Solvent-free Zinc-mediated Béchamp Reduction Using Mechanochemistry

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1. Chemicals and instrumentation

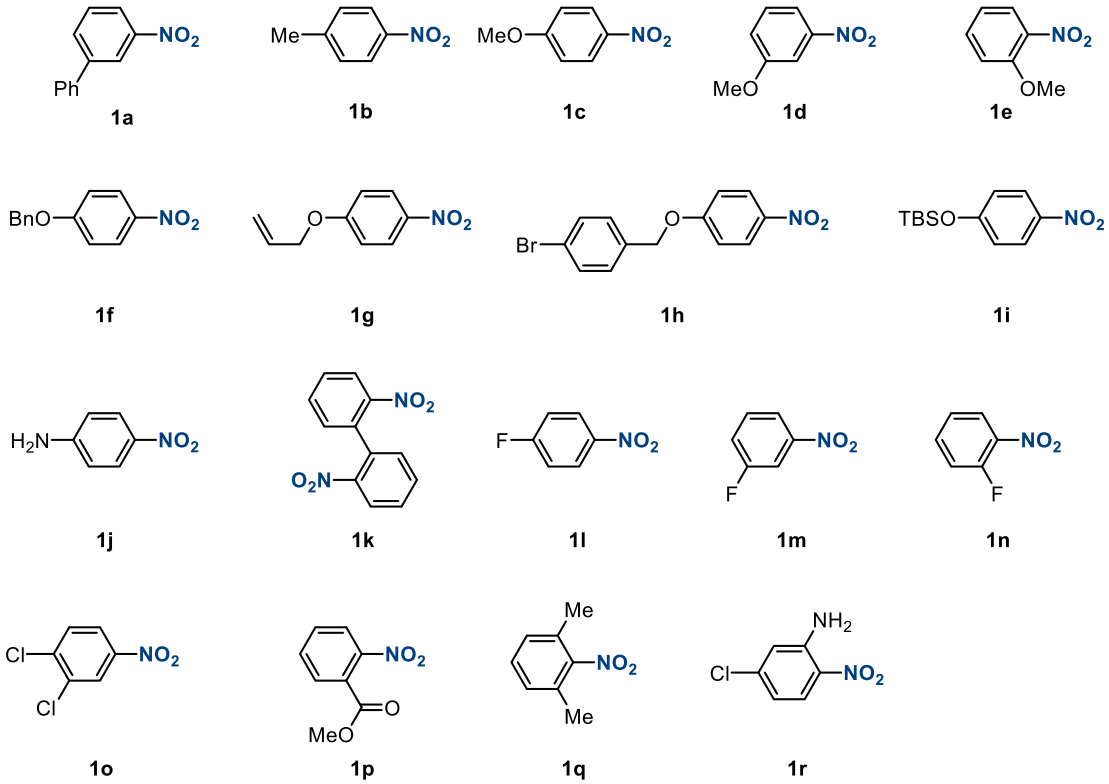
Materials were obtained from commercial suppliers and used as received. Zinc powder (<10 μm) was used in this study. Solvents for the synthesis of starting materials were also purchased from commercial suppliers and dried over molecular sieves (MS 4A) prior to use. All mechanochemical reactions were carried out using grinding vessels in a Retsch MM400 mill (Figure S1). Both jars (25 and 5 mL) and balls (10 mm) are made of stainless (SUS400B and SUS420J2, respectively) (Figure S2). A heat gun Takagi HG-1450B with a temperature control function was used for high-temperature ball-milling reactions (Figure S3). NMR spectra were recorded on JEOL JNM-EC X400P and JNM-ECS400 spectrometers (^{1}H: 396 or 401 MHz, ^{13}C: 392 MHz). Tetramethylsilane (^{1}H), $CDCl_3$ (^{13}C) were employed as external standards, respectively. Multiplicity was recorded as follows: s = singlet, brs = broad singlet, d = doublet, t = triplet, q = quartet, dd = double doublet, dt = double triplet, m = multiplet. Dibromomethane was used as an internal standard to determine NMR yields. High-resolution mass spectra were recorded at the Global Facility Center, Hokkaido University.

Figure S1. Retsch MM400 used in this study.

Figure S2. Stainless jar (25 mL and 5 mL) and balls (10 mm) used in this study.

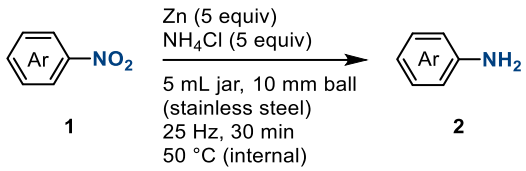
Figure S3. The temperature controllable heat gun Takagi HG-1450B used in this study.

2. List of substrates



1b–**1e** and **1j**–**1r** were purchased from commercial supplier and used as received. **1a** was prepared via Suzuki-Miyaura coupling of the corresponding bromide with phenylboronic acid. **1f** and **1h** were prepared via benzyl protection of the corresponding phenol with benzyl bromide and K_2CO_3. **1g** was prepared via allylation of the corresponding phenol with allyl bromide and K_2CO_3. **1i** was prepared via *tert*-butyl dimethyl silyl (TBS) protection of the corresponding phenol with TBS chloride and imidazole.

3. General procedure of zinc-mediated Béchamp reduction using mechanochemistry



General procedure A: Commercially available Zn metal powder (2.5 mmol, 5.0 equiv) , an aryl nitro compound (0.5 mmol, 1.0 equiv), an ammonium chloride (2.5 mmol, 5.0 equiv) were placed in a stainless milling jar (5 mL) with stainless ball (10 mm, diameter) in air. After the jar was closed without purging with inert gas, the jar was placed in the ball mill (Retsch MM 400, 30 min, 25 Hz). The heat gun was fixed with clamps and placed directly above the ball milling jar (distance between the heat gun and ball milling jar: ca. 1 cm, preset temperature: 70 °C, internal temperature in the jar : 50 °C). After grinding for 30 min, the jar was cooled to room temperature, opened in air, and then extracted with EtOAc or CH_2Cl_2 three times. The resultant solution was dried over $MgSO_4$, filtrated, and evaporated under a vacuum, after removal of the solvents under reduced pressure. NMR yields of the corresponding products were determined by ^1H NMR analysis with dibromomethane as the internal standard. The crude material was purified by flash chromatography (SiO_2, typically EtOAc/hexane, typically 0:100–10:90) to give the corresponding product.

General procedure B: Commercially available Zn metal powder (2.5 mmol, 5.0 equiv) , an aryl nitro compound (0.5 mmol, 1.0 equiv), an ammonium chloride (2.5 mmol, 5.0 equiv) were placed in a stainless milling jar (5 mL) with stainless ball (10 mm, diameter) in air. After the jar was closed without purging with inert gas, the jar was placed in the ball mill (Retsch MM 400, 30 min, 25 Hz). The heat gun was fixed with clamps and placed directly above the ball milling jar (distance between the heat gun and ball milling jar: ca. 1 cm, preset temperature: 70 °C, internal temperature in the jar : 50 °C). After grinding for 30 min, the jar was cooled to room temperature, opened in air, and then $CDCl_3$ was added in the jar. NMR yields of the corresponding products were determined by ^1H NMR analysis with dibromomethane as the internal standard.

Set-up procedure for high-temperature ball milling

The heat gun was fixed with clamps and placed directly above the ball milling jar (distance between the heat gun and ball milling jar: ca. 1 cm). The set-up procedure for high-temperature ball-milling reactions is shown in Figure S4. After the ball milling jar was closed, the jar was placed in the ball mill (Retsch MM400), and a heat gun was placed directly above the ball milling jar. The mechanochemical reduction of aryl nitro compound was conducted while applying heated air to the outside of the milling jar (the preset temperature at 70 °C). The temperature inside the milling jar of the mechanochemical reaction was confirmed by thermography immediately after opening the jar

(preset tempetature: 70 °C, internal temperature in the jar : 50 °C).

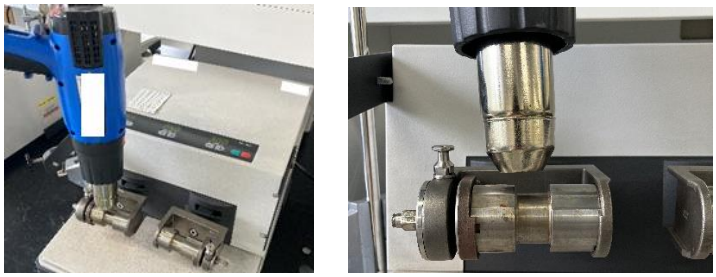
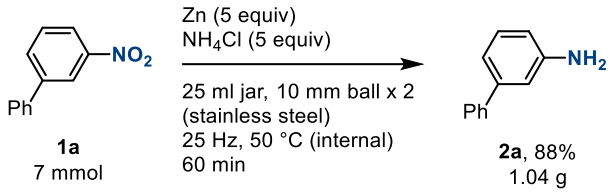


Figure S4. The set-up procedure for a heat gun on MM400.

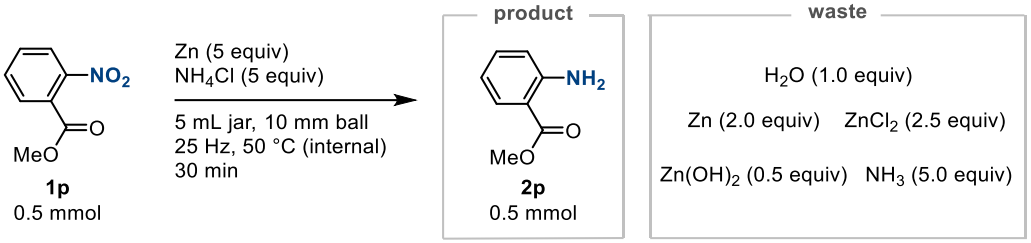
4. Procedure of gram-scale reaction



Commercially available Zn metal powder (2.5 mmol, 5.0 equiv) , an aryl nitro compound (0.5 mmol, 1.0 equiv), an ammonium chloride (2.5 mmol, 5.0 equiv) were placed in a stainless milling jar (5 mL) with stainless ball (10 mm, diameter) in air. After the jar was closed without purging with inert gas, the jar was placed in the ball mill (Retsch MM 400, 60 min, 25 Hz). A heat gun was set in a downward direction approximately 1 cm above the jar and was turned on (preset temperature: 70 °C, internal temperature in the jar: ca. 50 °C). After grinding for 60 min, the jar was cooled to room temperature, opened in air, and then extracted with EtOAc three times. The resultant solution was dried over $MgSO_4$, filtrated, and evaporated under a vacuum, after removal of the solvents under reduced pressure. NMR yields of the corresponding products were determined by ^1H NMR analysis with dibromomethane as the internal standard. The crude material was purified by flash chromatography (SiO_2, EtOAc/hexane, typically 0:100–5:95) to give the corresponding product **2a** (88%, 1.038 g) as a yellow oil.

5. Calculation of E-factors

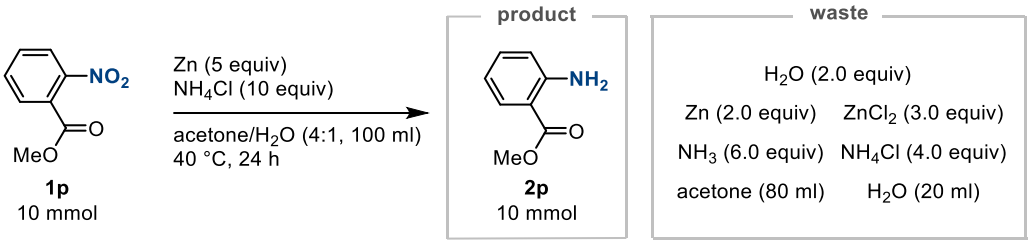
this work



	MW	mmol	mg
product (**2p**)	151.17	0.5	75.59
waste	MW	mmol	mg
H_2O	18.02	1.0	9.01
Zn	65.38	0.5	65.38
$ZnCl_2$	136.28	1.0	170.35
$Zn(OH)_2$	99.39	1.0	24.85
NH_3	17.03	2.0	42.58
total			312.17

E = 312.17 / 75.59 = 4.1

in solution

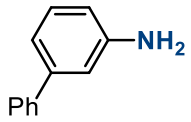


	MW	mmol	ml	density	g
product (**2p**)	151.17	10			1.51
waste	MW	mmol			g
H_2O	18.02	20			0.36
Zn	65.38	20			1.31
$ZnCl_2$	136.28	30			4.09
NH_3	17.03	60			1.02
NH_4Cl	53.49	40			2.14
acetone			80	0.79	63.04
H_2O			20	1.00	20.00
total					91.96

E = 91.96 / 1.51 = 60.9

6. Characterization of products

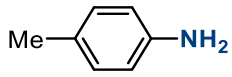
3-Aminobiphenyl (2a).



The reaction was conducted according to the general procedure A using 3-nitro-1,1'- biphenyl (**1a**, 99.5 mg, 0.5 mmol), Zn (164.2 mg, 2.5 mmol), and ammonium chloride (133.4 mg, 2.5 mmol). The resulting crude mixture was analyzed by ^{1}H NMR with dibromomethane as an internal standard to determine the NMR yield of **2a** (95%). The crude mixture was purified by silica-gel column chromatography (SiO_2, EtOAc/hexane, 0:100–10:90) to afford **2a** as a yellow oil (68.5 mg, 0.40 mmol, 81% yield). ^{1}H and ^{13}C NMR were in agreement with the literature.[1]

^{1}H NMR (396 MHz, $CDCl_3$, δ): 3.71 (brs, 2H), 6.65–6.73 (m, 1H), 6.90 (t, *J* = 1.6 Hz, 1H), 6.96–7.04 (m, 1H), 7.22 (t, *J* = 7.5 Hz, 1H), 7.29–7.37 (m, 1H), 7.37–7.48 (m, 2H), 7.53–7.63 (m, 2H). ^{13}C NMR (392 MHz, $CDCl_3$, δ): 113.8 (*C*H), 114.0 (*C*H), 117.6 (*C*H), 127.1 (*C*H), 127.2 (*C*H), 128.6 (*C*H), 129.6 (*C*H), 141.3 (*C*), 142.4 (*C*), 146.7 (*C*). HRMS-ESI (*m*/*z*): $[M+H]^+$ calcd for $C_{12}H_{12}N$, 170.0964; found, 170.0962.

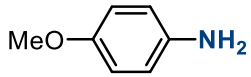
p-Toluidine (2b).



The reaction was conducted according to the general procedure A using 1-methyl-4-nitrobenzene (**2a**, 70.0 mg, 0.5 mmol), Zn (165.7 mg, 2.5 mmol), and ammonium chloride (134.2 mg, 2.5 mmol). The resulting crude mixture was analyzed by ^{1}H NMR with dibromomethane as an internal standard to determine the NMR yield of **2b** (97%). The crude mixture was purified by silica-gel column chromatography (SiO_2, EtOAc/hexane, 0:100–20:80) to afford **2b** as a yellow oil (43.4 mg, 0.41 mmol, 79% yield). ^{1}H and ^{13}C NMR were in agreement with the literature.[2]

^{1}H NMR (401 MHz, $CDCl_3$, δ): 2.24 (s, 3H), 3.52 (brs, 2H), 6.57–6.67 (m, 2H), 6.96 (d, *J* = 8.4 Hz, 2H). ^{13}C NMR (392 MHz, $CDCl_3$, δ): 20.4 (CH_3), 115.2 (*C*H), 127.7 (*C*), 129.7 (*C*H), 143.8 (*C*). HRMS-ESI (*m*/*z*): $[M+H]^+$ calcd for $C_7H_{10}N$, 108.0808; found, 108.0810.

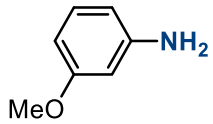
***p*-Anisidine (2c).**



The reaction was conducted according to the general procedure A using 4-nitroanisole (**1c**, 76.2 mg, 0.5 mmol), Zn (164.8 mg, 2.5 mmol), and ammonium chloride (134.6 mg, 2.5 mmol). The resulting crude mixture was analyzed by ^{1}H NMR with dibromomethane as an internal standard to determine the NMR yield of **2c** (78%). The crude mixture was purified by silica-gel column chromatography (SiO_2, EtOAc/hexane, 0:100–50:50) to afford **2c** as a brown solid (39.1 mg, 0.32 mmol, 61% yield). ^{1}H and ^{13}C NMR were in agreement with the literature.[1]

^{1}H NMR (396 MHz, $CDCl_3$, δ): 3.42 (brs, 2H), 3.75 (s, 3H), 6.63–6.69 (m, 2H), 6.72–6.77 (m, 2H). ^{13}C NMR (392 MHz, $CDCl_3$, δ): 55.7 (*C*H$_3$), 114.8 (*C*H), 116.4 (*C*H), 139.9 (*C*), 152.7 (*C*). HRMS-ESI (*m*/*z*): $[M+H]^+$ calcd for $C_7H_{10}ON$, 124.0757; found, 124.0757.

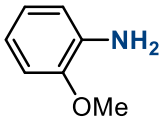
***m*-Anisidine (2d).**



The reaction was conducted according to the general procedure A using 3-nitroanisole (**1d**, 76.0 mg, 0.5 mmol), Zn (164.1 mg, 2.5 mmol), and ammonium chloride (134.1 mg, 2.5 mmol). The resulting crude mixture was analyzed by ^{1}H NMR with dibromomethane as an internal standard to determine the NMR yield of **2d** (80%). The crude mixture was purified by silica-gel column chromatography (SiO_2, EtOAc/hexane, 0:100–20:80) to afford **2d** as a brown oil (39.9 mg, 0.32 mmol, 65% yield). ^{1}H and ^{13}C NMR were in agreement with the literature.[3]

^{1}H NMR (396 MHz, $CDCl_3$, δ): 3.66 (brs, 2H), 3.76 (s, 3H), 6.24 (t, *J* = 2.4 Hz, 1H), 6.27–6.37 (m, 2H), 7.06 (t, *J* = 8.3 Hz, 1H). ^{13}C NMR (392 MHz, $CDCl_3$, δ): 55.1 (*C*H$_3$), 101.0 (*C*H), 103.9 (*C*H), 107.9 (*C*H), 130.1 (*C*H), 147.7 (*C*), 160.7 (*C*). HRMS-ESI (*m*/*z*): $[M+H]^+$ calcd for $C_7H_{10}ON$, 124.0757; found, 124.0757.

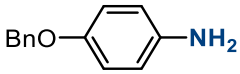
***o*-Anisidine (2e).**



The reaction was conducted according to the general procedure A using 2-nitroanisole (**1e**, 73.6 mg, 0.5 mmol), Zn (163.7 mg, 2.5 mmol), and ammonium chloride (134.7 mg, 2.5 mmol). The resulting crude mixture was analyzed by ^{1}H NMR with dibromomethane as an internal standard to determine the NMR yield of **2e** (97%). The crude mixture was purified by silica-gel column chromatography (SiO_2, EtOAc/hexane, 0:100–20:80) to afford **2e** as a yellow oil (45.3 mg, 0.37 mmol, 77% yield). ^{1}H and ^{13}C NMR were in agreement with the literature.[2]

^{1}H NMR (396 MHz, $CDCl_3$, δ): 3.74 (brs, 2H), 3.84 (s, 3H), 6.68–6.77 (m, 2H), 6.76–6.85 (m, 2H). ^{13}C NMR (392 MHz, $CDCl_3$, δ): 55.3 (*C*H$_3$), 110.3 (*C*H), 115.0 (*C*H), 118.4 (*C*H), 121.0 (*C*H), 136.1 (*C*), 147.2 (*C*). HRMS-ESI (*m*/*z*): $[M+H]^+$ calcd for $C_7H_{10}ON$, 124.0757; found, 124.0757.

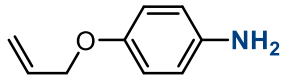
4-(Benzyloxy)aniline (2f).



The reaction was conducted according to the general procedure A using benzyl 4-nitrophenyl ether (**1f**, 113.7 mg, 0.5 mmol), Zn (164.9 mg, 2.5 mmol), and ammonium chloride (136.6 mg, 2.6 mmol). The resulting crude mixture was analyzed by ^{1}H NMR with dibromomethane as an internal standard to determine the NMR yield of **2f** (75%). The crude mixture was purified by silica-gel column chromatography (SiO_2, EtOAc/hexane, 0:100–10:90) to afford **2f** as a brown solid (62.6 mg, 0.31 mmol, 63% yield). ^{1}H and ^{13}C NMR were in agreement with the literature.[4]

^{1}H NMR (401 MHz, $CDCl_3$, δ): 3.43 (brs, 2H), 4.99 (s, 2H), 6.61–6.69 (m, 2H), 6.79–6.86 (m, 2H), 7.27–7.47 (m, 5H). ^{13}C NMR (392 MHz, $CDCl_3$, δ): 70.7 (*C*H$_2$), 116.0 (*C*H), 116.3 (*C*H), 127.5 (*C*H), 127.8 (*C*H), 128.5 (*C*H), 137.5 (*C*), 140.2 (*C*), 152.0 (*C*). HRMS-ESI (*m*/*z*): $[M+H]^+$ calcd for $C_{13}H_{14}ON$, 200.1070; found, 200.1065.

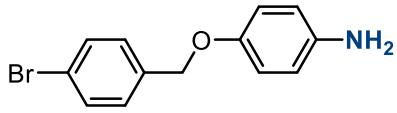
4-(Allyloxy)aniline (2g).



The reaction was conducted according to the general procedure A using 1-allyloxy-4-nitrobenzene (**1g**, 90.4 mg, 0.5 mmol), Zn (164.9 mg, 2.5 mmol), and ammonium chloride (135.4 mg, 2.5 mmol). The resulting crude mixture was analyzed by ^{1}H NMR with dibromomethane as an internal standard to determine the NMR yield of **2g** (97%). The crude mixture was purified by silica-gel column chromatography (SiO_2, EtOAc/hexane, 0:100–40:60) to afford **2g** as a brown oil (63.2 mg, 0.42 mmol, 84% yield). ^{1}H and ^{13}C NMR were in agreement with the literature.[5]

^{1}H NMR (401 MHz, $CDCl_3$, δ): 3.43 (brs, 2H), 4.43–4.52 (m, 2H), 5.23–5.29 (m, 1H), 5.35–5.43 (m, 1H), 5.99–6.11 (m, 1H), 6.61–6.68 (m, 2H), 6.73–6.80 (m, 2H). ^{13}C NMR (392 MHz, $CDCl_3$, δ): 69.5 (*C*H_2), 115.9 (*C*H), 116.3 (*C*H), 117.3 (*C*H_2), 133.8 (*C*H), 140.1 (*C*), 151.7 (*C*). HRMS-ESI (*m*/*z*): $[M+H]^+$ calcd for $C_9H_{12}ON$, 150.0913; found, 150.0911.

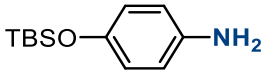
4-[(4-Bromobenzyl)oxy]aniline (2h).



The reaction was conducted according to the general procedure A (reaction time is 60 min) using 1-bromo-4-[(4-nitrophenoxy)methyl]benzene (**1h**, 153.2 mg, 0.5 mmol), Zn (164.8 mg, 2.5 mmol), and ammonium chloride (134.6 mg, 2.5 mmol). The crude mixture was purified by silica-gel column chromatography (SiO_2, EtOAc/hexane, 0:100–20:80) to afford **2h** as a brown solid (66.6 mg, 0.24 mmol, 48% yield).

^{1}H NMR (401 MHz, $CDCl_3$, δ): 3.44 (brs, 2H), 4.93 (s, 2H), 6.63 (d, *J* = 8.4 Hz, 2H), 6.78 (d, *J* = 8.8 Hz, 2H), 7.29 (d, *J* = 7.6 Hz, 2H), 7.49 (d, *J* = 8.4 Hz, 2H). ^{13}C NMR (392 MHz, $CDCl_3$, δ): 70.0 (*C*H_2), 116.0 (*C*H), 116.3 (*C*H), 121.6 (*C*), 129.1 (*C*H), 131.6 (*C*H), 136.5 (*C*), 140.4 (*C*), 151.6 (*C*). HRMS-ESI (*m*/*z*): $[M+H]^+$ calcd for $C_{13}H_{13}ONBr$, 278.0175; found, 278.0171.

4-[(*tert*-Butyldimethylsilyl)oxy]aniline (2i).

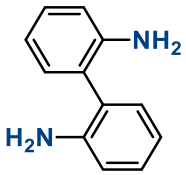


The reaction was conducted according to the general procedure A using *tert*-butyldimethyl(4-nitrophenoxy)silane (**1i**, 124.5 mg, 0.5 mmol), Zn (194.5 mg, 3.0 mmol), and ammonium chloride (137.2 mg, 2.6 mmol). The resulting crude mixture was analyzed by ^{1}H NMR with dibromomethane as an internal standard to determine the NMR yield of **2i** (99%). The crude mixture was purified by silica-gel column chromatography (SiO_2, EtOAc/hexane, 0:100–10:90) to afford **2i** as a brown oil (94.0 mg, 0.42 mmol, 86% yield). ^{1}H and ^{13}C NMR were in agreement with the literature.[6]
^{1}H NMR (396 MHz, $CDCl_3$, δ): 0.15 (s, 6H), 0.97 (s, 9H), 3.40 (brs, 2H), 6.55–6.60 (m, 2H), 6.63–6.68 (m, 2H). ^{13}C NMR (392 MHz, $CDCl_3$, δ): –4.5 (*C*H_3), 18.1 (*C*), 25.7 (*C*H_3), 116.2 (*C*H), 120.6 (*C*H), 140.2 (*C*), 148.1 (*C*). HRMS-ESI (*m*/*z*): $[M+H]^+$ calcd for $C_{12}H_{22}ONSi$, 224.1465; found, 224.1459.

1,4-Diaminobenzene (2j).

The reaction was conducted according to the general procedure A (work up was carried out by passing through a short silica gel column eluting with EtOAc) using 4-nitro-aniline (**1j**, 69.3 mg, 0.5 mmol), Zn (161.5 mg, 2.5 mmol), and ammonium chloride (135.4 mg, 2.5 mmol). The crude mixture was evaporated under a vacuum, after removal of the solvents under reduced pressure to afford **2j** as a brown solid (28.7 mg, 0.27 mmol, 53% yield). ^{1}H and ^{13}C NMR were in agreement with the literature.[1]
^{1}H NMR (401 MHz, $CDCl_3$, δ): 3.33 (brs, 4H), 6.52–6.63 (m, 4H). ^{13}C NMR (392 MHz, $CDCl_3$, δ): 116.7 (*C*H), 138.5 (*C*). HRMS-ESI (*m*/*z*): $[M+H]^+$ calcd for $C_6H_9N_2$, 109.0760; found, 109.0757.

1,1'-Biphenyl-2,2'-diamine (2k).

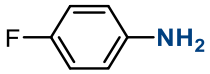


The reaction was conducted according to the general procedure A using 1-nitro-2-(2-nitrophenyl)benzene (**1k**, 122.5 mg, 0.5 mmol), Zn (325.5 mg, 5.0 mmol), and ammonium chloride (267.1 mg, 5.0 mmol). The resulting crude mixture was analyzed by ^{1}H NMR with dibromomethane

as an internal standard to determine the NMR yield of **2k** (94%). The crude mixture was purified by silica-gel column chromatography (SiO_2, EtOAc/CH_2Cl_2, 0:100–5:95) to afford **2k** as a white solid (71.5 mg, 0.39 mmol, 77% yield). ^1H and ^{13}C NMR were in agreement with the literature.[7]

^1H NMR (396 MHz, $CDCl_3$, δ): 3.71 (brs, 4H), 6.79 (dd, *J* = 0.79 Hz, 8.3 Hz, 2H), 6.84 (td, *J* = 1.6 Hz, 7.3 Hz, 2H), 7.13 (dd, *J* = 1.6 Hz, 7.7 Hz, 2H), 7.18 (td, *J* = 1.2 Hz, 7.7 Hz, 2H). ^{13}C NMR 392 MHz, $CDCl_3$, δ): 115.5 (*C*H), 118.7 (*C*H), 124.5 (*C*), 128.8 (*C*H), 131.0 (*C*H), 144.1 (*C*). HRMS-ESI (*m*/*z*): $[M+H]^+$ calcd for $C_{12}H_{13}N_2$, 185.1073; found, 185.1071.

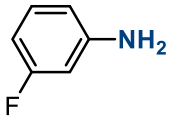
4-Fluoroaniline (2l).



The reaction was conducted according to the general procedure B using 4-fluoronitrobenzene (**1l**, 69.8 mg, 0.5 mmol), Zn (165.3 mg, 2.5 mmol), and ammonium chloride (133.4 mg, 2.5 mmol). The resulting crude mixture was analyzed by ^1H NMR with dibromomethane as an internal standard to determine the NMR yield of **2l** (65%). Because of the low boiling point of the product, only crude NMR yield was collected. ^1H was in agreement with the literature.[2]

^1H NMR (401 MHz, $CDCl_3$, δ): 3.54 (brs, 2H), 6.59–6.67 (m, 2H), 6.81–6.90 (m, 2H).

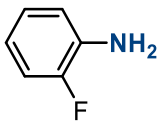
3-Fluoroaniline (2m).



The reaction was conducted according to the general procedure B using 3-fluoronitrobenzene (**1m**, 69.0 mg, 0.5 mmol), Zn (165.7 mg, 2.5 mmol), and ammonium chloride (135.7 mg, 2.5 mmol). The resulting crude mixture was analyzed by ^1H NMR with dibromomethane as an internal standard to determine the NMR yield of **2m** (85%). Because of the low boiling point of the product, only crude NMR yield was collected. ^1H was in agreement with the literature.[2]

^1H NMR (401 MHz, $CDCl_3$, δ): 3.76 (brs, 2H), 6.38 (dt, *J* = 2.4 Hz, 10.7 Hz, 1H), 6.40–6.48 (m, 2H), 7.04–7.12 (m, 1H).

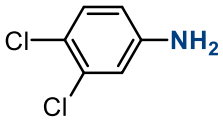
2-Fluoroaniline (2n).



The reaction was conducted according to the general procedure B using 2-fluoronitrobenzene (**1n**, 69.1 mg, 0.5 mmol), Zn (163.7 mg, 2.5 mmol), and ammonium chloride (132.9 mg, 2.5 mmol). The resulting crude mixture was analyzed by ^{1}H NMR with dibromomethane as an internal standard to determine the NMR yield of **2n** (89%). Because of the low boiling point of the product, only crude NMR yield was collected. ^{1}H was in agreement with the literature.[2]

^{1}H NMR (401 MHz, $CDCl_3$, δ): 3.71 (brs, 2H), 6.65–6.74 (m, 1H), 6.74–6.82 (m, 1H), 6.89–7.03 (m, 2H).

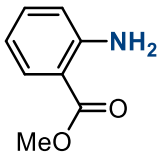
3,4-Dichloroaniline (2o).



The reaction was conducted according to the general procedure A using 3,4-dichloronitrobenzene (**1o**, 96.5 mg, 0.5 mmol), Zn (165.0 mg, 2.5 mmol), and ammonium chloride (134.0 mg, 2.5 mmol). The resulting crude mixture was analyzed by ^{1}H NMR with dibromomethane as an internal standard to determine the NMR yield of **2o** (96%). The crude mixture was purified by silica-gel column chromatography (SiO_2, EtOAc/hexane, 0:100–15:85) to afford **2o** as a yellow solid (66.4 mg, 0.41 mmol, 82% yield). ^{1}H and ^{13}C NMR were in agreement with the literature.[8]

^{1}H NMR (401 MHz, $CDCl_3$, δ): 3.72 (brs, 2H), 6.51 (dd, J = 2.4 Hz, 8.2 Hz, 1H), 6.77 (d, J = 2.8 Hz, 1H), 7.17 (d, J = 8.4 Hz, 1H). ^{13}C NMR (99 MHz, $CDCl_3$, δ): 114.6 (*C*H), 116.4 (*C*H), 121.0 (*C*), 130.7 (*C*H), 132.6 (*C*), 146.0 (*C*). HRMS-EI (*m*/*z*): $[M]^+$ calcd for $C_6H_5Cl_2N$, 160.9794; found, 160.9795.

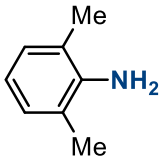
2-Carbomethoxyaniline (2p).



The reaction was conducted according to the general procedure A using methyl 2-nitrobenzoate (**1p**, 89.7 mg, 0.5 mmol), Zn (167.5 mg, 2.6 mmol), and ammonium chloride (135.2 mg, 2.5 mmol). The resulting crude mixture was analyzed by ^{1}H NMR with dibromomethane as an internal standard to determine the NMR yield of **2p** (97%). The crude mixture was purified by silica-gel column chromatography (SiO_2, EtOAc/hexane, 0:100–20:80) to afford **2p** as a yellow oil (65.4 mg, 0.43 mmol, 87% yield). ^{1}H and ^{13}C NMR were in agreement with the literature.[9]

^{1}H NMR (401 MHz, $CDCl_3$, δ): 3.87 (s, 3H), 5.71 (brs, 2H), 6.61–6.69 (m, 2H), 7.23–7.29 (m, 1H), 7.85 (dd, *J* = 1.6 Hz, 8.2 Hz, 1H). ^{13}C NMR (392 MHz, $CDCl_3$, δ): 51.5 (*C*H$_3$), 110.7 (*C*), 116.2 (*C*H), 116.6 (*C*H), 131.2 (*C*H), 134.0 (*C*H), 150.4 (*C*), 168.6 (*C*). HRMS-ESI (*m*/*z*): $[M+H]^+$ calcd for $C_8H_{10}O_2N$, 152.0706; found, 152.0704.

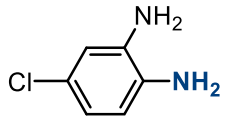
2,6-Dimethylaniline (2q).



The reaction was conducted according to the general procedure A using 2,6-dimethylnitrobenzene (**1q**, 75.0 mg, 0.5 mmol), Zn (166.5 mg, 2.5 mmol), and ammonium chloride (135.5 mg, 2.5 mmol). The resulting crude mixture was analyzed by ^{1}H NMR with dibromomethane as an internal standard to determine the NMR yield of **2q** (>99%). The crude mixture was purified by silica-gel column chromatography (SiO_2, EtOAc/hexane, 0:100–20:80) to afford **2q** as a pale yellow oil (50.7 mg, 0.42 mmol, 84% yield). ^{1}H and ^{13}C NMR were in agreement with the literature.[3]

^{1}H NMR (401 MHz, $CDCl_3$, δ): 2.18 (s, 6H), 3.55 (brs, 2H), 6.64 (t, *J* = 7.6 Hz, 1H), 6.94 (d, *J* = 7.6 Hz, 2H). ^{13}C NMR (392 MHz, $CDCl_3$, δ): 17.6 (*C*H$_3$), 117.9 (*C*H), 121.6 (*C*), 128.2 (*C*H), 142.7 (*C*). HRMS-ESI (*m*/*z*): $[M+H]^+$ calcd for $C_8H_{12}N$, 122.0964; found, 122.0965.

4-Chloro-1,2-phenylenediamine (2r).



The reaction was conducted according to the general procedure A using 5-chloro-2-nitroaniline (**1r**, 86.4 mg, 0.5 mmol), Zn (162.3 mg, 2.5 mmol), and ammonium chloride (134.3 mg, 2.5 mmol). The resulting crude mixture was analyzed by ^{1}H NMR with dibromomethane as an internal standard to determine the NMR yield of **2r** (77%). The crude mixture was purified by silica-gel column chromatography (SiO_2, MeOH/CH_2Cl_2, 0:100–3:97) to afford **2r** as a brown solid (48.9 mg, 0.34 mmol, 70% yield). ^{1}H and ^{13}C NMR were in agreement with the literature.[10]

^{1}H NMR (396 MHz, $CDCl_3$, δ): 3.40 (brs, 4H), 6.59–6.71 (m, 3H). ^{13}C NMR (392 MHz, $CDCl_3$, δ): 116.2 (*C*H), 117.5 (*C*H), 119.6 (*C*H), 124.8 (*C*), 133.0 (*C*), 136.1 (*C*). HRMS-ESI (*m*/*z*): $[M+H]^+$ calcd for $C_6H_8N_2Cl$, 143.0371; found, 143.0368.

7. References

1. Choi, K.; Brunn, J. N.; Borate, K.; Kaduskar, R.; Pueyo, C. L.; Shinde, H.; Goetz, R.; Hartwig, J. F. *J. Am. Chem. Soc.* **2024**, *146*, 19414–19424.
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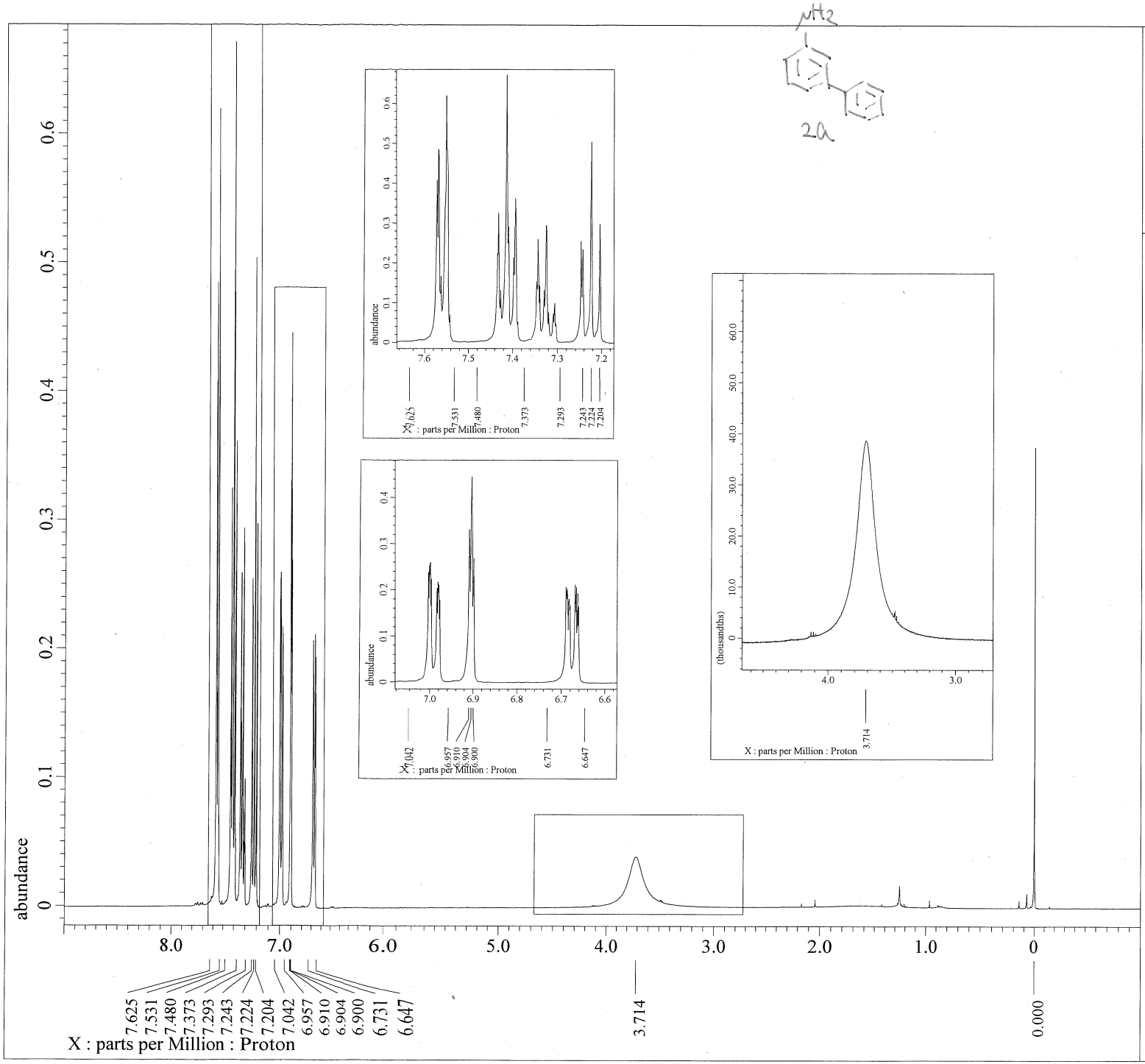
10. Goyal, V.; Bhatt, T.; Dewangan, C.; Narani, A.; Naik, G.; Balaraman, E.; Natte, K.; Jagadeesh, R. V. *J. Org. Chem*. **2023**, *88*, 2245–2259.

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Irr_Freq	= 395.88430144[MHz]
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X_Atn	= 5[dB]
X_Pulse	= 3.17[us]
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Tri_Mode	= Off
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Dante_Presat	= FALSE
Decimation_Rate	= 0
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Initial_Wait	= 1[s]
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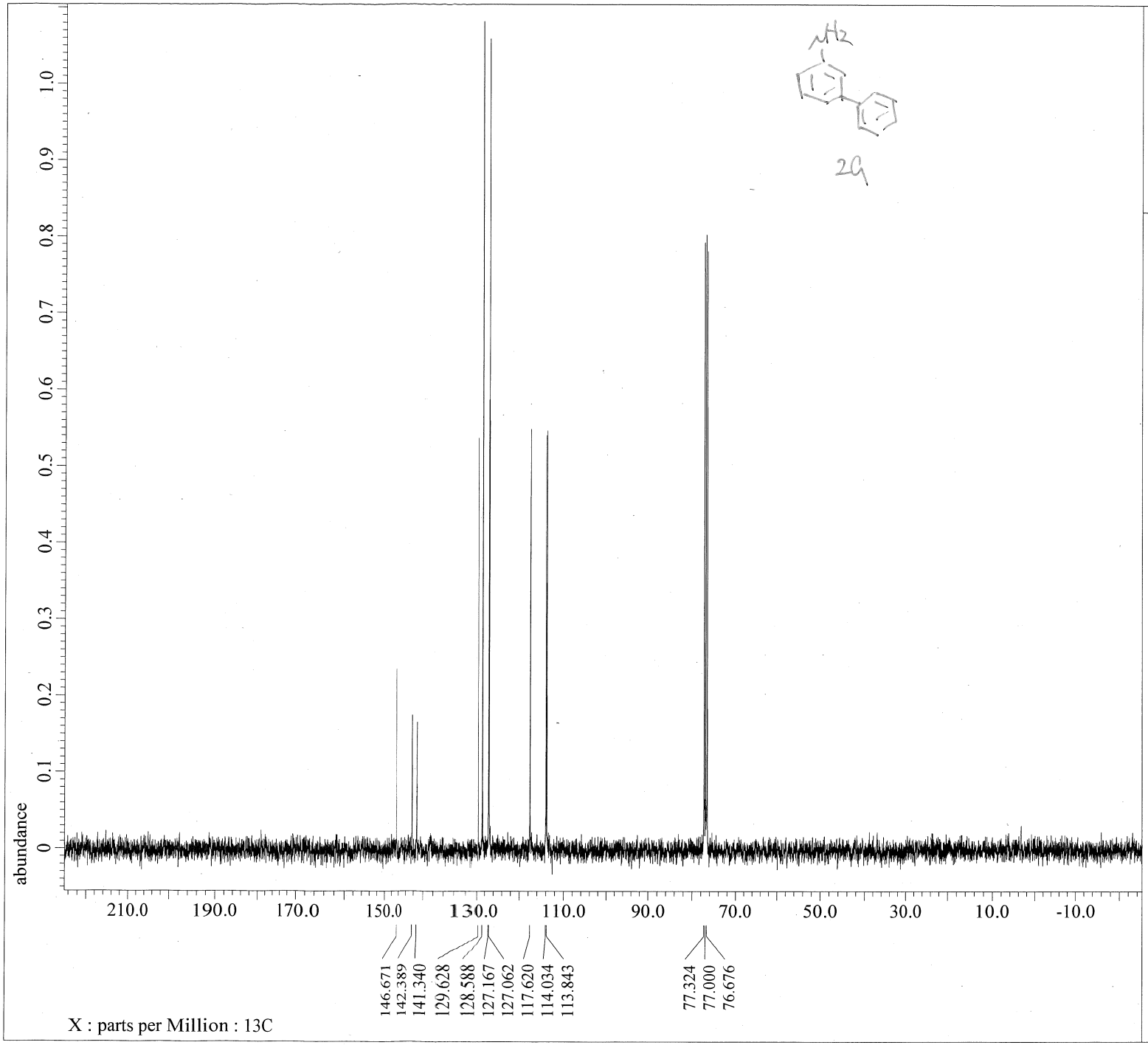
Presat_Time	= 5[s]



29

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fft( 1, TRUE, TRUE )
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ppm

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Experiment          = single_pulse_dec
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Revision_Time       =  5-AUG-2024 15:33:37

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X_Offset            = 100[ppm]
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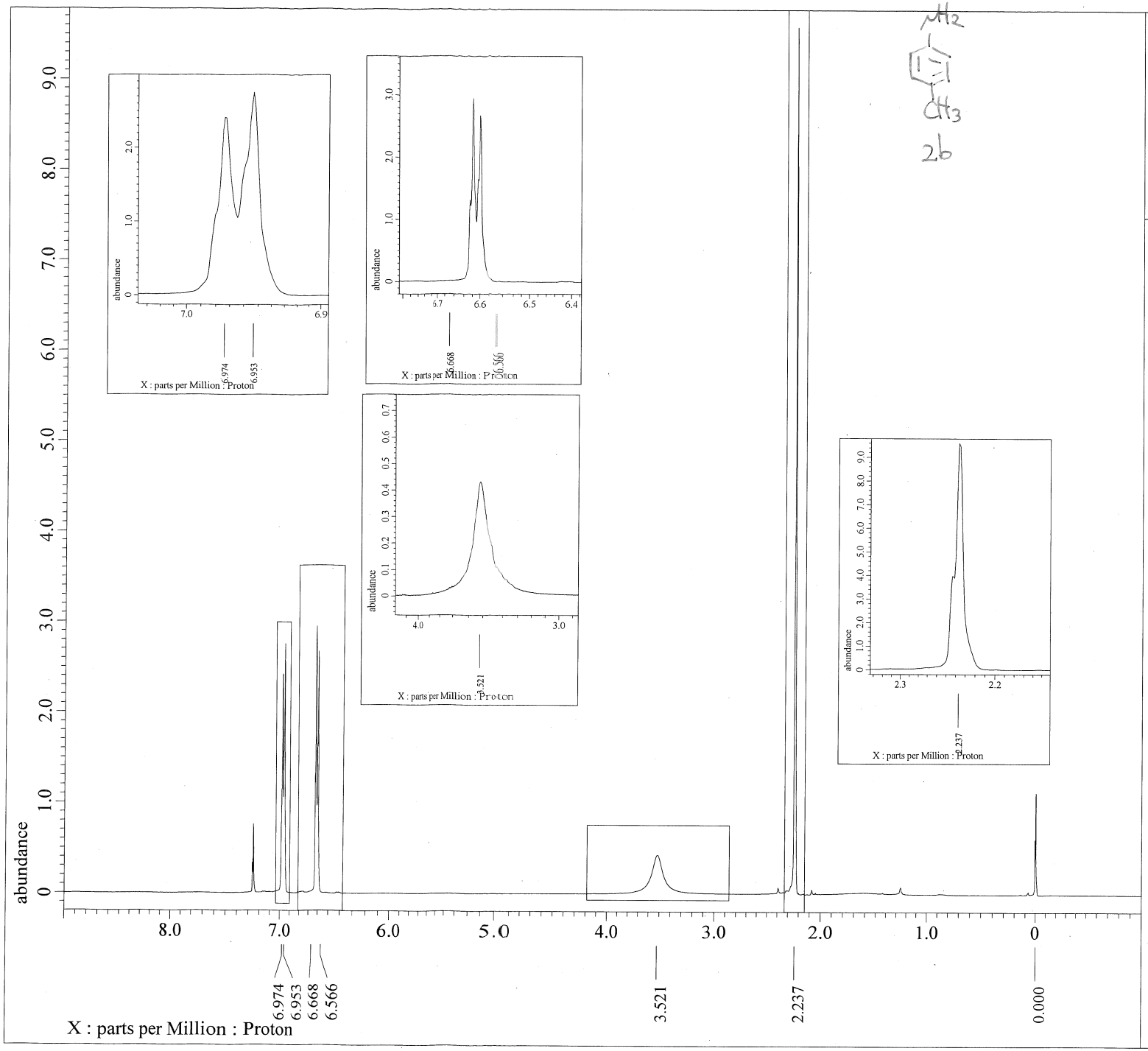


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machinephase
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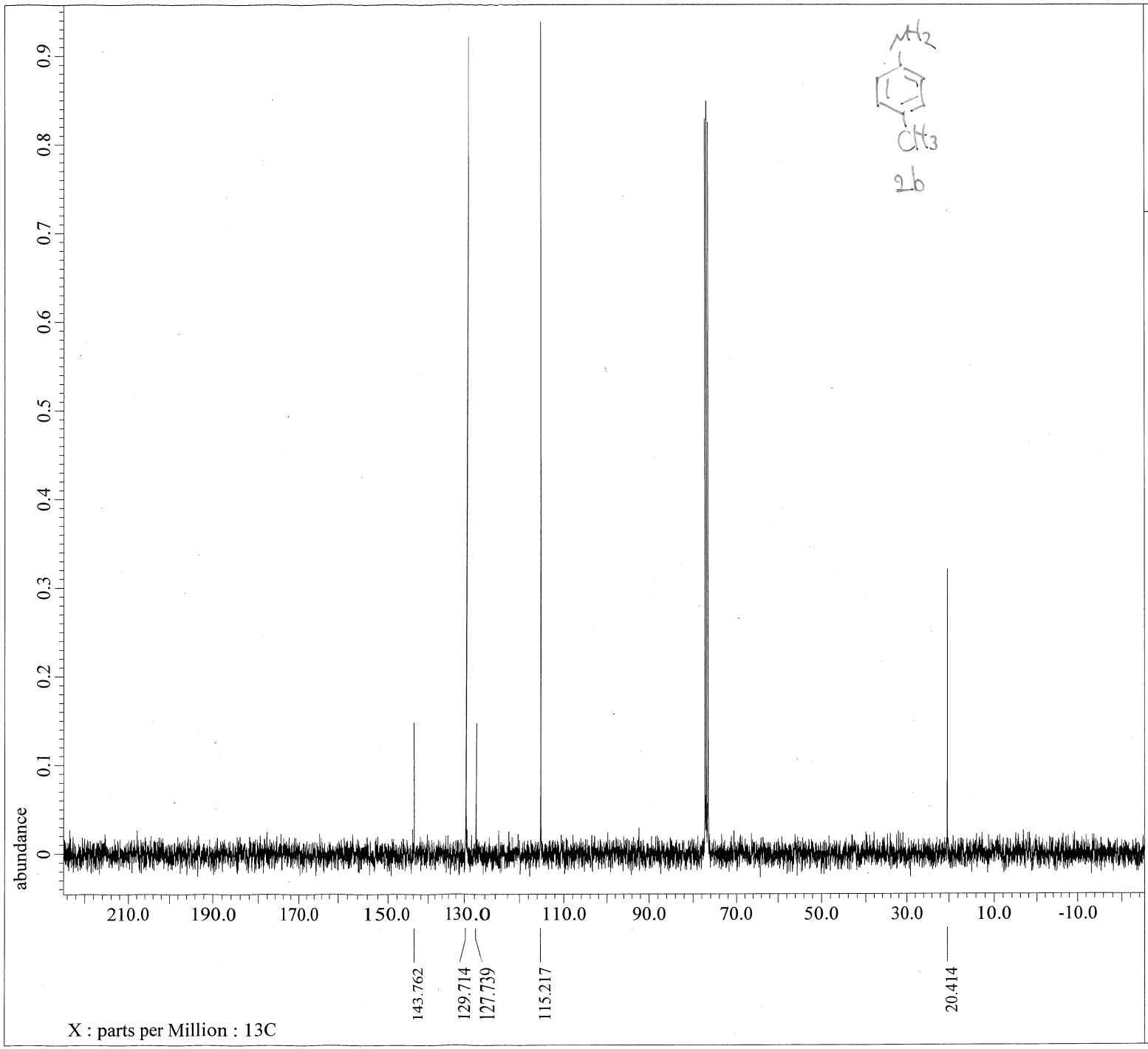


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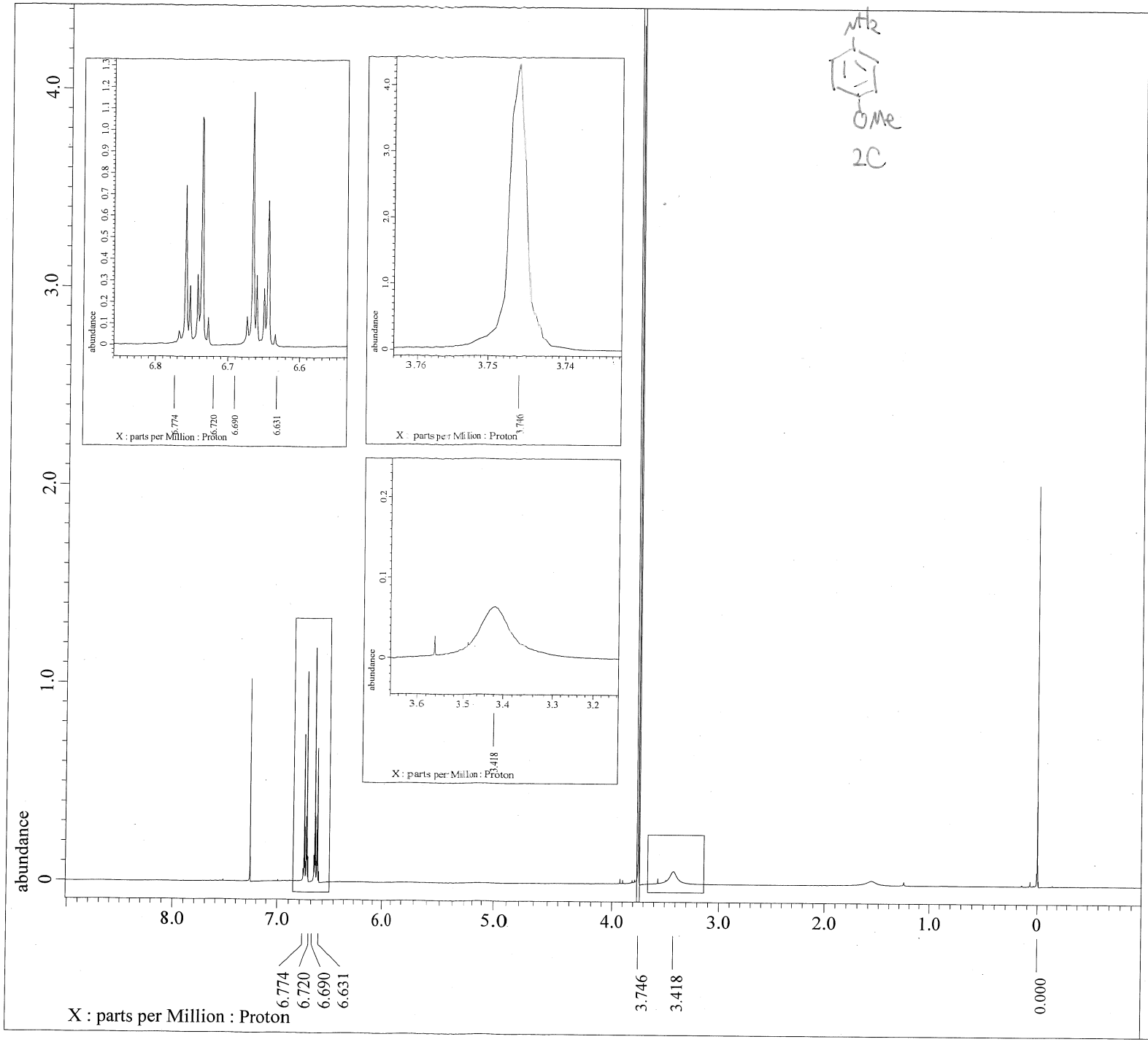


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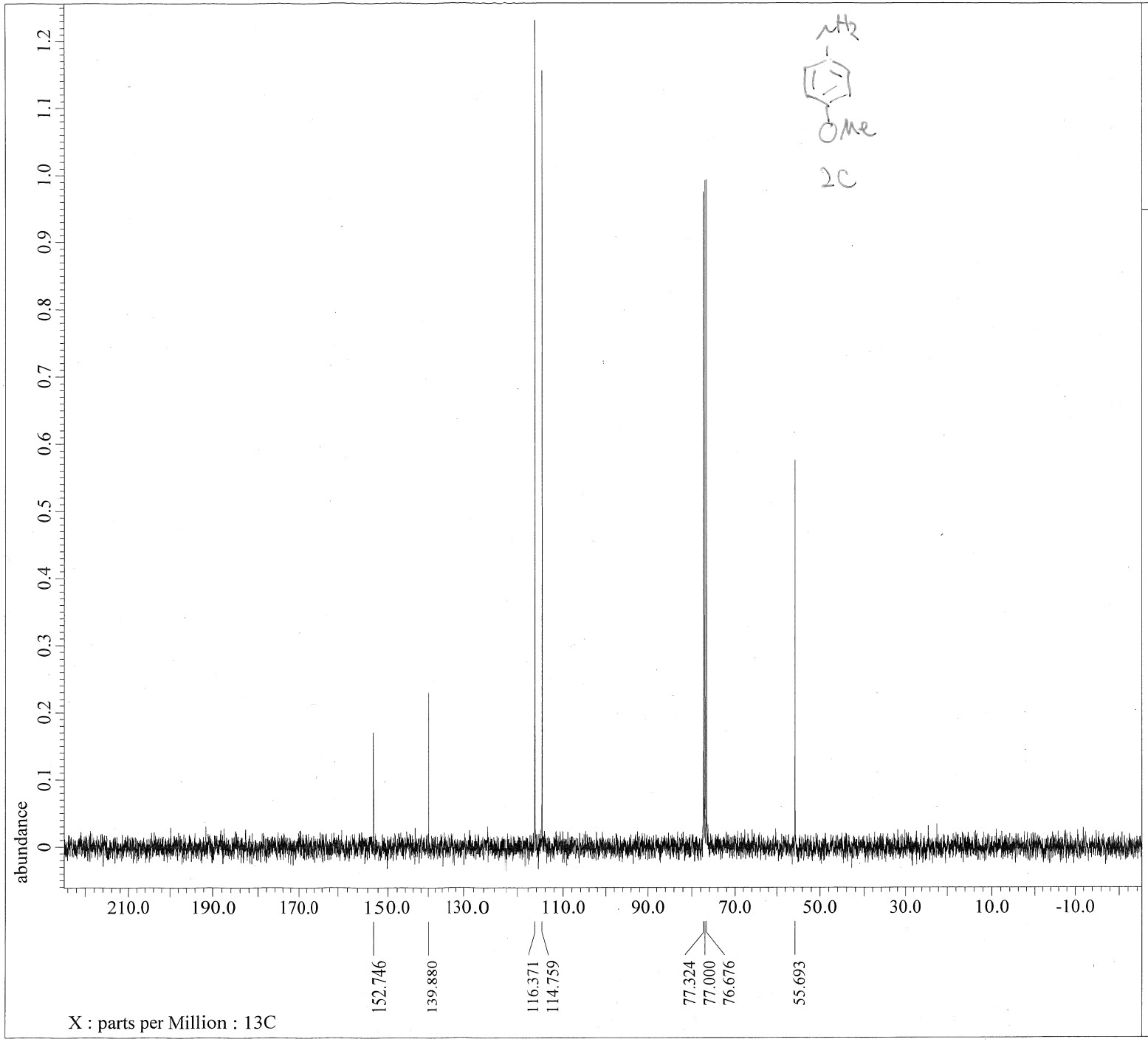
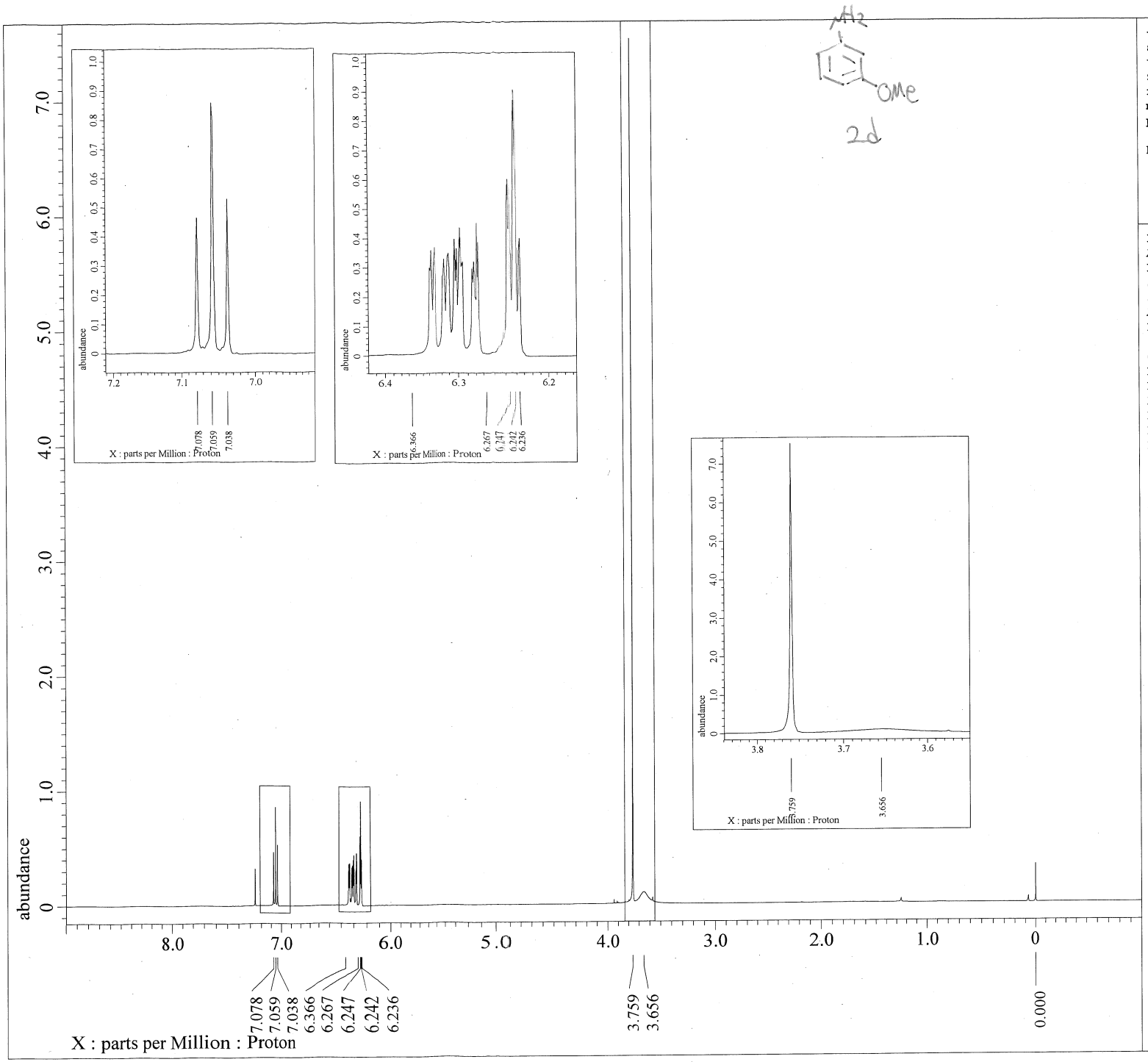
Experiment_Path	= C:\Program Files\JEOL
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180
Presat_Time	= 5[s]



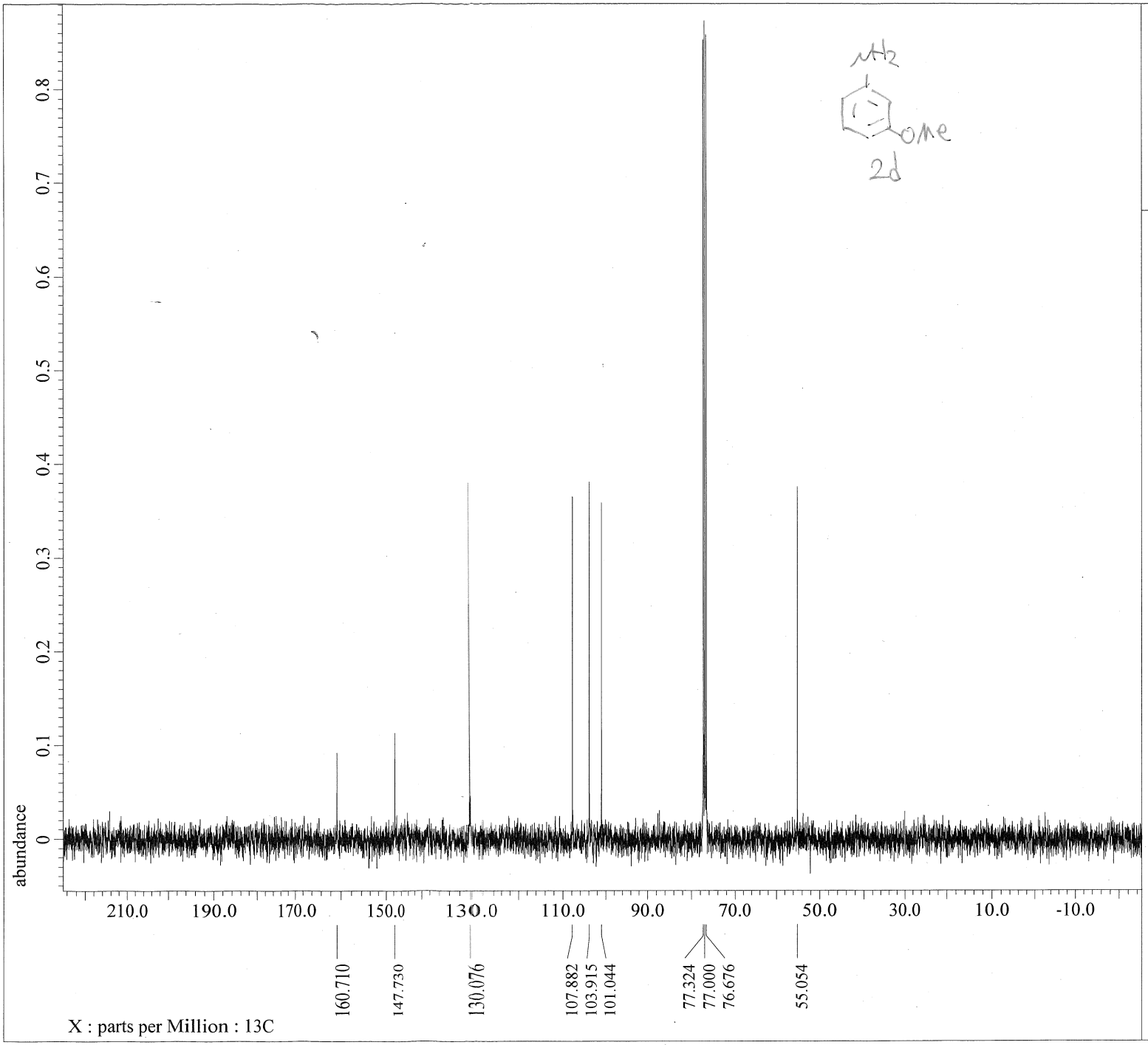

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-113-13C-1.jdf
```

```
Filename            = ASH-113-13C-2.jdf
Author              = element
Experiment          = single_pulse_dec
Sample_Id           = 1
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 29-JUL-2024 18:23:32
Revision_Time       = 29-JUL-2024 12:27:23

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               = 57
Total_Scans         = 57

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 22.2[dC]
X_90_Width          = 9.46[us]
X_Acq_Time          = 1.06430464[s]
X_Angle             = 30[deg]
X_Atn               = 4.9[dB]
X_Pulse             = 3.15333333[us]
Irr_Atn_Dec         = 22.45[dB]
Irr_Atn_Noe         = 22.45[dB]
Irr_Noise           = WALTZ
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.06430464[s]
```



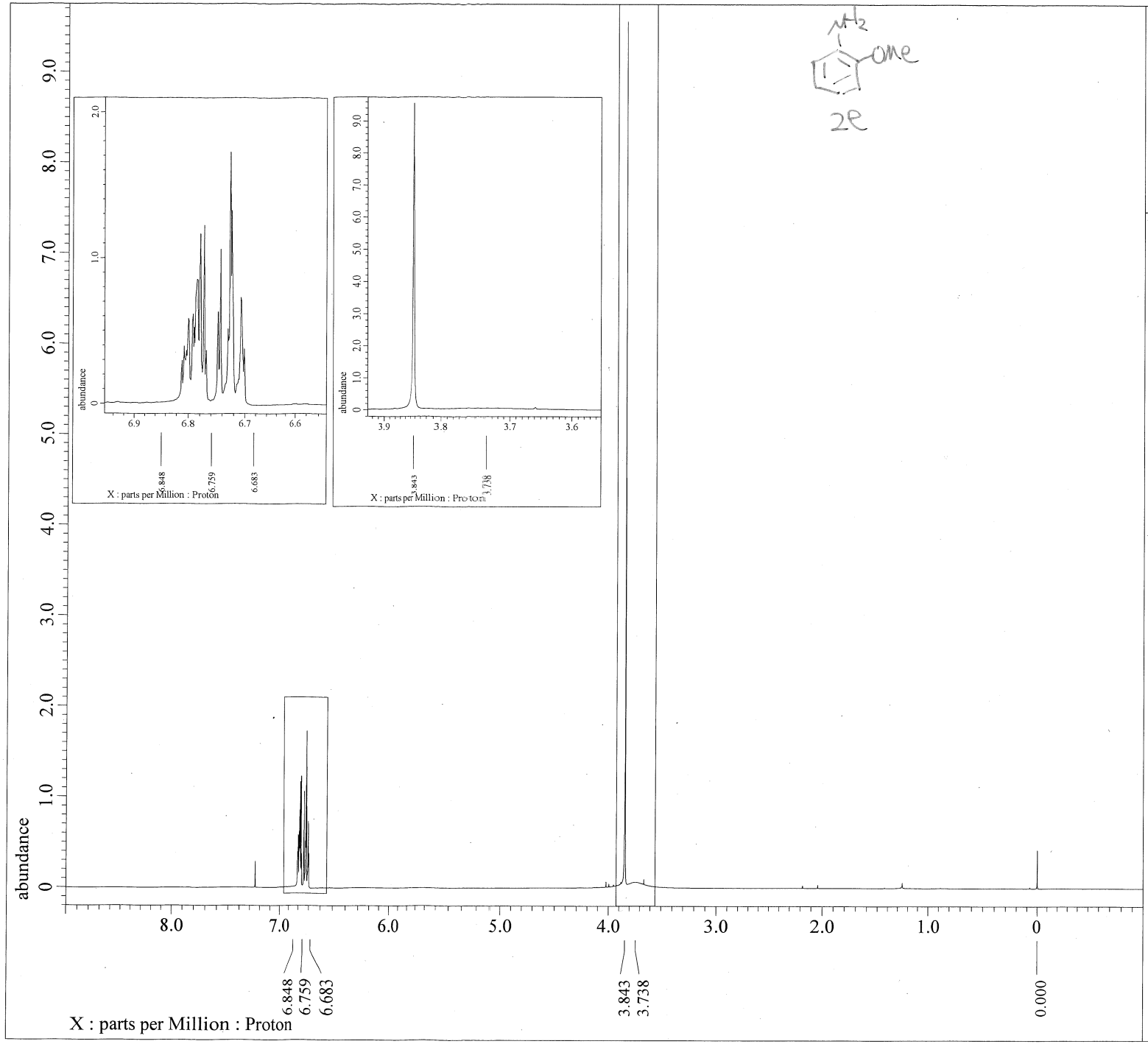

```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-111-pure3_Proton-1-1.jdf
```

Filename	= ASH-111-pure3_Proton-
Author	= element
Experiment	= proton_auto.jxp
Sample_Id	= ASH-105-pure3
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 27-JUL-2024 14:54:47
Revision_Time	= 12-NOV-2024 09:34:37
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	= 9.2982153[T] (400[MHz
X_Acq_Duration	= 2.20725248[s]
X_Domain	= Proton
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]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 46
Temp_Get	= 21.5[dC]
X_90_Width	= 6.34[us]
X_Acq_Time	= 2.20725248[s]
X_Angle	= 45[deg]
X_Atn	= 5[dB]
X_Pulse	= 3.17[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 500
Dante_Presat	= FALSE
Decimation_Rate	= 0
Experiment_Path	= C:\Program Files\JEOL
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180
Presat_Time	= 5[s]



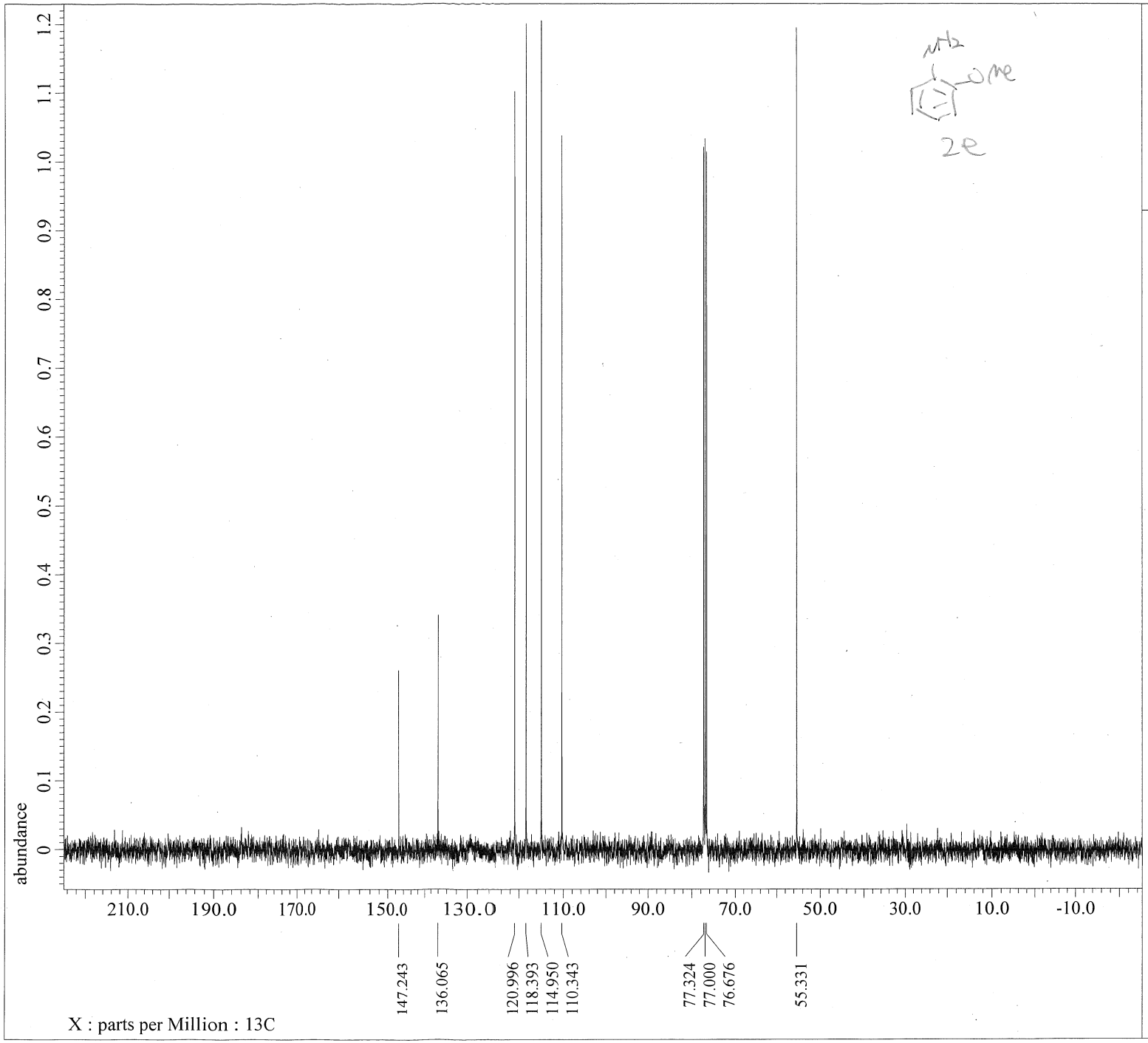

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-111-13C-1.jdf
```

```
Filename            = ASH-111-13C-2.jdf
Author              = element
Experiment          = single_pulse_dec
Sample_Id           = 1
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 27-JUL-2024 21:45:20
Revision_Time       = 27-JUL-2024 15:45:50

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               = 61
Total_Scans         = 61

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 22.7[dC]
X_90_Width          = 9.46[us]
X_Acq_Time          = 1.06430464[s]
X_Angle             = 30[deg]
X_Atn               = 4.9[dB]
X_Pulse             = 3.15333333[us]
Irr_Atn_Dec         = 22.45[dB]
Irr_Atn_Noe         = 22.45[dB]
Irr_Noise           = WALTZ
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.06430464[s]
```



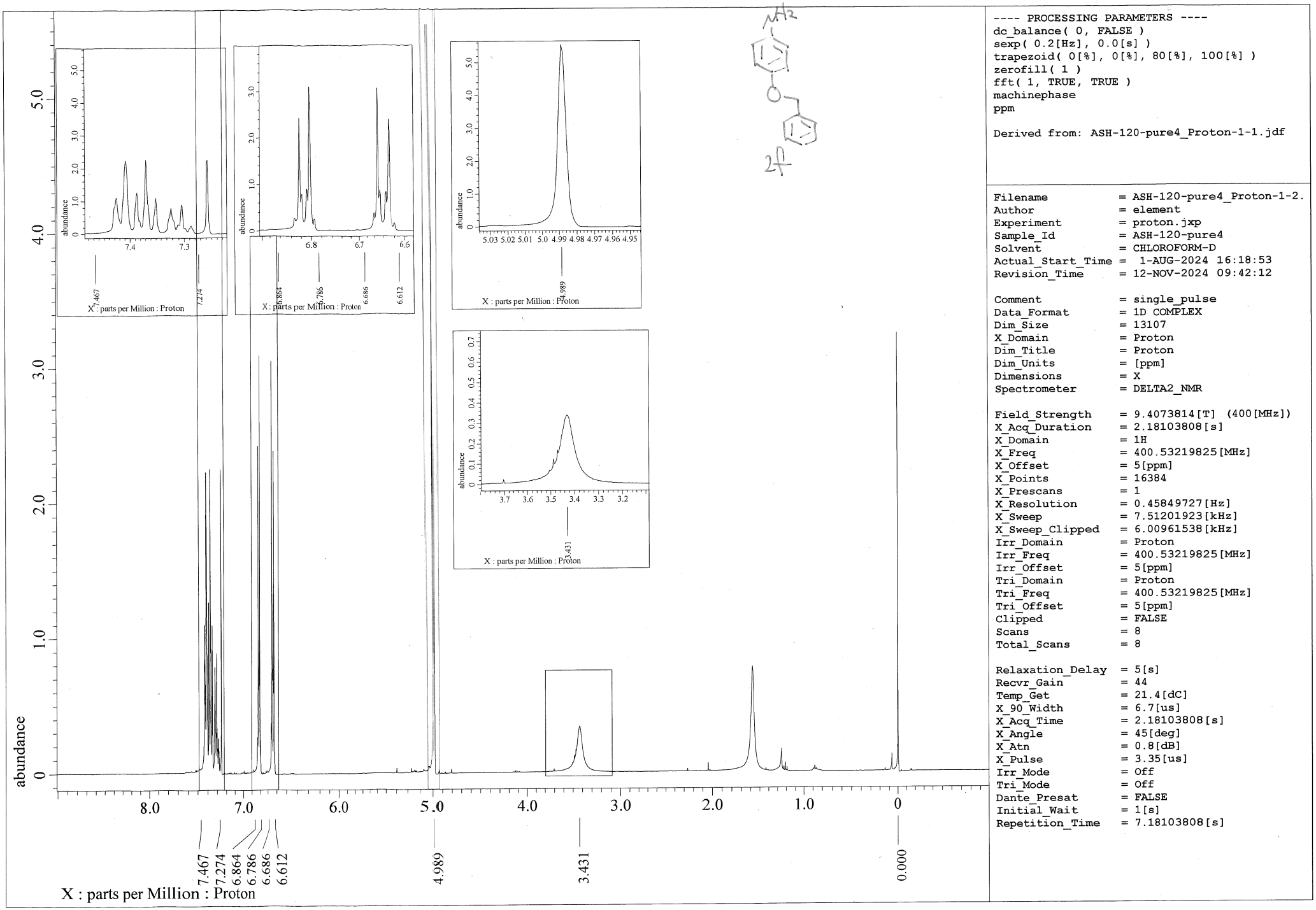

```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-110-pure2_Proton-1-1.jdf
```

Filename	= ASH-110-pure2_Proton-
Author	= element
Experiment	= proton_auto.jxp
Sample_Id	= ASH-110-pure2
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 23-JUL-2024 12:52:52
Revision_Time	= 12-NOV-2024 09:37:51
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	= 9.2982153[T] (400[MHz
X_Acq_Duration	= 2.20725248[s]
X_Domain	= Proton
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]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 46
Temp_Get	= 22.3[dC]
X_90_Width	= 6.34[us]
X_Acq_Time	= 2.20725248[s]
X_Angle	= 45[deg]
X_Atn	= 5[dB]
X_Pulse	= 3.17[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 500
Dante_Presat	= FALSE
Decimation_Rate	= 0
Experiment_Path	= C:\Program Files\JEOL
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180
Presat_Time	= 5[s]



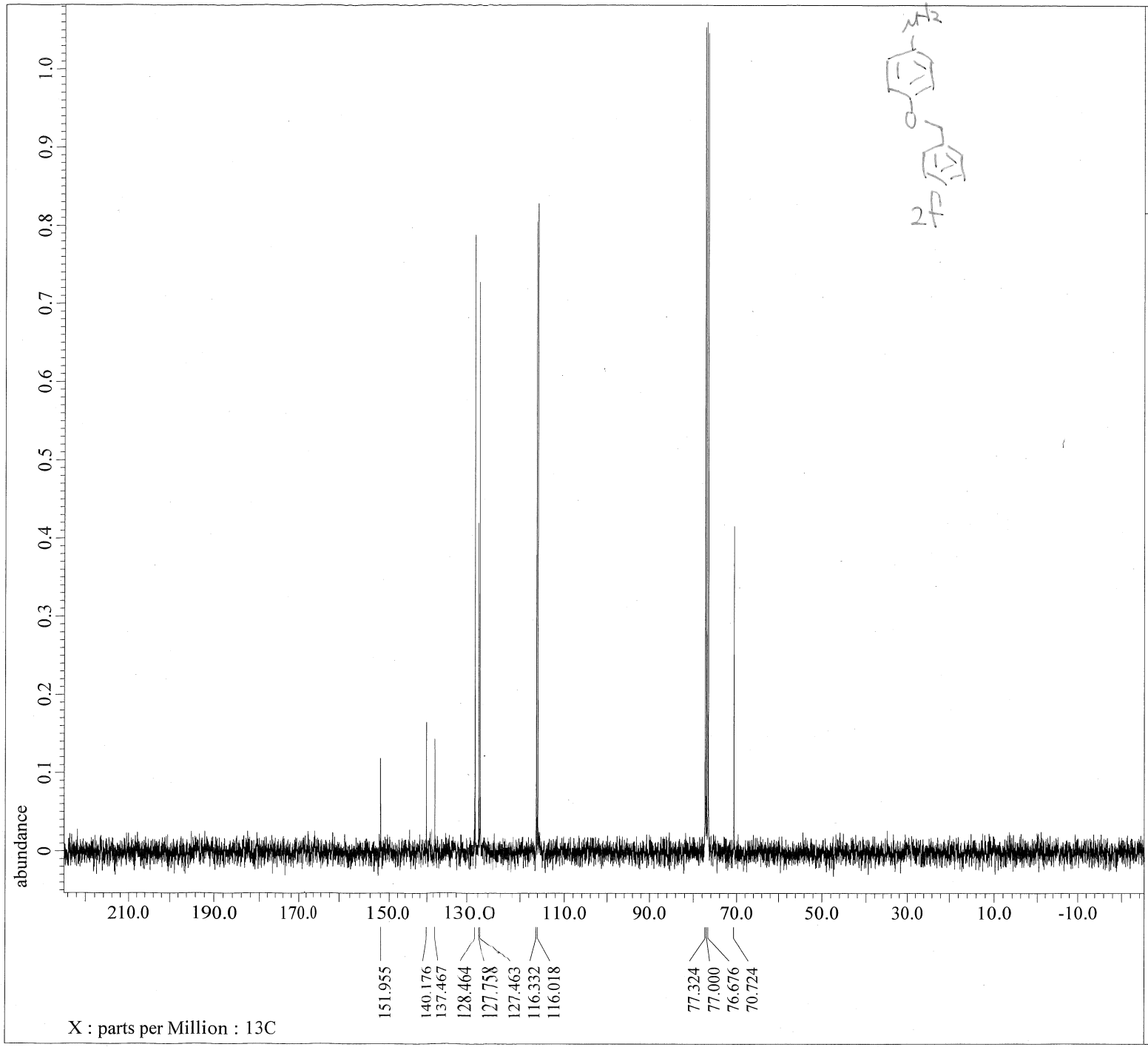

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-110-13C-1.jdf
```

```
Filename            = ASH-110-13C-2.jdf
Author              = element
Experiment          = single_pulse_dec
Sample_Id           = 1
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 29-JUL-2024 18:11:22
Revision_Time       = 29-JUL-2024 12:26:21

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               = 53
Total_Scans         = 53

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 22.1[dC]
X_90_Width          = 9.46[us]
X_Acq_Time          = 1.06430464[s]
X_Angle             = 30[deg]
X_Atn               = 4.9[dB]
X_Pulse             = 3.15333333[us]
Irr_Atn_Dec         = 22.45[dB]
Irr_Atn_Noe         = 22.45[dB]
Irr_Noise           = WALTZ
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.06430464[s]
```



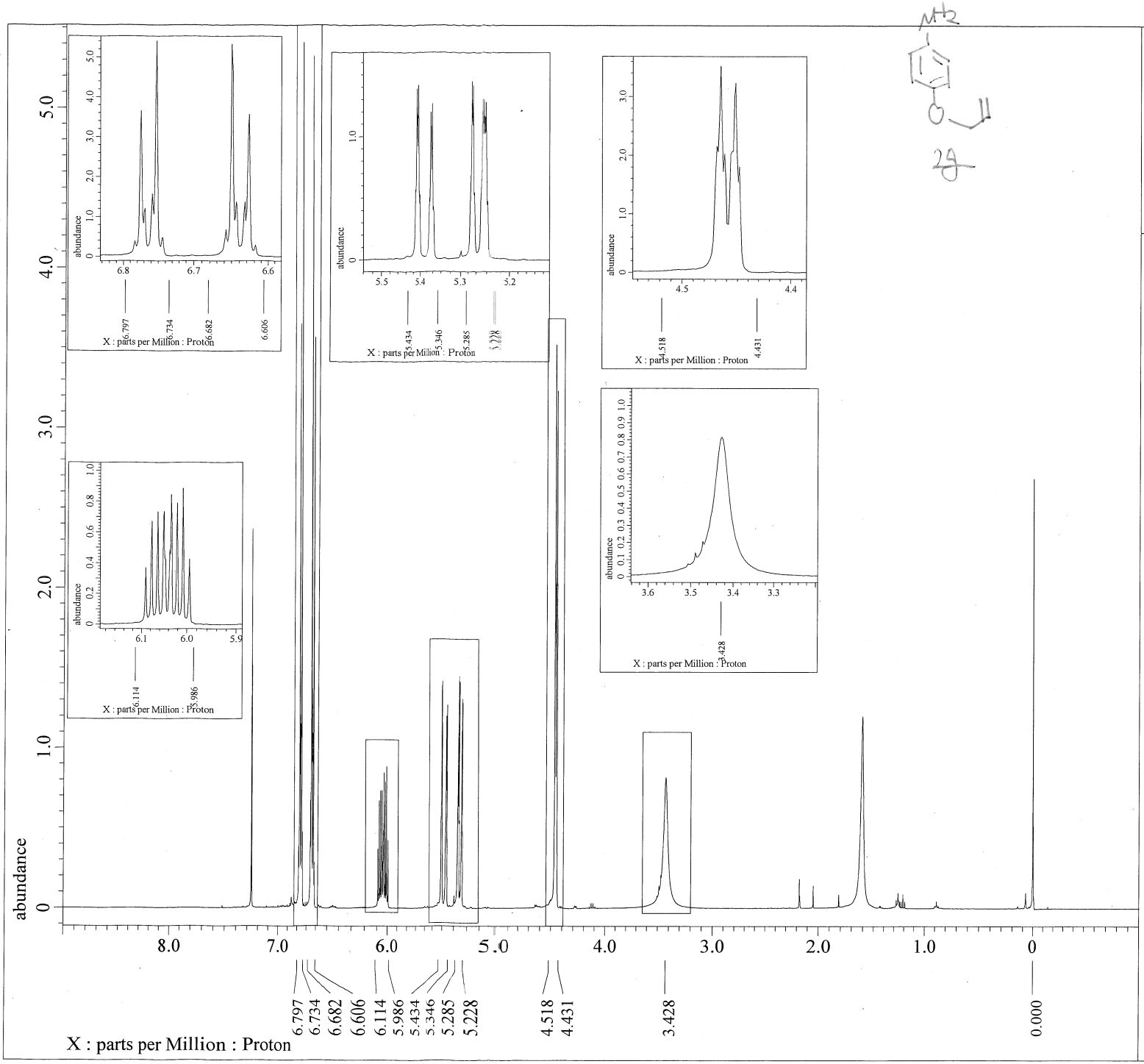

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-120-pure4_Proton-1-1.jdf
```

Filename	= ASH-120-pure4_Proton-1-2.
Author	= element
Experiment	= proton.jxp
Sample_Id	= ASH-120-pure4
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 1-AUG-2024 16:18:53
Revision_Time	= 12-NOV-2024 09:42:12
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	= 21.4[dC]
X_90_Width	= 6.7[us]
X_Acq_Time	= 2.18103808[s]
X_Angle	= 45[deg]
X_Atn	= 0.8[dB]
X_Pulse	= 3.35[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18103808[s]



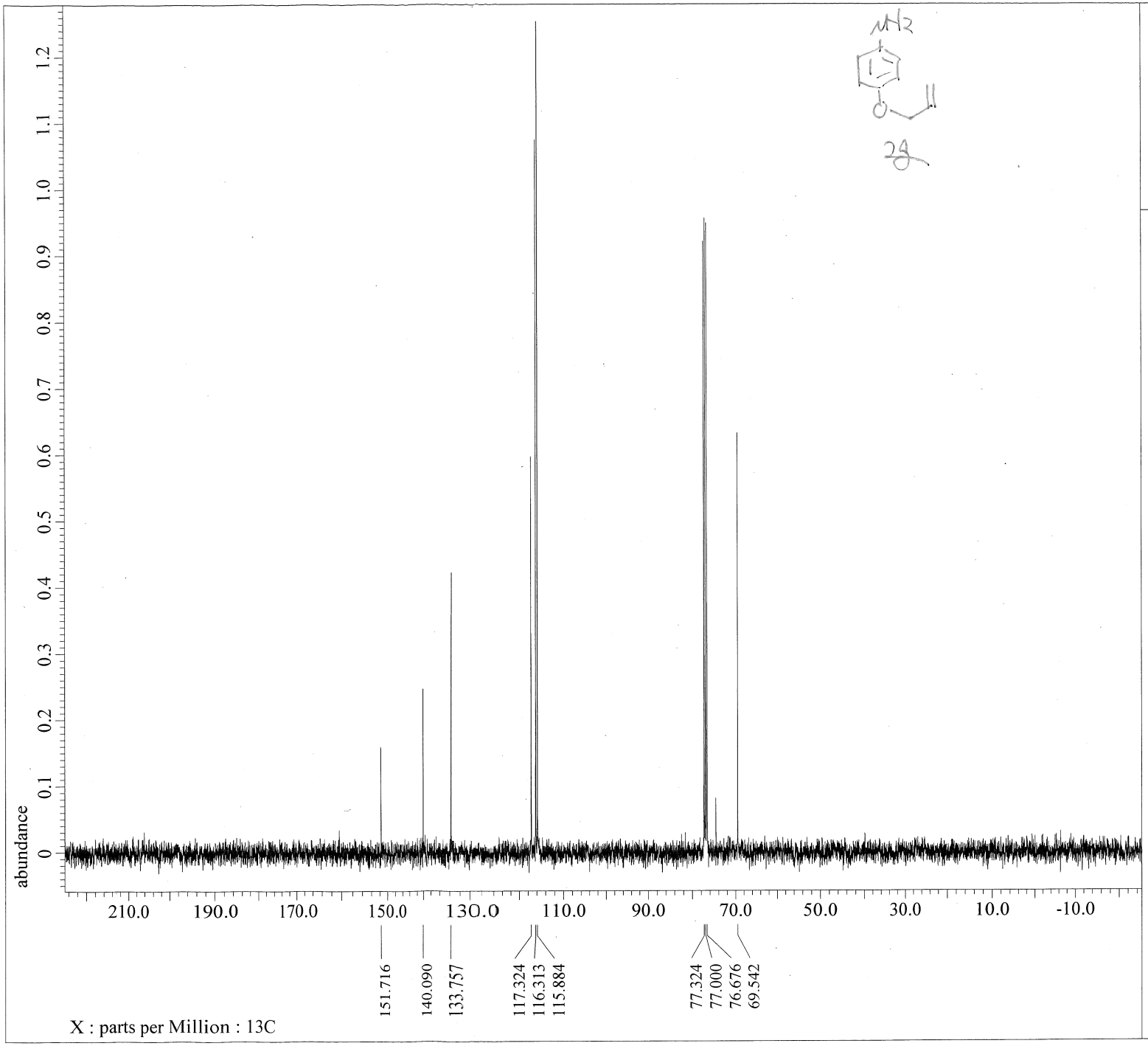

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-120-13C-1.jdf
```

```
Filename            = ASH-120-13C-2.jdf
Author              = element
Experiment          = single_pulse_dec
Sample_Id           = 1
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  1-AUG-2024 22:56:48
Revision_Time       =  1-AUG-2024 16:52:36

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               = 56
Total_Scans         = 56

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 22.5[dC]
X_90_Width          = 9.46[us]
X_Acq_Time          = 1.06430464[s]
X_Angle             = 30[deg]
X_Atn               = 4.9[dB]
X_Pulse             = 3.15333333[us]
Irr_Atn_Dec         = 22.45[dB]
Irr_Atn_Noe         = 22.45[dB]
Irr_Noise           = WALTZ
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.06430464[s]
```



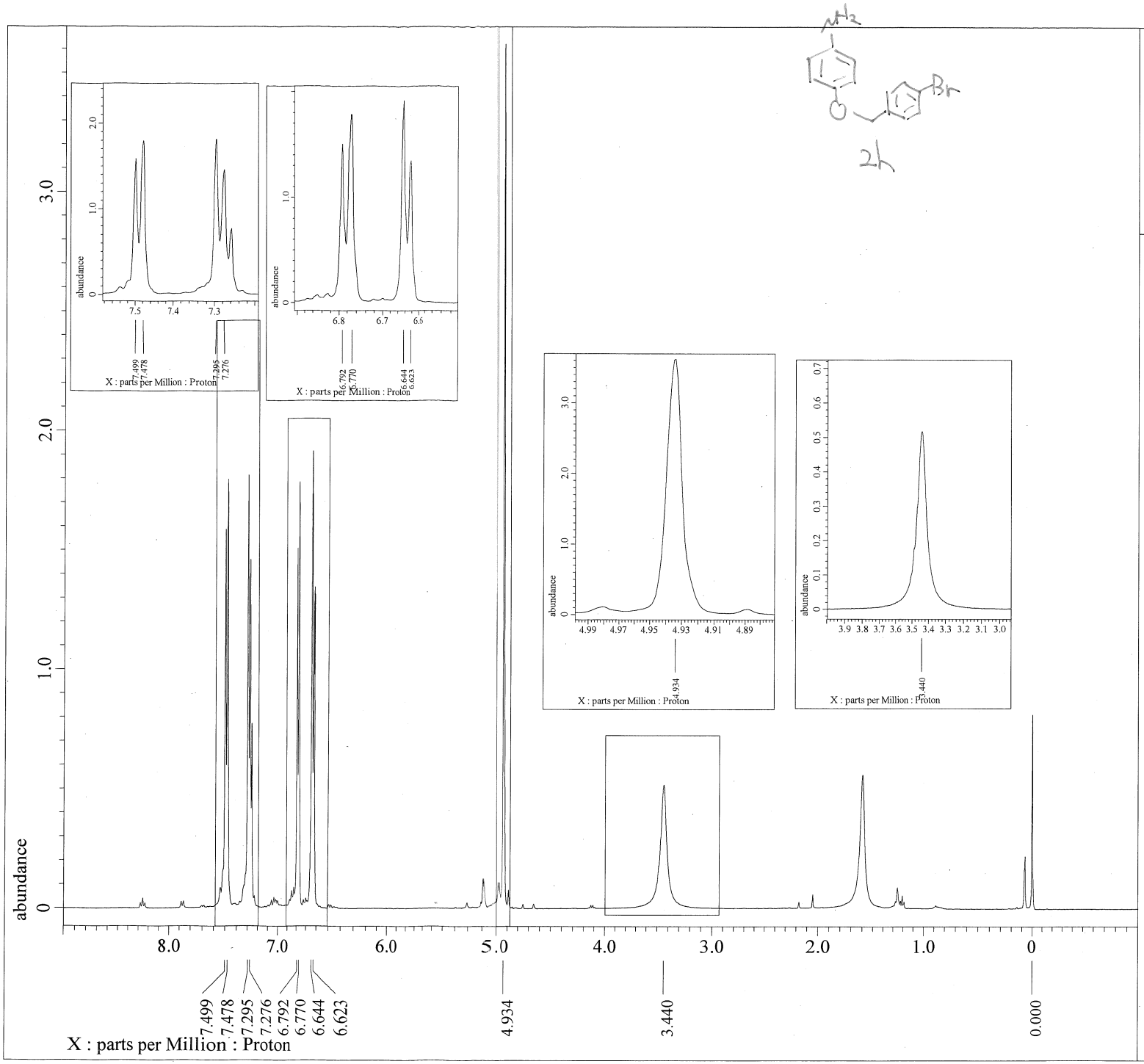

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-155-pure2_Proton-1-1.jdf
```

Filename	= ASH-155-pure2_Proton-1-2.
Author	= element
Experiment	= proton.jxp
Sample_Id	= ASH-155-pure2
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 21-AUG-2024 09:37:03
Revision_Time	= 12-NOV-2024 09:49:52
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	= 20.4[dC]
X_90_Width	= 6.7[us]
X_Acq_Time	= 2.18103808[s]
X_Angle	= 45[deg]
X_Atn	= 0.8[dB]
X_Pulse	= 3.35[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18103808[s]



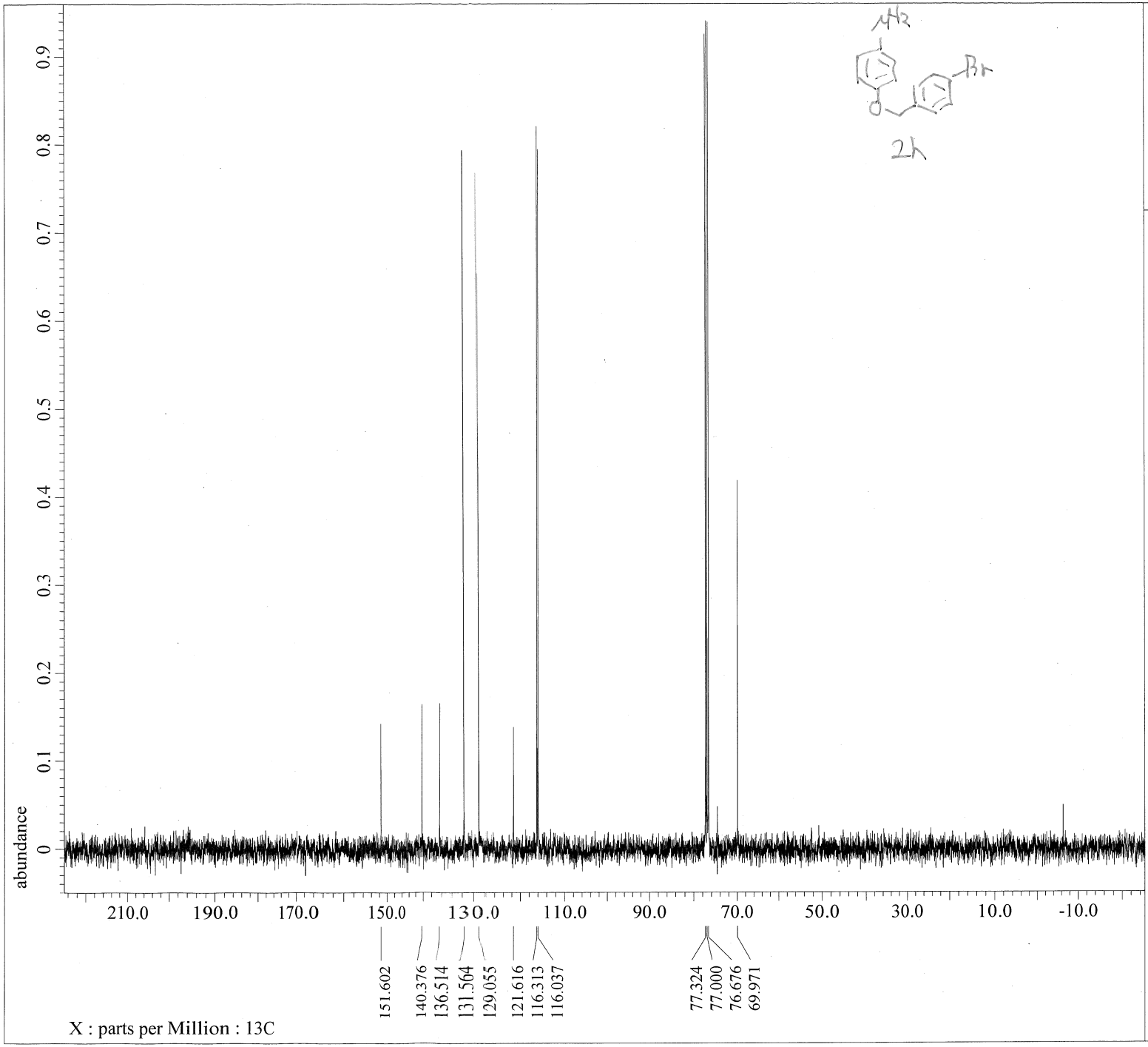

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-155-13C-1.jdf
```

```
Filename            = ASH-155-13C-2.jdf
Author              = element
Experiment          = single_pulse_dec
Sample_Id           = 1
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 21-AUG-2024 16:14:31
Revision_Time       = 21-AUG-2024 10:55:44

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               = 57
Total_Scans         = 57

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 21.8[dC]
X_90_Width          = 9.46[us]
X_Acq_Time          = 1.06430464[s]
X_Angle             = 30[deg]
X_Atn               = 4.9[dB]
X_Pulse             = 3.15333333[us]
Irr_Atn_Dec         = 22.45[dB]
Irr_Atn_Noe         = 22.45[dB]
Irr_Noise           = WALTZ
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.06430464[s]
```



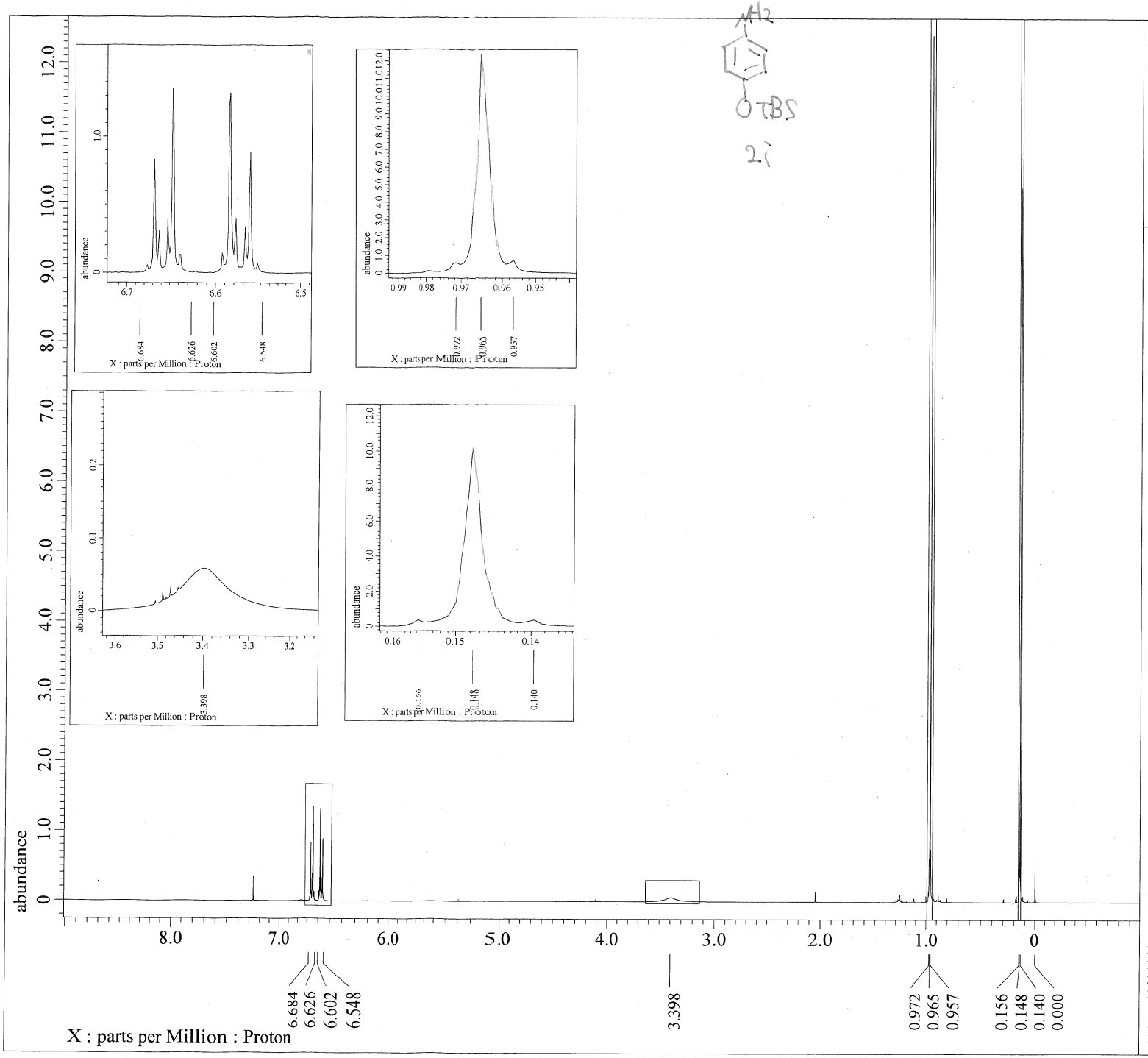

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-157-pure2_Proton-1-1.jdf
```

Filename	= ASH-157-pure2_Proton-1-2.
Author	= element
Experiment	= proton.jxp
Sample_Id	= ASH-157-pure2
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 22-AUG-2024 11:08:32
Revision_Time	= 12-NOV-2024 09:55:02
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	= 42
Temp_Get	= 20.8[dC]
X_90_Width	= 6.7[us]
X_Acq_Time	= 2.18103808[s]
X_Angle	= 45[deg]
X_Atn	= 0.8[dB]
X_Pulse	= 3.35[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18103808[s]



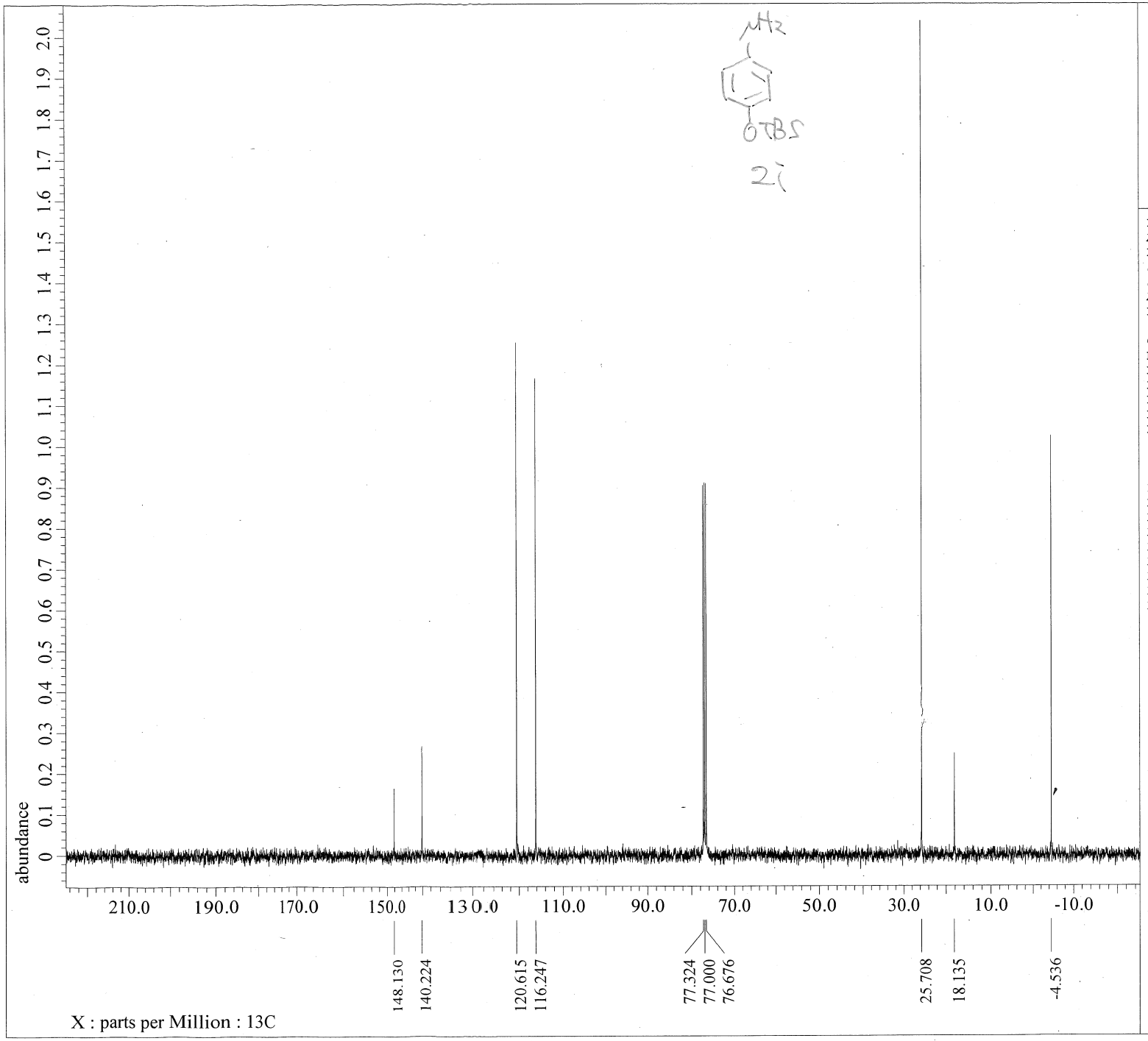

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-157-13C-1.jdf
```

Filename	= ASH-157-13C-2.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_Id	= 1
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 22-AUG-2024 17:02:47
Revision_Time	= 22-AUG-2024 11:04:17
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	= 72
Total_Scans	= 72
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 21.9[dC]
X_90_Width	= 9.46[us]
X_Acq_Time	= 1.06430464[s]
X_Angle	= 30[deg]
X_Atn	= 4.9[dB]
X_Pulse	= 3.15333333[us]
Irr_Atn_Dec	= 22.45[dB]
Irr_Atn_Noe	= 22.45[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.06430464[s]



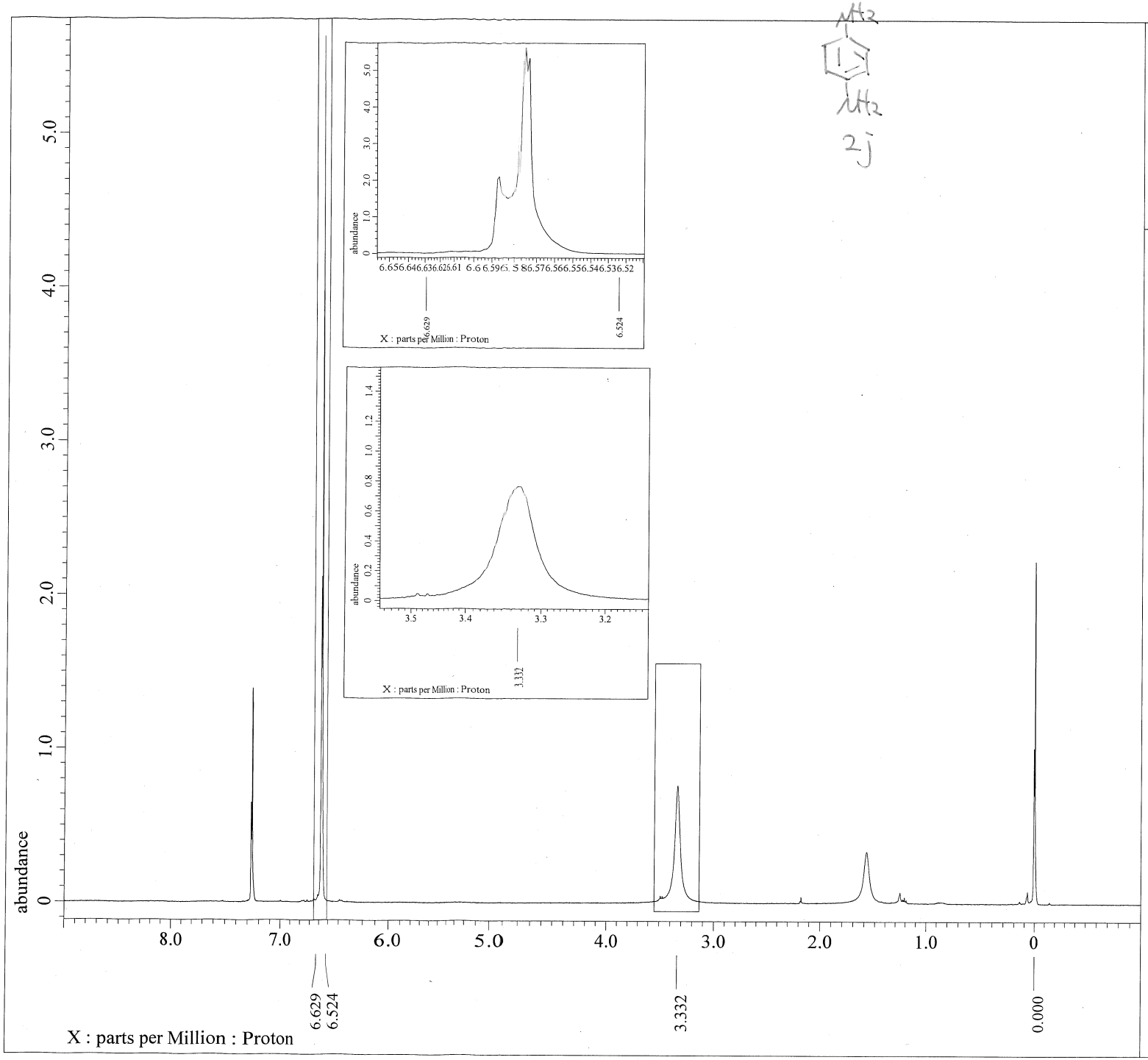

```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-124-pure 1_Proton-1-1.jdf
```

Filename	= ASH-124-pure 1_Proton
Author	= element
Experiment	= proton_auto.jxp
Sample_Id	= ASH-124-pure 1
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 4-AUG-2024 20:17:49
Revision_Time	= 12-NOV-2024 10:02:06
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	= 9.2982153[T] (400[MHz
X_Acq_Duration	= 2.20725248[s]
X_Domain	= Proton
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]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 46
Temp_Get	= 20.7[dC]
X_90_Width	= 6.34[us]
X_Acq_Time	= 2.20725248[s]
X_Angle	= 45[deg]
X_Atn	= 5[dB]
X_Pulse	= 3.17[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 500
Dante_Presat	= FALSE
Decimation_Rate	= 0
Experiment_Path	= C:\Program Files\JEOL
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180
Presat_Time	= 5[s]



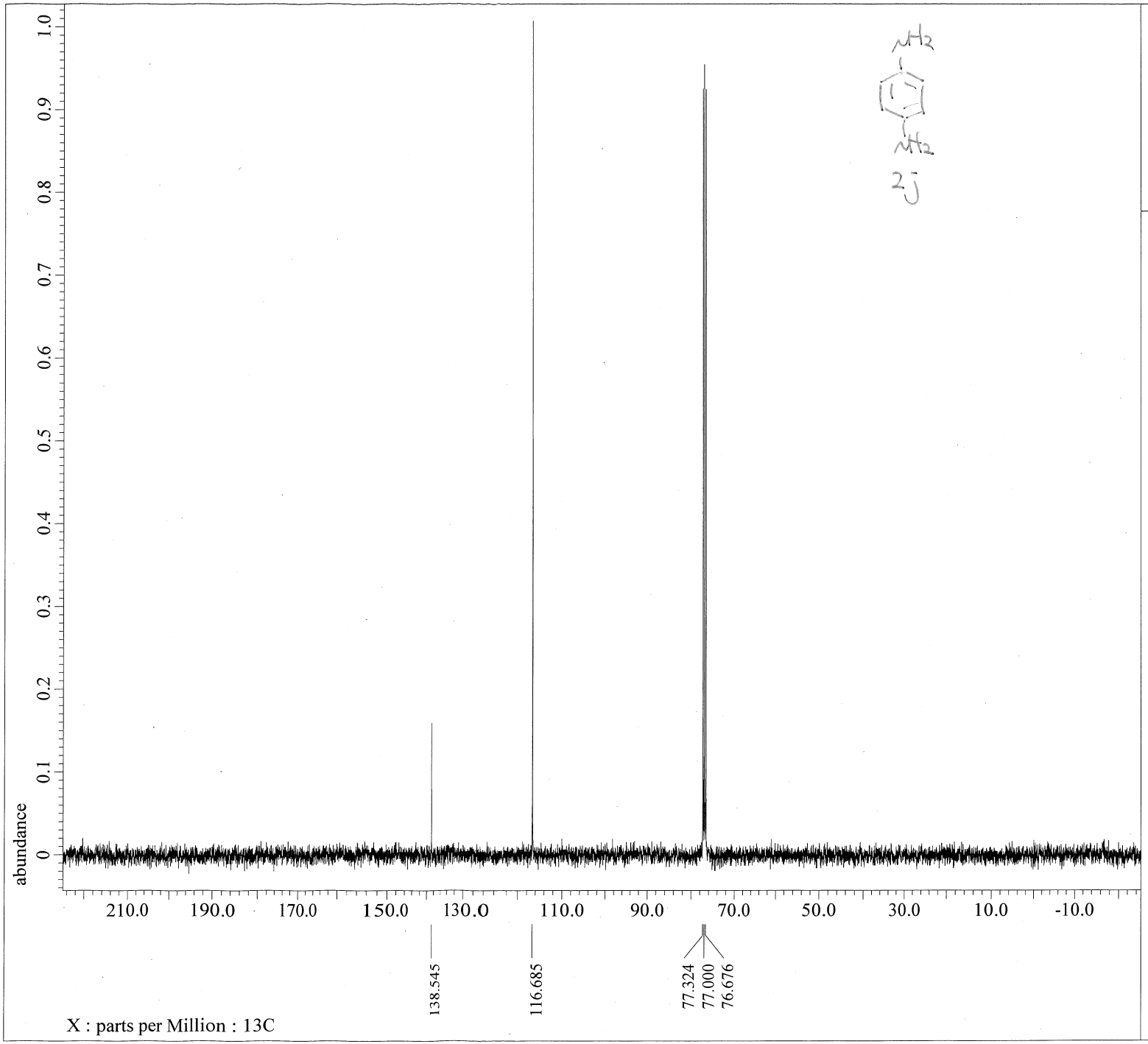

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-124-13C-1.jdf
```

```
Filename            = ASH-124-13C-2.jdf
Author              = element
Experiment          = single_pulse_dec
Sample_Id           = 1
Solvent             = CHLOROFORM-D
Actual_Start_Time   =  5-AUG-2024 16:04:15
Revision_Time       =  5-AUG-2024 10:21:37

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               = 62
Total_Scans         = 62

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 20.5[dC]
X_90_Width          = 9.46[us]
X_Acq_Time          = 1.06430464[s]
X_Angle             = 30[deg]
X_Atn               = 4.9[dB]
X_Pulse             = 3.15333333[us]
Irr_Atn_Dec         = 22.45[dB]
Irr_Atn_Noe         = 22.45[dB]
Irr_Noise           = WALTZ
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.06430464[s]
```



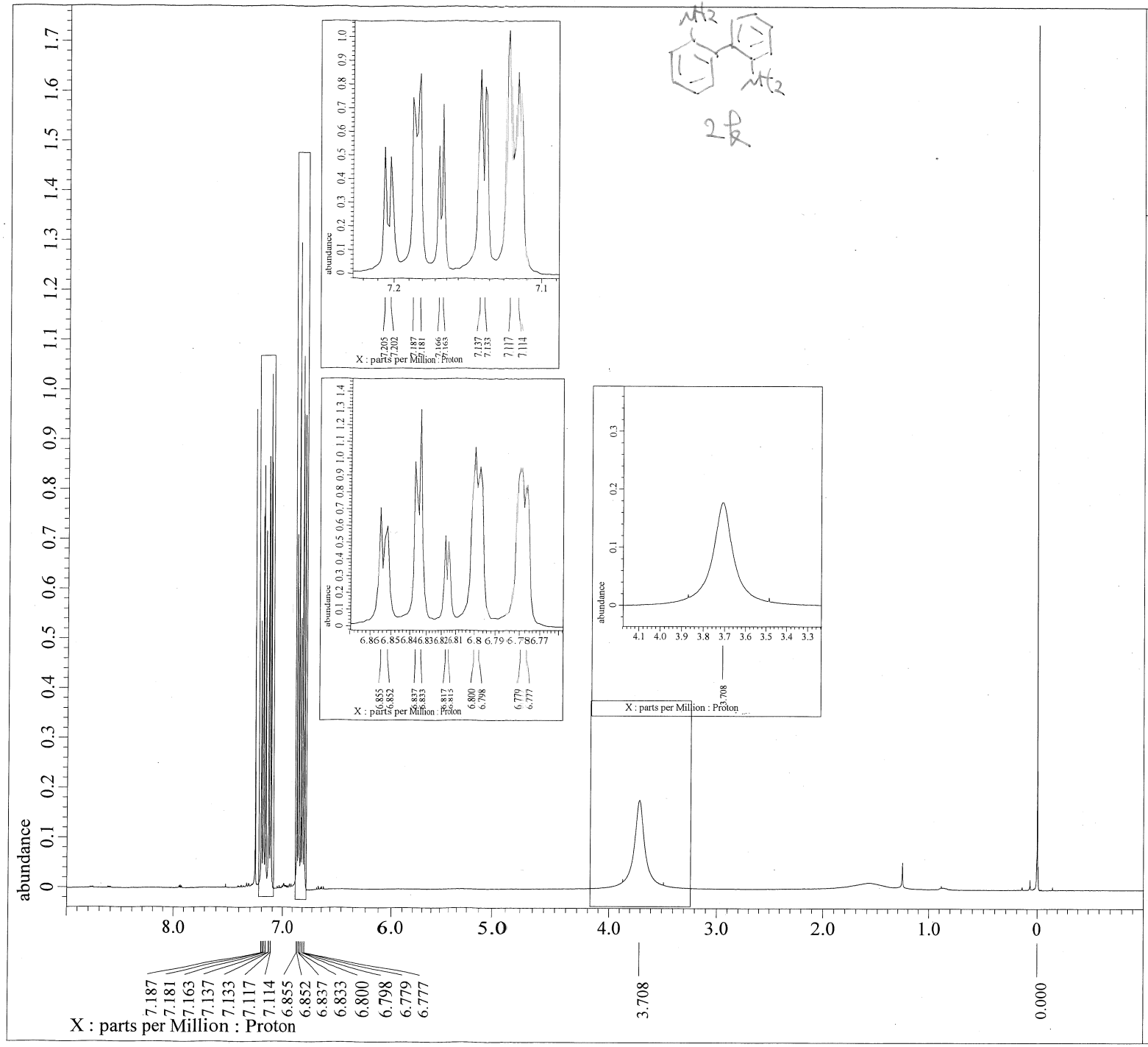

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-115-pure2_Proton-1-1.jdf
```

Filename	= ASH-115-pure2_Proton-1-2.
Author	= element
Experiment	= proton.jxp
Sample_Id	= ASH-115-pure2
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 26-JUL-2024 10:00:11
Revision_Time	= 12-NOV-2024 10:05:45
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	= 48
Temp_Get	= 20.4[dC]
X_90_Width	= 6.7[us]
X_Acq_Time	= 2.18103808[s]
X_Angle	= 45[deg]
X_Atn	= 0.8[dB]
X_Pulse	= 3.35[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18103808[s]



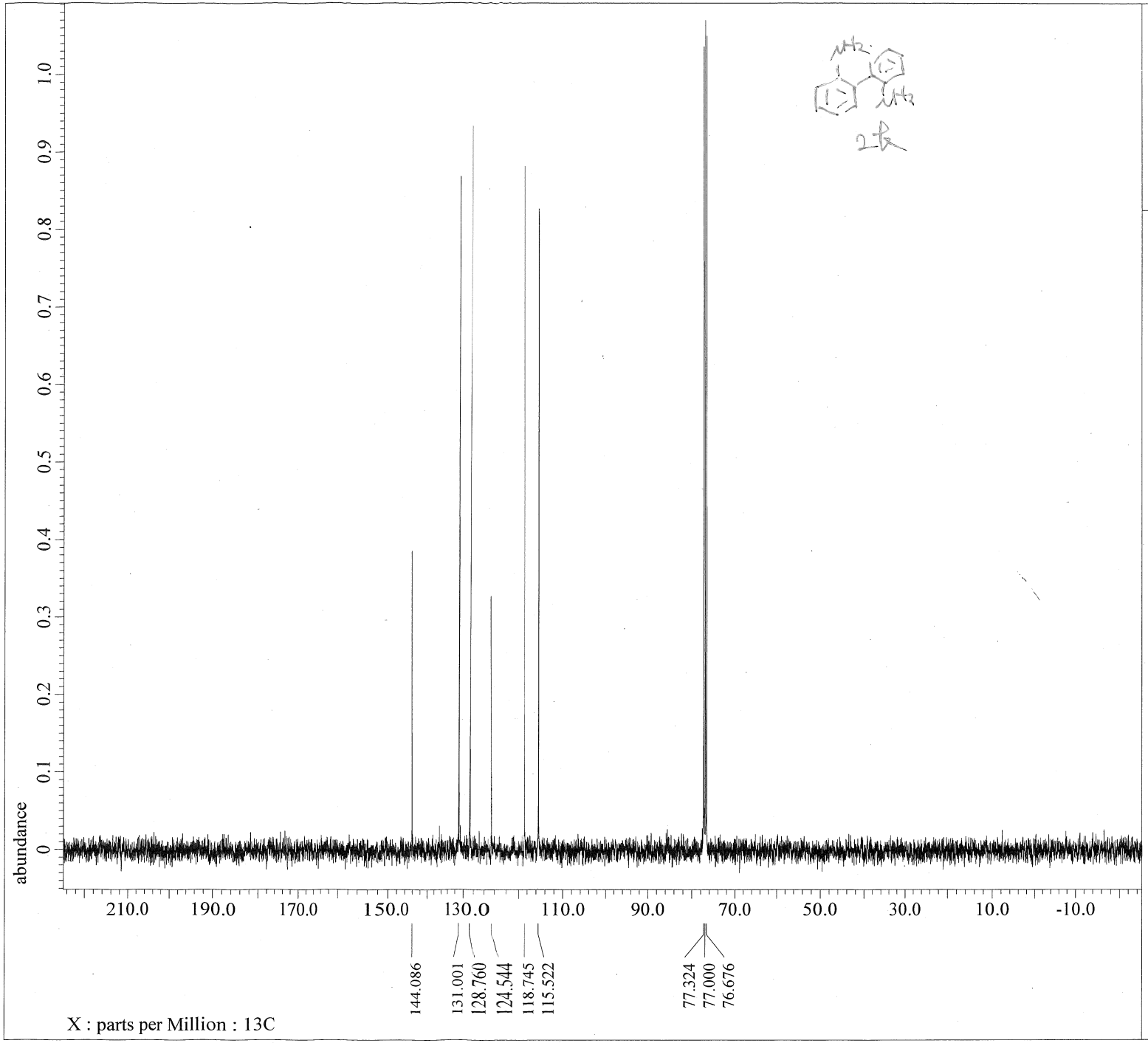

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-115-13C-2-1.jdf
```

Filename	= ASH-115-13C-2-2.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_Id	= 1
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 26-JUL-2024 16:25:23
Revision_Time	= 12-NOV-2024 11:09:52
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	= 133
Total_Scans	= 133
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 22.7[dC]
X_90_Width	= 9.46[us]
X_Acq_Time	= 1.06430464[s]
X_Angle	= 30[deg]
X_Atn	= 4.9[dB]
X_Pulse	= 3.15333333[us]
Irr_Atn_Dec	= 22.45[dB]
Irr_Atn_Noe	= 22.45[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.06430464[s]



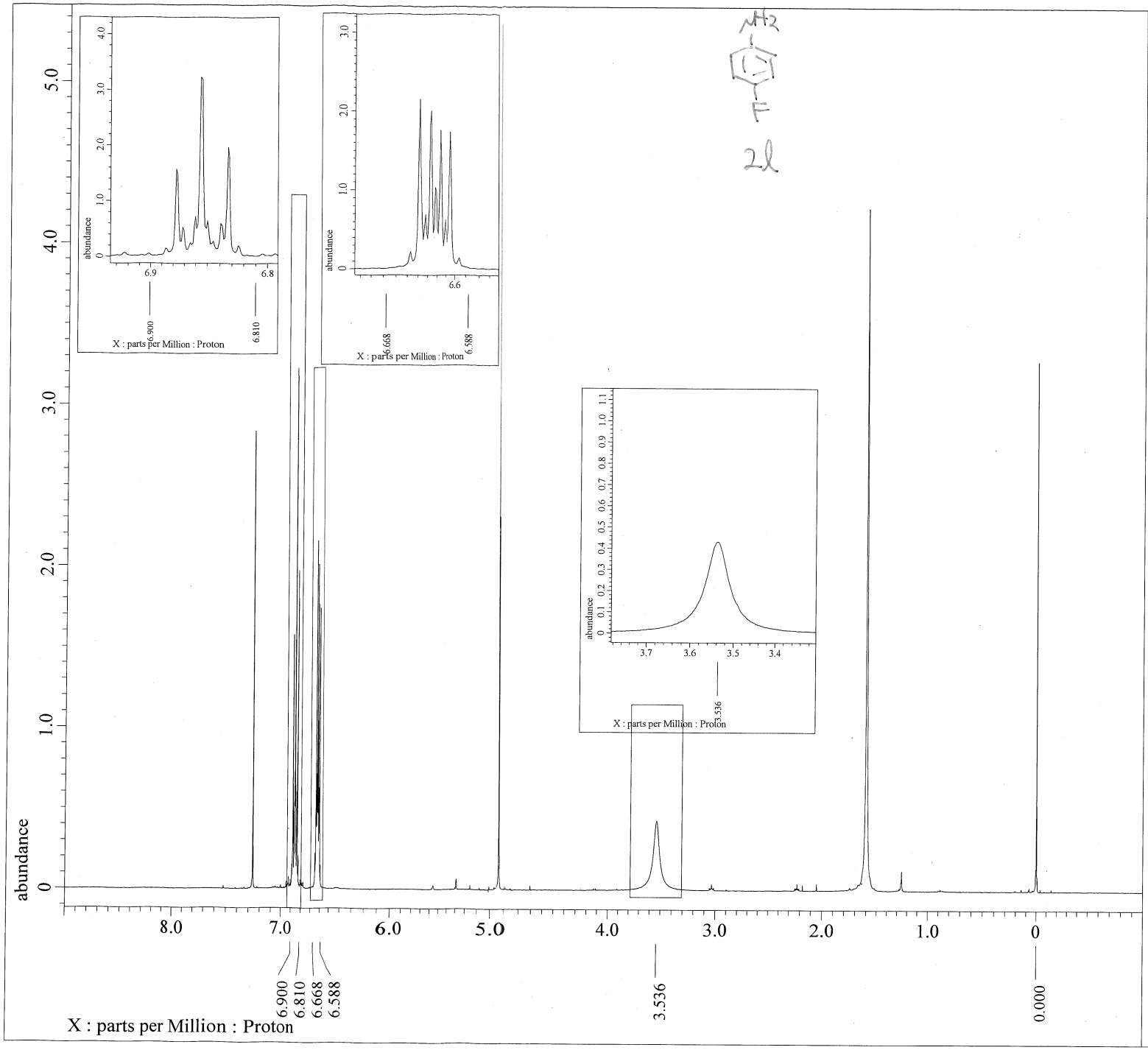

```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-101-pure2_Proton-1-1.jdf
```

Filename	= ASH-101-pure2_Proton-
Author	= element
Experiment	= proton_auto.jxp
Sample_Id	= ASH-101-pure2
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 27-JUL-2024 14:44:32
Revision_Time	= 12-NOV-2024 10:09:08
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	= 9.2982153[T] (400[MHz
X_Acq_Duration	= 2.20725248[s]
X_Domain	= Proton
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]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 56
Temp_Get	= 21.5[dC]
X_90_Width	= 6.34[us]
X_Acq_Time	= 2.20725248[s]
X_Angle	= 45[deg]
X_Atn	= 5[dB]
X_Pulse	= 3.17[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 500
Dante_Presat	= FALSE
Decimation_Rate	= 0
Experiment_Path	= C:\Program Files\JEOL
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180
Presat_Time	= 5[s]



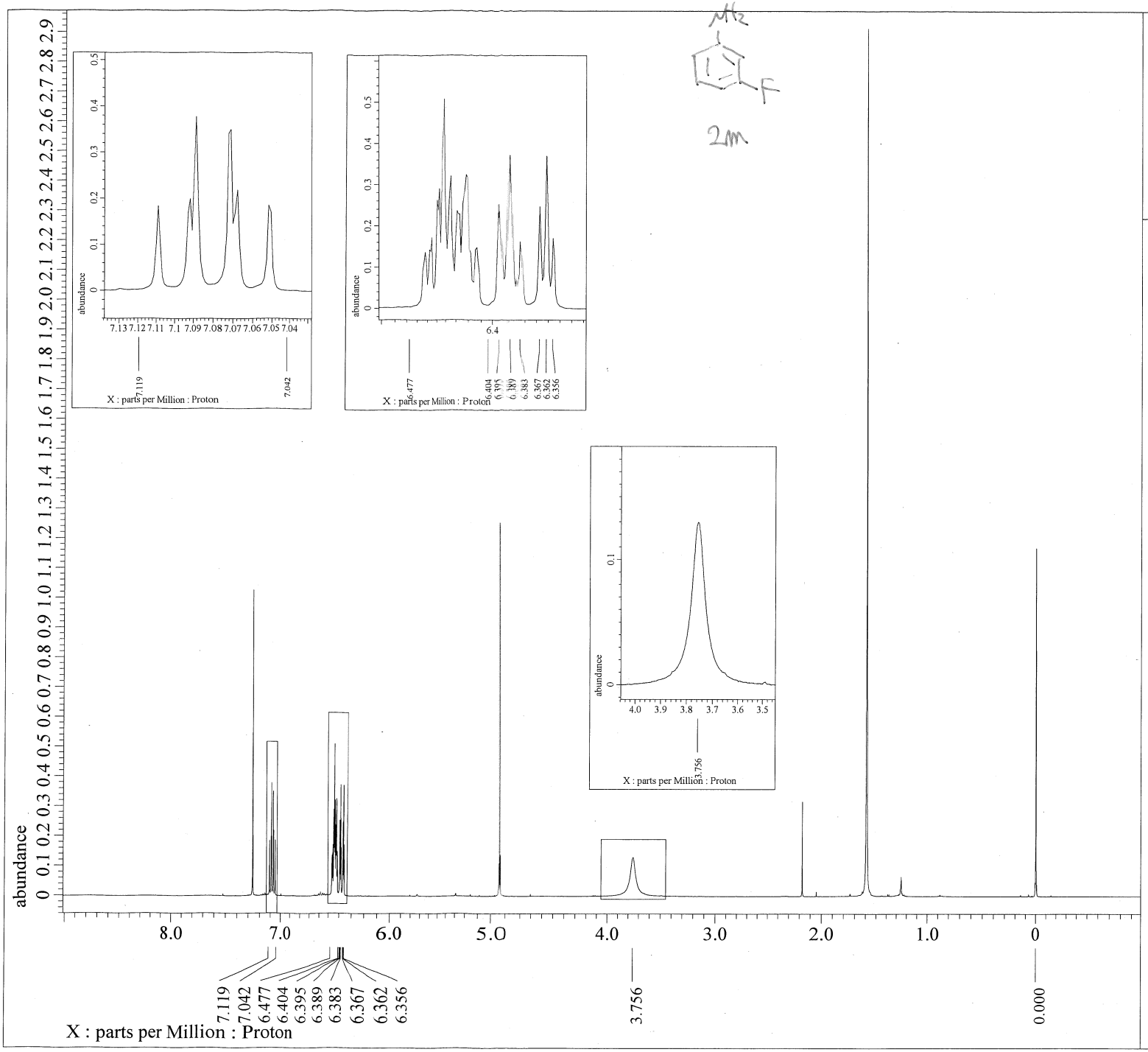

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-101-13C-1.jdf
```

```
Filename            = ASH-101-13C-2.jdf
Author              = element
Experiment          = single_pulse_dec
Sample_Id           = 1
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 27-JUL-2024 23:15:02
Revision_Time       = 27-JUL-2024 17:17:34

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               = 75
Total_Scans         = 75

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 22.7[dC]
X_90_Width          = 9.46[us]
X_Acq_Time          = 1.06430464[s]
X_Angle             = 30[deg]
X_Atn               = 4.9[dB]
X_Pulse             = 3.15333333[us]
Irr_Atn_Dec         = 22.45[dB]
Irr_Atn_Noe         = 22.45[dB]
Irr_Noise           = WALTZ
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.06430464[s]
```



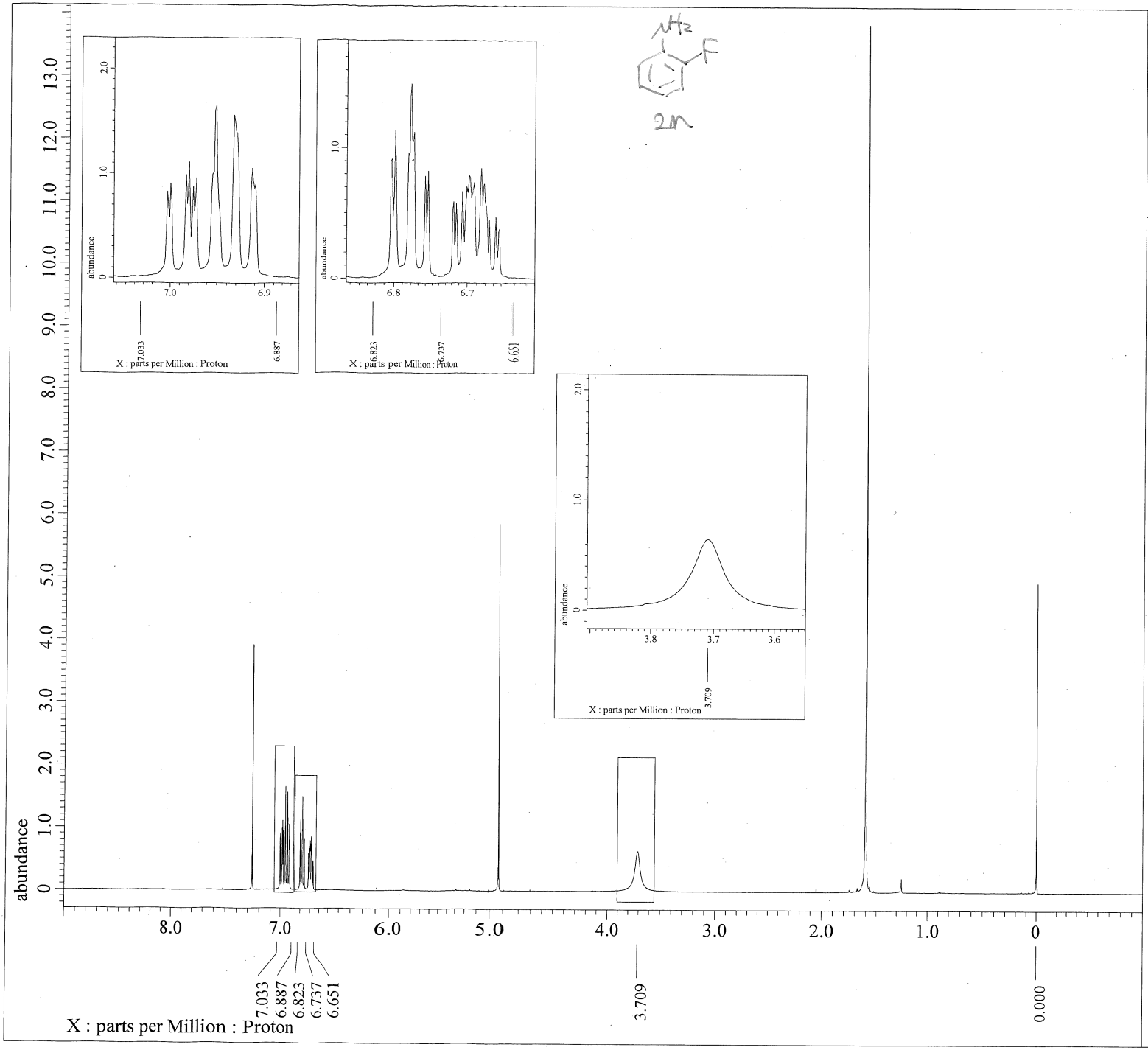

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-109-crude_Proton-1-1.jdf
```

Filename	= ASH-109-crude_Proton-1-2.
Author	= element
Experiment	= proton.jxp
Sample_Id	= ASH-109-crude
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 22-JUL-2024 11:09:06
Revision_Time	= 12-NOV-2024 10:12:48
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	= 21.7[dC]
X_90_Width	= 6.7[us]
X_Acq_Time	= 2.18103808[s]
X_Angle	= 45[deg]
X_Atn	= 0.8[dB]
X_Pulse	= 3.35[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18103808[s]



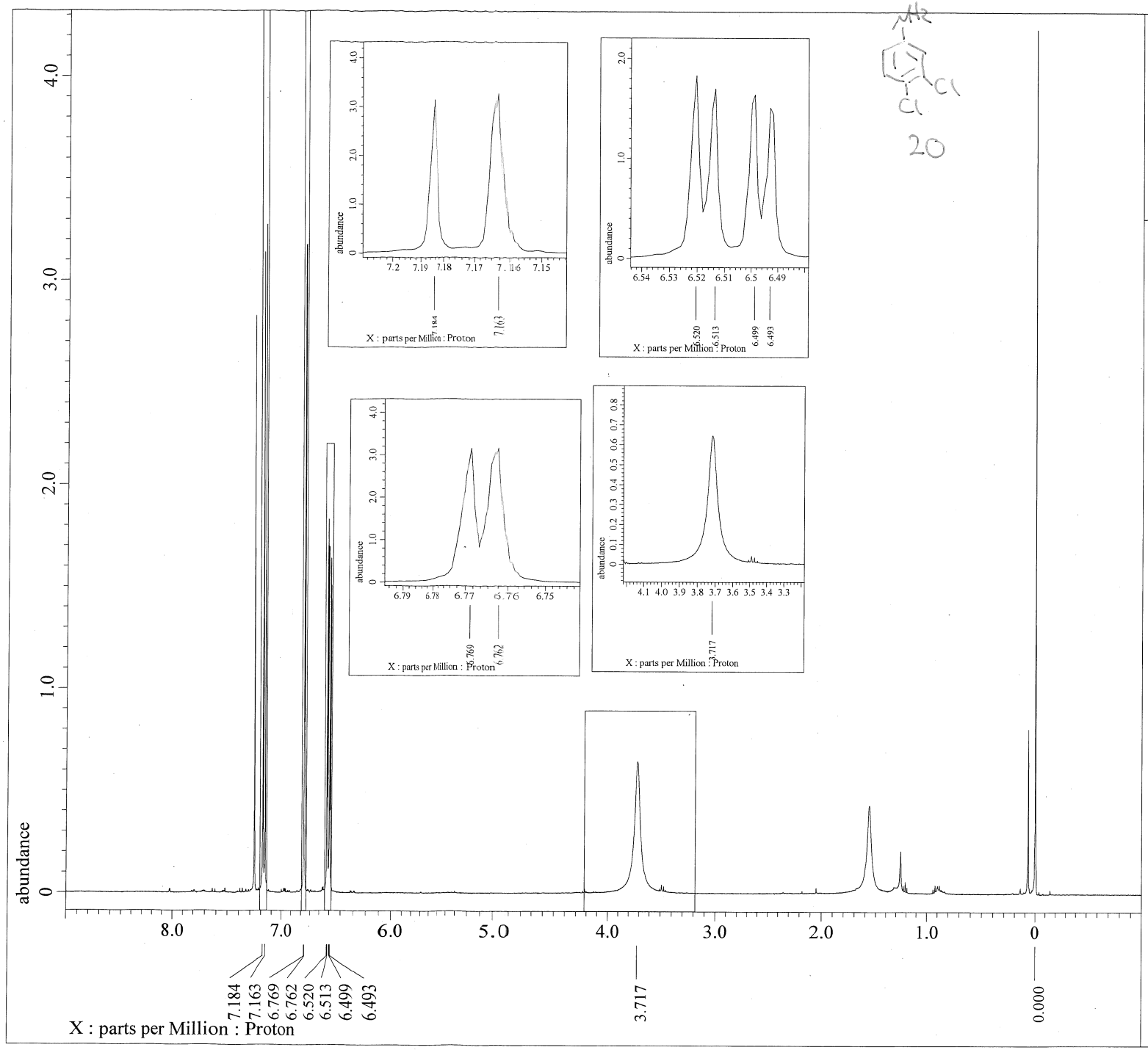

```
---- PROCESSING PARAMETERS ----
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-107-crude_Proton-1-1.jdf
```

Filename	= ASH-107-crude_Proton-
Author	= element
Experiment	= proton_auto.jxp
Sample_Id	= ASH-107-crude
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 20-JUL-2024 19:59:00
Revision_Time	= 12-NOV-2024 10:17:19
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	= 9.2982153[T] (400[MHz
X_Acq_Duration	= 2.20725248[s]
X_Domain	= Proton
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]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 56
Temp_Get	= 21.5[dC]
X_90_Width	= 6.34[us]
X_Acq_Time	= 2.20725248[s]
X_Angle	= 45[deg]
X_Atn	= 5[dB]
X_Pulse	= 3.17[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 500
Dante_Presat	= FALSE
Decimation_Rate	= 0
Experiment_Path	= C:\Program Files\JEOL
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180
Presat_Time	= 5[s]



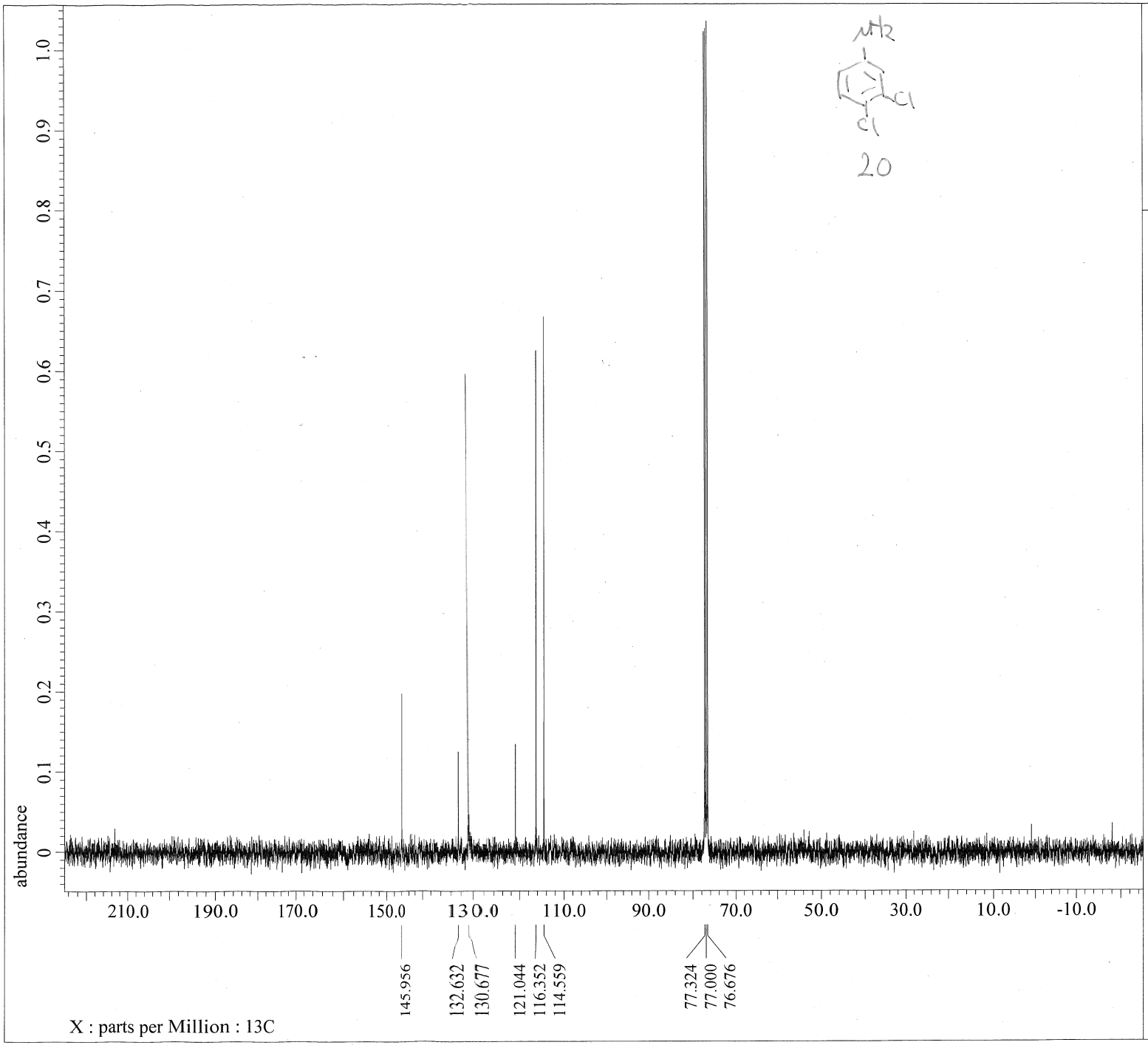

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-106-crude_Proton-1-1.jdf
```

Filename	= ASH-106-crude_Proton-1-2.
Author	= element
Experiment	= proton.jxp
Sample_Id	= ASH-106-crude
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 20-JUL-2024 17:54:06
Revision_Time	= 12-NOV-2024 10:21:25
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	= 46
Temp_Get	= 21.4[dC]
X_90_Width	= 6.7[us]
X_Acq_Time	= 2.18103808[s]
X_Angle	= 45[deg]
X_Atn	= 0.8[dB]
X_Pulse	= 3.35[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18103808[s]



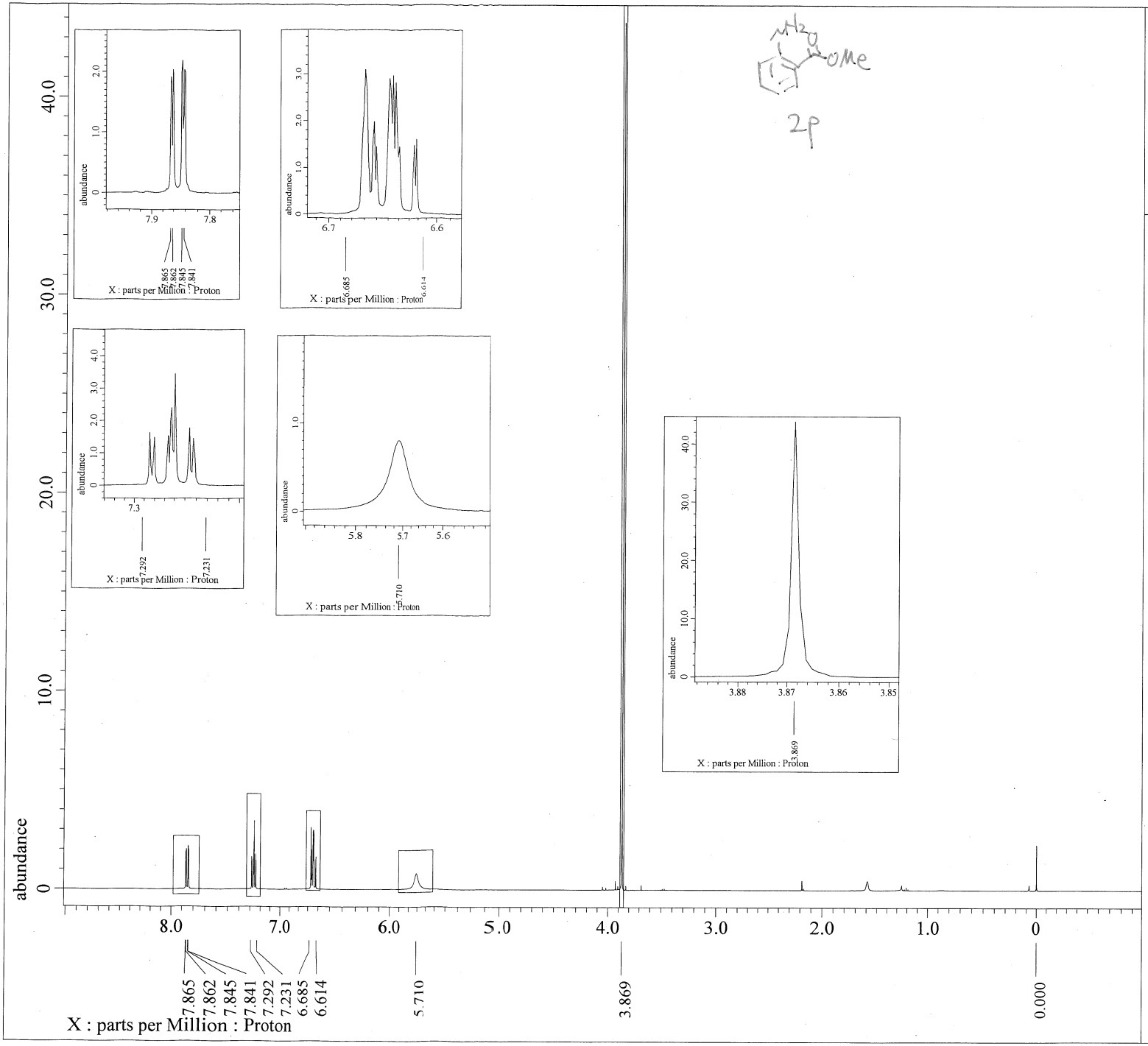

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-097-pure4_Proton-1-1.jdf
```

Filename	= ASH-097-pure4_Proton-1-2.
Author	= element
Experiment	= proton.jxp
Sample_Id	= ASH-097-pure3
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 27-JUL-2024 10:29:31
Revision_Time	= 12-NOV-2024 10:25:03
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	= 46
Temp_Get	= 20.6[dC]
X_90_Width	= 6.7[us]
X_Acq_Time	= 2.18103808[s]
X_Angle	= 45[deg]
X_Atn	= 0.8[dB]
X_Pulse	= 3.35[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18103808[s]



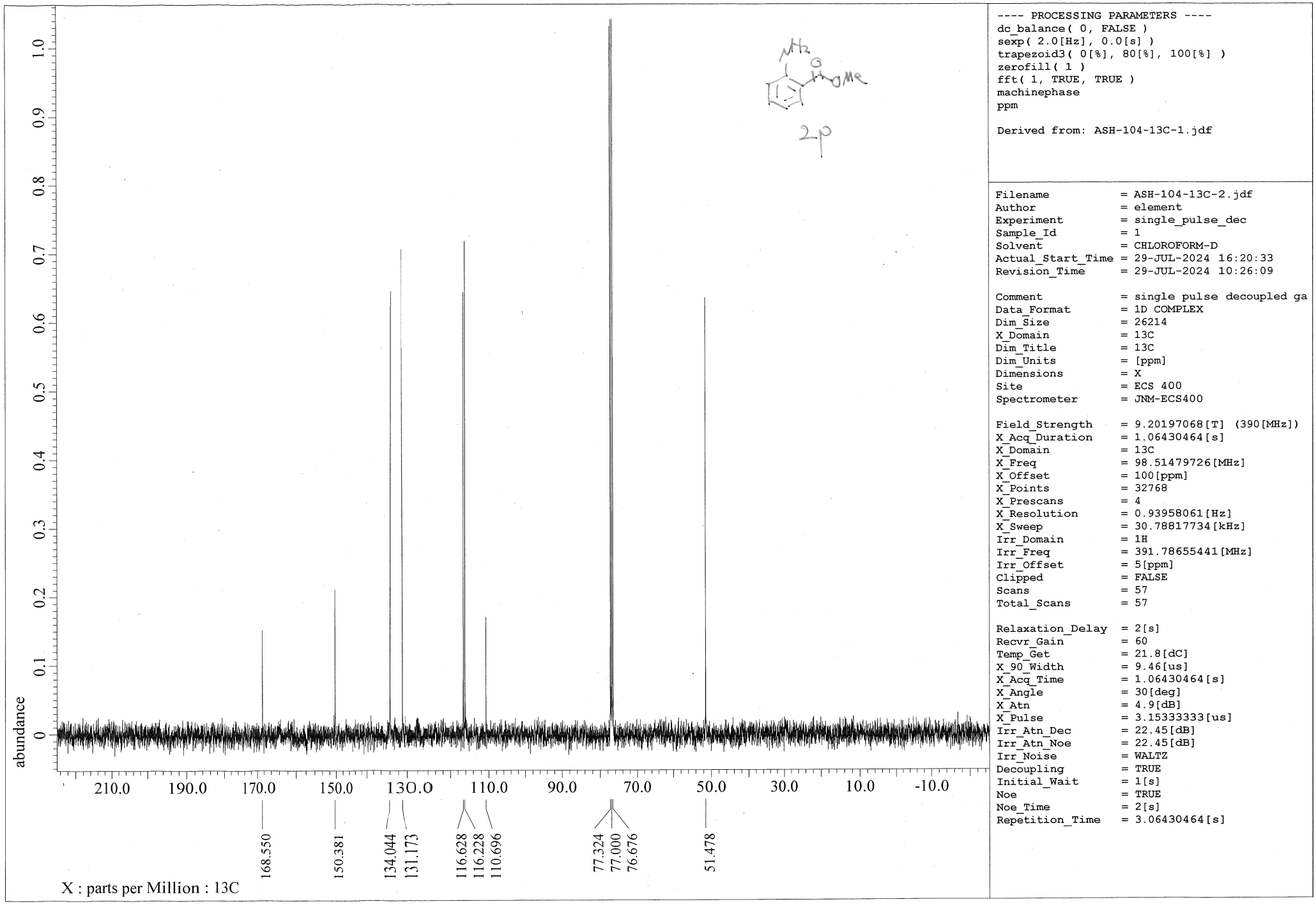

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-097-13C-1.jdf
```

Filename	= ASH-097-13C-2.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_Id	= S#451021
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 27-JUL-2024 18:48:15
Revision_Time	= 27-JUL-2024 13:59:19
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	= 73
Total_Scans	= 73
Relaxation_Delay	= 2[s]
Recvr_Gain	= 60
Temp_Get	= 22.8[dC]
X_90_Width	= 9.46[us]
X_Acq_Time	= 1.06430464[s]
X_Angle	= 30[deg]
X_Atn	= 4.9[dB]
X_Pulse	= 3.15333333[us]
Irr_Atn_Dec	= 22.45[dB]
Irr_Atn_Noe	= 22.45[dB]
Irr_Noise	= WALTZ
Decoupling	= TRUE
Initial_Wait	= 1[s]
Noe	= TRUE
Noe_Time	= 2[s]
Repetition_Time	= 3.06430464[s]



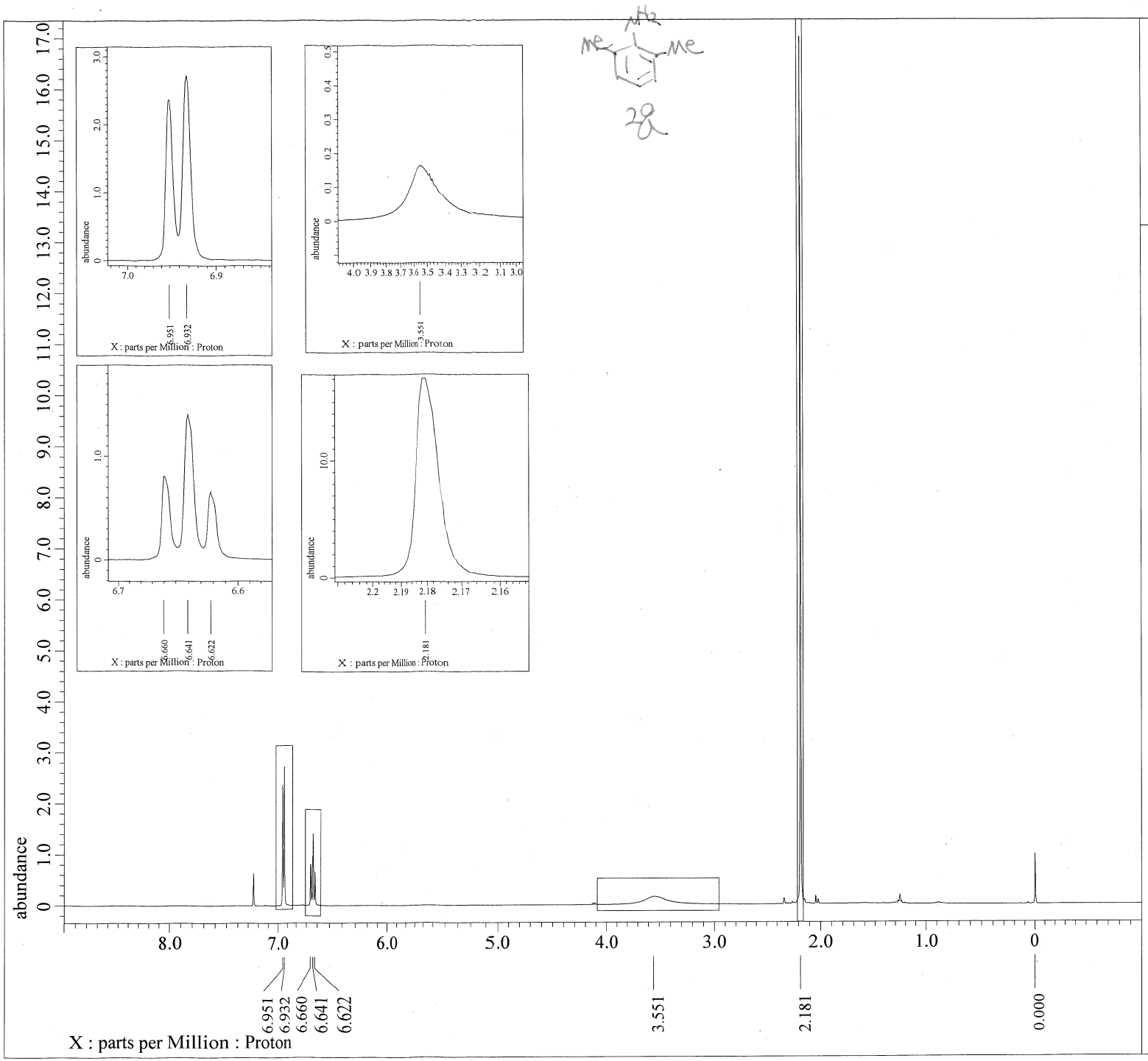

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-104-purelast_Proton-1-1.jdf
```

Filename	= ASH-104-purelast_Proton-1
Author	= element
Experiment	= proton.jxp
Sample_Id	= ASH-104-purelast
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 27-JUL-2024 19:07:25
Revision_Time	= 12-NOV-2024 10:29:17
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	= 36
Temp_Get	= 22.1[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
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.1889024[s]



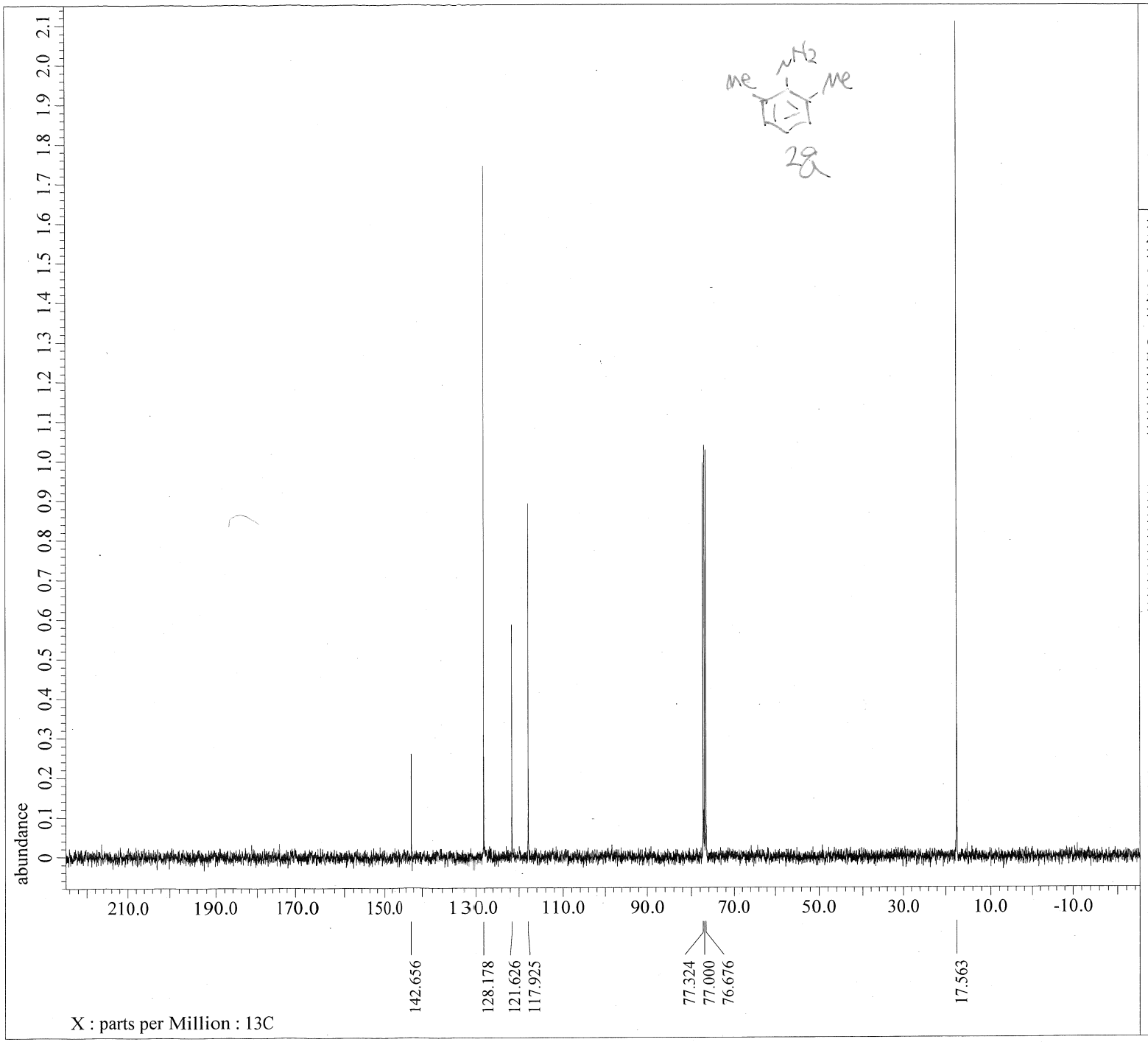

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-104-13C-1.jdf
```

```
Filename            = ASH-104-13C-2.jdf
Author              = element
Experiment          = single_pulse_dec
Sample_Id           = 1
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 29-JUL-2024 16:20:33
Revision_Time       = 29-JUL-2024 10:26:09

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               = 57
Total_Scans         = 57

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 21.8[dC]
X_90_Width          = 9.46[us]
X_Acq_Time          = 1.06430464[s]
X_Angle             = 30[deg]
X_Atn               = 4.9[dB]
X_Pulse             = 3.15333333[us]
Irr_Atn_Dec         = 22.45[dB]
Irr_Atn_Noe         = 22.45[dB]
Irr_Noise           = WALTZ
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.06430464[s]
```



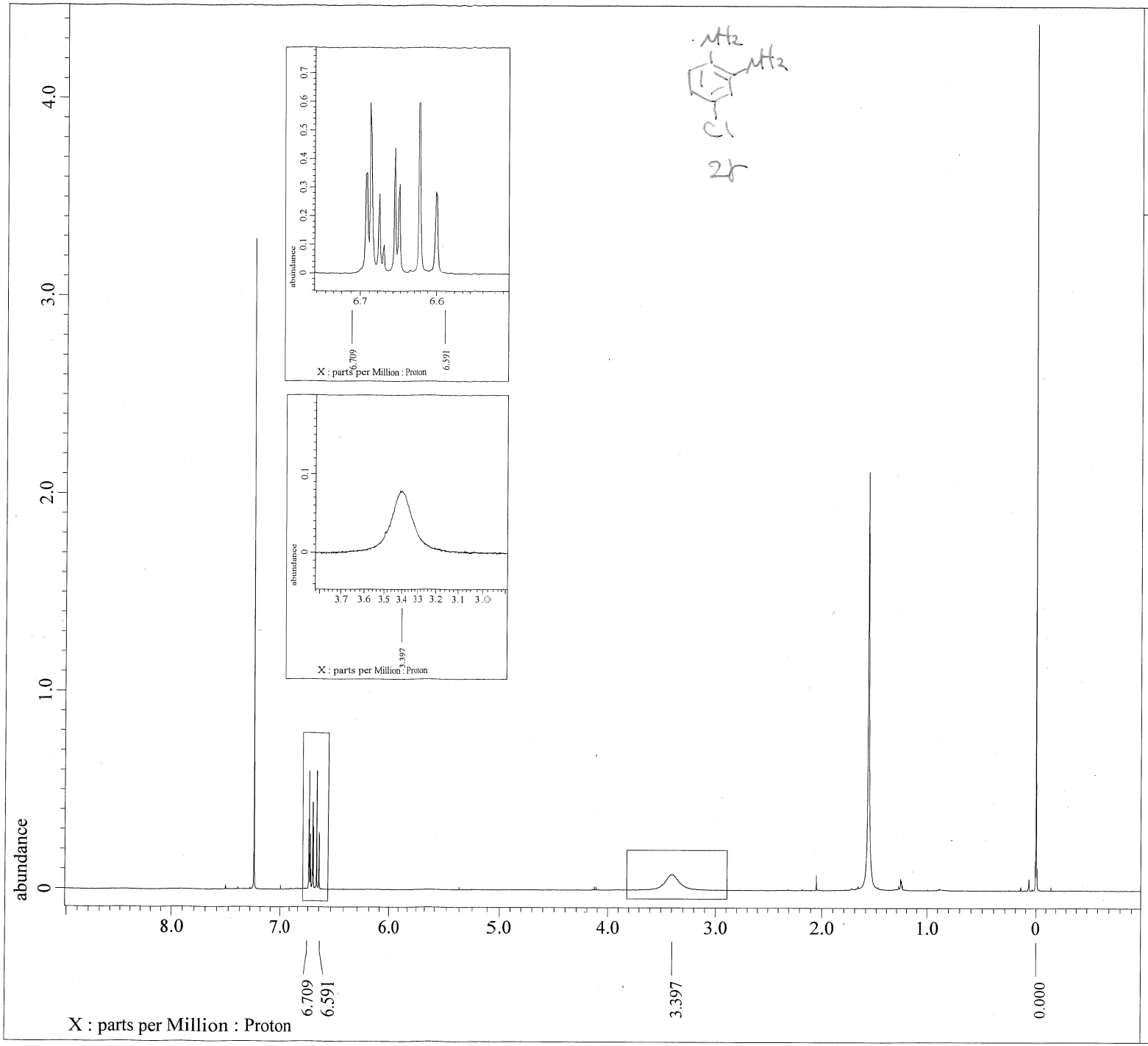

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 0.2[Hz], 0.0[s] )
trapezoid( 0[%], 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-105-pure2_Proton-1-1.jdf
```

Filename	= ASH-105-pure2_Proton-1-2.
Author	= element
Experiment	= proton.jxp
Sample_Id	= ASH-105-pure2
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 20-JUL-2024 15:38:15
Revision_Time	= 12-NOV-2024 10:34:24
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	= 40
Temp_Get	= 21.7[dC]
X_90_Width	= 6.7[us]
X_Acq_Time	= 2.18103808[s]
X_Angle	= 45[deg]
X_Atn	= 0.8[dB]
X_Pulse	= 3.35[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Presat	= FALSE
Initial_Wait	= 1[s]
Repetition_Time	= 7.18103808[s]



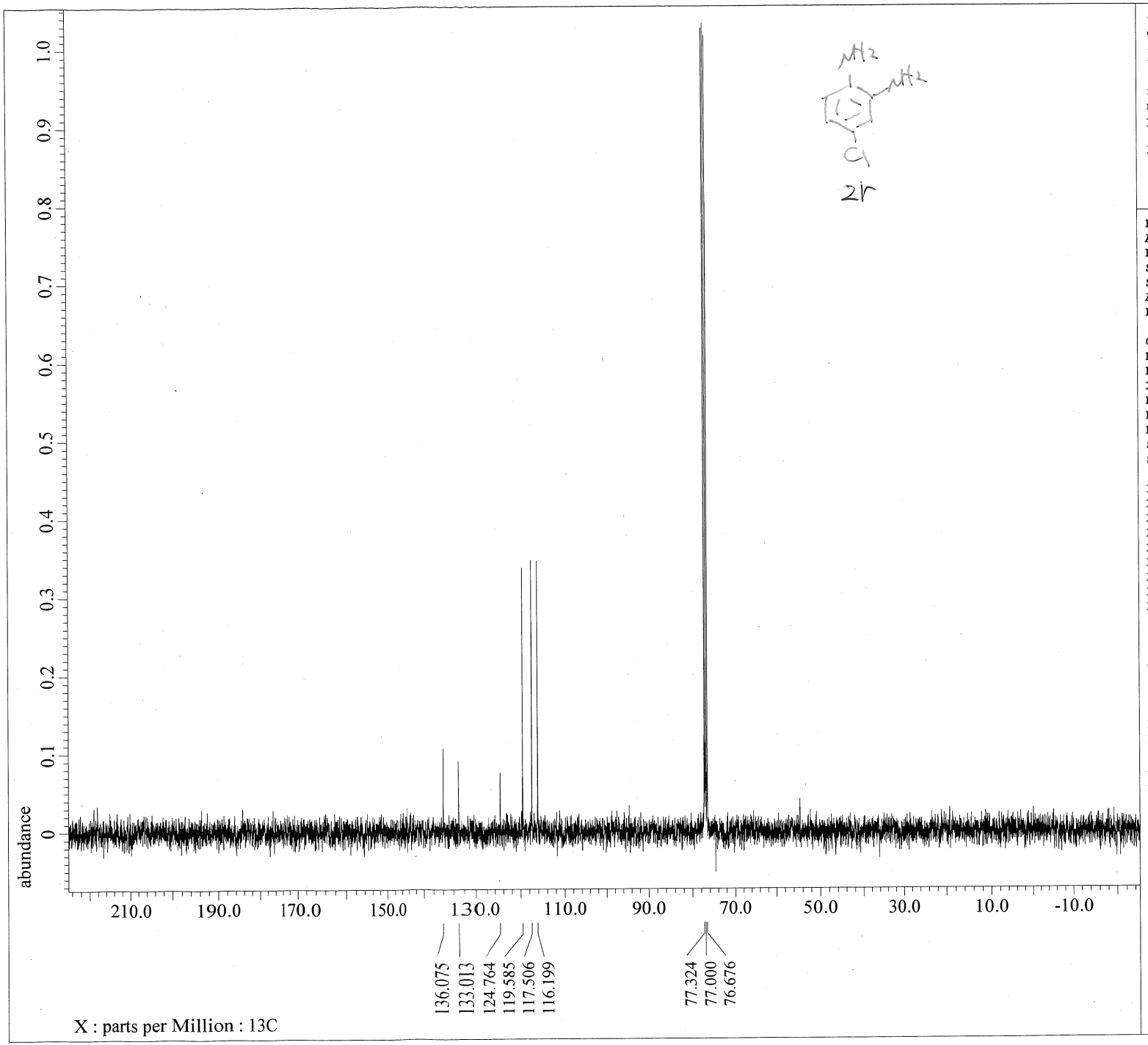

```
---- PROCESSING PARAMETERS ----
dc_balance( 0, FALSE )
sexp( 2.0[Hz], 0.0[s] )
trapezoid3( 0[%], 80[%], 100[%] )
zerofill( 1 )
fft( 1, TRUE, TRUE )
machinephase
ppm

Derived from: ASH-105-13C-1.jdf
```

```
Filename            = ASH-105-13C-2.jdf
Author              = element
Experiment          = single_pulse_dec
Sample_Id           = 1
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 29-JUL-2024 17:59:10
Revision_Time       = 29-JUL-2024 12:25:06

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               = 59
Total_Scans         = 59

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 22.2[dC]
X_90_Width          = 9.46[us]
X_Acq_Time          = 1.06430464[s]
X_Angle             = 30[deg]
X_Atn               = 4.9[dB]
X_Pulse             = 3.15333333[us]
Irr_Atn_Dec         = 22.45[dB]
Irr_Atn_Noe         = 22.45[dB]
Irr_Noise           = WALTZ
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.06430464[s]
```




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

Derived from: ASH-151-pure2_Proton-1-1.jdf
```

Filename	= ASH-151-pure2_Proton-
Author	= element
Experiment	= proton_auto.jxp
Sample_Id	= ASH-151-pure2
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 19-AUG-2024 21:16:11
Revision_Time	= 12-NOV-2024 10:40:18
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	= 9.2982153[T] (400[MHz
X_Acq_Duration	= 2.20725248[s]
X_Domain	= Proton
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]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 5[s]
Recvr_Gain	= 66
Temp_Get	= 19.9[dC]
X_90_Width	= 6.34[us]
X_Acq_Time	= 2.20725248[s]
X_Angle	= 45[deg]
X_Atn	= 5[dB]
X_Pulse	= 3.17[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 500
Dante_Presat	= FALSE
Decimation_Rate	= 0
Experiment_Path	= C:\Program Files\JEOL
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180
Presat_Time	= 5[s]




```
---- 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

Derived from: ASH-151-13C-1.jdf
```

```
Filename            = ASH-151-13C-2.jdf
Author              = element
Experiment          = single_pulse_dec
Sample_Id           = 1
Solvent             = CHLOROFORM-D
Actual_Start_Time   = 20-AUG-2024 03:48:35
Revision_Time       = 19-OCT-2024 13:35:25

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             = FALSE
Scans               = 52
Total_Scans         = 52

Relaxation_Delay    = 2[s]
Recvr_Gain          = 60
Temp_Get            = 21.2[dC]
X_90_Width          = 9.46[us]
X_Acq_Time          = 1.06430464[s]
X_Angle             = 30[deg]
X_Atn               = 4.9[dB]
X_Pulse             = 3.15333333[us]
Irr_Atn_Dec         = 22.45[dB]
Irr_Atn_Noe         = 22.45[dB]
Irr_Noise           = WALTZ
Decoupling          = TRUE
Initial_Wait        = 1[s]
Noe                 = TRUE
Noe_Time            = 2[s]
Repetition_Time     = 3.06430464[s]
```