Catalytic Asymmetric Synthesis of Chiral Allylic Esters

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Supporting Information

Experimental Section

General Procedures. All reactions were carried out using oven-dried glassware under an atmosphere of Ar or N_2 unless otherwise indicated. [(R_p,*S*)-COP-OAc]$_2$ (**2**), [(R_p,*S*)-COP-NHCOCCl$_3$]$_2$ (**14**) and [(R_p,*S*)-COP-Cl]$_2$ (**12**) and their enantiomers were prepared according to published procedures.[1] Currently, both enantiomers of [(R_p,*S*)COP-OAc]$_2$ are commercially available from Aldrich Chemical Co. (661716 and 661708). (*Z*)-Allylic trichloroacetimidates were synthesized by reaction of the corresponding allylic alcohols with trichloroacetonitrile in the presence of 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU).[2] 5-Phenylpent-2-yn-1-ol was synthesized according to a published procedure,[3] and converted to (*Z*)-5-phenylpent-2-en-1-ol by reduction with P2 nickel.[4] Solid carboxylic acids were recrystallized from methanol. Acetic acid was distilled from acetic anhydride and CrO_3. Tributyltin hydride was synthesized from lithium aluminum hydride and tributyltin chloride. Dichloromethane and diethyl ether were dried by passage through activated alumina using a GlassContour solvent purification system. Tetrahydrofuran (THF) was purified by distillation from sodium metal. Ethyl acetate was used without further purification from EMD Chemicals, Inc. All other commercial reagents were used as received unless otherwise noted. Analytical thin layer chromatography (TLC) was carried out using 0.25 mm silica plates purchased from Merck. Eluted plates were visualized using UV light and anisaldehyde stain. Silica gel chromatography was performed using 230–400 mesh silica gel purchased from Merck. Determination of enantiomeric excess was carried out using HPLC, GC, and SFC analysis using enantioselective stationary phases. Samples of racemates were used to

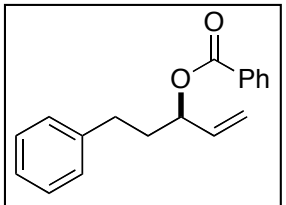
[1] (a) Anderson, C. E.; Kirsch, S. F.; Overman, L. E.; Richards, C. J.; Watson, M. P. *Org. Synth.* **2007**, *84*, 148–155 (b) Anderson, C. E.; Overman, L. E.; Richards, C. J.; Watson, M. P.; White, N. S. *Org. Synth.* **2007**, *84*, 139–147.
[2] Numata, M.; Sugimoto, M.; Koike, K.; Ogawa, T. *Carbohydr. Res.* **1987**, *163*, 209.
[3] Mori, M.; Tonogaki, K.; Kinoshita, A. *Org. Synth.* **2005**, *81*, 1–9.
[4] Brown, H. C.; Brown, C. A. *J. Am. Chem. Soc.* **1963**, *85*, 1005–1006.

calibrate enantioselective chromatographic analysis. Racemic mixtures of esters and aryl ethers were obtained by reaction of allylic trichloroacetimidates with acids in the presence of palladium(II) acetate [0.5 M CH_2Cl_2, rt, 5 mol% $Pd(OAc)_2$]. ^1H NMR (500 MHz) and ^{13}C NMR (125 MHz) spectra were obtained on Bruker FT NMR instruments. NMR spectra are reported as δ values in ppm relative to $CDCl_3$ calibrated to 7.27 ppm in ^1H NMR and 77.23 in ^{13}C NMR. Splitting patterns are abbreviated as follows: singlet (s), doublet (d), triplet (t), quartet (q), multiplet (m), broad (b), apparent (app) and combinations thereof. Infrared (IR) spectra were obtained using a Varian 640-IR FT-IR spectrometer as thin films from $CHCl_3$. Optical rotations were obtained using a JASCO J-1010 digital polarimeter. High-resolution mass spectra (HRMS) were obtained using a Waters-MicroMass Analytical LCT (ESI) spectrometer.

Experimental details and spectral data for the preparation of **4a**, **4b**, **4d**–**h**, **4j**, **4l**, **4r**, and **4s** can be found in the Supplementary Information that accompanies the preliminary communication.[5]

General Procedure Preparing Enantioenriched 3-Acyloxy-1-alkenes.

(*R*)-5-Phenyl-1-penten-3-yl Benzoate (4i).



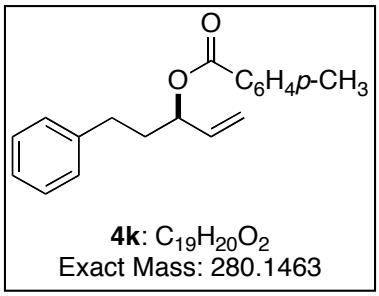
4i: $C_{18}H_{18}O_2$
Exact Mass: 266.1307

Allylic imidate **3e** (100 mg, 0.326 mmol) and benzoic acid (119 mg, 0.978 mmol) were dissolved in dichloromethane (0.33 mL). The flask was protected from light, and [(R_p,*S*)-COP-OAc]$_2$ (**2**) (5.0 mg, 0.0033 mmol) was added. After 17 h, ethylenediamine (0.10 mL, 1.63 mmol) was added. The crude reaction mixture was concentrated under reduced pressure and the resulting residue was purified by silica gel chromatography (10% ether/hexanes) to give 74 mg (85%) of **4i** as a clear, colorless oil. Enantioselective SFC analysis indicated a 93% enantiomeric

[5] Kirsch, S. F.; Overman, L. E. *J. Am. Chem. Soc.* **2005**, *127*, 2866–2867.

excess [OJ column; flow: 0.55 mL/min; 28% hexanes/72% CO_2; 230 nm; minor enantiomer, t_R = 17.1 min, major enantiomer, t_R= 18.8 min]: R_f 0.49 (25% EtOAc/hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.07 (d, *J* = 7.1 Hz, 2H,), 7.58 (t, *J* = 7.5 Hz, 1H), 7.47 (t, *J* = 7.9 Hz, 2H), 7.31–7.27 (m, 2H), 7.21–7.18 (m, 3H), 5.95 (ddd, *J* = 16.9, 10.6, 6.2 Hz, 1H), 5.55 (q, *J* = 6.3 Hz, 1H), 5.37 (d, *J* = 17.2 Hz, 1H), 5.26 (d, *J* = 10.6 Hz, 1H), 2.81–2.71 (m, 2H), 2.20–2.03 (m, 2H); ^{13}C NMR (125 MHz $CDCl_3$) δ 166.0. 141.5, 136.5, 133.1, 130.6, 129.8, 128.7, 128.6, 126.2, 117.2, 75.0, 36.2, 31.7; $[\alpha]^{25}_D$ –9.55 (*c* 0.99, $CHCl_3$); IR (thin film) 3064, 2928, 1719 cm^{-1}; HRMS (ESI) m/z calcd for $C_{18}H_{18}O_2Na$ (M + Na$^+$) 289.1205, found 289.1200.

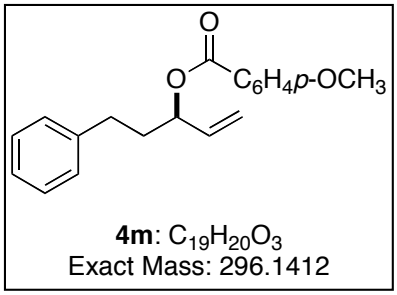
(*R*)-5-Phenyl-1-penten-3-yl *p*-Methylbenzoate (4k).



4k: $C_{19}H_{20}O_2$
Exact Mass: 280.1463

Following the general procedure, **4k** (40 mg, 85%) was obtained as a clear, colorless oil when **3e** (50 mg, 0.16 mmol) was reacted with *p*-methylbenzoic acid (65 mg, 0.48 mmol) and catalyst (R_p,*S*)-**2** (2 mg, 0.0016 mmol). Enantioselective SFC analysis indicated a 93% enantiomeric excess [OJ column; flow: 1.0 mL/min; 28% hexanes/72% CO_2; 230 nm; minor enantiomer, t_R = 15.3 min, major enantiomer, t_R = 16.8 min]: R_f 0.49 (25% EtOAc/hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.2 Hz, 2H), 7.32–7.28 (m, 4H), 7.23–7.20 (m, 3H), 5.96 (ddd, *J* = 17.0, 10.6, 6.1 Hz, 1H), 5.54 (q, *J* = 6.2 Hz, 1H), 5.36 (d, *J* = 17.2 Hz, 1H), 5.26 (d, *J* = 10.6 Hz, 1H), 2.79–2.75 (m, 2H), 2.45 (s, 3H), 2.19–2.07 (m, 2H); ^{13}C NMR (125 MHz $CDCl_3$) δ 166.1, 143.8, 141.6, 136.6, 129.9, 129.3, 128.7, 128.6, 127.9, 126.2, 117.1, 74.7, 36.2, 31.7, 21.9; $[\alpha]^{24}_D$ –7.81, $[\alpha]^{24}_{577}$ –10.2, $[\alpha]^{24}_{546}$ –14.0, $[\alpha]^{24}_{435}$ –26.1 (*c* 0.75, $CHCl_3$); IR (thin film) 3028, 2926, 2861, 1720, 1612 cm^{-1}; HRMS (ESI) m/z calcd for $C_{19}H_{20}O_2Na$ (M + Na$^+$) 303.1361, found 303.1351.

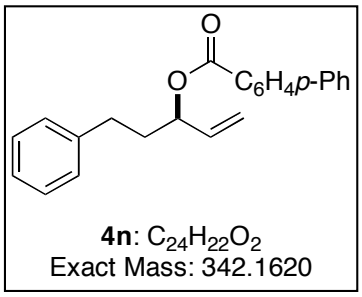
(*R*)-5-Phenyl-1-penten-3-yl 4-Methoxybenzoate (4m).



4m: $C_{19}H_{20}O_3$
Exact Mass: 296.1412

Following the general procedure, **4m** (38 mg, 79%) was obtained as a clear, colorless oil when **3e** (50 mg, 0.16 mmol) was reacted with *p*-methoxybenzoic acid (73 mg, 0.48 mmol) and catalyst (R_p,*S*)-**2** (2 mg, 0.0016 mmol). Enantioselective SFC analysis indicated a 95% enantiomeric excess [OJ column; flow: 1.0 mL/min; 28% hexanes/72% CO_2; 230 nm; minor enantiomer, t_R = 20.2 min, major enantiomer, t_R = 22.2 min]: R_f 0.49 (25% EtOAc/hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.03 (d, *J* = 8.9 Hz, 2H), 7.31–7.27 (m, 2H), 7.22–7.19 (m, 3H), 6.94 (d, *J* = 8.9 Hz, 2H), 5.94 (ddd, *J* = 17.1, 10.6, 6.1 Hz, 1H), 5.54 (q, *J* = 6.8 Hz, 1H), 5.36 (d, *J* = 17.2 Hz, 1H), 5.24 (d, *J* = 10.6 Hz, 1H), 3.88 (s, 3H), 2.78–2.74 (m, 2H), 2.17–2.05 (m, 2H); ^{13}C NMR (125 MHz $CDCl_3$) δ 165.8, 163.6, 141.6, 136.7, 131.8, 128.64, 128.57, 126.2, 123.1, 117.0, 113.8, 74.6, 55.6, 36.2, 31.7; $[\alpha]^{25}_D$ –1.98 (c = 0.94, $CHCl_3$); IR (thin film) 3079, 2936, 1712, 1606 cm^{-1}; HRMS (ESI) m/z calcd for $C_{19}H_{20}O_3Na$ ($M + Na^+$) 319.1310, found 319.1304.

(*R*)-5-Phenyl-1-penten-3-yl 4-Phenylbenzoate (4n).



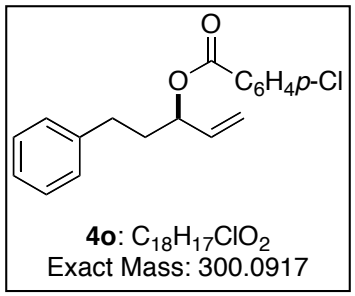
4n: $C_{24}H_{22}O_2$
Exact Mass: 342.1620

Following the general procedure, **4n** (53 mg, 95%) was obtained as a clear, colorless oil when **3e** (50 mg, 0.16 mmol) was reacted with *p*-phenylbenzoic acid (95 mg, 0.48 mmol) and catalyst (R_p,*S*)-**2** (2 mg, 0.0016 mmol). Enantioselective HPLC analysis indicated an 86% enantiomeric excess [OJ column; flow: 2.0 mL/min; 0.1% isopropanol/99.9% heptanes; 230 nm; minor enantiomer, t_R = 22.2 min, major enantiomer, t_R = 38.9 min][6]: R_f 0. 0.49 (25% EtOAc/hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.14 (d, *J* = 8.3 Hz, 2H), 7.69 (d, *J* = 8.3 Hz,

[6] Enantiomeric excess was determined after transesterification to **4i** (i. 1.2 equiv $LiAlH_4$, Et_2O; ii. 1.2 equiv BzCl, 1.2 equiv Et_3N, 0.1 equiv DMAP, CH_2Cl_2).

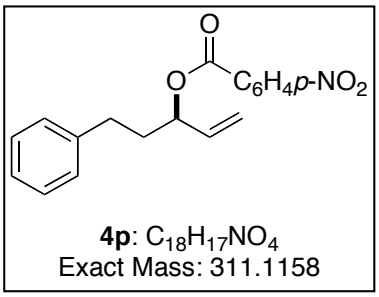
2H), 7.65 (d, *J* = 7.2 Hz, 2H), 7.50 (t, *J* = 7.3 Hz, 2H), 7.42 (t, *J* = 7.3 Hz, 1H), 7.32–7.20 (m, 5H), 5.97 (ddd, *J* = 16.9, 10.6, 6.2 Hz, 1H), 5.58 (q, *J* = 6.2 Hz, 1H), 5.39 (d, *J* = 17.2 Hz, 1H), 5.27 (d, *J* = 10.6 Hz, 1H), 2.81–2.73 (m, 2H), 2.23–2.06 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.9, 145.9, 141.5, 140.3, 136.5, 130.4, 129.4, 129.2, 128.7, 128.6, 128.4, 127.5, 127.3, 126.2, 117.2, 75.0, 36.2, 31.7; $[\alpha]^{24}_{D}$ –2.72, $[\alpha]^{24}_{577}$ –5.32, $[\alpha]^{24}_{546}$ –6.38, $[\alpha]^{24}_{435}$ –10.3, $[\alpha]^{24}_{405}$ –14.8 (c 0.78, $CHCl_3$); IR (thin film) 3026, 2925, 1948, 1723, 1613 cm^{-1}; HRMS (ESI) m/z calcd for $C_{24}H_{22}O_2Na$ ($M + Na^+$) 365.1518, found 365.1520.

(*R*)-5-Phenyl-1-penten-3-yl 4-Chlorobenzoate (4o).



Following the general procedure, **4o** (42 mg, 86%) was obtained as a clear, colorless oil when **3e** (50 mg, 0.16 mmol) was reacted with *p*-chlorobenzoic acid (75 mg, 0.48 mmol) and catalyst (R_p,*S*)-**2** (2 mg, 0.0016 mmol). Enantioselective SFC analysis indicated a 97% enantiomeric excess [OJ column; flow: 1.0 mL/min; 28% hexanes/72% CO_2; 230 nm; major enantiomer, t_R = 22.1 min, minor enantiomer, t_R = 23.8 min]: R_f 0. 0.49 (25% EtOAc/hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.5 Hz, 2H), 7.43 (d, *J* = 8.5 Hz, 2H), 7.29 (t, *J* = 7.6 Hz, 2H), 7.20 (m, 3H), 5.93 (ddd, *J* = 17.0, 10.6, 6.3 Hz, 1H), 5.53 (q, *J* = 6.3 Hz, 1H), 5.55 (d, 17.2 Hz, 1H), 5.27 (d, *J* = 10.6, 1H), 2.78–2.72 (m, 2H), 2.20–2.04 (m, 2H); ^{13}C NMR (125 MHz $CDCl_3$) δ 165.2, 141.4, 139.6, 136.2, 131.2, 129.1, 128.9, 128.7, 128.6, 126.2, 117.5, 75.3, 36.1, 31.7; $[\alpha]^{24}_{D}$ –6.86, $[\alpha]^{24}_{577}$ –6.49, $[\alpha]^{24}_{546}$ –8.97, $[\alpha]^{24}_{435}$ –18.1 (c = 1.08, $CHCl_3$); IR (thin film) 3026, 2927, 1720, 1594 cm^{-1}; HRMS (ESI) m/z calcd for $C_{18}H_{17}O_2ClNa$ ($M + Na^+$) 323.0815, found 323.0814.

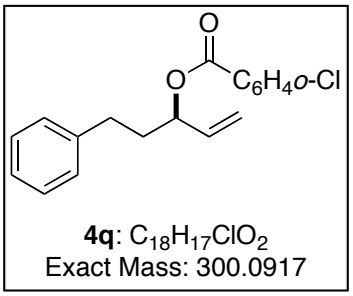
(*R*)-5-Phenyl-1-penten-3-yl *p*-Nitrobenzoate (4p).



4p: $C_{18}H_{17}NO_4$
Exact Mass: 311.1158

Following the general procedure, **4p** (33 mg, 65%) was obtained as a clear, colorless oil when **3e** (50 mg, 0.16 mmol) was reacted with *p*-nitrobenzoic acid (80 mg, 0.48 mmol) and catalyst (R_p,*S*)-**2** (2 mg, 0.0016 mmol). Enantioselective SFC analysis indicated 87% enantiomeric excess [OJ column; flow: 2.0 mL/min; 28% hexanes/72% CO_2; 230 nm; major enantiomer, t_R = 14.5 min, minor enantiomer, t_R = 19.1 min]: R_f 0.38 (9:1 hexanes/EtOAc); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.29 (d, *J* = 8.8 Hz, 2H), 8.18 (d, *J* = 8.8 Hz, 2H), 7.29 (app t, *J* = 7.7 Hz, 2H), 7.19 (m, 3H), 5.94 (ddd, *J* = 17.0, 10.6, 6.4 Hz, 1H), 5.56 (q, *J* = 6.4 Hz, 1H), 5.37 (d, 17.0 Hz, 1H), 5.30 (d, 10.6 Hz, 1H), 2.76 (t, *J* = 8.0 Hz), 2.22–2.09 (m, 2H); ^{13}C NMR (125 MHz $CDCl_3$) δ 164.1, 150.7, 141.2, 136.0, 135.8, 130.9, 128.8, 128.6, 126.3, 123.7, 118.1, 76.4, 35.9, 31.7; $[\alpha]^{24}_{D}$ –3.10, $[\alpha]^{24}_{577}$ –3.41, $[\alpha]^{24}_{546}$ –5.13, $[\alpha]^{24}_{435}$ –6.07 (c 0.28, $CHCl_3$); IR (thin film) 3027, 2928, 2860, 1725, 1528, 1275 cm^{-1}; HRMS (ESI) m/z calcd for $C_{18}H_{17}NO_4NH_4$ ($M + NH_4^+$) 329.1501, found 329.1509.

(*R*)-5-Phenyl-1-penten-3-yl *o*-Chlorobenzoate (4q).

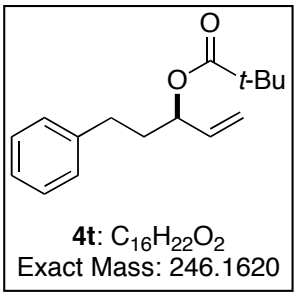


4q: $C_{18}H_{17}ClO_2$
Exact Mass: 300.0917

Following the general procedure, **4q** (12 mg, 24%) was obtained as a clear, colorless oil when **3e** (50 mg, 0.16 mmol) was reacted with *o*-chlorobenzoic acid (75 mg, 0.48 mmol) and catalyst (R_p,*S*)-**2** (2 mg, 0.0016 mmol). Enantioselective SFC analysis indicated 46% enantiomeric excess [OJ column; flow: 1.0 mL/min; 28% hexanes/72% CO_2; 230 nm; major enantiomer, t_R = 15.0 min, minor enantiomer, t_R = 16.3 min]: R_f 0.49 (25% EtOAc/hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.7 Hz, 1H), 7.48–7.42 (m, 2H), 7.35–7.27 (m, 3H), 7.22–7.20 (m, 3H), 5.94 (ddd, *J* = 17.1, 10.5, 6.5 Hz, 1H), 5.55

(q, J = 6.4 Hz, 1H), 5.39 (d, J = 17.2 Hz, 1H), 5.28 (d, J = 10.5 Hz, 1H), 2.79–2.74 (m, 2H), 2.19–2.05 (m, 2H); ^{13}C NMR (125 MHz $CDCl_3$) δ 165.3, 141.5, 136.1, 133.8, 132.7, 131.5, 131.3, 130.7, 128.7, 128.6, 126.8, 126.2, 117.9, 76.0, 36.1, 31.7; $[\alpha]^{24}_{D}$ –2.48, $[\alpha]^{24}_{577}$ –6.37, $[\alpha]^{24}_{546}$ –4.27, $[\alpha]^{24}_{435}$ –8.81 (c 0.38, $CHCl_3$); IR (thin film) 3022, 2921, 1733, 1599 cm^{-1}; HRMS (ESI) m/z calcd for $C_{18}H_{17}O_2ClNa$ (M + Na^+) 323.0815, found 323.0813.

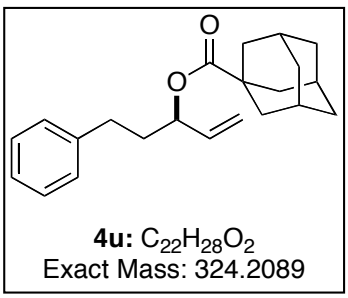
(*R*)-5-Phenyl-1-penten-3-yl Pivaloate (4t).



4t: $C_{16}H_{22}O_2$
Exact Mass: 246.1620

Following the general procedure, **4t** (38 mg, 95%) was obtained as a clear, colorless oil when **3e** (50 mg, 0.16 mmol) was reacted with pivalic acid (49 mg, 0.48 mmol) and catalyst (R_p,*S*)-**2** (2 mg, 0.0016 mmol).. Enantioselective HPLC analysis indicated a 94% enantiomeric excess [OJ column; flow: 2.0 mL/min; 0.1% isopropanol/99.9% heptanes; 230 nm; minor enantiomer, t_R = 22.2 min, major enantiomer, t_R = 38.9 min][6]: Spectral data matched that previously reported in the literature.[7] $[\alpha]^{24}_{D}$ –2.09, $[\alpha]^{24}_{577}$ –1.42, $[\alpha]^{24}_{546}$ –0.59 (c 1.03, $CHCl_3$).

(*R*)-5-Phenyl-1-penten-3-yl Adamantane-1-carboxylate (4u).



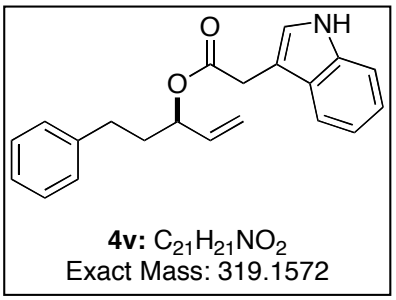
4u: $C_{22}H_{28}O_2$
Exact Mass: 324.2089

Following the general procedure, **4u** (36 mg, 68%) was obtained as a clear, colorless oil when **3e** (50 mg, 0.16 mmol) was reacted with 1-adamantanecarboxylic acid (86 mg, 0.48 mmol) and catalyst (R_p,*S*)-**2** (2 mg, 0.0016 mmol). **4u** was derivatized to the Mosher's ester (1. LAH, Et_2O, 2. (+)-MTPA, DCC, DMAP). ^{1}H NMR analysis indicated an enantiomeric excess of 86%:[8] R_f 0. 0.49 (25% EtOAc/hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30 (app q, J = 7.4 Hz, 2H), 7.22–7.17 (m, 3H), 5.83 (ddd, J = 17.0, 10.6, 5.9 Hz, 1H), 5.31–5.24 (m, 2H), 5.18

[7] Evans, P. A.; Leahy, D. K. *J. Am. Chem. Soc.* **2002**, *124*, 7882–7883.
[8] Dale, J. A.; Mosher, H. S. *J. Am. Chem. Soc.* **1973**, *95*, 512–519.

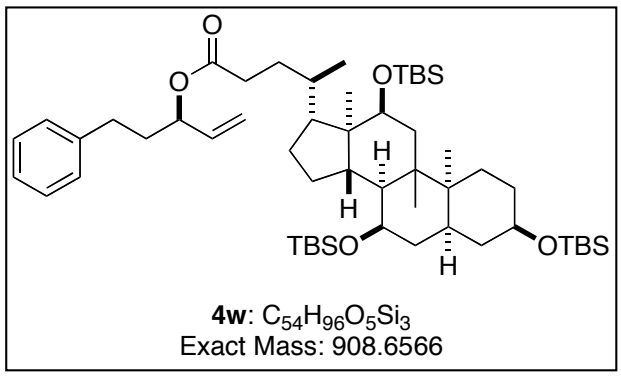
(d, J = 10.6 Hz, 1H), 2.70–2.63 (m, 2H), 2.05 (s, 3H), 2.00–1.90 (m, 8H), 1.75 (app t, J = 15.2 Hz, 6H); ^{13}C NMR (125 MHz $CDCl_3$) δ 177.0, 141.7, 136.8, 128.64, 128.55, 126.2, 116.4, 73.4, 41.1, 39.1, 36.7, 36.2, 31.6, 28.2; $[\alpha]^{24}_{D}$ –7.38, $[\alpha]^{24}_{577}$ –7.44, $[\alpha]^{24}_{546}$ –8.24, $[\alpha]^{24}_{435}$ –15.7, $[\alpha]^{24}_{405}$ –20.7 (c 1.72, $CHCl_3$); IR (thin film) 3024, 2906, 1726 cm^{-1}; HRMS (ESI) m/z calcd for $C_{22}H_{28}O_2Na$ (M + Na$^+$) 347.1987, found 347.1987.

(*R*)-5-Phenyl-1-penten-3-yl Indole-3-acetate (4v).



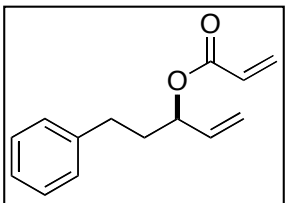
Following the general procedure, **4v** (34 mg, 65%) was obtained as a clear, colorless oil when **3e** (50 mg, 0.16 mmol) was reacted with indole-3-acetic acid (84 mg, 0.48 mmol) and catalyst (R_p,S)-**2** (2 mg, 0.0016 mmol). Enantioselective HPLC analysis indicated a 96% enantiomeric excess [OJ column; flow: 1.0 mL/min; 30% isopropanol/70% hexanes; 254 nm; minor enantiomer, t_R = 35.9 min, major enantiomer, t_R = 45.7 min]: R_f 0.27 (25% EtOAc/hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.14 (s, 1H), 7.70 (d, J = 7.9 Hz, 1H), 7.38 (d, J = 8.1 Hz, 1H), 7.30–7.09 (m, 6H), 7.08 (d, J = 7.2 Hz, 2H), 5.86 (ddd, J = 17.0, 10.6, 6.3 Hz, 1H), 5.34 (q, J = 6.3 Hz, 1H), 5.27 (d, J = 17.3 Hz, 1H), 5.21 (d, J = 10.6 Hz, 1H), 3.84 (s, 2H), 2.64–2.55 (m, 2H), 2.04–1.90 (m, 2H); ^{13}C NMR (125 MHz $CDCl_3$) δ 171.6, 141.5, 136.4, 136.3, 128.6, 128.5, 127.4, 126.1, 123.2, 122.4, 119.9, 119.1, 117.2, 111.4, 108.7, 74.7, 36.1, 31.8, 31.5; $[\alpha]^{24}_{D}$ 5.89, $[\alpha]^{24}_{577}$ 5.41, $[\alpha]^{24}_{546}$ 6.43, $[\alpha]^{24}_{435}$ 11.2 (c 1.10, $CHCl_3$); IR (thin film) 3415, 3059, 2927, 1726 cm^{-1}; HRMS (ESI) m/z calcd for $C_{21}H_{21}O_2NNa$ (M + Na$^+$) 342.1470, found 342.1466.

(*R*)-5-Phenyl-1-penten-3-yl Tris(tert-butyldimethylsilyl)cholate (4w).



Following the general procedure, **4w** (53 mg, 91%) was obtained as a clear, colorless oil when **3e** (20 mg, 0.065 mmol) was reacted with TBS-protected cholic acid (150 mg, 0.20 mmol) and catalyst (R_p,*S*)-**2** (2 mg, 0.00065 mmol). Enantioselective HPLC analysis indicated a 91% diastereomeric excess [OJ column; flow: 2.0 mL/min; 0.1% isopropanol/99.9% heptanes; 230 nm; minor enantiomer, t_R = 22.2 min, major enantiomer, t_R = 38.9 min][7]: R_f 0. 0.49 (25% EtOAc/hexanes); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 (t, *J* = 7.4 Hz, 2H), 7.22–7.17 (m, 3H), 5.83 (ddd, *J* = 17.1, 10.5, 6.4 Hz, 1H), 5.30–5.25 (m, 2H), 5.20 (d, *J* = 10.5 Hz, 1H), 3.98 (s, 1H), 3.80 (s, 1H), 3.39–3.30 (m, 1H), 2.69–2.63 (m, 2H), 2.39–2.14 (m, 5H), 2.00–1.85 (m, 6H), 1.71–1.59 (m, 2H), 1.55–1.40 (m, 7H), 1.39–1.23 (m, 4H), 0.97–0.86 (m, 36H), 0.67 (s, 3H), 0.10–0.01 (m, 18H); ^{13}C NMR (125 MHz $CDCl_3$) δ 173.7, 141.6, 136.7, 128.64, 128.57, 126.2, 117.2, 74.2, 74.0, 72.3, 69.8, 47.5, 46.2, 42.2, 41.6, 41.1, 40.8, 36.3, 36.1, 35.6, 35.1, 35.0, 32.3, 31.7, 31.5, 31.3, 29.9, 28.7, 27.9, 26.7, 26.6, 26.3, 25.9, 24.1, 23.2, 18.6, 18.1, 13.0, 1.3, –2.7, –4.0, –4.4, –5.2; $[\alpha]^{24}_D$ 22.6, $[\alpha]^{24}_{577}$ 24.0, $[\alpha]^{24}_{546}$ 26.2, $[\alpha]^{24}_{435}$ 41.9 (*c* 2.44, $CHCl_3$); IR (thin film) 2934, 2859, 1735, 1466 cm^{-1}; HRMS (ESI) m/z calcd for $C_{53}H_{94}O_5Si_3Na$ (M + Na^+) 917.6307, found 917.6313.

(*R*)-5-Phenyl-1-penten-3-yl Acrylate (4x).

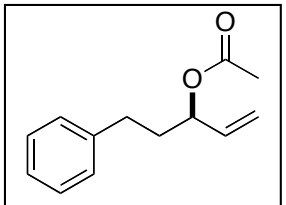


4x: $C_{14}H_{16}O_2$
Exact Mass: 216.1150

Following the general procedure, **4x** (13 mg, 37%) was obtained as a clear, colorless oil from **3e** (50 mg, 0.16 mmol) was reacted with acrylic acid (35 mg, 0.48 mmol) and catalyst (R_p,*S*)-**2** (2 mg, 0.0016 mmol): R_f 0.49 (25% EtOAc/hexanes); Spectral data matched those previously reported in the literature.[9]

General Procedure for Enantioselective One-Pot Synthesis of 3-Acyloxy-1-alkenes from Prochiral (*Z*)-Allylic Alcohols

(*R*)-5-Phenyl-1-penten-3-yl Acetate (4c).



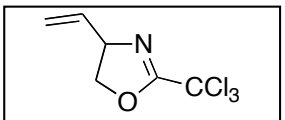
4c: $C_{13}H_{16}O_2$
Exact Mass: 204.1150

DBU (9 μL, 1M solution in CH_2Cl_2) was added to a solution of 5-phenylpent-2-en-1-ol (50 mg, 0.31 mmol), trichloroacetonitrile (31 μL, 0.31 mmol) and CH_2Cl_2 (0.3 mL). The solution was maintained at room temperature for 7 h, then acetic acid (53 μL, 0.93 mmol) was added, followed by [(R_p,*S*)-COP-OAc]$_2$ (**2**, 4.7 mg, 1 mol%). After 17 h, the solution was concentrated under reduced pressure and the residue was purified by silica gel chromatography (10% Et_2O/hexanes) to provide 49 mg (78%) of **4c** as a clear, colorless oil: Enantioselective GC analysis indicated 91% enantiomeric excess [Cyclosil-B; flow: 1.5 mL/min; 40 min at 130 °C; major enantiomer, t_R = 28.15 min, minor enantiomer, t_R = 30.36 min]: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30 (app t, *J* = 7.4 Hz, 2H), 7.20 (dd, *J* = 11.7, 7.2 Hz, 3H), 5.83 (ddd, *J* = 17.1, 10.6, 6.2 Hz, 1H), 5.29 (q, *J* = 6.5 Hz, 1H), 5.28 (d, *J* = 17.1 Hz, 1H), 5.22 (d, *J* = 10.5 Hz, 1H), 2.70–2.65 (m, 2H), 2.08 (s, 3H), 2.06–1.88 (m, 2H); ^{13}C NMR (125 MHz $CDCl_3$)

[9] Binder, J. T.; Kirsch, S. F. *Chem. Commun.* **2007**, 4164–4166.

δ 170.5, 141.5, 136.5, 128.6, 128.5, 126.2, 117.1, 74.5, 36.0, 31.6, 21.4; $[\alpha]^{24}_{D}$ 36.7, $[\alpha]^{24}_{577}$ 41.1 (c 1.31, $CHCl_3$); IR (thin film) 3028, 2950, 1739, 1604, 1238 cm^{-1}; HRMS (ESI) m/z calcd for $C_{13}H_{16}O_2Na$ ($M + Na^+$) 227.1048, found 227.1041.

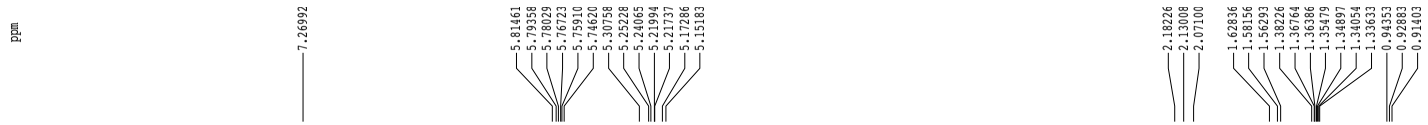
2-(Trichloromethyl)-4,5-dihydro-4-vinyloxazole (5).

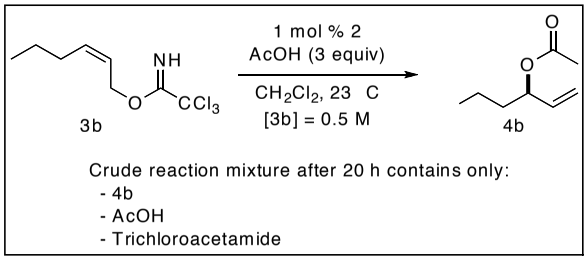


5: $C_6H_6Cl_3NO$
Exact Mass: 212.9515

(R_p,*S*)-[COP-OAc]$_2$ (**2**, 11.3 mg, 5 mol %) was added to a solution of imidate **3a** (40 mg, 0.15 mmol) in CH_2Cl_2 (0.3 mL, 0.5 M), and the solution was heated to 38 °C and overnight. At completion (TLC), the reaction mixture was concentrated under reduced pressure and the residue was purified by silica gel chromatography (15% EtOAc/hexanes) to give 13 mg (41%) of **5** as a clear, colorless oil and 10 mg (38%) of **4a** as a clear, colorless oil. Characterization data for **5**: R_f 0.56 (10% EtOAc/hexanes). ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.35 (t, *J* = 8.3 Hz, 1H), 4.74–4.78 (m, 1H), 4.84–4.89 (m, 1H), 5.25–5.34 (m, 2H), 5.88 (ddd, *J* = 17.2, 10.1, 6.3 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.1, 135.6, 118.1, 86.5, 75.7, 68.8; IR (thin film) 2988, 2907, 1770, 1707, 1514 cm^{-1} HRMS (ESI) m/z calcd for $C_6H_7Cl_3NO$ $(M + H)^+$ 213.9593, found 213.9589.

sk-ii-121 crude, 20 h
1H spectrum





Crude reaction mixture after 20 h contains only:
- 4b
- AcOH
- Trichloroacetamide

Current Data Parameters
USER cannon
NAME sk-ii-121crude
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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PROBHD 5 mm broadban
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 406.4
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

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P1 13.00 usec
PL1 -1.00 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200263 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
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F2P -1.062 ppm
F2 -531.20 Hz
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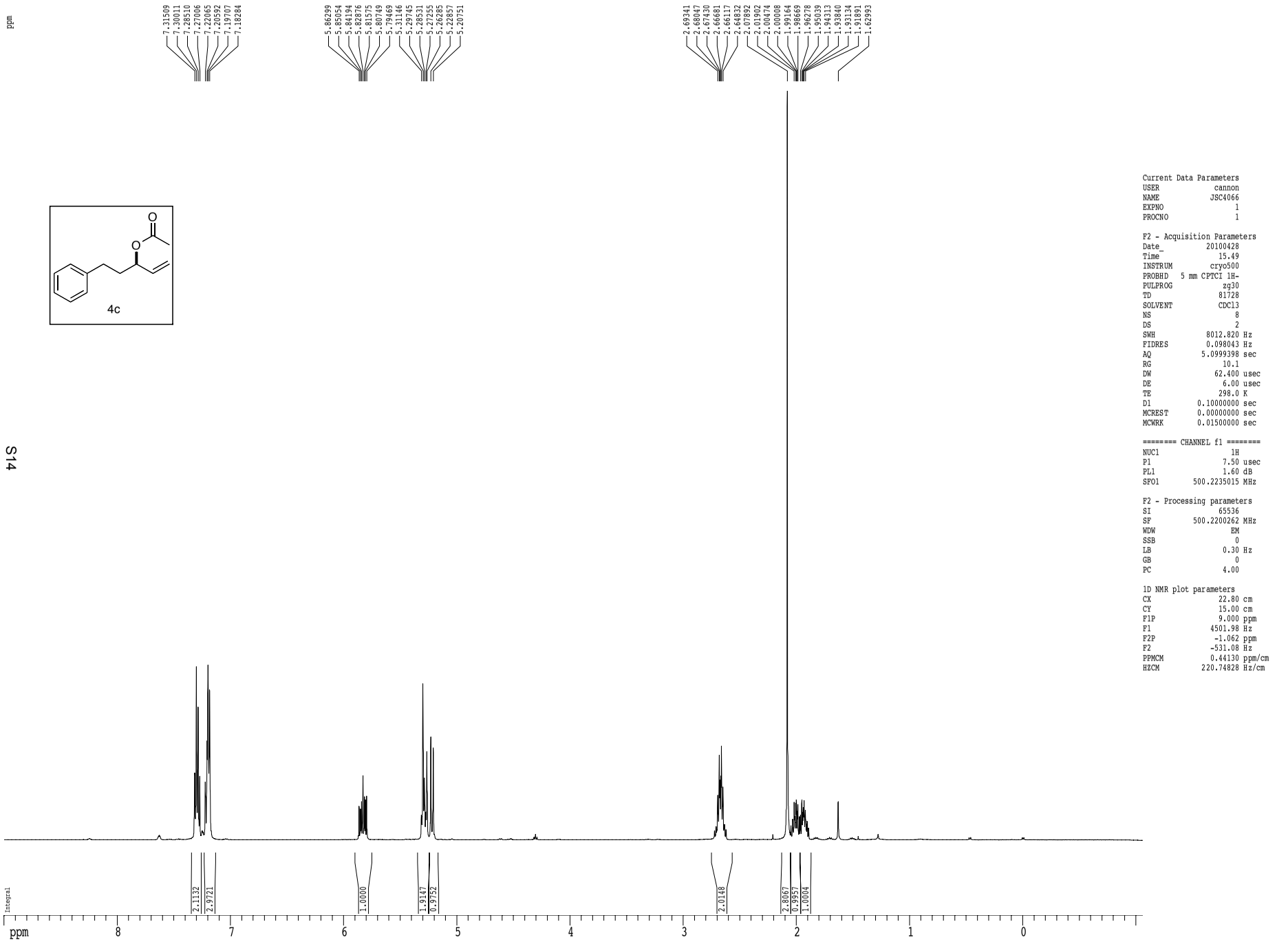
HZCM 220.75362 Hz/cm

1H spectrum



Current Data Parameters

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EXPNO	1
PROCNO	1

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TD	81728
SOLVENT	CDCl3
NS	8
DS	2
SWH	8012.820 Hz
FIDRES	0.098043 Hz
AQ	5.0999398 sec
RG	10.1
DW	62.400 usec
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======== CHANNEL f1 ========

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P1	7.50 usec
PL1	1.60 dB
SFO1	500.2235015 MHz

F2 - Processing parameters

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SF	500.2200262 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	22.80 cm
CY	15.00 cm
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F1	4501.98 Hz
F2P	-1.062 ppm
F2	-531.08 Hz
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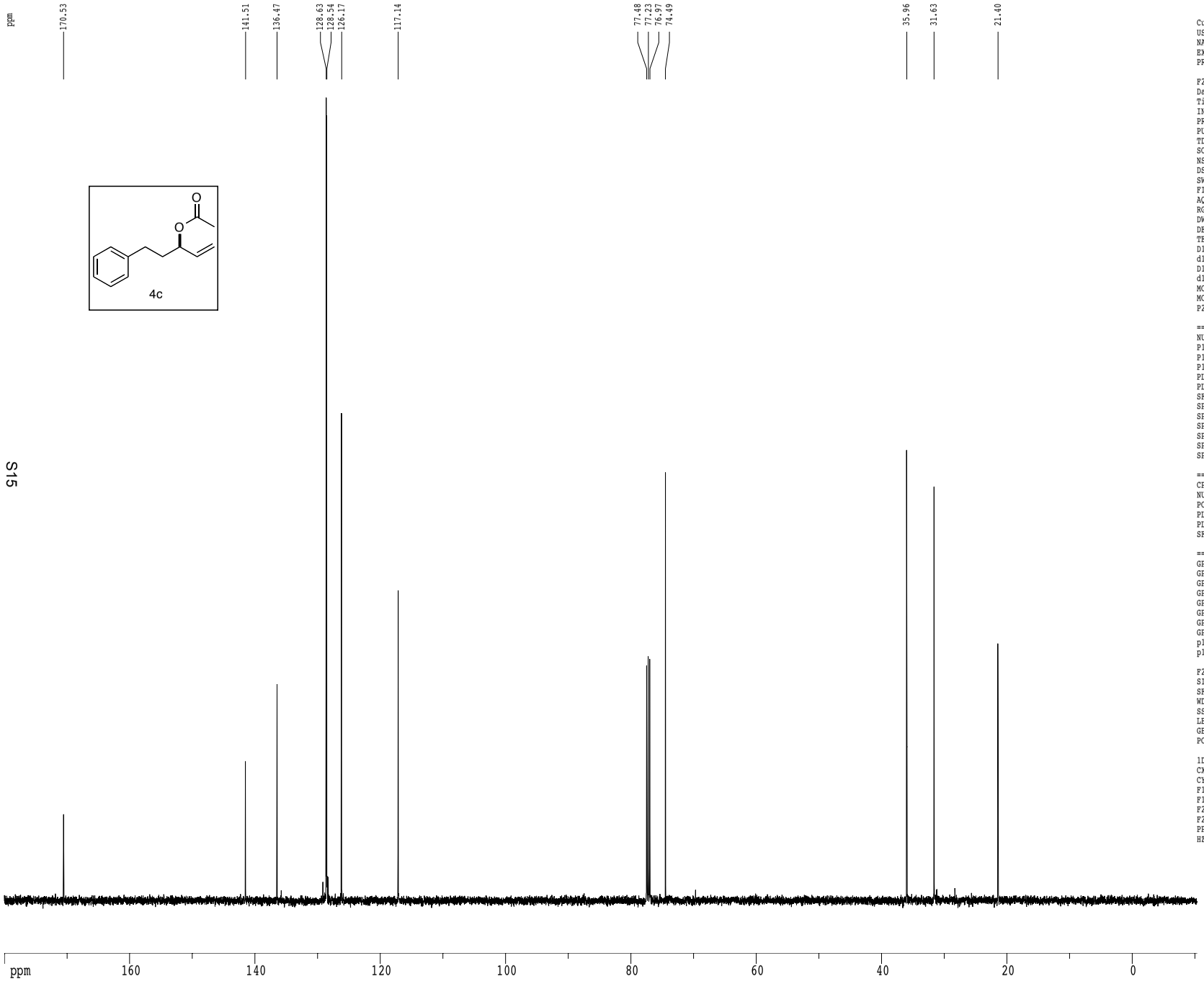
Z-restored spin-echo 13C spectrum with 1H decoupling

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EXPNO                 2
PROCNO                1

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PULPROG  SpinEchopg30gp.prd
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SOLVENT            CDCl3
NS                   96
DS                   16
SWH           30303.031 Hz
FIDRES        0.462388 Hz
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RG               5160.6
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d17           0.00019600 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
P2               31.00 usec

======== CHANNEL f1 ========
NUC1                 13C
P1                15.50 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1               3.20 dB
SP2               3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1               0.00 Hz
SPOFF2               0.00 Hz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1          SINE.100
GPNAM2          SINE.100
GPX1                 0.00 %
GPX2                 0.00 %
GPY1                 0.00 %
GPY2                 0.00 %
GPZ1                30.00 %
GPZ2                50.00 %
p15               500.00 usec
p16              1000.00 usec

F2 - Processing parameters
SI                   65536
SF           125.7804029 MHz
WDW                     EM
SSB                      0
LB                  1.00 Hz
GB                       0
PC                   2.00

1D NMR plot parameters
CX                  22.80 cm
CY                  15.65 cm
F1P               180.000 ppm
F1              22640.47 Hz
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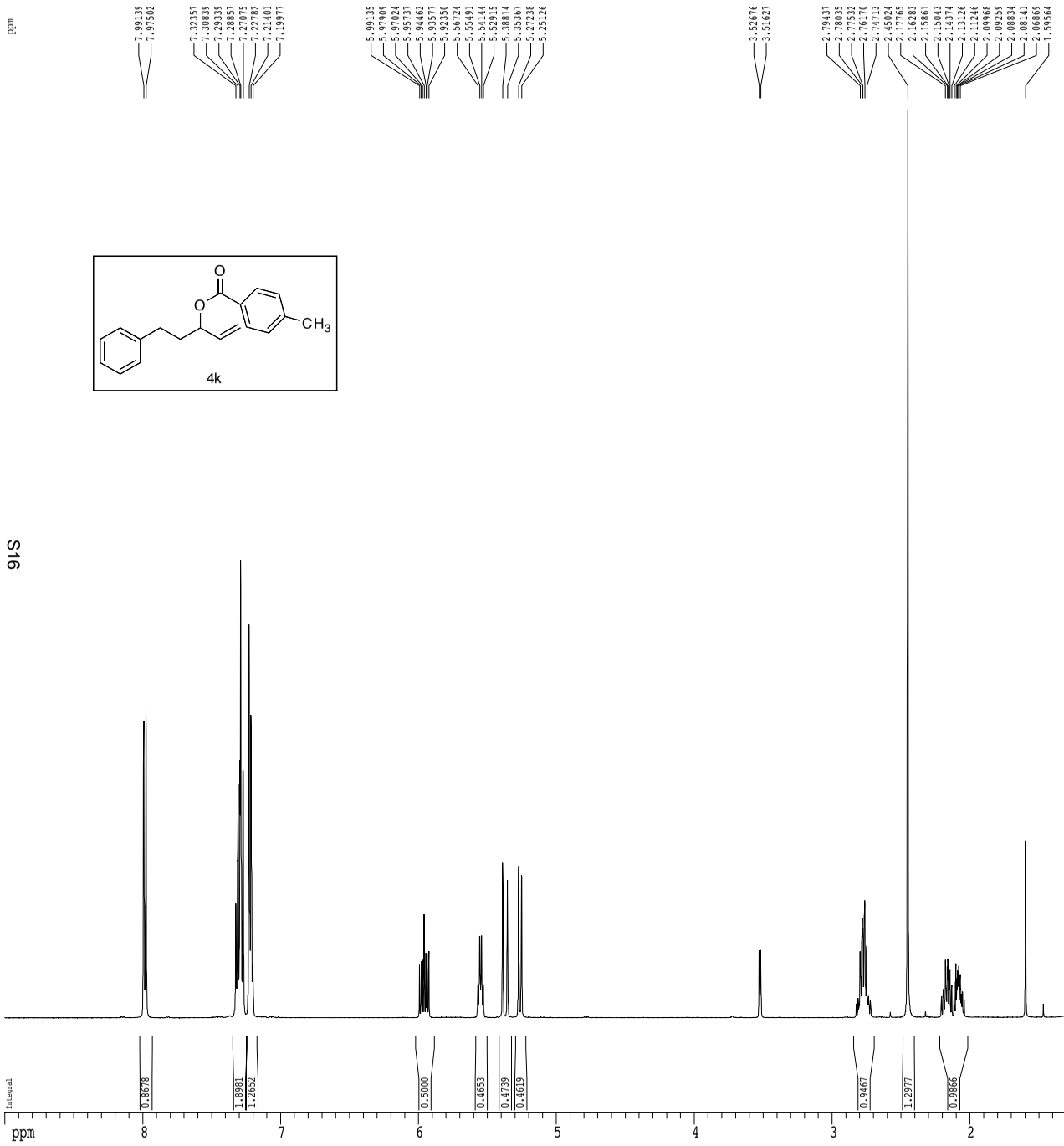
1H spectrum



S16

Current Data Parameters
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NAME JSC1092-1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
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Time 15.41
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PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 6.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200166 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -1.042 ppm
F2 -521.42 Hz
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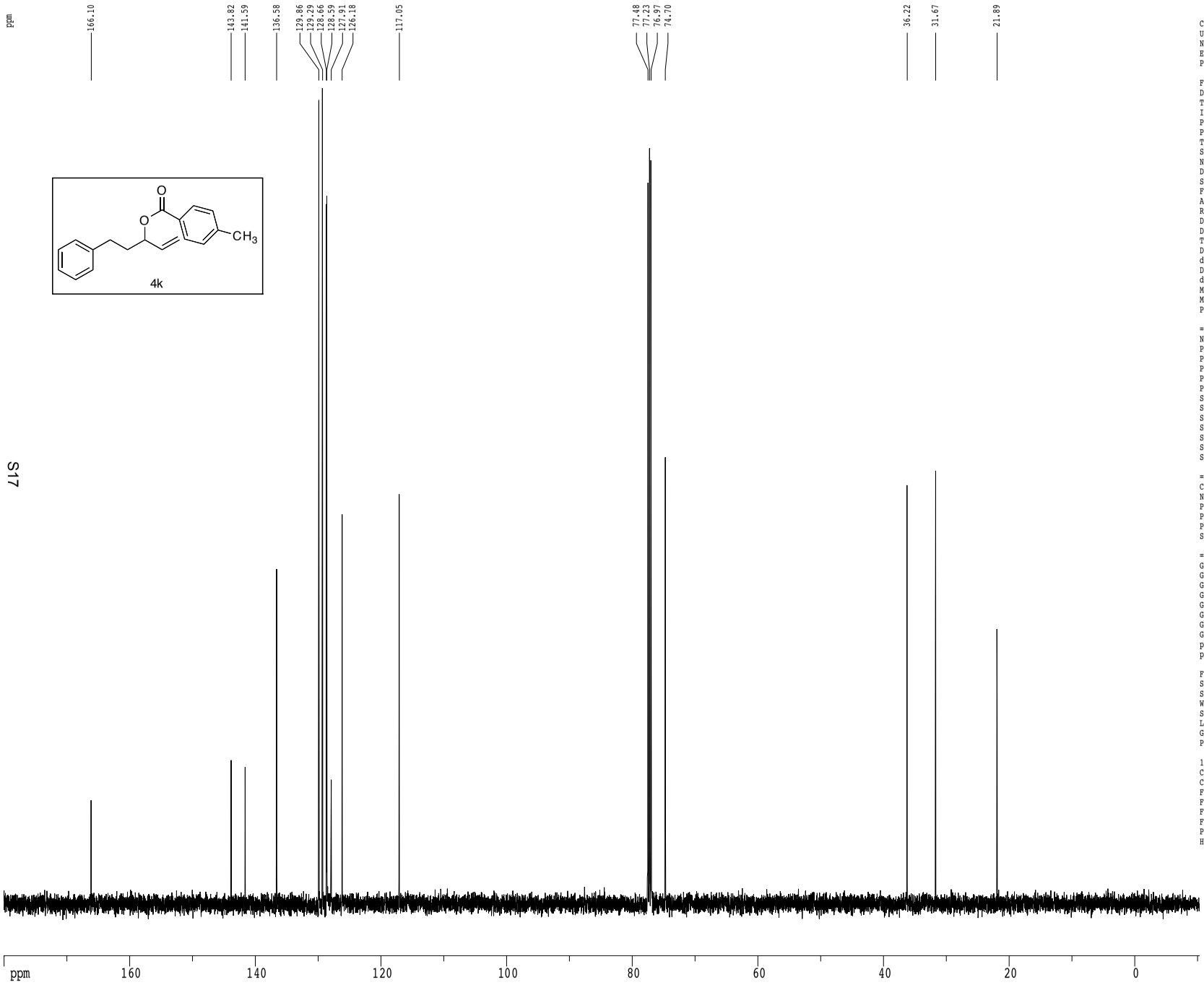
HZCM 220.32463 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling



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EXPNO 3
PROCNO 1

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PULPROG SpinEchopg30gp.prd
TD 65536
SOLVENT CDCl3
NS 200
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 10321.3
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 29.70 usec

======== CHANNEL f1 ========
NUC1 13C
P1 14.85 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.60 dB
SP2 3.60 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
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SPOFF2 0.00 Hz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

====== GRADIENT CHANNEL =====
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GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

F2 - Processing parameters
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SF 125.7803997 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
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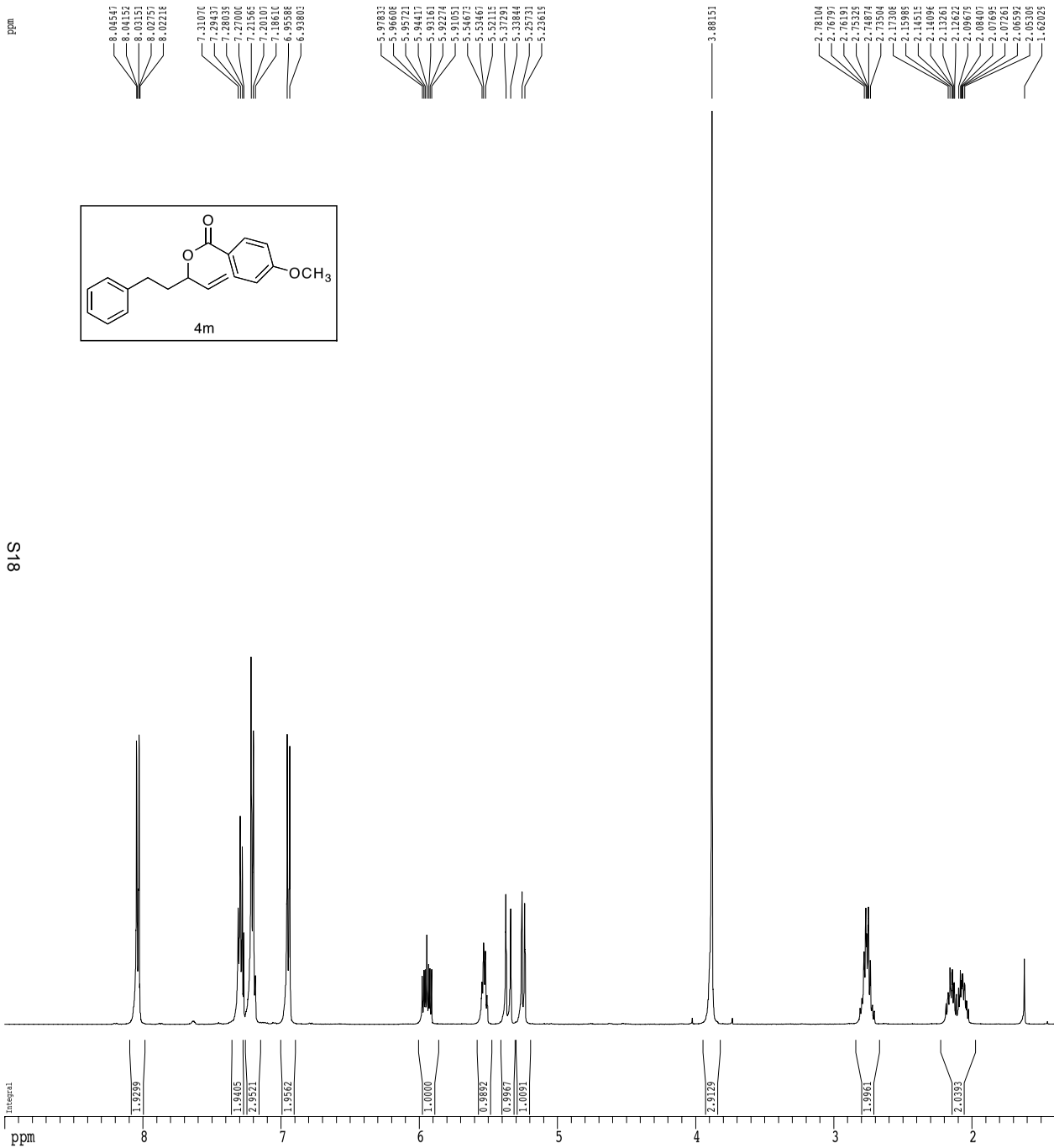
1H spectrum



Current Data Parameters
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PROCNO 1

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PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 14.3
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.38 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
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SF 500.2200254 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -1.060 ppm
F2 -530.22 Hz
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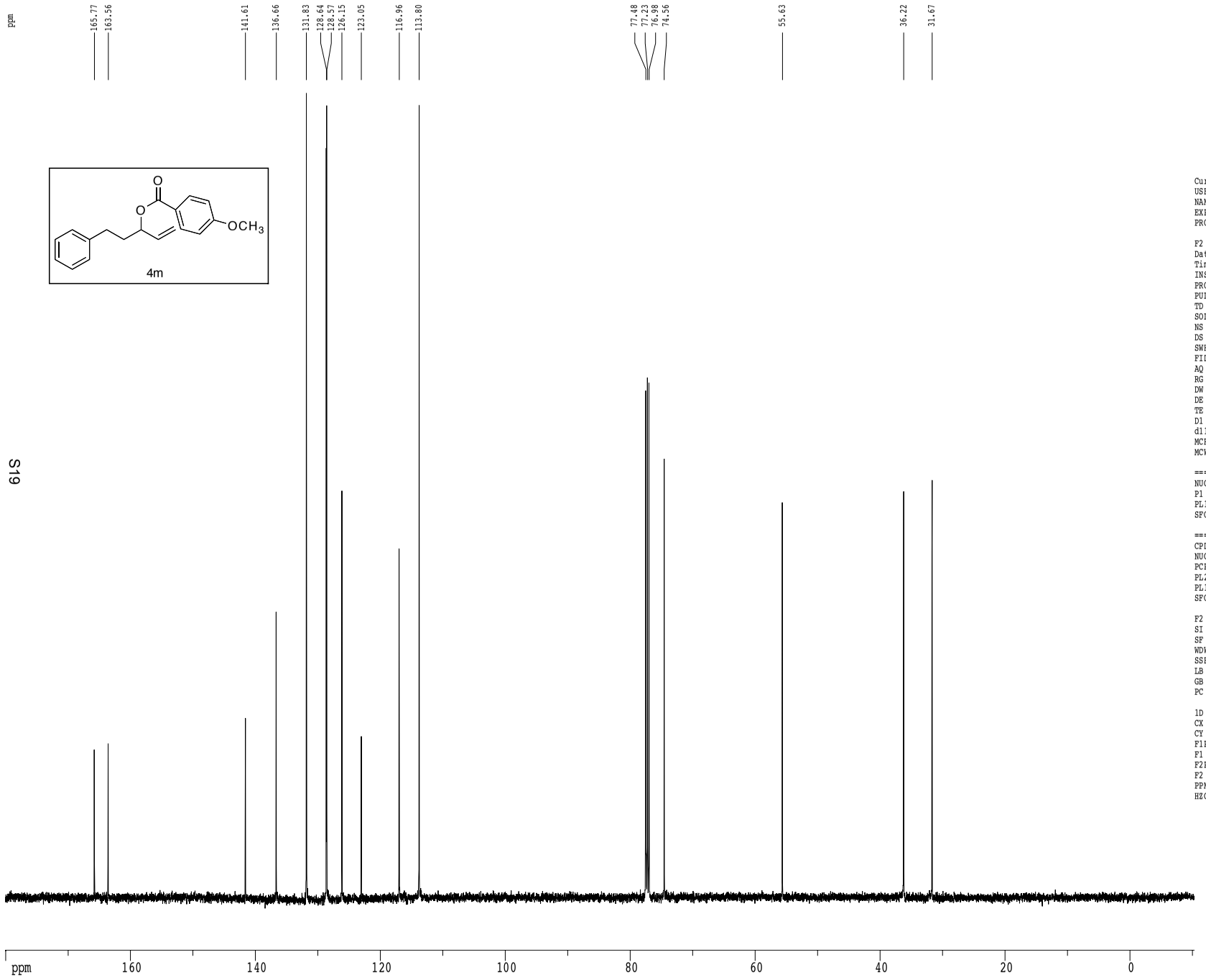
13C spectrum with 1H decoupling



Current Data Parameters

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NAME	JSC1100
EXPNO	5
PROCNO	1

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PULPROG	zgdc30
TD	65418
SOLVENT	CDCl3
NS	160
DS	4
SWH	30303.031 Hz
FIDRES	0.463222 Hz
AQ	1.0794470 sec
RG	9195.2
DW	16.500 usec
DE	6.00 usec
TE	298.0 K
D1	0.25000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	14.75 usec
PL1	-1.00 dB
SFO1	125.7942548 MHz

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

CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	1.60 dB
PL12	24.80 dB
SFO2	500.2225011 MHz

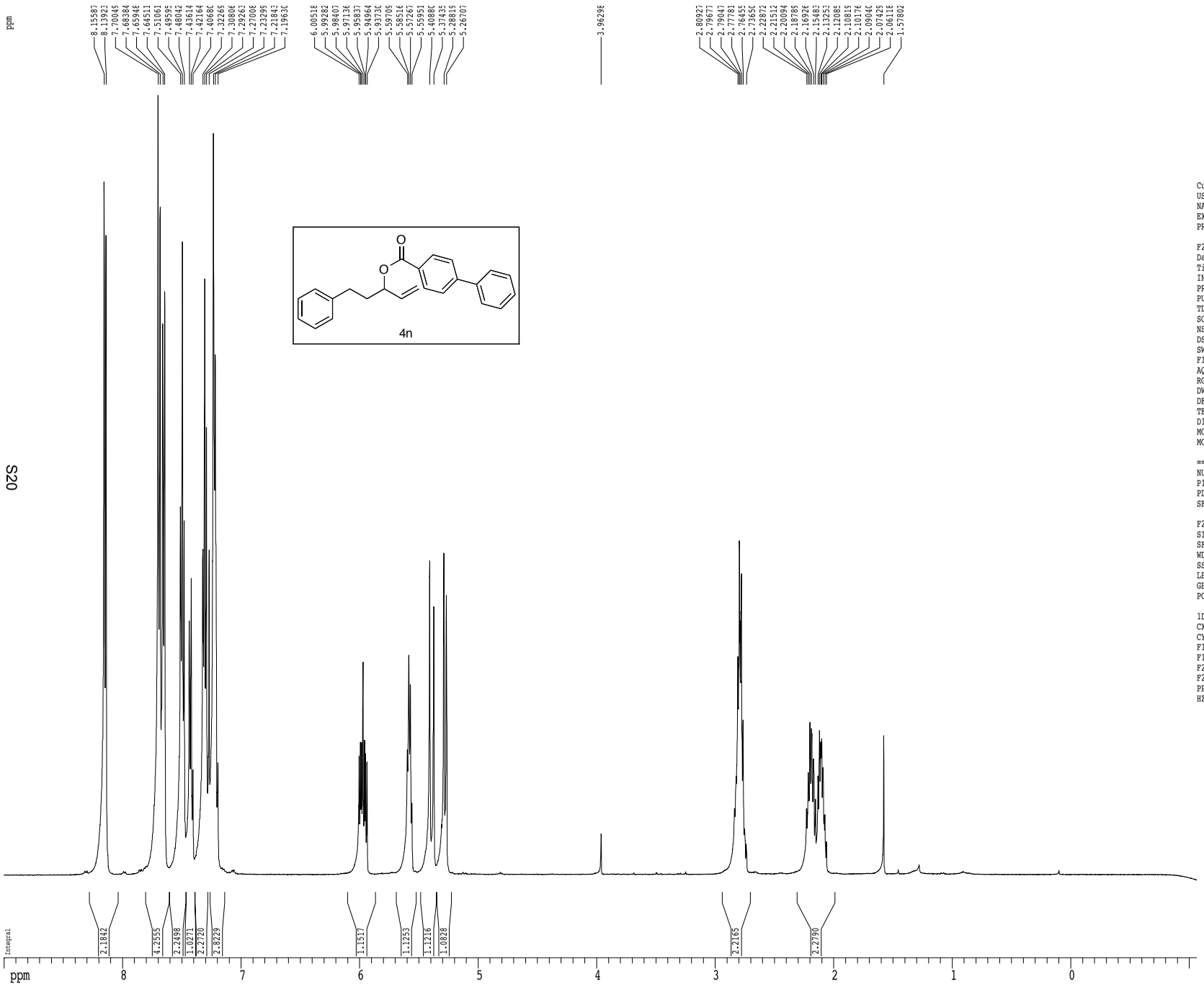
F2 - Processing parameters

SI	65536
SF	125.7804029 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	2.00

1D NMR plot parameters

CX	22.80 cm
CY	15.65 cm
F1P	180.000 ppm
F1	22640.47 Hz
F2P	-10.332 ppm
F2	-1299.59 Hz
PPMCM	8.34791 ppm/cm
HZCM	1050.00305 Hz/cm

1H spectrum



S20

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Current Data Parameters
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EXPNO                 1
PROCNO                1

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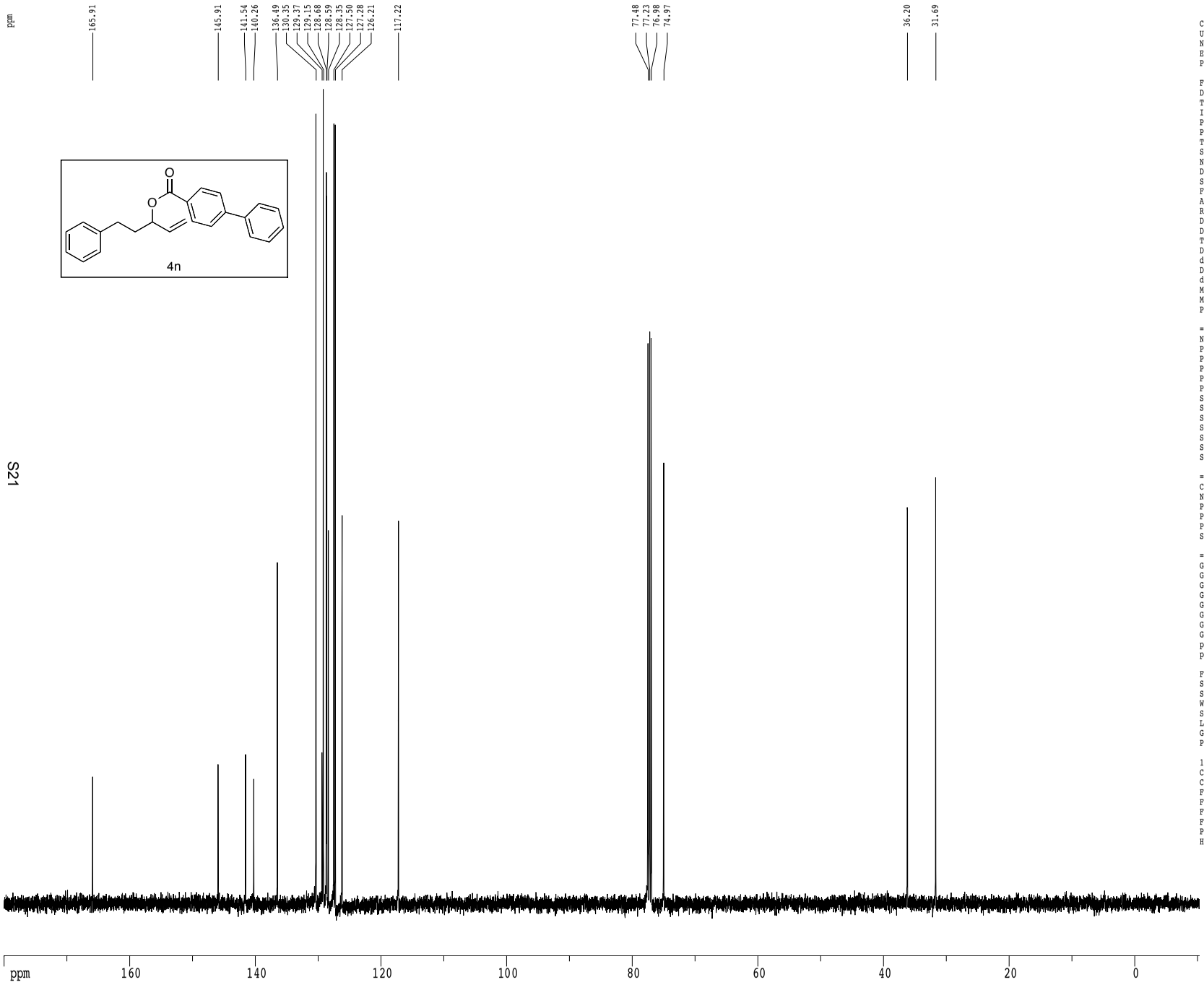
PULPROG            zg30
TD                81728
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ             5.0998774 sec
RG                    5
DW               62.400 usec
DE                6.00 usec
TE                298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.50 usec
PL1                1.60 dB
SFO1        500.2235015 MHz

F2 - Processing parameters
SI                 65536
SF          500.2200265 MHz
WDW                   EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 4.00

1D NMR plot parameters
CX                22.80 cm
CY                15.00 cm
F1P               9.000 ppm
F1              4501.98 Hz
F2P              -1.062 ppm
F2              -531.32 Hz
PPMCM          0.44132 ppm/cm
HZCM          220.75900 Hz/cm
```

Z-restored spin-echo 13C spectrum with 1H decoupling



S21

Current Data Parameters

USER	cannon
NAME	JSC1094-2
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20081013
Time	11.11
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	SpinEchopg30qp.prd
TD	65536
SOLVENT	CDCl3
NS	232
DS	16
SWH	30303.031 Hz
FIDRES	0.462388 Hz
AQ	1.0813940 sec
RG	7298.2
DW	16.500 usec
DE	6.00 usec
TE	298.0 K
D1	0.25000000 sec
d11	0.03000000 sec
D16	0.00020000 sec
d17	0.00019600 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec
P2	29.70 usec

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

NUC1	13C
P1	14.85 usec
P11	500.00 usec
P12	2000.00 usec
PL0	120.00 dB
PL1	-1.00 dB
SFO1	125.7942548 MHz
SP1	3.60 dB
SP2	3.60 dB
SPNAM1	Crp60,0.5,20.1
SPNAM2	Crp60comp.4
SPOFF1	0.00 Hz
SPOFF2	0.00 Hz

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

CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	1.60 dB
PL12	24.60 dB
SFO2	500.2225011 MHz

====== GRADIENT CHANNEL =====

GPNAM1	SINE.100
GPNAM2	SINE.100
GPX1	0.00 %
GPX2	0.00 %
GPY1	0.00 %
GPY2	0.00 %
GPZ1	30.00 %
GPZ2	50.00 %
p15	500.00 usec
p16	1000.00 usec

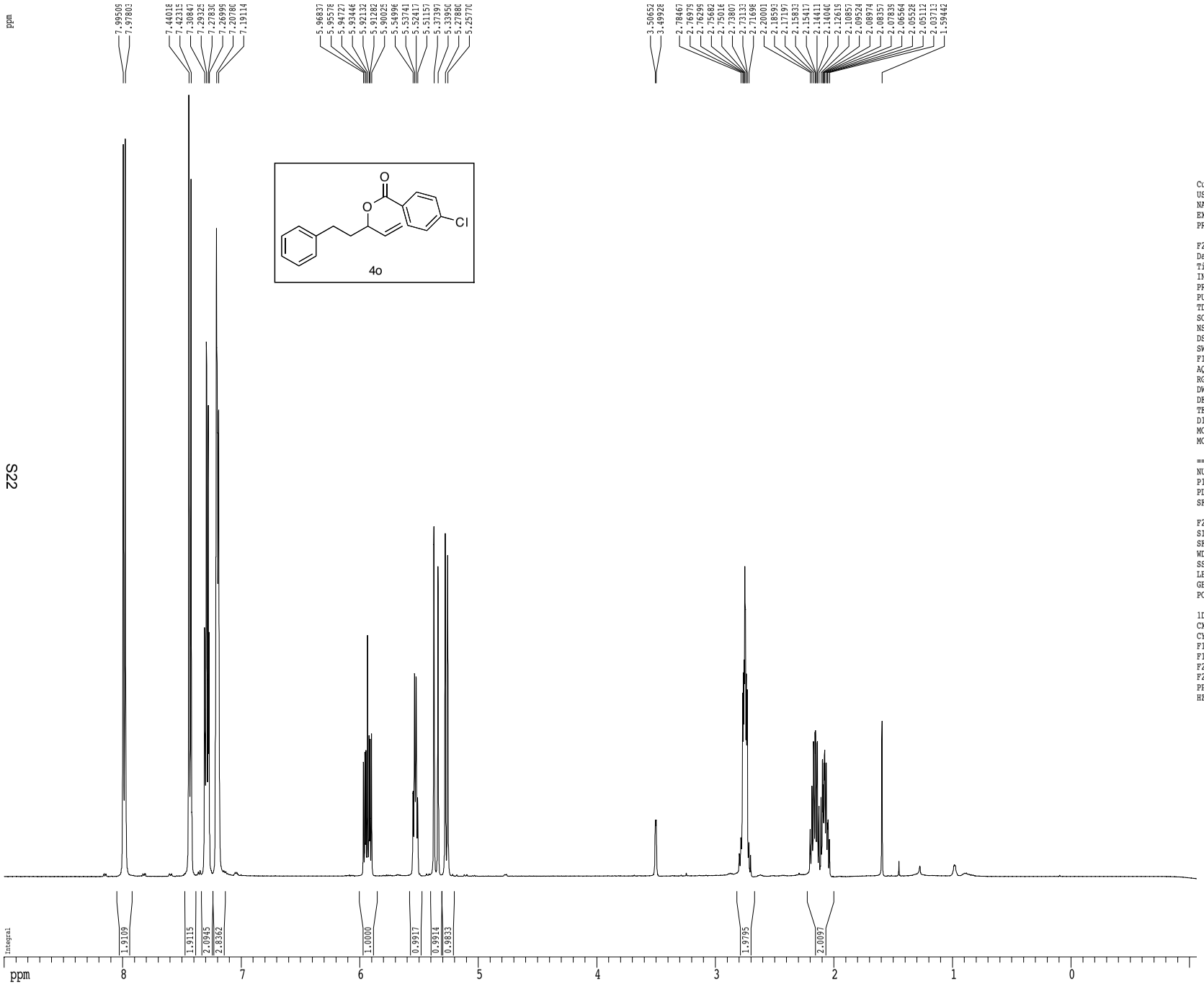
F2 - Processing parameters

SI	65536
SF	125.7804020 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	2.00

1D NMR plot parameters

CX	22.80 cm
CY	15.65 cm
F1P	180.000 ppm
F1	22640.47 Hz
F2P	-10.325 ppm
F2	-1298.67 Hz
PPMCM	8.34758 ppm/cm
HZCM	1049.96240 Hz/cm

1H spectrum



Current Data Parameters

USER	cannon
NAME	JSC1097-1
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20081008
Time	15.49
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	81728
SOLVENT	CDCl3
NS	8
DS	2
SWH	8012.820 Hz
FIDRES	0.098043 Hz
AQ	5.0998774 sec
RG	8
DW	62.400 usec
DE	6.00 usec
TE	298.0 K
D1	0.10000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	1H
P1	7.50 usec
PL1	1.60 dB
SFO1	500.2235015 MHz

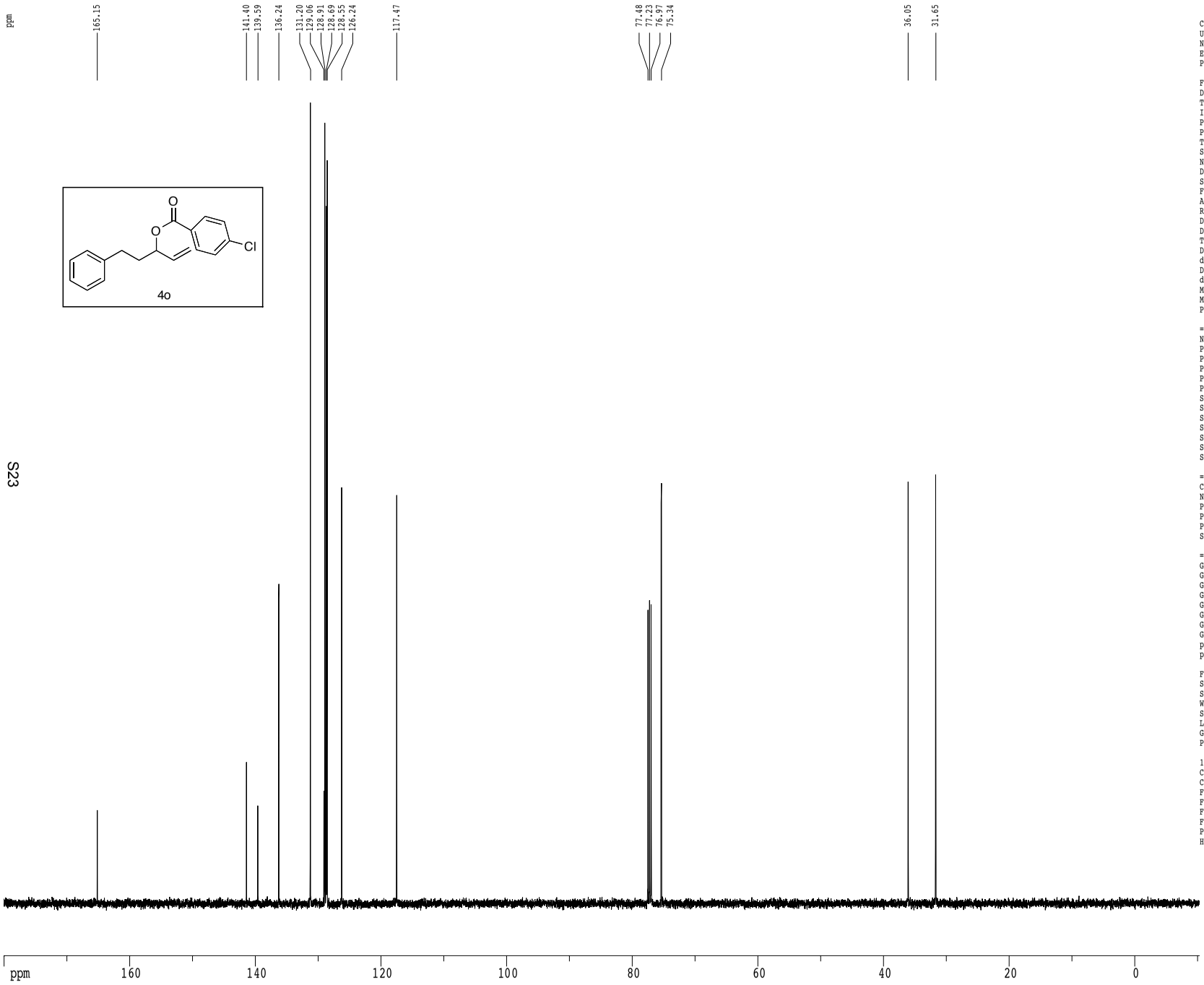
F2 - Processing parameters

SI	65536
SF	500.2200257 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	22.80 cm
CY	15.00 cm
F1P	9.000 ppm
F1	4501.98 Hz
F2P	-1.061 ppm
F2	-530.60 Hz
PPMCM	0.44126 ppm/cm
HZCM	220.72697 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling




```
Current Data Parameters
USER              cannon
NAME          JSC1097-1
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20081008
Time              15.50
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30qp.prd
TD                65536
SOLVENT            CDCl3
NS                  129
DS                   16
SWH           30303.031 Hz
FIDRES        0.462388 Hz
AQ            1.0813940 sec
RG               2896.3
DW               16.500 usec
DE                6.00 usec
TE                298.0 K
D1            0.25000000 sec
d11           0.03000000 sec
D16           0.00020000 sec
d17           0.00019600 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
P2                29.70 usec

======== CHANNEL f1 ========
NUC1                13C
P1                14.85 usec
P11              500.00 usec
P12             2000.00 usec
PL0              120.00 dB
PL1               -1.00 dB
SFO1        125.7942548 MHz
SP1               3.60 dB
SP2               3.60 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1               0.00 Hz
SPOFF2               0.00 Hz

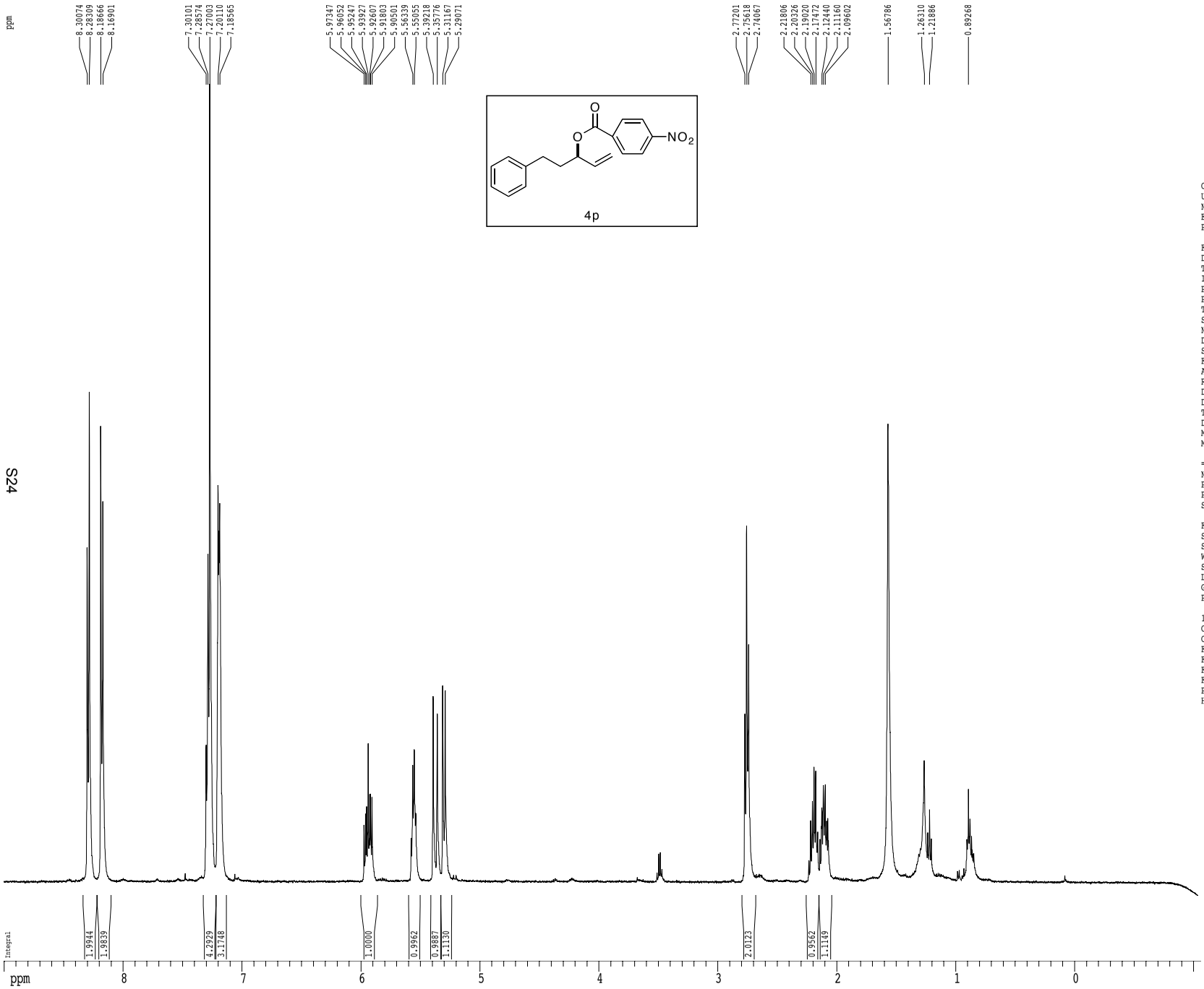
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2            100.00 usec
PL2                1.60 dB
PL12              24.60 dB
SFO2        500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1          SINE.100
GPNAM2          SINE.100
GPX1                0.00 %
GPX2                0.00 %
GPY1                0.00 %
GPY2                0.00 %
GPZ1               30.00 %
GPZ2               50.00 %
p15              500.00 usec
p16             1000.00 usec

F2 - Processing parameters
SI                  65536
SF          125.7804015 MHz
WDW                    EM
SSB                     0
LB                1.00 Hz
GB                      0
PC                2.00

1D NMR plot parameters
CX                22.80 cm
CY                15.65 cm
F1P              180.000 ppm
F1             22640.47 Hz
F2P              -10.321 ppm
F2             -1298.20 Hz
PPMCM             8.34742 ppm/cm
HZCM           1049.94214 Hz/cm
```

1H spectrum



S24

Current Data Parameters
USER cannon
NAME JSC2279-3
EXPNO 1
PROCNO 1

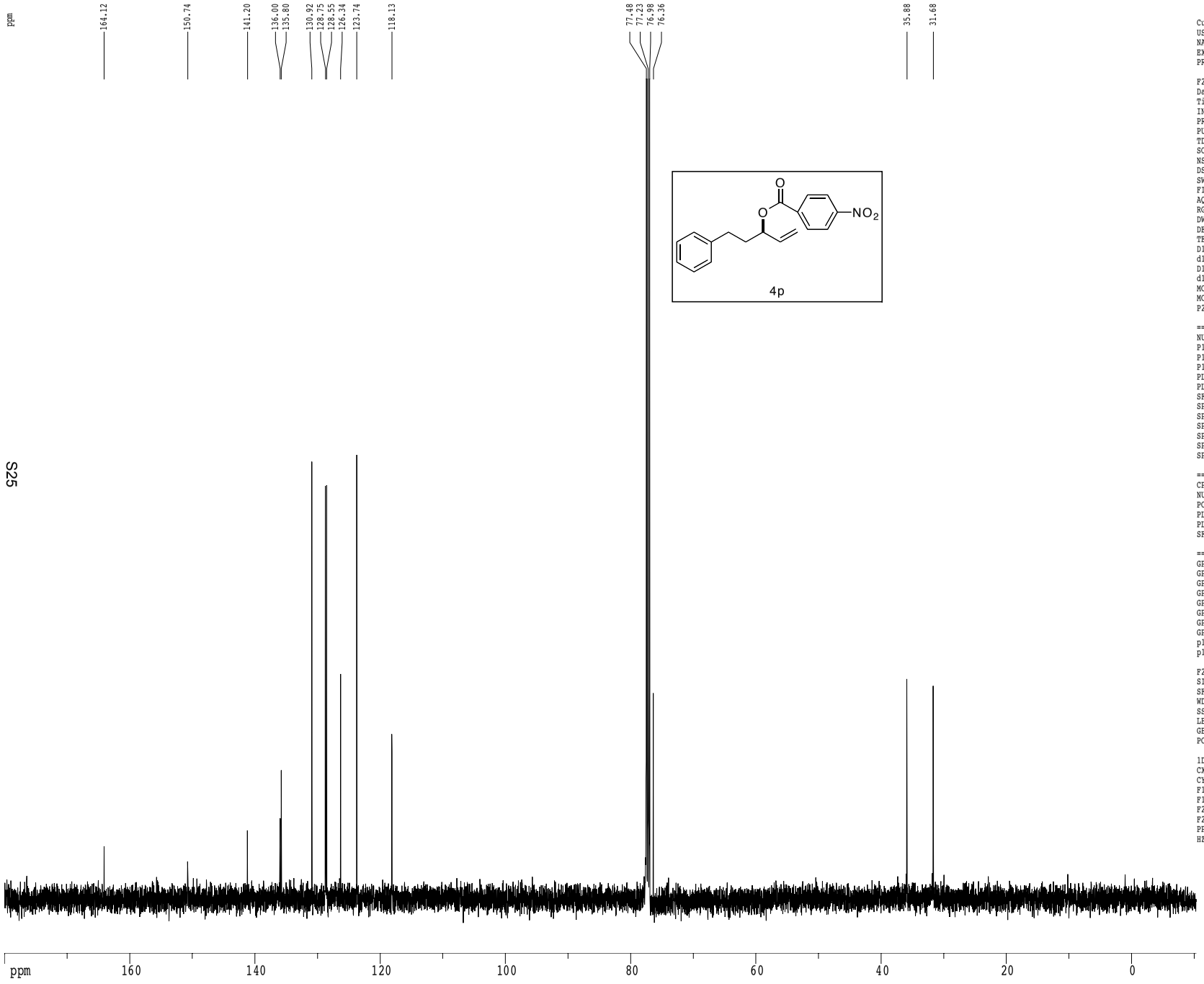
F2 - Acquisition Parameters
Date_ 20100428
Time 15.34
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0999398 sec
RG 8
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200267 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 30.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -1.063 ppm
F2 -531.57 Hz
PPMCM 0.44135 ppm/cm
HZCM 220.76974 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling




```
Current Data Parameters
USER              cannon
NAME          JSC2279-3
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20100428
Time              15.37
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG  SpinEchopg30gp.prd
TD                65536
SOLVENT           CDCl3
NS                  504
DS                   16
SWH           30303.031 Hz
FIDRES        0.462388 Hz
AQ            1.0813940 sec
RG                 4096
DW               16.500 usec
DE                6.00 usec
TE               298.0 K
D1            0.25000000 sec
d11           0.03000000 sec
D16           0.00020000 sec
d17           0.00019600 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec
P2               31.00 usec

======== CHANNEL f1 ========
NUC1                13C
P1               15.50 usec
P11             500.00 usec
P12            2000.00 usec
PL0             120.00 dB
PL1              -1.00 dB
SFO1        125.7942548 MHz
SP1              3.20 dB
SP2              3.20 dB
SPNAM1   Crp60,0.5,20.1
SPNAM2      Crp60comp.4
SPOFF1             0.00 Hz
SPOFF2             0.00 Hz

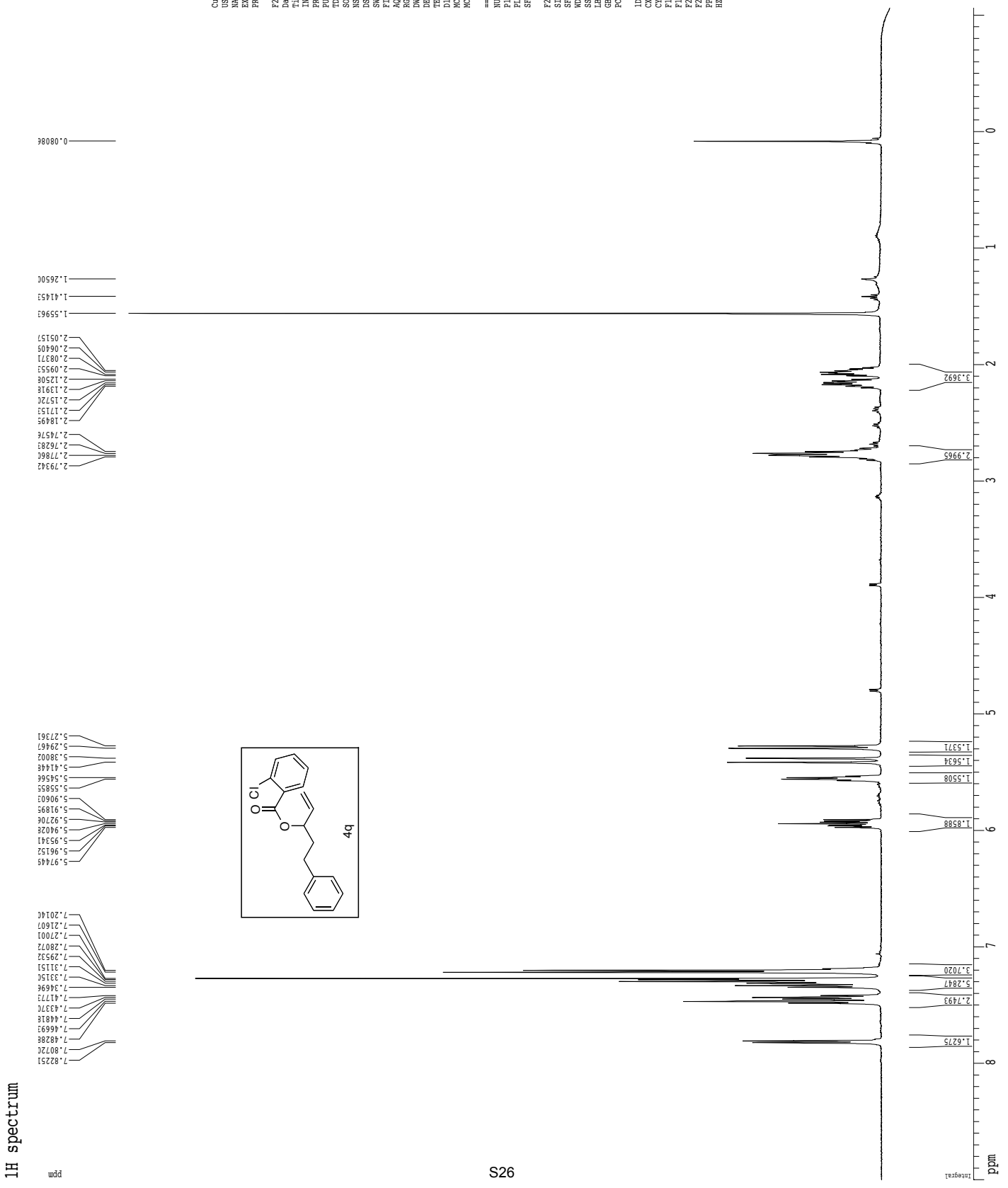
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2           100.00 usec
PL2               1.60 dB
PL12             24.60 dB
SFO2        500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1         SINE.100
GPNAM2         SINE.100
GPX1               0.00 %
GPX2               0.00 %
GPY1               0.00 %
GPY2               0.00 %
GPZ1              30.00 %
GPZ2              50.00 %
p15              500.00 usec
p16             1000.00 usec

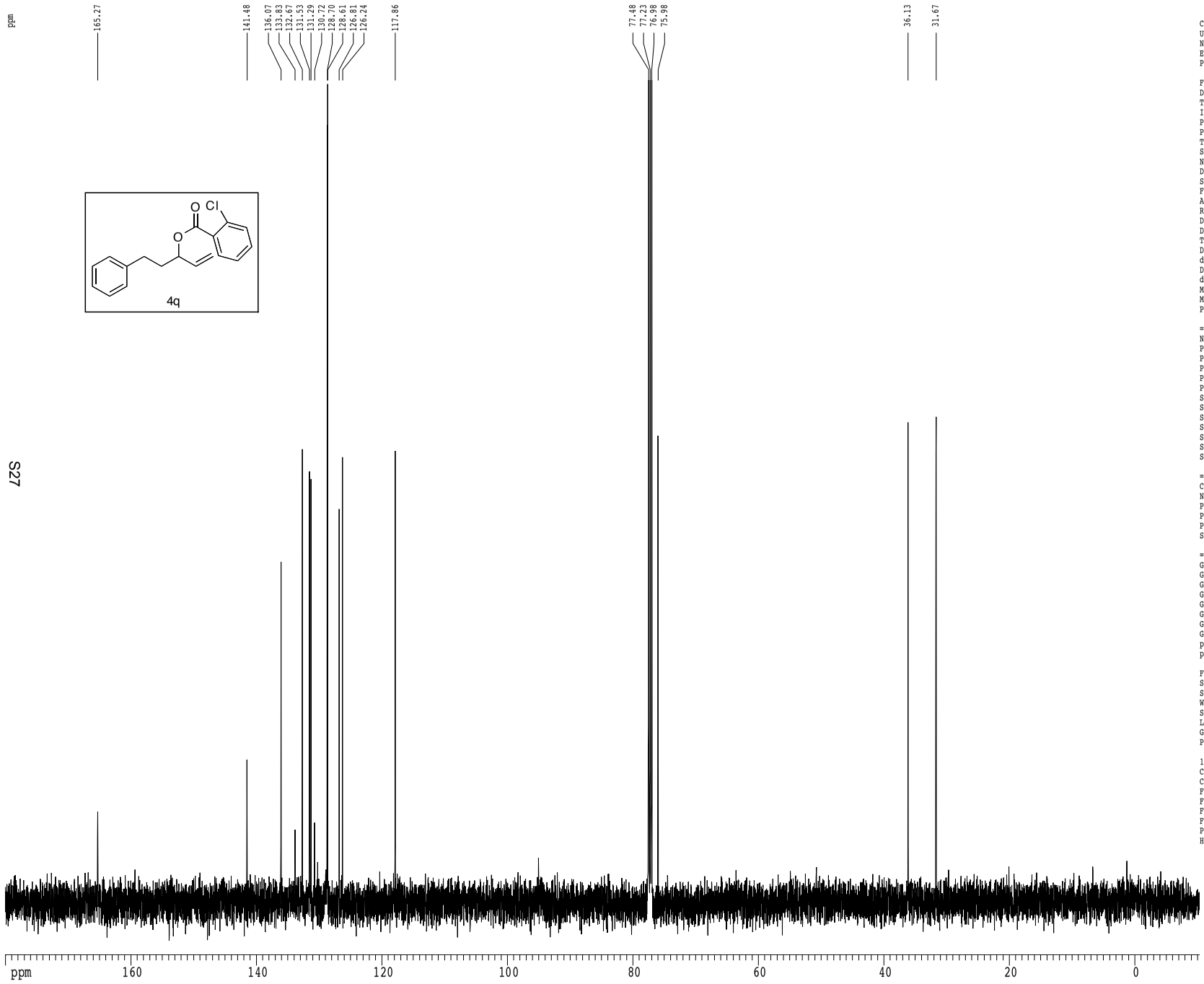
F2 - Processing parameters
SI                65536
SF          125.7803988 MHz
WDW                  EM
SSB                   0
LB                1.00 Hz
GB                    0
PC                1.00

1D NMR plot parameters
CX               22.80 cm
CY               31.00 cm
F1P             180.000 ppm
F1            22640.47 Hz
F2P             -10.460 ppm
F2            -1315.67 Hz
PPMCM          8.35351 ppm/cm
HZCM        1050.70789 Hz/cm
```

1H spectrum


Current Data Parameters
USER cannon
NAME JSC1104-2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081020
Time 15.30
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 7.50 usec
PL1 1.60 dB
SFO1 500.2235015 MHz
F2 - Processing parameters
SI 65536
SF 500.2200263 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00
1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -1.062 ppm
F2 -531.21 Hz
PPMCM 0.44131 ppm/cm
HZCM 220.75375 Hz/cm
0.08086
1.26500
1.41453
1.55963
2.05157
2.06405
2.08371
2.09553
2.12506
2.13918
2.15720
2.17153
2.18495
2.74576
2.76283
2.77860
2.79342
5.27361
5.29467
5.38002
5.41448
5.54566
5.55855
5.90603
5.91895
5.92706
5.94028
5.95341
5.96152
5.97449
7.20140
7.21607
7.27001
7.28072
7.29532
7.31151
7.33150
7.34696
7.41773
7.43370
7.44818
7.46693
7.48288
7.80720
7.82251
3.3692
2.9965
1.5371
1.5634
1.5508
1.8588
3.7020
5.2847
2.7493
1.6275
4q
O
O
Cl
ppm
Integral

Z-restored spin-echo 13C spectrum with 1H decoupling



Current Data Parameters
USER cannon
NAME JSC1104-2
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081020
Time 15.32
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30qp.prd
TD 65536
SOLVENT CDCl3
NS 660
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 7298.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 29.70 usec

======== CHANNEL f1 ========
NUC1 13C
P1 14.85 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.60 dB
SP2 3.60 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

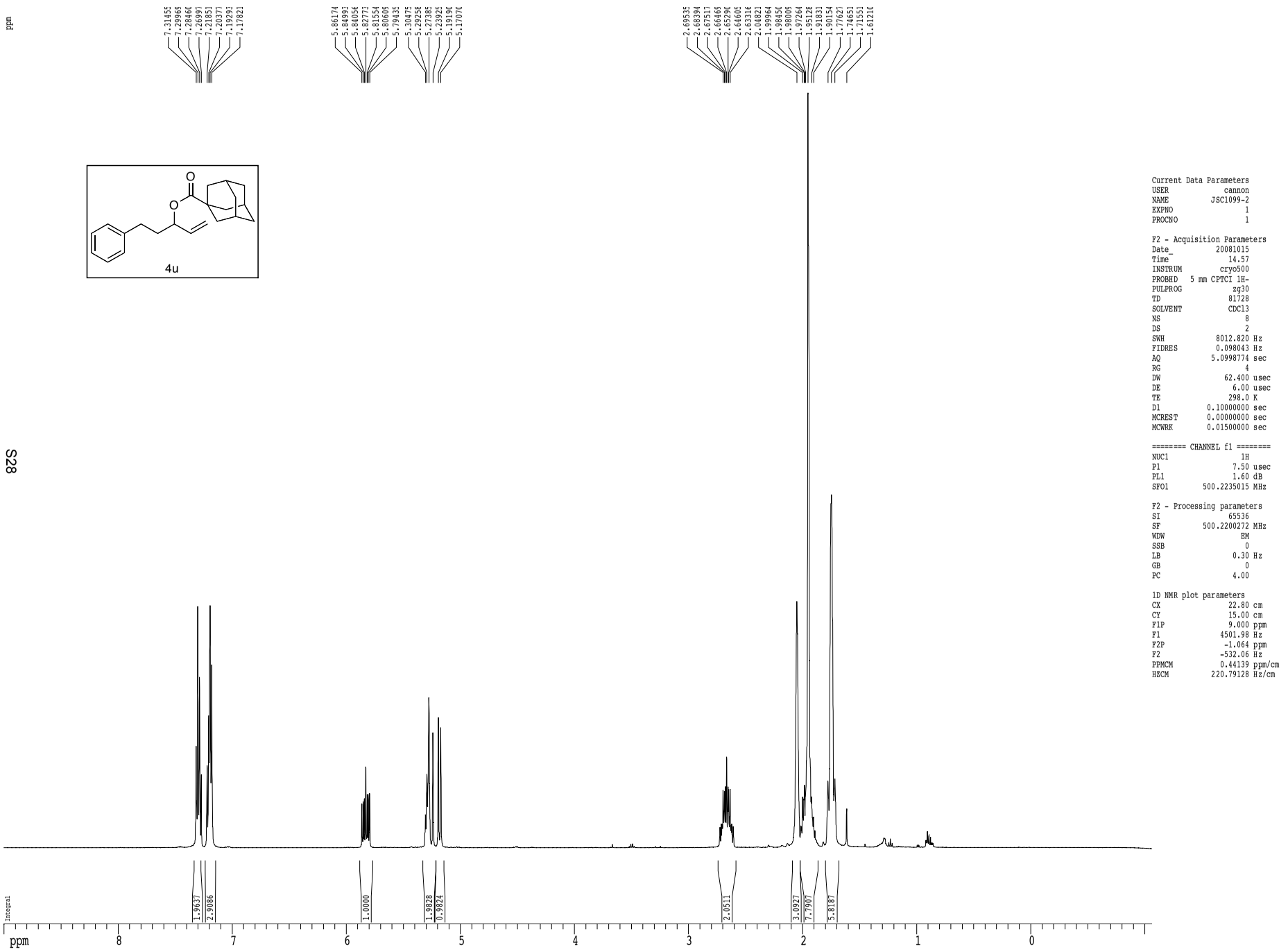
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 125.7803988 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.299 ppm
F2 -1295.43 Hz
PPMCM 8.34645 ppm/cm
HZCM 1049.82019 Hz/cm

1H spectrum



Current Data Parameters

USER	cannon
NAME	JSC1099-2
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20081015
Time	14.57
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	81728
SOLVENT	CDCl3
NS	8
DS	2
SWH	8012.820 Hz
FIDRES	0.098043 Hz
AQ	5.0998774 sec
RG	4
DW	62.400 usec
DE	6.00 usec
TE	298.0 K
D1	0.10000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	1H
P1	7.50 usec
PL1	1.60 dB
SFO1	500.2235015 MHz

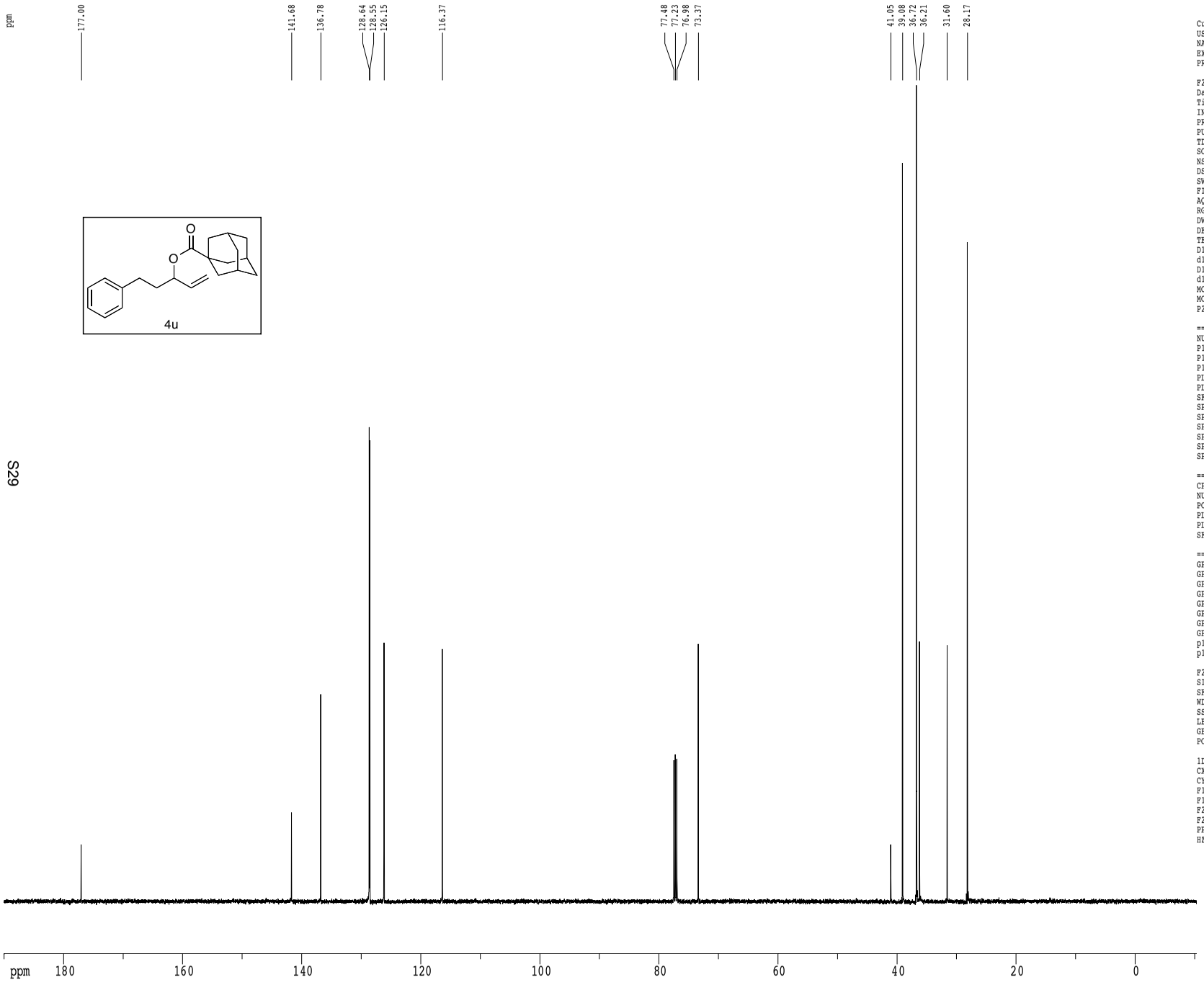
F2 - Processing parameters

SI	65536
SF	500.2200272 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	22.80 cm
CY	15.00 cm
F1P	9.000 ppm
F1	4501.98 Hz
F2P	-1.064 ppm
F2	-532.06 Hz
PPMCM	0.44139 ppm/cm
HZCM	220.79128 Hz/cm

Z-restored spin-echo 13C spectrum with 1H decoupling



S29

Current Data Parameters
USER cannon
NAME JSC1099-2
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081015
Time 14.58
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG SpinEchopg30qp.prd
TD 65536
SOLVENT CDCl3
NS 160
DS 16
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 7298.2
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
d17 0.00019600 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
P2 29.70 usec

======== CHANNEL f1 ========
NUC1 13C
P1 14.85 usec
P11 500.00 usec
P12 2000.00 usec
PL0 120.00 dB
PL1 -1.00 dB
SFO1 125.7942548 MHz
SP1 3.60 dB
SP2 3.60 dB
SPNAM1 Crp60,0.5,20.1
SPNAM2 Crp60comp.4
SPOFF1 0.00 Hz
SPOFF2 0.00 Hz

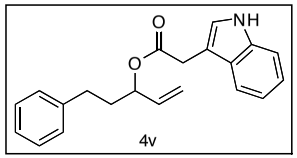
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.60 dB
SFO2 500.2225011 MHz

====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPX1 0.00 %
GPX2 0.00 %
GPY1 0.00 %
GPY2 0.00 %
GPZ1 30.00 %
GPZ2 50.00 %
p15 500.00 usec
p16 1000.00 usec

F2 - Processing parameters
SI 65536
SF 125.7804029 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 190.000 ppm
F1 23898.28 Hz
F2P -10.332 ppm
F2 -1299.59 Hz
PPMCM 8.78650 ppm/cm
HZCM 1105.16968 Hz/cm

1H spectrum



4v

S30

Current Data Parameters
USER cannon
NAME JSC1058
EXPNO 5
PROCNO 1

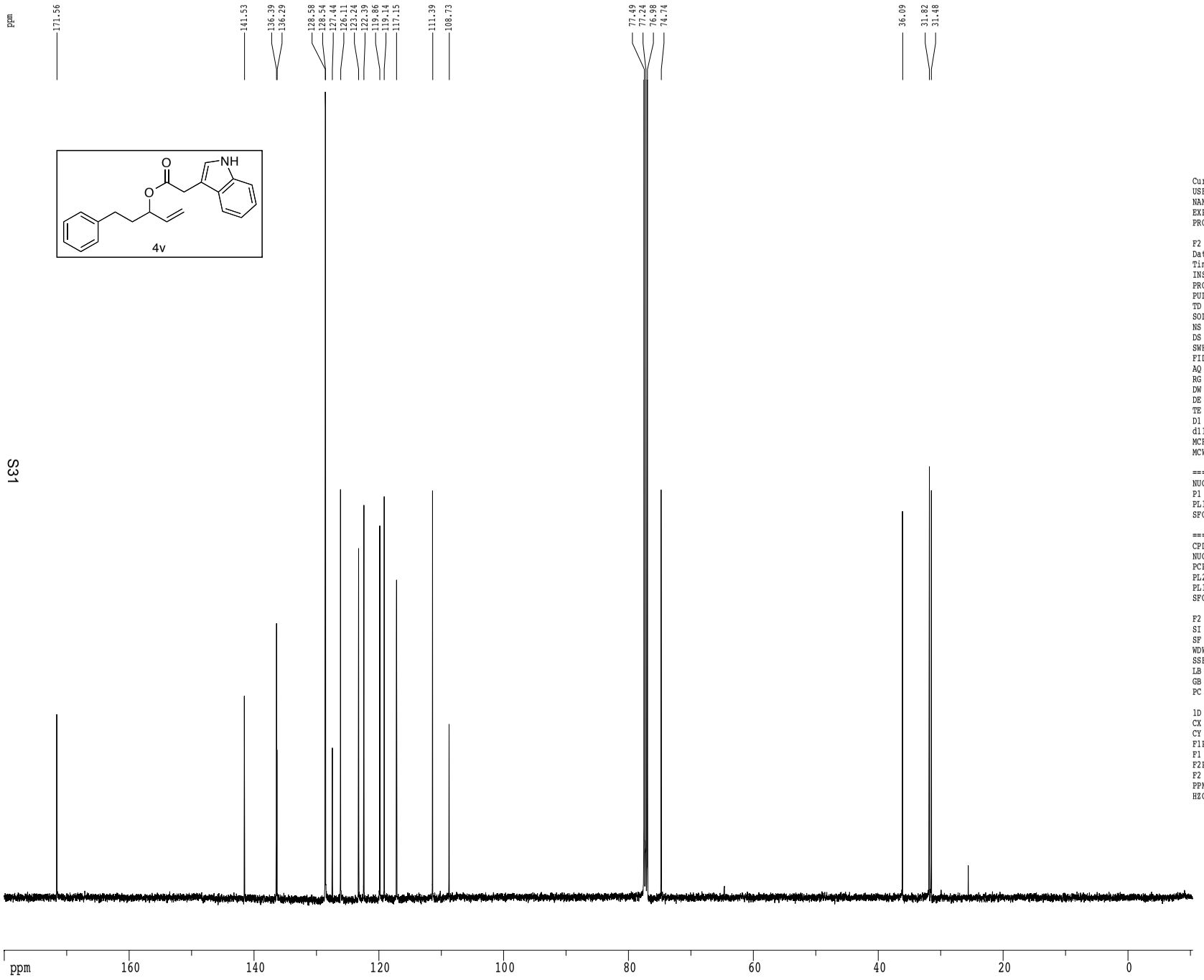
F2 - Acquisition Parameters
Date_ 20081003
Time 15.41
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 16
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.38 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200153 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -1.040 ppm
F2 -520.20 Hz
PPMCM 0.44035 ppm/cm
HZCM 220.27109 Hz/cm

13C spectrum with 1H decoupling



Current Data Parameters
USER cannon
NAME JSC1058
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081003
Time 15.36
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zgdc30
TD 65418
SOLVENT CDCl3
NS 160
DS 4
SWH 30303.031 Hz
FIDRES 0.463222 Hz
AQ 1.0794470 sec
RG 8192
DW 16.500 usec
DE 6.00 usec
TE 298.0 K
D1 0.25000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

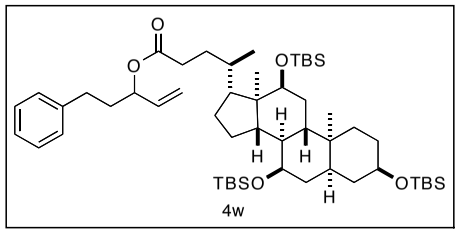
======== CHANNEL f1 ========
NUC1 13C
P1 14.75 usec
PL1 -1.00 dB
SFO1 125.7942548 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 1.60 dB
PL12 24.80 dB
SFO2 500.2225011 MHz

F2 - Processing parameters
SI 65536
SF 125.7804043 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 180.000 ppm
F1 22640.47 Hz
F2P -10.343 ppm
F2 -1300.99 Hz
PPMCM 8.34839 ppm/cm
HZCM 1050.06409 Hz/cm

1H spectrum



4w


```
Current Data Parameters
USER              cannon
NAME          JSC1133-2
EXPNO                 1
PROCNO                1

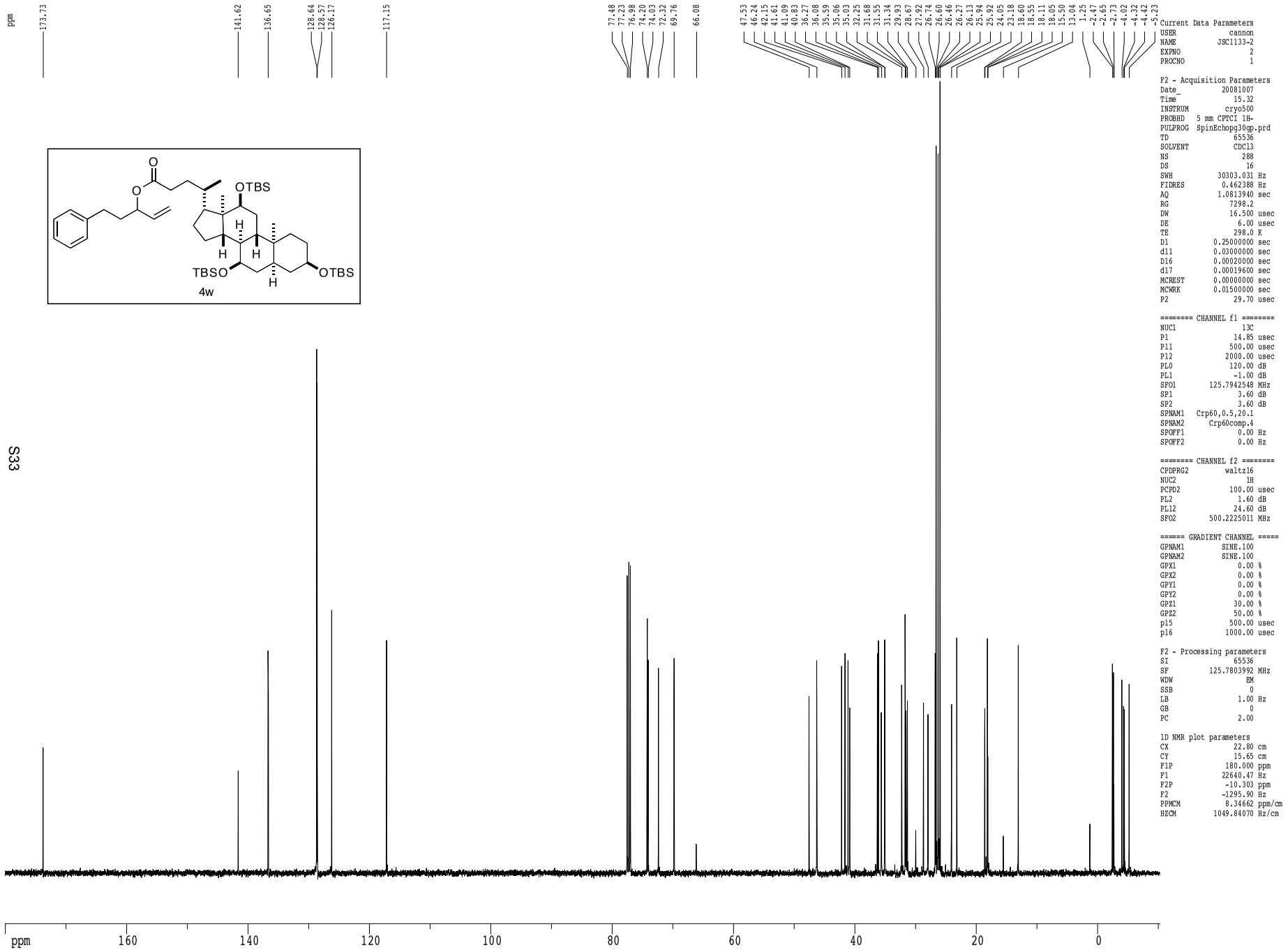
F2 - Acquisition Parameters
Date_          20081007
Time              15.31
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG             zg30
TD                81728
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ             5.0998774 sec
RG                  3.2
DW                62.400 usec
DE                 6.00 usec
TE                298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                7.50 usec
PL1               1.60 dB
SFO1        500.2235015 MHz

F2 - Processing parameters
SI                65536
SF          500.2200258 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                4.00

1D NMR plot parameters
CX               22.80 cm
CY               15.00 cm
F1P               9.000 ppm
F1             4501.98 Hz
F2P              -1.061 ppm
F2             -530.71 Hz
PPMCM          0.44127 ppm/cm
HZCM         220.73225 Hz/cm
```

Z-restored spin-echo 13C spectrum with 1H decoupling



Current Data Parameters

USER	cannon
NAME	JSC1133-2
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20081007
Time	15.32
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	SpinEchopg30qp.prd
TD	65536
SOLVENT	CDCl3
NS	288
DS	16
SWH	30303.031 Hz
FIDRES	0.462388 Hz
AQ	1.0813940 sec
RG	7298.2
DW	16.500 usec
DE	6.00 usec
TE	298.0 K
D1	0.25000000 sec
d11	0.03000000 sec
D16	0.00020000 sec
d17	0.00019600 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec
P2	29.70 usec

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

NUC1	13C
P1	14.85 usec
P11	500.00 usec
P12	2000.00 usec
PL0	120.00 dB
PL1	-1.00 dB
SFO1	125.7942548 MHz
SP1	3.60 dB
SP2	3.60 dB
SPNAM1	Crp60,0.5,20.1
SPNAM2	Crp60comp.4
SPOFF1	0.00 Hz
SPOFF2	0.00 Hz

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

CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	1.60 dB
PL12	24.60 dB
SFO2	500.2225011 MHz

====== GRADIENT CHANNEL =====

GPNAM1	SINE.100
GPNAM2	SINE.100
GPX1	0.00 %
GPX2	0.00 %
GPY1	0.00 %
GPY2	0.00 %
GPZ1	30.00 %
GPZ2	50.00 %
p15	500.00 usec
p16	1000.00 usec

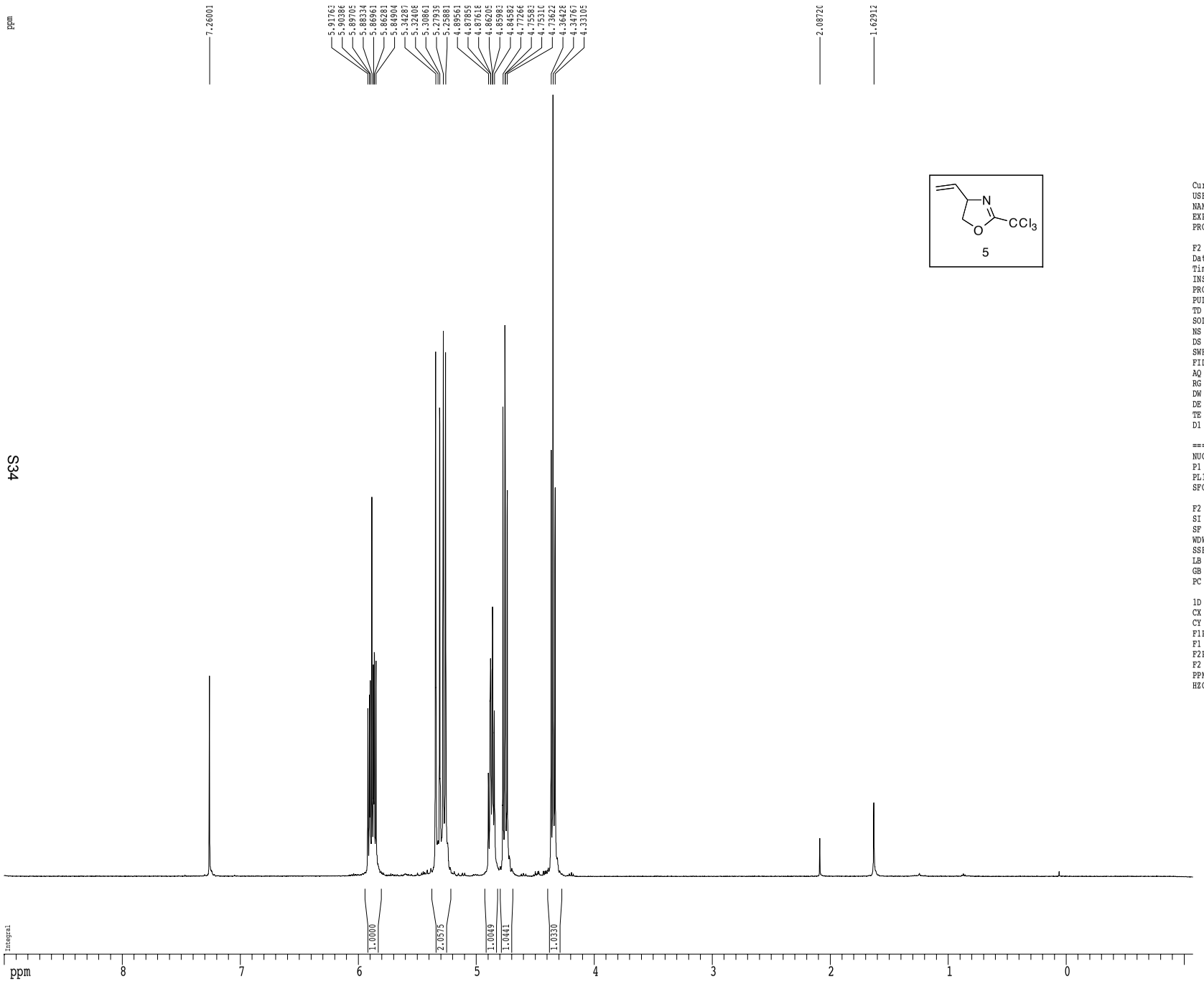
F2 - Processing parameters

SI	65536
SF	125.7803992 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	2.00

1D NMR plot parameters

CX	22.80 cm
CY	15.65 cm
F1P	180.000 ppm
F1	22640.47 Hz
F2P	-10.303 ppm
F2	-1295.90 Hz
PPMCM	8.34662 ppm/cm
HZCM	1049.84070 Hz/cm

sk-ii-96
1H spectrum



5

Current Data Parameters
USER cannon
NAME sk-ii-96
EXPNO 1
PROCNO 1

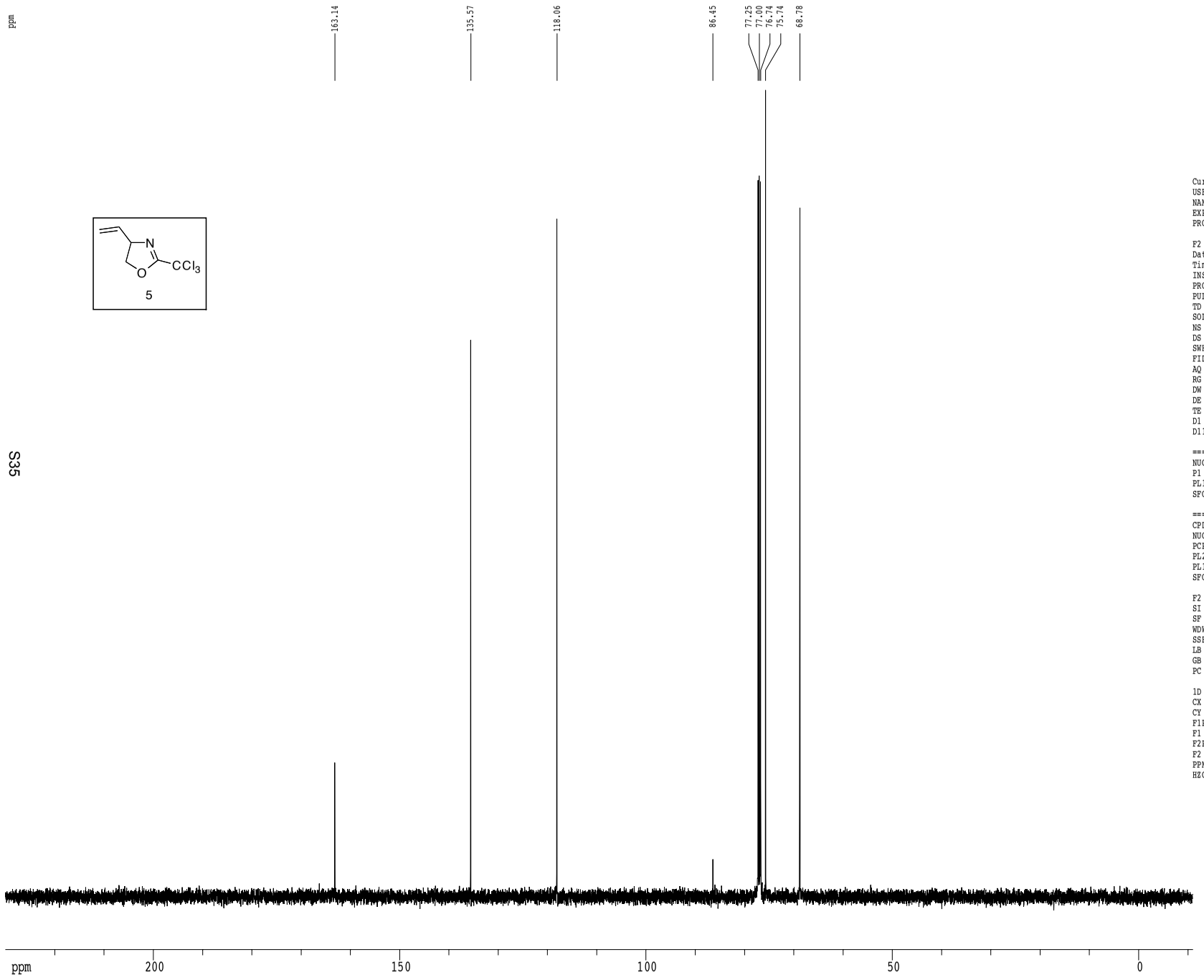
F2 - Acquisition Parameters
Date_ 20041021
Time 8.38
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 256
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

============ CHANNEL f1 ============
NUC1 1H
P1 13.00 usec
PL1 -1.00 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200312 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 9.000 ppm
F1 4501.98 Hz
F2P -1.072 ppm
F2 -536.08 Hz
PPMCM 0.44174 ppm/cm
HZCM 220.96764 Hz/cm

sk-ii-96
13C spectrum with 1H decoupling



Current Data Parameters
USER cannon
NAME sk-ii-96
EXPNO 2
PROCNO 1

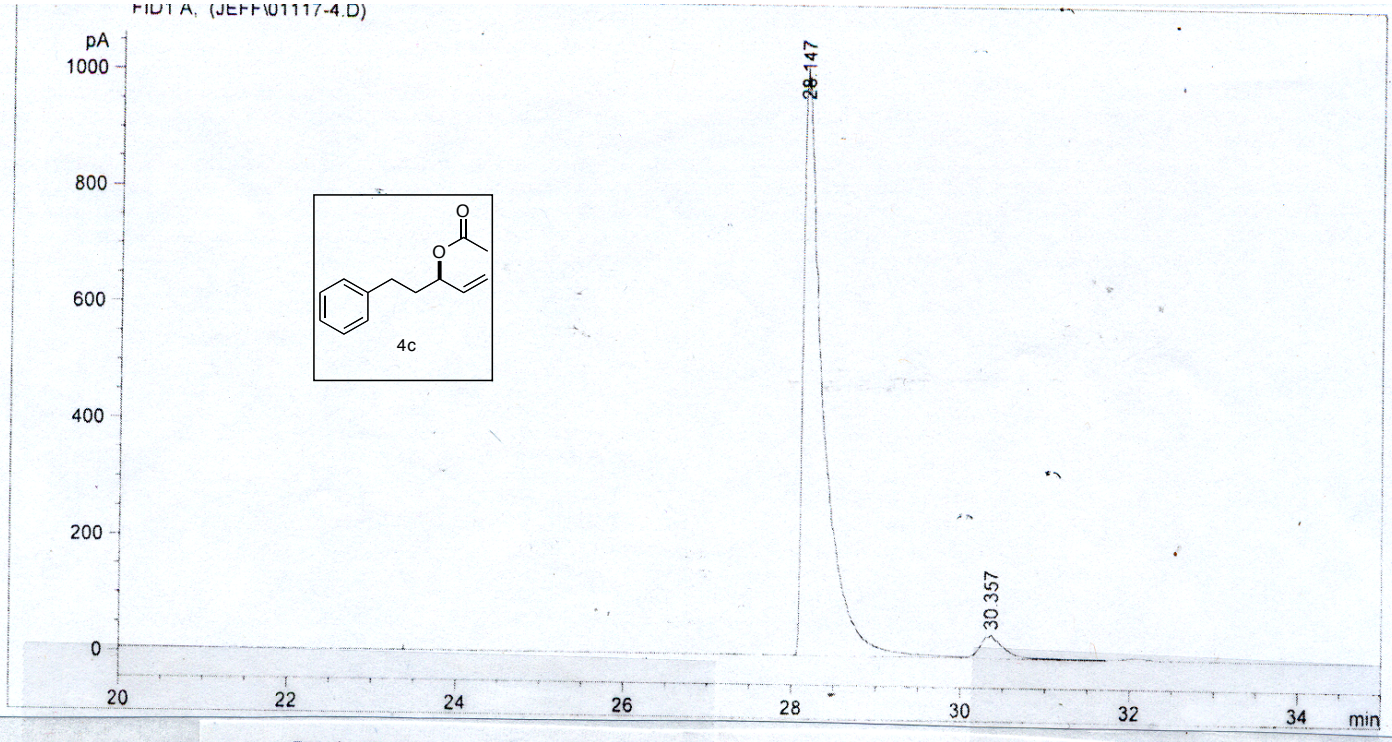
F2 - Acquisition Parameters
Date_ 20041021
Time 8.45
INSTRUM omega500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 828
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 5160.6
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec

========== CHANNEL f1 ==========
NUC1 13C
P1 22.50 usec
PL1 -6.00 dB
SFO1 125.7942048 MHz

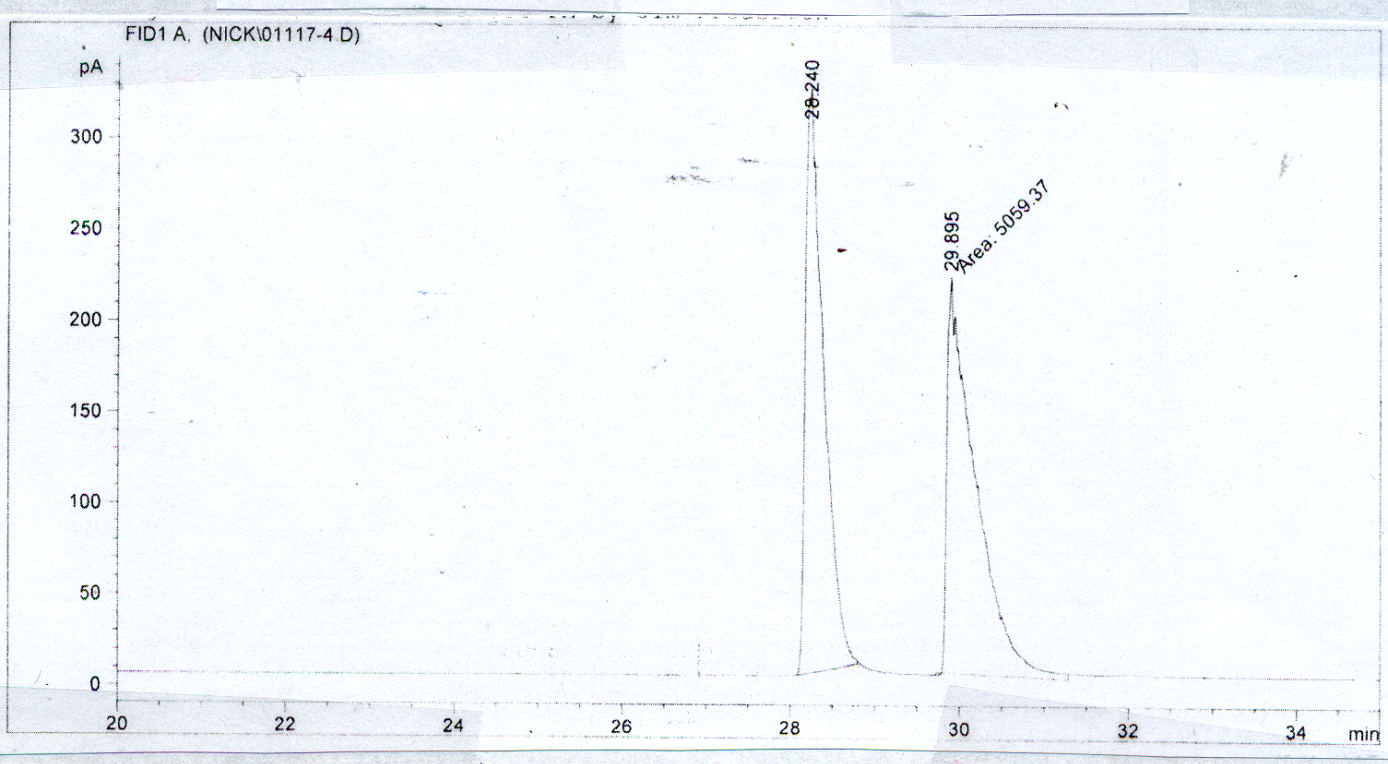
========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 14.40 dB
SFO2 500.2230013 MHz

F2 - Processing parameters
SI 65536
SF 125.7804290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
CY 15.65 cm
F1P 229.983 ppm
F1 28927.35 Hz
F2P -10.937 ppm
F2 -1375.68 Hz
PPMCM 10.56667 ppm/cm
HZCM 1329.08032 Hz/cm

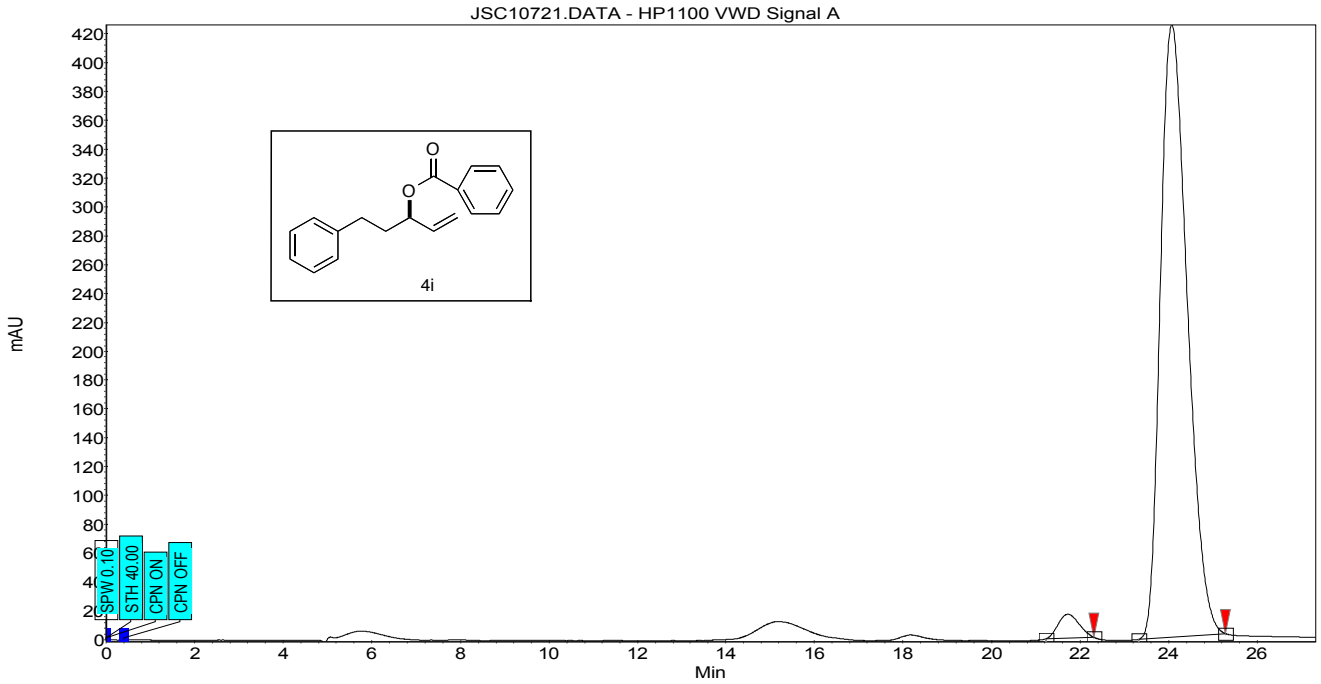


Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	28.147	VV	0.2239	1.64877e4	981.68097	95.40823
2	30.357	VV	0.2817	793.51178	38.94912	4.59177
Totals :				1.72812e4	1020.63009	



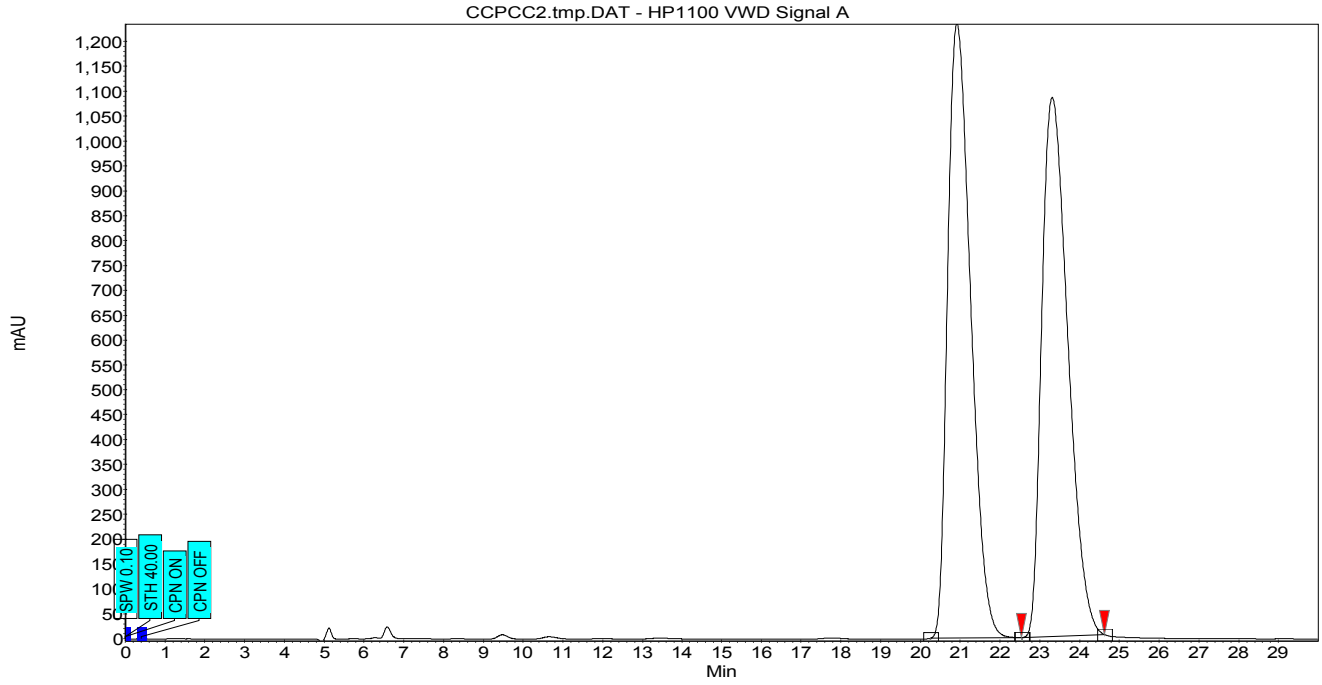
Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	28.240	PB	0.1814	4834.20068	319.73407	48.86204
2	29.895	MM	0.3882	5059.37109	217.20891	51.13796

Runinfo:28% hexanes, OJ column, 230nm 0.550 ml/min dihydrocinnamyl pivalate



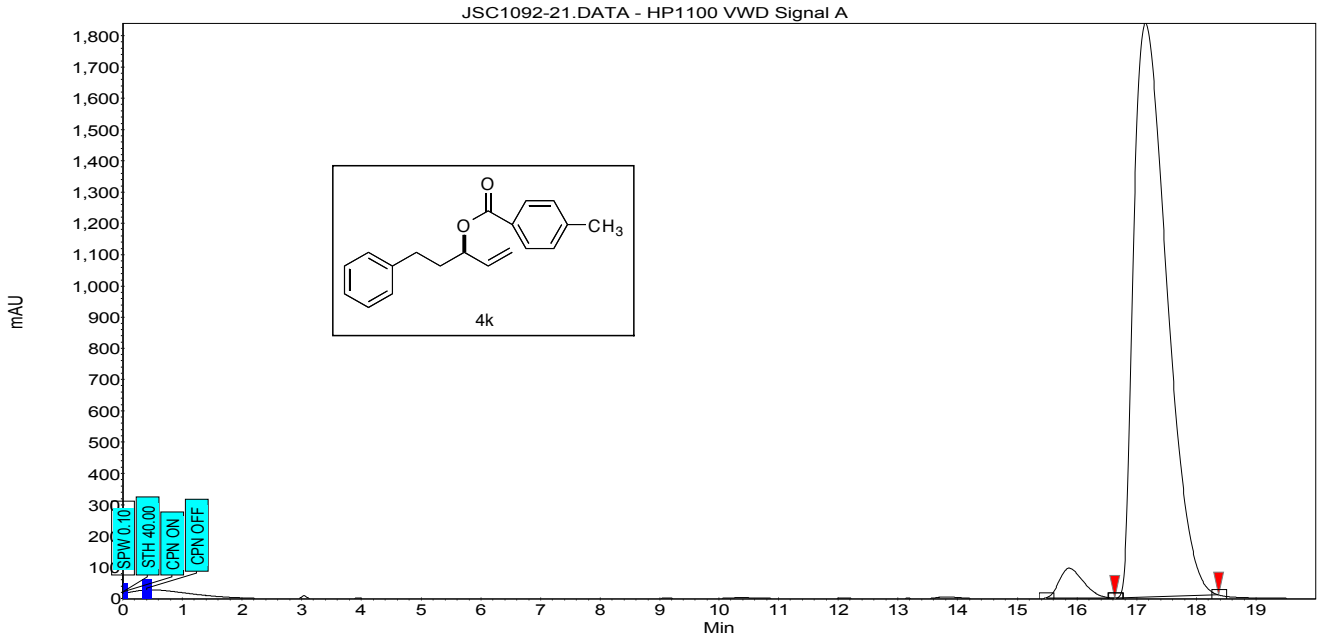
Index	Name	Time	Area	Height	Width	Area
		[Min]	[mAU*min]	[mAU]	[Min]	[%]
1	UNKNOWN	21.7151	8.9507	16.49	0.54	2.912
2	UNKNOWN	24.0698	298.4579	424.47	0.66	97.088
Total			307.4086			100.000

Runinfo:28% hexanes, OJ column, 230nm 0.550 ml/min dihydrocinnamyl pivalate



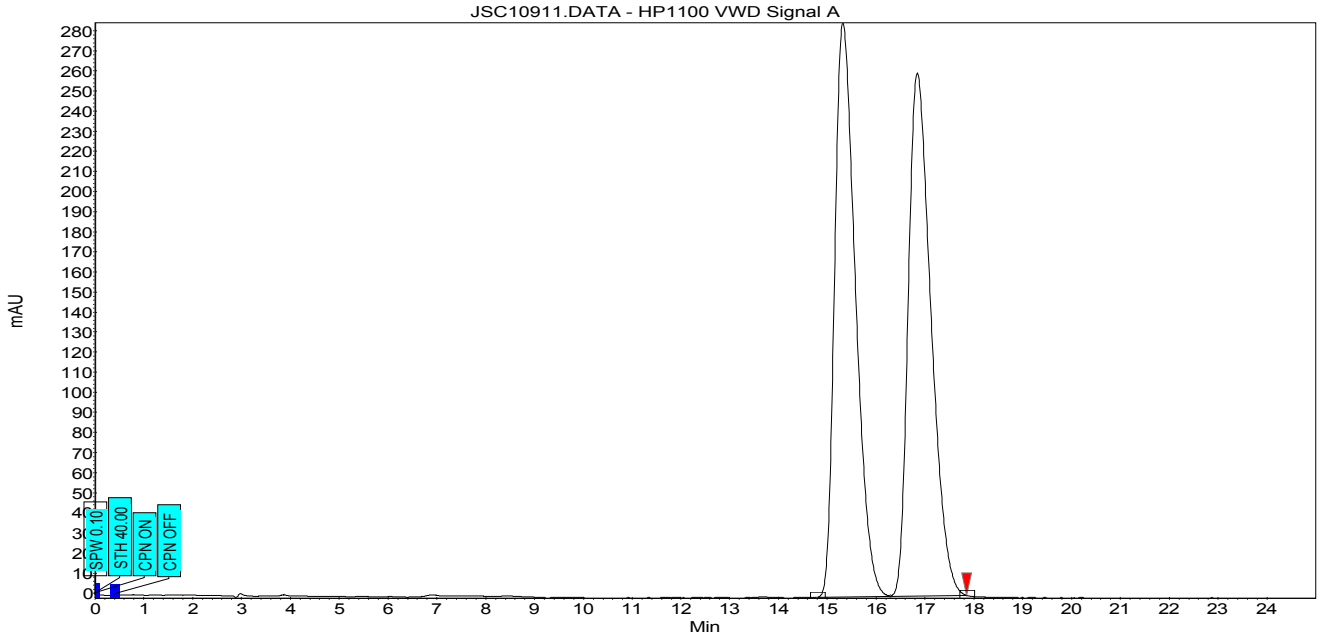
Index	Name	Time	Area	Height	Width	Area
		[Min]	[mAU*min]	[mAU]	[Min]	[%]
1	UNKNOWN	20.9205	826.4160	1234.04	0.64	50.103
2	UNKNOWN	23.3140	823.0108	1083.17	0.72	49.897
Total			1649.4268			100.000

Runinfo:28% hexanes, OJ column, 230nm 1 ml/min dihydrocinnamyl para-methylbenzoate



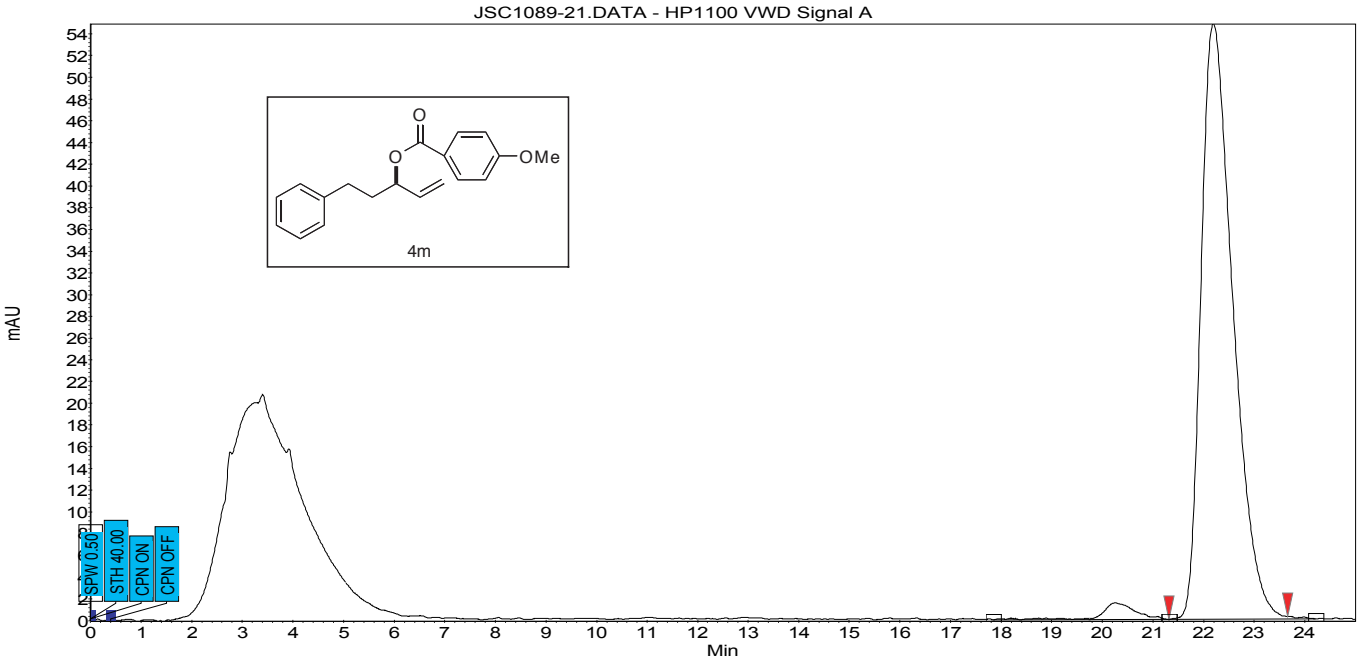
Index	Name	Time	Area	Height	Width	Area
		[Min]	[mAU*min]	[mAU]	[Min]	[%]
1	UNKNOWN	15.8721	44.4754	95.32	0.44	3.622
2	UNKNOWN	17.1512	1183.6092	1835.90	0.62	96.378
Total			1228.0846			100.000

Runinfo:28% hexanes, OJ column, 230nm 1 ml/min dihydrocinnamyl para-methylbenzoate



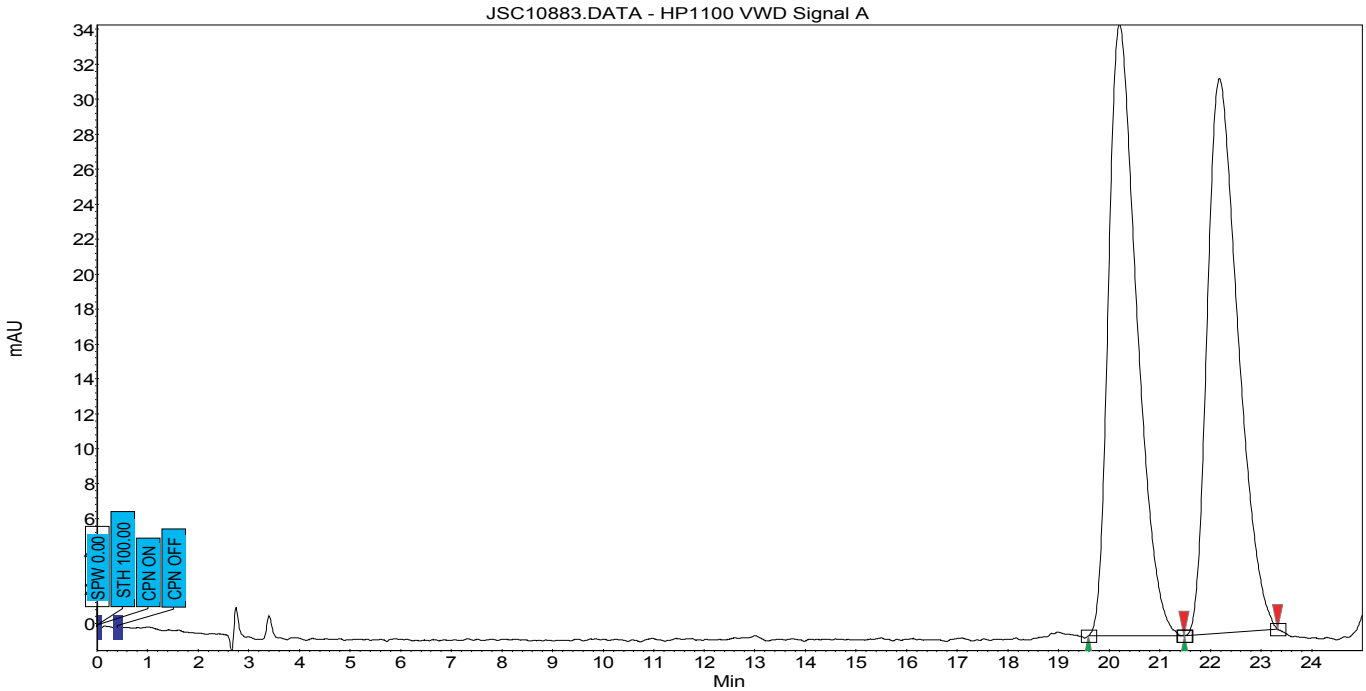
Index	Name	Time	Area	Height	Width	Area
		[Min]	[mAU*min]	[mAU]	[Min]	[%]
1	UNKNOWN	15.3198	140.9029	286.02	0.46	50.102
2	UNKNOWN	16.8411	140.3318	260.29	0.50	49.898
Total			281.2347			100.000

Runinfo:28% hexanes, OJ column, 230nm 1 ml/min dihydrocinnamyl para-methoxybenzoate



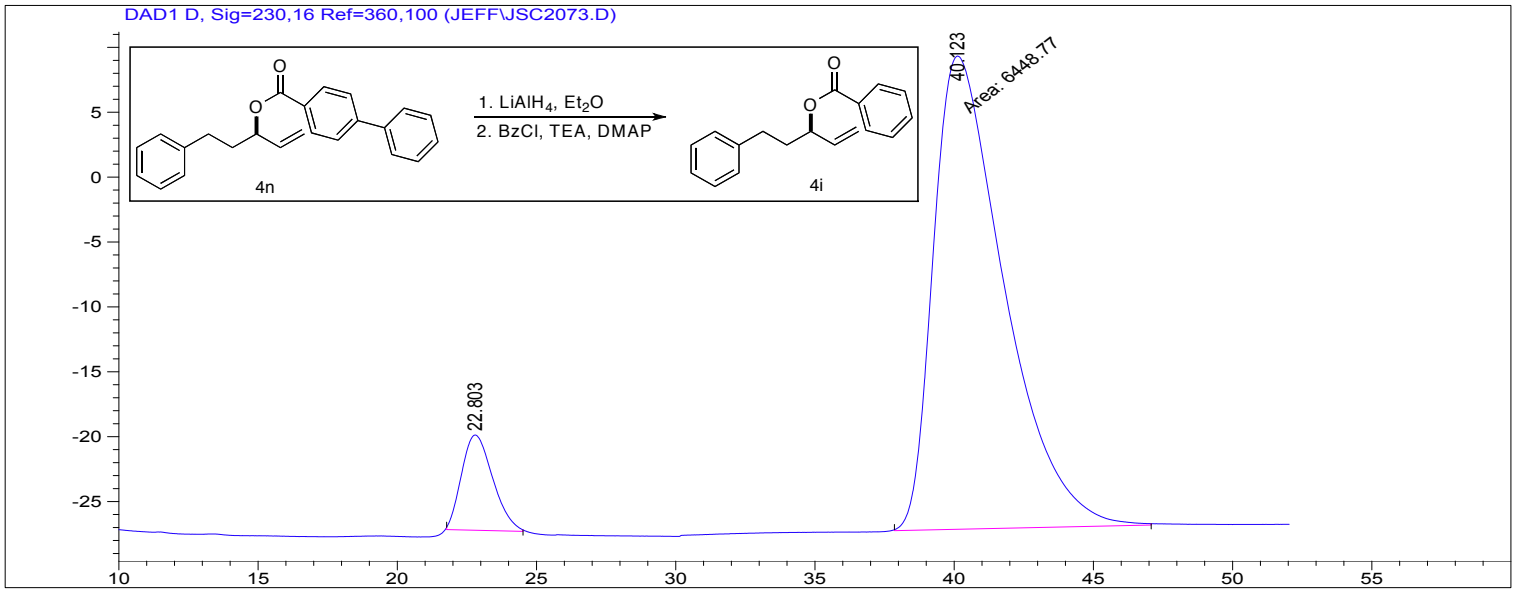
Index	Name	Time	Area	Height	Width	Area
		[Min]	[mAU*min]	[mAU]	[Min]	[%]
1	UNKNOWN	20.2519	1.0496	1.53	0.60	2.558
2	UNKNOWN	22.1899	39.9795	54.79	0.68	97.442
Total			41.0291			100.000

Runinfo:28% hexanes, OJ column, 230nm 1 ml/min dihydrocinnamyl para-methoxybenzoate



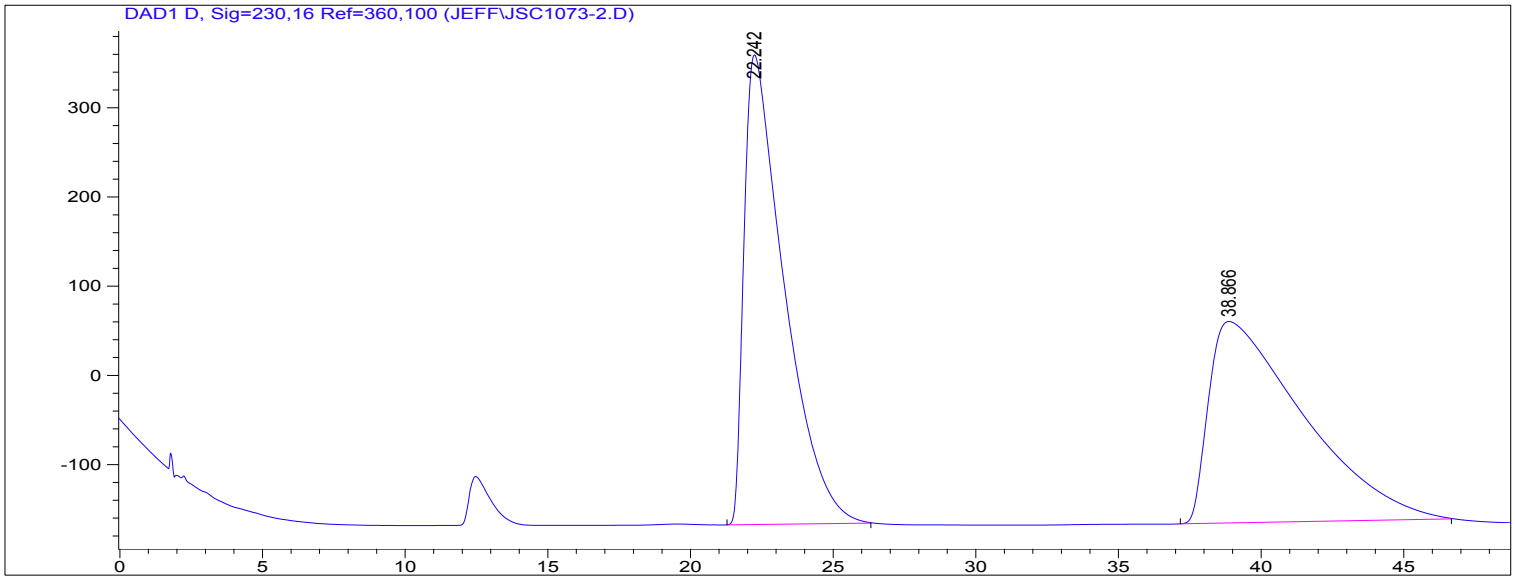
Index	Name	Time	Area	Height	Width	Area
		[Min]	[mAU*min]	[mAU]	[Min]	[%]
1	UNKNOWN	20.2035	22.5812	34.95	0.60	50.289
2	UNKNOWN	22.1802	22.3217	31.71	0.66	49.711
Total			44.9029			100.000

Method Info : OJ Column
0.1% IPA in heptanes
flow 2 mL/min
230 nm



Signal 1: DAD1 D, Sig=230,16 Ref=360,100

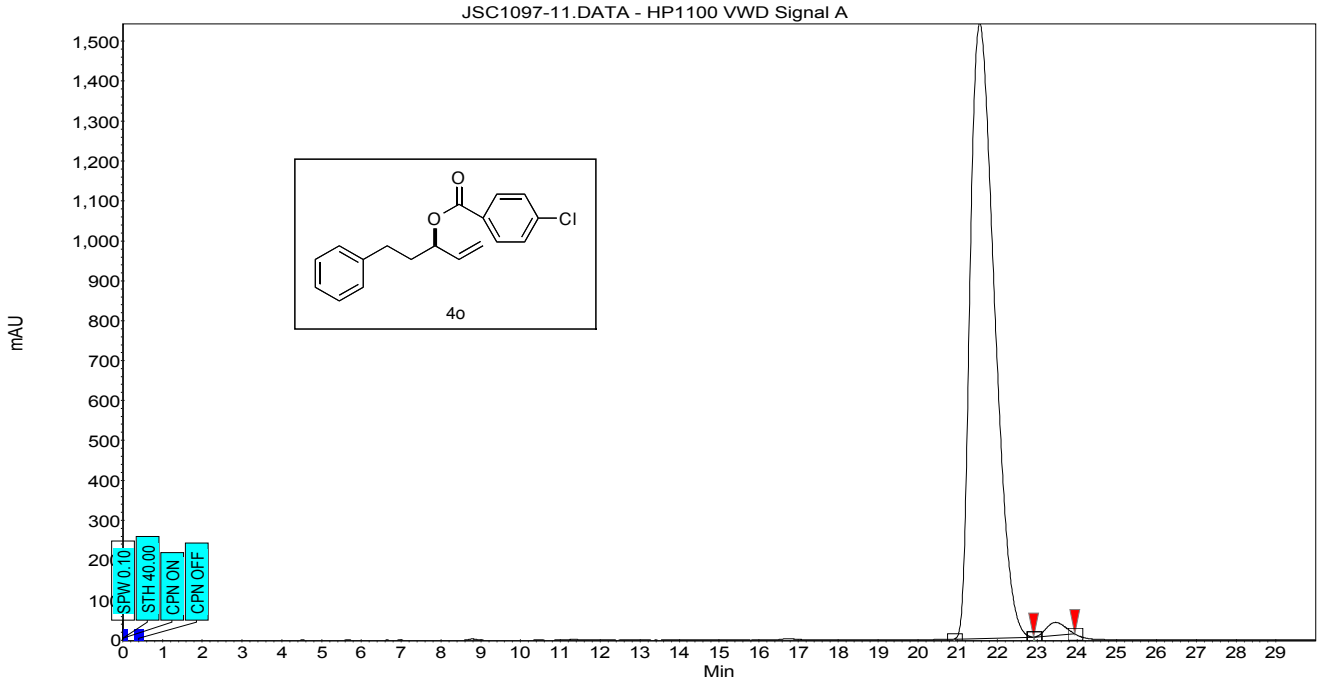
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.803	BB	0.9804	571.59839	7.34120	8.1420
2	40.123	MM	2.9478	6448.77197	36.46113	91.8580
Totals :				7020.37036	43.80233	



Signal 1: DAD1 D, Sig=230,16 Ref=360,100

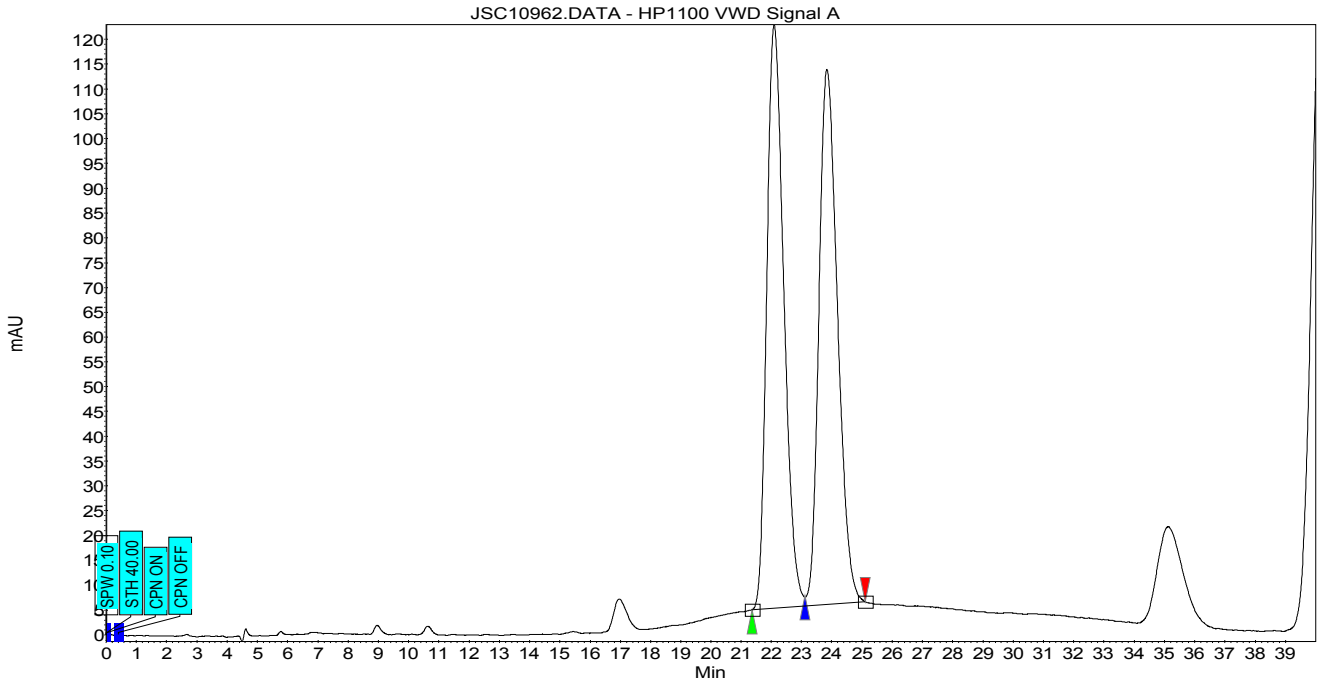
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.242	BB	1.4040	5.15449e4	526.59698	49.7296
2	38.866	BB	2.9971	5.21055e4	225.89069	50.2704
Totals :				1.03650e5	752.48767	

Runinfo:28% hexanes, OJ column, 230nm 1 ml/min dihydrocinnamyl para-chlorobenzoate



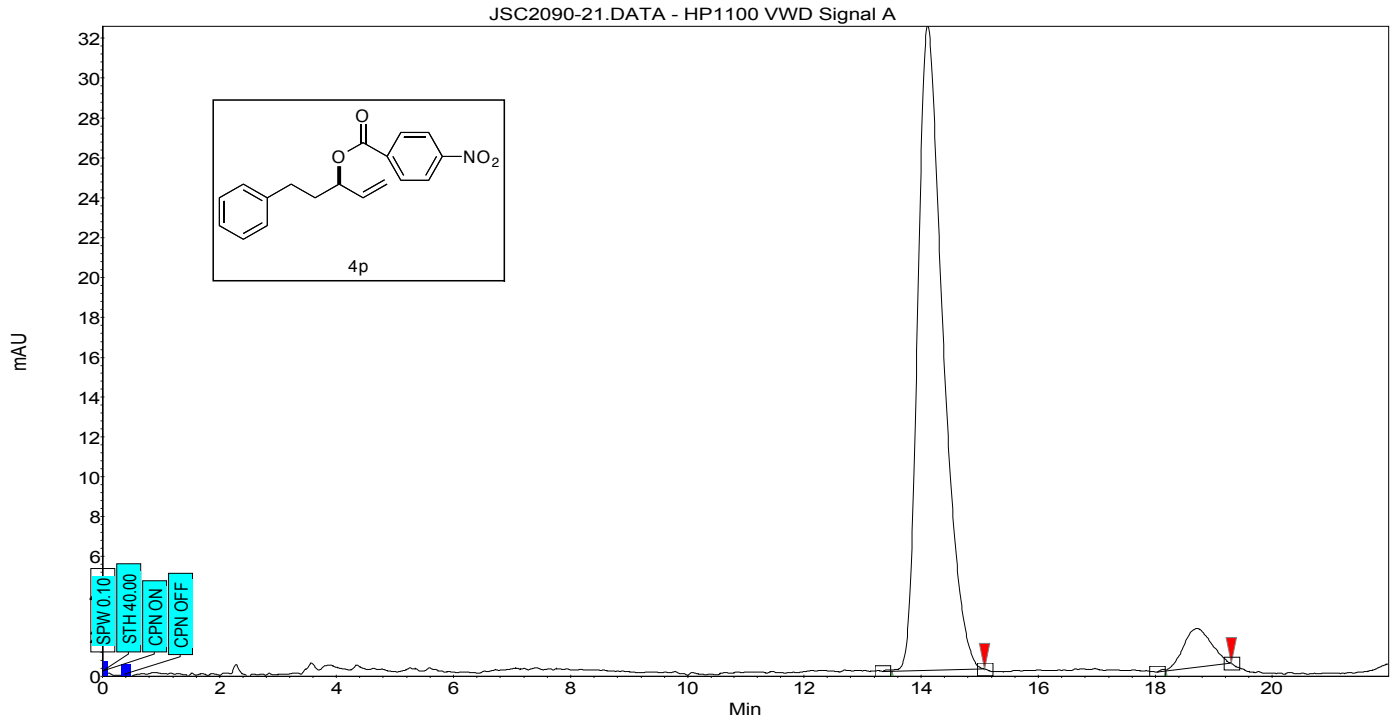
Index	Name	Time	Area	Height	Width	Area
		[Min]	[mAU*min]	[mAU]	[Min]	[%]
1	UNKNOWN	21.5504	1084.5034	1539.27	0.67	98.422
2	UNKNOWN	23.4496	17.3830	32.54	0.55	1.578
Total			1101.8864			100.000

Runinfo:28% hexanes, OJ column, 230nm 1 ml/min dihydrocinnamyl para-methoxybenzoate



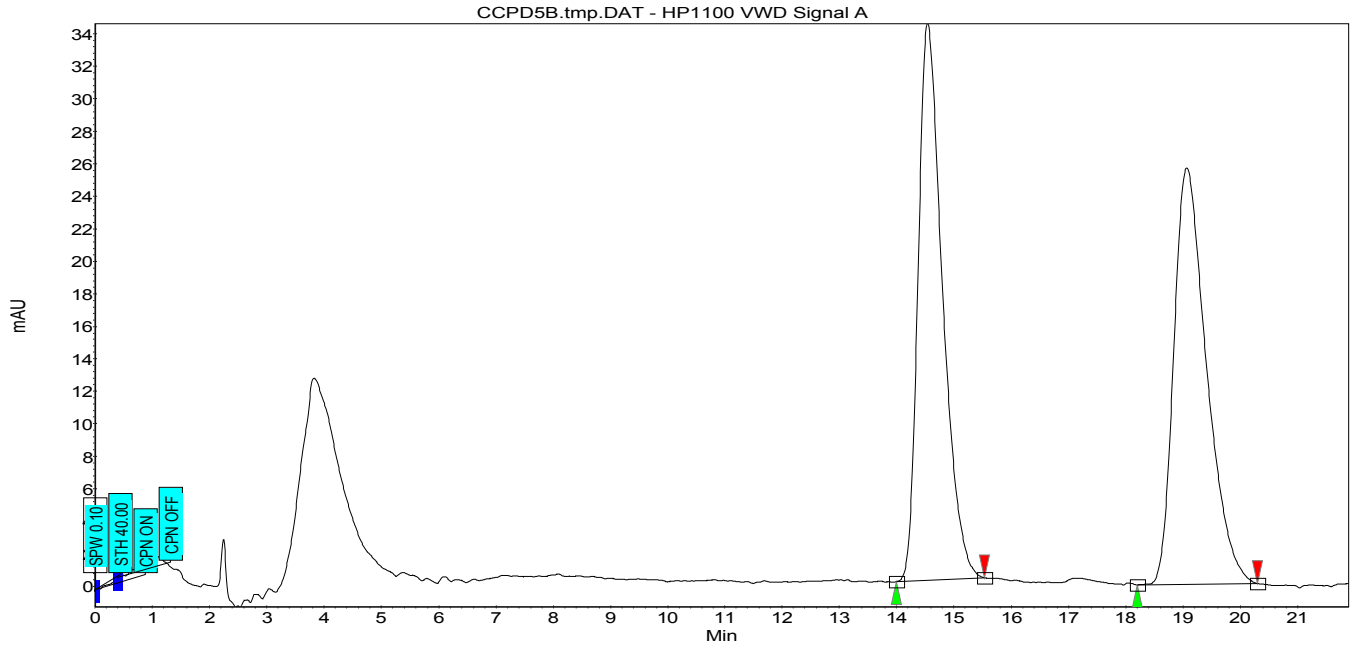
Index	Name	Time	Area	Height	Width	Area
		[Min]	[mAU*min]	[mAU]	[Min]	[%]
1	UNKNOWN	22.1027	76.5635	117.63	0.61	49.947
2	UNKNOWN	23.8469	76.7274	107.80	0.67	50.053
Total			153.2909			100.000

Runinfo:28% hexanes, OJ column, 230nm 2 ml/min, 35 degrees



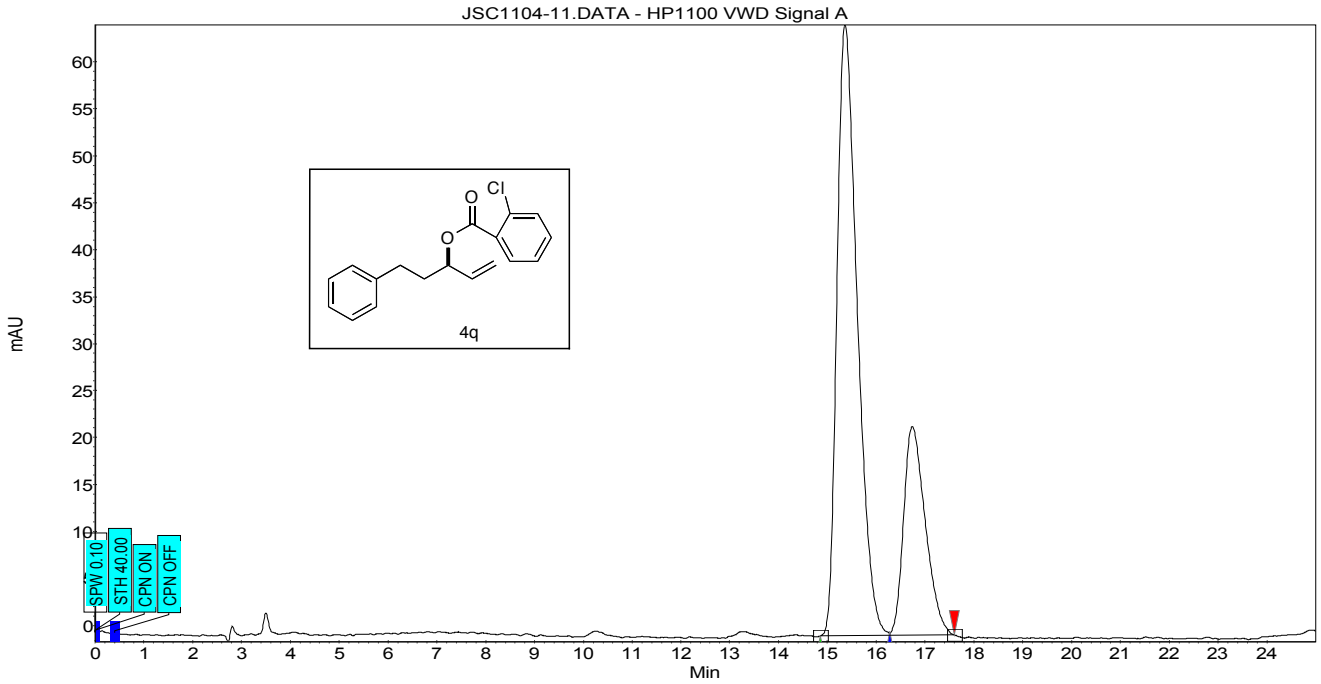
Index	Name	Time	Area	Height	Width	Area
		[Min]	[mAU*min]	[mAU]	[Min]	[%]
1	UNKNOWN	14.1085	16.1147	32.31	0.46	93.820
2	UNKNOWN	18.7016	1.0614	1.94	0.54	6.180
Total			17.1761			100.000

Runinfo:28% hexanes, OJ column, 230nm 2 ml/min, 35 degrees cinnamyl hydroxyl



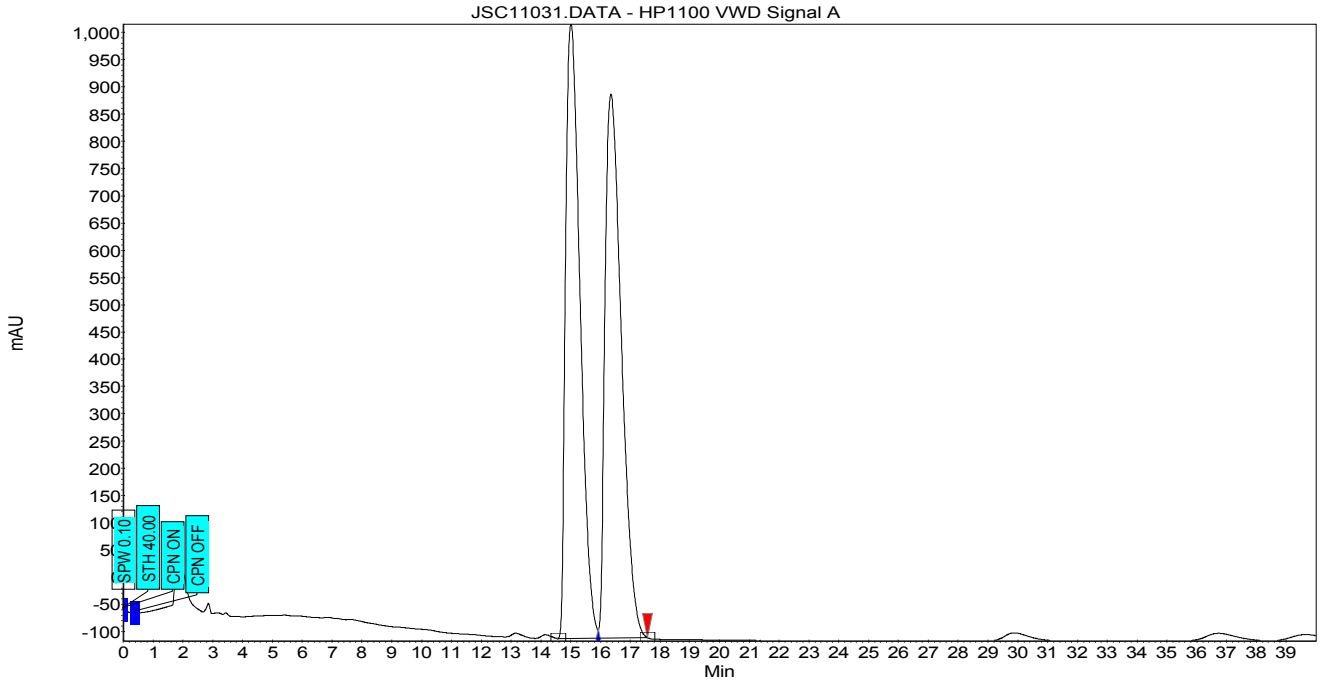
Index	Name	Time	Area	Height	Width	Area
		[Min]	[mAU*min]	[mAU]	[Min]	[%]
1	UNKNOWN	14.5446	16.8792	34.26	0.45	49.903
2	UNKNOWN	19.0601	16.9446	25.62	0.61	50.097
Total			33.8238			100.000

Runinfo:28% hexanes, OJ column, 230nm 1 ml/min dihydrocinnamyl para-chlorobenzoate



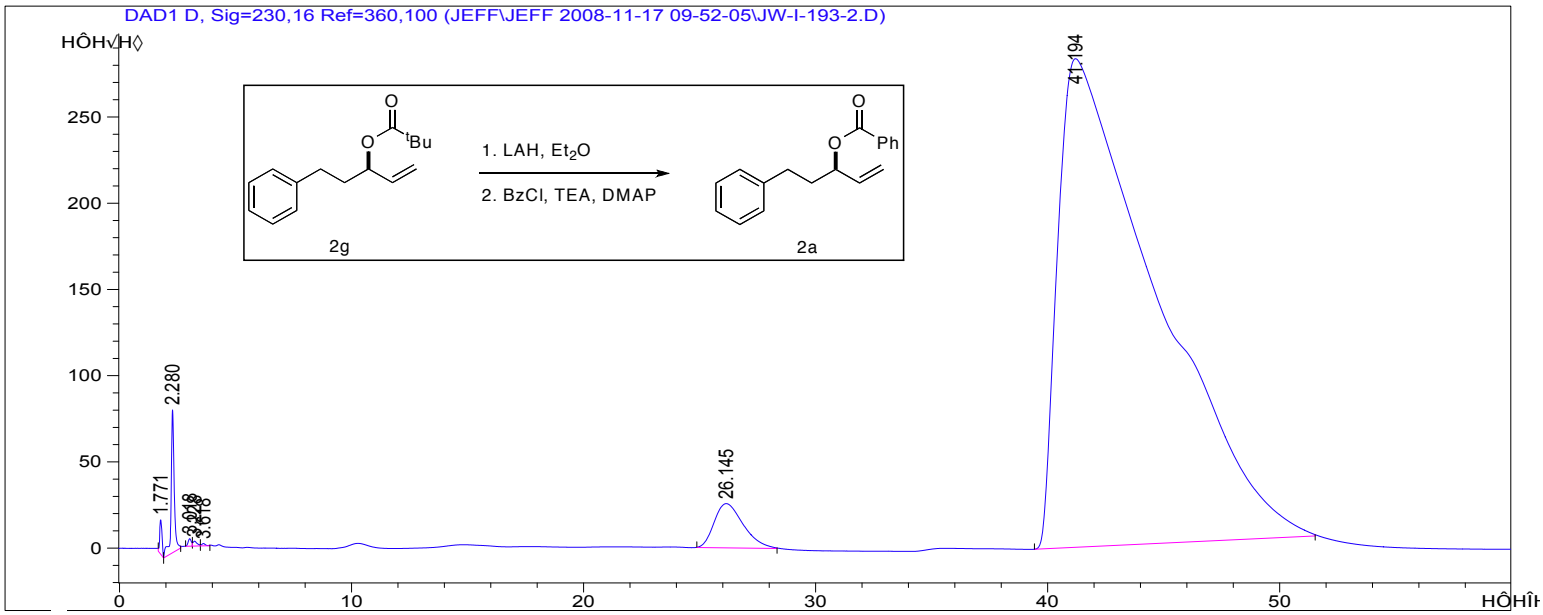
Index	Name	Time	Area	Height	Width	Area
		[Min]	[mAU*min]	[mAU]	[Min]	[%]
1	UNKNOWN	15.3682	31.3365	65.04	0.45	73.241
2	UNKNOWN	16.7442	11.4491	22.18	0.48	26.759
Total			42.7856			100.000

Runinfo:28% hexanes, OJ column, 230nm 1 ml/min dihydrocinnamyl benzoate



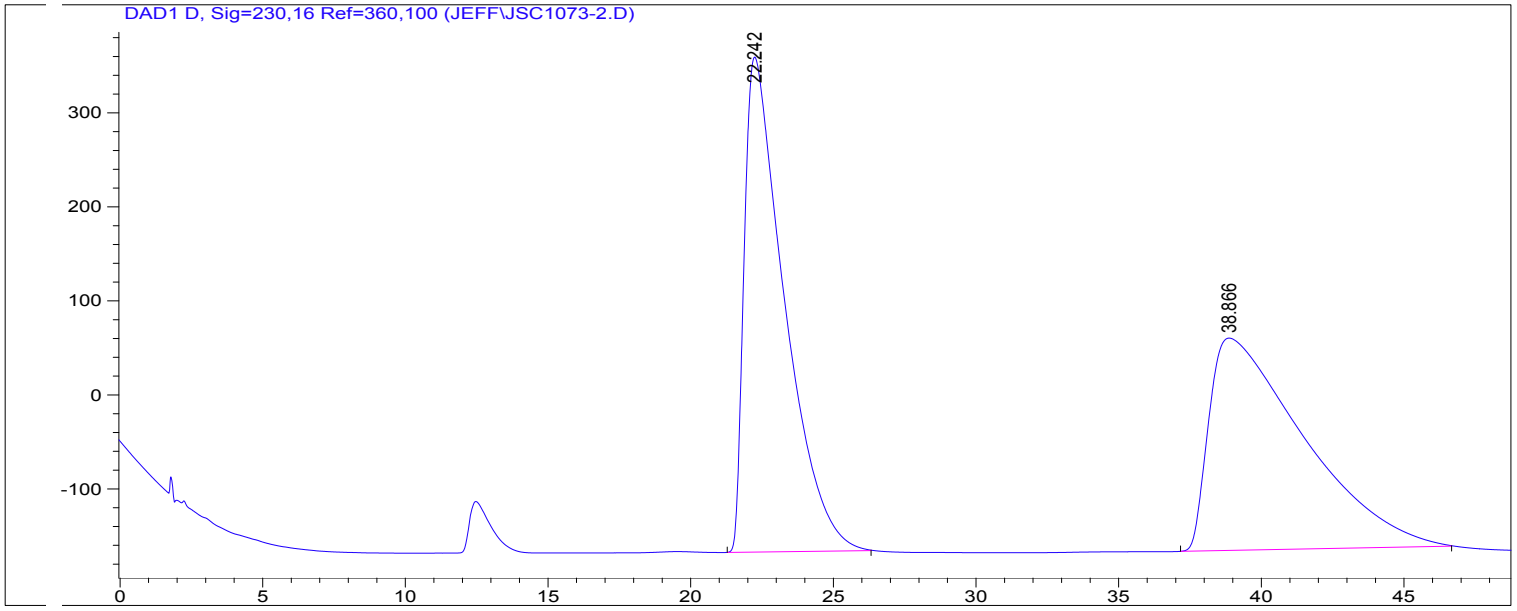
Index	Name	Time	Area	Height	Width	Area
		[Min]	[mAU*min]	[mAU]	[Min]	[%]
1	UNKNOWN	15.0097	634.7193	1127.80	0.54	49.873
2	UNKNOWN	16.3469	637.9589	997.82	0.61	50.127
Total			1272.6782			100.000

Method Info : OJ Column
0.1% IPA in heptanes
flow 2 mL/min
230 nm



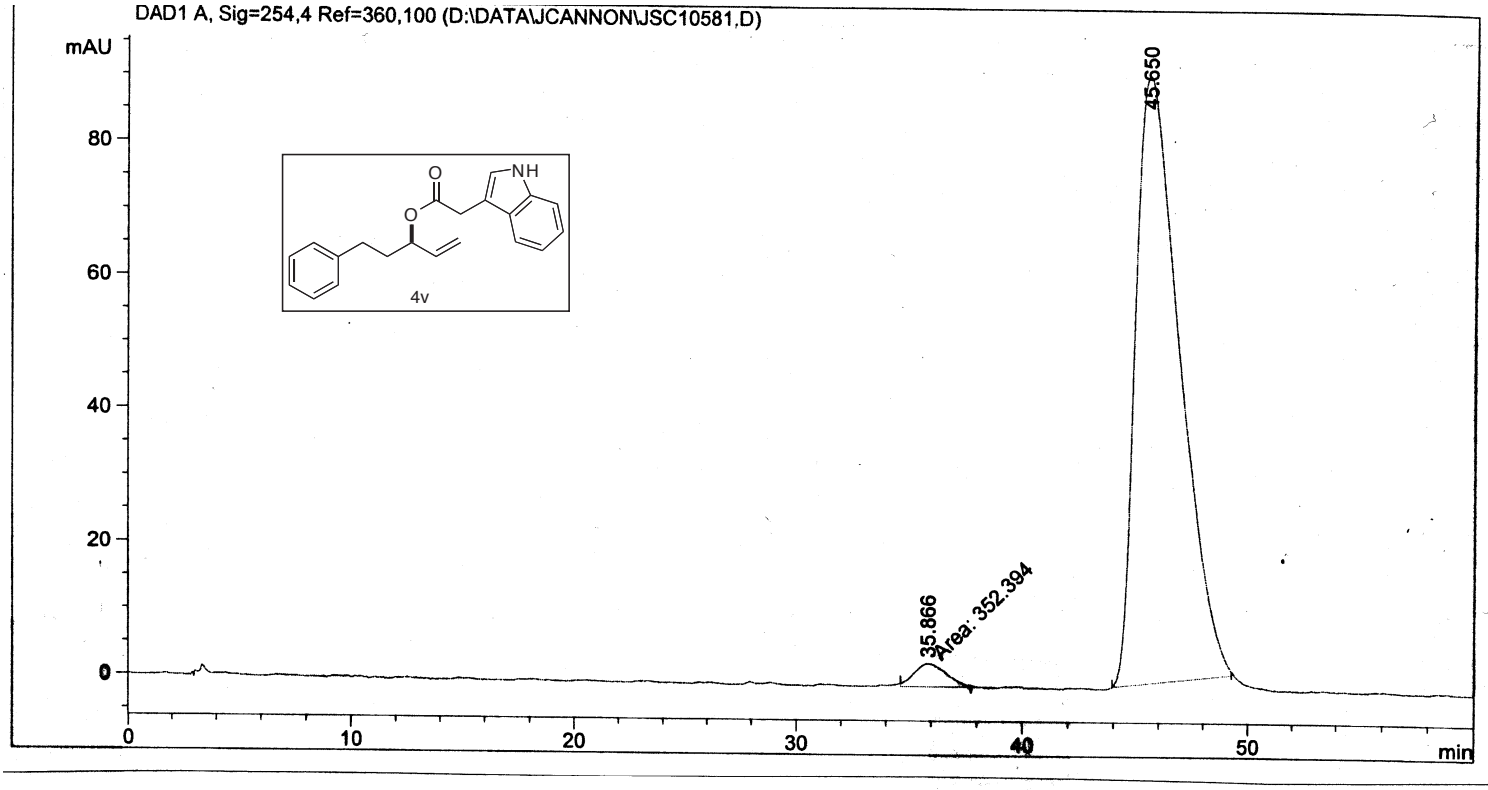
Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
6	26.145	BB	1.0220	2228.98437	25.64521	2.5665
7	41.194	BB	3.6474	8.36166e4	283.30359	96.2782



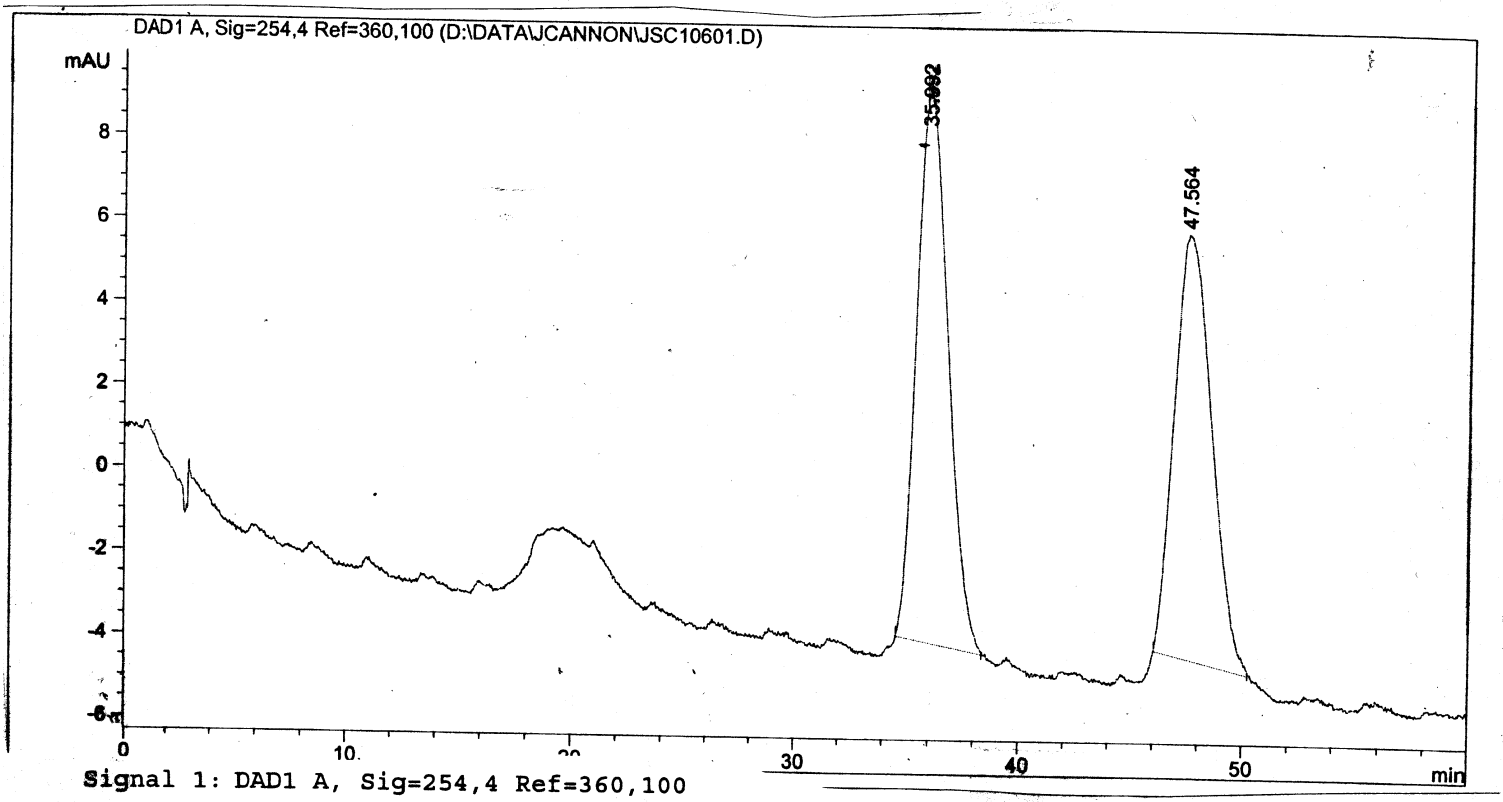
Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.242	BB	1.4040	5.15449e4	526.59698	49.7296
2	38.866	BB	2.9971	5.21055e4	225.89069	50.2704
Totals :				1.03650e5	752.48767	



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

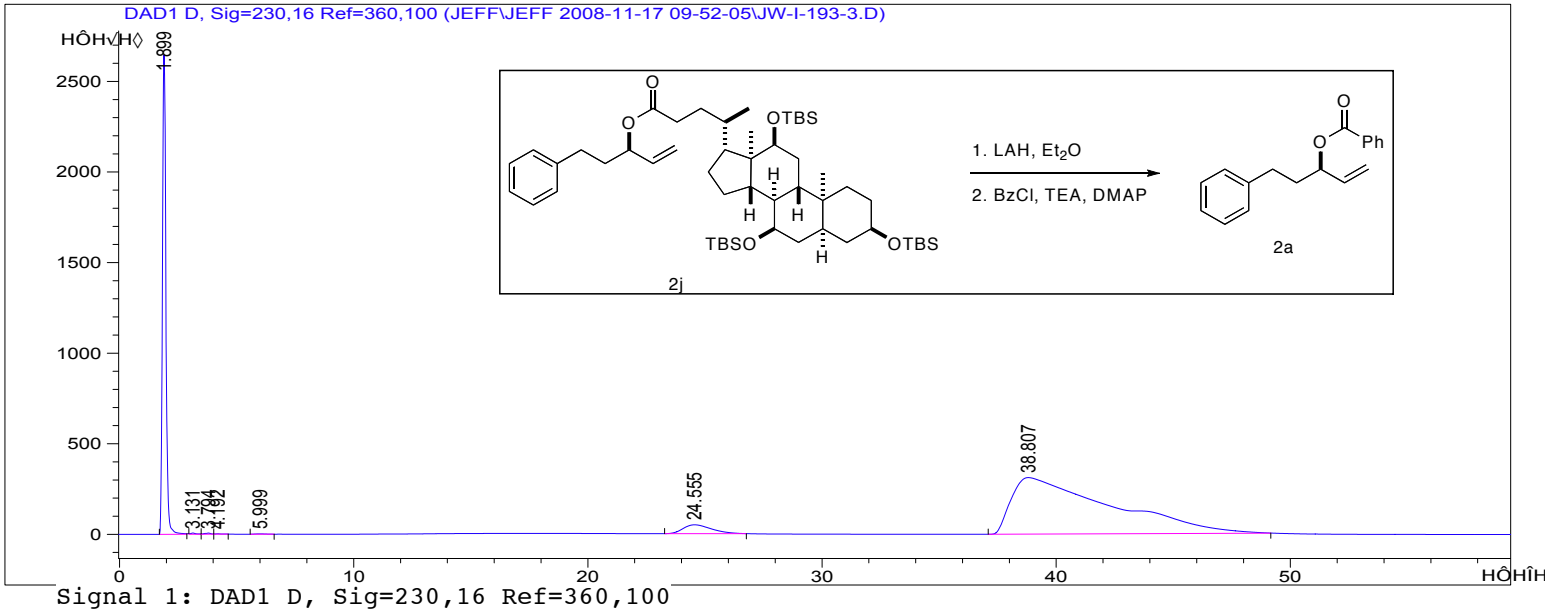
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	35.866	MM	1.6528	352.39398	3.55345	2.7464
2	45.650	BB	1.6192	1.24785e4	91.03713	97.2536
Totals :				1.28309e4	94.59059	



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

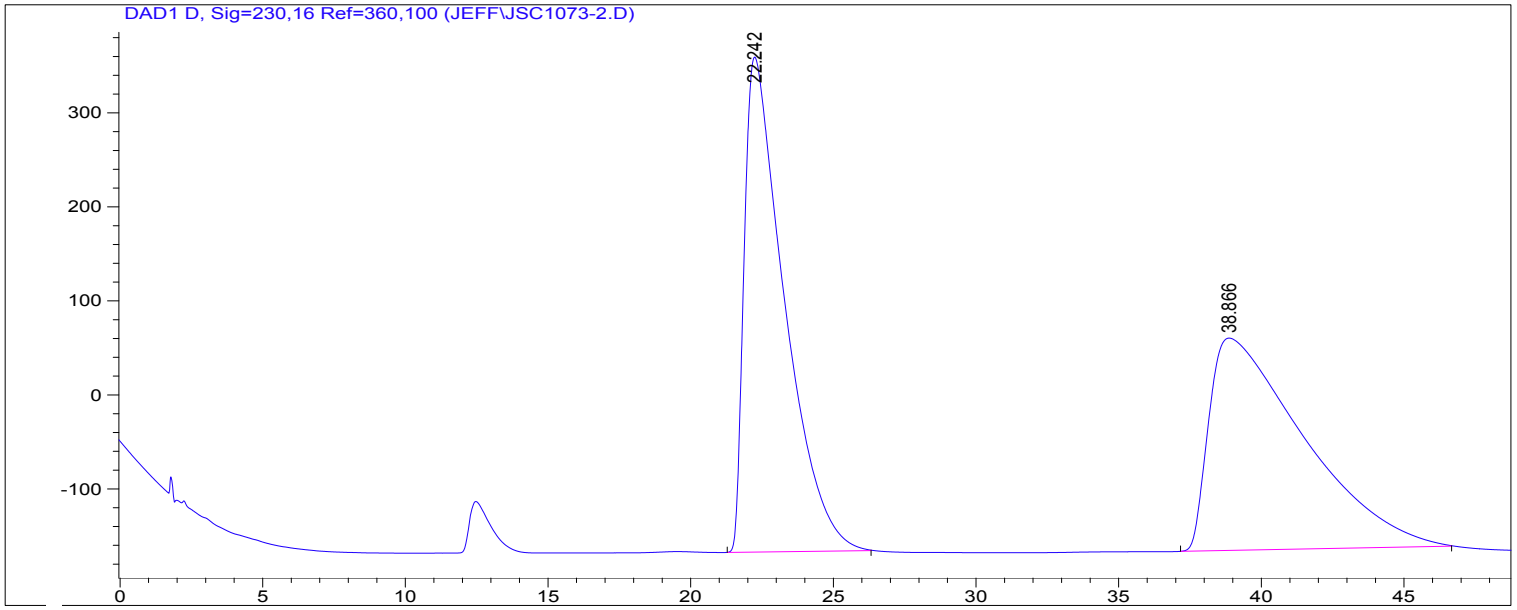
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	35.992	BB	1.1477	1286.02307	13.18718	52.0109
2	47.564	BB	1.3630	1186.58167	10.23144	47.9891

Method Info : OJ Column
0.1% IPA in heptanes
flow 2 mL/min
230 nm



Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
6	24.555	BB	1.1846	4140.99658	48.96955	3.3555
7	38.807	BB	3.5981	9.14118e4	312.18826	74.0730



Signal 1: DAD1 D, Sig=230,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.242	BB	1.4040	5.15449e4	526.59698	49.7296
2	38.866	BB	2.9971	5.21055e4	225.89069	50.2704

Totals : 1.03650e5 752.48767