Electronic Supplementary Material (ESI) for Chemical Communications

Supplementary Information

Asymmetric synthesis of *gem*-diaryl substituted cyclic sulfamidates and sulfamides by rhodium-catalyzed arylation of cyclic ketimines

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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 JEOL JNM ECA-600 spectrometer (600 MHz for ^{1}H, 150 MHz for ^{13}C). Chemical shifts are reported in δ (ppm) referenced to the residual peak of $CDCl_3$ (δ 7.26) 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, m: multiplet, br: broad. Optical rotations were measured on a JASCO P-2200 polarimeter. High-resolution mass spectra were obtained with a Bruker micrOTOF spectrometer. Flash column chromatography was performed with Silica Gel 60 N (spherical, neutral) (Cica-Reagent). Preparative thin-layer chromatography was performed with Silica Gel 60 PF_{254} (Merck). Alumina (activated 200) for column chromatography was purchased from Nacalai Tesque.

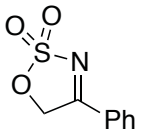
2. Materials

1,4-Dioxane and toluene were purified by passing through a neutral alumina column under N_2. *tert*-Amyl alcohol was dried over 4 Å molecular sieves under N_2. Rhodium complexes, $[RhCl(cod)]_2$ (CAS: 12092-47-6), $[Rh(OH)(cod)]_2$ (CAS: 73468-85-6),[1] $[Rh(OH)((R)\text{-binap})]_2$ (CAS: 805323-12-0),[2] $[Rh(OH)((R,R)\text{-Bn-tfb*})]_2$ (CAS: 1204591-09-2 for the (*S,S*)-complex),[3] $[Rh(OH)((R,R)\text{-Ph-tfb*})]_2$ (CAS: 1235989-05-5), [4] and $[Rh(OH)((S,S)\text{-Fc-tfb*})]_2$ [5] (CAS: 1204591-10-5) were prepared according to the reported procedures.

A diene ligand **L1**[6] (CAS: 1365284-85-0) were prepared according to the reported procedures. Ketimines **1a** (CAS: 1025506-03-9), **1b** (CAS: 4136-21-4), **1c** (CAS: 1025506-04-0), **1d** (CAS: 1244055-98-8), **1e** (CAS: 1204515-20-7), **1f** (CAS: 1280492-14-9), **1i** (CAS: 881652-47-7), and **1k** (CAS: 87216-60-2) were prepared according to the reported procedures.[7] Ketimines **1g**, **1h**, and **1j** were prepared as shown below. All other chemicals were purchased from commercial suppliers and used as received.

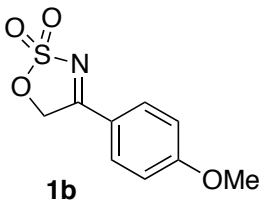
1 R. Uson, L. A. Oro and J. A. Cabeza, *Inorg. Synth.,* 1985, **23**, 129.
2 T. Hayashi, M. Takahashi, Y. Takaya and M. Ogasawara, *J. Am. Chem. Soc.,* 2002, **124**, 5052.
3 T. Nishimura, J. Wang, M. Nagaosa, K. Okamoto, R. Shintani, F. Kwong, W. Yu, A. S. C. Chan and T. Hayashi, *J. Am. Chem. Soc.,* 2010, **132**, 464.
4 R. Shintani, M. Takeda, T. Nishimura and T. Hayashi, *Angew. Chem. Int. Ed.,* 2010, **49**, 3969.
5 T. Nishimura, A. Kasai, M. Nagaosa and T. Hayashi, *Chem. Commun.*, 2011, **47**, 10488.
6 T. Nishimura, A. Noishiki, G. C. Tsui and T. Hayashi, *J. Am. Chem. Soc.*, 2012, **134**, 5056
7 *a*) S. Kang, J. Han, E. S. Lee, E. B. Choi and H.-H. Lee, *Org. Lett.,* 2010, **12**, 4184; *b*) S. A. Lee, S. H. Kwak and K.-I. Lee, *Chem. Commun.*, 2011, **47**, 2372; *c*) D. Freitag and P. Metz, *Tetrahedron*, 2006, **62**, 1799; *d*) A. M. Pietruszkiewicz, J. J. Baldwin, W. A. Bolhofer, J. M. Hoffman and W. C. Lumma, *J. Heterocycl. Chem.,* 1983, **20**, 821.

3. Characterization of ketimines



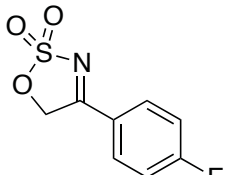
1a

Compound 1a (CAS: 1025506-03-9). ^{1}H NMR ($CDCl_3$) δ 5.59 (s, 2H), 7.58 (t, J = 7.8 Hz, 2H), 7.74 (t, J = 7.8 Hz, 1H), 7.91 (d, J = 7.8 Hz, 2H).



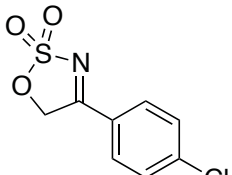
1b

Compound 1b (CAS: 4136-21-4). ^{1}H NMR ($CDCl_3$) δ 3.93 (s, 3H), 5.54 (s, 2H), 7.04 (d, J = 8.9 Hz, 2H), 7.87 (d, J = 8.9 Hz, 2H).



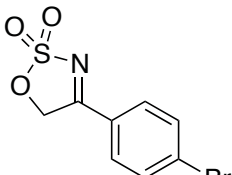
1c

Compound 1c (CAS: 1025506-04-0). ^{1}H NMR ($CDCl_3$) δ 5.56 (s, 2H), 7.27–7.31 (m, 2H), 7.94–7.98 (m, 2H).



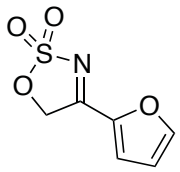
1d

Compound 1d (CAS: 1244055-98-8). ^{1}H NMR ($CDCl_3$) δ 5.55 (s, 2H), 7.58 (d, J = 8.1 Hz, 2H), 7.86 (d, J = 8.1 Hz, 2H).



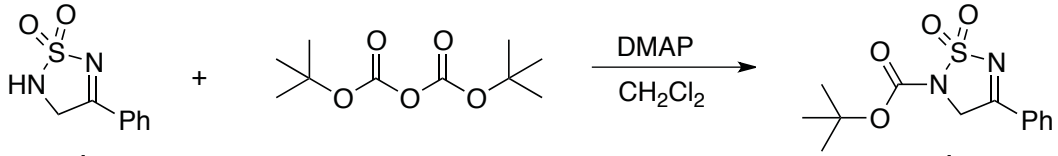
1e

Compound 1e (CAS: 1204515-20-7). ^{1}H NMR ($CDCl_3$) δ 5.55 (s, 2H), 7.74 (d, J = 8.9 Hz, 2H), 7.78 (d, J = 8.9 Hz, 2H).



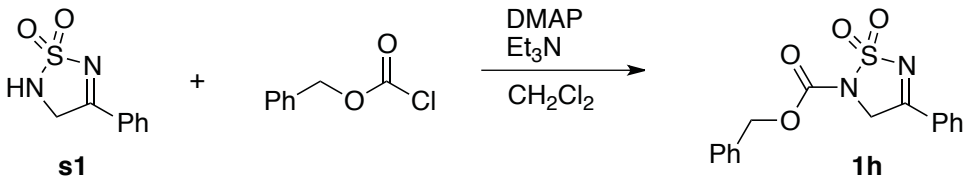
1f

Compound 1f (CAS: 1280492-14-9). ^{1}H NMR ($CDCl_3$) δ 5.48 (s, 2H), 6.77 (dd, J = 3.4, 1.4 Hz, 1H), 7.56 (d, J = 3.4 Hz, 1H), 7.82 (d, J = 1.4 Hz, 1H).



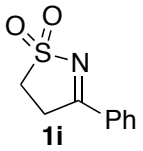
s1 **1g**

Compound 1g. To a solution of **s1**[7b] (CAS: 70404-58-9; 589 mg, 3.0 mmol) and di-*tert*-butyl dicarbonate (2.42 g, 6.0 mmol) in dichloromethane (6 mL) was added 4-dimethylaminopyridine (DMAP; 73 mg, 0.60 mmol), and the mixture was stirred at room temperature for 0.5 h. The mixture was concentrated on a rotary evaporator, and the residue was subjected to flash column chromatography on silica gel with hexane/ethyl acetate (4:1) to give **1g** (678 mg, 2.3 mmol, 76% yield). ^{1}H NMR ($CDCl_3$) δ 1.59 (s, 9H), 5.05 (s, 2H), 7.56 (t, J = 8.0 Hz, 2H), 7.71 (t, J = 8.0 Hz, 1H), 7.96 (d, J = 8.0 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 28.0, 52.9, 85.4, 128.6, 128.7, 129.5, 135.4, 148.8, 171.8. HRMS (ESI) calcd for $C_{13}H_{16}N_2NaO_4S$ $(M+Na)^+$ 319.0723, found 319.0724.



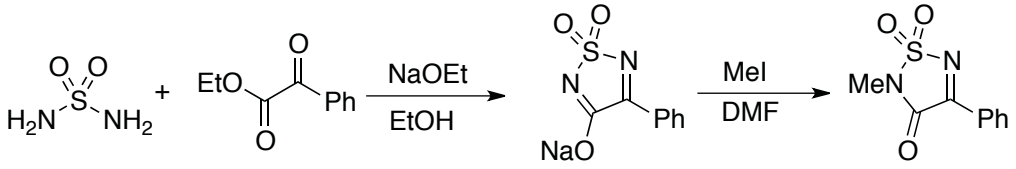
s1 **1h**

Compound 1h. To a solution of **s1**[7b] (462 mg, 2.4 mmol), 4-dimethylaminopyridine (DMAP; 24 mg, 0.2 mmol), and triethylamine (0.66 mL, 4.7 mmol) in dichloromethane (5 mL) was added dropwise benzyl chloroformate (604 mg, 3.5 mmol) at 0 °C, and the mixture was stirred for 1 h. The mixture was passed through a short column of silica gel with ethyl acetate and concentrated on a rotary evaporator. The residue was subjected to flash column chromatography on silica gel with hexane/chloroform/ethyl acetate (5:5:1) to give **1h** (711 mg, 2.2 mmol, 91% yield). ^{1}H NMR ($CDCl_3$) δ 5.10 (s, 2H), 5.37 (s, 2H), 7.33–7.42 (m, 3H), 7.46 (d, J = 7.5 Hz, 2H), 7.56 (t, J = 7.5 Hz, 2H), 7.71 (t, J = 7.5 Hz, 1H), 7.96 (d, J = 7.5 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 35.2, 69.4, 128.0, 128.4, 128.6, 128.7, 128.8, 129.5, 134.5, 135.7, 149.9, 171.9. HRMS (ESI) calcd for $C_{16}H_{14}N_2NaO_4S$ $(M+Na)^+$ 353.0566, found 353.0570.



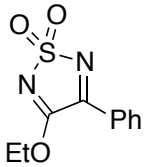
1i

Compound 1i (CAS: 881652-47-7). ^{1}H NMR ($CDCl_3$) δ 3.41–3.45 (m, 2H), 3.64–3.69 (m, 2H), 7.52 (t, *J* = 8.0 Hz, 2H), 7.63–7.67 (m, 1H), 8.00–8.03 (m, 2H).



s2 **1j**

Compound 1j. To a solution of sulfamide (1.92 g, 20 mmol) in ethanol (30 mL) was slowly added sodium ethoxide (1.36 g, 20 mmol) in ethanol (5 mL). The suspension was stirred at room temperature for 15 min and then ethyl phenylglyoxylate (3.56g, 20 mmol) in ethanol (15 mL) was added. After stirring for 15 min, the mixture was refluxed overnight and concentrated on a rotary evaporator. The residue was suspended in diethyl ether for 0.5 h and the resulting white solid was filtered, washed with diethyl ether, and dried under vacuum to give **s2** (3.95 g), which was used for the next reaction without further purification.[8] To a suspension of **s2** (1.16 g, 5.0 mmol as a pure **s1**) in DMF (10 mL) was added methyl iodide (0.47 mL, 7.5 mmol), and the mixture was stirred at room temperature for 17 h. H_2O was added to the mixture and it was extracted with ethyl acetate. The organic extracts were washed with H_2O, brine, dried over $MgSO_4$, filtered, and concentrated on a rotary evaporator. The residue was subjected to column chromatography on silica gel with hexane/$CHCl_3$/ethyl acetate (20:5:1) to give **1j** (301 mg, 1.34 mmol, 27% yield). **Compound 1j**: ^{1}H NMR ($CDCl_3$) δ 3.32 (s, 3H), 7.57 (t, *J* = 7.5 Hz, 2H), 7.75 (t, *J* = 7.5 Hz, 1H), 8.57 (d, *J* = 7.5 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 26.2, 126.8, 129.4, 132.2, 136.3, 156.0, 164.6. HRMS (ESI) calcd for $C_9H_8N_2NaO_3S$ $(M+Na)^+$ 247.0148, found 247.0147.



1k

Compound 1k (CAS: 87216-60-2). ^{1}H NMR ($CDCl_3$) δ 1.60 (t, *J* = 7.5 Hz, 3H), 4.73 (q, *J* = 7.5 Hz, 2H), 7.55 (t, *J* = 7.5 Hz, 2H), 7.71 (t, *J* = 7.5 Hz, 1H), 8.25 (d, *J* = 7.5 Hz, 2H).

4. A general procedure for Table 1

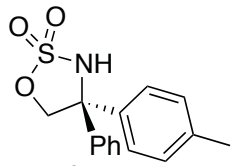
8 M. S. Pietruszkiewicz, J. J. Baldwin, W. A. Bolhofer, J. M. Hoffman, W. C. Lumma, *J. Heterocycl. Chem.*, 1983, **20**, 821.

A rhodium catalyst (0.0050 mmol of Rh), 4-phenyl-5*H*-[1,2,3]-oxathiazole 2,2-dioxide (**1a**) (19.7 mg, 0.10 mmol), and *p*-tolylboroxine (35.4 mg, 0.10 mmol) or *p*-tolylboronate (**2m**) (61.2 mg, 0.30 mmol) were placed in a Schlenk tube under nitrogen. A solvent was added and the mixture was stirred at 80 °C for 20 h. The mixture was passed through a short column of alumina with ethyl acetate as eluent. The solvent was removed on a rotary evaporator and the residue was subjected to preparative TLC on silica gel with ethyl acetate/hexane (1:2) to give **3am**. The ee of **3am** was determined by chiral HPLC.

5. A general procedure for Table 2

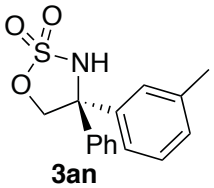
[Rh(OH)((*S,S*)-Fc-tfb*)]$_2$] (7.1 mg, 0.010 mmol of Rh), 4-aryl-5*H*-[1,2,3]-oxathiazole 2,2-dioxide **1** (0.20 mmol), and arylboronate **2** (0.60 mmol) were placed in a Schlenk tube under nitrogen. *tert*-Amyl alcohol (0.8 mL) and toluene (0.8 mL) were added and the mixture was stirred at 80 °C for 20 h. The mixture was passed through a short column of alumina with ethyl acetate as eluent. The solvent was removed on a rotary evaporator and the residue was subjected to preparative TLC on silica gel with ethyl acetate/hexane to give **3**.

6. Characterization of the products



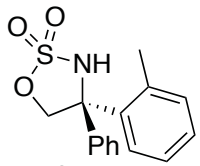
3am

Compound 3am. The ee was measured by HPLC (Chiralpak IC, hexane/$CHCl_3$/EtOH = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 12.7 min (*S*), t_2 = 13.7 min (*R*)); $[\alpha]^{20}_D$ +15 (*c* 1.00, $CHCl_3$) for 93% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 2.35 (s, 3H), 4.78 (br s, 1H), 5.06 (d, *J* = 9.5 Hz, 1H), 5.10 (d, *J* = 9.5 Hz, 1H), 7.20 (d, *J* = 8.2 Hz, 2H), 7.24 (d, *J* = 8.2 Hz, 2H), 7.33–7.42 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 21.0, 70.8, 79.2, 126.2, 126.3, 128.6, 129.1, 129.8, 138.1, 138.7, 141.1. HRMS (ESI) calcd for $C_{15}H_{15}NNaO_3S$ $(M+Na)^+$ 312.0665, found 312.0671.



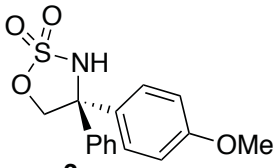
3an

Compound 3an. The ee was measured by HPLC (Chiralpak IB, hexane/$CHCl_3$/EtOH = 12:2:1, flow 0.6 mL/min, 254 nm, t_1 = 11.1 min (*S*), t_2 = 11.8 min (*R*)); $[\alpha]^{20}_D$ +9 (*c* 1.01, $CHCl_3$) for 92% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 2.36 (s, 3H), 4.84 (br s, 1H), 5.07 (d, *J* = 9.2 Hz, 1H), 5.10 (d, *J* = 9.2 Hz, 1H), 7.13 (d, *J* = 8.1 Hz, 1H), 7.16 (s, 1H), 7.17 (d, *J* = 8.1 Hz, 1H), 7.28 (t, *J* = 8.1 Hz, 1H), 7.33–7.42 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 21.7, 71.1, 79.2, 123.5, 126.4, 126.9, 128.7, 129.1, 129.2, 129.6, 139.2, 141.1, 141.3. HRMS (ESI) calcd for $C_{15}H_{15}NNaO_3S$ $(M+Na)^+$ 312.0665, found 312.0662.



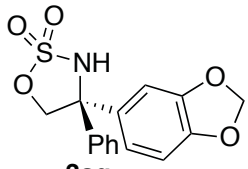
3ao

Compound 3ao. The ee was measured by HPLC (Chiralpak IC, hexane/$CHCl_3$/EtOH = 12:2:1, flow 0.6 mL/min, 254 nm, t_1 = 18.6 min (*R*), t_2 = 20.3 min (*S*)); $[\alpha]^{20}_D$ +27 (*c* 1.00, $CHCl_3$) for 98% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 1.91 (s, 3H), 4.71 (br s, 1H), 4.99 (d, *J* = 9.5 Hz, 1H), 5.20 (d, *J* = 9.5 Hz, 1H), 7.18 (d, *J* = 6.8 Hz, 1H), 7.30–7.40 (m, 7H), 7.76 (d, *J* = 7.5 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 21.0, 71.2, 78.5, 126.1, 126.3, 126.4, 128.7, 128.9, 129.2, 132.8, 135.2, 138.6, 141.4. HRMS (ESI) calcd for $C_{15}H_{15}NNaO_3S$ $(M+Na)^+$ 312.0665, found 312.0661.



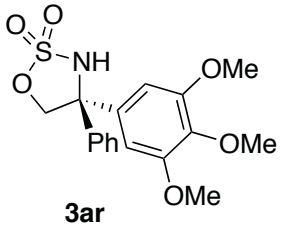
3ap

Compound 3ap. The ee was measured by HPLC (Chiralpak IC, hexane/$CHCl_3$/EtOH = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 15.0 min (*S*), t_2 = 17.3 min (*R*)); $[\alpha]^{20}_D$ +17 (*c* 1.01, $CHCl_3$) for 92% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 3.81 (s, 3H), 4.73 (br s, 1H), 5.03 (d, *J* = 9.2 Hz, 1H), 5.09 (d, *J* = 9.2 Hz, 1H), 6.90 (d, *J* = 8.9 Hz, 2H), 7.26 (d, *J* = 8.9 Hz, 2H), 7.34–7.42 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 55.3, 70.7, 79.3, 114.4, 126.3, 127.7, 128.6, 129.0, 133.0, 141.2, 159.7. HRMS (ESI) calcd for $C_{15}H_{15}NNaO_4S$ $(M+Na)^+$ 328.0614, found 328.0607.



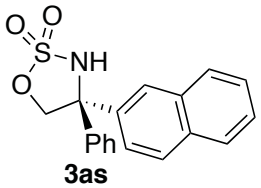
3aq

Compound 3aq. The ee was measured by HPLC (Chiralpak IC, hexane/$CHCl_3$/EtOH = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 16.2 min (*S*), t_2 = 18.2 min (*R*)); $[\alpha]^{20}_D$ +3 (*c* 1.01, $CHCl_3$) for 93% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 4.73 (br s, 1H), 5.03 (d, *J* = 9.2 Hz, 1H), 5.05 (d, *J* = 9.2 Hz, 1H), 5.98 (s, 2H), 6.80 (d, *J* = 7.5 Hz, 1H), 6.80 (d, *J* = 2.0 Hz, 1H), 6.85 (dd, *J* = 7.5, 2.0 Hz, 1H), 7.34–7.37 (m, 3H), 7.38–7.42 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 70.8, 79.1, 101.6, 107.1, 108.4, 120.0, 126.2, 128.7, 129.1, 134.9, 141.0, 147.8, 148.3. HRMS (ESI) calcd for $C_{15}H_{13}NNaO_5S$ $(M+Na)^+$ 342.0407, found 342.0404.



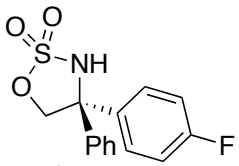
3ar

Compound 3ar. The ee was measured by HPLC (Chiralpak IC, hexane/$CHCl_3$/EtOH = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 14.5 min (*S*), t_2 = 17.2 min (*R*)); $[\alpha]^{20}_D$ –16 (*c* 0.99, $CHCl_3$) for 94% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 3.79 (s, 6H), 3.84 (s, 3H), 4.88 (br s, 1H), 5.03 (d, *J* = 9.5 Hz, 1H), 5.10 (d, *J* = 9.5 Hz, 1H), 6.54 (s, 2H), 7.35–7.43 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 56.2, 60.8, 71.0, 79.1, 103.9, 126.3, 128.9, 129.2, 136.6, 138.1, 140.9, 153.5. HRMS (ESI) calcd for $C_{17}H_{19}NNaO_6S$ $(M+Na)^+$ 388.0825, found 388.0821.



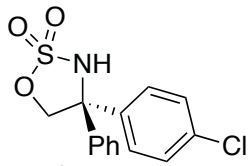
3as

Compound 3as. The ee was measured by HPLC (Chiralpak IC, hexane/$CHCl_3$/EtOH = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 13.5 min (*S*), t_2 = 15.2 min (*R*)); $[\alpha]^{20}_D$ –6 (*c* 0.99, $CHCl_3$) for 94% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 4.88 (s, 1H), 5.18 (d, *J* = 9.2 Hz, 1H), 5.20 (d, *J* = 9.2 Hz, 1H), 7.33 (dd, *J* = 8.5, 2.0 Hz, 1H), 7.35–7.43 (m, 5H), 7.52–7.56 (m, 2H), 7.82–7.88 (m, 3H), 7.95 (d, *J* = 2.0 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 71.1, 78.9, 123.7, 125.6, 126.4, 127.0, 127.1, 127.6, 128.4, 128.9, 129.2, 129.4, 132.9, 133.0, 138.0, 141.0. HRMS (ESI) calcd for $C_{18}H_{15}NNaO_3S$ $(M+Na)^+$ 348.0665, found 348.0662.



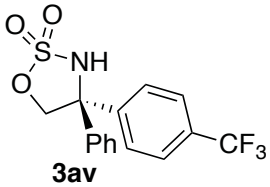
3at

Compound 3at. The ee was measured by HPLC (Chiralpak IC, hexane/$CHCl_3$/EtOH = 12:2:1, flow 0.6 mL/min, 254 nm, t_1 = 20.1 min (*S*), t_2 = 21.3 min (*R*)); $[\alpha]^{20}_D$ –18 (*c* 1.01, $CHCl_3$) for 93% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 4.85 (br s, 1H), 5.03 (d, *J* = 9.2 Hz, 1H), 5.09 (d, *J* = 9.2 Hz, 1H), 7.06–7.11 (m, 2H), 7.31–7.34 (m, 2H), 7.35–7.43 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 70.5, 79.1, 116.0 (d, $J_{F\text{-}C}$ = 23 Hz), 126.2, 128.4 (d, $J_{F\text{-}C}$ = 9 Hz), 129.0, 129.3, 136.9 (d, $J_{F\text{-}C}$ = 4 Hz), 140.8, 162.6 (d, $J_{F\text{-}C}$ = 250 Hz). HRMS (ESI) calcd for $C_{14}H_{12}FNNaO_3S$ $(M+Na)^+$ 316.0414, found 316.0410.



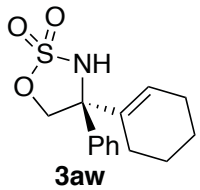
3au

Compound 3au. The ee was measured by HPLC (Chiralpak IC, hexane/$CHCl_3$/EtOH = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 11.9 min (*S*), t_2 = 12.8 min (*R*)); $[\alpha]^{20}_D$ –25 (*c* 1.00, $CHCl_3$) for 94% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 4.76 (br s, 1H), 5.01 (d, *J* = 9.2 Hz, 1H), 5.12 (d, *J* = 9.2 Hz, 1H), 7.29–7.36 (m, 4H), 7.37–7.43 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 70.5, 78.9, 126.1, 127.9, 129.0, 129.2, 129.4, 134.7, 139.5, 140.5. HRMS (ESI) calcd for $C_{14}H_{12}ClNNaO_3S$ $(M+Na)^+$ 332.0119, found 332.0116.



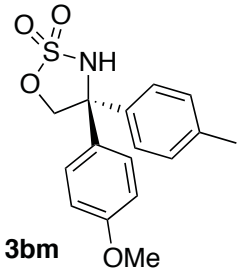
3av

Compound 3av. The ee was measured by HPLC (Chiralpak IC, hexane/$CHCl_3$/EtOH = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 9.4 min (*S*), t_2 = 9.9 min (*R*)); $[\alpha]^{20}_D$ –45 (*c* 0.57, $CHCl_3$) for 95% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 4.87 (s, 1H), 5.02 (d, *J* = 9.2 Hz, 1H), 5.19 (d, *J* = 9.2 Hz, 1H), 7.29 (d, *J* = 6.8 Hz, 2H), 7.37–7.45 (m, 3H), 7.57 (d, *J* = 8.5 Hz, 2H), 7.67 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 70.7, 78.7, 123.7 (q, $J_{F\text{-}C}$ = 273 Hz), 126.09 (q, $J_{F\text{-}C}$ = 7 Hz), 126.12, 126.9, 129.3, 129.5, 130.9 (q, $J_{F\text{-}C}$ = 33 Hz), 140.3, 144.9. HRMS (ESI) calcd for $C_{15}H_{12}F_3NNaO_3S$ $(M+Na)^+$ 366.0382, found 336.0377.



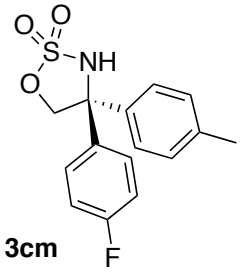
3aw

Compound 3aw. The isolated **3aw** contains a small amount (3%) of the double bond isomer. The ee was measured by HPLC (Chiralpak IC, hexane/$CHCl_3$/EtOH = 12:2:1, flow 0.6 mL/min, 254 nm, t_1 = 21.5 min (*R*), t_2 = 22.5 min (*S*)); $[\alpha]^{20}_D$ +24 (*c* 1.02, $CHCl_3$) for 99% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 1.31–1.63 (m, 4H), 1.73–1.87 (m, 2H), 2.14–2.19 (m, 2H), 4.56 (br s, 1H), 4.77 (d, *J* = 9.2 Hz, 1H), 4.92 (d, *J* = 9.2 Hz, 1H), 6.00–6.04 (m, 1H), 7.32–7.37 (m, 1H), 7.38–7.42 (m, 4H); ^{13}C NMR ($CDCl_3$) δ 21.7, 22.4, 24.9, 25.1, 72.1, 77.0, 125.9, 126.0, 128.5, 129.0, 135.5, 139.5. HRMS (ESI) calcd for $C_{14}H_{17}NNaO_3S$ $(M+Na)^+$ 302.0821, found 302.0822.



3bm

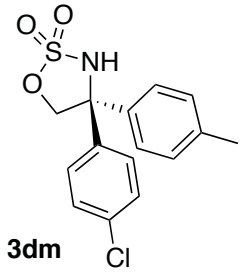
Compound 3bm. The ee was measured by HPLC (Chiralpak IC, hexane/$CHCl_3$/EtOH = 12:2:1, flow 0.6 mL/min, 254 nm, t_1 = 29.8 min (*S*), t_2 = 33.1 min (*R*)); $[\alpha]^{20}_D$ –2 (*c* 1.02, $CHCl_3$) for 94% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 2.35 (s, 3H), 3.91 (s, 3H), 4.69 (br s, 1H), 5.03 (d, *J* = 9.2 Hz, 1H), 5.95 (d, *J* = 9.2 Hz, 1H), 6.89 (d, *J* = 8.9 Hz, 2H), 7.19 (d, *J* = 8.1 Hz, 2H), 7.24 (d, *J* = 8.1 Hz, 2H), 7.27 (d, *J* = 8.9 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 21.0, 55.3, 70.6, 79.4, 114.3, 126.2, 127.7, 129.7, 133.1, 138.2, 138.6, 159.6. HRMS (ESI) calcd for $C_{16}H_{17}NNaO_4S$ $(M+Na)^+$ 342.0770, found 342.0762.



3cm

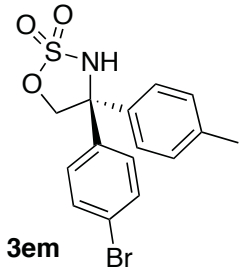
Compound 3cm. The ee was measured by HPLC (Chiralpak IA, hexane/$CHCl_3$/EtOH = 12:2:1, flow 0.6 mL/min, 254 nm, t_1 = 18.4 min (*S*), t_2 = 20.2 min (*R*)); $[\alpha]^{20}_D$ +31 (*c* 1.01, $CHCl_3$) for 94% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 2.36 (s, 3H), 4.70 (br s, 1H), 4.99 (d, *J* = 8.9 Hz, 1H), 5.10 (d, *J* = 8.9 Hz, 1H), 7.06–7.10 (m, 2H), 7.19 (d, *J* = 8.9 Hz, 2H), 7.21 (d, *J* = 8.9 Hz, 2H), 7.36–7.40 (m,

2H); ^{13}C NMR ($CDCl_3$) δ 21.0, 70.4, 79.2, 115.9 (d, J_{F-C} = 22 Hz), 126.1, 128.4 (d, J_{F-C} = 9 Hz), 129.9, 137.0 (d, J_{F-C} = 3 Hz), 137.9, 139.0, 162.5 (d, J_{F-C} = 250 Hz). HRMS (ESI) calcd for $C_{15}H_{14}FNNaO_3S$ $(M+Na)^+$ 330.0571, found 330.0576.



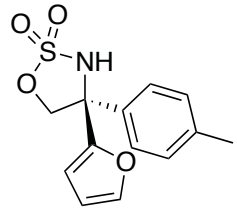
3dm

Compound 3dm. The ee was measured by HPLC (Chiralpak IC, hexane/$CHCl_3$/EtOH = 24:4:1, flow 0.6 mL/min, 254 nm, t_1 = 35.7 min (*S*), t_2 = 37.8 min (*R*)); $[\alpha]^{20}_D$ +36 (*c* 1.01, $CHCl_3$) for 94% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 2.36 (s, 3H), 4.70 (br s, 1H), 4.97 (d, *J* = 8.9 Hz, 1H), 5.11 (d, *J* = 8.9 Hz, 1H), 7.17 (d, *J* = 8.2 Hz, 2H), 7.21 (d, *J* = 8.2 Hz, 2H), 7.35 (d, *J* = 8.5 Hz, 2H), 7.37 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 21.0, 70.4, 79.0, 126.0, 127.9, 129.2, 130.0, 134.6, 137.6, 139.2, 139.7. HRMS (ESI) calcd for $C_{15}H_{14}ClNNaO_3S$ $(M+Na)^+$ 346.0275, found 346.0276.



3em

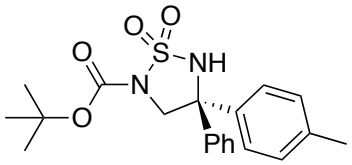
Compound 3em. The ee was measured by HPLC (Chiralpak IC, hexane/$CHCl_3$/EtOH = 12:2:1, flow 0.6 mL/min, 254 nm, t_1 = 21.1 min (*S*), t_2 = 22.6 min (*R*)); $[\alpha]^{20}_D$ +37 (*c* 1.00, $CHCl_3$) for 92% ee (*S*). ^{1}H NMR ($CDCl_3$) δ 2.36 (s, 3H), 4.71 (br s, 1H), 4.97 (d, *J* = 8.9 Hz, 1H), 5.11 (d, *J* = 8.9 Hz, 1H), 7.17 (d, *J* = 8.5 Hz, 2H), 7.21 (d, *J* = 8.5 Hz, 2H), 7.29 (d, *J* = 8.5 Hz, 2H), 7.53 (d, *J* = 8.5 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 21.0, 70.5, 78.9, 122.8, 126.1, 128.2, 130.0, 132.1, 137.6, 139.2, 140.2. HRMS (ESI) calcd for $C_{15}H_{14}BrNNaO_3S$ $(M+Na)^+$ 389.9770, found 389.9769.



3fm

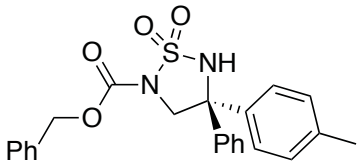
Compound 3fm. The ee was measured by HPLC (Chiralpak IC, hexane/$CHCl_3$/EtOH =

6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 12.5 min (*S*), t_2 = 13.9 min (*R*)); $[\alpha]^{20}_D$ –20 (*c* 0.71, $CHCl_3$) for 91% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 2.37 (s, 3H), 4.83 (d, *J* = 9.0 Hz, 1H), 5.02 (br s, 1H), 5.14 (d, *J* = 9.0 Hz, 1H), 6.28 (d, *J* = 3.4 Hz, 1H), 6.38 (dd, *J* = 3.4, 1.4 Hz, 1H), 7.23 (d, *J* = 8.1 Hz, 2H), 7.32 (d, *J* = 8.1 Hz, 2H), 7.46 (d, *J* = 1.4 Hz, 1H); ^{13}C NMR ($CDCl_3$) δ 21.1, 67.0, 77.8, 110.6, 110.9, 126.2, 129.6, 134.9, 139.2, 143.8, 151.6. HRMS (ESI) calcd for $C_{13}H_{13}NNaO_4S$ $(M+Na)^+$ 302.0457, found 302.0449.



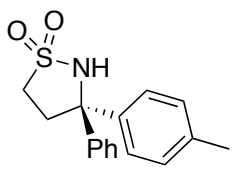
3gm

Compound 3gm. The ee was measured by HPLC (Chiralpak IC, hexane/$CHCl_3$/EtOH = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 9.6 min (*S*), t_2 = 10.7 min (*R*)); $[\alpha]^{20}_D$ +13 (*c* 1.04, $CHCl_3$) for 95% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 1.51 (s, 9H), 2.32 (s, 3H), 4.63 (d, *J* = 10.6 Hz, 1H), 4.66 (d, *J* = 10.6 Hz, 1H), 5.05 (br s, 1H), 7.16 (d, *J* = 8.1 Hz, 2H), 7.27–7.32 (m, 3H), 7.36 (t, *J* = 7.8 Hz, 2H), 7.16 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 21.0, 27.9, 57.8, 65.1, 84.8, 125.7, 125.8, 128.3, 129.0, 129.7, 138.27, 138.34, 141.4, 149.4. HRMS (ESI) calcd for $C_{20}H_{24}N_2NaO_4S$ $(M+Na)^+$ 411.1349, found 411.1344.



3hm

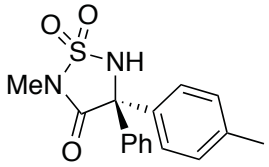
Compound 3hm. The ee was measured by HPLC (Chiralpak IA, hexane/$CHCl_3$/EtOH = 6:2:1, flow 0.6 mL/min, 254 nm, t_1 = 11.5 min (*R*), t_2 = 13.7 min (*S*)); $[\alpha]^{20}_D$ +10 (*c* 1.01, $CHCl_3$) for 93% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 2.31 (s, 3H), 4.71 (d, *J* = 10.2 Hz, 1H), 4.74 (d, *J* = 10.2 Hz, 1H), 5.10 (br s, 1H), 5.26 (s, 2H), 7.16 (d, *J* = 8.1 Hz, 2H), 7.27–7.40 (m, 10H), 7.42 (d, *J* = 7.5 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 21.0, 58.1, 65.7, 69.0, 125.7, 125.8, 127.8, 128.4, 128.6, 129.0, 129.7, 134.7, 138.0, 138.4, 141.1, 150.4. HRMS (ESI) calcd for $C_{23}H_{22}N_2NaO_4S$ $(M+Na)^+$ 445.1192, found 445.1187.



3im

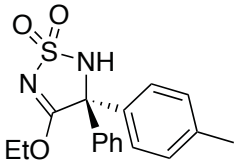
Compound 3im. The ee was measured by HPLC (Chiralpak IB, hexane/$CHCl_3$/EtOH =

30:10:1, flow 0.6 mL/min, 254 nm, t_1 = 12.9 min (*S*), t_2 = 14.1 min (*R*)); $[\alpha]^{20}_D$ +16 (*c* 1.01, $CHCl_3$) for 95% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 2.33 (s, 3H), 3.10 (dt, *J* = 17.2, 7.1 Hz, 1H), 3.14 (dt, *J* = 17.2, 7.1 Hz, 1H), 3.24 (t, *J* = 7.1 Hz, 2H), 4.56 (br s, 1H), 7.16 (d, *J* = 8.1 Hz, 2H), 7.23–7.30 (m, 3H), 7.35 (t, *J* = 7.8 Hz, 2H), 7.39 (d, *J* = 8.1 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 20.9, 37.2, 48.0, 68.8, 126.0, 126.1, 127.8, 128.8, 129.5, 137.8, 140.7, 143.9. HRMS (ESI) calcd for $C_{16}H_{17}NNaO_2S$ $(M+Na)^+$ 310.0872, found 310.0876.



3jm

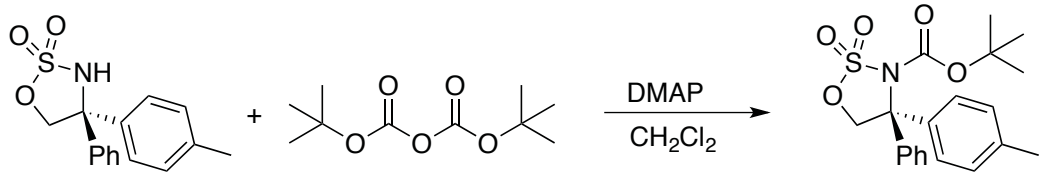
Compound 3jm. The ee was measured by HPLC (Chiralpak IB, hexane/$CHCl_3$/EtOH = 90:30:1, flow 0.6 mL/min, 254 nm, t_1 = 11.8 min (*S*), t_2 = 12.5 min (*R*)); $[\alpha]^{20}_D$ +9 (*c* 0.84, $CHCl_3$) for 79% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 2.36 (s, 3H), 3.18 (s, 3H), 5.13 (br s, 1H), 7.18 (d, *J* = 8.1 Hz, 2H), 7.32 (d, *J* = 8.1 Hz, 2H), 7.36–7.41 (m, 3H), 7.47–7.51 (m, 2H); ^{13}C NMR ($CDCl_3$) δ 21.1, 26.2, 75.4, 127.4, 127.5, 128.8, 129.0, 129.1, 129.6, 135.3, 138.1, 139.3. HRMS (ESI) calcd for $C_{16}H_{16}N_2NaO_3S$ $(M+Na)^+$ 339.0774, found 339.0768.



3km

Compound 3km. The ee was measured by HPLC (Chiralpak IB, hexane/$CHCl_3$/EtOH = 90:30:1, flow 0.6 mL/min, 254 nm, t_1 = 14.2 min (*S*), t_2 = 14.6 min (*R*)); $[\alpha]^{20}_D$ +8 (*c* 1.00, $CHCl_3$) for 91% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 1.37 (t, *J* = 7.2 Hz, 3H), 2.37 (s, 3H), 4.51 (q, *J* = 7.2 Hz, 2H), 4.84 (br s, 1H), 7.19 (d, *J* = 8.1 Hz, 2H), 7.25 (d, *J* = 8.1 Hz, 2H), 7.36–7.45 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 13.7, 21.1, 69.9, 77.3, 127.6, 127.8, 128.8, 129.1, 129.6, 135.3, 138.1, 139.3, 177.1. HRMS (ESI) calcd for $C_{17}H_{18}N_2NaO_3S$ $(M+Na)^+$ 353.0930, found 353.0929.

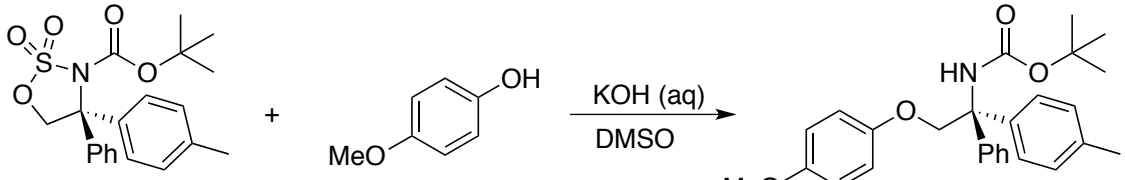
7. Transformations



3am (93% ee) **4**: 94%, 93% ee

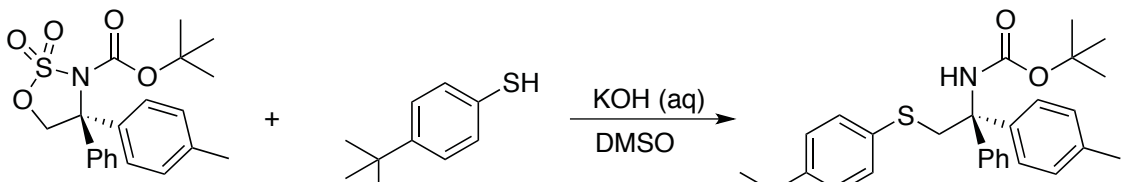
Compound 4. To a solution of **3am** (145 mg, 0.50 mmol, 93% ee) and

4-dimethylaminopyridine (12 mg, 0.10 mmol) in CH_2Cl_2 (1.0 mL) was added di-*tert*-butyl dicarbonate (218 mg, 1.0 mmol), and the mixture was stirred at room temperature overnight. The mixture was concentrated on a rotary evaporator and the residue was subjected to flash column chromatography on silica gel with hexane/ethyl acetate (5:1) to give **4** (184 mg, 0.47 mmol, 94% yield). The ee was measured by HPLC (Chiralpak IC, hexane/$CHCl_3$/EtOH = 90:30:1, flow 0.6 mL/min, 254 nm, t_1 = 26.5 min (*S*), t_2 = 29.8 min (*R*)); $[\alpha]^{20}_D$ +1 (*c* 1.46, $CHCl_3$) for 93% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 1.25 (s, 9H), 2.39 (s, 3H), 4.90 (d, *J* = 9.9 Hz, 1H), 4.92 (d, *J* = 9.9 Hz, 1H), 7.24 (d, *J* = 8.1 Hz, 2H), 7.31 (d, *J* = 8.1 Hz, 2H), 7.37–7.45 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 20.9, 27.5, 73.0, 79.2, 85.2, 127.78, 127.83, 128.4, 128.5, 129.1, 131.1, 137.4, 138.5, 148.3. HRMS (ESI) calcd for $C_{20}H_{23}NNaO_5S$ $(M+Na)^+$ 412.1189, found 412.1183.



4 (93% ee) **5**: 79%, 93% ee

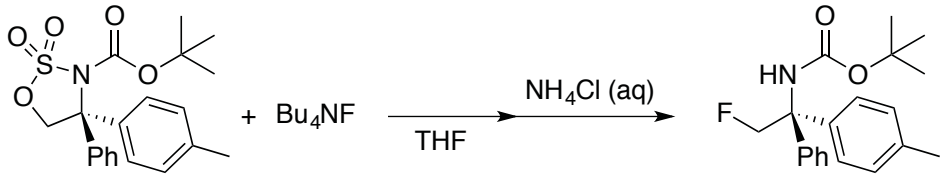
Compound 5. To a solution of **4** (104 mg, 0.27 mmol, 93% ee) and 4-methoxyphenol (66 mg, 0.53 mmol) in dimethyl sulfoxide (1.0 mL) was added 8 M KOH (aq) (66 μL, 0.53 mmol), and the mixture was stirred at room temperature for 15 h. Aqueous NH_4Cl solution was added to the mixture and it was extracted with diethyl ether. The organic extracts were washed with H_2O and brine, dried over $MgSO_4$, filtered, and concentrated on a rotary evaporator. The residue was subjected to preparative TLC on silica gel with hexane/ethyl acetate (9:1) to give **5** (91.4 mg, 0.21 mmol, 79% yield). The ee was measured by HPLC (Chiralcel OD-H × 3, hexane/2-propanol = 95:5, flow 0.4 mL/min, 254 nm, t_1 = 39.9 min (*R*), t_2 = 41.8 min (*S*)); $[\alpha]^{20}_D$ +2 (*c* 0.56, $CHCl_3$) for 93% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 1.35 (br s, 9H), 2.33 (s, 3H), 3.76 (s, 3H), 4.72 (br s, 2H), 5.52 (br s, 1H), 6.81 (d, *J* = 8.8 Hz, 2H), 6.85 (d, *J* = 8.8 Hz, 2H), 7.13 (d, *J* = 8.2 Hz, 2H), 7.23–7.29 (m, 3H), 7.32 (d, *J* = 7.5 Hz, 2H), 7.39 (d, *J* = 7.5 Hz, 2H); ^{13}C NMR ($CDCl_3$, 50 °C) δ 20.9, 28.3, 55.8, 64.4, 73.1 (br), 79.6, 114.8, 116.3, 127.2, 127.22, 127.31, 128.2, 128.9, 136.9, 140.3, 143.4, 153.0, 154.4, 154.7. HRMS (ESI) calcd for $C_{27}H_{31}NNaO_4$ $(M+Na)^+$ 456.2145, found 456.2144.



4 (93% ee) **6**: 89%, 93% ee

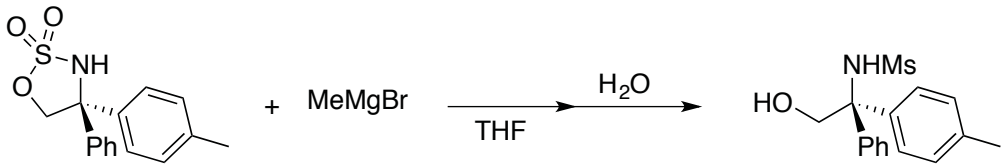
Compound 6. To a solution of **4** (81.1 mg, 0.21 mmol, 93% ee) and

4-*tert*-butylbenzenethiol (69 mg, 0.42 mmol) in dimethyl sulfoxide (1.0 mL) was added 8 M KOH (aq) (52 μL, 0.42 mmol), and the mixture was stirred at room temperature for 12 h. Aqueous NH_4Cl solution was added to the mixture and it was extracted with diethyl ether. The organic extracts were washed with H_2O and brine, dried over $MgSO_4$, filtered, and concentrated on a rotary evaporator. The residue was subjected to preparative TLC on silica gel with hexane/ethyl acetate (20:1) to give **6** (88.1 mg, 0.19 mmol, 89% yield). The ee was measured by HPLC (Chiralpak AD-H, hexane/2-propanol = 9:1, flow 0.6 mL/min, 254 nm, t_1 = 10.5 min (*S*), t_2 = 13.5 min (*R*)); $[\alpha]^{20}_D$ –3 (*c* 0.70, $CHCl_3$) for 93% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 1.29 (br s, 18H), 2.31 (s, 3H), 4.16 (br s, 2H), 5.69 (br s, 1H), 7.10 (d, *J* = 8.1 Hz, 2H), 7.19–7.27 (m, 7H), 7.29 (t, *J* = 7.5 Hz, 2H), 7.37 (d, *J* = 7.5 Hz, 2H); ^{13}C NMR ($CDCl_3$, 50 °C) δ 20.9, 28.2, 31.3, 34.4, 45.8 (br), 64.2, 79.6, 125.8, 126.7, 126.8, 127.0, 128.1, 128.9, 130.6, 133.4, 136.7, 141.2, 144.5, 149.6, 154.5. HRMS (ESI) calcd for $C_{30}H_{37}NNaO_2S_1$ $(M+Na)^+$ 498.2437, found 498.2430.



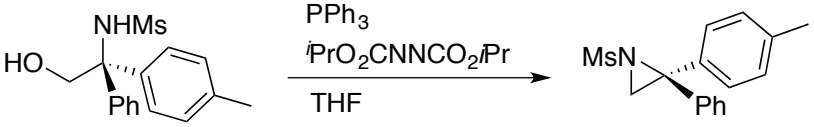
4 (93% ee) **7**: 80%, 93% ee

Compound 7. To a solution of **4** (66.8 mg, 0.17 mmol, 93% ee) in THF (0.8 mL) was added tetrabutylammonium fluoride solution (1 M in THF, 0.35 mL, 0.35 mmol), and the mixture was stirred at 60 °C for 16 h. Aqueous NH_4Cl solution was added to the mixture, and it was stirred at room temperature for 2 h. The mixture was extracted with diethyl ether, and the extracts were washed with H_2O and brine, dried over $MgSO_4$, filtered, and concentrated on a rotary evaporator. The residue was subjected to preparative TLC on silica gel with hexane/ethyl acetate (5:1) to give **7** (45.1 mg, 0.14 mmol, 80% yield). The ee was measured by HPLC (Chiralpak IA × 2, hexane/$CHCl_3$/EtOH = 90:30:1, flow 0.6 mL/min, 254 nm, t_1 = 13.1 min (*S*), t_2 = 13.6 min (*R*)); $[\alpha]^{20}_D$ +1 (*c* 0.69, $CHCl_3$) for 93% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 1.39 (br s, 9H), 2.33 (s, 3H), 5.22 (br s, 1H), 5.31 (br s, 2H), 7.14 (d, *J* = 8.1 Hz, 2H), 7.19 (d, *J* = 8.1 Hz, 2H), 7.26–7.37 (m, 5H); ^{13}C NMR ($CDCl_3$, 50 °C) δ 21.0, 28.3, 64.2 (d, $J_{F\text{-}C}$ = 17 Hz), 80.0, 85.7 (br d, $J_{F\text{-}C}$ = 181 Hz), 127.0, 127.1, 127.5, 128.4, 129.1, 137.3, 139.3, 142.4, 157.5. HRMS (ESI) calcd for $C_{20}H_{24}FNNaO_2$ $(M+Na)^+$ 352.1683, found 352.1682.



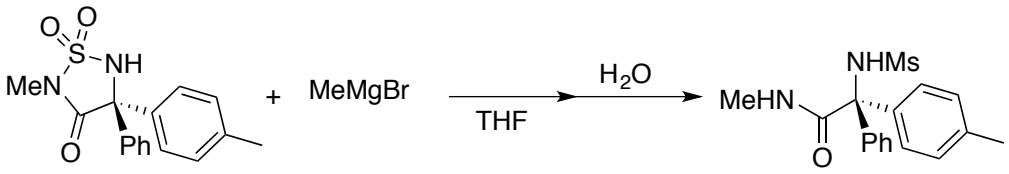
3am (93% ee) **8**: 80%, 93% ee

Compound 8. To a solution of **3am** (57.9 mg, 0.20 mmol, 93% ee) in THF (1.0 mL) was added MeMgBr (2 M in diethyl ether, 0.9 mL, 2.7 mmol), and the mixture was stirred at 60 °C for 12 h. The mixture was cooled to 0 °C and H_2O was slowly added. The mixture was extracted with diethyl ether, and the extracts were washed with H_2O and brine, dried over $MgSO_4$, filtered, and concentrated on a rotary evaporator. The residue was subjected to preparative TLC on silica gel with hexane/ethyl acetate (1:1) to give **8** (48.9 mg, 0.16 mmol, 80% yield). The ee was measured by HPLC (Chiralpak IA, hexane/$CHCl_3$/EtOH = 30:10:1, flow 0.6 mL/min, 254 nm, t_1 = 17.6 min (*R*), t_2 = 19.5 min (*S*)); $[\alpha]^{20}_D$ +3 (*c* 0.55, $CHCl_3$) for 93% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 2.26 (t, *J* = 7.5 Hz, 1H), 2.35 (s, 3H), 2.44 (s, 3H), 4.38 (dd, *J* = 11.9, 7.5 Hz, 1H), 4.41 (dd, *J* = 11.9, 7.5 Hz, 1H), 5.50 (s, 1H), 7.18 (d, *J* = 8.2 Hz, 2H), 7.26 (d, *J* = 8.2 Hz, 2H), 7.31–7.40 (m, 5H); ^{13}C NMR ($CDCl_3$) δ 21.0, 42.7, 68.3, 68.7, 128.08, 128.13, 128.4, 129.2, 138.05, 138.09, 141.4. HRMS (ESI) calcd for $C_{16}H_{19}NNaO_3S$ $(M+Na)^+$ 328.0978, found 328.0985.



8 (93% ee) **9**: 85%, 93% ee

Compound 9. To a solution of **8** (46.9 mg, 0.15 mmol, 93% ee) and PPh_3 (42.5 mg, 0.16 mmol) in THF (1.5 mL) was added diisopropyl azodicarboxylate (32 μL, 0.16 mmol) at 0 °C, and the mixture was stirred at room temperature for 15 h. The mixture was passed through a short alumina column and concentrated on a rotary evaporator. The residue was subjected to column chromatography on alumina with hexane/ethyl acetate (2:1) to give **9** (37.9 mg, 0.13 mmol, 85% yield). The ee was measured by HPLC (Chiralpak IA, hexane/ethyl acetate = 4:1, flow 0.6 mL/min, 254 nm, t_1 = 24.4 min (*R*), t_2 = 28.3 min (*S*)); $[\alpha]^{20}_D$ +29 (*c* 0.51, $CHCl_3$) for 93% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 2.33 (s, 3H), 3.01 (s, 3H), 3.06 (s, 1H), 3.21 (s, 1H), 7.14 (d, *J* = 7.5 Hz, 2H), 7.27–7.35 (m, 5H), 7.42 (d, *J* = 7.5 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 21.2, 40.6, 41.4, 56.4, 128.19, 128.24, 128.4, 128.9, 129.0, 134.4, 138.3, 138.5. HRMS (ESI) calcd for $C_{16}H_{17}NNaO_2S$ $(M+Na)^+$ 310.0872, found 310.0870.



3jm (79% ee) **10**: 90%, 79% ee

Compound 10. To a solution of **3jm** (51.6 mg, 0.16 mmol, 79% ee) in THF (1 mL) was added MeMgBr (3 M in diethyl ether, 0.73 mL, 2.2 mmol), and the mixture was stirred at 60 °C for 12 h. The mixture was cooled to 0 °C, and aqueous NH_4Cl solution was slowly added. The mixture was extracted with diethyl ether, and the extracts were washed with H_2O and brine, dried over $MgSO_4$, filtered, and concentrated on a rotary evaporator. The residue was subjected to preparative TLC on silica gel with hexane/ethyl acetate (1:1) to give **10** (48.8 mg, 0.15 mmol, 90% yield). The ee was measured by HPLC (Chiralpak IB, hexane/$CHCl_3$/EtOH = 30:10:1, flow 0.6 mL/min, 254 nm, t_1 = 16.3 min (*S*), t_2 = 18.5 min (*R*)); $[\alpha]^{20}_D$ –1 (*c* 1.00, $CHCl_3$) for 79% ee (*R*). ^{1}H NMR ($CDCl_3$) δ 2.02 (s, 3H), 2.37 (s, 3H), 2.76 (d, *J* = 4.8 Hz, 3H), 5.73 (br q, *J* = 4.8 Hz, 1H), 6.81 (s, 1H), 7.22 (d, *J* = 8.2 Hz, 2H), 7.35–7.43 (m, 3H), 7.46 (d, *J* = 8.2 Hz, 2H), 7.59 (d, *J* = 7.4 Hz, 2H); ^{13}C NMR ($CDCl_3$) δ 21.0, 27.4, 42.1, 71.5, 128.46, 128.53, 129.2, 129.37, 129.40, 135.6, 138.6, 138.8, 171.7. HRMS (ESI) calcd for $C_{17}H_{20}N_2NaO_3S$ $(M+Na)^+$ 355.1087, found 355.1084.

8. Data for X-ray crystal structure of 3au

Colorless crystals of **3au** suitable for X-ray crystallographic analysis were obtained by recrystallization from dichloroethane/hexane. The ORTEP drawings of **3au** are shown in Figure S1. The crystal structure has been deposited at the Cambridge Crystallographic Centre (deposition number: CCDC 924230). The data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif.

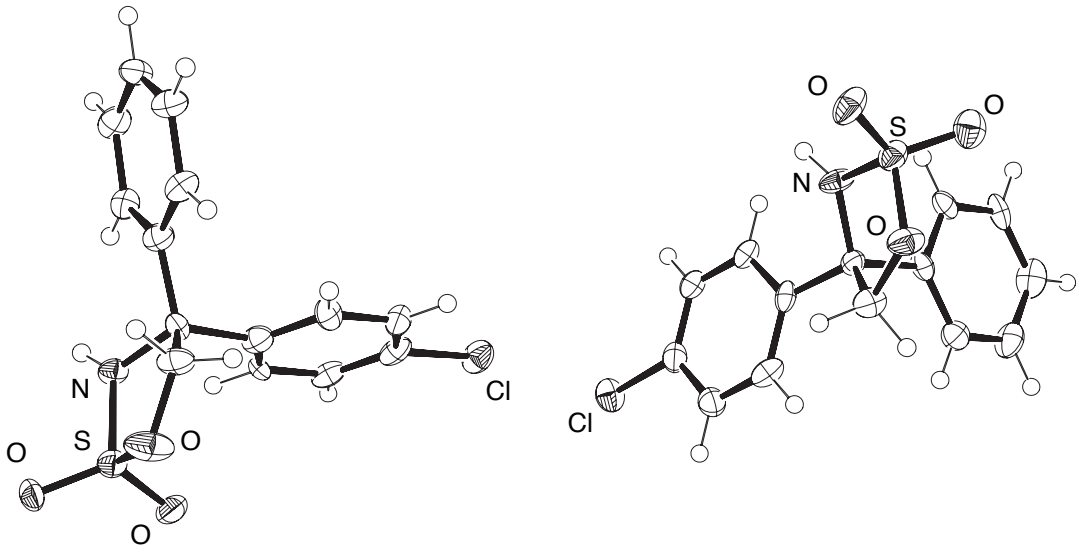


Figure S1. ORTEP illustration of **3au** with thermal ellipsoids drawn at 50% probability level.

X-Ray data were collected on a Rigaku RAXIS-RAPID imaging plate diffractometer using a graphite monochromater with Cu-*K*α radiation (λ = 1.54187 Å) at 93 K. The structure was solved by direct method (SHELXS-97) and refined with full-matrix least-square technique (SHELXL-97).[9] The absolute structure was deduced based on Flack parameter[10] –0.01(2). The data for **3au** are summarized in Table S1.

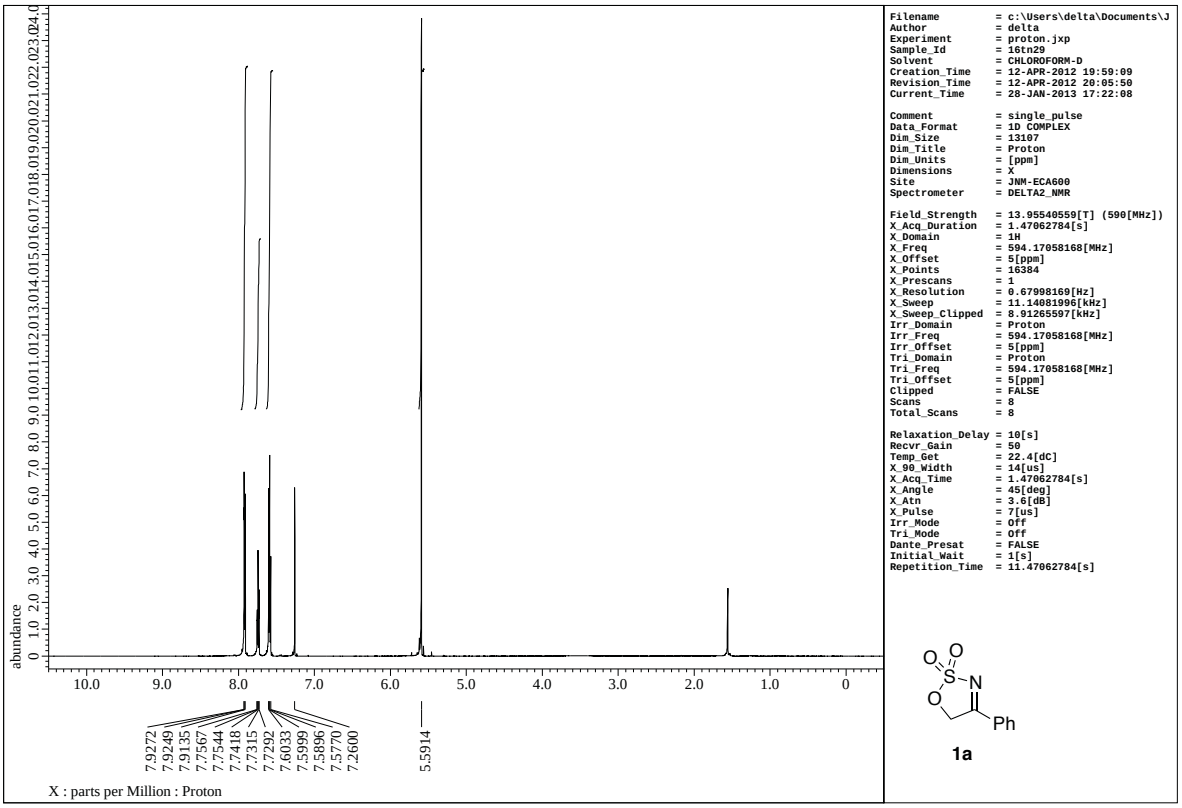
Table S1. Crystal data and structure refinement for **3au**.

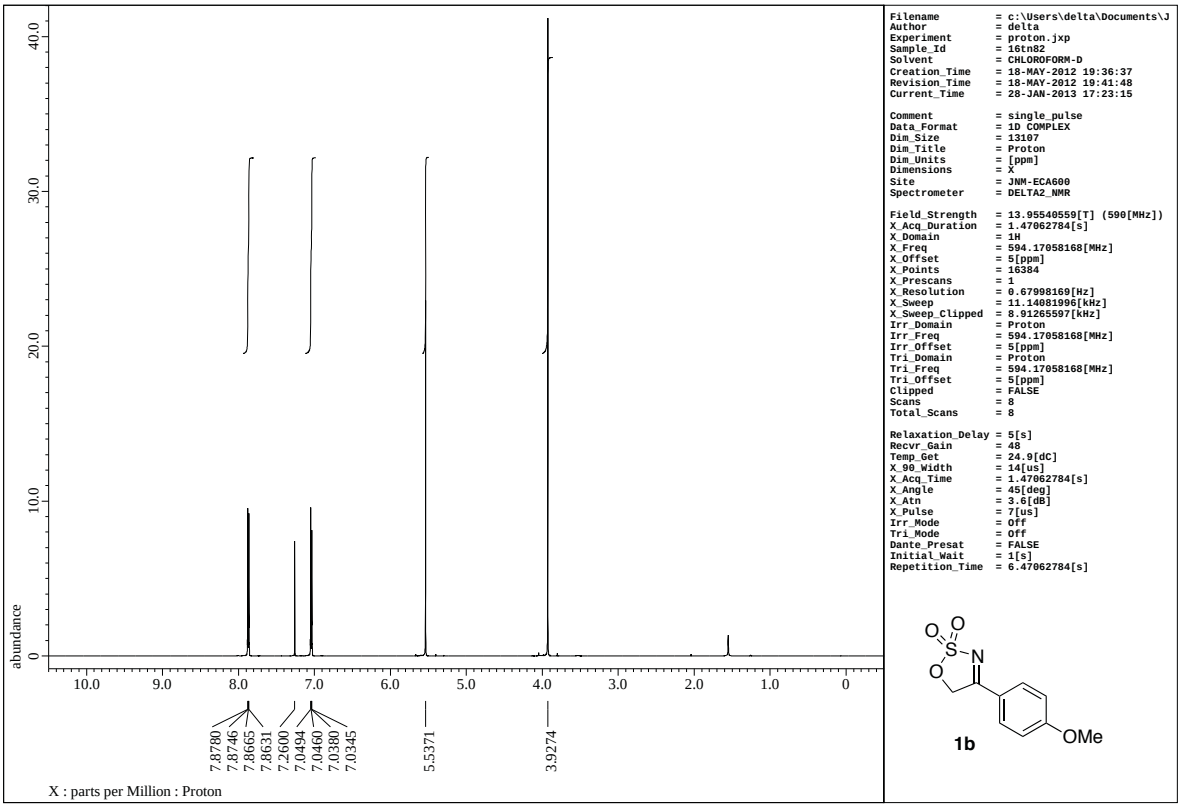
	3au
Empirical formula	$C_{14}H_{12}ClNO_3S$
Formula weight	309.76
Temperature	93(2) K
Crystal system	Monoclinic
Space group	$P2_1$ (#4)
Unit cell dimensions	a = 9.4413(2) Å
	b = 15.6524(3) Å
	c = 9.8374(2) Å
	β = 114.5778(14)°
Volume	1322.05(5) Å^3
Z	4
Density (calculated) [Mg/m^3]	1.556
μ (mm^{-1})	4.103
F(000)	640
Reflections collected	7504
Independent reflections	3777 [*R*(int) = 0.0696]
Completeness to θ (%)	95.9%
Goodness-of-fit	1.020
R_1 [I>2σ (I)]	0.0505
wR_2 (all data)	0.1379
Flack Parameter	–0.01(2)
Largest diff. peak and hole [e$^-$/Å^{-3}]	0.403 and – 0.433

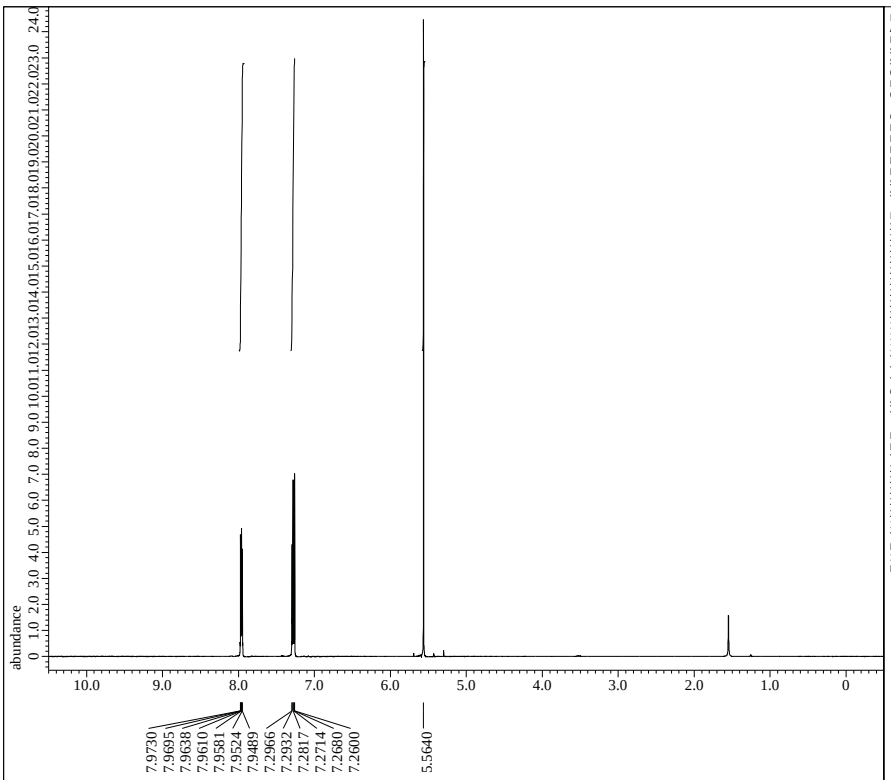
9 Sheldrick, G. M. Program for the solution and refinement of crystal structures, University of Göttingen, Göttingen, Germany, 1997.

10 Flack, H. D. *Acta Cryst*. 1983, **A39**, 876.

9. ^{1}H and ^{13}C NMR spectra and chiral HPLC charts





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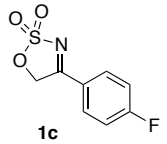

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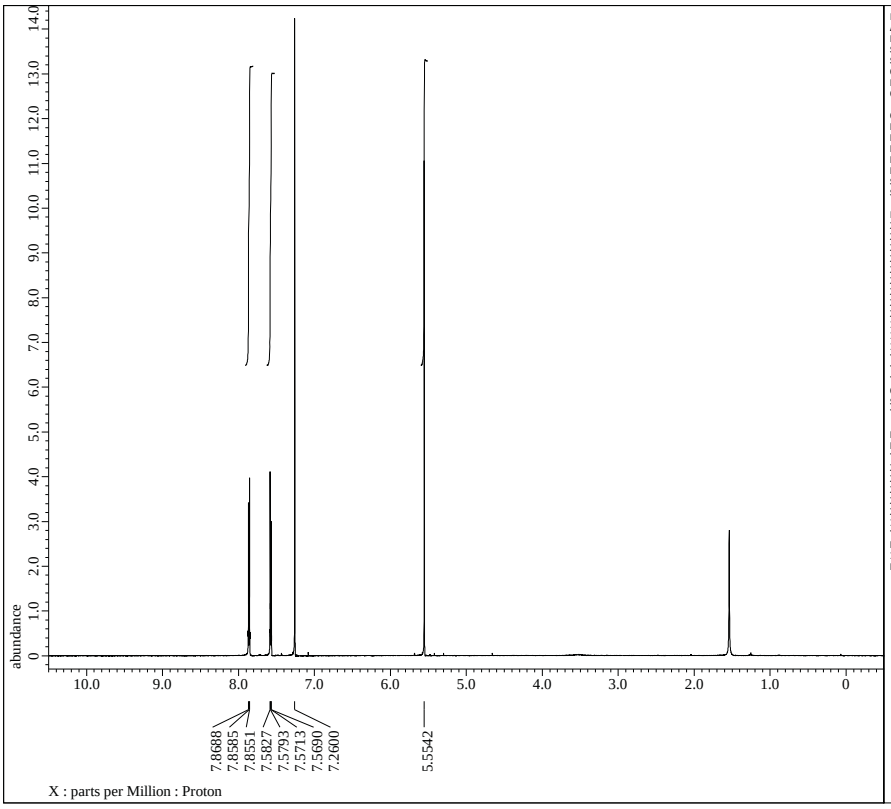


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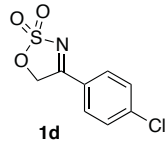

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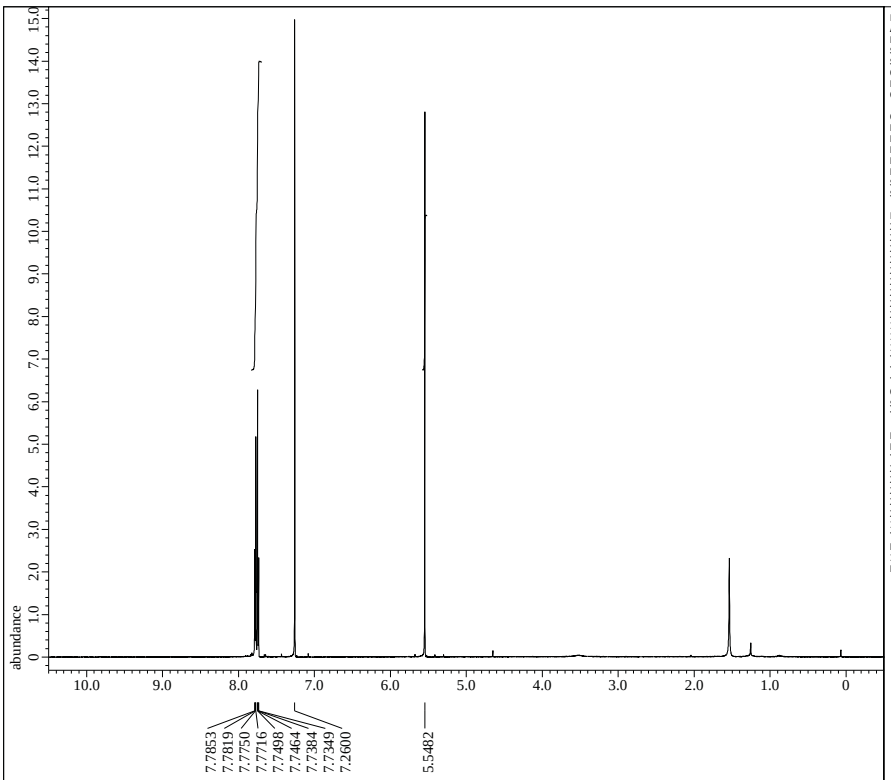


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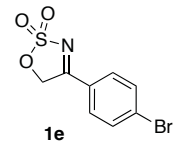

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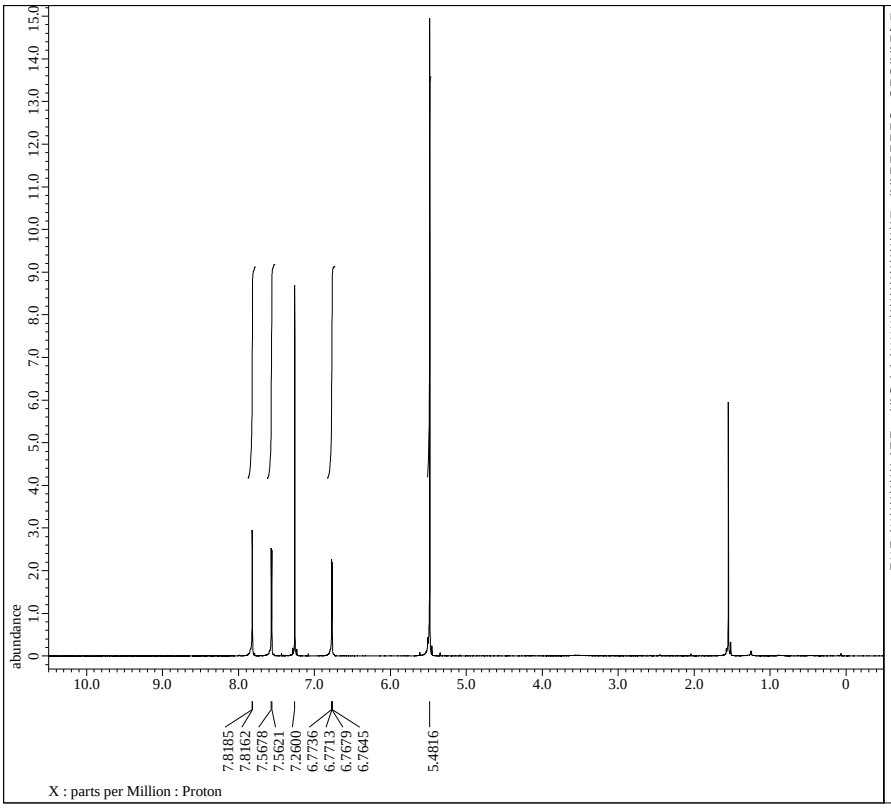


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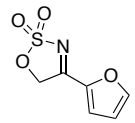
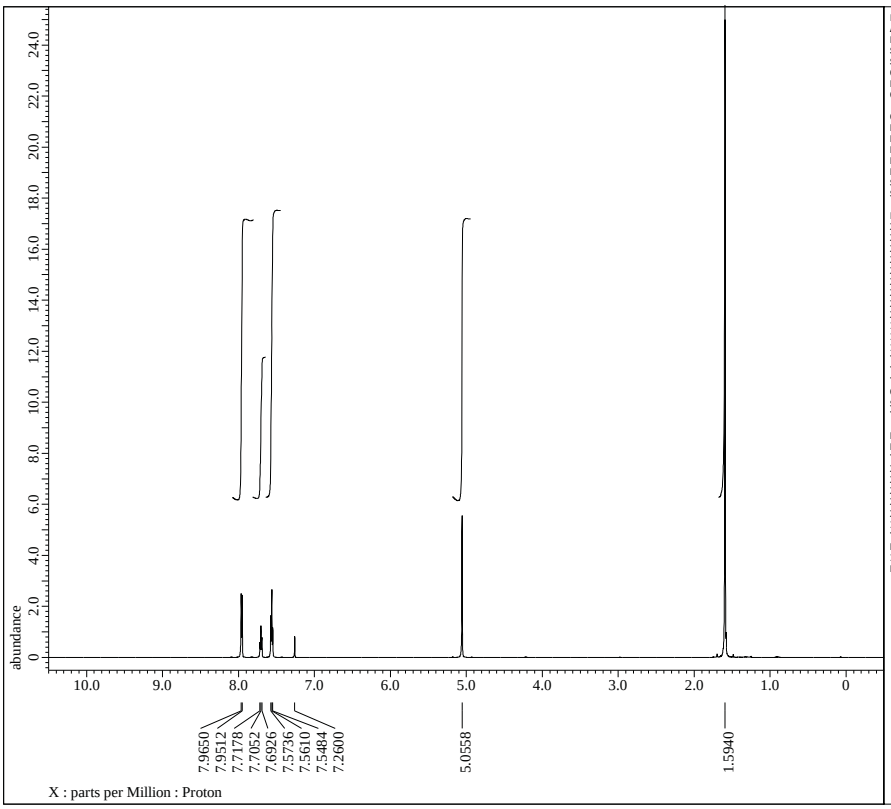
Tri_Domain        = Proton
Tri_Freq          = 594.17058168[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 54
Temp_Get          = 21.8[dC]
X_90_Width        = 14[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3.6[dB]
X_Pulse           = 7[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



1f

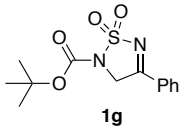



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = data hf1042
Solvent           = CHLOROFORM-D
Creation_Time     = 22-DEC-2012 16:50:45
Revision_Time     = 28-JAN-2013 17:49:39
Current_Time      = 28-JAN-2013 17:50:06

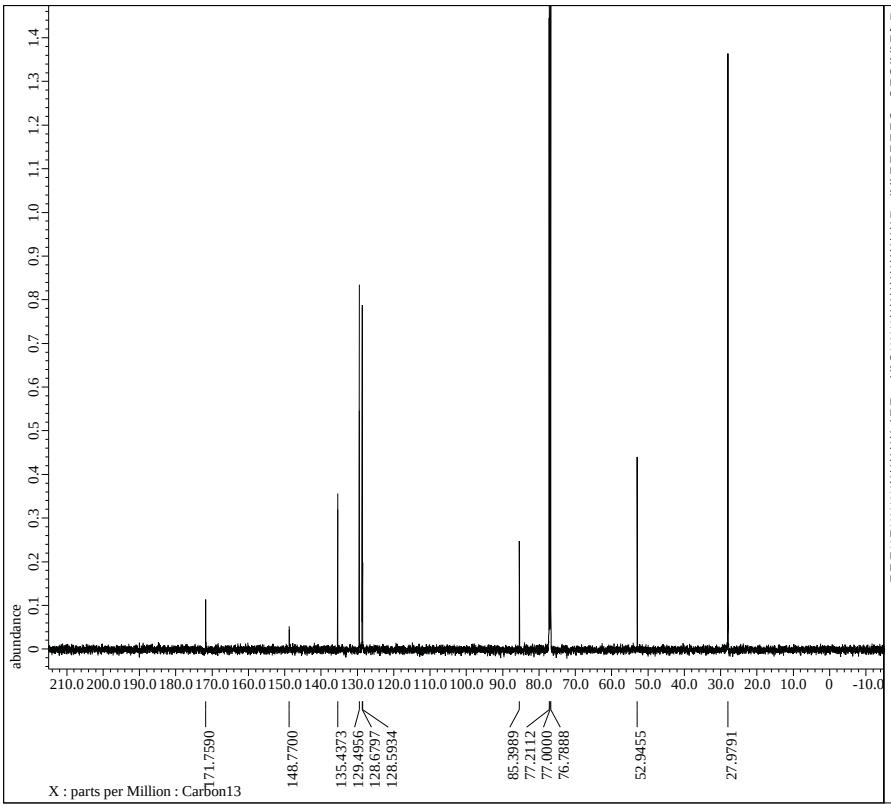
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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          = 23.4[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]
```



1g

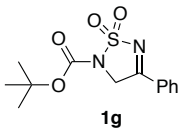



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = data hf1042
Solvent           = CHLOROFORM-D
Creation_Time     = 22-DEC-2012 16:53:33
Revision_Time     = 22-DEC-2012 17:33:20
Current_Time      = 28-JAN-2013 17:50:59

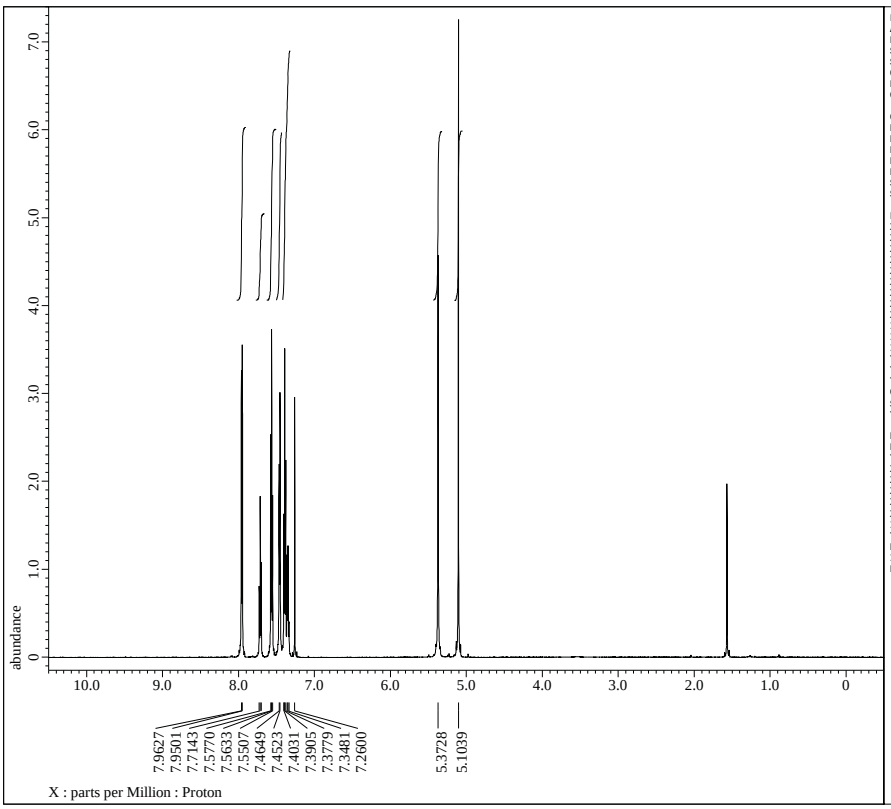
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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        = 58
Temp_Get          = 24.3[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]
```



1g

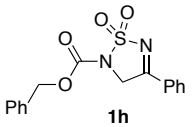



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 16tn30
Solvent           = CHLOROFORM-D
Creation_Time     = 12-APR-2012 20:05:58
Revision_Time     = 12-APR-2012 20:10:45
Current_Time      = 28-JAN-2013 17:53:16

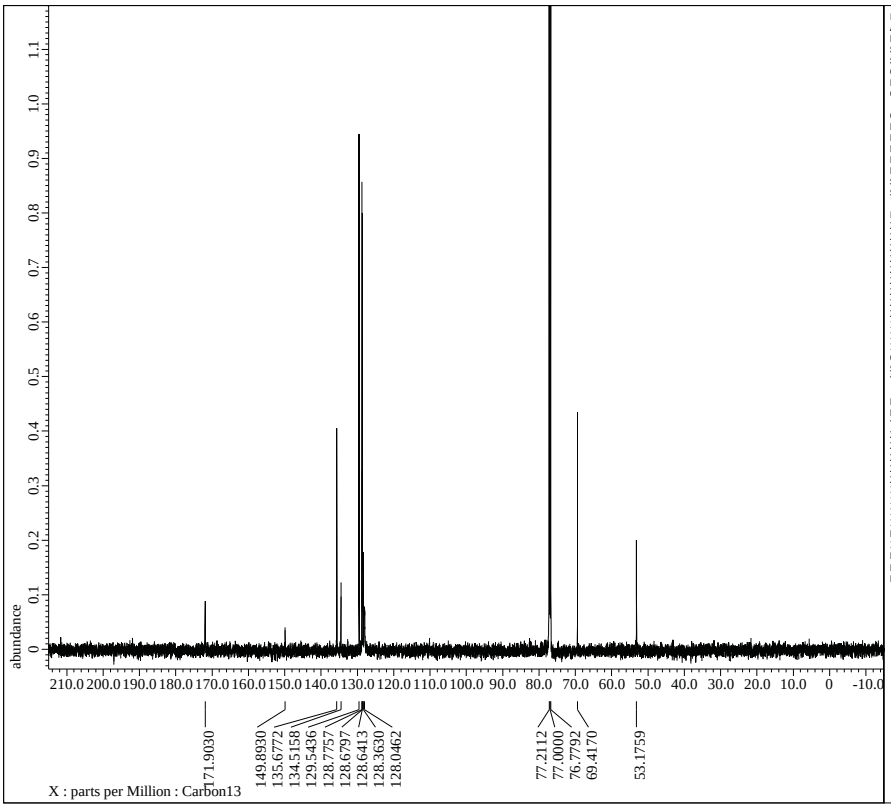
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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          = 22.4[dC]
X_90_Width        = 14[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3.6[dB]
X_Pulse           = 7[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



1h

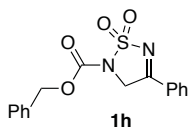



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 16tn30
Solvent           = CHLOROFORM-D
Creation_Time     = 12-APR-2012 20:09:25
Revision_Time     = 12-APR-2012 20:23:30
Current_Time      = 28-JAN-2013 17:54:16

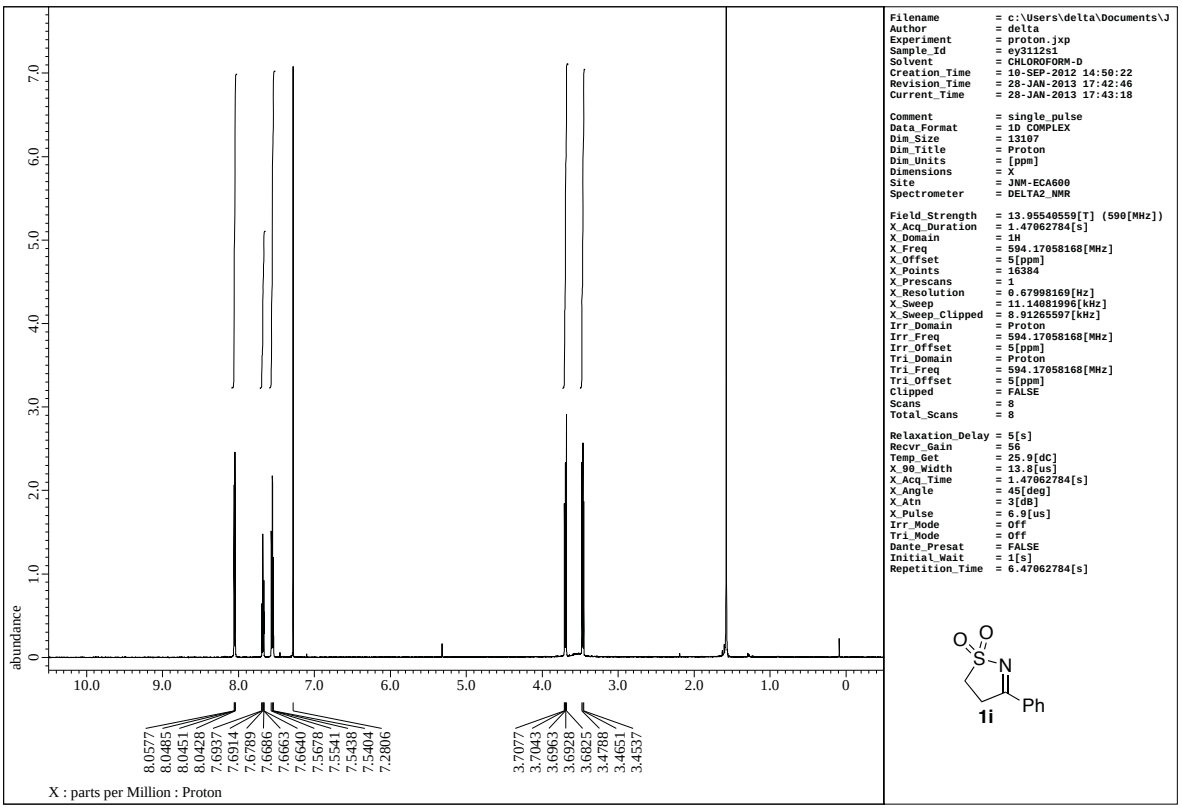
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

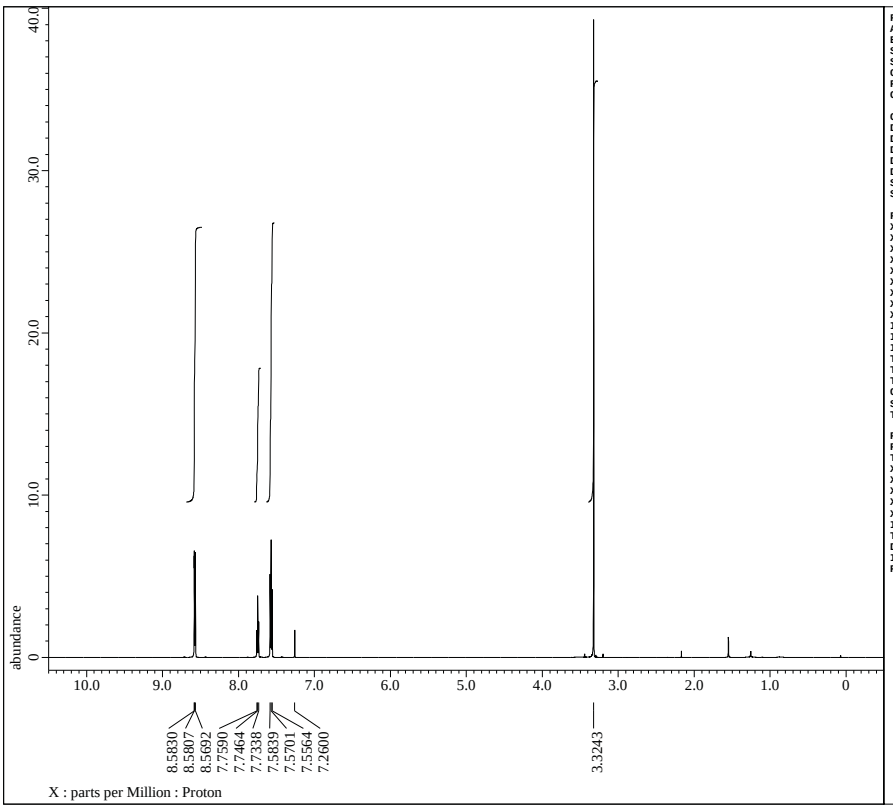
Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 0.69730304[s]
X_Domain          = 13C
X_Freq            = 149.40429612[MHz]
X_Offset          = 100.0[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.43409672[Hz]
X_Sweep           = 46.9924812[kHz]
X_Sweep_Clipped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

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.1[dC]
X_90_Width        = 9.9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 7[dB]
X_Pulse           = 3.3[us]
Irr_Atn_Dec       = 18.2[dB]
Irr_Atn_Noe       = 18.2[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]
```



1h


abundance
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.0577
8.0485
8.0451
8.0428
7.6937
7.6914
7.6789
7.6686
7.6663
7.6640
7.5678
7.5541
7.5438
7.5404
7.2806
3.7077
3.7043
3.6963
3.6928
3.6825
3.4788
3.4651
3.4537
X : parts per Million : Proton
Filename = c:\Users\delta\Documents\J
Author = delta
Experiment = proton.jxp
Sample_Id = ey3112s1
Solvent = CHLOROFORM-D
Creation_Time = 10-SEP-2012 14:50:22
Revision_Time = 28-JAN-2013 17:42:46
Current_Time = 28-JAN-2013 17:43:18
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 = 56
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]
O
O
S
N
Ph
1i

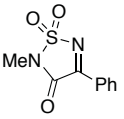



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = date hf1085gen
Solvent           = CHLOROFORM-D
Creation_Time     = 12-OCT-2012 18:37:35
Revision_Time     = 29-JAN-2013 19:30:59
Current_Time      = 29-JAN-2013 19:31:25

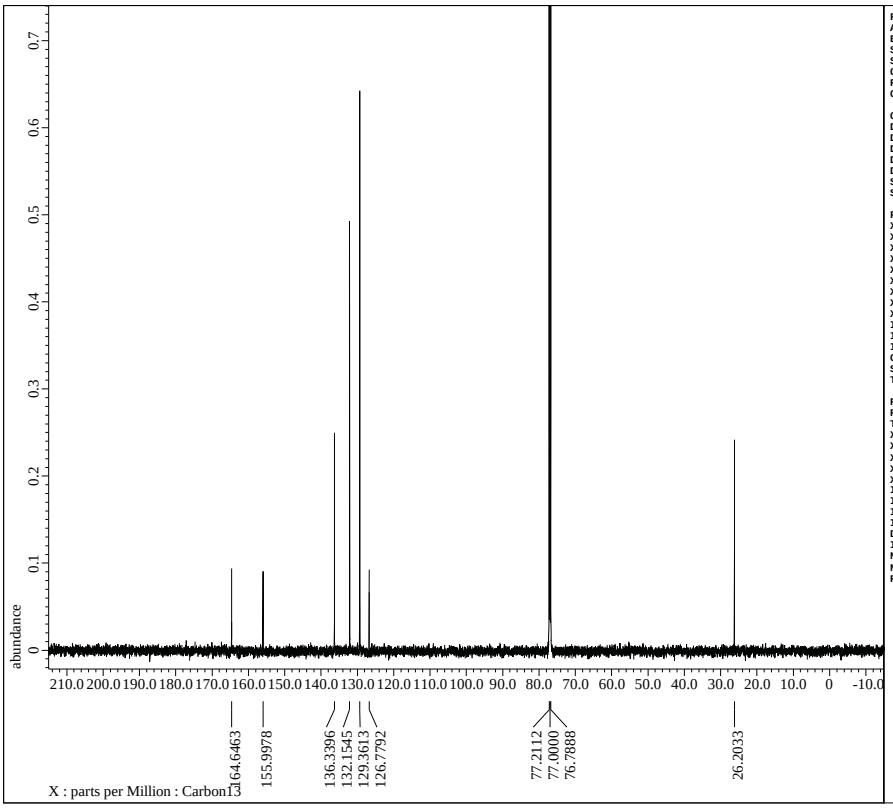
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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          = 24.6[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]
```



1j

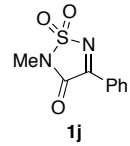



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = date hf1085gen
Solvent           = CHLOROFORM-D
Creation_Time     = 12-OCT-2012 18:41:25
Revision_Time     = 12-OCT-2012 18:58:25
Current_Time      = 29-JAN-2013 19:32:09

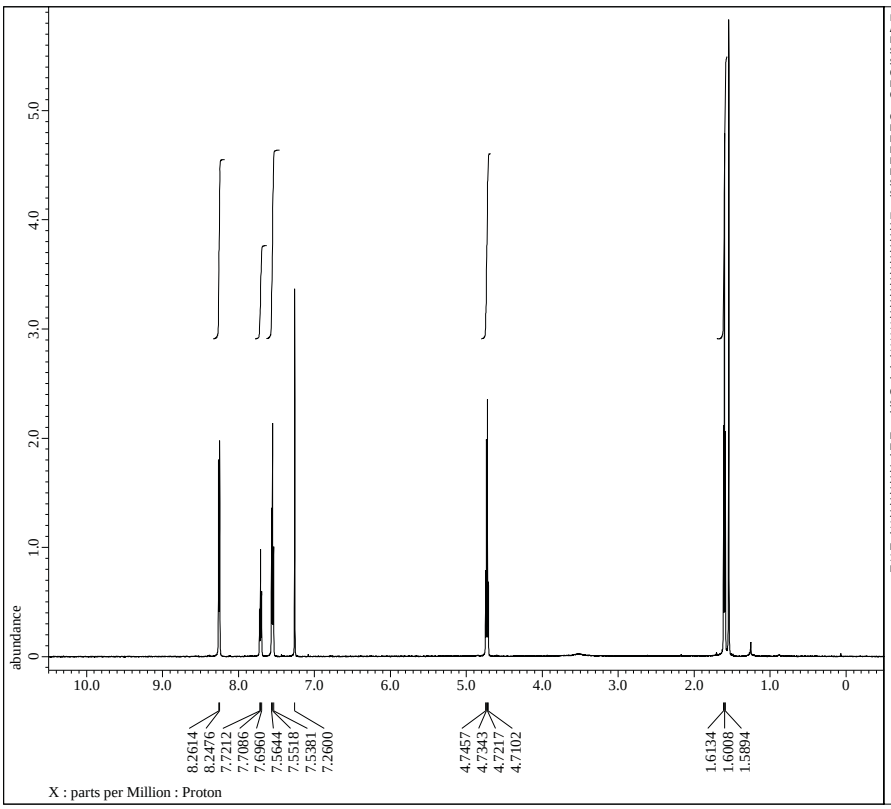
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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        = 54
Temp_Get          = 25.3[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]
```



1j

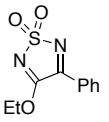



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = data ey3108
Solvent           = CHLOROFORM-D
Creation_Time     = 22-DEC-2012 16:42:59
Revision_Time     = 28-JAN-2013 17:38:08
Current_Time      = 28-JAN-2013 17:38:37

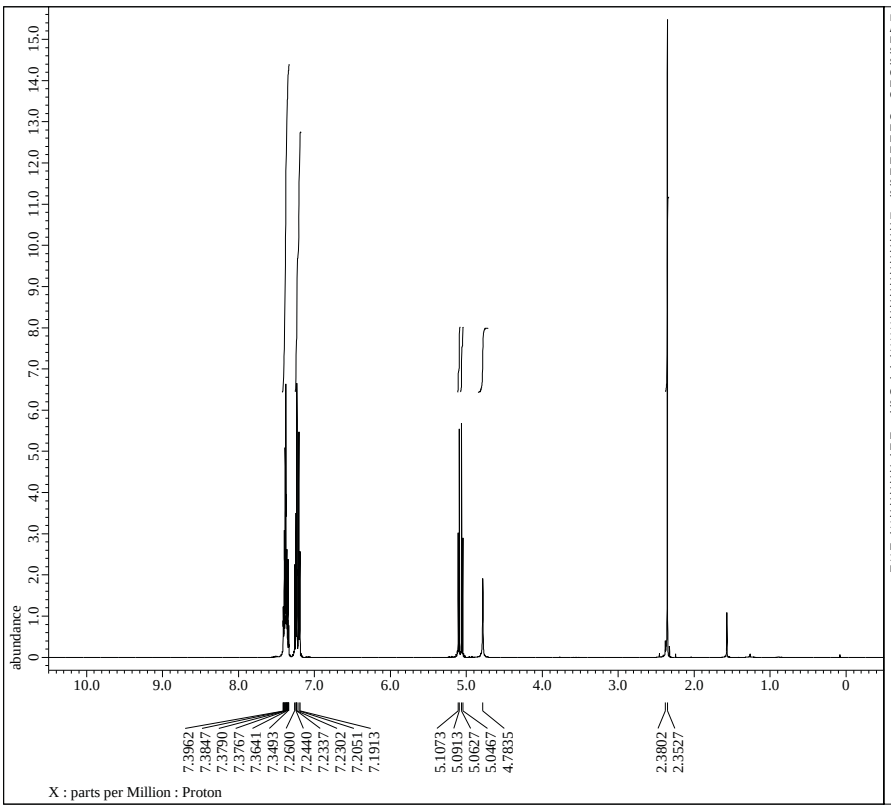
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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        = 56
Temp_Get          = 23.3[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]
```



1k

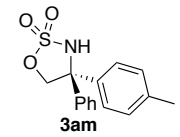



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 16tn12
Solvent           = CHLOROFORM-D
Creation_Time     =  2-APR-2012 19:15:34
Revision_Time     =  2-APR-2012 19:31:03
Current_Time      = 29-JAN-2013 19:33:50

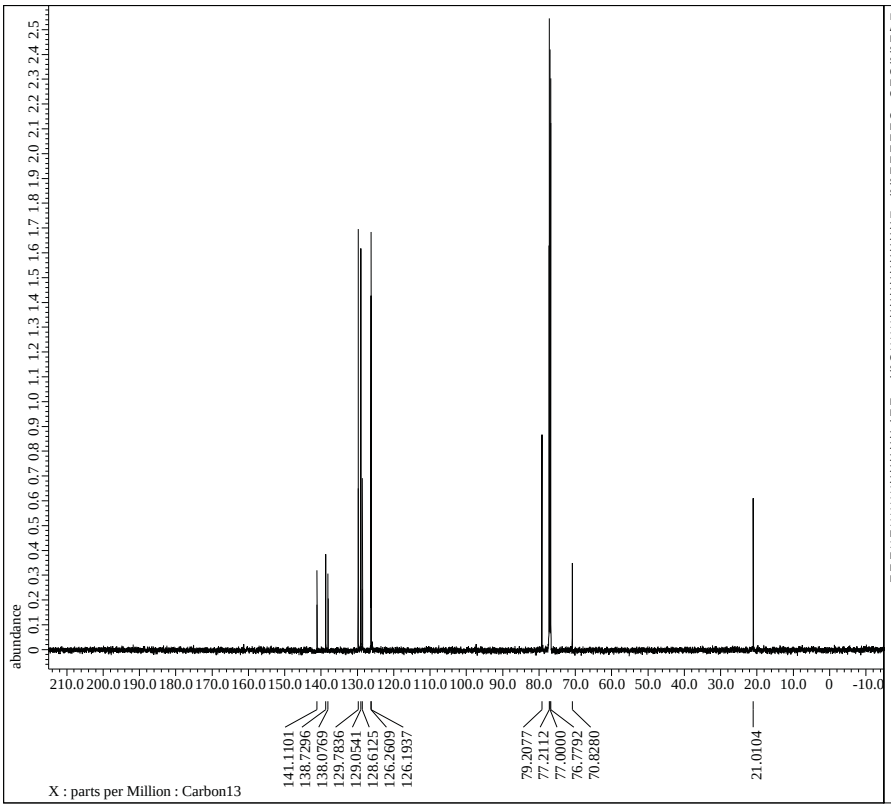
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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          = 21.9[dC]
X_90_Width        = 14[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3.6[dB]
X_Pulse           = 7[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



3am

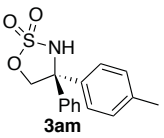



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 16tn12
Solvent           = CHLOROFORM-D
Creation_Time     =  2-APR-2012 19:18:28
Revision_Time     =  2-APR-2012 19:32:22
Current_Time      = 29-JAN-2013 19:34:29

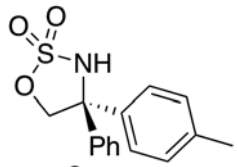
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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           = FALSE
Scans             = 256
Total_Scans       = 256

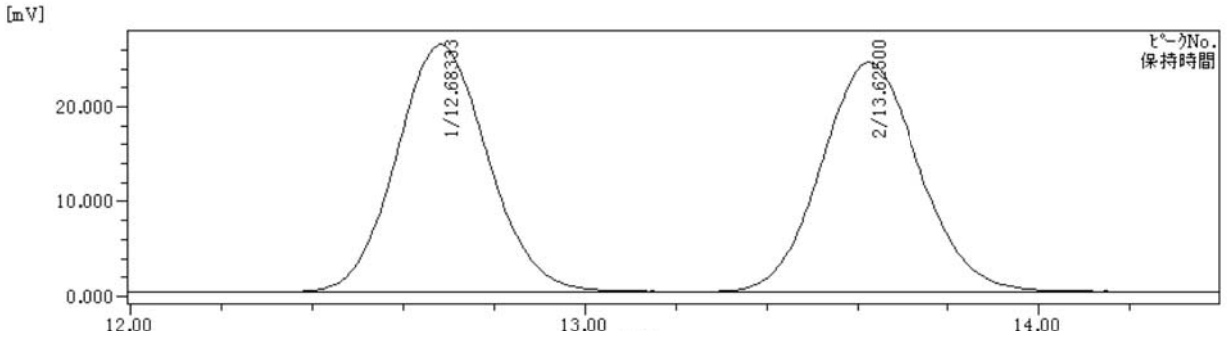
Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22.6[dC]
X_90_Width        = 9.9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 7[dB]
X_Pulse           = 3.3[us]
Irr_Atn_Dec       = 18.2[dB]
Irr_Atn_Noe       = 18.2[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



3am

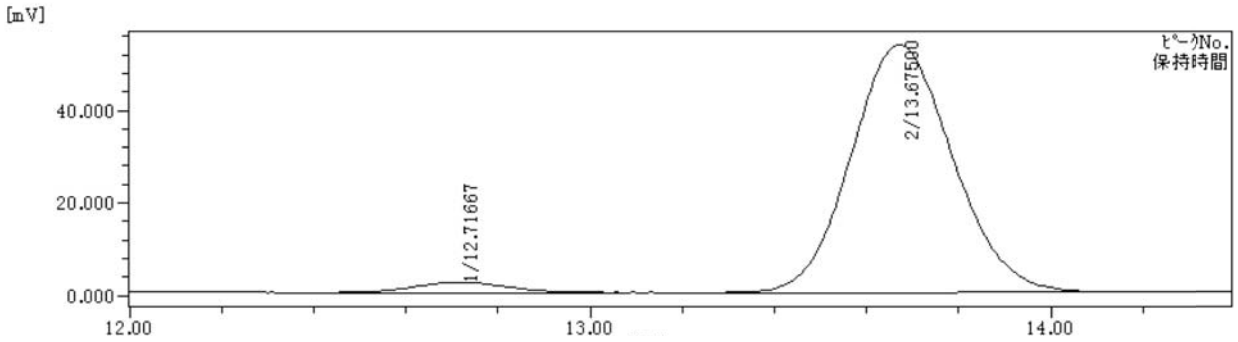


3am



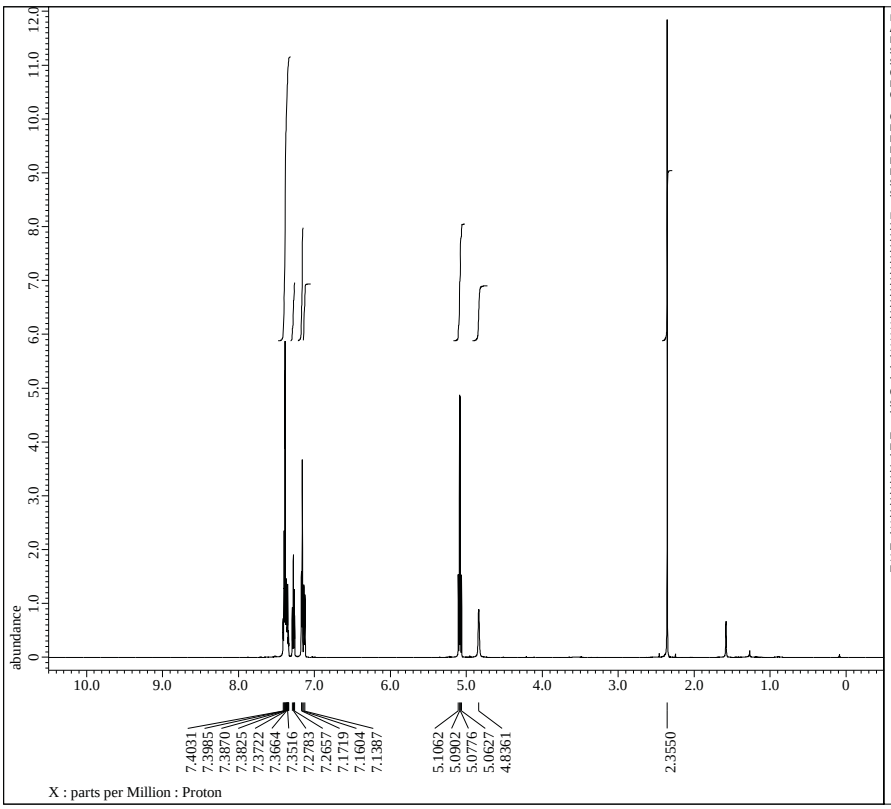
time (min)

Pk#	Retention Time	Area	Area (%)
1	12.683	365.446	49.957
2	13.625	366.082	50.043



time (min)

Pk#	Retention Time	Area	Area (%)
1	12.717	31.143	3.647
2	13.675	822.680	96.353

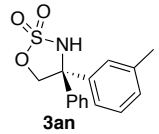



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = ey2009p1
Solvent           = CHLOROFORM-D
Creation_Time     = 12-JUN-2012 22:01:21
Revision_Time     = 29-JAN-2013 19:36:13
Current_Time      = 29-JAN-2013 19:36:55

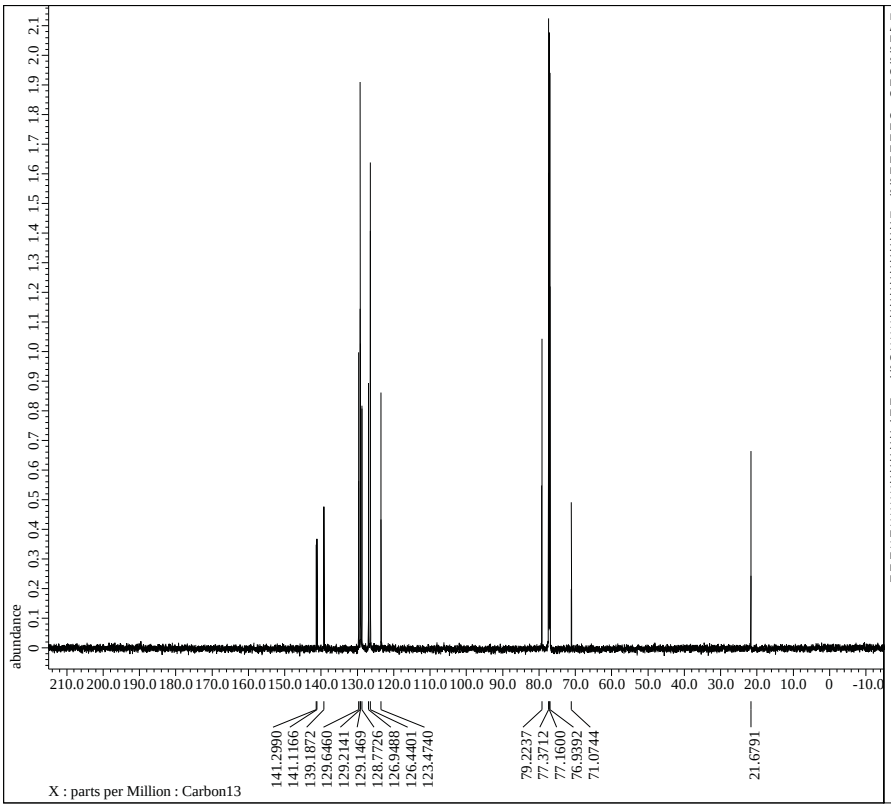
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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          = 25.4[dC]
X_90_Width        = 14[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3.6[dB]
X_Pulse           = 7[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



3an

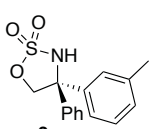



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = ey2009p1
Solvent           = CHLOROFORM-D
Creation_Time     = 12-JUN-2012 22:04:15
Revision_Time     = 12-JUN-2012 22:18:55
Current_Time      = 29-JAN-2013 19:38:15

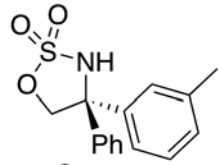
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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

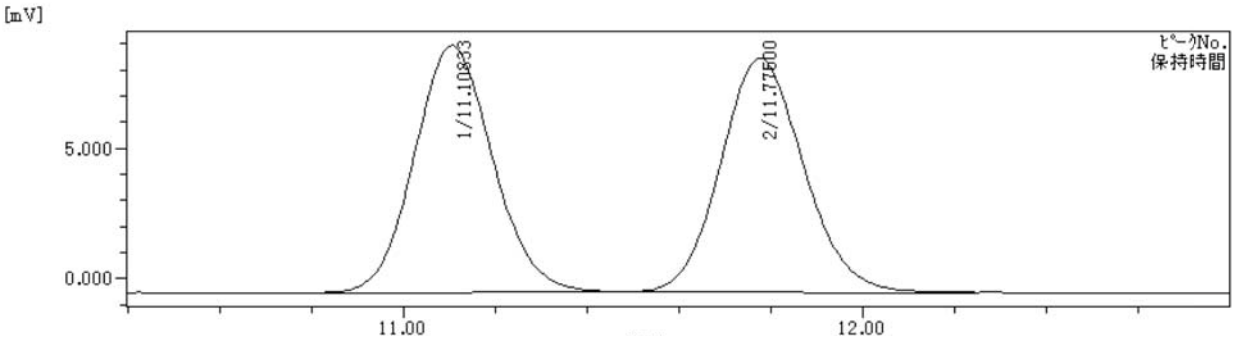
Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 26.1[dC]
X_90_Width        = 9.9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 7[dB]
X_Pulse           = 3.3[us]
Irr_Atn_Dec       = 18.2[dB]
Irr_Atn_Noe       = 18.2[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



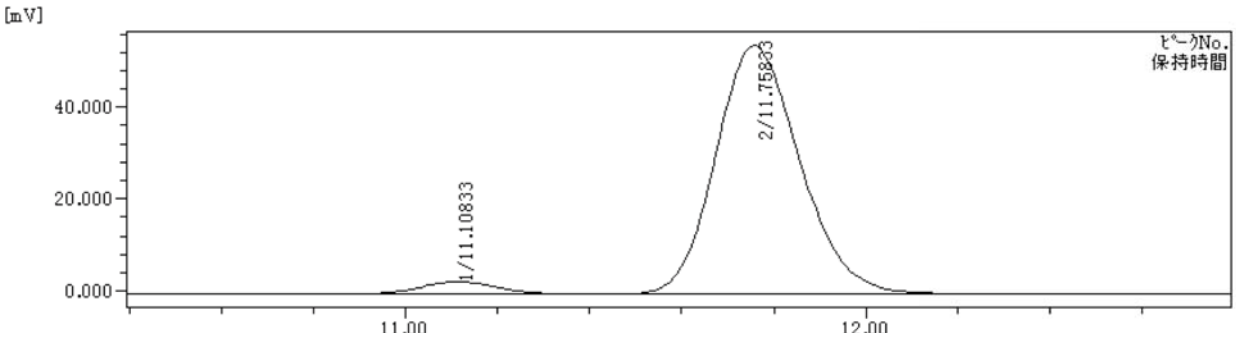
3an



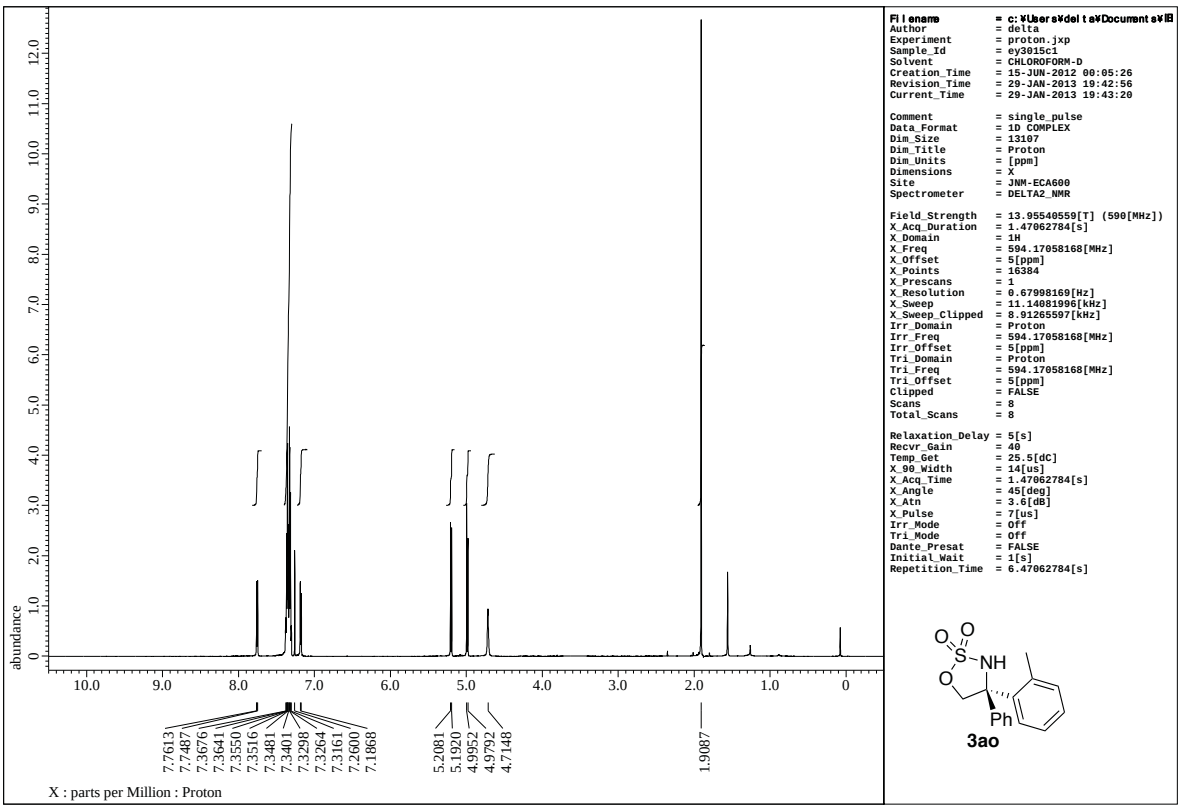
3an



Pk#	Retention Time	Area	Area (%)
1	11.108	112.412	50.023
2	11.775	112.310	49.977



Pk#	Retention Time	Area	Area (%)
1	11.108	30.417	4.212
2	11.758	691.716	95.788


abundance
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
7.7613 7.7487 7.3676 7.3641 7.3550 7.3516 7.3481 7.3401 7.3298 7.3264 7.3161 7.2600 7.1868
5.2081 5.1920 4.9952 4.9792 4.7148
1.9087
X : parts per Million : Proton
Filename = c:¥Users¥delta¥Documents¥
Author = delta
Experiment = proton.jxp
Sample_Id = ey3015c1
Solvent = CHLOROFORM-D
Creation_Time = 15-JUN-2012 00:05:26
Revision_Time = 29-JAN-2013 19:42:56
Current_Time = 29-JAN-2013 19:43:20
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 = 25.5[dC]
X_90_Width = 14[us]
X_Acq_Time = 1.47062784[s]
X_Angle = 45[deg]
X_Atn = 3.6[dB]
X_Pulse = 7[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.47062784[s]
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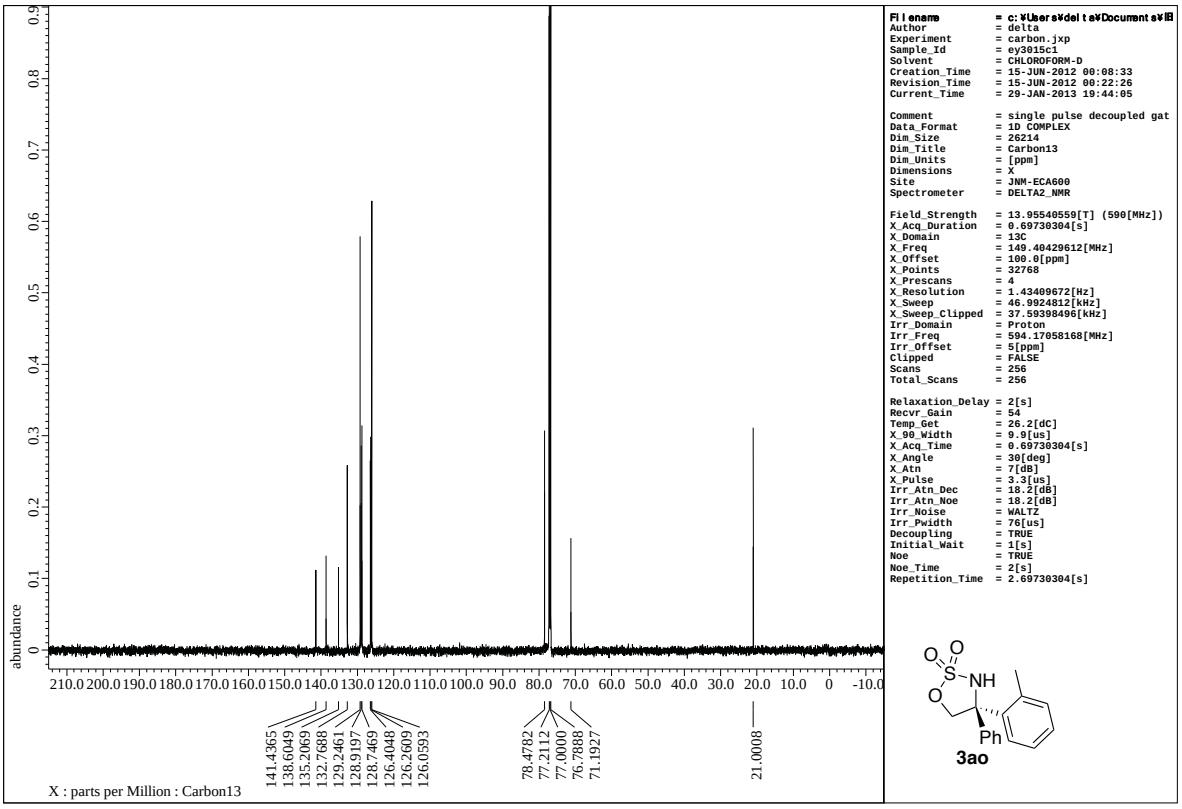
abundance
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
141.4365 138.6049 135.2069 132.7688 129.2461 128.9197 128.7469 126.4048 126.2609 126.0593
78.4782 77.2112 77.0000 76.7888 71.1927
21.0008
X : parts per Million : Carbon13
Filename = c:¥Users¥delta¥Documents¥
Author = delta
Experiment = carbon.jxp
Sample_Id = ey3015c1
Solvent = CHLOROFORM-D
Creation_Time = 15-JUN-2012 00:08:33
Revision_Time = 15-JUN-2012 00:22:26
Current_Time = 29-JAN-2013 19:44:05
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 = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 54
Temp_Get = 26.2[dC]
X_90_Width = 9.9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 7[dB]
X_Pulse = 3.3[us]
Irr_Atn_Dec = 18.2[dB]
Irr_Atn_Noe = 18.2[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]
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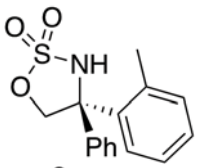

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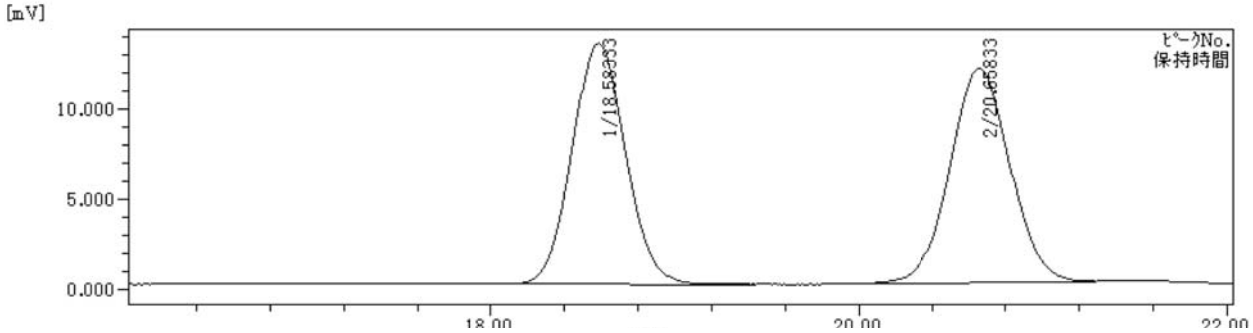
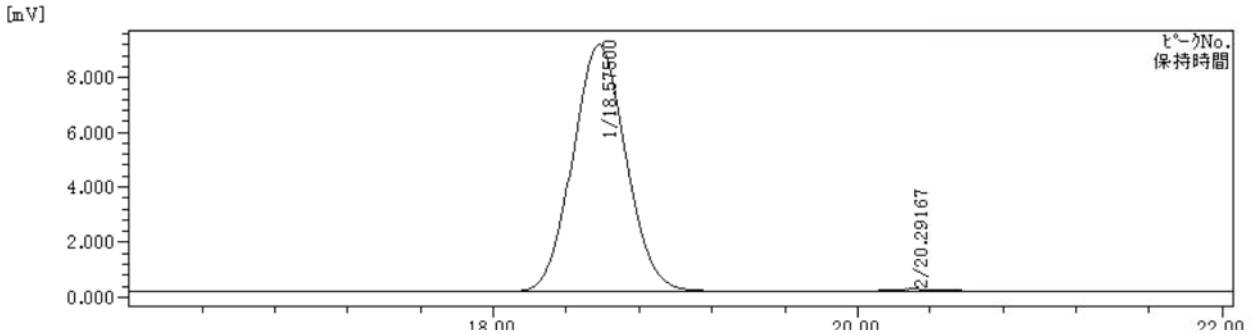


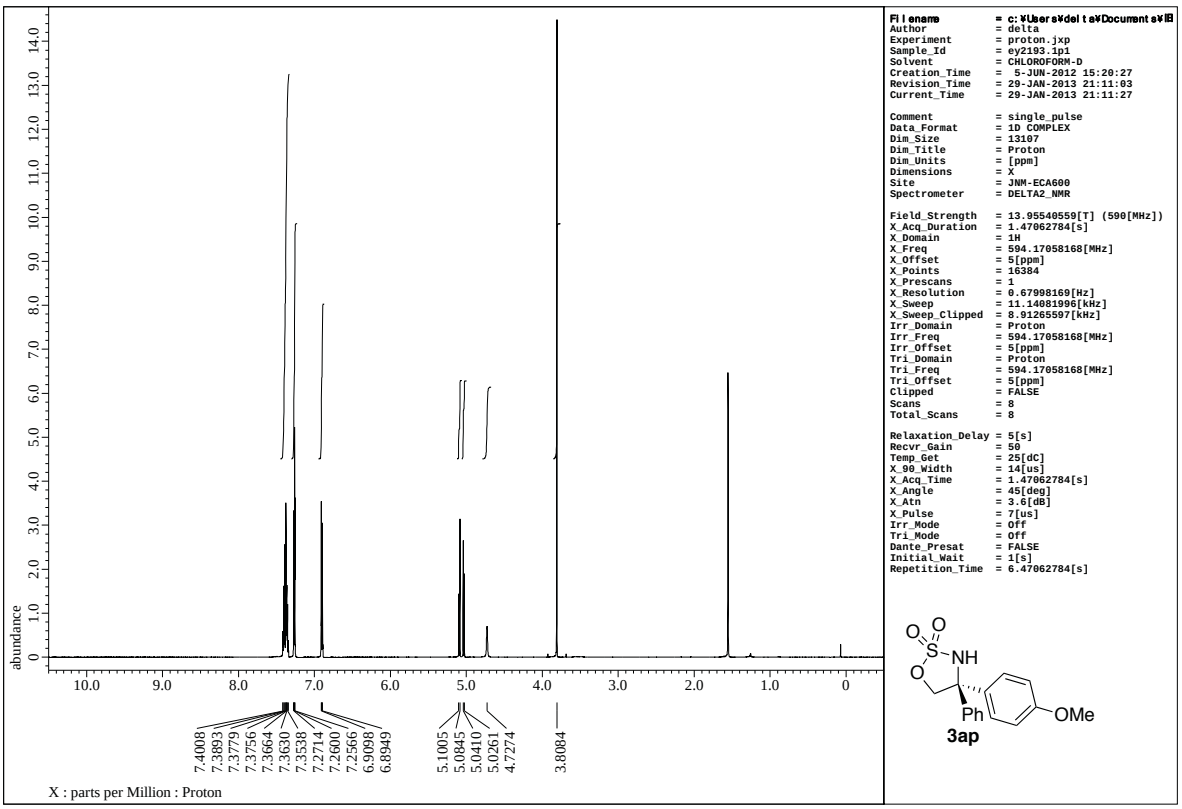
time (min)

Pk#	Retention Time	Area	Area (%)
1	18.583	268.825	49.823
2	20.658	270.738	50.177



time (min)

Pk#	Retention Time	Area	Area (%)
1	18.575	182.274	98.868
2	20.292	2.087	1.132


abundance
X : parts per Million : Proton
7.4008
7.3893
7.3779
7.3756
7.3664
7.3630
7.3538
7.2714
7.2600
7.2566
6.9098
6.8949
5.1005
5.0845
5.0410
5.0261
4.7274
3.8084
Filename = c:¥Users¥delta¥Documents¥NMR
Author = delta
Experiment = proton.jxp
Sample_Id = ey2193.1p1
Solvent = CHLOROFORM-D
Creation_Time = 5-JUN-2012 15:20:27
Revision_Time = 29-JAN-2013 21:11:03
Current_Time = 29-JAN-2013 21:11:27
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 = 50
Temp_Get = 25[dC]
X_90_Width = 14[us]
X_Acq_Time = 1.47062784[s]
X_Angle = 45[deg]
X_Atn = 3.6[dB]
X_Pulse = 7[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.47062784[s]
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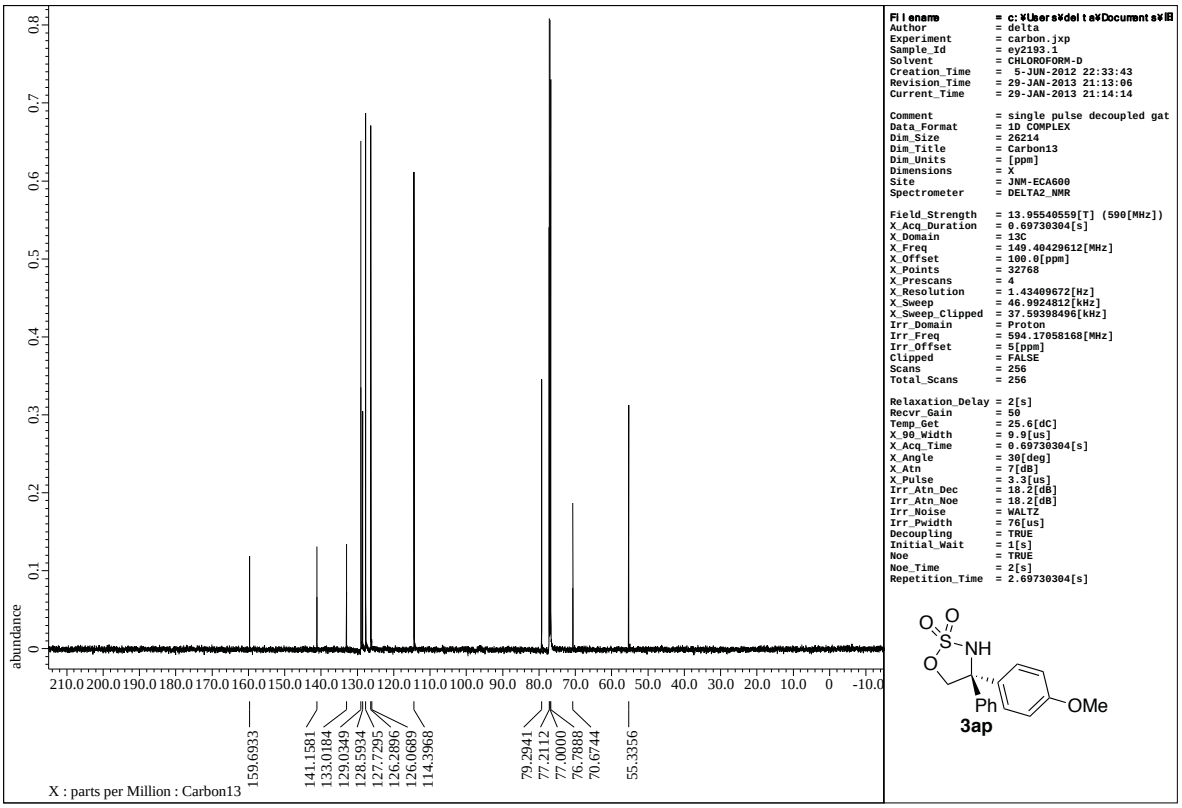
abundance
X : parts per Million : Carbon13
159.6933
141.1581
133.0184
129.0349
128.5934
127.7295
126.2896
126.0689
114.3968
79.2941
77.2112
77.0000
76.7888
70.6744
55.3356
Filename = c:¥Users¥delta¥Documents¥NMR
Author = delta
Experiment = carbon.jxp
Sample_Id = ey2193.1
Solvent = CHLOROFORM-D
Creation_Time = 5-JUN-2012 22:33:43
Revision_Time = 29-JAN-2013 21:13:06
Current_Time = 29-JAN-2013 21:14:14
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 = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 25.6[dC]
X_90_Width = 9.9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 7[dB]
X_Pulse = 3.3[us]
Irr_Atn_Dec = 18.2[dB]
Irr_Atn_Noe = 18.2[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]
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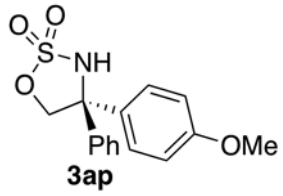
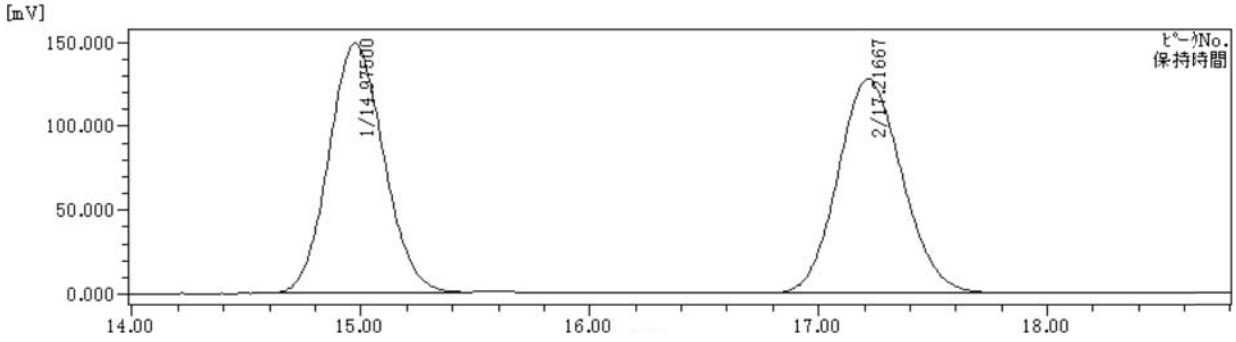
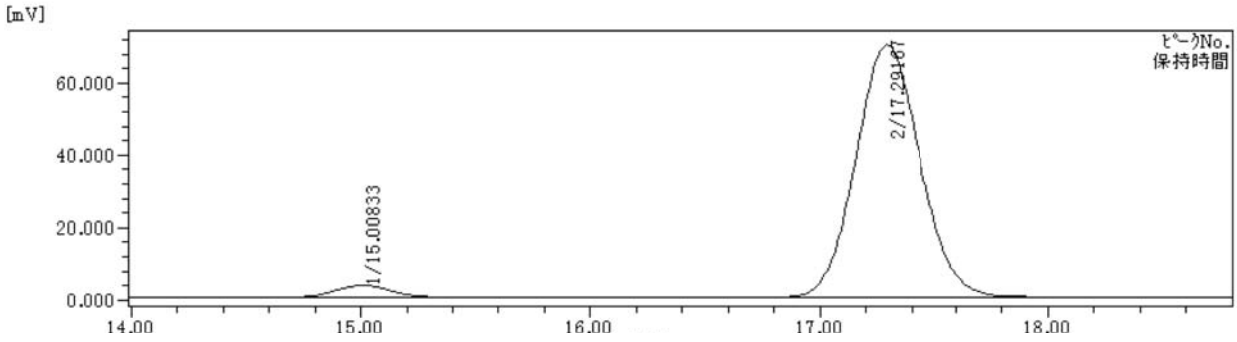


3ap



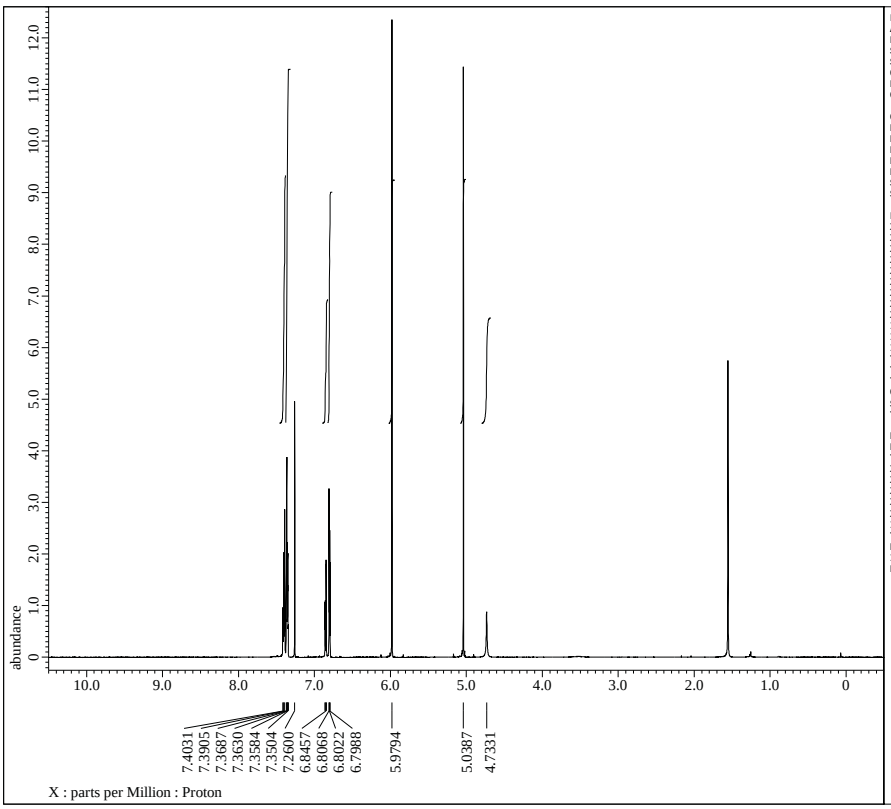
time (min)

Pk#	Retention Time	Area	Area (%)
1	14.975	2471.009	49.708
2	17.217	2500.057	50.292



time (min)

Pk#	Retention Time	Area	Area (%)
1	15.008	55.130	3.871
2	17.292	1369.055	96.129

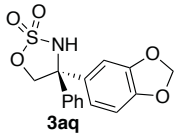



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = ey2193.2p1
Solvent           = CHLOROFORM-D
Creation_Time     =  5-JUN-2012 15:27:43
Revision_Time     = 30-JAN-2013 13:33:38
Current_Time      = 30-JAN-2013 13:33:59

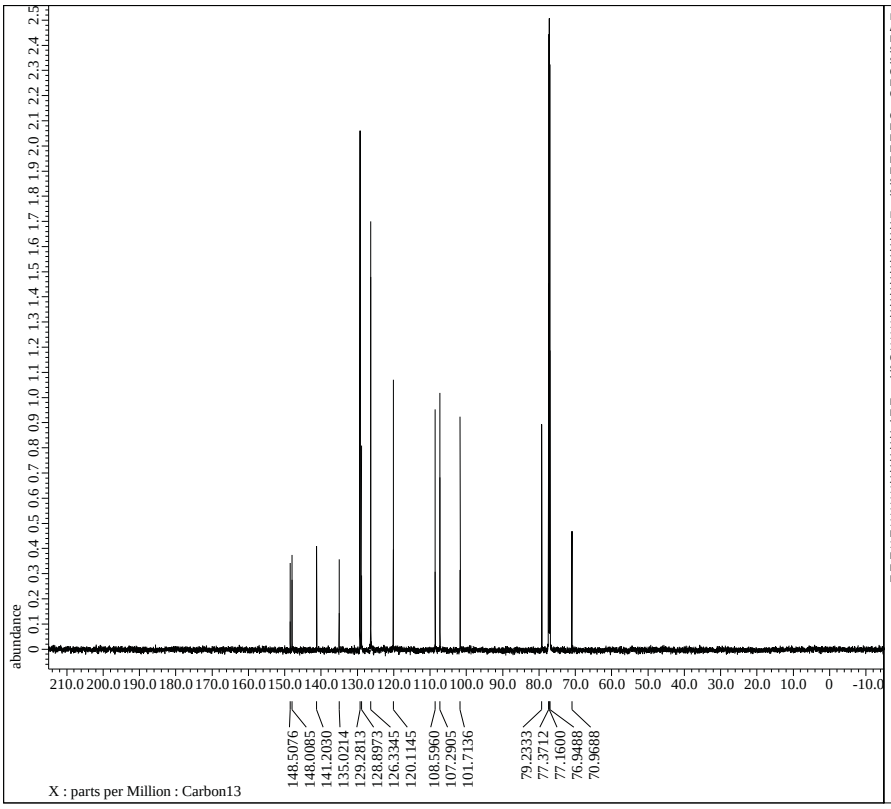
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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          = 25[dC]
X_90_Width        = 14[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3.6[dB]
X_Pulse           = 7[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



3aq

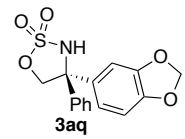



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = ey2193.2
Solvent           = CHLOROFORM-D
Creation_Time     =  5-JUN-2012 22:53:19
Revision_Time     =  5-JUN-2012 23:07:08
Current_Time      = 30-JAN-2013 13:34:38

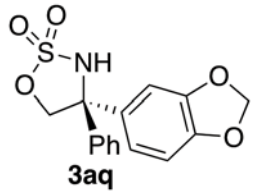
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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           = FALSE
Scans             = 256
Total_Scans       = 256

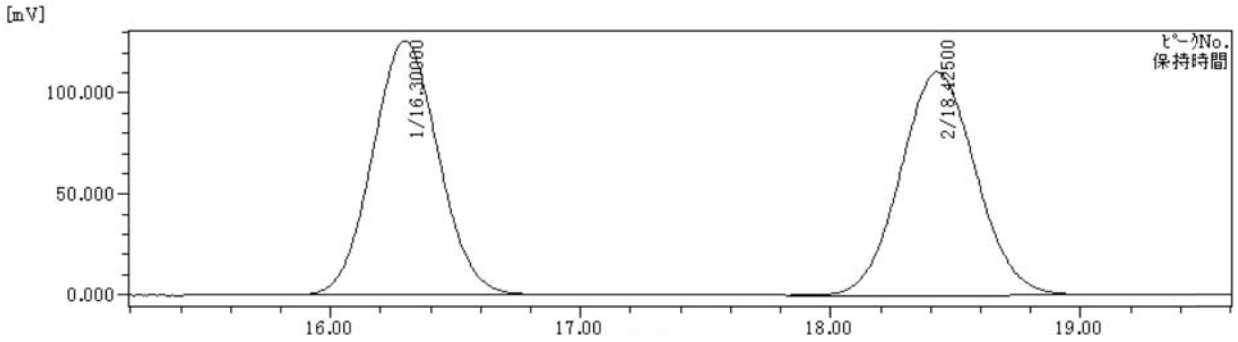
Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 25.7[dC]
X_90_Width        = 9.9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 7[dB]
X_Pulse           = 3.3[us]
Irr_Atn_Dec       = 18.2[dB]
Irr_Atn_Noe       = 18.2[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



3aq

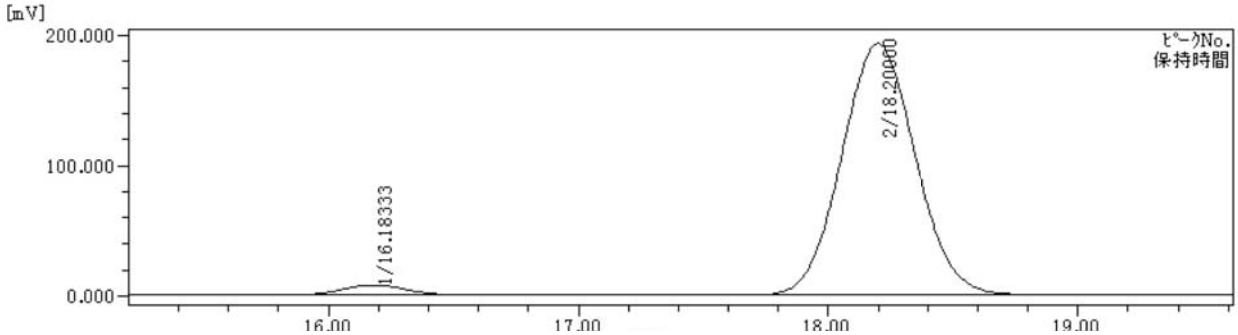


3aq



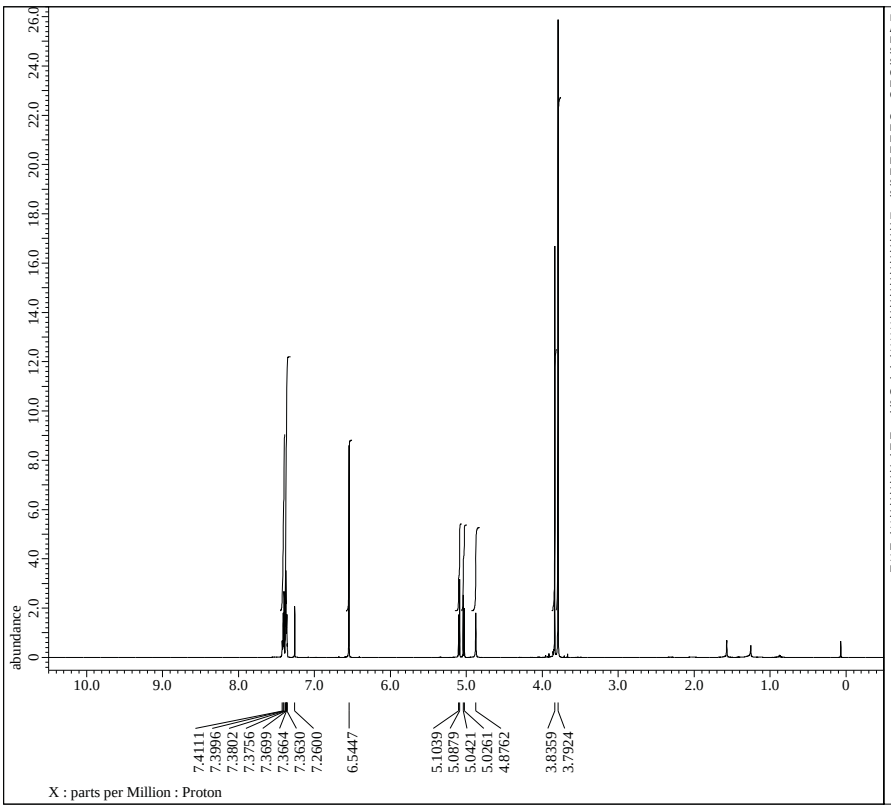
time (min)

Pk#	Retention Time	Area	Area (%)
1	16.300	2318.046	49.880
2	18.425	2329.213	50.120



time (min)

Pk#	Retention Time	Area	Area (%)
1	16.183	140.094	3.398
2	18.200	3983.220	96.602

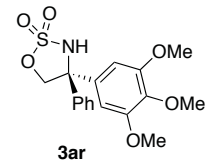



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = ey3014c1
Solvent           = CHLOROFORM-D
Creation_Time     = 14-JUN-2012 01:25:31
Revision_Time     = 30-JAN-2013 13:35:45
Current_Time      = 30-JAN-2013 13:36:14

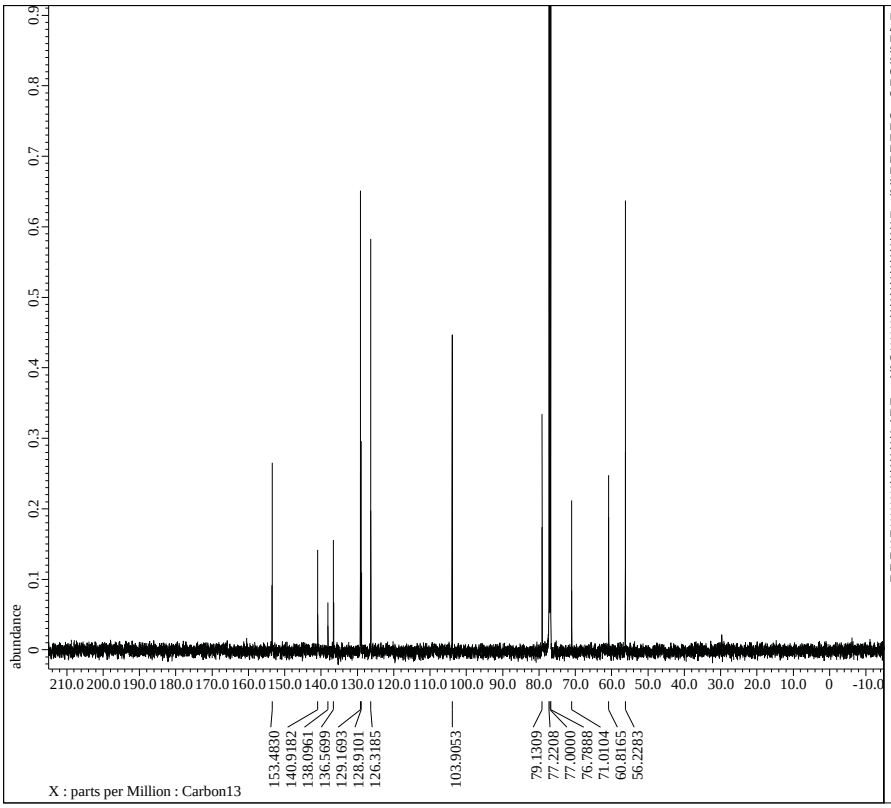
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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          = 25.4[dC]
X_90_Width        = 14[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3.6[dB]
X_Pulse           = 7[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



3ar

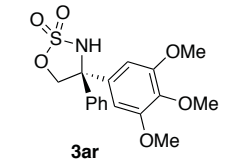



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = ey3014c1
Solvent           = CHLOROFORM-D
Creation_Time     = 14-JUN-2012 01:28:25
Revision_Time     = 30-JAN-2013 13:36:26
Current_Time      = 30-JAN-2013 13:37:03

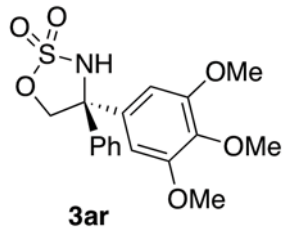
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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

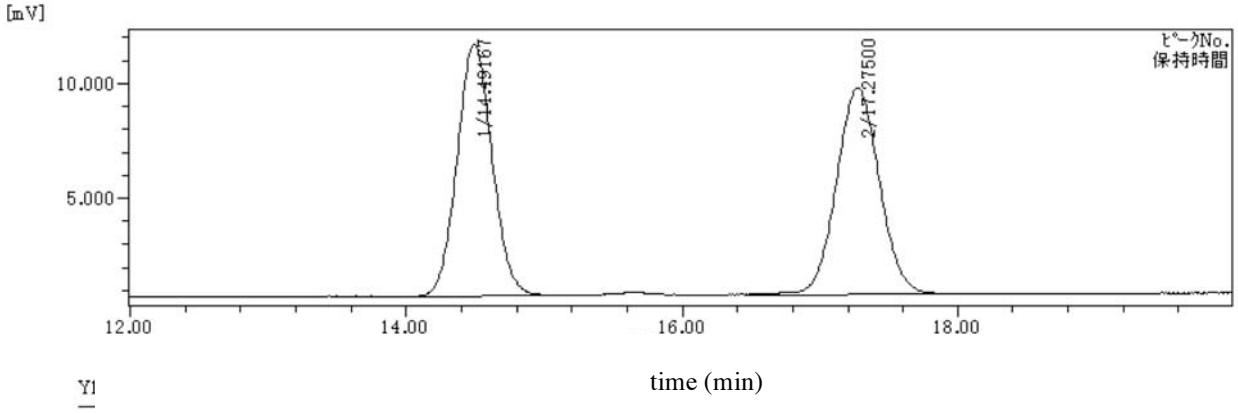
Relaxation_Delay  = 2[s]
Recvr_Gain        = 58
Temp_Get          = 26.1[dC]
X_90_Width        = 9.9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 7[dB]
X_Pulse           = 3.3[us]
Irr_Atn_Dec       = 18.2[dB]
Irr_Atn_Noe       = 18.2[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



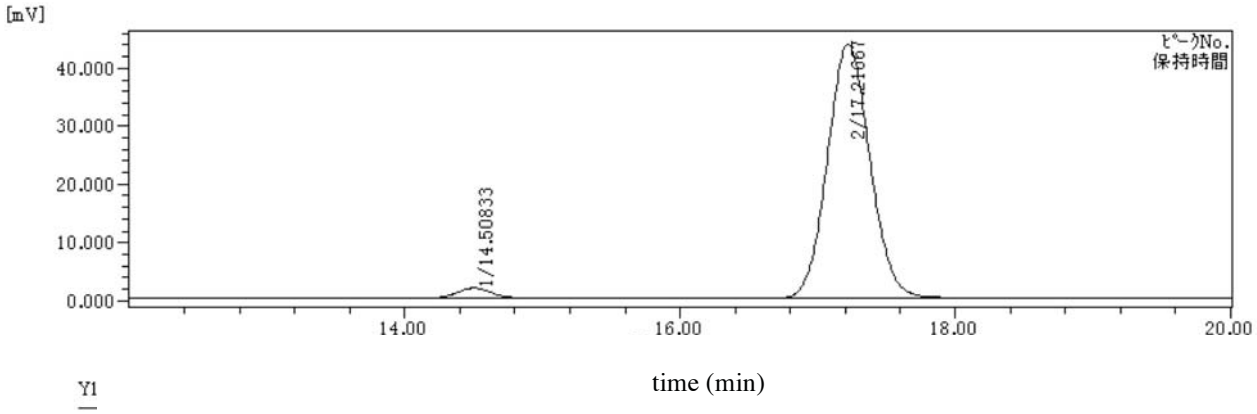
3ar



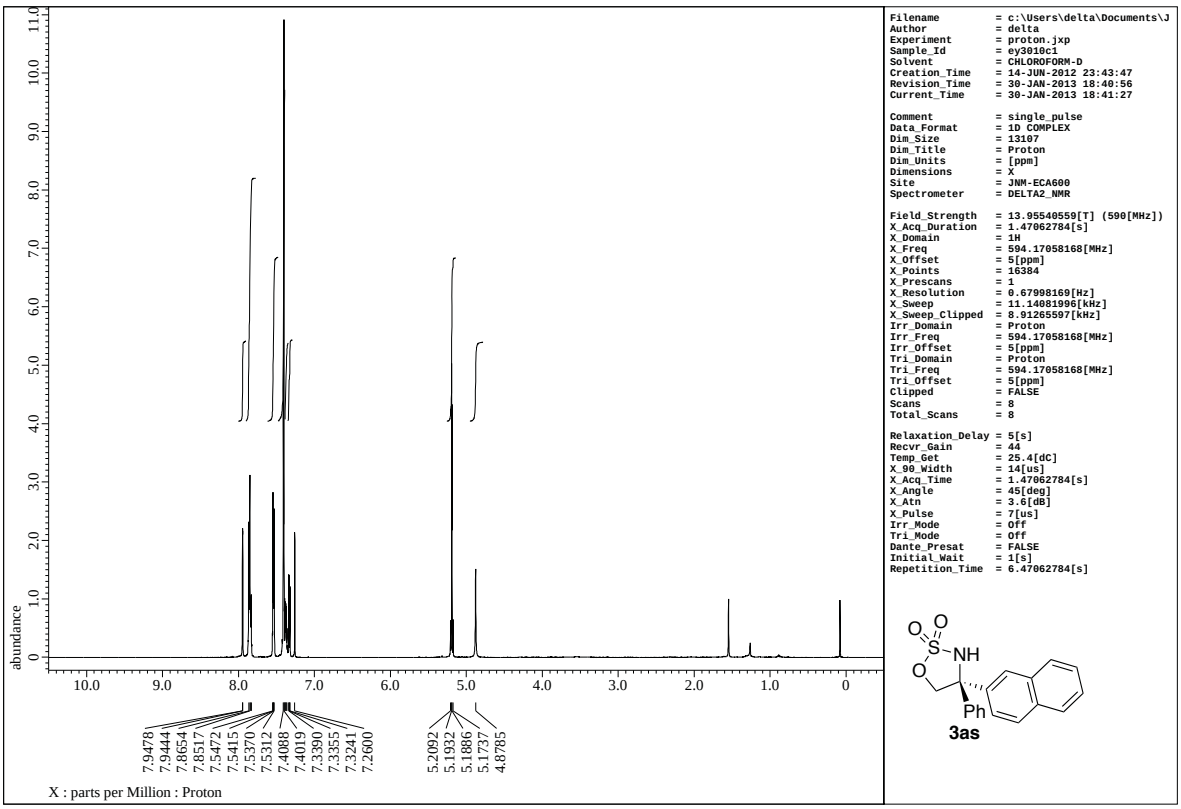
3ar

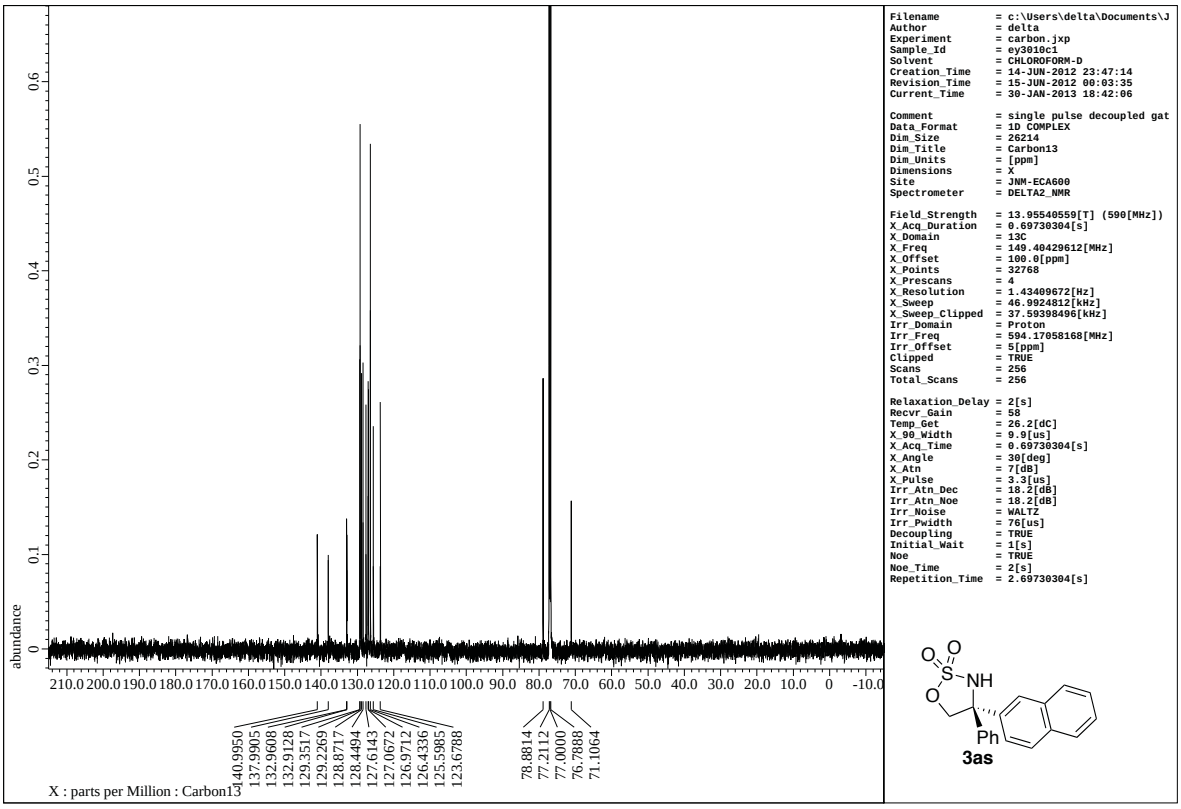


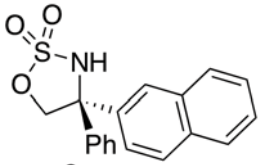
Pk#	Retention Time	Area	Area (%)
1	14.492	193.211	49.934
2	17.275	193.725	50.066



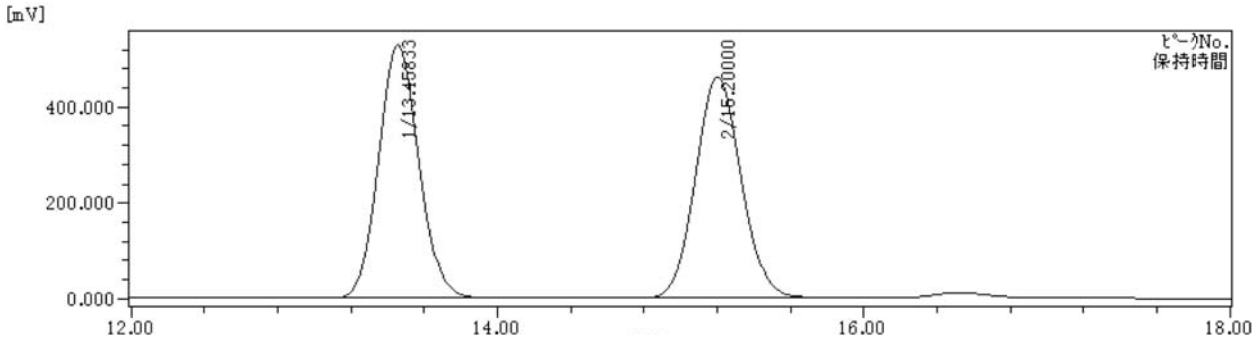
Pk#	Retention Time	Area	Area (%)
1	14.508	30.931	3.190
2	17.217	938.690	96.810


abundance
X : parts per Million : Proton
7.9478
7.9444
7.8654
7.8517
7.5472
7.5415
7.5370
7.5312
7.4088
7.4019
7.3390
7.3355
7.3241
7.2600
5.2092
5.1932
5.1886
5.1737
4.8785
Filename = c:\Users\delta\Documents\J
Author = delta
Experiment = proton.jxp
Sample_Id = ey3010c1
Solvent = CHLOROFORM-D
Creation_Time = 14-JUN-2012 23:43:47
Revision_Time = 30-JAN-2013 18:40:56
Current_Time = 30-JAN-2013 18:41:27
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 = 44
Temp_Get = 25.4[dC]
X_90_Width = 14[us]
X_Acq_Time = 1.47062784[s]
X_Angle = 45[deg]
X_Atn = 3.6[dB]
X_Pulse = 7[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.47062784[s]
NH
Ph
3as


abundance
X : parts per Million : Carbon13
140.9950
137.9905
132.9608
132.9128
129.3517
129.2269
128.8717
128.4494
127.6143
127.0672
126.9712
126.4336
125.5985
123.6788
78.8814
77.2112
77.0000
76.7888
71.1064
Filename = c:\Users\delta\Documents\J
Author = delta
Experiment = carbon.jxp
Sample_Id = ey3010c1
Solvent = CHLOROFORM-D
Creation_Time = 14-JUN-2012 23:47:14
Revision_Time = 15-JUN-2012 00:03:35
Current_Time = 30-JAN-2013 18:42:06
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 = 58
Temp_Get = 26.2[dC]
X_90_Width = 9.9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 7[dB]
X_Pulse = 3.3[us]
Irr_Atn_Dec = 18.2[dB]
Irr_Atn_Noe = 18.2[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]
NH
Ph
3as

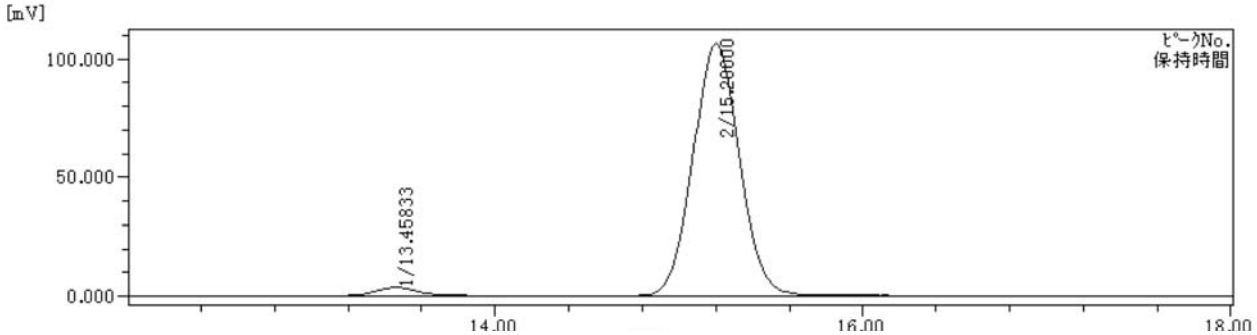


3as



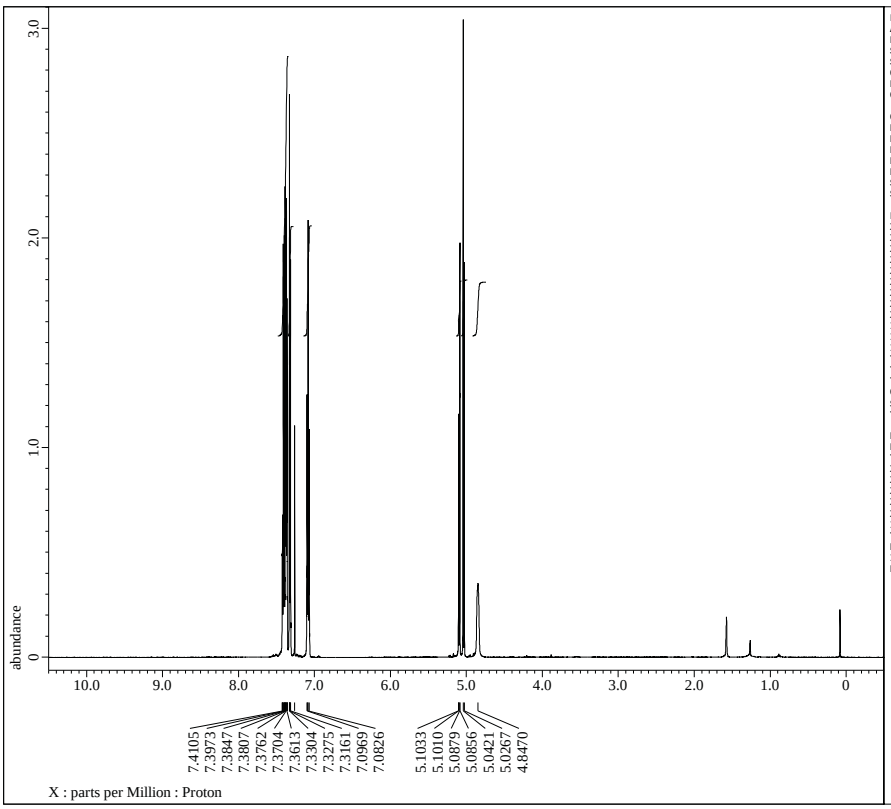
time (min)

Pk#	Retention Time	Area	Area (%)
1	13.458	8139.112	50.102
2	15.200	8105.957	49.898



time (min)

Pk#	Retention Time	Area	Area (%)
1	13.458	54.400	2.852
2	15.200	1852.954	97.148

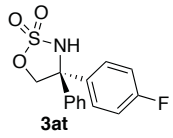



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = ey3025c1
Solvent           = CHLOROFORM-D
Creation_Time     = 21-JUN-2012 01:12:31
Revision_Time     = 30-JAN-2013 18:43:29
Current_Time      = 30-JAN-2013 18:43:46

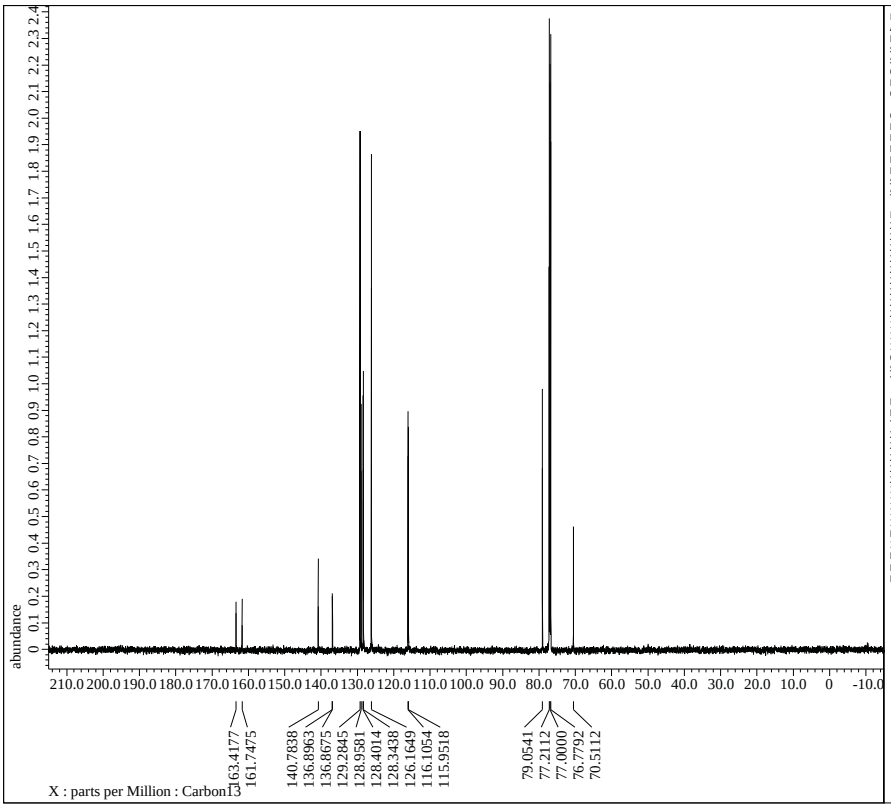
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 2.94125568[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.33999084[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          = 25.6[dC]
X_90_Width        = 14[us]
X_Acq_Time        = 2.94125568[s]
X_Angle           = 45[deg]
X_Atn             = 3.6[dB]
X_Pulse           = 7[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.94125568[s]
```



3at

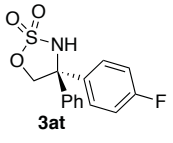



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = ey3025c1
Solvent           = CHLOROFORM-D
Creation_Time     = 21-JUN-2012 01:20:40
Revision_Time     = 17-JAN-2013 16:10:20
Current_Time      = 30-JAN-2013 18:47:39

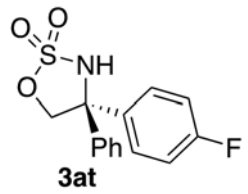
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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           = FALSE
Scans             = 256
Total_Scans       = 256

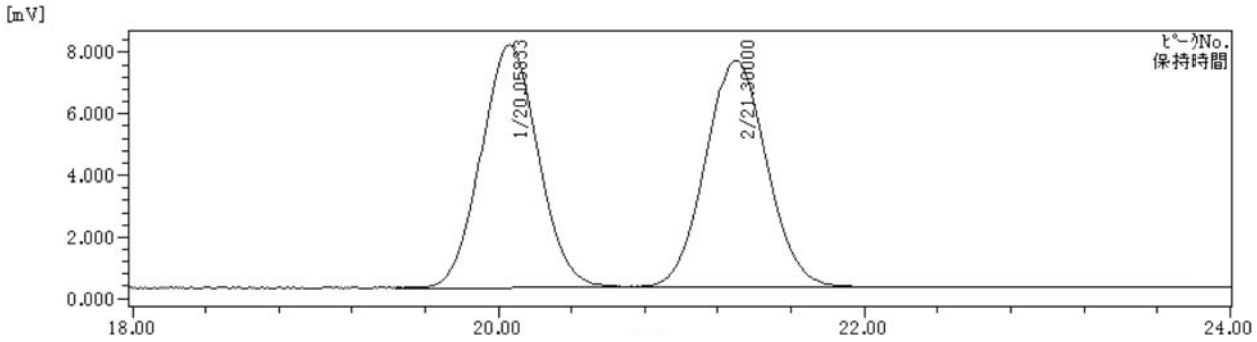
Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 26.2[dC]
X_90_Width        = 9.9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 7[dB]
X_Pulse           = 3.3[us]
Irr_Atn_Dec       = 18.2[dB]
Irr_Atn_Noe       = 18.2[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



3at

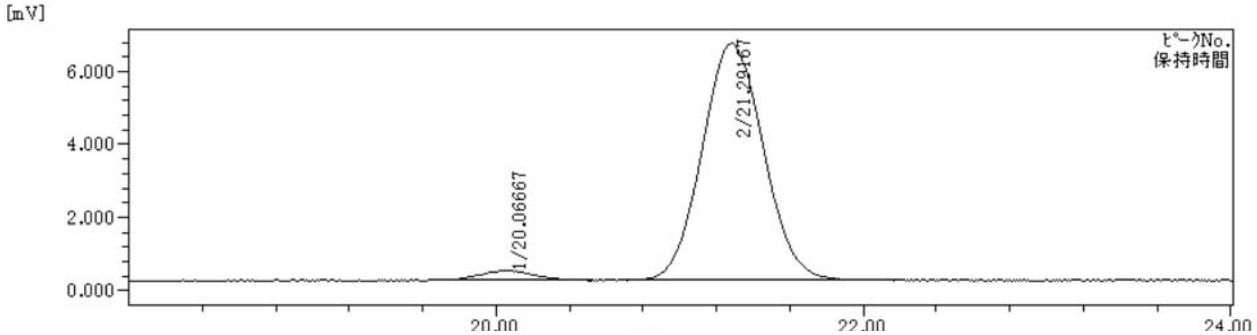


3at



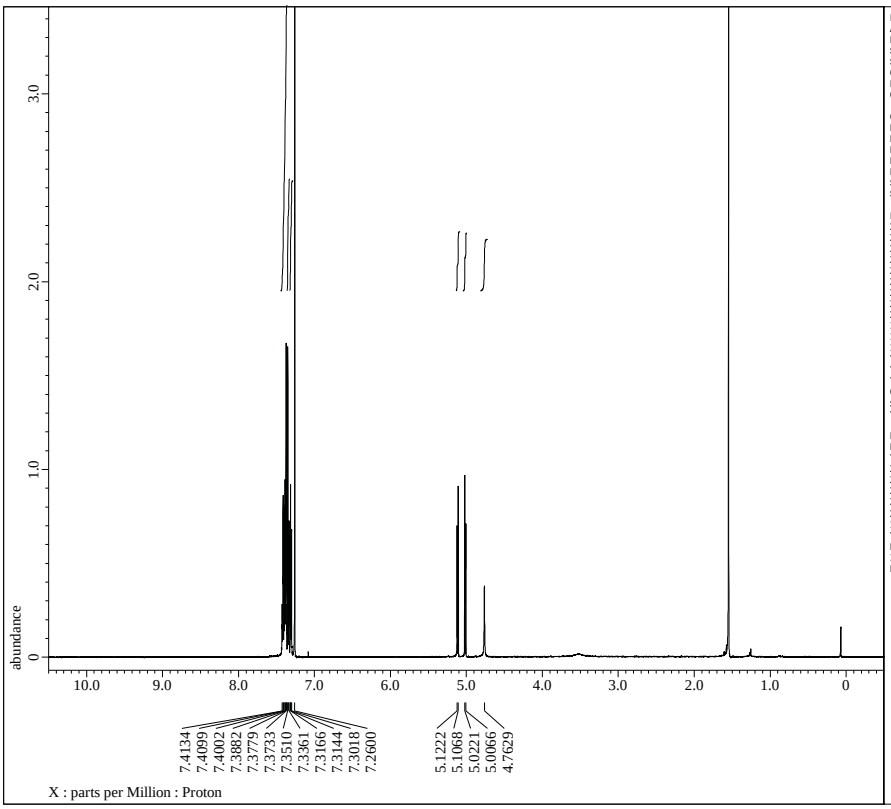
time (min)

Pk#	Retention Time	Area	Area (%)
1	20.058	170.814	50.085
2	21.300	170.237	49.915



time (min)

Pk#	Retention Time	Area	Area (%)
1	20.067	5.366	3.446
2	21.292	150.352	96.554

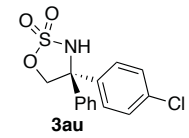



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = ey3027c1
Solvent           = CHLOROFORM-D
Creation_Time     = 22-JUN-2012 00:43:05
Revision_Time     = 30-JAN-2013 18:49:46
Current_Time      = 30-JAN-2013 18:50:07

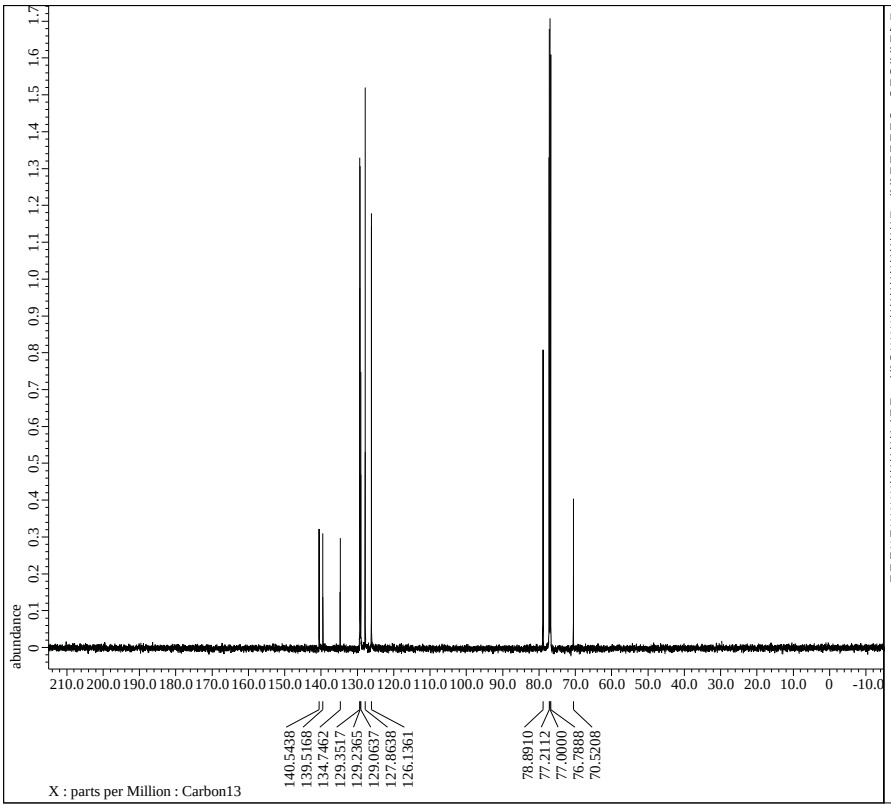
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 2.94125568[s]
X_Domain          = 1H
X_Freq            = 594.17058168[MHz]
X_Offset          = 5[ppm]
X_Points          = 32768
X_Prescans        = 1
X_Resolution      = 0.33999084[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        = 56
Temp_Get          = 25.5[dC]
X_90_Width        = 14[us]
X_Acq_Time        = 2.94125568[s]
X_Angle           = 45[deg]
X_Atn             = 3.6[dB]
X_Pulse           = 7[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.94125568[s]
```



3au

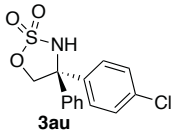



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = ey3027c1
Solvent           = CHLOROFORM-D
Creation_Time     = 22-JUN-2012 00:51:32
Revision_Time     = 22-JUN-2012 01:06:06
Current_Time      = 30-JAN-2013 18:50:40

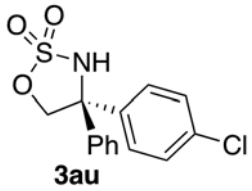
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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

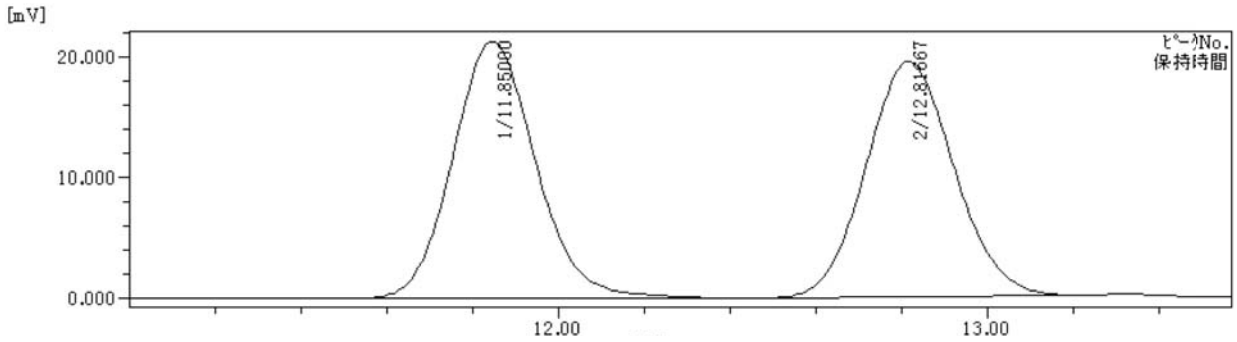
Relaxation_Delay  = 2[s]
Recvr_Gain        = 58
Temp_Get          = 26.1[dC]
X_90_Width        = 9.9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 7[dB]
X_Pulse           = 3.3[us]
Irr_Atn_Dec       = 18.2[dB]
Irr_Atn_Noe       = 18.2[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



3au

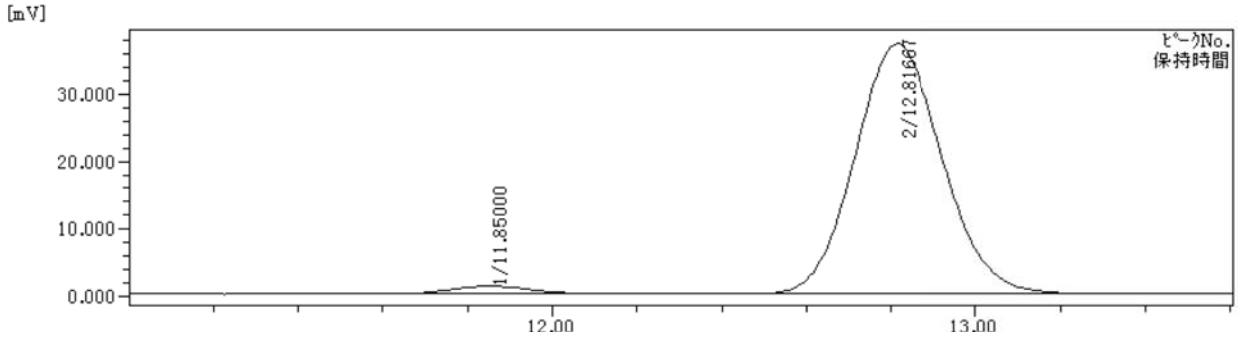


3au



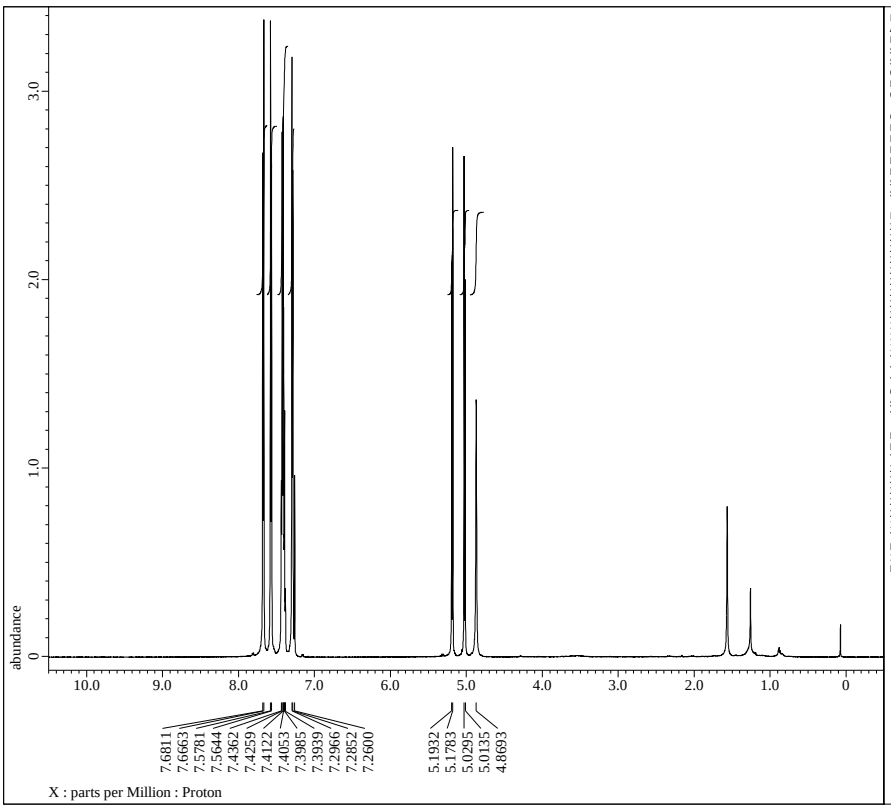
time (min)

Pk#	Retention Time	Area	Area (%)
1	11.850	286.746	50.755
2	12.817	278.218	49.245



time (min)

Pk#	Retention Time	Area	Area (%)
1	11.850	16.315	2.959
2	12.817	535.050	97.041

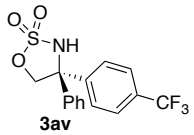



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = ey3007p1
Solvent           = CHLOROFORM-D
Creation_Time     = 30-NOV-2012 02:37:22
Revision_Time     = 30-JAN-2013 18:51:39
Current_Time      = 30-JAN-2013 18:52:07

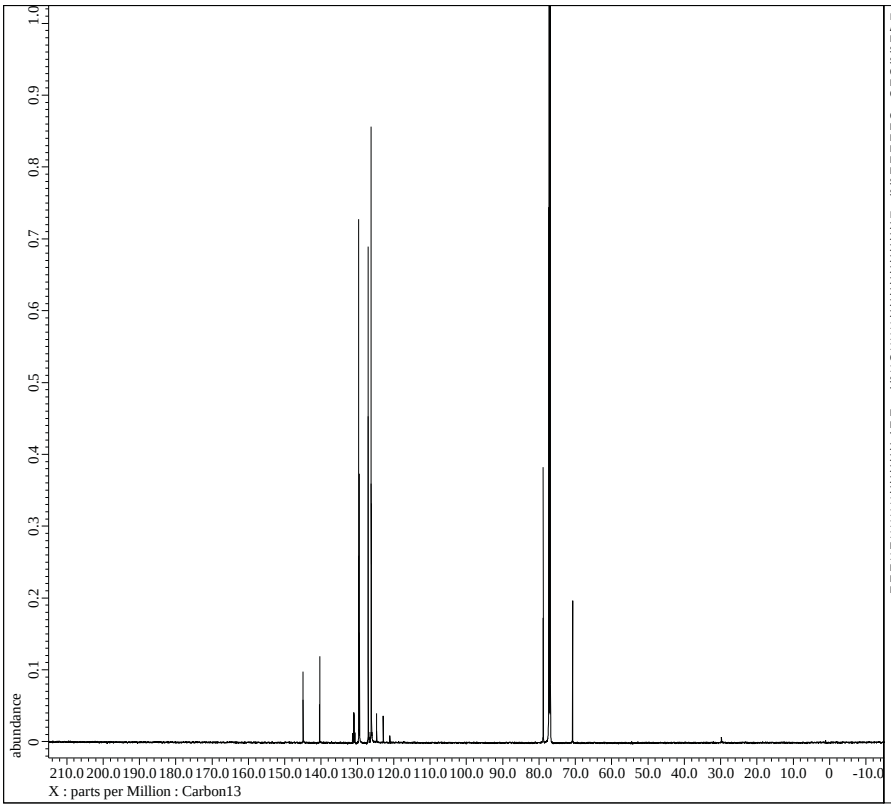
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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        = 44
Temp_Get          = 23.5[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]
```



3av

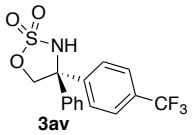



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = ey3007p1
Solvent           = CHLOROFORM-D
Creation_Time     = 30-NOV-2012 02:58:07
Revision_Time     = 30-NOV-2012 09:14:35
Current_Time      = 30-JAN-2013 18:53:01

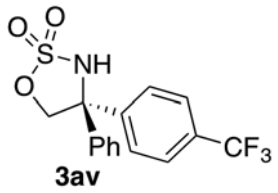
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 0[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
Incomplete_Copy   = TRUE
Scans             = 8328
Total_Scans       = 8328

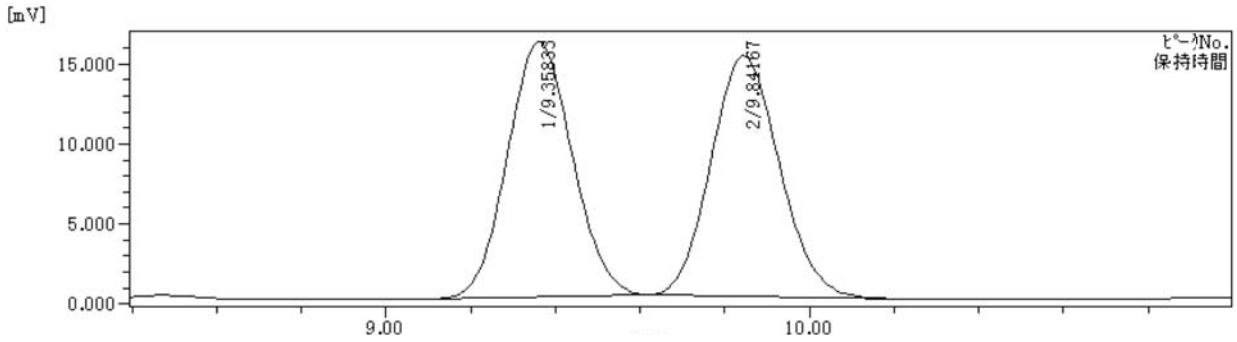
Relaxation_Delay = 2[s]
Recvr_Gain        = 56
Temp_Get          = 24.6[dC]
X_90_Width        = 0[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



3av

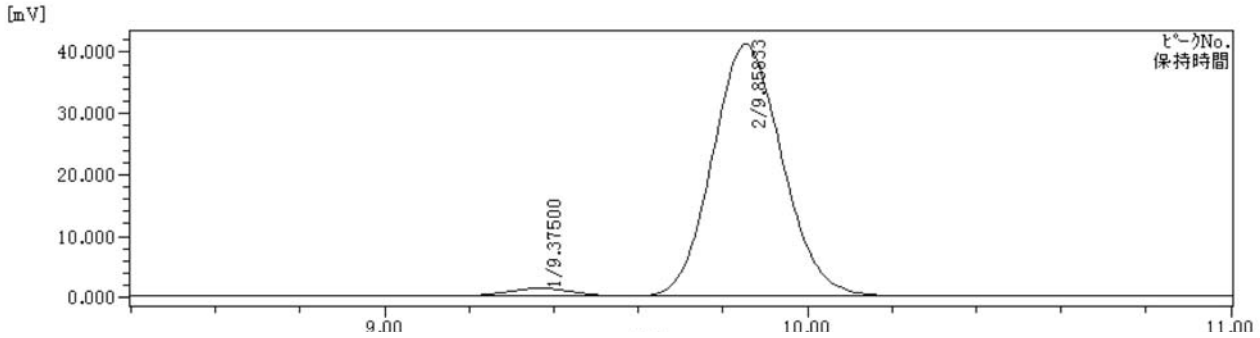


3av



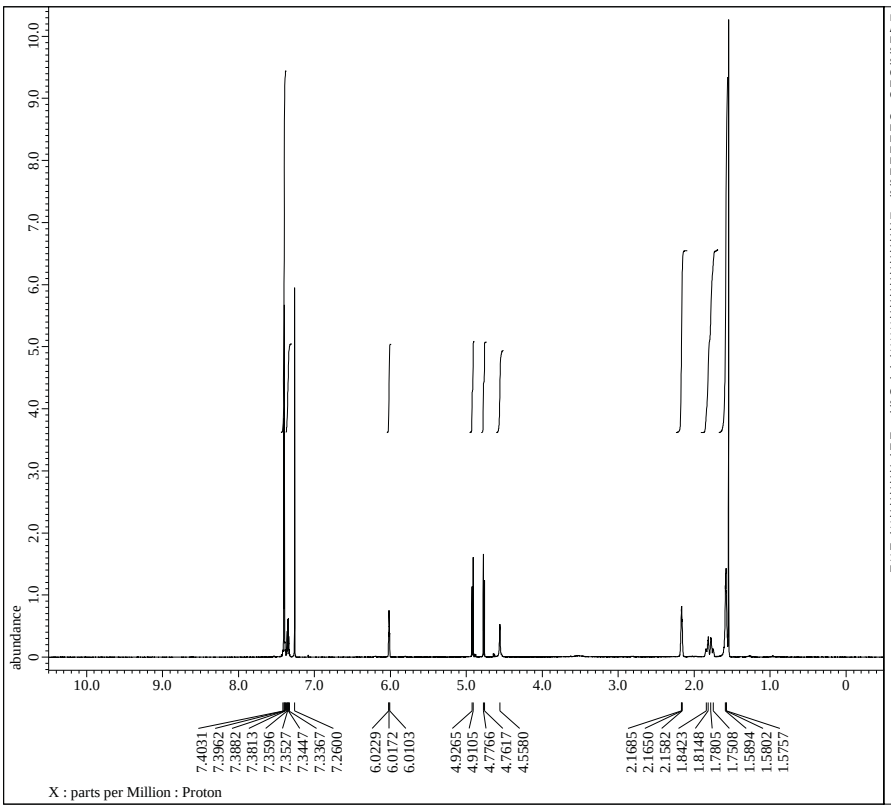
time (min)

Pk#	Retention Time	Area	Area (%)
1	9.358	168.930	50.075
2	9.842	168.425	49.925



time (min)

Pk#	Retention Time	Area	Area (%)
1	9.375	13.200	2.745
2	9.858	467.734	97.255

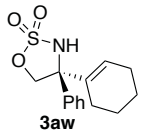



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = ey3042g1
Solvent           = CHLOROFORM-D
Creation_Time     =  5-JUL-2012 23:06:30
Revision_Time     = 31-JAN-2013 12:06:03
Current_Time      = 31-JAN-2013 12:06:08

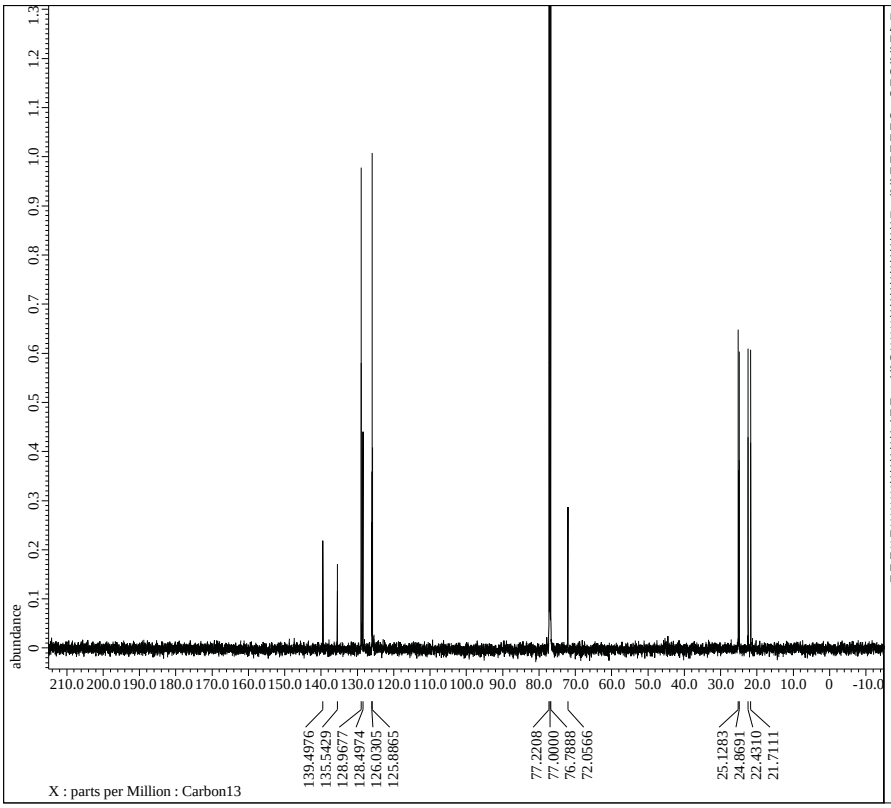
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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          = 25.6[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]
```



3aw

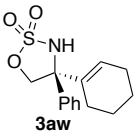



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = ey3042g1
Solvent           = CHLOROFORM-D
Creation_Time     =  5-JUL-2012 23:54:48
Revision_Time     =  6-JUL-2012 00:10:08
Current_Time      = 31-JAN-2013 12:06:48

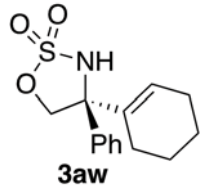
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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

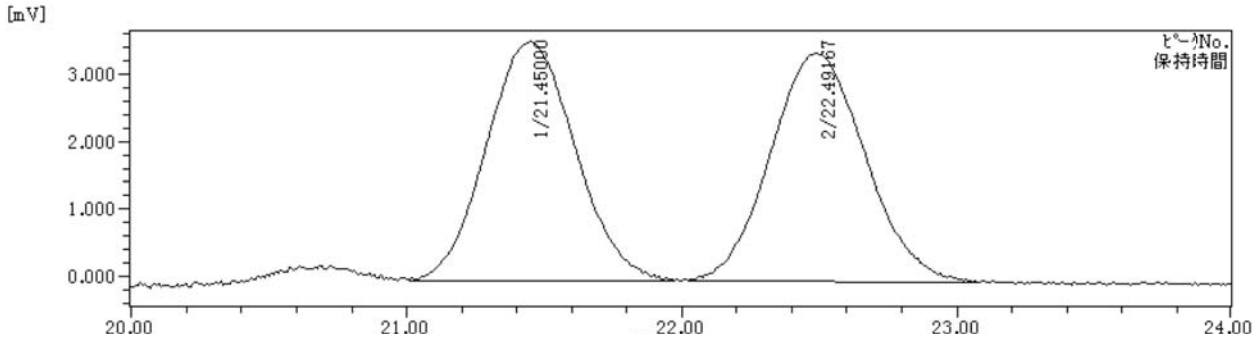
Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 26.3[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



3aw

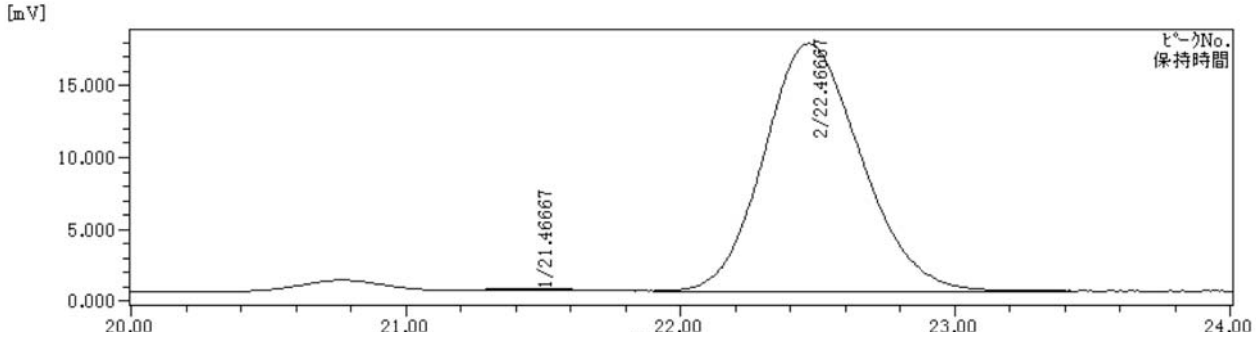


3aw



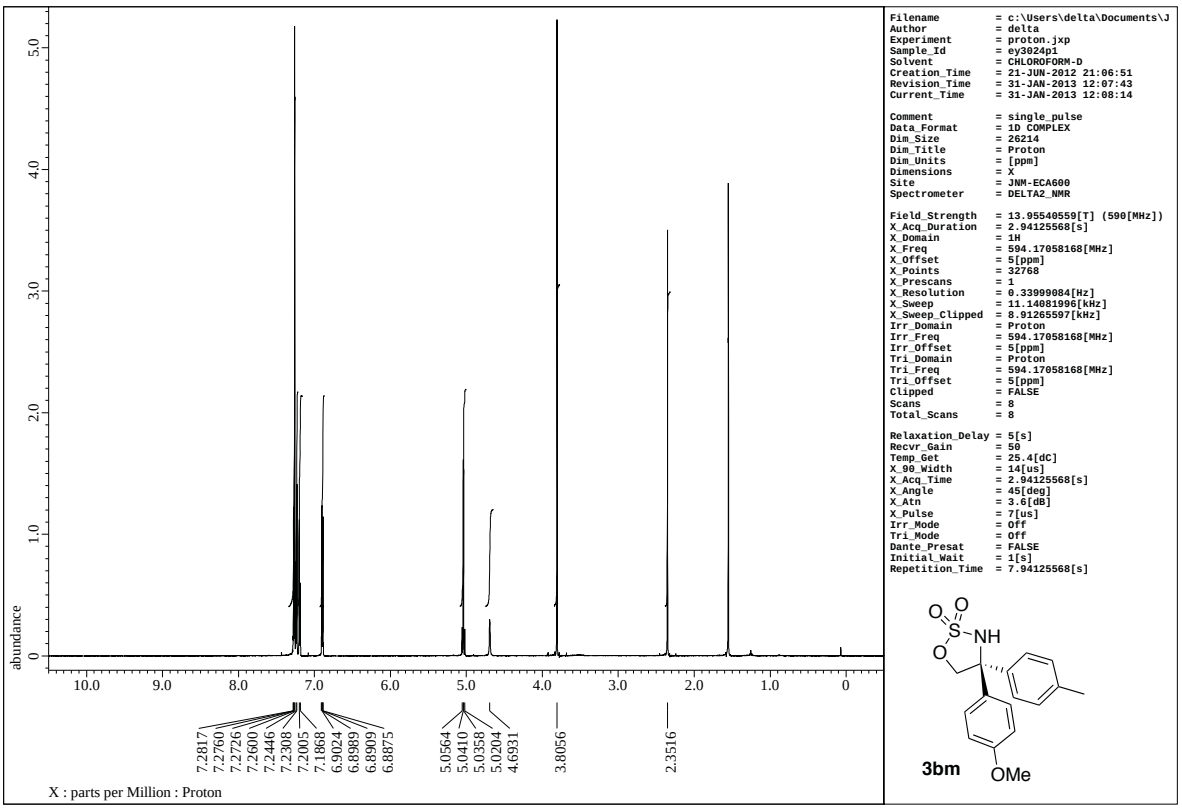
time (min)

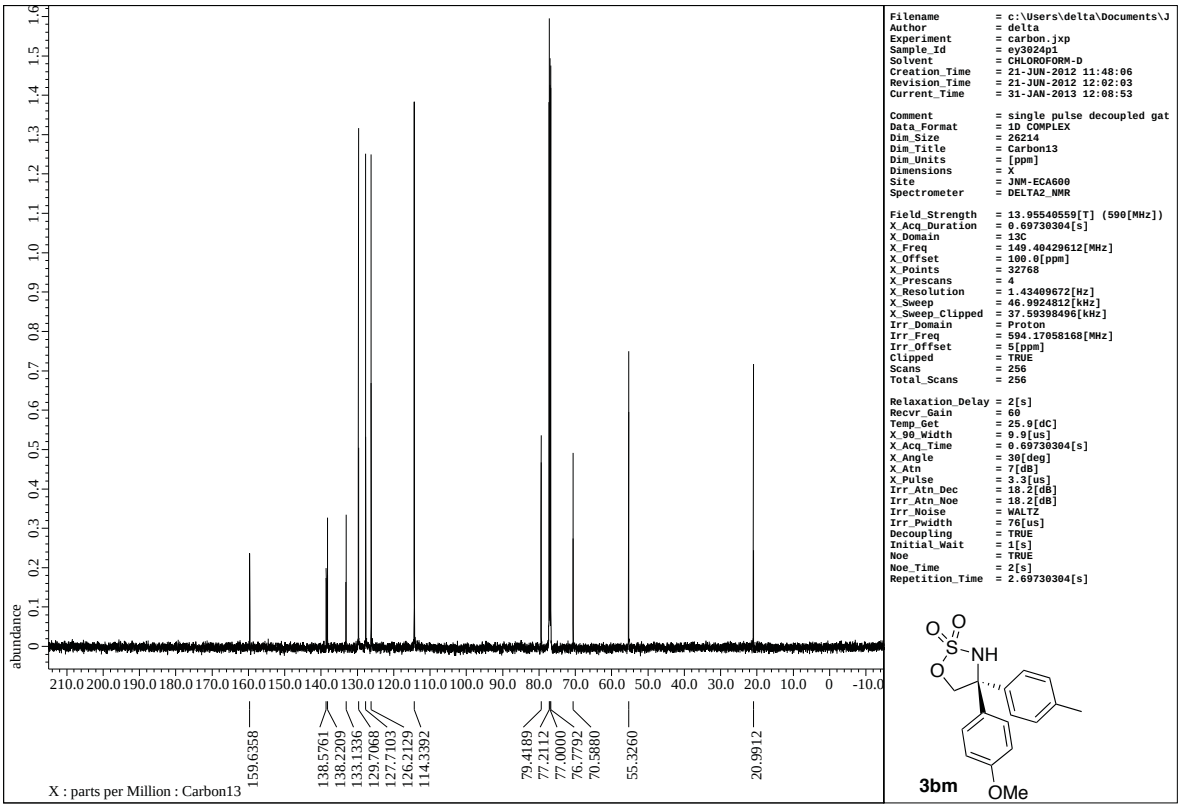
Pk#	Retention Time	Area	Area (%)
1	21.450	80.062	49.573
2	22.492	81.441	50.427

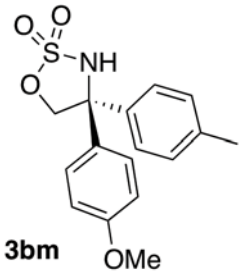


time (min)

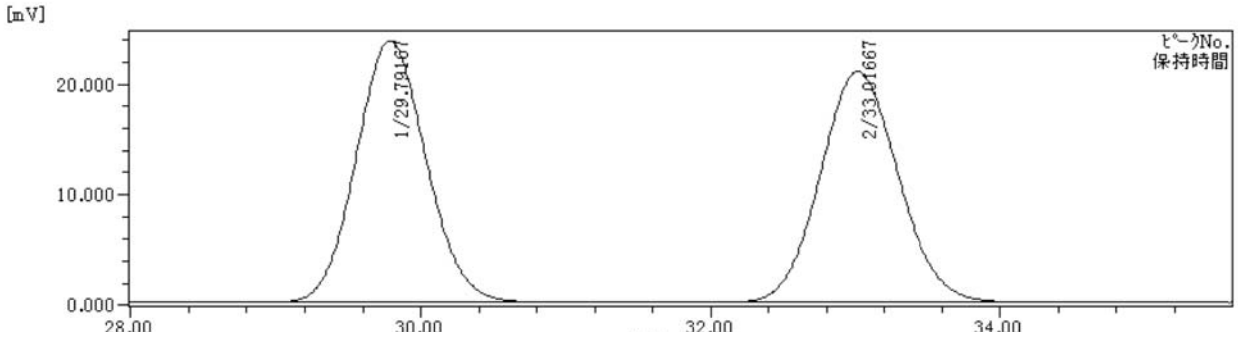
Pk#	Retention Time	Area	Area (%)
1	21.467	3.078	0.721
2	22.467	423.866	99.279


abundance
X : parts per Million : Proton
7.2817
7.2760
7.2726
7.2600
7.2446
7.2308
7.2005
7.1868
6.9024
6.8989
6.8909
6.8875
5.0564
5.0410
5.0358
5.0204
4.6931
3.8056
2.3516
Filename = c:\Users\delta\Documents\J
Author = delta
Experiment = proton.jxp
Sample_Id = ey3024p1
Solvent = CHLOROFORM-D
Creation_Time = 21-JUN-2012 21:06:51
Revision_Time = 31-JAN-2013 12:07:43
Current_Time = 31-JAN-2013 12:08:14
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 2.94125568[s]
X_Domain = 1H
X_Freq = 594.17058168[MHz]
X_Offset = 5[ppm]
X_Points = 32768
X_Prescans = 1
X_Resolution = 0.33999084[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 = 25.4[dC]
X_90_Width = 14[us]
X_Acq_Time = 2.94125568[s]
X_Angle = 45[deg]
X_Atn = 3.6[dB]
X_Pulse = 7[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 7.94125568[s]
3bm
OMe


abundance
X : parts per Million : Carbon13
159.6358
138.5761
138.2209
133.1336
129.7068
127.7103
126.2129
114.3392
79.4189
77.2112
77.0000
76.7792
70.5880
55.3260
20.9912
Filename = c:\Users\delta\Documents\J
Author = delta
Experiment = carbon.jxp
Sample_Id = ey3024p1
Solvent = CHLOROFORM-D
Creation_Time = 21-JUN-2012 11:48:06
Revision_Time = 21-JUN-2012 12:02:03
Current_Time = 31-JAN-2013 12:08:53
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 = 25.9[dC]
X_90_Width = 9.9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 7[dB]
X_Pulse = 3.3[us]
Irr_Atn_Dec = 18.2[dB]
Irr_Atn_Noe = 18.2[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]
3bm
OMe

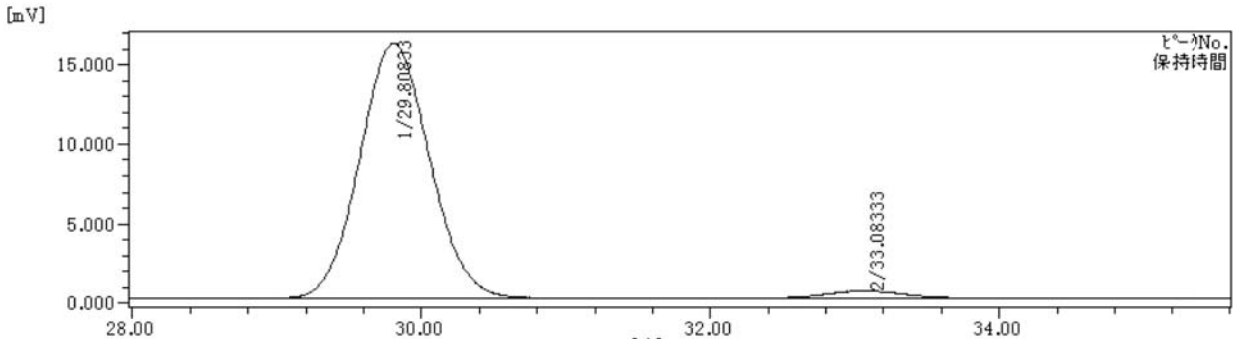


3bm



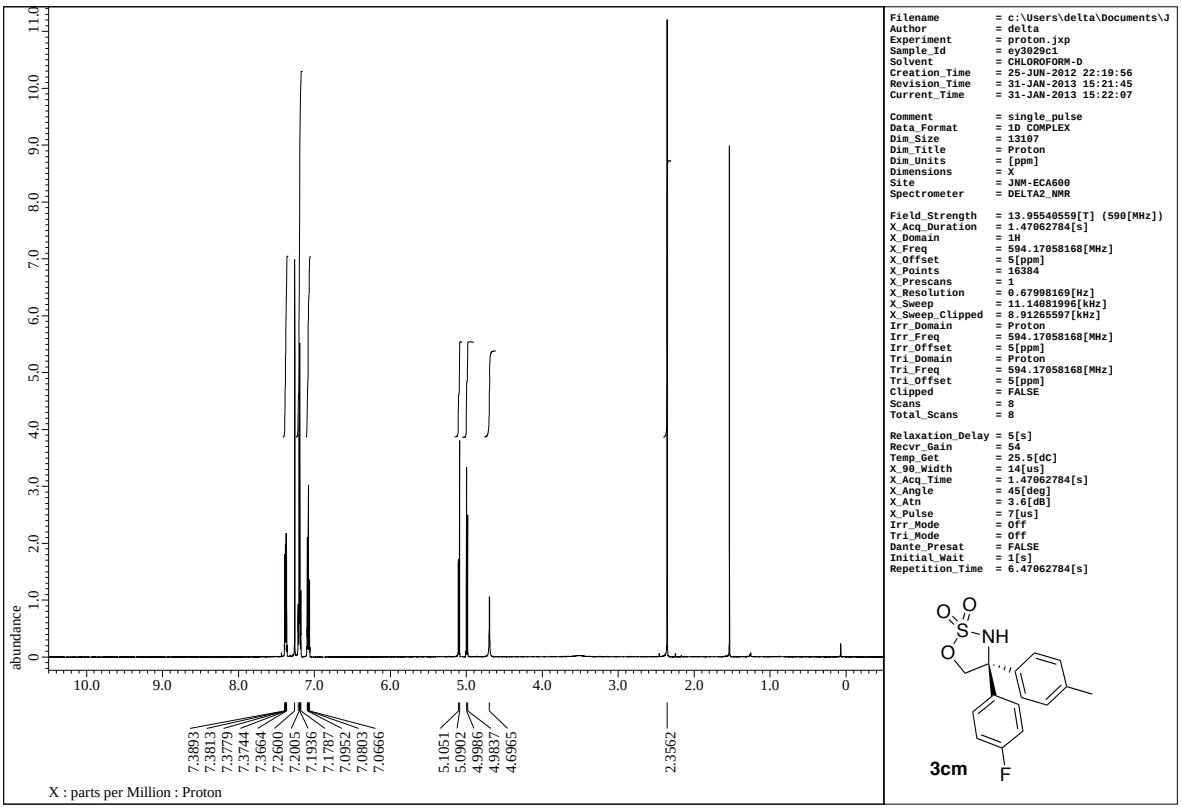
time (min)

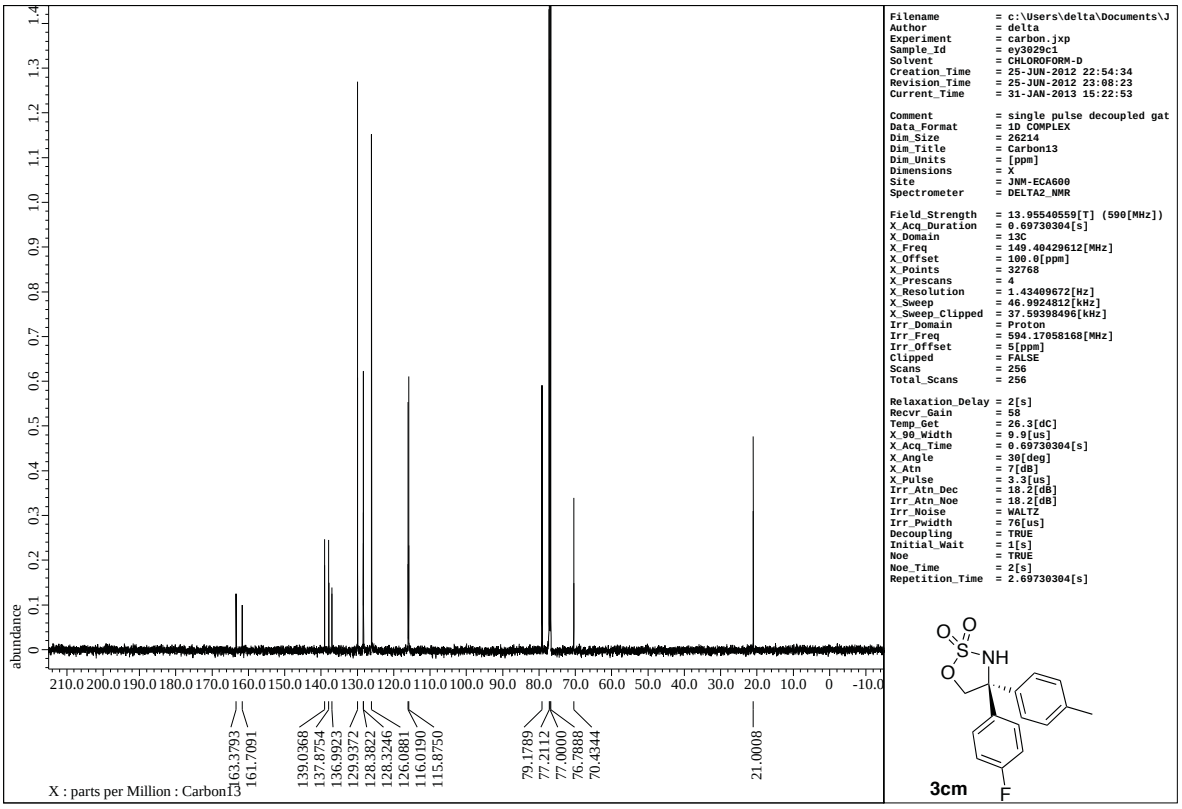
Pk#	Retention Time	Area	Area (%)
1	29.792	794.293	50.339
2	33.017	783.604	49.661

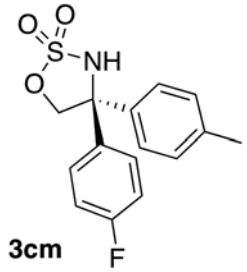


time (min)

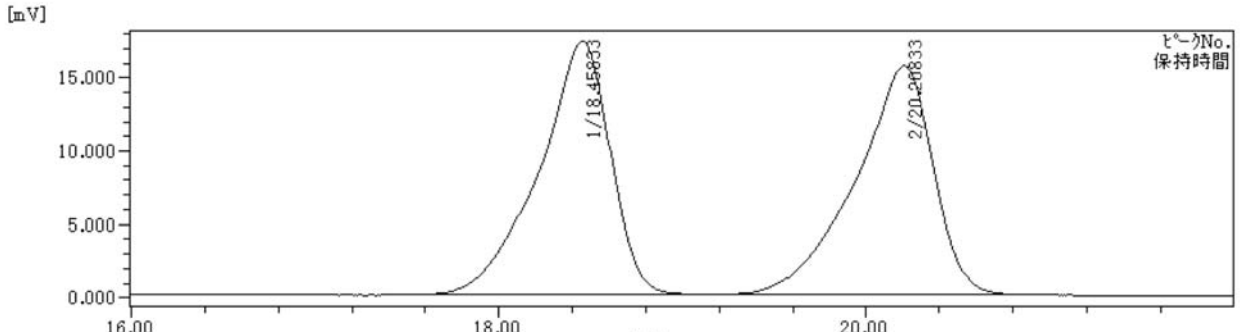
Pk#	Retention Time	Area	Area (%)
1	29.808	540.392	96.970
2	33.083	16.886	3.030


abundance
X : parts per Million : Proton
7.3893
7.3813
7.3779
7.3744
7.3664
7.2600
7.2005
7.1936
7.1787
7.0952
7.0803
7.0666
5.1051
5.0902
4.9986
4.9837
4.6965
2.3562
Filename = c:\Users\delta\Documents\J
Author = delta
Experiment = proton.jxp
Sample_Id = ey3029c1
Solvent = CHLOROFORM-D
Creation_Time = 25-JUN-2012 22:19:56
Revision_Time = 31-JAN-2013 15:21:45
Current_Time = 31-JAN-2013 15:22:07
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 = 54
Temp_Get = 25.5[dC]
X_90_Width = 14[us]
X_Acq_Time = 1.47062784[s]
X_Angle = 45[deg]
X_Atn = 3.6[dB]
X_Pulse = 7[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.47062784[s]
3cm


abundance
X : parts per Million : Carbon13
163.3793
161.7091
139.0368
137.8754
136.9923
129.9372
128.3822
128.3246
126.0881
116.0190
115.8750
79.1789
77.2112
77.0000
76.7888
70.4344
21.0008
Filename = c:\Users\delta\Documents\J
Author = delta
Experiment = carbon.jxp
Sample_Id = ey3029c1
Solvent = CHLOROFORM-D
Creation_Time = 25-JUN-2012 22:54:34
Revision_Time = 25-JUN-2012 23:08:23
Current_Time = 31-JAN-2013 15:22:53
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 = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 26.3[dC]
X_90_Width = 9.9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 7[dB]
X_Pulse = 3.3[us]
Irr_Atn_Dec = 18.2[dB]
Irr_Atn_Noe = 18.2[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]
3cm

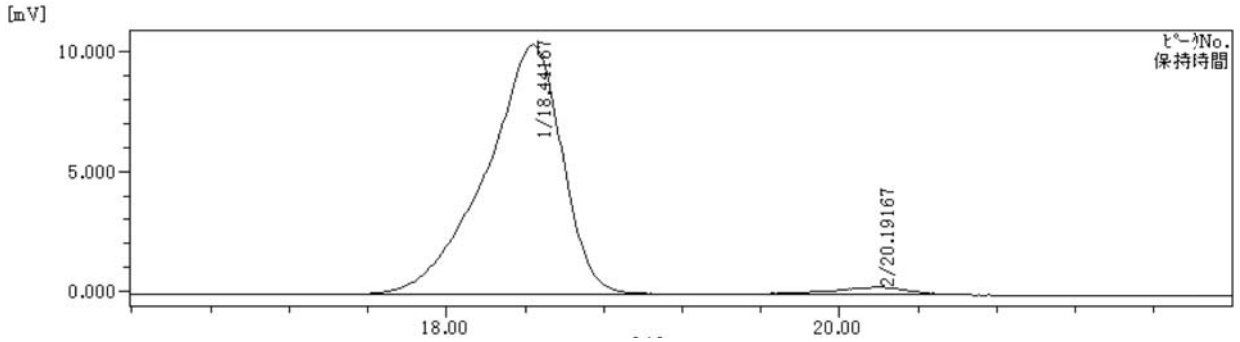


3cm



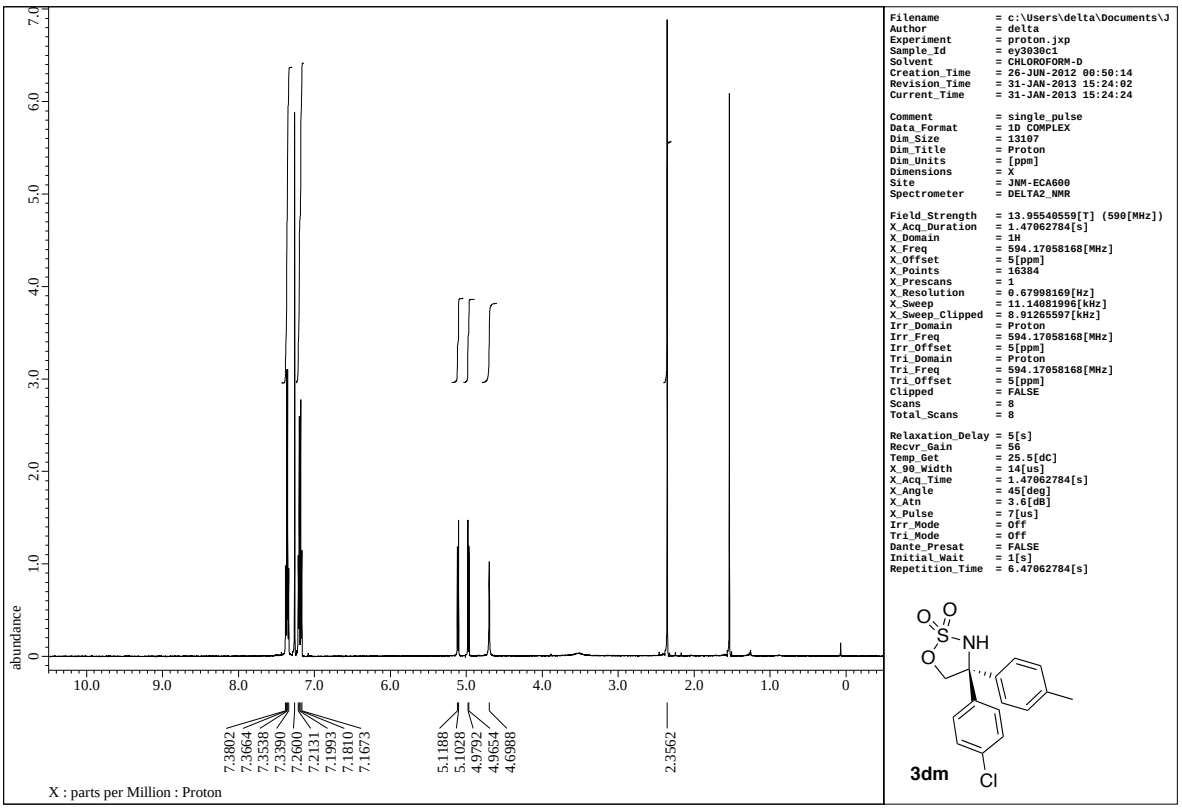
time (min)

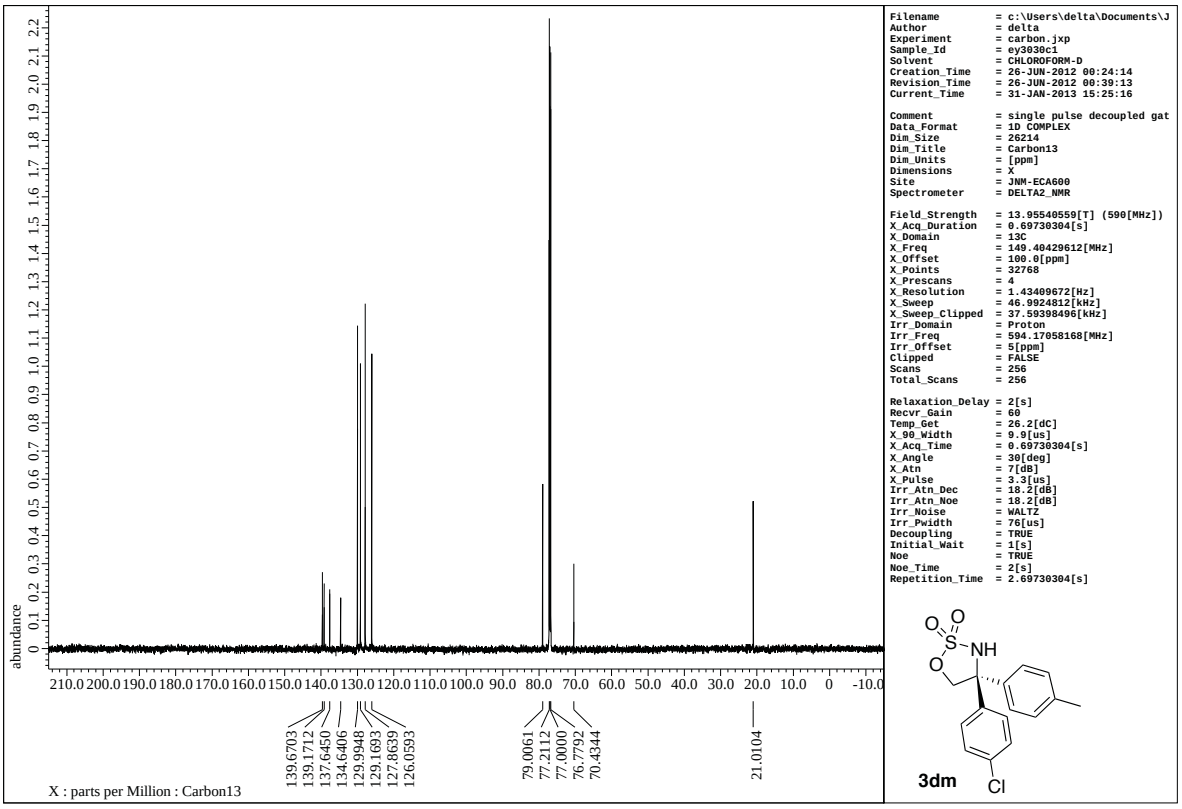
Pk#	Retention Time	Area	Area (%)
1	18.458	465.496	50.092
2	20.208	463.787	49.908

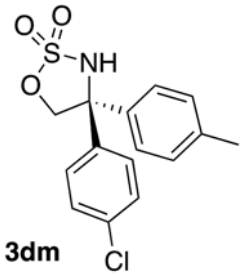


time (min)

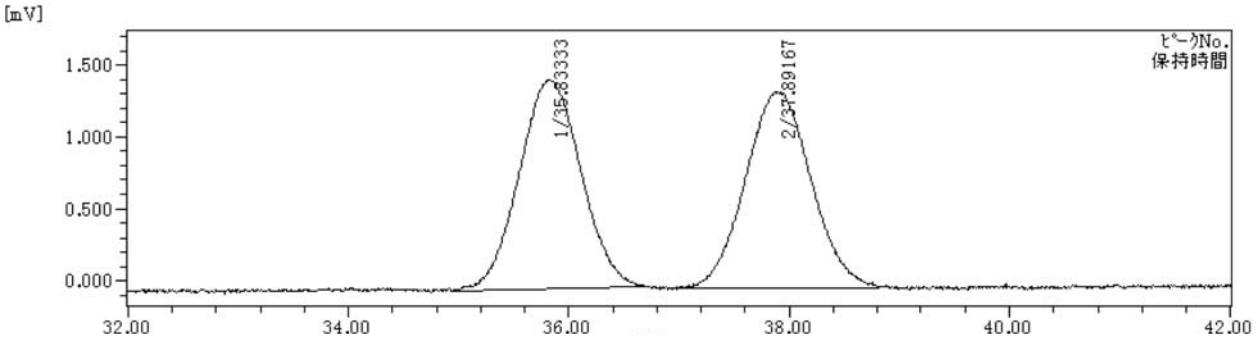
Pk#	Retention Time	Area	Area (%)
1	18.442	280.353	96.884
2	20.192	9.017	3.116


abundance
X : parts per Million : Proton
7.3802
7.3664
7.3538
7.3390
7.2600
7.2131
7.1993
7.1810
7.1673
5.1188
5.1028
4.9792
4.9654
4.6988
2.3562
Filename = c:\Users\delta\Documents\J
Author = delta
Experiment = proton.jxp
Sample_Id = ey3030c1
Solvent = CHLOROFORM-D
Creation_Time = 26-JUN-2012 00:50:14
Revision_Time = 31-JAN-2013 15:24:02
Current_Time = 31-JAN-2013 15:24:24
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 = 56
Temp_Get = 25.5[dC]
X_90_Width = 14[us]
X_Acq_Time = 1.47062784[s]
X_Angle = 45[deg]
X_Atn = 3.6[dB]
X_Pulse = 7[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
Initial_Wait = 1[s]
Repetition_Time = 6.47062784[s]
3dm


abundance
X : parts per Million : Carbon13
139.6703
139.1712
137.6450
134.6406
129.9948
129.1693
127.8639
126.0593
79.0061
77.2112
77.0000
76.7792
70.4344
21.0104
Filename = c:\Users\delta\Documents\J
Author = delta
Experiment = carbon.jxp
Sample_Id = ey3030c1
Solvent = CHLOROFORM-D
Creation_Time = 26-JUN-2012 00:24:14
Revision_Time = 26-JUN-2012 00:39:13
Current_Time = 31-JAN-2013 15:25:16
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 = FALSE
Scans = 256
Total_Scans = 256
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 26.2[dC]
X_90_Width = 9.9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 7[dB]
X_Pulse = 3.3[us]
Irr_Atn_Dec = 18.2[dB]
Irr_Atn_Noe = 18.2[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]
3dm

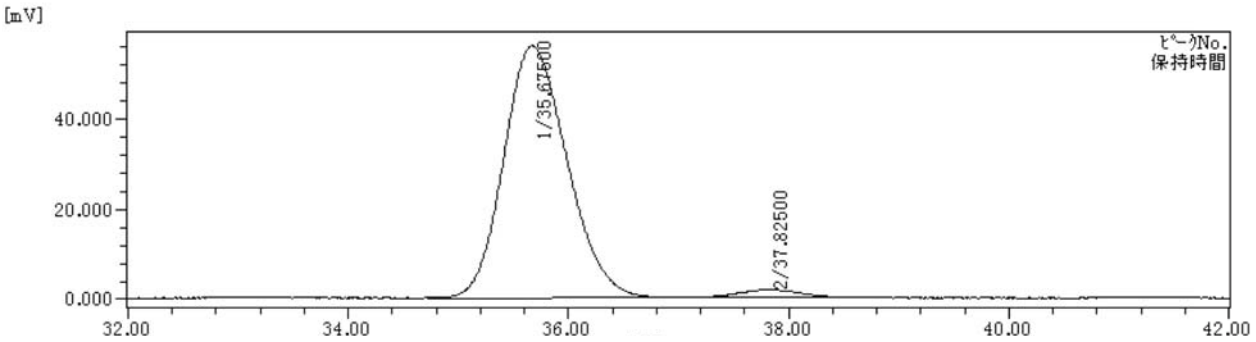


3dm



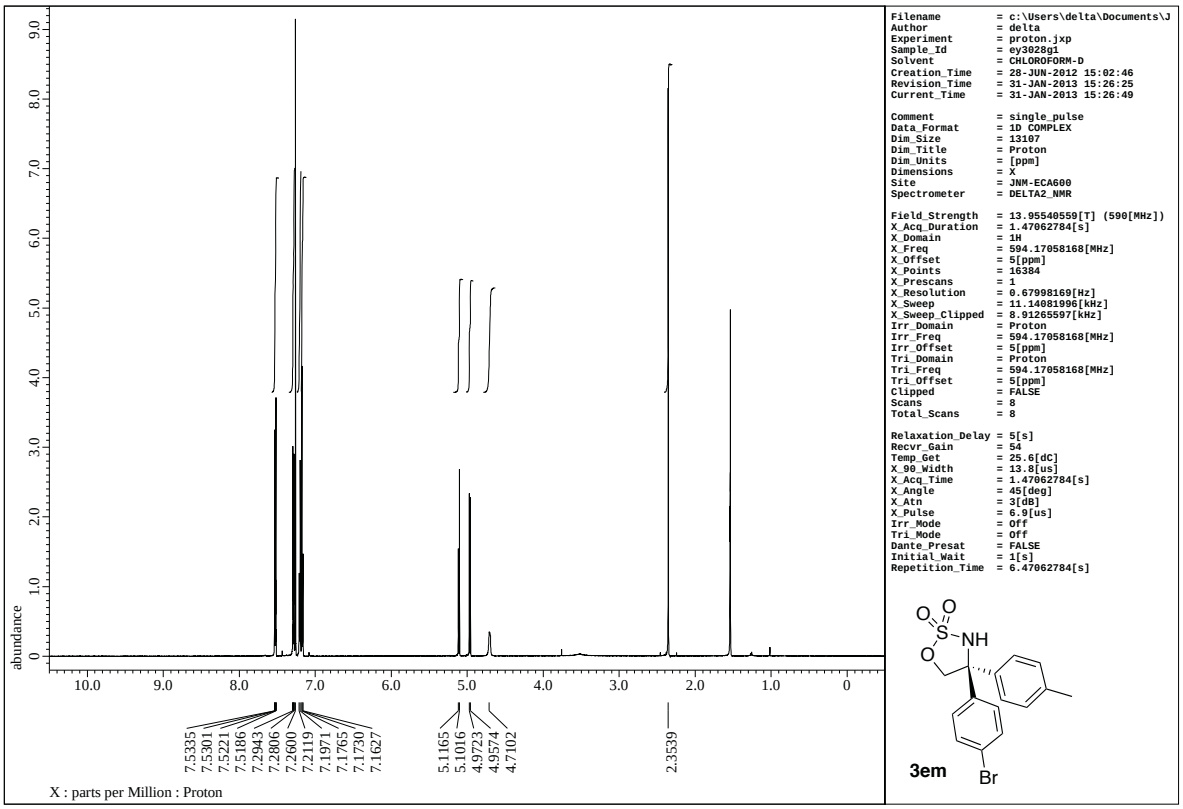
time (min)

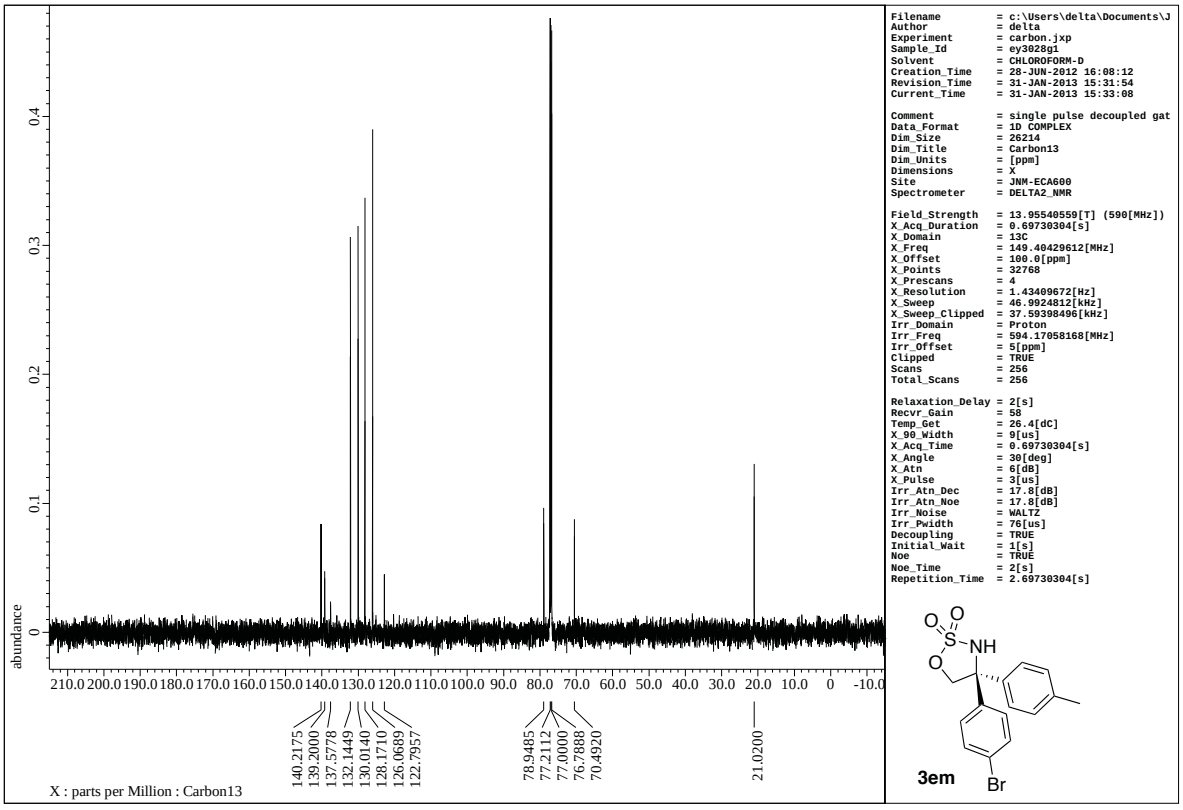
Pk#	Retention Time	Area	Area (%)
1	35.833	56.674	49.919
2	37.892	56.857	50.081

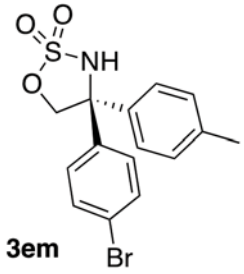


time (min)

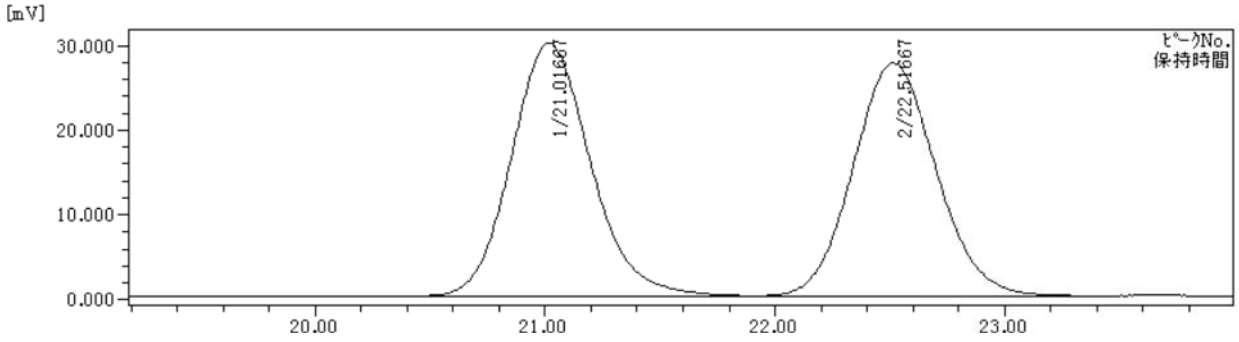
Pk#	Retention Time	Area	Area (%)
1	35.675	2230.541	96.936
2	37.825	70.494	3.064


abundance
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
7.5335 7.5301 7.5221 7.5186 7.2943 7.2806 7.2600 7.2119 7.1971 7.1765 7.1730 7.1627
5.1165 5.1016 4.9723 4.9574 4.7102
2.3539
X : parts per Million : Proton
Filename = c:\Users\delta\Documents\J
Author = delta
Experiment = proton.jxp
Sample_Id = ey3028g1
Solvent = CHLOROFORM-D
Creation_Time = 28-JUN-2012 15:02:46
Revision_Time = 31-JAN-2013 15:26:25
Current_Time = 31-JAN-2013 15:26:49
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 = 25.6[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]
O O S NH O
3em
Br


abundance
0.4 0.3 0.2 0.1 0
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
140.2175 139.2000 137.5778 132.1449 130.0140 128.1710 126.0689 122.7957
78.9485 77.2112 77.0000 76.7888 70.4920
21.0200
X : parts per Million : Carbon13
Filename = c:\Users\delta\Documents\J
Author = delta
Experiment = carbon.jxp
Sample_Id = ey3028g1
Solvent = CHLOROFORM-D
Creation_Time = 28-JUN-2012 16:08:12
Revision_Time = 31-JAN-2013 15:31:54
Current_Time = 31-JAN-2013 15:33:08
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 = 26.4[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]
O O S NH O
3em
Br

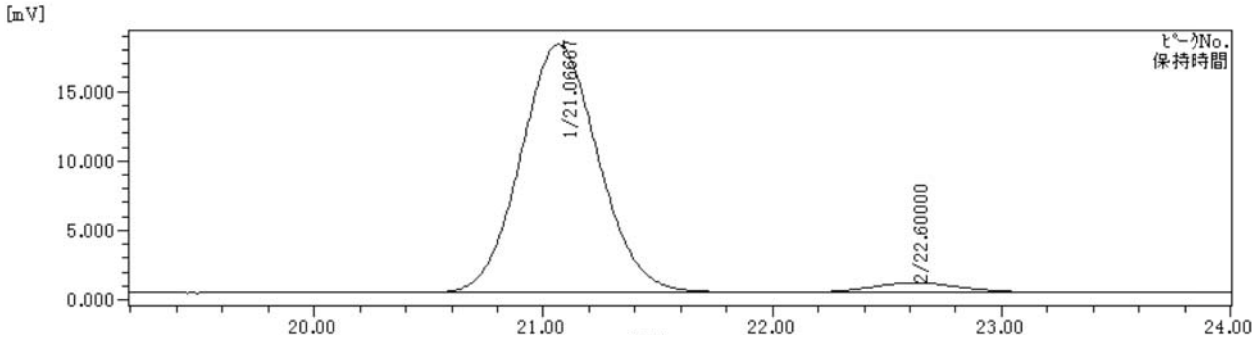


3em



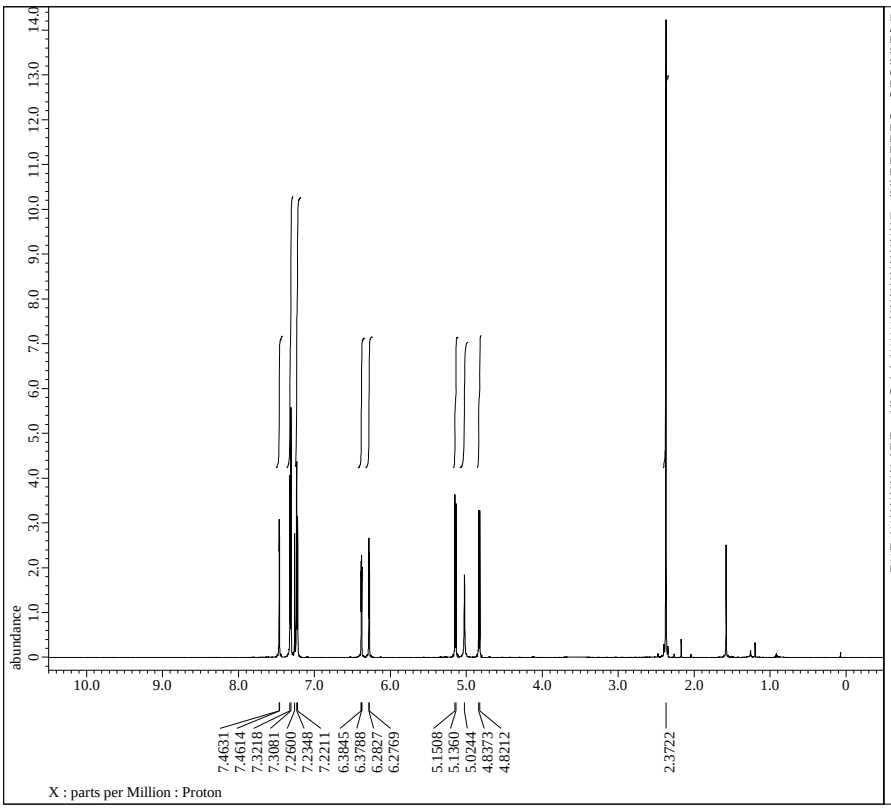
time (min)

Pk#	Retention Time	Area	Area (%)
1	21.017	715.388	50.858
2	22.517	691.238	49.142



time (min)

Pk#	Retention Time	Area	Area (%)
1	21.067	419.711	95.784
2	22.600	18.474	4.216

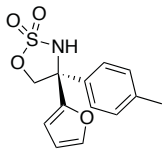



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = 16tn28
Solvent           = CHLOROFORM-D
Creation_Time     = 11-APR-2012 09:32:54
Revision_Time     = 11-APR-2012 09:38:30
Current_Time      = 31-JAN-2013 15:34:25

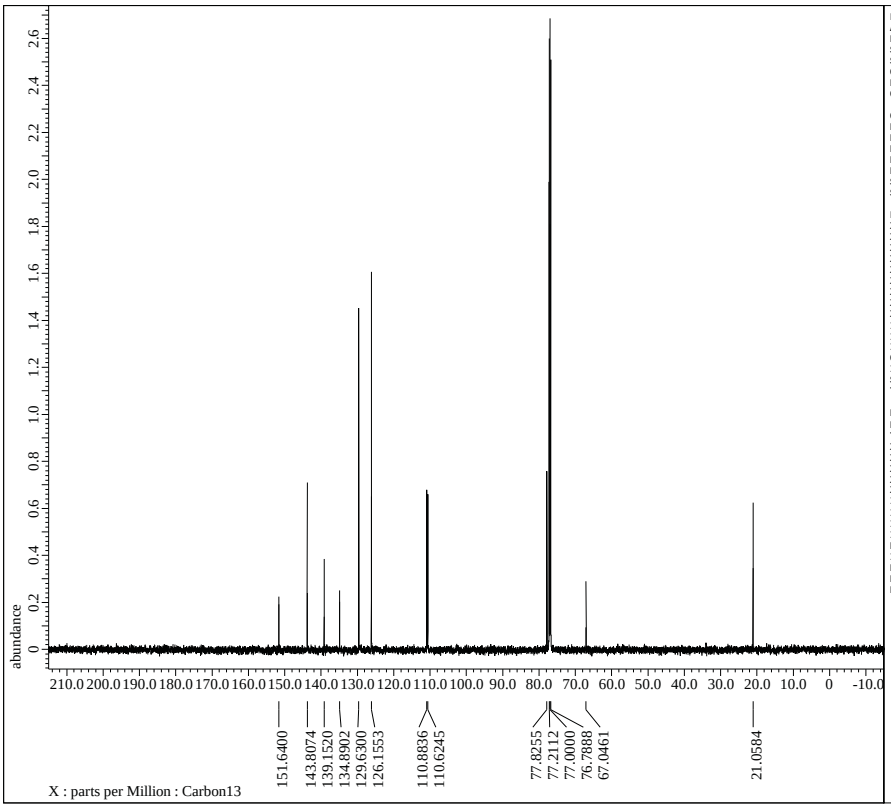
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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          = 21.8[dC]
X_90_Width        = 14[us]
X_Acq_Time        = 1.47062784[s]
X_Angle           = 45[deg]
X_Atn             = 3.6[dB]
X_Pulse           = 7[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.47062784[s]
```



3fm

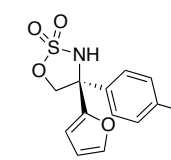



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 16tn28
Solvent           = CHLOROFORM-D
Creation_Time     = 10-APR-2012 14:54:13
Revision_Time     = 10-APR-2012 15:03:30
Current_Time      = 31-JAN-2013 15:35:06

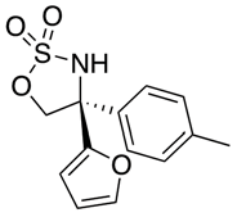
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 0[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           = FALSE
Incomplete_Copy   = TRUE
Scans             = 160
Total_Scans       = 160

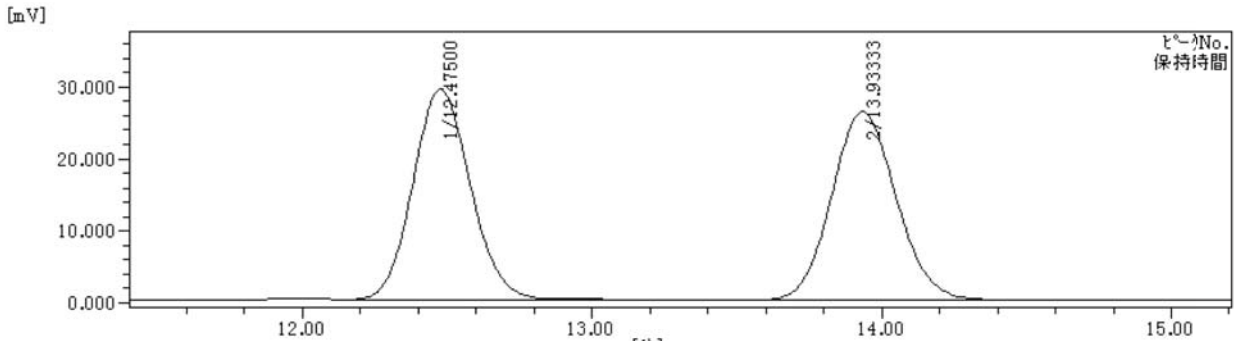
Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22.8[dC]
X_90_Width        = 9.9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 7[dB]
X_Pulse           = 3.3[us]
Irr_Atn_Dec       = 18.2[dB]
Irr_Atn_Noe       = 18.2[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



3fm

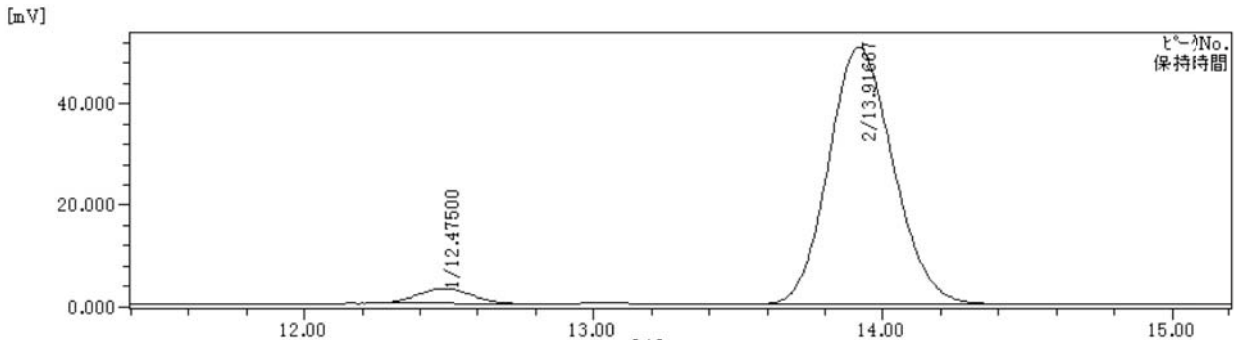


3fm



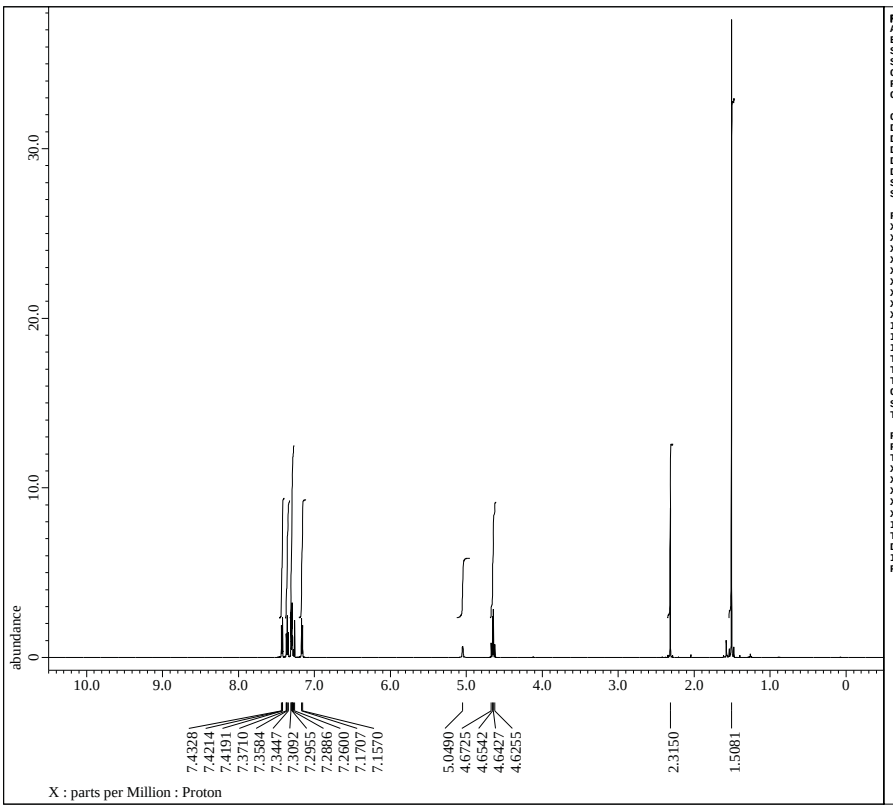
time (min)

Pk#	Retention Time	Area	Area (%)
1	12.475	407.077	50.002
2	13.933	407.046	49.998



time (min)

Pk#	Retention Time	Area	Area (%)
1	12.475	38.925	4.684
2	13.917	792.122	95.316

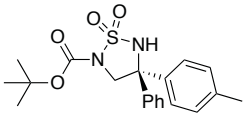



```
Filename        = c:¥Users¥delta¥Documents¥IB
Author          = delta
Experiment      = proton.jxp
Sample_Id       = 16tn17
Solvent         = CHLOROFORM-D
Creation_Time   =  5-APR-2012 13:07:07
Revision_Time   =  5-APR-2012 13:20:47
Current_Time    = 31-JAN-2013 15:39:02

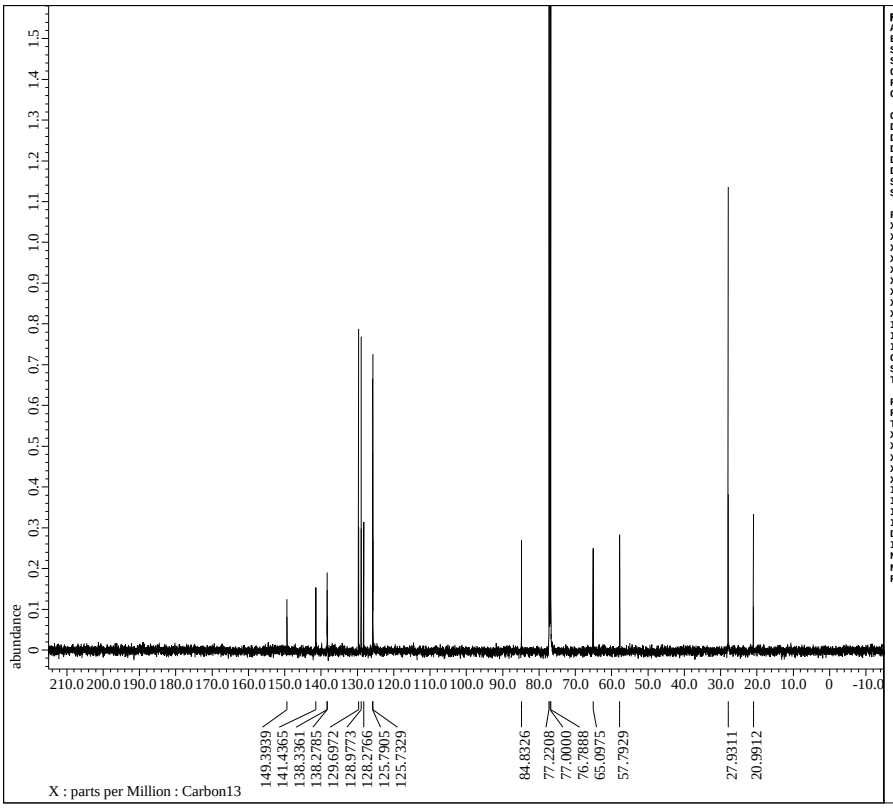
Comment         = single_pulse
Data_Format     = 1D COMPLEX
Dim_Size        = 13107
Dim_Title       = Proton
Dim_Units       = [ppm]
Dimensions      = X
Site            = JNM-ECA600
Spectrometer    = DELTA2_NMR

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        = 21.3[dC]
X_90_Width      = 14[us]
X_Acq_Time      = 1.47062784[s]
X_Angle         = 45[deg]
X_Atn           = 3.6[dB]
X_Pulse         = 7[us]
Irr_Mode        = Off
Tri_Mode        = Off
Dante_Presat    = FALSE
Initial_Wait    = 1[s]
Repetition_Time = 6.47062784[s]
```



3gm

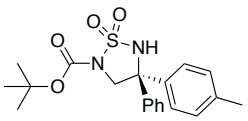



```
Filename        = c:¥Users¥delta¥Documents¥IB
Author          = delta
Experiment      = carbon.jxp
Sample_Id       = 16tn17
Solvent         = CHLOROFORM-D
Creation_Time   =  5-APR-2012 13:10:33
Revision_Time   =  5-APR-2012 13:26:21
Current_Time    = 31-JAN-2013 15:39:42

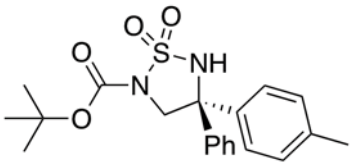
Comment         = single pulse decoupled gat
Data_Format     = 1D COMPLEX
Dim_Size        = 26214
Dim_Title       = Carbon13
Dim_Units       = [ppm]
Dimensions      = X
Site            = JNM-ECA600
Spectrometer    = DELTA2_NMR

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

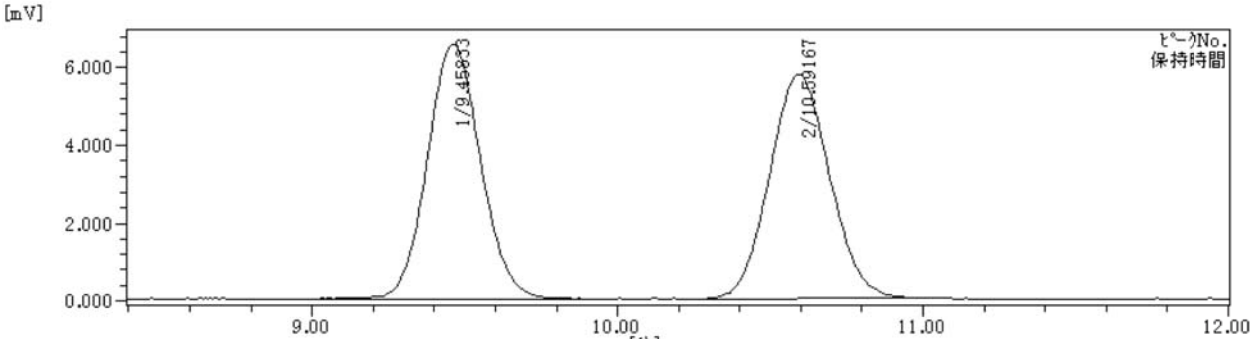
Relaxation_Delay = 2[s]
Recvr_Gain      = 60
Temp_Get        = 22[dC]
X_90_Width      = 9.9[us]
X_Acq_Time      = 0.69730304[s]
X_Angle         = 30[deg]
X_Atn           = 7[dB]
X_Pulse         = 3.3[us]
Irr_Atn_Dec     = 18.2[dB]
Irr_Atn_Noe     = 18.2[dB]
Irr_Noise       = WALTZ
Irr_Pwidth      = 76[us]
Decoupling      = TRUE
Initial_Wait    = 1[s]
Noe             = TRUE
Noe_Time        = 2[s]
Repetition_Time = 2.69730304[s]
```



3gm

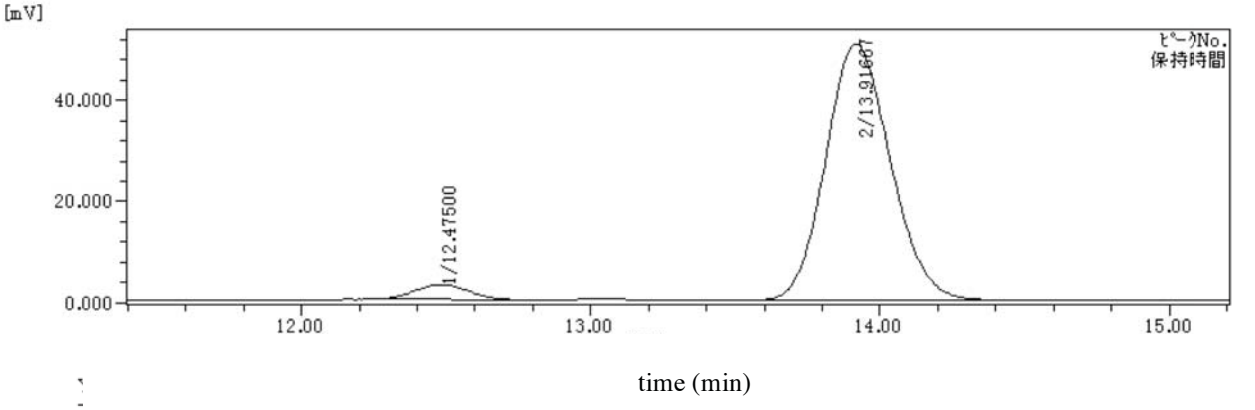


3gm



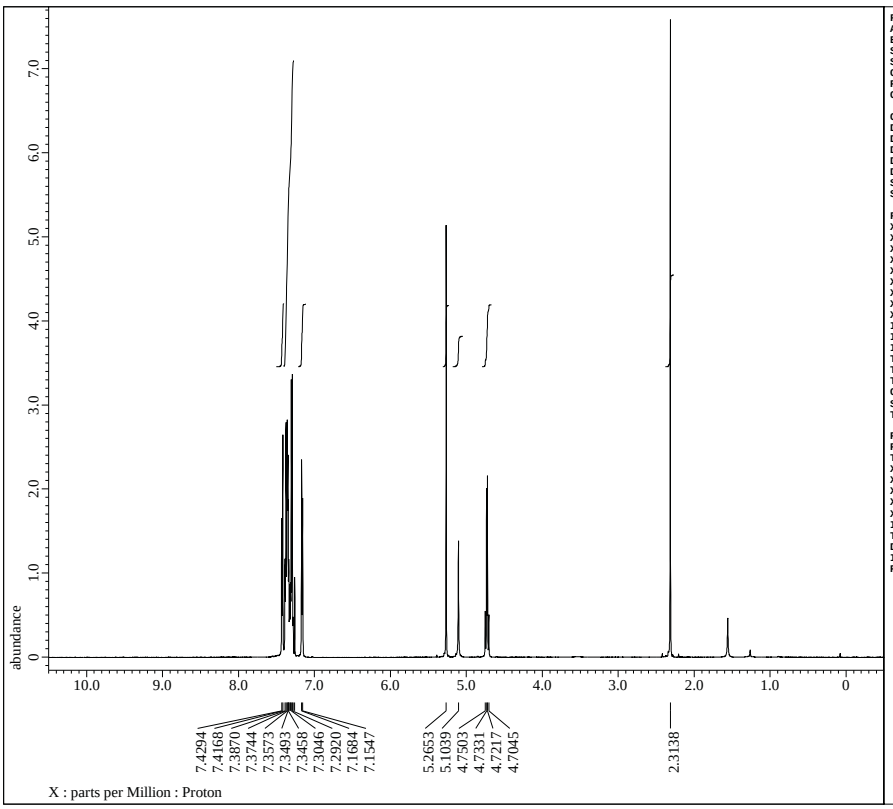
time (min)

Pk#	Retention Time	Area	Area (%)
1	9.458	80.026	50.320
2	10.592	79.007	49.680



time (min)

Pk#	Retention Time	Area	Area (%)
1	9.617	3.275	2.566
2	10.675	124.329	97.434

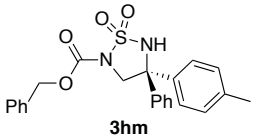



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = data z
Solvent           = CHLOROFORM-D
Creation_Time     = 19-DEC-2012 14:07:51
Revision_Time     = 31-JAN-2013 15:41:23
Current_Time      = 31-JAN-2013 15:41:50

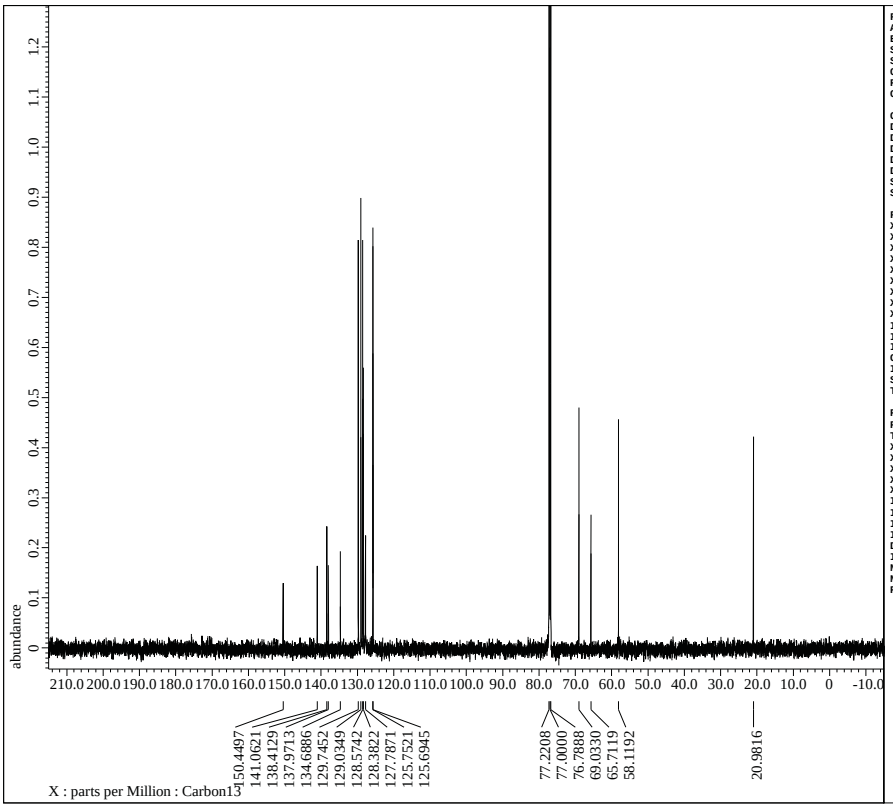
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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          = 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]
```



3hm

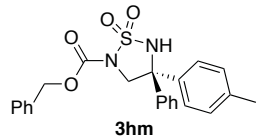



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = 16tn37
Solvent           = CHLOROFORM-D
Creation_Time     = 16-APR-2012 13:38:31
Revision_Time     = 16-APR-2012 13:47:49
Current_Time      = 31-JAN-2013 15:42:38

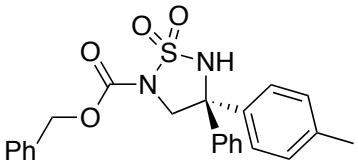
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 0[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           = FALSE
Incomplete_Copy   = TRUE
Scans             = 160
Total_Scans       = 160

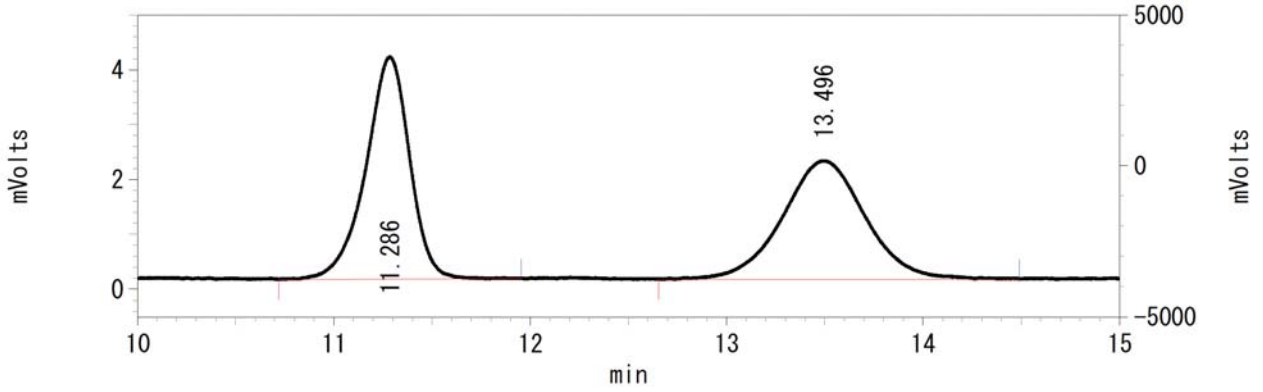
Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 23.1[dC]
X_90_Width        = 9.0[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 7[dB]
X_Pulse           = 3.3[us]
Irr_Atn_Dec       = 18.2[dB]
Irr_Atn_Noe       = 18.2[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



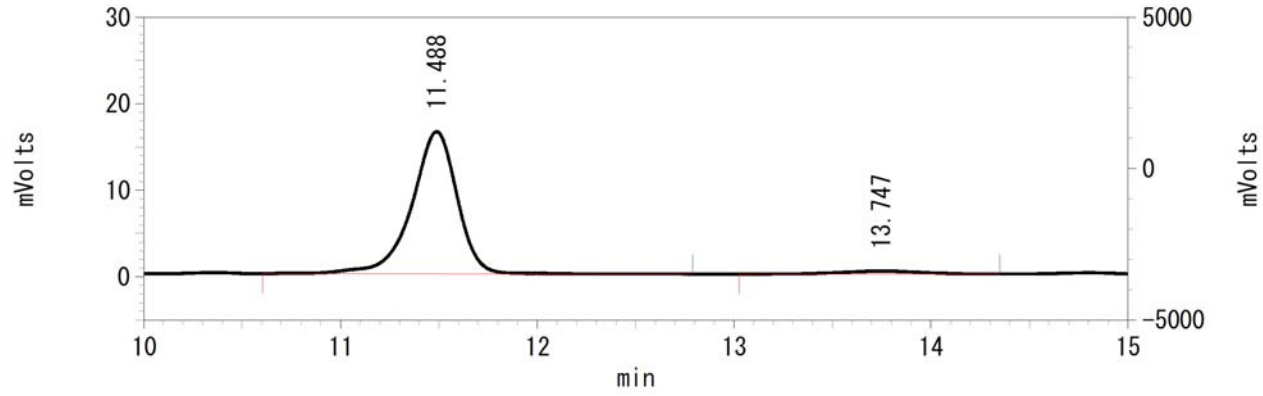
3hm



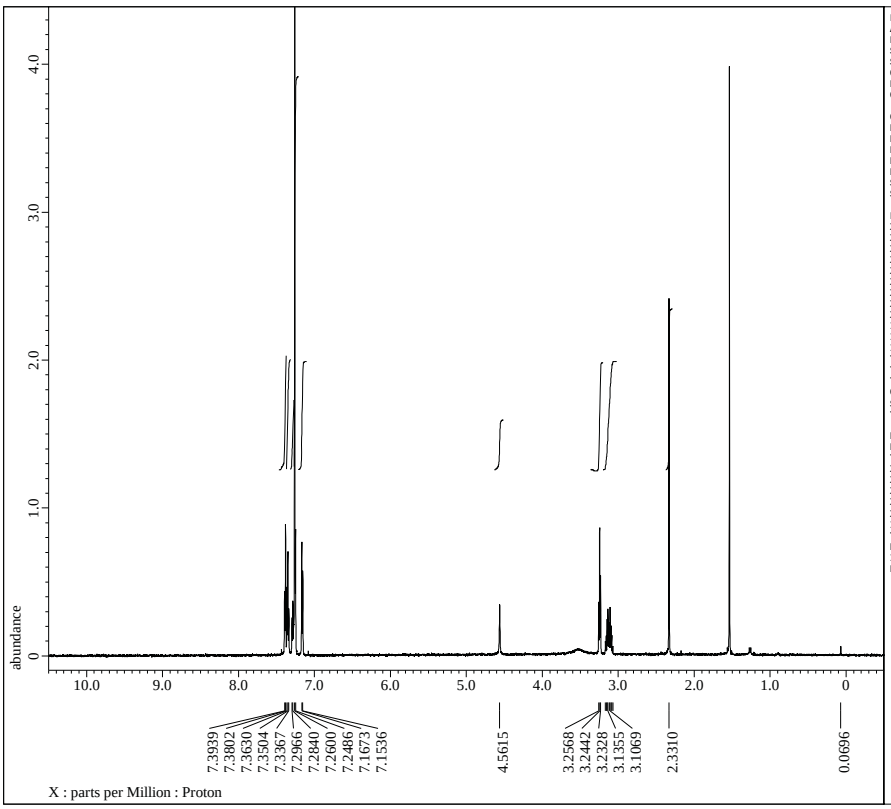
3hm

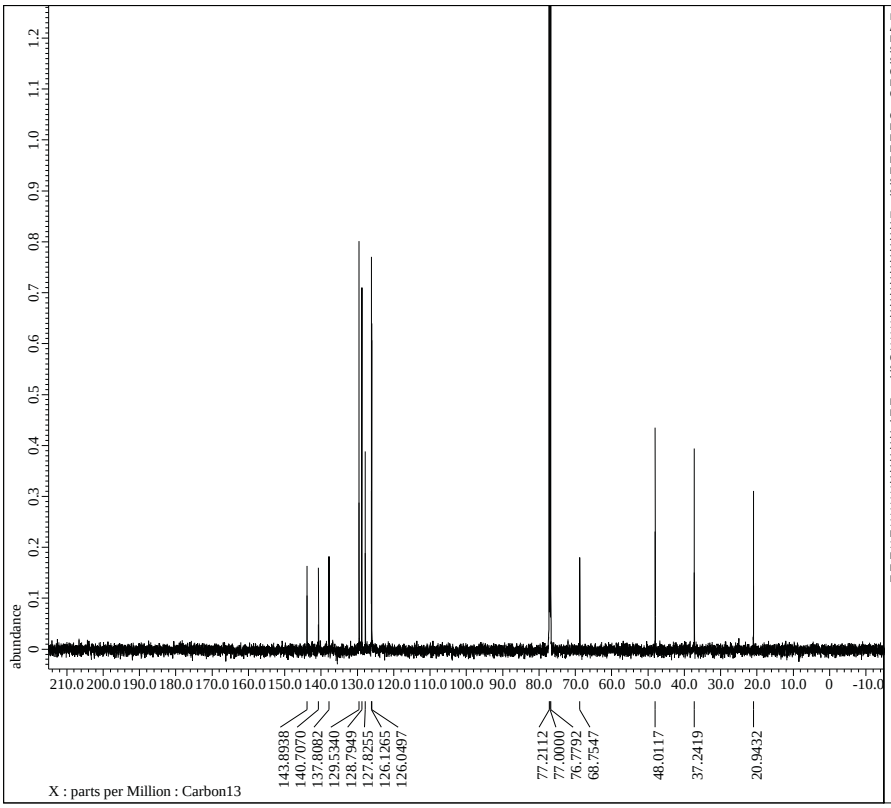


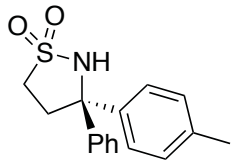
Pk #	Retention Time	Area	Area Percent
1	11.286	63164	49.940
2	13.496	63316	50.060



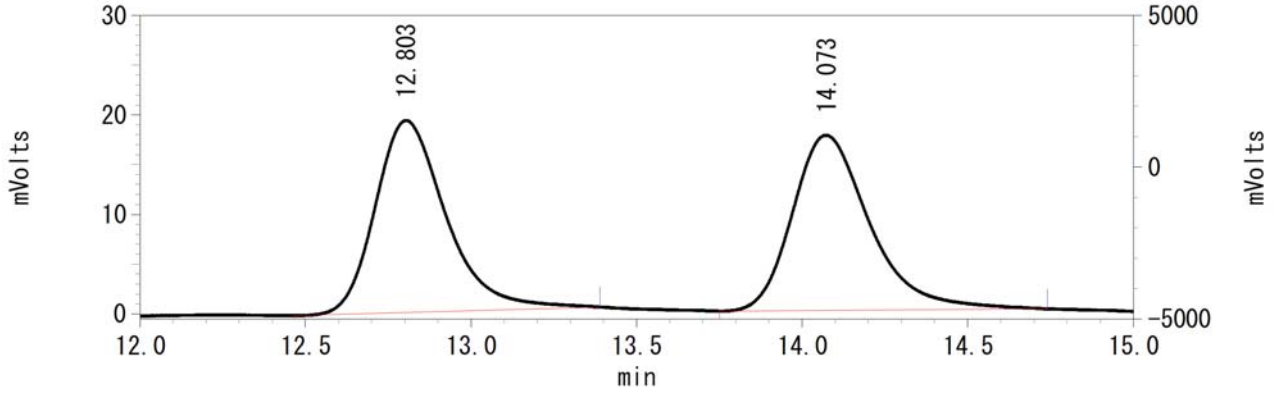
Pk #	Retention Time	Area	Area Percent
1	11.488	265144	96.315
2	13.747	10144	3.685


abundance
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
7.3939 7.3802 7.3630 7.3504 7.3367 7.2966 7.2840 7.2600 7.2486 7.1673 7.1536
4.5615
3.2568 3.2442 3.2328 3.1355 3.1069
2.3310
0.0696
X : parts per Million : Proton
Filename = c:\Users\delta\Documents\J
Author = delta
Experiment = proton.jxp
Sample_Id = data hf1153
Solvent = CHLOROFORM-D
Creation_Time = 19-DEC-2012 14:55:46
Revision_Time = 31-JAN-2013 15:45:03
Current_Time = 31-JAN-2013 15:45:39
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 = 60
Temp_Get = 23.5[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]
O O S NH Ph
3im

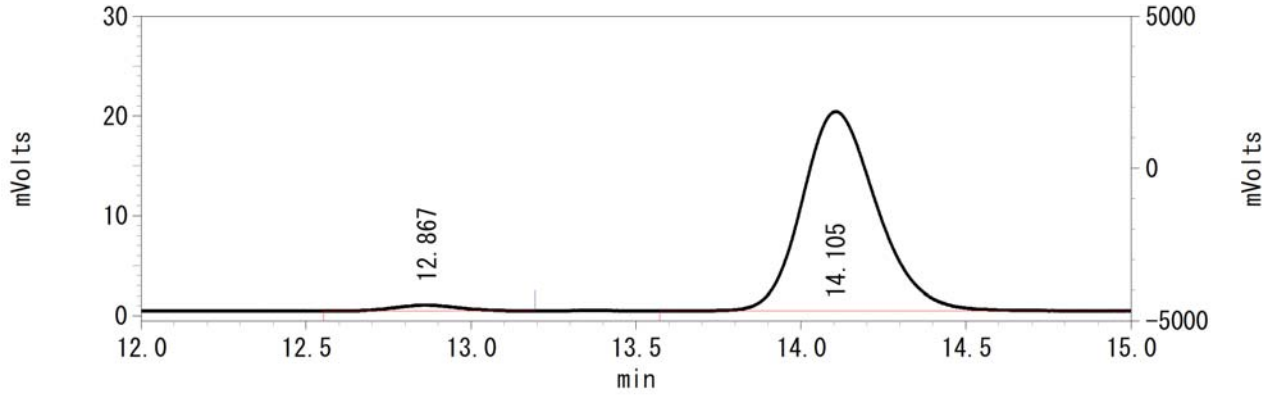

abundance
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
143.8938 140.7070 137.8082 129.5340 128.7949 127.8255 126.1265 126.0497
77.2112 77.0000 76.7792 68.7547
48.0117
37.2419
20.9432
X : parts per Million : Carbon13
Filename = c:\Users\delta\Documents\J
Author = delta
Experiment = carbon.jxp
Sample_Id = data hf1153
Solvent = CHLOROFORM-D
Creation_Time = 19-DEC-2012 15:52:11
Revision_Time = 19-DEC-2012 16:06:03
Current_Time = 31-JAN-2013 15:46:27
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 = 24.3[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]
O O S NH Ph
3im



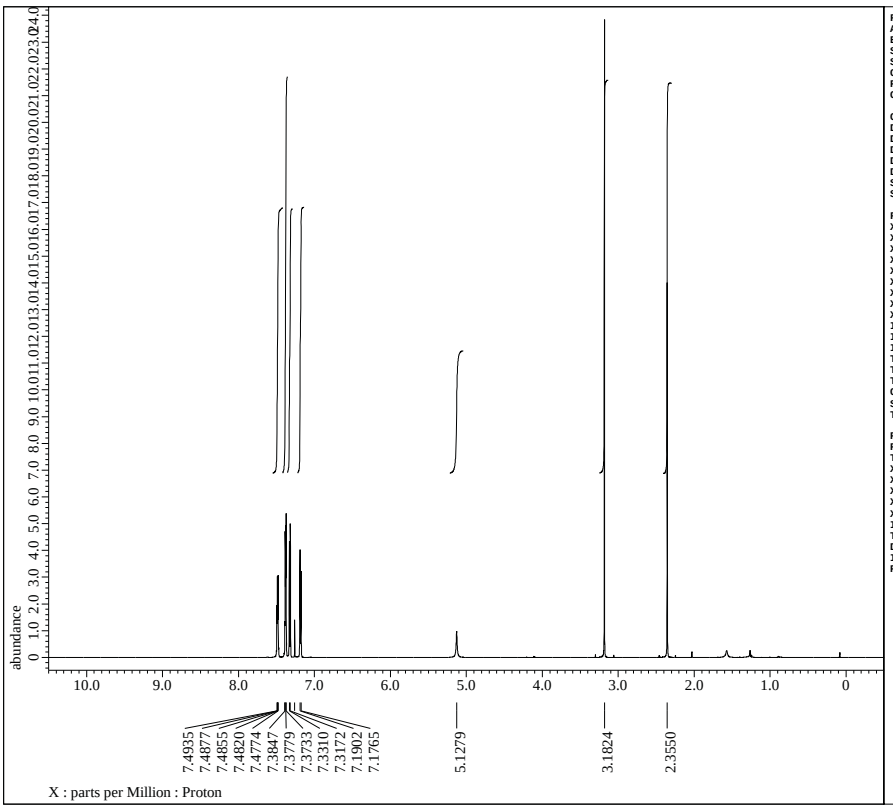
3im

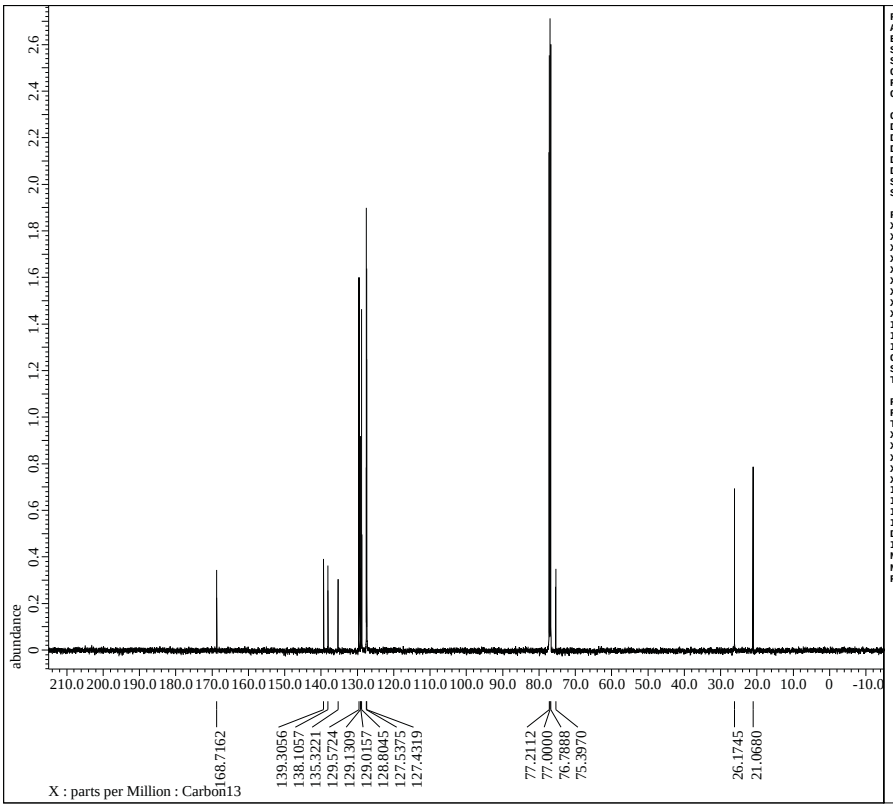


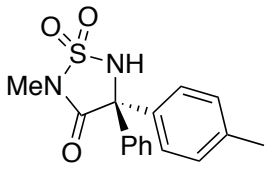
Pk #	Retention Time	Area	Area Percent
1	12.803	286322	50.094
2	14.073	285243	49.906



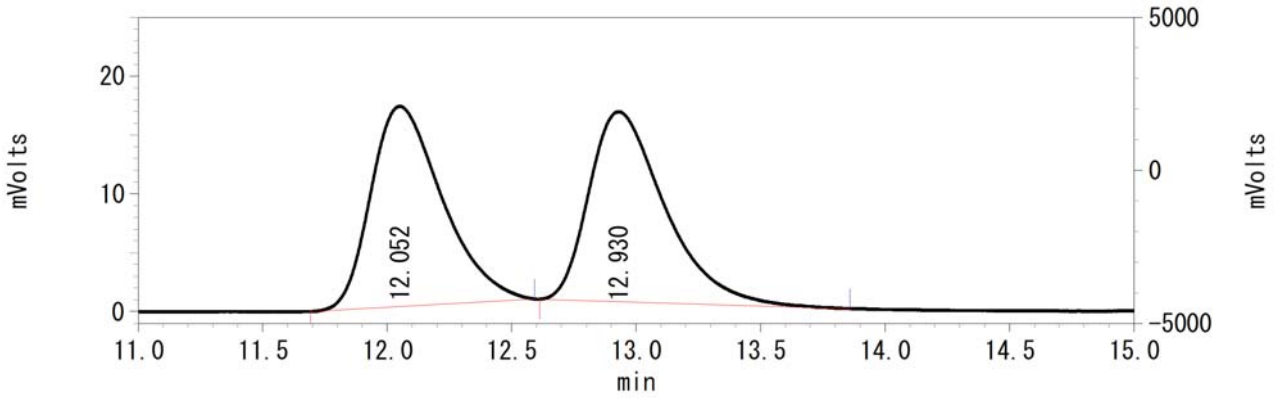
Pk #	Retention Time	Area	Area Percent
1	12.867	8113	2.525
2	14.105	313224	97.475


abundance
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
7.4935
7.4877
7.4855
7.4820
7.4774
7.3847
7.3779
7.3733
7.3310
7.3172
7.1902
7.1765
5.1279
3.1824
2.3550
X : parts in Million : Proton
Filename = c:\Users\delta\Documents\J
Author = delta
Experiment = proton.jxp
Sample_Id = data hf1097
Solvent = CHLOROFORM-D
Creation_Time = 15-OCT-2012 21:36:38
Revision_Time = 31-JAN-2013 15:47:32
Current_Time = 31-JAN-2013 15:47:51
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 = 24.3[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]
MeN
NH
Ph
3jm

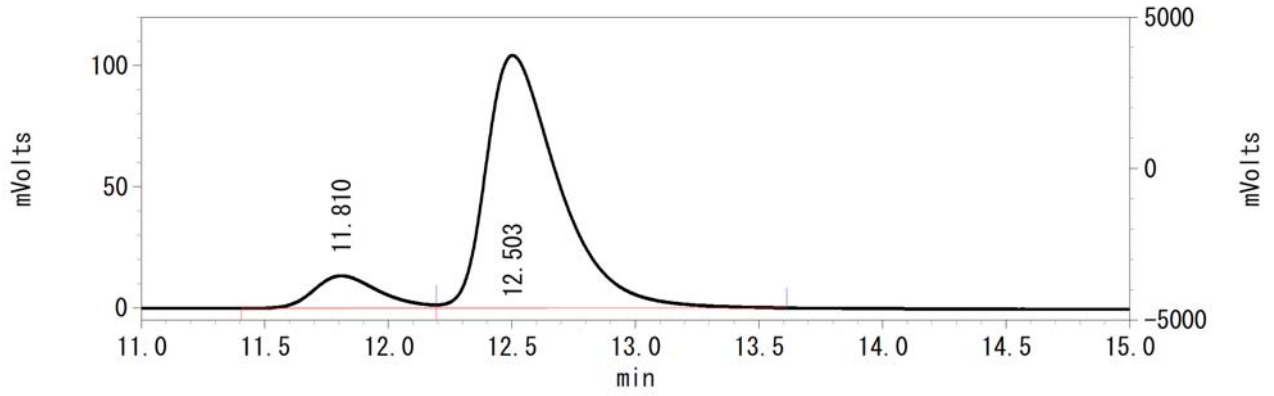

abundance
210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0
168.7162
139.3056
138.1057
135.3221
129.5724
129.1309
129.0157
128.8045
127.5375
127.4319
77.2112
77.0000
76.7888
75.3970
26.1745
21.0680
X : parts in Million : Carbon13
Filename = c:\Users\delta\Documents\J
Author = delta
Experiment = carbon.jxp
Sample_Id = data hf1097
Solvent = CHLOROFORM-D
Creation_Time = 15-OCT-2012 21:39:25
Revision_Time = 15-OCT-2012 21:53:31
Current_Time = 31-JAN-2013 15:48:25
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 = 25.1[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]
MeN
NH
Ph
3jm



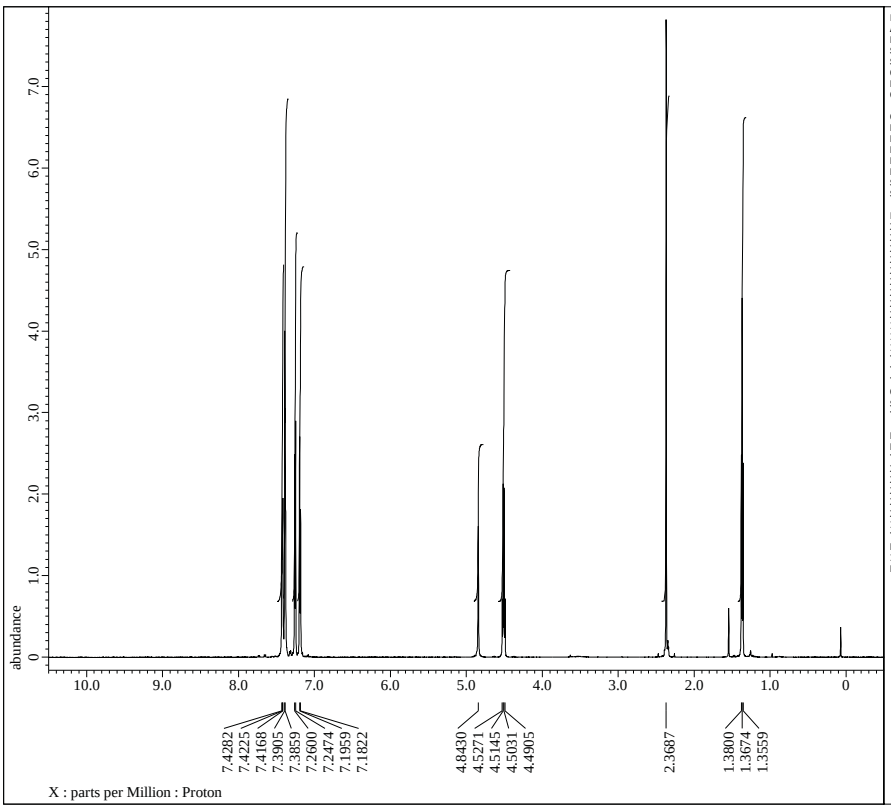
3jm



Pk #	Retention Time	Area	Area Percent
1	12.052	339634	49.800
2	12.930	342357	50.200



Pk #	Retention Time	Area	Area Percent
1	11.810	247019	10.399
2	12.503	2128499	89.601

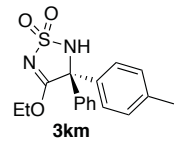



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = data hf1151
Solvent           = CHLOROFORM-D
Creation_Time     = 19-DEC-2012 15:30:25
Revision_Time     = 31-JAN-2013 15:49:06
Current_Time      = 31-JAN-2013 15:49:32

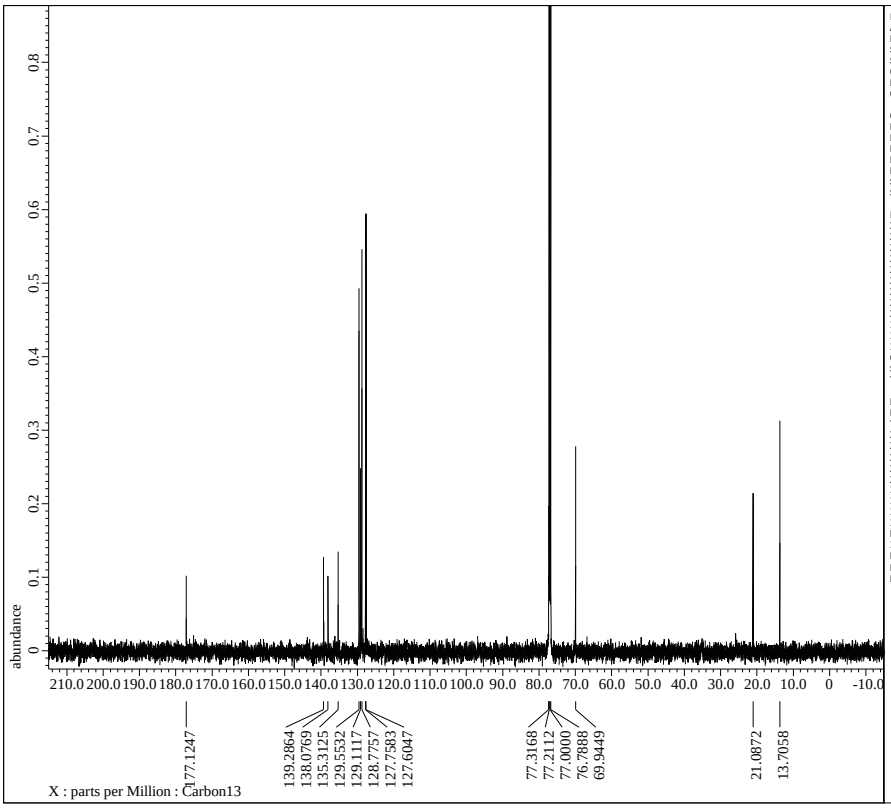
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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          = 23.5[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]
```



3km

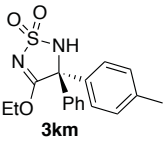



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = data hf1151
Solvent           = CHLOROFORM-D
Creation_Time     = 19-DEC-2012 15:33:14
Revision_Time     = 19-DEC-2012 15:47:59
Current_Time      = 31-JAN-2013 15:50:17

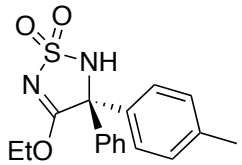
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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

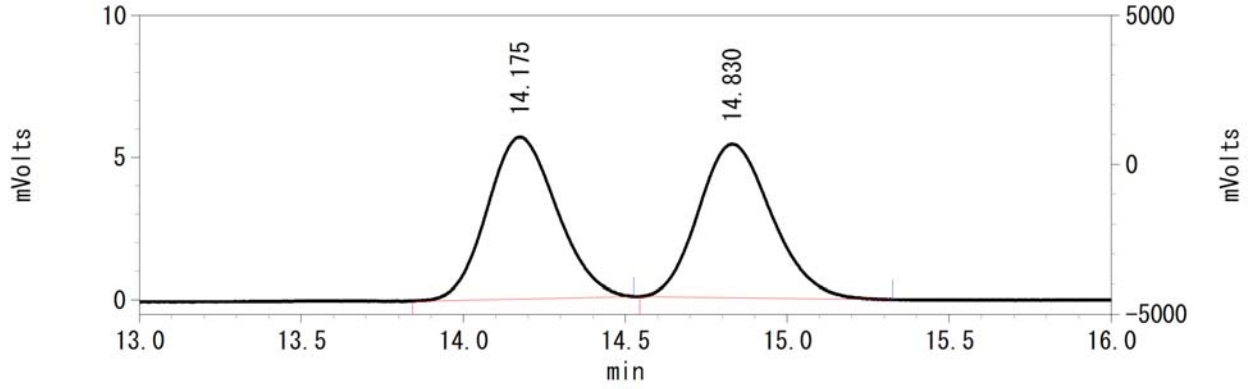
Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.2[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



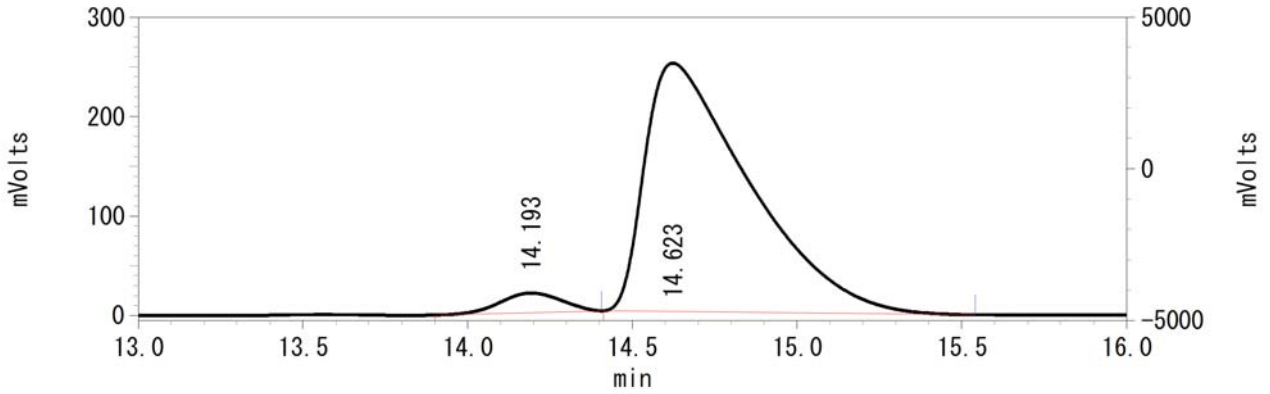
3km



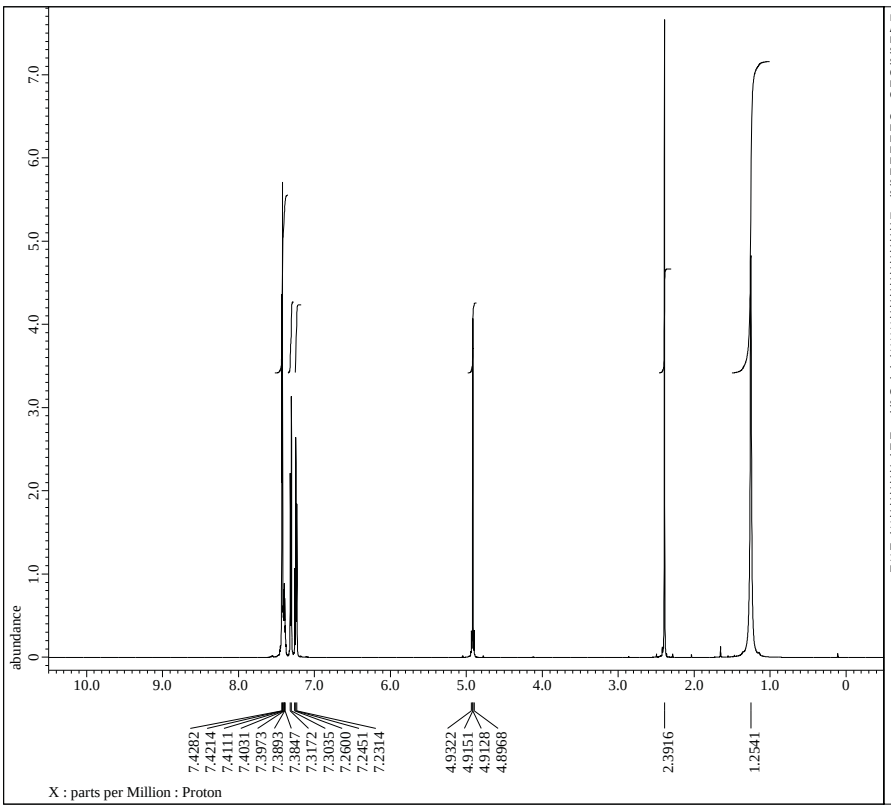
3km



Pk #	Retention Time	Area	Area Percent
1	14.175	84809	50.043
2	14.830	84662	49.957



Pk #	Retention Time	Area	Area Percent
1	14.193	254290	4.411
2	14.623	5510881	95.589

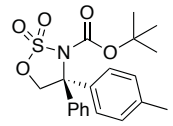



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = data hf1117
Solvent           = CHLOROFORM-D
Creation_Time     = 31-OCT-2012 23:15:29
Revision_Time     = 31-JAN-2013 15:51:56
Current_Time      = 31-JAN-2013 15:52:09

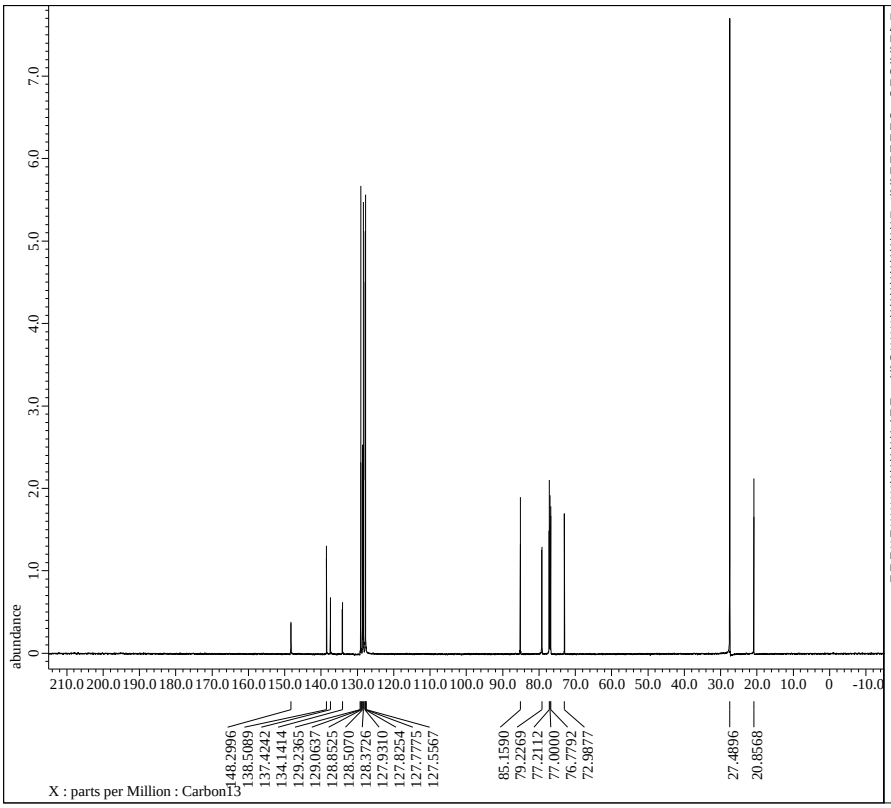
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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        = 22
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]
```



4

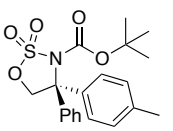



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = data hf1117
Solvent           = CHLOROFORM-D
Creation_Time     = 31-OCT-2012 23:18:18
Revision_Time     = 31-OCT-2012 23:31:54
Current_Time      = 31-JAN-2013 15:52:46

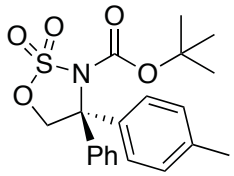
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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

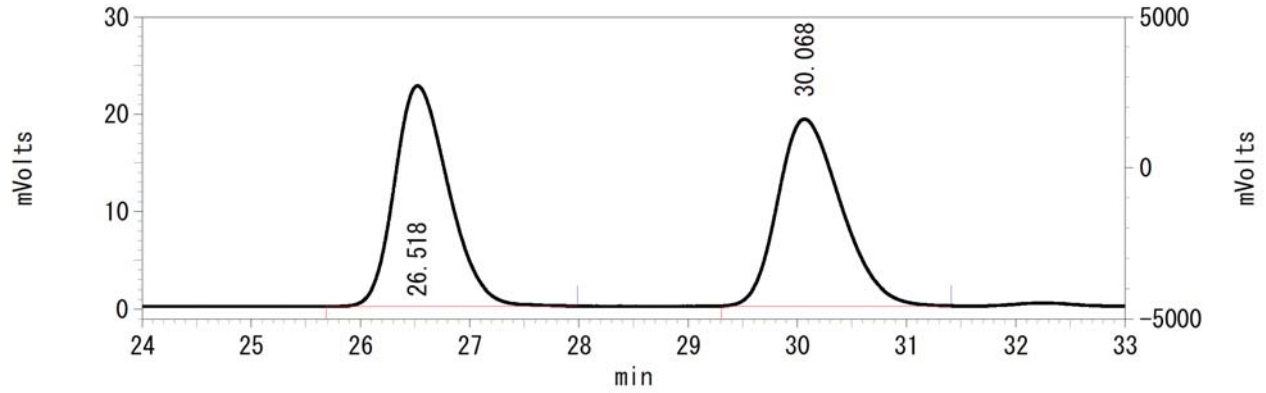
Relaxation_Delay  = 2[s]
Recvr_Gain        = 58
Temp_Get          = 24.5[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



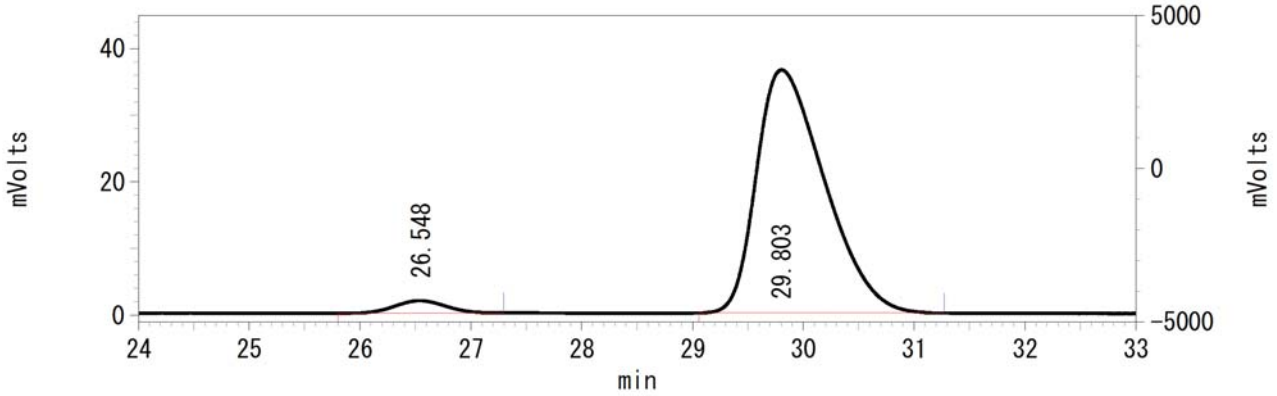
4



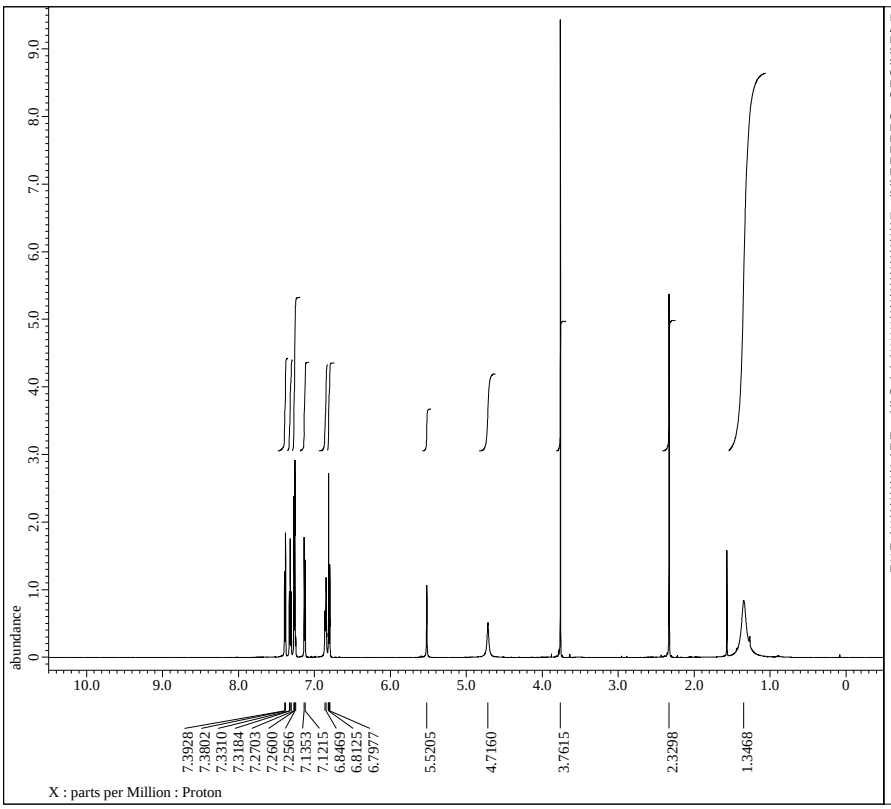
4



Pk #	Retention Time	Area	Area Percent
1	26.518	773551	50.144
2	30.068	769095	49.856



Pk #	Retention Time	Area	Area Percent
1	26.548	59116	3.651
2	29.803	1560182	96.349

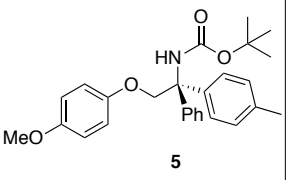



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = data hf1112
Solvent           = CHLOROFORM-D
Creation_Time     = 27-OCT-2012 11:46:06
Revision_Time     = 31-JAN-2013 19:13:28
Current_Time      = 31-JAN-2013 19:13:51

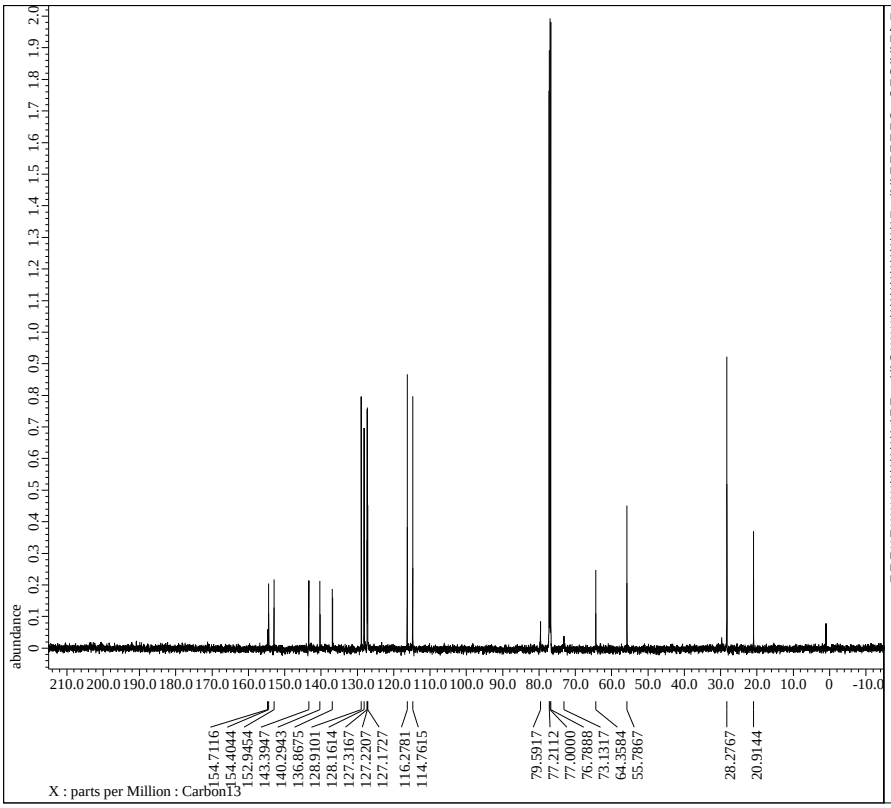
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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          = 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

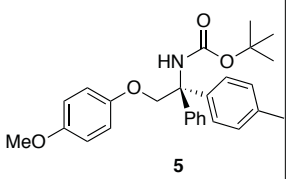



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = data hf1112re
Solvent           = CHLOROFORM-D
Creation_Time     = 21-JAN-2013 22:34:20
Revision_Time     = 31-JAN-2013 19:14:45
Current_Time      = 31-JAN-2013 19:15:11

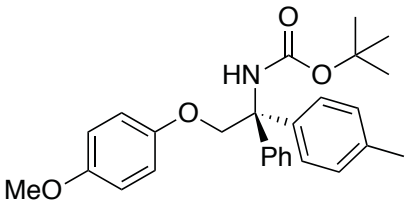
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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

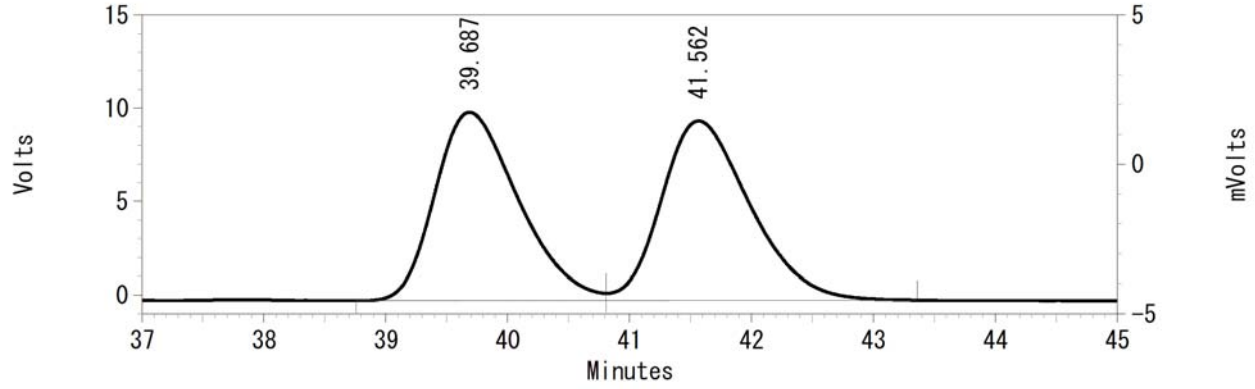
Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 50[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



5

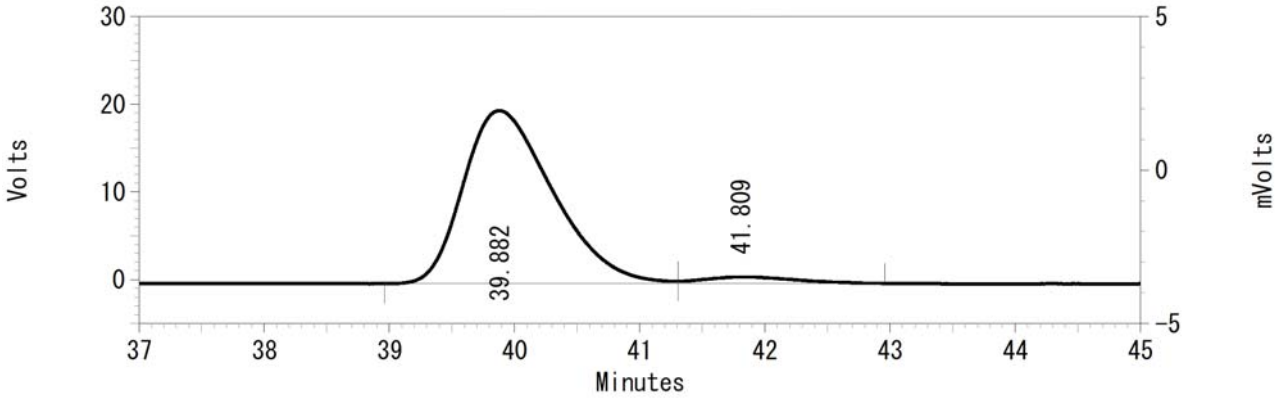


5



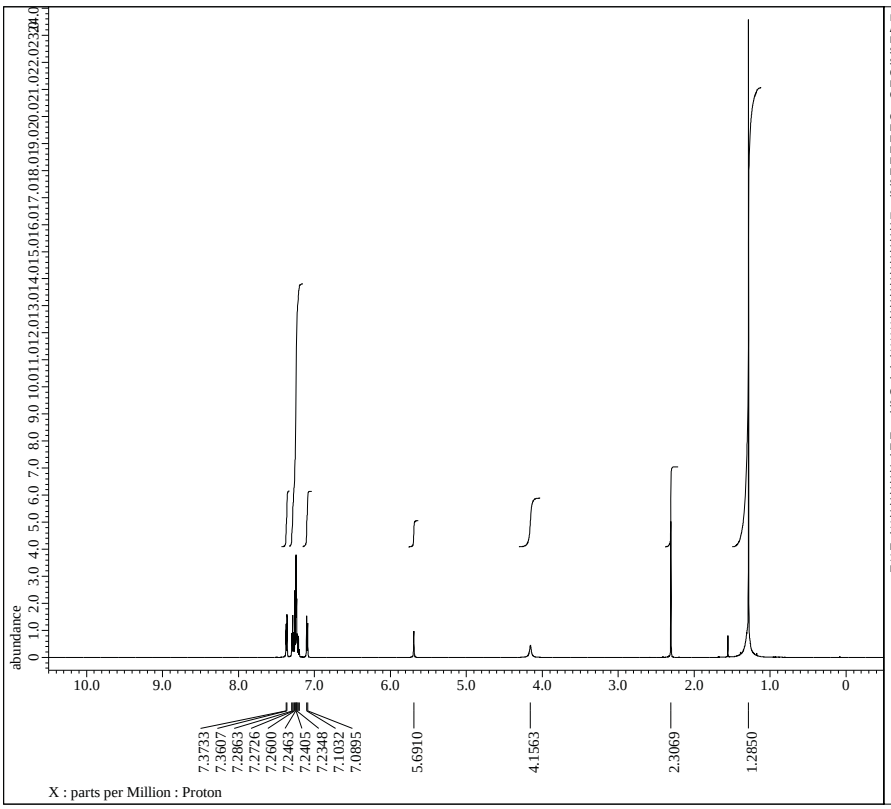
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	39.687	478359	49.737	10066
2	41.562	483426	50.263	9589



UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	39.882	979270	96.255	19726
2	41.809	38104	3.745	748

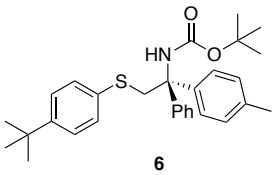



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = data hf1146
Solvent           = CHLOROFORM-D
Creation_Time     = 20-NOV-2012 19:19:11
Revision_Time     = 31-JAN-2013 19:18:10
Current_Time      = 31-JAN-2013 19:18:29

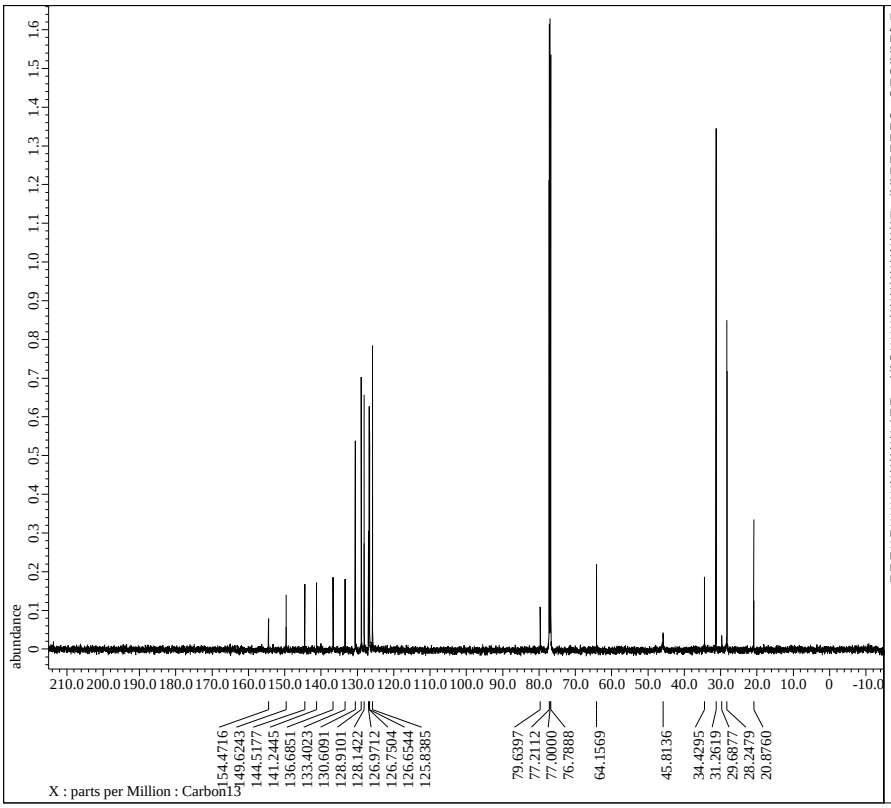
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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          = 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]
```



6

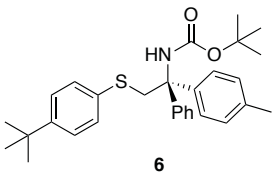



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = data hf1146re
Solvent           = CHLOROFORM-D
Creation_Time     = 23-JAN-2013 09:26:00
Revision_Time     = 23-JAN-2013 09:41:31
Current_Time      = 31-JAN-2013 19:17:14

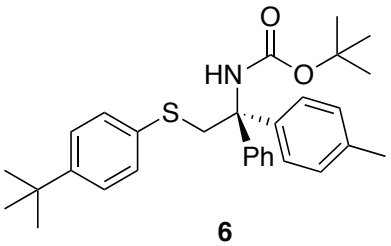
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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

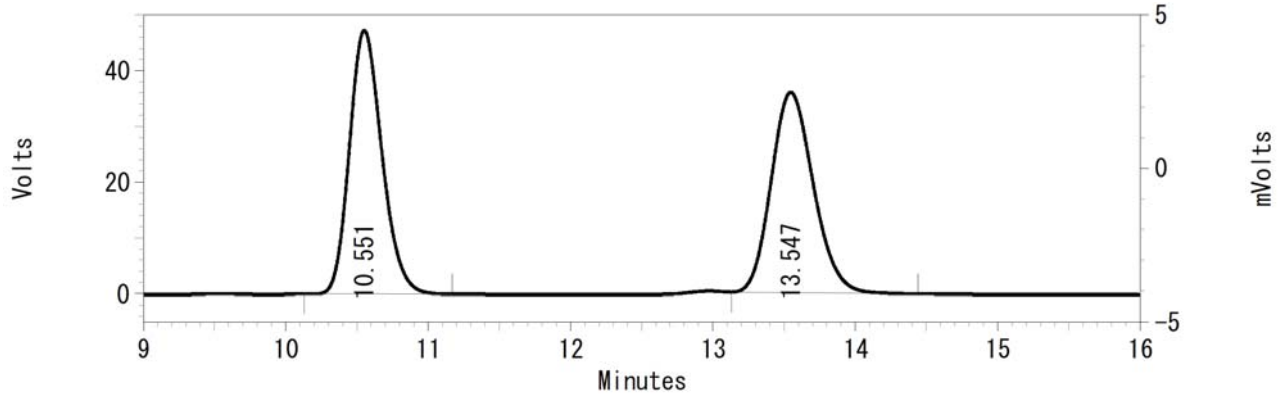
Relaxation_Delay = 2[s]
Recvr_Gain        = 58
Temp_Get          = 50[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



6

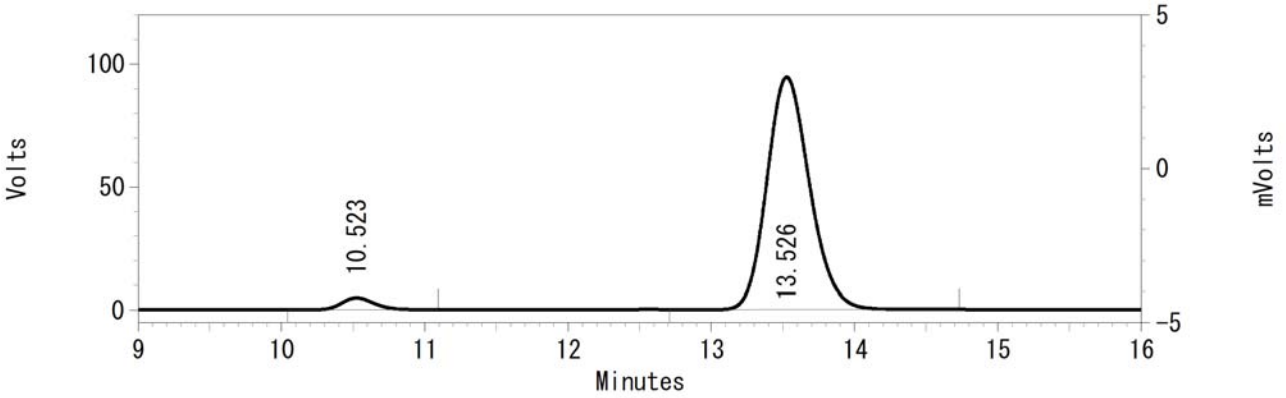


6



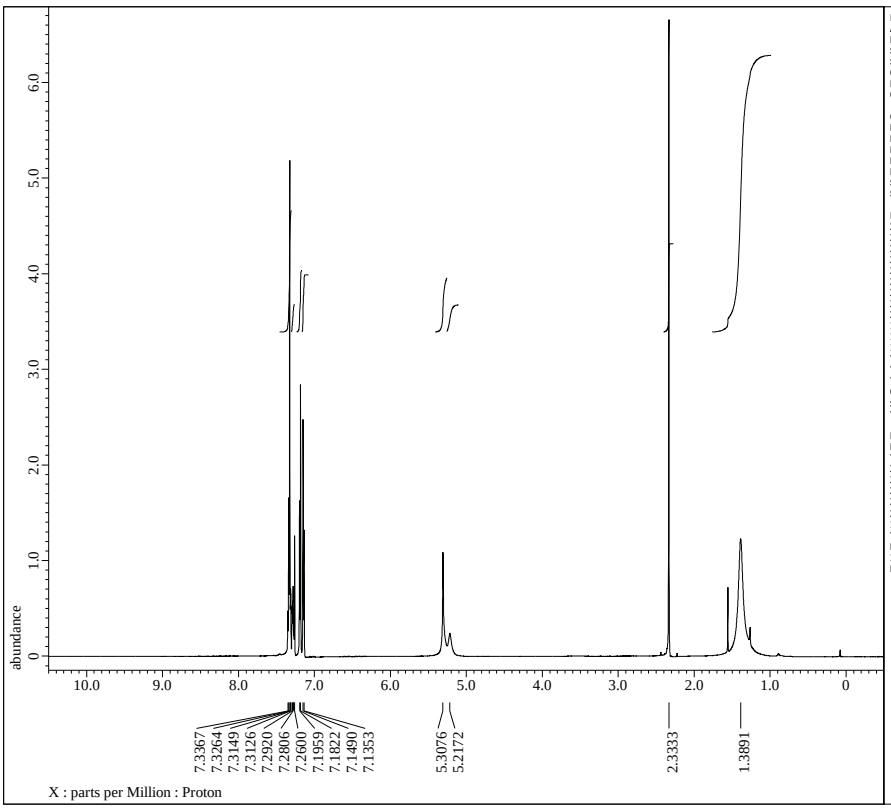
UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	10.551	748685	50.354	47214
2	13.547	738172	49.646	35899



UV Results

Pk #	Retention Time	Area	Area Percent	Height
1	10.523	75382	3.720	4787
2	13.526	1951122	96.280	94369

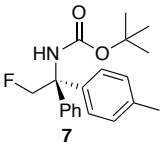



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = data hf1162
Solvent           = CHLOROFORM-D
Creation_Time     = 19-DEC-2012 22:29:31
Revision_Time     = 31-JAN-2013 19:24:58
Current_Time      = 31-JAN-2013 19:25:07

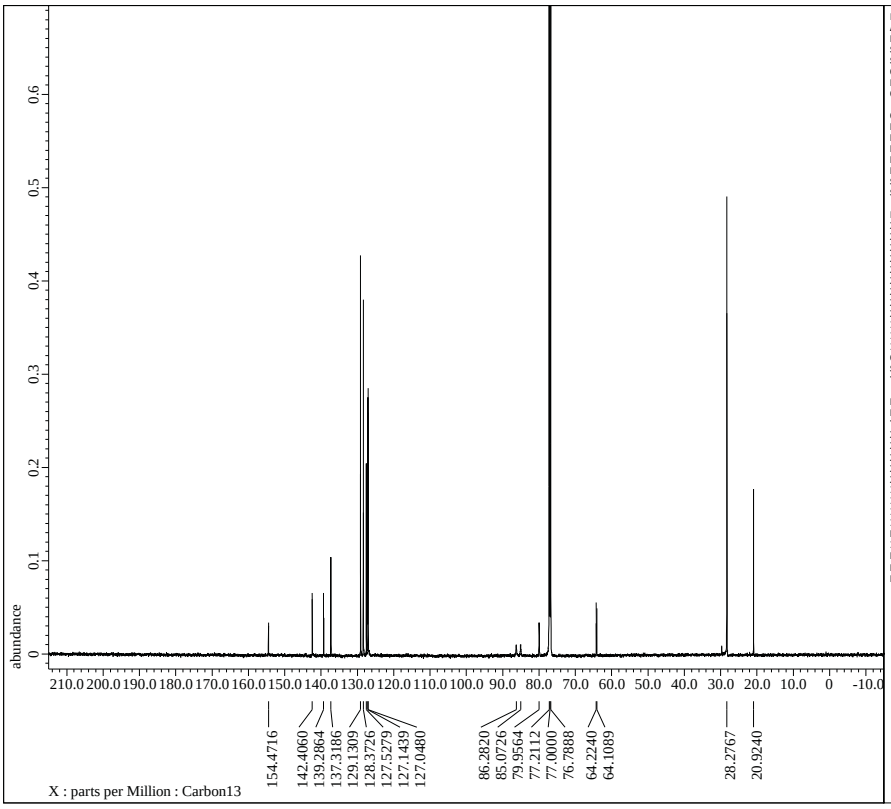
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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        = 40
Temp_Get          = 23.4[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]
```



7

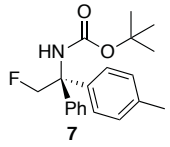



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = data hf1162rere
Solvent           = CHLOROFORM-D
Creation_Time     = 25-JAN-2013 23:54:36
Revision_Time     = 26-JAN-2013 08:26:44
Current_Time      = 31-JAN-2013 19:23:53

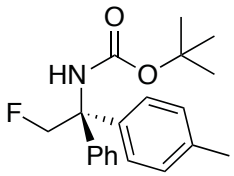
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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

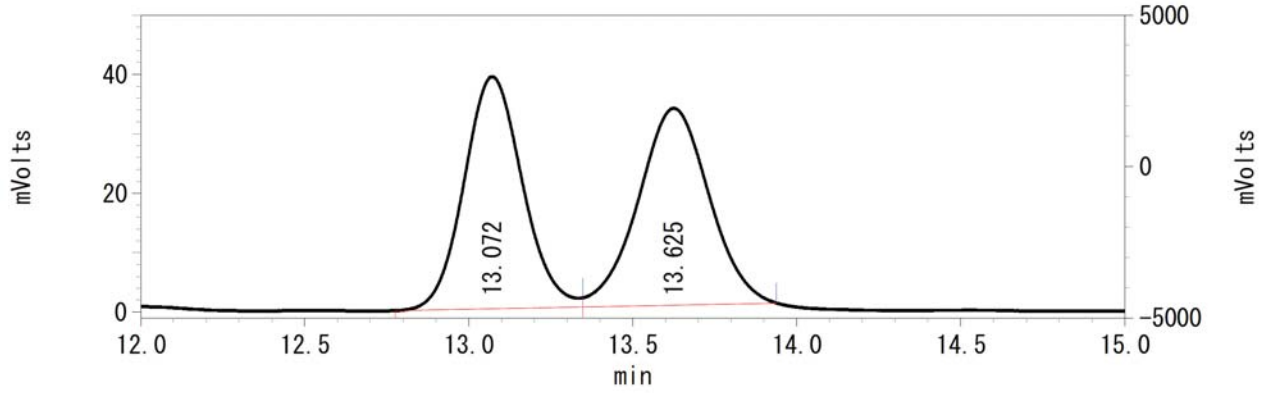
Relaxation_Delay  = 2[s]
Recvr_Gain        = 58
Temp_Get          = 50[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



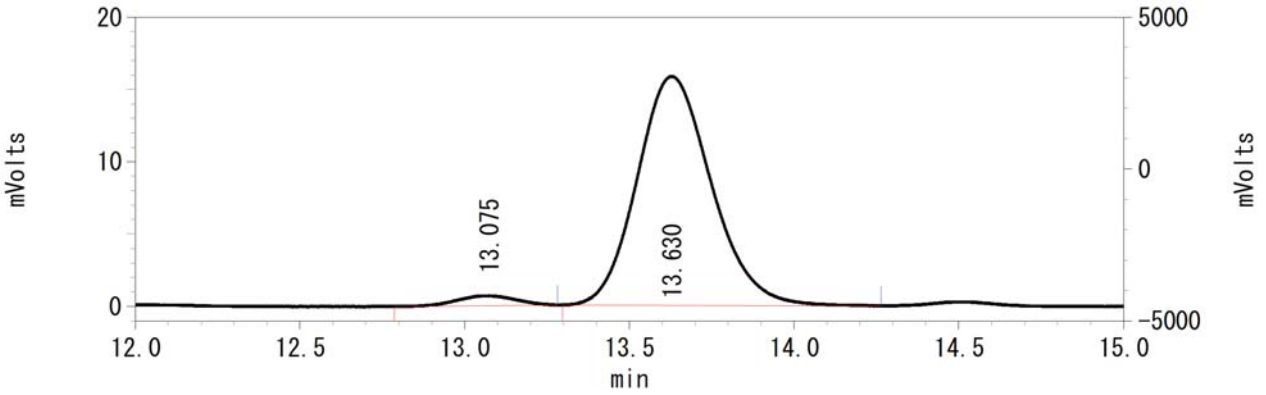
7



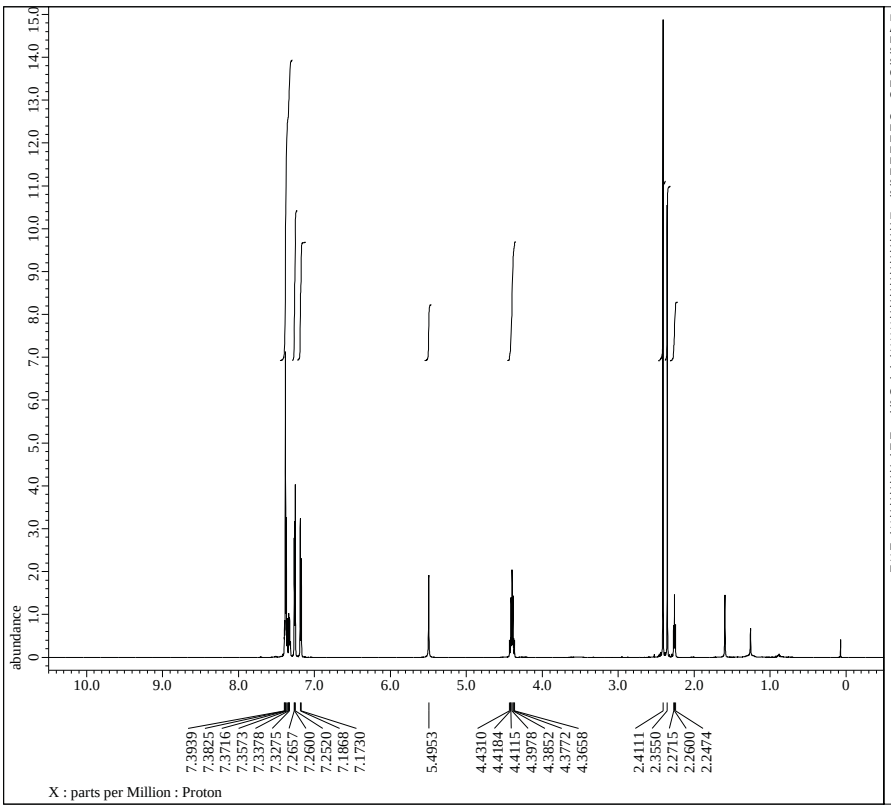
7



Pk #	Retention Time	Area	Area Percent
1	13.072	484204	49.275
2	13.625	498452	50.725



Pk #	Retention Time	Area	Area Percent
1	13.075	8541	3.327
2	13.630	248143	96.673

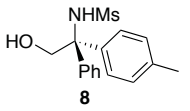



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = data hf1100
Solvent           = CHLOROFORM-D
Creation_Time     = 17-OCT-2012 20:26:23
Revision_Time     = 31-JAN-2013 19:27:33
Current_Time      = 31-JAN-2013 19:28:00

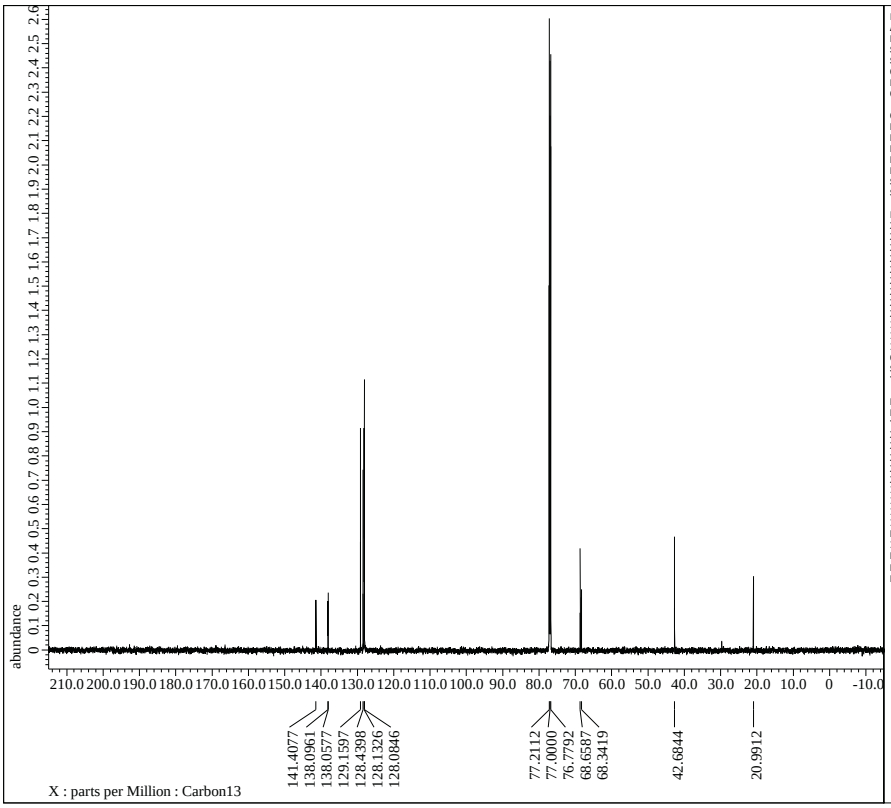
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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          = 24.5[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]
```



8

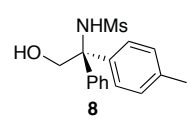



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = data hf1100
Solvent           = CHLOROFORM-D
Creation_Time     = 17-OCT-2012 20:29:11
Revision_Time     = 31-JAN-2013 19:25:41
Current_Time      = 31-JAN-2013 19:26:14

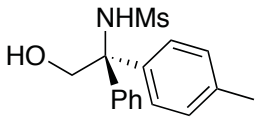
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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

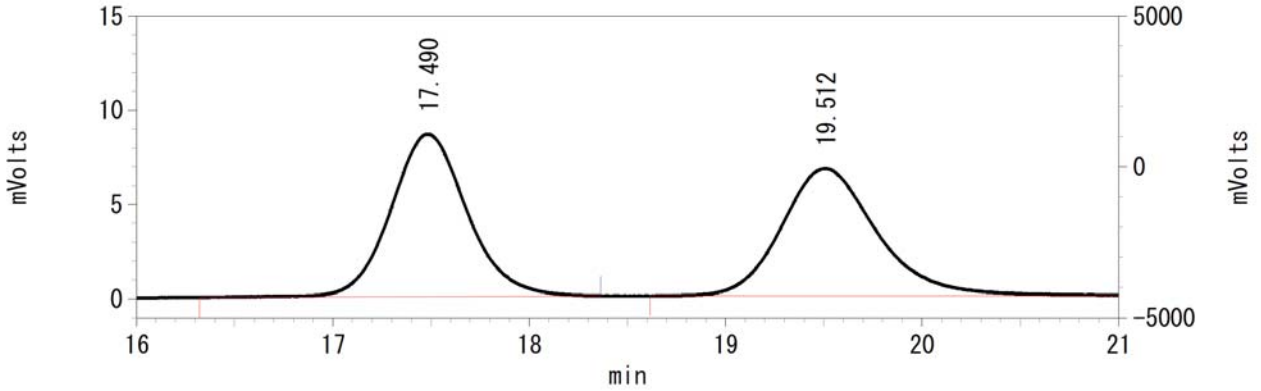
Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 25.3[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



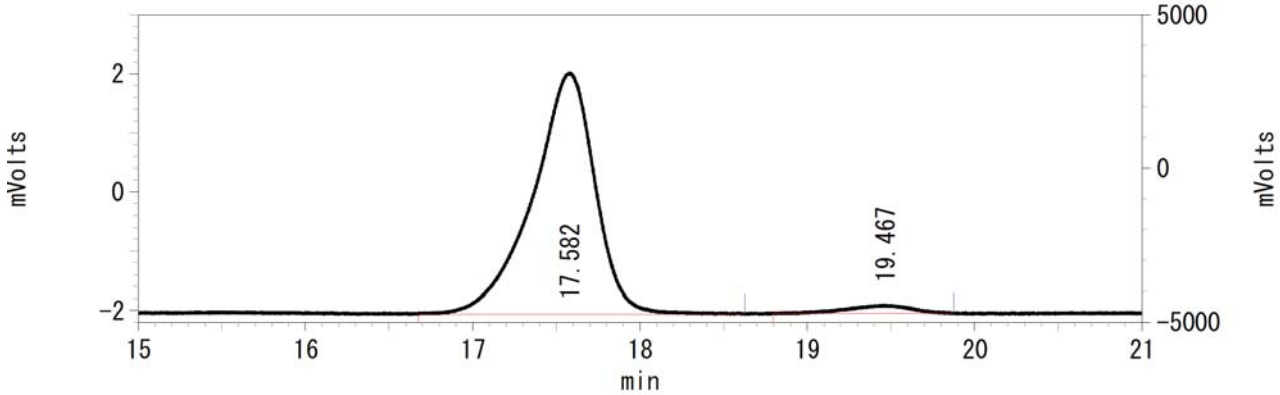
8



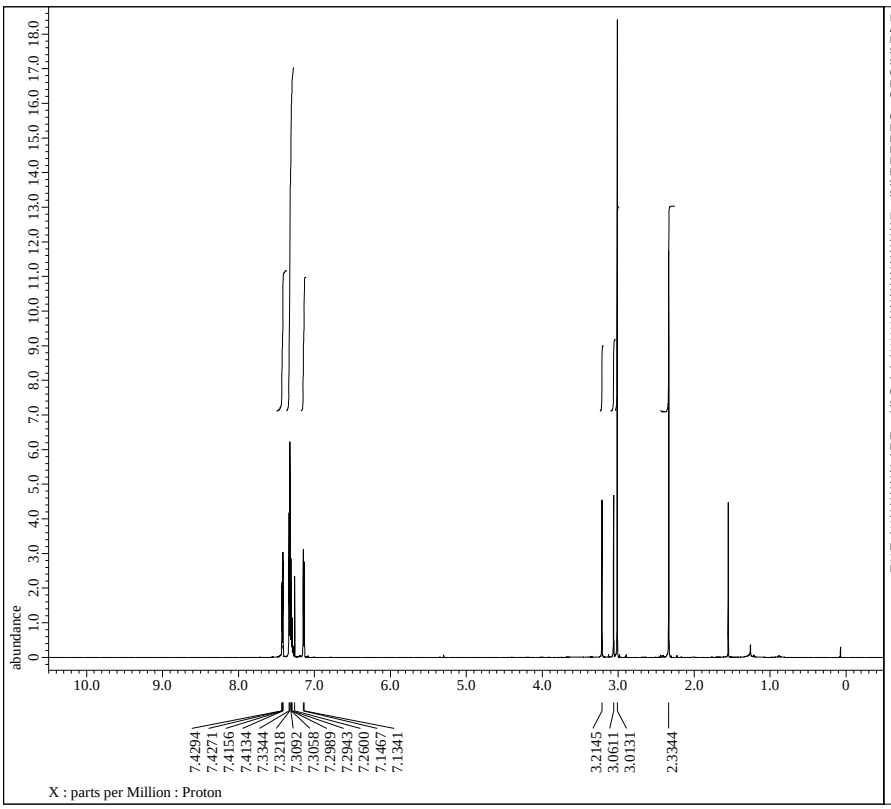
8



Pk #	Retention Time	Area	Area Percent
1	17.490	232196	50.336
2	19.512	229093	49.664



Pk #	Retention Time	Area	Area Percent
1	17.582	105322	96.698
2	19.467	3597	3.302

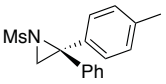



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = data hf1137
Solvent           = CHLOROFORM-D
Creation_Time     = 13-NOV-2012 21:39:33
Revision_Time     = 31-JAN-2013 19:29:45
Current_Time      = 31-JAN-2013 19:30:06

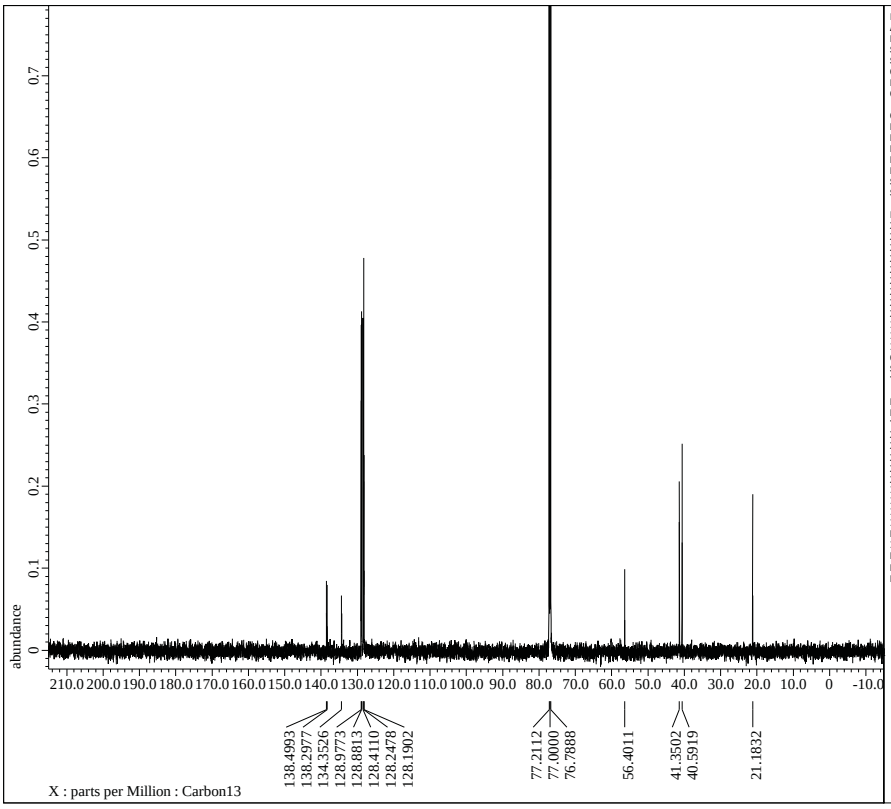
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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        = 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]
```



9

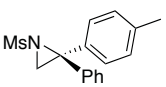



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = data hf1137
Solvent           = CHLOROFORM-D
Creation_Time     = 13-NOV-2012 21:42:22
Revision_Time     = 13-NOV-2012 21:56:33
Current_Time      = 31-JAN-2013 19:28:51

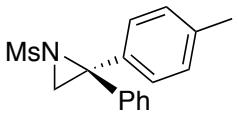
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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

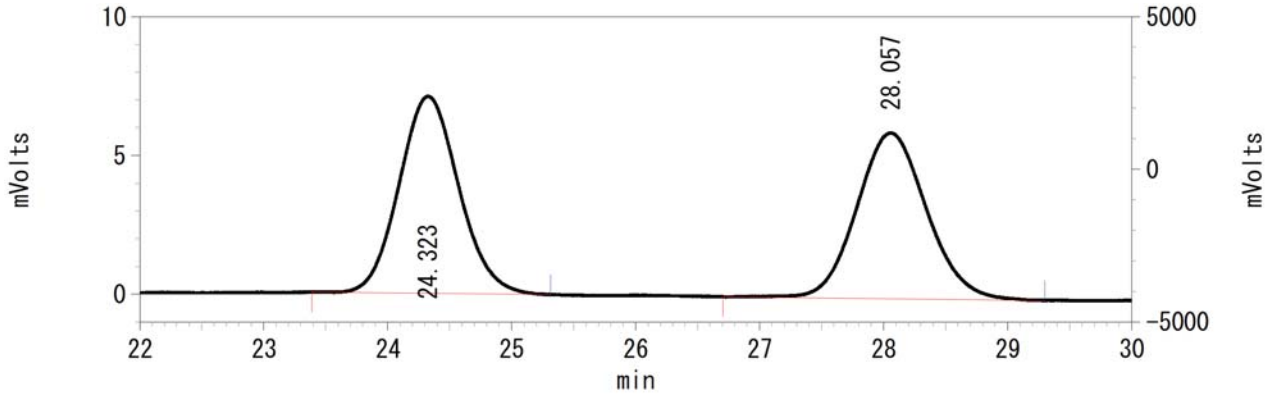
Relaxation_Delay = 2[s]
Recvr_Gain        = 58
Temp_Get          = 24.6[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



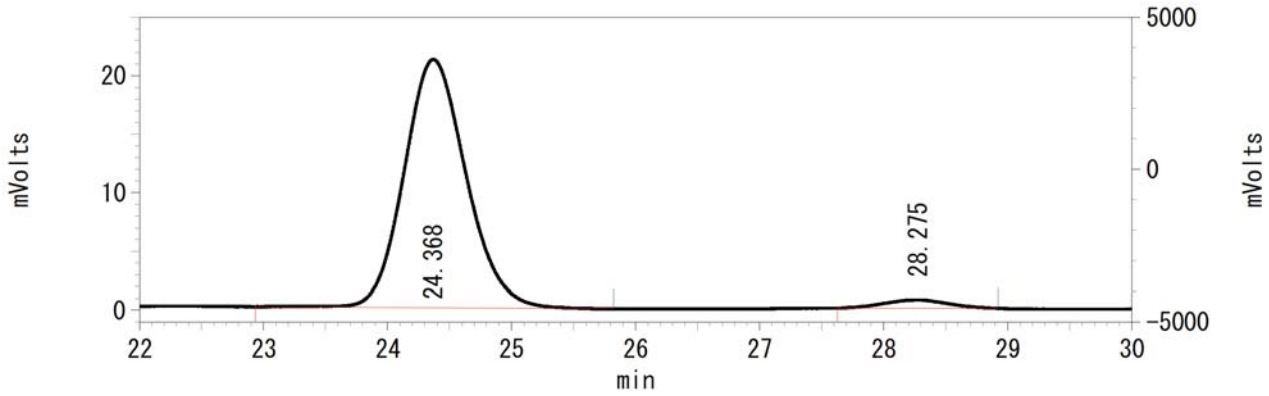
9



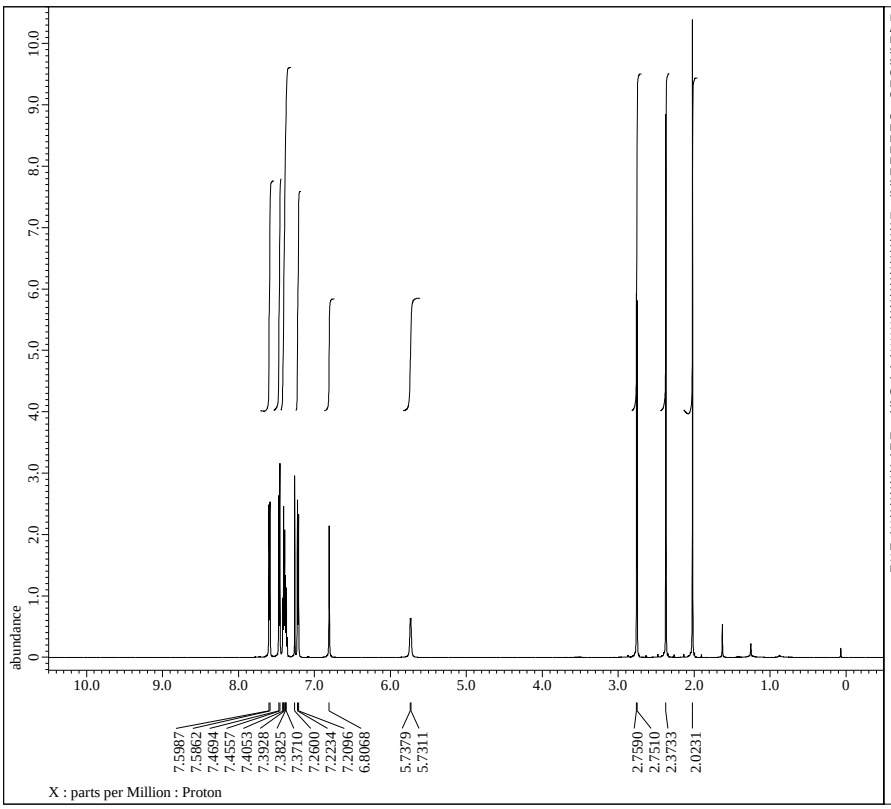
9



Pk #	Retention Time	Area	Area Percent
1	24.323	237533	50.348
2	28.057	234250	49.652



Pk #	Retention Time	Area	Area Percent
1	24.368	736358	96.653
2	28.275	25496	3.347

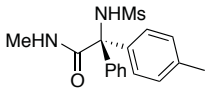



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = proton.jxp
Sample_Id         = data hf1135
Solvent           = CHLOROFORM-D
Creation_Time     = 14-NOV-2012 20:40:21
Revision_Time     = 31-JAN-2013 19:32:35
Current_Time      = 31-JAN-2013 19:33:04

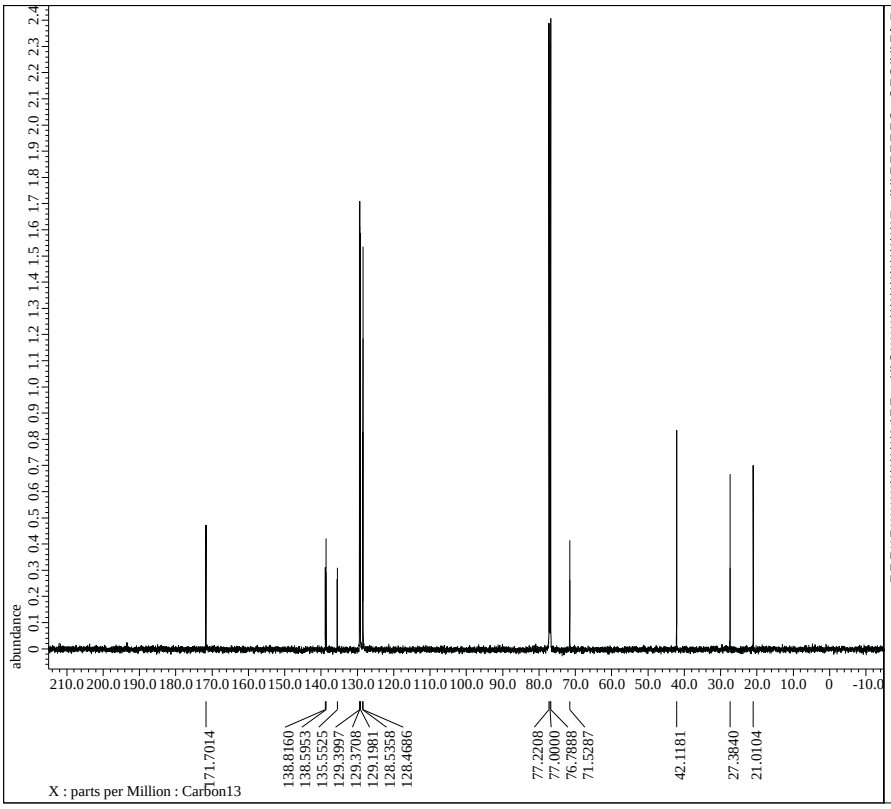
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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          = 23.8[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]
```



10

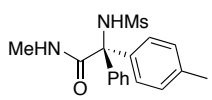



```
Filename          = c:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = data hf1135
Solvent           = CHLOROFORM-D
Creation_Time     = 14-NOV-2012 20:43:10
Revision_Time     = 14-NOV-2012 20:57:28
Current_Time      = 31-JAN-2013 19:31:21

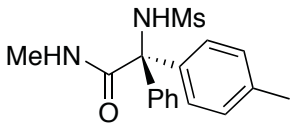
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

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

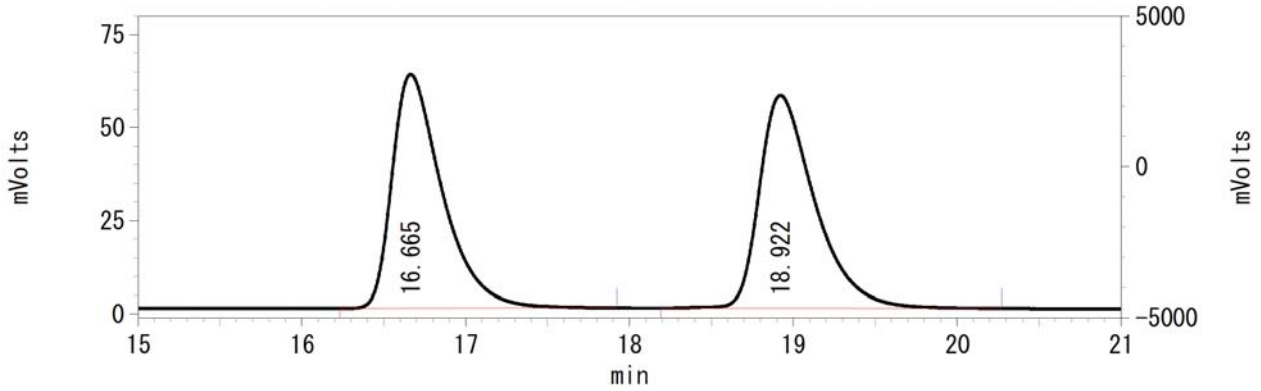
Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 24.7[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```



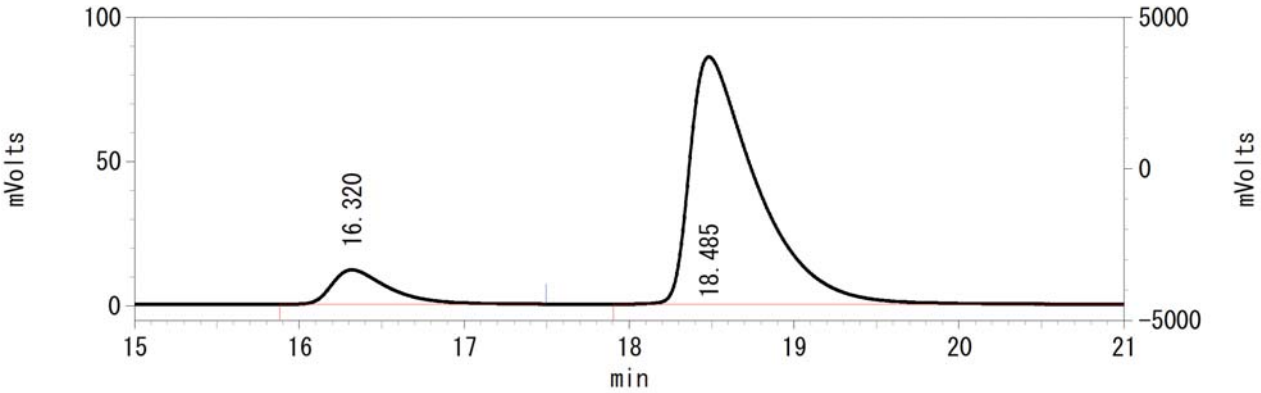
10



10



Pk #	Retention Time	Area	Area Percent
1	16.665	1336367	49.929
2	18.922	1340142	50.071



Pk #	Retention Time	Area	Area Percent
1	16.320	282395	10.466
2	18.485	2415730	89.534