SUPPORTING INFORMATION

Enantioselective Synthesis of Hindered Cyclic Dialkyl Ethers via Catalytic Oxa-Michael/Michael Desymmetrization

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Table of Contents

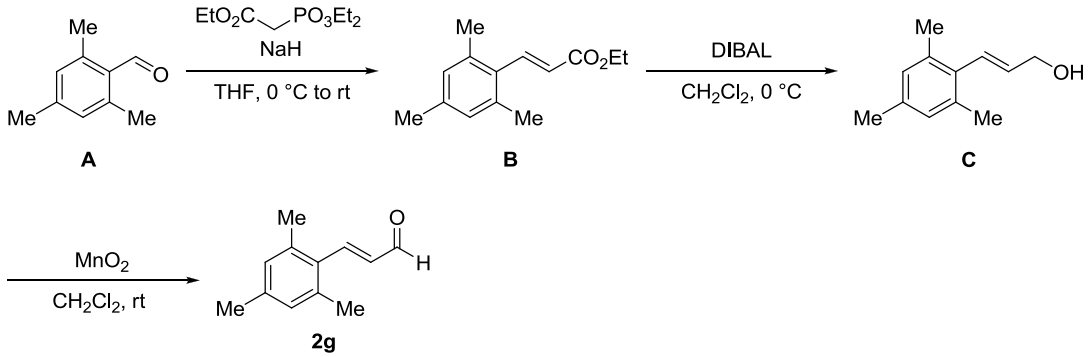
	Page
General Information (Methods and Materials)	S2
Synthesis of (*E*)-3-Mesitylacrylaldehyde (**2g**)	S3
General Procedure A for the Preparation of rac-**4**	S4
General Procedure B for the Preparation of **4**	S4-S9
Gram Scale Synthesis of **4d**	S9-S10
General Procedure C for the Reduction of Aldehydes **4** to Alcohols **5**	S10-S13
General Procedure D for the Conversion of **4h** and **4j** to **S1a** and **S1b**	S14-S15
Synthesis of Benzoate **6** from **5a**	S15-S16
Synthesis of Epoxide **7** from **4d**	S16
Synthesis of Diester **8** from **4n**	S16-S17
Synthesis of bis(Tetrahydrofuran) **9** from **4m**	S17
Synthesis of Tricycle **10** from **4d**	S18
Deuterium Labeling Experiment and Structure Determination of **10**	S18-S19
References	S20
^{1}H, ^{13}C, and NOESY NMR Spectra	S21-S56
HPLC/SFC Traces	S57-S70

General Information

Methods: Infrared (IR) spectra were obtained using a Jasco 260 Plus Fourier transform infrared spectrometer. Proton and carbon magnetic resonance spectra (^{1}H NMR, ^{13}C NMR, and ^{19}F NMR) were recorded on a Bruker model DRX 400 or 600 (^{1}H NMR at 400 MHz or 600 MHz, ^{13}C NMR at 101 MHz or 151 MHz, and ^{19}F NMR at 565 MHz) spectrometer with solvent resonance as the internal standard (^{1}H NMR: $CDCl_3$ at 7.26 ppm and ^{13}C NMR: $CDCl_3$ at 77.0 ppm). ^{1}H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, bs = broad singlet, d = doublet, dd = doublet of doublet, t = triplet, q = quartet, sept = septuplet, oct = octuplet, m = multiplet), coupling constants (Hz), and integration. HPLC analysis was performed on an Agilent Technologies 1200 System equipped with Chiralpak IA, IB, and IC columns (φ 4.6 mm x 250 mm, constant flow at 1.00 mL/min). Supercritical fluid chromatography (SFC) was performed on a Berger SFC system equipped with Chiralpak AD, AS, and OD (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. 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. Analytical thin layer chromatography (TLC) was performed on Sorbtech 0.25 mm silica gel 60 plates. Visualization was accomplished with UV light and/or aqueous ceric ammonium molybdate solution followed by heating. Purification of the reaction products was carried out by using Siliaflash-P60 silica gel (40-63 μm) purchased from Silicycle. All reactions were carried out with magnetic stirring. Yield refers to isolated yield of analytically pure material unless otherwise noted. Yields and diastereomeric ratios (dr) are reported for a specific experiment and as a result may differ slightly from those found in the tables, which are averages of at least two experiments.

Materials: *p*-Quinols **1**,[1,2] α,β-unsaturated aldehydes **2**,[3] and Jørgensen–Hayashi catalysts **3**[4] were prepared according to known literature procedures. Triethylamine (Et_3N) was freshly distilled from calcium hydride prior to use. Dichloromethane (CH_2Cl_2) and tetrahydrofuran (THF) were dried by passage through a column of neutral alumina under nitrogen prior to use. All other reagents were purchased from commercial sources and were used as received unless otherwise noted.

Synthesis of (*E*)-3-Mesitylacrylaldehyde (2g)

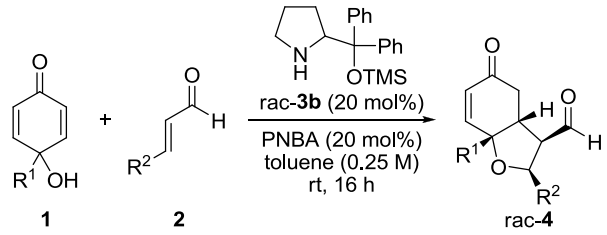


A flame-dried 100-mL round-bottom flask equipped with a magnetic stir bar was charged with NaH (60%) (0.5 g, 12.5 mmol, 1.25 equiv) suspended in THF (25 mL). The suspension was cooled to 0 ºC. Triethyl phosphonoacetate (2.5 mL, 12.5 mmol, 1.25 equiv) was added dropwise. The homogenous solution was allowed to stir at 0 ºC for 20 min before mesitaldehyde (**A**) (1.5 mL, 10.0 mmol, 1.00 equiv) was added dropwise. The ice bath was removed and the resulting solution was allowed to stir for 3 h as it slowly warmed to room temperature. The reaction was cooled to 0 ºC and quenched with sat. aq. NH_4Cl (25 mL). The reaction was diluted with Et_2O (100 mL) and washed with H_2O (40 mL) and brine (40 mL). The organic layer was dried over $MgSO_4$, filtered, and concentrated *in vacuo* to afford analytically pure unsaturated ester **B**, which was used without further purification.

A flame-dried 250-mL round-bottom flask equipped with a magnetic stir bar was charged with unsaturated ester **B** in CH_2Cl_2 (100 mL). The solution was cooled to 0 ºC. DIBAL (4.0 mL, 22.0 mmol, 2.20 equiv) was added dropwise. The reaction was allowed to stir at 0 ºC for 2 h. The reaction was quenched by sequential addition of acetone (25 mL) and 1 N HCl (100 mL). The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (2 x 75 mL). The combined organic extracts were dried over Na_2SO_4, filtered, and concentrated *in vacuo* to afford analytically pure allyl alcohol **C**, which was used without further purification.

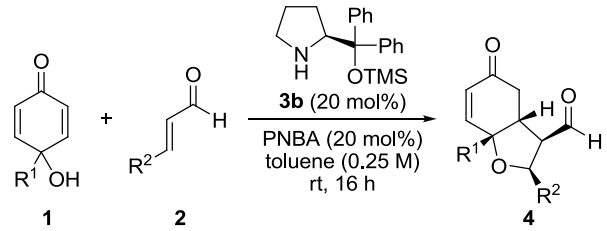
A flame-dried 250-mL round-bottom flask equipped with a magnetic stir bar was charged with allyl alcohol **C** in CH_2Cl_2 (100 mL). Activated MnO_2 (4.4 g, 50.0 mmol, 5.00 equiv) was added and the reaction was allowed to stir at room temperature for 16 h. The reaction was filtered through a pad of Celite® rinsing with CH_2Cl_2 (3 x 50 mL). The filtrate was concentrated *in vacuo*. The crude residue was purified by column chromatography on silica gel to afford (*E*)-3-mesitylacrylaldehyde (**2g**) (1.27 g, 73% yield) as a white solid (mp 72-73 ºC). Analytical data for **2g**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 9.70 (d, *J* = 7.7 Hz, 1H), 7.68 (d, *J* = 16.3 Hz, 1H), 6.94 (s, 2H), 6.41 (dd, *J* = 16.3, 7.7 Hz, 1H), 2.36 (s, 6H), 2.31 (s, 3H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 194.32,† 194.28,‡ 151.5, 139.5, 137.1, 133.5, 130.0, 129.5, 21.2, 21.1 (†Rotomer A, ‡Rotomer B); **IR** (thin film): 1662, 1626, 1136, 1020, 984, 845 cm^{-1}; **TLC** (20% ethyl acetate:hexanes): R_f = 0.49; **HRMS** (ESI): Calcd. for $C_{12}H_{15}O$ ($[M+H]^+$): 175.1124, Found: 175.1117.

General Procedure A for the Preparation of rac-4



A 20-mL scintillation vial equipped with a magnetic stir bar was charged with α,β-unsaturated aldehyde **2** (1.50 mmol, 1.5 equiv), rac-**3b** (70.5 mg, 0.20 mmol, 0.2 equiv), and 4-nitrobenzoic acid (33.4 mg, 0.20 mmol, 0.2 equiv) in toluene (4.0 mL, 0.25 M). The solution was stirred for 5 min at room temperature until homogeneous. *p*-Quinol **1** (1.00 mmol, 1.0 equiv) was added, the vial was capped, and the reaction was allowed to stir for 16 h at room temperature. The reaction was diluted with EtOAc (30 mL) and sequentially washed with sat. aq. $NaHCO_3$ (1 x 15 mL), H_2O (1 x 15 mL), and brine (1 x 15 mL). The organic layer was dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The diastereomeric ratio was determined by ^1H NMR analysis of the crude residue. The crude residue was purified by column chromatography on silica gel to afford rac-**4**.

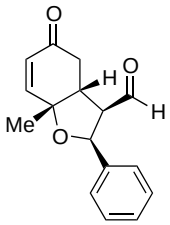
General Procedure B for the Preparation of 4



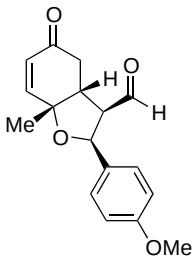
1a: R^1 = Me	**2a**: R^2 = C_6H_5	**2f**: R^2 = 2-$CF_3C_6H_4$
1b: R^1 = Et	**2b**: R^2 = 4-$OMeC_6H_4$	**2g**: R^2 = 2,4,6-$Me_3C_6H_2$
1c: R^1 = ^{i}Pr	**2c**: R^2 = 4-$NO_2C_6H_4$	**2h**: R^2 = *N*-Ts-indol-3-yl
1d: R^1 = CH_2CH_2OTBS	**2d**: R^2 = 2-ClC_6H_4	**2i**: R^2 = thien-2-yl
1e: R^1 = $CH_2CH_2CO_2Me$	**2e**: R^2 = 2-$OMeC_6H_4$	**2j**: R^2 = Me

Note: No precautions were taken to preclude water or air from the reactions. Reactions were performed in non-flame-dried glassware using reagent grade solvents as received under ambient atmosphere. Reactions can be performed employing CH_2Cl_2 or $CHCl_3$ in place of toluene with comparable levels of selectivity, but in slightly reduced yields.

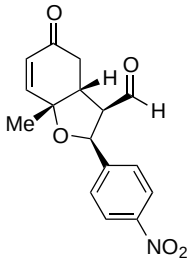
A 20-mL scintillation vial equipped with a magnetic stir bar was charged with α,β-unsaturated aldehyde **2** (1.50 mmol, 1.5 equiv), **3b** (70.5 mg, 0.20 mmol, 0.2 equiv), and 4-nitrobenzoic acid (33.4 mg, 0.20 mmol, 0.2 equiv) in toluene (4.0 mL, 0.25 M). The solution was stirred for 5 min at room temperature until homogeneous. *p*-Quinol **1** (1.00 mmol, 1.0 equiv) was added, the vial was capped, and the reaction was allowed to stir for 16 h at room temperature. The reaction was diluted with EtOAc (30 mL) and sequentially washed with sat. aq. $NaHCO_3$ (1 x 15 mL), H_2O (1 x 15 mL), and brine (1 x 15 mL). The organic layer was dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The diastereomeric ratio was determined by ^1H NMR analysis of the crude residue. The crude residue was purified by column chromatography on silica gel to afford **4**. The enantiomeric ratio was determined following reduction to **5** (with subsequent benzoate formation for **4h** and **4j**) or olefination to **8**.



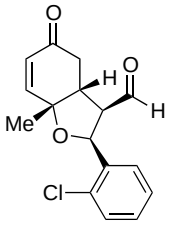
(2*S*,3*R*,3a*R*,7a*R*)-7a-Methyl-5-oxo-2-phenyl-2,3,3a,4,5,7a-hexahydrobenzofuran-3-carbaldehyde (4a): The title compound was prepared according to General Procedure B using *p*-quinol **1a** (124 mg, 1.00 mmol) and α,β-unsaturated aldehyde **2a** (198 mg, 1.50 mmol) affording a 15:1 (**4a**:Σothers) mixture of diastereomers. Purification provided **4a** (207 mg, 81% yield, >20:1 dr) as a pale yellow oil. The enantiomeric ratio was determined following reduction to **5a**. Analytical data for **4a**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.99 (d, *J* = 1.7 Hz, 1H), 7.36-7.33 (m, 2H), 7.29-7.27 (m, 3H), 6.71 (dd, *J* = 10.3, 1.5 Hz, 1H), 6.08 (d, *J* = 10.3 Hz, 1H), 5.23 (d, *J* = 9.7 Hz, 1H), 3.14 (dt, *J* = 9.5, 1.7 Hz, 1H), 3.06-3.03 (m, 1H), 2.70 (dd, *J* = 17.4, 5.4 Hz, 1H), 2.55 (d, *J* = 17.4 Hz, 1H), 1.67 (s, 3H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 198.9, 196.4, 152.0, 136.9, 129.8, 128.8, 128.4, 126.3, 79.6, 79.1, 60.4, 43.0, 37.3, 23.2; **IR** (thin film): 2973, 1718, 1679, 1494, 1455, 1122, 1045, 703 cm^{-1}; **TLC** (40% ethyl acetate:hexanes): R_f = 0.50; **HRMS** (ESI): Calcd. for $C_{16}H_{17}O_3$ ($[M+H]^+$): 257.1178, Found: 257.1173; **$[\alpha]_D$** -116 (*c* = 1.9, $CHCl_3$)**.**



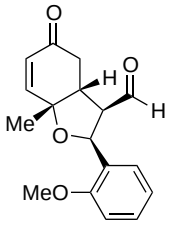
(2*S*,3*R*,3a*R*,7a*R*)-2-(4-Methoxyphenyl)-7a-methyl-5-oxo-2,3,3a,4,5,7a-hexahydrobenzofuran-3-carbaldehyde (4b): The title compound was prepared according to General Procedure B using *p*-quinol **1a** (124 mg, 1.00 mmol) and α,β-unsaturated aldehyde **2b** (243 mg, 1.50 mmol) affording a 9:1 (**4b**:Σothers) mixture of diastereomers. Purification provided **4b** (223 mg, 78% yield, >20:1 dr) as a pale yellow oil. The enantiomeric ratio was determined following reduction to **5b**. Analytical data for **4b**: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 9.02 (d, *J* = 1.8 Hz, 1H), 7.18 (d, *J* = 8.7 Hz, 2H), 6.87 (d, *J* = 8.7 Hz, 2H), 6.72 (dd, *J* = 10.3, 1.8 Hz, 1H), 6.08 (d, *J* = 10.3 Hz, 1H), 5.19 (d, *J* = 9.3 Hz, 1H), 3.78 (s, 3H), 3.14-3.04 (m, 2H), 2.70 (dd, *J* = 17.4, 5.2 Hz, 1H), 2.55 (d, *J* = 17.4 Hz, 1H), 1.66 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 199.1, 196.5, 159.5, 152.1, 129.8, 128.9, 127.7, 114.2, 79.3, 78.9, 60.4, 55.2, 42.9, 37.3, 23.2; **IR** (thin film): 2930, 1717, 1682, 1612, 1513, 1249, 1032, 836 cm^{-1}; **TLC** (40% ethyl acetate:hexanes): R_f = 0.36; **HRMS** (ESI): Calcd. for $C_{17}H_{19}O_4$ ($[M+H]^+$): 287.1284, Found: 287.1279; **$[\alpha]_D$** -87 (*c* = 1.6, $CHCl_3$).



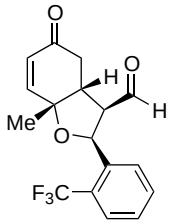
(2*S*,3*R*,3a*R*,7a*R*)-7a-Methyl-2-(4-nitrophenyl)-5-oxo-2,3,3a,4,5,7a-hexahydrobenzofuran-3-carbaldehyde (4c): The title compound was prepared according to General Procedure B using *p*-quinol **1a** (124 mg, 1.00 mmol) and α,β-unsaturated aldehyde **2c** (266 mg, 1.50 mmol) affording a 17:1 (**4c**:Σothers) mixture of diastereomers. Purification provided **4c** (214 mg, 77% yield, >20:1 dr) as a pale yellow oil. The enantiomeric ratio was determined following reduction to **5c**. Analytical data for **4c**: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 9.02 (d, *J* = 2.5 Hz, 1H), 8.22 (d, *J* = 8.8 Hz, 2H), 7.48 (d, *J* = 8.6 Hz, 2H), 6.72 (dd, *J* = 10.3, 2.0 Hz, 1H), 6.13 (dd, *J* = 10.3, 1.0 Hz, 1H), 5.32 (d, *J* = 9.6 Hz, 1H), 3.25 (dt, *J* = 9.5, 2.5 Hz, 1H), 3.08-3.03 (m, 1H), 2.73 (dd, *J* = 17.4, 5.4 Hz, 1H), 2.56 (ddd, *J* = 17.4, 2.1, 1.1 Hz, 1H), 1.71 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 198.0, 195.9, 151.2, 147.8, 144.2, 130.3, 127.2, 124.1, 80.3, 78.3, 60.5, 43.4, 37.2, 23.3; **IR** (thin film): 2089, 1716, 1681, 1645, 1520, 1457, 1348, 529 cm^{-1}; **TLC** (40% ethyl acetate:hexanes): R_f = 0.24; **HRMS** (ESI): Calcd. for $C_{16}H_{16}NO_5$ ($[M+H]^+$): 302.1029, Found: 302.1019; **$[\alpha]_D$** -115 (*c* = 0.5, $CHCl_3$)**.**



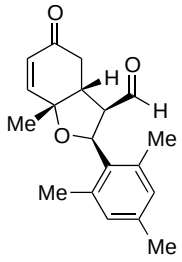
(2*S*,3*R*,3a*R*,7a*R*)-2-(2-Chlorophenyl)-7a-methyl-5-oxo-2,3,3a,4,5,7a-hexahydrobenzofuran-3-carbaldehyde (4d): The title compound was prepared according to General Procedure B using *p*-quinol **1a** (124 mg, 1.00 mmol) and α,β-unsaturated aldehyde **2d** (250 mg, 1.50 mmol) affording a 17:1 (**4d**:Σothers) mixture of diastereomers. Purification provided **4d** (273 mg, 94% yield, >20:1 dr) as an off-white solid (mp 150-153 ºC). The enantiomeric ratio was determined following reduction to **5d**. Analytical data for **4d**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 9.09 (d, *J* = 2.0 Hz, 1H), 7.52 (d, *J* = 7.7 Hz, 1H), 7.34 (d, *J* = 7.9 Hz, 1H), 7.28 (t, *J* = 7.6 Hz, 1H), 7.25-7.22 (m, 1H), 6.71 (dd, *J* = 10.3, 1.9 Hz, 1H), 6.12 (d, *J* = 10.3 Hz, 1H), 5.47 (d, *J* = 9.5 Hz, 1H), 3.35 (dt, *J* = 9.5, 2.0 Hz, 1H), 3.01-2.98 (m, 1H), 2.69 (dd, *J* = 17.5, 5.5 Hz, 1H), 2.56 (dd, *J* = 17.5, 1.0 Hz, 1H), 1.68 (s, 3H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 198.6,† 198.5,‡ 196.4, 151.7, 135.0, 131.3, 130.4, 129.4, 129.3, 127.34, 127.29, 79.5, 75.9, 58.6, 43.0, 37.3, 23.1 (†Rotomer A, ‡Rotomer B); **IR** (thin film): 1717, 1682, 1653, 1474, 1374, 1120, 1049 cm^{-1}; **TLC** (40% ethyl acetate:hexanes): R_f = 0.51; **HRMS** (ESI): Calcd. for $C_{16}H_{16}ClO_3$ ($[M+H]^+$): 291.0789, Found: 291.0785; **$[\alpha]_D$** -205 (*c* = 1.4, $CHCl_3$).



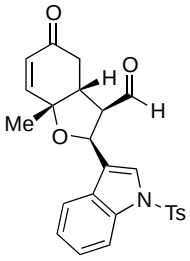
(2*S*,3*R*,3a*R*,7a*R*)-2-(2-Methoxyphenyl)-7a-methyl-5-oxo-2,3,3a,4,5,7a-hexahydrobenzofuran-3-carbaldehyde (4e): The title compound was prepared according to General Procedure B using *p*-quinol **1a** (124 mg, 1.00 mmol) and α,β-unsaturated aldehyde **2e** (243 mg, 1.50 mmol) affording a 14:1 (**4e**:Σothers) mixture of diastereomers. The enantiomeric ratio was determined following reduction to **5e**. Purification provided **4e** (247 mg, 86% yield, >20:1 dr) as a white solid (mp 101-103 ºC). Analytical data for **4e**: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 9.00 (d, *J* = 2.5 Hz, 1H), 7.42 (d, *J* = 7.0 Hz, 1H), 7.23 (dt, *J* = 8.0, 1.6 Hz, 1H), 6.94 (t, *J* = 7.5 Hz, 1H), 6.80 (d, *J* = 10.3, 1.9 Hz, 1H), 6.68 (dd, *J* = 10.3, 1.9 Hz, 1H), 6.06 (dd, *J* = 10.3, 0.7 Hz, 1H), 5.36 (d, *J* = 9.3 Hz, 1H), 3.77 (s, 3H), 3.35 (dt, *J* = 9.2, 2.5 Hz, 1H), 2.89-2.84 (m, 1H), 2.65 (dd, *J* = 17.4, 5.4 Hz, 1H), 2.52 (d, *J* = 17.4 Hz, 1H), 1.63 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 199.5, 196.6, 155.1, 152.2, 129.9, 129.0, 126.1, 125.5, 120.7, 109.8, 79.1, 74.5, 58.6, 55.0, 43.1, 37.2, 23.1; **IR** (thin film): 2839, 1717, 1684, 1490, 1296, 1116, 1046, 757 cm^{-1}; **TLC** (40% ethyl acetate:hexanes): R_f = 0.46; **HRMS** (ESI): Calcd. for $C_{17}H_{19}O_4$ ($[M+H]^+$): 287.1284, Found: 287.1279; **$[\alpha]_D$** -182 (*c* = 1.8, $CHCl_3$).



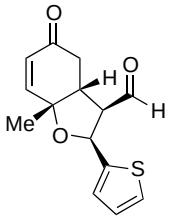
(2*S*,3*R*,3a*R*,7a*R*)-7a-Methyl-5-oxo-2-(2-(trifluoromethyl)phenyl)-2,3,3a,4,5,7a-hexahydrobenzofuran-3-carbaldehyde (4f): The title compound was prepared according to General Procedure B using *p*-quinol **1a** (124 mg, 1.00 mmol) and α,β-unsaturated aldehyde **2f** (300 mg, 1.50 mmol) affording a >20:1 (**4f**:Σothers) mixture of diastereomers. Purification provided **4f** (295 mg, 91% yield, >20:1 dr) as a white solid (mp 99-100 ºC). The enantiomeric ratio was determined following reduction to **5f**. Analytical data for **4f**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.95 (d, *J* = 1.1 Hz, 1H), 7.65 (d, *J* = 7.9 Hz, 1H), 7.61 (d, *J* = 7.9 Hz, 1H), 7.57 (t, *J* = 7.7 Hz, 1H), 7.41 (t, *J* = 7.6 Hz, 1H), 6.73 (dd, *J* = 10.3, 2.0 Hz, 1H), 6.12 (d, *J* = 10.3 Hz, 1H), 5.52 (d, *J* = 9.8 Hz, 1H), 3.21 (t, *J* = 9.5 Hz, 1H), 3.10-3.07 (m, 1H), 2.70 (dd, *J* = 17.5, 5.4 Hz, 1H), 2.53 (dt, *J* = 17.5, 1.1 Hz, 1H), 1.68 (s, 3H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 198.4,† 198.3,‡ 196.3, 151.5, 135.8, 132.6, 130.4, 128.5, 128.0, 127.1 (q, J_{C-F} = 30.2 Hz), 125.8 (q, J_{C-F} = 6.0 Hz), 124.0 (q, J_{C-F} = 273.3 Hz), 79.6, 74.61,† 74.60,‡ 60.5, 42.6, 37.3, 23.1 (†Rotomer A, ‡Rotomer B); **^{19}F NMR** (565 MHz, $CDCl_3$): δ -59.3; **IR** (thin film): 1717, 1683, 1653, 1314, 1165, 1121, 770 cm^{-1}; **TLC** (40% ethyl acetate:hexanes): R_f = 0.49; **HRMS** (ESI): Calcd. for $C_{17}H_{16}F_3O_3$ ($[M+H]^+$): 325.1052, Found: 325.1048; **$[\alpha]_D$** -127 (*c* = 0.8, $CHCl_3$).



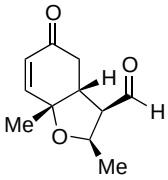
(2*S*,3*R*,3a*R*,7a*R*)-2-Mesityl-7a-methyl-5-oxo-2,3,3a,4,5,7a-hexahydrobenzofuran-3-carbaldehyde (4g): The title compound was prepared according to General Procedure B using *p*-quinol **1a** (124 mg, 1.00 mmol) and α,β-unsaturated aldehyde **2g** (261 mg, 1.50 mmol) affording a 4:1 (**4g**:Σothers) mixture of diastereomers. Purification provided **4g** (215 mg, 72% yield, >20:1 dr) as a pale yellow oil. The enantiomeric ratio was determined following reduction to **5g**. Analytical data for **4g**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 9.04 (d, *J* = 1.3 Hz, 1H), 6.79 (bs, 2H), 6.68 (dd, *J* = 10.3, 2.0 Hz, 1H), 6.11 (dd, *J* = 10.3, 0.8 Hz, 1H), 5.47 (d, *J* = 10.5 Hz, 1H), 3.18-3.15 (m, 1H), 3.04 (dt, *J* = 8.9, 1.3 Hz, 1H), 2.73 (dd, *J* = 17.3, 5.3 Hz, 1H), 2.60 (dd, *J* = 17.3, 0.9 Hz, 1H), 2.27 (bs, 6H), 2.22 (s, 3H), 1.63 (s, 3H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 199.0, 196.8, 152.4, 137.7, 136.2,† 135.3,‡ 131.6,† 130.5, 129.4,‡ 128.7, 79.2, 76.3, 59.02,† 58.98, 43.2,† 43.1,‡ 37.8, 22.7, 22.1,† 20.8,‡ 20.71,† 20.68‡ (†Rotomer A, ‡Rotomer B); **IR** (thin film): 2929, 1732, 1684, 1653, 1558, 1507, 1220, 1124 cm^{-1}; **TLC** (40% ethyl acetate:hexanes): R_f = 0.56; **HRMS** (ESI): Calcd. for $C_{19}H_{23}O_3$ ($[M+H]^+$): 299.1648, Found: 299.1643; **$[\alpha]_D$** -81 (*c* = 1.5, $CHCl_3$).



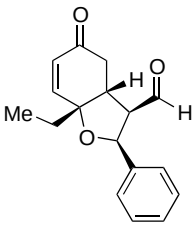
(2*S*,3*R*,3a*R*,7a*R*)-7a-Methyl-5-oxo-2-(1-tosyl-1*H*-indol-3-yl)-2,3,3a,4,5,7a-hexahydrobenzofuran-3-carbaldehyde (4h): The title compound was prepared according to General Procedure B using *p*-quinol **1a** (124 mg, 1.00 mmol) and α,β-unsaturated aldehyde **2h** (488 mg, 1.50 mmol) affording a 5:1 (**4h**:Σothers) mixture of diastereomers. Purification provided **4h** (328 mg, 73% yield, >20:1 dr) as a viscous pale yellow oil. The enantiomeric ratio was determined following reduction and benzoate formation to **S1a**. Analytical data for **4h**: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 9.00 (d, *J* = 2.0 Hz, 1H), 7.94 (d, *J* = 8.3 Hz, 1H), 7.72 (d, *J* = 8.4 Hz, 2H), 7.61 (s, 1H), 7.39 (d, *J* = 7.8 Hz, 1H), 7.31 (d, *J* = 7.4 Hz, 1H), 7.22-7.19 (m, 3H), 6.70 (dd, *J* = 10.3, 1.7 Hz, 1H), 6.11 (d, *J* = 10.3 Hz, 1H), 5.40 (d, *J* = 8.6 Hz, 1H), 3.18-3.08 (m, 2H), 2.73 (dd, *J* = 17.9, 5.1 Hz, 1H), 2.57 (d, *J* = 17.1 Hz, 1H), 2.31 (s, 3H), 1.69 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 198.3, 196.2, 151.5, 145.2, 135.3, 134.8, 130.4, 129.9, 127.7, 126.7, 125.3, 124.2, 123.6, 119.7, 118.7, 113.8, 79.6, 77.2, 73.1, 59.6, 43.3, 37.3, 23.2, 21.5; **IR** (thin film): 2917, 1717, 1698, 1684, 1507, 1457, 1373, 1174 cm^{-1}; **TLC** (40% ethyl acetate:hexanes): R_f = 0.29; **HRMS** (ESI): Calcd. for $C_{50}H_{47}N_2O_{10}S_2$ ($[2M+H]^+$): 899.2673, Found: 899.2696; **$[\alpha]_D$** -98 (*c* = 1.2, $CHCl_3$).



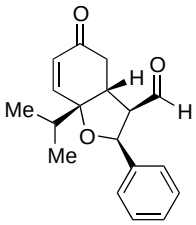
(2*S*,3*R*,3a*R*,7a*R*)-7a-Methyl-5-oxo-2-(thiophen-2-yl)-2,3,3a,4,5,7a-hexahydrobenzofuran-3-carbaldehyde (4i): The title compound was prepared according to General Procedure B using *p*-quinol **1a** (124 mg, 1.00 mmol) and α,β-unsaturated aldehyde **2i** (207 mg, 1.50 mmol) affording a 7:1 (**4i**:Σothers) mixture of diastereomers. Purification provided **4i** (171 mg, 65% yield, >20:1 dr) as a pale yellow oil. The enantiomeric ratio was determined following reduction to **5i**. Analytical data for **4i**: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 9.17 (d, *J* = 1.9 Hz, 1H), 7.28-7.26 (m, 1H), 6.97-6.96 (m, 2H), 6.69 (dd, *J* = 10.3, 1.9 Hz, 1H), 6.04 (dd, *J* = 10.3, 0.9 Hz, 1H), 5.57 (d, *J* = 9.3 Hz, 1H), 3.15 (dt, *J* = 9.8, 1.9 Hz, 1H), 3.10-3.05 (m, 1H), 2.71 (dd, *J* = 17.5, 5.2 Hz, 1H), 2.58 (ddd, *J* = 17.5, 2.0, 1.1 Hz, 1H), 1.65 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 198.2, 196.2, 151.7, 141.2, 129.4, 127.2, 125.9, 125.0, 79.9, 75.4, 60.0, 42.9, 36.9, 23.5; **IR** (thin film): 2973, 1868, 1717, 1683, 1558, 1457, 1374, 1120 cm^{-1}; **TLC** (40% ethyl acetate:hexanes): R_f = 0.39; **HRMS** (ESI): Calcd. for $C_{14}H_{15}O_3S$ ($[M+H]^+$): 263.0743, Found: 263.0736; **$[\alpha]_D$** -90 (*c* = 1.3, $CHCl_3$).



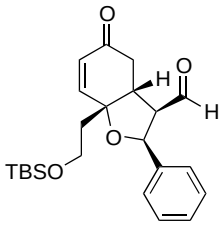
(2*R*,3*R*,3a*R*,7a*R*)-2,7a-Dimethyl-5-oxo-2,3,3a,4,5,7a-hexahydrobenzofuran-3-carbaldehyde (4j): The title compound was prepared according to General Procedure B using *p*-quinol **1a** (124 mg, 1.00 mmol) and α,β-unsaturated aldehyde **2j** (105 mg, 1.50 mmol) affording a 7:1 (**4j**:Σothers) mixture of diastereomers. Purification provided **4j** (116 mg, 60% yield, >20:1 dr) as a pale yellow oil. The enantiomeric ratio was determined following reduction and benzoate formation to **S1b**. Analytical data for **4j**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 9.72 (d, *J* = 2.1 Hz, 1H), 6.60 (dd, *J* = 10.3, 1.5 Hz, 1H), 5.99 (d, *J* = 10.3 Hz, 1H), 4.32 (dq, *J* = 6.6, 2.2 Hz, 1H), 2.96-2.94 (m, 1H), 2.90 (dt, *J* = 9.0, 2.0 Hz, 1H), 2.64 (dd, *J* = 17.3, 5.4 Hz, 1H), 2.51 (d, *J* = 17.3 Hz, 1H), 1.52 (s, 3H), 1.27 (d, *J* = 6.6 Hz, 3H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 200.10,[†] 200.06,[‡] 196.7, 152.5, 129.4, 78.9, 73.7, 59.2, 43.1, 37.3, 23.5, 18.0 ([†]Rotomer A, [‡]Rotomer B); **IR** (thin film): 1716, 1683, 1653, 1541, 1457, 1367, 709 cm^{-1}; **TLC** (40% ethyl acetate:hexanes): R_f = 0.29; **HRMS** (ESI): Calcd. for $C_{11}H_{15}O_3$ ($[M+H]^+$): 195.1022, Found: 195.0995; **$[\alpha]_D$** -38 (*c* = 0.7, $CHCl_3$).



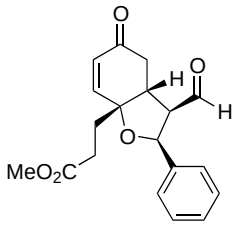
(2*S*,3*R*,3a*R*,7a*R*)-7a-Ethyl-5-oxo-2-phenyl-2,3,3a,4,5,7a-hexahydrobenzofuran-3-carbaldehyde (4k): The title compound was prepared according to General Procedure B using *p*-quinol **1b** (138 mg, 1.00 mmol) and α,β-unsaturated aldehyde **2a** (198 mg, 1.50 mmol) affording a 12:1 (**4k**:Σothers) mixture of diastereomers. Purification provided **4k** (212 mg, 78% yield, >20:1 dr) as a pale yellow oil. The enantiomeric ratio was determined following reduction to **5k**. Analytical data for **4k**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 9.02 (d, *J* = 2.7 Hz, 1H), 7.39-7.29 (m, 5H), 6.77 (dd, *J* = 15.5, 2.5 Hz, 1H), 6.18 (d, *J* = 15.5 Hz, 1H), 5.24 (d, *J* = 13.3 Hz, 1H), 3.21-3.13 (m, 2H), 2.71 (dd, *J* = 26.4, 7.9 Hz, 1H), 2.55 (d, *J* = 26.4 Hz, 1H), 2.13-1.93 (m, 2H), 1.20 (t, *J* = 11.3 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 199.0, 196.6, 151.4, 137.0, 130.6, 128.8, 128.3, 126.3, 81.6, 78.8, 60.7, 40.3, 37.6, 29.8, 8.1; **IR** (thin film): 2972, 1718, 1683, 1576, 1507, 1457, 1396, 1219 cm^{-1}; **TLC** (40% ethyl acetate:hexanes): R_f = 0.55; **HRMS** (ESI): Calcd. for $C_{17}H_{19}O_3$ ($[M+H]^+$): 271.1335, Found: 271.1329; **$[\alpha]_D$** -110 (*c* = 1.3, $CHCl_3$).



(2*S*,3*R*,3a*R*,7a*R*)-7a-Isopropyl-5-oxo-2-phenyl-2,3,3a,4,5,7a-hexahydrobenzofuran-3-carbaldehyde (4l): The title compound was prepared according to General Procedure B using *p*-quinol **1c** (152 mg, 1.00 mmol) and α,β-unsaturated aldehyde **2a** (198 mg, 1.50 mmol) affording a 6:1 (**4l**:Σothers) mixture of diastereomers. Purification provided **4l** (202 mg, 71% yield, 10:1 dr) as a pale yellow oil. The enantiomeric ratio was determined following reduction to **5l**. Analytical data for **4l**: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 9.02 (d, *J* = 2.0 Hz, 1H), 7.39-7.29 (m, 5H), 6.75 (dd, *J* = 10.4, 1.7 Hz, 1H), 6.28 (d, *J* = 10.4 Hz, 1H), 5.19 (d, *J* = 9.3 Hz, 1H), 3.34-3.31 (m, 1H), 3.12-3.07 (m, 1H), 2.74 (dd, *J* = 17.7, 6.0 Hz, 1H), 2.53 (d, *J* = 17.7 Hz, 1H), 2.34-2.23 (m, 1H), 1.21 (dd, *J* = 10.1, 7.0 Hz, 6H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 199.0, 197.1, 150.5, 136.8, 131.9, 128.9, 128.4, 126.4, 83.6, 78.6, 62.1, 39.1, 38.2, 35.4, 18.0, 17.1; **IR** (thin film): 2967, 1717, 1683, 1636, 1457, 1268, 1222 cm^{-1}; **TLC** (40% ethyl acetate:hexanes): R_f = 0.60; **HRMS** (ESI): Calcd. for $C_{18}H_{21}O_3$ ($[M+H]^+$): 285.1491, Found: 285.1486; **$[\alpha]_D$** -122 (*c* = 0.8, $CHCl_3$).

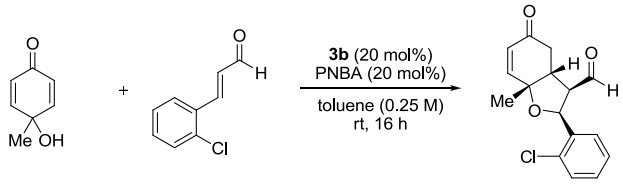


(2*S*,3*R*,3a*R*,7a*R*)-7a-(2-((*tert*-Butyldimethylsilyl)oxy)ethyl)-5-oxo-2-phenyl-2,3,3a,4,5,7a-hexahydrobenzofuran-3-carbaldehyde (4m): The title compound was prepared according to General Procedure B using *p*-quinol **1d** (268 mg, 1.00 mmol) and α,β-unsaturated aldehyde **2a** (198 mg, 1.50 mmol) affording a 13:1 (**4m**:Σothers) mixture of diastereomers. Purification provided **4m** (329 mg, 82% yield, >20:1 dr) as a pale yellow oil. The enantiomeric ratio was determined following reduction to **5m**. Analytical data for **4m**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.99 (d, *J* = 2.3 Hz, 1H), 7.36-7.34 (m, 2H), 7.30-7.27 (m, 3H), 6.75 (dd, *J* = 10.3, 1.9 Hz, 1H), 6.12 (d, *J* = 10.3 Hz, 1H), 5.22 (d, *J* = 9.8 Hz, 1H), 4.03-3.99 (m, 1H), 3.91-3.87 (m, 1H), 3.37-3.34 (m, 1H), 3.13 (dt, *J* = 9.6, 2.3 Hz, 1H), 2.83 (dd, *J* = 17.5, 5.5 Hz, 1H), 2.53 (dd, *J* = 17.5, 1.1 Hz, 1H), 2.24-2.19 (m, 1H), 2.15-2.11 (m, 1H), 0.90 (s, 9H), 0.08 (d, *J* = 6.9 Hz, 3H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 199.0, 197.1, 151.6, 137.0, 130.2, 128.9, 128.5, 126.4, 81.1, 79.0, 60.5, 58.4, 41.4, 39.9, 37.4, 25.8, 18.1, -5.5; **IR** (thin film): 2953, 2856, 1717, 1684, 1472, 1255, 1089, 837 cm^{-1}; **TLC** (40% ethyl acetate:hexanes): R_f = 0.69; **HRMS** (ESI): Calcd. for $C_{23}H_{33}O_4Si$ ($[M+H]^+$): 401.2149, Found: 401.2144; **$[\alpha]_D$** -69 (*c* = 1.2, $CHCl_3$).



Methyl 3-((2*S*,3*R*,3a*R*,7a*R*)-3-formyl-5-oxo-2-phenyl-2,3,3a,4,5,7a-hexahydrobenzofuran-7a-yl)propanoate (4n): The title compound was prepared according to General Procedure B using *p*-quinol **1e** (196 mg, 1.00 mmol) and α,β-unsaturated aldehyde **2a** (198 mg, 1.50 mmol) affording a 6:1 (**4n**:Σothers) mixture of diastereomers. Purification provided **4n** (246 mg, 75% yield, >20:1 dr) as a pale yellow oil. The enantiomeric ratio was determined following olefination to **8**. Analytical data for **4n**: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.96 (s, 1H), 7.36-7.22 (m, 5H), 6.71 (d, *J* = 10.4 Hz, 1H), 6.14 (d, *J* = 10.3 Hz, 1H), 5.19 (dd, *J* = 14.8, 1.8 Hz, 1H), 3.68 (s, 3H), 3.15-3.09 (m, 2H), 2.73-2.62 (m, 3H), 2.52 (d, *J* = 17.4 Hz, 1H), 2.38-2.22 (m, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 198.6, 196.3, 173.2, 150.3, 136.6, 131.0, 128.9, 128.5, 126.4, 80.5, 79.1, 60.3, 51.8, 40.6, 37.3, 31.4, 28.4; **IR** (thin film): 2952, 1733, 1717, 1683, 1636, 1200, 1028, 755 cm^{-1}; **TLC** (40% ethyl acetate:hexanes): R_f = 0.32; **HRMS** (ESI): Calcd. for $C_{19}H_{21}O_5$ ($[M+H]^+$): 329.1390, Found: 329.1385; **$[\alpha]_D$** -78 (*c* = 0.9, $CHCl_3$)**.**

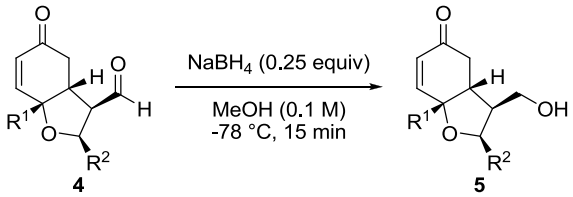
Gram Scale Synthesis of 4d



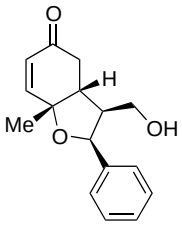
A 250-mL round bottom flask equipped with a magnetic stir bar was charged with α,β-unsaturated aldehyde **2d** (5.00 g, 30.0 mmol, 1.5 equiv), **3b** (1.41 g, 4.0 mmol, 0.2 equiv), and 4-nitrobenzoic acid (0.67 g, 4.0 mmol, 0.2 equiv) in toluene (80.0 mL, 0.25 M). The solution was stirred for 5 min at room temperature until homogeneous. *p*-Quinol **1a** (2.48 g, 20.0 mmol, 1.0 equiv) was added and the reaction was allowed to stir for 16 h at room temperature. The reaction was diluted with EtOAc (400 mL) and sequentially washed with sat. aq. $NaHCO_3$ (1 x 100 mL), H_2O (1 x 100 mL), and brine

(1 x 100 mL). The organic layer was dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The diastereomeric ratio was determined by ^1H NMR analysis of the crude residue. The crude residue was purified by column chromatography on silica gel eluting with 20% ethyl acetate:hexanes. The obtained solid was recrystallized from 10% ethyl acetate:hexanes to afford **4d** (4.76 g, 82% yield, >20:1 dr, >99.5:0.5 er) as colorless crystals (mp 150-153 ºC).

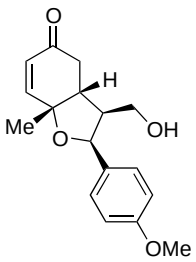
General Procedure C for the Reduction of Aldehydes 4 to Alcohols 5



A 10-mL round-bottom flask equipped with a magnetic stir bar was charged with aldehyde **4** (1.00 equiv) in MeOH (0.1 M). The solution was cooled to -78 ºC. $NaBH_4$ (0.25 equiv) was added and the reaction was allowed to stir for 15 min at -78 ºC. The reaction was quenched with sat. aq. NH_4Cl (2 mL) at -78 ºC and allowed to warm to room temperature. After stirring at room temperature for 30 min, the reaction was partitioned between CH_2Cl_2 (15 mL) and H_2O (30 mL). The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (2 x 15 mL). The combined organic extracts were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by column chromatography on silica gel to afford alcohol **5**.

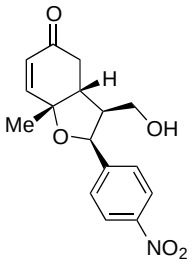


(2*S*,3*S*,3a*R*,7a*R*)-3-(Hydroxymethyl)-7a-methyl-2-phenyl-2,3,3a,4-tetrahydrobenzofuran-5(7a*H*)-one (5a): The title compound was prepared according to General Procedure C using aldehyde **4a** (90 mg, 0.35 mmol) and $NaBH_4$ (3.3 mg, 0.09 mmol) affording **5a** (83 mg, 92% yield) as a pale yellow oil. Analytical data for **5a**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.35-7.32 (m, 2H), 7.30-7.27 (m, 3H), 6.68 (dd, *J* = 10.3, 1.4 Hz, 1H), 6.02 (d, *J* = 10.3 Hz, 1H), 5.00 (d, *J* = 8.4 Hz, 1H), 3.25-3.19 (m, 2H), 2.71 (d, *J* = 17.2 Hz, 1H), 2.65 (dd, *J* = 16.8, 4.9 Hz, 1H), 2.46-2.39 (m, 2H), 1.62 (s, 3H), 1.40 (bs, 1H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 197.7, 152.8, 138.6, 129.0, 128.3, 127.8, 126.4, 80.3, 79.0, 62.5, 49.7, 46.7, 37.9, 23.4; **IR** (thin film): 3445, 2937, 1683, 1456, 1387, 1027, 703 cm^{-1}; **TLC** (50% ethyl acetate:hexanes): R_f = 0.38; **HRMS** (ESI): Calcd. for $C_{32}H_{36}NaO_6$ ($[2M+Na]^+$): 539.2410, Found: 539.2419; **SFC** Chiralpak OD, 9% MeOH, pressure = 150 bar, flow rate = 1.5 mL/min, λ = 210 nm, $t_{R\ (minor)}$ 13.6 min, $t_{R\ (major)}$ 14.7 min, >99.5:0.5 er; **$[\alpha]_D$** -117 (*c* = 1.2, $CHCl_3$).

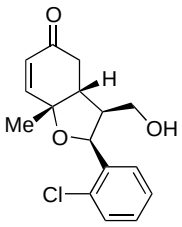


(2*S*,3*S*,3a*R*,7a*R*)-3-(Hydroxymethyl)-2-(4-methoxyphenyl)-7a-methyl-2,3,3a,4-tetrahydrobenzofuran-5(7a*H*)-one (5b): The title compound was prepared according to General Procedure C using aldehyde **4b** (129 mg, 0.45 mmol) and $NaBH_4$ (4.1 mg, 0.11 mmol) affording **5b** (108 mg, 83% yield) as a pale yellow oil. Analytical data for **5b**: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.21 (d, *J* = 7.9 Hz, 2H), 6.86 (d, *J* = 7.7 Hz, 2H), 6.67 (d, *J* = 10.2 Hz, 1H), 6.01 (d, *J* = 10.2 Hz, 1H), 4.97 (d, *J* = 7.6 Hz, 1H), 3.77 (s, 3H), 3.24-3.23 (m, 2H), 2.72-2.61 (m, 2H), 2.43-2.38 (m, 2H), 1.60 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 197.7, 159.1, 152.9, 130.6, 129.0, 127.6, 113.7, 80.0, 78.9, 62.5, 55.2, 49.8, 46.6, 37.8, 23.5; **IR** (thin film): 3420, 2928, 1671, 1514,

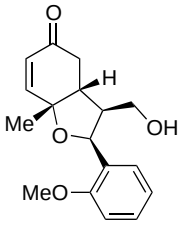
1248, 1174, 1033, 835 cm^{-1}; **TLC** (50% ethyl acetate:hexanes): R_f = 0.24; **HRMS** (ESI): Calcd. for $C_{17}H_{20}NaO_4$ ($[M+Na]^+$): 311.1260, Found: 311.1255; **HPLC** Chiralpak IC, H:IPA = 55:45, flow rate = 1.0 mL/min, λ = 210 nm, $t_{R\ (major)}$ 16.0 min, $t_{R\ (minor)}$ 20.7 min, 98:2 er; **$[\alpha]_D$** -99 (*c* = 1.0, $CHCl_3$).



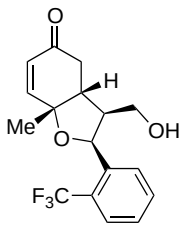
(2*S*,3*S*,3a*R*,7a*R*)-3-(Hydroxymethyl)-7a-methyl-2-(4-nitrophenyl)-2,3,3a,4-tetrahydrobenzofuran-5(7a*H*)-one (5c): The title compound was prepared according to General Procedure C using aldehyde **4c** (135 mg, 0.45 mmol) and $NaBH_4$ (4.1 mg, 0.11 mmol) affording **5c** (109 mg, 80% yield) as a white solid (mp 135-137 ºC). Analytical data for **5c**: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.17 (d, *J* = 8.7 Hz, 2H), 7.49 (d, *J* = 8.5 Hz, 2H), 6.68 (dd, *J* = 10.2, 1.7 Hz, 1H), 6.03 (d, *J* = 10.2 Hz, 1H), 5.11 (d, *J* = 9.0 Hz, 1H), 3.24-3.16 (m, 2H), 2.74-2.63 (m, 2H), 2.58-2.50 (m, 1H), 2.42-2.39 (m, 1H), 1.65 (s, 3H), 1.59 (bs, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 197.4, 152.3, 147.3, 146.4, 129.3, 127.6, 123.2, 79.6, 79.6, 62.3, 49.8, 46.9, 37.9, 23.5; **IR** (thin film): 3431, 1671, 1520, 1347, 1231, 1121, 1047 cm^{-1}; **TLC** (50% ethyl acetate:hexanes): R_f = 0.22; **HRMS** (ESI): Calcd. for $C_{16}H_{17}NNaO_5$ ($[M+Na]^+$): 326.1005, Found: 326.0999; **SFC** Chiralpak OD, 9% MeOH, pressure = 150 bar, flow rate = 3.0 mL/min, λ = 210 nm, $t_{R\ (minor)}$ 26.1 min, $t_{R\ (major)}$ 28.4 min, >99.5:0.5 er; **$[\alpha]_D$** -98 (*c* = 1.5, $CHCl_3$).



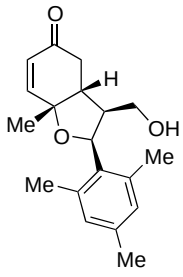
(2*S*,3*S*,3a*R*,7a*R*)-2-(2-Chlorophenyl)-3-(hydroxymethyl)-7a-methyl-2,3,3a,4-tetrahydrobenzofuran-5(7a*H*)-one (5d): The title compound was prepared according to General Procedure C using aldehyde **4d** (250 mg, 0.86 mmol) and $NaBH_4$ (8.1 mg, 0.21 mmol) affording **5d** (234 mg, 93% yield) as a white solid (mp 147-148 ºC). Analytical data for **5d**: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.58 (dd, *J* = 7.7, 1.6 Hz, 1H), 7.33-7.19 (m, 3H), 6.67 (dd, *J* = 10.2, 1.1 Hz, 1H), 6.09 (dd, *J* = 10.2, 1.0 Hz, 1H), 5.20 (dd, *J* = 13.8, 5.8 Hz, 1H), 3.32-3.29 (m, 1H), 3.16-3.13 (m, 1H), 2.77 (d, *J* = 17.3 Hz, 1H), 2.67 (dd, *J* = 17.3, 5.3 Hz, 1H), 2.58-2.50 (m, 2H), 1.63 (s, 3H), 1.30 (bs, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 197.9, 152.3, 136.6, 131.9, 130.2, 129.2, 128.7, 127.4, 126.8, 78.8, 76.8, 62.9, 48.5, 47.3, 38.4, 23.3; **IR** (thin film): 3420, 1683, 1558, 1473, 1374, 1121, 1049, 748 cm^{-1}; **TLC** (50% ethyl acetate:hexanes): R_f = 0.38; **HRMS** (ESI): Calcd. for $C_{16}H_{18}ClO_3$ ($[M+H]^+$): 293.0945, Found: 293.0939; **SFC** Chiralpak OD, 9% MeOH, pressure = 150 bar, flow rate = 1.5 mL/min, λ = 210 nm, $t_{R\ (minor)}$ 10.2 min, $t_{R\ (major)}$ 13.1 min, >99.5:0.5 er; **$[\alpha]_D$** -201 (*c* = 0.7, $CHCl_3$).



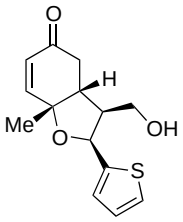
(2*S*,3*S*,3a*R*,7a*R*)-3-(Hydroxymethyl)-2-(2-methoxyphenyl)-7a-methyl-2,3,3a,4-tetrahydrobenzofuran-5(7a*H*)-one (5e): The title compound was prepared according to General Procedure C using aldehyde **4e** (200 mg, 0.70 mmol) and $NaBH_4$ (6.7 mg, 0.17 mmol) affording **5e** (176 mg, 87% yield) as a white solid (mp 124-126 ºC). Analytical data for **5e**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.51 (d, *J* = 7.4 Hz, 1H), 7.30-7.27 (m, 1H), 7.01 (t, *J* = 7.5 Hz, 1H), 6.89 (d, *J* = 8.2 Hz, 1H), 6.71 (dd, *J* = 10.2, 1.1 Hz, 1H), 6.08 (d, *J* = 10.3 Hz, 1H), 5.26 (d, *J* = 7.9 Hz, 1H), 3.82 (s, 3H), 3.25 (bs, 2H), 2.74-2.67 (m, 2H), 2.53-2.48 (m, 2H), 1.62 (s, 3H), 1.36 (bs, 1H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 198.0, 155.5, 153.0, 129.5, 128.6, 127.3, 126.2, 120.8, 110.1, 78.5, 75.2, 62.5, 55.29,† 55.25,‡ 49.2, 46.1, 38.1, 23.4 (†Rotomer A, ‡Rotomer B); **IR** (thin film): 3446, 2929, 1682, 1491, 1244, 1125, 1046, 756 cm^{-1}; **TLC** (50% ethyl acetate:hexanes): R_f = 0.30; **HRMS** (ESI): Calcd. for $C_{17}H_{20}NaO_4$ ($[M+Na]^+$): 311.1260, Found: 311.1254; **SFC** Chiralpak OD, 9% MeOH, pressure = 150 bar, flow rate = 1.5 mL/min, λ = 210 nm, $t_{R\ (minor)}$ 10.9 min, $t_{R\ (major)}$ 12.0 min, >99.5:0.5 er; **$[\alpha]_D$** -175 (*c* = 1.5, $CHCl_3$).



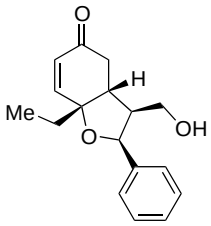
(2*S*,3*S*,3a*R*,7a*R*)-3-(Hydroxymethyl)-7a-methyl-2-(2-(trifluoromethyl)phenyl)-2,3,3a,4-tetrahydrobenzofuran-5(7a*H*)-one (5f): The title compound was prepared according to General Procedure C using aldehyde **4f** (195 mg, 0.60 mmol) and $NaBH_4$ (5.6 mg, 0.15 mmol) affording **5f** (161 mg, 82% yield) as a white solid (mp 133-135 ºC). Analytical data for **5f**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.74 (d, *J* = 7.9 Hz, 1H), 7.61 (d, *J* = 7.9 Hz, 1H), 7.54 (t, *J* = 7.6 Hz, 1H), 7.38 (t, *J* = 7.6 Hz, 1H), 6.68 (dd, *J* = 10.3, 2.0 Hz, 1H), 6.08 (dd, *J* = 10.3, 0.9 Hz, 1H), 5.23 (d, *J* = 9.0 Hz, 1H), 3.18 (dd, *J* = 10.9, 5.0 Hz, 1H), 3.08-3.05 (m, 1H), 2.80 (ddd, *J* = 17.5, 2.0, 1.1 Hz, 1H), 2.66 (d, *J* = 17.3 Hz, 1H), 2.50-2.47 (m, 1H), 2.42-2.37 (m, 1H), 1.64 (s, 3H), 1.63 (bs, 1H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 198.0, 152.3, 136.7, 131.6, 130.2, 128.1, 127.8, 127.4 (q, J_{C-F} = 30.4 Hz), 125.7 (q, J_{C-F} = 5.6 Hz), 124.0 (q, J_{C-F} = 274.2 Hz), 79.0, 75.8, 63.7, 49.7, 48.5, 38.5, 23.3; **^{19}F NMR** (565 MHz, $CDCl_3$): δ -59.2; **IR** (thin film): 3432, 2931, 1677, 1313, 1163, 1123, 1035, 771 cm^{-1}; **TLC** (50% ethyl acetate:hexanes): R_f = 0.37; **HRMS** (ESI): Calcd. for $C_{17}H_{17}F_3NaO_3$ ($[M+Na]^+$): 349.1028, Found: 349.1020; **SFC** Chiralpak OD, 9% MeOH, pressure = 150 bar, flow rate = 1.5 mL/min, λ = 210 nm, $t_{R\ (minor)}$ 5.3 min, $t_{R\ (major)}$ 6.6 min, 99.5:0.5 er; **[α]$_D$** -124 (*c* = 1.5, $CHCl_3$).



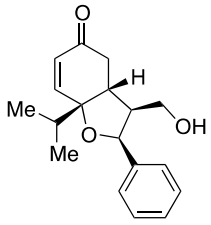
(2*S*,3*S*,3a*R*,7a*R*)-3-(Hydroxymethyl)-2-mesityl-7a-methyl-2,3,3a,4-tetrahydrobenzofuran-5(7a*H*)-one (5g): The title compound was prepared according to General Procedure C using aldehyde **4g** (142 mg, 0.48 mmol) and $NaBH_4$ (4.5 mg, 0.12 mmol) affording **5g** (127 mg, 88% yield) as a pale yellow oil. Analytical data for **5g**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 6.80 (s, 1H), 6.77 (s, 1H), 6.66 (dd, *J* = 10.3, 1.9 Hz, 1H), 6.07 (d, *J* = 10.3 Hz, 1H), 5.24 (d, *J* = 9.3 Hz, 1H), 3.31-3.28 (m, 2H), 2.78 (d, *J* = 17.2 Hz, 1H), 2.66 (dd, *J* = 17.2, 5.2 Hz, 1H), 2.48-2.43 (m, 1H), 2.45 (s, 3H), 2.38-2.36 (m, 1H), 2.22 (s, 3H), 2.18 (s, 3H), 1.35 (bs, 1H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 197.9, 153.0, 136.9, 136.4, 135.5, 131.4, 130.3, 130.0, 129.2, 78.7, 77.67,† 77.66,‡ 63.5, 49.3, 48.2, 38.4, 22.9,† 22.8,‡ 20.70,† 20.69,‡ 20.67,† 20.66‡ (†Rotomer A, ‡Rotomer B); **IR** (thin film): 3444, 2925, 1673, 1457, 1371, 1126, 1052, 754 cm^{-1}; **TLC** (50% ethyl acetate:hexanes): R_f = 0.42; **HRMS** (ESI): Calcd. for $C_{19}H_{25}O_3$ ($[M+H]^+$): 301.1804, Found: 301.1799; **SFC** Chiralpak OD, 9% MeOH, pressure = 150 bar, flow rate = 1.5 mL/min, λ = 210 nm, $t_{R\ (minor)}$ 10.1 min, $t_{R\ (major)}$ 12.1 min, 99:1 er; **[α]$_D$** -136 (*c* = 1.5, $CHCl_3$).



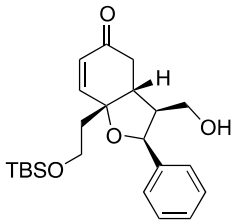
(2*S*,3*S*,3a*R*,7a*R*)-3-(Hydroxymethyl)-7a-methyl-2-(thiophen-2-yl)-2,3,3a,4-tetrahydrobenzofuran-5(7a*H*)-one (5i): The title compound was prepared according to General Procedure C using aldehyde **4i** (95 mg, 0.36 mmol) and $NaBH_4$ (3.4 mg, 0.09 mmol) affording **5i** (83 mg, 87% yield) as a white solid (mp 99-101 ºC). Analytical data for **5i**: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.28-7.26 (m, 1H), 7.00-6.96 (m, 2H), 6.67 (dd, *J* = 10.2 Hz, 1H), 5.97 (d, *J* = 10.2 Hz, 1H), 5.39 (d, *J* = 7.9 Hz, 1H), 3.44 (bs, 2H), 2.74-2.63 (m, 2H), 2.57-2.46 (m, 2H), 1.62 (s, 3H), 1.37 (bs, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 197.2, 152.7, 143.3, 128.3, 126.9, 125.1, 124.4, 79.7, 77.5, 61.8, 49.7, 45.6, 37.3, 23.8; **IR** (thin film): 3430, 2924, 1672, 1374, 1236, 1115, 1027, 708 cm^{-1}; **TLC** (50% ethyl acetate:hexanes): R_f = 0.32; **HRMS** (ESI): Calcd. for $C_{14}H_{16}O_3NaS$ ($[M+Na]^+$): 287.0718, Found: 287.0713; **SFC** Chiralpak OD, 9% MeOH, pressure = 150 bar, flow rate = 1.5 mL/min, λ = 210 nm, $t_{R\ (minor)}$ 16.8 min, $t_{R\ (major)}$ 18.6 min, 97:3 er; **[α]$_D$** -77 (*c* = 1.2, $CHCl_3$).



(2*S*,3*S*,3a*R*,7a*R*)-7a-Ethyl-3-(hydroxymethyl)-2-phenyl-2,3,3a,4-tetrahydrobenzofuran-5(7a*H*)-one (5k): The title compound was prepared according to General Procedure C using aldehyde **4k** (189 mg, 0.70 mmol) and $NaBH_4$ (6.6 mg, 0.18 mmol) affording **5k** (141 mg, 74% yield) as a pale yellow oil. Analytical data for **5k**: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.37-7.26 (m, 5H), 6.72 (dd, *J* = 10.3, 1.8 Hz, 1H), 6.11 (d, *J* = 10.3 Hz, 1H), 5.01 (d, *J* = 8.7 Hz, 1H), 3.27-3.19 (m, 2H), 2.71 (dd, *J* = 17.3, 1.5 Hz, 1H), 2.64 (dd, *J* = 17.4, 1.1 Hz, 1H), 2.52-2.40 (m, 2H), 2.08-1.86 (m, 2H), 1.49 (bs, 1H), 1.16 (t, *J* = 7.5 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 198.1, 152.2, 138.6, 130.0, 128.3, 127.7, 126.4, 81.0, 79.8, 77.2, 62.7, 50.1, 44.3, 38.4, 30.1, 8.1; **IR** (thin film): 3420, 2925, 1683, 1558, 1457, 1027, 703 cm^{-1}; **TLC** (50% ethyl acetate:hexanes): R_f = 0.34; **HRMS** (ESI): Calcd. for $C_{17}H_{20}NaO_3$ ($[M+Na]^+$): 295.1310, Found: 295.1305; **SFC** Chiralpak OD, 9% MeOH, pressure = 150 bar, flow rate = 1.5 mL/min, λ = 210 nm, $t_{R\ (minor)}$ 12.5 min, $t_{R\ (major)}$ 14.6 min, >99.5:0.5 er; **[α]$_D$** -126 (*c* = 1.6, $CHCl_3$).

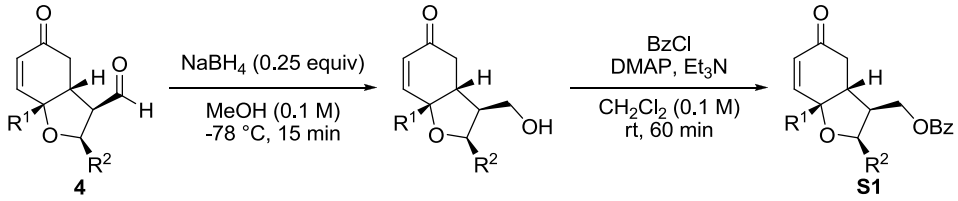


(2*S*,3*S*,3a*R*,7a*R*)-3-(Hydroxymethyl)-7a-isopropyl-2-phenyl-2,3,3a,4-tetrahydrobenzofuran-5(7a*H*)-one (5l): The title compound was prepared according to General Procedure C using aldehyde **4l** (108 mg, 0.38 mmol) and $NaBH_4$ (3.6 mg, 0.09 mmol) affording **5l** (81 mg, 74% yield) as a pale yellow oil. Analytical data for **5l**: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.37-7.27 (m, 5H), 6.69 (dd, *J* = 10.4, 1.4 Hz, 1H), 6.23 (d, *J* = 10.4 Hz, 1H), 4.97 (d, *J* = 8.5 Hz, 1H), 3.32-3.24 (m, 2H), 2.69-2.59 (m, 3H), 2.40-2.32 (m, 1H), 2.27-2.17 (m, 1H), 1.16 (dd, *J* = 12.4, 6.9 Hz, 6H), 0.92 (bs, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 198.2, 150.9, 138.4, 131.5, 128.5, 127.9, 126.3, 82.9, 79.1, 62.7, 51.8, 42.2, 39.8, 35.6, 18.0, 17.1; **IR** (thin film): 3420, 2963, 1683, 1558, 1387, 1265, 1051, 714 cm^{-1}; **TLC** (50% ethyl acetate:hexanes): R_f = 0.41; **HRMS** (ESI): Calcd. for $C_{18}H_{22}NaO_3$ ($[M+Na]^+$): 309.1467, Found: 309.1461; **SFC** Chiralpak OD, 9% MeOH, pressure = 150 bar, flow rate = 1.5 mL/min, λ = 210 nm, $t_{R\ (minor)}$ 11.2 min, $t_{R\ (major)}$ 13.9 min, 99:1 er; **[α]$_D$** -131 (*c* = 2.0, $CHCl_3$).



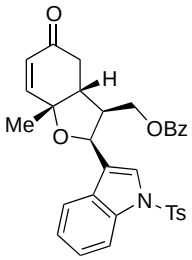
(2*S*,3*S*,3a*R*,7a*R*)-7a-(2-((*tert*-Butyldimethylsilyl)oxy)ethyl)-3-(hydroxymethyl)-2-phenyl-2,3,3a,4-tetrahydrobenzofuran-5(7a*H*)-one (5m): The title compound was prepared according to General Procedure C using aldehyde **4m** (274 mg, 0.68 mmol) and $NaBH_4$ (6.5 mg, 0.17 mmol) affording **5m** (238 mg, 87% yield) as a pale yellow oil. Analytical data for **5m**: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.37-7.27 (m, 5H), 6.72 (dd, *J* = 10.3, 1.8 Hz, 1H), 6.07 (d, *J* = 10.3 Hz, 1H), 5.03 (d, *J* = 9.0 Hz, 1H), 4.02-3.97 (m, 1H), 3.93-3.87 (m, 1H), 3.27 (t, *J* = 5.3 Hz, 2H), 2.78 (dd, *J* = 17.5, 5.6 Hz, 1H), 2.69-2.64 (m, 2H), 2.49-2.41 (m, 1H), 2.22-2.07 (m, 2H), 0.97 (bs, 1H), 0.89 (s, 9H), 0.08 (d, *J* = 3.4 Hz, 6H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 198.0, 152.1, 138.7, 129.6, 128.4, 127.9, 126.5, 80.4, 80.0, 62.5, 58.6, 50.0, 44.9, 40.4, 38.0, 25.8, 18.1, -5.43, -5.44; **IR** (thin film): 3444, 2953, 1671, 1472, 1255, 1091, 836 cm^{-1}; **TLC** (50% ethyl acetate:hexanes): R_f = 0.50; **HRMS** (ESI): Calcd. for $C_{23}H_{34}O_4NaSi$ ($[M+Na]^+$): 425.2124, Found: 425.2119; **SFC** Chiralpak AD, 9% MeOH, pressure = 150 bar, flow rate = 1.5 mL/min, λ = 210 nm, $t_{R\ (major)}$ 7.7 min, $t_{R\ (minor)}$ 9.3 min, 99:1 er; **[α]$_D$** -71 (*c* = 1.0, $CHCl_3$).

General Procedure D for the Conversion of 4h and 4j to S1a and S1b

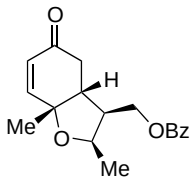


A 10-mL round-bottom flask equipped with a magnetic stir bar was charged with aldehyde **4** (1.00 equiv) in MeOH (0.1 M). The solution was cooled to -78 ºC. $NaBH_4$ (0.25 equiv) was added and the reaction was allowed to stir for 15 min at -78 ºC. The reaction was quenched with sat. aq. NH_4Cl (2 mL) at -78 ºC and allowed to warm to room temperature. After stirring at room temperature for 30 min, the reaction was partitioned between CH_2Cl_2 (15 mL) and H_2O (30 mL). The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (2 x 15 mL). The combined organic extracts were dried over Na_2SO_4, filtered, and concentrated *in vacuo* to afford the crude alcohol, which was used without further purification.

A flame-dried 10-mL round-bottom flask equipped with a magnetic stir bar was charged with the crude alcohol (1.00 equiv) in CH_2Cl_2 (0.1 M). 4-Dimethylaminopyridine (0.10 equiv), benzoyl chloride (1.20 equiv), and triethylamine (3.00 equiv) were added sequentially. The reaction was allowed to stir for 60 min at room temperature. The reaction was quenched with sat. aq. NH_4Cl (2 mL). The reaction was partitioned between CH_2Cl_2 (15 mL) and H_2O (30 mL). The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (2 x 15 mL). The combined organic extracts were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by column chromatography on silica gel to afford benzoate **S1**.

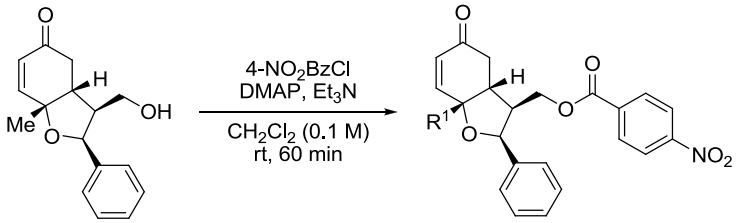


((2*S*,3*S*,3a*R*,7a*R*)-7a-Methyl-5-oxo-2-(1-tosyl-1*H*-indol-3-yl)-2,3,3a,4,5,7a-hexahydrobenzofuran-3-yl)methyl benzoate (S1a): The title compound was prepared according to General Procedure D using aldehyde **4h** (300 mg, 0.67 mmol) affording **S1a** (193 mg, 52% yield) as a viscous pale yellow oil. Analytical data for **S1a**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.92 (d, *J* = 8.4 Hz, 1H), 7.67 (d, *J* = 8.4 Hz, 2H), 7.64 (s, 1H), 7.54 (d, *J* = 7.1 Hz, 2H), 7.48-7.45 (m, 2H), 7.31-7.27 (m, 3H), 7.16 (t, *J* = 7.5 Hz, 1H), 7.08 (d, *J* = 8.2 Hz, 2H), 6.70 (dd, *J* = 10.3, 1.7 Hz, 1H), 6.14 (d, *J* = 10.3 Hz, 1H), 5.29 (d, *J* = 8.4 Hz, 1H), 4.04 (dd, *J* = 11.3, 7.4 Hz, 1H), 3.90 (dd, *J* = 11.3, 6.8 Hz, 1H), 2.81-2.72 (m, 3H), 2.49-2.47 (m, 1H), 2.23 (s, 3H), 1.67 (s, 3H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 196.9, 165.8, 151.8, 144.9, 135.0, 134.7, 132.8, 130.2, 129.7, 129.2, 128.9, 128.2, 126.5, 124.9, 123.8, 123.7, 123.4, 119.7, 119.5, 113.8, 78.8, 73.8, 64.74, 64.68,† 64.6,‡ 48.3, 46.7, 38.2, 23.5, 21.42,† 21.39‡ (†Rotomer A, ‡Rotomer B); **IR** (thin film): 2974, 1716, 1683, 1372, 1271, 1174, 1120, 750 cm^{-1}; **TLC** (40% ethyl acetate:hexanes): R_f = 0.26; **HRMS** (ESI): Calcd. for $C_{64}H_{58}N_2NaO_{12}S_2$ ($[2M+Na]^+$): 1133.3330, Found: 1133.3330; **SFC** Chiralpak AD, 10% MeOH, pressure = 150 bar, flow rate = 3.0 mL/min, λ = 210 nm, $t_{R\ (major)}$ 18.8 min, $t_{R\ (minor)}$ 27.4 min, >99.5:0.5 er; **$[\alpha]_D$** -52 (*c* = 1.6, $CHCl_3$).



((2*R*,3*S*,3a*R*,7a*R*)-2,7a-Dimethyl-5-oxo-2,3,3a,4,5,7a-hexahydrobenzofuran-3-yl)methyl benzoate (S1b): The title compound was prepared according to General Procedure D using aldehyde **4j** (90 mg, 0.46 mmol) affording **S1b** (32 mg, 23% yield) as a pale yellow oil. Analytical data for **S1b**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 8.02 (dd, *J* = 8.4, 1.1 Hz, 2H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.47-7.44 (m, 2H), 6.62 (dd, *J* = 10.3, 1.8 Hz, 1H), 6.01 (dd, *J* = 10.3, 0.7 Hz, 1H), 4.41 (dd, *J* = 11.2, 6.9 Hz, 1H), 4.30 (d, *J* = 11.2, 7.0 Hz, 1H), 4.22-4.17 (m, 1H), 2.75 (dd, *J* = 17.2, 1.0 Hz, 1H), 2.67 (dd, *J* = 17.2, 5.4 Hz, 1H), 2.51-2.46 (m, 1H), 2.35-2.32 (m, 1H), 1.51 (s, 3H), 1.28 (d, *J* = 6.6 Hz, 3H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 197.3, 166.4, 152.9, 133.2, 129.6, 129.5, 128.9, 128.5, 78.3, 74.31,† 74.28,‡ 64.3, 47.6, 45.3, 37.9, 24.1, 16.7 (†Rotomer A, ‡Rotomer B); **IR** (thin film): 2973, 1717, 1684, 1456, 1273, 1114, 713 cm^{-1}; **TLC** (40% ethyl acetate:hexanes): R_f = 0.31; **HRMS** (ESI): Calcd. for $C_{18}H_{20}NaO_4$ ([M+Na]$^+$): 323.1260, Found: 323.1252; **SFC** Chiralpak AD, 9% MeOH, pressure = 150 bar, flow rate = 1.5 mL/min, λ = 210 nm, $t_{R\ (major)}$ 6.4 min, $t_{R\ (minor)}$ 8.1 min, 99.5:0.5 er; **[α]$_D$** -9 (*c* = 1.4, $CHCl_3$).

Synthesis of Benzoate 6 from 5a



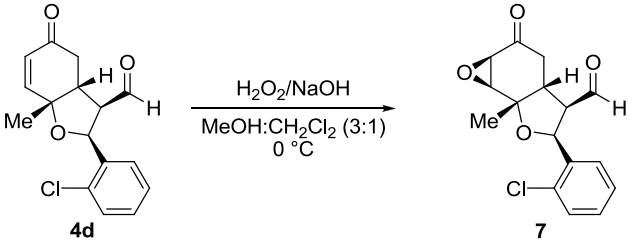
5a **6**

((2*S*,3*S*,3a*R*,7a*R*)-7a-Methyl-5-oxo-2-phenyl-2,3,3a,4,5,7a-hexahydrobenzofuran-3-yl)methyl 4-nitrobenzoate (6): A flame-dried 10-mL round-bottom flask equipped with a magnetic stir bar was charged with alcohol **5a** (77 mg, 0.30 mmol, 1.00 equiv) in CH_2Cl_2 (3 mL, 0.1 M). 4-Dimethylaminopyridine (3.7 mg, 0.03 mmol, 0.10 equiv), 4-nitrobenzoyl chloride (67 mg, 0.36 mmol, 1.20 equiv), and triethylamine (125 μL, 0.90 mmol, 3.00 equiv) were added sequentially. The reaction was allowed to stir for 60 min at room temperature. The reaction was quenched with sat. aq. NH_4Cl (2 mL). The reaction was partitioned between CH_2Cl_2 (15 mL) and H_2O (30 mL). The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (2 x 15 mL). The combined organic extracts were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by column chromatography on silica gel to afford benzoate **6** (102 mg, 83% yield) as a white solid (mp 125-126 ºC). Analytical data for **6**: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 8.15 (d, *J* = 8.9 Hz, 2H), 7.90 (d, *J* = 8.8 Hz, 2H), 7.26-7.10 (m, 5H), 6.64 (dd, *J* = 10.2, 1.8 Hz, 1H), 5.99 (d, *J* = 10.2 Hz, 1H), 5.03 (d, *J* = 8.8 Hz, 1H), 3.95-3.83 (m, 2H), 2.74-2.64 (m, 3H), 2.38-2.35 (m, 1H), 1.58 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 196.8, 164.0, 152.4, 150.5, 137.8, 134.9, 130.5, 129.3, 128.3, 128.0, 126.5, 123.5, 80.2, 79.2, 65.6, 47.8, 46.7, 38.0, 23.6; **IR** (thin film): 2975, 1725, 1683, 1529, 1371, 1279, 1171, 1133 cm^{-1}; **TLC** (40% ethyl acetate:hexanes): R_f = 0.33; **HRMS** (ESI): Calcd. for $C_{46}H_{42}N_2NaO_{12}$ ([2M+Na]$^+$): 837.2636, Found: 837.2626; **[α]$_D$** -50 (*c* = 0.5, $CHCl_3$)**.**

X-ray suitable crystals were grown by dissolving **6** (100 mg) in a minimal amount of acetone (~0.2 mL) in a 20-mL scintillation vial. Without disturbing the acetone layer, hexanes (~4 mL) was carefully

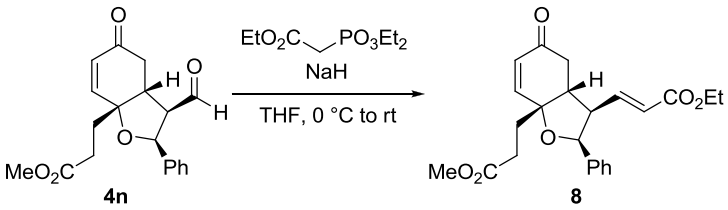
pipetted on top to form a second layer. The vial was capped and carefully transferred to a freezer (-10 ºC) where it was left to age overnight.

Synthesis of Epoxide 7 from 4d



(1a*R*,3a*R*,4*R*,5*S*,6a*S*,6b*S*)-5-(2-Chlorophenyl)-6a-methyl-2-oxooctahydrooxireno[2,3-*g*]benzofuran-4-carbaldehyde (7): A 10-mL round-bottom flask equipped with a magnetic stir bar was charged with aldehyde **4d** (116 mg, 0.40 mmol, 1.0 equiv) in MeOH:CH_2Cl_2 (3:1) (1.6 mL, 0.25 M). The solution was cooled to 0 ºC. H_2O_2 (30 wt. % in H_2O) (0.8 mL) and NaOH (20 wt. % in H_2O) (0.2 mL) were sequentially added. The reaction was allowed to at 0 ºC for 12 h. The reaction was carefully quenched with sat. aq. $Na_2S_2O_3$ (5 mL) to remove excess peroxides. The reaction was partitioned between CH_2Cl_2 (15 mL) and H_2O (30 mL). The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (2 x 15 mL). The combined organic extracts were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by column chromatography on silica gel to afford epoxide **7** (117 mg, 95% yield, >20:1 dr) as a white solid (mp 73 ºC). Analytical data for **7**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 9.03 (d, *J* = 1.4 Hz, 1H), 7.47 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.36 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.30-7.24 (m, 2H), 5.54 (d, *J* = 8.3 Hz, 1H), 3.50 (dd, *J* = 3.7, 1.8 Hz, 1H), 3.41 (dd, *J* = 3.7, 0.7 Hz, 1H), 3.33-3.31 (m, 1H), 3.13-3.10 (m, 1H), 3.07 (dd, *J* = 13.8, 5.3 Hz, 1H), 2.07 (ddd, *J* = 13.8, 2.6, 0.9 Hz, 1H), 1.77 (s, 3H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 206.0, 197.9, 134.1, 131.3, 129.5, 129.4, 127.4, 127.2, 78.2, 76.8, 64.3, 58.7, 55.7, 46.1, 35.3, 23.0; **IR** (thin film): 2978, 1720, 1684, 1653, 1541, 1473, 1375, 1127, 1034 cm^{-1}; **TLC** (20% ethyl acetate:hexanes): R_f = 0.28; **HRMS** (ESI): Calcd. for $C_{32}H_{30}Cl_2NaO_8$ ($[2M+Na]^+$): 635.1216, Found: 635.1217; **$[\alpha]_D$** -104 (*c* = 1.3, $CHCl_3$).

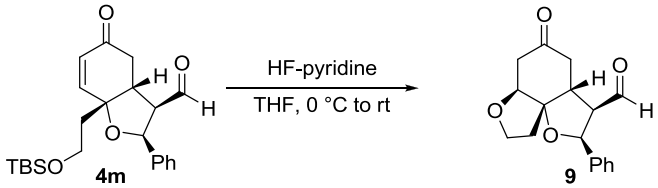
Synthesis of Diester 8 from 4n



(*E*)-Ethyl 3-((2*S*,3*R*,3a*R*,7a*R*)-7a-(3-methoxy-3-oxopropyl)-5-oxo-2-phenyl-2,3,3a,4,5,7a-hexahydrobenzofuran-3-yl)acrylate (8): A flame-dried 10-mL round-bottom flask equipped with a magnetic stir bar was charged with NaH (60%) (23 mg, 0.57 mmol, 1.25 equiv) suspended in THF (2

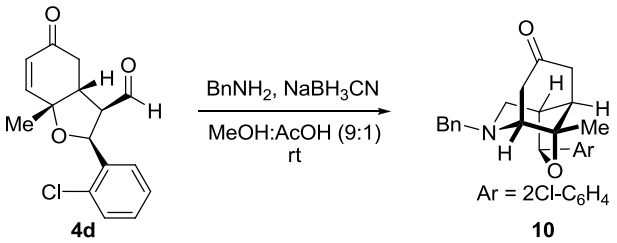
mL). The suspension was cooled to 0 ºC. Triethyl phosphonoacetate (115 μL, 0.57 mmol, 1.25 equiv) was added dropwise. The homogenous solution was allowed to stir at 0 ºC for 20 min before a solution of aldehyde **4n** (150 mg, 0.46 mmol, 1.00 equiv) in THF (0.5 mL) was added dropwise. The ice bath was removed and the resulting solution was allowed to stir for 3 h as it slowly warmed to room temperature. The reaction was cooled to 0 ºC and quenched with sat. aq. NH_4Cl (5 mL). The reaction was diluted with Et_2O (30 mL) and washed with H_2O (15 mL) and brine (15 mL). The organic layer was dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude residue was purified by column chromatography on silica gel to afford diester **8** (176 mg, 96% yield, >20:1 *E*:*Z*) as a pale yellow oil. Analytical data for **8**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.31 (t, *J* = 7.4 Hz, 2H), 7.26-7.23 (m, 1H), 7.17 (d, *J* = 7.4 Hz, 2H), 6.78 (dd, *J* = 10.3, 2.0 Hz, 1H), 6.12-6.08 (m, 2H), 5.65 (d, *J* = 15.5 Hz, 1H), 5.13 (d, *J* = 9.1 Hz, 1H), 4.10-3.99 (m, 2H), 3.70 (s, 3H), 3.06 (q, *J* = 9.7 Hz, 1H), 2.74-2.61 (m, 3H), 2.49 (d, *J* = 17.8 Hz, 1H), 2.46-2.43 (m, 1H), 2.38-2.26 (m, 2H), 1.17 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 196.3, 173.4, 165.4, 150.9, 145.7, 138.0, 130.1, 128.4, 127.9, 126.3, 123.2, 81.7, 80.9, 60.3, 52.1, 51.9, 47.2, 36.1, 32.4, 28.6, 14.0; **IR** (thin film): 1734, 1716, 1683, 1456, 1387, 1248, 1175, 1028 cm^{-1}; **TLC** (50% ethyl acetate:hexanes): R_f = 0.50; **HRMS** (ESI): Calcd. for $C_{23}H_{26}NaO_6$ ($[M+Na]^+$): 421.1627, Found: 421.1622; **SFC** Chiralpak OD, 9% MeOH, pressure = 150 bar, flow rate = 1.5 mL/min, λ = 210 nm, $t_{R\ (major)}$ 9.4 min, $t_{R\ (minor)}$ 10.4 min, >99.5:0.5 er; **$[\alpha]_D$** -17 (*c* = 1.6, $CHCl_3$).

Synthesis of bis(Tetrahydrofuran) 9 from 4m



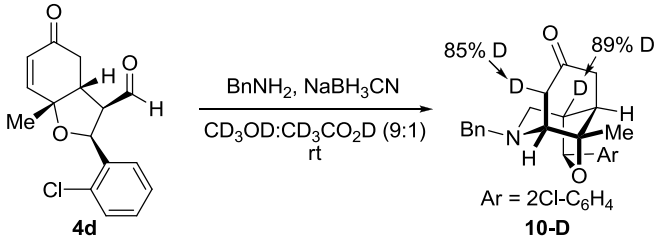
(3a*S*,5*S*,6*R*,6a*R*,9a*S*)-8-Oxo-5-phenyloctahydro-2*H*-benzo[1,2-*b*:2,3-*b'*]difuran-6-carbaldehyde (9): A 20-mL Nalgene® scintillation vial equipped with a magnetic stir bar was charged with silyl ether **4m** (400 mg, 1.00 mmol, 1.00 equiv) in THF (10 mL, 0.1 M). The solution was cooled to 0 ºC. HF-pyridine (70% HF) (4 mL) was slowly added to the reaction. The ice bath was removed and the resulting solution was allowed to stir for 3 h as it slowly warmed to room temperature. The reaction was quenched by carefully pipetting the reaction mixture into a beaker containing sat. aq. $NaHCO_3$ (60 mL). The biphasic solution was diluted with H_2O (40 mL) and extracted with CH_2Cl_2 (3 x 40 mL). The combined organic extracts were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by column chromatography on silica gel to afford tricycle **9** (235 mg, 82% yield, >20:1 dr) as a pale yellow oil. Analytical data for **9**: **^{1}H NMR** (400 MHz, $CDCl_3$): δ 9.05 (s, 1H), 7.38-7.34 (m, 2H), 7.31-7.28 (m, 3H), 5.47 (d, *J* = 8.5 Hz, 1H), 4.17 (t, *J* = 5.4 Hz, 1H), 4.07 (dt, *J* = 8.6, 3.1 Hz, 1H), 3.98 (q, *J* = 8.9 Hz, 1H), 3.20 (q, *J* = 6.0 Hz, 1H), 3.09 (t, *J* = 7.2 Hz, 1H), 2.70-2.57 (m, 3H), 2.55-2.49 (m, 1H), 2.43-2.34 (m, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$): δ 207.3, 198.8, 136.7, 128.8, 128.4, 125.9, 89.1, 80.4, 80.1, 66.5, 60.6, 43.1, 40.8, 38.9, 38.3; **IR** (thin film): 1716, 1653, 1636, 1541, 1457, 1397, 1209, 1065 cm^{-1}; **TLC** (40% ethyl acetate:hexanes): R_f = 0.25; **HRMS** (ESI): Calcd. for $C_{17}H_{18}NaO_4$ ($[M+Na]^+$): 309.1103, Found: 309.1100; **$[\alpha]_D$** -72 (*c* = 1.2, $CHCl_3$)**.**

Synthesis of Tricycle 10 from 4d



(2*S*,3*R*,3a*R*,7*R*,7a*S*)-8-Benzyl-2-(2-chlorophenyl)-7a-methylhexahydro-7,3-(epiminomethano)benzofuran-5(6*H*)-one (10): A 10-mL round-bottom flask equipped with a magnetic stir bar was charged with aldehyde **4d** (116 mg, 0.40 mmol, 1.00 equiv) and benzylamine (86 mg, 0.80 mmol, 2.00 equiv) in MeOH:AcOH (9:1) (4 mL, 0.1 M). The solution was allowed to stir at room temperature for 4 h. $NaBH_3CN$ (50 mg, 0.80 mmol, 2.00 equiv) was added in one portion resulting in vigorous gas formation. After stirring at room temperature for 30 min, the reaction was quenched with sat. aq. $NaHCO_3$ (4 mL). The reaction was partitioned between CH_2Cl_2 (15 mL) and sat. aq. $NaHCO_3$ (30 mL). The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (2 x 15 mL). The combined organic extracts were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by column chromatography on silica gel to afford tricycle **10** (136 mg, 89% yield, >20:1 dr) as a pale yellow oil. Analytical data for **10**: **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.59 (d, *J* = 7.7 Hz, 1H), 7.44 (d, *J* = 7.2 Hz, 2H), 7.34-7.28 (m, 4H), 7.27-7.24 (m, 1H), 7.20 (dt, *J* = 7.7, 1.4 Hz, 1H), 5.48 (s, 1H), 3.80 (dd, *J* = 22.4, 13.7 Hz, 2H), 3.17 (bs, 1H), 2.93-2.88 (m, 2H), 2.61-2.58 (m, 3H), 2.43 (t, *J* = 3.8 Hz, 1H), 2.30 (dd, *J* = 17.3, 3.8 Hz, 1H), 2.29-2.27 (m, 1H), 1.71 (s, 3H); **^{13}C NMR** (151 MHz, $CDCl_3$): δ 209.5, 141.0, 138.3, 131.3, 129.3, 128.4, 128.3, 128.0, 127.0, 126.4, 80.7, 79.7, 62.4, 57.1, 47.7, 46.9, 40.3, 38.7, 38.5, 22.0; **IR** (thin film): 2905, 2813, 1707, 1558, 1457, 1338, 1121, 751 cm^{-1}; **TLC** (20% ethyl acetate:hexanes): R_f = 0.28; **HRMS** (ESI): Calcd. for $C_{23}H_{27}ClNO_2$ ($[M+H]^+$): 382.1575, Found: 382.1568; **$[\alpha]_D$** +38 (*c* = 1.1, $CHCl_3$).

Deuterium Labeling Experiment and Structure Determination



A 10-mL round-bottom flask equipped with a magnetic stir bar was charged with aldehyde **4d** (58 mg, 0.20 mmol, 1.00 equiv) and benzylamine (43 mg, 0.40 mmol, 1.00 equiv) in CD_3OD:CD_3CO_2D (9:1) (2 mL, 0.1 M). The solution was allowed to stir at room temperature for 4 h. $NaBH_3CN$ (25 mg, 0.40 mmol, 2.00 equiv) was added in one portion resulting in vigorous gas formation. After stirring at room temperature for 30 min, the reaction was quenched with sat. aq. $NaHCO_3$ (2 mL). The reaction was partitioned between CH_2Cl_2 (15 mL) and sat. aq. $NaHCO_3$ (30 mL). The layers were separated

and the aqueous layer was extracted with CH_2Cl_2 (2 x 15 mL). The combined organic extracts were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The crude residue was purified by column chromatography on silica gel to afford tricycle **10** (64 mg, 84% yield, >20:1 dr) as a pale yellow oil.

Analysis: Based on deuterium incorporation α-aldehyde, it can be assumed that under the acidic conditions epimerization occurs via enamine/iminum tautomerization. This observation is also consistent with experimental observations related to the time required for condensation/epimerization to occur. When the aldehyde and amine are premixed for shorter periods of time before $NaBH_3CN$ is added, the yield of **10** is lower with the remaining mass being the formation of **5d** via direct reduction of the aldehyde. Deuterium incorporation α-ketone is consistent with protonation following aza-Michael addition, with protonation of the enol from the convex face preferentially. 2D-NMR analysis showed strong nOe interactions consistent with the proposed structure (see p. S56).

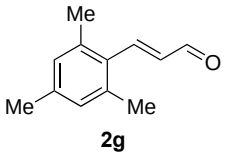
References

1) M. C. Carreño, M. González-López, A. Urbano, *Angew. Chem*. **2006**, *118*, 2803–2807; *Angew. Chem. Int. Ed*. **2006**, *45*, 2737–2741.

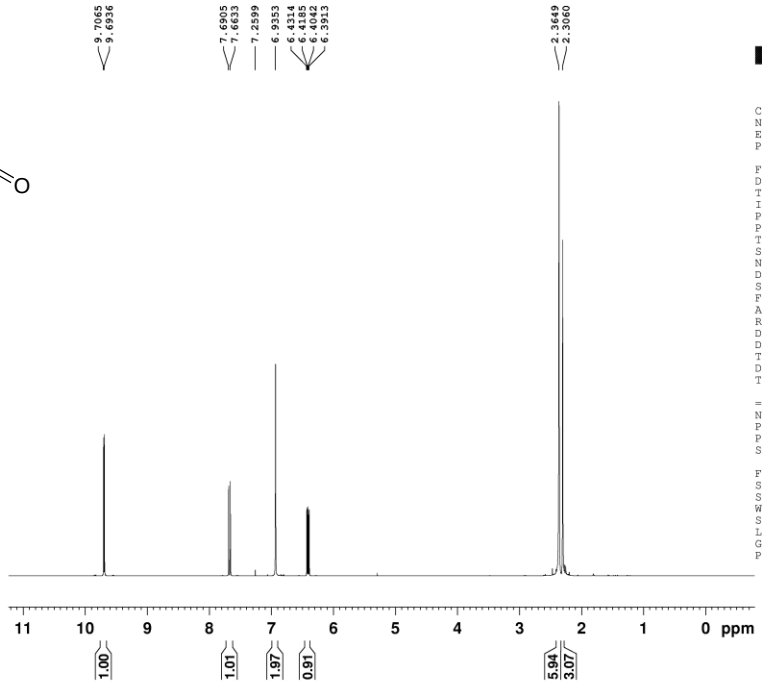
2) P. D. Brown, A. C. Willis, M. S. Sherburn, A. L. Lawrence, *Org. Lett*. **2012**, *14*, 4537–4539.

3) N. T. Patil, V. Singh, *Chem. Comm*. **2011**, *47*, 11116–11118.

4) M. Marigo, T. C. Wabnitz, D. Fielenbach, K. A. Jørgensen, *Angew. Chem*. **2005**, *117*, 804–807; *Angew. Chem. Int. Ed*. **2005**, *44*, 794–797.



2g

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DS                    0
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FIDRES         0.188225 Hz
AQ             2.6564426 sec
RG                 14.2
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TD0                    1

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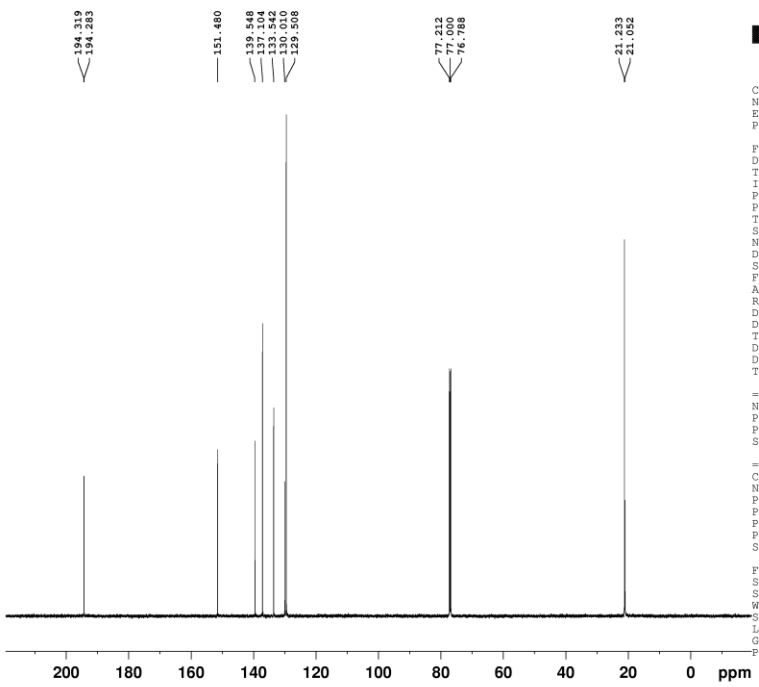


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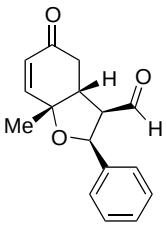

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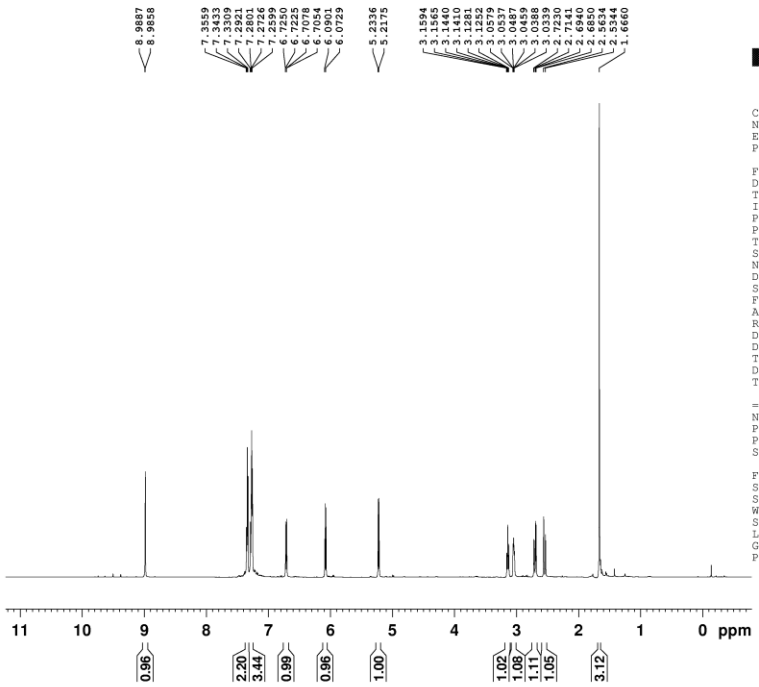

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TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           12335.526 Hz
FIDRES        0.188225 Hz
AQ            2.6564426 sec
RG                   32
DW              40.533 usec
DE               6.50 usec
TE              295.0 K
D1           1.00000000 sec
TD0                   1

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

F2 - Processing parameters
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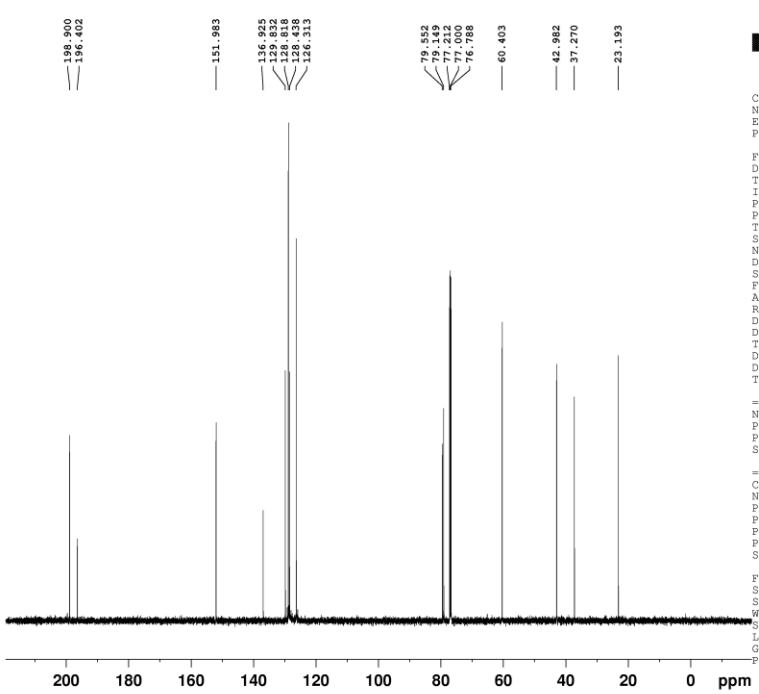

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PROCNO                 1

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TD                65536
SOLVENT           CDCl3
NS                   65
DS                    0
SWH           36057.691 Hz
FIDRES        0.550197 Hz
AQ            0.9088159 sec
RG                  203
DW              13.867 usec
DE               10.00 usec
TE              294.9 K
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======== CHANNEL f1 ========
NUC1                 13C
P1                10.50 usec
PLW1       110.00000000 W
SFO1        150.9178981 MHz

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

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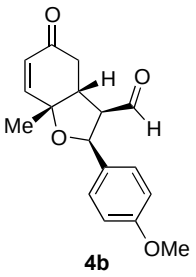

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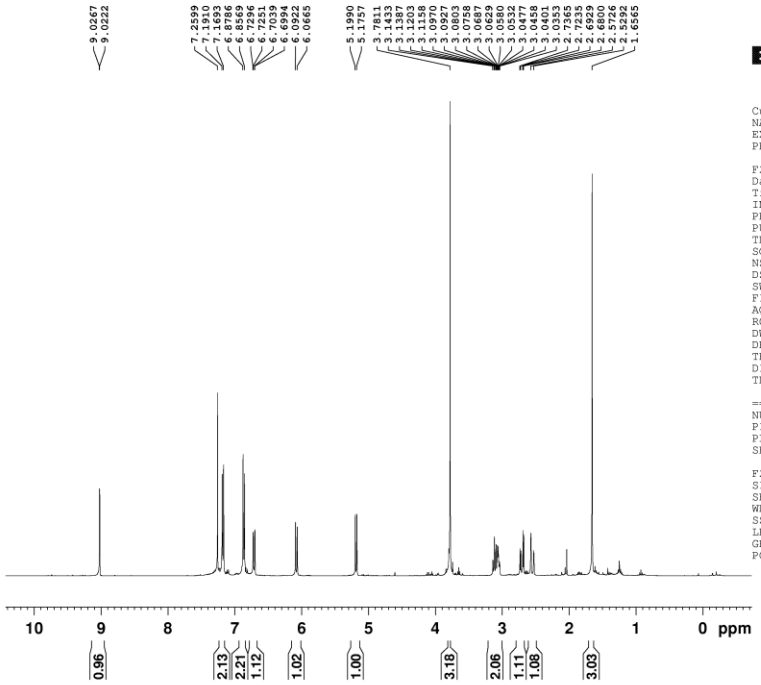

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PROCNO                 1

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PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
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FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                 25.11
DW                60.800 usec
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TE                297.2 K
D1             1.00000000 sec
TD0                    1

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

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SF            400.0900118 MHz
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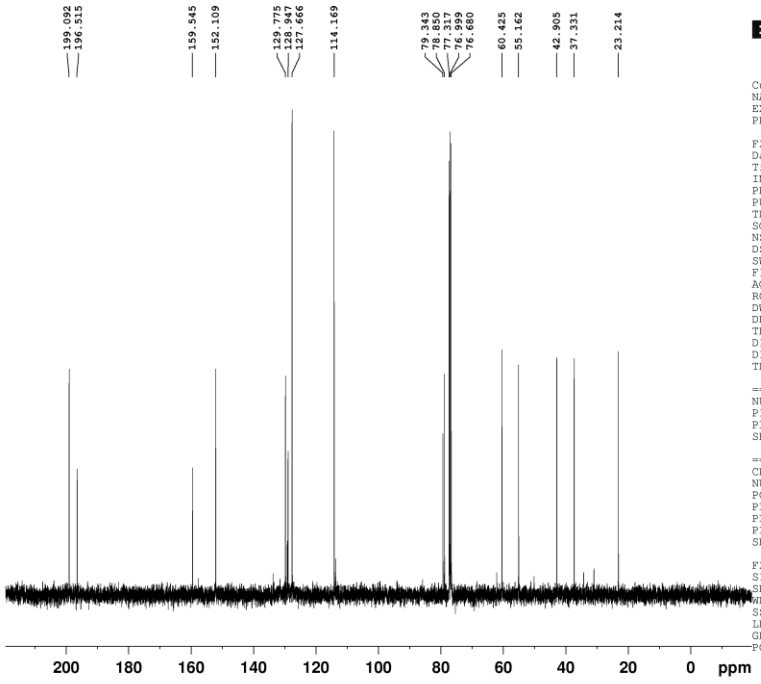

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PROCNO                 1

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PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    55
DS                     0
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                200.09
DW                20.800 usec
DE                 6.50 usec
TE                297.7 K
D1             2.00000000 sec
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P1                 7.50 usec
PLW1          61.20000076 W
SFO1         100.6127703 MHz

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

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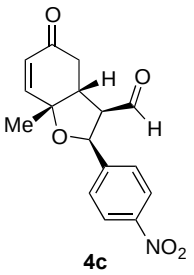

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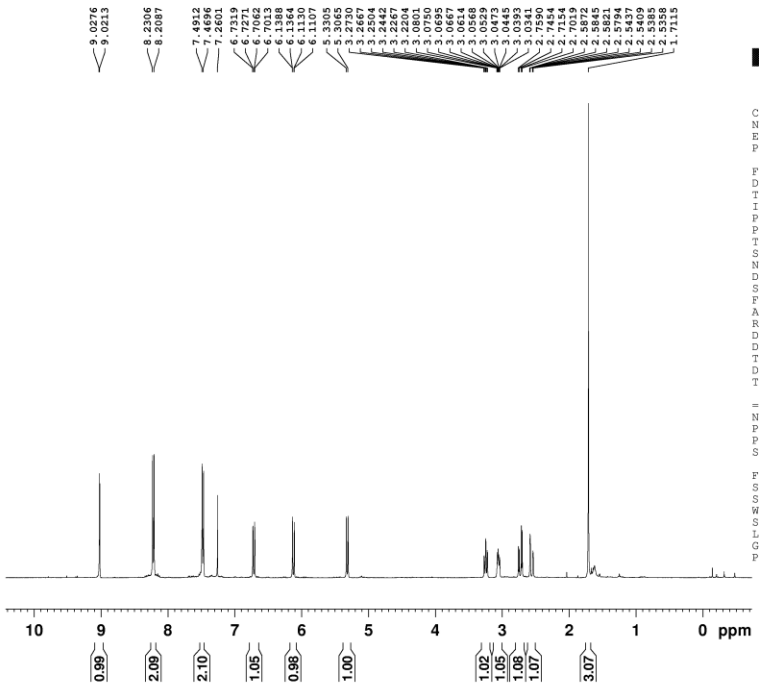

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TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
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TD0                    1

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PLW1          11.19999981 W
SFO1          400.0924707 MHz

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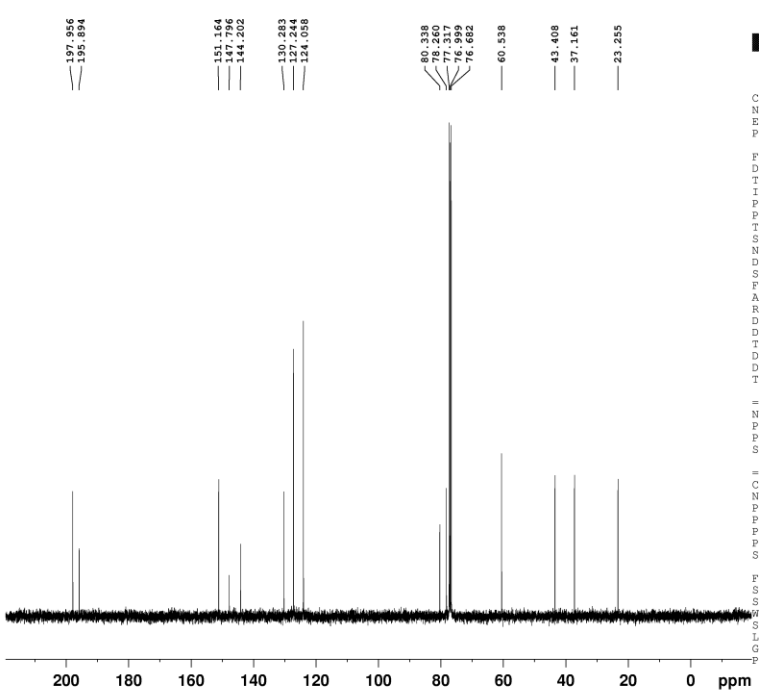

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SOLVENT            CDCl3
NS                   100
DS                     0
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FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                200.09
DW                20.800 usec
DE                 6.50 usec
TE                298.7 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

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

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

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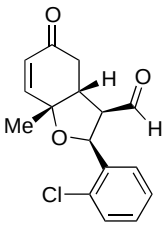

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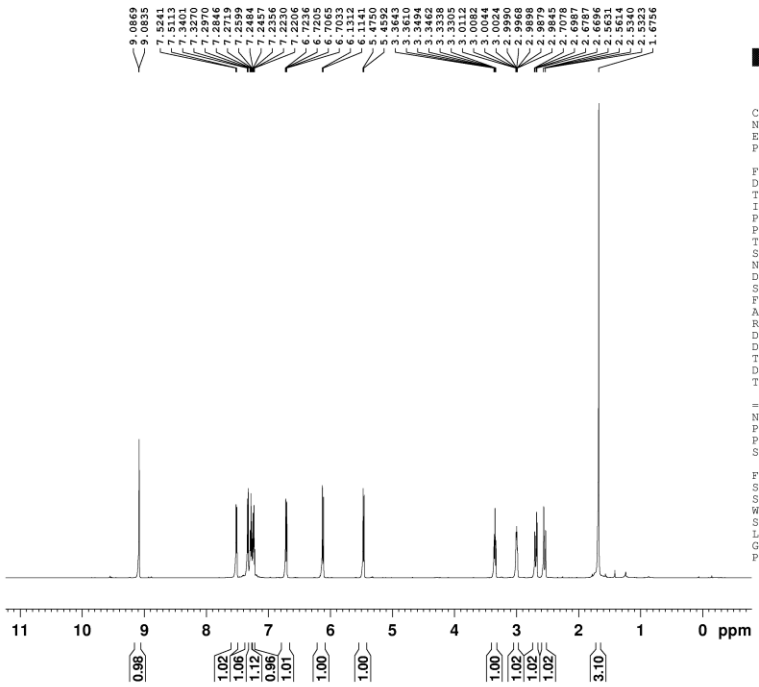

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FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                 20.2
DW               40.533 usec
DE               10.00 usec
TE               295.0 K
D1            1.00000000 sec
TD0                    1

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

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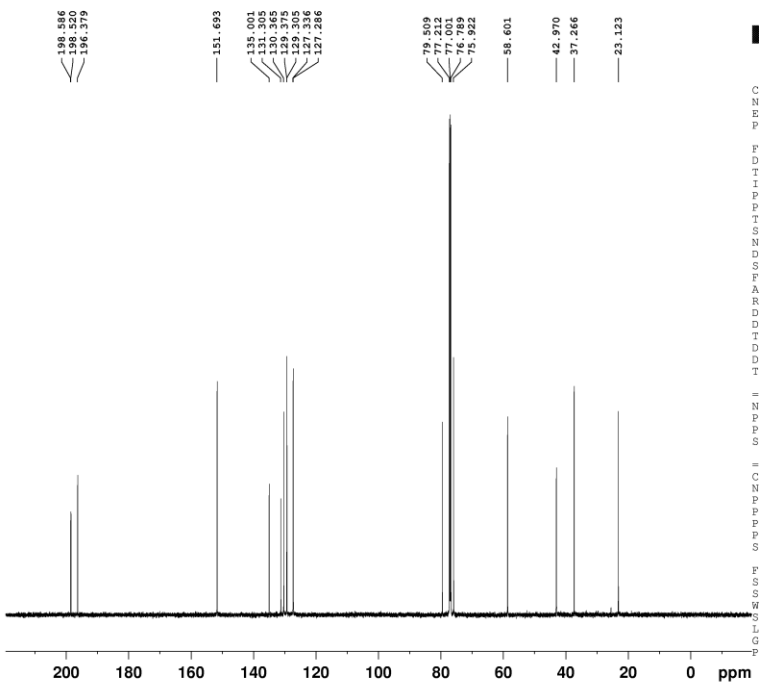

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TE               295.0 K
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D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
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P1                 11.00 usec
PLW1        230.00000000 W
SFO1        150.9178981 MHz

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NUC2                  1H
PCPD2              90.00 usec
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PLW12         0.25077000 W
PLW13         0.20313001 W
SFO2        600.1324005 MHz

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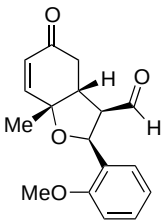

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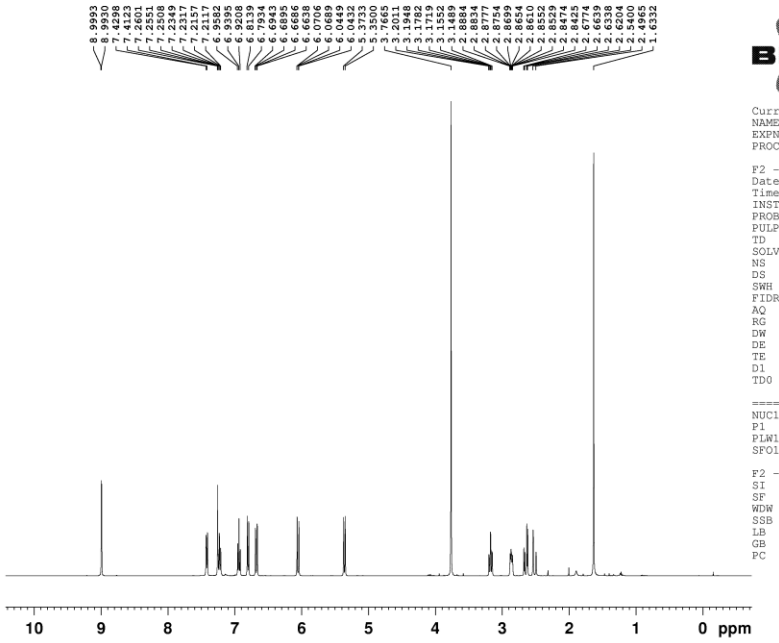


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TD                 65536
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FIDRES          0.125483 Hz
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RG                 32.04
DW                60.800 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1

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

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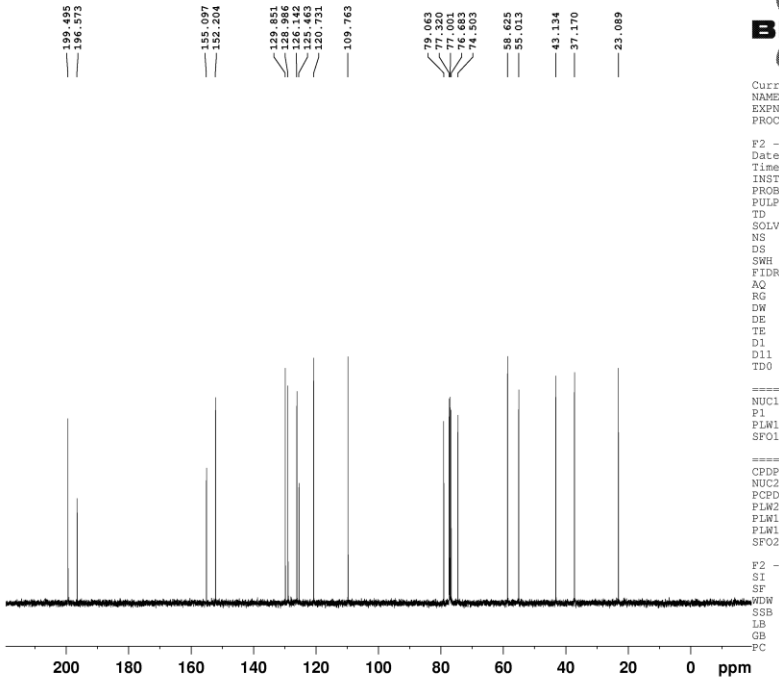


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DS                     0
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FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                200.09
DW                20.800 usec
DE                 6.50 usec
TE                298.8 K
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D11            0.03000000 sec
TD0                    1

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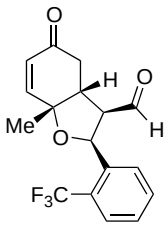
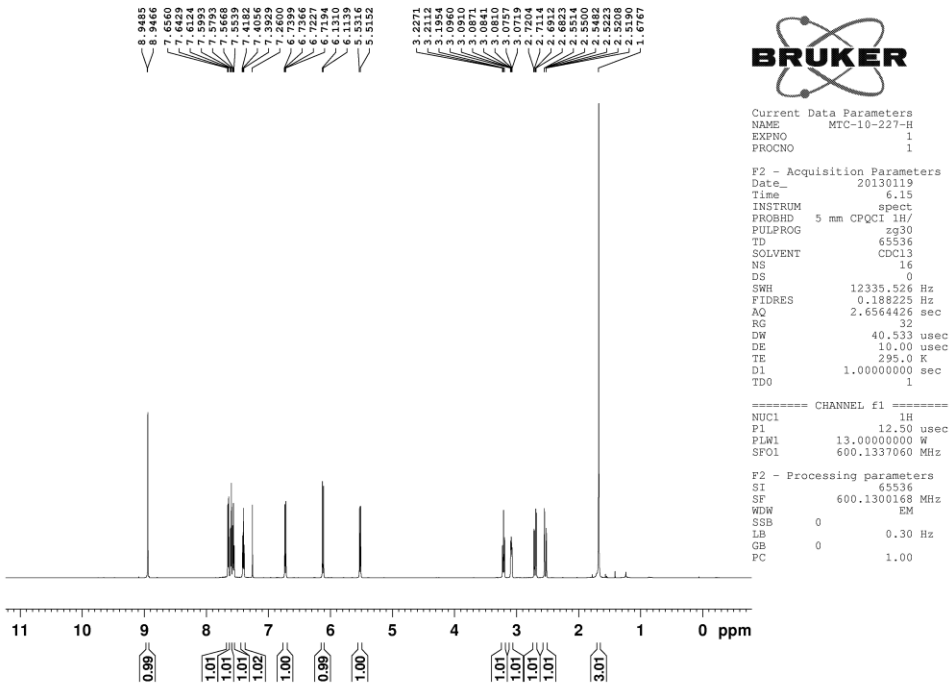
P1                 7.50 usec
PLW1         61.20000076 W
SFO1        100.6127703 MHz

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

F2 - Processing parameters
SI                 32768
SF          100.6027274 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



4f



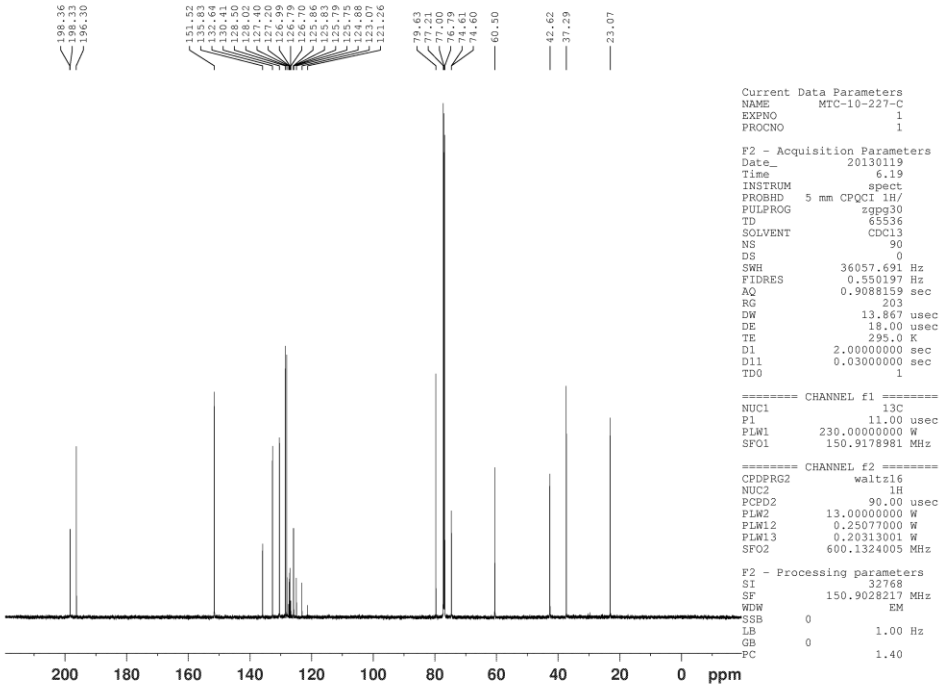
BRUKER

```
Current Data Parameters
NAME        MTC-10-227-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130119
Time                6.15
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                   32
DW               40.533 usec
DE               10.00 usec
TE                295.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                12.50 usec
PLW1        13.00000000 W
SFO1       600.1337060 MHz

F2 - Processing parameters
SI                65536
SF           600.1300168 MHz
WDW                  EM
SSB       0
LB              0.30 Hz
GB        0
PC                1.00
```



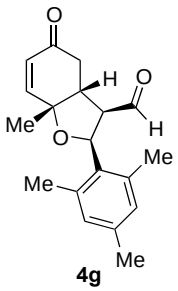

```
Current Data Parameters
NAME        MTC-10-227-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130119
Time                6.19
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   90
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9088159 sec
RG                  203
DW               13.867 usec
DE                18.00 usec
TE                295.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

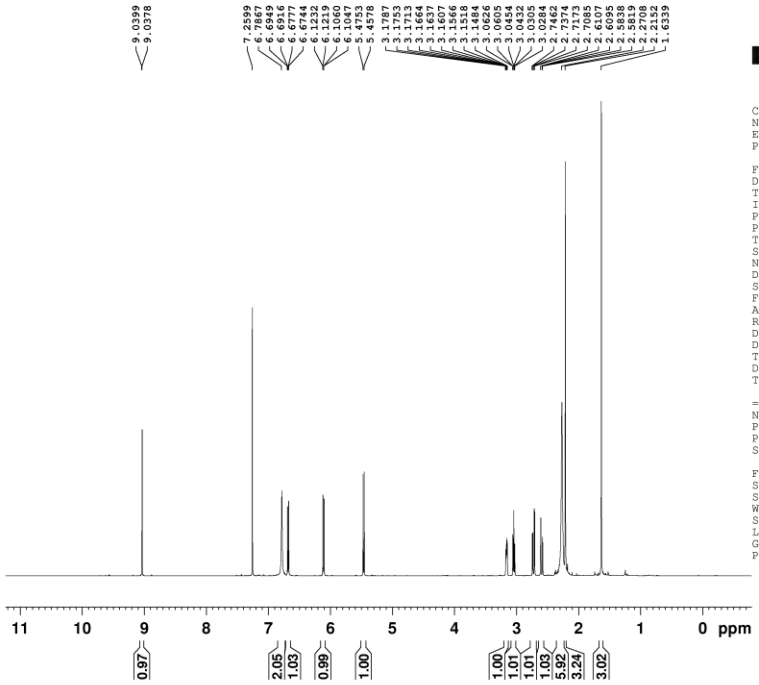
======== CHANNEL f1 ========
NUC1                13C
P1                11.00 usec
PLW1       230.00000000 W
SFO1       150.9178981 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2        13.00000000 W
PLW12        0.25077000 W
PLW13        0.20313001 W
SFO2       600.1324005 MHz

F2 - Processing parameters
SI                32768
SF           150.9028217 MHz
WDW                  EM
SSB       0
LB              1.00 Hz
GB        0
PC                1.40
```



4g

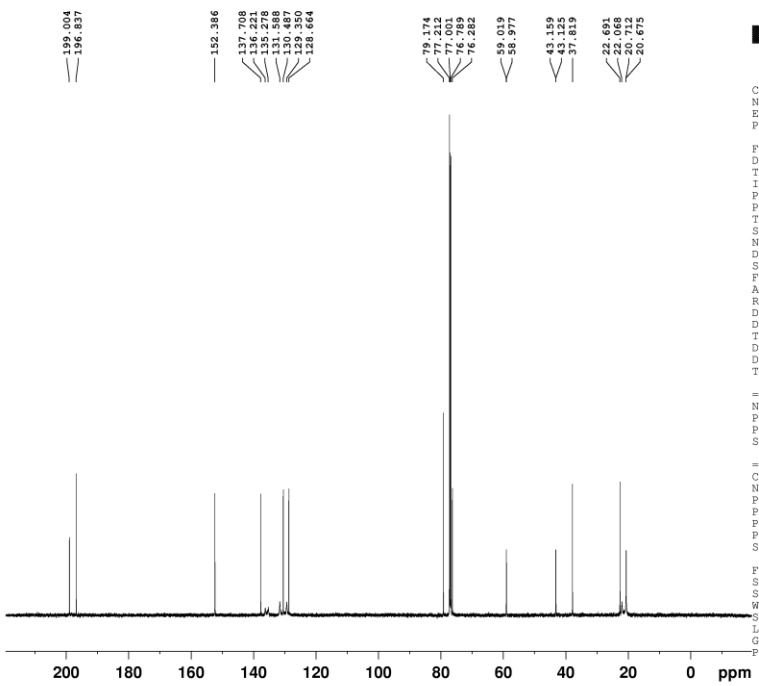


```
Current Data Parameters
NAME      MTC-10-233-Major-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130120
Time               10.44
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                 14.2
DW               40.533 usec
DE               10.00 usec
TE               295.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.50 usec
PLW1         13.00000000 W
SFO1        600.1337060 MHz

F2 - Processing parameters
SI                 65536
SF           600.1300164 MHz
WDW                   EM
SSB       0
LB             0.30 Hz
GB        0
PC                 1.00
```



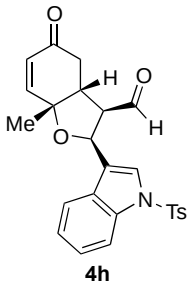
```
Current Data Parameters
NAME      MTC-10-233-Major-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130120
Time               10.49
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   65
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9088159 sec
RG                  203
DW               13.867 usec
DE               18.00 usec
TE               295.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

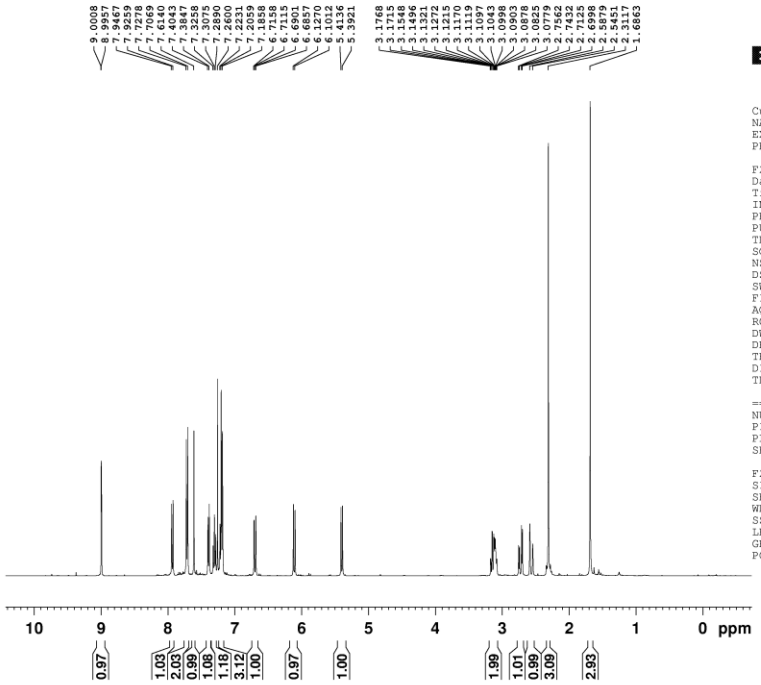
======== CHANNEL f1 ========
NUC1                  13C
P1                 11.00 usec
PLW1        230.00000000 W
SFO1        150.9178981 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2         13.00000000 W
PLW12         0.25077000 W
PLW13         0.20313001 W
SFO2        600.1324005 MHz

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



4h



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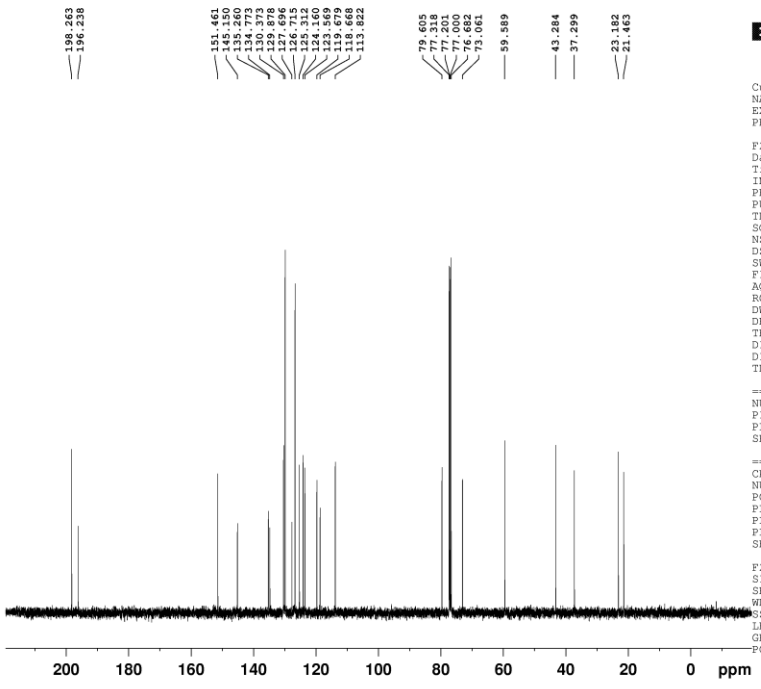
```
Current Data Parameters
NAME        MTC-10-236-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130123
Time                7.04
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                 32.04
DW                60.800 usec
DE                 6.50 usec
TE                298.5 K
D1             1.00000000 sec
TD0                    1

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

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



BRUKER

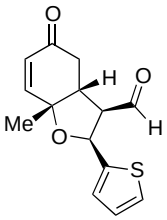
```
Current Data Parameters
NAME        MTC-10-236-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130123
Time                7.10
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    75
DS                     0
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                200.09
DW                20.800 usec
DE                 6.50 usec
TE                299.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

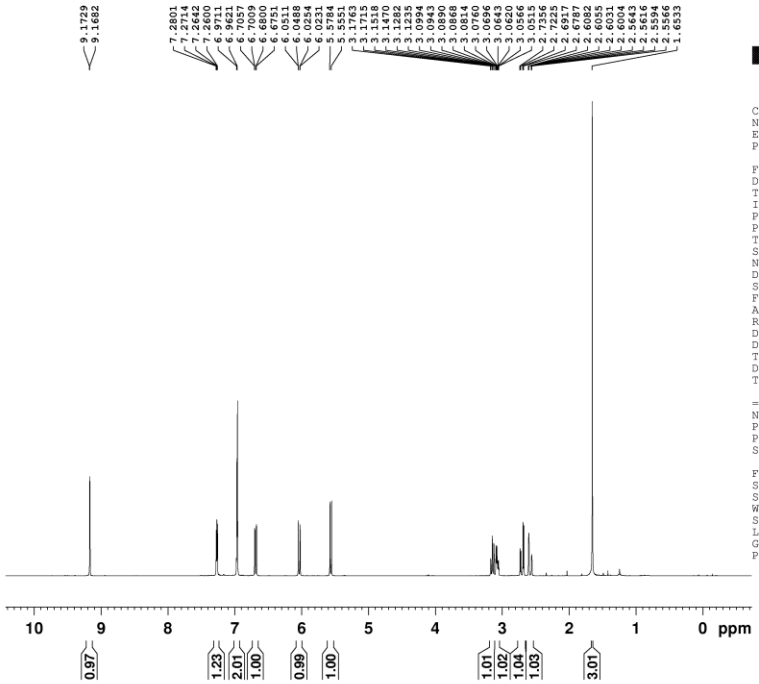
======== CHANNEL f1 ========
NUC1                  13C
P1                 7.50 usec
PLW1          61.20000076 W
SFO1          100.6127703 MHz

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

F2 - Processing parameters
SI                 32768
SF            100.6027219 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



4i



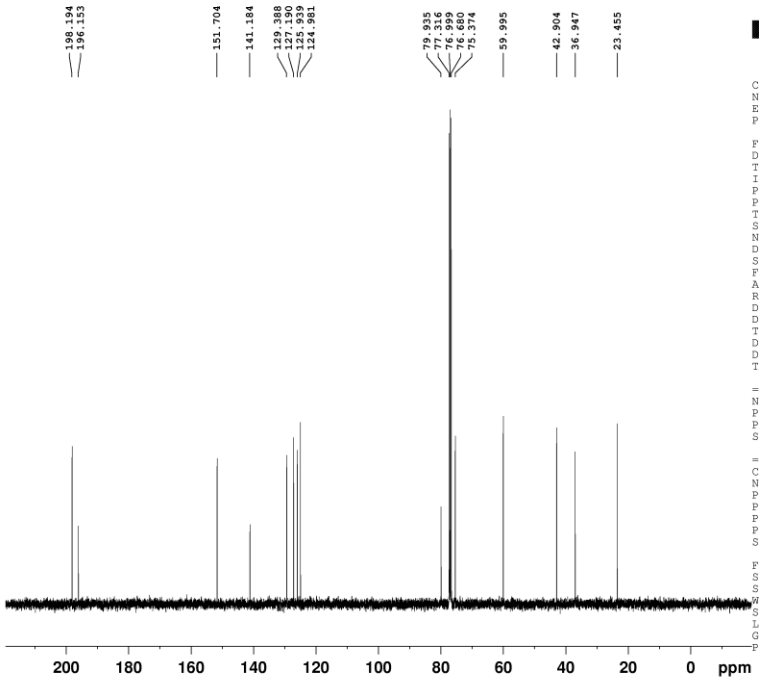
BRUKER

```
Current Data Parameters
NAME        MTC-10-202-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130112
Time                5.49
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                110.42
DW                60.800 usec
DE                 6.50 usec
TE                298.4 K
D1             1.00000000 sec
TD0                    1

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

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



BRUKER

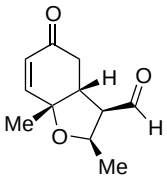
```
Current Data Parameters
NAME        MTC-10-202-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130112
Time                5.55
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   100
DS                     0
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                200.09
DW                20.800 usec
DE                 6.50 usec
TE                299.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

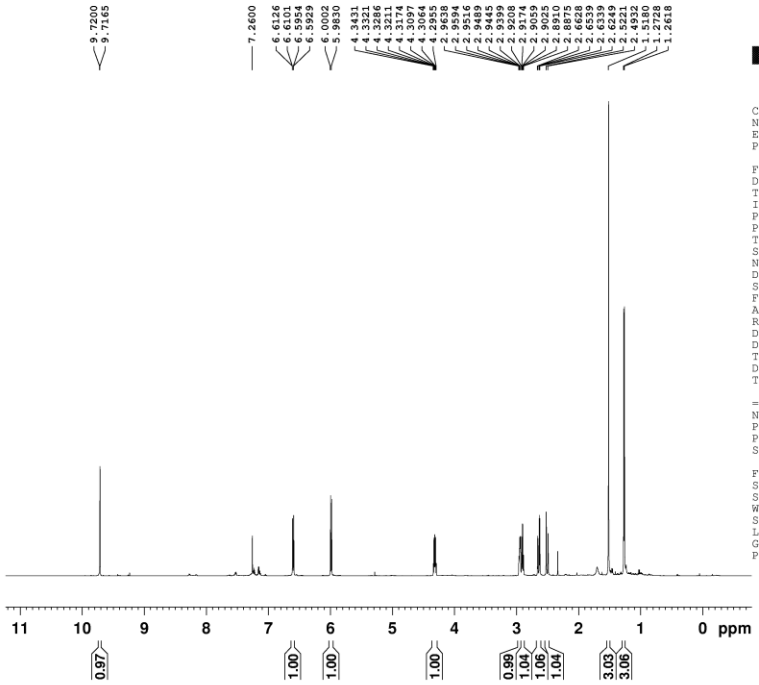
======== CHANNEL f1 ========
NUC1                  13C
P1                 7.50 usec
PLW1          61.20000076 W
SFO1         100.6127703 MHz

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

F2 - Processing parameters
SI                 32768
SF            100.6027171 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



4j

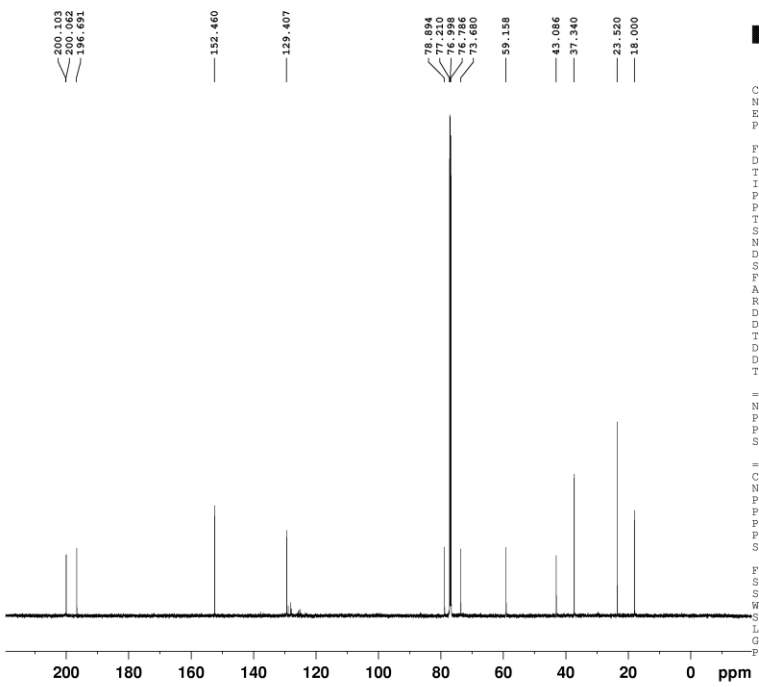


```
Current Data Parameters
NAME        MTC-10-216-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130116
Time                9.42
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                 20.2
DW               40.533 usec
DE               10.00 usec
TE               295.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.50 usec
PLW1         13.00000000 W
SFO1        600.1337060 MHz

F2 - Processing parameters
SI                 65536
SF           600.1300167 MHz
WDW                   EM
SSB       0
LB             0.30 Hz
GB        0
PC             1.00
```



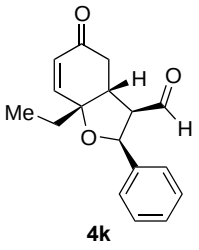
```
Current Data Parameters
NAME        MTC-10-216-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130116
Time                9.44
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   80
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9088159 sec
RG                  203
DW               13.867 usec
DE                18.00 usec
TE                295.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

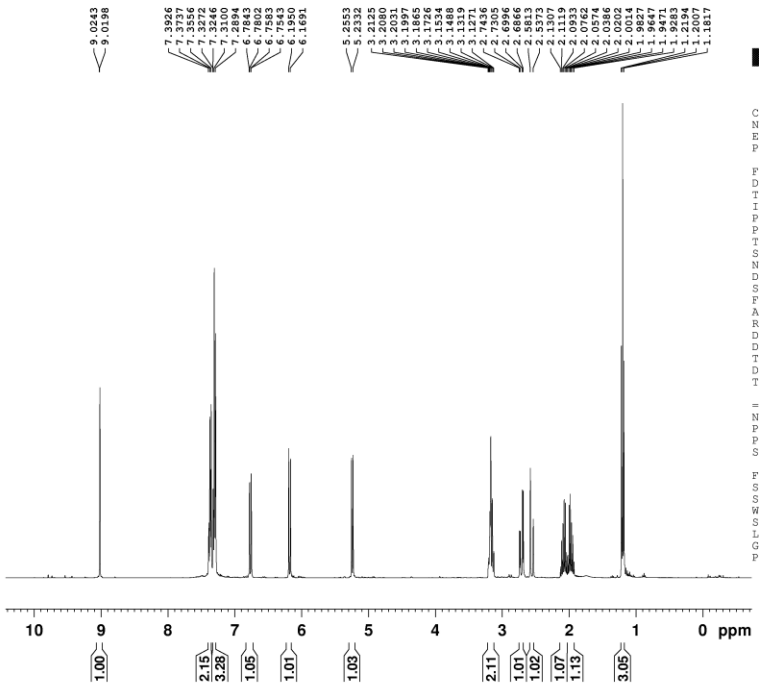
======== CHANNEL f1 ========
NUC1                  13C
P1                 11.00 usec
PLW1        230.00000000 W
SFO1        150.9178981 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2         13.00000000 W
PLW12         0.25077000 W
PLW13         0.20313001 W
SFO2        600.1324005 MHz

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



4k

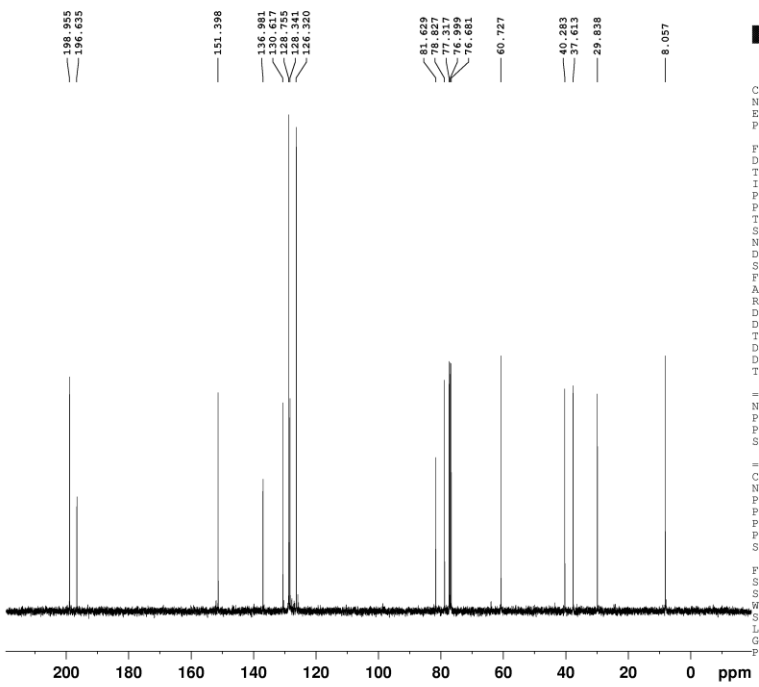


```
Current Data Parameters
NAME       MTC-11-129-H3
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130211
Time               11.33
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                71.37
DW               60.800 usec
DE                6.50 usec
TE                298.4 K
D1           1.00000000 sec
TD0                   1

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

F2 - Processing parameters
SI                65536
SF          400.0900000 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



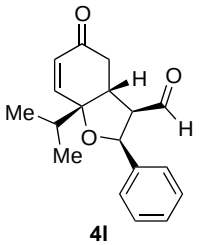
```
Current Data Parameters
NAME       MTC-10-129-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121208
Time                9.29
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   50
DS                    0
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                200.09
DW               20.800 usec
DE                6.50 usec
TE                297.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

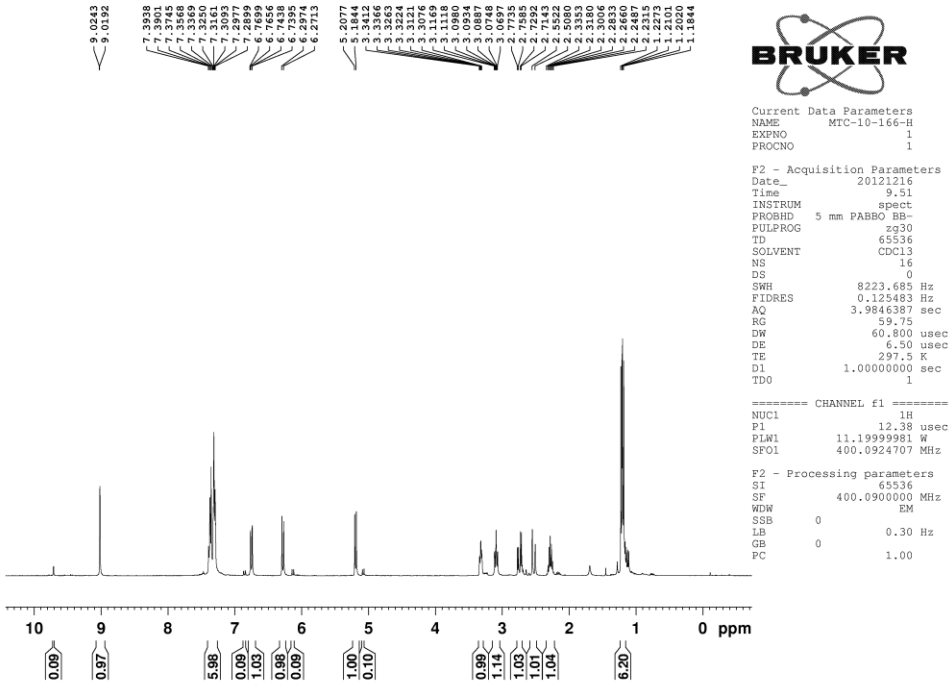
======== CHANNEL f1 ========
NUC1                13C
P1                7.50 usec
PLW1        61.20000076 W
SFO1       100.6127703 MHz

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

F2 - Processing parameters
SI                32768
SF         100.6027270 MHz
WDW                  EM
SSB       0
LB             1.00 Hz
GB        0
PC                1.40
```



4l



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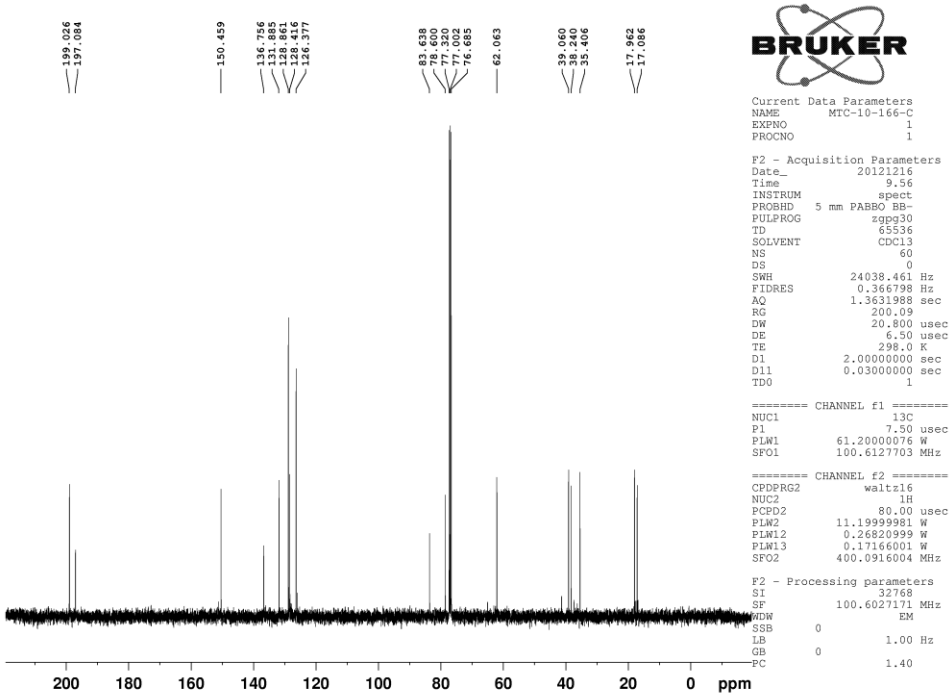
```
Current Data Parameters
NAME        MTC-10-166-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121216
Time                9.51
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                 59.75
DW                60.800 usec
DE                 6.50 usec
TE                297.5 K
D1             1.00000000 sec
TD0                    1

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

F2 - Processing parameters
SI                 65536
SF            400.0900000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



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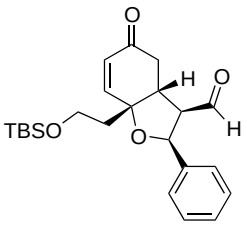
```
Current Data Parameters
NAME        MTC-10-166-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121216
Time                9.56
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    60
DS                     0
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                200.09
DW                20.800 usec
DE                 6.50 usec
TE                298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

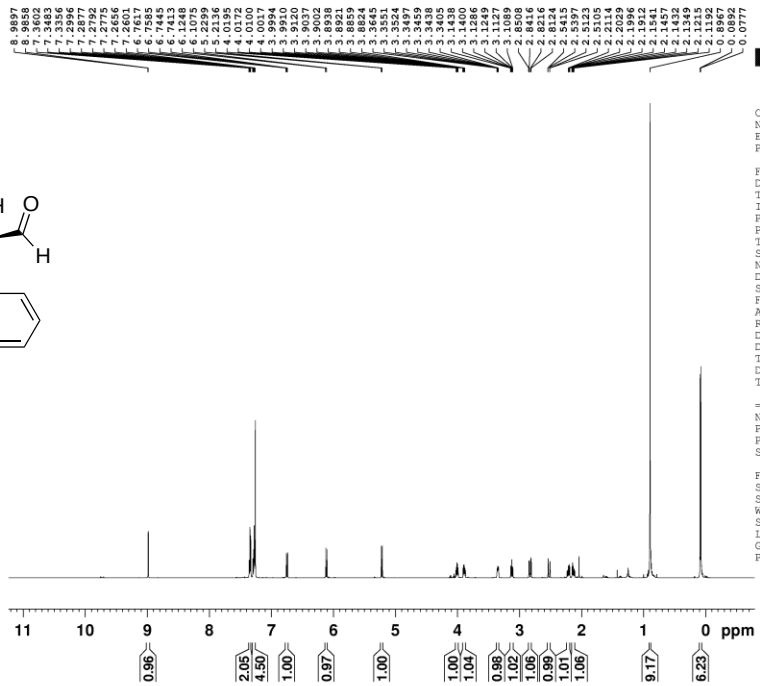
======== CHANNEL f1 ========
NUC1                  13C
P1                 7.50 usec
PLW1          61.20000076 W
SFO1         100.6127703 MHz

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

F2 - Processing parameters
SI                 32768
SF            100.6027171 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



4m

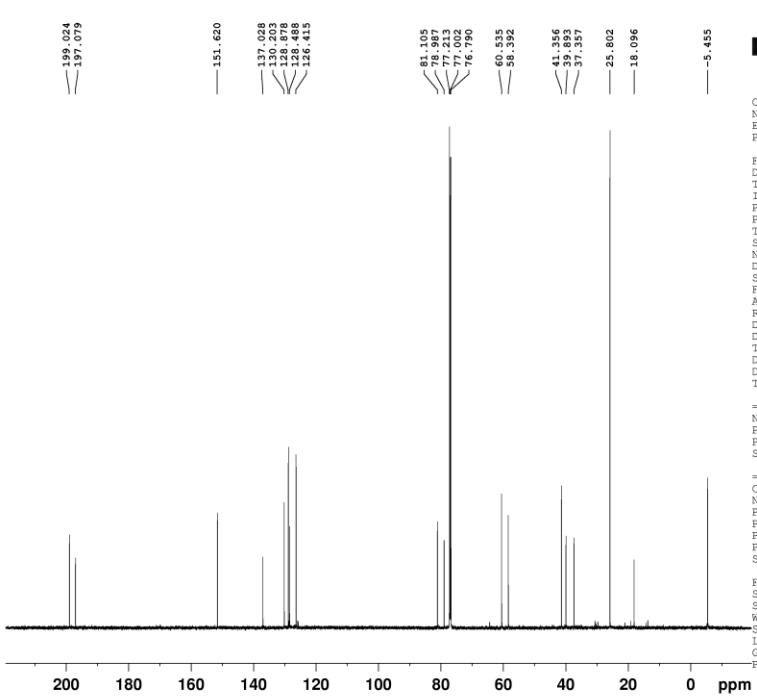



```
Current Data Parameters
NAME       MTC-10-262-H2
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20130205
Time               17.07
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                   16
DW               40.533 usec
DE               10.00 usec
TE                295.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                19.25 usec
PLW1        13.00000000 W
SFO1       600.1337060 MHz

F2 - Processing parameters
SI                65536
SF           600.1300163 MHz
WDW                  EM
SSB       0
LB             0.30 Hz
GB        0
PC                1.00
```



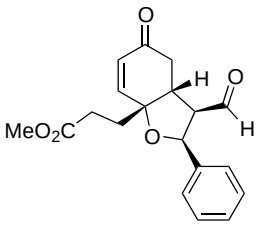

```
Current Data Parameters
NAME       MTC-10-262-C2
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20130205
Time               17.11
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   75
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9088159 sec
RG                  203
DW               13.867 usec
DE                18.00 usec
TE                295.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

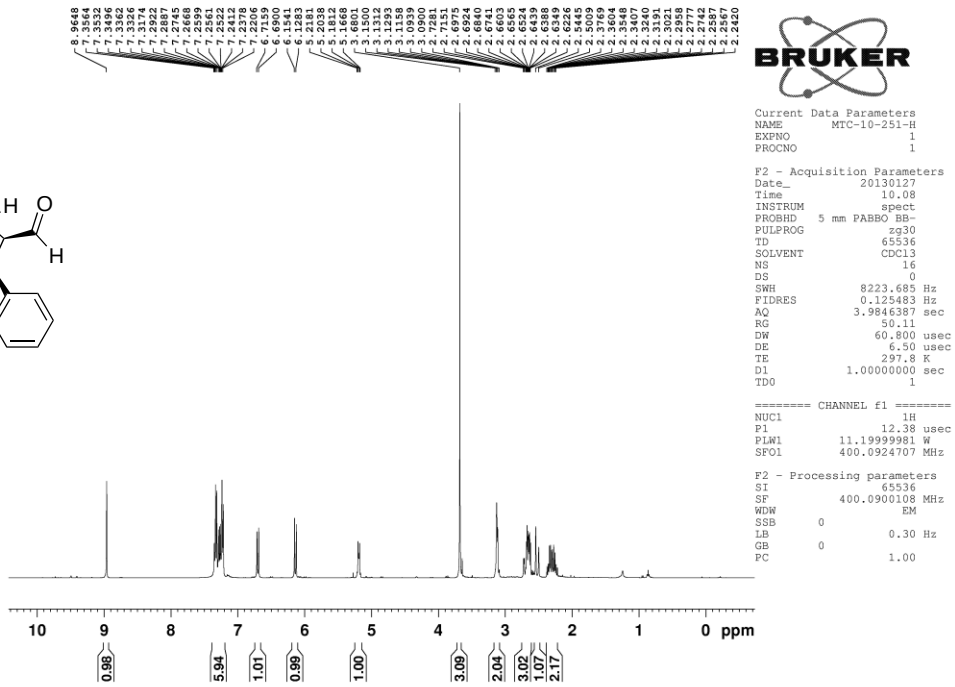
======== CHANNEL f1 ========
NUC1                 13C
P1                11.00 usec
PLW1       230.00000000 W
SFO1       150.9178981 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2        13.00000000 W
PLW12        0.59473002 W
PLW13        0.48173001 W
SFO2       600.1324005 MHz

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



4n

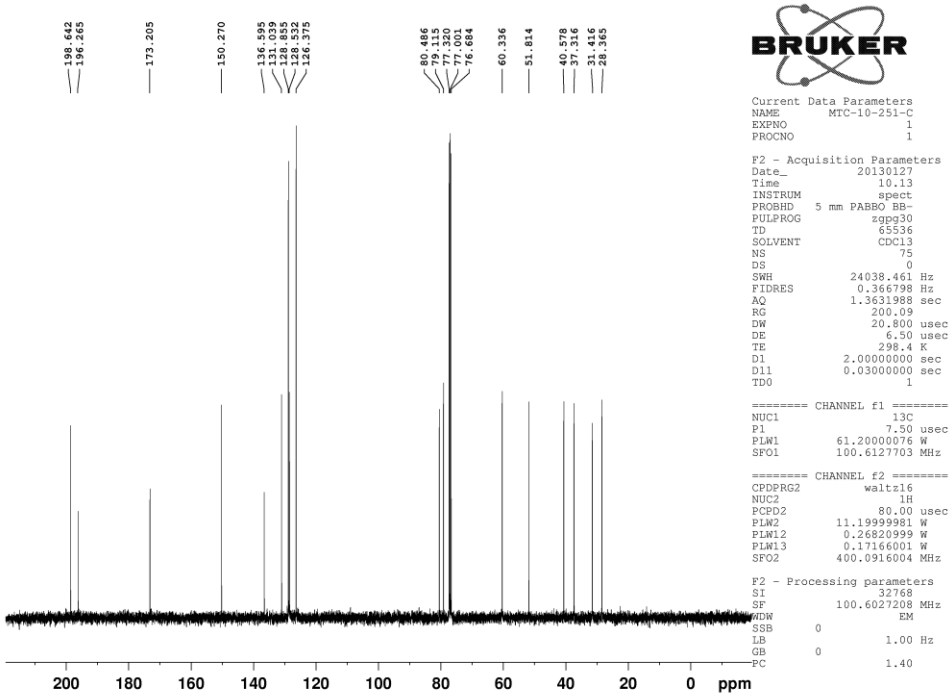



```
Current Data Parameters
NAME        MTC-10-251-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130127
Time               10.08
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                50.11
DW               60.800 usec
DE                6.50 usec
TE                297.8 K
D1           1.00000000 sec
TD0                   1

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

F2 - Processing parameters
SI                65536
SF          400.0900108 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



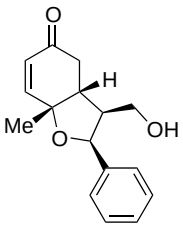

```
Current Data Parameters
NAME        MTC-10-251-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130127
Time               10.13
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                   75
DS                    0
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                200.09
DW               20.800 usec
DE                6.50 usec
TE                298.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

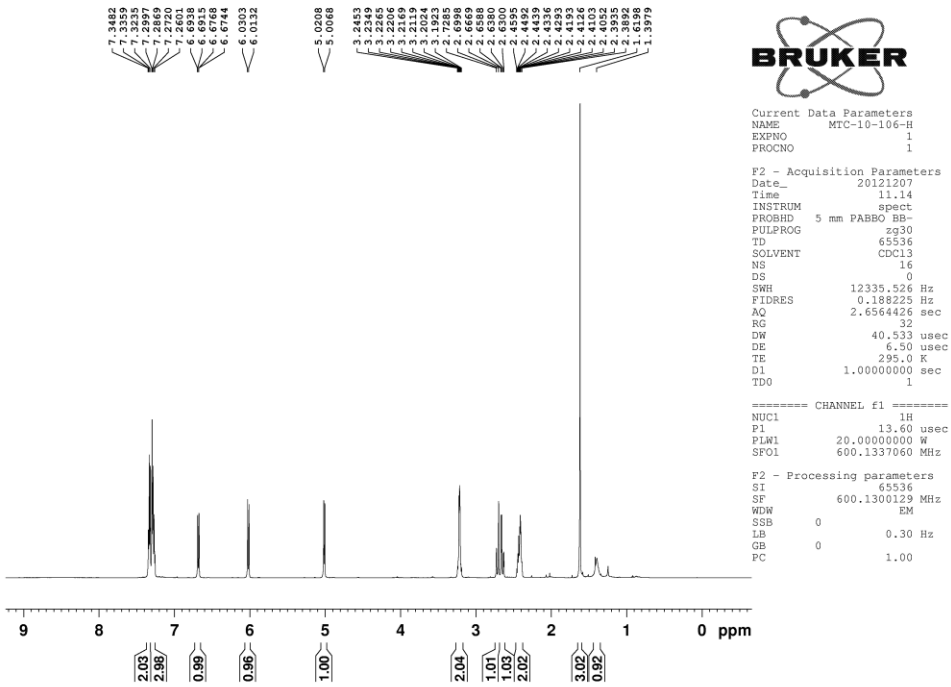
======== CHANNEL f1 ========
NUC1                 13C
P1                 7.50 usec
PLW1        61.20000076 W
SFO1        100.6127703 MHz

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

F2 - Processing parameters
SI                32768
SF          100.6027208 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



5a

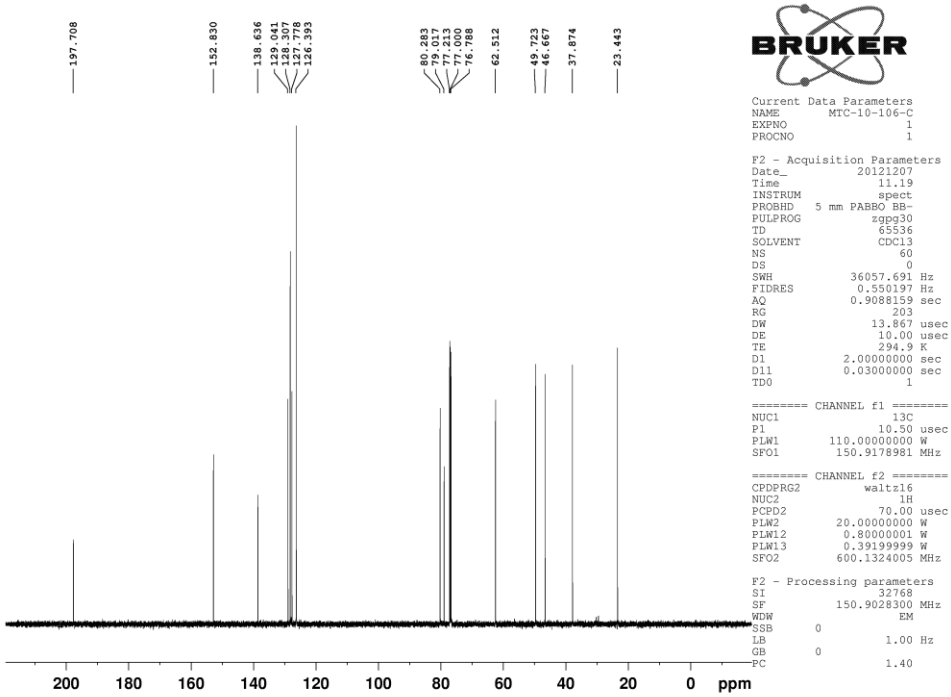



```
Current Data Parameters
NAME        MTC-10-106-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121207
Time               11.14
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                    32
DW               40.533 usec
DE                6.50 usec
TE                295.0 K
D1           1.00000000 sec
TD0                    1

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

F2 - Processing parameters
SI                 65536
SF          600.1300129 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



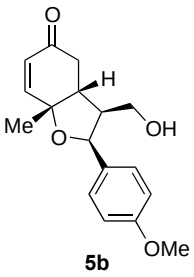

```
Current Data Parameters
NAME        MTC-10-106-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121207
Time               11.19
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    60
DS                     0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9088159 sec
RG                   203
DW               13.867 usec
DE                10.00 usec
TE                294.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

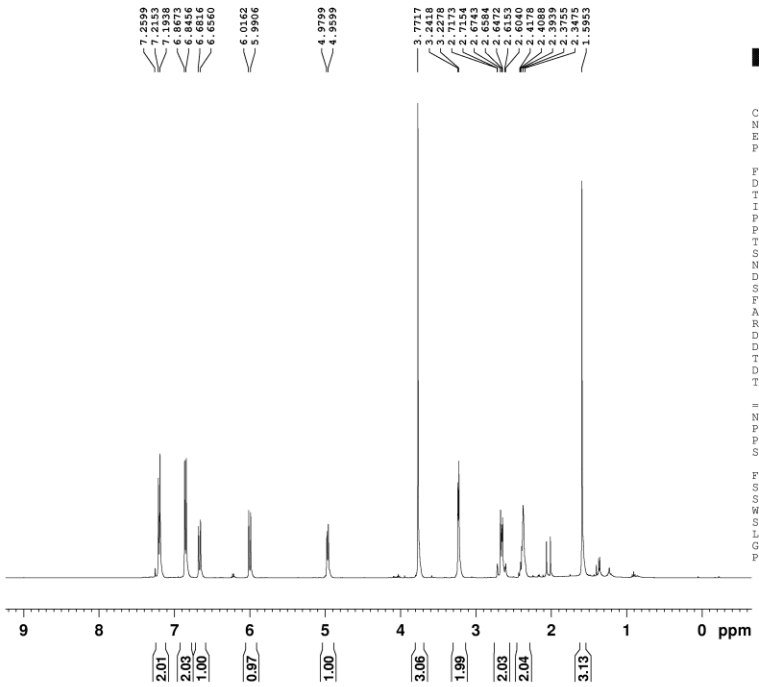
======== CHANNEL f1 ========
NUC1                 13C
P1                10.50 usec
PLW1       110.00000000 W
SFO1       150.9178981 MHz

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

F2 - Processing parameters
SI                 32768
SF          150.9028300 MHz
WDW                   EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



5b

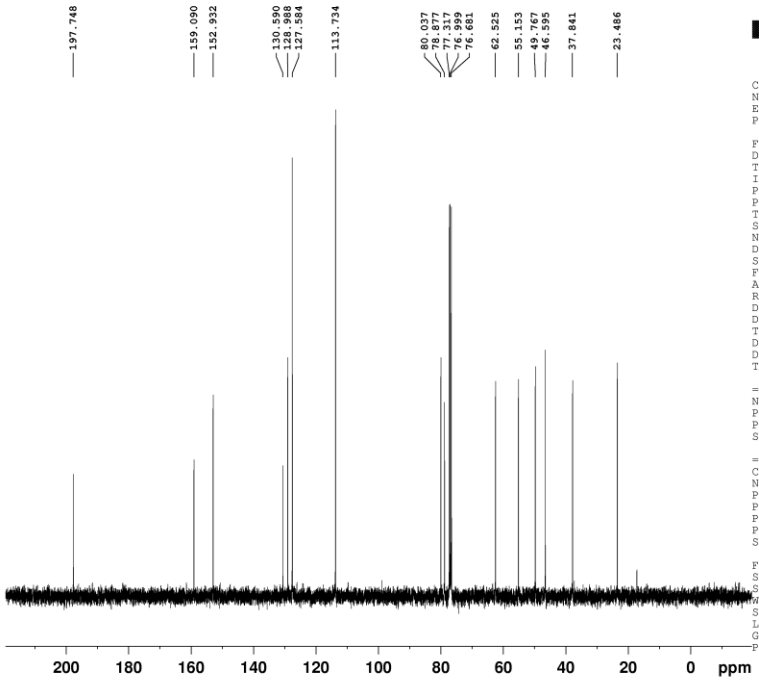


```
Current Data Parameters
NAME       MTC-10-146-H2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121212
Time                7.54
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                 25.11
DW                60.800 usec
DE                 6.50 usec
TE                297.2 K
D1             1.00000000 sec
TD0                    1

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

F2 - Processing parameters
SI                 65536
SF            400.0900125 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



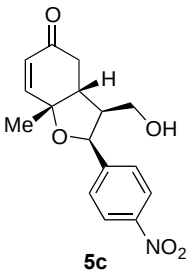
```
Current Data Parameters
NAME       MTC-10-146-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121211
Time               15.01
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    56
DS                     0
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                200.09
DW                20.800 usec
DE                 6.50 usec
TE                297.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

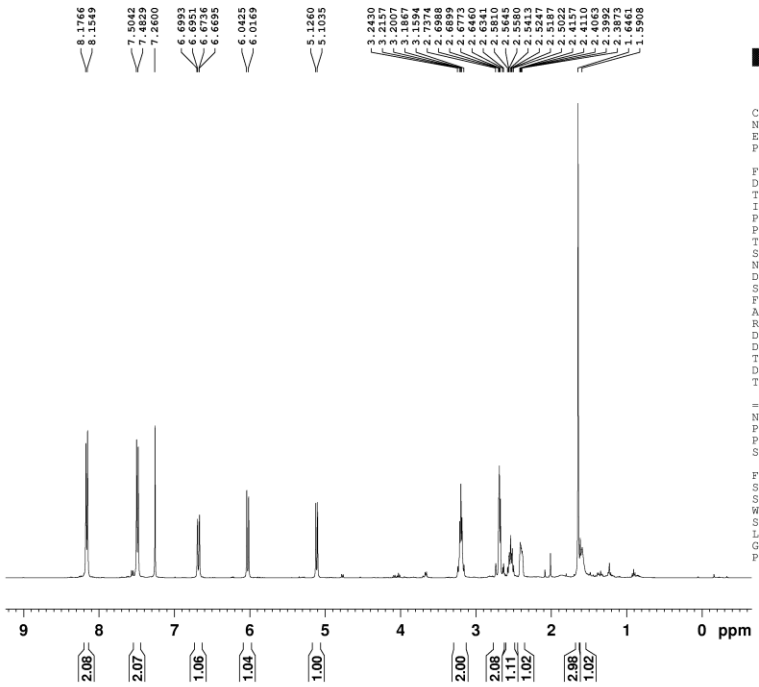
======== CHANNEL f1 ========
NUC1                  13C
P1                 7.50 usec
PLW1          61.20000076 W
SFO1          100.6127703 MHz

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

F2 - Processing parameters
SI                 32768
SF            100.6027226 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



5c

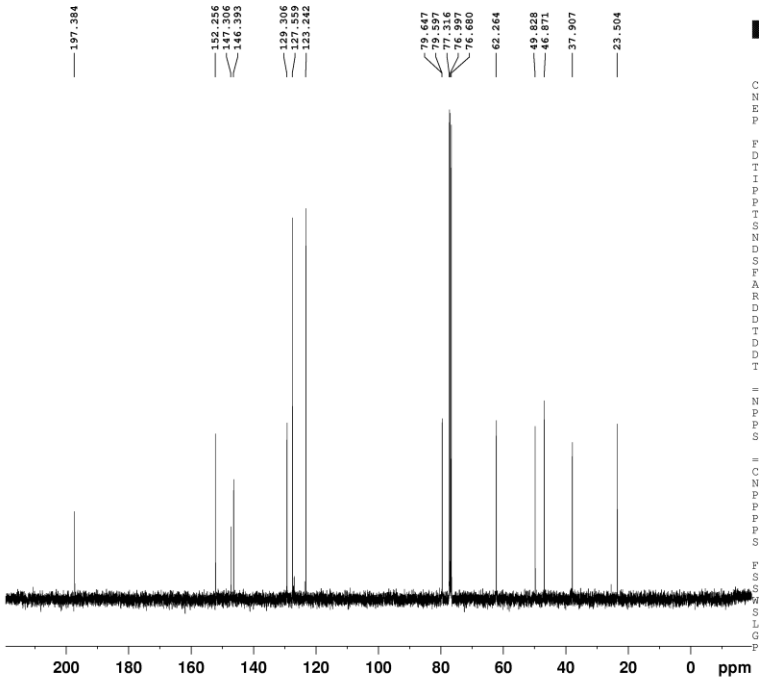


```
Current Data Parameters
NAME        MTC-10-198-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130109
Time                7.21
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                 53.79
DW                60.800 usec
DE                 6.50 usec
TE                298.2 K
D1             1.00000000 sec
TD0                    1

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

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



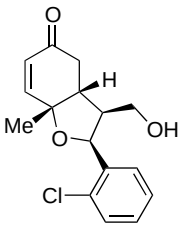
```
Current Data Parameters
NAME        MTC-10-194-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130107
Time               18.21
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    60
DS                     0
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                200.09
DW                20.800 usec
DE                 6.50 usec
TE                299.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

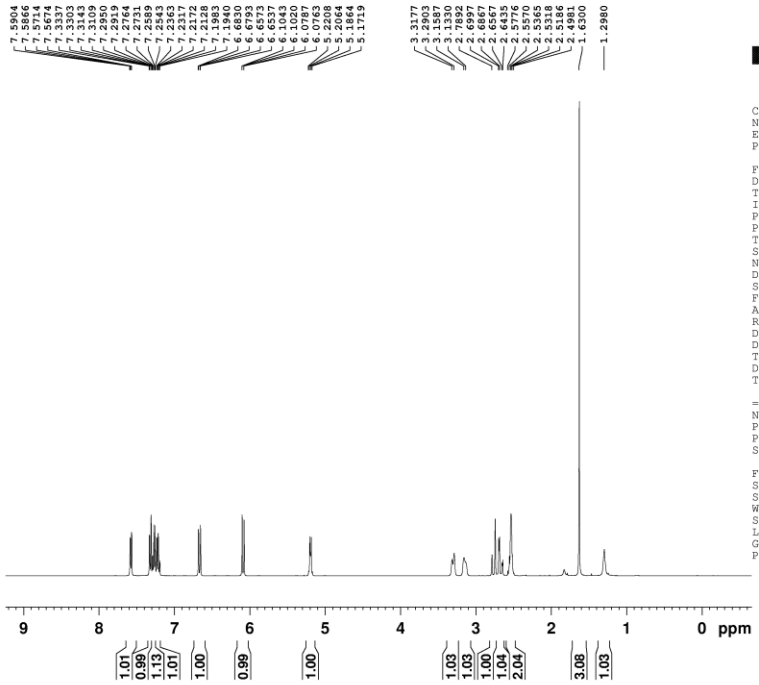
======== CHANNEL f1 ========
NUC1                  13C
P1                 7.50 usec
PLW1          61.20000076 W
SFO1          100.6127703 MHz

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

F2 - Processing parameters
SI                 32768
SF          100.6027183 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



5d

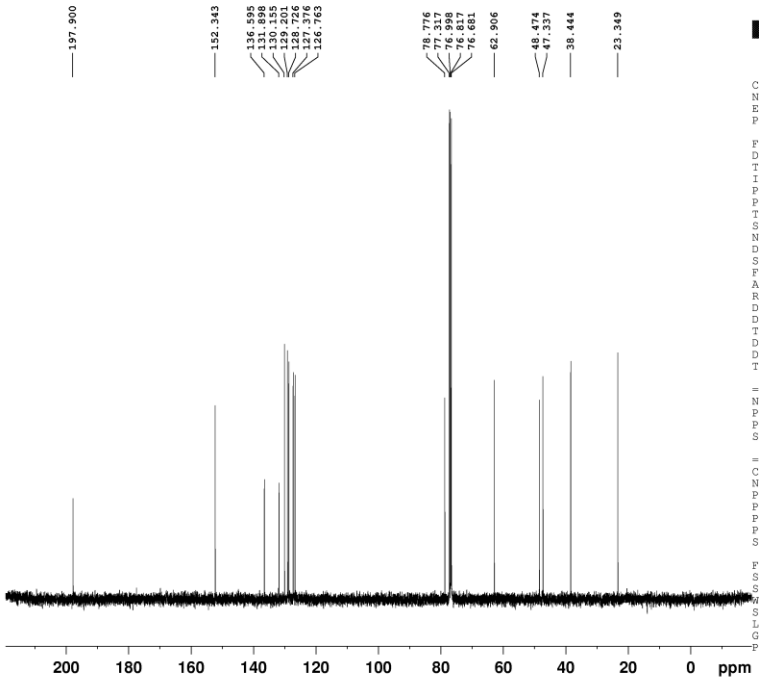



```
Current Data Parameters
NAME        MTC-10-201-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130110
Time                7.07
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                 53.79
DW                60.800 usec
DE                 6.50 usec
TE                298.9 K
D1             1.00000000 sec
TD0                    1

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

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



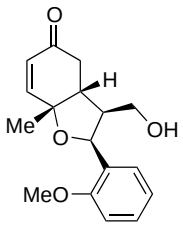

```
Current Data Parameters
NAME        MTC-10-201-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130110
Time                7.13
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    70
DS                     0
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                200.09
DW                20.800 usec
DE                 6.50 usec
TE                299.6 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

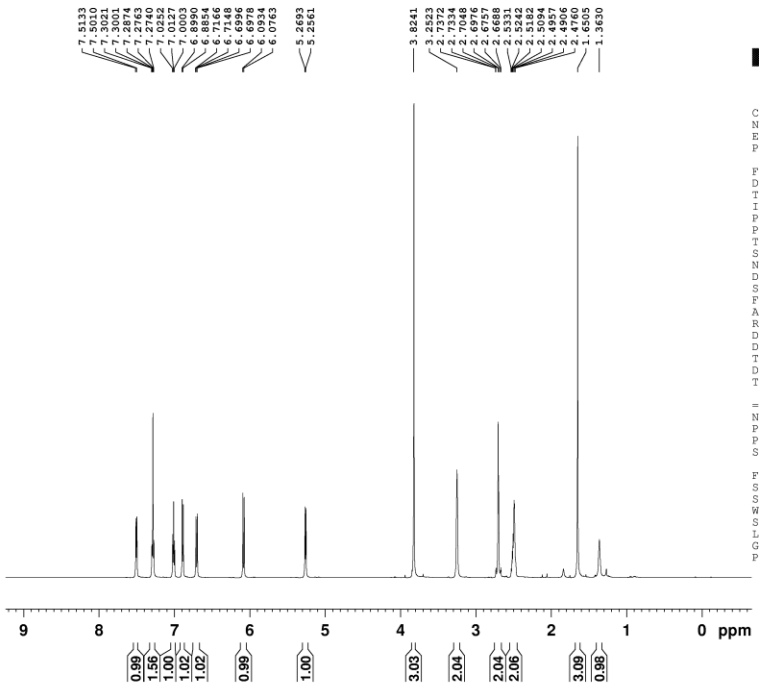
======== CHANNEL f1 ========
NUC1                  13C
P1                 7.50 usec
PLW1          61.20000076 W
SFO1         100.6127703 MHz

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

F2 - Processing parameters
SI                 32768
SF            100.6027182 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



5e

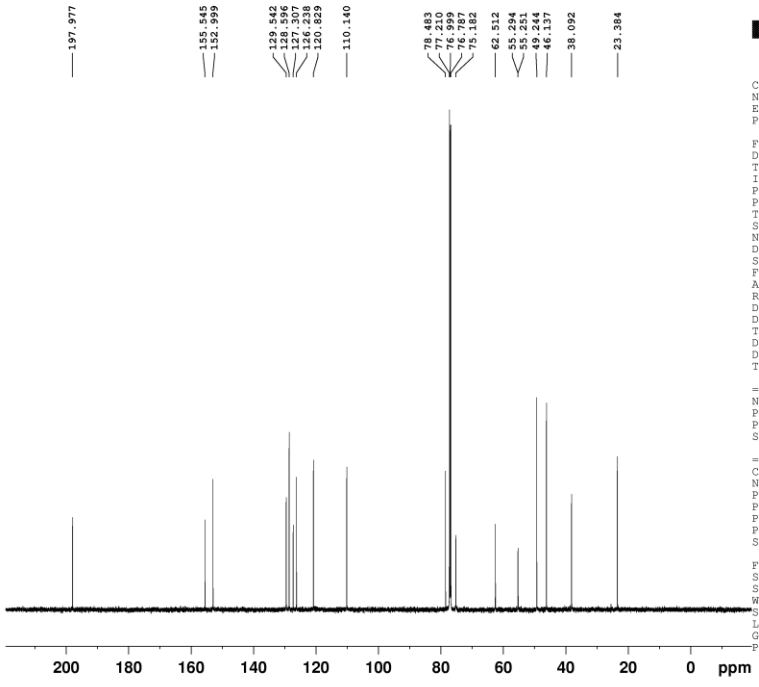


```
Current Data Parameters
NAME        MTC-10-218-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130114
Time               13.35
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                   16
DW               40.533 usec
DE               10.00 usec
TE               295.0 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                12.50 usec
PLW1        13.00000000 W
SFO1       600.1337060 MHz

F2 - Processing parameters
SI                65536
SF           600.1300000 MHz
WDW                  EM
SSB       0
LB              0.30 Hz
GB        0
PC                1.00
```



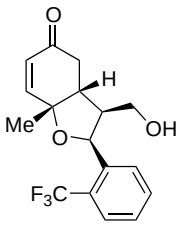
```
Current Data Parameters
NAME        MTC-10-218-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130114
Time               13.39
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   50
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9088159 sec
RG                  203
DW               13.867 usec
DE                18.00 usec
TE                295.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

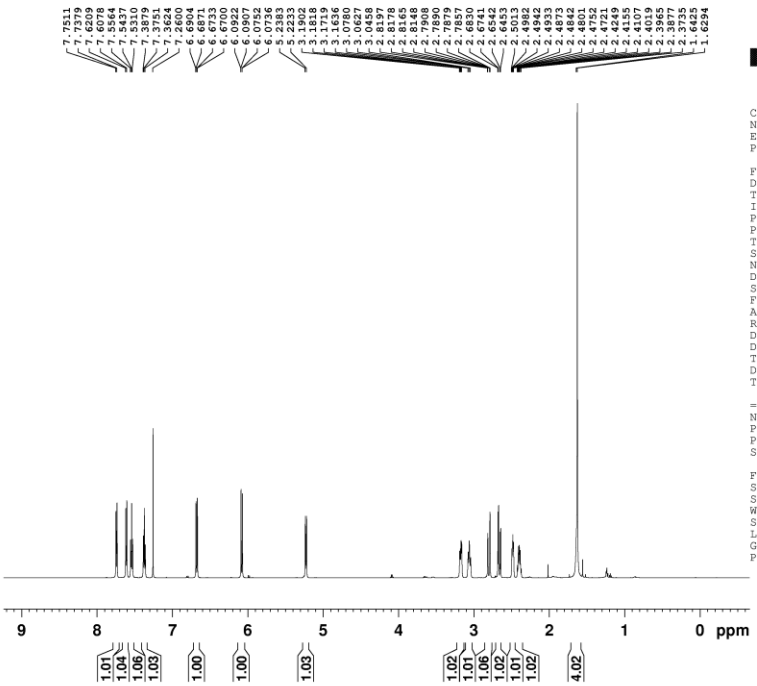
======== CHANNEL f1 ========
NUC1                 13C
P1                11.00 usec
PLW1       230.00000000 W
SFO1       150.9178981 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PLW2        13.00000000 W
PLW12        0.25077000 W
PLW13        0.20313001 W
SFO2       600.1324005 MHz

F2 - Processing parameters
SI                32768
SF           150.9028233 MHz
WDW                  EM
SSB       0
LB              1.00 Hz
GB        0
PC                1.40
```



5f

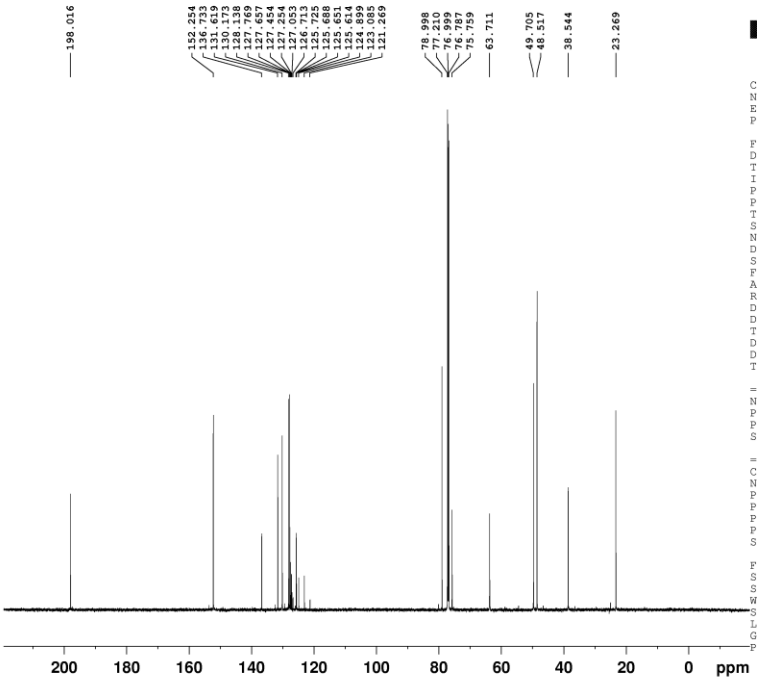



```
Current Data Parameters
NAME        MTC-10-230-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130119
Time                6.24
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                 25.4
DW               40.533 usec
DE               10.00 usec
TE               295.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.50 usec
PLW1         13.00000000 W
SFO1        600.1337060 MHz

F2 - Processing parameters
SI                 65536
SF          600.1300165 MHz
WDW                   EM
SSB       0
LB             0.30 Hz
GB        0
PC             1.00
```



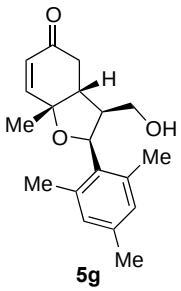

```
Current Data Parameters
NAME        MTC-10-230-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130119
Time                6.29
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   90
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9088159 sec
RG                  203
DW               13.867 usec
DE               18.00 usec
TE               295.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

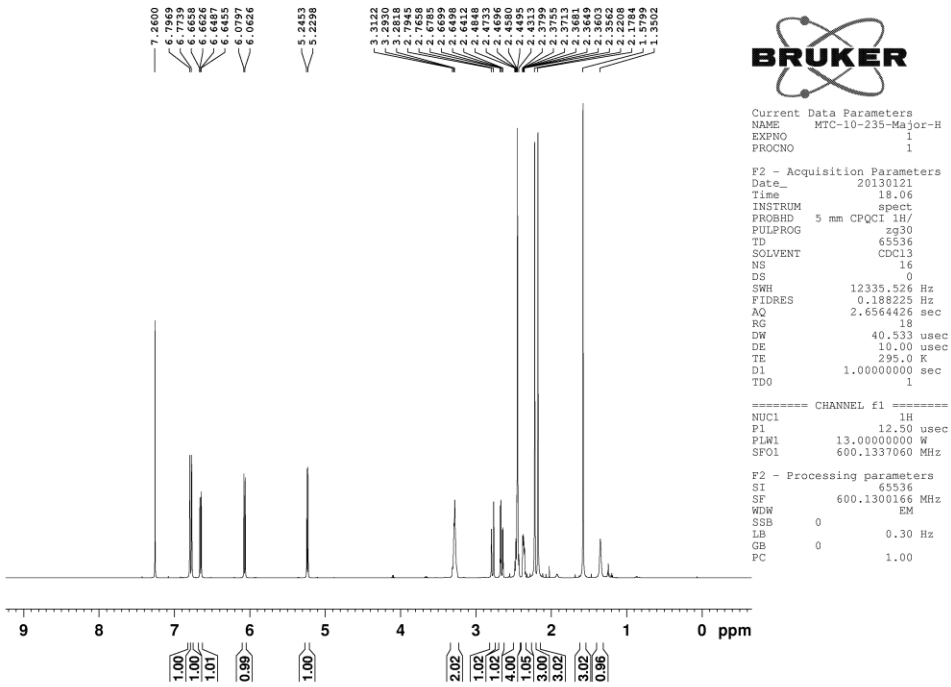
======== CHANNEL f1 ========
NUC1                 13C
P1                 11.00 usec
PLW1        230.00000000 W
SFO1        150.9178981 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2         13.00000000 W
PLW12         0.25077000 W
PLW13         0.20313001 W
SFO2        600.1324005 MHz

F2 - Processing parameters
SI                 32768
SF          150.9028228 MHz
WDW                   EM
SSB       0
LB             1.00 Hz
GB        0
PC             1.40
```



5g

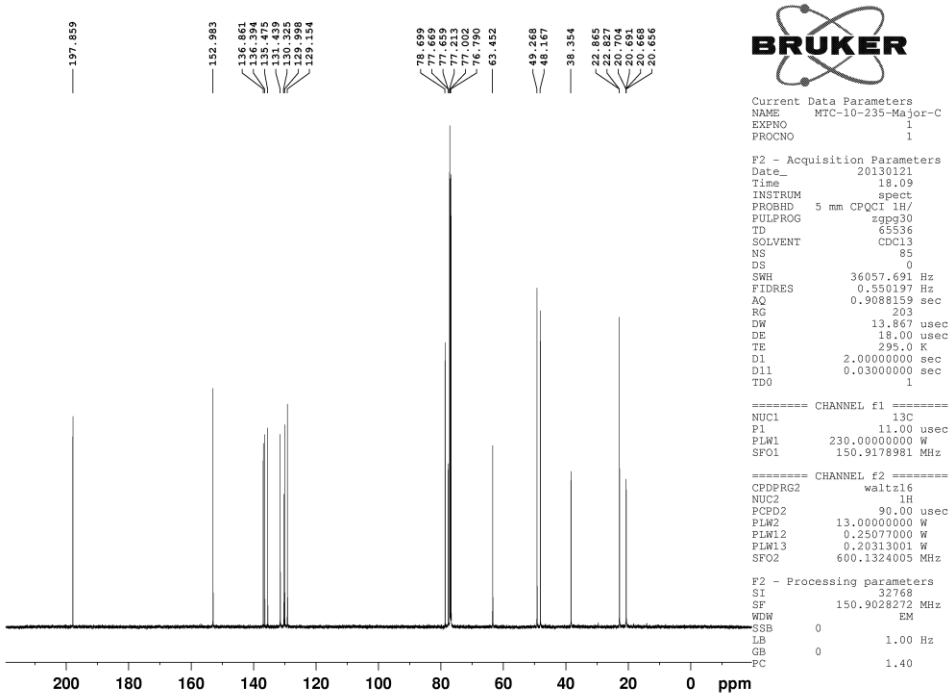



```
Current Data Parameters
NAME      MTC-10-235-Major-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130121
Time              18.06
INSTRUM           spect
PROBHD   5 mm CPQCI 1H/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   0
SWH          12335.526 Hz
FIDRES        0.188225 Hz
AQ            2.6564426 sec
RG                  18
DW              40.533 usec
DE              10.00 usec
TE              295.0 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1               12.50 usec
PLW1       13.00000000 W
SFO1       600.1337060 MHz

F2 - Processing parameters
SI               65536
SF          600.1300166 MHz
WDW                 EM
SSB       0
LB             0.30 Hz
GB        0
PC             1.00
```



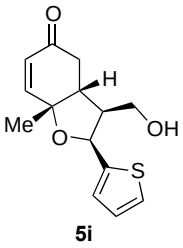

```
Current Data Parameters
NAME      MTC-10-235-Major-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130121
Time              18.09
INSTRUM           spect
PROBHD   5 mm CPQCI 1H/
PULPROG          zgpg30
TD               65536
SOLVENT          CDCl3
NS                  85
DS                   0
SWH          36057.691 Hz
FIDRES        0.550197 Hz
AQ            0.9088159 sec
RG                 203
DW              13.867 usec
DE              18.00 usec
TE              295.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

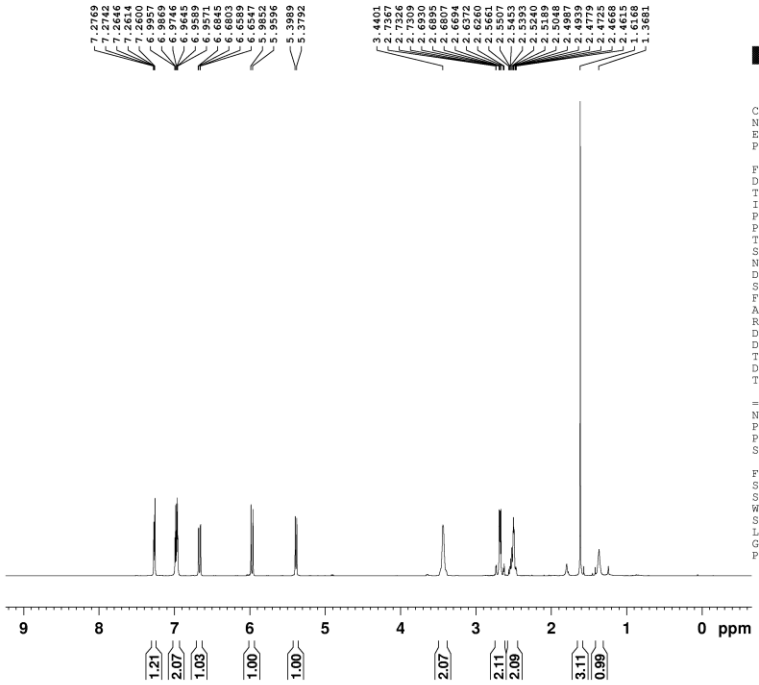
======== CHANNEL f1 ========
NUC1                13C
P1               11.00 usec
PLW1      230.00000000 W
SFO1       150.9178981 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2            90.00 usec
PLW2       13.00000000 W
PLW12       0.25077000 W
PLW13       0.20313001 W
SFO2       600.1324005 MHz

F2 - Processing parameters
SI               32768
SF          150.9028272 MHz
WDW                 EM
SSB       0
LB             1.00 Hz
GB        0
PC             1.40
```



5i

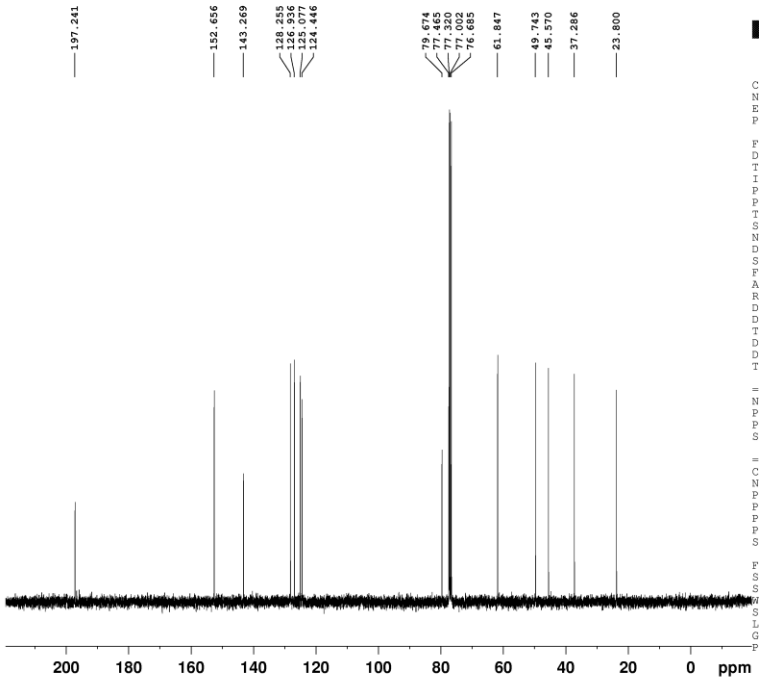



```
Current Data Parameters
NAME        MTC-10-208-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130112
Time                6.00
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                 71.37
DW                60.800 usec
DE                 6.50 usec
TE                298.5 K
D1             1.00000000 sec
TD0                    1

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

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



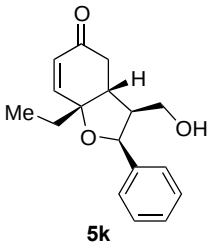

```
Current Data Parameters
NAME        MTC-10-208-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130112
Time                6.08
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    99
DS                     0
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                200.09
DW                20.800 usec
DE                 6.50 usec
TE                299.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

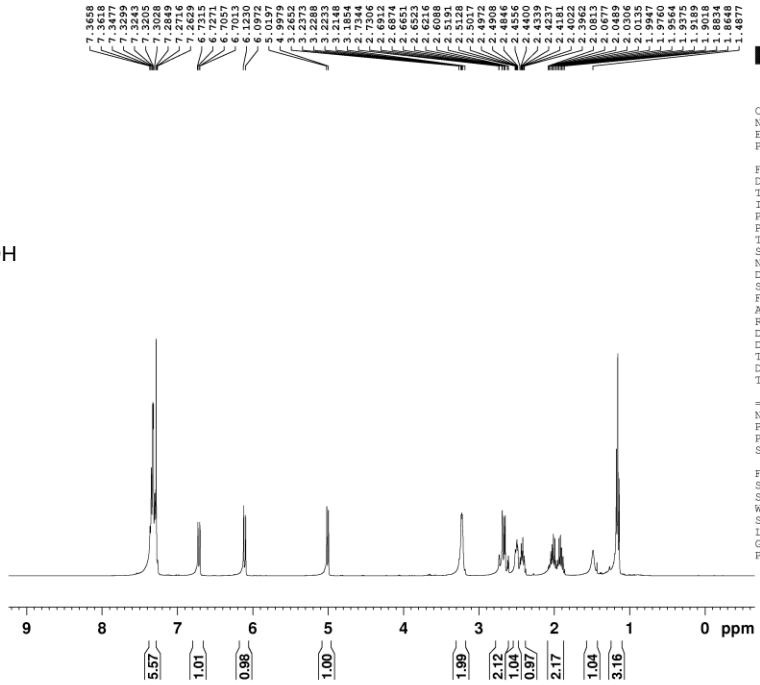
======== CHANNEL f1 ========
NUC1                  13C
P1                 7.50 usec
PLW1          61.20000076 W
SFO1         100.6127703 MHz

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

F2 - Processing parameters
SI                 32768
SF           100.6027179 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



5k



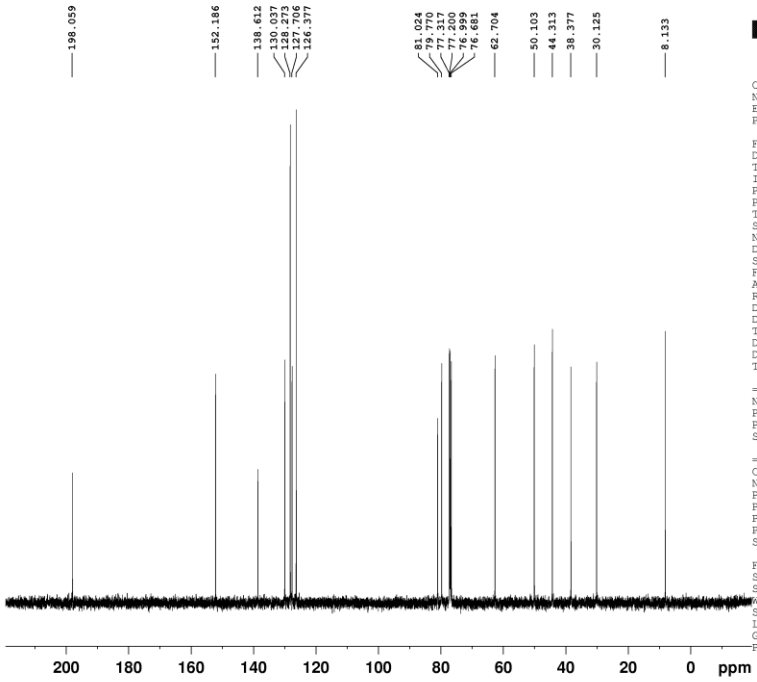
BRUKER

```
Current Data Parameters
NAME        MTC-10-134-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121208
Time               12.54
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                32.04
DW               60.800 usec
DE                6.50 usec
TE                297.1 K
D1           1.00000000 sec
TD0                   1

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

F2 - Processing parameters
SI                65536
SF          400.0900000 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



BRUKER

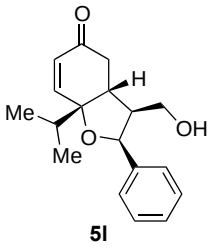
```
Current Data Parameters
NAME        MTC-10-134-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121208
Time               13.00
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   50
DS                    0
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                200.09
DW               20.800 usec
DE                6.50 usec
TE                297.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

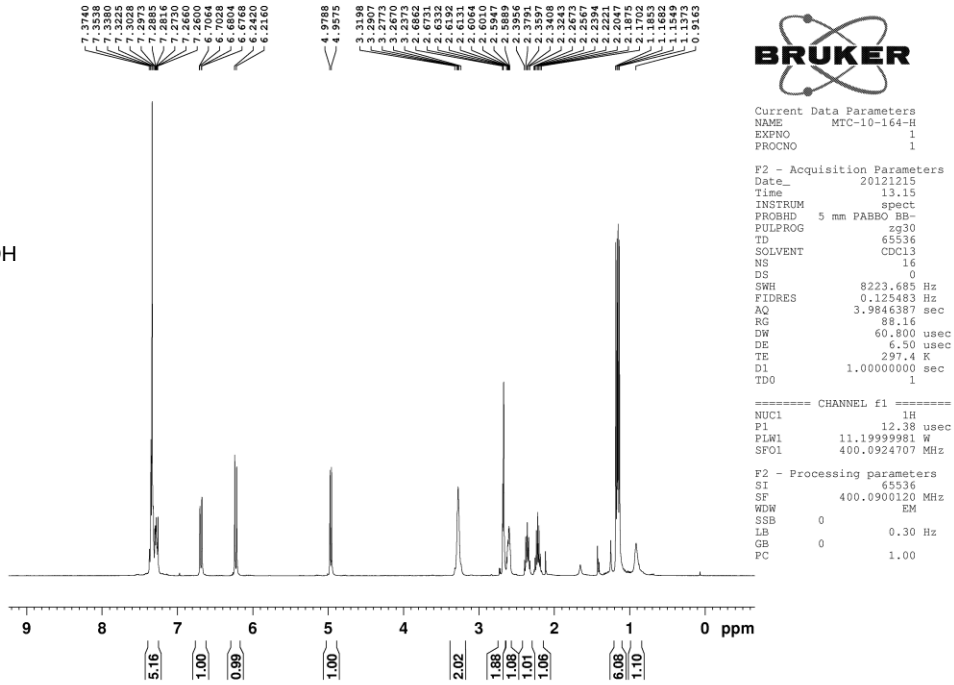
======== CHANNEL f1 ========
NUC1                 13C
P1                 7.50 usec
PLW1         61.20000076 W
SFO1        100.6127703 MHz

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

F2 - Processing parameters
SI                32768
SF          100.6027263 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



5l

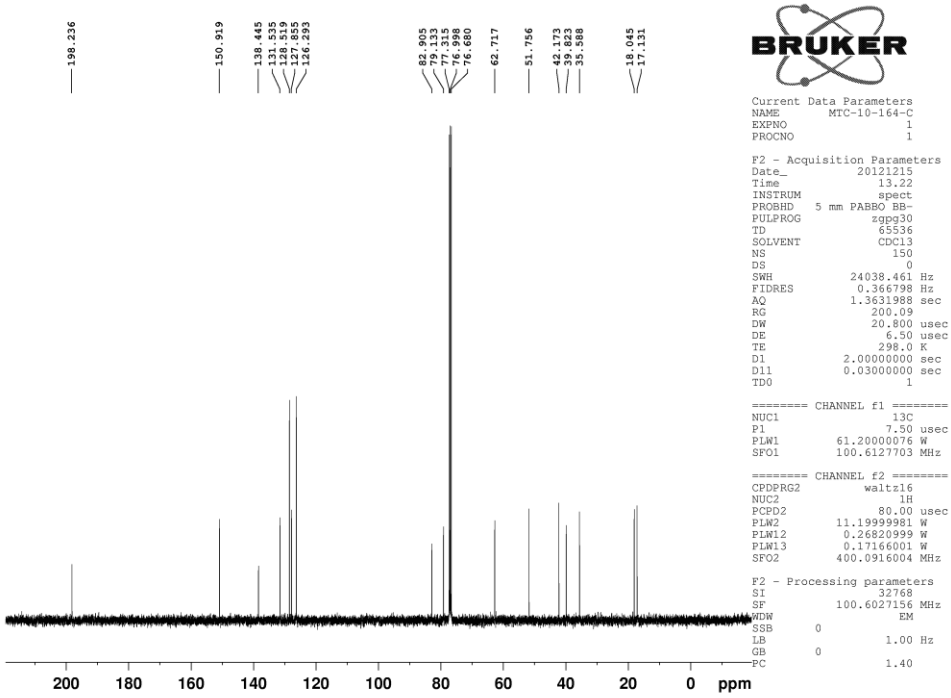



```
Current Data Parameters
NAME        MTC-10-164-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121215
Time               13.15
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                88.16
DW               60.800 usec
DE                6.50 usec
TE               297.4 K
D1            1.00000000 sec
TD0                   1

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

F2 - Processing parameters
SI                65536
SF          400.0900120 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



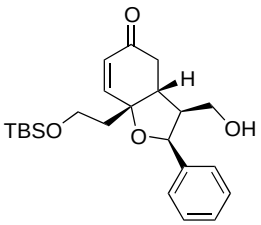

```
Current Data Parameters
NAME        MTC-10-164-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20121215
Time               13.22
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  150
DS                    0
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                200.09
DW               20.800 usec
DE                6.50 usec
TE               298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

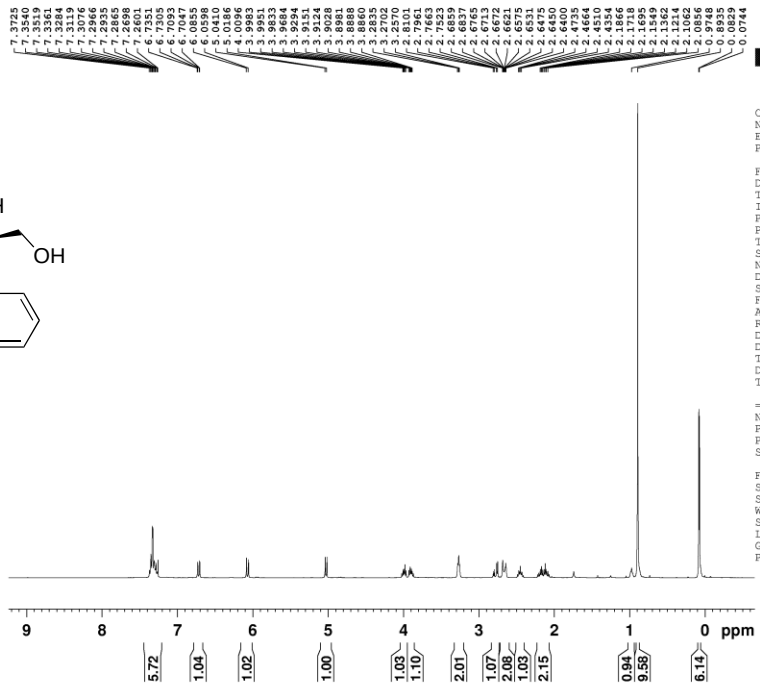
======== CHANNEL f1 ========
NUC1                13C
P1                7.50 usec
PLW1        61.20000076 W
SFO1        100.6127703 MHz

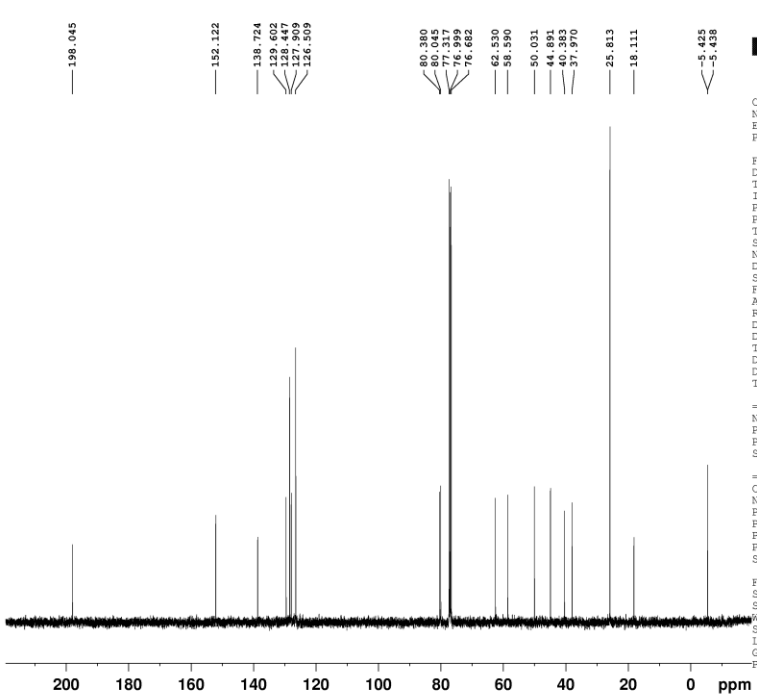
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            80.00 usec
PLW2        11.19999981 W
PLW12        0.26820999 W
PLW13        0.17166001 W
SFO2        400.0916004 MHz

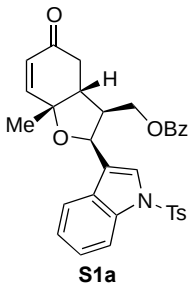
F2 - Processing parameters
SI                32768
SF          100.6027156 MHz
WDW                  EM
SSB       0
LB             1.00 Hz
GB        0
PC                1.40
```


O
H
TBSO
O
OH

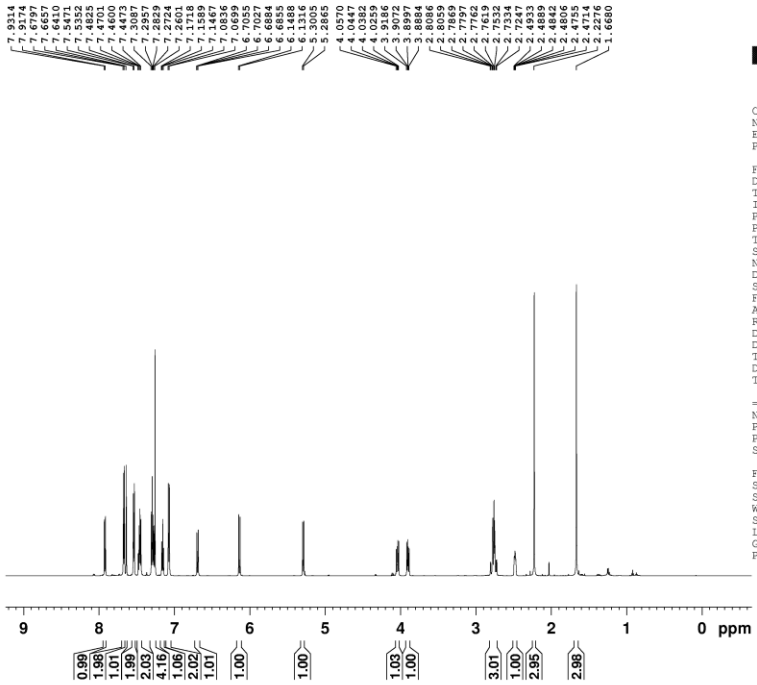
5m


7.3725
7.3540
7.3519
7.3361
7.3284
7.3119
7.3076
7.2966
7.2935
7.2865
7.2688
7.2601
6.7351
6.7305
6.7093
6.7047
6.0855
6.0598
5.0410
5.0186
5.0086
4.0083
3.9951
3.9833
3.9684
3.9644
3.9251
3.9124
3.9028
3.8981
3.8888
3.8860
3.8835
3.2702
3.2570
2.7901
2.7663
2.7523
2.6859
2.6787
2.6705
2.6713
2.6672
2.6621
2.6575
2.6531
2.6475
2.6450
2.6400
2.4725
2.4664
2.4510
2.4354
2.1866
2.1758
2.1695
2.1549
2.1362
2.1214
2.1062
2.0866
0.9748
0.8935
0.0829
0.0744
BRUKER
Current Data Parameters
NAME MTC-10-193-H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130108
Time 7.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 32.04
DW 60.800 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.38 usec
PLW1 11.19999981 W
SFO1 400.0924707 MHz
F2 - Processing parameters
SI 65536
SF 400.0900111 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
5.72 1.04 1.02 1.00 1.03 1.10 2.01 1.07 2.08 1.03 2.15 0.94 9.58 6.14


198.045
152.122
138.724
129.602
128.447
127.909
126.509
80.380
80.045
77.337
77.019
76.682
62.530
58.590
50.031
44.891
40.383
37.970
25.813
18.111
-5.425
-5.438
BRUKER
Current Data Parameters
NAME MTC-10-193-C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130108
Time 7.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 110
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 200.09
DW 20.800 usec
DE 6.50 usec
TE 298.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.50 usec
PLW1 61.20000076 W
SFO1 100.6127703 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 11.19999981 W
PLW12 0.26820999 W
PLW13 0.17166001 W
SFO2 400.0916004 MHz
F2 - Processing parameters
SI 32768
SF 100.6027167 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm



S1a

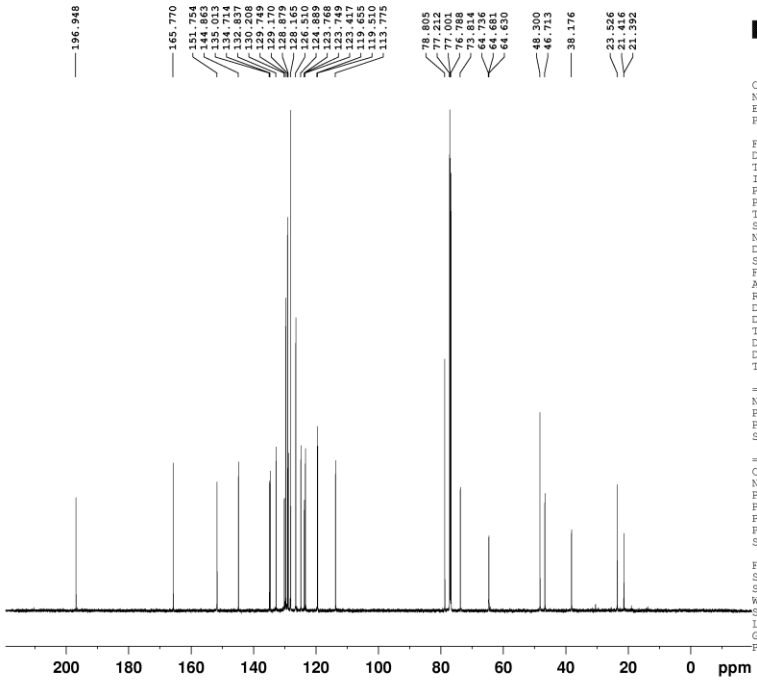



```
Current Data Parameters
NAME        MTC-10-241-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130124
Time               10.05
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                 14.2
DW               40.533 usec
DE               10.00 usec
TE               295.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.50 usec
PLW1         13.00000000 W
SFO1        600.1337060 MHz

F2 - Processing parameters
SI                 65536
SF          600.1300161 MHz
WDW                   EM
SSB       0
LB             0.30 Hz
GB        0
PC                 1.00
```



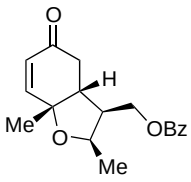

```
Current Data Parameters
NAME        MTC-10-241-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130124
Time               10.11
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  100
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9088159 sec
RG                  203
DW               13.867 usec
DE               18.00 usec
TE               295.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

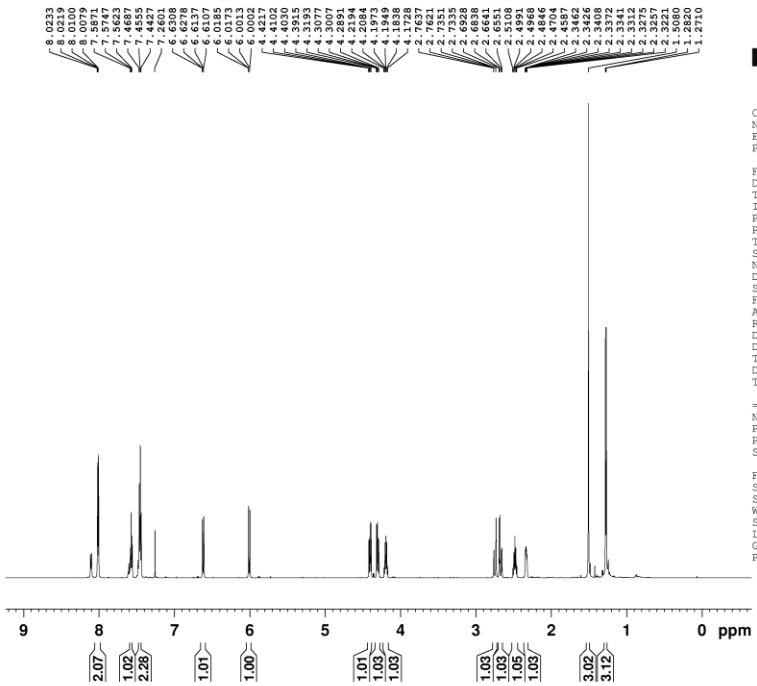
======== CHANNEL f1 ========
NUC1                  13C
P1                 11.00 usec
PLW1        230.00000000 W
SFO1        150.9178981 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2         13.00000000 W
PLW12         0.25077000 W
PLW13         0.20313001 W
SFO2        600.1324005 MHz

F2 - Processing parameters
SI                 32768
SF          150.9028322 MHz
WDW                   EM
SSB       0
LB             1.00 Hz
GB        0
PC                 1.40
```



S1b

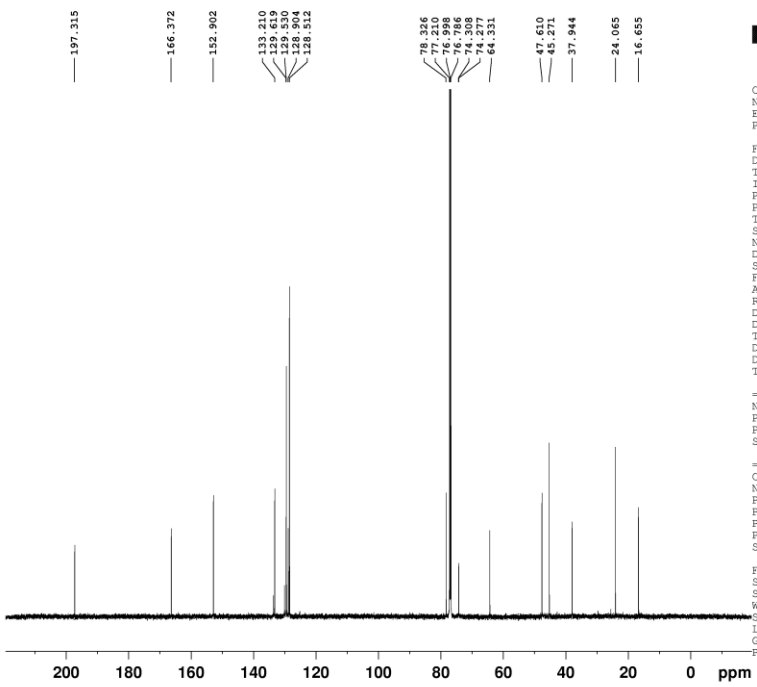


```
Current Data Parameters
NAME        MTC-10-228-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130119
Time                6.04
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                 20.2
DW               40.533 usec
DE               10.00 usec
TE               295.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.50 usec
PLW1         13.00000000 W
SFO1        600.1337060 MHz

F2 - Processing parameters
SI                 65536
SF          600.1300166 MHz
WDW                   EM
SSB       0
LB             0.30 Hz
GB        0
PC                 1.00
```



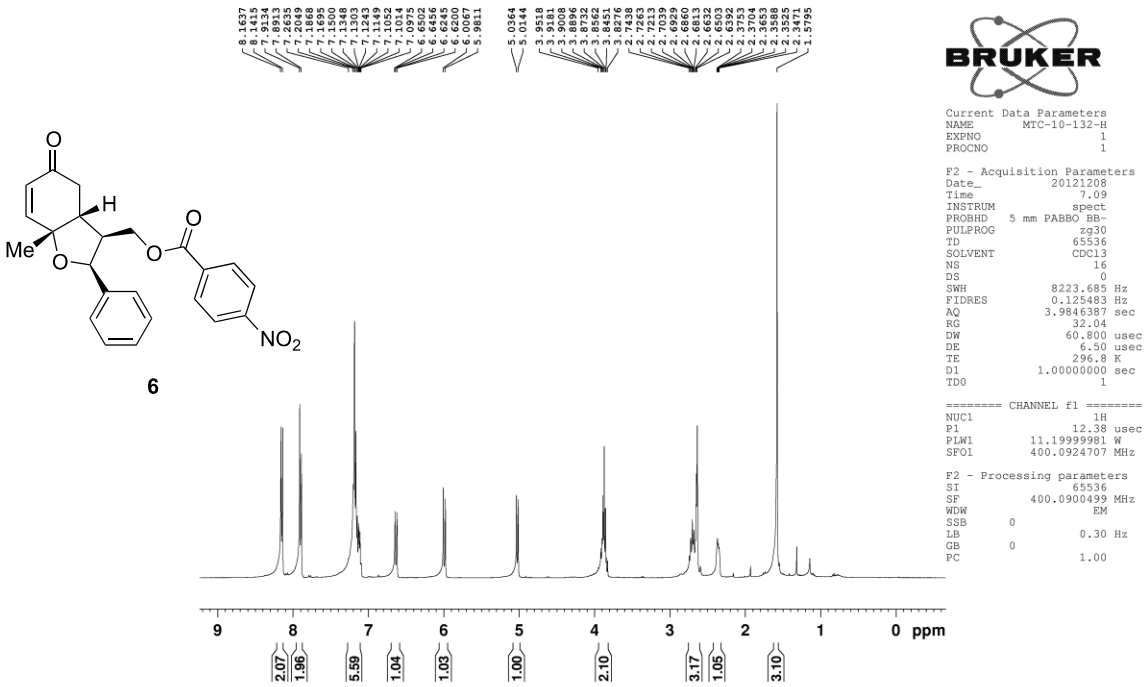
```
Current Data Parameters
NAME        MTC-10-228-C
EXPNO                  1
PROCNO                 1

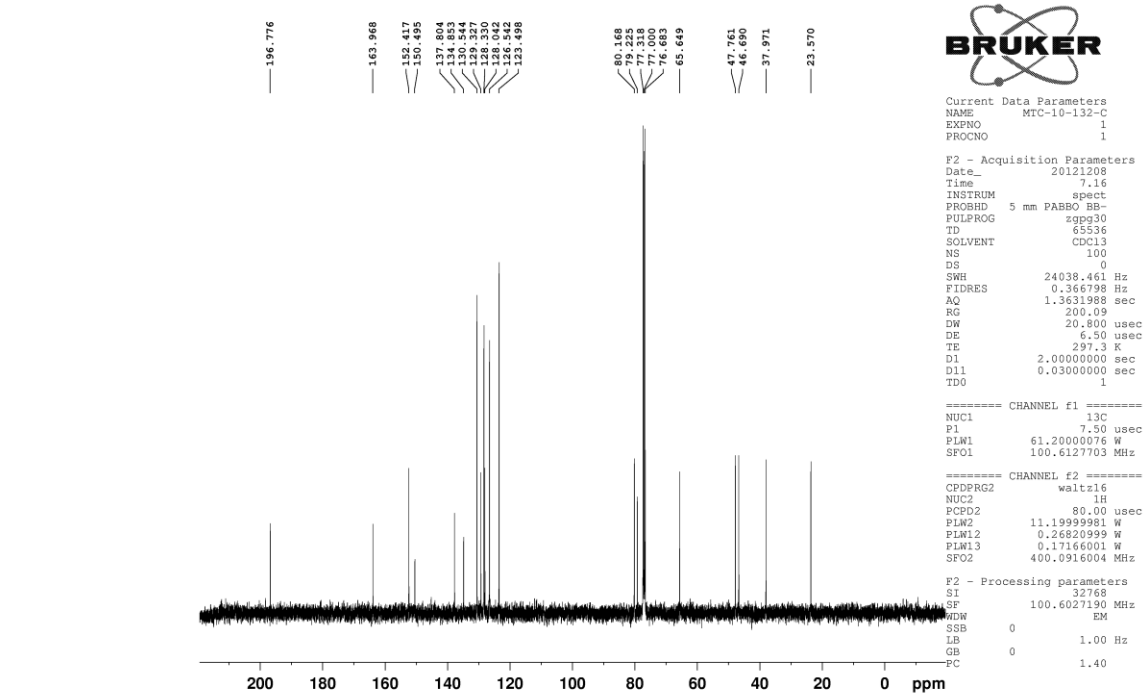
F2 - Acquisition Parameters
Date_           20130119
Time                6.09
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  150
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9088159 sec
RG                  203
DW               13.867 usec
DE               18.00 usec
TE               295.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

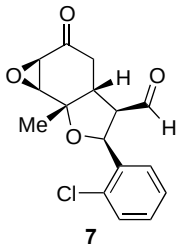
======== CHANNEL f1 ========
NUC1                  13C
P1                 11.00 usec
PLW1        230.00000000 W
SFO1        150.9178981 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2         13.00000000 W
PLW12         0.25077000 W
PLW13         0.20313001 W
SFO2        600.1324005 MHz

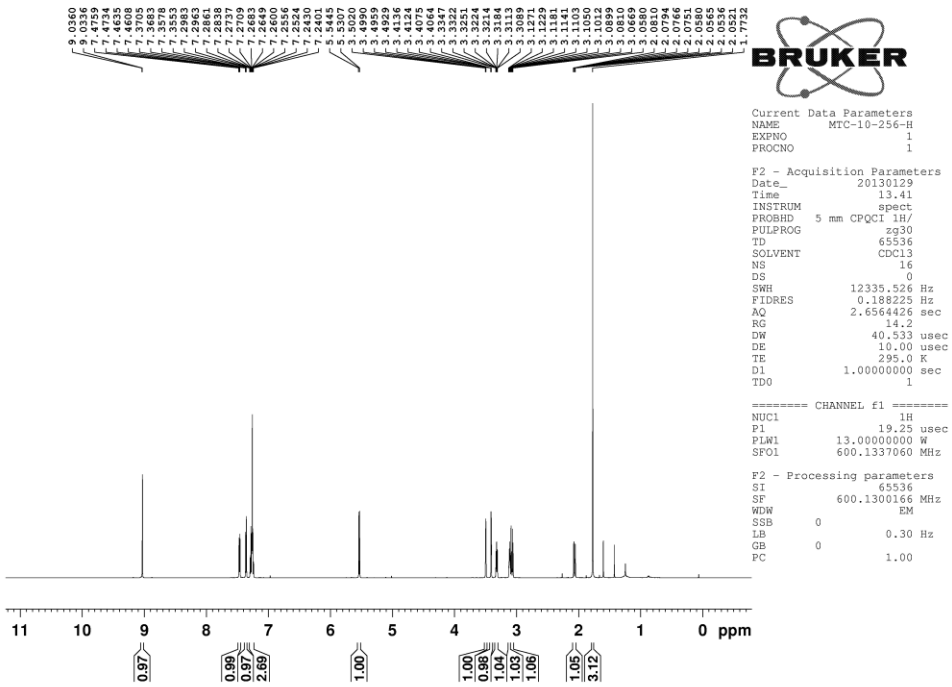
F2 - Processing parameters
SI                 32768
SF          150.9028184 MHz
WDW                   EM
SSB       0
LB             1.00 Hz
GB        0
PC                 1.40
```


8.1637
8.1415
7.9134
7.8933
7.8735
7.2049
7.1868
7.1695
7.1520
7.1346
7.1303
7.1243
7.1149
7.1052
7.1004
7.0975
6.6502
6.6456
6.6245
6.6200
6.0067
5.9811
5.0364
5.0144
3.9518
3.9181
3.9008
3.8896
3.8732
3.8562
3.8451
3.8276
2.7438
2.7263
2.7103
2.7039
2.6929
2.6860
2.6813
2.6652
2.6503
2.6392
2.3753
2.3704
2.3653
2.3588
2.3525
2.3471
1.5795
O
H
Me
O
O
O
NO2
6
9
8
7
6
5
4
3
2
1
0 ppm
2.07
1.96
5.59
1.04
1.03
1.00
2.10
3.17
1.05
3.10
BRUKER
Current Data Parameters
NAME MTC-10-132-H
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121208
Time 7.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 32.04
DW 60.800 usec
DE 6.50 usec
TE 296.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.38 usec
PLW1 11.19999981 W
SFO1 400.0924707 MHz
F2 - Processing parameters
SI 65536
SF 400.0900499 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


196.776
163.968
152.417
150.495
137.804
134.853
130.544
129.327
128.330
128.042
126.542
123.498
80.168
79.225
77.318
77.000
76.683
65.649
47.761
46.690
37.971
23.570
200
180
160
140
120
100
80
60
40
20
0 ppm
BRUKER
Current Data Parameters
NAME MTC-10-132-C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121208
Time 7.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 100
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 200.09
DW 20.800 usec
DE 6.50 usec
TE 297.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.50 usec
PLW1 61.20000076 W
SFO1 100.6127703 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 11.19999981 W
PLW12 0.26820999 W
PLW13 0.17166001 W
SFO2 400.0916004 MHz
F2 - Processing parameters
SI 32768
SF 100.6027190 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



7



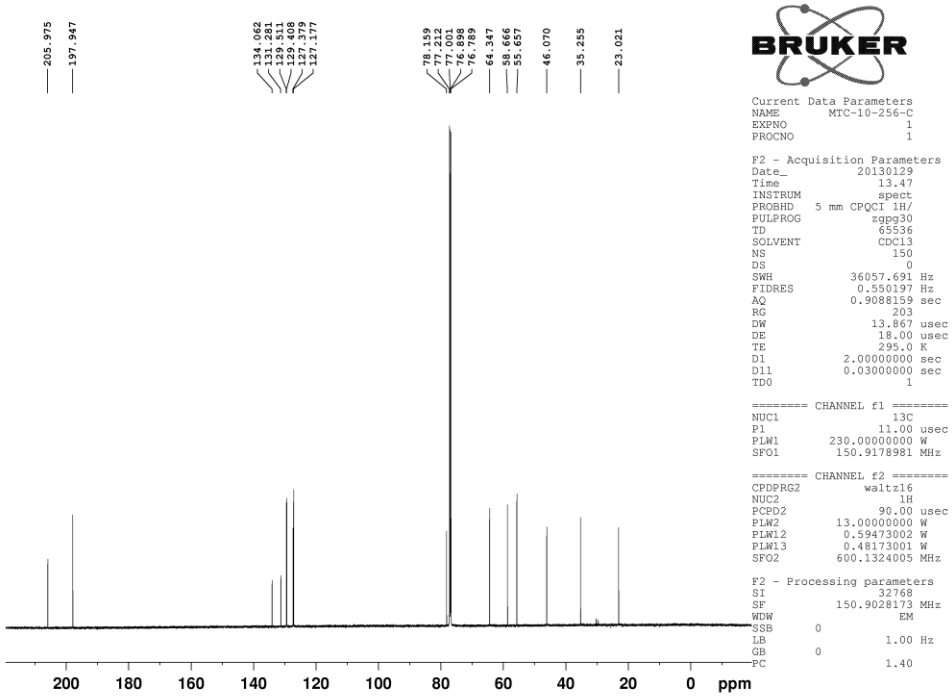
BRUKER

```
Current Data Parameters
NAME        MTC-10-256-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130129
Time               13.41
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                 14.2
DW               40.533 usec
DE               10.00 usec
TE                295.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                19.25 usec
PLW1        13.00000000 W
SFO1       600.1337060 MHz

F2 - Processing parameters
SI                65536
SF           600.1300166 MHz
WDW                  EM
SSB       0
LB              0.30 Hz
GB        0
PC              1.00
```



BRUKER

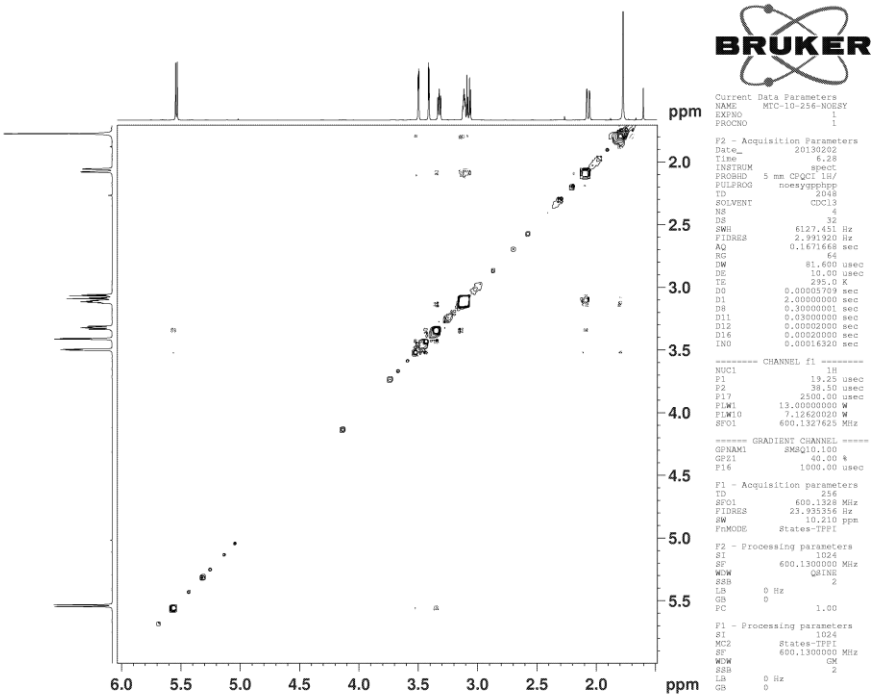
```
Current Data Parameters
NAME        MTC-10-256-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130129
Time               13.47
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  150
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9088159 sec
RG                  203
DW               13.867 usec
DE                18.00 usec
TE                295.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                11.00 usec
PLW1       230.00000000 W
SFO1       150.9178981 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2        13.00000000 W
PLW12        0.59473002 W
PLW13        0.48173001 W
SFO2       600.1324005 MHz

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



BRUKER

Current Data Parameters
NAME MTC-10-256-NOESY
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130202
Time 6.28
INSTRUM spect
PROBHD 5 mm CPQCI 1H/
PULPROG noesygpphpp
TD 2048
SOLVENT CDCl3
NS 4
DS 32
SWH 6127.451 Hz
FIDRES 2.991920 Hz
AQ 0.1671668 sec
RG 64
DW 81.600 usec
DE 10.00 usec
TE 295.0 K
D0 0.00005709 sec
D1 2.00000000 sec
D8 0.30000001 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00016320 sec

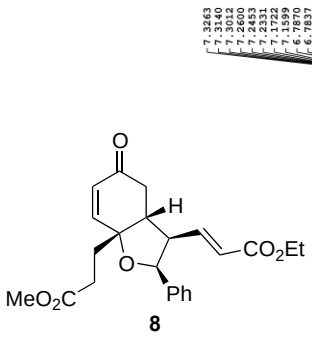
======== CHANNEL f1 ========
NUC1 1H
P1 19.25 usec
P2 38.50 usec
P17 2500.00 usec
PLW1 13.00000000 W
PLW10 7.12620020 W
SFO1 600.1327625 MHz

====== GRADIENT CHANNEL =====
GPNAM1 SMSQ10.100
GPZ1 40.00 %
P16 1000.00 usec

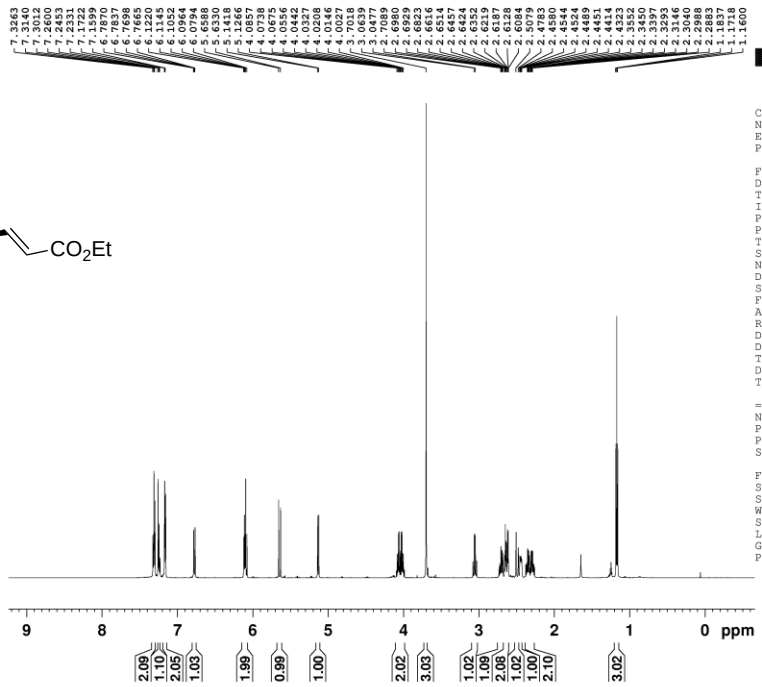
F1 - Acquisition parameters
TD 256
SFO1 600.1328 MHz
FIDRES 23.935356 Hz
SW 10.210 ppm
FnMODE States-TPPI

F2 - Processing parameters
SI 1024
SF 600.1300000 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 States-TPPI
SF 600.1300000 MHz
WDW GM
SSB 2
LB 0 Hz
GB 0



8

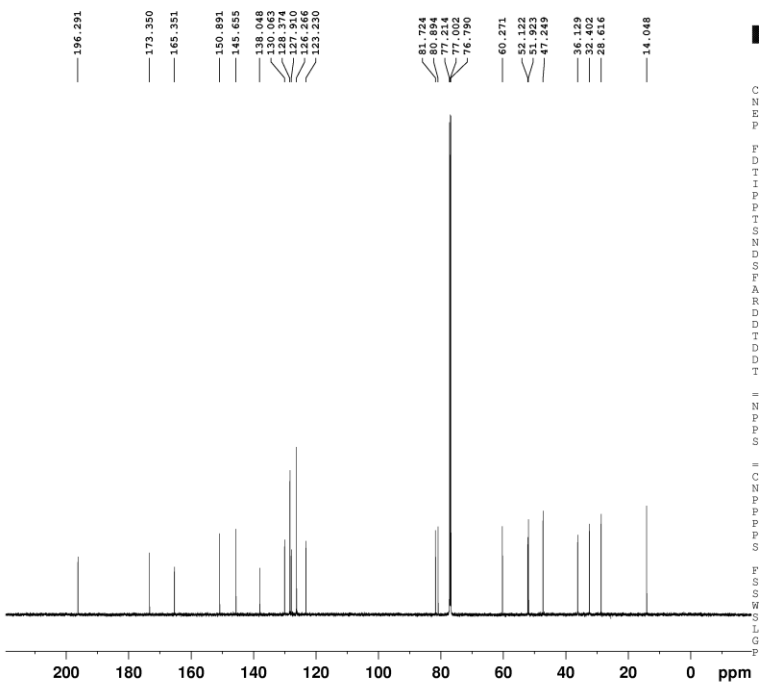



```
Current Data Parameters
NAME        MTC-10-260-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130202
Time                6.16
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                 12.7
DW               40.533 usec
DE               10.00 usec
TE               295.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 19.25 usec
PLW1         13.00000000 W
SFO1        600.1337060 MHz

F2 - Processing parameters
SI                 65536
SF           600.1300165 MHz
WDW                   EM
SSB       0
LB             0.30 Hz
GB        0
PC                 1.00
```



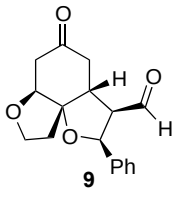

```
Current Data Parameters
NAME        MTC-10-260-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130202
Time                6.21
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  100
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9088159 sec
RG                  203
DW               13.867 usec
DE               18.00 usec
TE               295.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

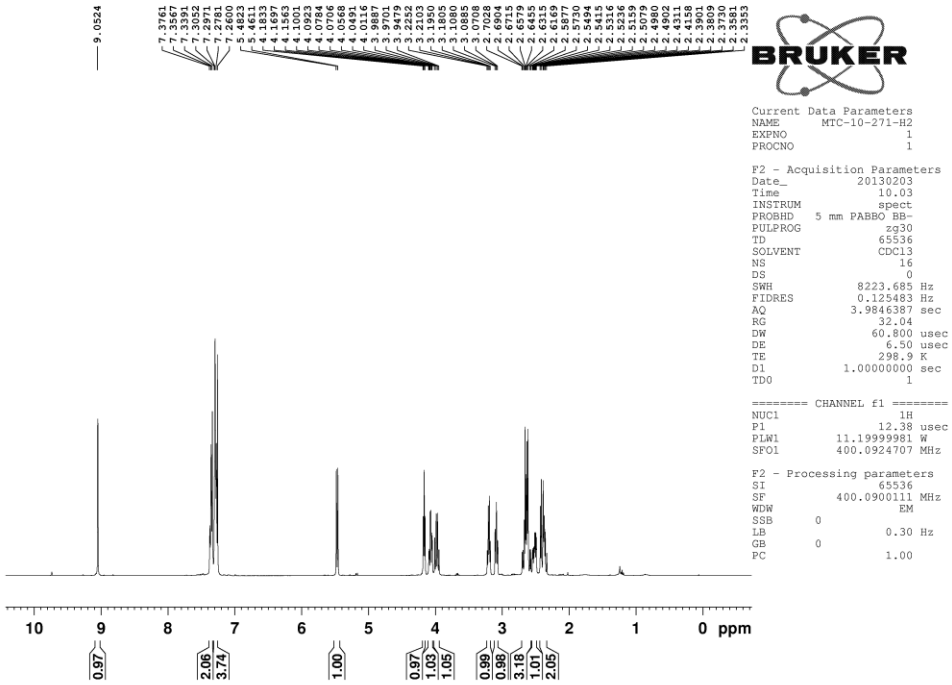
======== CHANNEL f1 ========
NUC1                  13C
P1                 11.00 usec
PLW1        230.00000000 W
SFO1        150.9178981 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2         13.00000000 W
PLW12         0.59473002 W
PLW13         0.48173001 W
SFO2        600.1324005 MHz

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



9



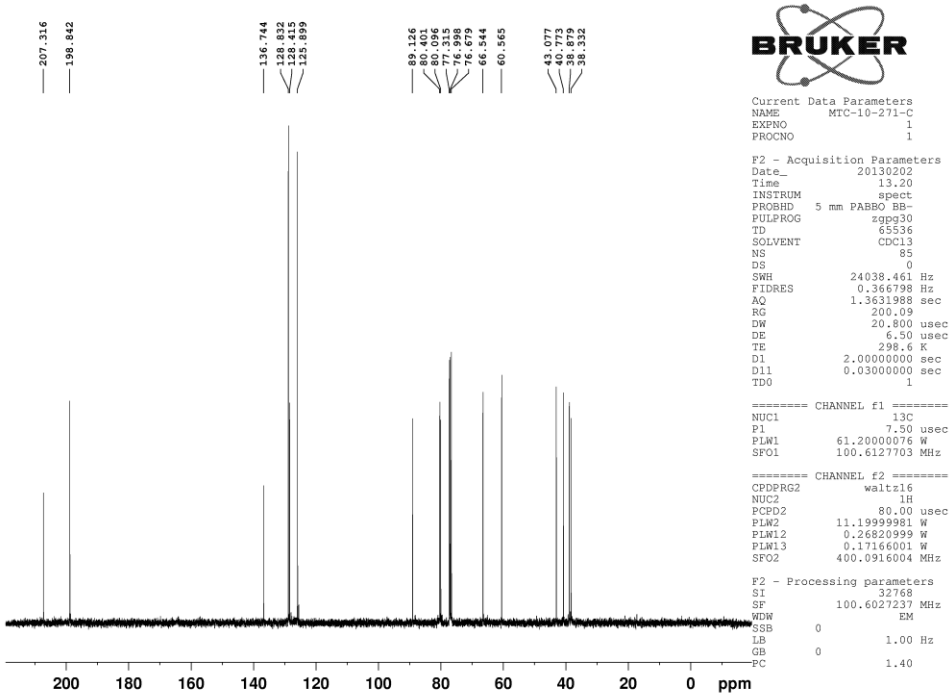
BRUKER

```
Current Data Parameters
NAME       MTC-10-271-H2
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20130203
Time               10.03
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                32.04
DW               60.800 usec
DE                6.50 usec
TE                298.9 K
D1            1.00000000 sec
TD0                   1

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

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



BRUKER

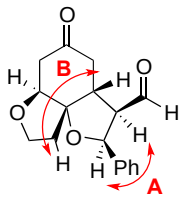
```
Current Data Parameters
NAME       MTC-10-271-C
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20130202
Time               13.20
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   85
DS                    0
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                200.09
DW               20.800 usec
DE                6.50 usec
TE                298.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

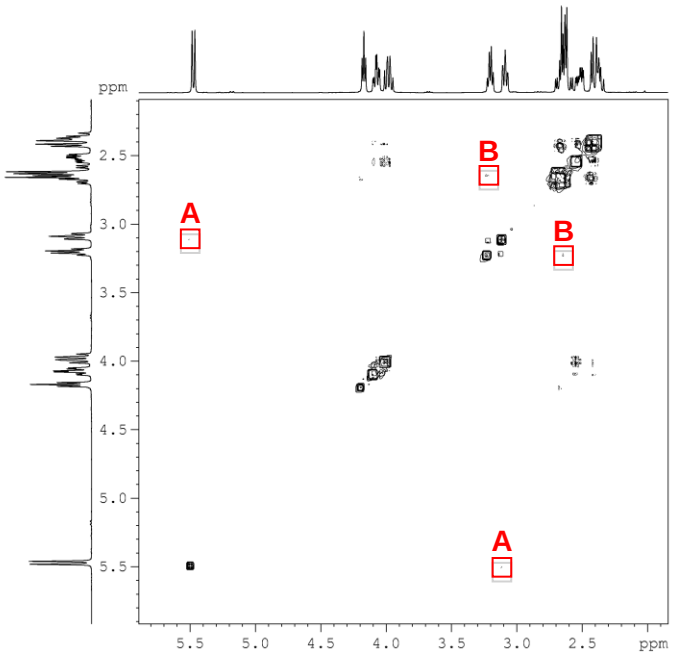
======== CHANNEL f1 ========
NUC1                 13C
P1                7.50 usec
PLW1         61.20000076 W
SFO1        100.6127703 MHz

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

F2 - Processing parameters
SI                32768
SF          100.6027237 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



9




```
Current Data Parameters
NAME      MTC-10-271-NOESY
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20130204
Time                   7.02
INSTRUM               spect
PROBHD   5 mm PABBO BA-
PULPROG       noesygpphpp
TD                    2048
SOLVENT               CDCl3
NS                       4
DS                       0
SWH               4084.967 Hz
FIDRES            1.996613 Hz
AQ                0.2506752 sec
RG                    59.75
DW                  122.400 usec
DE                    6.50 usec
TE                  297.9 K
D0                0.00010664 sec
D1                2.00000000 sec
D8                0.30000001 sec
D11               0.03000000 sec
D12               0.00002000 sec
D16               0.00020000 sec
IN0               0.00024480 sec

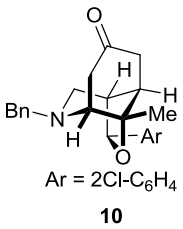
======== CHANNEL f1 ========
NUC1                     1H
P1                    12.38 usec
P2                    24.76 usec
P17                 2500.00 usec
PLW1              11.19999981 W
PLW10              1.67930002 W
SFO1              400.0918417 MHz

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

F1 - Acquisition parameters
TD                       256
SFO1              400.0918 MHz
FIDRES            15.956903 Hz
SW                   10.210 ppm
FnMODE           States-TPPI

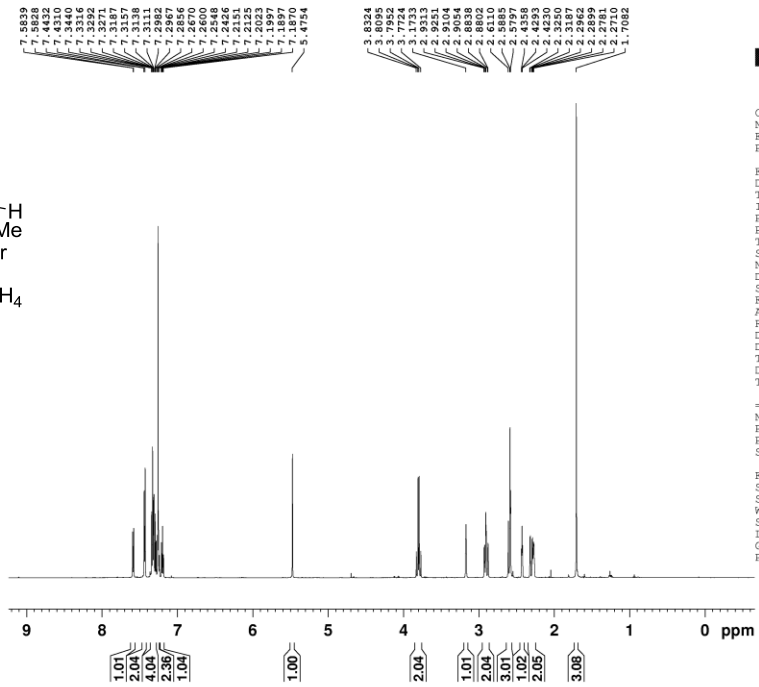
F2 - Processing parameters
SI                       1024
SF                400.0900000 MHz
WDW                     QSINE
SSB                         2
LB           0 Hz
GB           0
PC                       1.00

F1 - Processing parameters
SI                       1024
MC2              States-TPPI
SF                400.0900000 MHz
WDW                     QSINE
SSB                         2
LB           0 Hz
GB           0
```



Ar = 2Cl-C_6H_4

10

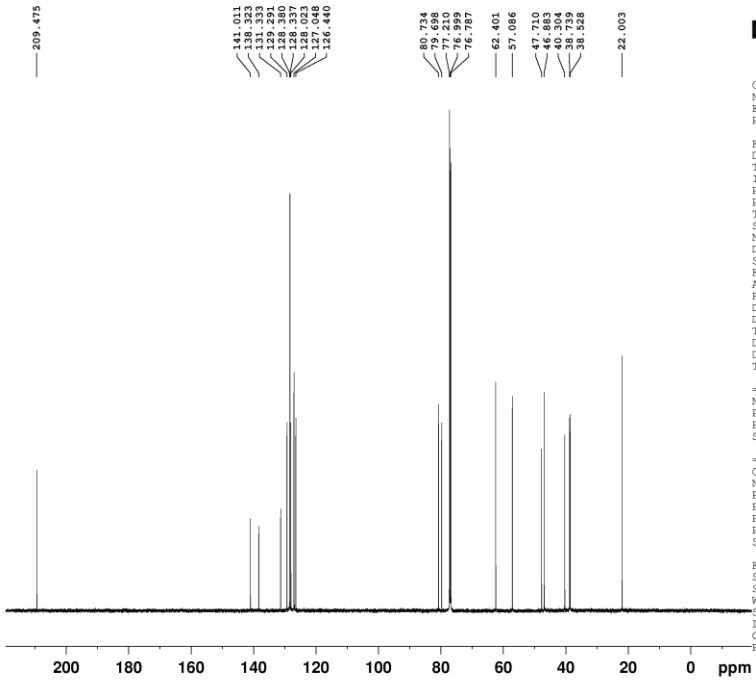


```
Current Data Parameters
NAME         MTC-11-6B-H
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130212
Time                9.01
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    0
SWH           12335.526 Hz
FIDRES         0.188225 Hz
AQ            2.6564426 sec
RG                 12.7
DW               40.533 usec
DE               10.00 usec
TE               295.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 19.25 usec
PLW1         13.00000000 W
SFO1        600.1337060 MHz

F2 - Processing parameters
SI                 65536
SF          600.1300165 MHz
WDW                   EM
SSB       0
LB               0.30 Hz
GB        0
PC               1.00
```



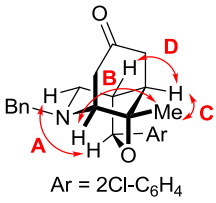
```
Current Data Parameters
NAME         MTC-11-6B-C
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20130212
Time                9.05
INSTRUM            spect
PROBHD   5 mm CPQCI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   55
DS                    0
SWH           36057.691 Hz
FIDRES         0.550197 Hz
AQ            0.9088159 sec
RG                   203
DW               13.867 usec
DE               18.00 usec
TE               295.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 11.00 usec
PLW1        230.00000000 W
SFO1        150.9178981 MHz

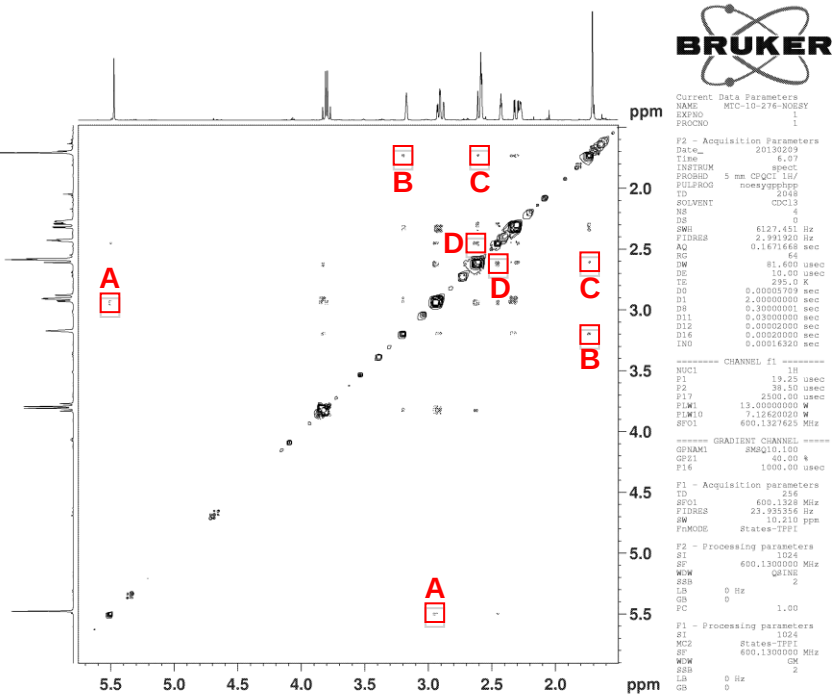
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2         13.00000000 W
PLW12         0.59473002 W
PLW13         0.48173001 W
SFO2        600.1324005 MHz

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



Ar = 2Cl-C_6H_4

10



Current Data Parameters
NAME MTC-10-276-NOESY
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130209
Time 6.07
INSTRUM spect
PROBHD 5 mm CPQCI 1H/
PULPROG noesygpphpp
TD 2048
SOLVENT CDCl3
NS 4
DS 0
SWH 6127.451 Hz
FIDRES 2.991920 Hz
AQ 0.1671668 sec
RG 64
DW 81.600 usec
DE 10.00 usec
TE 295.0 K
D0 0.00005709 sec
D1 2.00000000 sec
D8 0.30000001 sec
D11 0.03000000 sec
D12 0.00002000 sec
D16 0.00020000 sec
IN0 0.00016320 sec

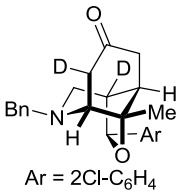
======== CHANNEL f1 ========
NUC1 1H
P1 19.25 usec
P2 38.50 usec
P17 2500.00 usec
PLW1 13.00000000 W
PLW10 7.12620020 W
SFO1 600.1327625 MHz

====== GRADIENT CHANNEL =====
GPNAM1 SMSQ10.100
GPZ1 40.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 256
SFO1 600.1328 MHz
FIDRES 23.935356 Hz
SW 10.210 ppm
FnMODE States-TPPI

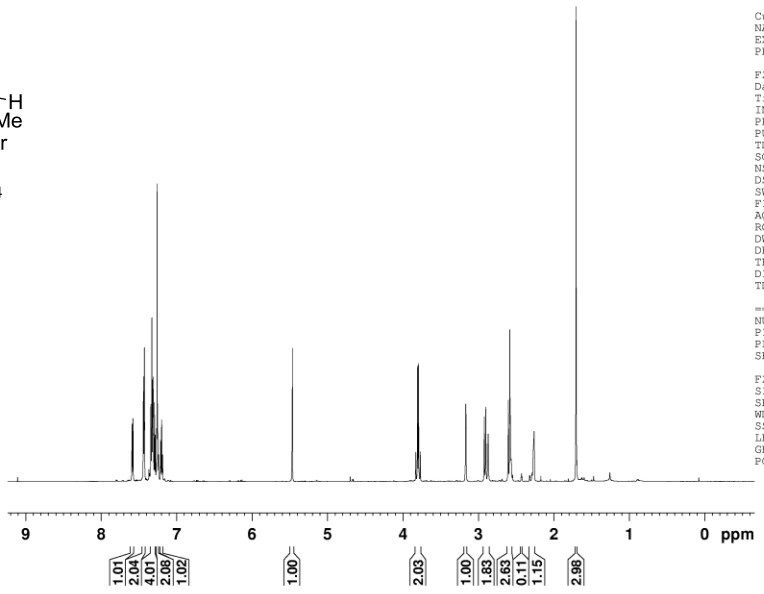
F2 - Processing parameters
SI 1024
SF 600.1300000 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 1024
MC2 States-TPPI
SF 600.1300000 MHz
WDW GM
SSB 2
LB 0 Hz
GB 0



Ar = 2Cl-C_6H_4

10-D

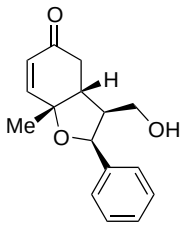


Current Data Parameters
NAME MTC-11-7-H
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130212
Time 15.16
INSTRUM spect
PROBHD 5 mm CPQCI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 14.2
DW 40.533 usec
DE 10.00 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 19.25 usec
PLW1 13.00000000 W
SFO1 600.1337060 MHz

F2 - Processing parameters
SI 65536
SF 600.1300164 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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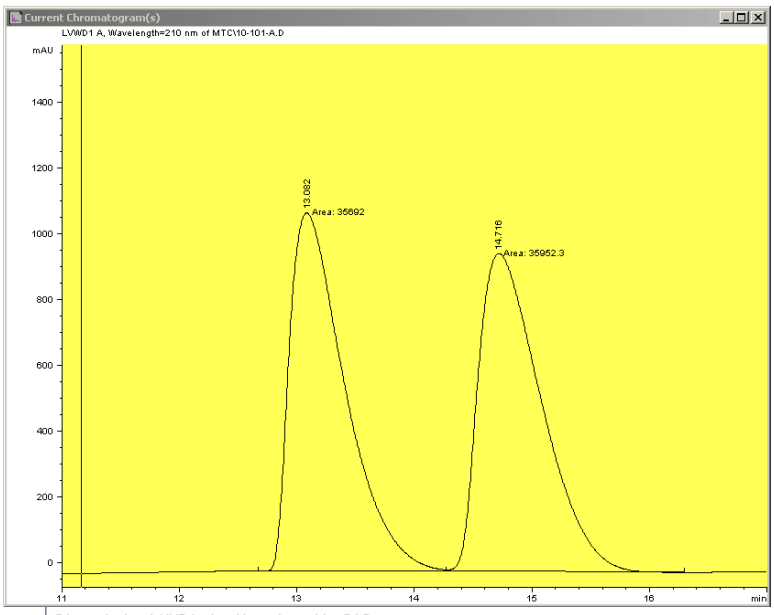
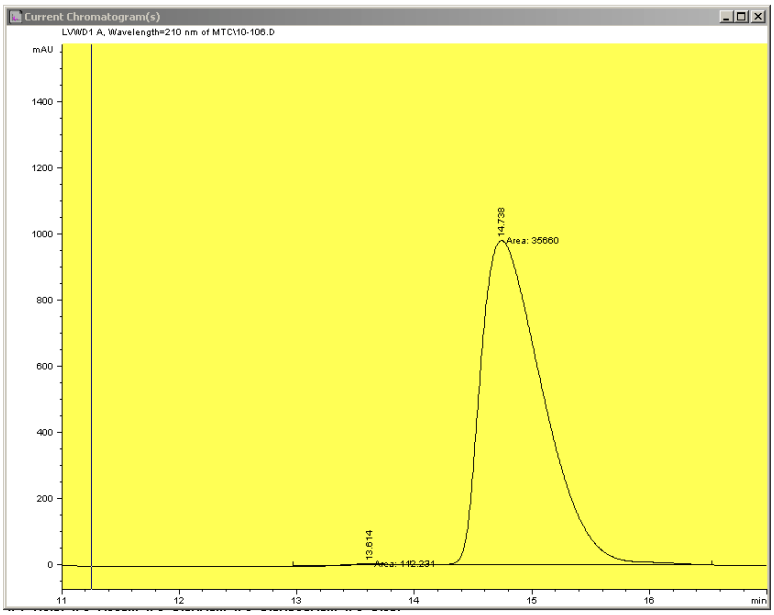


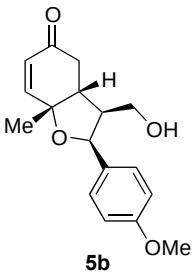
Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	13.082	MF	0.546	35692.03906	1089.90076	49.8184
2	14.716	FM	0.619	35952.25781	967.94873	50.1816
Totals :				71644.29687	2057.84961	



Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	13.614	MM	0.423	112.23133	4.41831	0.3137
2	14.738	MM	0.606	35660.02734	980.88129	99.6863
Totals :				35772.25781	985.29962	



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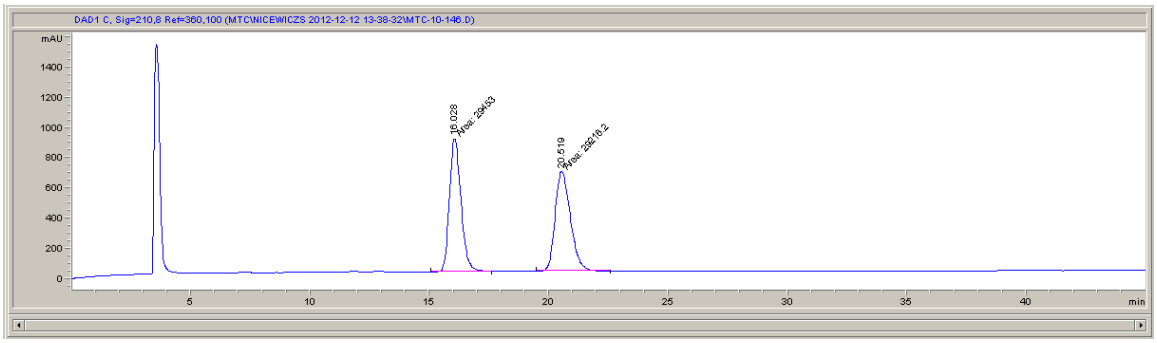
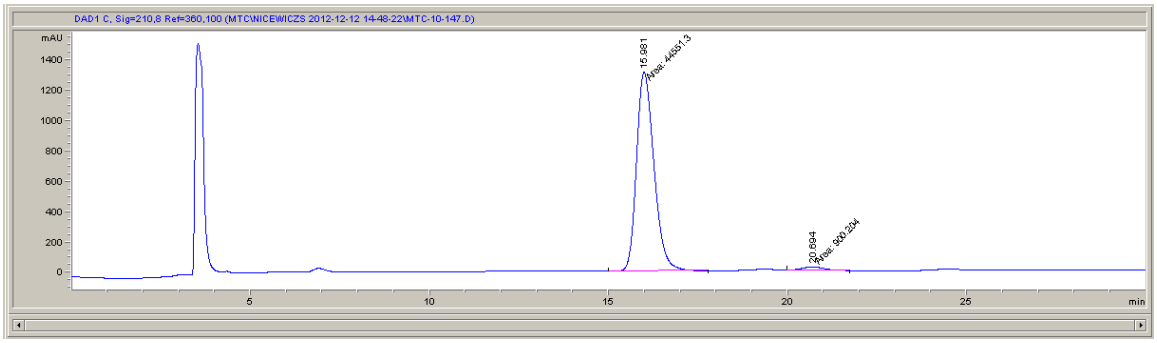
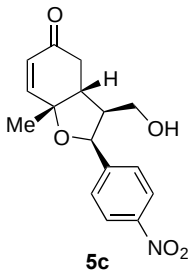


#	Time	Area	Height	Width	Area%	Symmetry
1	16.028	29453	879.7	0.558	50.202	0.762
2	20.519	29216.2	657.8	0.7403	49.798	0.733



#	Time	Area	Height	Width	Area%	Symmetry
1	15.981	44551.3	1307.7	0.5678	98.019	0.734
2	20.694	900.2	21.7	0.6915	1.981	0.87



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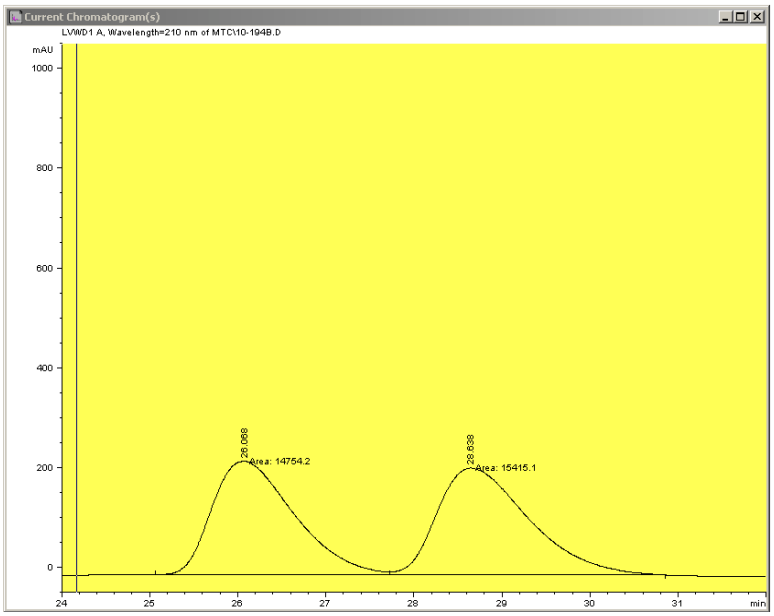
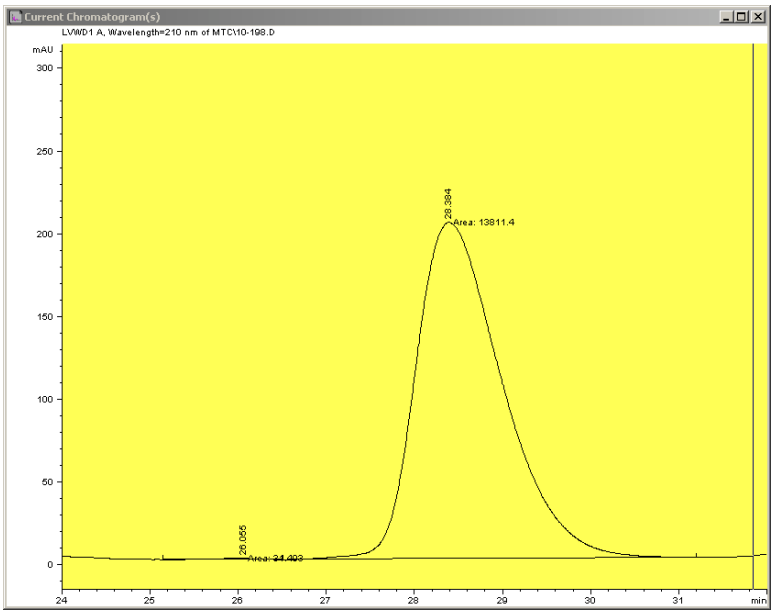


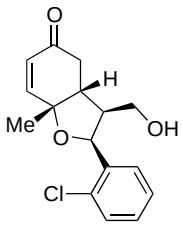
Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	26.068	MF	1.079	14754.19629	227.85472	48.9047
2	28.638	FM	1.199	15415.10938	214.35262	51.0953
Totals :				30169.30469	442.20734	



Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	26.055	MM	0.664	31.40295	7.88319e-1	0.2269
2	28.384	MM	1.131	13811.40625	203.54843	99.7731
Totals :				13842.80957	204.33675	



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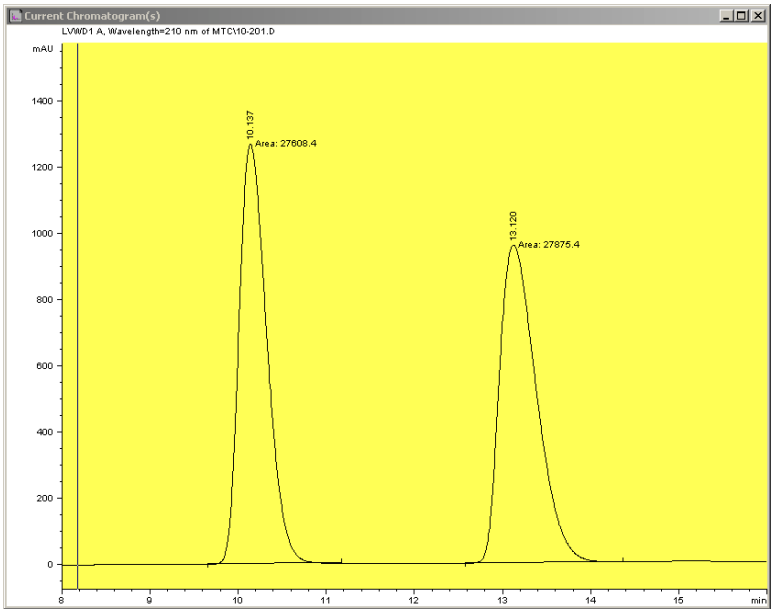
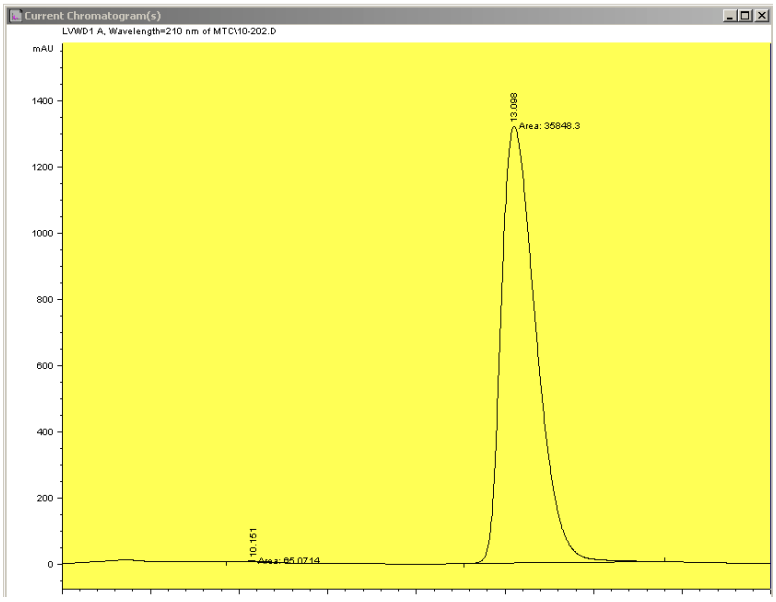


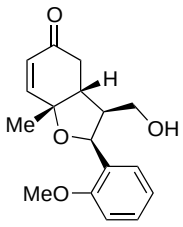
Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	10.137	MM	0.363	27608.39648	1268.78271	49.7594
2	13.120	MM	0.484	27875.42188	959.61017	50.2406
Totals :				55483.82031	2228.39282	



Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	10.151	MM	0.293	65.07142	3.70250	0.1812
2	13.098	MM	0.453	35848.25391	1320.22339	99.8188
Totals :				35913.32422	1323.92590	



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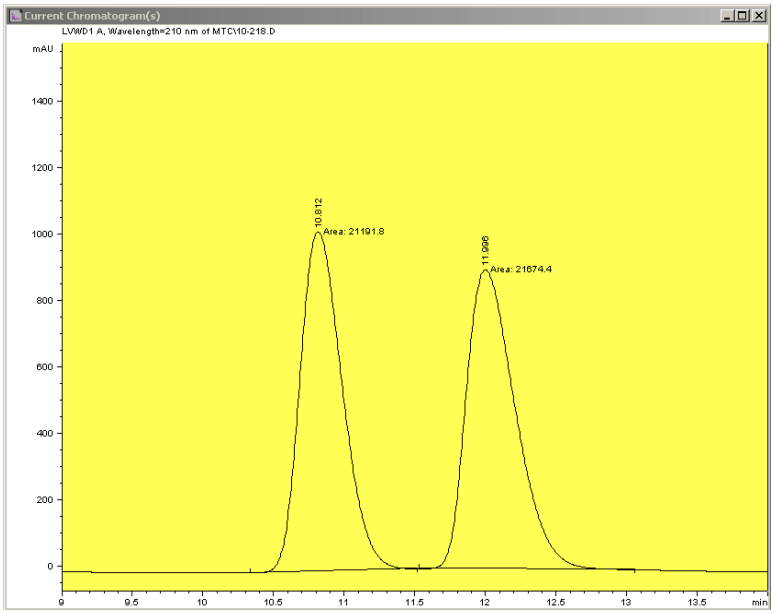
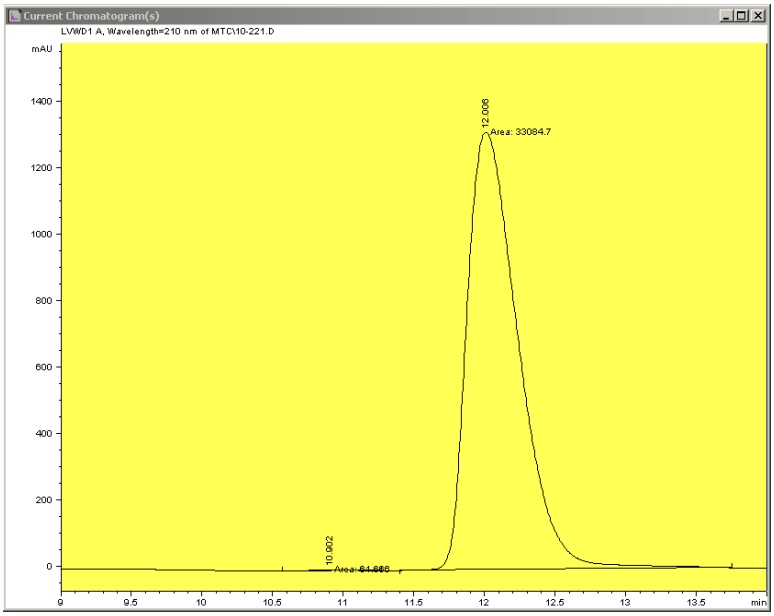


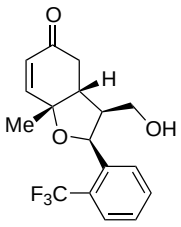
Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	10.812	MM	0.345	21191.79883	1023.13025	49.4371
2	11.996	MM	0.400	21674.41602	902.10376	50.5629
Totals :				42866.21484	1925.23401	



Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	10.902	MM	0.280	61.66599	3.67305	0.1860
2	12.006	MM	0.418	33084.66016	1318.03906	99.8140
Totals :				33146.32422	1321.71216	



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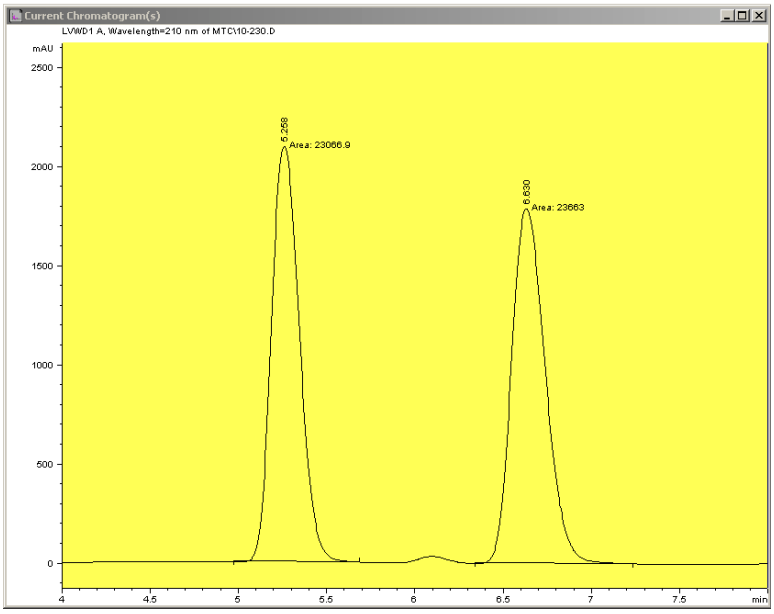
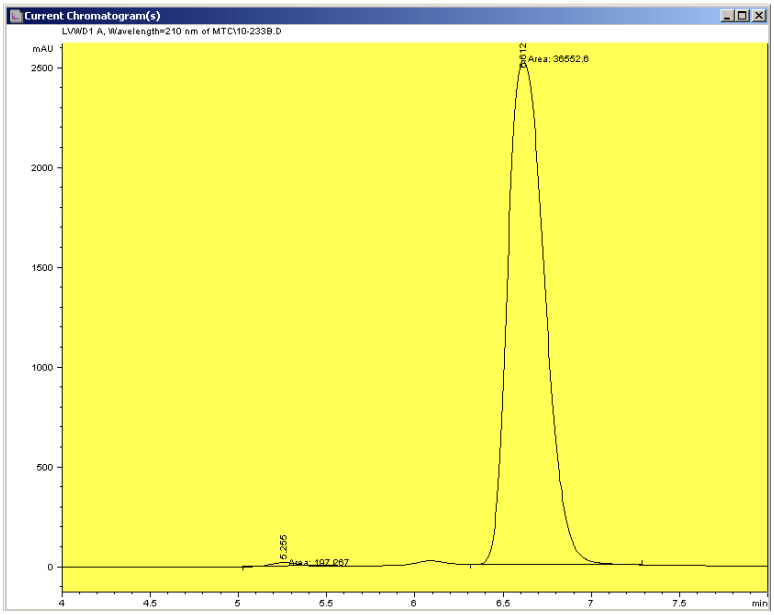


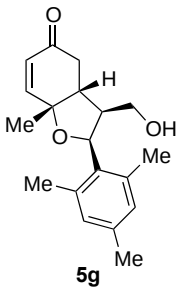
Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	5.258	MM	0.183	23066.86523	2099.76001	49.3622
2	6.630	MM	0.220	23662.95703	1793.04492	50.6378
Totals :				46729.82031	3892.80493	



Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	5.255	MM	0.173	197.26682	18.96338	0.5368
2	6.612	MM	0.241	36552.58984	2530.95337	99.4632
Totals :				36749.85547	2549.91675	



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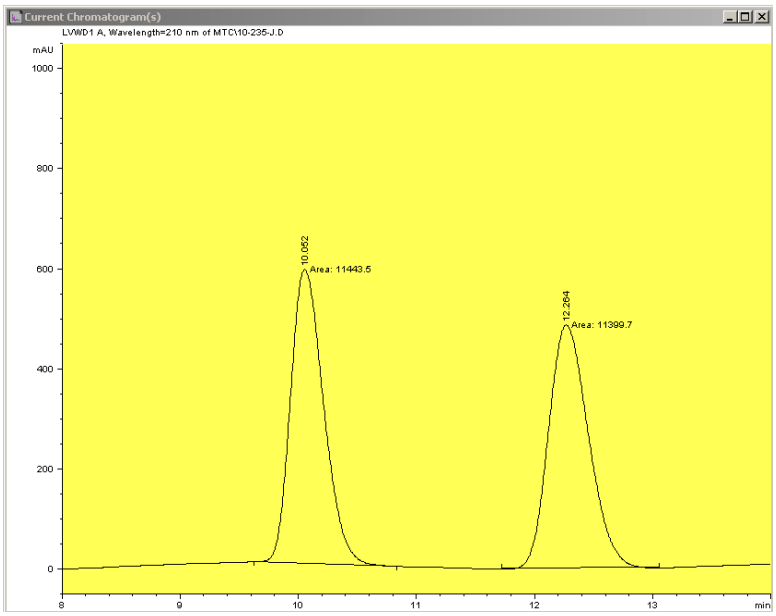
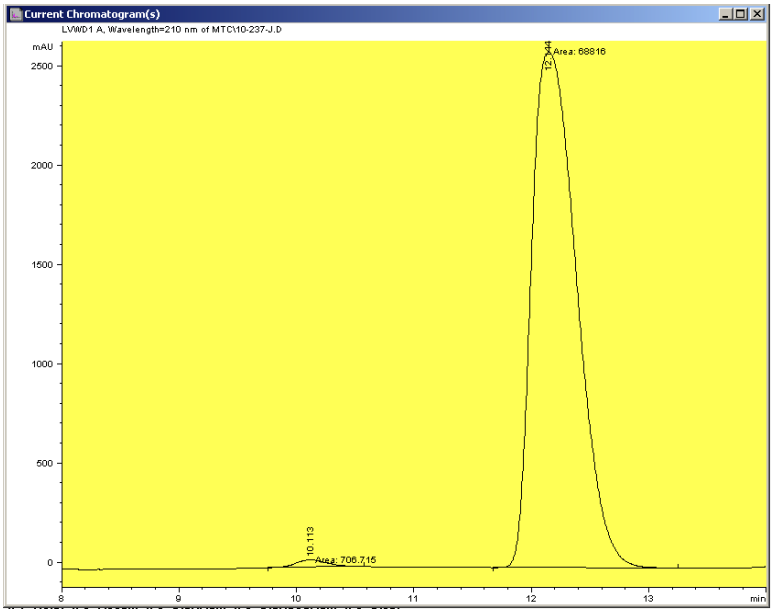


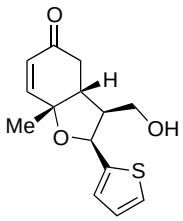
Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	10.052	MM	0.324	11443.54102	588.20996	50.0960
2	12.264	MM	0.390	11399.69922	487.26517	49.9040
Totals :				22843.24023	1075.47510	

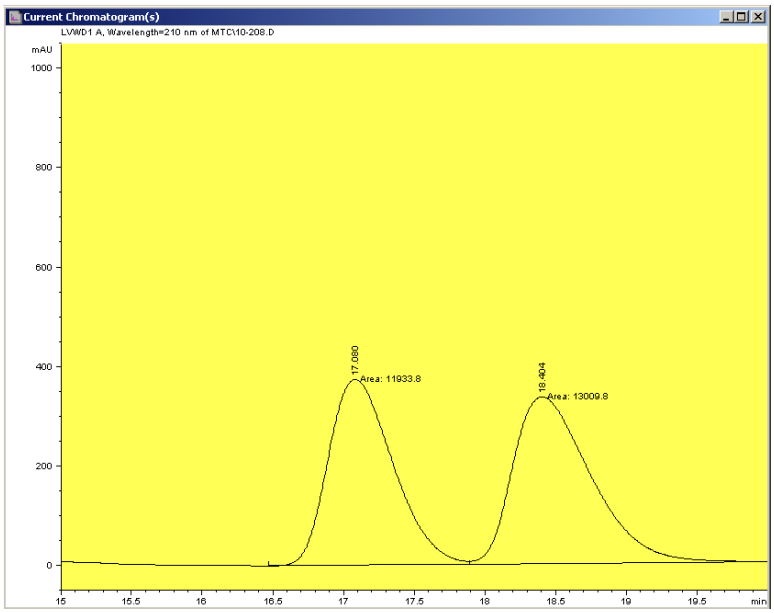


Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	10.113	MM	0.318	706.71490	37.01941	1.0165
2	12.144	MM	0.441	68816.02344	2597.90308	98.9835
Totals :				69522.74219	2634.92236	

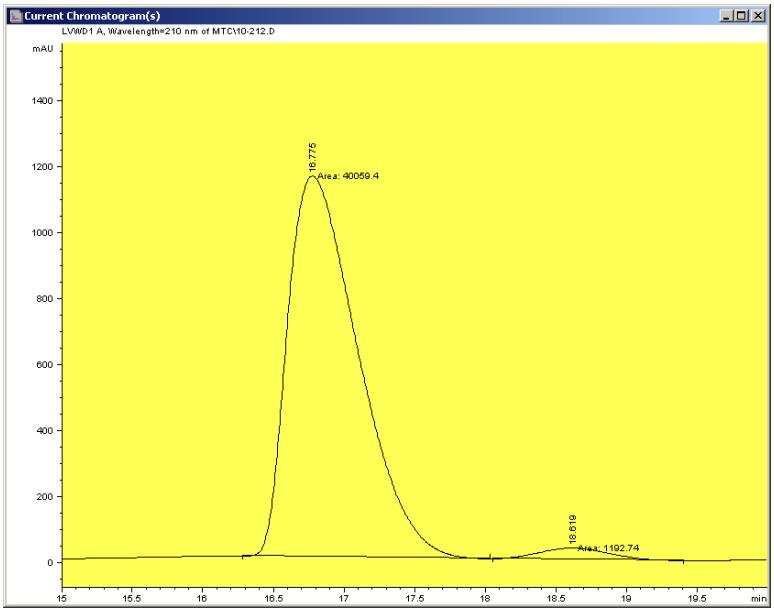


5i



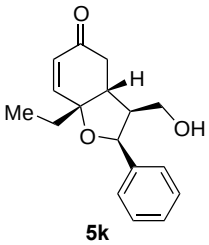
Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	17.080	MF	0.531	11933.84375	374.55807	47.8432
2	18.404	FM	0.643	13009.82324	337.10199	52.1568
Totals :				24943.66797	711.66003	

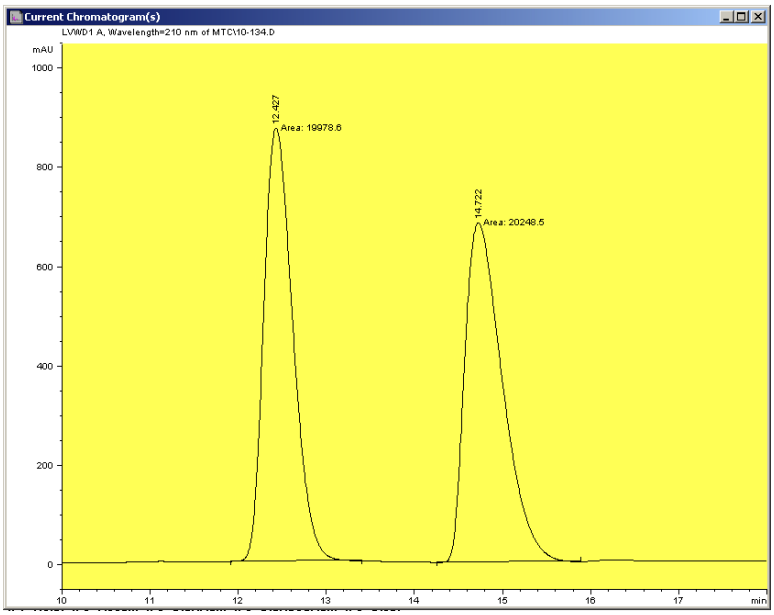


Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	16.775	MM	0.578	40059.43750	1154.59070	97.1087
2	18.619	MM	0.572	1192.73767	34.78268	2.8913
Totals :				41252.17578	1189.37341	

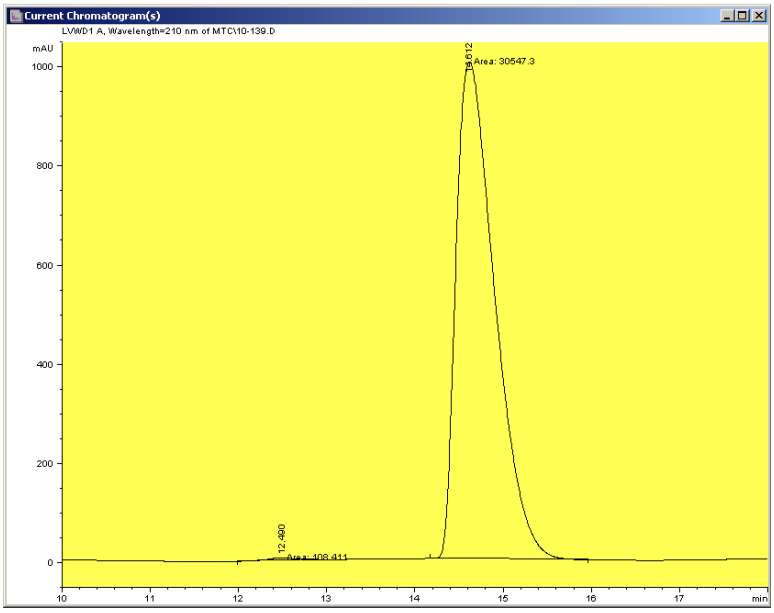


5k



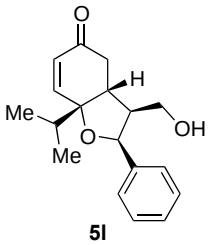
Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	12.427	MM	0.381	19978.60156	874.14465	49.6645
2	14.722	MM	0.493	20248.55078	684.43555	50.3355
Totals :				40227.15234	1558.58020	

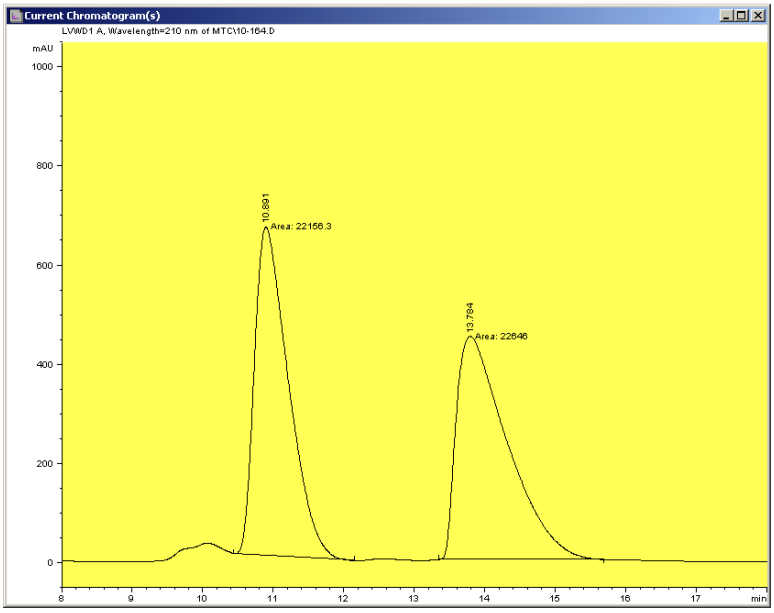


Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	12.490	MM	0.336	108.41068	5.38079	0.3536
2	14.612	MM	0.507	30547.30859	1004.37323	99.6464
Totals :				30655.71875	1009.75403	

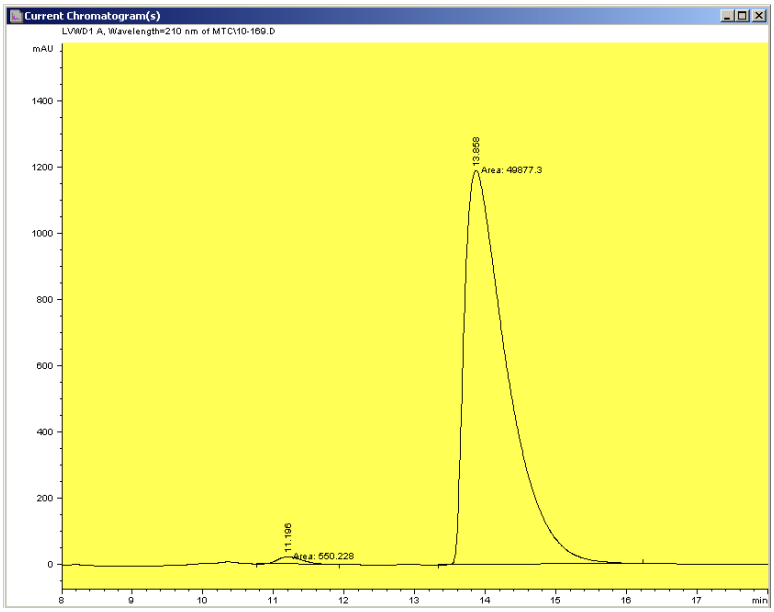


5l



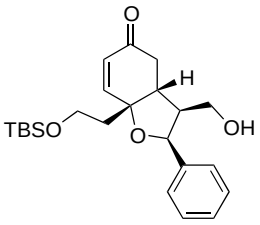
Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	10.891	MM	0.556	22156.27539	664.01410	49.4535
2	13.784	MM	0.838	22645.96875	450.47635	50.5465
Totals :				44802.24219	1114.49048	

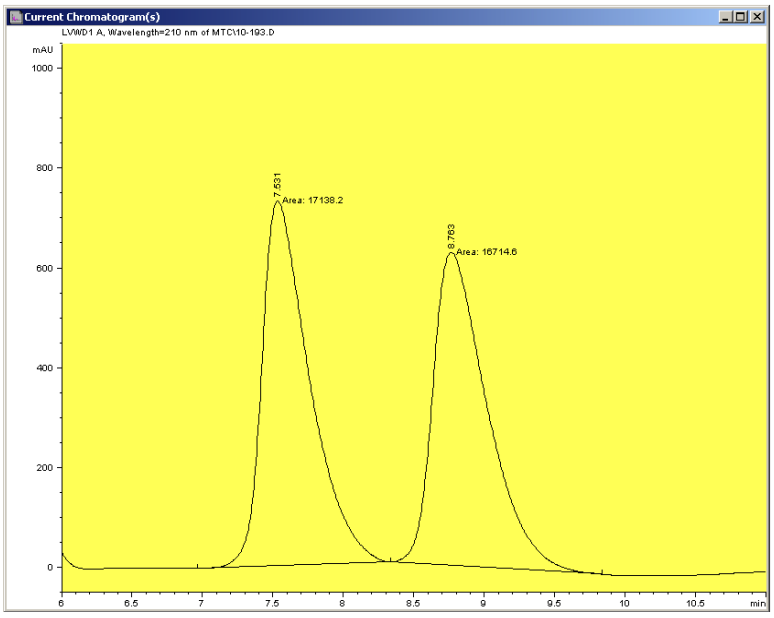


Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	11.196	MM	0.410	550.22803	22.34335	1.0911
2	13.858	MM	0.698	49877.27734	1191.04919	98.9089
Totals :				50427.50391	1213.39258	

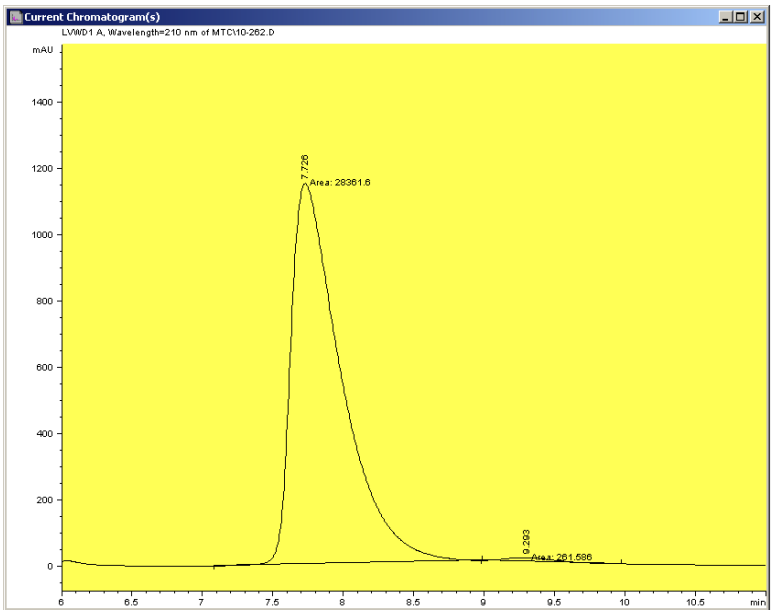


5m



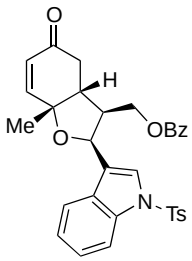
Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	7.531	MM	0.390	17138.22656	732.43677	50.6256
2	8.763	MM	0.443	16714.64258	628.76929	49.3744
Totals :				33852.86719	1361.20605	

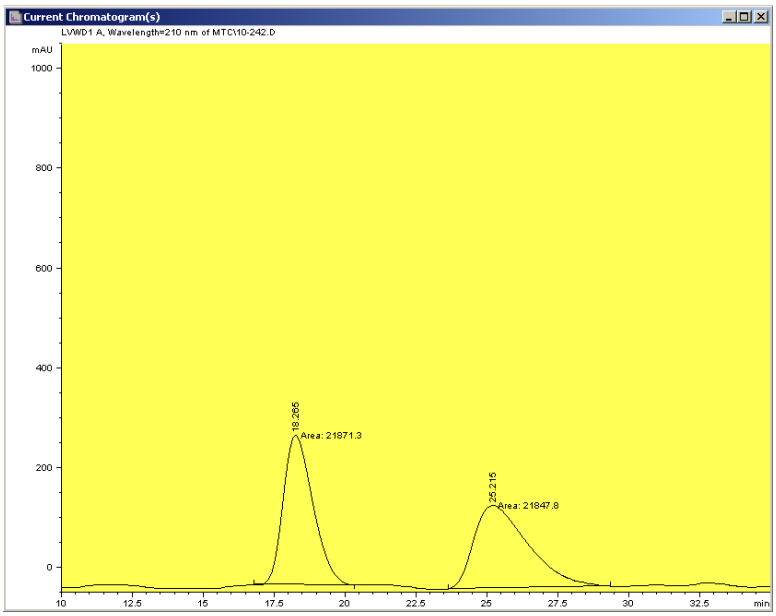


Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	7.726	MM	0.411	28361.55469	1149.12598	99.0861
2	9.293	MM	0.382	261.58557	11.41118	0.9139
Totals :				28623.14063	1160.53711	

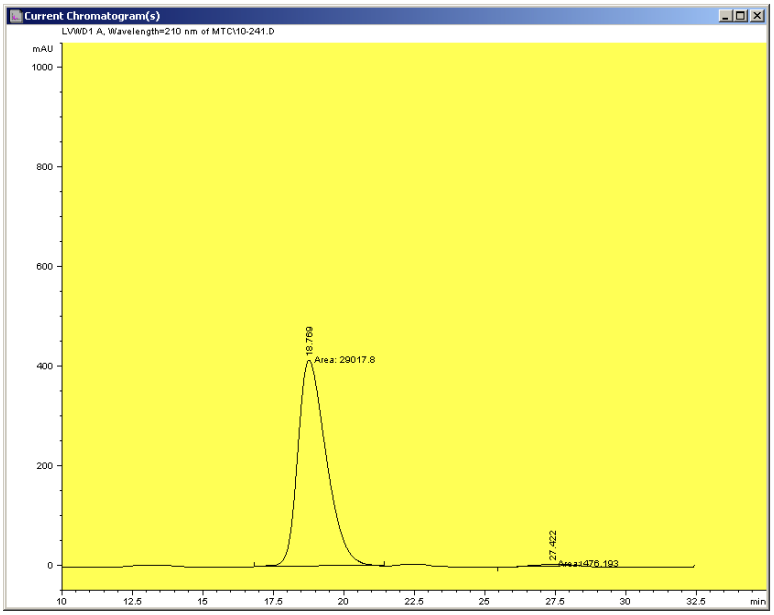


S1a



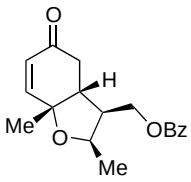
Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	18.265	MM	1.215	21871.28125	300.09641	50.0268
2	25.215	MM	2.201	21847.83203	165.43869	49.9732
Totals :				43719.11328	465.53510	

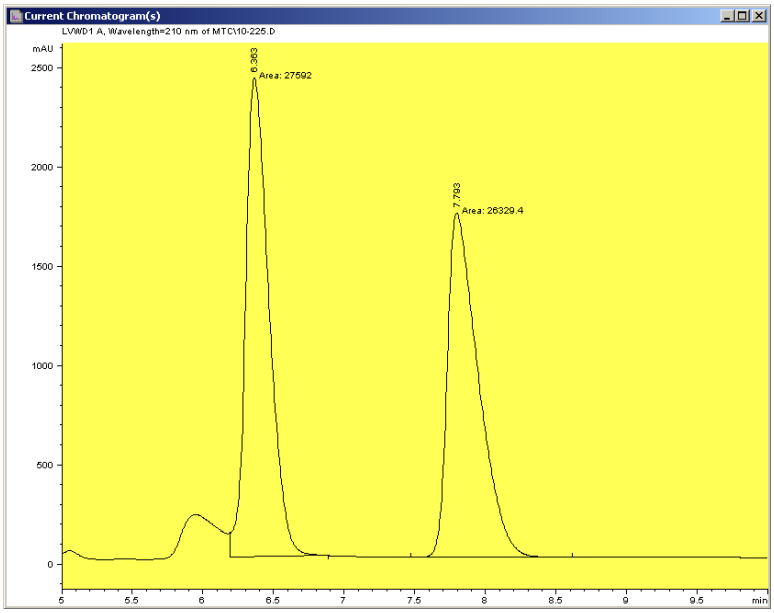


Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	18.769	MM	1.168	29017.80273	414.17654	98.3855
2	27.422	MM	1.757	476.19266	4.51676	1.6145
Totals :				29493.99609	418.69330	

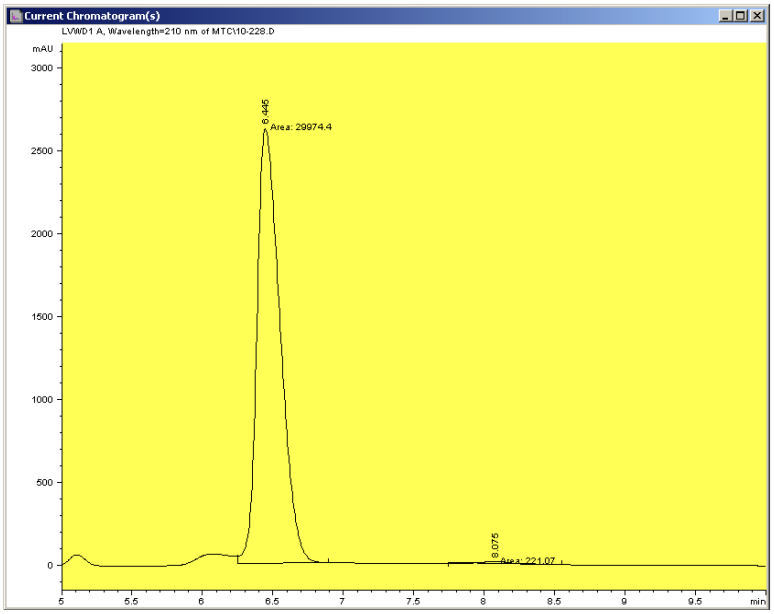


S1b



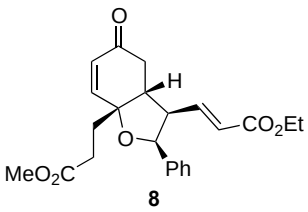
Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	6.363	FM	0.190	27591.97070	2424.08276	51.1707
2	7.793	MM	0.252	26329.44531	1742.78735	48.8293
Totals :				53921.41406	4166.87012	

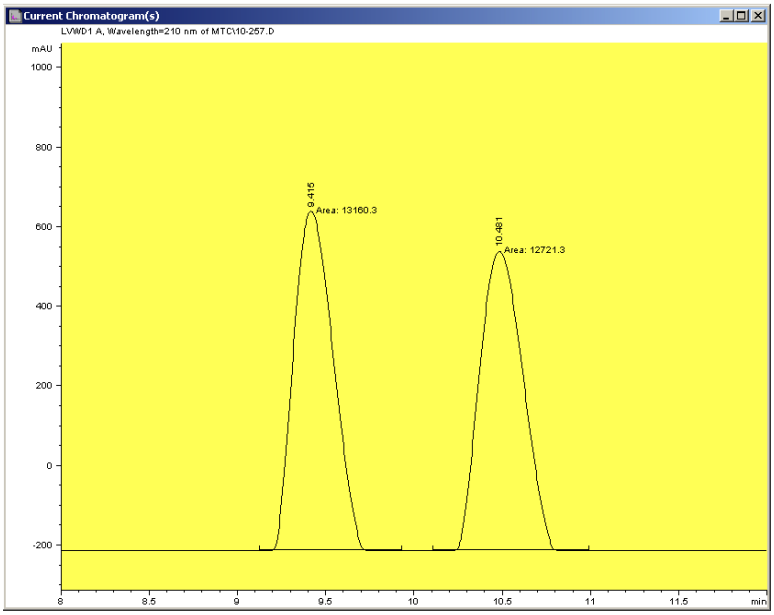


Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	6.445	FM	0.190	29974.37109	2632.51978	99.2679
2	8.075	MM	0.236	221.06999	15.60210	0.7321
Totals :				30195.44141	2648.12183	

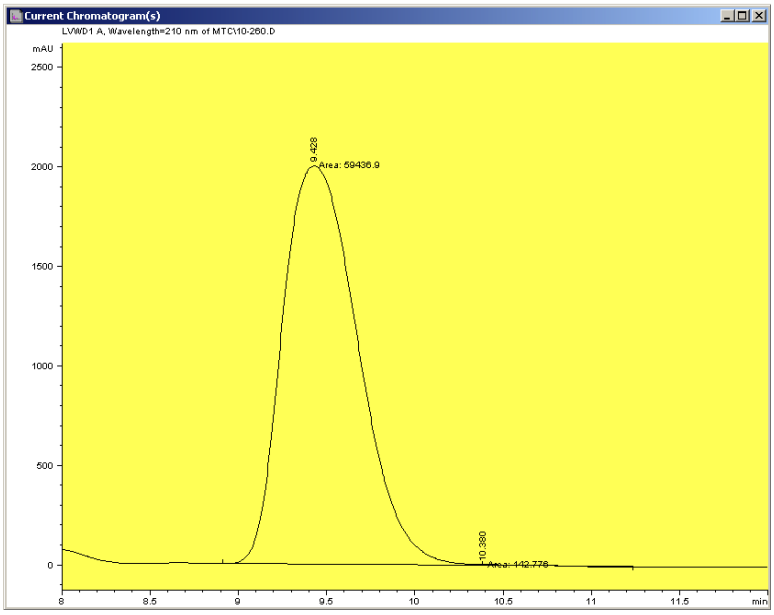


8



Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	9.415	MM	0.257	13160.33301	853.28540	50.8481
2	10.481	MM	0.282	12721.31250	753.00775	49.1519
Totals :				25881.64453	1606.29321	



Signal 1: LVWD1 A, Wavelength=210 nm

Peak #	RT [min]	Type	Width [min]	Area [mAU*sec]	Height [mAU]	Area %
1	9.428	MF	0.494	59436.91406	2004.04883	99.7604
2	10.380	FM	0.378	142.77565	6.29405	0.2396
Totals :				59579.69141	2010.34290	