DE               6.50 usec
TE               299.4 K
D1           3.50000000 sec
d11          0.03000000 sec
DELTA        3.40000010 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1               13C
P1                4.80 usec
PL1               0.00 dB
SFO1        150.6100903 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            92.00 usec
PL2             120.00 dB
PL12             11.30 dB
PL13             14.00 dB
SFO2        598.9029990 MHz

F2 - Processing parameters
SI                65536
SF          150.5935265 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                20.00 cm
F1P              200.000 ppm
F1             30118.71 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM         10.00000 ppm/cm
HZCM        1505.93518 Hz/cm
```

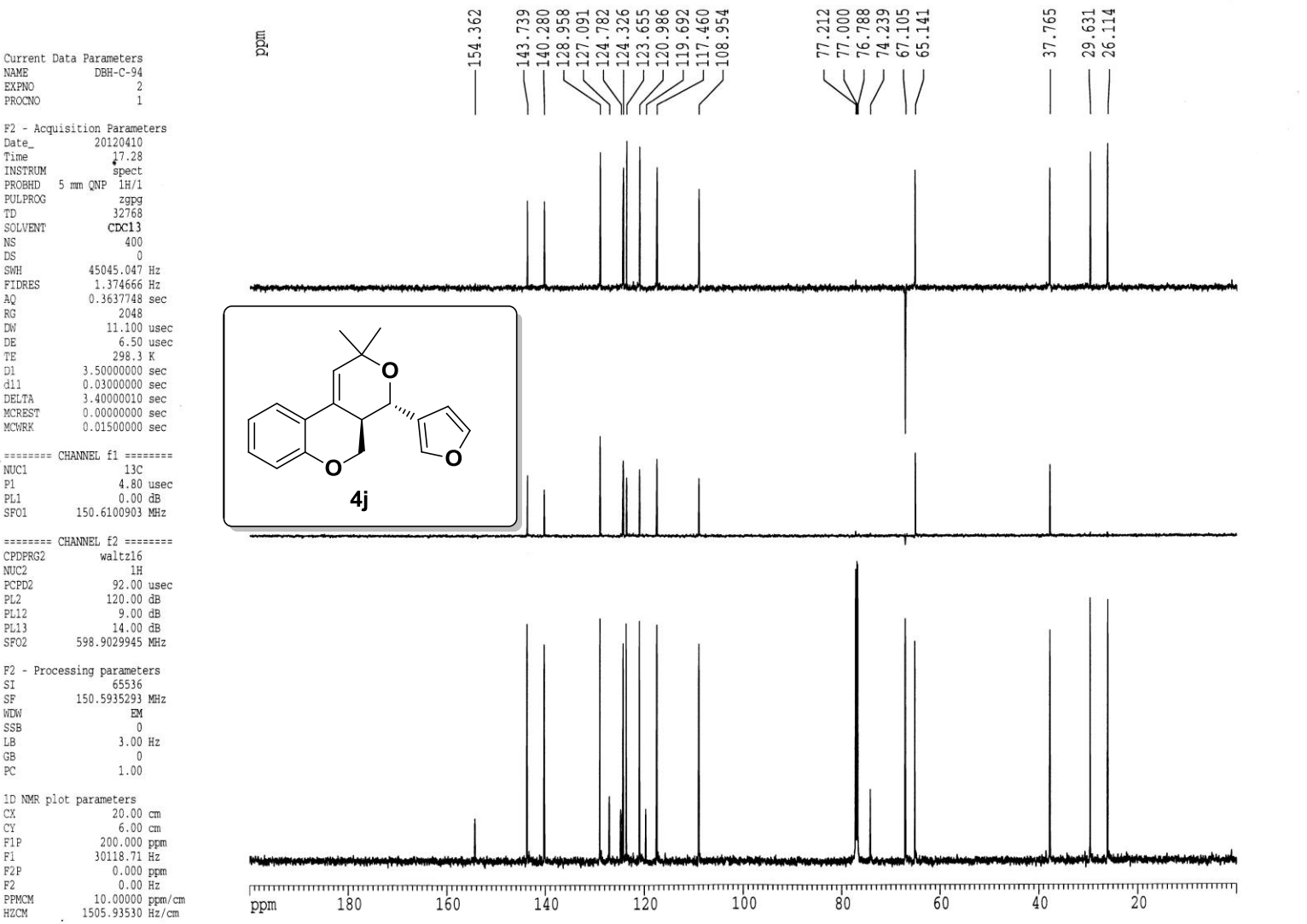


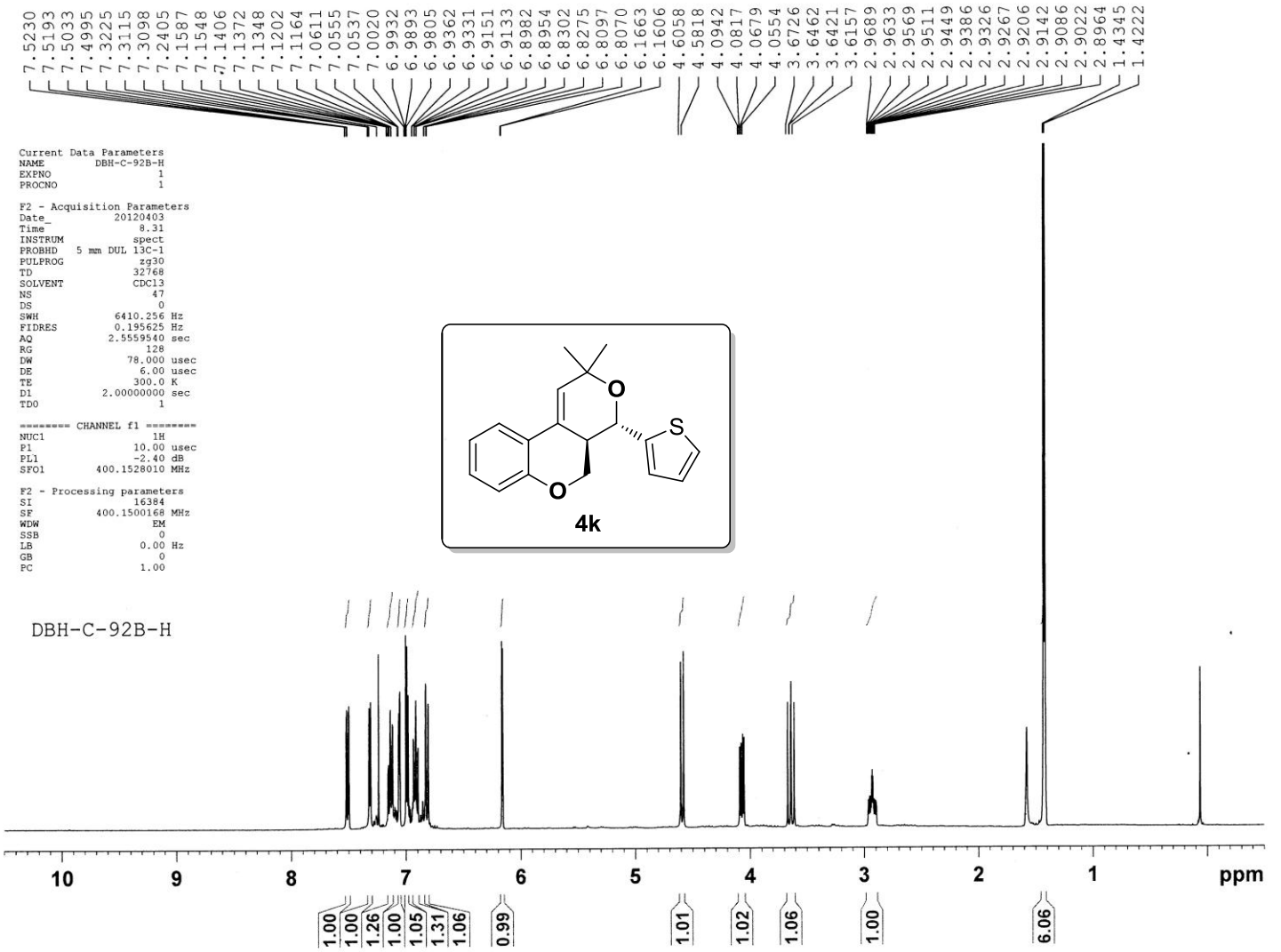


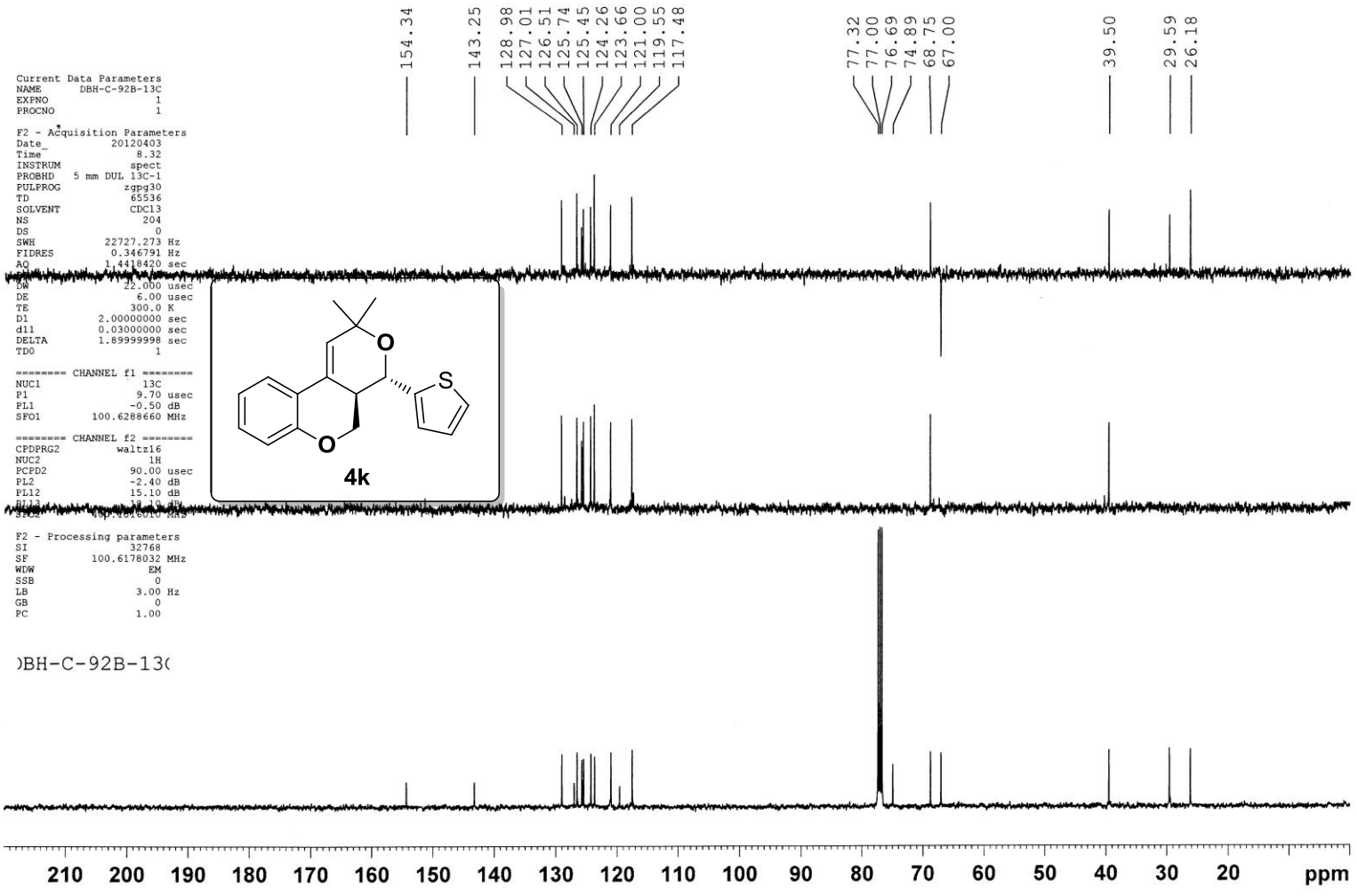


```
Current Data Parameters
NAME                  DBH-C-94
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_              20120410
Time                    17.09
INSTRUM                 spect
PROBHD    5 mm QNP  1H/1
PULPROG                 zg
TD                      32768
SOLVENT                 CDCl3
NS                         16
DS                          0
SWH                  8389.262 Hz
FIDRES               0.256020 Hz
AQ                   1.9530228 sec
RG                        128
DW                     59.600 usec
DE                      6.50 usec
TE                     297.3 K
D1                3.50000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                       9.50 usec
PL1                      0.00 dB
SFO1              598.9029945 MHz

F2 - Processing parameters
SI                      32768
SF                598.9000238 MHz
WDW                        no
SSB                         0
LB                       0.00 Hz
GB                          0
PC                       1.00

1D NMR plot parameters
CX                      20.00 cm
CY                      10.00 cm
F1P                     10.000 ppm
F1                    5989.00 Hz
F2P                     -0.500 ppm
F2                    -299.45 Hz
PPMCM                 0.52500 ppm/cm
HZCM                314.42252 Hz/cm
```

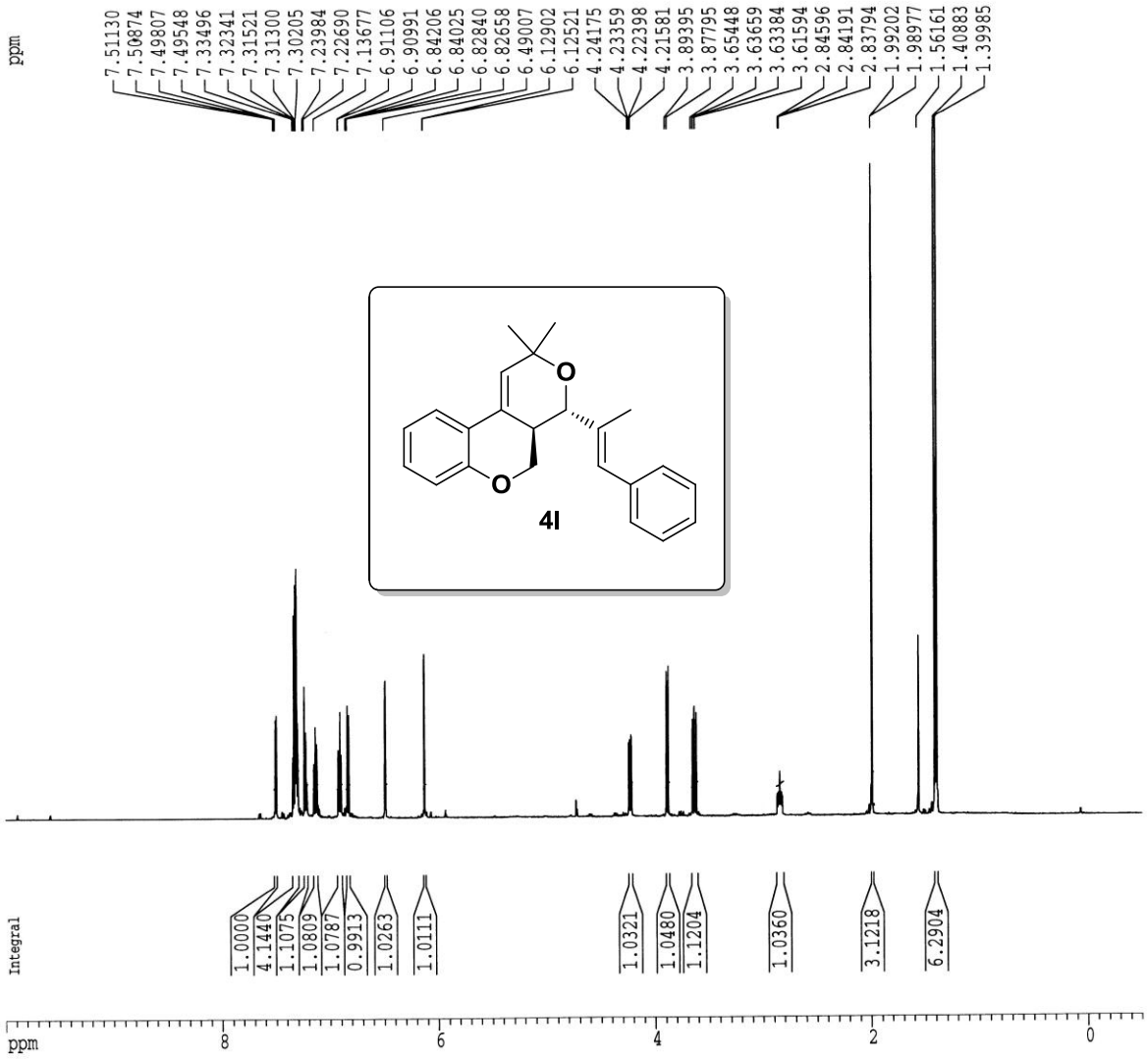




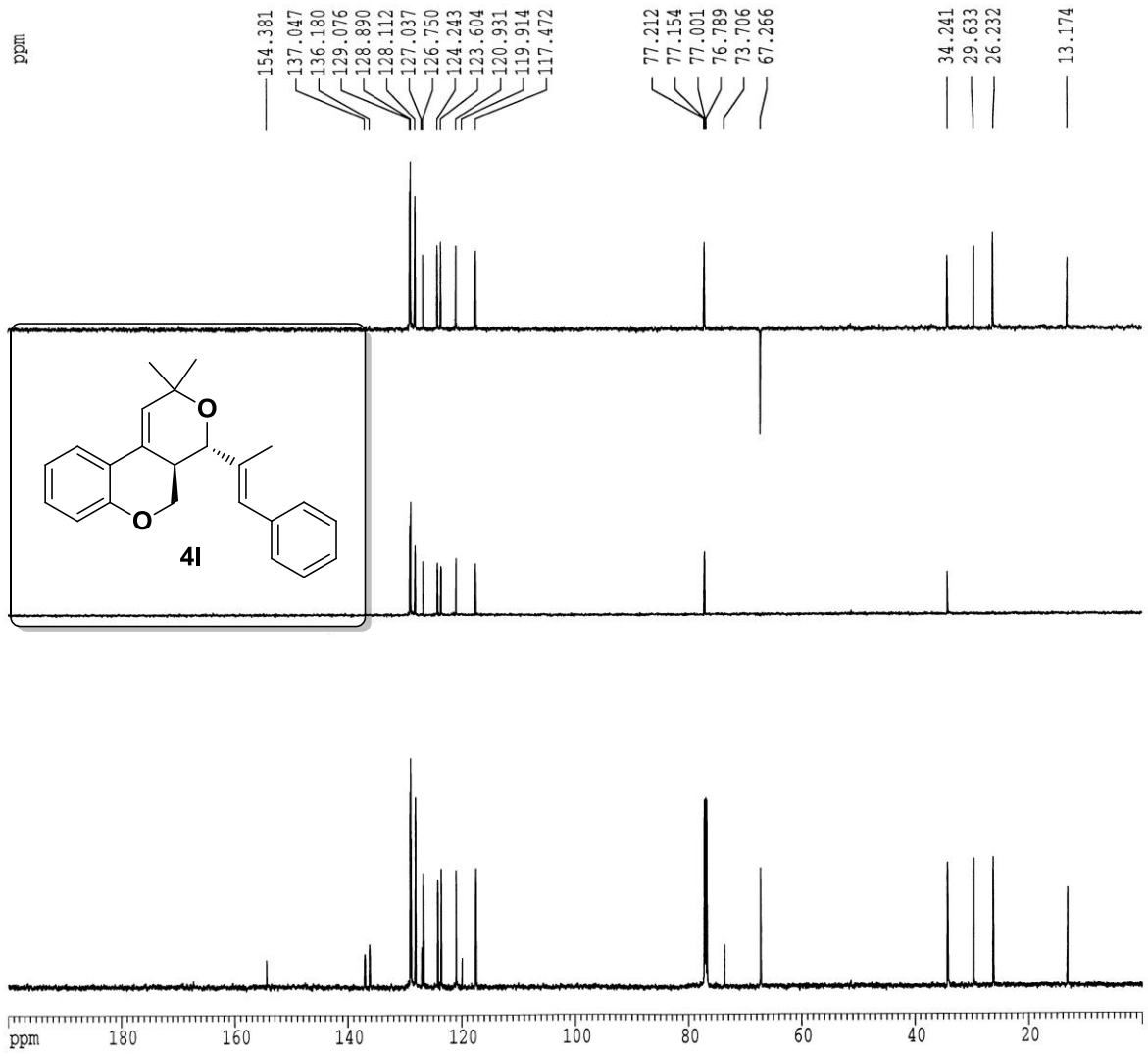




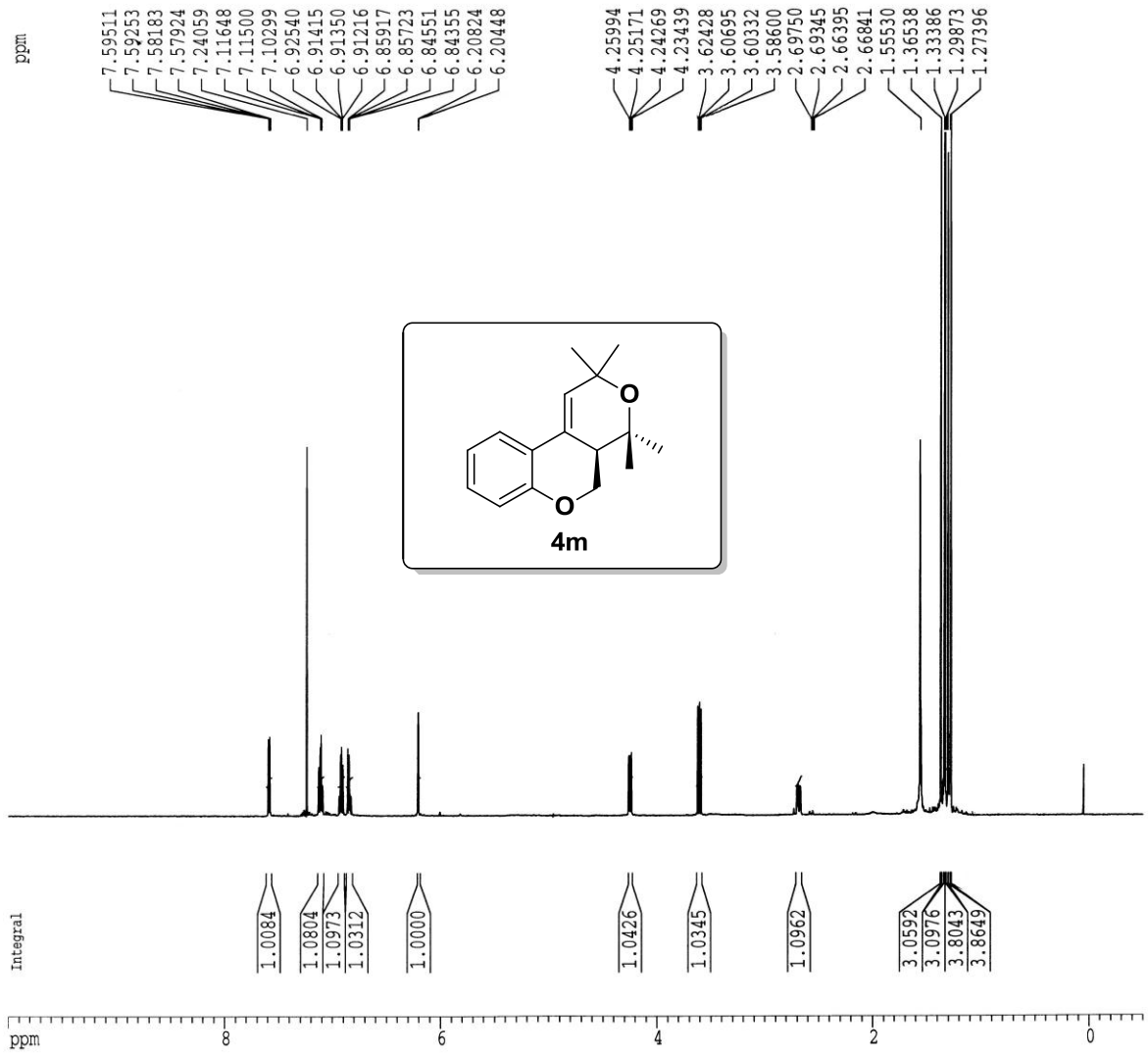
```
Current Data Parameters
NAME                  DBH-C-97
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_              20120412
Time                   15.30
INSTRUM                spect
PROBHD    5 mm QNP  1H/1
PULPROG                   zg
TD                     32768
SOLVENT                CDCl3
NS                        16
DS                         0
SWH                 8389.262 Hz
FIDRES              0.256020 Hz
AQ                  1.9530228 sec
RG                       128
DW                    59.600 usec
DE                     6.50 usec
TE                    296.7 K
D1                 3.50000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                      9.50 usec
PL1                     0.00 dB
SFO1               598.9029945 MHz

F2 - Processing parameters
SI                     32768
SF                598.9000251 MHz
WDW                       no
SSB                        0
LB                      0.00 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                     20.00 cm
CY                     15.00 cm
F1P                   10.000 ppm
F1                  5989.00 Hz
F2P                   -0.500 ppm
F2                  -299.45 Hz
PPMCM                0.52500 ppm/cm
HZCM               314.42252 Hz/cm
```



```
Current Data Parameters
NAME            DBH-C-97
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120412
Time               12.46
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG             zgpg
TD                 32768
SOLVENT           CD2Cl2
NS                    400
DS                      0
SWH            45045.047 Hz
FIDRES          1.374666 Hz
AQ              0.3637748 sec
RG                   2048
DW                11.100 usec
DE                 6.50 usec
TE                297.5 K
D1            3.50000000 sec
d11           0.03000000 sec
DELTA         3.40000010 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  4.80 usec
PL1                 0.00 dB
SFO1         150.6100903 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              92.00 usec
PL2               120.00 dB
PL12                9.00 dB
PL13               14.00 dB
SFO2         598.9029945 MHz

F2 - Processing parameters
SI                  65536
SF           150.5935286 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  20.00 cm
CY                   4.00 cm
F1P                200.000 ppm
F1               30118.71 Hz
F2P                  0.000 ppm
F2                   0.00 Hz
PPMCM             10.00000 ppm/cm
HZCM            1505.93518 Hz/cm
```



```
Current Data Parameters
NAME                 DBH-C-111
EXPNO                        1
PROCNO                       1

F1 - Acquisition Parameters
Date_               20120503
Time                   14.52
INSTRUM                spect
PROBHD    5 mm QNP  1H/1
PULPROG                   zg
TD                     32768
SOLVENT                CDCl3
NS                        16
DS                         0
SWH                 8389.262 Hz
FIDRES                0.256020 Hz
AQ                 1.9530228 sec
RG                       512
DW                    59.600 usec
DE                     6.50 usec
TE                     297.2 K
D1                2.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                      9.80 usec
PL1                     0.00 dB
SFO1              598.9029945 MHz

F2 - Processing parameters
SI                         32768
SF                598.9000256 MHz
WDW                           no
SSB                            0
LB                          0.00 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                         20.00 cm
CY                         15.00 cm
F1P                        10.000 ppm
F1                       5989.00 Hz
F2P                        -0.500 ppm
F2                       -299.45 Hz
PPMCM                     0.52500 ppm/cm
HZCM                    314.42252 Hz/cm
```



```
Current Data Parameters
NAME          DBH-C-111
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20120503
Time               4.24
INSTRUM           spect
PROBHD   5 mm QNP  1H/1
PULPROG           zgpg
TD               32768
SOLVENT          CDCl3
NS                5120
DS                   0
SWH          45045.047 Hz
FIDRES        1.374666 Hz
AQ            0.3637748 sec
RG                4096
DW              11.100 usec
DE               6.50 usec
TE              298.7 K
D1           3.50000000 sec
d11          0.03000000 sec
DELTA        3.40000010 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1               13C
P1               4.50 usec
PL1              0.00 dB
SFO1       150.6100903 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2               1H
PCPD2           92.00 usec
PL2            120.00 dB
PL12            11.30 dB
PL13            14.00 dB
SFO2       598.9029990 MHz

F2 - Processing parameters
SI                65536
SF          150.5935272 MHz
WDW                  EM
SSB                   0
LB                3.00 Hz
GB                    0
PC                1.00

1D NMR plot parameters
CX               20.00 cm
CY               25.00 cm
F1P             200.000 ppm
F1             30118.71 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM         10.00000 ppm/cm
HZCM        1505.93518 Hz/cm
```