Electronic Supplementary Material (ESI) for ChemComm.

Supplementary Information

Iridium-catalyzed asymmetric cyclization of alkenoic acids leading to γ-lactones

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Contents of Supplementary Information:

1. General

All anaerobic and moisture-sensitive manipulations were carried out with standard Schlenk techniques under predried nitrogen. NMR spectra were recorded on a JEOL JNM LA-500 spectrometer (500 MHz for ^{1}H) or a JEOL JNM ECA-600 spectrometer (600 MHz for ^{1}H, 91 MHz for ^{2}H, 150 MHz for ^{13}C, and 240 MHz for ^{31}P). Chemical shifts are reported in δ (ppm) referenced to the residual peaks of $CDCl_3$ (δ 7.26) and DMSO-d_6 (δ 2.50) for ^{1}H NMR, and $CDCl_3$ (δ 77.00) for ^{13}C NMR. The following abbreviations are used; s, singlet: d, doublet: t, triplet: q, quartet: sept, septet: m, multiplet: br, broad. High-resolution mass spectra were obtained with a Bruker micrOTOF spectrometer. Optical rotations were measured on a JASCO P-2200 polarimeter. Preparative thin-layer chromatography was performed with Silica Gel 60 PF_{254} (Merck). Alumina (activated 200) for column chromatography was purchased from Nacalai Tesque. Preparative recycling gel permeation chromatography was performed with JAI LC-908 equipped with JAIGEL-1H and -2H using chloroform as eluent.

2. Materials

Toluene was purified by passing through a neutral alumina column under N_2. Iridium complexes, $[IrCl(cod)]_2$[1] and $[IrCl(coe)_2]_2$,[2] were prepared according to the reported procedures.

3. Reaction optimization

$[IrCl(coe)_2]_2$ (2.2 mg, 0.0050 mmol of Ir, 5 mol% of Ir) and a ligand (0.0050 mmol, 5 mol%) were placed in a Schlenk tube under nitrogen. A solvent (0.4 mL) was added and the mixture was stirred at room temperature for 10 min. 2,2-Diphenyl-4-pentenoic acid (**1a**: 25.2 mg, 0.10 mmol) was added and the schlenk tube was capped with a glass stopper and heated at 80 °C for 20 h with stirring. The mixture was diluted with Et_2O, washed with H_2O and brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The yields of the products were determined by ^{1}H NMR using 1,4-dimethoxybenzene as an internal standard. The residue was subjected to preparative TLC on silica gel with EtOAc/hexane (1:5) to give **2a**. The ee was determined by chiral HPLC analysis (DAICEL Chiralcel OD-H).

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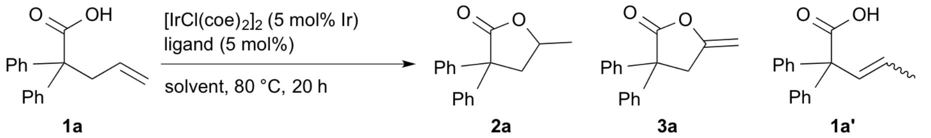
[1] J. L. Herde, J. C. Lambert and C. V. Senoff, *Inorg. Synth.,* 1974, **15**, 18.

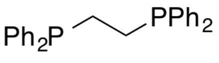
[2] R, Uson, L. A. Oro and J. A. Cabeza, *Inorg. Synth.,* 1985, **23**, 126.

Table S1. Reaction Optimization



Entry	Ligand	Solvent	**2a** (%)^b	**3a** (%)^b	**1a'** (%)^b	Ee (%)
1	dppe	1,4-Dioxane	0			
2	dppb	1,4-Dioxane	0	3		
3	dppf	1,4-Dioxane	12	16		
4^c	dppbenz	1,4-Dioxane	20			
5	(*R*,*R*)-siphos-PE	1,4-Dioxane	0		80	
6	IPr	1,4-Dioxane	<1		19	
7	(*R*)-DTBM-segphos	1,4-Dioxane	13			58
8	(*R*)-DTBM-segphos	1,2-DCE	25			28
9	(*R*)-DTBM-segphos	MeCN	36			25
10	(*R*)-DTBM-segphos	DMSO	5			N.D.^{d}
11	(*R*)-DTBM-segphos	TMU	30			76
12	(*R*)-DTBM-segphos	DMA	21			79
13	(*R*)-DTBM-segphos	NMP	39			80

^{a}Reaction conditions: **1a** (0.10 mmol), [IrCl(coe)$_2$]$_2$ (5 mol% Ir), and a ligand (5 mol%) at 80 °C for 20 h. ^{b}The yields were determined by ^{1}H NMR using 1,4-dimethoxybenene as an internal standard. ^{c}The formation of **4a** (61%) was observed. ^{d}Not determined. 1,2-DCE = 1,2-dichloroethane. TMU = 1,1,3,3-tetramethylurea. DMA = *N*,*N*-dimethylacetamide.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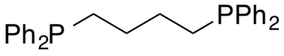
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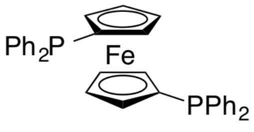
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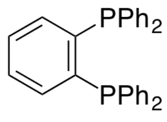
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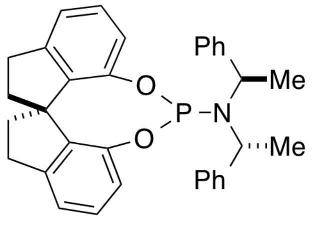
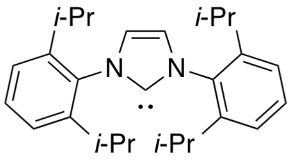
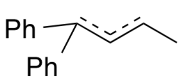
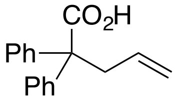

(*R*,*R*)-siphos-PE


IPr


4a

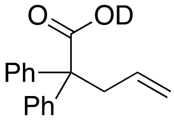
4. Preparation of alkenoic acids 1

Compound **1a** (CAS: 23132-29-8),[3] **1h** (CAS: 27326-43-8),[4] and **1j** (CAS: 46475-02-9)[5] were prepared according to the reported procedures. Compounds **1b** (CAS: 138952-03-1), **1c**, **1d** (CAS: 1397711-73-7), **1e**, **1f**, and **1g** were prepared according to the procedures for **1a**.



1a

Compound 1a. To a solution of diisopropylamine (5.3 mL, 38 mmol) in THF (9 mL) at 0 °C was slowly added *n*-BuLi (1.63 M in hexane, 23 mL, 37 mmol), and the mixture was stirred at 0 °C for 1 h. To the solution was added diphenylacetic acid (3.18 g, 15.0 mmol) in THF (18 mL), and the mixture was stirred at 45 °C for 1.5 h. Allyl bromide (2.6 mL, 30 mmol) was added to the solution and the mixture was stirred at 45 °C overnight. H_2O was added to the mixture and the aqueous layer was washed with Et_2O. The aqueous layer was acidified with aq. HCl, and it was extracted with Et_2O three times. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The solid residue was recrystallized from $CHCl_3$ and hexane to give **1a** as a white solid (3.66 g, 14.5 mmol, 97% yield). ^1H NMR (600 MHz, $CDCl_3$) δ 3.16 (d, *J* = 7.5 Hz, 2H), 4.91–4.96 (m, 2H), 5.55–5.62 (m, 1H), 7.24–7.31 (m, 10H).



1a-d_1

Compound 1a-d_1. **1a** (126 mg, 0.50 mmol) was placed in a Schlenk tube under nitrogen. Acetone-d_6 (1.5 mL) and D_2O (1.5 mL) was added and the Schlenk tube was capped with a glass stopper and stirred at room temperature for 48 h. The mixture was concentrated and dried under vacuum at 50 °C for 1.5 h then freeze-dried to give **1a-d_1**, which was used immediately for the reaction. ^1H NMR (600 MHz, DMSO-d_6) δ 3.09 (d, *J* = 7.9 Hz, 2H), 4.87–4.92 (m, 2H), 5.50–5.55 (m, 1H), 7.20–7.31 (m, 5H).

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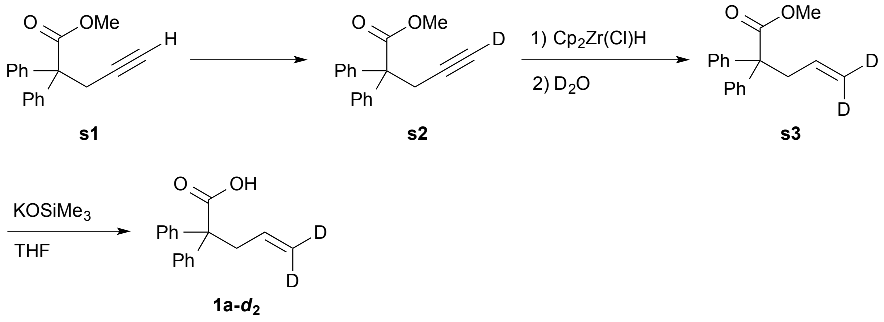
[3] N. T. Barczak and E. R. Jarvo, *Chem. Eur. J.,* 2011, **17**, 12912.

[4] I. F. Cottrel, A. R. Cowley, L. J. Croft, L. Hymns, M. G. Moloney, E. J. Netteleton, H. K. Smithies and A. L. Thompson, *Tetrahedron,* 2009, **65**, 2537.

[5] M. Wilking, C. G. Daniliuc, U. Hennecke, U. *Synlett,* 2014, **25**, 1701.



Compound 1a-*d*$_2$. Compound **s1** (CAS: 906542-82-3, 529 mg, 2.00 mmol) and K_2CO_3 (415 mg, 3.0 mmol) in MeCN (4.0 mL) was stirred at room temperature for 15 min. D_2O (1.0 mL) was added and the mixture was stirred at room temperature for 2 h.[6] The mixture was extracted with dry Et_2O and the organic layer was dried by passing through a short column of $MgSO_4$ and concentrated to give **s2** as a colorless oil (501 mg, 1.89 mmol, 94% yield, 96% D). ^1H NMR (600 MHz, $CDCl_3$) δ 3.29 (s, 2H), 3.74 (s, 3H), 7.27–7.34 (m, 10H). HRMS (ESI) calcd for $C_{18}H_{15}D_1NaO_2$ $(M+Na)^+$ 288.1105, found 288.1109.

To a solution of $Cp_2Zr(Cl)H$ (193 mg, 0.75 mmol) in CH_2Cl_2 (1.5 mL) at 0 °C was slowly added a solution of **s2** (133 mg, 0.50 mmol) in CH_2Cl_2 (1.5 mL), and the mixture was stirred at 0 °C for 80 min. To the solution at 0 °C was added D_2O (0.70 mL), and the mixture was passed through a pad of silica gel with CH_2Cl_2 and concentrated on a rotary evaporator. The residue was subjected to column chromatography on silica gel with EtOAc/hexane (1/10) to give **s3** a colorless oil (120 mg, 0.44 mmol, 88% yield, 93%D of the terminal methylene protons). ^1H NMR (600 MHz, $CDCl_3$) δ 3.17 (d, J = 6.8 Hz, 2H), 3.70 (s, 3H), 5.62–5.56 (m, 1H), 7.22–7.30 (m, 10H). HRMS (ESI) calcd for $C_{18}H_{16}D_2NaO_2$ $(M+Na)^+$ 291.1325, found 291.1327.

$KOSiMe_3$ (283 mg, 2.2 mmol) and **s3** (120 mg, 0.44 mmol) were placed in a Schlenk tube under nitrogen. THF (3.5 mL) was added and the Schlenk tube was capped with a glass stopper and heated at 80 °C for 20 h with stirring. 1 N aq. NaOH was added, and the mixture was washed with hexane. The aqueous layer was acidified with aq. HCl and extracted with Et_2O. The organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The residue was passed through a pad of silica gel with EtOAc and recrystallized from $CHCl_3$ and hexane to give **1a-*d*$_2$** as a white solid (87.1 mg, 0.34 mmol, 78% yield, 93%D of the terminal methylene proton). ^1H NMR (600 MHz, $CDCl_3$) δ 3.17 (d, J = 7.5 Hz, 2H), 5.56–5.62 (m, 1H), 7.25–7.33 (m, 10H). ^2H NMR (91 MHz, $CHCl_3$) δ 4.96. HRMS (ESI) calcd for $C_{17}H_{14}D_2NaO_2$ $(M+Na)^+$ 277.1168, found 277.1174.

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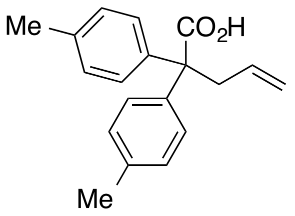
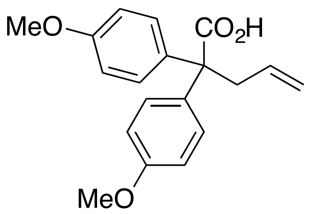
[6] S. P. Bew, G. D. Hiatt-Gipson, J. A. Lovell and C. Poullain, *Org. Lett.,* 2012, **14**, 456.



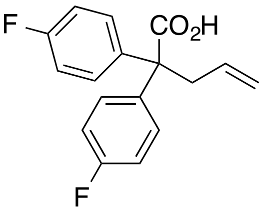
1b

Compound 1b (white solid). ^{1}H NMR (600 MHz, $CDCl_3$) δ 2.35 (s, 6H), 3.14 (d, *J* = 6.8 Hz, 2H), 4.90–5.00 (m, 2H), 5.56–5.65 (m, 1H), 7.12 (d, *J* = 8.1 Hz, 4H), 7.19 (d, *J* = 8.1 Hz, 4H).



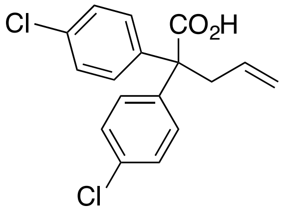
1c

Compound 1c (white solid). This compound was prepared by the reaction of di(4-methoxylphenyl)acetic acid according to the procedures for **1a** (88% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 3.12 (d, *J* = 6.8 Hz, 2H), 3.81 (s, 6H), 4.93–4.97 (m, 2H), 5.55–5.63 (m, 1H), 6.84 (d, *J* = 8.9 Hz, 4H), 7.22 (d, *J* = 8.9 Hz, 4H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 42.6, 55.2, 58.8, 113.2, 118.3, 130.1, 134.1, 134.2, 158.4, 180.3. HRMS (ESI) calcd for $C_{19}H_{20}NaO_4$ $(M+Na)^+$ 355.1254, found 355.1256.



1d

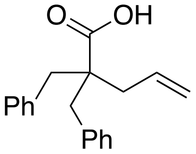
Compound 1d (white solid). ^{1}H NMR (600 MHz, $CDCl_3$) δ 3.10 (d, *J* = 6.8 Hz, 2H), 4.92 (d. *J* = 17.0 Hz, 1H), 4.95 (d, *J* = 10.2 Hz, 1H), 5.50–5.59 (m, 1H), 6.99 (dd, *J* = 8.5, 8.5 Hz, 4H), 7.24 (dd, *J* = 7.8, 5.1 Hz, 4H).



1e

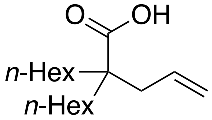
Compound 1e (white solid). This compound was prepared by the reaction of di(4-chlorolphenyl)acetic acid according to the procedures for **1a** (83% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 3.10 (d, *J* = 6.8 Hz, 2H), 4.93 (d, *J* = 17.0 Hz, 1H), 4.97 (d, *J* = 9.5 Hz, 1H), 5.49–

5.57 (m, 1H), 7.20 (d, *J* = 8.5 Hz, 4H), 7.29 (d, *J* = 8.5 Hz, 4H); ^{13}C NMR (150 MHz, CDCl$_3$) δ 42.3, 59.4, 119.4, 128.2, 130.4, 132.9, 133.4, 140.0, 178.9. HRMS (ESI) calcd for $C_{17}H_{14}Cl_2NaO_2$ (M+Na)$^+$ 343.0263, found 343.0267.



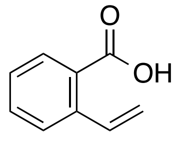
1f

Compound 1f (white solid). This compound was prepared by the reaction of dibenzylacetic acid according to the procedures for **1a** (69% yield). ^{1}H NMR (600 MHz, CDCl$_3$) δ 2.31 (d, *J* = 6.8 Hz, 2H), 2.92 (d, *J* = 14.0 Hz, 2H), 3.12 (d, *J* = 14.0 Hz, 2H), 5.18 (d, *J* = 17.7 Hz, 1H), 5.26 (d, *J* = 10.2 Hz, 1H), 5.96–6.04 (m, 1H), 7.20–7.29 (m, 10H); ^{13}C NMR (150 MHz, CDCl$_3$) δ 35.5, 41.8, 51.8, 119.3, 126.7, 128.2, 130.2, 133.8, 137.0, 181.9. HRMS (ESI) calcd for $C_{19}H_{20}NaO_2$ (M+Na)$^+$ 303.1356, found 303.1364.



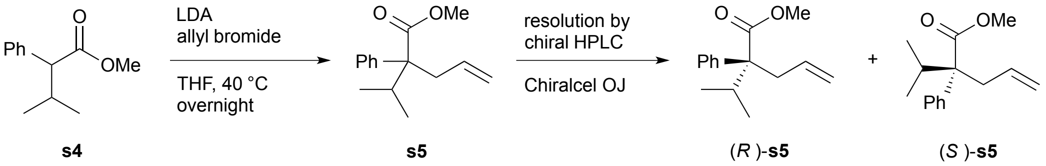
1g

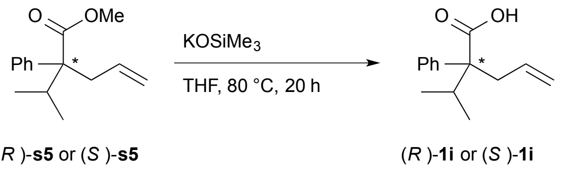
Compound 1g (white solid). This compound was prepared by the reaction of 2-hexyloctanoic acid according to the procedures for **1a** (93% yield). ^{1}H NMR (600 MHz, CDCl$_3$) δ 0.88 (t, *J* = 6.8 Hz, 6H), 1.16–1.33 (m, 18H), 1.49–1.58 (m, 2H), 2.35 (d, *J* = 7.5 Hz, 2H), 5.05–5.11 (m, 2H), 5.67–5.75 (m, 1H); ^{13}C NMR (150 MHz, CDCl$_3$) δ 14.0, 22.6, 23.8, 29.7, 31.7, 34.8, 38.0, 49.0, 117.9, 133.8, 183.7. HRMS (ESI) calcd for $C_{17}H_{32}NaO_2$ (M+Na)$^+$ 291.2295, found 291.2290.



1h

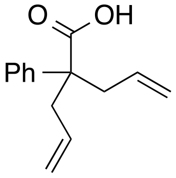
Compound 1h (white solid). ^{1}H NMR (600 MHz, CDCl$_3$) δ 5.39 (d, *J* = 10.2 Hz, 1H), 5.68 (d, *J* = 17.6 Hz, 1H), 7.37 (t, *J* = 7.5 Hz, 1H), 7.53–7.63 (m, 3H), 8.04 (d, *J* = 7.5 Hz, 1H).





Compound 1i (white solid). To a solution of diisopropylamine (1.6 mL, 11 mmol) in THF (7 mL) at 0 °C was slowly added *n*-BuLi (1.60 M in hexane, 6.4 mL, 10 mmol), and the mixture was stirred at 0 °C for 1 h. To the solution was added **s4** (CAS: 72615-27-1, 2.1 g, 8.5 mmol) in THF (5 mL), and the mixture was stirred at 40 °C for 1.5 h. Allyl bromide (0.96 mL, 11 mmol) was added to the solution and the mixture was stirred at 40 °C overnight. The mixture was quenched with aq. HCl and extracted with Et_2O three times. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The residue was subjected to column chromatography on silica gel with EtOAc/hexane (1/25) to give racemic **s5** (1.64 g, 7.1 mmol, 83% yield) as a colorless oil. Optical resolution was carried out by use of a chiral stationary phase column (Chiralcel OJ, hexane/2-propanol = 90:10, t_1 = 20 min for (*R*)-**s5**, t_2 = 38 min for (*S*)-**s5**) to give both enantiomers (*R*)-**s5** and (*S*)-**s5**. (*R*)-**s5**: ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.84 (d, *J* = 6.8 Hz, 3H), 0.88 (d, *J* = 6.8 Hz, 3H), 2.48 (sept, *J* = 6.8 Hz, 1H), 2.79 (dd, *J* = 14.3, 7.5 Hz, 1H), 2.86 (dd, *J* = 14.3, 6.8 Hz, 1H), 3.70 (s, 3H), 4.99 (d, *J* = 10.2 Hz, 1H), 5.03 (d, *J* = 17.0 Hz, 1H), 5.60–5.68 (m, 1H), 7.17 (d, *J* = 7.6 Hz, 2H), 7.24 (t, *J* = 7.6 Hz, 1H), 7.31 (t, *J* = 7.6 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 18.0, 19.0, 32.5, 40.4, 51.4, 59.3, 117.6, 126.4, 127.5, 128.4, 134.3, 139.6, 175.3. HRMS (ESI) calcd for $C_{15}H_{20}NaO_2$ $(M+Na)^+$ 255.1356, found 255.1362. $[\alpha]^{20}_D$ +8 (*c* 0.95, $CHCl_3$). (*S*)-**s5**: $[\alpha]^{20}_D$ –7 (*c* 0.86, $CHCl_3$).

To a solution of (*R*)-**s5** (41.3 mg, 0.18 mmol) in THF (1.0 mL) was added $KOSiMe_3$ (116 mg, 0.90 mmol), and the mixture was heated at 100 °C for 20 h with stirring. 1 N aq. NaOH was added, and the mixture was washed with hexane. The aqueous layer was acidified with aq. HCl and extracted with Et_2O. The organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The residue was subjected to preparative TLC on silica gel with EtOAc/hexane (1:3) to give (*R*)-**1i** as a white solid (22.2 mg, 0.10 mmol, 56% yield). (*R*)-**1i**: ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.87 (d, *J* = 6.8 Hz, 3H), 0.94 (d, *J* = 6.8 Hz, 3H), 2.50 (sept, *J* = 6.8 Hz, 1H), 2.81–2.90 (m, 2H), 5.01 (d, *J* = 9.5 Hz, 1H), 5.07 (d, *J* = 17.0 Hz, 1H), 5.64–5.73 (m, 1H), 7.23–7.29 (m, 3H), 7.32 (t, *J* = 7.8 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 18.0, 18.9, 32.8, 40.2, 59.3, 117.9, 126.7, 127.6, 128.6, 134.1, 138.8, 180.9. HRMS (ESI) calcd for $C_{14}H_{18}NaO_2$ $(M+Na)^+$ 241.1199, found 241.1194. $[\alpha]^{20}_D$ = +10 (*c* 1.11, $CHCl_3$). (*S*)-**1i**: $[\alpha]^{20}_D$ –10 (*c* 1.33, $CHCl_3$).



1j

Compound 1j. ^{1}H NMR ($CDCl_3$) δ 2.77 (dd, J = 14.1, 6.8 Hz, 2H), 2.83 (dd, J = 14.1, 7.8 Hz, 2H), 5.05–5.11 (m, 4H), 5.51–5.59 (m, 2H), 7.28 (t, J = 7.5 Hz, 1H), 7.31 (t, J = 7.5 Hz, 2H), 7.35 (d, J = 7.5 Hz, 2H).

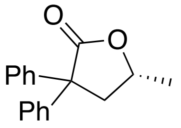
5. General procedure for Ir-catalyzed asymmetric cyclization (Table 1)

(*R*)-DTBM-segphos (5.9 mg, 0.0050 mmol, 5 mol%) and $[IrCl(coe)_2]_2$ (2.2 mg, 0.0050 mmol of Ir, 5 mol% of Ir) were placed in a Schlenk tube under nitrogen. NMP (0.4 mL) was added and the mixture was stirred at room temperature for 10 min. 2,2-Diphenyl-4-pentenoic acid (**1a**: 25.2 mg, 0.10 mmol) was added and the Schlenk tube was capped with a glass stopper and heated at 80 °C for 20 h with stirring. The mixture was diluted with Et_2O, washed with H_2O and brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The yields of the products were determined by ^{1}H NMR using 1,4-dimethoxybenzene as an internal standard. The residue was subjected to preparative TLC on silica gel with EtOAc/hexane (1:5) to give **2a**.

6. General procedure for Ir-catalyzed asymmetric cyclization (Table 2)

(*R*)-DTBM-segphos (11.8 mg, 0.010 mmol, 5 mol%) and $[IrCl(coe)_2]_2$ (4.5 mg, 0.010 mmol of Ir, 5 mol% of Ir) were placed in a Schlenk tube under nitrogen. NMP (0.8 mL) was added and the mixture was stirred at room temperature for 10 min. Carboxylic acid **1** (0.20 mmol) was added and the Schlenk tube was capped with a glass stopper and heated at 100 °C for 20 h with stirring. The mixture was diluted with Et_2O, washed with H_2O and brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The residue was subjected to preparative TLC on silica gel and GPC with $CHCl_3$ to give **2**.

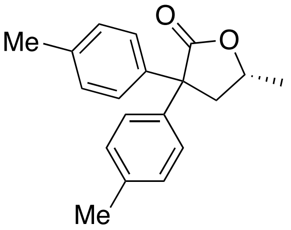
7. Characterization of the products



2a

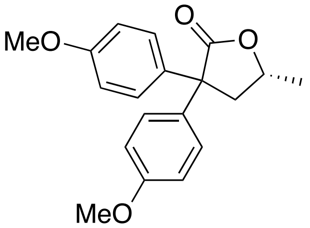
Compound 2a (colorless oil, 46.5 mg, 92% yield, 81% ee, CAS: 1729-20-0 for racemic **2a**). A solution of EtOAc/hexane (1:5) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OD-H column × 2, hexane/2-propanol = 100:1, flow 0.5 mL/min, 254 nm, t_1 = 27.2 min (*R*), t_2 = 28.6 min (*S*); $[\alpha]^{20}_{D}$ −102 (*c* 0.99, $CHCl_3$) for 81% ee (*R*). ^{1}H NMR

(600 MHz, $CDCl_3$) δ 1.46 (d, *J* = 6.1 Hz, 3H), 2.59 (dd, *J* = 12.9, 10.2 Hz, 1H), 3.06 (dd, *J* = 12.9, 4.8 Hz, 1H), 4.45–4.51 (m, 1H), 7.22–7.38 (m, 10H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 20.4, 45.4, 58.6, 73.5, 127.2, 127.3, 127.6, 127.7, 128.3, 128.9, 139.9, 142.1, 177.1. HRMS (ESI) calcd for $C_{17}H_{16}NaO_2$ $(M+Na)^+$ 275.1043, found 275.1048.



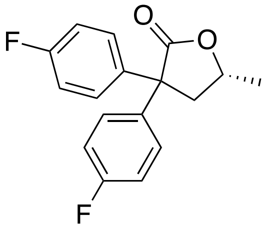
2b

Compound 2b (colorless oil, 55.1 mg, 98% yield, 85% ee). A solution of EtOAc/hexane (1:4) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak AD-H column, hexane/2-propanol = 90:10, flow 0.5 mL/min, 254 nm, t_1 = 17.3 min (*R*), t_2 = 22.2 min (*S*); $[\alpha]^{20}_D$ –95 (*c* 1.02, $CHCl_3$) for 85% ee (*R*). ^1H NMR (600 MHz, $CDCl_3$) δ 1.46 (d, *J* = 6.1 Hz, 3H), 2.31 (s, 3H), 2.34 (s, 3H), 2.55 (dd, *J* = 12.9, 10.2 Hz, 1H), 3.02 (dd, *J* = 12.9, 4.8 Hz, 1H), 4.44–4.50 (m, 1H), 7.11 (d, *J* = 8.1 Hz, 2H), 7.16 (d, *J* = 8.1 Hz, 2H), 7.19 (d, *J* = 8.1 Hz, 2H), 7.25 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 20.3, 20.9, 45.4, 58.1, 73.4, 127.2, 127.5, 129.0, 129.4, 136.7, 137.0, 137.3, 139.3, 177.4. HRMS (ESI) calcd for $C_{19}H_{20}NaO_2$ $(M+Na)^+$ 303.1356, found 303.1348.



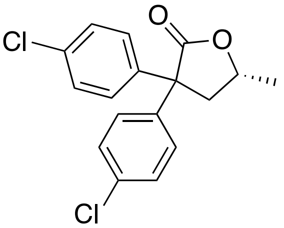
2c

Compound 2c (colorless oil, 59.8 mg, 96% yield, 83% ee). A solution of EtOAc/hexane (1:4) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OD-H column, hexane/2-propanol = 90:10, flow 0.5 mL/min, 254 nm, t_1 = 22.1 min (*S*), t_2 = 25.3 min (*R*); $[\alpha]^{20}_D$ –83 (*c* 1.05, $CHCl_3$) for 83% ee (*R*). ^1H NMR (600 MHz, $CDCl_3$) δ 1.44 (d, *J* = 6.1 Hz, 3H), 2.51 (dd, *J* = 12.9, 10.2 Hz, 1H), 2.99 (dd, *J* = 12.9, 4.8 Hz, 1H), 3.76 (s, 3H), 3.78 (s, 3H), 4.42–4.48 (m, 1H), 6.82 (d, *J* = 8.9 Hz, 2H), 6.86 (d, *J* = 8.9 Hz, 2H), 7.19 (d, *J* = 8.9 Hz, 2H), 7.26 (d, *J* = 8.9 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 20.3, 45.6, 55.2, 55.3, 57.4, 73.4, 113.7, 114.2, 128.4, 128.7, 132.0, 134.6, 158.5, 158.9, 177.7. HRMS (ESI) calcd for $C_{19}H_{20}NaO_4$ $(M+Na)^+$ 335.1254, found 335.1246.



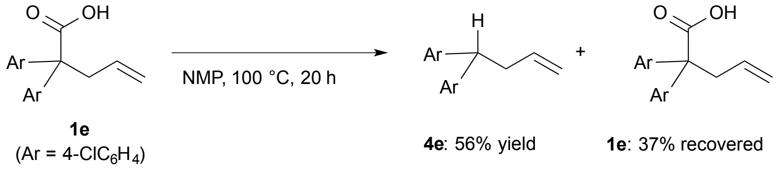
2d

Compound 2d (colorless oil, 49.7 mg, 86% yield, 70% ee). A solution of EtOAc/hexane (1:4) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OJ-H column, hexane/2-propanol = 90:10, flow 0.5 mL/min, 254 nm, t_1 = 33.6 min (*S*), t_2 = 47.2 min (*R*); $[\alpha]^{20}_{D}$ –74 (*c* 0.78, $CHCl_3$) for 70% ee (*R*). ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.46 (d, *J* = 6.1 Hz, 3H), 2.53 (dd, *J* = 12.9, 10.2 Hz, 1H), 3.01 (dd, *J* = 12.9, 4.8 Hz, 1H), 4.43–4.49 (m, 1H), 6.99 (t, *J* = 8.7 Hz, 2H), 7.04 (t, *J* = 8.7 Hz, 2H), 7.24–7.28 (m, 2H), 7.29–7.33 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 20.3, 45.4, 57.5, 73.5, 115.5 (d, $J_{F\text{–}C}$ = 90 Hz), 115.7 (d, $J_{F\text{–}C}$ = 90 Hz), 129.1 (d, $J_{F\text{–}C}$ = 45 Hz), 129.2 (d, $J_{F\text{–}C}$ = 45 Hz), 135.5, 137.7, 161.9 (d, $J_{F\text{–}C}$ = 247 Hz), 162.1 (d, $J_{F\text{–}C}$ = 247 Hz), 176.8. HRMS (ESI) calcd for $C_{17}H_{14}F_2NaO_2$ $(M+Na)^+$ 311.0854, found 311.0862.

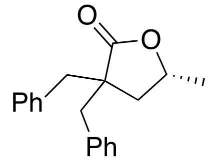


2e

Compound 2e (colorless oil, 34.0 mg, 53% yield, 61% ee). A solution of EtOAc/hexane (1:4) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak AD-H column, hexane/2-propanol = 90:10, flow 0.5 mL/min, 254 nm, t_1 = 17.7 min (*R*), t_2 = 18.8 min (*S*); $[\alpha]^{20}_{D}$ –64 (*c* 0.87, $CHCl_3$) for 61% ee (*R*). ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.46 (d, *J* = 6.1 Hz, 3H), 2.53 (dd, *J* = 12.9, 10.9 Hz, 1H), 2.99 (dd, *J* = 12.9, 4.8 Hz, 1H), 4.43–4.49 (m, 1H), 7.22–7.29 (m, 6H), 7.32 (d, *J* = 8.8 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 20.3, 45.1, 57.7, 73.6, 128.7, 129.0, 129.2, 133.5, 134.0, 138.1, 140.1, 176.4. HRMS (ESI) calcd for $C_{17}H_{14}Cl_2NaO_2$ $(M+Na)^+$ 343.0263, found 343.0260.

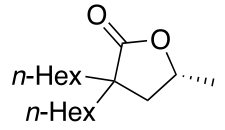


Decarboxylation of 1e in the absence of the Ir catalyst: **1e** (32.1 mg, 0.10 mmol) was placed in a Schlenk tube under nitrogen. NMP (0.4 mL) was added, then the Schlenk tube was capped with a glass stopper and heated at 100 °C for 20 h with stirring. The mixture was diluted with Et_2O, washed with H_2O and brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The yields of **4e** and recovered **1e** were estimated by ^{1}H NMR using 1,4-dimethoxybenzene as an internal standard. The ^{1}H NMR spectrum of **4e** was identical to that previously reported.[7]



2f

Compound 2f (white solid, 24.5 mg obtained in the reaction of 0.10 mmol of **1f**, 87% yield, 86% ee). A solution of EtOAc/hexane (1:4) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel OD-H column, hexane/2-propanol = 90:10, flow 0.5 mL/min, 254 nm, t_1 = 14.8 min (*S*), t_2 = 15.8 min (*R*); $[\alpha]^{20}_D$ +27 (*c* 0.97, $CHCl_3$) for 86% ee (*R*). ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.84 (d, *J* = 6.8 Hz, 3H), 1.77 (dd, *J* = 13.2, 9.2 Hz, 1H), 2.17 (dd, *J* = 13.2, 7.1 Hz, 1H), 2.71 (d, *J* = 13.2 Hz, 1H), 2.77 (d, *J* = 13.2 Hz, 1H), 3.16 (d, *J* = 13.5 Hz, 1H), 3.27 (d, *J* = 13.5 Hz, 1H), 3.35–3.41 (m, 1H), 7.17–7.31 (m, 10H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 21.3, 36.8, 43.8, 44.5, 52.2, 73.7, 126.9, 127.2, 128.4, 128.6, 129.9, 130.4, 136.4, 136.9, 180.8. HRMS (ESI) calcd for $C_{19}H_{20}NaO_2$ $(M+Na)^+$ 303.1356, found 303.1355.



2g

Compound 2g (colorless oil, 39.8 mg, 74% yield, 89% ee). The crude product was subjected to column chromatography on silica gel with EtOAc/hexane (1:20). The ee of **2g** was determined by chiral HPLC analysis of compound **5** derived from **2g** (vide infra). $[\alpha]^{20}_D$ +5 (*c* 0.85, $CHCl_3$) for 89% ee (*R*). ^{1}H NMR (600 MHz, $CDCl_3$) δ 0.85–0.90 (m, 6H), 1.12–1.20 (m, 1H), 1.23–1.37 (m, 15H), 1.38 (d, *J* = 6.1 Hz, 3H), 1.49–1.61 (m, 4H), 1.75 (dd, *J* = 13.0, 9.3 Hz, 1H), 2.15 (dd, *J* = 13.0, 6.6 Hz, 1H), 4.49–4.55 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 14.0, 21.7, 22.5, 22.6, 24.28, 24.32, 29.61, 29.63, 31.59, 31.64, 36.3, 36.9, 40.3, 48.4, 73.5, 181.1. HRMS (ESI) calcd for $C_{17}H_{32}NaO_2$ $(M+Na)^+$ 291.2295, found 291.2289.

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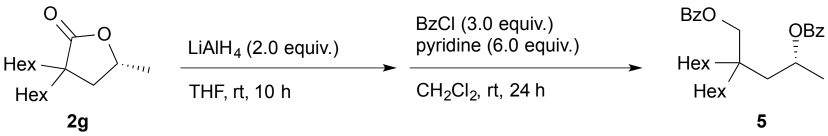
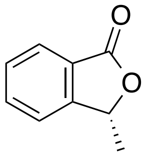
[7] S. J. Mahoney, T. Lou, G. Bondarenko and E. Fillion, *Org. Lett.*, 2012, **14**, 3474.



Transformation of 2g into 5. To a suspension of LiAlH$_4$ (10.6 mg, 0.28 mmol) in THF (0.2 mL) was added a solution of **2g** (39.7 mg, 0.15 mmol) in THF (0.5 mL) at 0 °C, and the mixture was stirred at room temperature for 10 h. H$_2$O (20 μL), 10% aq. NaOH (20 μL), and H$_2$O (65 μL) were added successively at 0 °C, and the mixture was passed through a pad of Celite with Et$_2$O as an eluent and concentrated on a rotary evaporator. To the solution of the crude product and pyridine (62 μL, 0.77 mmol) in CH$_2$Cl$_2$ (0.6 mL) was added benzoyl chloride (45 μL, 0.40 mmol) at 0 °C, and the mixture was stirred at room temperature for 24 h. H$_2$O was added to the mixture and it was extracted with EtOAc. The organic layer was washed with brine, dried over Na$_2$SO$_4$, filtered, and concentrated on a rotary evaporator. The residue was subjected to preparative TLC on silica gel with EtOAc/hexane (1:10) to give **5** as a white solid (52.8 mg, 0.11 mmol, 86% yield). The ee was measured by HPLC (Chiralpak AD-H column, hexane/2-propanol = 100:1, flow 0.5 mL/min, 254 nm, t_1 = 13.1 min (*R*), t_2 = 15.3 min (*S*); $[\alpha]^{20}_{\mathrm{D}}$ –29 (*c* 0.93, CHCl$_3$) for 89% ee (*R*). ^{1}H NMR (600 MHz, CDCl$_3$) δ 0.79–0.85 (m, 6H), 1.13–1.25 (m, 14H), 1.26–1.41 (m, 9H), 1.60 (d, *J* = 15.2 Hz, 1H), 2.07 (dd, *J* = 15.2, 9.2 Hz, 1H), 4.10 (d, *J* = 11.3 Hz, 1H), 4.13 (d, *J* = 11.3 Hz, 1H), 5.37–5.42 (m, 1H), 7.34 (t, *J* = 7.5 Hz, 2H), 7.45 (t, *J* = 7.5 Hz, 2H), 7.50 (t, *J* = 7.5 Hz, 1H), 7.56 (t, *J* = 7.5 Hz, 1H), 7.97 (d, *J* = 7.5 Hz, 2H), 8.01 (d, *J* = 7.5 Hz, 2H); ^{13}C NMR (150 MHz, CDCl$_3$) δ 14.0, 22.4, 22.6, 23.0, 30.1, 31.77, 31.79, 34.7, 34.9, 38.6, 40.7, 68.4, 68.9, 128.2, 128.4, 129.48, 129.49, 130.4, 130.6, 132.7, 132.8, 166.0, 166.3. HRMS (ESI) calcd for $C_{31}H_{44}NaO_4$ $(M+Na)^+$ 503.3132, found 503.3141.



2h

Compound 2h (colorless oil, 16.8 mg, 57% yield, 49% ee, CAS: 3413-14-7 for (*R*)-**2h**). A solution of EtOAc/hexane (1:3) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralcel AS-H column, hexane/2-propanol = 90:10, flow 0.5 mL/min, 254 nm, t_1 = 35.8 min (*R*), t_2 = 37.9 min (*S*); $[\alpha]^{20}_{\mathrm{D}}$ +23 (*c* 0.83, CHCl$_3$) for 49% ee (*R*). The absolute configuration was determined by comparison of the optical rotation with the reported value.[8] ^{1}H NMR (600 MHz, CDCl$_3$) δ 1.64 (d, *J* = 6.4 Hz, 3H), 5.57 (q, *J* = 6.4 Hz, 1H), 7.44 (d, *J* = 8.0 Hz, 1H), 7.53 (t, *J* = 8.0 Hz, 1H), 7.68 (t, *J* = 8.0 Hz, 1H), 7.90 (d, *J* = 8.0 Hz, 1H).

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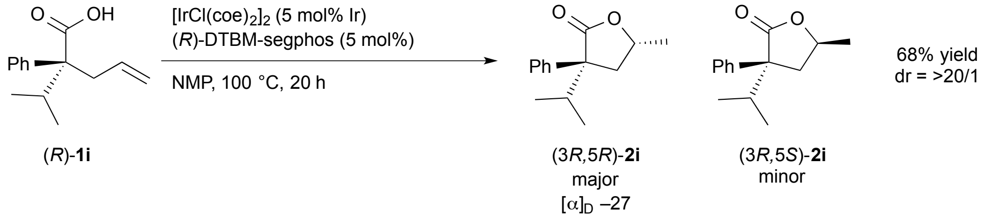
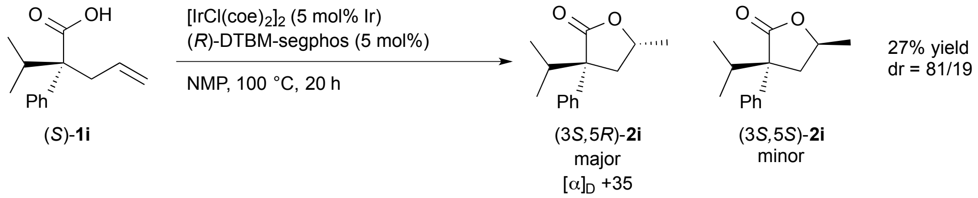
[8] J. Yang and N. Yoshikai, *J. Am. Chem. Soc.*, 2014, **136**, 16748.





Compound 2i (eqns (1) and (2)). (*R*)-DTBM-segphos (5.7 mg, 0.0050 mmol, 5 mol%) and $[IrCl(coe)_2]_2$ (2.2 mg, 0.0050 mmol of Ir, 5 mol% of Ir) were placed in a Schlenk tube under nitrogen. NMP (0.4 mL) was added and the mixture was stirred at room temperature for 10 min. (*R*)-**1i** (22.0 mg, 0.10 mmol) was added and the Schlenk tube was capped with a glass stopper and heated at 100 °C for 20 h with stirring. The mixture was diluted with Et_2O, washed with H_2O and brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The residue was subjected to preparative TLC on silica gel with EtOAc/hexane (5:1) and GPC with $CHCl_3$ to give (3*R*,5*R*)-**2i** as a colorless oil (14.9 mg, 68% yield, dr = >20:1). $[\alpha]^{20}_{D} = -27$ (*c* 0.74, $CHCl_3$). The reaction of (*S*)-**1i** gave **2i** as a mixture of the diastereomers (colorless oil, 7.3 mg, 27% yield, dr = 81:19). The isolated mixture of (3*S*,5*R*)-**2i** and (3*S*,5*S*)-**2i** was subjected to preparative TLC on silica gel with EtOAc/hexane (5:1) to give the major diastereomer (3*S*,5*R*)-**2i** (dr = 96:4).

Compound (3*R*,5*R*)-2i. $[\alpha]^{20}_{D}$ −27 (*c* 0.74, $CHCl_3$). The absolute configuration was determined by comparison of the optical rotation with the reported value.[9] ^1H NMR (500 MHz, $CDCl_3$) δ 0.72 (d, *J* = 6.5 Hz, 3H), 1.02 (d, *J* = 6.5 Hz, 3H), 1.43 (d, *J* = 6.0 Hz, 3H), 2.02 (dd, *J* = 13.5, 11.0 Hz, 1H), 2.36 (sept, *J* = 6.5 Hz, 1H), 2.71 (dd, *J* = 13.5, 5.0 Hz, 1H), 4.32–4.39 (m, 1H), 7.29 (t, *J* = 7.6 Hz, 1H), 7.36 (t, *J* = 7.6 Hz, 2H), 7.50 (d, *J* = 7.6 Hz, 2H).

Compound (3*S*,5*R*)-2i. $[\alpha]^{20}_{D}$ +35 (*c* 0.30, $CHCl_3$) for 92% de. The absolute configuration was determined by comparison of the optical rotation with the reported value.[9] ^1H NMR (500 MHz, $CDCl_3$) δ 0.84 (d, *J* = 7.0 Hz, 3H), 0.95 (d, *J* = 7.0 Hz, 3H), 1.29 (d, *J* = 6.0 Hz, 3H), 2.25 (sept, *J* = 7.0 Hz, 1H), 2.36 (dd, *J* = 13.5, 6.5 Hz, 1H), 2.73 (dd, *J* = 13.5, 7.5 Hz, 1H), 4.61–4.68 (m, 1H), 7.26 (t, *J* = 7.8 Hz, 1H), 7.34 (t, *J* = 7.8 Hz, 2H), 7.59 (d, *J* = 7.8 Hz, 2H).

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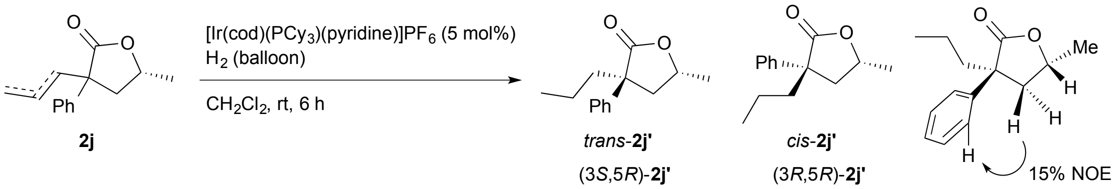
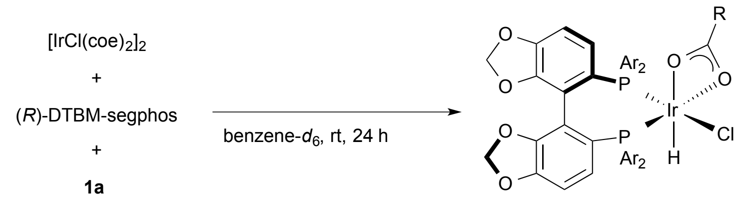
[9] F. Antonietti, E. Brenna, C. Fuganti, F. G. Gatti, T. Giovenzana, V. Grande and L. Malpezzi, *Synthesis,* 2005, 1148.



Compound 2j'. A solution of the crude mixture of **2j** and [Ir(cod)(PCy$_3$)(pyridine)]PF$_6$ (8.0 mg, 5 mol%) in CH_2Cl_2 (0.4 mL) was vigorously stirred under H_2 (balloon) for 6 h. The mixture was passed through a pad of Celite with Et_2O as an eluent, and the solvent was removed on a rotary evaporator. The residue was subjected to preparative TLC on silica gel with EtOAc/hexane (1:4) as an eluent and GPC with $CHCl_3$ to give **2j'** as a mixture of the diastereomers (38.7 mg, 0.177 mmol, 89% yield). The ee was measured by HPLC (Chiralpak ID column × 2, hexane/2-propanol = 98:2, flow 0.45 mL/min, 254 nm, t_1 = 47.7 min (3*R*,5*S*), t_2 = 50.4 min (3*S*,5*R*), t_3 = 53.5 min (3*R*,5*R*), t_4 = 56.8 min (3*S*,5*S*). *trans*-**2j'** (major isomer): ^{1}H NMR (CDCl$_3$) δ 0.87 (t, *J* = 7.5 Hz, 3H), 1.10–1.32 (m, 2H), 1.42 (d, *J* = 6.1 Hz, 3H), 1.79 (ddd, *J* = 14.0, 12.2, 4.5 Hz, 1H), 1.95–2.00 (m, 1H), 1.98 (dd, *J* = 13.0, 10.5 Hz, 1H), 2.80 (dd, *J* = 13.0, 4.8 Hz, 1H), 4.32–4.38 (m, 1H), 7.27 (t, *J* = 7.4 Hz, 1H), 7.36 (t, *J* = 7.4 Hz, 2H), 7.42 (d, *J* = 7.4 Hz, 2H). *cis*-**2j'** (minor isomer): ^{1}H NMR (CDCl$_3$) δ 0.85 (t, *J* = 7.5 Hz, 3H), 1.10–1.32 (m, 2H), 1.35 (d, *J* = 6.1 Hz, 3H), 1.84–1.94 (m, 2H), 2.29 (dd, *J* = 13.0, 8.5 Hz, 1H), 2.64 (dd, *J* = 13.0, 6.8 Hz, 1H), 4.65–4.71 (m, 1H), 7.26 (t, *J* = 7.4 Hz, 1H), 7.35 (t, *J* = 7.4 Hz, 2H), 7.52 (d, *J* = 7.4 Hz, 2H). HRMS (ESI) calcd for $C_{14}H_{18}NaO_2$ (M+Na)$^+$ 241.1199, found 241.1202.

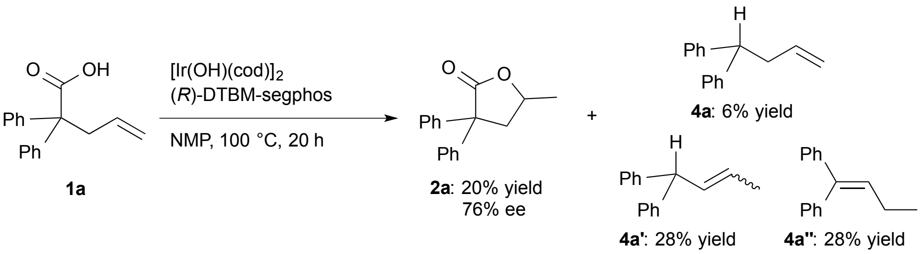
8. Stoichiometric reaction of 1a with [IrCl(coe)$_2$]$_2$ and (*R*)-DTBM-segphos



Ar = 3,5-(*t*-Bu)$_2$-4-MeOC$_6$H$_2$

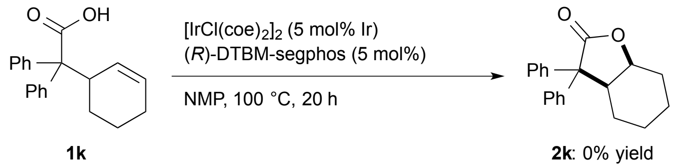
Compound **1a** (2.5 mg, 0.010 mmol) and hexamethybenzene (1.6 mg, 0.010 mmol as an internal standard) were placed in an NMR tube under nitrogen. (*R*)-DTBM-segphos (11.8 mg, 0.010 mmol) and [IrCl(coe)$_2$]$_2$ (4.5 mg, 0.0050 mmol, 0.010 mmol of Ir) were placed in a Schlenk tube under nitrogen. C_6D_6 (0.8 mL) was added to the Schlenk tube and the mixture was stirred at room temperature for 20 min. The resulting solution was transferred into the NMR tube by use of a syringe pump, and the tube was capped with a rubber septum. The signal of the hydride was observed at –24.9 ppm (virtual t, $J_{P–H}$ = 22 Hz, a minor isomer) and –27.1 ppm (virtual t, $J_{P–H}$ = 22 Hz, a major isomer). The NMR (600 MHz) yield of the iridium-hydride complexes was estimated to be 13% and 35%, respectively, after 24 h. The stereochemistry of the major isomer shown in the above scheme was tentatively assigned by comparison of the chemical shifts of the hydride peaks with the reported values of [IrHCl(OAc)((*S*)-binap)].[10]

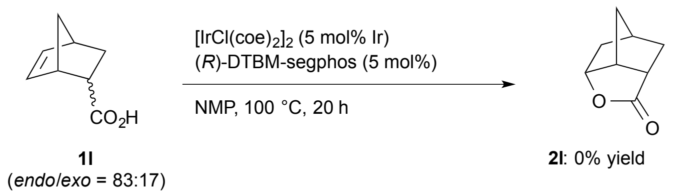
9. The use of $[Ir(OH)(cod)]_2$ as a catalyst precursor



(*R*)-DTBM-segphos (5.9 mg, 0.0050 mmol, 5 mol %) and $[Ir(OH)(cod)]_2$ (1.6 mg, 0.0050 mmol of Ir, 5 mol % of Ir) were placed in a Schlenk tube under nitrogen. NMP (0.4 mL) was added and the mixture was stirred at room temperature for 10 min. Compound **1a** (25.2 mg, 0.10 mmol) was added and the Schlenk tube was capped with a glass stopper and heated at 100 °C for 20 h with stirring. The mixture was diluted with Et_2O, washed with H_2O and brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The yields of decarboxylation products were determined by ^1H NMR using 1,4-dimethoxybenzene as an internal standard. The residue was subjected to preparative TLC on silica gel to give **2a** as a colorless oil (5.1 mg, 0.020 mmol, 20% yield) and **4a** as a mixture of double bond isomers. The ee of **2a** was measured by HPLC (Chiralcel OD-H column × 3, hexane/2-propanol = 90:10, flow 0.5 mL/min, 254 nm, t_1 = 50.5 min (*R*), t_2 = 53.3 min (*S*) (81% ee (*R*)).

10. Intramolecular cyclization of internal alkenes





The reactions of **1k** (CAS: 1311320-00-9) and **1l** (CAS: 934-30-5)[11] were conducted according to the general procedures. No reaction occurred in either case.

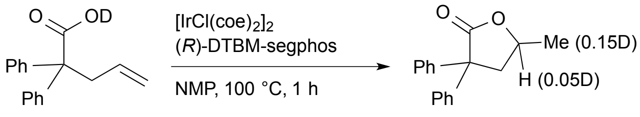
---

[10] T. Yamagata, H. Tadaoka, M. Nagata, T. Hirao, Y. Kataoka, V. Ratovelomanana-Vidal, J. P. Genet and K. Mashima, *Organometallics,* 2006, **25**, 2505.

[11] (*a*) R. E. M. Brooner and R. A. Widenfoefer, *Chem. Eur. J.,* 2011, **17**, 6170; (*b*) H. Kitahara and H. Sakurai, *Molecules,* 2012, **17**, 2579.

11. Deuterium-labeling experiments (eqns (4) and (5))

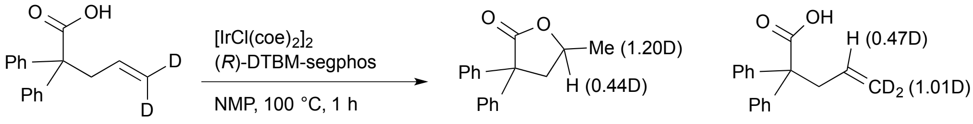
eqn (4)



1a-*d*$_1$ (0.85D) **2a-*d***: 9% yield

(*R*)-DTBM-segphos (11.8 mg, 0.010 mmol, 5 mol%) and $[IrCl(coe)_2]_2$ (4.5 mg, 0.010 mmol of Ir, 5 mol% of Ir) were placed in a Schlenk tube under nitrogen. NMP (0.8 mL) was added and the mixture was stirred at room temperature for 10 min. Compound **1a-*d*$_1$** (0.20 mmol) was added and the Schlenk tube was capped with a glass stopper and heated at 100 °C for 1 h with stirring. The mixture was diluted with Et_2O, washed with H_2O and brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The yields were determined by ^{1}H NMR using 1,4-dimethoxybenzene as an internal standard. The residue was subjected to preparative TLC on silica gel to give **2a** with impurities, which was subjected to GPC with $CHCl_3$ to give **2a** in a pure form. ^{2}H NMR (91 MHz, $CHCl_3$) δ 1.48, 4.50.

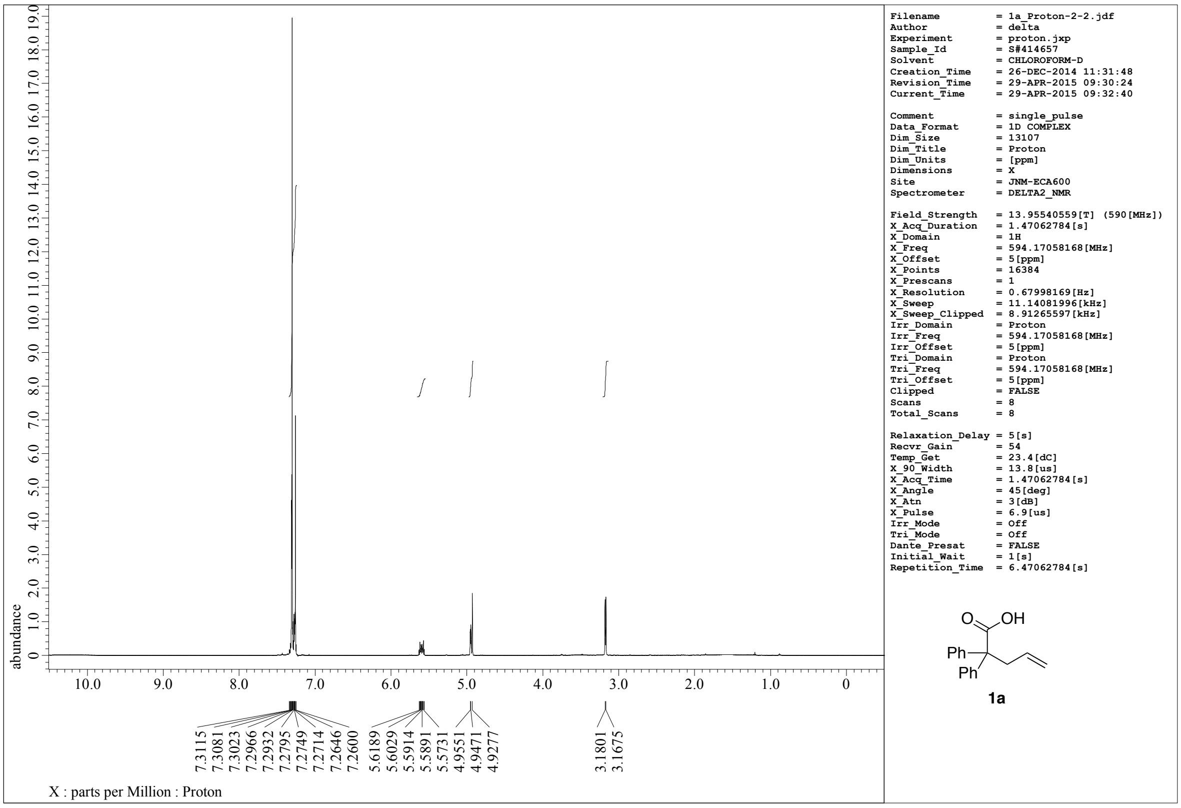
eqn (5)



1a-*d*$_2$ (1.87D) **2a-*d***: 4% yield **1a-*d***: 76% recovered

(*R*)-DTBM-segphos (11.8 mg, 0.010 mmol, 5 mol%) and $[IrCl(coe)_2]_2$ (4.5 mg, 0.010 mmol of Ir, 5 mol% of Ir) were placed in a Schlenk tube under nitrogen. NMP (0.8 mL) was added and the mixture was stirred at room temperature for 10 min. **1a-*d*$_2$** (0.20 mmol) was added and the Schlenk tube was capped with a glass stopper and heated at 100 °C for 1 h with stirring. The mixture was diluted with Et_2O, washed with H_2O and brine, dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. The yields were determined by ^{1}H NMR using 1,4-dimethoxybenzene as an internal standard. The residue was subjected to preparative TLC on silica gel to give unreacted **1a** and **2a** with impurities, which was subjected to GPC with $CHCl_3$ to give **2a** in a pure form. ^{2}H NMR (91 MHz, $CHCl_3$) δ 1.48, 4.50.

12. ^{1}H and ^{13}C NMR spectra and chiral HPLC charts



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X_Sweep_Clipped   = 8.91265597[kHz]
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X_Angle           = 45[deg]
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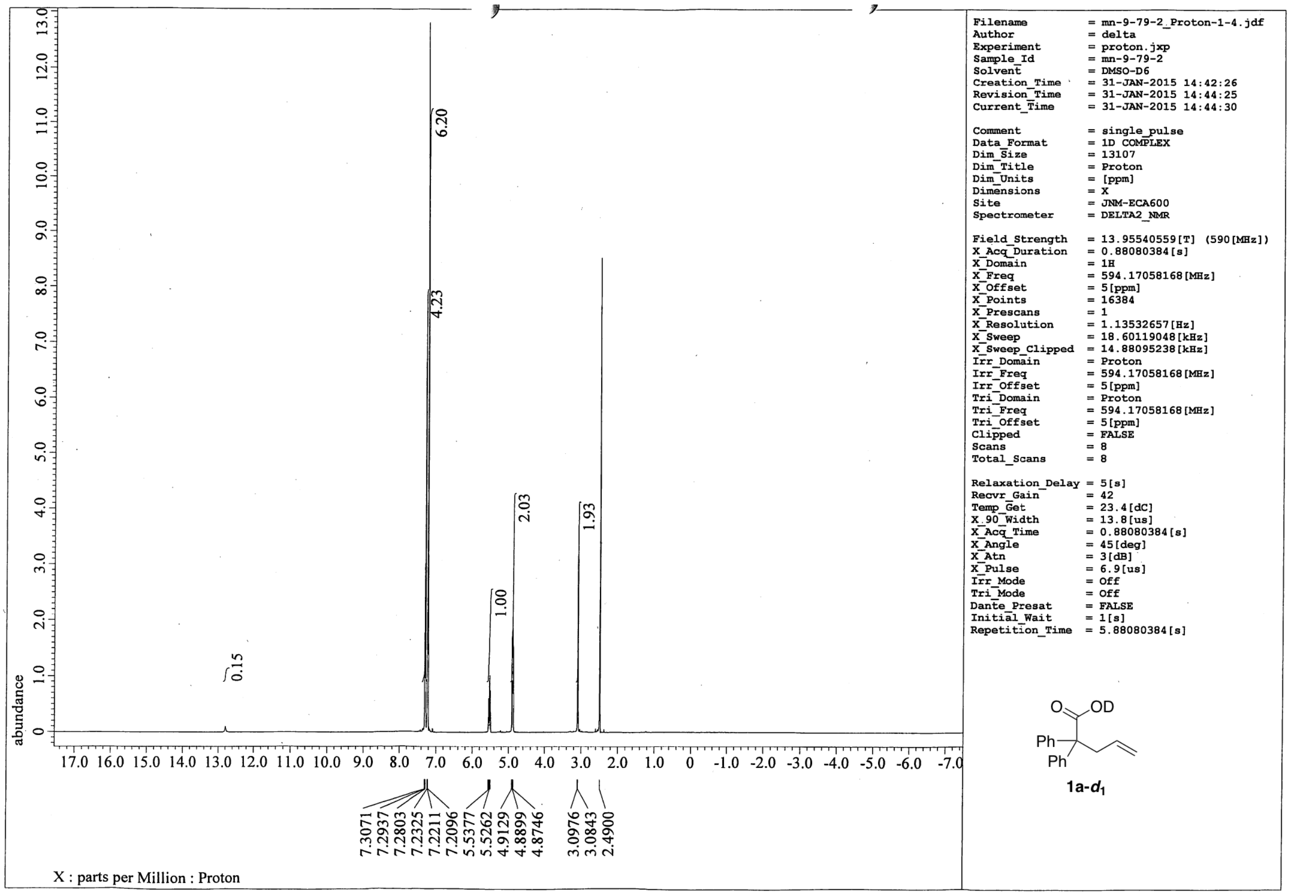
1a



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Current_Time      = 31-JAN-2015 14:44:30

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Spectrometer      = DELTA2_NMR

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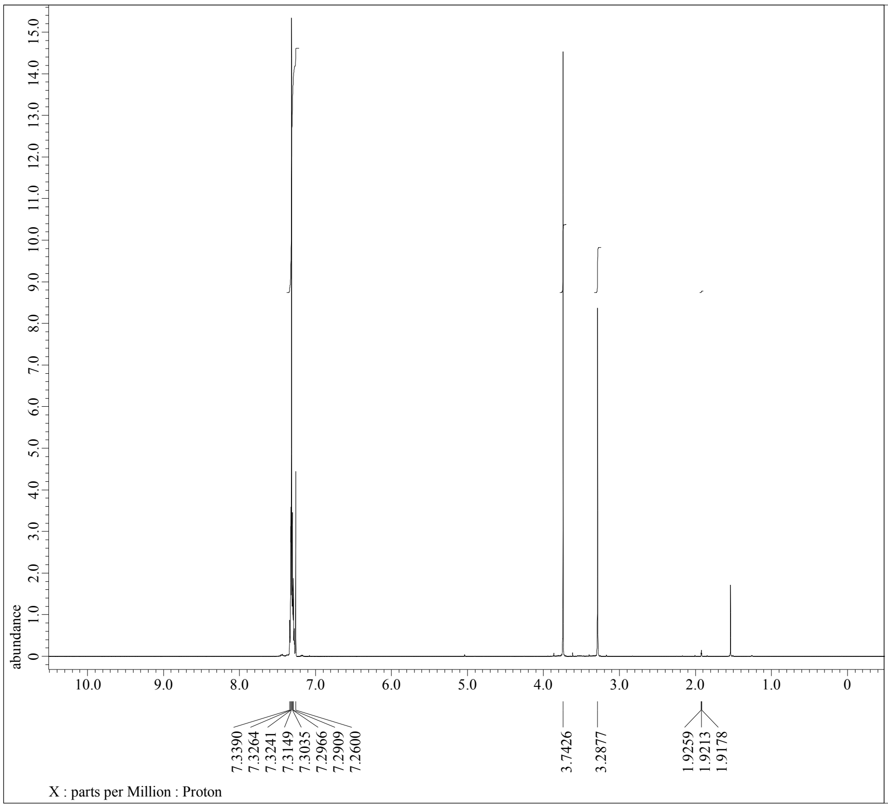
1a-d_1



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Spectrometer      = DELTA2_NMR

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Irr_Freq          = 594.17058168[MHz]
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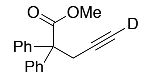
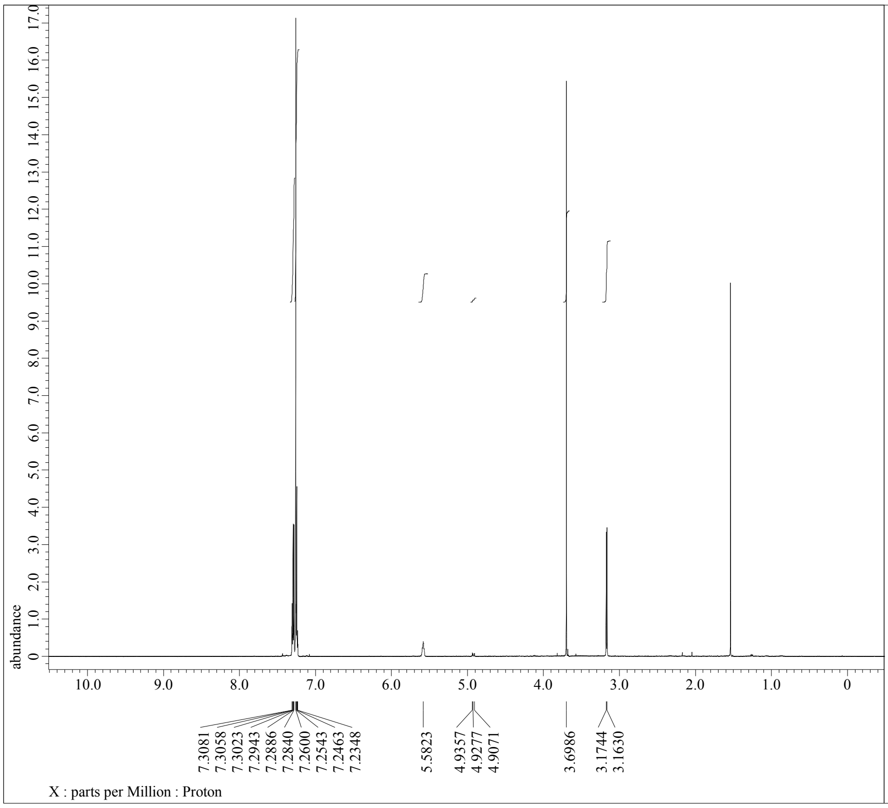


s2



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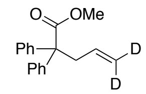

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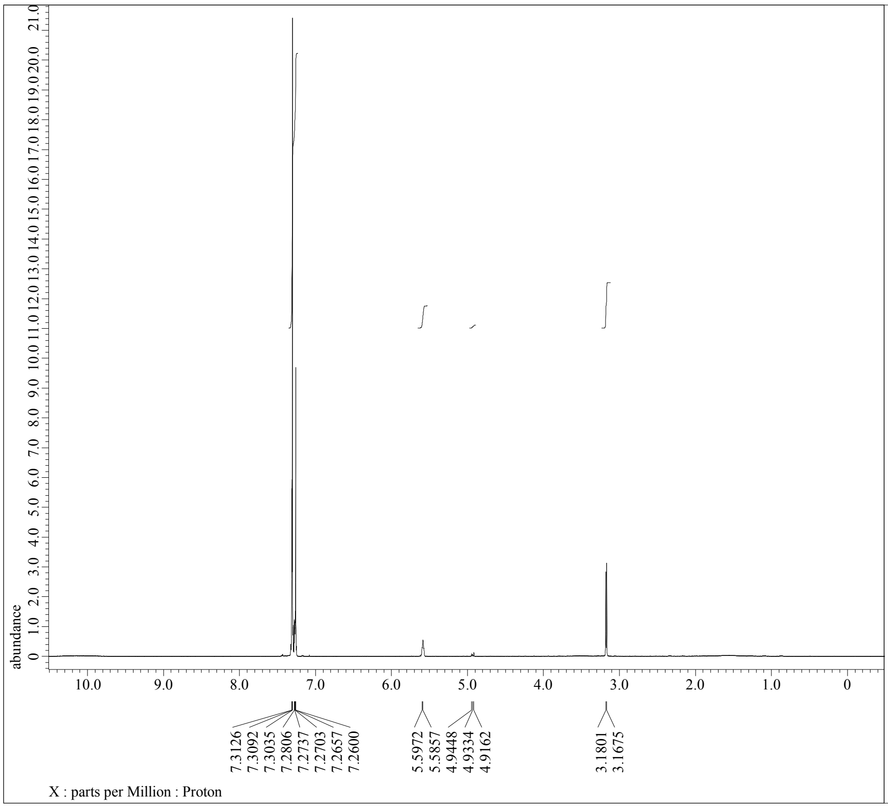

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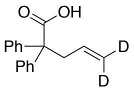
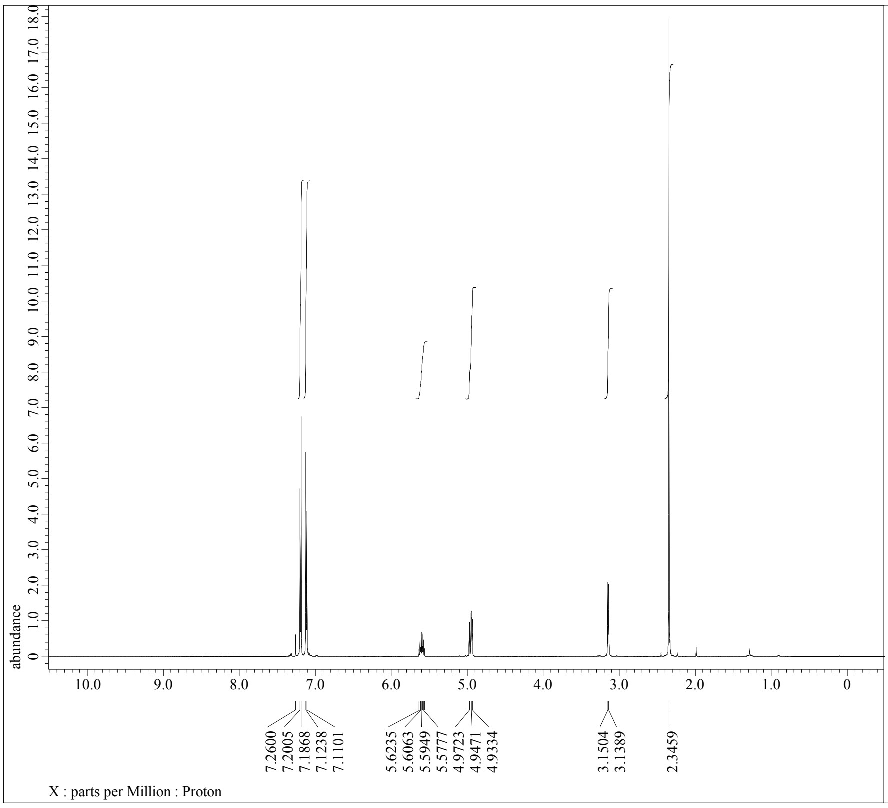


1a-d_2



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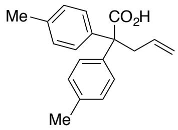
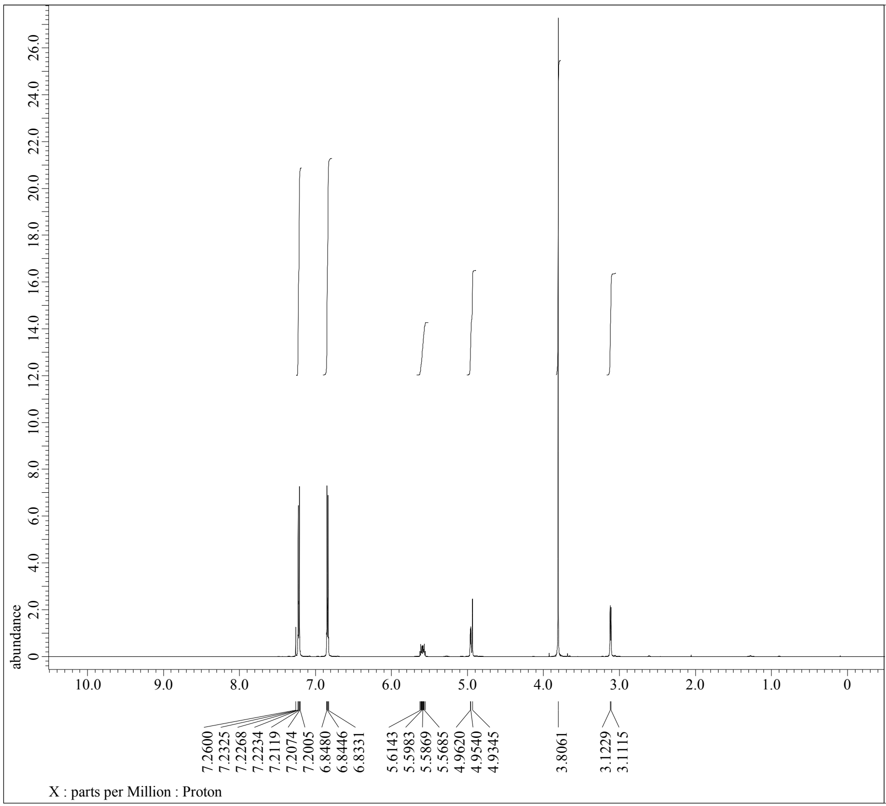


1b



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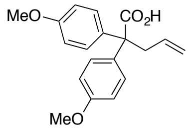
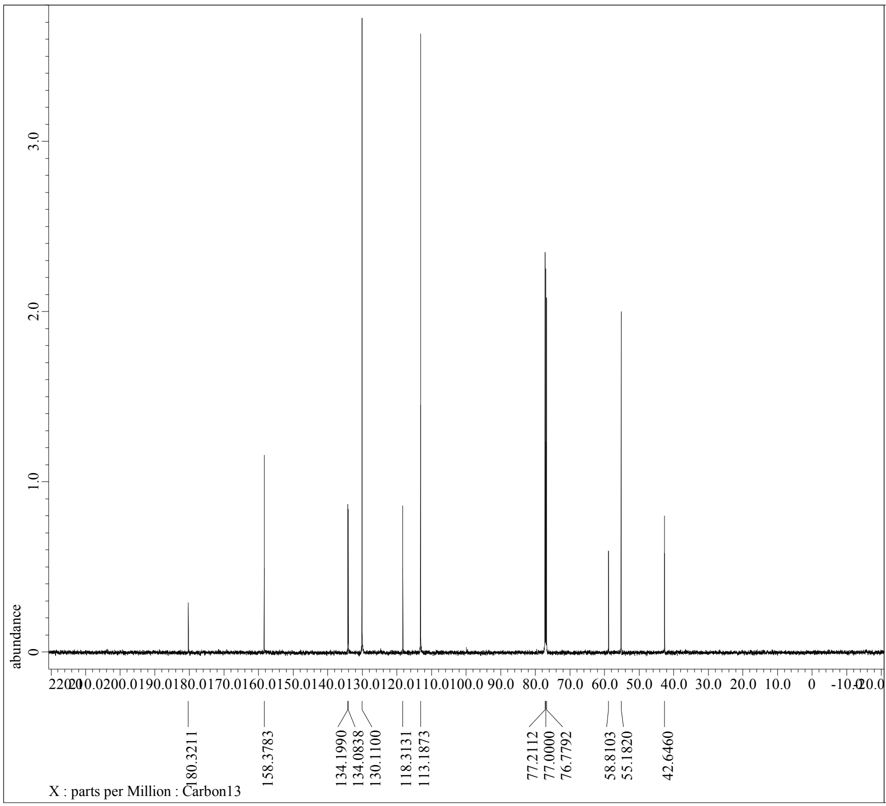
X_Sweep_Clipped   = 8.91265597[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 34
Temp_Get          = 26.9[dC]
X_90_Width        = 13.8[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.9[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



1c

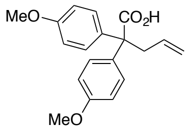


```
Filename          = 20tn148_Carbon-1-1.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 20tn148
Solvent           = CHLOROFORM-D
Creation_Time     =  7-JUL-2014 18:37:51
Revision_Time     =  7-JUL-2014 18:49:44
Current_Time      = 29-APR-2015 09:46:28

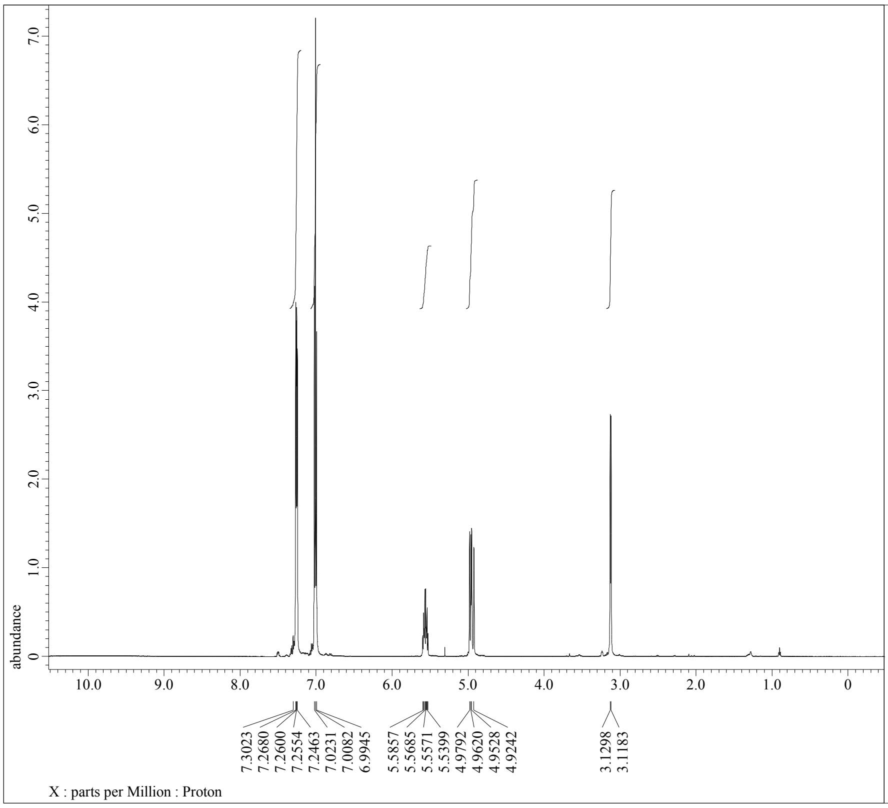
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 0.69730304[s]
X_Domain          = 13C
X_Freq            = 149.40429612[MHz]
X_Offset          = 100.0[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.43409672[Hz]
X_Sweep           = 46.9924812[kHz]
X_Sweep_Clipped   = 37.59398496[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 256
Total_Scans       = 256

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 27.8[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



1c

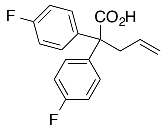


```
Filename          = 20tnp-f_Proton-1-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 20tnp-f
Solvent           = CHLOROFORM-D
Creation_Time     = 30-JUN-2014 15:48:53
Revision_Time     = 12-MAY-2015 17:23:40
Current_Time      = 12-MAY-2015 17:24:13

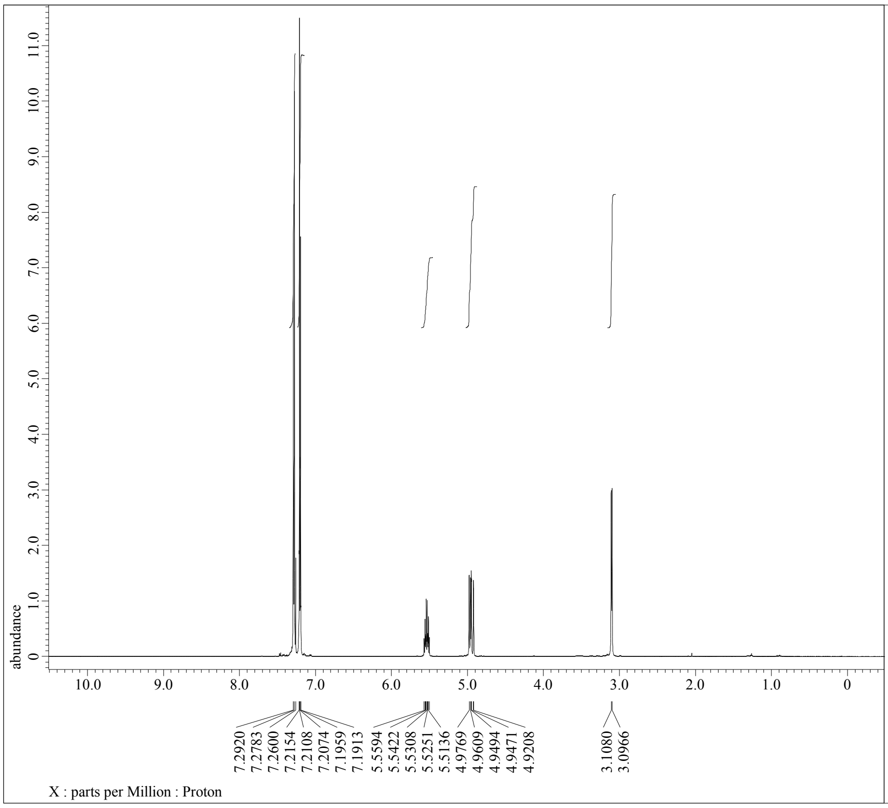
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 1.47062784[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.67998169[Hz]
X_Sweep           = 11.14081996[kHz]
X_Sweep_Clipped   = 8.91265597[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 38
Temp_Get          = 26.3[dC]
X_90_Width        = 13.8[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.9[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



1d

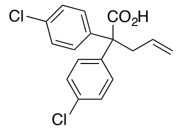


```
Filename          = 20tn144_Proton-1-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 20tn144
Solvent           = CHLOROFORM-D
Creation_Time     =  3-JUL-2014 09:32:24
Revision_Time     = 29-APR-2015 09:48:14
Current_Time      = 29-APR-2015 09:48:21

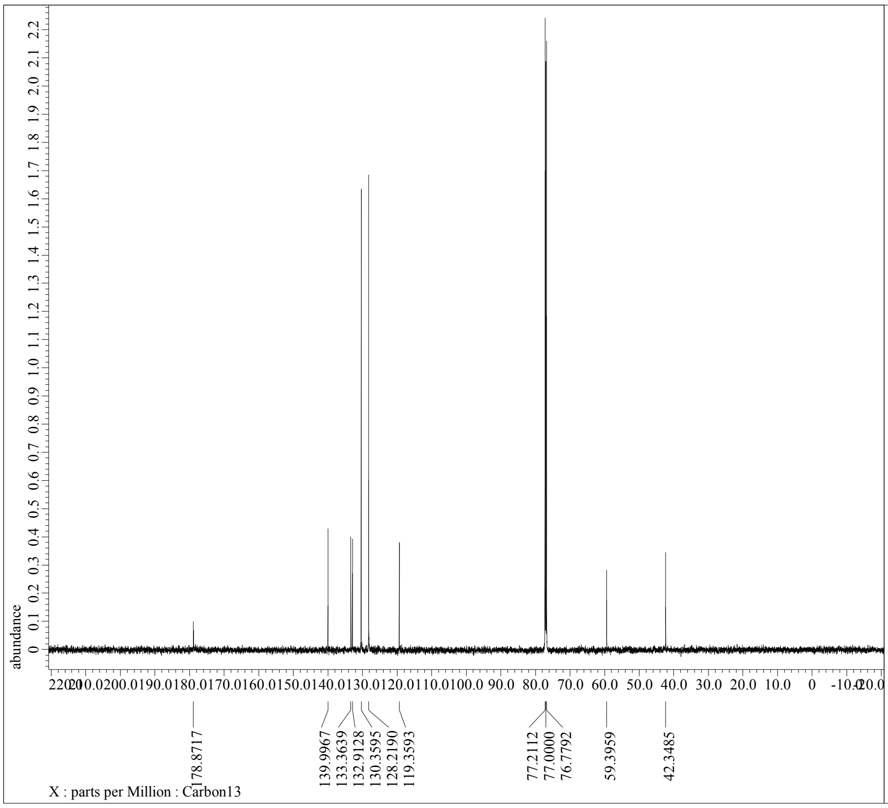
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 1.47062784[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.67998169[Hz]
X_Sweep           = 11.14081996[kHz]
X_Sweep_Clipped   = 8.91265597[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 44
Temp_Get          = 26.6[dC]
X_90_Width        = 13.8[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.9[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



1e

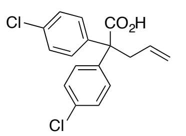


```
Filename          = 20tn144_Carbon-1-1.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 20tn144
Solvent           = CHLOROFORM-D
Creation_Time     =  3-JUL-2014 09:35:11
Revision_Time     =  3-JUL-2014 09:48:26
Current_Time      = 29-APR-2015 09:48:51

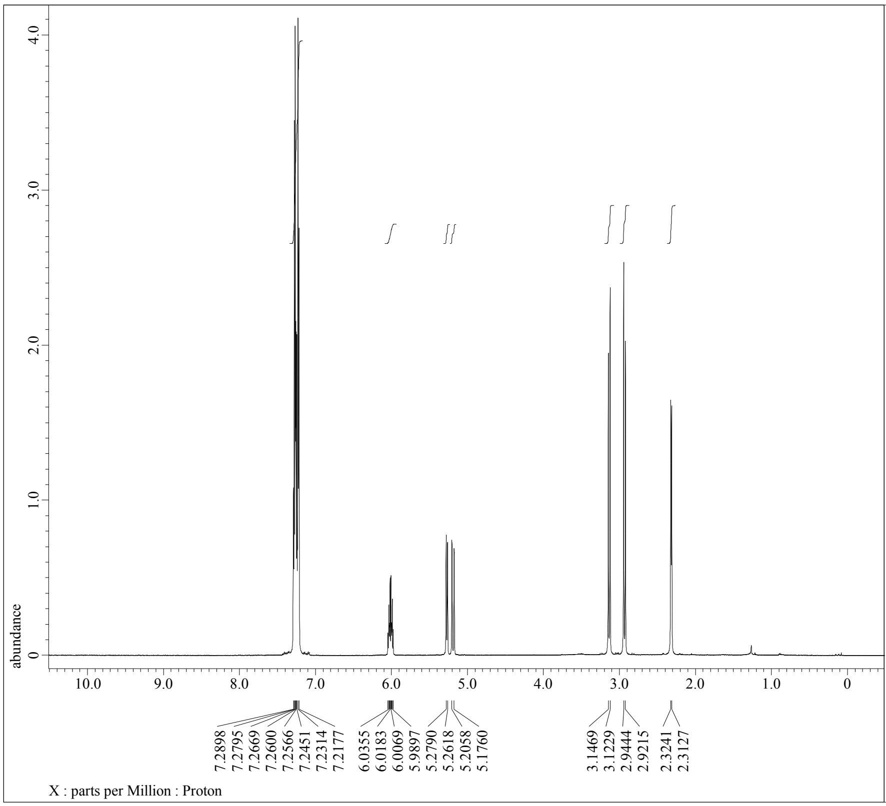
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 0.69730304[s]
X_Domain          = 13C
X_Freq            = 149.40429612[MHz]
X_Offset          = 100.0[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.43409672[Hz]
X_Sweep           = 46.9924812[kHz]
X_Sweep_Clipped   = 37.59398496[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 256
Total_Scans       = 256

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 27.6[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



1e

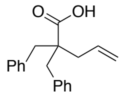


```
Filename          = mn-7-44_Proton-2-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = mn-7-44
Solvent           = CHLOROFORM-D
Creation_Time     = 12-JUN-2014 17:35:19
Revision_Time     = 29-APR-2015 09:50:08
Current_Time      = 29-APR-2015 09:50:21

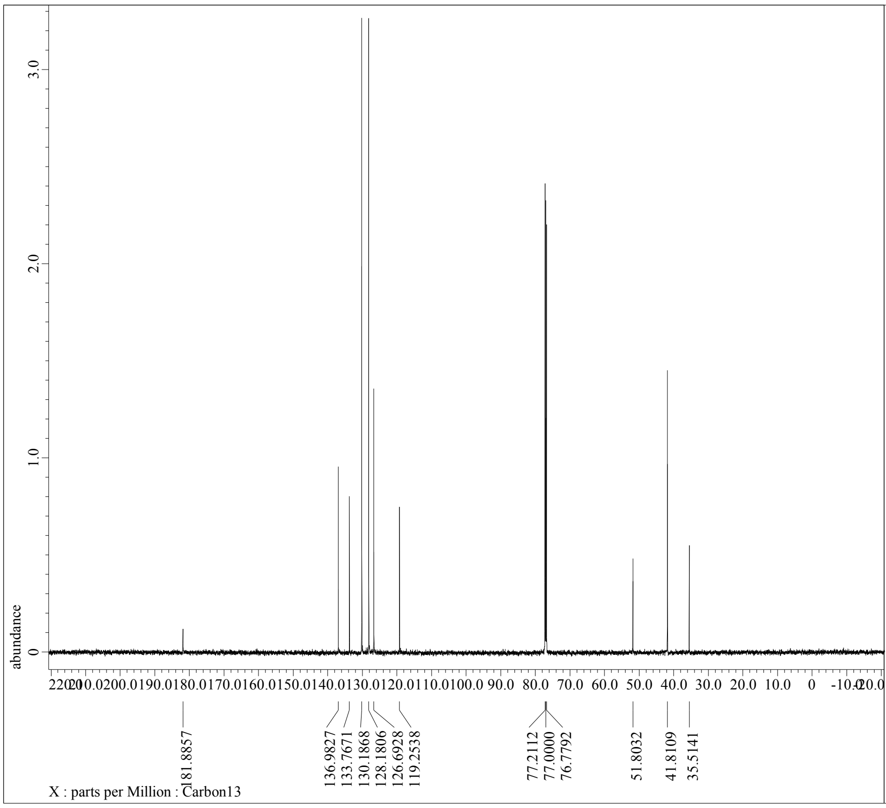
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 1.47062784[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.67998169[Hz]
X_Sweep           = 11.14081996[kHz]
X_Sweep_Clipped   = 8.91265597[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 42
Temp_Get          = 25.9[dC]
X_90_Width        = 13.8[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.9[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



1f

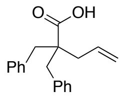


```
Filename          = mn-7-44_Carbon-1-5.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = mn-7-44
Solvent           = CHLOROFORM-D
Creation_Time     = 25-NOV-2014 21:59:04
Revision_Time     = 29-APR-2015 09:51:16
Current_Time      = 29-APR-2015 09:51:31

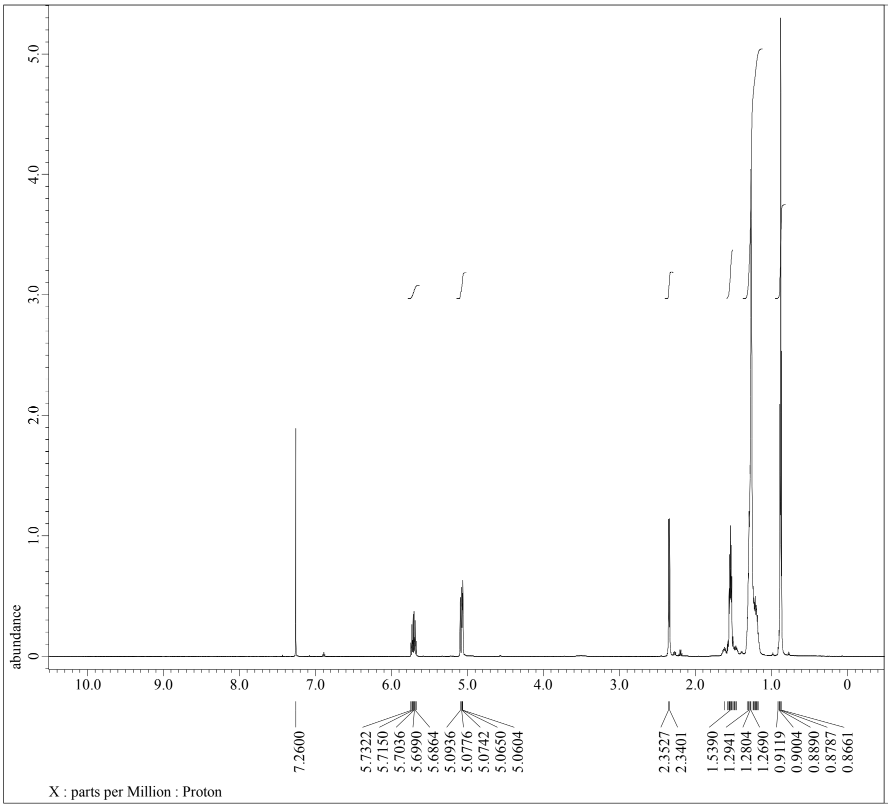
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 0.69730304[s]
X_Domain          = 13C
X_Freq            = 149.40429612[MHz]
X_Offset          = 100.0[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.43409672[Hz]
X_Sweep           = 46.9924812[kHz]
X_Sweep_Clipped   = 37.59398496[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 256
Total_Scans       = 256

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 25[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



1f

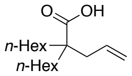


```
Filename          = mn-7-78_Proton-1-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = mn-7-78
Solvent           = CHLOROFORM-D
Creation_Time     = 26-JUN-2014 14:59:54
Revision_Time     = 29-APR-2015 09:52:58
Current_Time      = 29-APR-2015 09:53:15

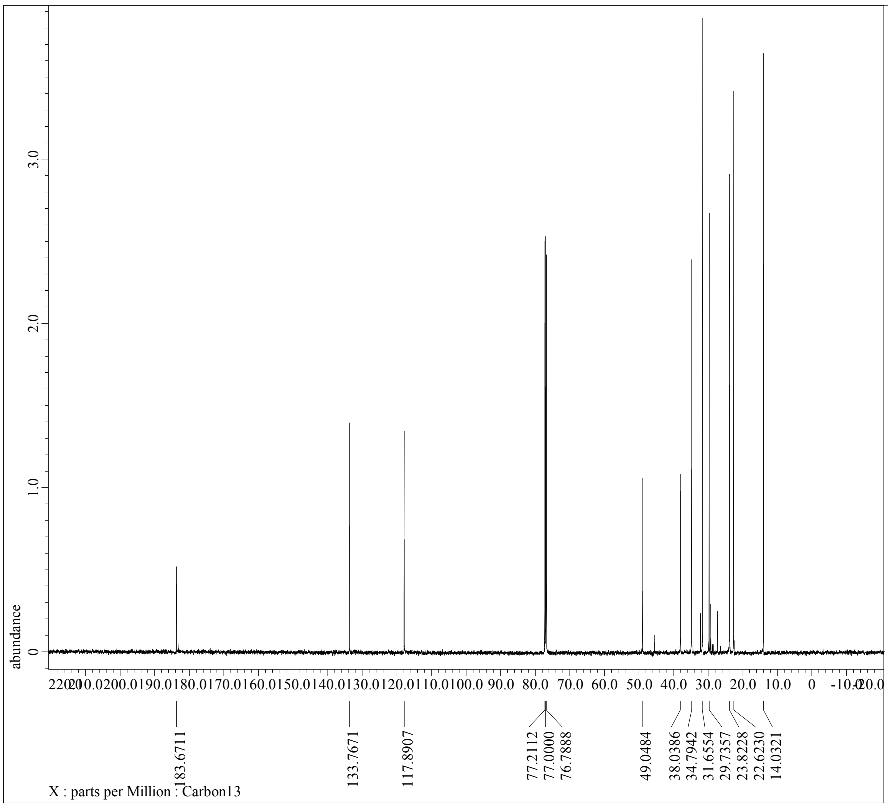
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 1.47062784[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.67998169[Hz]
X_Sweep           = 11.14081996[kHz]
X_Sweep_Clipped   = 8.91265597[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 16
Total_Scans       = 16

Relaxation_Delay = 5[s]
Recvr_Gain        = 42
Temp_Get          = 26.1[dC]
X_90_Width        = 13.8[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.9[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



1g

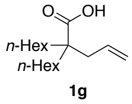


```
Filename          = mn-7-78_Carbon-5-2.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = S#718538
Solvent           = CHLOROFORM-D
Creation_Time     = 26-NOV-2014 19:59:33
Revision_Time     = 29-APR-2015 09:54:29
Current_Time      = 29-APR-2015 09:54:46

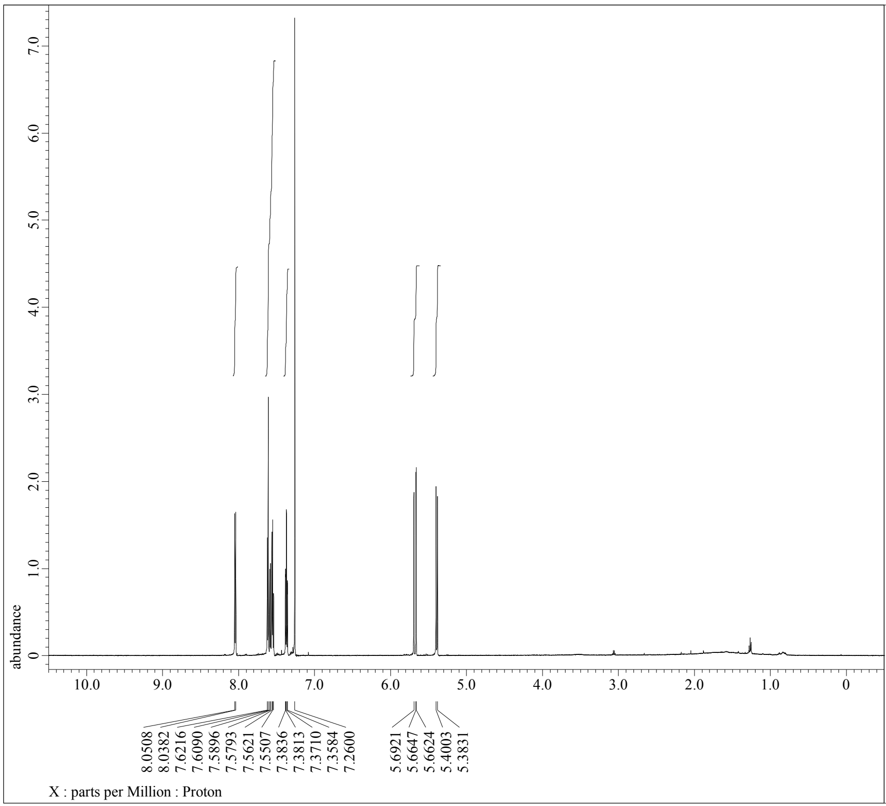
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 0.69730304[s]
X_Domain          = 13C
X_Freq            = 149.40429612[MHz]
X_Offset          = 100.0[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.43409672[Hz]
X_Sweep           = 46.9924812[kHz]
X_Sweep_Clipped   = 37.59398496[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 256
Total_Scans       = 256

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 25[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



1g

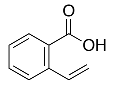


```
Filename          = mn-7-6_Proton-1-4.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = mn-7-6
Solvent           = CHLOROFORM-D
Creation_Time     = 13-MAY-2015 20:36:15
Revision_Time     = 13-MAY-2015 20:39:50
Current_Time      = 13-MAY-2015 20:40:01

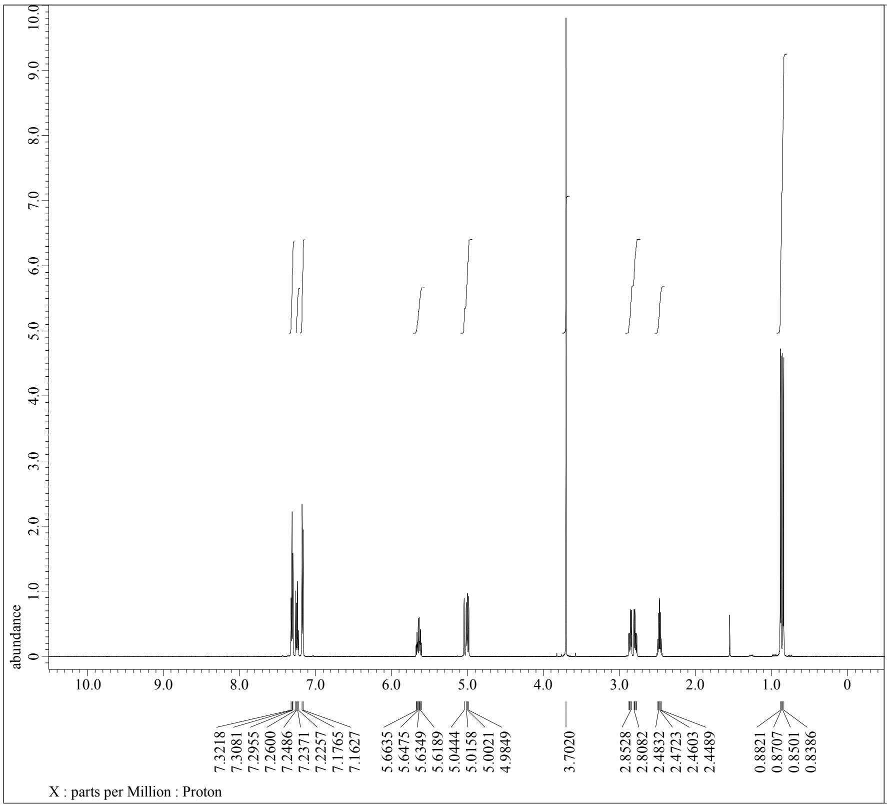
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 1.47062784[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.67998169[Hz]
X_Sweep           = 11.14081996[kHz]
X_Sweep_Clipped   = 8.91265597[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 54
Temp_Get          = 23.4[dC]
X_90_Width        = 14[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 7[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



1h

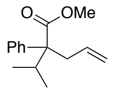


```
Filename          = mn-9-55_Proton-1-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = mn-9-55
Solvent           = CHLOROFORM-D
Creation_Time     = 21-JAN-2015 21:00:12
Revision_Time     = 29-APR-2015 09:55:39
Current_Time      = 29-APR-2015 09:55:56

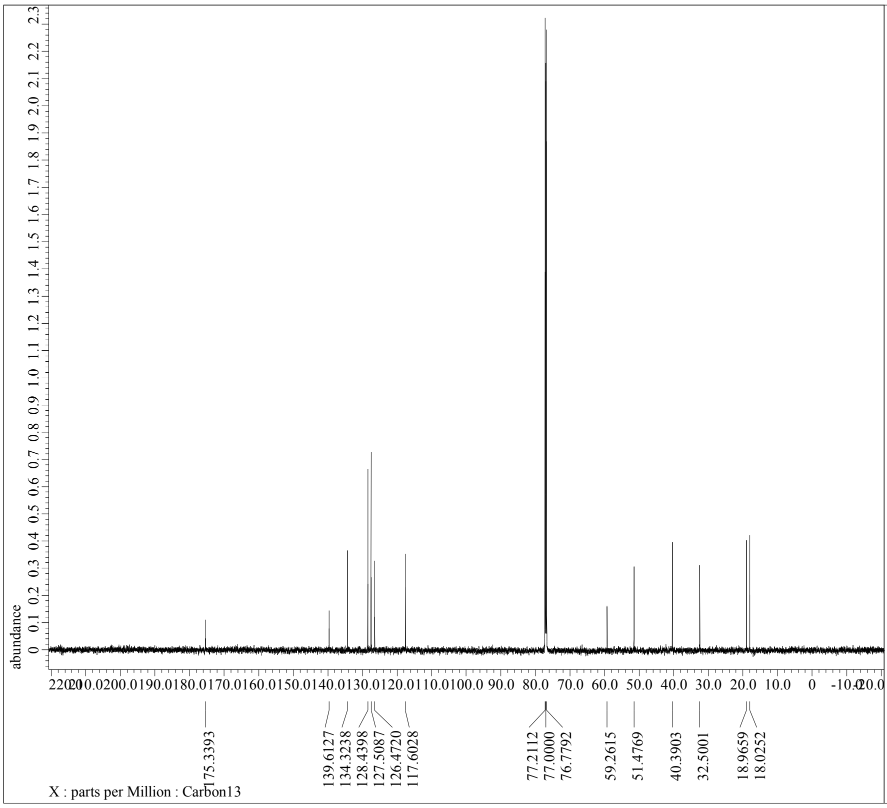
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 1.47062784[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.67998169[Hz]
X_Sweep           = 11.14081996[kHz]
X_Sweep_Clipped   = 8.91265597[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 42
Temp_Get          = 23.6[dC]
X_90_Width        = 13.8[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.9[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



s5

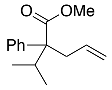


```
Filename          = mn-9-55_Carbon-1-1.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = mn-9-55
Solvent           = CHLOROFORM-D
Creation_Time     = 21-JAN-2015 21:03:03
Revision_Time     = 21-JAN-2015 21:15:24
Current_Time      = 29-APR-2015 09:56:27

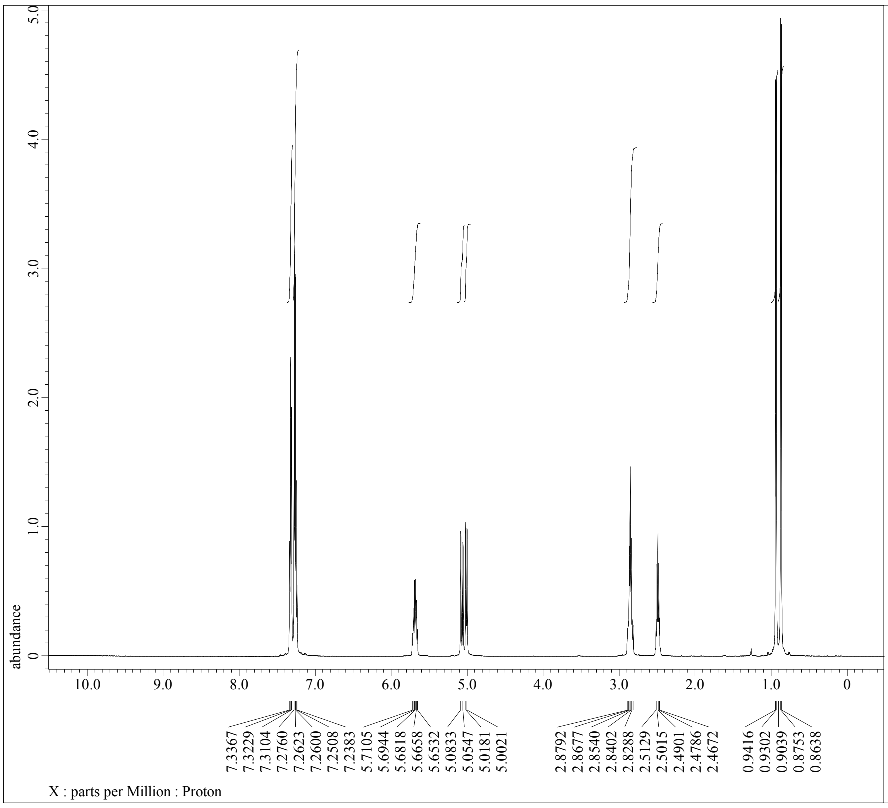
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 0.69730304[s]
X_Domain          = 13C
X_Freq            = 149.40429612[MHz]
X_Offset          = 100.0[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.43409672[Hz]
X_Sweep           = 46.9924812[kHz]
X_Sweep_Clipped   = 37.59398496[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 256
Total_Scans       = 256

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.4[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



s5

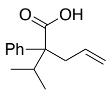


```
Filename          = mn-9-64_Proton-1-2.jdf
Author            = console
Experiment        = proton.jxp
Sample_Id         = mn-9-64
Solvent           = CHLOROFORM-D
Creation_Time     = 24-JAN-2015 17:49:44
Revision_Time     = 29-APR-2015 09:57:07
Current_Time      = 29-APR-2015 09:57:21

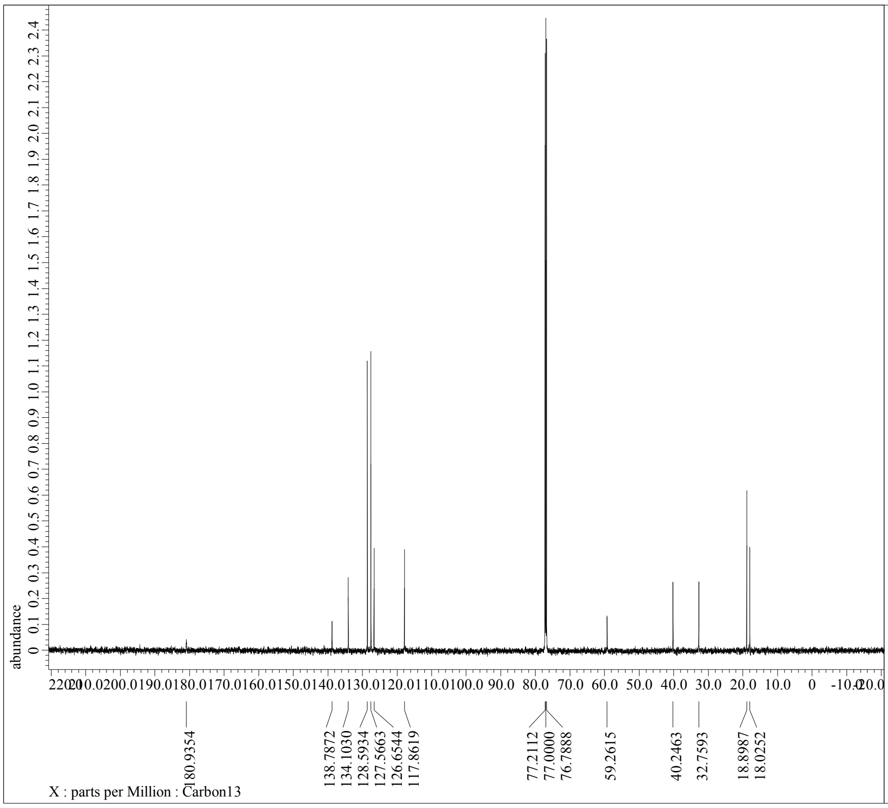
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 1.47062784[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.67998169[Hz]
X_Sweep           = 11.14081996[kHz]
X_Sweep_Clipped   = 8.91265597[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 40
Temp_Get          = 23.7[dC]
X_90_Width        = 13.8[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.9[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



1i

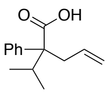


```
Filename          = mn-9-64_Carbon-1-2.jdf
Author            = console
Experiment        = carbon.jxp
Sample_Id         = mn-9-64
Solvent           = CHLOROFORM-D
Creation_Time     = 24-JAN-2015 17:52:50
Revision_Time     = 29-APR-2015 09:57:50
Current_Time      = 29-APR-2015 09:58:06

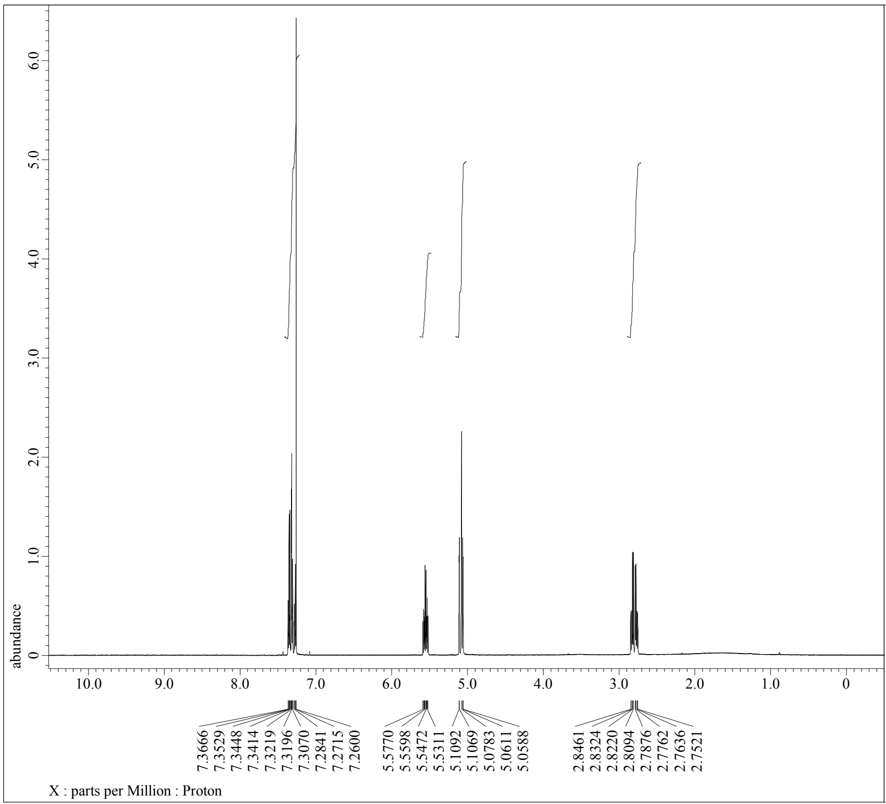
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 0.69730304[s]
X_Domain          = 13C
X_Freq            = 149.40429612[MHz]
X_Offset          = 100.0[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.43409672[Hz]
X_Sweep           = 46.9924812[kHz]
X_Sweep_Clipped   = 37.59398496[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 256
Total_Scans       = 256

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.4[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



1i

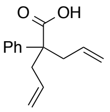


```
Filename          = mn-10-104_Proton-1-4.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = mn-10-104
Solvent           = CHLOROFORM-D
Creation_Time     = 17-JUN-2015 08:55:49
Revision_Time     = 29-JUN-2015 14:41:15
Current_Time      = 29-JUN-2015 14:41:19

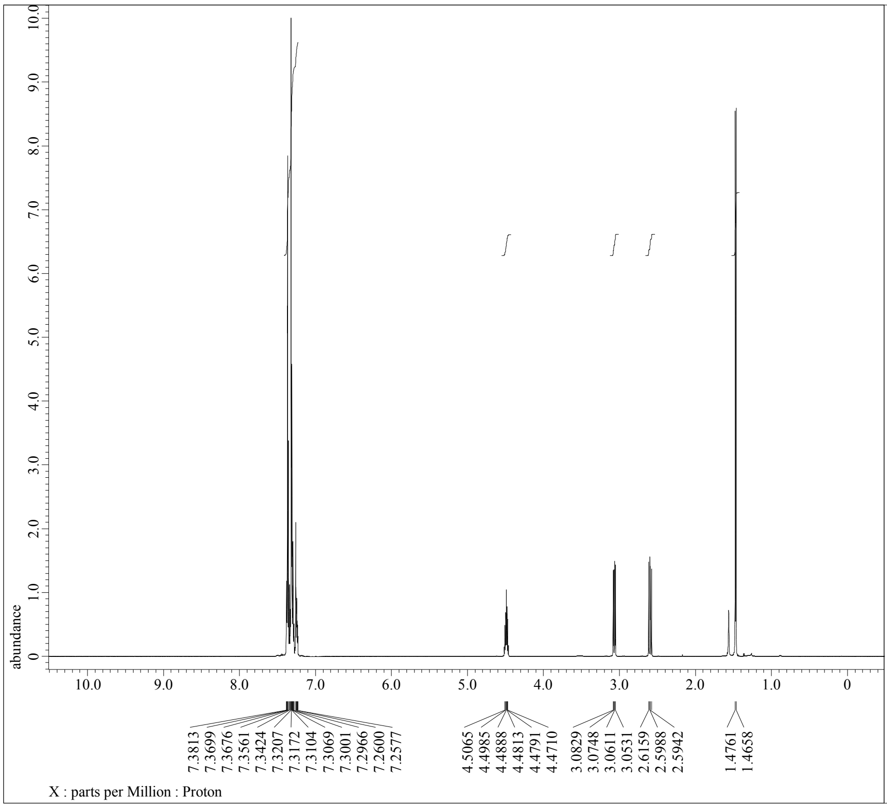
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 1.4680064[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.68119594[Hz]
X_Sweep           = 11.16071429[kHz]
X_Sweep_Clipped   = 8.92857143[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 50
Temp_Get          = 23.6[dC]
X_90_Width        = 14[us]
X_Acq_Time        = 1.4680064[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 7[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.4680064[s]
```



1j

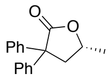


```
Filename          = mn-7-79_Proton-1-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = mn-7-79
Solvent           = CHLOROFORM-D
Creation_Time     = 26-JUN-2014 17:32:50
Revision_Time     = 29-APR-2015 09:59:10
Current_Time      = 29-APR-2015 09:59:23

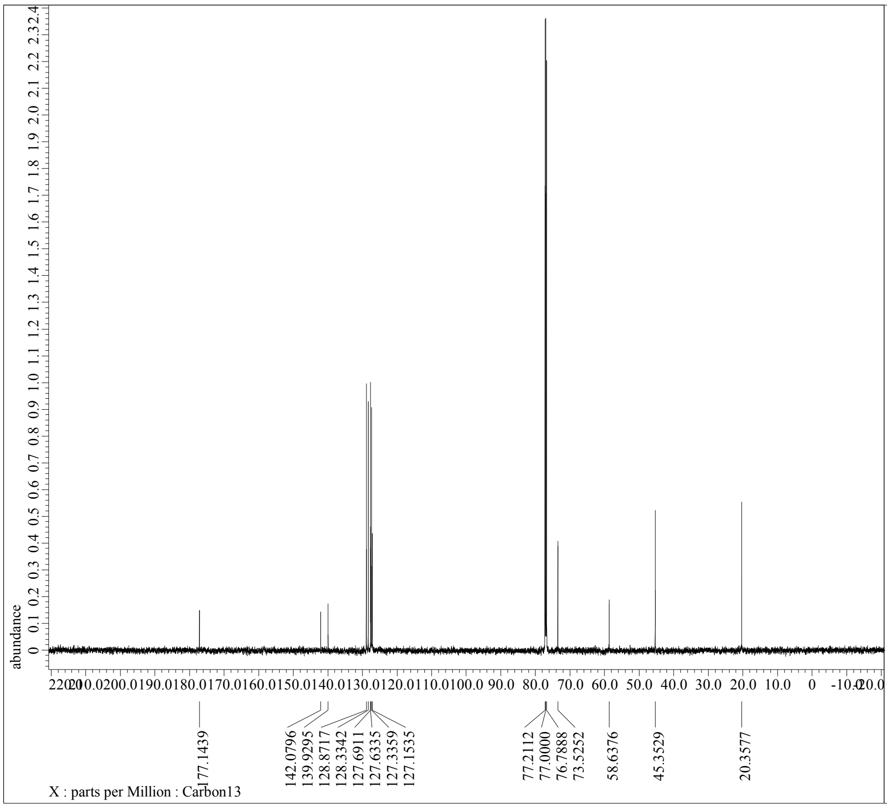
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 1.47062784[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.67998169[Hz]
X_Sweep           = 11.14081996[kHz]
X_Sweep_Clipped   = 8.91265597[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 42
Temp_Get          = 25.8[dC]
X_90_Width        = 13.8[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.9[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



2a

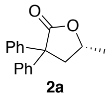


```
Filename          = mn-7-79_Carbon-1-2.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = mn-7-79
Solvent           = CHLOROFORM-D
Creation_Time     = 26-JUN-2014 17:35:35
Revision_Time     = 29-APR-2015 09:59:49
Current_Time      = 29-APR-2015 10:00:05

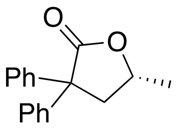
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 0.69730304[s]
X_Domain          = 13C
X_Freq            = 149.40429612[MHz]
X_Offset          = 100.0[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.43409672[Hz]
X_Sweep           = 46.9924812[kHz]
X_Sweep_Clipped   = 37.59398496[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 256
Total_Scans       = 256

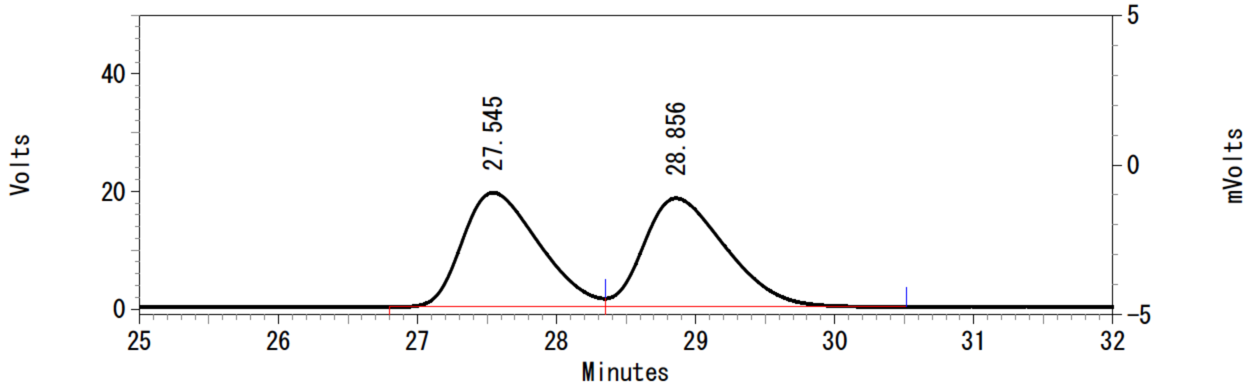
Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 26.8[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



2a

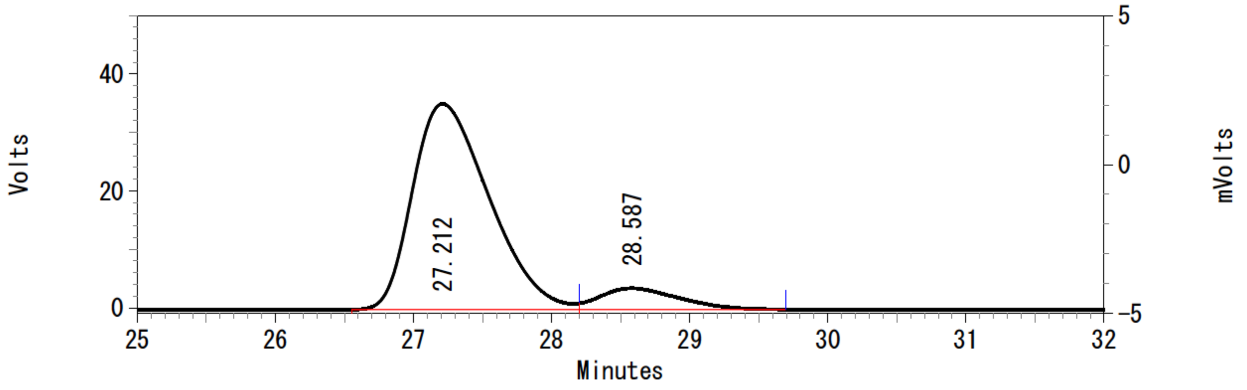


2a



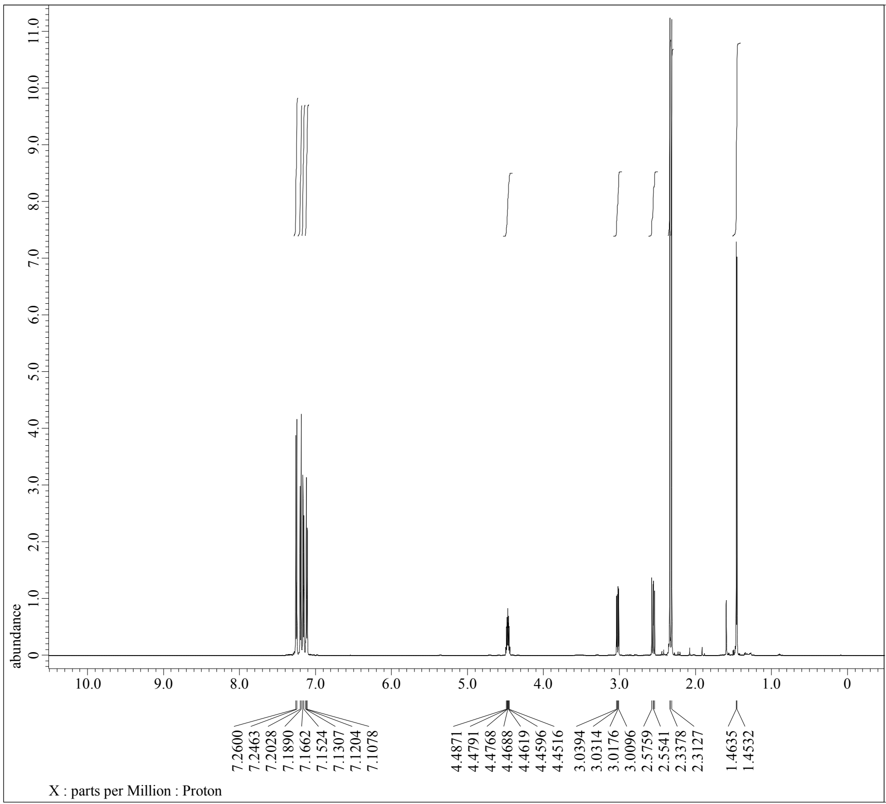
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	27.545	745579	49.536	19434
2	28.856	759533	50.464	18472
Totals		1505112	100.000	37906



UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	27.212	1355500	90.451	35207
2	28.587	143099	9.549	3634
Totals		1498599	100.000	38841

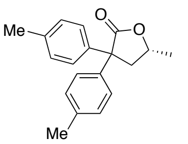


```
Filename          = mn-7-111_Proton-1-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = mn-7-111
Solvent           = CHLOROFORM-D
Creation_Time     = 10-JUL-2014 18:25:00
Revision_Time     = 29-APR-2015 10:00:56
Current_Time      = 29-APR-2015 10:01:11

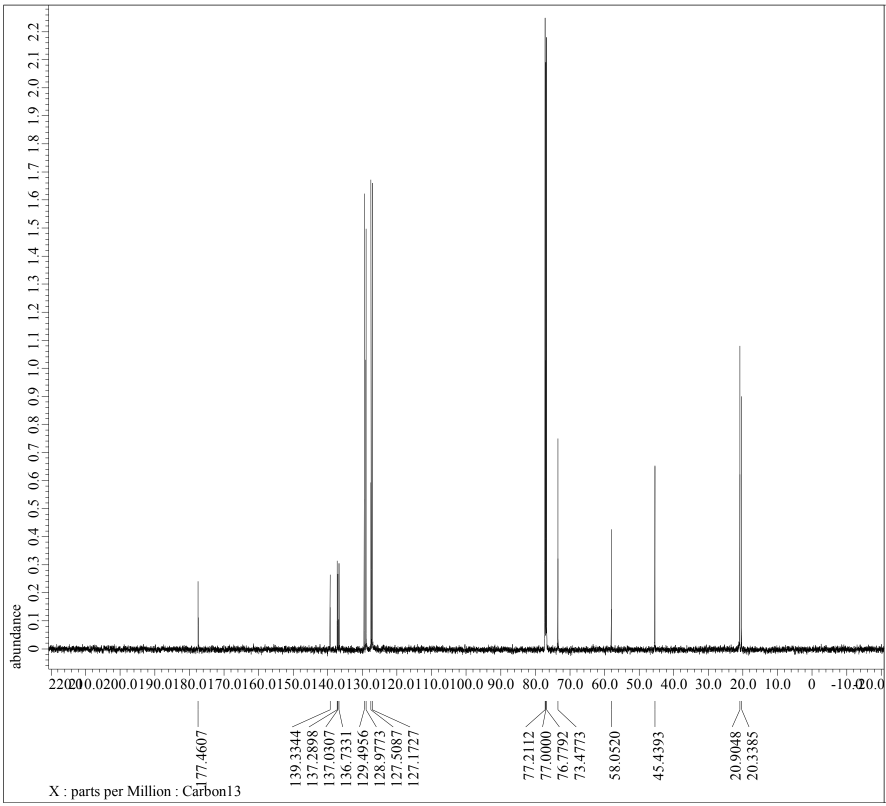
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 1.47062784[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.67998169[Hz]
X_Sweep           = 11.14081996[kHz]
X_Sweep_Clipped   = 8.91265597[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 36
Temp_Get          = 27.5[dC]
X_90_Width        = 13.8[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.9[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



2b

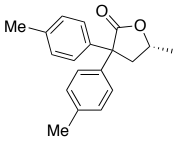


```
Filename          = mn-7-111_Carbon-1-2.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = mn-7-111
Solvent           = CHLOROFORM-D
Creation_Time     = 10-JUL-2014 18:27:42
Revision_Time     = 29-APR-2015 10:01:35
Current_Time      = 29-APR-2015 10:01:50

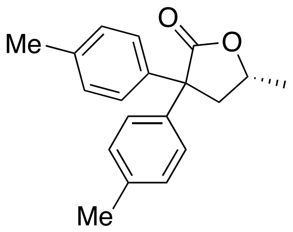
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 0.69730304[s]
X_Domain          = 13C
X_Freq            = 149.40429612[MHz]
X_Offset          = 100.0[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.43409672[Hz]
X_Sweep           = 46.9924812[kHz]
X_Sweep_Clipped   = 37.59398496[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 256
Total_Scans       = 256

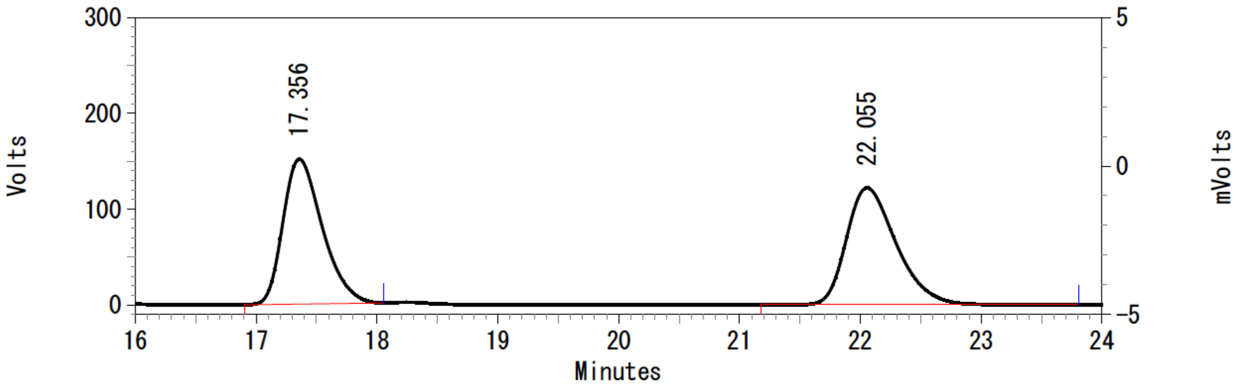
Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 28.4[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



2b

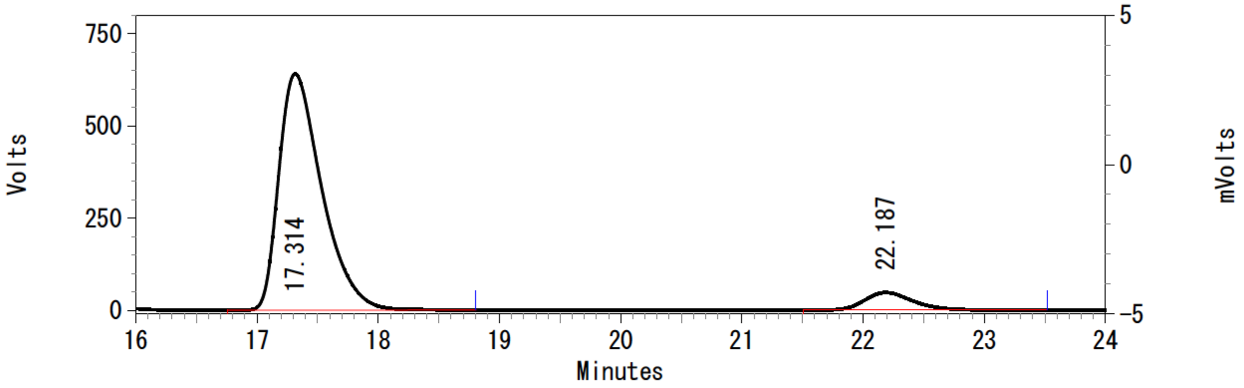


2b



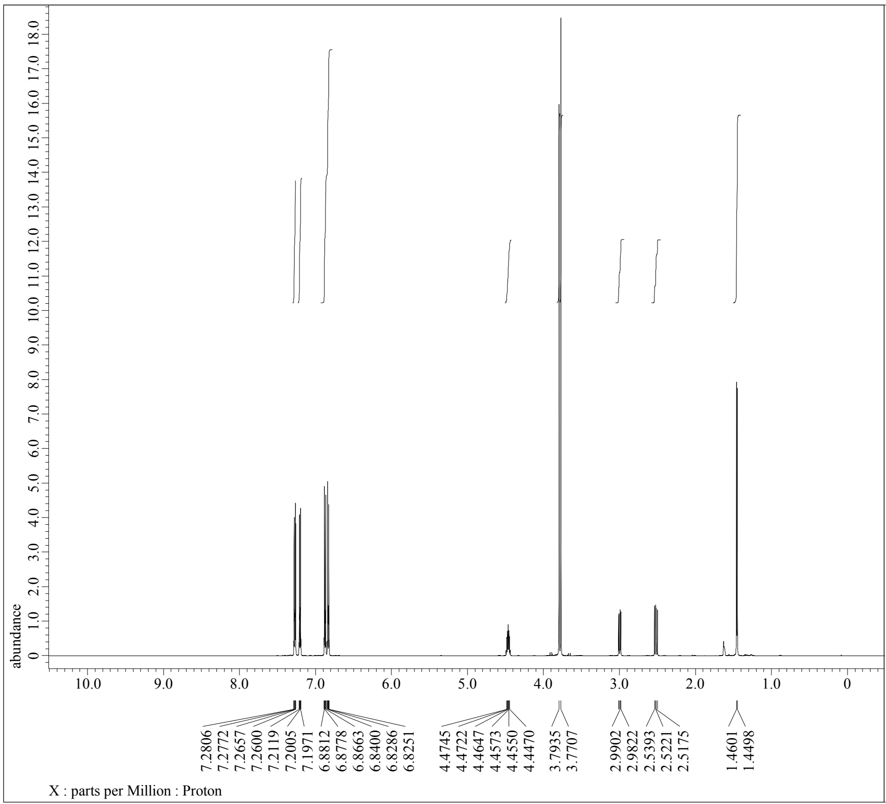
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	17.356	3417801	49.491	151317
2	22.055	3488102	50.509	121935
Totals		6905903	100.000	273252



UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	17.314	15723711	92.296	640452
2	22.187	1312481	7.704	47503
Totals		17036192	100.000	687955

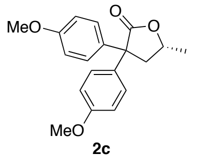


```
Filename          = mn-7-110_Proton-1-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = mn-7-110
Solvent           = CHLOROFORM-D
Creation_Time     = 10-JUL-2014 16:12:58
Revision_Time     = 29-APR-2015 10:02:54
Current_Time      = 29-APR-2015 10:03:07

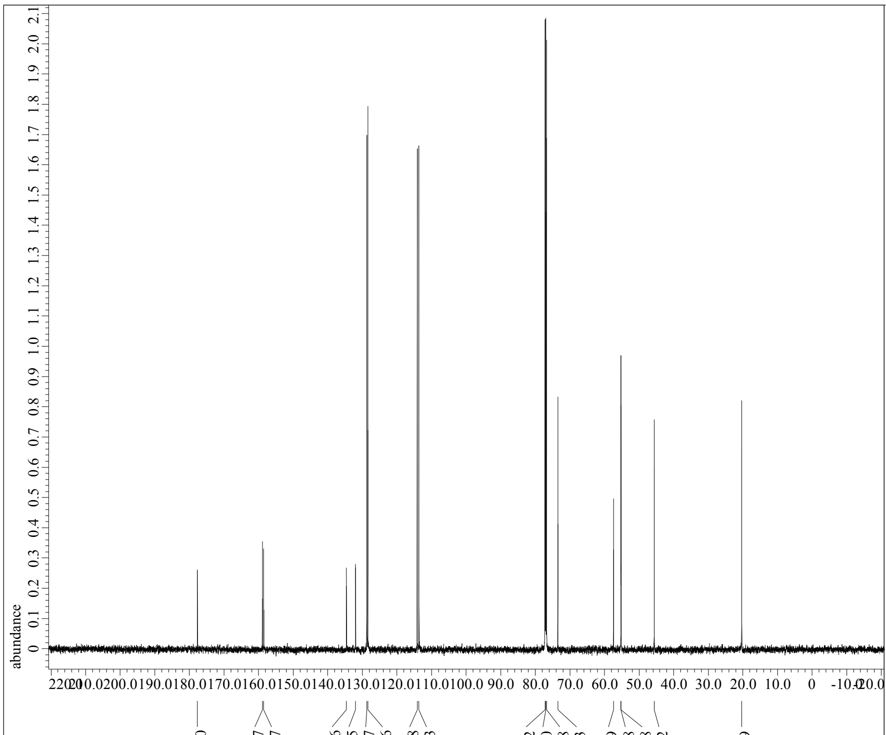
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 1.47062784[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.67998169[Hz]
X_Sweep           = 11.14081996[kHz]
X_Sweep_Clipped   = 8.91265597[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 36
Temp_Get          = 27.3[dC]
X_90_Width        = 13.8[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.9[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



2c

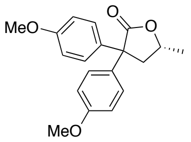


```
Filename          = mn-7-110_Carbon-1-2.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = mn-7-110
Solvent           = CHLOROFORM-D
Creation_Time     = 10-JUL-2014 16:16:05
Revision_Time     = 29-APR-2015 10:03:31
Current_Time      = 29-APR-2015 10:03:48

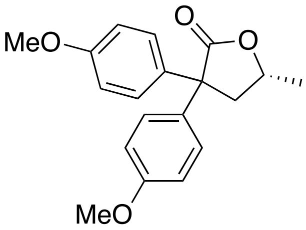
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 0.69730304[s]
X_Domain          = 13C
X_Freq            = 149.40429612[MHz]
X_Offset          = 100.0[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.43409672[Hz]
X_Sweep           = 46.9924812[kHz]
X_Sweep_Clipped   = 37.59398496[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 256
Total_Scans       = 256

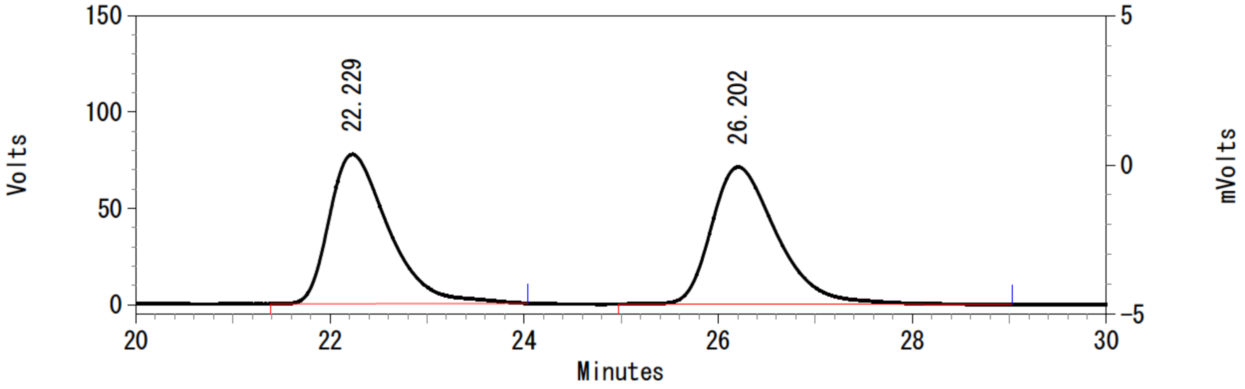
Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 28.3[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



2c

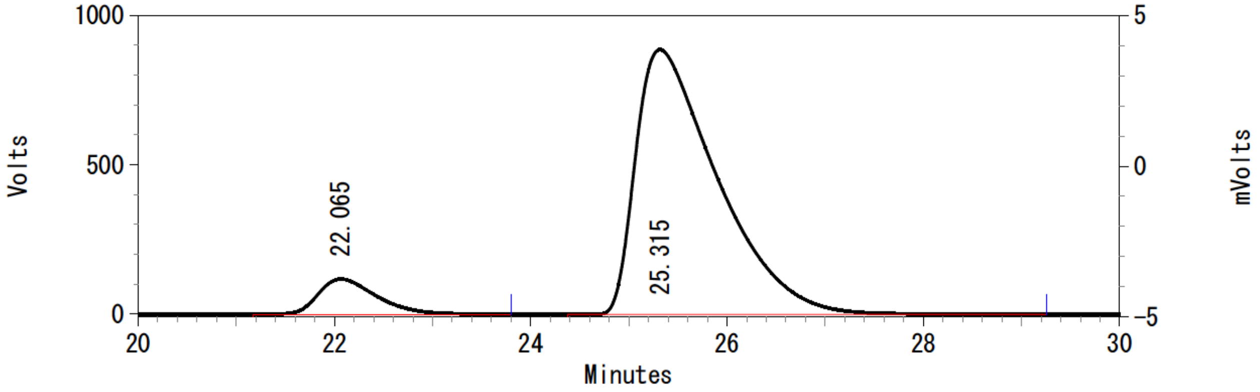


2c



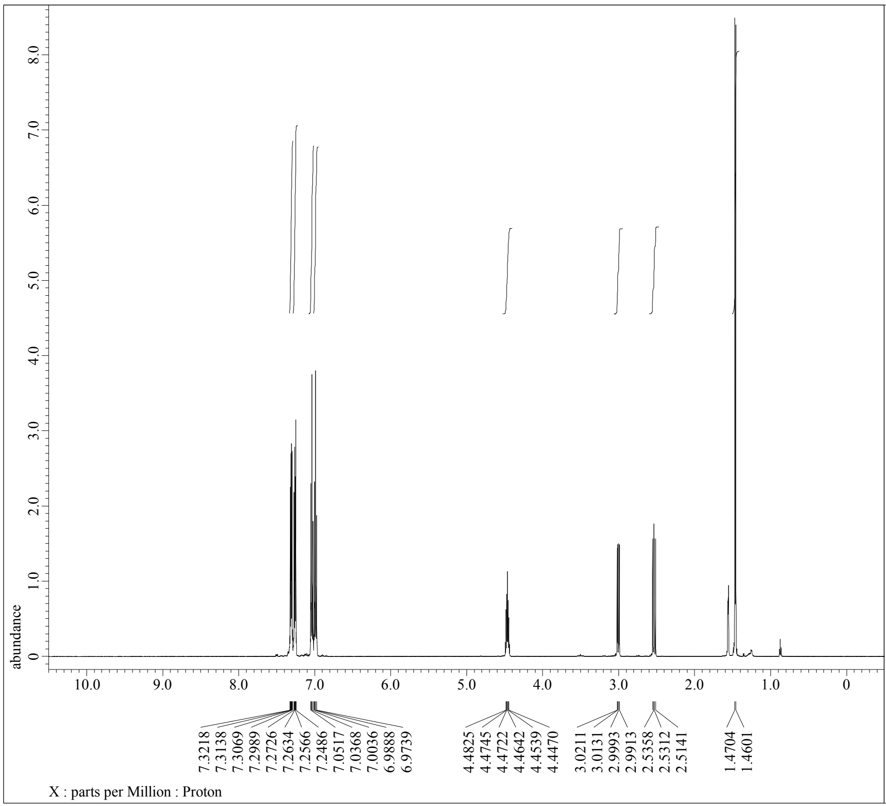
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	22.229	3290559	49.689	77463
2	26.202	3331771	50.311	71130
Totals		6622330	100.000	148593



UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	22.065	4884347	8.695	118026
2	25.315	51291926	91.305	886146
Totals		56176273	100.000	1004172

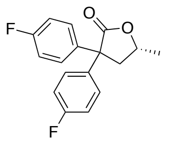


```
Filename          = mn-7-92_Proton-1-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = mn-7-92
Solvent           = CHLOROFORM-D
Creation_Time     =  2-JUL-2014 23:31:50
Revision_Time     = 29-APR-2015 10:04:55
Current_Time      = 29-APR-2015 10:05:09

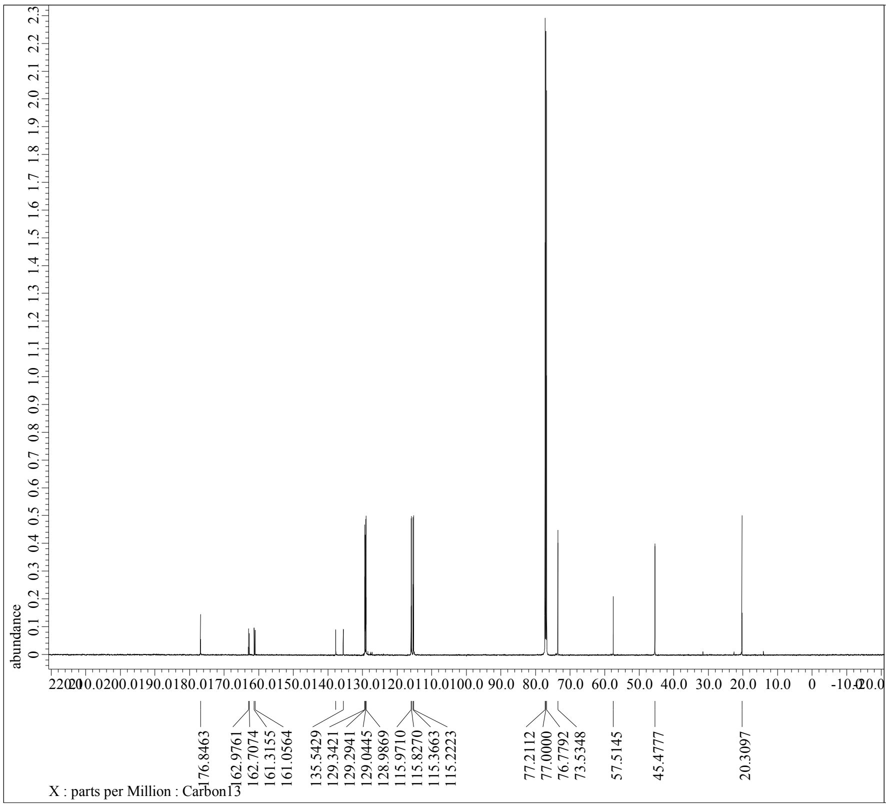
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 1.47062784[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.67998169[Hz]
X_Sweep           = 11.14081996[kHz]
X_Sweep_Clipped   = 8.91265597[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 42
Temp_Get          = 26.7[dC]
X_90_Width        = 13.8[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.9[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



2d

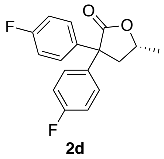


```
Filename          = mn-7-92_Carbon-1-2.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = mn-7-92
Solvent           = CHLOROFORM-D
Creation_Time     =  2-JUL-2014 23:34:36
Revision_Time     = 29-APR-2015 10:05:29
Current_Time      = 29-APR-2015 10:05:45

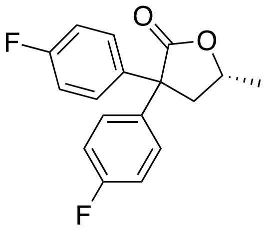
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 0.69730304[s]
X_Domain          = 13C
X_Freq            = 149.40429612[MHz]
X_Offset          = 100.0[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.43409672[Hz]
X_Sweep           = 46.9924812[kHz]
X_Sweep_Clipped   = 37.59398496[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 8000
Total_Scans       = 8000

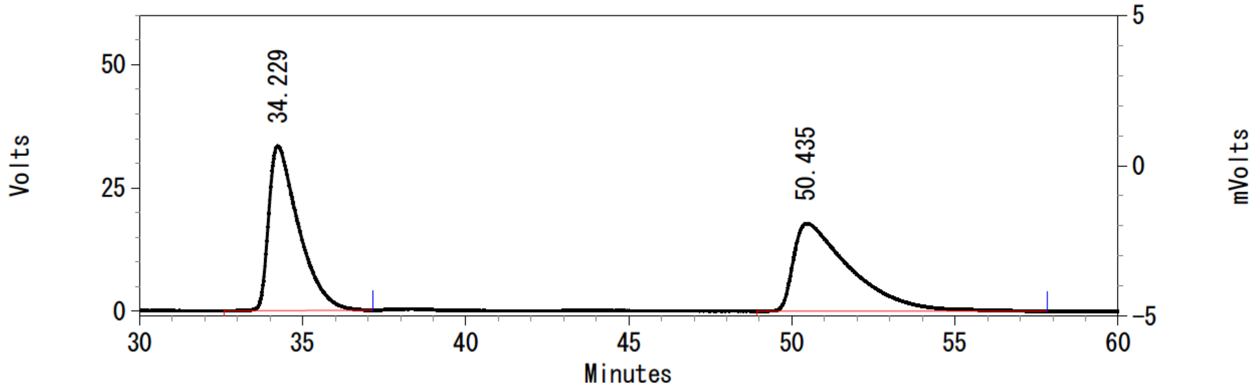
Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 27.8[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



2d

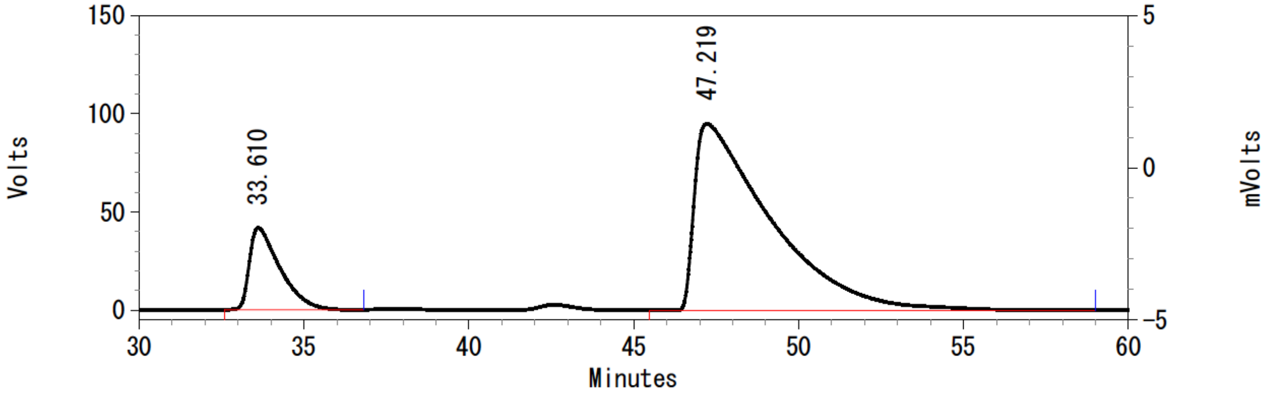


2d



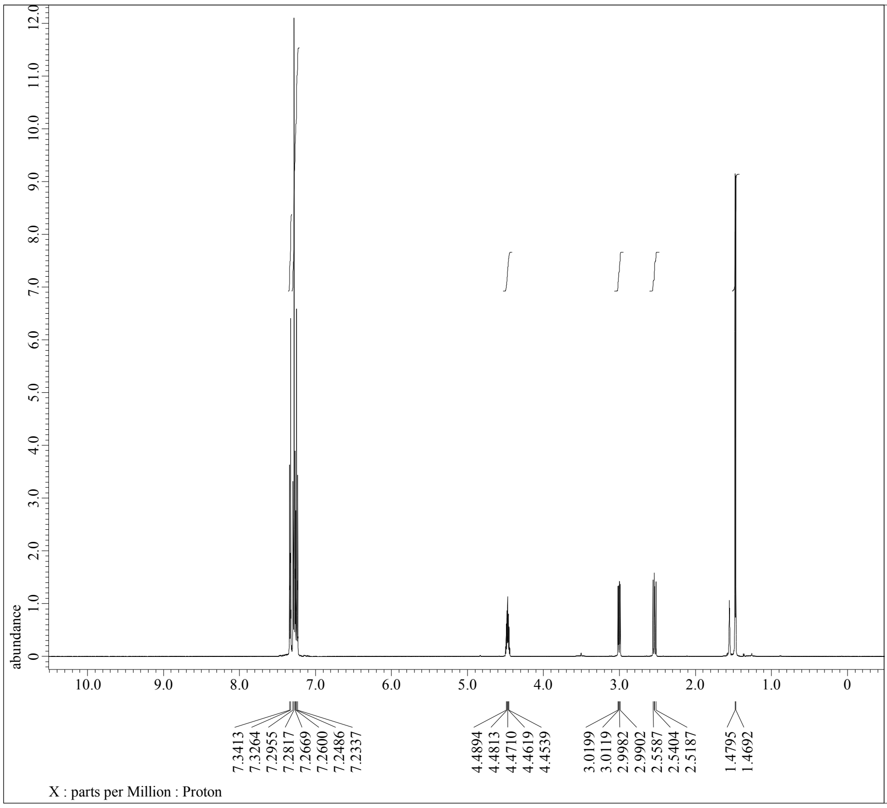
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	34.229	2132017	49.864	33355
2	50.435	2143645	50.136	17742
Totals		4275662	100.000	51097



UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	33.610	2696741	15.095	41830
2	47.219	15168719	84.905	94897
Totals		17865460	100.000	136727

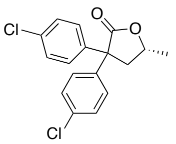


```
Filename          = mn-7-105_Proton-1-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = mn-7-105
Solvent           = CHLOROFORM-D
Creation_Time     =  9-JUL-2014 18:42:43
Revision_Time     = 29-APR-2015 10:06:29
Current_Time      = 29-APR-2015 10:06:43

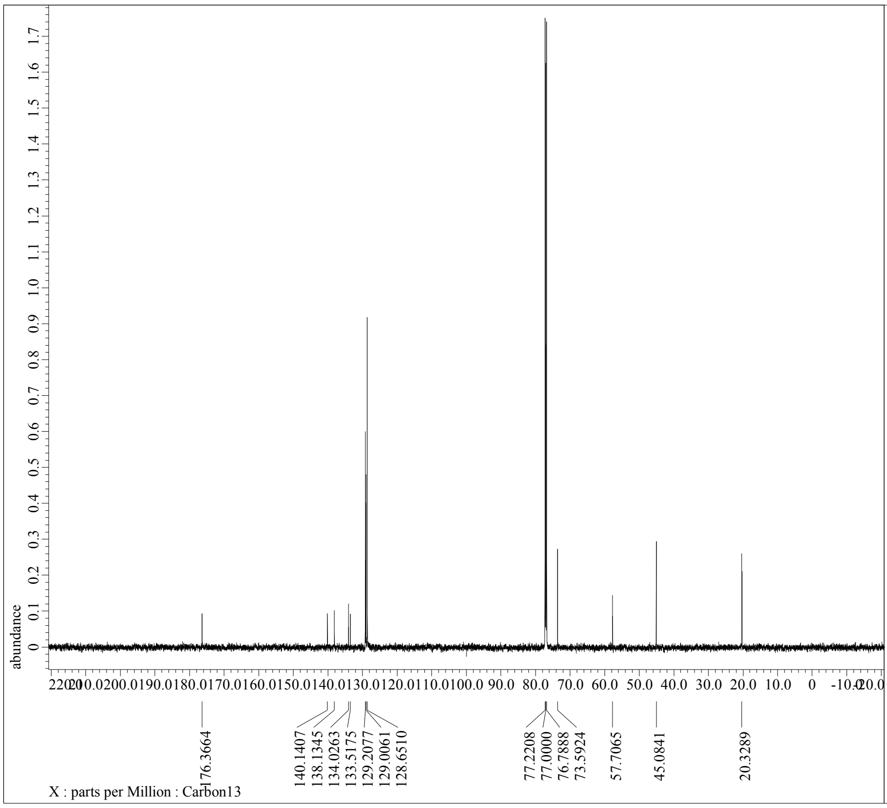
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 1.47062784[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.67998169[Hz]
X_Sweep           = 11.14081996[kHz]
X_Sweep_Clipped   = 8.91265597[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 46
Temp_Get          = 27.4[dC]
X_90_Width        = 13.8[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.9[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



2e

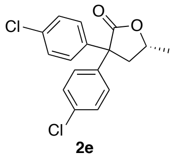


```
Filename          = mn-7-105_Carbon-1-2.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = mn-7-105
Solvent           = CHLOROFORM-D
Creation_Time     =  9-JUL-2014 18:45:30
Revision_Time     = 29-APR-2015 10:07:07
Current_Time      = 29-APR-2015 10:07:24

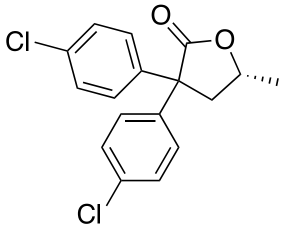
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 0.69730304[s]
X_Domain          = 13C
X_Freq            = 149.40429612[MHz]
X_Offset          = 100.0[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.43409672[Hz]
X_Sweep           = 46.9924812[kHz]
X_Sweep_Clipped   = 37.59398496[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 256
Total_Scans       = 256

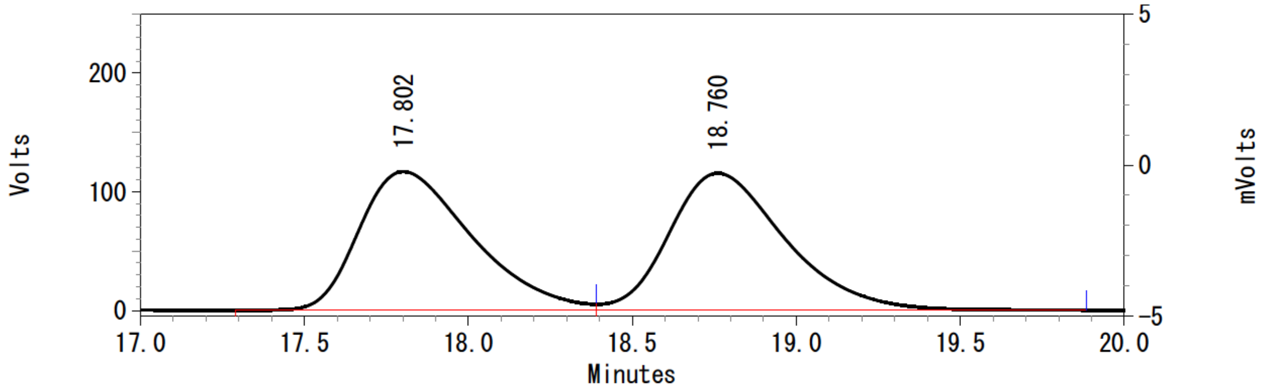
Relaxation_Delay = 2[s]
Recvr_Gain        = 58
Temp_Get          = 28.4[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



2e

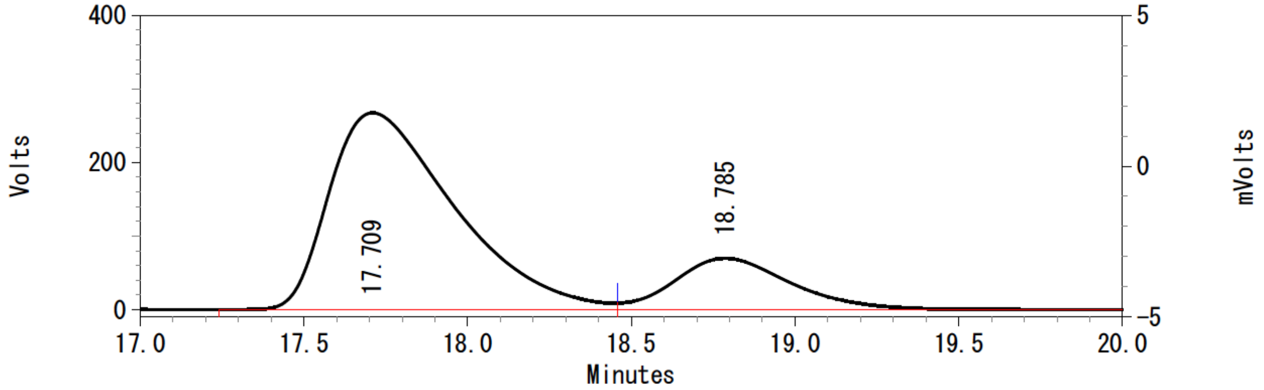


2e



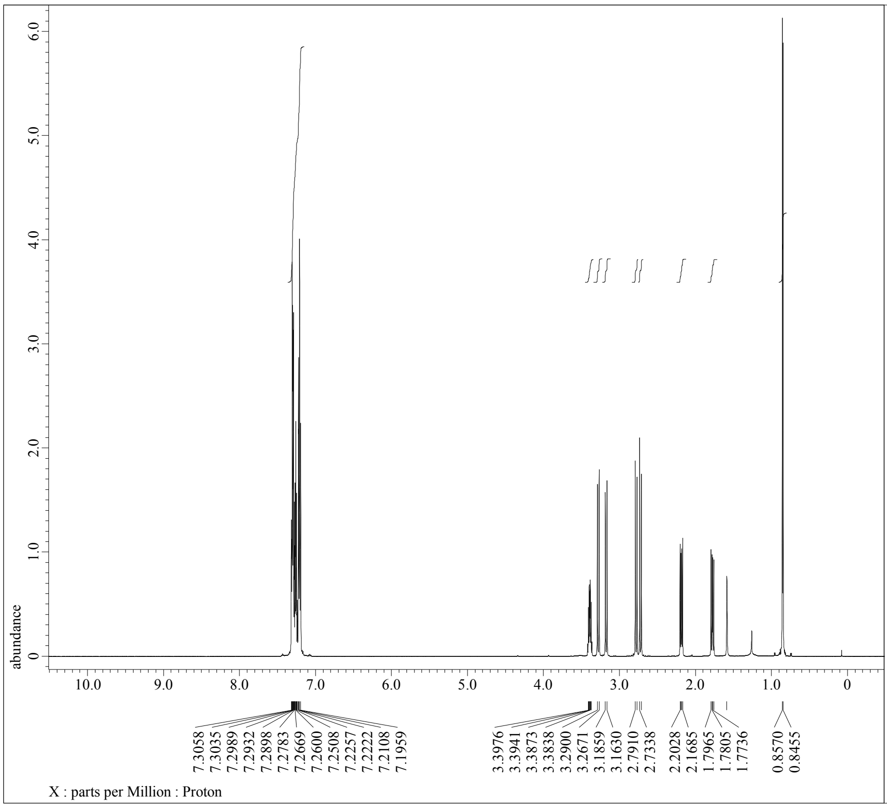
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	17.802	2798375	49.664	116698
2	18.760	2836223	50.336	115293
Totals		5634598	100.000	231991



UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	17.709	7107268	80.463	267344
2	18.785	1725650	19.537	69732
Totals		8832918	100.000	337076

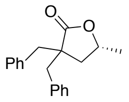


```
Filename           = mn-7-61_Proton-1-2.jdf
Author             = delta
Experiment         = proton.jxp
Sample_Id          = mn-7-61
Solvent            = CHLOROFORM-D
Creation_Time      = 17-JUN-2014 16:42:56
Revision_Time      = 29-APR-2015 10:08:14
Current_Time       = 29-APR-2015 10:08:27

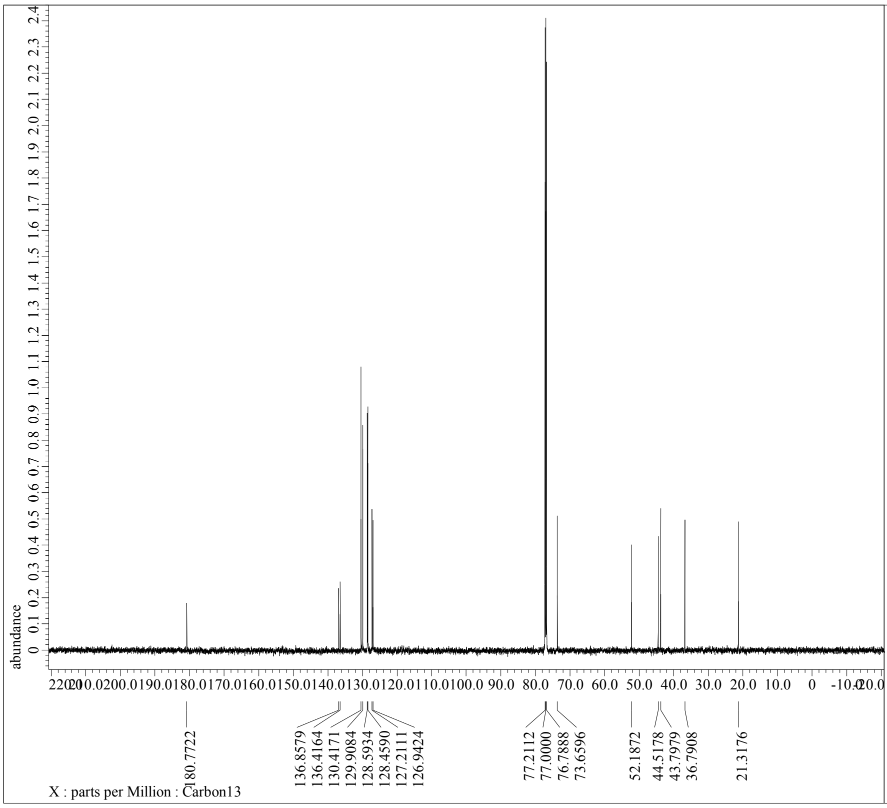
Comment            = single_pulse
Data_Format        = 1D COMPLEX
Dim_Size           = 13107
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECA600
Spectrometer       = DELTA2_NMR

Field_Strength     = 13.95540559[T] (590[MHz])
X_Acq_Duration     = 1.47062784[s]
X_Domain           = 1H
X_Freq             = 594.17058168[MHz]
X_Offset           = 5[ppm]
X_Points           = 16384
X_Prescans         = 1
X_Resolution       = 0.67998169[Hz]
X_Sweep            = 11.14081996[kHz]
X_Sweep_Clipped    = 8.91265597[kHz]
Irr_Domain         = Proton
Irr_Freq           = 594.17058168[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 594.17058168[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay = 5[s]
Recvr_Gain         = 40
Temp_Get           = 26.1[dC]
X_90_Width         = 13.8[us]
X_Acq_Time         = 1.47062784[s]
X_Angle            = 45[deg]
X_Atn              = 3[dB]
X_Pulse            = 6.9[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
Initial_Wait       = 1[s]
Repetition_Time    = 6.47062784[s]
```



2f

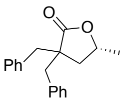


```
Filename           = mn-7-61_Carbon-1-2.jdf
Author             = delta
Experiment         = carbon.jxp
Sample_Id          = mn-7-61
Solvent            = CHLOROFORM-D
Creation_Time      = 17-JUN-2014 16:45:44
Revision_Time      = 29-APR-2015 10:08:46
Current_Time       = 29-APR-2015 10:08:59

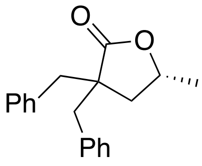
Comment            = single pulse decoupled gat
Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECA600
Spectrometer       = DELTA2_NMR

Field_Strength     = 13.95540559[T] (590[MHz])
X_Acq_Duration     = 0.69730304[s]
X_Domain           = 13C
X_Freq             = 149.40429612[MHz]
X_Offset           = 100.0[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 1.43409672[Hz]
X_Sweep            = 46.9924812[kHz]
X_Sweep_Clipped    = 37.59398496[kHz]
Irr_Domain         = Proton
Irr_Freq           = 594.17058168[MHz]
Irr_Offset         = 5[ppm]
Clipped            = TRUE
Scans              = 256
Total_Scans        = 256

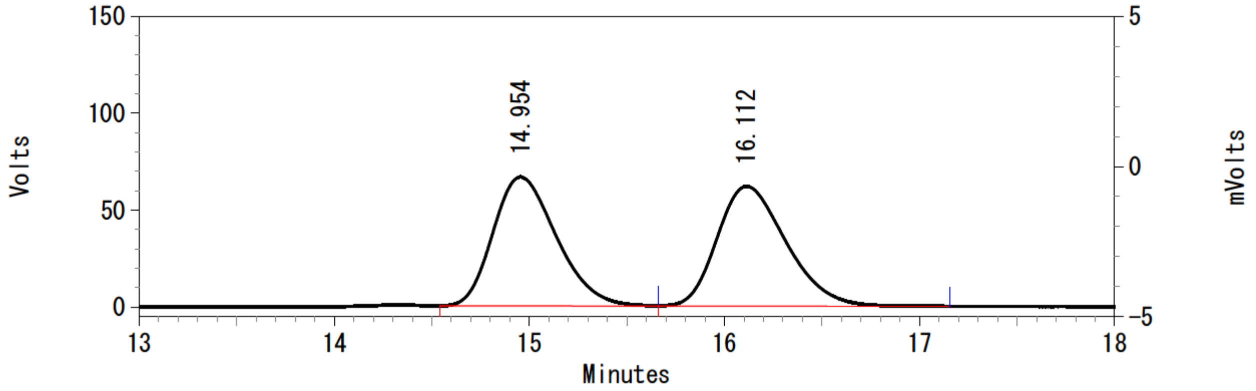
Relaxation_Delay = 2[s]
Recvr_Gain         = 60
Temp_Get           = 27.2[dC]
X_90_Width         = 9[us]
X_Acq_Time         = 0.69730304[s]
X_Angle            = 30[deg]
X_Atn              = 6[dB]
X_Pulse            = 3[us]
Irr_Atn_Dec        = 17.8[dB]
Irr_Atn_Noe        = 17.8[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 76[us]
Decoupling         = TRUE
Initial_Wait       = 1[s]
Noe                = TRUE
Noe_Time           = 2[s]
Repetition_Time    = 2.69730304[s]
```



2f



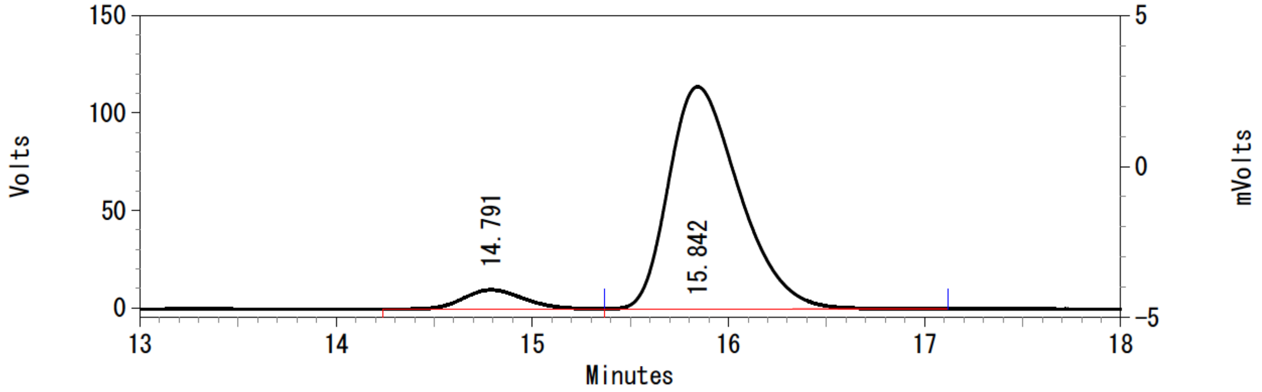
2f



UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	14.954	1503524	49.741	66535
2	16.112	1519182	50.259	61804

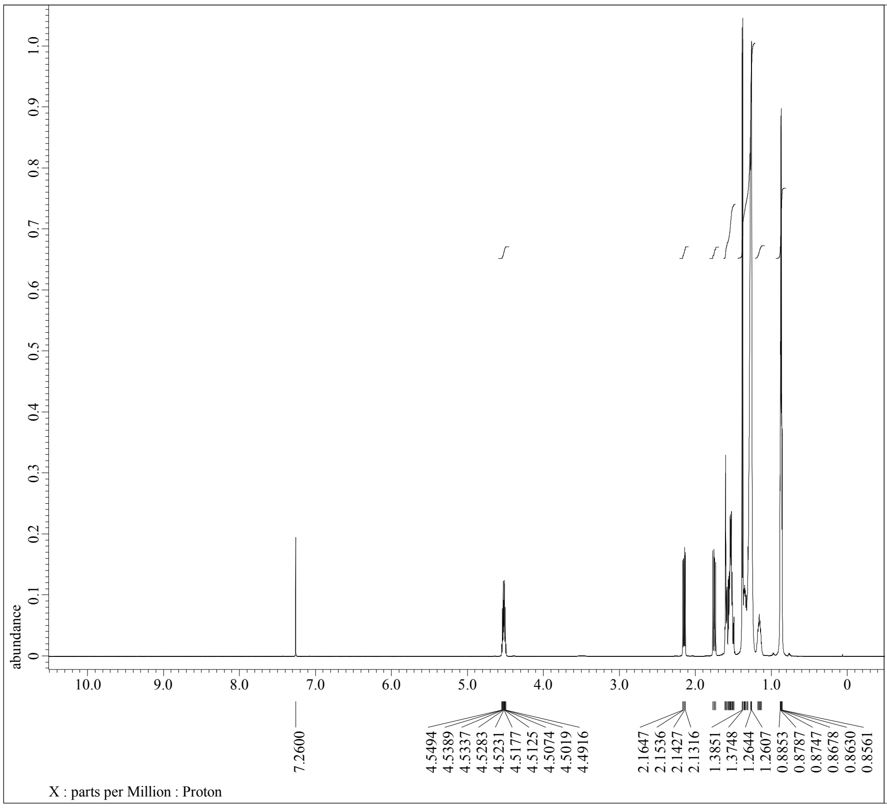
Totals				
		3022706	100.000	128339



UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	14.791	213836	6.976	9859
2	15.842	2851518	93.024	114010

Totals				
		3065354	100.000	123869

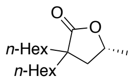


```
Filename          = mn-7-98_Proton-1-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = mn-7-98
Solvent           = CHLOROFORM-D
Creation_Time     =  4-JUL-2014 13:24:51
Revision_Time     = 29-APR-2015 10:09:34
Current_Time      = 29-APR-2015 10:09:48

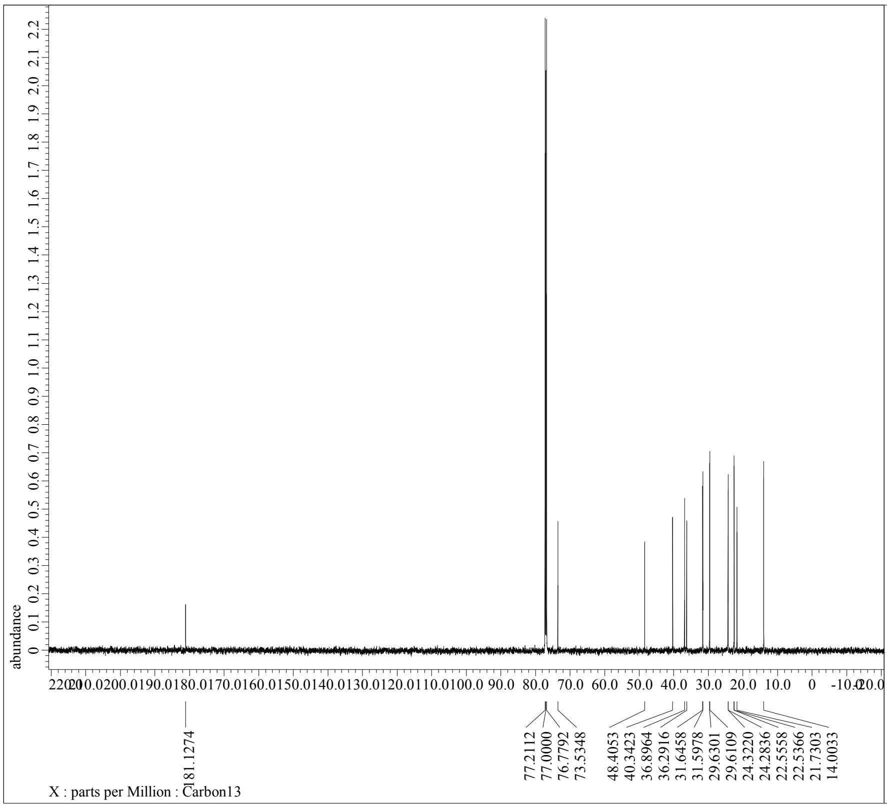
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 52429
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 5.88251136[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 65536
X_Prescans        = 1
X_Resolution      = 0.16999542[Hz]
X_Sweep           = 11.14081996[kHz]
X_Sweep_Clipped   = 8.91265597[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 36
Temp_Get          = 26.7[dC]
X_90_Width        = 13.8[us]
X_Acq_Time        = 5.88251136[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.9[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 10.88251136[s]
```



2g

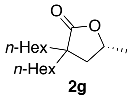


```
Filename          = mn-7-98_Carbon-1-2.jdf
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = mn-7-98
Solvent           = CHLOROFORM-D
Creation_Time     =  4-JUL-2014 13:28:38
Revision_Time     = 29-APR-2015 10:10:09
Current_Time      = 29-APR-2015 10:10:22

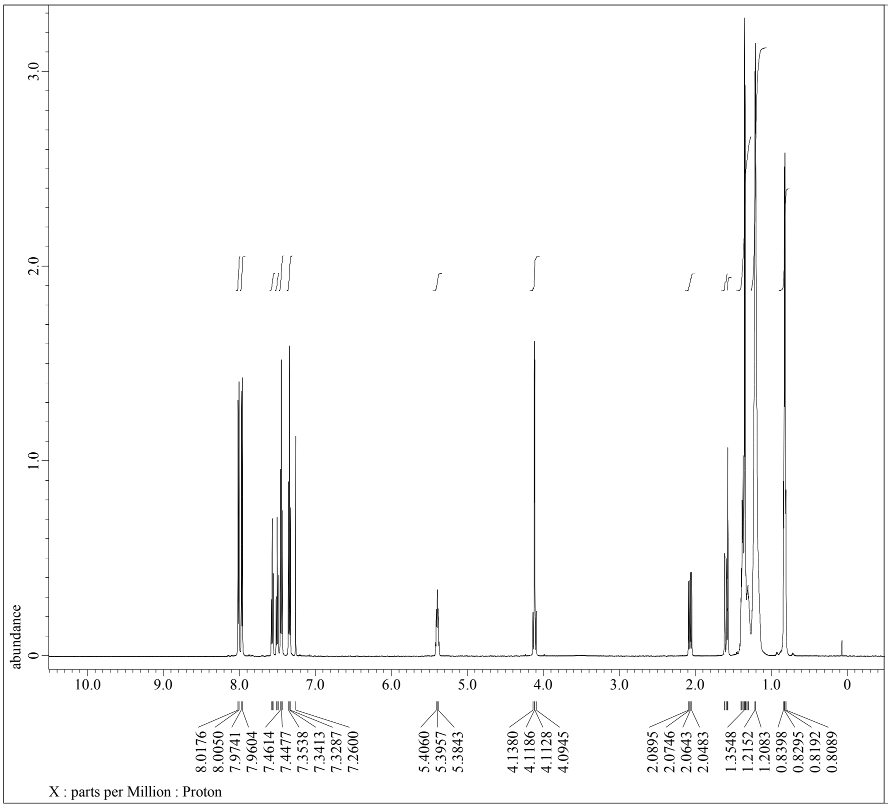
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 0.69730304[s]
X_Domain          = 13C
X_Freq            = 149.40429612[MHz]
X_Offset          = 100.0[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.43409672[Hz]
X_Sweep           = 46.9924812[kHz]
X_Sweep_Clipped   = 37.59398496[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 256
Total_Scans       = 256

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 27.7[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



2g

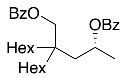


```
Filename         = mn-8-123_Proton-1-4.jdf
Author           = delta
Experiment       = proton.jxp
Sample_Id        = mn-8-123
Solvent          = CHLOROFORM-D
Creation_Time    = 30-OCT-2014 18:28:44
Revision_Time    = 29-APR-2015 10:12:13
Current_Time     = 29-APR-2015 10:12:27

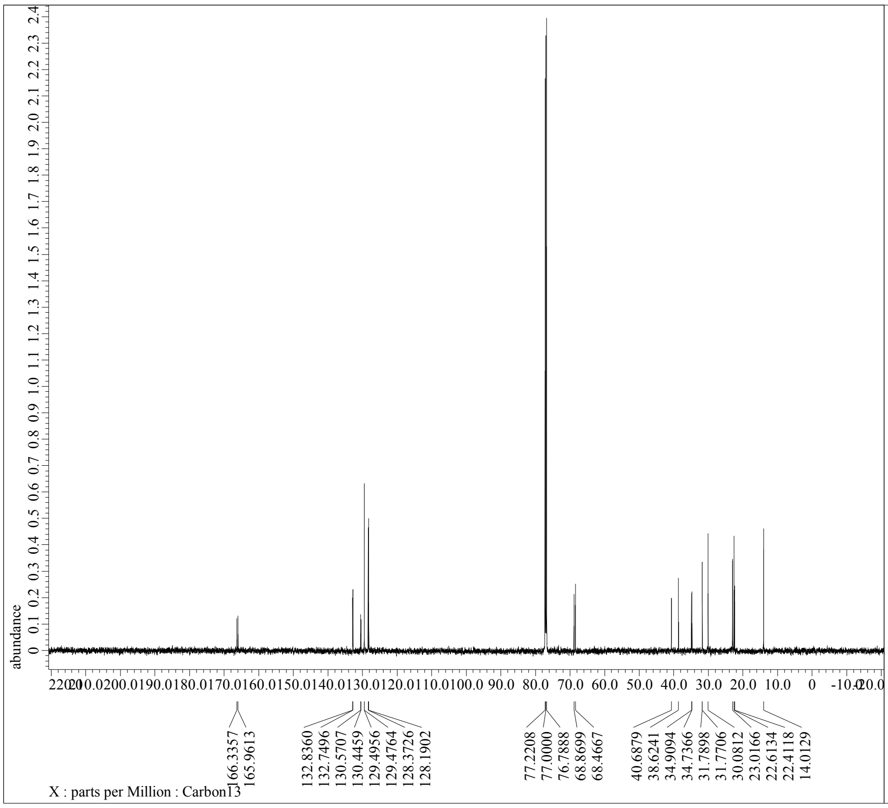
Comment          = single_pulse
Data_Format      = 1D COMPLEX
Dim_Size         = 13107
Dim_Title        = Proton
Dim_Units        = [ppm]
Dimensions       = X
Site             = JNM-ECA600
Spectrometer     = DELTA2_NMR

Field_Strength   = 13.95540559[T] (590[MHz])
X_Acq_Duration   = 1.47062784[s]
X_Domain         = 1H
X_Freq           = 594.17058168[MHz]
X_Offset         = 5[ppm]
X_Points         = 16384
X_Prescans       = 1
X_Resolution     = 0.67998169[Hz]
X_Sweep          = 11.14081996[kHz]
X_Sweep_Clipped  = 8.91265597[kHz]
Irr_Domain       = Proton
Irr_Freq         = 594.17058168[MHz]
Irr_Offset       = 5[ppm]
Tri_Domain       = Proton
Tri_Freq         = 594.17058168[MHz]
Tri_Offset       = 5[ppm]
Clipped          = FALSE
Scans            = 8
Total_Scans      = 8

Relaxation_Delay = 5[s]
Recvr_Gain       = 40
Temp_Get         = 24.7[dC]
X_90_Width       = 13.8[us]
X_Acq_Time       = 1.47062784[s]
X_Angle          = 45[deg]
X_Atn            = 3[dB]
X_Pulse          = 6.9[us]
Irr_Mode         = Off
Tri_Mode         = Off
Dante_Presat     = FALSE
Initial_Wait     = 1[s]
Repetition_Time  = 6.47062784[s]
```



5

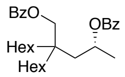


```
Filename         = mn-8-123_Carbon-1-2.jdf
Author           = delta
Experiment       = carbon.jxp
Sample_Id        = mn-8-123
Solvent          = CHLOROFORM-D
Creation_Time    = 30-OCT-2014 18:31:29
Revision_Time    = 29-APR-2015 10:12:50
Current_Time     = 29-APR-2015 10:13:00

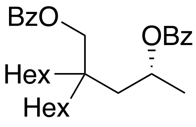
Comment          = single pulse decoupled gat
Data_Format      = 1D COMPLEX
Dim_Size         = 26214
Dim_Title        = Carbon13
Dim_Units        = [ppm]
Dimensions       = X
Site             = JNM-ECA600
Spectrometer     = DELTA2_NMR

Field_Strength   = 13.95540559[T] (590[MHz])
X_Acq_Duration   = 0.69730304[s]
X_Domain         = 13C
X_Freq           = 149.40429612[MHz]
X_Offset         = 100.0[ppm]
X_Points         = 32768
X_Prescans       = 4
X_Resolution     = 1.43409672[Hz]
X_Sweep          = 46.9924812[kHz]
X_Sweep_Clipped  = 37.59398496[kHz]
Irr_Domain       = Proton
Irr_Freq         = 594.17058168[MHz]
Irr_Offset       = 5[ppm]
Clipped          = TRUE
Scans            = 256
Total_Scans      = 256

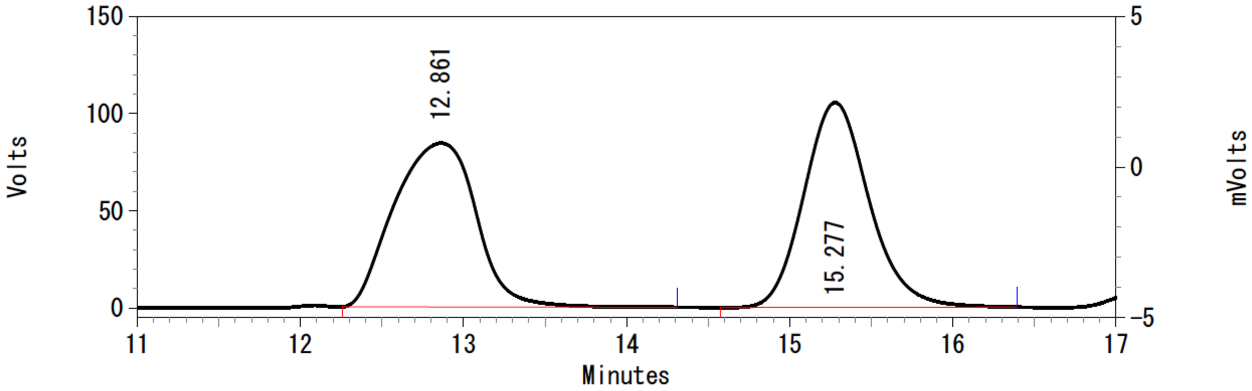
Relaxation_Delay = 2[s]
Recvr_Gain       = 60
Temp_Get         = 25.4[dC]
X_90_Width       = 9[us]
X_Acq_Time       = 0.69730304[s]
X_Angle          = 30[deg]
X_Atn            = 6[dB]
X_Pulse          = 3[us]
Irr_Atn_Dec      = 17.8[dB]
Irr_Atn_Noe      = 17.8[dB]
Irr_Noise        = WALTZ
Irr_Pwidth       = 76[us]
Decoupling       = TRUE
Initial_Wait     = 1[s]
Noe              = TRUE
Noe_Time         = 2[s]
Repetition_Time  = 2.69730304[s]
```



5

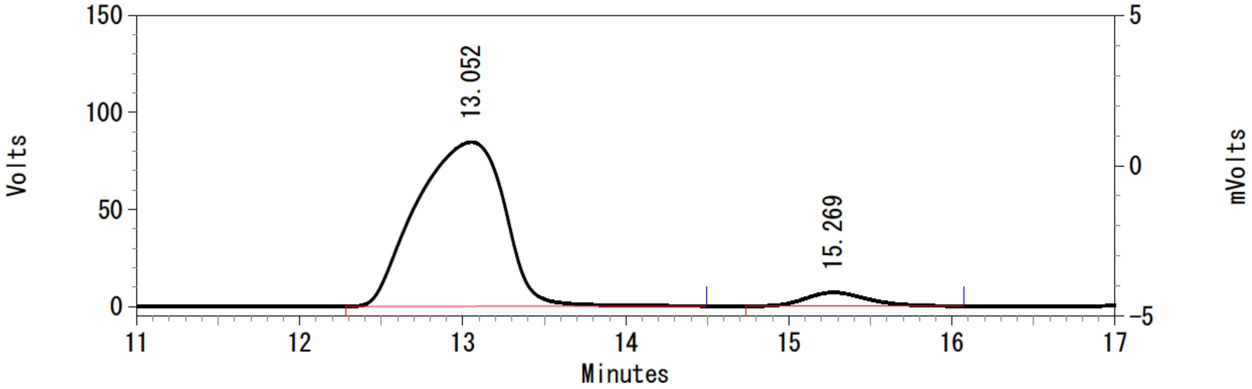


5



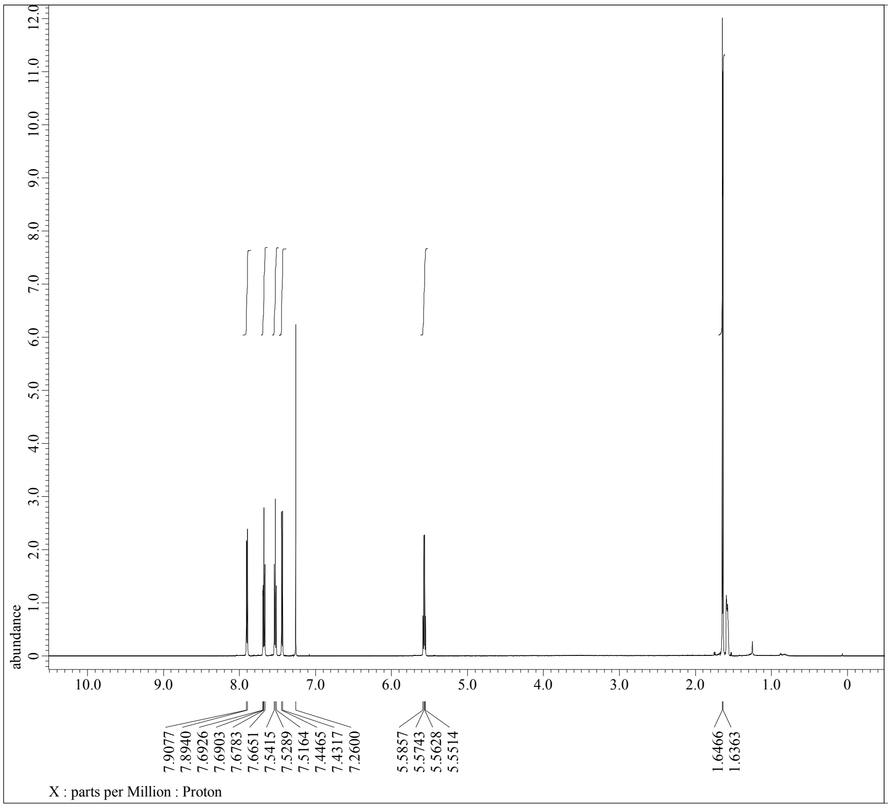
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	12.861	2939090	50.053	84173
2	15.277	2932898	49.947	105366
Totals		5871988	100.000	189539



UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	13.052	3151435	94.460	84473
2	15.269	184821	5.540	7024
Totals		3336256	100.000	91497

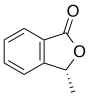


```
Filename          = mn-9-162_Proton-2-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = mn-9-162
Solvent           = CHLOROFORM-D
Creation_Time     = 12-MAY-2015 20:06:05
Revision_Time     = 12-MAY-2015 20:09:35
Current_Time      = 12-MAY-2015 20:09:59

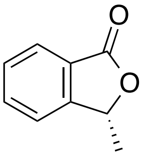
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 1.47062784[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.67998169[Hz]
X_Sweep           = 11.14081996[kHz]
X_Sweep_Clipped   = 8.91265597[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

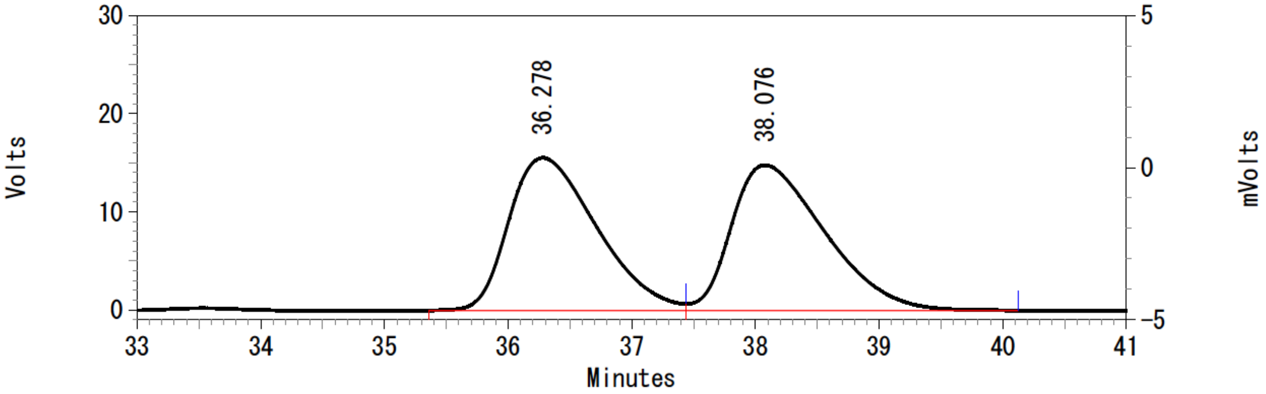
Relaxation_Delay = 5[s]
Recvr_Gain        = 50
Temp_Get          = 23.3[dC]
X_90_Width        = 14[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 7[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



2h



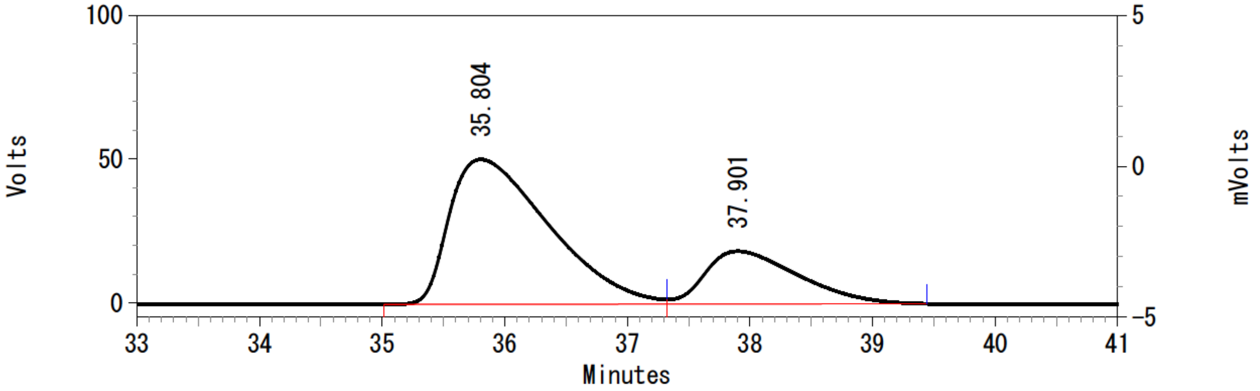
2h



UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	36.278	769594	49.730	15544
2	38.076	777952	50.270	14801

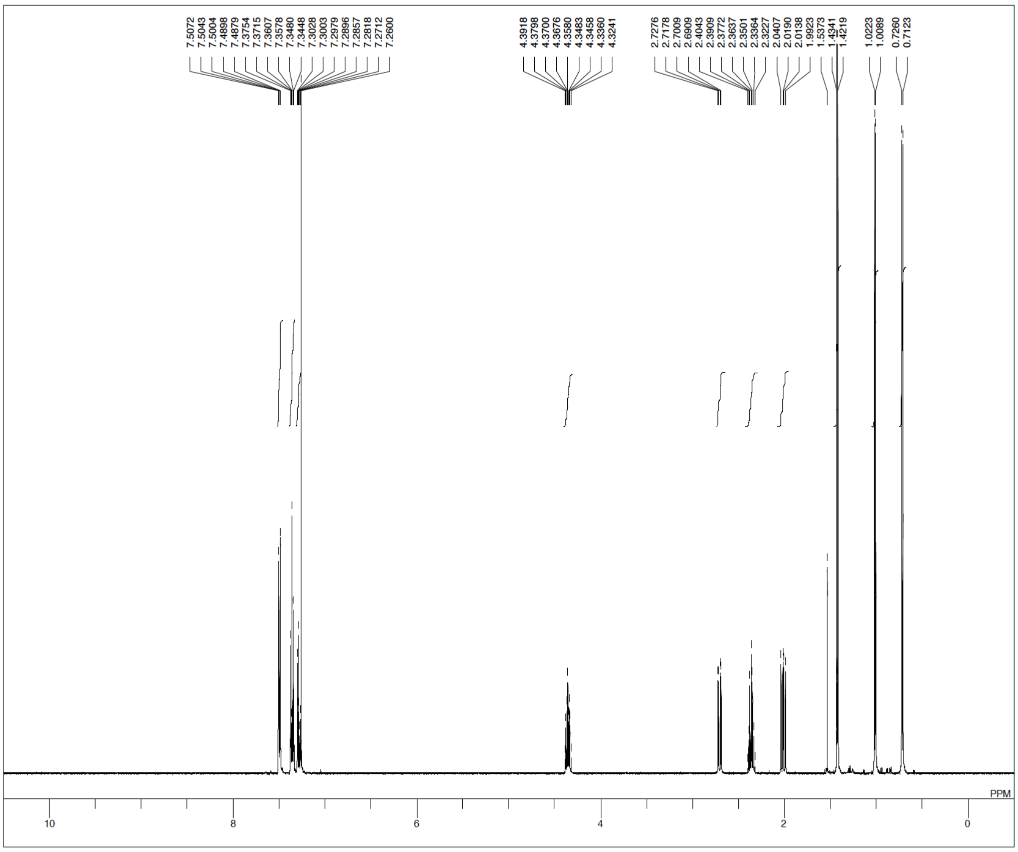
Totals		1547546	100.000	30345



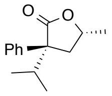
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	35.804	2848252	74.571	50378
2	37.901	971261	25.429	18353

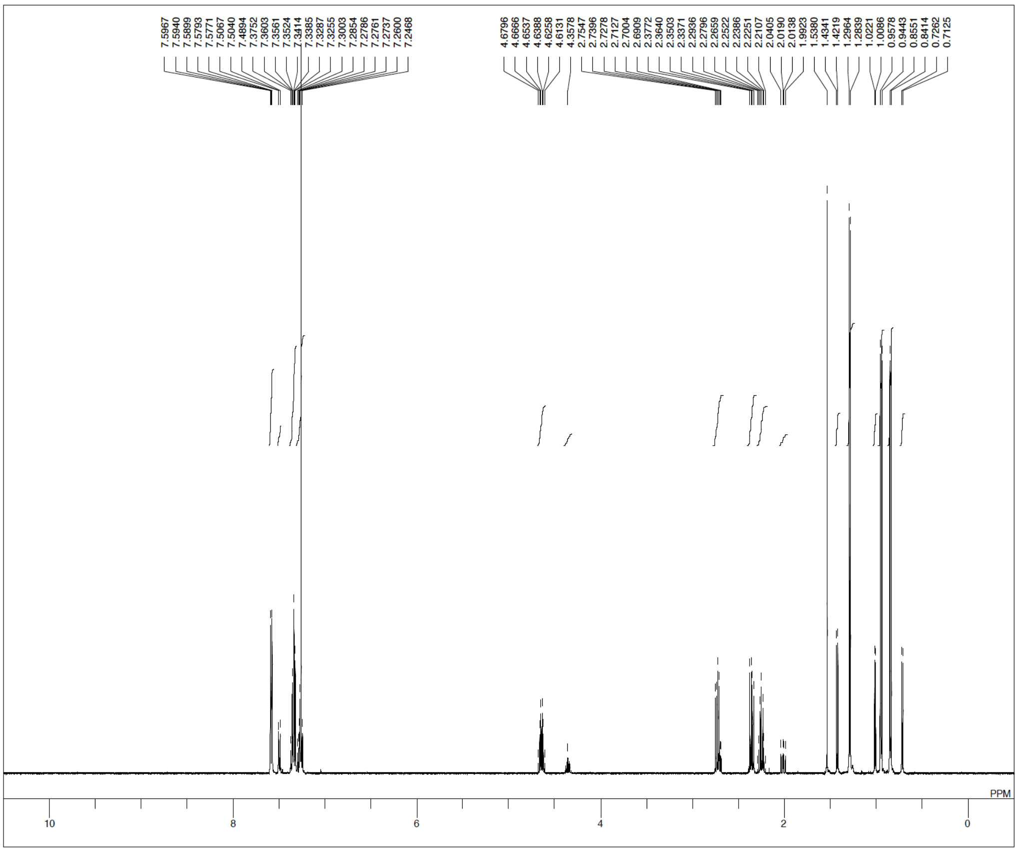
Totals		3819513	100.000	68731



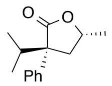
MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	23
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling & nonpresaturation
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-9-75-gpc.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	6
LKSIG	839
CSPED	13 Hz
FILDC	
FILDF	
SLVNT	CDCL3



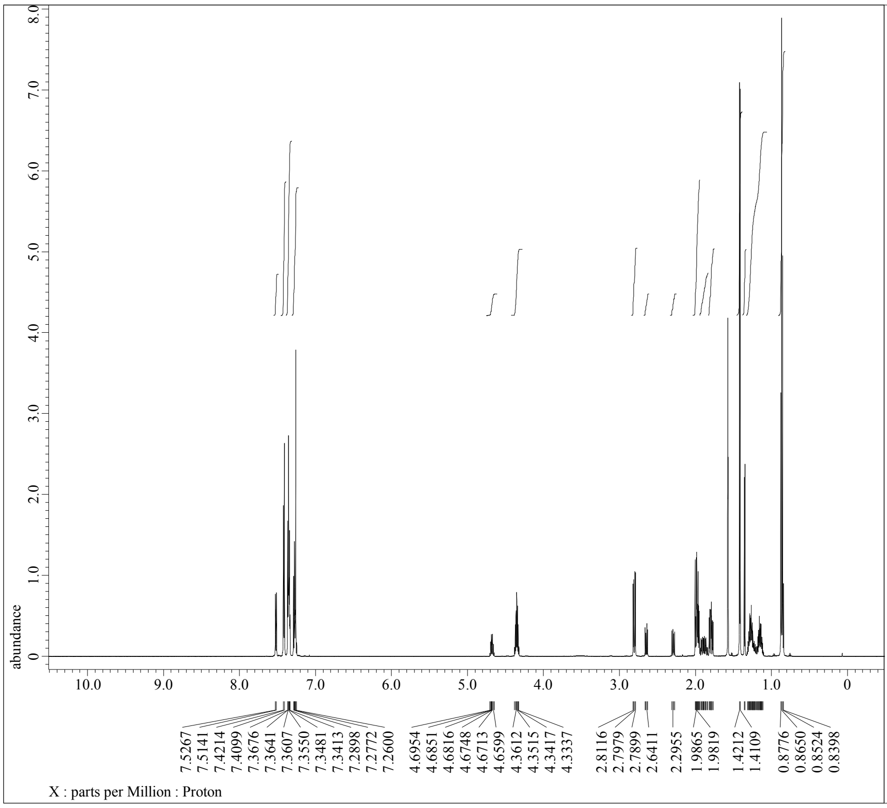
(3*R*,5*R*)-**2i**



MENUF	1H
OBNUC	1H
OFR	499.10 MHz
OBSET	0.00 KHz
OBFIN	128250.00 Hz
PW1	5.50 usec
DEADT	72.30 usec
PREDL	10.00000 msec
IWT	0.5000 sec
POINT	65536
SPO	65536
TIMES	8
DUMMY	0
FREQU	8000.00 Hz
FLT	4000 Hz
DELAY	50.00 usec
ACQTM	8.1920 sec
PD	1.0000 sec
ADBIT	16
RGAIN	23
BF	0.12 Hz
T1	0.00
T2	0.00
T3	90.00
T4	100.00
EXMOD	non
EXPCM	single pulse nondecoupling & nonpresaturation
IRNUC	1H
IFR	499.10 MHz
IRSET	0.00 KHz
IRFIN	128250.00 Hz
IRRPW	50 usec
IRATN	511
DFILE	mn-9-70-gpc.als
SF	th5at
LKSET	0.00 KHz
LKFIN	26934.0 Hz
LKLEV	200
LGAIN	23
LKPHS	6
LKSIG	826
CSPED	14 Hz
FILDC	
FILDF	
SLVNT	CDCL3



(3*S*,5*R*)-**2i**

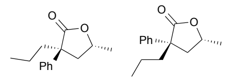


```
Filename          = mn-1-108-gpc_Proton-1-2.jd
Author            = delta
Experiment        = proton.jxp
Sample_Id         = mn-1-108-gpc
Solvent           = CHLOROFORM-D
Creation_Time     = 22-JUN-2015 13:47:12
Revision_Time     = 29-JUN-2015 14:42:37
Current_Time      = 29-JUN-2015 14:42:54

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

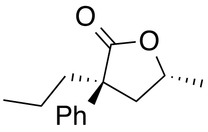
Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 1.47062784[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.67998169[Hz]
X_Sweep           = 11.14081996[kHz]
X_Sweep_Clipped   = 8.91265597[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 46
Temp_Get          = 23.9[dC]
X_90_Width        = 14[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 7[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```

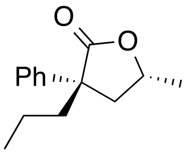


trans-**2j'** (3*S*,5*R*)-**2j'**

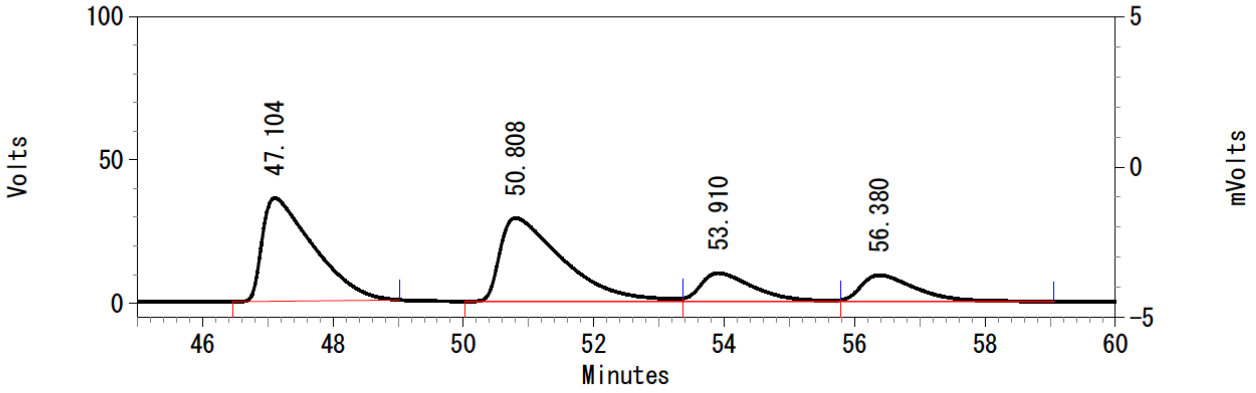
cis-**2j'** (3*R*,5*R*)-**2j'**



trans-**2j'**
(3*S*,5*R*)-**2j'**

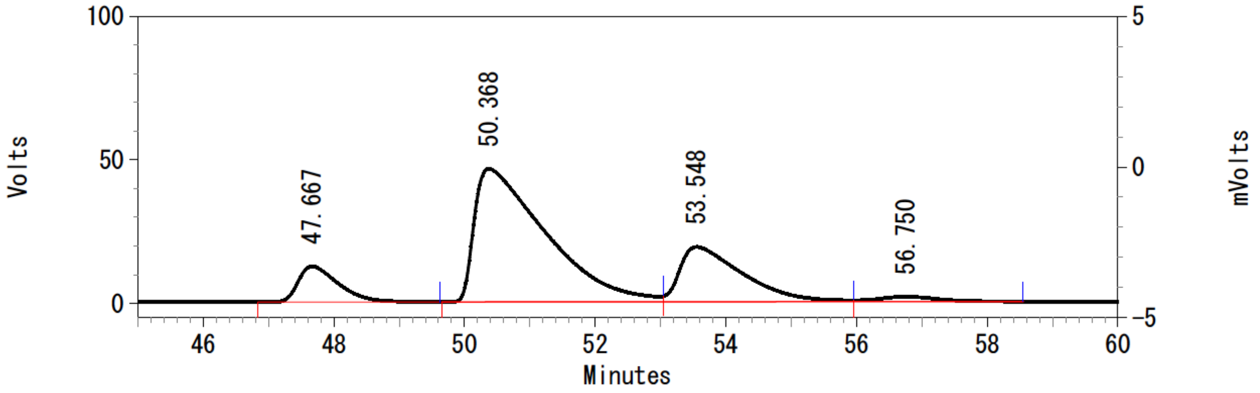


cis-**2j'**
(3*R*,5*R*)-**2j'**



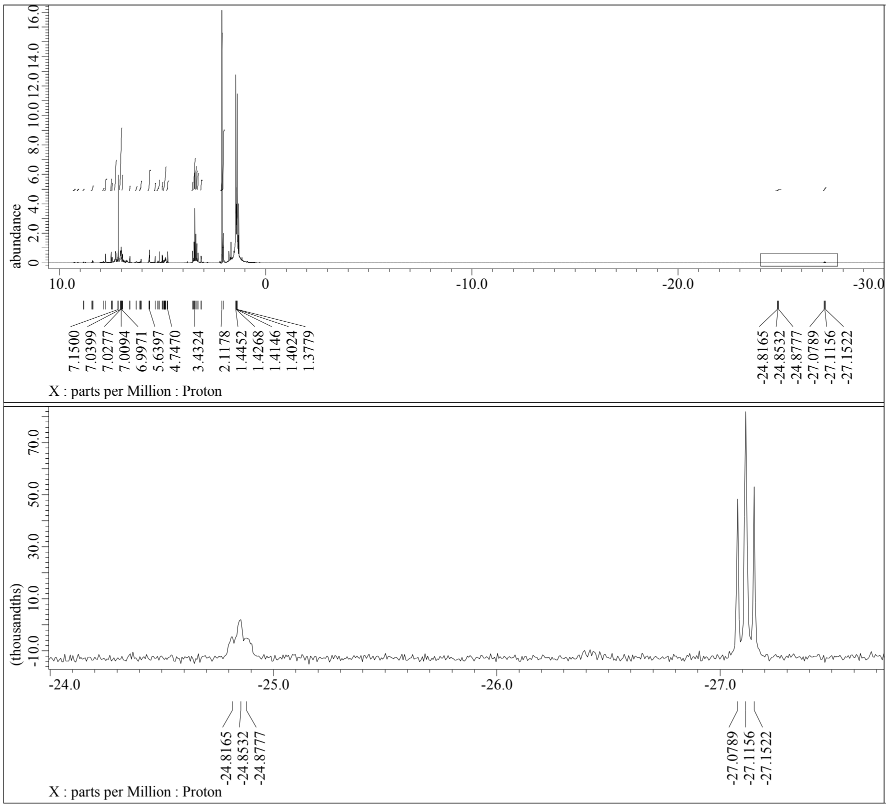
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	47.104	1958028	39.223	35990
2	50.808	1945429	38.971	29076
3	53.910	556055	11.139	9886
4	56.380	532498	10.667	9200
Totals		4992010	100.000	84152



UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	47.667	529509	9.775	12454
2	50.368	3516391	64.917	46338
3	53.548	1267324	23.396	19103
4	56.750	103562	1.912	1701
Totals		5416786	100.000	79596

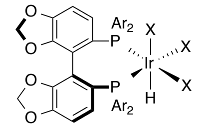


```
Filename          = mn-9-47-24h_Proton-2-2.jdf
Author            = delta
Experiment        = proton.jxp
Sample_Id         = mn-9-47-24h
Solvent           = BENZENE-D6
Creation_Time     = 12-JAN-2015 13:13:28
Revision_Time     = 25-MAY-2015 17:39:07
Current_Time      = 25-MAY-2015 17:40:12

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 0.2752512[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 3.63304501[Hz]
X_Sweep           = 59.52380952[kHz]
X_Sweep_Clipped   = 47.61904762[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 36
Temp_Get          = 23.6[dC]
X_90_Width        = 13.8[us]
X_Acq_Time        = 0.2752512[s]
X_Angle           = 45[deg]
X_Atn             = 3[dB]
X_Pulse           = 6.9[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 5.2752512[s]
```



X = Cl, OCOR

Ar = 3,5-(t-Bu)$_2$-4-MeOC$_6$H$_2$