Supporting Information

A New Cobalt-Salen Catalyst for Asymmetric Cyclopropanation. Synthesis of the Serotonin-Norepinephrine Reuptake Inhibitor (+)-Synosutine

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Experimental Section

General

Starting materials and reagents were obtained from commercial sources and were used without further purification. Solvents were dried by distillation from the appropriate drying reagents immediately prior to use. All solvents used for routine isolation of products and chromatography were reagent grade. Moisture- and air-sensitive reactions were carried out under an atmosphere of argon. Reaction flasks were flame dried under a stream of argon gas, and glass syringes were oven dried at 120 °C prior to use.

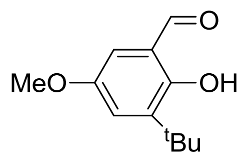
Unless otherwise stated, concentration under reduced pressure refers to a rotary evaporator at water aspirator pressure. Residual solvent was removed by vacuum pump at a pressure less than 0.25 mm of mercury.

Analytical thin-layer chromatography (TLC) was conducted using precoated TLC plates (0.2 mm layer thickness of silica gel 60 F-254). Compounds were visualized by ultraviolet light and/or by heating the plate after dipping in a 3-5% solution of phosphomolybdic acid in ethanol, 10% ammonium molybdate in water, a 1% solution of vanillin in 0.1 M sulfuric acid in methanol or 2.5% *p*-anisaldehyde in 88% ethanol, 5% water, 3.5% concentrated sulfuric acid and 1% acetic acid. Flash chromatography was performed with the indicated eluents on 230 - 400 mesh silica gel.

Optical rotations were measured with a polarimeter at ambient temperature using a 0.9998 dm cell with 1 mL capacity. Infrared (IR) spectra were recorded on a FT-IR spectrometer. Proton and carbon nuclear magnetic resonance (NMR) spectra were obtained using either a 400, 500 or 700 MHz spectrometer. All chemical shifts were reported in parts per million (ppm) downfield from tetramethylsilane using the δ scale. ^{1}H NMR spectral data are reported in the order: chemical shift, multiplicity (s = singlet, d = doublet, m = multiplet, and b = broad), coupling constant (J, in Hertz), and number of protons.

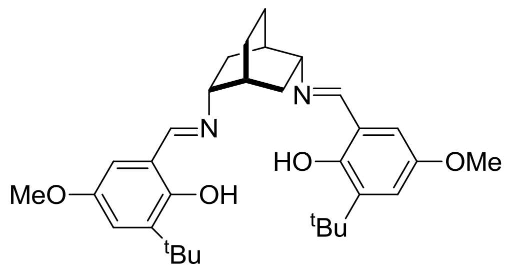
Low (MS) and high (HRMS) resolution mass spectra are reported with ion mass/charge (*m/z*) ratios as values in atomic mass units. α-Methylstyrenes were prepared following literature method.[1]

3-(*tert*-Butyl)-2-hydroxy-5-methoxybenzaldehyde (10)



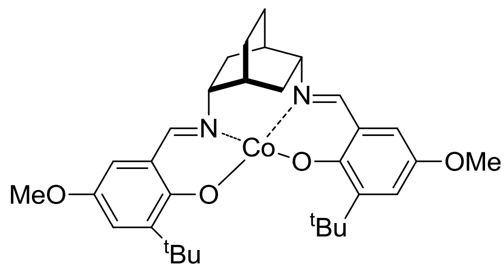
To a solution of 2-(*tert*-butyl)-4-methoxyphenol (**9**, 721 mg, 4 mmol) in CH_3CN (20 mL) at room temperature were added Et_3N (2.8 mL, 20 mmol) and $MgCl_2$ (456 mg, 4.8 mmol) and the mixture was stirred for 15 min. Paraformaldehyde (600 mg, 20 mmol) was added and the solution was refluxed for 10 h. The solution was cooled to room temperature, poured into 1M aqueous HCl (100 mL) and stirred for 30 min at room temperature. The reaction mixture was extracted with ether (4 x 100 mL) and the organic layer was washed with brine (20 mL), dried (Na_2SO_4) and concentrated under reduced pressure. The crude residue was purified by flash chromatography on silica gel (5% ether/hexanes) to obtain **10** (609 mg, 73%) as a yellow oil: IR (neat) 3534, 3301, 3062, 2925, 2854, 1622, 1598, 1508, 1472, 1422, 1378, 1206, 1149, 1967, 1030, 854, 836, 814, 749, 693 cm^{-1}; ^{1}H NMR (700 MHz, $CDCl_3$) δ 1.42 (s, 9H), 3.04 (s, 3H), 7.40 (s, 1H), 7.62 (s, 1H), 9.91 (s, 1H), 11.68 (s, 1H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 29.7, 32.9, 54.7, 113.4, 119.3, 129.8, 140.3, 150.5, 152.1, 207.4.

(+)-2,2'-[(1*R*,2*R*,4*R*,5*R*)-Bicyclo[2.2.2]octane-2,5-diylbis(nitrilomethylidine)]bis-2-*tert*-butyl-4-methoxylphenol [(+)-11]



To a solution of (-)-**1** (86 mg, 0.61 mmol) in EtOH (15 mL) was added anhydrous $MgSO_4$ (367 mg, 3.05 mmol) followed by a solution of **10** (256 mg 1.23 mmol) in EtOH (5 mL). The suspension was refluxed for 4 h at which time a yellow precipitate had formed. The mixture was cooled to room temperature and the precipitate was filtered off. The crude solid was purified by flash chromatography on silica gel (5% EtOAc/hexanes) to give (+)-**11** (293 mg, 92%) as an amorphous yellow solid: mp 162 - 163 °C; $[\alpha]^{25}_{D}$ + 96.6 (*c* 0.5 , CH_2Cl_2); IR (neat) 3358 (b), 1744, 1467, 1375, 1286, 1169, 1137, 1030, 891 cm^{-1}; ^{1}H NMR (700 MHz, $CDCl_3$) δ 1.38 (s, 9H), 1.73 (s, 3H), 1.91 - 2.01 (m, 1H), 2.12 - 2.24 (m, 1H) , 3.53 - 3.57 (m, 1H), 3.92 (s, 3H), 7.12 (d, *J* = 2.4 Hz, 1H), 7.38 (d, *J* = 2.4 Hz, 1H), 8.43 (s, 1H), 13.86 (s, 1H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 23.9, 31.3, 31.6, 33.9, 34.2, 57.7, 67.5, 118.4, 128.6, 128.8, 137.1, 139.7, 159.0, 165.6; HRMS (EI) calcd for $C_{32}H_{44}N_2O_4$ *m/z* 520.7131, found 520.7128.

(+)-(1*R*,2*R*,4*R*,5*R*)-*N*,*N*'-Bis-(3-*tert*-butyl-5-methoxylsalicylidene)-2,5-diaminobicyclo[2.2.2]octane Cobalt(II) [(+)-12]



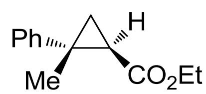
To a solution of (+)-**11** (290 mg, 0.56 mmol) in EtOH (15 mL) was added a solution of $Co(OAc)_2$ (99 mg, 0.56 mmol) in EtOH (2 mL) and the mixture was heated at reflux for 6 h, at which time an orange precipitate had formed. The mixture was cooled to room temperature and concentrated under reduced pressure. The crude residue was dissolved in CH_2Cl_2 and filtered. Concentration of the filtrate under vacuum provided (+)-**12** (324 mg, 94%) as an orange solid: mp > 260 °C; $[\alpha]^{25}_{D}$ + 86.0 (*c* 0.26 $CHCl_3$); IR (neat) 2949, 2859, 1606, 1594, 1548, 1528, 1458, 1411, 1361, 1314, 1252, 1178, 1108, 1084, 1011, 960, 867, 839 cm^{-1}; HRMS (EI) calcd for $C_{32}H_{42}CoN_2O_4$ *m/z* 577.4733, found 577.4716.

Representative Procedure for the Asymmetric Cyclopropanation of 1,1-Disubstituted ethylenes Catalyzed by Co-salen Complex (+)-1**2:**

To a solution of (+)-**12** (15 μmol) in CH_2Cl_2 (1.5 mL) was added KSAc (2 mg, 15 μmol) and the mixture was stirred at room temperature for 1 h. Ethyl diazoacetate (32 μL, 0.3 mmol) and 1,1-disubstituted ethylenes (0.45 mmol) were added to the reaction mixture and stirring was continued for the length of time specified in Tables 1-3.. The reaction mixture was passed through a short column of Celite which was eluted with CH_2Cl_2. The effluent was evaporated and the crude residue was purified by flash chromatography (SiO_2, hexanes) to give the product. The enantiomeric excess of the pure product was determined by HPLC on a Daicel Chiralcel OD, AD, OJ, OD-H or AS-H column.

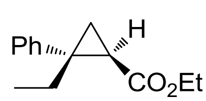
A procedure at 5 mmol scale was carried out with α-methylstyrene (967 μL, 7.5 mmol) and ethyl diazoacetate (533 μL, 5 mmol) using (+)-1**2** (150 mg, 0.25 mmol, 5 mol%) and KSAc (33.3 mg, 0.25 mmol, 5 mol%) in anhydrous CH_2Cl_2 (25 mL). The cyclopropane **(*E*)-7** was obtained in 92% yield and 93% ee.

(1*R*,2*R*)-Ethyl 2-Methyl-2-phenylcyclopropanecarboxylate [(*E*)-7]



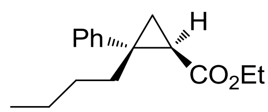
Pale yellow oil; *E*:*Z* ratio 31:1; 93% ee [Chiralcel OD-H, hexane:*i*-propanol 95:5, 0.5 mL/min, 215 nm, t_R [(1*R*,2*R*), major] 14.4 min, t_R [(1*S*,2*S*), minor] 16.9 min]; $[\alpha]_D^{22}$ - 291.1 (*c* 0.5, CH_2Cl_2), [lit[2]for (1*S*,2*S*) isomer $[\alpha]_D^{20}$ + 286.0 (*c* 0.3, $CHCl_3$); IR (neat) 2977, 2935, 1724, 1615, 1252, 1178, 1086, 1034, 848, 816, 790 cm^{-1}; ^1H NMR (700 MHz, $CDCl_3$) δ 1.26 (t, *J* = 7.4 Hz, 3H), 1.38 - 1.41 (m, 2H), 1.52 (s, 3H), 1.97 (dd, *J* = 8.2, 6.0 Hz, 1H), 4.10 - 4.19 (m, 2H), 7.16 - 7.24 (m, 2H), 7.27 - 7.41 (m, 3H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 15.3, 19.8, 21.3, 27.8, 30.6, 60.6, 127.3, 128.6, 128.9, 146.2, 173.1.

(1*R*,2*R*)-Ethyl 2-ethyl-2-phenylcyclopropanecarboxylate (14)



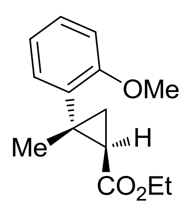
Yellow oil; *E*:*Z* ratio 26:1; 92% ee [Chiralcel OD-H, hexane:*i*-propanol 93:7, 0.5 mL/min, 215 nm, t_R [(1*R*,2*R*), major] 12.9 min, t_R [(1*S*,2*S*), minor] 15.7 min]; $[\alpha]_D^{16}$ - 168.6 (*c* 0.25, $CHCl_3$); IR (neat) 2985, 1732, 1370, 1332, 1268, 1188, 1152, 1035, 936 cm^{-1}; ^1H NMR (700 MHz, $CDCl_3$) δ 0.86 - 0.92 (m, 3H), 1.27 (t, *J* = 7.4 Hz, 3H), 1.53 - 1.56 (m, 2H), 1.84 - 1.93 (m, 1H), 1.97 - 2.03 (m, 1H), 2.74 - 2.82 (m, 1H), 3.87 - 3.95 (m, 2H), 7.42 - 7.48 (m, 2H), 7.53 - 7.61 (m, 1H), 7.96 - 8.01(m, 2H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 8.7, 14.2, 27.3, 31.9, 35.2, 63.9, 128.5, 129.2, 134.0, 136.6, 173.9; HRMS (EI) calcd for $C_{14}H_{19}O_2$ (M+H) *m/z* 219.0786, found 219.0778.

(1*R*,2*R*)-Ethyl 2-butyl-2-phenylcyclopropanecarboxylate (15)



Yellow oil; *E*:*Z* ratio 23:1; 90% ee [Chiralcel AS-H, hexane:*i*-propanol 98:2, 0.5 mL/min, 215 nm, t_R [(1*R*,2*R*), major] 8.5 min, t_R [(1*S*,2*S*), minor] 9.8 min]; $[\alpha]_D^{16}$ - 139.0 (*c* 0.4, $CHCl_3$); IR (neat) 2970, 2908, 1738, 1466, 1447, 1393, 1370, 1331, 1270, 1190, 1149, 1097, 1035, 948, 901, 889, 847 cm^{-1}; ^{1}H NMR (700 MHz, $CDCl_3$) δ 0.87 - 0.92 (m, 3H), 1.22 - 1.31 (m, 7H), 1.36 - 1.42 (m, 2H), 1.63 - 1.71 (m, 1H), 1.90 - 1.95 (m, 1H), 2.91 - 2.97 (m, 1H), 4.22 - 4.28 (m, 2H), 7.38 - 7.45 (m, 2H), 7.48 - 7.55 (m, 1H), 7.91 - 7.96 (m, 2H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 14.5, 15.6, 22.2, 23.1, 24.4, 25.9, 27.1, 38.6, 61.8, 127.7, 128.4, 129.4, 137.7, 176.8; HRMS (EI) calcd for $C_{16}H_{23}O_2$ (M+H) *m/z* 247.1698, found 247.1706.

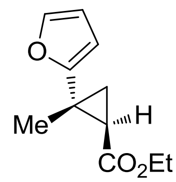
(1*R*,2*R*)-Ethyl 2-(2-methoxyphenyl)-2-methylcyclopropanecarboxylate (16)



Colorless oil; *E*:*Z* ratio 30:1; 96% ee [Chiralcel AD, hexane:*i*-propanol 96.6:3.4, 0.5 mL/min, 215 nm, t_R [(1*R*,2*R*), major] 14.4 min, t_R [(1*S*,2*S*), minor] 17.2 min]; $[\alpha]_D^{16}$ - 264.2 (*c* 0.15, $CHCl_3$); IR (neat) 2976, 2943, 1732, 1466, 1447, 1414, 1393, 1370, 1331, 1270, 1190, 1150, 1097, 1035 cm^{-1}; ^{1}H NMR (700 MHz, $CDCl_3$) δ 0.80 - 0.93 (m, 3H), 1.83 - 1.89 (m, 1H), 1.96 -

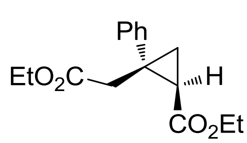
2.05 (m, 1H), 2.52 (s, 3H), 2.72 - 2.74 (m, 1H), 3.79 - 3.85 (m, 2H), 4.16 (s, 3H), 7.43 - 7.49 (m, 1H), 7.65 - 7.72 (m, 1H), 7.92 - 7.99 (m, 1H), 8.03 - 8.09 (m, 1H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 12.2, 23.2, 23.9, 28.7, 30.5, 56.1, 62.3, 113.2, 120.8, 129.7, 130.6, 138.0, 158.7, 174.3; HRMS (EI) calcd for $C_{14}H_{19}O_3$ (M+H) *m/z* 235.1334, found 235.1327.

(1*R*,2*R*)-Ethyl 2-(furan-2-yl)-2-methylcyclopropanecarboxylate (17)



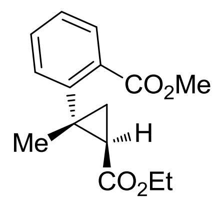
Colorless oil; *E*:*Z* ratio 23:1; 92% ee [Chiralcel OD-H, hexane:*i*-propanol 98:2, 0.5 mL/min, 215 nm, t_R [(1*R*,2*R*), major] 19.3 min, t_R [(1*S*,2*S*), minor] 22.5 min]; $[\alpha]_D^{16}$ - 156.6 (*c* 0.12, $CHCl_3$); IR (neat) 2985, 1735, 1466, 1447, 1414, 1370, 1332, 1270, 1190, 1150, 1035, 956, 866, 845 cm^{-1}; ^{1}H NMR (700 MHz, $CDCl_3$) δ 0.84 - 0.93 (m, 3H), 1.44 (s, 3H), 1.79 - 1.83 (m, 1H), 1.87 - 1.94 (m, 1H), 2.49 - 2.58 (m, 1H), 4.03 - 4.09 (m, 2H), 6.82 - 6.88 (m, 1H), 7.49 - 7.56 (m, 1H), 7.68 -7.73 (m, 1H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 14.1, 23.4, 23.9, 28.6, 29.5, 61.8, 121.6, 123.6, 124.2, 146.7, 173.9; HRMS (EI) calcd for $C_{11}H_{15}O_3$ (M+H) *m/z* 195.1021, found 195.1020.

(1*R*,2*R*)-Ethyl 2-(2-ethoxy-2-oxoethyl)-2-phenylcyclopropanecarboxylate (18)



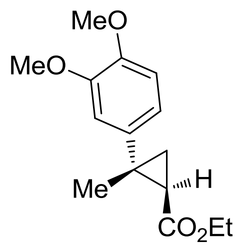
Yellow oil; *E*:*Z* ratio 25:1; 91% ee [Chiralcel OD-H, hexane:*i*-propanol 92:8, 0.5 mL/min, 215 nm, t_R [(1*R*,2*R*), major] 10.3 min, t_R [(1*S*,2*S*), minor] 11.9 min]; $[\alpha]_D^{16}$ - 136.2 (*c* 0.22, $CHCl_3$); IR (neat) 2970, 1741, 1466, 1447, 1414, 1370, 1332, 1268, 1189, 1152, 1035, 845 cm^{-1}; ^{1}H NMR (700 MHz, $CDCl_3$) δ 0.92 - 1.03 (m, 6H), 1.40 - 1.48 (m, 1H), 1.74 - 1.82 (m, 1H), 2.63 - 2.71 (m, 1H), 2.96 - 3.03 (m, 2H), 3.88 - 3.96 (m, 4H), 7.42 - 7.49 (m, 1H), 7.54 - 7.59 (m, 1H), 7.98 - 8.03 (m, 2H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 14.3, 14.6, 25.0, 28.2, 28.5, 58.4, 58.8, 126.3, 127.2, 129.6, 148.1, 170.9, 174.3; HRMS (EI) calcd for $C_{16}H_{20}O_4Na$ (M+Na) *m/z* 299.1259, found 299.1263.

Methyl 2-((1*R*,2*R*)-2-(ethoxycarbonyl)-1-methylcyclopropyl)benzoate (19)



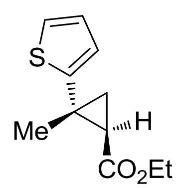
Pale yellow oil; *E*:*Z* ratio 30:1; 95% ee [Chiralcel OD-H, hexane:*i*-propanol 99:1, 0.5 mL/min, 215 nm, t_R [(1*R*,2*R*), major] 25.2 min, t_R [(1*S*,2*S*), minor] 31.4 min]; $[\alpha]_D^{16}$ - 92.7 (*c* 0.39, $CHCl_3$); IR (neat) 3012, 2976, 1735, 1466, 1448, 1370, 1332, 1269, 1190, 1150, 1097, 1036, 894, 836 cm^{-1}; ^{1}H NMR (700 MHz, $CDCl_3$) δ 1.03 - 1.11 (m, 3H), 1.62 - 1.67 (m, 1H), 1.89 - 1.97 (m, 1H), 2.16 (s, 3H), 2.80 - 2.89 (m, 1H), 3.92 - 4.01 (m, 5H), 7.38 - 7.47 (m, 1H), 7.57 - 7.66 (m, 2H), 7.83 - 7.91 (m, 1H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 13.0, 23.3, 24.0, 26.8, 27.4, 56.9, 61.2, 125.3, 126.7, 129.0, 133.4, 139.2, 156.8, 170.5, 173.4; HRMS (EI) calcd for $C_{15}H_{18}O_4$ *m/z* 262.3002, found 262.2997.

(1*R*,2*R*)-Ethyl 2-(3,4-dimethoxyphenyl)-2-methylcyclopropanecarboxylate (20)



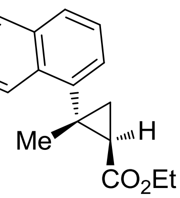
Colorless oil; *E*:*Z* ratio 32:1; 94% ee [Chiralcel OD-H, hexane:*i*-propanol 97.5:2.5, 0.5 mL/min, 215 nm, t_R [(1*R*,2*R*), major] 14.6 min, t_R [(1*S*,2*S*), minor] 17.2 min]; $[\alpha]_D^{16}$ - 226.1 (*c* 0.15, $CHCl_3$); IR (neat) 2985, 2942, 2908, 1735, 1466, 1447, 1414, 1393, 1370, 1332, 1268, 1189, 1152, 1096, 1035, 955, 845, 786, 675 cm^{-1}; ^1H NMR (700 MHz, $CDCl_3$) δ 0.82 - 0.99 (m, 3H), 1.45 (s, 3H), 1.81 - 1.90 (m, 1H), 1.97 - 2.09 (m, 1H), 2.88 - 2.97 (m, 1H), 3.94 - 4.18 (m, 8H), 6.88 - 6.98 (m, 1H), 7.53 - 7.75 (m, 2H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 13.9, 25.6, 28.0, 29.7, 32.1, 56.4, 63.3, 111.9, 113.9, 117.5, 143.2, 147.3, 149.9, 173.2; HRMS (EI) calcd for $C_{15}H_{20}O_4Na$ (M+Na) *m/z* 287.1259, found 287.1262.

(1*R*,2*R*)-Ethyl 2-methyl-2-(thiophen-2-yl)cyclopropanecarboxylate (21)



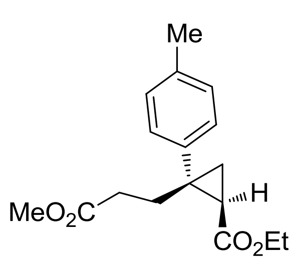
Colorless oil; *E*:*Z* ratio 25:1; 90% ee [Chiralcel OD-H, hexane:*i*-propanol 96.5:3.5, 0.5 mL/min, 215 nm, t_R [(1*R*,2*R*), major] 15.1 min, t_R [(1*S*,2*S*), minor] 18.9 min]; $[\alpha]_D^{16}$ - 201.5 (*c* 0.12, $CHCl_3$); IR (neat) 2985, 1740, 1370, 1332, 1269, 1190, 1150, 1036, 952, 837, 780 cm^{-1}; ^1H NMR (700 MHz, $CDCl_3$) δ 0.92 - 1.06 (m, 3H), 1.46 (s, 3H), 1.72 - 1.81 (m, 1H), 1.96 - 2.06 (m, 1H), 2.70 - 2.79 (m, 1H), 3.96 - 4.09 (m, 2H), 7.13 - 7.22 (m, 1H), 7.64 - 7.69 (m, 1H), 7.70 - 7.81 (m, 1H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 13.5, 24.0, 25.9, 27.3, 29.3, 62.4, 128.6, 133.4, 134.7, 145.2, 191.9; HRMS (EI) calcd for $C_{11}H_{14}O_2S$ *m/z* 210.2853, found 210.2856.

(1*R*,2*R*)-Ethyl 2-methyl-2-(naphthalen-1-yl)cyclopropanecarboxylate (22)



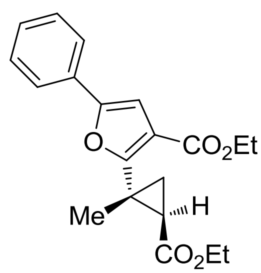
Colorless oil; *E*:*Z* ratio 33:1; 96% ee [Chiralcel OD-H, hexane:*i*-propanol 99.5:0.5, 0.5 mL/min, 215 nm, t_R [(1*R*,2*R*), major] 40.7 min, t_R [(1*S*,2*S*), minor] 45.8 min]; $[\alpha]_D^{16}$ - 121.0 (*c* 0.25, $CHCl_3$); IR (neat) 2979, 1751, 1463, 1441, 1370, 1332, 1264, 1185, 1099, 1034, 900, 845, 794 cm^{-1}; ^{1}H NMR (700 MHz, $CDCl_3$) δ 0.88 - 0.97 (m, 3H), 1.55 (s, 3H), 1.74 - 1.83 (m, 1H), 1.88 - 1.94 (m, 1H), 2.50 - 2.57 (m, 1H), 4.16 - 4.22 (m, 2H), 6.92 (d, *J* = 8.2 Hz, 1H), 7.36 - 7.43 (m, 1H), 7.53 (d, *J* = 8.2 Hz, 1H), 7.58 - 7.63 (m, 2H), 7.93 (d, *J* = 7.8 Hz, 1H), 8.17 (d, *J* = 7.8 Hz, 1H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 13.7, 25.2, 27.2, 28.1, 28.9, 62.7, 121.8, 125.4, 126.0, 126.4, 127.0, 127.9, 135.4, 148.8, 171.7; HRMS (EI) calcd for $C_{17}H_{18}O_2Na$ (M+Na) *m/z* 277.1204, found 277.1196.

(1*R*,2*R*)-Ethyl 2-(3-methoxy-3-oxopropyl)-2-(p-tolyl)cyclopropanecarboxylate (23)



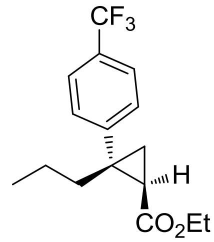
Pale yellow oil; *E*:*Z* ratio 27:1; 94% ee [Chiralcel OD-H, hexane:*i*-propanol 94:6, 0.5 mL/min, 215 nm, t_R [(1*R*,2*R*), major] 13.2 min, t_R [(1*S*,2*S*), minor] 15.1 min]; $[\alpha]_D^{16}$ - 128.6 (*c* 0.4, $CHCl_3$); IR (neat) 2986, 1735, 1466, 1447, 1414, 1370, 1332, 1268, 1186, 1152, 1096, 1035, 845 cm^{-1}; ^1H NMR (700 MHz, $CDCl_3$) δ 1.60 - 1.76 (m, 5H), 1.83 - 1.91 (m, 2H), 2.08 - 2.11 (m, 1H), 2.17 - 2.23 (m, 1H), 2.42 (s, 3H), 2.82 - 2.88 (m, 1H), 3.83 (s, 3H), 3.97 - 4.04 (2H), 7.31 (d, *J* = 7.9 Hz, 1H), 7.93 (d, *J* = 7.9 Hz, 1H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 22.1, 24.2, 27.8, 30.9, 31.6, 33.0, 58.6, 64.2, 129.2, 130.3, 135.0, 145.7, 174.6, 178.1; HRMS (EI) calcd for $C_{17}H_{22}O_4$ *m/z* 290.1692, found 290.1698.

Ethyl 2-((1*R*,2*R*)-2-(ethoxycarbonyl)-1-methylcyclopropyl)-5-phenylfuran-3-carboxylate (24)



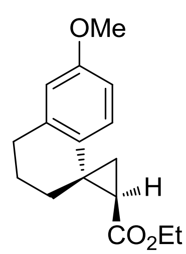
Yellow oil; *E*:*Z* ratio 26:1; 97% ee [Chiralcel OD-H, hexane:*i*-propanol 90:10, 0.5 mL/min, 215 nm, t_R [(1*R*,2*R*), major] 9.6 min, t_R [(1*S*,2*S*), minor] 10.4 min]; $[\alpha]_D^{16}$ - 176.2 (*c* 0.61, $CHCl_3$); IR (neat) 2976, 2908, 1732, 1466, 1447, 1414, 1393, 1370, 1331, 1270, 1190, 1150, 1097, 1035, 934, 867, 821, 781 cm^{-1}; ^1H NMR (700 MHz, $CDCl_3$) δ 1.31 - 1.47 (m, 6H), 1.80 - 1.87 (m, 1H), 2.02 - 2.09 (m, 1H), 2.68 (s, 3H), 2.89 - 2.99 (m, 1H), 4.27 - 4.46 (m, 4H), 6.58 (s, 1H), 7.29 - 7.36 (m, 1H), 7.39 - 7.48 (m, 2H), 7.62 - 7.73 (m, 2H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 13.9, 14.2, 24.9, 26.0, 26.7, 27.0, 57.8, 61.2, 106.8, 108.1, 124.0, 127.4, 128.1, 130.3, 158.5, 165.2, 170.3, 173.4; HRMS (EI) calcd for $C_{20}H_{22}O_5Na$ (M+Na) *m/z* 365.1385, found 375.1383.

(1*R*,2*R*)-Ethyl 2-propyl-2-(4-(trifluoromethyl)phenyl)cyclopropanecarboxylate (25)



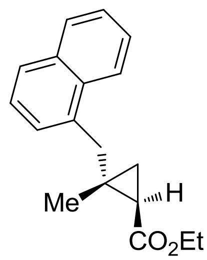
Pale yellow oil; *E*:*Z* ratio 21:1; 92% ee [Chiralcel OD-H, hexane:*i*-propanol 95:5, 0.5 mL/min, 215 nm, t_R [(1*R*,2*R*), major] 19.4 min, t_R [(1*S*,2*S*), minor] 24.0 min]; $[\alpha]_D^{16}$ - 79.3 (*c* 0.41, $CHCl_3$); IR (neat) 2985, 1735, 1414, 1392, 1330, 1372, 1266, 1193, 1151, 1030, 920, 855 cm^{-1}; ^1H NMR (700 MHz, $CDCl_3$) δ 0.89 - 0.97 (m, 5H), 1.24 - 1.31 (m, 3H), 1.54 - 1.64 (m, 2H), 1.88 - 1.97 (m, 1H), 2.07 - 2.14 (m, 1H), 2.75 - 2.86 (m, 1H), 4.18 - 4.26 (m, 2H), 7.56 (d, *J* = 8.0 Hz, 1H), 7.95 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 13.3, 13.9, 19.7, 21.1, 16.0, 32.4, 43.5, 62.2, 120.8, 122.2, 127.1, 128.0, 153.9, 171.7; HRMS (EI) calcd for $C_{16}H_{19}F_3O_2Na$ (M+Na) *m/z* 323.1223, found 323.1225.

(1*R*,2*R*)-Ethyl 6'-methoxy-3',4'-dihydro-2'H-spiro[cyclopropane-1,1'-naphthalene]-2-carboxylate (26)



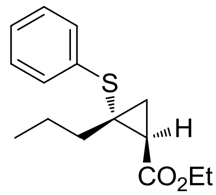
Colorless oil; single diastereomer; 98% ee [Chiralcel OD-H, hexane:*i*-propanol 95:5, 0.5 mL/min, 215 nm, t_R [(1*R*,2*R*), major] 23.8 min, t_R [(1*S*,2*S*), minor] 30.2 min]; $[\alpha]_D^{16}$ - 128.6 (*c* 0.4, $CHCl_3$); IR (neat) 2985, 2942, 2908, 1732, 1466, 1447, 1414, 1393, 1370, 1332, 1270, 1190, 1150, 1035, 955, 866, 845 cm^{-1}; ^{1}H NMR (700 MHz, $CDCl_3$) δ 1.12 - 1.20 (m, 3H), 1.22 - 1.36 (m, 6H), 1.63 - 1.71 (m, 1H), 1.85 - 1.90 (m, 1H), 2.73 - 2.79 (m, 1H), 3.77 (s, 3H), 4.17 - 4.26 (m, 2H), 6.87 (s, 1H), 7.04 (d, *J* = 8.0 Hz, 1H), 8.03 (d, *J* = 8.0 Hz, 1H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 13.3, 19.1, 23.5, 26.5, 30.1, 30.4, 34.8, 56.0, 61.2, 113.4, 113.8, 127.0, 136.9, 142.1, 158.7, 173.2; HRMS (EI) calcd for $C_{16}H_{21}O_3$ (M+H) *m/z* 261.1509, found 261.1508.

(1*R*,2*R*)-Ethyl 2-methyl-2-(naphthalen-1-ylmethyl)cyclopropanecarboxylate (27)



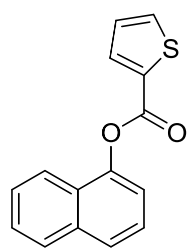
Yellow oil; *E*:*Z* ratio 18:1; 83% ee [Chiralcel OD-H, hexane:*i*-propanol 97:3, 0.5 mL/min, 215 nm, t_R [(1*R*,2*R*), major] 18.8 min, t_R [(1*S*,2*S*), minor] 23.7 min]; $[\alpha]_D^{16}$ - 46.8 (*c* 0.15, $CHCl_3$); IR (neat) 3261, 2985, 2907, 1716, 1580, 1504, 1447, 1370, 1331, 1096, 1033, 847, 817, 763, 699 cm^{-1}; ^{1}H NMR (700 MHz, $CDCl_3$) δ 1.05 - 1.17 (m, 3H), 1.69 - 1.76 (m, 1H), 1.86 - 1.92 (m, 1H), 2.12 (s, 3H), 2.40 (s, 2H), 2.74 - 2.80 (m, 1H), 4.13 - 4.21 (m, 2H), 7.41 - 7.60 (m, 4H), 7.82 - 7.87 (m, 1H), 7.92 (d, *J* = 8.1 Hz, 1H), 8.03 (d, *J* = 8.1 Hz, 1H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 17.0, 25.7, 26.2, 27.3, 31.9, 48.3, 62.9, 120.3, 125.0, 125.9, 126.3, 127.6, 128.4, 129.7, 132.8, 133.3, 134.0, 171.8; HRMS (EI) calcd for $C_{18}H_{20}O_2Na$ (M+Na) *m/z* 291.1381, found 291.1387.

(1*S*,2*R*)-Ethyl 2-(phenylthio)-2-propylcyclopropanecarboxylate (28)



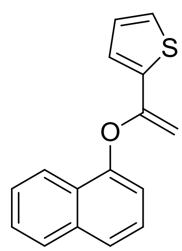
Pale yellow oil; *E*:*Z* ratio 16:1; 88% ee [Chiralcel OD-H, hexane:*i*-propanol 98:2, 0.5 mL/min, 215 nm, t_R [(1*R*,2*R*), major] 20.6 min, t_R [(1*S*,2*S*), minor] 27.3 min]; $[\alpha]_D^{16}$ - 69.3 (*c* 0.4, $CHCl_3$); IR (neat) 2985, 1751, 1467, 1447, 1392, 1370, 1332, 1265, 1185, 1096, 1034, 901, 858 cm^{-1}; ^1H NMR (700 MHz, $CDCl_3$) δ 0.92 - 0.96 (m, 3H), 0.97 - 1.02 (m, 3H), 1.39 - 1.58 (m, 2H), 1.62 - 1.72 (m, 2H), 1.81 - 1.87 (m, 1H), 1.93 - 2.02 (m, 1H), 2.89 - 2.97 (m, 1H), 4.22 - 4.27 (m, 2H), 7.37 - 7.46 (m, 2H), 7.49 - 7.56 (m, 1H), 7.91 - 7.97 (m, 2H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 16.5, 20.9, 28.3, 28.9, 32.2, 41.1, 64.5, 136.7, 141.9, 144.2, 146.9, 174.7; HRMS (EI) calcd for $C_{15}H_{20}O_2SNa$ (M+Na) *m/z* 287.1082, found 287.1089.

1-Naphthyl Thiophen-2-carboxylate (32)



To a solution of 2-thiophenecarbonyl chloride (**31**, 1.25 g, 8.53 mmol) in THF (30 mL) at 0^{o}C was added a solution of 1-naphthol (**30**, 2.76 g, 19.15 mmol) in THF (20 mL) dropwise. After addition was complete, the solution was stirred at room temperature for 10 min and Et_3N (2.8 mL, 20 mmol) was added. A colorless solid was precipitated immediately and the suspension was stirred for 14 h. The reaction mixture was quenched with 5M aquoues HCl (20 mL) and was extracted with CH_2Cl_2 (4 x 120 mL). The organic layer was dried (Na_2SO_4) and evaporated in vacuo, and the crude residue was purified by flash chromatography (SiO_2-hexanes) to afford **32** (2.16 g, 100%) as an amorphous colorless solid; mp 73 - 74 ^oC, [lit[3] 70 - 75 ^oC]; IR (neat) 3295, 2980, 2934, 1738, 1698, 1650, 1580, 1560, 1463, 1428, 1408, 1367, 1244, 1195, 1151, 1084, 1022, 990, 941, 808, 760, 736 cm^{-1}; ^1H NMR (700 MHz, $CDCl_3$) δ 7.29 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.43 - 7.58 (m, 1H), 7.52 - 7.58 (m, 3H), 7.75 (d, *J* = 7.8 Hz, 1H), 7.80 - 7.84 (m, 1H), 7.92 (d, *J* = 8 Hz, 1H), 7.97 - 8.05 (m, 1H), 8.12 - 8.16 (m, 1H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 118.3, 121.6, 125.2, 126.0, 126.4, 126.7, 128.0, 128.2, 132.7, 133.5, 134.7, 134.9, 146.5, 161.0.

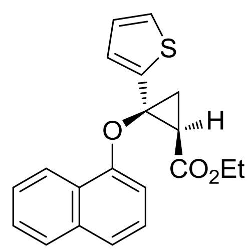
2-(1-(Naphthalen-1-yloxy)vinyl)thiophene (33)



A solution of **32** (1.27 g, 5.0 mmol) in THF (10 mL) was syringed into a flask containing Tebbe reagent,[4] prepared from titanocene dichloride (1.91 g, 7.70 mmol) and trimethylaluminum (7.70 mL of 2M solution in toluene, 112 mmol), at room temperature. The slurry was stirred for 24 h at room temperature and was diluted with ether (15 mL). The reaction mixture was extracted with ether (2 x 10 mL), washed with 1M aqueous NaOH (2 x 10 mL), dried (Na_2SO_4) and evaporated. The crude residue was passed through a short path of neutral silica, eluting with ether (120 mL) containing 5% Et_3N. The effluent was concentrated in vacuo and the crude residue was purified by flash chromatography on silica gel (95% hexanes, 5% Et_3N) to give **33** (986 mg, 78%) as a brown oil; IR (neat) 2929, 2858, 1652, 1457, 1258, 1073, 750 cm^{-1}; ^1H NMR (700 MHz, $CDCl_3$) δ 4.29 (d, *J* = 3.1 Hz, 1H), 4.99 (d, *J* =3.1 Hz, 1H), 7.05 - 7.10 (m, 1H), 7.26 - 7.29 (m, 1H), 7.30

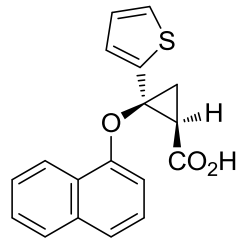
- 7.34 (m, 1H), 7.42 -7.50 (m, 2H), 7.51 - 7.59 (m, 2H), 7.68 - 7.74 (m, 1H), 7.89 - 7.96 (m, 1H), 8.18 - 8.23 (m, 1H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 90.7, 114.8, 116.9, 122.3, 124.7, 124.9, 125.8, 125.9, 126.3, 127.0, 127.6, 128.0, 135.3, 139.5, 151.7, 155.8.

(1*R*,2*S*)-Ethyl 2-(Naphthalen-1-yloxy)-2-(thiophen-2-yl)cyclopropanecarboxylate [(+)-34]



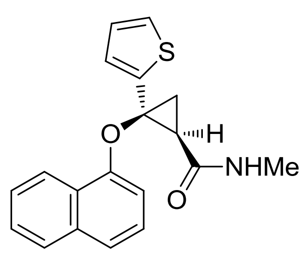
Yellow oil; *Z*:*E* ratio 17:1; 94% ee [Chiralcel OJ, hexane:*i*-propanol 94.5:5.5, 0.5 mL/min, 215 nm, t_R [(1*R*,2*R*), major] 21.8 min, t_R [(1*S*,2*S*), minor] 28.4 min]; $[\alpha]_D^{22}$ + 36.3 (*c* 0.5, $CHCl_3$); IR (neat) 3473, 3029, 2931, 1735, 1496, 1454, 1380, 1260, 1174, 1037, 734, 697 cm^{-1}; ^{1}H NMR (700 MHz, $CDCl_3$) δ 1.21 (t, *J* = 6.9 Hz, 3H), 1.88 - 1.97 (m, 1H), 2.37 (s, 3H), 1.97 (dd, *J* = 8.2, 6.0 Hz, 1H), 2.38 (t, *J* = 6.7 Hz, 1H), 2.79 (dd, *J* = 9.9, 7.5 Hz, 1H), 4.01 (q, *J* = 6.9 Hz, 2H), 6.81 - 6.91 (m, 1H), 7.02 - 7.05 (m, 1H), 7.05 - 7.15 (m, 1H), 7.16 -7.21 (m, 1H), 7.22 - 7.27 (m, 1H), 7.28 - 7.30 (m, 1H), 7.32 - 7.40 (m, 2H), 7.41 - 7.47 (m, 1H), 7.67 - 7.72 (m, 1H), 8.12 - 8.22 (m, 1H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 15.5, 24.2, 32.9, 61.8, 63.4, 108.4, 121.6, 122.0, 124.8, 125.2, 125.9, 126.2, 126.9, 127.7, 127.9, 128.1, 134.5, 135.6, 151.8, 169.3; HRMS (EI) calcd for $C_{20}H_{19}O_3S$ (M+H) *m/z* 339.1055, found 339.1055.

(1*R*,2*S*)-2-(Naphthalen-1-yloxy)-2-(thiophen-2-yl)cyclopropanecarboxylic Acid [(+)-35]



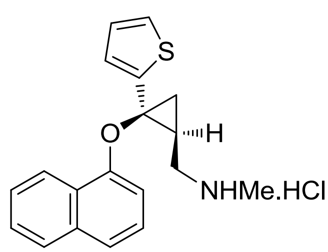
To a solution of (+)-**34** (113 mg, 0.33 mmol) in THF (8 mL) was added LiOH.H_2O (48 mg, 2 mmol) followed by H_2O (2 mL). The mixture was stirred at room temperature for 24 h and was acidified with 1M HCl to pH 6. The reaction mixture was extracted with ether (2 x 50 mL), dried (Na_2SO_4) and evaporated to obtain (+)-**35** (99 mg, 96%) as a white solid; mp 169 - 170 °C, [lit[3] for racemate 167 - 168 °C]; $[\alpha]_D^{20}$ + 54.2 (*c* 0.4, MeOH), [lit[3] for (1*S*,2*R*) isomer $[\alpha]_D^{25}$ - 51.4 (*c* 0.07, $CHCl_3$)]; IR (neat) 3537, 2983, 2938, 1739, 1465, 1445, 1407, 1368, 1282, 1176, 1114, 1027 cm^{-1}; ^1H NMR (700 MHz, $CDCl_3$) δ 1.99 (dd, *J* = 9.1, 6.2 Hz, 1H), 2.12 (t, *J* = 7.3 Hz, 1H), 2.83 (dd, *J* = 9.1, 8.3 Hz, 1H), 6.94 (dd, *J* = 5.2, 4.7 Hz, 1H), 7.13 (d, J = 8.0 Hz, 1H), 7.20 - 7.29 (m, 3H), 7.42 (d, *J* = 8.0 Hz, 1H), 7.51 (t, *J* = 3.1 Hz, 1H), 7.62 (d, *J* = 3.1 Hz, 1H), 7.72 - 7.76 (m, 1H), 8.18 - 8.22 (m, 1H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 20.9, 33.7, 64.2, 110.2, 120.4, 120.8, 121.3, 121.7, 122.0, 123.4, 125.6, 128.6, 129.8, 129.7, 138.7, 144.3, 158..7, 173.5.

(1*R*,2*S*)-N-Methyl-2-(naphthalen-1-yloxy)-2-(thiophen-2-yl)cyclopropanecarboxamide [(-)-36]



To a solution of (+)-**35** (95 mg, 0.306 mmol) in THF (6 mL) was added Hunig's base (160 μL, 0.918 mmol) followed by 4-dimethylaminopyridine (4 mg, 0.31 mmol), methylamine hydrochloride (62 mg, 0.918 mmol), 1-[3-(Dimethylamino)propyl]-3-ethylcarbodiimide methiodide (182 mg, 0.612 mg) and the reaction mixture was stirred at room temperature for 13 h. The solvent of the reaction mixture was removed under reduced pressure and the crude residue was purified by flash chromatography on silica gel (0 - 10% ethyl acetate in hexanes) to give (-)-**36** (98 mg, 99%) as a pale yellow oil; $[\alpha]_D^{16}$ - 59.9 (*c* 0.22, $CHCl_3$), [lit[3] for (1*S*,2*R*) isomer $[\alpha]_D^{25}$ + 59.4 (*c* 0.36, $CHCl_3$)]; IR (neat) 3210, 1769, 1701, 1427, 1320, 1294, 1244, 1320, 1294, 1244, 1179, 917, 852, 815 cm^{-1}; ^1H NMR (700 MHz, $CDCl_3$) δ 2.11 - 2.24 (m, 2H), 2.33 - 2.41 (m, 1H), 2.92 (d, *J* = 4.2 Hz, 3H), 5.93 (br. S, 1H), 6.95 - 7.09 (m, 3H), 7.18 - 7.32 (m, 2H), 7.41 - 7.50 (m, 2H), 7.82 (dd, *J* = 6.2, 3.1 Hz, 1H), 8.23 (dd, *J* = 6.1, 3.1 Hz, 1H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 21.7, 15.9, 34.8, 62.6, 108.7, 121.8, 123.4, 123.9, 124.3, 124.5, 125.9, 126.7, 127.8, 128.4, 129.1, 134.5, 144.8, 153.4, 168.0.

***N*-Methyl-1-((1*S*,2*S*)-2-(naphthalen-1-yloxy)-2-(thiophen-2-yl)cyclopropyl)methanamine hydrochloride [(+)-29]**



To a solution of (-)-**36** (96 mg, 0.296 mmol) in THF (5 mL) was added lithium aluminium hydride (45 mg, 1.187 mmol) and the mixture was refluxed for 4 h. The reaction mixture was cooled to 0 °C and was quenched by aqueous ammonium chloride. The reaction mixture was extracted in ethyl acetate (2x25 mL), evaporated and treated with 1(M) HCl in ether (1 mL). A white precipitate formed which was filtered, washed with ether and dried to obtain (+)-**29** (89 mg, 87%) as a colorless solid; mp 251 - 252 ^{o}C [lit.[3] 252 ^{o}C]; $[\alpha]_D^{16}$ + 51.2 (*c* 0.22, $CHCl_3$), [lit[3] $[\alpha]_D^{20}$ + 51.4 (*c* 0.07, $CHCl_3$)]; IR (neat) 3529, 3346, 3052, 2661, 1630, 1596, 1577, 1514, 1494, 1457, 1276, 1241, 1206, 1148, 1083, 1043, 1015, 961, 878, 794, 770, 699 cm^{-1}; ^{1}H NMR (700 MHz, $CDCl_3$) δ 1.49 - 1.57 (m, 1H), 1.97 - 2.10 (m, 2H), 2.81 - 2.94 (m, 3H), 3.36 - 3.45 (m, 2H), 6.90 - 6.94 (m, 1H), 6.95 - 7.02 (m, 1H), 7.04 - 7.24 (m, 3H), 7.42 (d, *J* = 8.1 Hz, 1H), 7.45 - 7.53 (m, 2H), 7.80 (d, *J* = 8.1 Hz, 1H), 8.22 - 8.28 (m, 1H), 9.81 (br, s, 2H); ^{13}C NMR (175 MHz, $CDCl_3$) δ 21.8, 26.3, 48.1, 61.4, 83.6, 108.8, 121.3, 121.9, 122.4, 122.8, 125.4, 125.5, 126.0, 126.9, 127.3, 128.0, 134.5, 143.7, 151.9.

References:

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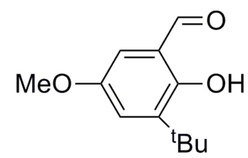
2. Berkessel, A.; Kaiser, P.; Lex, J. *Chem Eur. J.* **2003**, *9*, 4746.
3. White, J. D.; Juniku, R.; Huang, K.; Yang., J.; Wong, D. T. *J. Med. Chem.* **2009**, *52*, 5872.
4. Tebbe, F. N.; Parshall, G. W.; Reddy, G. S. *J. Am. Chem. Soc.* **1978**, *100*, 3611.



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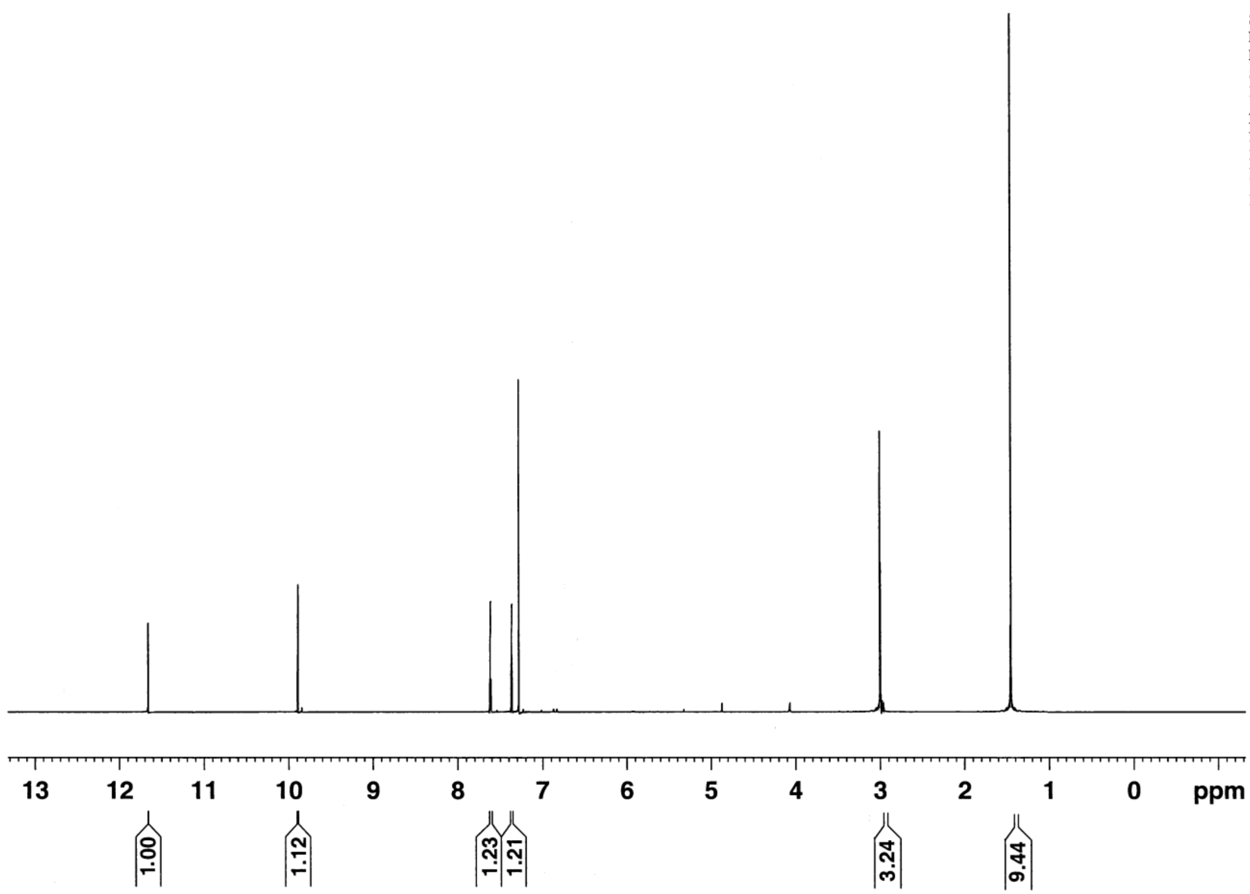


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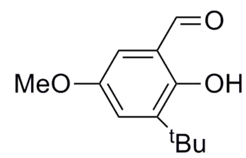
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```



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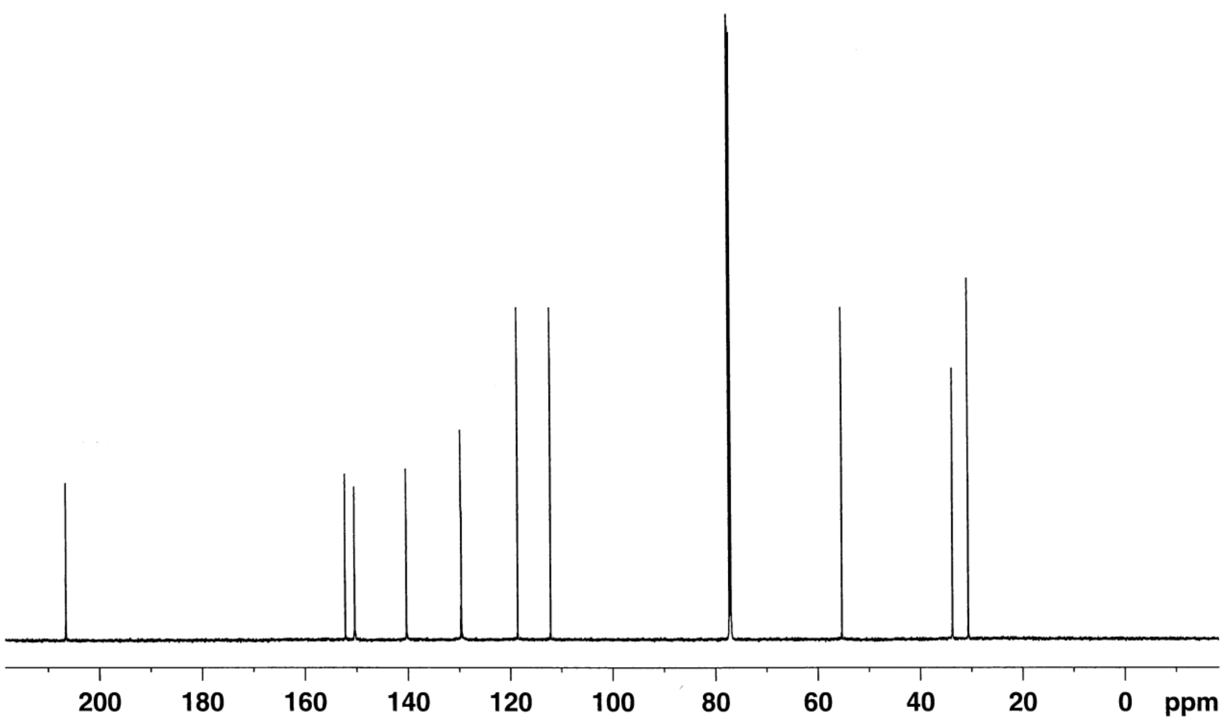
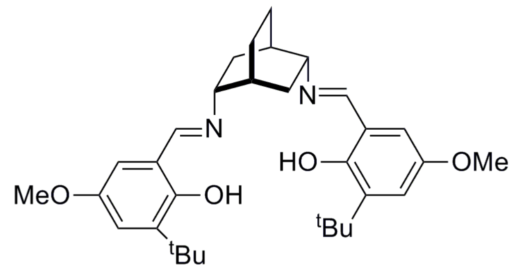


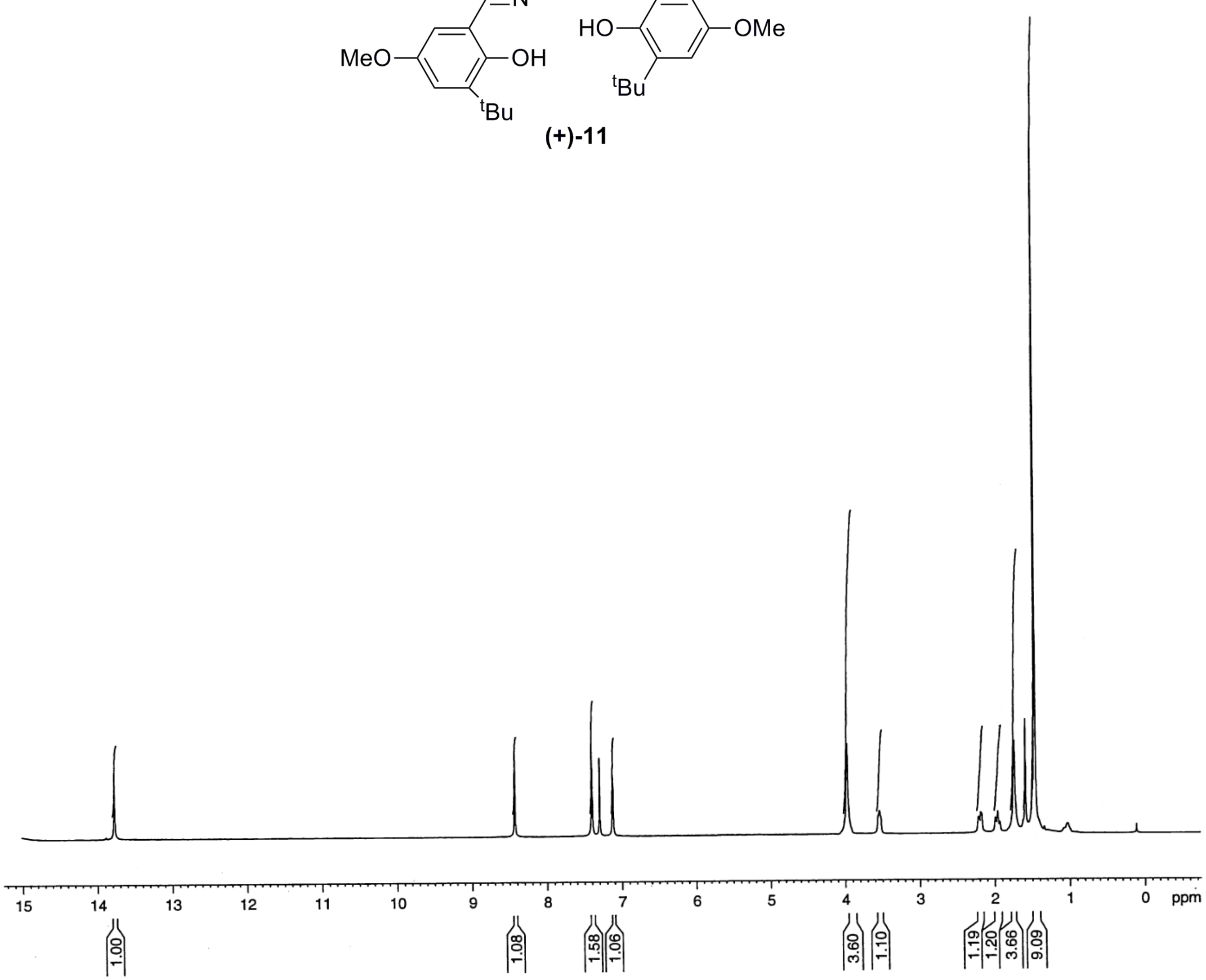

```
NAME            SS-06-110
EXPNO                   2
PROCNO                  1
Date_            20130323
Time                17.08
INSTRUM             spect
PROBHD   5 mm CPDCH 13C
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     14
DS                      4
SWH            41666.668 Hz
FIDRES          0.635783 Hz
AQ             0.7864820 sec
RG                    203
DW                 12.000 usec
DE                  16.50 usec
TE                  298.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.00 usec
PL1                  4.50 dB
PL1W          38.14553833 W
SFO1          176.0697436 MHz

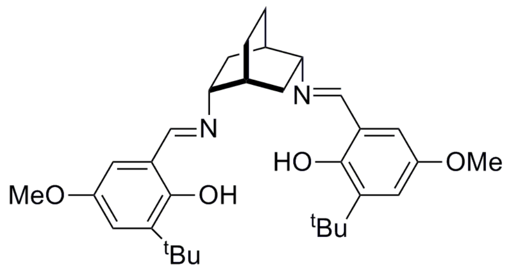
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               65.00 usec
PL2                 -3.20 dB
PL12                13.60 dB
PL13               120.00 dB
PL2W          33.59817505 W
PL12W          0.70196527 W
PL13W          0.00000000 W
SFO2          700.1499406 MHz
SI                  32768
SF            176.0521380 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.00
```



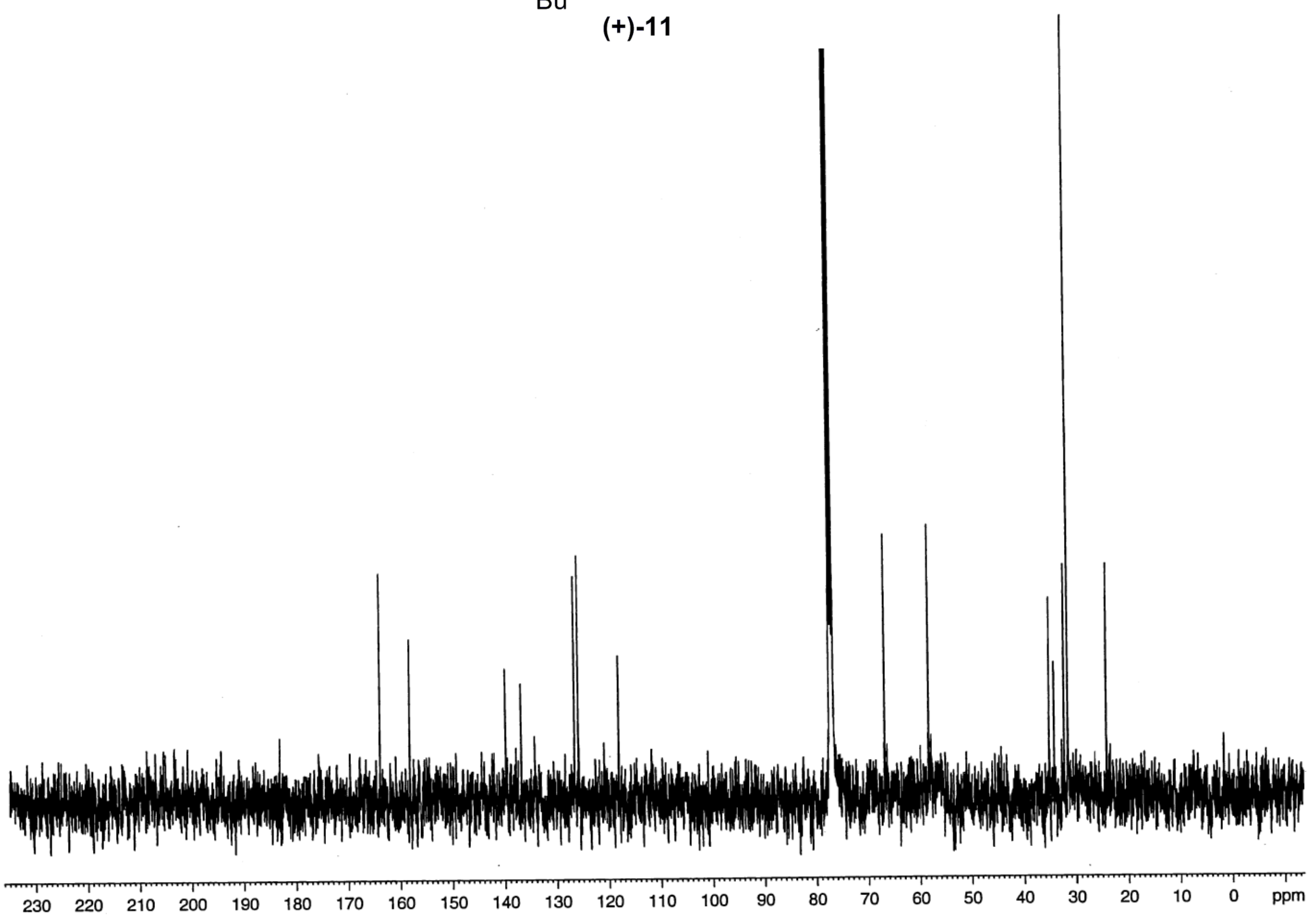
(+)-11



Current Data Parameters		
NAME	SS-6-130	
EXPNO	1	
PROCNO	1	
F2 - Acquisition Parameters		
Date_	20130309	
Time	17.43	
INSTRUM	spect	
PROBHD	5 mm PATXI 1H/	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8012.820	Hz
FIDRES	0.122266	Hz
AQ	4.0894465	sec
RG	97.9	
DW	62.400	usec
DE	6.50	usec
TE	298.0	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
SFO1	500.1330008	MHz
NUC1	1H	
P1	7.80	usec
PLW1	12.00000000	W
F2 - Processing parameters		
SI	65536	
SF	500.1300000	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	



(+)-11



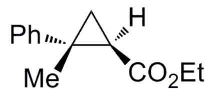
NAME	SS-06-130
EXPNO	2
PROCNO	1
Date_	20130323
Time	18.57
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	20
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 sec
RG	203
DW	12.000 use
DE	16.50 use
TE	298.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

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

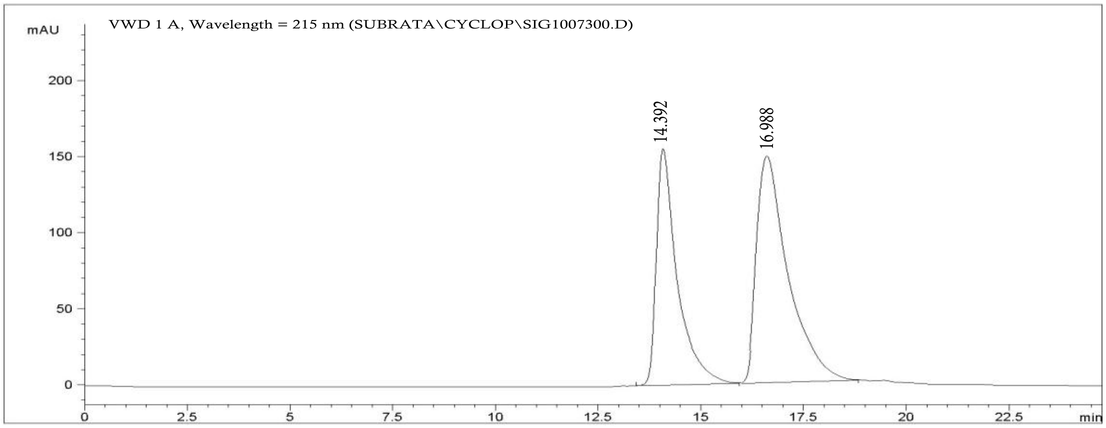
NUC1	13C
P1	9.00 use
PL1	4.50 dB
PL1W	38.14553833 W
SFO1	176.0697436 MHz

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

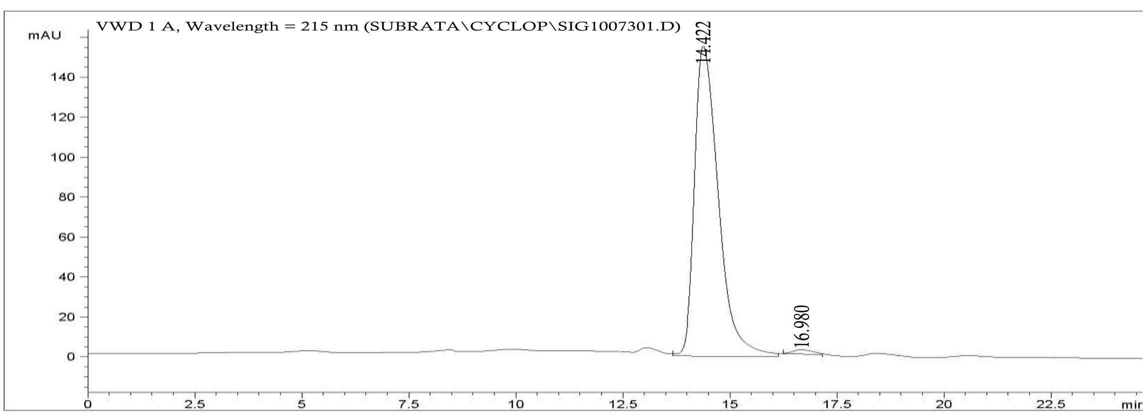
CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 use
PL2	-3.20 dB
PL12	13.60 dB
PL13	120.00 dB
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1499406 MHz
SI	32768
SF	176.0521380 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00



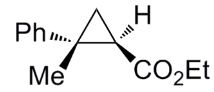
(*E*)-7



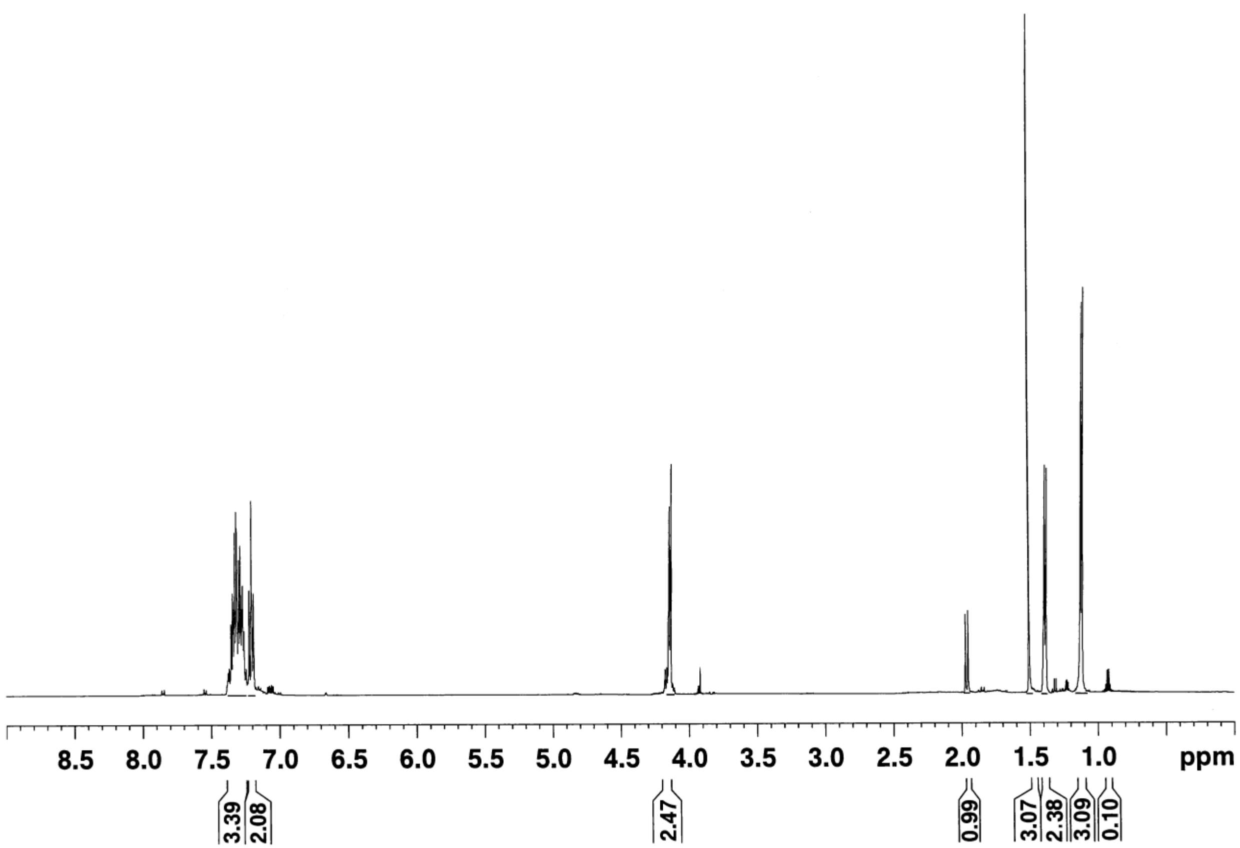
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.392	BB	0.8228	1.27544e4	223.39753	48.0518
2	16.988	VB	1.0933	1.37886e4	180.97344	51.9481



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.422	BB	0.6139	5009.19349	154.47652	96.1410
2	16.980	VB	0.5782	201.06383	2.21761	3.8590



(*E*)-7

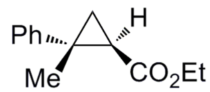



```
Current Data Parameters
NAME            SS-6-137
EXPNO                  2
PROCNO                 1

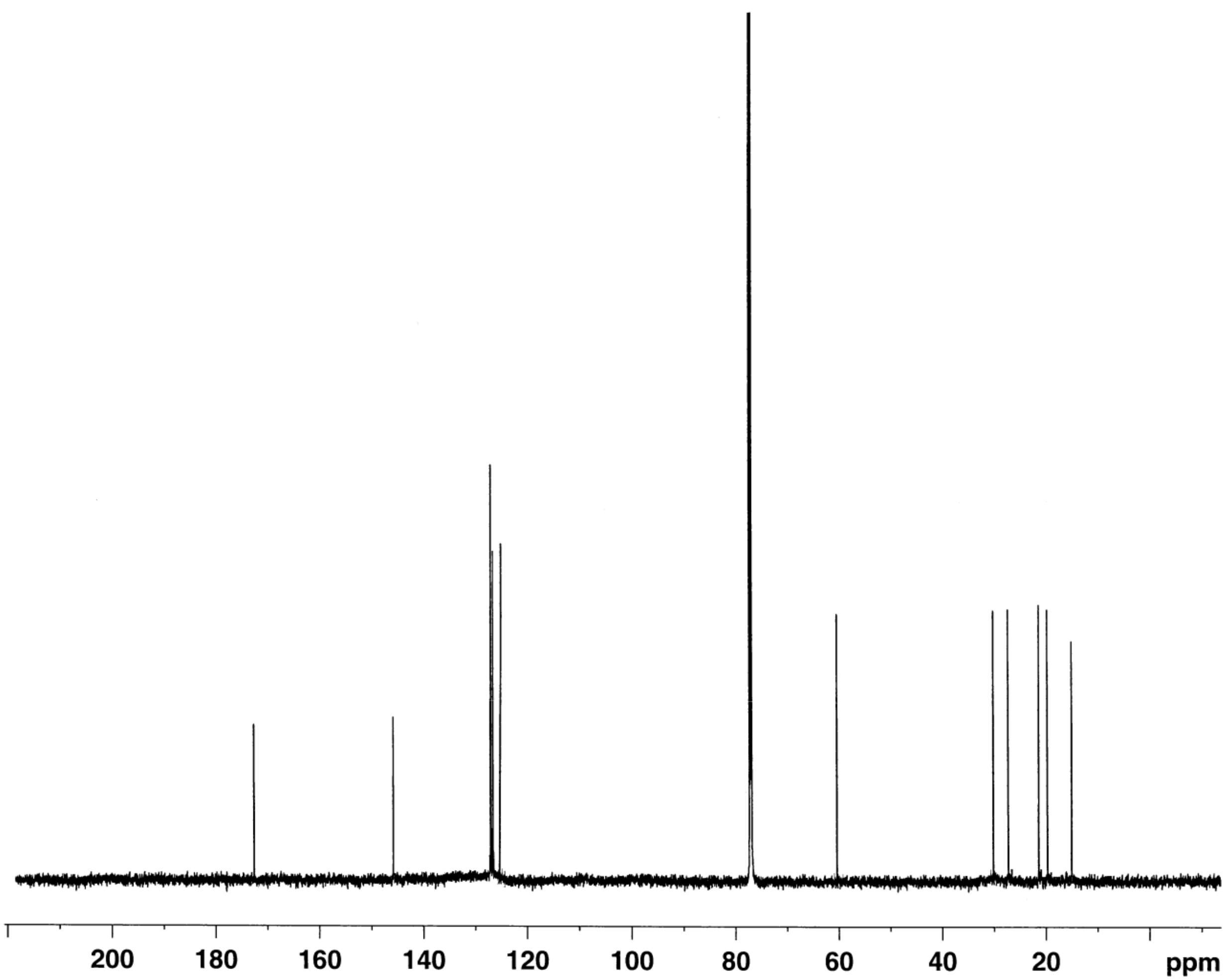
F2 - Acquisition Parameters
Date_           20130316
Time               18.26
INSTRUM            spect
PROBHD   5 mm PATXI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                 46.39
DW                62.400 use
DE                  6.50 use
TE                 297.9 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 =======
SFO1         500.1330008 MHz
NUC1                  1H
P1                  7.80 use
PLW1         12.00000000 W

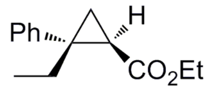
F2 - Processing parameters
SI                 65536
SF           500.1300000 MHz
WDW                   EM
SSB      0
LB                  0.30 Hz
GB       0
PC                  1.00
```



(*E*)-7



NAME	SS-06-137
EXPNO	1
PROCNO	1
Date_	20130323
Time	19.26
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	20
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 sec
RG	203
DW	12.000 usec
DE	16.50 usec
TE	298.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.00 usec
PL1	4.50 dB
PL1W	38.14553833 W
SFO1	176.0697436 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 usec
PL2	-3.20 dB
PL12	13.60 dB
PL13	120.00 dB
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1499406 MHz
SI	32768
SF	176.0521380 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00



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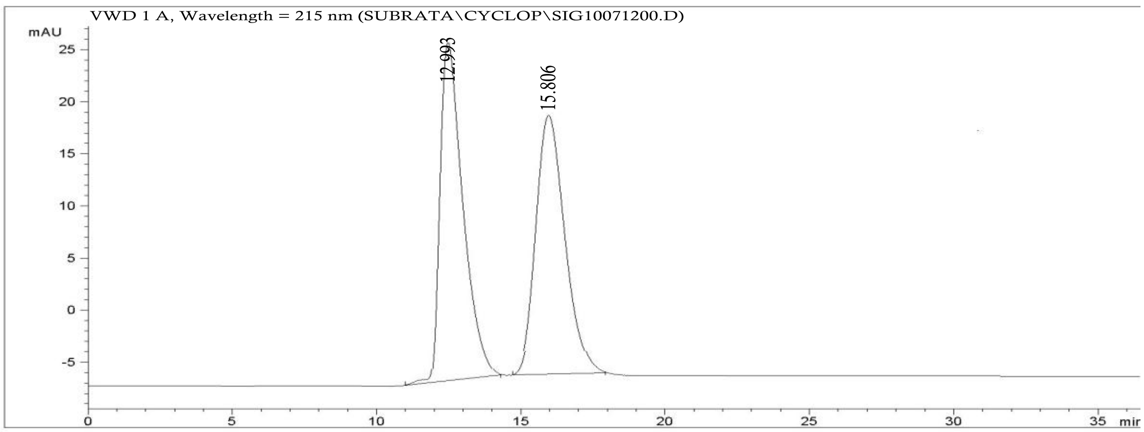
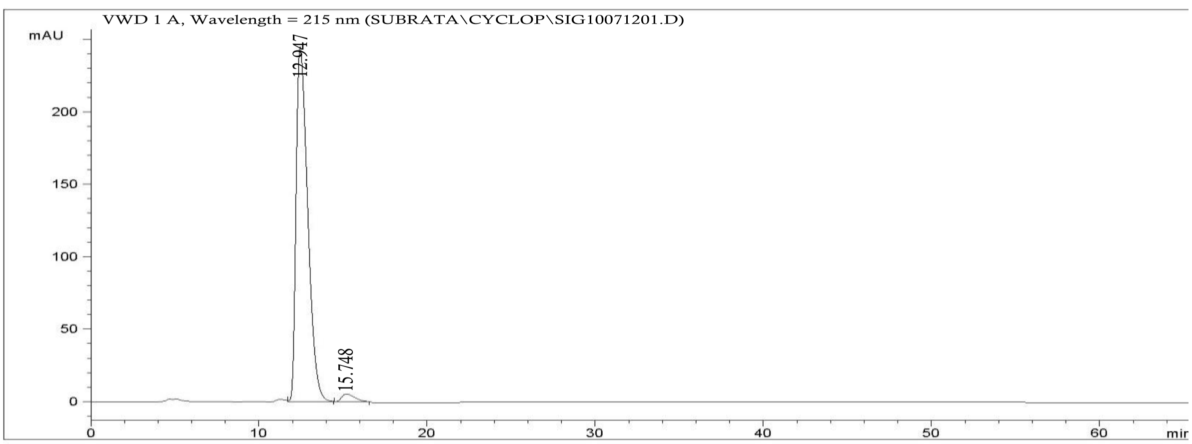
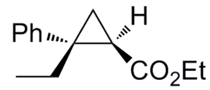


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.993	VB	0.8172	1878.72502	26.53098	51.6084
2	15.806	VB	1.0434	1761.62088	19.82355	48.3916



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.947	BB	0.7656	1.22744e4	244.95091	96.1410
2	15.748	VB	0.7599	492.68168	5.74825	3.8590



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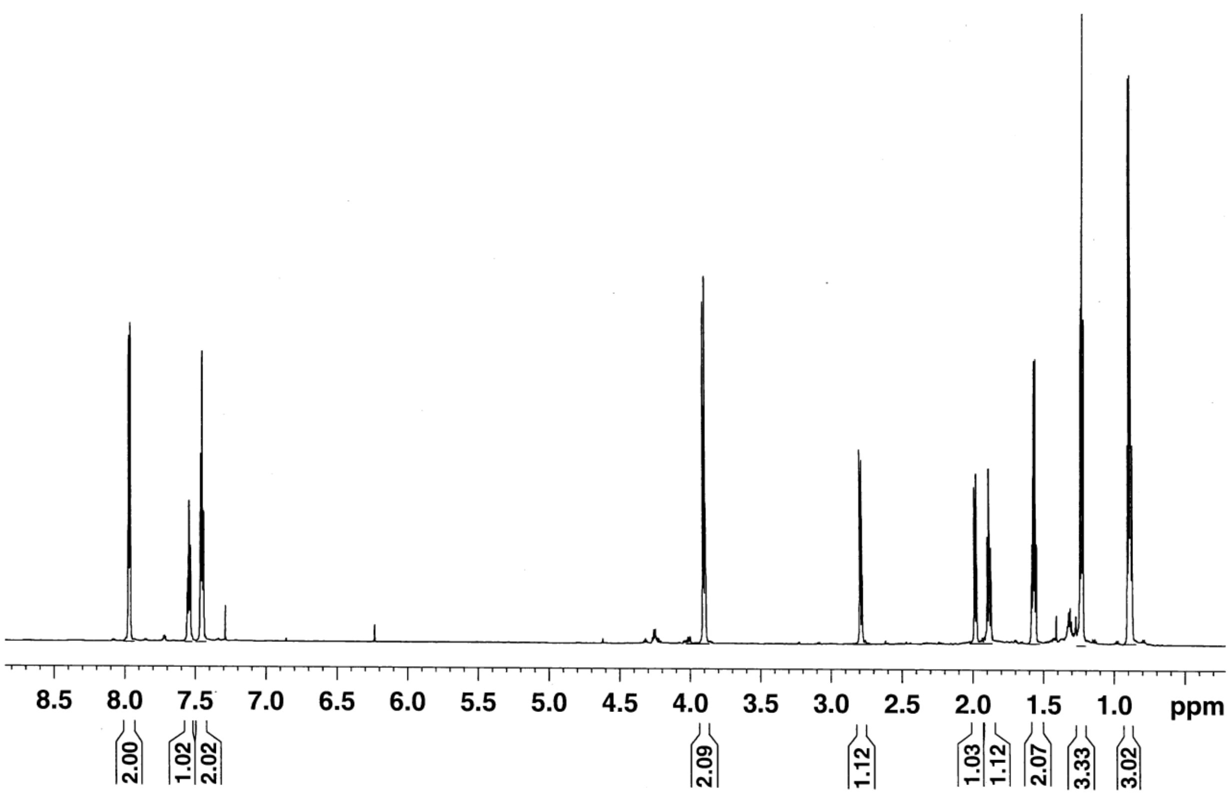
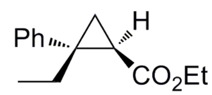


NAME	SS-05-120
EXPNO	1
PROCNO	1
Date_	20131009
Time	20.14
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zg30
TD	95236
SOLVENT	CDCl3
NS	14
DS	2
SWH	11904.762 Hz
FIDRES	0.125003 Hz
AQ	3.9999621 sec
RG	11.3
DW	42.000 use
DE	6.50 use
TE	298.4 K
D1	2.00000000 sec
TD0	1

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

NUC1	1H
P1	9.40 use
PL1	-3.20 dB
PL1W	33.59817505 W
SFO1	700.1516910 MHz
SI	131072
SF	700.1471400 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



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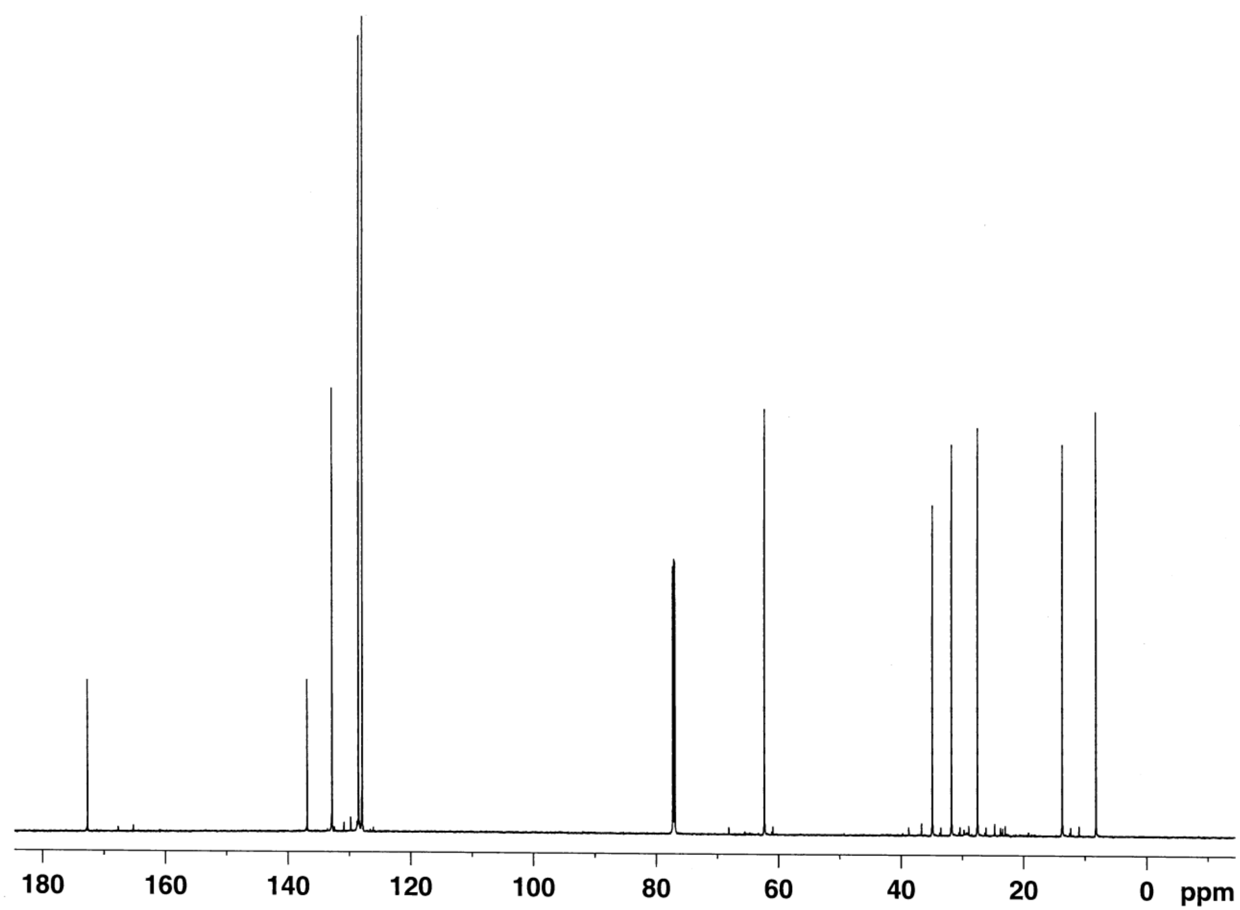
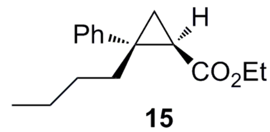
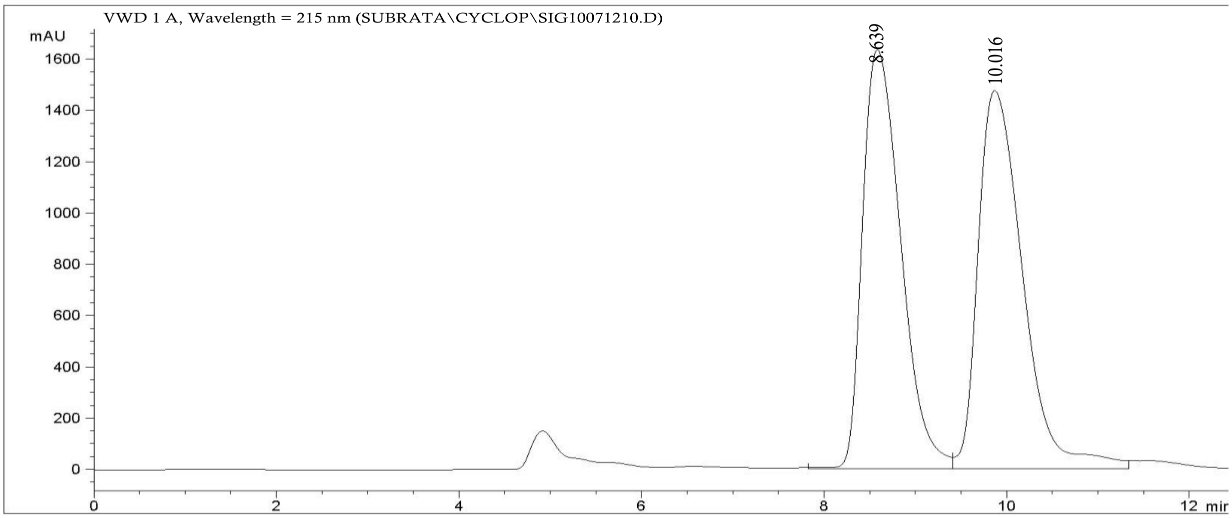


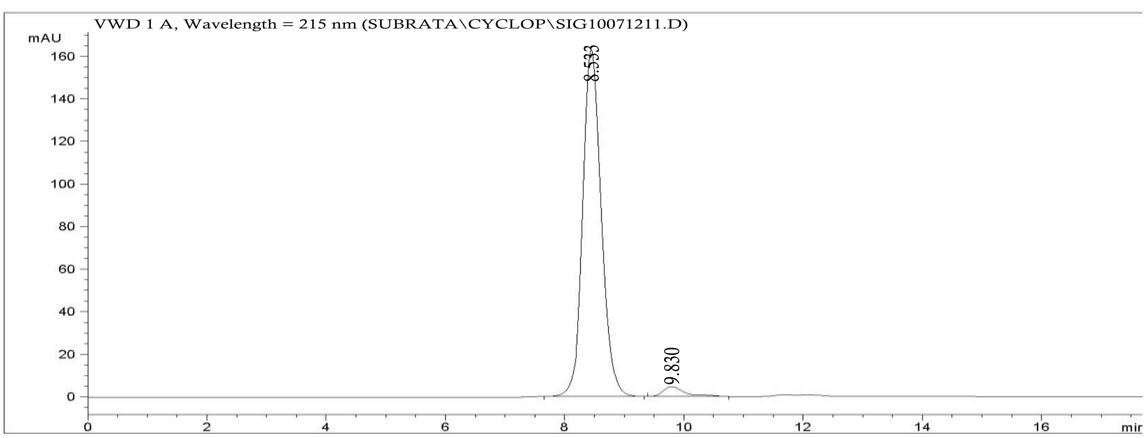
NAME	SS-05-120	
EXPNO	2	
PROCNO	1	
Date_	20131009	
Time	20.17	
INSTRUM	spect	
PROBHD	5 mm CPDCH 13C	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	19	
DS	4	
SWH	41666.668	Hz
FIDRES	0.635783	Hz
AQ	0.7864820	sec
RG	203	
DW	12.000	usec
DE	16.50	usec
TE	298.3	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	9.00	usec
PL1	4.50	dB
PL1W	38.14553833	W
SFO1	176.0697436	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	65.00	usec
PL2	-3.20	dB
PL12	13.60	dB
PL13	120.00	dB
PL2W	33.59817505	W
PL12W	0.70196527	W
PL13W	0.00000000	W
SFO2	700.1499406	MHz
SI	32768	
SF	176.0521380	MHz
WDW	EM	
SSB	0	
LB	3.00	Hz
GB	0	
PC	1.40	



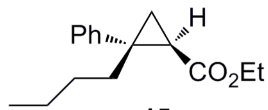
15



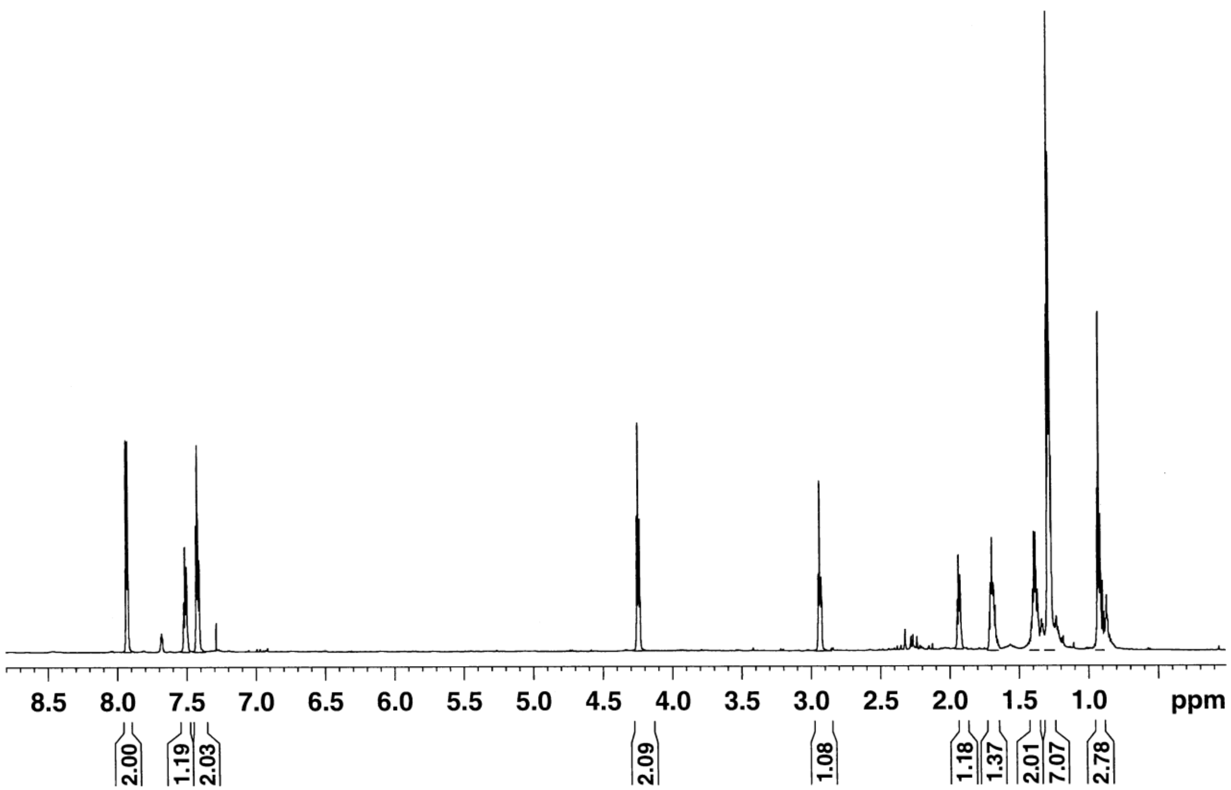
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.639	VB	0.4655	4.79542e4	1636.32328	48.6788
2	10.016	VB	0.5284	5.04875e4	1477.97354	51.3212



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.533	VB	0.3335	2999.53722	162.79829	95.0048
2	9.830	BB	0.3877	157.71085	4.62816	4.9952

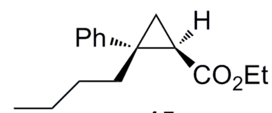


15

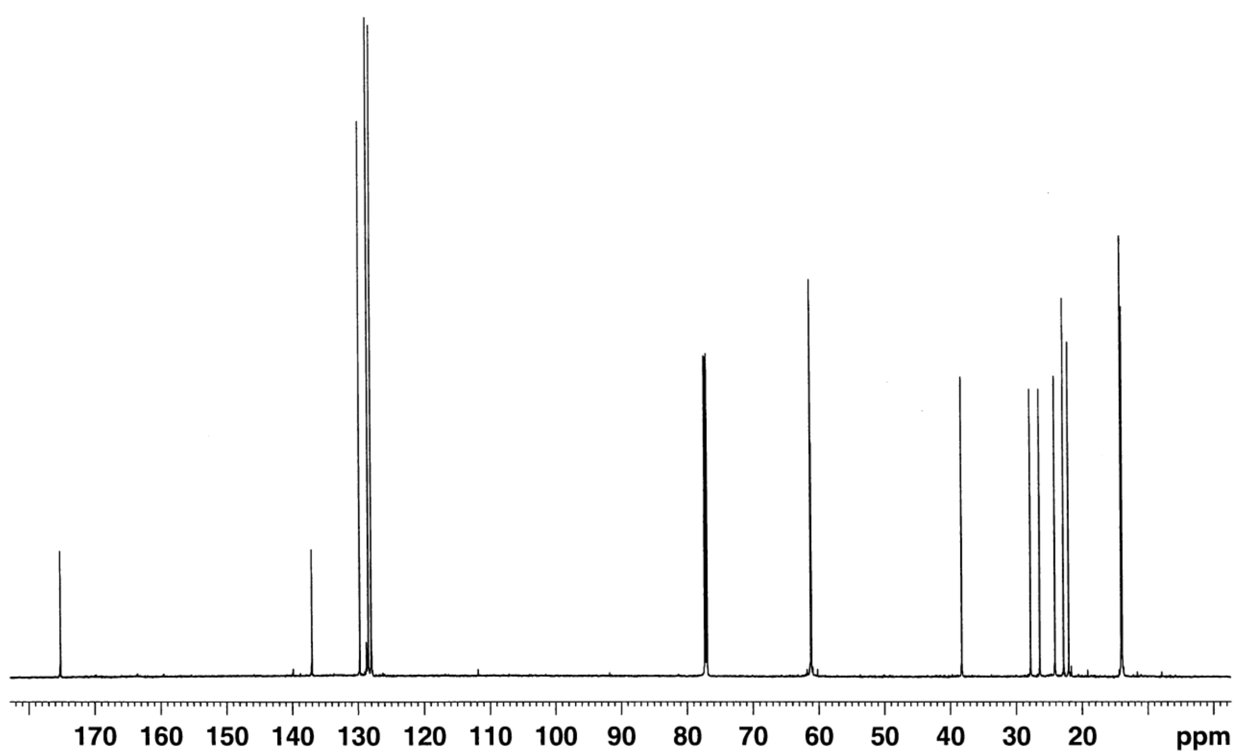



```
NAME          SS-05-121
EXPNO                 1
PROCNO                1
Date_          20131009
Time              20.21
INSTRUM           spect
PROBHD   5 mm CPDCH 13C
PULPROG            zg30
TD                95236
SOLVENT           CDCl3
NS                   28
DS                    2
SWH          11904.762 Hz
FIDRES        0.125003 Hz
AQ           3.9999621 sec
RG                   10
DW              42.000 usec
DE                6.50 usec
TE               298.2 K
D1          2.00000000 sec
TD0                   1

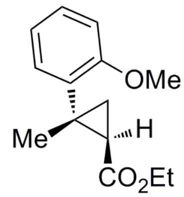
======== CHANNEL f1 ========
NUC1                 1H
P1                 9.40 usec
PL1               -3.20 dB
PL1W        33.59817505 W
SFO1        700.1516910 MHz
SI               131072
SF          700.1471400 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



15



NAME	SS-05-121
EXPNO	2
PROCNO	1
Date_	20131009
Time	20.26
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	32
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 sec
RG	203
DW	12.000 use
DE	16.50 use
TE	298.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 =======	
NUC1	13C
P1	9.00 use
PL1	4.50 dB
PL1W	38.14553833 W
SFO1	176.0697436 MHz
======== CHANNEL f2 =======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 use
PL2	-3.20 dB
PL12	13.60 dB
PL13	120.00 dB
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1499406 MHz
SI	32768
SF	176.0521380 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40



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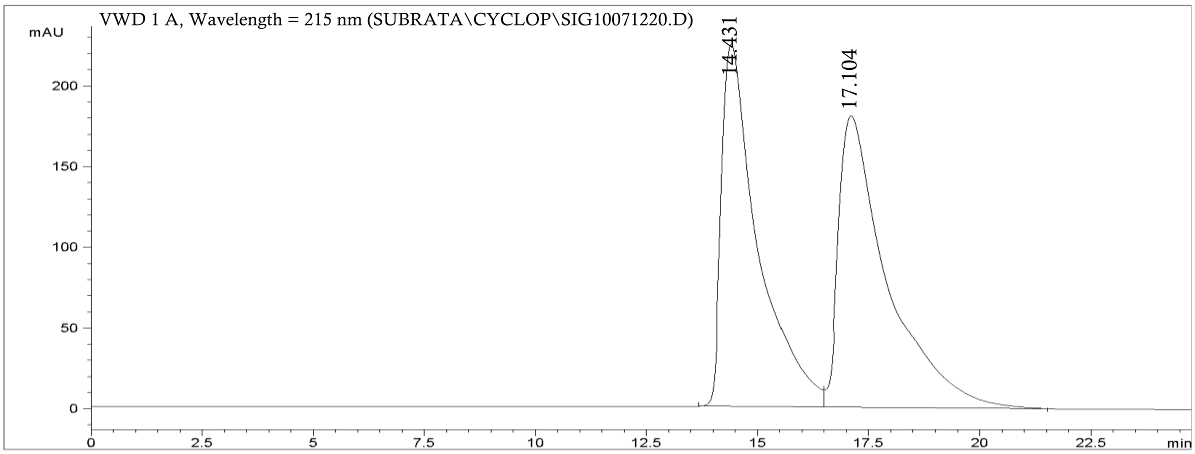
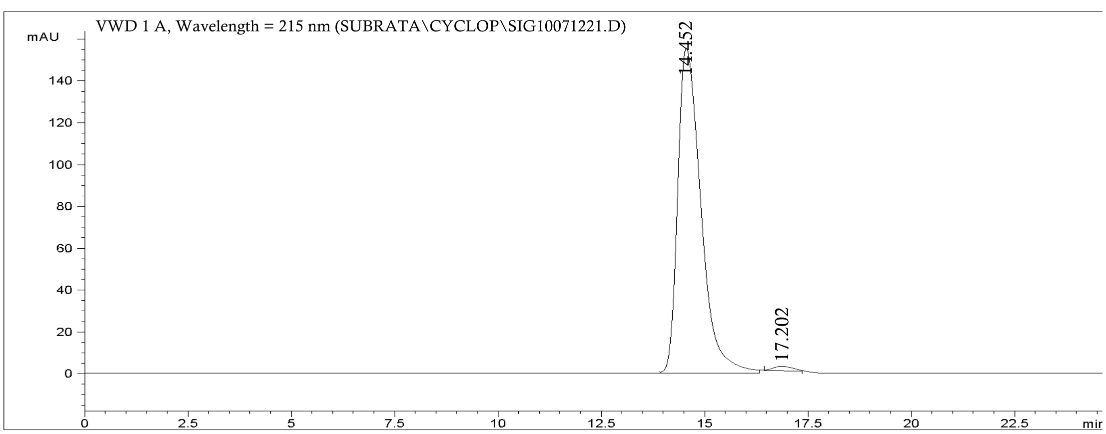
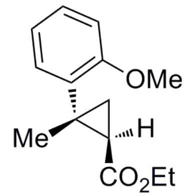


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.431	BV	0.8233	1.28534e4	224.65383	48.2447
2	17.104	VB	1.0356	1.37853e4	180.28644	51.7553



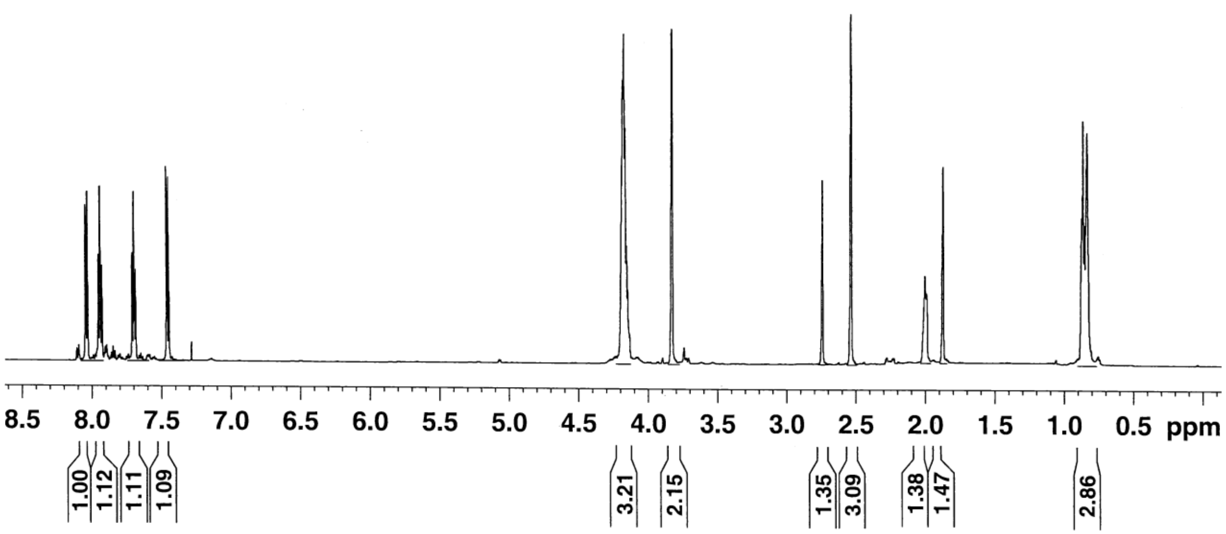
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.452	VB	0.6122	8645.42608	155.16352	98.0968
2	17.202	VB	0.5777	167.73202	2.67524	1.9032

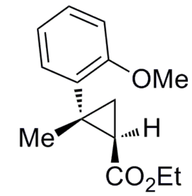


16

NAME	SS-05-122
EXPNO	1
PROCNO	1
Date_	20131009
Time	20.35
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zg30
TD	95236
SOLVENT	CDCl3
NS	32
DS	2
SWH	11904.762 Hz
FIDRES	0.125003 Hz
AQ	3.9999621 sec
RG	7.12
DW	42.000 usec
DE	6.50 usec
TE	298.2 K
D1	2.00000000 sec
TD0	1

======== CHANNEL f1 ========	
NUC1	1H
P1	9.40 usec
PL1	-3.20 dB
PL1W	33.59817505 W
SFO1	700.1516910 MHz
SI	131072
SF	700.1471400 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00





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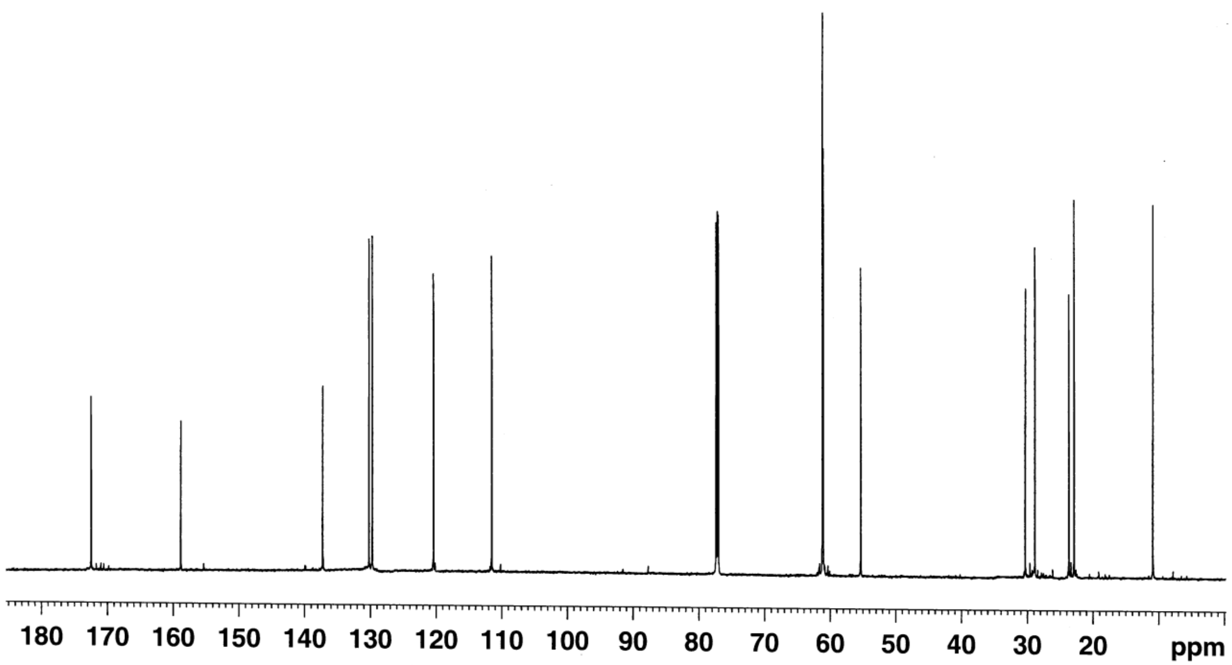
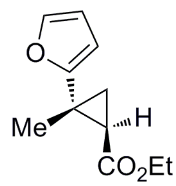



```
NAME            SS-05-122
EXPNO                   2
PROCNO                  1
Date_            20131009
Time                20.41
INSTRUM             spect
PROBHD   5 mm CPDCH 13C
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     33
DS                      4
SWH             41666.668 Hz
FIDRES           0.635783 Hz
AQ              0.7864820 sec
RG                    203
DW                 12.000 use
DE                  16.50 use
TE                  298.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 =======
NUC1                  13C
P1                   9.00 use
PL1                  4.50 dB
PL1W          38.14553833 W
SFO1          176.0697436 MHz

======== CHANNEL f2 =======
CPDPRG2           waltz16
NUC2                   1H
PCPD2               65.00 use
PL2                 -3.20 dB
PL12                13.60 dB
PL13               120.00 dB
PL2W          33.59817505 W
PL12W          0.70196527 W
PL13W          0.00000000 W
SFO2          700.1499406 MHz
SI                  32768
SF            176.0521380 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.40
```



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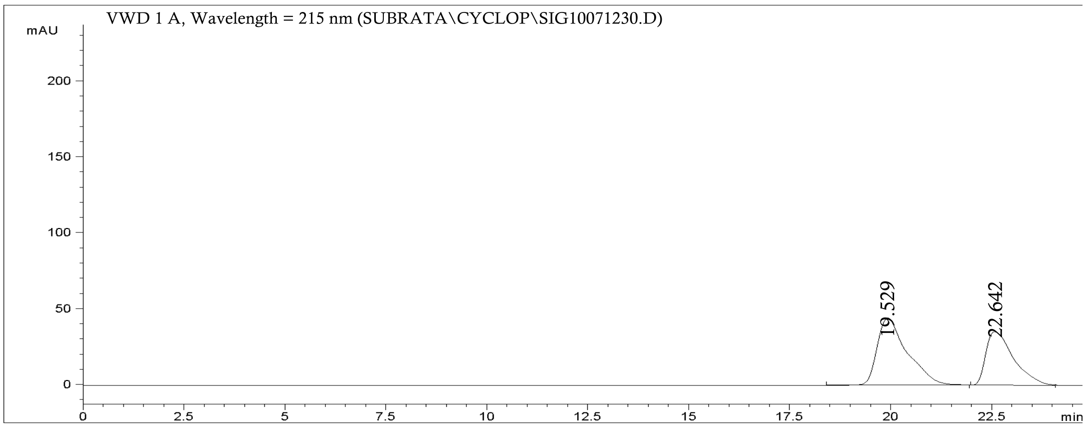
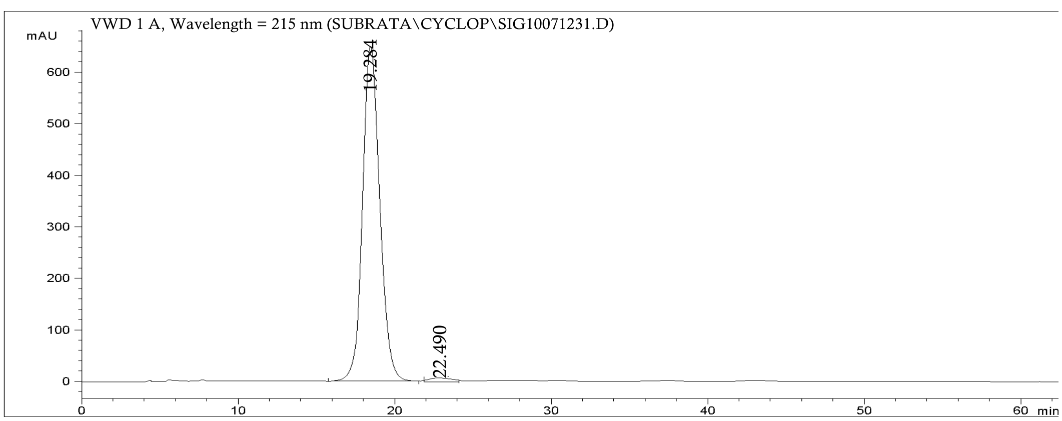
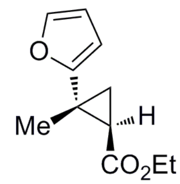


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	19.529	BB	0.7988	1067.36854	48.10531	55.5083
2	22.642	VB	0.7633	855.52073	42.60073	44.4917



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	19.284	BB	1.2692	5850.33187	645.43922	96.2231
2	22.490	VB	0.8973	229.63424	9.03535	3.7769



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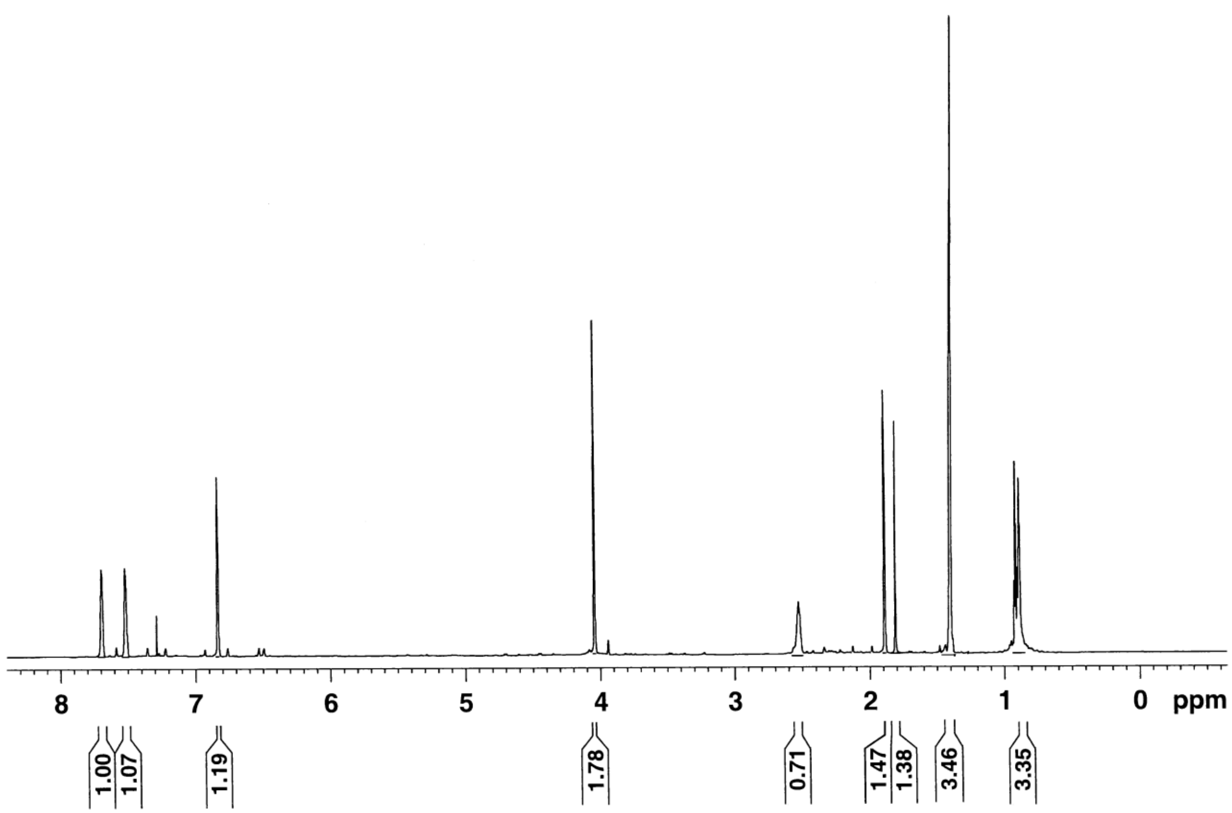
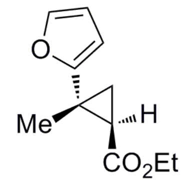
NAME	SS-05-123
EXPNO	1
PROCNO	1
Date_	20131009
Time	20.49
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zg30
TD	95236
SOLVENT	CDCl3
NS	13
DS	2
SWH	11904.762 Hz
FIDRES	0.125003 Hz
AQ	3.9999621 sec
RG	12.7
DW	42.000 usec
DE	6.50 usec
TE	298.2 K
D1	2.00000000 sec
TD0	1

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

NUC1	1H
P1	9.40 usec
PL1	-3.20 dB
PL1W	33.59817505 W
SFO1	700.1516910 MHz
SI	131072
SF	700.1471400 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00





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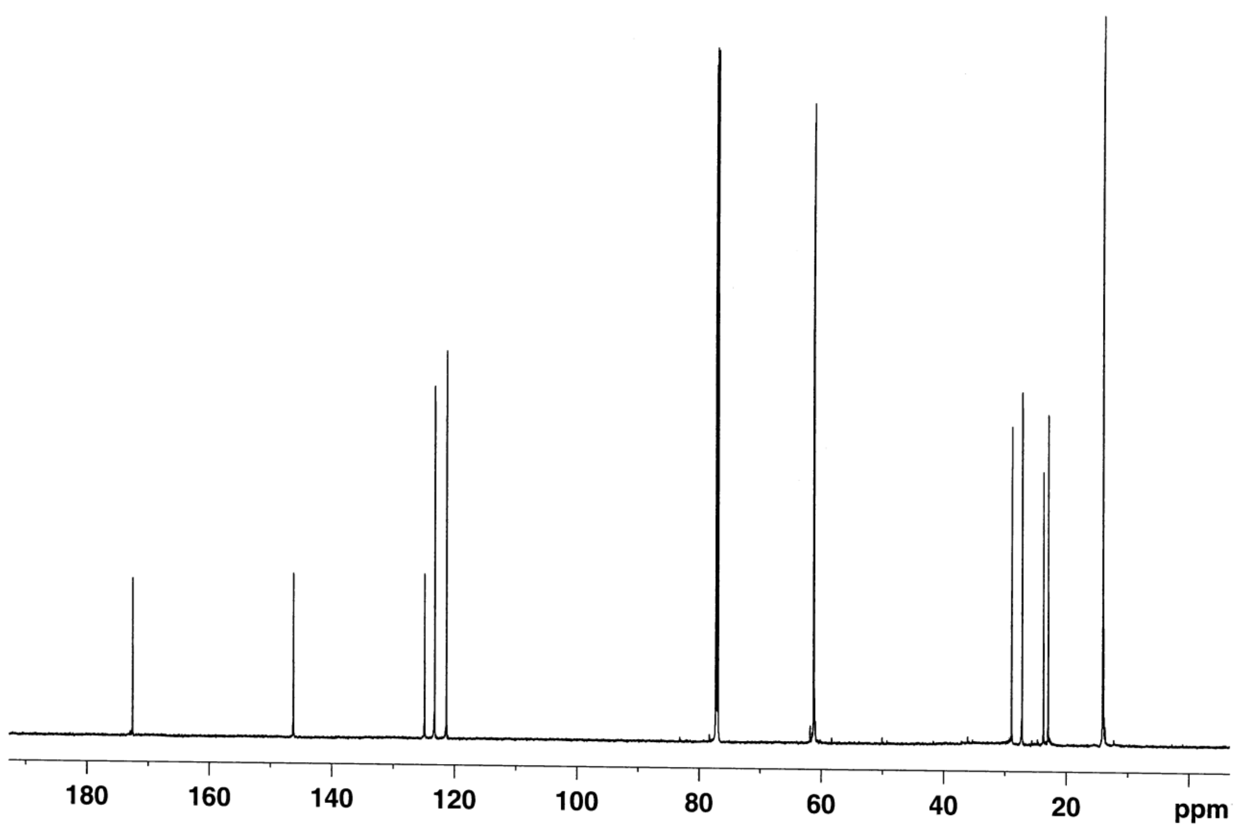
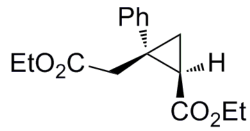


NAME	SS-05-123
EXPNO	2
PROCNO	1
Date_	20131009
Time	20.55
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	83
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 sec
RG	203
DW	12.000 use
DE	16.50 use
TE	298.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 =======	
NUC1	13C
P1	9.00 use
PL1	4.50 dB
PL1W	38.14553833 W
SFO1	176.0697436 MHz
======== CHANNEL f2 =======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 use
PL2	-3.20 dB
PL12	13.60 dB
PL13	120.00 dB
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1499406 MHz
SI	32768
SF	176.0521380 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40



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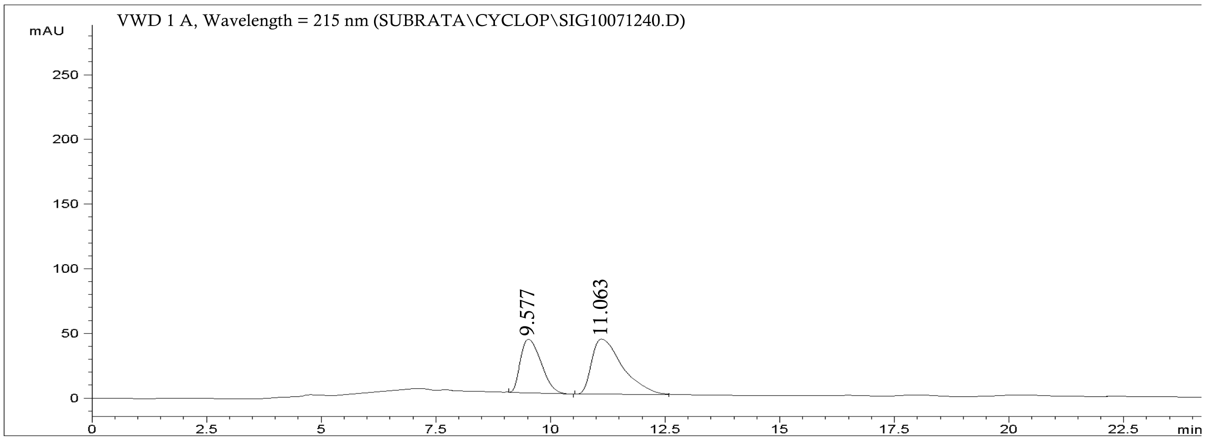
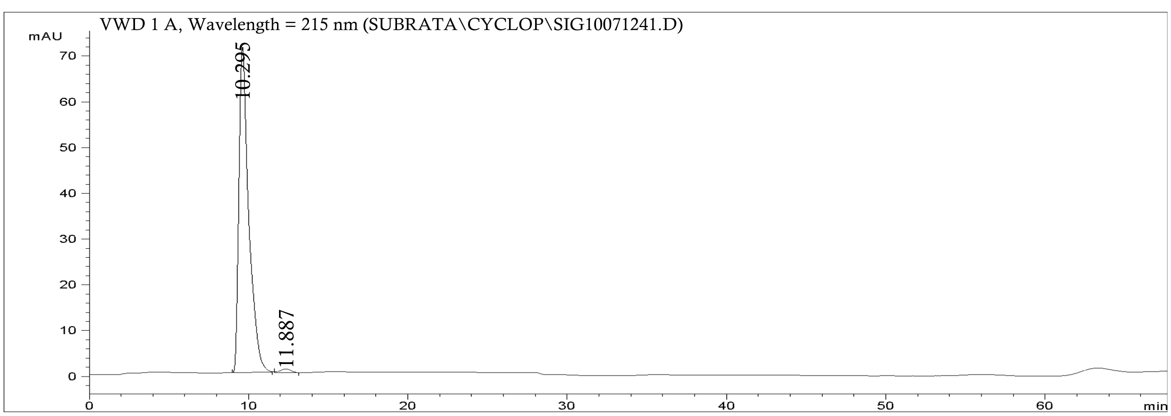
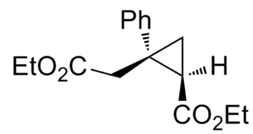


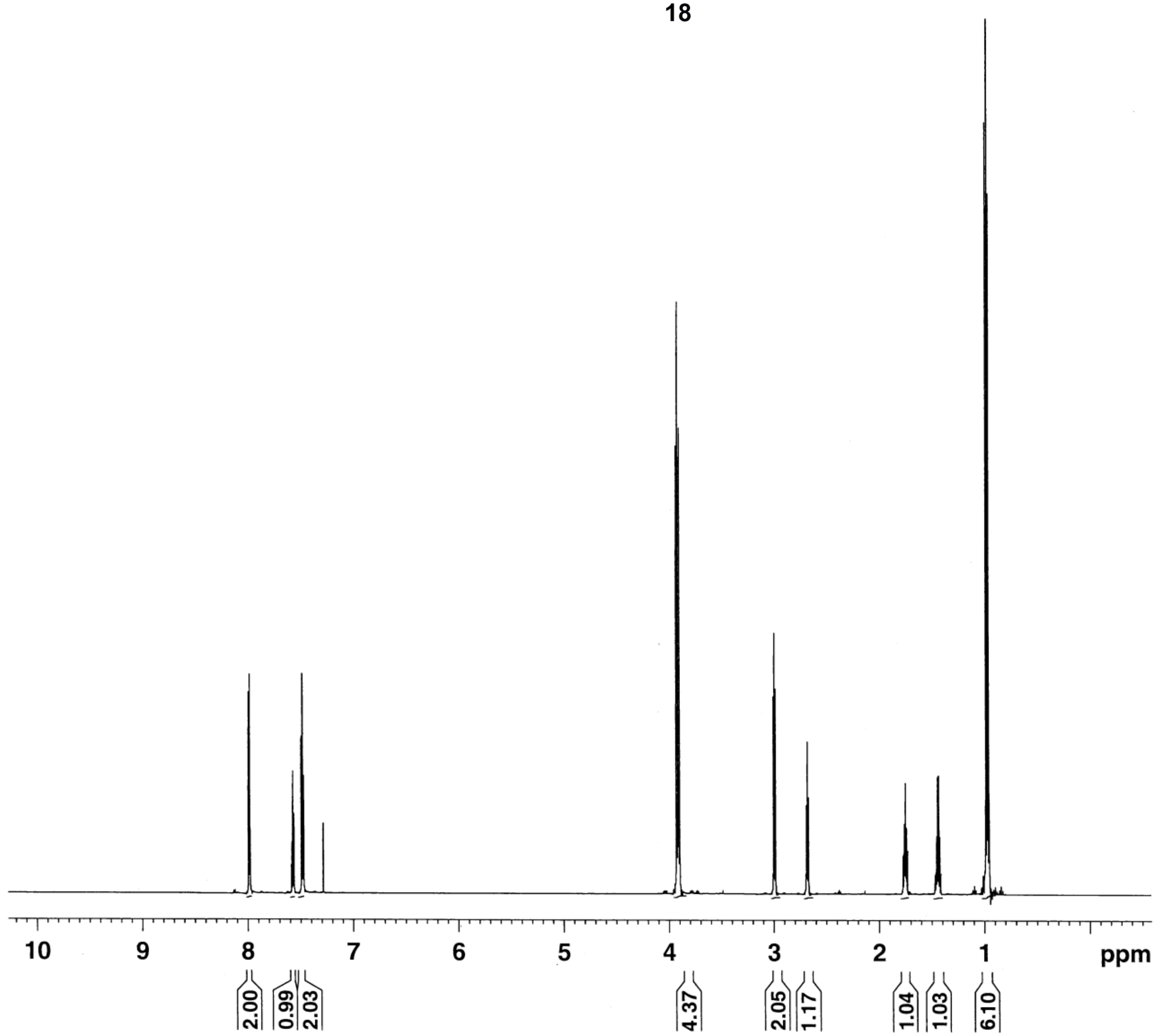
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.577	BB	0.5193	1845.65065	41.59241	48.9333
2	11.063	VB	0.7344	2015.63928	42.69965	51.0667



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.295	VB	0.6421	1171.60377	71.34489	95.4481
2	11.887	BB	0.0942	55.87354	0.36073	4.5519

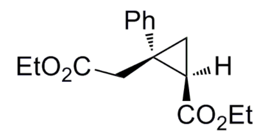


18

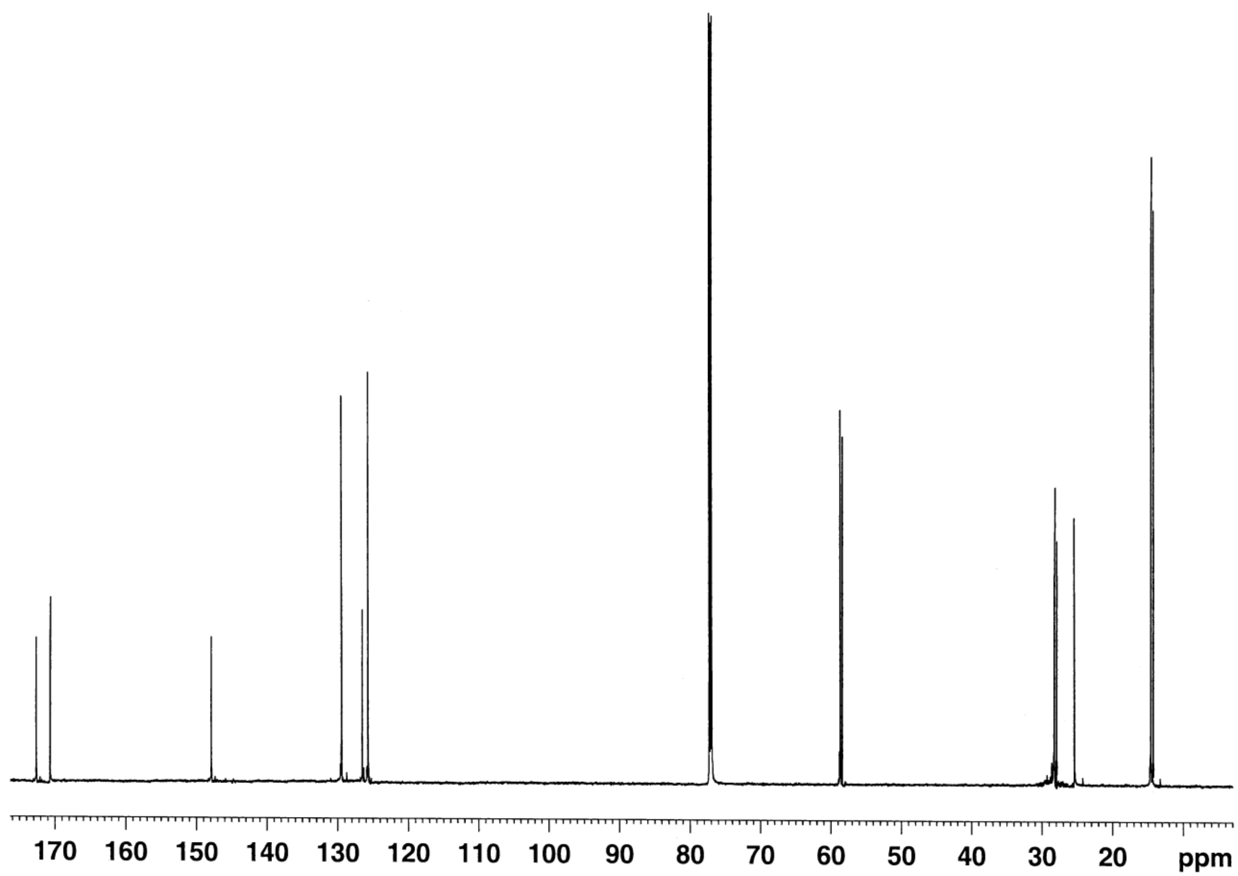



```
NAME          SS-05-124
EXPNO                 1
PROCNO                1
Date_          20140106
Time              20.19
INSTRUM           spect
PROBHD   5 mm CPDCH 13C
PULPROG            zg30
TD                95236
SOLVENT           CDCl3
NS                   13
DS                    2
SWH           11904.762 Hz
FIDRES         0.125003 Hz
AQ            3.9999621 sec
RG                 20.2
DW               42.000 usec
DE                 6.50 usec
TE                298.2 K
D1           2.00000000 sec
TD0                   1

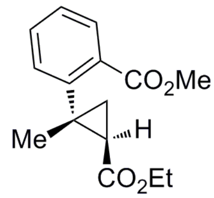
======== CHANNEL f1 ========
NUC1                 1H
P1                 9.40 usec
PL1               -3.20 dB
PL1W        33.59817505 W
SFO1        700.1516910 MHz
SI               131072
SF          700.1471400 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



18



NAME	SS-05-138
EXPNO	2
PROCNO	1
Date_	20140106
Time	20.43
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	52
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 sec
RG	203
DW	12.000 usec
DE	16.50 usec
TE	298.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.00 usec
PL1	4.50 dB
PL1W	38.14553833 W
SFO1	176.0697436 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 usec
PL2	-3.20 dB
PL12	13.60 dB
PL13	120.00 dB
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1499406 MHz
SI	32768
SF	176.0521380 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40



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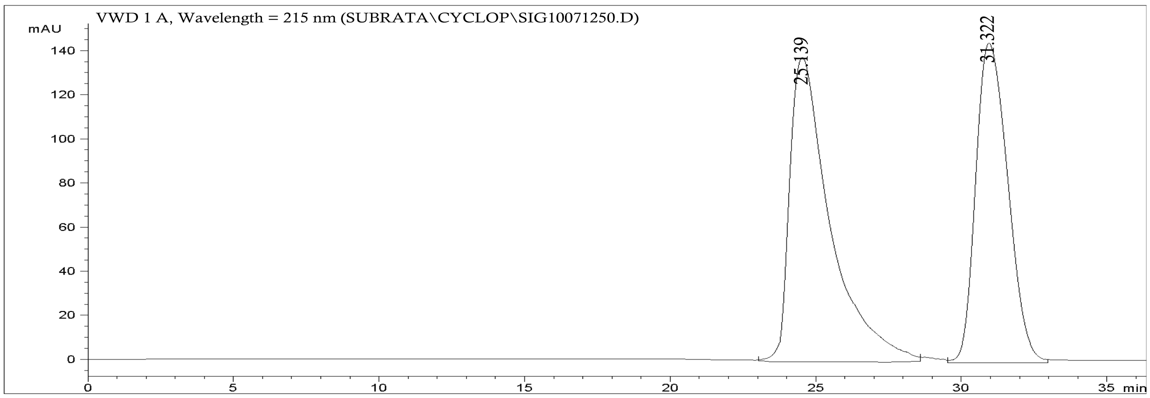
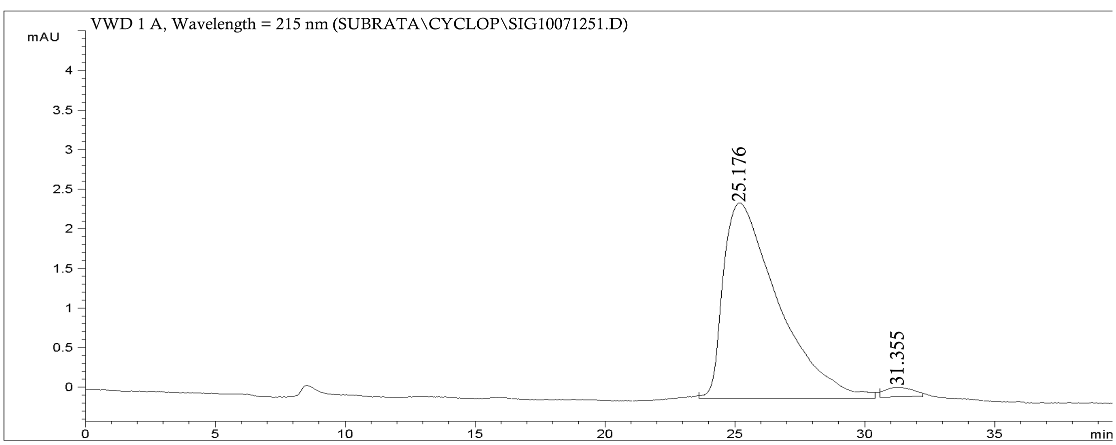
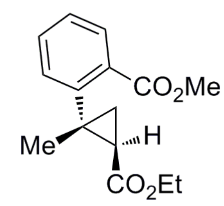


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	25.139	VB	0.9678	4689.28931	136.97383	50.8633
2	31.322	BB	0.3753	4530.10192	143.18244	49.1367



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	25.176	BB	1.1236	659.83539	2.54536	97.6133
2	31.355	VB	0.3925	16.12112	0.13628	2.3867



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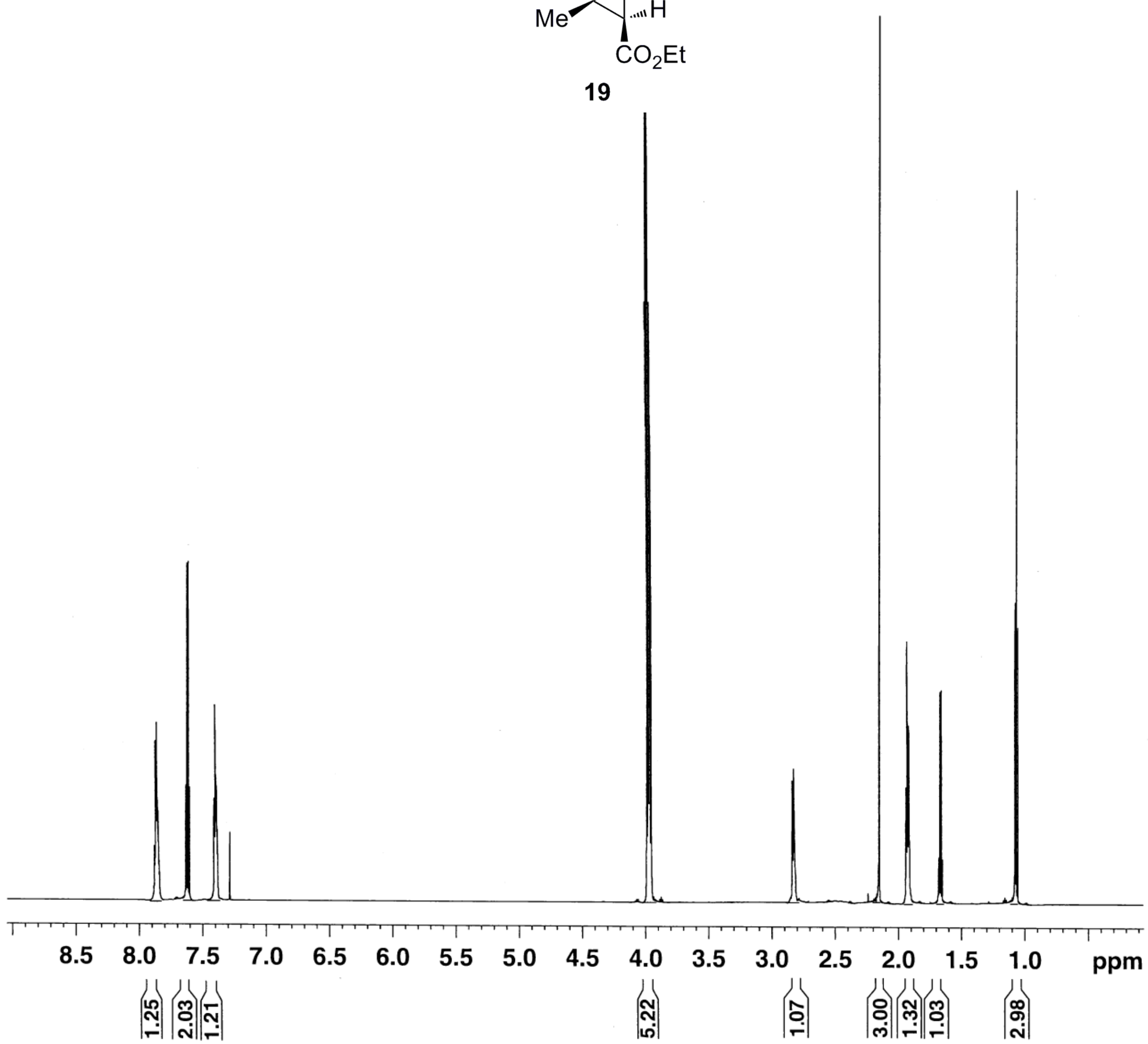
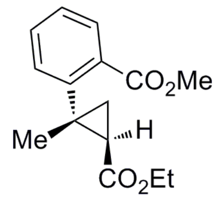



```
NAME             SS-05-135
EXPNO                    3
PROCNO                   1
Date_             20140106
Time                 19.17
INSTRUM              spect
PROBHD     5 mm CPDCH 13C
PULPROG               zg30
TD                   95236
SOLVENT              CDCl3
NS                      11
DS                       2
SWH              11904.762 Hz
FIDRES            0.125003 Hz
AQ               3.9999621 sec
RG                    22.6
DW                  42.000 usec
DE                    6.50 usec
TE                   298.2 K
D1              2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                    9.40 usec
PL1                  -3.20 dB
PL1W           33.59817505 W
SFO1           700.1516910 MHz
SI                  131072
SF             700.1471400 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



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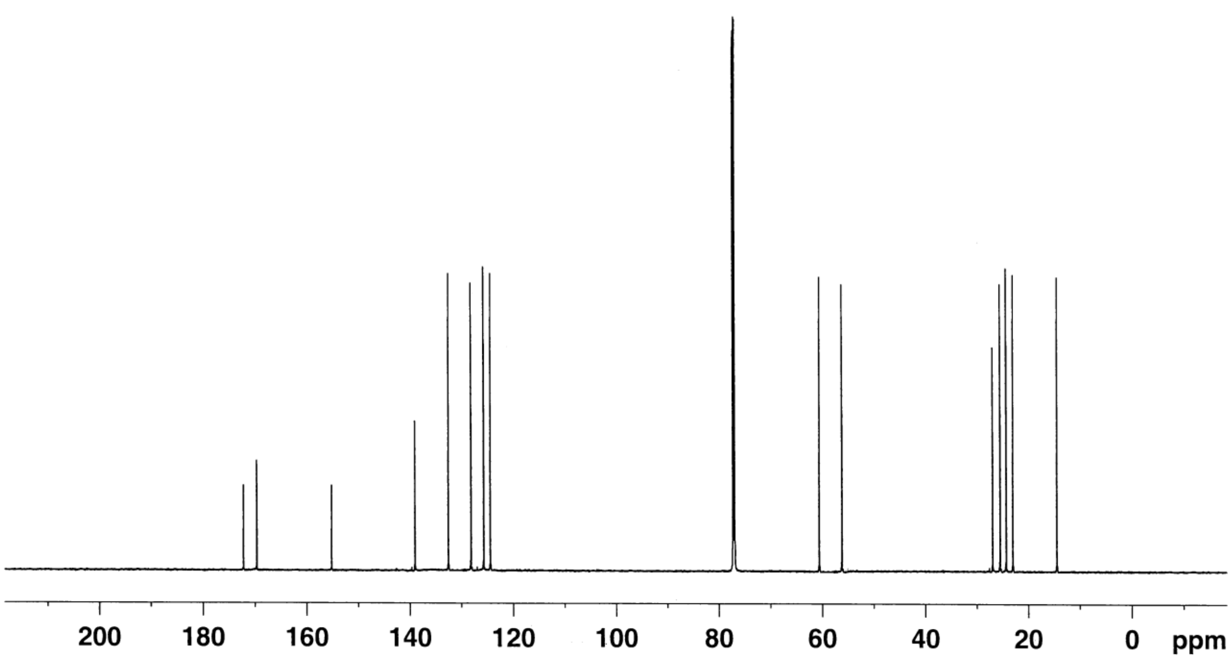
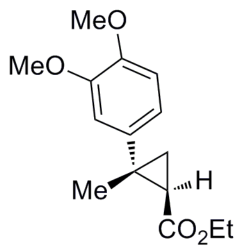


NAME	SS-05-135
EXPNO	2
PROCNO	1
Date_	20140106
Time	19.12
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	58
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 sec
RG	203
DW	12.000 use
DE	16.50 use
TE	298.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 =======	
NUC1	13C
P1	9.00 use
PL1	4.50 dB
PL1W	38.14553833 W
SFO1	176.0697436 MHz
======== CHANNEL f2 =======	
CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 use
PL2	-3.20 dB
PL12	13.60 dB
PL13	120.00 dB
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1499406 MHz
SI	32768
SF	176.0521380 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40



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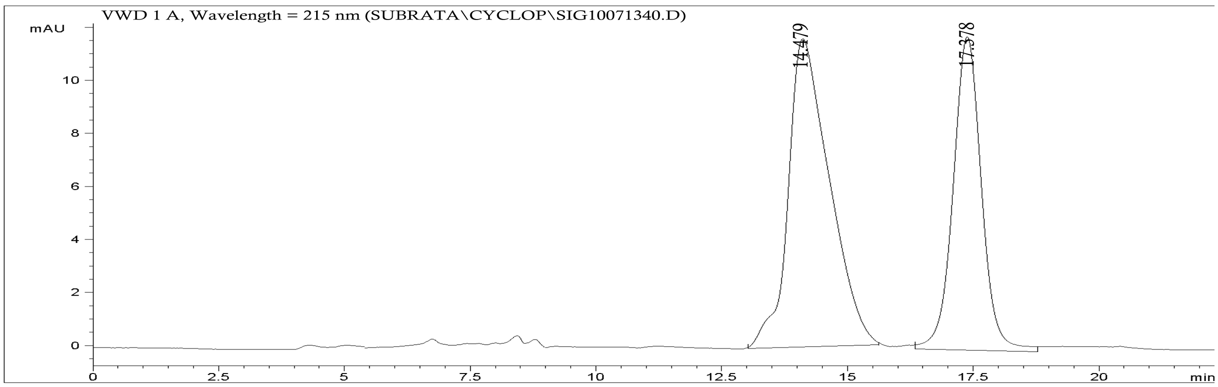
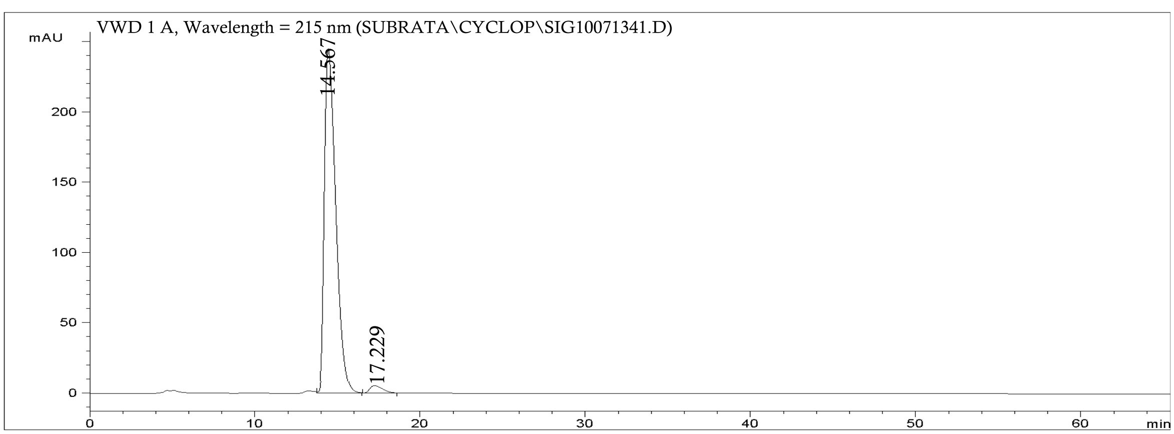
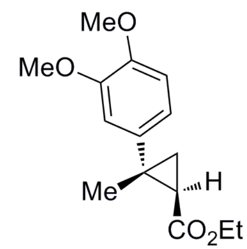


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.479	BB	0.7619	1953.03871	11.53826	54.2109
2	17.378	VB	0.4011	1649.62861	11.19861	45.7891



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.567	BB	0.6882	8674.72143	238.97346	96.8427
2	17.229	VB	0.6509	282.81634	9.94255	3.1573



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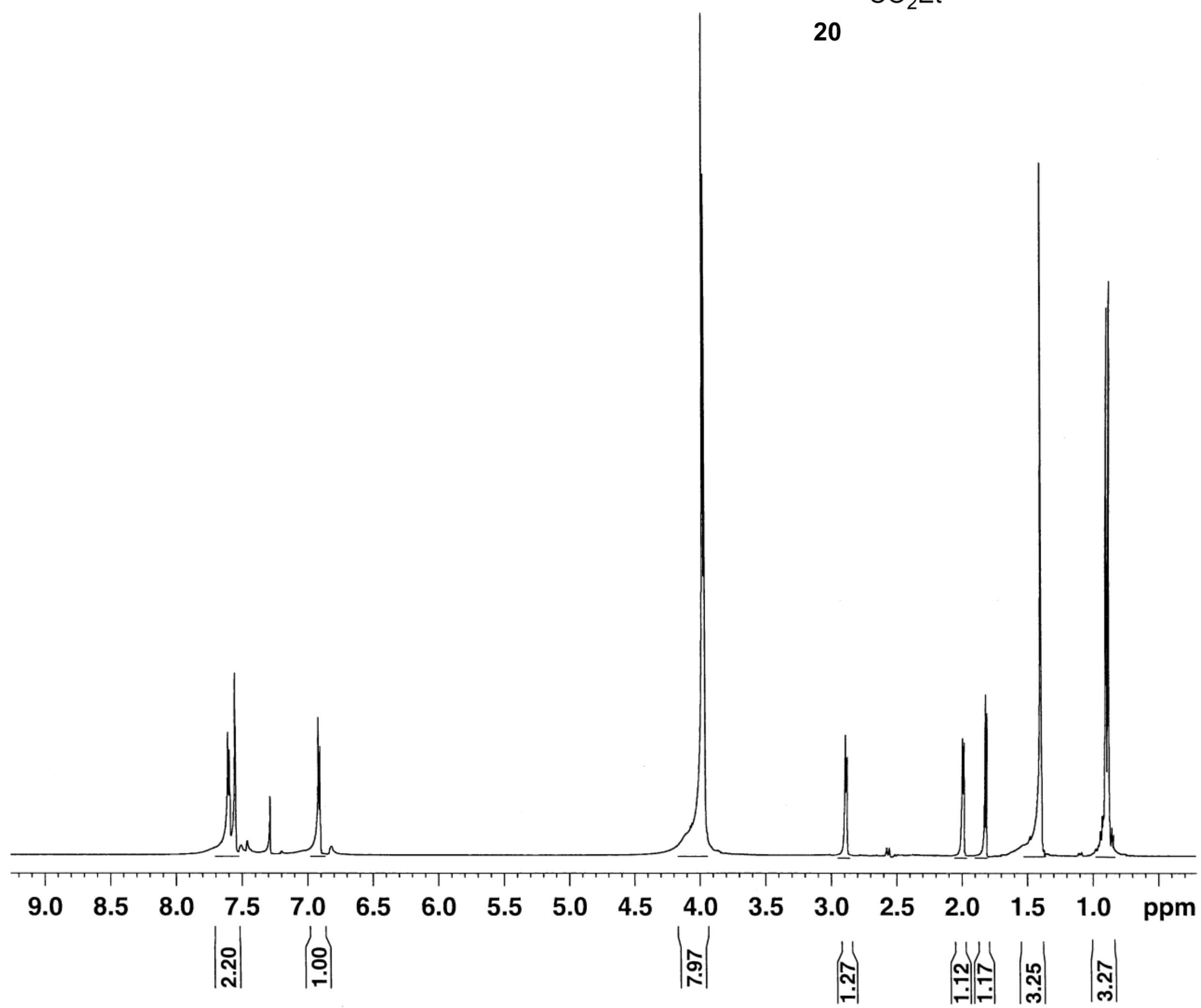
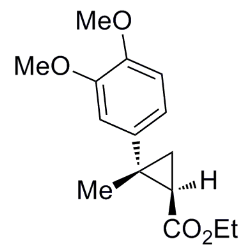
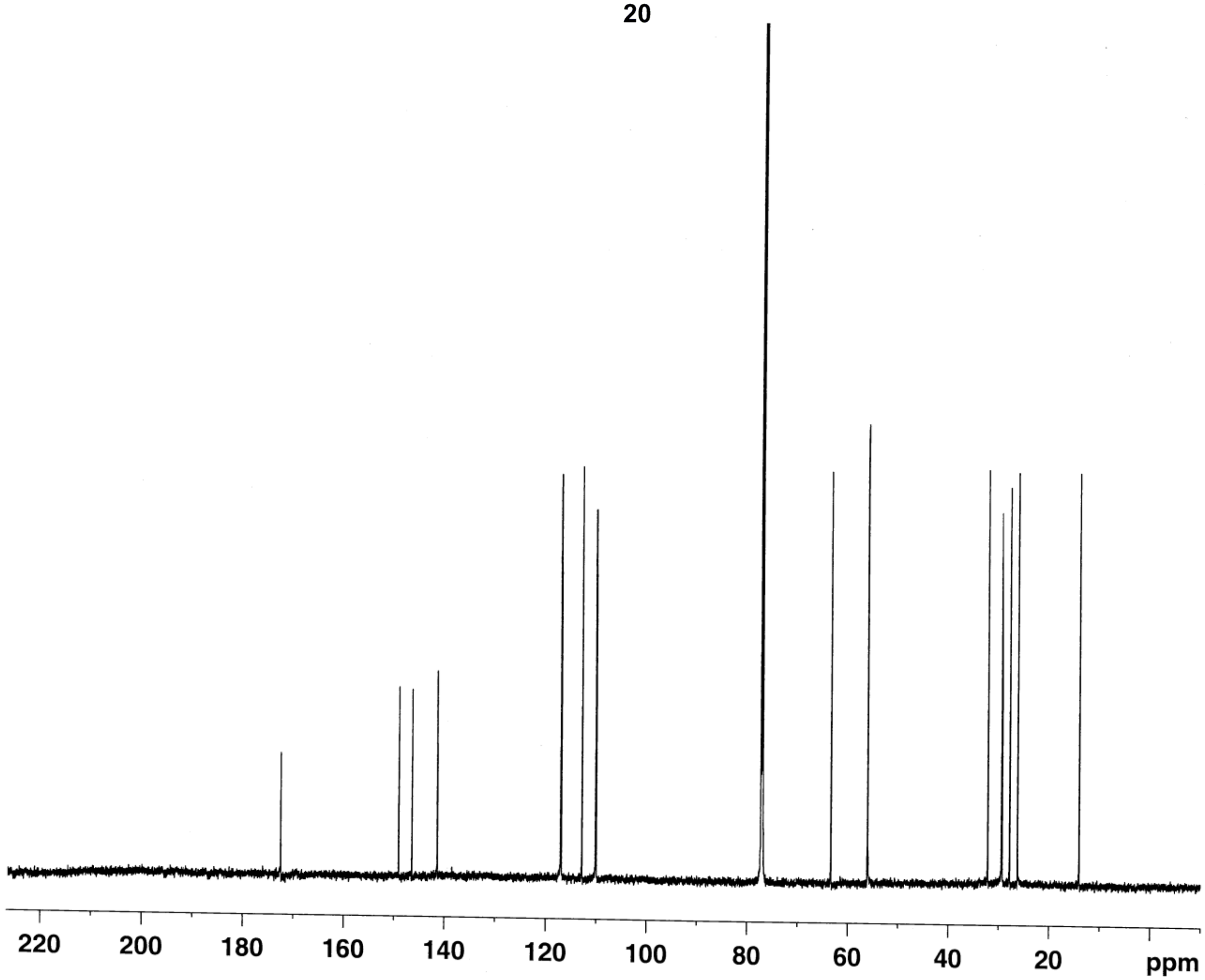



```
NAME          SS-05-134
EXPNO                 1
PROCNO                1
Date_          20140106
Time              20.02
INSTRUM           spect
PROBHD   5 mm CPDCH 13C
PULPROG            zg30
TD                95236
SOLVENT           CDCl3
NS                    9
DS                    2
SWH           11904.762 Hz
FIDRES         0.125003 Hz
AQ            3.9999621 sec
RG                   32
DW               42.000 usec
DE                 6.50 usec
TE                298.2 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.40 usec
PL1               -3.20 dB
PL1W        33.59817505 W
SFO1        700.1516910 MHz
SI               131072
SF          700.1471400 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



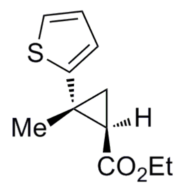
20




```
NAME              SS-05-134
EXPNO                     2
PROCNO                    1
Date_              20140106
Time                  20.06
INSTRUM               spect
PROBHD      5 mm CPDCH 13C
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       48
DS                        4
SWH              41666.668 Hz
FIDRES            0.635783 Hz
AQ               0.7864820 sec
RG                      203
DW                   12.000 use
DE                   16.50 use
TE                   298.2 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 =======
NUC1                    13C
P1                     9.00 use
PL1                    4.50 dB
PL1W            38.14553833 W
SFO1            176.0697436 MHz

======== CHANNEL f2 =======
CPDPRG2             waltz16
NUC2                     1H
PCPD2                 65.00 use
PL2                   -3.20 dB
PL12                  13.60 dB
PL13                 120.00 dB
PL2W            33.59817505 W
PL12W            0.70196527 W
PL13W            0.00000000 W
SFO2            700.1499406 MHz
SI                    32768
SF              176.0521380 MHz
WDW                      EM
SSB                       0
LB                     3.00 Hz
GB                        0
PC                     1.40
```



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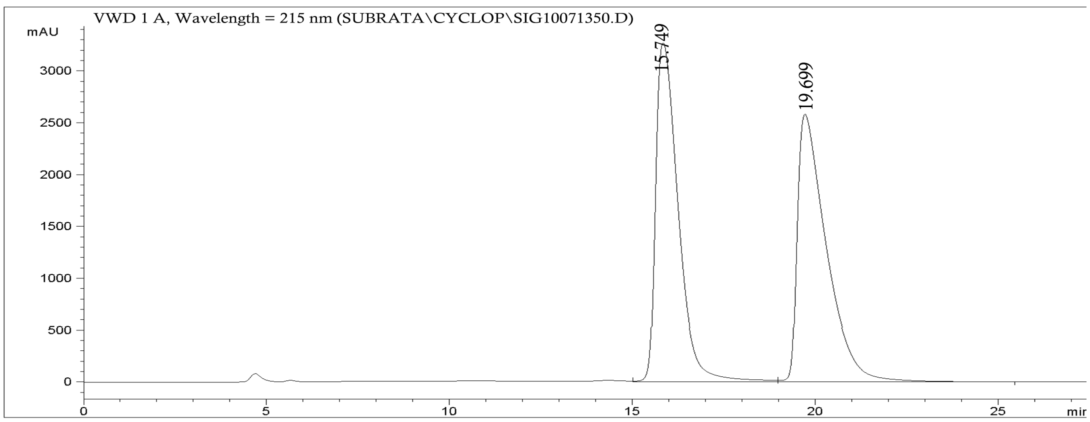
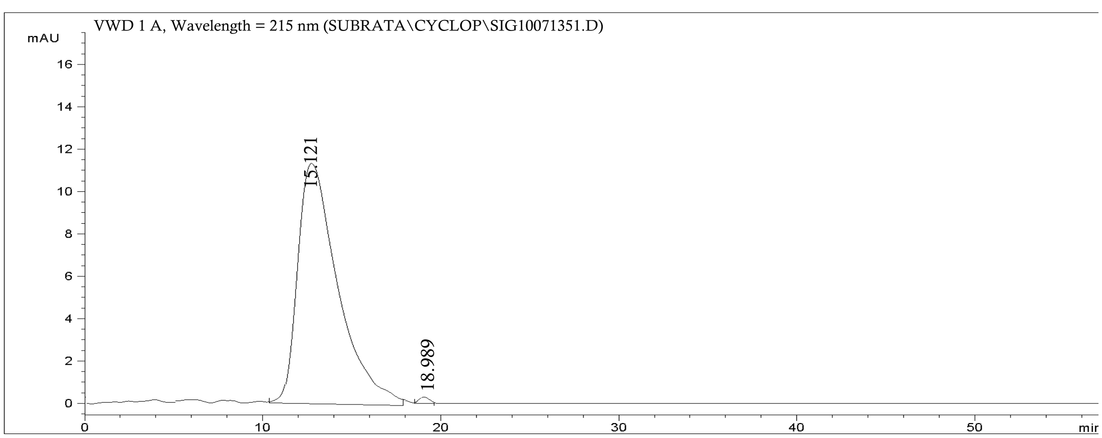
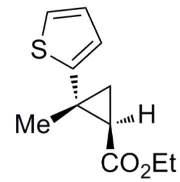


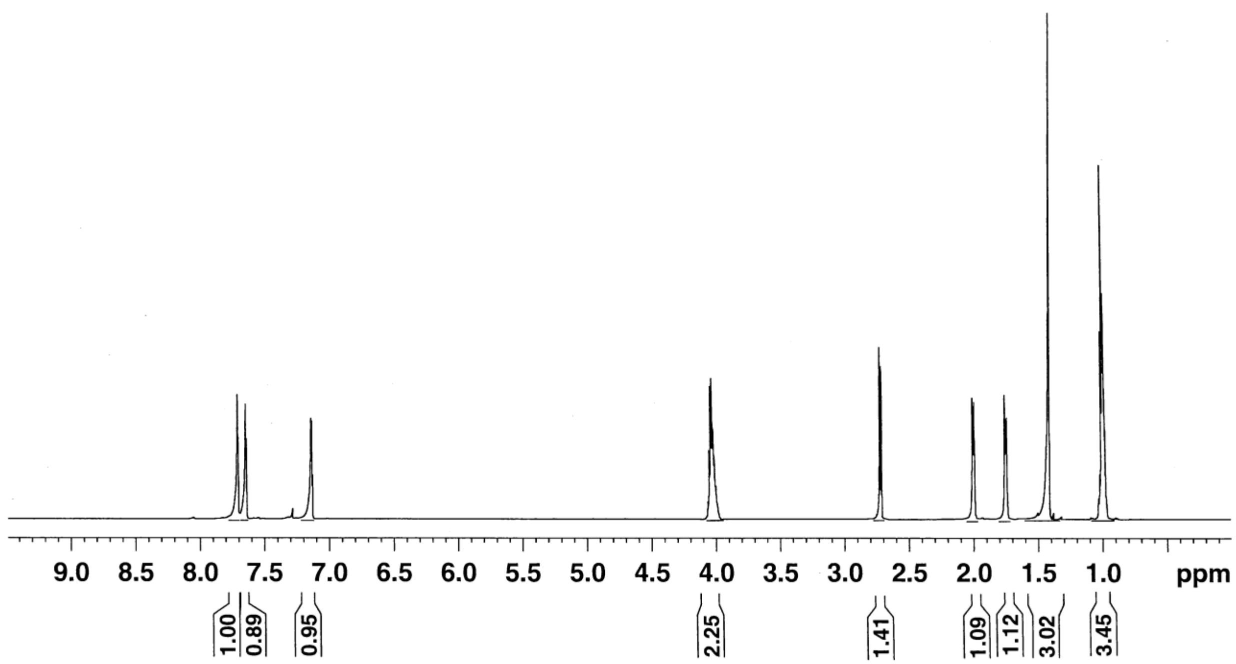
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	15.749	VV	0.6481	1.40223e5	3269.96245	49.5932
2	19.699	VB	0.8073	1.42523e5	2568.60079	50.4068



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	15.121	BB	2.6793	1291.94814	11.42963	95.0792
2	18.989	VB	0.0166	66.86445	0.10436	4.9208



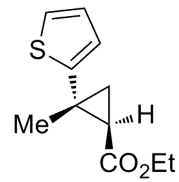
21



NAME	SS-05-135
EXPNO	1
PROCNO	1
Date_	20140106
Time	19.08
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zg30
TD	95236
SOLVENT	CDCl3
NS	16
DS	2
SWH	11904.762 Hz
FIDRES	0.125003 Hz
AQ	3.9999621 sec
RG	28.5
DW	42.000 usec
DE	6.50 usec
TE	298.1 K
D1	2.00000000 sec
TD0	1

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

NUC1	1H
P1	9.40 usec
PL1	-3.20 dB
PL1W	33.59817505 W
SFO1	700.1516910 MHz
SI	131072
SF	700.1471400 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



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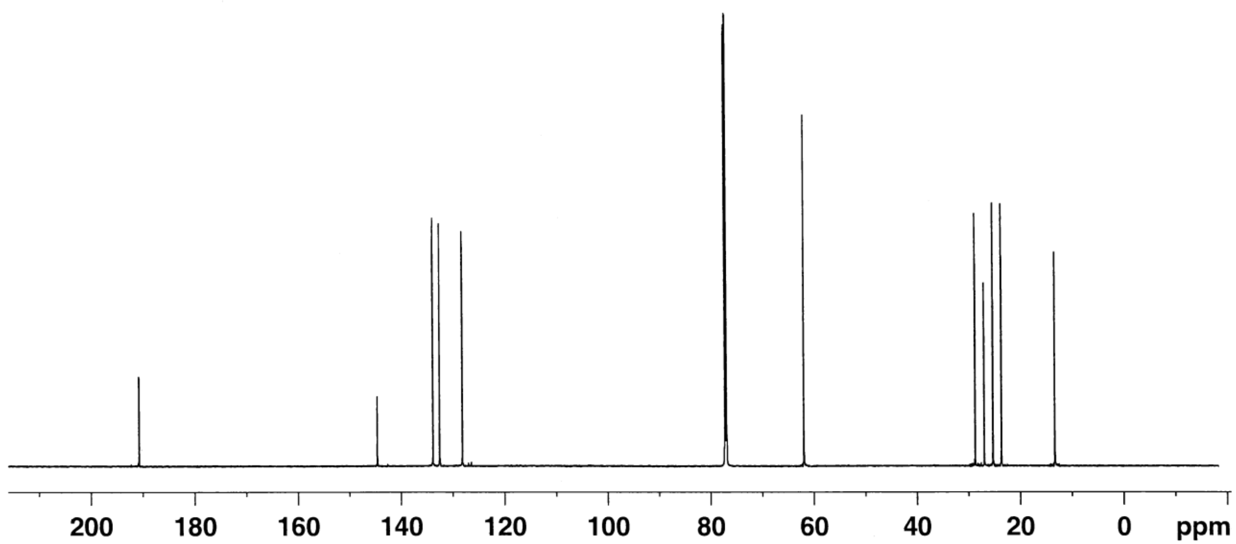
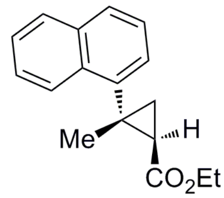
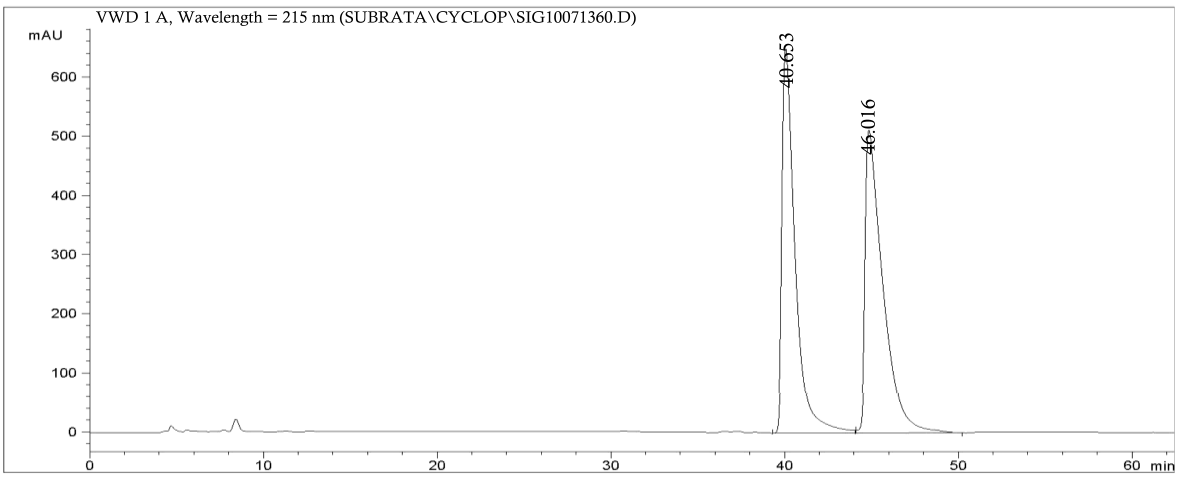


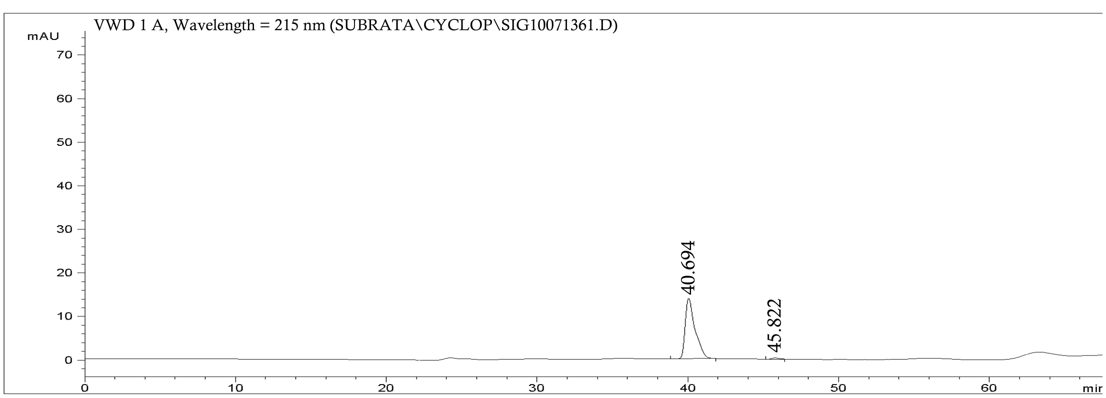
NAME	SS-05-135	
EXPNO	2	
PROCNO	1	
Date_	20140106	
Time	19.12	
INSTRUM	spect	
PROBHD	5 mm CPDCH 13C	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	43	
DS	4	
SWH	41666.668	Hz
FIDRES	0.635783	Hz
AQ	0.7864820	sec
RG	203	
DW	12.000	usec
DE	16.50	usec
TE	298.1	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	9.00	usec
PL1	4.50	dB
PL1W	38.14553833	W
SFO1	176.0697436	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	65.00	usec
PL2	-3.20	dB
PL12	13.60	dB
PL13	120.00	dB
PL2W	33.59817505	W
PL12W	0.70196527	W
PL13W	0.00000000	W
SFO2	700.1499406	MHz
SI	32768	
SF	176.0521380	MHz
WDW	EM	
SSB	0	
LB	3.00	Hz
GB	0	
PC	1.40	



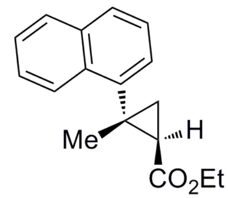
22



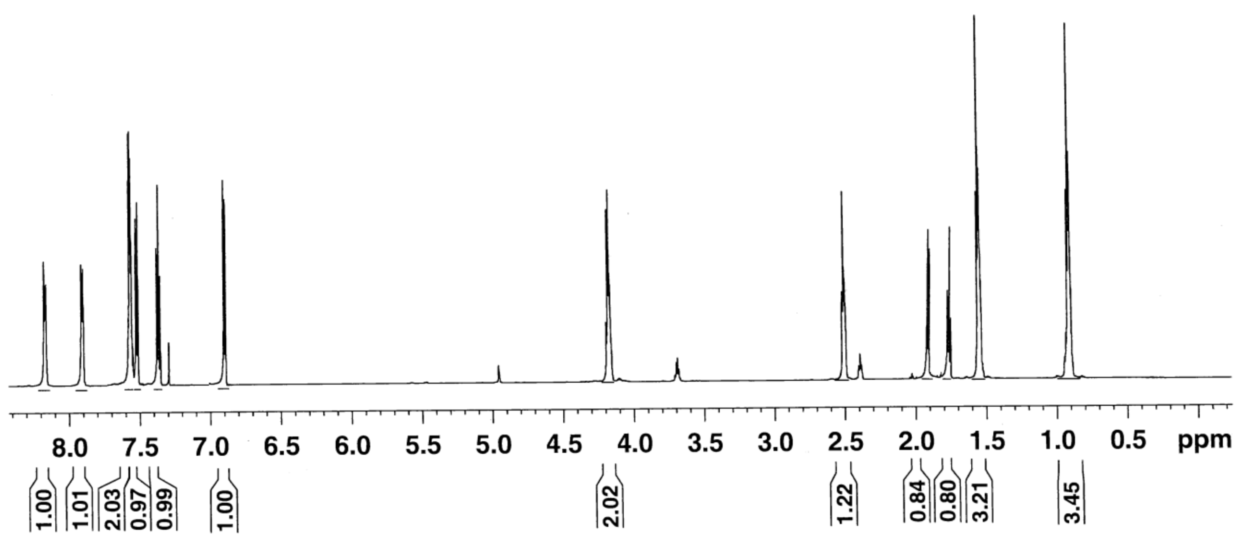
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	40.653	BB	0.7408	3.34754e4	648.95361	44.0168
2	46.016	VB	1.2012	3.65632e4	514.84083	55.9832



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	40.694	BV	0.4296	7964.43606	13.15377	97.8066
2	45.822	VB	0.0846	178.60956	0.10983	2.1934

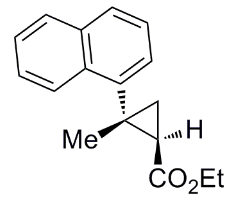


22

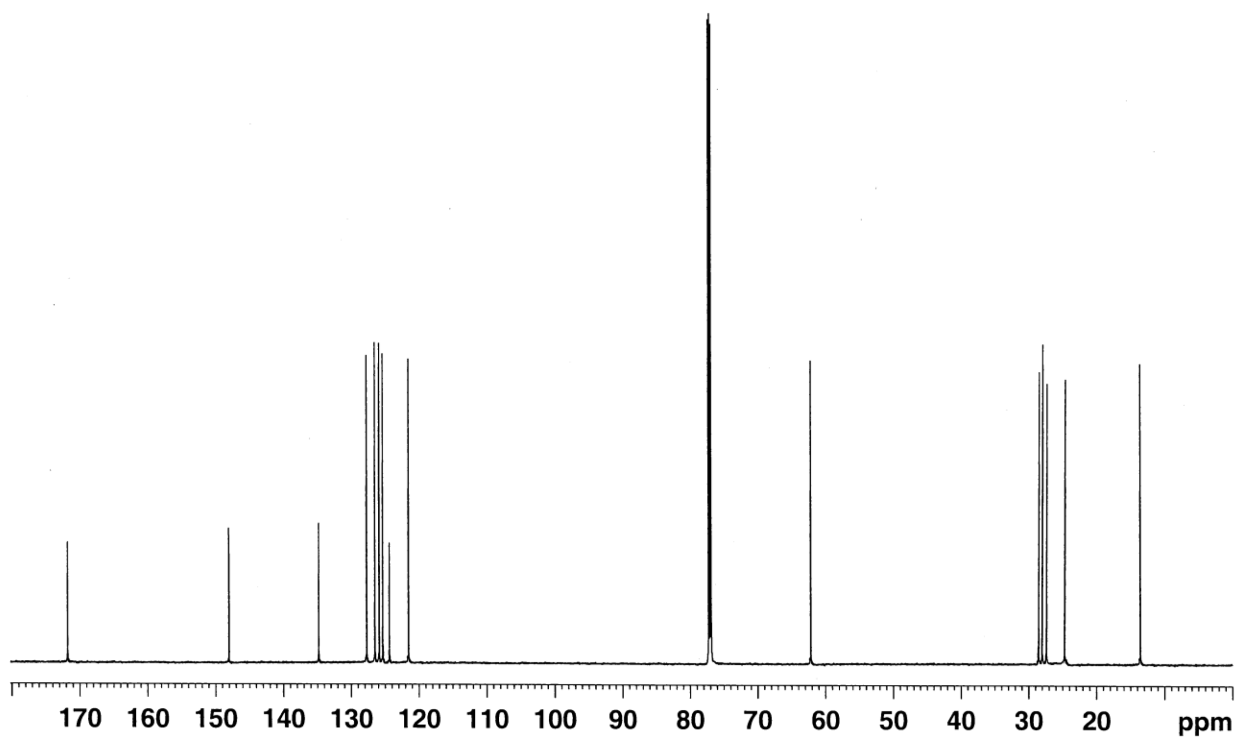



```
NAME            SS-05-136
EXPNO                   1
PROCNO                  1
Date_            20140106
Time                19.24
INSTRUM             spect
PROBHD   5 mm CPDCH 13C
PULPROG              zg30
TD                  95236
SOLVENT             CDCl3
NS                     10
DS                      2
SWH             11904.762 Hz
FIDRES           0.125003 Hz
AQ              3.9999621 sec
RG                   25.4
DW                 42.000 use
DE                   6.50 use
TE                  298.2 K
D1             2.00000000 sec
TD0                     1

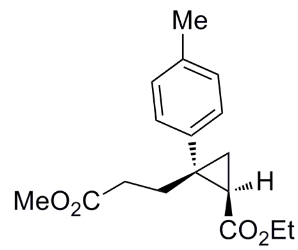
======== CHANNEL f1 ========
NUC1                   1H
P1                   9.40 use
PL1                 -3.20 dB
PL1W          33.59817505 W
SFO1          700.1516910 MHz
SI                 131072
SF            700.1471400 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



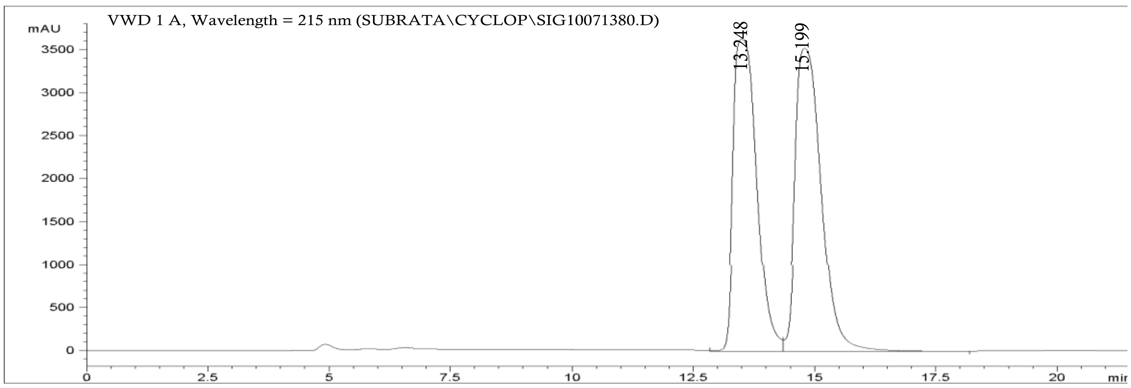
22



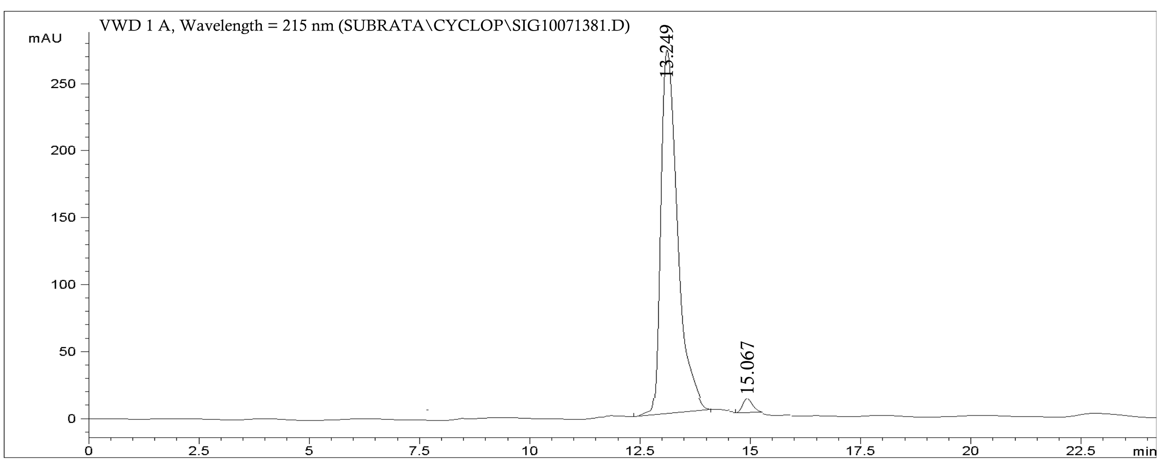
NAME	SS-05-136	
EXPNO	2	
PROCNO	1	
Date_	20140106	
Time	19.29	
INSTRUM	spect	
PROBHD	5 mm CPDCH 13C	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	60	
DS	4	
SWH	41666.668	Hz
FIDRES	0.635783	Hz
AQ	0.7864820	sec
RG	203	
DW	12.000	use
DE	16.50	use
TE	298.2	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1 =======		
NUC1	13C	
P1	9.00	use
PL1	4.50	dB
PL1W	38.14553833	W
SFO1	176.0697436	MHz
======== CHANNEL f2 =======		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	65.00	use
PL2	-3.20	dB
PL12	13.60	dB
PL13	120.00	dB
PL2W	33.59817505	W
PL12W	0.70196527	W
PL13W	0.00000000	W
SFO2	700.1499406	MHz
SI	32768	
SF	176.0521380	MHz
WDW	EM	
SSB	0	
LB	3.00	Hz
GB	0	
PC	1.40	



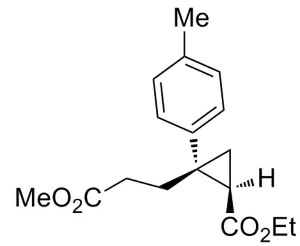
23



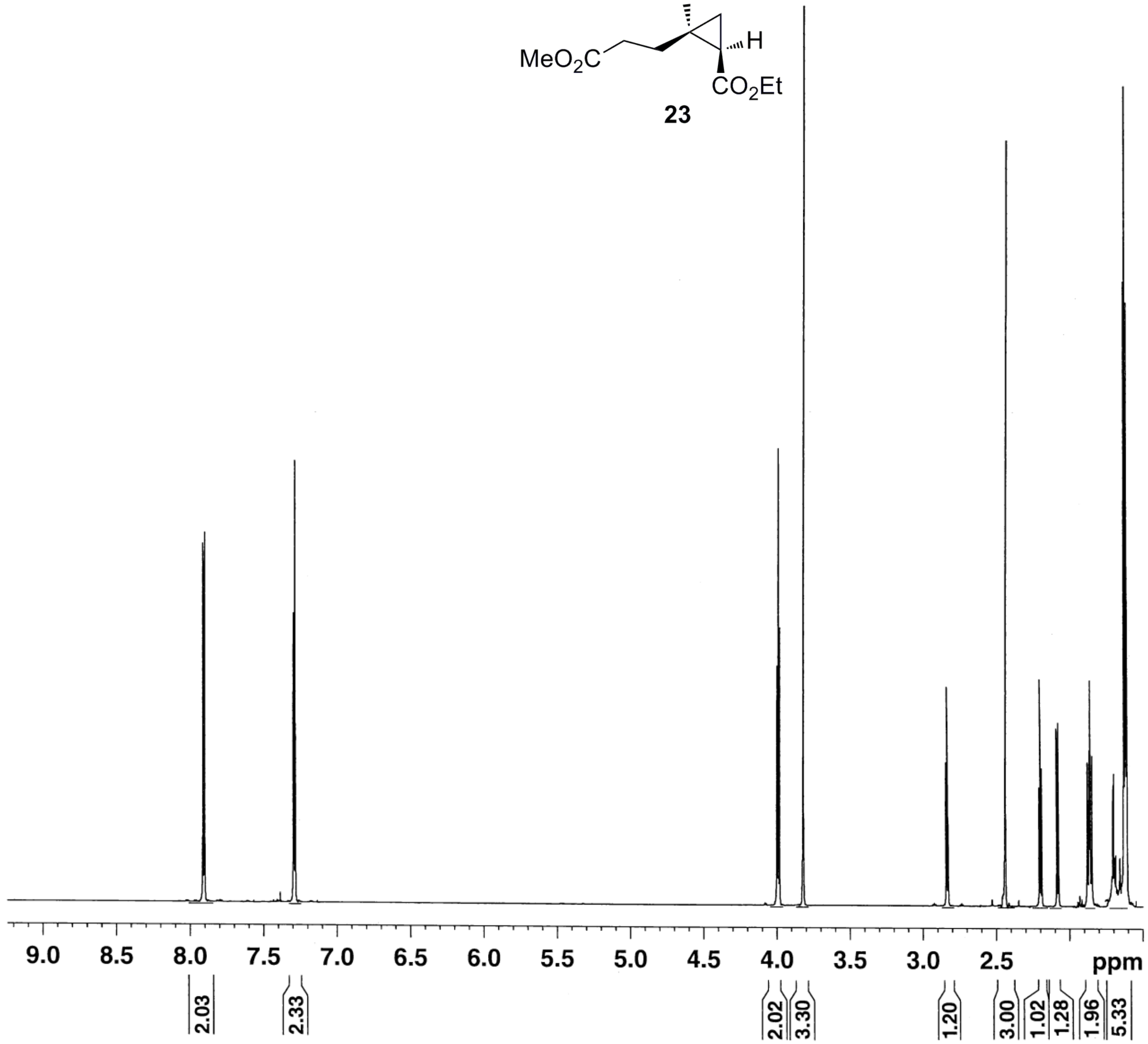
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.248	BB	0.4536	1.22381e5	3633.09243	48.1463
2	15.199	VB	0.5702	1.31805e5	3502.98901	51.8537



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.249	VB	0.4183	13497.85965	271.07908	97.1794
2	15.067	BB	0.0810	391.77092	8.92509	2.8206

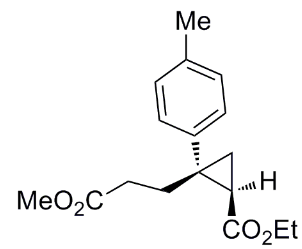


23

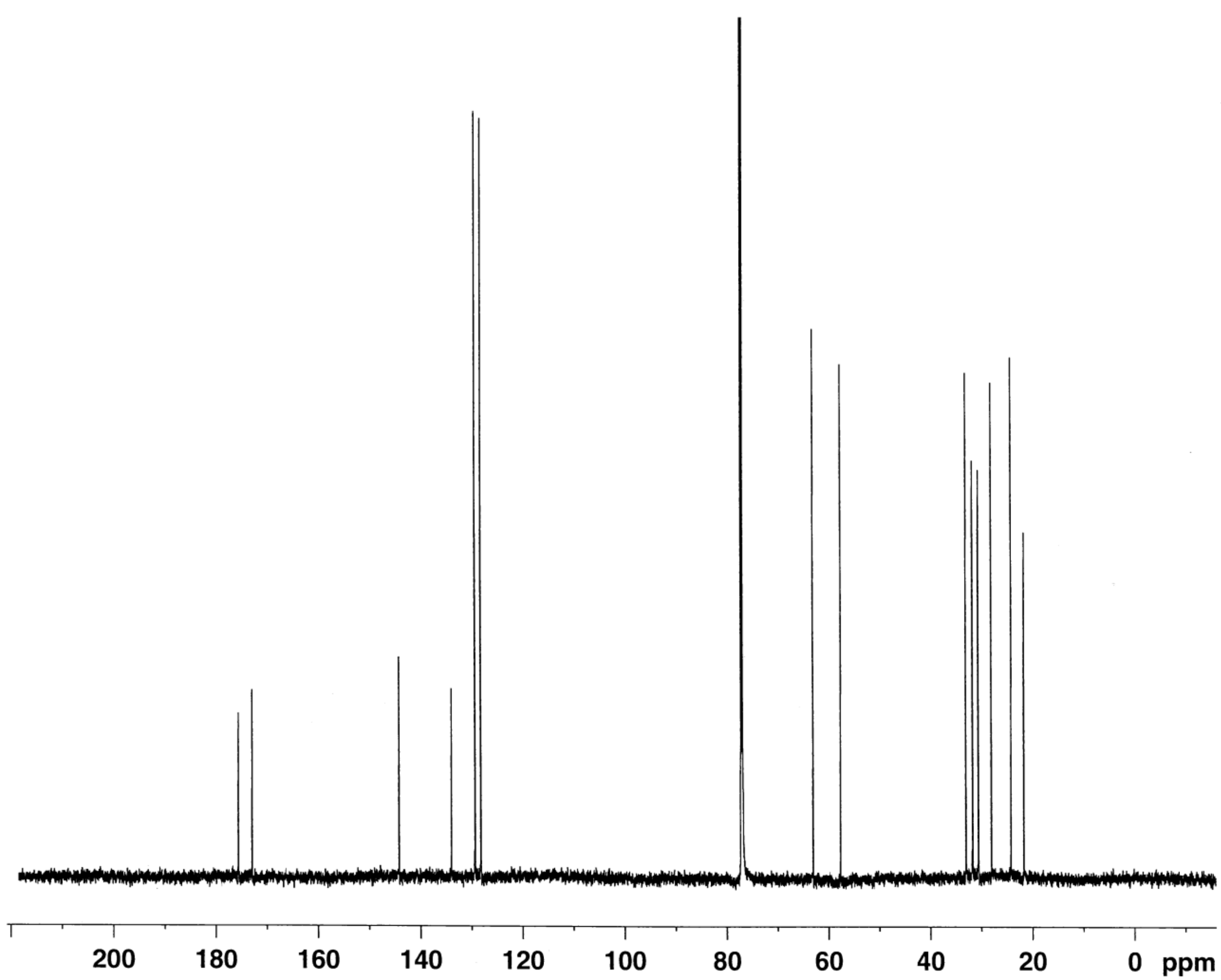



```
NAME            SS-05-138
EXPNO                   3
PROCNO                  1
Date_            20140106
Time                20.49
INSTRUM             spect
PROBHD   5 mm CPDCH 13C
PULPROG              zg30
TD                  95236
SOLVENT             CDCl3
NS                     10
DS                      2
SWH             11904.762 Hz
FIDRES           0.125003 Hz
AQ              3.9999621 sec
RG                     32
DW                 42.000 usec
DE                   6.50 usec
TE                  298.1 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.40 usec
PL1                 -3.20 dB
PL1W          33.59817505 W
SFO1          700.1516910 MHz
SI                 131072
SF            700.1471400 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



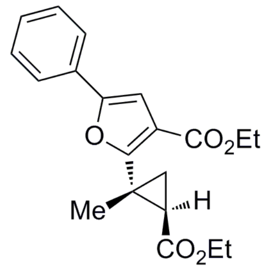
23



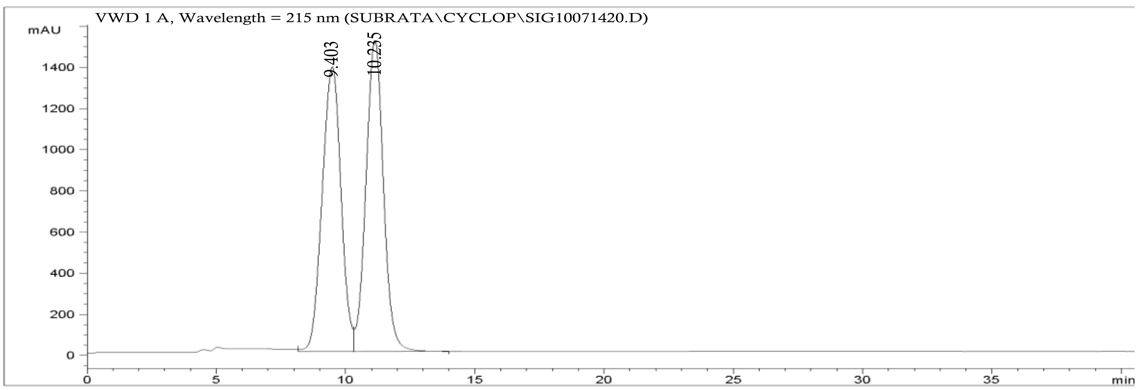

```
NAME           SS-05-138
EXPNO                  4
PROCNO                 1
Date_           20140106
Time               20.53
INSTRUM            spect
PROBHD   5 mm CPDCH 13C
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    41
DS                     4
SWH            41666.668 Hz
FIDRES          0.635783 Hz
AQ             0.7864820 sec
RG                   101
DW                12.000 usec
DE                 16.50 usec
TE                 298.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.00 usec
PL1                 4.50 dB
PL1W         38.14553833 W
SFO1         176.0697436 MHz

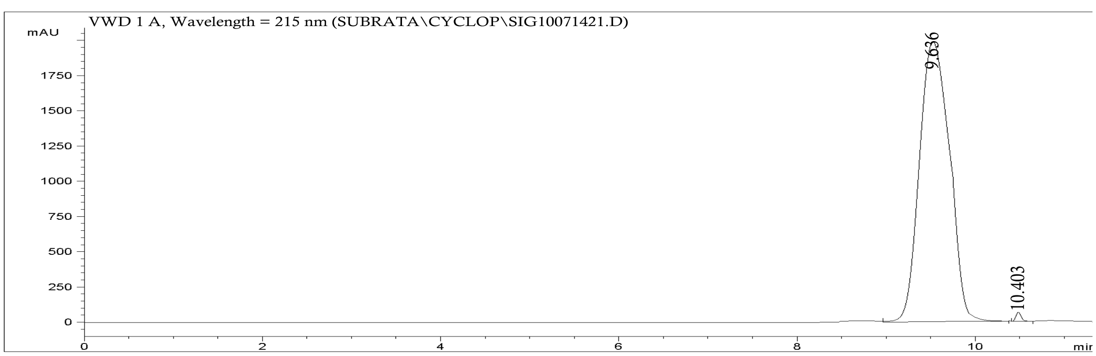
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              65.00 usec
PL2                -3.20 dB
PL12               13.60 dB
PL13              120.00 dB
PL2W         33.59817505 W
PL12W         0.70196527 W
PL13W         0.00000000 W
SFO2         700.1499406 MHz
SI                 32768
SF           176.0521380 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.40
```



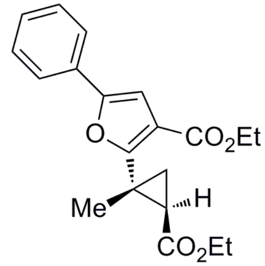
24



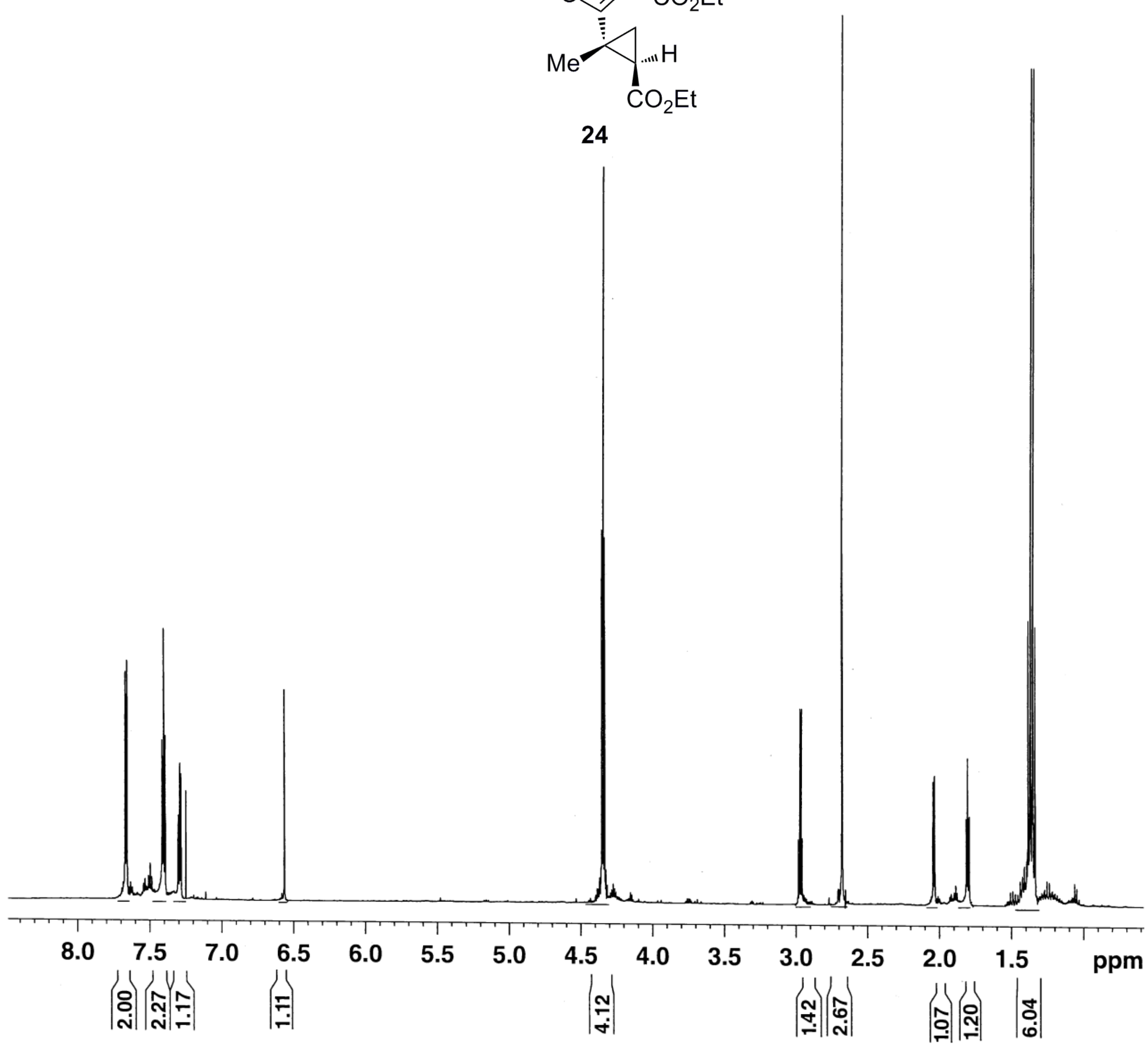
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.403	VB	0.5204	2.35595e4	1442.39208	49.2681
2	10.235	BB	0.4941	2.42397e4	1583.49035	50.7319



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.636	VB	0.4682	1972.63315	1982.06928	98.5182
2	10.403	VB	0.0787	29.67013	48.78802	1.4818

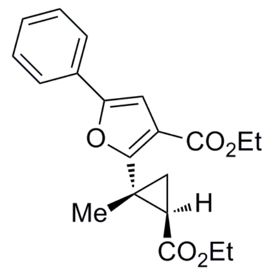


24

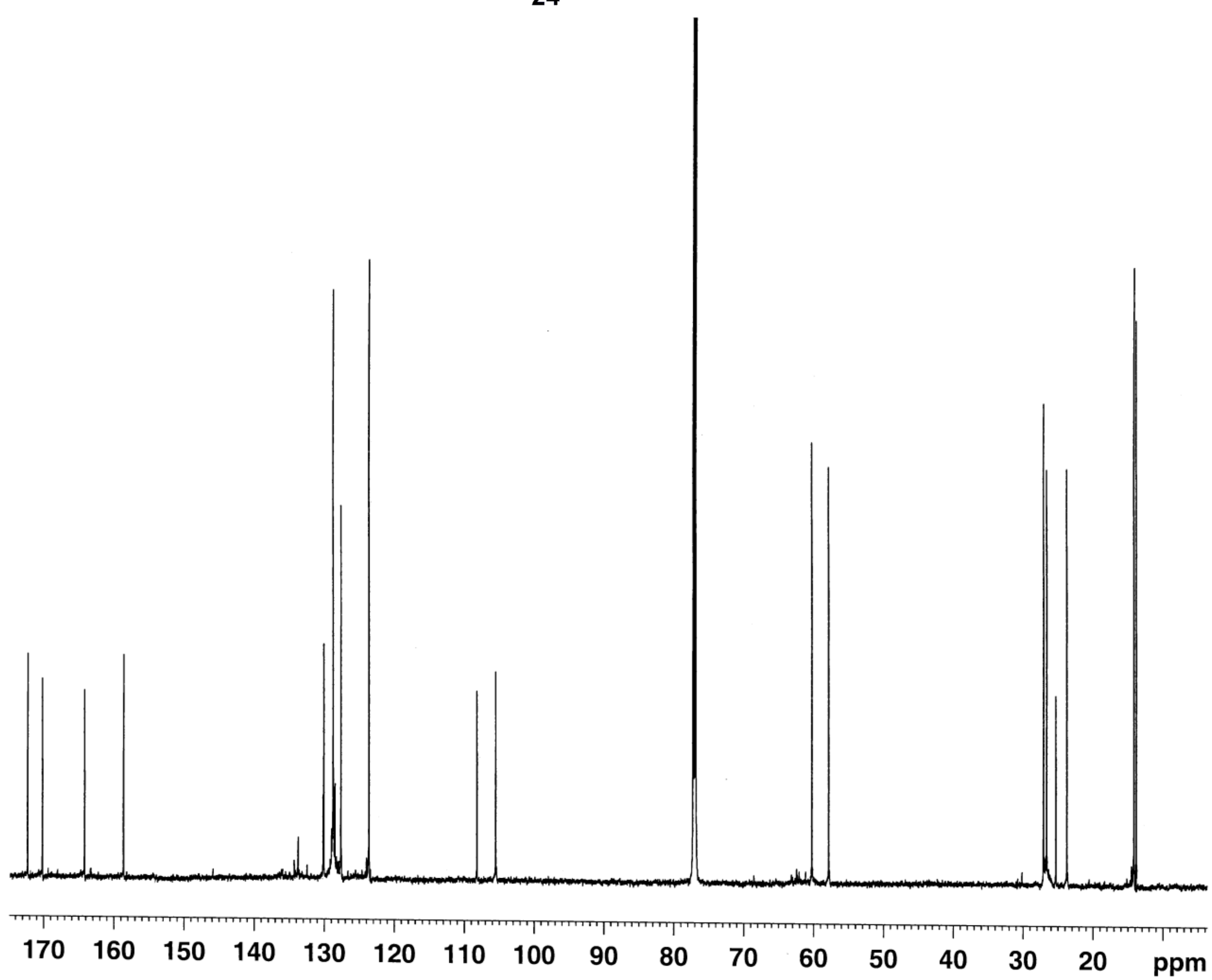



```
NAME              SS-05-142
EXPNO                     1
PROCNO                    1
Date_              20140106
Time                  19.42
INSTRUM               spect
PROBHD    5 mm CPDCH 13C
PULPROG                zg30
TD                    95236
SOLVENT               CDCl3
NS                       11
DS                        2
SWH              11904.762 Hz
FIDRES            0.125003 Hz
AQ               3.9999621 sec
RG                     25.4
DW                   42.000 usec
DE                     6.50 usec
TE                    298.1 K
D1               2.00000000 sec
TD0                       1

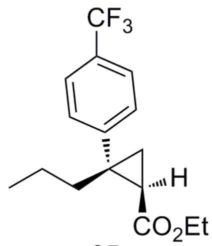
======== CHANNEL f1 ========
NUC1                     1H
P1                     9.40 usec
PL1                   -3.20 dB
PL1W            33.59817505 W
SFO1            700.1516910 MHz
SI                   131072
SF              700.1471400 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```



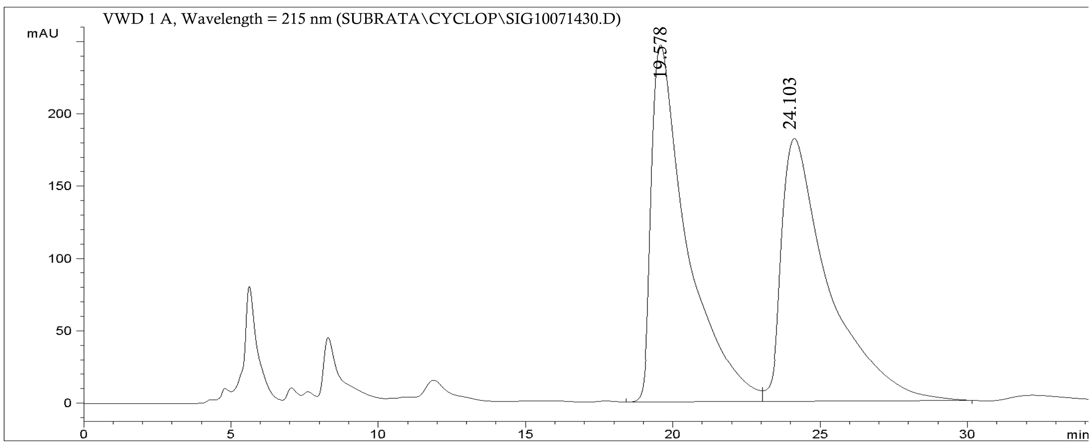
24



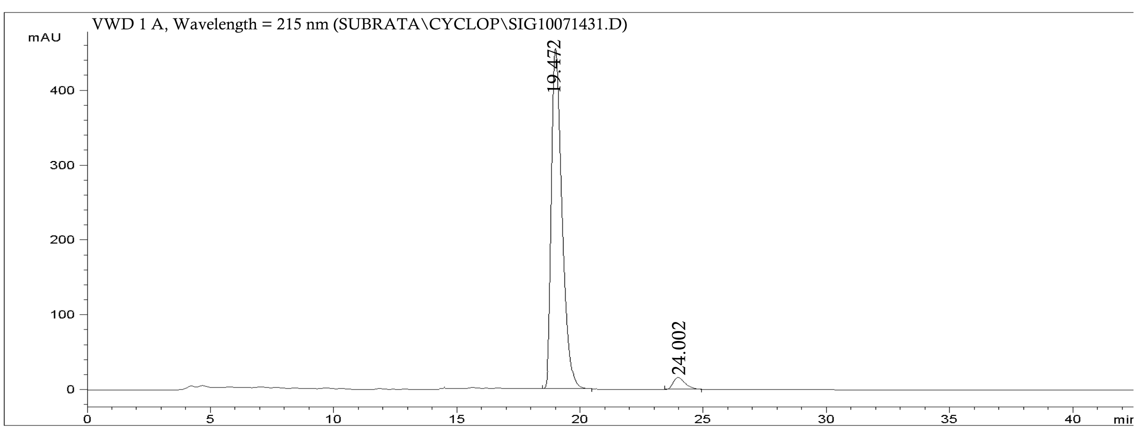
NAME	SS-05-142
EXPNO	2
PROCNO	1
Date_	20140106
Time	19.46
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	57
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 sec
RG	203
DW	12.000 usec
DE	16.50 usec
TE	298.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.00 usec
PL1	4.50 dB
PL1W	38.14553833 W
SFO1	176.0697436 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 usec
PL2	-3.20 dB
PL12	13.60 dB
PL13	120.00 dB
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1499406 MHz
SI	32768
SF	176.0521380 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40



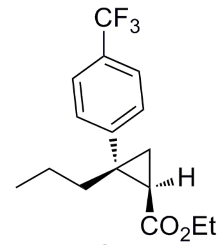
25



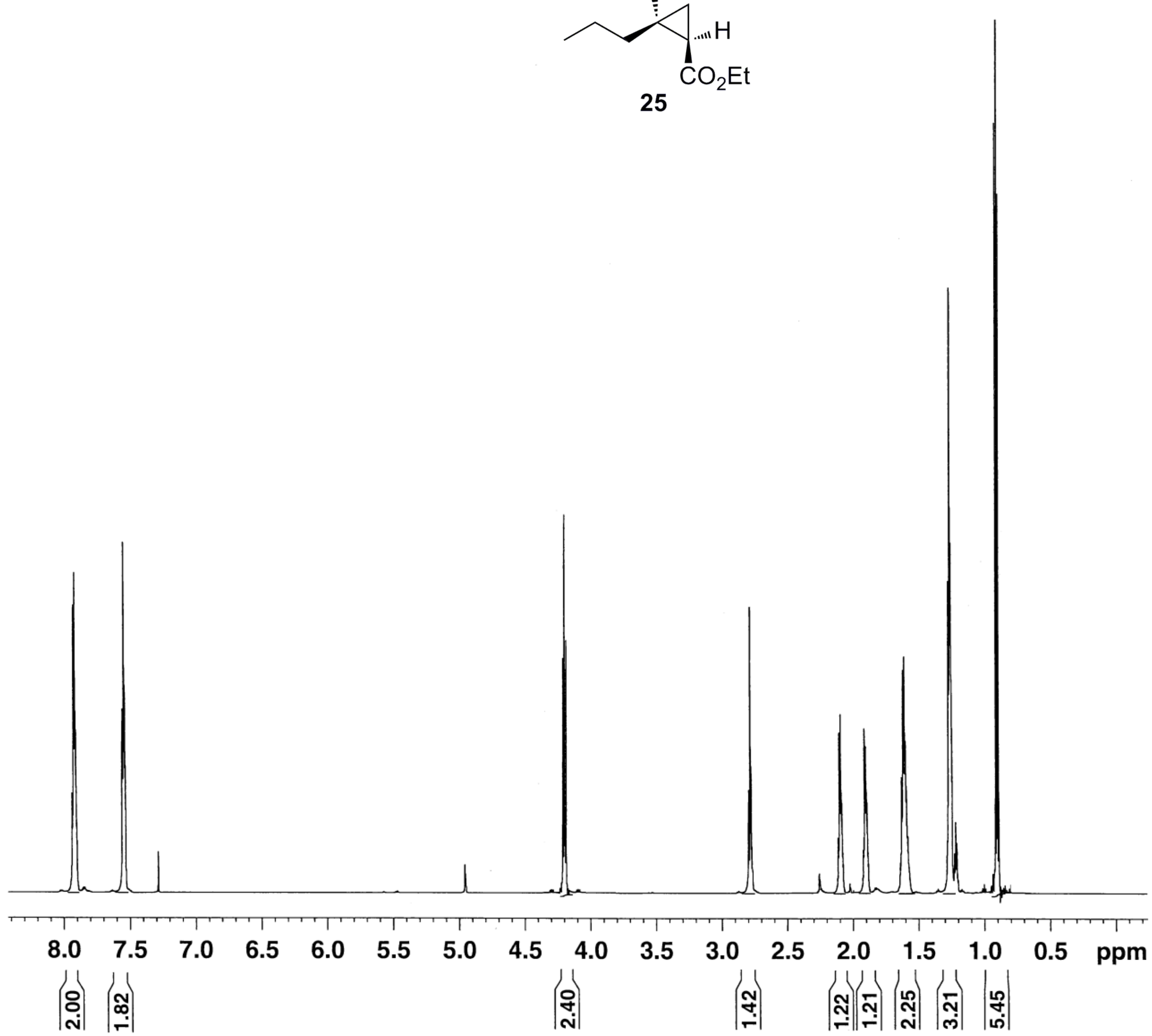
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	19.578	VB	1.2289	2.10043e4	246.88022	57.5866
2	24.103	BB	1.5899	2.04256e4	181.98033	42.4134



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	19.472	BB	0.4586	6974.3335	453.98066	95.8967
2	24.002	VB	0.5507	298.42302	5.75003	4.1033

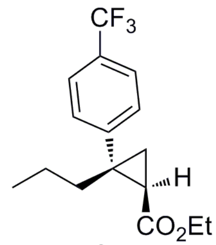


25

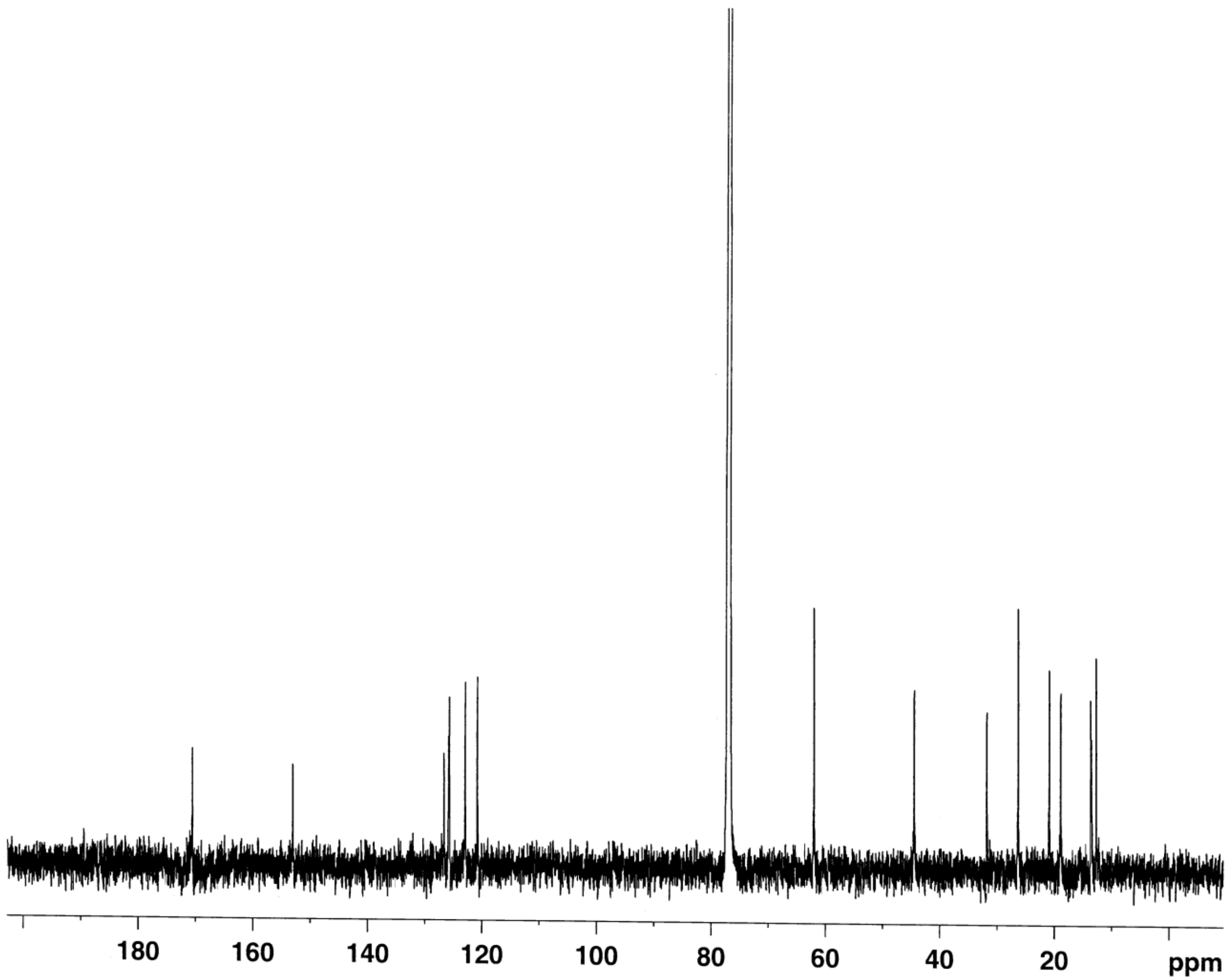



```
NAME            SS-05-143
EXPNO                   1
PROCNO                  1
Date_            20140106
Time                18.37
INSTRUM             spect
PROBHD   5 mm CPDCH 13C
PULPROG              zg30
TD                  95236
SOLVENT             CDCl3
NS                     12
DS                      2
SWH             11904.762 Hz
FIDRES           0.125003 Hz
AQ              3.9999621 sec
RG                   25.4
DW                 42.000 usec
DE                   6.50 usec
TE                  298.2 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.40 usec
PL1                 -3.20 dB
PL1W          33.59817505 W
SFO1          700.1516910 MHz
SI                 131072
SF            700.1471400 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



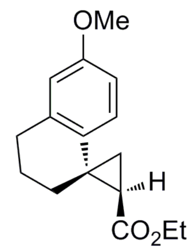
25



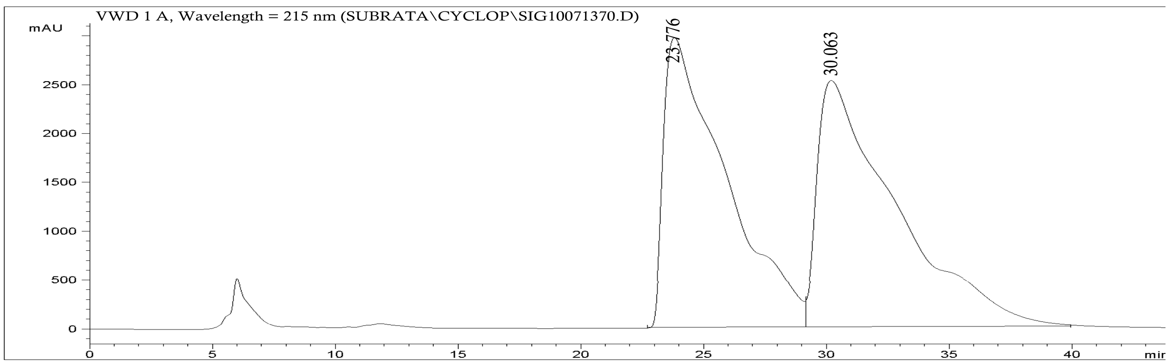

```
NAME               SS-05-143
EXPNO                      2
PROCNO                     1
Date_               20140106
Time                   18.40
INSTRUM                spect
PROBHD     5 mm CPDCH 13C
PULPROG              zgpg30
TD                     65536
SOLVENT                CDCl3
NS                        67
DS                         4
SWH               41666.668 Hz
FIDRES             0.635783 Hz
AQ                0.7864820 sec
RG                       203
DW                    12.000 usec
DE                     16.50 usec
TE                     298.2 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                      9.00 usec
PL1                     4.50 dB
PL1W             38.14553833 W
SFO1             176.0697436 MHz

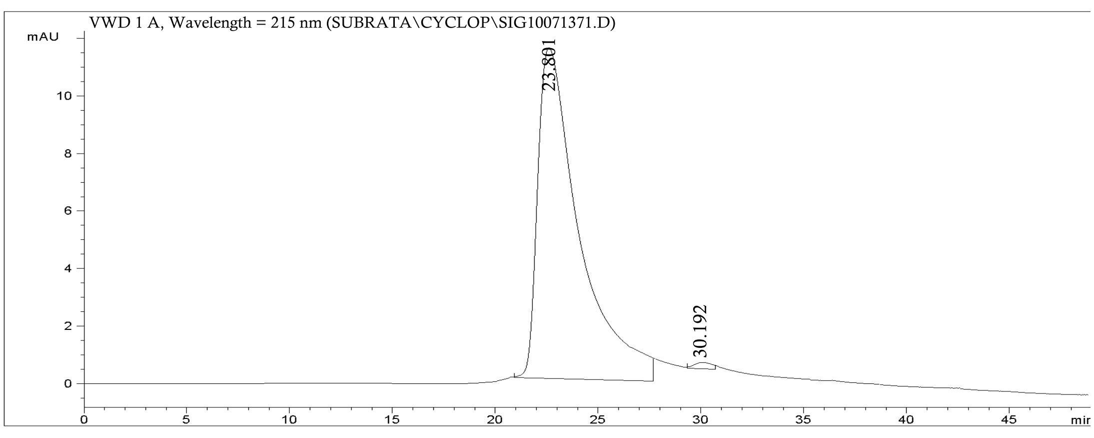
======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                  65.00 usec
PL2                    -3.20 dB
PL12                   13.60 dB
PL13                  120.00 dB
PL2W             33.59817505 W
PL12W             0.70196527 W
PL13W             0.00000000 W
SFO2             700.1499406 MHz
SI                     32768
SF               176.0521380 MHz
WDW                       EM
SSB                        0
LB                      3.00 Hz
GB                         0
PC                      1.40
```



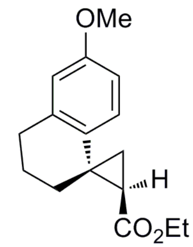
26



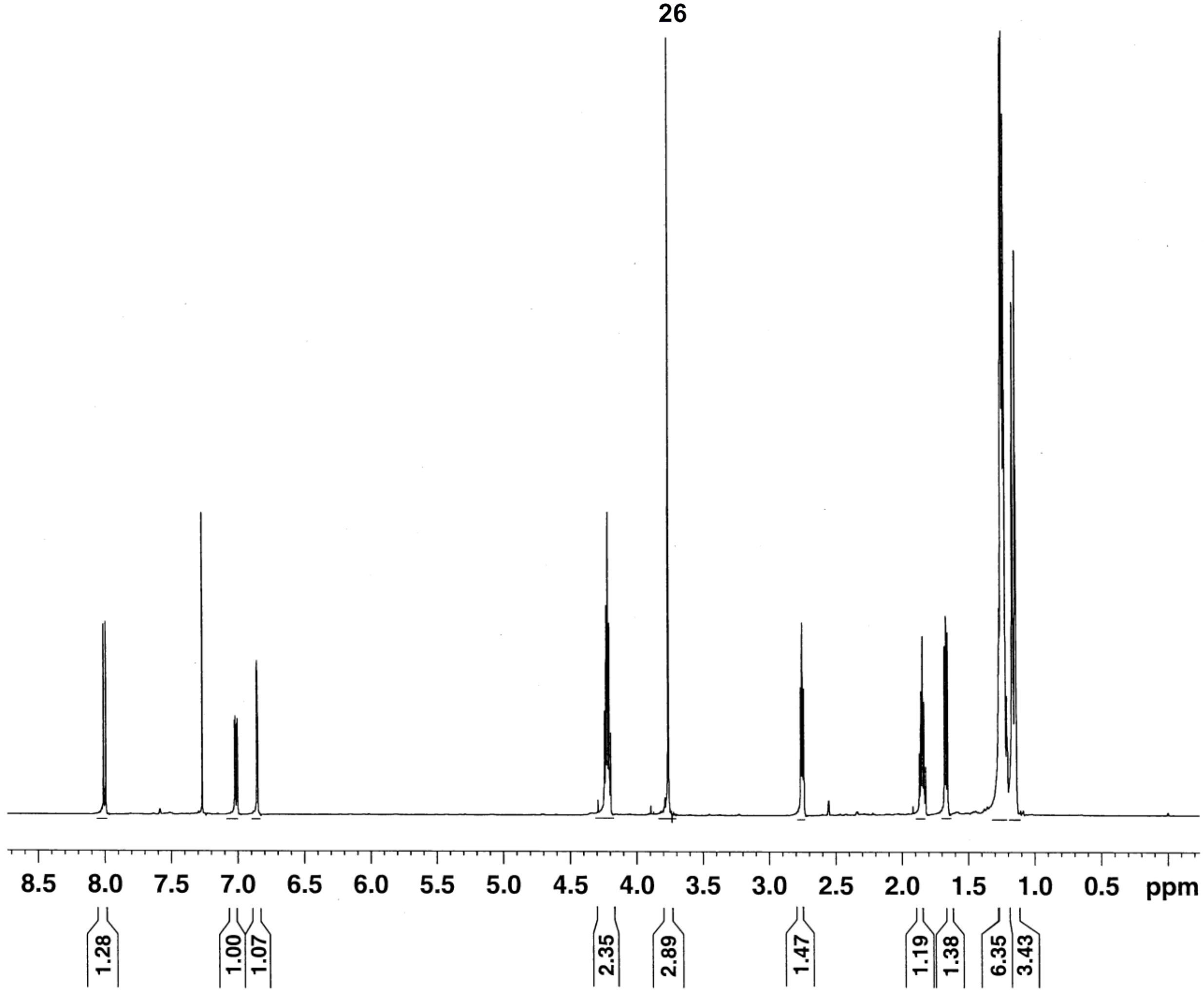
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	23.776	MM	2.9312	5.22162e5	2967.07038	48.3427
2	30.063	MM	3.6729	5.57949e5	2520.92117	51.6573



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	23.801	MM	2.2207	1.26471e4	11.44930	99.2366
2	30.192	MM	3.6601	97.29106	0.22981	0.7634

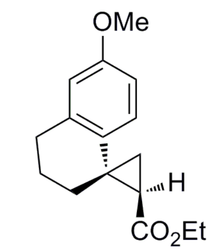


26

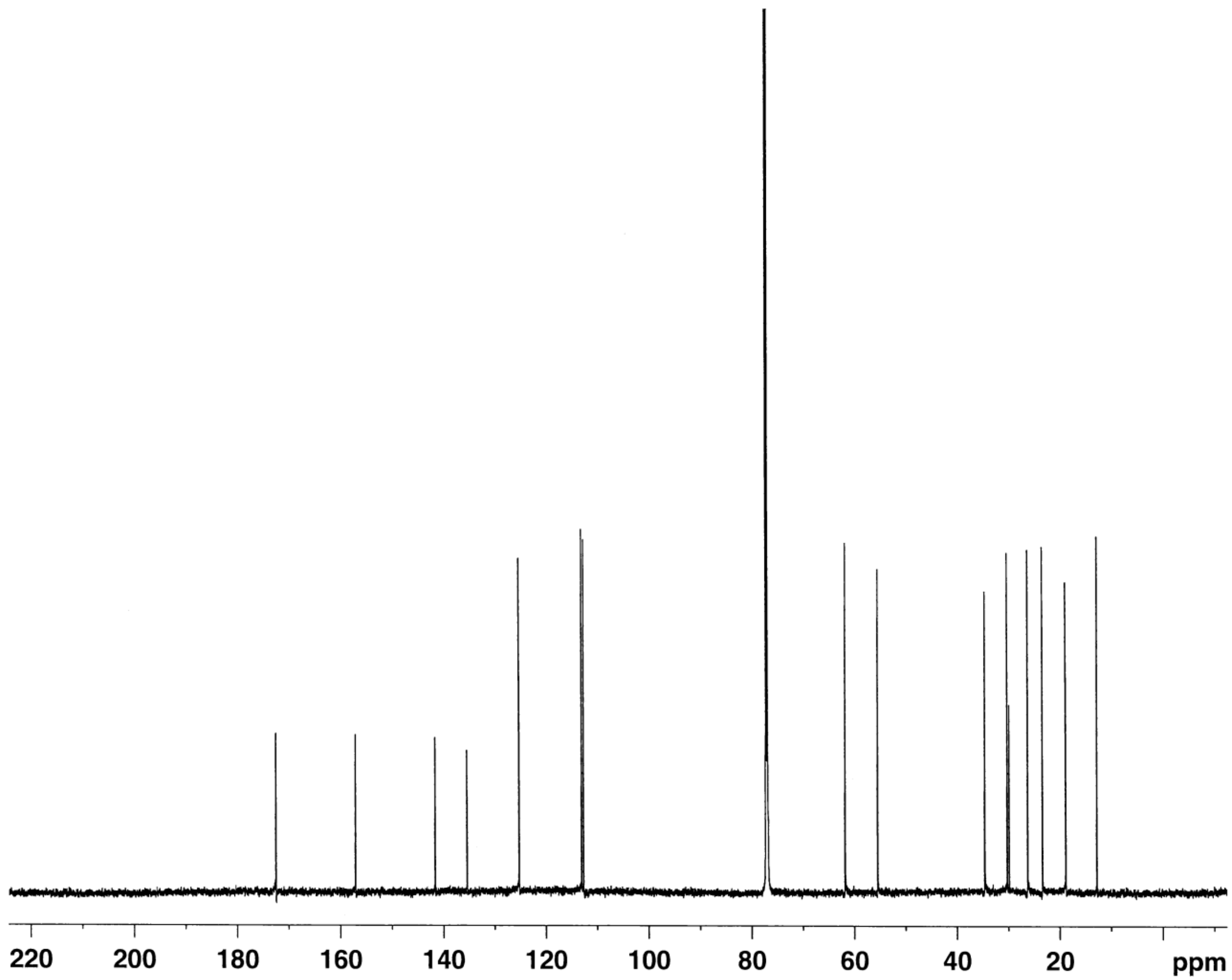



```
NAME            SS-05-137
EXPNO                   1
PROCNO                  1
Date_            20140106
Time                19.32
INSTRUM             spect
PROBHD   5 mm CPDCH 13C
PULPROG              zg30
TD                  95236
SOLVENT             CDCl3
NS                      8
DS                      2
SWH             11904.762 Hz
FIDRES           0.125003 Hz
AQ              3.9999621 sec
RG                     36
DW                 42.000 usec
DE                   6.50 usec
TE                  298.2 K
D1             2.00000000 sec
TD0                     1

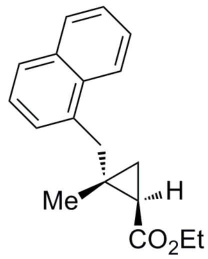
======== CHANNEL f1 ========
NUC1                   1H
P1                   9.40 usec
PL1                 -3.20 dB
PL1W          33.59817505 W
SFO1          700.1516910 MHz
SI                 131072
SF            700.1471400 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



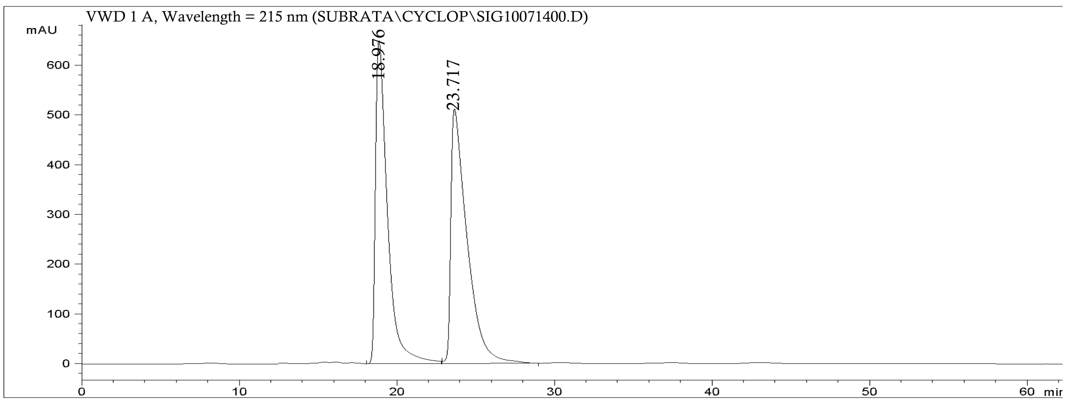
26



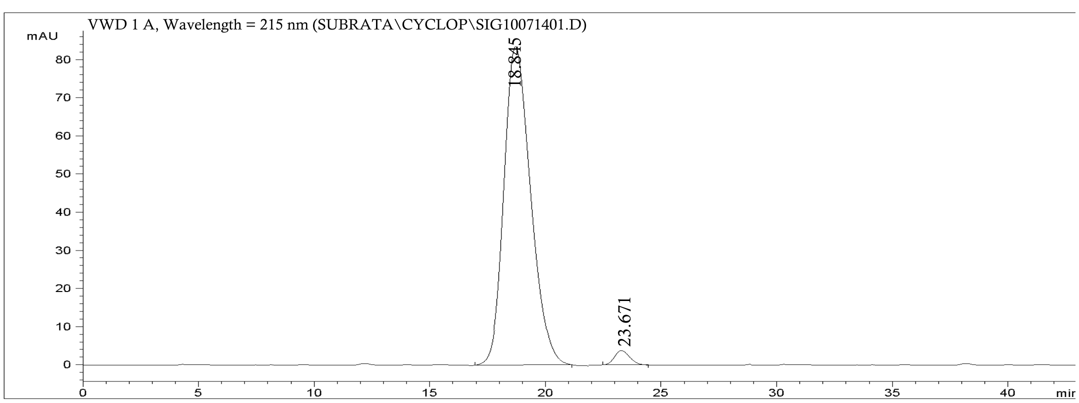
NAME	SS-05-137
EXPNO	2
PROCNO	1
Date_	20140106
Time	19.38
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	93
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 sec
RG	203
DW	12.000 usec
DE	16.50 usec
TE	298.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.00 usec
PL1	4.50 dB
PL1W	38.14553833 W
SFO1	176.0697436 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 usec
PL2	-3.20 dB
PL12	13.60 dB
PL13	120.00 dB
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1499406 MHz
SI	32768
SF	176.0521380 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40



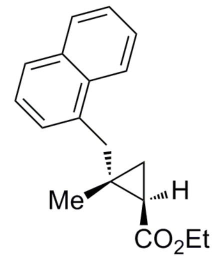
27



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	18.976	BB	0.7792	3.44675e4	648.81031	48.5624
2	23.717	BB	1.1092	3.65049e4	511.17269	51.4376



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	18.845	BB	1.2196	3098.28171	84.34238	91.6493
2	23.671	BB	0.6367	282.30244	3.94012	8.3507



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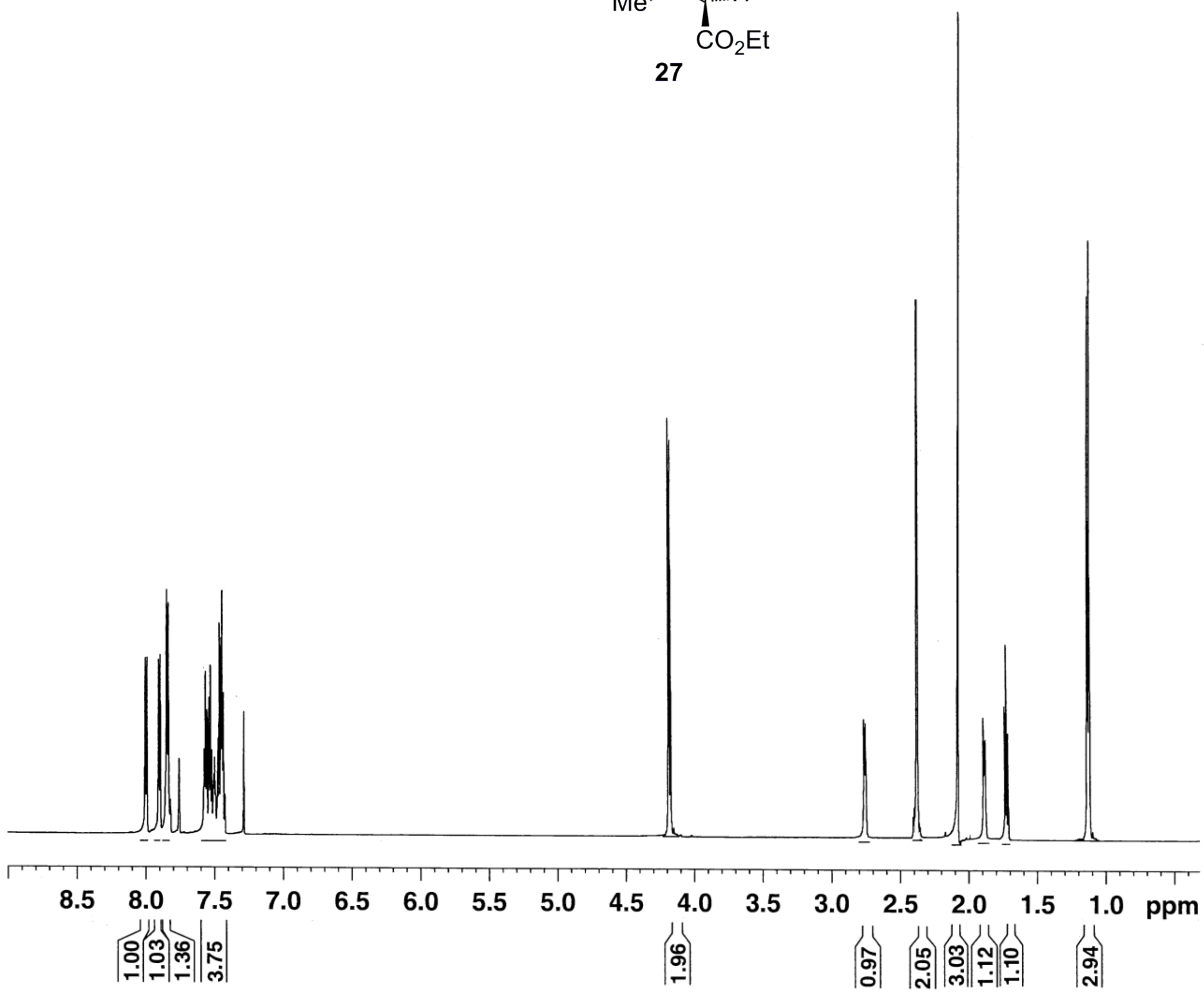
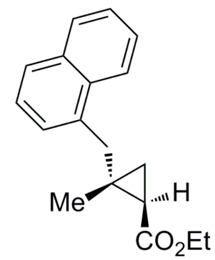
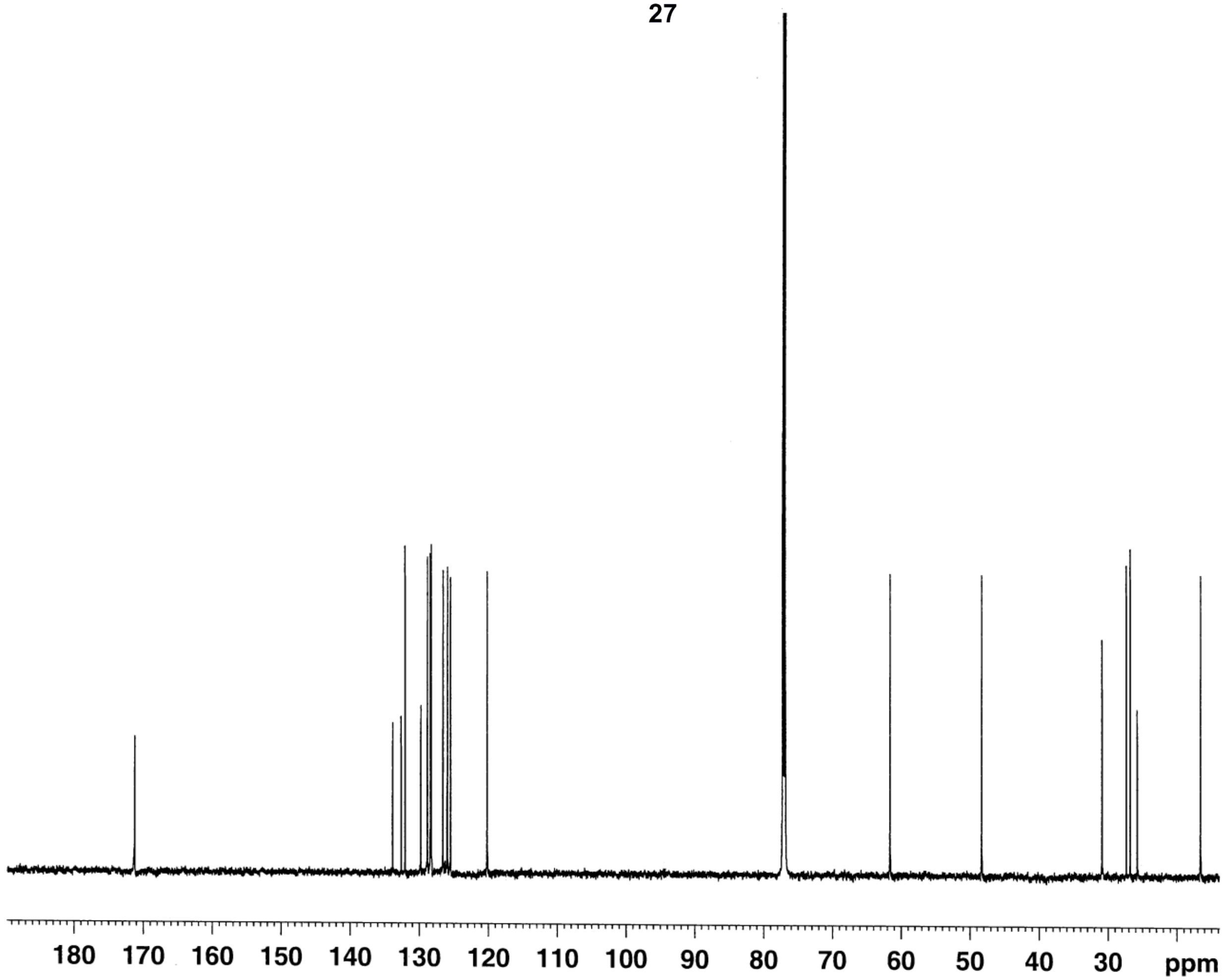



```
NAME          SS-05-140
EXPNO                 1
PROCNO                1
Date_          20140106
Time              18.49
INSTRUM           spect
PROBHD   5 mm CPDCH 13C
PULPROG            zg30
TD                95236
SOLVENT           CDCl3
NS                   13
DS                    2
SWH           11904.762 Hz
FIDRES         0.125003 Hz
AQ            3.9999621 sec
RG                 22.6
DW               42.000 usec
DE                 6.50 usec
TE                298.2 K
D1           2.00000000 sec
TD0                   1

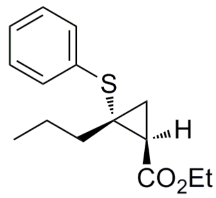
======== CHANNEL f1 ========
NUC1                 1H
P1                 9.40 usec
PL1               -3.20 dB
PL1W        33.59817505 W
SFO1        700.1516910 MHz
SI               131072
SF          700.1471400 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



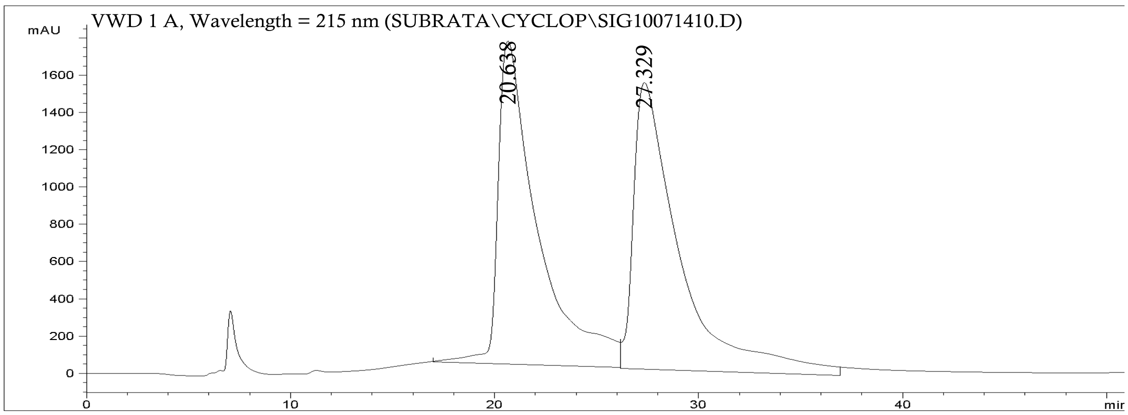
27



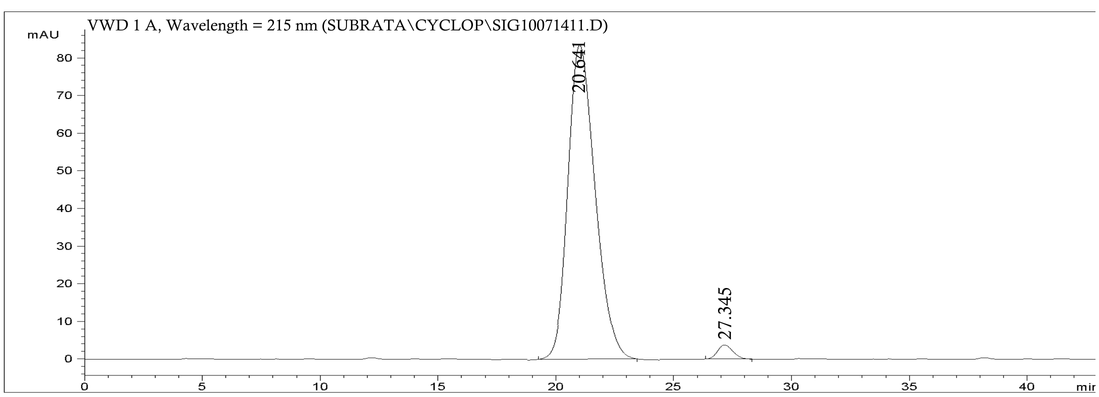
NAME	SS-05-140	
EXPNO	2	
PROCNO	1	
Date_	20140106	
Time	18.52	
INSTRUM	spect	
PROBHD	5 mm CPDCH 13C	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	38	
DS	4	
SWH	41666.668	Hz
FIDRES	0.635783	Hz
AQ	0.7864820	sec
RG	203	
DW	12.000	usec
DE	16.50	usec
TE	298.1	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	9.00	usec
PL1	4.50	dB
PL1W	38.14553833	W
SFO1	176.0697436	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	65.00	usec
PL2	-3.20	dB
PL12	13.60	dB
PL13	120.00	dB
PL2W	33.59817505	W
PL12W	0.70196527	W
PL13W	0.00000000	W
SFO2	700.1499406	MHz
SI	32768	
SF	176.0521380	MHz
WDW	EM	
SSB	0	
LB	3.00	Hz
GB	0	
PC	1.40	



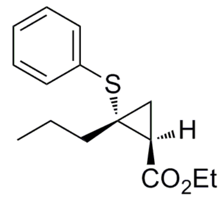
28



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	20.638	MM	2.2827	2.34028e5	1733.98661	49.2661
2	27.329	MM	2.5823	2.40823e5	1577.83504	50.7339



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	20.641	BB	1.2407	6323.95514	83.76202	94.2327
2	27.345	BB	0.6692	387.04342	3.72017	5.7673



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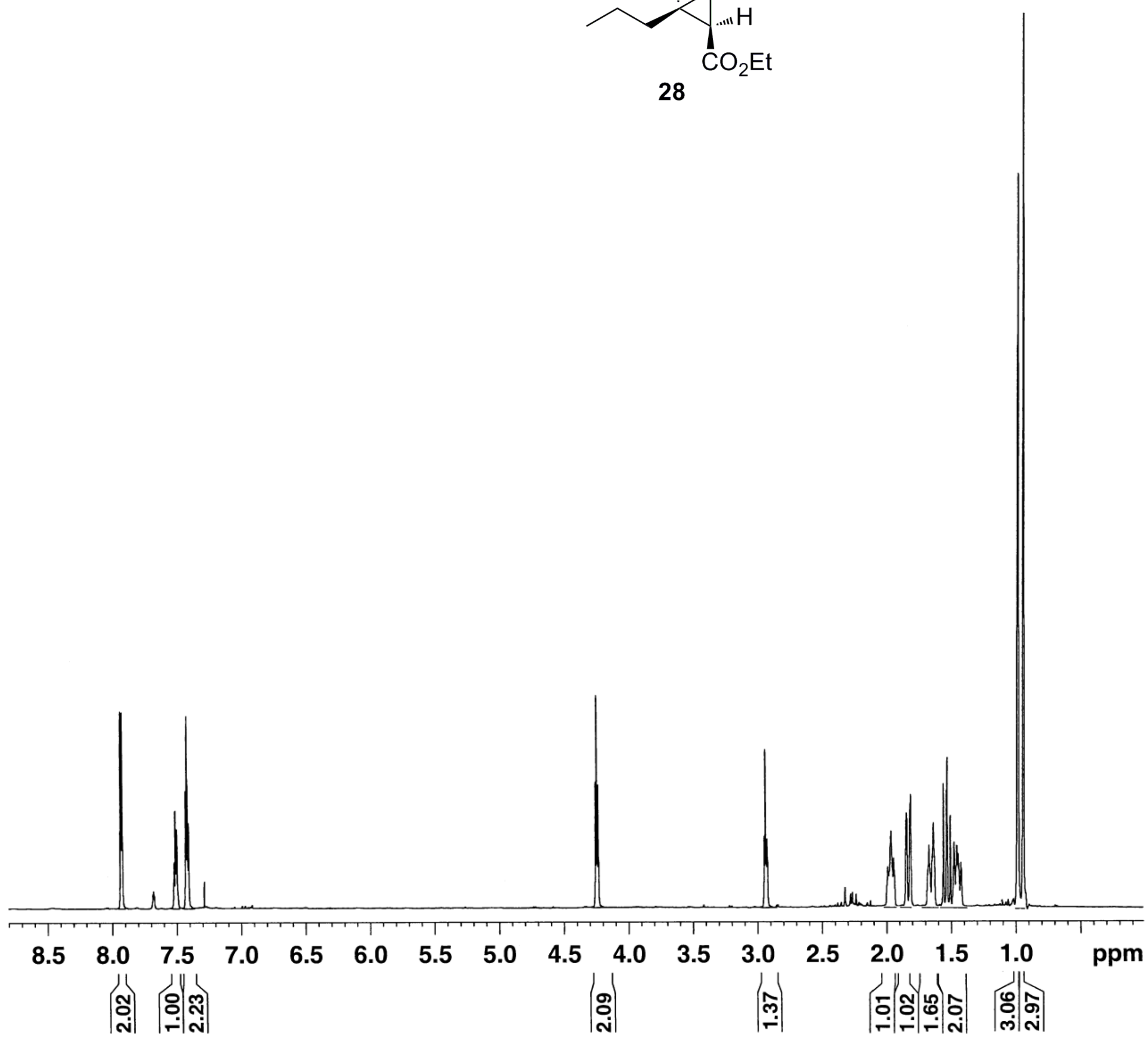
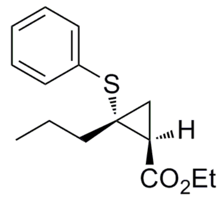


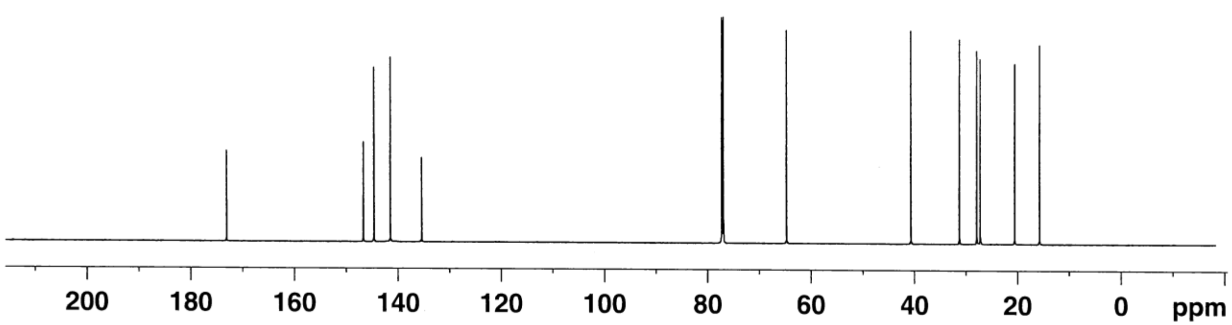
NAME	SS-05-141	
EXPNO	1	
PROCNO	1	
Date_	20140106	
Time	18.57	
INSTRUM	spect	
PROBHD	5 mm CPDCH 13C	
PULPROG	zg30	
TD	95236	
SOLVENT	CDCl3	
NS	8	
DS	2	
SWH	11904.762	Hz
FIDRES	0.125003	Hz
AQ	3.9999621	sec
RG	20.2	
DW	42.000	use
DE	6.50	use
TE	298.1	K
D1	2.00000000	sec
TD0	1	

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

NUC1	1H	
P1	9.40	use
PL1	-3.20	dB
PL1W	33.59817505	W
SFO1	700.1516910	MHz
SI	131072	
SF	700.1471400	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	



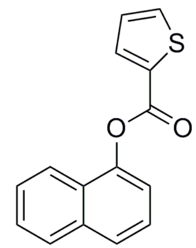
28




```
NAME          SS-05-141
EXPNO                 2
PROCNO                1
Date_          20140106
Time              19.02
INSTRUM           spect
PROBHD   5 mm CPDCH 13C
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                   70
DS                    4
SWH          41666.668 Hz
FIDRES        0.635783 Hz
AQ           0.7864820 sec
RG                  203
DW               12.000 usec
DE                16.50 usec
TE                298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.00 usec
PL1                4.50 dB
PL1W        38.14553833 W
SFO1        176.0697436 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                 1H
PCPD2             65.00 usec
PL2               -3.20 dB
PL12              13.60 dB
PL13             120.00 dB
PL2W        33.59817505 W
PL12W        0.70196527 W
PL13W        0.00000000 W
SFO2        700.1499406 MHz
SI                32768
SF          176.0521380 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.40
```



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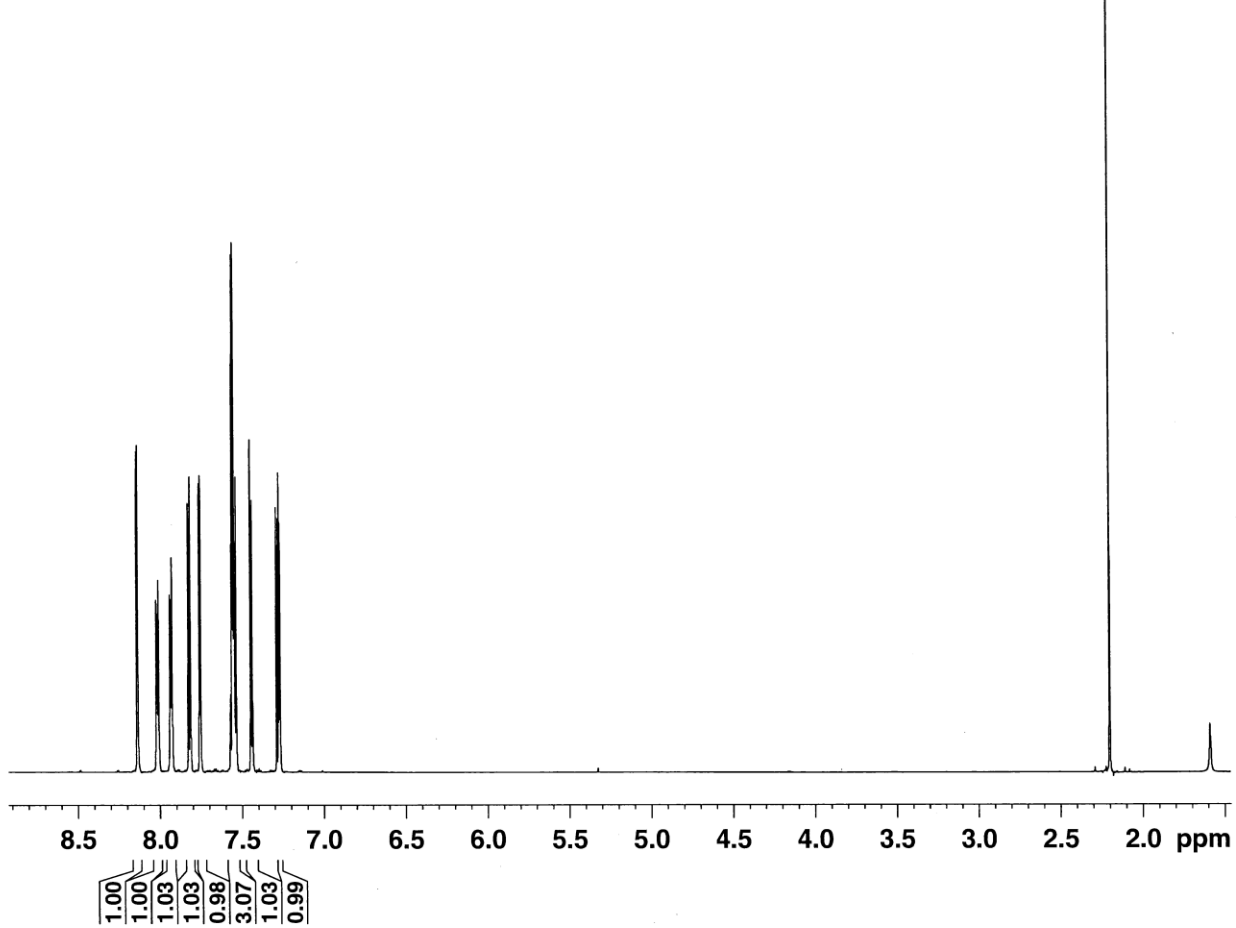
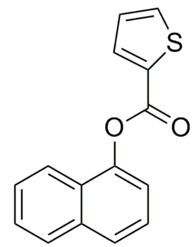
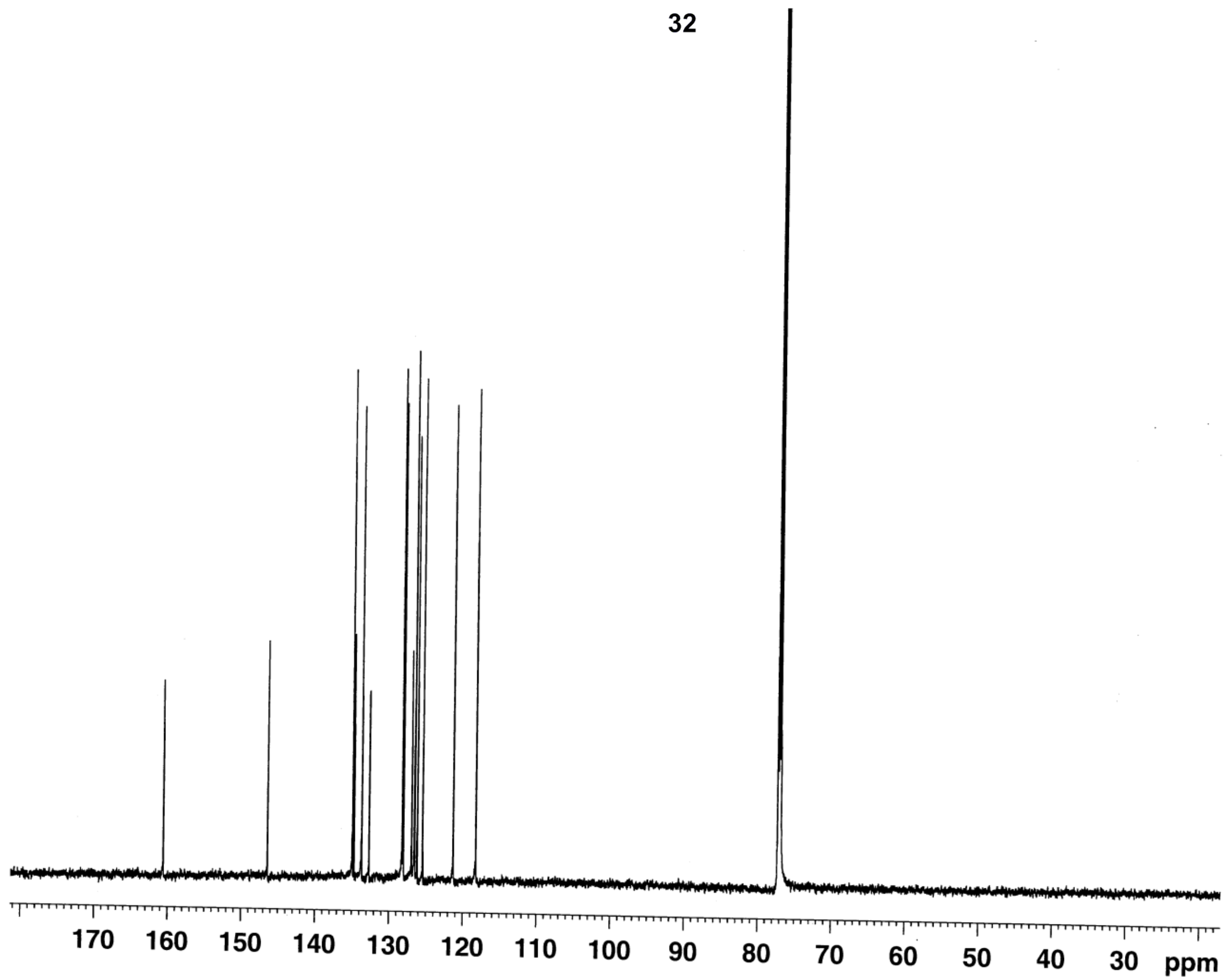


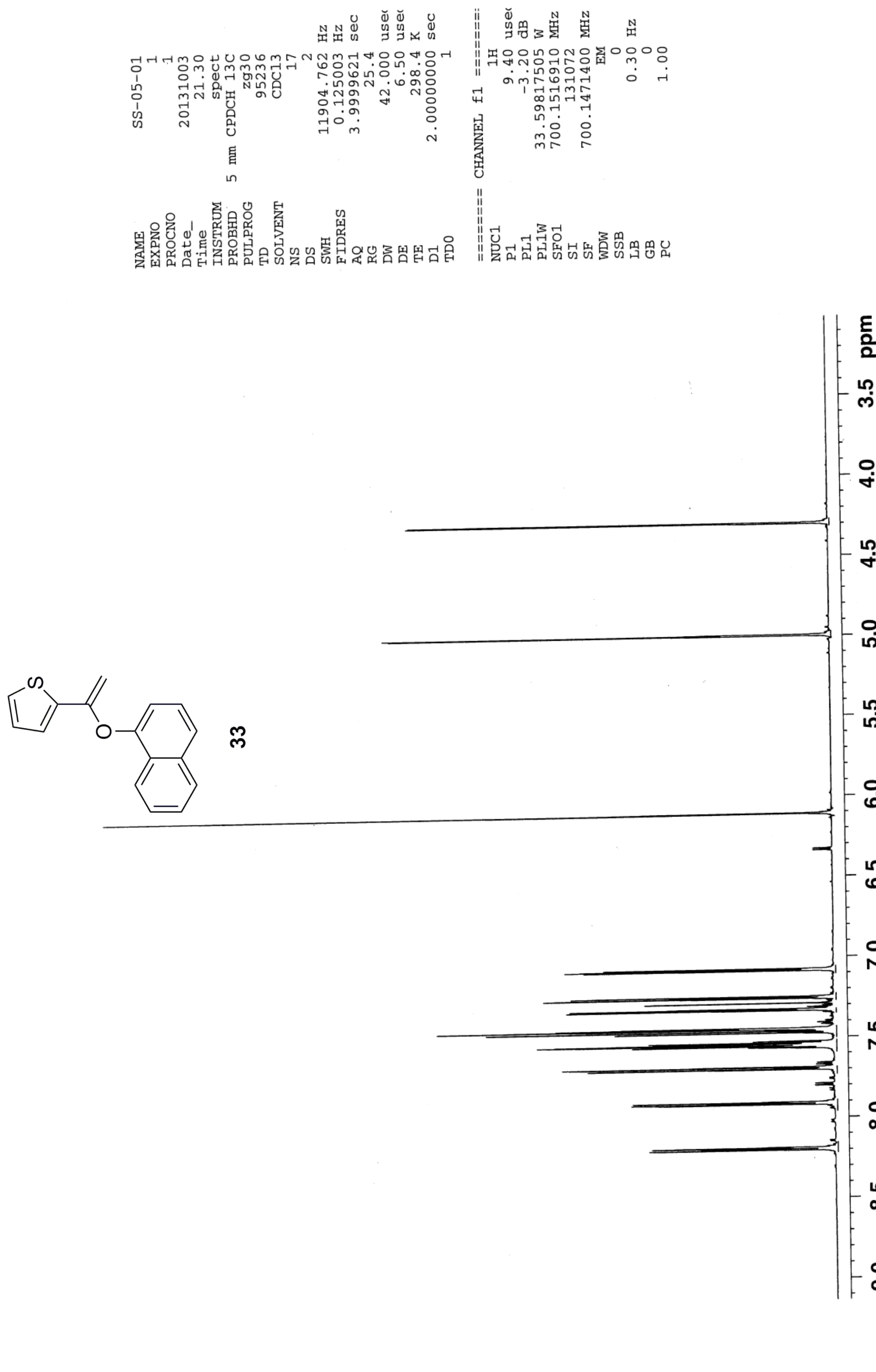
NAME	SS-05-128
EXPNO	1
PROCNO	1
Date_	20131005
Time	12.35
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zg30
TD	95236
SOLVENT	CDCl3
NS	10
DS	2
SWH	11904.762 Hz
FIDRES	0.125003 Hz
AQ	3.9999621 sec
RG	20.2
DW	42.000 usec
DE	6.50 usec
TE	298.4 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	9.40 usec
PL1	-3.20 dB
PL1W	33.59817505 W
SFO1	700.1516910 MHz
SI	131072
SF	700.1471400 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



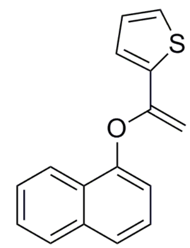
32



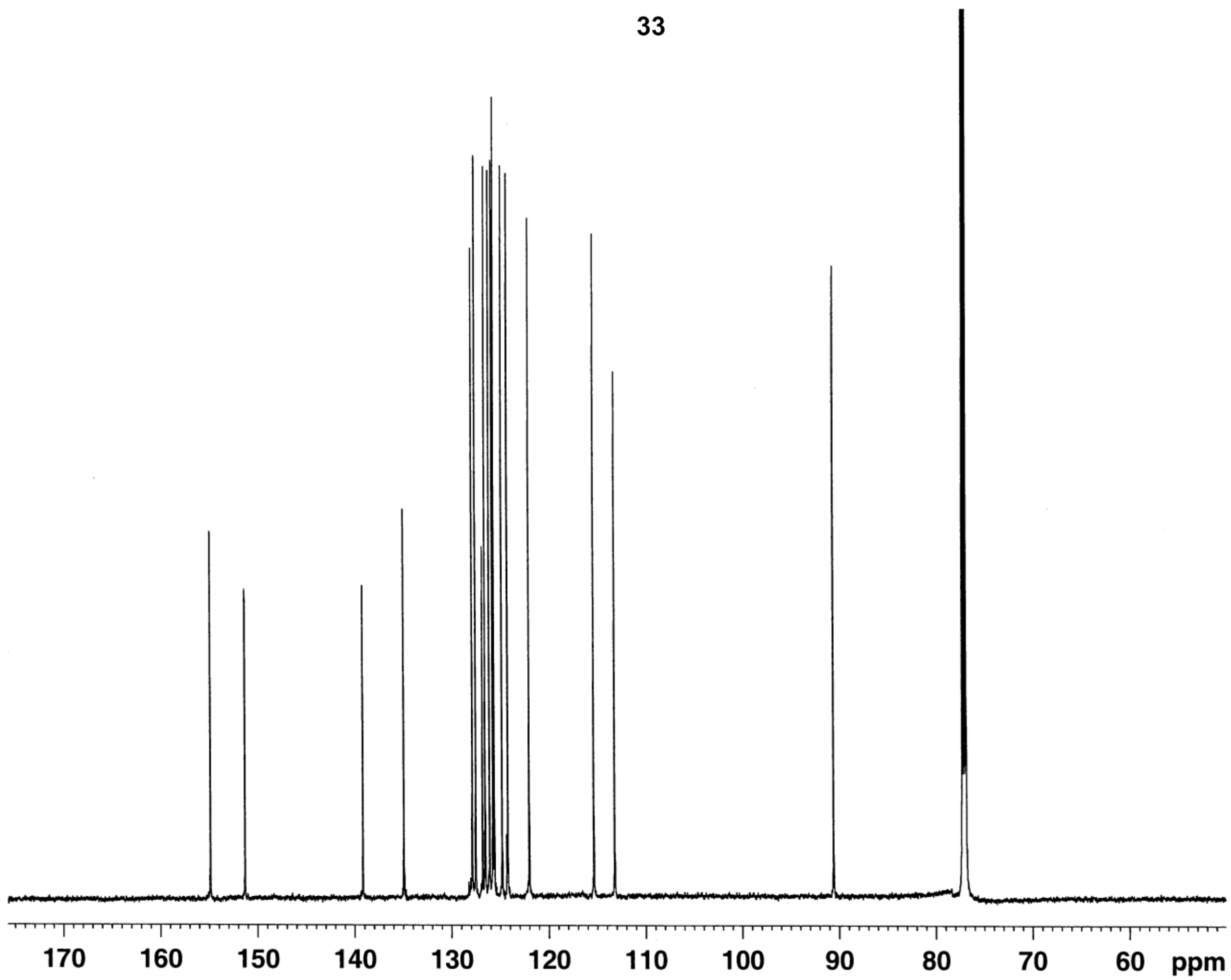
NAME	SS-05-128
EXPNO	2
PROCNO	1
Date_	20131005
Time	12.40
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	117
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 sec
RG	203
DW	12.000 usec
DE	16.50 usec
TE	298.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.00 usec
PL1	4.50 dB
PL1W	38.14553833 W
SFO1	176.0697436 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 usec
PL2	-3.20 dB
PL12	13.60 dB
PL13	120.00 dB
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1499406 MHz
SI	32768
SF	176.0521380 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40


NAME SS-05-01
EXPNO 1
PROCNO 1
Date_ 20131003
Time 21.30
INSTRUM spect
PROBHD 5 mm CPDCH 13C
PULPROG zg30
TD 95236
SOLVENT CDCl3
NS 17
DS 2
SWH 11904.762 Hz
FIDRES 0.125003 Hz
AQ 3.9999621 sec
RG 25.4
DW 42.000 use
DE 6.50 use
TE 298.4 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.40 use
PL1 -3.20 dB
PL1W 33.59817505 W
SFO1 700.1516910 MHz
SI 131072
SF 700.1471400 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
S
O
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
ppm
1.00
1.04
1.02
2.14
2.14
1.07
1.01
1.00
1.00
1.01

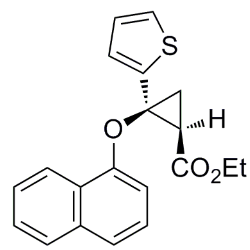
33



33



NAME	SS-05-01
EXPNO	2
PROCNO	1
Date_	20131003
Time	21.34
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	224
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 sec
RG	203
DW	12.000 usec
DE	16.50 usec
TE	298.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.00 usec
PL1	4.50 dB
PL1W	38.14553833 W
SFO1	176.0697436 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 usec
PL2	-3.20 dB
PL12	13.60 dB
PL13	120.00 dB
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1499406 MHz
SI	32768
SF	176.0521380 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40



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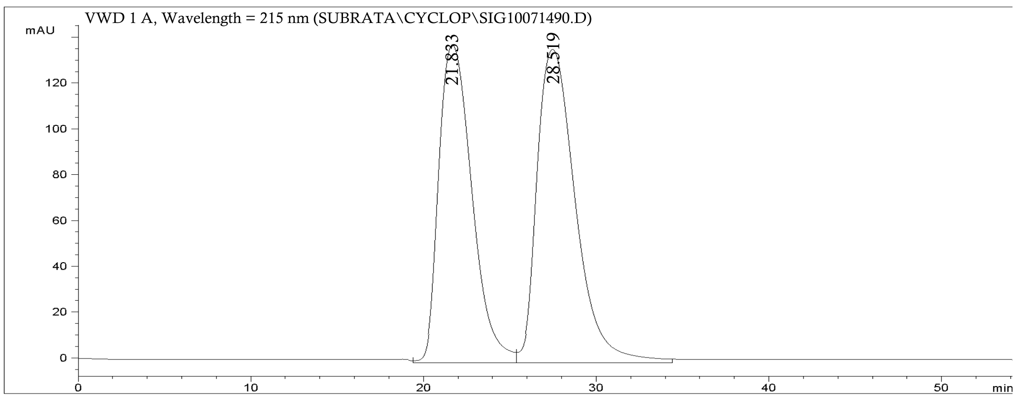
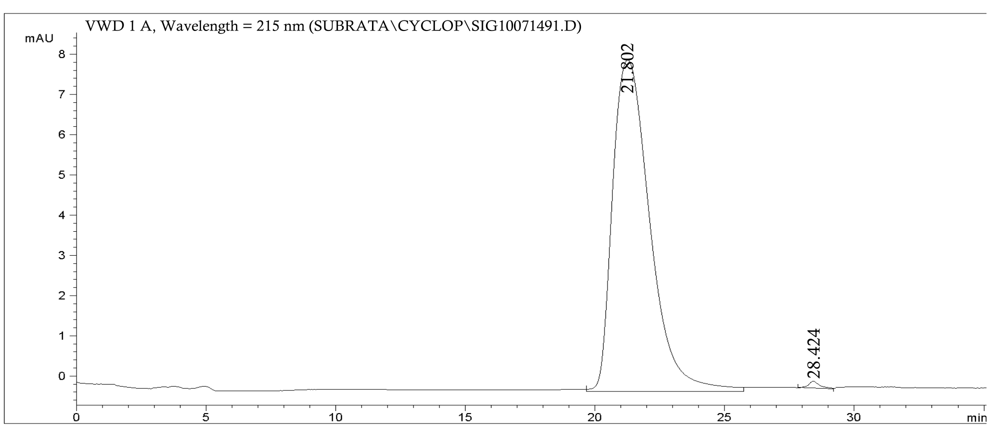
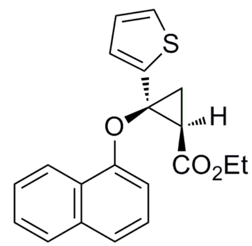


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	21.833	BB	1.0599	9634.32427	141.50624	49.1068
2	28.519	VB	1.1204	9984.80032	136.52637	50.8932



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	21.802	BB	1.1293	1319.93324	7.81106	97.1634
2	28.424	VB	0.0192	38.53429	0.17283	2.8366



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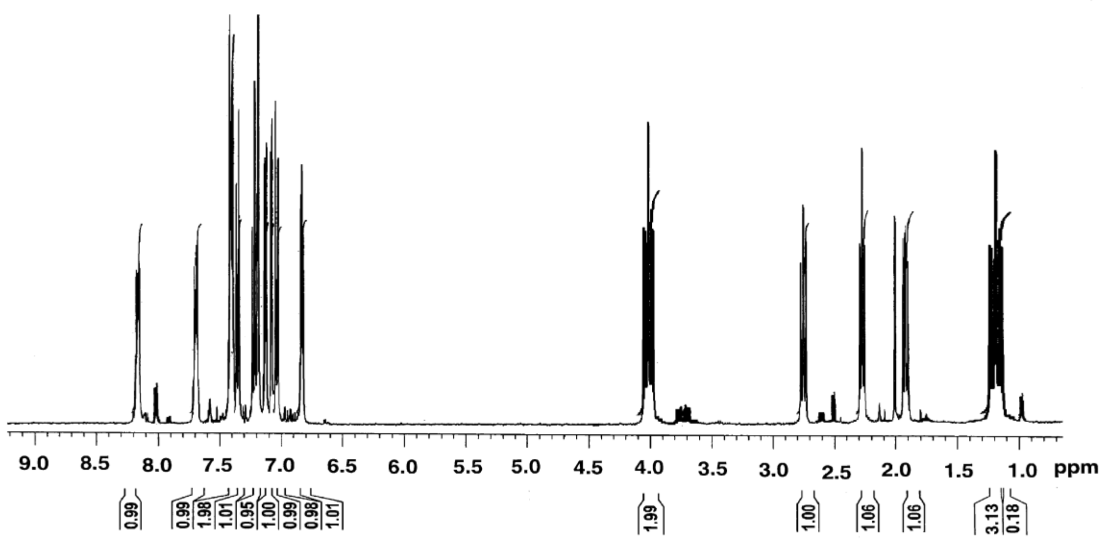
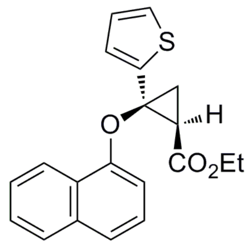



```
NAME            SS-05-130
EXPNO                   1
PROCNO                  1
Date_            20131005
Time                12.49
INSTRUM             spect
PROBHD   5 mm CPDCH 13C
PULPROG              zg30
TD                  95236
SOLVENT             CDCl3
NS                     32
DS                      2
SWH             11904.762 Hz
FIDRES           0.125003 Hz
AQ              3.9999621 sec
RG                   20.2
DW                 42.000 use
DE                   6.50 use
TE                  298.2 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.40 use
PL1                 -3.20 dB
PL1W          33.59817505 W
SFO1          700.1516910 MHz
SI                 131072
SF            700.1471400 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



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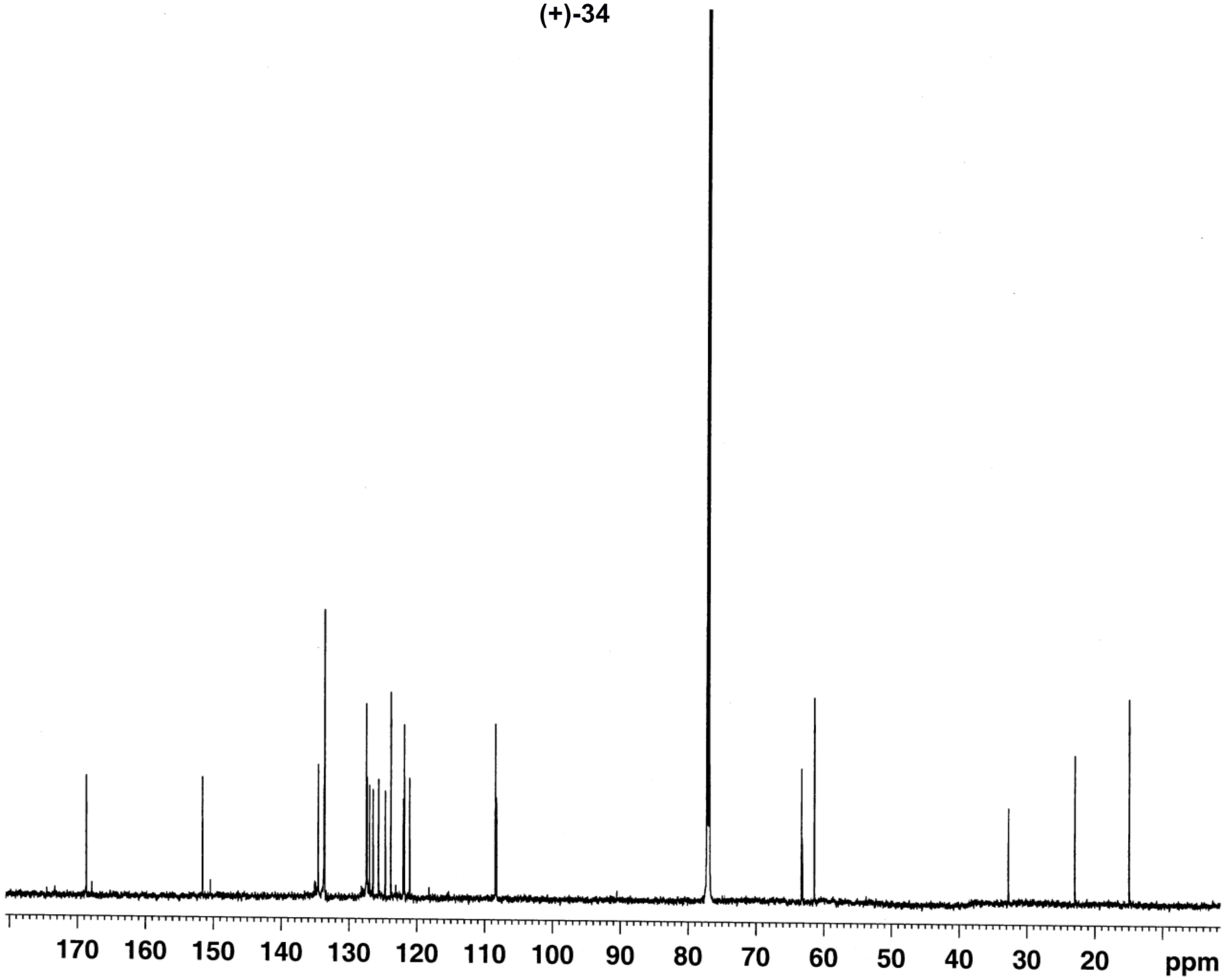
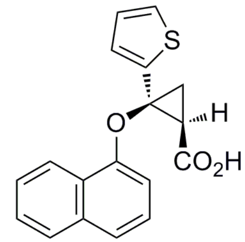



```
NAME              SS-05-130
EXPNO                     2
PROCNO                    1
Date_              20131005
Time                  12.57
INSTRUM               spect
PROBHD   5 mm CPDCH 13C
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       86
DS                        4
SWH              41666.668 Hz
FIDRES            0.635783 Hz
AQ               0.7864820 sec
RG                      203
DW                   12.000 use
DE                    16.50 use
TE                    298.2 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 =======
NUC1                    13C
P1                     9.00 use
PL1                    4.50 dB
PL1W            38.14553833 W
SFO1            176.0697436 MHz

======== CHANNEL f2 =======
CPDPRG2             waltz16
NUC2                     1H
PCPD2                 65.00 use
PL2                   -3.20 dB
PL12                  13.60 dB
PL13                 120.00 dB
PL2W            33.59817505 W
PL12W            0.70196527 W
PL13W            0.00000000 W
SFO2            700.1499406 MHz
SI                    32768
SF              176.0521380 MHz
WDW                      EM
SSB                       0
LB                     3.00 Hz
GB                        0
PC                     1.40
```



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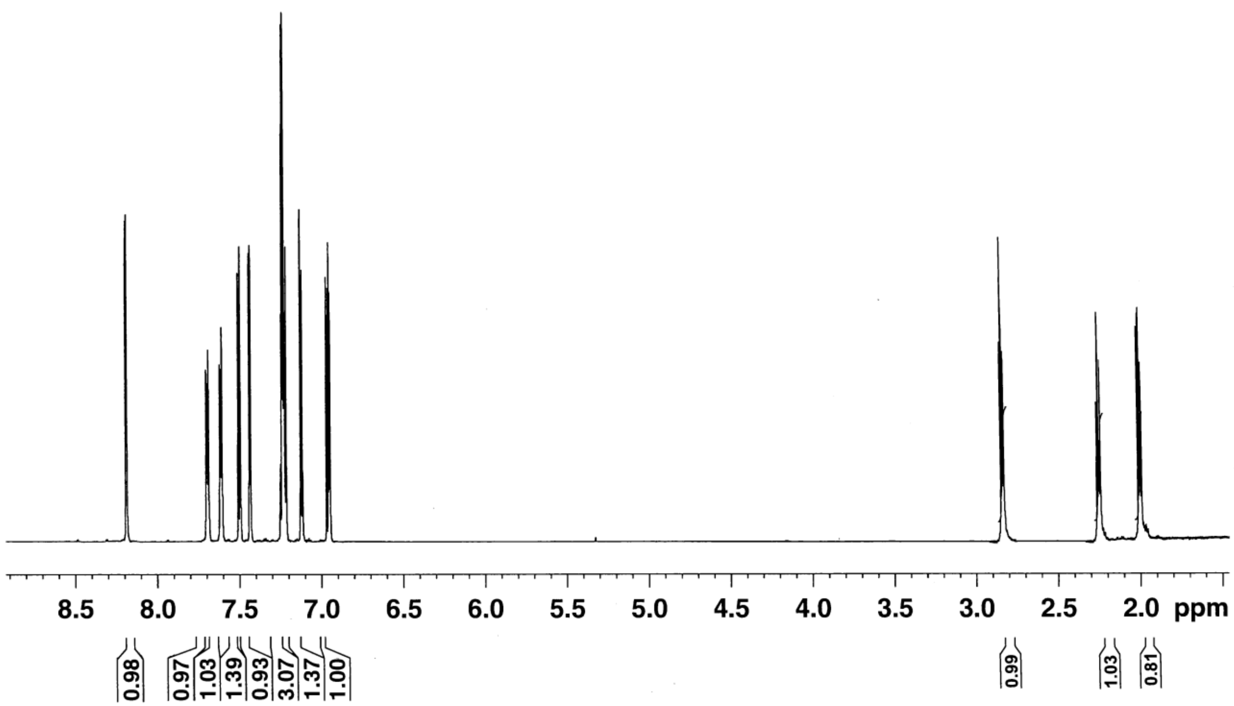
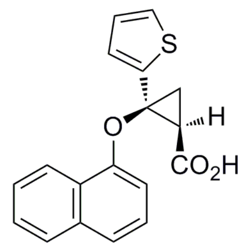



```
Current Data Parameters
NAME             SS-6-116
EXPNO                   1
PROCNO                  1

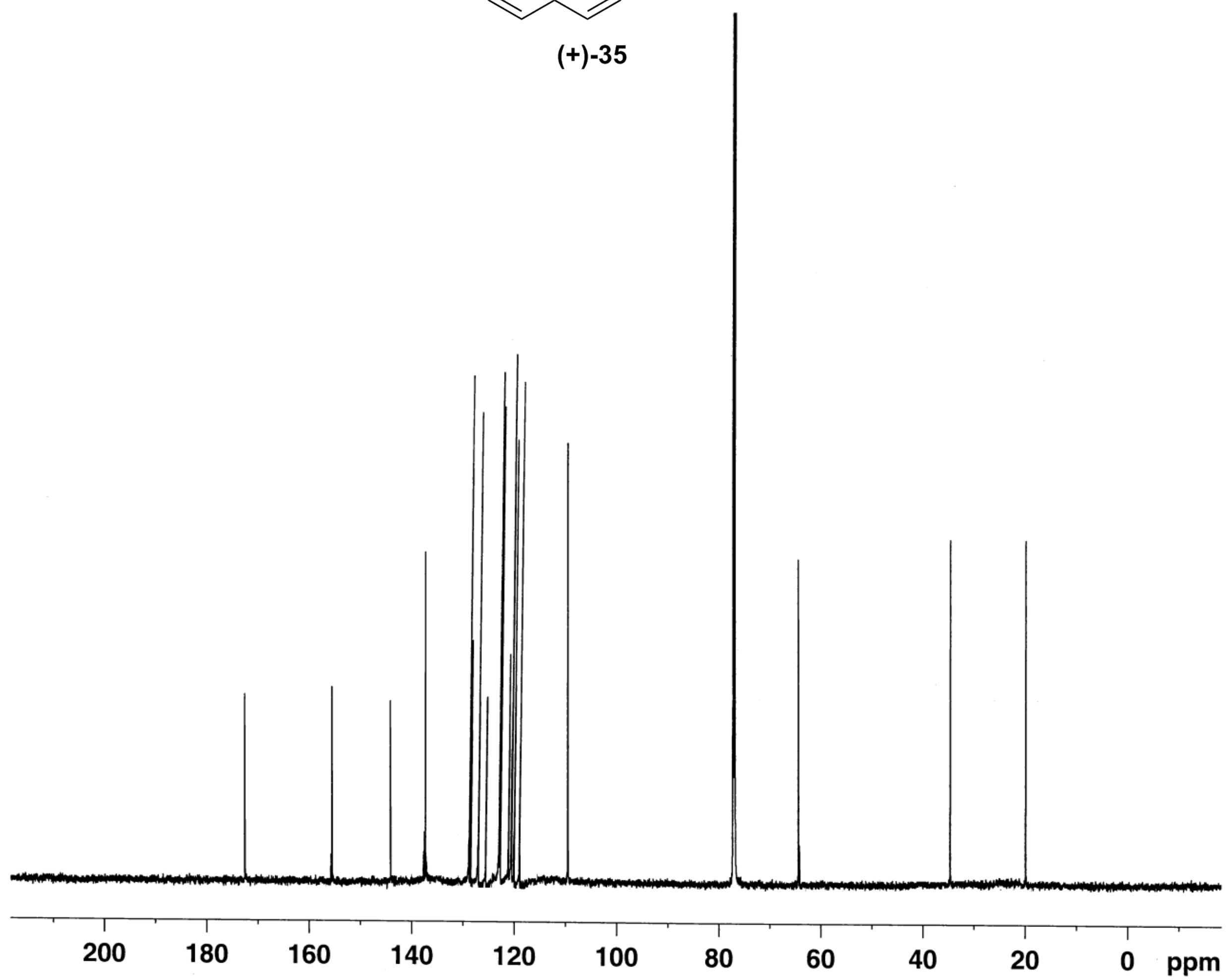
F2 - Acquisition Parameters
Date_            20130302
Time                21.39
INSTRUM             spect
PROBHD   5 mm PATXI 1H/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ              4.0894465 sec
RG                 131.79
DW                 62.400 usec
DE                   6.50 usec
TE                  298.0 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         500.1330008 MHz
NUC1                  1H
P1                  7.80 usec
PLW1         12.00000000 W

F2 - Processing parameters
SI                  65536
SF           500.1300000 MHz
WDW                    EM
SSB      0
LB                   0.30 Hz
GB       0
PC                   1.00
```



(+)-35



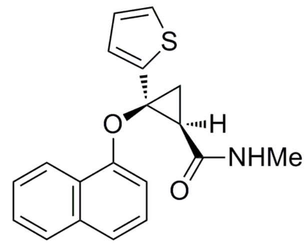
NAME	SS-06-116
EXPNO	2
PROCNO	1
Date_	20130323
Time	18.18
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	56
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 sec
RG	203
DW	12.000 use(
DE	16.50 use(
TE	298.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

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

NUC1	13C
P1	9.00 use(
PL1	4.50 dB
PL1W	38.14553833 W
SFO1	176.0697436 MHz

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

CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 use(
PL2	-3.20 dB
PL12	13.60 dB
PL13	120.00 dB
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1499406 MHz
SI	32768
SF	176.0521380 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00



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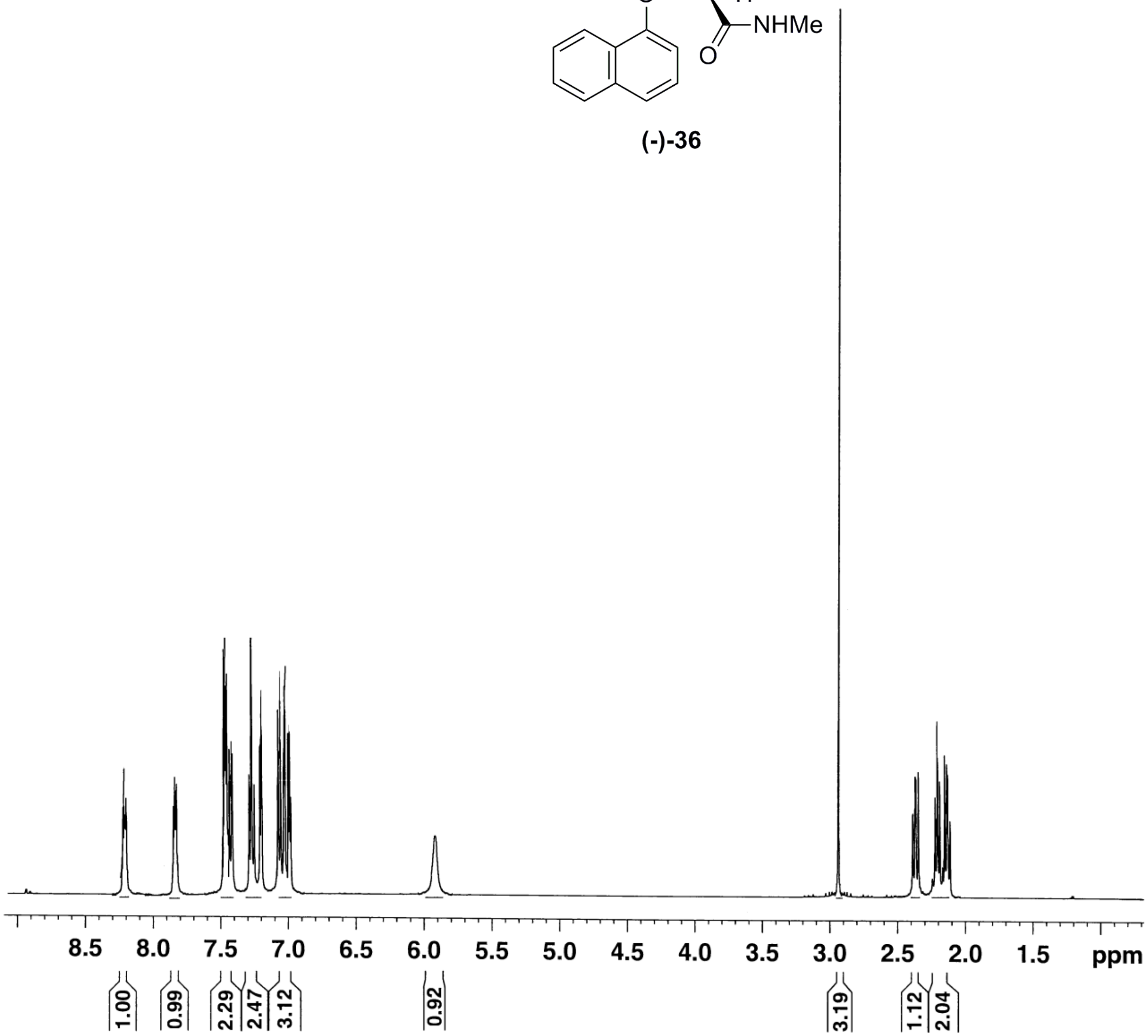
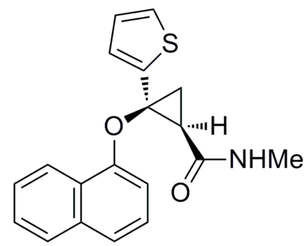



```
NAME              SS-08-09
EXPNO                    1
PROCNO                   1
Date_             20140109
Time                 19.12
INSTRUM              spect
PROBHD   5 mm CPDCH 13C
PULPROG               zg30
TD                   95236
SOLVENT              CDCl3
NS                      10
DS                       2
SWH             11904.762 Hz
FIDRES           0.125003 Hz
AQ              3.9999621 sec
RG                      18
DW                  42.000 usec
DE                    6.50 usec
TE                   298.5 K
D1              2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                    9.40 usec
PL1                  -3.20 dB
PL1W           33.59817505 W
SFO1           700.1516910 MHz
SI                  131072
SF             700.1471400 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



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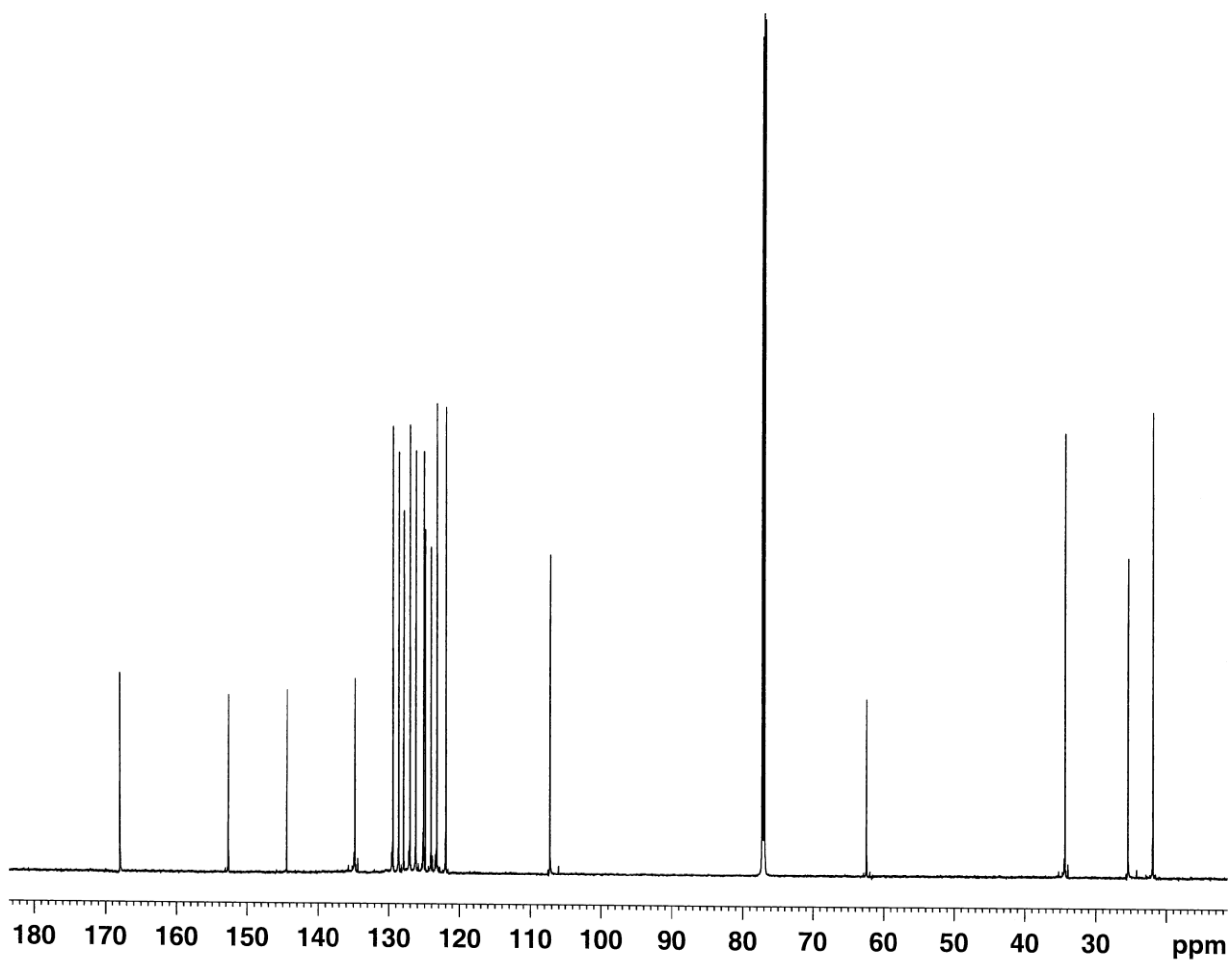


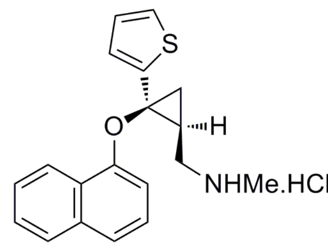
NAME	SS-08-09
EXPNO	2
PROCNO	1
Date_	20140109
Time	19.15
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	65
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 sec
RG	203
DW	12.000 usec
DE	16.50 usec
TE	298.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

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

NUC1	13C
P1	9.00 usec
PL1	4.50 dB
PL1W	38.14553833 W
SFO1	176.0697436 MHz

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

CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 usec
PL2	-3.20 dB
PL12	13.60 dB
PL13	120.00 dB
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1499406 MHz
SI	32768
SF	176.0521380 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40



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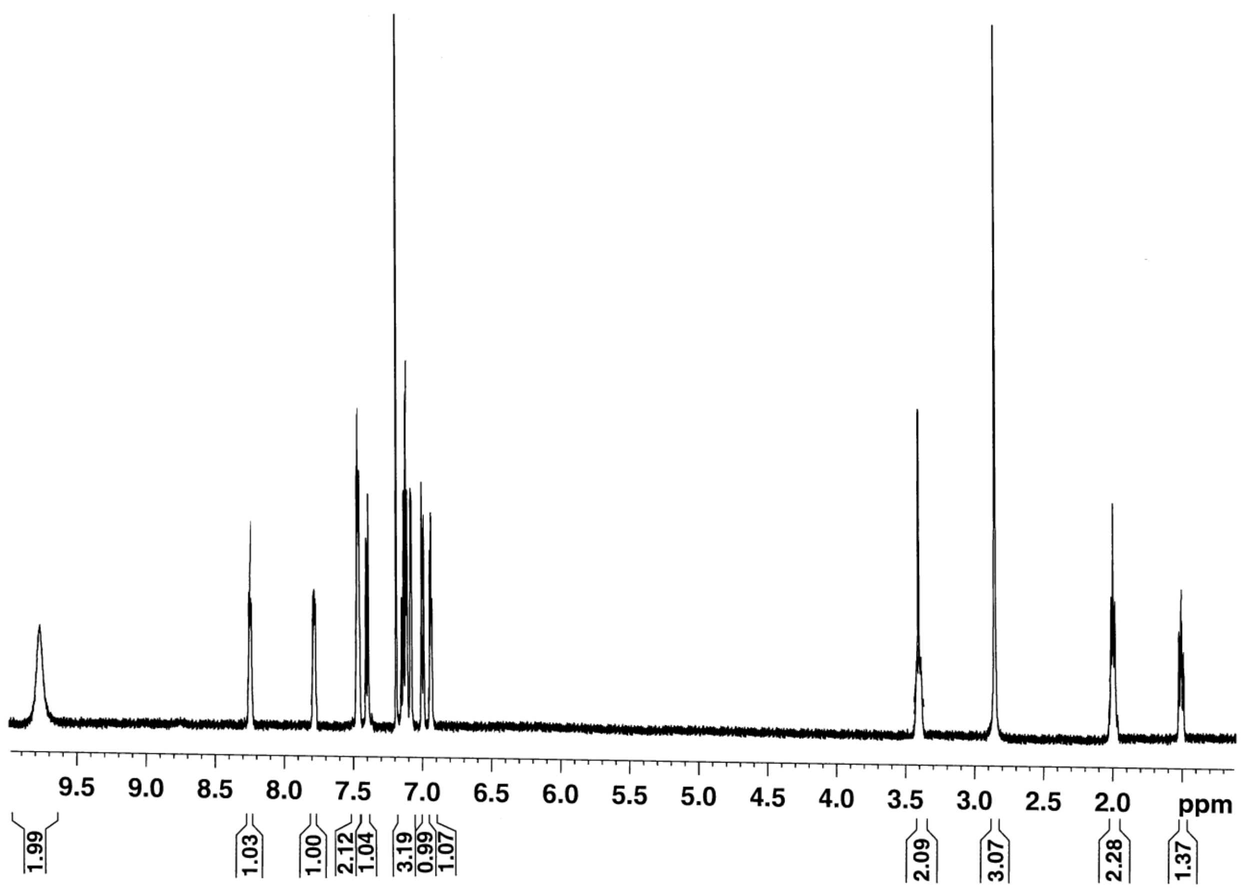
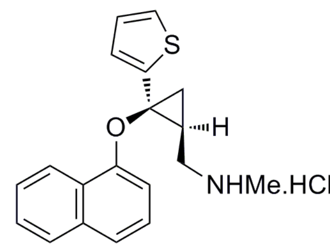


NAME	SS-08-10
EXPNO	1
PROCNO	1
Date_	20140109
Time	19.25
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zg30
TD	95236
SOLVENT	CDCl3
NS	32
DS	2
SWH	11904.762 Hz
FIDRES	0.125003 Hz
AQ	3.9999621 sec
RG	20.2
DW	42.000 usec
DE	6.50 usec
TE	298.2 K
D1	2.00000000 sec
TD0	1

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

NUC1	1H
P1	9.40 usec
PL1	-3.20 dB
PL1W	33.59817505 W
SFO1	700.1516910 MHz
SI	131072
SF	700.1471400 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



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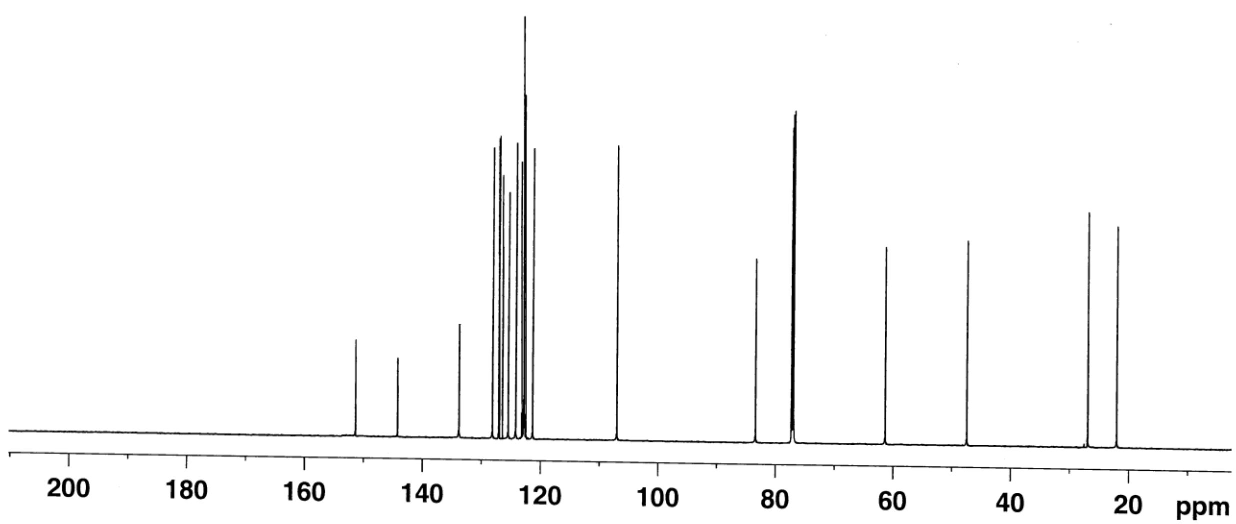

NAME	SS-08-10
EXPNO	2
PROCNO	1
Date_	20140109
Time	19.31
INSTRUM	spect
PROBHD	5 mm CPDCH 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	68
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 sec
RG	203
DW	12.000 usec
DE	16.50 usec
TE	298.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.00 usec
PL1	4.50 dB
PL1W	38.14553833 W
SFO1	176.0697436 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	65.00 usec
PL2	-3.20 dB
PL12	13.60 dB
PL13	120.00 dB
PL2W	33.59817505 W
PL12W	0.70196527 W
PL13W	0.00000000 W
SFO2	700.1499406 MHz
SI	32768
SF	176.0521380 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40