Supporting Information

Asymmetric Total Syntheses of Megacerotonic Acid and Shimobashiric Acid A

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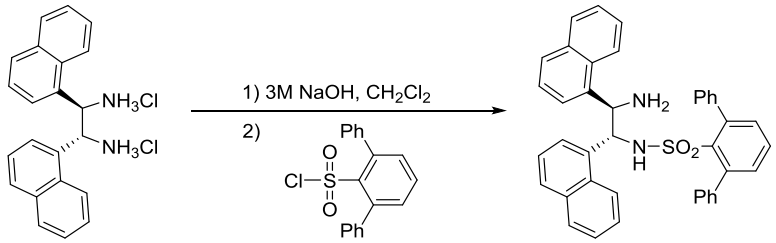
General Information

Methods: Infrared (IR) spectra were obtained using a Jasco 260 Plus Fourier transform infrared spectrometer. Proton and carbon nuclear magnetic resonance spectra (^{1}H NMR and ^{13}C NMR) were recorded on a Bruker DRX 400 or 600 (^{1}H NMR at 400 MHz or 600 MHz and ^{13}C NMR at 100 or 150 MHz) spectrometer with solvent resonance as the internal standard (^{1}H NMR: $CDCl_3$ at 7.26 ppm, acetone-d_6 at 2.05 ppm, methanol-d_4 at 3.30 ppm, D_2O at 4.79 ppm; ^{13}C NMR: $CDCl_3$ at 77.0 ppm, acetone-d_6 at 29.8 ppm, methanol-d_4 at 49.0) or tetramethylsilane as the internal standard at 0.00 ppm. ^{1}H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, bs = broad singlet, d = doublet, dd = doublet of doublet, dt = doublet of triplets, ddd = doublet of doublet of doublets, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), and integration. Supercritical fluid chromatography was performed on a Berger SFC system equipped with Chiralcel AD, AS, OD, and WO columns (φ 4.6 mm x 250 mm). Samples were eluted with SFC grade CO_2 at the indicated percentage of MeOH with an oven temperature of 40 °C. 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 Thermo Scientific LTQ FT Ultra instrument with electrospray ionization (Note: All samples prepared in methanol with ammonium acetate, formic acid, or cesium acetate additives). Melting point data was collected on a Thomas Hoover Capillary Melting Point Apparatus and are uncorrected. Analytical thin layer chromatography (TLC) was performed on Sorbent Technologies Silica G 0.20 mm silica gel plates. Visualization was accomplished with UV light, aqueous basic potassium permanganate solution, or Seebach's TLC stain followed by heating. Flash chromatography was performed using Silia-P flash silica gel (40-63 μm) purchased from Silicycle. Yield refers to isolated yield of analytically pure material unless otherwise noted.

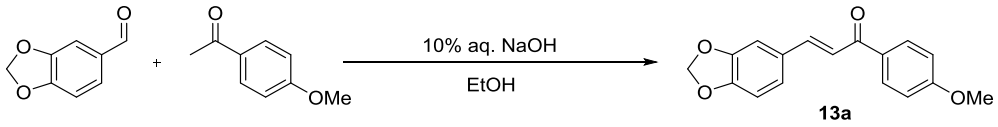
Materials: Triethylamine (Et_3N) and *tert*-amyl alcohol (^{t}AmylOH) were freshly distilled from calcium hydride prior to use. Sodium *tert*-amylate ($NaO^{t}Amyl$) solution was prepared by reaction of sodium metal (cut into thin strips) with freshly distilled *tert*-amyl alcohol at 50 °C. Dichloromethane (CH_2Cl_2), diethyl ether (Et_2O), tetrahydrofuran (THF), and toluene were dried by passage through a column of neutral alumina under nitrogen prior to use. Ethyl glyoxylate was purchased from Sigma Aldrich or Alfa Aesar and distilled according to a literature procedure immediately before use (the concentration after distillation was determined by ^{1}H NMR spectroscopy).[1] (*R,R*)-1,2-di(naphthyl)-1,2-ethanediamine-dihydrochloride salt[2] and *m*-terphenyl sulfonyl chloride[3] were prepared according to literature procedures. All other reagents were purchased from commercial sources and used as received unless otherwise noted. Anhydrous dimethylformamide (DMF) and dimethylsulfoxide (DMSO) were purchased from Sigma Aldrich or Fisher Scientific.

Preparation of (*R,R*)-*N*-(2-amino-1,2-di(naphthalene-1-yl)ethyl)-[*m*-terphenyl]-2′-sulfonamide, L1



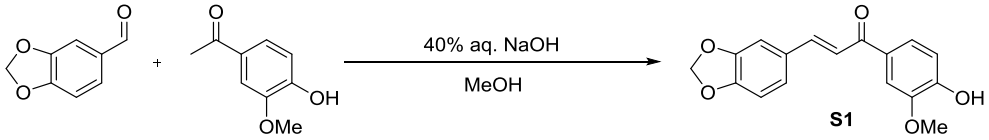
To a 100 mL round-bottomed flask equipped with oversized magnetic stir bar containing 3M NaOH (18 mL) and dichloromethane (18 mL) was added (*R,R*)-1,2-di(naphthyl)-1,2-ethanediamine-dihydrochloride salt (1.38 g, 3.58 mmol, 1.0 equiv). The resulting mixture was stirred vigorously for 14 h. The reaction was cooled to 0 °C and *m*-terphenyl sulfonyl chloride (1.18 g, 3.58 mmol, 1.0 equiv) was added. The reaction was allowed to warm to room temperature over 30 min. After 1.5 h at room temperature, the reaction was diluted with water (15 mL) and extracted with ethyl acetate (2 x 20 mL). The combined organic extracts were washed with brine, dried (Na_2SO_4) and concentrated. Flash chromatography (50% EtOAc/hexanes) provided **L1** (1.04 g, 1.72 mmol, 48%) as an off-white solid. Analytical data for **L1** matched that previously reported.[3]

Preparation of Enone 13a



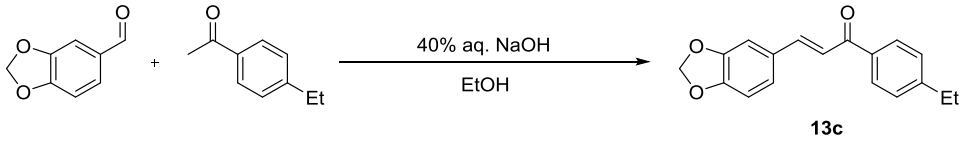
To a solution of piperonal (12.29 g, 81.9 mmol, 1.0 equiv) and 4′-methoxyacetophenone (12.29 g, 81.9 mmol, 1.0 equiv) in ethanol (40 mL) was added 10% aqueous sodium hydroxide (2.4 mL). After 22 h, the yellow suspension was diluted with ethanol/water (50/50, 50 mL). Suction filtration and washing with ethanol/water (50/50, 150 mL) provided **13a** (22.7 g, 80.3 mmol, 98% yield) as a yellow solid. Analytical data for **13a**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.06 – 7.96 (m, 2H), 7.72 (d, *J* = 15.5 Hz, 1H), 7.38 (d, *J* = 15.5 Hz, 1H), 7.16 (d, *J* = 1.7 Hz, 1H), 7.14 – 7.09 (m, 1H), 6.97 (d, *J* = 8.8 Hz, 2H), 6.83 (d, *J* = 8.0 Hz, 1H), 6.02 (s, 2H), 3.88 (s, 3H).; **^{13}C NMR** (150 MHz, $CDCl_3$) δ 188.5, 163.3, 149.7, 148.3, 143.8, 131.2, 130.7, 129.5, 125.0, 119.8, 113.8, 108.6, 106.6, 101.6, 55.5; **IR** (thin film, cm^{-1}): 1652, 1602, 1506, 1036; **m.p.** 130-131 °C; **TLC** (20% EtOAc/hexanes) R_f : 0.22; **HRMS** (ESI): Calculated for [M+H]+ $C_{17}H_{15}O_4$: 283.0970, Found: 283.0965.

Preparation of Enone S1



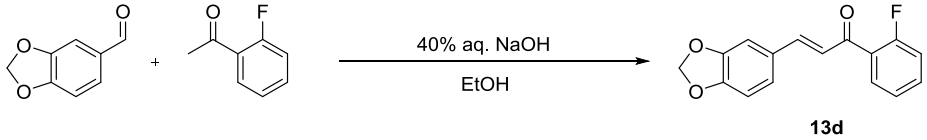
To a solution of piperonal (750 mg, 5.0 mmol, 1.0 equiv) and acetovanillone (830 mg, 5.0 mmol, 1.0 equiv) in methanol (15 mL) was added 40% aqueous sodium hydroxide (5 mL). After 25 h, the reaction was diluted with water (20 mL) and brought to pH = 0 with concentrated hydrochloric acid resulting in warming of the reaction solution. After cooling to room temperature and stirring for 4.5 h, suction filtration provided **S1** (692 mg, 2.3 mmol, 46% yield) as a bright yellow solid. Analytical data for **S1** matched that previously reported.[4]

Preparation of Enone 13c



To a solution of piperonal (2.25 g, 15.0 mmol, 1.0 equiv) and 4′-ethylacetophenone (2.22 g, 15.0 mmol, 1.0 equiv) in ethanol (40 mL) was added 40% aqueous sodium hydroxide (1.5 mL). After 15 h, the yellow suspension was diluted with water (30 mL). Suction filtration and washing with ethanol/water (50/50, 40 mL) provided **13c** (3.95 g, 14.1 mmol, 94% yield) as a yellow solid. Analytical data for **13c**: **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.98 – 7.93 (m, 2H), 7.73 (d, *J* = 15.6 Hz, 1H), 7.38 (d, *J* = 15.5 Hz, 1H), 7.34 – 7.30 (m, 2H), 7.17 (d, *J* = 1.7 Hz, 1H), 7.12 (dd, *J* = 8.0, 1.7 Hz, 1H), 6.85 (d, *J* = 8.0 Hz, 1H), 6.03 (s, 2H), 2.73 (q, *J* = 7.6 Hz, 2H), 1.28 (t, *J* = 7.6 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 189.9, 149.8, 149.6, 148.4, 144.2, 136.0, 129.5, 128.6, 128.1, 125.1, 120.2, 108.6, 106.6, 101.6, 29.0, 15.2; **IR** (thin film, cm^{-1}): 1652, 1580, 1500, 1362, 991; **m.p.** 105-106 °C; **TLC** (20% EtOAc/hexanes) R_f : 0.38; **HRMS** (ESI): Calculated for [M+H]+ $C_{18}H_{17}O_3$: 281.1178, Found: 281.1173.

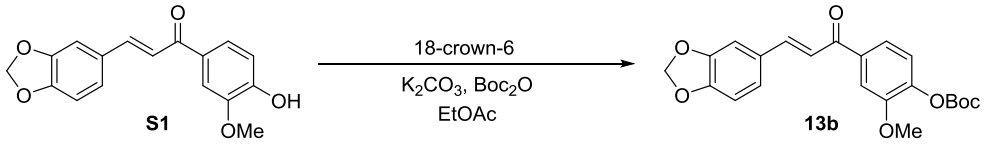
Preparation of Enone 13d



To a solution of piperonal (1.50 g, 9.99 mmol, 1.0 equiv) and 2′-fluoroacetophenone (1.38 g, 9.99 mmol, 1.0 equiv) in ethanol (15 mL) was added 40% aqueous sodium hydroxide (1 mL). After 5 h, the yellow suspension was diluted with water (45 mL). Suction filtration provided **13d**

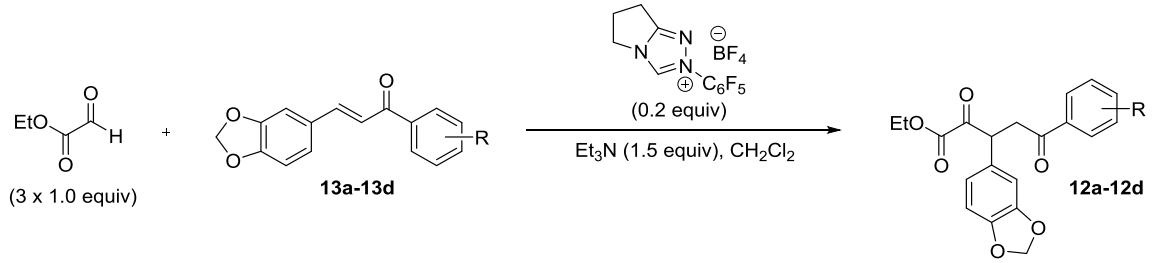
(2.56 g, 9.47 mmol, 95% yield) as a yellow solid. Analytical data for **13d**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.80 (td, *J* = 7.5, 1.9 Hz, 1H), 7.66 (dd, *J* = 15.7, 1.8 Hz, 1H), 7.51 (dddd, *J* = 8.3, 7.1, 5.0, 1.8 Hz, 1H), 7.28 – 7.13 (m, 4H), 7.12 – 7.09 (m, 1H), 6.84 (d, *J* = 8.0 Hz, 1H), 6.02 (s, 2H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 188.9 (d, *J* = 2.4 Hz), 161.1 (d, *J* = 253.0 Hz), 150.1, 148.4, 144.8, 133.7 (d, *J* = 8.8 Hz), 130.9 (d, *J* = 2.7 Hz), 129.2, 127.3 (d, *J* = 13.4 Hz), 125.5, 124.5 (d, *J* = 3.4 Hz), 123.7 (d, *J* = 6.6 Hz), 116.5 (d, *J* = 23.2 Hz), 108.7, 106.8, 101.6; **IR** (thin film, cm^{-1}): 1655, 1454, 930, 766; **m.p.** 127-128 °C; **TLC** (20% EtOAc/hexanes) R_f : 0.35; **HRMS** (ESI): Calculated for [M+H]+ $C_{16}H_{12}FO_3$: 271.0770, Found: 271.0765.

***Tert*-Butoxycarbonylation of Enone 13b**



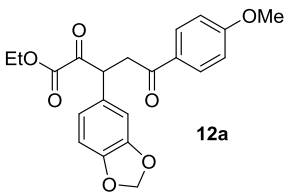
To 50 mL round-bottomed flask equipped with magnetic stir bar containing a semi-solution of enone **S1** (676 mg, 2.27 mmol, 1.0 equiv) and 18-crown-6 (60 mg, 0.23 mmol, 0.10 equiv) in ethyl acetate (10 mL) was added potassium carbonate (471 mg, 3.41 mmol, 1.5 equiv) followed by di-*tert*-butyl dicarbonate (495 mg, 2.27 mmol, 1.0 equiv). The resulting orange suspension was stirred at room temperature. After 27 h, the reaction was partitioned between ethyl acetate (25 mL) and water (25 mL). The aqueous layer was extracted with ethyl acetate (10 mL). The combined organic extracts were washed with brine (15 mL), dried (Na_2SO_4) and concentrated to provide **13b** (904 mg, 2.27 mmol, 100% yield) as a crystalline, yellow solid. Analytical data for **13b**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.74 (d, *J* = 15.5 Hz, 1H), 7.64 (d, *J* = 1.9 Hz, 1H), 7.60 (dd, *J* = 8.2, 1.9 Hz, 1H), 7.33 (d, *J* = 15.5 Hz, 1H), 7.24 (d, *J* = 8.2 Hz, 1H), 7.16 (d, *J* = 1.7 Hz, 1H), 7.12 (dd, *J* = 8.2, 1.7 Hz, 1H), 6.85 (d, *J* = 8.0 Hz, 1H), 6.03 (s, 2H), 3.94 (s, 3H), 1.56 (s, 9H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ 189.0, 151.6, 150.8, 150.0, 148.4, 144.9, 143.8, 137.0, 129.3, 125.3, 122.3, 121.5, 119.7, 112.1, 108.7, 106.6, 101.6, 83.9, 56.1, 27.6; **IR** (thin film, cm^{-1}): 1761, 1661, 1585, 1506, 805; **m.p.** 141-143 °C; **TLC** (20% EtOAc/hexanes) R_f : 0.22; **HRMS** (ESI): Calculated for [M+H]+ $C_{22}H_{23}O_7$: 399.1444, Found: 399.1440.

General Procedure A for the Preparation of β-Substituted-α,δ-Diketo Esters 12a-d



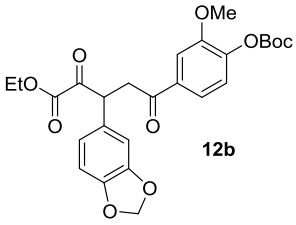
To a flame-dried round-bottomed flask equipped with magnetic stir bar were added enone **13a-d** (1.0 equiv), triazolium salt (0.2 equiv), and freshly distilled ethyl glyoxylate solution (1.0 equiv, ~90% in toluene). The flask was sealed with a rubber septum and purged with nitrogen. Dichloromethane (0.5 M) followed by triethylamine (1.5 equiv) were then added. After 1.5 h and

5 h additional ethyl glyoxylate solution (1.0 equiv, respectively) was added. After 24 h, the reaction was concentrated *in vacuo* and purified by flash chromatography using the indicated solvent system.



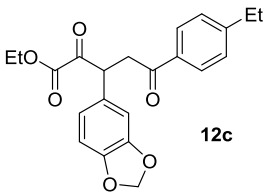
12a

Ethyl 3-(benzo[d][1,3]dioxol-5-yl)-5-(4-methoxyphenyl)-2,5-dioxopentanoate (12a): The title compound was prepared according to General Procedure A: enone **13a** (5.00 g, 17.7 mmol), triazolium salt (1.28 g, 3.53 mmol), ethyl glyoxylate (3 x 2.0 g, 3 x 17.7 mmol), dichloromethane (35.3 mL), triethylamine (3.69 mL, 26.5 mmol). Flash chromatography (20-40% EtOAc/hexanes) provided **12a** (6.70 g, contained 4% enone which could not be efficiently separated) as a clear, viscous oil. This material was reduced in its entirety (*vide infra*). In another experiment an analytically pure sample was obtained by careful selection of a clean fraction. Analytical data for **12a**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.98 – 7.70 (m, 2H), 6.98 – 6.87 (m, 2H), 6.83 (d, *J* = 1.7 Hz, 1H), 6.81 – 6.70 (m, 2H), 6.01 – 5.86 (m, 2H), 5.04 (dd, *J* = 10.6, 3.9 Hz, 1H), 4.43 – 4.20 (m, 2H), 3.91 (dd, *J* = 17.9, 10.6 Hz, 1H), 3.86 (d, *J* = 1.6 Hz, 3H), 3.36 (dd, *J* = 17.9, 3.9 Hz, 1H), 1.32 (t, *J* = 7.2 Hz, 2H). **^{13}C NMR** (150 MHz, $CDCl_3$): δ 195.8, 192.0, 163.8, 160.4, 148.2, 147.3, 130.4, 128.9, 128.8, 122.3, 113.7, 109.1, 108.7, 101.2, 62.5, 55.5, 47.8, 42.9, 13.9; **IR** (thin film, cm^{-1}): 2906, 1728, 1670, 1601, 1488, 1171, 1038; **TLC** (30% EtOAc/hexanes) R_f : 0.29; **HRMS** (ESI): Calculated for [M+Cs]+ $C_{21}H_{20}CsO_7$: 517.0263, Found: 517.0265.



12b

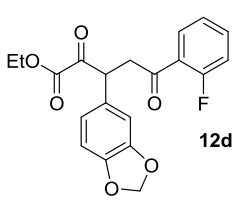
Ethyl 3-(benzo[d][1,3]dioxol-5-yl)-5-(4-((tert-butoxycarbonyl)oxy)-3-methoxyphenyl)-2,5-dioxopentanoate (12b): The title compound was prepared according to General Procedure A: enone **13b** (850 mg, 2.13 mmol), triazolium salt (155 mg, 0.426 mmol), ethyl glyoxylate (3 x 242 mg, 3 x 0.426 mmol), dichloromethane (4.2 mL), triethylamine (0.45 mL, 3.2 mmol). Flash chromatography (20-25% EtOAc/hexanes) provided **12b** (1.01 g, 2.02 mmol, 95% yield) as pale yellow solid. Analytical data for **12b**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.56 – 7.53 (m, 2H), 7.22 – 7.14 (m, 1H), 6.81 (d, *J* = 1.5 Hz, 1H), 6.80 – 6.74 (m, 2H), 5.94 (d, *J* = 2.6 Hz, 2H), 5.04 (dd, *J* = 10.4, 4.0 Hz, 1H), 4.33 – 4.24 (m, 2H), 3.91 (dd, *J* = 18.1, 10.4 Hz, 1H), 3.88 (s, 3H), 3.38 (dd, *J* = 18.1, 4.0 Hz, 1H), 1.54 (s, 9H), 1.32 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 196.2, 191.8, 160.3, 151.5, 150.6, 148.2, 147.4, 144.4, 134.5, 128.4, 122.4, 122.4, 121.5, 111.5, 109.0, 108.8, 101.2, 84.0, 62.5, 56.0, 47.9, 42.9, 27.5, 13.9; **IR** (thin film, cm^{-1}): 2982, 1762, 1730, 1683, 1506, 1488, 1146, 1039, 738; **m.p.** 108-109 °C **TLC** (30% EtOAc/hexanes) R_f : 0.29; **HRMS** (ESI): Calculated for [M+H]+ $C_{26}H_{29}O_{10}$: 501.1761, Found: 501.1758.



12c

Ethyl 3-(benzo[d][1,3]dioxol-5-yl)-5-(4-ethylphenyl)-2,5-dioxopentanoate (12c): The title compound was prepared according to General Procedure A: enone **13c** (3.90 g, 13.9 mmol), triazolium salt (1.01 g, 2.78 mmol), ethyl glyoxylate (3 x 1.58 g, 3 x 13.9 mmol), dichloromethane (28 mL), triethylamine (2.9 mL, 21 mmol). Flash chromatography (10-15% EtOAc/hexanes) provided **12c** (5.30 g, 13.9 mmol, 99% yield) as a viscous yellow oil. Analytical data for **12c**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.87 (d, *J* = 8.3 Hz, 2H), 7.28 – 7.26 (d, *J* = 8.4 Hz, 2H), 6.83 (d, *J* = 1.8 Hz, 1H), 6.81 – 6.73

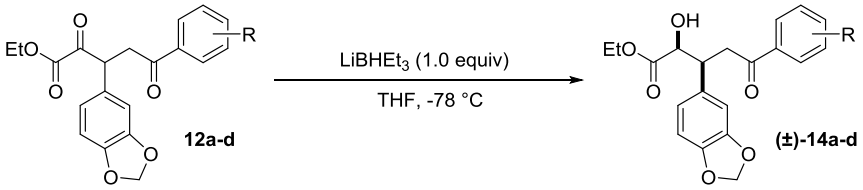
(m, 2H), 5.94 (d, *J* = 3.0 Hz, 2H), 5.05 (dd, *J* = 10.5, 3.9 Hz, 1H), 4.35 – 4.24 (m, 2H), 3.94 (dd, *J* = 18.1, 10.5 Hz, 1H), 3.39 (dd, *J* = 18.1, 4.0 Hz, 1H), 2.69 (q, *J* = 7.6 Hz, 2H), 1.32 (t, *J* = 7.2 Hz, 3H), 1.24 (t, *J* = 7.6 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 197.0, 191.9, 160.4, 150.5, 148.2, 147.3, 133.6, 128.7, 128.4, 128.1, 122.3, 109.1, 108.7, 101.2, 62.5, 47.8, 43.0, 28.9, 15.1, 13.9; **IR** (thin film, cm^{-1}): 2968, 2935, 2905, 1729, 1678, 1607, 1488, 1038, 766; **TLC** (30% EtOAc/hexanes) R_f : 0.28; **HRMS** (ESI): Calculated for [M+H]+ $C_{22}H_{23}O_6$: 383.1495, Found: 383.1492.



12d

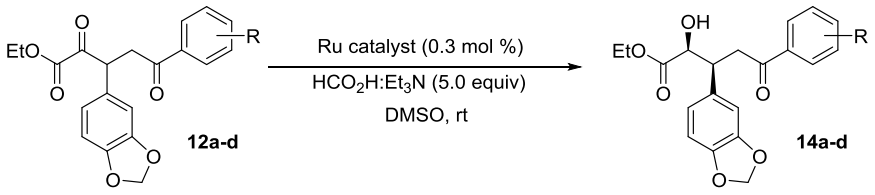
Ethyl 3-(benzo[d][1,3]dioxol-5-yl)-5-(2-fluorophenyl)-2,5-dioxopentanoate (12d): The title compound was prepared according to General Procedure A: enone **13d** (500 mg, 1.85 mmol), triazolium salt (134 mg, 0.37 mmol), ethyl glyoxylate (3 x 230 mg, 3 x 1.85 mmol), dichloromethane (3.7 mL), triethylamine (0.39 mL, 2.8 mmol). Flash chromatography (15-20% EtOAc/hexanes) provided **12d** (685 mg, 1.84 mmol, 99% yield) as a viscous yellow oil. Analytical data for **12d**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.86 (td, *J* = 7.6, 1.9 Hz, 1H), 7.57 – 7.48 (m, 1H), 7.23 – 7.19 (m, 1H), 7.12 (ddd, *J* = 11.3, 8.3, 1.1 Hz, 1H), 6.80 (d, *J* = 1.7 Hz, 1H), 6.78 – 6.74 (m, 2H), 5.94 – 5.92 (m, 2H), 5.05 (dd, *J* = 10.8, 3.7 Hz, 1H), 4.33 – 4.22 (m, 2H), 3.95 (ddd, *J* = 18.9, 10.8, 3.3 Hz, 1H), 3.39 (dt, *J* = 18.9, 3.6 Hz, 1H), 1.32 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 195.4 (d, *J* = 4.1 Hz), 191.7, 162.2 (d, *J* = 255.7 Hz), 160.4, 148.1, 147.4, 135.1 (d, *J* = 9.1 Hz), 130.6 (d, *J* = 2.3 Hz), 128.4, 124.5 (d, *J* = 3.3 Hz), 124.3 (d, *J* = 12.6 Hz), 122.4, 116.7 (d, *J* = 23.7 Hz), 109.0, 108.7, 101.2, 62.5, 47.8 (d, *J* = 2.3 Hz), 47.6 (d, *J* = 8.5 Hz), 13.9; **IR** (thin film, cm^{-1}): 2984, 2904, 1739, 1683, 1610, 1487, 1453, 1038, 768; **TLC** (20% EtOAc/hexanes) R_f : 0.21; **HRMS** (ESI): Calculated for [M+H]+ $C_{20}H_{18}FO_6$: 373.1087, Found: 373.1087.

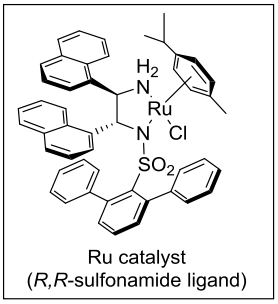
General Procedure B for Preparation of Racemic δ-Keto-α-Hydroxy Esters (±)-14a-d



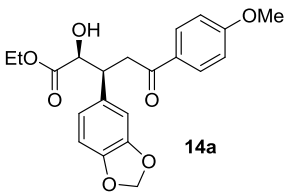
Lithium triethylborohydride (1.0 equiv, 1.0 M solution in THF) was added to a solution of β-substituted α,δ-diketo ester **12a-d** (1.0 equiv) in THF (0.5 M concentration) at -78 °C. The reaction was allowed to stir at this temperature for 10 min and quenched with saturated ammonium chloride. The reaction 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 α-keto δ-hydroxy esters **x** which were purified by flash chromatography using the indicated solvent systems.

General Procedure C for Preparation of Enantioenriched δ-Keto-α-Hydroxy Esters 14a-d

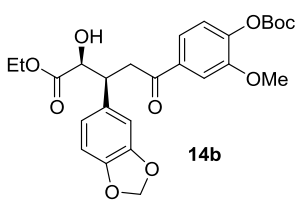




To a flame-dried 20 mL scintillation vial equipped with magnetic stir bar were added $[RuCl_2(p\text{-cymene})]_2$ (0.3-0.4 mol %) and **L1** (1.2-1.6 mol %). The vial was purged with nitrogen and DMSO (0.01 M) was added. The resulting solution was heated to 70 °C. After 1 h, the red solution was cooled to room temperature and transferred via cannula to a solution of α-keto ester **12a-d** (1.0 equiv) in DMSO (0.6 M) under nitrogen. Formic acid/triethylamine azeotrope (5:2, 5.0 equiv) was added. After the indicated time period the reaction was diluted with water and extracted with ethyl acetate (2x). The combined organic extracts were washed with brine, dried (Na_2SO_4) and concentrated *in vacuo*. The crude product was purified by flash chromatography using the indicated solvent systems.

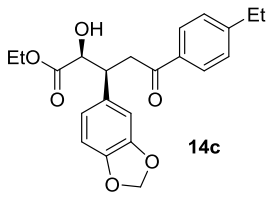


(2*S*,3*S*)-Ethyl 3-(benzo[d][1,3]dioxol-5-yl)-2-hydroxy-5-(4-methoxyphenyl)-5-oxopentanoate (14a): The title compound was prepared according to General Procedure C: $[RuCl_2(p\text{-cymene})]_2$ (32.5 mg, 0.053 mmol, 0.3 mol %), **L1** (128 mg, 0.212 mmol, 12 mol %), DMSO (5.3 mL); α-keto ester **12a** (6.70 g, contained 4% enone from preparation, *vide supra*), DMSO (30 mL), formic acid/triethylamine azeotrope (5:2, 7.64 mL, 88.4 mmol, 5.0 equiv); 24 h. The diastereomer ratio (>20:1 dr) was determined by ^{1}H NMR spectroscopic analysis of the crude reaction mixture in which no diastereomer was observed. Flash chromatography provided **14a** (6.31 g, 16.3 mmol, 92% yield over 2 steps, >20:1 dr) as a pale yellow, viscous oil. Analytical data for **14a**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.98 – 7.95 (m, 2H), 6.95 – 6.89 (m, 2H), 6.84 (d, *J* = 1.7 Hz, 1H), 6.75 – 6.67 (m, 2H), 5.90 (q, *J* = 1.6 Hz, 2H), 4.50 (dd, *J* = 6.1, 3.1 Hz, 1H), 4.14 (qq, *J* = 7.4, 3.6 Hz, 2H), 3.86 (s, 4H), 3.62 (dd, *J* = 17.5, 8.1 Hz, 1H), 3.34 (dd, *J* = 17.5, 6.1 Hz, 1H), 2.88 (d, *J* = 6.1 Hz, 1H), 1.26 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 196.7, 173.7, 163.5, 147.4, 146.6, 132.7, 130.3, 130.0, 121.8, 113.7, 108.9, 108.0, 100.9, 72.6, 61.8, 55.4, 43.4, 40.2, 14.2; **IR** (thin film, cm^{-1}): 3503, 2903, 1733, 1673, 1601, 1506, 1489, 1037; **TLC** (30% EtOAc/hexanes) R_f : 0.19; **HRMS** (ESI): Calculated for [M+H]+ $C_{21}H_{23}O_7$: 387.1444, Found: 387.1440; **SFC Analysis**: AD column, 20% MeOH, 1.5 mL/min, 150 bar, 210 nm; t_{major} = 10.1 min, t_{minor} = 13.5 min, 96:4 er; **$[\alpha]_D^{25}$** -17.9 (*c* = 0.26, MeOH).



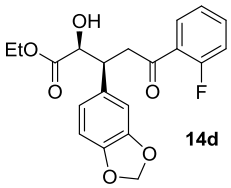
14b

(2*S*,3*S*)-Ethyl 3-(benzo[d][1,3]dioxol-5-yl)-5-(4-((tert-butoxycarbonyl)oxy)-3-methoxyphenyl)-2-hydroxy-5-oxopentanoate (14b): The title compound was prepared according to General Procedure C: $[RuCl_2(p\text{-cymene})]_2$ (14.8 mg, 0.024 mmol, 0.4 mol %), **L1** (58.6 mg, 0.097 mmol, 16 mol %), DMSO (2.4 mL); α-keto ester **12b** (3.03 g, 6.06 mmol, 1.0 equiv), DMSO (9.7 mL), formic acid/triethylamine azeotrope (5:2, 2.62 mL, 30.3 mmol, 5.0 equiv); 28 h. The diastereomer ratio (>20:1 dr) was determined by ^{1}H NMR spectroscopic analysis of the crude reaction mixture in which no diastereomer was observed. Flash chromatography provided **14b** (2.92 g, 5.81 mmol, 96%, >20:1 dr) as a sticky, yellow foam. Analytical data for **14b**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.63 – 7.56 (m, 2H), 7.19 (d, *J* = 8.2 Hz, 1H), 6.82 (d, *J* = 1.7 Hz, 1H), 6.74 – 6.67 (m, 2H), 5.90 (s, 2H), 4.49 (dd, *J* = 5.9, 3.1 Hz, 1H), 4.14 (qq, *J* = 7.4, 3.6 Hz, 2H), 3.88 (s, 3H), 3.84 (ddd, *J* = 7.6, 6.4, 3.1 Hz, 1H), 3.61 (dd, *J* = 17.6, 7.7 Hz, 1H), 3.39 (dd, *J* = 17.6, 6.4 Hz, 1H), 2.90 (d, *J* = 6.0 Hz, 1H), 1.54 (s, 9H), 1.25 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 197.0, 173.5, 151.4, 150.7, 147.5, 146.7, 144.2, 135.5, 132.4, 122.4, 121.8, 121.4, 111.5, 108.9, 108.0, 100.9, 83.9, 72.6, 61.8, 56.0, 43.4, 40.5, 27.5, 14.2; **IR** (thin film, cm^{-1}): 3503, 2981, 1762, 1684, 1506; **TLC** (30% EtOAc/hexanes) R_f : 0.19; **HRMS** (ESI): Calculated for [M+H]+ $C_{26}H_{31}O_{10}$: 503.1917, Found: 503.1921; **SFC Analysis**: AD column, 15% MeOH, 1.5 mL/min, 150 bar, 210 nm; t_{major} = 7.4 min, t_{minor} = 8.8 min, 97:3 er; **$[\alpha]_D^{25}$** +3.2 (*c* = 0.34, MeOH).



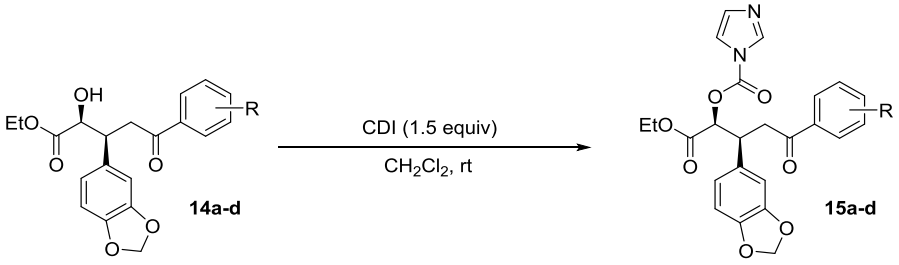
14c

(2*S*,3*S*)-Ethyl 3-(benzo[d][1,3]dioxol-5-yl)-5-(4-ethylphenyl)-2-hydroxy-5-oxopentanoate (14c): The title compound was prepared according to General Procedure C: $[RuCl_2(p\text{-cymene})]_2$ (9.0 mg, 0.015 mmol, 0.3 mol %), **L1** (35.5 mg, 0.059 mmol, 12 mol %), DMSO (1.5 mL); α-keto ester **12c** (1.87 g, 4.89 mmol, 1.0 equiv), DMSO (8.3 mL), formic acid/triethylamine azeotrope (5:2, 2.12 mL, 24.5 mmol, 5.0 equiv); 22 h. The diastereomer ratio (>20:1 dr) was determined by ^{1}H NMR spectroscopic analysis of the crude reaction mixture in which no diastereomer was observed. Flash chromatography provided **14c** (1.84 g, 4.79 mmol, 98%, >20:1 dr) as a viscous pale yellow oil. Analytical data for **14c**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.91 (d, *J* = 8.3 Hz, 2H), 7.27 (d, *J* = 8.2 Hz, 2H), 6.84 (d, *J* = 1.7 Hz, 1H), 6.77 – 6.65 (m, 2H), 5.90 (q, *J* = 1.5 Hz, 2H), 4.51 (dd, *J* = 6.1, 3.1 Hz, 1H), 4.15 (qq, *J* = 7.2, 3.6 Hz, 2H), 3.87 (ddd, *J* = 8.0, 6.1, 3.1 Hz, 1H), 3.65 (dd, *J* = 17.7, 8.0 Hz, 1H), 3.38 (dd, *J* = 17.7, 6.1 Hz, 1H), 2.90 (d, *J* = 6.1 Hz, 1H), 2.69 (q, *J* = 7.6 Hz, 2H), 1.25 (td, *J* = 7.4, 6.3 Hz, 6H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 197.8, 173.7, 150.2, 147.4, 146.6, 134.6, 132.6, 128.3, 128.0, 121.8, 108.9, 108.0, 100.9, 72.6, 61.8, 43.3, 40.5, 28.9, 15.1, 14.2; **IR** (thin film, cm^{-1}): 3502, 2968, 1733, 1683, 1505, 1489, 1038, 813; **TLC** (30% EtOAc/hexanes) R_f : 0.28; **HRMS** (ESI): Calculated for [M+H]+ $C_{22}H_{25}O_6$: 385.1651, Found: 385.1648; **SFC Analysis**: AD column, 20% MeOH, 1.5 mL/min, 150 bar, 210 nm; t_{major} = 8.6 min, t_{minor} = 10.6 min, 97:3 er; **$[\alpha]_D^{25}$** -11.8 (*c* = 0.12, MeOH).

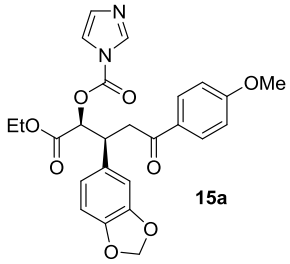


(2*S*,3*S*)-Ethyl 3-(benzo[d][1,3]dioxol-5-yl)-5-(2-fluorophenyl)-2-hydroxy-5-oxopentanoate (14d): The title compound was prepared according to General Procedure C: $[RuCl_2(p\text{-cymene})]_2$ (14.4 mg, 0.023 mmol, 0.3 mol %), **L1** (56.8 mg, 0.094 mmol, 12 mol %), DMSO (2.3 mL); α-keto ester **12d** (2.91 g, 7.82 mmol, 1.0 equiv), DMSO (13.3 mL), formic acid/triethylamine azeotrope (5:2, 3.38 mL, 39.1 mmol, 5.0 equiv); 23 h. The diastereomer ratio (>20:1 dr) was determined by ^{1}H NMR spectroscopic analysis of the crude reaction mixture in which no diastereomer was observed. Flash chromatography provided **14d** (2.77 g, 7.40 mmol, 95%, >20:1 dr) as a viscous pale yellow oil. Analytical data for **14d**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.82 (td, *J* = 7.6, 1.9 Hz, 1H), 7.51 (dddd, *J* = 8.3, 7.1, 5.0, 1.9 Hz, 1H), 7.20 (ddd, *J* = 8.0, 7.3, 1.1 Hz, 1H), 7.13 (ddd, *J* = 11.3, 8.3, 1.1 Hz, 1H), 6.81 (d, *J* = 1.5 Hz, 1H), 6.74 – 6.65 (m, 2H), 5.91 – 5.89 (m, 2H), 4.52 (dd, *J* = 6.2, 3.2 Hz, 1H), 4.15 (qq, *J* = 7.1, 3.6 Hz, 2H), 3.85 (dddd, *J* = 7.5, 6.2, 3.3, 1.0 Hz, 1H), 3.67 (ddd, *J* = 18.5, 7.9, 2.9 Hz, 1H), 3.43 (ddd, *J* = 18.5, 6.2, 2.9 Hz, 1H), 2.84 (d, *J* = 6.3 Hz, 1H), 1.27 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 196.5 (d, *J* = 4.3 Hz), 173.7, 161.9 (d, *J* = 255.2 Hz), 147.4, 146.6, 134.6 (d, *J* = 9.0 Hz), 132.4, 130.5 (d, *J* = 2.6 Hz), 125.6 (d, *J* = 12.9 Hz), 124.4 (d, *J* = 3.4 Hz), 121.9, 116.7 (d, *J* = 23.7 Hz), 109.0, 108.0, 100.9, 72.6, 61.8, 45.5 (d, *J* = 7.7 Hz), 43.1, 14.2; **IR** (thin film, cm^{-1}): 3502, 2983, 2902, 1733, 1685, 1609, 1488, 1452, 1098, 1038, 766; **TLC** (20% EtOAc/hexanes) R_f : 0.21; **HRMS** (ESI): Calculated for [M+H]+ $C_{20}H_{19}FNaO_6$: 397.1063, Found: 397.1060; **SFC Analysis**: AD column, 20% MeOH, 1.5 mL/min, 150 bar, 210 nm; t_{minor} = 6.8 min, t_{major} = 8.5 min, 98:2 er; **$[\alpha]_D^{25}$** -17.0 (*c* = 0.22, MeOH).

General Procedure D for Preparation of δ-Keto-α-Carboxy Imidazoles 15a-d

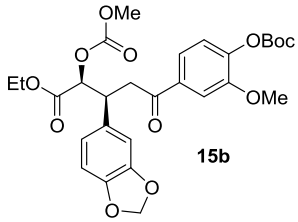


To a flame-dried round-bottomed flask equipped with magnetic stir bar under nitrogen was added 1,1′-carbonyldiimidazole (1.5 equiv) and dichloromethane. To the resulting suspension was added a solution of δ-keto-α-hydroxy ester **14a-d** (1.0 equiv) in dichloromethane. The reaction was stirred for the indicated time period then diluted with dichloromethane and water. The aqueous layer was extracted with dichloromethane. The combined organic extracts were dried (Na_2SO_4) and concentrated *in vacuo*. The crude product was purified by flash chromatography using the indicated solvent systems.



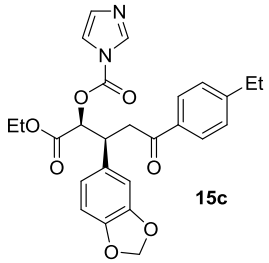
15a

(2*S*,3*S*)-3-(Benzo[d][1,3]dioxol-5-yl)-1-ethoxy-5-(4-methoxyphenyl)-1,5-dioxopentan-2-yl 1H-imidazole-1-carboxylate (15a): The title compound was prepared according to General Procedure D: 1,1′-carbonyldiimidazole (3.90 g, 24.1 mmol), dichloromethane (32 mL), δ-keto-α-hydroxy ester **14a** (6.20 g, 16.1 mmol); 19 h. Flash chromatography provided **15a** (7.25 g, 15.1 mmol, 94% yield) as a white foam. Analytical data for **15a**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.11-8.10 (m, Hz, 1H), 7.93 – 7.88 (m, 2H), 7.39-7.38 (m, 1H), 7.09 – 7.04 (m, 1H), 6.93 – 6.87 (m, 2H), 6.81 (d, *J* = 1.7 Hz, 1H), 6.80 – 6.70 (m, 2H), 5.92 (s, 2H), 5.55 (d, *J* = 4.9 Hz, 1H), 4.23 – 4.02 (m, 3H), 3.84 (s, 3H), 3.50 (dd, *J* = 17.6, 8.0 Hz, 1H), 3.39 (dd, *J* = 17.5, 6.0 Hz, 1H), 1.17 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 195.1, 167.3, 163.8, 147.9, 147.8, 147.0, 137.0, 131.8, 130.8, 130.2, 129.3, 121.6, 117.1, 113.8, 108.4, 108.3, 101.1, 77.9, 62.0, 55.4, 41.2, 39.8, 13.9; **IR** (thin film, cm^{-1}): 2981, 1752, 1674, 1601, 1397, 1172, 1038, 733; **TLC** (50% EtOAc/hexanes) R_f : 0.11; **HRMS** (ESI): Calculated for [M-$C_3H_3N_2$+MeOH]+ $C_{23}H_{25}O_9$: 445.1499, Found: 445.1495; **$[\alpha]_D^{25}$** -6.3 (*c* = 0.26, MeOH).



15b

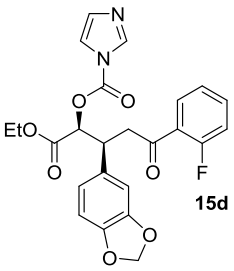
(2*S*,3*S*)-3-(Benzo[d][1,3]dioxol-5-yl)-5-(4-((tert-butoxycarbonyl)oxy)-3-methoxyphenyl)-1-ethoxy-1,5-dioxopentan-2-yl 1H-imidazole-1-carboxylate (15b): The title compound was prepared according to General Procedure D: 1,1′-carbonyldiimidazole (248 mg, 1.53 mmol), dichloromethane (2.0 mL), δ-keto-α-hydroxy ester **14b** (512 mg, 1.02 mmol); 2 h. Flash chromatography provided **15b** (550 mg, 0.93 mmol, 91% yield) as a white foam. Analytical data for **15b**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.12 (t, *J* = 1.0 Hz, 1H), 7.55 (d, *J* = 1.9 Hz, 1H), 7.53 (d, *J* = 8.2 Hz, 1H), 7.39 (t, *J* = 1.5 Hz, 1H), 7.19 (d, *J* = 8.2 Hz, 1H), 7.08 (dd, *J* = 1.7, 0.8 Hz, 1H), 6.82 – 6.79 (m, 1H), 6.79 – 6.72 (m, 2H), 5.94 (s, 2H), 5.55 (d, *J* = 5.0 Hz, 1H), 4.25 – 4.09 (m, 3H), 3.88 (s, 3H), 3.51 (dd, *J* = 17.8, 7.6 Hz, 1H), 3.45 (dd, *J* = 17.7, 6.4 Hz, 1H), 1.54 (s, 9H), 1.20 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 195.5, 167.2, 151.6, 150.6, 148.0, 147.9, 147.2, 144.5, 137.1, 134.9, 131.6, 131.0, 122.5, 121.7, 121.3, 117.1, 111.5, 108.4, 108.4, 101.2, 84.1, 77.8, 62.1, 56.1, 41.2, 40.2, 27.5, 14.0; **IR** (thin film, cm^{-1}): 2981, 1760, 164, 1506, 1396, 1145, 737; **TLC** (50% EtOAc/hexanes) R_f : 0.19; **HRMS** (ESI): Calculated for [M-$C_3H_3N_2$+MeOH]+ $C_{28}H_{30}O_{12}$: 561.1972, Found: 561.1970; **$[\alpha]_D^{25}$** +10.8 (*c* = 0.32, MeOH).



15c

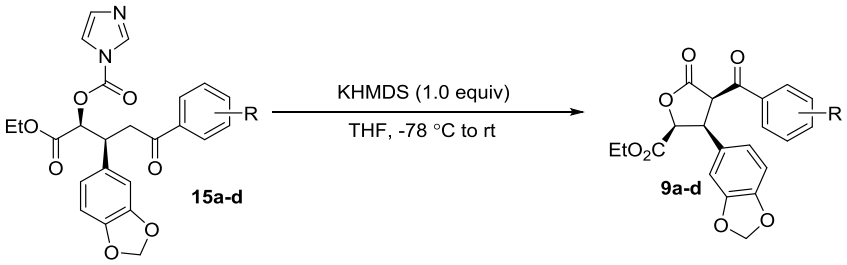
(2*S*,3*S*)-3-(Benzo[d][1,3]dioxol-5-yl)-1-ethoxy-5-(4-ethylphenyl)-1,5-dioxopentan-2-yl 1H-imidazole-1-carboxylate (15c): The title compound was prepared according to General Procedure D: 1,1′-carbonyldiimidazole (1.12 g, 6.91 mmol), dichloromethane (9.2 mL), δ-keto-α-hydroxy ester **14c** (1.77 g, 4.60 mmol); 3 h. Flash chromatography provided **15c** (2.16 g, 4.51 mmol, 98% yield) as a white foam. Analytical data for **15c**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.11 (t, *J* = 1.0 Hz, 1H), 7.89 – 7.83 (m, 2H), 7.39 (t, *J* = 1.5 Hz, 1H), 7.30 – 7.23 (m, 2H), 7.08 (dd, *J* = 1.7, 0.8 Hz, 1H), 6.81 (d, *J* = 1.7 Hz, 1H), 6.80 – 6.73 (m, 2H), 5.94 (s, 2H), 5.56 (d, *J* = 4.9 Hz, 1H), 4.27 – 4.05 (m, 3H), 3.54 (dd, *J* = 17.7, 7.9 Hz, 1H), 3.43 (dd, *J* = 17.7, 6.1 Hz, 1H), 2.69 (q, *J* = 7.6 Hz, 2H), 1.24 (t, *J* = 7.6 Hz, 3H), 1.19 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 196.3, 167.3,

150.7, 148.0, 147.9, 147.1, 137.1, 134.1, 131.8, 130.9, 128.2, 121.7, 117.1, 108.5, 108.4, 101.2, 77.9, 62.1, 41.2, 40.1, 28.9, 15.1, 14.0; **IR** (thin film, cm^{-1}): 2969, 2935, 1735, 1682, 1606, 1490, 1396, 1239, 1039, 735; **TLC** (30% EtOAc/hexanes) R_f : 0.08; **HRMS** (ESI): Calculated for [M-$C_3H_3N_2$+MeOH]+ $C_{24}H_{27}O_8$: 443.1706, Found: 443.1702; **$[\alpha]_D^{25}$** +1.8 (*c* = 0.29, MeOH).

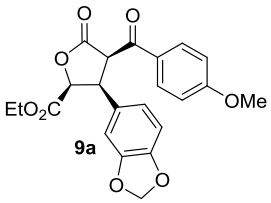


(2*S*,3*S*)-3-(Benzo[d][1,3]dioxol-5-yl)-1-ethoxy-5-(2-fluorophenyl)-1,5-dioxopentan-2-yl 1H-imidazole-1-carboxylate (15d): The title compound was prepared according to General Procedure D: 1,1′-carbonyldiimidazole (1.73 g, 10.7 mmol), dichloromethane (14.2 mL), δ-keto-α-hydroxy ester **14d** (2.66 g, 7.11 mmol); 24 h. Flash chromatography provided **15d** (3.19 g, 6.81 mmol, 96% yield) as a viscous pale yellow oil. Analytical data for **15d**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.10 (t, *J* = 1.1 Hz, 1H), 7.79 (td, *J* = 7.6, 1.9 Hz, 1H), 7.54 – 7.46 (m, 1H), 7.38 (t, *J* = 1.5 Hz, 1H), 7.22 – 7.17 (m, 1H), 7.10 (ddd, *J* = 11.4, 8.3, 1.1 Hz, 1H), 7.06 (dd, *J* = 1.7, 0.9 Hz, 1H), 6.79 (d, *J* = 1.7 Hz, 1H), 6.77 – 6.71 (m, 2H), 5.92 (s, 2H), 5.53 (d, *J* = 5.0 Hz, 1H), 4.24 – 4.05 (m, 3H), 3.57 (ddd, *J* = 18.4, 7.7, 2.8 Hz, 1H), 3.46 (ddd, *J* = 18.4, 6.4, 2.7 Hz, 1H), 1.19 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 195.0 (d, *J* = 3.9 Hz), 167.2 , 161.8 (d, *J* = 254.2 Hz), 148.0 , 147.8 , 147.0 , 137.0 , 135.0 (d, *J* = 9.1 Hz), 131.5 , 130.8 , 130.6 (d, *J* = 2.5 Hz), 124.9 (d, *J* = 12.6 Hz), 124.6 (d, *J* = 3.3 Hz), 121.7 , 117.0 , 116.7 (d, *J* = 24.0 Hz), 108.4 , 108.2 , 101.1 , 77.8 , 62.0 , 45.2 (d, *J* = 8.2 Hz), 41.0 (d, *J* = 2.0 Hz), 13.9; **IR** (thin film, cm^{-1}): 2905, 1749, 1685, 1609, 1489, 1452, 1039, 765; **TLC** (30% EtOAc/hexanes) R_f : 0.08; **HRMS** (ESI): Calculated for [M-$C_3H_3N_2$+MeOH]+ $C_{22}H_{22}FO_8$: 433.1299, Found:433.1295; **$[\alpha]_D^{25}$** -10.0 (*c* = 0.21, MeOH).

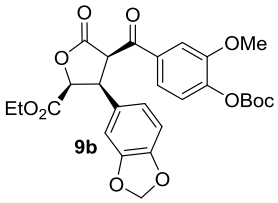
General Procedure E for Preparation of β-Keto-γ-Butyrolactones 9a-d



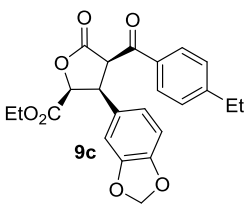
To a flame-dried, oversized round-bottomed flask (500 mL-1000 mL) equipped with magnetic stir bar under nitrogen was added a solution of δ-keto-β-carboxy imidazole **15a-d** (1 equiv) in anhydrous tetrahydrofuran (0.1 M). The flask was cooled to -78 °C and a solution of potassium bis(trimethylsilyl)amide (0.5 M in toluene) was added slowly as a steady stream. After 15 min, the reaction was warmed to 0 °C in an ice bath. After 1 h, the reaction was warmed to room temperature. After an additional 30 min, the reaction was quenched with aqueous saturated ammonium chloride, diluted with water, and extracted with diethyl ether (2x). The combined organic extracts were washed with brine, dried ($MgSO_4$), and concentrated *in vacuo*. The crude product was purified by flash chromatography using the indicated solvent systems.



Ethyl (2*S*,3*S*,4*R*)-3-(benzo[d][1,3]dioxol-5-yl)-4-(4-methoxybenzoyl)-5-oxotetrahydrofuran-2-carboxylate (9a): The title compound was prepared according to General Procedure E: δ-keto-β-carboxy imidazole **15a** (3.17 g, 6.60 mmol), tetrahydrofuran (72 mL), potassium bis(trimethylsilyl)amide (13.2 mL, 6.60 mmol). The diastereomer ratio (>20:1 dr) was determined by ^{1}H NMR spectroscopic analysis of the crude reaction mixture in which no diastereomer was observed. Flash chromatography provided **9a** (2.02 g, 4.90 mmol, 74% yield, >20:1 dr) as colorless viscous oil. Analytical data for **9a**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.09 – 8.01 (m, 2H), 7.01 – 6.96 (m, 2H), 6.77 – 6.64 (m, 3H), 5.26 (d, *J* = 8.2 Hz, 1H), 4.94 (d, *J* = 8.7 Hz, 1H), 4.66 (t, *J* = 8.4 Hz, 1H), 3.97 (ddq, *J* = 42.5, 10.8, 7.1 Hz, 2H), 3.88 (s, 3H), 1.00 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 189.5, 171.1, 168.0, 164.5, 148.0, 147.6, 132.1, 128.3, 128.1, 121.1, 114.1, 108.5, 108.0, 101.3, 79.3, 61.6, 55.6, 52.3, 46.8, 13.7; **IR** (thin film, cm^{-1}): 2982, 1785, 1742, 1671, 1601, 1507, 1037; **TLC** (30% EtOAc/hexanes) R_f : 0.24; **HRMS** (ESI): Calculated for [M+H]+ $C_{22}H_{21}O_8$: 413.1236, Found: 413.1237; **$[\alpha]_D^{25}$** +97.8 (*c* = 0.26, MeOH).

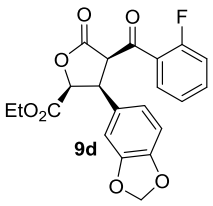


Ethyl (2*S*,3*S*,4*R*)-3-(benzo[d][1,3]dioxol-5-yl)-4-(4-((tert-butoxycarbonyl)oxy)-3-methoxybenzoyl)-5-oxotetrahydrofuran-2-carboxylate (9b): The title compound was prepared according to General Procedure E: δ-keto-β-carboxy imidazole **15b** (2.28 g, 3.83 mmol), tetrahydrofuran (38 mL), potassium bis(trimethylsilyl)amide (7.67 mL, 3.83 mmol); The diastereomer ratio (>20:1 dr) was determined by ^{1}H NMR spectroscopic analysis of the crude reaction mixture in which no diastereomer was observed. Flash chromatography provided **9b** (2.02 g, 4.90 mmol, 74% yield, >20:1 dr) as a colorless solid. Analytical data for **9b**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.71 (dd, *J* = 8.3, 2.0 Hz, 1H), 7.65 (d, *J* = 2.0 Hz, 1H), 7.30 – 7.24 (m, 1H), 6.72 (d, *J* = 8.0 Hz, 1H), 6.69 (d, *J* = 1.8 Hz, 1H), 6.65 (dd, *J* = 8.1, 1.9 Hz, 1H), 5.93 (q, *J* = 1.4 Hz, 2H), 5.24 (d, *J* = 8.3 Hz, 1H), 4.97 (d, *J* = 9.2 Hz, 1H), 4.65 (t, *J* = 8.7 Hz, 1H), 4.03 – 3.91 (m, 2H), 3.90 (s, 3H), 1.54 (s, 9H), 0.99 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 190.4, 170.7, 168.0, 151.6, 150.5, 148.1, 147.7, 145.1, 133.9, 127.5, 123.4, 122.7, 121.0, 112.6, 108.5, 107.9, 101.3, 84.1, 79.2, 61.7, 56.1, 52.4, 47.0, 27.5, 13.7; **IR** (thin film, cm^{-1}): 2982, 1762, 1684, 1507, 889; **m.p.** 68-70 °C; **TLC** (30% EtOAc/hexanes) R_f : 0.27; **HRMS** (ESI): Calculated for [M+Na]+ $C_{27}H_{28}NaO_{11}$: 551.1529, Found: 551.1530; **$[\alpha]_D^{25}$** +156.0 (*c* = 0.14, MeOH).



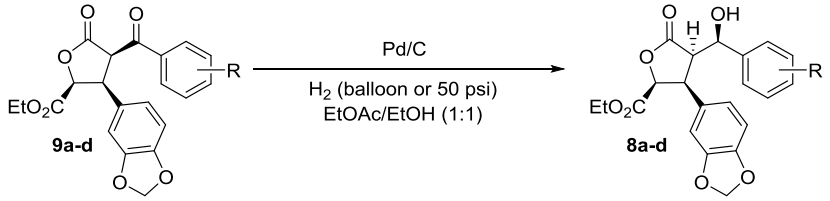
Ethyl (2*S*,3*S*,4*R*)-3-(benzo[d][1,3]dioxol-5-yl)-4-(4-ethylbenzoyl)-5-oxotetrahydrofuran-2-carboxylate (9c): The title compound was prepared according to General Procedure E: δ-keto-β-carboxy imidazole **15c** (2.08 g, 4.34 mmol), tetrahydrofuran (47 mL), potassium bis(trimethylsilyl)amide (8.70 mL, 4.34 mmol). The diastereomer ratio (>20:1 dr) was determined by ^{1}H NMR spectroscopic analysis of the crude reaction mixture by comparison of the integration of resonances at δ 5.26 (major diastereomer) and δ 5.10 (minor diastereomer). Flash chromatography provided **9c** (1.23 g, 3.00 mmol, 69%

yield, >20:1 dr) as a colorless, viscous oil. Analytical data for **9c**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.00 (d, *J* = 8.4 Hz, 1H), 7.36 – 7.30 (m, 2H), 6.74 – 6.65 (m, 3H), 5.92 (t, *J* = 1.1 Hz, 2H), 5.25 (d, *J* = 8.3 Hz, 1H), 5.00 (d, *J* = 8.9 Hz, 1H), 4.66 (t, *J* = 8.5 Hz, 1H), 3.97 (ddq, *J* = 41.5, 10.7, 7.1 Hz, 2H), 2.72 (q, *J* = 7.6 Hz, 2H), 1.25 (t, *J* = 7.6 Hz, 3H), 0.99 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 190.9, 170.9, 168.0, 151.6, 148.0, 147.6, 133.0, 129.8, 128.4, 127.9, 121.0, 108.5, 108.0, 101.3, 79.2, 61.6, 52.3, 46.8, 29.0, 15.0, 13.7; **IR** (thin film, cm^{-1}): 2969, 1787, 1741, 1680, 1606, 1505, 1038, 737; **TLC** (30% EtOAc/hexanes) R_f : 0.35; **HRMS** (ESI): Calculated for [M+H]+ $C_{23}H_{23}O_7$: 411.1444, Found: 411.1441; **$[\alpha]_D^{25}$** +131.1 (*c* = 0.58, MeOH).

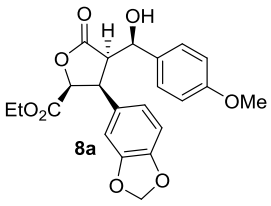


Ethyl (2*S*,3*S*,4*R*)-3-(benzo[d][1,3]dioxol-5-yl)-4-(2-fluorobenzoyl)-5-oxotetrahydrofuran-2-carboxylate (9d): The title compound was prepared according to General Procedure E: δ-keto-β-carboxy imidazole **15d** (3.09 g, 6.60 mmol), tetrahydrofuran (66 mL), potassium bis(trimethylsilyl)amide (13.2 mL, 6.60 mmol);The keto/enol ratio (7:1) was determined by ^{1}H NMR spectroscopic analysis by comparison of the integration of resonances at δ 5.94 (keto) and δ 5.79 (enol). The diastereomer ratio (>20:1 dr) was determined by ^{1}H NMR spectroscopic analysis of the crude reaction mixture in which no diastereomer was observed. Flash chromatography provided **9d** (1.62 g, 4.05 mmol, 61% yield, 4:1 mixture of keto (>20:1 dr) to enol tautomers) as a colorless, viscous oil. Analytical data for **9d**: **^{1}H NMR** (Keto tautomer, 600 MHz, $CDCl_3$): δ 7.89 (td, *J* = 7.6, 1.8 Hz, 1H), 7.63 – 7.57 (m, 1H), 7.30 – 7.25 (m, 1H), 7.19 (ddd, *J* = 11.6, 8.3, 1.1 Hz, 1H), 6.77 – 6.68 (m, 3H), 5.94 (s, 2H), 5.17 (d, *J* = 8.4 Hz, 1H), 5.14 (dd, *J* = 10.1, 2.2 Hz, 1H), 4.64 (ddd, *J* = 10.4, 8.4, 2.3 Hz, 1H), 3.98 (ddq, *J* = 47.0, 10.8, 7.2 Hz, 2H), 1.02 (t, *J* = 7.2 Hz, 3H); **^{1}H NMR** (Enol tautomer, 600 MHz, $CDCl_3$): δ 11.66 (s, 1H), 7.02 (td, *J* = 7.6, 1.1 Hz, 1H), 6.86 (ddd, *J* = 10.6, 8.3, 1.1 Hz, 1H), 6.82 – 6.78 (m, 1H), 6.45 (d, *J* = 7.9 Hz, 1H), 6.42 – 6.36 (m, 2H), 5.79 (d, *J* = 10.7 Hz, 1H), 5.25 (d, *J* = 9.5 Hz, 1H), 4.72 (d, *J* = 9.5 Hz, 1H), 3.78 (ddq, *J* = 69.5, 10.9, 7.2 Hz, 2H), 0.91 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (Keto tautomer, 150 MHz, $CDCl_3$): δ 190.1 (d, *J* = 3.5 Hz), 170.5, 167.9, 162.2 (d, *J* = 256.1 Hz), 148.1, 147.7, 136.0 (d, *J* = 9.5 Hz), 131.4 (d, *J* = 1.7 Hz), 127.3, 124.8 (d, *J* = 3.3 Hz), 121.1, 117.1, 116.9, 108.5, 108.0, 101.3, 78.9, 61.7, 55.4 (d, *J* = 8.3 Hz), 46.5, 13.7; **^{13}C NMR** (Enol tautomer, 150 MHz, $CDCl_3$): δ 175.5, 167.2, 164.4, 158.8 (d, *J* = 251.8 Hz), 147.7, 132.5 (d, *J* = 8.7 Hz), 129.7, 129.3 (d, *J* = 2.6 Hz), 124.2 (d, *J* = 10.7 Hz), 124.0 (d, *J* = 3.4 Hz), 122.2, 115.7, 115.6, 108.7, 107.6, 101.4, 100.9, 79.9, 61.4, 46.0 (d, *J* = 7.8 Hz), 13.6; **IR** (thin film, cm^{-1}): 1791, 1747, 1685, 1609, 1506, 1490, 1454, 1038, 766; **TLC** (30% EtOAc/hexanes) R_f : 0.27; **HRMS** (ESI): Calculated for [M+H]+ $C_{21}H_{18}FO_7$: 401.1037, Found: 401.1033; **$[\alpha]_D^{25}$** +66.4 (*c* = 0.14, MeOH).

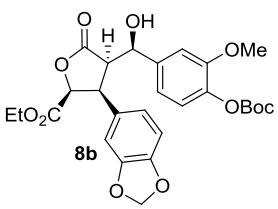
General Procedure F for Reduction of β-Keto-γ-Butyrolactones 8a-d



To a round-bottomed flask equipped with magnetic stir bar containing a suspension of 10% palladium on carbon (100 mg/mmol) in absolute ethanol under nitrogen was added a solution of β-keto-γ-butyrolactones **9a-9d** in ethyl acetate. The flask was either purged with hydrogen (balloon) and vigorously stirred at room temperature under a hydrogen atmosphere for 1 h or placed in Parr bomb, cycled with nitrogen (3 x 25 psi), hydrogen (3 x 50 psi), then held at 50 psi hydrogen and stirred vigorously for 1h. The crude reaction mixture was filtered over a plug of silica gel eluting with ethyl acetate and concentrated *in vacuo*. The alcohol was used without further purification unless otherwise noted.

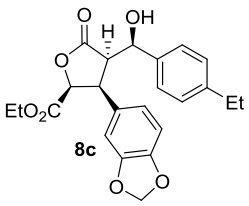


Ethyl (2*S*,3*S*,4*R*)-3-(benzo[d][1,3]dioxol-5-yl)-4-((*R*)-hydroxy(4-methoxyphenyl)methyl)-5-oxotetrahydrofuran-2-carboxylate (8a): The title compound was prepared according to General Procedure F: Pd/C (153 mg), ethanol (6.0 mL), β-keto-γ-butyrolactone **9a** (629 mg, 1.53 mmol), ethyl acetate (6.0 mL), hydrogen (balloon). Filtration and concentration provided **8a** (620 mg, 1.50 mmol, 98%, 8:1 dr). The diastereomer ratio was determined by ^{1}H NMR spectroscopic analysis of the crude reaction mixture by comparison of the integration of resonances at δ 4.95 (major diastereomer) and δ 4.97 (minor diastereomer). Analytical data for **8a**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.15 (d, *J* = 8.7 Hz, 2H), 6.72 – 6.68 (m, 2H), 6.60 (d, *J* = 8.0 Hz, 1H), 6.44 (dd, *J* = 8.0, 1.9 Hz, 1H), 6.39 (d, *J* = 1.8 Hz, 1H), 5.88 (d, *J* = 0.9 Hz, 2H), 4.94 (d, *J* = 6.4 Hz, 1H), 4.83 (d, *J* = 8.8 Hz, 1H), 3.97 – 3.78 (m, 2H), 3.73 (s, 3H), 3.69 (dd, *J* = 11.1, 8.8 Hz, 1H), 3.44 (dd, *J* = 11.1, 6.5 Hz, 1H), 0.94 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 176.7, 168.1, 159.5, 147.5, 147.0, 131.4, 127.8, 127.6, 121.4, 113.7, 113.6, 108.1, 108.0, 101.1, 78.9, 73.8, 61.6, 55.2, 49.6, 47.0, 13.7; **IR** (thin film, cm^{-1}): 3503, 2903, 1781, 1613, 1513, 1446, 1038, 736; **TLC** (30% EtOAc/hexanes) R_f : 0.16; **HRMS** (ESI): Calculated for [M+Na]+ $C_{22}H_{22}NaO_8$: 437.1212, Found: 437.1211; **$[\alpha]_D^{25}$** +158.2 (*c* = 0.18, MeOH).

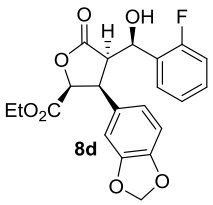


Ethyl (2*S*,3*S*,4*R*)-3-(benzo[d][1,3]dioxol-5-yl)-4-((*R*)-(4-((tert-butoxycarbonyl)oxy)-3-methoxyphenyl)(hydroxy)methyl)-5-oxotetrahydrofuran-2-carboxylate (8b): The title compound was prepared according to General Procedure F: Pd/C (9.5 mg), ethanol (0.5 mL), β-keto-γ-butyrolactone **9b** (50 mg, 0.095 mmol), ethyl acetate (0.5 mL), hydrogen (50 psi). Filtration and concentration provided **8b** (50 mg, 0.094 mmol, 99%, >20:1 dr). The diastereomer ratio was determined by ^{1}H NMR spectroscopic analysis of the crude reaction mixture. Analytical data for **8b**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 6.91 (d, *J* = 8.1 Hz, 1H), 6.88 (d, *J* = 2.0 Hz, 1H), 6.76 (dd, *J* = 8.2, 1.9 Hz, 1H), 6.60 (d, *J* = 7.9 Hz,

1H), 6.46 – 6.41 (m, 2H), 5.89 (s, 2H), 4.94 (d, *J* = 6.3 Hz, 1H), 4.83 (d, *J* = 8.9 Hz, 1H), 3.95 – 3.77 (m, 2H), 3.74 (s, 3H), 3.72 (d, *J* = 2.6 Hz, 1H), 3.71 – 3.65 (m, 1H), 3.46 (dd, *J* = 11.5, 6.2 Hz, 1H), 1.51 (s, 9H), 0.92 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 176.5, 168.1, 151.14, 151.06, 147.7, 147.2, 139.9, 138.2, 127.1, 122.0, 121.2, 118.8, 110.8, 108.1, 107.7, 101.2, 83.3, 78.8, 73.7, 61.6, 55.8, 49.0, 46.9, 27.5, 13.6; **IR** (thin film, cm^{-1}): 3503, 2982, 1761, 1507, 1146, 1037, 738; **TLC** (30% EtOAc/hexanes) R_f : 0.14; **HRMS** (ESI): Calculated for [M+Na]+ $C_{27}H_{30}NaO_{11}$: 553.1686, Found: 553.1685; **$[\alpha]_D^{25}$** +110.7 (*c* = 0.18, MeOH).

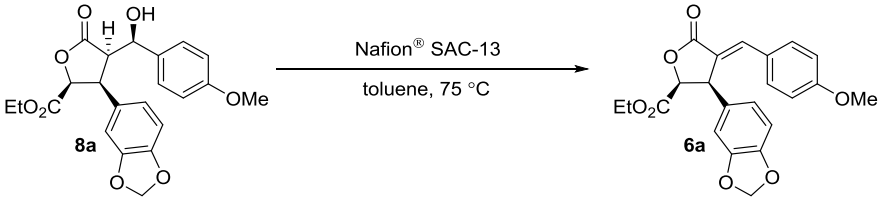


Ethyl (2*S*,3*S*,4*R*)-3-(benzo[d][1,3]dioxol-5-yl)-4-((*R*)-(4-ethylphenyl)(hydroxy)methyl)-5-oxotetrahydrofuran-2-carboxylate (8c): The title compound was prepared according to General Procedure F: Pd/C (380 mg), ethanol (15 mL), β-keto-γ-butyrolactone **9c** (1.56 g, 3.80 mmol), ethyl acetate (15 mL), hydrogen (50 psi). Flash chromatography provided **8c** (1.15 g, 2.79 mmol, 73%, 3:1 dr). Analytical data for **8c**: **^{1}H NMR** (Major Diastereomer, 600 MHz, $CDCl_3$): δ 7.13 (d, *J* = 7.9 Hz, 2H), 6.99 (d, *J* = 7.9 Hz, 2H), 6.57 (d, *J* = 8.0 Hz, 1H), 6.43 – 6.38 (m, 2H), 5.86 (s, 2H), 4.94 (d, *J* = 6.5 Hz, 1H), 4.82 (d, *J* = 8.9 Hz, 1H), 3.95 – 3.77 (m, 2H), 3.71 (dd, *J* = 11.1, 8.8 Hz, 1H), 3.65 – 3.63 (bs, 1H), 3.44 (dd, *J* = 11.1, 6.5 Hz, 1H), 2.52 (q, *J* = 7.5 Hz, 3H), 1.12 (t, *J* = 7.6 Hz, 3H), 0.92 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (Major Diastereomer, 150 MHz, $CDCl_3$): δ 176.7, 168.1, 147.4, 146.9, 144.3, 136.4, 127.7, 126.5, 125.4, 121.3, 108.02, 107.99, 101.0, 78.8, 73.9, 61.5, 49.4, 46.9, 28.4, 15.6, 13.6; **IR** (thin film, cm^{-1}): 3504, 2965, 1781, 1506, 1491, 1446, 1039, 737; **TLC** (30% EtOAc/hexanes) R_f : 0.27; **HRMS** (ESI): Calculated for [M+Na]+ $C_{23}H_{24}NaO_7$: 435.1420, Found: 435.1416; **$[\alpha]_D^{25}$** +150.9 (*c* = 0.34, MeOH).



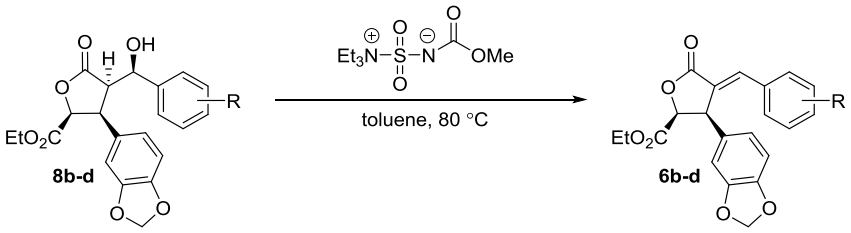
Ethyl (2*S*,3*S*,4*R*)-3-(benzo[d][1,3]dioxol-5-yl)-4-((*R*)-(2-fluorophenyl)(hydroxy)methyl)-5-oxotetrahydrofuran-2-carboxylate (8d): The title compound was prepared according to General Procedure F: Pd/C (138 mg), ethanol (15 mL), β-keto-γ-butyrolactone **9d** (1.38 g, 3.45 mmol), ethyl acetate (15 mL), hydrogen (balloon). Filtration and concentration provided **8d** (1.38 g, 3.42 mmol, 99%, 7:1 dr). Analytical data for **8d**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.47 (td, *J* = 7.5, 1.9 Hz, 1H), 7.09 – 7.03 (m, 1H), 7.01 (td, *J* = 7.5, 1.3 Hz, 1H), 6.70 (ddd, *J* = 10.7, 8.1, 1.3 Hz, 1H), 6.54 (d, *J* = 7.9 Hz, 1H), 6.43 – 6.38 (m, 2H), 5.85 (dd, *J* = 8.1, 1.5 Hz, 2H), 5.21 (dd, *J* = 7.6, 2.5 Hz, 1H), 4.96 (d, *J* = 8.9 Hz, 1H), 4.10 (d, *J* = 2.8 Hz, 1H), 3.97 – 3.77 (m, 3H), 3.42 (dd, *J* = 12.1, 7.6 Hz, 1H), 0.92 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 177.0, 168.1, 159.3 (d, *J* = 244.9 Hz), 147.4, 147.0, 129.5 (d, *J* = 8.6 Hz), 127.9 (d, *J* = 3.8 Hz), 126.9 (d, *J* = 12.6 Hz), 126.3, 124.2 (d, *J* = 3.3 Hz), 121.2, 114.8 (d, *J* = 21.8 Hz), 108.0, 107.7, 101.1, 78.9, 67.3 (d, *J* = 2.9 Hz), 61.6, 48.0, 47.0, 13.6; **IR** (thin film, cm^{-1}): 3503, 2902, 1790, 1743, 1506, 1490, 1454, 1038, 761; **TLC** (30% EtOAc/hexanes) R_f : 0.19; **HRMS** (ESI): Calculated for [M+Na]+ $C_{21}H_{20}FO_7$: 403.1193, Found: 403.1193; **$[\alpha]_D^{25}$** +42.1 (*c* = 0.15, MeOH).

Preparation of α-Benzylidene-γ-Butyrolactone 6a

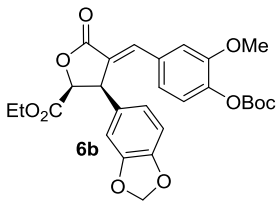


To a flame-dried 100 mL round-bottomed flask equipped with magnetic stir bar were added alcohol **8a** (1.80 g, 4.34 mmol) and dry toluene (43.4 mL). To the resulting solution was added Nafion® SAC-13 (434 mg, 100 mg/mmol). The flask was sealed with a rubber septum, purged with nitrogen, stirred, and heated to 75 °C. After 23 h the reaction was filtered (sintered glass funnel, ethyl acetate rinse). Flash chromatography (25-30% ethyl acetate/hexanes) provided **6a** (1.20 g, 3.03 mmol, 70%, >20:1 *E*:*Z*). Analytical data for **6a**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.73 (d, *J* = 2.1 Hz, 1H), 7.29 (d, *J* = 8.9 Hz, 2H), 6.80 (d, *J* = 8.8 Hz, 2H), 6.70 (d, *J* = 1.2 Hz, 2H), 6.66 (d, *J* = 1.2 Hz, 1H), 5.90 (dd, *J* = 13.9, 1.5 Hz, 2H), 5.16 (d, *J* = 8.1 Hz, 1H), 4.77 (dd, *J* = 8.1, 2.2 Hz, 1H), 3.95 – 3.79 (m, 2H), 3.78 (s, 3H), 1.03 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 171.4, 167.1, 161.3, 147.9, 147.4, 139.7, 132.8, 129.5, 125.9, 122.5, 122.3, 114.2, 108.8, 108.3, 101.2, 78.6, 61.4, 55.3, 47.0, 13.8; **IR** (thin film, cm^{-1}): 1757, 1645, 1602, 1558, 1166, 1037; **m.p.** 109-110 °C; **TLC** (30% EtOAc/hexanes) R_f : 0.18; **HRMS** (ESI): Calculated for [M+H]+ $C_{22}H_{21}O_7$: 397.1287, Found: 397.1284; **$[\alpha]_D^{25}$** +389.5 (*c* = 0.20, MeOH).

General Procedure G for Preparation of α-Benzylidene-γ-Butyrolactones 6b-6d

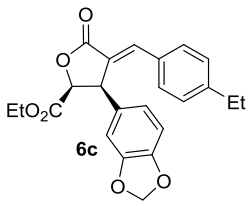


To a flame-dried scintillation vial equipped with magnetic stir bar were added alcohol **8b-d** (275 mg, 1.0 equiv) and toluene. To the resulting solution was added methyl *N*-(triethylammoniumsulfonyl)carbamate (2.0 equiv). The resulting mixture was heated to 80 °C under nitrogen. After 1.5 h the reaction was concentrated and purified by flash chromatography using the indicated solvent systems.

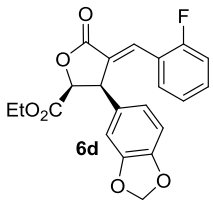


Ethyl (2*S*,3*R*)-3-(benzo[d][1,3]dioxol-5-yl)-4-((*E*)-4-((tert-butoxycarbonyl)oxy)-3-methoxybenzylidene)-5-oxotetrahydrofuran-2-carboxylate (6b): The title compound was prepared according to General Procedure G: alcohol **8b** (275 mg, 0.518 mmol, 1.0 equiv), toluene (5.2 mL), methyl *N*-(triethylammoniumsulfonyl)carbamate (Burgess reagent, 247 mg, 1.04 mmol, 2.0 equiv). Flash chromatography (20% acetone/petroleum ether) provided **6b** (163 mg, 0.318 mmol, 61%, >20:1 *E*:*Z*) as a white foam. Analytical data for **6b**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.74 (d, *J* = 2.2 Hz, 1H), 7.06 (d, *J* =

8.2 Hz, 1H), 6.97 (dd, *J* = 8.3, 2.0 Hz, 1H), 6.85 (d, *J* = 2.0 Hz, 1H), 6.73 – 6.67 (m, 2H), 6.64 (d, *J* = 1.6 Hz, 1H), 5.91 (dd, *J* = 6.6, 1.4 Hz, 2H), 5.16 (d, *J* = 8.0 Hz, 1H), 4.77 (dd, *J* = 8.0, 2.2 Hz, 1H), 3.87 (ddq, *J* = 59.4, 10.8, 7.1 Hz, 2H), 3.63 (s, 3H), 1.52 (s, 9H), 1.03 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 170.9, 166.9, 151.2, 150.9, 148.1, 147.6, 141.7, 139.4, 131.9, 129.4, 125.4, 124.5, 122.8, 122.2, 113.9, 108.7, 108.3, 101.3, 83.9, 78.7, 61.5, 55.9, 47.0, 27.5, 13.8; **IR** (thin film, cm^{-1}): 1761, 1507, 1256, 1146, 1036, 737; **TLC** (30% EtOAc/hexanes) R_f : 018; **HRMS** (ESI): Calculated for [M+H]+ $C_{27}H_{29}O_{10}$: 513.1761, Found: 513.1759; **$[\alpha]_D^{25}$** +133.3 (*c* = 0.14, MeOH).

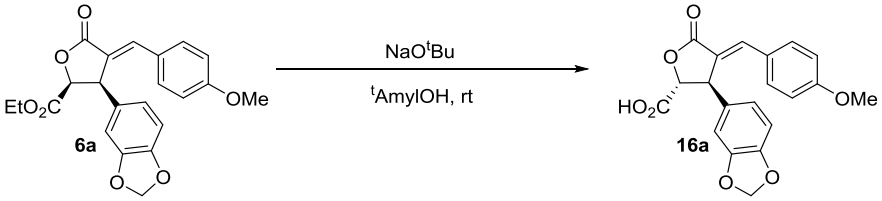


Ethyl (2*S*,3*R*)-3-(benzo[d][1,3]dioxol-5-yl)-4-((*E*)-4-ethylbenzylidene)-5-oxotetrahydrofuran-2-carboxylate (6c): The title compound was prepared according to General Procedure G: alcohol **8c** (200 mg, 0.485 mmol, 1.0 equiv), toluene (4.9 mL), methyl *N*-(triethylammoniumsulfonyl)carbamate (Burgess reagent, 231 mg, 0.97 mmol, 2.00 equiv). Flash chromatography (15% acetone/petroleum ether) provided **6c** (80 mg, 0.203 mmol, 42%, >20:1 *E*:*Z*) as a white foam. Analytical data for **6c**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.76 (d, *J* = 2.1 Hz, 1H), 7.27 (d, *J* = 8.2 Hz, 2H), 7.12 (d, *J* = 8.0 Hz, 2H), 6.71 (d, *J* = 1.3 Hz, 2H), 6.66 (t, *J* = 1.1 Hz, 1H), 5.90 (dd, *J* = 16.4, 1.4 Hz, 2H), 5.16 (d, *J* = 8.0 Hz, 1H), 4.78 (dd, *J* = 8.0, 2.2 Hz, 1H), 3.88 (ddq, *J* = 55.3, 10.7, 7.2 Hz, 2H), 2.61 (q, *J* = 7.6 Hz, 2H), 1.19 (t, *J* = 7.6 Hz, 3H), 1.04 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 171.2, 167.0, 147.9, 147.3, 147.3, 140.0, 131.0, 130.6, 129.6, 128.3, 124.5, 122.3, 108.8, 108.3, 101.2, 78.7, 61.4, 47.1, 28.7, 15.1, 13.8; **IR** (thin film, cm^{-1}): 2968, 2932, 1760, 1645, 1488, 1171, 1038, 736; **m.p.** 106-107 °C; **TLC** (30% EtOAc/hexanes) R_f : 0.26; **HRMS** (ESI): Calculated for [M+H]+ $C_{23}H_{23}O_6$: 395.1495, Found: 395.1492; **$[\alpha]_D^{25}$** +333.7 (*c* = 0.17, MeOH).



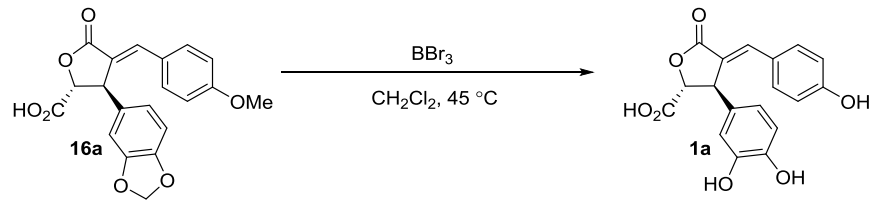
Ethyl (2*S*,3*R*)-3-(benzo[d][1,3]dioxol-5-yl)-4-((*E*)-2-fluorobenzylidene)-5-oxotetrahydrofuran-2-carboxylate (6d): The title compound was prepared according to General Procedure G: alcohol **8d** (402 mg, 1.00 mmol, 1.00 equiv), toluene (10 mL), methyl *N*-(triethylammoniumsulfonyl)carbamate (Burgess reagent, 477 mg, 2.00 mmol, 2.00 equiv). Flash chromatography (20% acetone/petroleum ether) provided **6d** (213 mg, 0.554 mmol, 55%, >20:1 *E*:*Z*) as a white foam. Analytical data for **6d**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.98 (d, *J* = 2.5 Hz, 1H), 7.29 – 7.24 (m, 1H), 7.16 (td, *J* = 7.6, 1.7 Hz, 1H), 7.03 – 6.94 (m, 2H), 6.67 – 6.55 (m, 3H), 5.88 (dd, *J* = 17.4, 1.4 Hz, 2H), 5.18 (d, *J* = 8.4 Hz, 1H), 4.77 (dd, *J* = 8.4, 2.6 Hz, 1H), 3.85 (ddq, *J* = 58.5, 10.8, 7.2 Hz, 2H), 1.00 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 170.4, 167.1, 161.0 (d, *J* = 254.1 Hz), 147.8, 147.3, 132.0 (d, *J* = 8.7 Hz), 131.9 (d, *J* = 5.1 Hz), 130.2 (d, *J* = 2.0 Hz), 128.7, 128.0, 123.9 (d, *J* = 3.7 Hz), 122.3, 121.5 (d, *J* = 12.1 Hz), 115.8, 115.7, 108.8, 108.2, 101.2, 78.6, 61.5, 47.3 (d, *J* = 2.5 Hz), 13.7; **IR** (thin film, cm^{-1}): 2984, 1764, 1653, 1488, 1445, 1238, 1198, 1175, 1038, 760; **m.p.** 113-115 °C; **TLC** (30% EtOAc/hexanes) R_f : 0.22; **HRMS** (ESI): Calculated for [M+H]+ $C_{21}H_{18}FO_6$: 385.1087, Found: 385.1083; **$[\alpha]_D^{25}$** +151.5 (*c* = 0.20, MeOH).

Epimerization/Hydrolysis of α-Benzylidene-γ-Butyrolactone 16a



To a flame-dried 25 mL round-bottomed flask were added α-benzylidene-γ-butyrolactone **6a** (250 mg, 0.631 mmol, 1.0 equiv) and *tert*-amyl alcohol (12.5 mL). To the resulting semi-solution under nitrogen was added sodium *tert*-butoxide (121 mg, 1.26 mmol, 2.0 equiv). The cream-colored suspension was stirred vigorously at room temperature. After 6 h, the reaction was cooled to 0 °C and quenched with 1M HCl (10 mL). The reaction was extracted with ethyl acetate (2 x 15 mL). The combined organic extracts were washed with brine, dried (Na_2SO_4), and concentrated *in vacuo*. The crude product was purified by flash chromatography (96.5:3:0.5 dichloromethane:methanol:acetic acid). The residue obtained was taken up in ethyl acetate (20 mL) and washed with water (3 x 10 mL), brine (10 mL), dried (Na_2SO_4) and concentrated *in vacuo* to give acid **16a** (172 mg, 0.467 mmol, 74%, 14:1 dr) as a cream colored foam. Analytical data for **16a**: **^{1}H NMR** (600 MHz, acetone-d_6): δ 7.70 (d, *J* = 2.0 Hz, 1H), 7.50 (d, *J* = 8.9 Hz, 2H), 6.94 – 6.80 (m, 5H), 6.00 (dd, *J* = 12.5, 1.1 Hz, 2H), 4.88 (d, *J* = 2.3 Hz, 1H), 4.81 (t, *J* = 2.3 Hz, 1H), 3.81 (s, 3H); **^{13}C NMR** (150 MHz, acetone-d_6): δ 171.0, 170.3, 161.6, 148.6, 147.3, 139.0, 133.9, 132.9, 126.0, 122.1, 120.3, 114.3, 108.6, 107.3, 101.5, 80.8, 54.9, 47.9; **IR** (thin film, cm^{-1}): 2905, 1759, 1645, 1601, 1514, 1168, 1038, 736; **TLC** (94.5:5:0.1 CH_2Cl_2:MeOH:AcOH) R_f : 0.36; **HRMS** (ESI): Calculated for [M+H]+ $C_{20}H_{17}O_7$: 369.0974, Found: 369.0970; **$[\alpha]_D^{25}$** +275.2 (*c* = 0.10, MeOH). **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.80 (d, J = 2.0 Hz, 1H), 7.30 (d, J = 8.6 Hz, 2H), 6.80 (d, J = 8.7 Hz, 2H), 6.79 – 6.74 (m, 3H), 5.96 (dd, J = 12.8, 1.4 Hz, 2H), 4.85 (d, J = 2.3 Hz, 1H), 4.60 (d, J = 2.2 Hz, 1H), 3.78 (s, 3H). **^{13}C NMR** (150 MHz, $CDCl_3$): δ 173.9, 171.6, 161.6, 148.7, 147.5, 141.1, 133.0, 132.8, 125.8, 120.2, 120.0, 114.4, 109.0, 107.2, 101.4, 80.7, 55.4, 48.1. Data reported by Brown, *et al.*:[5] **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.80 (d, *J* = 1.5 Hz, 1H), 7.30 (d, *J* = 8.8 Hz, 2H), 6.81 (d, *J* = 8.8 Hz, 2H), 6.77 (m, 3H), 5.97-5.95 (2d, *J* = 1.2 Hz, 2H), 4.84 (d, *J* = 2.1 Hz, 1H), 4.60 (s, 1H), 3.79 (s, 3H). **^{13}C NMR** not reported.

Preparation of Megacerotonic Acid 1a



To a flame-dried 10 mL round-bottomed flask equipped with magnetic stir bar were added acid **16a** (25.0 mg, 0.068 mmol, 1.0 equiv) and dichloromethane (2.5 mL). To the resulting solution under nitrogen at 0 °C was added dropwise boron tribromide (105 μL, 272 mg, 1.09 mmol, 16

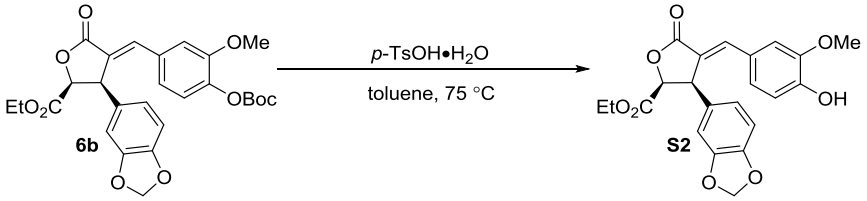
equiv). After 15 min the reaction was fitted with a reflux condenser and heated to 45 °C under nitrogen. After 6 h the reaction was cooled to room temperature, quenched through the condenser with dropwise addition of 0.1M NaOH (8 mL), and stirred vigorously for 20 min. The resulting mixture was extracted with ethyl acetate (2 x 10 mL). The combined organic extracts were washed with brine, dried (Na_2SO_4) and concentrated *in vacuo*. A portion of the crude product (5/6) was purified by semi-preparative HPLC (A: 10 mM NH_4OAc in water, B: 90:10 acetonitrile:10 mM NH_4OAc in water; A:B 90:10 to 50:50 over 41 min). The fractions collected were concentrated on rotatory evaporator to remove acetonitrile and extracted with ethyl acetate (10 mL). The aqueous layer was brought to pH = 0 with concentrated sulfuric acid and extracted with ethyl acetate (2 x 10 mL). The combined organic extracts (excluding the first) were washed with brine, dried (Na_2SO_4) and concentrated to yield megacerotonic acid **1a** (6.6 mg, 0.019 mmol, 34%) as colorless, amorphous solid. Analytical data for **1a**: **^{1}H NMR** (600 MHz, D_2O, referenced to TMS): δ 7.72 (s, 1H), 7.33 (d, *J* = 8.5 Hz, 2H), 6.88 (s, 1H), 6.82 (d, *J* = 8.2 Hz, 1H), 6.77 (d, *J* = 8.7 Hz, 2H), 6.73 (d, *J* = 8.2 Hz, 1H) 4.79 (overlapped with H_2O, 1H), 4.65 (s, 1H); **^{1}H NMR** (600 MHz, MeOD): δ 7.67 (d, *J* = 2.0 Hz, 1H), 7.29 (d, *J* = 8.8 Hz, 2H), 6.74 (d, *J* = 8.1 Hz, 1H), 6.73 (d, *J* = 2.2 Hz, 1H), 6f.70 (d, *J* = 8.7 Hz, 2H), 6.63 (dd, *J* = 8.2, 2.2 Hz, 1H), 4.78 (d, *J* = 2.2 Hz, 1H), 4.51, (s, 1H); **^{13}C NMR** (150 MHz, D_2O, referenced to TMS): δ 176.4, 176.1, 159.1, 145.4, 144.4, 141.9, 134.3, 133.2, 126.2, 122.7, 120.3, 117.5, 116.6, 115.9, 84.5, 49.0; **IR** (thin film, cm^{-1}): 3420, 2360, 2341, 1733, 1717, 1636, 1603, 1517, 1254, 1191, 1171, 1058, 834; **HRMS** (ESI): Calculated for [M+Na]+ $C_{18}H_{14}NaO_7$: 365.0637, Found: 365.0638; **$[\alpha]_D^{26}$** +171.9 (*c* = 0.14, 5% AcOH/H_2O).

Comparison of Physicochemical Properties for Megacerotonic Acid			
Data Type	**Data Reported in Isolation Paper**	**Our Synthetic Sample**	**Data Reported for Papin Synthetic Sample**
^{1}H NMR (D_2O)	^{1}H NMR (300 MHz, D_2O) 4.52 (1H, dd, *J*= 1.6, 3.0 Hz) 4.74 (1H, d, *J*= 3.0 Hz) 6.53 (1H, dd, *J*= 2.0,6.2 Hz) 6.62 (2H, d, *J*= 6.6 Hz) 6.70 (1H, d, *J*= 6.2 Hz) 6.82 (1H, d, *J*= 2.0 Hz) 7.14 (2H, d. *J*= 6.6 Hz) 7.58 (1H, d, *J*= 1.6 Hz)	^{1}H NMR (600 MHz, D_2O, TMS) 4.65 (s, 1H) Overlapped with H_2O 6.73 (d, *J* = 8.2 Hz, 1H) 6.77 (d, *J* = 8.7 Hz, 2H) 6.82 (d, *J* = 8.2 Hz, 1H) 6.88 (s, 1H) 7.33 (d, *J* = 8.5 Hz, 2H) 7.72 (s, 1H)	^{1}H NMR (400 MHz, D_2O) 4.63 (ddt, *J* = 2.5 Hz, 1H) 4.83 (d, *J* = 3.1 Hz, 1H) 6.66 (dd, *J* = 8.3, 2.2 Hz, 1H) 6.72 (d, *J* =8.8 Hz, 2H) 6.80 (d, *J* = 8.3 Hz, 1H) 6.87 (d, *J* = 2.2 Hz, 1H) 7.24 (d, *J* =8.8 Hz, 2H) 7.66 (d, *J* = 2.0 Hz, 1H)
^{1}H NMR (MeOD)	Not reported	4.51 (s, 1H) 4.78 (d, *J* = 2.2 Hz, 1H) 6.63 (dd, *J* = 8.2, 2.2 Hz, 1H) 6.70 (d, *J* = 8.7 Hz, 2H) 6.73 (d, *J* = 2.2 Hz, 1H) 6.74 (d, *J* = 8.1 Hz, 1H) 7.29 (d, *J* = 8.8 Hz, 2H) 7.67 (d, *J* = 2.0 Hz, 1H).	Not reported
^{13}C NMR	^{13}C NMR (75.5MHz, D_2O) 177.6, 177.5, 161.0, 147.3, 146.3, 144.0, 136.2, 134.8, 127.8, 123.9, 122.0, 119.3, 118.4, 117.8, 85.8, 50.6	^{13}C NMR (150 MHz, D_2O) 176.4, 176.1, 159.1, 145.4, 144.4, 141.9, 134.3, 133.2, 126.2, 122.7, 120.3, 117.5, 116.6, 115.9, 84.5, 49.0.	^{13}C NMR (100 MHz, acetone-d_6) 172.4, 171.5, 160.6, 146.6, 145.7, 139.9, 134.1, 132.7, 126.2, 122.5, 119.3, 116.8, 116.7, 114.7, 82.0, 48.8
Rotation (5% AcOH)	+233.0 (c 1.66)	+171.9 (c 0.14)	NA
IR	1735	3420, 2360, 2341, 1733, 1717, 1636, 1603, 1517, 1254, 1191, 1058, 834	1735
MS	343 (M+H)	360.1083 (M+Na = 360.1086)	Not reported

The spectral properties of synthetic megacerotonic acid vary significantly from both those reported by Takeda in its isolation as well as that reported by Brown, *et al.* in their racemic total synthesis. We were unable to obtain either a natural sample or synthetic sample. We postulate based on our experience with shimobashiric acid A (*vide infra*) that the differences in NMR shifts observed are a result of concentration differences. A concentration vs. chemical shift study (below) appears to support this hypothesis. Finally, we note that our ^{1}H NMR data for **16a** matches that of the pentultimate intermediate in the published racemic synthesis (*vide supra*).

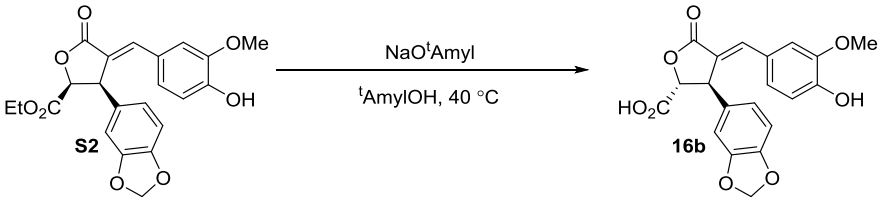
^{1}H NMR shifts of our synthetic sample as a function of concentration (600 MHz, D_2O)	
Concentration	**Resonance (δ)**
11.6 mg/mL	4.56, 4.73, 6.68, 6.73, 6.78, 6.86, 7.30, 7.67
5.8 mg/mL	4.59, 4.74, 6.72, 6.77, 6.81, 6.87, 7.34, 7.70
2.9 mg/mL	4.60, 4.75, 6.74, 6.78, 6.82, 6.87, 7.36, 7.72
1.5 mg/mL	4.60, 4.75, 6.75, 6.80, 6.83, 6.87, 7.37, 7.73
0.7 mg/mL	4.61, 4.75, 6.77, 6.80, 6.83, 6.87, 7.38, 7.73

Hydrolysis of *Tert*-Butoxycarbonyl α-Benzylidene-γ-Butyrolactone S2



To a scintillation vial were added α-benzylidene-γ-butyrolactone **6b** (50 mg, 0.098 mmol, 1.0 equiv) and toluene (2.0 mL). To the resulting solution was added *p*-toluenesulfonic acid monohydrate (37.1 mg, 0.195 mmol, 2.0 equiv). The resulting mixture was heated to 75 °C. After 4 h the reaction was cooled to room temperature, diluted with 1M HCl (10 mL), and extracted with ethyl acetate (2 x 10 mL). The combined organic extracts were washed with brine, dried (Na_2SO_4) and concentrated *in vacuo*. The crude product was purified by flash chromatography (30-40% ethyl acetate/hexanes) to give α-benzylidene-γ-butyrolactone **S2** (36 mg, 0.087 mmol, 89%) as a pale yellow, viscous oil. Analytical data for **S2**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.71 (d, *J* = 2.2 Hz, 1H), 6.95 (dd, *J* = 8.3, 2.0 Hz, 1H), 6.83 (d, *J* = 8.2 Hz, 1H), 6.77 (d, *J* = 2.0 Hz, 1H), 6.73 – 6.69 (m, 2H), 6.67 (d, *J* = 1.6 Hz, 1H), 5.98 (s, 1H), 5.90 (dd, *J* = 6.9, 1.4 Hz, 2H), 5.17 (d, *J* = 8.2 Hz, 1H), 4.79 (dd, *J* = 8.3, 2.2 Hz, 1H), 3.95 – 3.76 (m, 2H), 3.67 (s, 3H), 1.03 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 171.4, 167.2, 148.1, 148.0, 147.4, 146.5, 140.4, 129.6, 126.7, 125.7, 122.3, 121.9, 114.7, 112.3, 108.8, 108.3, 101.3, 78.6, 61.4, 55.9, 47.0, 13.7; **IR** (thin film, cm^{-1}): 3420, 2917, 1750, 1636, 1517, 1164, 736; **TLC** (50% EtOAc/hexanes) R_f : 0.18; **HRMS** (ESI): Calculated for [M+H]+ $C_{22}H_{21}O_8$: 413.1236, Found: 413.1233; **$[\alpha]_D^{25}$** +426.0 (*c* = 0.11, MeOH).

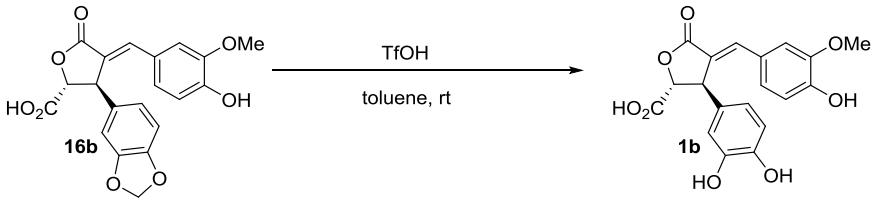
Epimerization/Hydrolysis of α-Benzylidene-γ-Butyrolactone 16b



To a flame-dried 10 mL round-bottomed flask were added α-benzylidene-γ-butyrolactone **S1** (20 mg, 0.048 mmol, 1.0 equiv) and *tert*-amyl alcohol (2.5 mL). To the resulting solution, after heating to 40 °C, a solution of sodium *tert*-amylate in *tert*-amyl alcohol (0.87 mL, 0.145 mmol, 3.0 equiv) was added dropwise. After 6.5 h, the reaction was cooled to 0 °C and quenched with 1M HCl (3 mL). The reaction was extracted with ethyl acetate (2 x 5 mL). The combined organic extracts were washed with brine, dried (Na_2SO_4), and concentrated *in vacuo*. The crude product was purified by flash chromatography (98:2:0.3 dichloromethane:methanol:acetic acid). The residue obtained was taken up in ethyl acetate (10 mL) and washed with water (5 x 8 mL), brine (5 mL), dried (Na_2SO_4) and concentrated *in vacuo* to give acid **16b** (13.7 mg, 0.036 mmol, 73%, 14:1 dr) as a clear oil. Analytical data for **16b**: **^{1}H NMR** (600 MHz, Acetone-d_6) δ 7.67 (d, *J* = 1.5 Hz, 1H), 7.09 (dd, *J* = 8.3, 2.1 Hz, 1H), 7.02 (d, *J* = 2.0 Hz, 1H), 6.92 – 6.85 (m, 3H), 6.82 (d, *J* =

8.2 Hz, 1H), 6.00 (dd, *J* = 7.3, 1.1 Hz, 2H), 4.82 (s, 2H), 3.66 (s, 3H).**^{13}C NMR** (150 MHz, $CDCl_3$): δ 172.1, 171.5, 150.0, 149.5, 148.3, 148.2, 140.5, 135.2, 127.2, 126.4, 122.2, 121.3, 116.1, 114.2, 109.6, 108.1, 102.4, 81.8, 56.2, 48.8; **IR** (thin film, cm^{-1}): 3502, 2917, 2360, 1749, 1716, 1636, 1518, 1254, 1163, 1036, 817; **TLC** (94.5:5:0.1 CH_2Cl_2:MeOH:AcOH) R_f : 0.27; **HRMS** (ESI): Calculated for [M+H]+ $C_{20}H_{17}O_8$: 385.0923, Found: 385.0919; **$[\alpha]_D^{25}$** +222.6 (*c* = 0.17, MeOH).

Preparation of Shimobashiric Acid A 1b

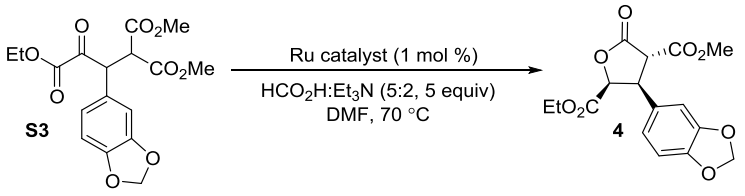


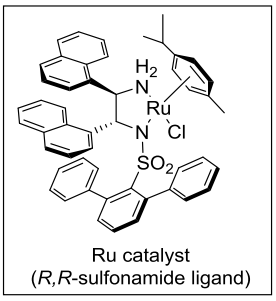
To a solution of acid **16b** (9.6 mg, 0.025 mmol) in toluene (2.0 mL) at room temperature was added trifluoromethanesulfonic acid (excess, ~20 drops). After 2 min, the reaction was diluted with water (5 mL) and extracted with ethyl acetate (2 x 5 mL). The combined organic extracts were washed with brine, dried (Na_2SO_4) and concentrated. A portion of the crude product (5/6) was purified by semi-preparative HPLC (A: 10 mM NH_4OAc in water, B: 90:10 acetonitrile:10 mM NH_4OAc in water; A:B 90:10 to 50:50 over 41 min). The fractions collected were concentrated on rotatory evaporator to remove acetonitrile and extracted with ethyl acetate (10 mL). The aqueous layer was brought to pH=0 with concentrated sulfuric acid and extracted with ethyl acetate (2 x 10 mL). The combined organic extracts (excluding the first) were washed with brine, dried (Na_2SO_4) and concentrated to yield shimobashiric acid A **1b** (4.5 mg, 0.012 mmol, 48%) as a colorless oil. Analytical data for **1b**: **^{1}H NMR** (600 MHz, MeOD): δ 7.68 (d, *J* = 2.0 Hz, 1H), 6.98 (dd, *J* = 8.3, 2.0 Hz, 1H), 6.88 (d, *J* = 2.0 Hz, 1H), 6.77 – 6.75 (m, 2H), 6.75 (d, *J* = 6.6 Hz, 1H), 6.66 (dd, *J* = 8.2, 2.2 Hz, 1H), 4.78 (d, *J* = 2.3 Hz, 1H), 4.54 (d, *J* = 2.3 Hz, 1H), 3.55 (s, 3H); **^{13}C NMR** (150 MHz, MeOD) δ 174.4, 173.2, 150.7, 149.0, 147.3, 146.3, 141.8, 132.9, 128.0, 126.7, 122.0, 119.2, 117.1, 116.4, 114.9, 114.1, 83.1, 56.3, 49.7; **IR** (thin film, cm^{-1}): 3392, 2919, 2849, 1734, 1635, 1597, 1518, 1289, 1204, 1179, 1059, 820; **HRMS** (ESI): Calculated for [M+Na]+ $C_{19}H_{16}NaO_8$: 395.0743, Found: 395.0740; **$[\alpha]_D^{26}$** +295.6 (*c* = 0.20, MeOH).

Comparison of Physicochemical Properties for Shimobashiric Acid A			
Data Type	**Data Reported in Isolation Paper**	**Our Synthetic Sample**	**Obtained Natural Sample**
^{1}H NMR (MeOD)	^{1}H NMR (400 MHz, MeOD) 3.56 (s, 3H) 4.55 (bs, 1H) 4.78 (overlapped, 1H) 6.67 (dd, *J* = 8.0, 2.0 Hz, 1H) 6.75 (d, *J* = 8.5 Hz, 1H) 6.77 (d, *J* = 2.0 Hz, 1H) 6.77 (d, *J* = 8.0 Hz, 1H) 6.89 (d, *J* = 2.0 Hz, 1H) 6.99 (dd, *J* = 8.5, 2.0 Hz, 1H) 7.68 (d, *J* = 2.0 Hz, 1H)	^{1}H NMR (600 MHz, MeOD) 3.55 (s, 3H) 4.54 (d, *J* = 2.3 Hz, 1H) 4.78 (d, *J* = 2.3 Hz, 1H) 6.66 (dd, *J* = 8.2, 2.2 Hz, 1H) 6.75 (d, *J* = 6.6 Hz, 1H) 6.77 – 6.75 (m, 2H) 6.88 (d, *J* = 2.0 Hz, 1H) 6.98 (dd, *J* = 8.3, 2.0 Hz, 1H) 7.68 (d, *J* = 2.0 Hz, 1H)	^{1}H NMR (600 MHz, MeOD) 3.55 (s, 3H) 4.52 (s, 1H) 4.59 (d, *J* = 2.2 Hz, 1H) 6.68 (dd, *J* = 8.1, 2.2 Hz, 1H) 6.73 (s, 1H) 6.74 (s, 1H) 6.81 (d, *J* = 2.2 Hz, 1H) 6.90 (d, *J* = 2.0 Hz, 1H) 6.96 (dd, *J* = 8.1, 2.0 Hz, 1H) 7.61 (d, *J* =2.1 Hz, 1H)
^{13}C NMR (MeOD)	^{13}C NMR (100 MHz, MeOD) 174.5, 173.7, 150.7, 149.1, 147.4, 146.3, 141.7, 133.7, 128.0, 126.9, 122.5, 119.4, 117.2, 116.4, 115.1, 114.3, 83.6, 56.4, 49.5	^{13}C NMR (150 MHz, MeOD) 174.4, 173.2, 150.7, 149.0, 147.3, 146.3, 141.8, 132.9, 128.0, 126.7, 122.0, 119.2, 117.1, 116.4, 114.9, 114.1, 83.1, 56.3, 49.7	NA
Rotation (MeOH)	+205.0 (c 0.16)	+295.6 (c 0.20)	NA
IR	Not Reported	3392, 2919, 2849, 1734, 1635, 1597, 1518, 1289, 1204, 1179, 1059, 820	NA
MS	373.0928	373.0921 (M+H = 373.0923)	NA

The data reported by Murata and coworkers for natural shimobashiric acid A agrees well with data obtained with our synthetic sample. We obtained a natural sample of shimobashiric acid A (~0.16 mg) from Murata and the ^{1}H NMR spectrum of this sample varied significantly for several resonances relative to those reported in its isolation (e.g. 4.58 ppm vs 4.78 ppm). Postulating the presence of concentration dependent shifts, we analyzed an equal mixture of natural (0.08 mg) and synthetic shimobashiric acid A (0.08 mg) by ^{1}H NMR spectroscopy. This analysis clearly showed coalescence of the resonances in question, indicating concentration dependent changes in NMR resonances. This feature may be relevant in the analysis of megacerotonic acid (*vide supra*).

Preparation of γ-Butyrolactone 4

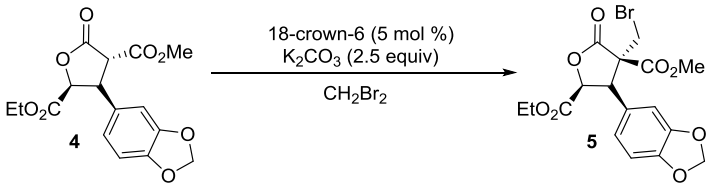




To a flame-dried 250 mL round-bottomed flask equipped with magnetic stir bar were added $[RuCl_2(p\text{-cymene})]_2$ (65.6 mg, 0.107 mmol, 0.005 equiv), ligand **L1** (259 mg, 0.428 mmol, 0.02 equiv), and anhydrous DMF (30 mL). The resulting solution was stirred under a N_2 atmosphere at 70 °C. After 1 h, the reaction solution was cooled to room temperature and a solution of α-keto-β-aryl ester **S3** (7.85 g, 21.4 mmol, 1.0 equiv) in DMF (20 mL) was added. The reaction solution was diluted with DMF (57 mL) and formic acid:triethylamine azeotrope (5:2, 9.3 mL, 107 mmol, 5.0 equiv) was added. The resulting solution was heated to 70 °C. After 20 h, the reaction was cooled to room temperature and diluted with water (200 mL) and extracted with ethyl acetate (250 mL, 50 mL). The combined organic extracts were washed with water (2 x 150 mL), brine (75 mL), dried (Na_2SO_4) and concentrated to give crude γ-butyrolactone **4** as a 19:1 mixture of diastereomers. The diastereomer ratio was determined by ^1H NMR spectroscopic analysis of the crude reaction mixture by comparison of the integration of resonances at δ 5.11 (major diastereomer) and δ 4.82 (minor diastereomer). **HPLC Analysis**: Chiralpak IB column, 15% Isopropanol/Hexanes, 1.0 mL/min, 210 nm; t_{major} = 15.2 min t_{minor} = 18.4 min, 90:10 er.

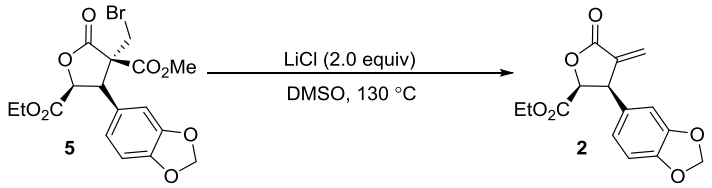
The crude product was eluted over a plug of silica gel with a mixture of ethyl acetate/hexanes (30/70, 300 mL). The resulting oil was recrystallized from diethyl ether/hexanes to yield γ-butyrolactone **4** (3.55 g, 10.6 mmol, 49% yield, >20:1 dr) as white crystalline solid. Analytical data for **4**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 6.76 (d, *J* = 7.8 Hz, 1H), 6.68-6.66 (m, 2H), 5.95 (s, 2H), 5.12 (d, *J* = 8.5 Hz, 1H), 4.41 (dd, *J* = 11.5, 8.5 Hz, 1H), 4.13 (d, *J* = 11.5 Hz, 1H), 4.00-3.94 (m, 1H), 3.92-3.87 (m, 1H), 3.79 (s, 3H), 0.97 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 170.3, 167.7, 167.2, 148.2, 147.8, 126.1, 120.9, 108.6, 107.8, 101.4, 78.9, 61.8, 53.3, 49.1, 47.6, 13.7; **IR** (thin film, cm^{-1}): 1793, 1741, 1507, 1143, 1038; **m.p.** 85-87 °C; **TLC** (30% EtOAc/hexanes) R_f : 0.26; **HRMS** (ESI): Calculated for [M+Na]+ $C_{16}H_{16}NaO_8$: 359.0743, Found: 359.0738; **HPLC Analysis**: Chiralpak IB column, 15% Isopropanol/Hexanes, 1.0 mL/min, 210 nm; t_{major} = 15.2 min t_{minor} = 18.4 min, 93:7 er; **$[\alpha]_D^{25}$** +195.8 (*c* = 0.25, MeOH).

Preparation of γ-Butyrolactone 5



To a 1-dram vial were added γ-butyrolactone **4**, potassium carbonate (51.4 mg, 0.372 mmol, 2.5 equiv), 18-crown-6 (2.0 mg, 0.007 mmol, 0.05 equiv), and dibromomethane (0.5 mL). The resulting slurry was stirred vigorously. After 4.5 days, the reaction was partitioned between ethyl acetate (5 mL) and water (5 mL). The aqueous layer was separated and extracted with ethyl acetate (5 mL). The combined organic extracts were washed with brine (5 mL), dried (Na_2SO_4) and concentrated to give crude γ-butyrolactone **5** as a single diastereomer. Flash chromatography (25-30% ethyl acetate/hexanes) provided γ-butyrolactone **5** (55.8 mg, 0.130 mmol, 87% yield) as white solid. Analytical data for **5**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 6.71-6.69 (m, 1H), 6.64-6.63 (m, 2H), 5.93 (d, *J* = 1.3 Hz, 1H), 5.36 (d, *J* = 8.4 Hz, 1H), 4.14 (d, *J* = 8.4 Hz, 1H), 4.02-3.94 (m, 3H), 3.90 (d, *J* = 10.8 Hz, 1H), 3.43 (s, 3H), 0.97 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 170.3, 166.3, 166.2, 147.8, 147.6, 127.3, 122.8, 109.2, 108.1, 101.4, 78.1, 61.7, 61.6, 52.9, 52.3, 33.1, 13.7; **IR** (thin film, cm^{-1}): 2983, 1799, 1742, 1490, 1447, 1038, 736; **m.p.** 130-132 °C; **TLC** (30% EtOAc/hexanes) R_f : 0.21; **HRMS** (ESI): Calculated for [M+Na]+ $C_{17}H_{17}BrNaO_8$: 451.0004, Found: 451.0001; **$[\alpha]_D^{25}$** +110.3 (*c* = 0.20, MeOH).

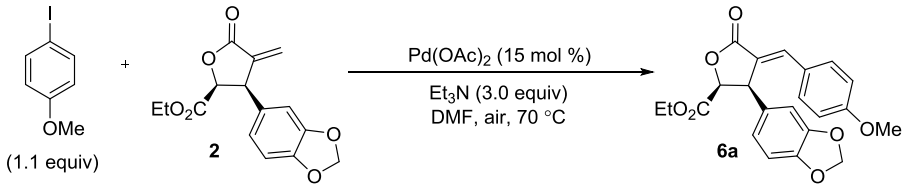
Preparation of α-Methylidene-γ-Butyrolactone 2



To a 20 mL scintillation vial were added γ-butyrolactone **5** (639.5 mg, 1.49 mmol, 1.0 equiv), lithium chloride (126 mg, 2.98 mmol, 2.0 equiv), and anhydrous DMSO (10 mL). The reaction vial was purged with N_2 and heated to 130 °C. After 5.5 h the reaction was cooled to room temperature and partitioned between ethyl acetate (25 mL) and water (25 mL). The aqueous layer was separated and extracted with ethyl acetate (15 mL). The combined organic extracts were washed with water (10 mL), brine (10 mL), dried (Na_2SO_4) and concentrated. Flash chromatography (20-30% ethyl acetate/hexanes) provided α-methylidene-γ-butyrolactone **2** (245 mg, 0.844 mmol, 57% yield) as a viscous very pale orange oil. Analytical data for **2**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 6.77 (d, *J* = 8.0 Hz, 1H), 6.68 (dd, *J* = 8.0, 1.8 Hz, 1H), 6.64 (d, *J* = 1.8 Hz, 1H), 6.51 (d, *J* = 3.3 Hz, 1H), 5.95 (d, *J* = 1.8 Hz, 2H), 5.62 (d, *J* = 3.0 Hz, 1H), 5.13 (d, *J* = 9.3 Hz, 1H), 4.52, (dt, *J* = 9.3, 3.1 Hz, 1H), 3.94-3.89 (m, 1H), 3.84-3.79 (m, 1H), 0.97 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 169.2, 167.7, 148.0, 147.6, 135.9, 128.6, 125.0, 122.7, 108.9, 108.4, 101.3, 78.4, 61.6, 48.3, 13.7; **IR** (thin film, cm^{-1}): 2985, 2904, 1780, 1746,

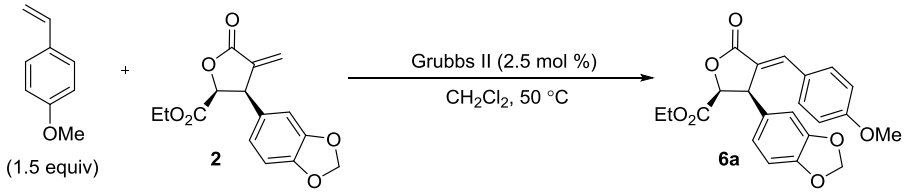
1490, 1446, 1095, 1068, 1038, 736; **TLC** (30% EtOAc/hexanes) R_f : 0.26; **HRMS** (ESI): Calculated for [M+H]+ $C_{15}H_{15}O_6$: 291.0869, Found: 291.0864 **$[\alpha]_D^{25}$** +256.2 (*c* = 0.13, MeOH).

Preparation of α-Benzylidene-γ-Butyrolactone 6a Via Heck Coupling



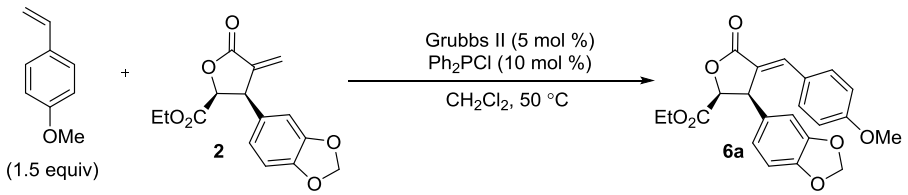
To a flame-dried 1-dram vial equipped with magnetic stir bar were added α-methylene-γ-butyrolactone **2** (26 mg, 0.09 mmol, 1.0 equiv), 4-iodoanisole (23.4 mg, 0.10 mmol, 1.1 equiv), palladium(II) acetate (3.0 mg, 0.013 mmol, 15 mol %) and DMF (400 μL) under an atmosphere of air. Triethylamine (38 μL, 0.27 mmol, 3.0 equiv) was added and the resulting mixture heated to 70 °C. After 20 h, the reaction was cooled to rt and diluted with ethyl acetate (5 mL), washed with water (2 x 5 mL), brine (5 mL), dried (Na_2SO_4) and concentrated. The crude product was purified by flash chromatography to yield **6a** (6.6 mg, 0.017 mmol, 19%) as a clear oil. Analytical data for **6a** matched that reported above.

Attempt 1 at Preparation of α-Benzylidene-γ-Butyrolactone 6a Via Cross Metathesis



To a flame-dried 1-dram vial sealed with Teflon puncture cap under N_2 equipped with magnetic stir bar containing Grubbs second generation catalyst (1.8 mg, 0.002 mmol, 2.5 mol %) was added a solution of α-methylene-γ-butyrolactone **2** (25 mg, 0.09 mmol, 1.0 equiv) and 4-vinylanisole (17 μg, 0.13 mmol, 1.5 equiv) in dichloromethane (200 μL) at room temperature. The resulting solution was heated to 50 °C. After 20 h the reaction was concentrated on rotary evaporator. ^{1}H NMR of the crude product showed no conversion of α-methylene-γ-butyrolactone **2** to desired product **6a**.

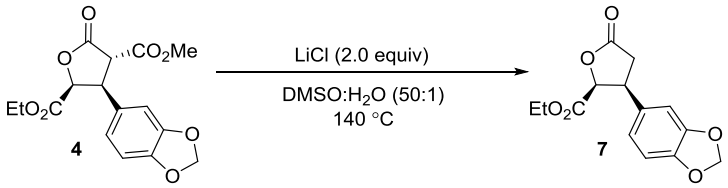
Attempt 2 at Preparation of α-Benzylidene-γ-Butyrolactone 6a Via Cross Metathesis



To a flame-dried 1-dram vial sealed with Teflon puncture cap under N_2 equipped with magnetic stir bar containing Grubbs second generation catalyst (3.6 mg, 0.004 mmol, 5.0 mol %) and

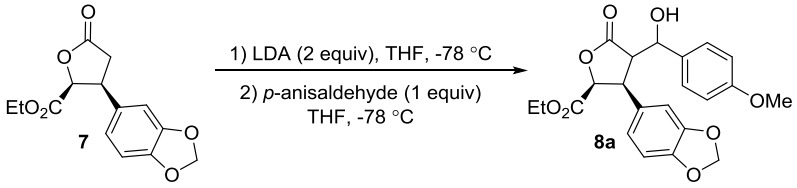
chlorodiphenylphosphine (1.2 μL, 0.10 mmol, 10 mol %) was added a solution of α-methylene-γ-butyrolactone **2** (25 mg, 0.09 mmol, 1.0 equiv) and 4-vinylanisole (17 μg, 0.13 mmol, 1.5 equiv) in dichloromethane (200 μL) at room temperature. The resulting solution was heated to 50 °C. After 20 h the reaction was concentrated on rotary evaporator. ^{1}H NMR of the crude product showed no conversion of α-methylene-γ-butyrolactone **2** to desired product **6a**.

Preparation of γ-Butyrolactone 7



To a 100 mL round-bottomed flask were added γ-butyrolactone **4** (1.00 g, 2.97 mmol, 1.0 equiv), lithium chloride (252 mg, 5.95 mmol, 2.0 equiv), anhydrous DMSO (30 mL), and water (0.6 mL). The reaction flask was purged with N_2 and heated to 140 °C. After 19 h, the reaction was cooled to room temperature and partitioned between ethyl acetate (50 mL) and water (50 mL). The aqueous layer was separated and extracted with ethyl acetate (25 mL). The combined organic extracts were washed with water (2 x 20 mL), brine (15 mL), dried (Na_2SO_4) and concentrated. Flash chromatography (30% ethyl acetate/hexanes) provided γ-butyrolactone **7** (723 mg, 2.60 mmol, 87% yield) as a pale yellow solid. Analytical data for **7**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 6.75 (d, *J* = 7.9 Hz, 1H), 6.68-6.65 (m, 2H), 5.94 (s, 2H), 5.07 (d, *J* = 8.2 Hz, 1H), 4.03-3.98 (m, 2H), 3.98-3.92 (m, 1H), 3.90-3.85 (m, 1H), 2.98 (dd, *J* = 17.3, 10.1 Hz, 1H), 2.81 (dd, *J* = 17.3, 8.7 Hz, 1H), 0.97 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$): δ 175.1, 167.9, 148.0, 147.4, 128.4, 120.8, 108.4, 107.8, 101.3, 80.1, 61.4, 43.8, 32.5, 13.7; **IR** (thin film, cm^{-1}): 2984, 2904, 1793, 1742, 1506, 1447, 1038, 818; **m.p.** 80-81 °C; **TLC** (30% EtOAc/hexanes) R_f : 0.21; **HRMS** (ESI): Calculated for [M+H]+ $C_{14}H_{15}O_6$: 279.0869, Found: 279.0866; **$[\alpha]_D^{25}$** +118.5 (*c* = 0.22, MeOH).

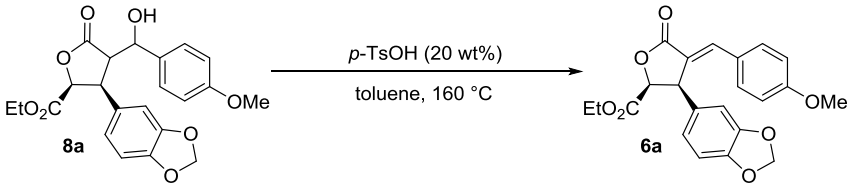
Preparation of γ-Butyrolactone 8a Via Aldol Reaction



To a flame-dried 1 dram vial equipped with magnetic stir bar containing diisopropylamine (53 μL, 0.38 mmol, 2.1 equiv) at 0 °C was added a solution of *n*-butyl lithium in hexanes (0.21 mL, 2.0 equiv). After 15 min, tetrahydrofuran (0.4 mL) was added. The resulting solution was cooled to -78 °C and a solution of γ-butyrolactone **7** (50 mg, 0.180 mmol, 1.0 equiv) in tetrahydrofuran (1.0 mL) was added dropwise. After 15 min, neat *p*-anisaldehyde (22 μL, 0.180 mmol, 1.0 equiv) was added dropwise. After 10 min, the reaction was quenched with aqueous saturated ammonium chloride (2 mL) and allowed to warm to room temperature. The resulting mixture

was diluted with water (2 mL) and extracted with ethyl acetate (2 x 5 mL). The combined organic extracts were washed with brine, dried (Na_2SO_4) and concentrated. Flash chromatography (20-40% ethyl acetate/hexanes) provided **8a** (33 mg, 0.079 mmol, 44%) as a 2:1 mixture of diastereomers.

Preparation of α-Benzylidene-γ-Butyrolactone 6a Via Elimination



To a solution of alcohol **8a** (27 mg, 0.065 mmol, 1.0 equiv) in toluene (5 mL) was added *p*-toluenesulfonic acid (5 mg, 20 wt%). The resulting mixture was heated to 160 °C under nitrogen. After 1 h the reaction was cooled to room temperature and concentrated. Flash chromatography (30% ethyl acetate/hexanes) provided **6a** (13.4 mg, 0.034 mmol, 52%, >20:1 *E:Z*) as a clear oil. Analytical data for **6a** matched that shown above.

References

(1) Evans, D. A.; Burgey, C. S.; Paras, N. A.; Vojkovsky, T.; Tregay, S. W. *J. Am. Chem. Soc.***1998**, *120*, 5824.

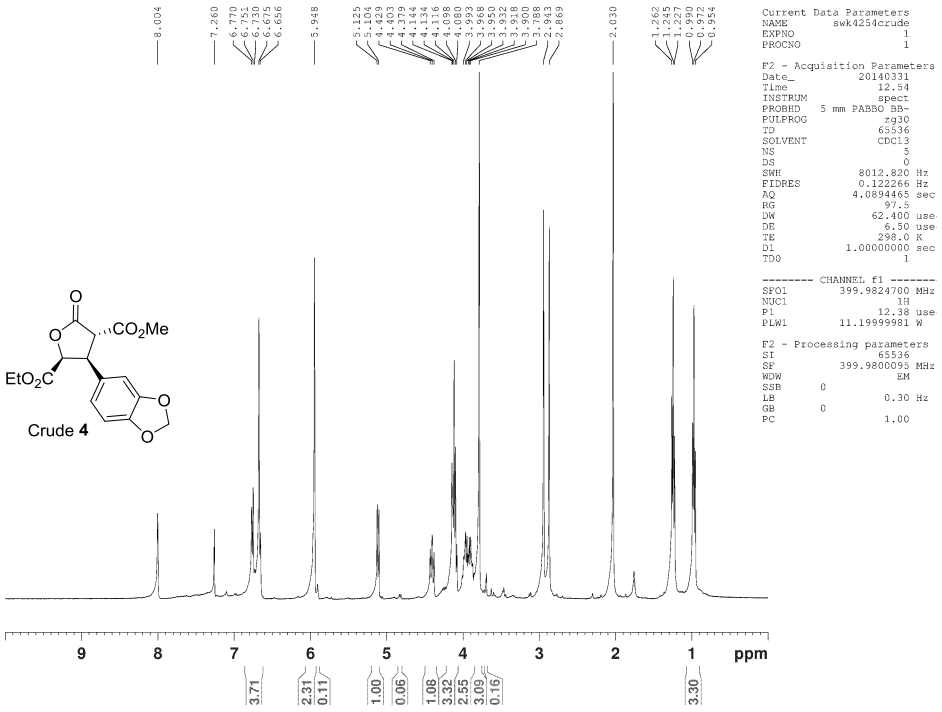
(2) Kim, H.; Nguyen, Y.; Yen, C. P.-H.; Chagal, L.; Lough, A. J.; Kim, B. M.; Chin, J. *J. Am. Chem. Soc.* **2008**, *130*, 12184.

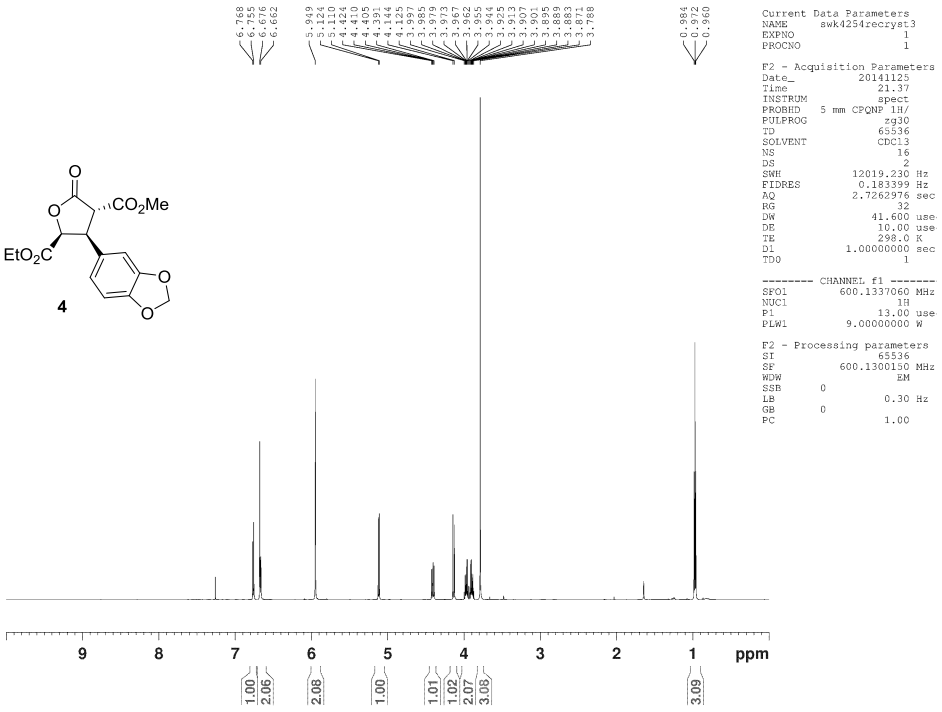
(3) Steward, K. M.; Gentry, E. C.; Johnson, J. S. *J. Am. Chem. Soc.* **2012**, *134*, 7329.

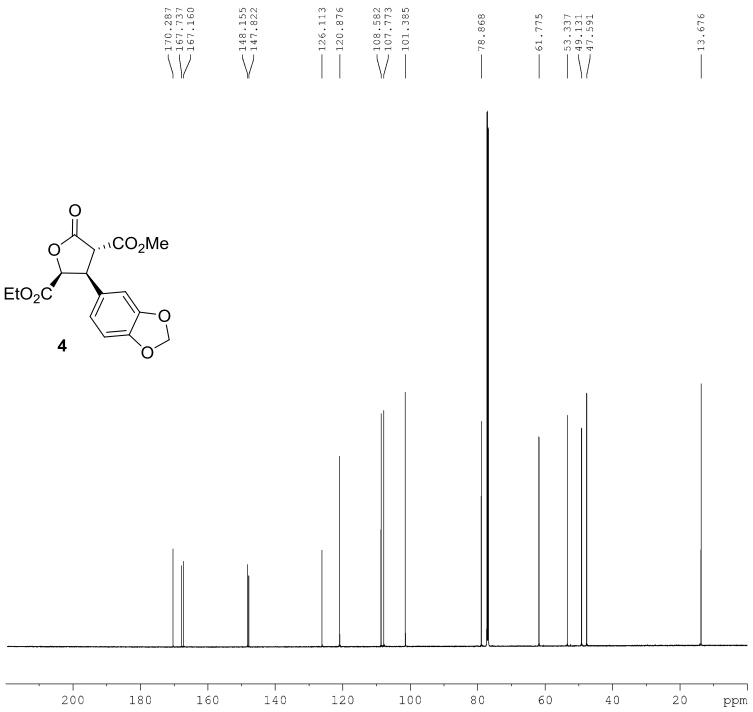
(4) Chiaradia, L. D.; Mascarello, A.; Purificacao, M.; Vernal, J.; Cordeiro, M. N. S.; Zenteno, M. E.; Villarino, A.; Nunes, R. J.; Yunes, R. A.; Terenzi, H. *Bioorg. Med. Chem. Lett.* **2008**, *18*, 6227.

(5) Brown, E.; Dhal, R.; Papin, N. *Tetrahedron* **1995**, *51*, 13061.

X. NMR Spectra





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DS                       4
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FIDRES          0.550197 Hz
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RG                     203
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PCPD2               80.00 usec
PLW2          9.00000000 W
PLW12         0.26587000 W
PLW13         0.17016000 W

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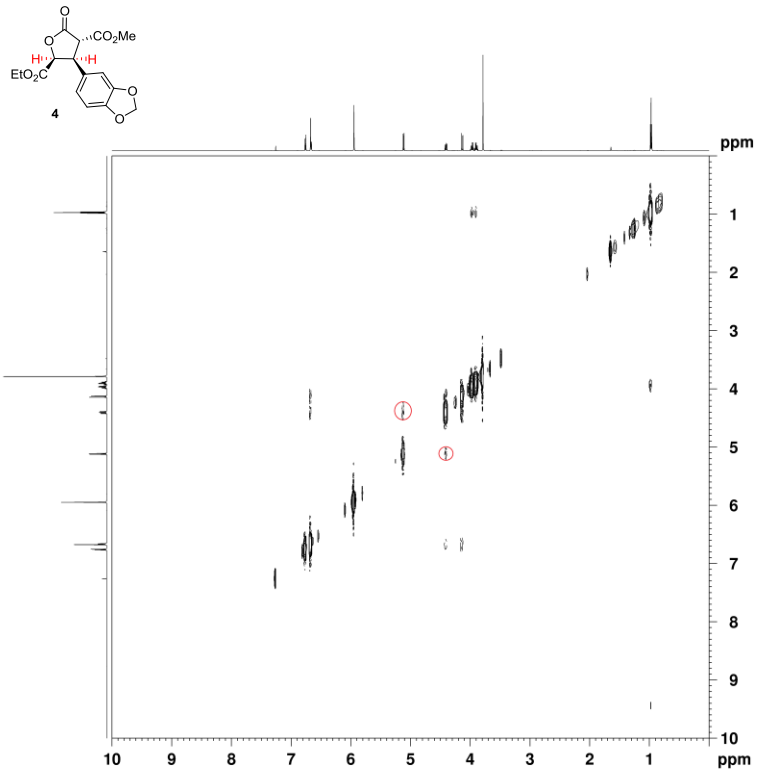


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SOLVENT              CDCl3
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DS                      32
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D1             2.00000000 sec
D8             0.30000001 sec
D11            0.03000000 sec
D12            0.00002000 sec
D16            0.00020000 sec
IN0            0.00016640 sec

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P17              2500.00 use
PLW1          9.00000000 W
PLW10         2.25000000 W

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FIDRES         46.950119 Hz
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FnMODE       States-TPPI

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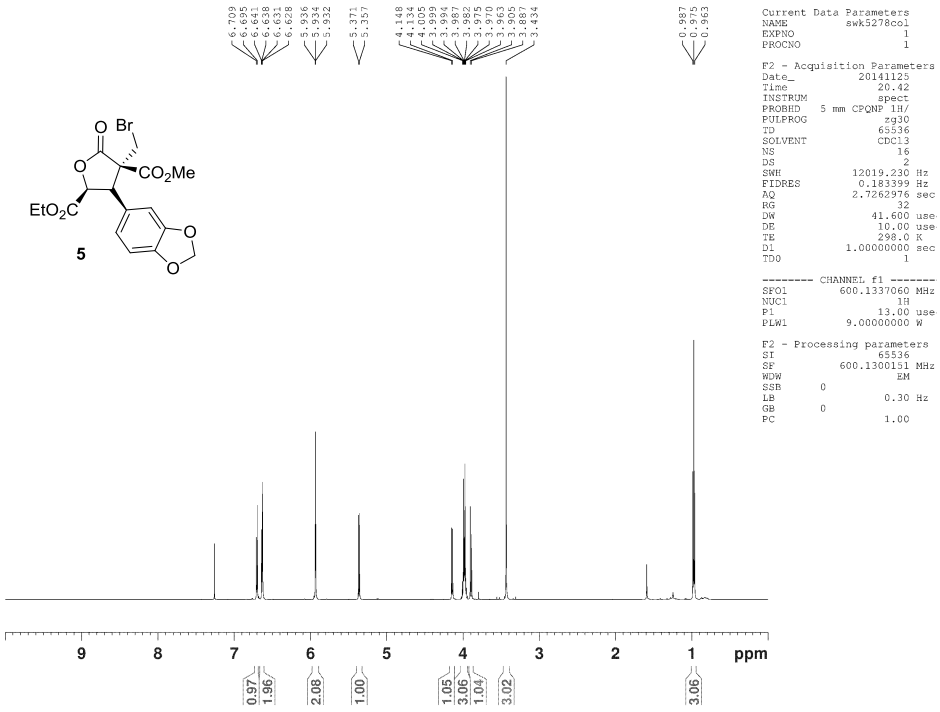


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PROCNO                         1

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SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                  12019.230 Hz
FIDRES                0.183399 Hz
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RG                          32
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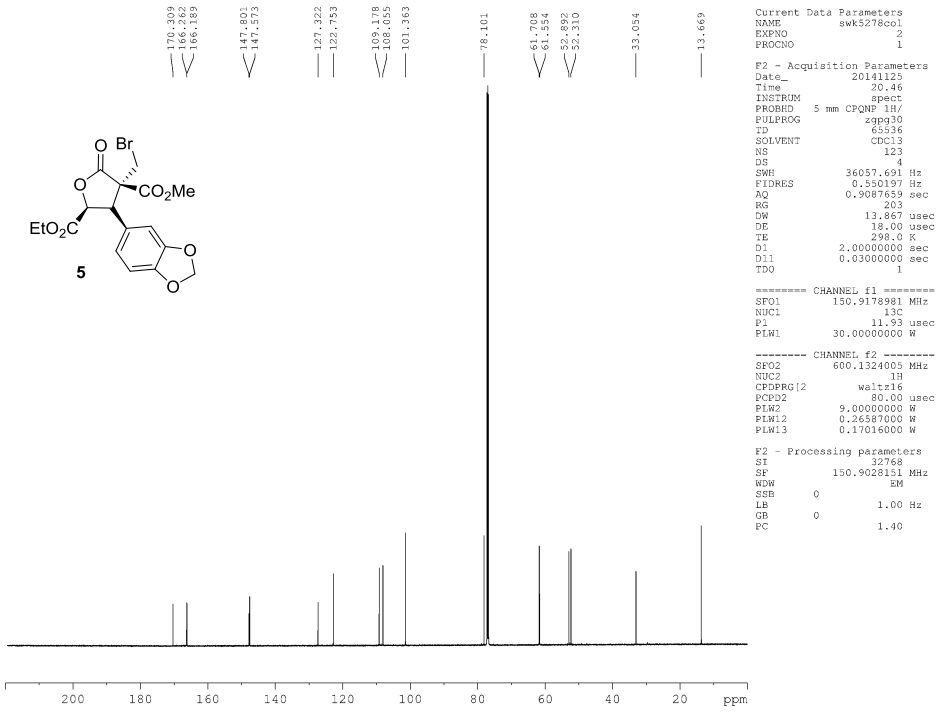


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SOLVENT                  CDCl3
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NUC1                        13C
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PLW1               30.00000000 W

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NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    80.00 usec
PLW2                9.00000000 W
PLW12               0.26587000 W
PLW13               0.17016000 W

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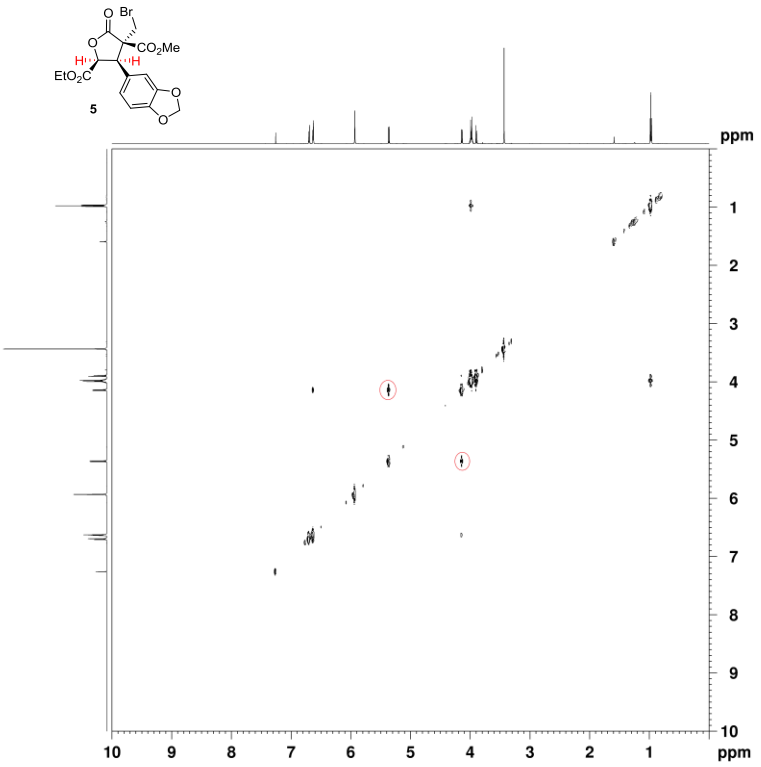


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SOLVENT                 CDCl3
NS                            4
DS                           32
SWH                  6009.615 Hz
FIDRES               2.934382 Hz
AQ                   0.1703936 sec
RG                          144
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D1                   2.00000000 sec
D8                   0.30000001 sec
D11                  0.03000000 sec
D12                  0.00002000 sec
D16                  0.00020000 sec
IN0                  0.00016640 sec

======== CHANNEL f1 =======
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NUC1                        1H
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PLW1                9.00000000 W
PLW10               2.25000000 W

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FnMODE           States-TPPI

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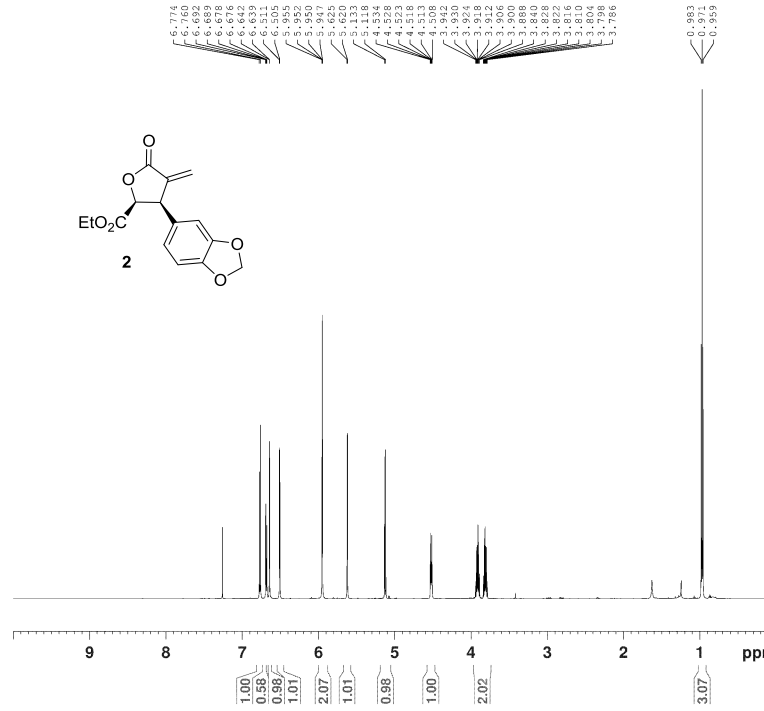


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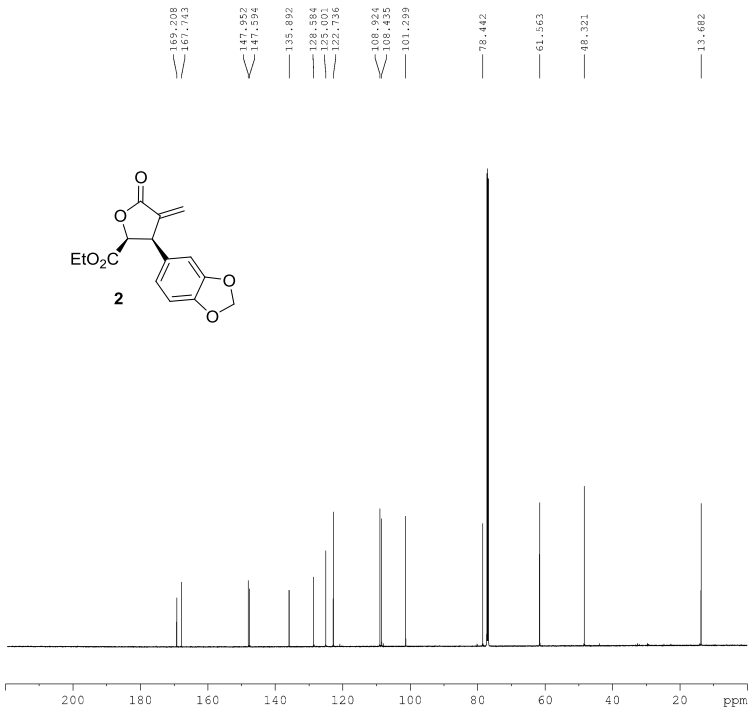


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TE                    298.0 K
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NUC1                      13C
P1                      11.93 usec
PLW1              30.00000000 W

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PCPD2                   80.00 usec
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PLW12              0.26587000 W
PLW13              0.17016000 W

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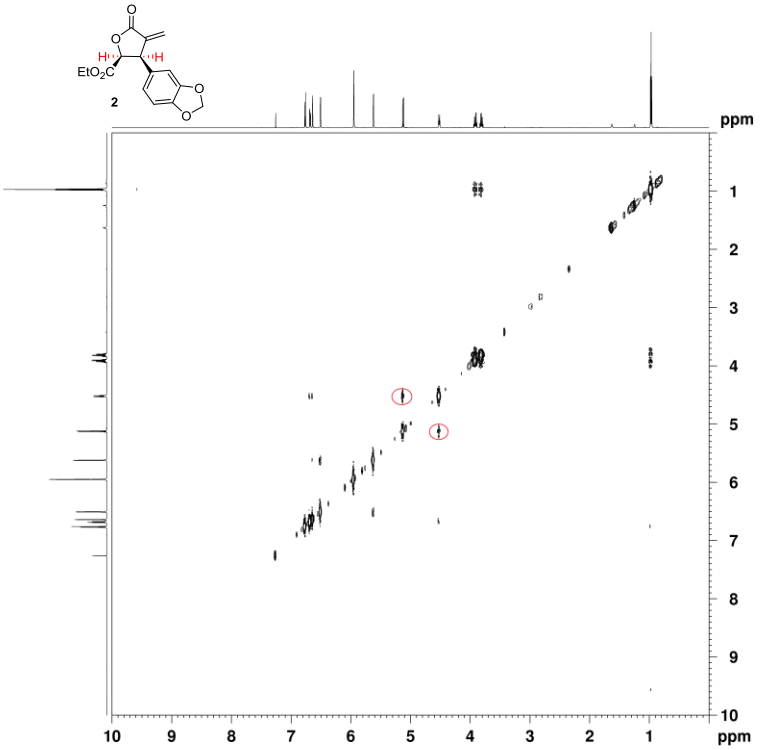


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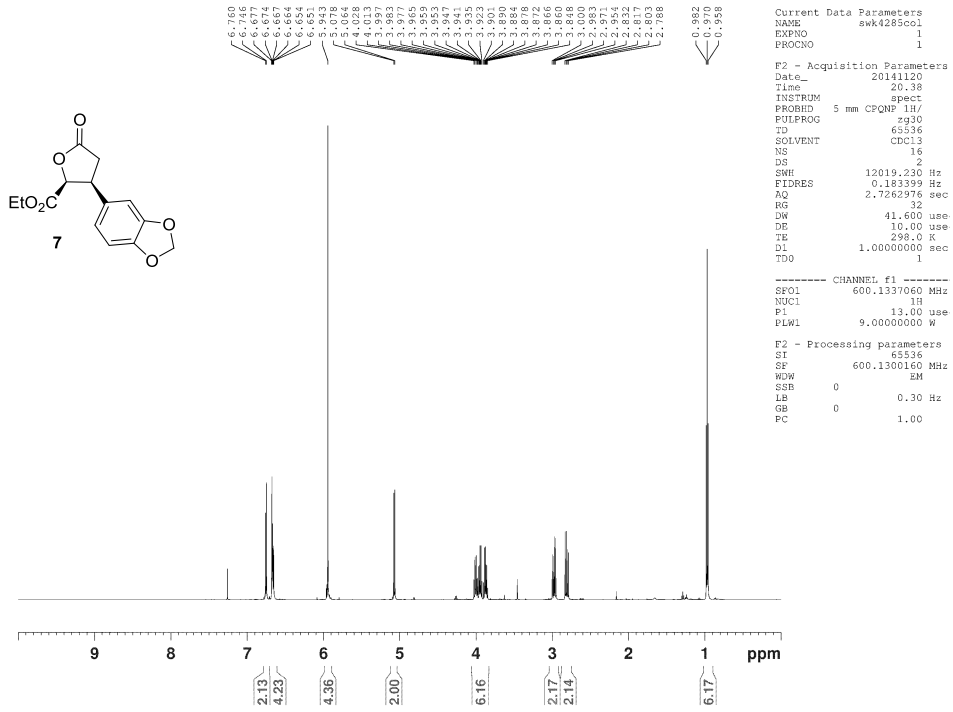


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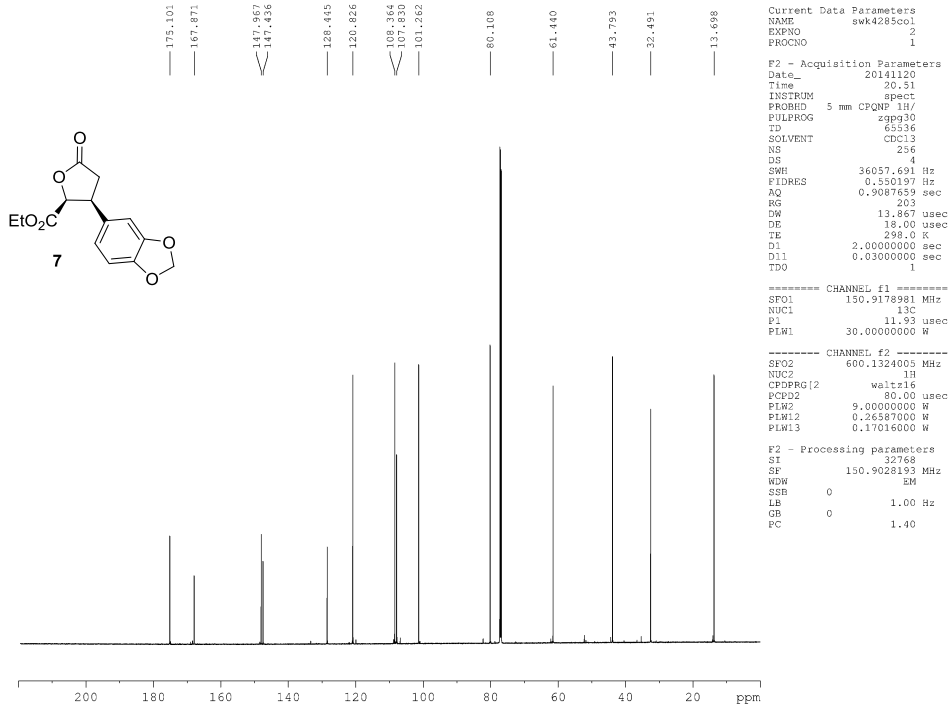


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PROCNO                        1

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FIDRES             0.550197 Hz
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RG                      203
DW                    13.867 usec
DE                    18.00 usec
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D1                 2.00000000 sec
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TD0                       1

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NUC1                      13C
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PLW1              30.00000000 W

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CPDPRG[2               waltz16
PCPD2                  80.00 usec
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PLW12              0.26587000 W
PLW13              0.17016000 W

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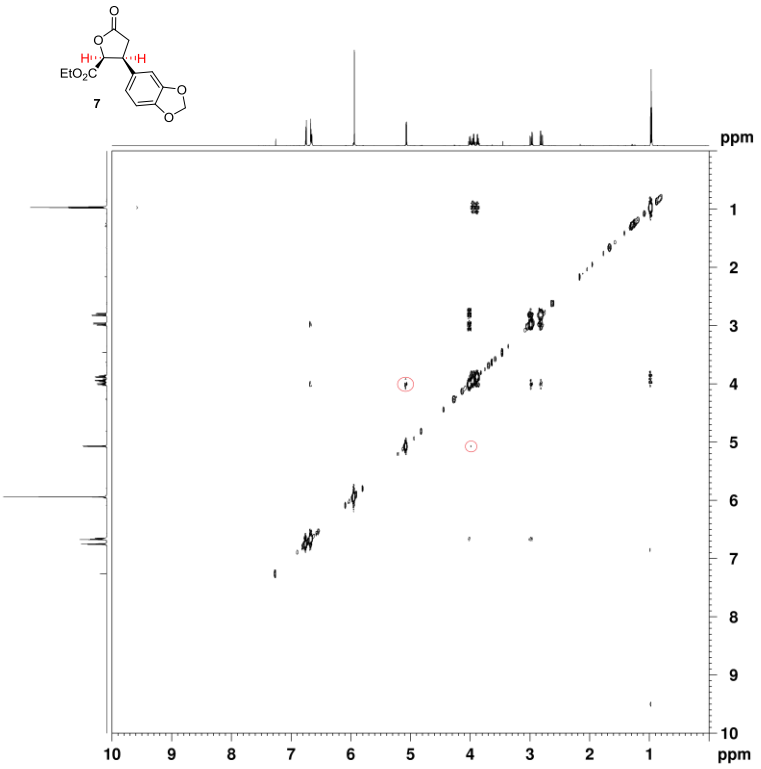


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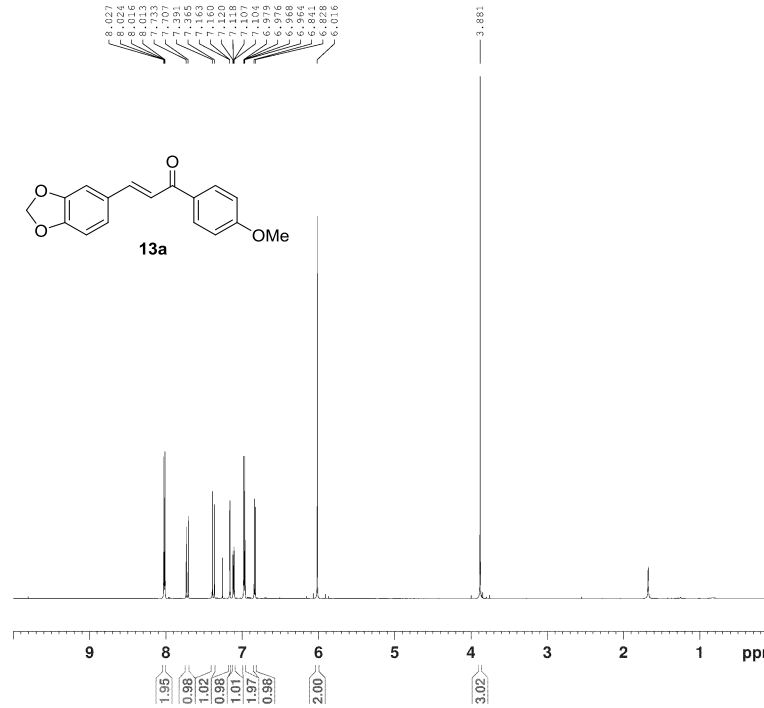


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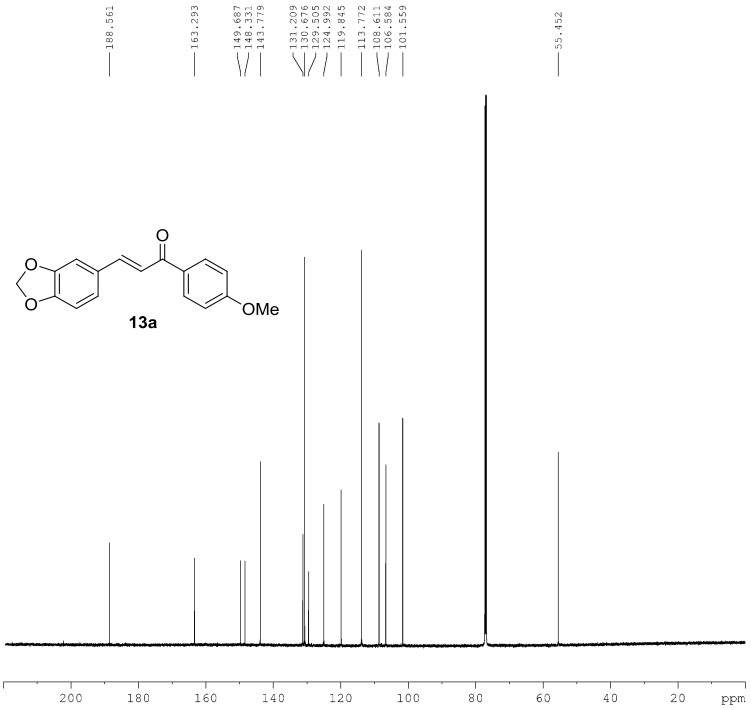
INSTRUM               spect
PROBHD    5 mm CPQCI 1H/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               12019.230 Hz
FIDRES             0.183399 Hz
AQ                 2.7262976 sec
RG                      12.7
DW                    41.600 use
DE                    10.00 use
TE                   298.0 K
D1                 1.00000000 sec
TD0                       1

======== CHANNEL f1 =======
SFO1              600.1337060 MHz
NUC1                      1H
P1                    10.35 use
PLW1              13.00000000 W

F2 - Processing parameters
SI                       65536
SF                600.1300151 MHz
WDW                      EM
SSB         0
LB                     0.30 Hz
GB          0
PC                      1.00
```



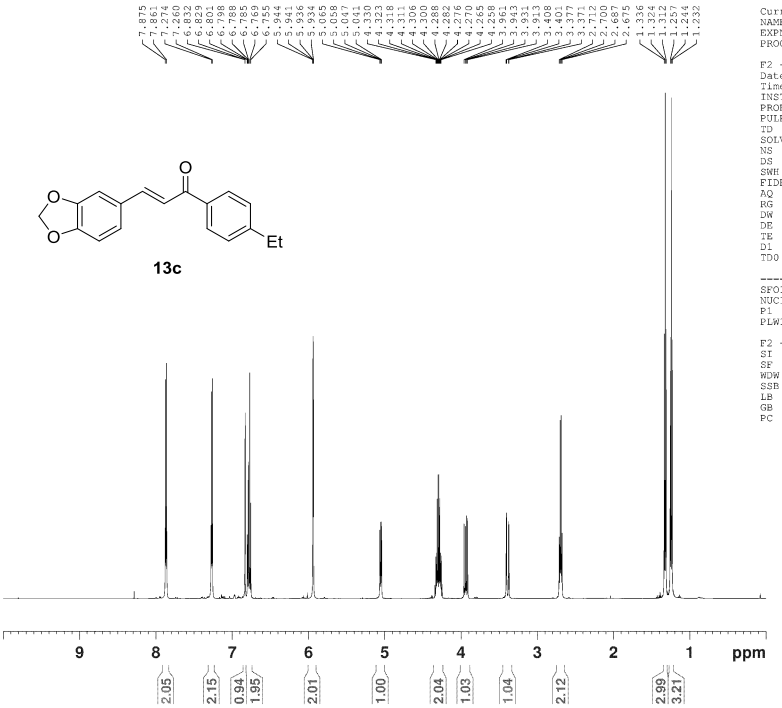
Current Data Parameters
NAME swk4288crude13C
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140928
Time 9.14
INSTRUM spect
PROBHD 5 mm CPQCI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 203
DW 13.867 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 11.35 usec
PLW1 230.00000000 W

-------- CHANNEL f2 --------
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.17192000 W
PLW13 0.13925999 W

F2 - Processing parameters
SI 32768
SF 150.9028163 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

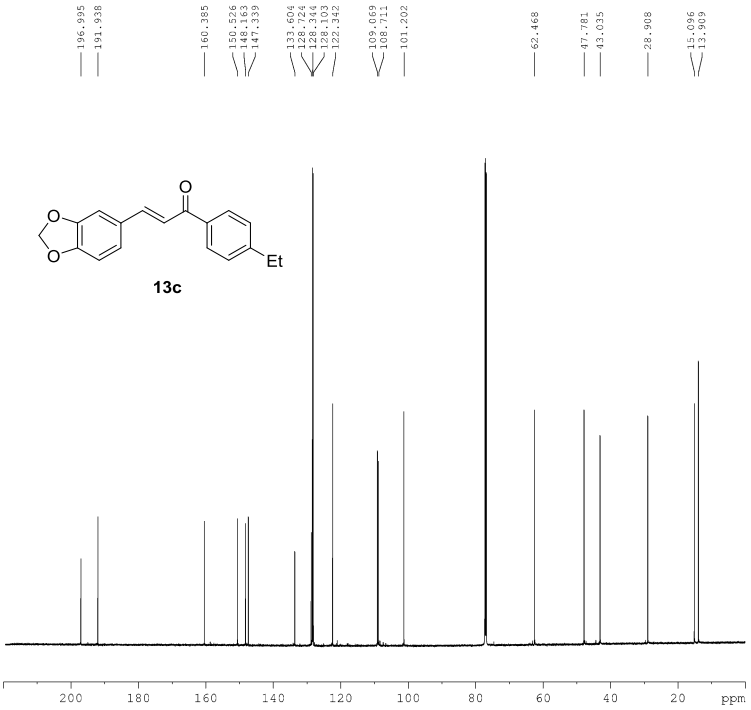


Current Data Parameters
NAME swk5107col
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140709
Time 22.07
INSTRUM spect
PROBHD 5 mm CPQCI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 12.7
DW 41.600 use
DE 10.00 use
TE 298.2 K
D1 1.00000000 sec
TD0 1

-------- CHANNEL f1 --------
SFO1 600.1337060 MHz
NUC1 1H
P1 12.10 use
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 600.1300139 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



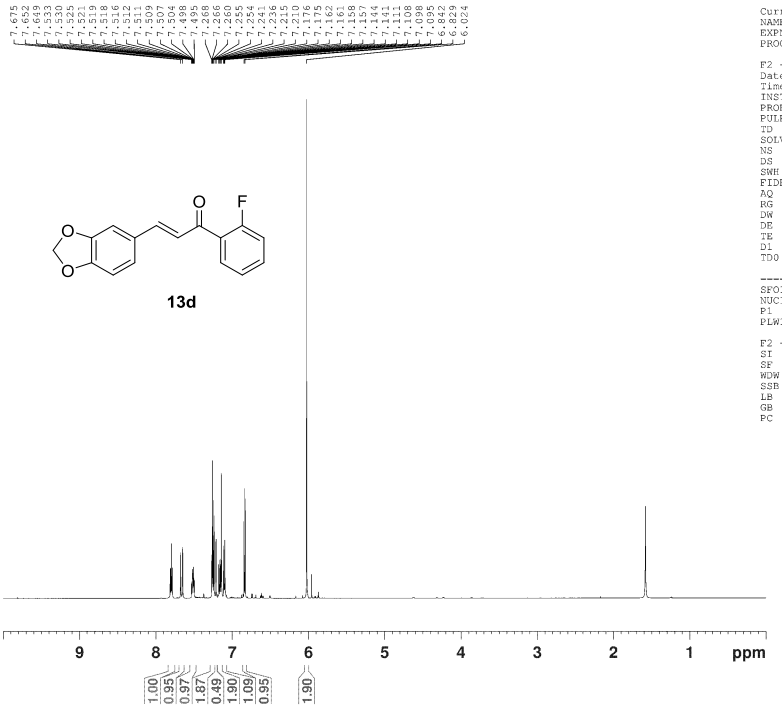

```
Current Data Parameters
NAME            swk5107co1
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_             20140710
Time                  2.46
INSTRUM              spect
PROBHD   5 mm CPQCI 1H/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     512
DS                       4
SWH            36057.691 Hz
FIDRES          0.550197 Hz
AQ              0.9087659 sec
RG                     203
DW                13.867 usec
DE                 18.00 usec
TE                 298.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          150.9178981 MHz
NUC1                   13C
P1                   11.35 usec
PLW1        230.00000000 W

-------- CHANNEL f2 --------
SFO2          600.1324005 MHz
NUC2                   1H
CPDPRG[2            waltz16
PCPD2                90.00 usec
PLW2          13.00000000 W
PLW12          0.23498000 W
PLW13          0.19033000 W

F2 - Processing parameters
SI                   32768
SF            150.9028195 MHz
WDW                     EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

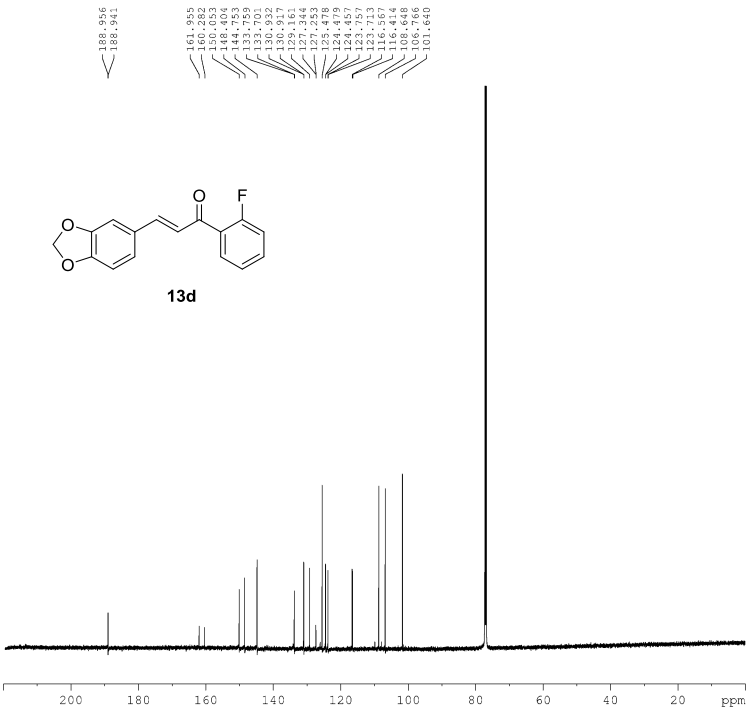



```
Current Data Parameters
NAME            swk5149crude
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_             20140709
Time                 21.37
INSTRUM              spect
PROBHD   5 mm CPQCI 1H/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       0
SWH            12019.230 Hz
FIDRES          0.183399 Hz
AQ              2.7262976 sec
RG                      18
DW                41.600 use
DE                10.00 use
TE                298.1 K
D1             1.00000000 sec
TD0                      1

-------- CHANNEL f1 --------
SFO1          600.1337060 MHz
NUC1                   1H
P1                  12.10 use
PLW1        13.00000000 W

F2 - Processing parameters
SI                   65536
SF            600.1300140 MHz
WDW                     EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```



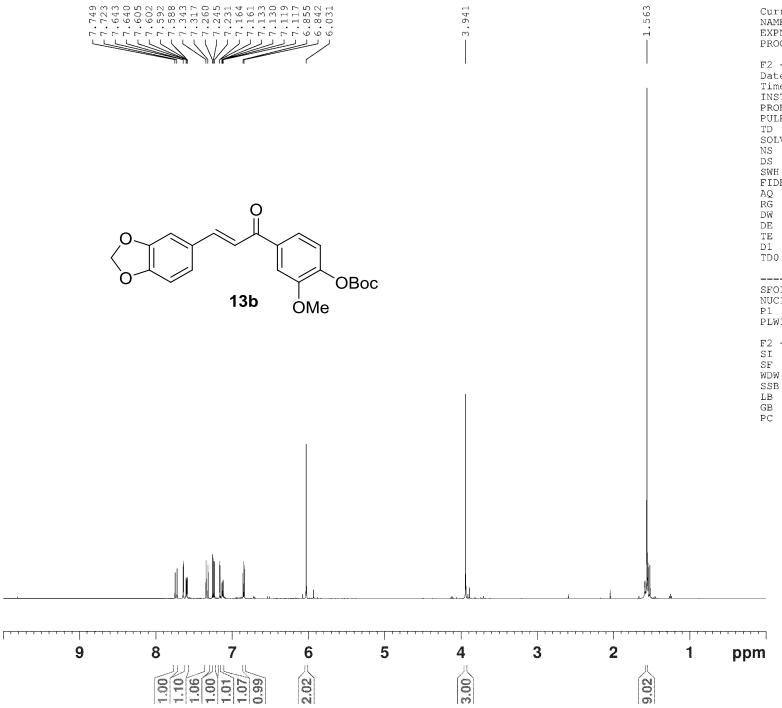

```
Current Data Parameters
NAME        swk5149crude
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140709
Time               23.46
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ             0.9087659 sec
RG                  203
DW               13.867 usec
DE                18.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        150.9178981 MHz
NUC1                  13C
P1                 11.35 usec
PLW1       230.00000000 W

-------- CHANNEL f2 --------
SFO2        600.1324005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2        13.00000000 W
PLW12        0.23498000 W
PLW13        0.19033000 W

F2 - Processing parameters
SI                 32768
SF          150.9028129 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

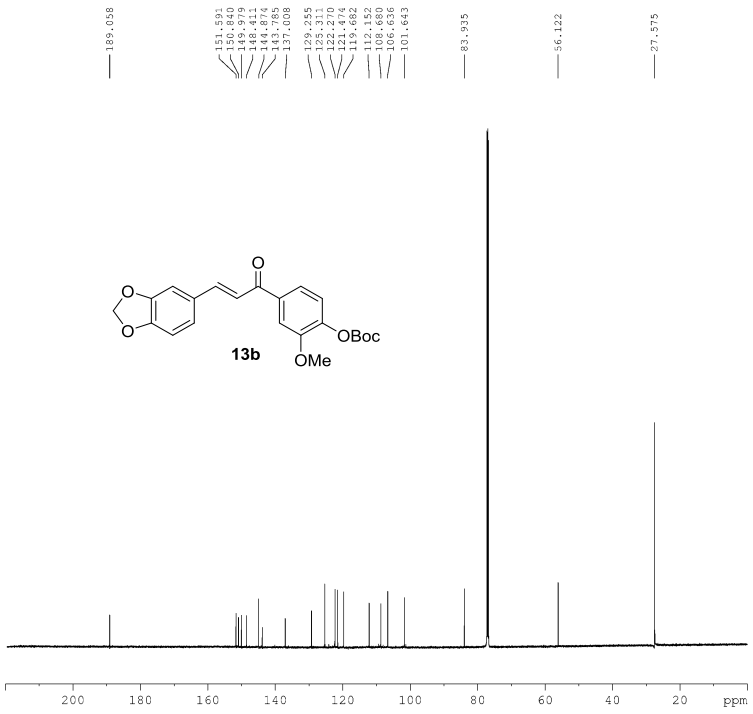



```
Current Data Parameters
NAME        swk5143crude
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140709
Time               21.44
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG              zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           12019.230 Hz
FIDRES         0.183399 Hz
AQ             2.7262976 sec
RG                   16
DW               41.600 use
DE                10.00 use
TE                298.2 K
D1            1.00000000 sec
TD0                    1

-------- CHANNEL f1 --------
SFO1        600.1337060 MHz
NUC1                  1H
P1                12.10 use
PLW1       13.00000000 W

F2 - Processing parameters
SI                 65536
SF          600.1300139 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



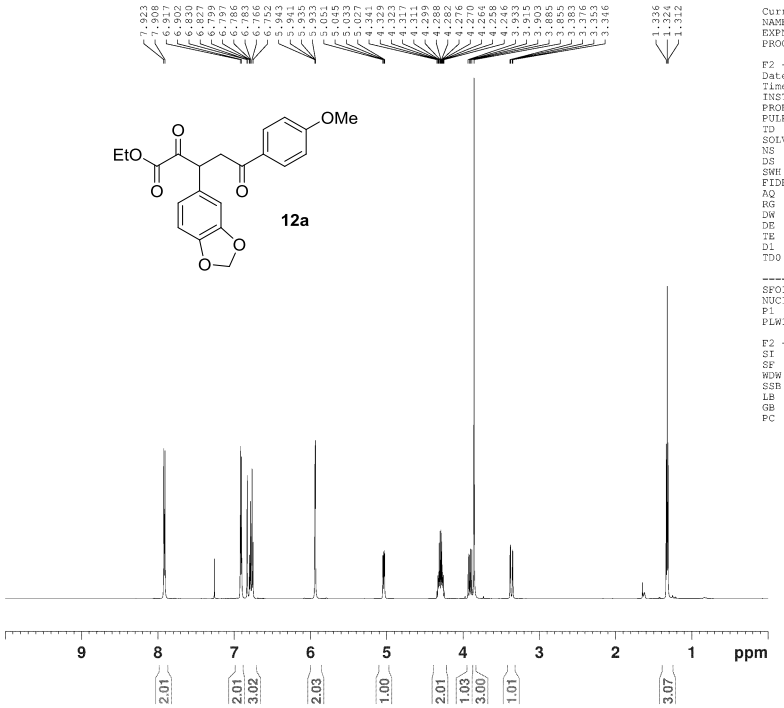

```
Current Data Parameters
NAME        swk5143crude
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140710
Time                0.13
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9087659 sec
RG                  203
DW               13.867 usec
DE                18.00 usec
TE                298.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        150.9178981 MHz
NUC1               13C
P1               11.35 usec
PLW1       230.00000000 W

-------- CHANNEL f2 --------
SFO2        600.1324005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        13.00000000 W
PLW12        0.23498000 W
PLW13        0.19033000 W

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

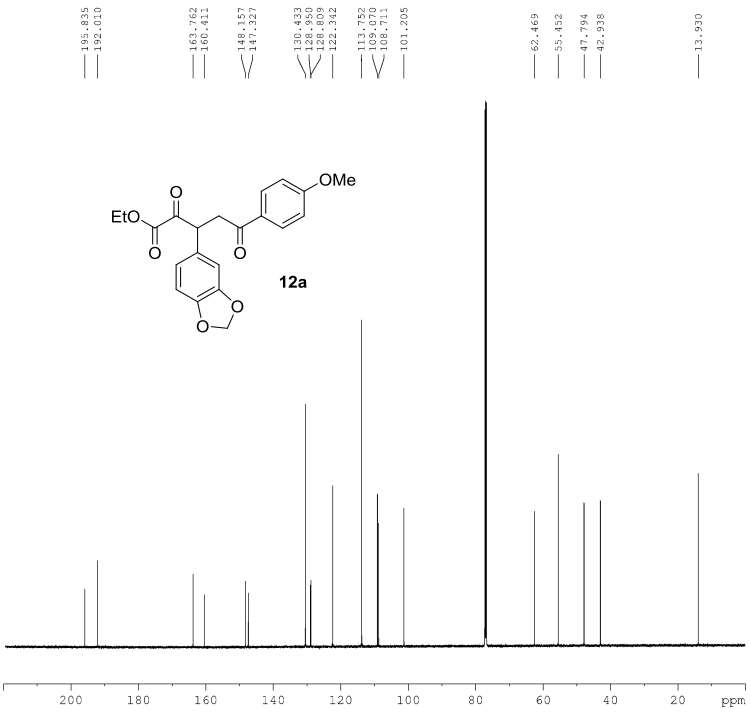



```
Current Data Parameters
NAME      swk5081fr40-42
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20141108
Time                8.29
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                    6
DS                    0
SWH           12019.230 Hz
FIDRES         0.183399 Hz
AQ            2.7262976 sec
RG                   32
DW               41.600 usec
DE                10.00 usec
TE                298.0 K
D1           1.00000000 sec
TD0                    1

-------- CHANNEL f1 --------
SFO1        600.1337060 MHz
NUC1               1H
P1               13.00 usec
PLW1        9.00000000 W

F2 - Processing parameters
SI                 65536
SF          600.1300141 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



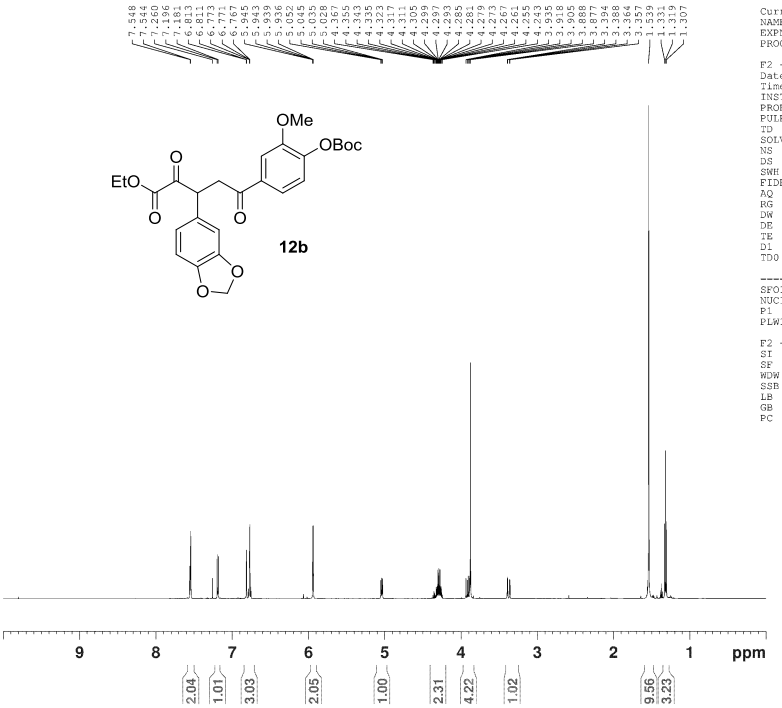

```
Current Data Parameters
NAME       swk5081fr40-42
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_               20141108
Time                    8.31
INSTRUM                spect
PROBHD   5 mm CPQNP 1H/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      36
DS                       0
SWH              36057.691 Hz
FIDRES            0.550197 Hz
AQ                0.9087659 sec
RG                     203
DW                  13.867 usec
DE                  18.00 usec
TE                  298.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          150.9178981 MHz
NUC1                    13C
P1                   11.93 usec
PLW1         30.00000000 W

-------- CHANNEL f2 --------
SFO2          600.1324005 MHz
NUC2                    1H
CPDPRG[2             waltz16
PCPD2                80.00 usec
PLW2          9.00000000 W
PLW12         0.26587000 W
PLW13         0.17016000 W

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

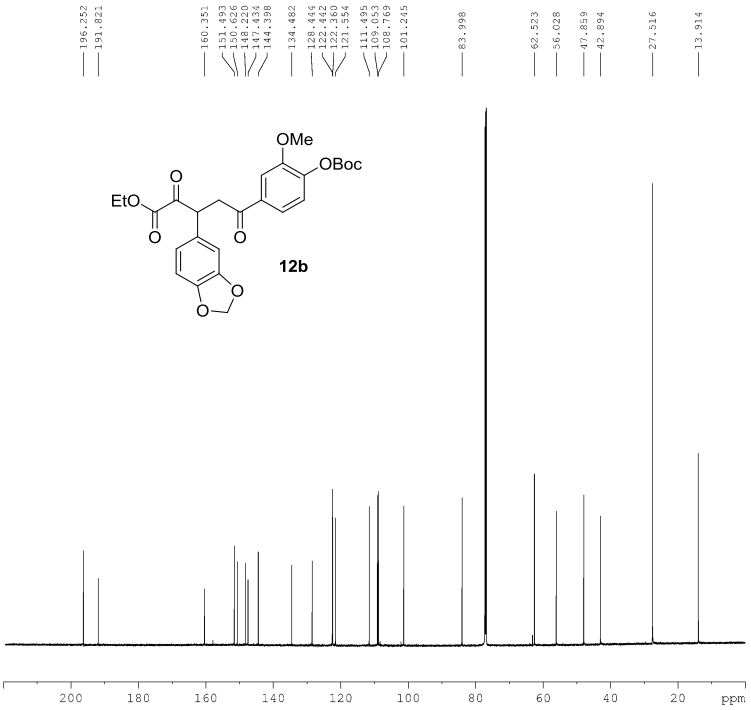



```
Current Data Parameters
NAME        swk5193crude
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_               20140925
Time                   19.08
INSTRUM                spect
PROBHD   5 mm CPQCI 1H/
PULPROG                 zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH              12019.230 Hz
FIDRES            0.183399 Hz
AQ                2.7262976 sec
RG                    22.6
DW                  41.600 use
DE                  10.00 use
TE                  298.0 K
D1               1.00000000 sec
TD0                     1

-------- CHANNEL f1 --------
SFO1          600.1337060 MHz
NUC1                    1H
P1                  10.35 use
PLW1         13.00000000 W

F2 - Processing parameters
SI                   65536
SF            600.1300151 MHz
WDW                     EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```



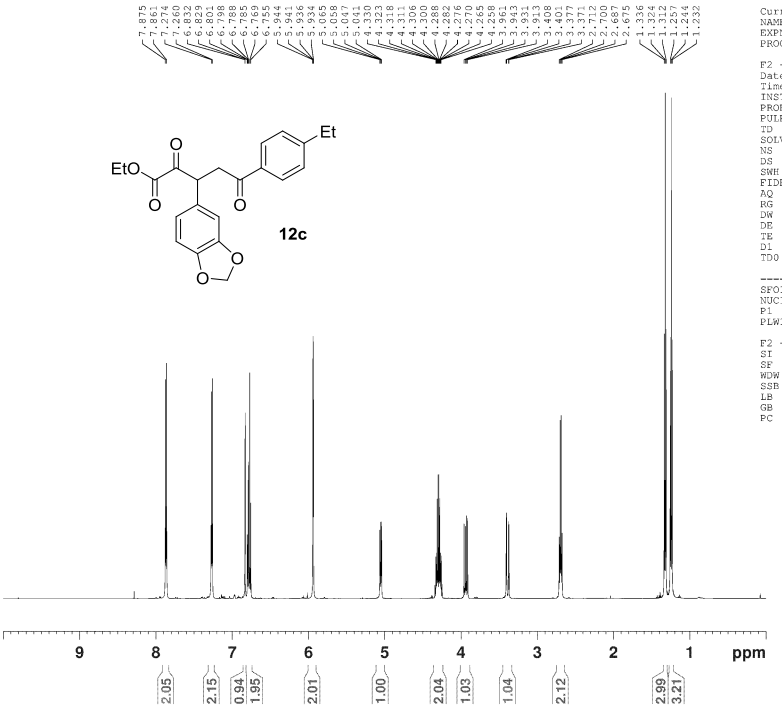

```
Current Data Parameters
NAME          swk5193crude
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_              20140925
Time                  19.33
INSTRUM               spect
PROBHD   5 mm CPQCI 1H/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      512
DS                        4
SWH               36057.691 Hz
FIDRES             0.550197 Hz
AQ                 0.9087659 sec
RG                      203
DW                   13.867 usec
DE                   18.00 usec
TE                   298.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1          150.9178981 MHz
NUC1                    13C
P1                   11.35 usec
PLW1         230.00000000 W

-------- CHANNEL f2 --------
SFO2          600.1324005 MHz
NUC2                    1H
CPDPRG[2             waltz16
PCPD2                90.00 usec
PLW2         13.00000000 W
PLW12         0.17192000 W
PLW13         0.13925999 W

F2 - Processing parameters
SI                    32768
SF          150.9028177 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

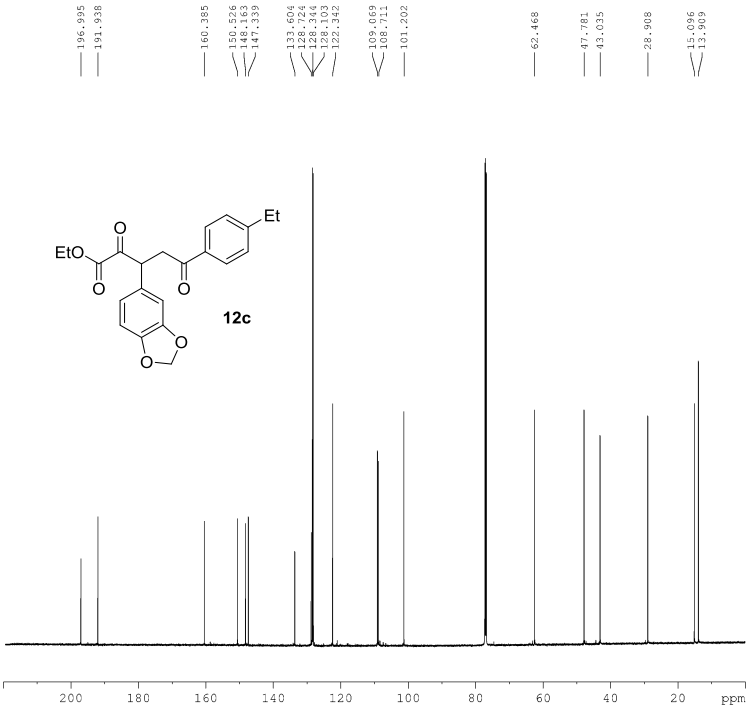



```
Current Data Parameters
NAME          swk5107col
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20140709
Time                  22.07
INSTRUM               spect
PROBHD   5 mm CPQCI 1H/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               12019.230 Hz
FIDRES             0.183399 Hz
AQ                 2.7262976 sec
RG                     12.7
DW                   41.600 use
DE                   10.00 use
TE                   298.2 K
D1                1.00000000 sec
TD0                       1

-------- CHANNEL f1 --------
SFO1          600.1337060 MHz
NUC1                    1H
P1                   12.10 use
PLW1         13.00000000 W

F2 - Processing parameters
SI                    65536
SF          600.1300139 MHz
WDW                      EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```



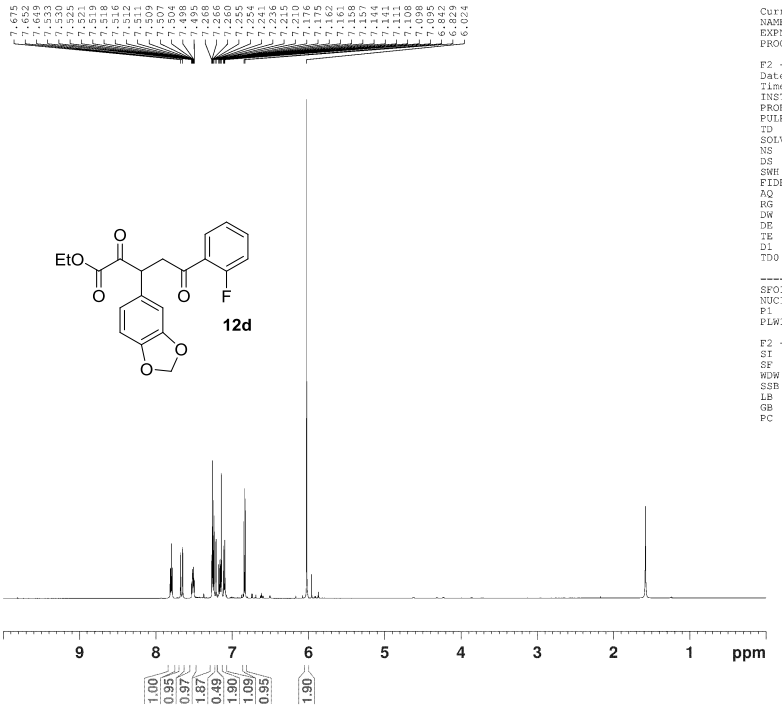

```
Current Data Parameters
NAME            swk5107co1
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_             20140710
Time                  2.46
INSTRUM              spect
PROBHD   5 mm CPQCI 1H/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     512
DS                       4
SWH            36057.691 Hz
FIDRES          0.550197 Hz
AQ              0.9087659 sec
RG                     203
DW                13.867 usec
DE                 18.00 usec
TE                 298.1 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          150.9178981 MHz
NUC1                   13C
P1                  11.35 usec
PLW1        230.00000000 W

-------- CHANNEL f2 --------
SFO2          600.1324005 MHz
NUC2                   1H
CPDPRG[2            waltz16
PCPD2               90.00 usec
PLW2          13.00000000 W
PLW12          0.23498000 W
PLW13          0.19033000 W

F2 - Processing parameters
SI                   32768
SF            150.9028195 MHz
WDW                     EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```

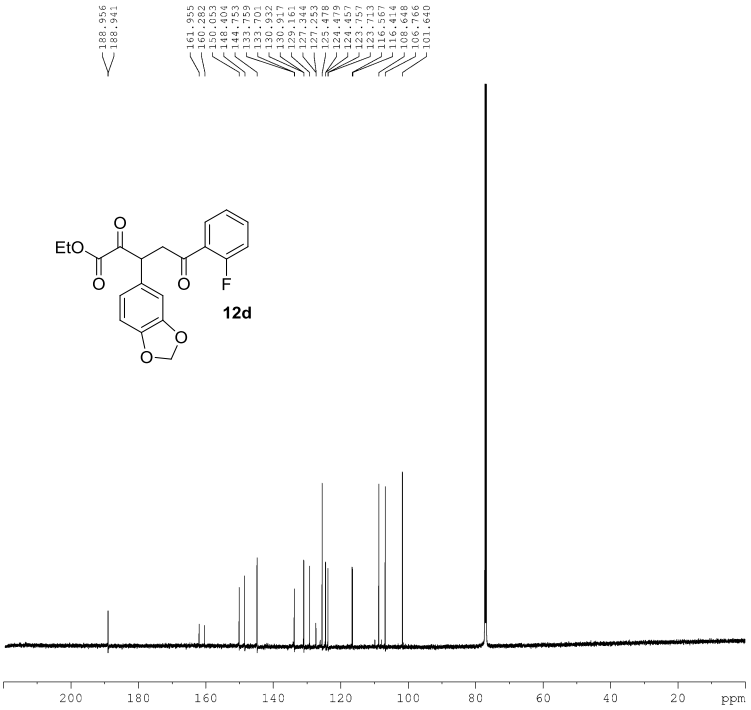



```
Current Data Parameters
NAME            swk5149crude
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_             20140709
Time                 21.37
INSTRUM              spect
PROBHD   5 mm CPQCI 1H/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       0
SWH            12019.230 Hz
FIDRES          0.183399 Hz
AQ              2.7262976 sec
RG                      18
DW                41.600 use
DE                10.00 use
TE                298.1 K
D1              1.00000000 sec
TD0                      1

-------- CHANNEL f1 --------
SFO1          600.1337060 MHz
NUC1                   1H
P1                 12.10 use
PLW1        13.00000000 W

F2 - Processing parameters
SI                   65536
SF            600.1300140 MHz
WDW                     EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```



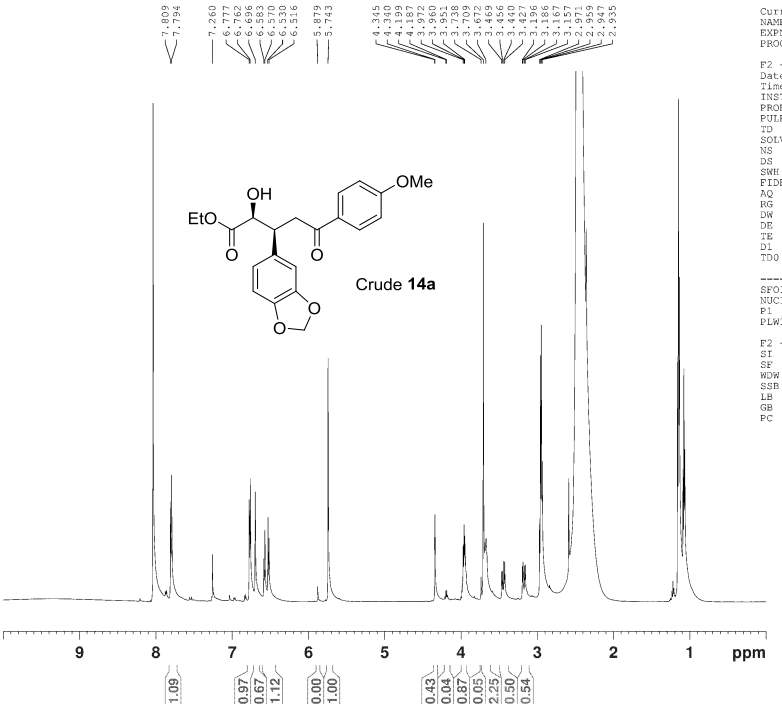

```
Current Data Parameters
NAME        swk5149crude
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140709
Time               23.46
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ             0.9087659 sec
RG                  203
DW               13.867 usec
DE                18.00 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        150.9178981 MHz
NUC1                  13C
P1                11.35 usec
PLW1       230.00000000 W

-------- CHANNEL f2 --------
SFO2        600.1324005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2        13.00000000 W
PLW12        0.23498000 W
PLW13        0.19033000 W

F2 - Processing parameters
SI                32768
SF          150.9028129 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

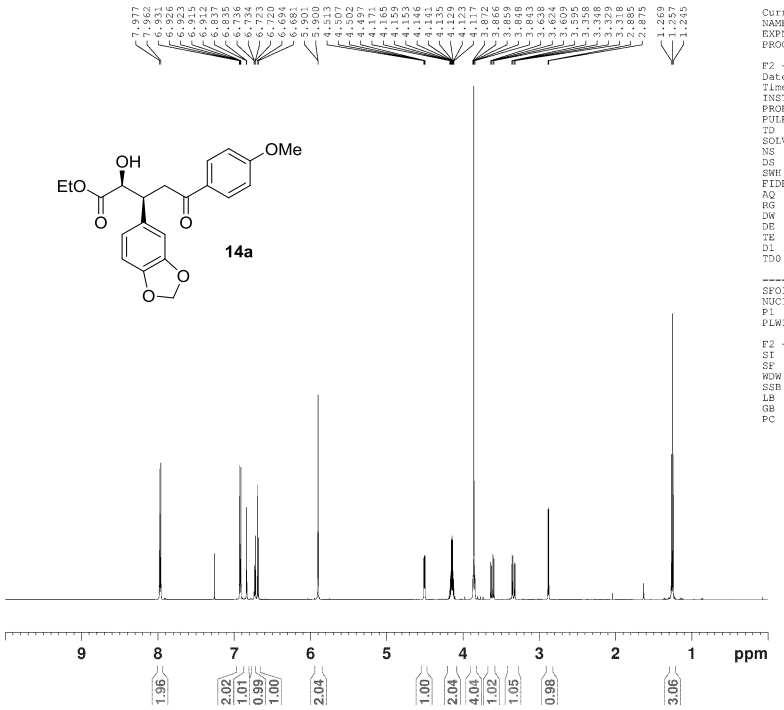



```
Current Data Parameters
NAME       swk5155fol22g
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140708
Time               10.54
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                    9
DS                    0
SWH           12019.230 Hz
FIDRES         0.183399 Hz
AQ             2.7262976 sec
RG                    9
DW               41.600 use
DE                10.00 use
TE                298.1 K
D1            1.00000000 sec
TD0                   1

-------- CHANNEL f1 --------
SFO1        600.1337060 MHz
NUC1                  1H
P1                12.10 use
PLW1       13.00000000 W

F2 - Processing parameters
SI                65536
SF          600.1300143 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

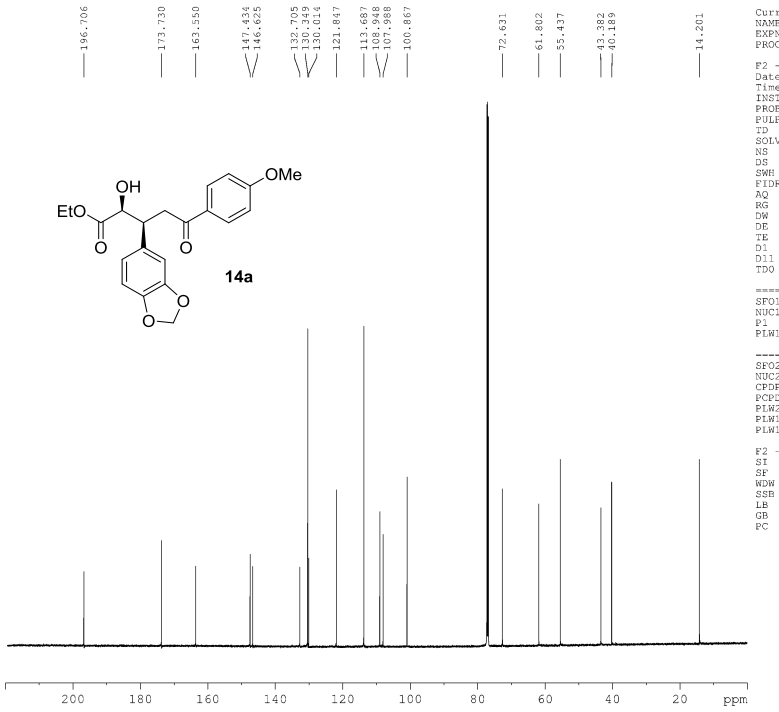



```
Current Data Parameters
NAME                 swk5155col
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_               20140709
Time                   22.19
INSTRUM                spect
PROBHD    5 mm CPQCI 1H/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                12019.230 Hz
FIDRES                0.183399 Hz
AQ                 2.7262976 sec
RG                       14.2
DW                    41.600 use
DE                    10.00 use
TE                    298.1 K
D1                 1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1              600.1337060 MHz
NUC1                        1H
P1                       12.10 use
PLW1              13.00000000 W

F2 - Processing parameters
SI                        65536
SF                600.1300139 MHz
WDW                         EM
SSB          0
LB                     0.30 Hz
GB           0
PC                        1.00
```



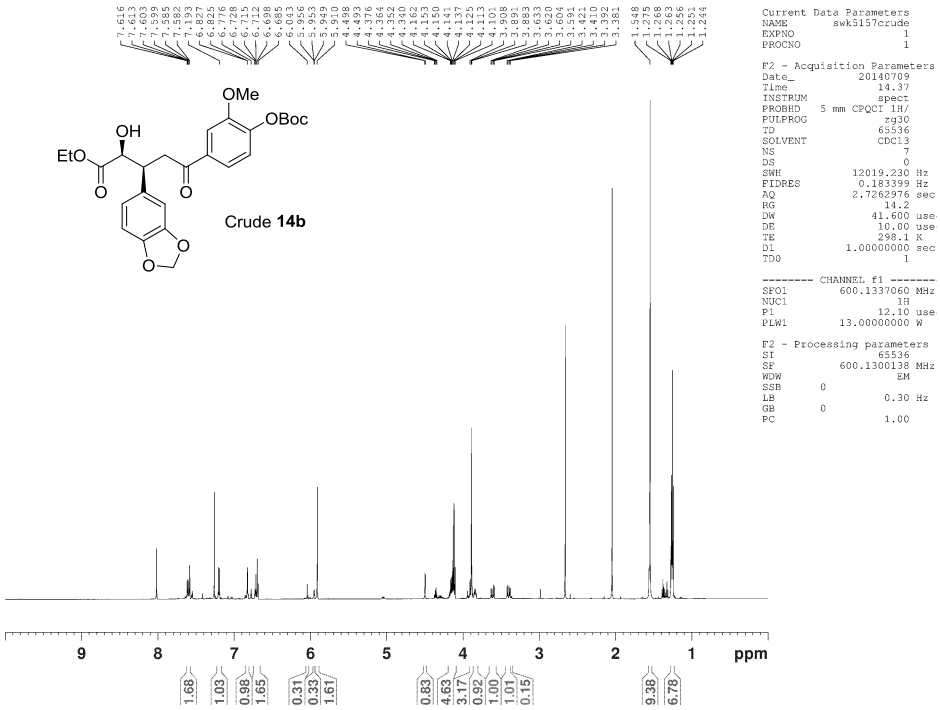

```
Current Data Parameters
NAME                 swk5155col
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_               20140710
Time                    3.13
INSTRUM                spect
PROBHD    5 mm CPQCI 1H/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      512
DS                        4
SWH                36057.691 Hz
FIDRES                0.550197 Hz
AQ                 0.9087659 sec
RG                      203
DW                   13.867 usec
DE                    18.00 usec
TE                    298.1 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1              150.9178981 MHz
NUC1                       13C
P1                       11.35 usec
PLW1          230.00000000 W

======== CHANNEL f2 ========
SFO2              600.1324005 MHz
NUC2                        1H
CPDPRG[2             waltz16
PCPD2                 90.00 usec
PLW2              13.00000000 W
PLW12              0.23498000 W
PLW13              0.19033000 W

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

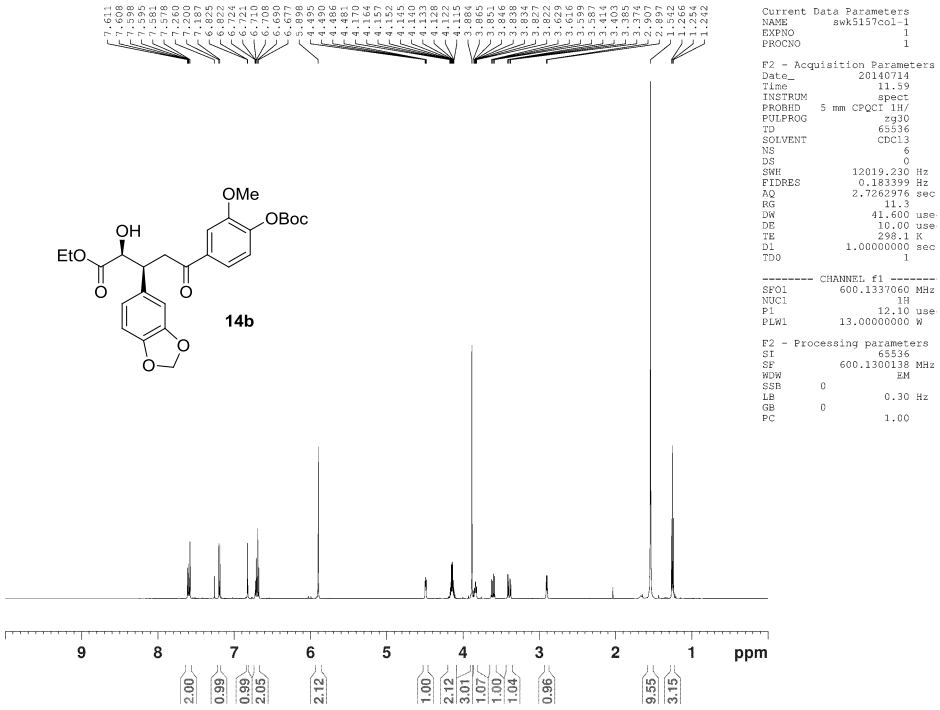



```
Current Data Parameters
NAME         swk5157crude
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_              20140709
Time                 14.37
INSTRUM              spect
PROBHD    5 mm CPQCI 1H/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      7
DS                      0
SWH             12019.230 Hz
FIDRES              0.183399 Hz
AQ                2.7262976 sec
RG                   14.2
DW                  41.600 use
DE                  10.00 use
TE                  298.1 K
D1              1.00000000 sec
TD0                     1

-------- CHANNEL f1 --------
SFO1            600.1337060 MHz
NUC1                    1H
P1                   12.10 use
PLW1           13.00000000 W

F2 - Processing parameters
SI                  65536
SF              600.1300138 MHz
WDW                    EM
SSB         0
LB                   0.30 Hz
GB          0
PC                   1.00
```

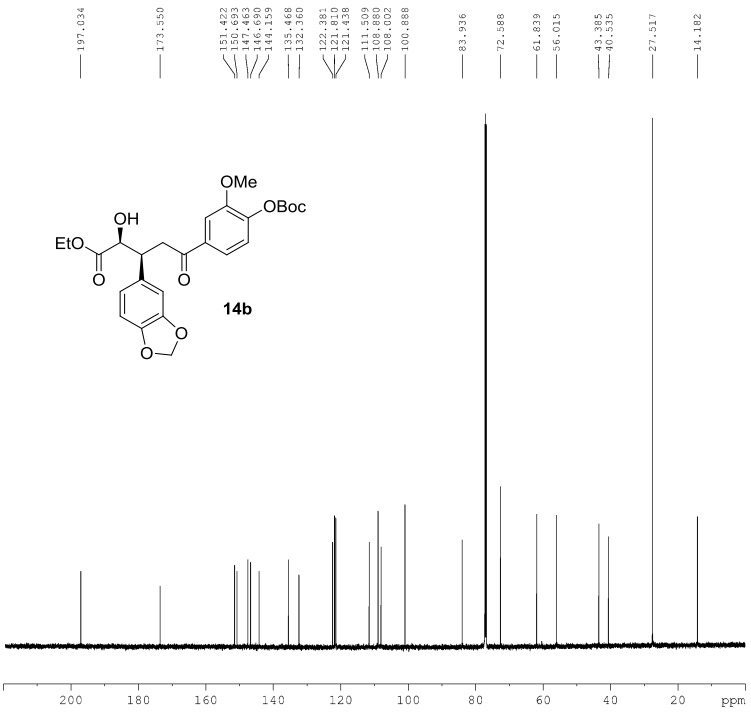



```
Current Data Parameters
NAME         swk5157col-1
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_              20140714
Time                 11.59
INSTRUM              spect
PROBHD    5 mm CPQCI 1H/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      6
DS                      0
SWH             12019.230 Hz
FIDRES              0.183399 Hz
AQ                2.7262976 sec
RG                   11.3
DW                  41.600 use
DE                  10.00 use
TE                  298.1 K
D1              1.00000000 sec
TD0                     1

-------- CHANNEL f1 --------
SFO1            600.1337060 MHz
NUC1                    1H
P1                   12.10 use
PLW1           13.00000000 W

F2 - Processing parameters
SI                  65536
SF              600.1300138 MHz
WDW                    EM
SSB         0
LB                   0.30 Hz
GB          0
PC                   1.00
```



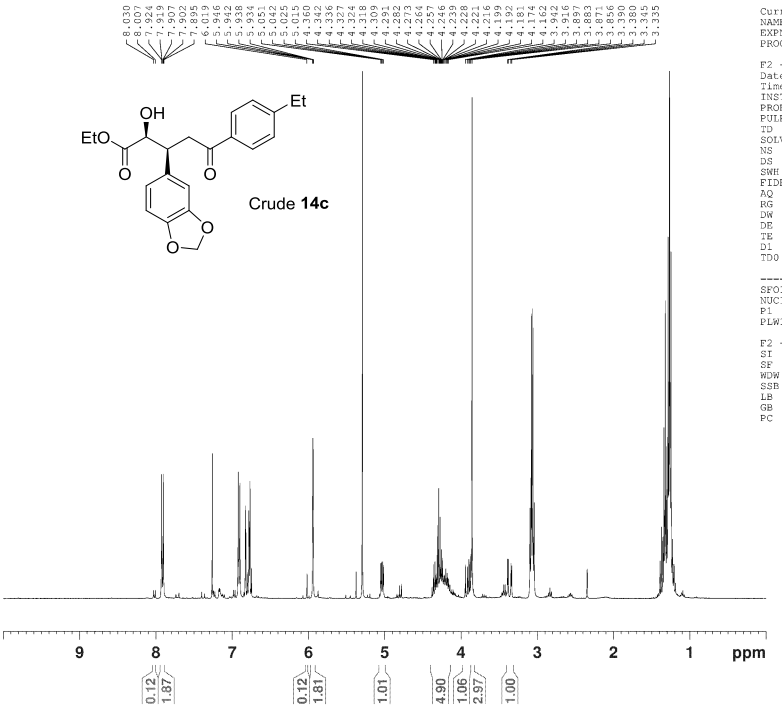

```
Current Data Parameters
NAME        swk5157col-1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140714
Time               12.02
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   42
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9087659 sec
RG                  203
DW               13.867 usec
DE               18.00 usec
TE               298.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        150.9178981 MHz
NUC1               13C
P1               11.35 usec
PLW1       230.00000000 W

-------- CHANNEL f2 --------
SFO2        600.1324005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        13.00000000 W
PLW12        0.23498000 W
PLW13        0.19033000 W

F2 - Processing parameters
SI                 32768
SF          150.9028184 MHz
WDW                   EM
SSB       0
LB               1.00 Hz
GB        0
PC               1.40
```

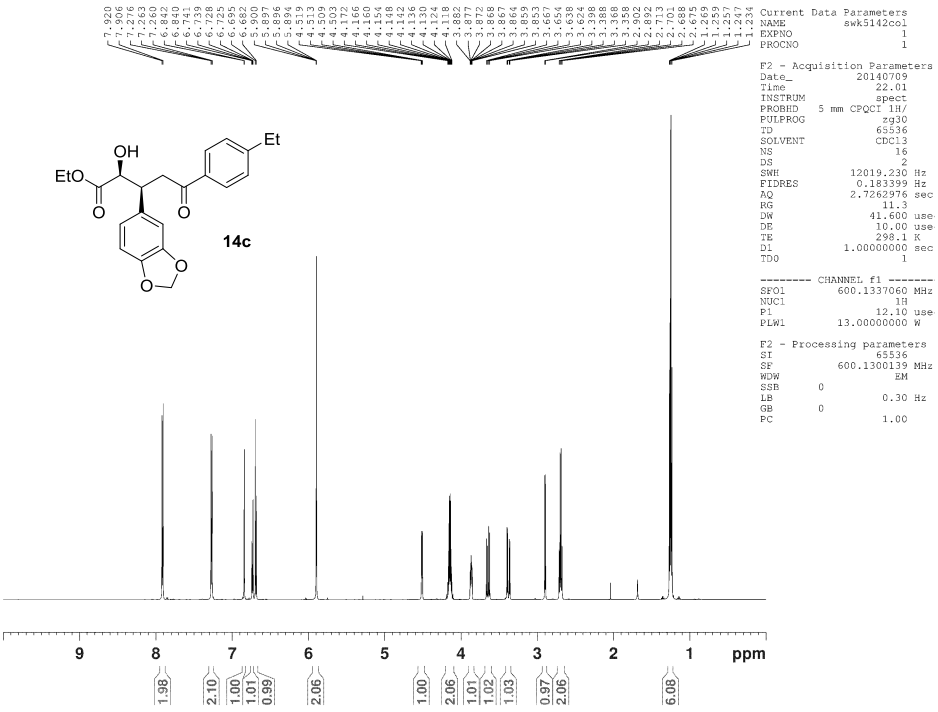


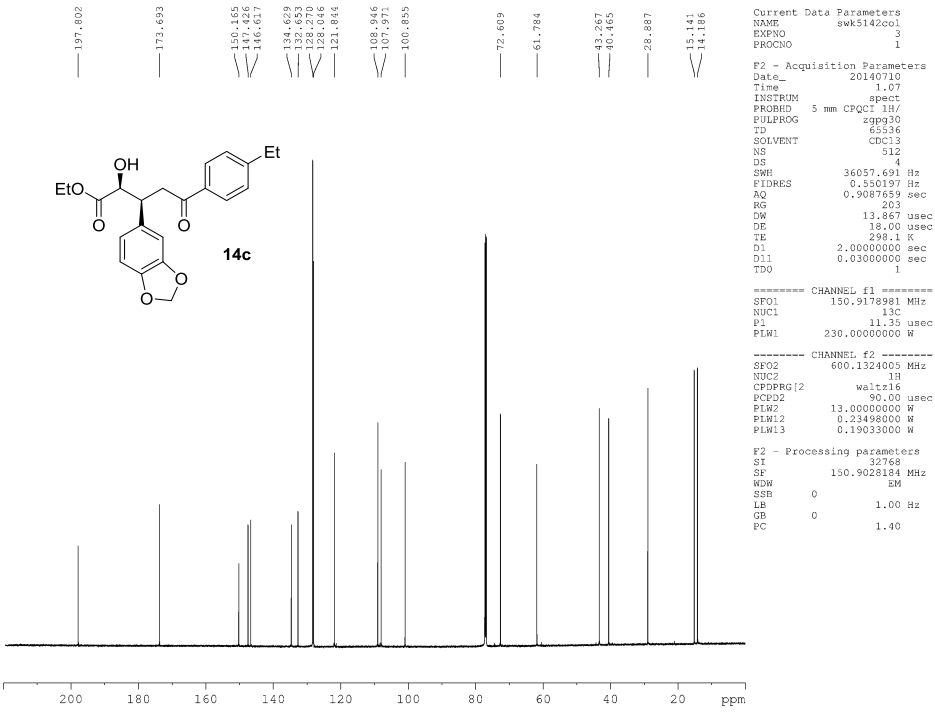

```
Current Data Parameters
NAME       swk5142fol42h
EXPNO                  1
PROCNO                 1

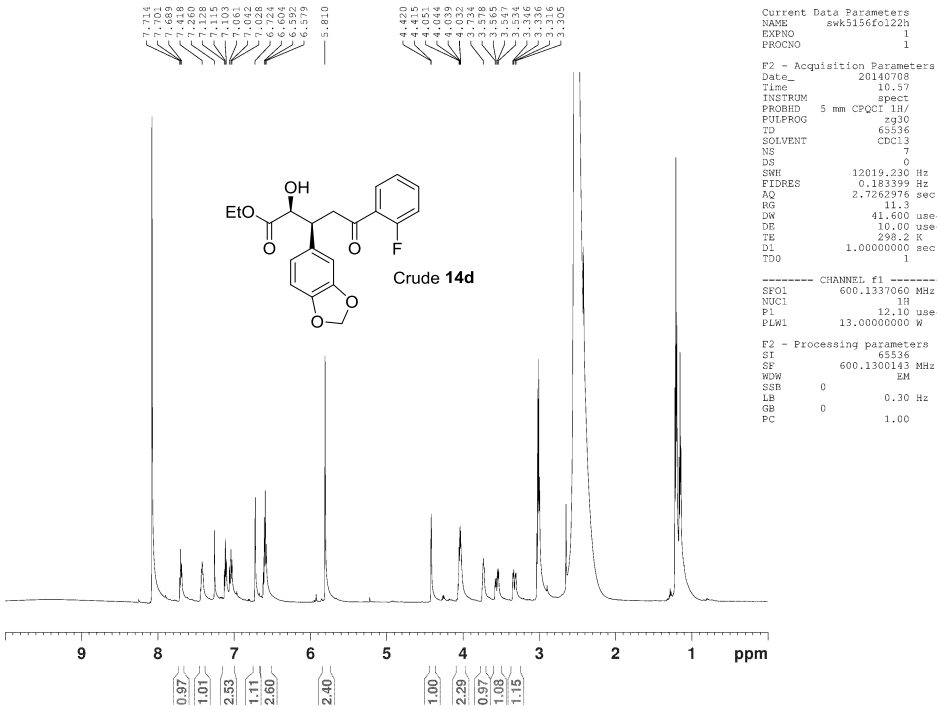
F2 - Acquisition Parameters
Date_           20140704
Time               8.46
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                    5
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                120.26
DW                62.400 use
DE                 6.50 use
TE                295.7 K
D1             1.00000000 sec
TD0                    1

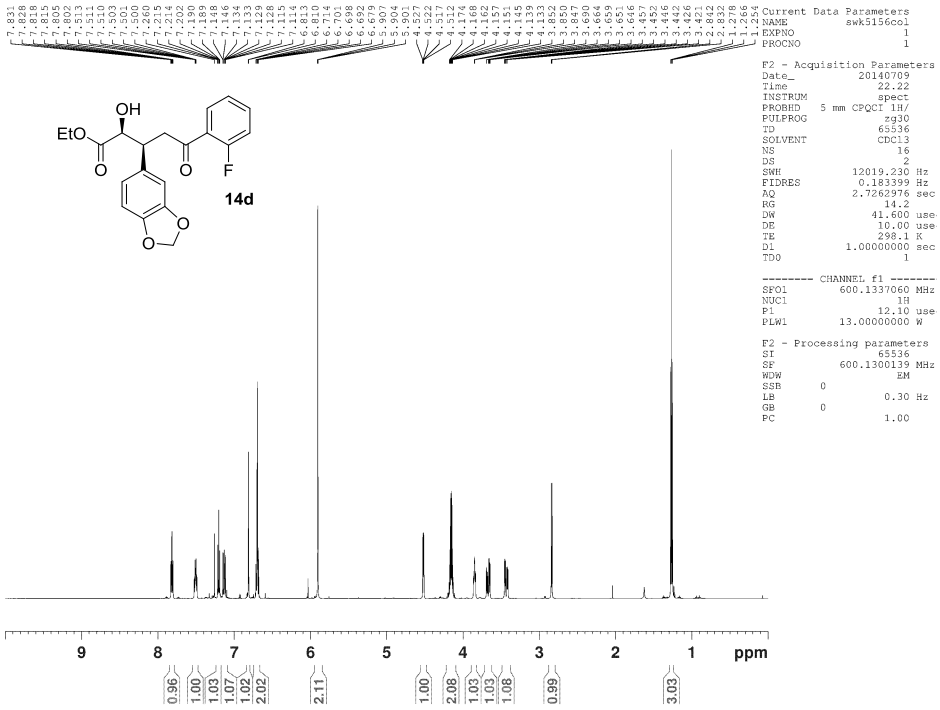
-------- CHANNEL f1 --------
SFO1        399.9824700 MHz
NUC1               1H
P1               12.38 use
PLW1       11.19999981 W

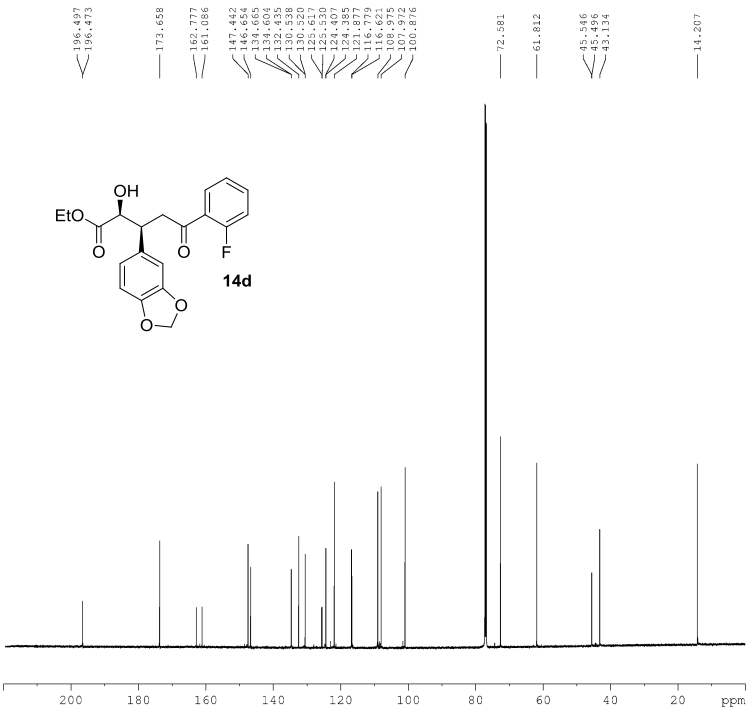
F2 - Processing parameters
SI                 65536
SF          399.9800093 MHz
WDW                   EM
SSB       0
LB               0.30 Hz
GB        0
PC               1.00
```


7.920
7.906
7.276
7.263
7.260
6.842
6.840
6.741
6.739
6.728
6.725
6.685
6.682
5.900
5.897
5.896
5.894
4.519
4.513
4.509
4.503
4.172
4.166
4.160
4.154
4.148
4.142
4.136
4.130
4.124
4.118
3.882
3.877
3.872
3.868
3.867
3.866
3.859
3.853
3.667
3.654
3.648
3.624
3.398
3.388
3.368
3.358
2.902
2.892
2.713
2.701
2.688
2.675
1.269
1.259
1.257
1.247
1.234
OH
EtO
O
O
Et
O
O
O
14c
Current Data Parameters
NAME swk5142co1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140709
Time 22.01
INSTRUM spect
PROBHD 5 mm CPQCI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 11.3
DW 41.600 use
DE 10.00 use
TE 298.1 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 600.1337060 MHz
NUC1 1H
P1 12.10 use
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 600.1300139 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
1.98
2.10
1.00
1.01
0.99
2.06
1.00
2.06
1.01
1.02
1.03
0.97
2.06
6.08


197.802
173.693
150.165
147.426
146.617
134.629
132.653
128.270
128.046
121.844
108.946
107.971
100.855
72.609
61.784
43.267
40.465
28.887
15.141
14.186
OH
EtO
O
O
Et
O
O
O
14c
Current Data Parameters
NAME swk5142co1
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140710
Time 1.07
INSTRUM spect
PROBHD 5 mm CPQCI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 203
DW 13.867 usec
DE 18.00 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
SFO1 150.9178981 MHz
NUC1 13C
P1 11.35 usec
PLW1 230.00000000 W
CHANNEL f2
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.23498000 W
PLW13 0.19033000 W
F2 - Processing parameters
SI 32768
SF 150.9028184 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
ppm


7.714
7.701
7.689
7.618
7.260
7.128
7.115
7.103
7.061
7.052
7.028
6.724
6.604
6.592
6.579
5.810
4.420
4.415
4.051
4.044
4.039
4.032
3.734
3.578
3.565
3.547
3.534
3.346
3.336
3.316
3.305
OH
EtO
O
O
O
F
O
O
Crude 14d
Current Data Parameters
NAME swk5156fol22h
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140708
Time 10.57
INSTRUM spect
PROBHD 5 mm CPQCI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 7
DS 0
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 11.3
DW 41.600 use
DE 10.00 use
TE 298.2 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
SFO1 600.1337060 MHz
NUC1 1H
P1 12.10 use
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 600.1300143 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
0.97
1.01
2.53
1.11
2.60
2.40
1.00
2.29
0.97
1.08
1.15


7.831
7.828
7.818
7.815
7.805
7.802
7.513
7.511
7.510
7.503
7.501
7.500
7.260
7.215
7.214
7.202
7.190
7.189
7.148
7.146
7.134
7.133
7.129
7.128
7.115
7.114
6.813
6.810
6.714
6.701
6.698
6.682
6.679
5.907
5.904
5.901
4.527
4.522
4.517
4.512
4.174
4.168
4.162
4.157
4.151
4.145
4.139
4.133
3.852
3.850
3.847
3.840
3.664
3.659
3.651
3.646
3.457
3.452
3.446
3.442
3.426
3.421
2.842
2.832
1.278
1.266
1.254
OH
EtO
O
O
O
F
O
O
14d
Current Data Parameters
NAME swk5156col
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140709
Time 22.22
INSTRUM spect
PROBHD 5 mm CPQCI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 14.2
DW 41.600 use
DE 10.00 use
TE 298.1 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
SFO1 600.1337060 MHz
NUC1 1H
P1 12.10 use
PLW1 13.00000000 W
F2 - Processing parameters
SI 65536
SF 600.1300139 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm
0.96
1.00
1.03
1.07
1.02
2.02
2.11
1.00
2.08
1.03
1.03
1.08
0.99
3.03



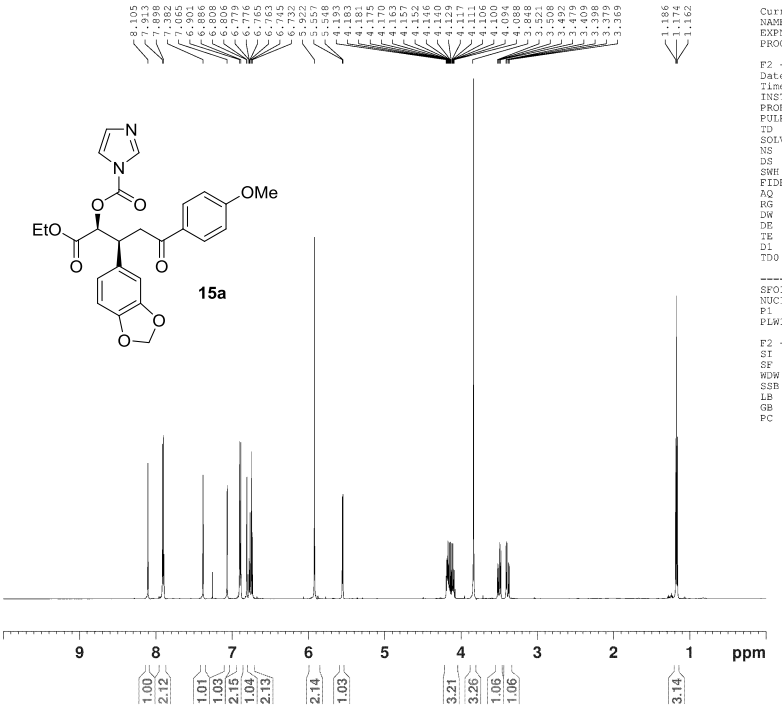

```
Current Data Parameters
NAME                 swk5156col
EXPNO                         3
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20140710
Time                       3.41
INSTRUM                   spect
PROBHD    5 mm CPQCI 1H/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        512
DS                          4
SWH                36057.691 Hz
FIDRES              0.550197 Hz
AQ                  0.9087659 sec
RG                        203
DW                     13.867 usec
DE                     18.00 usec
TE                     298.1 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1            150.9178981 MHz
NUC1                      13C
P1                     11.35 usec
PLW1           230.00000000 W

-------- CHANNEL f2 --------
SFO2            600.1324005 MHz
NUC2                      1H
CPDPRG[2               waltz16
PCPD2                  90.00 usec
PLW2           13.00000000 W
PLW12           0.23498000 W
PLW13           0.19033000 W

F2 - Processing parameters
SI                        32768
SF              150.9028161 MHz
WDW                          EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```

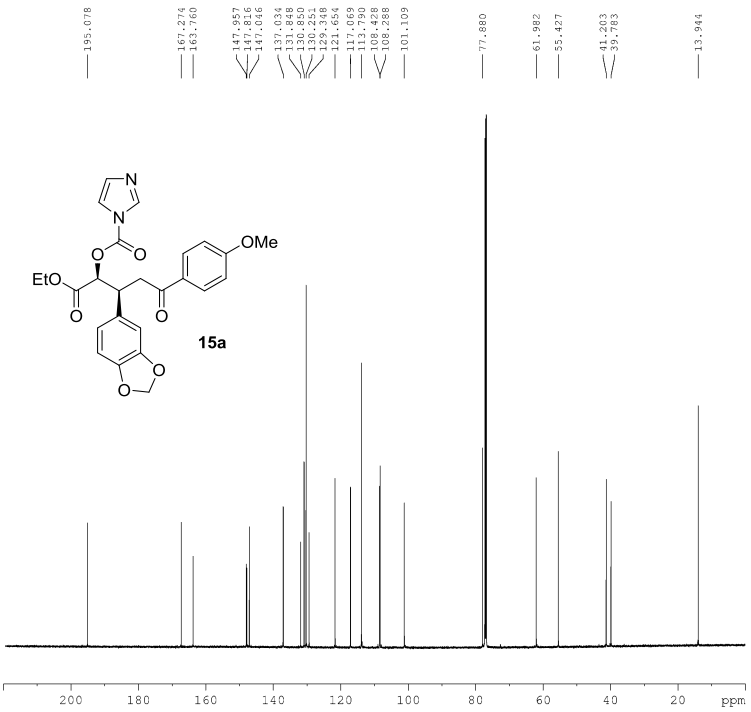



```
Current Data Parameters
NAME                 swk5175colH
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20140928
Time                       9.33
INSTRUM                   spect
PROBHD    5 mm CPQCI 1H/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          2
SWH                12019.230 Hz
FIDRES              0.183399 Hz
AQ                  2.7262976 sec
RG                       20.2
DW                     41.600 use
DE                     10.00 use
TE                     298.0 K
D1                 1.00000000 sec
TD0                           1

-------- CHANNEL f1 --------
SFO1            600.1337060 MHz
NUC1                      1H
P1                     10.35 use
PLW1           13.00000000 W

F2 - Processing parameters
SI                        65536
SF              600.1300150 MHz
WDW                          EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```



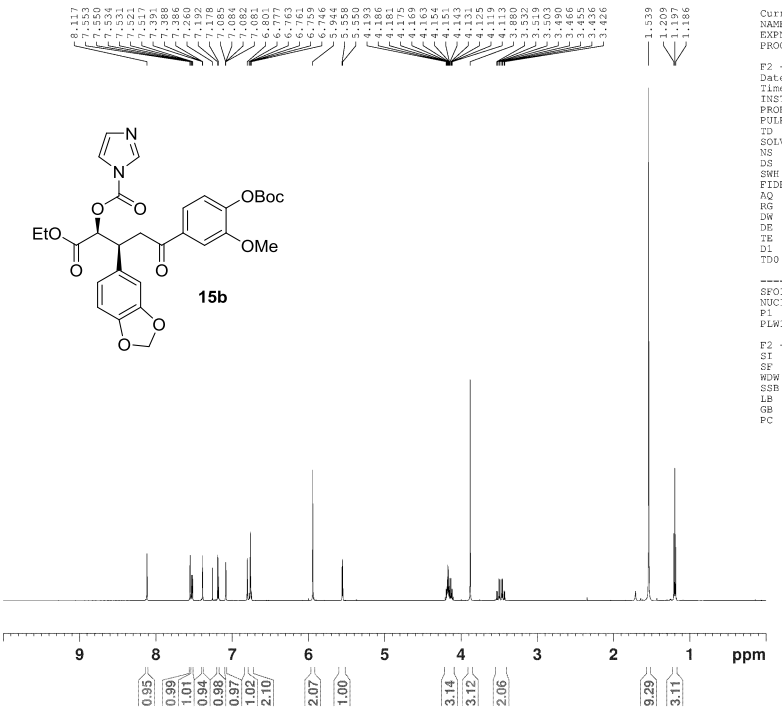

```
Current Data Parameters
NAME        swk5175col13C
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_              20140928
Time                  9.46
INSTRUM              spect
PROBHD   5 mm CPQCI 1H/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     256
DS                       4
SWH              36057.691 Hz
FIDRES            0.550197 Hz
AQ                0.9087659 sec
RG                     203
DW                  13.867 usec
DE                  18.00 usec
TE                  298.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          150.9178981 MHz
NUC1                    13C
P1                   11.35 usec
PLW1        230.00000000 W

-------- CHANNEL f2 --------
SFO2          600.1324005 MHz
NUC2                    1H
CPDPRG[2            waltz16
PCPD2                90.00 usec
PLW2          13.00000000 W
PLW12          0.17192000 W
PLW13          0.13925999 W

F2 - Processing parameters
SI                     32768
SF            150.9028231 MHz
WDW                       EM
SSB         0
LB                    1.00 Hz
GB          0
PC                    1.40
```

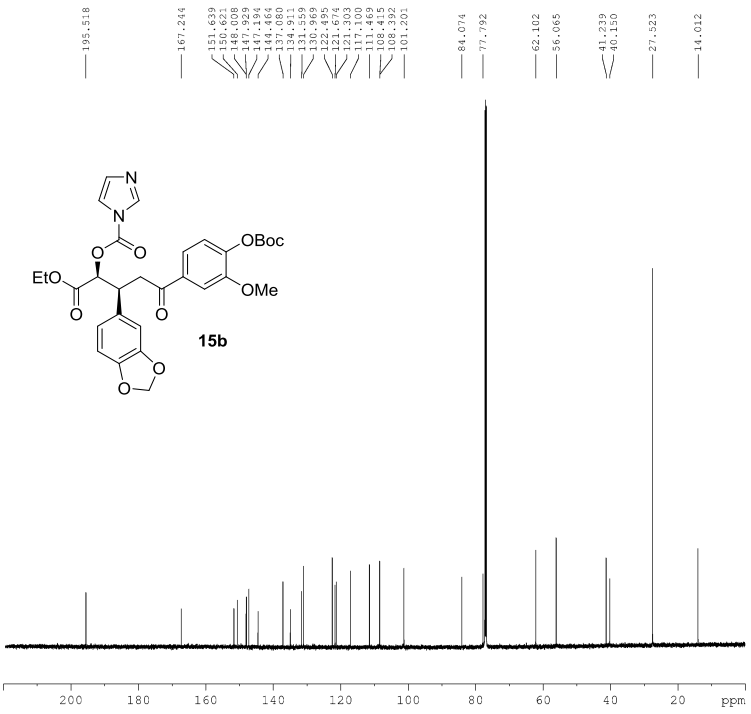



```
Current Data Parameters
NAME        swk5164col-1
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_              20140714
Time                 12.06
INSTRUM              spect
PROBHD   5 mm CPQCI 1H/
PULPROG                zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       2
SWH              12019.230 Hz
FIDRES            0.183399 Hz
AQ                2.7262976 sec
RG                    22.6
DW                 41.600 use
DE                 10.00 use
TE                 298.2 K
D1               1.00000000 sec
TD0                      1

-------- CHANNEL f1 -------
SFO1          600.1337060 MHz
NUC1                    1H
P1                  12.10 use
PLW1         13.00000000 W

F2 - Processing parameters
SI                     65536
SF            600.1300139 MHz
WDW                       EM
SSB         0
LB                    0.30 Hz
GB          0
PC                    1.00
```



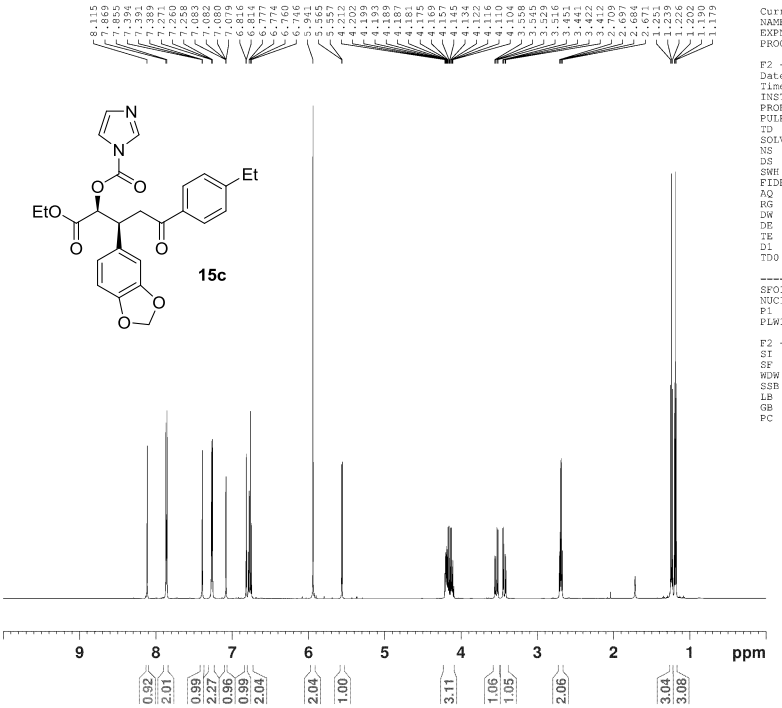
Current Data Parameters
NAME swk5164col-1
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140714
Time 12.09
INSTRUM spect
PROBHD 5 mm CPQCI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 92
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 203
DW 13.867 usec
DE 18.00 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 11.35 usec
PLW1 230.00000000 W

-------- CHANNEL f2 --------
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.23498000 W
PLW13 0.19033000 W

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

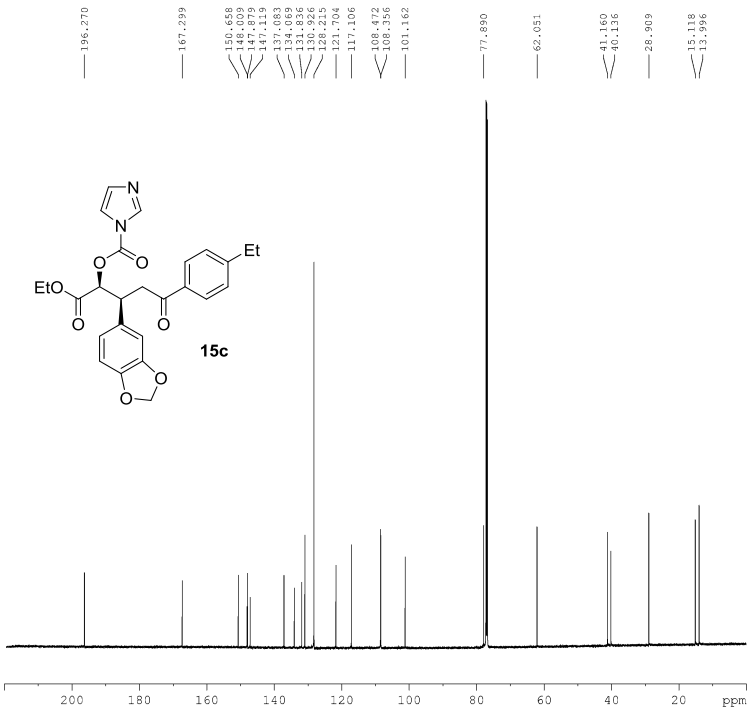


Current Data Parameters
NAME swk5147crude
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140709
Time 21.49
INSTRUM spect
PROBHD 5 mm CPQCI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 9
DS 0
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 14.2
DW 41.600 use
DE 10.00 use
TE 298.1 K
D1 1.00000000 sec
TD0 1

-------- CHANNEL f1 --------
SFO1 600.1337060 MHz
NUC1 1H
P1 12.10 use
PLW1 13.00000000 W

F2 - Processing parameters
SI 65536
SF 600.1300138 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



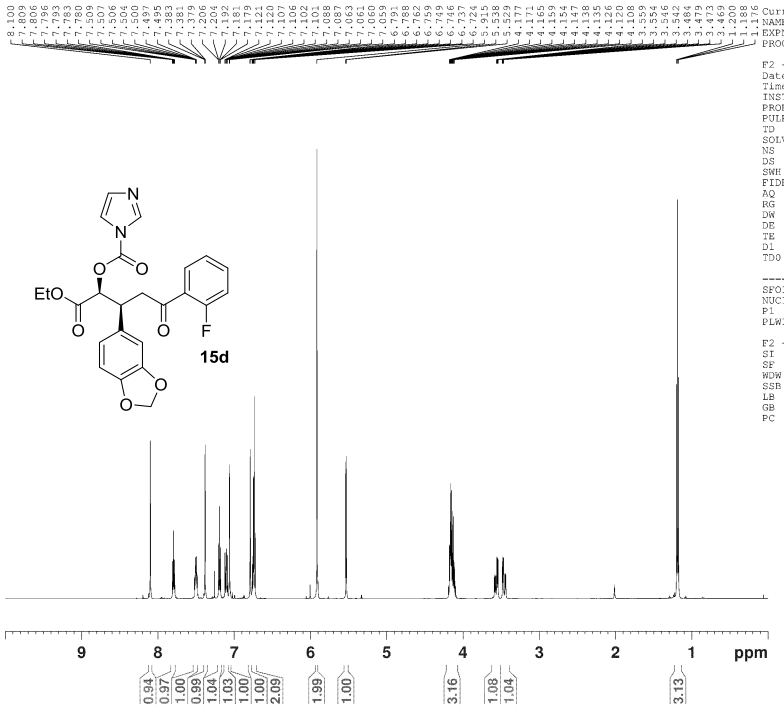

```
Current Data Parameters
NAME                 swk5147col
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20140710
Time                       0.40
INSTRUM                   spect
PROBHD    5 mm CPQCI 1H/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         512
DS                           4
SWH                  36057.691 Hz
FIDRES                0.550197 Hz
AQ                    0.9087659 sec
RG                         203
DW                      13.867 usec
DE                       18.00 usec
TE                       298.2 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                150.9178981 MHz
NUC1                        13C
P1                       11.35 usec
PLW1              230.00000000 W

-------- CHANNEL f2 --------
SFO2                600.1324005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2               13.00000000 W
PLW12               0.23498000 W
PLW13               0.19033000 W

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

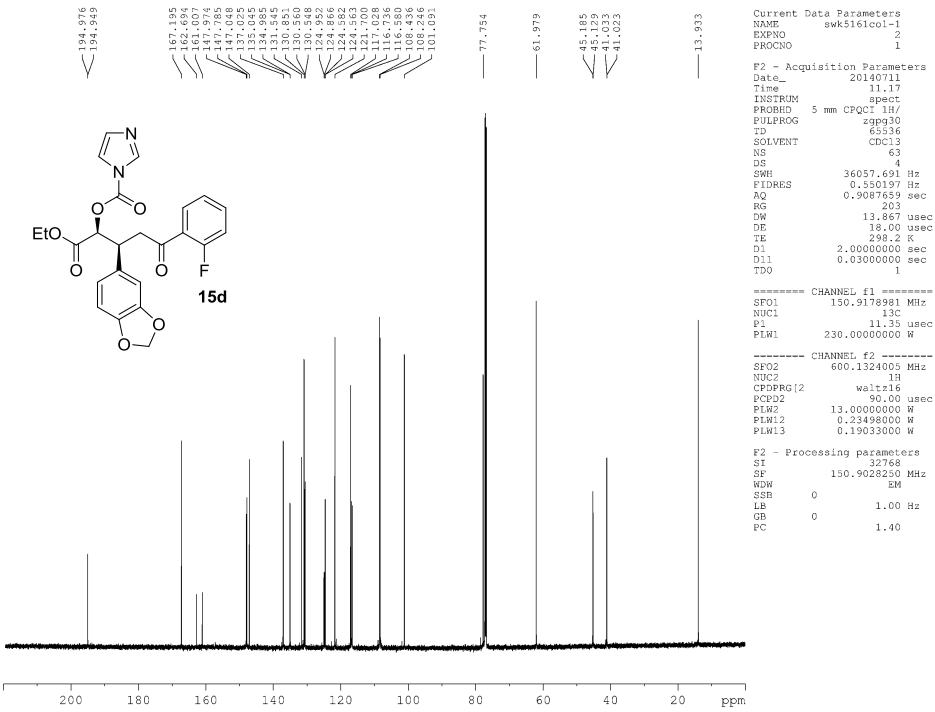



```
Current Data Parameters
NAME                 swk5161col-1
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20140711
Time                      11.13
INSTRUM                   spect
PROBHD    5 mm CPQCI 1H/
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                           8
DS                           2
SWH                  12019.230 Hz
FIDRES                0.183399 Hz
AQ                    2.7262976 sec
RG                           9
DW                      41.600 use
DE                      10.00 use
TE                       298.2 K
D1                    1.00000000 sec
TD0                          1

-------- CHANNEL f1 --------
SFO1                600.1337060 MHz
NUC1                        1H
P1                      12.10 use
PLW1              13.00000000 W

F2 - Processing parameters
SI                       65536
SF                  600.1300137 MHz
WDW                         EM
SSB                  0
LB                       0.30 Hz
GB                   0
PC                       1.00
```



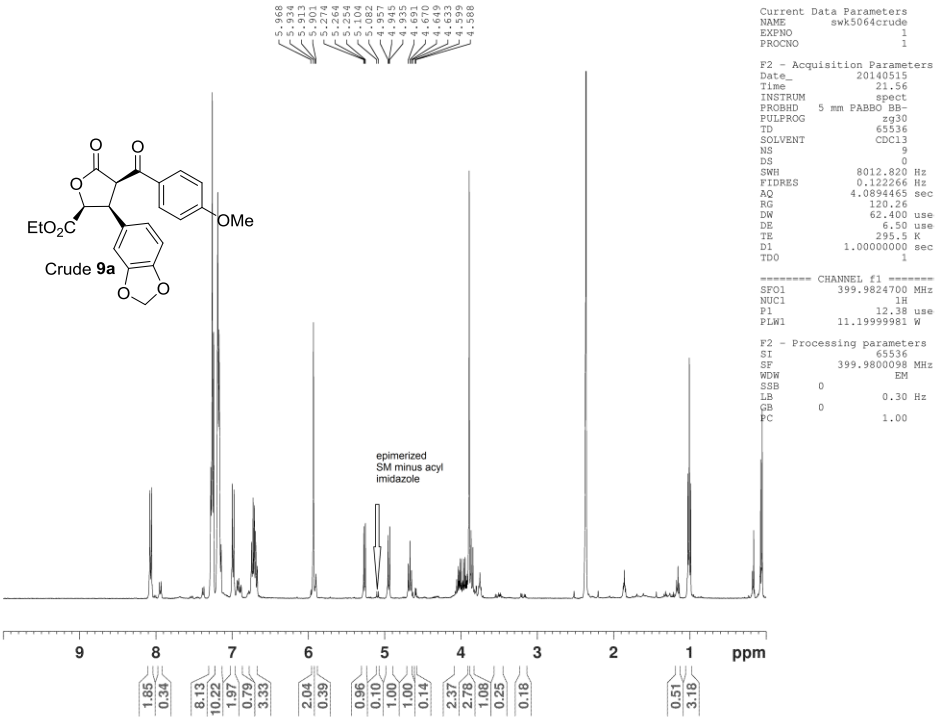
Current Data Parameters
NAME swk5161col-1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140711
Time 11.17
INSTRUM spect
PROBHD 5 mm CPQCI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 63
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 203
DW 13.867 usec
DE 18.00 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9178981 MHz
NUC1 13C
P1 11.35 usec
PLW1 230.00000000 W

-------- CHANNEL f2 --------
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 13.00000000 W
PLW12 0.23498000 W
PLW13 0.19033000 W

F2 - Processing parameters
SI 32768
SF 150.9028250 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

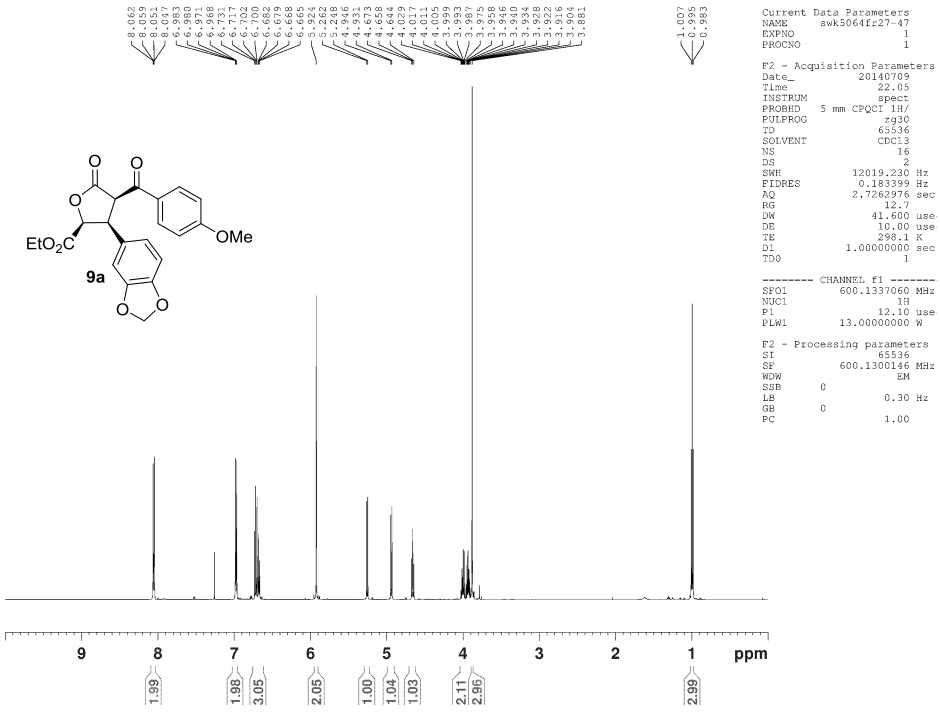


Current Data Parameters
NAME swk5064crude
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140515
Time 21.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 9
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 120.26
DW 62.400 use
DE 6.50 use
TE 295.5 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 399.9824700 MHz
NUC1 1H
P1 12.38 use
PLW1 11.19999981 W

F2 - Processing parameters
SI 65536
SF 399.9800098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

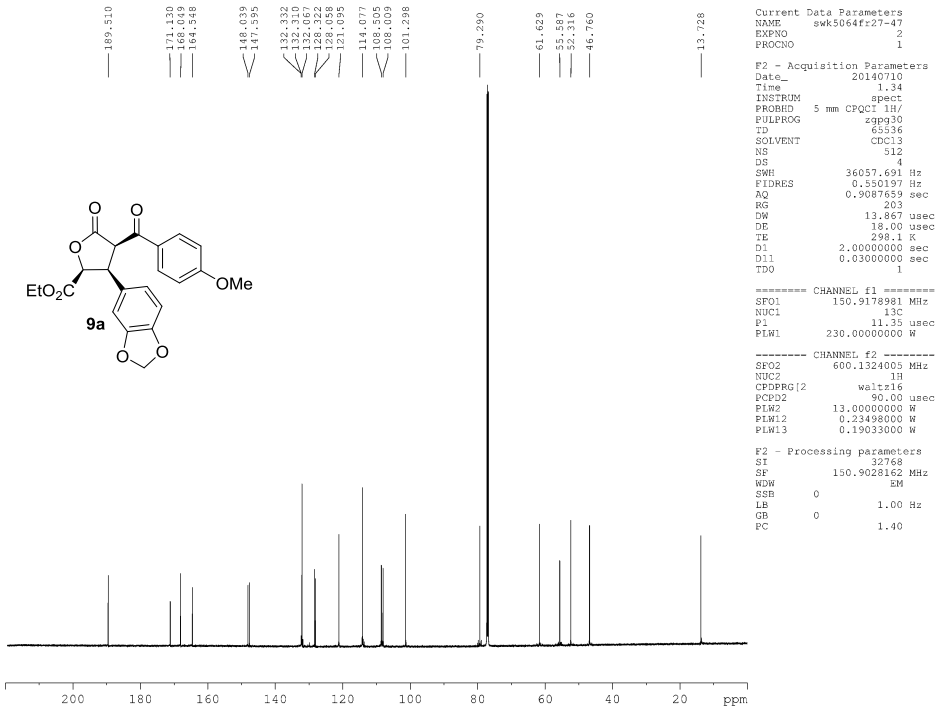



```
Current Data Parameters
NAME      swk5064fr27-47
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20140709
Time               22.05
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG            zg30
TD                65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            12019.230 Hz
FIDRES             0.183399 Hz
AQ             2.7262976 sec
RG                  12.7
DW                 41.600 usec
DE                 10.00 usec
TE                 298.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          600.1337060 MHz
NUC1                  1H
P1                 12.10 usec
PLW1          13.00000000 W

F2 - Processing parameters
SI                 65536
SF            600.1300146 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



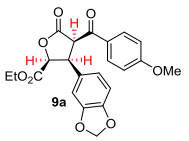

```
Current Data Parameters
NAME      swk5064fr27-47
EXPNO                 2
PROCNO                1

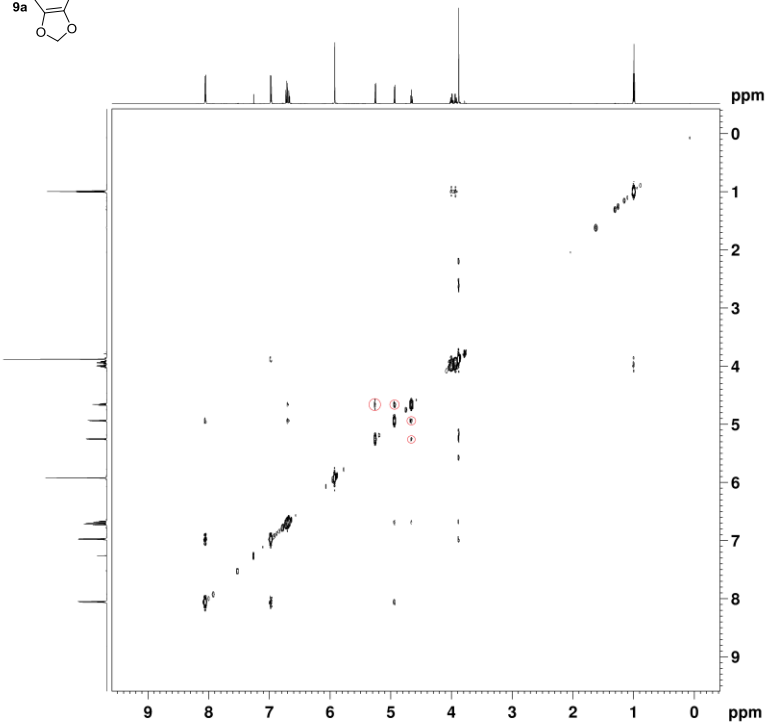
F2 - Acquisition Parameters
Date_           20140710
Time               1.34
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT            CDCl3
NS                  512
DS                    4
SWH            36057.691 Hz
FIDRES             0.550197 Hz
AQ             0.9087659 sec
RG                  203
DW                 13.867 usec
DE                 18.00 usec
TE                 298.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          150.9178981 MHz
NUC1                  13C
P1                 11.35 usec
PLW1       230.00000000 W

======== CHANNEL f2 ========
SFO2          600.1324005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          13.00000000 W
PLW12          0.23498000 W
PLW13          0.19033000 W

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      swk5064fr27-48
EXPNO                  3
PROCNO                 1

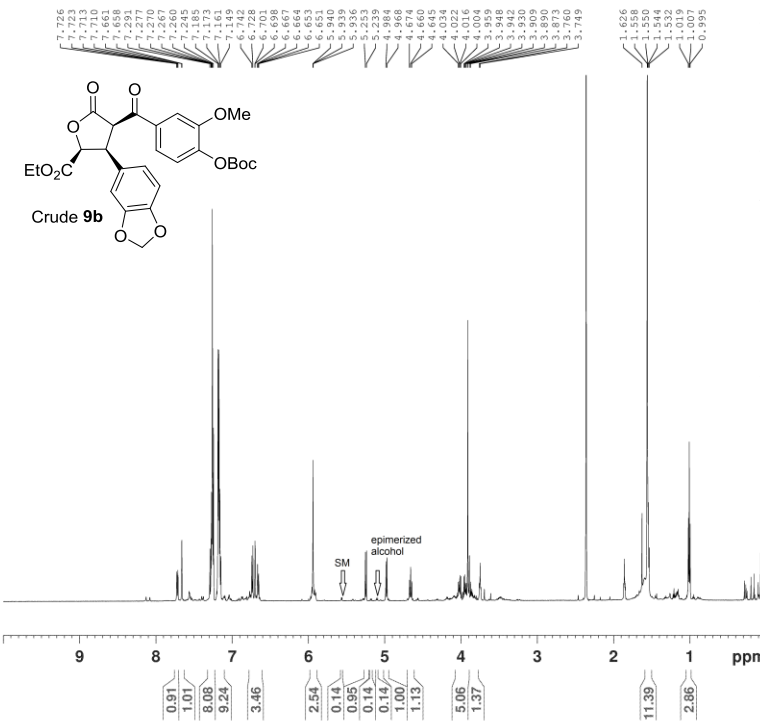
F2 - Acquisition Parameters
Date_           20140710
Time               1.37
INSTRUM           spect
PROBHD   5 mm CPQCI 1H/
PULPROG     noesygpphpp
TD                 2048
SOLVENT            CDCl3
NS                    4
DS                   32
SWH             6009.615 Hz
FIDRES          2.934382 Hz
AQ              0.1703936 sec
RG                   64
DW                83.200 use
DE                10.00 use
TE                298.2 K
D0             0.00006779 sec
D1             2.00000000 sec
D8             0.30000001 sec
D11            0.03000000 sec
D12            0.00002000 sec
D16            0.00020000 sec
IN0            0.00016640 sec

======== CHANNEL f1 =======
SFO1          600.1327625 MHz
NUC1                  1H
P1                 12.10 use
P2                 24.20 use
P17              2500.00 use
PLW1          13.00000000 W
PLW10          2.81559992 W

====== GRADIENT CHANNEL ===
GPNAM[1]      SMSQ10.100
GPZ1               40.00 %
P16              1000.00 use

F1 - Acquisition parameters
TD                   256
SFO1            600.1328 MHz
FIDRES         23.475060 Hz
SW                10.014 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                  1024
SF            600.1300108 MHz
WDW                 QSINE
SSB                     2
LB         0 Hz
GB         0
PC                   1.00
```

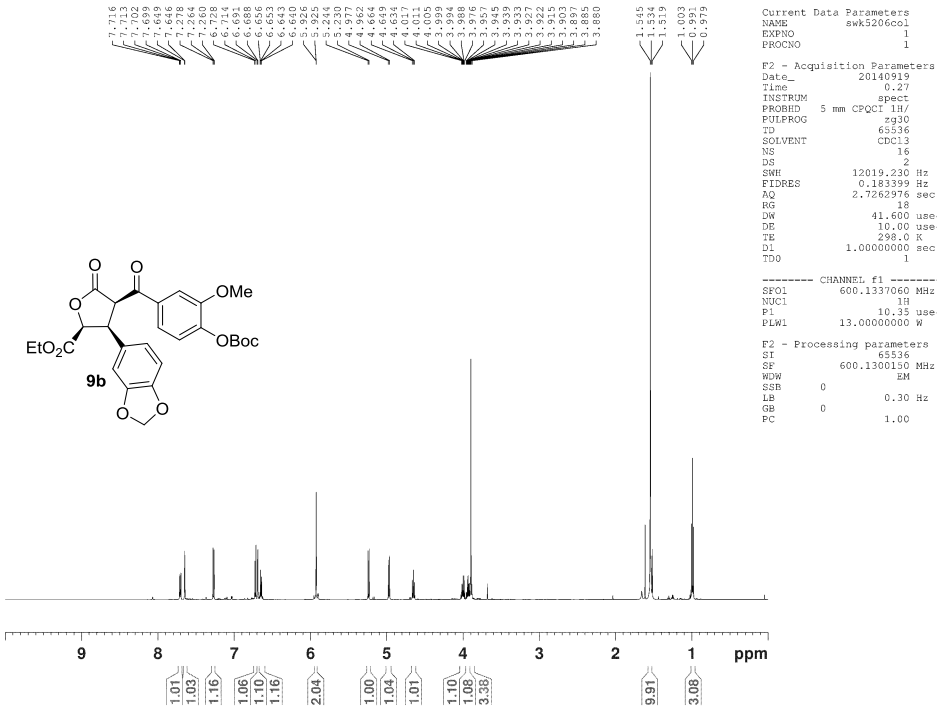



```
Current Data Parameters
NAME       swk5206crude
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20140827
Time              15.16
INSTRUM           spect
PROBHD   5 mm CPQCI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    6
DS                    0
SWH             12019.230 Hz
FIDRES          0.183399 Hz
AQ              2.7262976 sec
RG                   18
DW                41.600 use
DE                10.00 use
TE                298.2 K
D1             1.00000000 sec
TD0                   1

======== CHANNEL f1 =======
SFO1          600.1337060 MHz
NUC1                  1H
P1                 10.35 use
PLW1          13.00000000 W

F2 - Processing parameters
SI                 65536
SF            600.1300136 MHz
WDW                   EM
SSB        0
LB                 0.30 Hz
GB         0
PC                   1.00
```

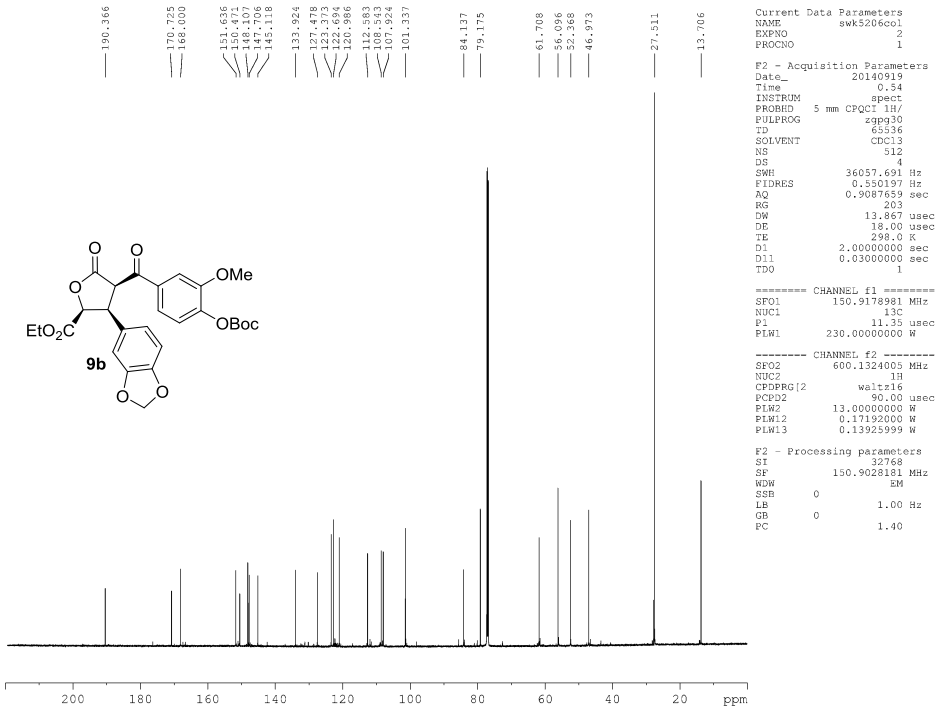



```
Current Data Parameters
NAME                 swk5206col
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_               20140919
Time                    0.27
INSTRUM                spect
PROBHD    5 mm CPQCI 1H/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                12019.230 Hz
FIDRES                0.183399 Hz
AQ                 2.7262976 sec
RG                       18
DW                    41.600 use
DE                    10.00 use
TE                    298.0 K
D1                 1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1              600.1337060 MHz
NUC1                        1H
P1                       10.35 use
PLW1              13.00000000 W

F2 - Processing parameters
SI                        65536
SF                600.1300150 MHz
WDW                         EM
SSB          0
LB                        0.30 Hz
GB           0
PC                        1.00
```



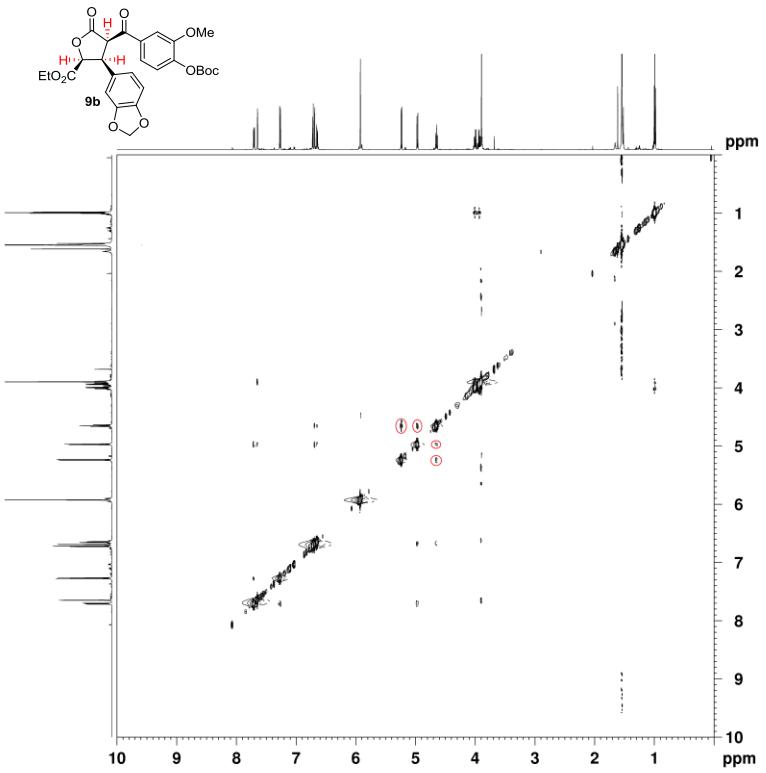

```
Current Data Parameters
NAME                 swk5206col
EXPNO                         2
PROCNO                        1

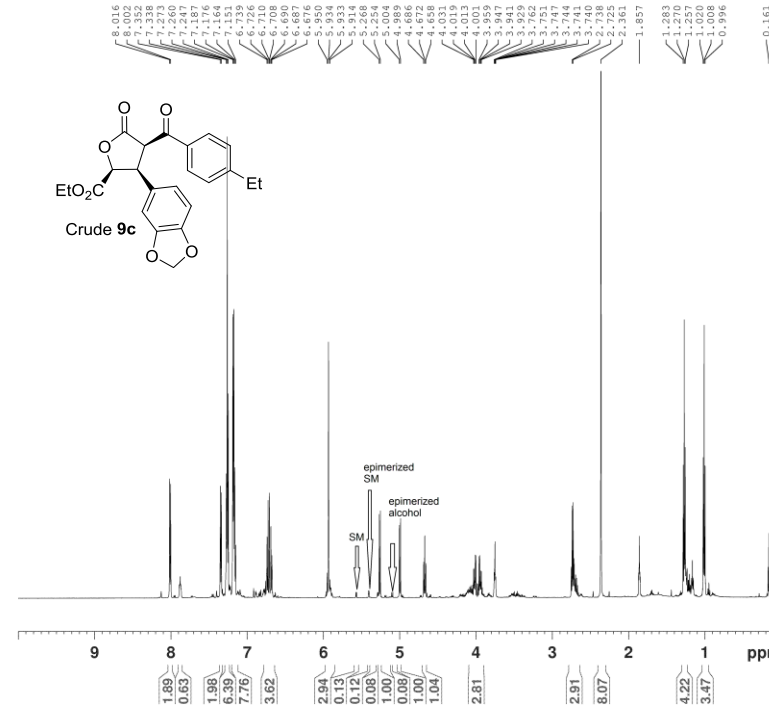
F2 - Acquisition Parameters
Date_               20140919
Time                    0.54
INSTRUM                spect
PROBHD    5 mm CPQCI 1H/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      512
DS                        4
SWH                36057.691 Hz
FIDRES                0.550197 Hz
AQ                 0.9087659 sec
RG                      203
DW                    13.867 usec
DE                    18.00 usec
TE                    298.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                           1

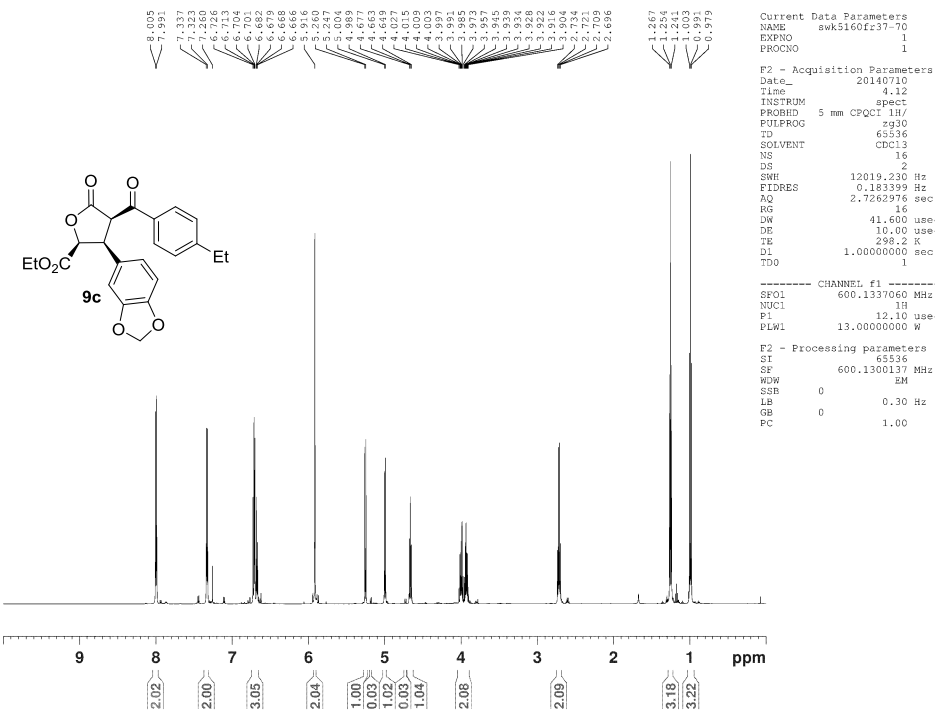
======== CHANNEL f1 ========
SFO1              150.9178981 MHz
NUC1                       13C
P1                       11.35 usec
PLW1          230.00000000 W

======== CHANNEL f2 ========
SFO2              600.1324005 MHz
NUC2                        1H
CPDPRG[2               waltz16
PCPD2                    90.00 usec
PLW2              13.00000000 W
PLW12              0.17192000 W
PLW13              0.13925999 W

F2 - Processing parameters
SI                        32768
SF                150.9028181 MHz
WDW                         EM
SSB          0
LB                        1.00 Hz
GB           0
PC                        1.40
```


OMe
OBoc
EtO2C
9b
ppm


EtO2C
Et
Crude 9c
epimerized SM
epimerized alcohol
SM
ppm

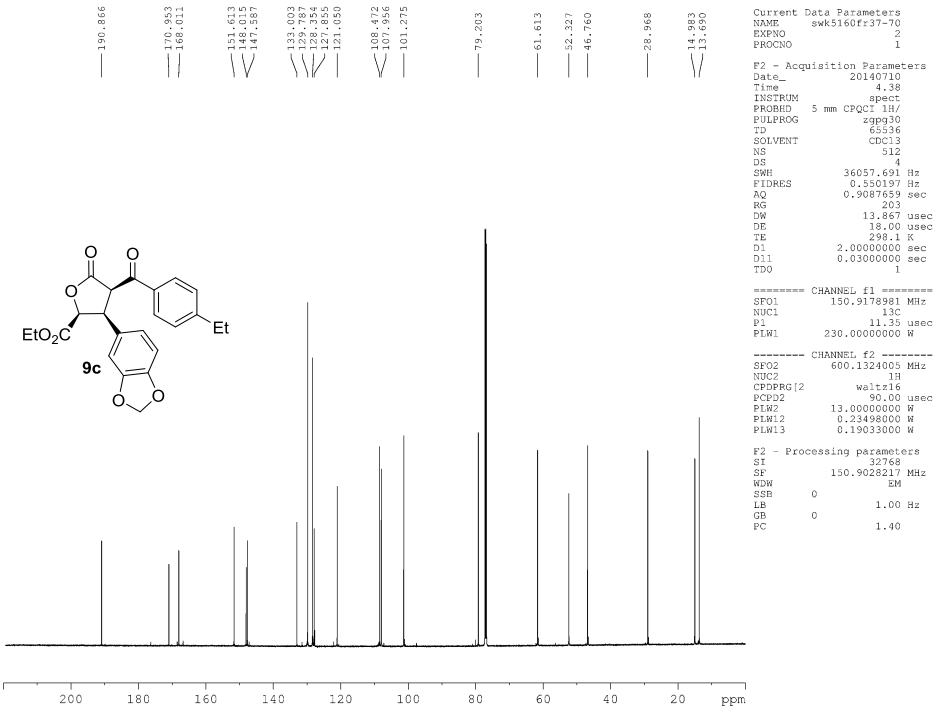



```
Current Data Parameters
NAME      swk5160fr37-70
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140710
Time                4.12
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           12019.230 Hz
FIDRES         0.183399 Hz
AQ             2.7262976 sec
RG                   16
DW                41.600 use
DE                10.00 use
TE                298.2 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.1337060 MHz
NUC1                 1H
P1                12.10 use
PLW1         13.00000000 W

F2 - Processing parameters
SI                65536
SF           600.1300137 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



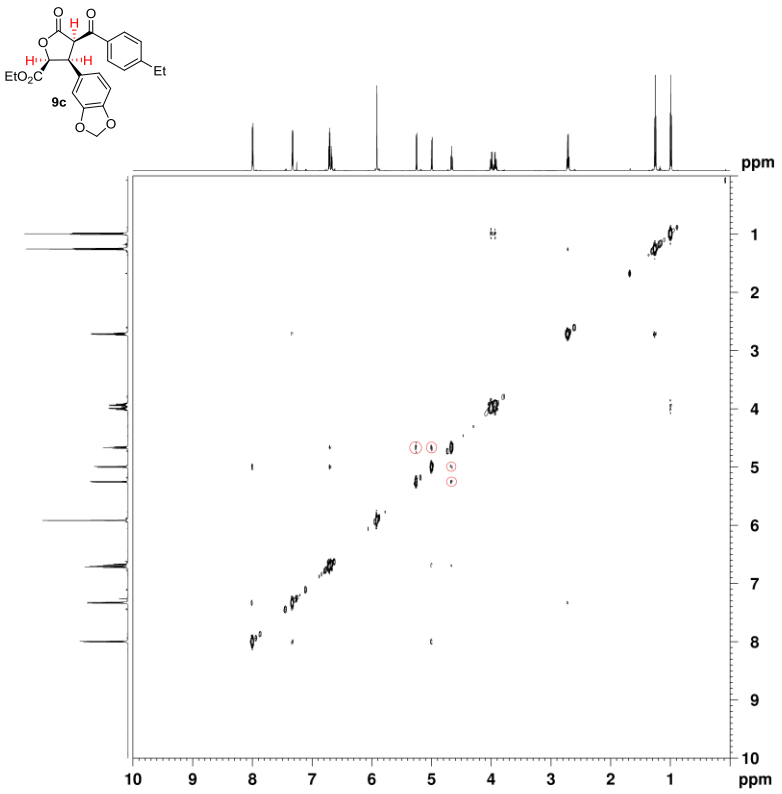

```
Current Data Parameters
NAME      swk5160fr37-70
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140710
Time                4.38
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ             0.9087659 sec
RG                  203
DW                13.867 usec
DE                18.00 usec
TE                298.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         150.9178981 MHz
NUC1                 13C
P1                11.35 usec
PLW1        230.00000000 W

======== CHANNEL f2 ========
SFO2         600.1324005 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         13.00000000 W
PLW12         0.23498000 W
PLW13         0.19033000 W

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      swk5160fr37-70
EXPNO                 3
PROCNO                1

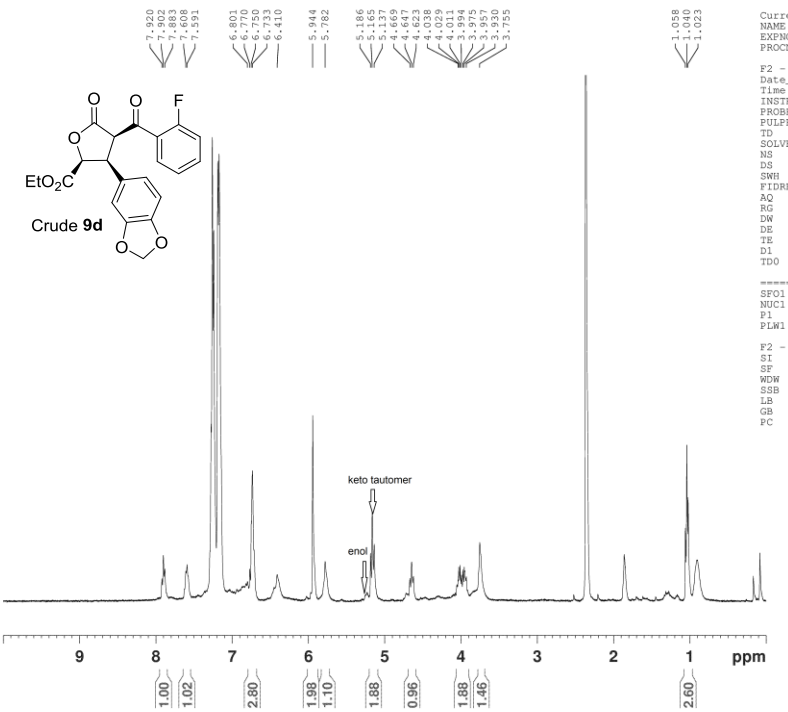
F2 - Acquisition Parameters
Date_          20140710
Time               4.40
INSTRUM           spect
PROBHD   5 mm CPQCI 1H/
PULPROG     noesygpphpp
TD                 2048
SOLVENT            CDCl3
NS                    4
DS                   32
SWH             6009.615 Hz
FIDRES          2.934382 Hz
AQ             0.1703936 sec
RG                   36
DW                83.200 use
DE                10.00 use
TE                298.1 K
D0             0.00006779 sec
D1             2.00000000 sec
D8             0.30000001 sec
D11            0.03000000 sec
D12            0.00002000 sec
D16            0.00020000 sec
IN0            0.00016640 sec

======== CHANNEL f1 =======
SFO1          600.1327625 MHz
NUC1                   1H
P1                  12.10 use
P2                  24.20 use
P17               2500.00 use
PLW1          13.00000000 W
PLW10          2.81559992 W

====== GRADIENT CHANNEL ===
GPNAM[1]      SMSQ10.100
GPZ1               40.00 %
P16              1000.00 use

F1 - Acquisition parameters
TD                  256
SFO1          600.1328 MHz
FIDRES        23.475060 Hz
SW                10.014 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                 1024
SF            600.1300107 MHz
WDW                QSINE
SSB                    2
LB         0 Hz
GB         0
PC                 1.00
```

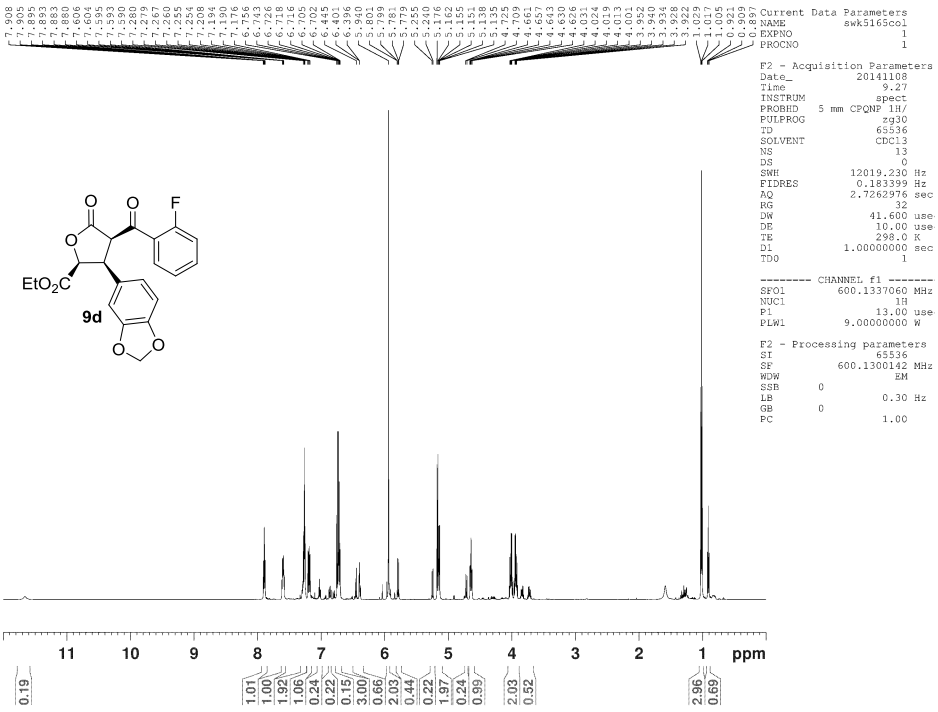


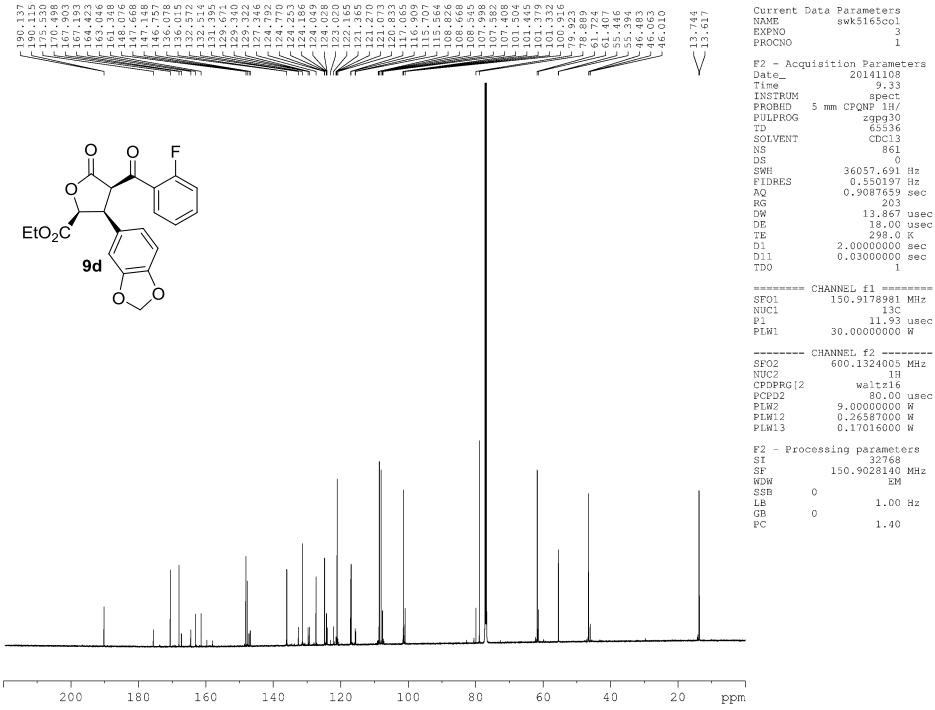

```
Current Data Parameters
NAME        swk5165crude
EXPNO                   1
PROCNO                  1

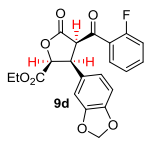
F2 - Acquisition Parameters
Date_          20140711
Time              21.21
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                178.34
DW                62.400 use
DE                 6.50 use
TE                313.5 K
D1             1.00000000 sec
TD0                    1

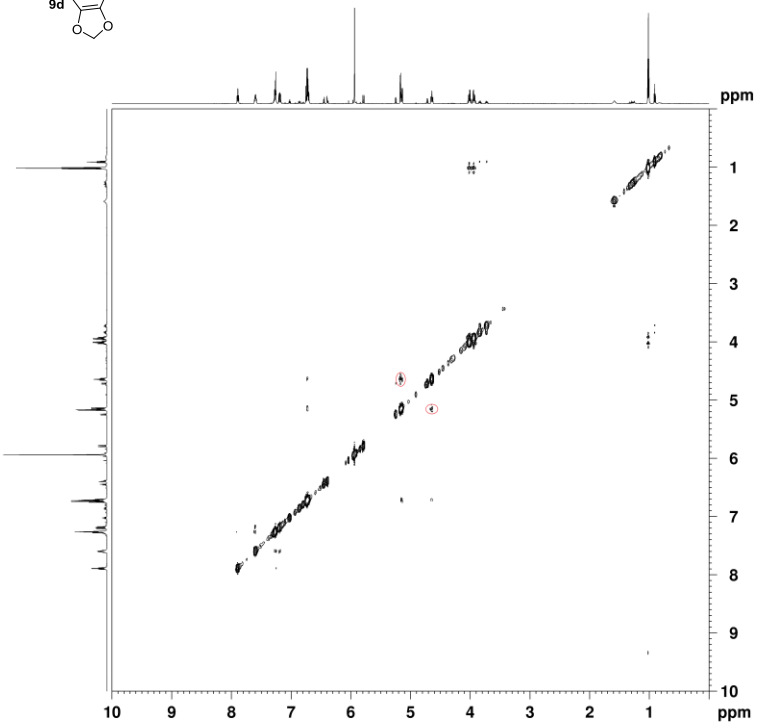
======== CHANNEL f1 =======
SFO1          399.9824700 MHz
NUC1                   1H
P1                  12.38 use
PLW1          11.19999981 W

F2 - Processing parameters
SI                 65536
SF            399.9800081 MHz
WDW                   EM
SSB        0
LB                 0.30 Hz
GB         0
PC                 1.00
```


9d
Current Data Parameters
NAME swk5165col
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141108
Time 9.27
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 13
DS 0
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 32
DW 41.600 use
DE 10.00 use
TE 298.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 600.1337060 MHz
NUC1 1H
P1 13.00 use
PLW1 9.00000000 W
F2 - Processing parameters
SI 65536
SF 600.1300142 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm


9d
Current Data Parameters
NAME swk5165col
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20141108
Time 9.33
INSTRUM spect
PROBHD 5 mm CPQNP 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 861
DS 0
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 203
DW 13.867 usec
DE 18.00 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
SFO1 150.9178981 MHz
NUC1 13C
P1 11.93 usec
PLW1 30.00000000 W
CHANNEL f2
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 9.00000000 W
PLW12 0.26587000 W
PLW13 0.17016000 W
F2 - Processing parameters
SI 32768
SF 150.9028140 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm






```
Current Data Parameters
NAME                 swk5165col
EXPNO                         4
PROCNO                        1

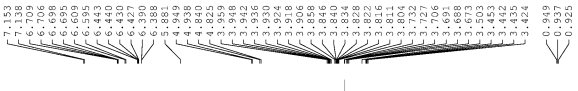
F2 - Acquisition Parameters
Date_               20141108
Time                   10.18
INSTRUM                spect
PROBHD    5 mm CPQNP 1H/
PULPROG          noesygpphpp
TD                       2048
SOLVENT                 CDCl3
NS                            4
DS                           32
SWH                  6009.615 Hz
FIDRES               2.934382 Hz
AQ                   0.1703936 sec
RG                          203
DW                     83.200 use
DE                     10.00 use
TE                     298.0 K
D0                   0.00006665 sec
D1                   2.00000000 sec
D8                   0.30000001 sec
D11                  0.03000000 sec
D12                  0.00002000 sec
D16                  0.00020000 sec
IN0                  0.00016640 sec

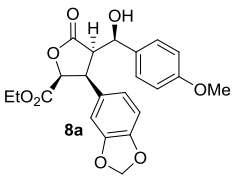
======== CHANNEL f1 =======
SFO1                600.1327625 MHz
NUC1                        1H
P1                     13.00 use
P2                     26.00 use
P17                  2500.00 use
PLW1                 9.00000000 W
PLW10                2.25000000 W

====== GRADIENT CHANNEL ===
GPNAM[1]            SMSQ10.100
GPZ1                   40.00 %
P16                  1000.00 use

F1 - Acquisition parameters
TD                           256
SFO1                  600.1328 MHz
FIDRES               23.475060 Hz
SW                       10.014 ppm
FnMODE            States-TPPI

F2 - Processing parameters
SI                          1024
SF                  600.1300140 MHz
WDW                       QSINE
SSB                           2
LB          0 Hz
GB          0
PC                         1.00
```



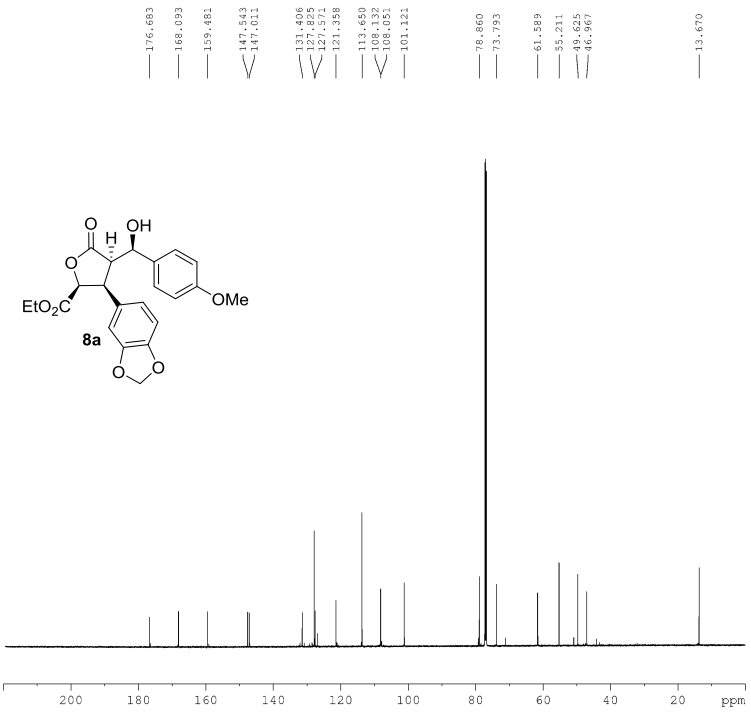



```
Current Data Parameters
NAME          swk5276crude
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_              20141117
Time                  21.46
INSTRUM               spect
PROBHD   5 mm CPQNP 1H/
PULPROG                zg30
TD                    65536
SOLVENT              CDCl3
NS                       16
DS                        2
SWH                12019.230 Hz
FIDRES             0.183399 Hz
AQ                 2.7262976 sec
RG                       32
DW                    41.600 use
DE                    10.00 use
TE                    298.0 K
D1                 1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1              600.1337060 MHz
NUC1                      1H
P1                   13.00 use
PLW1               9.00000000 W

F2 - Processing parameters
SI                       65536
SF                600.1300155 MHz
WDW                      EM
SSB          0
LB                    0.30 Hz
GB           0
PC                       1.00
```



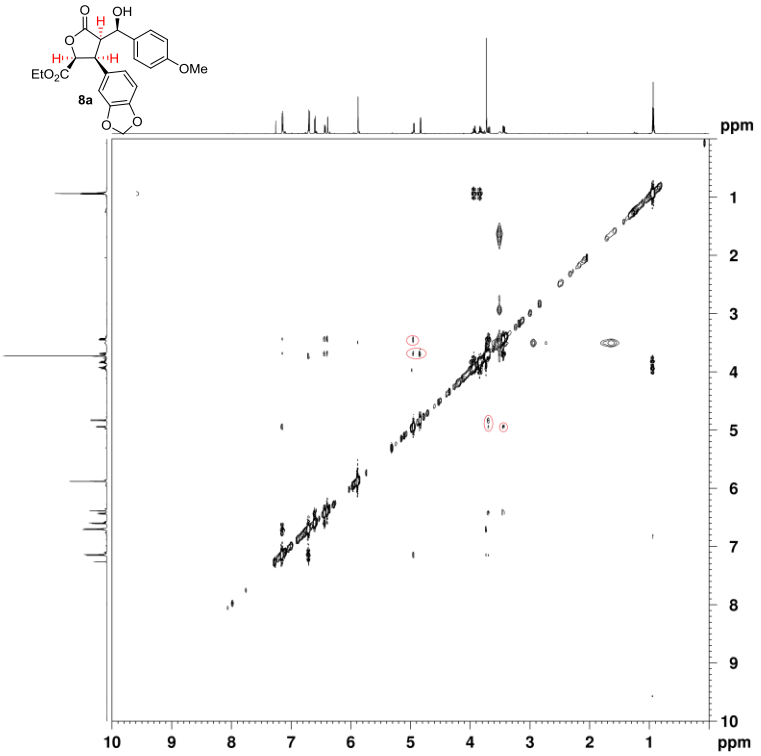

```
Current Data Parameters
NAME          swk5276crude
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_              20141117
Time                  21.49
INSTRUM               spect
PROBHD   5 mm CPQNP 1H/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       60
DS                        4
SWH               36057.691 Hz
FIDRES             0.550197 Hz
AQ                0.9087659 sec
RG                      203
DW                   13.867 usec
DE                   18.00 usec
TE                   298.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1          150.9178981 MHz
NUC1                    13C
P1                    11.93 usec
PLW1          30.00000000 W

-------- CHANNEL f2 --------
SFO2          600.1324005 MHz
NUC2                    1H
CPDPRG[2            waltz16
PCPD2                80.00 usec
PLW2           9.00000000 W
PLW12          0.26587000 W
PLW13          0.17016000 W

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          swk5276crude
EXPNO                    4
PROCNO                   1

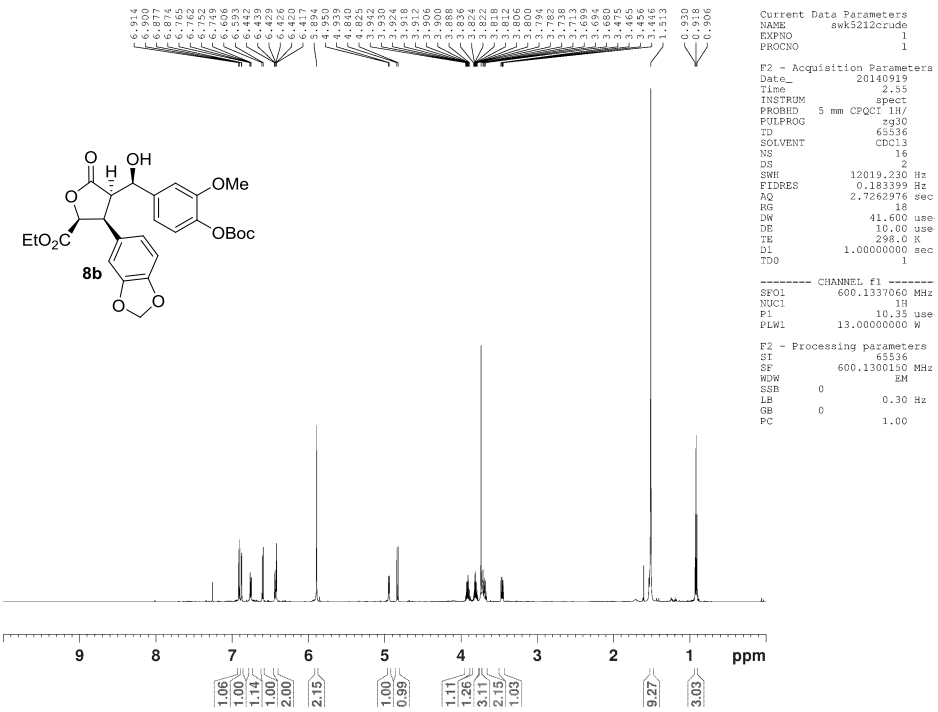
F2 - Acquisition Parameters
Date_              20141117
Time                  21.52
INSTRUM               spect
PROBHD   5 mm CPQNP 1H/
PULPROG          noesygpphpp
TD                     2048
SOLVENT               CDCl3
NS                        4
DS                       32
SWH                6009.615 Hz
FIDRES             2.934382 Hz
AQ                0.1703936 sec
RG                      101
DW                   83.200 use
DE                   10.00 use
TE                   298.0 K
D0                0.00006665 sec
D1                2.00000000 sec
D8                0.30000001 sec
D11               0.03000000 sec
D12               0.00002000 sec
D16               0.00020000 sec
IN0               0.00016640 sec

======== CHANNEL f1 ========
SFO1          600.1327625 MHz
NUC1                    1H
P1                    13.00 use
P2                    26.00 use
P17                 2500.00 use
PLW1           9.00000000 W
PLW10          2.25000000 W

====== GRADIENT CHANNEL ===
GPNAM[1]       SMSQ10.100
GPZ1                  40.00 %
P16                 1000.00 use

F1 - Acquisition parameters
TD                       256
SFO1              600.1328 MHz
FIDRES            23.475060 Hz
SW                   10.014 ppm
FnMODE         States-TPPI

F2 - Processing parameters
SI                       1024
SF            600.1300110 MHz
WDW                    QSINE
SSB                        2
LB          0 Hz
GB          0
PC                     1.00
```

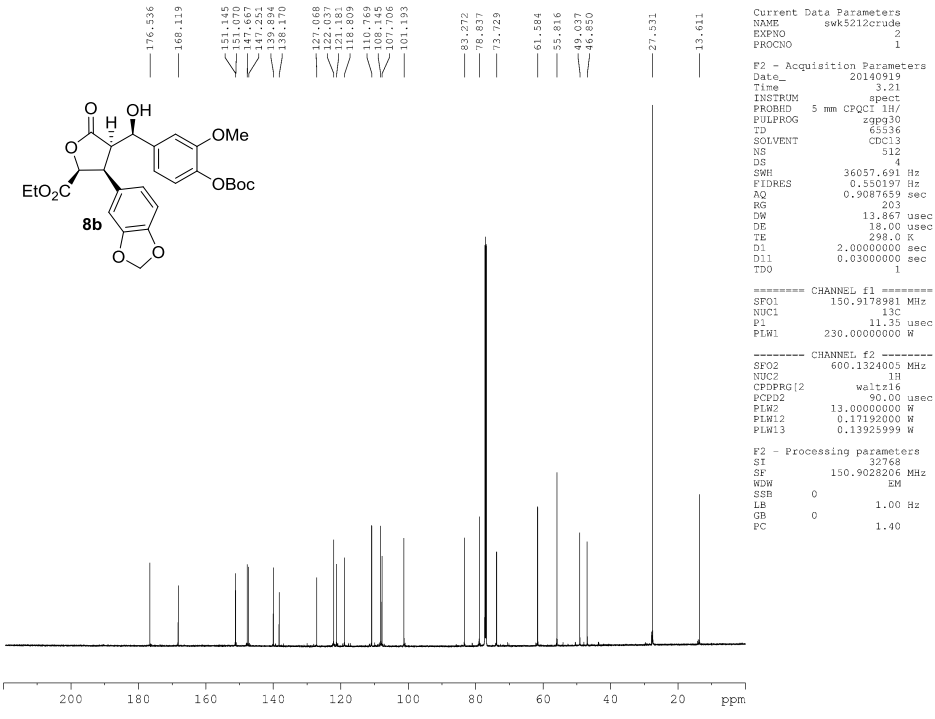



```
Current Data Parameters
NAME         swk5212crude
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_             20140919
Time                  2.55
INSTRUM              spect
PROBHD    5 mm CPQCI 1H/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH           12019.230 Hz
FIDRES         0.183399 Hz
AQ             2.7262976 sec
RG                     18
DW                41.600 use
DE                10.00 use
TE                298.0 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         600.1337060 MHz
NUC1                   1H
P1                 10.35 use
PLW1        13.00000000 W

F2 - Processing parameters
SI                  65536
SF           600.1300150 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



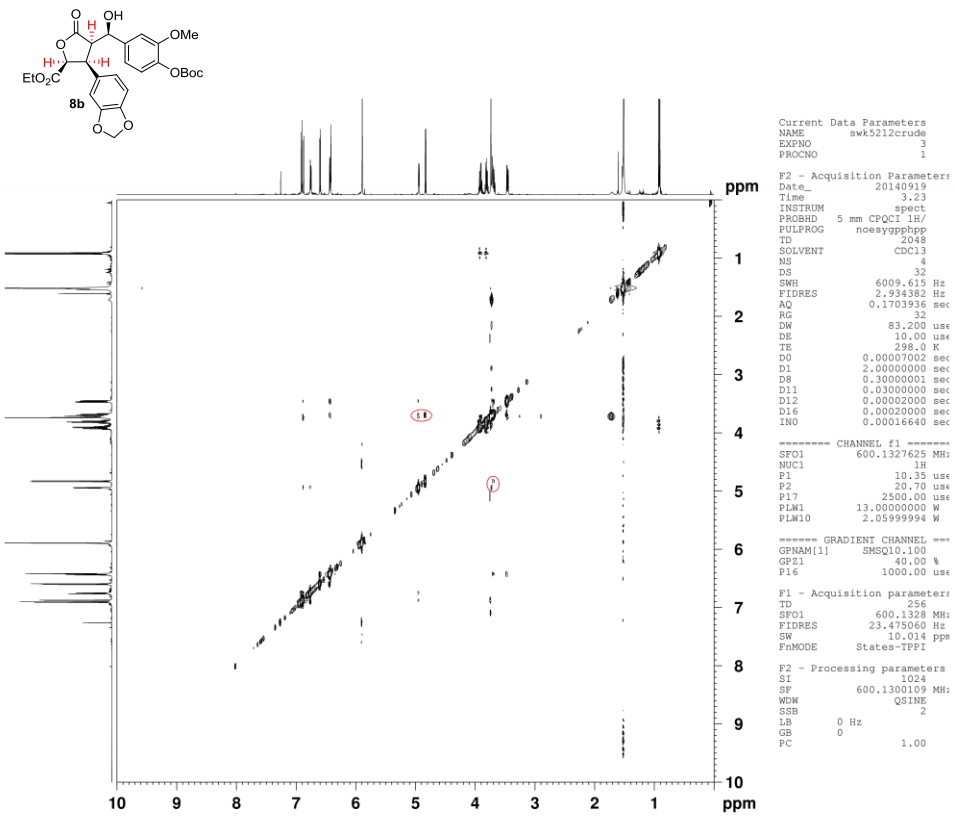

```
Current Data Parameters
NAME         swk5212crude
EXPNO                   2
PROCNO                  1

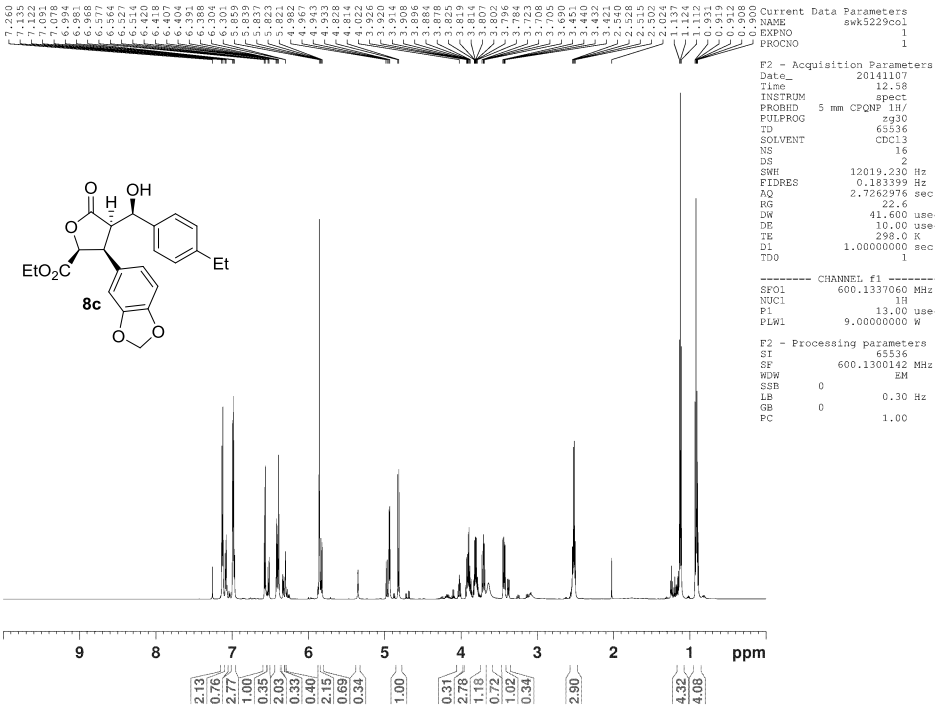
F2 - Acquisition Parameters
Date_             20140919
Time                  3.21
INSTRUM              spect
PROBHD    5 mm CPQCI 1H/
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    512
DS                      4
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ             0.9087659 sec
RG                    203
DW                13.867 usec
DE                18.00 usec
TE                298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

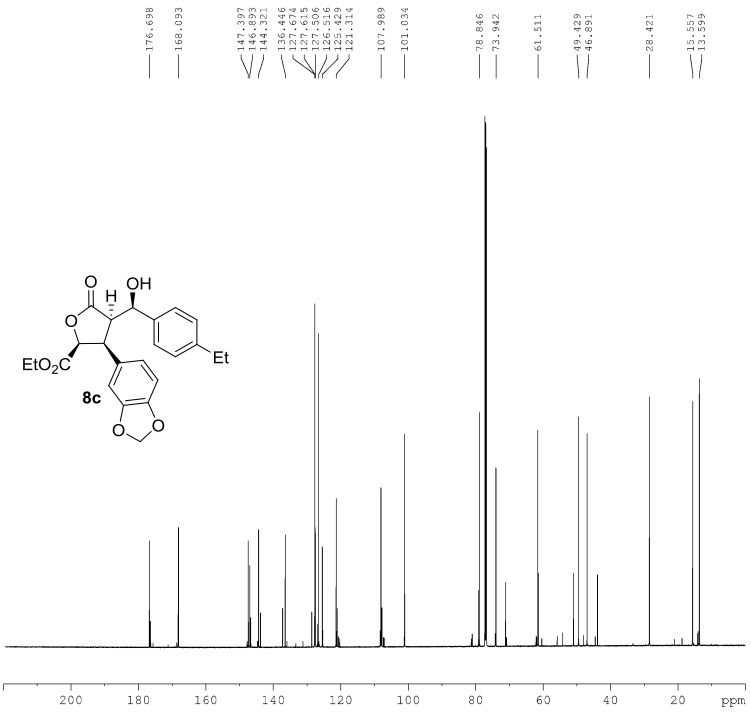
======== CHANNEL f1 ========
SFO1        150.9178981 MHz
NUC1                   13C
P1                 11.35 usec
PLW1       230.00000000 W

======== CHANNEL f2 ========
SFO2         600.1324005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2        13.00000000 W
PLW12        0.17192000 W
PLW13        0.13925999 W

F2 - Processing parameters
SI                  32768
SF          150.9028206 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```


OH
OMe
OBoc
EtO2C
8b
ppm


OH
Et
EtO2C
8c
ppm



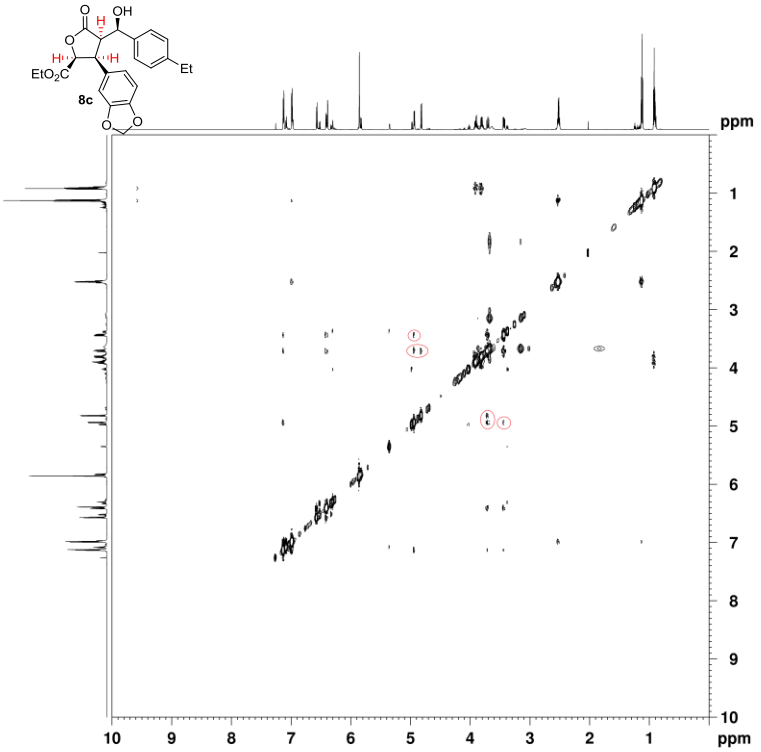

```
Current Data Parameters
NAME                 swk5229co1
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20141107
Time                      13.12
INSTRUM                   spect
PROBHD    5 mm CPQNP 1H/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         256
DS                           4
SWH                  36057.691 Hz
FIDRES                0.550197 Hz
AQ                   0.9087659 sec
RG                         203
DW                    13.867 usec
DE                     18.00 usec
TE                     298.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1           150.9178981 MHz
NUC1                     13C
P1                     11.93 usec
PLW1          30.00000000 W

-------- CHANNEL f2 --------
SFO2           600.1324005 MHz
NUC2                     1H
CPDPRG[2              waltz16
PCPD2                  80.00 usec
PLW2           9.00000000 W
PLW12          0.26587000 W
PLW13          0.17016000 W

F2 - Processing parameters
SI                       32768
SF             150.9028238 MHz
WDW                         EM
SSB         0
LB                      1.00 Hz
GB          0
PC                      1.40
```




```
Current Data Parameters
NAME                 swk5229co1
EXPNO                         3
PROCNO                        1

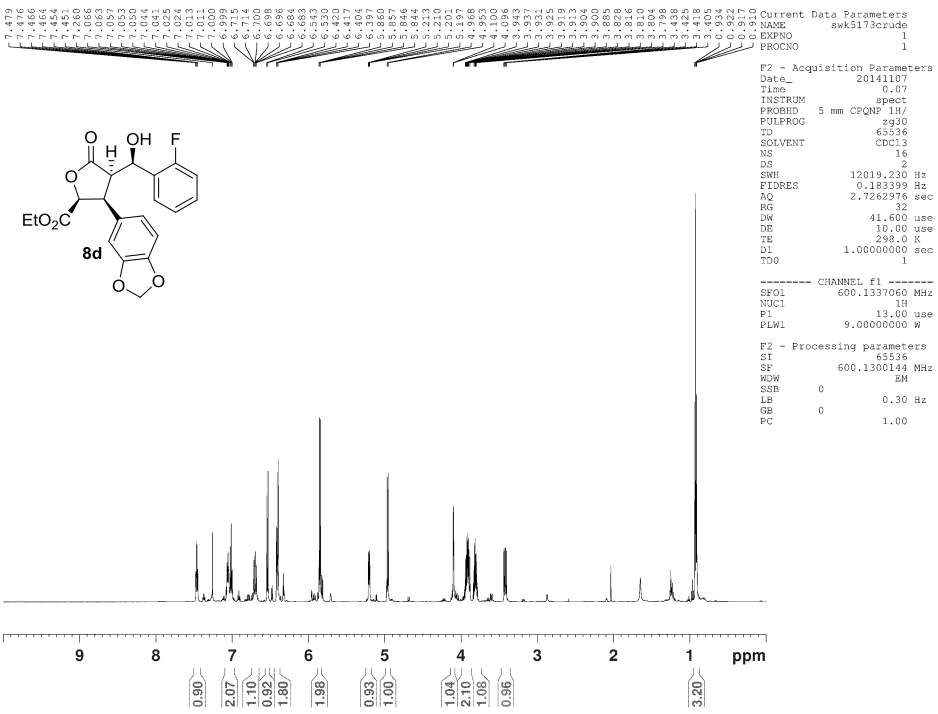
F2 - Acquisition Parameters
Date_                  20141117
Time                      22.38
INSTRUM                   spect
PROBHD    5 mm CPQNP 1H/
PULPROG          noesygpphpp
TD                        2048
SOLVENT                  CDCl3
NS                           4
DS                          32
SWH                  6009.615 Hz
FIDRES               2.934382 Hz
AQ                   0.1703936 sec
RG                        28.5
DW                    83.200 use
DE                     10.00 use
TE                     298.0 K
D0                  0.00006665 sec
D1                  2.00000000 sec
D8                  0.30000001 sec
D11                 0.03000000 sec
D12                 0.00002000 sec
D16                 0.00020000 sec
IN0                 0.00016640 sec

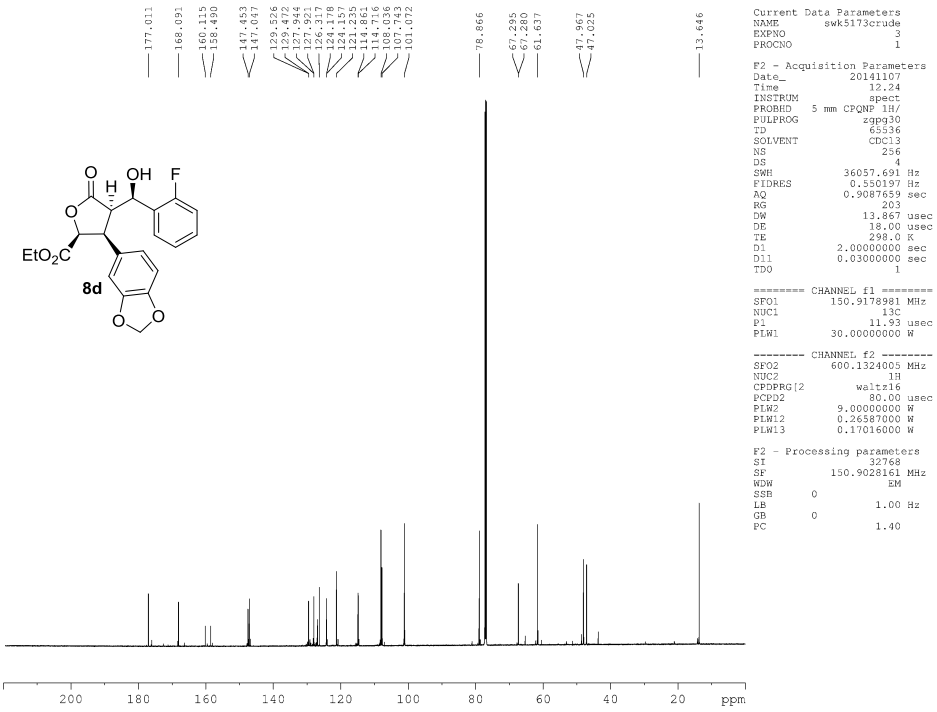
======== CHANNEL f1 ========
SFO1           600.1327625 MHz
NUC1                     1H
P1                     13.00 use
P2                     26.00 use
P17                  2500.00 use
PLW1           9.00000000 W
PLW10          2.25000000 W

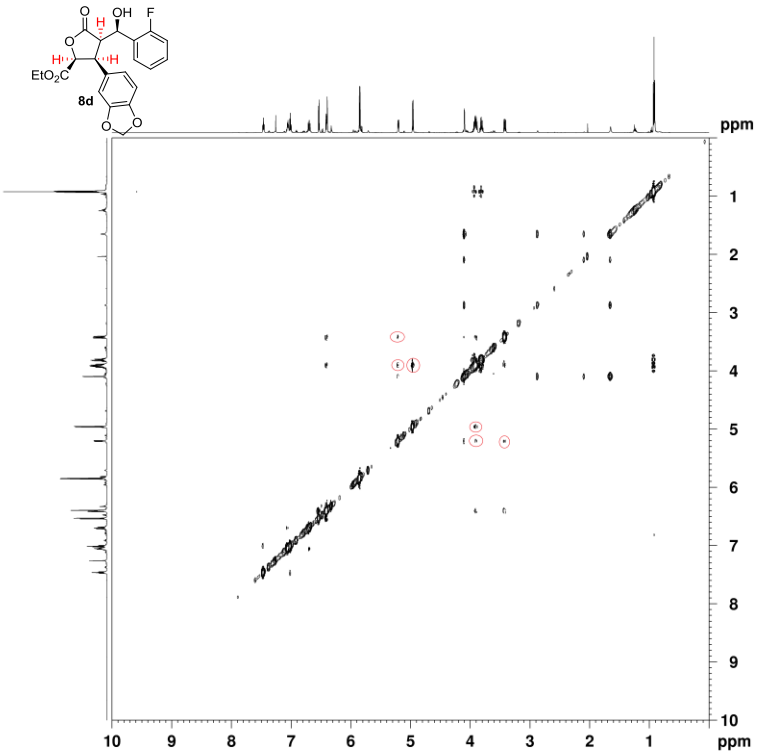
====== GRADIENT CHANNEL ===
GPNAM[1]       SMSQ10.100
GPZ1                  40.00 %
P16                 1000.00 use

F1 - Acquisition parameters
TD                       256
SFO1             600.1328 MHz
FIDRES           23.475060 Hz
SW                   10.014 ppm
FnMODE         States-TPPI

F2 - Processing parameters
SI                      1024
SF             600.1300108 MHz
WDW                   QSINE
SSB                       2
LB          0 Hz
GB          0
PC                      1.00
```


8d
ppm
0.90
2.07
1.10
0.92
1.80
1.98
0.93
1.00
1.04
2.10
1.08
0.96
3.20


8d
ppm




```
Current Data Parameters
NAME        swk5173crude
EXPNO                  2
PROCNO                 1

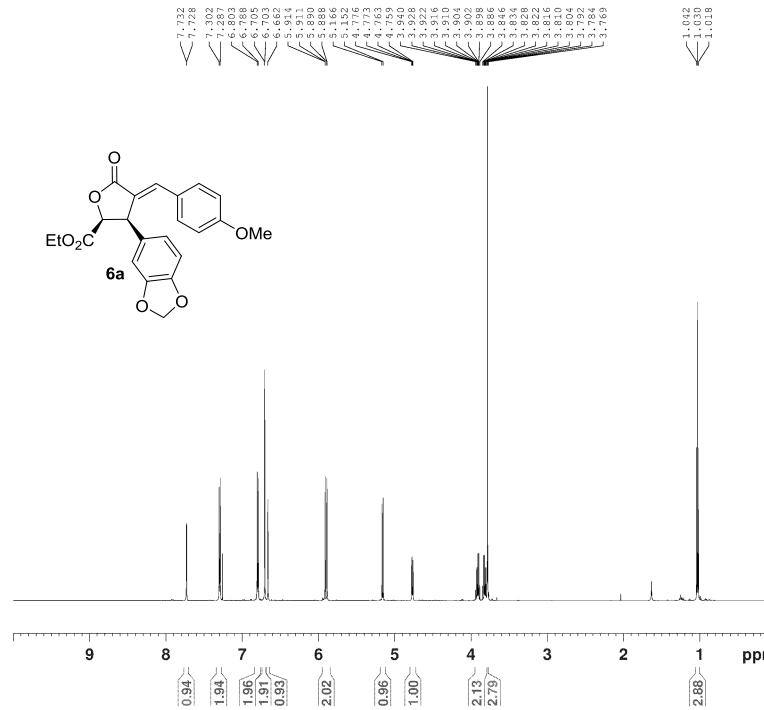
F2 - Acquisition Parameters
Date_           20141107
Time                0.08
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG     noesygpphpp
TD                  2048
SOLVENT             CDCl3
NS                     4
DS                    32
SWH             6009.615 Hz
FIDRES          2.934382 Hz
AQ              0.1703936 sec
RG                    64
DW                83.200 use
DE                10.00 use
TE                298.0 K
D0             0.00006665 sec
D1             2.00000000 sec
D8             0.30000001 sec
D11            0.03000000 sec
D12            0.00002000 sec
D16            0.00020000 sec
IN0            0.00016640 sec

======== CHANNEL f1 =======
SFO1          600.1327625 MHz
NUC1                   1H
P1                  13.00 use
P2                  26.00 use
P17               2500.00 use
PLW1         9.00000000 W
PLW10        2.25000000 W

====== GRADIENT CHANNEL ===
GPNAM[1]       SMSQ10.100
GPZ1                40.00 %
P16               1000.00 use

F1 - Acquisition parameters
TD                   256
SFO1          600.1328 MHz
FIDRES        23.475060 Hz
SW                10.014 ppm
FnMODE        States-TPPI

F2 - Processing parameters
SI                  1024
SF            600.1300104 MHz
WDW                 QSINE
SSB                     2
LB        0 Hz
GB        0
PC                  1.00
```

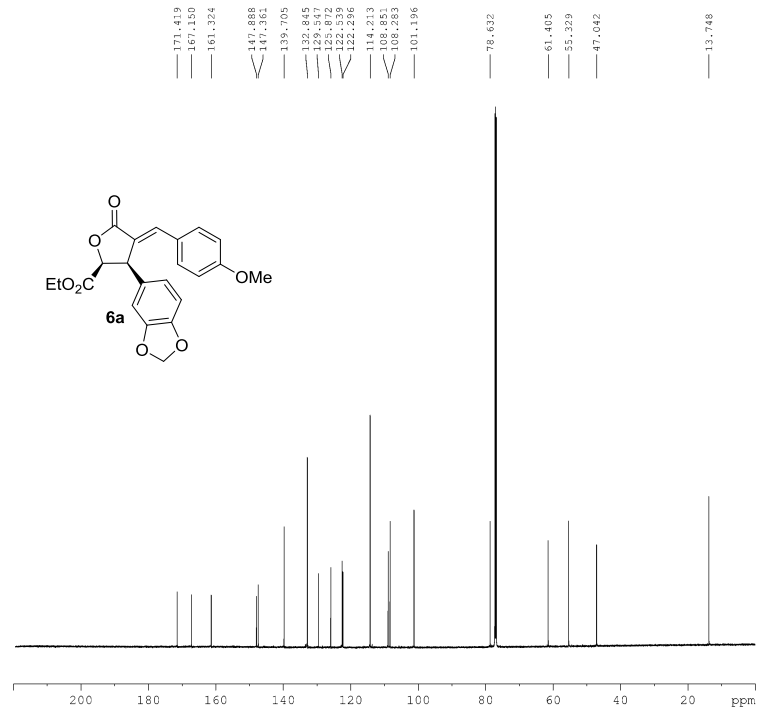



```
Current Data Parameters
NAME      swk5139fr113-165
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20140709
Time               21.02
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH            12019.230 Hz
FIDRES          0.183399 Hz
AQ              2.7262976 sec
RG                   12.7
DW                41.600 use
DE                10.00 use
TE                298.1 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 =======
SFO1          600.1337060 MHz
NUC1                   1H
P1                 12.10 use
PLW1         13.00000000 W

F2 - Processing parameters
SI                  65536
SF            600.1300138 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



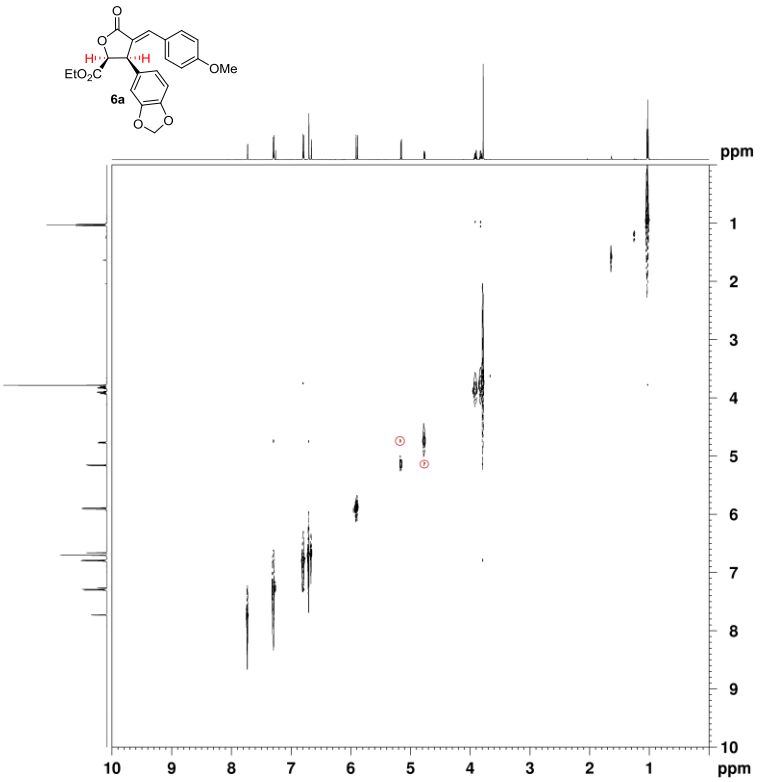

```
Current Data Parameters
NAME      swk5139fr113-165
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_              20140709
Time                 21.26
INSTRUM              spect
PROBHD   5 mm CPQCI 1H/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    512
DS                      4
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9087659 sec
RG                    203
DW               13.867 usec
DE                18.00 usec
TE                298.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1        150.9178981 MHz
NUC1                  13C
P1                 11.35 usec
PLW1       230.00000000 W

-------- CHANNEL f2 --------
SFO2        600.1324005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2        13.00000000 W
PLW12        0.23498000 W
PLW13        0.19033000 W

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      swk5139fr113-165
EXPNO                   3
PROCNO                  1

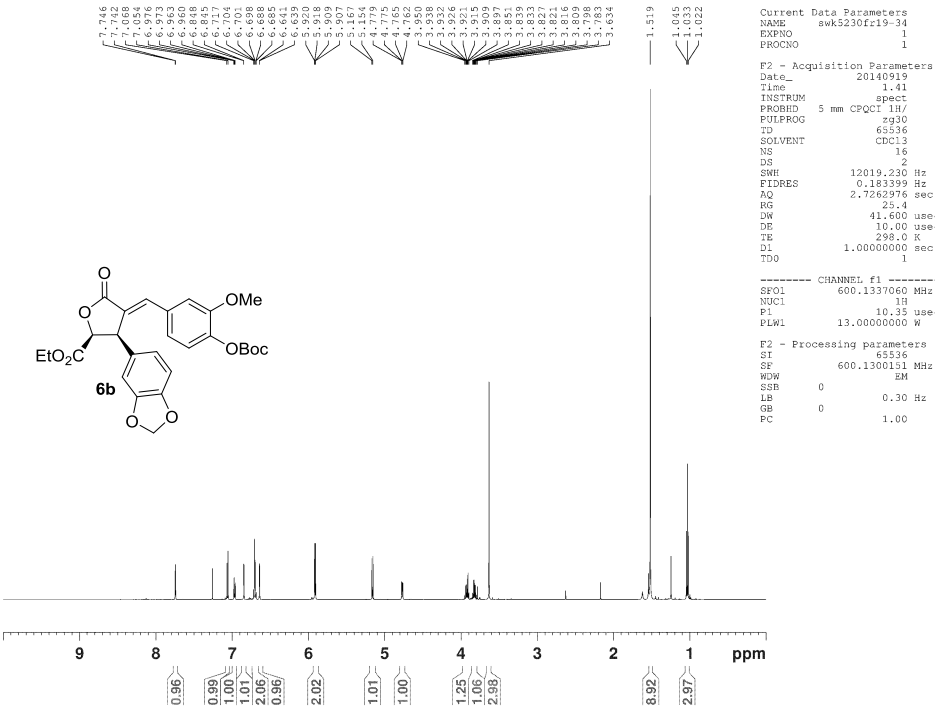
F2 - Acquisition Parameters
Date_              20140709
Time                 22.36
INSTRUM              spect
PROBHD   5 mm CPQCI 1H/
PULPROG      noesygpphpp
TD                   2048
SOLVENT             CDCl3
NS                      4
DS                     32
SWH            6009.615 Hz
FIDRES         2.934382 Hz
AQ            0.1703936 sec
RG                     64
DW               83.200 use
DE                10.00 use
TE                298.2 K
D0            0.00006779 sec
D1            2.00000000 sec
D8            0.30000001 sec
D11           0.03000000 sec
D12           0.00002000 sec
D16           0.00020000 sec
IN0           0.00016640 sec

======== CHANNEL f1 ========
SFO1        600.1327625 MHz
NUC1                  1H
P1                 12.10 use
P2                 24.20 use
P17              2500.00 use
PLW1        13.00000000 W
PLW10        2.81559992 W

====== GRADIENT CHANNEL ===
GPNAM[1]      SMSQ10.100
GPZ1                40.00 %
P16               1000.00 use

F1 - Acquisition parameters
TD                   256
SFO1           600.1328 MHz
FIDRES        23.475060 Hz
SW                10.014 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                  1024
SF           600.1300107 MHz
WDW                QSINE
SSB                    2
LB          0 Hz
GB          0
PC                  1.00
```

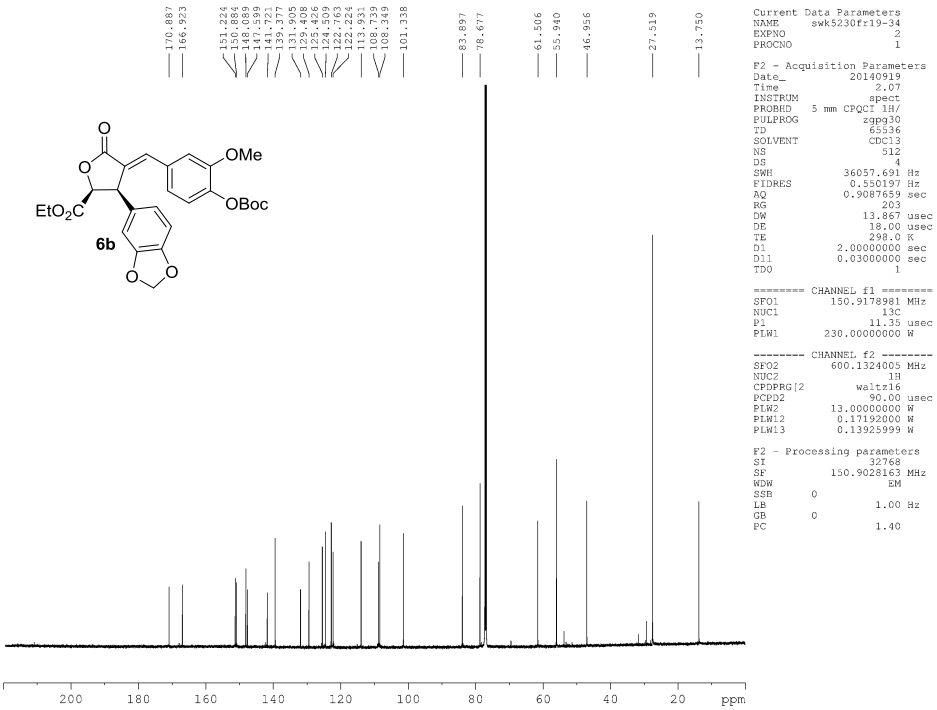



```
Current Data Parameters
NAME      swk5230fr19-34
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140919
Time                1.41
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           12019.230 Hz
FIDRES        0.183399 Hz
AQ            2.7262976 sec
RG                 25.4
DW                41.600 use
DE                10.00 use
TE                298.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         600.1337060 MHz
NUC1                   1H
P1                 10.35 use
PLW1        13.00000000 W

F2 - Processing parameters
SI                 65536
SF           600.1300151 MHz
WDW                   EM
SSB          0
LB                 0.30 Hz
GB           0
PC                 1.00
```



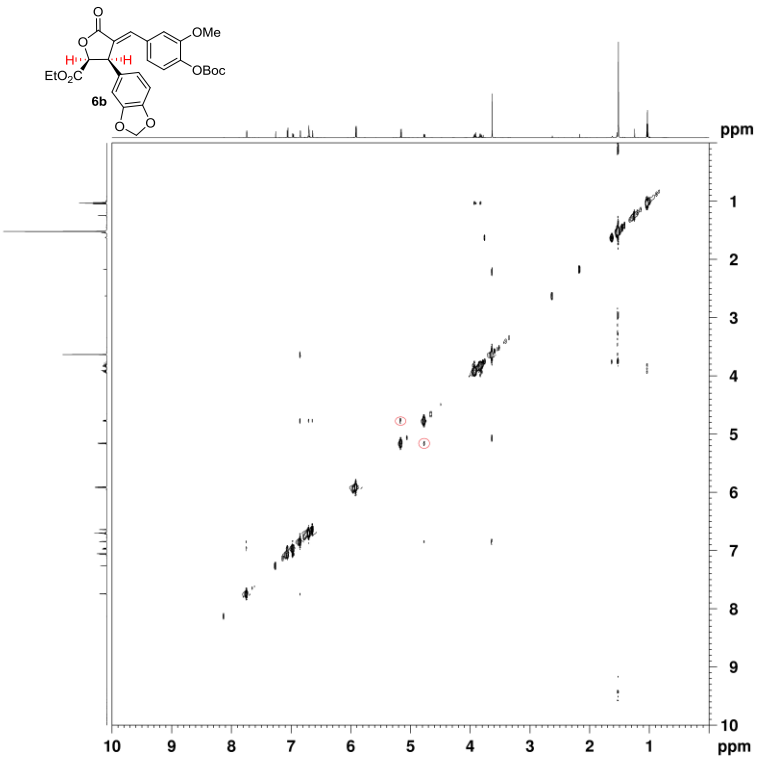

```
Current Data Parameters
NAME      swk5230fr19-34
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140919
Time                2.07
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           36057.691 Hz
FIDRES        0.550197 Hz
AQ            0.9087659 sec
RG                  203
DW                13.867 usec
DE                18.00 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         150.9178981 MHz
NUC1                  13C
P1                 11.35 usec
PLW1       230.00000000 W

======== CHANNEL f2 ========
SFO2         600.1324005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2        13.00000000 W
PLW12        0.17192000 W
PLW13        0.13925999 W

F2 - Processing parameters
SI                 32768
SF           150.9028163 MHz
WDW                   EM
SSB          0
LB                 1.00 Hz
GB           0
PC                 1.40
```




```
Current Data Parameters
NAME      swk5230fr19-34
EXPNO                 3
PROCNO                1

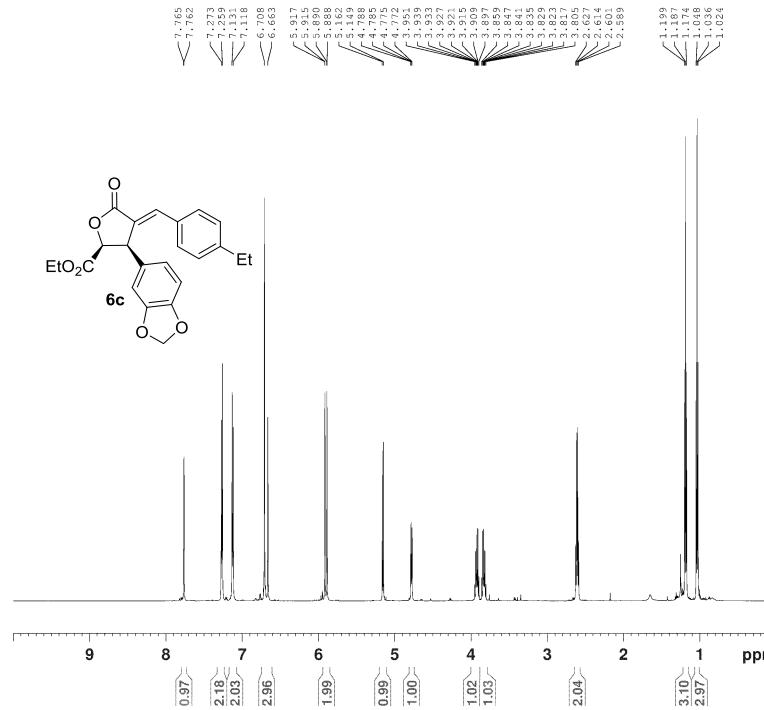
F2 - Acquisition Parameters
Date_          20140919
Time               2.09
INSTRUM           spect
PROBHD   5 mm CPQCI 1H/
PULPROG     noesygpphpp
TD                 2048
SOLVENT            CDCl3
NS                    4
DS                   32
SWH             6009.615 Hz
FIDRES          2.934382 Hz
AQ              0.1703936 sec
RG                   64
DW                83.200 use
DE                10.00 use
TE                298.0 K
D0             0.00007002 sec
D1             2.00000000 sec
D8             0.30000001 sec
D11            0.03000000 sec
D12            0.00002000 sec
D16            0.00020000 sec
IN0            0.00016640 sec

======== CHANNEL f1 =======
SFO1          600.1327625 MHz
NUC1               1H
P1                10.35 use
P2                20.70 use
P17             2500.00 use
PLW1          13.00000000 W
PLW10          2.05999994 W

====== GRADIENT CHANNEL ===
GPNAM[1]      SMSQ10.100
GPZ1              40.00 %
P16             1000.00 use

F1 - Acquisition parameters
TD                  256
SFO1          600.1328 MHz
FIDRES        23.475060 Hz
SW               10.014 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                 1024
SF            600.1300113 MHz
WDW                QSINE
SSB                    2
LB         0 Hz
GB         0
PC                 1.00
```

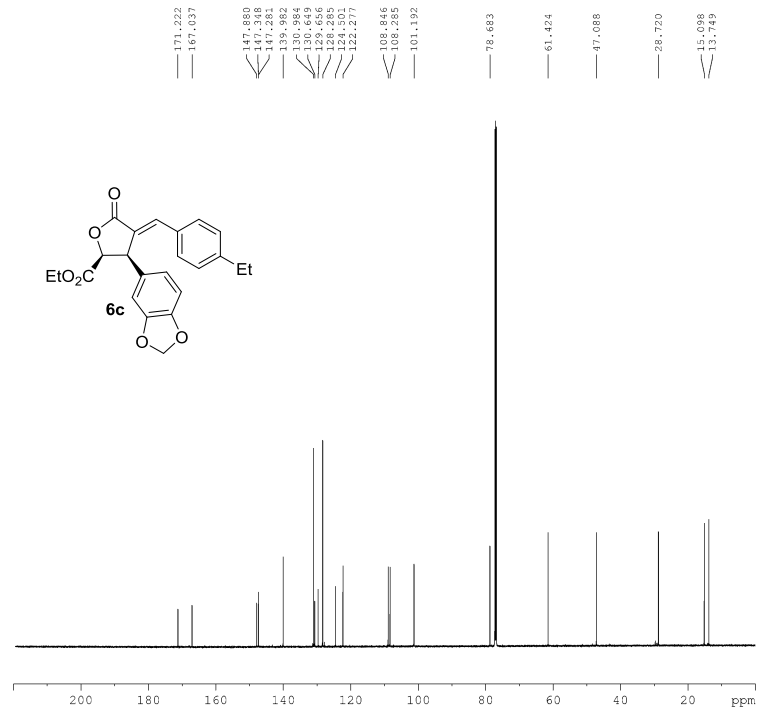



```
Current Data Parameters
NAME          swk5231col
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_          20141108
Time               8.37
INSTRUM           spect
PROBHD   5 mm CPQNP 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    7
DS                    0
SWH            12019.230 Hz
FIDRES          0.183399 Hz
AQ              2.7262976 sec
RG                   32
DW                41.600 use
DE                10.00 use
TE                298.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 =======
SFO1          600.1337060 MHz
NUC1               1H
P1                13.00 use
PLW1           9.00000000 W

F2 - Processing parameters
SI                65536
SF            600.1300132 MHz
WDW                   EM
SSB        0
LB                 0.30 Hz
GB         0
PC                 1.00
```



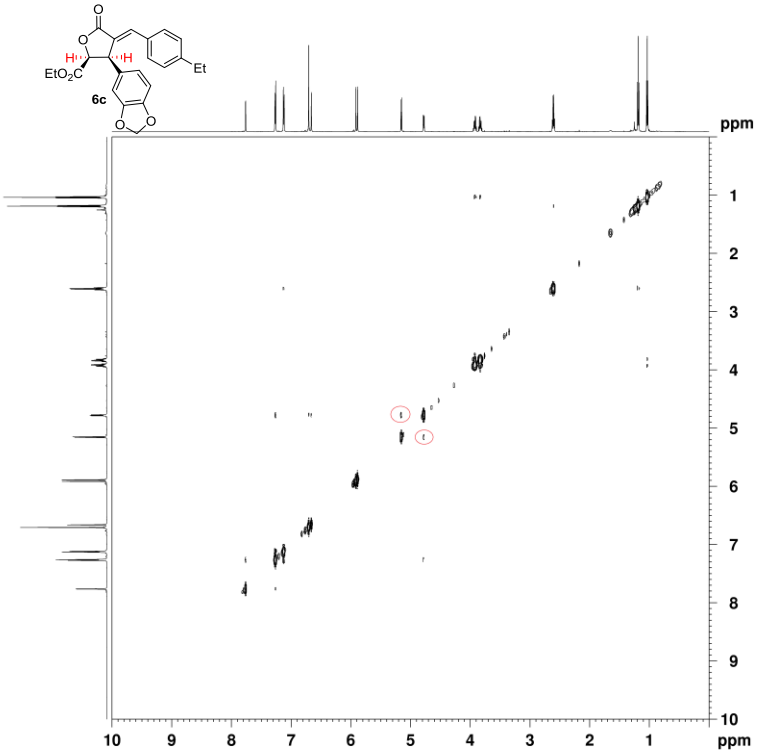

```
Current Data Parameters
NAME                 swk5231co1
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20141108
Time                       8.40
INSTRUM                   spect
PROBHD    5 mm CPQNP 1H/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                          36
DS                           0
SWH                  36057.691 Hz
FIDRES                0.550197 Hz
AQ                   0.9087659 sec
RG                         203
DW                      13.867 usec
DE                      18.00 usec
TE                      298.0 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               150.9178981 MHz
NUC1                       13C
P1                       11.93 usec
PLW1              30.00000000 W

-------- CHANNEL f2 --------
SFO2               600.1324005 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    80.00 usec
PLW2               9.00000000 W
PLW12              0.26587000 W
PLW13              0.17016000 W

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




```
Current Data Parameters
NAME                 swk5231co1
EXPNO                         3
PROCNO                        1

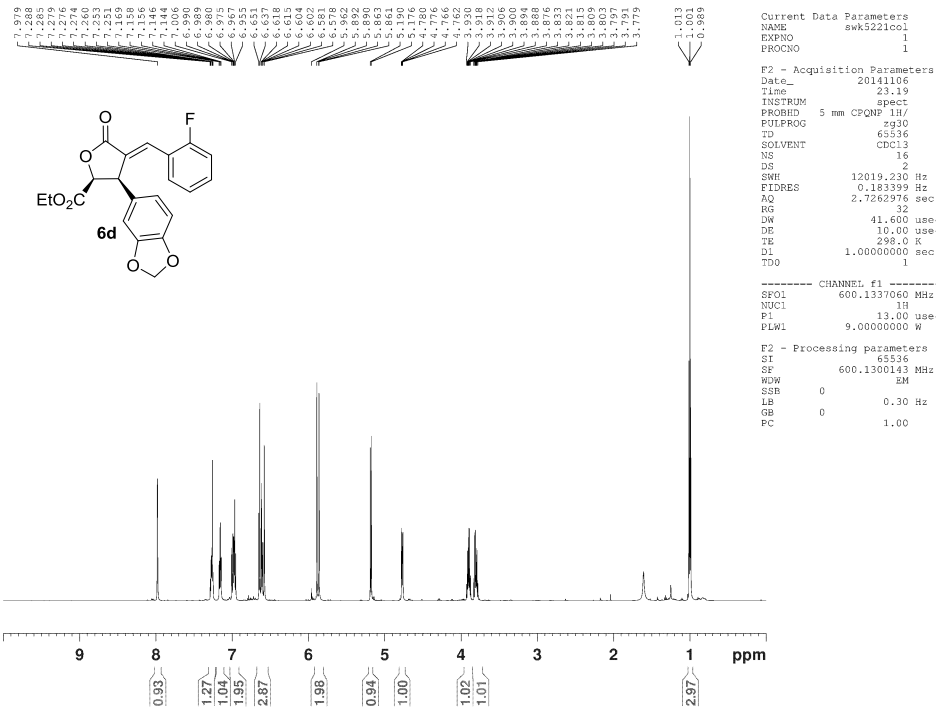
F2 - Acquisition Parameters
Date_                  20141108
Time                       8.45
INSTRUM                   spect
PROBHD    5 mm CPQNP 1H/
PULPROG          noesygpphpp
TD                        2048
SOLVENT                  CDCl3
NS                           4
DS                          32
SWH                  6009.615 Hz
FIDRES               2.934382 Hz
AQ                   0.1703936 sec
RG                          64
DW                      83.200 use
DE                      10.00 use
TE                      298.0 K
D0                   0.00006665 sec
D1                   2.00000000 sec
D8                   0.30000001 sec
D11                  0.03000000 sec
D12                  0.00002000 sec
D16                  0.00020000 sec
IN0                  0.00016640 sec

======== CHANNEL f1 ========
SFO1               600.1327625 MHz
NUC1                       1H
P1                       13.00 use
P2                       26.00 use
P17                    2500.00 use
PLW1               9.00000000 W
PLW10              2.25000000 W

====== GRADIENT CHANNEL ====
GPNAM[1]         SMSQ10.100
GPZ1                    40.00 %
P16                   1000.00 use

F1 - Acquisition parameters
TD                         220
SFO1                600.1328 MHz
FIDRES              27.316433 Hz
SW                      10.014 ppm
FnMODE           States-TPPI

F2 - Processing parameters
SI                        1024
SF                 600.1300135 MHz
WDW                      QSINE
SSB                          2
LB                0 Hz
GB                0
PC                       1.00
```

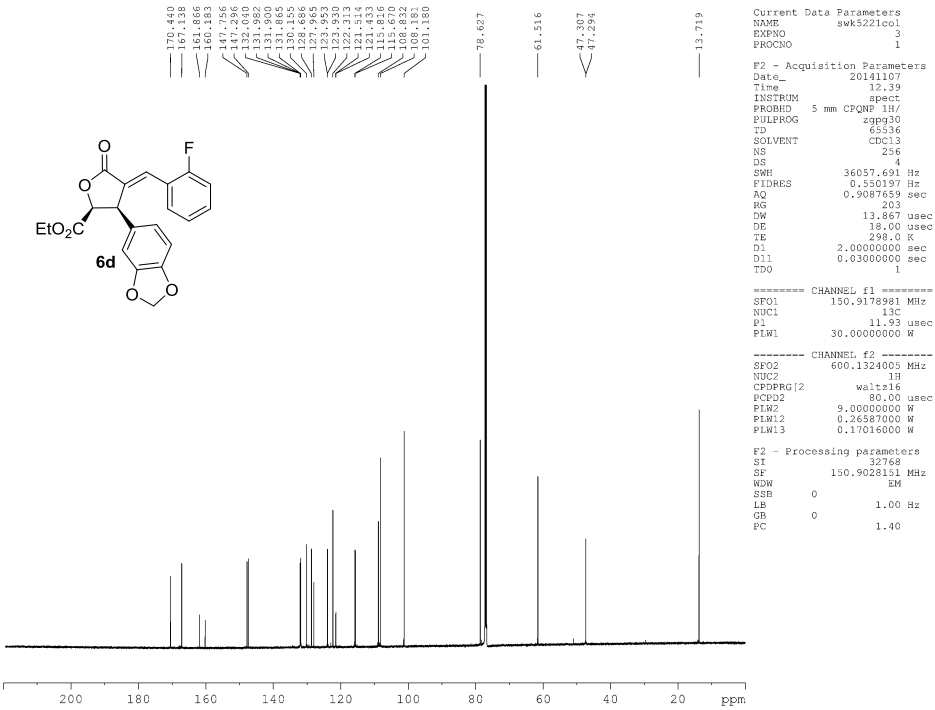



```
Current Data Parameters
NAME                  swk5221col
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_                 20141106
Time                      23.19
INSTRUM                   spect
PROBHD    5 mm CPQNP 1H/
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                          16
DS                           2
SWH                  12019.230 Hz
FIDRES                0.183399 Hz
AQ                    2.7262976 sec
RG                          32
DW                       41.600 use
DE                       10.00 use
TE                       298.0 K
D1                  1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                600.1337060 MHz
NUC1                        1H
P1                       13.00 use
PLW1                9.00000000 W

F2 - Processing parameters
SI                       65536
SF                  600.1300143 MHz
WDW                         EM
SSB                  0
LB                        0.30 Hz
GB                   0
PC                        1.00
```



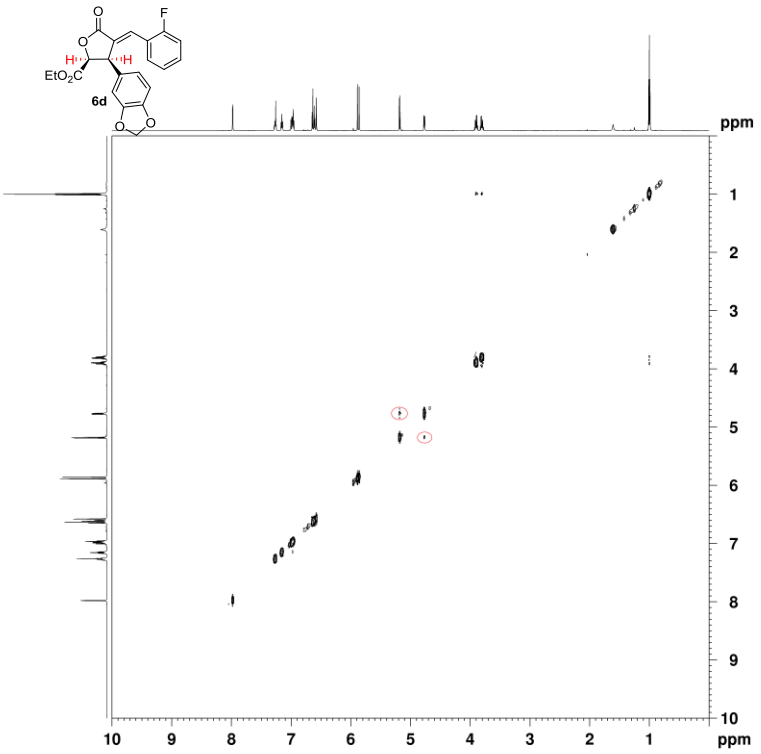

```
Current Data Parameters
NAME                  swk5221col
EXPNO                          3
PROCNO                         1

F2 - Acquisition Parameters
Date_                 20141107
Time                      12.39
INSTRUM                   spect
PROBHD    5 mm CPQNP 1H/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         256
DS                           4
SWH                  36057.691 Hz
FIDRES                0.550197 Hz
AQ                    0.9087659 sec
RG                         203
DW                       13.867 usec
DE                       18.00 usec
TE                       298.0 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                150.9178981 MHz
NUC1                        13C
P1                       11.93 usec
PLW1               30.00000000 W

======== CHANNEL f2 ========
SFO2                600.1324005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    80.00 usec
PLW2                9.00000000 W
PLW12               0.26587000 W
PLW13               0.17016000 W

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                 swk5221co1
EXPNO                         2
PROCNO                        1

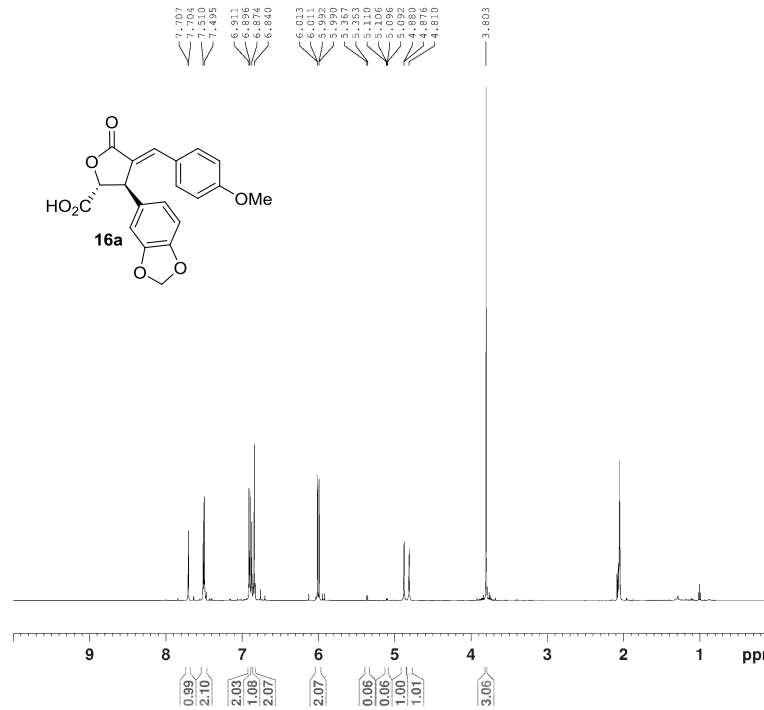
F2 - Acquisition Parameters
Date_               20141106
Time                   23.21
INSTRUM                spect
PROBHD    5 mm CPQNP 1H/
PULPROG          noesygpphpp
TD                       2048
SOLVENT                 CDCl3
NS                          4
DS                         32
SWH                6009.615 Hz
FIDRES             2.934382 Hz
AQ                 0.1703936 sec
RG                       203
DW                    83.200 use
DE                    10.00 use
TE                    298.0 K
D0                 0.00006665 sec
D1                 2.00000000 sec
D8                 0.30000001 sec
D11                0.03000000 sec
D12                0.00002000 sec
D16                0.00020000 sec
IN0                0.00016640 sec

======== CHANNEL f1 =======
SFO1              600.1327625 MHz
NUC1                       1H
P1                      13.00 use
P2                      26.00 use
P17                   2500.00 use
PLW1               9.00000000 W
PLW10              2.25000000 W

====== GRADIENT CHANNEL ===
GPNAM[1]          SMSQ10.100
GPZ1                   40.00 %
P16                  1000.00 use

F1 - Acquisition parameters
TD                        256
SFO1                600.1328 MHz
FIDRES             23.475060 Hz
SW                     10.014 ppm
FnMODE           States-TPPI

F2 - Processing parameters
SI                       1024
SF                600.1300151 MHz
WDW                     QSINE
SSB                         2
LB          0 Hz
GB          0
PC                       1.00
```

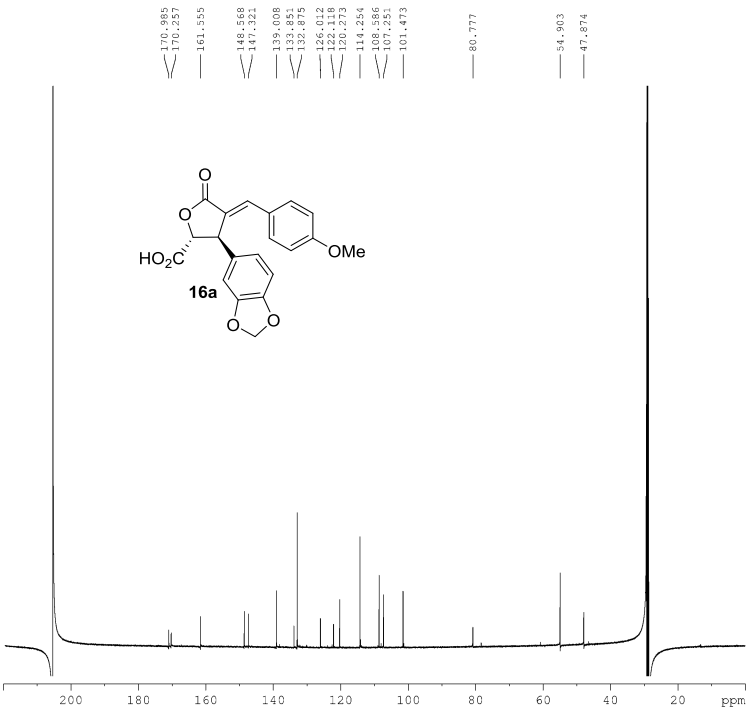



```
Current Data Parameters
NAME                 swk5144wash
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_               20140919
Time                    4.10
INSTRUM                spect
PROBHD    5 mm CPQCI 1H/
PULPROG                 zg30
TD                     65536
SOLVENT              Acetone
NS                          16
DS                           2
SWH               12019.230 Hz
FIDRES             0.183399 Hz
AQ                 2.7262976 sec
RG                        25.4
DW                    41.600 use
DE                    10.00 use
TE                    298.0 K
D1                 1.00000000 sec
TD0                          1

======== CHANNEL f1 =======
SFO1              600.1337060 MHz
NUC1                       1H
P1                      10.35 use
PLW1              13.00000000 W

F2 - Processing parameters
SI                       65536
SF                600.1300104 MHz
WDW                        EM
SSB         0
LB                      0.30 Hz
GB          0
PC                       1.00
```



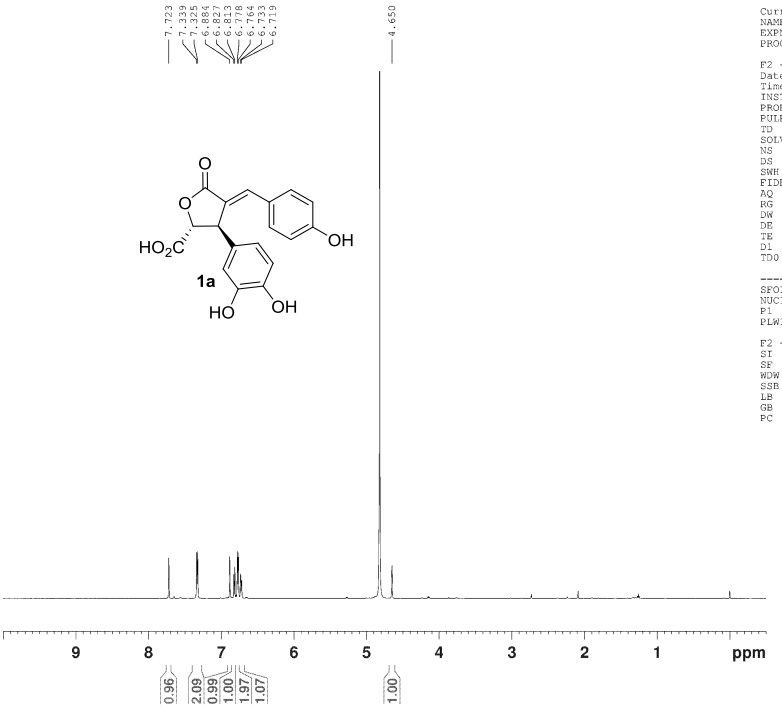

```
Current Data Parameters
NAME          swk5144wash
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_              20140919
Time                   4.36
INSTRUM               spect
PROBHD   5 mm CPQCI 1H/
PULPROG             zgpg30
TD                   65536
SOLVENT            Acetone
NS                       512
DS                         4
SWH              36057.691 Hz
FIDRES            0.550197 Hz
AQ                0.9087659 sec
RG                       203
DW                  13.867 usec
DE                   18.00 usec
TE                   298.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1          150.9178981 MHz
NUC1                    13C
P1                   11.35 usec
PLW1        230.00000000 W

-------- CHANNEL f2 --------
SFO2          600.1324005 MHz
NUC2                    1H
CPDPRG[2             waltz16
PCPD2                90.00 usec
PLW2          13.00000000 W
PLW12          0.17192000 W
PLW13          0.13925999 W

F2 - Processing parameters
SI                     32768
SF            150.9028090 MHz
WDW                       EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

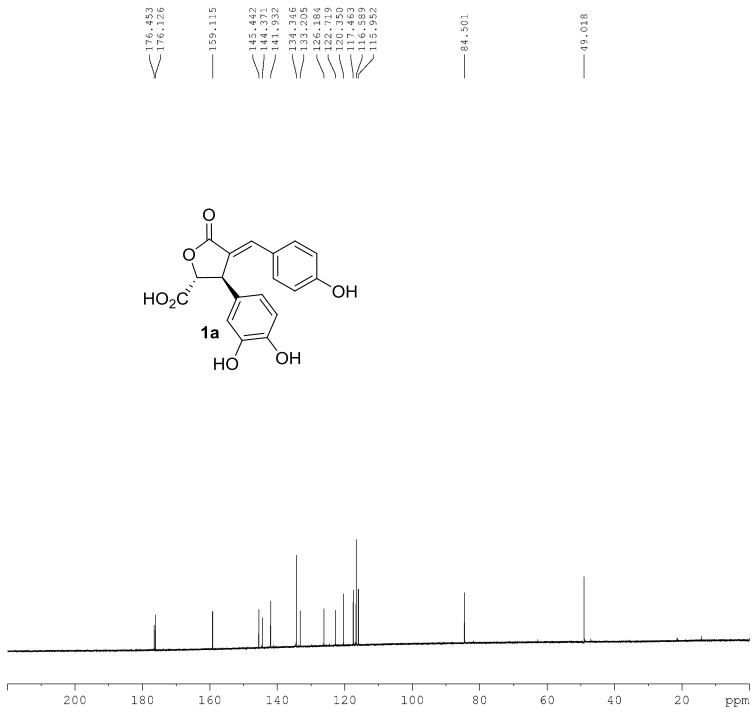



```
Current Data Parameters
NAME          swk5292HPLC
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_              20141219
Time                   0.01
INSTRUM               spect
PROBHD   5 mm CPQNP 1H/
PULPROG               zg30
TD                   65536
SOLVENT                D2O
NS                        16
DS                         2
SWH              12019.230 Hz
FIDRES            0.183399 Hz
AQ                2.7262976 sec
RG                     71.8
DW                  41.600 use
DE                   10.00 use
TE                   298.1 K
D1                1.00000000 sec
TD0                        1

-------- CHANNEL f1 --------
SFO1          600.1337060 MHz
NUC1                    1H
P1                   13.00 use
PLW1          9.00000000 W

F2 - Processing parameters
SI                     65536
SF            600.1299301 MHz
WDW                       EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```



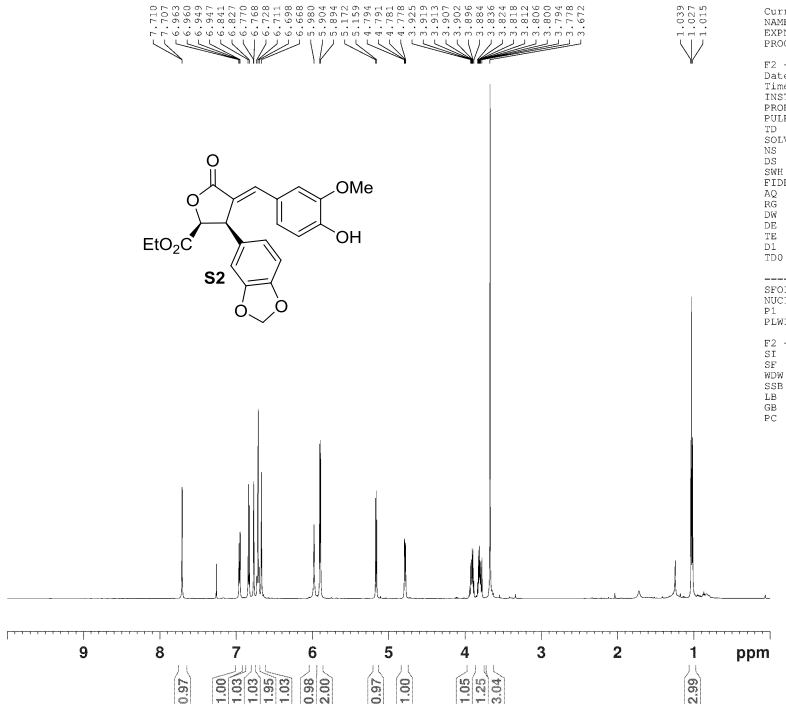

```
Current Data Parameters
NAME          swk5292HPLC
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_              20141219
Time                   2.57
INSTRUM               spect
PROBHD   5 mm CPQNP 1H/
PULPROG             zgpg30
TD                   65536
SOLVENT                D2O
NS                    3500
DS                       4
SWH            36057.691 Hz
FIDRES          0.550197 Hz
AQ             0.9087659 sec
RG                     203
DW                13.867 usec
DE                 18.00 usec
TE                 298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         150.9178981 MHz
NUC1                  13C
P1                 11.00 usec
PLW1         30.00000000 W

-------- CHANNEL f2 --------
SFO2         600.1324005 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2              80.00 usec
PLW2          9.00000000 W
PLW12         0.26587000 W
PLW13         0.17016000 W

F2 - Processing parameters
SI                    32768
SF           150.9026548 MHz
WDW                     EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

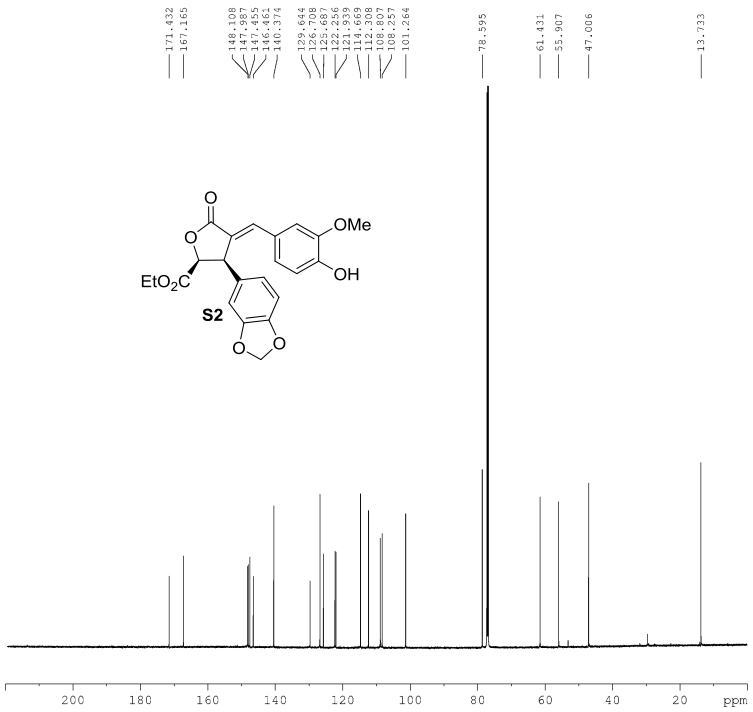



```
Current Data Parameters
NAME          swk5251col
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_              20140930
Time                  21.05
INSTRUM               spect
PROBHD   5 mm CPQCI 1H/
PULPROG                zg30
TD                   65536
SOLVENT              CDCl3
NS                       6
DS                       0
SWH            12019.230 Hz
FIDRES          0.183399 Hz
AQ             2.7262976 sec
RG                    12.7
DW                41.600 use
DE                10.00 use
TE                298.0 K
D1            1.00000000 sec
TD0                     1

-------- CHANNEL f1 --------
SFO1         600.1337060 MHz
NUC1                   1H
P1                10.35 use
PLW1         13.00000000 W

F2 - Processing parameters
SI                    65536
SF           600.1300151 MHz
WDW                     EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



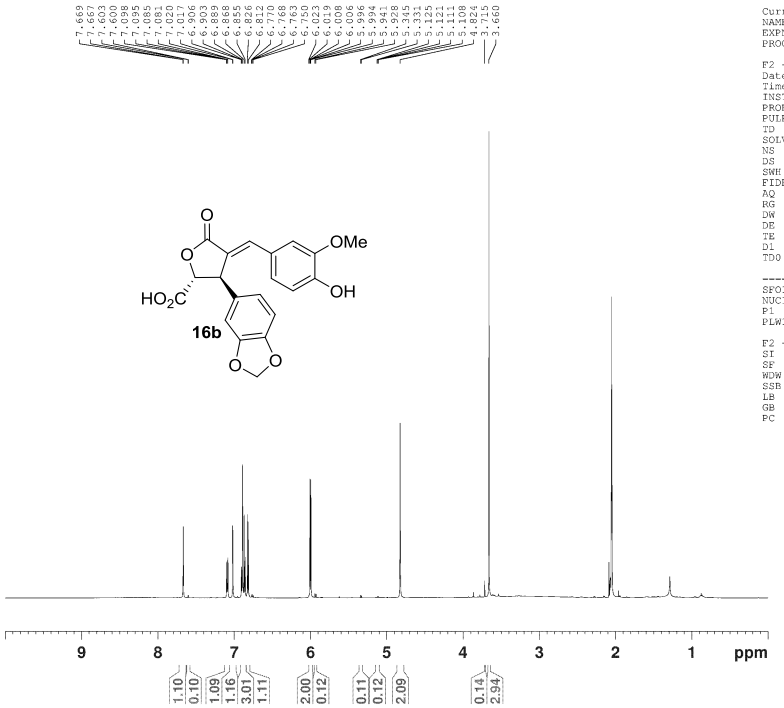

```
Current Data Parameters
NAME          swk5251co1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140930
Time               21.13
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  516
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9087659 sec
RG                  203
DW               13.867 usec
DE               18.00 usec
TE               298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        150.9178981 MHz
NUC1               13C
P1               11.35 usec
PLW1       230.00000000 W

-------- CHANNEL f2 --------
SFO2        600.1324005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        13.00000000 W
PLW12        0.17192000 W
PLW13        0.13925999 W

F2 - Processing parameters
SI                 32768
SF          150.9028187 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

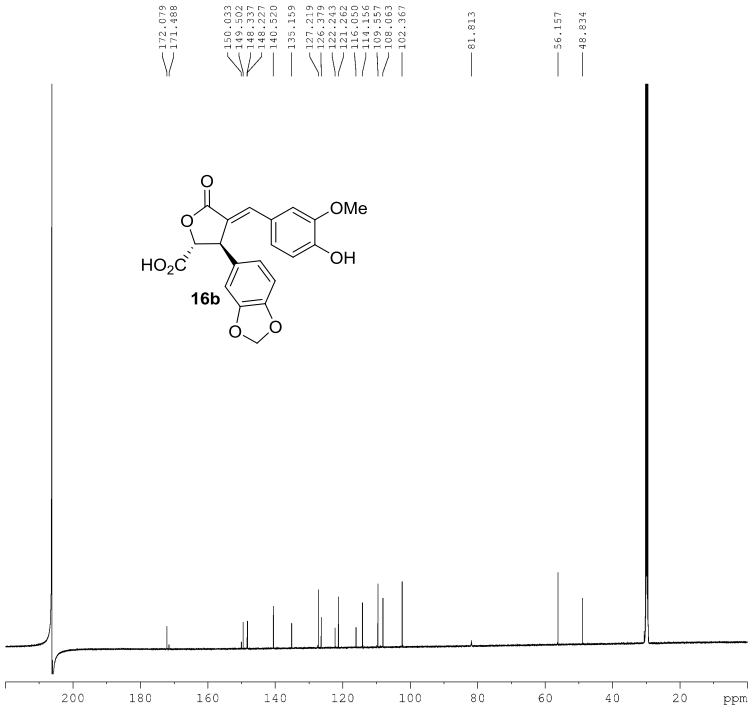



```
Current Data Parameters
NAME      swk5286recovered
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20141202
Time               15.54
INSTRUM            spect
PROBHD   5 mm CPQNP 1H/
PULPROG             zg30
TD                65536
SOLVENT         Acetone
NS                   16
DS                    2
SWH           12019.230 Hz
FIDRES         0.183399 Hz
AQ            2.7262976 sec
RG                   57
DW               41.600 use
DE               10.00 use
TE               298.0 K
D1            1.00000000 sec
TD0                    1

-------- CHANNEL f1 --------
SFO1        600.1337060 MHz
NUC1               1H
P1               13.00 use
PLW1        9.00000000 W

F2 - Processing parameters
SI                 65536
SF          600.1300102 MHz
WDW                EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



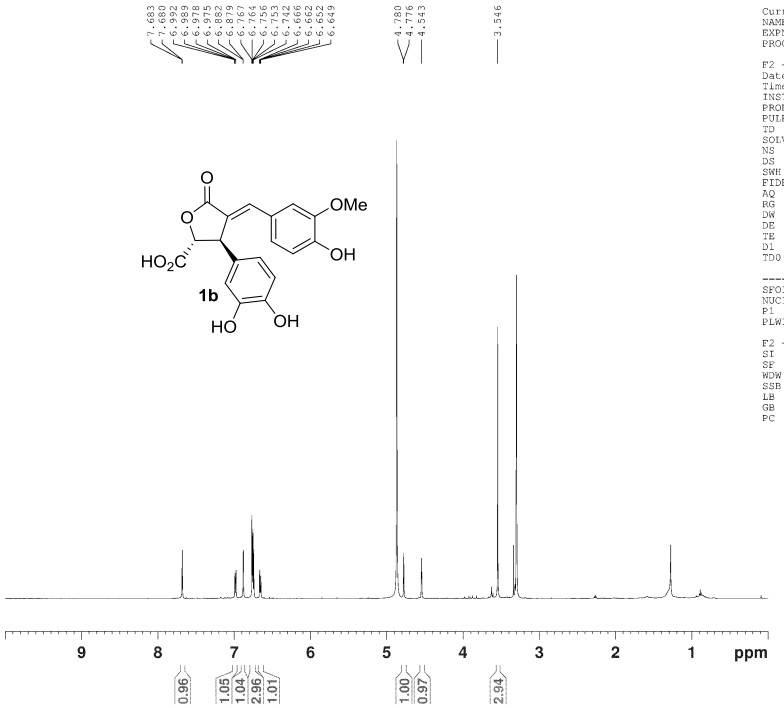

```
Current Data Parameters
NAME      swk5286recovered
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_              20141202
Time                  15.58
INSTRUM               spect
PROBHD   5 mm CPQNP 1H/
PULPROG           zgpg30
TD                 65536
SOLVENT          Acetone
NS                    1024
DS                       0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9087659 sec
RG                     203
DW                13.867 usec
DE                 18.00 usec
TE                 298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1        150.9178981 MHz
NUC1                   13C
P1                   11.93 usec
PLW1        30.00000000 W

-------- CHANNEL f2 --------
SFO2        600.1324005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2          9.00000000 W
PLW12         0.26587000 W
PLW13         0.17016000 W

F2 - Processing parameters
SI                   32768
SF          150.9026735 MHz
WDW                     EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

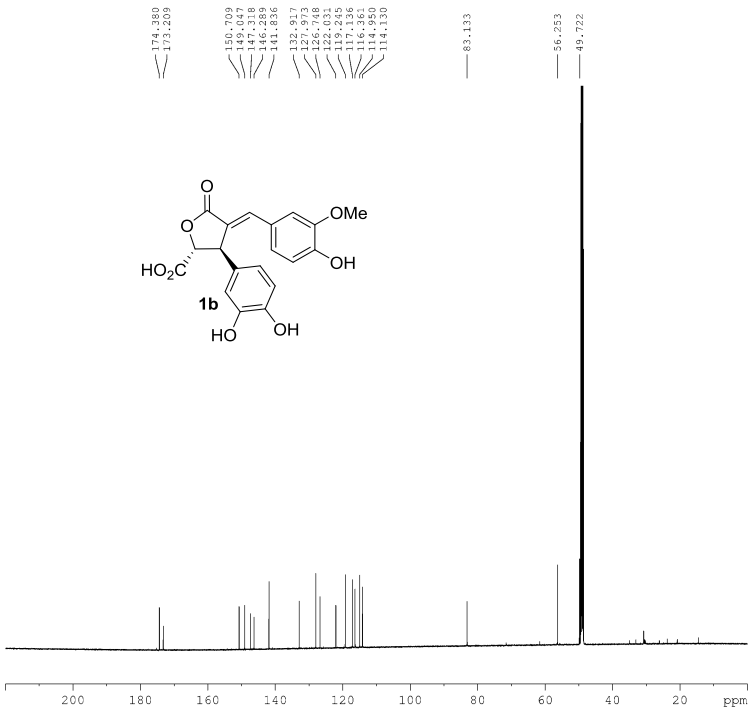



```
Current Data Parameters
NAME         swk5295HPLC
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_              20141218
Time                  21.01
INSTRUM               spect
PROBHD   5 mm CPQNP 1H/
PULPROG               zg30
TD                 65536
SOLVENT             MeOD
NS                    16
DS                     2
SWH           12019.230 Hz
FIDRES         0.183399 Hz
AQ            2.7262976 sec
RG                  71.8
DW                41.600 use
DE                 10.00 use
TE                 298.1 K
D1            1.00000000 sec
TD0                     1

-------- CHANNEL f1 --------
SFO1        600.1337060 MHz
NUC1                   1H
P1                  13.00 use
PLW1          9.00000000 W

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      swk5295HPLCextract2
EXPNO                       2
PROCNO                      1

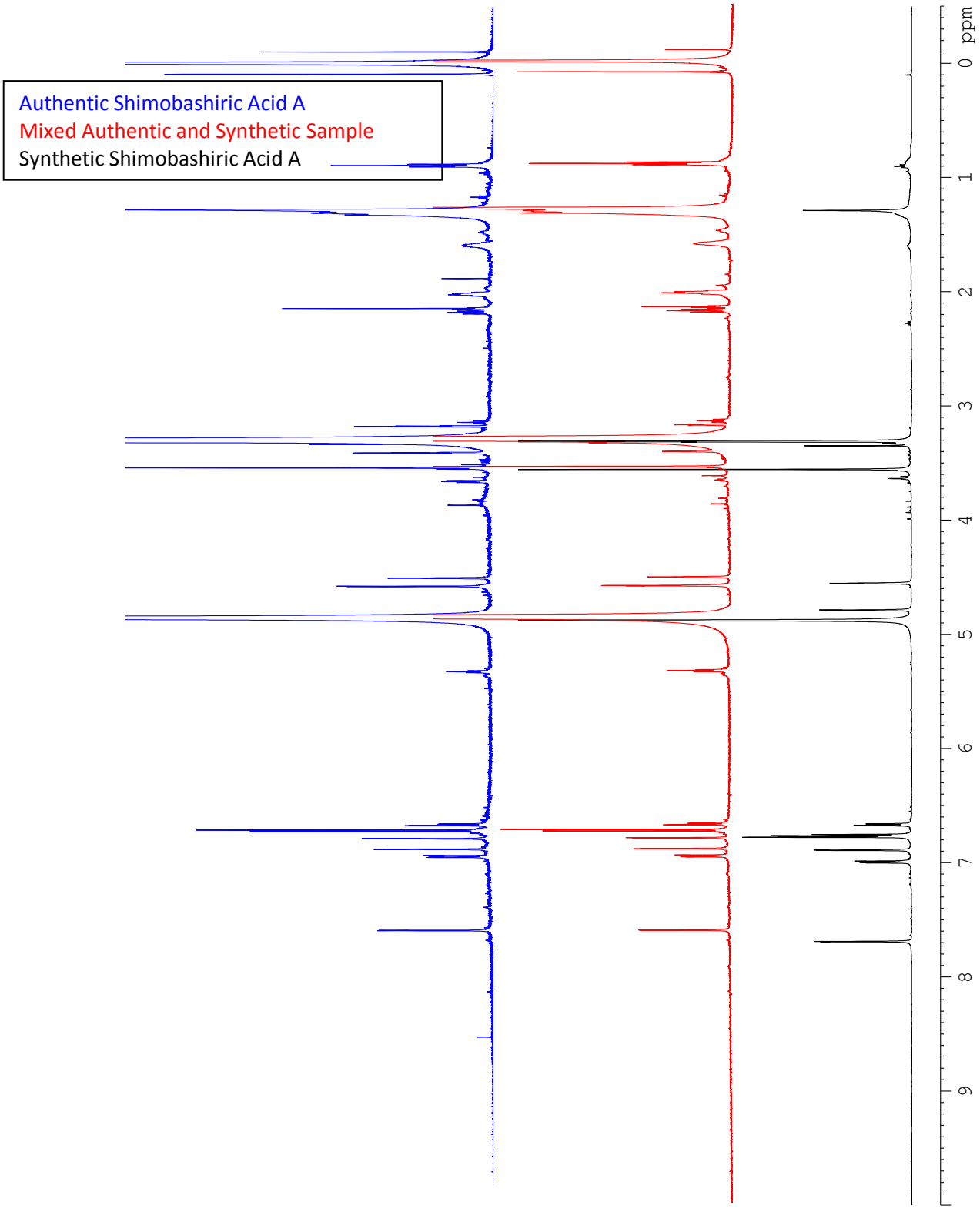
F2 - Acquisition Parameters
Date_              20141215
Time                  22.56
INSTRUM               spect
PROBHD   5 mm CPQNP 1H/
PULPROG             zgpg30
TD                   65536
SOLVENT               MeOD
NS                    2372
DS                       4
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9087659 sec
RG                     203
DW                13.867 usec
DE                 18.00 usec
TE                 298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1        150.9178981 MHz
NUC1                  13C
P1                  11.93 usec
PLW1        30.00000000 W

-------- CHANNEL f2 --------
SFO2        600.1324005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              80.00 usec
PLW2         9.00000000 W
PLW12        0.26587000 W
PLW13        0.17016000 W

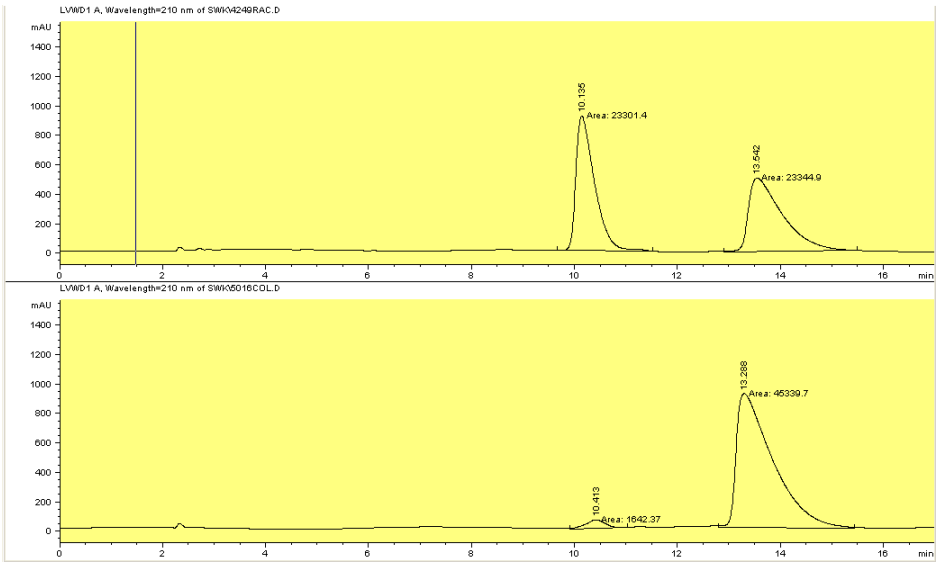
F2 - Processing parameters
SI                   32768
SF          150.9025970 MHz
WDW                     EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```

Comparison of Authentic Shimobashiric Acid A to Synthetic Sample



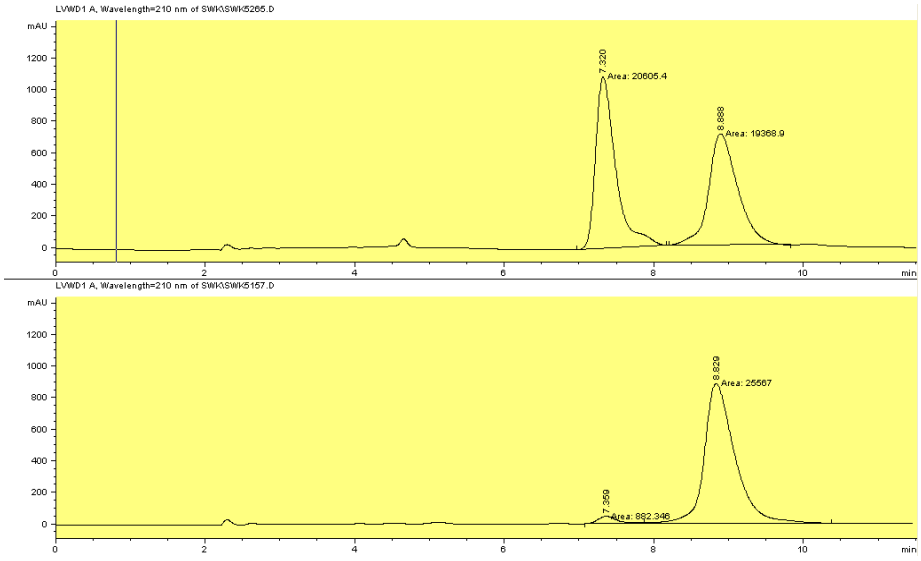
SFC Traces

SFC Analysis of 14a



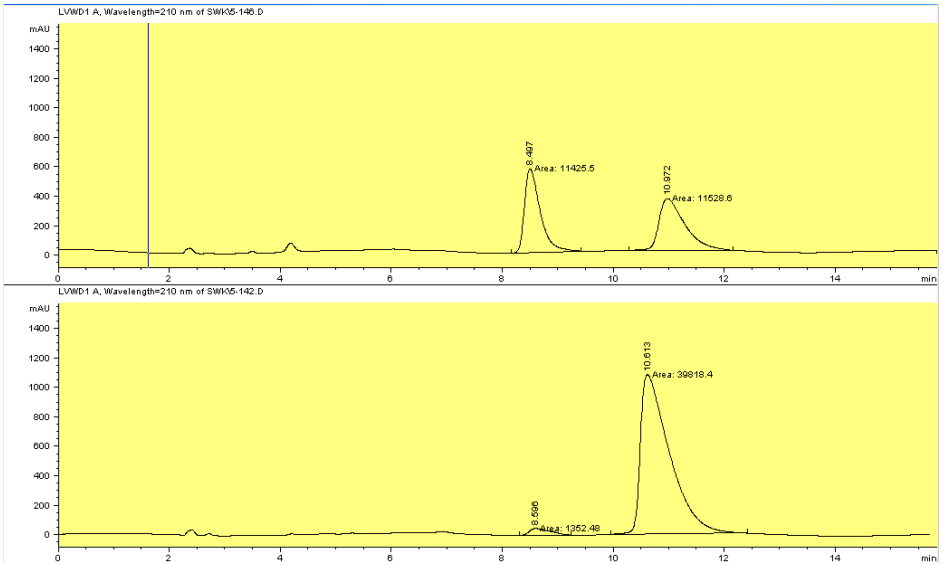
Peak #	RT [min]	Area (mAU*sec)	Area [%]
1	10.1	1642.37	3.5
2	13.3	45339.7	96.5

SFC Analysis of 14b



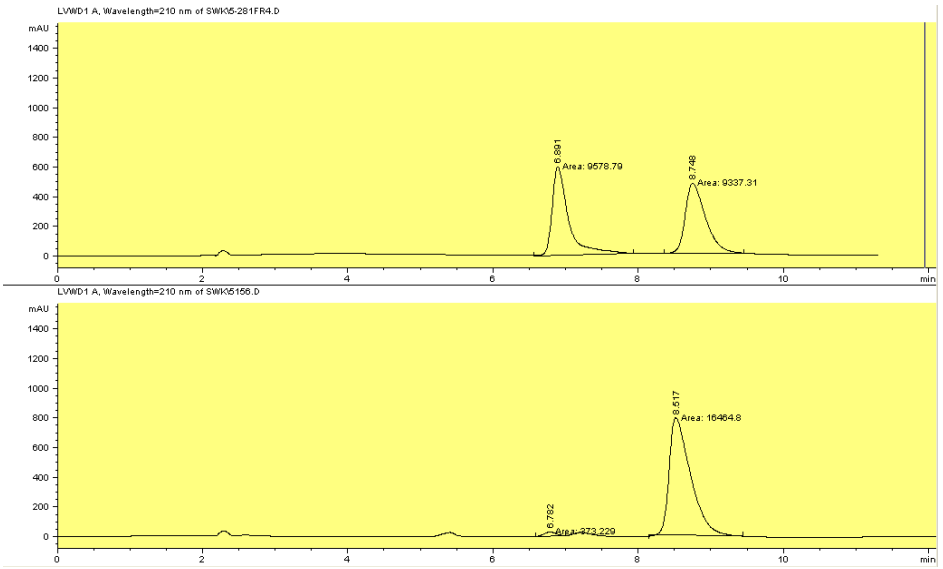
Peak #	RT [min]	Area (mAU*sec)	Area [%]
1	7.4	882.346	3.3
2	8.8	25567.0	96.7

SFC Analysis of 14c



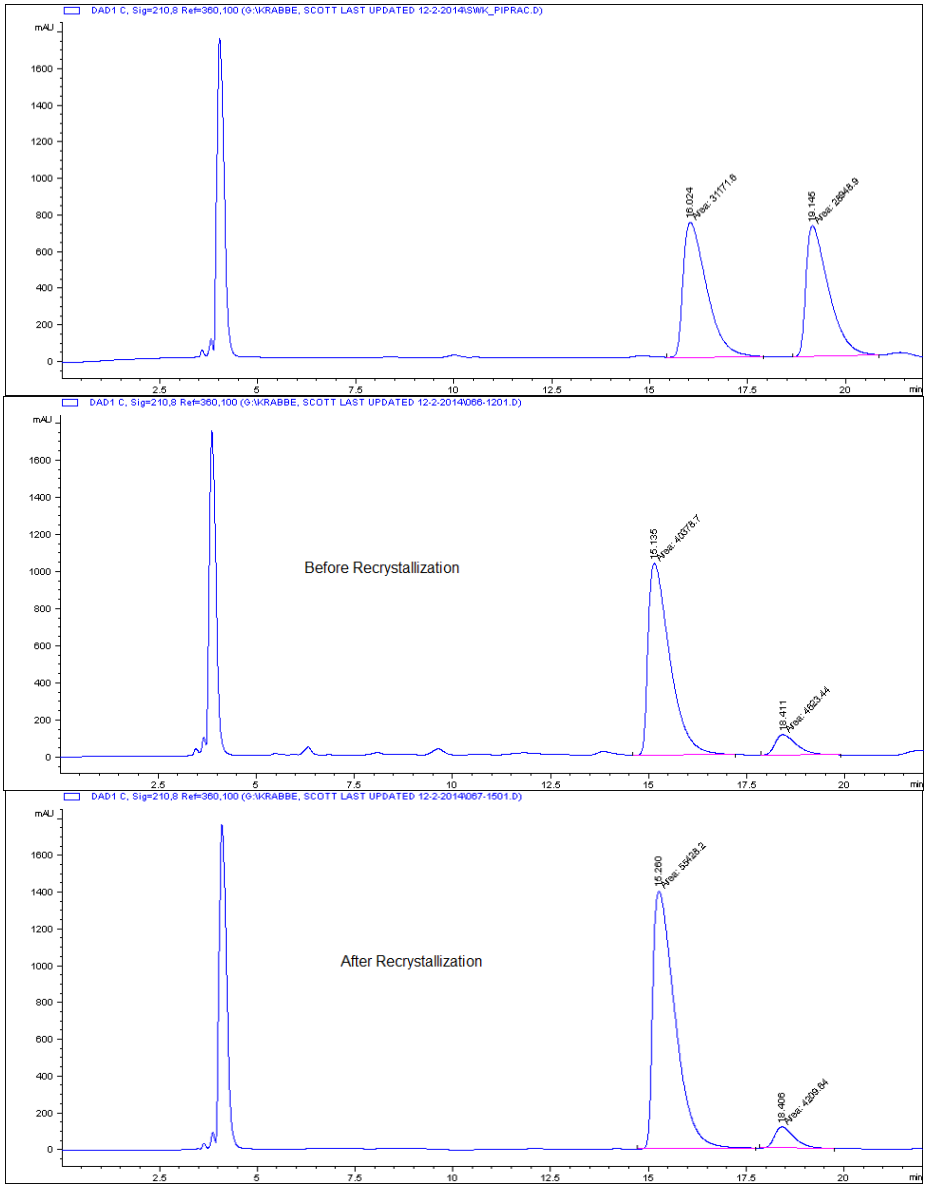
Peak #	RT [min]	Area (mAU*sec)	Area [%]
1	8.6	1352.48	3.3
2	10.6	39818.4	96.7

SFC Analysis of 14d



Peak #	RT [min]	Area (mAU*sec)	Area [%]
1	6.8	373.229	2.2
2	8.5	16464.8	97.8

HPLC Analysis of 4



Before Recrystallization			
Peak #	**RT [min]**	**Area (mAU*sec)**	**Area [%]**
1	15.1	40378.7	89.7
2	18.4	4623.44	10.3
After Recrystallization			
Peak #	**RT [min]**	**Area (mAU*sec)**	**Area [%]**
1	15.3	55428.2	92.9
2	18.4	4209.64	7.1