Electronic Supplementary Material (ESI) for Chemical Communications

Supplementary Information

Stereocontrolled Generation of Highly Nucleophilic (*Z*)- or (*E*)-α-Fluoroalkenylchromium Reagents *via* Carbon-Fluorine Bond Activation: Highly Stereoselective Syntheses of (*E*)- or (*Z*)-β-Fluoroallylic Alcohols

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Content

General methods

Infrared spectra (IR) were determined in a liquid film on a NaCl plate or KBr disk method with a JASCO FT/IR-4100 typeA spectrometer. ^{1}H and ^{13}C NMR spectra were measured with a JEOL JNM-AL 400 NMR spectrometer in a chloroform-*d* ($CDCl_3$) solution with $CHCl_3$ (7.26) and $CDCl_3$ (77.0) as an internal reference. A JEOL JNM-EX90A (84.21 MHz) FT-NMR spectrometer and a JEOL JNM-AL 400 NMR spectrometer were used for determining the yield of the products with hexafluorobenzene (C_6F_6). ^{19}F NMR (376.05 MHz) spectra were measured with a JEOL JNM-AL 400 NMR spectrometer in a chloroform-*d* ($CDCl_3$) solution with trichlorofluoromethane ($CFCl_3$) as an internal standard. High-resolution mass spectra (HRMS) were taken on a JEOL JMS-700MS spectrometer by electron impact (EI), chemical ionization (CI), and fast atom bombardment (FAB) methods.

All reactions were routinely monitored by ^{19}F NMR spectroscopy or TLC, and carried out under an atmosphere of argon.

Materials

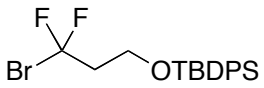
DMF were fleshly distilled from calcium hydride (CaH_2). All chemicals were of reagent grade and, if necessary, were purified in the usual manner prior to use. Thin-layer chromatography (TLC) was done with Merck silica gel 60 F_{254} plates, and column chromatography was carried out with Wako gel C-200. The compounds, **1A**,[1] **1C**,[2] and **1D**[3] were prepared according to the literature.

Preparation of *tert*-butyldiphenylsilyloxy-1-bromo-1,1-difluoropropane (1B) :

Under an argon atmosphere, a solution of ethyl 3-bromo-3,3-difluoropropanoate[2] (0.65 g, 3.0 mmol) in CH_2Cl_2 was dropwise added to diisobutylaluminium hydride (DIBAL-H) (1.0 M Hexane solution, 6.9 mL, 6.9 mmol) at −78 °C. After stirring at that temperature for 2 h, the mixture was stirred at 0 °C for 3 h. The reaction was quenched with 10% HCl aq., and the mixture was extracted with CH_2Cl_2. The organic layers were dried over anhydrous sodium sulfate, filtered, and concentrated under reduce pressure. The resulting residue was used without purification.

A mixture of the crude materials, *tert*-buthyldiphenylchlorosilane (1.65 g, 6.0 mmol), and imidazole (0.41 g, 6.0 mmol) in THF (6.0 mL) was stirred at room temperature for 20 h. Addition of water followed by extractive workup and purification by column chromatography (Hexane/AcOEt = 80:1) afforded the desired product (0.60 g, 1.46 mmol, 49% yield).

tert-Butyldiphenylsilyloxy-1-bromo-1,1-difluoropropane (1B)



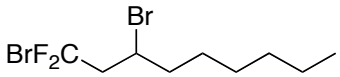
^{1}H NMR ($CDCl_3$) δ = 1.07 (9H, s), 2.63–2.69 (2H, m), 3.93 (2H, t, *J* = 6.59 Hz), 7.39–7.46 (6H, m), 7.67–7.69 (4H, m); ^{13}C NMR ($CDCl_3$) δ = 19.1, 26.7, 46.7 (t, *J* = 20.6 Hz), 58.8 (t, *J* = 4.1 Hz), 121.2 (t, *J* = 304.2 Hz), 127.8, 129.8, 133.1, 135.5; ^{19}F NMR ($CDCl_3$) δ = –42.39 (1F, t, *J* = 13.35 Hz); IR (neat) : 3428, 3072, 3000, 2932, 2858, 1960, 1590, 1472, 1428, 1362, 1283, 1188, 1114, 998, 880, 823, 702, 613 cm^{-1}; HRMS (FAB) calcd for $C_{19}H_{24}BrF_2OSi$ (M+H) 413.0748, found 413.0751.

Preparation of 1-bromo-1,1-difluorononane (1E) :

Under an argon atmosphere, a three-necked, round-bottomed flask equipped with an efficient magnetic stirring bar was charged with copper(I) chloride (0.040 g, 0.4 mmol), ethanol amine (1.22 g, 20.0 mmol), *tert*-butanol (6.6 mL), 1-octene (4.49 g, 40 mmol), and dibromodifluoromethane (16.79 g, 80 mmol). After stirring of the reaction mixture at 85 °C for 48 h, all organic materials were filtered through silica gel, which was rinsed with hexanes. Then the resulting colorless filtrate was concentrated by evaporation. The residure was subjected to fractional distillation to afford 1,3-dibromo-1,1-difluorononane (8.86 g, 27.7 mmol, 69% yield) as a colorless liquid.

Under an argon atmosphere, a three-necked round-bottomed flask equipped with an ice-H_2O condenser was charged with the above obtained 1,3-dibromo-1,1-difluorononane and 28 mL of anhydrous DMSO. Sodium borohydride (1.51 g, 40 mmol) was then added in small portions with vigorous stirring over the course of 15 min. After addition was complete, the bath temperature was raised to 70 °C and the reaction mixture was stirred at the temperature for 6 h. Then, the reaction was cooled to room temperature, and the contents were transferred to erlenmeyer flask, the reaction being quenched with chips of ice. The resulting mixture was carefully acidified with conc. HCl aq., and the aqueous DMSO layer was extracted with Et_2O three times. The combined Et_2O layers were washed with H_2O, dried over anhydrous Na_2SO_4, and subjected to ambient pressure fractional distillation to afford 1-bromo-1,1-difluorononane (contaminated with a small amount of 1,1-difluorononane) (1.87 g, 7.7 mmol, 38% yield) as a colorless liquid.

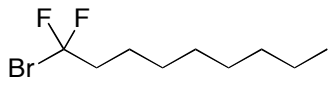
1,3-Dibromo-1,1-difluorononane



^{1}H NMR ($CDCl_3$) δ = 0.89 (3H, t, *J* = 6.79 Hz), 1.27–1.58 (8H, m), 1.82–1.89 (2H, m), 2.92–3.10 (2H, m), 4.22–4.25 (1H, m); ^{13}C NMR ($CDCl_3$) δ = 14.0, 22.5, 27.0 28.4, 31.6, 38.4, 46.9, 52.6 (t, *J* = 21.08 Hz), 120.6 (t, *J* = 305.8 Hz); ^{19}F NMR

($CDCl_3$) δ = –43.24 (2F, t, *J* = 13.35 Hz); IR (neat) : 2929, 2859, 1466, 1430, 1378, 1198, 1129, 1091, 979, 925, 769, 725 cm^{-1}; HRMS (EI) calcd for $C_9H_{16}Br_2F_2$ (M^+) 319.9587, found 319.9594.

1-Bromo-1,1-difluorononane (1E)

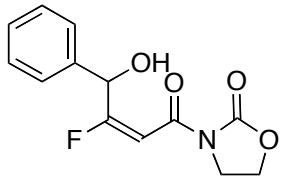


^{1}H NMR ($CDCl_3$) δ = 0.89 (3H, t, *J* = 6.79 Hz), 1.26–1.38 (10H, m), 1.57–1.65 (2H, m), 2.28–2.35 (2H, m); ^{13}C NMR ($CDCl_3$) δ = 14.1, 22.7, 23.9 (t, *J* = 2.81 Hz), 28.5, 29.1, 29.2, 31.8, 44.3 (t, *J* = 21.08 Hz), 123.3 (t, *J* = 303.8 Hz); ^{19}F NMR ($CDCl_3$) δ = –43.82 (2F, t, *J* = 14.67 Hz); IR (neat) : 2927, 2858, 1467, 1378, 1197, 1129, 1092, 962, 912 cm^{-1}

Typical procedure for the *E*-selective chromium(II)-mediated reductive coupling of 3-(3-bromo-3,3-difluoropropanoyl)-2-oxazolidinone (1A) with benzaldehyde

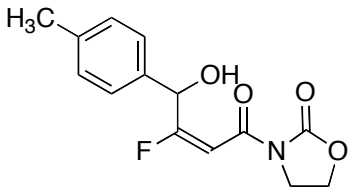
To a suspentsion of $CrCl_2$ (0.25 g, 2.0 mmol) and LiI (0.013 g, 0.1 mmol) in DMF (1.5 mL) were added benzaldehyde (0.11 g, 1.0 mmol) and 3-(3-bromo-3,3-difluoropropanoyl)-2-oxazolinone (**1A**) (0.13 g, 0.5 mmol) at 0 °C. After stirring at that temperature for 4 h, the reaction was quenched with ice-cold water. The whole was extracted with Et_2O three times. The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated in *vacuo*. The resulting residue was purified by silica gel column chromatography (Benzen/AcOEt = 3/1) to afford the pure product **2Aa** (0.11 g, 0.42 mmol, 84% yield).

3-[(*E*)-3-Fluoro-4-hydroxy-4-phenyl-2-butenoyl]-2-oxazolidinone (2Aa)



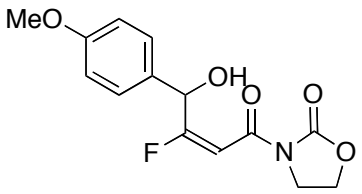
Yield : 84%; ^{1}H NMR (Acetone-d_6) δ = 4.12 (2H, t, *J* = 8.03 Hz), 4.48–4.52 (2H, m), 5.31 (1H, d, *J* = 6.00 Hz), 6.47 (1H, dd, *J* = 6.00, 27.23 Hz), 7.04 (1H, d, *J* = 21.33 Hz), 7.31–7.59 (5H, m); ^{13}C NMR (Acetone-d_6) δ = 43.6, 63,2, 69.0 (d, *J* = 22.0 Hz), 101.8 (d, *J* = 30.2 Hz), 127.2, 128.7, 129.1, 140.5 (d, *J* = 1.3 Hz), 154.4, 164.5 (d, *J* = 24.7 Hz), 175.4 (d, *J* = 280.6 Hz); ^{19}F NMR (Acetone-d_6) δ = –97.43 (1F, dd, *J* = 21.33, 27.23 Hz); HRMS (FAB) calcd for $C_{13}H_{12}FNO_4$ (M^+) 265.0750, found 265.0750.

3-[(*E*)-3-Fluoro-4-hydroxy-4-(4-methylphenyl)-2-butenoyl]-2-oxazolidinone (2Ab)



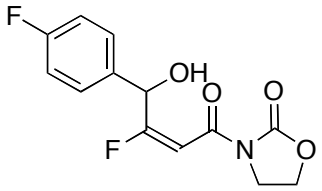
Yield : 64%; ^{1}H NMR (Acetone-d_6) δ = 2.36 (3H, s), 4.13 (2H, t, *J* = 8.06 Hz), 4.48–4.52 (2H, m), 5.26 (1H, d, *J* = 6.11 Hz), 6.47 (1H, dd, *J* = 6.11, 27.13 Hz), 7.11 (1H, d, *J* = 21.36 Hz), 7.22 (2H, d, *J* = 7.97 Hz), 7.51 (2H, d, *J* = 7.97 Hz); ^{13}C NMR (Acetone-d_6) δ = 21.1, 43.6, 63.1, 69.0 (d, *J* = 22.1 Hz), 101.6 (d, *J* = 30.2 Hz), 127.2, 129.7, 137.5, 138.3, 154.4, 164.5 (d, *J* = 24.7 Hz), 175.6 (d, *J* = 280.6 Hz); ^{19}F NMR (Acetone-d_6) δ = –98.73 (1F, dd, *J* = 21.36, 27.13 Hz); HRMS (FAB) calcd for $C_{14}H_{14}FNO_4$ (M^+) 279.0907, found 279.0916.

3-[(*E*)-3-Fluoro-4-hydroxy-4-(4-methoxylphenyl)-2-butenoyl]-2-oxazolidinone (2Ac)



Yield : 69%; ^{1}H NMR (Acetone-d_6) δ = 3.79 (3H, s), 4.11 (2H, t, *J* = 8.05 Hz), 4.46–4.51 (2H, m), 5.18 (1H, d, *J* = 6.00 Hz), 6.39 (1H, dd, *J* = 6.00, 27.01 Hz), 6.92 (2H, d, *J* = 8.58 Hz), 7.04 (1H, d, *J* = 21.31 Hz), 7.49 (d, *J* = 8.58 Hz); ^{13}C NMR (Acetone-d_6) δ = 43.6, 55.5, 63.1, 68.7 (d, *J* = 22.0 Hz), 101.4 (d, *J* = 30.2 Hz), 114.5, 128.5, 132.5, 154.4, 160.5, 164.1 (d, *J* = 24.7 Hz), 175.6 (d, *J* = 280.6 Hz); ^{19}F NMR (Acetone-d_6) δ = –98.67 (1F, dd, *J* = 21.31, 27.01 Hz); HRMS (FAB) calcd for $C_{14}H_{14}FNO_5$ (M^+) 295.0856, found 295.0863.

3-[(*E*)-3-Fluoro-(4-fluorophenyl)-4-hydroxy-2-butenoyl]-2-oxazolidinone (2Ad)

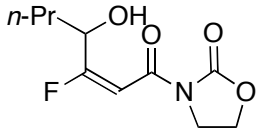


Yield : 90%; ^{1}H NMR (Acetone-d_6) δ = 4.13 (2H, t, *J* = 8.17 Hz), 4.47–4.53 (2H, m), 5.37 (1H, d, *J* = 5.84 Hz), 6.46 (1H, dd, *J* = 5.84, 27.09 Hz), 7.06 (1H, d, *J* = 21.29 Hz), 7.14 (2H, m), 7.58–7.63 (2H, m); ^{13}C NMR (Acetone-d_6) δ = 43.6, 63.2, 68.3 (d, *J* = 21.9 Hz), 101.9 (d, *J* = 29.8 Hz), 115.8 (d, *J* = 21.6 Hz), 129.17 (d, *J* = 8.2 Hz), 136.6, 154.4, 163.3 (d, *J* = 244.4 Hz), 164.5 (d, *J* = 24.4 Hz), 175.6 (d, *J* = 280.4 Hz); ^{19}F NMR (Acetone-d_6) δ = –115.06 to –114.87 (1F, m), –98.29 (1F, dd, *J* = 21.29, 27.09 Hz); HRMS (FAB) calcd for $C_{13}H_{12}F_2NO_4$ (M^+) 284.0734, found 284.0726.

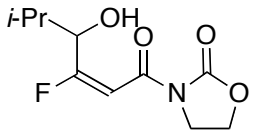
3-[(*E*)-3-Fluoro-4-hydroxy-2-heptenoyl]-2-oxazolidinone (2Af)

Yield : 68%; ^{1}H NMR (Acetone-d_6) δ = 0.94 (3H, t, *J* = 7.38 Hz), 1.35–1.78 (4H, m), 4.07 (2H, t, *J* = 8.05 Hz), 4.43–4.49 (3H, m), 5.10–5.25 (1H, m),

6.99 (1H, d, J = 22.83 Hz); ^{13}C NMR (Acetone-d_6) δ = 14.1, 19.3, 36.6, 43.5, 63.1, 67.6 (d, J = 22.9 Hz), 101.3 (d, J = 30.9 Hz), 154.4, 164.2 (d, J = 25.02 Hz), 177.5 (d, J = 280.4 Hz); ^{19}F NMR (Acetone-d_6) δ = –97.38 (1F, dd, J = 22.83, 26.89 Hz).

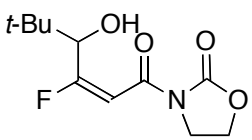


3-[(*E*)-3-Fluoro-4-hydroxy-5-methyl-2-hexenoyl]-2-oxazolidinone (2Ag)



Yield : 77%; ^{1}H NMR (Acetone-d_6) δ = 0.92 (3H, d, J = 6.82 Hz), 1.05 (3H, d, J = 6.66 Hz), 1.91–1.99 (1H, m), 4.06 (2H, t, J = 8.03 Hz), 4.48 (2H, t, J = 8.03 Hz), 4.57 (1H, d, J = 7.08 Hz), 4.80–4.90 (1H, m), 7.05 (d, J = 21.83 Hz); ^{13}C NMR (Acetone-d_6) δ = 18.85, 18.86, 32.4, 43.5, 63.0, 73.1 (d, J = 22.5 Hz), 102.2 (d, J = 30.7 Hz), 154.3, 164.4 (d, J = 25.5 Hz), 177.0 (d, J = 280.6 Hz); ^{19}F NMR (Acetone-d_6) δ = –95.00 (1F, dd, J = 21.83, 29.33 Hz); HRMS (FAB) calcd for $C_{10}H_{15}FNO_4$ (M+H) 232.0985, found 232.0984.

3-[(*E*)-3-Fluoro-4-hydroxy-5,5-dimethyl-2-hexenoyl]-2-oxazolidinone (2Ah)



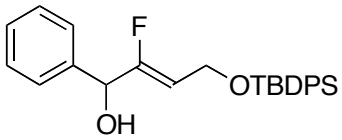
Yield : 77%; ^{1}H NMR (Acetone-d_6) δ = 1.00 (9H, s), 4.07 (2H, dt, J = 2.11, 8.00 Hz), 4.48 (2H, t, J = 8.00 Hz), 4.63 (1H, d, J = 6.93 Hz), 5.04 (1H, dd, J = 6.93, 30.24 Hz), 7.09 (1H, d, J = 23.08 Hz); ^{13}C NMR (Acetone-d_6) δ = 26.5, 36.3, 43.6, 63.0, 74.4 (d, J = 20.5 Hz), 103.0 (d, J = 31.3 Hz), 154.4, 164.5 (d, J = 25.9 Hz), 176.6 (d, J = 282.8 Hz); ^{19}F NMR (Acetone-d_6) δ = –87.58 (1F, dd, J = 23.08, 30.24 Hz); HRMS (FAB) calcd for $C_{11}H_{17}FNO_4$ (M+H) 246.1142, found 246.1141.

Typical procedure for the *Z*-selective chromium(II)-mediated reductive coupling of 3-(*tert*-butyldiphenylsilyloxy) -1-bromo-1,1-difluoropropane (1B) with benzaldehyde

To a suspension of $CrCl_2$ (0.22 g, 1.8 mmol) and LiI (0.020 g, 0.15 mmol) in DMF (1.2 mL) were added benzaldehyde (0.064 g, 0.6 mmol) and 3-(*tert*-butyldiphenylsilyloxy)-1-bromo-1,1-difluloropropane (**1B**) (0.12 g, 0.3 mmol) at 0 °C. After stirring at room temperature for 4 h, the reaction was quenched with water. The whole was extracted with Et_2O three times. The combined organic layers were dried over anhydrous Na_2SO_4,

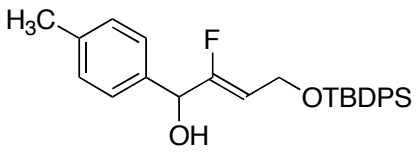
filtered, and concentrated in *vacuo*. The resulting residue was purified by silica gel column chromatography (Hexane/AcOEt = 5/1) to afford the pure product (0.071 g, 0.17 mmol, 56% yield).

(*Z*)-4-(*tert*-Butyldiphenylsilyloxy)-2-fluoro-1-phenyl-2-buten-1-ol (2Ba)



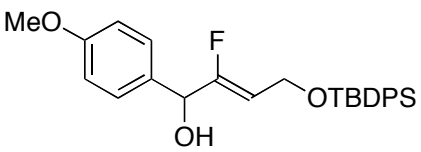
Yield : 56%; ^{1}H NMR ($CDCl_3$) δ = 0.95 (9H, s), 2.01 (1H, br), 4.26 (2H, dd, *J* = 2.00, 6.79 Hz), 5.03 (1H, d, *J* = 11.91 Hz), 5.09 (1H, dt, *J* = 36.72, 6.79 Hz), 7.20–7.34 (11H, m), 7.56–7.57 (4H, m); ^{13}C NMR ($CDCl_3$) δ = 19.1, 26.8, 57.1 (d, *J* = 6.6 Hz), 72.3 (d, *J* = 32.2 Hz), 107.0, (d, *J* = 10.7 Hz), 126.7, 127.6, 128.4, 128.5, 129.6, 133.5, 135.5, 139.1, 158.79 (d, *J* = 260.3 Hz); ^{19}F NMR ($CDCl_3$) δ = –119.05 (1F, dd, *J* = 11.91, 36.72 Hz); IR (neat) : 3392, 3070, 2931, 2858, 1709, 1589, 1494, 1472, 1428, 1262, 1164, 1111, 1060, 885, 740, 701, 612 cm^{-1}; HRMS (FAB) calcd for $C_{26}H_{29}FNaO_2Si$ (M+Na) 443.1819, found 443.1815.

(*Z*)-4-(*tert*-Butyldiphenylsilyloxy)-2-fluoro-1-(4-methylphenyl)-2-buten-1-ol (2Bb)



Yield : 48%; ^{1}H NMR ($CDCl_3$) δ = 1.05 (9H, s), 2.31 (1H, br), 2.35 (3H, s), 4.36 (2H, dd, *J* = 1.60, 6.49 Hz), 5.09 (1H, d, *J* = 11.86 Hz), 5.19 (1H, dt, *J* = 36.52, 6.49 Hz), 7.15–7.44 (10H, m), 7.64–7.70 (4H, m); ^{13}C NMR ($CDCl_3$) δ = 19.1, 21.2, 26.7, 57.1 (d, *J* = 6.6 Hz), 72.2 (d, *J* = 32.2 Hz), 106.9 (d, *J* = 9.9 Hz), 126.6, 127.6, 129.2, 129.6, 133.6, 135.6, 136.2, 138.2, 158.8 (d, *J* = 259.5 Hz); ^{19}F NMR ($CDCl_3$) δ = –119.12 (1F, dd, *J* = 11.86, 36.52 Hz); IR (neat) : 3395, 3071, 2930, 2858, 1079, 1513, 1472, 1428, 1362,1163, 1111, 1062, 800, 740, 702 cm^{-1}; HRMS (FAB) calcd for $C_{27}H_{31}FNaO_2Si$ (M+Na) 457.1975, found 457.1972.

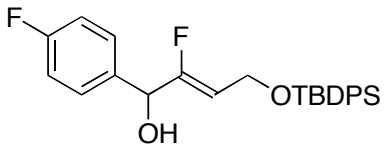
(*Z*)-4-(*tert*-Butyldiphenylsilyloxy)-2-fluoro-1-(4-methoxyphenyl)-2-buten-1-ol (2Bc)



Yield : 52%; ^{1}H NMR ($CDCl_3$) δ = 1.04 (9H, s), 2.00 (1H, br), 3.82 (3H, s), 4.36 (2H, dd, *J* = 2.20, 6.59 Hz), 5.09 (1H, dd, *J* = 3.20, 10.09 Hz), 5.19 (1H, dt, *J* = 36.63, 6.59 Hz), 6.88–6.90 (2H, m), 7.23–7.44 (8H, m), 7.63–7.68 (4H, m); ^{13}C NMR ($CDCl_3$) δ = 19.1, 26.8, 55.3, 57.8 (d, *J* = 6.6 Hz), 72.0 (d, *J* = 32.2 Hz), 106.8 (d, *J* = 11.6 Hz), 113.9, 127.6, 128.1, 129.6, 131.3, 133.6, 135.6, 158.9 (d, *J* = 259.5 Hz), 159.7; ^{19}F NMR ($CDCl_3$) δ = –118.95 (1F, dd, *J* = 10.09, 36.63 Hz); IR (neat) : 3419, 3071, 2931, 2857, 1709, 1611, 1512, 1464, 1428, 1250, 1111, 1036, 739, 703 cm^{-1}; HRMS (FAB) calcd for

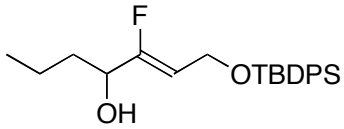
$C_{27}H_{30}FO_3Si$ (M-H) 449.1948, found 449.1938.

(*Z*)-4-(*tert*-Butyldiphenylsilyloxy)-2-fluoro-1-(4-fluorophenyl)-2-buten-1-ol (2Bd)



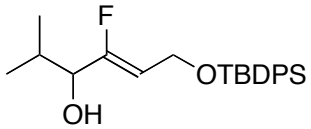
Yield : 56%; ^{1}H NMR ($CDCl_3$) δ = 1.06 (9H, s), 2.17 (1H, br), 4.37 (2H, d, *J* = 6.39 Hz), 5.12 (1H, d, *J* = 12.11 Hz), 5.18 (1H, dt, *J* = 36.72, 6.39 Hz), 7.03–7.07 (2H, m), 7.32–7.46 (8H, m), 7.64–7.72 (4H, m); ^{13}C NMR ($CDCl_3$) δ = 19.1, 26.8, 57.0 (d, *J* = 6.6 Hz), 71.7 (d, *J* = 32.2 Hz), 107.3 (d, *J* = 11.7 Hz), 115.4 (d, *J* = 27.8 Hz), 127.6, 128.5 (d, *J* = 8.2 Hz), 129.7, 133.5, 134.8 (d, *J* = 2.4 Hz), 135.5, 158.5 (d, *J* = 258.5 Hz), 162.6 (d, *J* = 246.2 Hz); ^{19}F NMR ($CDCl_3$) δ = –119.38 (1F, dd, *J* = 12.11, 36.72 Hz); IR (neat) : 3402, 3071, 2931, 2858, 1709, 1605, 1509, 1472, 1428, 1227, 1111, 1064, 908, 739, 703 cm^{-1}; HRMS (FAB) calcd for $C_{26}H_{28}F_2O_2Si$ (M^+) 438.1827, found 438.1833.

(*Z*)-1-(*tert*-Butyldiphenylsilyloxy)-3-fluoro-2-hepten-4-ol (2Bf)



Yield : 57%; ^{1}H NMR ($CDCl_3$) δ = 0.95 (3H, t, *J* = 7.39 Hz), 1.07 (9H, s), 1.26–1.70 (4H, m), 1.79 (1H, br), 4.00–4.07 (1H, m), 4.37 (2H, dd, *J* = 2.20, 6.59 Hz), 5.07 (1H, dt, *J* = 37.41, 6.59 Hz), 7.36–7.47 (6H, m), 7.68–7.73 (4H, m); ^{13}C NMR ($CDCl_3$) δ = 13.8, 18.5, 19.1, 26.8, 35.8, 57.0 (d, *J* = 7.43 Hz), 70.2 (d, *J* = 29.8 Hz), 106.1 (d, *J* = 11.7 Hz), 127.6, 129.6, 133.6, 135.5, 159.8 (d, *J* = 261.24 Hz); ^{19}F NMR ($CDCl_3$) δ = –122.19 (1F, dd, *J* = 15.61, 37.41 Hz); IR (neat) : 3389, 3071, 3050, 2999, 2959 , 2932, 2859, 1709, 1589, 1471, 1428, 1305, 1264, 1185, 1112, 939, 852, 822, 740, 702 cm^{-1}; HRMS (FAB) calcd for $C_{23}H_{31}FNaO_2Si$ (M+Na) 409.1975, found 409.1976.

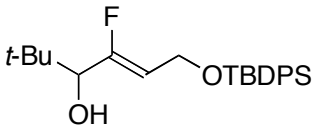
(*Z*)-6-(*tert*-Butyldiphenylsilyloxy)-4-fluoro-2-methyl-4-buten-3-ol (2Bg)



Yield : 59%; ^{1}H NMR ($CDCl_3$) δ = 0.92 (3H, d, *J* = 6.79 Hz), 0.97 (3H, d, *J* = 6.79 Hz), 1.07 (9H, s), 1.69 (1H, br), 1.87-1.91 (1H, m), 3.73 (1H, dd, *J* = 6.59, 17.05 Hz), 4.37 (2H, d, *J* = 6.49 Hz), 5.07 (1H, dt, *J* = 37.12, 6.49 Hz), 7.37–7.46 (6H, m), 7.69–7.71 (4H, m); ^{13}C NMR ($CDCl_3$) δ = 17.5, 18.8, 19.1, 26.5, 26.8, 57.0 (d, *J* = 7.4 Hz), 75.8 (d, *J* = 28.9 Hz), 107.1 (d, *J* = 11.6 Hz), 127.7, 129.7, 133.7, 135.6, 159.1 (d, *J* = 261.1 Hz); ^{19}F NMR ($CDCl_3$) δ = –121.25 (1F, dd, *J* = 17.05, 37.12 Hz); IR : (neat) 3404, 3071, 3050, 2960, 2931, 2858, 1708, 1471, 1428, 1264, 1112, 1059, 1026, 939, 876, 822, 782, 740, 702 cm^{-1}; HRMS

(FAB) calcd for $C_{23}H_{31}FNaO_2Si$ (M+Na) 409.1975, found 409.1976.

(*Z*)-6-(*tert*-Butyldiphenylsilyloxy)-4-fluoro-2,2-dimethyl-4-hexen-3-ol (2Bh)

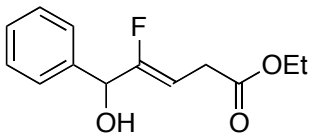


Yield : 63%; ^{1}H NMR ($CDCl_3$) δ = 0.94 (9H, s), 1.05 (9H, s), 1.71 (1H, br), 3.68 (1H, d, *J* = 18.81 Hz), 4.35–4.36 (2H, m), 5.03 (1H, dt, *J* = 37.38, 6.79 Hz), 7.37–7.44 (6H, m), 7.67–7.71 (4H, m); ^{13}C NMR ($CDCl_3$) δ = 19.1, 25.8, 26.8, 34.78, 60.0 (d, *J* = 7.43 Hz), 78.2 (d, *J* = 27.31 Hz), 107.5 (d, *J* = 11.7 Hz), 127.7, 129.6, 133.6, 133.7, 159.1 (d, *J* = 262.0 Hz); ^{19}F NMR ($CDCl_3$) δ = –144.51 (1F, dd, *J* = 18.81, 37.38 Hz); IR (neat) : 3442, 3071, 2957, 2958, 1702, 1671, 1472, 1428, 1264, 1186, 1070, 1014, 937, 822, 788, 740, 612 cm^{-1}; HRMS (FAB) calcd for $C_{24}H_{32}FO_2Si$ (M-H) 399.2156, found 399.2152.

Synthetic procedure for the *Z*-selective chromium(II)-mediated reductive coupling reaction of ethyl 4-bromo-4,4-difluorobutyrate (1D) with benzaldehyde

To a suspension of $CrCl_2$ (0.22 g, 1.8 mmol) and LiI (0.020 g, 0.15 mmol) in DMF (1.2 mL) were added benzaldehyde (0.064 g, 0.6 mmol) and ethyl 4-bromo-4,4-difluorobutylate (**1D**) (0.065 g, 0.3 mmol) at 0 °C. After stirring at room temperature for 4 h, the reaction was quenched with water. The whole was extracted with Et_2O three times. The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated in *vacuo*. The resulting residue was purified by silica gel column chromatography (Hexane/AcOEt = 3/1) to afford (*Z*)-ethyl 4-fluoro-5-hydroxy-5-phenyl-3-pentenoate (**2Da**) (0.040 g, 0.17 mmol, 55% yield).

(*Z*)-Ethyl 4-fluoro-5-hydroxy-5-phenyl-3-pentenoate (2Da)



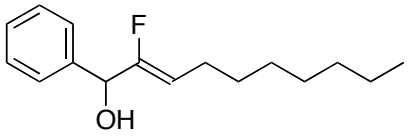
Yield : 55%; ^{1}H NMR ($CDCl_3$) δ = 1.26 (3H, t, *J* = 7.09 Hz), 2.50 (1H, br s), 3.17 (2H, d, *J* = 7.19 Hz), 4.15 (2H, q, *J* = 7.09 Hz), 5.22 (1H, dt, *J* = 35.26, 7.19 Hz), 5.24 (1H, d, *J* = 11.25 Hz), 7.29–7.45 (5H, m); ^{13}C NMR ($CDCl_3$) δ = 14.1, 29.2, 60.9, 72.4 (d, *J* = 31.4 Hz), 99.5 (d, *J* = 9.8 Hz), 126.7, 128.4, 128.5, 139.1, 160.4 (d, *J* = 260.3 Hz), 171.2; ^{19}F NMR ($CDCl_3$) δ = –119.34 (1F, dd, *J* = 11.25, 35.26 Hz); IR (neat) : 3448, 3032, 2983, 1736, 1495, 1454, 1372, 1266, 1189, 1094,

1060, 1027, 955, 887, 804, 701 cm^{-1}; HRMS (FAB) calcd for $C_{13}H_{15}FO_3$ (M^+) 238.1005, found 238.1009.

Typical procedure for the *Z*-selective chromium(II)-mediated reductive coupling of 1-bromo-1,1-difluorononane (1E) with benzaldehyde

To a suspension of $CrCl_2$ (0.22 g, 1.8 mmol) and LiI (0.020 g, 0.15 mmol) in DMF (1.2 mL) were added benzaldehyde (0.064 g, 0.6 mmol) and 1-bromo-1,1-difluorononane (**1E**) (0.073 g, 0.3 mmol) at 0 °C. After stirring at room temperature for 4 h, the reaction was quenched with water. The whole was extracted with Et_2O three times. The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated in *vacuo*. The resulting residue was purified by silica gel column chromatography (Hexane/AcOEt = 3/1) to afford the pure product **2Ea** (0.056 g, 0.22 mmol, 76% yield).

(*Z*)-2-Fluoro-1-phenyl-2-decen-1-ol (2Ea)



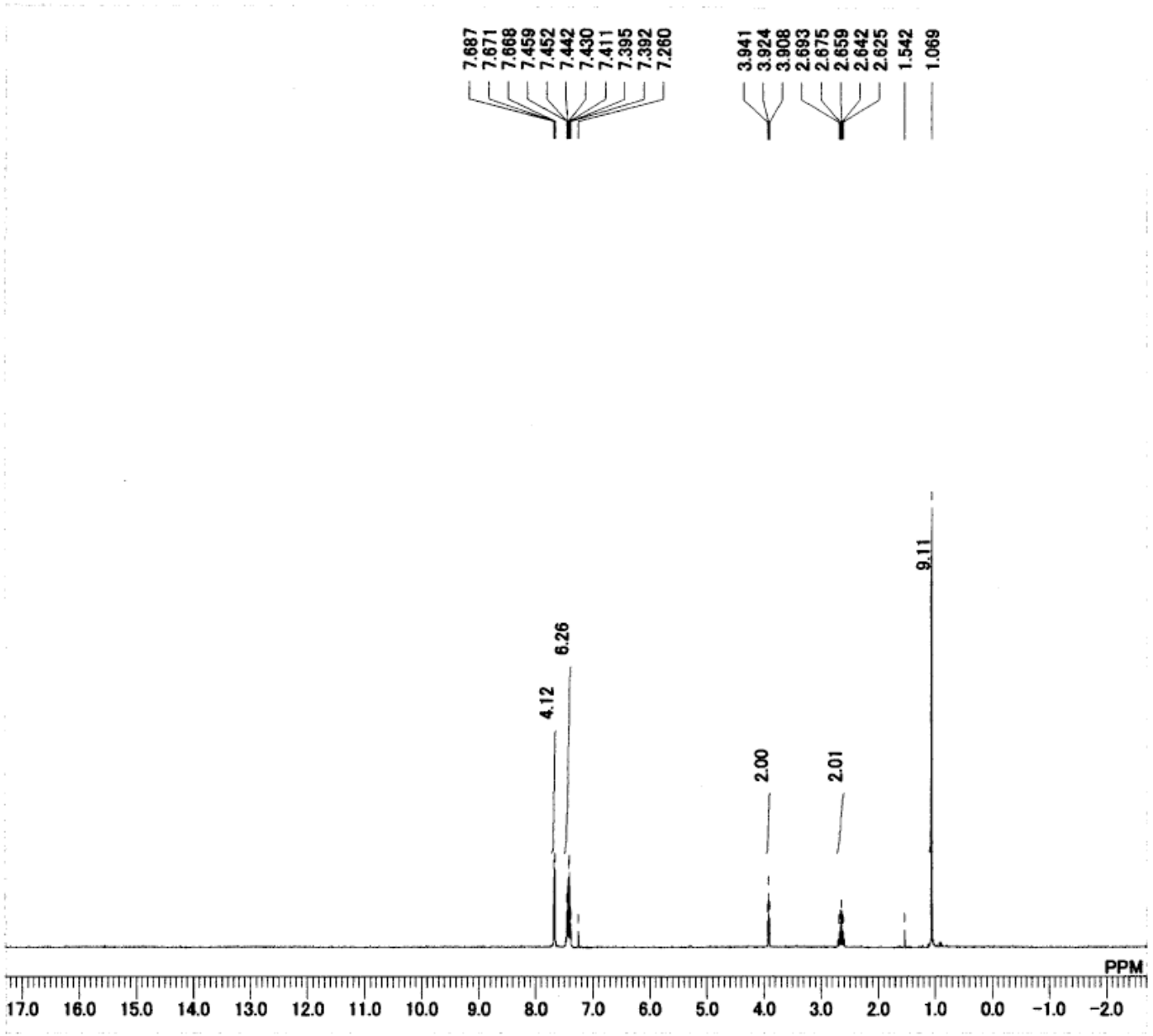
^{1}H NMR ($CDCl_3$) δ = 0.88 (3H, t, *J* = 6.79 Hz), 1.27–1.39 (10H, m), 2.08–2.18 (3H, m), 4.90 (1H, dt, *J* = 37.87, 7.19 Hz), 5.21 (1H, d, *J* = 13.17 Hz), 7.31–7.45 (5H, m); ^{13}C NMR ($CDCl_3$) δ = 14.1, 22.6, 23.3, 29.0, 29.1, 29.2, 31.8, 72.8 (t, *J* = 32.2 Hz), 107.8 (d, *J* = 13.3 Hz), 126.6, 128.2, 128.5, 139.7, 158.3 (t, *J* = 255.4 Hz); ^{19}F NMR ($CDCl_3$) δ = –124.22 (1F, dd, *J* = 13.17, 37.87 Hz); IR (neat) : 3364, 3032, 2926, 2856, 1708, 1495, 1455, 1379, 1273, 1192, 1111, 1024, 700 cm^{-1}; HRMS (FAB) calcd for $C_{16}H_{23}FO$ (M^+) 250.1733, found 250.1729.

Reference

1) Shimada, T.; Konno, T.; Ishihara, T. *Chem. Lett*. **2007**, *36*, 636-637.

2) Peng, S.; Qing, F.; Li, Y.; Hu, C. *J. Org. Chem*. **2000**, *65*, 694-700.

3) Chen, J.; Hu, C.-M. *J. Chem. Soc., Perkin Trans. 1* **1994**, 1111-1114.



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OBSET	135.40 KHz
OBFIN	24.90 Hz
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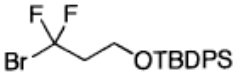
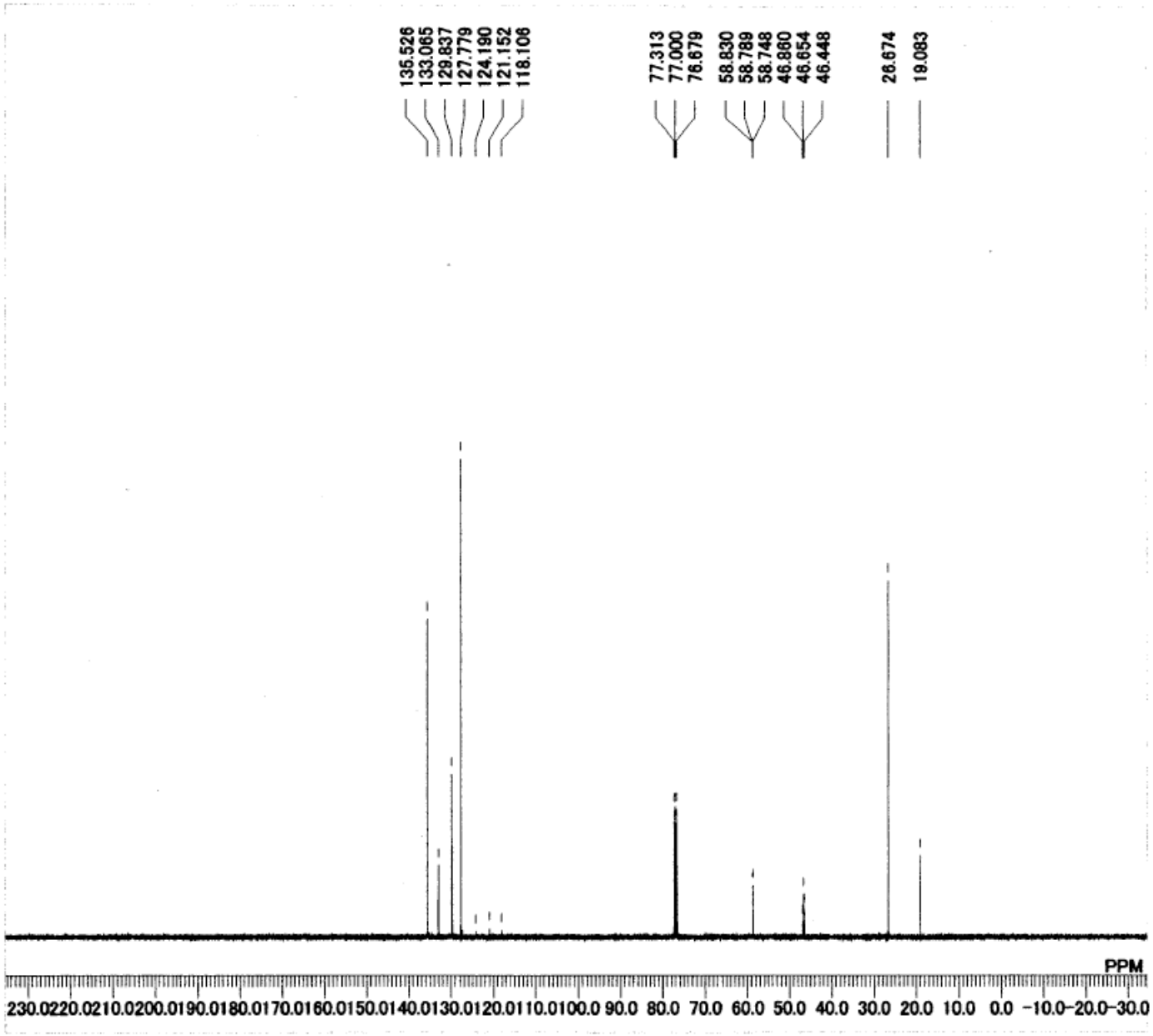
DEADT	72.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	13
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	原料 H 拡大.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	22
LKPHS	250
LKSIG	804
CSPED	12 Hz
FILDC	
FILDF	





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PREDL	0.20000 msec
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POINT	32768
SPO	32768
TIMES	256
DUMMY	1
FREQU	27118.64 Hz
FLT	13550 Hz
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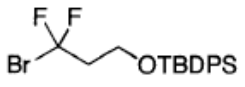
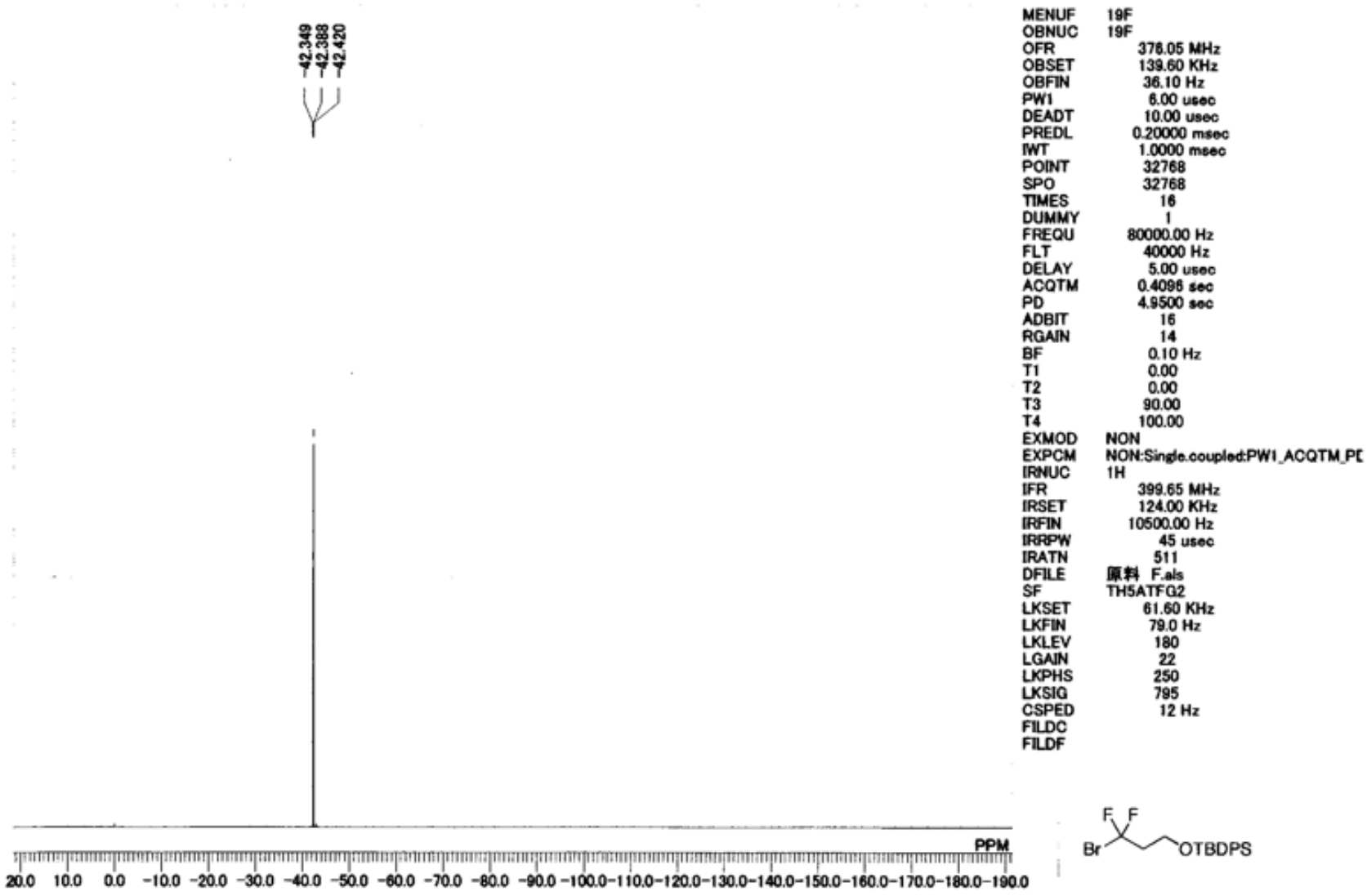
PD	1.7920 sec
ADBIT	16
RGAIN	25
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRF
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	原料 C 拡大.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	22
LKPHS	250
LKSIG	803
CSPED	12 Hz
FILDC	
FILDF	





MENUF	19F
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OBSET	139.60 KHz
OBFIN	36.10 Hz
PW1	6.00 usec
DEADT	10.00 usec
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IWT	1.0000 msec
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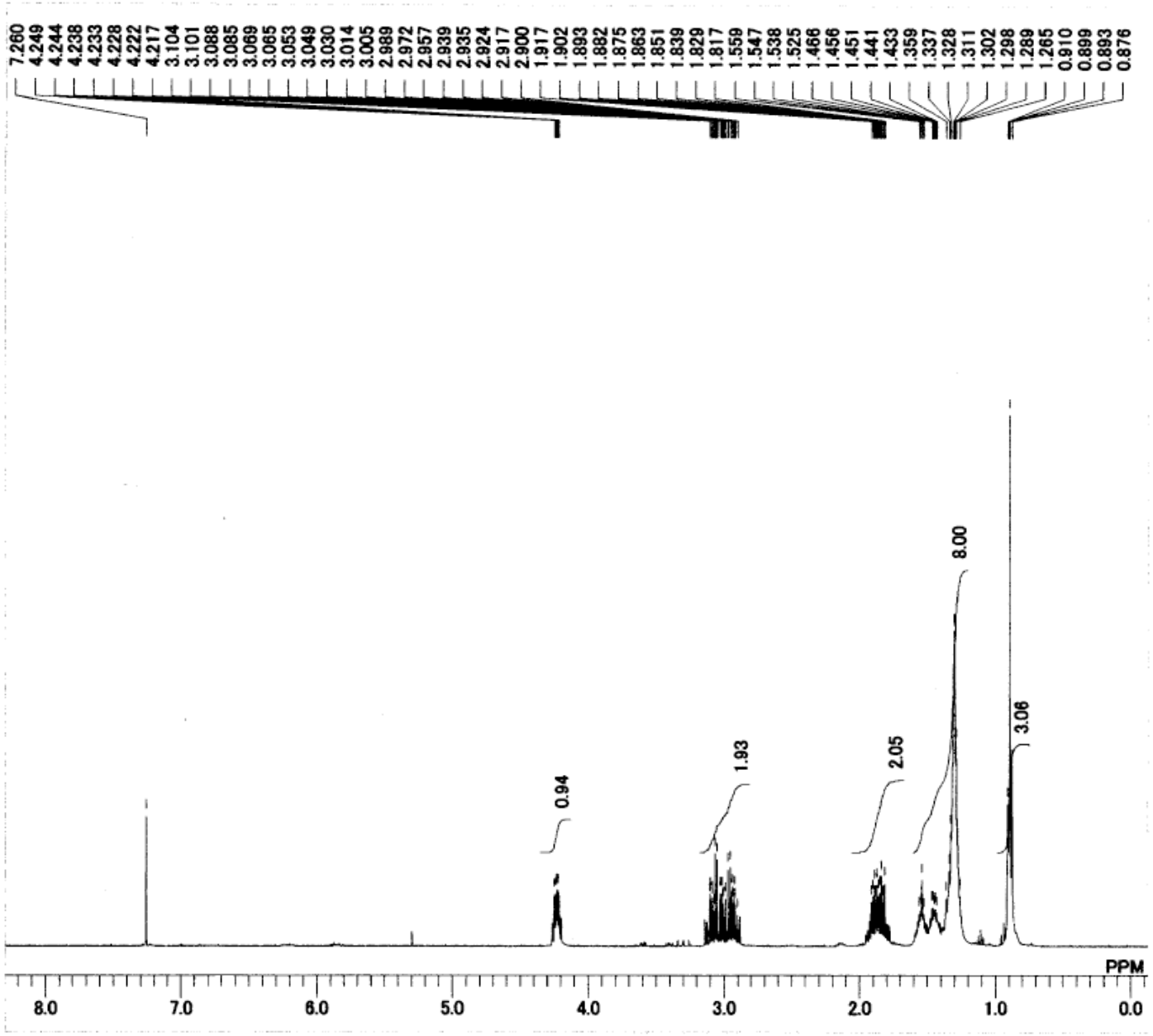
SPO	32768
TIMES	16
DUMMY	1
FREQU	80000.00 Hz
FLT	40000 Hz
DELAY	5.00 usec
ACQTM	0.4096 sec
PD	4.9500 sec
ADBIT	16
RGAIN	14
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	原料 F.als
SF	TH5ATFQ2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	22
LKPHS	250
LKSIG	795
CSPED	12 Hz
FILDC	
FILDF	



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OBSET	135.40 KHz
OBFIN	24.90 Hz
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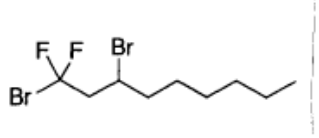
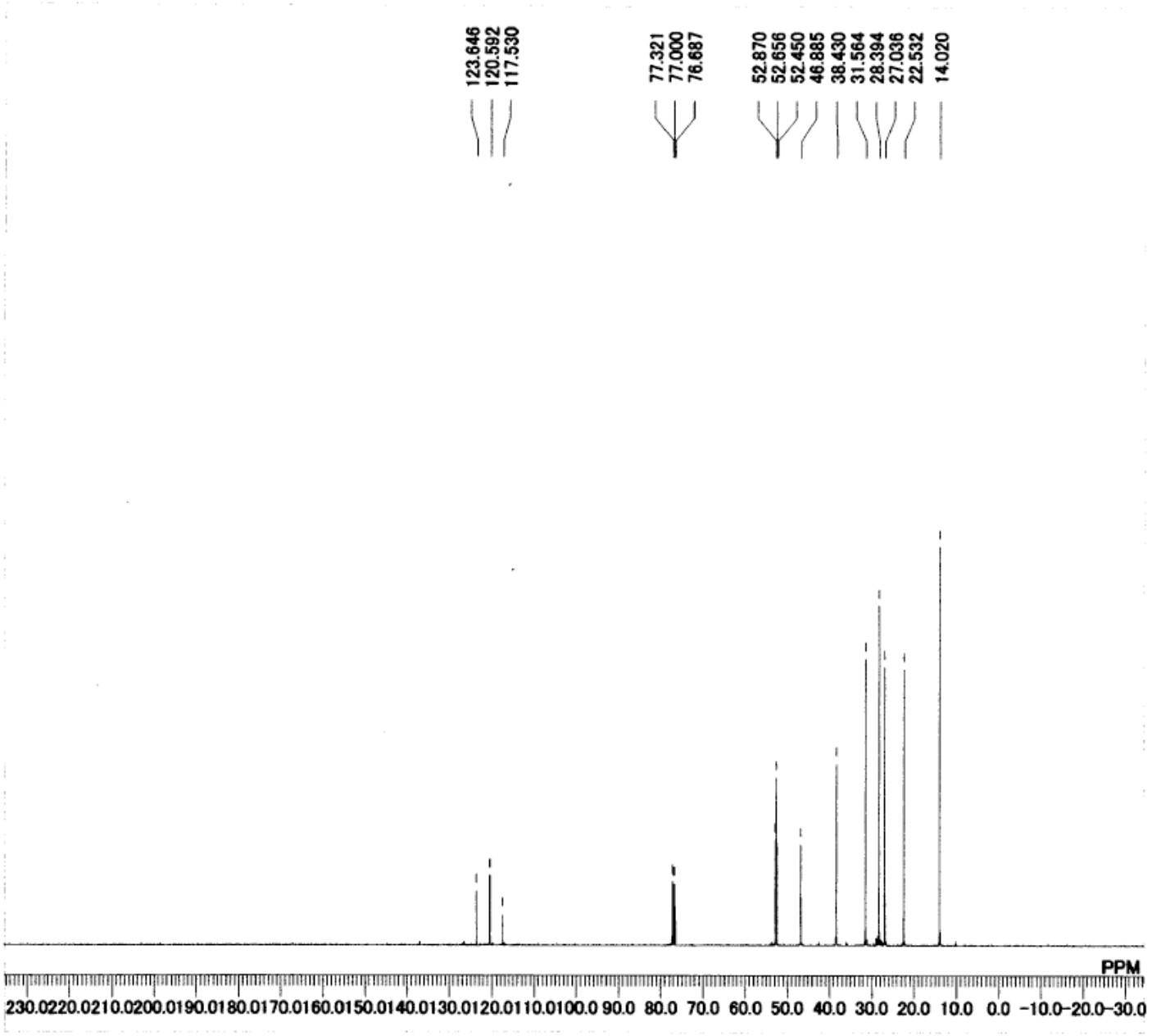
DEADT	72.20 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	14
BF	0.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	Rxn.459 nonane 1段階目　蒸留 H.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	23
LKPHS	240
LKSIG	1083
CSPED	12 Hz
FILDC	
FILDF	





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OBSET	125.00 KHz
OBFIN	10500.00 Hz
PW1	6.20 usec
DEADT	19.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	20000
DUMMY	1
FREQU	27118.64 Hz
FLT	13550 Hz
DELAY	14.80 usec
ACQTM	1.2083 sec
PD	1.7920 sec
ADBIT	16
RGAIN	24
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRF
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	_DEFAULT.ALS
SF	TH5ATFG2
LKSET	61.60 KHz
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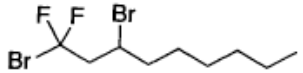
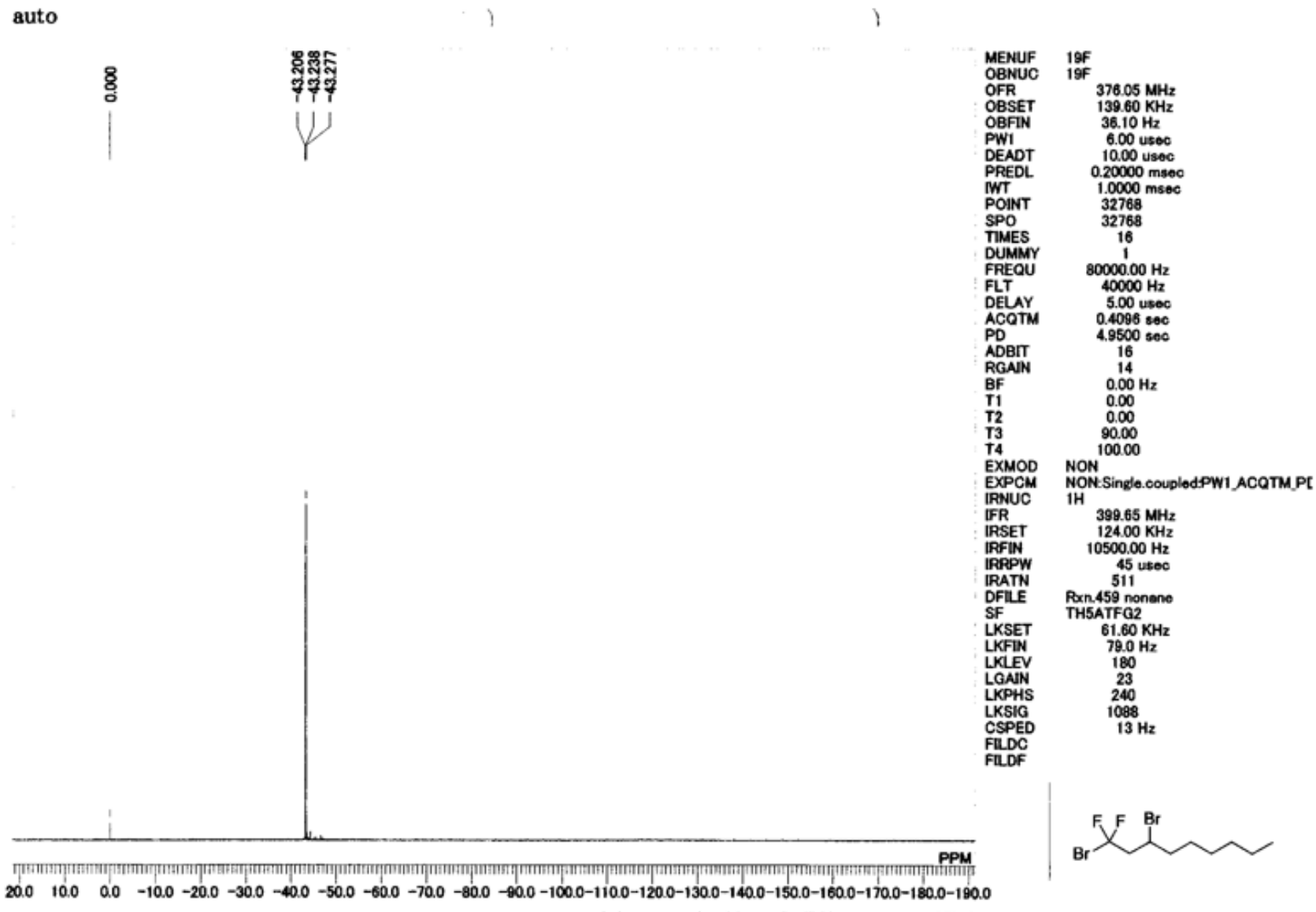
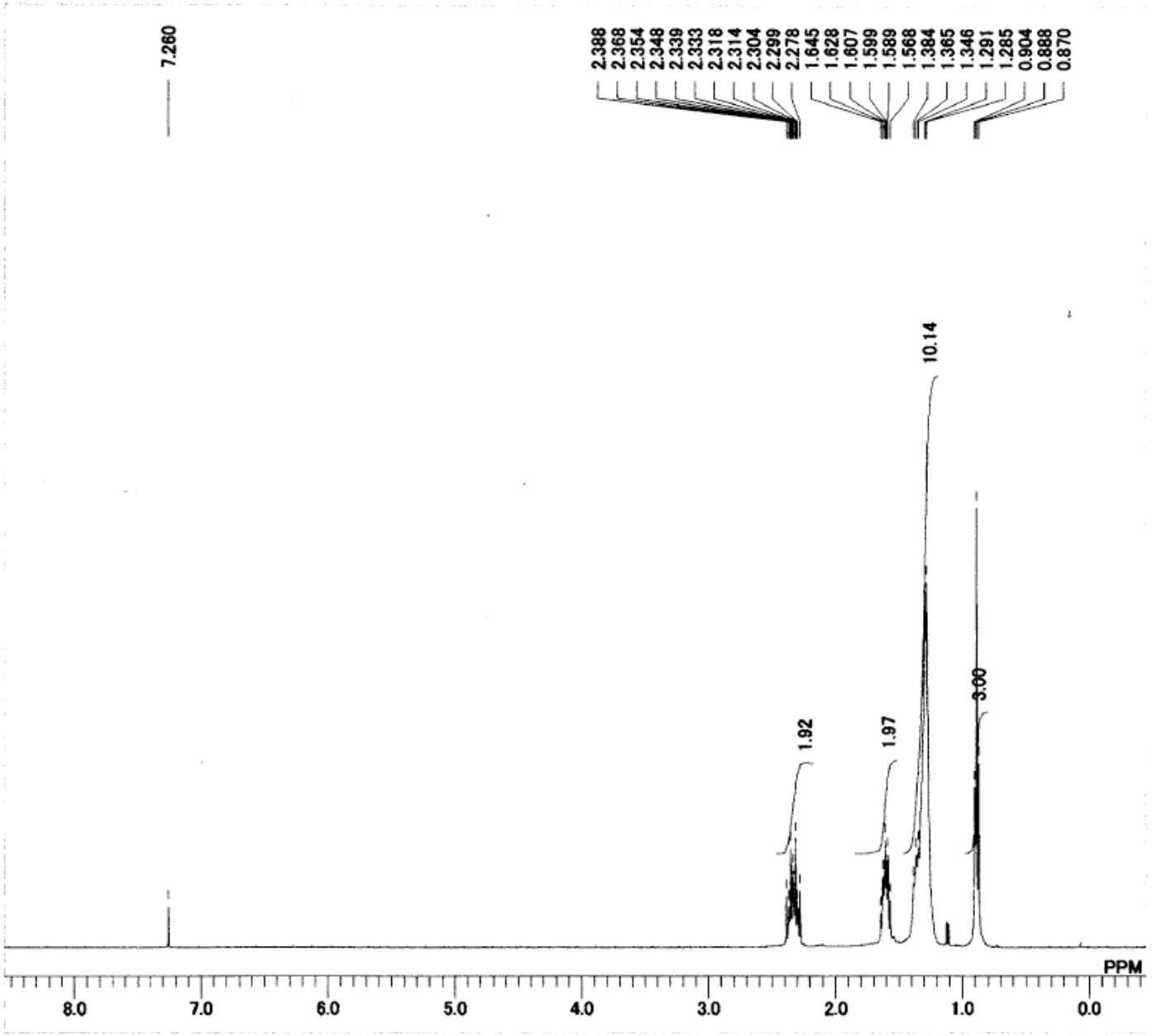
FILDF	







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OBSET	135.40 KHz
OBFIN	24.90 Hz
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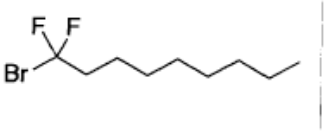
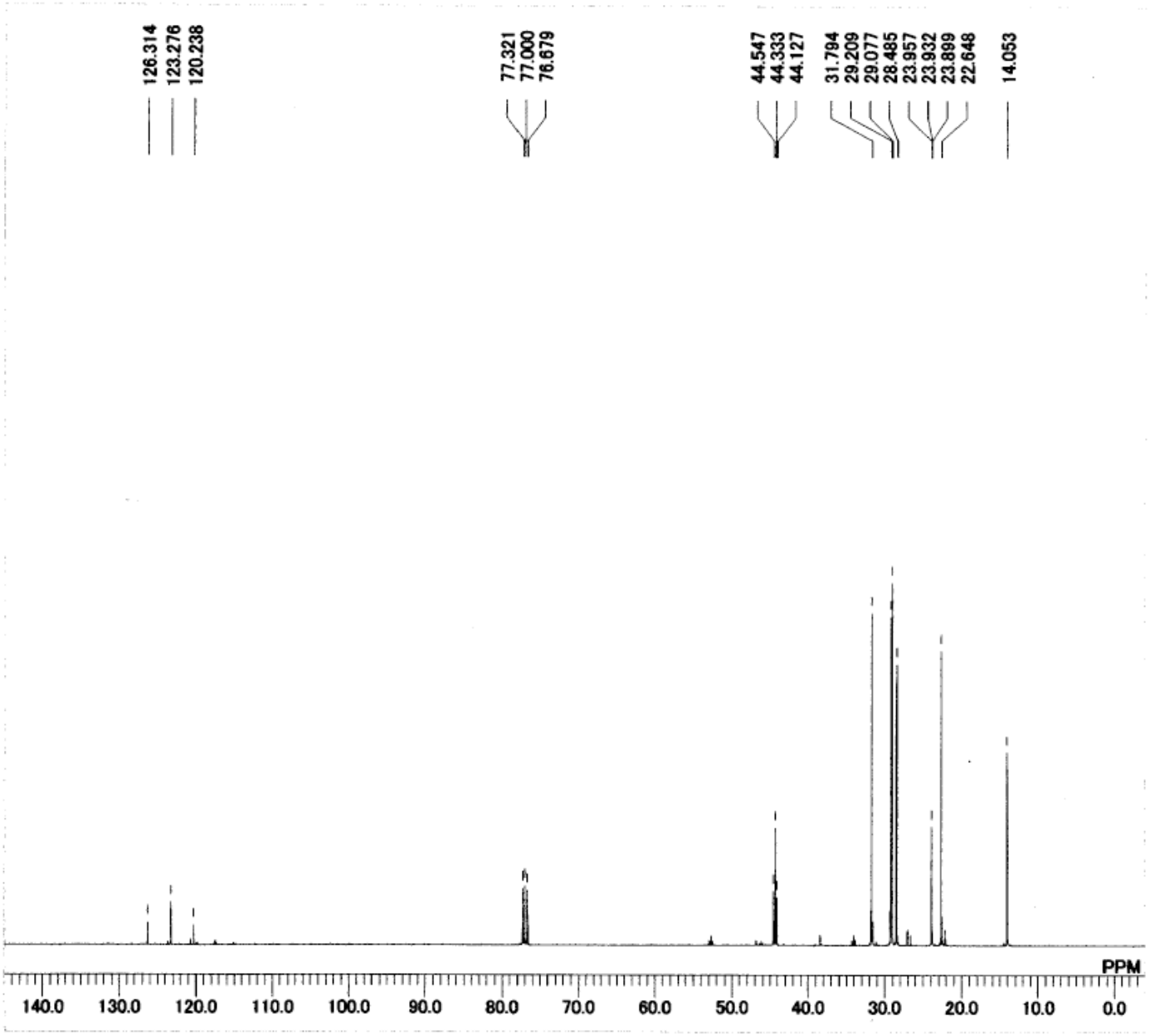
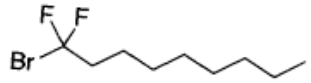
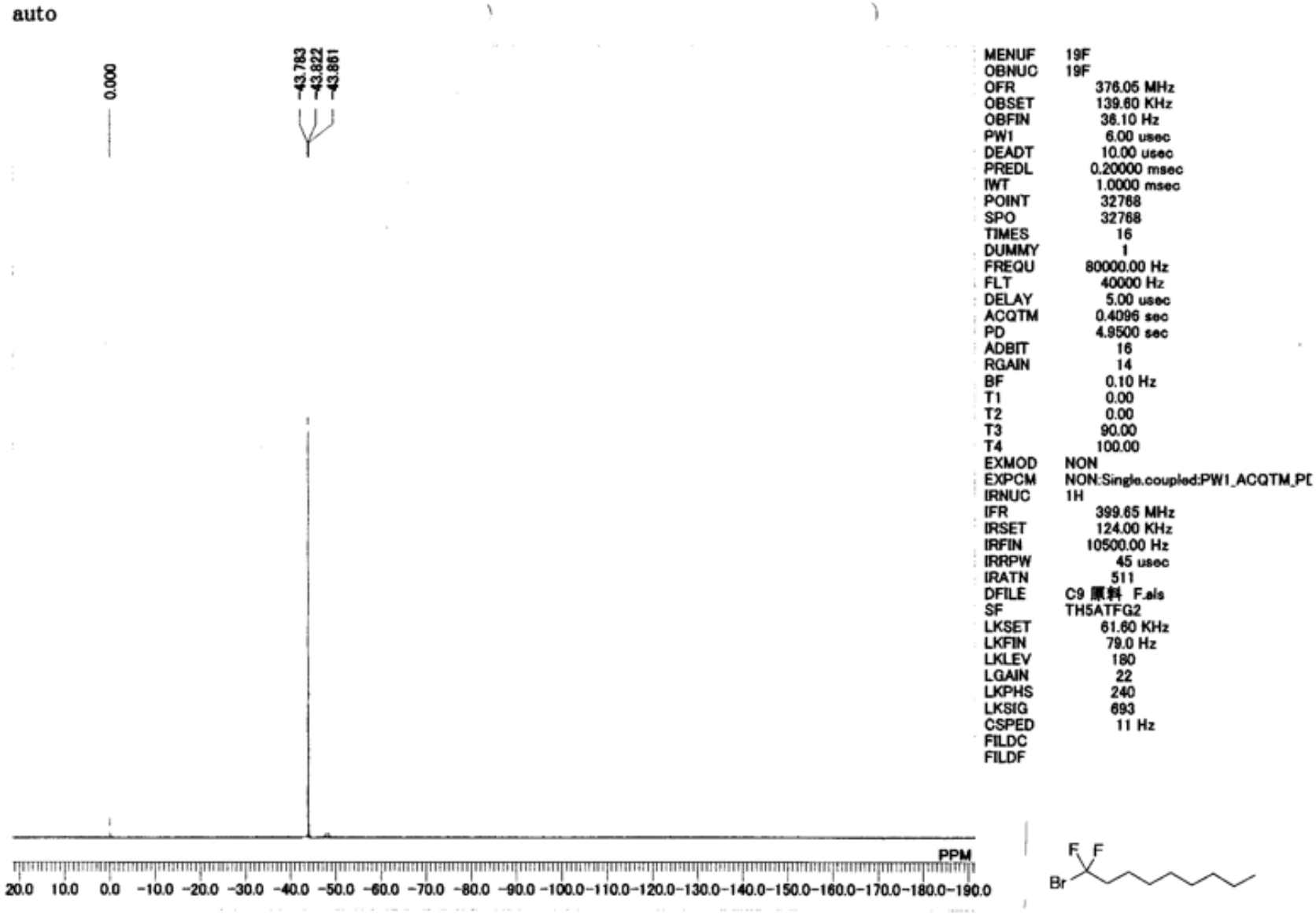
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	11
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	C9 原料 H.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	22
LKPHS	240
LKSIG	687
CSPED	10 Hz
FILDC	
FILDF	

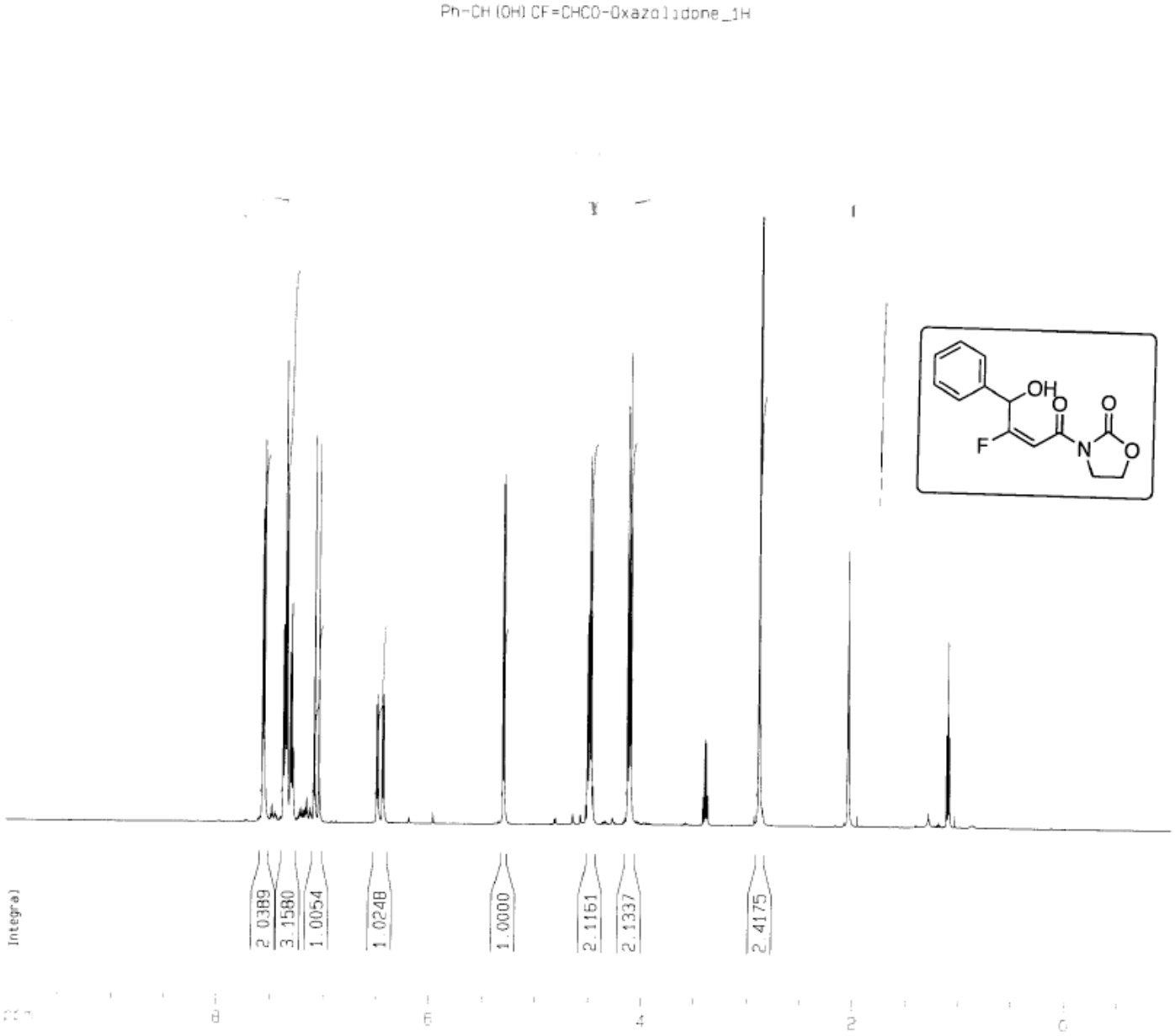




MENUF	13C
OBNUC	13C
OFR	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
PW1	6.20 usec
DEADT	19.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	20000
DUMMY	1
FREQU	27118.64 Hz
FLT	13550 Hz
DELAY	14.80 usec
ACQTM	1.2083 sec
PD	1.7920 sec
ADBIT	16
RGAIN	24
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRF
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	C9 原料 C over night.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	24
LKPHS	240
LKSIG	1190
CSPED	12 Hz
FILDC	
FILDF	

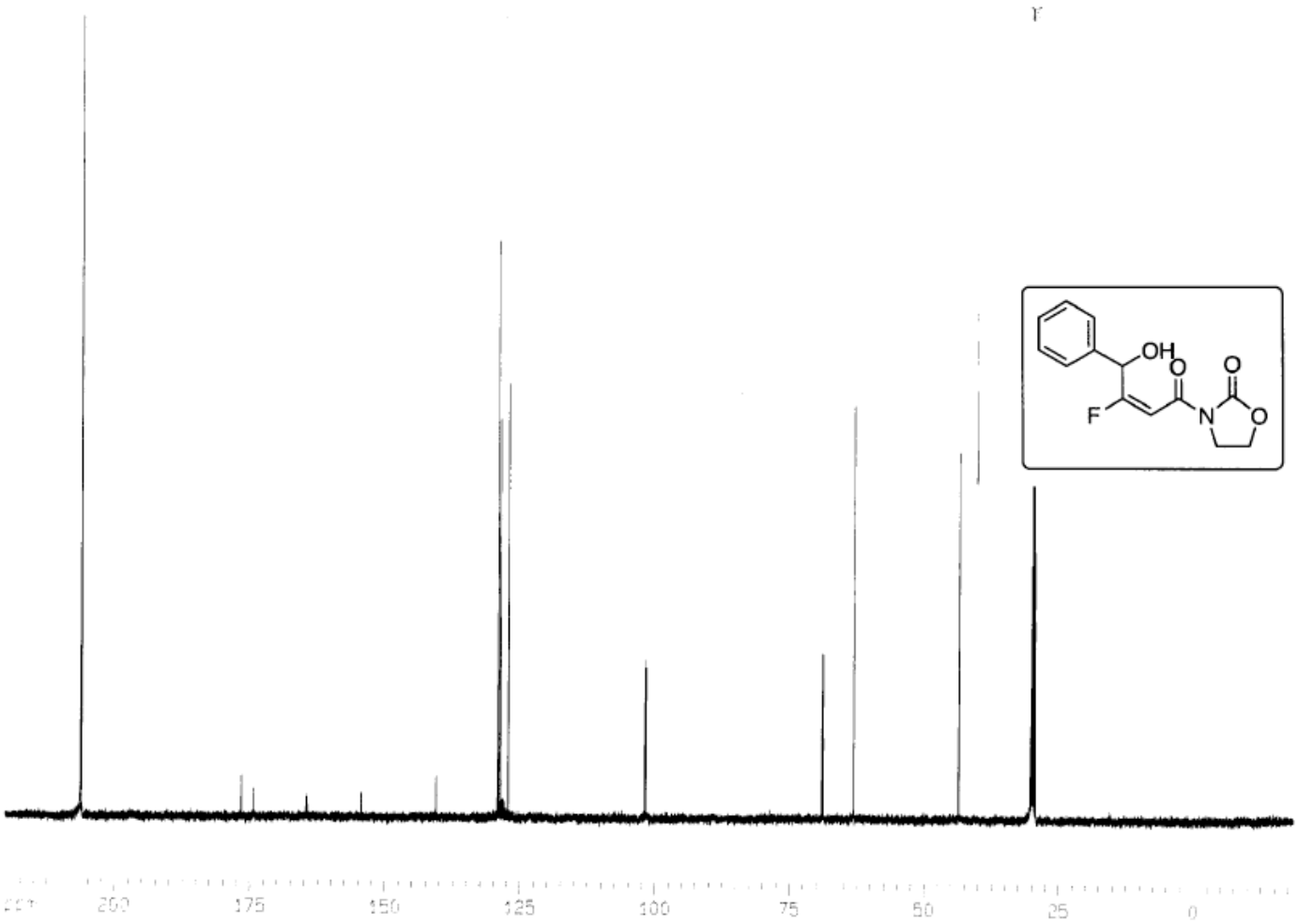




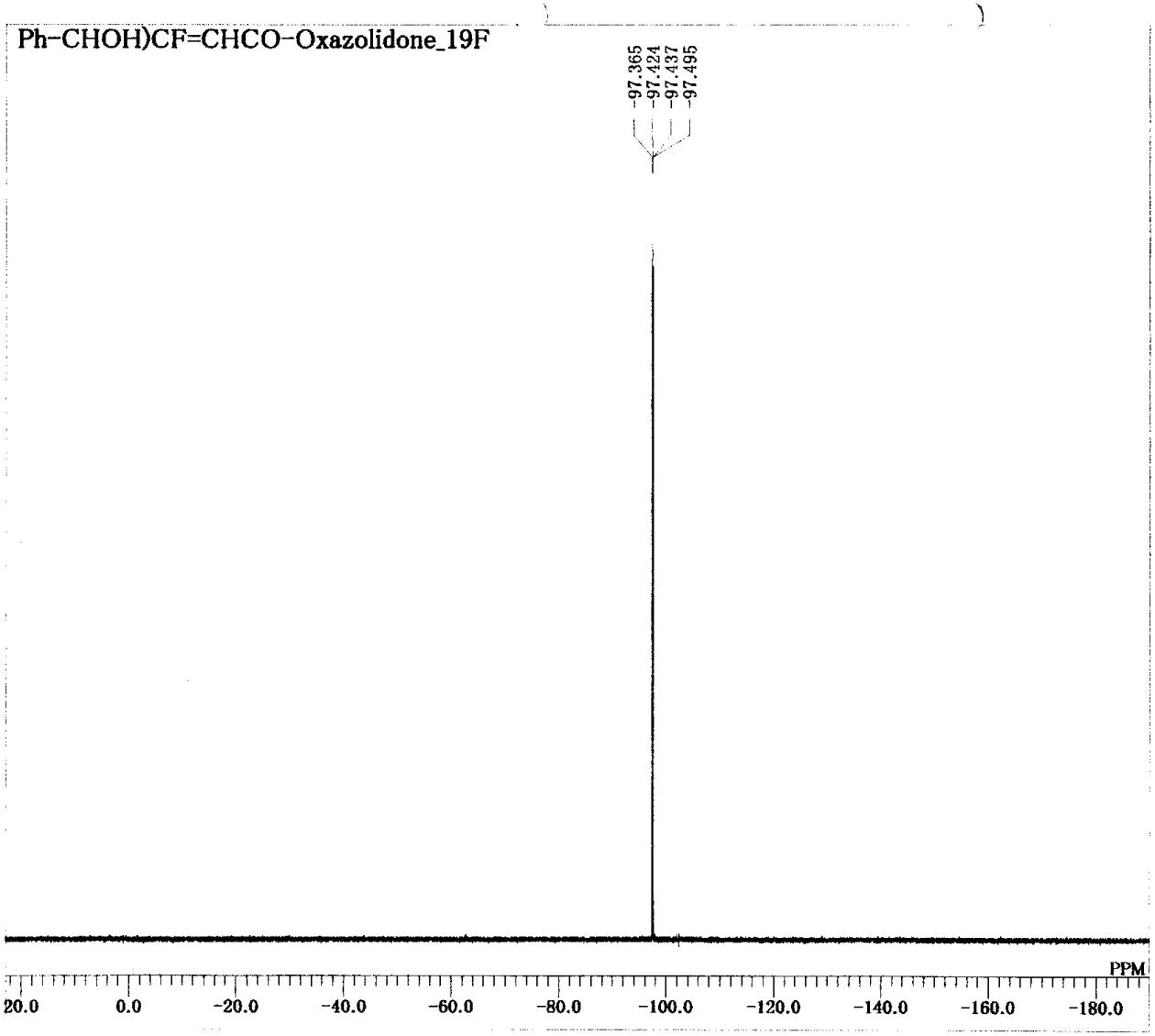



```
Current Data Parameters
NAME                  shima-061016
EXPNO                            3
PROCNO                           1

F2 - Acquisition Parameters
Date_                     20061016
Time                         15.23
INSTRUM                    drx500
PROBHD     5 mm Multinucl
PULPROG                      zg30
TD                          65536
SOLVENT                   Acetone
NS                               34
DS                               2
SWH                   10330.578 Hz
FIDRES                0.157632 Hz
AQ                    3.1719923 sec
RG                           203.2
DW                       48.400 usec
DE                        6.00 usec
TE                          296.8 K
D1                    1.00000000 sec
MCREST                0.00000000 sec
MCWRK                 0.01500000 sec

======== CHANNEL f1 ========
NUC1                          1H
P1                       10.00 usec
PL1                      -6.00 dB
SFO1                500.1330885 MHz

F2 - Processing parameters
SI                           32768
SF                  500.1300099 MHz
WDW                            EM
SSB                             0
LB                        0.30 Hz
GB                              0
PC                        0.00

1D NMR plot parameters
CX                       20.00 cm
CY                       29.41 cm
F1P                     10.000 ppm
F1                     5001.30 Hz
F2P                     -1.000 ppm
F2                     -500.13 Hz
PPMCM                 0.55000 ppm/cm
HZCM                275.07150 Hz/cm
```

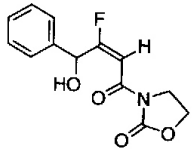
Ph-CH(OH)CF=CHCO-Oxazolidone_13C

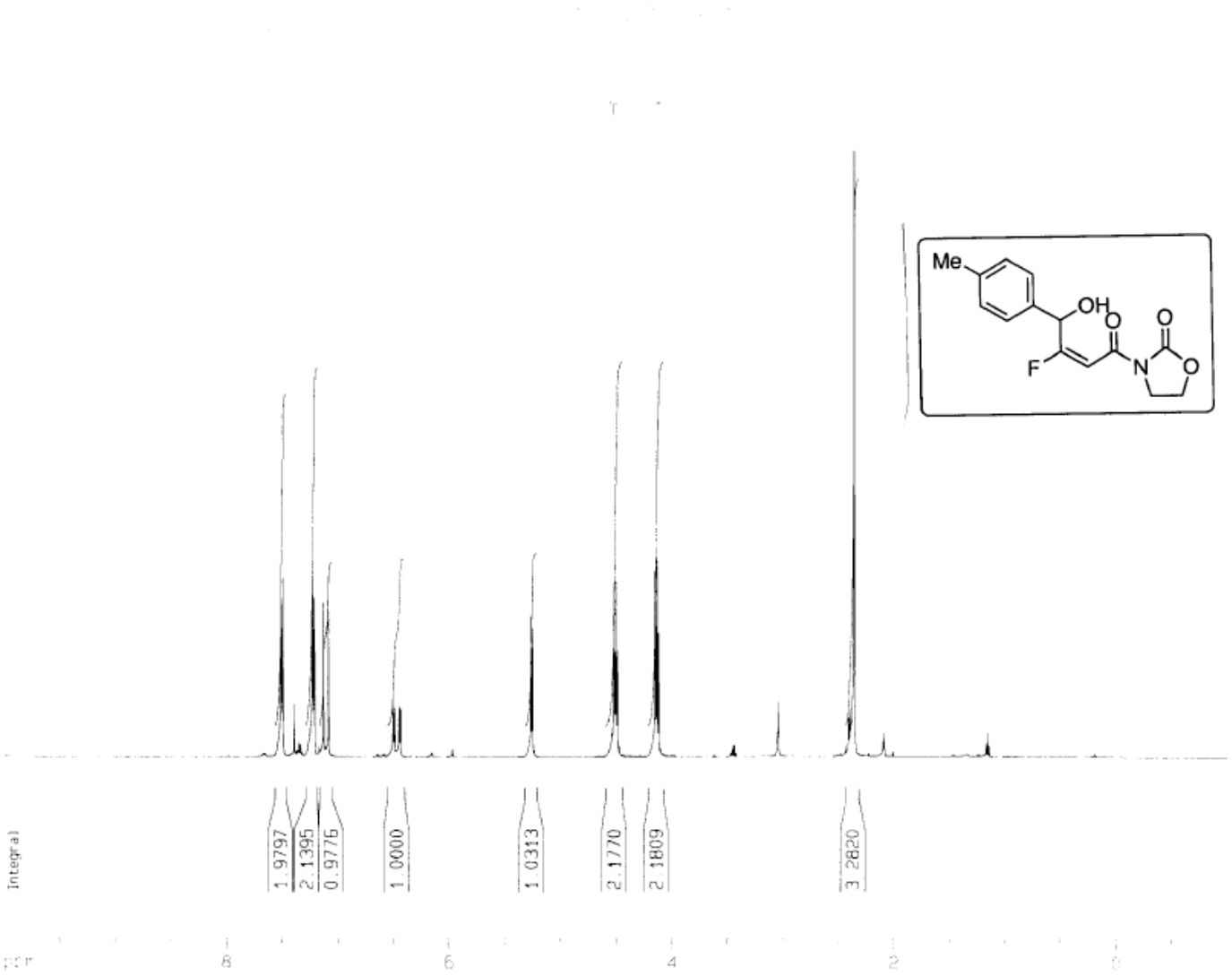



```
Current Data Parameters
NAME        shima-061016
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20061016
Time               15.29
INSTRUM           drx500
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  274
DS                    2
SWH           30030.029 Hz
FIDRES        0.458222 Hz
AQ            1.0912244 sec
RG                 4096
DW               16.650 usec
DE                6.00 usec
TE               297.1 K
D1            0.50000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                11.00 usec
PL1               -3.00 dB
SFO1        125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            100.00 usec
PL2               -6.00 dB
PL12              17.00 dB
PL13              19.00 dB
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7576791 MHz
WDW                   EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 0.00

1D NMR plot parameters
CX                20.00 cm
CY                14.56 cm
F1P              220.266 ppm
F1               27700.20 Hz
F2P              -18.526 ppm
F2               -2329.83 Hz
PPMCM            11.93964 ppm/cm
HZCM          1501.50159 Hz/cm
```

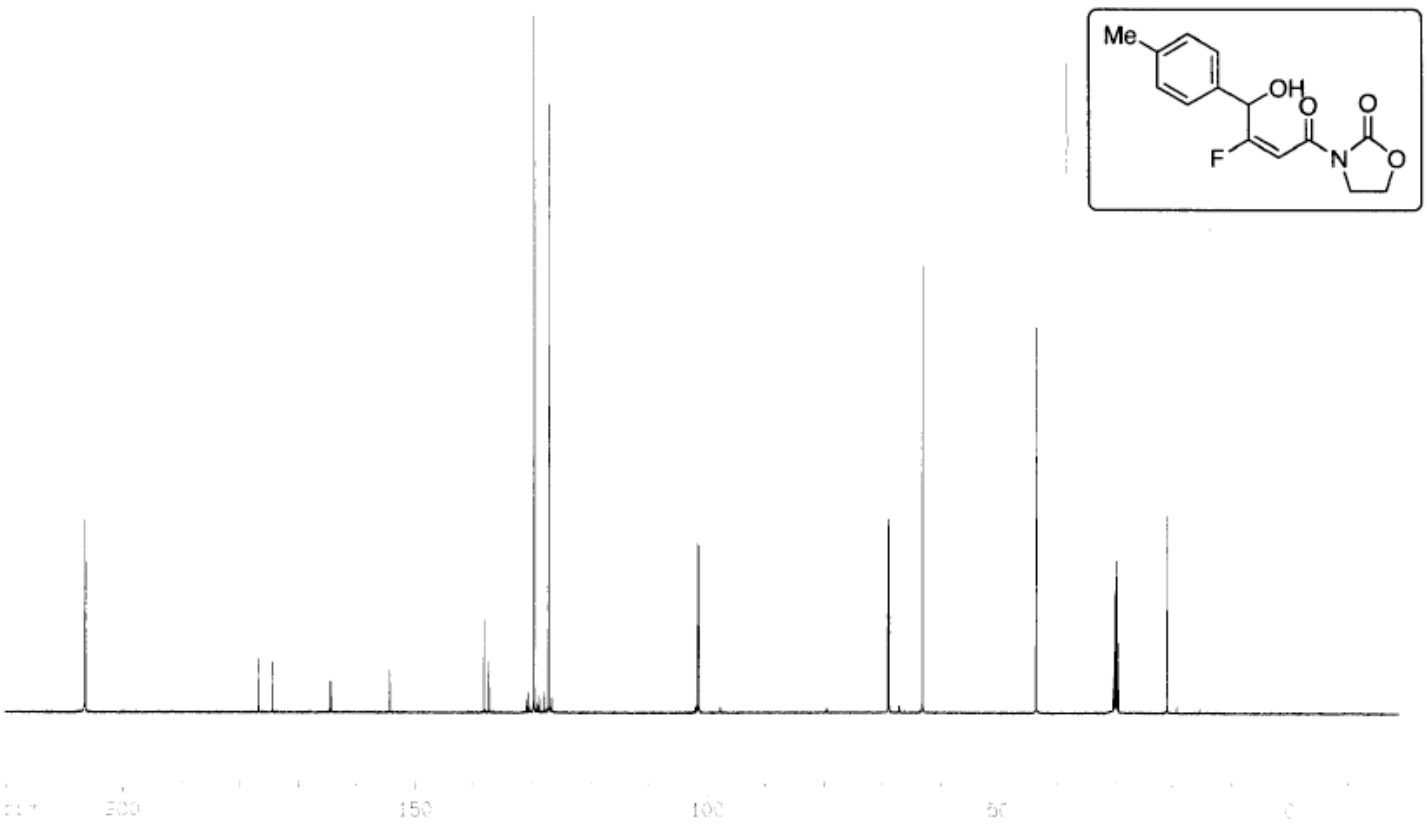


DFILE	_DEFAULT.ALS
COMNT	Ph-CHOH)CF=CHCO-Ox
DATIM	Mon Oct 16 15:31:38 200
OBNUC	19F
EXMOD	NON
OBFRQ	376.05 MHz
OBSET	139.60 KHz
OBFIN	36.10 Hz
POINT	32768
FREQU	80000.00 Hz
SCANS	32
ACQTM	0.4096 sec
PD	4.9500 sec
PW1	5.50 usec
IRNUC	1H
CTEMP	23.0 c
SLVNT	ACETN
EXREF	0.00 ppm
BF	0.10 Hz
RGAIN	17

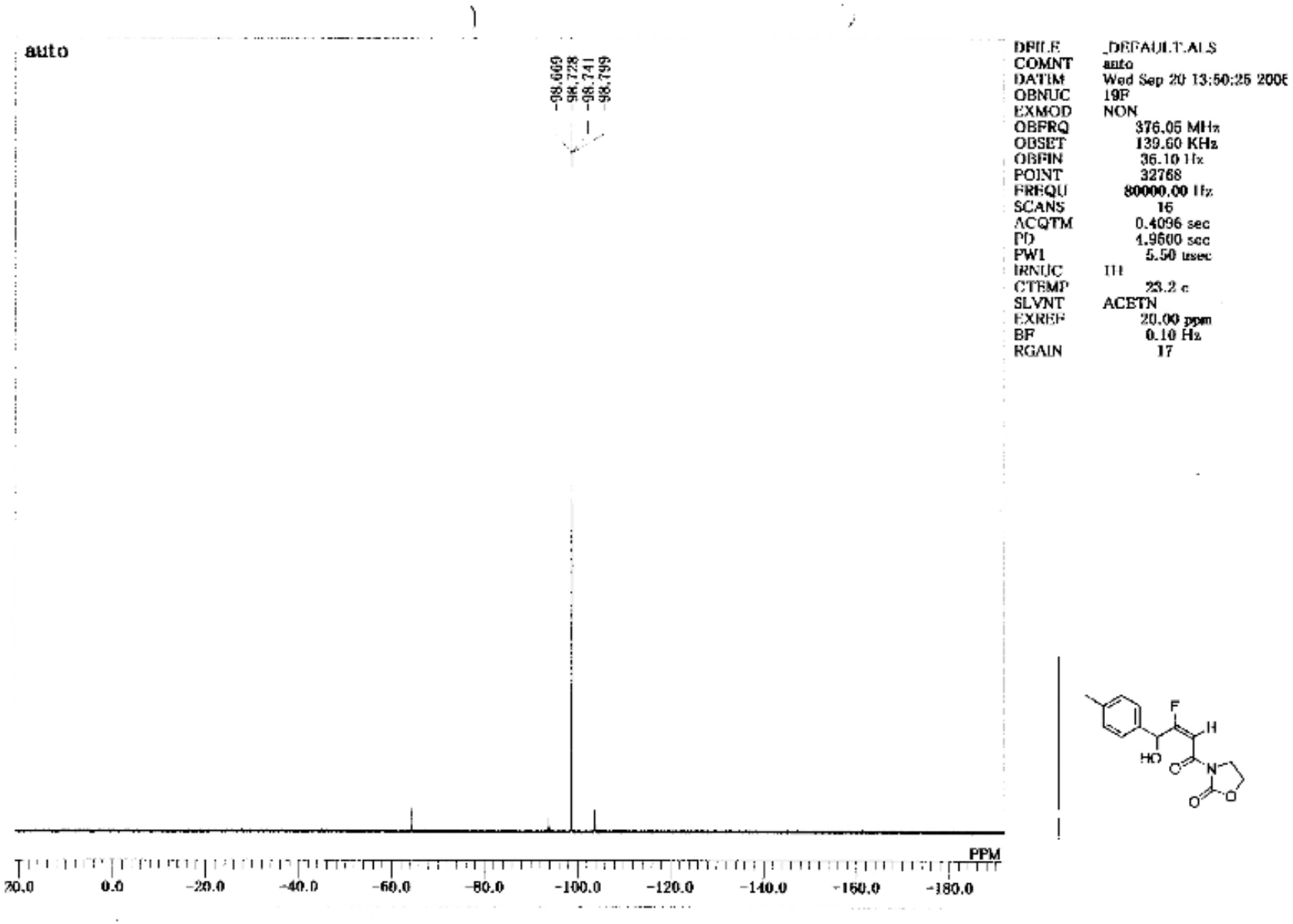




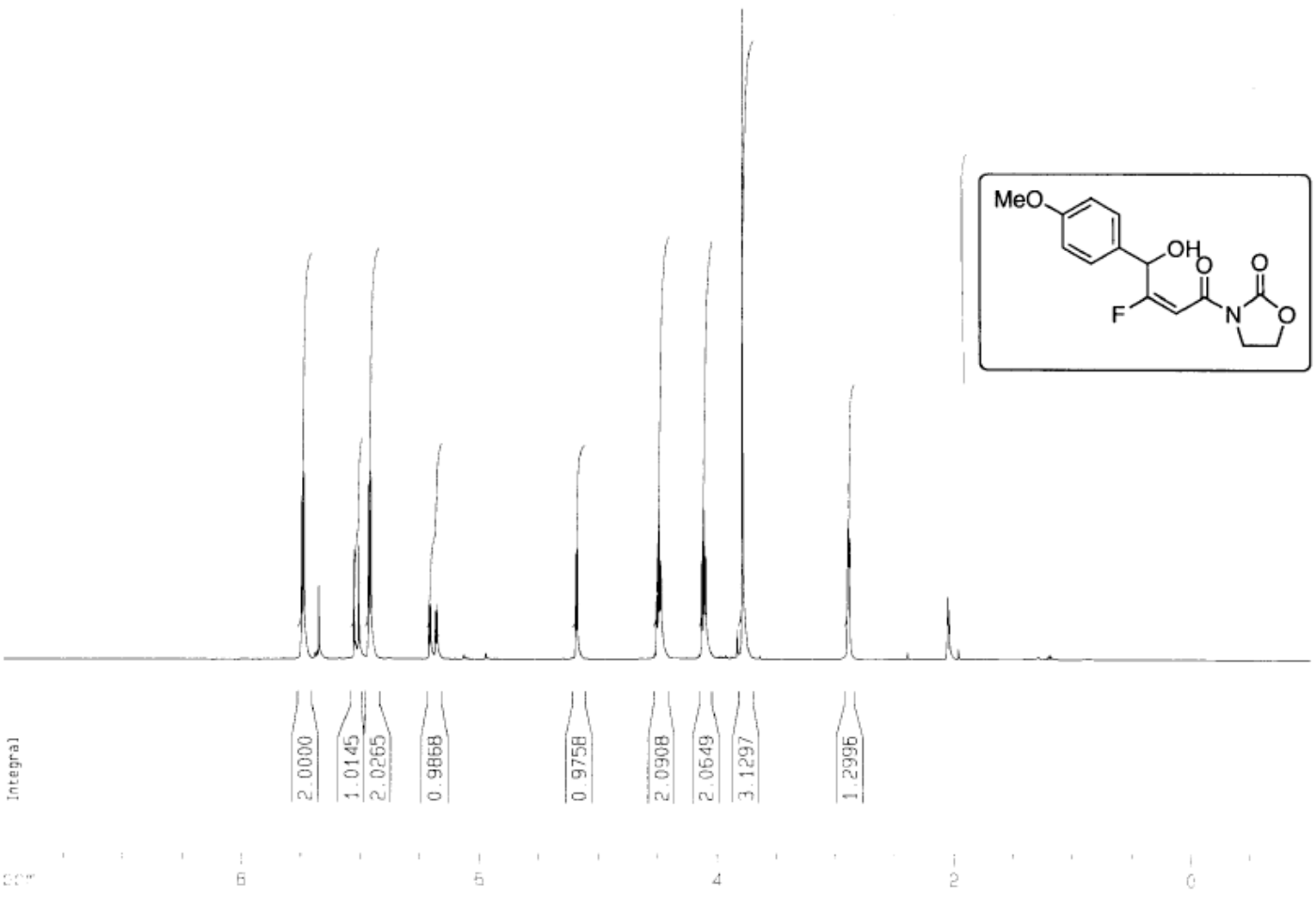

```
Current Data Parameters
NAME        shima-060920
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060920
Time               12.32
INSTRUM          drx500
PROBHD   5 mm Multinucl
PULPROG            zg30
TD                65536
SOLVENT         Acetone
NS                    75
DS                     2
SWH          10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1719923 sec
RG                    57
DW              48.400 usec
DE               6.00 usec
TE              299.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                10.00 usec
PL1               -6.00 dB
SFO1        500.1330885 MHz

F2 - Processing parameters
SI                 32768
SF          500.1299899 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  0.00

1D NMR plot parameters
CX                 20.00 cm
CY                  9.99 cm
F1P               10.000 ppm
F1              5001.30 Hz
F2P               -1.000 ppm
F2              -500.13 Hz
PPMCM              0.55000 ppm/cm
HZCM             275.07150 Hz/cm
```




```
Current Data Parameters
NAME        shima-060920
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060920
Time               12.43
INSTRUM          drx500
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  556
DS                    2
SWH           30030.029 Hz
FIDRES        0.458222 Hz
AQ            1.0912244 sec
RG               2896.3
DW              16.650 usec
DE               6.00 usec
TE              299.6 K
D1           0.50000000 sec
D11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1               11.00 usec
PL1              -3.00 dB
SFO1        125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2           100.00 usec
PL2              -6.00 dB
PL12             17.00 dB
PL13             19.00 dB
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7575835 MHz
WDW                EM
SSB                0
LB               1.00 Hz
GB                 0
PC              0.00

1D NMR plot parameters
CX              20.00 cm
CY              10.06 cm
F1P            220.231 ppm
F1           27695.73 Hz
F2P            -18.562 ppm
F2           -2334.30 Hz
PPMCM          11.93964 ppm/cm
HZCM         1501.50159 Hz/cm
```

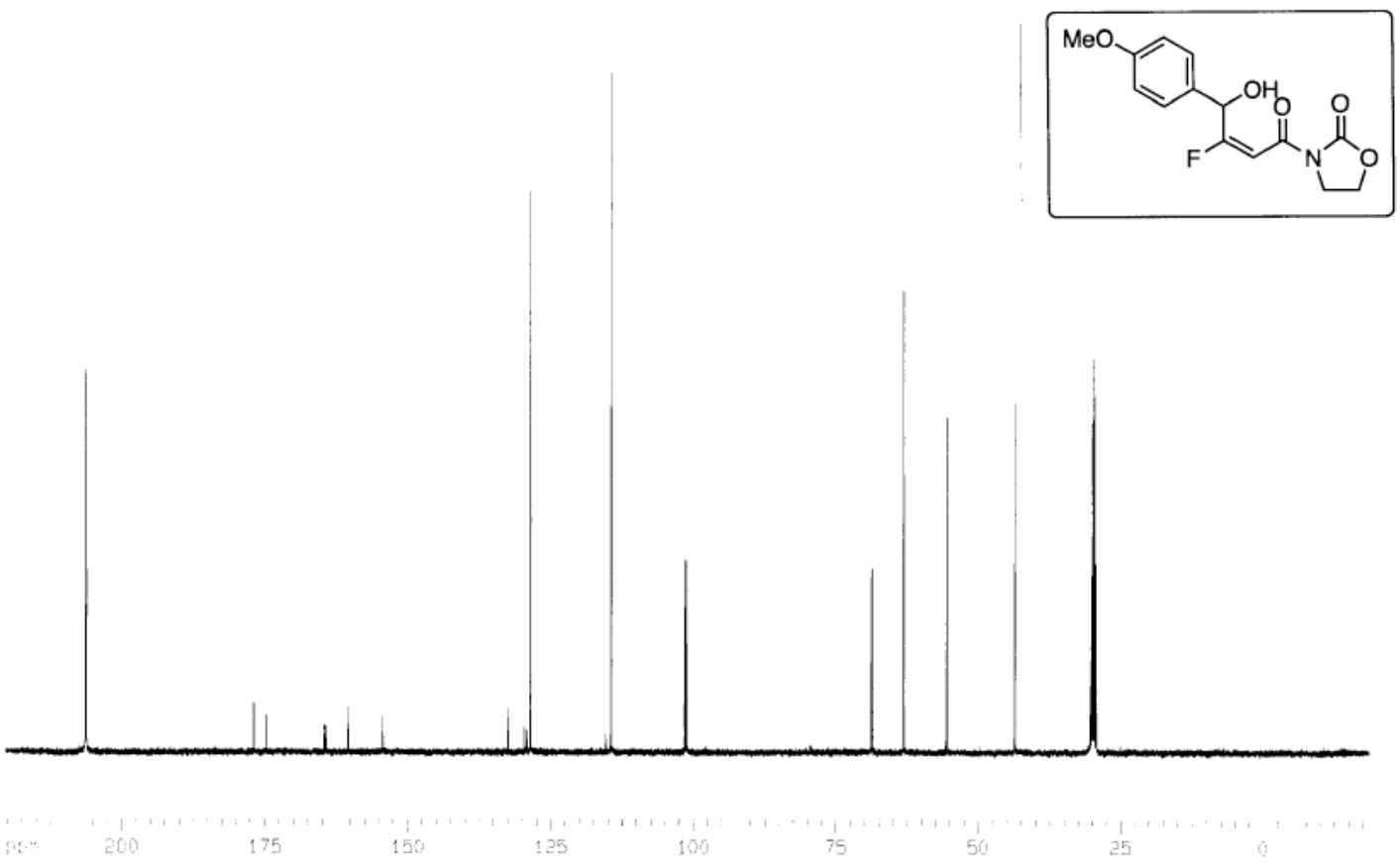

auto
-98.669
-98.728
-98.741
-98.799
DFILE _DEFAULT.ALS
COMNT auto
DATIM Wed Sep 20 13:50:25 2006
OBNUC 19F
EXMOD NON
OBFRQ 376.05 MHz
OBSET 139.60 KHz
OBFIN 36.10 Hz
POINT 32768
FREQU 80000.00 Hz
SCANS 16
ACQTM 0.4096 sec
PD 1.9600 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 23.2 c
SLVNT ACETN
EXREF 20.00 ppm
BF 0.10 Hz
RGAIN 17
F
H
HO
O
N
O
O
PPM
20.0
0.0
-20.0
-40.0
-60.0
-80.0
-100.0
-120.0
-140.0
-160.0
-180.0

p-Anis-CH(OH)CF=CHCO-Oxazilidone_1H

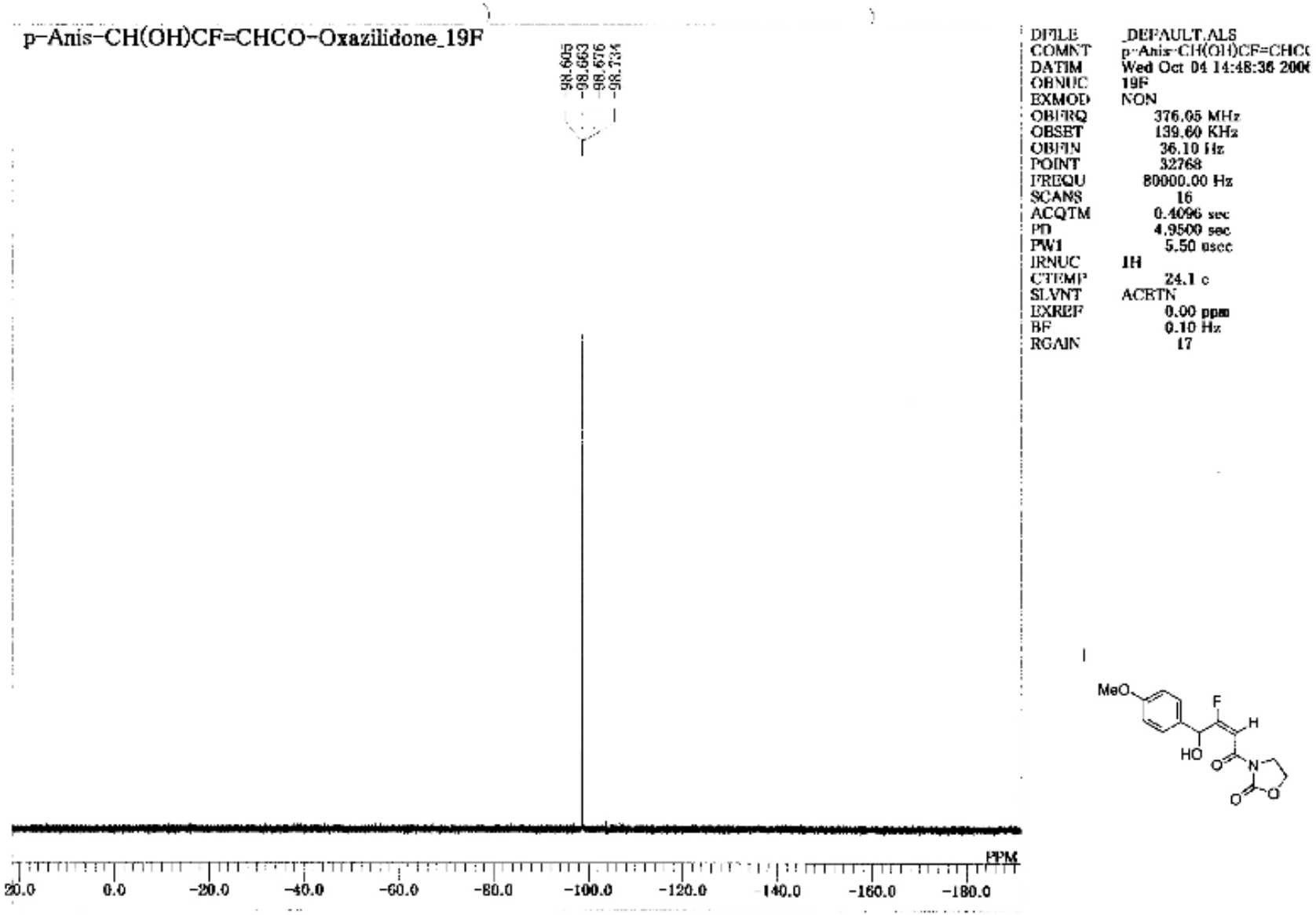



```
Current Data Parameters
NAME        shima-061004
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20061004
Time               16.02
INSTRUM          drx500
PROBHD   5 mm Multinucl
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                    23
DS                     2
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1719923 sec
RG                   114
DW               48.400 usec
DE                6.00 usec
TE              297.5 K
D1            1.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                10.00 usec
PL1               -6.00 dB
SFO1         500.1330885 MHz

F2 - Processing parameters
SI                 32768
SF           500.1300099 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                  0.00

1D NMR plot parameters
CX                20.00 cm
CY                10.01 cm
F1P              10.000 ppm
F1              5001.30 Hz
F2P              -1.000 ppm
F2              -500.13 Hz
PPMCM            0.55000 ppm/cm
HZCM           275.07150 Hz/cm
```

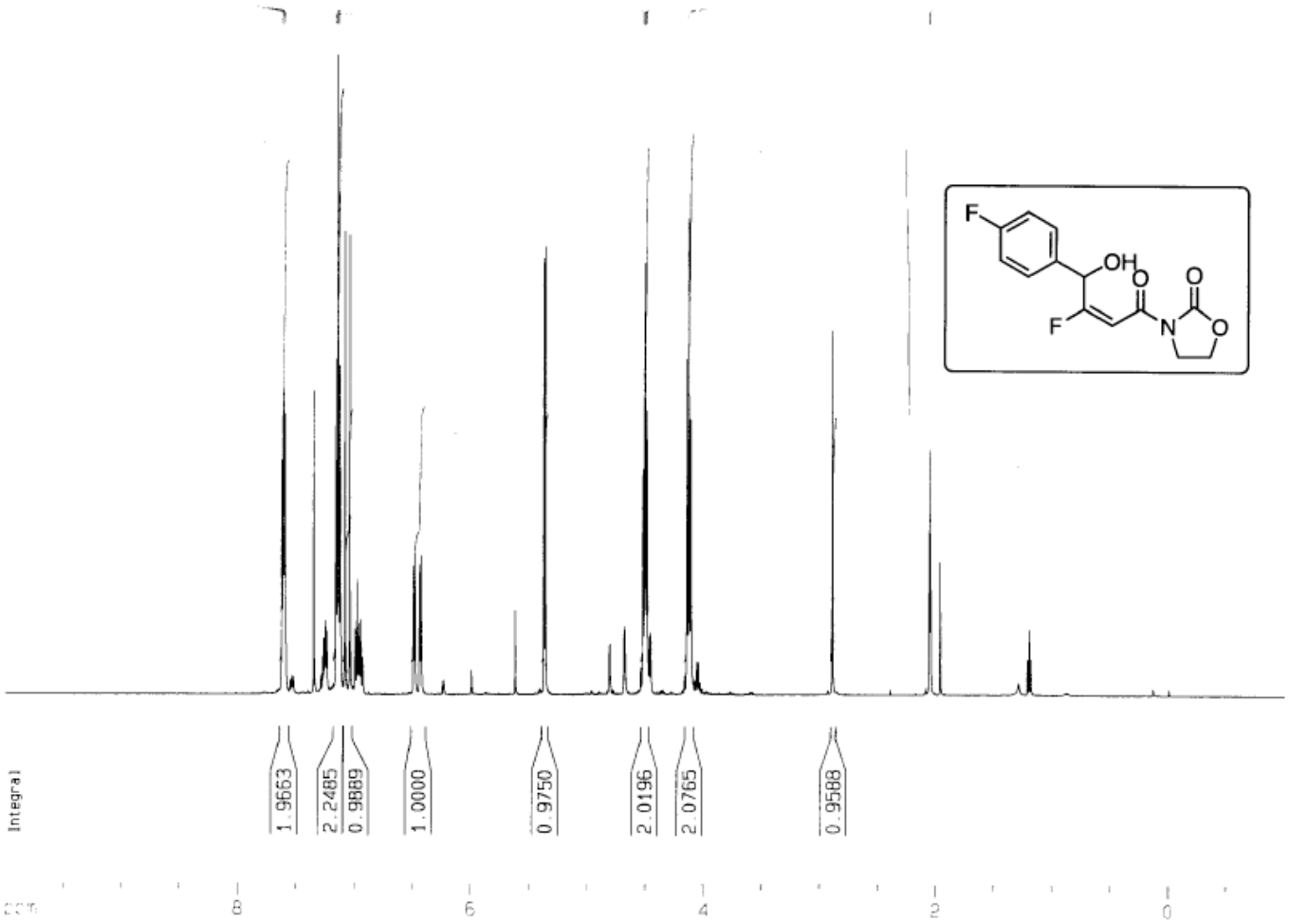
p-Anis-CH(OH)CF=CHCO-Oxazolidone_13C




```
Current Data Parameters
NAME        shima-061004
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20061004
Time               16.09
INSTRUM          drx500
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                65536
SOLVENT            CDCl3
NS                   376
DS                     2
SWH            30030.029 Hz
FIDRES         0.458222 Hz
AQ             1.0912244 sec
RG                1290.2
DW                16.650 usec
DE                 6.00 usec
TE                297.8 K
D1             0.50000000 sec
D11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                 11.00 usec
PL1                -3.00 dB
SFO1          125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                -6.00 dB
PL12               17.00 dB
PL13               19.00 dB
SFO2          500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7576791 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  0.00

1D NMR plot parameters
CX                 20.00 cm
CY                 10.03 cm
F1P               220.266 ppm
F1                27700.20 Hz
F2P               -18.526 ppm
F2                -2329.83 Hz
PPMCM             11.93964 ppm/cm
HZCM          1501.50159 Hz/cm
```

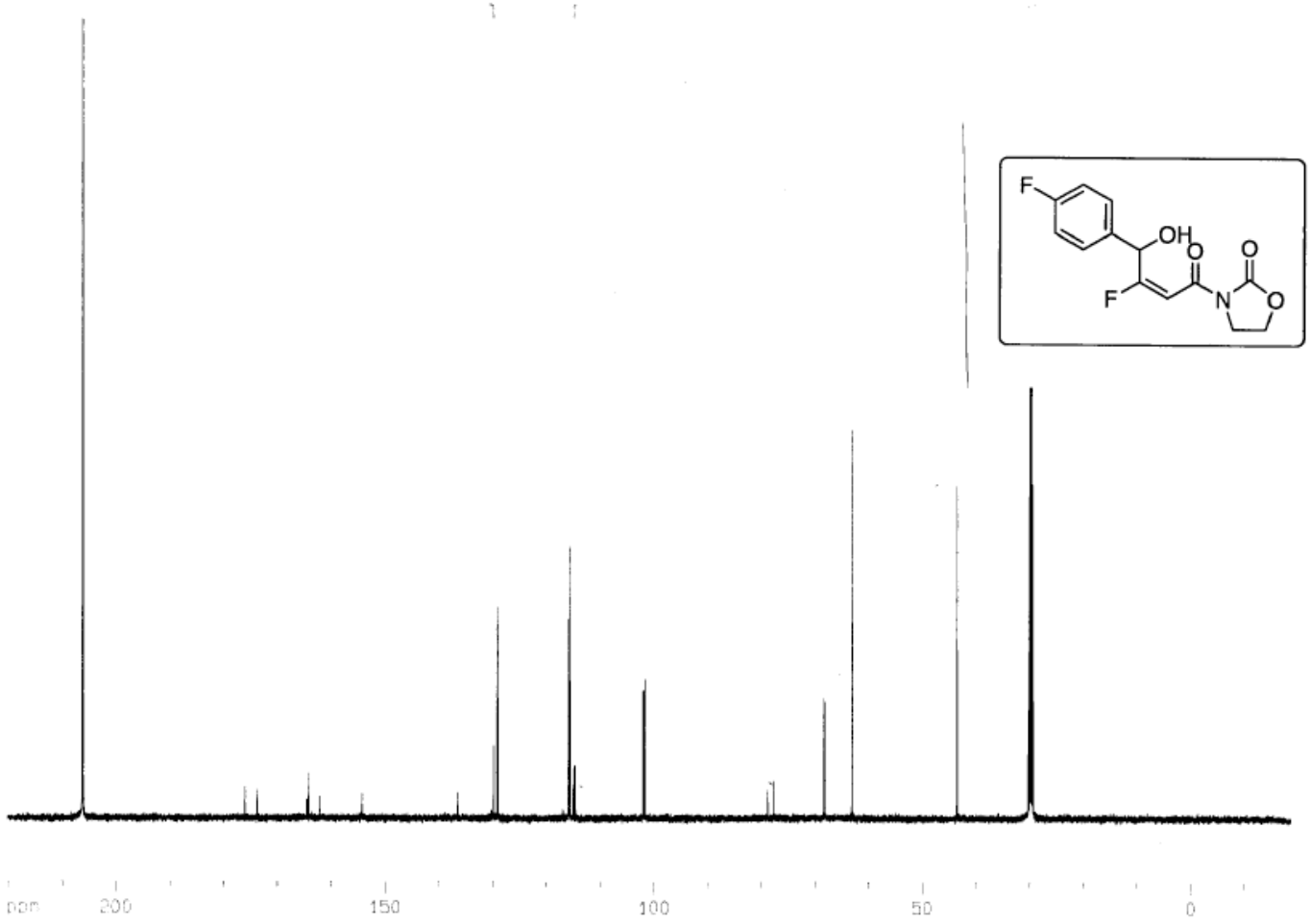


p-FC6H4CHOHCF=CHCO-Oxa_1H

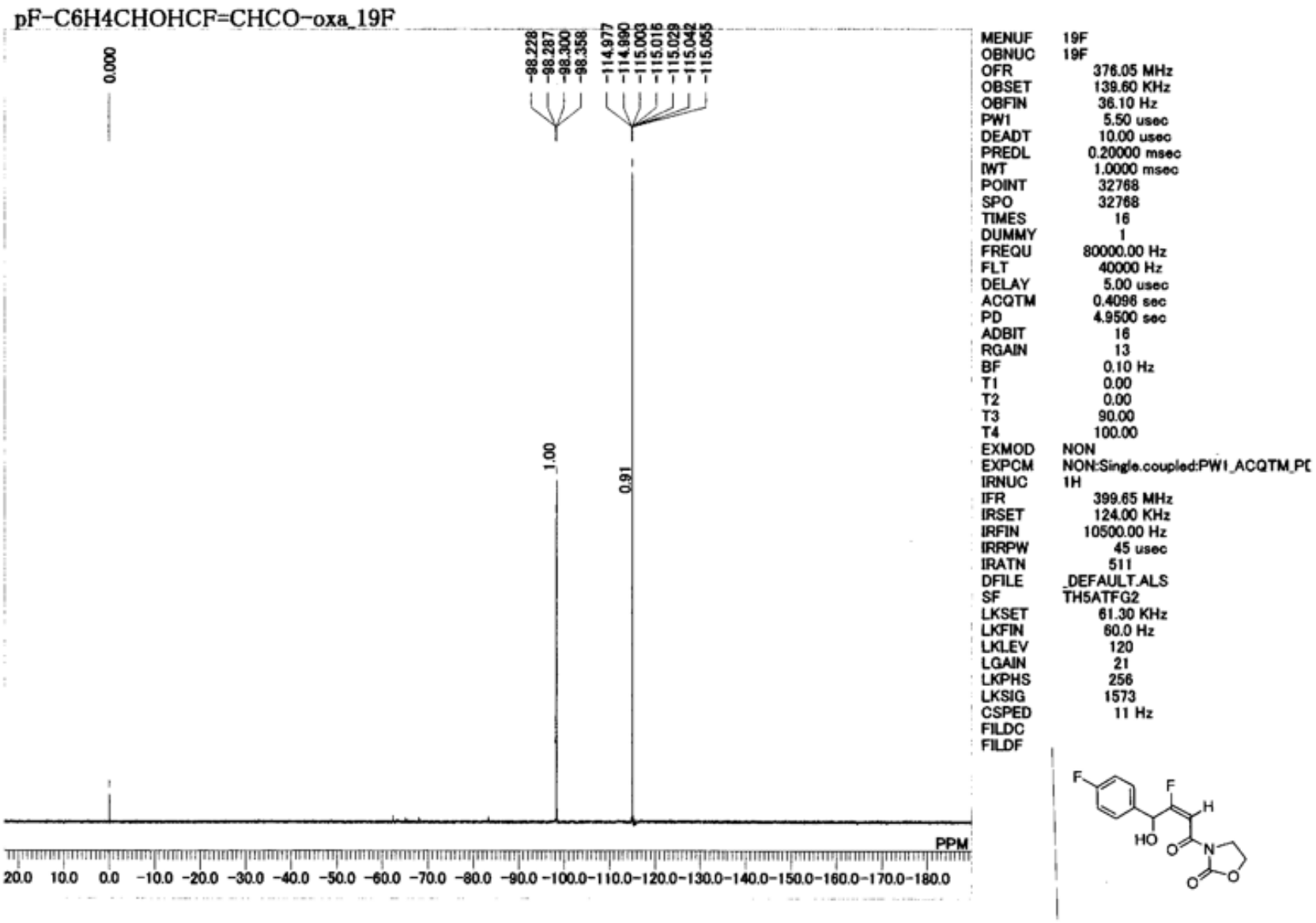


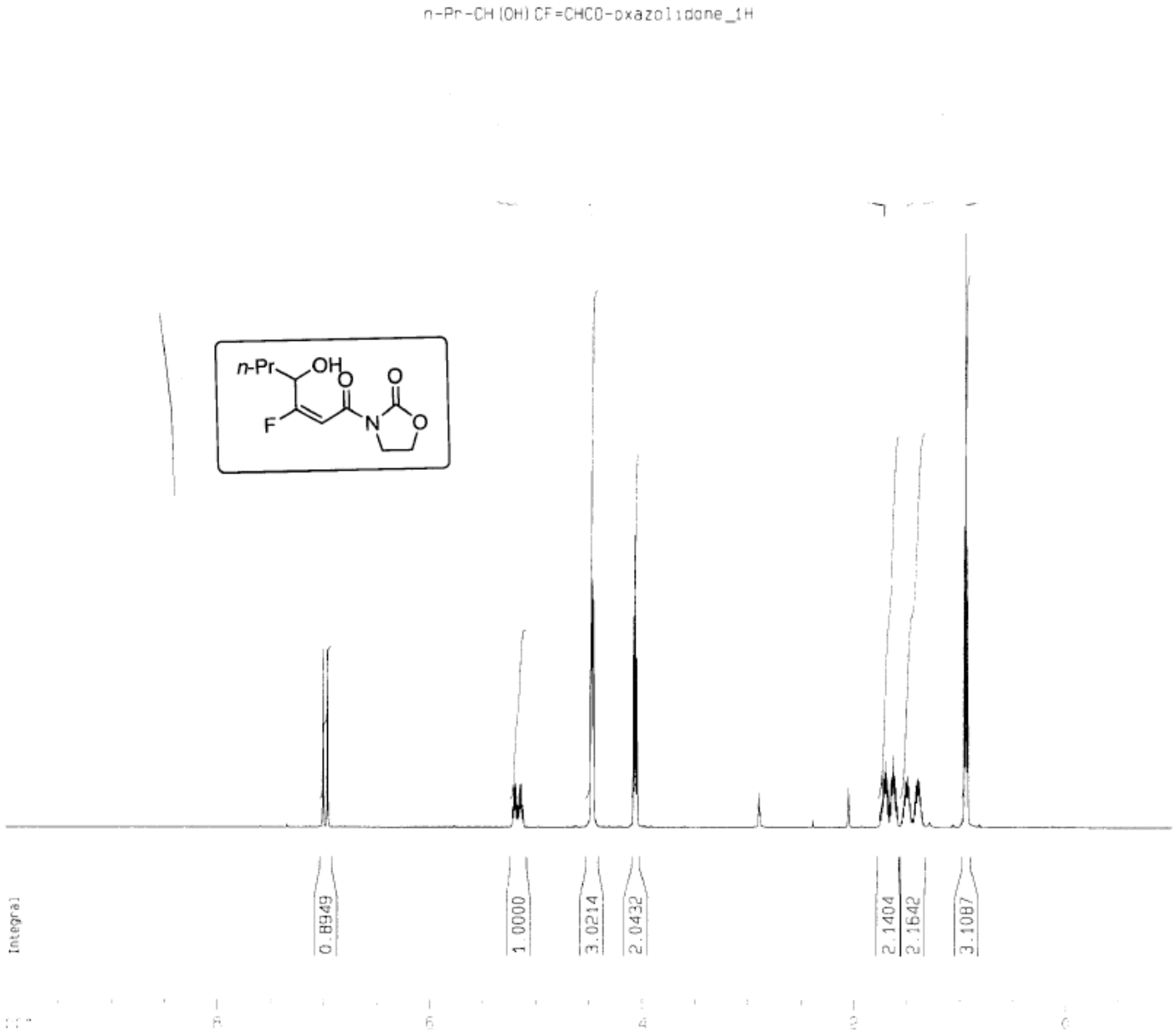

```
Current Data Parameters
NAME        shima-070128
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070129
Time                4.21
INSTRUM           drx500
PROBHD   5 mm Multinucl
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                    34
DS                     2
SWH          10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1719923 sec
RG                   181
DW               48.400 usec
DE                6.00 usec
TE                293.6 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                10.00 usec
PL1                -6.00 dB
SFO1         500.1330885 MHz

F2 - Processing parameters
SI                 32768
SF          500.1300099 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                0.00

1D NMR plot parameters
CX                20.00 cm
CY                10.06 cm
F1P              10.000 ppm
F1              5001.30 Hz
F2P              -1.000 ppm
F2              -500.13 Hz
PPMCM            0.55000 ppm/cm
HZCM           275.07150 Hz/cm
```

p-FC6H4CHOHCF=CHCO-Oxa_13C



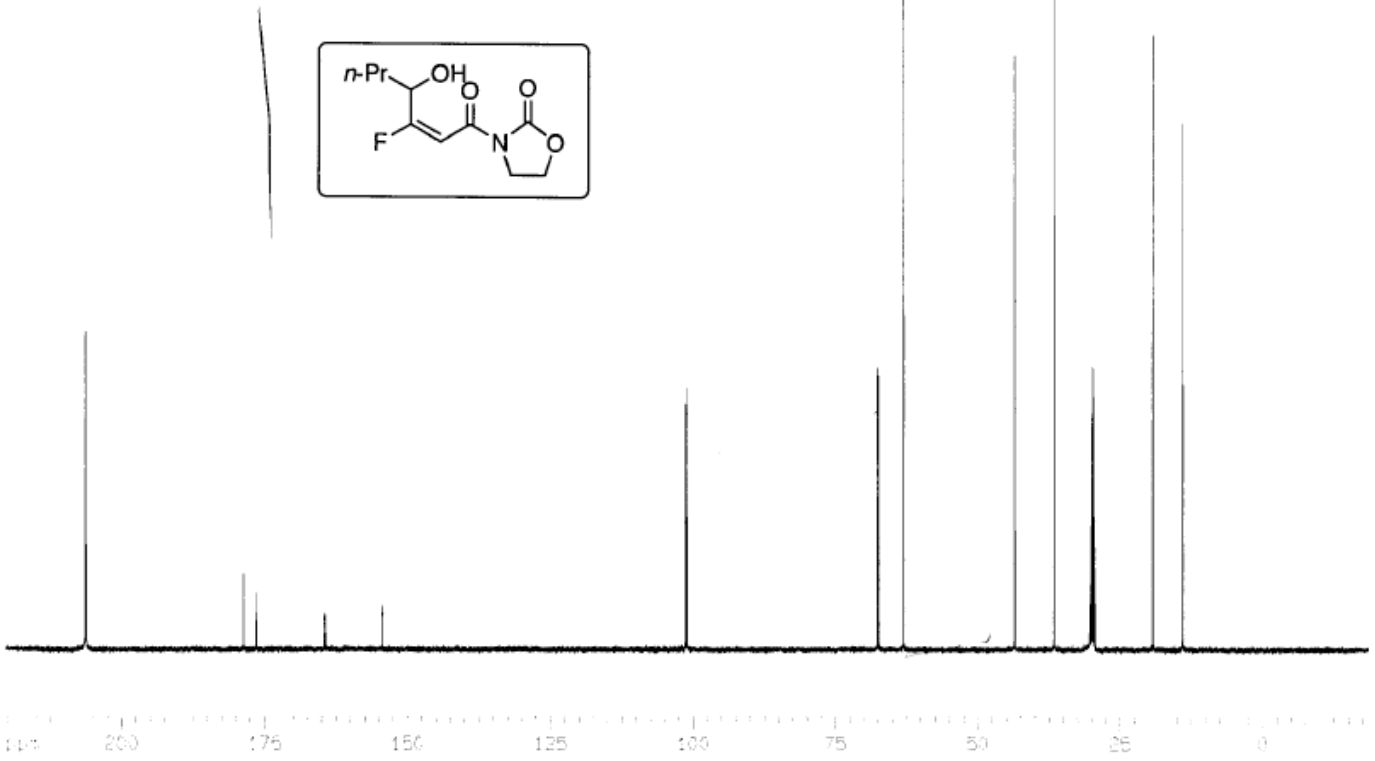

```
Current Data Parameters
NAME        shima-070128
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070129
Time                4.29
INSTRUM          drx500
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  542
DS                    2
SWH           30030.029 Hz
FIDRES        0.458222 Hz
AQ            1.0912244 sec
RG                3649.1
DW               16.650 usec
DE                6.00 usec
TE                294.0 K
D1            0.50000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                11.00 usec
PL1               -3.00 dB
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2               -6.00 dB
PL12              17.00 dB
PL13              19.00 dB
SFO2         500.1320005 MHz

F2 - Processing parameters
SI                  32768
SF           125.7576819 MHz
WDW                    EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  0.00

1D NMR plot parameters
CX                 20.00 cm
CY                 14.20 cm
F1P               220.245 ppm
F1               27697.45 Hz
F2P               -18.548 ppm
F2               -2332.58 Hz
PPMCM             11.93964 ppm/cm
HZCM           1501.50159 Hz/cm
```



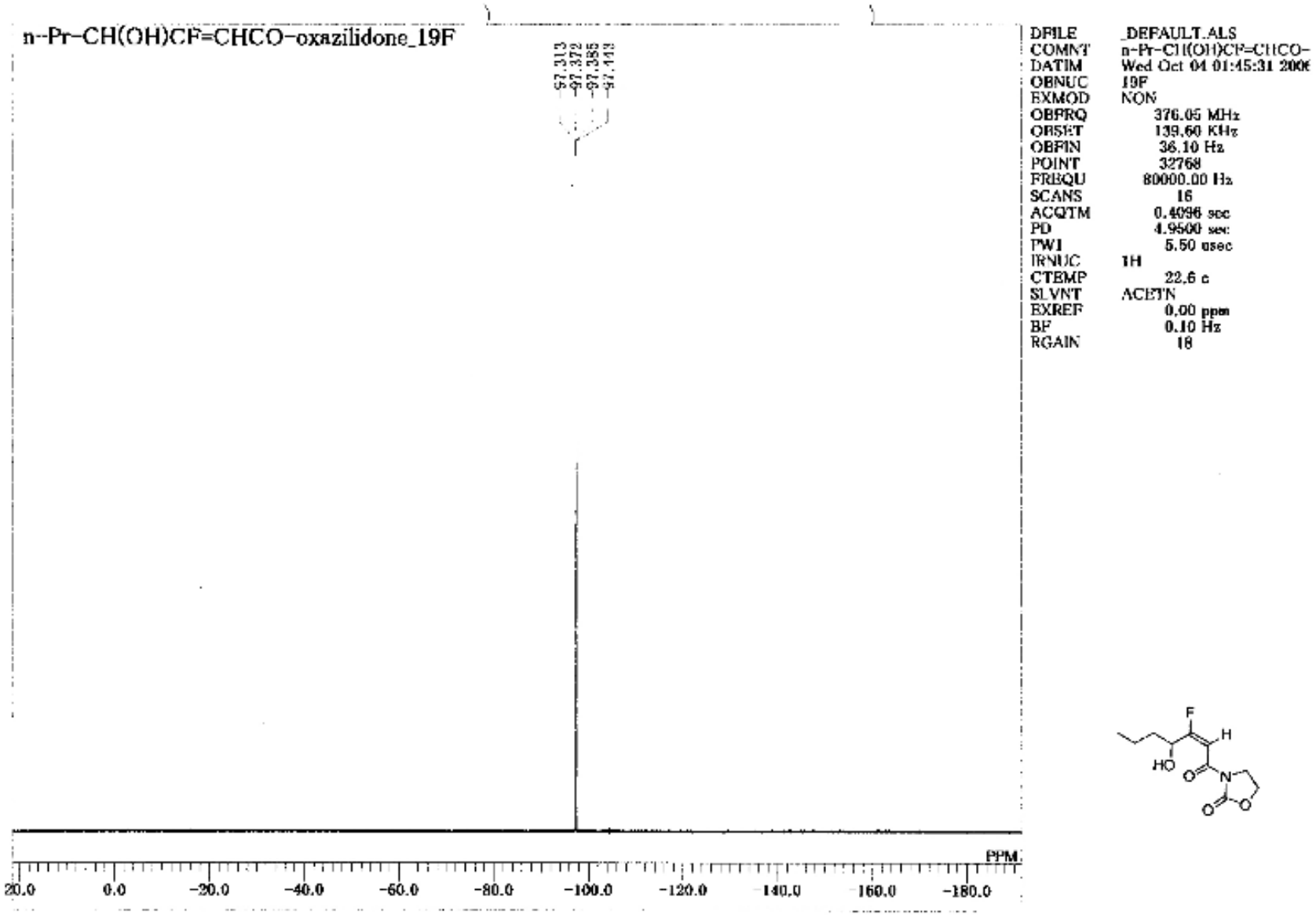


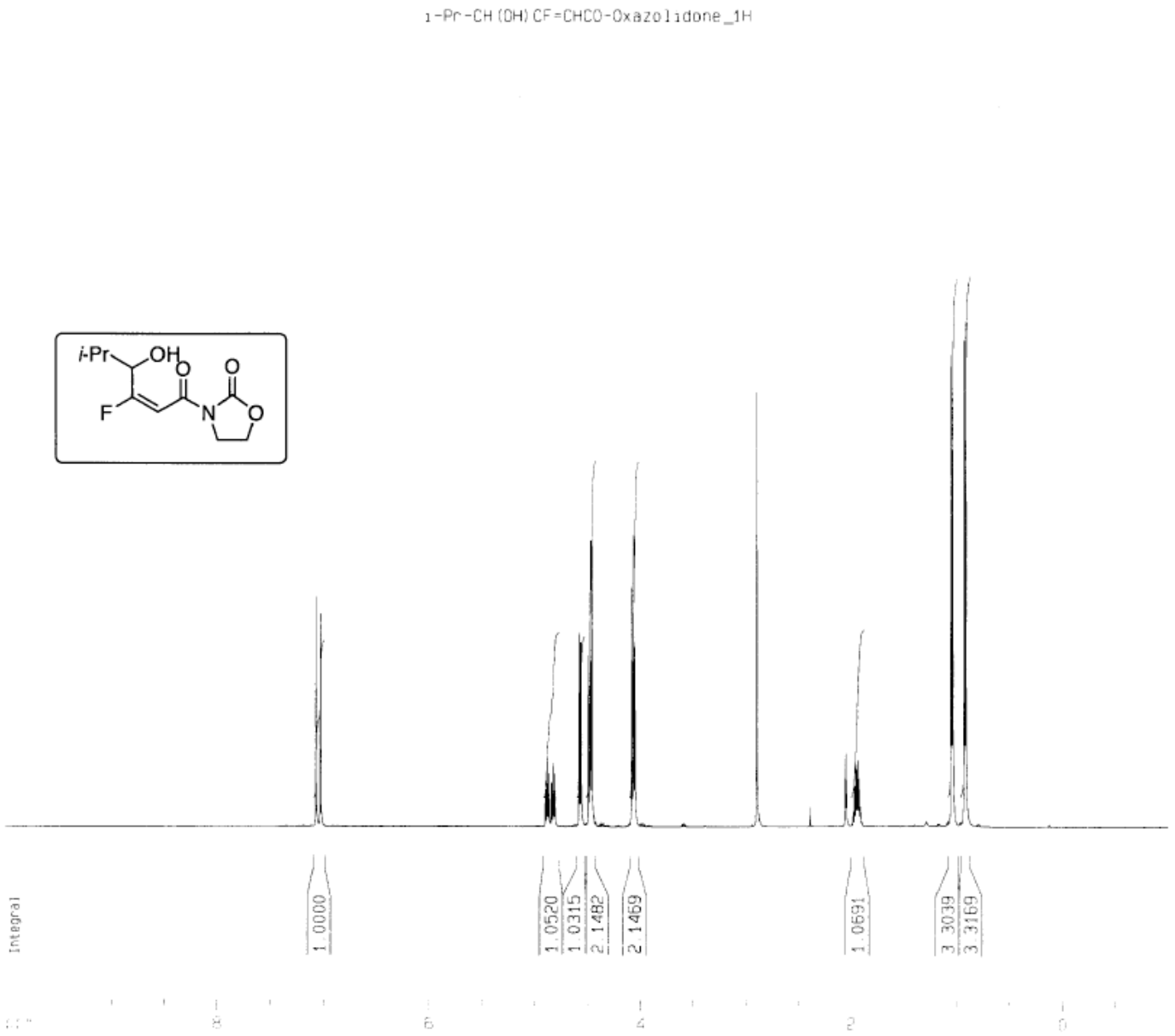

```
Current Data Parameters
NAME        shima-060929
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060930
Time                0.04
INSTRUM          qnx500
PROBHD   5 mm Multinucl
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                    46
DS                     2
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1719923 sec
RG                  71.8
DW                48.400 usec
DE                 6.00 usec
TE                297.4 K
D1            1.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.00 usec
PL1                -6.00 dB
SFO1         500.1330885 MHz

F2 - Processing parameters
SI                 32768
SF          500.1300099 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  0.00

1D NMR plot parameters
CX                 20.00 cm
CY                 10.27 cm
F1P               10.000 ppm
F1                5001.30 Hz
F2P               -1.000 ppm
F2                -500.13 Hz
PPMCM             0.55000 ppm/cm
HZCM            275.07150 Hz/cm
```

n-Pr-CH(OH)CF=CHCO-oxazolidone_13C



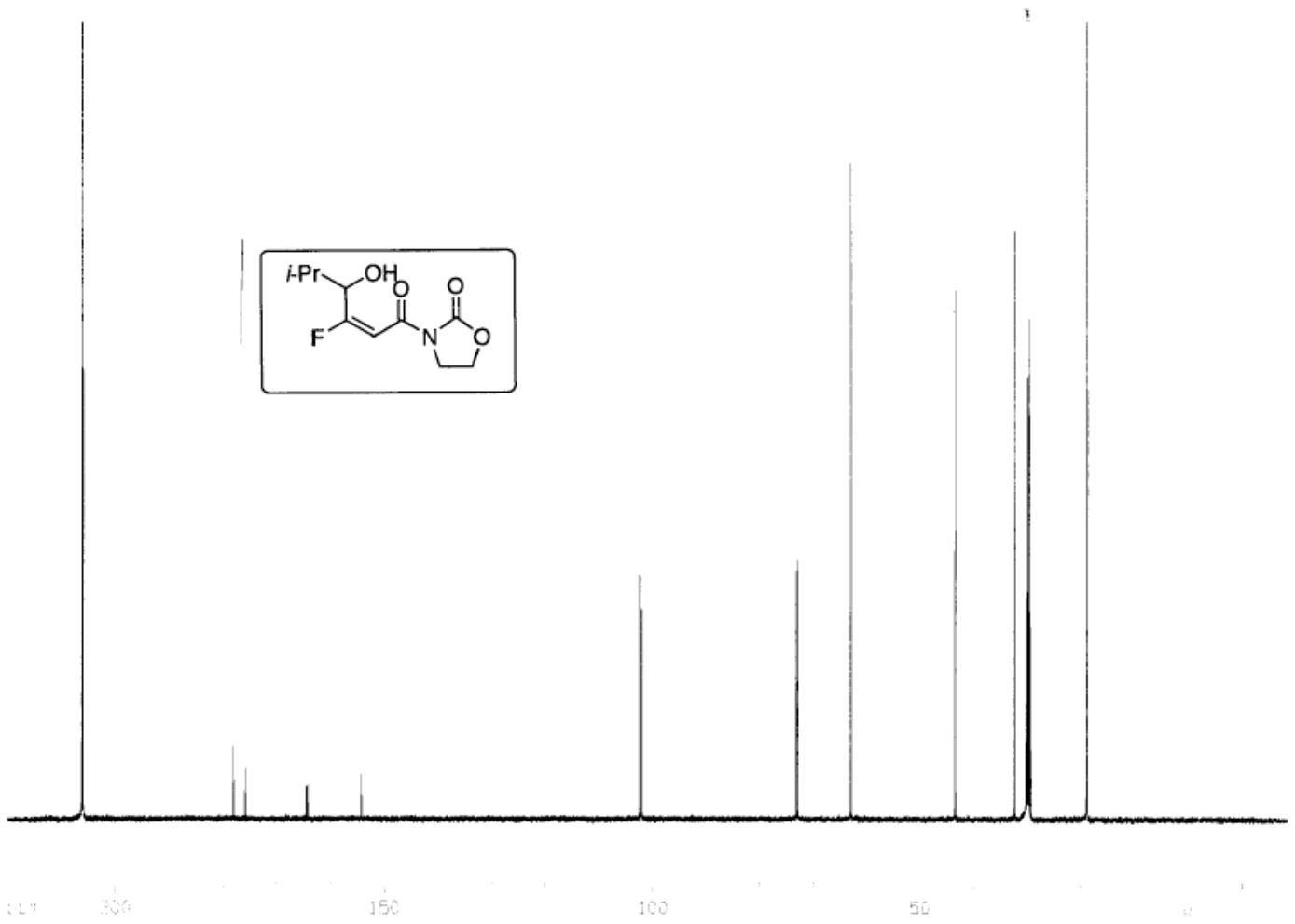

```
Current Data Parameters
NAME        shima-060929
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060930
Time                0.15
INSTRUM          drx500
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  286
DS                    2
SWH           30030.029 Hz
FIDRES         0.458222 Hz
AQ             1.0912244 sec
RG                 2048
DW               16.650 usec
DE                6.00 usec
TE                297.8 K
D1            0.50000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                11.00 usec
PL1               -3.00 dB
SFO1        125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2           100.00 usec
PL2               -6.00 dB
PL12              17.00 dB
PL13              19.00 dB
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7576800 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                  0.00

1D NMR plot parameters
CX                20.00 cm
CY                10.17 cm
F1P              220.259 ppm
F1              27699.29 Hz
F2P              -18.534 ppm
F2              -2330.75 Hz
PPMCM          11.93964 ppm/cm
HZCM         1501.50159 Hz/cm
```



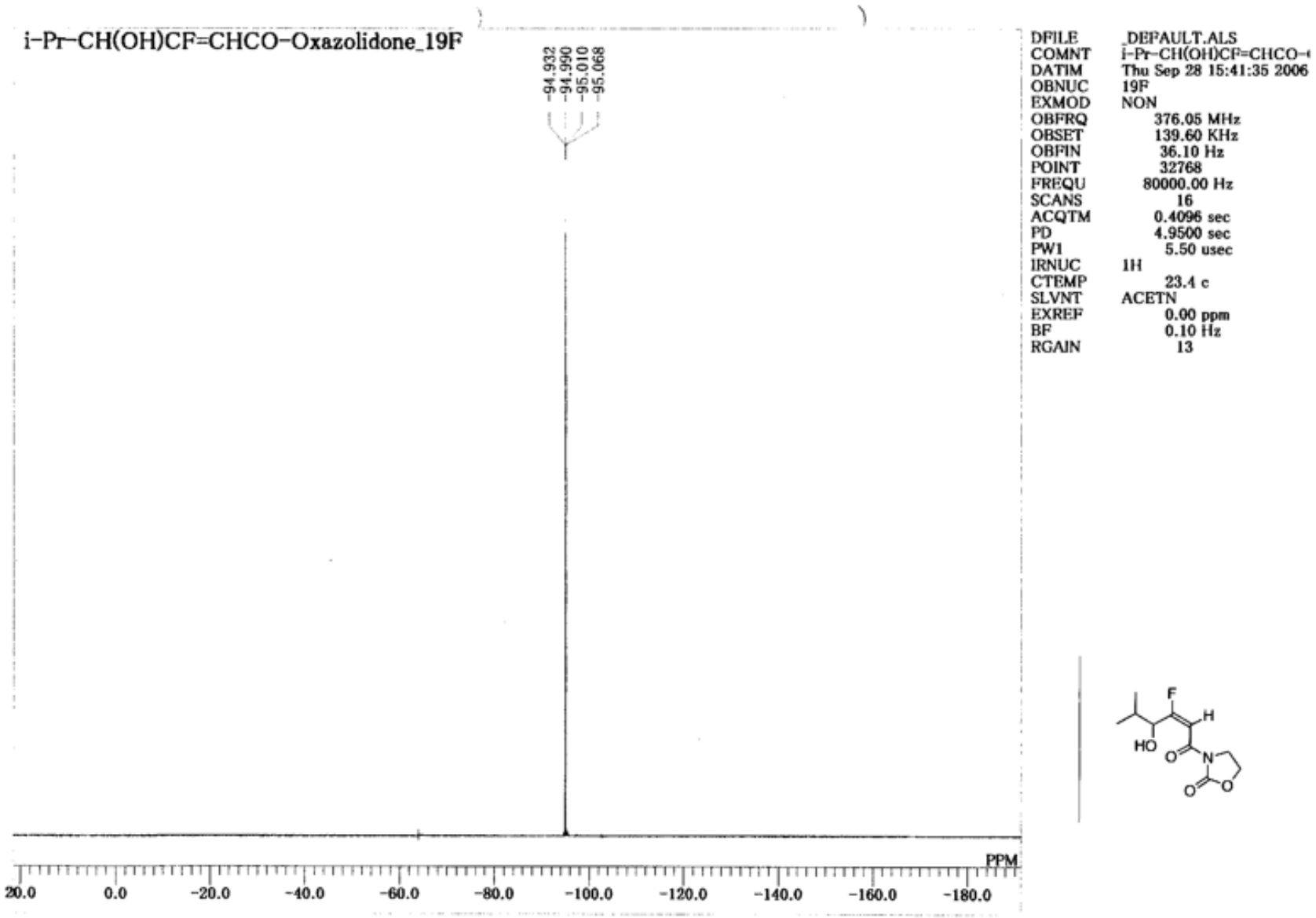



```
Current Data Parameters
NAME        shima-060927
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060928
Time                0.25
INSTRUM          drx500
PROBHD   5 mm Multinucl
PULPROG             zg30
TD                 65536
SOLVENT          Acetone
NS                    40
DS                     2
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1719923 sec
RG                  80.6
DW                48.400 usec
DE                 6.00 usec
TE                297.6 K
D1            1.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 10.00 usec
PL1                -6.00 dB
SFO1          500.1330885 MHz

F2 - Processing parameters
SI                  32768
SF            500.1300099 MHz
WDW                    EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  0.00

1D NMR plot parameters
CX                 20.00 cm
CY                  8.75 cm
F1P               10.000 ppm
F1              5001.30 Hz
F2P               -1.000 ppm
F2              -500.13 Hz
PPMCM             0.55000 ppm/cm
HZCM            275.07150 Hz/cm
```

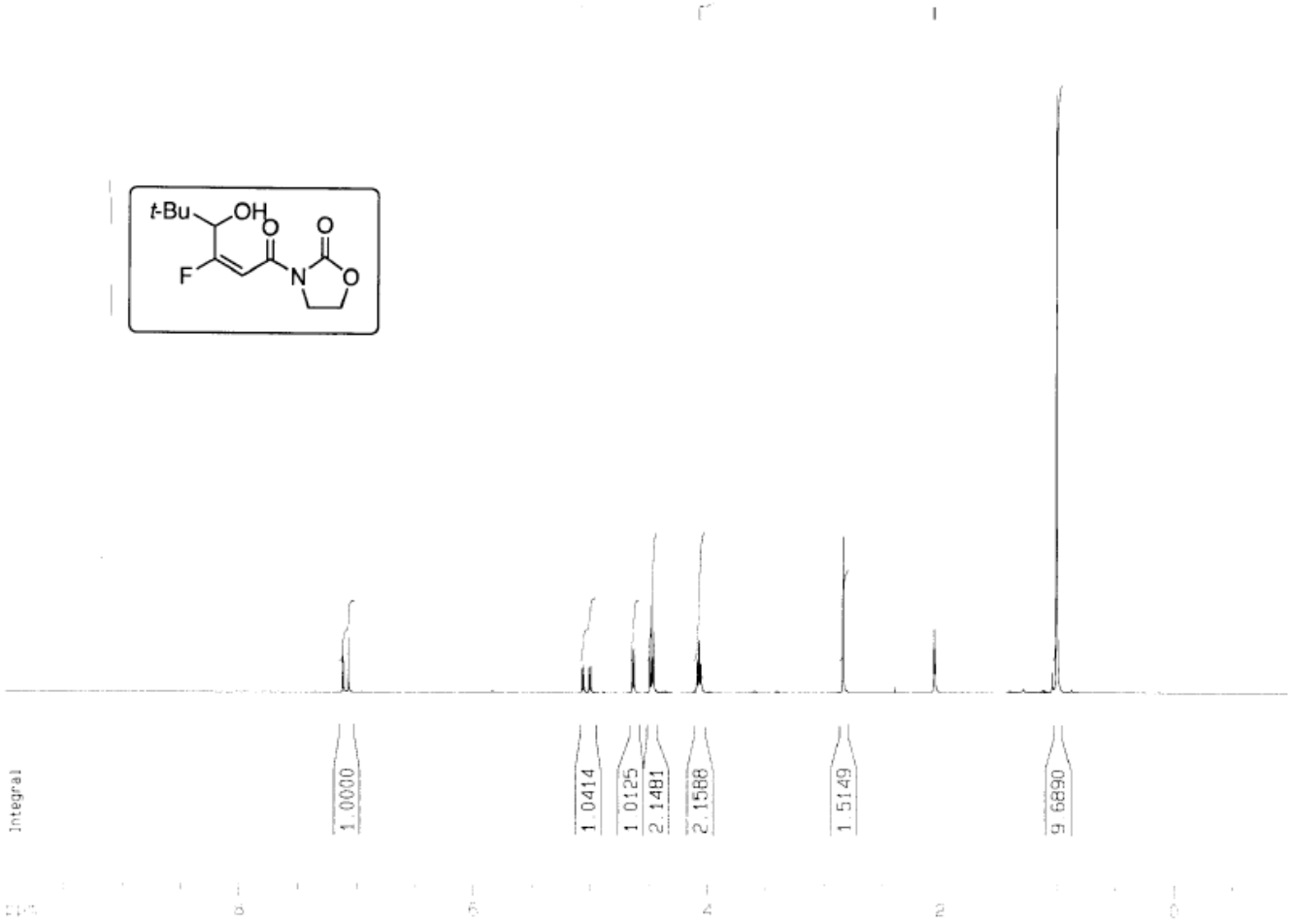
1-Pr-CH(OH)CF=CHCO-Oxazolidone_13C



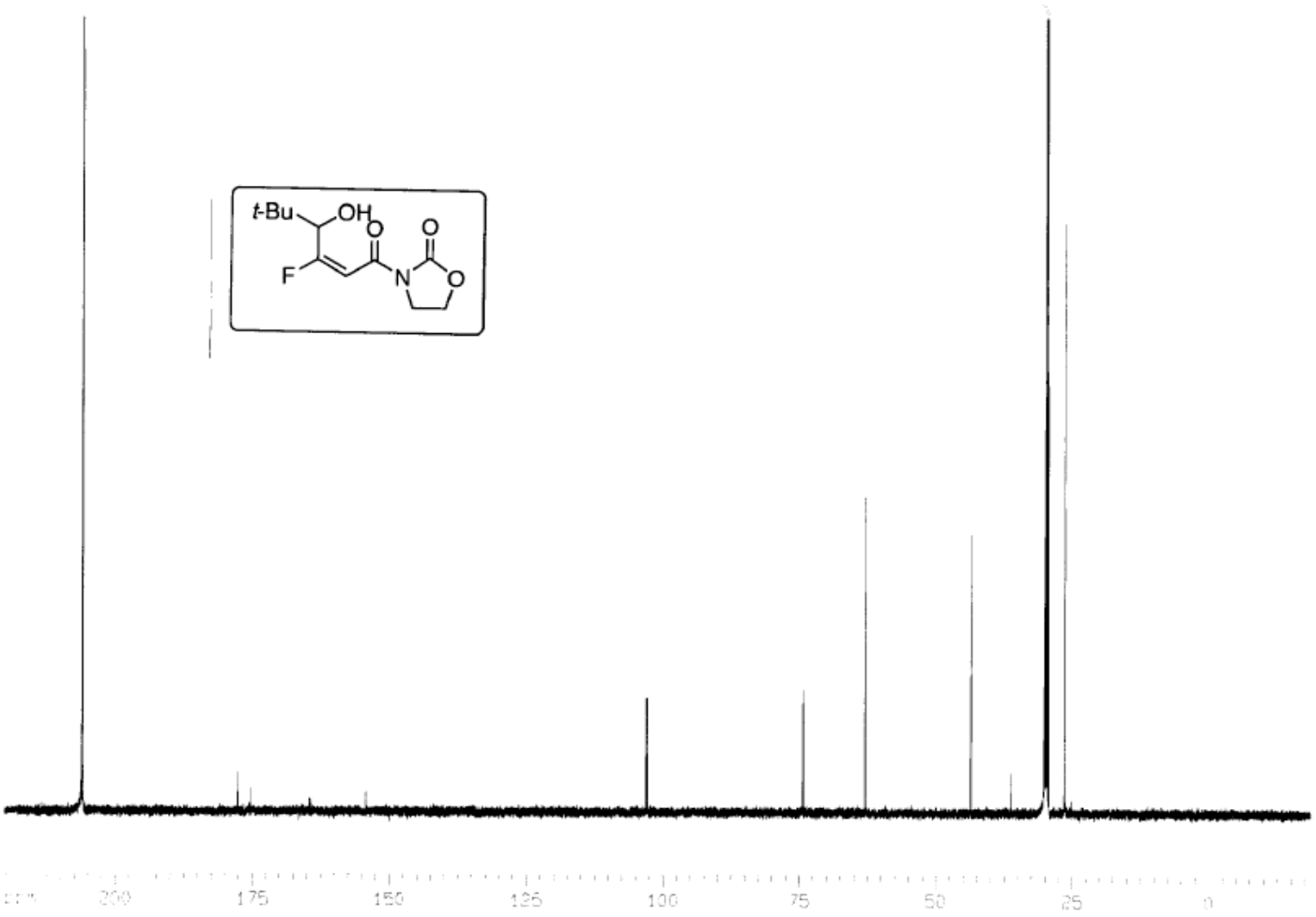

```
Current Data Parameters
NAME        shina-060927
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060928
Time                0.44
INSTRUM           drx500
PROBHD   5 mm Multinucl
PULPROG           zgpg30
TD                 65536
SOLVENT             CDCl3
NS                    625
DS                      2
SWH             30030.029 Hz
FIDRES          0.458222 Hz
AQ              1.0912244 sec
RG                 1625.5
DW                 16.650 usec
DE                  6.00 usec
TE                 298.0 K
D1              0.50000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                  11.00 usec
PL1                 -3.00 dB
SFO1          125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                 -6.00 dB
PL12                17.00 dB
PL13                19.00 dB
SFO2          500.1320005 MHz

F2 - Processing parameters
SI                    32768
SF            125.7576851 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     0.00

1D NMR plot parameters
CX                    20.00 cm
CY                    12.82 cm
F1P                 220.219 ppm
F1                27694.26 Hz
F2P                 -18.574 ppm
F2                -2335.78 Hz
PPMCM              11.93964 ppm/cm
HZCM            1501.50159 Hz/cm
```



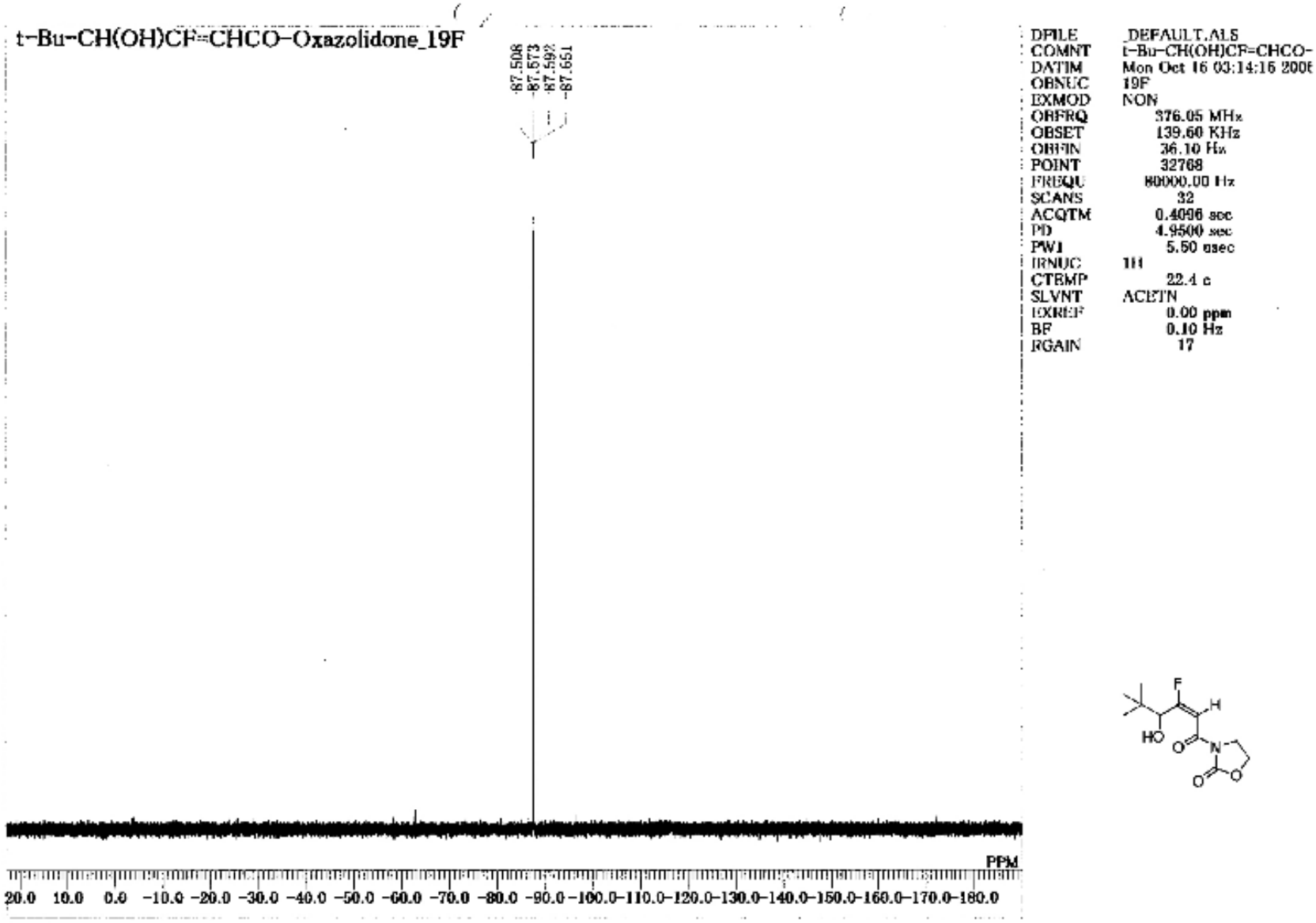
t-Bu-CH(OH)CF=CHCO-Oxazolidone_1H

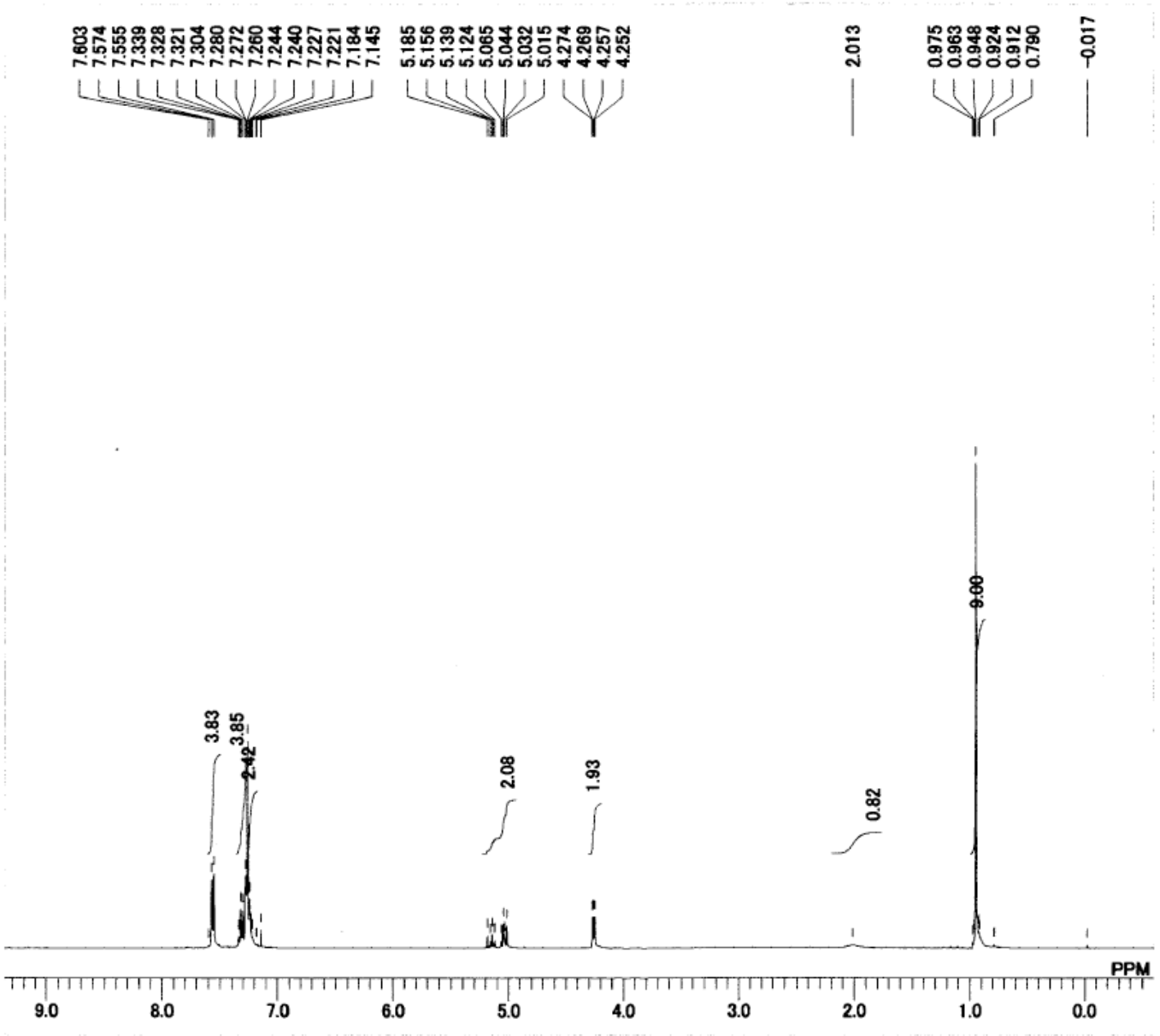



```
Current Data Parameters
NAME        shima-061016
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20061016
Time               12.32
INSTRUM          drx500
PROBHD   5 mm Multinucl
PULPROG            zg30
TD                65536
SOLVENT         Acetone
NS                    25
DS                     2
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1719923 sec
RG                   256
DW                48.400 usec
DE                 6.00 usec
TE                298.7 K
D1            1.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                10.00 usec
PL1               -6.00 dB
SFO1         500.1330885 MHz

F2 - Processing parameters
SI                 32768
SF          500.1300099 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 0.00

1D NMR plot parameters
CX                20.00 cm
CY                 9.52 cm
F1P               10.000 ppm
F1              5001.30 Hz
F2P               -1.000 ppm
F2              -500.13 Hz
PPMCM          0.55000 ppm/cm
HZCM          275.07150 Hz/cm
```

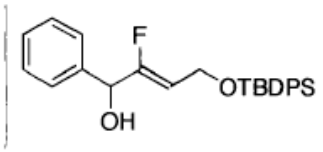


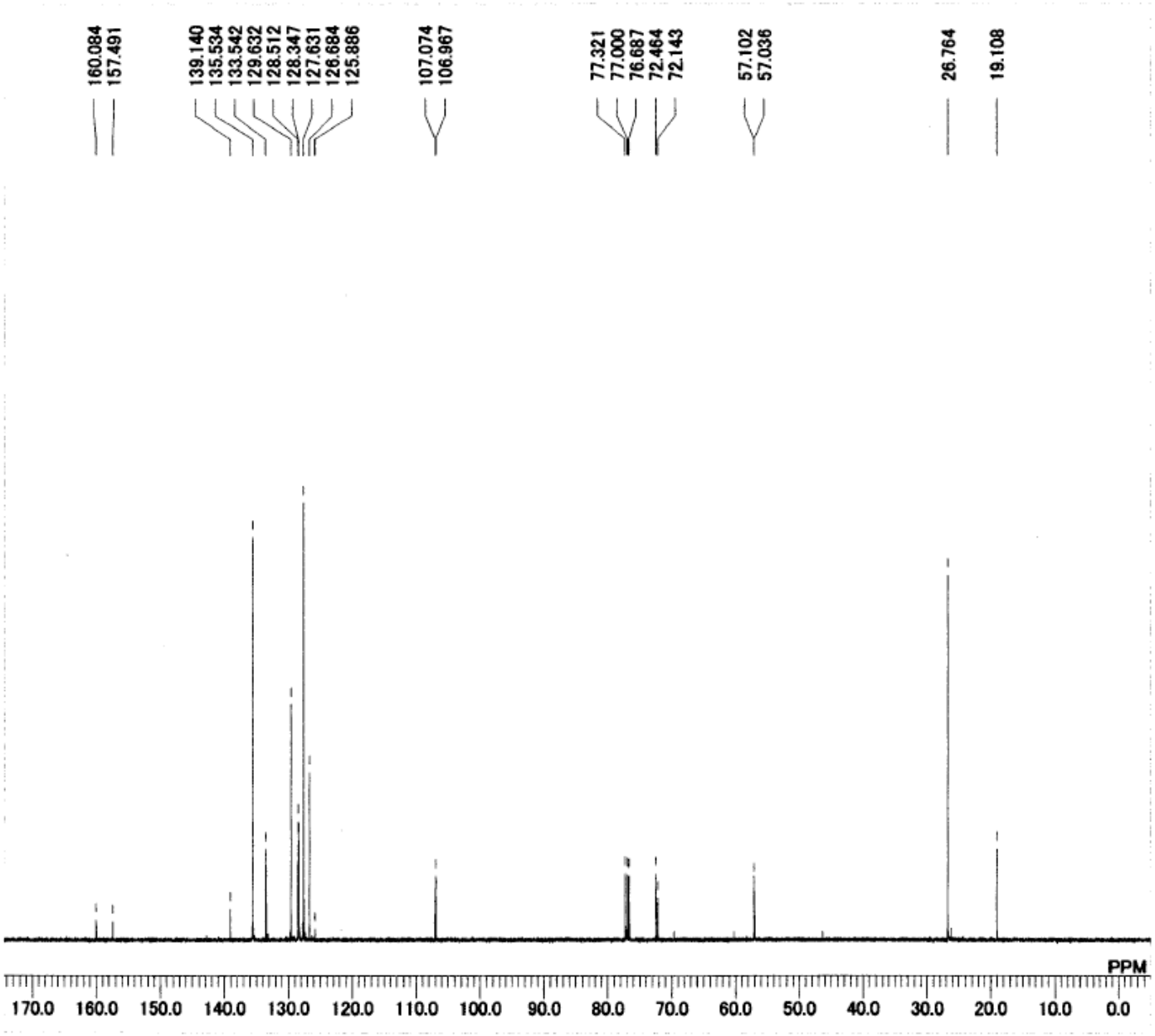

```
Current Data Parameters
NAME        shima-061016
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20061016
Time                12.39
INSTRUM          drx500
PROBHD   5 mm Multinucl
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  548
DS                    2
SWH           30030.029 Hz
FIDRES        0.458222 Hz
AQ            1.0912244 sec
RG                 2048
DW               16.650 usec
DE                6.00 usec
TE              296.9 K
D1            0.50000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                11.00 usec
PL1               -3.00 dB
SFO1        125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2            100.00 usec
PL2               -6.00 dB
PL12              17.00 dB
PL13              19.00 dB
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                 32768
SF          125.7576773 MHz
WDW                   EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 0.00

1D NMR plot parameters
CX                20.00 cm
CY                23.63 cm
F1P             220.281 ppm
F1              27702.04 Hz
F2P             -18.512 ppm
F2              -2327.99 Hz
PPMCM          11.93964 ppm/cm
HZCM        1501.50146 Hz/cm
```



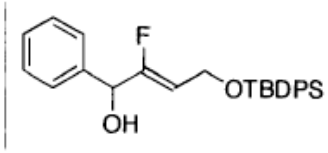


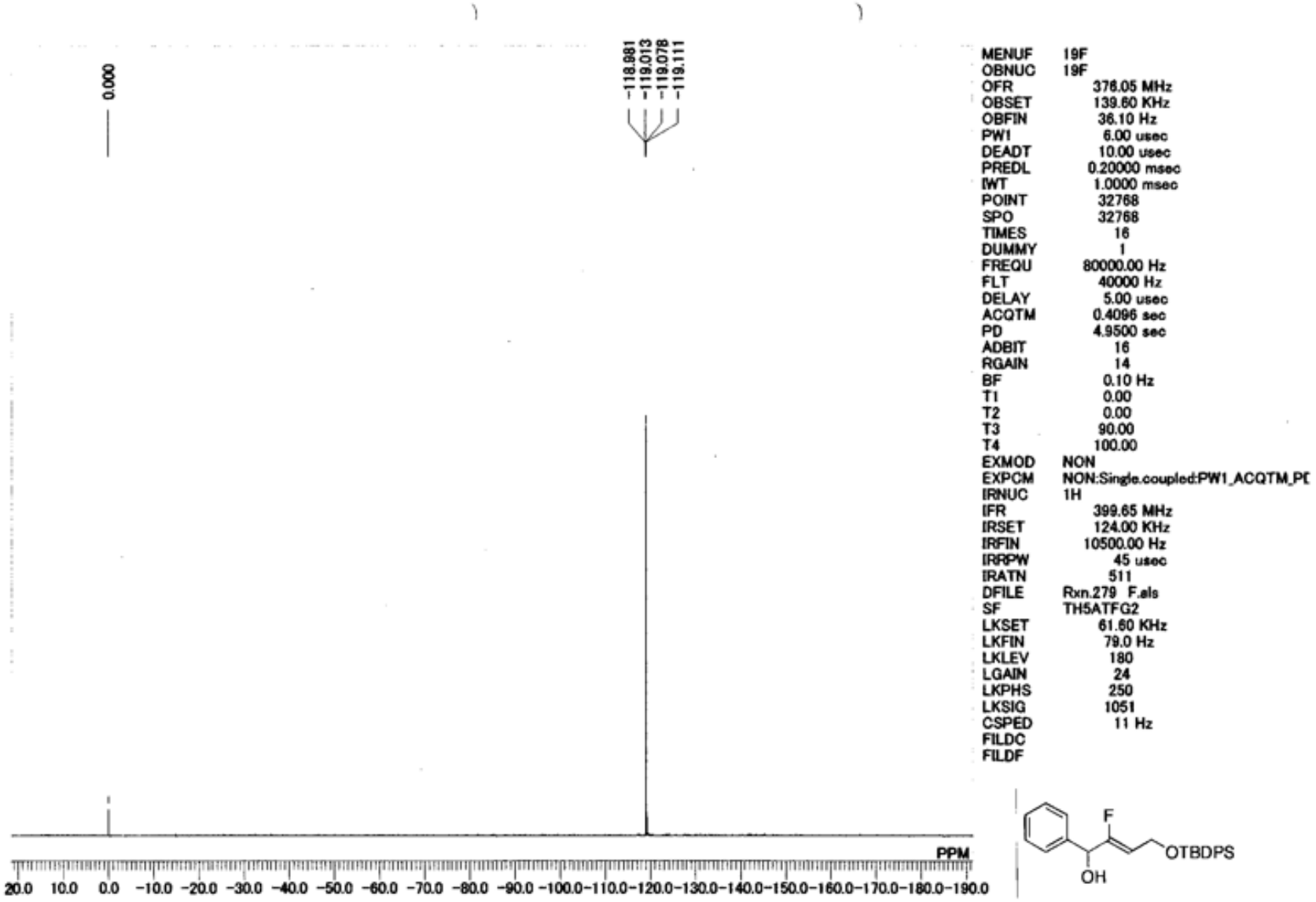
MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.50 usec
DEADT	72.20 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	13
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	11.01.08 Ph C3 H.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	23
LKPHS	240
LKSIG	854
CSPED	10 Hz
FILDC	
FILDF	

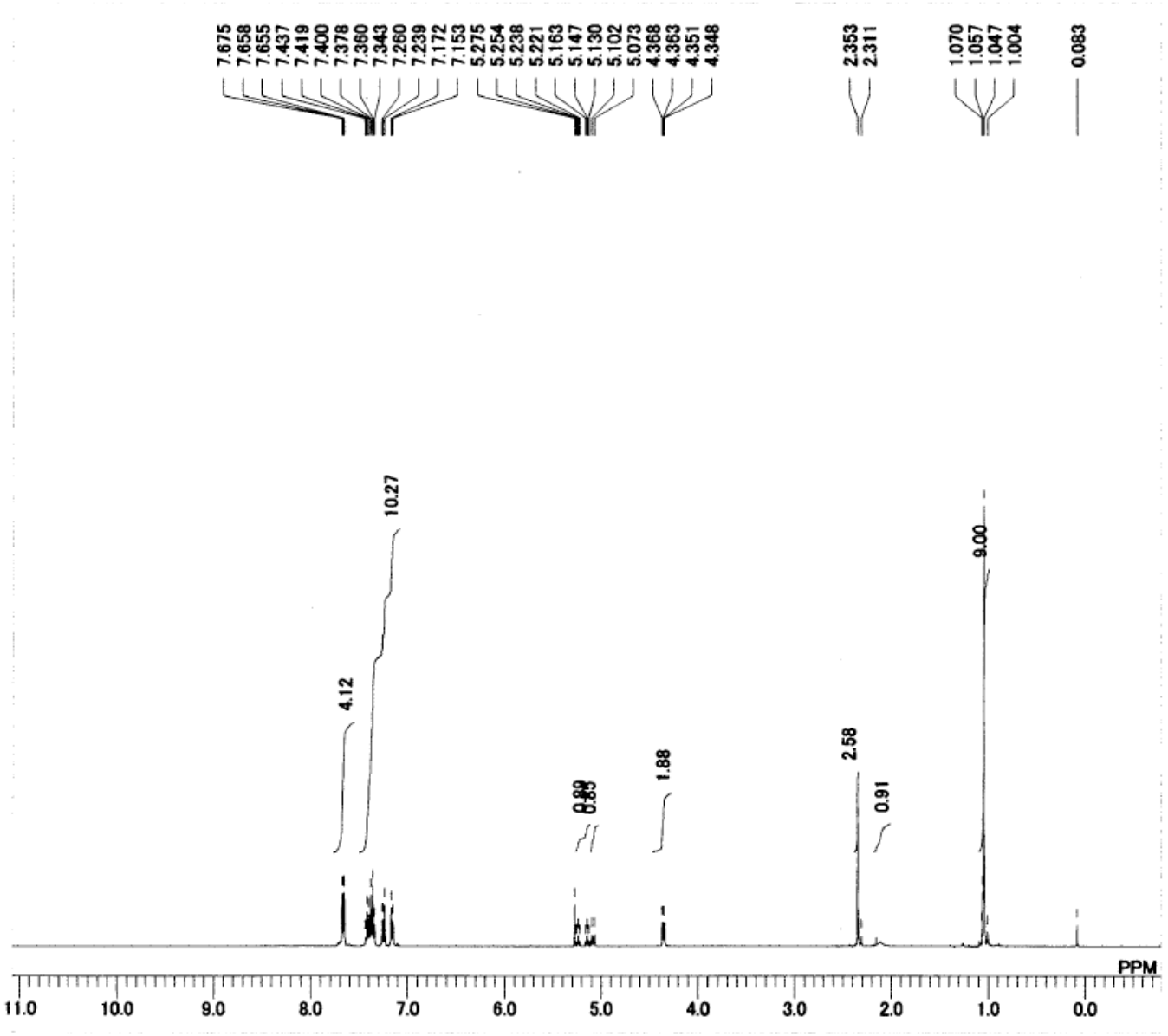




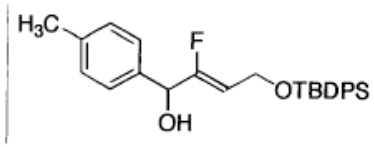
MENUF	13C
OBNUC	13C
OFR	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
PW1	6.00 usec
DEADT	19.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	256
DUMMY	1
FREQU	27118.64 Hz
FLT	13550 Hz
DELAY	14.80 usec
ACQTM	1.2083 sec
PD	1.7920 sec
ADBIT	16
RGAIN	24
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRF
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	Rxn.279 C.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	24
LKPHS	250
LKSIG	1031
CSPED	12 Hz
FILDC	
FILDF	

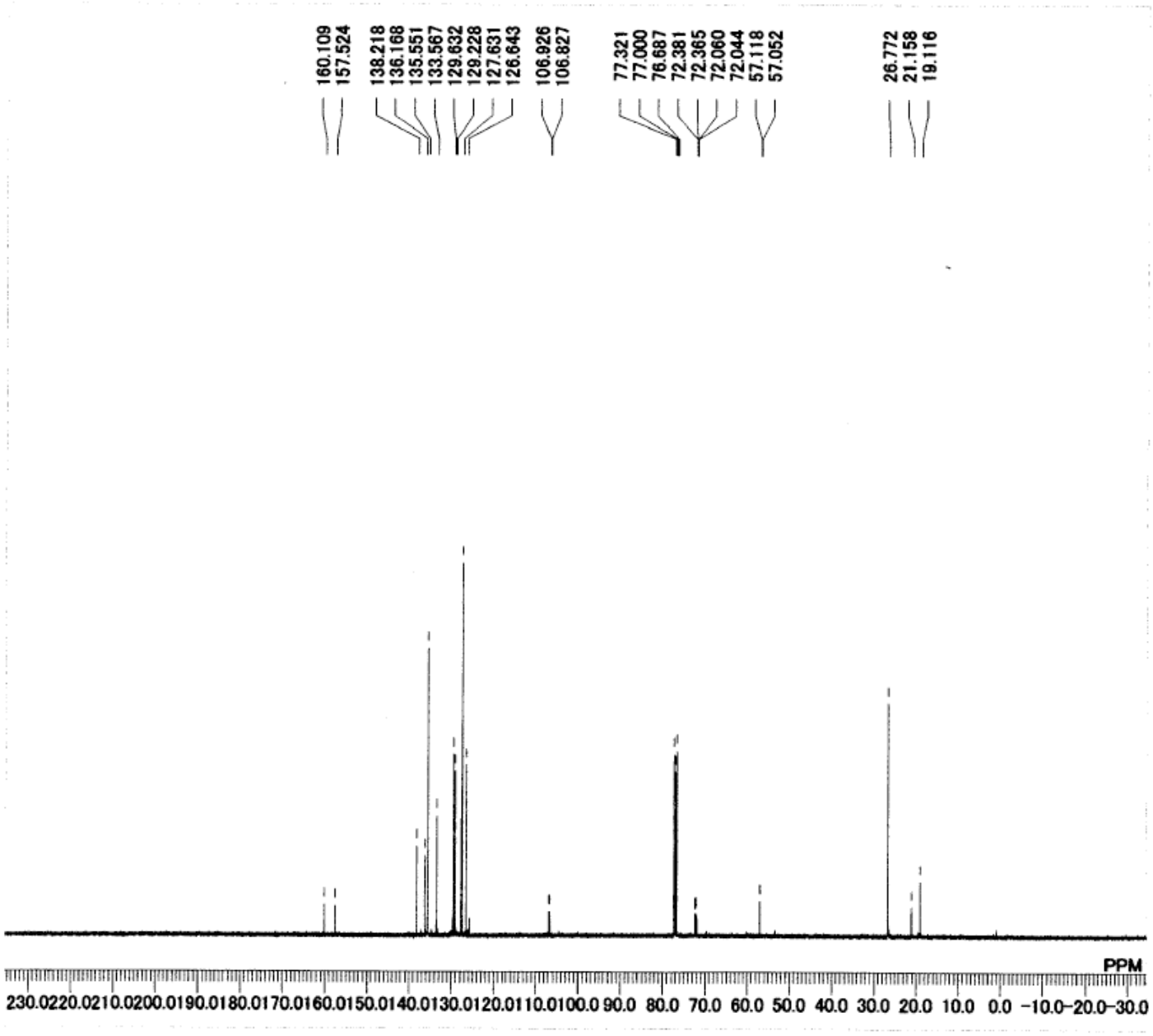




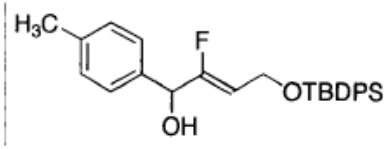


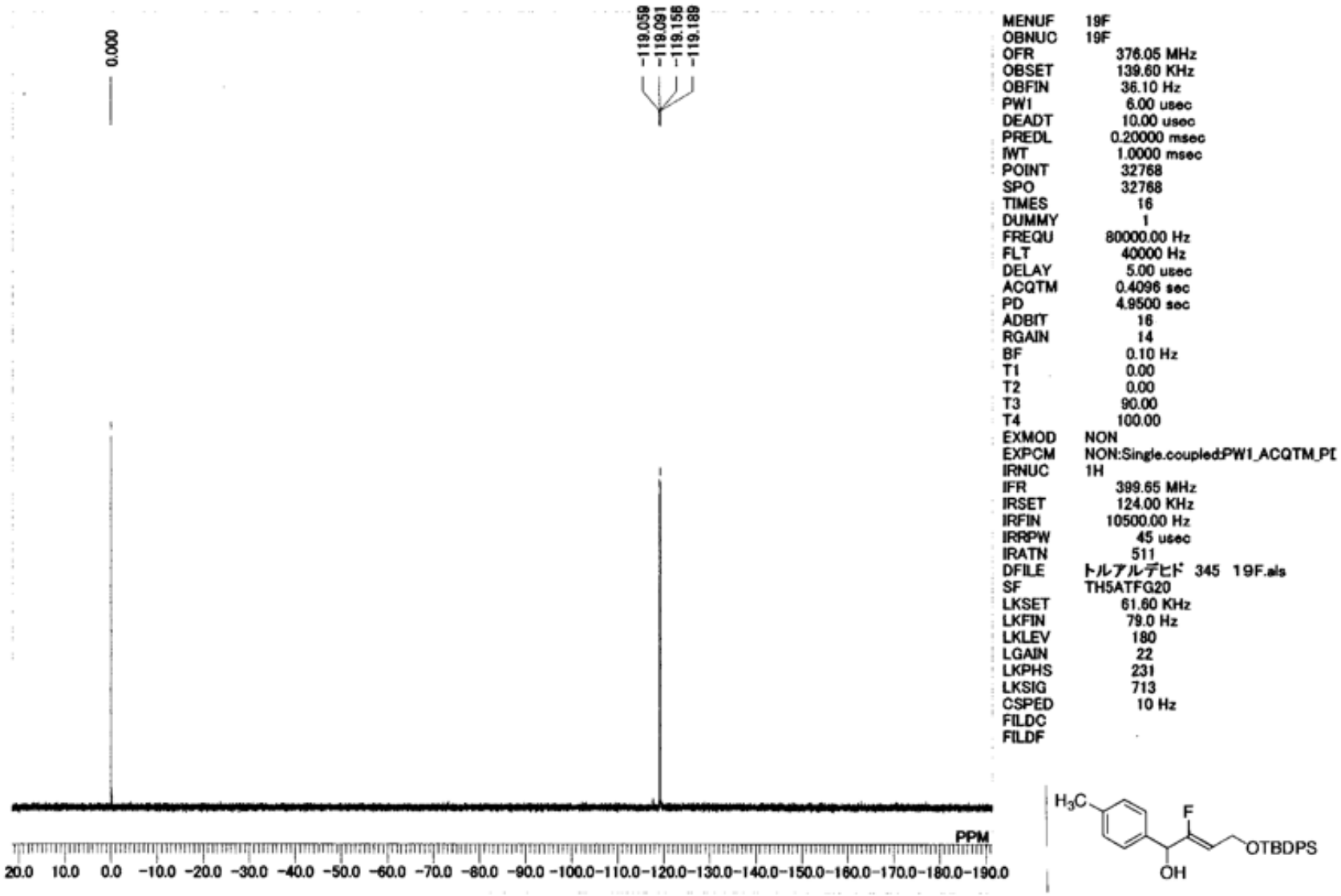
MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.50 usec
DEADT	72.20 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	13
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	トルアルデヒド 345 1H とりなおし.
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	26
LKPHS	150
LKSIG	1044
CSPED	13 Hz
FILDC	
FILDF	

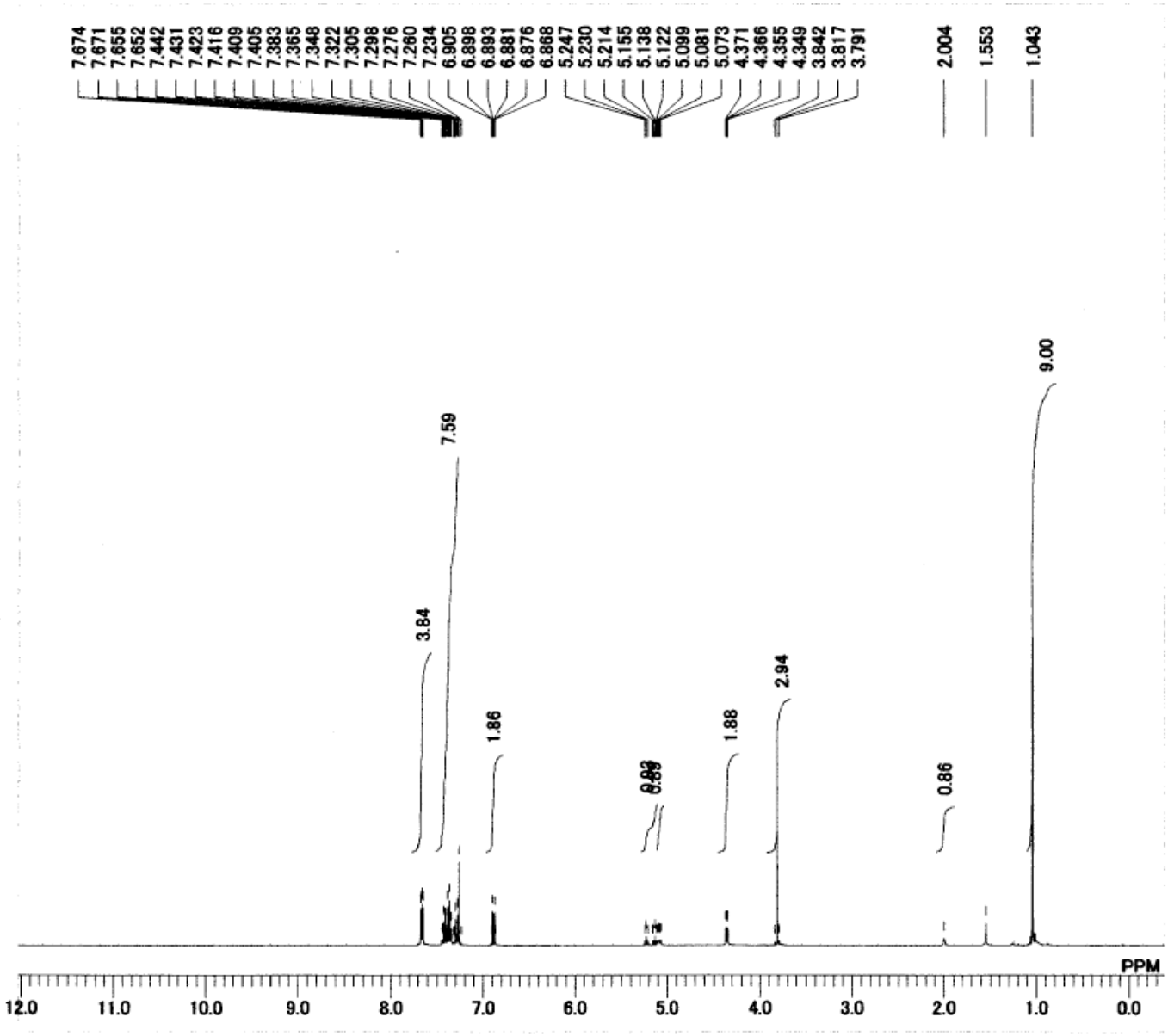




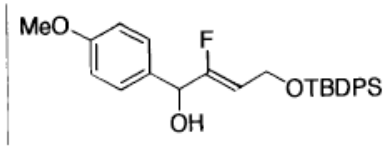
MENUF	13C
OBNUC	13C
OFR	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
PW1	6.20 usec
DEADT	19.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	10000
DUMMY	1
FREQU	27118.64 Hz
FLT	13550 Hz
DELAY	14.80 usec
ACQTM	1.2083 sec
PD	1.7920 sec
ADBIT	16
RGAIN	25
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRF
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	345 トルアルデヒド C .als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	26
LKPHS	150
LKSIG	1048
CSPED	14 Hz
FILDC	
FILDF	

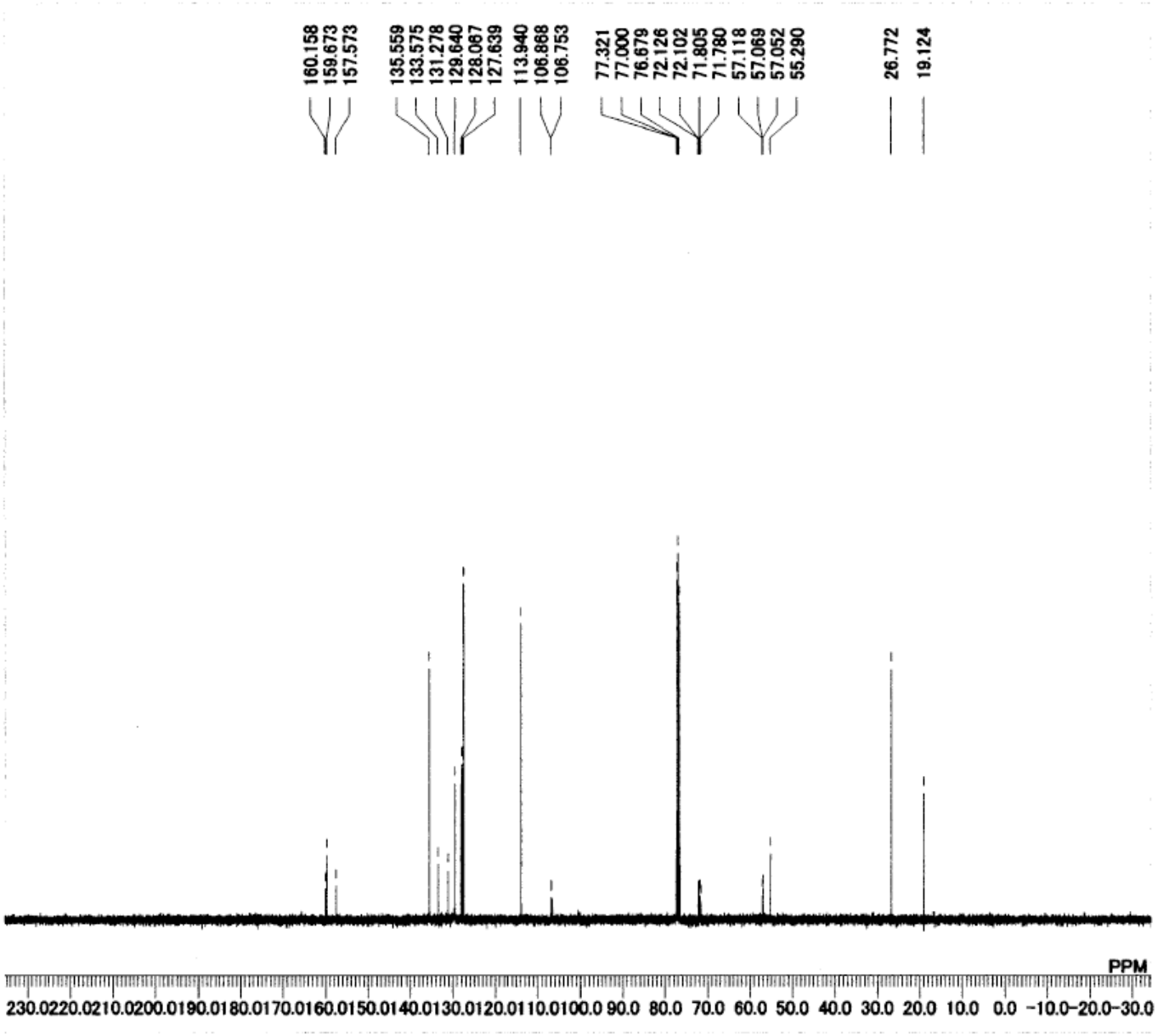




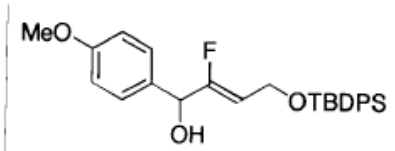


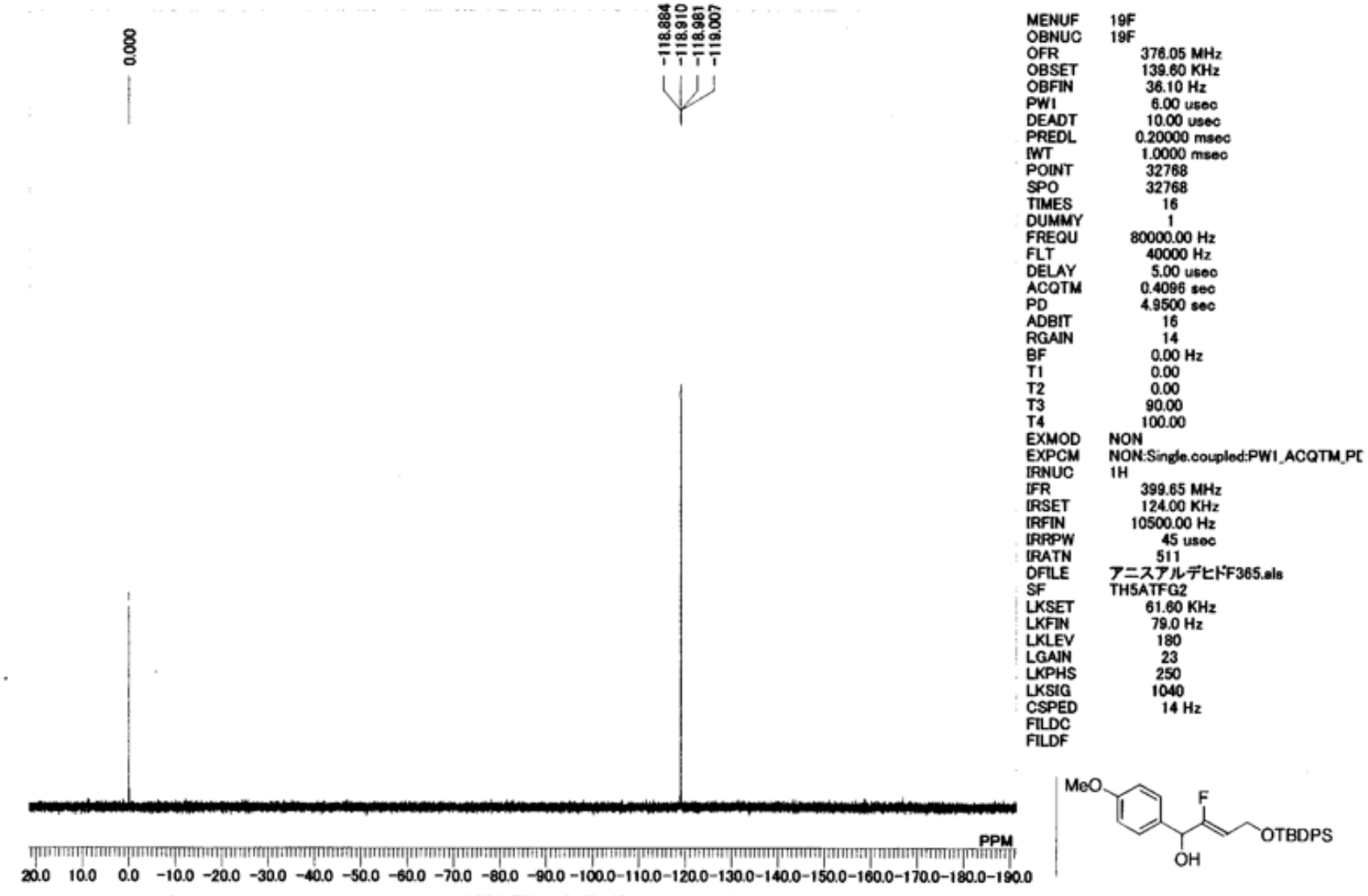
MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.80 usec
DEADT	72.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	18
BF	0.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	アニスアルデヒドH365 sai.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	23
LKPHS	250
LKSIG	1038
CSPED	11 Hz
FILDC	
FILDF	



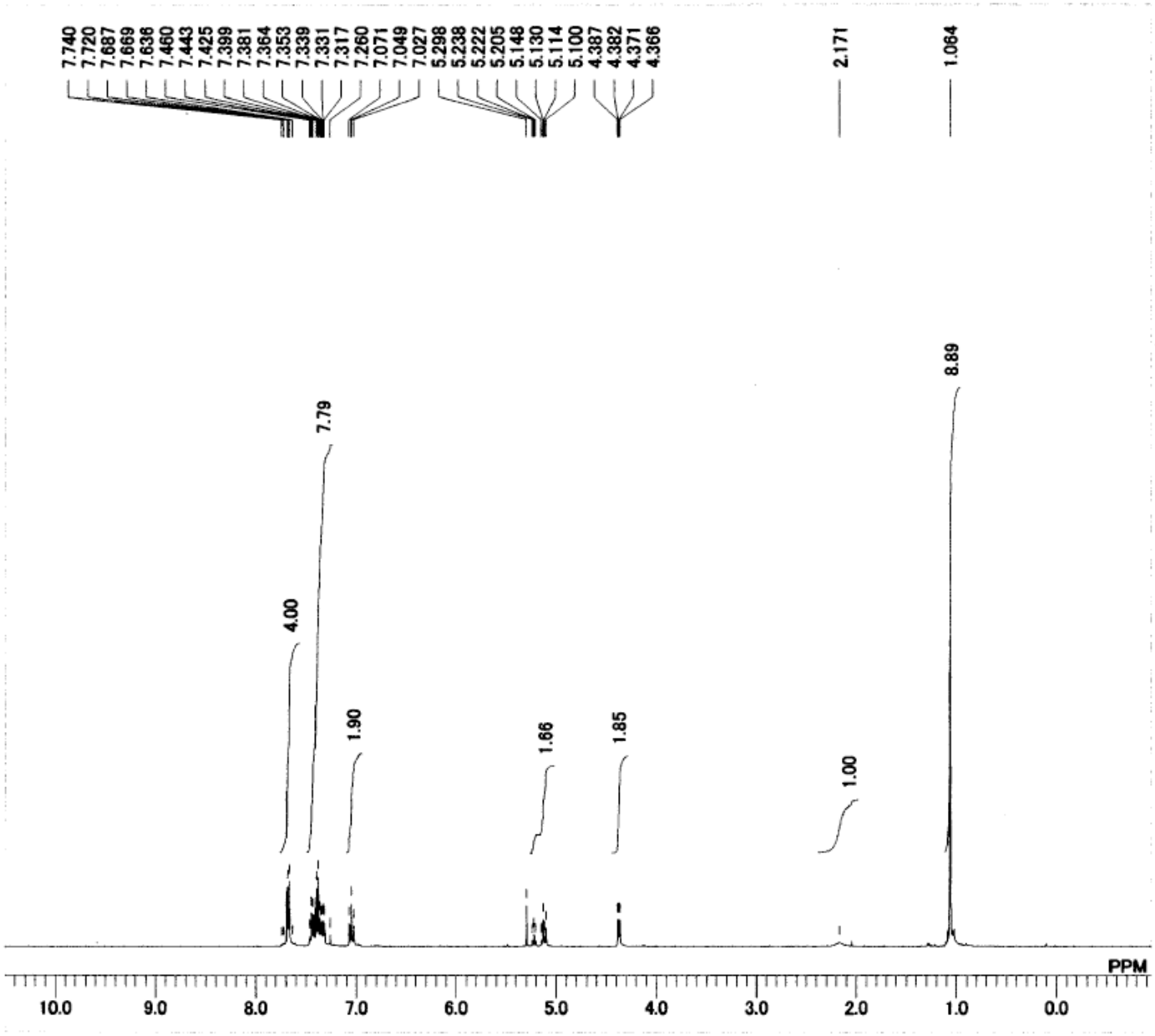


MENUF	13C
OBNUC	13C
OFR	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
PW1	6.20 usec
DEADT	19.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	1000000
DUMMY	1
FREQU	27118.64 Hz
FLT	13550 Hz
DELAY	14.80 usec
ACQTM	1.2083 sec
PD	1.7920 sec
ADBIT	16
RGAIN	26
BF	0.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRF
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	365 C.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	24
LKPHS	150
LKSIG	590
CSPED	13 Hz
FILDC	
FILDF	

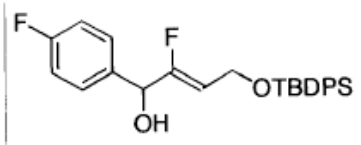


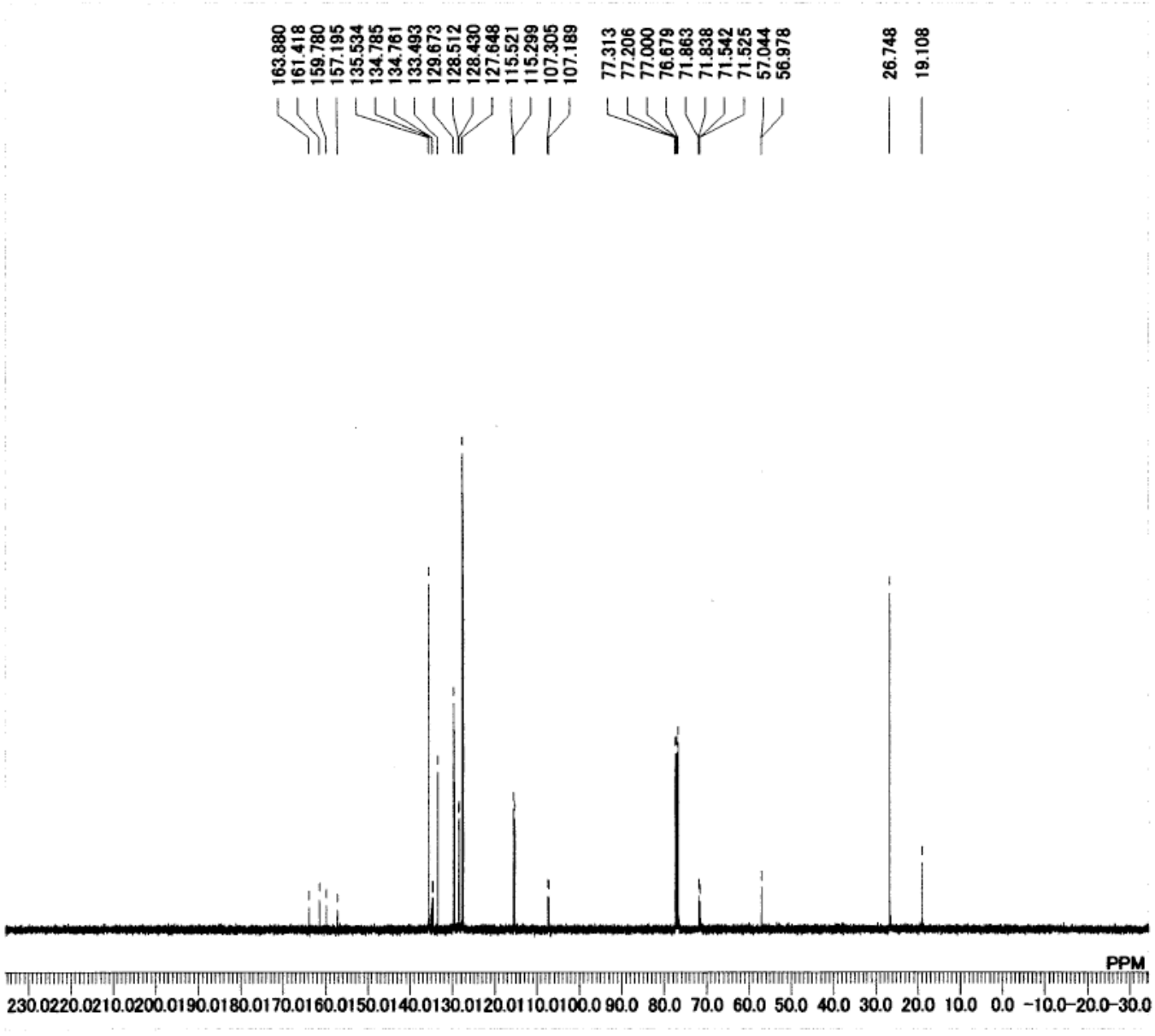


MENUF	19F
OBNUC	19F
OFR	376.05 MHz
OBSET	139.60 KHz
OBFIN	36.10 Hz
PW1	6.00 usec
DEADT	10.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	16
DUMMY	1
FREQU	80000.00 Hz
FLT	40000 Hz
DELAY	5.00 usec
ACQTM	0.4096 sec
PD	4.9500 sec
ADBIT	16
RGAIN	14
BF	0.00 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	アニスアルデヒドF365.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	23
LKPHS	250
LKSIG	1040
CSPED	14 Hz
FILDC	
FILDF	

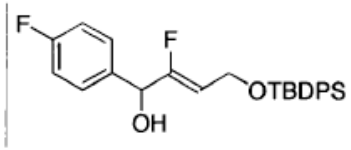


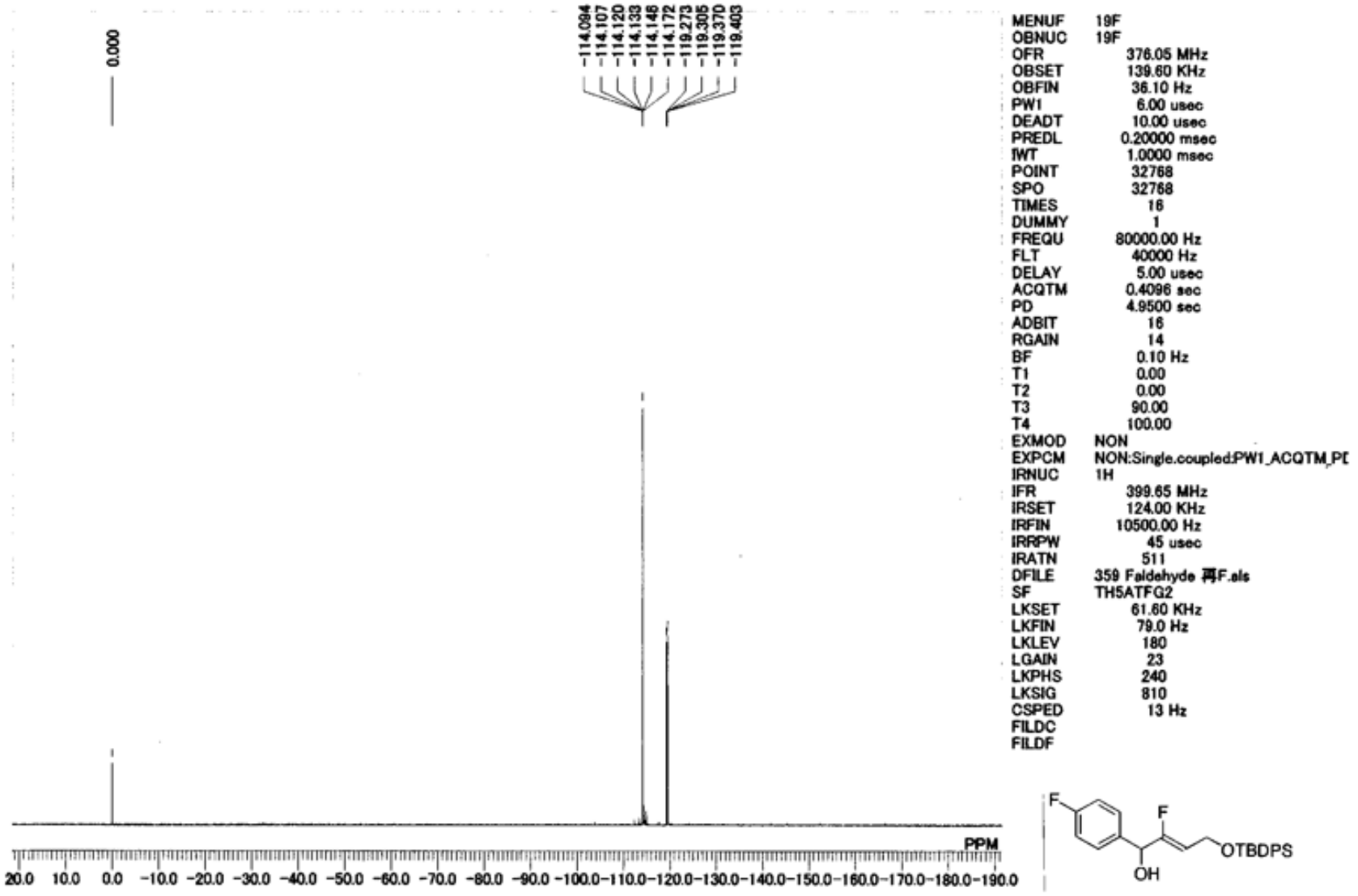
MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.50 usec
DEADT	72.20 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	12
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	359 Faldehyde 再H.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	23
LKPHS	240
LKSIG	808
CSPED	14 Hz
FILDC	
FILDF	

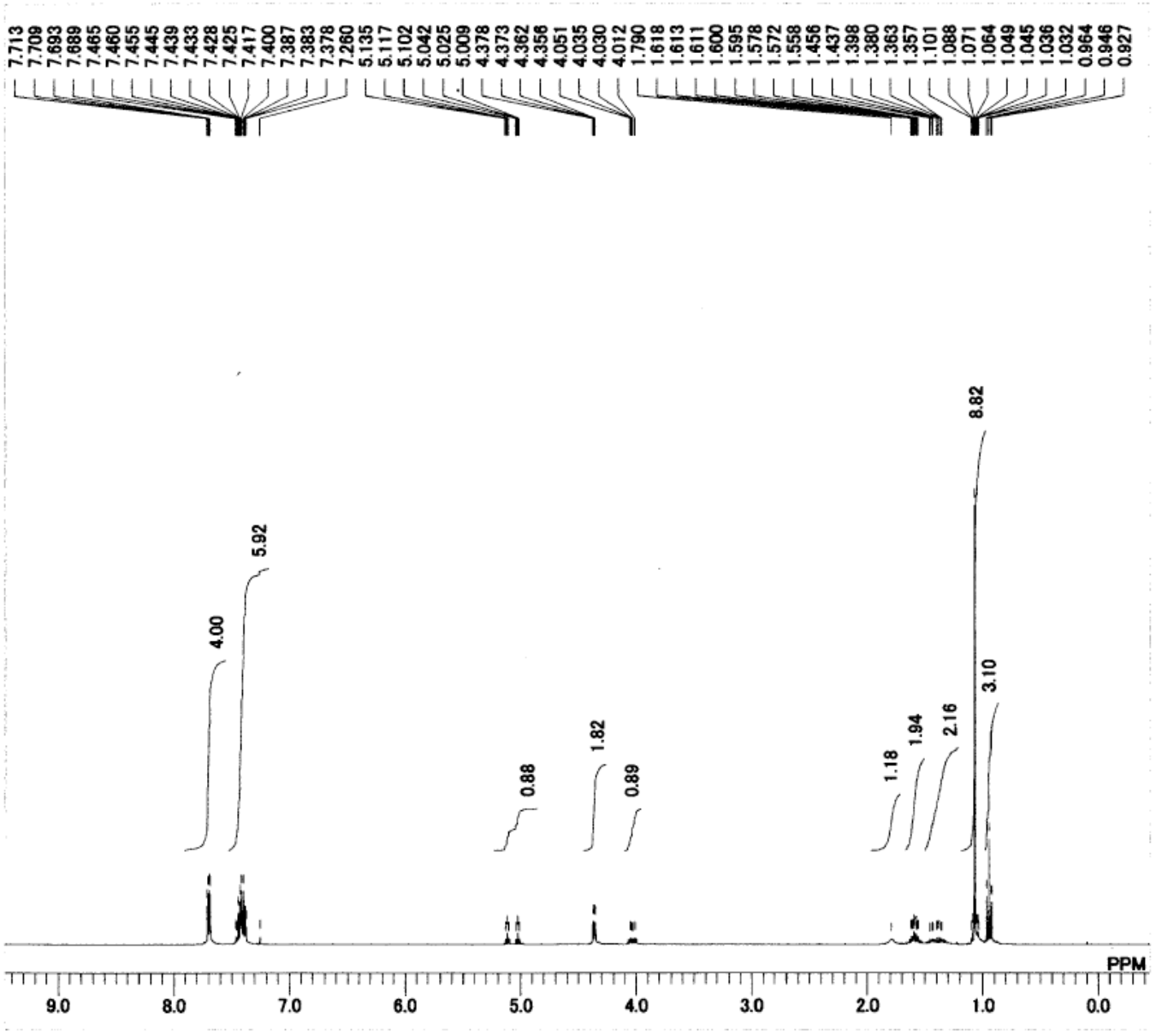




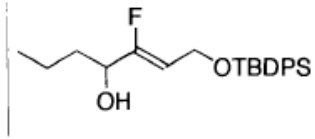
MENUF	13C
OBNUC	13C
OFR	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
PW1	6.20 usec
DEADT	19.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	20000
DUMMY	1
FREQU	27118.64 Hz
FLT	13550 Hz
DELAY	14.80 usec
ACQTM	1.2083 sec
PD	1.7920 sec
ADBIT	16
RGAIN	25
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRF
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	359 Faldehyde 再C.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	23
LKPHS	240
LKSIG	804
CSPED	13 Hz
FILDC	
FILDF	

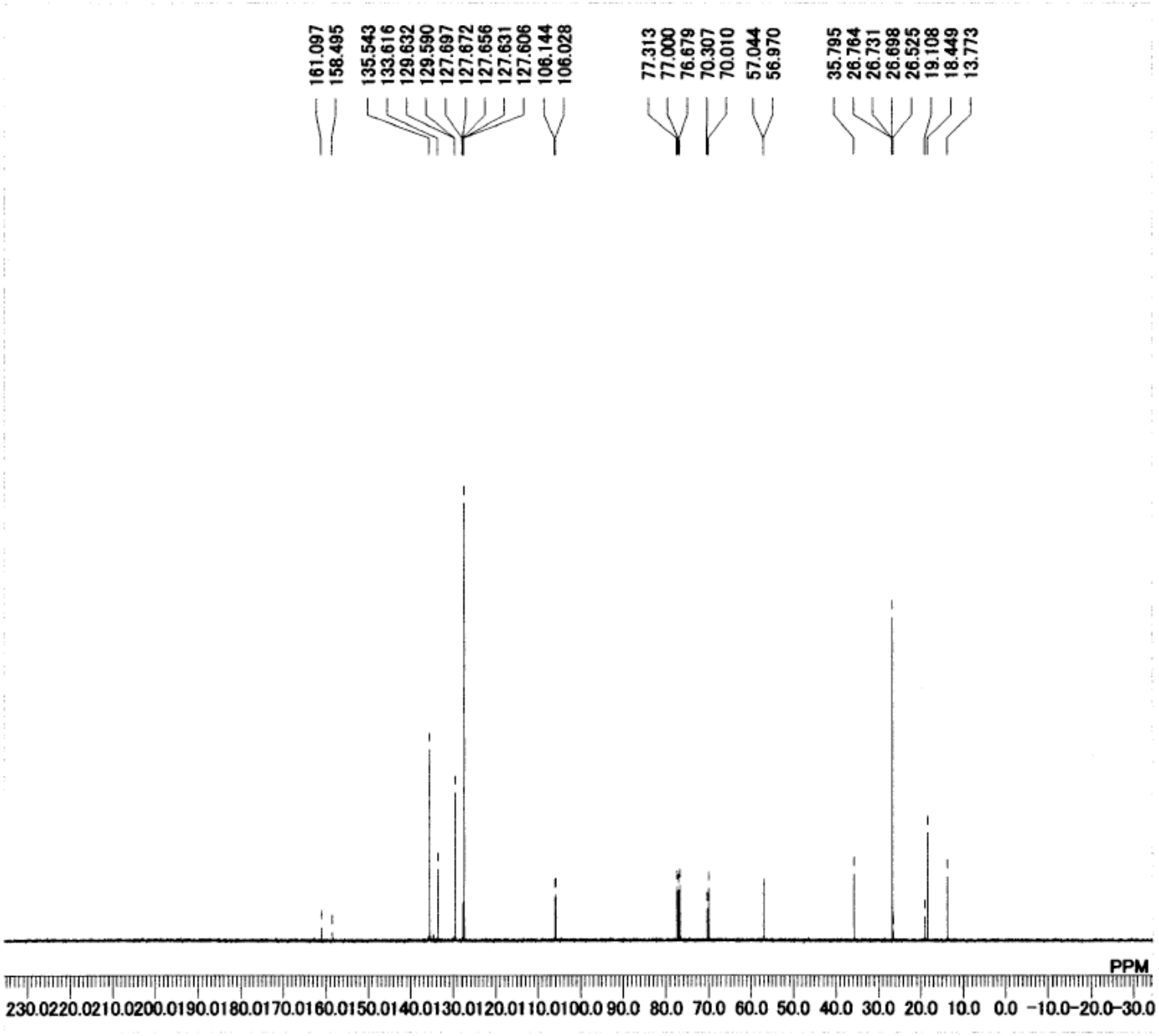




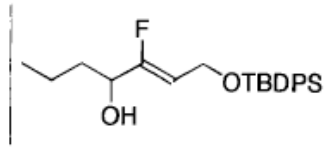


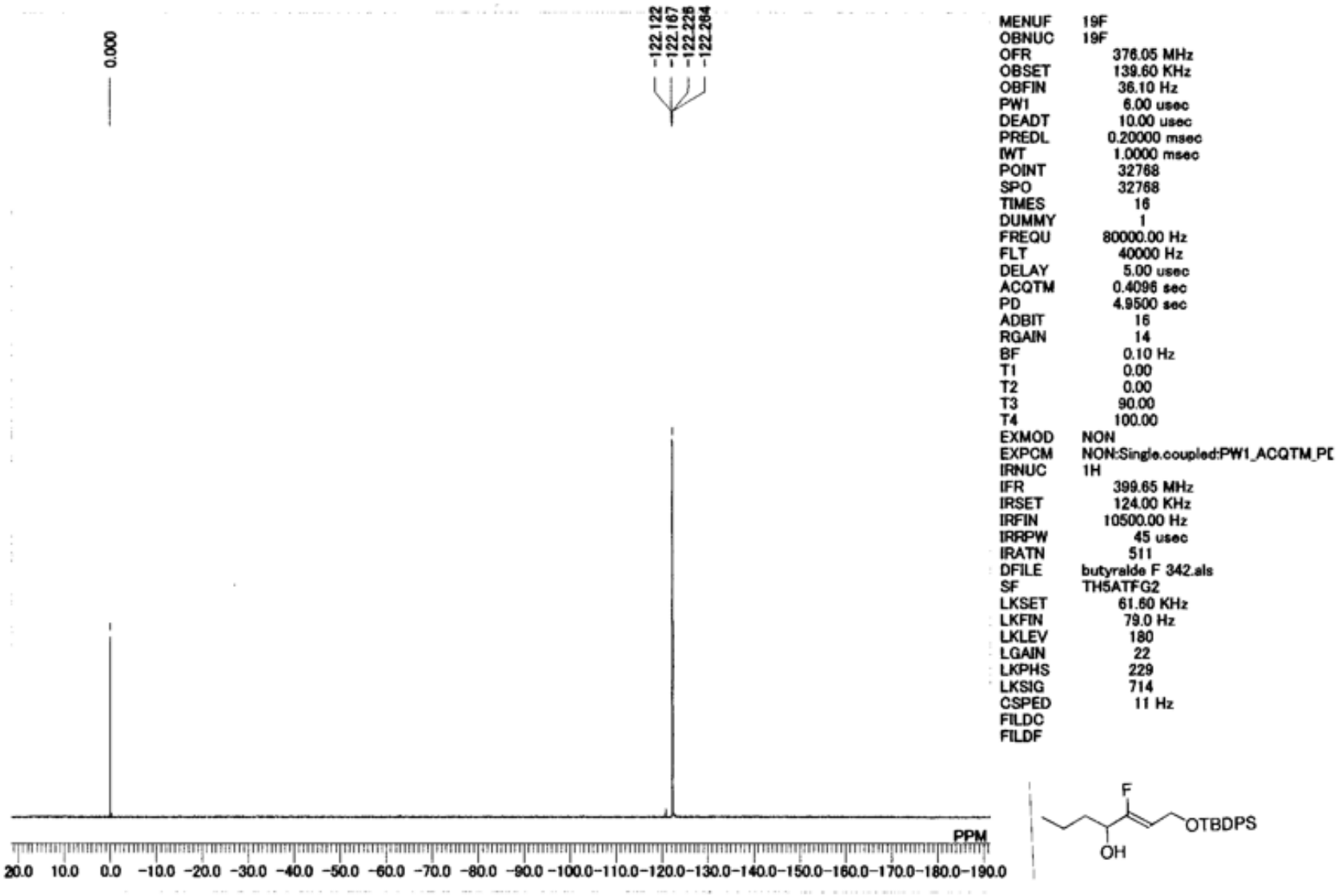
MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.80 usec
DEADT	72.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	10
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PI
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	butyralde H 342Vew.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	22
LKPHS	229
LKSIG	701
CSPED	12 Hz
FILDC	
FILDF	

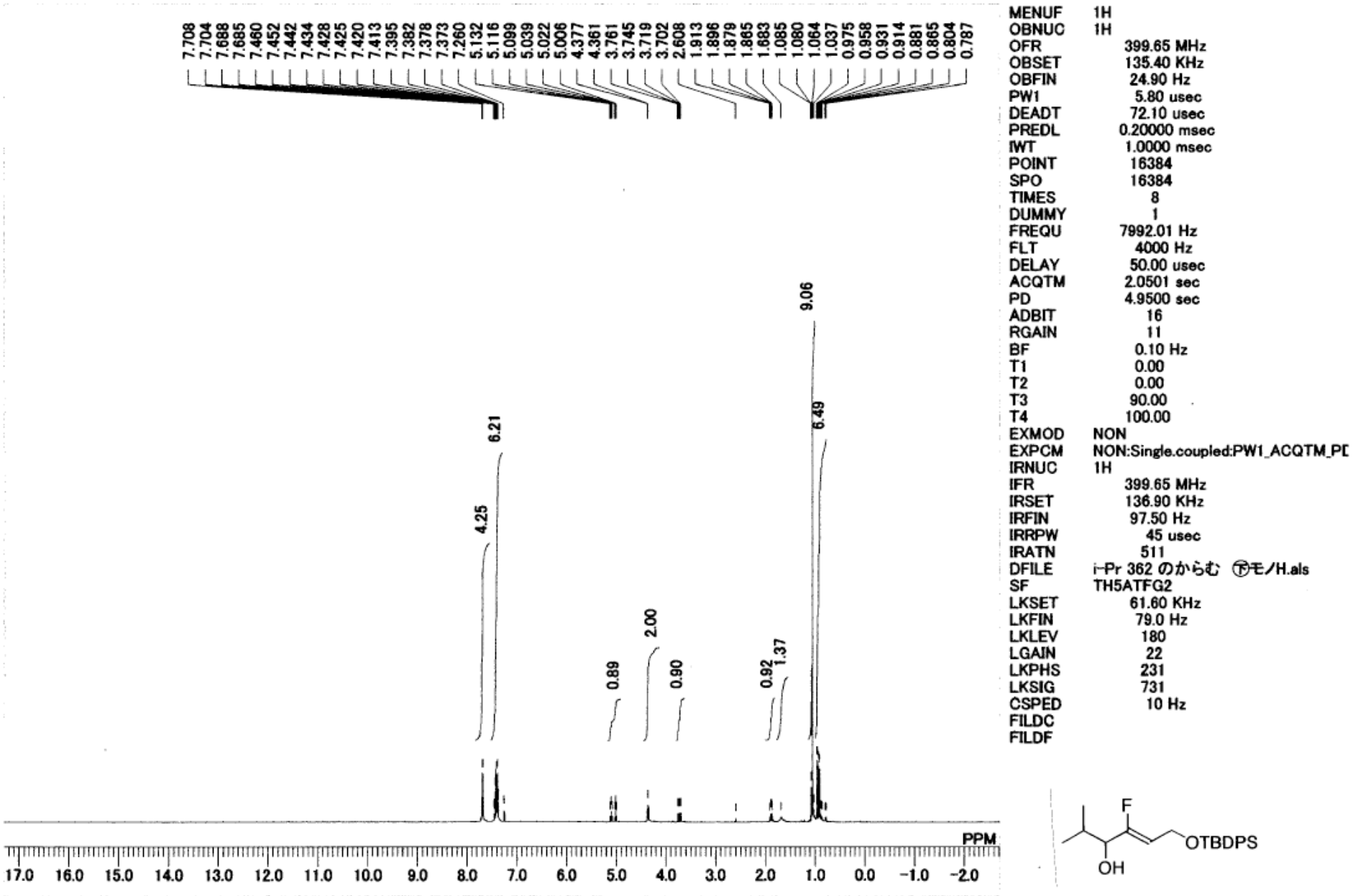


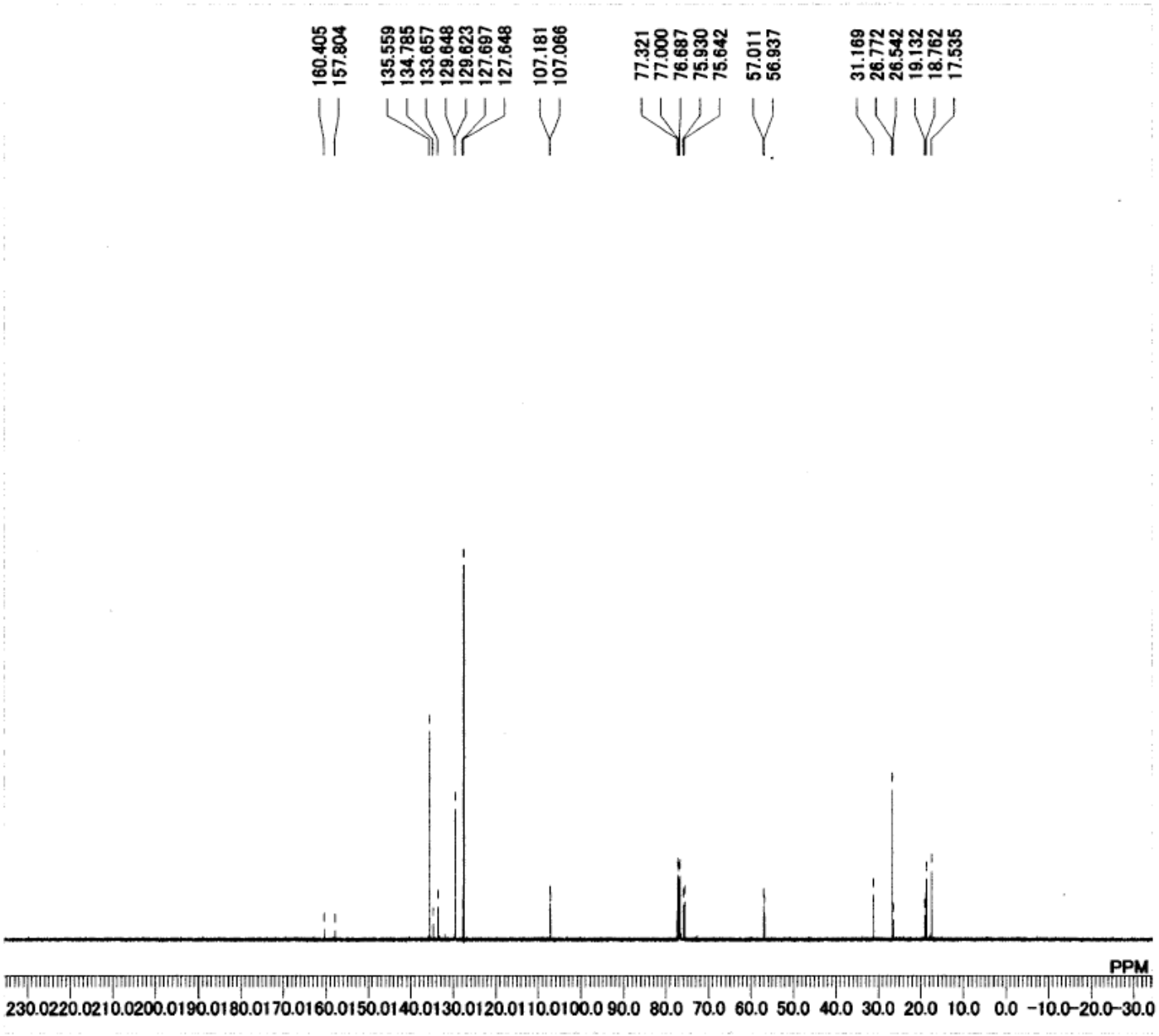


MENUF	13C
OBNUC	13C
OFR	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
PW1	6.00 usec
DEADT	19.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	256
DUMMY	1
FREQU	27118.64 Hz
FLT	13550 Hz
DELAY	14.80 usec
ACQTM	1.2083 sec
PD	1.7920 sec
ADBIT	16
RGAIN	24
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRF
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	butyralde C 342.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	22
LKPHS	229
LKSIG	687
CSPED	12 Hz
FILDC	
FILDF	

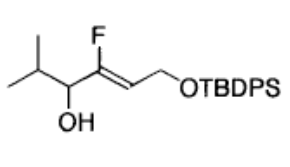


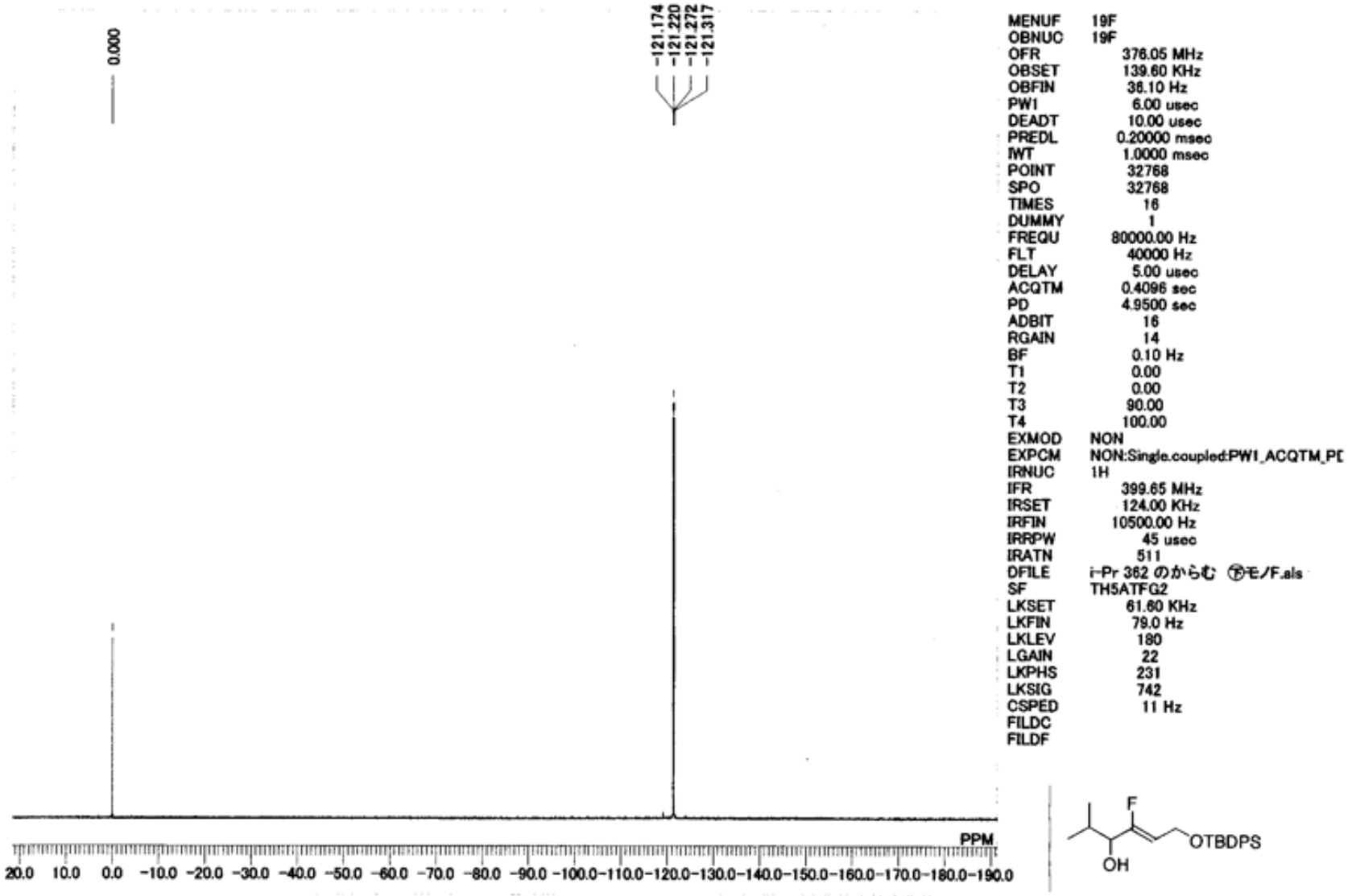


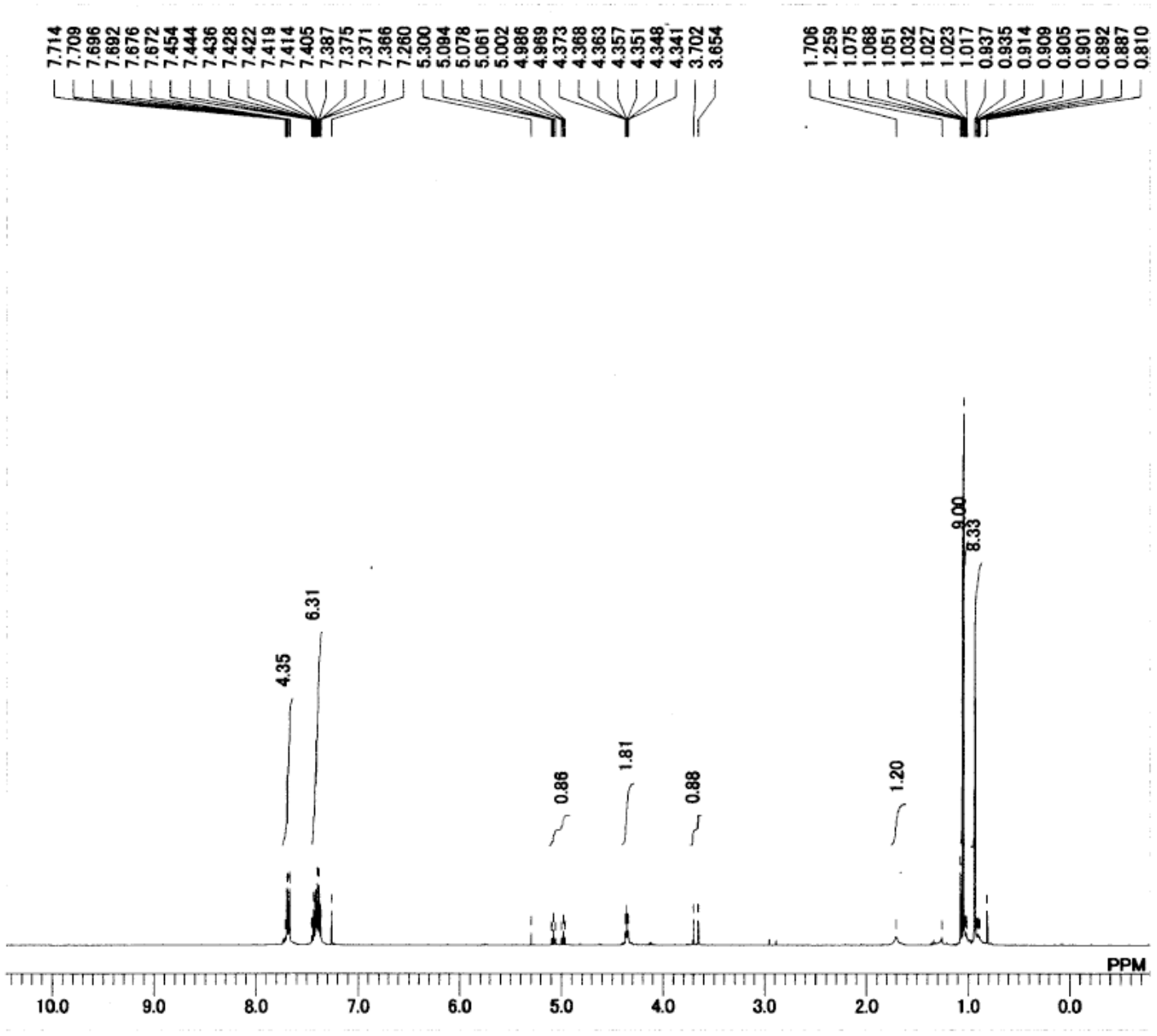




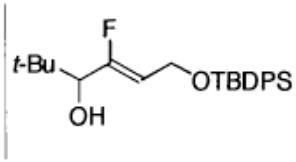
MENUF	13C
OBNUC	13C
OFR	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
PW1	6.00 usec
DEADT	19.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	256
DUMMY	1
FREQU	27118.64 Hz
FLT	13550 Hz
DELAY	14.80 usec
ACQTM	1.2083 sec
PD	1.7920 sec
ADBIT	16
RGAIN	25
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRF
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRPW	45 usec
IRATN	511
DFILE	i-Pr 362 のからむ ㊦モノC.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	22
LKPHS	231
LKSIG	719
CSPED	10 Hz
FILDC	
FILDF	

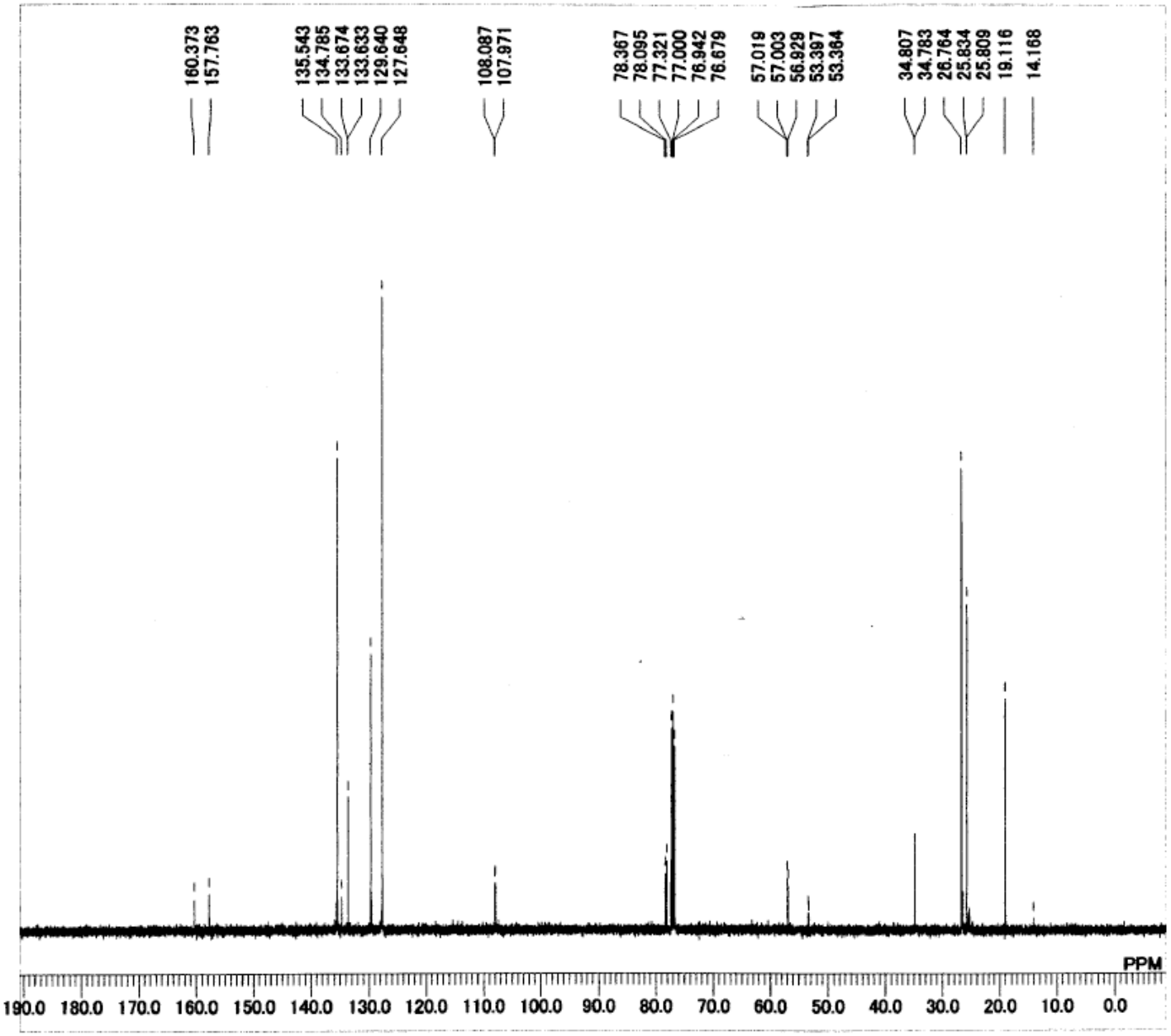




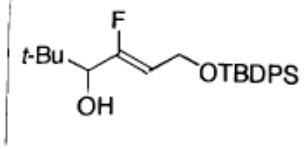


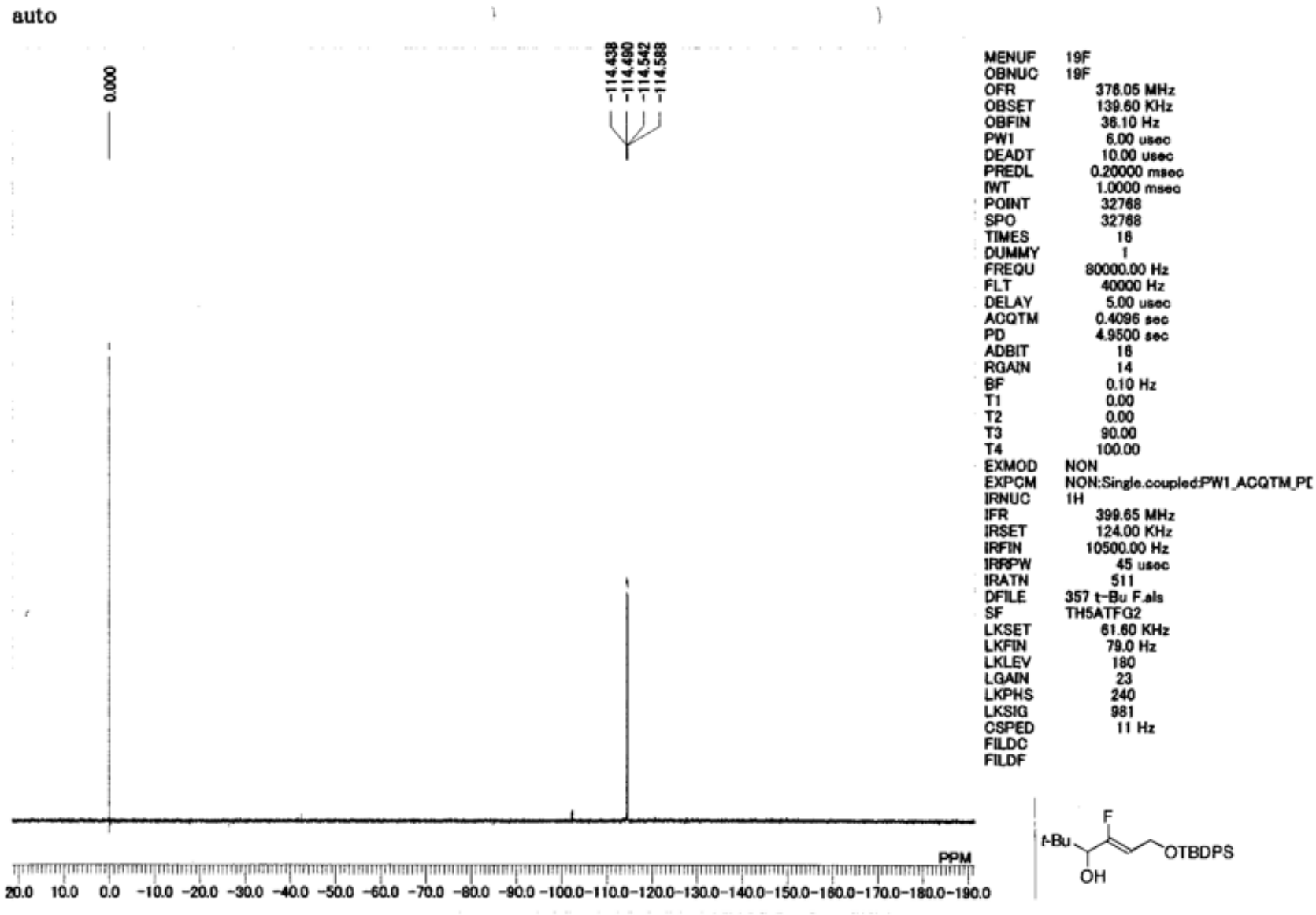
MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.50 usec
DEADT	72.20 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	15
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	357 t-Bu H.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	23
LKPHS	240
LKSIG	978
CSPED	11 Hz
FILDC	
FILDF	

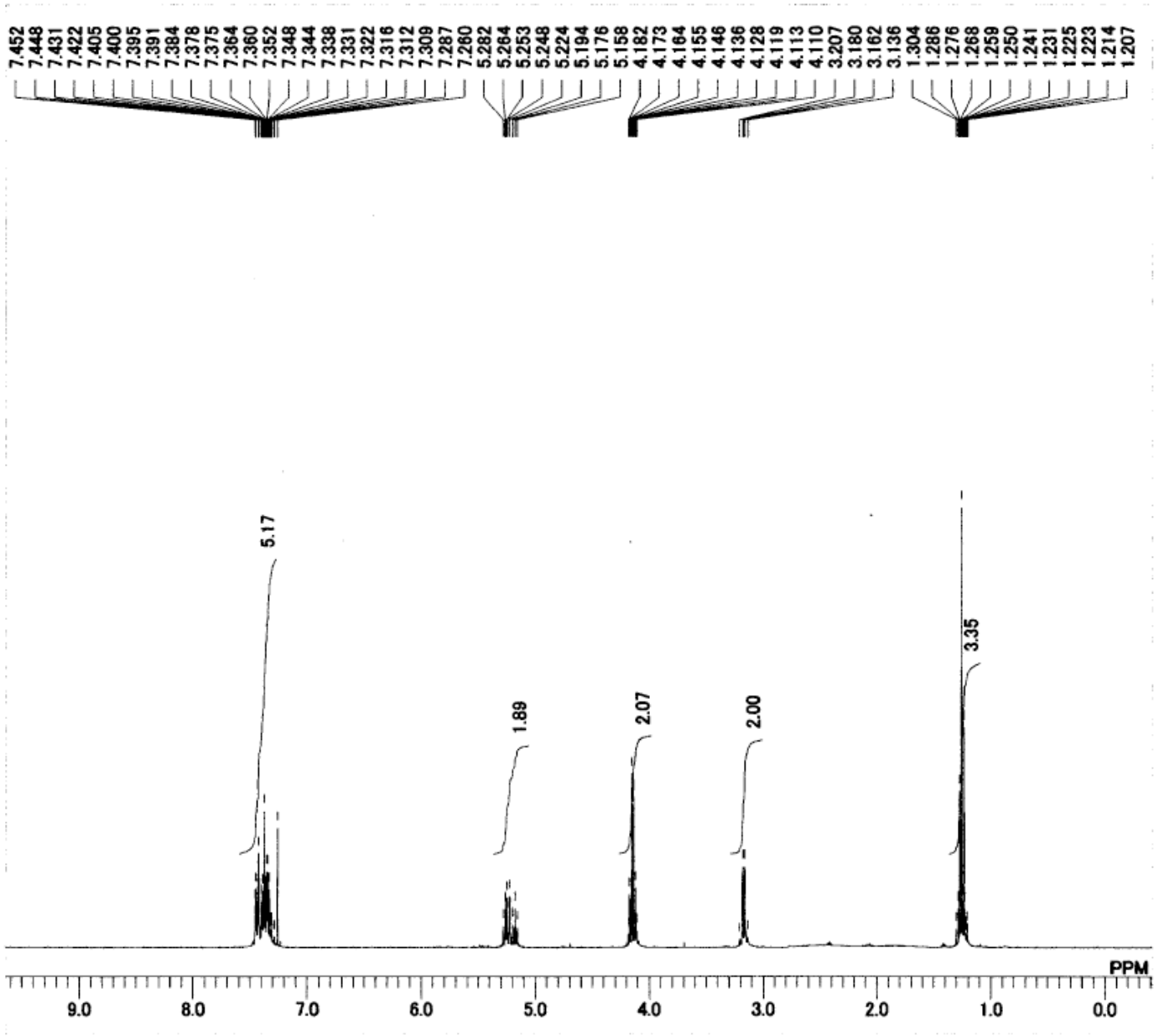




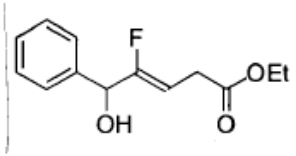
MENUF	13C
OBNUC	13C
OFR	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
PW1	6.20 usec
DEADT	19.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	20000
DUMMY	1
FREQU	27118.64 Hz
FLT	13550 Hz
DELAY	14.80 usec
ACQTM	1.2083 sec
PD	1.7920 sec
ADBIT	16
RGAIN	25
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRF
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	382 t-Bu TBDPS 13Cals.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	23
LKPHS	240
LKSIG	915
CSPED	10 Hz
FILDC	
FILDF	

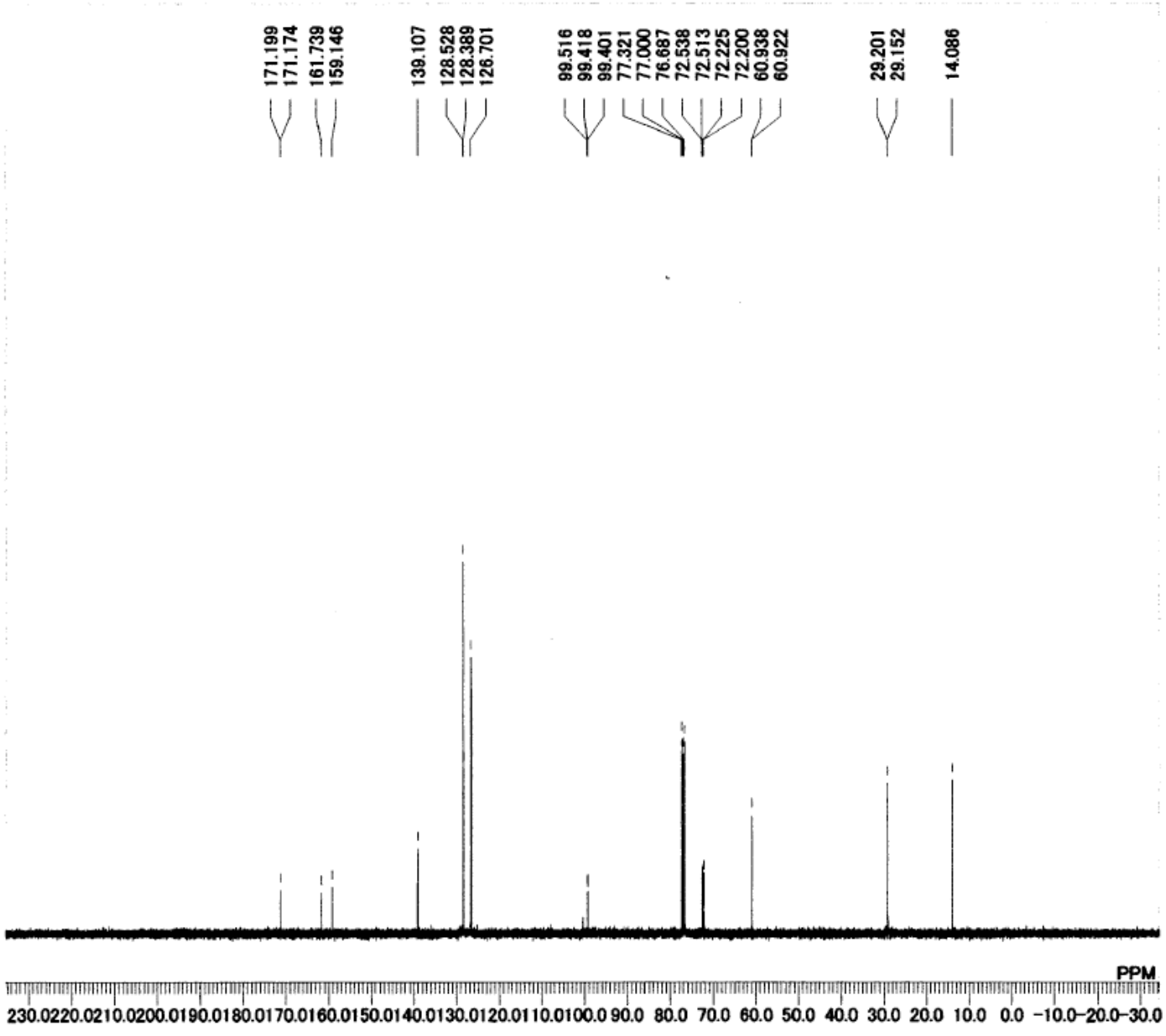




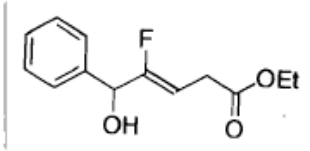


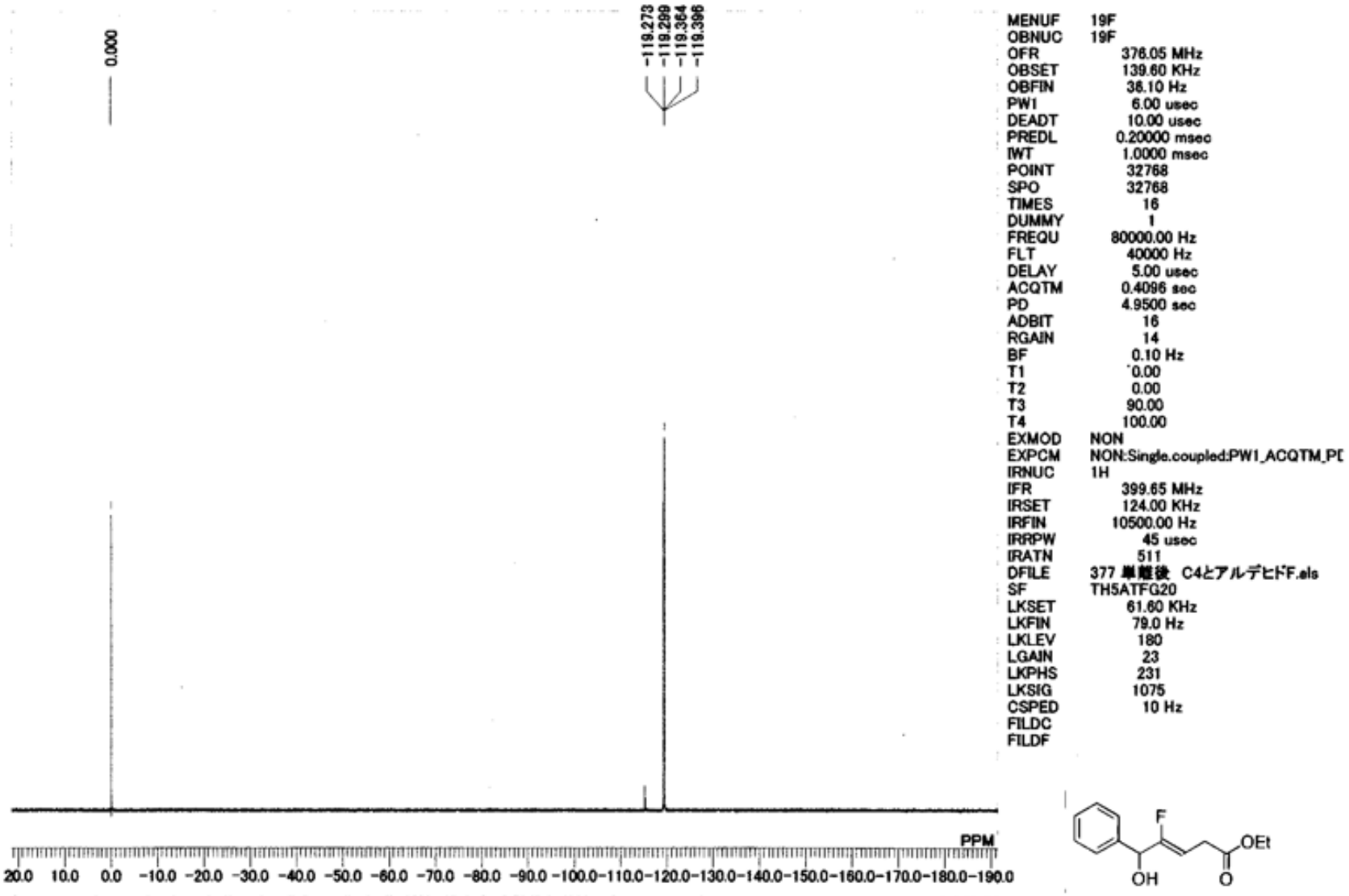
MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.80 usec
DEADT	72.10 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	16
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	377 単離後 C4とアルデヒドH.als
SF	TH5ATFG20
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	23
LKPHS	231
LKSIG	1073
CSPED	11 Hz
FILDC	
FILDF	

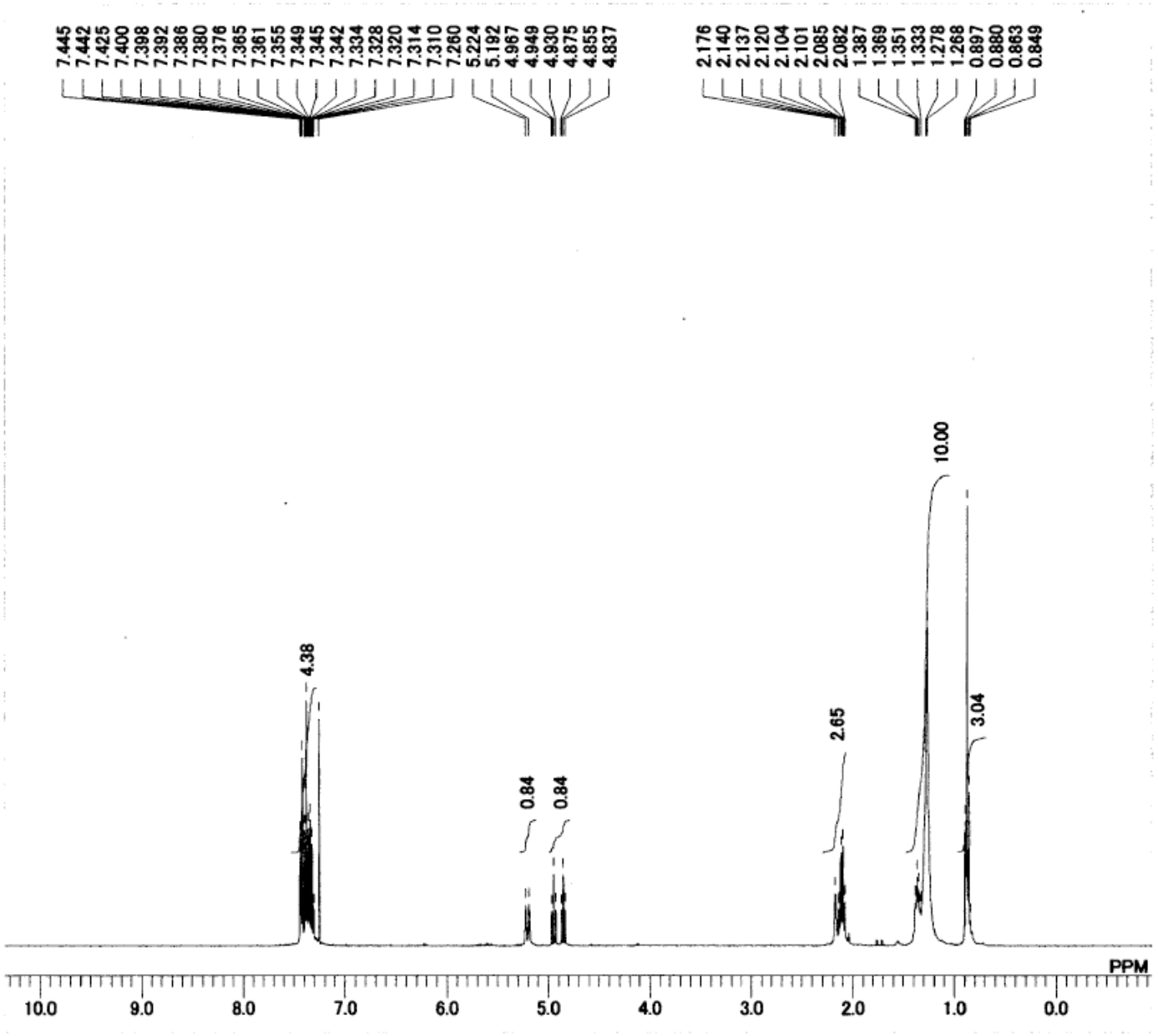




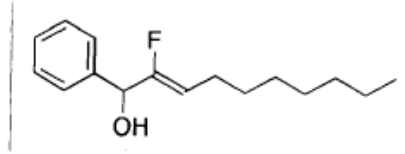
MENUF	13C
OBNUC	13C
OFR	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
PW1	6.20 usec
DEADT	19.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	10000
DUMMY	1
FREQU	27118.64 Hz
FLT	13550 Hz
DELAY	14.80 usec
ACQTM	1.2083 sec
PD	1.7920 sec
ADBIT	16
RGAIN	25
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRF
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	377 単離後 C4とアルデヒド C.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	26
LKPHS	150
LKSIG	968
CSPED	11 Hz
FILDC	
FILDF	

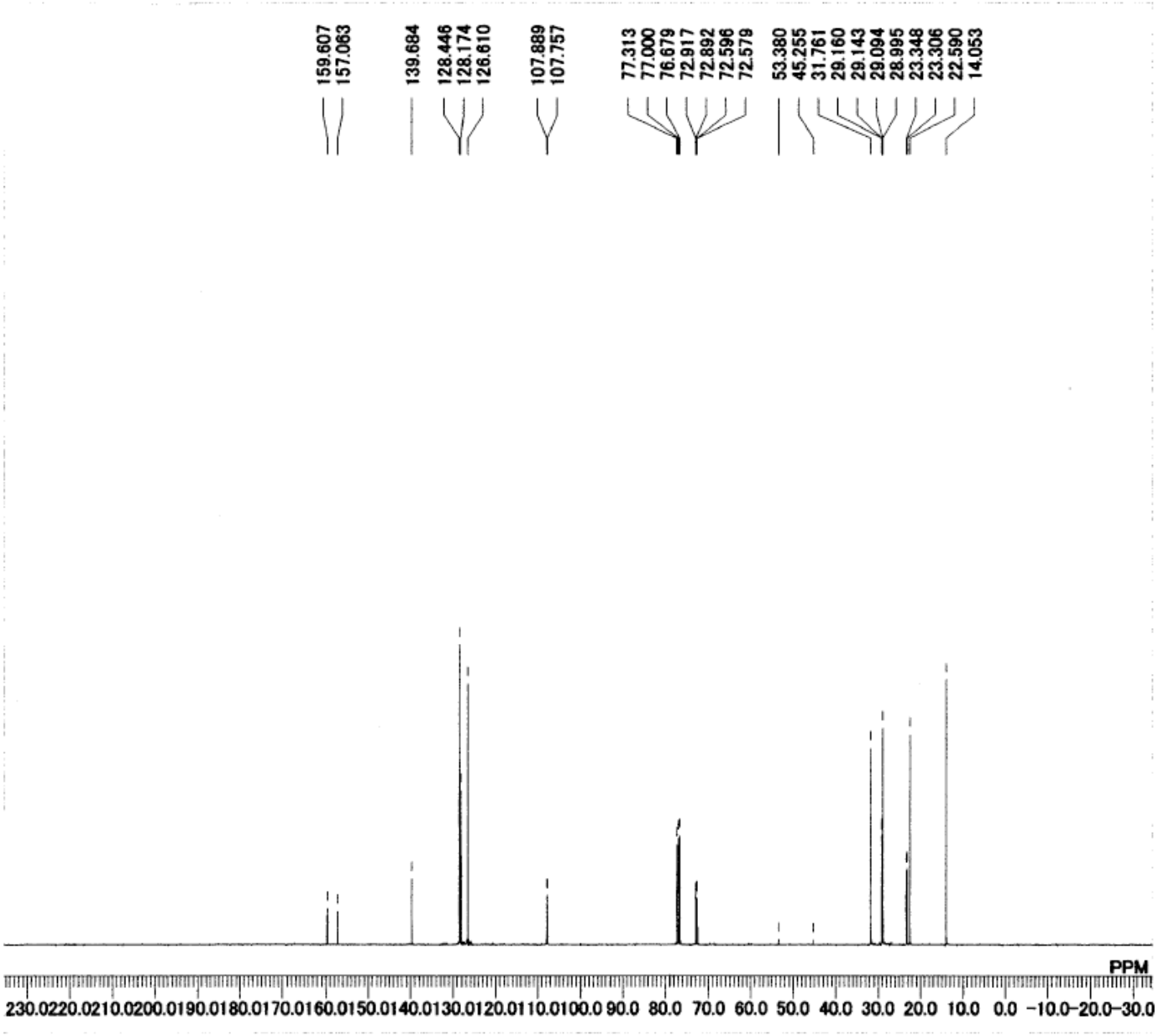




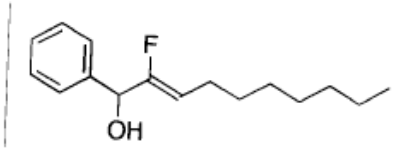


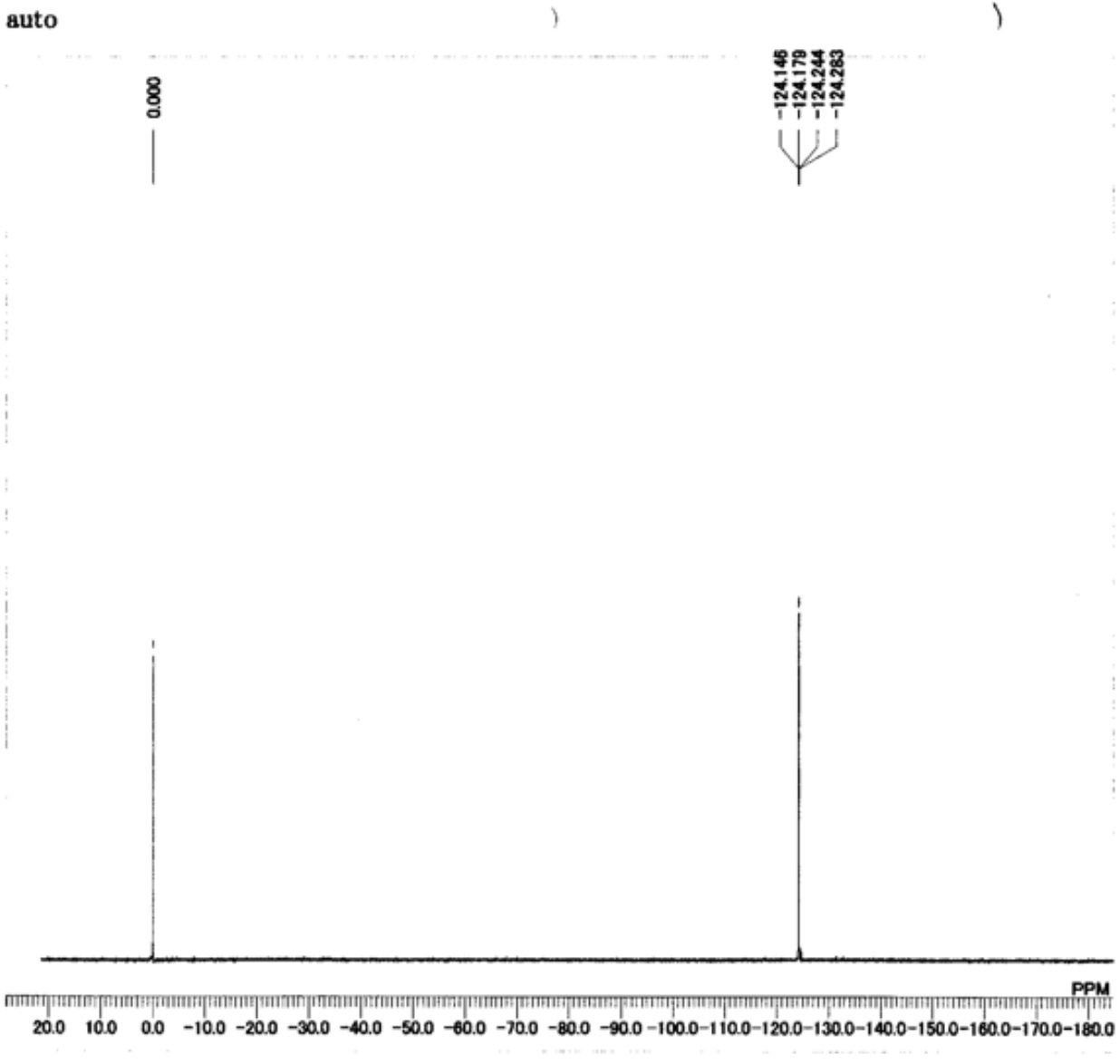
MENUF	1H
OBNUC	1H
OFR	399.65 MHz
OBSET	135.40 KHz
OBFIN	24.90 Hz
PW1	5.50 usec
DEADT	72.20 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	16384
SPO	16384
TIMES	8
DUMMY	1
FREQU	7992.01 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	2.0501 sec
PD	4.9500 sec
ADBIT	16
RGAIN	16
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD
IRNUC	1H
IFR	399.65 MHz
IRSET	136.90 KHz
IRFIN	97.50 Hz
IRRPW	45 usec
IRATN	511
DFILE	C9 とベンズアルデヒド H.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	22
LKPHS	240
LKSIG	820
CSPED	12 Hz
FILDC	
FILDF	



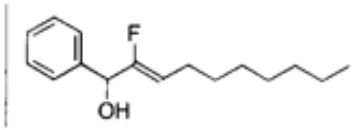


MENUF	13C
OBNUC	13C
OFR	100.40 MHz
OBSET	125.00 KHz
OBFIN	10500.00 Hz
PW1	6.20 usec
DEADT	19.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	20000
DUMMY	1
FREQU	27118.64 Hz
FLT	13550 Hz
DELAY	14.80 usec
ACQTM	1.2083 sec
PD	1.7920 sec
ADBIT	16
RGAIN	25
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	BCM
EXPCM	Bilevel.complete.decoupling:Set_IRRF
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRRPW	45 usec
IRATN	511
DFILE	_DEFAULT.ALS
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	24
LKPHS	240
LKSIG	1243
CSPED	11 Hz
FILDC	
FILDF	

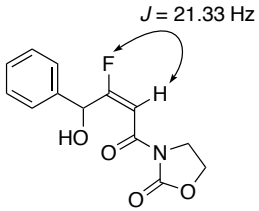




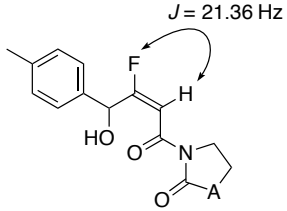
MENUF	19F
OBNUC	19F
OFR	376.05 MHz
OBSET	139.60 KHz
OBFIN	36.10 Hz
PW1	6.00 usec
DEADT	10.00 usec
PREDL	0.20000 msec
IWT	1.0000 msec
POINT	32768
SPO	32768
TIMES	16
DUMMY	1
FREQU	80000.00 Hz
FLT	40000 Hz
DELAY	5.00 usec
ACQTM	0.4096 sec
PD	4.9500 sec
ADBIT	16
RGAIN	14
BF	0.10 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	NON
EXPCM	NON:Single.coupled:PW1_ACQTM_PD
IRNUC	1H
IFR	399.65 MHz
IRSET	124.00 KHz
IRFIN	10500.00 Hz
IRPW	45 usec
IRATN	511
DFILE	C9 とベンズアルデヒド F.als
SF	TH5ATFG2
LKSET	61.60 KHz
LKFIN	79.0 Hz
LKLEV	180
LGAIN	22
LKPHS	240
LKSIG	819
CSPED	12 Hz
FILDC	
FILDF	



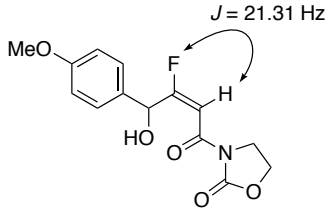
*Determination of the Stereochemistry for **2A***



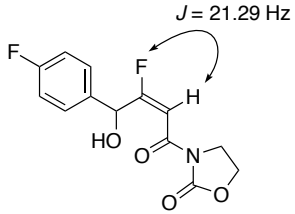
2Aa : 89% (84%)
E/*Z* = 100/0



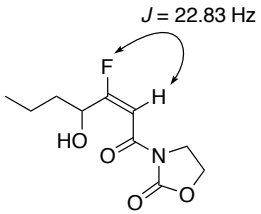
2Ab : 77% (64%)
E/*Z* = 100/0



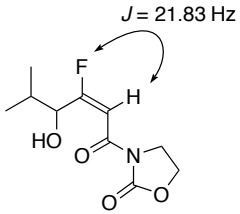
2Ac : 73% (69%)
E/*Z* = 100/0



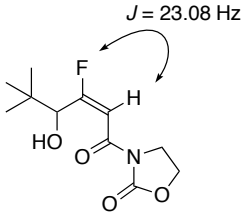
2Ad : 94% (90%)
E/*Z* = 100/0



2Af : 70% (68%)
E/*Z* = 100/0

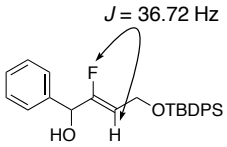


2Ag : 91% (77%)
E/*Z* = 100/0

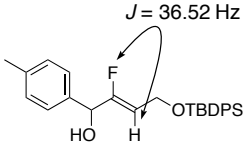


2Ah : 88% (77%)
E/*Z* = 100/0

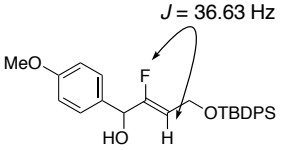
*Determination of the Stereochemistry for **2B***



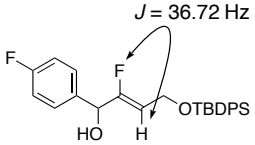
2Ba : 68% (56%)
E/*Z* = 0/100



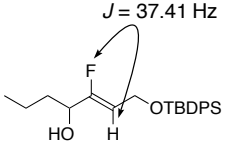
2Bb : 64% (48%)
E/*Z* = 0/100



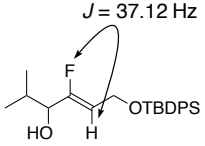
2Bc : 78% (52%)
E/*Z* = 0/100



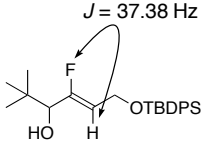
2Bd : 64% (56%)
E/*Z* = 0/100



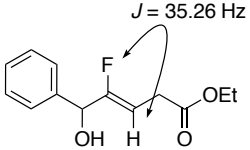
2Bf : 79% (57%)
E/*Z* = 1/99



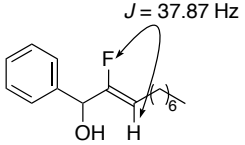
2Bg : 77% (59%)
E/*Z* = 1/99



2Bh : 78% (63%)
E/*Z* = 0/100



2Da : 76% (55%
E/*Z* = 2/98



2Ea : 94% (76%
E/*Z* = 2/98