Synthesis of 2-alkyl-2-boryl-substituted-tetrahydrofurans via copper(I)-catalyzed borylative cyclization of aliphatic ketones

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Table of Contents

1. Instrumentation and Chemicals

Materials were obtained from commercial suppliers and purified by standard procedures unless otherwise noted. Solvents were also purchased from commercial suppliers, degassed via three freeze-pump-thaw cycles, and further dried over molecular sieves (MS 4A). NMR spectra were recorded on JEOL JNM-ECX400P and JNM-ECS400 spectrometers (^{1}H: 401 or 400 or 396 or 392 MHz, ^{13}C: 99 MHz, ^{19}F: 373 MHz ^{11}B: 127 MHz,) Tetramethylsilane (^{1}H), $CDCl_3$ (^{13}C), fluorobenzene (^{19}F), $BF_3 \cdot Et_2O$ (^{11}B) were employed as the external standards, respectively. CuCl (ReagentPlus® grade, 224332-25G, ≥99%) and K(O-*t*-Bu) / THF (1.0 M, 328650-50ML) were purchased from Sigma-Aldrich Co. and used as received. Dibromomethane was used as an internal standard to determine ^{1}H NMR yields. Recycle preparative gel permeation chromatography (GPC) was conducted with JAI LC-9101 using $CHCl_3$ as an eluent. GLC analyses were conducted with a Shimadzu GC-2014 or GC-2025 equipped with a ULBON HR-1 glass capillary column (Shinwa Chemical Industries) and a FID detector. High-resolution mass spectra was recorded at the Global Facility Center for Instrumental Analysis, Hokkaido University.

2. General Experimental Procedures

Procedure for the Copper(I)-Catalyzed Borylative Cyclization of 1a without MeOH (Table 1).

Copper chloride (2.5 mg, 0.025 mmol) and bis(pinacolato)diboron (139.7 mg, 0.55 mmol), 1,3-di-*tert*-butylimidazolium tetrafluoroborate (6.7 mg, 0.025 mmol) were placed in an oven-dried reaction vial. After the vial was sealed with a screw cap containing a Teflon-coated rubber septum, the vial was connected to a vacuum/nitrogen manifold through a needle. It was evacuated and then backfilled with nitrogen. This cycle was repeated three times. Dry THF (450 μL) and K(O-*t*-Bu)/THF (1.0 M, 550 μL, 0.55 mmol) were added in the vial through the rubber septum using a syringe. The dark brown color solution was formed. **1a** (60.3 mg, 0.50 mmol) was then added dropwised to the reaction mixture and the mixture was stirred for 4 h. After the reaction was complete, the reaction mixture was passed through a short silica gel eluting with EtOAc. The crude mixture was purified by flash column chromatography [SiO_2, EtOAc/hexane (dried over K_2CO_3 before used), 0:100–12:88] to give the corresponding borylation product **3a** as a colorless oil. The flash column chromatography should be completed within 10 min to minimize decomposition of the product.

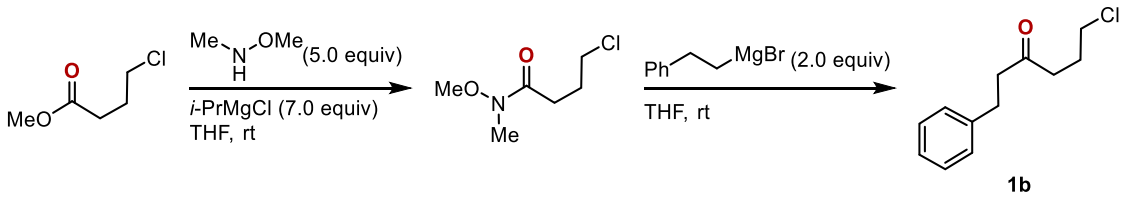
Procedure for the Copper(I)-Catalyzed Borylative Cyclization of 1b with MeOH (Table 2).

Copper chloride (2.5 mg, 0.025 mmol) and bis(pinacolato)diboron (139.7 mg, 0.55 mmol), 1,3-di-*tert*-butylimidazolium tetrafluoroborate (6.7 mg, 0.025 mmol) were placed in an oven-dried reaction vial. After the vial was sealed with a screw cap containing a Teflon-coated rubber septum, the vial was connected to a vacuum/nitrogen manifold through a needle. It was evacuated and then backfilled with nitrogen. This cycle was repeated three times. Dry THF (450 μL) and K(O-*t*-Bu)/THF (1.0 M, 550 μL, 0.55 mmol) were added in the vial through the rubber septum using a syringe. The dark brown color solution was formed. After **1b** (105.4 mg, 0.50 mmol) was added to the mixture at 30°C, MeOH (40.5 μL, 1.0 mmol) was added dropwise. After the reaction was complete, the reaction mixture was passed through a short silica gel eluting with EtOAc. The crude mixture was purified by flash column chromatography [SiO_2, EtOAc/hexane (dried over K_2CO_3 before used), 0:100–12:88] to give the corresponding borylation product **3b** as a colorless oil. The flash column chromatography should be completed within 10 min to minimize decomposition of the product.

3. Substrate Preparation

The substrate **1a** was purchased from commercial suppliers and used received.

Preparation of 6-chloro-1-phenylhexan-3-one (1b).

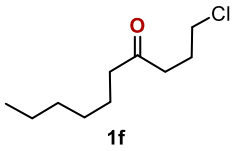


In an oven-dried 300 mL round bottomed flask, methyl 4-chlorobutyrate (1.86 g, 13.6 mmol) was added to a suspension of *N,O*-dimethylhydroxylamine in THF (36 mL) at room temperature under nitrogen atmosphere and the mixture was cooled to –20°C. A THF solution of Isopropylmagnesium chloride (2.0 M, 48 mL, 96 mmol) was then added dropwised for 15 min to the reaction mixture and the mixture was stirred for 1 h. After the reaction mixture was warmed to 0°C, the mixture was stirred for 4 h. Then the reaction mixture was warmed to room temperature, the mixture was stirred for 12 h. The resulting suspension was quenched by the addition of saturated aqueous NH_4Cl. The mixture was extracted with CH_2Cl_2 three times and dried over $MgSO_4$, filtered and concentrated under reduced pressure. The crude product was used for the next step without purification.

In an oven-dried 100 mL round bottomed flask, the amide (993.1 mg, 6.0 mmol) was dissolved in THF (14 mL) under nitrogen atmosphere. After the reaction mixture was cooled to 0°C, a THF solution of phenethylmagnesium bromide (1.5 M, 8.0 mL, 12 mmol) was added dropwised to the reaction mixture. After stirred for 18 h at room temperature, the resulting suspension was quenched by the addition of saturated aqueous NH_4Cl. The mixture was the extracted with EtOAc three times and dried over $MgSO_4$, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography (SiO_2, EtOAc/hexane, 0:100–7:93) to afford the corresponding ketone **1b** (214.0 mg, 1.0 mmol, 17%) as a colorless oil.

^{1}H NMR (401 MHz, $CDCl_3$, δ): 2.03 (quint, J = 6.5 Hz, 2H), 2.59 (t, J = 7.0 Hz, 2H), 2.76 (t, J = 7.6 Hz, 2H), 2.91 (t, J = 7.6 Hz, 2H), 3.55 (t, J = 6.4 Hz, 2H), 7.17–7.22 (m, 3H), 7.26–7.30 (m, 2H). ^{13}C NMR (99 MHz, $CDCl_3$, δ): 26.0 (CH_2), 29.5 (CH_2), 39.2 (CH_2), 44.1 (CH_2), 44.2 (CH_2), 125.9 (*C*H), 128.1 (*C*H), 128.3 (*C*H), 140.7 (*C*), 208.4 (*C*). HRMS–EI (*m/z*): $[M]^+$ calcd for $C_{12}H_{15}ClO$, 210.0811; found, 210.0812.

Preparation of 1-chlorodecan-4-one (1f).

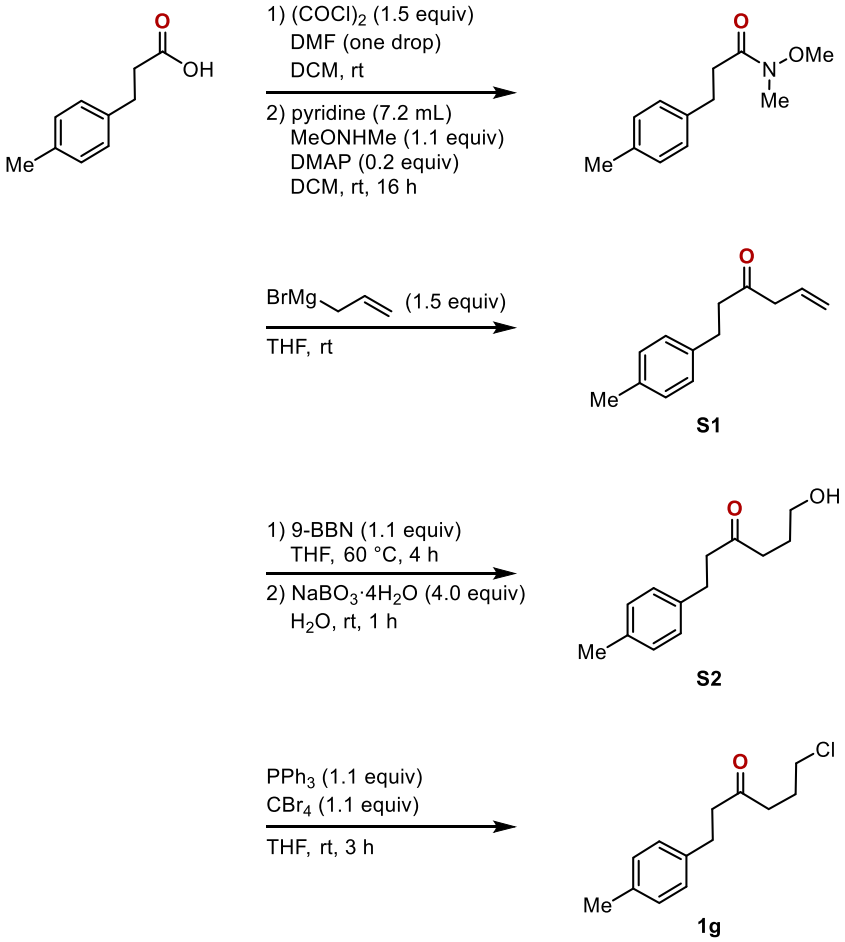


1f

1f was prepared from the corresponding alkyl halide according to the procedure for the synthesis of **1b** as a colorless oil.

^{1}H NMR (392 MHz, $CDCl_3$, δ): 0.88 (t, J = 7.0 Hz, 3H), 1.25–1.34 (m, 6H), 1.54–1.61 (m, 2H), 2.04 (quint, J = 6.6 Hz, 2H), 2.42 (t, J = 7.4 Hz, 2H), 2.61 (t, J = 7.0 Hz, 2H), 3.58 (t, J = 6.3 Hz, 2H). ^{13}C NMR (99 MHz, $CDCl_3$, δ): 14.0 (CH_3), 22.4 (CH_2), 23.8 (CH_2), 26.2 (CH_2), 28.8 (CH_2), 31.5 (CH_2), 39.1 (CH_2), 43.0 (CH_2), 44.5 (CH_2), 210.1 (C). HRMS–EI (m/z): $[M]^+$ calcd for $C_{10}H_{19}ClO$, 190.1124; found, 190.1129.

Preparation of 6-chloro-1-(4-methylphenyl)hexan-3-one (1g).



The amidation was performed according to the literature procedure.[1] In an oven-dried 100 mL round bottomed flask, 3-(*p*-tolyl)propionic acid (3.23 g, 20 mmol) was dissolved in CH_2Cl_2 (30 mL) and one

drop of DMF was added under nitrogen atmosphere. Then oxalyl chloride (3.79 g, 30 mmol) was added dropwised to the reaction mixture. The resulting mixture was stirred for 4 h at room temperature and then concentrated under reduced pressure to give the corresponding acid chloride. The reaction mixture was used for the next step without purification. The acid chloride was dissolved in CH_2Cl_2 (30 mL). After the reaction mixture was cooled to 0°C, *N,O*-dimethylhydroxylamine (2.20 g, 22 mmol), pyridine (4.8 mL) and DMAP (472.1 mg, 4 mmol) was added to the reaction mixture and the mixture was warmed to room temperature. After stirred for 15 h, the resulting suspension was quenched by the addition of 3 M HCl. The mixture was the extracted with CH_2Cl_2 three times and dried over $MgSO_4$, filtered and concentrated under reduced pressure. The crude product was used for the next step without purification.

In an oven-dried 200 mL round bottomed flask, the amide was dissolved in THF (60 mL) under nitrogen atmosphere. After the reaction mixture was cooled to 0°C, a Et_2O solution of allylmagnesium bromide (0.7 M, 34 mL, 24 mmol) was added dropwise to the reaction mixture and the mixture was warmed to room temperature. After stirred for 16 h, the resulting suspension was quenched by the addition of saturated aqueous NH_4Cl. The mixture was the extracted with EtOAc three times and dried over $MgSO_4$, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography (SiO_2, EtOAc/hexane, 0:100–4:96) to afford the corresponding ketone **S1** (3.29 g, 17 mmol, 87%) as a colorless oil.

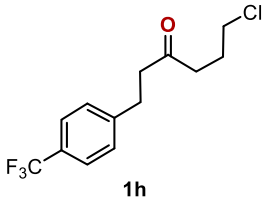
The hydroboration was performed according to the literature procedure.[2,3] In an oven-dried 200 mL round bottomed flask, **S1** (1.91 g, 10 mmol) was dissolved in THF (22 mL) under nitrogen atmosphere. After the reaction mixture was cooled to 0°C, a THF solution of 9-borabicyclo[3.3.1]nonane (0.5 M, 22 mL, 11 mmol) was added dropwised to the reaction mixture and the reaction mixture heated to 60°C. After stirred for 3 h, $NaBO_3 \cdot 4H_2O$ (6.15 g, 40 mmol) and H_2O (44 mL) were added to the mixture at 0°C. After stirred for 2 h at room temperature, the reaction mixture was extracted with CH_2Cl_2 three times and dried over $MgSO_4$, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography (SiO_2, EtOAc/hexane, 0:100–40:60) to afford the corresponding alcohol **S2** (1.29 g, 6.2 mmol, 62%) as a colorless oil.

The chlorination was performed according to the literature procedure.[4] In an oven-dried 50 mL round bottomed flask, triphenylphosphine (2.41 g, 9.3 mmol), **S2** (1.29 g, 6.2 mmol) and carbon tetrachloride (1.48 g, 9.3 mmol) were dissolved in acetonitrile (19 mL) under nitrogen atmosphere at 80°C. After stirred for 5 h, the resulting suspension was the extracted with CH_2Cl_2 three times and dried over MgSO4, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography (SiO_2, EtOAc/hexane, 0:100–2:98) to afford the corresponding ketone **1g** (491.6 mg, 2.2 mmol, 35%) as a yellow oil.

^{1}H NMR (392 MHz, $CDCl_3$, δ): 2.03 (quint, J = 6.6 Hz, 2H), 2.31 (s, 3H), 2.58 (t, J = 7.1 Hz, 2H), 2.73 (t, J = 7.6 Hz, 2H), 2.87 (t, J = 7.3 Hz, 2H), 3.55 (t, J = 6.3 Hz, 2H), 7.06–7.12 (m, 4H). ^{13}C NMR

(99 MHz, $CDCl_3$, δ): 20.9 (*C*H_3), 26.1 (*C*H_2), 29.2 (*C*H_2), 39.3 (*C*H_2), 44.3 (*C*H_2), 44.4 (*C*H_2), 128.0 (*C*H), 129.0 (*C*H), 135.5 (*C*), 137.6 (*C*), 208.7 (*C*). HRMS–EI (*m/z*): $[M]^+$ calcd for $C_{13}H_{17}ClO$, 224.0968; found, 224.0975.

Preparation of 6-chloro-1-[3-(trifluoromethyl)phenyl]hexan-3-one (1h).

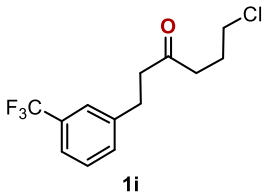


1h

1h was prepared from the corresponding calboxylic acid according to the procedure for the synthesis of **1g** as a colorless oil.

^1H NMR (392 MHz, $CDCl_3$, δ): 2.04 (quint, *J* = 6.6 Hz, 2H), 2.61 (t, *J* = 7.0 Hz, 2H), 2.78 (t, *J* = 7.6 Hz, 2H), 2.97 (t, *J* = 7.4 Hz, 2H), 3.56 (t, *J* = 6.3 Hz, 2H), 7.30 (d, *J* = 8.1 Hz, 2H), 7.54 (d, *J* = 8.1 Hz, 2H). ^{13}C NMR (99 MHz, $CDCl_3$, δ): 26.1 (*C*H_2), 29.3 (*C*H_2), 39.4 (*C*H_2), 43.7 (*C*H_2), 44.3 (*C*H_2), 124.2 (q, *J* = 273.2 Hz, *C*), 125.3 (q, *J* = 3.8 Hz, *C*H), 128.4 (q, *J* = 32.4 Hz, *C*), 128.6 (*C*H), 145.0 (*C*), 208.0 (*C*). HRMS–EI (*m/z*): $[M]^+$ calcd for $C_{13}H_{14}ClF_3O$, 278.0685; found, 278.0683.

Preparation of 6-chloro-1-[4-(trifluoromethyl)phenyl]hexan-3-one (1i).

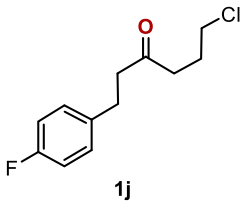


1i

1i was prepared from the corresponding alkyl halide according to the procedure for the synthesis of **1b** as a colorless oil.

^1H NMR (392 MHz, $CDCl_3$, δ): 2.05 (quint, *J* = 6.6 Hz, 2H), 2.61 (t, *J* = 7.0 Hz, 2H), 2.79 (t, *J* = 7.4 Hz, 2H), 2.97 (t, *J* = 7.4 Hz, 2H), 3.56 (t, *J* = 6.3 Hz, 2H), 7.36–7.40 (m, 2H), 7.42–7.47 (m, 2H). ^{13}C NMR (99 MHz, $CDCl_3$, δ): 26.1 (*C*H_2), 29.2 (*C*H_2), 39.3 (*C*H_2), 43.8 (*C*H_2), 44.3 (*C*H_2), 122.9 (q, *J* = 3.8 Hz, *C*H), 124.1 (q, *J* = 273.5 Hz, *C*), 124.9 (q, *J* = 3.5 Hz, *C*H), 128.8 (*C*H), 130.6 (q, *J* = 32.1 Hz, *C*), 131.8 (*C*H), 141.8 (*C*), 208.0 (*C*). HRMS–EI (*m/z*): $[M]^+$ calcd for $C_{13}H_{14}ClF_3O$, 278.0685; found, 278.0686.

Preparation of 6-chloro-1-(4-fluorophenyl)hexan-3-one (1j).



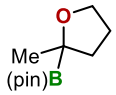
1j

1j was prepared from the corresponding alkyl halide according to the procedure for the synthesis of **1b** as a colorless oil.

^{1}H NMR (392 MHz, $CDCl_3$, δ): 2.03 (quint, J = 6.6 Hz, 2H), 2.59 (t, J = 7.0 Hz, 2H), 2.74 (t, J = 7.4 Hz, 2H), 2.88 (t, J = 7.4 Hz, 2H), 3.56 (t, J = 6.1 Hz, 2H), 6.93–6.99 (m, 2H), 7.11–7.16 (m, 2H). ^{13}C NMR (99 MHz, $CDCl_3$, δ): 26.1 (CH_2), 28.7 (CH_2), 39.4 (CH_2), 44.2 (CH_2), 44.3 (CH_2), 115.0 (*C*H), 115.2 (*C*H), 129.6 (d, J = 8.5 Hz, *C*H), 136.4 (d, J = 2.8 Hz, *C*), 161.2 (d, J = 244.5 Hz, *C*), 208.4 (*C*). HRMS–EI (*m/z*): $[M]^+$ calcd for $C_{12}H_{14}ClFO$, 228.0717; found, 228.0718.

4. Borylation Product Characterization

2-methyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)tetrahydrofuran (3a).

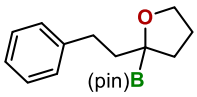


3a

The reaction was conducted with 60.5 mg (0.50 mmol) of **1a**. The product **3a** was obtained in 62% yield (64.9 mg, 0.31 mmol) as a yellow oil.

^{1}H NMR (401 MHz, $CDCl_3$, δ): 1.21 (s, 3H), 1.27 (s, 12H), 1.56–1.65 (m, 1H), 1.82–1.89 (m, 1H), 1.91–2.02 (m, 2H), 3.78–3.91 (m, 2H). ^{13}C NMR (99 MHz, $CDCl_3$, δ): 22.9 (*C*H_3), 24.5 (*C*H_3), 25.8 (*C*H_2), 35.0 (*C*H_2), 67.1 (*C*H_2), 83.7 (*C*). HRMS–EI (*m/z*): $[M–CH_3]^+$ calcd for $C_{10}H_{18}{}^{11}BO_3$, 197.0351; found, 197.1352.

2-phenethyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)tetrahydrofuran (3b).

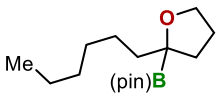


3b

The reaction was conducted with 103.0 mg (0.50 mmol) of **1b**. The product **3b** was obtained in 54% yield (80.6 mg, 0.27 mmol) as a colorless oil.

^{1}H NMR (392 MHz, $CDCl_3$, δ): 1.29 (s, 12H), 1.65–1.72 (m, 1H), 1.76–2.03 (m, 5H), 2.52–2.62 (m, 1H), 2.76–2.84 (m, 1H), 3.87 (t, J = 7.0 Hz, 2H), 7.15–7.29 (m, 5H). ^{13}C NMR (99 MHz, $CDCl_3$, δ): 24.5 (*C*H_3), 24.7 (*C*H_3), 25.8 (*C*H_2), 32.3 (*C*H_2), 34.0 (*C*H_2), 39.5 (*C*H_2), 67.1 (*C*H_2), 83.8 (*C*), 125.5 (*C*H), 128.1 (*C*H), 128.2 (*C*H), 142.8 (*C*). HRMS–ESI (*m/z*): $[M+Na]^+$ calcd for $C_{18}H_{27}{}^{11}BO_3Na$, 325.1949; found, 325.1952.

2-hexyl-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)tetrahydrofuran (3f).

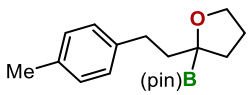


3f

The reaction was conducted with 190.7 mg (0.50 mmol) of **1f**. The product **3f** was obtained in 36% yield (49.9 mg, 0.18 mmol) as a colorless oil.

^{1}H NMR (392 MHz, $CDCl_3$, δ): 0.85–0.89 (m, 3H), 1.27 (s, 20H), 1.40–1.55 (m, 3H), 1.77–1.85 (m, 1H), 1.87–2.00 (m, 2H), 3.82 (t, J = 6.9 Hz, 2H). ^{13}C NMR (99 MHz, $CDCl_3$, δ): 14.0 (*C*H_3), 22.5 (*C*H_2), 24.6 (*C*H_3), 24.6 (*C*H_3), 25.9 (*C*H_2), 26.0 (*C*H_2), 29.8 (*C*H_2), 31.7 (*C*H_2), 34.1 (*C*H_2), 37.6 (*C*H_2), 67.0 (*C*H_2), 83.7 (*C*). HRMS–EI (*m/z*): $[M–CH_3]^+$ calcd for $C_{15}H_{28}{}^{11}BO_3$, 267.2134; found, 267.2133.

2-(4'-methylphenethyl)- 2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)tetrahydrofuran (3g).

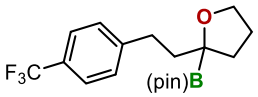


3g

The reaction was conducted with 114.3 mg (0.50 mmol) of **1g**. The product **3g** was obtained in 59% yield (94.0 mg, 0.30 mmol) as a colorless oil.

^{1}H NMR (392 MHz, $CDCl_3$, δ): 1.29 (s, 12H), 1.64–1.70 (m, 1H), 1.77–2.02 (m, 5H), 2.31 (s, 3H), 2.48–2.56 (m, 1H), 2.69–2.81 (m, 1H), 3.86 (t, *J* = 6.7 Hz, 2H), 7.06–7.11 (m, 4H). ^{13}C NMR (99 MHz, $CDCl_3$, δ): 20.9 (*C*H_3), 24.5 (*C*H_3), 24.7 (*C*H_3), 25.8 (*C*H_2), 31.9 (*C*H_2), 34.0 (*C*H_2), 39.6 (*C*H_2), 67.1 (*C*H_2), 75.2 (brs, B–*C*), 83.8 (*C*), 128.1 (*C*H), 128.8 (*C*H), 134.8 (*C*), 139.7 (*C*). HRMS–EI (*m/z*): $[M]^+$ calcd for $C_{19}H_{29}{}^{11}BO_3$, 316.2213; found, 316.2212.

2-[4'-(trifluoromethyl)phenethyl]-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)tetrahydrofuran (3h).

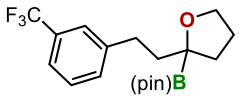


3h

The reaction was conducted with 138.8 mg (0.50 mmol) of **1h**. The product **3h** was obtained in 46% yield (84.3 mg, 0.23 mmol) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 1.29 (s, 6H), 1.29 (s, 6H), 1.64–1.72 (m, 1H), 1.74–2.04 (m, 5H), 2.62 (dt, *J* = 5.2, 12.7 Hz, 1H), 2.85 (dt, *J* = 5.5, 12.9 Hz, 1H), 3.85–3.89 (m, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 7.51 (d, *J* = 7.6 Hz, 2H). ^{13}C NMR (99 MHz, $CDCl_3$, δ): 24.6 (*C*H_3), 24.7 (*C*H_3), 25.8 (*C*H_2), 32.3 (*C*H_2), 34.2 (*C*H_2), 39.2 (*C*H_2), 67.3 (*C*H_2), 75.0 (brs, B–*C*), 84.0 (*C*), 124.4 (q, *J* = 272.9 Hz, *C*), 125.1 (q, *J* = 3.8 Hz, *C*H), 127.9 (q, *J* = 32.1 Hz, *C*), 128.6 (*C*H), 147.0 (*C*). HRMS–ESI (*m/z*): $[M+Na]^+$ calcd for $C_{19}H29^{11}BF_3O_3Na$, 393.1823; found, 393.1821.

2-[3'-(trifluoromethyl)phenethyl]-2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)tetrahydrofuran (3i).



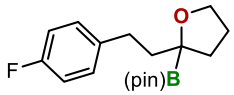
3i

The reaction was conducted with 122.7 mg (0.50 mmol) of **1i**. The product **3i** was obtained in 63% yield (102.4 mg, 0.28 mmol) as a colorless oil. This product contains small amount of unremovable impurities.

^{1}H NMR (392 MHz, $CDCl_3$, δ): 1.29 (s, 12H), 1.62–1.73 (m, 1H), 1.75–2.04 (m, 5H), 2.58–2.66 (m, 1H), 2.81–2.89 (m, 1H), 3.87 (td, *J* = 2.2, 6.8 Hz, 2H), 7.26–7.47 (m, 4H). ^{13}C NMR (99 MHz, $CDCl_3$,

δ): 24.5 (*C*H$_3$), 24.7 (*C*H$_3$), 25.8 (*C*H$_2$), 32.2 (*C*H$_2$), 34.1 (*C*H$_2$), 39.3 (*C*H$_2$), 67.3 (*C*H$_2$), 83.9 (*C*), 122.4 (q, *J* = 6.6 Hz, *C*H), 124.2 (q, *J* = 273.5 Hz, *C*), 124.9 (q, *J* = 3.8 Hz, *C*H), 128.6 (*C*H), 130.4 (q, *J* = 31.8 Hz, *C*), 131.8 (*C*H), 143.7 (*C*). HRMS–ESI (*m/z*): [M+Na]$^+$ calcd for $C_{19}H_{26}{}^{11}BF_3O_3$, 393.1823; found, 393.1830.

2-(4'-fluorophenethyl)- 2-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)tetrahydrofuran (3j).



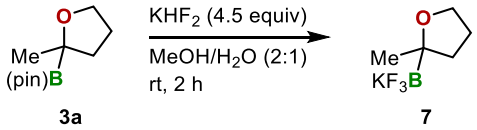
3j

The reaction was conducted with 122.9 mg (0.50 mmol) of **1j** product **3j** was obtained in 47% yield (75.1 mg, 0.24 mmol) as a colorless oil.

^{1}H NMR (392 MHz, $CDCl_3$, δ): 1.29 (s, 12H), 1.64–1.70 (m, 1H), 1.72–2.03 (m, 5H), 2.49–2.567 (m, 1H), 2.72–2.80 (m, 1H), 3.81–3.90 (m, 2H), 6.94 (tt, *J* = 5.4, 7.6 Hz, 2H), 7.11–7.16 (m, 2H). ^{13}C NMR (99 MHz, $CDCl_3$, δ): 24.5 (*C*H$_3$), 24.7 (*C*H$_3$), 25.8 (*C*H$_2$), 31.6 (*C*H$_2$), 34.1 (*C*H$_2$), 39.7 (*C*H$_2$), 67.2 (*C*H$_2$), 75.5 (brs, B–*C*), 83.9 (*C*), 114.8 (d, *J* = 20.8 Hz, *C*H), 129.5 (d, *J* = 7.6 Hz, *C*H), 138.4 (*C*), 161.0 (d, *J* = 243.6 Hz, *C*). HRMS–EI (*m/z*): [M–CH$_3$]$^+$ calcd for $C_{17}H_{23}{}^{11}BFO_3$, 305.1727; found, 305.1722.

5. Borylation Product Functionalization Procedure

Procedure for the synthesis of trifluoroborate salt 7.

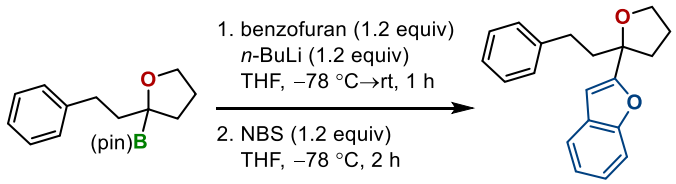


3a **7**

KHF_2 (351.5 mg, 4.5 mmol) was placed in an oven-dried reaction vial. After the vial was sealed with a screw cap containing a teflon-coated rubber septum, the vial was connected to a vacuum/nitrogen manifold through a needle. It was evacuated and then backfilled with nitrogen. This cycle was repeated three times. MeOH (2.0 mL) was added in the vial through the rubber septum using a syringe. After **3a** (212.1 mg, 1.0 mmol) was added to the mixture at room temperature, H_2O (1.0 mL) was added dropwise. After 2 h, the reaction mixture was concerted in vacuo. The resulting solid was filtered with MeOH. The filtrate was concerted in vacuo. The resulting solid was washed with Et_2O to give **7** (132.5 mg, 0.69 mmol, 69%) as a white powder.

^{1}H NMR (396 MHz, CD_3CN, δ): 0.88 (s, 3H), 1.25–1.32 (m, 1H), 1.68–1.87 (m, 3H), 3.55–3.61 (m, 1H), 3.63–3.70 (m, 1H). ^{13}C NMR (99 MHz, DMSO-d_6, δ): 23.6 (*C*H_3), 26.1 (*C*H_2), 34.2 (*C*H_2), 65.4 (*C*H_2). ^{11}B NMR (127 MHz, CD_3CN, δ): 4.30 (q, J = 44.8 Hz). ^{19}F NMR (373 MHz, CD_3CN, δ): –151.3 (q, J = 56.9 Hz). HRMS–ESI (*m/z*): $[M–K]^-$ calcd for $C_5H_9{}^{10}BF_3O$, 152.0740; found, 152.0741.

Procedure for the synthesis of arylated product 8 through the stereospecific cross-coupling of 3b with benzofuran.

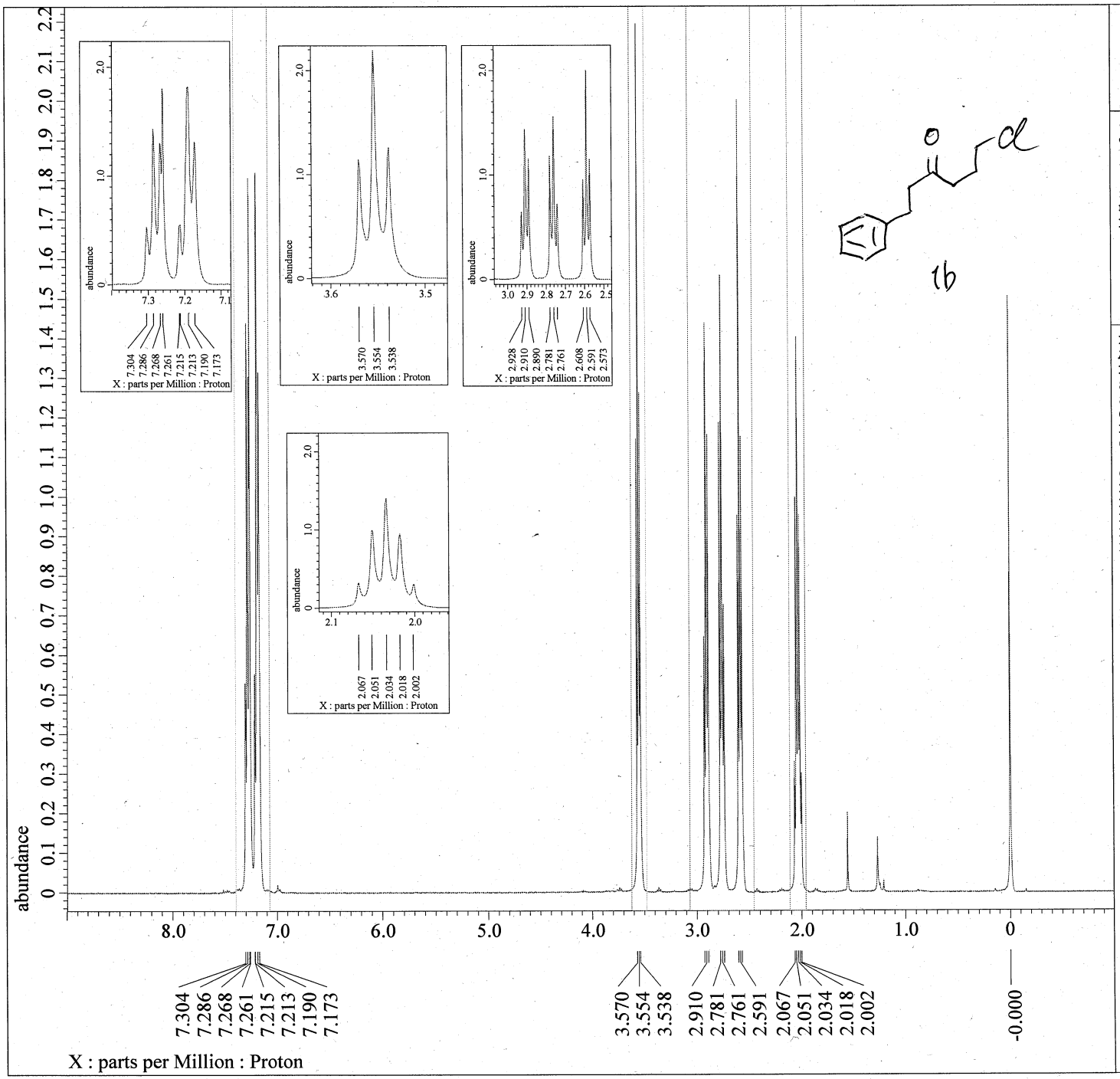


3b **8**

The stereospecific cross-coupling was performed according to the literature.[5] A solution of benzofuran (18.4 mg, 0.24 mmol) in THF (800 µL) was cooled to –78°C and treated with *n*-BuLi in hexane (1.57 M, 153 µL, 0.24 mmol). The reaction mixture was warmed to room temperature and stirred for 1 h. The mixture was then cooled to –78°C and the **3b** (60.4 mg, 0.2 mmol) was added to the mixture as a solution in THF (400 µL) and the reaction stirred at the same temperature for 1 h. A solution of NBS (42.7 mg, 0.24 mmol) in THF (800 µL) was then added dropwise to the mixture. After 2 h at –78°C, aqueous $Na_2S_2O_3$ was added and the reaction mixture was allowed to warm to room temperature. The mixture was extracted three times with Et_2O, dried over $MgSO_4$, and filtered. The resulting crude product was purified by flash column chromatography (SiO_2, Et_2O/hexane, 0:100–2:98) to give the corresponding arylated product **8** (10.1 mg, 0.03 mmol, 17% yield) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$, δ): 1.94–2.07 (m, 3H), 2.17–2.24 (m, 1H), 2.34 (td, J = 4.6, 12.7 Hz, 1H), 2.38–2.43 (m, 1H), 2.48 (td, J = 4.7, 12.8 Hz, 1H), 2.71 (td, J = 4.7, 12.7 Hz, 1H), 4.03 (t, J = 6.5 Hz, 2H), 6.65 (d, J = 0.8 Hz, 1H), 7.13–7.16 (m, 3H), 7.19–7.27 (m, 4H), 7.46 (d, J = 7.8 Hz, 1H), 7.56–7.55 (m, 1H). ^{13}C NMR (99 MHz, $CDCl_3$, δ): 25.7 (CH_2), 30.9 (CH_2), 36.2 (CH_2), 41.3 (CH_2), 68.4 (CH_2), 83.5 (*C*), 102.6 (*C*H), 111.1 (*C*H), 120.7 (*C*H), 122.6 (*C*H), 123.6 (*C*H), 125.7 (*C*H), 128.3 (*C*H), 128.4 (*C*), 142.1 (*C*), 155.0 (*C*), 160.7 (*C*). HRMS–EI (*m/z*): $[M]^+$ calcd for $C_{20}H_{20}O_2$, 292.1463; found, 292.1464.

6. References

1) Wang, Y.; Zarca, M.; Gong, L.–Z.; Zhang, L. *J. Am. Chem. Soc.* **2016**, *138*, 7516.
2) Organ, M. G.; Ghasemt, H. *J. Org. Chem.* **2004**, *69*, 695.
3) Kubota, K.; Watanabe, Y.; Hayama, K.; Ito, H. *J. Am. Chem. Soc.* **2016**, *138*, 4338.
4) Kalkeren, H. A.; Delft, F. L.; Rutjes, F. P. J. T. *Pure Appl. Chem.* **2012**, *85*, 817.
5) Hilt, G.; Bolze, P.; Harms, K. *Chem. Eur. J.* **2007**, *13*, 4312.



JEOL RESONANCE

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

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Author	= element
Experiment	= proton.jxp
Sample_Id	= UES-051-pureH
Solvent	= CHLOROFORM-D
Creation_Time	= 26-JAN-2019 13:36:59
Revision_Time	= 22-APR-2019 16:50:03
Current_Time	= 22-APR-2019 16:51:24
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Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
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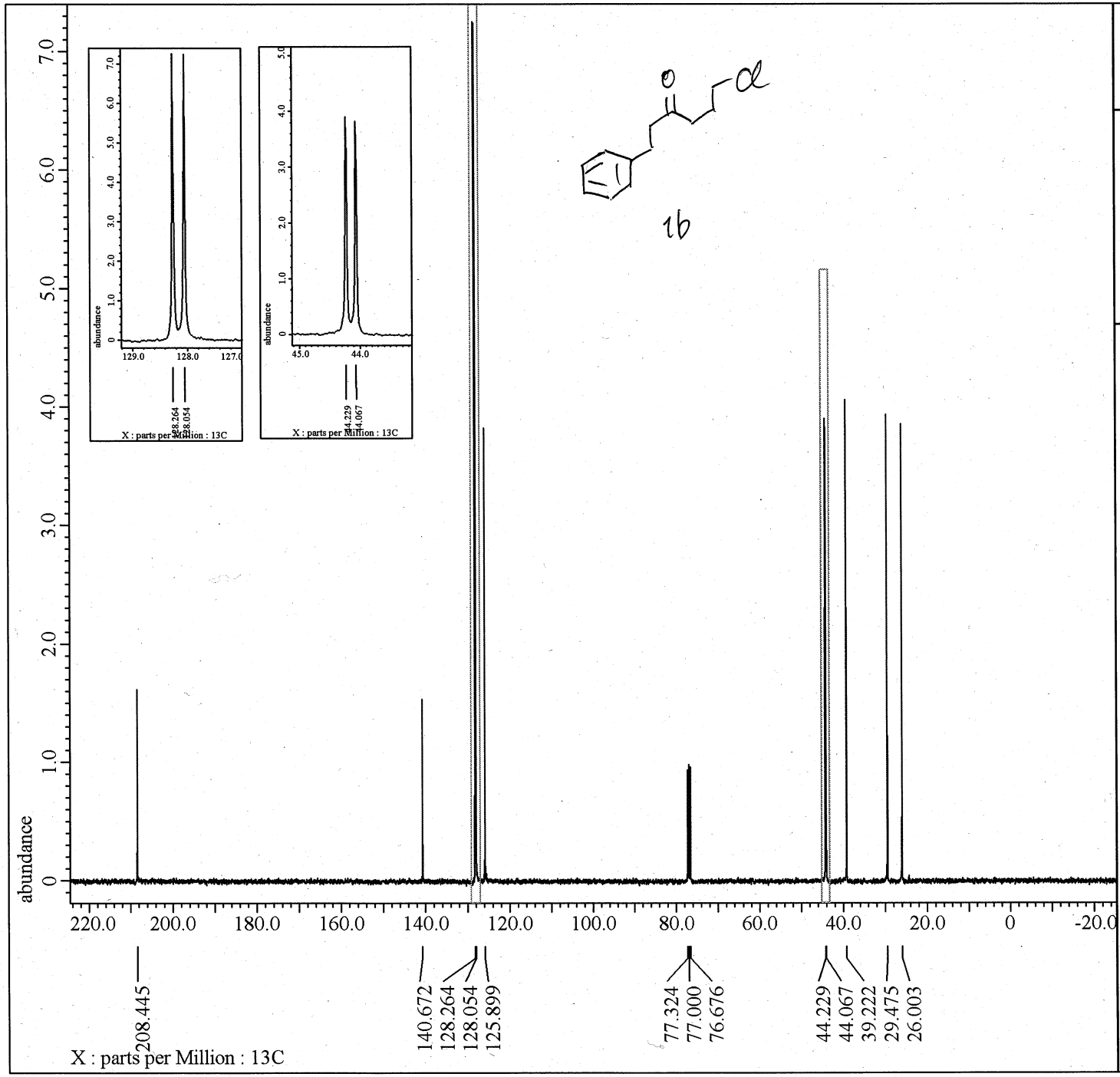
X_Acq_Duration	= 2.18103808[s]
X_Domain	= 1H
X_Freq	= 400.53219825[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45849727[Hz]
X_Sweep	= 7.51201923[kHz]
X_Sweep_Clipped	= 6.00961538[kHz]
Irr_Domain	= Proton
Irr_Freq	= 400.53219825[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 400.53219825[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 44
Temp_Get	= 18[dC]
X_90_Width	= 6[us]
X_Acq_Time	= 2.18103808[s]
X_Angle	= 45[deg]
X_Atn	= 0.8[dB]
X_Pulse	= 3[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE



JEOL RESONANCE

```
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dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: UES-051-gpcC-2.jdf
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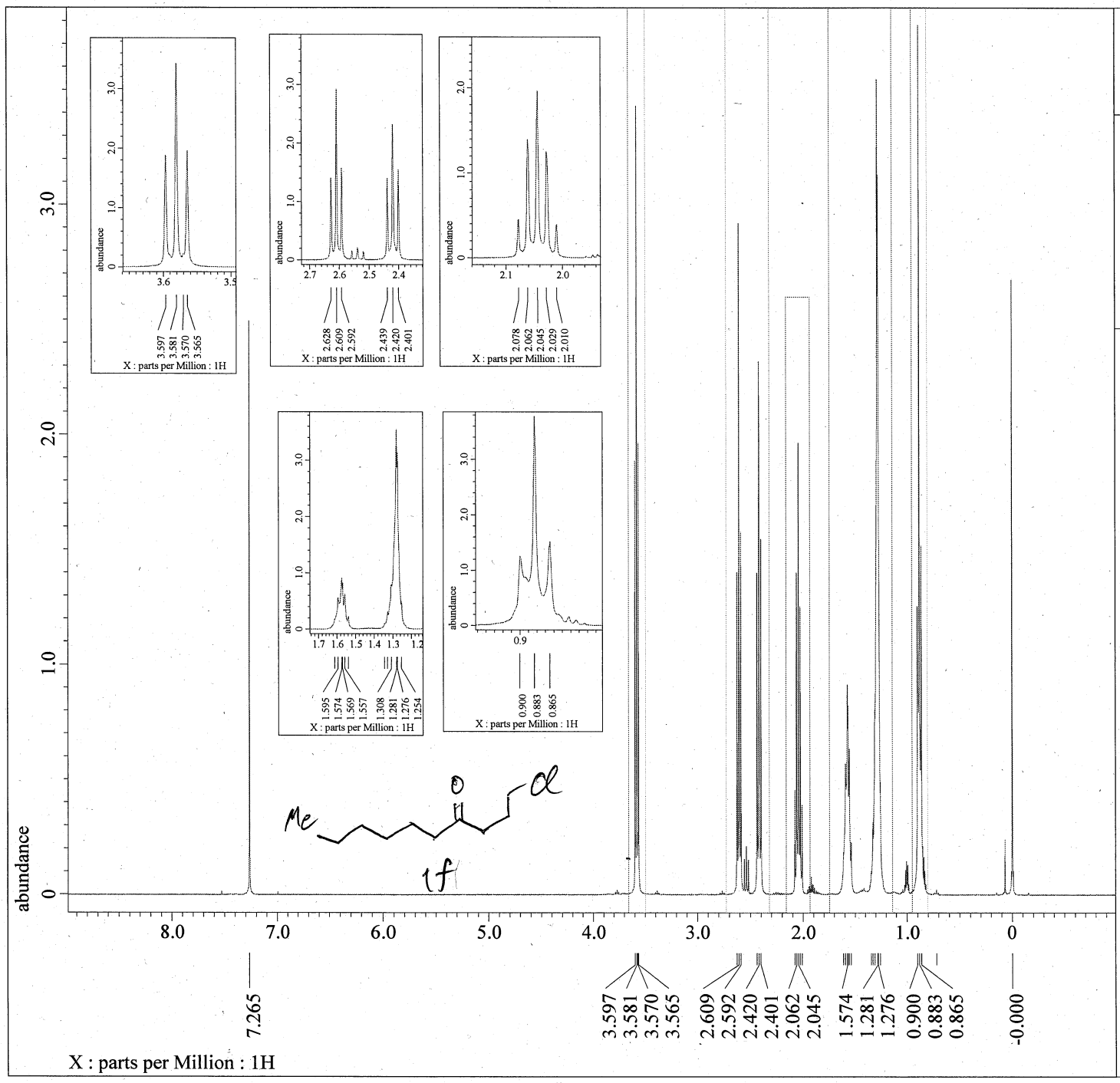
Author	= element
Experiment	= single_pulse_dec
Sample_Id	= S#494988
Solvent	= CHLOROFORM-D
Creation_Time	= 12-JUL-2018 12:03:09
Revision_Time	= 22-APR-2019 16:32:31
Current_Time	= 22-APR-2019 16:33:05
Comment	= single pulse decoupled g
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 1.06430464[s]
X_Domain	= 13C
X_Freq	= 98.51479726[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.93958061[Hz]
X_Sweep	= 30.78817734[kHz]
Irr_Domain	= 1H
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 50
Total_Scans	= 50
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 20[dC]
X_90_Width	= 9.11[us]
X_Acq_Time	= 1.06430464[s]
X_Angle	= 30[deg]
X_Atn	= 4.9[dB]
X_Pulse	= 3.03666667[us]
Irr_Atn_Dec	= 22.255[dB]
Irr_Atn_Noe	= 22.255[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: UES-047-pureH-3.jdf
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Filename	= UES-047-pureH-5.jdf
Author	= element
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Solvent	= CHLOROFORM-D
Creation_Time	= 25-JAN-2019 12:28:07
Revision_Time	= 22-APR-2019 16:45:46
Current_Time	= 22-APR-2019 16:48:01
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Dim_Size	= 13107
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 2.228224[s]
X_Domain	= 1H
X_Freq	= 391.78655441[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.44878791[Hz]
X_Sweep	= 7.35294118[kHz]
Irr_Domain	= 1H
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 391.78655441[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 44
Temp_Get	= 18.4[dC]
X_90_Width	= 11.04[us]
X_Acq_Time	= 2.228224[s]
X_Angle	= 45[deg]
X_Atn	= 1.9[dB]
X_Pulse	= 5.52[us]
Irr_Mode	= Off
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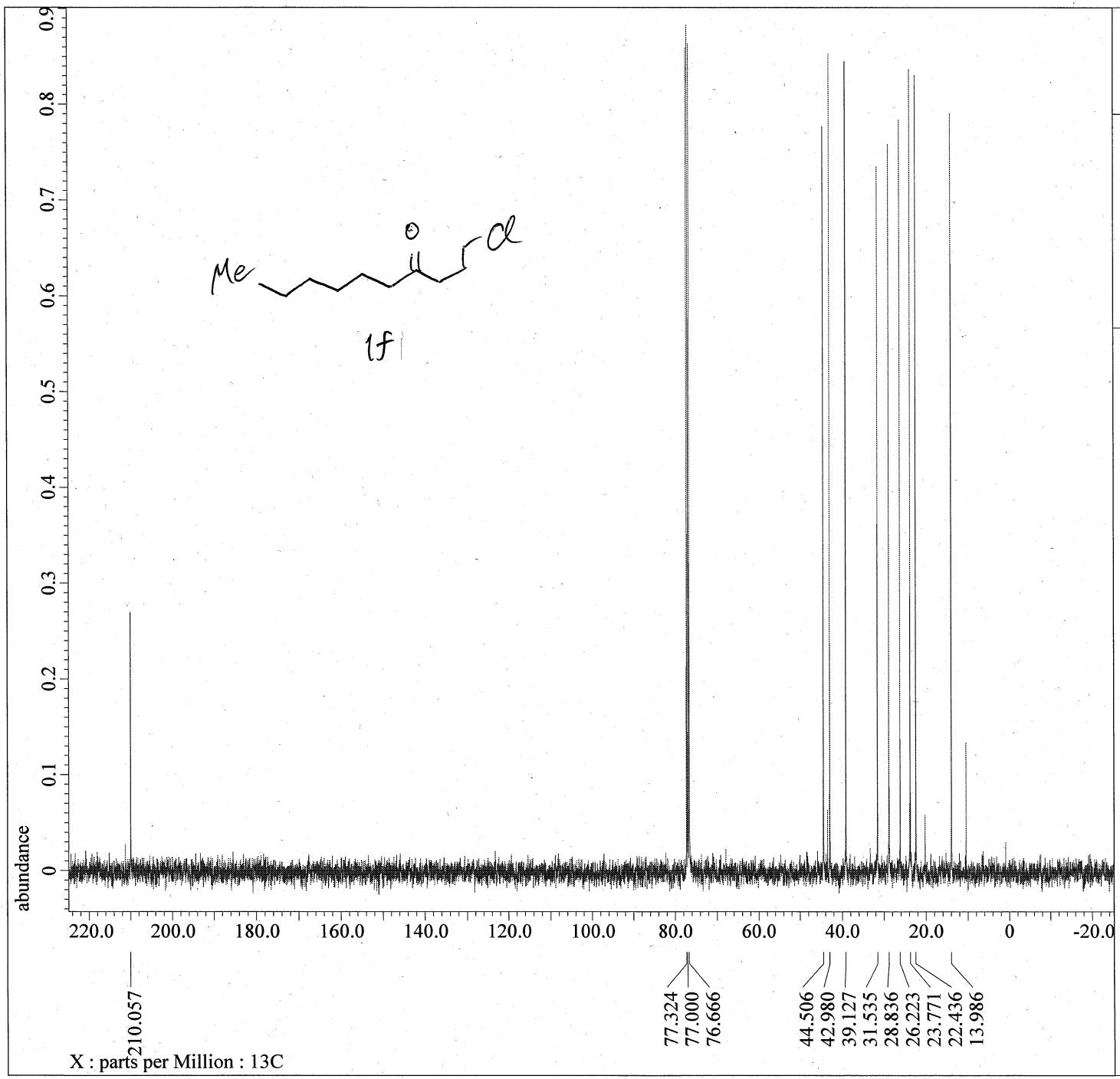
Dante_Presat	= FALSE



JEOL RESONANCE

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---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: UES-047-pureC-2.jdf
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Filename	= UES-047-pureC-4.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_Id	= S#534167
Solvent	= CHLOROFORM-D
Creation_Time	= 25-JAN-2019 13:22:30
Revision_Time	= 22-APR-2019 16:41:08
Current_Time	= 22-APR-2019 16:41:54
Comment	= single pulse decoupled g
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 1.06430464[s]
X_Domain	= 13C
X_Freq	= 98.51479726[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.93958061[Hz]
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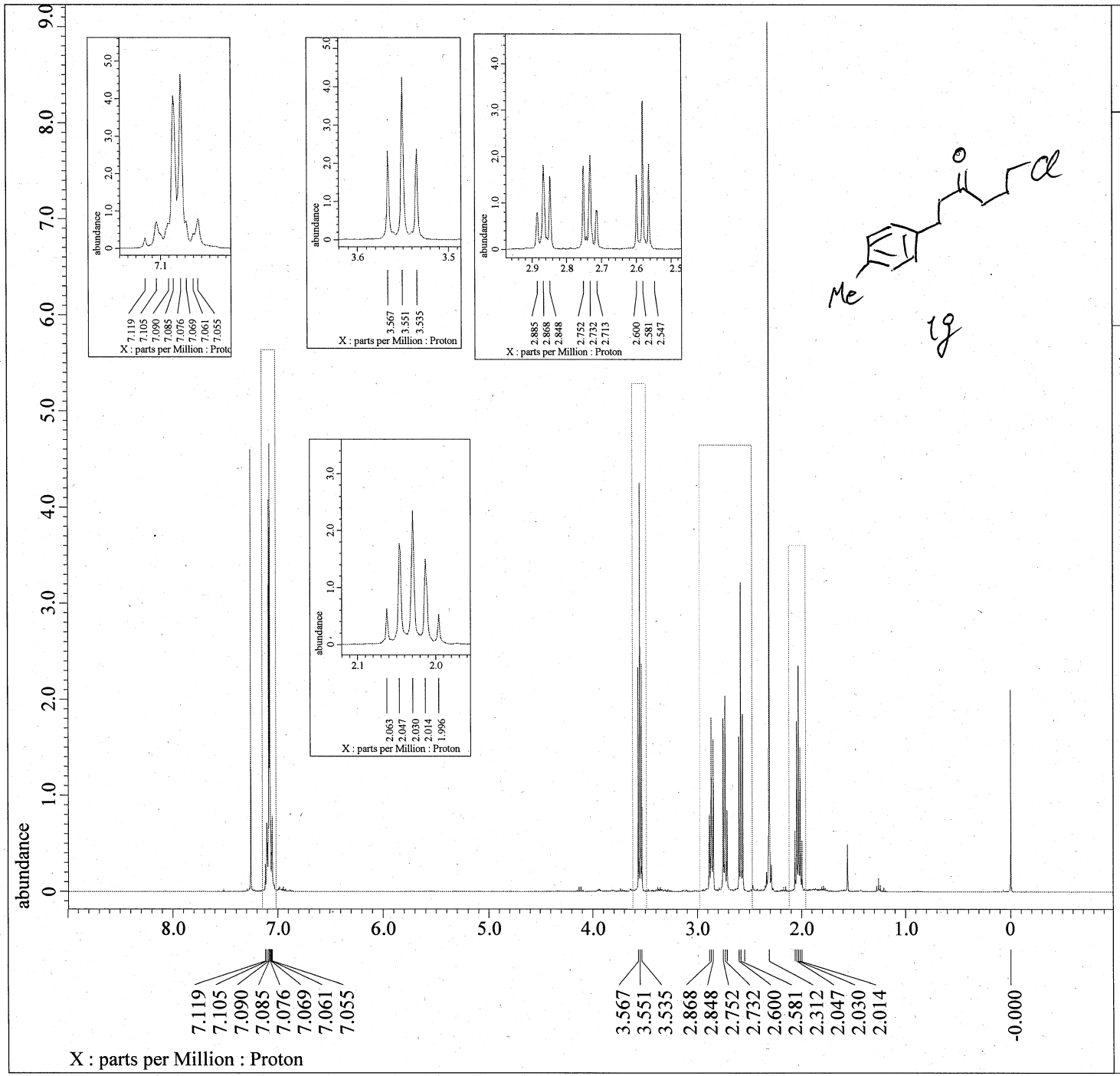

JEOL RESONANCE

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

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Revision_Time	= 22-APR-2019 17:22:05
Current_Time	= 22-APR-2019 17:22:42
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Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.4073814[T] (400[MHz])
X_Acq_Duration	= 2.18103808[s]
X_Domain	= 1H
X_Freq	= 400.53219825[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45849727[Hz]
X_Sweep	= 7.51201923[kHz]
X_Sweep_Clipped	= 6.00961538[kHz]
Irr_Domain	= Proton
Irr_Freq	= 400.53219825[MHz]
Irr_Offset	= 5[ppm]
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Tri_Offset	= 5[ppm]
Clipped	= FALSE
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Relaxation_Delay	= 5[s]
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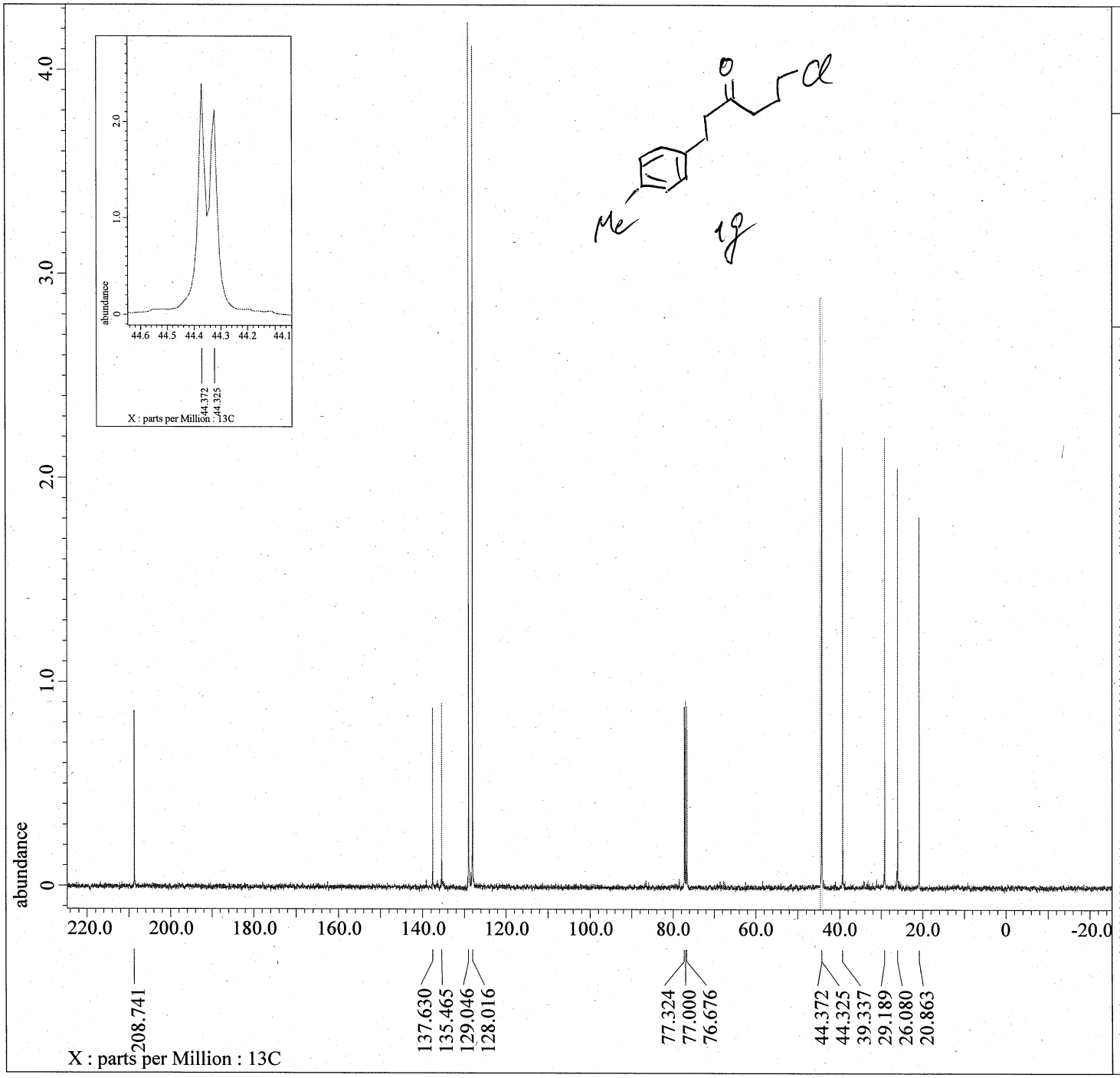
Dante_Presat	= FALSE



JEOL RESONANCE

```
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fft( 1, TRUE, TRUE )
machinephase
ppm

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Dim_Units	= [ppm]
Dimensions	= X
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Spectrometer	= JNM-ECS400
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X_Acq_Duration	= 1.06430464[s]
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X_Freq	= 98.51479726[MHz]
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Irr_Offset	= 5[ppm]
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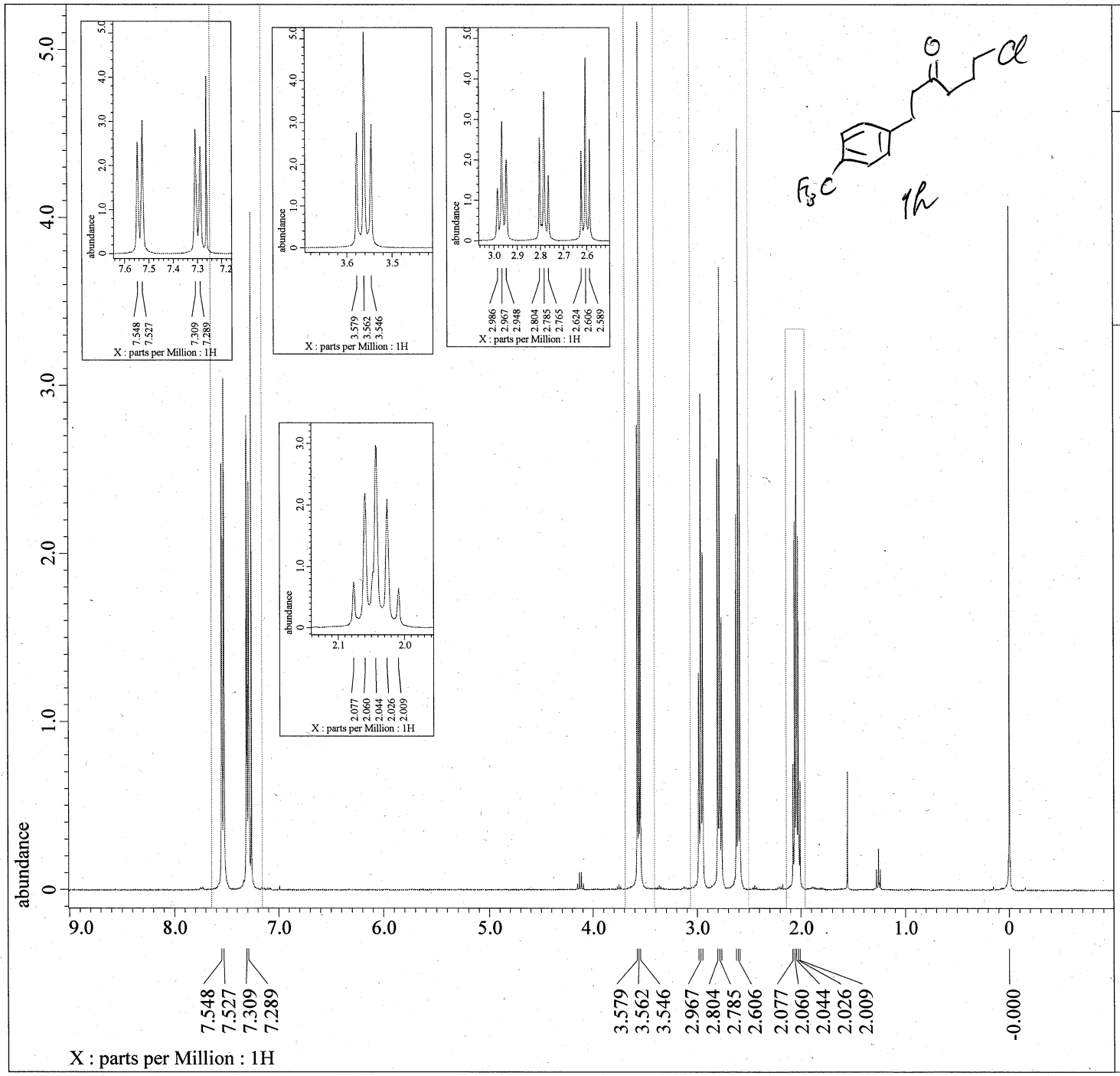
Recvr_Gain	= 60
Temp_Get	= 18.8[dC]
X_90_Width	= 9.11[us]
X_Acq_Time	= 1.06430464[s]
X_Angle	= 30[deg]
X_Atn	= 4.9[dB]
X_Pulse	= 3.03666667[us]
Irr_Atn_Dec	= 22.255[dB]
Irr_Atn_Noe	= 22.255[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE

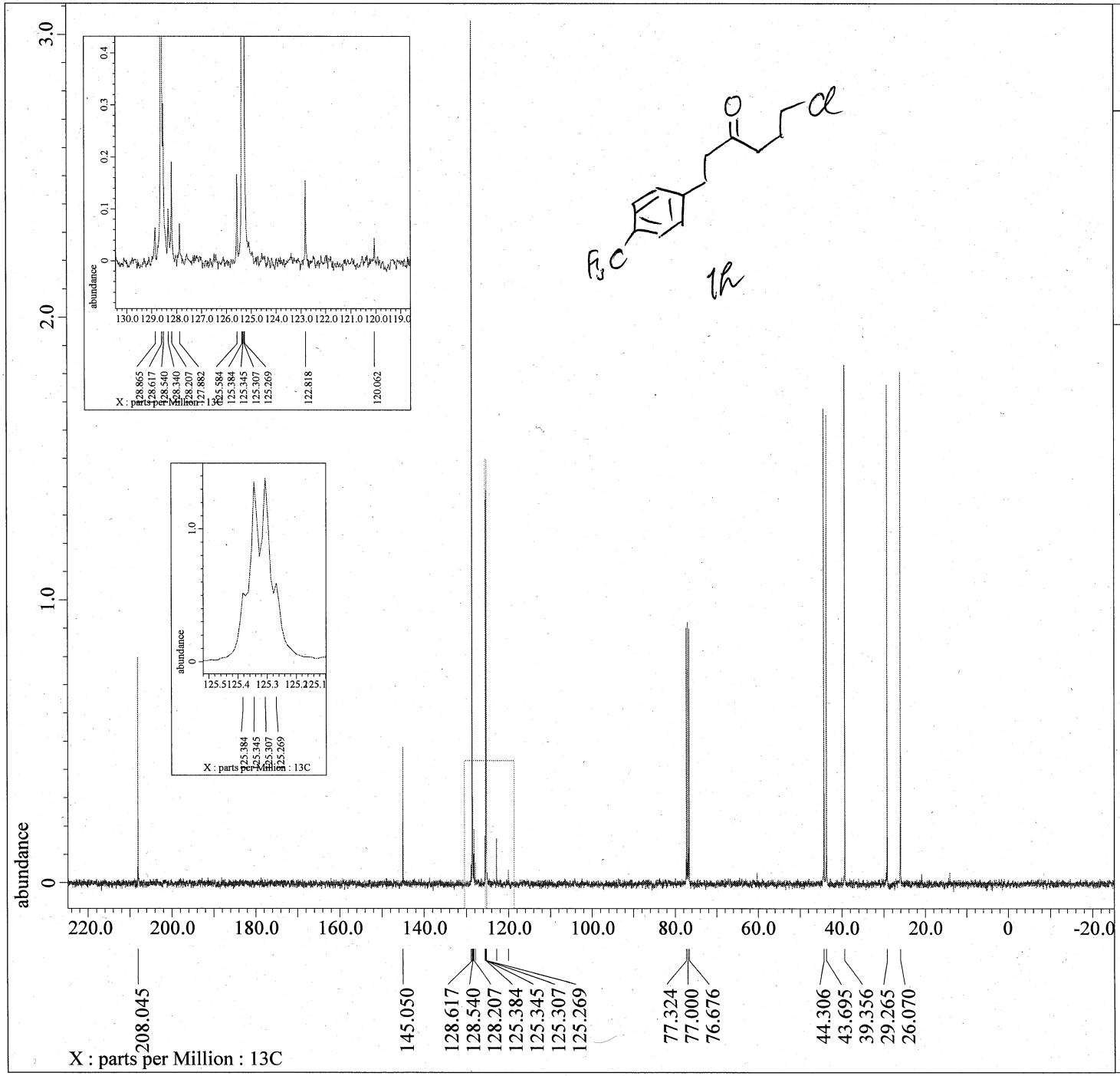


JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: UES-091-pureH-2.jdf
```

Filename	= UES-091-pureH-4.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_Id	= S#507482
Solvent	= CHLOROFORM-D
Creation_Time	= 25-JAN-2019 12:34:49
Revision_Time	= 22-APR-2019 20:55:14
Current_Time	= 22-APR-2019 20:56:06
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 2.228224[s]
X_Domain	= 1H
X_Freq	= 391.78655441[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.44878791[Hz]
X_Sweep	= 7.35294118[kHz]
Irr_Domain	= 1H
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 391.78655441[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 50
Temp_Get	= 18.8[dC]
X_90_Width	= 11.04[us]
X_Acq_Time	= 2.228224[s]
X_Angle	= 45[deg]
X_Atn	= 1.9[dB]
X_Pulse	= 5.52[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE

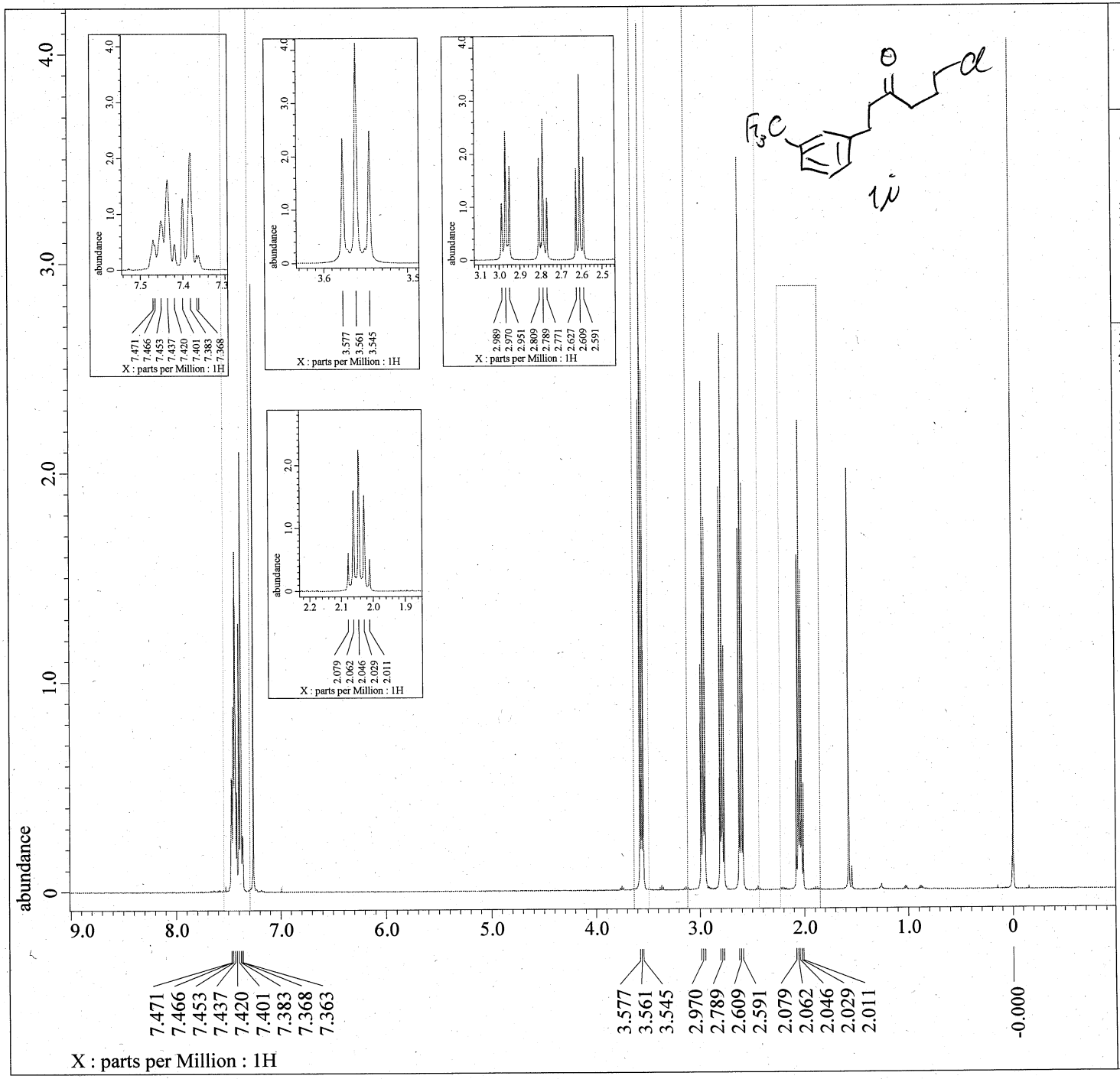


JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: UES-091-pureC-2.jdf
```

Filename	= UES-091-pureC-4.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_Id	= S#542099
Solvent	= CHLOROFORM-D
Creation_Time	= 25-JAN-2019 13:35:44
Revision_Time	= 22-APR-2019 20:57:41
Current_Time	= 22-APR-2019 21:03:40
Comment	= single pulse decoupled g
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 1.06430464[s]
X_Domain	= 13C
X_Freq	= 98.51479726[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.93958061[Hz]
X_Sweep	= 30.78817734[kHz]
Irr_Domain	= 1H
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 100
Total_Scans	= 100
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 19.1[dC]
X_90_Width	= 9.11[us]
X_Acq_Time	= 1.06430464[s]
X_Angle	= 30[deg]
X_Atn	= 4.9[dB]
X_Pulse	= 3.03666667[us]
Irr_Atn_Dec	= 22.255[dB]
Irr_Atn_Noe	= 22.255[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE

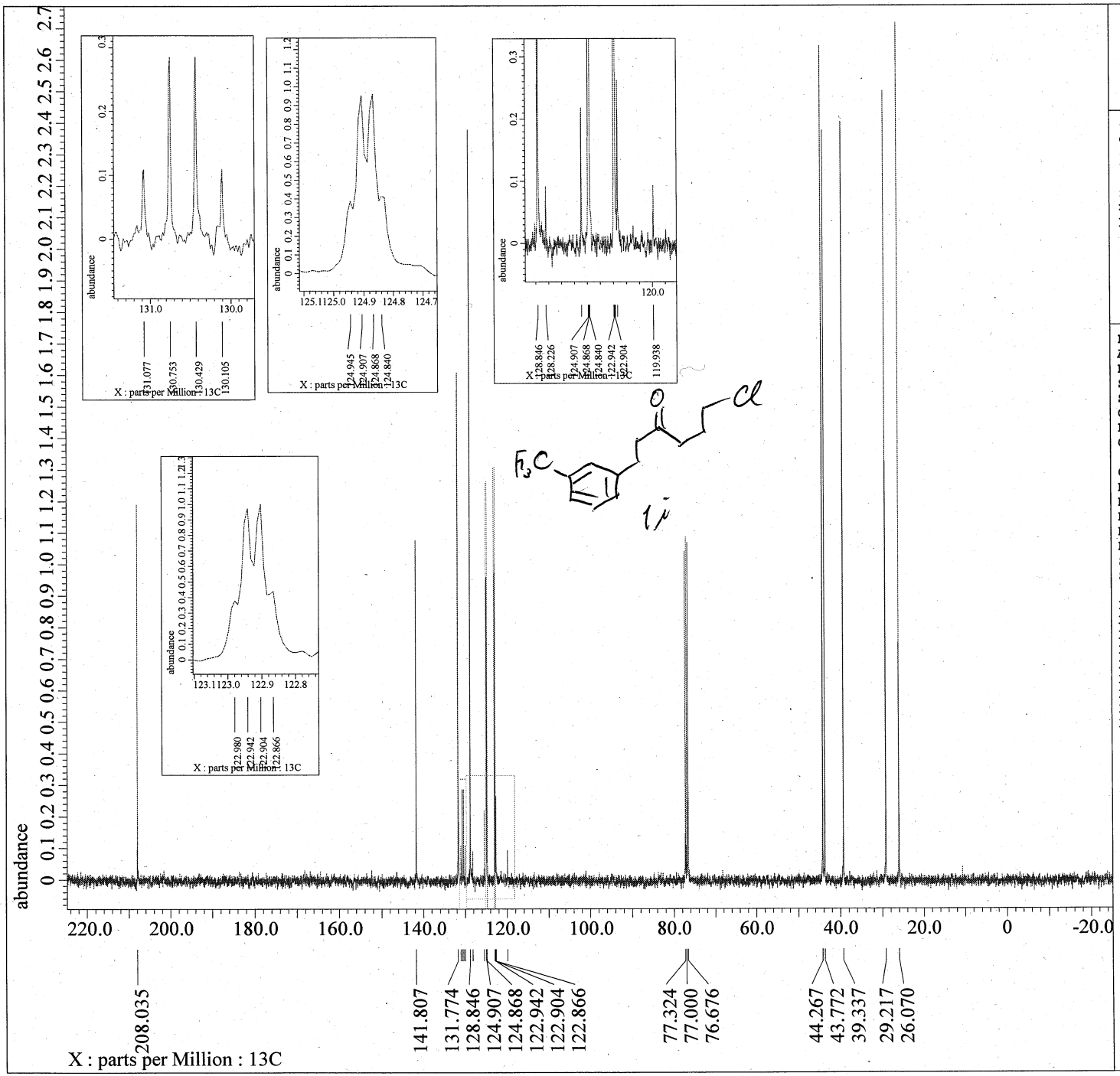


JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: UES-061-gpcH-2.jdf
```

Filename	= UES-061-gpcH-4.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_Id	= S#335478
Solvent	= CHLOROFORM-D
Creation_Time	= 19-JUL-2018 07:35:50
Revision_Time	= 22-APR-2019 21:07:03
Current_Time	= 22-APR-2019 21:07:54
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 2.228224[s]
X_Domain	= 1H
X_Freq	= 391.78655441[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.44878791[Hz]
X_Sweep	= 7.35294118[kHz]
Irr_Domain	= 1H
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 391.78655441[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 44
Temp_Get	= 19.7[dC]
X_90_Width	= 11.04[us]
X_Acq_Time	= 2.228224[s]
X_Angle	= 45[deg]
X_Atn	= 1.9[dB]
X_Pulse	= 5.52[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE

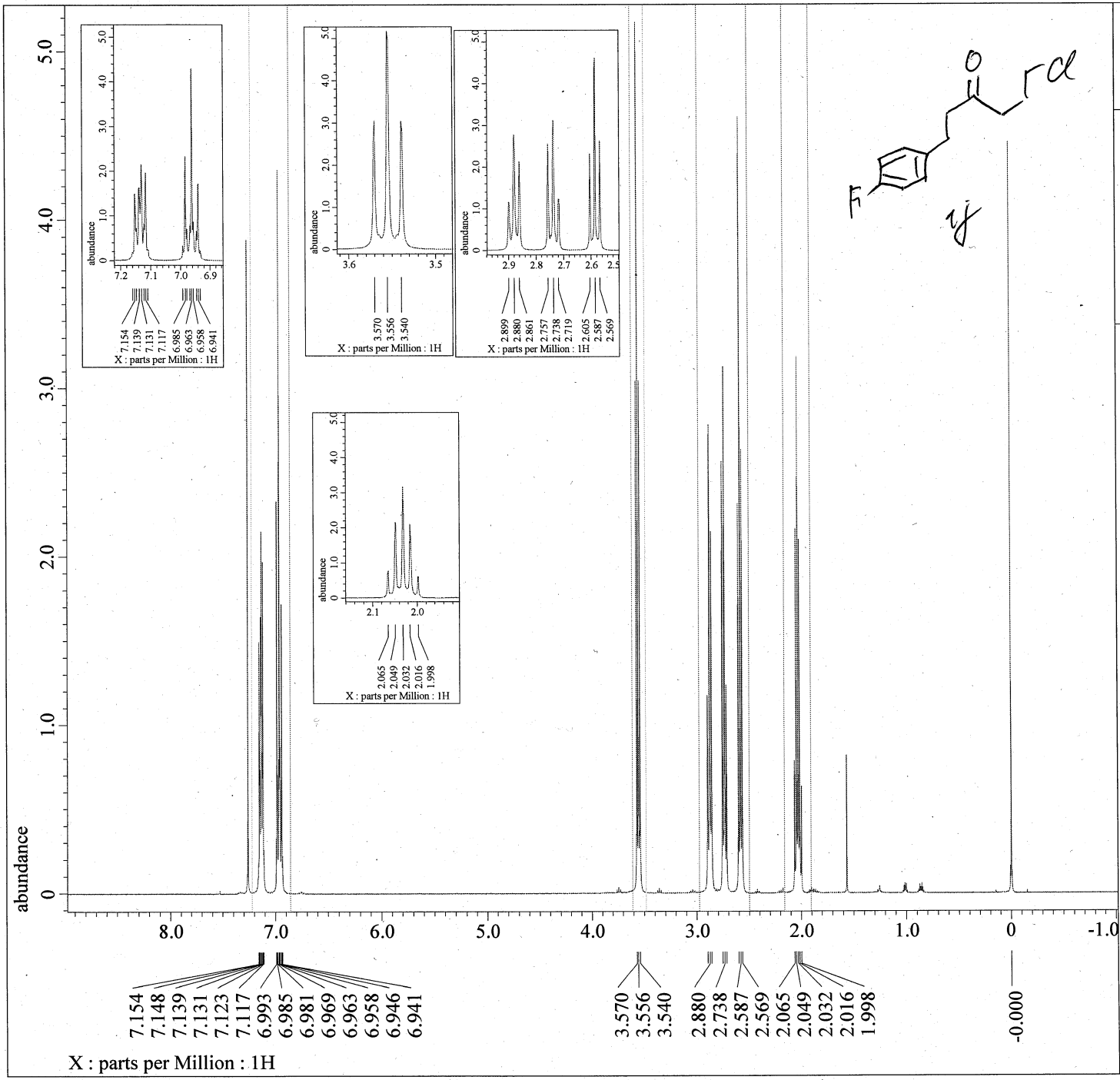


JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: UES-061-gpcC-2.jdf
```

Filename	= UES-061-gpcC-4.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_Id	= S#350686
Solvent	= CHLOROFORM-D
Creation_Time	= 19-JUL-2018 08:01:42
Revision_Time	= 22-APR-2019 21:11:32
Current_Time	= 22-APR-2019 21:13:29
Comment	= single pulse decoupled g
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 1.06430464[s]
X_Domain	= 13C
X_Freq	= 98.51479726[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.93958061[Hz]
X_Sweep	= 30.78817734[kHz]
Irr_Domain	= 1H
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 50
Total_Scans	= 50
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 20[dC]
X_90_Width	= 9.11[us]
X_Acq_Time	= 1.06430464[s]
X_Angle	= 30[deg]
X_Atn	= 4.9[dB]
X_Pulse	= 3.03666667[us]
Irr_Atn_Dec	= 22.255[dB]
Irr_Atn_Noe	= 22.255[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE

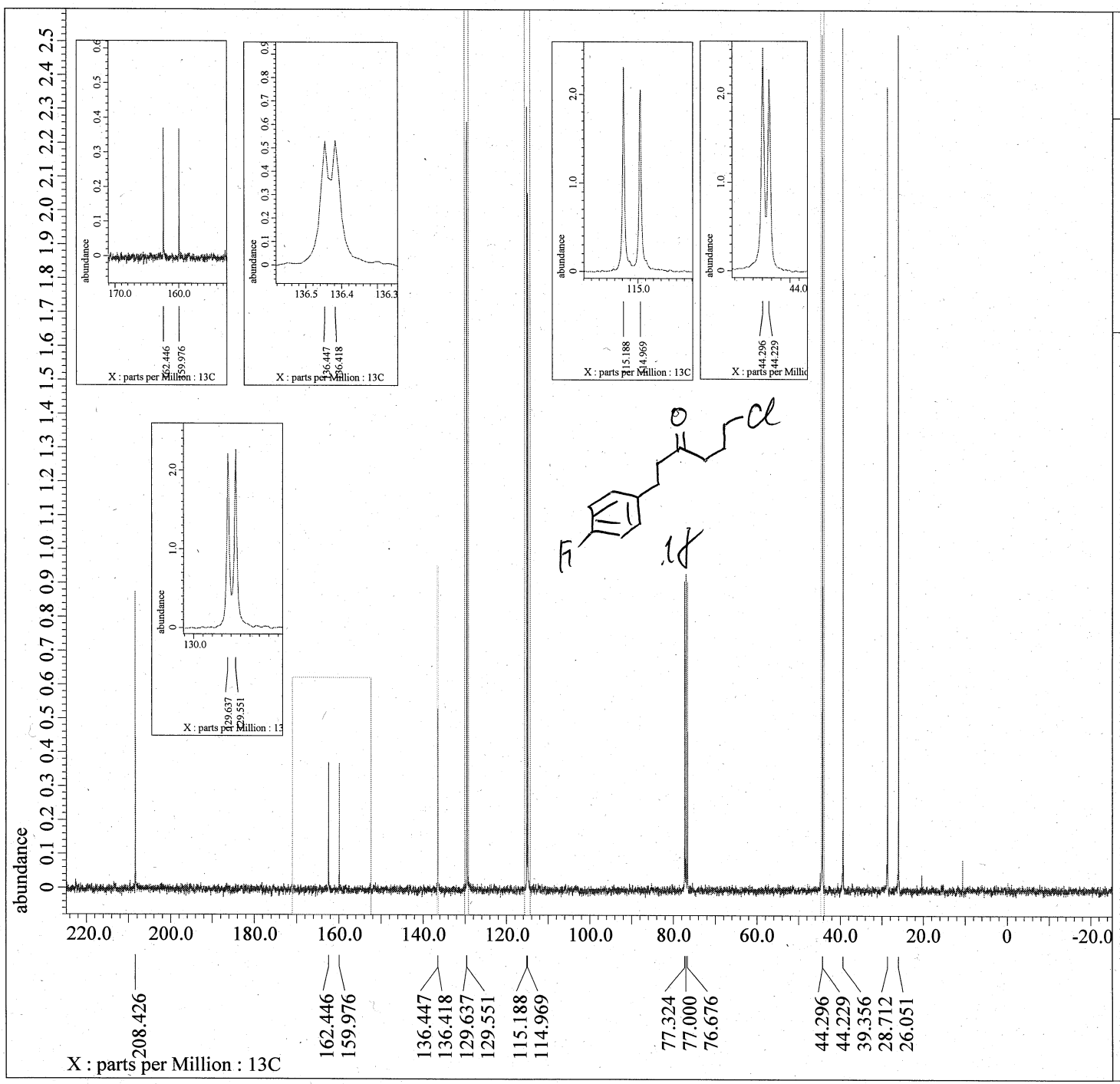


JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: UES-039-pureH-2.jdf
```

Filename	= UES-039-pureH-4.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_Id	= S#499178
Solvent	= CHLOROFORM-D
Creation_Time	= 25-JAN-2019 12:21:05
Revision_Time	= 22-APR-2019 21:17:37
Current_Time	= 22-APR-2019 21:17:52
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 2.228224[s]
X_Domain	= 1H
X_Freq	= 391.78655441[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.44878791[Hz]
X_Sweep	= 7.35294118[kHz]
Irr_Domain	= 1H
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 391.78655441[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 46
Temp_Get	= 18.4[dC]
X_90_Width	= 11.04[us]
X_Acq_Time	= 2.228224[s]
X_Angle	= 45[deg]
X_Atn	= 1.9[dB]
X_Pulse	= 5.52[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE

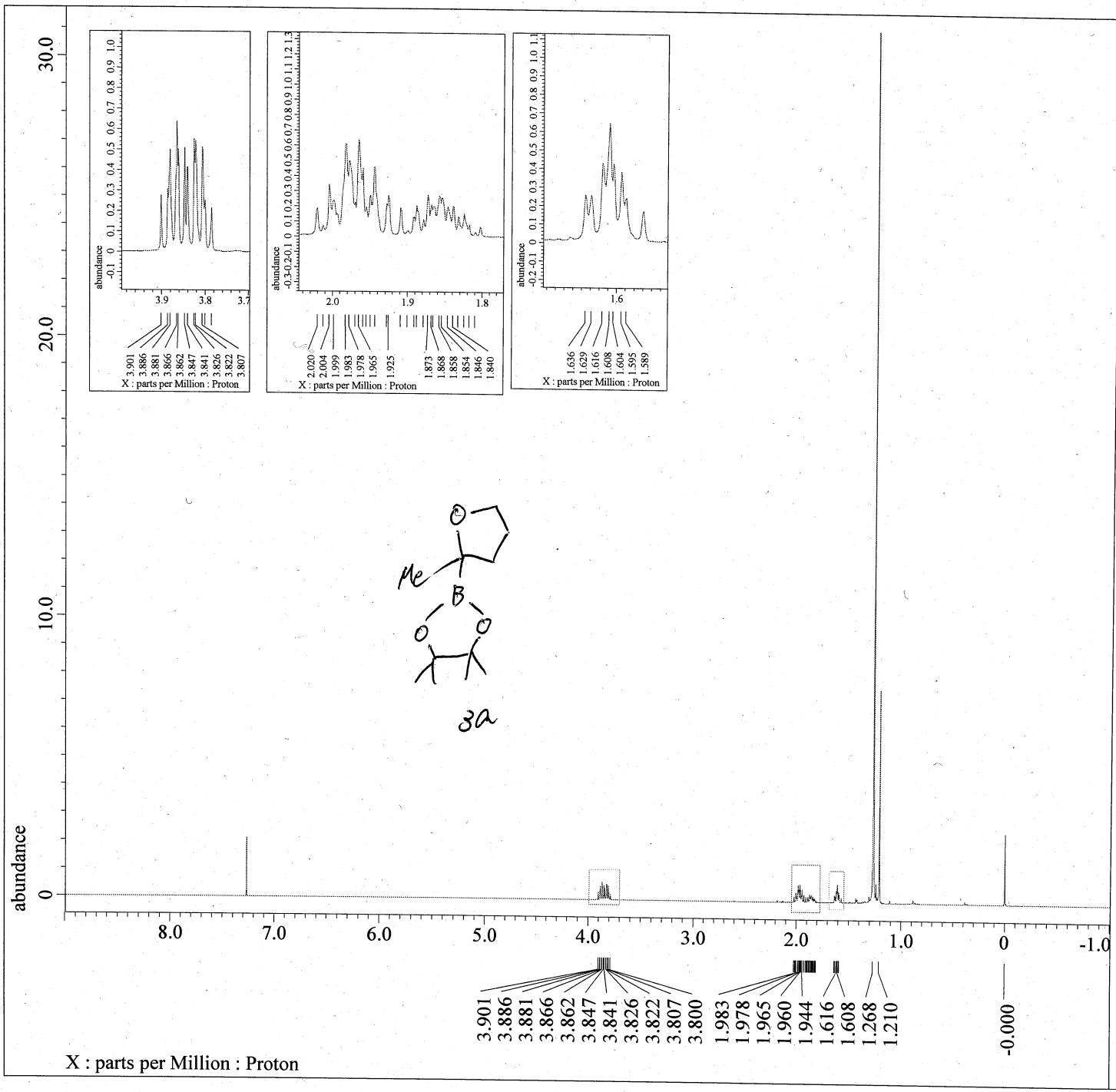


JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: UES-039-pureC-2.jdf
```

Filename	= UES-039-pureC-4.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_Id	= S#525013
Solvent	= CHLOROFORM-D
Creation_Time	= 25-JAN-2019 13:07:15
Revision_Time	= 22-APR-2019 21:18:47
Current_Time	= 22-APR-2019 21:20:07
Comment	= single pulse decoupled g
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 1.06430464[s]
X_Domain	= 13C
X_Freq	= 98.51479726[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.93958061[Hz]
X_Sweep	= 30.78817734[kHz]
Irr_Domain	= 1H
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 100
Total_Scans	= 100
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 20[dC]
X_90_Width	= 9.11[us]
X_Acq_Time	= 1.06430464[s]
X_Angle	= 30[deg]
X_Atn	= 4.9[dB]
X_Pulse	= 3.03666667[us]
Irr_Atn_Dec	= 22.255[dB]
Irr_Atn_Noe	= 22.255[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE

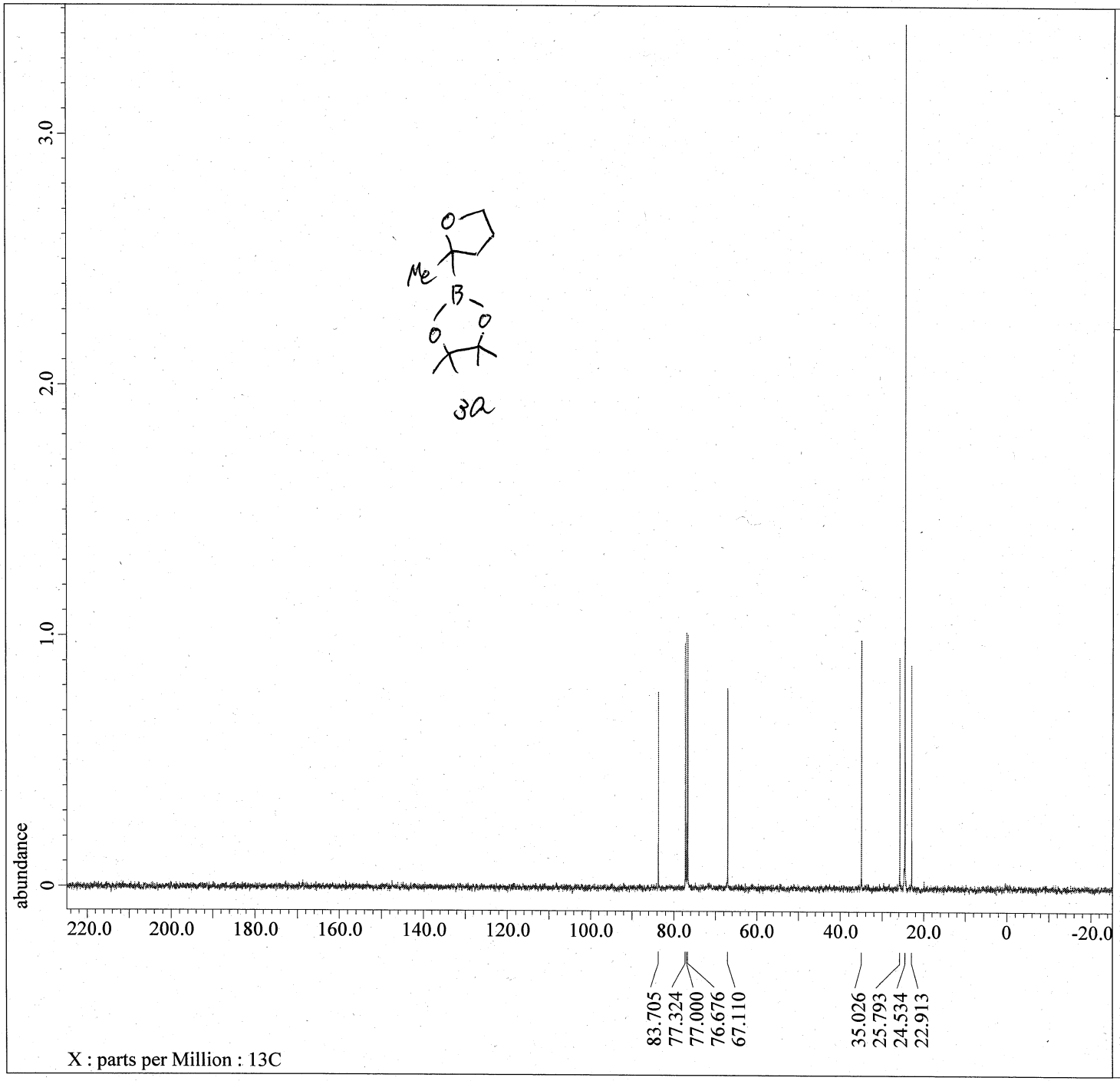


JEOL RESONANCE

```
---- 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

以下に由来:: UES-104-pureH_Proton-1-1.jdf
```

Filename	= UES-104-pureH_Proton-1-4
Author	= element
Experiment	= proton.jxp
Sample_Id	= UES-104-pureH
Solvent	= CHLOROFORM-D
Creation_Time	= 12-OCT-2018 14:29:30
Revision_Time	= 23-APR-2019 12:05:59
Current_Time	= 23-APR-2019 12:07:41
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.4073814[T] (400[MHz])
X_Acq_Duration	= 2.18103808[s]
X_Domain	= 1H
X_Freq	= 400.53219825[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45849727[Hz]
X_Sweep	= 7.51201923[kHz]
X_Sweep_Clipped	= 6.00961538[kHz]
Irr_Domain	= Proton
Irr_Freq	= 400.53219825[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 400.53219825[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 38
Temp_Get	= 19.3[dC]
X_90_Width	= 6.22[us]
X_Acq_Time	= 2.18103808[s]
X_Angle	= 45[deg]
X_Atn	= 0.8[dB]
X_Pulse	= 3.11[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE



JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

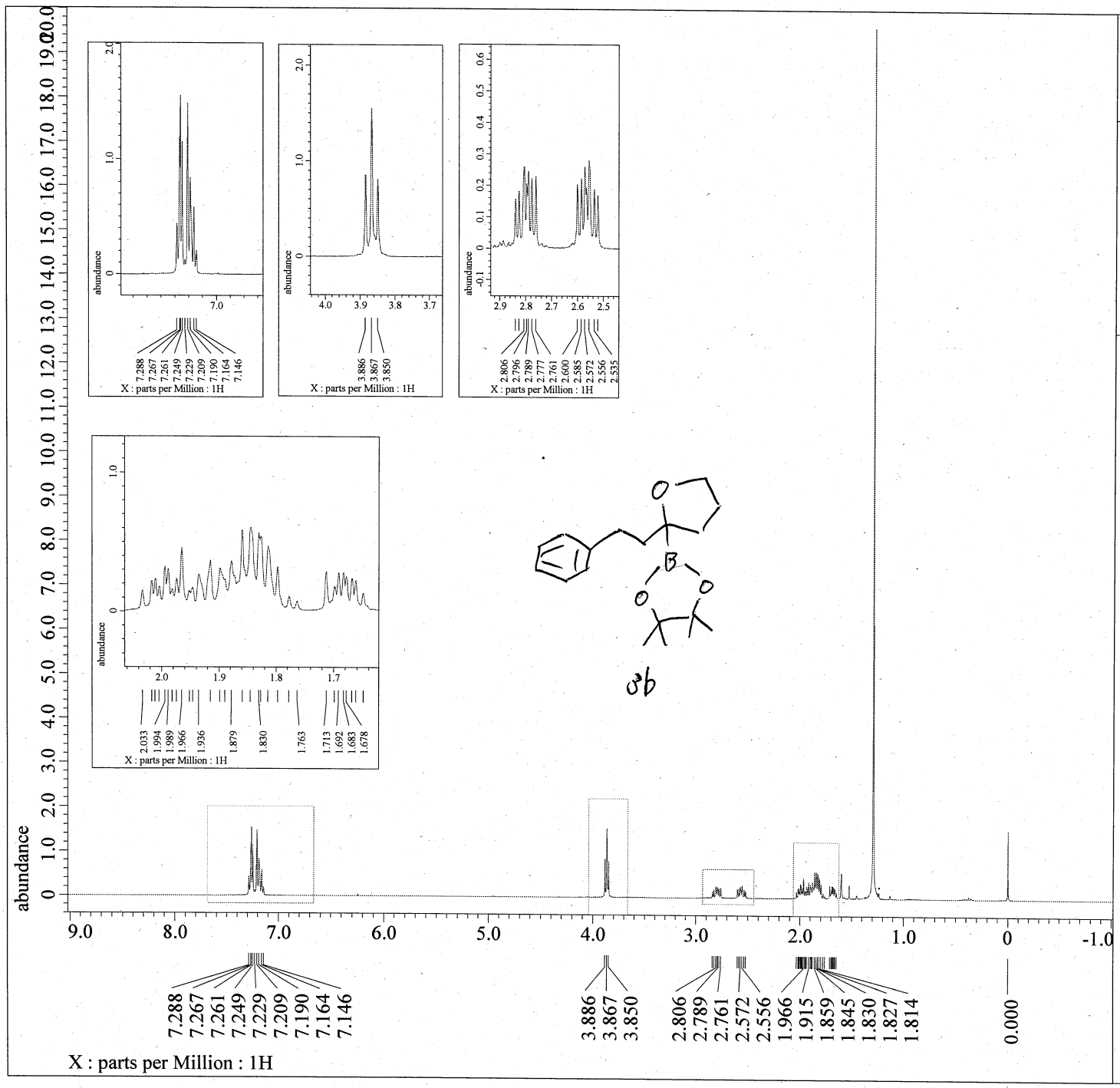
以下に由来:: UES-104-pureC-2.jdf
```

```
Filename          = UES-104-pureC-4.jdf
Author            = element
Experiment        = single_pulse_dec
Sample_Id         = S#548840
Solvent           = CHLOROFORM-D
Creation_Time     = 12-OCT-2018 13:50:24
Revision_Time     = 23-APR-2019 12:08:41
Current_Time      = 23-APR-2019 12:08:53

Comment           = single pulse decoupled g
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = 13C
Dim_Units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_Strength    = 9.20197068[T] (390[MHz])
X_Acq_Duration    = 1.06430464[s]
X_Domain          = 13C
X_Freq            = 98.51479726[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 0.93958061[Hz]
X_Sweep           = 30.78817734[kHz]
Irr_Domain        = 1H
Irr_Freq          = 391.78655441[MHz]
Irr_Offset        = 5[ppm]
Clipped           = FALSE
Scans             = 100
Total_Scans       = 100

Relaxation_Delay  = 2[s]
Recvr_Gain        = 60
Temp_Get          = 21.3[dC]
X_90_Width        = 9.11[us]
X_Acq_Time        = 1.06430464[s]
X_Angle           = 30[deg]
X_Atn             = 4.9[dB]
X_Pulse           = 3.03666667[us]
Irr_Atn_Dec       = 22.255[dB]
Irr_Atn_Noe       = 22.255[dB]
Irr_Noise         = WALTZ
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
```

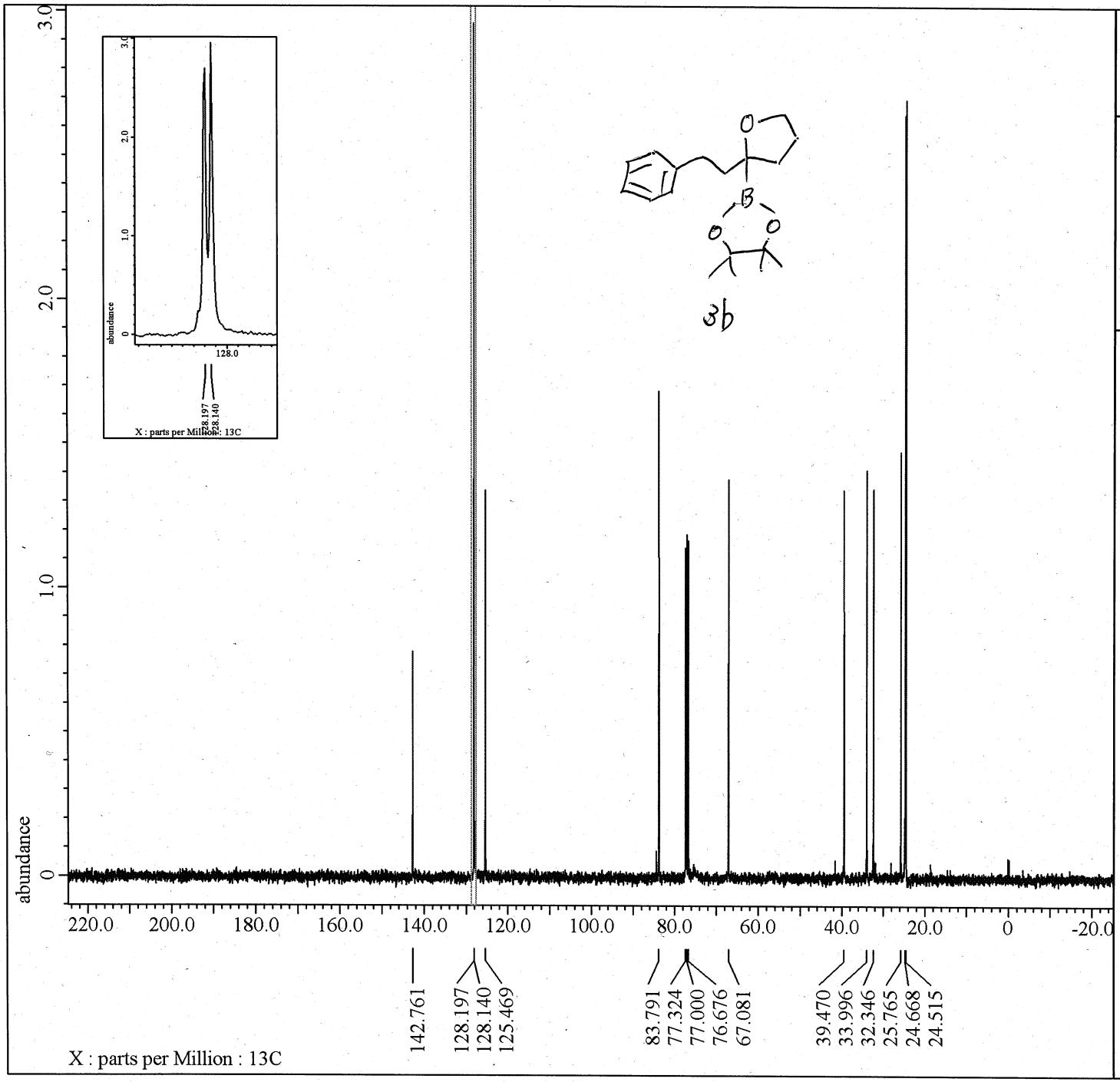


JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: UES-036-pureH-2.jdf
```

Filename	= UES-036-pureH-4.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_Id	= S#413618
Solvent	= CHLOROFORM-D
Creation_Time	= 11-JUL-2018 09:47:41
Revision_Time	= 23-APR-2019 12:19:17
Current_Time	= 23-APR-2019 12:21:51
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 2.228224[s]
X_Domain	= 1H
X_Freq	= 391.78655441[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.44878791[Hz]
X_Sweep	= 7.35294118[kHz]
Irr_Domain	= 1H
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 391.78655441[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 40
Temp_Get	= 19.4[dC]
X_90_Width	= 11.04[us]
X_Acq_Time	= 2.228224[s]
X_Angle	= 45[deg]
X_Atn	= 1.9[dB]
X_Pulse	= 5.52[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE

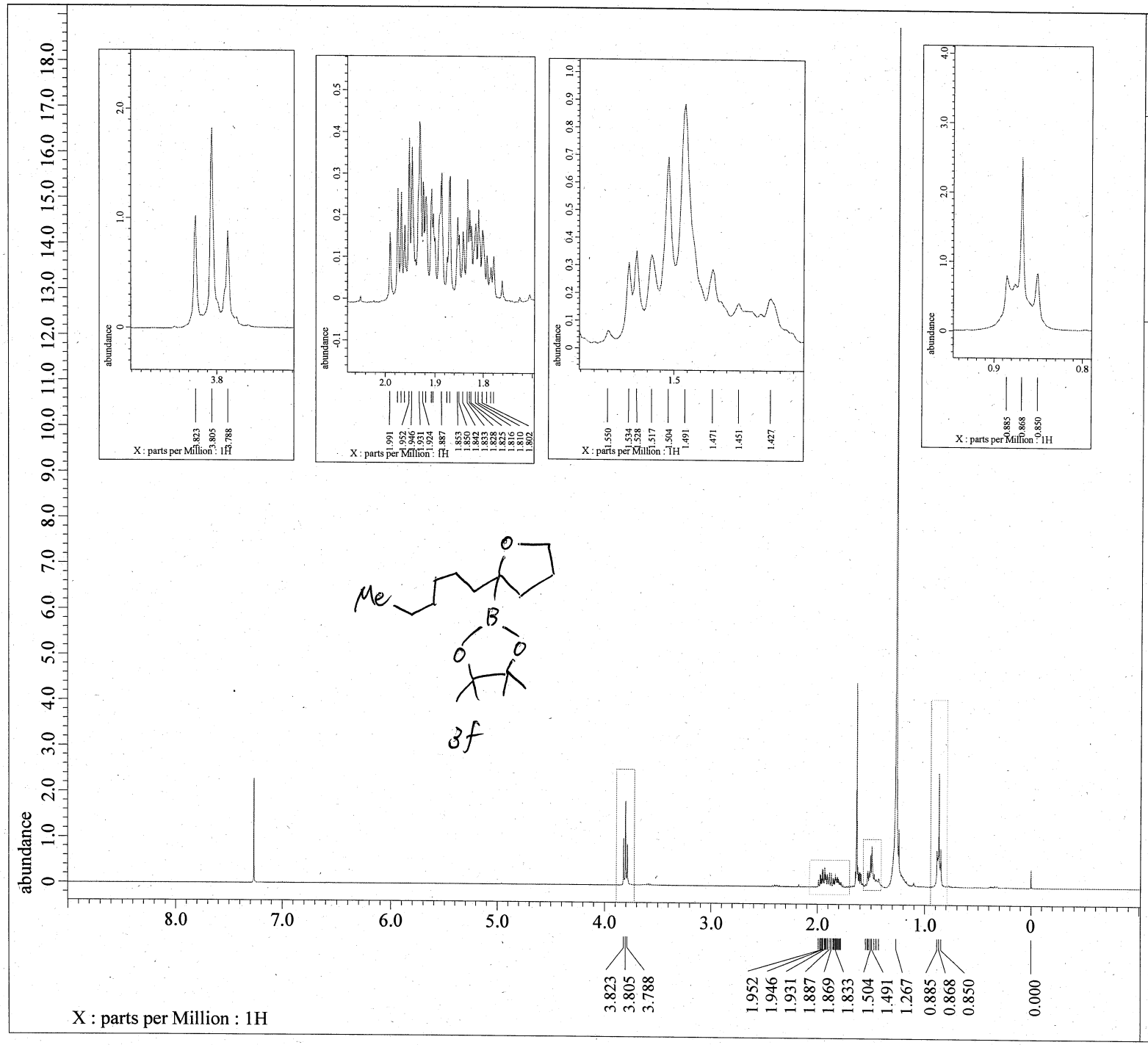


JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: UES-036-pureC-2.jdf
```

Filename	= UES-036-pureC-4.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_Id	= S#425248
Solvent	= CHLOROFORM-D
Creation_Time	= 11-JUL-2018 10:07:04
Revision_Time	= 23-APR-2019 12:23:51
Current_Time	= 23-APR-2019 12:24:29
Comment	= single pulse decoupled g
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 1.06430464[s]
X_Domain	= 13C
X_Freq	= 98.51479726[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.93958061[Hz]
X_Sweep	= 30.78817734[kHz]
Irr_Domain	= 1H
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 50
Total_Scans	= 50
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 19.6[dC]
X_90_Width	= 9.11[us]
X_Acq_Time	= 1.06430464[s]
X_Angle	= 30[deg]
X_Atn	= 4.9[dB]
X_Pulse	= 3.03666667[us]
Irr_Atn_Dec	= 22.255[dB]
Irr_Atn_Noe	= 22.255[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE

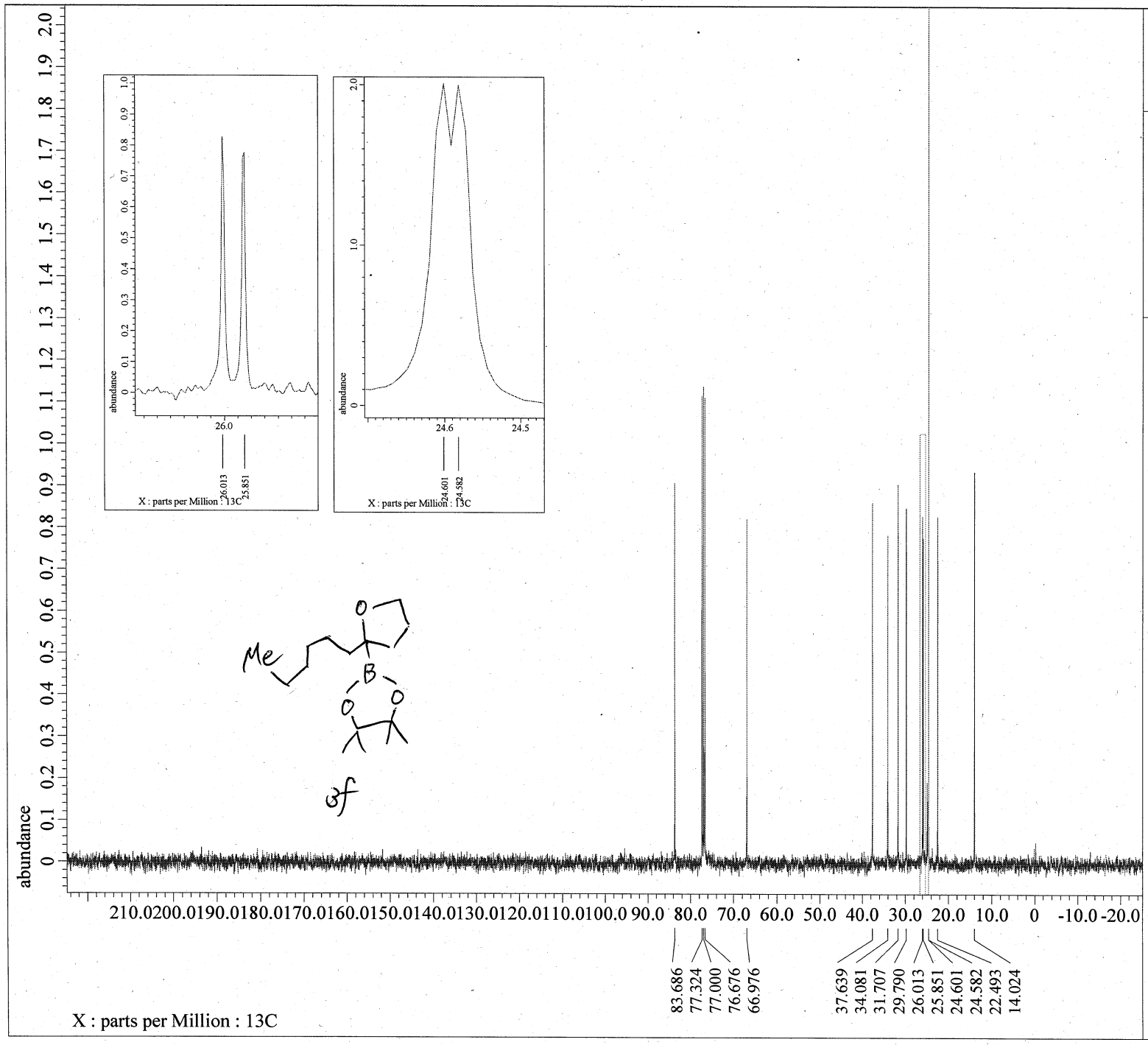


JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: UES-052-pureH-1.jdf
```

Filename	= UES-052-pureH-5.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_Id	= S#347797
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 11-JUL-2018 16:56:36
Revision_Time	= 23-APR-2019 12:35:47
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= 1H
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 2.228224[s]
X_Domain	= 1H
X_Freq	= 391.78655441[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.44878791[Hz]
X_Sweep	= 7.35294118[kHz]
Irr_Domain	= 1H
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 391.78655441[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 40
Temp_Get	= 19.3[dC]
X_90_Width	= 11.04[us]
X_Acq_Time	= 2.228224[s]
X_Angle	= 45[deg]
X_Atn	= 1.9[dB]
X_Pulse	= 5.52[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE

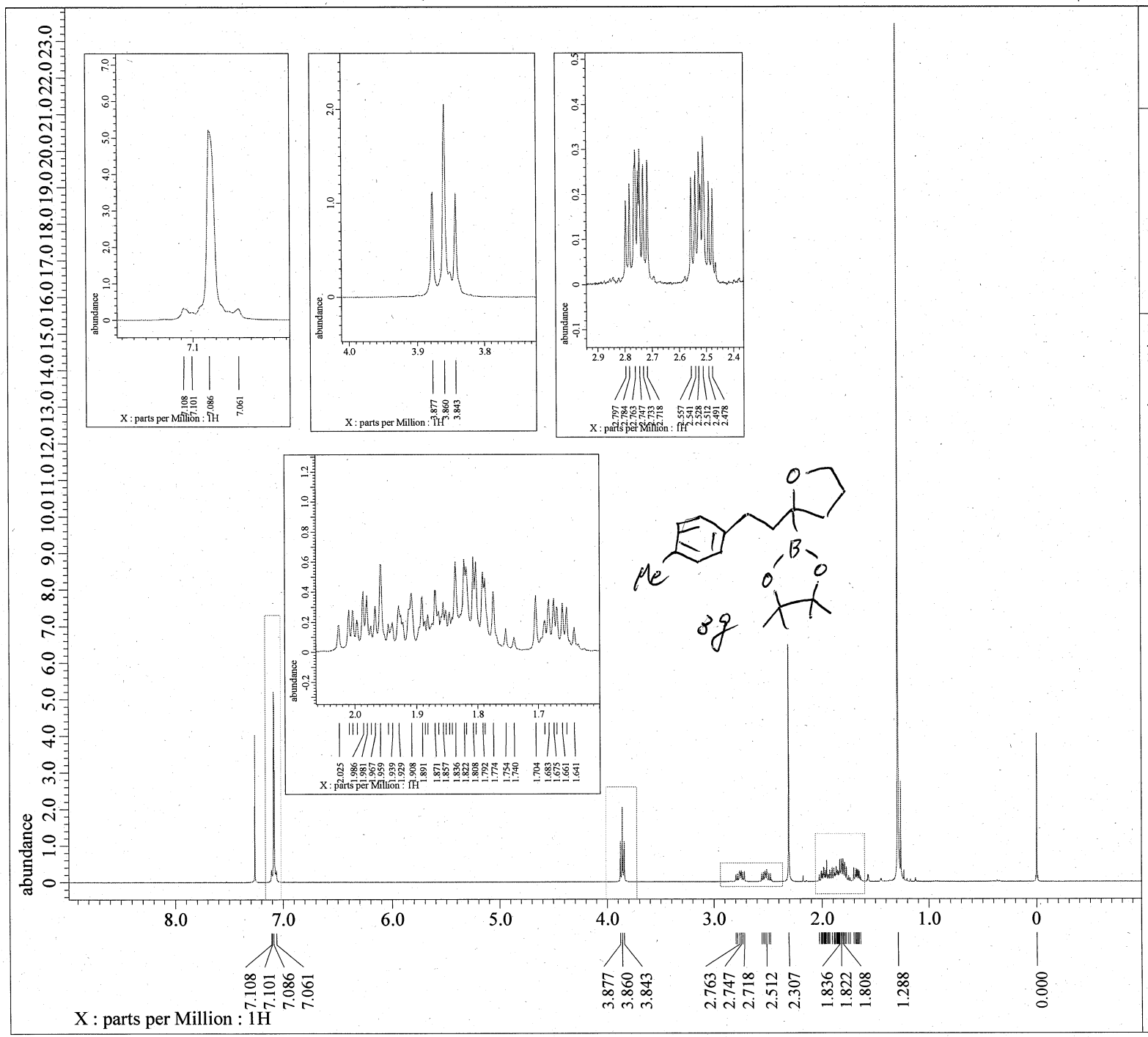


JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: UES-052-pureC-1.jdf
```

Filename	= UES-052-pureC-2.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_Id	= S#361150
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 11-JUL-2018 17:17:29
Revision_Time	= 23-APR-2019 12:38:19
Comment	= single pulse decoupled ga
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= 13C
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 1.06430464[s]
X_Domain	= 13C
X_Freq	= 98.51479726[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.93958061[Hz]
X_Sweep	= 30.78817734[kHz]
Irr_Domain	= 1H
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 50
Total_Scans	= 50
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 19.6[dC]
X_90_Width	= 9.11[us]
X_Acq_Time	= 1.06430464[s]
X_Angle	= 30[deg]
X_Atn	= 4.9[dB]
X_Pulse	= 3.03666667[us]
Irr_Atn_Dec	= 22.255[dB]
Irr_Atn_Noe	= 22.255[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE

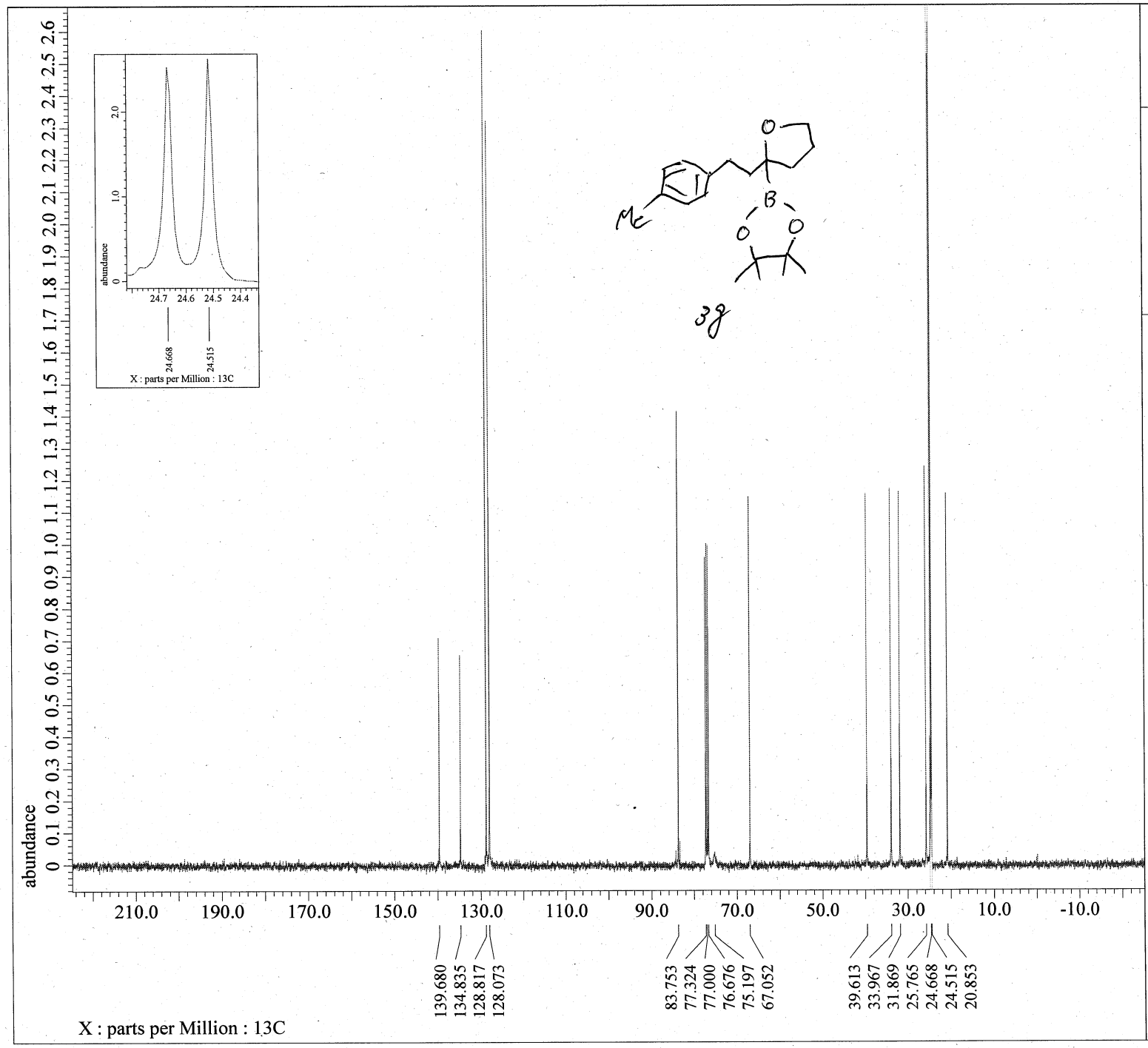


JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: UES-174-pureH-1.jdf
```

Filename	= UES-174-pureH-2.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_Id	= S#375925
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 19-JAN-2019 17:55:34
Revision_Time	= 23-APR-2019 12:44:41
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= 1H
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 2.228224[s]
X_Domain	= 1H
X_Freq	= 391.78655441[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.44878791[Hz]
X_Sweep	= 7.35294118[kHz]
Irr_Domain	= 1H
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 391.78655441[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 46
Temp_Get	= 19.4[dC]
X_90_Width	= 11.04[us]
X_Acq_Time	= 2.228224[s]
X_Angle	= 45[deg]
X_Atn	= 1.9[dB]
X_Pulse	= 5.52[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE

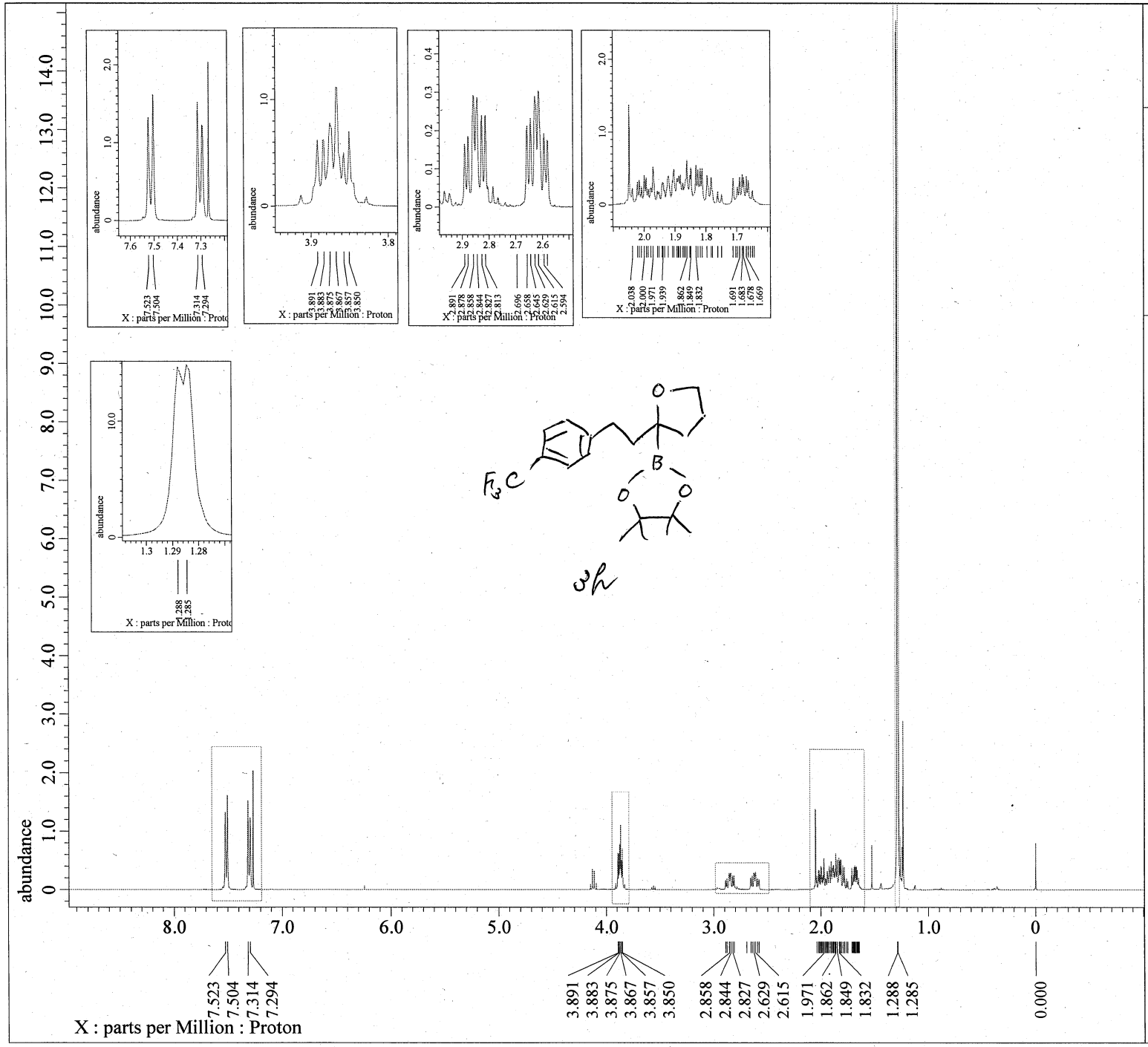


JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: UES-174-pureC-1.jdf
```

Filename	= UES-174-pureC-2.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_Id	= S#382393
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 19-JAN-2019 18:05:05
Revision_Time	= 23-APR-2019 16:14:25
Comment	= single pulse decoupled ga
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= 13C
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 1.06430464[s]
X_Domain	= 13C
X_Freq	= 98.51479726[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.93958061[Hz]
X_Sweep	= 30.78817734[kHz]
Irr_Domain	= 1H
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Clipped	= TRUE
Scans	= 100
Total_Scans	= 100
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 19.9[dC]
X_90_Width	= 9.11[us]
X_Acq_Time	= 1.06430464[s]
X_Angle	= 30[deg]
X_Atn	= 4.9[dB]
X_Pulse	= 3.03666667[us]
Irr_Atn_Dec	= 22.255[dB]
Irr_Atn_Noe	= 22.255[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE

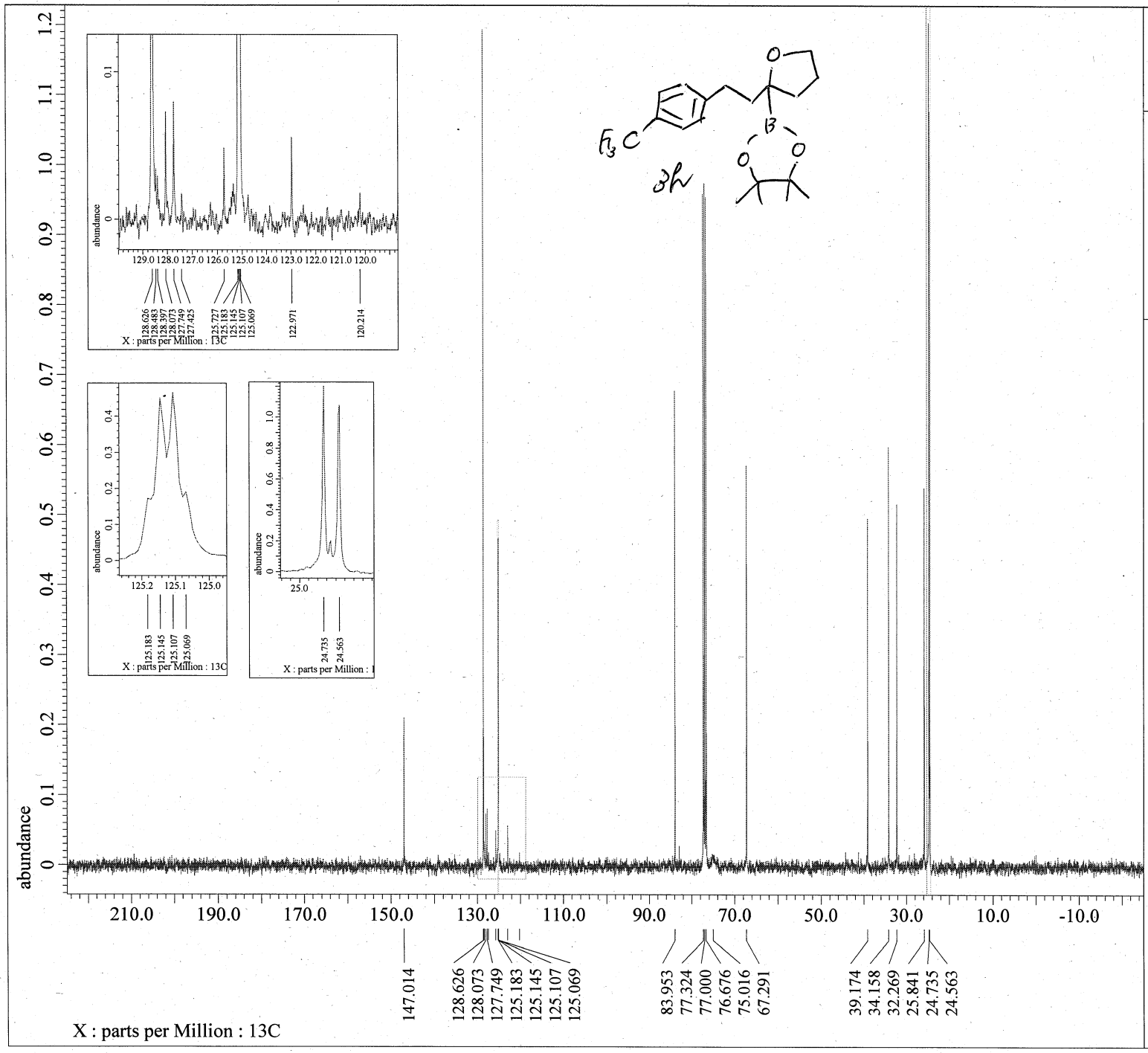


JEOL

```
---- 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

以下に由来:: UES-093-pureH_Proton-1-1.jdf
```

Filename	= UES-093-pureH_Proton-1-2.
Author	= element
Experiment	= proton.jxp
Sample_Id	= UES-093-pureH
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 28-SEP-2018 09:52:07
Revision_Time	= 23-APR-2019 16:18:35
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Site	= JNM-ECS400
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.37221[T] (400[MHz])
X_Acq_Duration	= 2.1889024[s]
X_Domain	= 1H
X_Freq	= 399.03472754[MHz]
X_Offset	= 5.0[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45684997[Hz]
X_Sweep	= 7.48502994[kHz]
X_Sweep_Clipped	= 5.98802395[kHz]
Irr_Domain	= Proton
Irr_Freq	= 399.03472754[MHz]
Irr_Offset	= 5.0[ppm]
Tri_Domain	= Proton
Tri_Freq	= 399.03472754[MHz]
Tri_Offset	= 5.0[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 30
Temp_Get	= 18.1[dC]
X_90_Width	= 6.6[us]
X_Acq_Time	= 2.1889024[s]
X_Angle	= 45[deg]
X_Atn	= 1[dB]
X_Pulse	= 3.3[us]
Irr_Mode	= Off
Tri_Mode	= Off

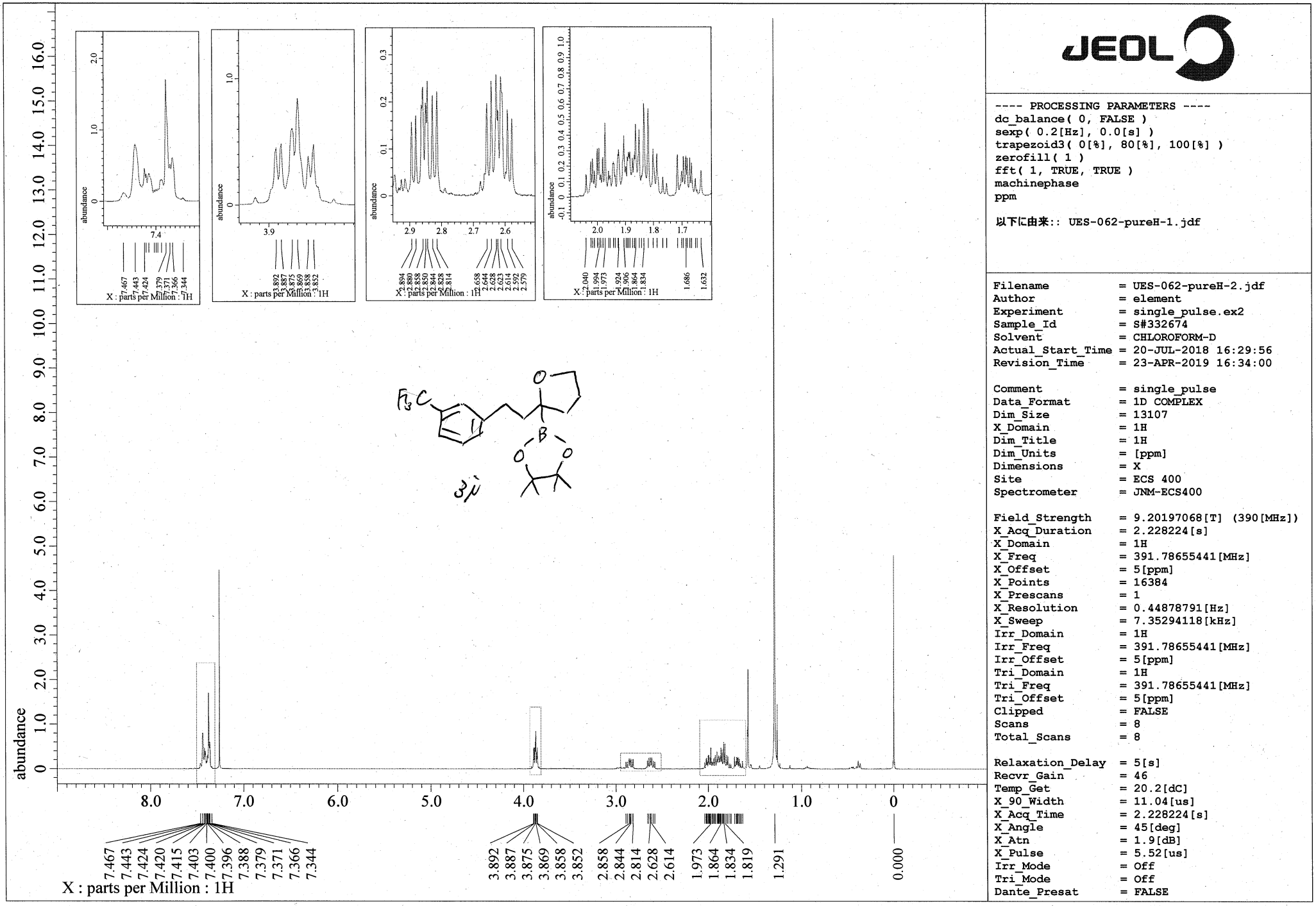


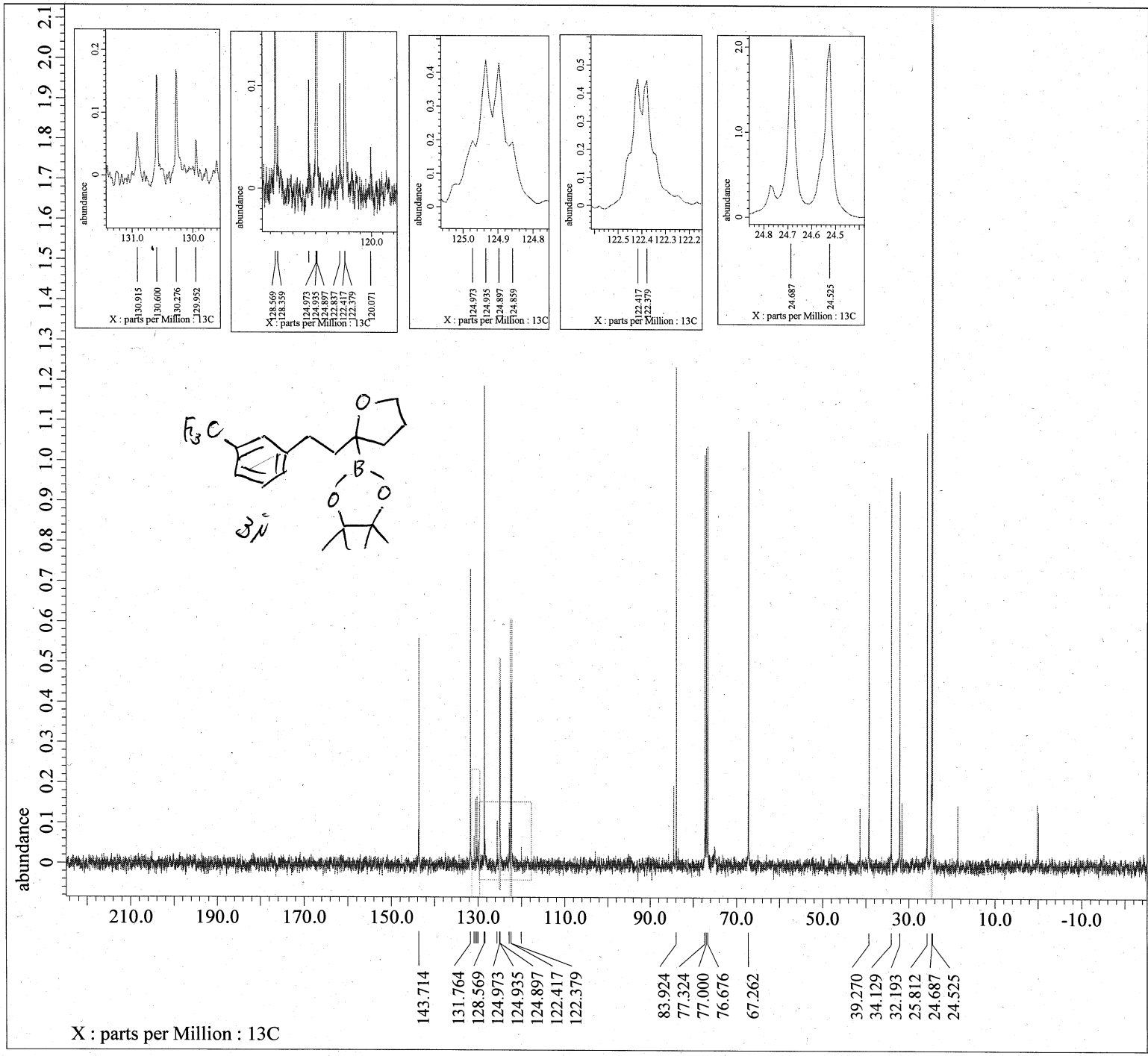
JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: UES-093-pureC-2.jdf
```

Filename	= UES-093-pureC-3.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_Id	= S#727530
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 18-JAN-2019 03:40:33
Revision_Time	= 23-APR-2019 16:21:12
Comment	= single pulse decoupled ga
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= 13C
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 1.06430464[s]
X_Domain	= 13C
X_Freq	= 98.51479726[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.93958061[Hz]
X_Sweep	= 30.78817734[kHz]
Irr_Domain	= 1H
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 200
Total_Scans	= 200
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 20.2[dC]
X_90_Width	= 9.11[us]
X_Acq_Time	= 1.06430464[s]
X_Angle	= 30[deg]
X_Atn	= 4.9[dB]
X_Pulse	= 3.03666667[us]
Irr_Atn_Dec	= 22.255[dB]
Irr_Atn_Noe	= 22.255[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE


JEOL
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
以下に由来:: UES-062-pureH-1.jdf
Filename = UES-062-pureH-2.jdf
Author = element
Experiment = single_pulse.ex2
Sample_Id = S#332674
Solvent = CHLOROFORM-D
Actual_Start_Time = 20-JUL-2018 16:29:56
Revision_Time = 23-APR-2019 16:34:00
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 13107
X_Domain = 1H
Dim_Title = 1H
Dim_Units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = JNM-ECS400
Field_Strength = 9.20197068[T] (390[MHz])
X_Acq_Duration = 2.228224[s]
X_Domain = 1H
X_Freq = 391.78655441[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Prescans = 1
X_Resolution = 0.44878791[Hz]
X_Sweep = 7.35294118[kHz]
Irr_Domain = 1H
Irr_Freq = 391.78655441[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = 1H
Tri_Freq = 391.78655441[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recvr_Gain = 46
Temp_Get = 20.2[dC]
X_90_Width = 11.04[us]
X_Acq_Time = 2.228224[s]
X_Angle = 45[deg]
X_Atn = 1.9[dB]
X_Pulse = 5.52[us]
Irr_Mode = Off
Tri_Mode = Off
Dante_Presat = FALSE
abundance
X : parts per Million : 1H
7.467 7.443 7.424 7.420 7.415 7.403 7.400 7.396 7.388 7.379 7.371 7.366 7.344
3.892 3.887 3.875 3.869 3.858 3.852
2.858 2.844 2.814 2.628 2.614
1.973 1.864 1.834 1.819
1.291
0.000

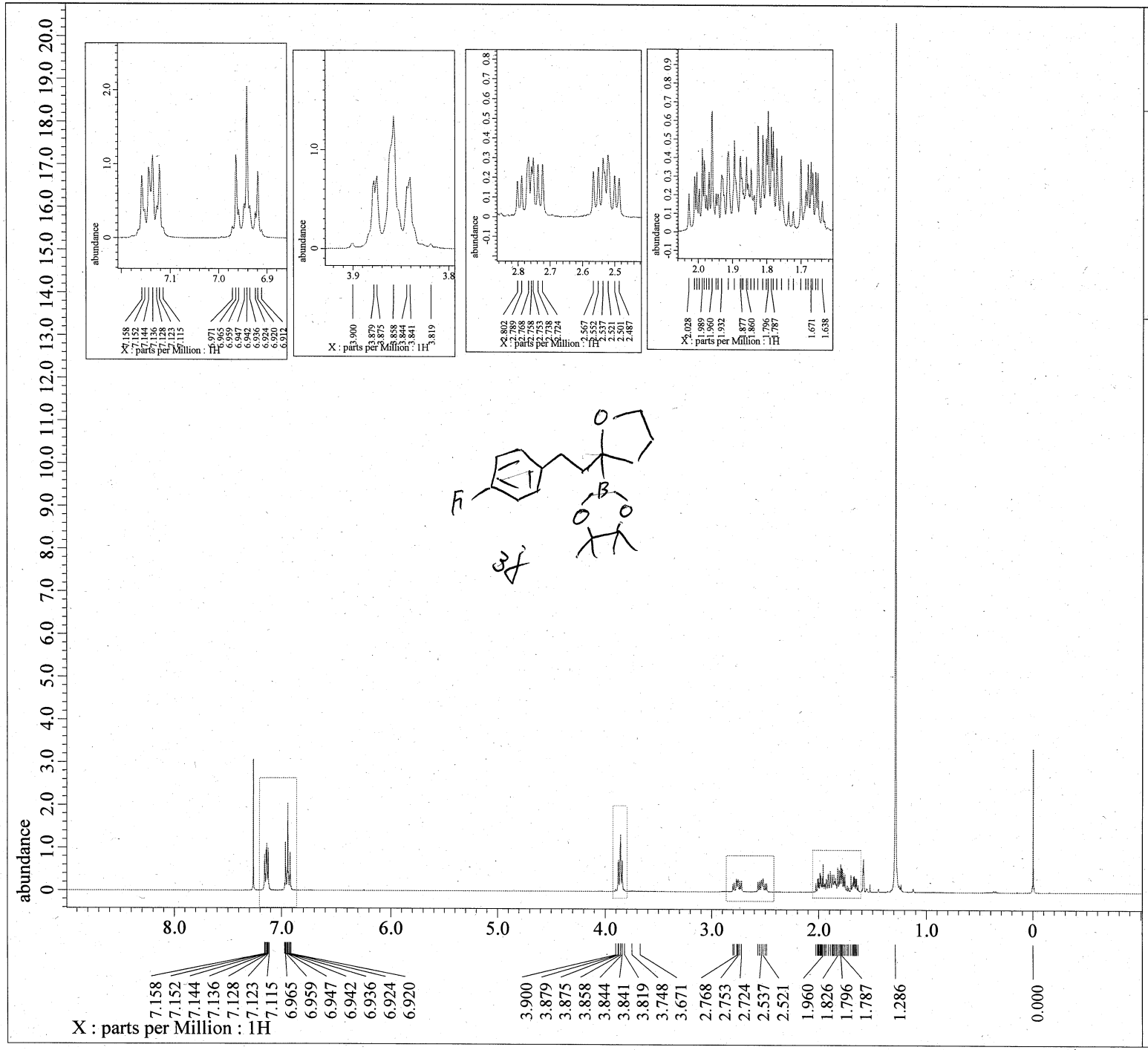


JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: UES-062-pureC-1.jdf
```

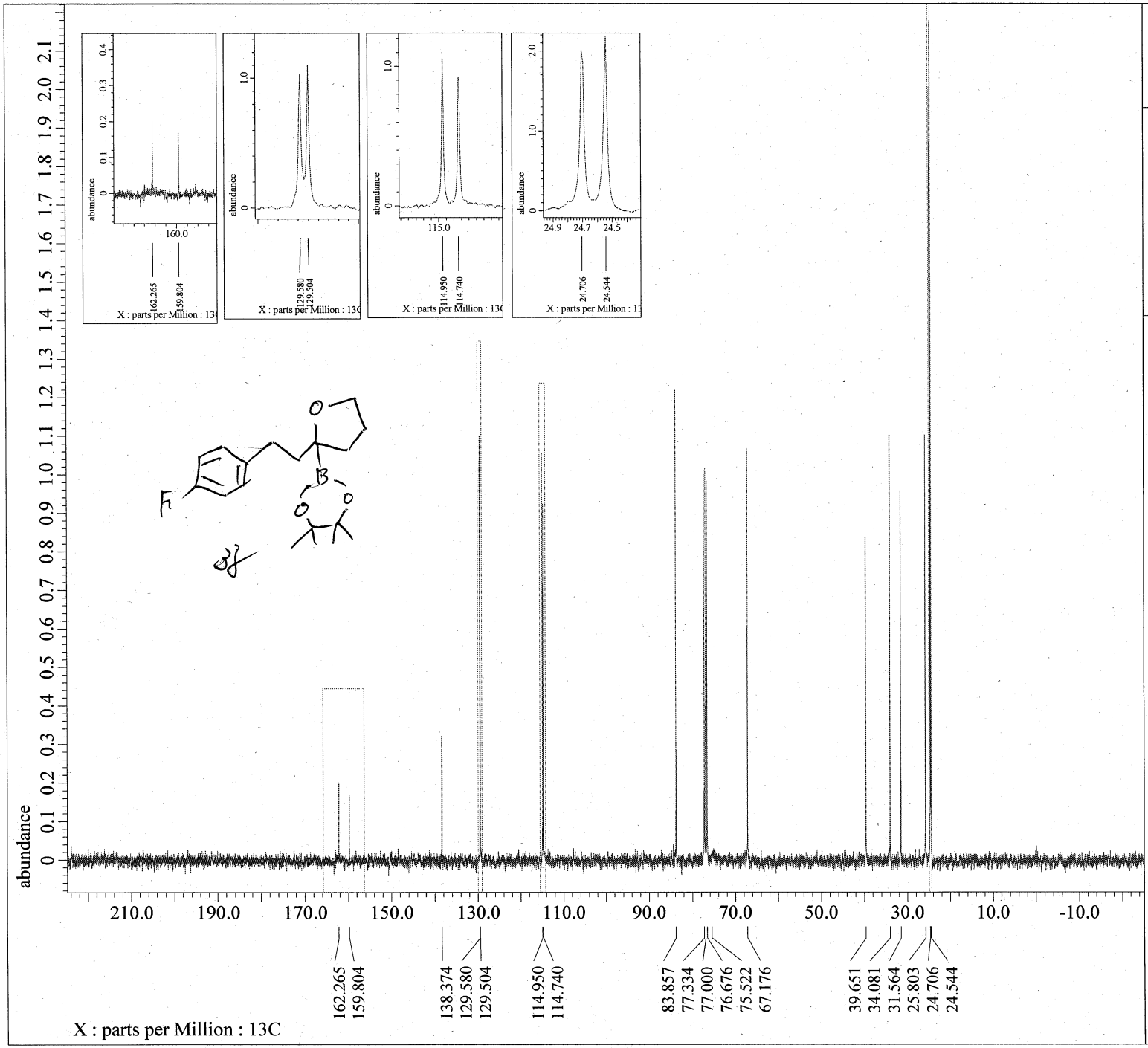
Filename	= UES-062-pureC-2.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_Id	= S#343907
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 20-JUL-2018 16:47:28
Revision_Time	= 23-APR-2019 16:38:28
Comment	= single pulse decoupled ga
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= 13C
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 1.06430464[s]
X_Domain	= 13C
X_Freq	= 98.51479726[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.93958061[Hz]
X_Sweep	= 30.78817734[kHz]
Irr_Domain	= 1H
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 50
Total_Scans	= 50
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 20.4[dC]
X_90_Width	= 9.11[us]
X_Acq_Time	= 1.06430464[s]
X_Angle	= 30[deg]
X_Atn	= 4.9[dB]
X_Pulse	= 3.03666667[us]
Irr_Atn_Dec	= 22.255[dB]
Irr_Atn_Noe	= 22.255[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE



---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm

以下に由来:: UES-041-pureH-1.jdf

Filename	= UES-041-pureH-2.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_Id	= S#488930
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 11-JUL-2018 20:51:48
Revision_Time	= 23-APR-2019 16:43:11
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= 1H
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 2.228224[s]
X_Domain	= 1H
X_Freq	= 391.78655441[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.44878791[Hz]
X_Sweep	= 7.35294118[kHz]
Irr_Domain	= 1H
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 391.78655441[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 44
Temp_Get	= 19.5[dC]
X_90_Width	= 11.04[us]
X_Acq_Time	= 2.228224[s]
X_Angle	= 45[deg]
X_Atn	= 1.9[dB]
X_Pulse	= 5.52[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE

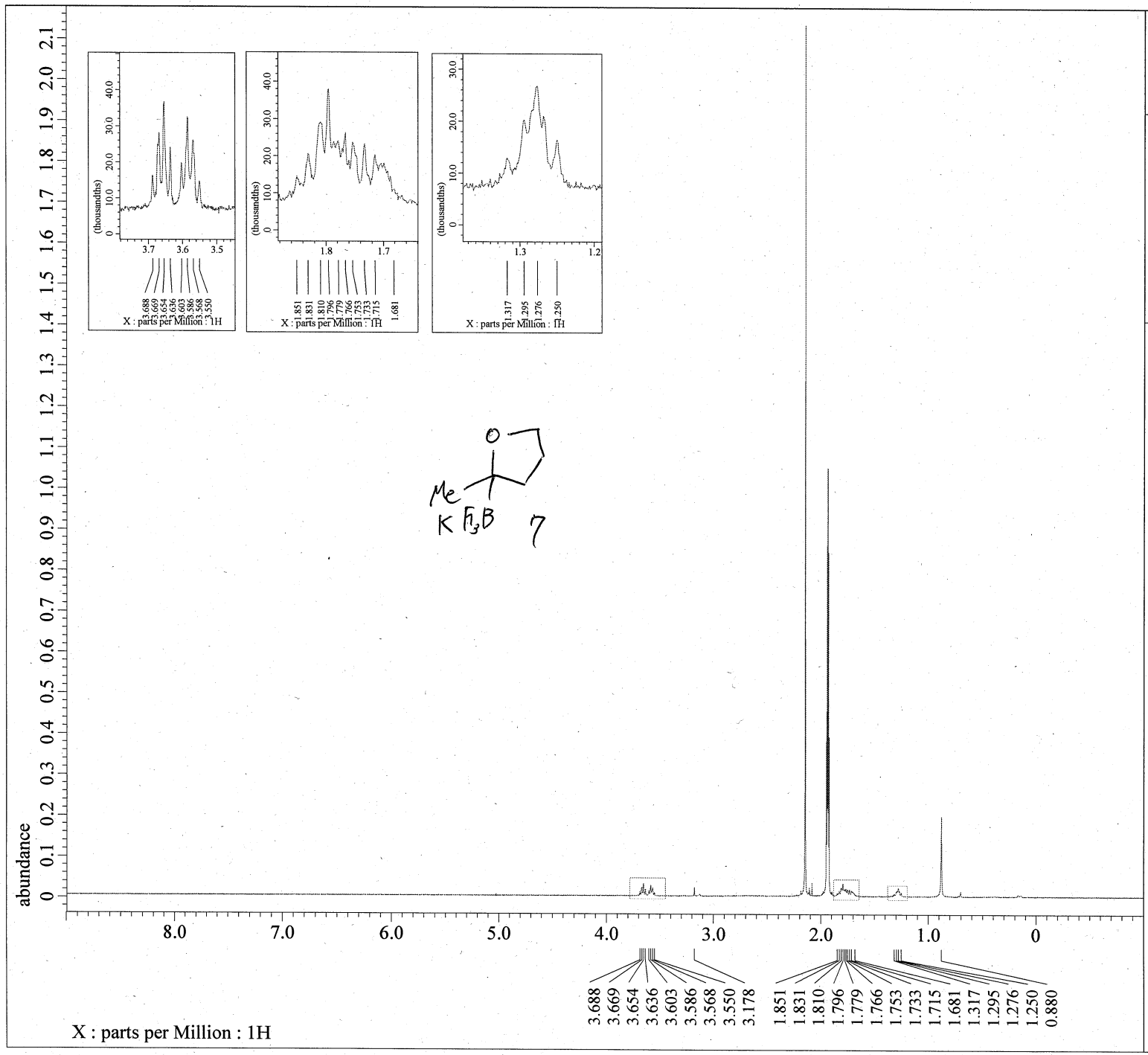


JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: UES-041-pureC-1.jdf
```

Filename	= UES-041-pureC-2.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_Id	= S#500723
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 11-JUL-2018 21:10:05
Revision_Time	= 23-APR-2019 16:45:22
Comment	= single pulse decoupled ga
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= 13C
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 1.06430464[s]
X_Domain	= 13C
X_Freq	= 98.51479726[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.93958061[Hz]
X_Sweep	= 30.78817734[kHz]
Irr_Domain	= 1H
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 50
Total_Scans	= 50
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 19.8[dC]
X_90_Width	= 9.11[us]
X_Acq_Time	= 1.06430464[s]
X_Angle	= 30[deg]
X_Atn	= 4.9[dB]
X_Pulse	= 3.03666667[us]
Irr_Atn_Dec	= 22.255[dB]
Irr_Atn_Noe	= 22.255[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE

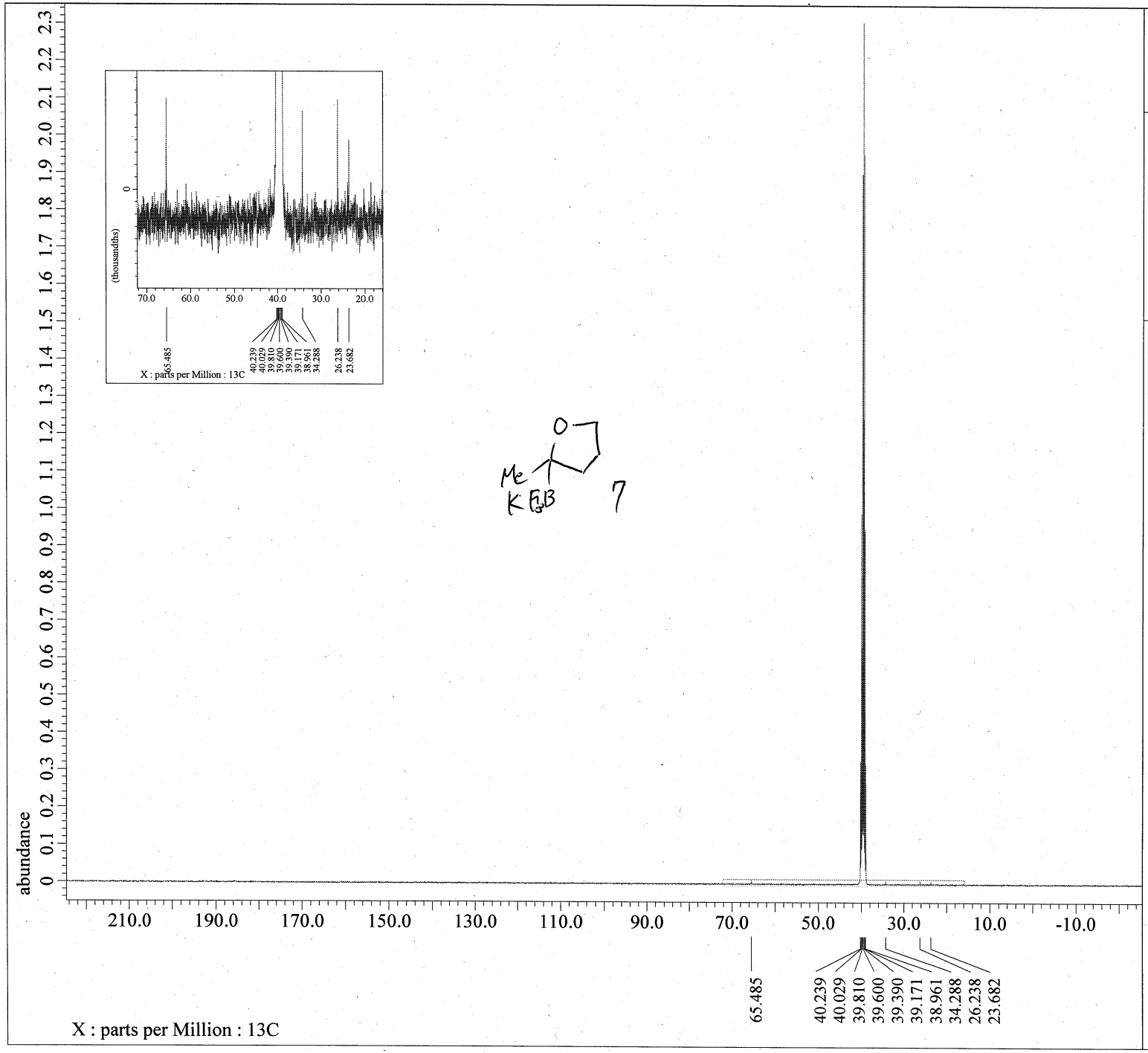


JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: UES-193-pureH-1.jdf
```

Filename	= UES-193-pureH-2.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_Id	= 1
Solvent	= ACETONITRILE-D3
Actual_Start_Time	= 11-MAR-2019 22:39:16
Revision_Time	= 23-APR-2019 16:53:13
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= 1H
Dim_Title	= 1H
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400P
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]
Irr_Domain	= 1H
Irr_Freq	= 395.88430144[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 1H
Tri_Freq	= 395.88430144[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 38
Temp_Get	= 20.3[dC]
X_90_Width	= 13.2[us]
X_Acq_Time	= 2.20725248[s]
X_Angle	= 45[deg]
X_Atn	= 3.5[dB]
X_Pulse	= 6.6[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE

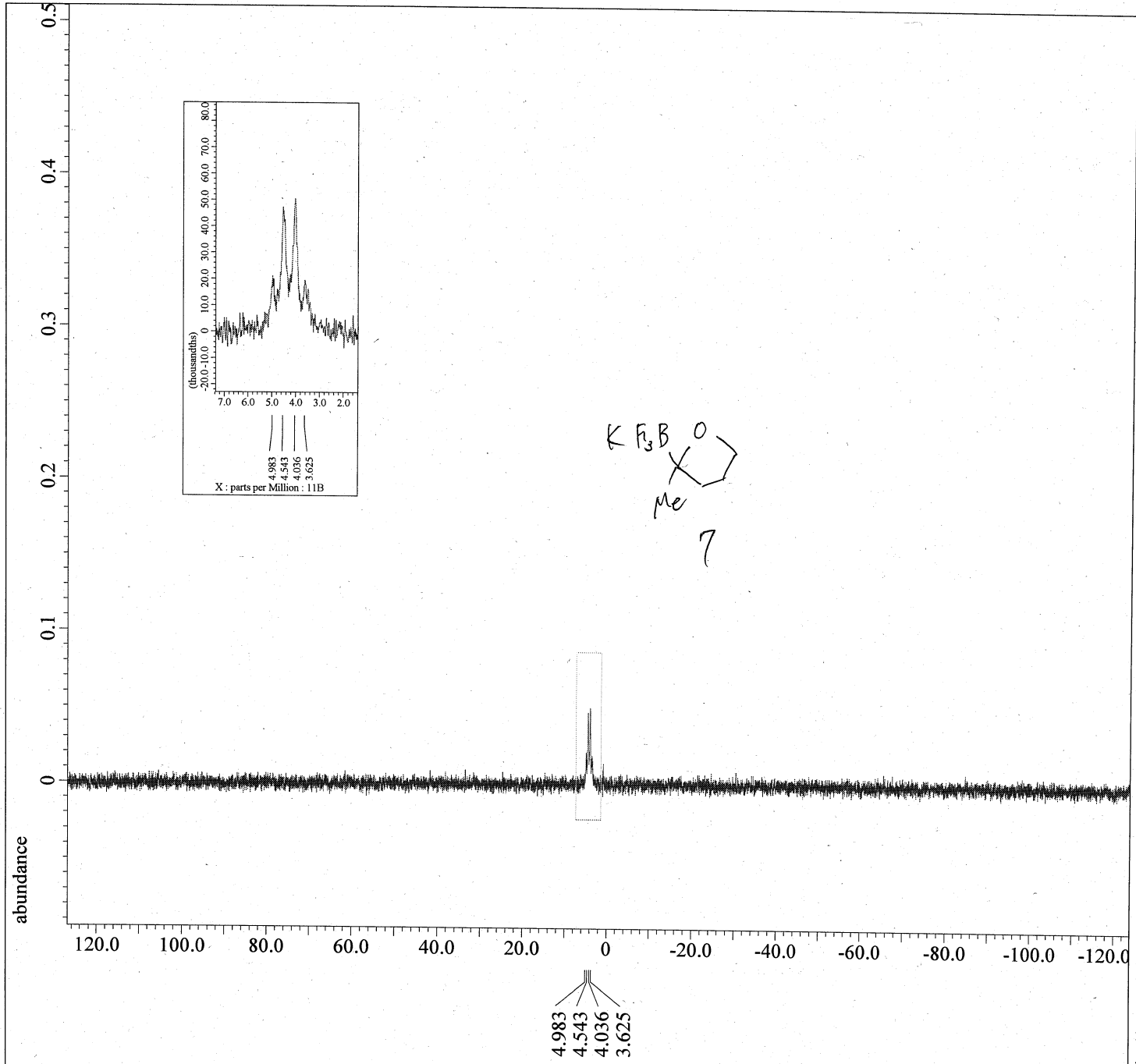


JEOL

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: UES-193-pureC-1.jdf
```

Filename	= UES-193-pureC-2.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_Id	= 1
Solvent	= DMSO-D6
Actual_Start_Time	= 12-MAR-2019 04:43:07
Revision_Time	= 23-APR-2019 16:58:31
Comment	= single pulse decoupled ga
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= 13C
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 1.06430464[s]
X_Domain	= 13C
X_Freq	= 98.51479726[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.93958061[Hz]
X_Sweep	= 30.78817734[kHz]
Irr_Domain	= 1H
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 6000
Total_Scans	= 6000
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 19.1[dC]
X_90_Width	= 9.11[us]
X_Acq_Time	= 1.06430464[s]
X_Angle	= 30[deg]
X_Atn	= 4.9[dB]
X_Pulse	= 3.03666667[us]
Irr_Atn_Dec	= 22.255[dB]
Irr_Atn_Noe	= 22.255[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE

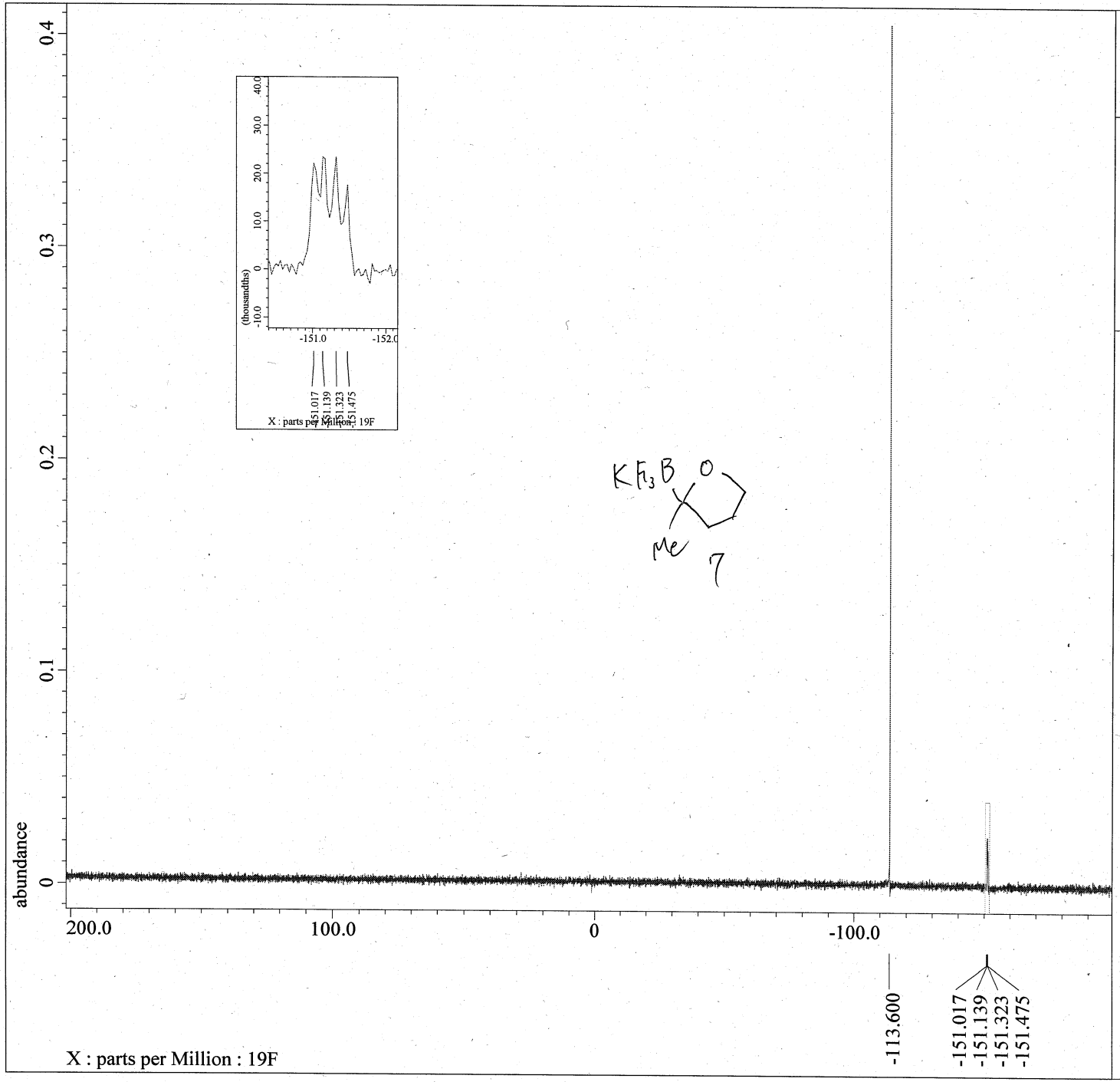


JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

以下に由来:: UES-193-pureB-2.jdf
```

Filename	= UES-193-pureB-5.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_Id	= S#520790
Solvent	= ACETONITRILE-D3
Creation_Time	= 11-MAR-2019 14:17:56
Revision_Time	= 17-APR-2019 15:14:34
Current_Time	= 17-APR-2019 15:14:56
Comment	= single pulse decoupled g
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
Dim_Title	= 11B
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400P
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.2982153[T] (400[MHz])
X_Acq_Duration	= 0.82313216[s]
X_Domain	= 11B
X_Freq	= 127.01553457[MHz]
X_Offset	= 0[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.21487174[Hz]
X_Sweep	= 39.8089172[kHz]
Irr_Domain	= 1H
Irr_Freq	= 395.88430144[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 400
Total_Scans	= 400
Relaxation_Delay	= 2[s]
Recvr_Gain	= 56
Temp_Get	= 20.9[dC]
X_90_Width	= 10[us]
X_Acq_Time	= 0.82313216[s]
X_Angle	= 30[deg]
X_Atn	= 4.8[dB]
X_Pulse	= 3.33333333[us]
Irr_Atn_Dec	= 22.3[dB]
Irr_Atn_Noe	= 22.3[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE

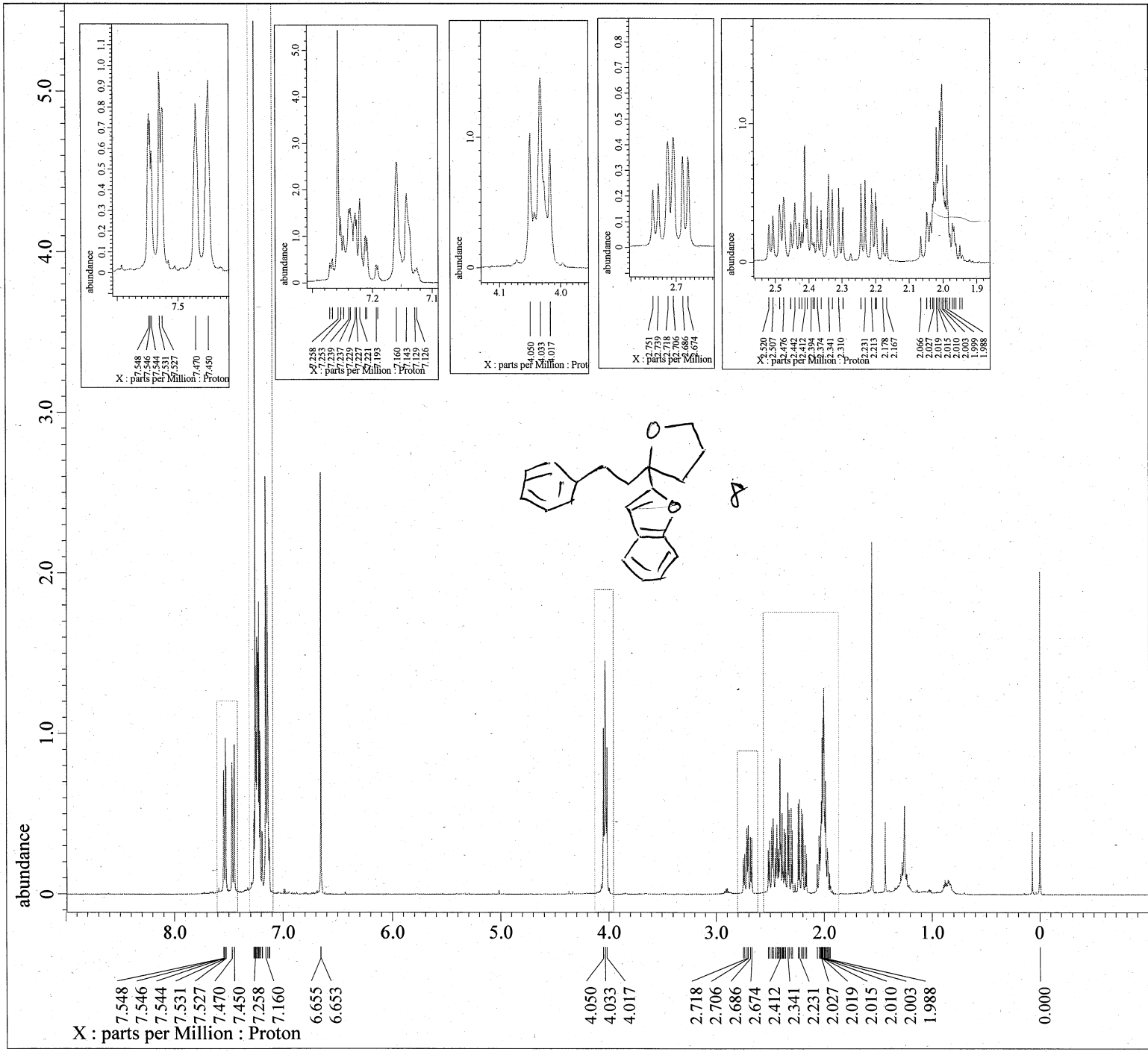


JEOL RESONANCE

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
blip( 16, 64, 30 )
fft( 1, TRUE, TRUE )
machinephase
ppm
phase( -24.77227, 0, 50[%] )

以下に由来:: UES-193-pureFIS-6.jdf
```

Filename	= UES-193-pureFIS-11.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_Id	= S#538015
Solvent	= ACETONITRILE-D3
Creation_Time	= 11-MAR-2019 14:28:08
Revision_Time	= 17-APR-2019 15:19:54
Current_Time	= 17-APR-2019 15:20:11
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
Dim_Title	= 19F
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECX 400P
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]
Irr_Domain	= 19F
Irr_Freq	= 372.50336686[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= 19F
Tri_Freq	= 372.50336686[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 24
Temp_Get	= 20.5[dC]
X_90_Width	= 13.9[us]
X_Acq_Time	= 87.81824[ms]
X_Angle	= 45[deg]
X_Atn	= 4[dB]
X_Pulse	= 6.95[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE

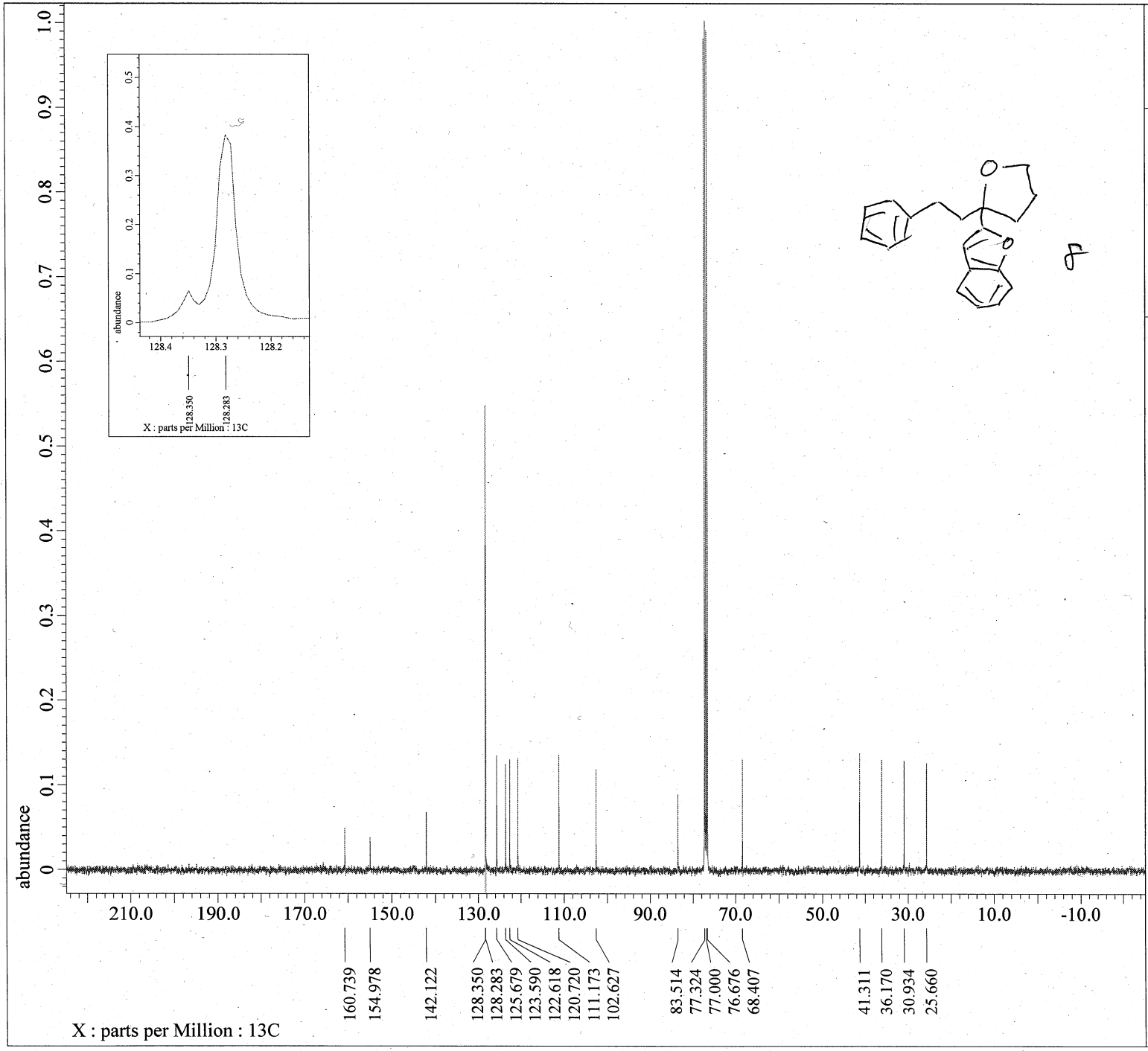


JEOL

```
---- 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

以下に由来:: UES-151-pure12_Proton-1-1.jdf
```

Filename	= UES-151-pure12_Proton-1-2
Author	= element
Experiment	= proton.jxp
Sample_Id	= UES-151-pure12
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 4-DEC-2018 13:15:30
Revision_Time	= 23-APR-2019 17:12:35
Comment	= single_pulse
Data_Format	= 1D COMPLEX
Dim_Size	= 13107
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= DELTA2_NMR
Field_Strength	= 9.4073814[T] (400[MHz])
X_Acq_Duration	= 2.18103808[s]
X_Domain	= 1H
X_Freq	= 400.53219825[MHz]
X_Offset	= 5[ppm]
X_Points	= 16384
X_Prescans	= 1
X_Resolution	= 0.45849727[Hz]
X_Sweep	= 7.51201923[kHz]
X_Sweep_Clipped	= 6.00961538[kHz]
Irr_Domain	= Proton
Irr_Freq	= 400.53219825[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 400.53219825[MHz]
Tri_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 44
Temp_Get	= 19.2[dC]
X_90_Width	= 6.22[us]
X_Acq_Time	= 2.18103808[s]
X_Angle	= 45[deg]
X_Atn	= 0.8[dB]
X_Pulse	= 3.11[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE



8

---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid3(0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm

以下に由来:: UES-151-pureC-1.jdf

Filename	= UES-151-pureC-2.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_Id	= S#333067
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 5-DEC-2018 16:37:36
Revision_Time	= 23-APR-2019 17:15:59
Comment	= single pulse decoupled ga
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= 13C
Dim_Title	= 13C
Dim_Units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_Strength	= 9.20197068[T] (390[MHz])
X_Acq_Duration	= 1.06430464[s]
X_Domain	= 13C
X_Freq	= 98.51479726[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 0.93958061[Hz]
X_Sweep	= 30.78817734[kHz]
Irr_Domain	= 1H
Irr_Freq	= 391.78655441[MHz]
Irr_Offset	= 5[ppm]
Clipped	= FALSE
Scans	= 800
Total_Scans	= 800
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 20.1[dC]
X_90_Width	= 9.11[us]
X_Acq_Time	= 1.06430464[s]
X_Angle	= 30[deg]
X_Atn	= 4.9[dB]
X_Pulse	= 3.03666667[us]
Irr_Atn_Dec	= 22.255[dB]
Irr_Atn_Noe	= 22.255[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE