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Supplementary Information

Iridium-catalysed asymmetric addition of imides to alkenes

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Contents of Supplementary Information:

1. General

All anaerobic and moisture-sensitive manipulations were carried out with standard Schlenk techniques under pre-dried nitrogen. NMR spectra were recorded on either a JEOL JNM ECZ-400 spectrometer (400 MHz for ^{1}H, 100 MHz for ^{13}C) or a Bruker Avance III HD 400 spectrometer (400 MHz for ^{1}H, 100 MHz for ^{13}C, 162 MHz for ^{31}P). Chemical shifts are reported in δ (ppm) referenced to the residual peaks of $CDCl_3$ (δ 7.26) and CD_3CN (δ 1.93) 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, quint: quintet, sext: sextet, sept: septet, m: multiplet, br: broad. Optical rotations were measured on a JASCO P-2200 polarimeter. High-resolution mass spectra were obtained with JEOL AccuTOF LC-plus 4G spectrometer. Flash column chromatography was performed with Silica Gel 60 N (Wako). Preparative thin-layer chromatography was performed with Wakogel® B-5F (Wako). Preparative recycling gel permeation chromatography (GPC) was performed using Shodex GPC FP-2002 (x 2) using chloroform as eluent.

2. Materials

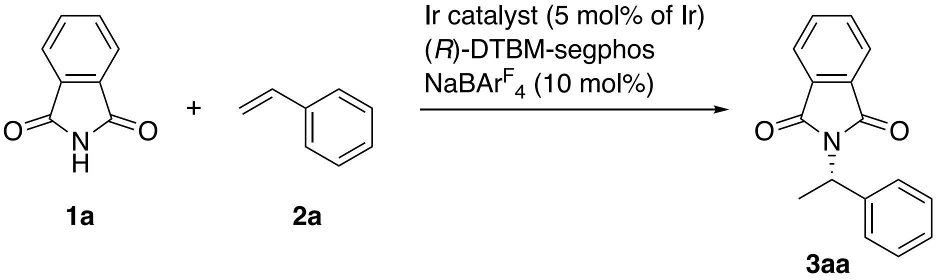
Dehydrated solvents were purchased and used after being deoxygenated by bubbling N_2. $[IrCl(cod)]_2$,[1] $[IrCl(coe)_2]_2$,[2] and $NaBAr^F_4$ [$Ar^F = 3,5\text{-}(CF_3)_2C_6H_3$][3] were prepared according to the reported procedures. Ligands (*R*)-DTBM-segphos, (*S*)-DTBM-segphos, (*S*)-segphos, (*R*)-DTBM-binap, (*R*)-binap, and (*S*)-DTBM-MeO-biphep were purchased from commercial suppliers and used as received. Imides **1b**,[4] **1c**,[5] and **1d**[6] were prepared according to the reported procedures. Alkenes **2a**, **2b**, **2e**, **2g**, **2h**, **2i**, **2j**, **2l**, **2q**, and **2t** were purchased from commercial suppliers and used after vacuum distillation. Known alkenes **2c**, **2d**, **2f**, **2k**, **2m**, **2n**, **2o**, and **2p** were prepared from the corresponding aldehydes with methyltriphenylphosphonium bromide and potassium *t*-butoxide in Et_2O. Allylsilanes **2r** and **2s** were prepared according to the reported procedures.[7] Other chemicals were purchased from commercial suppliers and used as received. Racemic compounds of **3** were synthesized by using pseudo racemic DTBM-segphos, which was prepared from an equivalent amount of (*R*)- and (*S*)-DTBM-segphos.

3. Procedure for Table S1

A mixture of $[IrCl(cod)]_2$ (1.7 mg, 0.0025 mmol, 5 mol% Ir), (*R*)-DTBM-segphos (7.1 mg, 0.0060 mmol, 6 mol%), and $NaBAr^F_4$ (9.2 mg, 0.010 mmol, 10 mol%) in CH_2Cl_2 (0.2 mL) in a Schlenk with a Teflon valve was stirred at room temperature for 10 min under N_2. Then, the green suspension was concentrated under vacuum and the resulting solid was dried under vacuum at room temperature for 10 min. The Schlenk tube was refilled with N_2, and phthalimide (**1a**, 14.7 mg, 0.10 mmol), solvent (0.2 mL), and styrene (**2a**, 31.2 mg, 0.30 mmol) were added to the tube. Then, the

Teflon valve was closed, and the mixture was stirred at 120 °C in an oil bath for 48 h. After the reaction mixture was concentrated under vacuum, the residue was subjected to preparative TLC on silica gel eluted with hexane/EtOAc (5:1) to give the addition product **3aa**. The ee was measured by HPLC with a chiral stationary column (Daicel Chiralpak ID).

Table S1 Reaction optimization



entry	Ir catalyst	solvent	temp (°C)	time (h)	yield (%)[b]	ee (%)[c]
1	$[IrCl(cod)]_2$	1,2-dichloroethane	120	18	16	N.D
2	$[IrCl(coe)_2]_2$	1,2-dichloroethane	120	18	trace	N.D
3	$[IrCl(cod)_2]BF_4$	1,2-dichloroethane	120	18	0	–
4	$[IrCl(cod)_2]OTf$	1,2-dichloroethane	120	18	0	–
5	$[IrCl(cod)]_2$	1,2-dichloroethane	120	72	59	96
6	$[IrCl(cod)]_2$	toluene	120	48	36	96
7	$[IrCl(cod)]_2$	1,2-dimethoxyethane	120	72	0	–
8	$[IrCl(cod)]_2$	CH_3CN	120	72	0	–
9	$[IrCl(cod)]_2$	*N*-methylpyrrolidone	120	72	0	–
10	$[IrCl(cod)]_2$	cyclohexane	120	48	50	96
11	$[IrCl(cod)]_2$	methylcyclohexane	120	48	63	95
12	$[IrCl(cod)]_2$	methylcyclohexane	120	18	6	N.D
13[d]	$[IrCl(cod)]_2$	methylcyclohexane	120	18	30	N.D
14[d,e]	$[IrCl(cod)]_2$	methylcyclohexane	120	18	27	N.D
15[d,e]	$[IrCl(cod)]_2$	methylcyclohexane	140	48	91	N.D
16[d,e]	$[IrCl(cod)]_2$	methylcyclohexane	140	18	35	N.D
17[d,e,f]	$[IrCl(cod)]_2$	methylcyclohexane	140	18	65	93
18[d,e,f,g]	$[IrCl(cod)]_2$	methylcyclohexane	140	18	0	–

[a] Reaction conditions: **1a** (0.10 mmol), **2a** (0.30 mmol), Ir catalyst (5 mol% of Ir), (*R*)-DTBM-segphos (0.0060 mmol, 6 mol%), and $NaBAr^F_4$ (0.020 mmol, 10 mol%) in solvent (0.2 mL). N.D.: Not determined. [b] Isolated yields. [c] Dertermined by HPLC with a chiral stationary phase column: Chiralpak ID. [d] Distilled **1a** was used. [e] Performed in methylcyclohexane (0.4 mL). [f] Performed with 5 mol% of (*R*)-DTBM-segphos. [g] Without $NaBAr^F_4$.

4. Procedure for Table 1

For Entries 1-5

A mixture of $[IrCl(cod)]_2$ (1.7 mg, 0.0025 mmol, 5 mol% Ir), ligand (0.0050 mmol, 5 mol%), and $NaBAr^F{}_4$ (9.2 mg, 0.010 mmol, 10 mol%) in CH_2Cl_2 (0.2 mL) in a Schlenk tube with a Teflon valve was stirred at room temperature for 5 min under N_2. Then, the green suspension was concentrated under vacuum and the resulting solid was dried under vacuum at room temperature for 5 min. The Schlenk tube was refilled with N_2, and phthalimide (**1a**, 14.7 mg, 0.10 mmol), methylcyclohexane (0.4 mL), and styrene (**2a**, 31.2 mg, 0.30 mmol) were added to the tube. Then, the Teflon valve was closed, and the mixture was stirred at 140 °C in an oil bath or in a heating block for 48 h. After the reaction mixture was concentrated under vacuum, the residue was subjected to preparative TLC on silica gel eluted with hexane/EtOAc (5:1) to give the addition product **3aa**. The ee was measured by HPLC with a chiral stationary column (Daicel Chiralpak ID).

For Entry 6

A mixture of $[Ir(cod)_2]BAr^F{}_4$ (6.4 mg, 0.0050 mmol, 5 mol%), (*S*)-DTBM-segphos (5.9 mg, 0.0050 mmol, 5 mol%) in CH_2Cl_2 (0.2 mL) in a Schlenk tube was stirred at room temperature for 5 min under N_2. Then, the suspension was concentrated under vacuum and the resulting solid was dried under vacuum at room temperature for 5 min. The Schlenk tube was refilled with N_2, and phthalimide (**1a**, 14.7 mg, 0.10 mmol), methylcyclohexane (0.4 mL), and styrene (**2a**, 31.2 mg, 0.30 mmol) were added to the tube. Then, the Teflon valve was closed, and the mixture was stirred at 140 °C in a heating block for 48 h. After the reaction mixture was concentrated under vacuum, the residue was subjected to preparative TLC on silica gel eluted with hexane/EtOAc (5:1) to give the addition product **3aa**.

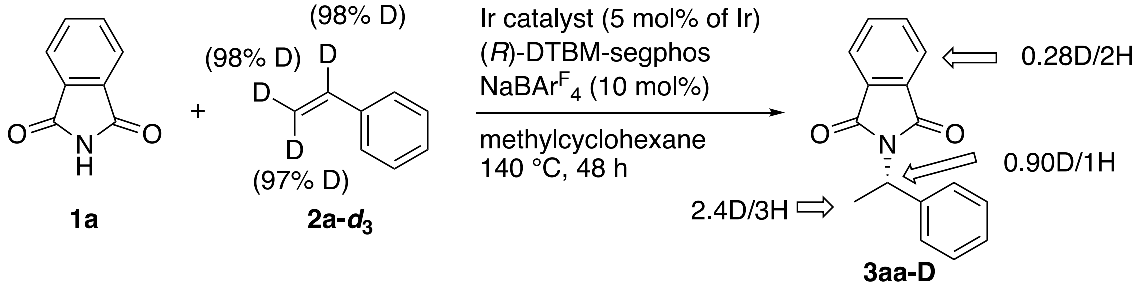
5. Procedure for Schemes 2 and 3

A mixture of $[IrCl(cod)]_2$ (1.7 mg, 0.0025 mmol, 5 mol% Ir), (*S*)-DTBM-segphos (5.9 mg, 0.0050 mmol, 5 mol%), and $NaBAr^F{}_4$ (9.2 mg, 0.010 mmol, 10 mol%) in CH_2Cl_2 (0.2 mL) in a Schlenk tube with a Teflon valve was stirred at room temperature for 5 min under N_2. Then, the green suspension was concentrated under vacuum and the resulting solid was dried under vacuum at room temperature for 5 min. The Schlenk tube was refilled with N_2, and phthalimide **1** (0.10 mmol), methylcyclohexane (0.4 mL), and alkene **2** (0.30 mmol) were added to the tube. Then, the Teflon valve was closed, and the mixture was stirred at 140 °C in an oil bath or in a heating block for 48 h. After the reaction mixture was concentrated under vacuum, the residue was subjected to preparative TLC on silica gel eluted with hexane/EtOAc to give the addition product **3**. The ee was measured by HPLC with chiral stationary columns.

6. NMR experiments for the reaction of a cationic iridium complex with phthalimide

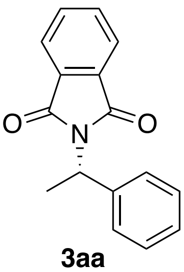
A mixture of [IrCl(coe)$_2$]$_2$ (4.4 mg, 0.0050 mmol), (*S*)-DTBM-segphos (11.8 mg, 0.010 mmol, 5 mol%), NaBAr$^F{}_4$ (9.2 mg, 0.010 mmol), and phthalimide (2.9 mg, 0.020 mmol, 2 equiv) in CD$_3$CN (0.6 mL) in an NMR tube was heated at 80 °C in an oil bath for 15 min under N$_2$. After cooling to room temperature, nitromethane (4.7 mg) was added as an internal standard, and the sample was measured by ^{1}H NMR at room temperature. Major two peaks of the hydrides are as follows; ^{1}H NMR (400 MHz, CD$_3$CN) δ –18.0 (t, $J_{\text{P-H}}$ = 15 Hz, 1H), –20.0 (dd, $J_{\text{P-H}}$ = 23, 14 Hz, 0.17H); ^{31}P {^{1}H} NMR (162 MHz, CD$_3$CN) δ –0.90 (d, $J_{\text{P-P}}$ = 21 Hz), –2.96 (d, $J_{\text{P-P}}$ = 21 Hz).

7. Reaction of phthalimide with styrene-*d*$_3$



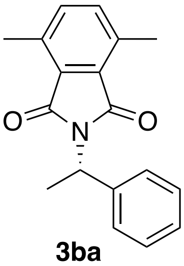
A mixture of [IrCl(cod)]$_2$ (1.7 mg, 0.0025 mmol, 5 mol% Ir), (*S*)-DTBM-segphos (5.9 mg, 0.0050 mmol, 5 mol%), and NaBAr$^F{}_4$ (9.2 mg, 0.010 mmol, 10 mol%) in CH$_2$Cl$_2$ (0.2 mL) in a Schlenk tube with a Teflon valve was stirred at room temperature for 5 min under N$_2$. Then, the green suspension was concentrated under vacuum and the resulting solid was dried under vacuum at room temperature for 5 min. The Schlenk tube was refilled with N$_2$, and phthalimide **1** (14.7 mg, 0.10 mmol), methylcyclohexane (0.4 mL), and styrene-*d*$_3$[8] (**2a-*d*$_3$**, 32.2 mg, 0.30 mmol) were added to the tube. Then, the Teflon valve was closed, and the mixture was stirred at 140 °C in an oil bath or in a heating block for 48 h. After the reaction mixture was concentrated under vacuum, the residue was subjected to preparative TLC on silica gel eluted with hexane/EtOAc (5:1) to give the addition product **3aa-D** (colorless oil, 9.7 mg, 40% yield). colorless oil, 9.7 mg, 40% yield). ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.83–7.77 (m, 1.72H), 7.73–7.65 (m, 2H), 7.50 (d, *J* = 7.2 Hz, 2H), 7.33–7.30 (m, 2H), 7.29–7.23 (m, 1H), 5.60–5.53 (m, 0.10H), 1.93–1.87 (m, 0.61H); ^{2}H NMR (61 MHz, CHCl$_3$) δ 7.85 (s, 0.28D), 5.55 (s, 0.90D), 1.90 (s, 2.4D).

8. Characterization of the products



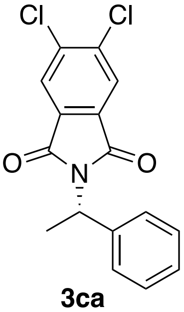
3aa

Compound 3aa (CAS: 3976-26-9 for (*S*)-**3aa**, Table 1, entry 1, colorless oil, 21.4 mg, 85% yield, 93% ee (*S*)). A solution of EtOAc/hexane (1:5) was used as an eluent for preparative TLC. The ee was measured by HPLC [Chiralpak ID, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 14.3 min (minor), t_2 = 15.1 min (major)]. $[\alpha]^{25}_{D}$ –60 (*c* 0.96, $CHCl_3$) for 93% ee (*S*); ^1H NMR (400 MHz, $CDCl_3$) δ 7.83–7.77 (m, 2H), 7.73–7.65 (m, 2H), 7.50 (d, *J* = 7.2 Hz, 2H), 7.33 (t, *J* = 7.2 Hz, 2H), 7.26 (t, *J* = 7.2 Hz, 1H), 5.58 (q, *J* = 7.6 Hz, 1H), 1.93 (d, *J* = 7.6 Hz, 3H). The absolute configuration of **3aa** was determined to be *S* by comparison of the specific rotation with the reported values ($[\alpha]^{23}_{D}$ –31.4 (*c* 0.5, $CHCl_3$) for (*S*)-**3aa**,[9] $[\alpha]^{25}_{D}$ +86.3 (*c* 0.31, $CHCl_3$) for (*R*)-**3aa**[10]). For other compounds except for **3at**, the absolute configurations were assigned by analogy with **3aa**.



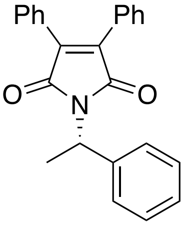
3ba

Compound 3ba (Scheme 2, colorless solid, 23.2 mg, 83% yield, 82% ee (*S*)). A solution of EtOAc/hexane (1:5) was used as an eluent for preparative TLC. The ee was measured by HPLC [Chiralpak ID, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 10.9 min (minor), t_2 = 11.8 min (major)]. $[\alpha]^{25}_{D}$ –64 (*c* 0.95, $CHCl_3$) for 82% ee (*S*); ^1H NMR (400 MHz, $CDCl_3$) δ 7.49 (d, *J* = 9.6 Hz, 2H), 7.36–7.19 (m, 5H), 5.53 (q, *J* = 9.6 Hz, 1H), 2.60 (s, 6H), 1.89 (d, *J* = 9.6 Hz, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 168.9, 140.5, 135.9, 135.2, 128.7, 128.4, 127.5, 127.4, 48.9, 17.4, 17.3; HRMS (DART-TOF) m/z: $[M + H]^+$ Calcd for $C_{18}H_{18}NO_2$ 280.1332; Found 280.1329.



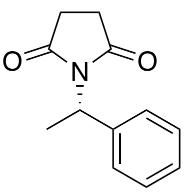
3ca

Compound 3ca (Scheme 2, colorless solid, 6.4 mg, which was obtained after purification by GPC to remove a small amount of impurities, 20% yield, 92% ee (*S*)). A solution of EtOAc/hexane (1:5) was used as an eluent for preparative TLC. The ee was measured by HPLC [Chiralpak ID, hexane/2-propanol = 19:1, flow 0.5 mL/min, 254 nm, $t_1 = 13.8$ min (major), $t_2 = 15.8$ min (minor)]. $[\alpha]^{25}_{D}$ –41 (*c* 0.56, $CHCl_3$) for 92% ee (*S*); ^1H NMR (400 MHz, $CDCl_3$) δ 7.88 (s, 2H), 7.47 (d, *J* = 7.6 Hz, 2H), 7.36–7.24 (m, 3H), 5.54 (q, *J* = 7.2 Hz, 1H), 1.92 (d, *J* = 7.2 Hz, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 166.1, 139.7, 138.8, 131.1, 128.6, 127.9, 127.4, 125.3, 50.2, 17.4; HRMS (DART-TOF) m/z: $[M + H]^+$ Calcd for $C_{16}H_{12}{}^{35}ClNO_2$ 320.0230; Found 320.0240.



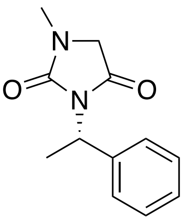
3da

Compound 3da (Scheme 2, yellow solid, 23.4 mg, which was obtained after purification by GPC to remove a small amount of impurities, 66% yield, 92% ee (*S*)). A solution of EtOAc/hexane (1:5) was used as an eluent for preparative TLC. The ee was measured by HPLC [Chiralpak AD-H, hexane/2-propanol = 30:1, flow 0.5 mL/min, 254 nm, $t_1 = 15.4$ min (minor), $t_2 = 22.5$ min (major)]. $[\alpha]^{25}_{D}$ –44 (*c* 1.10, $CHCl_3$) for 92% ee (*S*); ^1H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 7.2 Hz, 2H), 7.48–7.43 (m, 4H), 7.40–7.27 (m, 9H), 5.53 (q, *J* = 7.6 Hz, 1H), 1.93 (d, *J* = 7.6 Hz, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 170.4, 140.4, 135.9, 129.9, 129.7, 128.54, 128.49, 128.46, 127.7, 127.6, 50.1, 17.6; HRMS (DART-TOF) m/z: $[M + H]^+$ Calcd for $C_{24}H_{20}NO_2$ 354.1489; Found 354.1481.



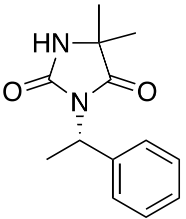
3ea

Compound 3ea (CAS: 62993-44-6 for (*S*)-**3ea**, Scheme 2: colorless solid, 13.2 mg, 65% yield, 90% ee (*S*)). A solution of MeOH/CH_2Cl_2 (1:100) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IB, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 22.9 min (major), t_2 = 27.6 min (minor)). $[\alpha]^{25}_{D}$ –78 (*c* 0.80, $CHCl_3$) for 90% ee (*S*); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, *J* = 7.6 Hz, 2H), 7.30 (t, *J* = 7.6 Hz, 2H), 7.26 (d, *J* = 7.6 Hz, 1H), 5.40 (q, *J* = 7.2 Hz, 1H), 2.62 (s, 4H), 1.79 (d, *J* = 7.2 Hz, 3H).



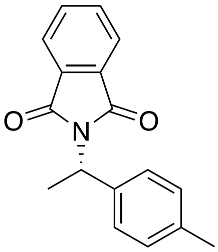
3fa

Compound 3fa (Scheme 2: colorless solid, 14.6 mg, 67% yield, 89% ee (*S*)). A solution of MeOH/CH_2Cl_2 (1:50) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak ID, hexane/2-propanol = 4:1, flow 0.5 mL/min, 254 nm, t_1 = 17.4 min (minor), t_2 = 18.9 min (major)). $[\alpha]^{25}_{D}$ –51 (*c* 0.64, $CHCl_3$) for 89% ee (*S*); ^{1}H NMR ($CDCl_3$) δ 7.49 (d, *J* = 7.2 Hz, 2H), 7.34 (t, *J* = 7.2 Hz, 2H), 7.27 (t, *J* = 7.2 Hz, 1H), 5.33 (q, *J* = 7.6 Hz, 1H), 3.82 (d, *J* = 17.4 Hz, 1H), 3.76 (d, *J* = 17.4 Hz, 1H), 2.95 (s, 3H), 1.84 (d, *J* = 7.6 Hz, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 169.5, 156.5, 140.0, 128.4, 127.8, 127.5, 51.3, 50.6, 29.5, 17.0; HRMS (DART-TOF) m/z: $[M + H]^+$ Calcd for $C_{12}H_{15}N_2O_2$ 219.1128; Found 219.1122.



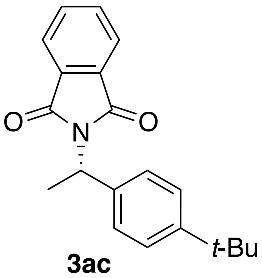
3ga

Compound 3ga (Scheme 2: colorless solid, 20.7 mg, 89% yield, 93% ee (*S*)). The ee was measured by HPLC (Chiralpak ID, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 13.3 min (minor), t_2 = 14.3 min (major)). $[\alpha]^{25}_{D}$ –39 (*c* 0.66, $CHCl_3$) for 93% ee (*S*); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, *J* = 7.6 Hz, 2H), 7.32 (t, *J* = 7.6 Hz, 2H), 7.26 (t, *J* = 7.6 Hz, 1H), 5.59 (brs, 1H), 5.32 (q, J = 7.2 Hz, 1H), 1.83 (d, J = 7.2 Hz, 3H), 1.40 (s, 3H), 1.37 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 177.0, 156.0, 140.1, 128.5, 127.6, 127.1, 58.0, 50.0, 25.1, 25.0, 17.2; HRMS (DART-TOF) m/z: $[M + H]^+$ Calcd for $C_{13}H_{17}N_2O_2$ 233.1285; Found 233.1290.



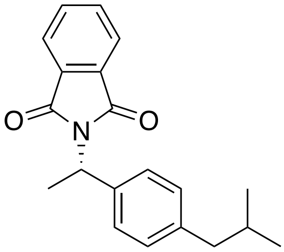
3ab

Compound 3ab (CAS: 36244-75-4 for **3ab**, Scheme 3: colorless oil, 24.8 mg, 93% yield, 93% ee). A solution of EtOAc/hexane (1:10) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak AD-H, hexane/2-propanol = 30:1, flow 0.5 mL/min, 254 nm, t_1 = 22.5 min (major), t_2 = 25.1 min (minor)). $[\alpha]^{25}_D$ –66 (c 1.17, $CHCl_3$) for 93% ee (S); ^1H NMR (400 MHz, $CDCl_3$) δ 7.83–7.76 (m, 2H), 7.71–7.65 (m, 2H), 7.41 (d, J = 8.0 Hz, 2H), 7.13 (d, J = 8.0 Hz, 2H), 5.54 (q, J = 7.2 Hz, 1H), 2.31 (s, 3H), 1.91 (d, J = 7.2 Hz, 3H).



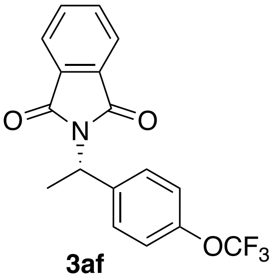
3ac

Compound 3ac (CAS: 2763070-09-1 for **3ac**, Scheme 3: colorless oil, 28.1 mg, 91% yield, 90% ee (S)). A solution of EtOAc/hexane (1:5) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak AD-H, hexane/2-propanol = 100:1, flow 1.0 mL/min, 254 nm, t_1 = 11.5 min (major), t_2 = 12.4 min (minor)). $[\alpha]^{25}_D$ –52 (c 1.40, $CHCl_3$) for 90% ee (S); ^1H NMR (400 MHz, $CDCl_3$) δ 7.84–7.76 (m, 2H), 7.72–7.63 (m, 2H), 7.45 (d, J = 8.4 Hz, 2H), 7.34 (d, J = 8.4 Hz, 2H), 5.55 (q, J = 7.2 Hz, 1H), 1.92 (d, J = 7.2 Hz, 3H), 1.29 (s, 9H).



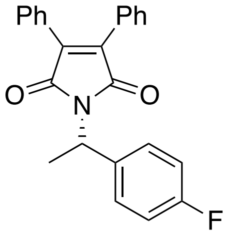
3ad

Compound 3ad (Scheme 3: colorless solid, 24.3 mg, 79% yield, 93% ee (*S*)). A solution of EtOAc/hexane (1:5) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak ID, hexane/2-propanol = 100:1, flow 0.5 mL/min, 254 nm, t_1 = 22.3 min (major), t_2 = 24.7 min (minor)). $[\alpha]^{25}_{D}$ –55 (*c* 1.14, $CHCl_3$) for 93% ee (*S*); ^1H NMR (400 MHz, $CDCl_3$) δ 7.83–7.76 (m, 2H), 7.70–7.65 (m, 2H), 7.41 (d, *J* = 7.8 Hz, 2H), 7.10 (d, *J* = 7.8 Hz, 2H), 5.55 (q, *J* = 7.2 Hz, 1H), 2.43 (d, *J* = 7.6 Hz, 2H), 1.92 (d, *J* = 7.2 Hz, 3H), 1.83 (sept, *J* = 6.4 Hz, 1H), 0.88 (d, *J* = 6.4 Hz, 6H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 168.2, 141.1, 137.5, 133.8, 132.0, 129.1, 127.2, 123.1, 49.4, 45.0, 30.1, 22.4, 17.6; HRMS (DART-TOF) m/z: $[M + H]^+$ Calcd for $C_{20}H_{22}NO_2$ 308.1645; Found 308.1643.



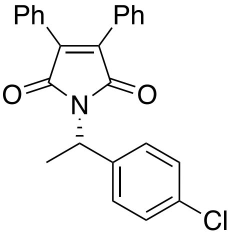
3af

Compound 3af (Scheme 3: colorless oil, 6.8 mg, 20% yield, 90% ee (*S*)). A solution of EtOAc/hexane (1:10) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak ID, hexane/2-propanol = 200:1, flow 0.5 mL/min, 254 nm, t_1 = 23.4 min (major), t_2 = 25.8 min (minor)). $[\alpha]^{25}_{D}$ –57 (*c* 0.34, $CHCl_3$) for 90% ee (*S*); ^1H NMR (400 MHz, $CDCl_3$) δ 7.85–7.78 (m, 2H), 7.75–7.67 (m, 2H), 7.54 (d, *J* = 8.4 Hz, 2H), 7.17 (d, *J* = 8.4 Hz, 2H), 5.56 (q, *J* = 7.4 Hz, 1H), 1.92 (d, *J* = 7.4 Hz, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 168.0, 148.5, 138.9, 134.0, 131.8, 129.0, 123.3, 120.9, 120.4 (q, $J_{F\text{-}C}$ = 257 Hz), 48.9, 17.5; HRMS (DART-TOF) m/z: $[M + H]^+$ Calcd for $C_{17}H_{13}F_3NO_3$ 336.0842; Found 336.0828.



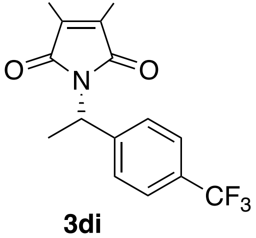
3dg

Compound 3dg (Scheme 3: yellow solid, 32.1 mg, 86% yield, 93% ee (*S*)). A solution of EtOAc/hexane (1:5) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak AD–H ×2, hexane/2-propanol = 30:1, flow 0.5 mL/min, 254 nm, t_1 = 31.9 min (minor), t_2 = 40.7 min (major)). $[\alpha]^{25}_{D}$ –46 (*c* 1.29, $CHCl_3$) for 93% ee (*S*); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56–7.49 (m, 2H), 7.45 (dd, *J* = 8.0, 1.6 Hz, 4H), 7.40–7.30 (m, 6H), 7.03 (t, *J* = 8.8 Hz, 2H), 5.49 (q, *J* = 7.6 Hz, 1H), 1.91 (d, *J* = 7.6 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 170.4, 162.2 (d, $J_{C\text{–}F}$ = 245 Hz), 136.2, 136.0, 129.9, 129.8, 129.3 (d, $J_{C\text{–}F}$ = 9 Hz), 128.5, 115.3 (d, $J_{C\text{–}F}$ = 21 Hz), 49.4, 17.8; HRMS (DART-TOF) m/z: $[M + H]^+$ Calcd for $C_{24}H_{19}FNO_2$ 372.1394; Found 372.1387.



3dh

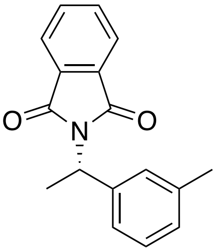
Compound 3dh (Scheme 3: yellow solid, 28.8 mg, 74% yield, 94% ee (*S*)). A solution of EtOAc/hexane (1:5) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak AD–H ×2, hexane/2-propanol = 30:1, flow 0.5 mL/min, 254 nm, t_1 = 36.4 min (minor), t_2 = 43.4 min (major)). $[\alpha]^{25}_{D}$ –44 (*c* 1.29, $CHCl_3$) for 94% ee (*S*); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51–7.41 (m, 6H), 7.41–7.28 (m, 8H), 5.48 (q, *J* = 7.6 Hz, 1H), 1.90 (d, *J* = 7.6 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ 170.4, 138.8, 136.0, 133.6, 129.9, 129.8, 129.0, 128.6, 128.5, 128.4, 49.4, 17.6; HRMS (DART-TOF) m/z: $[M + H]^+$ Calcd for $C_{24}H_{19}{}^{35}ClNO_2$ 388.1099; Found 388.1091.



3di

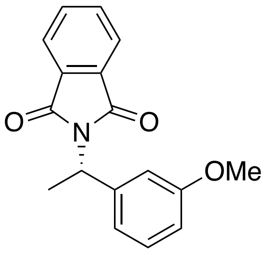
Compound 3di (Scheme 3: yellow solid, 10.1 mg, 24% yield, 93% ee (*S*)). A solution of EtOAc/hexane (1:5) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak AD–H ×2, hexane/2-propanol = 30:1, flow 0.5 mL/min, 254 nm, t_1 = 27.0 min (minor), t_2

= 30.5 min (major)). $[\alpha]^{25}_{D}$ –47 (*c* 0.37, $CHCl_3$) for 93% ee (*S*); ^1H NMR (400 MHz, $CDCl_3$) δ 7.69–7.57 (m, 4H), 7.45 (d, *J* = 6.8 Hz, 4H), 7.42–7.29 (m, 6H), 5.55 (q, *J* = 7.6 Hz, 1H), 1.94 (d, *J* = 7.6 Hz, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 170.3, 144.2, 136.0, 130.1, 129.9, 128.5, 128.4, 127.9, 125.5 (d, $J_{C–F}$ = 4 Hz), 124.0 (d, $J_{C–F}$ = 271 Hz), 49.6, 17.5; HRMS (DART-TOF) m/z: $[M + H]^+$ Calcd for $C_{25}H_{19}F_3NO_2$ 422.1366; Found 422.1362.



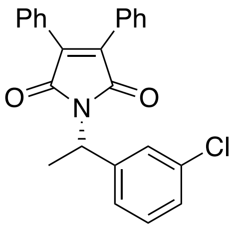
3aj

Compound 3aj (CAS: 875738-08-2 for (*R*)-**3aj**, Scheme 3: colorless oil, 21.3 mg, 80% yield, 92% ee (*S*)). A solution of EtOAc/hexane (1:10) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IB ×2, hexane/2-propanol = 100:1, flow 0.5 mL/min, 254 nm, t_1 = 32.3 min (minor), t_2 = 33.2 min (major)). $[\alpha]^{25}_{D}$ –62 (*c* 1.07, $CHCl_3$) for 92% ee (*S*); ^1H NMR (400 MHz, $CDCl_3$) δ 7.84–7.78 (m, 2H), 7.72–7.65 (m, 2H), 7.32 (d, *J* = 7.6 Hz, 1H), 7.30 (s, 1H), 7.22 (t, *J* = 7.6 Hz, 1H), 7.07 (d, *J* = 7.6 Hz, 1H), 5.54 (q, *J* = 7.2 Hz, 1H), 2.34 (s, 3H), 1.92 (d, *J* = 7.2 Hz, 3H).



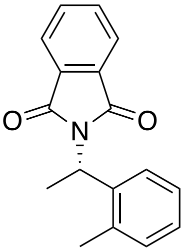
3ak

Compound 3ak (CAS: 365515-83-9 for **3ak**, Scheme 3: colorless oil, 26.1 mg, 93% yield, 89% ee (*S*)). A solution of EtOAc/hexane (1:5) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IB, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 12.8 min (minor), t_2 = 14.3 min (major)). $[\alpha]^{25}_{D}$ –52 (*c* 1.30, $CHCl_3$) for 89% ee (*S*); ^1H NMR (400 MHz, $CDCl_3$) δ 7.84–7.78 (m, 2H), 7.72–7.65 (m, 2H), 7.24 (t, *J* = 6.8 Hz, 1H), 7.12–7.03 (m, 2H), 6.80 (dd, *J* = 8.4, 2.0 Hz, 1H), 5.53 (q, *J* = 7.6 Hz, 1H), 3.79 (s, 3H), 1.91 (d, *J* = 7.6 Hz, 3H).



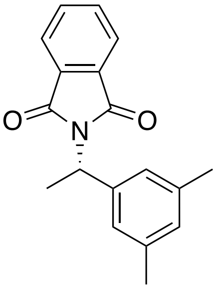
3dl

Compound 3dl (Scheme 3: yellow solid, 22.3 mg, 57% yield, 93% ee (*S*)). A solution of EtOAc/hexane (1:5) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak AD-H×2, hexane/2-propanol = 30:1, flow 0.5 mL/min, 254 nm, t_1 = 22.7 min (minor), t_2 = 29.1 min (major)). $[\alpha]^{25}_{D}$ –46 (*c* 0.90, $CHCl_3$) for 93% ee (*S*); ^1H NMR (400 MHz, $CDCl_3$) δ 7.54–7.20 (m, 14H), 5.45 (q, *J* = 7.2 Hz, 1H), 1.88 (d, *J* = 7.2 Hz, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 170.3, 142.3, 136.0, 134.4, 129.9, 129.86, 129.80, 128.5, 128.0, 127.9, 125.7, 49.5, 17.5; HRMS (DART-TOF) m/z: $[M + H]^+$ Calcd for $C_{24}H_{19}{}^{35}ClNO_2$ 388.1099; Found 388.1103.



3am

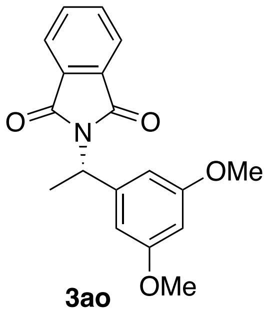
Compound 3am (CAS: 2147750-06-7 for **3am**, Scheme 3: colorless oil, 12.1 mg, 46% yield, 89% ee (*S*)). A solution of EtOAc/hexane (1:10) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IB, hexane/2-propanol = 100:1, flow 0.5 mL/min, 254 nm, t_1 = 16.5 min (minor), t_2 = 18.4 min (major)). $[\alpha]^{25}_{D}$ –92 (*c* 0.61, $CHCl_3$) for 89% ee (*S*); ^1H NMR (400 MHz, $CDCl_3$) δ 7.83–7.76 (m, 3H), 7.72–7.65 (m, 2H), 7.26 (td, *J* = 7.6, 1.2 Hz, 1H), 7.18 (td, *J* = 7.6, 1.2 Hz, 1H), 7.26 (d, *J* = 7.6 Hz, 1H), 5.71 (q, *J* = 7.6 Hz, 1H), 2.37 (s, 3H), 1.88 (d, *J* = 7.6 Hz, 3H).



3an

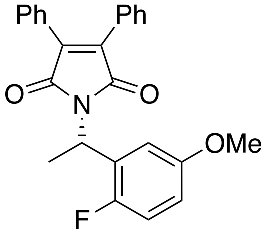
Compound 3an (Scheme 3: colorless oil, 25.8 mg, 92% yield, 89% ee (*S*)). A solution of EtOAc/hexane (1:5) was used as an eluent for preparative TLC. The ee was measured by HPLC

(Chiralpak IB, hexane/2-propanol = 100:1, flow 0.5 mL/min, 254 nm, t_1 = 14.8 min (minor), t_2 = 15.8 min (major)). $[\alpha]^{25}_D$ –65 (*c* 1.07, $CHCl_3$) for 89% ee (*S*); ^1H NMR (400 MHz, $CDCl_3$) δ 7.84–7.77 (m, 2H), 7.73–7.66 (m, 2H), 7.12 (s, 2H), 6.90 (s, 1H), 5.50 (q, *J* = 7.4 Hz, 1H), 2.30 (s, 6H), 1.91 (d, *J* = 7.4 Hz, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 168.2, 140.2, 137.9, 133.8, 132.0, 129.3, 125.2, 123.1, 49.6, 21.3, 17.6; HRMS (DART-TOF) m/z: $[M + H]^+$ Calcd for $C_{18}H_{18}NO_2$ 280.1332; Found 280.1344.



3ao

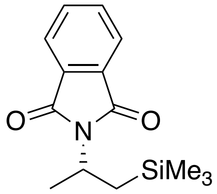
Compound 3ao (Scheme 3: colorless oil, 28.5 mg, 92% yield, 74% ee (*S*)). A solution of EtOAc/hexane (1:5) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IB, hexane/2-propanol = 9:1, flow 0.5 mL/min, 254 nm, t_1 = 14.9 min (minor), t_2 = 19.9 min (major)). $[\alpha]^{25}_D$ –38 (*c* 1.43, $CHCl_3$) for 74% ee (*S*); ^1H NMR (400 MHz, $CDCl_3$) δ 7.87–7.77 (m, 2H), 7.72–7.66 (m, 2H), 6.66 (d, *J* = 2.4 Hz, 2H), 6.36 (t, *J* = 2.4 Hz, 1H), 5.48 (q, *J* = 7.6 Hz, 1H), 3.77 (s, 6H), 1.90 (d, *J* = 7.6 Hz, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 168.1, 160.7, 142.7, 133.9, 131.9, 123.2, 105.6, 99.4, 55.3, 49.7, 17,6; HRMS (DART-TOF) m/z: $[M + H]^+$ Calcd for $C_{18}H_{18}NO_4$ 312.1230; Found 312.1223.



3dp

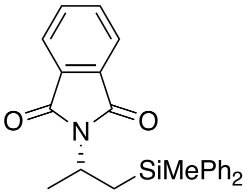
Compound 3dp (Scheme 3: colorless oil, 24.7 mg, 62% yield, 87% ee (*S*)). A solution of EtOAc/hexane (1:10) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak IB, hexane/2-propanol = 100:1, flow 0.5 mL/min, 254 nm, t_1 = 14.8 min (minor), t_2 = 15.8 min (major)). $[\alpha]^{25}_D$ –87 (*c* 1.24, $CHCl_3$) for 87% ee (*S*); ^1H NMR (400 MHz, $CDCl_3$) δ 7.48–7.43 (m, 4H), 7.40–7.30 (m, 6H), 7.23 (dd, *J* = 6.0, 3.4 Hz, 1H), 6.95 (t, *J* = 9.4 Hz, 1H), 6.77 (dt, *J* = 8.4, 3.4 Hz, 1H), 5.79 (q, *J* = 7.2 Hz, 1H), 3.80 (s, 3H), 1.87 (d, *J* = 7.2 Hz, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 170.1, 155.6 (d, $J_{C\text{–}F}$ = 240 Hz), 155.5, 135.9, 129.9, 129.8, 128.6, 128.5, 127.8 (d,

$J_{C–F}$ = 15 Hz), 115.8 (d, $J_{C–F}$ = 24 Hz), 114.5 (d, $J_{C–F}$ = 3 Hz), 113.8 (d, $J_{C–F}$ = 9 Hz), 55.7, 43.4 (d, $J_{C–F}$ = 4 Hz), 17.4; HRMS (DART-TOF) m/z: $[M + H]^+$ Calcd for $C_{25}H_{21}FNO_3$ 402.1500; Found 402.1506.



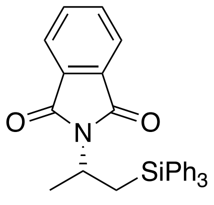
3aq

Compound 3aq (Scheme 3: colorless oil, 18.7 mg, 72% yield, 80% ee (*S*)). A solution of EtOAc/hexane (1:5) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak AD-H, hexane/2-propanol = 100:1, flow 0.5 mL/min, 254 nm, t_1 = 12.3 min (major), t_2 = 13.5 min (minor)). $[\alpha]^{25}_D$ +8 (*c* 0.88, $CHCl_3$) for 80% ee (*S*); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84–7.77 (m, 2H), 7.71–7.65 (m, 2H), 4.61–4.48 (m, 1H), 1.49 (d, *J* = 7.2 Hz, 3H), 1.47 (dd, *J* = 14.8, 9.8 Hz, 1H), 1.11 (dd, *J* = 14.8, 7.2 Hz, 1H), –0.03 (s, 9H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 168.4, 133.7, 132.1, 123.0, 44.6, 22.9, 22.1, –1.4; HRMS (DART-TOF) m/z: $[M + H]^+$ Calcd for $C_{14}H_{20}NO_2Si$ 262.1257; Found 262.1258.



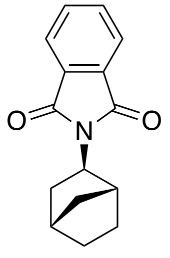
3ar

Compound 3ar (Scheme 3: colorless oil, 28.2 mg, 73% yield, 86% ee (*S*)). A solution of EtOAc/hexane (1:10) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak AD-H, hexane/2-propanol = 19:1, flow 0.5 mL/min, 254 nm, t_1 = 12.3 min (major), t_2 = 14.0 min (minor)). $[\alpha]^{25}_D$ –19 (*c* 1.15, $CHCl_3$) for 86% ee (*S*); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28–7.23 (m, 3H), 7.05–6.98 (m, 2H), 6.94 (tt, *J* = 7.6, 1.9 Hz, 1H), 4.73–4.59 (m, 1H), 2.28 (dd, *J* = 14.8, 11.2 Hz, 1H), 1.510 (d, *J* = 6.8 Hz, 1H), 1.505 (dd, *J* = 14.8, 4.4 Hz, 1H), 0.63 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 168.1, 136.8, 135.3, 134.2, 133.2, 131.8, 129.1, 128.7, 127.8, 127.5, 122.6, 43.9, 22.7, 20.3, –4.7. Anal. Calcd for $C_{24}H_{23}NO_2Si$: C, 74.77; H, 6.01; N, 3.63. Found: C, 74.75; H, 6.28; N, 3.66.

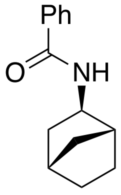


3as

Compound 3as (Scheme 3: colorless solid, 10.7 mg obtained after PTLC and GPC, 24% yield, 85% ee (*S*)). A solution of EtOAc/hexane (1:5) was used as an eluent for preparative TLC. The ee was measured by HPLC (Chiralpak AD-H, hexane/2-propanol = 19:1, flow 0.5 mL/min, 254 nm, t_1 = 12.0 min (major), t_2 = 20.3 min (minor)). $[\alpha]^{25}_{D}$ +57 (*c* 0.30, $CHCl_3$) for 85% ee (*S*); ^1H NMR (400 MHz, $CDCl_3$) δ 7.60–7.47 (m, 10H), 7.25–7.17 (m, 9H), 4.91–4.78 (m, 1H), 2.56 (dd, *J* = 15.0, 11.0 Hz, 1H), 1.87 (dd, *J* = 15.0, 4.2 Hz, 1H), 1.51 (d, *J* = 7.2 Hz, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 128.1, 135.5, 134.1, 133.2, 131.9, 129.2, 127.8, 122.6, 43.8, 23.1, 19.0; HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{29}H_{25}NNaO_2Si$ 470.1547; Found 470.1549.



3at



4

Compound 3at (Scheme 3: colorless oil, 21.4 mg, 96% yield, 95% ee (1*R*,2*R*,4*S*)). A solution of EtOAc/hexane (1:5) was used as an eluent for preparative TLC. A 228 mg of **3at** was isolated by column chromatography on silica gel in the 1.0 mmol scale (94% yield, 96% ee (1*R*,2*R*,4*S*)). The ee was measured by HPLC (Chiralpak AD-H, hexane/2-propanol = 100:1, flow 1.0 mL/min, 254 nm, t_1 = 20.7 min (major), t_2 = 22.1 min (minor)). $[\alpha]^{25}_{D}$ –8 (*c* 1.03, $CHCl_3$) for 95% ee (*S*); ^1H NMR (400 MHz, $CDCl_3$) δ 7.82–7.75 (m, 2H), 7.71–7.64 (m, 2H), 4.13 (dd, *J* = 8.0, 5.6 Hz, 1H), 2.41 (d, *J* = 13.2 Hz, 2H), 2.31–2.19 (m, 2H), 1.76–1.66 (m, 1H), 1.62–1.49 (m, 2H), 1.40–1.30 (m, 1H), 1.30–1.16 (m, 2H).

Compound 4.[11] To a solution of **3at** (24.1 mg, 0.10 mmol) in THF (0.4 mL) and ethanol (0.4 mL) in a Schlenk tube was added hydrazine hydrate (12.4 μL, 0.50 mmol), and the mixture was heated in an oil bath at 80 °C for 3 h. After cooling to room temperature, the precipitate was removed by filtration and washed with CH_2Cl_2. The filtrate was washed with 1 M NaOHaq., dried over Na_2SO_4, filtered, and concentrated on a rotary evaporator. To the residue in CH_2Cl_2 (1 mL) were added triethylamine (42 μL, 0.20 mmol) and benzoyl chloride (23 μL, 0.30 mmol), and the mixture was stirred at room temperature overnight. A small amount of water was added to the mixture, and the resulting solution was passed through a short column of silica gel eluted with ethyl acetate. The solution was concentrated on a rotary evaporator and the residue was subjected to preparative TLC on silica gel with hexane/ethyl acetate (3:1) to give **4** (17.6 mg, 82% yield) as a colorless solid. The absolute configuration of **4** was determined to be (1*R*,2*R*,4*S*) by comparison of the specific rotation with the reported value ($[\alpha]^{23}_{D}$ –15.2 (*c* 2.0, $CHCl_3$) for 91% ee (1*R*,2*R*,4*S*)).[12] $[\alpha]^{25}_{D}$ –13 (*c* 0.78, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.75–7.69 (m, 2H), 7.50–7.36 (m, 3H), 5.97 (br s, 1H), 3.90 (td, *J* = 7.6, 3.6 Hz, 1H), 2.31 (d, *J* = 4.4 Hz, 2H), 2.82 (ddd, *J* = 13.2, 8.0, 2.0 Hz, 1H), 1.58–1.43 (m, 2H), 1.40–1.10 (m, 5H).

9. References

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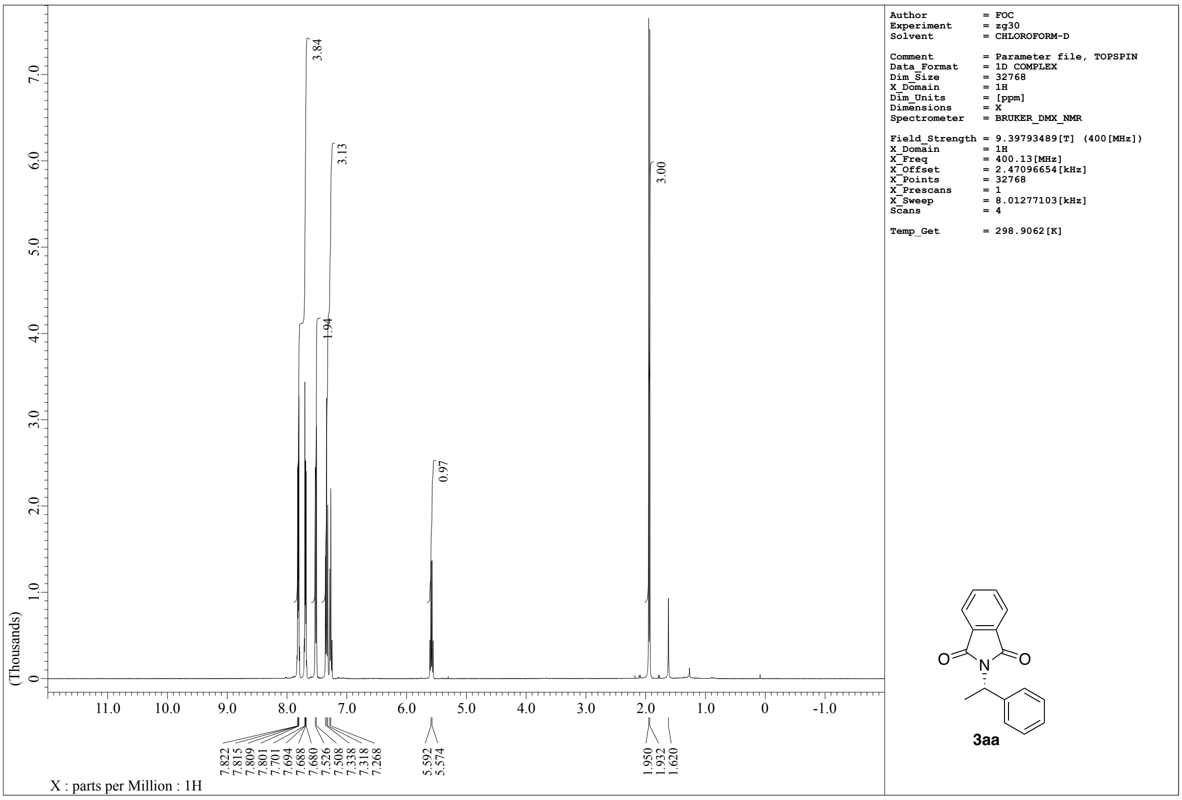
10. NMR and HPLC charts



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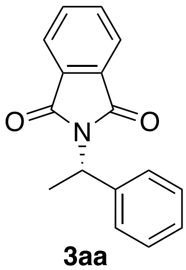

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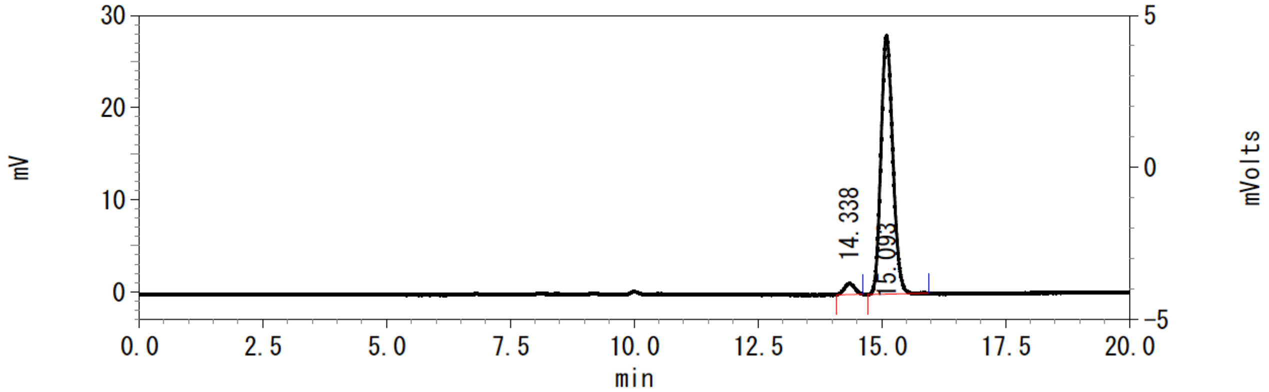

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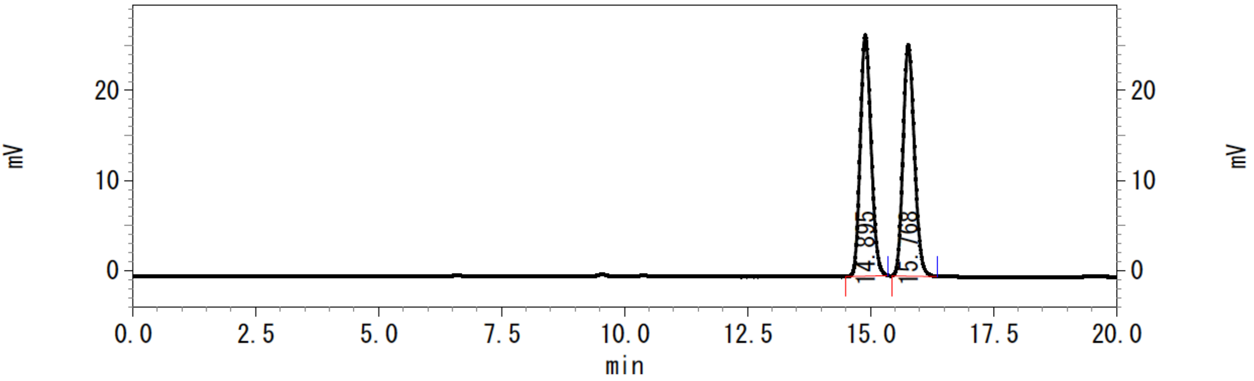

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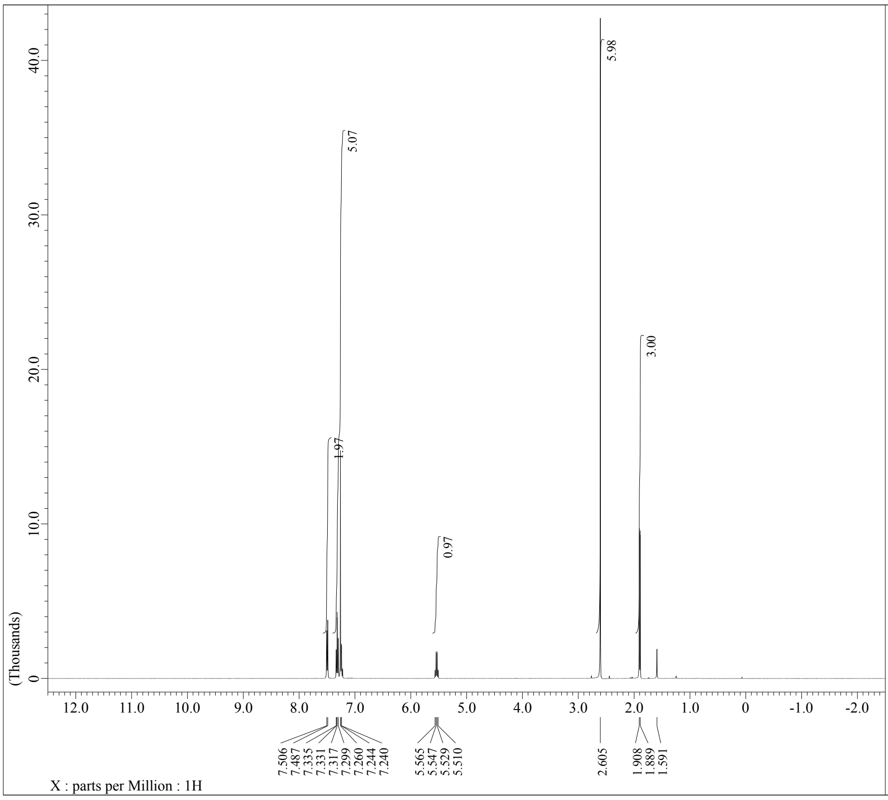

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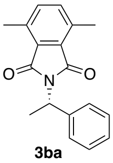

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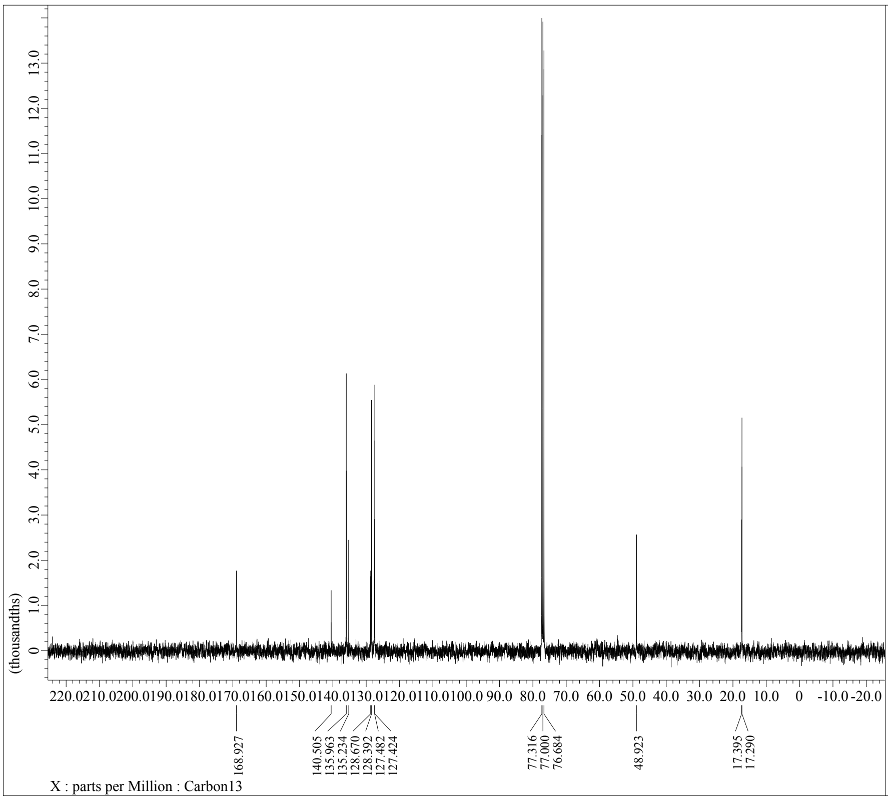

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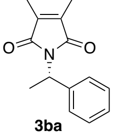

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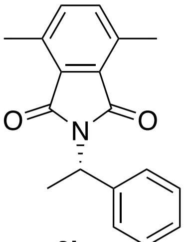

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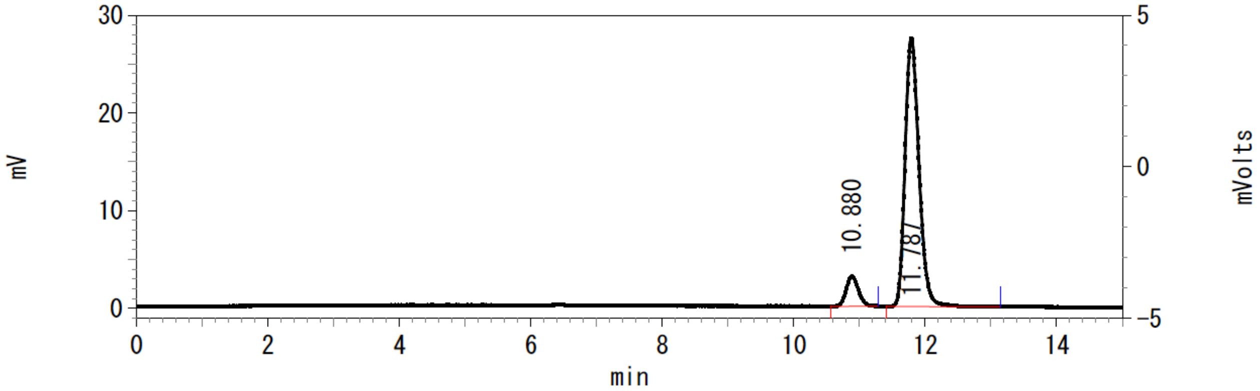

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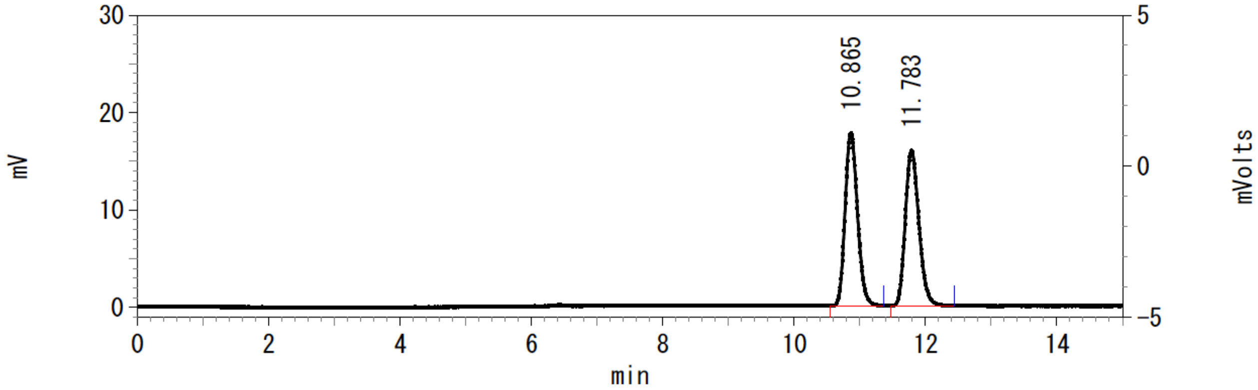

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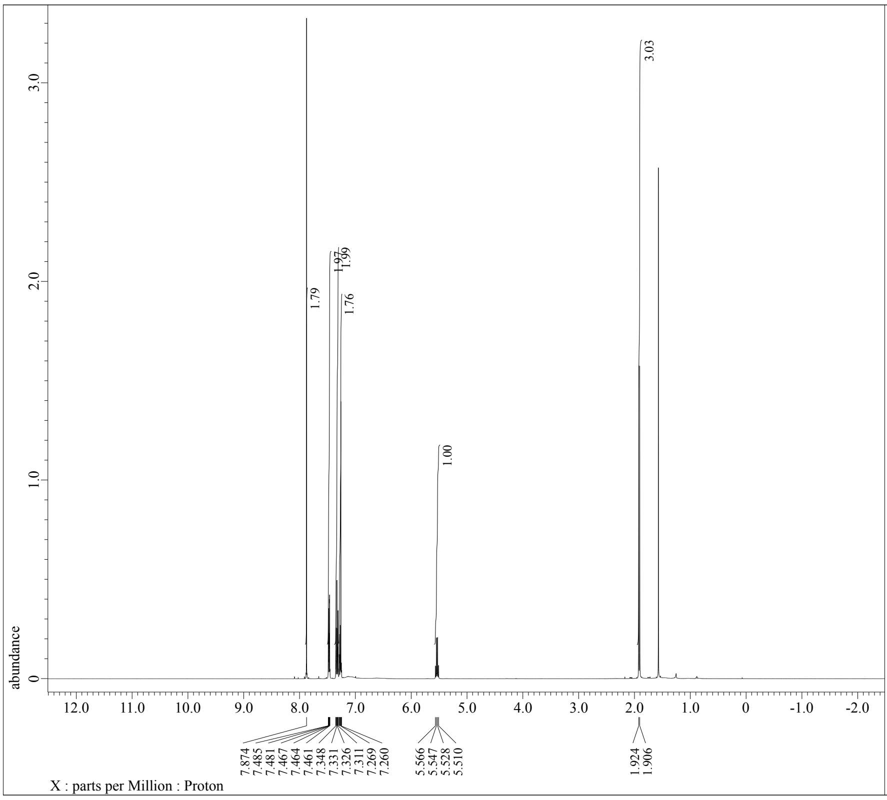

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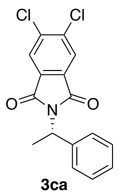

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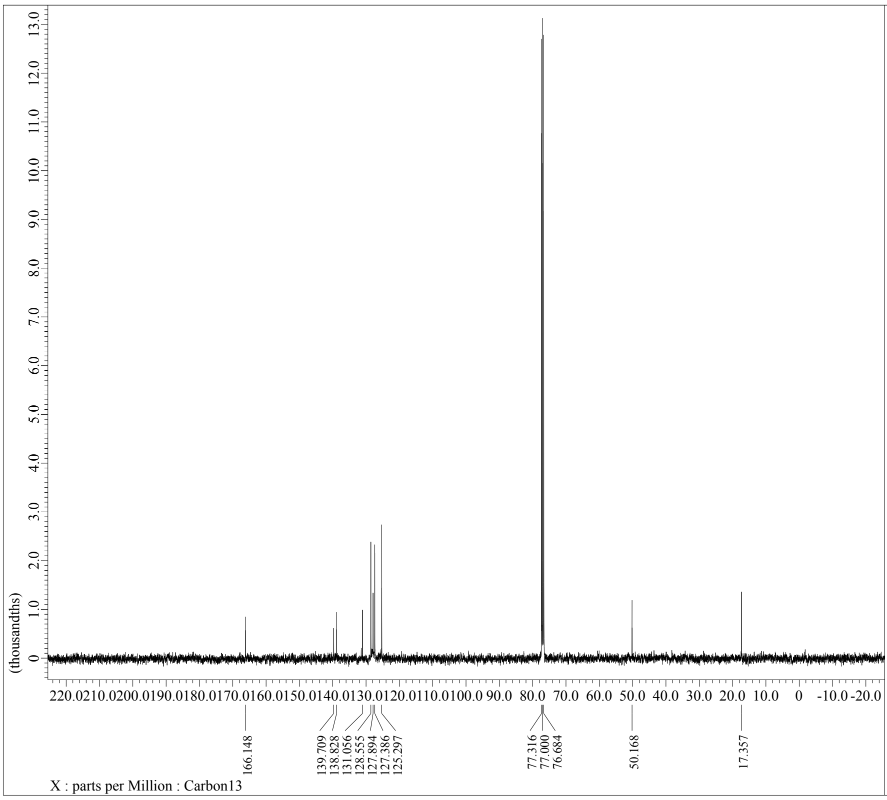

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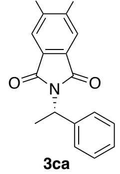

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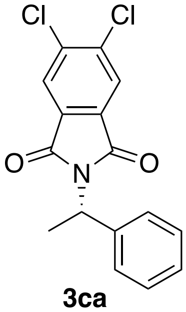

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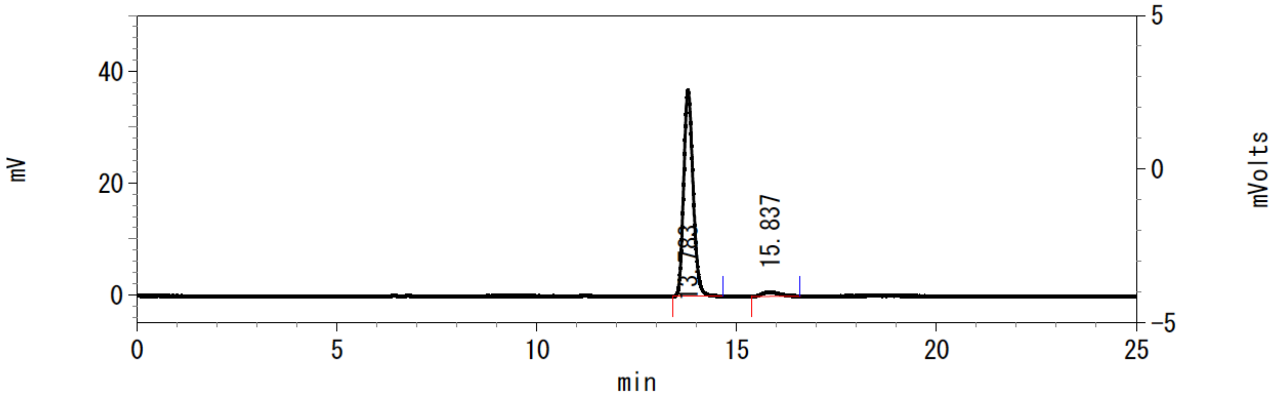

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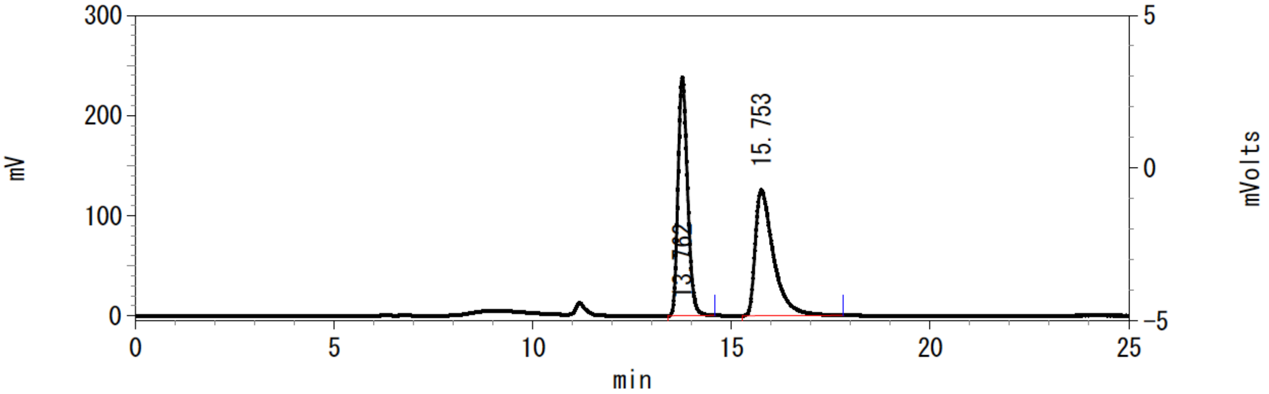

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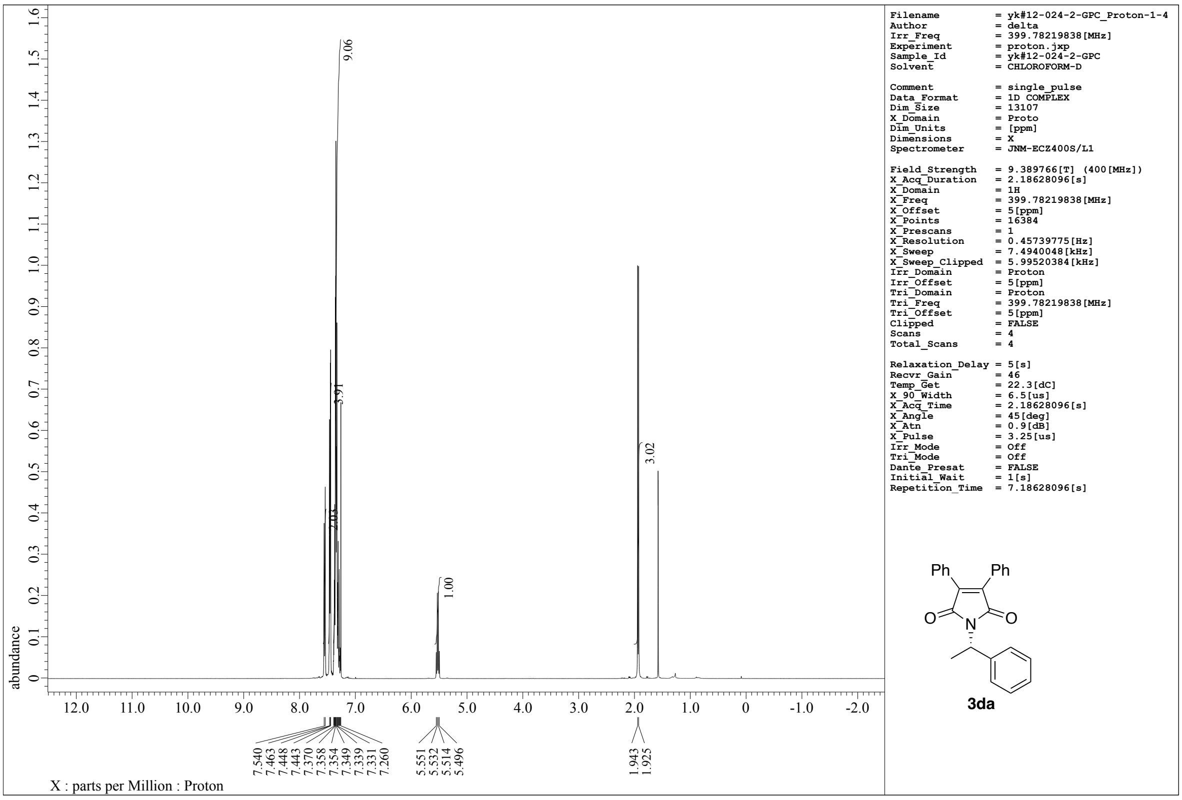

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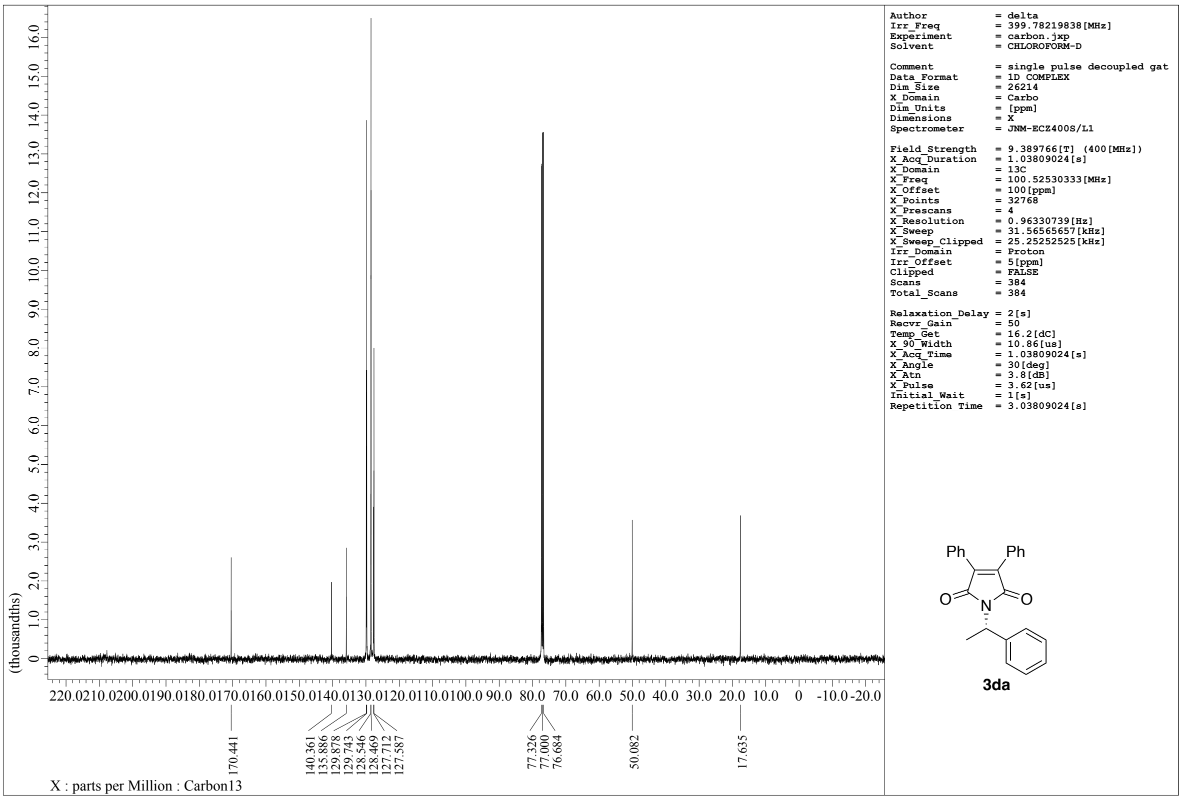

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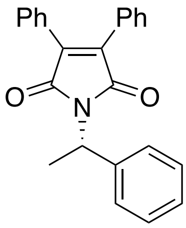

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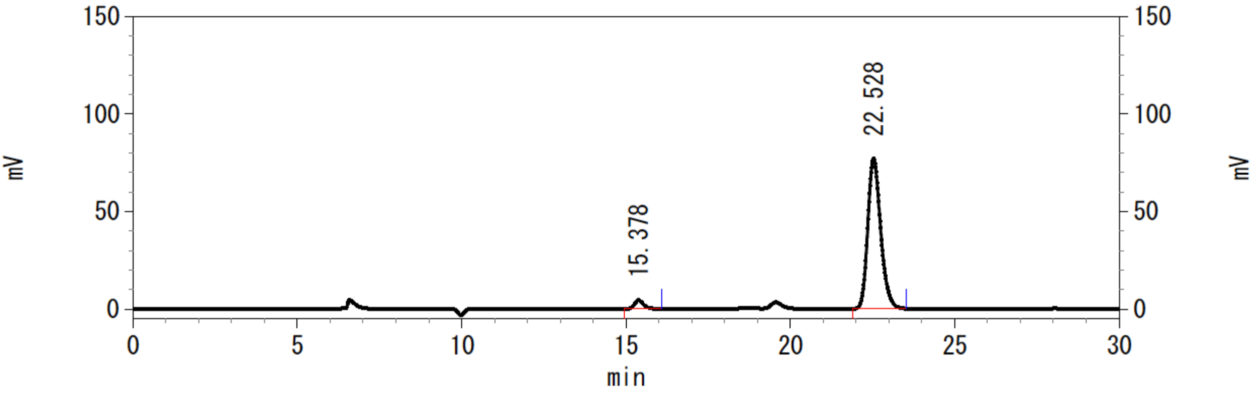

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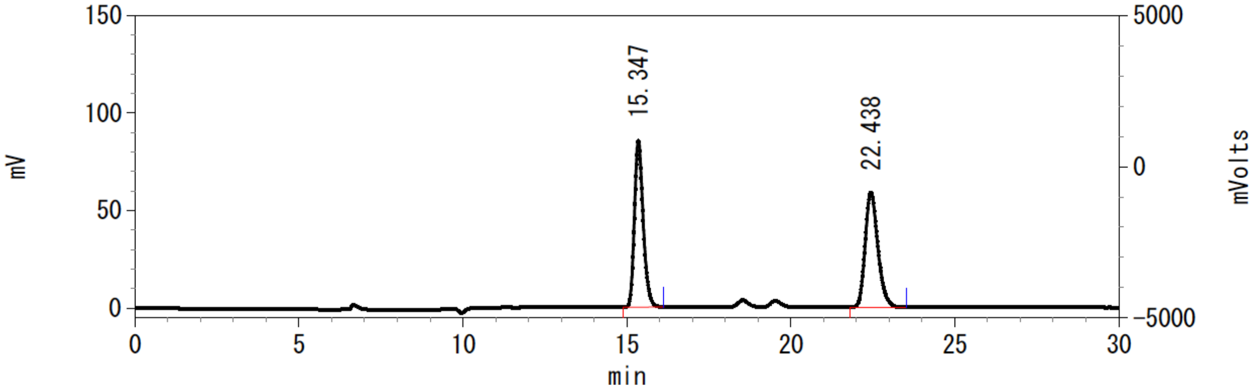

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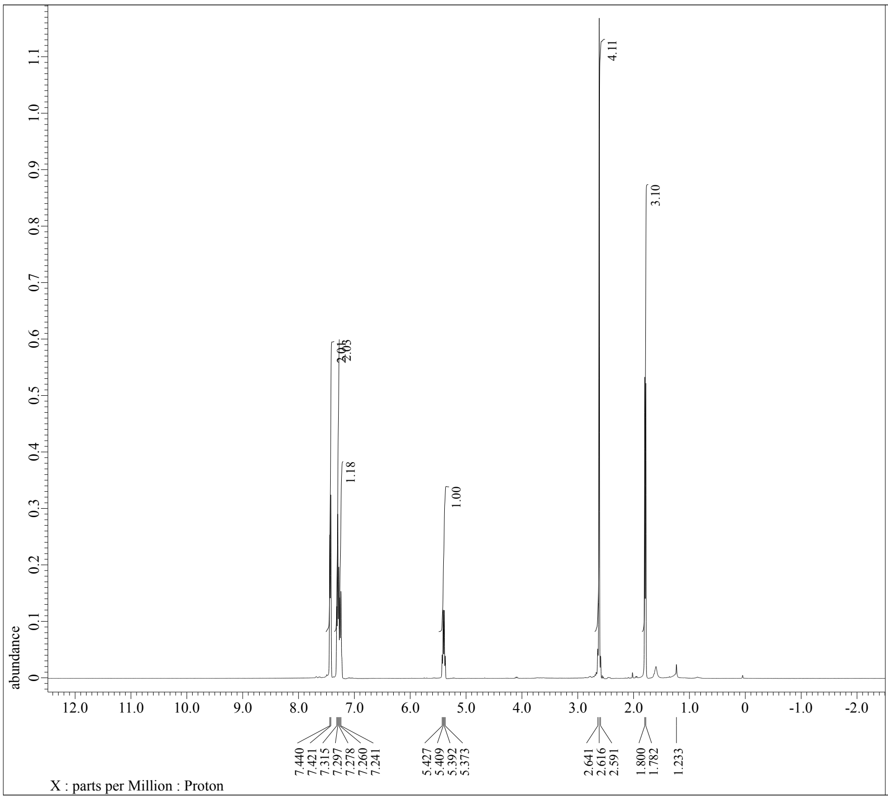

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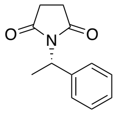

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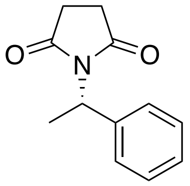

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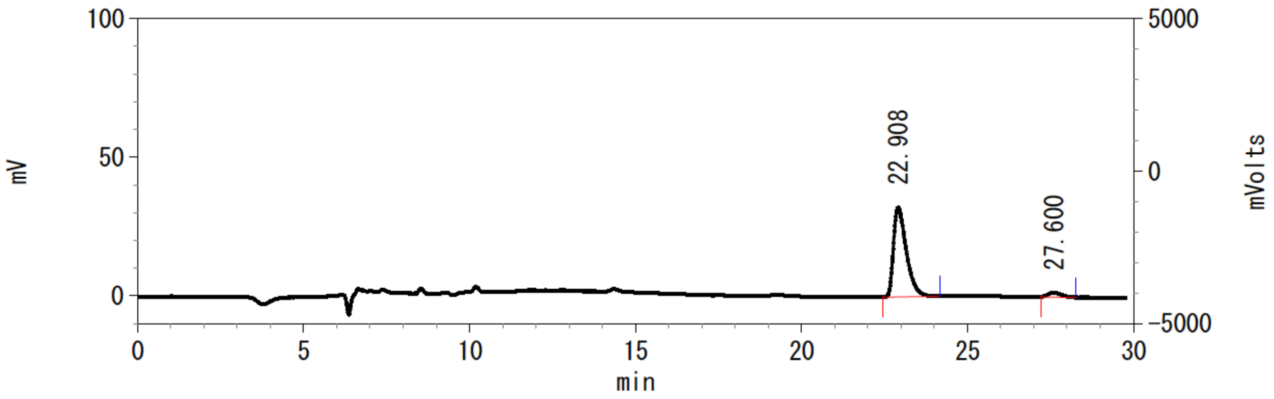
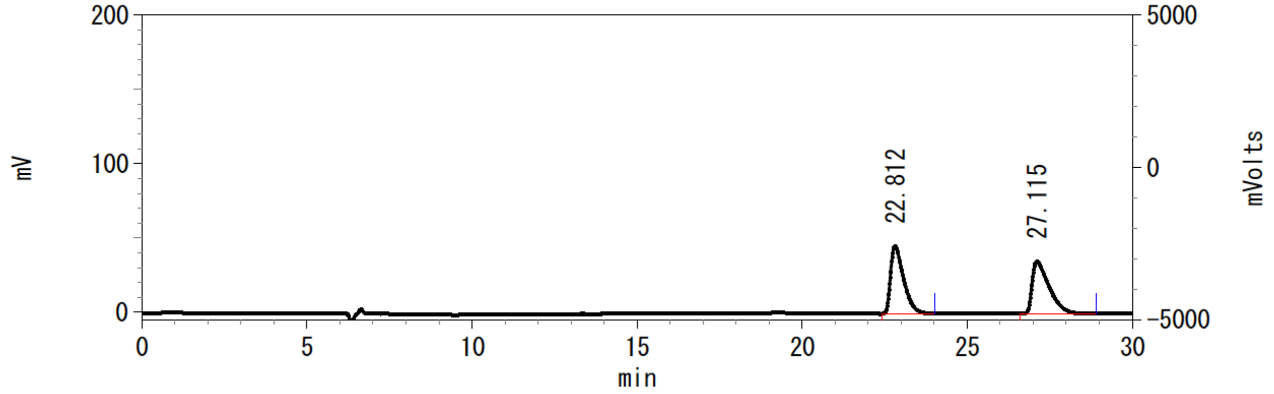
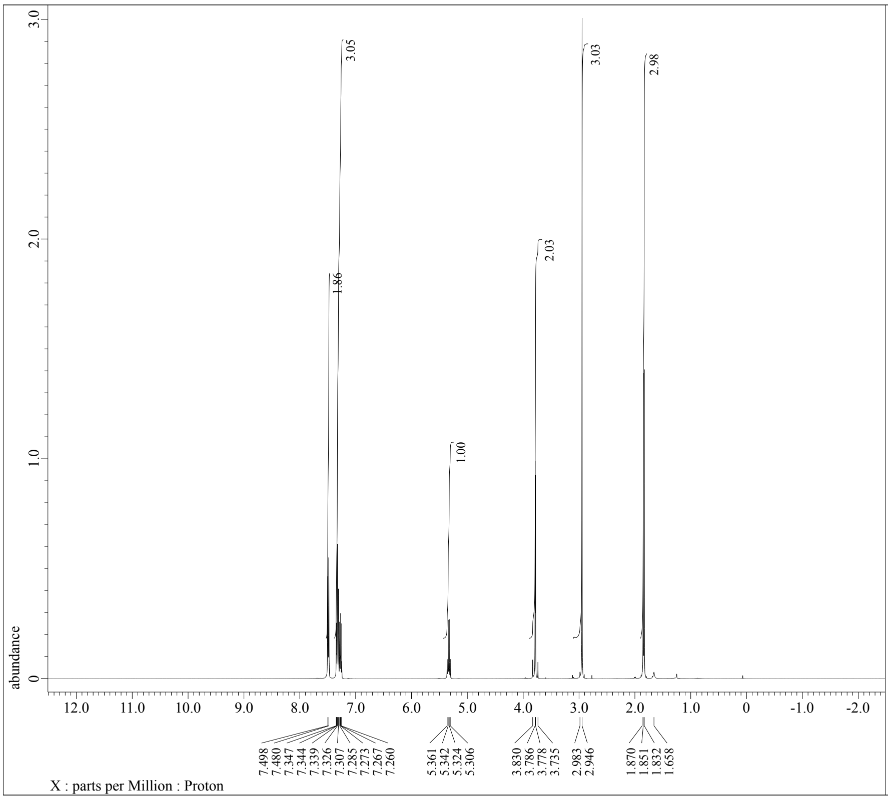


Pk #	Retention Time	Area	Area Percent
1	22.908	868205	94.879
2	27.600	46861	5.121



Pk #	Retention Time	Area	Area Percent
1	22.812	1253831	50.064
2	27.115	1250649	49.936



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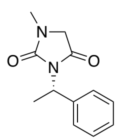
Author           = delta
Irr_Freq         = 399.78219838[MHz]
Experiment       = proton.jxp
Solvent          = CHLOROFORM-D

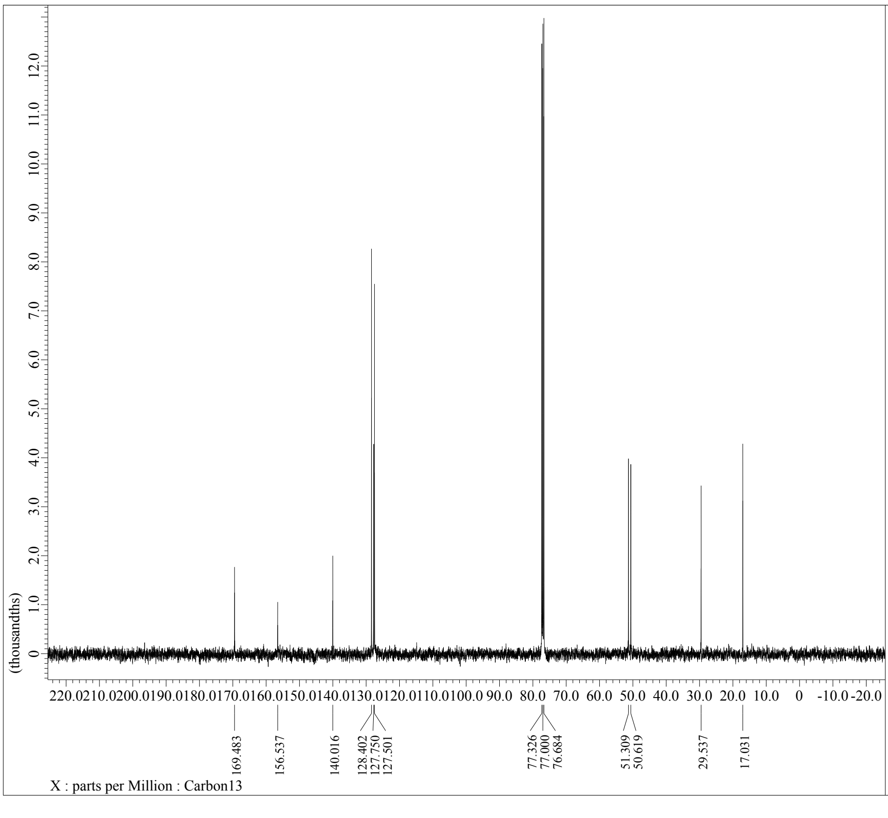
Comment          = single_pulse
Data_Format      = 1D COMPLEX
Dim_Size         = 13107
X_Domain         = Proto
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = JNM-ECZ400S/L1

Field_Strength   = 9.389766[T] (400[MHz])
X_Acq_Duration   = 2.18628096[s]
X_Domain         = 1H
X_Freq           = 399.78219838[MHz]
X_Offset         = 5[ppm]
X_Points         = 16384
X_Prescans       = 1
X_Resolution     = 0.45739775[Hz]
X_Sweep          = 7.4940048[kHz]
X_Sweep_Clipped  = 5.99520384[kHz]
Irr_Domain       = Proton
Irr_Offset       = 5[ppm]
Tri_Domain       = Proton
Tri_Freq         = 399.78219838[MHz]
Tri_Offset       = 5[ppm]
Clipped          = FALSE
Scans            = 8
Total_Scans      = 8

Relaxation_Delay = 5[s]
Recvr_Gain       = 46
Temp_Get         = 18.3[dC]
X_90_Width       = 6.3[us]
X_Acq_Time       = 2.18628096[s]
X_Angle          = 45[deg]
X_Atn            = 0.9[dB]
X_Pulse          = 3.15[us]
Irr_Mode         = Off
Tri_Mode         = Off
Dante_Presat     = FALSE
Initial_Wait     = 1[s]
Repetition_Time  = 7.18628096[s]
```



3fa

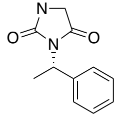


```
Author           = delta
Irr_Freq         = 399.78219838[MHz]
Experiment       = carbon.jxp
Solvent          = CHLOROFORM-D

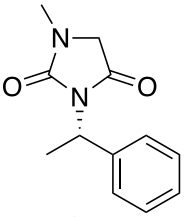
Comment          = single pulse decoupled gat
Data_Format      = 1D COMPLEX
Dim_Size         = 26214
X_Domain         = Carbo
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = JNM-ECZ400S/L1

Field_Strength   = 9.389766[T] (400[MHz])
X_Acq_Duration   = 1.03809024[s]
X_Domain         = 13C
X_Freq           = 100.52530333[MHz]
X_Offset         = 100[ppm]
X_Points         = 32768
X_Prescans       = 4
X_Resolution     = 0.96330739[Hz]
X_Sweep          = 31.56565657[kHz]
X_Sweep_Clipped  = 25.25252525[kHz]
Irr_Domain       = Proton
Irr_Offset       = 5[ppm]
Clipped          = FALSE
Scans            = 203
Total_Scans      = 203

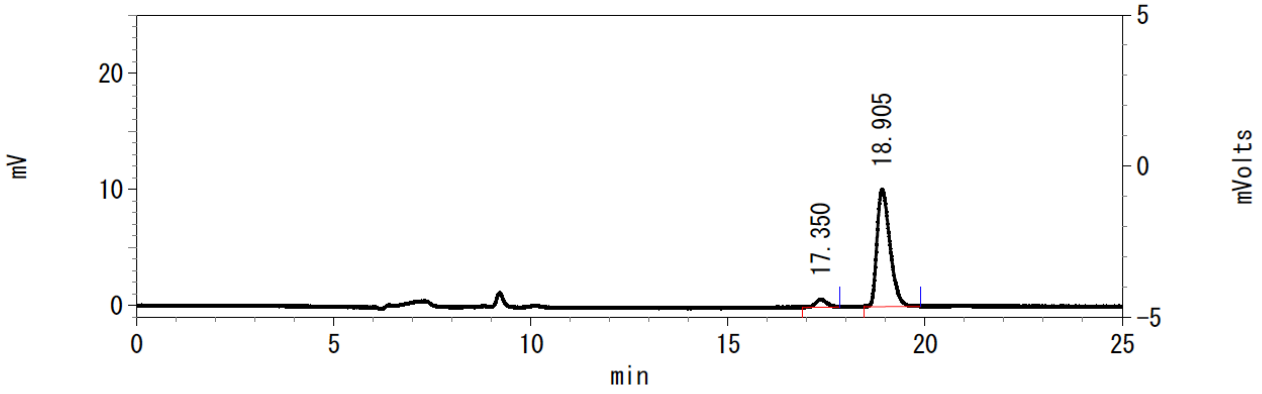
Relaxation_Delay = 2[s]
Recvr_Gain       = 50
Temp_Get         = 18.2[dC]
X_90_Width       = 10.86[us]
X_Acq_Time       = 1.03809024[s]
X_Angle          = 30[deg]
X_Atn            = 3.8[dB]
X_Pulse          = 3.62[us]
Initial_Wait     = 1[s]
Repetition_Time  = 3.03809024[s]
```



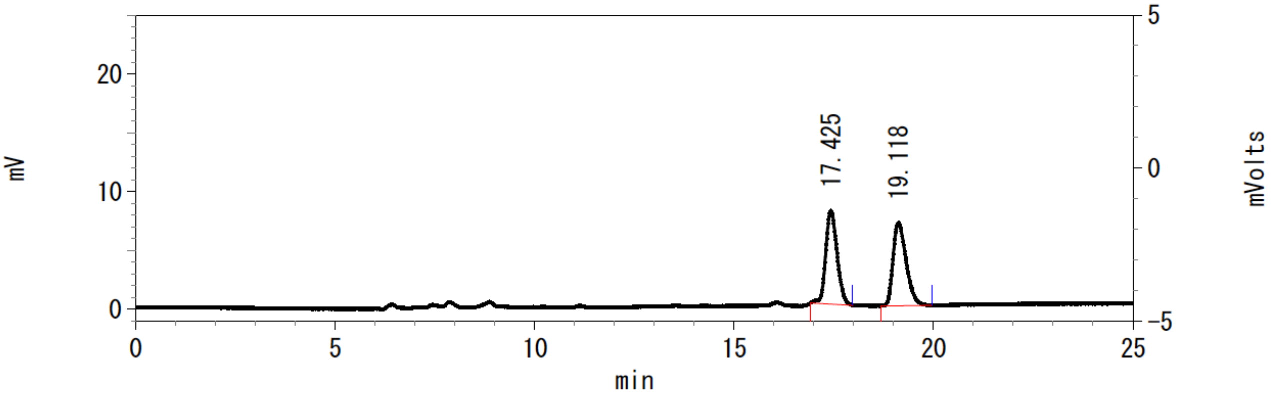
3fa



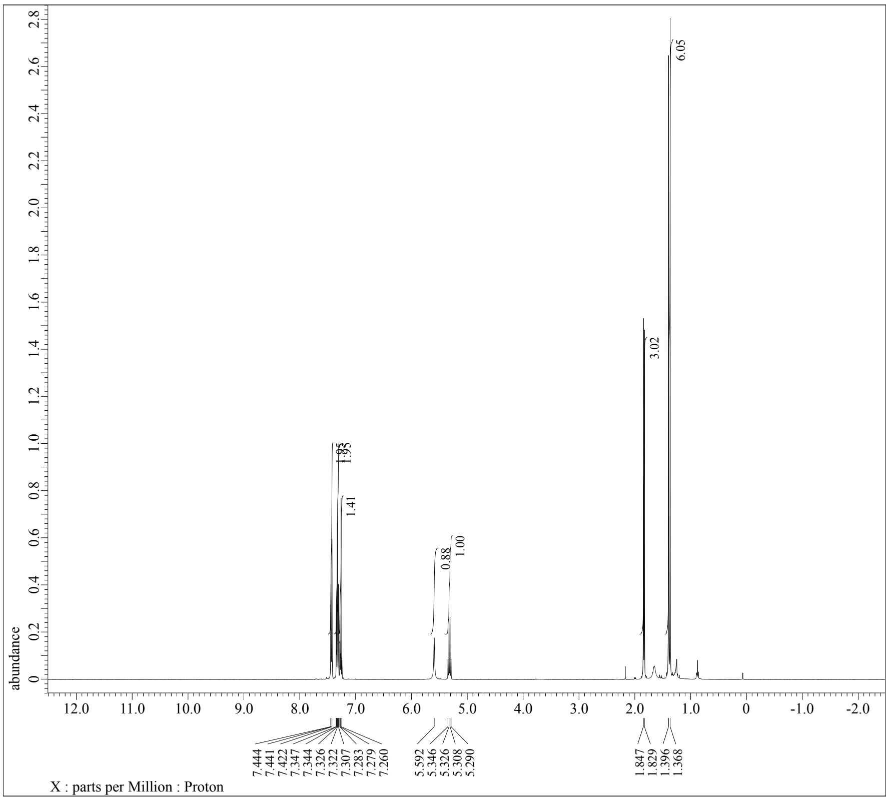
3fa



Pk #	Retention Time	Area	Area Percent
1	17.350	13463	5.535
2	18.905	229781	94.465



Pk #	Retention Time	Area	Area Percent
1	17.425	158152	49.537
2	19.118	161110	50.463

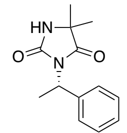


```
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Solvent           = CHLOROFORM-D

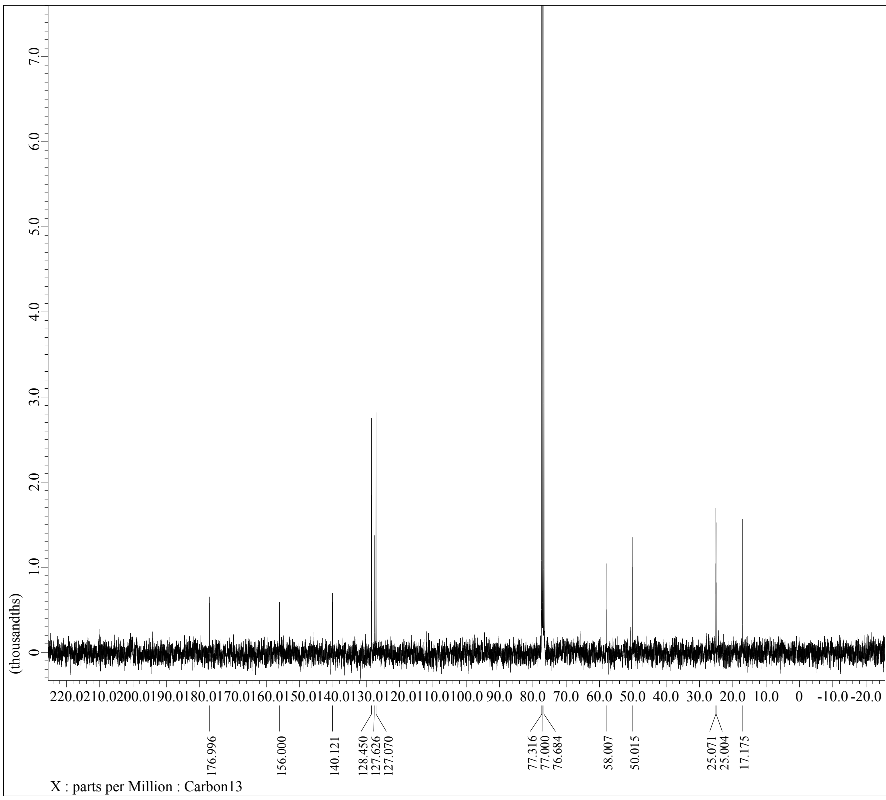
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proto
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18628096[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45739775[Hz]
X_Sweep           = 7.4940048[kHz]
X_Sweep_Clipped   = 5.99520384[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 4
Total_Scans       = 4

Relaxation_Delay = 5[s]
Recvr_Gain        = 56
Temp_Get          = 18.8[dC]
X_90_Width        = 6.3[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 3.15[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18628096[s]
```



3ga

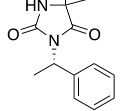


```
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Solvent           = CHLOROFORM-D

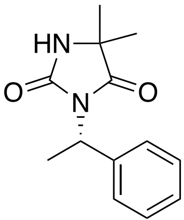
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
X_Domain          = Carbo
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.03809024[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.96330739[Hz]
X_Sweep           = 31.56565657[kHz]
X_Sweep_Clipped   = 25.25252525[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 156
Total_Scans       = 156

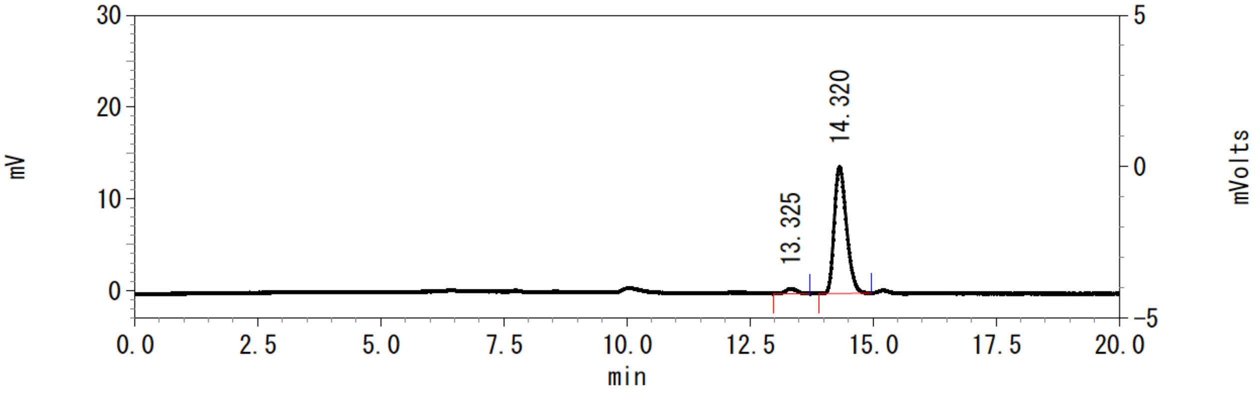
Relaxation_Delay = 2[s]
Recvr_Gain        = 50
Temp_Get          = 18.8[dC]
X_90_Width        = 10.86[us]
X_Acq_Time        = 1.03809024[s]
X_Angle           = 30[deg]
X_Atn             = 3.8[dB]
X_Pulse           = 3.62[us]
Initial_Wait      = 1[s]
Repetition_Time   = 3.03809024[s]
```



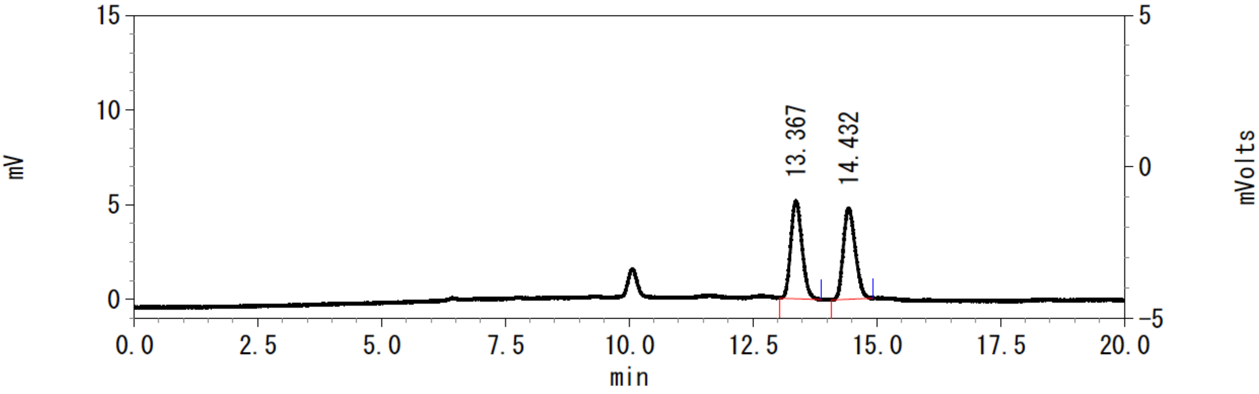
3ga



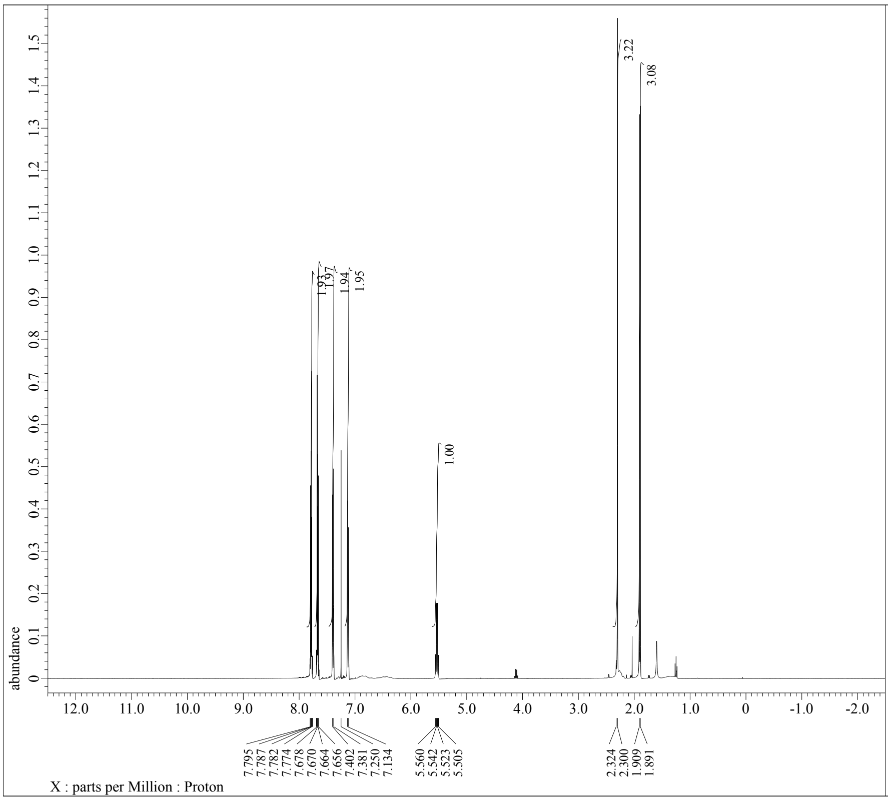
3ga



Pk #	Retention Time	Area	Area Percent
1	13.325	8901	3.584
2	14.320	239470	96.416



Pk #	Retention Time	Area	Area Percent
1	13.367	81656	50.167
2	14.432	81113	49.833

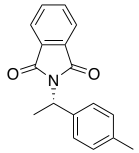


```
Author           = delta
Irr_Freq         = 399.78219838[MHz]
Experiment       = proton.jxp
Solvent          = CHLOROFORM-D

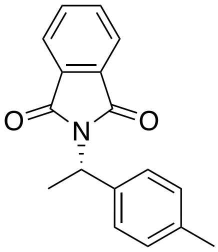
Comment          = single_pulse
Data_Format      = 1D COMPLEX
Dim_Size         = 13107
X_Domain         = Proto
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = JNM-ECZ400S/L1

Field_Strength   = 9.389766[T] (400[MHz])
X_Acq_Duration   = 2.18628096[s]
X_Domain         = 1H
X_Freq           = 399.78219838[MHz]
X_Offset         = 5[ppm]
X_Points         = 16384
X_Prescans       = 1
X_Resolution     = 0.45739775[Hz]
X_Sweep          = 7.4940048[kHz]
X_Sweep_Clipped  = 5.99520384[kHz]
Irr_Domain       = Proton
Irr_Offset       = 5[ppm]
Tri_Domain       = Proton
Tri_Freq         = 399.78219838[MHz]
Tri_Offset       = 5[ppm]
Clipped          = FALSE
Scans            = 4
Total_Scans      = 4

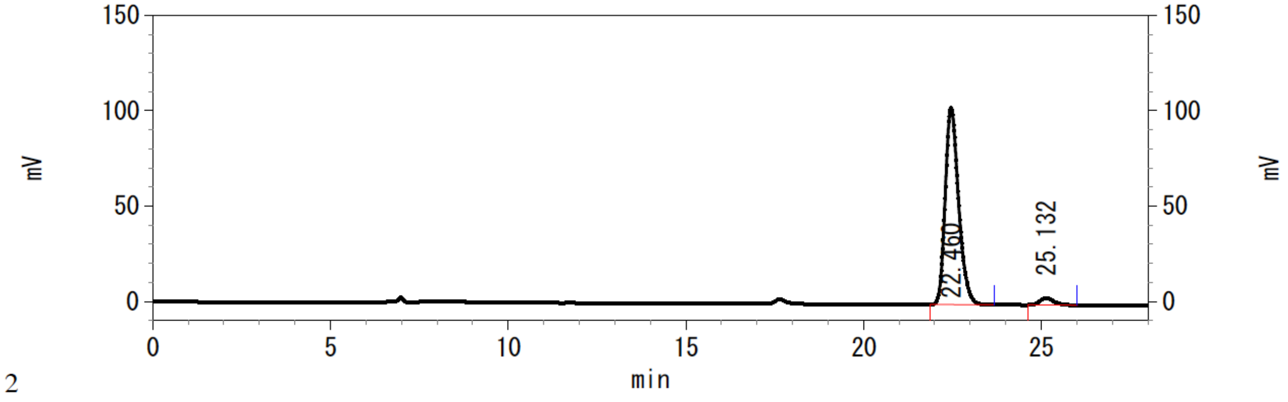
Relaxation_Delay = 5[s]
Recvr_Gain       = 46
Temp_Get         = 21.4[dC]
X_90_Width       = 6.5[us]
X_Acq_Time       = 2.18628096[s]
X_Angle          = 45[deg]
X_Atn            = 0.9[dB]
X_Pulse          = 3.25[us]
Irr_Mode         = Off
Tri_Mode         = Off
Dante_Presat     = FALSE
Initial_Wait     = 1[s]
Repetition_Time  = 7.18628096[s]
```



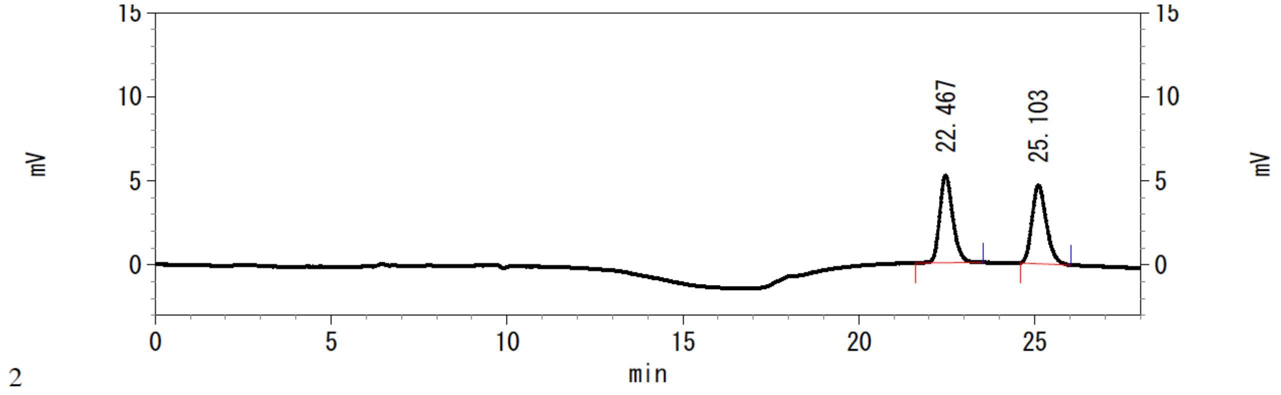
3ab



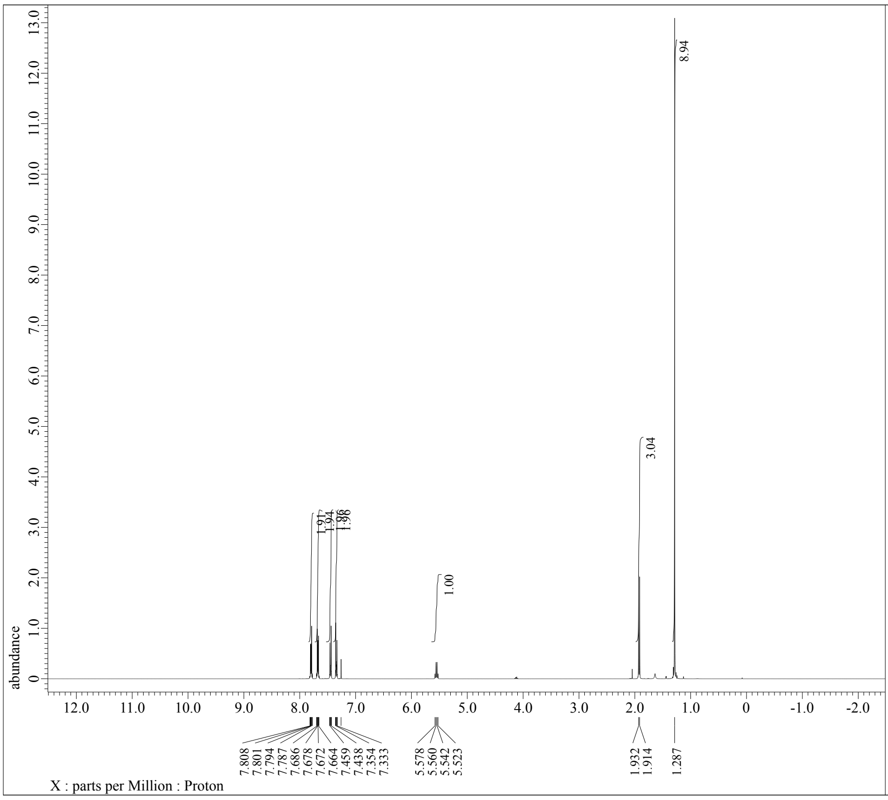
3ab



Pk #	Retention Time	Area	Area Percent
1	22.460	2693412	96.355
2	25.132	101891	3.645



Pk #	Retention Time	Area	Area Percent
1	22.467	134909	50.506
2	25.103	132207	49.494

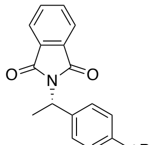


```
Author           = delta
Irr_Freq         = 399.78219838[MHz]
Experiment       = proton.jxp
Solvent          = CHLOROFORM-D

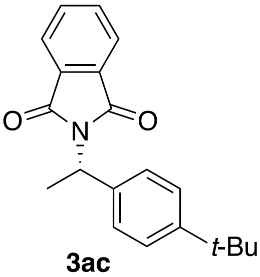
Comment          = single_pulse
Data_Format      = 1D COMPLEX
Dim_Size         = 13107
X_Domain         = Proto
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = JNM-ECZ400S/L1

Field_Strength   = 9.389766[T] (400[MHz])
X_Acq_Duration   = 2.18628096[s]
X_Domain         = 1H
X_Freq           = 399.78219838[MHz]
X_Offset         = 5[ppm]
X_Points         = 16384
X_Prescans       = 1
X_Resolution     = 0.45739775[Hz]
X_Sweep          = 7.4940048[kHz]
X_Sweep_Clipped  = 5.99520384[kHz]
Irr_Domain       = Proton
Irr_Offset       = 5[ppm]
Tri_Domain       = Proton
Tri_Freq         = 399.78219838[MHz]
Tri_Offset       = 5[ppm]
Clipped          = FALSE
Scans            = 4
Total_Scans      = 4

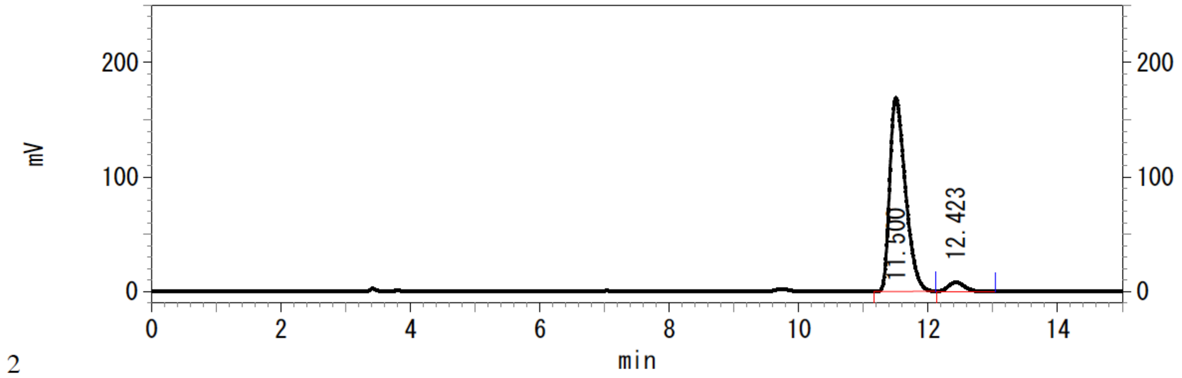
Relaxation_Delay = 5[s]
Recvr_Gain       = 46
Temp_Get         = 21.5[dC]
X_90_Width       = 6.5[us]
X_Acq_Time       = 2.18628096[s]
X_Angle          = 45[deg]
X_Atn            = 0.9[dB]
X_Pulse          = 3.25[us]
Irr_Mode         = Off
Tri_Mode         = Off
Dante_Presat     = FALSE
Initial_Wait     = 1[s]
Repetition_Time  = 7.18628096[s]
```



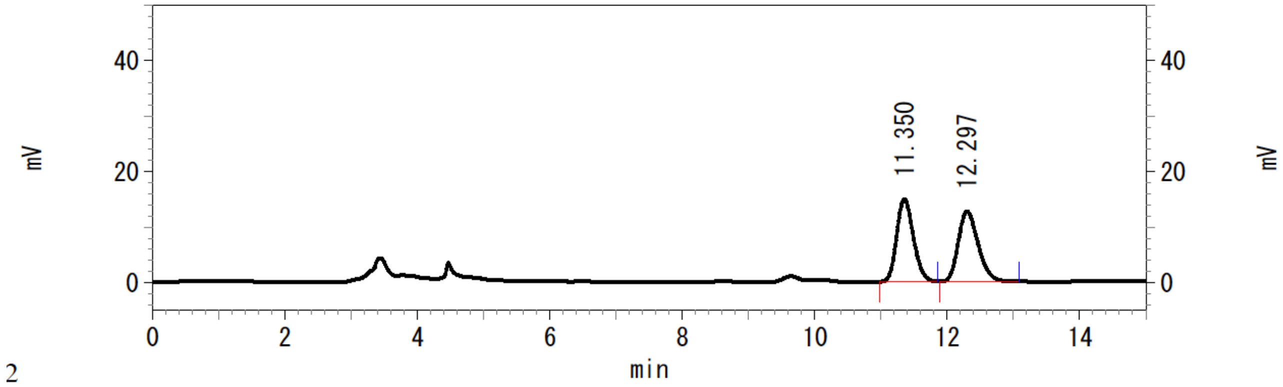
3ac



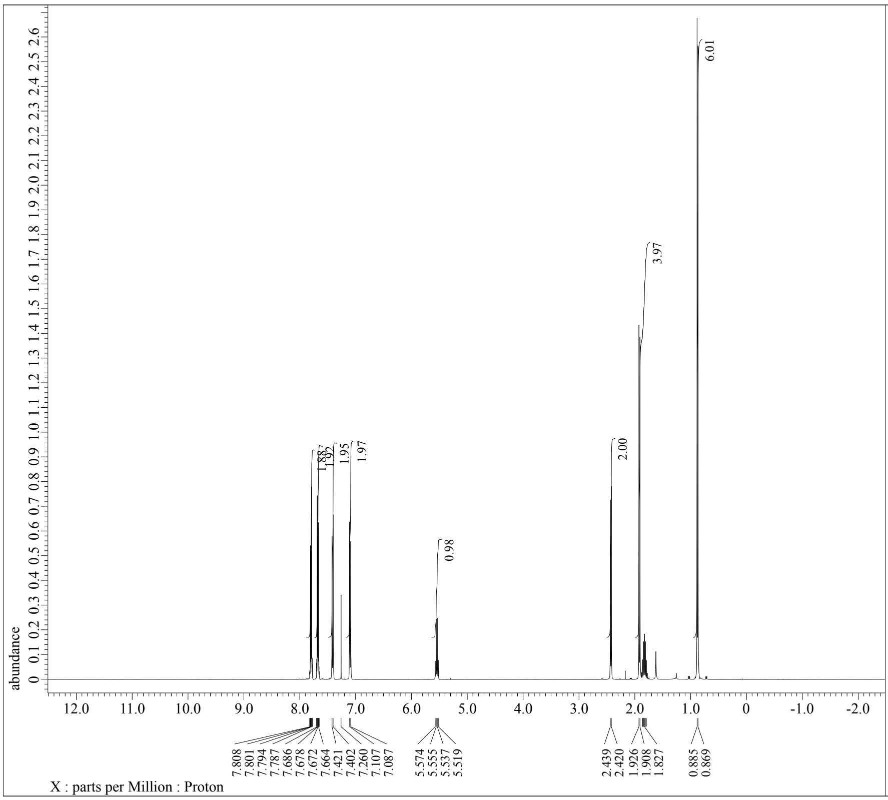
3ac



Pk #	Retention Time	Area	Area Percent
1	11.500	2852384	95.019
2	12.423	149538	4.981



Pk #	Retention Time	Area	Area Percent
1	11.350	261109	49.761
2	12.297	263616	50.239

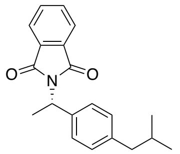


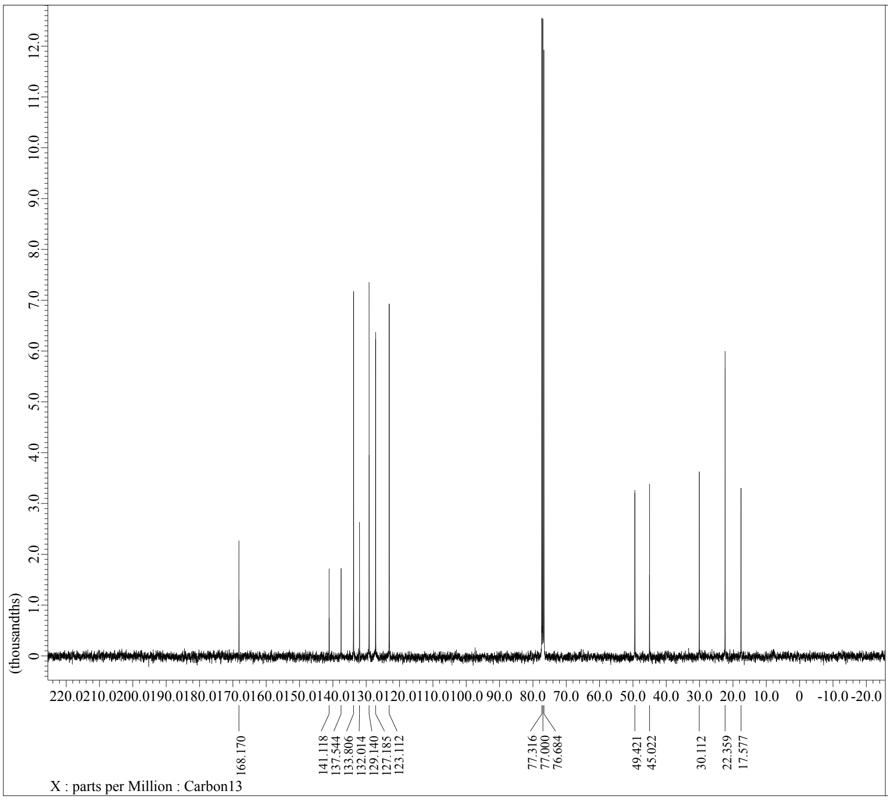
```
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Solvent           = CHLOROFORM-D

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proto
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18628096[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45739775[Hz]
X_Sweep           = 7.4940048[kHz]
X_Sweep_Clipped   = 5.99520384[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 4
Total_Scans       = 4

Relaxation_Delay  = 5[s]
Recvr_Gain        = 46
Temp_Get          = 22.3[dC]
X_90_Width        = 6.5[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 3.25[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18628096[s]
```


3ad

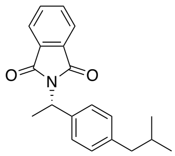


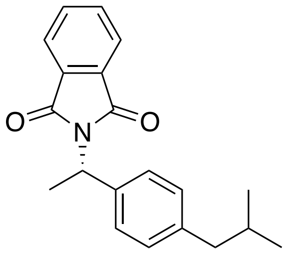
```
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Solvent           = CHLOROFORM-D

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
X_Domain          = Carbo
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

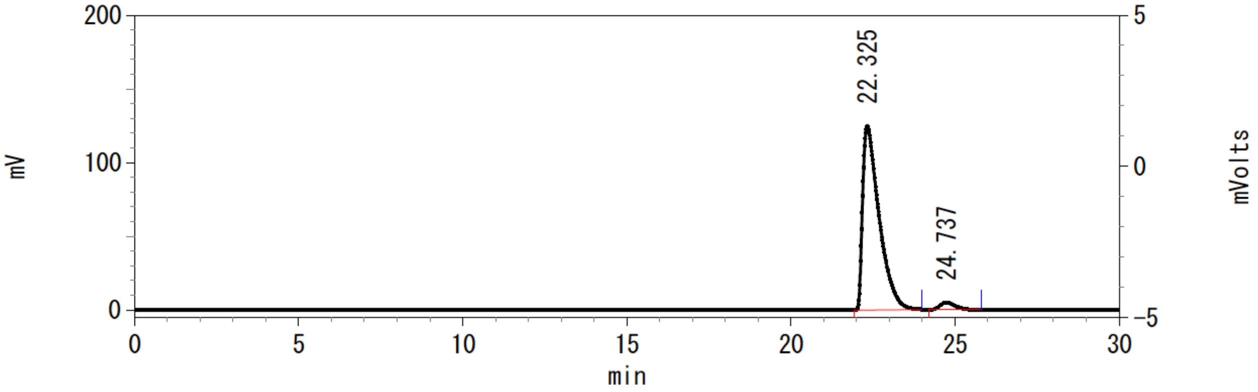
Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.03809024[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.96330739[Hz]
X_Sweep           = 31.56565657[kHz]
X_Sweep_Clipped   = 25.25252525[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 355
Total_Scans       = 355

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 22.8[dC]
X_90_Width        = 10.5[us]
X_Acq_Time        = 1.03809024[s]
X_Angle           = 30[deg]
X_Atn             = 3.8[dB]
X_Pulse           = 3.5[us]
Initial_Wait      = 1[s]
Repetition_Time   = 3.03809024[s]
```

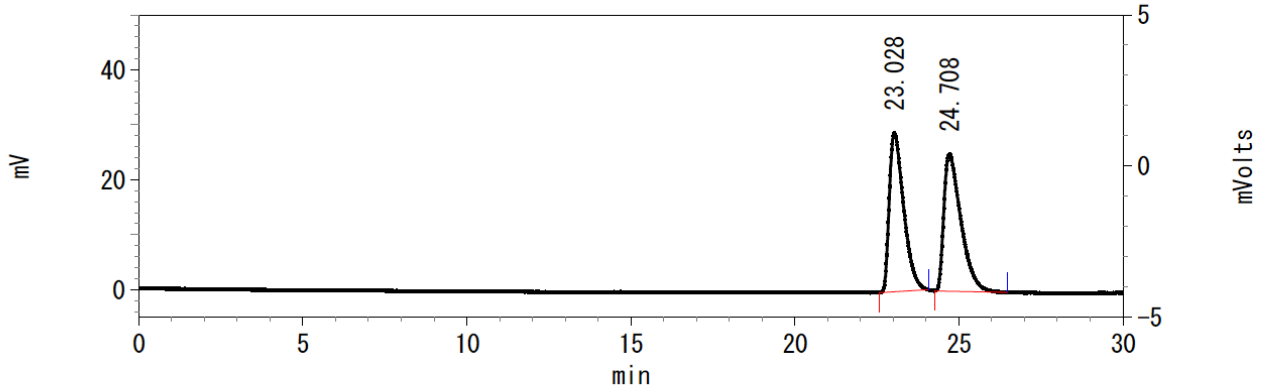

3ad



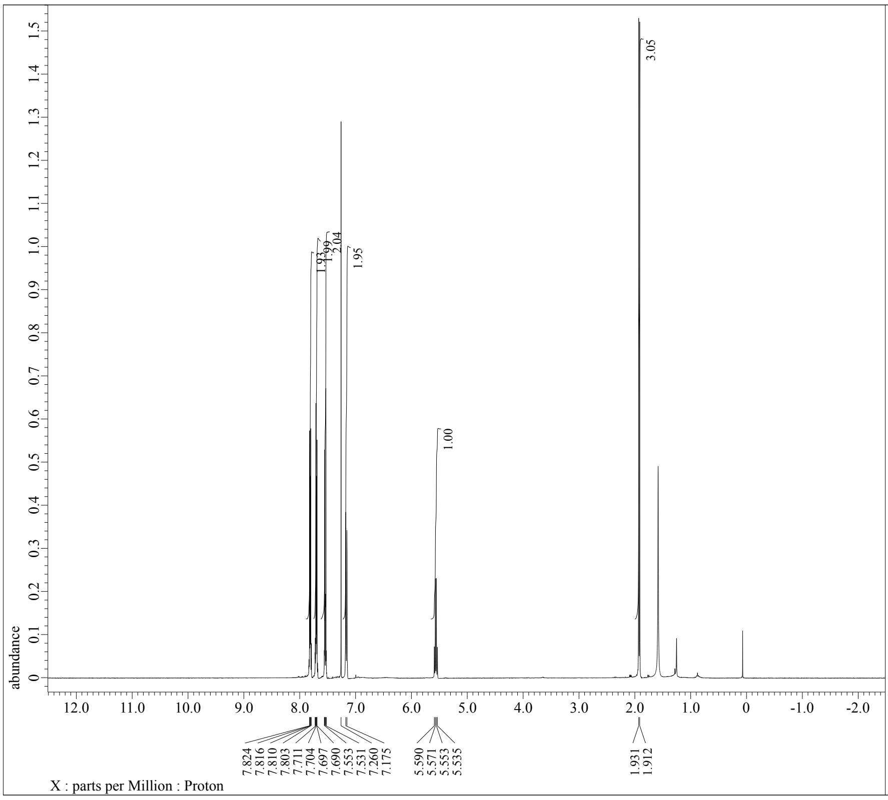
3ad



Pk #	Retention Time	Area	Area Percent
1	22.325	4352199	96.366
2	24.737	164108	3.634



Pk #	Retention Time	Area	Area Percent
1	23.028	883939	49.398
2	24.708	905495	50.602

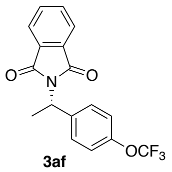


```
Author           = delta
Irr_Freq         = 399.78219838[MHz]
Experiment       = proton.jxp
Solvent          = CHLOROFORM-D

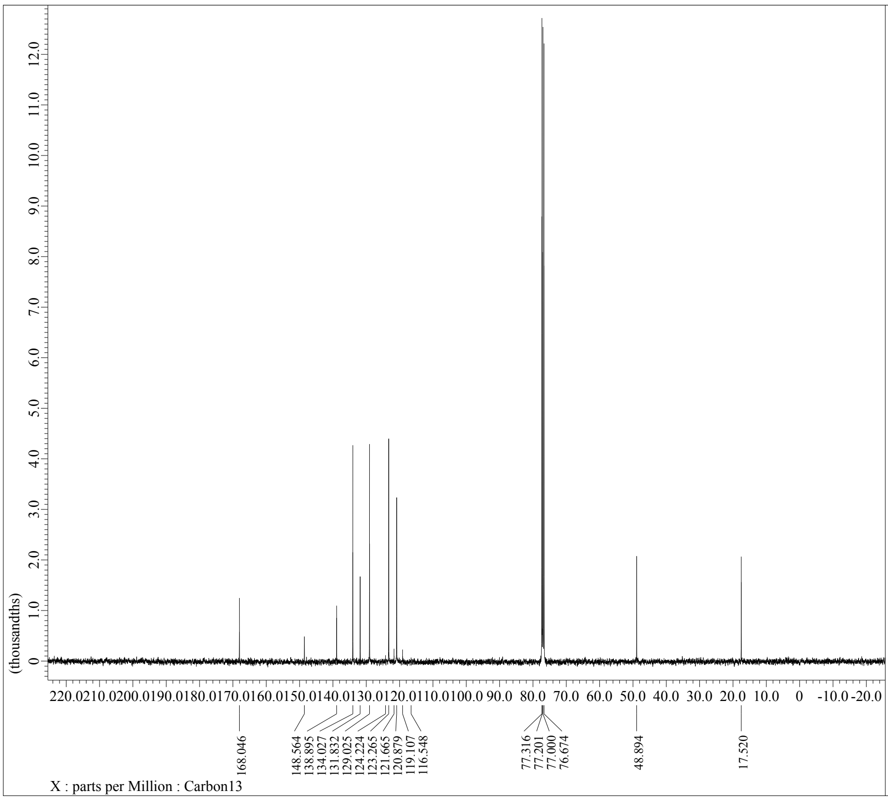
Comment          = single_pulse
Data_Format      = 1D COMPLEX
Dim_Size         = 13107
X_Domain         = Proto
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = JNM-ECZ400S/L1

Field_Strength   = 9.389766[T] (400[MHz])
X_Acq_Duration   = 2.18628096[s]
X_Domain         = 1H
X_Freq           = 399.78219838[MHz]
X_Offset         = 5[ppm]
X_Points         = 16384
X_Prescans       = 1
X_Resolution     = 0.45739775[Hz]
X_Sweep          = 7.4940048[kHz]
X_Sweep_Clipped  = 5.99520384[kHz]
Irr_Domain       = Proton
Irr_Offset       = 5[ppm]
Tri_Domain       = Proton
Tri_Freq         = 399.78219838[MHz]
Tri_Offset       = 5[ppm]
Clipped          = FALSE
Scans            = 4
Total_Scans      = 4

Relaxation_Delay = 5[s]
Recvr_Gain       = 56
Temp_Get         = 22.5[dC]
X_90_Width       = 6.5[us]
X_Acq_Time       = 2.18628096[s]
X_Angle          = 45[deg]
X_Atn            = 0.9[dB]
X_Pulse          = 3.25[us]
Irr_Mode         = Off
Tri_Mode         = Off
Dante_Presat     = FALSE
Initial_Wait     = 1[s]
Repetition_Time  = 7.18628096[s]
```



3af

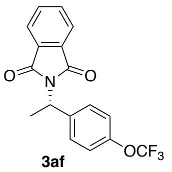


```
Author           = delta
Irr_Freq         = 399.78219838[MHz]
Experiment       = carbon.jxp
Solvent          = CHLOROFORM-D

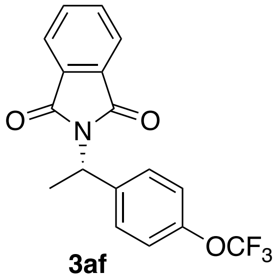
Comment          = single pulse decoupled gat
Data_Format      = 1D COMPLEX
Dim_Size         = 26214
X_Domain         = Carbo
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = JNM-ECZ400S/L1

Field_Strength   = 9.389766[T] (400[MHz])
X_Acq_Duration   = 1.03809024[s]
X_Domain         = 13C
X_Freq           = 100.52530333[MHz]
X_Offset         = 100[ppm]
X_Points         = 32768
X_Prescans       = 4
X_Resolution     = 0.96330739[Hz]
X_Sweep          = 31.56565657[kHz]
X_Sweep_Clipped  = 25.25252525[kHz]
Irr_Domain       = Proton
Irr_Offset       = 5[ppm]
Clipped          = FALSE
Scans            = 935
Total_Scans      = 935

Relaxation_Delay = 2[s]
Recvr_Gain       = 50
Temp_Get         = 21.4[dC]
X_90_Width       = 10.5[us]
X_Acq_Time       = 1.03809024[s]
X_Angle          = 30[deg]
X_Atn            = 3.8[dB]
X_Pulse          = 3.5[us]
Initial_Wait     = 1[s]
Repetition_Time  = 3.03809024[s]
```



3af



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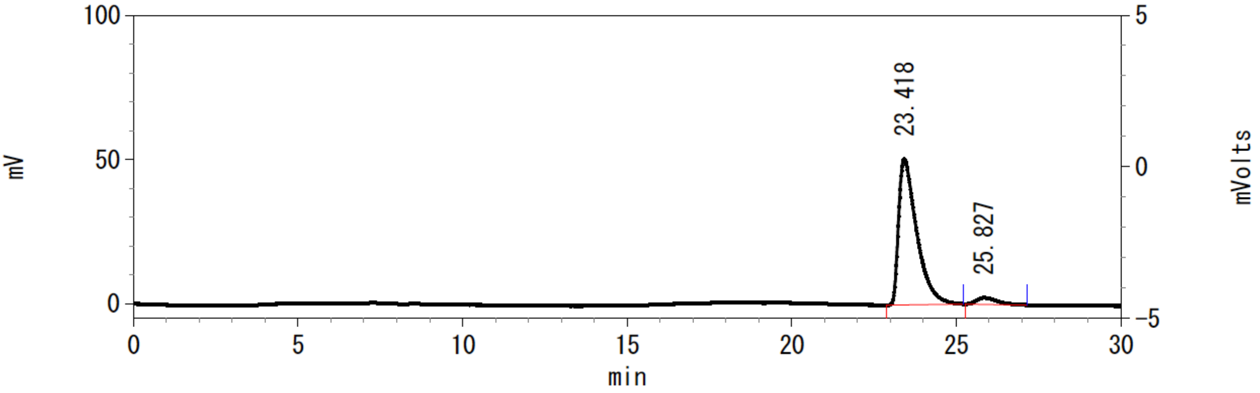
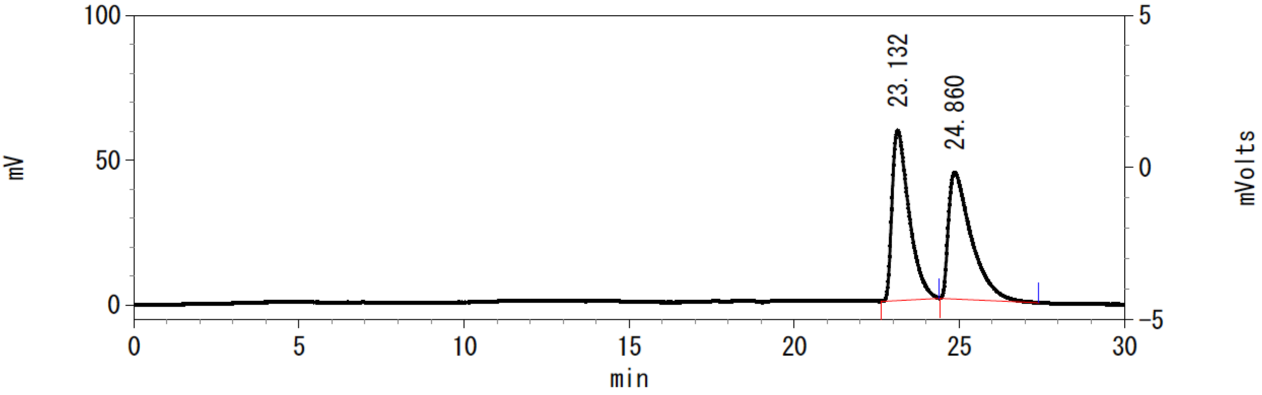
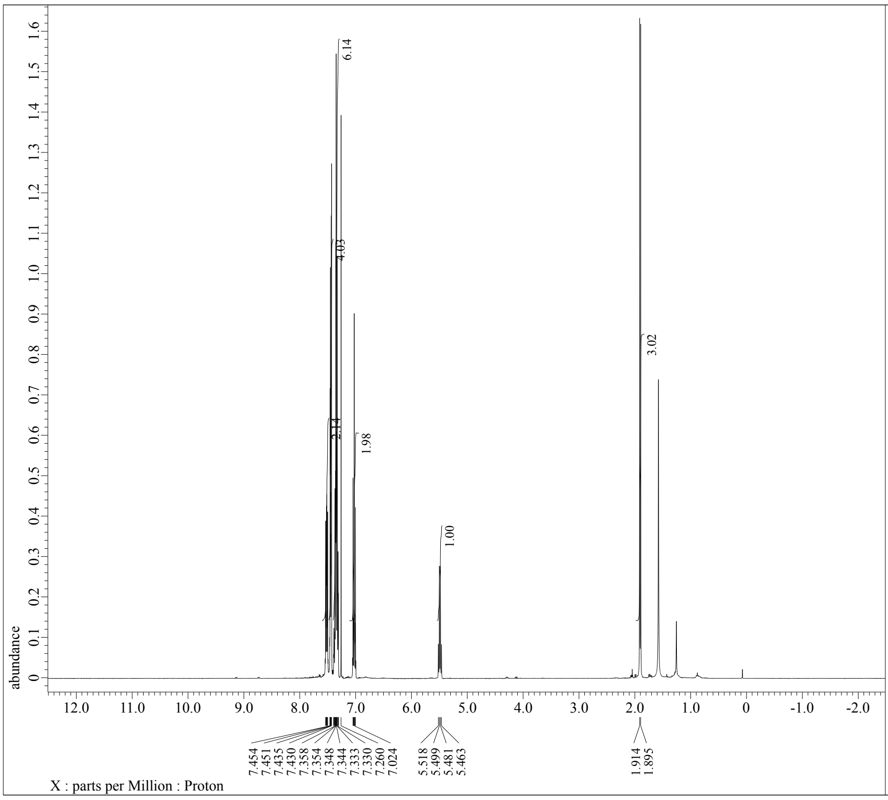


Pk #	Retention Time	Area	Area Percent
1	23.418	1936431	95.080
2	25.827	100200	4.920



Pk #	Retention Time	Area	Area Percent
1	23.132	2099939	49.870
2	24.860	2110883	50.130

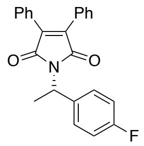


```
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Solvent           = CHLOROFORM-D

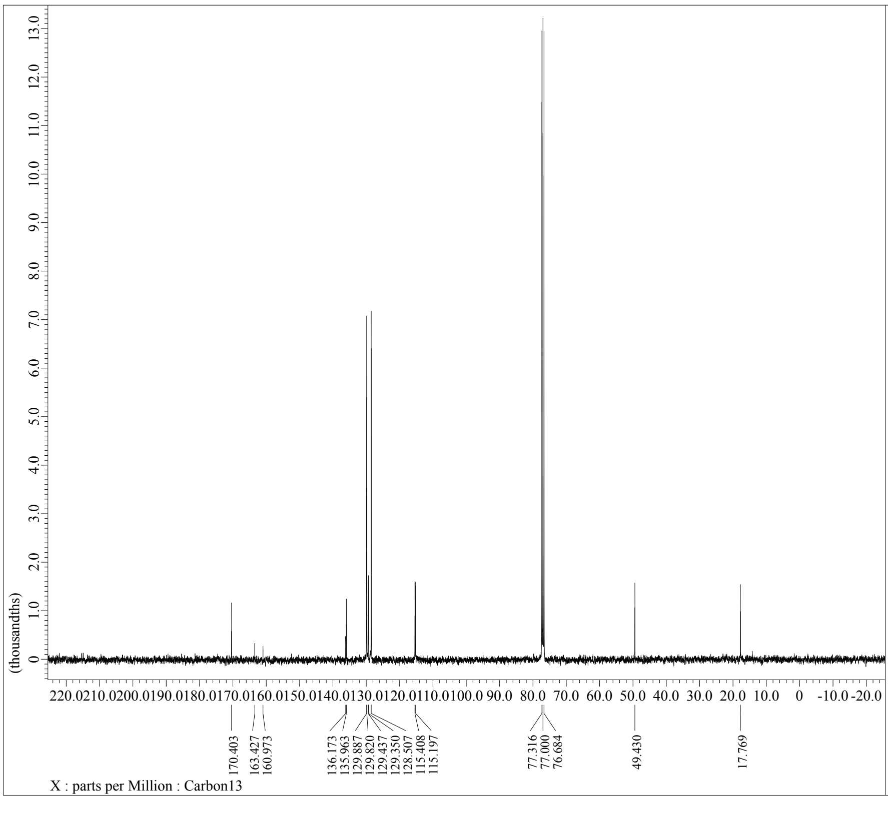
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proto
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18628096[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45739775[Hz]
X_Sweep           = 7.4940048[kHz]
X_Sweep_Clipped   = 5.99520384[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 4
Total_Scans       = 4

Relaxation_Delay  = 5[s]
Recvr_Gain        = 56
Temp_Get          = 21.2[dC]
X_90_Width        = 6.5[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 3.25[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18628096[s]
```



3dg

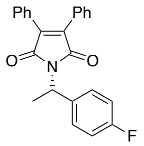


```
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Solvent           = CHLOROFORM-D

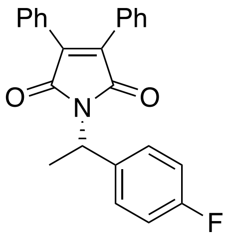
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
X_Domain          = Carbo
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.03809024[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.96330739[Hz]
X_Sweep           = 31.56565657[kHz]
X_Sweep_Clipped   = 25.25252525[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 570
Total_Scans       = 570

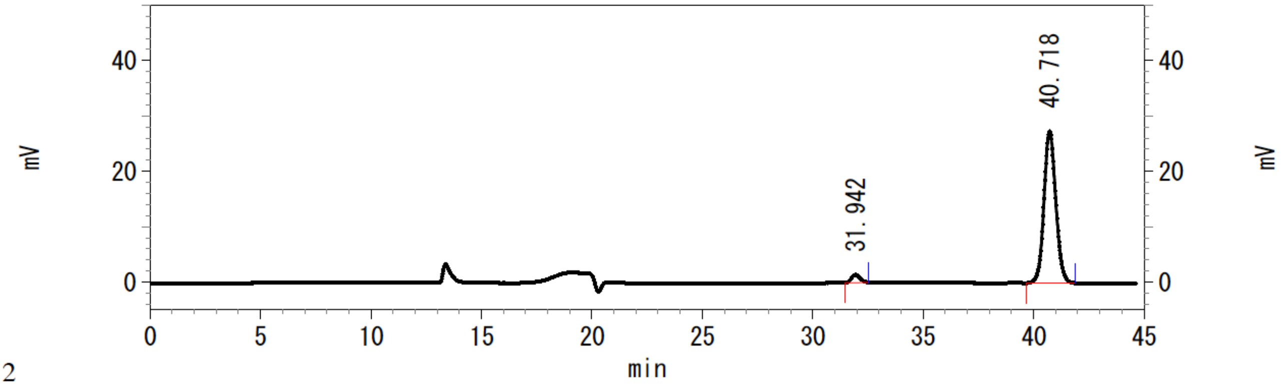
Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 22.2[dC]
X_90_Width        = 10.86[us]
X_Acq_Time        = 1.03809024[s]
X_Angle           = 30[deg]
X_Atn             = 3.8[dB]
X_Pulse           = 3.62[us]
Initial_Wait      = 1[s]
Repetition_Time   = 3.03809024[s]
```



3dg

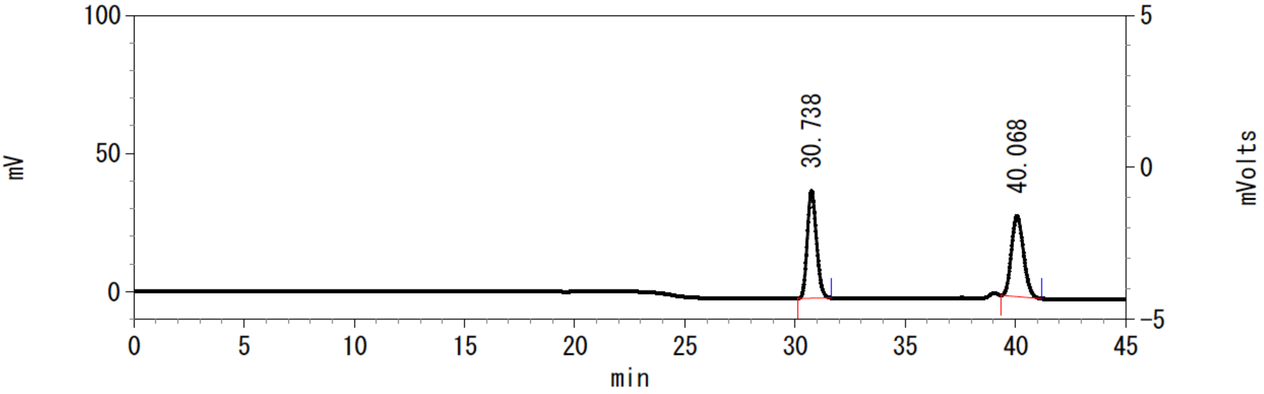


3dg

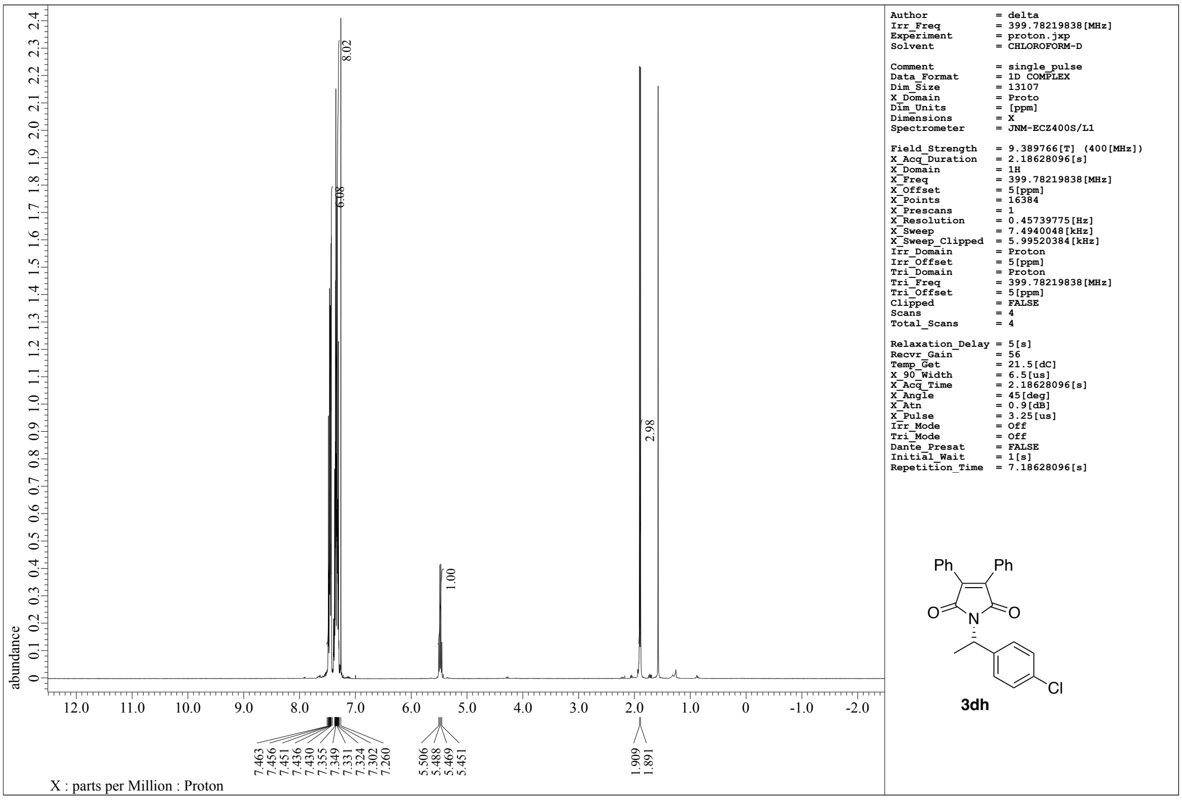


2

Pk #	Retention Time	Area	Area Percent
1	31.942	38091	3.595
2	40.718	1021509	96.405



Pk #	Retention Time	Area	Area Percent
1	30.738	1131868	50.397
2	40.068	1114040	49.603



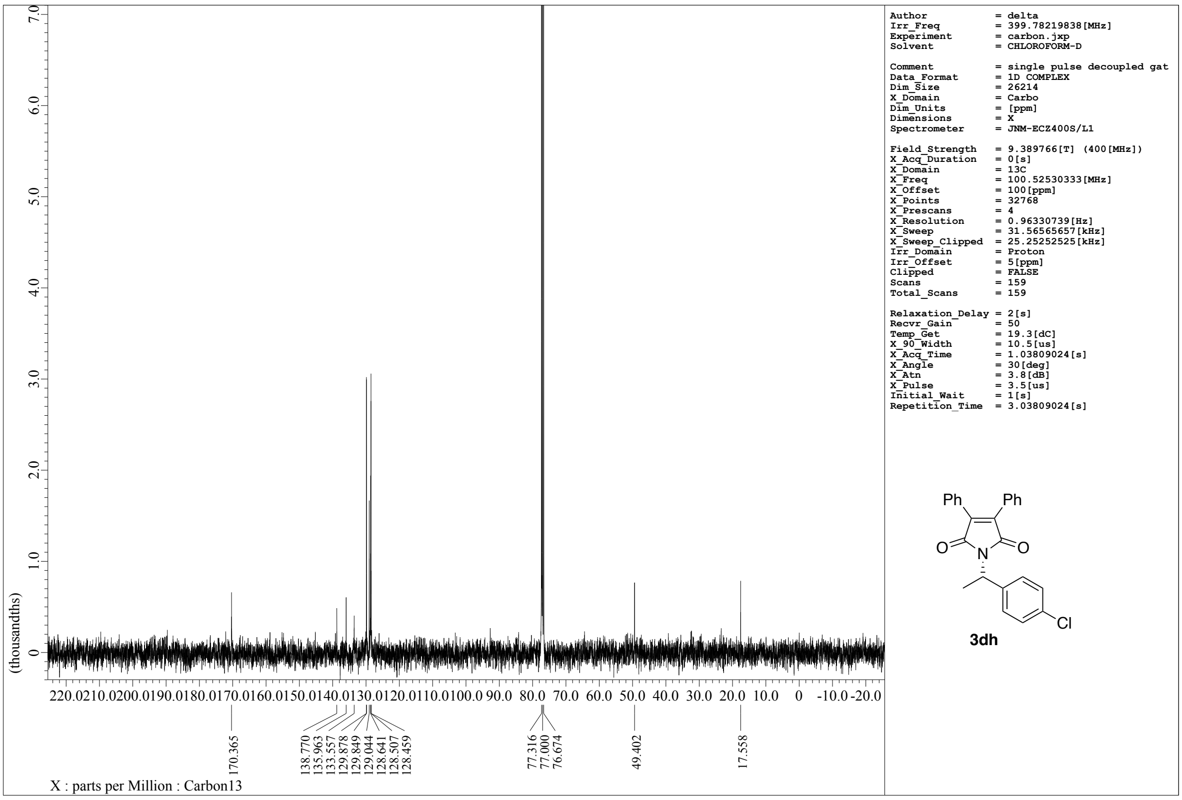
```
Author           = delta
Irr_Freq         = 399.78219838[MHz]
Experiment       = proton.jxp
Solvent          = CHLOROFORM-D

Comment          = single_pulse
Data_Format      = 1D COMPLEX
Dim_Size         = 13107
X_Domain         = Proto
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = JNM-ECZ400S/L1

Field_Strength   = 9.389766[T] (400[MHz])
X_Acq_Duration   = 2.18628096[s]
X_Domain         = 1H
X_Freq           = 399.78219838[MHz]
X_Offset         = 5[ppm]
X_Points         = 16384
X_Prescans       = 1
X_Resolution     = 0.45739775[Hz]
X_Sweep          = 7.4940048[kHz]
X_Sweep_Clipped  = 5.99520384[kHz]
Irr_Domain       = Proton
Irr_Offset       = 5[ppm]
Tri_Domain       = Proton
Tri_Freq         = 399.78219838[MHz]
Tri_Offset       = 5[ppm]
Clipped          = FALSE
Scans            = 4
Total_Scans      = 4

Relaxation_Delay = 5[s]
Recvr_Gain       = 56
Temp_Get         = 21.5[dC]
X_90_Width       = 6.5[us]
X_Acq_Time       = 2.18628096[s]
X_Angle          = 45[deg]
X_Atn            = 0.9[dB]
X_Pulse          = 3.25[us]
Irr_Mode         = Off
Tri_Mode         = Off
Dante_Presat     = FALSE
Initial_Wait     = 1[s]
Repetition_Time  = 7.18628096[s]
```

3dh



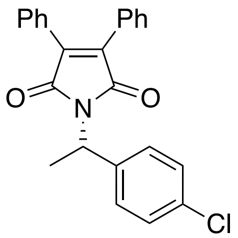
```
Author           = delta
Irr_Freq         = 399.78219838[MHz]
Experiment       = carbon.jxp
Solvent          = CHLOROFORM-D

Comment          = single pulse decoupled gat
Data_Format      = 1D COMPLEX
Dim_Size         = 26214
X_Domain         = Carbo
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = JNM-ECZ400S/L1

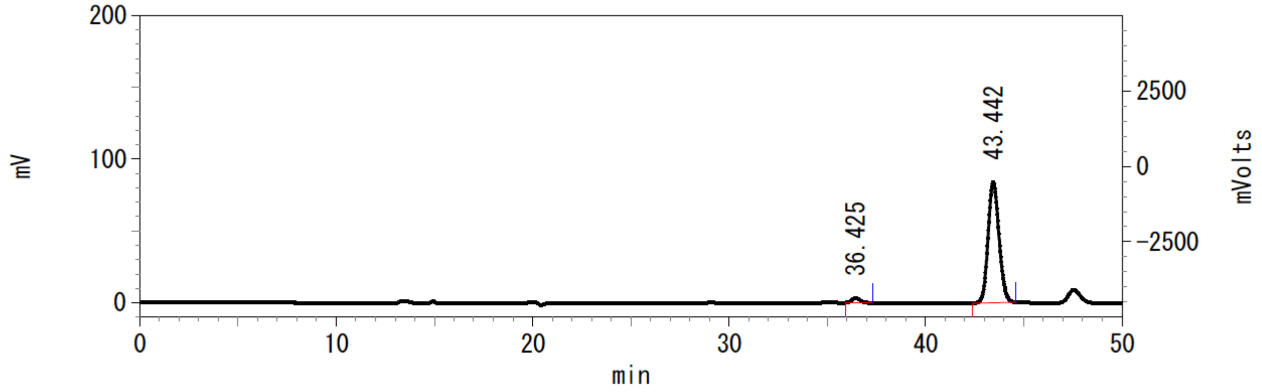
Field_Strength   = 9.389766[T] (400[MHz])
X_Acq_Duration   = 0[s]
X_Domain         = 13C
X_Freq           = 100.52530333[MHz]
X_Offset         = 100[ppm]
X_Points         = 32768
X_Prescans       = 4
X_Resolution     = 0.96330739[Hz]
X_Sweep          = 31.56565657[kHz]
X_Sweep_Clipped  = 25.25252525[kHz]
Irr_Domain       = Proton
Irr_Offset       = 5[ppm]
Clipped          = FALSE
Scans            = 159
Total_Scans      = 159

Relaxation_Delay = 2[s]
Recvr_Gain       = 50
Temp_Get         = 19.3[dC]
X_90_Width       = 10.5[us]
X_Acq_Time       = 1.03809024[s]
X_Angle          = 30[deg]
X_Atn            = 3.8[dB]
X_Pulse          = 3.5[us]
Initial_Wait     = 1[s]
Repetition_Time  = 3.03809024[s]
```

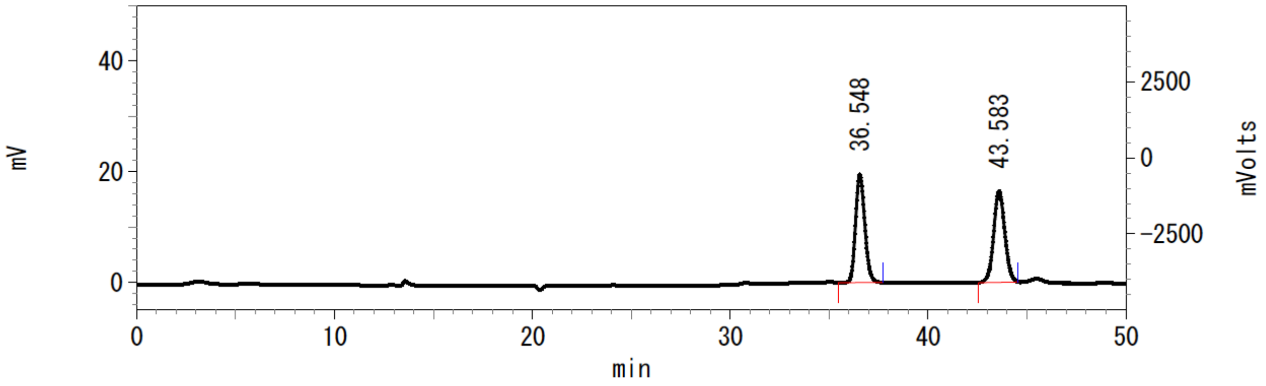
3dh



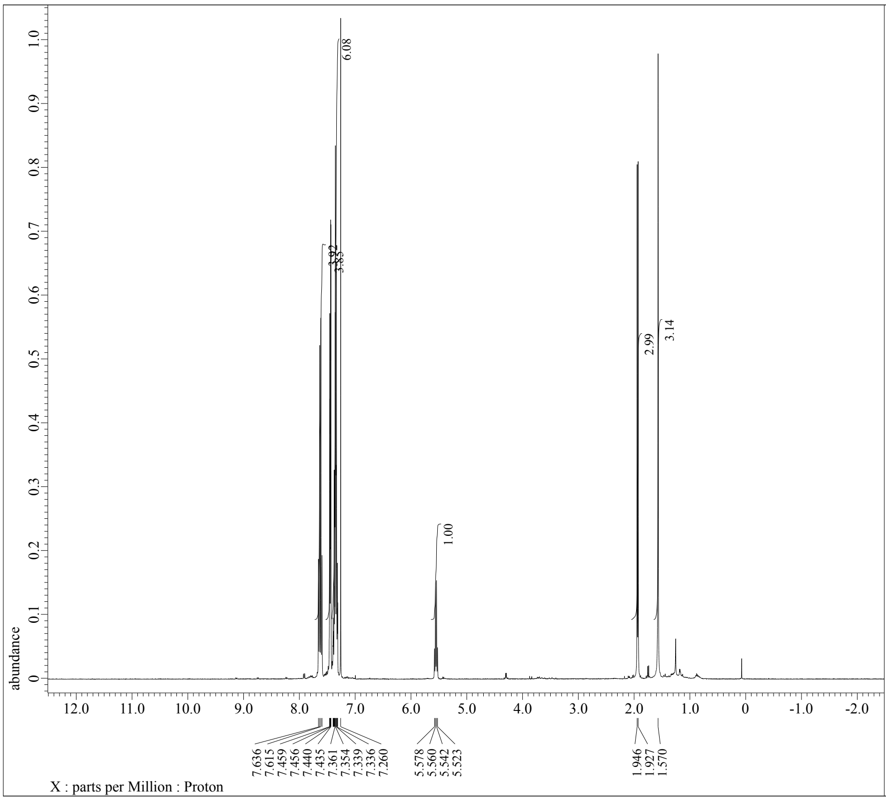
3dh



Pk #	Retention Time	Area	Area Percent
1	36.425	104543	3.110
2	43.442	3256699	96.890



Pk #	Retention Time	Area	Area Percent
1	36.548	633092	49.510
2	43.583	645619	50.490

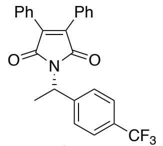


```
Author           = delta
Irr_Freq         = 399.78219838[MHz]
Experiment       = proton.jxp
Solvent          = CHLOROFORM-D

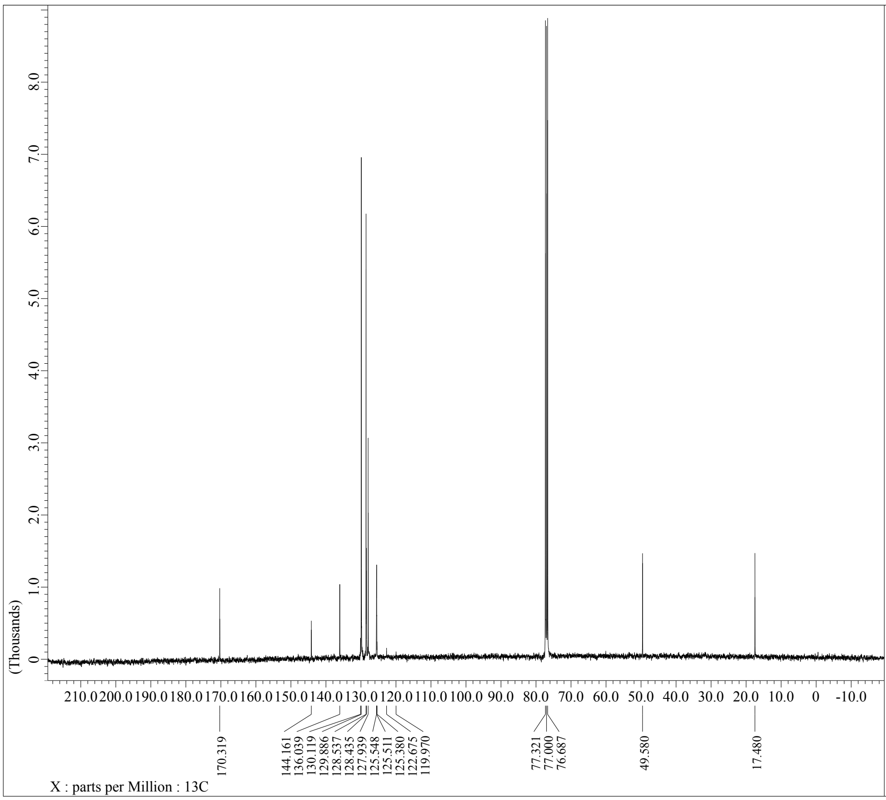
Comment          = single_pulse
Data_Format      = 1D COMPLEX
Dim_Size         = 13107
X_Domain         = Proto
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = JNM-ECZ400S/L1

Field_Strength   = 9.389766[T] (400[MHz])
X_Acq_Duration   = 2.18628096[s]
X_Domain         = 1H
X_Freq           = 399.78219838[MHz]
X_Offset         = 5[ppm]
X_Points         = 16384
X_Prescans       = 1
X_Resolution     = 0.45739775[Hz]
X_Sweep          = 7.4940048[kHz]
X_Sweep_Clipped  = 5.99520384[kHz]
Irr_Domain       = Proton
Irr_Offset       = 5[ppm]
Tri_Domain       = Proton
Tri_Freq         = 399.78219838[MHz]
Tri_Offset       = 5[ppm]
Clipped          = FALSE
Scans            = 4
Total_Scans      = 4

Relaxation_Delay = 5[s]
Recvr_Gain       = 56
Temp_Get         = 21.6[dC]
X_90_Width       = 6.5[us]
X_Acq_Time       = 2.18628096[s]
X_Angle          = 45[deg]
X_Atn            = 0.9[dB]
X_Pulse          = 3.25[us]
Irr_Mode         = Off
Tri_Mode         = Off
Dante_Presat     = FALSE
Initial_Wait     = 1[s]
Repetition_Time  = 7.18628096[s]
```



3di

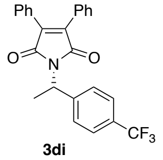


```
Author           = FOC
Experiment       = zgpg30
Solvent          = CHLOROFORM-D

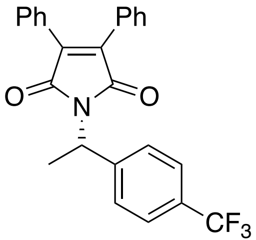
Comment          = Parameter file, TOPSPIN
Data_Format      = 1D COMPLEX
Dim_Size         = 32768
X_Domain         = 13C
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain         = 13C
X_Freq           = 100.61276853[MHz]
X_Offset         = 10.06080281[kHz]
X_Points         = 32768
X_Prescans       = 1
X_Sweep          = 24.03605805[kHz]
Scans            = 918

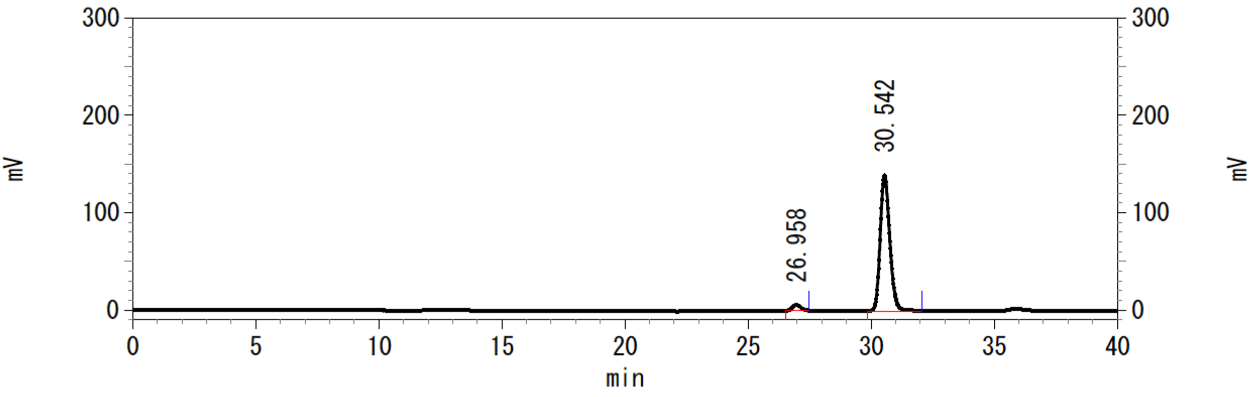
Temp_Get         = 298.5853[K]
```



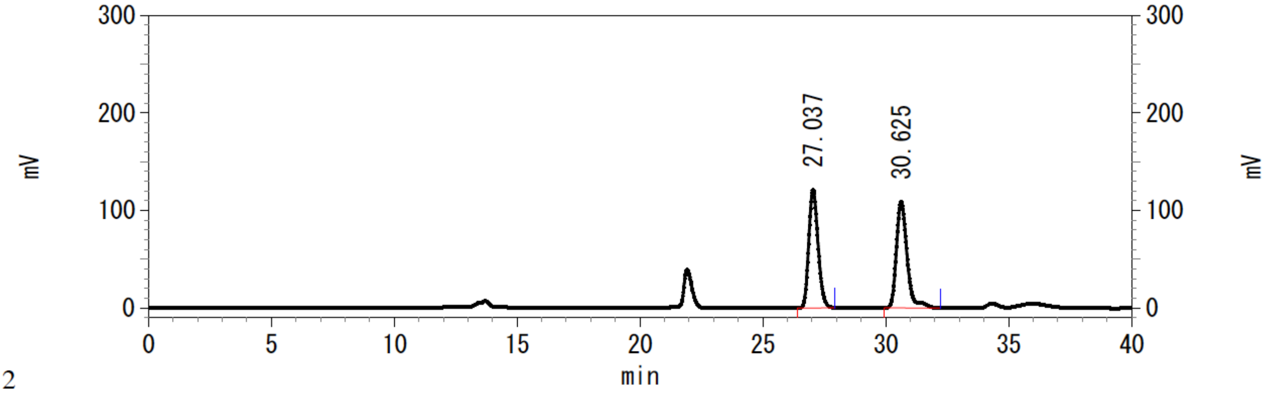
3di



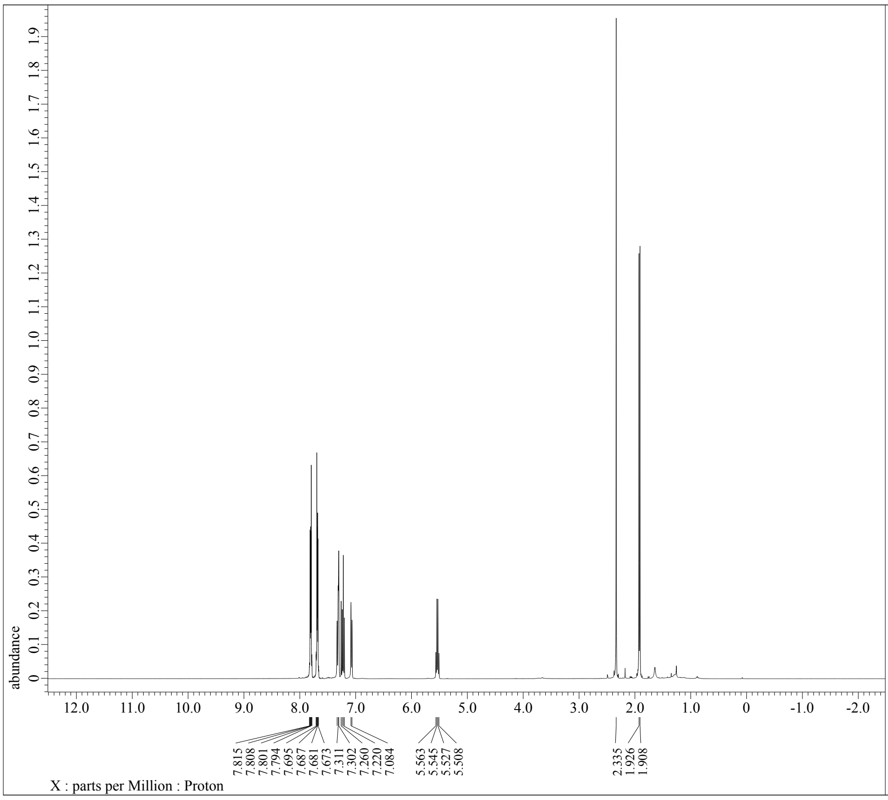
3di



Pk #	Retention Time	Area	Area Percent
1	26.958	138684	3.574
2	30.542	3741559	96.426



Pk #	Retention Time	Area	Area Percent
1	27.037	3201197	49.407
2	30.625	3277994	50.593

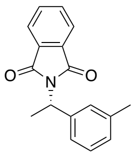


```
Author           = delta
Irr_Freq         = 399.78219838[MHz]
Experiment       = proton.jxp
Solvent          = CHLOROFORM-D

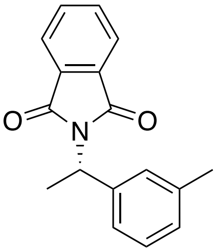
Comment          = single_pulse
Data_Format      = 1D COMPLEX
Dim_Size         = 13107
X_Domain         = Proto
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = JNM-ECZ400S/L1

Field_Strength   = 9.389766[T] (400[MHz])
X_Acq_Duration   = 2.18628096[s]
X_Domain         = 1H
X_Freq           = 399.78219838[MHz]
X_Offset         = 5[ppm]
X_Points         = 16384
X_Prescans       = 1
X_Resolution     = 0.45739775[Hz]
X_Sweep          = 7.4940048[kHz]
X_Sweep_Clipped  = 5.99520384[kHz]
Irr_Domain       = Proton
Irr_Offset       = 5[ppm]
Tri_Domain       = Proton
Tri_Freq         = 399.78219838[MHz]
Tri_Offset       = 5[ppm]
Clipped          = FALSE
Scans            = 4
Total_Scans      = 4

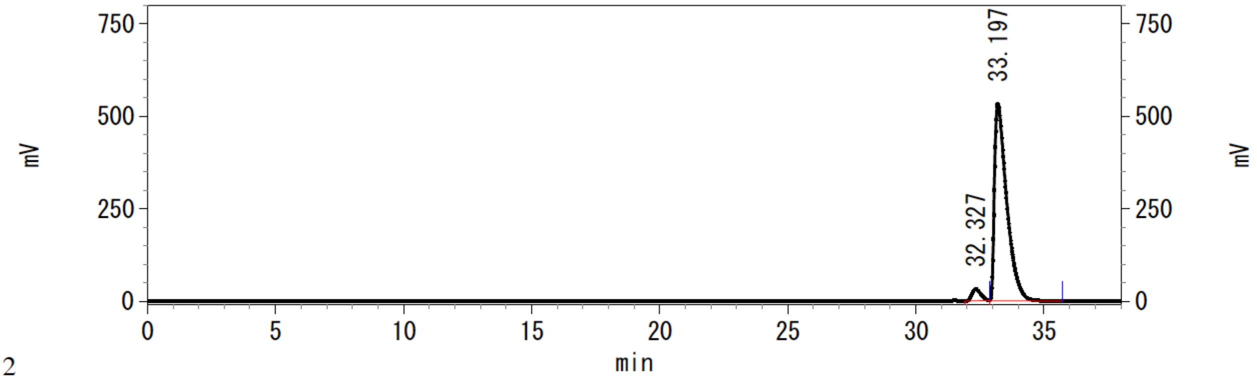
Relaxation_Delay = 5[s]
Recvr_Gain       = 46
Temp_Get         = 18.8[dC]
X_90_Width       = 6.3[us]
X_Acq_Time       = 2.18628096[s]
X_Angle          = 45[deg]
X_Atn            = 0.9[dB]
X_Pulse          = 3.15[us]
Irr_Mode         = Off
Tri_Mode         = Off
Dante_Presat     = FALSE
Initial_Wait     = 1[s]
Repetition_Time  = 7.18628096[s]
```



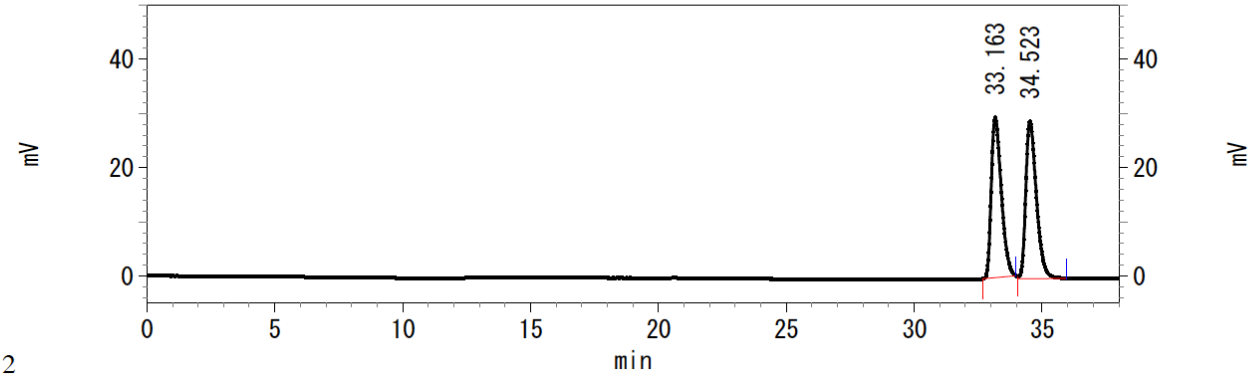
3aj



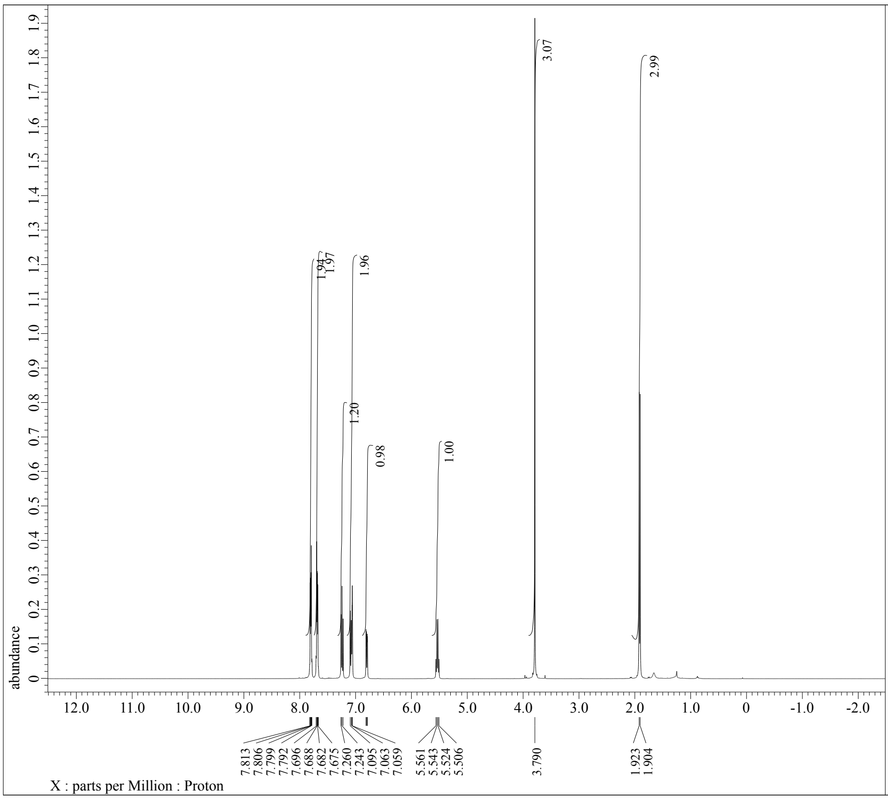
3aj



Pk #	Retention Time	Area	Area Percent
1	32.327	758331	4.109
2	33.197	17695734	95.891



Pk #	Retention Time	Area	Area Percent
1	33.163	807516	49.049
2	34.523	838822	50.951

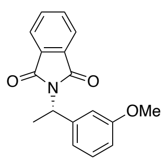


```
Author           = delta
Irr_Freq         = 399.78219838[MHz]
Experiment       = proton.jxp
Solvent          = CHLOROFORM-D

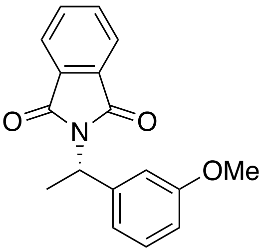
Comment          = single_pulse
Data_Format      = 1D COMPLEX
Dim_Size         = 13107
X_Domain         = Proto
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = JNM-ECZ400S/L1

Field_Strength   = 9.389766[T] (400[MHz])
X_Acq_Duration   = 2.18628096[s]
X_Domain         = 1H
X_Freq           = 399.78219838[MHz]
X_Offset         = 5[ppm]
X_Points         = 16384
X_Prescans       = 1
X_Resolution     = 0.45739775[Hz]
X_Sweep          = 7.4940048[kHz]
X_Sweep_Clipped  = 5.99520384[kHz]
Irr_Domain       = Proton
Irr_Offset       = 5[ppm]
Tri_Domain       = Proton
Tri_Freq         = 399.78219838[MHz]
Tri_Offset       = 5[ppm]
Clipped          = FALSE
Scans            = 8
Total_Scans      = 8

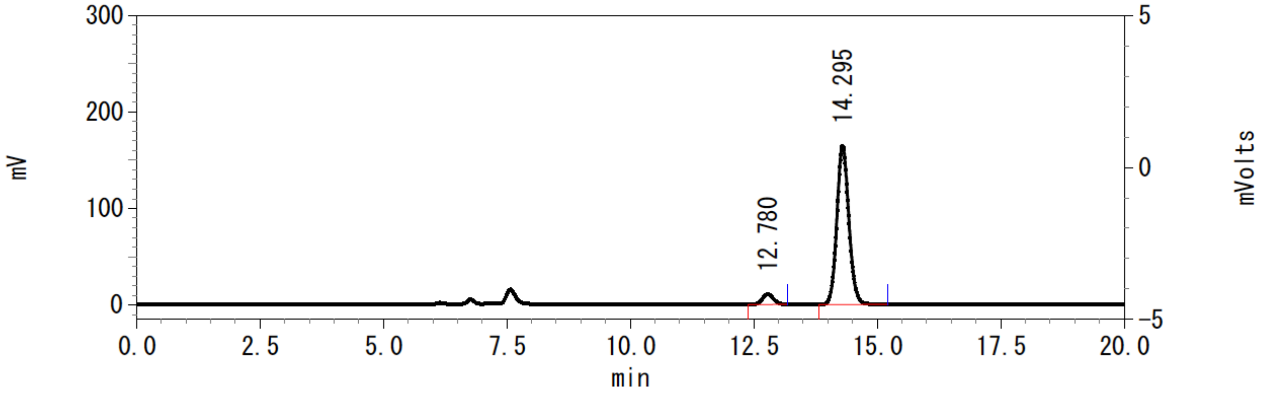
Relaxation_Delay = 5[s]
Recvr_Gain       = 46
Temp_Get         = 15.6[dC]
X_90_Width       = 6.3[us]
X_Acq_Time       = 2.18628096[s]
X_Angle          = 45[deg]
X_Atn            = 0.9[dB]
X_Pulse          = 3.15[us]
Irr_Mode         = Off
Tri_Mode         = Off
Dante_Presat     = FALSE
Initial_Wait     = 1[s]
Repetition_Time  = 7.18628096[s]
```



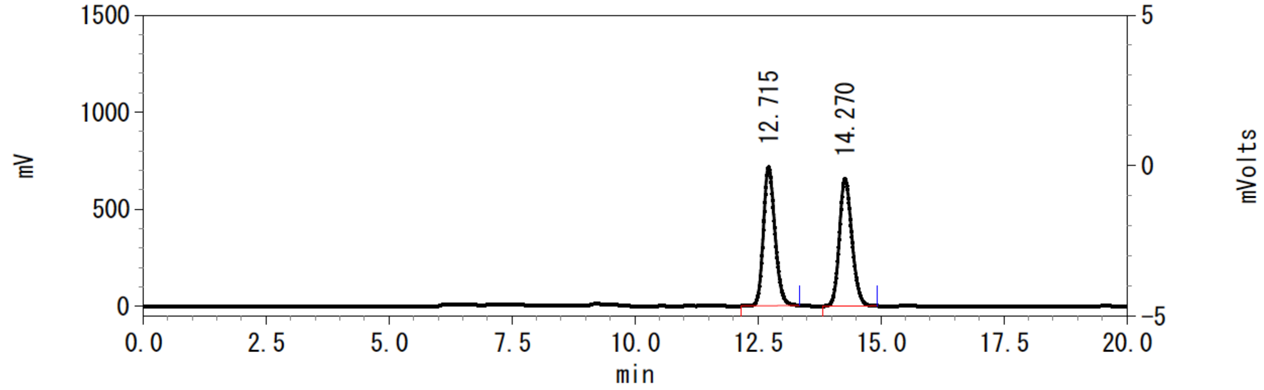
3ak



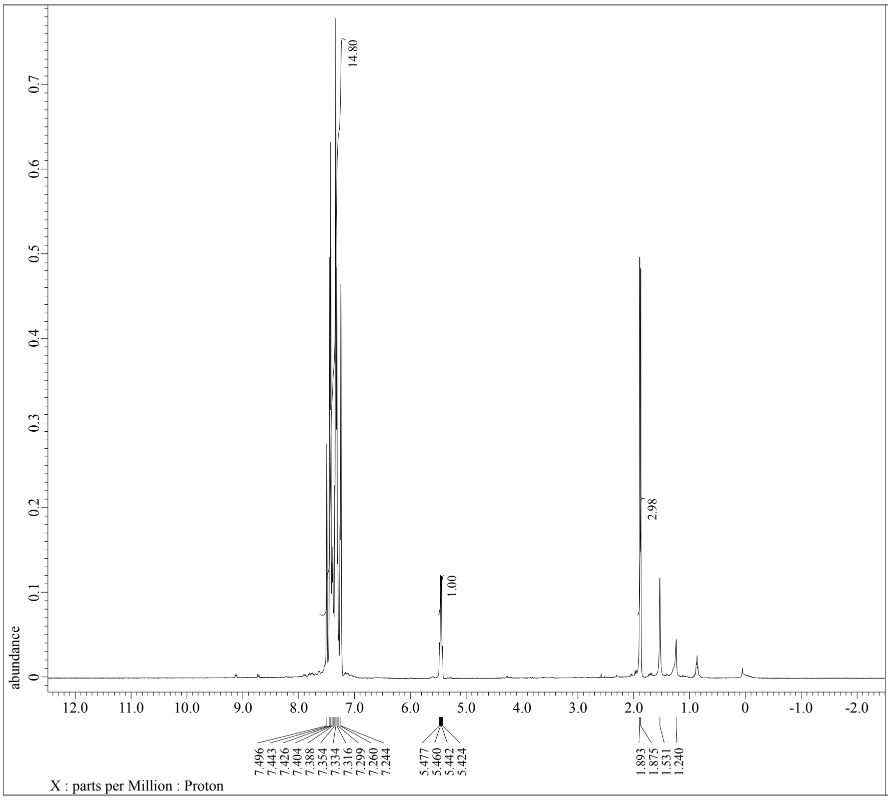
3ak



Pk #	Retention Time	Area	Area Percent
1	12.780	168097	5.691
2	14.295	2785583	94.309



Pk #	Retention Time	Area	Area Percent
1	12.715	11675874	49.975
2	14.270	11687549	50.025

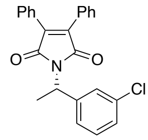


```
Author           = delta
Irr_Freq         = 399.78219838[MHz]
Experiment       = proton.jxp
Solvent          = CHLOROFORM-D

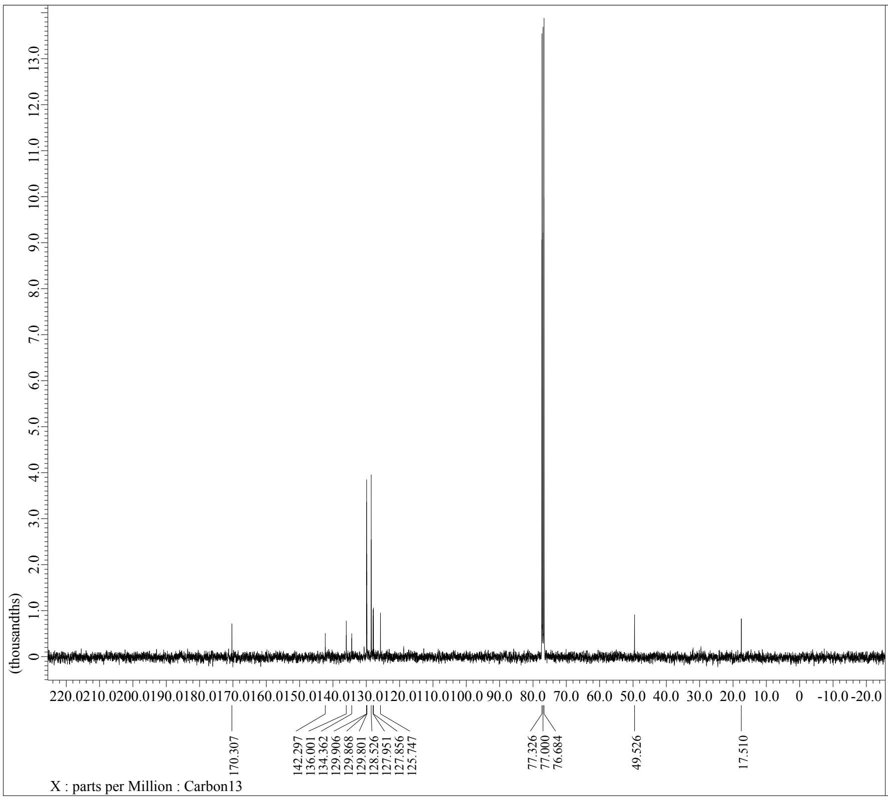
Comment          = single_pulse
Data_Format      = 1D COMPLEX
Dim_Size         = 13107
X_Domain         = Proto
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = JNM-ECZ400S/L1

Field_Strength   = 9.389766[T] (400[MHz])
X_Acq_Duration   = 2.18628096[s]
X_Domain         = 1H
X_Freq           = 399.78219838[MHz]
X_Offset         = 5[ppm]
X_Points         = 16384
X_Prescans       = 1
X_Resolution     = 0.45739775[Hz]
X_Sweep          = 7.4940048[kHz]
X_Sweep_Clipped  = 5.99520384[kHz]
Irr_Domain       = Proton
Irr_Offset       = 5[ppm]
Tri_Domain       = Proton
Tri_Freq         = 399.78219838[MHz]
Tri_Offset       = 5[ppm]
Clipped          = FALSE
Scans            = 8
Total_Scans      = 8

Relaxation_Delay = 5[s]
Recvr_Gain       = 56
Temp_Get         = 22.1[dC]
X_90_Width       = 6.3[us]
X_Acq_Time       = 2.18628096[s]
X_Angle          = 45[deg]
X_Atn            = 0.9[dB]
X_Pulse          = 3.15[us]
Irr_Mode         = Off
Tri_Mode         = Off
Dante_Presat     = FALSE
Initial_Wait     = 1[s]
Repetition_Time  = 7.18628096[s]
```



3dl

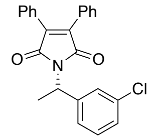


```
Author           = delta
Irr_Freq         = 399.78219838[MHz]
Experiment       = carbon.jxp
Solvent          = CHLOROFORM-D

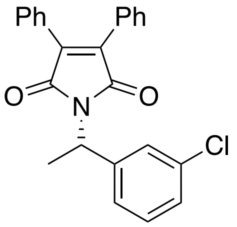
Comment          = single pulse decoupled gat
Data_Format      = 1D COMPLEX
Dim_Size         = 26214
X_Domain         = Carbo
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = JNM-ECZ400S/L1

Field_Strength   = 9.389766[T] (400[MHz])
X_Acq_Duration   = 1.03809024[s]
X_Domain         = 13C
X_Freq           = 100.52530333[MHz]
X_Offset         = 100[ppm]
X_Points         = 32768
X_Prescans       = 4
X_Resolution     = 0.96330739[Hz]
X_Sweep          = 31.56565657[kHz]
X_Sweep_Clipped  = 25.25252525[kHz]
Irr_Domain       = Proton
Irr_Offset       = 5[ppm]
Clipped          = FALSE
Scans            = 248
Total_Scans      = 248

Relaxation_Delay = 2[s]
Recvr_Gain       = 50
Temp_Get         = 22.1[dC]
X_90_Width       = 10.86[us]
X_Acq_Time       = 1.03809024[s]
X_Angle          = 30[deg]
X_Atn            = 3.8[dB]
X_Pulse          = 3.62[us]
Initial_Wait     = 1[s]
Repetition_Time  = 3.03809024[s]
```



3dl



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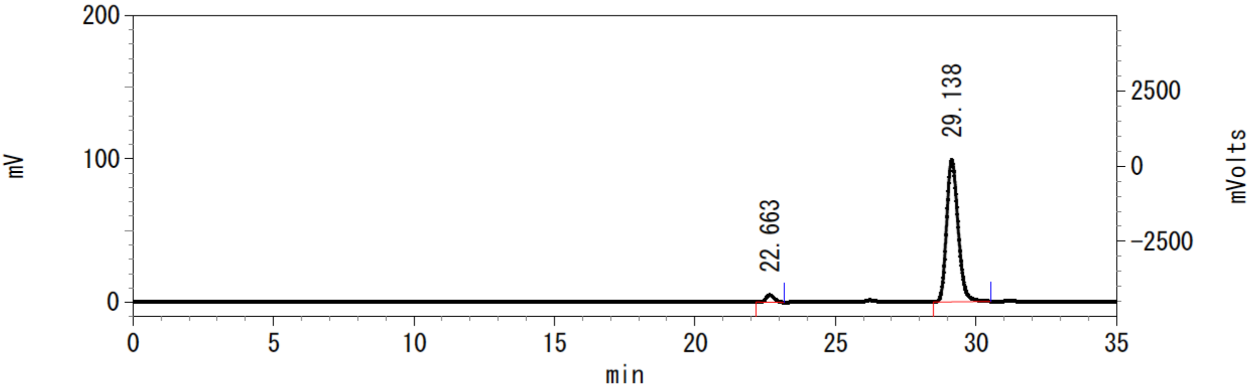
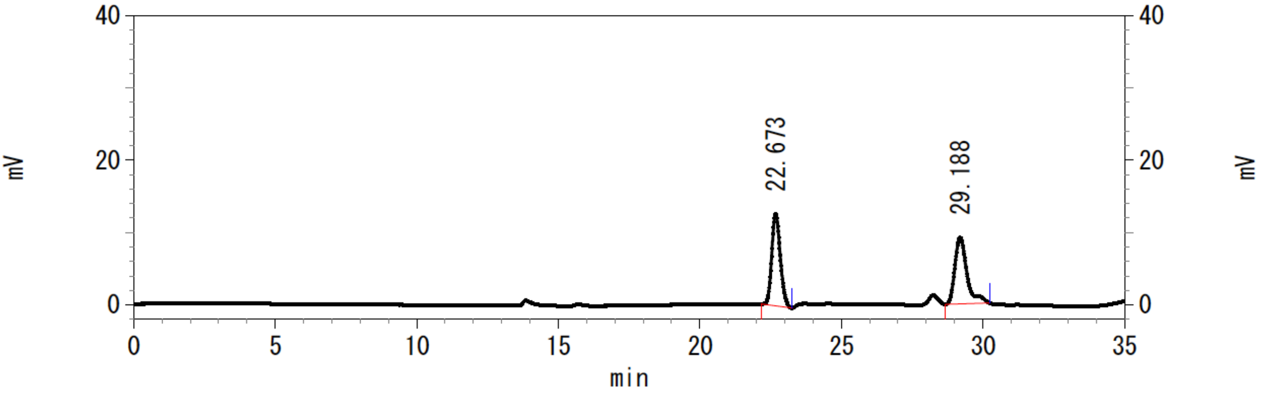
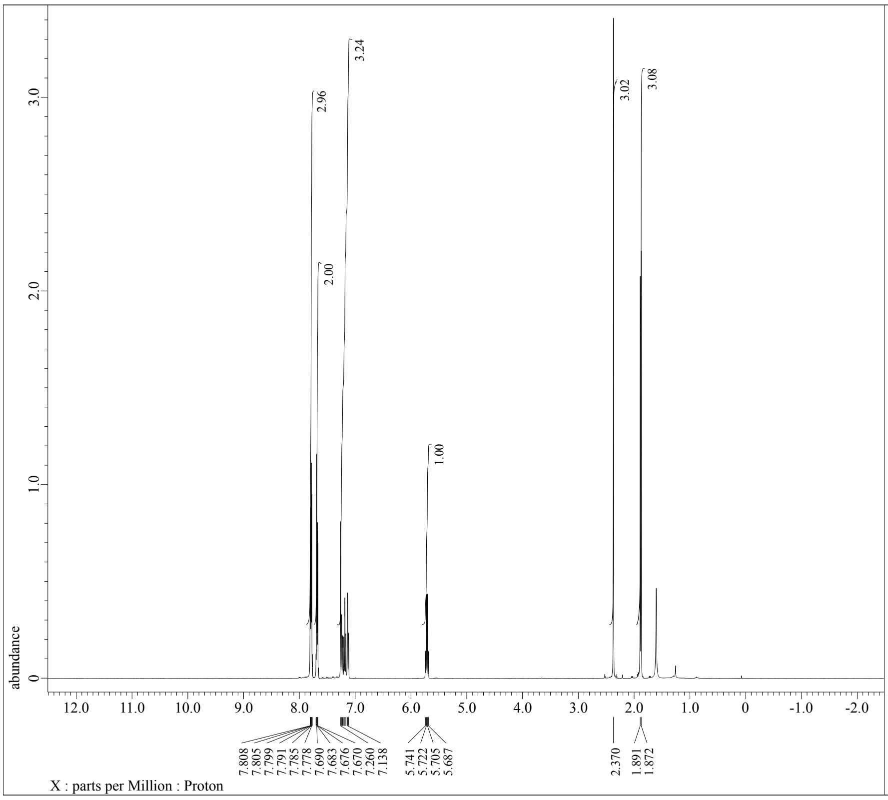


Pk #	Retention Time	Area	Area Percent
1	22.663	103030	3.537
2	29.138	2810302	96.464



2

Pk #	Retention Time	Area	Area Percent
1	22.673	266343	49.516
2	29.188	271552	50.484

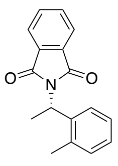


```
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Solvent           = CHLOROFORM-D

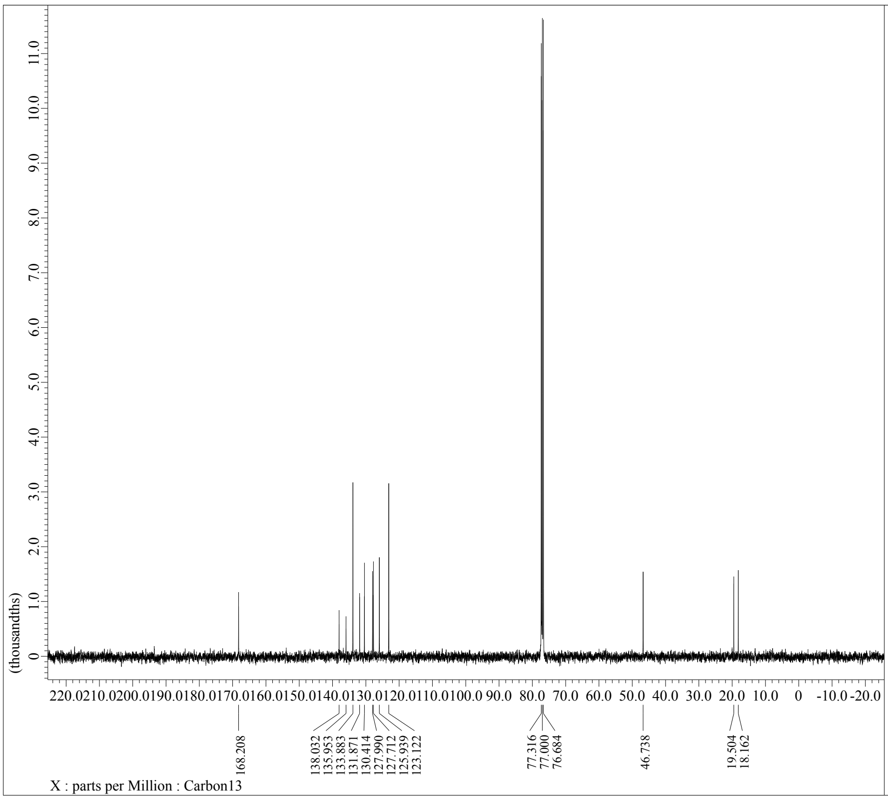
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proto
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18628096[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45739775[Hz]
X_Sweep           = 7.4940048[kHz]
X_Sweep_Clipped   = 5.99520384[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 4
Total_Scans       = 4

Relaxation_Delay  = 5[s]
Recvr_Gain        = 56
Temp_Get          = 22.4[dC]
X_90_Width        = 6.5[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 3.25[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18628096[s]
```



3am

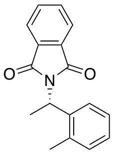


```
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Solvent           = CHLOROFORM-D

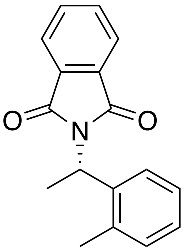
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
X_Domain          = Carbo
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.03809024[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.96330739[Hz]
X_Sweep           = 31.56565657[kHz]
X_Sweep_Clipped   = 25.25252525[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 350
Total_Scans       = 350

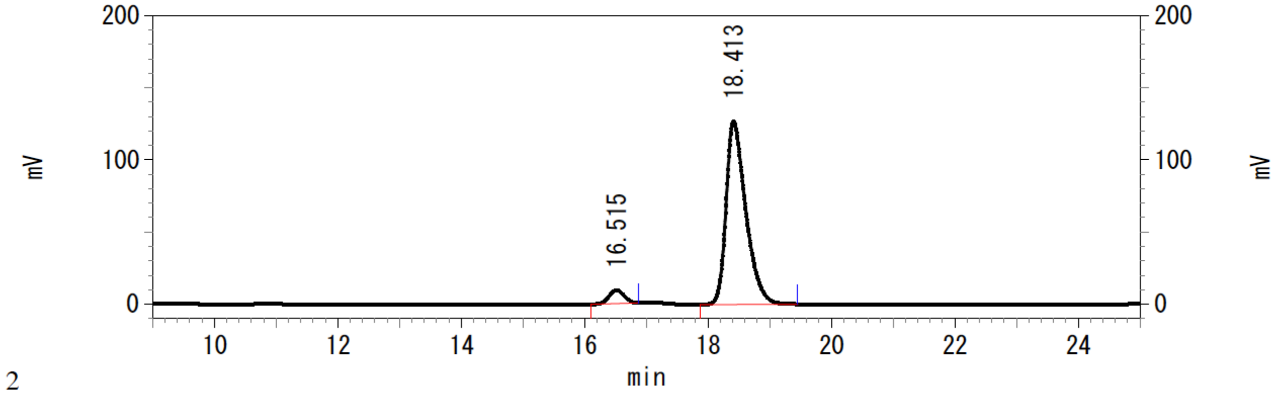
Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 22.5[dC]
X_90_Width        = 10.5[us]
X_Acq_Time        = 1.03809024[s]
X_Angle           = 30[deg]
X_Atn             = 3.8[dB]
X_Pulse           = 3.5[us]
Initial_Wait      = 1[s]
Repetition_Time   = 3.03809024[s]
```



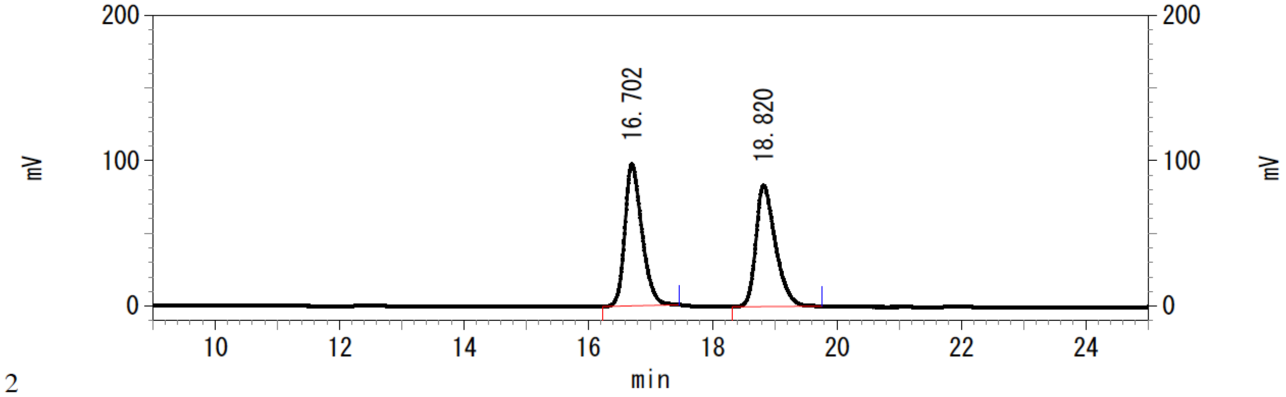
3am



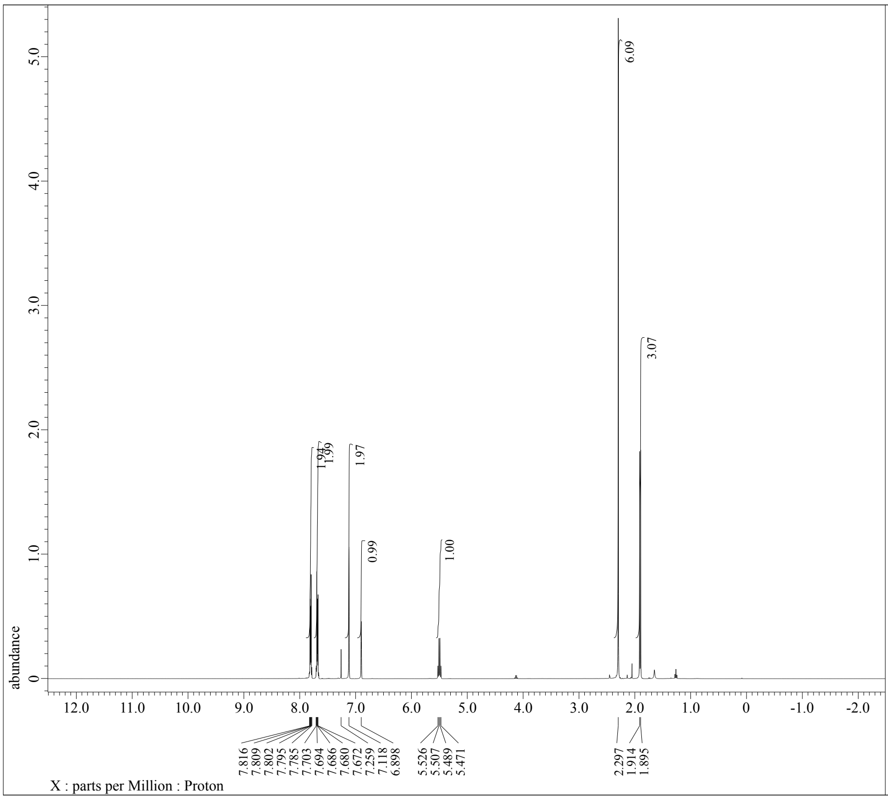
3am



Pk #	Retention Time	Area	Area Percent
1	16.515	161879	5.440
2	18.413	2813765	94.560



Pk #	Retention Time	Area	Area Percent
1	16.702	1863212	50.541
2	18.820	1823293	49.459

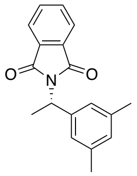


```
Author          = delta
Irr_Freq        = 399.78219838[MHz]
Experiment      = proton.jxp
Solvent         = CHLOROFORM-D

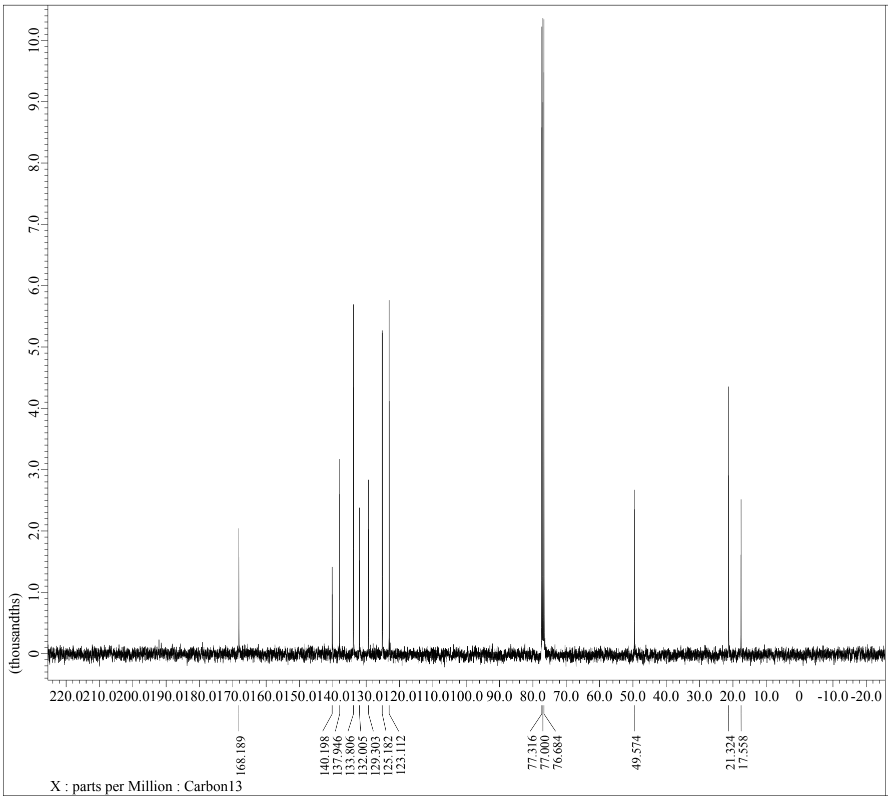
Comment         = single_pulse
Data_Format     = 1D COMPLEX
Dim_Size        = 13107
X_Domain        = Proto
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = JNM-ECZ400S/L1

Field_Strength  = 9.389766[T] (400[MHz])
X_Acq_Duration  = 2.18628096[s]
X_Domain        = 1H
X_Freq          = 399.78219838[MHz]
X_Offset        = 5[ppm]
X_Points        = 16384
X_Prescans      = 1
X_Resolution    = 0.45739775[Hz]
X_Sweep         = 7.4940048[kHz]
X_Sweep_Clipped = 5.99520384[kHz]
Irr_Domain      = Proton
Irr_Offset      = 5[ppm]
Tri_Domain      = Proton
Tri_Freq        = 399.78219838[MHz]
Tri_Offset      = 5[ppm]
Clipped         = FALSE
Scans           = 4
Total_Scans     = 4

Relaxation_Delay = 5[s]
Recvr_Gain      = 46
Temp_Get        = 21.4[dC]
X_90_Width      = 6.5[us]
X_Acq_Time      = 2.18628096[s]
X_Angle         = 45[deg]
X_Atn           = 0.9[dB]
X_Pulse         = 3.25[us]
Irr_Mode        = Off
Tri_Mode        = Off
Dante_Presat    = FALSE
Initial_Wait    = 1[s]
Repetition_Time = 7.18628096[s]
```



3an

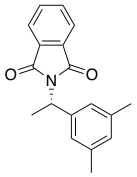


```
Author          = delta
Irr_Freq        = 399.78219838[MHz]
Experiment      = carbon.jxp
Solvent         = CHLOROFORM-D

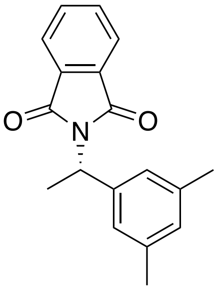
Comment         = single pulse decoupled gat
Data_Format     = 1D COMPLEX
Dim_Size        = 26214
X_Domain        = Carbo
Dim_Units       = [ppm]
Dimensions      = X
Spectrometer    = JNM-ECZ400S/L1

Field_Strength  = 9.389766[T] (400[MHz])
X_Acq_Duration  = 1.03809024[s]
X_Domain        = 13C
X_Freq          = 100.52530333[MHz]
X_Offset        = 100[ppm]
X_Points        = 32768
X_Prescans      = 4
X_Resolution    = 0.96330739[Hz]
X_Sweep         = 31.56565657[kHz]
X_Sweep_Clipped = 25.25252525[kHz]
Irr_Domain      = Proton
Irr_Offset      = 5[ppm]
Clipped         = FALSE
Scans           = 256
Total_Scans     = 256

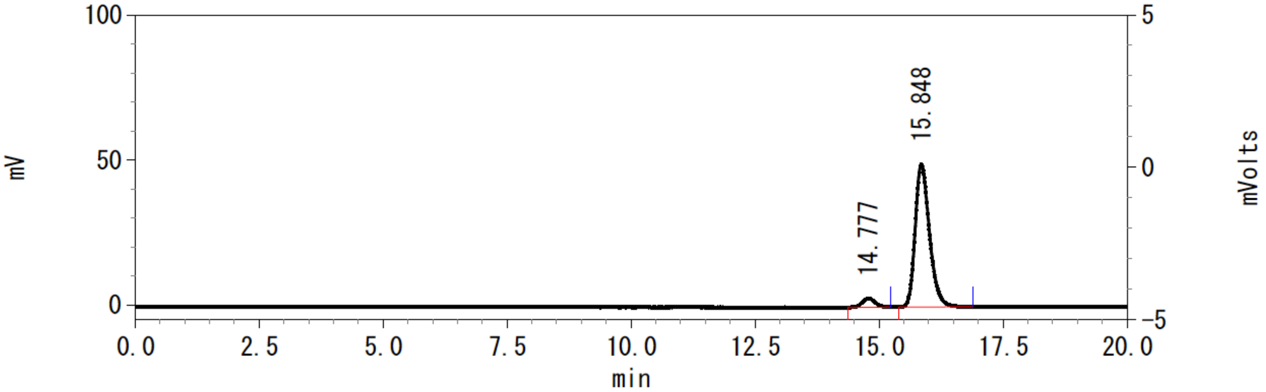
Relaxation_Delay = 2[s]
Recvr_Gain      = 50
Temp_Get        = 21.5[dC]
X_90_Width      = 10.5[us]
X_Acq_Time      = 1.03809024[s]
X_Angle         = 30[deg]
X_Atn           = 3.8[dB]
X_Pulse         = 3.5[us]
Initial_Wait    = 1[s]
Repetition_Time = 3.03809024[s]
```



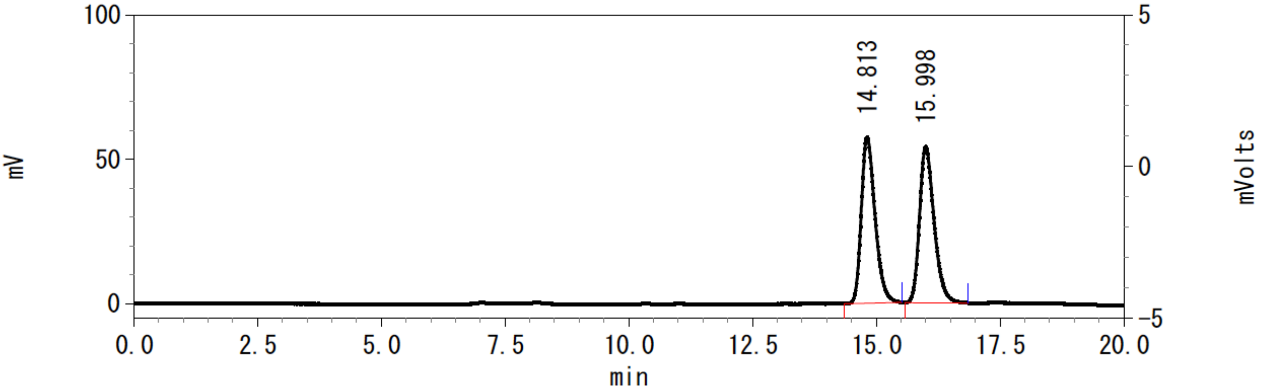
3an



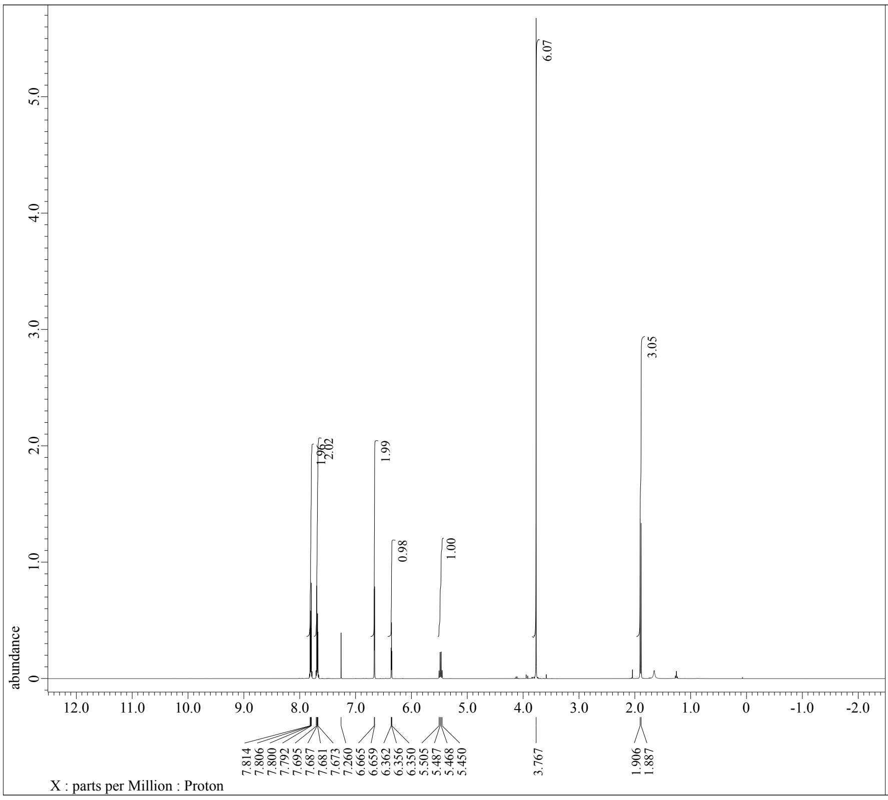
3an



Pk #	Retention Time	Area	Area Percent
1	14.777	55876	5.342
2	15.848	990191	94.658



Pk #	Retention Time	Area	Area Percent
1	14.813	1113706	50.091
2	15.998	1109666	49.909

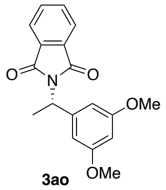


```
Author             = delta
Irr_Freq           = 399.78219838[MHz]
Experiment         = proton.jxp
Solvent            = CHLOROFORM-D

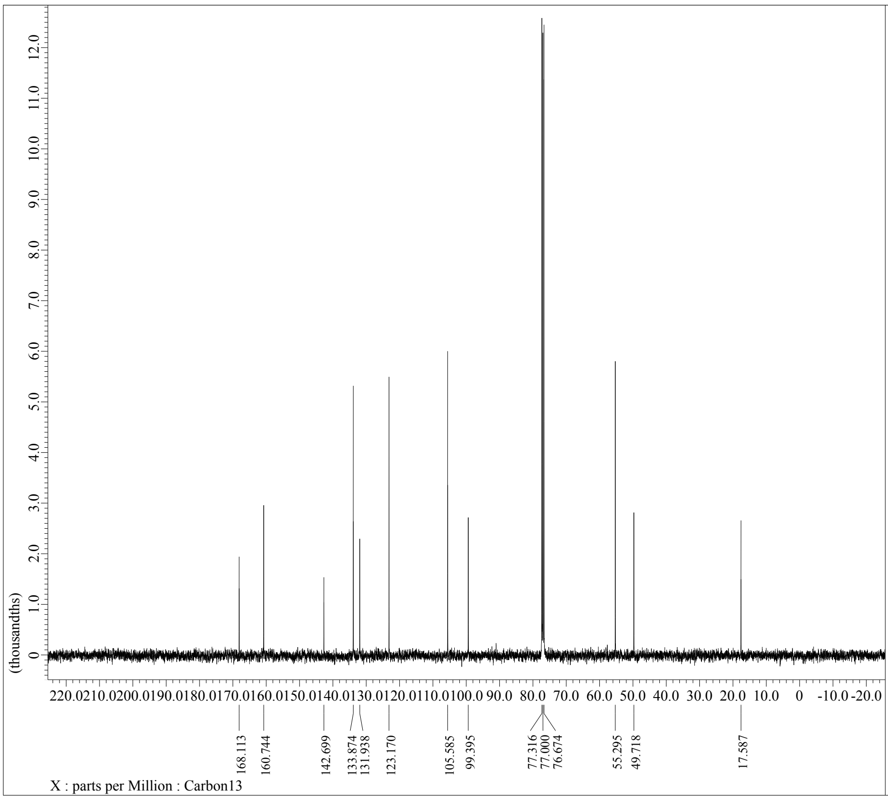
Comment            = single_pulse
Data_Format        = 1D COMPLEX
Dim_Size           = 13107
X_Domain           = Proto
Dim_Units          = [ppm]
Dimensions         = X
Spectrometer       = JNM-ECZ400S/L1

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 2.18628096[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 16384
X_Prescans         = 1
X_Resolution       = 0.45739775[Hz]
X_Sweep            = 7.4940048[kHz]
X_Sweep_Clipped    = 5.99520384[kHz]
Irr_Domain         = Proton
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 399.78219838[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 4
Total_Scans        = 4

Relaxation_Delay = 5[s]
Recvr_Gain         = 46
Temp_Get           = 22.8[dC]
X_90_Width         = 6.5[us]
X_Acq_Time         = 2.18628096[s]
X_Angle            = 45[deg]
X_Atn              = 0.9[dB]
X_Pulse            = 3.25[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
Initial_Wait       = 1[s]
Repetition_Time    = 7.18628096[s]
```



3ao

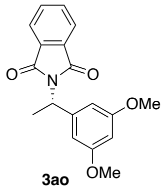


```
Author             = delta
Irr_Freq           = 399.78219838[MHz]
Experiment         = carbon.jxp
Solvent            = CHLOROFORM-D

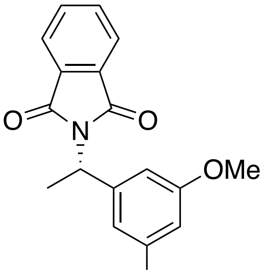
Comment            = single pulse decoupled gat
Data_Format        = 1D COMPLEX
Dim_Size           = 26214
X_Domain           = Carbo
Dim_Units          = [ppm]
Dimensions         = X
Spectrometer       = JNM-ECZ400S/L1

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.03809024[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.96330739[Hz]
X_Sweep            = 31.56565657[kHz]
X_Sweep_Clipped    = 25.25252525[kHz]
Irr_Domain         = Proton
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 256
Total_Scans        = 256

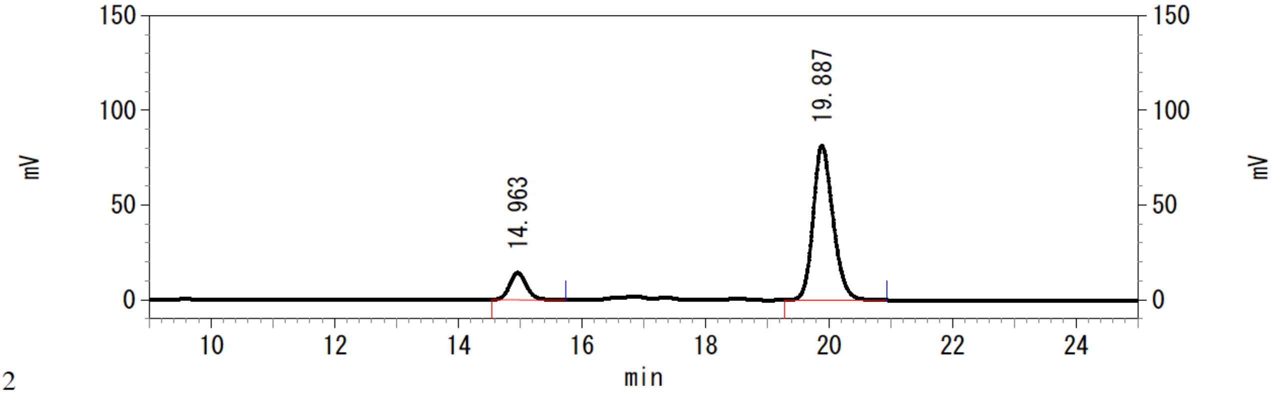
Relaxation_Delay = 2[s]
Recvr_Gain         = 50
Temp_Get           = 22.8[dC]
X_90_Width         = 10.5[us]
X_Acq_Time         = 1.03809024[s]
X_Angle            = 30[deg]
X_Atn              = 3.8[dB]
X_Pulse            = 3.5[us]
Initial_Wait       = 1[s]
Repetition_Time    = 3.03809024[s]
```



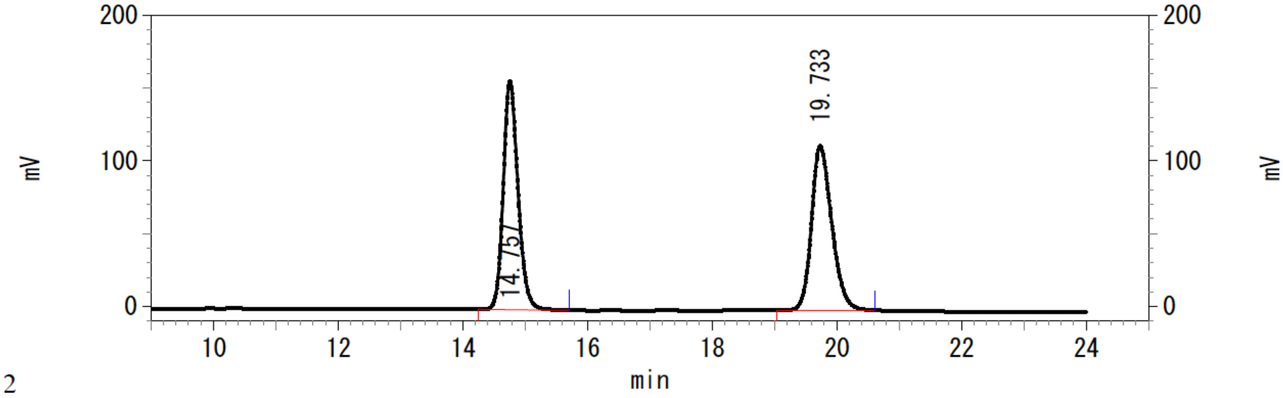
3ao



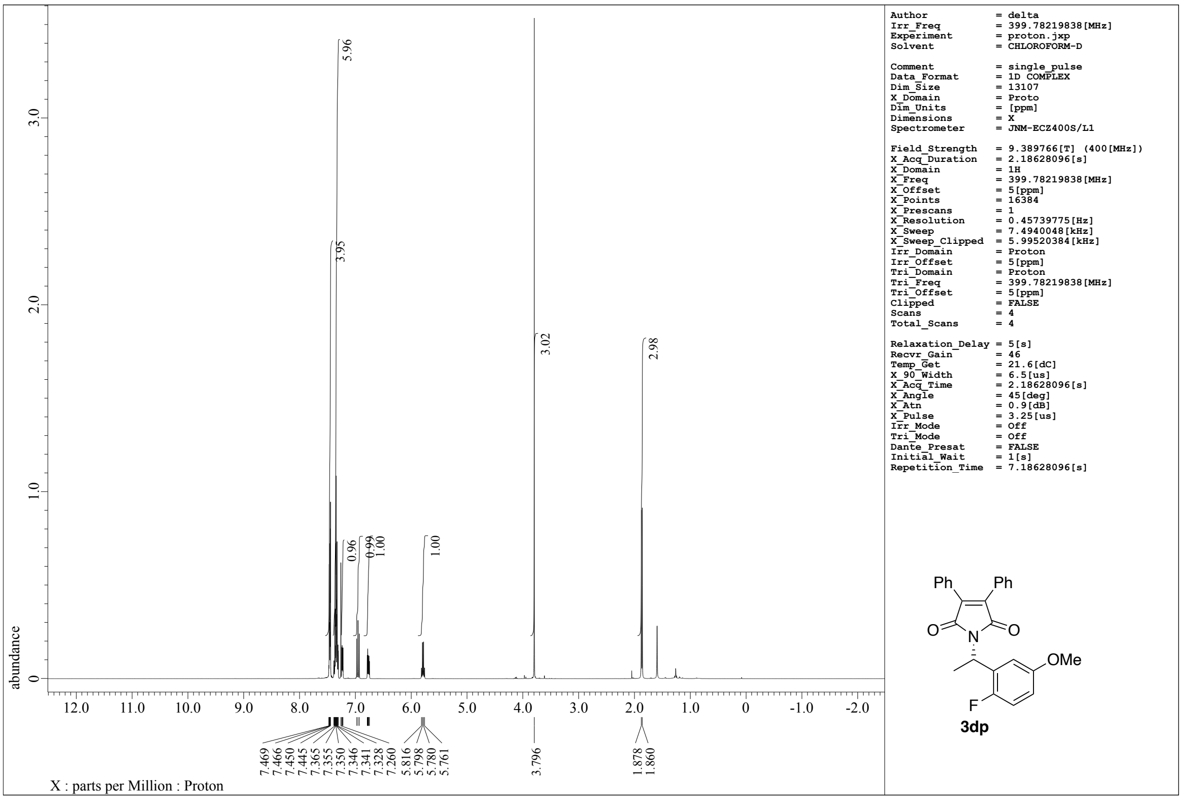
3ao



Pk #	Retention Time	Area	Area Percent
1	14.963	271578	12.944
2	19.887	1826539	87.056



Pk #	Retention Time	Area	Area Percent
1	14.757	2585538	50.293
2	19.733	2555378	49.707



```
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Solvent           = CHLOROFORM-D

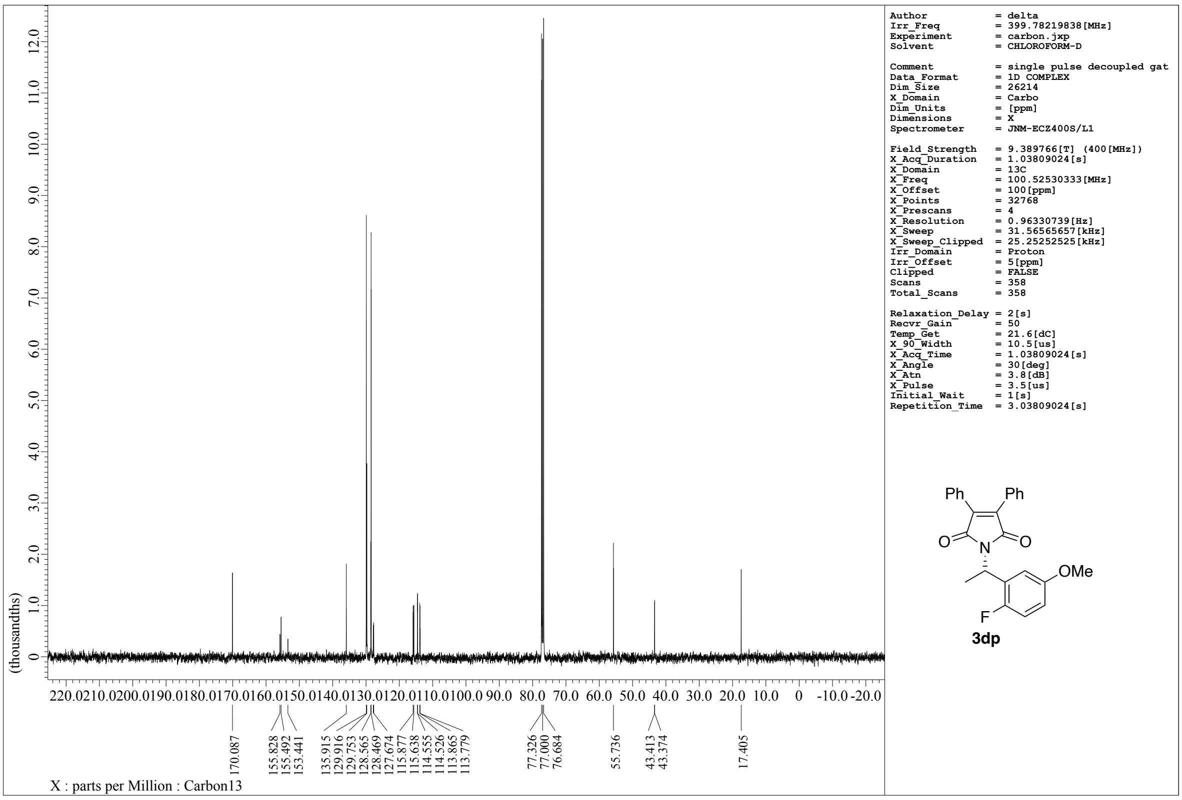
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proto
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18628096[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45739775[Hz]
X_Sweep           = 7.4940048[kHz]
X_Sweep_Clipped   = 5.99520384[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 4
Total_Scans       = 4

Relaxation_Delay = 5[s]
Recvr_Gain        = 46
Temp_Get          = 21.6[dC]
X_90_Width        = 6.5[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 3.25[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time  = 7.18628096[s]
```

3dp

X : parts per Million : Proton



```
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Solvent           = CHLOROFORM-D

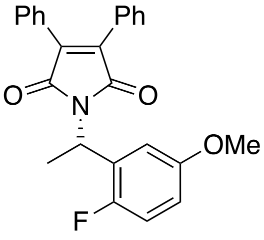
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
X_Domain          = Carbo
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.03809024[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.96330739[Hz]
X_Sweep           = 31.56565657[kHz]
X_Sweep_Clipped   = 25.25252525[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 358
Total_Scans       = 358

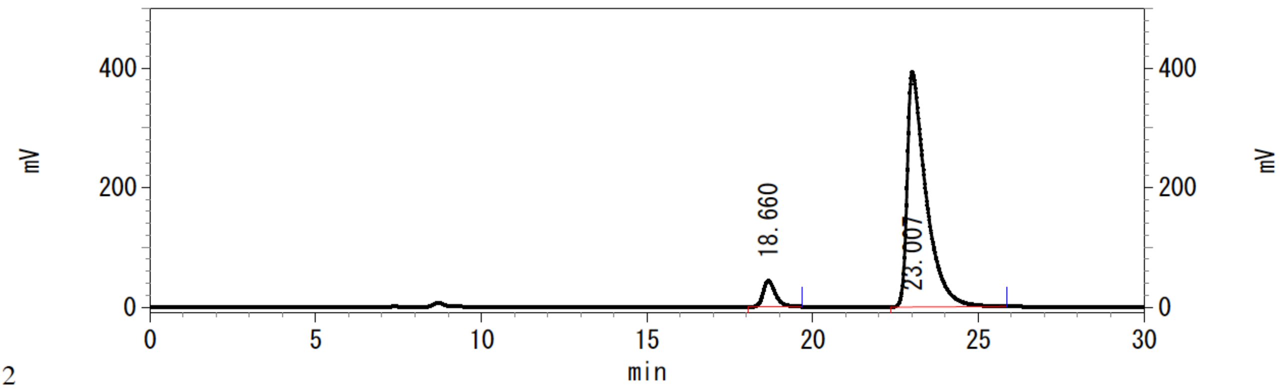
Relaxation_Delay = 2[s]
Recvr_Gain        = 50
Temp_Get          = 21.6[dC]
X_90_Width        = 10.5[us]
X_Acq_Time        = 1.03809024[s]
X_Angle           = 30[deg]
X_Atn             = 3.8[dB]
X_Pulse           = 3.5[us]
Initial_Wait      = 1[s]
Repetition_Time  = 3.03809024[s]
```

3dp

X : parts per Million : Carbon13

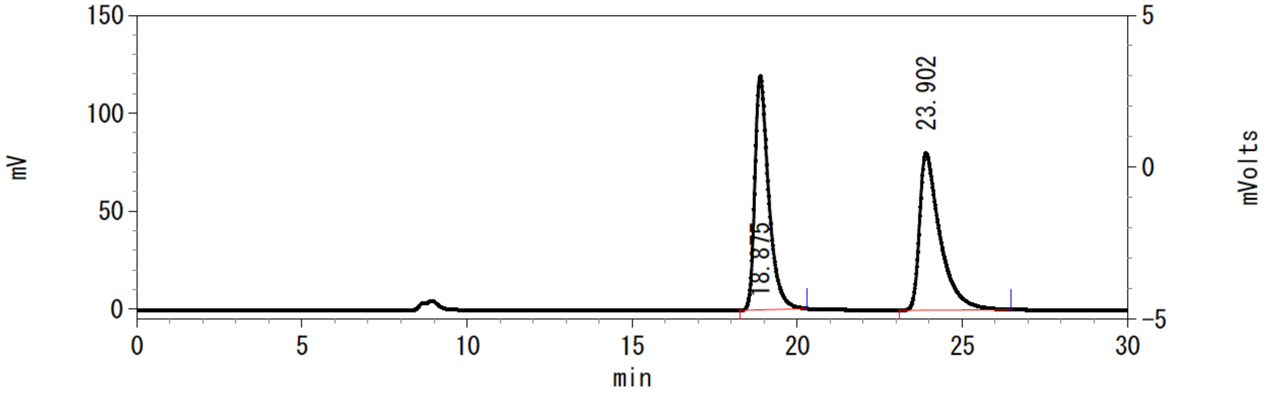


3dp

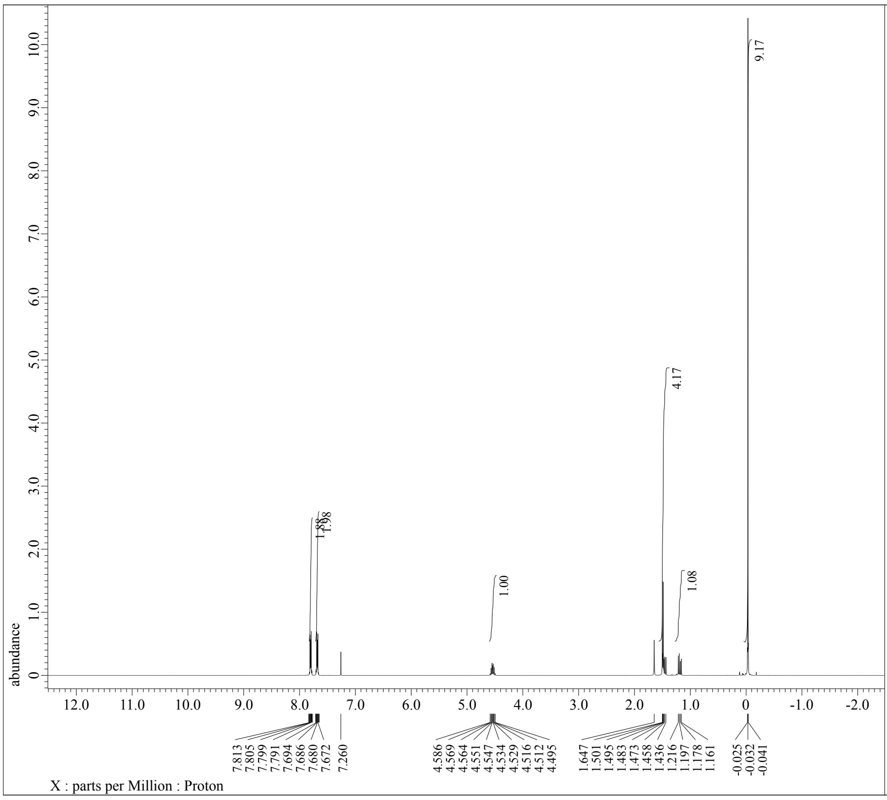


2

Pk #	Retention Time	Area	Area Percent
1	18.660	1115846	6.714
2	23.007	15502725	93.286



Pk #	Retention Time	Area	Area Percent
1	18.875	3474653	50.147
2	23.902	3454237	49.853

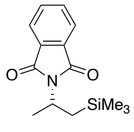


```
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Solvent           = CHLOROFORM-D

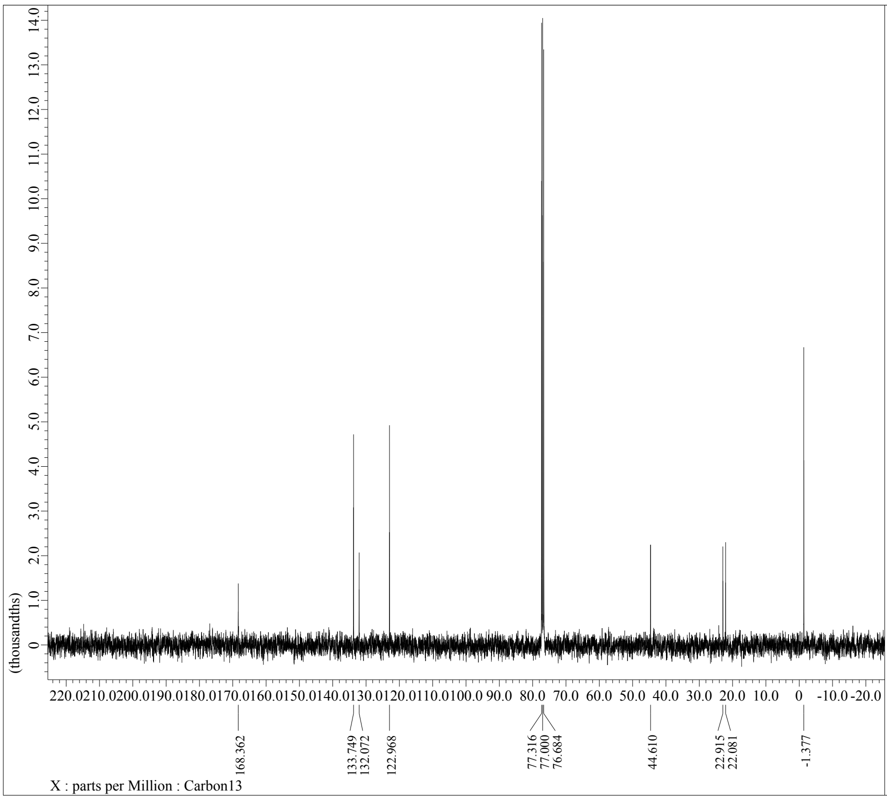
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proto
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18628096[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45739775[Hz]
X_Sweep           = 7.4940048[kHz]
X_Sweep_Clipped   = 5.99520384[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 4
Total_Scans       = 4

Relaxation_Delay  = 5[s]
Recvr_Gain        = 46
Temp_Get          = 22.1[dC]
X_90_Width        = 6.5[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 3.25[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18628096[s]
```



3aq

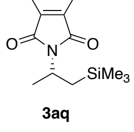


```
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Solvent           = CHLOROFORM-D

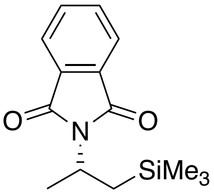
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
X_Domain          = Carbo
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.03809024[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.96330739[Hz]
X_Sweep           = 31.56565657[kHz]
X_Sweep_Clipped   = 25.25252525[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 47
Total_Scans       = 47

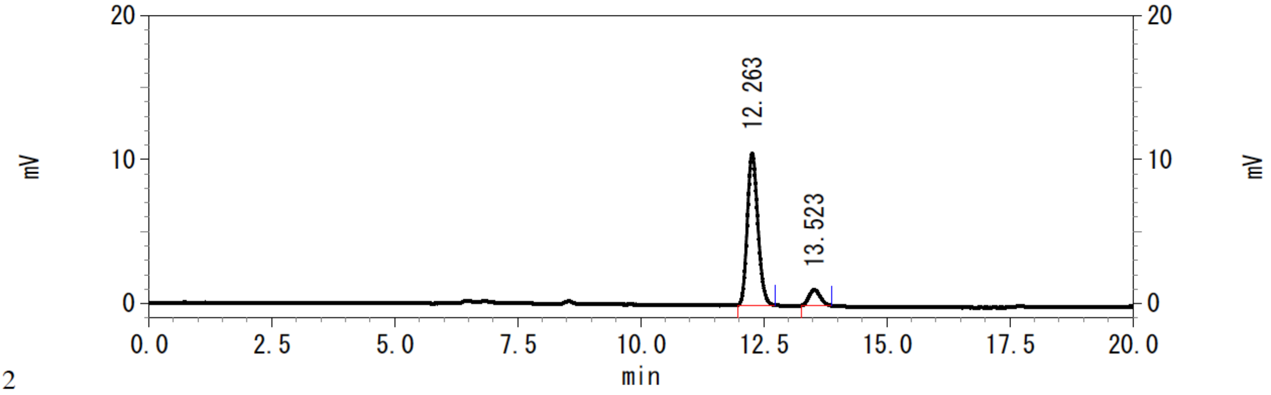
Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 22[dC]
X_90_Width        = 10.5[us]
X_Acq_Time        = 1.03809024[s]
X_Angle           = 30[deg]
X_Atn             = 3.8[dB]
X_Pulse           = 3.5[us]
Initial_Wait      = 1[s]
Repetition_Time   = 3.03809024[s]
```



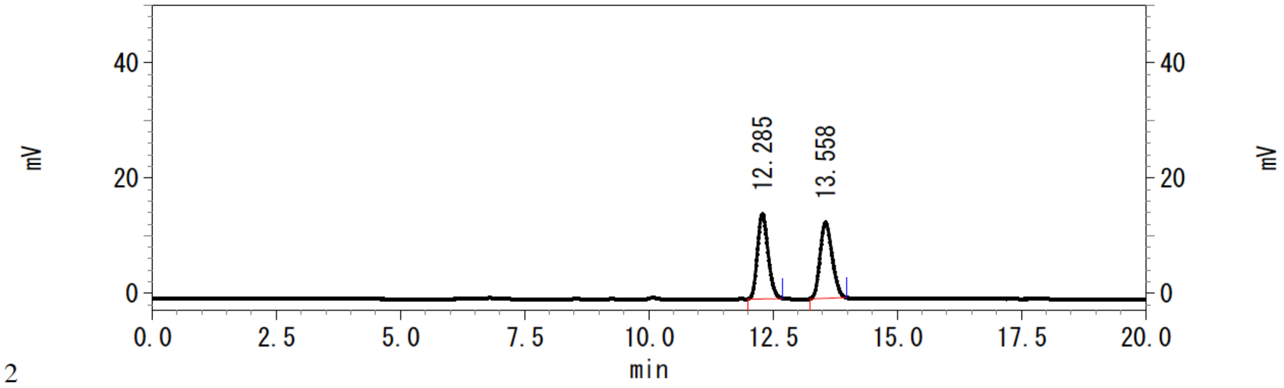
3aq



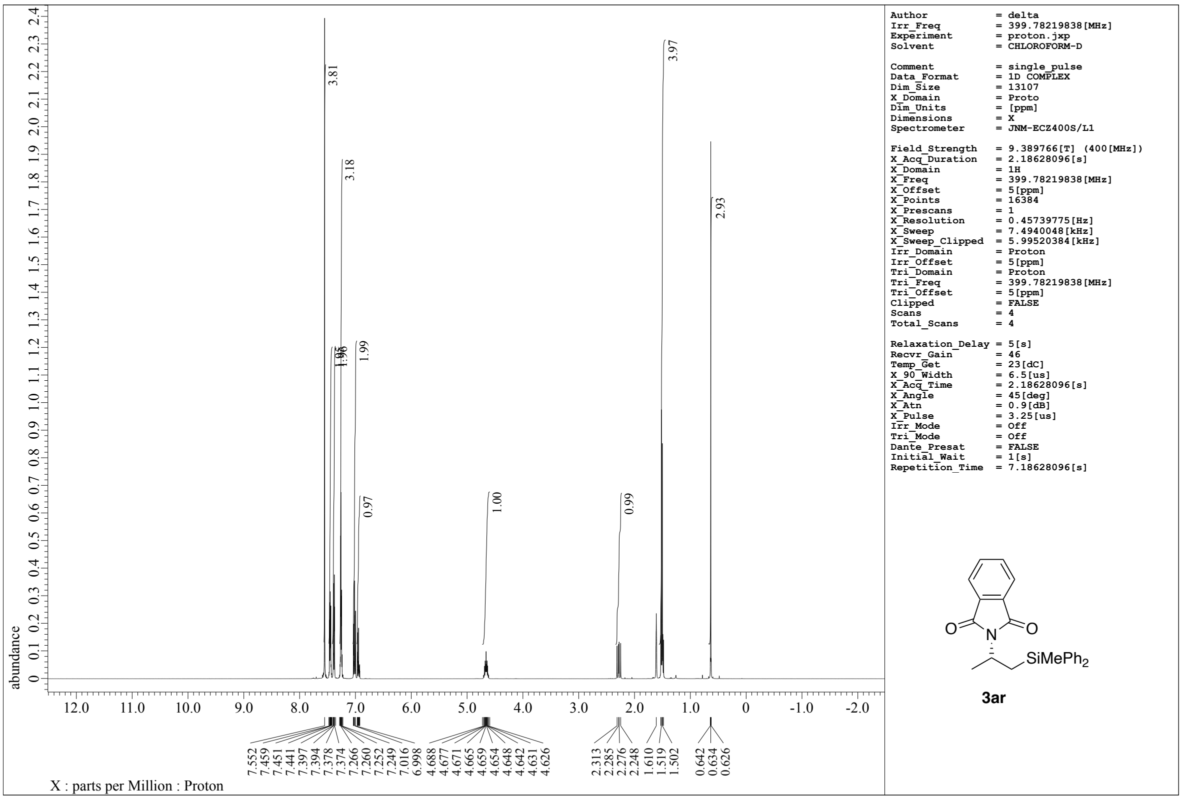
3aq



Pk #	Retention Time	Area	Area Percent
1	12.263	156755	90.035
2	13.523	17349	9.965



Pk #	Retention Time	Area	Area Percent
1	12.285	220166	49.618
2	13.558	223556	50.382

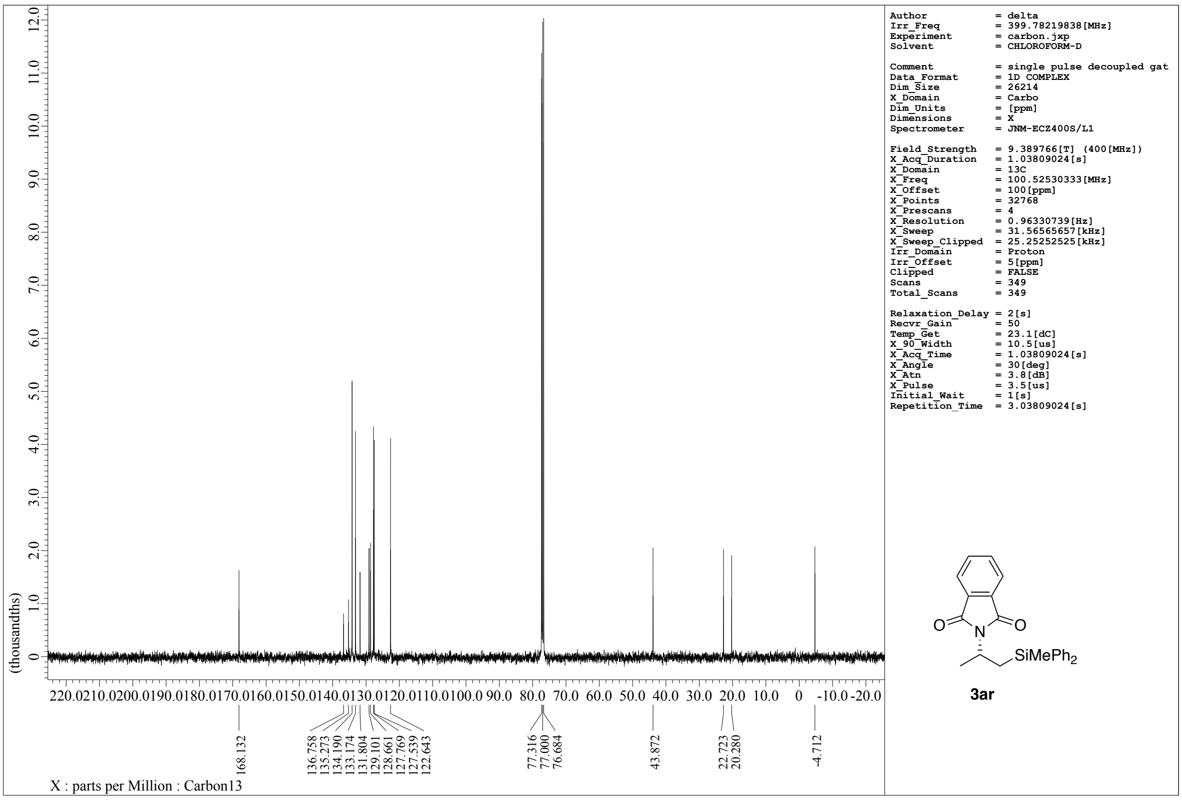


```
Author           = delta
Irr_Freq         = 399.78219838[MHz]
Experiment       = proton.jxp
Solvent          = CHLOROFORM-D

Comment          = single_pulse
Data_Format      = 1D COMPLEX
Dim_Size         = 13107
X_Domain         = Proto
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = JNM-ECZ400S/L1

Field_Strength   = 9.389766[T] (400[MHz])
X_Acq_Duration   = 2.18628096[s]
X_Domain         = 1H
X_Freq           = 399.78219838[MHz]
X_Offset         = 5[ppm]
X_Points         = 16384
X_Prescans       = 1
X_Resolution     = 0.45739775[Hz]
X_Sweep          = 7.4940048[kHz]
X_Sweep_Clipped  = 5.99520384[kHz]
Irr_Domain       = Proton
Irr_Offset       = 5[ppm]
Tri_Domain       = Proton
Tri_Freq         = 399.78219838[MHz]
Tri_Offset       = 5[ppm]
Clipped          = FALSE
Scans            = 4
Total_Scans      = 4

Relaxation_Delay = 5[s]
Recvr_Gain       = 46
Temp_Get         = 23[dC]
X_90_Width       = 6.5[us]
X_Acq_Time       = 2.18628096[s]
X_Angle          = 45[deg]
X_Atn            = 0.9[dB]
X_Pulse          = 3.25[us]
Irr_Mode         = Off
Tri_Mode         = Off
Dante_Presat     = FALSE
Initial_Wait     = 1[s]
Repetition_Time  = 7.18628096[s]
```

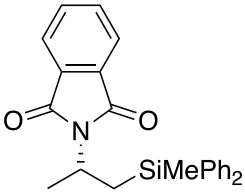


```
Author           = delta
Irr_Freq         = 399.78219838[MHz]
Experiment       = carbon.jxp
Solvent          = CHLOROFORM-D

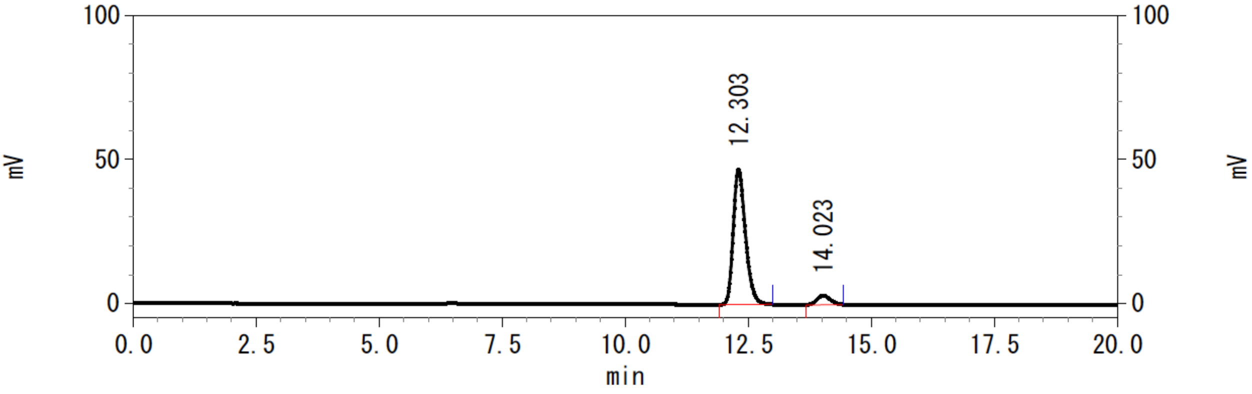
Comment          = single pulse decoupled gat
Data_Format      = 1D COMPLEX
Dim_Size         = 26214
X_Domain         = Carbo
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = JNM-ECZ400S/L1

Field_Strength   = 9.389766[T] (400[MHz])
X_Acq_Duration   = 1.03809024[s]
X_Domain         = 13C
X_Freq           = 100.52530333[MHz]
X_Offset         = 100[ppm]
X_Points         = 32768
X_Prescans       = 4
X_Resolution     = 0.96330739[Hz]
X_Sweep          = 31.56565657[kHz]
X_Sweep_Clipped  = 25.25252525[kHz]
Irr_Domain       = Proton
Irr_Offset       = 5[ppm]
Clipped          = FALSE
Scans            = 349
Total_Scans      = 349

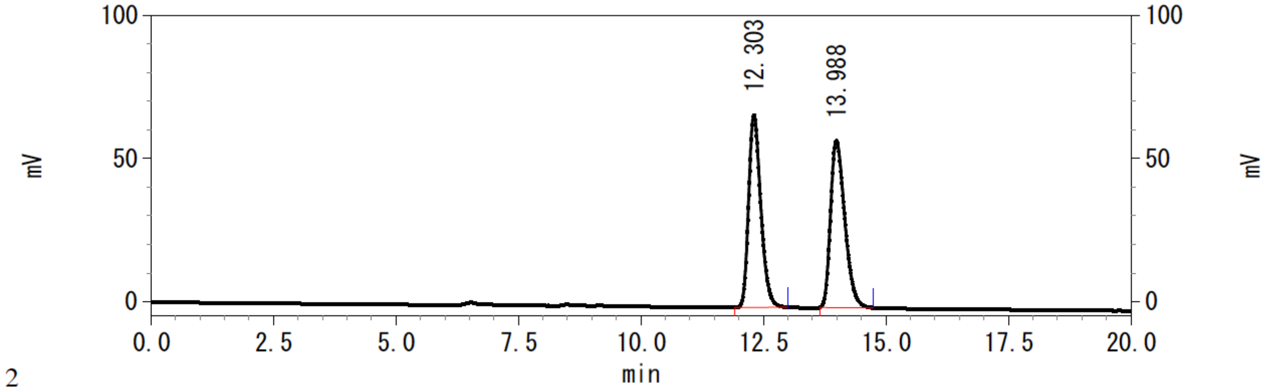
Relaxation_Delay = 2[s]
Recvr_Gain       = 50
Temp_Get         = 23.1[dC]
X_90_Width       = 10.5[us]
X_Acq_Time       = 1.03809024[s]
X_Angle          = 30[deg]
X_Atn            = 3.8[dB]
X_Pulse          = 3.5[us]
Initial_Wait     = 1[s]
Repetition_Time  = 3.03809024[s]
```



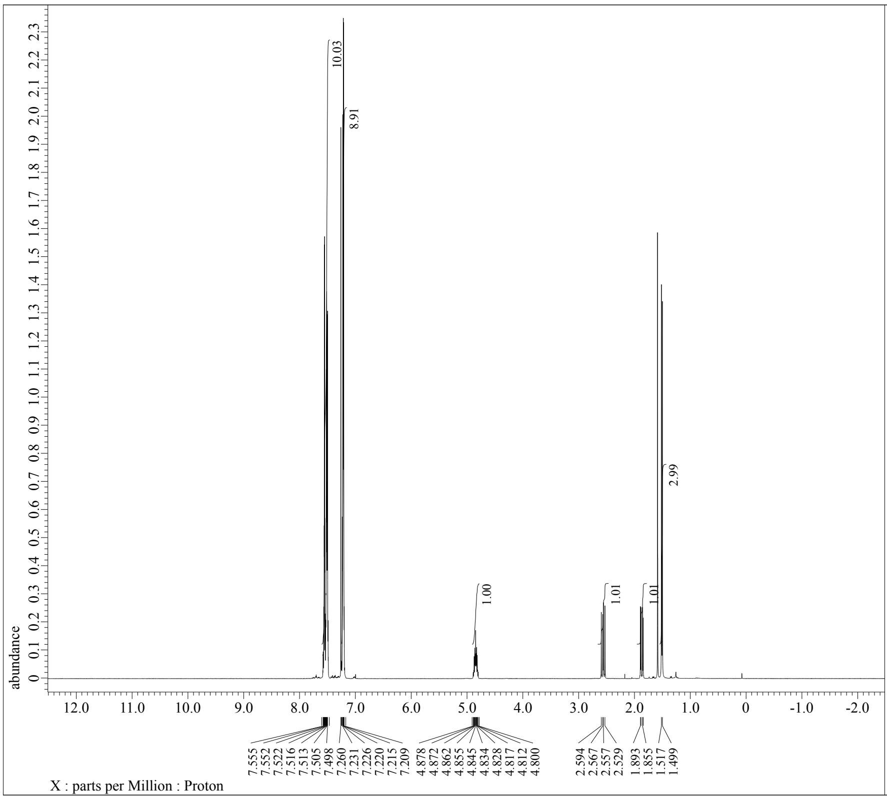
3ar



Pk #	Retention Time	Area	Area Percent
1	12.303	805172	93.023
2	14.023	60386	6.977



Pk #	Retention Time	Area	Area Percent
1	12.303	1159921	49.550
2	13.988	1180994	50.450

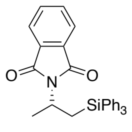


```
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Solvent           = CHLOROFORM-D

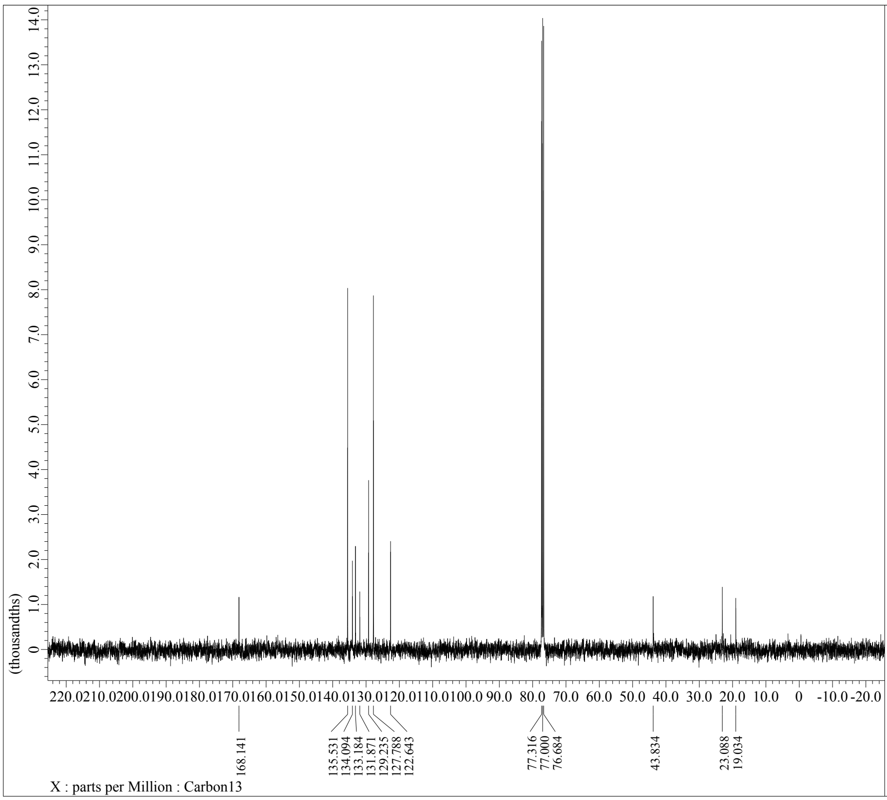
Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proto
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18628096[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45739775[Hz]
X_Sweep           = 7.4940048[kHz]
X_Sweep_Clipped   = 5.99520384[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 4
Total_Scans       = 4

Relaxation_Delay = 5[s]
Recvr_Gain        = 56
Temp_Get          = 22.2[dC]
X_90_Width        = 6.5[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 3.25[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18628096[s]
```



3as

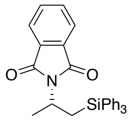


```
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = carbon.jxp
Solvent           = CHLOROFORM-D

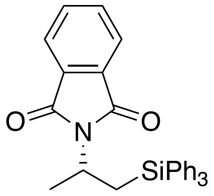
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
X_Domain          = Carbo
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 1.03809024[s]
X_Domain          = 13C
X_Freq            = 100.52530333[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.96330739[Hz]
X_Sweep           = 31.56565657[kHz]
X_Sweep_Clipped   = 25.25252525[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 79
Total_Scans       = 79

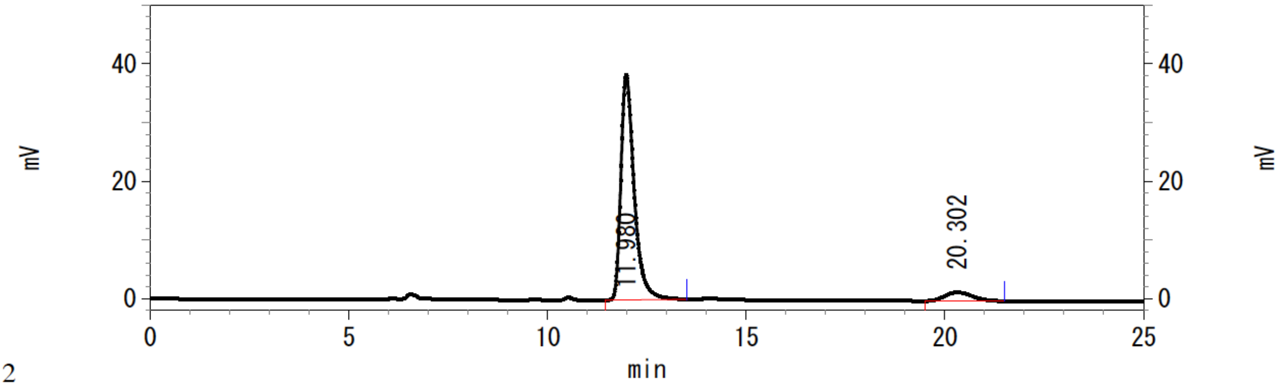
Relaxation_Delay = 2[s]
Recvr_Gain        = 50
Temp_Get          = 22.4[dC]
X_90_Width        = 10.5[us]
X_Acq_Time        = 1.03809024[s]
X_Angle           = 30[deg]
X_Atn             = 3.8[dB]
X_Pulse           = 3.5[us]
Initial_Wait      = 1[s]
Repetition_Time   = 3.03809024[s]
```



3as

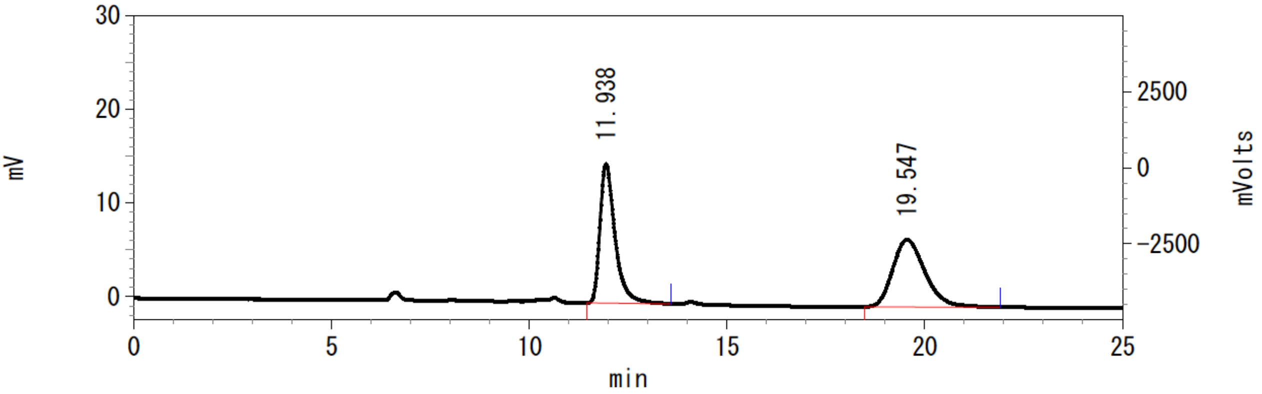


3as

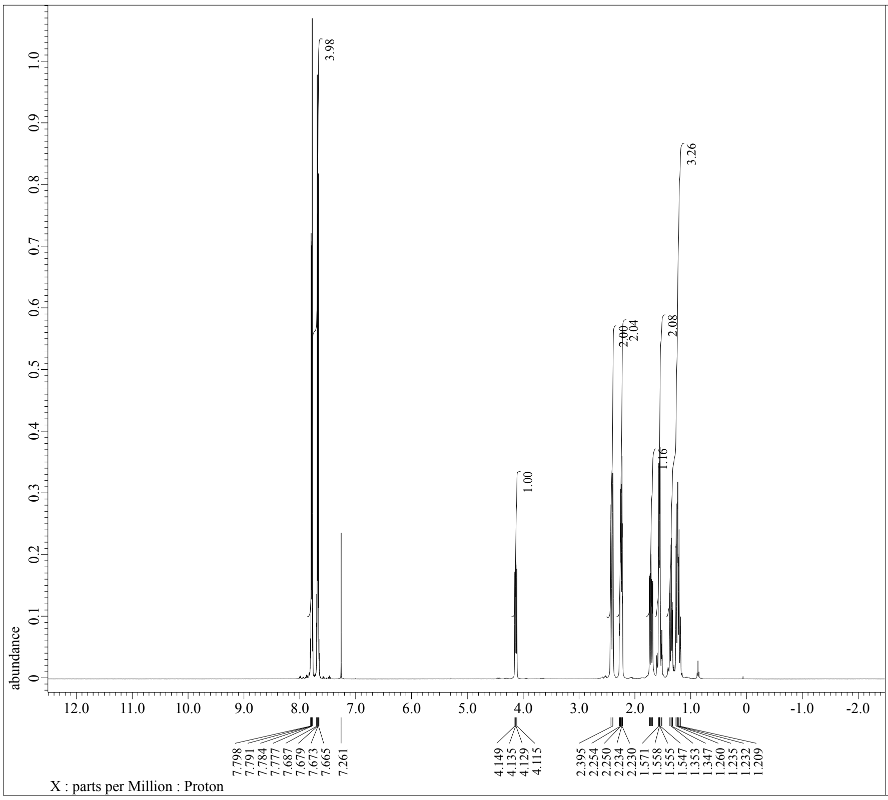


2

Pk #	Retention Time	Area	Area Percent
1	11.980	890054	92.531
2	20.302	71845	7.469



Pk #	Retention Time	Area	Area Percent
1	11.938	394973	49.203
2	19.547	407767	50.797

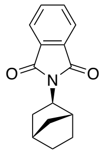


```
Author           = delta
Irr_Freq         = 399.78219838[MHz]
Experiment       = proton.jxp
Solvent          = CHLOROFORM-D

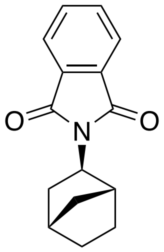
Comment          = single_pulse
Data_Format      = 1D COMPLEX
Dim_Size         = 13107
X_Domain         = Proto
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = JNM-ECZ400S/L1

Field_Strength   = 9.389766[T] (400[MHz])
X_Acq_Duration   = 2.18628096[s]
X_Domain         = 1H
X_Freq           = 399.78219838[MHz]
X_Offset         = 5[ppm]
X_Points         = 16384
X_Prescans       = 1
X_Resolution     = 0.45739775[Hz]
X_Sweep          = 7.4940048[kHz]
X_Sweep_Clipped  = 5.99520384[kHz]
Irr_Domain       = Proton
Irr_Offset       = 5[ppm]
Tri_Domain       = Proton
Tri_Freq         = 399.78219838[MHz]
Tri_Offset       = 5[ppm]
Clipped          = FALSE
Scans            = 8
Total_Scans      = 8

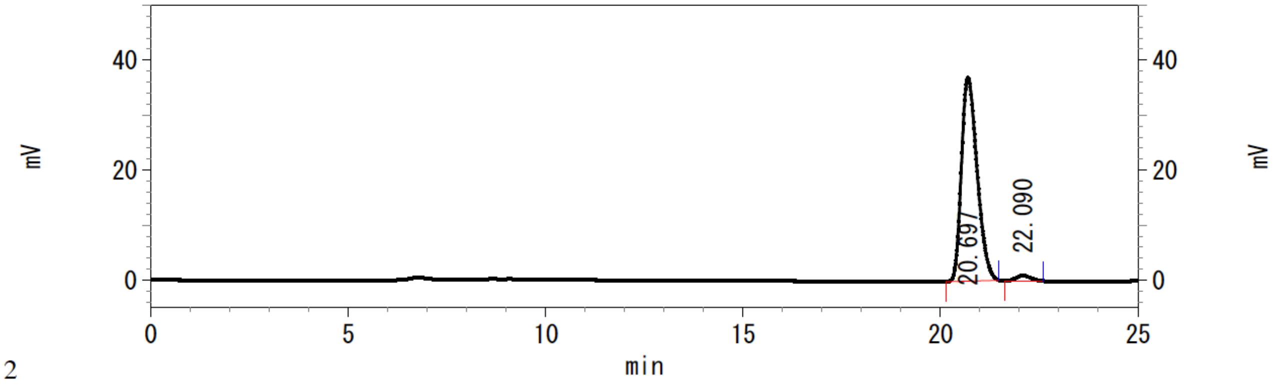
Relaxation_Delay = 5[s]
Recvr_Gain       = 46
Temp_Get         = 18.2[dC]
X_90_Width       = 6.3[us]
X_Acq_Time       = 2.18628096[s]
X_Angle          = 45[deg]
X_Atn            = 0.9[dB]
X_Pulse          = 3.15[us]
Irr_Mode         = Off
Tri_Mode         = Off
Dante_Presat     = FALSE
Initial_Wait     = 1[s]
Repetition_Time  = 7.18628096[s]
```



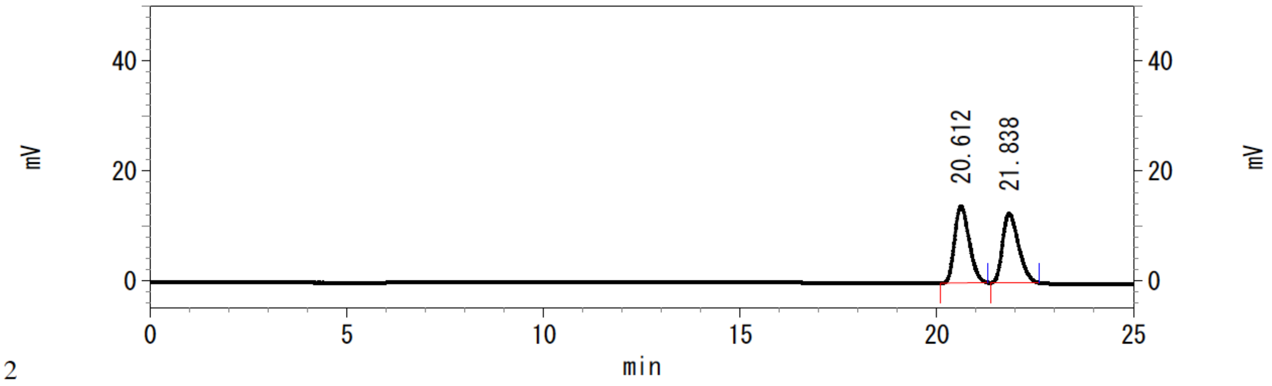
3at



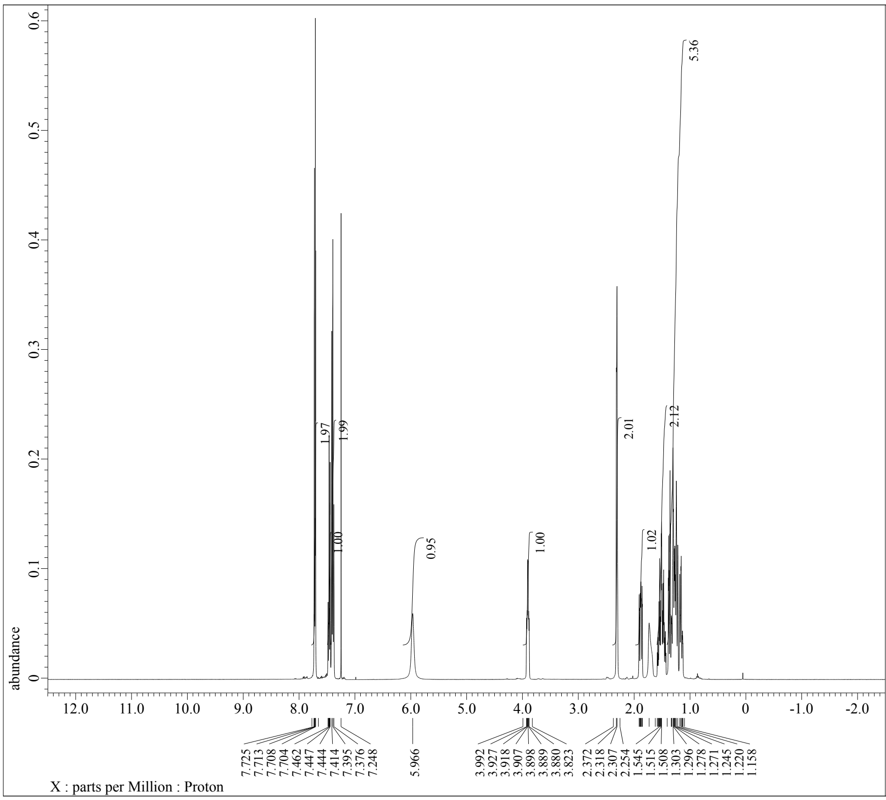
3at



Pk #	Retention Time	Area	Area Percent
1	20.697	1020326	97.553
2	22.090	25592	2.447



Pk #	Retention Time	Area	Area Percent
1	20.612	360321	50.296
2	21.838	356075	49.704

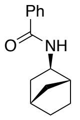


```
Author           = delta
Irr_Freq         = 399.78219838[MHz]
Experiment       = proton.jxp
Solvent          = CHLOROFORM-D

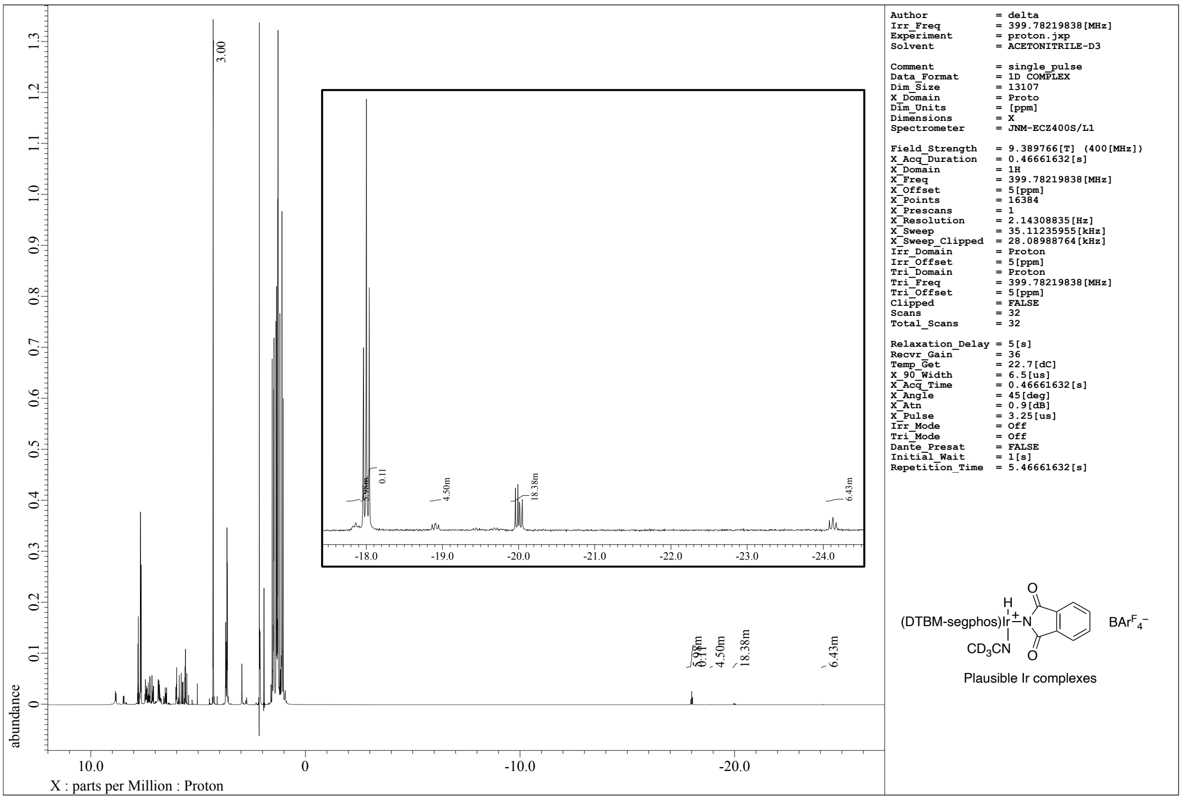
Comment          = single_pulse
Data_Format      = 1D COMPLEX
Dim_Size         = 13107
X_Domain         = Proto
Dim_Units        = [ppm]
Dimensions       = X
Spectrometer     = JNM-ECZ400S/L1

Field_Strength   = 9.389766[T] (400[MHz])
X_Acq_Duration   = 2.18628096[s]
X_Domain         = 1H
X_Freq           = 399.78219838[MHz]
X_Offset         = 5[ppm]
X_Points         = 16384
X_Prescans       = 1
X_Resolution     = 0.45739775[Hz]
X_Sweep          = 7.4940048[kHz]
X_Sweep_Clipped  = 5.99520384[kHz]
Irr_Domain       = Proton
Irr_Offset       = 5[ppm]
Tri_Domain       = Proton
Tri_Freq         = 399.78219838[MHz]
Tri_Offset       = 5[ppm]
Clipped          = FALSE
Scans            = 16
Total_Scans      = 16

Relaxation_Delay = 5[s]
Recvr_Gain       = 46
Temp_Get         = 21.7[dC]
X_90_Width       = 6.5[us]
X_Acq_Time       = 2.18628096[s]
X_Angle          = 45[deg]
X_Atn            = 0.9[dB]
X_Pulse          = 3.25[us]
Irr_Mode         = Off
Tri_Mode         = Off
Dante_Presat     = FALSE
Initial_Wait     = 1[s]
Repetition_Time  = 7.18628096[s]
```



4



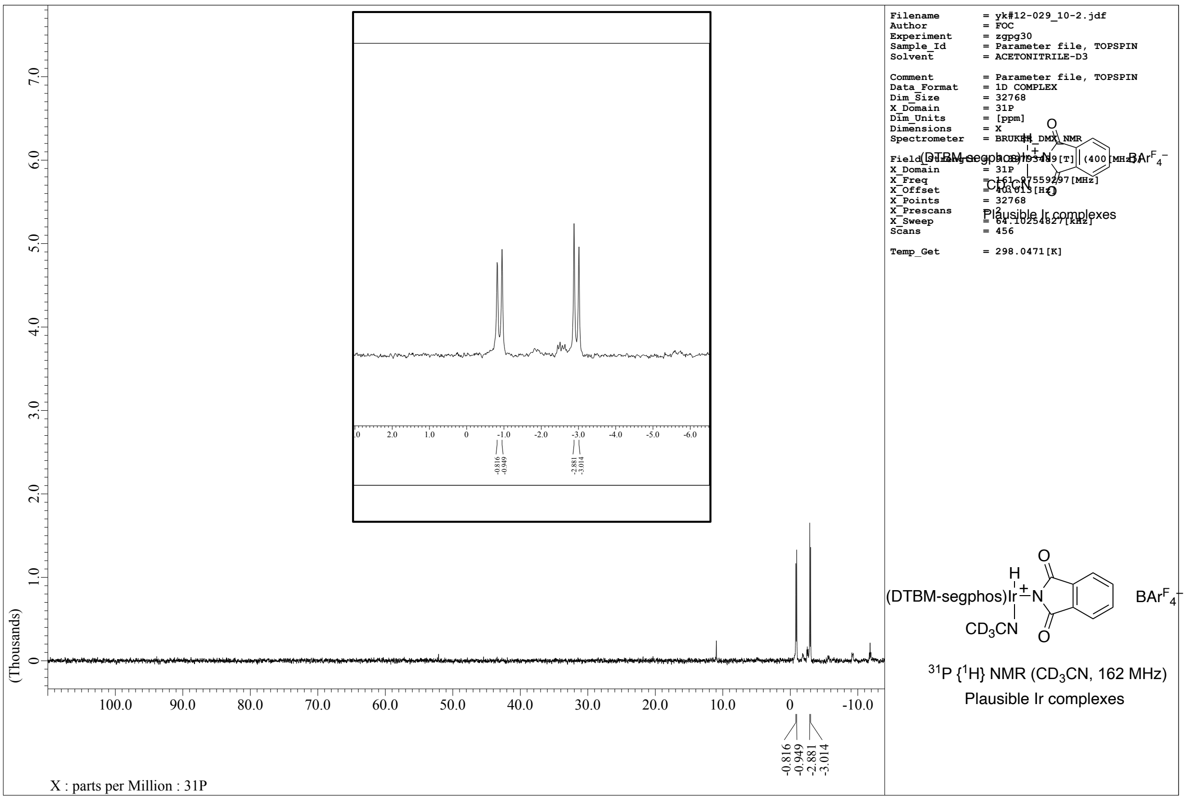
```
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Solvent           = ACETONITRILE-D3

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proto
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 0.46661632[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 2.14308835[Hz]
X_Sweep           = 35.11235955[kHz]
X_Sweep_Clipped   = 28.08988764[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 32
Total_Scans       = 32

Relaxation_Delay = 5[s]
Recvr_Gain        = 36
Temp_Get          = 22.7[dC]
X_90_Width        = 6.5[us]
X_Acq_Time        = 0.46661632[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 3.25[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 5.46661632[s]
```

Plausible Ir complexes



```
Filename          = yk#12-029_10-2.jdf
Author            = FOC
Experiment        = zgpg30
Sample_Id         = Parameter file, TOPSPIN
Solvent           = ACETONITRILE-D3

Comment           = Parameter file, TOPSPIN
Data_Format       = 1D COMPLEX
Dim_Size          = 32768
X_Domain          = 31P
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = BRUKER_DMX_NMR

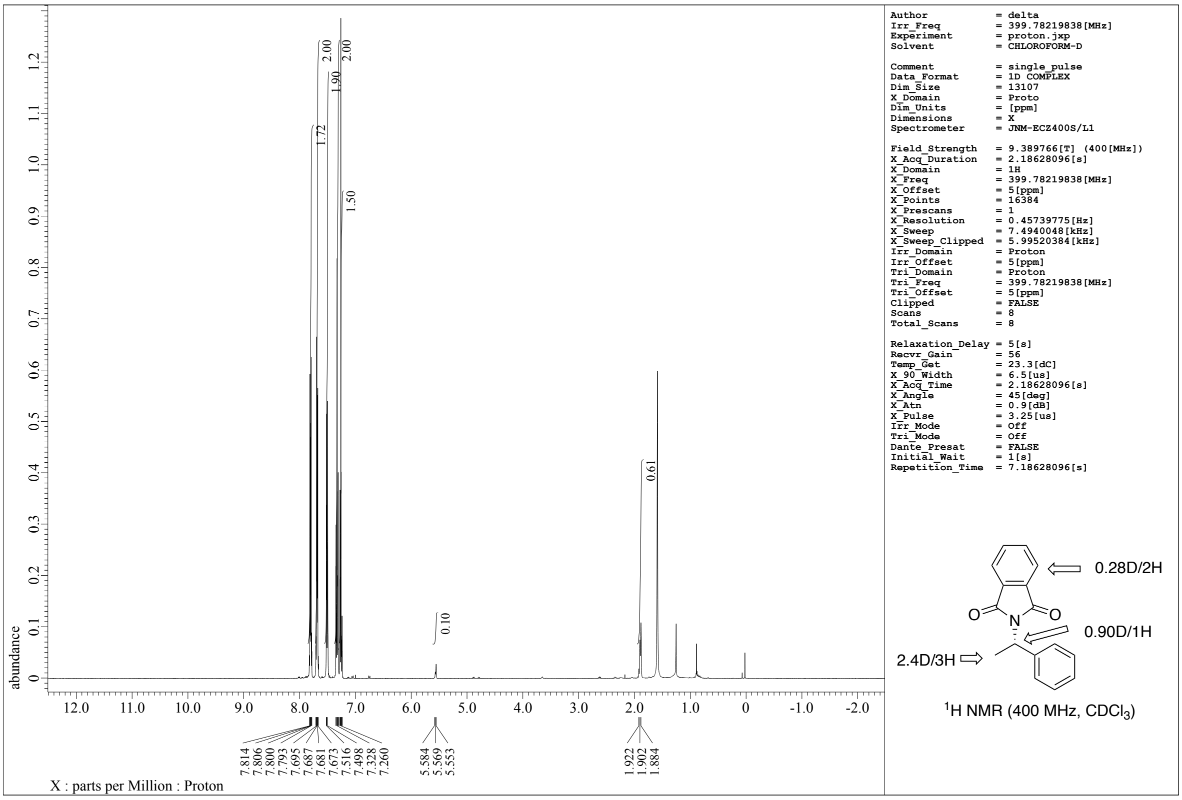
Field_Strength    = 9.39793489[T] (400[MHz])
X_Domain          = 31P
X_Freq            = 161.97559297[MHz]
X_Offset          = 403.013[Hz]
X_Points          = 32768
X_Prescans        = 2
X_Sweep           = 64.10254827[kHz]
Scans             = 456

Temp_Get          = 298.0471[K]
```

Plausible Ir complexes

^{31}P {^{1}H} NMR (CD_3CN, 162 MHz)

Plausible Ir complexes



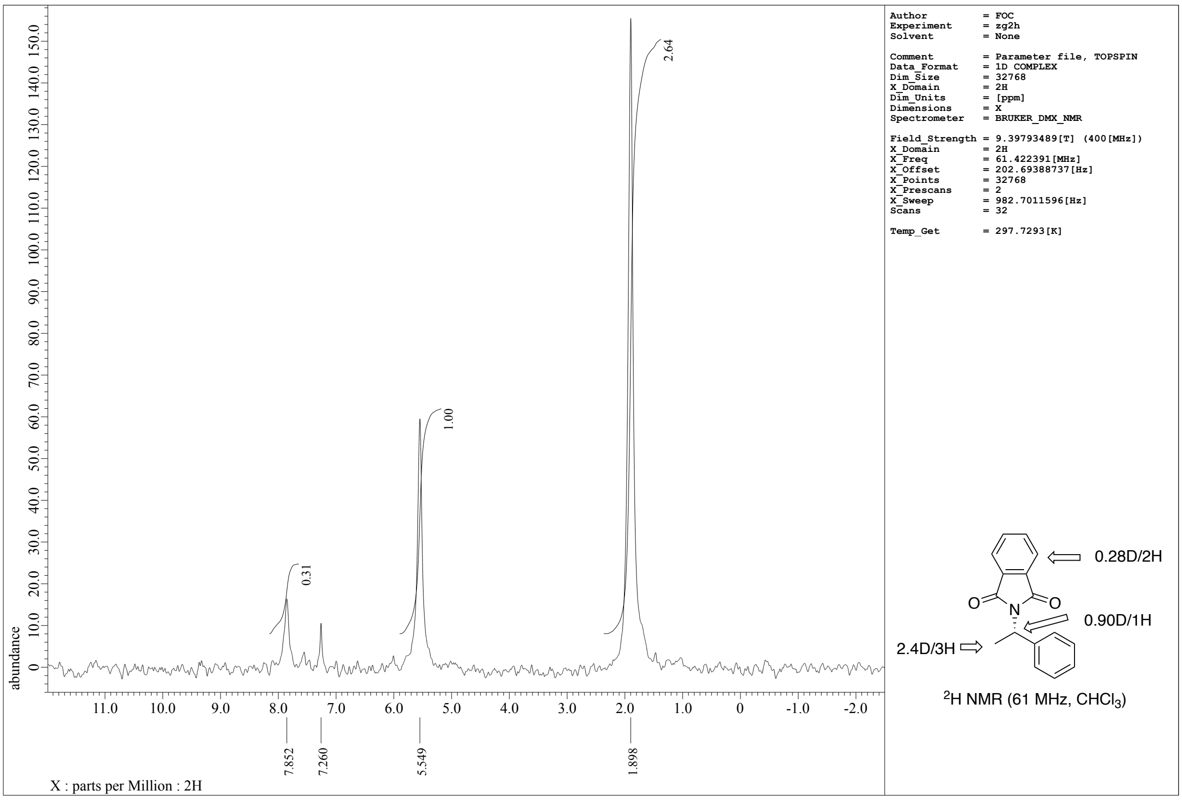
```
Author            = delta
Irr_Freq          = 399.78219838[MHz]
Experiment        = proton.jxp
Solvent           = CHLOROFORM-D

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
X_Domain          = Proto
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = JNM-ECZ400S/L1

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18628096[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45739775[Hz]
X_Sweep           = 7.4940048[kHz]
X_Sweep_Clipped   = 5.99520384[kHz]
Irr_Domain        = Proton
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 399.78219838[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        = 6.5[us]
X_Acq_Time        = 2.18628096[s]
X_Angle           = 45[deg]
X_Atn             = 0.9[dB]
X_Pulse           = 3.25[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18628096[s]
```

^{1}H NMR (400 MHz, $CDCl_3$)



```
Author            = FOC
Experiment        = zg2h
Solvent           = None

Comment           = Parameter file, TOPSPIN
Data_Format       = 1D COMPLEX
Dim_Size          = 32768
X_Domain          = 2H
Dim_Units         = [ppm]
Dimensions        = X
Spectrometer      = BRUKER_DMX_NMR

Field_Strength = 9.39793489[T] (400[MHz])
X_Domain          = 2H
X_Freq            = 61.422391[MHz]
X_Offset          = 202.69388737[Hz]
X_Points          = 32768
X_Prescans        = 2
X_Sweep           = 982.7011596[Hz]
Scans             = 32

Temp_Get          = 297.7293[K]
```

^{2}H NMR (61 MHz, $CHCl_3$)