

Supporting Information

Dynamic Kinetic Resolution of α -Keto Esters via Asymmetric Transfer Hydrogenation

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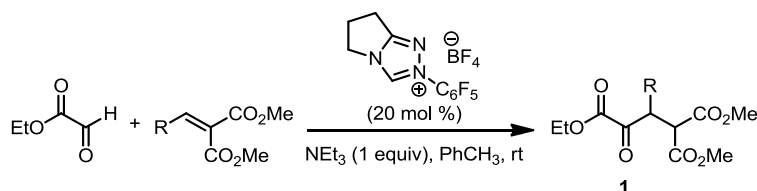
General Information

Methods: Infrared (IR) spectra were obtained using a Jasco 260 Plus Fourier transform infrared spectrometer. Proton and carbon magnetic resonance spectra (^1H NMR and ^{13}C NMR) were recorded on a Bruker model DRX 400 or 600 (^1H NMR at 400 MHz or 600 MHz and ^{13}C NMR at 100 MHz or 150 MHz) spectrometer with solvent resonance as the internal standard (^1H NMR: CDCl_3 at 7.26 ppm and ^{13}C NMR: CDCl_3 at 77.0 ppm). ^1H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, bs = broad singlet, d = doublet, dd = doublet of doublet, t = triplet, q = quartet, sept = septuplet, oct = octuplet, m = multiplet), coupling constants (Hz), and integration. Supercritical fluid chromatography was performed on a Berger SFC system equipped with a Chiralcel WO column. Samples were eluted with SFC grade CO_2 at the indicated percentage of MeOH. HPLC analysis was performed on an Agilent Technologies 1200 System equipped with Chiralpak IA, IB, and IC columns (constant flow at 1.00 mL/min). Optical rotations were measured using a 2 mL cell with a 1 dm path length on a Jasco DIP 1000 digital polarimeter. Mass spectra were obtained using a Micromass Quattro II (triple quad) instrument with nanoelectrospray ionization (Note: All samples prepared in methanol). Analytical thin layer chromatography (TLC) was performed on Whatman 0.25 mm silica gel 60 plates. Visualization was accomplished with UV light and/or aqueous ceric ammonium molybdate solution followed by heating. Purification of the reaction

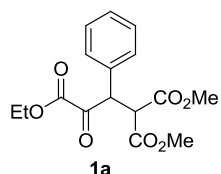
products was carried out by using Siliaflash-P60 silica gel (40-63 μ m) purchased from Silicycle. All reactions were carried out under an atmosphere of nitrogen in flame-dried glassware with magnetic stirring. Yield refers to isolated yield of analytically pure material unless otherwise noted. Yields and diastereomeric ratios (dr) are reported for a specific experiment and as a result may differ slightly from those found in the tables, which are averages of at least two experiments.

Materials: Benzylidene malonates were prepared according to known procedures. Ethyl glyoxylate was purchased from Sigma Aldrich as a 40% solution in toluene and distilled under reduced pressure prior to use (the concentration after distillation was determined by ^1H NMR). *N,N*-Dimethylformamide (DMF) was distilled from phosphorous pentoxide and stored under nitrogen over 3Å molecular sieves. Triethylamine (NEt_3) was freshly distilled from calcium hydride prior to use. Toluene (PhCH_3) and tetrahydrofuran (THF) were dried by passage through a column of neutral alumina under nitrogen prior to use. All other reagents were purchased from commercial sources and were used as received unless otherwise noted.

General Procedure A for the Preparation of β -Aryl α -Keto Esters 1a-1j

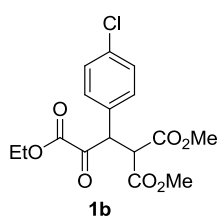


To a flame-dried 10-mL round-bottomed flask equipped with a magnetic stir bar were added benzylidene malonate (2.0 mmol, 1.0 equiv), ethyl glyoxylate (4.0 mmol, 2.0 equiv) and triazolium salt (0.40 mmol, 0.20 equiv). The flask was sealed with a rubber septum and purged with nitrogen. Toluene (0.5 M concentration with respect to benzylidene malonate) followed by triethylamine (2.0 mmol, 1.0 equiv) were then added. The reaction was stirred at room temperature for 16 h and diluted with ethyl acetate and water. The organic layer was washed with brine and dried over sodium sulfate. Concentration *in vacuo* afforded the β -aryl α -keto esters which were purified by flash chromatography using the indicated solvent systems.



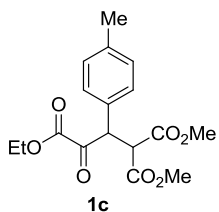
3-Ethyl 1,1-dimethyl 3-oxo-2-phenylpropane-1,1,3-tricarboxylate (1a): The title compound was prepared according to General Procedure A. Flash chromatography (20% EtOAc/hexanes) provided **1a** (0.618 g, 1.92 mmol, 96% yield) as a colorless oil. Analytical data for **1a**: $^1\text{H NMR}$ (600 MHz, CDCl_3): δ 7.31-7.27 (m, 3H), 7.25-7.24 (m, 2H), 5.22 (d, J = 12 Hz, 1H), 4.30 (d, J = 12 Hz, 1H), 4.26-4.19 (m, 2H), 3.74 (s, 3H), 3.46 (s, 3H), 1.26 (t, J = 7.2 Hz, 3 H); ^{13}C

NMR (150 MHz, CDCl_3): δ 190.2, 168.2, 167.4, 159.5, 131.4, 129.4, 129.1, 128.6, 62.7, 54.5, 53.2, 52.7, 52.6, 13.8; **IR** (thin film cm^{-1}): 2956, 1731, 1495, 1435, 1256, 1153, 1103, 1052, 854, 753, 700; **TLC** (20% EtOAc/hexanes): R_f : 0.27; **LRMS** (ESI): Calculated for $[\text{M} + \text{H}]^+$ $\text{C}_{16}\text{H}_{18}\text{O}_7$: 323.11, Found: 323.07.



3-Ethyl 1,1-dimethyl 2-(4-chlorophenyl)-3-oxopropane-1,1,3-tricarboxylate (1b): The title compound was prepared according to General Procedure A. Flash chromatography (20% EtOAc/hexanes) provided **1b** (0.660 g, 1.9 mmol, 95% yield) as a colorless oil. Analytical data for **1b**: $^1\text{H NMR}$ (600 MHz, CDCl_3): δ 7.30 (d, J = 7.8 Hz, 2H), 7.21 (d, J = 8.4 Hz, 2H), 5.22 (d, J = 12 Hz, 1H), 4.29 (d, J = 12 Hz, 1H), 4.28-4.22 (m, 2H), 3.75 (s, 3H), 3.51 (s, 3H), 1.29 (t, J = 7.2 Hz, 3H);

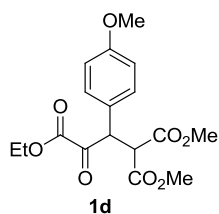
$^{13}\text{C NMR}$ (150 MHz, CDCl_3): δ 189.9, 168.0, 167.2, 159.4, 134.8, 130.7, 130.1, 129.4, 62.9, 54.5, 53.3, 52.7, 51.8, 13.9; **IR** (thin film, cm^{-1}): 3649, 2956, 1732, 1491, 1435, 1258, 1154, 1094, 1051, 936, 854, 717, 597; **TLC** (20% EtOAc/hexanes): R_f : 0.32; **LRMS** (ESI): Calculated for $[\text{M} + \text{H}]^+$ $\text{C}_{16}\text{H}_{18}\text{ClO}_7$: 357.07, Found: 357.02.



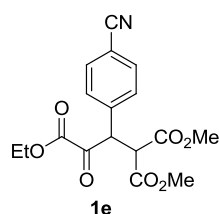
3-Ethyl 1,1-dimethyl 3-oxo-2-(p-tolyl)propane-1,1,3-tricarboxylate (1c): The title compound was prepared according to General Procedure A. Flash chromatography (20% EtOAc/hexanes) provided **1c** (0.618 g, 1.84 mmol, 92% yield) as a colorless oil. Analytical data for **1c**: $^1\text{H NMR}$ (600 MHz, CDCl_3): δ 7.12 (m, 4H), 5.19 (d, J = 12 Hz, 1H), 4.29 (d, J = 12 Hz, 1H), 4.27-4.18 (m, 2H), 3.75 (s, 3H), 3.49 (s, 3H), 2.30 (s, 3H), 1.28 (t, J = 7.2 Hz, 3H); $^{13}\text{C NMR}$ (150 MHz,

CDCl_3): δ 190.2, 168.3, 167.5, 159.6, 138.5, 129.9, 129.3, 128.2, 62.7, 54.5, 53.1, 52.6, 52.3, 21.1, 13.9; **IR** (thin film, cm^{-1}): 3649, 2956, 1731, 1513, 1436, 1257, 1153, 1100, 1053, 854, 801, 718, 601;

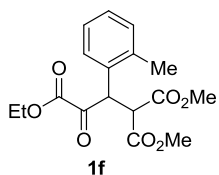
TLC (20% EtOAc/hexanes): R_f : 0.32; **LRMS** (ESI): Calculated for $[M+H]^+$ $C_{17}H_{21}O_7$: 337.13, Found: 337.15.



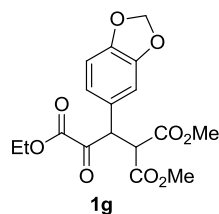
3-Ethyl 1,1-dimethyl 2-(4-methoxyphenyl)-3-oxopropane-1,1,3-tricarboxylate (1d): The title compound was prepared according to General Procedure A. Flash chromatography (30% EtOAc/hexanes) provided **1d** (0.655 g, 1.86 mmol, 93% yield) as a colorless oil. Analytical data for **1d**: 1H NMR (600 MHz, $CDCl_3$): δ 7.16 (d, J = 9.0 Hz, 2H), 6.83 (d, J = 8.4 Hz, 2H), 5.17 (d, J = 12 Hz, 1H), 4.27 (d, J = 12 Hz, 1H), 4.26-4.19 (m, 2H), 3.76 (s, 3H), 3.74 (s, 3H), 3.49 (s, 3H), 1.27 (t, J = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 190.0, 168.2, 167.5, 159.7, 159.6, 130.6, 123.0, 114.6, 62.7, 55.2, 54.4, 53.1, 52.6, 51.8, 13.9; **IR** (thin film, cm^{-1}): 2956, 1731, 1609, 1511, 1436, 1304, 1256, 1180, 1153, 1099, 1052, 1031, 832, 741, 602; **TLC** (30% EtOAc/hexanes): R_f : 0.34; **LRMS** (ESI): Calculated for $[M+H]^+$ $C_{17}H_{21}O_8$: 353.12, Found: 353.17.



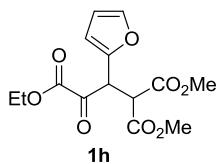
3-Ethyl 1,1-dimethyl 2-(4-cyanophenyl)-3-oxopropane-1,1,3-tricarboxylate (1e): The title compound was prepared according to General Procedure A. Flash chromatography (30% EtOAc/hexanes) provided **1e** (0.625 g, 1.80 mmol, 90% yield) as a white solid. Analytical data for **1e**: 1H NMR (600 MHz, $CDCl_3$): δ 7.62 (d, J = 7.8 Hz, 2H), 7.42 (d, J = 7.8 Hz, 2H), 5.29 (d, J = 12 Hz, 1H), 4.32 (d, J = 12 Hz, 1H), 4.30-4.23 (m, 2H), 3.75 (s, 3H), 3.50 (s, 3H), 1.30 (t, J = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 189.5, 167.8, 166.9, 159.2, 137.3, 132.7, 130.1, 118.0, 112.7, 63.1, 54.5, 53.4, 52.8, 52.2, 13.8; **IR** (thin film, cm^{-1}): 2957, 2230, 1732, 1506, 1436, 1259, 1156, 1097, 1051, 836; **m.p.** 71-73 $^{\circ}C$; **TLC** (30% EtOAc/hexanes): R_f : 0.32; **LRMS** (ESI): Calculated for $[M+H]^+$ $C_{17}H_{18}NO_7$: 348.11, Found: 348.14.



3-Ethyl 1,1-dimethyl 3-oxo-2-(o-tolyl)propane-1,1,3-tricarboxylate (1f): The title compound was prepared according to General Procedure A. Flash chromatography (20% EtOAc/hexanes) provided **1f** (0.524 g, 1.56 mmol, 78% yield) as a colorless oil. Analytical data for **1f**: 1H NMR (600 MHz, $CDCl_3$): δ 7.18-7.12 (m, 3H), 7.11-7.01 (m, 2H), 5.47 (d, J = 11.6 Hz, 1H), 4.29 (d, J = 11.6 Hz, 1H), 4.25-4.16 (m, 2H), 3.76 (s, 3H), 3.42 (s, 3H), 2.54 (s, 3H), 1.24 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.3, 168.3, 167.6, 159.8, 138.5, 131.4, 129.7, 128.5, 128.2, 126.5, 62.7, 54.4, 53.1, 52.4, 48.3, 19.6, 13.8; **IR** (thin film, cm^{-1}): 3578, 2964, 1734, 1523, 1441, 1264, 1148, 1110, 1057, 852, 806, 714, 605; **TLC** (20% EtOAc/hexanes): R_f : 0.33; **LRMS** (ESI): Calculated for $[M+H]^+$ $C_{17}H_{21}O_7$: 337.13, Found: 337.14.

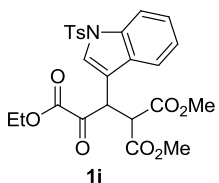


3-Ethyl 1,1-dimethyl 2-(benzo[d][1,3]dioxol-5-yl)-3-oxopropane-1,1,3-tricarboxylate (1g): The title compound was prepared according to General Procedure A on a 10 mmol scale. Flash chromatography (30% EtOAc/hexanes) provided **1g** (3.33 g, 9.10 mmol, 91% yield) as a viscous yellow oil. Analytical data for **1g**: 1H NMR (600 MHz, $CDCl_3$): δ 6.74-6.70 (m, 3H), 5.95 (bs, 2H), 5.14 (d, J = 12 Hz, 1H), 4.24 (d, J = 12 Hz, 1H), 4.29-4.22 (m, 2H), 3.74 (s, 3H), 3.54 (s, 3H), 1.29 (t, J = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 189.9, 168.2, 167.4, 159.6, 148.2, 147.9, 124.6, 123.2, 109.5, 108.8, 101.4, 62.8, 54.6, 53.2, 52.7, 52.0, 13.9; **IR** (thin film, cm^{-1}): 2956, 2360, 1731, 1505, 1489, 1440, 1249, 1225, 1153, 1095, 1037, 932, 857, 812, 638; **TLC** (30% EtOAc/hexanes): R_f : 0.35; **LRMS** (ESI): Calculated for $[M+H]^+$ $C_{17}H_{19}O_9$: 367.10, Found: 367.01.



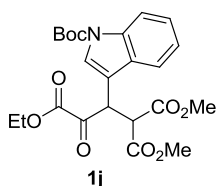
3-Ethyl 1,1-dimethyl 2-(furan-2-yl)-3-oxopropane-1,1,3-tricarboxylate (1h):

The title compound was prepared according to General Procedure A. Flash chromatography (20% EtOAc/hexanes) provided **1h** (0.543 g, 1.74 mmol, 87% yield) as an orange oil. Analytical data for **1h**: $^1\text{H NMR}$ (600 MHz, CDCl_3): δ 7.37 (s, 1H), 6.32-6.30 (m, 2H), 5.40 (d, J = 12 Hz, 1H), 4.36 (d, J = 12 Hz, 1H), 4.33-4.25 (m, 2H), 3.75 (s, 3H), 3.62 (s, 3H), 1.32 (t, J = 7.2 Hz, 3H); $^{13}\text{C NMR}$ (150 MHz, CDCl_3): δ 187.3, 167.9, 167.3, 159.5, 145.0, 143.9, 111.0, 110.7, 62.9, 53.2, 52.9, 52.4, 46.5, 13.9; **IR** (thin film, cm^{-1}): 2957, 1736, 1499, 1436, 1252, 1153, 1100, 1051, 1014, 749, 599; **TLC** (20% EtOAc/hexanes): R_f : 0.27; **LRMS** (ESI): Calculated for $[\text{M}+\text{H}]^+$ $\text{C}_{14}\text{H}_{17}\text{O}_8$: 313.09, Found: 313.13.



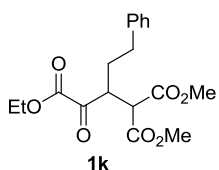
3-Ethyl 1,1-dimethyl 3-oxo-2-(1-tosyl-1H-indol-3-yl)propane-1,1,3-tricarboxylate (1i):

The title compound was prepared according to General Procedure A. Flash chromatography (30% EtOAc/hexanes) provided **1i** (0.918 g, 1.78 mmol, 89% yield) as a viscous yellow oil. Analytical data for **1i**: $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.94 (d, J = 8.4 Hz, 1H), 7.70 (d, J = 8.4 Hz, 2H), 7.67 (m, 1H), 7.51 (s, 1H), 7.33-7.30 (m, 2H), 7.23-7.21 (m, 2H), 5.48 (d, J = 11.6 Hz, 1H), 4.39 (d, J = 11.6 Hz, 1H), 4.21-4.12 (m, 2H), 3.77 (s, 3H), 3.25 (s, 3H), 2.33 (s, 3H), 1.15 (t, J = 7.2 Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 188.9, 168.0, 167.2, 159.5, 145.1, 135.1, 134.9, 129.9, 126.8, 126.4, 125.4, 123.7, 119.9, 113.6, 113.4, 62.8, 53.9, 53.2, 52.5, 43.5, 21.5, 13.7; **IR** (thin film, cm^{-1}): 2955, 1732, 1596, 1447, 1372, 1286, 1251, 1176, 1123, 1093, 1050, 979, 814, 748, 704, 669, 573, 538; **TLC** (30% EtOAc/hexanes): R_f : 0.25; **LRMS** (ESI): Calculated for $[\text{M}+\text{Na}]$ $\text{C}_{25}\text{H}_{25}\text{NO}_9\text{SNa}$ + MeOH: 570.14, Found: 570.03.



3-Ethyl 1,1-dimethyl 2-(1-(tert-butoxycarbonyl)-1H-indol-3-yl)-3-oxopropane-1,1,3-tricarboxylate (1j):

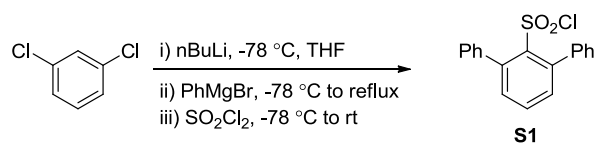
The title compound was prepared according to General Procedure A. Flash chromatography (20% EtOAc/hexanes) provided **1j** (0.775 g, 1.68 mmol, 84% yield) as a pale yellow solid. Analytical data for **1j**: $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 8.12 (d, J = 8.0 Hz, 1H), 7.70-7.68 (m, 1H), 7.45 (s, 1H), 7.35-7.27 (m, 2H), 5.53 (d, J = 11.6 Hz, 1H), 4.39 (d, J = 11.6 Hz, 1H), 4.25-4.15 (m, 2H), 3.78 (s, 3H), 3.43 (s, 3H), 1.65 (s, 9H), 1.22 (t, J = 7.2 Hz, 3H); $^{13}\text{C NMR}$ (150 MHz, CDCl_3): δ 188.9, 168.2, 167.2, 159.5, 149.1, 135.5, 128.7, 125.9, 125.0, 123.0, 119.3, 115.2, 111.0, 84.3, 62.8, 54.0, 53.2, 52.7, 43.3, 28.1, 13.7; **IR** (thin film, cm^{-1}): 2980, 1733, 1453, 1367, 1257, 1154, 1083, 1051, 748; **m.p.**: 96-97.5 $^{\circ}\text{C}$; **TLC** (20% EtOAc/hexanes) R_f : 0.29; **LRMS** (ESI): Calculated for $[\text{M}+\text{Na}]$ $\text{C}_{23}\text{H}_{17}\text{NO}_9\text{Na}$ + MeOH: 516.18, Found: 516.21.



3-Ethyl 1,1-dimethyl 3-oxo-2-phenethylpropane-1,1,3-tricarboxylate (1k):

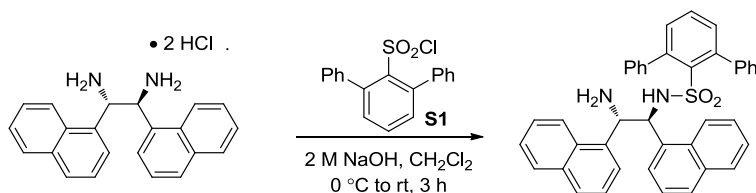
The title compound was prepared according to General Procedure A. Flash chromatography (20% EtOAc/hexanes) provided **1k** (0.775 g, 1.86 mmol, 93% yield) as a colorless oil. Analytical data for **1k**: $^1\text{H NMR}$ (600 MHz, CDCl_3): δ 7.29-7.26 (m, 2H), 7.21-7.18 (m, 1H), 7.12-7.11 (m, 2H), 4.41-4.33 (m, 2H), 4.13 (ddd, J = 10.8, 8.4, 4.2 Hz, 1H), 3.97 (d, J = 10.8 Hz, 1H), 3.98 (s, 3H), 3.97 (s, 3H), 2.59-2.56 (m, 2H), 1.99-1.87 (m, 2H), 1.40 (t, J = 7.2 Hz, 3H); $^{13}\text{C NMR}$ (150 MHz, CDCl_3): δ 194.8, 168.6, 168.1, 140.6, 128.5, 128.3, 126.3, 62.7, 54.2, 53.1, 52.9, 45.2, 32.4, 31.7, 14.0; **IR** (thin film, cm^{-1}): 2955, 1797, 1730, 1603, 1496, 1455, 1436, 1257, 1155, 1097, 1064, 752, 700; **TLC** (20% EtOAc/hexanes) R_f : 0.29; **LRMS** (ESI): Calculated for $[\text{M}+\text{H}]^+$ $\text{C}_{18}\text{H}_{23}\text{O}_7$: 351.14, Found: 351.16.

Preparation of *m*-Terphenyl Sulfonyl Chloride **S1**



To a flame-dried 50-mL round-bottomed flask equipped with a magnetic stir bar and rubber septum were added 1,3-dichlorobenzene (1.47 g, 10.0 mmol, 1.0 equiv) and THF (25 mL). The solution was cooled to $-78\text{ }^\circ\text{C}$ and *n*BuLi (1.6 M in hexanes, 11.0 mmol, 1.1 equiv) was added dropwise over 10 min. The resultant white slurry was stirred at $-78\text{ }^\circ\text{C}$ for 1.5 hrs. While being kept at $-78\text{ }^\circ\text{C}$, this reaction mixture was then added to a room temperature solution of phenylmagnesium bromide [prepared from bromobenzene (3.14 g, 20.0 mmol, 2.0 equiv) and magnesium (578 mg, 24.0 mmol, 2.4 equiv)] in 30 mL of THF via cannula. The mixture was heated at reflux overnight, cooled to ambient temperature, and then to $-78\text{ }^\circ\text{C}$. Sulfuryl chloride (1 M in CH_2Cl_2 , 20.0 mmol, 2.0 equiv) was added via syringe in a single portion and the reaction was warmed slowly to room temperature overnight. After being cooled to $0\text{ }^\circ\text{C}$, the reaction mixture was diluted with 1 M HCl and extracted three times with diethyl ether. The combined organic extracts were washed with brine, dried over sodium sulfate and concentrated *in vacuo*. The crude brown solid was recrystallized from hexanes and CHCl_3 to give the desired sulfonyl chloride (2.0 g, 6.1 mmol, 61% yield) whose spectral properties matched those reported in the literature.¹

Preparation of **L8**

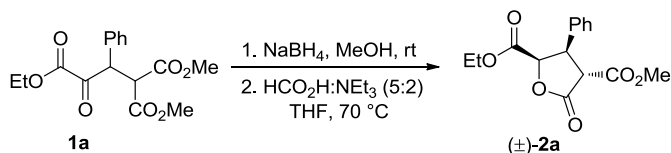


A 10-mL round-bottomed flask equipped with a magnetic stir bar was charged with (1*S*,2*S*)-1,2-di(1-naphthyl)-1,2-ethanediamine dihydrochloride (250 mg, 0.649 mmol, 1.0 equiv). Dichloromethane (3 mL) and 2 M NaOH (3 mL) were added sequentially and the biphasic mixture was cooled to $0\text{ }^\circ\text{C}$. *m*-Terphenyl sulfonyl chloride **S1** (213 mg, 0.649 mmol, 1.0 equiv) was added and the reaction was warmed to room temperature and stirred for 3 h. The reaction mixture was diluted with ethyl acetate and water. The organic was washed with brine, dried over sodium sulfate and concentrated *in vacuo*. Flash chromatography (50% EtOAc/hexanes) provided **L8** (330 mg, 0.545 mmol, 84% yield) as a white solid. Analytical data for **L8**: $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 8.07 (d, $J = 8.0\text{ Hz}$, 1H), 7.78 (d, $J = 8.0\text{ Hz}$, 1H), 7.74 (d, $J = 7.6\text{ Hz}$, 1H), 7.72-7.67 (m, 2H), 7.66-7.02 (m, 20H), 6.97 (bs, 1H), 6.72 (bs, 1H), 5.57 (d, $J = 6.4\text{ Hz}$, 1H), 4.95 (d, $J = 7.6\text{ Hz}$), 4.68 (bs, 1H), 1.75 (bs, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 141.9, 140.9, 139.7, 137.8, 135.3, 133.8, 133.6, 131.6, 131.0, 130.7, 130.0, 129.2, 128.8, 128.6, 128.2, 127.9, 127.7, 127.7, 126.1, 126.0, 125.4, 125.3, 125.1, 125.0, 124.3, 123.6, 122.9, 122.4; **IR** (thin film, cm^{-1}): 2926, 2357, 1868, 1716, 1608, 1541, 1507, 1456, 1338, 1158, 1028, 929, 778, 759, 700, 664, 592, 529; **m.p.** 158-160 $^\circ\text{C}$; **TLC** (50% EtOAc/hexanes) R_f :

¹ Kosugi, Y.; Akakura, M.; Ishihara, K. *Tetrahedron* **2007**, 63, 6191.

0.25; **LRMS** (ESI): Calculated for $[M+H]^+$ $C_{40}H_{33}N_2O_2S$: 605.23, Found: 605.29; $[\alpha]_D^{25} +170.6$ ($c = 1.30$, $CHCl_3$).

General Procedure B for the Preparation of Racemic γ -Butyrolactones 2a-2j



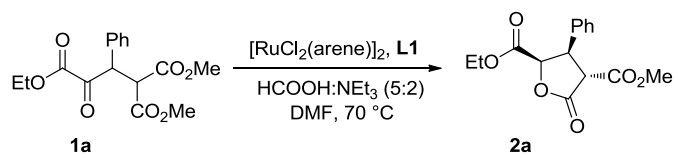
Sodium borohydride (1.0 equiv) was added to a solution of β -aryl α -keto ester (1.0 equiv) in methanol (0.5 M concentration) at room temperature. When gas evolution ceased, 1 mL saturated ammonium chloride was added. The reaction was further diluted with diethyl ether and water. The organic layer was washed with brine, dried over sodium sulfate and concentrated *in vacuo* to give a mixture of α -hydroxy ester and γ -butyrolactone. The crude reaction mixture was dissolved in tetrahydrofuran (0.5 M concentration) and formic acid:triethylamine 5:2 azeotrope (1.0 equiv) was added. The reaction heated at 70 °C for 1 hour, at which point the reaction mixture was allowed to cool to ambient temperature and diluted with diethyl ether and water. The organic layer was washed with brine and dried over sodium sulfate. Concentration *in vacuo* afforded the γ -butyrolactones which were purified by flash chromatography using the indicated solvent systems.

General Procedure C for the ATH-DKR of β -Aryl α -Keto Esters

To a flame-dried 1-dram vial equipped with a magnetic stir bar were added $[RuCl(arene)]_2$ (0.02 equiv) and ligand (0.08 equiv). The vial was sealed with a rubber septum and purged with nitrogen. DMF (0.5 mL) was added and the rubber septum was quickly replaced with a PTFE-lined screw cap. The mixture was heated at 70 °C for 30 min and cooled to ambient temperature. A solution of β -aryl α -keto ester (1.0 equiv in 1.0 mL DMF) followed by formic acid:triethylamine 5:2 azeotrope (5.0 equiv) were added. The vial was purged with nitrogen and the reaction was heated at 70 °C for 16 h, at which point the reaction mixture was allowed to cool to ambient temperature and diluted with ethyl acetate and water. The organic layer was washed with water (x2), brine, and dried over sodium sulfate. Concentration *in vacuo* afforded the γ -butyrolactones which were purified by flash chromatography using the indicated solvent systems.

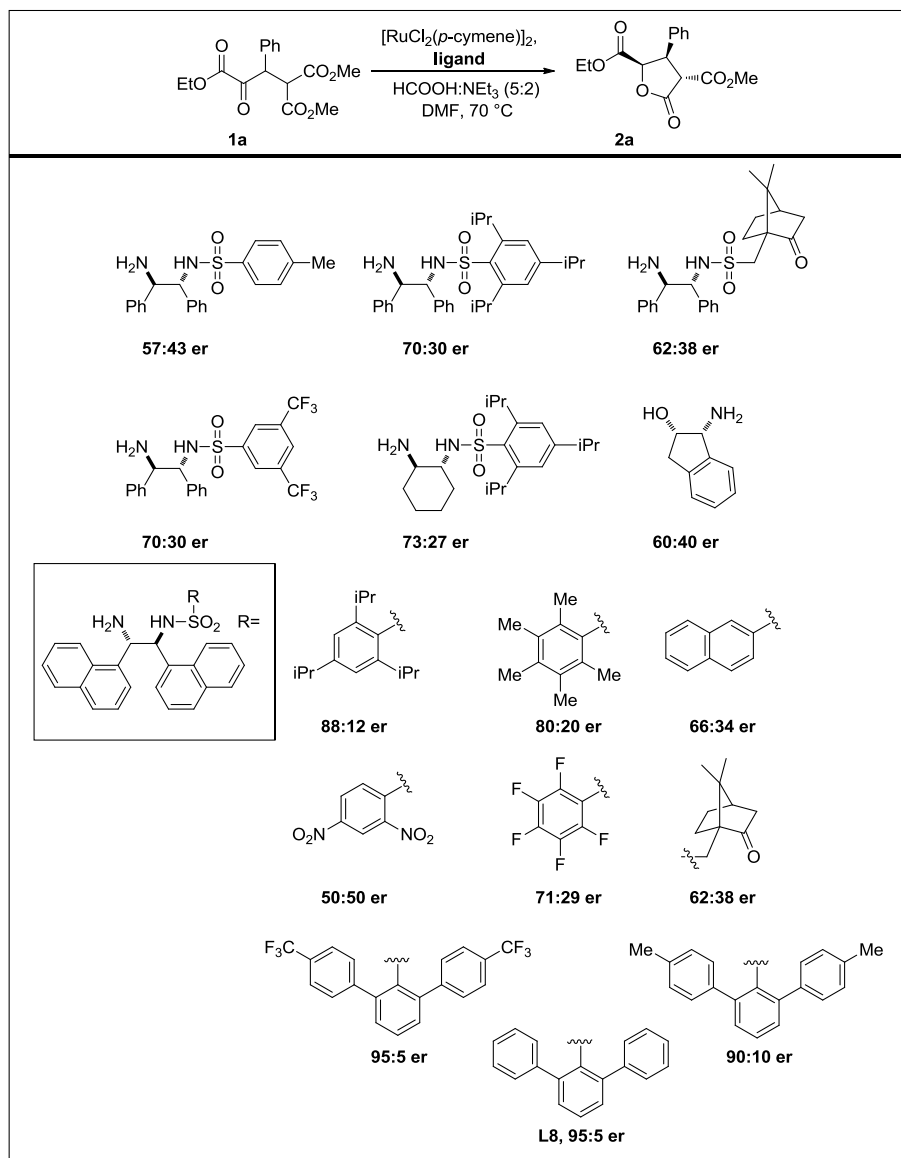
Catalyst Optimization Studies

Optimization of $[\text{RuCl}_2(\text{arene})]_2$ with 1a and L1

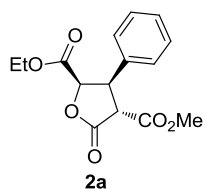


$[\text{RuCl}_2(\text{arene})]_2$	e.r.
$[\text{RuCl}_2(\text{hexamethylbenzene})]_2$	62:38
$[\text{RuCl}_2(p\text{-cymene})]_2$	70:30
$[\text{RuCl}_2(\text{benzene})]_2$	71:29

Full Ligand Screen

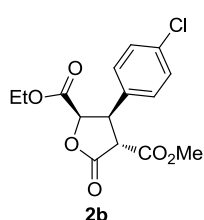


Procedures for Synthesis of γ -Butyrolactones 2a-2j



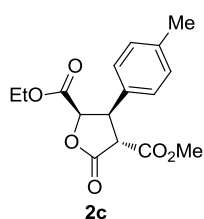
(2R,3R,4R)-2-ethyl 4-methyl 5-oxo-3-phenyltetrahydrofuran-2,4-dicarboxylate (2a): The title compound was prepared according to General Procedure C using β -aryl α -keto ester **1a** (50 mg, 0.155 mmol, 1.0 equiv), $[\text{RuCl}_2(p\text{-cymene})]_2$ (1.9 mg, 0.0031 mmol, 0.02 equiv), **L8** (7.5 mg, 0.0124 mmol, 0.08 equiv), and $\text{HCOOH}:\text{NEt}_3$ 5:2 azeotrope (67 mg, 0.775 mmol, 5.0 equiv).

Flash chromatography (20% EtOAc/hexanes) provided γ -butyrolactone **2a** (42.0 mg, 0.143 mmol, 92% yield) as a white solid. Analytical data for **2a**: $^1\text{H NMR}$ (600 MHz, CDCl_3): δ 7.35-7.32 (m, 3H), 7.22-7.20 (m, 2H), 5.18 (d, J = 8.4 Hz, 1H), 4.52 (dd, J = 12, 8.4 Hz, 1H), 4.25 (d, J = 12 Hz, 1H), 3.92-3.87 (m, 1H), 3.87 (s, 3H), 3.86-3.74 (m, 1H), 0.84 (t, J = 7.2 Hz, 3H); $^{13}\text{C NMR}$ (150 MHz, CDCl_3): δ 170.5, 167.7, 167.2, 132.6, 129.0, 128.7, 127.4, 78.9, 61.7, 53.4, 48.7, 47.8, 13.5; **IR** (thin film, cm^{-1}): 2963, 1797, 1742, 1455, 1437, 1382, 1280, 1218, 1133, 1075, 994, 940, 751, 699; **m.p.** 100-102 $^\circ\text{C}$; **TLC** (20% EtOAc/hexanes) R_f : 0.26; **LRMS** (ESI): Calculated for $[\text{M}+\text{H}]^+$ $\text{C}_{15}\text{H}_{17}\text{O}_6$: 293.10, Found: 293.16 **SFC Analysis**: WO column, 2% MeOH, 1.5 mL/min, 150 bar, 210 nm; t_{minor} = 12.5 min t_{major} = 14.5, 95:5 er; $[\alpha]_{\text{D}}^{25}$ -150.9 (c = 1.70, CHCl_3).



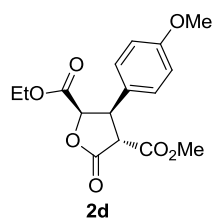
(2R,3R,4R)-2-ethyl 4-methyl 3-(4-chlorophenyl)-5-oxotetrahydrofuran-2,4-dicarboxylate (2b): The title compound was prepared according to General Procedure C using β -aryl α -keto ester **1b** (55 mg, 0.155 mmol, 1.0 equiv), $[\text{RuCl}_2(p\text{-cymene})]_2$ (1.9 mg, 0.0031 mmol, 0.02 equiv), **L8** (7.5 mg, 0.0124 mmol, 0.08 equiv), and $\text{HCOOH}:\text{NEt}_3$ 5:2 azeotrope (67 mg, 0.775 mmol, 5.0 equiv).

Flash chromatography provided γ -butyrolactone **2b** (47.6 mg, 0.146 mmol, 94% yield) as a white solid. Analytical data for **2b**: $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.34 (d, J = 8.8 Hz, 2H), 7.16 (d, J = 8.8 Hz, 2H), 5.16 (d, J = 8.4 Hz, 1H), 4.48 (dd, J = 11.2, 8.4 Hz, 1H), 4.18 (d, J = 11.2 Hz, 1H), 3.98-3.90 (m, 1H), 3.87-3.82 (m, 1H), 3.81 (s, 3H), 0.92 (t, J = 7.2 Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 170.0, 167.6, 167.0, 134.8, 131.3, 129.6, 129.2, 128.9, 128.5, 78.6, 61.9, 53.5, 48.9, 47.2, 13.6; **IR** (thin film, cm^{-1}): 2957, 1796, 1741, 1437, 1382, 1302, 1213, 1128, 1060, 840, 708, 564; **m.p.** 94-94 $^\circ\text{C}$; **TLC** (20% EtOAc/hexanes) R_f : 0.31; **LRMS** (ESI): Calculated for $[\text{M}+\text{H}]^+$ $\text{C}_{15}\text{H}_{16}\text{ClO}_6$: 327.06, Found: 327.10; **SFC Analysis**: WO column, 2% MeOH, 1.5 mL/min, 150 bar, 210 nm; t_{minor} = 13.4 min t_{major} = 17.3, 96:4 er; $[\alpha]_{\text{D}}^{25}$ -143.1 (c = 1.5, CHCl_3).



(2R,3R,4R)-2-ethyl 4-methyl 5-oxo-3-(p-tolyl)tetrahydrofuran-2,4-dicarboxylate (2c): The title compound was prepared according to General Procedure C using β -aryl α -keto ester **1c** (52 mg, 0.155 mmol, 1.0 equiv), $[\text{RuCl}_2(p\text{-cymene})]_2$ (1.9 mg, 0.0031 mmol, 0.02 equiv), **L8** (7.5 mg, 0.0124 mmol, 0.08 equiv), and $\text{HCOOH}:\text{NEt}_3$ 5:2 azeotrope (67 mg, 0.775 mmol, 5.0 equiv).

Flash chromatography provided γ -butyrolactone **2c** (40 mg, 0.130 mmol, 84% yield) as a white solid. Analytical data for **2c**: $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.15 (d, J = 8.0 Hz, 2H), 7.09 (d, J = 8.0 Hz, 2H), 5.15 (d, J = 8.8 Hz, 1H), 4.47 (dd, J = 11.2, 8 Hz, 1H), 4.21 (d, J = 11.2 Hz, 1H), 3.93-3.89 (m, 1H), 3.82-3.78 (m, 1H), 3.79 (s, 3H), 0.88 (t, J = 7.2 Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 170.5, 167.8, 167.3, 138.5, 129.6, 127.3, 79.0, 61.7, 53.3, 49.0, 47.6, 21.0, 13.5; **IR** (thin film, cm^{-1}): 2957, 1796, 1742, 1519, 1438, 1381, 1306, 1213, 1131, 1058, 828, 517; **m.p.** 85-86 $^\circ\text{C}$; **TLC** (30% EtOAc/hexanes) R_f : 0.38; **LRMS** (ESI): Calculated for $[\text{M}+\text{H}]^+$ $\text{C}_{16}\text{H}_{19}\text{O}_6$: 307.12, Found: 307.16; **HPLC Analysis**: Chiralpak IB, 10% i PrOH/hexanes, constant flow at 1.00 mL/min, 210 nm; t_{minor} = 12.5 min t_{major} = 15.8 min, 95.5:4.5 er; $[\alpha]_{\text{D}}^{25}$ -191.3 (c = 1.7, CHCl_3).

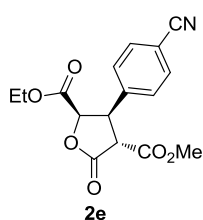


(2R,3R,4R)-2-ethyl 4-methyl 3-(4-methoxyphenyl)-5-oxotetrahydrofuran-2,4-dicarboxylate (2d):

The title compound was prepared according to General Procedure C using β -aryl α -keto ester **1d** (55 mg, 0.155 mmol, 1.0 equiv), $[\text{RuCl}_2(p\text{-cymene})]_2$ (1.9 mg, 0.0031 mmol, 0.02 equiv), **L8** (7.5 mg, 0.0124 mmol, 0.08 equiv), and $\text{HCOOH}:\text{NEt}_3$ 5:2 azeotrope (67 mg, 0.775 mmol, 5.0 equiv).

Flash chromatography provided γ -butyrolactone **2d** (45 mg, 0.140 mmol, 90% yield) as a white solid. Analytical data for **2d**: $^1\text{H NMR}$ (600 MHz, CDCl_3): δ 7.13

(d, J = 9.0 Hz, 2H), 6.87 (d, J = 9.0 Hz, 2H), 5.14 (d, J = 8.4 Hz, 1H), 4.45 (dd, J = 11.4, 8.4 Hz, 1H), 4.19 (d, J = 11.4 Hz, 1H), 3.95-3.80 (m, 1H), 3.84-3.8 (m, 1H), 3.79 (s, 3H), 3.78 (s, 3H), 0.93 (t, J = 7.2 Hz, 3H); $^{13}\text{C NMR}$ (150 MHz, CDCl_3): δ 170.5, 167.8, 167.3, 159.8, 128.6, 124.5, 114.3, 79.0, 61.7, 55.3, 53.4, 49.1, 47.2, 13.6; **IR** (thin film, cm^{-1}): 2958, 1796, 1742, 1613, 1518, 1440, 1382, 1256, 1218, 1182, 1132, 1096, 1058, 1030, 835, 750, 683; **m.p.** 106-107 $^\circ\text{C}$; **TLC** (30% EtOAc/hexanes) R_f : 0.31; **LRMS** (ESI): Calculated for $[\text{M}+\text{Na}]$ $\text{C}_{16}\text{H}_{18}\text{O}_7\text{Na}$: 345.10, Found: 345.18; **HPLC Analysis**: Chiralpak IB, 10% i PrOH/hexanes, constant flow at 1.00 mL/min, 210 nm; t_{minor} = 18.7 min t_{major} = 23.6 min, 95:5 er; $[\alpha]_{\text{D}}^{25}$ -179.2 (c = 1.9, CHCl_3).

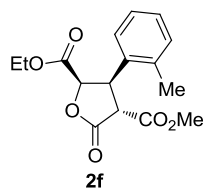


(2R,3R,4R)-2-ethyl 4-methyl 3-(4-cyanophenyl)-5-oxotetrahydrofuran-2,4-dicarboxylate (2e):

The title compound was prepared according to General Procedure C using β -aryl α -keto ester **1e** (54 mg, 0.155 mmol, 1.0 equiv), $[\text{RuCl}_2(p\text{-cymene})]_2$ (1.9 mg, 0.0031 mmol, 0.02 equiv), **L8** (7.5 mg, 0.0124 mmol, 0.08 equiv), and $\text{HCOOH}:\text{NEt}_3$ 5:2 azeotrope (67 mg, 0.775 mmol, 5.0 equiv).

Flash chromatography provided γ -butyrolactone **2d** (43 mg, 0.136 mmol, 88%

yield) as a white solid. Analytical data for **2e**: $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.67 (d, J = 8.0 Hz, 2H), 7.35 (d, J = 8.0 Hz, 2H), 5.21 (d, J = 8.8 Hz, 1H), 4.56 (dd, J = 11.2, 8.8 Hz, 1H), 4.21 (d, J = 11.2 Hz, 1H), 3.96-3.90 (m, 1H), 3.85-3.79 (m, 1H), 3.82 (s, 3H), 0.90 (t, J = 7.2 Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 169.4, 167.3, 166.7, 138.2, 132.7, 128.4, 117.9, 112.8, 78.1, 62.1, 53.6, 48.6, 47.6, 13.6; **IR** (thin film, cm^{-1}): 2360, 2333, 1796, 1742, 1312, 1271, 1134, 995, 836, 670; **m.p.** 104-107 $^\circ\text{C}$; **TLC** (30% EtOAc/hexanes) R_f : 0.25; **LRMS** (ESI): Calculated for $[\text{M}+\text{Na}]$ $\text{C}_{16}\text{H}_{15}\text{NO}_6\text{Na}$: 340.08, Found: 340.18; **HPLC Analysis**: Chiralpak IB, 25% i PrOH/hexanes, constant flow at 1.00 mL/min, 210 nm; t_{minor} = 18.6 min t_{major} = 22.4 min, 95:5 er; $[\alpha]_{\text{D}}^{25}$ -177.4 (c = 1.7, CHCl_3).

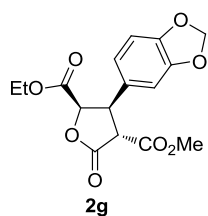


(2R,3R,4R)-2-ethyl 4-methyl 5-oxo-3-(o-tolyl)tetrahydrofuran-2,4-dicarboxylate (2f):

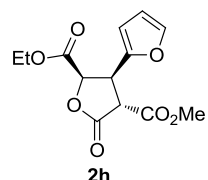
The title compound was prepared according to General Procedure C using β -aryl α -keto ester **1f** (52 mg, 0.155 mmol, 1.0 equiv), $[\text{RuCl}_2(p\text{-cymene})]_2$ (1.9 mg, 0.0031 mmol, 0.02 equiv), **L8** (7.5 mg, 0.0124 mmol, 0.08 equiv), and $\text{HCOOH}:\text{NEt}_3$ 5:2 azeotrope (67 mg, 0.775 mmol, 5.0 equiv).

Flash chromatography provided γ -butyrolactone **2d** (39 mg, 0.127 mmol, 82%

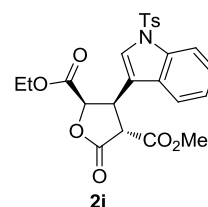
yield) as a white solid. Analytical data for **2f**: $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.23-7.21 (m, 2H), 7.19-7.16 (m, 1H), 7.09-7.07 (m, 1H), 5.24 (d, J = 8.8 Hz, 1H), 4.72 (dd, J = 12, 8.8 Hz, 1H), 4.34 (d, J = 12 Hz, 1H), 3.86-3.81 (m, 1H), 3.79 (s, 3H), 3.74-3.7 (m, 1H), 0.80 (t, J = 7.2 Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 170.5, 167.7, 167.3, 137.4, 131.0, 130.7, 128.5, 126.5, 125.0, 77.3, 61.6, 53.3, 48.3, 44.8, 19.6, 13.4; **IR** (thin film, cm^{-1}): 2359, 2341, 1797, 1742, 1440, 1381, 1317, 1212, 1135, 1021, 752, 538; **m.p.** 103-104 $^\circ\text{C}$; **TLC** (20% EtOAc/hexanes) R_f : 0.29; **LRMS** (ESI): Calculated for $[\text{M}+\text{H}]^+$ $\text{C}_{16}\text{H}_{19}\text{O}_6$: 307.12, Found: 307.16; **HPLC Analysis**: Chiralpak IB, 10% i PrOH/hexanes, constant flow at 1.00 mL/min, 210 nm; t_{minor} = 12.1 min t_{major} = 15.0 min, 89:11 er; $[\alpha]_{\text{D}}^{25}$ -136.4 (c = 1.4, CHCl_3).



(2R,3R,4R)-2-ethyl 4-methyl 3-(benzo[d][1,3]dioxol-5-yl)-5-oxotetrahydrofuran-2,4-dicarboxylate (2g): The title compound was prepared according to General Procedure C using β -aryl α -keto ester **1g** (57 mg, 0.155 mmol, 1.0 equiv), $[\text{RuCl}_2(p\text{-cymene})]_2$ (1.9 mg, 0.0031 mmol, 0.02 equiv), **L8** (7.5 mg, 0.0124 mmol, 0.08 equiv), and $\text{HCOOH}:\text{NEt}_3$ 5:2 azeotrope (67 mg, 0.775 mmol, 5.0 equiv). Flash chromatography provided γ -butyrolactone **2d** (43 mg, 0.129 mmol, 83% yield) as a white solid. Analytical data for **2g**: $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 6.77 (d, J = 8.4 Hz, 1H), 6.68-6.67 (m, 2H), 5.96 (s, 2H), 5.12 (d, J = 8.4 Hz, 1H), 4.41 (dd, J = 11.6, 8.4 Hz, 1H), 4.14 (d, J = 11.6 Hz, 1H), 4.05-3.87 (m, 2H), 3.80 (s, 3H), 0.98 (t, J = 7.2 Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 170.3, 167.7, 167.2, 148.2, 147.9, 126.2, 120.9, 108.6, 107.8, 101.4, 78.9, 61.8, 53.3, 49.2, 47.7, 13.7; **IR** (thin film, cm^{-1}): 2906, 2363, 2349, 1797, 1740, 1506, 1494, 1446, 1304, 1256, 1237, 1142, 1093, 1037, 931, 801; **m.p.** 86-87 $^\circ\text{C}$; **TLC** (30% EtOAc/hexanes) R_f : 0.31; **LRMS** (ESI): Calculated for $[\text{M}+\text{H}]^+$ $\text{C}_{16}\text{H}_{17}\text{O}_8$: 337.09, Found: 337.09; **HPLC Analysis**: Chiralpak IB, 10% i -PrOH/hexanes, constant flow at 1.00 mL/min, 210 nm; t_{minor} = 21.8 min t_{major} = 25.8 min, 96:4 er; $[\alpha]_{\text{D}}^{25}$ -206.3 (c = 2.1, CHCl_3).

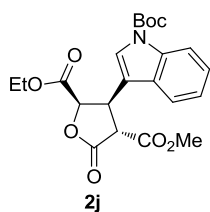


(2R,3S,4R)-2-ethyl 4-methyl 3-(furan-2-yl)-5-oxotetrahydrofuran-2,4-dicarboxylate (2h): The title compound was prepared according to General Procedure C using β -aryl α -keto ester **1h** (48 mg, 0.155 mmol, 1.0 equiv), $[\text{RuCl}_2(p\text{-cymene})]_2$ (1.9 mg, 0.0031 mmol, 0.02 equiv), **L8** (7.5 mg, 0.0124 mmol, 0.08 equiv), and $\text{HCOOH}:\text{NEt}_3$ 5:2 azeotrope (67 mg, 0.775 mmol, 5.0 equiv). Flash chromatography provided γ -butyrolactone **2h** (40 mg, 0.141 mmol, 91% yield) as a clear oil. Analytical data for **2h**: $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.38-7.37 (m, 1H), 6.34-6.33 (m, 1H), 6.28-6.27 (m, 1H), 5.14 (d, J = 8.4 Hz, 1H), 4.56 (dd, J = 11.6, 8.4 Hz, 1H), 4.16 (d, J = 11.6, 1H), 4.10-4.04 (m, 1H), 3.99-3.92 (m, 1H), 3.82 (s, 3H), 1.09 (t, J = 7.2 Hz, 3H); $^{13}\text{C NMR}$ (150 MHz, CDCl_3): δ 169.6, 167.6, 166.8, 146.7, 143.2, 110.7, 109.0, 77.2, 62.2, 53.5, 48.2, 41.9, 13.7; **IR** (thin film, cm^{-1}): 2963, 1797, 1743, 1440, 1382, 1305, 1215, 1133, 1061, 841, 708, 560; **TLC** (20% EtOAc/hexanes) R_f : 0.21; **LRMS** (ESI): Calculated for $[\text{M}+\text{H}]^+$ $\text{C}_{13}\text{H}_{15}\text{O}_7$: 283.08, Found: 283.03; **HPLC Analysis**: Chiralpak IA, 10% i -PrOH/hexanes, constant flow at 1.00 mL/min, 210 nm; t_{minor} = 11.4 min t_{major} = 12.5 min, 95:5 er; $[\alpha]_{\text{D}}^{25}$ -52.3 (c = 1.2, CHCl_3).



(2R,3R,4R)-2-ethyl 4-methyl 5-oxo-3-(1-tosyl-1H-indol-3-yl)tetrahydrofuran-2,4-dicarboxylate (2i): The title compound was prepared according to General Procedure C using β -aryl α -keto ester **1i** (80 mg, 0.155 mmol, 1.0 equiv), $[\text{RuCl}_2(p\text{-cymene})]_2$ (1.9 mg, 0.0031 mmol, 0.02 equiv), **L8** (7.5 mg, 0.0124 mmol, 0.08 equiv), and $\text{HCOOH}:\text{NEt}_3$ 5:2 azeotrope (67 mg, 0.775 mmol, 5.0 equiv). Flash chromatography provided γ -butyrolactone **2h** (68 mg, 0.141 mmol, 91% yield) as a white solid. Analytical data for **2i**: $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.99 (d, J = 8.4 Hz, 1H), 7.76 (d, J = 8.4 Hz, 2H), 7.52 (d, J = 7.6 Hz, 1H), 7.42 (s, 1H), 7.37-7.35 (m, 1H), 7.32-7.30 (m, 1H), 7.54-7.23 (m, 1H), 5.30 (d, J = 8.4 Hz, 1H), 4.64 (dd, J = 11.6, 8.4 Hz, 1H), 4.20 (d, J = 11.6 Hz, 1H), 3.82 (s, 3H), 3.64-3.56 (m, 1H), 3.36-3.28 (m, 1H), 2.35 (s, 3H), 0.41 (t, J = 6.8 Hz, 3H); $^{13}\text{C NMR}$ (150 MHz, CDCl_3): δ 169.8, 167.6, 167.0, 145.3, 135.0, 134.8, 130.0, 129.2, 126.9, 125.6, 123.6, 123.4, 119.5, 115.5, 113.7, 77.6, 61.4, 53.5, 48.4, 39.9, 30.2, 21.5, 12.9; **IR** (thin film, cm^{-1}): 2978, 2348, 1800, 1742, 1595, 1449, 1373, 1290, 1215, 1175, 1139, 1092, 1075, 1021, 974, 912, 816, 762, 747, 703, 680, 655; **m.p.** 99-100 $^\circ\text{C}$; **TLC** (30% EtOAc/hexanes) R_f : 0.28; **LRMS** (ESI): Calculated for $[\text{M}+\text{Na}]$ $\text{C}_{24}\text{H}_{23}\text{NO}_8\text{SNa}$: 508.10, Found: 508.10; **SFC Analysis**: WO column, 10%

MeOH, 1.5 mL/min, 150 bar 210 nm; $t_{\text{minor}} = 16.5$ min $t_{\text{major}} = 22.5$ min, 96.5:3.5 er; $[\alpha]_{\text{D}}^{25} -81.2$ ($c = 1.7$, CHCl_3).

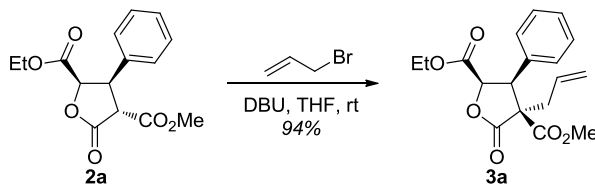


(2R,3R,4R)-2-ethyl 4-methyl 3-(1-(tert-butoxycarbonyl)-1H-indol-3-yl)-5-oxotetrahydrofuran-2,4-dicarboxylate (2j): The title compound was prepared according to General Procedure C using β -aryl α -keto ester **1j** (71.5 mg, 0.155 mmol, 1.0 equiv), $[\text{RuCl}_2(p\text{-cymene})]_2$ (1.9 mg, 0.0031 mmol, 0.02 equiv), **L8** (7.5 mg, 0.0124 mmol, 0.08 equiv), and $\text{HCOOH}:\text{NEt}_3$ 5:2 azeotrope (67 mg, 0.775 mmol, 5.0 equiv). Flash chromatography provided γ -butyrolactone **2h** (59 mg, 0.136 mmol, 88% yield) as a white solid. Analytical data for **2j**: $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 8.11 (d, $J = 8.4$ Hz, 1H), 7.57-7.55 (m, 1H), 7.44 (s, 1H), 7.37-7.30 (m, 2H), 5.35 (d, $J = 8.4$ Hz, 1H), 4.70 (dd, $J = 11.6, 8.4$ Hz, 1H), 4.24 (d, $J = 11.6$, 1H), 3.85-3.80 (m, 2H), 3.82 (s, 3H), 0.69 (t, $J = 6.8$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 170.1, 167.9, 167.2, 149.2, 135.4, 129.0, 125.3, 123.2, 123.0, 118.9, 115.4, 113.5, 84.5, 77.9, 61.7, 53.4, 48.8, 40.0, 28.1, 13.2; **IR** (thin film, cm^{-1}): 2979, 1800, 1739, 1454, 1375, 1338, 1310, 1273, 1258, 1220, 1155, 1078, 1018, 857, 840, 763, 748; **m.p.** 84-85 $^\circ\text{C}$; **TLC** (30% EtOAc/hexanes): R_f : 0.4; **LRMS** (ESI): Calculated for $[\text{M}+\text{H}]^+$ $\text{C}_{22}\text{H}_{26}\text{NO}_8$: 432.17, Found: 432.18; **HPLC Analysis**: Chiralpak IB, 10% i -PrOH/hexanes, constant flow at 1.0 mL/min, 210 nm; $t_{\text{minor}} = 11.7$ min $t_{\text{major}} = 12.6$ min, 96:4 er; $[\alpha]_{\text{D}}^{25} -75.7$ ($c = 2.3$, CHCl_3).

Large Scale Preparation of γ -Butyrolactone 2g

To a flame-dried 250-mL round-bottomed flask were added $[\text{RuCl}_2(p\text{-cymene})]_2$ (83.6 mg, 0.137 mmol, 0.005 equiv), **L8** (330 mg, 0.546 mmol, 0.020 equiv) and 50 mL of DMF. The mixture was heated at 70 $^\circ\text{C}$ under nitrogen for 45 min and cooled to ambient temperature. A solution of β -aryl α -keto ester **1g** (10.0 g, 27.3 mmol, 1.0 equiv) in 20 mL DMF was added via syringe and the reaction mixture was further diluted with 66 mL DMF. Formic acid:triethylamine 5:2 azeotrope (11.8 g, 136.5 mmol, 5.0 equiv) was added in a single portion and the reaction was heated at 70 $^\circ\text{C}$ for 16 h, at which point the reaction was cooled to ambient temperature and diluted with ethyl acetate and water. The aqueous layer was extracted with ethyl acetate (x2) and the combined organics were washed with water (x3) followed by brine (x1), dried over sodium sulfate and concentrated *in vacuo*. A 150-mL frit funnel (7 cm diameter) was charged with silica gel (4 cm in height). The crude reaction mixture was dissolved in the minimum amount of dichloromethane and passed through the pad of silica gel using 150 mL of 30% EtOAc/hexanes; the filtrate was concentrated *in vacuo*. The resultant residue was dissolved in the minimum amount of diethyl ether. Hexanes were added until the solution became cloudy. This solution was stored in the freezer overnight. The crystals were collected by filtration to yield enantiopure **1g** (6.61 g, 19.6 mmol, 72% yield).

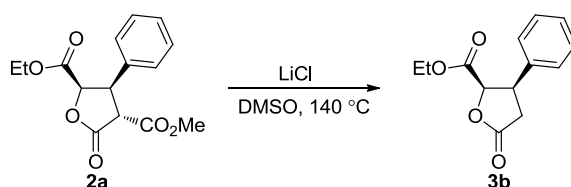
Preparation of γ -Butyrolactone 3a



To a 1-dram vial equipped with a magnetic stir bar were added **2a** (29.2 mg, 0.100 mmol, 1.0 equiv), allyl bromide (24.2 mg, 0.200 mmol, 2.0 equiv) and THF (1 mL). DBU (30.4 mg, 0.200 mmol, 2.0

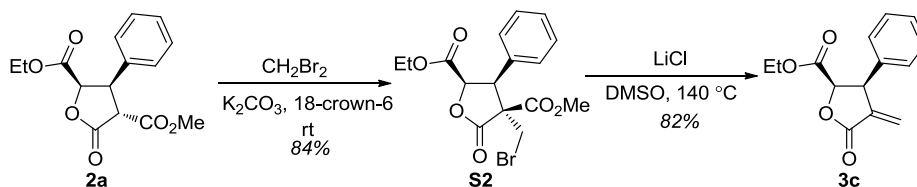
equiv) was added and the reaction was stirred at room temperature for 2 h and then diluted with water and ethyl acetate. The organic was washed with water followed by brine, dried over sodium sulfate and concentrated *in vacuo*. Flash chromatography provided **3a** (31.2 mg, 0.940 mmol, 94% yield) as a white solid. Analytical data for **3a**: $^1\text{H NMR}$ (600 MHz, CDCl_3): δ 7.25-7.24 (m, 3H), 7.15-7.13 (m, 2H), 5.90-5.85 (m, 1H), 5.35-5.30 (m, 2H), 5.22 (d, $J = 7.2$ Hz, 1H), 3.91-3.81 (m, 2H), 3.89 (d, $J = 7.2$ Hz, 1H), 3.22 (s, 3H), 2.97 (dd, $J = 14.1, 7.2$ Hz, 1H), 2.85 (dd, $J = 14.1, 7.2$ Hz, 1H), 0.83 (t, $J = 7.2$ Hz, 3H); $^{13}\text{C NMR}$ (150 MHz, CDCl_3): δ 171.6, 167.6, 166.3, 134.8, 130.5, 128.6, 128.4, 128.2, 121.5, 77.3, 61.6, 60.4, 53.6, 52.1, 40.1, 13.5; **IR** (thin film, cm^{-1}): 2921, 2359, 1797, 1759, 1740, 1436, 1221, 1168, 1125, 1087, 1028, 929, 705; **m.p.** 100-101.5 $^\circ\text{C}$; **TLC** (20% EtOAc/hexanes) R_f : 0.22; **LRMS** (ESI): Calculated for $[\text{M}+\text{H}]^+$ $\text{C}_{18}\text{H}_{21}\text{O}_6$: 333.13, Found: 333.16; $[\alpha]_D^{25}$ -35.5 ($c=6.5$, CHCl_3).

Preparation of γ -Butyrolactone **3b**



To a 1-dram vial equipped with a magnetic stir bar were added **2a** (45.0 mg, 0.155 mmol, 1.0 equiv), lithium chloride (71.0 mg, 0.310 mmol, 2.0 equiv), and DMSO (1 mL). The vial was fitted with a PTFE screw-cap and heated in an oil bath at 140 $^\circ\text{C}$ for 16 h, at which point the reaction was cooled to ambient temperature and diluted with water and ethyl acetate. The organic layer was washed with water (x2) followed by brine (x1), dried over sodium sulfate and concentrated *in vacuo*. Flash chromatography (20% EtOAc/hexanes) provided **3b** (31.2 mg, 0.133 mmol, 86% yield) as a colorless oil. Analytical data for **3b**: $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.36-7.30 (m, 3H), 7.22-7.20 (m, 2H), 5.13 (d, $J = 8.4$ Hz, 1H), 4.13-4.06 (m, 1H), 3.91-3.83 (m, 1H), 3.80-3.72 (m, 1H), 3.09 (dd, $J = 17.2, 10$ Hz, 1H), 2.84 (dd, $J = 17.2, 8.8$ Hz, 1H), 0.86 (t, $J = 7.2$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 175.2, 167.9, 134.9, 128.8, 128.3, 127.5, 80.1, 61.4, 44.1, 32.2, 13.5; **IR** (thin film, cm^{-1}): 2985, 2359, 2342, 1793, 1742, 1498, 1455, 1381, 1214, 1151, 1094, 1074, 1051, 700, 544; **TLC** (20% EtOAc/hexanes) R_f : 0.19; **LRMS** (ESI): Calculated for $[\text{M}+\text{H}]^+$ $\text{C}_{13}\text{H}_{15}\text{O}_4$: 235.10, Found: 235.08; $[\alpha]_D^{25}$ -68.4 ($c=1.6$, CHCl_3).

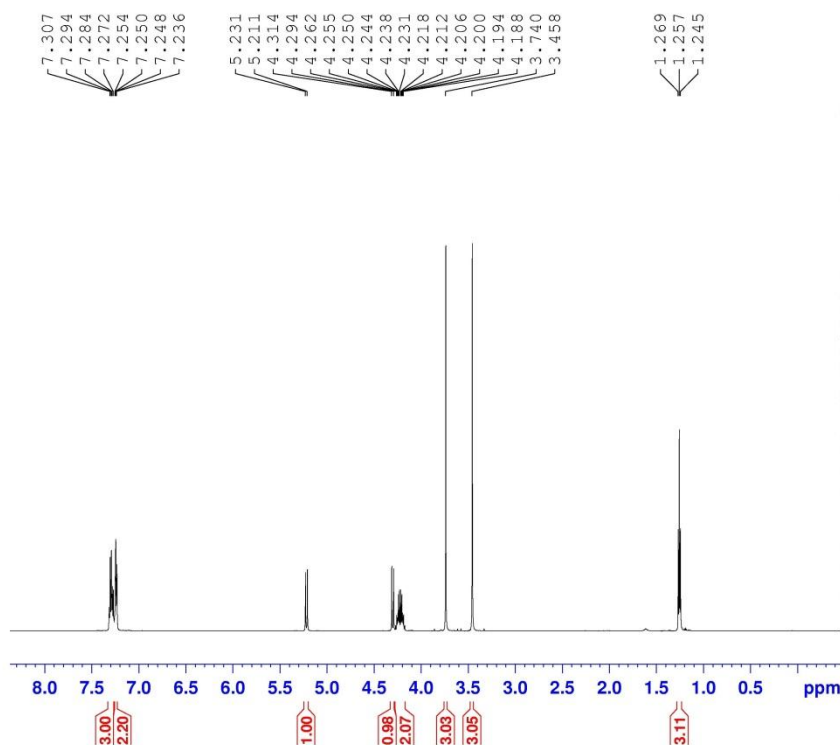
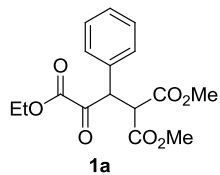
Preparation of α -methylene γ -Butyrolactone **3c**



To a flame-dried 1-dram vial equipped with a magnetic stir bar were added **2a** (29.2 mg, 0.100 mmol, 1.0 equiv) and dibromomethane (0.125 mL). 18-crown-6 (1.30 mg, 0.005 mmol, 0.05 equiv) and potassium carbonate (35 mg, 0.25 mmol, 2.5 equiv) were added sequentially and the reaction was stirred at ambient temperature for 36 h. The reaction was diluted with water and ethyl acetate and the organic was washed with water followed by brine, dried over sodium sulfate and

concentrated *in vacuo*. Flash chromatography (30% EtOAc/hexanes) provided **S2** (32 mg, 0.084 mmol, 84% yield) as a white solid. Analytical data for **S2**: $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.28-7.26 (m, 3H), 7.17-7.15 (m, 2H), 5.42 (d, $J = 8.4$ Hz, 1H), 4.20 (d, $J = 8.4$ Hz, 1H), 4.01 (d, $J = 10.8$ Hz, 1H), 3.94 (d, $J = 10.8$ Hz, 1H), 3.92-3.86 (m, 2H), 3.30 (s, 3H), 0.84 (t, $J = 7.2$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 170.3, 166.2, 134.2, 128.9, 128.7, 128.3, 78.2, 61.8, 61.6, 52.8, 52.7, 33.2, 13.5; **IR** (thin film, cm^{-1}): 2360, 1795, 1751, 1736, 1232, 1146, 1086, 1041, 709; **m.p.** 161-162 °C; **TLC** (30% EtOAc/hexanes) R_f : 0.32; **LRMS** (ESI): Calculated for $[\text{M}+\text{Na}]$ $\text{C}_{16}\text{H}_{17}\text{BrO}_6\text{Na}$: 407.01, Found: 407.01; $[\alpha]_D^{25} = -37.3$ ($c = 2.0$, CHCl_3).

To a 1-dram vial equipped with a magnetic stir bar were added **S2** (26 mg, 0.067 mmol, 1.0 equiv), lithium chloride (6.0 mg, 0.134 mmol, 2.0 equiv) and DMSO (1 mL). The vial was fitted with a PTFE screw-cap and heated in an oil bath at 140 °C for 16 h, at which point the reaction was cooled to ambient temperature and diluted with water and ethyl acetate. The organic was washed with water (x2) followed by brine (x1), dried over sodium sulfate and concentrated *in vacuo*. Flash chromatography (20% EtOAc/hexanes) provided **3c** (14 mg, 0.057 mmol, 85% yield) as a white solid. Analytical data for **3c**: $^1\text{H NMR}$ (600 MHz, CDCl_3): δ 7.33-7.30 (m, 3H), 7.21-7.19 (m, 2H), 6.54 (d, $J = 3$ Hz, 1H), 5.63 (d, $J = 3$ Hz, 1H), 5.18 (d, $J = 9$ Hz, 1H), 4.62-4.59 (dt, $J = 9.6, 3$ Hz, 1H), 3.85-3.80 (m, 1H), 3.68-3.63 (m, 1H), 0.84 (t, $J = 7.2$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 169.3, 167.7, 135.8, 135.3, 129.0, 128.8, 128.4, 125.1, 78.4, 61.5, 48.6, 13.5; **IR** (thin film, cm^{-1}): 2983, 2359, 1780, 1747, 1402, 1188, 1095, 1067, 757, 702; **m.p.** 113-114 °C; **TLC** (20% EtOAc/hexanes) R_f : 0.25; **LRMS** (ESI): Calculated for $[\text{M}+\text{H}]^+$ $\text{C}_{14}\text{H}_{15}\text{O}_4$: 247.10, Found: 247.12; $[\alpha]_D^{25} = -62.3$ ($c = 2.2$, CHCl_3).

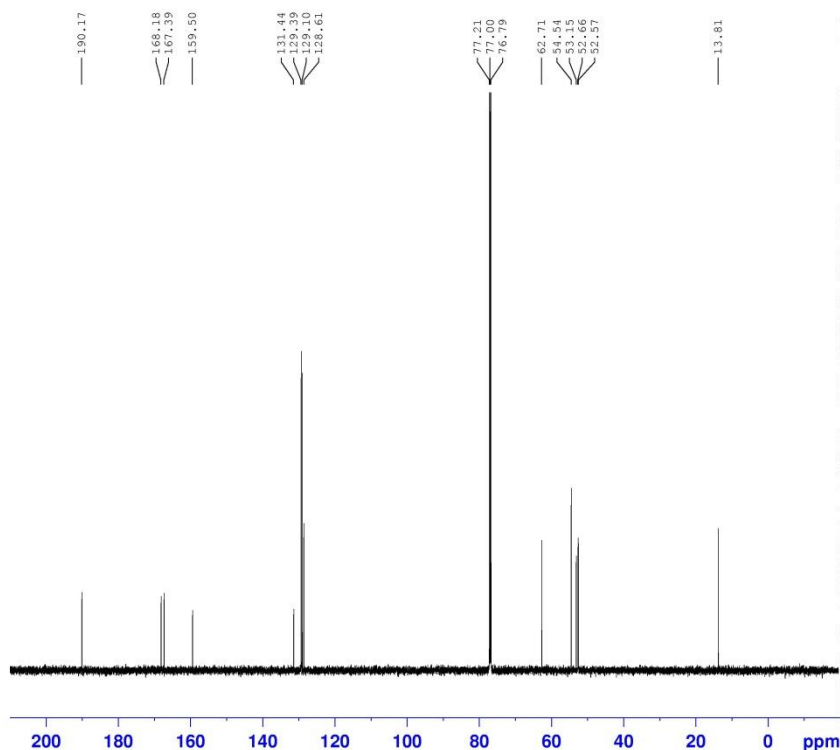


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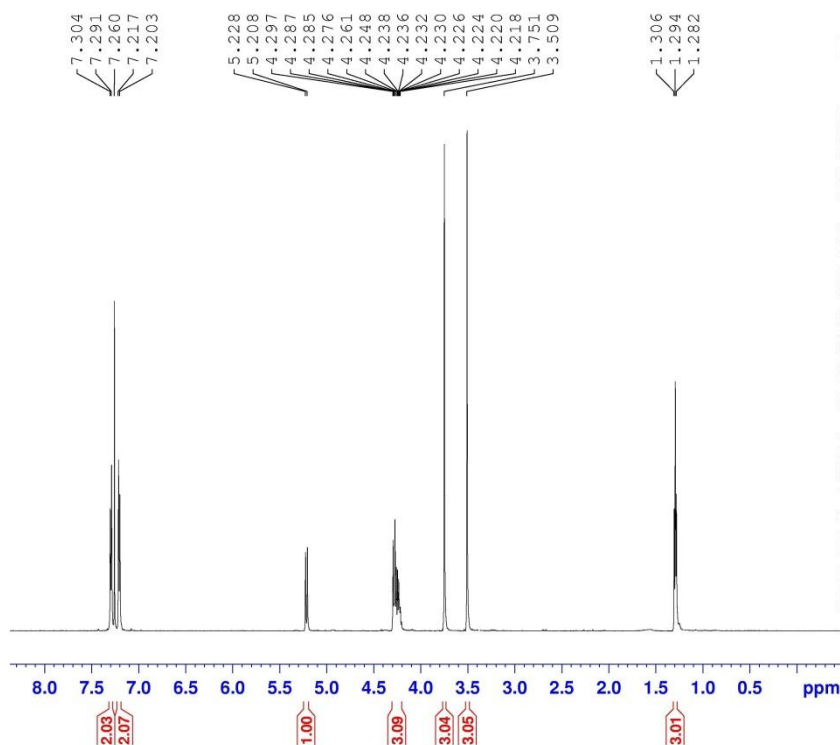
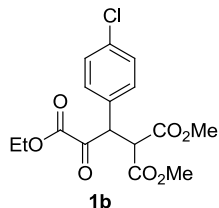
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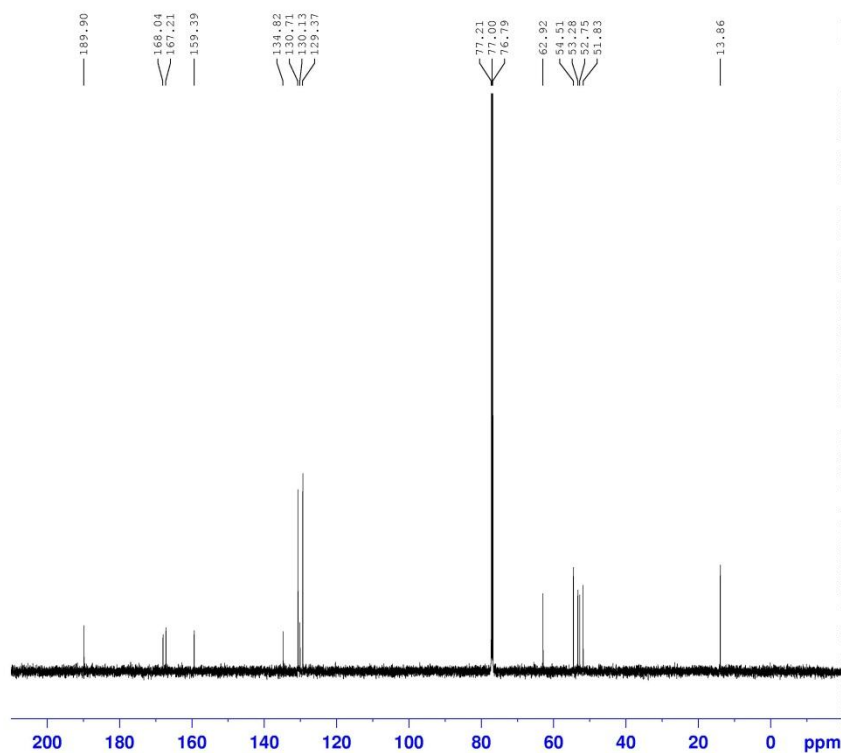


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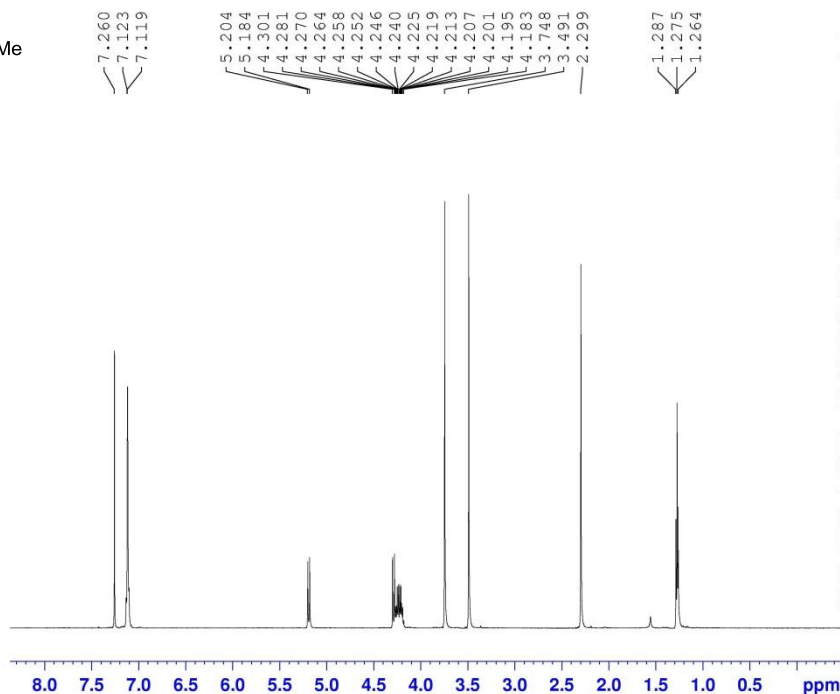
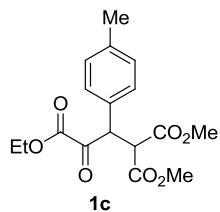
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DE 10.00 usec
TE 294.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

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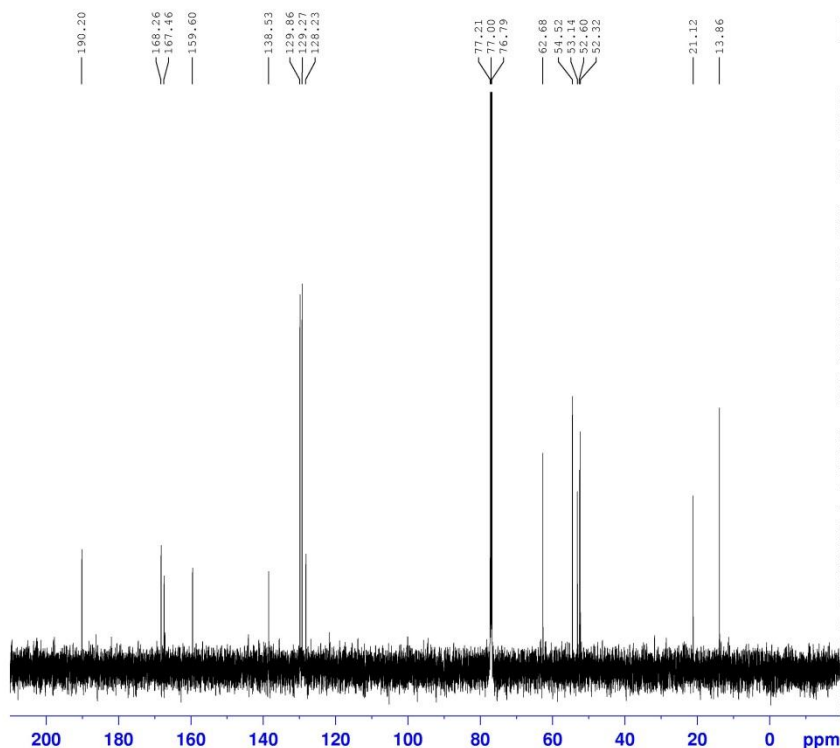


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TE 292.8 K
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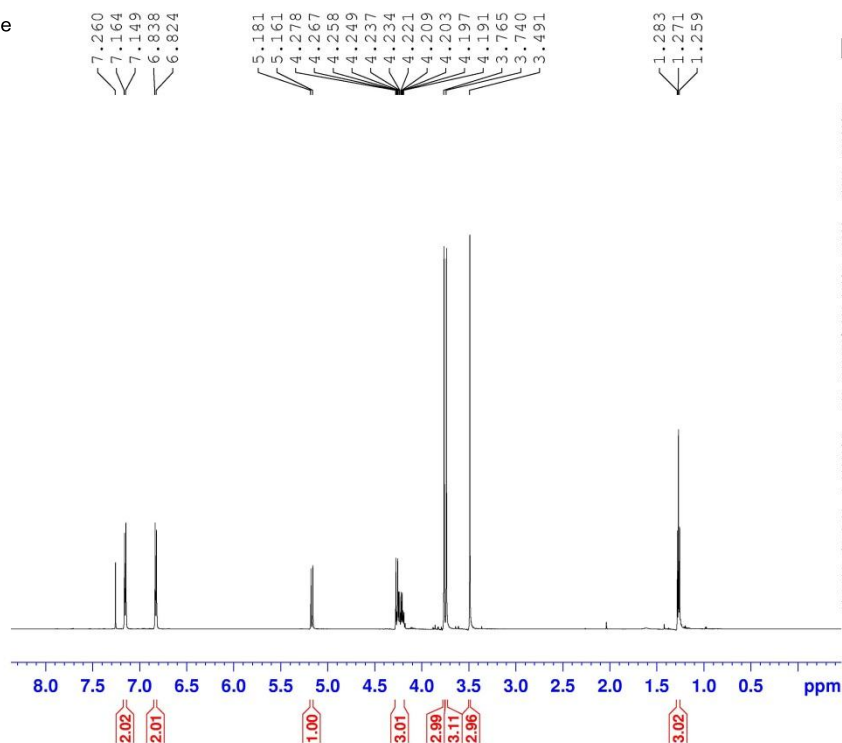
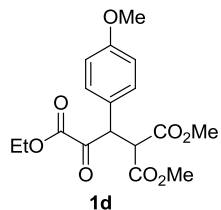
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D11 0.03000000 sec
TD0 1

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PLW1 110.00000000 W
SFO1 150.9178981 MHz

===== CHANNEL f2 =====
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NUC2 1H
PCPD2 70.00 usec
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F2 - Processing parameters
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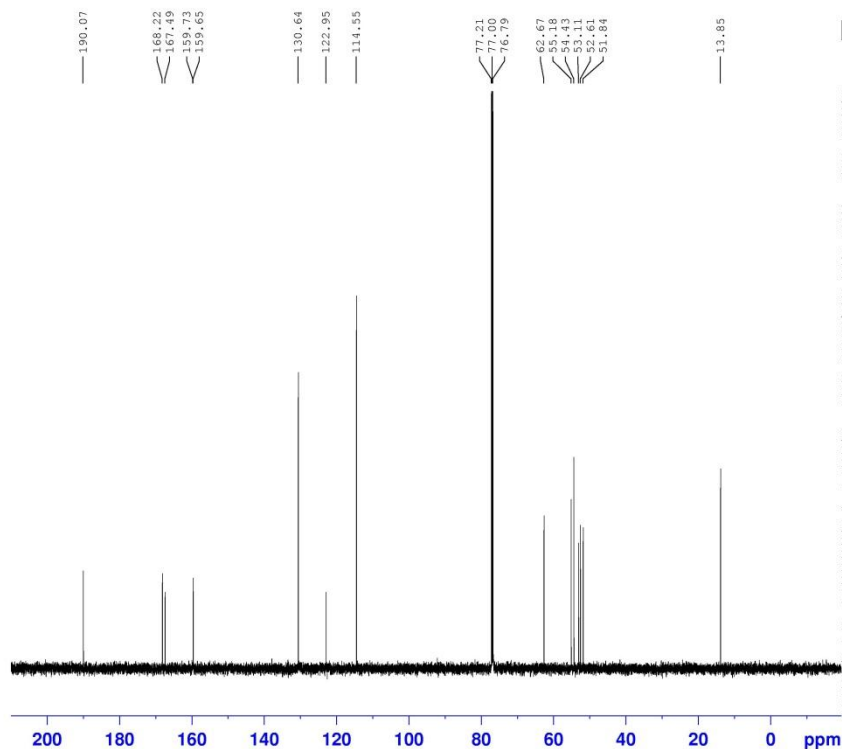


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SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 161
DW 40.533 usec
DE 6.50 usec
TE 292.9 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.60 usec
PLW1 20.00000000 W
SFO1 600.1337060 MHz

F2 - Processing parameters
SI 65536
SF 600.1300178 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



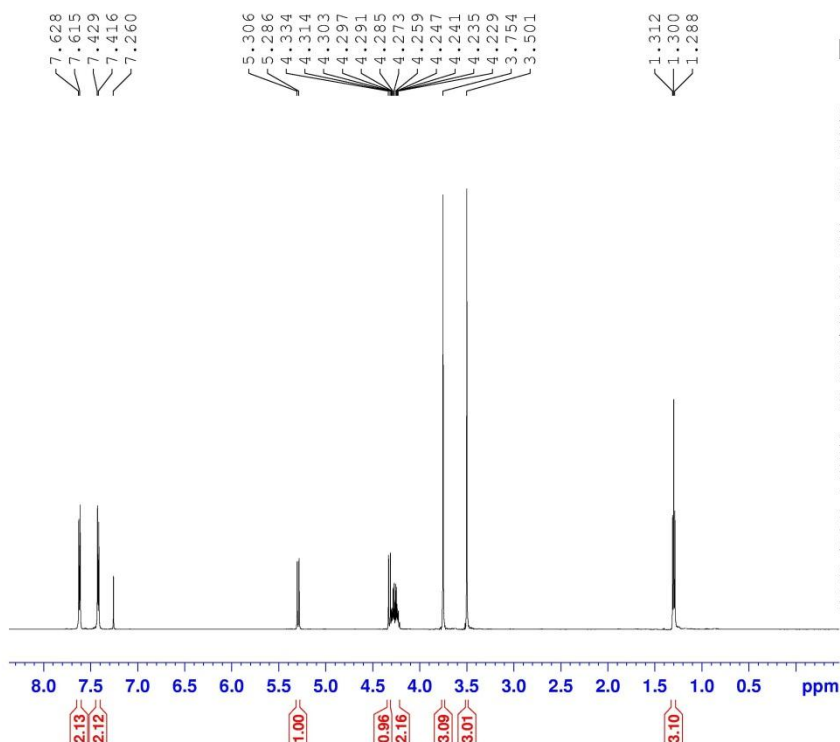
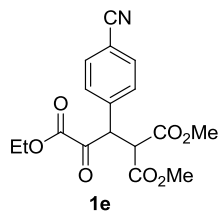
Current Data Parameters
NAME 6KMS107
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111210
Time 15.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 123
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 203
DW 13.867 usec
DE 10.00 usec
TE 294.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PLW1 110.00000000 W
SFO1 150.9178981 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PLW2 20.00000000 W
PLW12 0.80000001 W
PLW13 0.39199999 W
SFO2 600.1324005 MHz

F2 - Processing parameters
SI 32768
SF 150.9028162 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

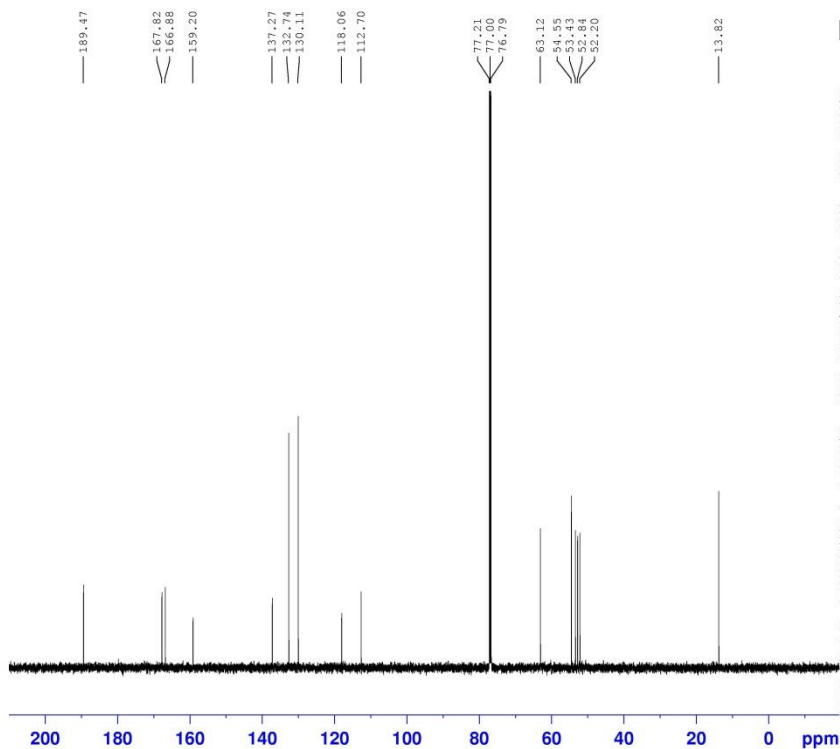


Current Data Parameters
NAME 6KMS204
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111210
Time 15.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 181
DW 40.533 usec
DE 6.50 usec
TE 292.6 K
D1 1.00000000 sec
TDO 1

CHANNEL f1
NUC1 1H
P1 13.60 usec
PLW1 20.00000000 W
SFO1 600.1337060 MHz

F2 - Processing parameters
SI 65536
SF 600.1300177 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



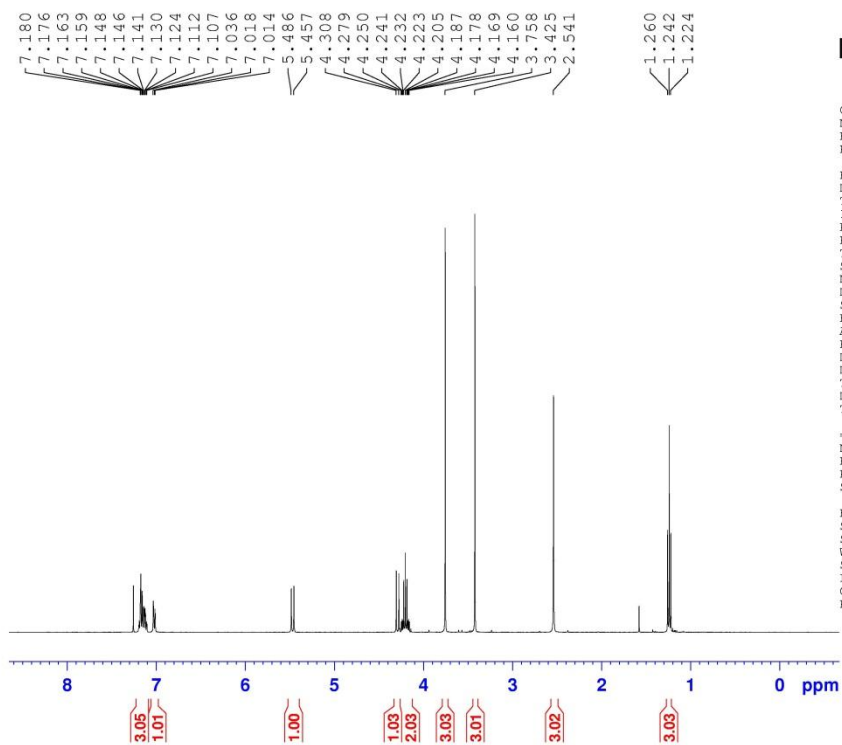
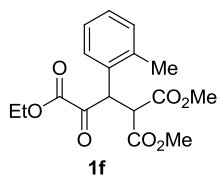
Current Data Parameters
NAME 6KMS204
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111210
Time 15.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 139
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 203
DW 13.867 usec
DE 10.00 usec
TE 294.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

CHANNEL f1
NUC1 13C
P1 10.50 usec
PLW1 110.00000000 W
SFO1 150.9178981 MHz

CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PLW2 20.00000000 W
PLW12 0.80000001 W
PLW13 0.39199999 W
SFO2 600.1324005 MHz

F2 - Processing parameters
SI 32768
SF 150.9028183 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

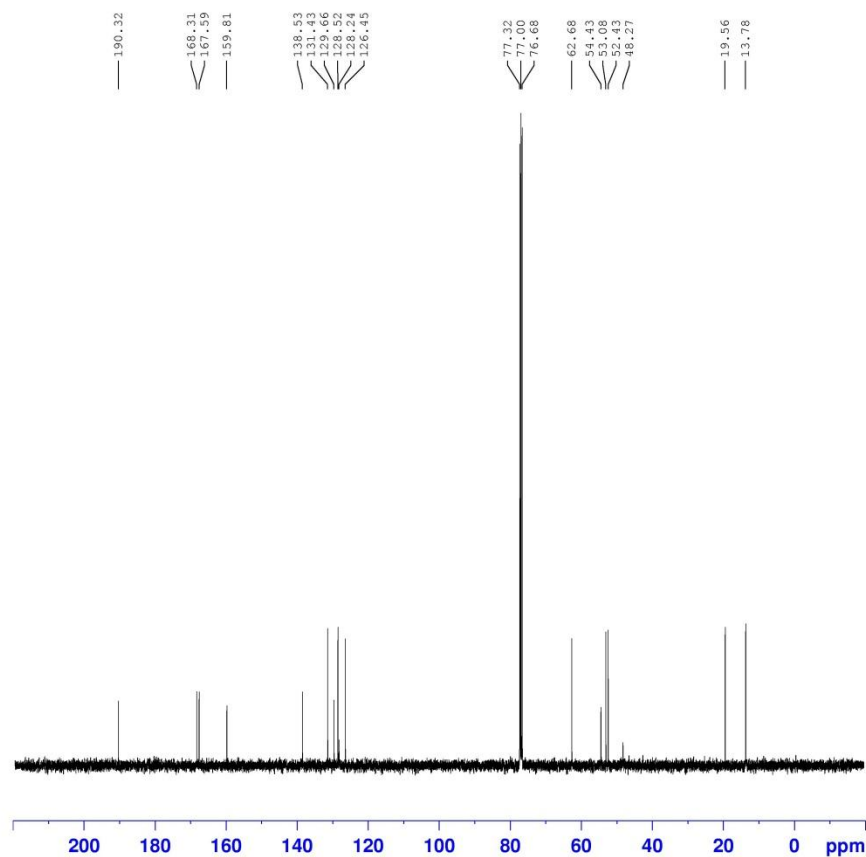


Current Data Parameters
NAME 6KMS269
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120302
Time 17.28
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 97.5
DW 60.800 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.38 usec
PLW1 11.19999981 W
SFO1 400.0924707 MHz

F2 - Processing parameters
SI 65536
SF 400.0900112 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



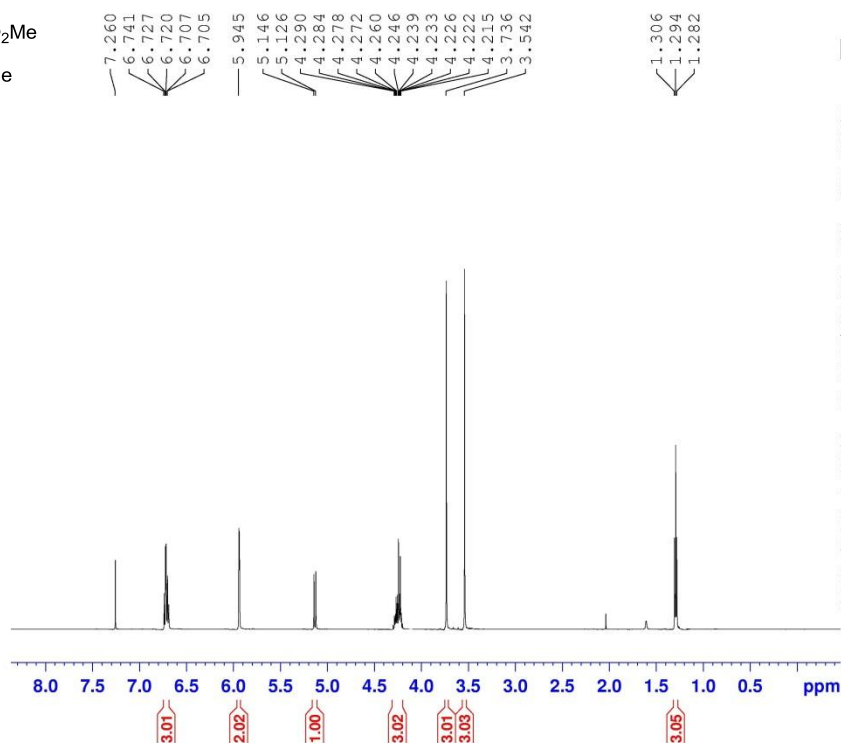
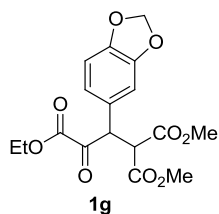
Current Data Parameters
NAME 6KMS269
EXPNO 2
PROCNO 1

F2 - Acquisition Parameter
Date_ 20120302
Time 17.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 221
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 200.09
DW 20.800 us
DE 6.50 us
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 7.50 us
PLW1 61.20000076 W
SFO1 100.6127703 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 us
PLW2 11.19999981 W
PLW12 0.26820999 W
PLW13 0.17160001 W
SFO2 400.0916004 MHz

F2 - Processing parameters
SI 32768
SF 100.6027142 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

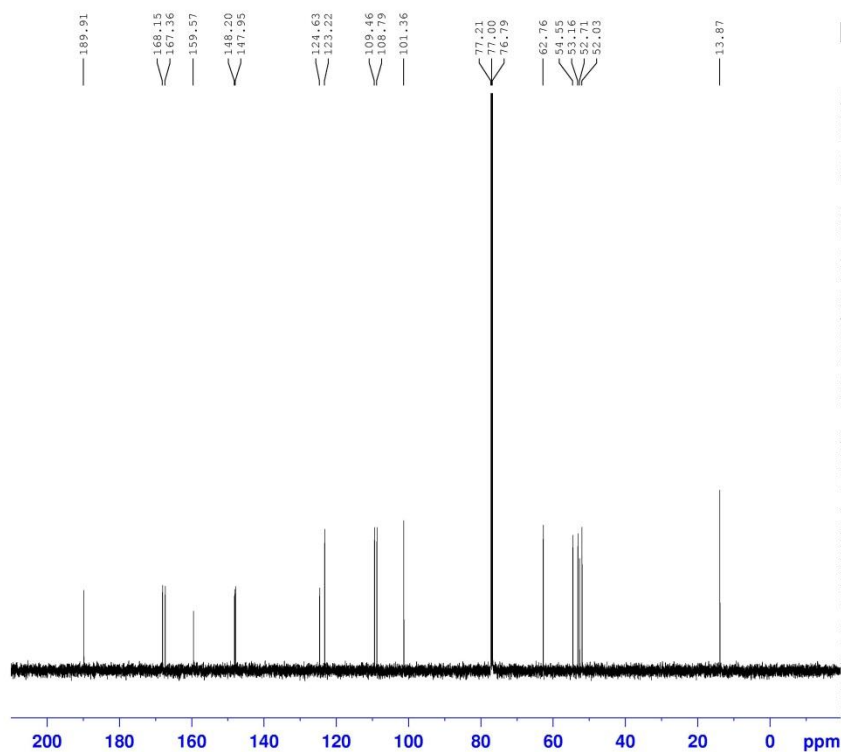


Current Data Parameters
NAME 6KMS260
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120130
Time 12.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 203
DW 40.533 usec
DE 6.50 usec
TE 292.8 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.60 usec
PLW1 20.00000000 W
SFO1 600.1337060 MHz

F2 - Processing parameters
SI 65536
SF 600.1300178 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



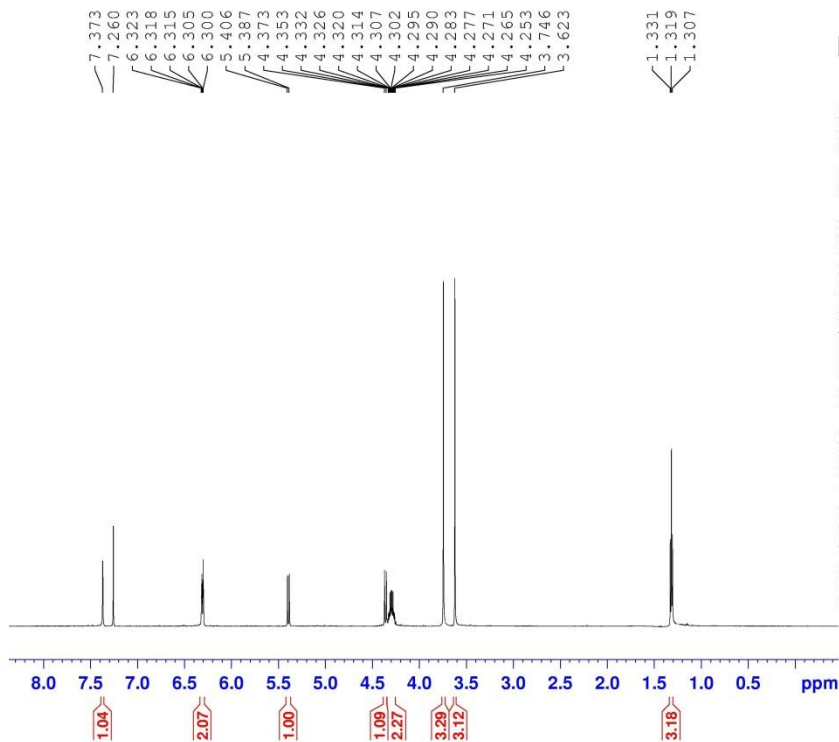
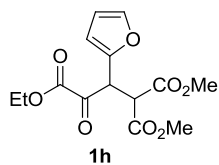
Current Data Parameters
NAME 6KMS260
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120130
Time 12.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 102
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 203
DW 13.867 usec
DE 10.00 usec
TE 293.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PLW1 110.00000000 W
SFO1 150.9178981 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PLW2 20.00000000 W
PLW12 0.80000001 W
PLW13 0.39199999 W
SFO2 600.1324005 MHz

F2 - Processing parameters
SI 32768
SF 150.9028162 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

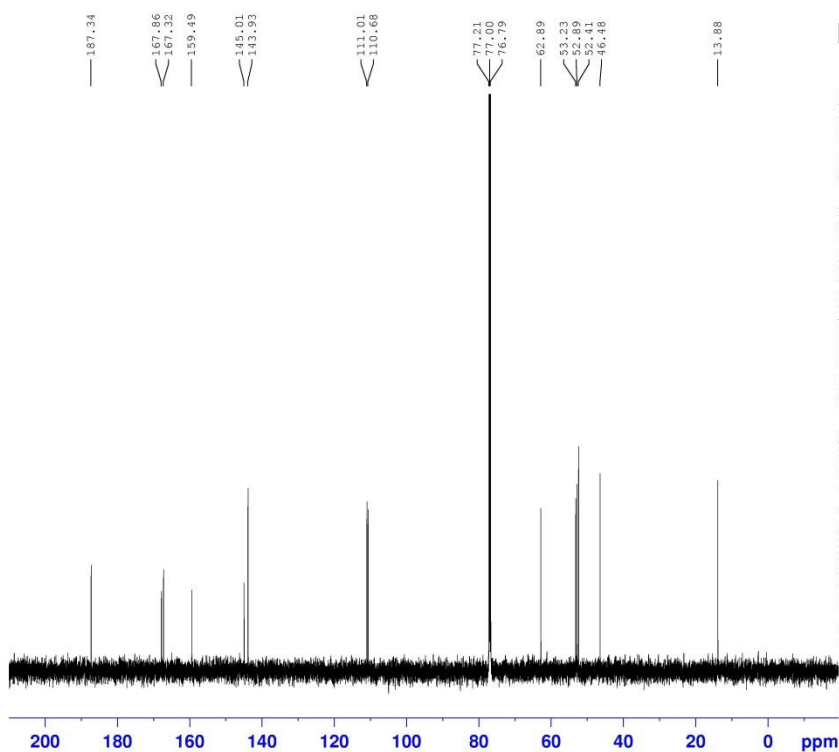


Current Data Parameters
NAME 6KMS205
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111212
Time 14.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 203
DW 40.533 usec
DE 6.50 usec
TE 292.7 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.60 usec
PLW1 20.00000000 W
SFO1 600.1337060 MHz

F2 - Processing parameters
SI 65536
SF 600.1300177 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



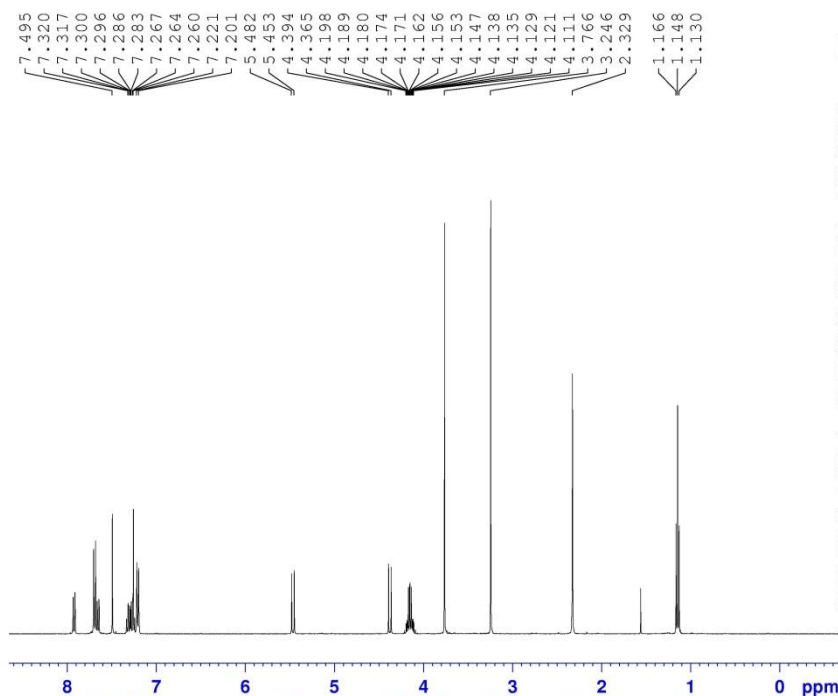
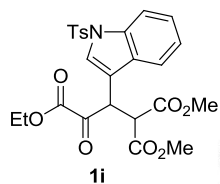
Current Data Parameters
NAME 6KMS205
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20111212
Time 14.17
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 230
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 203
DW 13.867 usec
DE 10.00 usec
TE 294.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PLW1 110.00000000 W
SFO1 150.9178981 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PLW2 20.00000000 W
PLW12 0.80000001 W
PLW13 0.39199999 W
SFO2 600.1324005 MHz

F2 - Processing parameters
SI 32768
SF 150.9028151 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

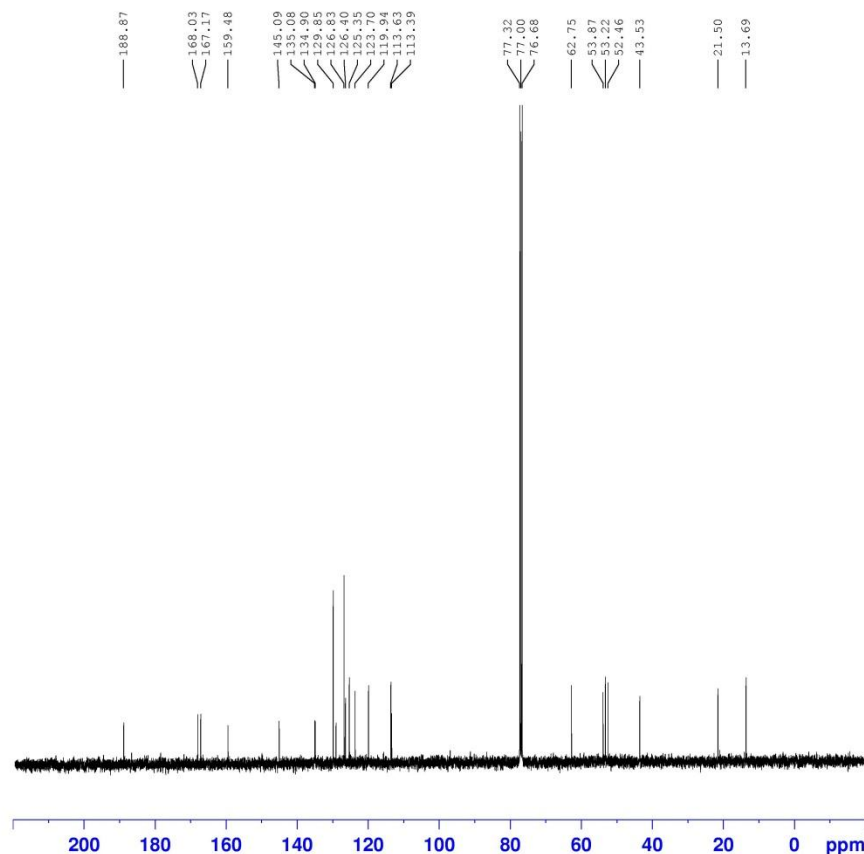


Current Data Parameters
 NAME 7KMS14
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120302
 Time 12.12
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 134.63
 DW 60.800 usec
 DE 6.50 usec
 TE 297.1 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 12.38 usec
 PLW1 11.19999981 W
 SFO1 400.0924707 MHz

F2 - Processing parameters
 SI 65536
 SF 400.0900114 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



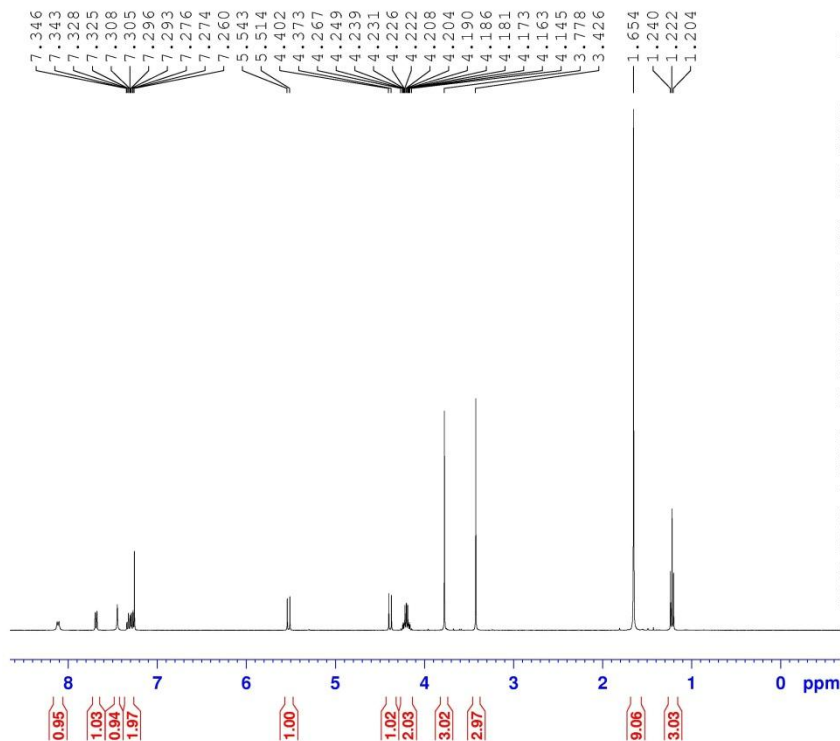
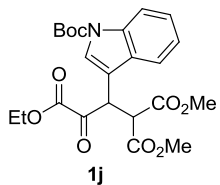
Current Data Parameters
 NAME 7MS14
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameter
 Date_ 20120214
 Time 15.35
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 163
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 200.09
 DW 20.800 us
 DE 6.50 us
 TE 302.6 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

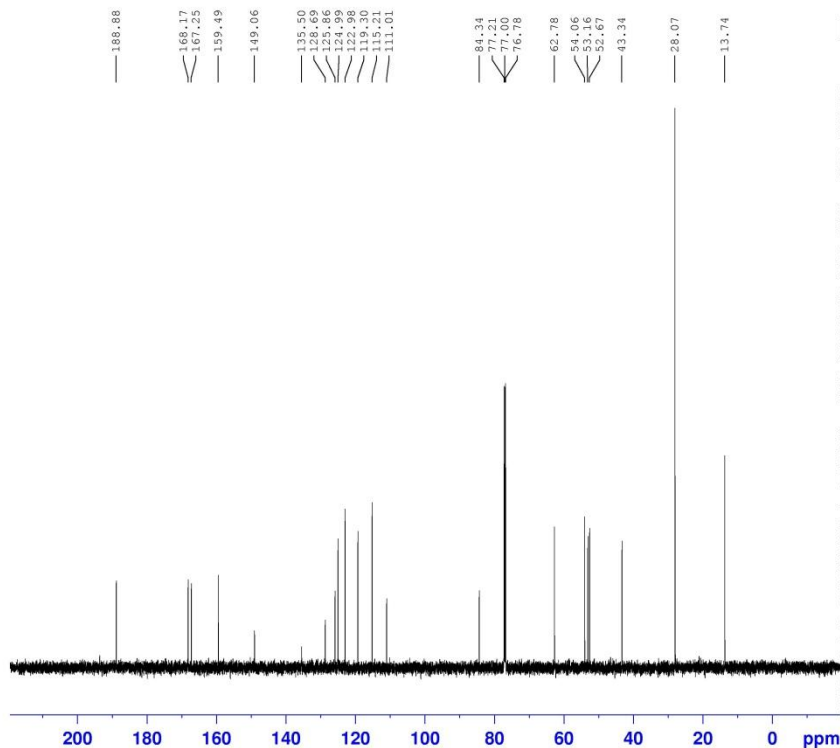
===== CHANNEL f1 =====
 NUC1 13C
 P1 7.50 us
 PLW1 61.20000076 W
 SFO1 100.6127703 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 us
 PLW2 11.19999981 W
 PLW12 0.26820999 W
 PLW13 0.17166001 W
 SFO2 400.0916004 MHz

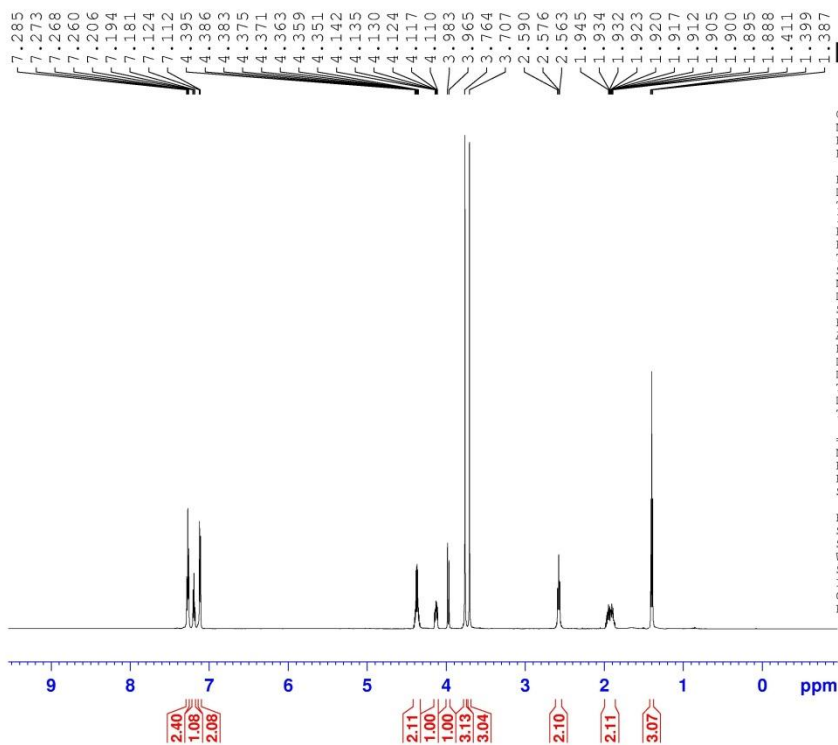
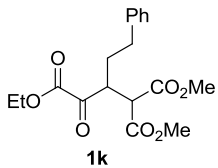
F2 - Processing parameters
 SI 32768
 SF 100.6027134 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



Current Data Parameters
NAME 7RMS15
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120302
Time 12.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 134.63
DW 60.800 usec
DE 6.50 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 1H
P1 12.38 usec
PLW1 11.19999981 W
SF01 400.0924707 MHz
F2 - Processing parameters
SI 65536
SF 400.0900113 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Current Data Parameters
NAME 7RMS15
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120302
Time 12.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 80
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 203
DW 13.867 usec
DE 10.00 usec
TE 294.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PLW1 110.00000000 W
SF01 150.9178981 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PLW2 20.00000000 W
PLW12 0.80000001 W
PLW13 0.39199999 W
SF02 600.1324005 MHz
F2 - Processing parameters
SI 32768
SF 150.9028217 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

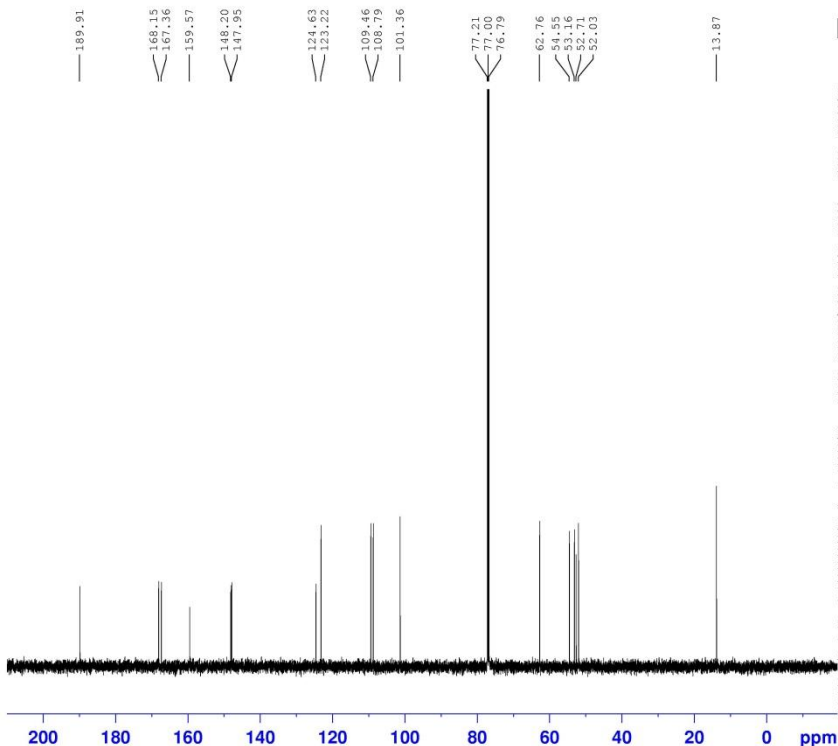


Current Data Parameters
NAME 6KMS206
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120406
Time 15.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 128
DW 40.533 usec
DE 6.50 usec
TE 293.7 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.60 usec
PLW1 20.00000000 W
SFO1 600.1337060 MHz

F2 - Processing parameters
SI 65536
SF 600.1300134 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



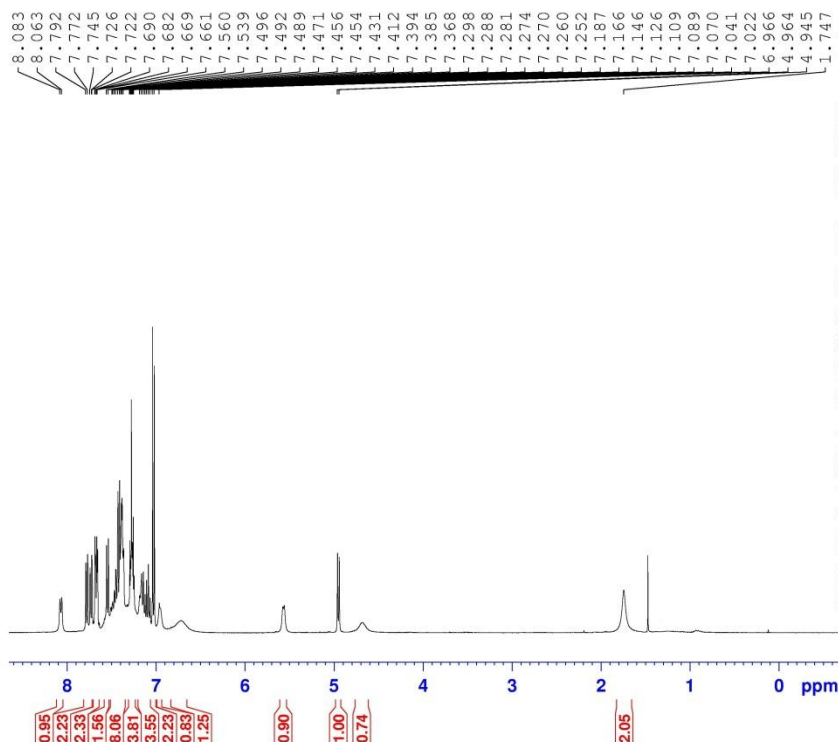
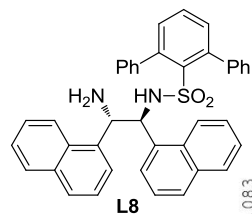
Current Data Parameters
NAME 6KMS260
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120130
Time 12.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 102
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 203
DW 13.867 usec
DE 10.00 usec
TE 293.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PLW1 110.00000000 W
SFO1 150.9178981 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PLW2 20.00000000 W
PLW12 0.80000000 W
PLW13 0.39199999 W
SFO2 600.1324005 MHz

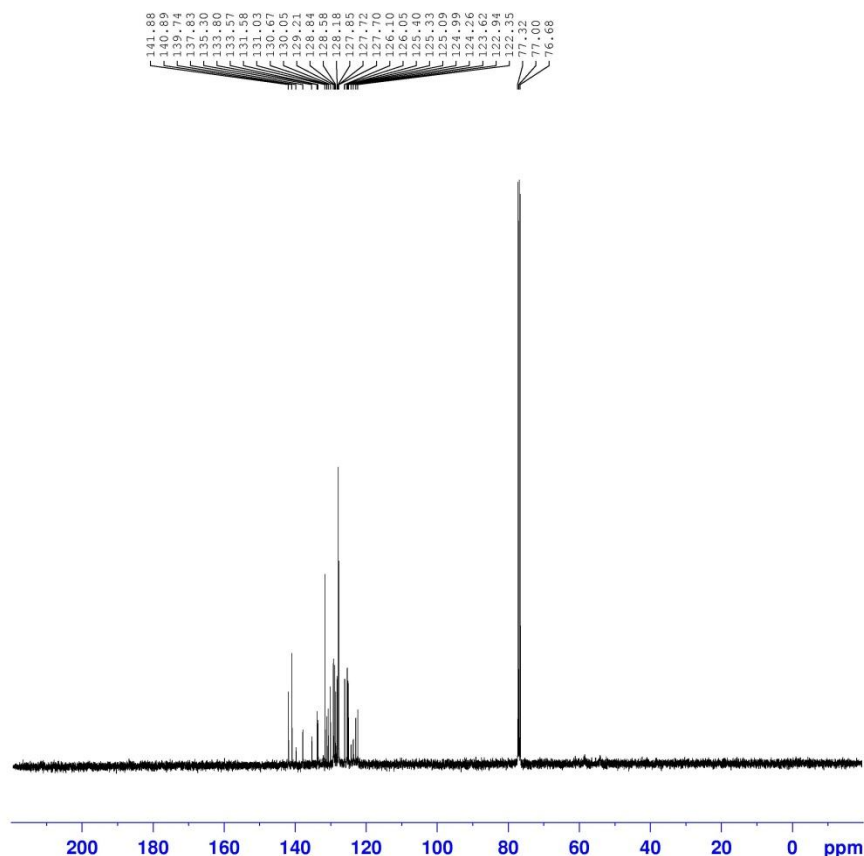
F2 - Processing parameters
SI 32768
SF 150.9028162 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



Current Data Parameters
NAME 6KMS262
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120214
Time 18.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 78.87
DW 60.800 usec
DE 6.50 usec
TE 301.8 K
D1 1.0000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.38 usec
PLW1 11.19999981 W
SFO1 400.0924707 MHz
F2 - Processing parameters
SI 65536
SF 400.0900029 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

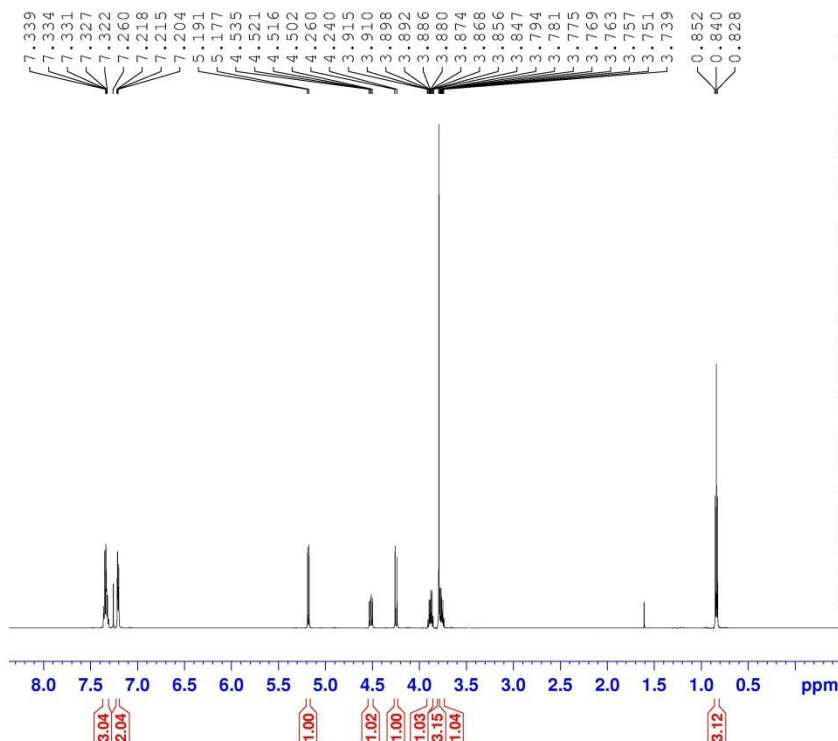
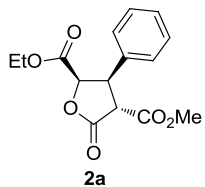


Current Data Parameters
NAME 6KMS262
EXPNO 2
PROCNO 1

F2 - Acquisition Parameter
Date_ 20120214
Time 18.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 320
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 200.09
DW 20.800 us
DE 6.50 us
TE 303.0 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 7.50 us
PLW1 61.20000076 W
SFO1 100.6127703 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 us
PLW2 11.19999981 W
PLW12 0.26820999 W
PLW13 0.17166001 W
SFO2 400.0916004 MHz

F2 - Processing parameters
SI 32768
SF 100.6027164 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

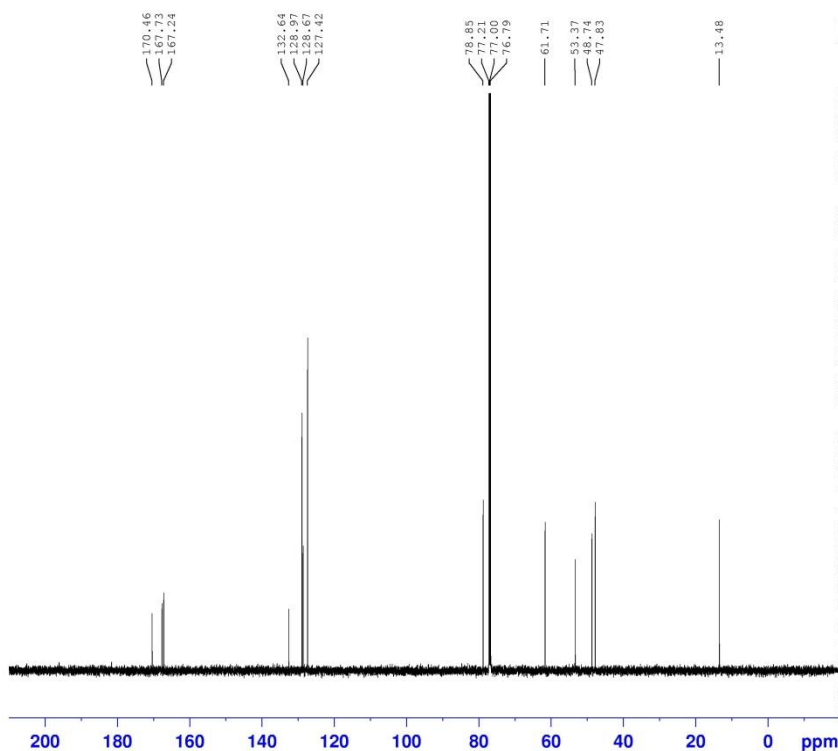


Current Data Parameters
NAME 6KMS184
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120208
Time 13.50
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 128
DW 40.533 usec
DE 6.50 usec
TE 293.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.60 usec
PLW1 20.00000000 W
SFO1 600.1337060 MHz

F2 - Processing parameters
SI 65536
SF 600.1300179 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



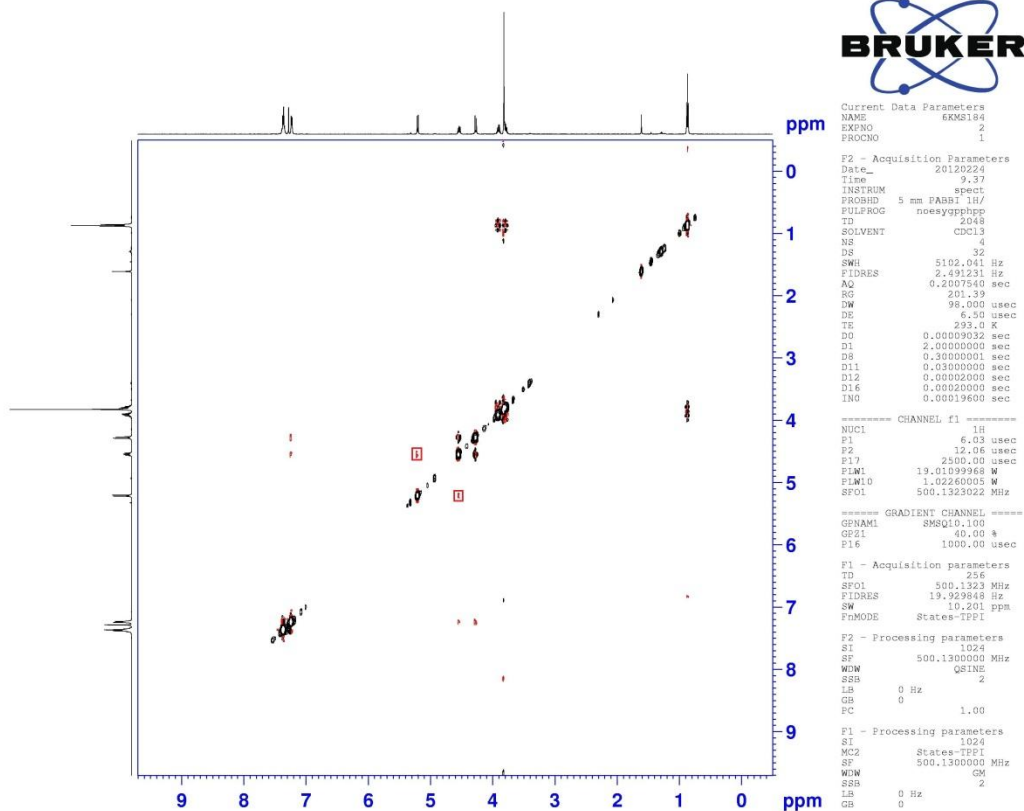
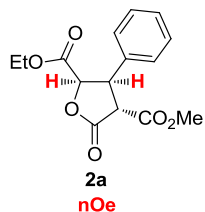
Current Data Parameters
NAME 6KMS184
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120208
Time 13.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 115
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 203
DW 13.867 usec
DE 10.00 usec
TE 294.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

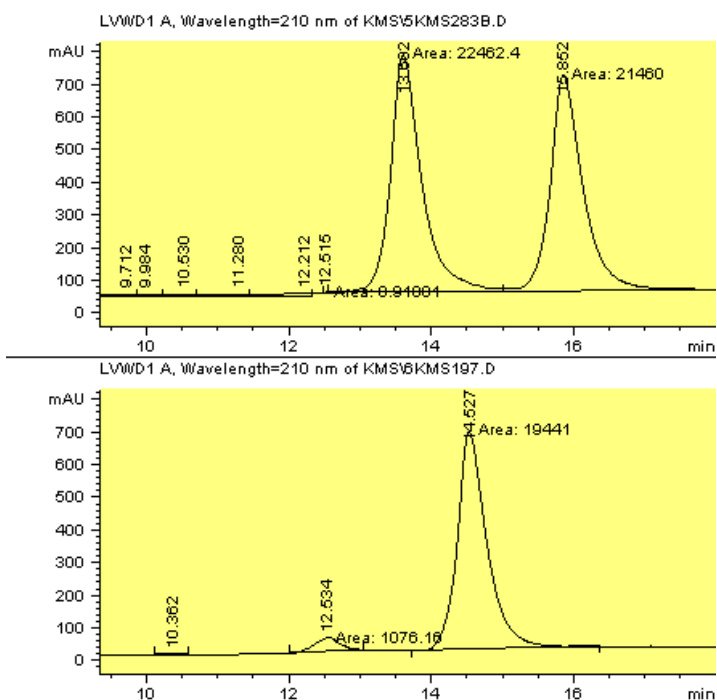
===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PLW1 110.00000000 W
SFO1 150.9178981 MHz

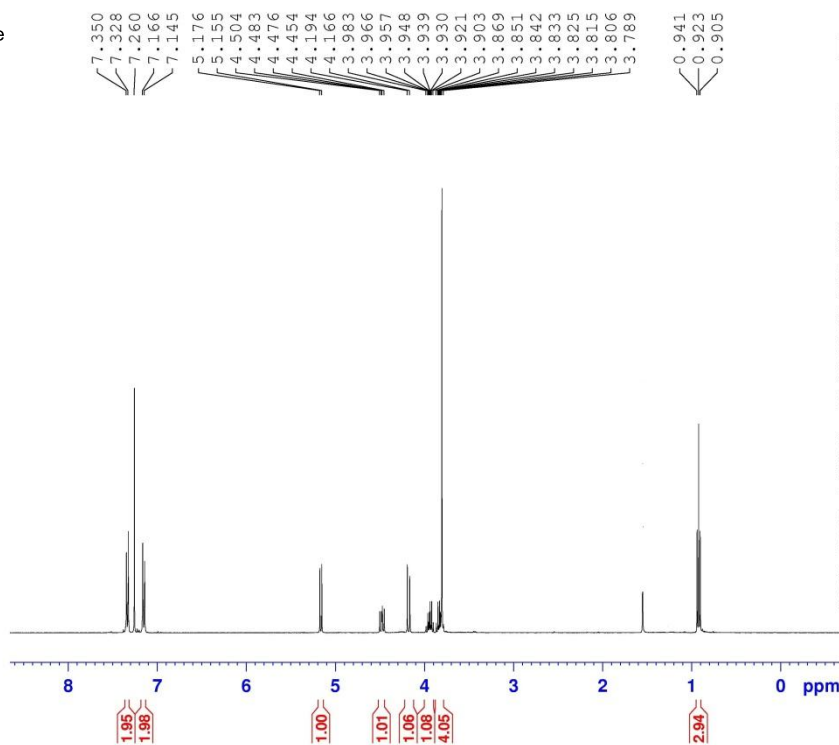
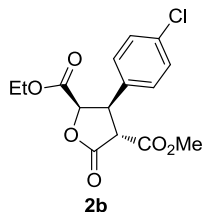
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PLW2 20.00000000 W
PLW12 0.80000001 W
PLW13 0.39199999 W
SFO2 600.1324005 MHz

F2 - Processing parameters
SI 32768
SF 150.9028173 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



SFC Analysis for 2a:



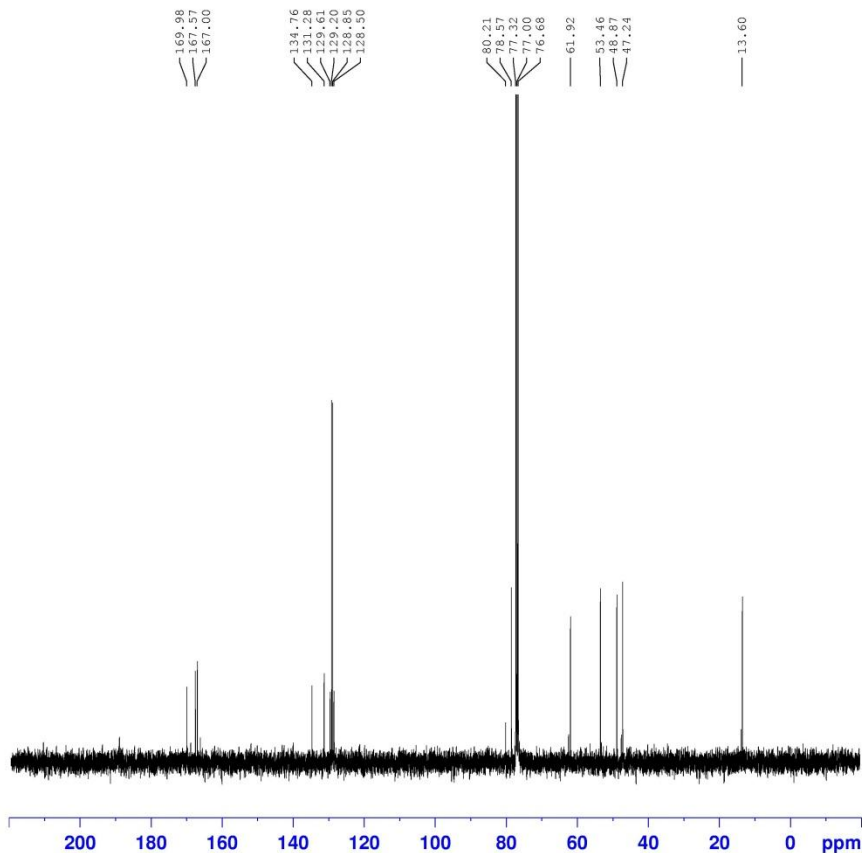


Current Data Parameters
NAME 6KMS266
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120303
Time 12.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 200.09
DW 60.800 usec
DE 6.50 usec
TE 297.4 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.38 usec
PLW1 11.19999981 W
SFO1 400.0924707 MHz

F2 - Processing parameters
SI 65536
SF 400.0900115 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



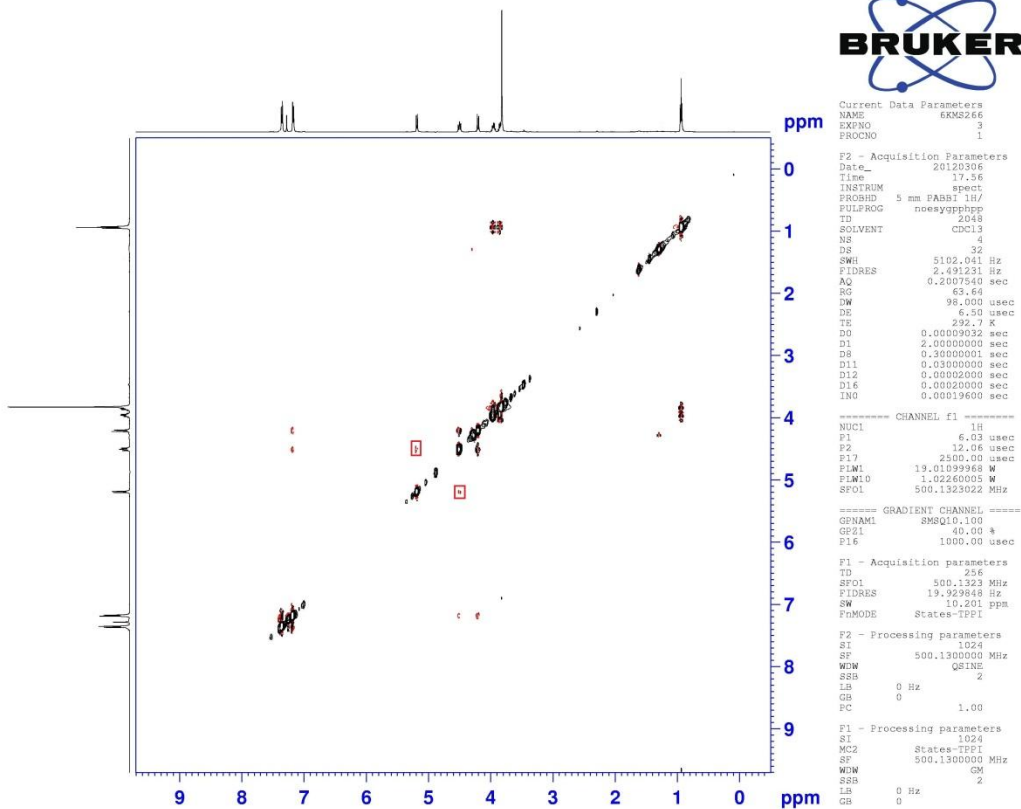
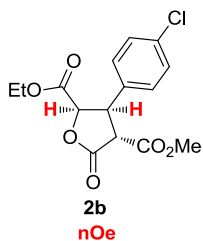
Current Data Parameters
NAME 6KMS266
EXPNO 2
PROCNO 1

F2 - Acquisition Parameter
Date_ 20120303
Time 14.36
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 201
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 200.09
DW 20.800 us
DE 6.50 us
TE 298.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

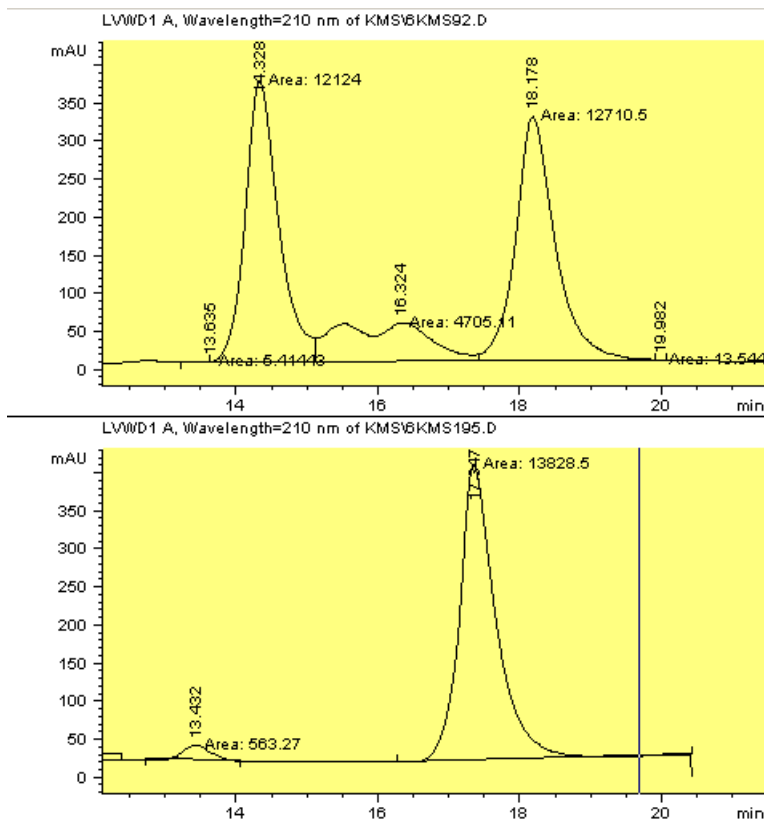
===== CHANNEL f1 =====
NUC1 13C
P1 7.50 us
PLW1 61.20000076 W
SFO1 100.6127703 MHz

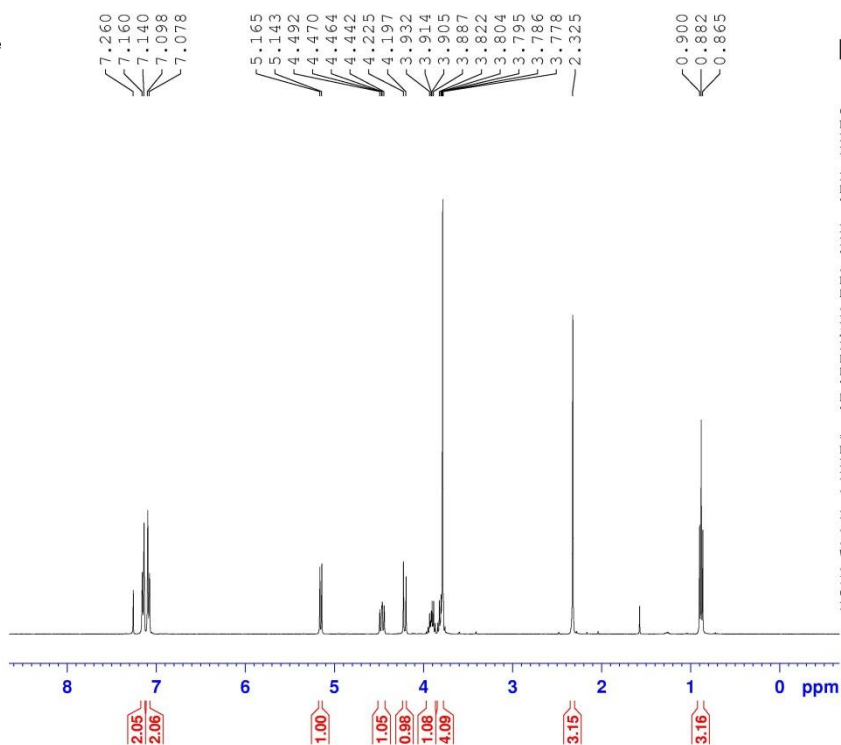
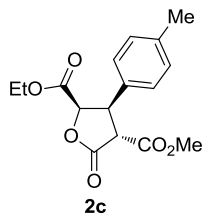
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 us
PLW2 11.19999981 W
PLW12 0.26820999 W
PLW13 0.17166001 W
SFO2 400.0916004 MHz

F2 - Processing parameters
SI 32768
SF 100.6027135 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



SCF Analysis fo 2b:



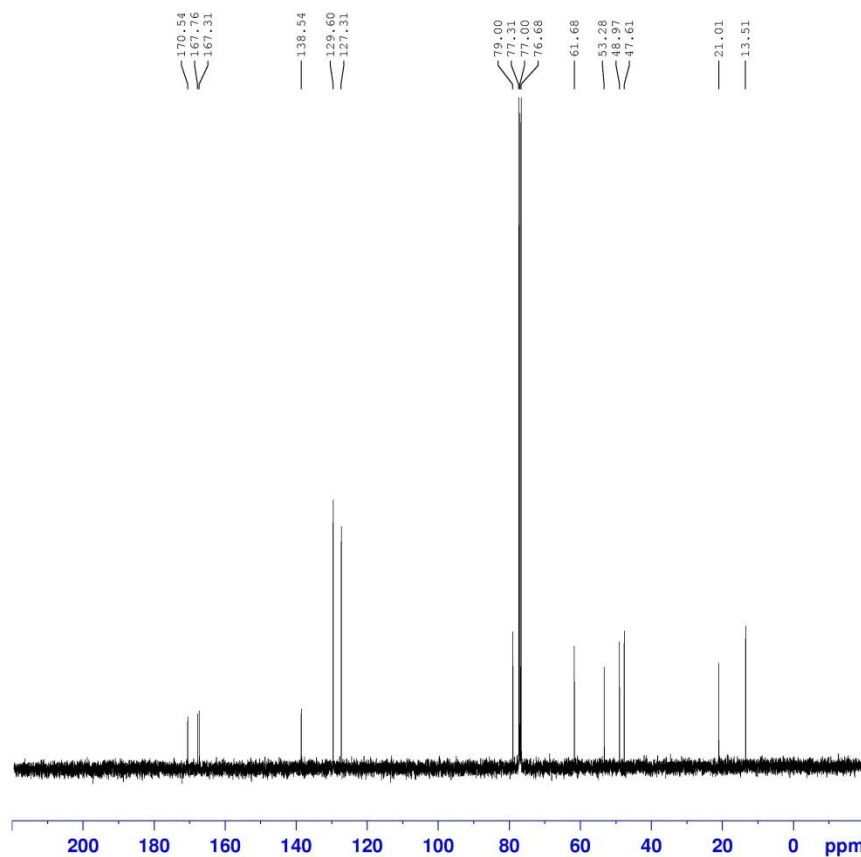


Current Data Parameters
NAME 6KMS298
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120210
Time 11.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 110.42
DW 60.800 usec
DE 6.50 usec
TE 302.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.38 usec
PLW1 11.19999981 W
SFO1 400.0924707 MHz

F2 - Processing parameters
SI 65536
SF 400.0900105 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



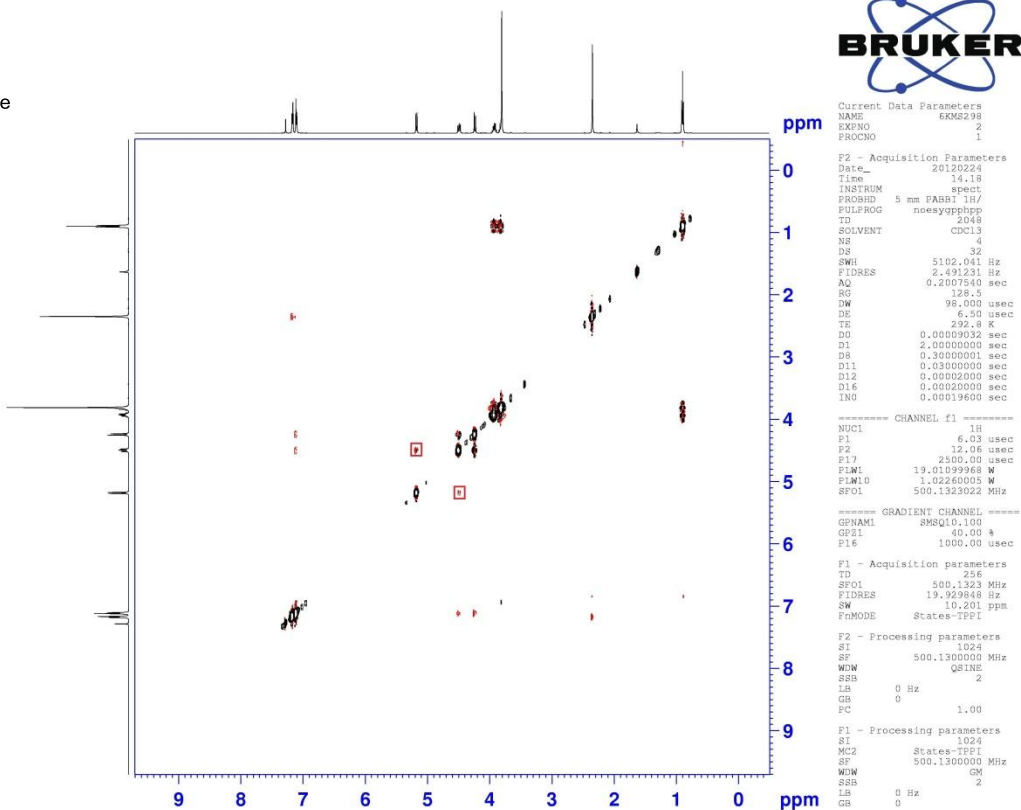
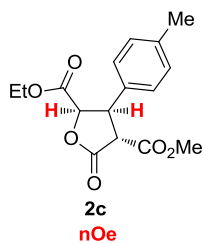
Current Data Parameters
NAME 6KMS298
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120210
Time 11.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 100
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 200.09
DW 20.800 usec
DE 6.50 usec
TE 302.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

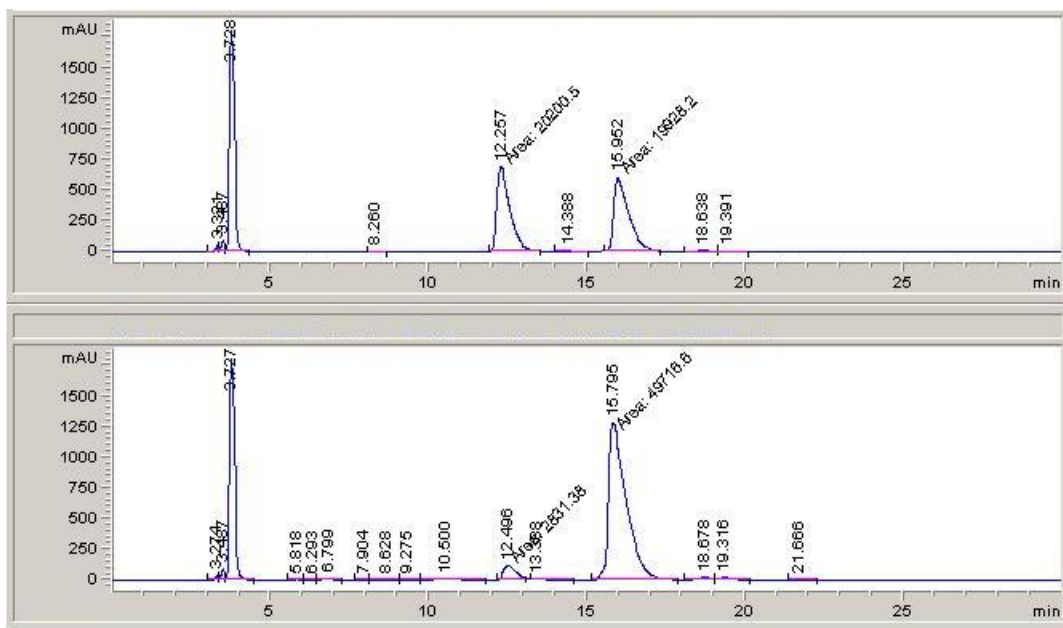
===== CHANNEL f1 =====
NUC1 13C
P1 7.50 usec
PLW1 61.20000076 W
SFO1 100.6127703 MHz

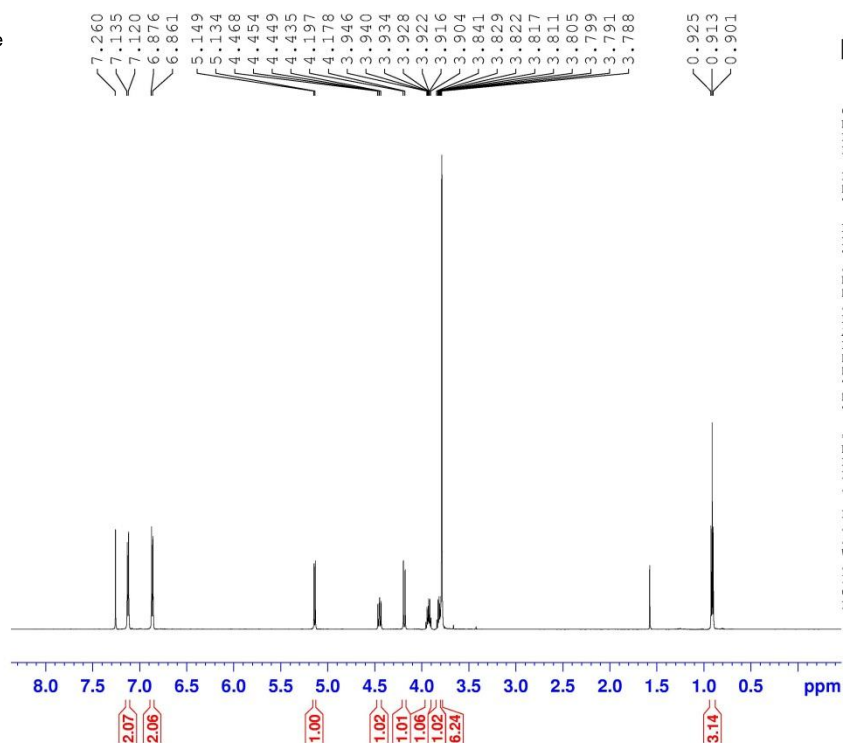
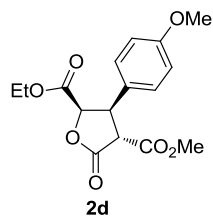
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 11.19999981 W
PLW12 0.26820999 W
PLW13 0.17166001 W
SFO2 400.0916004 MHz

F2 - Processing parameters
SI 32768
SF 100.6027135 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



HPLC Analysis for 2c:



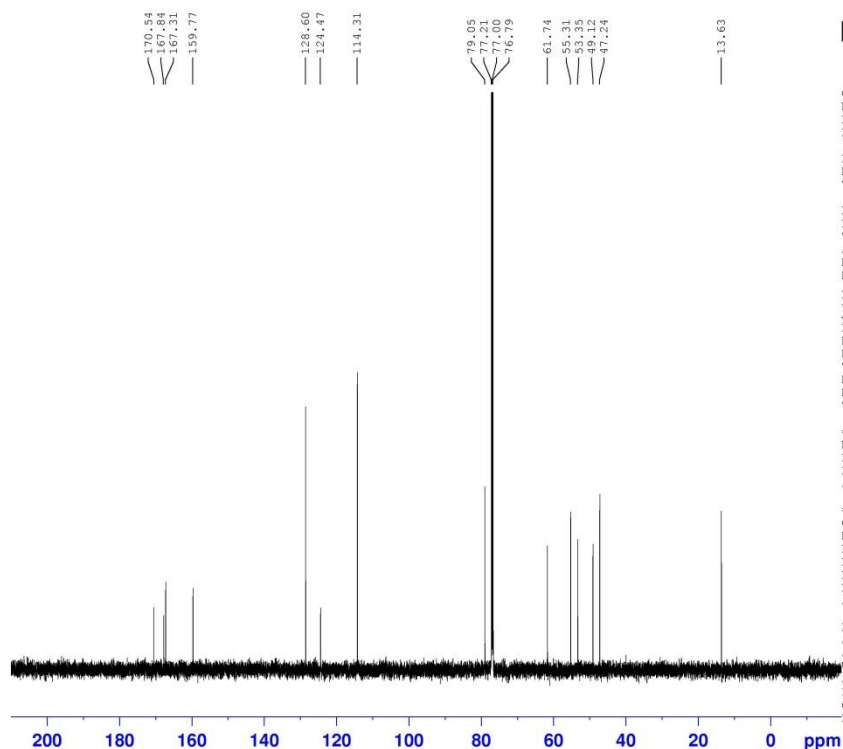


Current Data Parameters
NAME 6KMS203
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120210
Time 14.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 203
DW 40.533 usec
DE 6.50 usec
TE 293.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.60 usec
PLW1 20.00000000 W
SF01 600.1337060 MHz

F2 - Processing parameters
SI 65536
SF 600.1300179 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



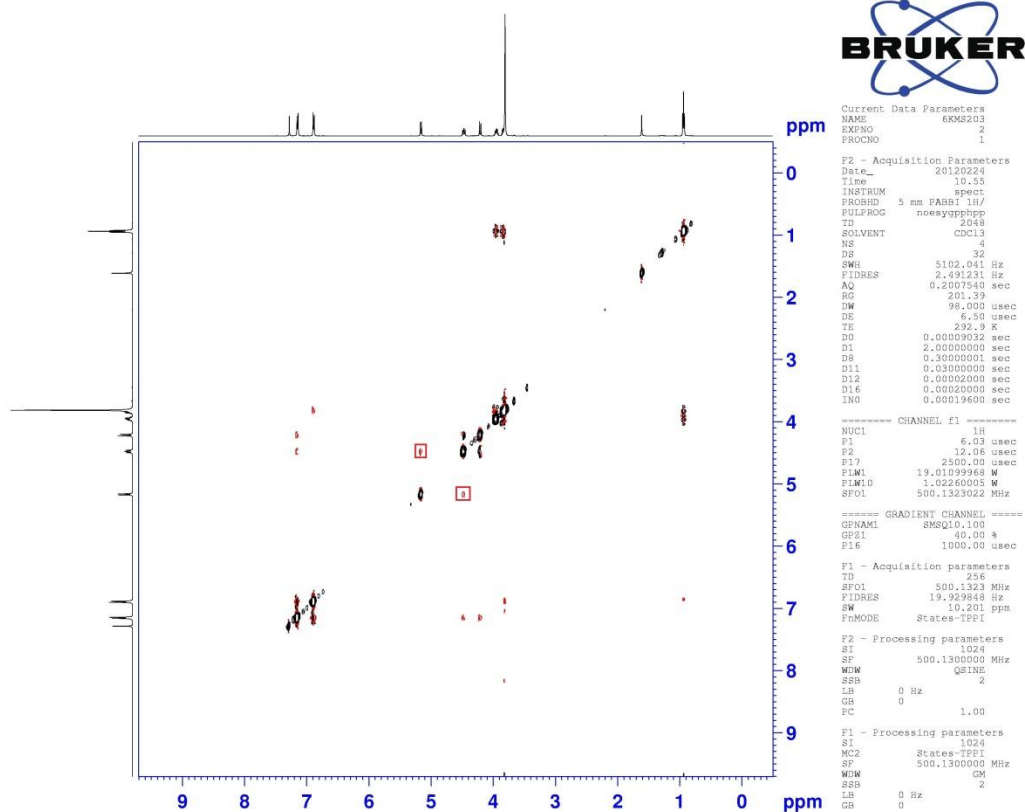
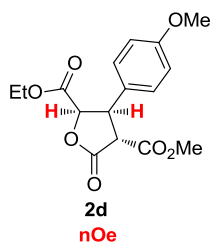
Current Data Parameters
NAME 6KMS203
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120210
Time 14.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 270
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 203
DW 13.867 usec
DE 10.00 usec
TE 294.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

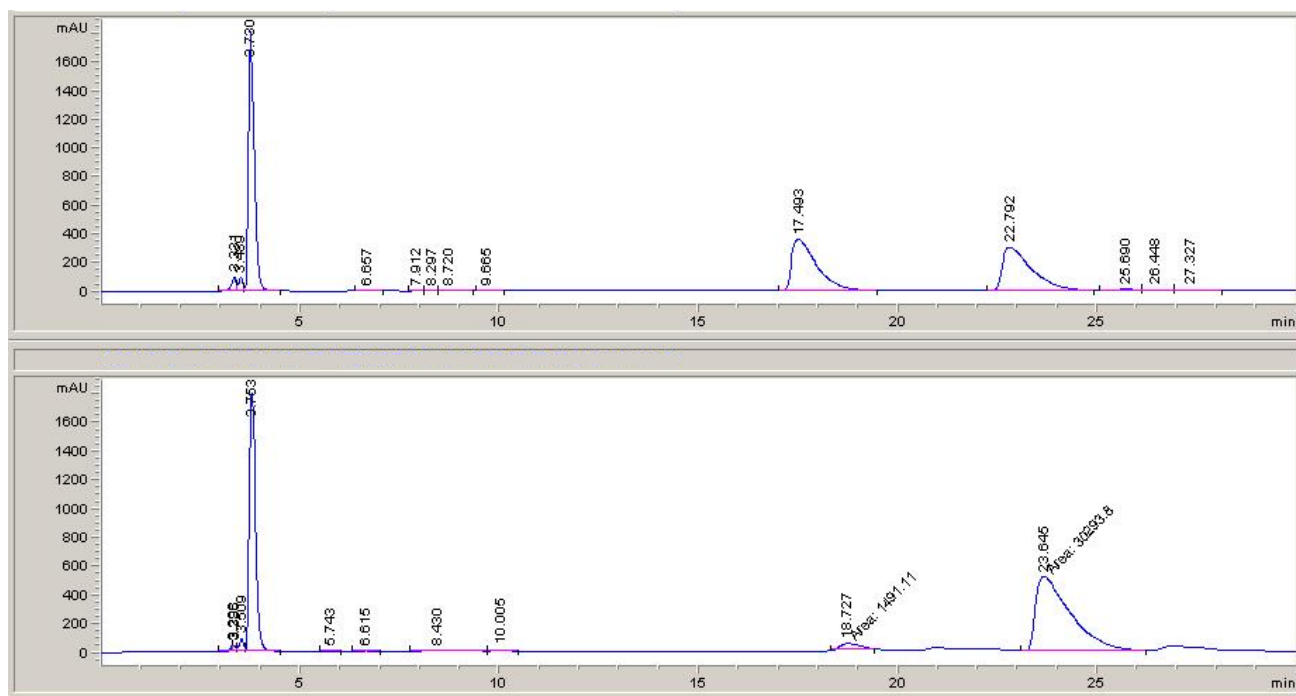
===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PLW1 110.00000000 W
SF01 150.9178981 MHz

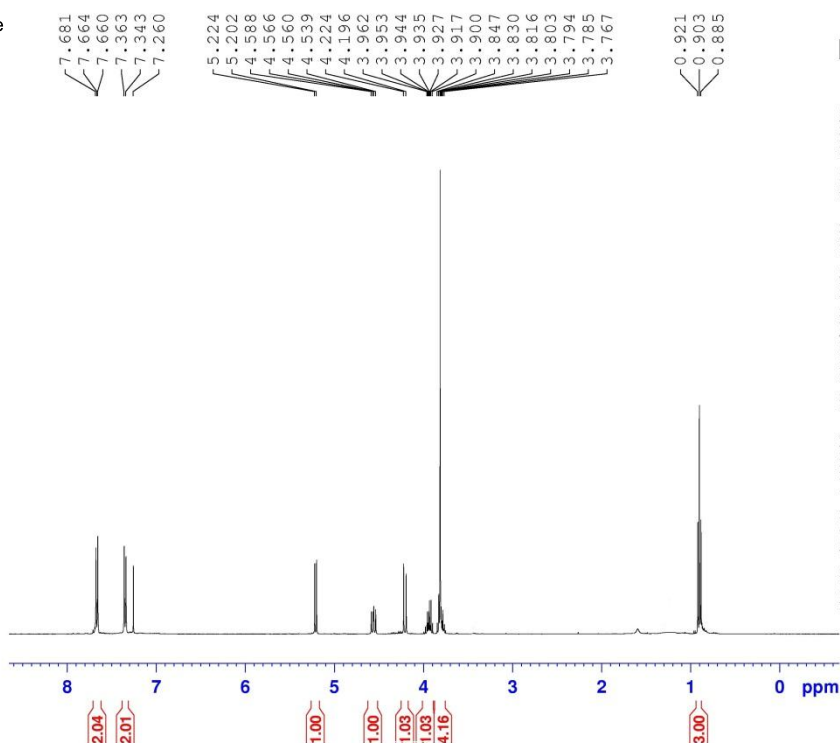
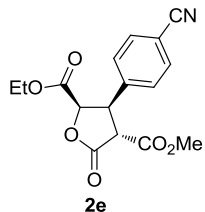
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PLW2 20.00000000 W
PLW12 0.800000001 W
PLW13 0.39199999 W
SF02 600.1324005 MHz

F2 - Processing parameters
SI 32768
SF 150.9028151 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



HPLC Analysis for 2d:



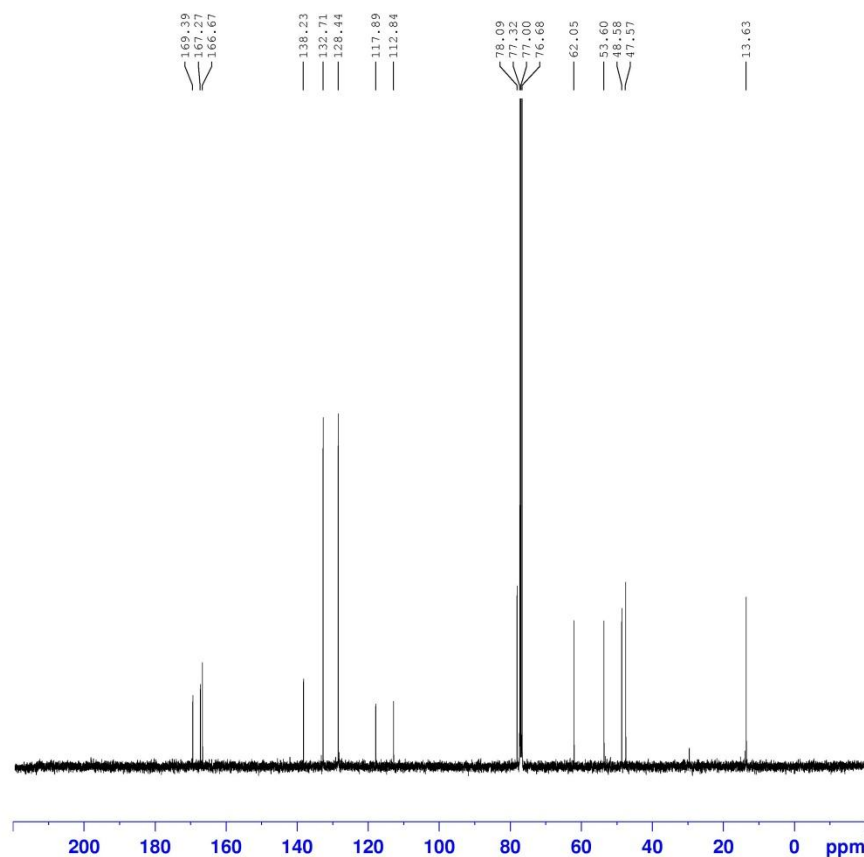


Current Data Parameters
NAME 6KMS233
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120302
Time 19.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 110.42
DW 60.800 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.38 usec
PLW1 11.19999981 W
SFO1 400.0924707 MHz

F2 - Processing parameters
SI 65536
SF 400.0900114 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



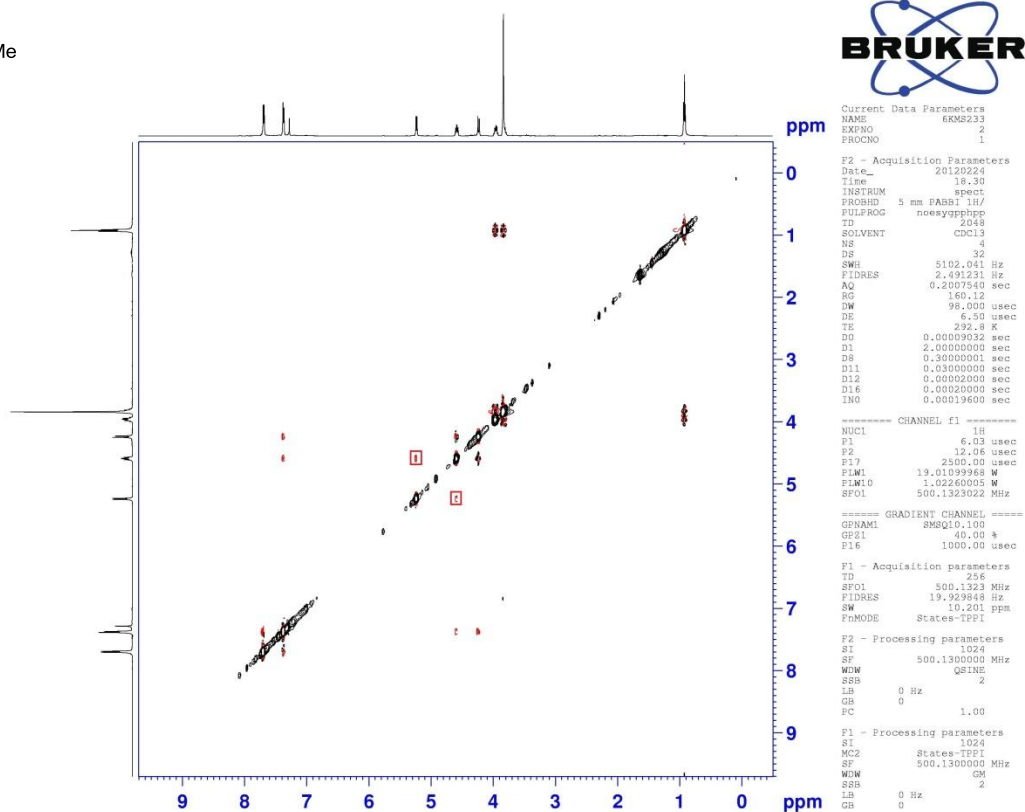
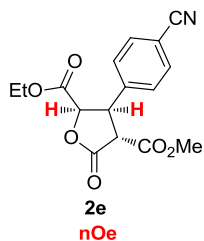
Current Data Parameters
NAME 6KMS233
EXPNO 2
PROCNO 1

F2 - Acquisition Parameter
Date_ 20120302
Time 19.53
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 608
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 200.09
DW 20.800 us
DE 6.50 us
TE 298.5 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1

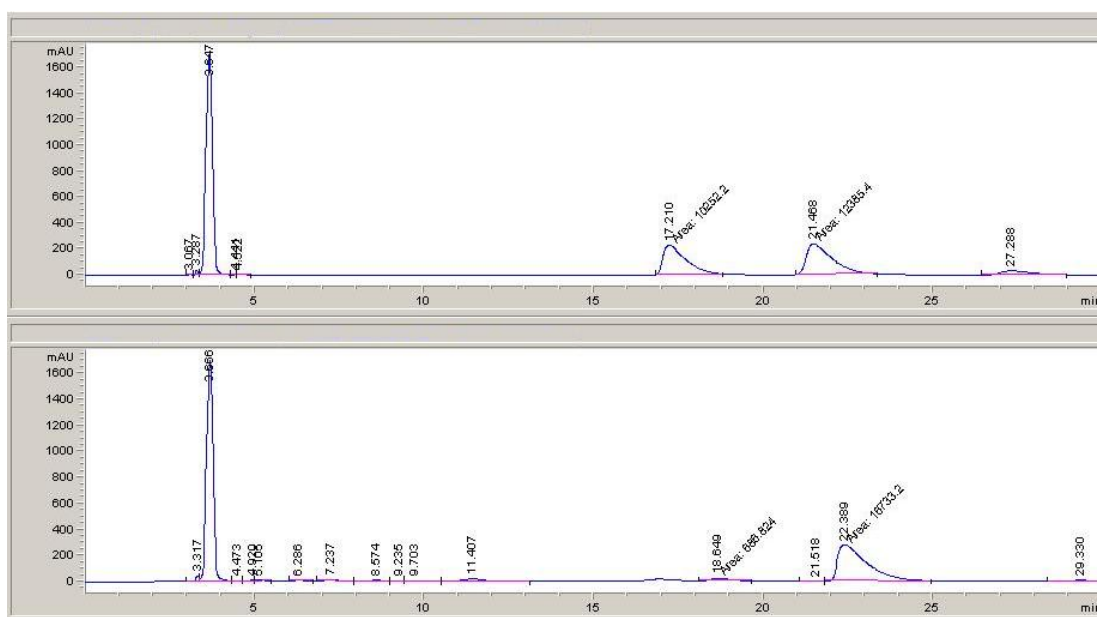
===== CHANNEL f1 =====
NUC1 13C
P1 7.50 us
PLW1 61.20000076 W
SFO1 100.6127703 MHz

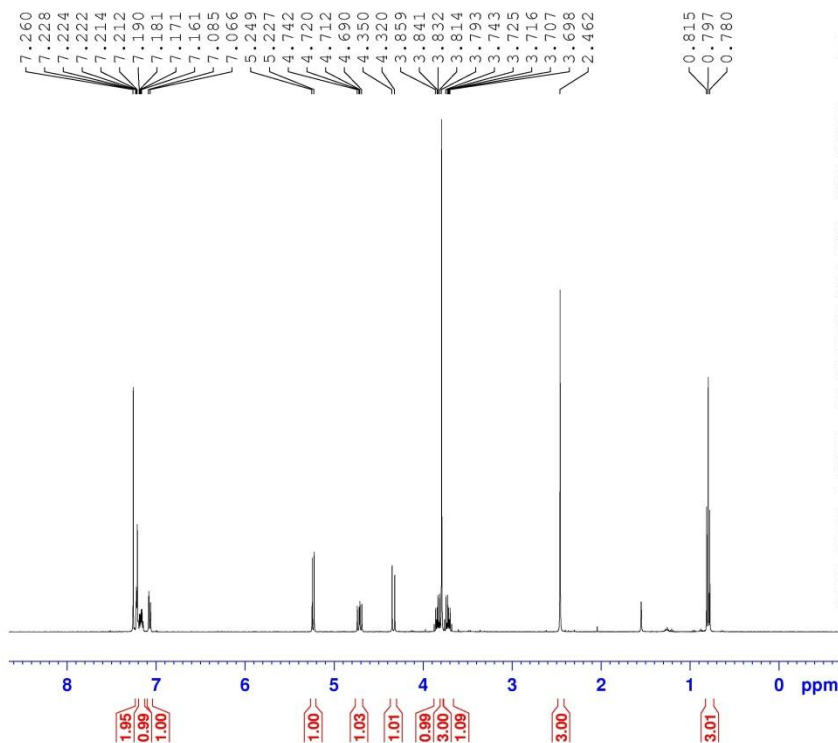
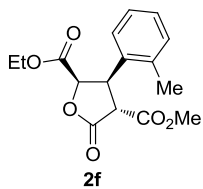
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 us
PLW2 11.19999981 W
PLW12 0.26820999 W
PLW13 0.17166001 W
SFO2 400.0916004 MHz

F2 - Processing parameters
SI 32768
SF 100.6027149 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



HPLC Analysis for 2e:



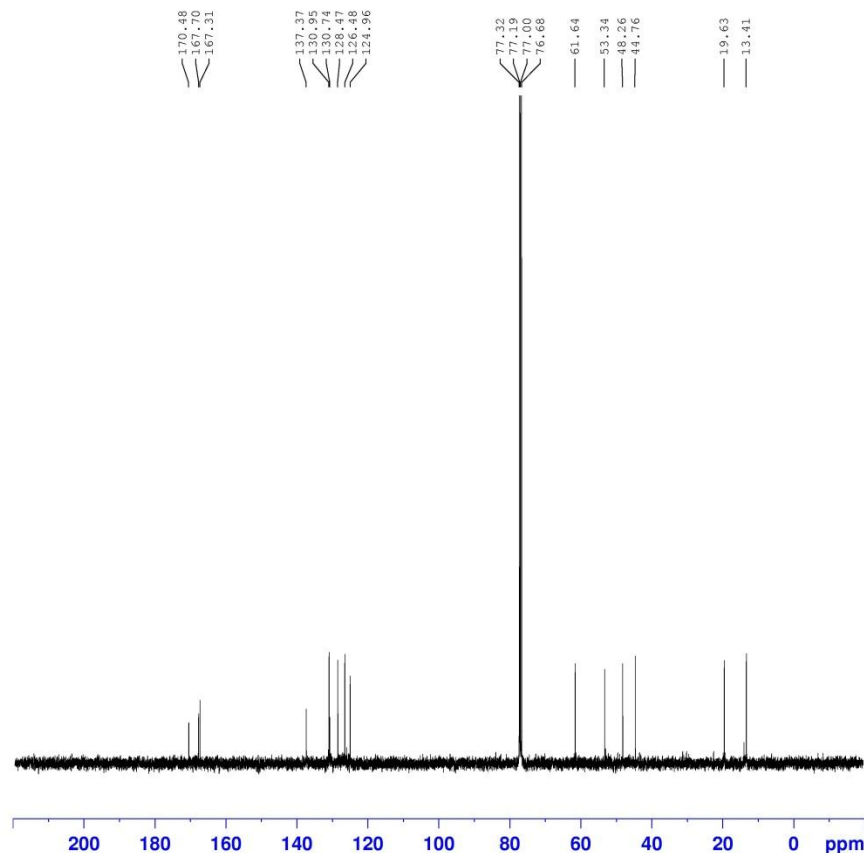


Current Data Parameters
NAME 7KMS61
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120303
Time 14.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 14
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 200.09
DW 60.800 usec
DE 6.50 usec
TE 297.9 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.38 usec
PLW1 11.19999981 W
SFO1 400.0924707 MHz

F2 - Processing parameters
SI 65536
SF 400.0900115 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



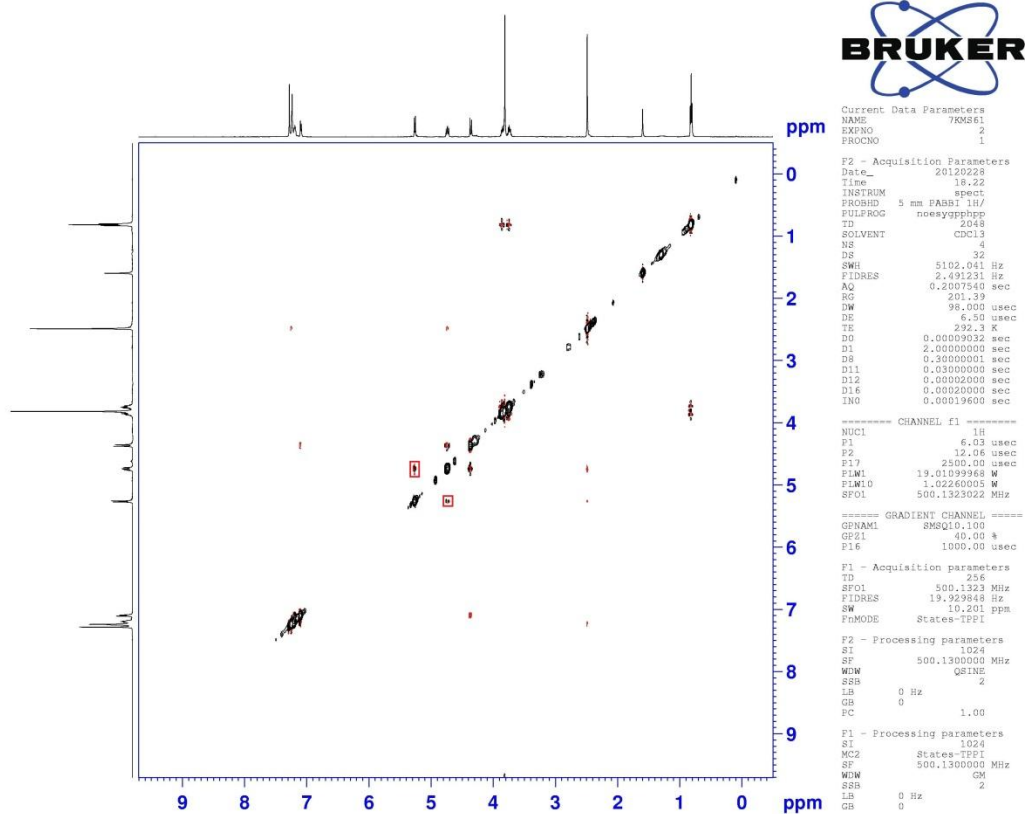
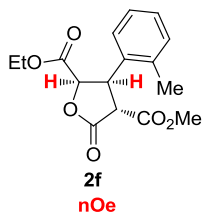
Current Data Parameters
NAME 7KMS61
EXPNO 2
PROCNO 1

F2 - Acquisition Parameter
Date_ 20120303
Time 12.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 254
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 200.09
DW 20.800 us
DE 6.50 us
TE 298.6 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1

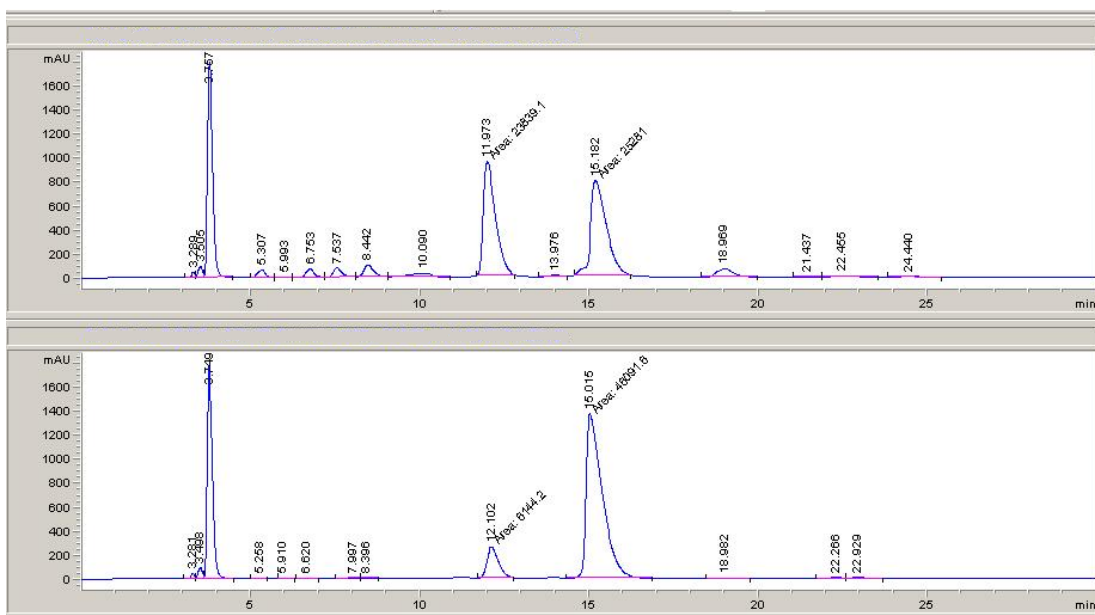
===== CHANNEL f1 =====
NUC1 13C
P1 7.50 us
PLW1 61.20000076 W
SFO1 100.6127703 MHz

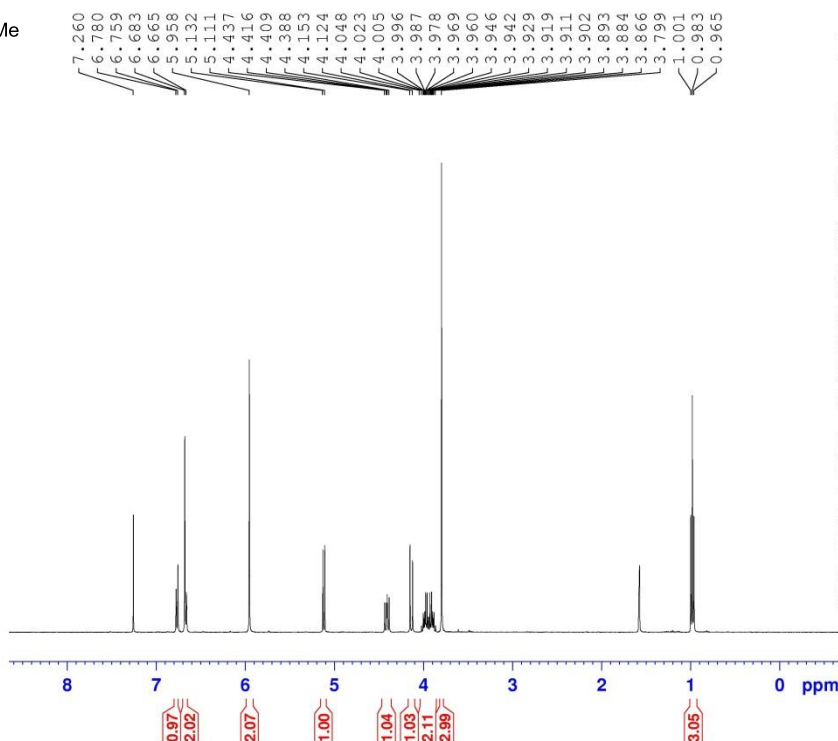
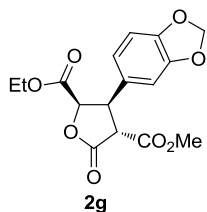
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 us
PLW2 11.19999981 W
PLW12 0.26820999 W
PLW13 0.17166001 W
SFO2 400.0916004 MHz

F2 - Processing parameters
SI 32768
SF 100.6027149 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



HPLC Analysis for 2f:



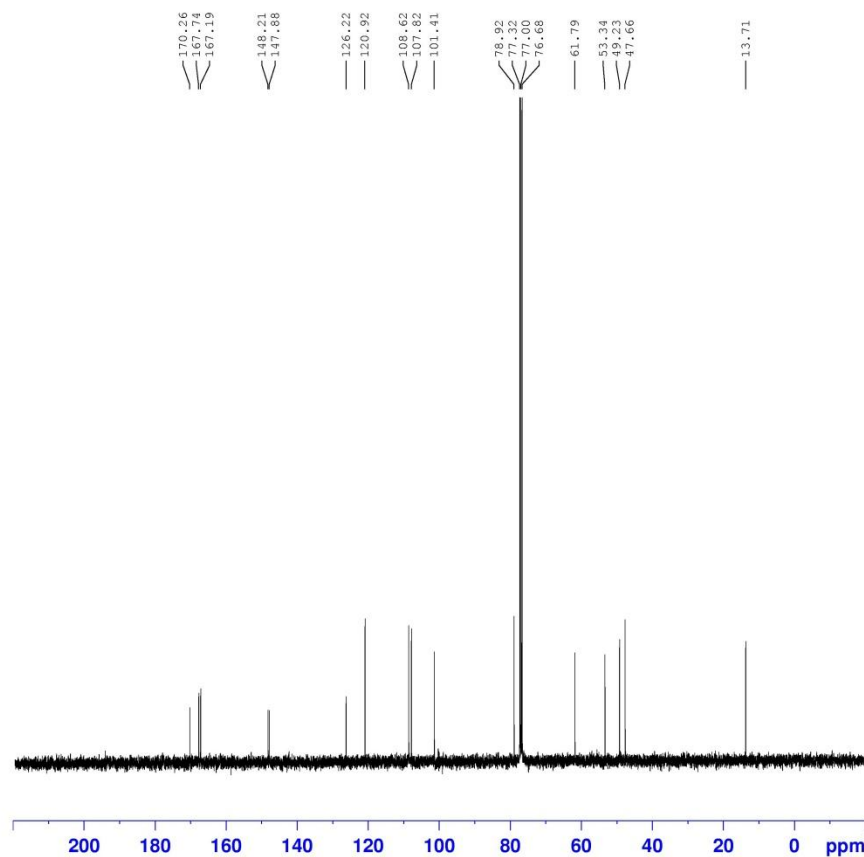


Current Data Parameters
 NAME 7KM25
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120302
 Time 9.25
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 156.94
 DW 60.800 usec
 DE 6.50 usec
 TE 297.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 12.38 usec
 PLW1 11.1999981 W
 SFO1 400.0924707 MHz

F2 - Processing parameters
 SI 65536
 SF 400.0900114 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



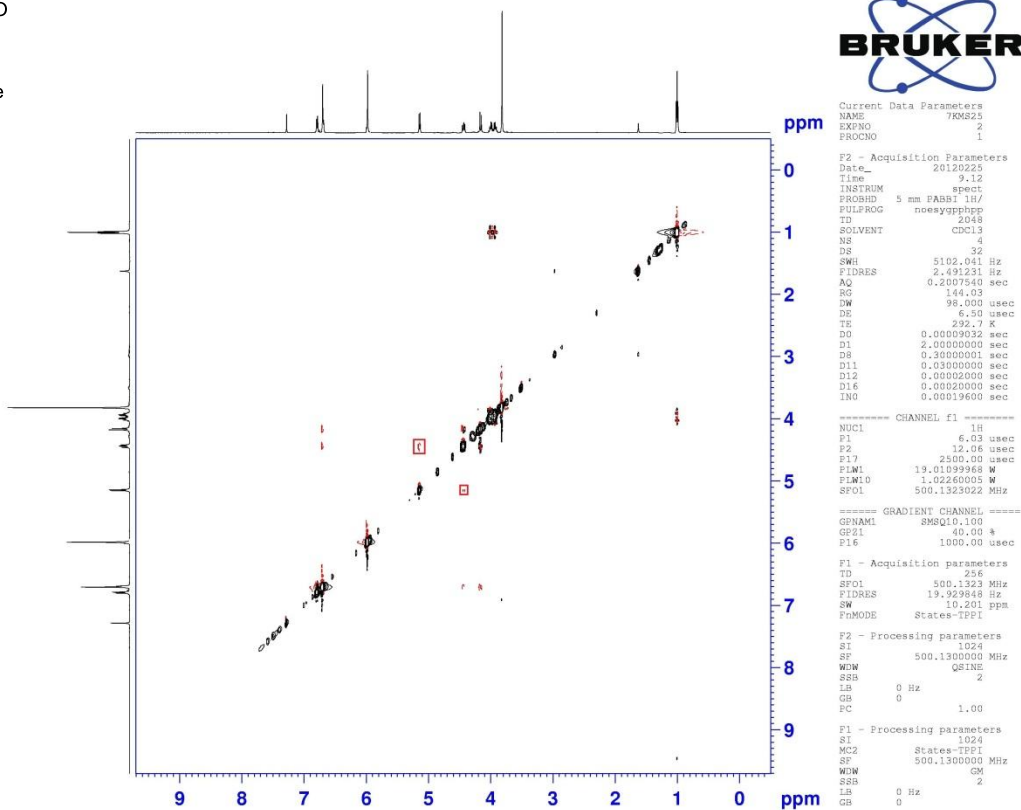
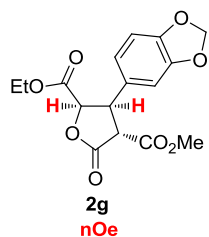
Current Data Parameters
 NAME 7KM25
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameter
 Date_ 20120215
 Time 15.30
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 201
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 se
 RG 200.09
 DW 20.800 us
 DE 6.50 us
 TE 302.4 K
 D1 2.00000000 se
 D11 0.03000000 se
 TD0 1

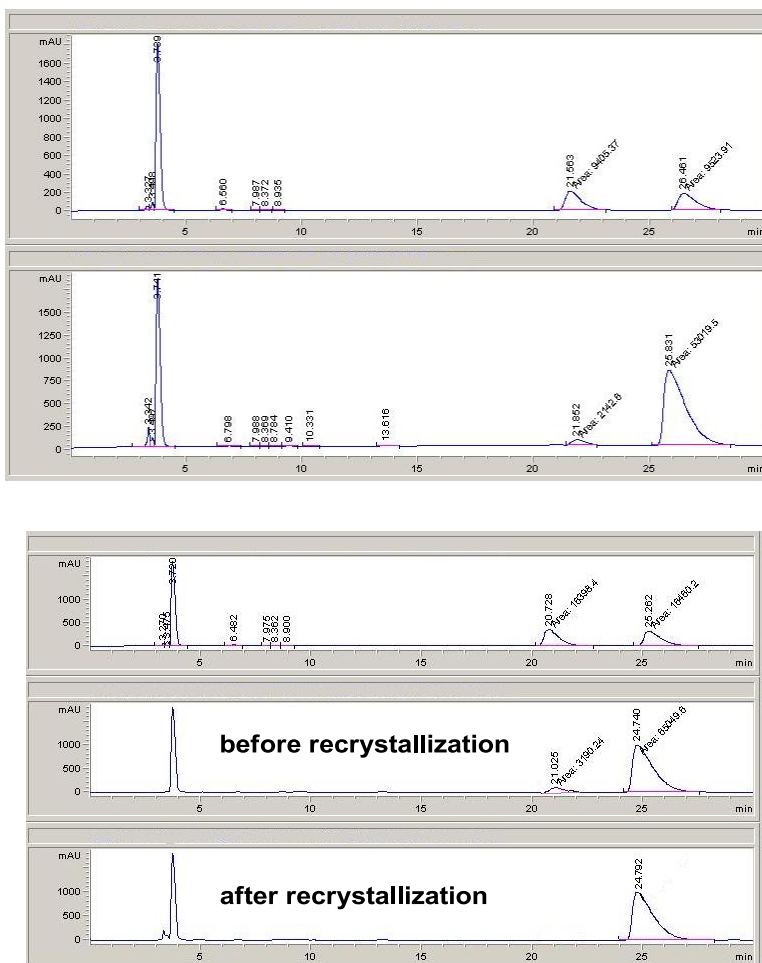
===== CHANNEL f1 =====
 NUC1 13C
 P1 7.50 usec
 PLW1 61.20000076 W
 SFO1 100.6127703 MHz

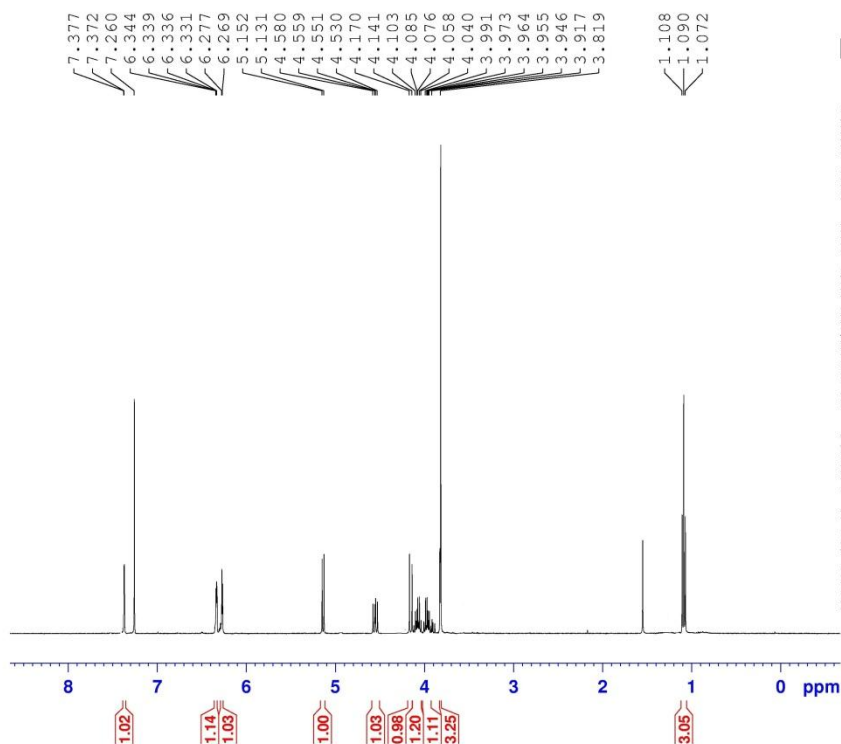
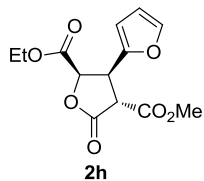
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PLW2 11.1999981 W
 PLW12 0.26820999 W
 PLW13 0.17160001 W
 SFO2 400.0916004 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6027131 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



HPLC Analysis for 2g (top: small scale; bottom: large scale)



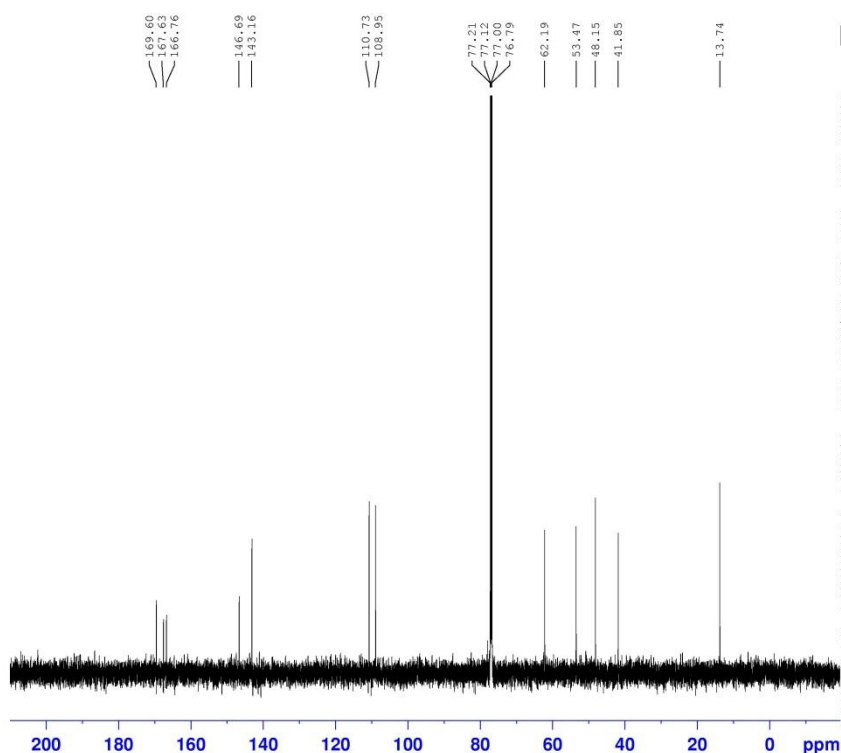


Current Data Parameters
NAME 7KMS59
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120305
Time 17.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 200.09
DW 60.800 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.38 usec
PLW1 11.1999981 W
SFO1 400.0924707 MHz

F2 - Processing parameters
SI 65536
SF 400.0900113 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



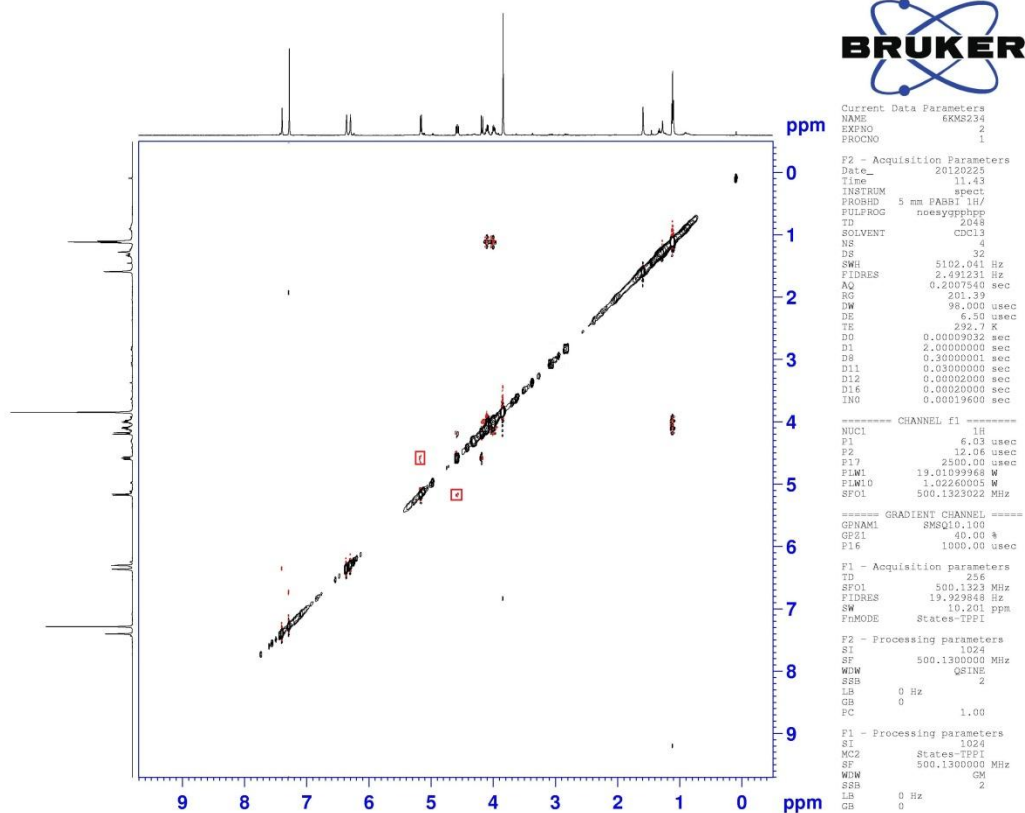
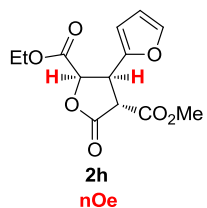
Current Data Parameters
NAME 7KMS59
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120309
Time 11.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 340
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 203
DW 13.867 usec
DE 10.00 usec
TE 295.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

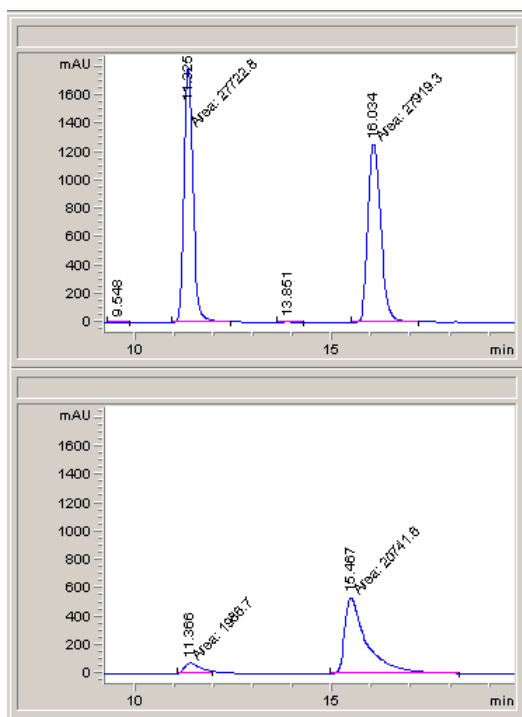
===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PLW1 110.0000000 W
SFO1 150.9178981 MHz

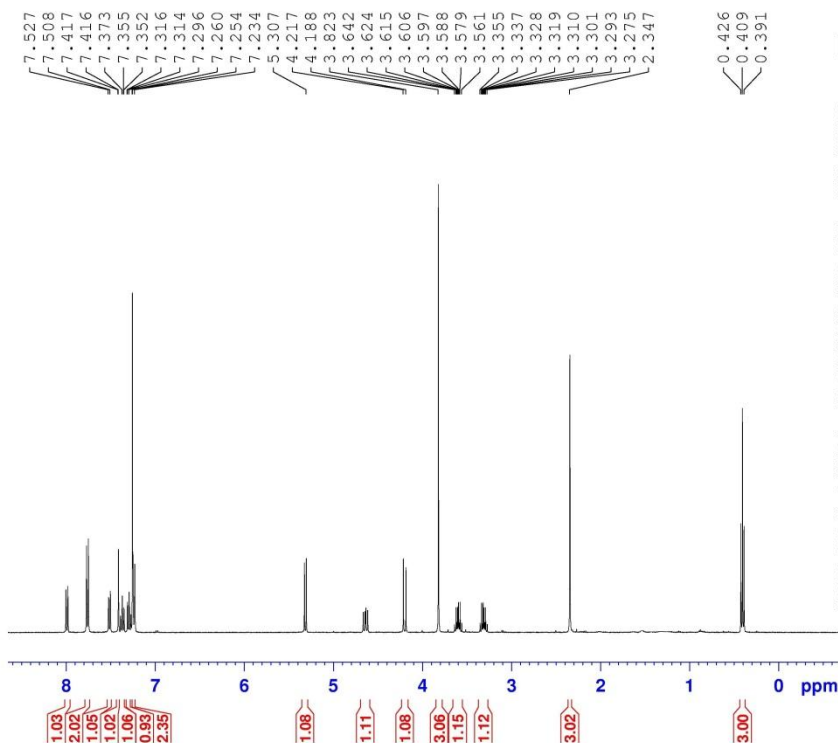
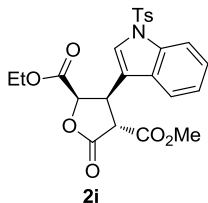
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PLW2 20.0000000 W
PLW12 0.8000000 W
PLW13 0.3919999 W
SFO2 600.1324005 MHz

F2 - Processing parameters
SI 32768
SF 150.9028140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



HPLC Analysis of 2h:



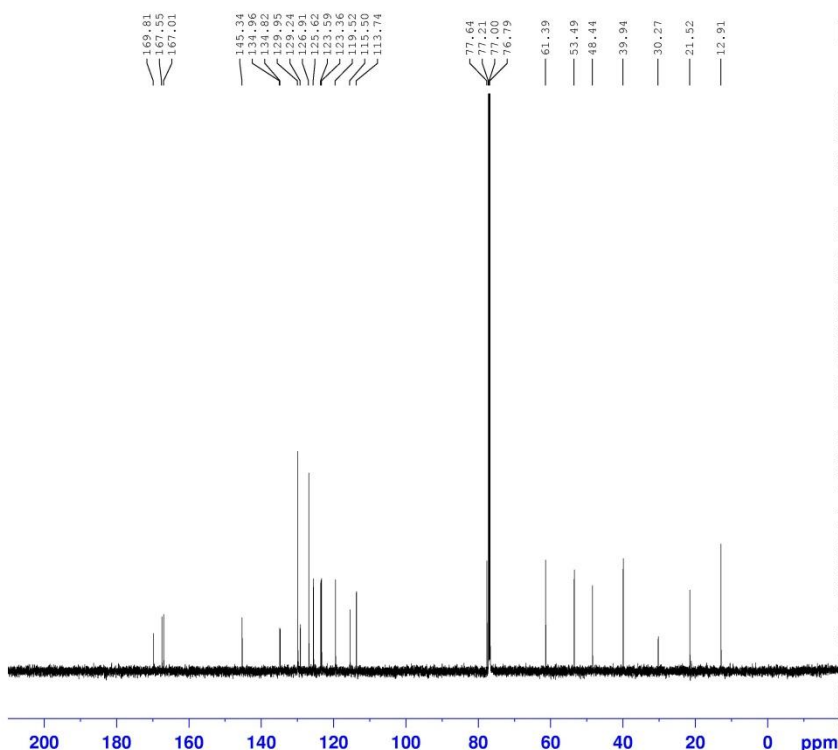


Current Data Parameters
NAME 7KM26
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120215
Time 15.19
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 200.09
DW 60.800 usec
DE 6.50 usec
TE 301.6 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.38 usec
PLW1 11.19999981 W
SFO1 400.0924707 MHz

F2 - Processing parameters
SI 65536
SF 400.0900113 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



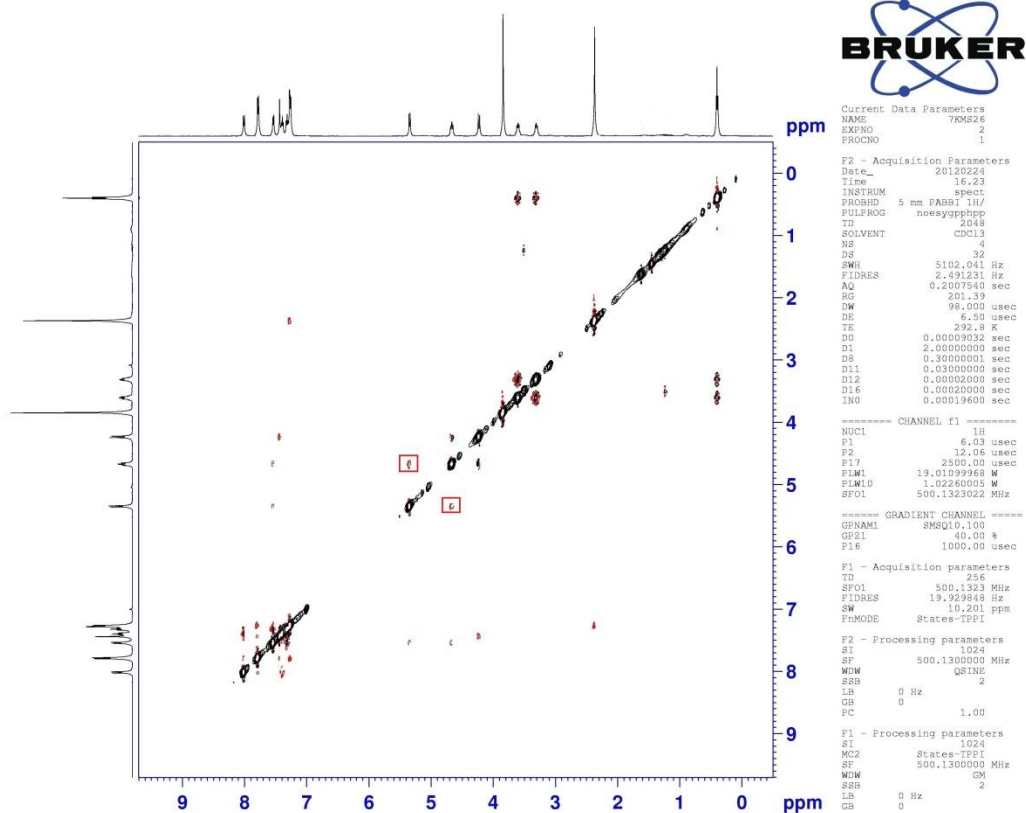
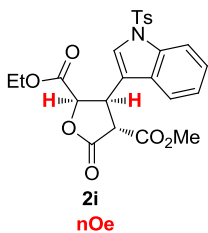
Current Data Parameters
NAME 7KM26
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120315
Time 17.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 172
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 203
DW 13.867 usec
DE 10.00 usec
TE 295.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

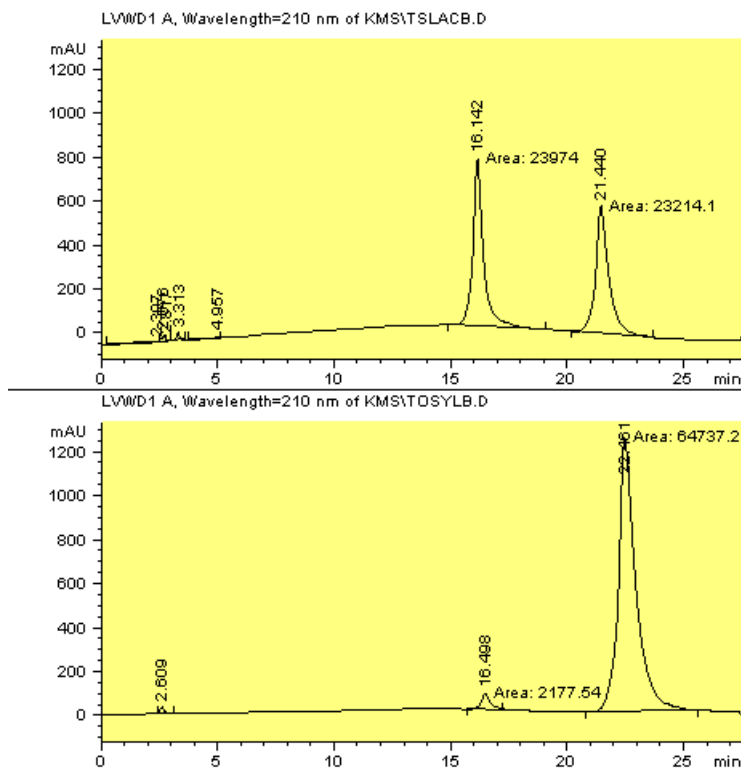
===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PLW1 110.00000000 W
SFO1 150.9178981 MHz

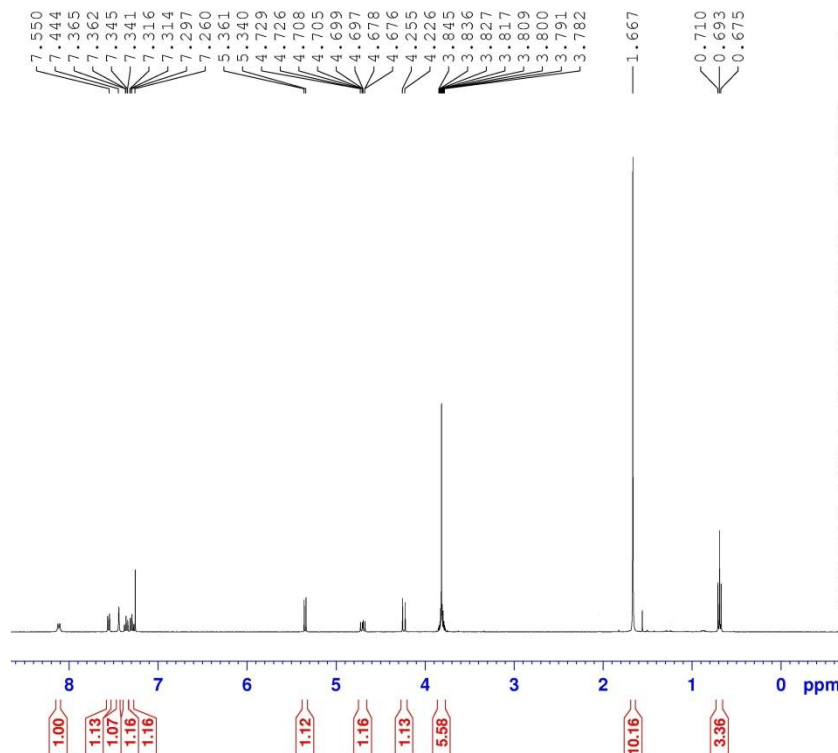
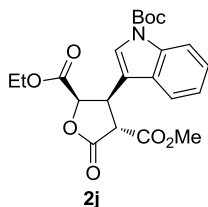
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PLW2 20.00000000 W
PLW12 0.80000001 W
PLW13 0.39199999 W
SFO2 600.1324005 MHz

F2 - Processing parameters
SI 32768
SF 150.9028173 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



SFC Analysis of 2i:



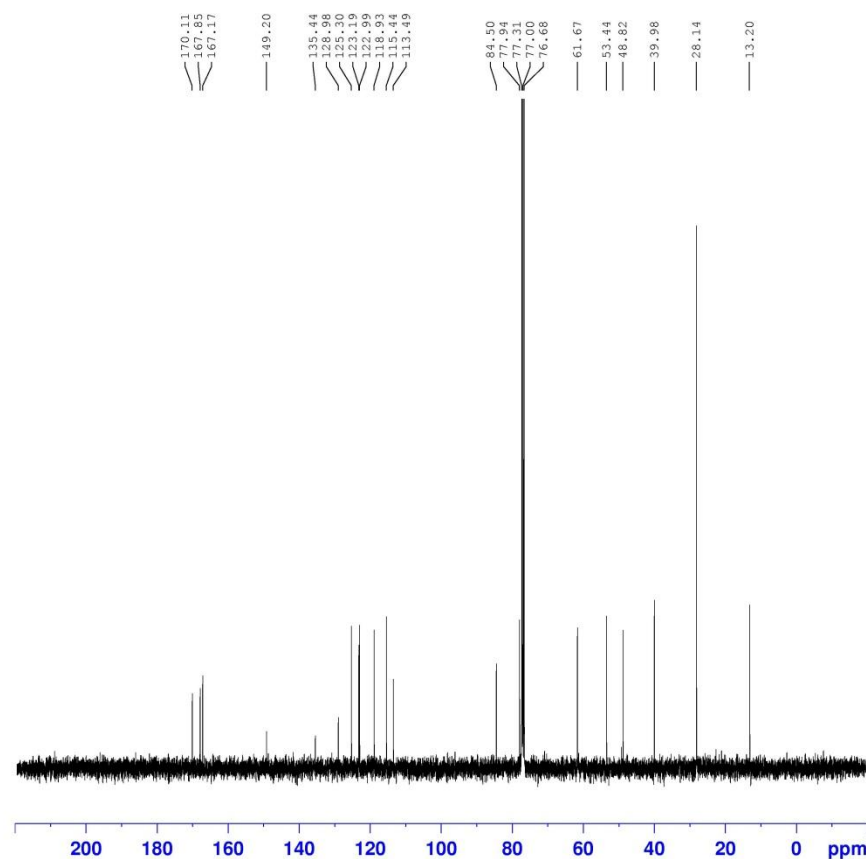


Current Data Parameters
NAME 7KMS26
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120218
Time 12.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 134.63
DW 60.800 usec
DE 6.50 usec
TE 297.4 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.38 usec
PLW1 11.19999981 W
SFO1 400.0924707 MHz

F2 - Processing parameters
SI 65536
SF 400.0900114 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



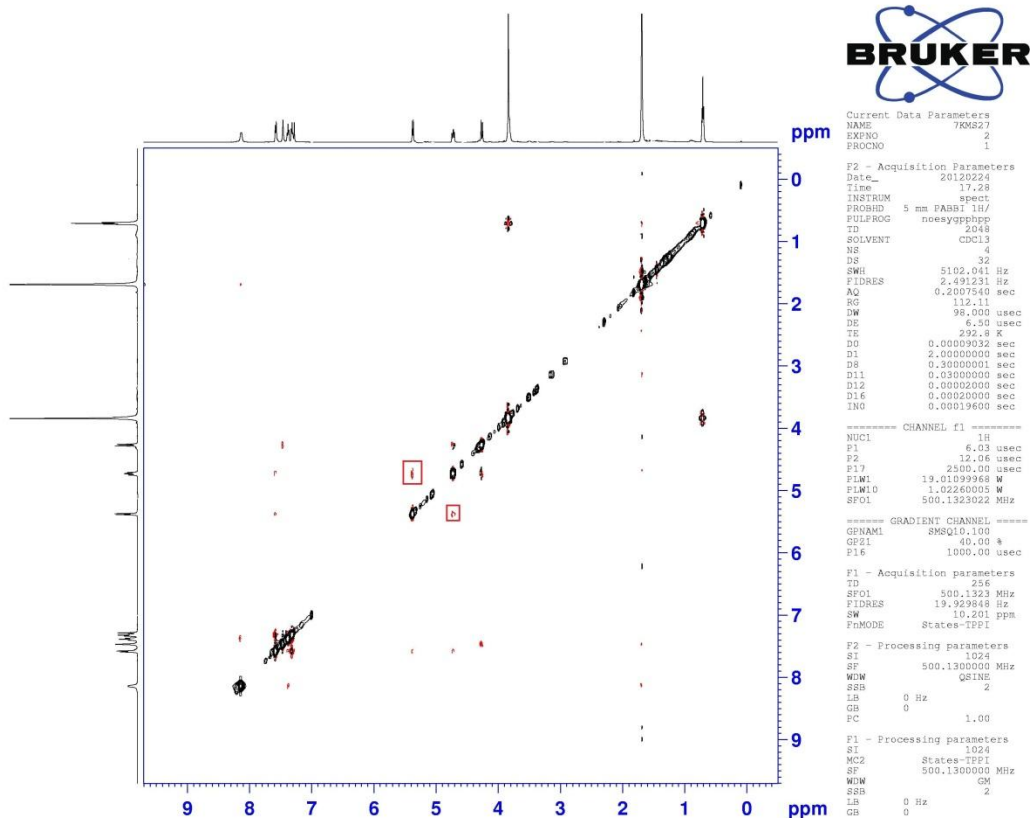
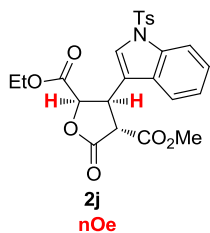
Current Data Parameters
NAME 7KMS26
EXPNO 2
PROCNO 1

F2 - Acquisition Parameter
Date_ 20120218
Time 12.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 559
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 se
RG 200.09
DW 20.800 us
DE 6.50 us
TE 298.6 K
D1 2.00000000 se
D11 0.03000000 se
TD0 1

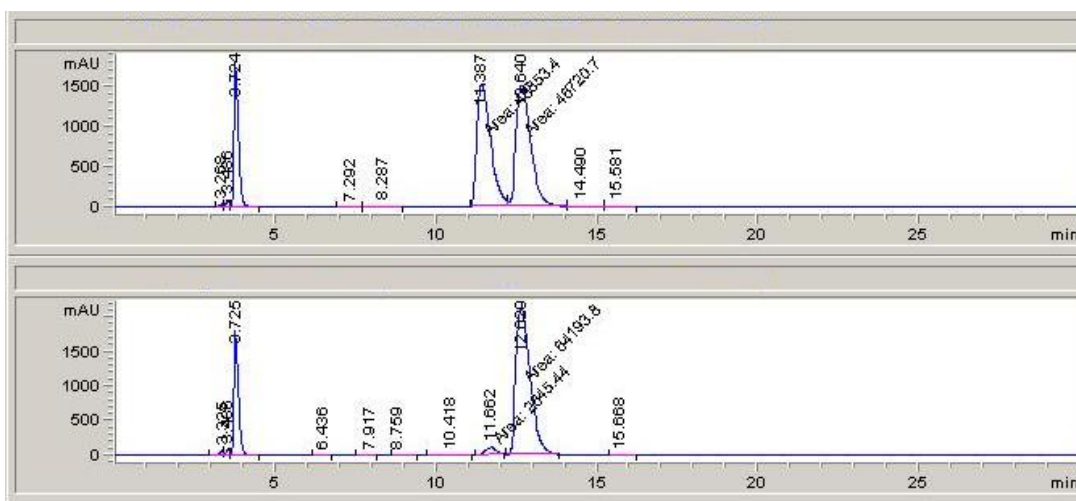
===== CHANNEL f1 =====
NUC1 13C
P1 7.50 us
PLW1 61.20000076 W
SFO1 100.6127703 MHz

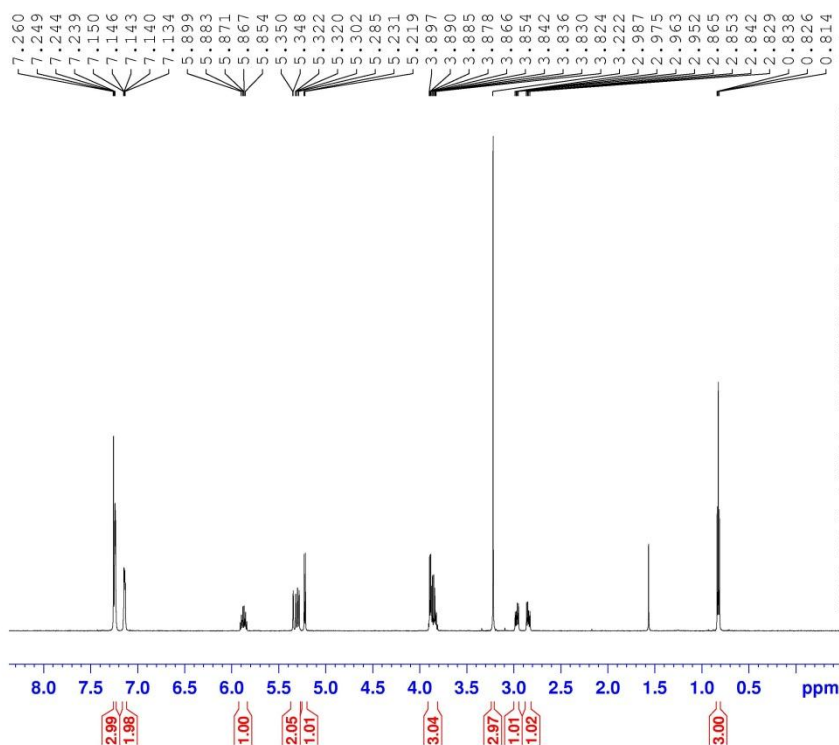
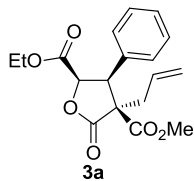
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 us
PLW2 11.19999981 W
PLW12 0.26820999 W
PLW13 0.17166001 W
SFO2 400.0916004 MHz

F2 - Processing parameters
SI 32768
SF 100.6027135 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



HPLC Analysis of 2j:



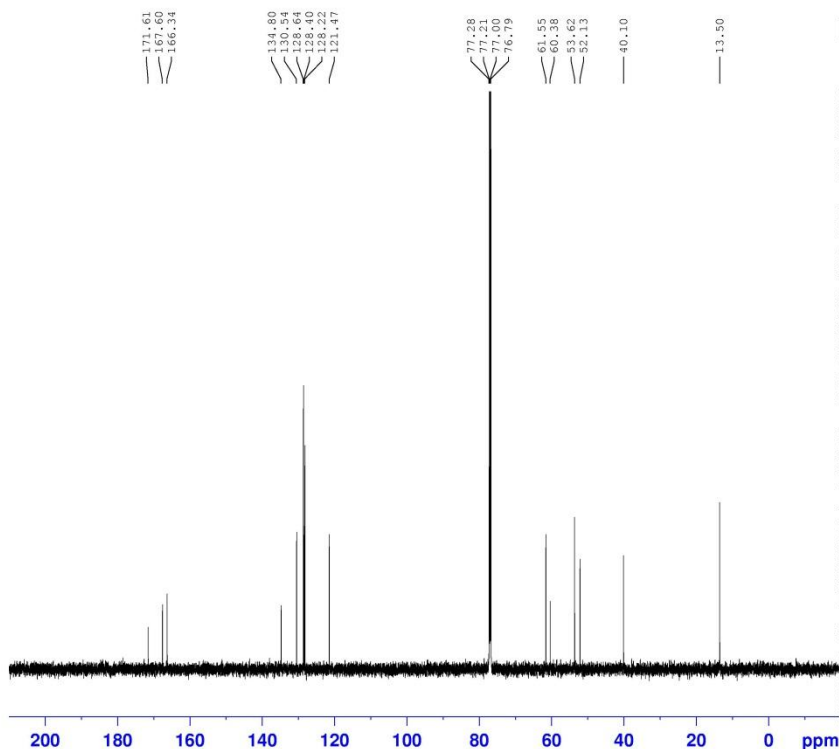


Current Data Parameters
NAME 6KMS240
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120305
Time 14.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 203
DW 40.533 usec
DE 6.50 usec
TE 293.5 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.60 usec
PLW1 20.00000000 W
SFO1 600.1337060 MHz

F2 - Processing parameters
SI 65536
SF 600.1300178 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



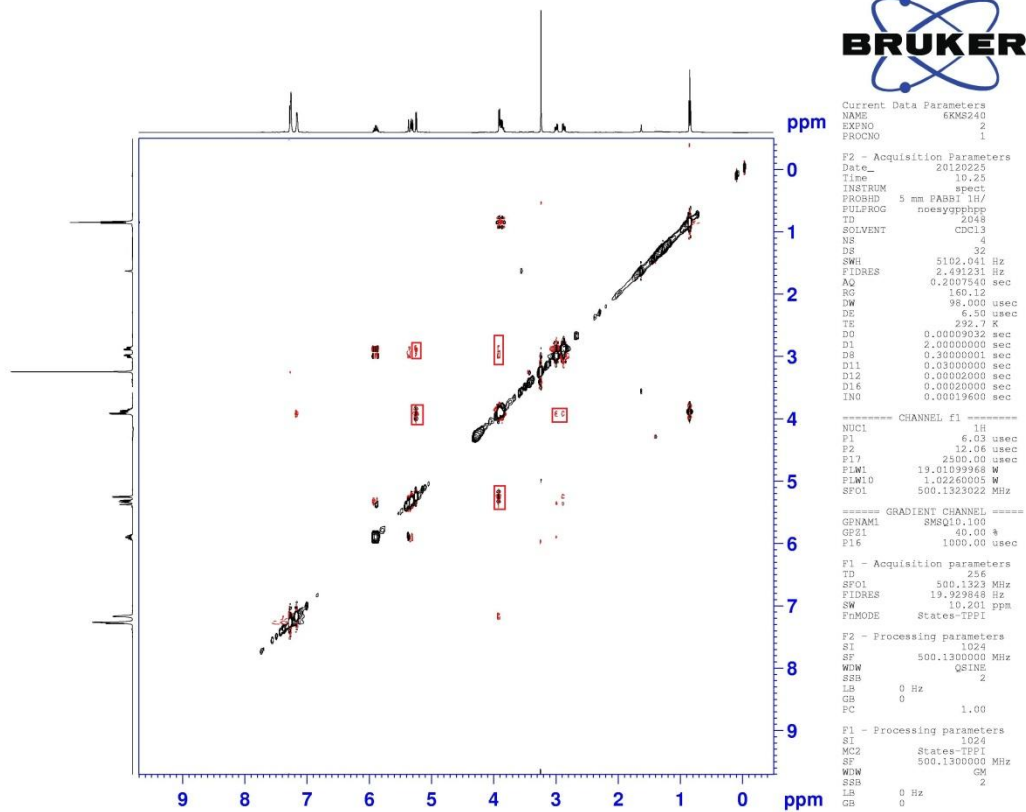
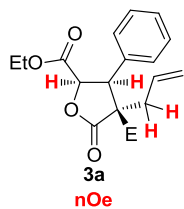
Current Data Parameters
NAME 6KMS240
EXPNO 4
PROCNO 1

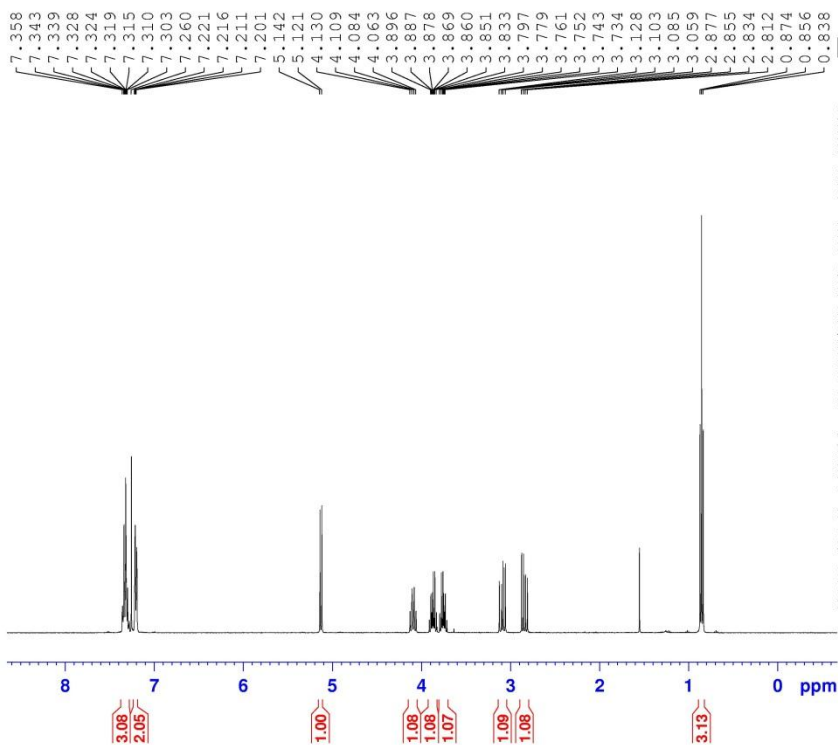
F2 - Acquisition Parameters
Date_ 20120305
Time 15.40
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 842
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 203
DW 13.867 usec
DE 10.00 usec
TE 295.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 10.50 usec
PLW1 110.00000000 W
SFO1 150.9178981 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PLW2 20.00000000 W
PLW12 0.80000001 W
PLW13 0.39199999 W
SFO2 600.1324005 MHz

F2 - Processing parameters
SI 32768
SF 150.9028139 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





```
Current Data Parameters
NAME      Decarboxylation
EXPNO      1
PROCNO     1
```

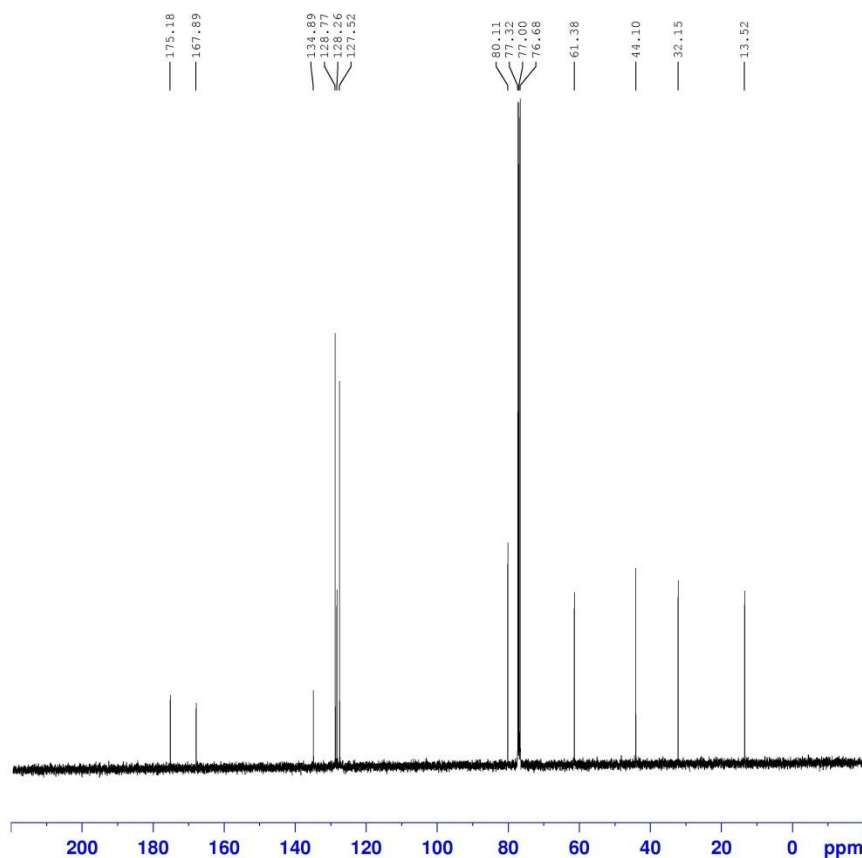
```

F2 - Acquisition Parameters
Date_      20120213
Time       19.05
INSTRUM    spect
PULPROG    5 mm PABBO BB-
TD          zg30
FIDRES     65536
SOLVENT    CDCl3
NS          16
DS          2
SWH         8223.685 Hz
FIDRES      0.125483 Hz
AQ          3.9846387 sec
RG          178.34
DW          60.800 usec
DE          5.50 usec
TE          301.6 K
D1          1.00000000 sec
TD0         1

```

```
===== CHANNEL f1 =====
NUC1                1H
P1                  12.38 usec
PLW1                11.19999981 W
SFO1                400.0924707 MHz
```

```
F2 - Processing parameters
SI                65536
SF                400.0900112 MHz
WDW               EM
SSB               0
LB                0.30 Hz
GB               0
PC                1.00
```



```
Current Data Parameters
NAME      Decarboxylation
EXPNO      2
PROCNO     1
```

```

F2 - Acquisition Parameters
Date_          20120213
Time           19.26
INSTRUM        spect
PROBHD         5 mm PABBO BB-
PULPROG        zgpg30
TD             65536
SOLVENT        CDC13
NS             306
DS             4
SWH            24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG            200.09
DW            20.800 us
DE            6.50 us
TE            302.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

```

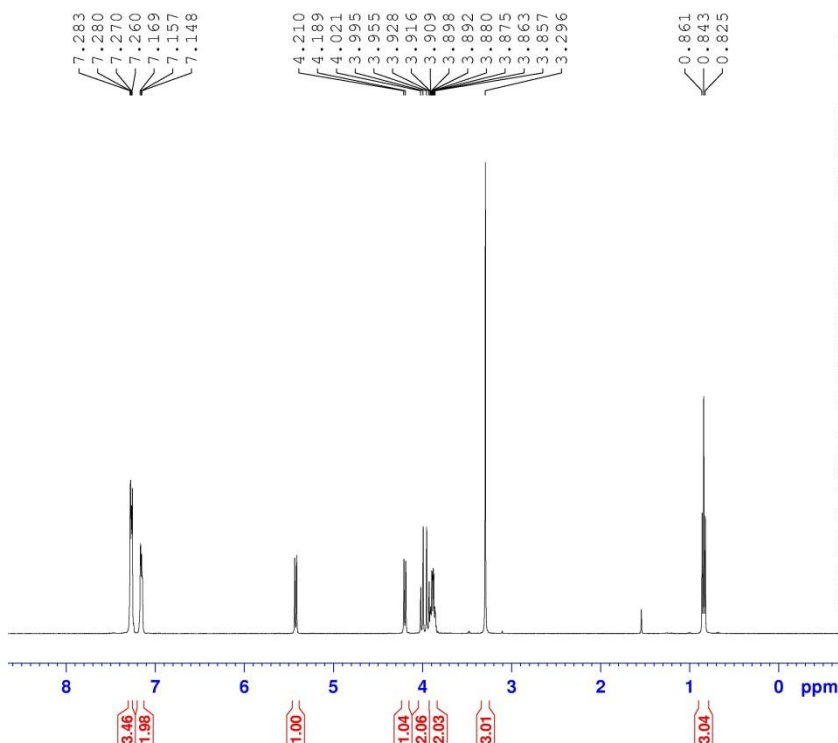
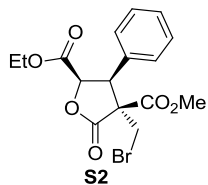
```
===== CHANNEL f1 =====
NUC1                13C
P1                  7.50 us
PLW1                61.20000076 W
SFO1                100.6127703 MHz
```

```

===== CHANNEL f2 =====
CPDPRG2          waltz16
NUC2              1H
PCPD2            80.00 us
PLW2            11.19999981 W
PLW12           0.26820999 W
PLW13           0.17166001 W
SFO2            400.0916004 MHz

```

F2 - Processing parameters		
SI		32768
SF		100.6027138 MHz
WDW		EM
SSB	0	
LB		1.00 Hz
GB	0	
PC		1.40

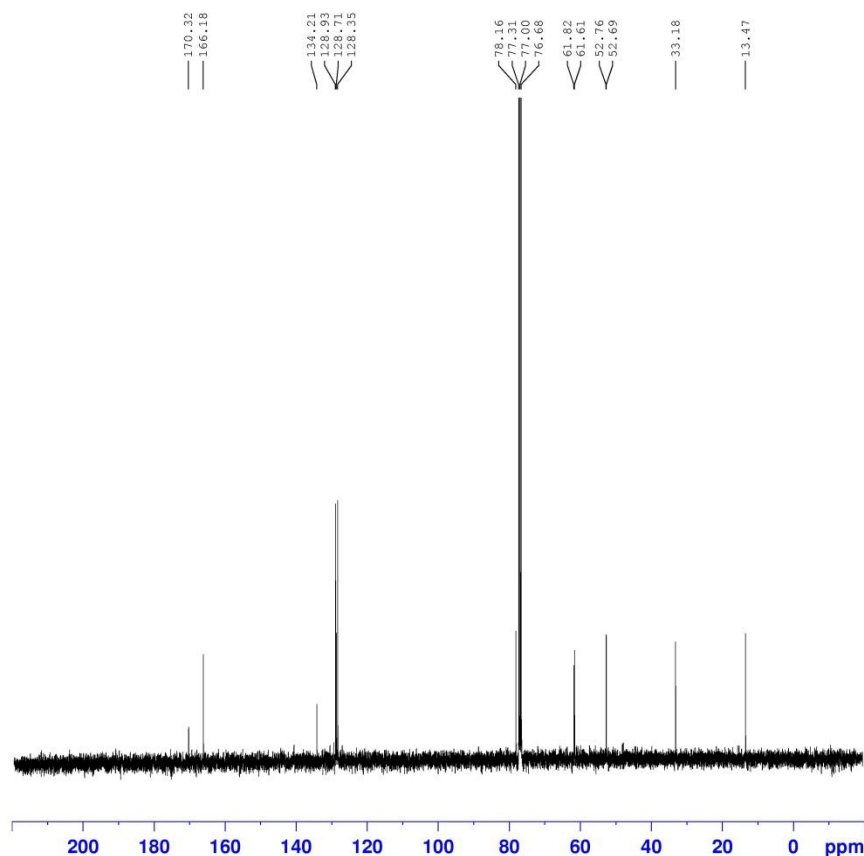


Current Data Parameters
 NAME 7KMS36
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20120215
 Time 18.25
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 178.34
 DW 60.800 usec
 DE 6.50 usec
 TE 301.9 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 12.38 usec
 PLW1 11.19999981 W
 SFO1 400.0924707 MHz

F2 - Processing parameters
 SI 65536
 SF 400.0900111 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



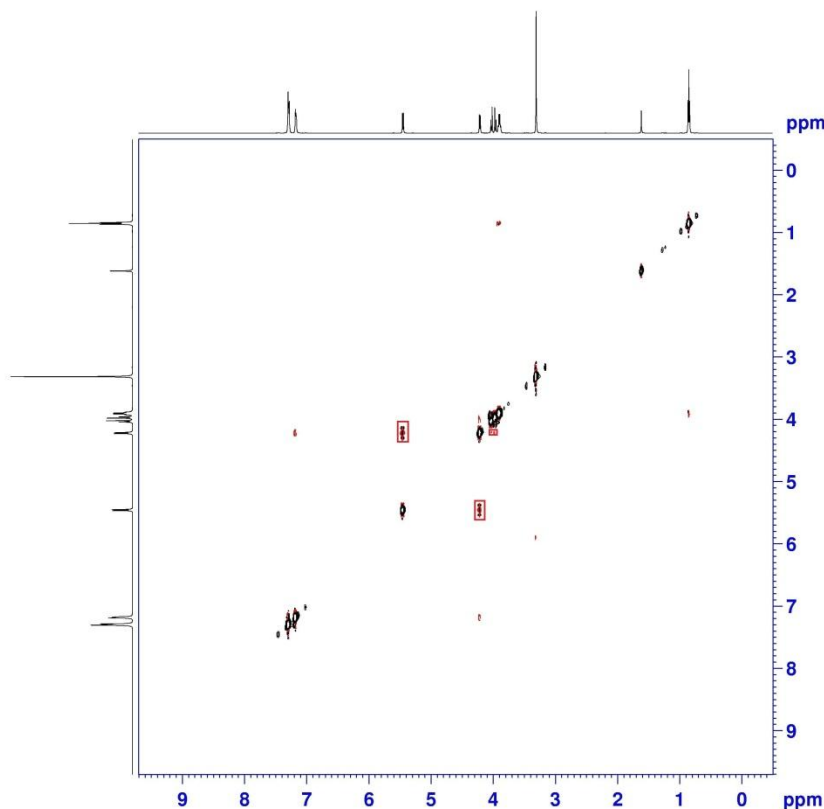
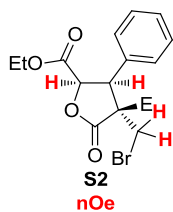
Current Data Parameters
 NAME 7KMS36
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameter
 Date_ 20120214
 Time 19.41
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 231
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 se
 RG 200.09
 DW 20.800 us
 DE 6.50 us
 TE 302.9 K
 D1 2.00000000 se
 D11 0.03000000 se
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 7.50 us
 PLW1 61.20000076 W
 SFO1 100.6127703 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 us
 PLW2 11.19999981 W
 PLW12 0.26820999 W
 PLW13 0.17166001 W
 SFO2 400.0916004 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6027127 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



Current Data Parameters
NAME 7KXG36
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120224
Time 12:31
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG noesypphpgp
TD 2048
SOLVENT CDCl3
NS 4
DS 32
SWH 5102.041 Hz
FIDRES 2.491231 Hz
AQ 0.2007540 sec
RG 201.39
DW 98.000 usec
DE 6.50 usec
TE 290.2 K
D0 0.00000032 sec
D1 2.00000000 sec
D8 0.30000001 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00019600 sec

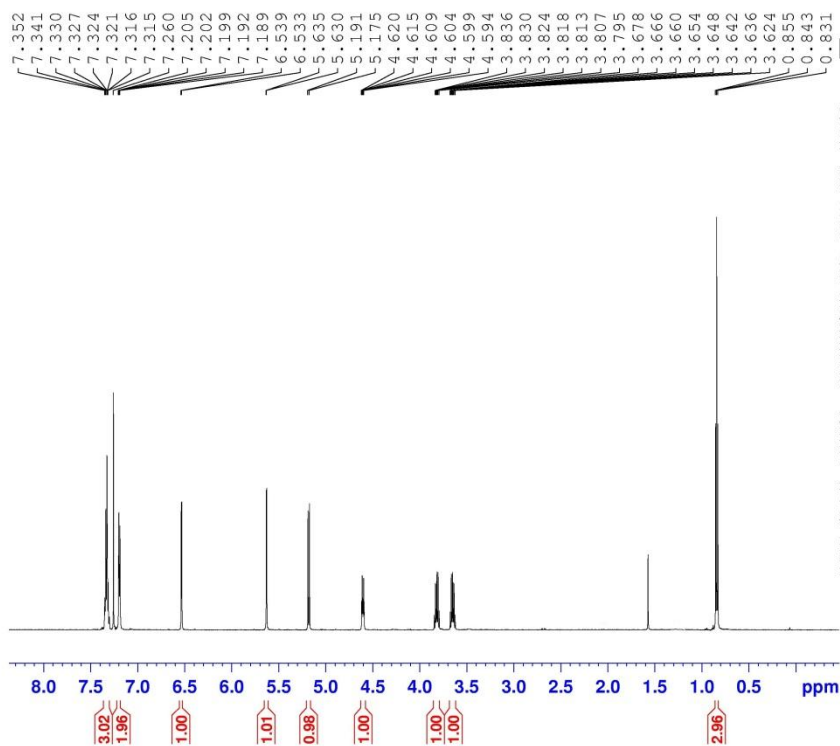
===== CHANNEL f1 =====
NUC1 1H
P1 6.03 usec
P2 12.06 usec
P17 2500.00 usec
PLM1 19.01099968 W
PLM10 1.02260005 W
SFO1 500.1323022 MHz

===== GRADIENT CHANNEL =====
GENAM1 SMSQ10.100
GFG1 40.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 256
SFO1 500.1323 MHz
FIDRES 19.929848 Hz
SW 10.201 ppm
FMODE States-TPPI

F2 - Processing parameters
SI 1024
SF 500.1300000 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 States-TPPI
SF 500.1300000 MHz
WDW GM
SSB 2
LB 0 Hz
GB 0



```
Current Data Parameters
NAME      Dehalodecarboxylation
EXPNO      1
PROCNO     1
```

```

F2 - Acquisition Parameters
Date_      20120213
Time       15.37
INSTRUM    spect
PROBH      5 mm PABBO B5
PULPROG    zg30
TD          65536
SOLVENT    CDCl3
NS          16
DS          2
SWH         12335.526 Hz
FIDRES      0.188225 Hz
AQ          2.6564426 sec
RG          203
DE          40.53 usec
DW          6.50 usec
TE          293.1 K
D1          1.00000000 sec
TDO         1

```

```

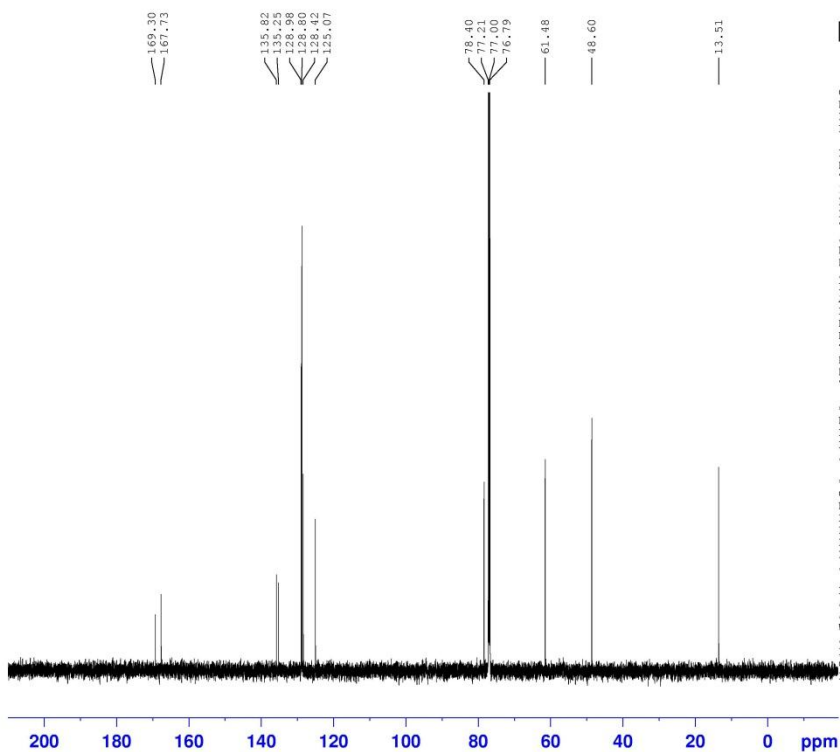
----- CHANNEL f1 -----
NUC1              1H
P1                13.60 usec
PLW1             20.00000000 W
SFO1             600.1337060 MHz

```

```

F2 - Processing parameters
SI                65536
SF                600.1300180 MHz
WDW               EM
SSB               0
LB                0.30 Hz
GB               0
PC                1.00

```



```
Current Data Parameters
NAME      Dehalodecarboxylation
EXPNO      2
PROCNO     1
```

```

F2 - Acquisition Parameters
Time_                20120213
Date_                16.00
INSTRUM              spect
PROBHD      5 mm PABBO BB-
PULPROG              zgpg30
TD                   65536
SOLVENT              CDC13
NS                   510
DS                   4
SWH                  36057.691 Hz
FIDRES              0.550197 Hz
AQ                   0.908815 sec
RG                   203
DW                   13.867 use
DE                   10.00 use
TE                   294.6 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                   1

```

```
===== CHANNEL f1 =====
NUC1                13C
P1                  10.50 usec
PLW1                110.00000000 W
SFO1                150.9178981 MHz
```

```

    CHANNEL f2
CPDPRG2          waltz16
NUC2             1H
PCPD2           70.00 usec
PLW2            20.00000000 W
PLW12           0.80000001 W
PLW13           0.39199999 W
SFO2            600.1324005 MHz

```

```

F2 - Processing parameters
SI                      32768
SF                      150.9028140 MHz
WDW                      EM
SSB      0
LB                      1.00 Hz
GB      0
PC                      1.40

```

