Palladium-Catalyzed Synthesis of α-Diimines from Triarylbismuthines and Isocyanides

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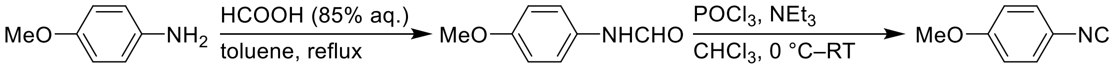
Supporting Information

1. General Methods and Instrumentation

Triphenylbismuthine (**1a**) was obtained from commercial supply. The other bismuthines were synthesized according to the literature. All aliphatic isocyanides and 2,6-xylylisocyanide (**2g**) were obtained from commercial supplies. The other isocyanides were synthesized according to the literatures. All solvents were distilled before use. ^{1}H NMR spectra were recorded on JEOL JNM-ECX400 (400 MHz) FT NMR or JEOL JNM-ECS400 (400 MHz) FT NMR in $CDCl_3$ with Me_4Si as an internal standard. ^{13}C NMR spectra were recorded on JEOL JNM-ECX400 (100 MHz) FT NMR or JEOL JNM-ECS400 (100 MHz) FT NMR in $CDCl_3$. ^{19}F NMR spectra were recorded on JEOL JNM-ECX400 (376 MHz) FT NMR or JEOL JNM-ECS400 (376 MHz) FT NMR in $CDCl_3$ with $CFCl_3$ as an external standard. High resolution mass spectra were obtained on JEOL JMS700. GC-MS spectra were obtained on SHIMADZU GCMS-QP5000. HPLC (recycle GPC) method for isolation was performed on JAPAN ANALYTICAL INDUSTRY LC-908 with JAIGEL-2HH (polystyrene-based column).

2. Synthesis of Isocyanides

• Synthesis of 4-methoxyphenylisocyanide (2f)

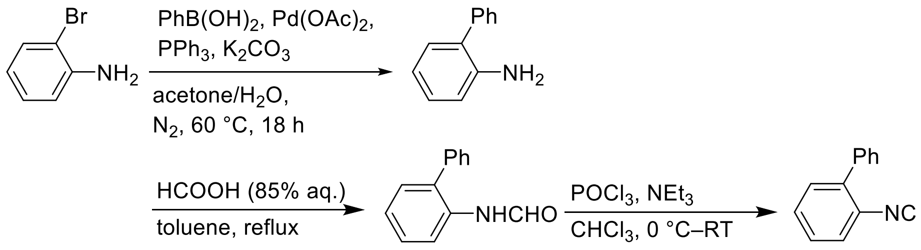


4-methoxyphenylisocyanide (**2f**) was synthesized by minor modified method of the literature.[1]

A solution of 4-methoxyaniline (1.85 g, 15 mmol) and formic acid (85% aq.) (1.0 mL, 22.5 mmol) in toluene (15 mL) is refluxed under N_2 atmosphere. The reaction was monitored by TLC. After the reaction, volatile materials were evaporated under reduced pressure and crude product was purified recrystallization from $CHCl_3$/hexane. 4-Methoxyformanilide was obtained in 97% yield as white to pale blue crystal.

In 100 mL two-necked flask after heating for drying, 4-methoxyformanilide (1.51 g, 10 mmol) was dissolved in $CHCl_3$ (15 mL) and NEt_3 (4.2 mL, 30 mmol) under N_2 atmosphere and cooled to 0 °C by ice bath. To the mixture, phosphoryl chloride (1.2 mL, 12 mmol, 5 mL $CHCl_3$ solution) was added dropwise slowly. After stirring for 1 h, the reaction temperature was allowed to RT and the mixture was stirred for more 1 h. After the reaction was completed, the resulting mixture was cooled to 0 °C and aqueous saturated solution of sodium carbonate (10 mL) was added dropwise to quench the reaction. After vigorous stirring for 1 h, more water (ca. 20 mL) was added and organic layer was separated. Aqueous layer was extracted by $CHCl_3$ (20 mL) twice. Combined organic layer was dried over sodium sulfate, and solvent was removed by evaporation. The residue was purified by silica gel column chromatography (eluent: hexane/AcOEt = 9/1). 4-methoxyphenylisocyanide (**1e**) was obtained in 90% yield (1.20 g) as white smelling solid but rapidly change to green liquid.

• Synthesis of 2-biphenylisocyanide (2h)



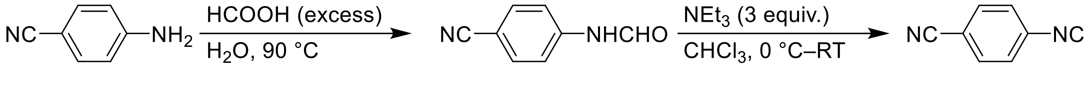
2-biphenylisocyanide (**2h**) was synthesized by minor modified method in the literature.[2]

2-Bromoaniline (1.38 g, 8.0 mmol), phenylboronic acid (1.17 g, 9.6 mmol) and potassium carbonate (2.21 g, 16 mmol) were placed in 50 mL two-necked flask and dissolved in acetone/H_2O = 2/1 solution (10 mL) under N_2 atmosphere. After stirred for 1 h at RT, palladium acetate (90 mg, 0.4 mmol) and triphenylphosphine (210 mg, 0.8 mmol) were added to the solution. The mixture was stirred at 60 °C for 18 h. After the reaction, water was added and extracted by AcOEt twice. Combined organic layer was dried over magnesium sulfate, and solvent was removed by evaporation. The residue was purified by silica gel column chromatography (eluent: hexane/AcOEt = 5/1). 2-biphenylaniline was obtained in 79% yield (1.07 g) as white solid.

In 50 mL two-necked flask, a solution of 2-biphenylaniline (0.85 g, 5 mmol) and formic acid (85% aq.) (1.0 mL, 22.5 mmol) in toluene (5 mL) is refluxed under N_2 atmosphere. The reaction was monitored by TLC. After the reaction, volatile materials were evaporated in reduced pressure. More purification was not operated. 2-phenylformanilide was obtained in 99% yield (0.98 g) as white solid.

In 100 mL two-necked flask after heating for drying, 2-phenylformanilide (0.98 g, 5 mmol) was dissolved in $CHCl_3$ (30 mL) and NEt_3 (3 mL, 20 mmol) under N_2 atmosphere and cooled to 0 °C by ice bath. To the mixture, phosphoryl chloride (1.2 mL, 12 mmol, 5 mL $CHCl_3$ solution) was added dropwise slowly. After stirring 1 h, the reaction temperature was allowed to RT and mixture was stirrer for more 1 h. After the reaction was completed, the resulting mixture was cooled to 0 °C and aqueous saturated solution of sodium carbonate (5 mL) was added dropwise to quench the reaction. After vigorous stirring for 1 h, more water (ca. 20 mL) was added and organic layer was separated. Aqueous layer was extracted by $CHCl_3$ (20 mL) twice. Combined organic layer was dried over sodium sulfate, and solvent was removed by evaporation. The residue was purified by silica gel column chromatography (eluent: hexane/AcOEt = 10/1). 2-biphenylisocyanide (**2h**) was obtained in 96% yield (0.85 g) as green liquid.

• Synthesis of 4-cyanophenylisocyanide (2i)



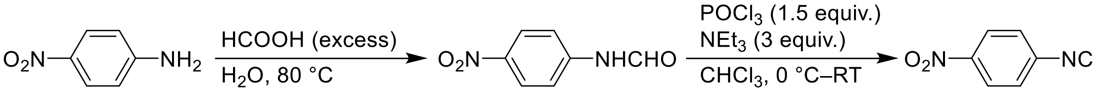
4-cyanophenylisocyanide (**2i**) was synthesized by minor modified method in the literatures.[3]

In 50 mL 2-neck flask, a suspension of 4-cyanoaniline (2.36 g, 20 mmol) and formic acid (85% aq.) (10 mL, 67.5 mmol) in water (5 mL) is stirred at 90 °C under N_2 atmosphere overnight. The reaction was monitored by

TLC. After the reaction, all volatile materials were removed in reduced pressure. The residue was recrystallized from water. Obtained white crystal was corrected by filtration under reduced pressure. 4-cyanoformanilide was obtained in 43% yield (1.26 g).

In 100 mL two-necked flask after heating for drying, 4-cyanoformanilide (1.00 g, 6.8 mmol) was dissolved in $CHCl_3$ (30 mL) and NEt_3 (3.0 mL, 20 mmol) under N_2 atmosphere and cooled to 0 °C. After stirred for 3 h at RT, phosphoryl chloride (0.69 mL, 7.4 mmol, 10 mL $CHCl_3$ solution) was added dropwise slowly. After 2 h, the reaction temperature was allowed to RT and mixture was stirred for more 2 h. After the reaction was completed, the resulting mixture was cooled to 0 °C and aqueous saturated solution of sodium carbonate (10 mL) was added dropwise to quench the reaction. After vigorous stirring for 1 h, more water (ca. 20 mL) was added and organic layer was separated. Aqueous layer was extracted by $CHCl_3$ (20 mL) twice. Combined organic layer was dried over sodium sulfate, and solvent was removed by evaporation. The residue was purified by silica gel column chromatography (eluent: hexane/AcOEt = 5/2). 4-cyanophenylisocyanide (**2i**) was obtained in 67% yield (0.584 g) as white solid.

• Synthesis of 4-nitrophenylisocyanide (2j)

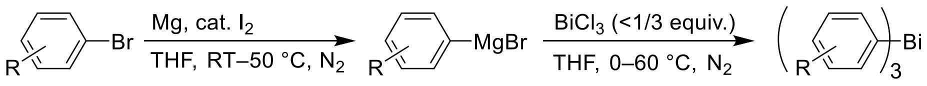


4-nitrophenylisocyanide (**2j**) was synthesized by minor modified method in the literatures.[3]

In 50 mL two-necked flask, a suspension of 4-nitroaniline (2.07 g, 15 mmol) and formic acid (85% aq.) (3.0 mL, 67.5 mmol) in water (3 mL) is stirred at 80 °C under N_2 atmosphere overnight. The reaction was monitored by TLC. After the reaction, AcOEt (30 mL) was poured. Organic layer was washed by sat. $NaHCO_3$ aqueous solution, followed by brine. Organic layer was separated and dried over sodium sulfate. Solvent was removed under reduced pressure, and obtained solid was washed by methanol. 4-nitroformanilide was obtained in 88% yield as yellow solid.

In 100 mL two-necked flask after heating for drying, 4-nitroformanilide (1.02 g, 6 mmol) was dissolved in $CHCl_3$ (60 mL) and NEt_3 (2.6 mL, 18 mmol) under N_2 atmosphere and cooled to 0 °C. After stirred for 3 h at RT, phosphoryl chloride (0.87 mL, 9 mmol, 15 mL $CHCl_3$ solution) was added dropwise slowly. After 1 h, the reaction temperature was allowed to RT and mixture was stirrer for more 2 h. After the reaction was completed, the resulting mixture was cooled to 0 °C and aqueous saturated solution of sodium carbonate (10 mL) was added dropwise to quench the reaction. After vigorous stirring for 1 h, more water (ca. 20 mL) was added and organic layer was separated. Aqueous layer was extracted by $CHCl_3$ (20 mL) twice. Combined organic layer was dried over sodium sulfate, and solvent was removed by evaporation. The residue was purified by silica gel column chromatography (eluent: hexane/AcOEt = 7/3). 4-nitrophenylisocyanide (**2j**) was obtained in 78% yield (0.693 g) as pale yellow solid.

3. Synthesis of Bismuthines



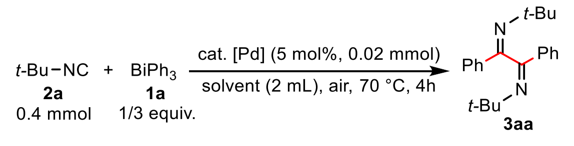
All triarylbismuthines except **1a** were synthesized by minor modified method in the literatures.[4]

Magnesium (1.53 g, 63 mmol) was placed in 200 mL two-necked flask, and heated for drying under N_2. Small amount of dry THF and iodine was added to the flask. The corresponding aryl bromide (63 mmol) was added through dropwise funnel for generation of Grignard reagent, and the reaction mixture was diluted by dry THF. The mixture was heated at 50 °C until the reaction was completed. After all magnesium was consumed, the mixture was cooled to 0 °C by ice bath. THF suspension of $BiCl_3$ (6.3 g, 20 mmol) was added to solution of Grignard reagent slowly. The resulting mixture was stirred for 4 h at 60 °C. After the reaction, sat. NH_4Cl aq. was added and extracted by $CHCl_3$ twice. Combined organic layer was dried over sodium sulfate, and solvent was removed in reduced pressure. Pure triarylbismuthines was obtained in 40-60% yield by recrystallization of crude product from hot ethanol.

4. Typical Reaction Procedure of α-Diimine Synthesis (Scheme 1)

To 10 mL flask, Triphenylbismuthine **1a** (176.1 mg, 0.4 mmol) and *tert*-butylisocyanide **2a** (135.7 μL, 1.2 mmol) were dissolved in MeCN (2 mL). Palladium diacetate (5.4 mg, 0.024 mmol) was added to the mixture. The resulting mixture was stirred for 4 h at 70 °C. After the reaction, the crude product was filtered through Celite pat. All volatile was evaporated under reduced pressure, and the crude product was purified by recycle GPC (eluent: $CHCl_3$). *N,N'*-bis(1,1-dimethylethyl)-1,2-diphenylethane-1,2-diimine (**3aa**) was obtained in 80% yield (154 mg).

5. Investigation of Palladium Catalysts and Solvents

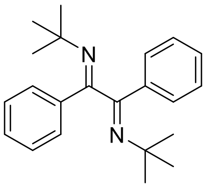


entry	cat. [Pd]	solvent	yield[a]
1	Pd(OAc)$_2$	benzene	93%
2	PdCl$_2$	benzene	67%
3	PdCl$_2$(PPh$_3$)$_2$	benzene	60%
4	PdCl$_2$(PhCN)$_2$	benzene	69%
5	PdCl$_2$(cod)	benzene	63%
6	Pd(PPh$_3$)$_4$	benzene	69%
7[b]	Pd$_2$(dba)$_3$·CHCl$_3$	benzene	78%
8	Pd(OAc)$_2$	toluene	94%
9	Pd(OAc)$_2$	MeCN	97%
10	Pd(OAc)$_2$	THF	96%
11	Pd(OAc)$_2$	acetone	31%
12	Pd(OAc)$_2$	MeOH	18%
13[c]	**Pd(OAc)$_2$ (2 mol%)**	**MeCN**	**>99% (80%)**
14[c]	Pd(OAc)$_2$ (1 mol%)	MeCN	29%

[a]Determined by ^{1}H NMR. Isolated yield is shown in the parentheses. [b]Pd$_2$(dba)$_3$·CHCl$_3$ (2.5 mmol) was used. [c]Reaction conditions: **2a** (1.2 mmol), **1a** (0.4 mmol), MeCN (2 mL).

6. Compounds Characterization Data

- *N,N'*-bis(1,1-dimethylethyl)-1,2-diphenylethane-1,2-diimine (**3aa**)

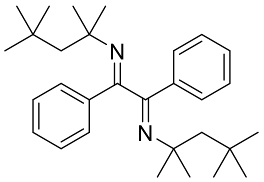


CAS [38015-77-9]

3aa is a known compound.[5] However, **3aa** was not fully characterized in the literature. This compound was isolated in 80% yield (153.8 mg) by recycle GPC (eluent: $CHCl_3$)

White solid; m.p. 107.5–109.0 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.83–7.74 (m, 4H), 7.37–7.27 (m, 6H), 1.24 (s, 18H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.1, 140.4, 129.6, 128.2, 127.7, 57.9, 30.0; HRMS (FAB) Calcd for $C_{22}H_{29}N_2$ $[M+H]^+$: 321.2331, Found: 321.2320.

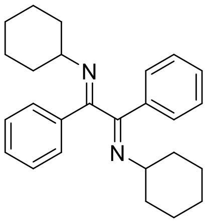
- *N,N'*-bis(1,1,3,3-tetramethylpropyl)-1,2-diphenylethane-1,2-diimine (**3ab**)



3ab is a new compound. This compound was isolated in 80% yield (207.7 mg) by recycle GPC (eluent: $CHCl_3$).

White solid; m.p. 108.0–109.0 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.81–7.74 (m, 4H), 7.34–7.28 (m, 6H), 1.50 (s, 4H), 1.38 (s, 6H), 1.16 (s, 6H), 1.04 (s, 18H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.6, 140.7, 129.5, 128.1, 127.9, 62.0, 57.1, 32.1, 32.0, 30.0, 29.5; HRMS (FAB) Calcd for $C_{30}H_{45}N_2$ $[M+H]^+$: 433.3583, Found: 433.3580.

- *N,N'*-dicyclohexyl-1,2-diphenylethane-1,2-diimine (**3ac**)

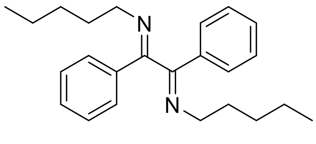


CAS [20586-41-8]

3ac is a known compound.[6] However **3ac** was not fully characterized in the literature. This compound was isolated in 82% yield (183.3 mg) by recycle GPC (eluent: $CHCl_3$).

White solid; m.p. 89.5–91.0 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.84–7.74 (m, 4H), 7.40–7.29 (m, 6H), 3.25 (tt, $J = 4.1, 9.6$ Hz, 2H), 1.89–1.84 (m, 2H), 1.73–1.18 (m, 16H), 1.14–0.99 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.1, 137.1, 130.2, 128.4, 127.6, 62.8, 33.9, 32.9, 25.6, 24.1, 24.0; HRMS (FAB) Calcd for $C_{26}H_{33}N_2$ $[M+H]^+$: 373.2644, Found: 373.2644.

- *N,N'*-dipentyl-1,2-diphenylethane-1,2-diimine (**3ad**)

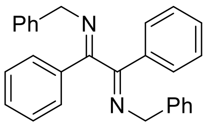


3ad is a new compound.

This compound was isolated in 76% yield (158.9 mg) by recycle GPC (eluent: $CHCl_3$).

Pale yellow oil; ^1H NMR (400 MHz, $CDCl_3$) δ 7.77–7.72 (m, 4H), 7.39–7.31 (m, 6H), 3.33 (t, $J = 7.3$ Hz, 4H), 1.74–1.65 (m, 4H), 1.37–1.23 (m, 8H), 0.86 (t, *J* = 7.3 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.0, 136.1, 130.5, 128.6, 127.1, 54.5, 30.5, 29.8, 22.5, 14.0; HRMS (FAB) Calcd for $C_{24}H_{33}N_2$ $[M+H]^+$: 349.2644, Found: 349.2663.

- *N,N'*-bis(phenylmethyl)-1,2-diphenylethane-1,2-diimine (**3ae**)

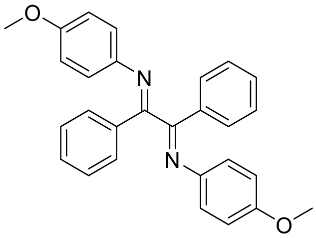


CAS [59508-51-9]

3ae is a known compound. This compound was isolated in 60% yield (139.9 mg) by recycle GPC (eluent: $CHCl_3$).

White solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88–7.83 (m, 4H), 7.43–7.16 (m, 16H), 4.60 (d, *J* = 15.9 Hz, 2H), 4.53 (d, *J* = 15.9 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.8, 139.5, 135.7, 131.0, 128.8, 128.3, 127.9, 127.4, 126.8, 57.7; GC-MS (EI) *m/z* = 388 (M^+).

- *N,N'*-bis(4-methoxyphenyl)-1,2-diphenylethane-1,2-diimine (**3af**)

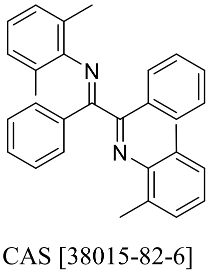


CAS [32349-49-8]

3af is a known compound. This compound was isolated in 58% yield (48.8 mg) by preparative TLC (eluent: hexane/AcOEt = 1/1).

Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88–7.83 (m, 4H), 7.43–7.33 (m, 6H), 6.70–6.61 (m, 8H), 3.72 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.2, 157.3, 142.4, 137.4 130.8, 128.7, 128.0, 122.1, 113.7, 55.3; GC-MS (EI) *m/z* = 420 (M^+).

- *N,N'*-bis(2,6-dimethylphenyl)-1,2-diphenylethane-1,2-diimine (**3ag**)

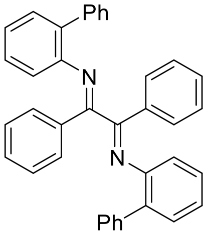


CAS [38015-82-6]

3ag is a known compound. This compound was isolated as mixture of two geometrical isomers (isomers ratio is 4:1) in 85% yield (70.8 mg) by preparative TLC (eluent: hexane/AcOEt = 8/1).

Viscous yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27–8.20 (m, 4H), 7.60–6.68 (m, 12H), 2.13–1.75 (m, 12H); GC-MS (EI) *m/z* = 416 (M^+).

- *N,N'*-bis(2-biphenyl)-1,2-diphenylethane-1,2-diimine (**3ah**)

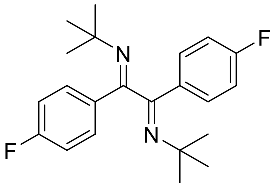


3ah is a new compound.

This compound was isolated in 62% yield (63.6 mg) by preparative TLC (eluent: hexane/AcOEt = 6/1).

Yellow solid; m.p. 184.5–185.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62–7.55 (m, 4H), 7.41–7.33 (m, 2H), 7.28–7.21 (m, 6H), 7.20–7.10 (m, 6H), 7.11–7.01 (m, 2H), 6.91–6.80 (m, 6H), 6.50–6.42 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.9, 145.8, 139.4, 137.6, 134.8, 130.6, 130.3, 129.7, 128.6, 128.3, 127.4, 127.4, 126.3, 125.8, 118.3; HRMS (FAB) Calcd for $C_{38}H_{29}N_2$ $[M+H]^+$: 513.2331, Found: 513.2340.

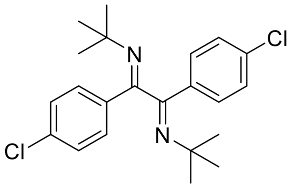
- *N,N'*-bis(1,1-dimethylethyl)-1,2-bis(4-fluorophenyl)ethane-1,2-diimine (**3ba**)



3ba is a new compound. This compound was isolated in 80% yield (171.1 mg) by recycle GPC (eluent: $CHCl_3$).

White solid; m.p. 99.5–102.0 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78–7.71 (m, 4H), 7.03–6.97 (m, 4H), 1.23 (s, 18H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.8 (d, $J_{C\text{–}F}$ = 250.8 Hz), 157.5, 136.3 (d, $J_{C\text{–}F}$ = 2.9 Hz), 129.5 (d, $J_{C\text{–}F}$ = 8.6 Hz), 115.2 (d, $J_{C\text{–}F}$ = 21.9 Hz), 58.0, 29.9; ^{19}F NMR (376 MHz, $CDCl_3$) δ -111.3; HRMS (FAB) Calcd for $C_{22}H_{27}F_2N_2$ $[M+H]^+$: 357.2142, Found: 357.2133.

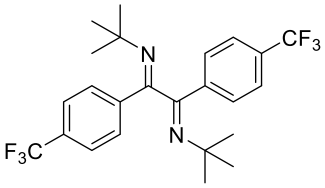
- *N,N'*-bis(1,1-dimethylethyl)-1,2-bis(4-chlorophenyl)ethane-1,2-diimine (**3ca**)



3ca is a new compound. This compound was isolated in 77% yield (179.9 mg) by recycle GPC (eluent: $CHCl_3$).

Pale yellow solid; m.p. 120.0–121.5 °C; ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.71–7.65 (m, 4H), 7.32–7.26 (m, 4H), 1.23 (s, 18H); ^{13}C NMR (100 MHz, CDCl$_3$) δ157.4, 138.4, 135.9, 128.8, 128.6, 58.2, 29.9; HRMS (FAB) Calcd for $C_{22}H_{27}Cl_2N_2$ [M+H]$^+$: 389.1551, Found: 389.1546.

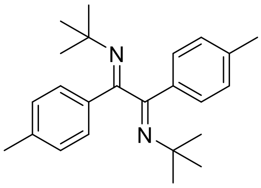
- *N,N'*-bis(1,1-dimethylethyl)-1,2-bis(4-trifluoromethylphenyl)ethane-1,2-diimine (**3da**)



3da is a new compound. This compound was isolated in 65% yield (178.0 mg) by recycle GPC (eluent: CHCl$_3$).

White solid; m.p. 107.0–109.0 °C; ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.87 (d, J = 8.2 Hz, 4H), 7.59 (d, J = 8.2 Hz, 4H), 1.26 (s, 18H); ^{13}C NMR (100 MHz, CDCl$_3$) δ 157.4, 138.4, 131.7 (q, $J_{C–F}$ = 32.4 Hz), 127.8, 125.4 (q, $J_{C–F}$ = 3.8 Hz), 124.1 (q, $J_{C–F}$ = 272.8 Hz), 58.6, 29.9; HRMS (FAB) Calcd for $C_{24}H_{27}F_6N_2$ [M+H]$^+$: 457.2078, Found: 457.2068.

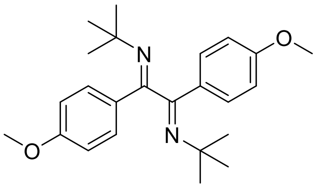
- *N,N'*-bis(1,1-dimethylethyl)-1,2-bis(4-methylphenyl)ethane-1,2-diimine (**3ea**)



3ea is a new compound. This compound was isolated in 81% yield (169.4 mg) by recycle GPC (eluent: CHCl$_3$).

White solid; m.p. 83.5–85.0 °C; ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.69–7.62 (m, 4H), 7.13–7.08 (m, 4H), 2.32 (s, 6H), 1.23 (s, 18H); ^{13}C NMR (100 MHz, CDCl$_3$) δ 159.1, 139.6, 137.9, 128.9, 127.6, 57.7, 30.0, 21.3; HRMS (FAB) Calcd for $C_{24}H_{33}N_2$ [M+H]$^+$: 349.2644, Found: 349.2646.

- *N,N'*-bis(1,1-dimethylethyl)-1,2-bis(4-methoxyphenyl)ethane-1,2-diimine (**3fa**)



3fa is a new compound. This compound was isolated in 87% yield (198.6 mg) by recycle GPC (eluent: CHCl$_3$).

White solid; m.p. 80.0–84.0 °C; ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.73–7.68 (m, 4H), 6.84–6.80 (m, 4H), 3.78 (s, 6H), 1.24 (s, 18H); ^{13}C NMR (100 MHz, CDCl$_3$) δ 160.7, 158.6, 133.2, 113.4, 127.6 57.6, 55.2, 30.0; HRMS

(FAB) Calcd for $C_{24}H_{33}N_2O_2$ $[M+H]^+$: 381.2542, Found: 381.2539.

7. References

(1) Li, J.; Liu, Y.; Li, C.; Jia, X. *Adv. Synth. Catal.* **2011**, *353*, 913.

(2) Zhang, B.; Fan, C.; Yin, H.; Qin, C.; Zhang, G.; Zhang, X.; Yi, H.; Studer, A. *Angew. Chem. Int. Ed.* **2013**, *52*, 10792.

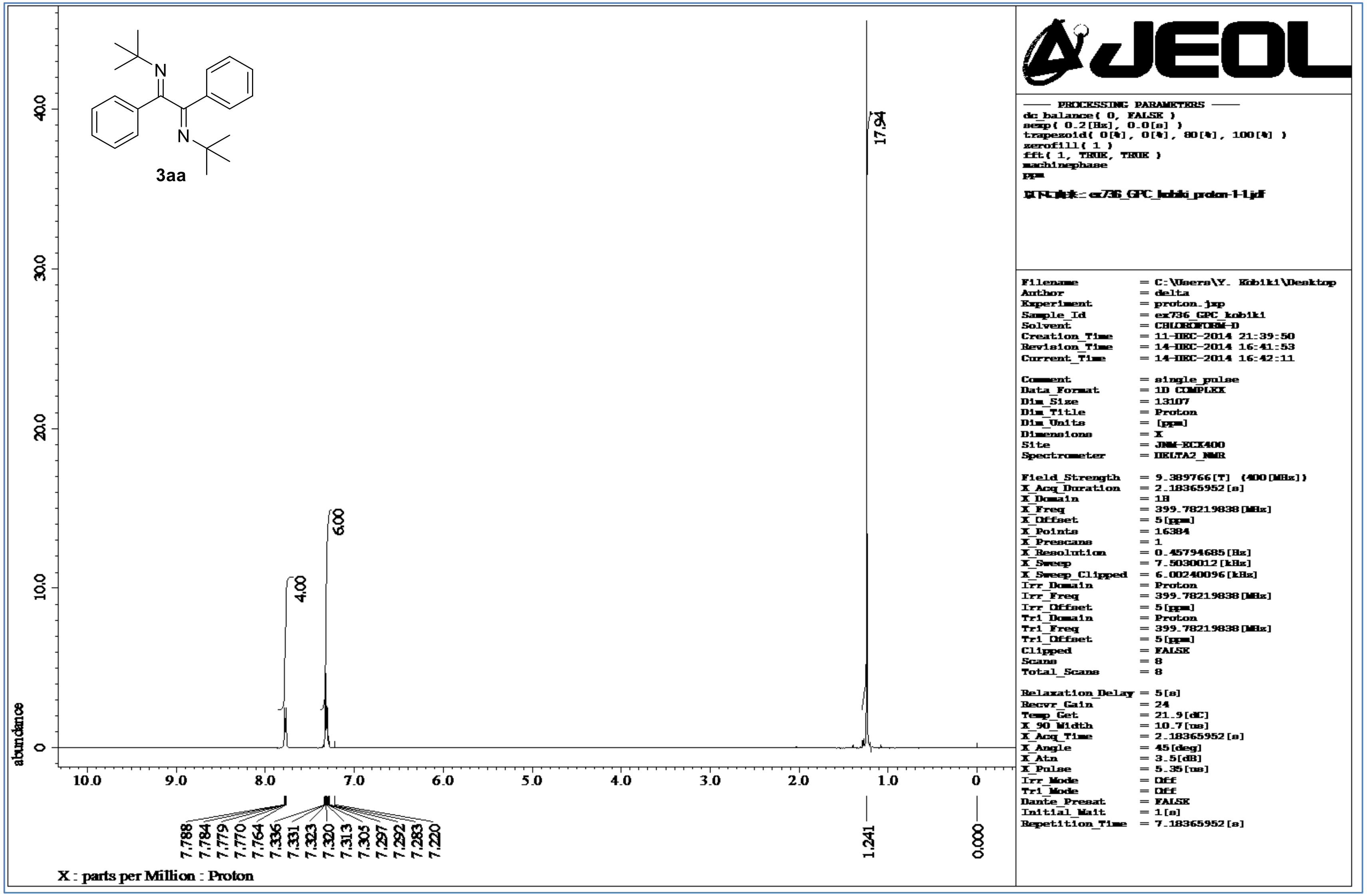
(3) (a) Rahman, M.; Kundu, D.; Hajra, A.; Majee, A. *Tetrahedron Lett.* **2010**, *51*, 2896. (b) Braune, S.; Pohlman, M.; Kazmaier, U. *J. Org. Chem.* **2004**, *69*, 468.

(4) Barton, D. H. R.; Bhatnagar, N. Y.; Finet, J. P.; Motherwell, W. B. *Tetrahedron* **1986**, *42*, 3111.

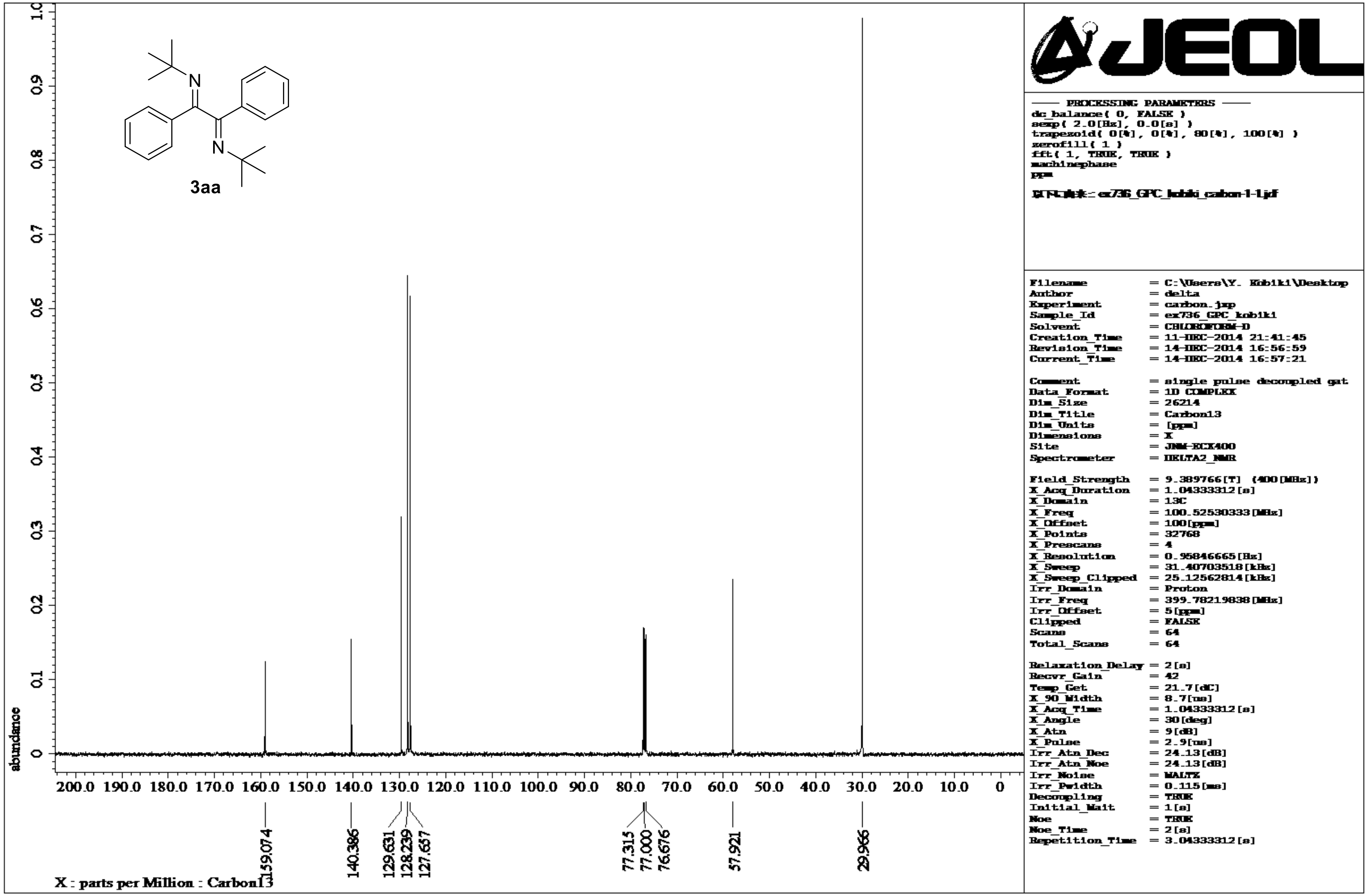
(5) Müller, E.; Nespital, V. *Chem.-Ztg.* **1972**, *96*, 529.

(6) Ugi, I.; Fetzer, U. *Chem. Ber.* **1961**, *94*, 2239.

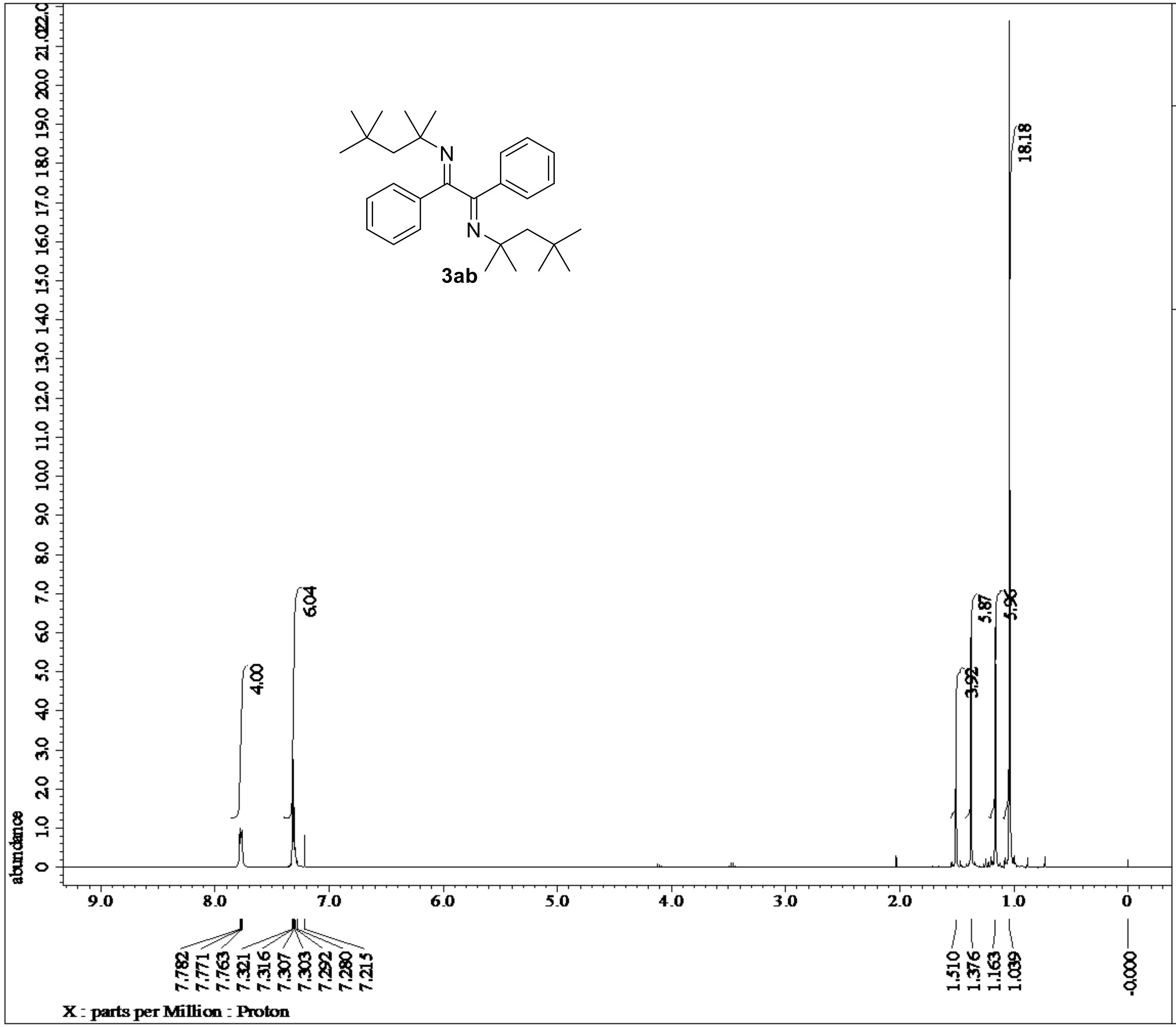
7. Copies of NMR Spectra of Compounds



```
—— PROCESSING PARAMETERS ——
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

[illegible]: ex736_GPC_kobiki_proton-1-1.jdf

Filename           = C:\Users\Y. Kobiki\Desktop
Author             = delta
Experiment         = proton.jxp
Sample_Id          = ex736_GPC_kobiki
Solvent            = CHLOROFORM-D
Creation_Time      = 11-DEC-2014 21:39:50
Revision_Time      = 14-DEC-2014 16:41:53
Current_Time       = 14-DEC-2014 16:42:11

Comment            = single_pulse
Data_Format        = 1D COMPLEX
Dim_Size           = 13107
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECX400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 2.18365952[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 16384
X_Prescans         = 1
X_Resolution       = 0.45794685[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
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         = 24
Temp_Get           = 21.9[dC]
X_90_Width         = 10.7[us]
X_Acq_Time         = 2.18365952[s]
X_Angle            = 45[deg]
X_Atn              = 3.5[dB]
X_Pulse            = 5.35[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
Initial_Wait       = 1[s]
Repetition_Time    = 7.18365952[s]
```



```
—— PROCESSING PARAMETERS ——
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

[illegible]: ex736_GPC_kobiki_carbon-1-1.jdf

Filename           = C:\Users\Y. Kobiki\Desktop
Author             = delta
Experiment         = carbon.jxp
Sample_Id          = ex736_GPC_kobiki
Solvent            = CHLOROFORM-D
Creation_Time      = 11-DEC-2014 21:41:45
Revision_Time      = 14-DEC-2014 16:56:59
Current_Time       = 14-DEC-2014 16:57:21

Comment            = single pulse decoupled gat
Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECX400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 64
Total_Scans        = 64

Relaxation_Delay   = 2[s]
Recvr_Gain         = 42
Temp_Get           = 21.7[dC]
X_90_Width         = 8.7[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 9[dB]
X_Pulse            = 2.9[us]
Irr_Atn_Dec        = 24.13[dB]
Irr_Atn_Noe        = 24.13[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
Noe                = TRUE
Noe_Time           = 2[s]
Repetition_Time    = 3.04333312[s]
```



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—— PROCESSING PARAMETERS ——
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

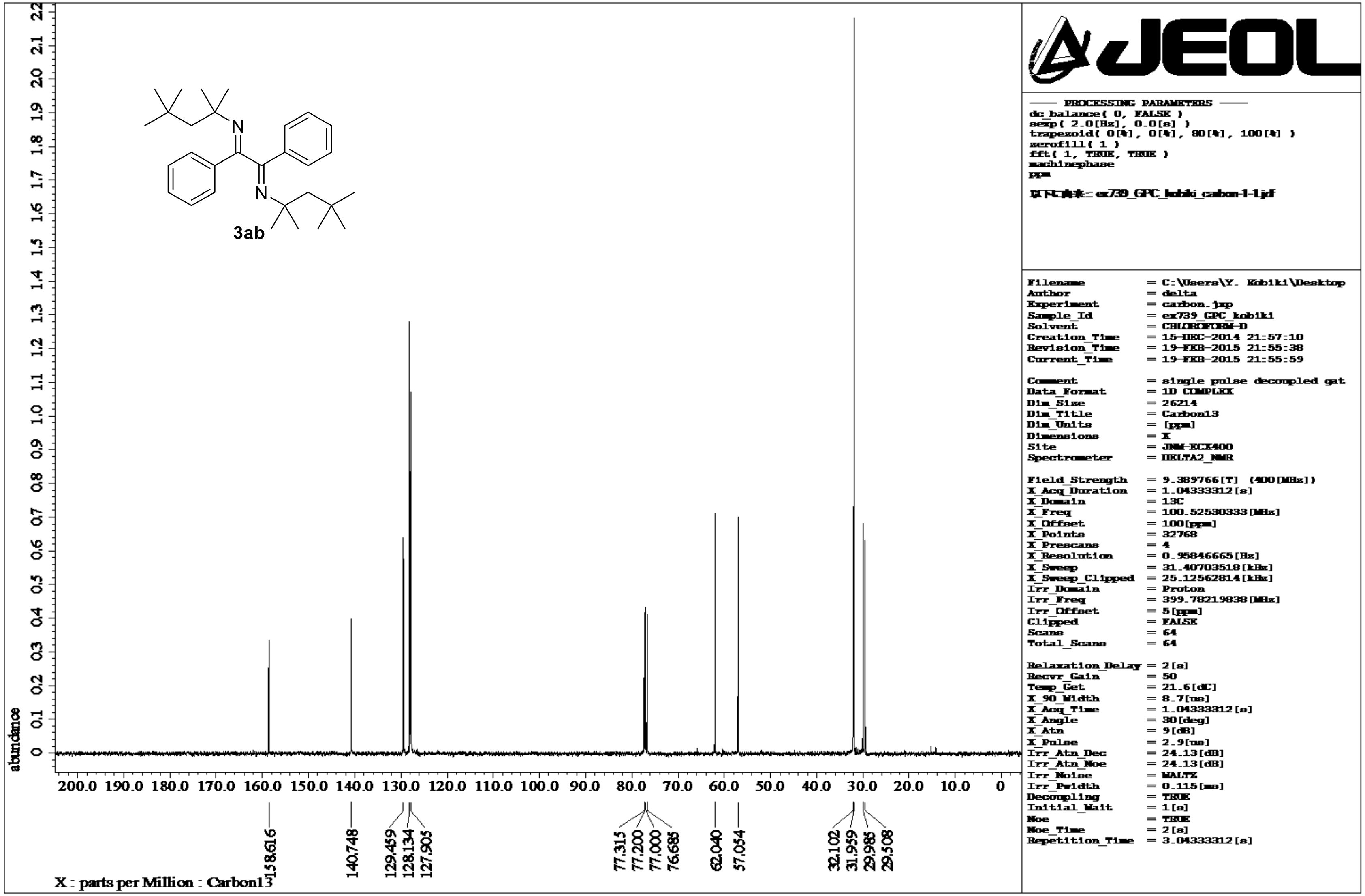
[illegible]: ex739_GPC_kobiki_proton-1-1.jdf

Filename           = C:\Users\Y. Kobiki\Desktop
Author             = delta
Experiment         = proton.jxp
Sample_Id          = ex739_GPC_kobiki
Solvent            = CHLOROFORM-D
Creation_Time      = 15-DEC-2014 21:55:25
Revision_Time      = 19-FEB-2015 15:53:01
Current_Time       = 19-FEB-2015 15:53:21

Comment            = single_pulse
Data_Format        = 1D COMPLEX
Dim_Size           = 13107
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECX400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 2.18365952[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 16384
X_Prescans         = 1
X_Resolution       = 0.45794685[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
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         = 20
Temp_Get           = 22[dC]
X_90_Width         = 10.7[us]
X_Acq_Time         = 2.18365952[s]
X_Angle            = 45[deg]
X_Atn              = 3.5[dB]
X_Pulse            = 5.35[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
Initial_Wait       = 1[s]
Repetition_Time    = 7.18365952[s]
```



```
——— PROCESSING PARAMETERS ———
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

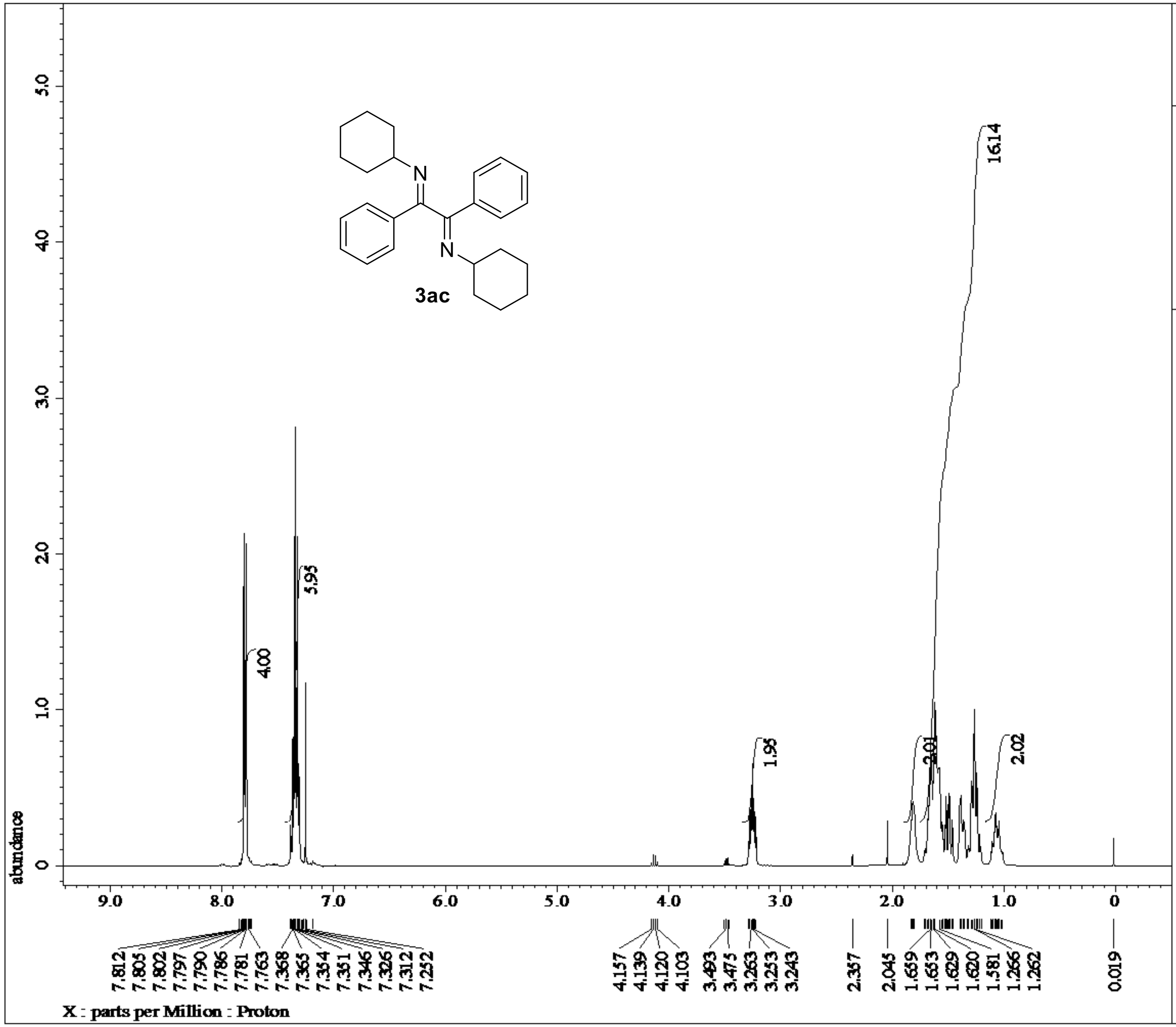
[illegible]: ex739_GPC_kobiki_cabon-1-1jdf

Filename           = C:\Users\Y. Kobiki\Desktop
Author             = delta
Experiment         = carbon.jxp
Sample_Id          = ex739_GPC_kobiki
Solvent            = CHLOROFORM-D
Creation_Time      = 15-DEC-2014 21:57:10
Revision_Time      = 19-FEB-2015 21:55:38
Current_Time       = 19-FEB-2015 21:55:59

Comment            = single pulse decoupled gat
Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECX400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 64
Total_Scans        = 64

Relaxation_Delay   = 2[s]
Recvr_Gain         = 50
Temp_Get           = 21.6[dC]
X_90_Width         = 8.7[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 9[dB]
X_Pulse            = 2.9[us]
Irr_Atn_Dec        = 24.13[dB]
Irr_Atn_Noe        = 24.13[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
Noe                = TRUE
Noe_Time           = 2[s]
Repetition_Time    = 3.04333312[s]
```



3ac

```
—— PROCESSING PARAMETERS ——
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

[illegible]: ex738_GOC_kobiki_proton-1-1.jdf
```

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Filename            = C:\Users\Y. Kobiki\Desktop
Author              = delta
Experiment          = proton.jxp
Sample_Id           = ex738_GOC_kobiki
Solvent             = CHLOROFORM-D
Creation_Time       = 15-DEC-2014 21:44:51
Revision_Time       = 19-FEB-2015 17:10:14
Current_Time        = 19-FEB-2015 17:33:26

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Data_Format         = 1D COMPLEX
Dim_Size            = 13107
Dim_Title           = Proton
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECX400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.389766[T] (400[MHz])
X_Acq_Duration      = 2.18365952[s]
X_Domain            = 1H
X_Freq              = 399.78219838[MHz]
X_Offset            = 5[ppm]
X_Points            = 16384
X_Prescans          = 1
X_Resolution        = 0.45794685[Hz]
X_Sweep             = 7.5030012[kHz]
X_Sweep_Clipped     = 6.00240096[kHz]
Irr_Domain          = Proton
Irr_Freq            = 399.78219838[MHz]
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          = 20
Temp_Get            = 21.8[dC]
X_90_Width          = 10.7[us]
X_Acq_Time          = 2.18365952[s]
X_Angle             = 45[deg]
X_Atn               = 3.5[dB]
X_Pulse             = 5.35[us]
Irr_Mode            = Off
Tri_Mode            = Off
Dante_Presat        = FALSE
Initial_Wait        = 1[s]
Repetition_Time     = 7.18365952[s]
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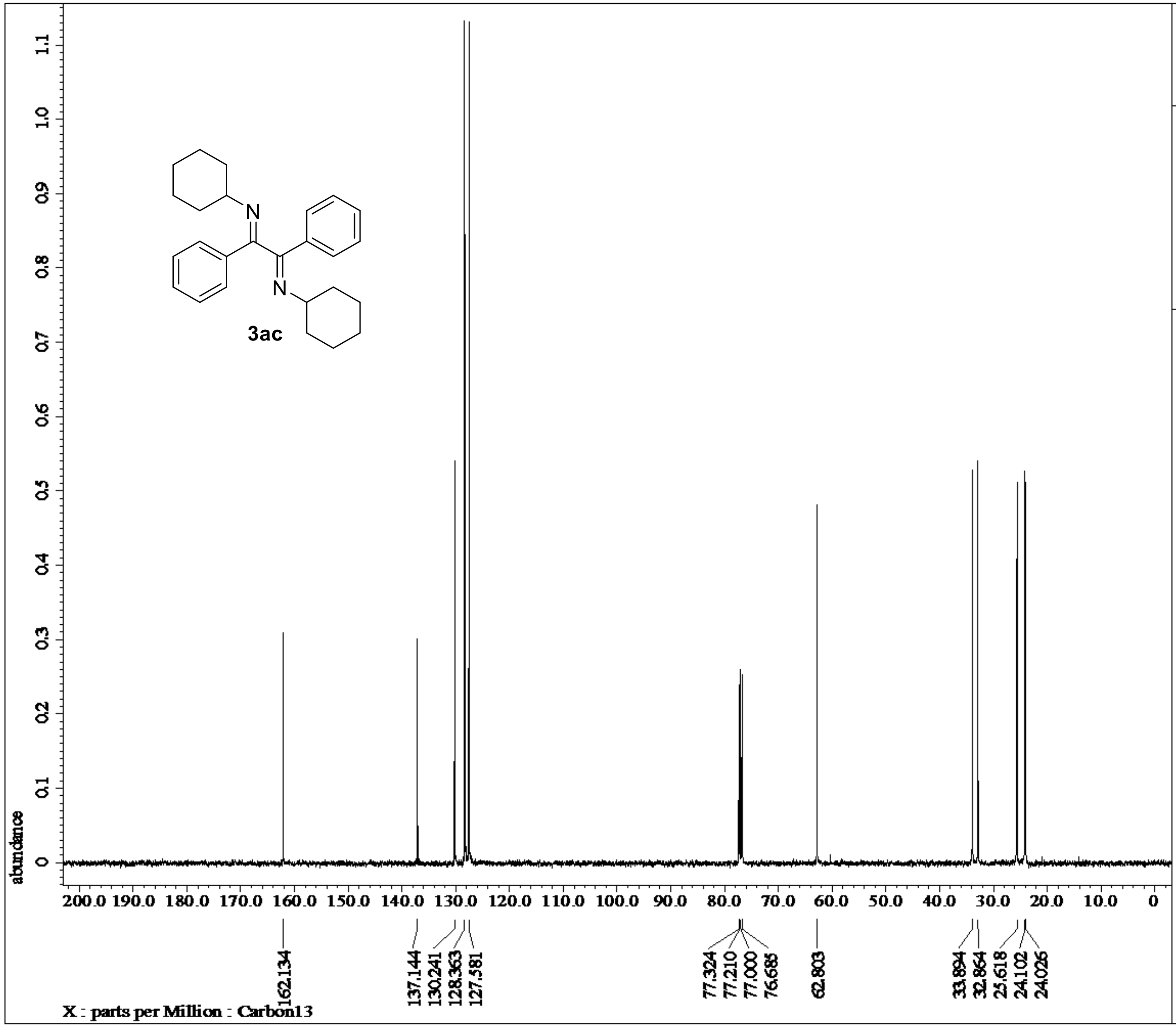

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—— PROCESSING PARAMETERS ——
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sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

[illegible] : ex738_GDC_kobiki_carbon-1-1.jdf
```

```
Filename           = C:\Users\Y. Kobiki\Desktop
Author             = delta
Experiment         = carbon.jxp
Sample_Id          = ex738_GDC_kobiki
Solvent            = CHLOROFORM-D
Creation_Time      = 15-DEC-2014 21:46:49
Revision_Time      = 19-FEB-2015 21:49:35
Current_Time       = 19-FEB-2015 21:53:14

Comment            = single pulse decoupled gat
Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECX400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 64
Total_Scans        = 64

Relaxation_Delay = 2[s]
Recvr_Gain         = 46
Temp_Get           = 22[dC]
X_90_Width         = 8.7[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 9[dB]
X_Pulse            = 2.9[us]
Irr_Atn_Dec        = 24.13[dB]
Irr_Atn_Noe        = 24.13[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
Noe                = TRUE
Noe_Time           = 2[s]
Repetition_Time    = 3.04333312[s]
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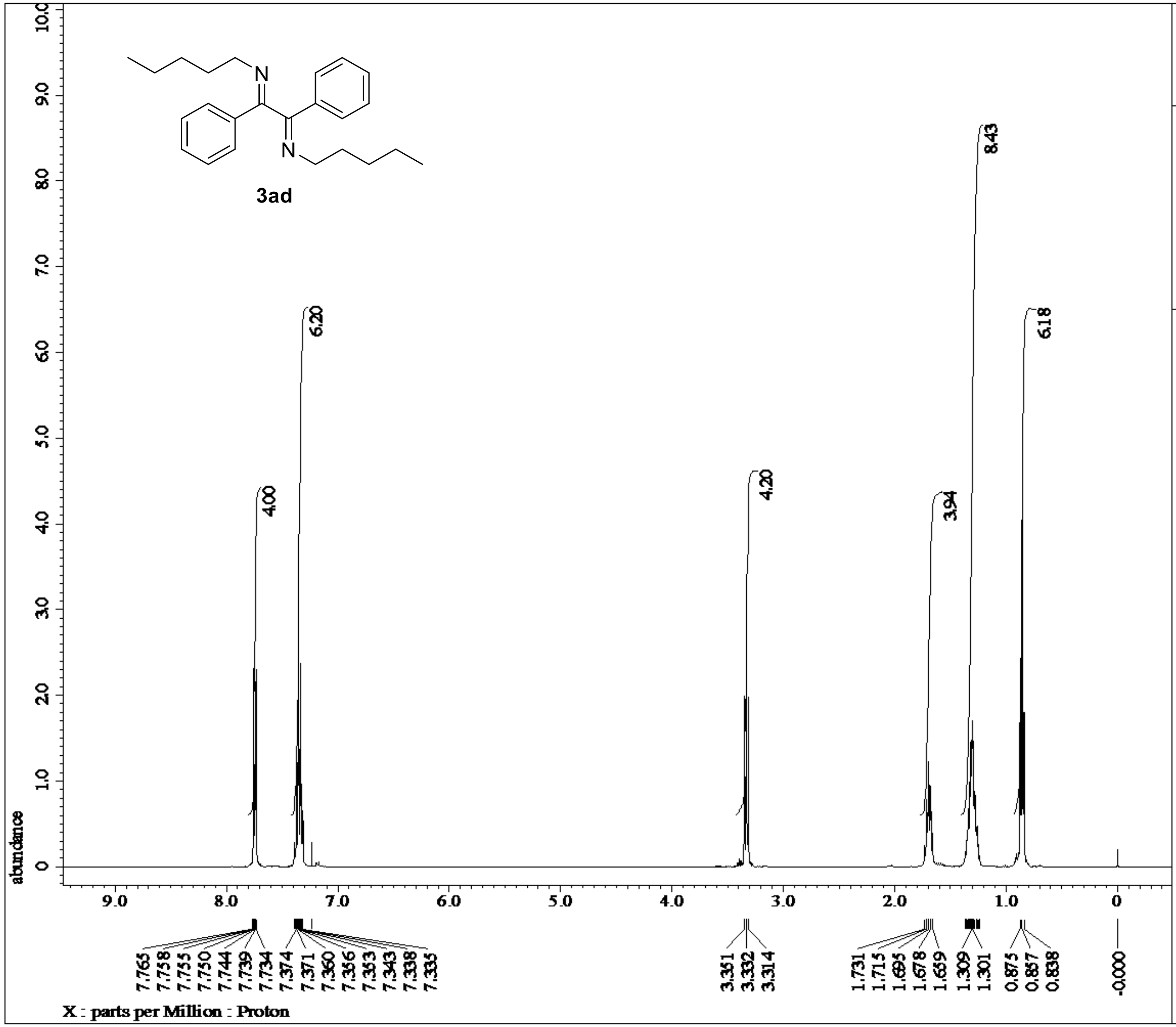

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—— PROCESSING PARAMETERS ——
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trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

[illegible]: ex741_GPC_kobiki_proton-1-1.jdf

Filename           = C:\Users\Y. Kobiki\Desktop
Author             = delta
Experiment         = proton.jxp
Sample_Id          = ex741_GPC_kobiki
Solvent            = CHLOROFORM-D
Creation_Time      = 19-DEC-2014 18:43:18
Revision_Time      = 22-FEB-2015 19:43:29
Current_Time       = 22-FEB-2015 19:43:52

Comment            = single_pulse
Data_Format        = 1D COMPLEX
Dim_Size           = 13107
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECX400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 2.18365952[s]
X_Domain           = 1H
X_Freq             = 399.78219838[MHz]
X_Offset           = 5[ppm]
X_Points           = 16384
X_Prescans         = 1
X_Resolution       = 0.45794685[Hz]
X_Sweep            = 7.5030012[kHz]
X_Sweep_Clipped    = 6.00240096[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
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         = 20
Temp_Get           = 21.5[dC]
X_90_Width         = 10.7[us]
X_Acq_Time         = 2.18365952[s]
X_Angle            = 45[deg]
X_Atn              = 3.5[dB]
X_Pulse            = 5.35[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
Initial_Wait       = 1[s]
Repetition_Time    = 7.18365952[s]
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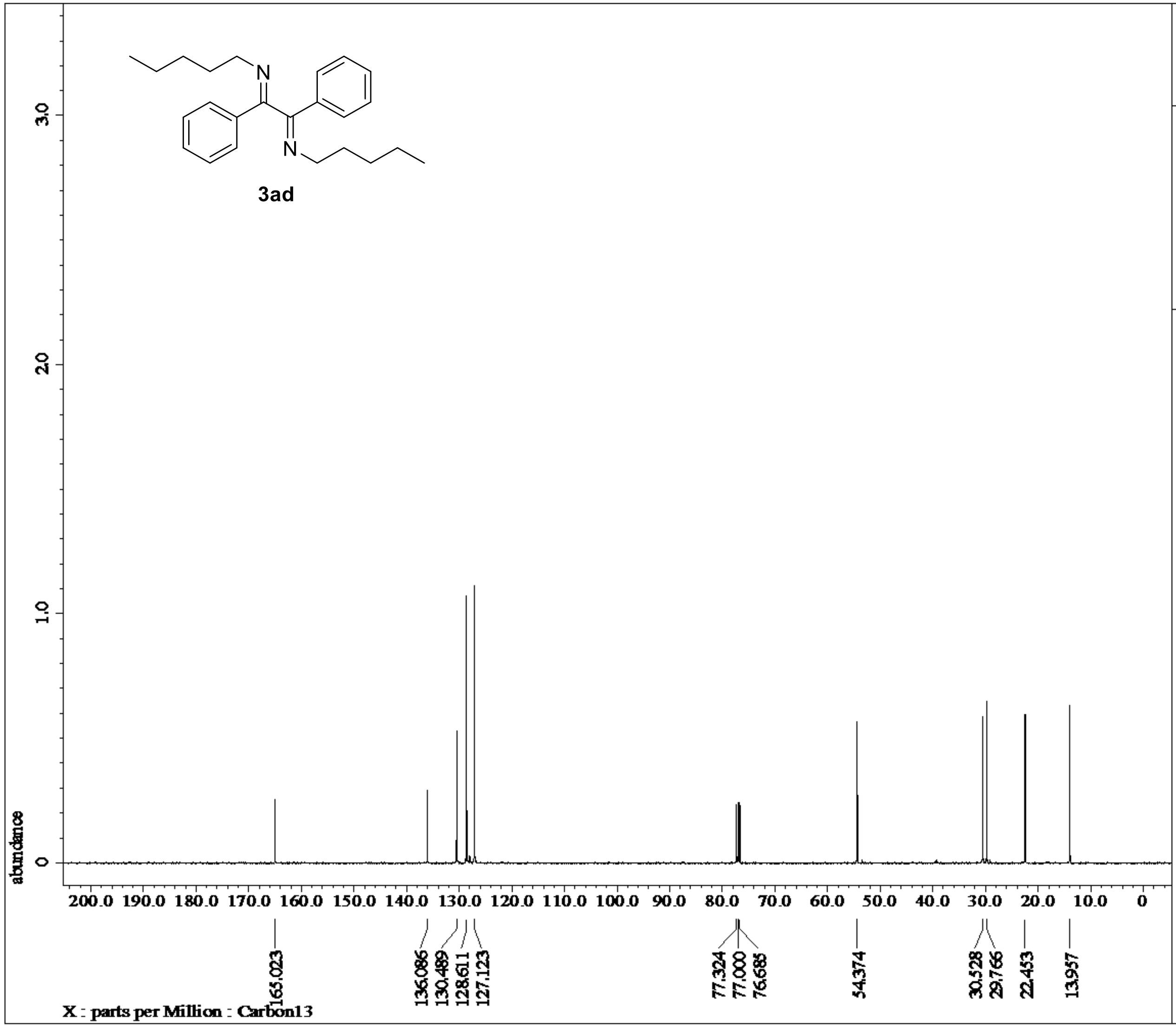

3ad

```
—— PROCESSING PARAMETERS ——
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

[illegible]: ex741_GPC_kobiki_carbon-1-1.jdf

Filename           = C:\Users\Y. Kobiki\Desktop
Author             = delta
Experiment         = carbon.jxp
Sample_Id          = ex741_GPC_kobiki
Solvent            = CHLOROFORM-D
Creation_Time      = 19-DEC-2014 18:45:29
Revision_Time      = 22-FEB-2015 19:47:24
Current_Time       = 22-FEB-2015 19:50:39

Comment            = single pulse decoupled gat
Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECX400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 69
Total_Scans        = 69

Relaxation_Delay = 2[s]
Recvr_Gain         = 46
Temp_Get           = 21.8[dC]
X_90_Width         = 8.7[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 9[dB]
X_Pulse            = 2.9[us]
Irr_Atn_Dec        = 24.13[dB]
Irr_Atn_Noe        = 24.13[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
Noe                = TRUE
Noe_Time           = 2[s]
Repetition_Time    = 3.04333312[s]
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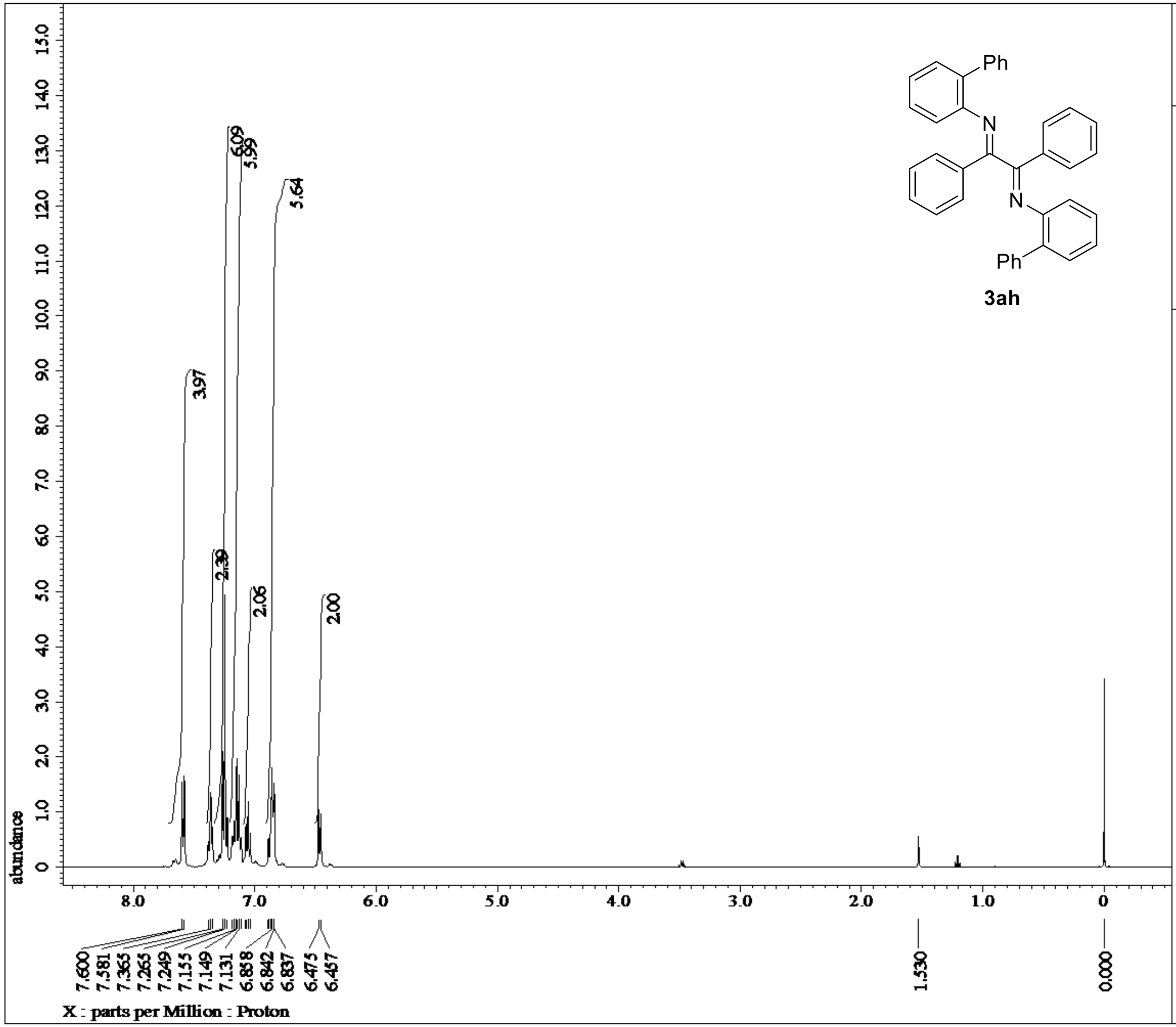
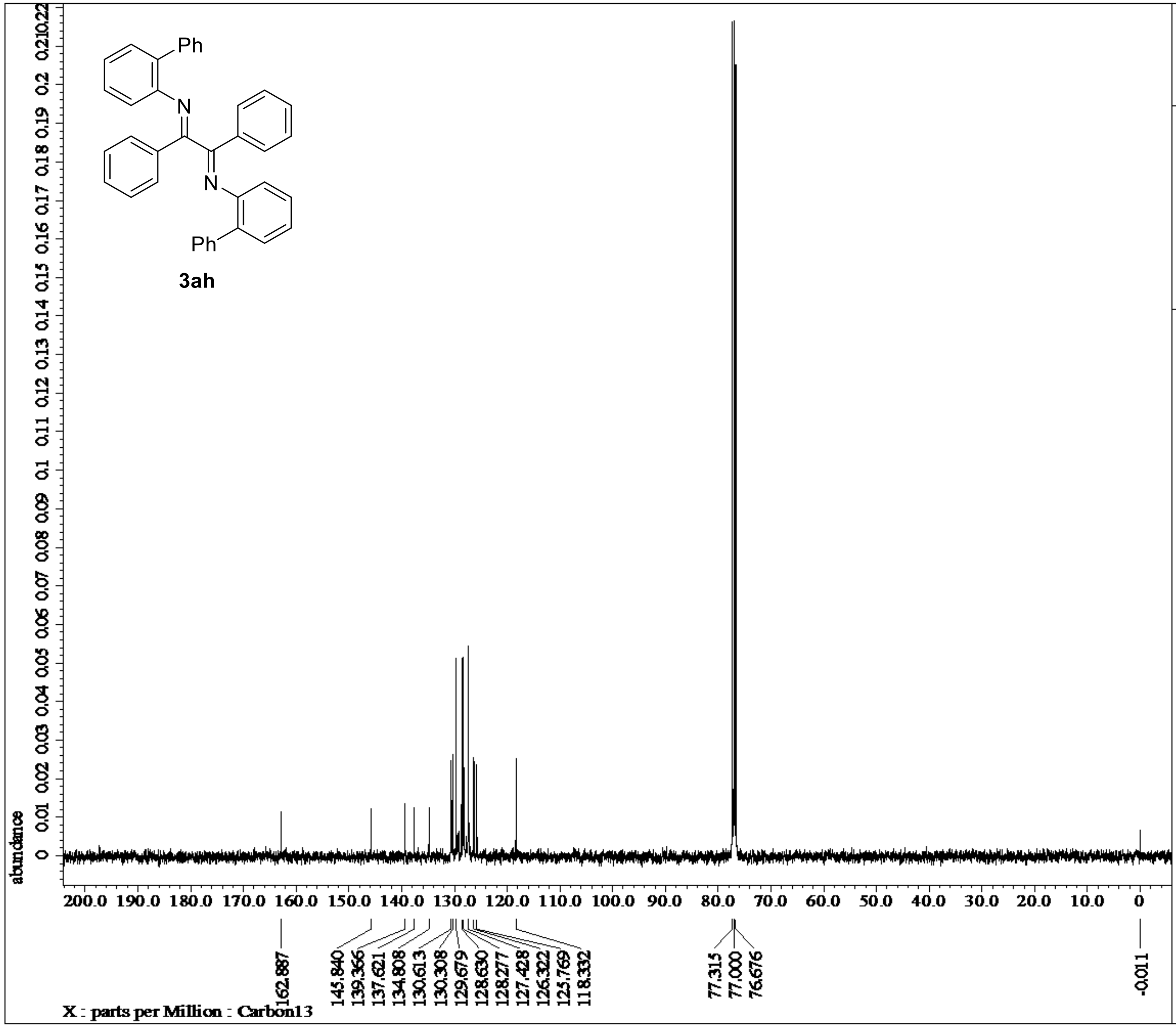


3ah

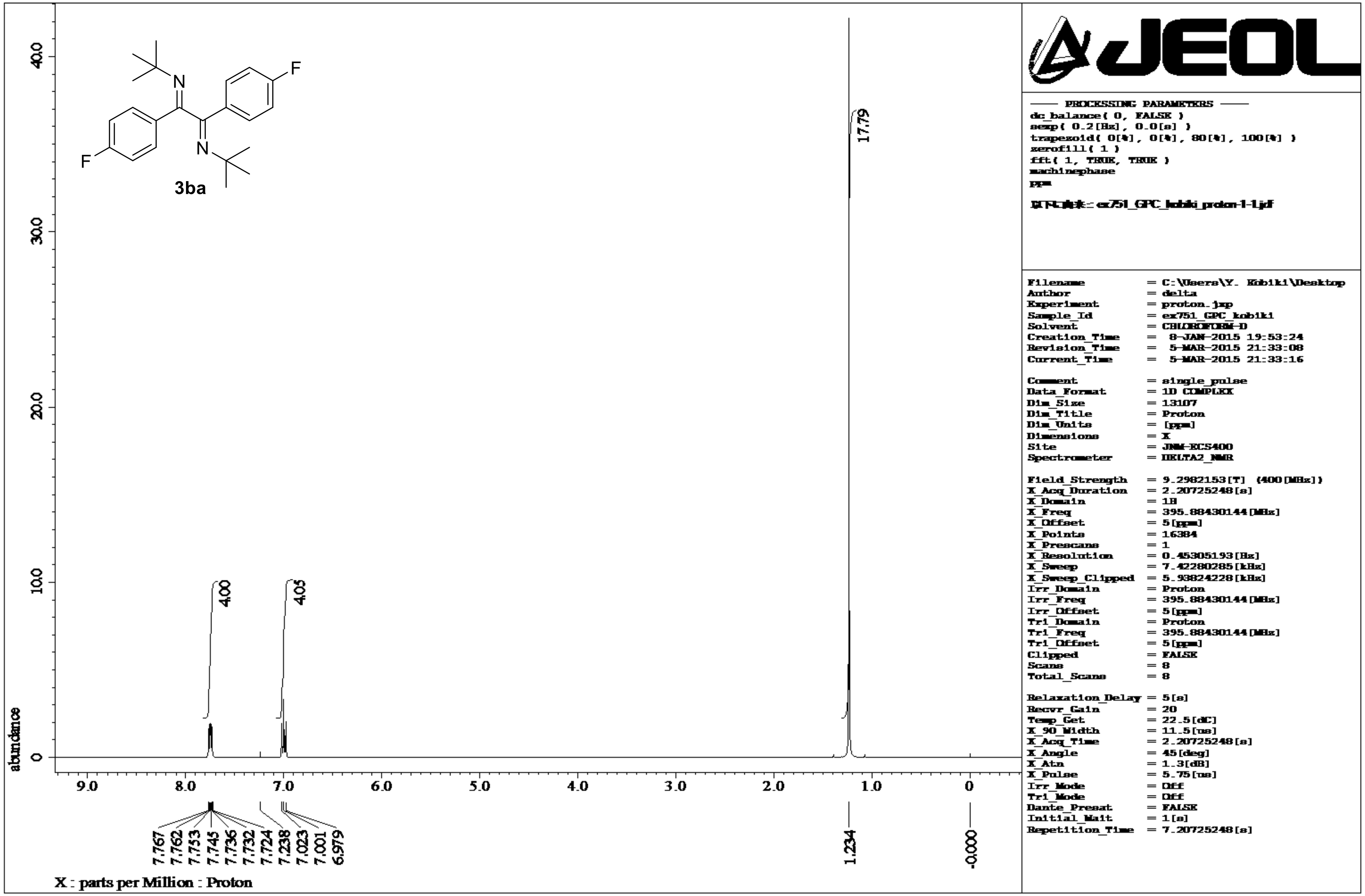
```
—— PROCESSING PARAMETERS ——
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

[illegible] ex832_kobiki_proton-1-1.jdf

Filename          = C:\Users\Y. Kobiki\Desktop
Author            = delta
Experiment        = proton.jxp
Sample_Id         = ex832_kobiki
Solvent           = CHLOROFORM-D
Creation_Time     = 20-FEB-2015 22:08:29
Revision_Time     =  5-MAR-2015 21:07:45
Current_Time      =  5-MAR-2015 21:12:25

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECX400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
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        = 42
Temp_Get          = 21.5[dC]
X_90_Width        = 10.7[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.5[dB]
X_Pulse           = 5.35[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



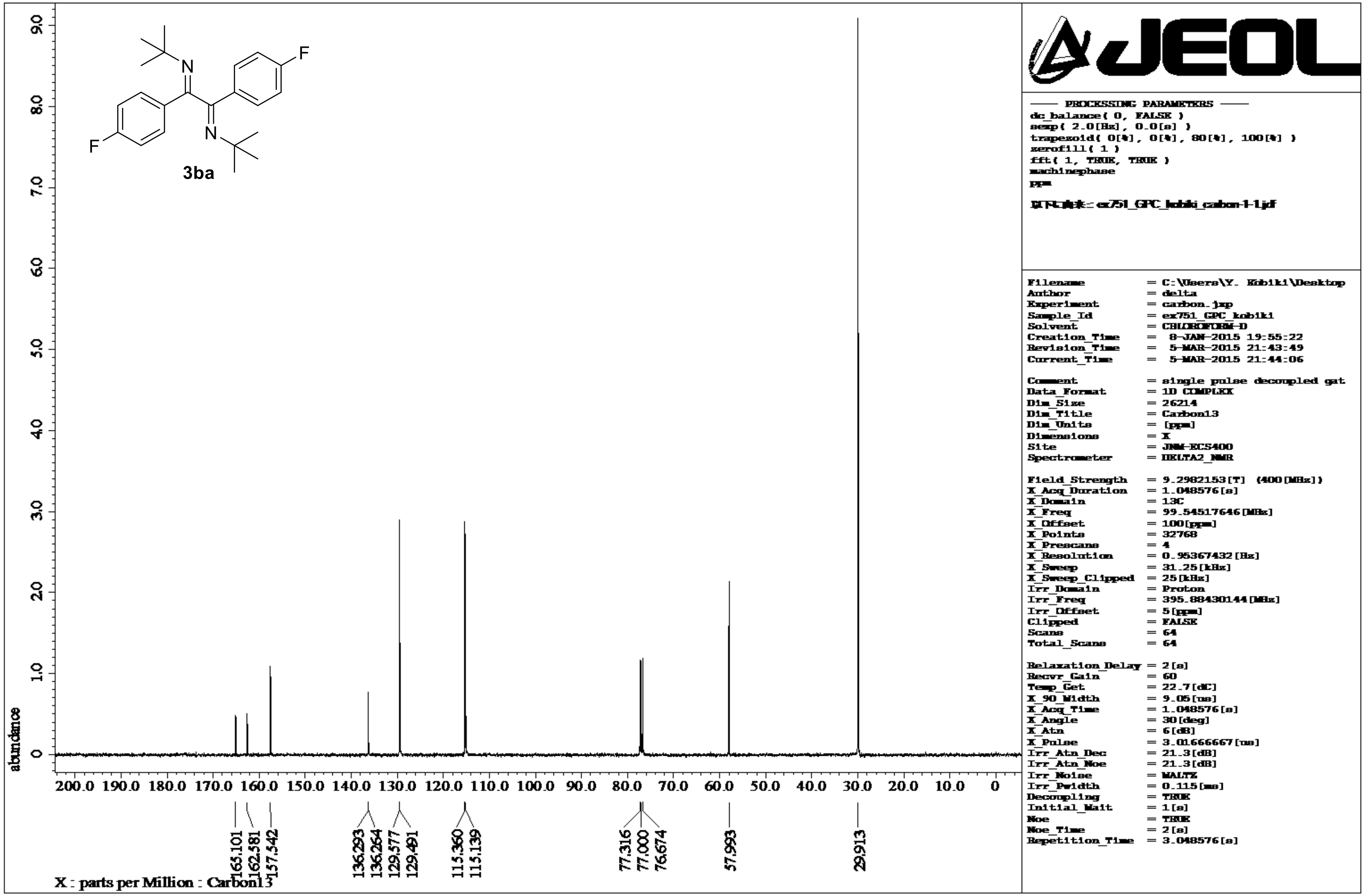
3ah

JEOL

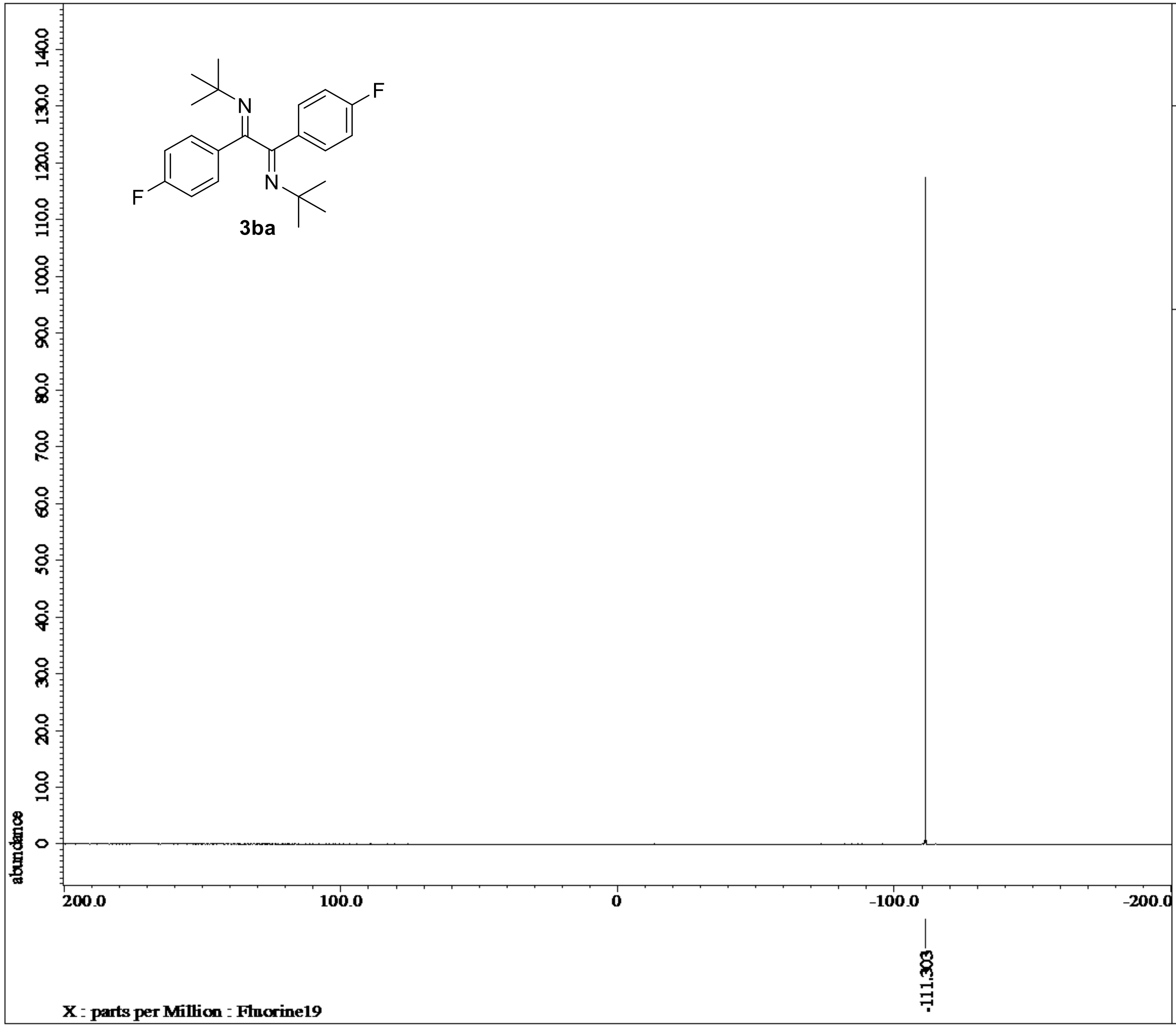
```
—— PROCESSING PARAMETERS ——
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

[illegible] ex832_kobiki_carbon-1-1.jdf

Filename           = C:\Users\Y. Kobiki\Desktop
Author             = delta
Experiment         = carbon.jxp
Sample_Id          = ex832_kobiki
Solvent            = CHLOROFORM-D
Creation_Time      = 20-FEB-2015 22:10:42
Revision_Time      =  5-MAR-2015 21:18:25
Current_Time       =  5-MAR-2015 21:18:40

Comment            = single pulse decoupled gat
Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECX400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.389766[T] (400[MHz])
X_Acq_Duration     = 1.04333312[s]
X_Domain           = 13C
X_Freq             = 100.52530333[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95846665[Hz]
X_Sweep            = 31.40703518[kHz]
X_Sweep_Clipped    = 25.12562814[kHz]
Irr_Domain         = Proton
Irr_Freq           = 399.78219838[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 257
Total_Scans        = 257

Relaxation_Delay   = 2[s]
Recvr_Gain         = 46
Temp_Get           = 22.3[dC]
X_90_Width         = 8.7[us]
X_Acq_Time         = 1.04333312[s]
X_Angle            = 30[deg]
X_Atn              = 9[dB]
X_Pulse            = 2.9[us]
Irr_Atn_Dec        = 24.13[dB]
Irr_Atn_Noe        = 24.13[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
Noe                = TRUE
Noe_Time           = 2[s]
Repetition_Time    = 3.04333312[s]
```



```
—— PROCESSING PARAMETERS ——
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

[illegible]: ex751_GPC_kobiki_proton-1-1.jdf

Filename          = C:\Users\Y. Kobiki\Desktop
Author            = delta
Experiment        = proton.jxp
Sample_Id         = ex751_GPC_kobiki
Solvent           = CHLOROFORM-D
Creation_Time     =  8-JAN-2015 19:53:24
Revision_Time     =  5-MAR-2015 21:33:08
Current_Time      =  5-MAR-2015 21:33:16

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.2982153[T] (400[MHz])
X_Acq_Duration    = 2.20725248[s]
X_Domain          = 1H
X_Freq            = 395.88430144[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45305193[Hz]
X_Sweep           = 7.42280285[kHz]
X_Sweep_Clipped   = 5.93824228[kHz]
Irr_Domain        = Proton
Irr_Freq          = 395.88430144[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 395.88430144[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 20
Temp_Get          = 22.5[dC]
X_90_Width        = 11.5[us]
X_Acq_Time        = 2.20725248[s]
X_Angle           = 45[deg]
X_Atn             = 1.3[dB]
X_Pulse           = 5.75[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.20725248[s]
```

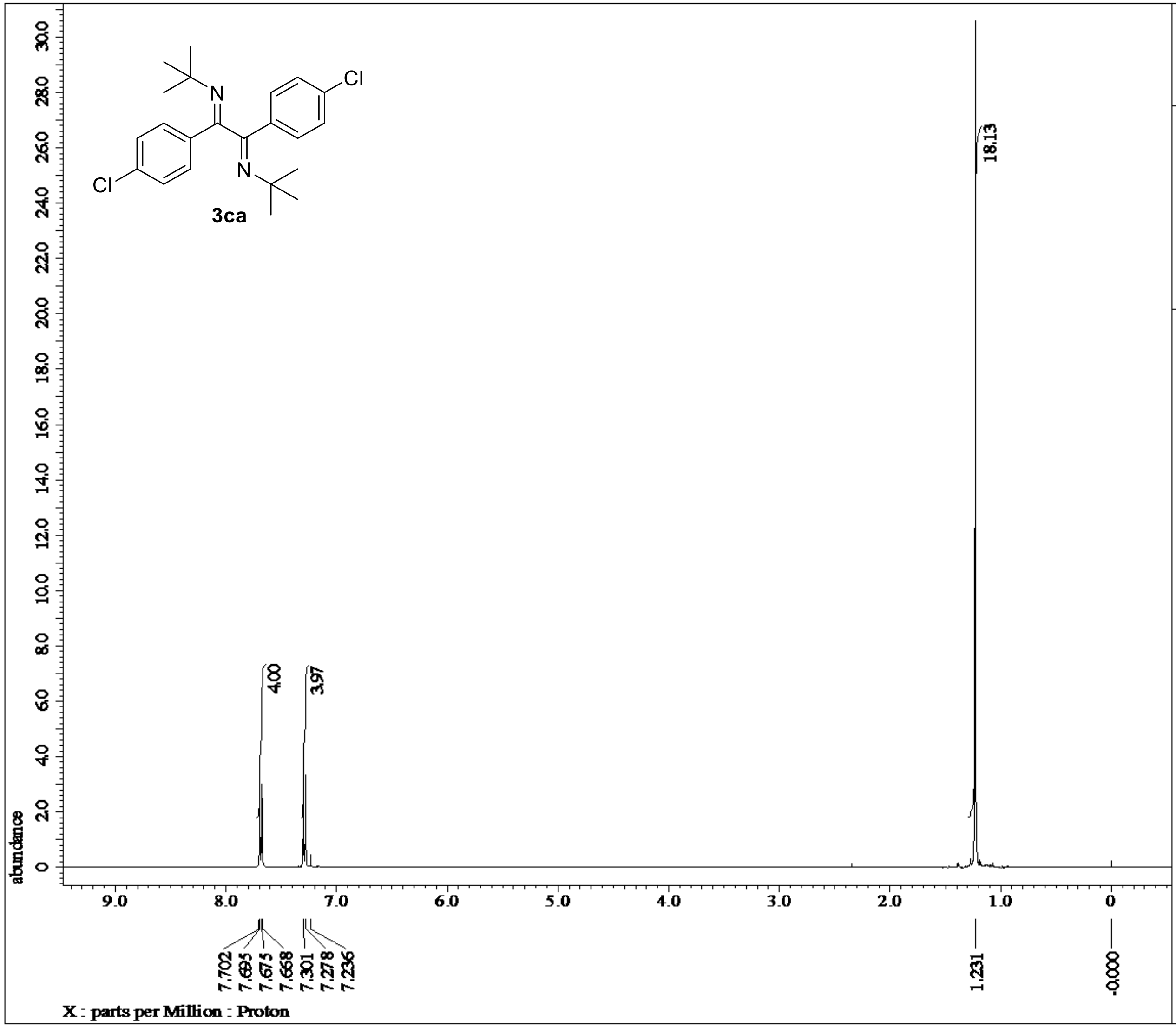


```
—— PROCESSING PARAMETERS ——
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

[illegible]：ex751_GPC_kobiki_cabon-1-1.jdf

Filename           = C:\Users\Y. Kobiki\Desktop
Author             = delta
Experiment         = carbon.jxp
Sample_Id          = ex751_GPC_kobiki
Solvent            = CHLOROFORM-D
Creation_Time      =  8-JAN-2015 19:55:22
Revision_Time      =  5-MAR-2015 21:43:49
Current_Time       =  5-MAR-2015 21:44:06

Comment            = single pulse decoupled gat
Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.2982153[T] (400[MHz])
X_Acq_Duration     = 1.048576[s]
X_Domain           = 13C
X_Freq             = 99.54517646[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95367432[Hz]
X_Sweep            = 31.25[kHz]
X_Sweep_Clipped    = 25[kHz]
Irr_Domain         = Proton
Irr_Freq           = 395.88430144[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 64
Total_Scans        = 64

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 22.7[dC]
X_90_Width         = 9.05[us]
X_Acq_Time         = 1.048576[s]
X_Angle            = 30[deg]
X_Atn              = 6[dB]
X_Pulse            = 3.01666667[us]
Irr_Atn_Dec        = 21.3[dB]
Irr_Atn_Noe        = 21.3[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
Noe                = TRUE
Noe_Time           = 2[s]
Repetition_Time    = 3.048576[s]
```

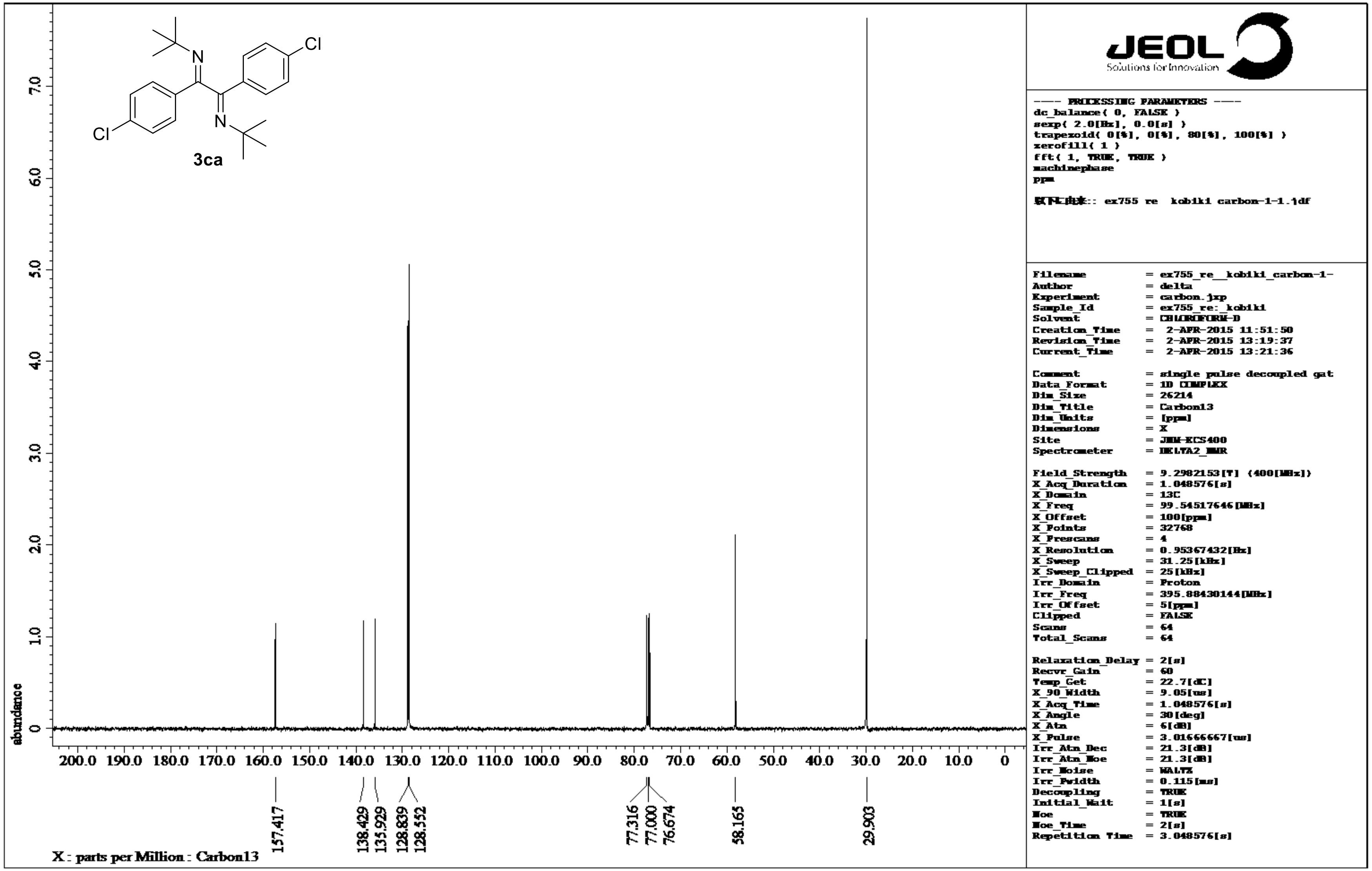


3ba

```
——— PROCESSING PARAMETERS ———
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( 34, 0, 50[%] )

[illegible]: ex751_GPC_kobiki_Fluorine-1-1.jdf

Filename          = C:\Users\Y. Kobiki\Desktop
Author            = delta
Experiment        = proton.jxp
Sample_Id         = ex751_GPC_kobiki
Solvent           = CHLOROFORM-D
Creation_Time     =  8-JAN-2015 20:00:52
Revision_Time     =  5-MAR-2015 21:46:00
Current_Time      =  5-MAR-2015 21:46:29

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Fluorine19
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.2982153[T] (400[MHz])
X_Acq_Duration    = 87.81824[ms]
X_Domain          = 19F
X_Freq            = 372.50336686[MHz]
X_Offset          = 0[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 11.38715602[Hz]
X_Sweep           = 186.56716418[kHz]
X_Sweep_Clipped   = 149.25373134[kHz]
Irr_Domain        = Fluorine19
Irr_Freq          = 372.50336686[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Fluorine19
Tri_Freq          = 372.50336686[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 18
Total_Scans       = 18

Relaxation_Delay  = 5[s]
Recvr_Gain        = 46
Temp_Get          = 22.5[dC]
X_90_Width        = 11.8[us]
X_Acq_Time        = 87.81824[ms]
X_Angle           = 45[deg]
X_Atn             = 1.2[dB]
X_Pulse           = 5.9[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 5.08781824[s]
```

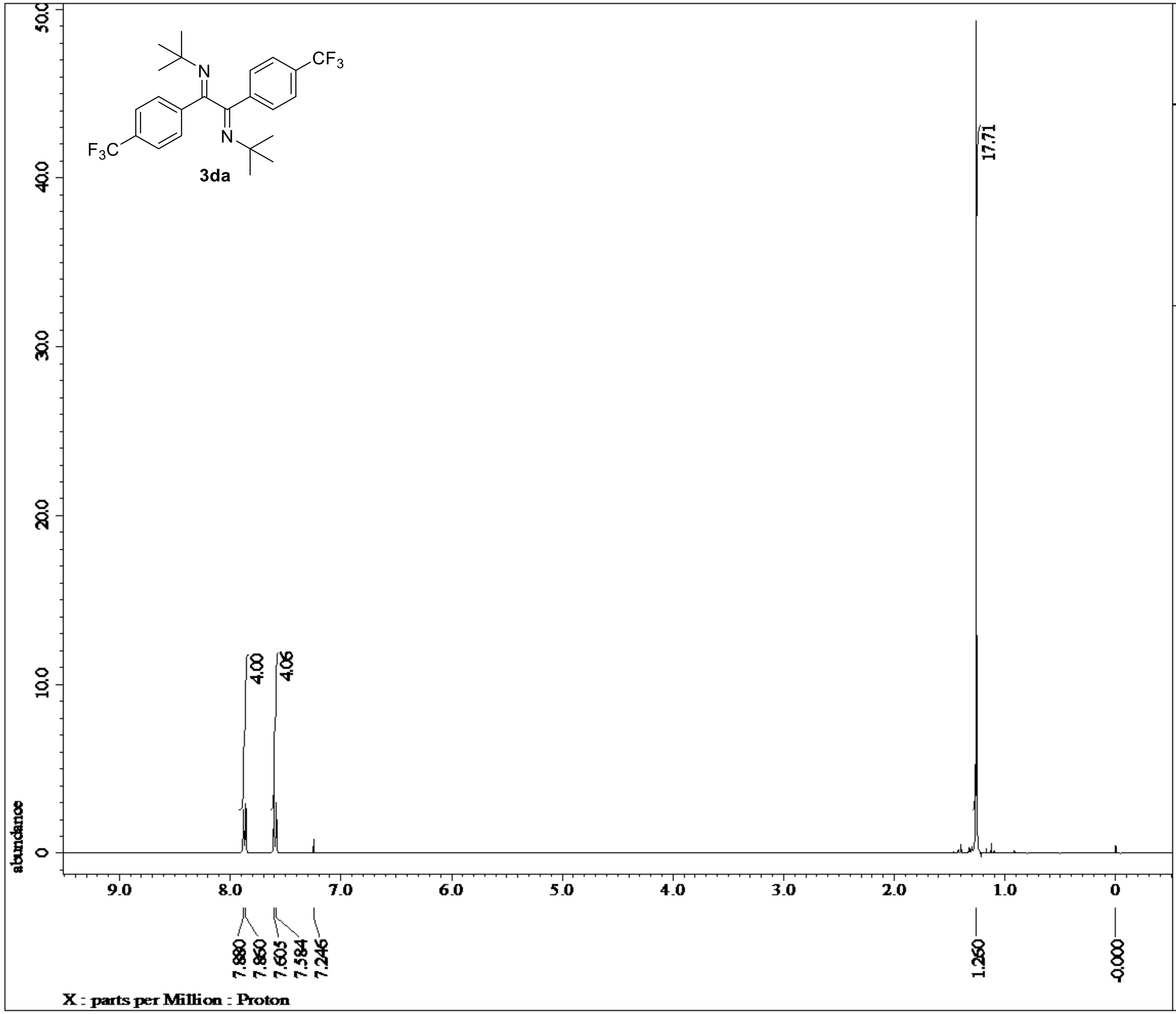


```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

[illegible]: ex755_GPC_kobiki_proton-1-1.jdf

Filename          = C:\Users\Y. Kobiki\Desktop
Author            = delta
Experiment        = proton.jxp
Sample_Id         = ex755_GPC_kobiki
Solvent           = CHLOROFORM-D
Creation_Time     = 13-JAN-2015 00:04:16
Revision_Time     =  5-MAR-2015 21:55:13
Current_Time      =  5-MAR-2015 21:55:25

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECX400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.389766[T] (400[MHz])
X_Acq_Duration    = 2.18365952[s]
X_Domain          = 1H
X_Freq            = 399.78219838[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45794685[Hz]
X_Sweep           = 7.5030012[kHz]
X_Sweep_Clipped   = 6.00240096[kHz]
Irr_Domain        = Proton
Irr_Freq          = 399.78219838[MHz]
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        = 28
Temp_Get          = 21[dC]
X_90_Width        = 10.7[us]
X_Acq_Time        = 2.18365952[s]
X_Angle           = 45[deg]
X_Atn             = 3.5[dB]
X_Pulse           = 5.35[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.18365952[s]
```



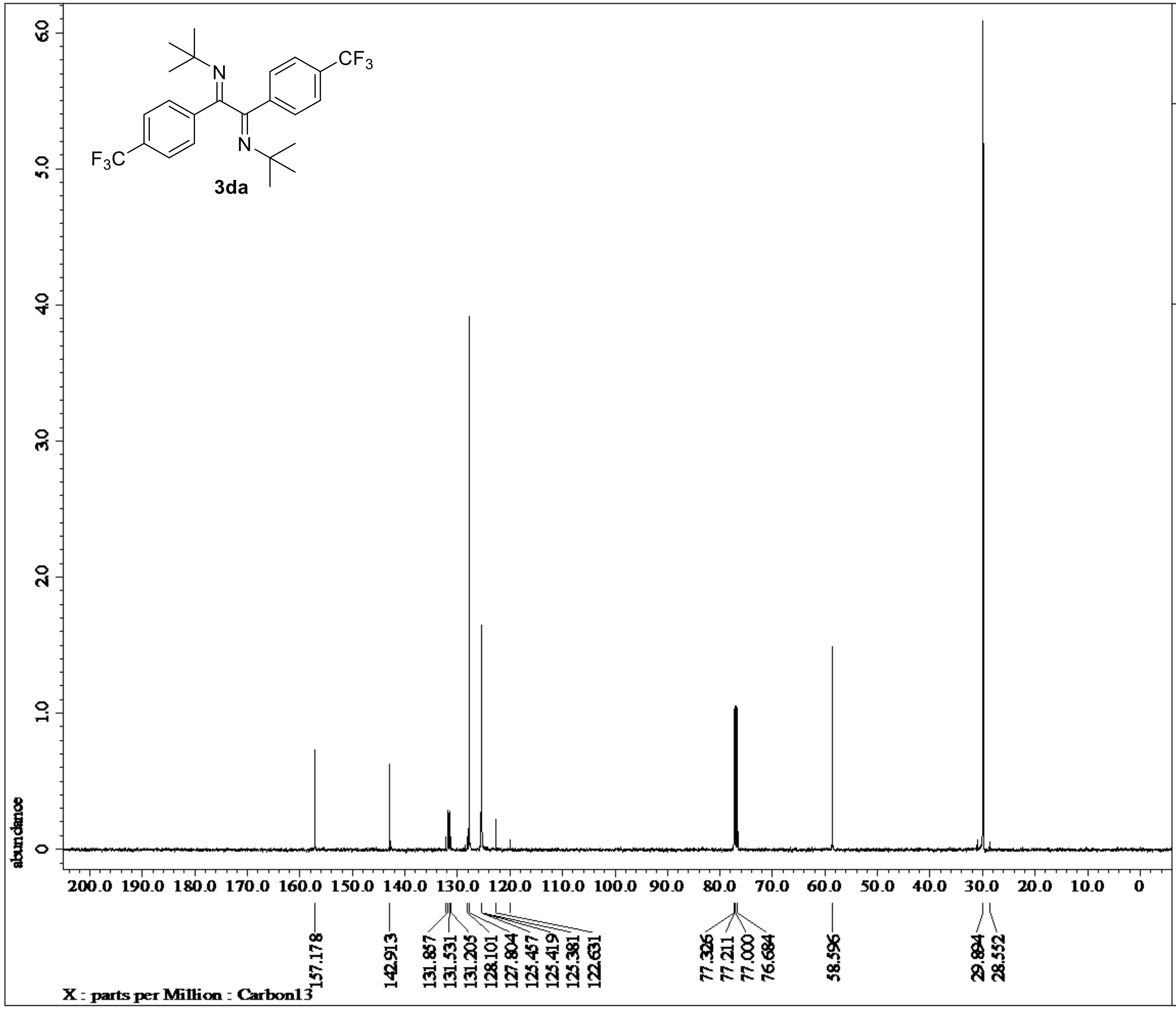
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

[illegible]:: ex755 re  kobiki carbon-1-1.jdf
```

```
Filename          = ex755_re__kobiki_carbon-1-
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = ex755_re:_kobiki
Solvent           = CHLOROFORM-D
Creation_Time     =  2-APR-2015 11:51:50
Revision_Time     =  2-APR-2015 13:19:37
Current_Time      =  2-APR-2015 13:21:36

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.2982153[T] (400[MHz])
X_Acq_Duration    = 1.048576[s]
X_Domain          = 13C
X_Freq            = 99.54517646[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95367432[Hz]
X_Sweep           = 31.25[kHz]
X_Sweep_Clipped   = 25[kHz]
Irr_Domain        = Proton
Irr_Freq          = 395.88430144[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 64
Total_Scans       = 64

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22.7[dC]
X_90_Width        = 9.05[us]
X_Acq_Time        = 1.048576[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.01666667[us]
Irr_Atn_Dec       = 21.3[dB]
Irr_Atn_Noe       = 21.3[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition Time   = 3.048576[s]
```



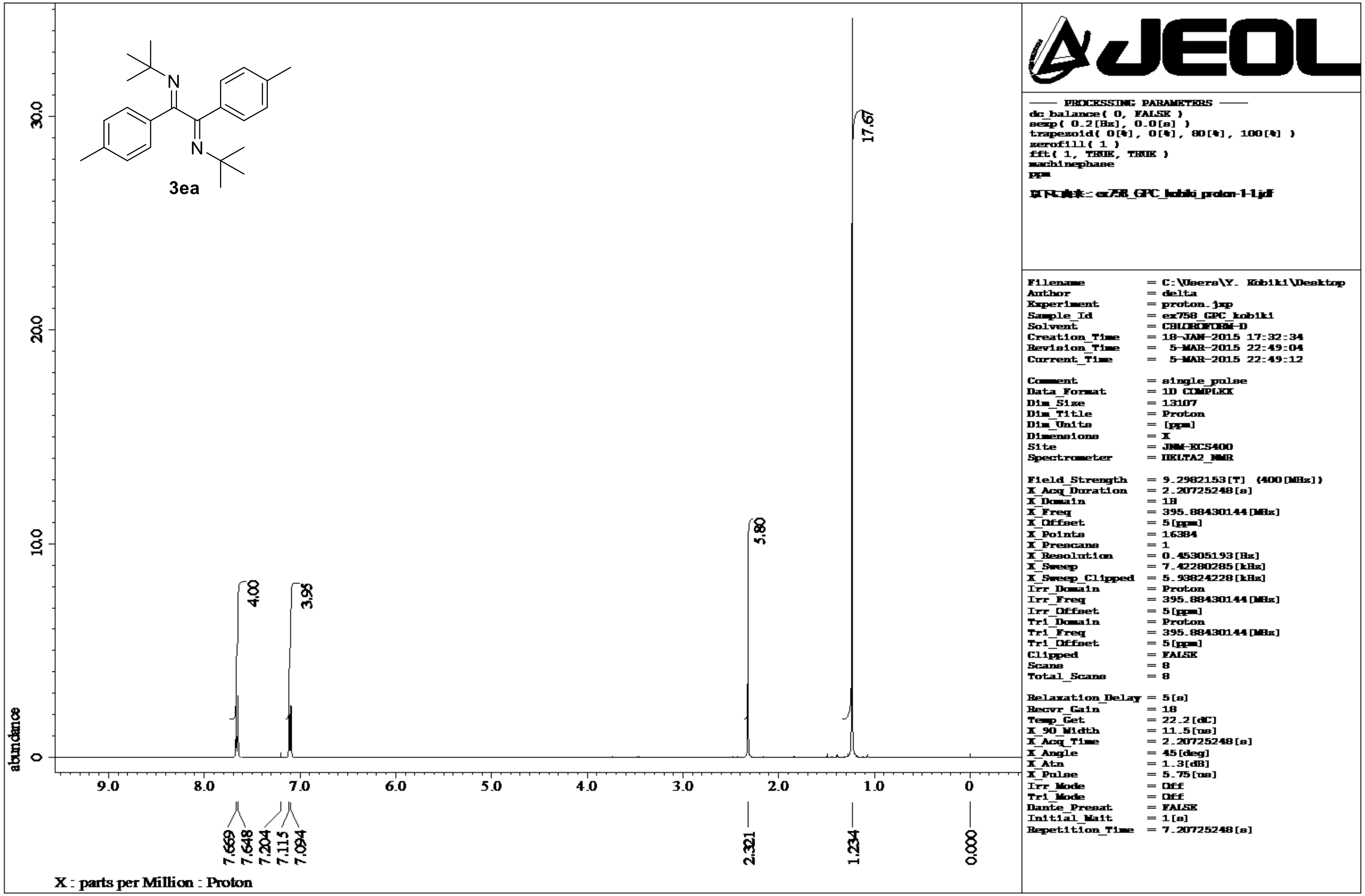
```
—— PROCESSING PARAMETERS ——
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

試料名称：ex756_GPC_kobiki_proton-1-1.jdf
```

```
Filename           = C:\Users\Y. Kobiki\Desktop
Author             = delta
Experiment         = proton.jxp
Sample_Id          = ex756_GPC_kobiki
Solvent            = CHLOROFORM-D
Creation_Time      = 13-JAN-2015 22:15:24
Revision_Time      =  5-MAR-2015 22:30:46
Current_Time       =  5-MAR-2015 22:31:24

Comment            = single_pulse
Data_Format        = 1D COMPLEX
Dim_Size           = 13107
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.2982153[T] (400[MHz])
X_Acq_Duration     = 2.20725248[s]
X_Domain           = 1H
X_Freq             = 395.88430144[MHz]
X_Offset           = 5[ppm]
X_Points           = 16384
X_Prescans         = 1
X_Resolution       = 0.45305193[Hz]
X_Sweep            = 7.42280285[kHz]
X_Sweep_Clipped    = 5.93824228[kHz]
Irr_Domain         = Proton
Irr_Freq           = 395.88430144[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 395.88430144[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay   = 5[s]
Recvr_Gain         = 24
Temp_Get           = 22.2[dC]
X_90_Width         = 11.5[us]
X_Acq_Time         = 2.20725248[s]
X_Angle            = 45[deg]
X_Atn              = 1.3[dB]
X_Pulse            = 5.75[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
Initial_Wait       = 1[s]
Repetition_Time    = 7.20725248[s]
```

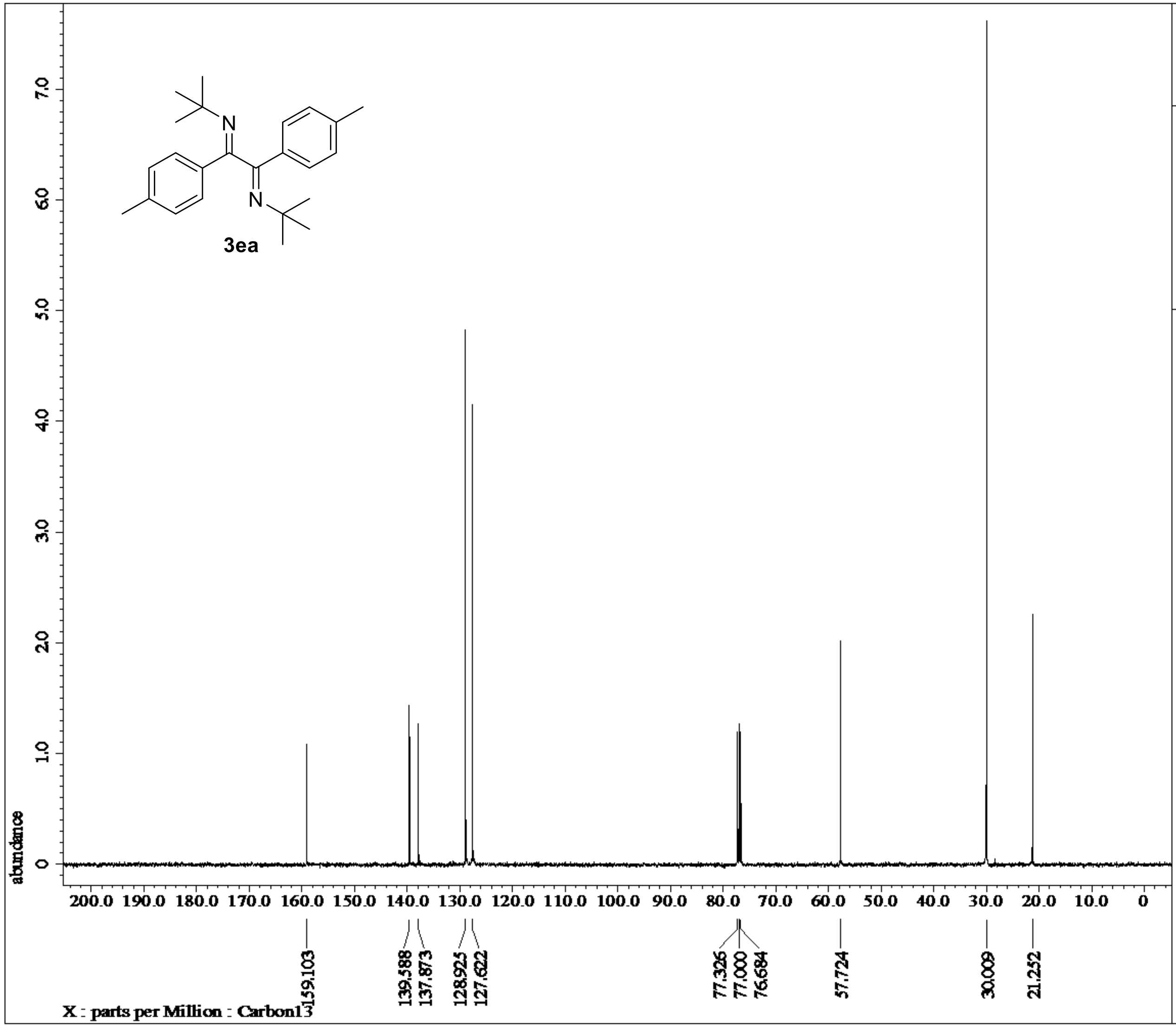


```
—— PROCESSING PARAMETERS ——
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

[illegible]：ex756_GPC_kobiki_cabon-1-1.jdf
```

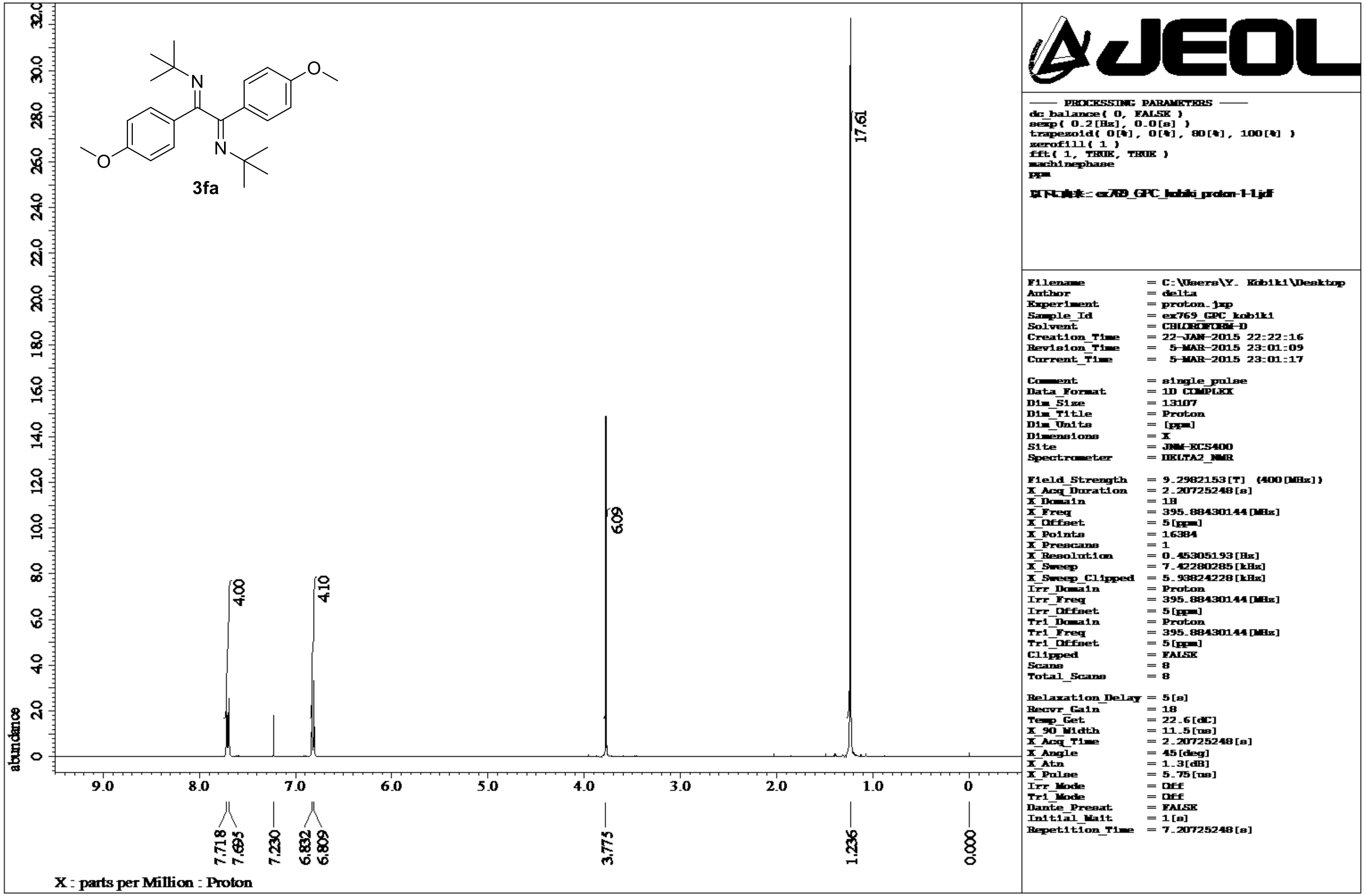
```
Filename          = C:\Users\Y. Kobiki\Desktop
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = ex756_GPC_kobiki
Solvent           = CHLOROFORM-D
Creation_Time     = 13-JAN-2015 22:17:14
Revision_Time     =  5-MAR-2015 22:39:31
Current_Time      =  5-MAR-2015 22:39:48

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.2982153[T] (400[MHz])
X_Acq_Duration    = 1.048576[s]
X_Domain          = 13C
X_Freq            = 99.54517646[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.95367432[Hz]
X_Sweep           = 31.25[kHz]
X_Sweep_Clipped   = 25[kHz]
Irr_Domain        = Proton
Irr_Freq          = 395.88430144[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 148
Total_Scans       = 148

Relaxation_Delay = 2[s]
Recvr_Gain        = 60
Temp_Get          = 22.5[dC]
X_90_Width        = 9.05[us]
X_Acq_Time        = 1.048576[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3.01666667[us]
Irr_Atn_Dec       = 21.3[dB]
Irr_Atn_Noe       = 21.3[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 0.115[ms]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 3.048576[s]
```


3ea

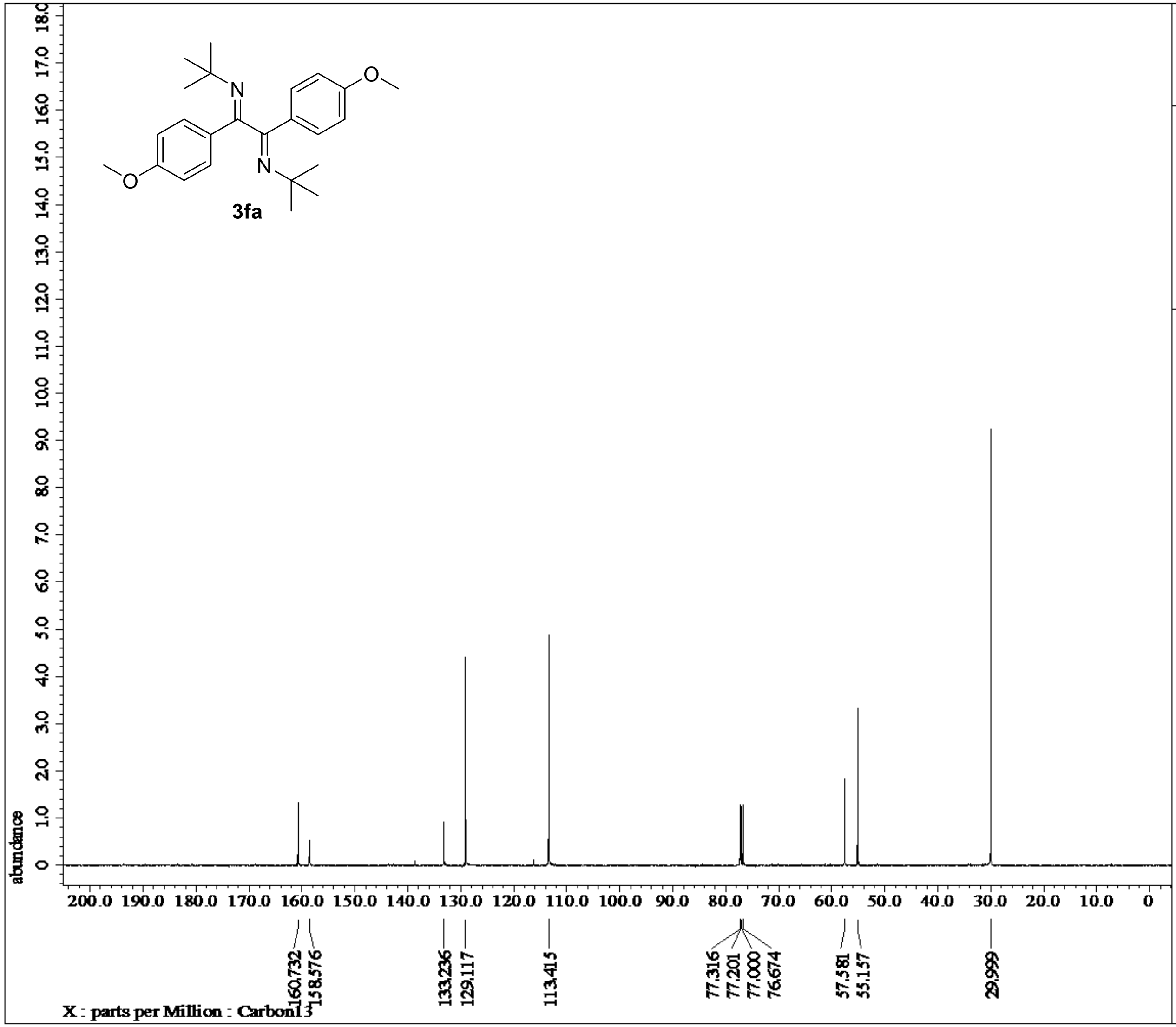
```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

[illegible] : ex758_GPC_kobiki_proton-1-1.jdf

Filename           = C:\Users\Y. Kobiki\Desktop
Author             = delta
Experiment         = proton.jxp
Sample_Id          = ex758_GPC_kobiki
Solvent            = CHLOROFORM-D
Creation_Time      = 18-JAN-2015 17:32:34
Revision_Time      =  5-MAR-2015 22:49:04
Current_Time       =  5-MAR-2015 22:49:12

Comment            = single_pulse
Data_Format        = 1D COMPLEX
Dim_Size           = 13107
Dim_Title          = Proton
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.2982153[T] (400[MHz])
X_Acq_Duration     = 2.20725248[s]
X_Domain           = 1H
X_Freq             = 395.88430144[MHz]
X_Offset           = 5[ppm]
X_Points           = 16384
X_Prescans         = 1
X_Resolution       = 0.45305193[Hz]
X_Sweep            = 7.42280285[kHz]
X_Sweep_Clipped    = 5.93824228[kHz]
Irr_Domain         = Proton
Irr_Freq           = 395.88430144[MHz]
Irr_Offset         = 5[ppm]
Tri_Domain         = Proton
Tri_Freq           = 395.88430144[MHz]
Tri_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 8
Total_Scans        = 8

Relaxation_Delay   = 5[s]
Recvr_Gain         = 18
Temp_Get           = 22.2[dC]
X_90_Width         = 11.5[us]
X_Acq_Time         = 2.20725248[s]
X_Angle            = 45[deg]
X_Atn              = 1.3[dB]
X_Pulse            = 5.75[us]
Irr_Mode           = Off
Tri_Mode           = Off
Dante_Presat       = FALSE
Initial_Wait       = 1[s]
Repetition_Time    = 7.20725248[s]
```


3ea

```
—— PROCESSING PARAMETERS ——
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

[illegible]: ex758_GPC_kobiki_cabon-1-1.jdf

Filename           = C:\Users\Y. Kobiki\Desktop
Author             = delta
Experiment         = carbon.jxp
Sample_Id          = ex758_GPC_kobiki
Solvent            = CHLOROFORM-D
Creation_Time      = 18-JAN-2015 17:34:05
Revision_Time      =  5-MAR-2015 22:51:11
Current_Time       =  5-MAR-2015 22:52:38

Comment            = single pulse decoupled gat
Data_Format        = 1D COMPLEX
Dim_Size           = 26214
Dim_Title          = Carbon13
Dim_Units          = [ppm]
Dimensions         = X
Site               = JNM-ECS400
Spectrometer       = DELTA2_NMR

Field_Strength     = 9.2982153[T] (400[MHz])
X_Acq_Duration     = 1.048576[s]
X_Domain           = 13C
X_Freq             = 99.54517646[MHz]
X_Offset           = 100[ppm]
X_Points           = 32768
X_Prescans         = 4
X_Resolution       = 0.95367432[Hz]
X_Sweep            = 31.25[kHz]
X_Sweep_Clipped    = 25[kHz]
Irr_Domain         = Proton
Irr_Freq           = 395.88430144[MHz]
Irr_Offset         = 5[ppm]
Clipped            = FALSE
Scans              = 64
Total_Scans        = 64

Relaxation_Delay   = 2[s]
Recvr_Gain         = 60
Temp_Get           = 22.6[dC]
X_90_Width         = 9.05[us]
X_Acq_Time         = 1.048576[s]
X_Angle            = 30[deg]
X_Atn              = 6[dB]
X_Pulse            = 3.01666667[us]
Irr_Atn_Dec        = 21.3[dB]
Irr_Atn_Noe        = 21.3[dB]
Irr_Noise          = WALTZ
Irr_Pwidth         = 0.115[ms]
Decoupling         = TRUE
Initial_Wait       = 1[s]
Noe                = TRUE
Noe_Time           = 2[s]
Repetition_Time    = 3.048576[s]
```



```
—— PROCESSING PARAMETERS ——
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

[illegible]: ex769_GPC_kobiki_proton-1-1.jdf

Filename          = C:\Users\Y. Kobiki\Desktop
Author            = delta
Experiment        = proton.jxp
Sample_Id         = ex769_GPC_kobiki
Solvent           = CHLOROFORM-D
Creation_Time     = 22-JAN-2015 22:22:16
Revision_Time     =  5-MAR-2015 23:01:09
Current_Time      =  5-MAR-2015 23:01:17

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECS400
Spectrometer      = DELTA2_NMR

Field_Strength    = 9.2982153[T] (400[MHz])
X_Acq_Duration    = 2.20725248[s]
X_Domain          = 1H
X_Freq            = 395.88430144[MHz]
X_Offset          = 5[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.45305193[Hz]
X_Sweep           = 7.42280285[kHz]
X_Sweep_Clipped   = 5.93824228[kHz]
Irr_Domain        = Proton
Irr_Freq          = 395.88430144[MHz]
Irr_Offset        = 5[ppm]
Tri_Domain        = Proton
Tri_Freq          = 395.88430144[MHz]
Tri_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay  = 5[s]
Recvr_Gain        = 18
Temp_Get          = 22.6[dC]
X_90_Width        = 11.5[us]
X_Acq_Time        = 2.20725248[s]
X_Angle           = 45[deg]
X_Atn             = 1.3[dB]
X_Pulse           = 5.75[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 7.20725248[s]
```



3fa

```
—— PROCESSING PARAMETERS ——
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

[illegible]：ex769_GPC_kobiki_carbon-1-1.jdf
```

```
Filename            = C:\Users\Y. Kobiki\Desktop
Author              = delta
Experiment          = carbon.jxp
Sample_Id           = ex769_GPC_kobiki
Solvent             = CHLOROFORM-D
Creation_Time       = 22-JAN-2015 22:23:47
Revision_Time       =  5-MAR-2015 23:03:13
Current_Time        =  5-MAR-2015 23:04:52

Comment             = single pulse decoupled gat
Data_Format         = 1D COMPLEX
Dim_Size            = 26214
Dim_Title           = Carbon13
Dim_Units           = [ppm]
Dimensions          = X
Site                = JNM-ECS400
Spectrometer        = DELTA2_NMR

Field_Strength      = 9.2982153[T] (400[MHz])
X_Acq_Duration      = 1.048576[s]
X_Domain            = 13C
X_Freq              = 99.54517646[MHz]
X_Offset            = 100[ppm]
X_Points            = 32768
X_Prescans          = 4
X_Resolution        = 0.95367432[Hz]
X_Sweep             = 31.25[kHz]
X_Sweep_Clipped     = 25[kHz]
Irr_Domain          = Proton
Irr_Freq            = 395.88430144[MHz]
Irr_Offset          = 5[ppm]
Clipped             = FALSE
Scans               = 79
Total_Scans         = 79

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 22.8[dC]
X_90_Width          = 9.05[us]
X_Acq_Time          = 1.048576[s]
X_Angle             = 30[deg]
X_Atn               = 6[dB]
X_Pulse             = 3.01666667[us]
Irr_Atn_Dec         = 21.3[dB]
Irr_Atn_Noe         = 21.3[dB]
Irr_Noise           = WALTZ
Irr_Pwidth          = 0.115[ms]
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.048576[s]
```