

Supporting Information

Synthesis of Alkyl Aryl Ketones by Pd/Light Induced Carbonylative Cross-Coupling of Alkyl Iodides and Arylboronic Acids

Shuhei Sumino, Takahito Ui, and Ilhyong Ryu*

Department of Chemistry, Graduate School of Science, Osaka Prefecture University
Sakai, Osaka 599-8531, Japan
ryu@c.s.osakafu-u.ac.jp

Table of Contents

General Information.....	S2
Typical procedure for the synthesis of 3a	S2
Procedure for the synthesis of 6	S2
Spectral data of 3a-q , 6	S3-S8
Spectra (¹ H, ¹³ C, and ¹⁹ F).....	S9-S46

General Information

Thin layer chromatography (TLC) was performed on Merck precoated plates (silica gel 60 F254, Art 5715, 0.25 mm) and were visualized by fluorescence quenching under UV light or by staining with *p*-anisaldehyde/AcOH/H₂SO₄/EtOH, or 12MoO₃.H₃PO₄/EtOH. The products were purified by flash chromatography on silica gel (Kanto Chem. Co. Silica Gel 60N (spherical, neutral, 40-50 µm)) and, if necessary, were further purified by recycling preparative HPLC (Japan Analytical Industry Co. Ltd., LC-918) equipped with GPC columns (JAIGEL-1H + JAIGEL-2H columns) using CHCl₃ as eluent. ¹H NMR spectra were recorded with a JEOL JNM-ECS400 (400 MHz) spectrometer and referenced to the peak of the residual TMS at 0.00 ppm. ¹³C NMR spectra were recorded with a JEOL JNM-ECS400 (100 MHz) spectrometer and referenced to the solvent peak at 77.16 ppm. Splitting patterns are indicated as follows: br, broad; s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. Infrared spectra were recorded on a JASCO FT/IR-4100 spectrometer and are reported as wavenumber (cm⁻¹). Conventional and high-resolution mass spectra were recorded with a JEOL MS700 spectrometer.

Typical procedure for the synthesis of nonanophenone (**3a**)

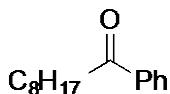
A magnetic stirring bar, iodoctane (**1a**) (121.9mg, 0.5 mmol), phenylboronic acid (**2a**) (91.7mg, 0.75 mmol), PdCl₂(PPh₃)₂ (16.4 mg, 0.025 mmol), K₂CO₃ (139.1 mg, 1.0 mmol), C₆H₆ (4 mL), and H₂O (2 mL) were placed in a stainless steel autoclave for photoreaction equipped with an inserted Pyrex glass liner. The autoclave was closed, purged three times with carbon monoxide, pressurized with 45 atm of CO and then irradiated by Xenon arc lamp (500 W) with stirring for 16 h. Excess CO was discharged at room temperature after the reaction. The reaction mixture was poured into water (20 mL) and extracted with ether (20 mL × 3). The combined ether layer was washed with brine and dried over MgSO₄, then filtered and concentrated *in vacuo* to give a residue, which was subjected to silica gel column chromatography using hexan/EtOAc = 250/1 as eluent affording nonanophenone (**3a**) (100.6 mg, 91%)

Procedure for the synthesis of ethyl 4-benzoyldecanoate (**6**)

A magnetic stirring bar, ethyl iodoacetate (**4**) (110.0 mg, 0.5 mmol), 1-octene (**5**) (566.9 mg, 5.1 mmol), phenylboronic acid (**2a**) (94.1 mg, 0.77 mmol), PdCl₂(PPh₃)₂ (16.1 mg, 0.025 mmol), K₂CO₃ (136.2 mg, 1.0 mmol), C₆H₆ (4 mL) and H₂O (2 mL) were placed in a stainless steel autoclave for photoreaction equipped with an inserted Pyrex glass liner. The autoclave was closed, purged three times with carbon monoxide, pressurized with 45 atm of CO and then irradiated by Xenon arc lamp (500 W) with stirring for 16 h. Excess CO was discharged at room temperature after the reaction. The reaction mixture was poured into water (20 mL) and extracted with ether (20 mL × 3). The

combined ether layer was washed with brine, and dried over MgSO_4 , then filtered and concentrated *in vacuo* to give a residue, which was subjected to silica gel column chromatography using hexan/EtOAc = 200/1 as eluent affording ethyl 4-benzoyldecanoate (**6**) (96.5 mg, 62 %)

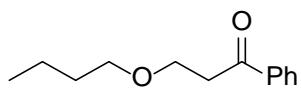
Nonanophenone (3a)



This compound is previously known and commercially available: Vautravers, N. R.; Regent, D. D.; Breit, B. *Chem. Commun.* **2011**, 47, 6635.

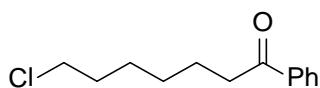
Colorless oil; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ 7.97-7.95 (m, 2H), 7.57-7.53 (m, 1H), 7.48-7.44 (m, 2H), 2.98-2.95 (m, 2H), 1.79-1.70 (m, 2H), 1.41-1.27 (m, 10H), 0.88 (m, 3H); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ 200.61, 137.14, 132.91, 128.60, 128.11, 38.70, 31.93, 29.54, 29.46, 29.26, 24.44, 22.74, 14.19.

3-Butoxy-1-phenylpropan-1-one (3b)



Colorless oil; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ 7.98-7.96 (m, 2H), 7.59-7.55 (m, 1H), 7.49-7.45 (m, 2H), 3.87-3.84 (m, 2H), 3.47 (t, $J = 6.8$ Hz, 2H), 3.28-3.24 (m, 2H), 1.57-1.51 (m, 2H), 1.38-1.31 (m, 2H), 0.92-0.88 (m, 3H); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ 198.65, 137.13, 133.21, 128.68, 128.24, 71.21, 66.11, 39.04, 31.83, 19.40, 14.02; IR (neat) 2958 cm^{-1} , 1682 cm^{-1} , 1112 cm^{-1} ; MS (relative intensity) m/z 206 (M^+ , 13), 151 (34), 133 (66), 105 (100), 77 (78); HRMS (EI) m/z calcd for $\text{C}_{13}\text{H}_{18}\text{O}_2$ 206.1307, found 206.1308.

7-Chloro-1-phenylheptan-1-one (3c)

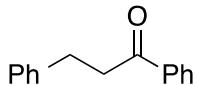


This compound is previously known and commercially available: Rieke, R. D.; Stack, D. E.; Dawson, B. T.; Wu, T.-C. *J. Org. Chem.* **1993**, 58, 2483.

White solid (m.p. = 32.7-33.6 °C); $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ 7.97-7.95 (m, 2H), 7.58-7.54 (m, 1H), 7.48-7.45 (m, 2H), 3.54 (t, $J = 6.6$ Hz, 2H), 3.00-2.97 (m, 2H), 1.82-1.73 (m, 4H), 1.50-1.41 (m, 4H); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ 200.40, 137.11, 133.08, 128.69, 128.14, 45.18, 38.49, 32.52,

28.67, 26.84, 24.17.

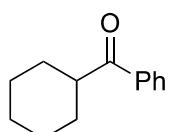
1,3-Diphenyl-propan-1-one (**3d**)



This compound is previously known: Colbon, P.; Ruan, J.; Purdie, M.; Xiao, J. *Org. Lett.* **2010**, *12*, 3670.

White solid (m.p. = 66.9-68.1 °C); ¹H-NMR (CDCl₃, 400 MHz) δ 7.97-7.95 (m, 2H), 7.56-7.47 (m, 1H), 7.45-7.43 (m, 2H), 7.30-7.21 (m, 5H), 3.33-3.29 (m, 2H), 3.09-3.05 (m, 2H); ¹³C-NMR (CDCl₃, 100 MHz) δ 199.30, 141.38, 136.91, 133.17, 128.70, 128.63, 128.53, 128.13, 126.23, 40.55, 30.20.

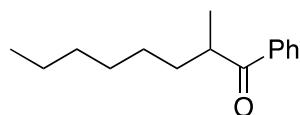
Cyclohexylphenylmethanone (**3e**)



This compound is previously known and commercially available: Hsieh, J.-C.; Chem, Y.-C.; Cheng, A.-Y.; Tseng, H.-C. *Org. Lett.* **2012**, *14*, 1282.

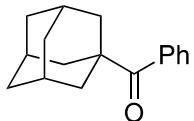
White solid (m.p. = 55.6-56.1 °C); ¹H-NMR (CDCl₃, 400 MHz) δ 7.96-7.94 (m, 2H), 7.56-7.53 (m, 1H), 7.48-7.44 (m, 2H), 3.30-3.23 (m, 1H), 1.91-1.83 (m, 3H), 1.78-1.72 (m, 2H), 1.57-1.21 (m, 5H); ¹³C-NMR (CDCl₃, 100 MHz) δ 203.94, 136.38, 132.80, 128.65, 128.32, 45.67, 29.49, 26.05, 25.93.

2-Methyl-1-phenyloctan-1-one (**3f**)



Colorless oil; ¹H-NMR (CDCl₃, 400 MHz) δ 7.97-7.94 (m, 2H), 7.58-7.54 (m, 1H), 7.49-7.45 (m, 2H), 3.51-3.42 (m, 1H), 1.83-1.76 (m, 1H), 1.48-1.41 (m, 1H), 1.34-1.22 (m, 8H), 1.19 (d, *J* = 6.8 Hz, 3H), 0.86 (t, *J* = 6.8 Hz, 3H); ¹³C-NMR (CDCl₃, 100 MHz) δ 204.68, 136.87, 132.89, 128.70, 128.33, 40.67, 33.85, 31.80, 29.51, 27.49, 22.70, 17.34, 14.17; IR (neat) 2930 cm⁻¹, 2856 cm⁻¹, 1682 cm⁻¹, 703 cm⁻¹; MS (relative intensity) m/z 218 (M⁺, 10), 134 (48), 105 (100), 77 (40); HRMS (EI) m/z calcd for C₁₅H₂₂O 218.1671, found 218.1676.

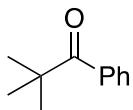
1-Benzoyladamantane (3g)



This compound is previously known: Lo Fiego, M. J.; Lockhart, M. T.; Chopra, A. B. *J. Organomet. Chem.* **2009**, *694*, 3674.

White solid (m.p. = 49.3-50.1 °C); ^1H -NMR (CDCl_3 , 400 MHz) δ 7.55-7.53 (m, 2H), 7.45-7.36 (m, 3H), 2.10-2.00 (m, 10H), 1.80-1.69 (m, 5H); ^{13}C -NMR (CDCl_3 , 100 MHz) δ 210.22, 139.65, 130.21, 128.00, 127.17, 46.95, 39.14, 36.59, 28.18.

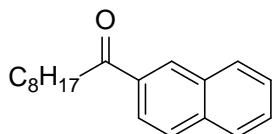
2,2-Dimethyl-1-phenyl-propan-1-one (3h)



This compound is previously known: Zhu, Y.; Zhao, B.; Shi, Y. *Org. Lett.* **2013**, *15*, 992.

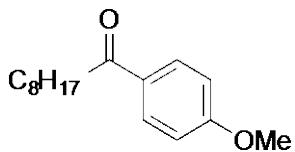
Colorless oil; ^1H -NMR (CDCl_3 , 400 MHz) δ 7.70-7.68 (m, 2H), 7.48-7.38 (m, 3H), 1.35 (s, 9H); ^{13}C -NMR (CDCl_3 , 100 MHz) δ 209.48, 138.72, 130.95, 128.18, 127.96, 28.15.

1-(2-Naphthyl)-nonan-1-one (3i)



White solid (m.p. = 54.1-54.7 °C); ^1H -NMR (CDCl_3 , 400 MHz) δ 8.47 (s, 1H), 8.05-8.03 (m, 1H), 7.96 (d, J = 8.0 Hz, 1H), 7.91-7.87 (m, 2H), 7.61-7.51 (m, 2H), 3.12-3.08 (m, 2H), 1.85-1.76 (m, 2H), 1.44-1.22 (m, 10H), 0.90-0.87 (m, 3H); ^{13}C -NMR (CDCl_3 , 100 MHz) δ 200.76, 135.64, 134.55, 132.69, 129.75, 129.67, 128.52, 128.47, 127.90, 126.84, 124.11, 38.87, 31.99, 29.63, 29.58, 29.35, 24.69, 22.82, 14.27; IR (neat) 2933 cm^{-1} , 2849 cm^{-1} , 1683 cm^{-1} , 1463 cm^{-1} , 803 cm^{-1} , 755 cm^{-1} ; MS (relative intensity) m/z 268 (M^+ , 19), 170 (100), 155 (83), 127 (49); HRMS (EI) m/z calcd for $C_{19}H_{24}O$ 268.1827, found 268.1833.

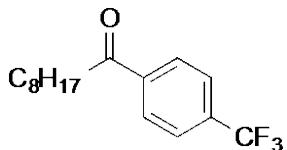
1-(4-Methoxyphenyl)nonan-1-one (3j)



This compound is previously known: Vautravers, N. R.; Reqert, D. D.; Breit, B. *Chem. Commun.* **2011**, 47, 6635.

White solid (m.p. = 43-44 °C); ¹H-NMR (CDCl₃, 400 MHz) δ 7.94 (d, *J* = 8.0 Hz, 2H), 6.93 (d, *J* = 7.6 Hz, 2H), 3.87 (s, 3H), 2.93-2.88 (m, 2H), 1.79-1.69 (m, 2H), 1.42-1.20 (m, 10H), 0.88 (t, *J* = 6.8 Hz, 3H); ¹³C-NMR (CDCl₃, 100 MHz) δ 199.29, 163.36, 130.38, 130.24, 113.72, 55.50, 38.38, 31.94, 29.56, 29.53, 29.27, 24.71, 22.74, 14.19.

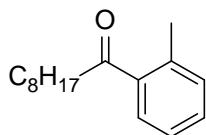
1-(4-Trifluoromethylphenyl)nonan-1-one (3k)



This Compound is previously known: Dohi, S.: Moriyama, K.; Togo, H. *Tetrahedron* **2012**, 68, 6557.

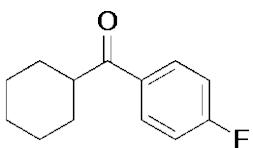
White solid (m.p. = 35.5-36.1 °C); ¹H-NMR (CDCl₃, 400 MHz) δ 8.07-8.05 (m, 2H), 7.74-7.72 (m, 2H), 3.00-2.97 (m, 2H), 1.80-1.71 (m, 2H), 1.39-1.27 (m, 10H), 0.88 (t, *J* = 6.8 Hz, 3H); ¹³C-NMR (CDCl₃, 100 MHz) δ 199.70, 139.34 (*q*, *J*_{C-F} = 32.5 Hz), 128.51, 125.79 (*q*, *J*_{C-F} = 3.8 Hz), 123.77 (*q*, *J*_{C-F} = 270.2 Hz), 39.08, 31.97, 29.56, 29.44, 29.30, 24.28, 22.80, 14.24; ¹⁹F-NMR (CDCl₃, 377 MHz) δ -62.98.

1-(*o*-Tolyl)nonan-1-one (3l)



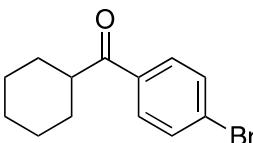
Colorless oil; ¹H-NMR (CDCl₃, 400 MHz) δ 7.62-7.60 (m, 1H), 7.37-7.34 (m, 1H), 7.26-7.23 (m, 2H), 2.90-2.86 (m, 2H), 2.48 (s, 3H), 1.75-1.66 (m, 2H), 1.39-1.27 (m, 10H), 0.89-0.87 (m, 3H); ¹³C-NMR (CDCl₃, 100 MHz) δ 205.09, 138.46, 137.88, 131.96, 131.09, 128.36, 125.71, 41.80, 31.95, 29.56, 29.45, 29.29, 24.55, 22.77, 21.30, 14.22; IR (neat) 2925 cm⁻¹, 2854 cm⁻¹, 1687 cm⁻¹, 1456 cm⁻¹, 753 cm⁻¹; MS (relative intensity) m/z 232 (M⁺, 8), 134 (47), 119 (100), 91 (59); HRMS (EI) m/z calcd for C₁₆H₂₄O 232.1827, found 232.1820.

Cyclohexyl-(4-fluorophenyl)methanone (**3m**)



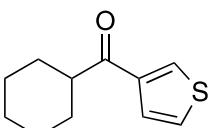
Colorless oil; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ 7.99-7.95 (m, 2H), 7.13 (t, $J = 8.4$ Hz, 2H), 3.25-2.17 (m, 1H), 1.90-1.82 (m, 4H), 1.78-1.70 (m, 1H), 1.55-1.22 (m, 5H); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ 202.36, 165.65 (d, $J_{\text{C-F}} = 251.1$ Hz), 132.76 (d, $J_{\text{C-F}} = 2.9$ Hz), 130.97 (d, $J_{\text{C-F}} = 8.6$ Hz), 115.74 (d, $J_{\text{C-F}} = 22.0$ Hz), 45.68, 29.52, 26.03, 25.94; $^{19}\text{F-NMR}$ (CDCl_3 , 377 MHz) δ -105.87; IR (neat) 2943 cm^{-1} , 2855 cm^{-1} , 1681 cm^{-1} , 1236 cm^{-1} , 843 cm^{-1} ; MS (relative intensity) m/z 206 (M^+ , 25), 151 (20), 123 (100), 95 (21); HRMS (EI) m/z calcd for $\text{C}_{13}\text{H}_{15}\text{FO}$ 206.1107, found 206.1102.

Cyclohexyl-(4-bromophenyl)methanone (**3n**)



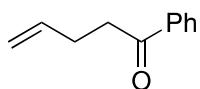
White solid (m.p. = 76.5-77.0 °C); $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ 7.81 (d, $J = 8.4$ Hz, 2H), 7.60 (d, $J = 8.0$ Hz, 2H), 3.30-3.23 (m, 1H), 1.90-1.81 (m, 4H), 1.79-1.70 (m, 1H), 1.54-1.21 (m, 5H); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ 202.92, 135.11, 132.00, 129.95, 127.94, 45.71, 29.45, 26.01, 25.91; IR (KBr) 2928 cm^{-1} , 1680 cm^{-1} , 833 cm^{-1} ; MS (relative intensity) m/z 268 (M^+ , 20), 266 (M^+ , 21), 187 (40), 185 (100), 183 (99), 157 (20), 155 (20); HRMS (EI) m/z calcd for $\text{C}_{13}\text{H}_{15}{^{79}\text{BrO}}$ 266.0306, found 266.0297.

Cyclohexyl-(3-thienyl)methanone (**3o**)



White solid (m.p. = 55.0-55.8 °C); $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ 8.05 (t, $J = 1.4$ Hz, 1H), 7.55 (d, $J = 4.8$ Hz, 1H), 7.32-7.30 (m, 1H), 3.10-3.03 (m, 1H), 1.90-1.81 (m, 4H), 1.76-1.70 (m, 1H), 1.56-1.21 (m, 5H); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ 198.31, 141.64, 131.79, 127.38, 126.32, 47.71, 29.45, 25.98, 25.90; IR (KBr) 1654 cm^{-1} , 2850 cm^{-1} ; MS (relative intensity) m/z 194 (M^+ , 45), 111 (100); HRMS (EI) m/z calcd for $\text{C}_{11}\text{H}_{14}\text{OS}$ 194.0765, found 194.0758.

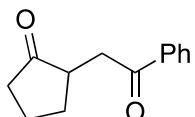
1-Phenyl-4-penten-1-one (3p)



This product is previously known and commercially available: Pirtsch, M.; Paria, S.; Matsuno, T.; Reiser, O.; Isobe, H. *Chem. -Eur. J.* **2012**, *18*, 7336.

Colorless oil; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ 7.98-7.96 (m, 2H), 7.60-7.54 (m, 1H), 7.49-7.45 (m, 2H), 5.92 (ddt, $J = 16.8$ Hz, 10.0 Hz, 6.4 Hz, 1H), 5.09 (ddd, $J = 16.8$ Hz, 3.2 Hz, 1.6 Hz, 1H), 5.02 (ddd, $J = 10.0$ Hz, 3.2 Hz, 1.6 Hz, 1H), 3.10-3.07 (m, 2H), 2.52-2.47 (m, 2H); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ 199.54, 137.41, 137.01, 133.12, 128.70, 128.13, 115.40, 37.84, 28.24.

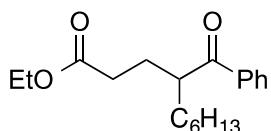
2-Phenacyl-cyclopentanone (3q)



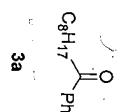
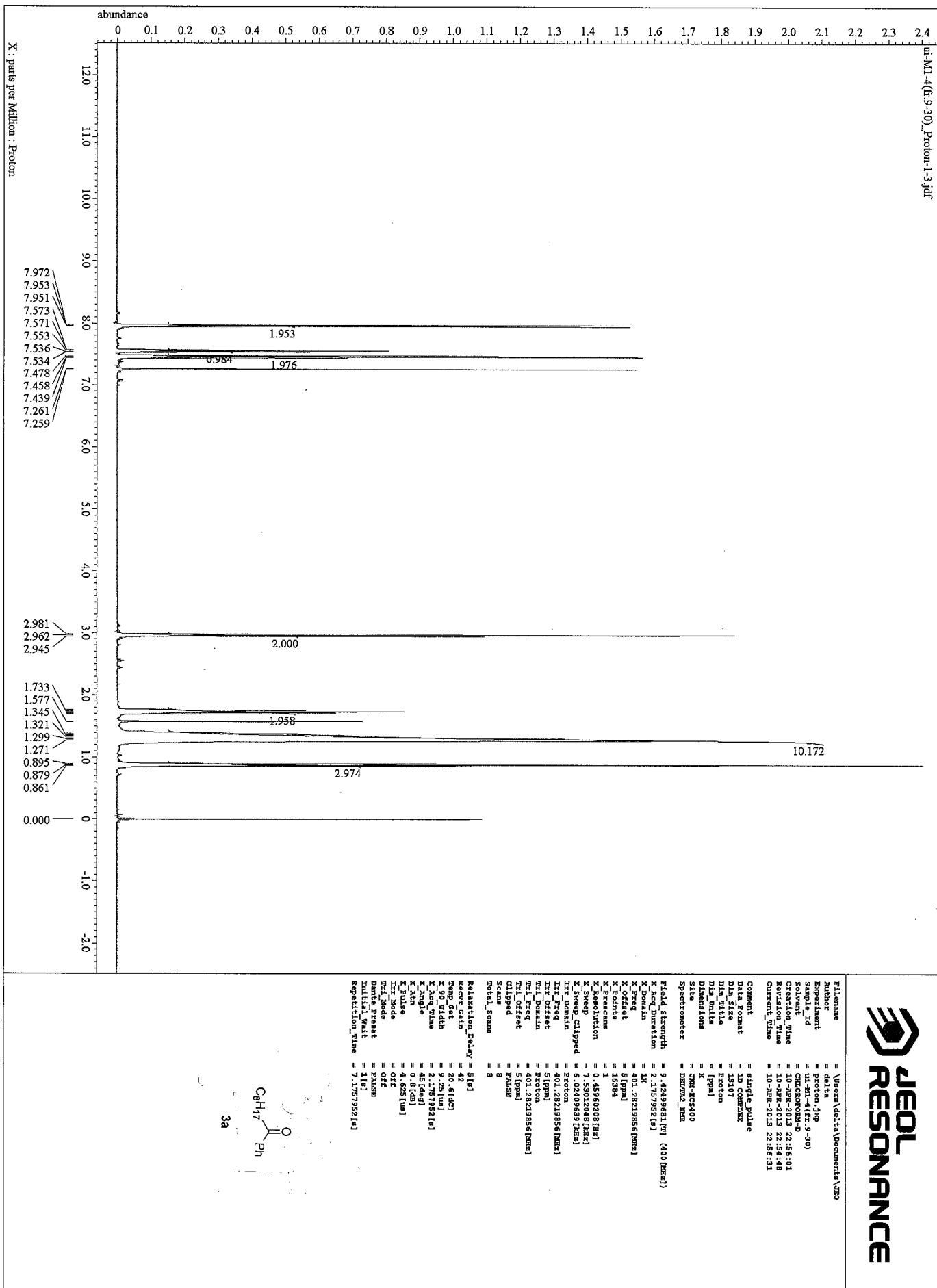
This product is previously known; Thompson, B. B.; Montgomery, J. *Org. Lett.* **2011**, *13*, 3289.

Colorless oil; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ 7.96 (d, $J = 7.6$ Hz, 2H), 7.59-7.56 (m, 1H), 7.46 (t, $J = 7.6$ Hz, 2H), 3.54 (dd, $J = 3.2$ Hz, 18.4 Hz, 1H), 3.06 (dd, $J = 8.4$ Hz, 17.6 Hz, 1H), 2.61-2.69 (m, 1H), 2.45-2.26 (m, 3H), 2.13-2.06 (m, 1H), 1.95-1.81 (m, 1H), 1.69-1.57 (m, 1H); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ 220.53, 198.04, 136.63, 133.32, 128.69, 128.10, 45.15, 38.70, 37.64, 29.76, 20.91.

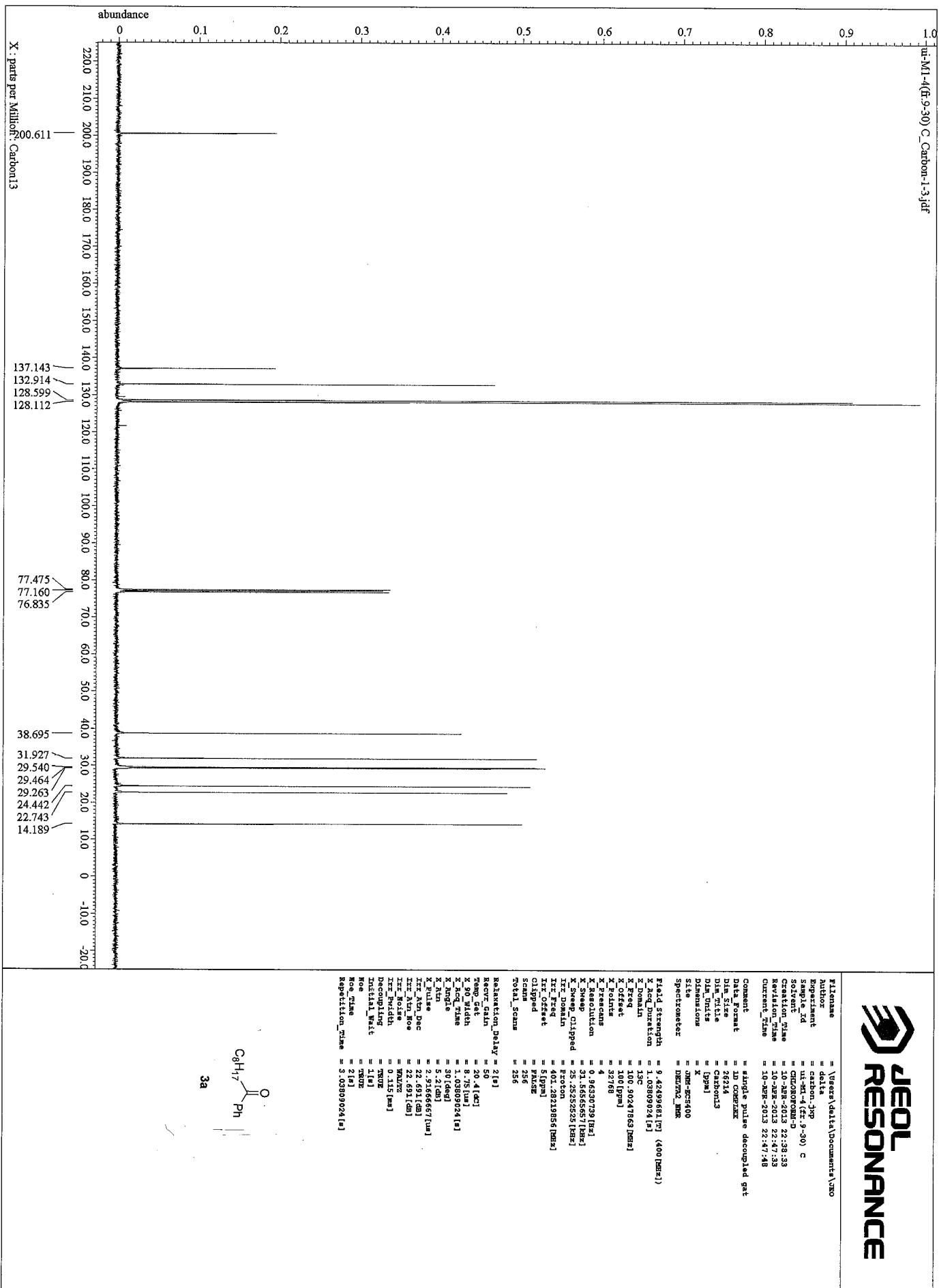
Ethyl 4-benzoyldecanoate (6)



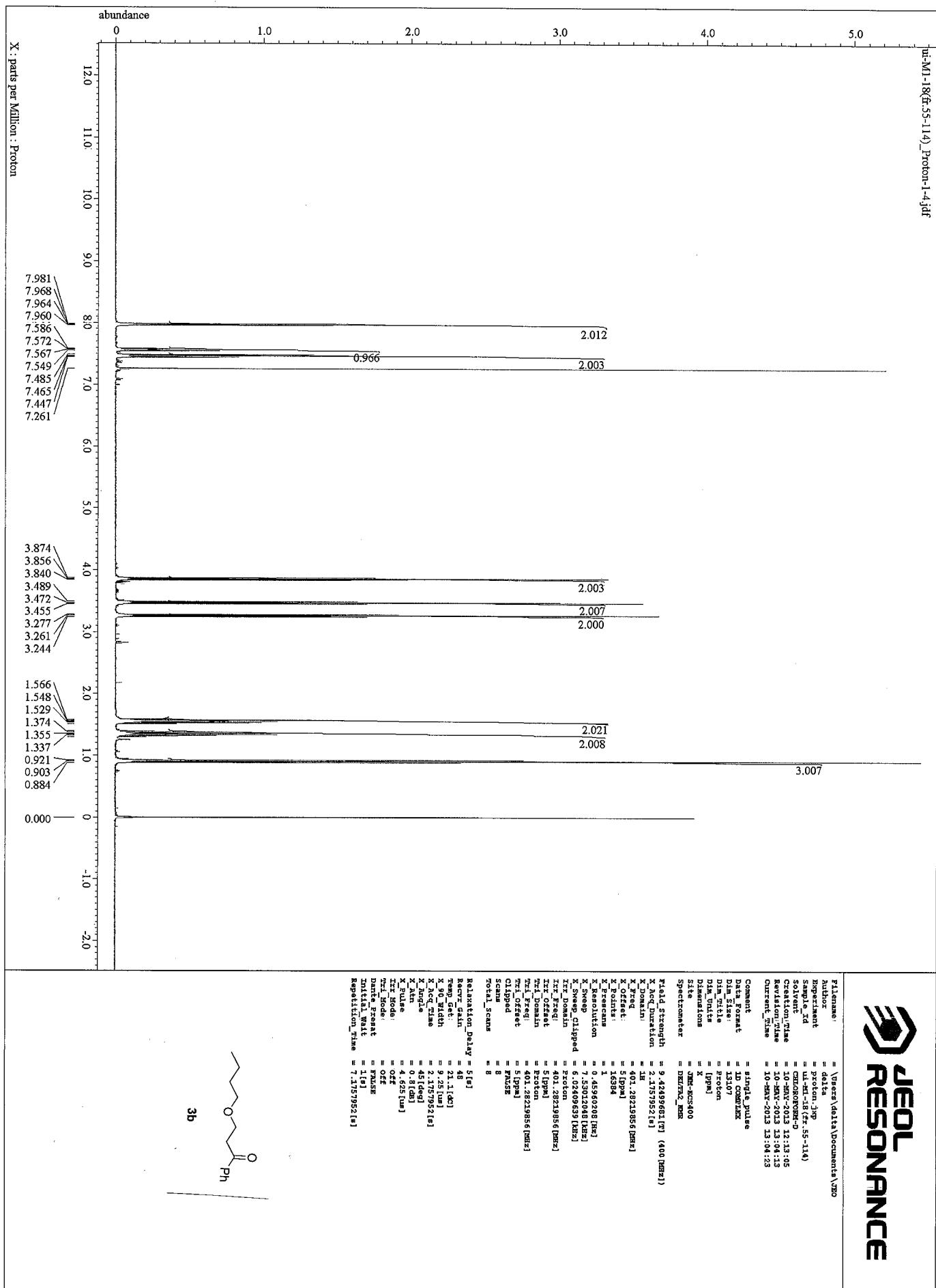
Colorless oil; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ 7.97-7.95 (m, 2H), 7.59-7.55 (m, 1H), 7.49-7.45 (m, 2H), 4.09 (q, $J = 6.9$ Hz, 2H), 3.55-3.49 (m, 1H), 2.38-2.30 (m, 1H), 2.26-2.20 (m, 1H), 2.18-2.06 (m, 1H), 1.90-1.81 (m, 1H), 1.77-1.72 (m, 1H), 1.51-1.45 (m, 1H), 1.26-1.19 (m, 1H), 0.86-0.82 (m, 3H); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ 203.90, 173.40, 137.91, 133.13, 128.77, 128.32, 60.43, 45.02, 32.56, 31.97, 31.69, 29.48, 27.37, 26.93, 22.64, 14.27, 14.13; IR (neat) 1216 cm^{-1} , 1680 cm^{-1} , 1731 cm^{-1} , 2929 cm^{-1} , 704 cm^{-1} ; MS (relative intensity) m/z 304 (M^+ , 3), 259 (10), 220 (29), 105 (100), 77 (19); HRMS (EI) m/z calcd for $C_{19}H_{28}O_3$ 304.2038, found 304.2031.

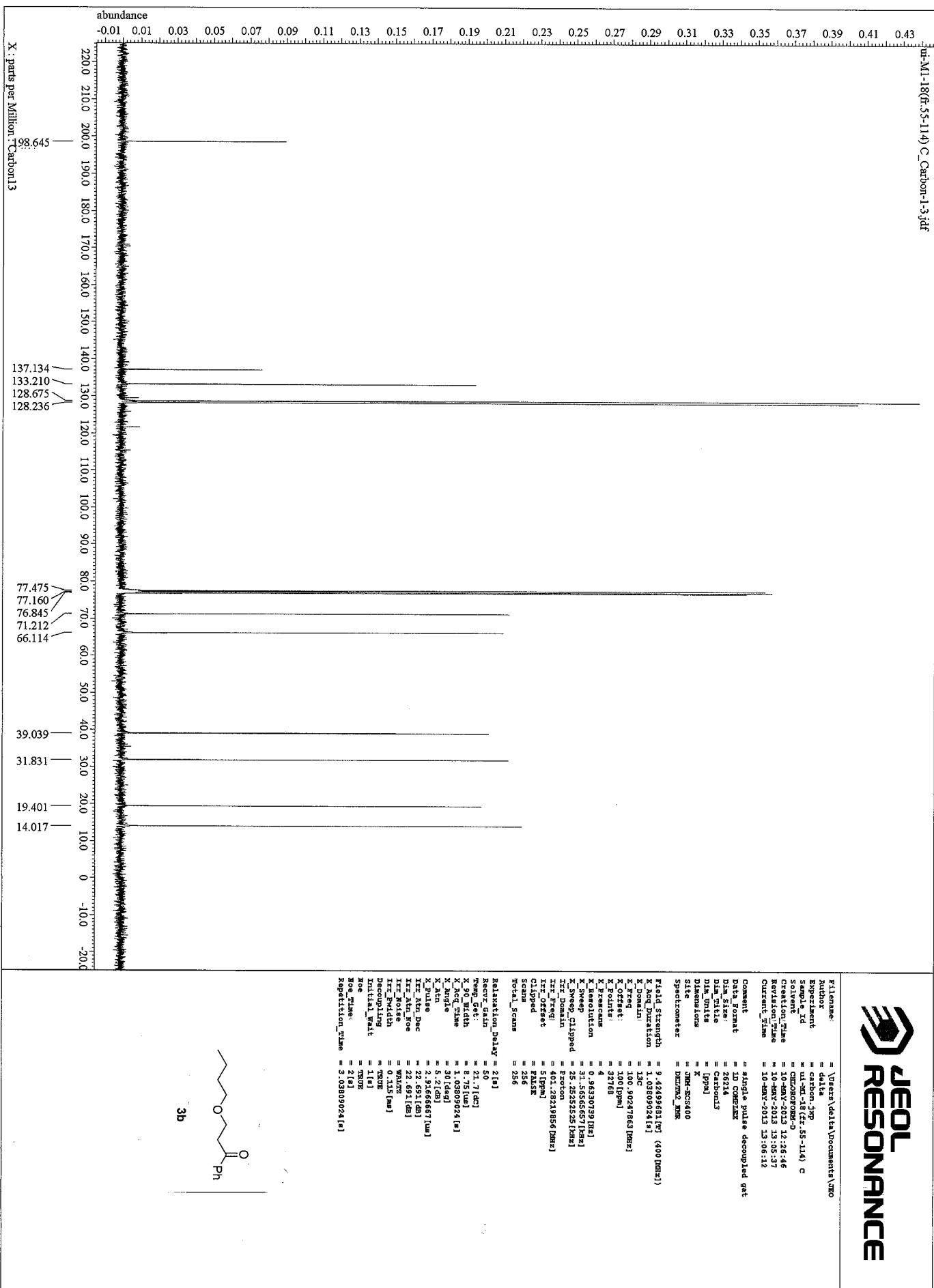


32



JEOL
RESONANCE





JEOL
RESONANCE

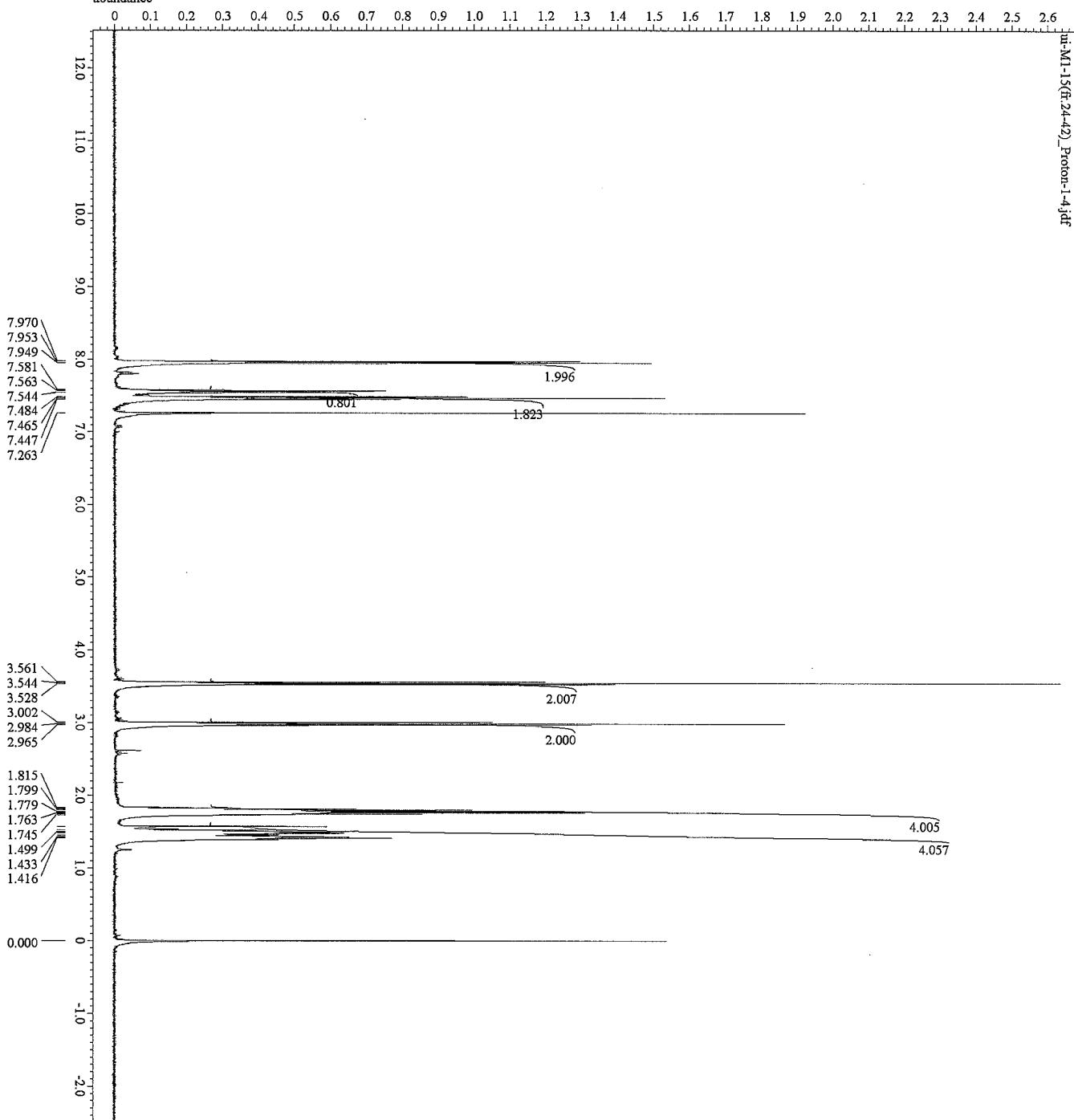
```

filename = C:\Users\delta\Documents\J
author = delta
experiment = proton.JDP
sample_id = ui-M1-15(G-24-42)
solvent = CHLOROFORM-D
creation_time = 25-APR-2013 23:48:49
revision_time = 14-MAY-2013 20:23:48
current_time = 14-MAY-2013 20:23:48

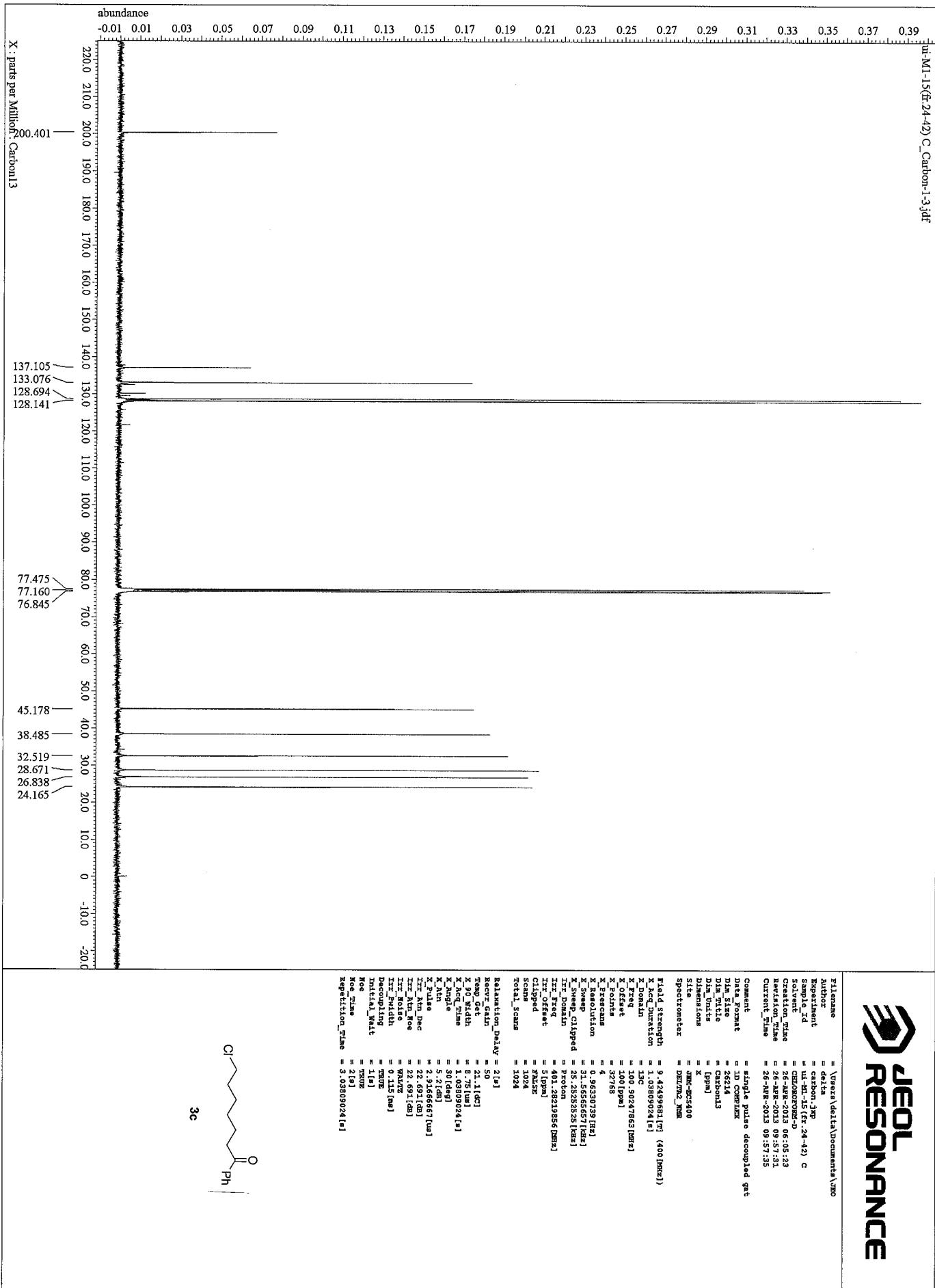
comment = single pulse
data_format = 1D complex
dim_size = 13107
dim_title = Proton
dim_units = [PPM]
dimensions = X
site = JMS-XE3400
specrometer = JEOLME2_NMR
field_strength = 9.42499611[MHz] (400 MHz)
XAccq_Direction = 1
Xdomain = 1H
Xfreq = 401.28239856[MHz]
Xoffset = 5[ppm]
Xpoints = 16384
Xprescans = 1
Xresolution = 0.45566208[Hz]
Xsweep = 7.53012048[ppm]
Xsweep_clipped = 7.53012048[ppm]
Xzdomain = Proton
Xzfreq = 6.02409659[MHz]
Xzoffset = 401.28239856[MHz]
Zdomain = Proton
Zfreq = 401.28239856[MHz]
Tr1_offset = 5[ppm]
Tr1_offset_clipped = 5[ppm]
scans = 2
total_scans = 2

relaxation_delay = 5[s]
servo_gain = 46
temp_get = 20.5[deg]
Xpp_width = 9.25[us]
Xacc_rate = 2.1757955[us]
Xangle = 45.0[deg]
Xatt = 0.8[sec]
Xpulse = 4.625[us]
IR_Mode = off
Pr1_Mode = off
Pr1_Offset = FALSE
Pr1_Offset_Value = 1[us]
Repetition_Time = 7.1757952[us]

```



X : parts per Million : Proton



JEOL
RESONANCE

File Name = \Users\alok\Documents\J00

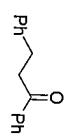
Author = dexter
Experiment = proton_1d
Sample_ID = 11-sec71f-5-47
Slient = CDR0000000
Creation_Time = 27-Mar-2013 16:54:40
Revision_Time = 27-Mar-2013 16:55:39
Current_Time = 27-Mar-2013 16:55:39

Comment = single_pulse
Data_Format = 1D_COSINEK
D1_Gain = 13.07
D1_Middle = FR1ON
D1_Monita = [ppm]
D1_Minimum = X
D1_Note = JMR-2125-400
D1_Offset = 1620ppm_ASK

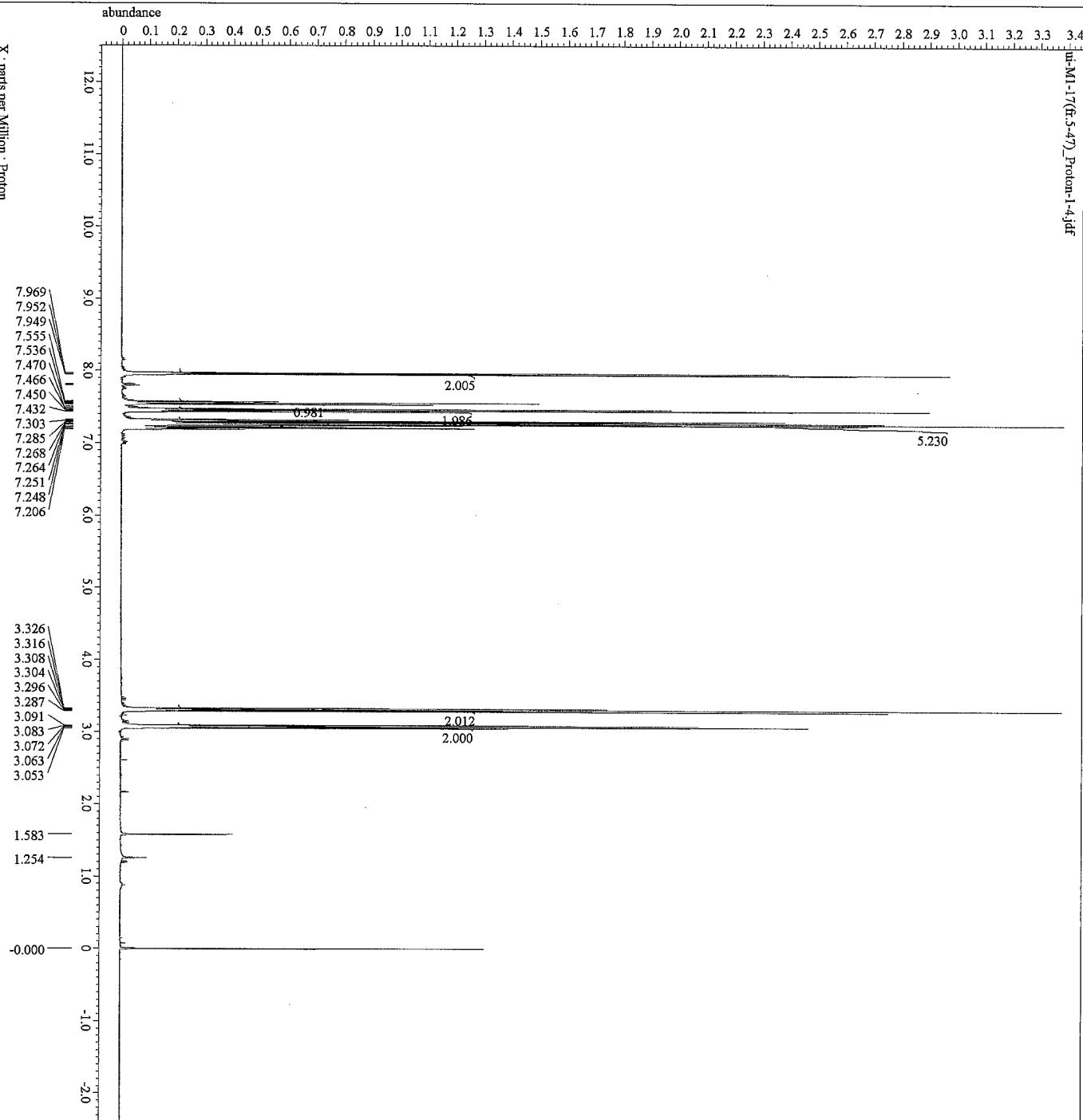
Field_Strength = 9.42996621[T] (400 [MHz])
Freq_Duration = 2.1757952 [Hz]

Integration = 1H
Kern = 401.28219856 [MHz]
Offset = 1684
Pulse = 1
PulseTime = 0.4560208 [Hz]
ReSolution = 7.33012048 [Hz]
Sweep_Clipped = 6.02409659 [Hz]
Tx_Domain = Proton
Tx_Freq = 401.28219856 [MHz]
Tx_Offset = 5 [ppm]
Tx_Domain = Proton
Tx_Freq = 401.28219856 [MHz]
Tx_Offset = 5 [ppm]
Clipped = PULSE
Scans = 8
Total_Scans = 8

Relaxation_Delay = 5 [s]
Recv_Gain = 40
Temp_Get = 20.8 [deg]
X1_0_Width = 9.25 [us]
X1q_q_Tune = 2.1757952 [s]
X1qAngle = 45 [deg]
X1qInv = 0.8 [us]
X1qRate = 4.625 [us]
Tx1_Mode = off
Tx1_Pulse = FALSE
Dantic_Preset = 1 [s]
Initial_Wait = 7.1757952 [s]
Repetition_Time =



3d



X : parts per Million : Proton

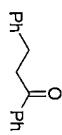
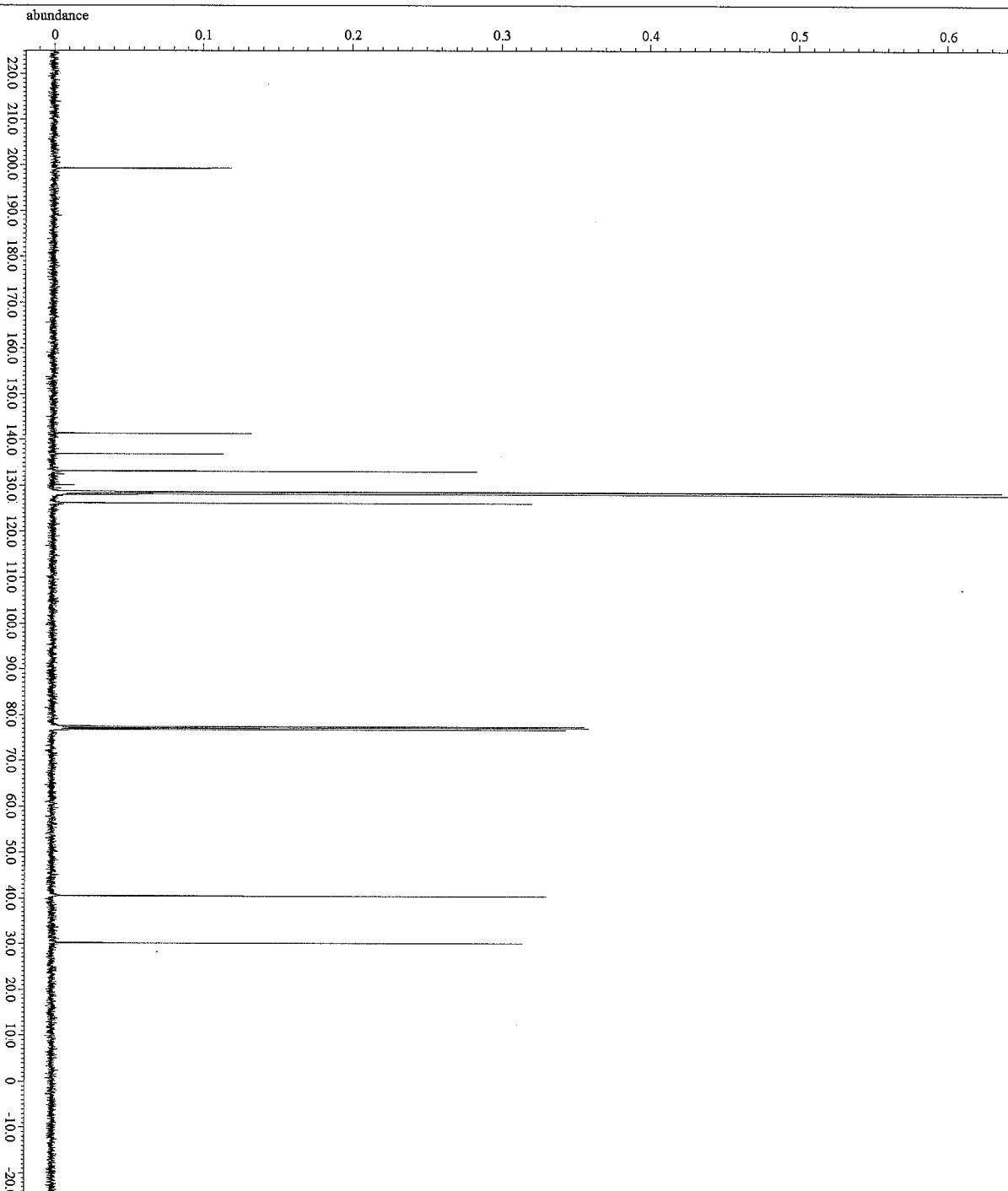
JEOL
RESONANCE

```

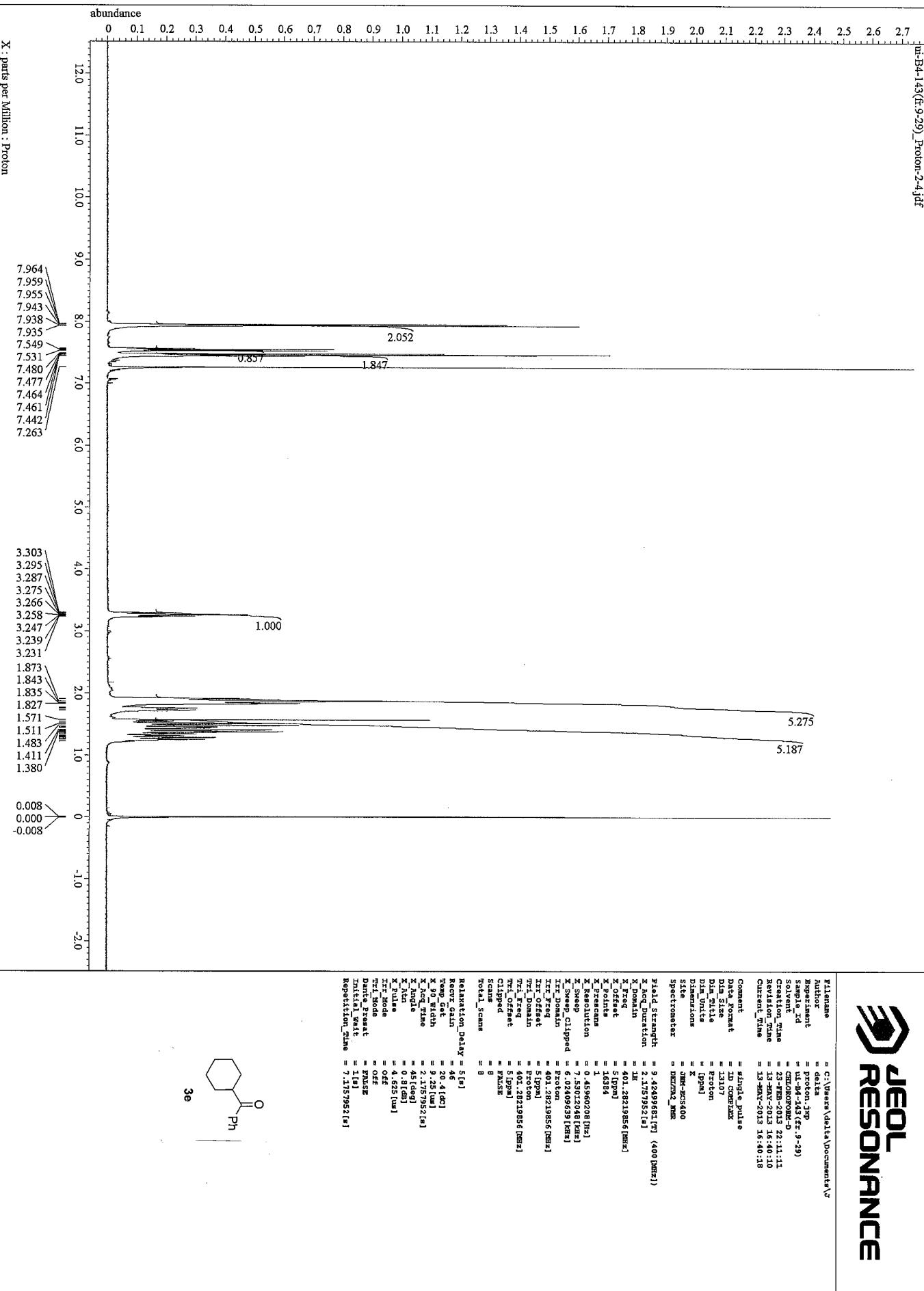
File name: = \Users\delta\Documents\J00
Author: = delta_JP
Sample Id: = ui-M1-17(f, 5-47) C
Solvent: = CHLOROFORM-D
Creation Time: = 27-APR-2013 17:01:53
Revision Time: = 8-MAY-2013 19:54:53
Current Time: = 8-MAY-2013 19:55:59

Comment: = single pulse decoupled gat
Data Format: = 1D spectrum
Dim Size: = 262144
Dim Units: = [PPM]
Dimensions: = X [PPM]
Spectrum: = JMR-EX400
Spectrometer: = JEOL-EX400
Pulse Strength: = 9.424996511[m] (400[PPM])
X_Scale[Duration: = 1.0308024[s]
X_Domain: = 130
X_Freq: = 100 [PPM]
X_Offset: = 100 [PPM]
X_Points: = 32768
X_Pulse: = 9
X_Probe: = 0.9430739[PPM]
X_Relaxation: = 31.5555551[PPM]
X_Sweep: = 25.282828281[PPM]
X_Sweep_Offset: = 40.12819856[PPM]
X_T1: = 5 [PPM]
X_T2: = PAUSE
Q1_Gated: = 256
Q1_Sums: = 256
Relaxation_Delay: = 2[s]
Recv_Gain: = 50
Recv_Gain_Offset: = 20.9 [deg]
X_90_Width: = 8.75 [us]
X_90_Width_Offset: = 1.0308024[s]
X_Angle: = 30.0 [deg]
X_Atm: = 5.2 [cm]
X_Elapsed: = 2.93666671 [us]
X_FID_Atm_Dic: = 22.6911 [db]
X_FID_Atm_Offset: = 22.6911 [db]
X_FID_Offset: = 0.0 [db]
X_FID_Width: = 0.15 [us]
Decoupling: = TRUE
Initial_Wait: = 1[s]
Mo_e_time: = TRUE
Mo_e_time: = 2[s]
Repetition_Time: = 3.0369024[s]

```



JEOL
RESONANCE



JEOL
RESONANCE

```

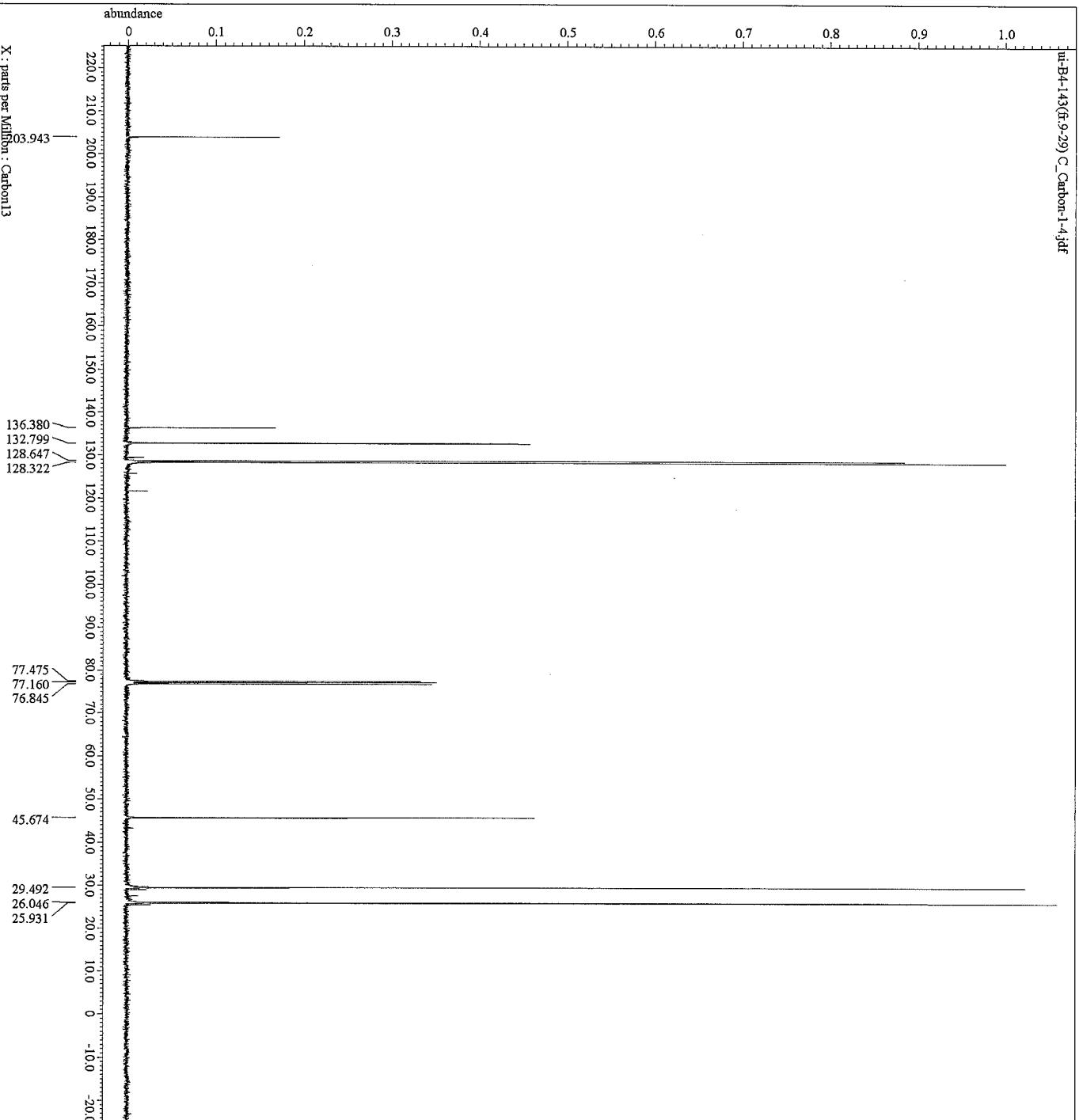
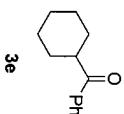
filename = C:\Users\deltaika\Documents\J
Author = Delta
Experiment = carbon.jdp
sample_id = UL-B4-143(f,9-29) C
solvent = CHLOROFORM-D
creation_time = 23-FEB-2013 21:42:12
revision_time = 13-MAY-2013 16:40:38
current_time = 13-MAY-2013 16:40:38

comment = single pulse decoupled gat
data_format = 1D COMPLEX
bin_size = 2614
dim_nucle = Carbon13
dim_units = [ppm]
dimensions = X
site = JMR-XECA00
spectrometer = DIEMR2_NMR

field_strength = 9.42499651 [T] (400 [MHz])
X_Avg_Portion = 1.0
X_Domain = 13C
X_Freq = 100.90247863 [MHz]
X_Offset = 100 [ppm]
X_Points = 32768
X_Precision = 4
X_Resolution = 0.96307519 [Hz]
X_Sweep = 31.5550557 [Hz]
X_Sweep_Clipped = 25.25252525 [Hz]
Xc_Domain = Proton
Xc_Freq = 401.28219856 [MHz]
Xc_Offset = 5 [ppm]
clipped = FALSE
scans = 256
total_scans = 256

relaxation_delay = 2 [s]
recvr_gain = 50
transc_Gain = 20.2 [dB]
X_B0_With = 8.75 [us]
X_B0_Rate = 1.03080024 [s]
X_Angle = 30 [deg]
X_Ann = 5.2 [mJ]
X_Pulse = 2.9166667 [us]
TR_ABDec = 22.6911 [us]
TR_AB2Dec = 22.6911 [us]
TE_Millis = 0.002 [ms]
TE_Width = 0.15 [ms]
Decimation = 1 [s]
Initial_Wait = 1 [s]
No_Gauss = 2 [s]
Repetition_Time = 3.03080024 [s]

```



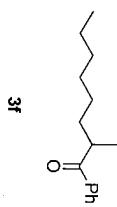
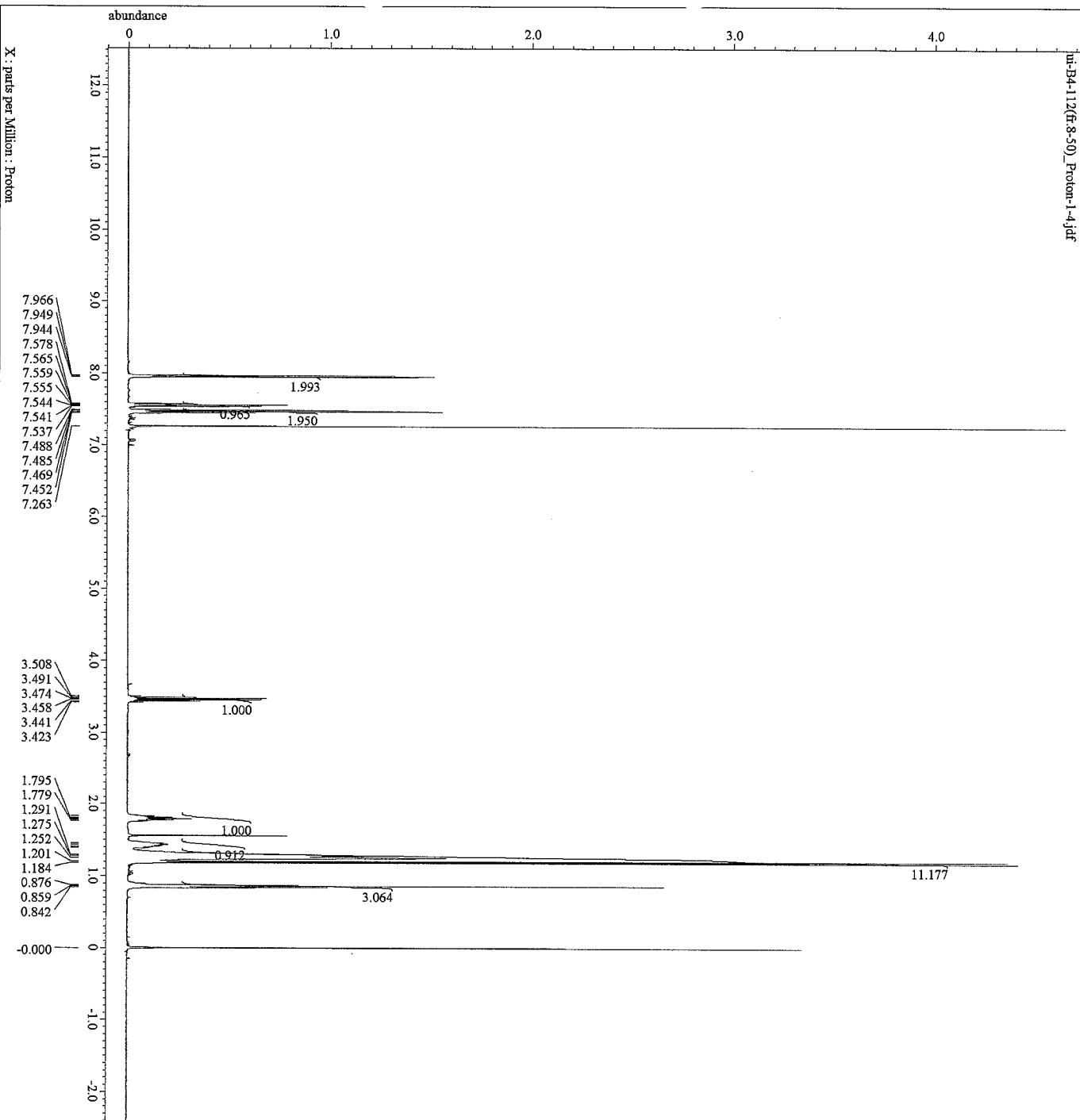
X : parts per Million : Carbon13

JEOL
RESONANCE

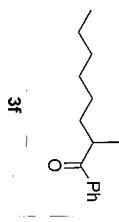
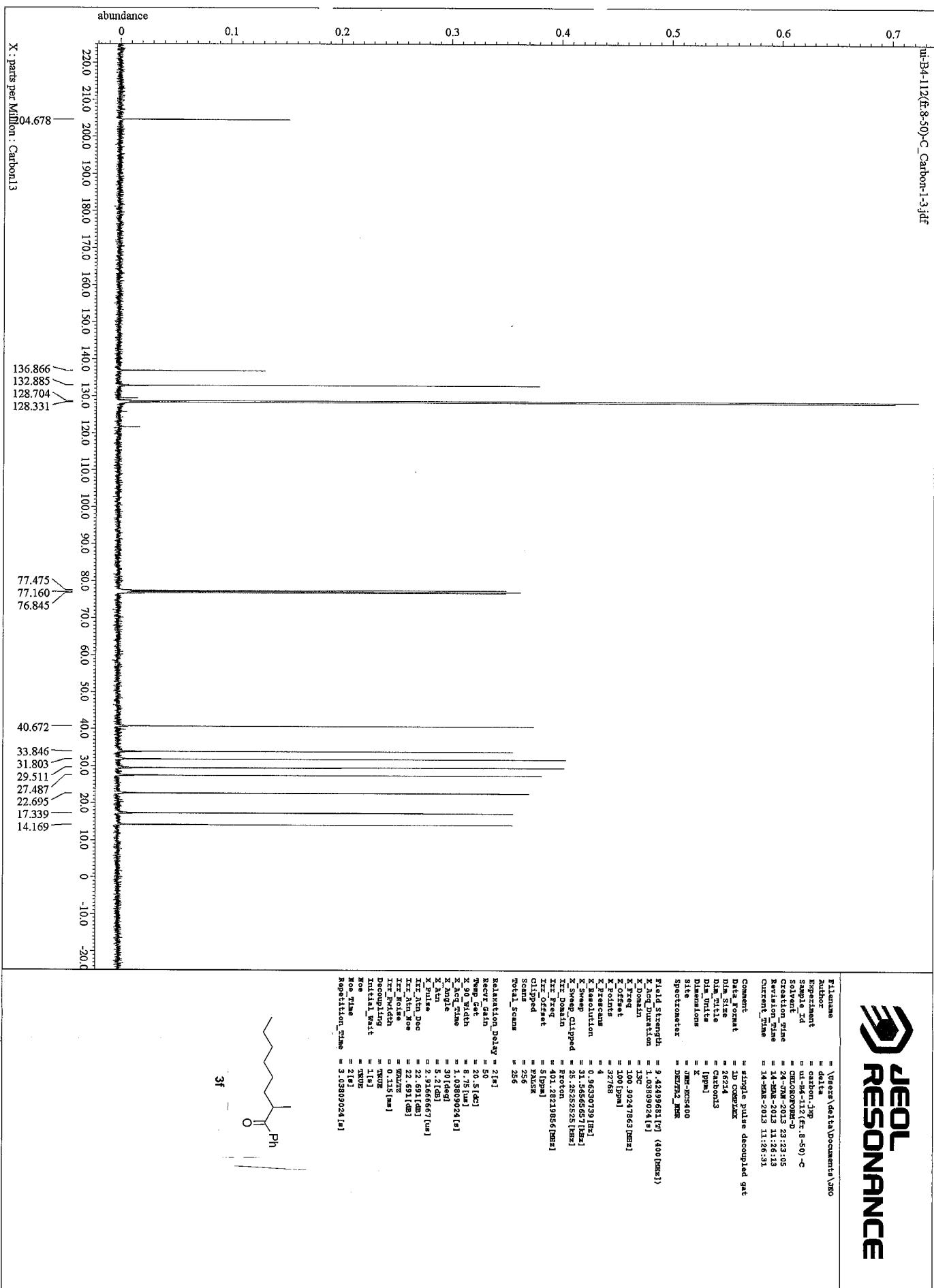
```

filename = "Unica\delta\ta\documents\100
Author = delta
Experiment = proton_jdp
Sample_Id = ui-B4-112(f-8-50)
Solvent = CDCl3
Creation_Time = 24-JUN-2013 18:03:43
Last_Modified = 14-SEP-2013 11:25:19
Current_Time = 14-SEP-2013 11:25:27
Comment = single_pulse
Data_Format = 1D_GATED
Data_Size = 13,070
Dim_Rule = proton
Dim_Units = [PPM]
Dimensions = 1
Instrument = JES-600A400
Spectrometer = INNOVA2_MER
Field_Strength = 9.42999611 [T] (400 [MHz])
X2_3Q_Duration = 2.73775161
X2_Domain = 1K
X_Freq = 401.38219856 [MHz]
X_Offset = 5184
X_Points = 2
X_Pulse = 0.45560208 [Hz]
X_Sweep = 1.3312018 [kHz]
X_Sweep_Clipped = 0.02408659 [kHz]
Xr_Domain = proton
Xr_Freq = 401.38219856 [MHz]
Xr_Offset = 5184
Xr_Domain = proton
Xr1_Freq = 401.38219856 [MHz]
Xr1_Offset = 5184
Xr1_Clipped = PAUSE
Scans = 16
Total_Scans = 16
Relaxation_Delay = 5 [s]
Recv_Gain = 48
Recv_Cet = 20.3 [sec]
X90_Witch = 9.35 [us]
X_Acq_Probe = 2.178795 [s]
X_Angle = 45 [deg]
X_Atn = 0.8 [us]
X_Full = 4.62 [us]
Xr_Mode = off
Trig_Mode = off
Dw_Preset = FALSE
Initial_Wait = 1 [s]
Repetition_Time = 7.1787952 [s]

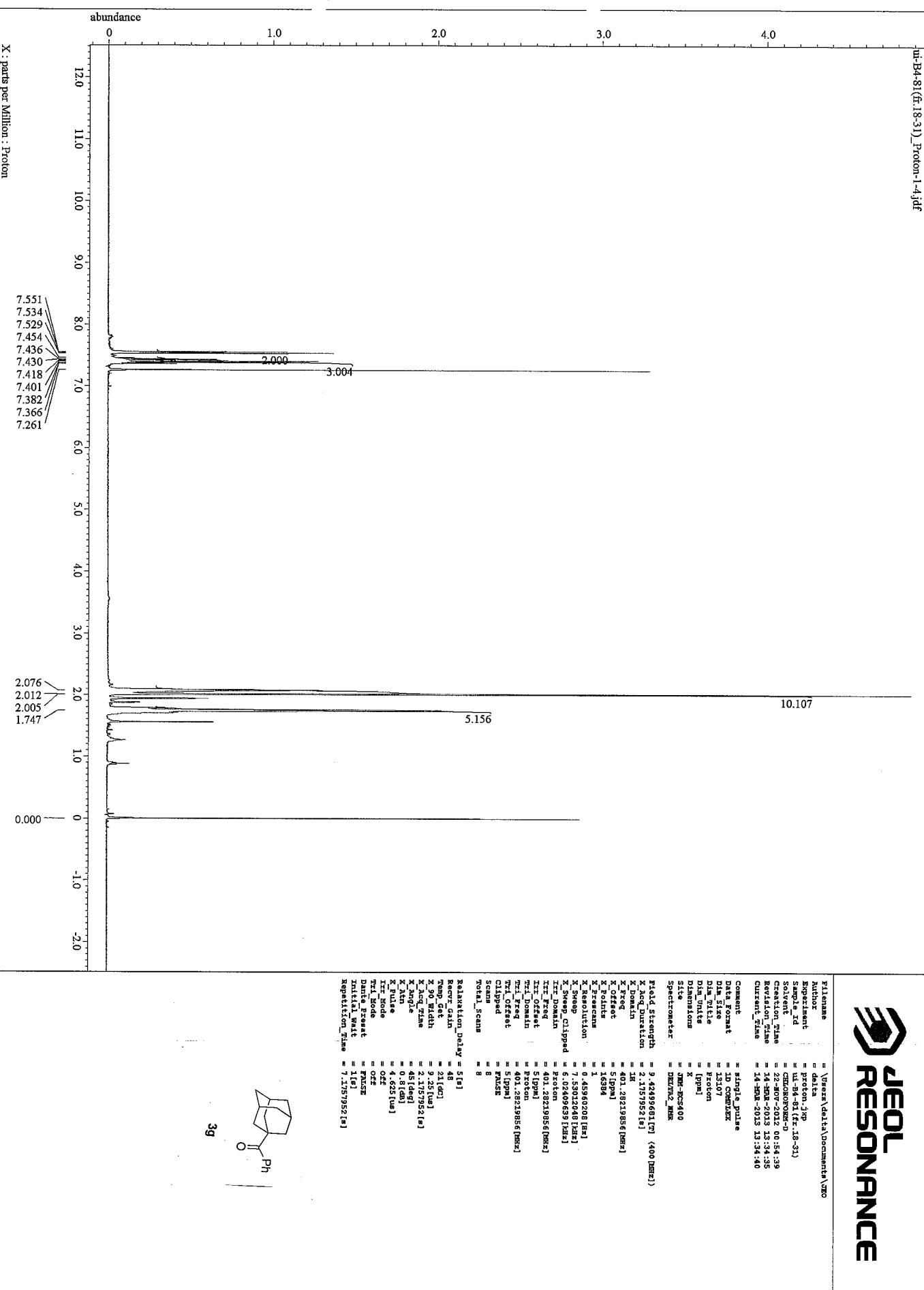
```



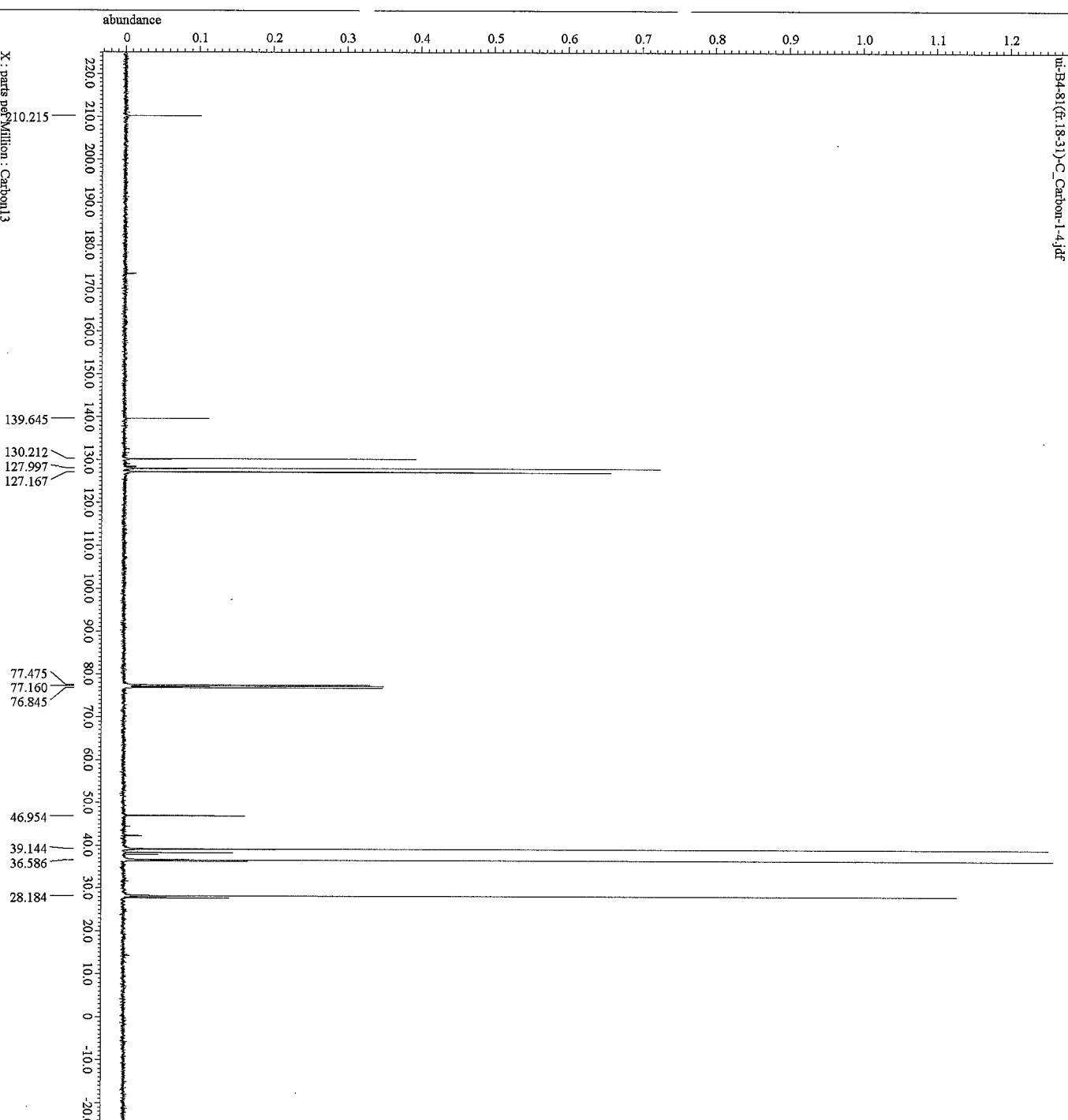
X : parts per Million : Proton.



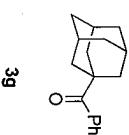
JEOL
RESONANCE



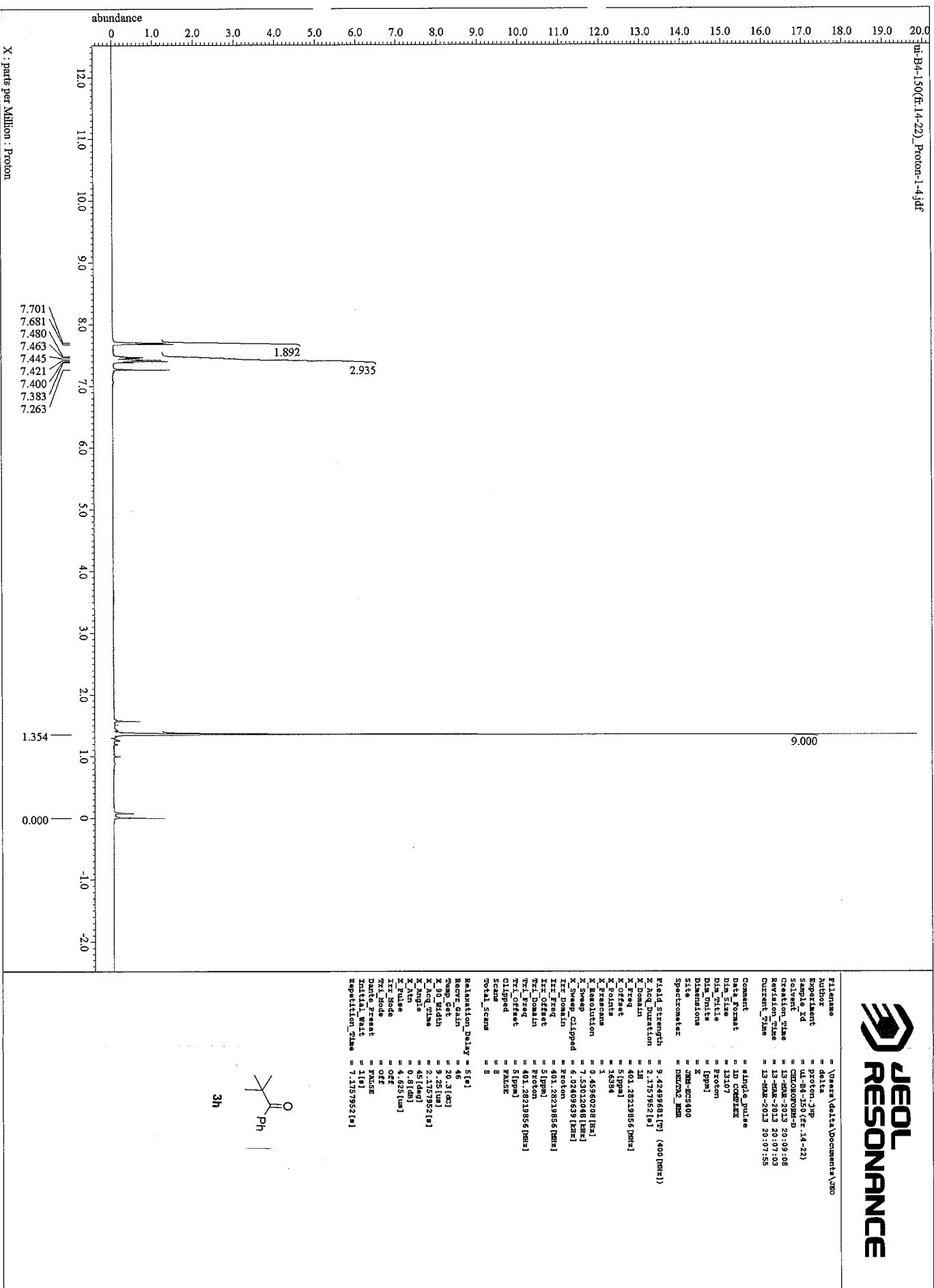
JEOL
RESONANCE



filename	= "User\dalton\documents\J00
Author	= delta
Experiment	= carbon_3p (fr.-31)-C
Sample_Id	= ui-B4-81(fr.-31)-C
Solvent	= CHCl3/CDCl3
Creation_Time	= 22-MAY-2012 01:10:13:35
Revision_Time	= 14-MAR-2013 13:35:36
Current_Time	= 14-MAR-2013 13:35:47
Comment	= single pulse decoupled gat
Data_Format	= IN_CHEM3D
DIM_Size	= 26214
DIM_Title	= Carbon13
DIM_Units	= [ppm]
DIMENSIONS	= X
site	= JMM-SCI400
Specrometer	= NEXUS2_WBZ
Field_Strength	= 9.42299651 [T] (400 [MHz])
X_Sweep_Duration	= 1.03600024 [s]
X_Domain	= 13C
X_Freq	= 100.00247863 [MHz]
X_Offset	= 100 [ppm]
X_Results	= 4.760
X_Resolutions	= 0.96320799 [ppm]
X_Sweep	= 21.56555557 [ppm]
X_Sweep_Clipped	= 25.24232525 [ppm]
IR_Domain	= Fixton
IR_Freq	= 401.29121896 [MHz]
IR_Offset	= 519ppm
QCPD	= FABZ
SCana	= 250
SCAL1_Scans	= 256
Relaxation_Delay	= 2 [s]
Recv_Gain	= 50
Temp_Set	= 21 [deg]
X90_Width	= 8.75 [us]
X_Acq_Pulse	= 1.03000024 [s]
X_Angle	= 30.0deg
X_Atn	= 5.21[deg]
X_Pulse	= 2.91666667 [us]
IR_Atn_Dec	= 22.6511[deg]
IR_Atn_Fee	= 22.6511[deg]
IR_Echo	= 512[us]
IR_Pulse	= 0.118 [us]
Decoupling	= TMS
Initial_Wait	= 1 [s]
Nois_Canc	= 32 [s]
Repetition_Rate	= 3.03600241 [s]



JEOL
RESONANCE



JEOL
RESONANCE

```

filename = \Users\delta\Documents\J00
author = delta
Reportment = carbon_3D
Sample_Id = ui-Ba-150(fr.14-22) C
Solvent = CDCl3/CH2Cl2-D
Creation_Time = 14-MAR-2013 07:34:27
Revision_Time = 14-MAR-2013 10:07:52
Current_Time = 14-MAR-2013 10:08:07

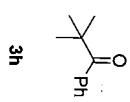
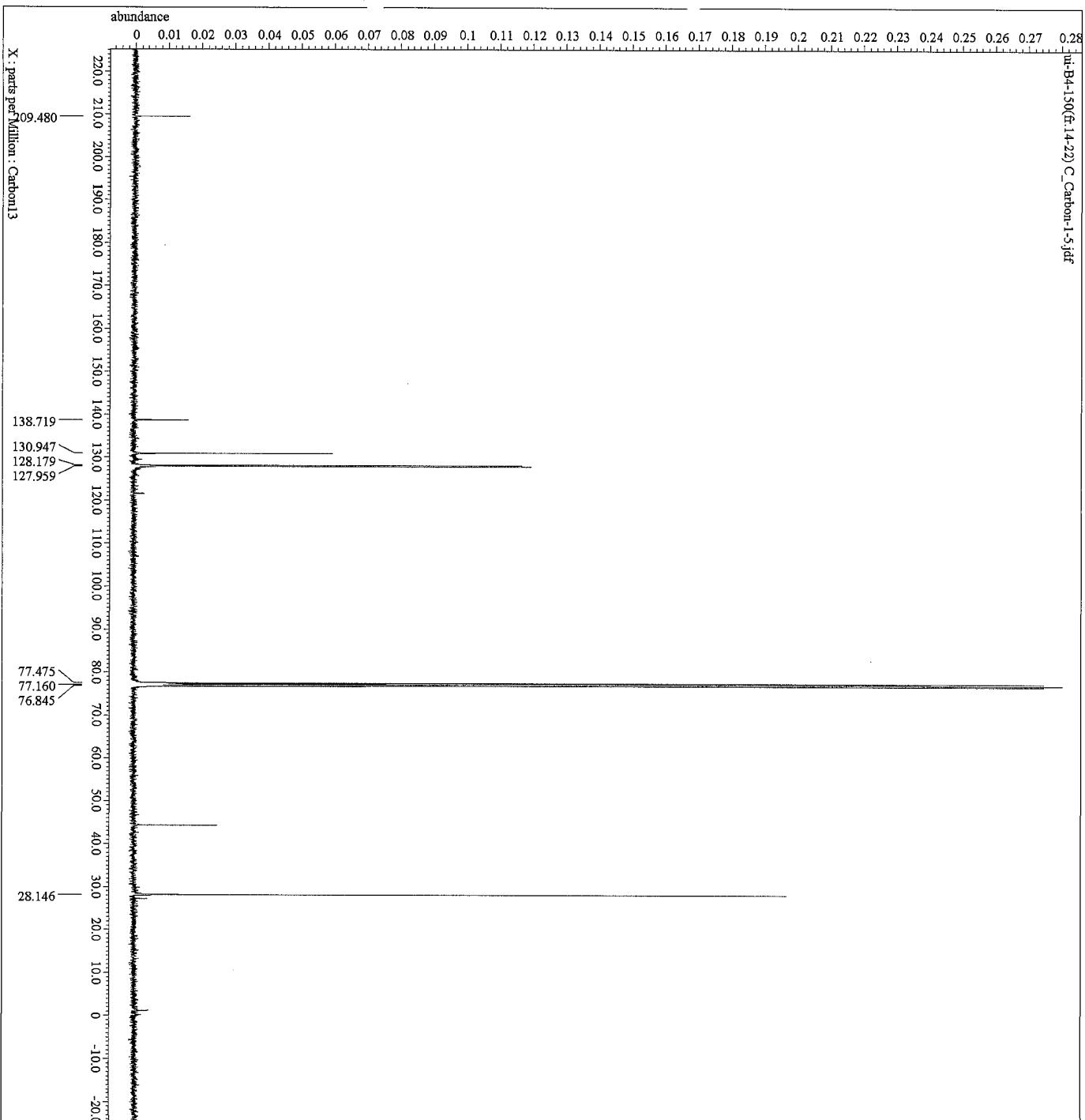
Comment = single pulse decoupled gat
Data_Format = 1D_COSYEX
NIM_Size = 26214
NIM_Tutis = Carbon13
NIM_Units = [PPM]
Dimensions = X,Y,Z
SpectrumID = DMR2_400
Spectrometer = DMR2_400

Field_Strength = 9.424996611 [T] (400 [MHz])
X_Duration = 1.0300024 [s]
X_Domain = 130
X_Offset = 100 [PPM]
X_Points = 32768

X_Scale = 1
X_Ascension = 9.6639079 [Hz]
X_Sweep = 31.555555 [PPM]
X_Sweep_Clipped = 25.22222225 [PPM]
X_FDomain = 401.2812856 [PPM]
X_Freq = 51 [PPM]
X_LOCKET = PAUSE
CLIPPED = 2056
CLIPEND = 2056
CLOCK_SOURC = 2056

Relaxation_Delay = 2 [s]
Recv_Gain = 50
Temp_Gain = 20.1 [dB]
X90_Width = 8.75 [us]
X90_GateTime = 1.0300024 [s]
XAngle = 30 [deg]
X_Alt = 5.2 [cm]
X_Pulse = 2.91666667 [us]
X_Alt_Dic = 22.691 [cm]
X_Alt_Rce = 22.691 [cm]
X_Raise = WHZ
X_Pitch = 0.115 [us]
Decoupling = TRUE
Initial_Wait = 1 [s]
No_TIME = TRUE
No_TUNE = 2 [s]
Repetition_Time = 3.0309024 [s]

```

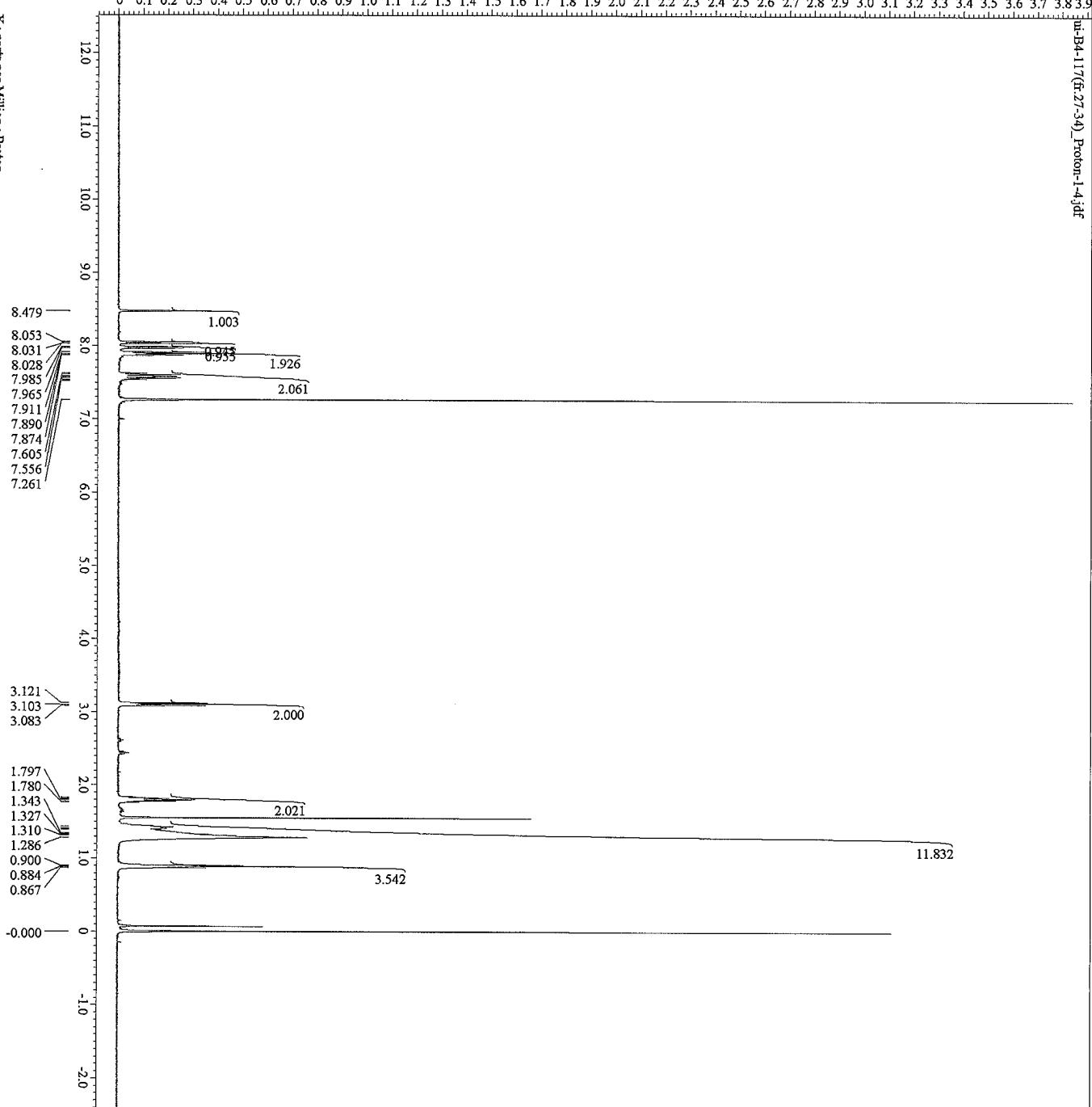
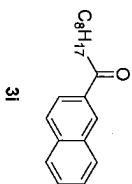


JEOL
RESONANCE

```

File name = \Users\deltaita\Documents\1.j00
Autosave = 0
Experiment = Proton_1D
Sample_id = ui-B4-117(fr-27-34)
Solvent = CDCl3/CD3OD/CD3OD
Creation_time = 31-JUN-2013 16:09:45
Last_modification = 14-MAR-2013 11:13:55
Current_time = 14-MAR-2013 11:13:55
Comment = single_pulse
Data_Format = 1D_Spectrum
Data_Scale = ppm
Pulse_Train = Proton
Data_Units = [ppm]
Dimensions = 1
Spectrum_ID = 00000000000000000000000000000000
SectionMarker = DATA02.REK
Field_Strength = 9.4299661 [T] (400 [MHz])
X_Avg_Duration = 2.1757952 [s]
X_Domain = 1H
X_Freq = 401.28219856 [MHz]
X_Offset = 5 [ppm]
X_Points = 16384
X_Precision = 1
X_Projection = 0.45569208 [Hz]
X_Sweep = 7.33012018 [Hz]
X_Sweep_Clipped = 6.02405659 [Hz]
Xr_Domain = Proton
Xr_Freq = 401.28219856 [MHz]
Xr_Offset = 5 [ppm]
Xr_Domain = Proton
Xr1_Freq = 401.28219856 [MHz]
Xr1_Offset = 5 [ppm]
Total_Scans = 16
Scans = 16
Relaxation_Delay = 5 [s]
Kevr_Gain = 52
Qewv_Gain = 20 [dB]
X90_width = 9.15 [us]
X_JEOL_name = 2.1757952 [s]
X_Angle = 45 [deg]
X_Alt = 0.8 [deg]
X_pulse = 4.62 [us]
Xr_Mode = off
Tri.Mode = off
Data_Preset = PAUSE
Initial_Wait = 1 [s]
Repetition_Rate = 7.1757952 [s]

```



JEOL
RESONANCE

```

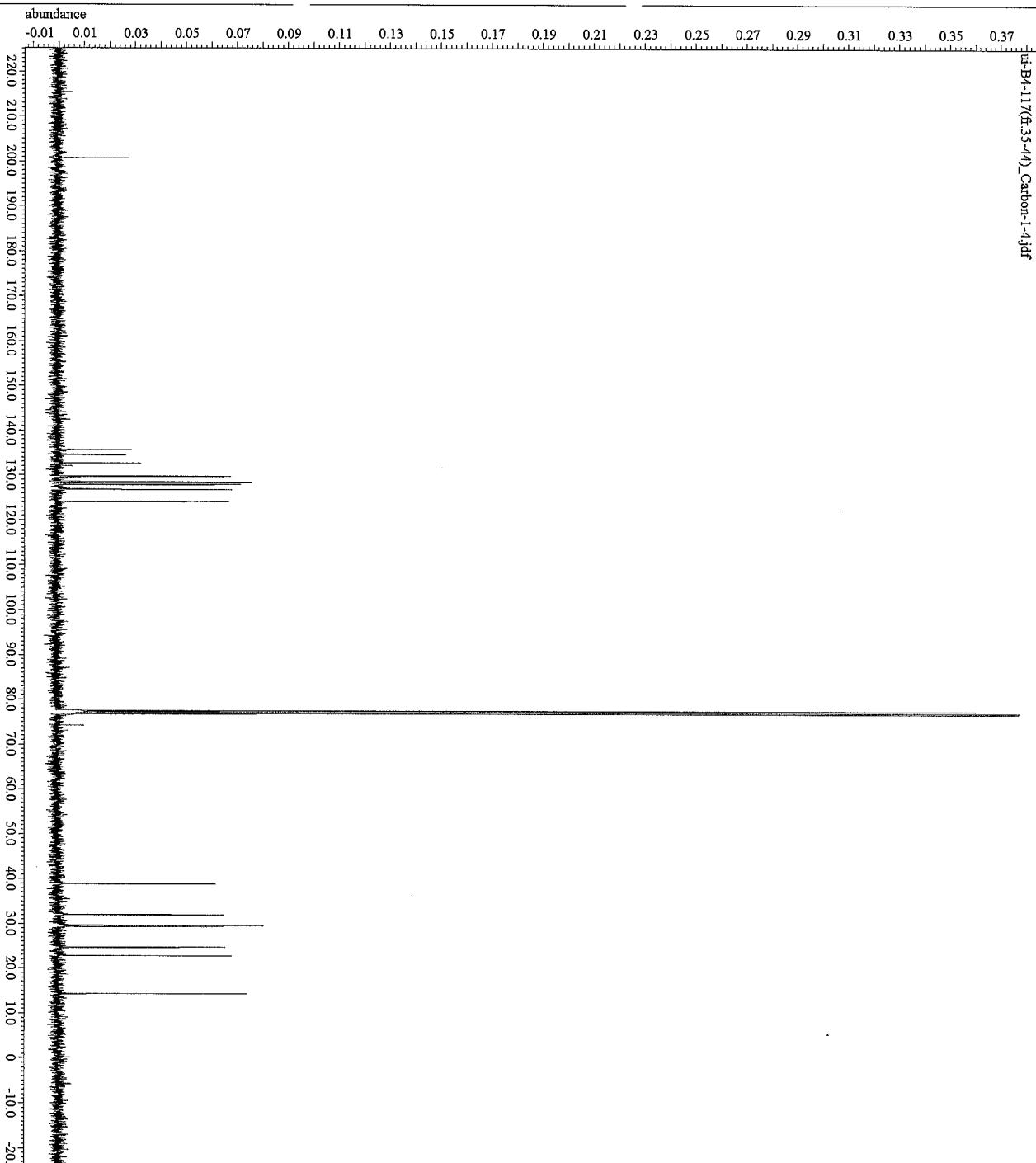
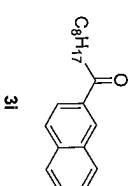
filename          = \Users\deltaite\Documents\JDP
Author            = delta
Experiment       = carbon_JDP
Sample_Id        = ui-B4-117(f.35-44)
Solvent          = CDCl3
Creation_Time   = 31-JUN-2013 18:07:05
Revision_Time   = 14-MAR-2013 11:13:32
Current_Time    = 14-MAR-2013 11:13:53

comment          = single pulse decoupled gpc
data_Format     = 1D complex
data_Size       = 26214
data_Tittle     = carbon13
data_Units      = [ppm]
dimensions      = 2
site             = JNM-GRX400
spectrometer    = JEOL2_MRS

Field_Strength   = 9.42998631 [T] (400 [MHz])
z_dcq_Duration = 1.0300004 [s]
z_dcq_Domain   = 132
z_Offset        = 100.90247863 [ppm]
z_Points        = 32760
z_Spectrum      = 4
z_Spinlock      = 4.9639779 [Hz]
z_Sweep          = 31.535555 [sec]
z_Sweep_clipped = 25.23232325 [sec]
zr_Domain       = 401..4913986 [ppm]
zr_Freq          = 51 [ppm]
zr_Offset        = 1.0300004
zr_Scale         = 250
zr_Total_Scans  = 256

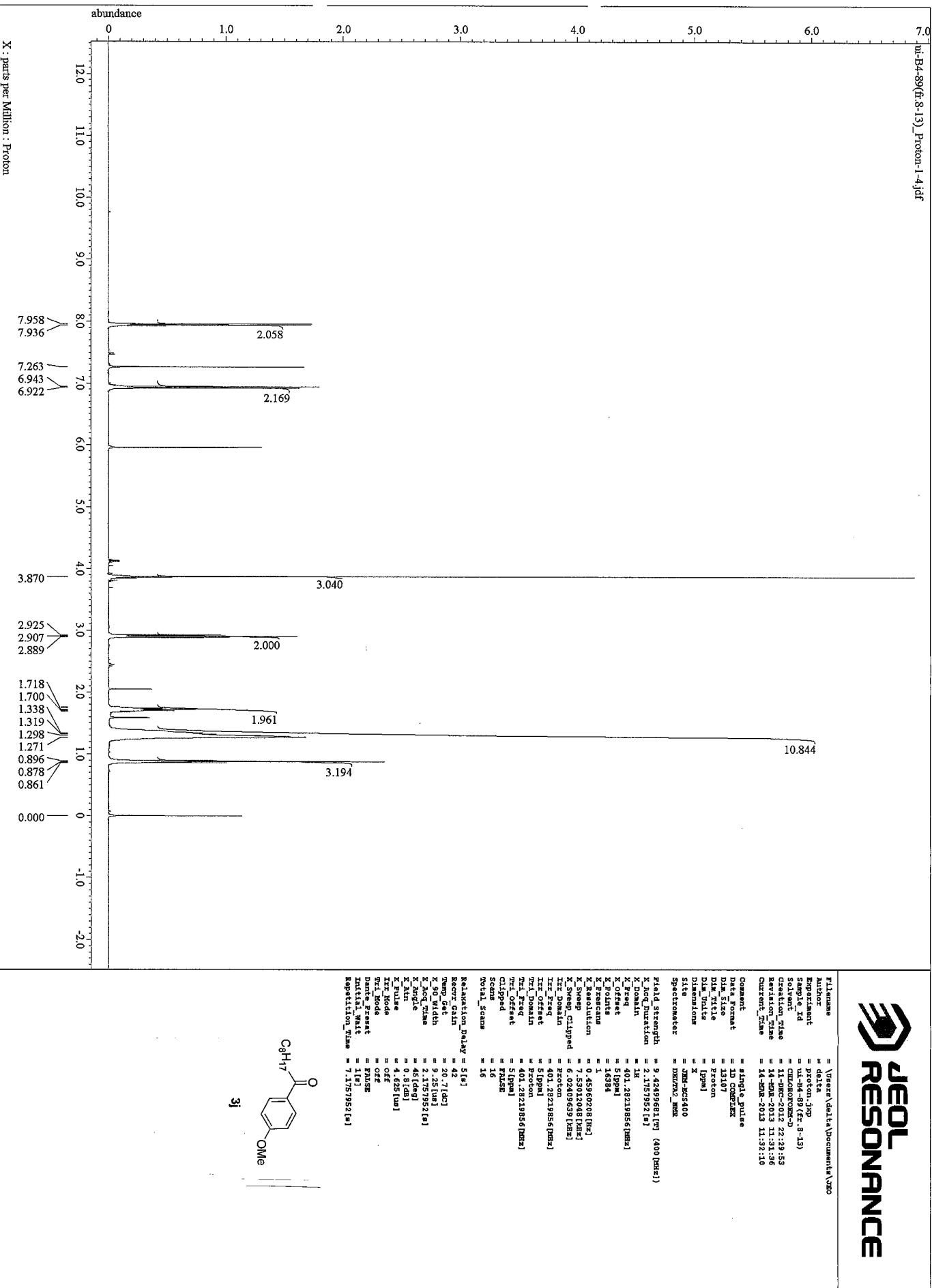
relaxation_Delay = 2 [s]
recvr_Gain       = 50
temp_Set         = 20.3 [deg]
temp_13C         = 6.75 [um]
z_dcq_Domain   = 1.03000024 [s]
z_dcq_Offset    = 30 [deg]
z_Alt            = 5.2 [deg]
z_Pulse          = 2.91666667 [us]
IR_Alt_Dec      = 22.651 [db]
IR_Alt_Ice      = 22.651 [db]
IR_Reliqe       = WALTZ
IR_Pw13          = 0.15 [us]
Decoupling       = TONE
Initial_Wait    = 1 [s]
Rce_Tune         = TONE
Rce_Time         = 2 [s]
Kepotition_Nise = 3.0380924 [s]

```

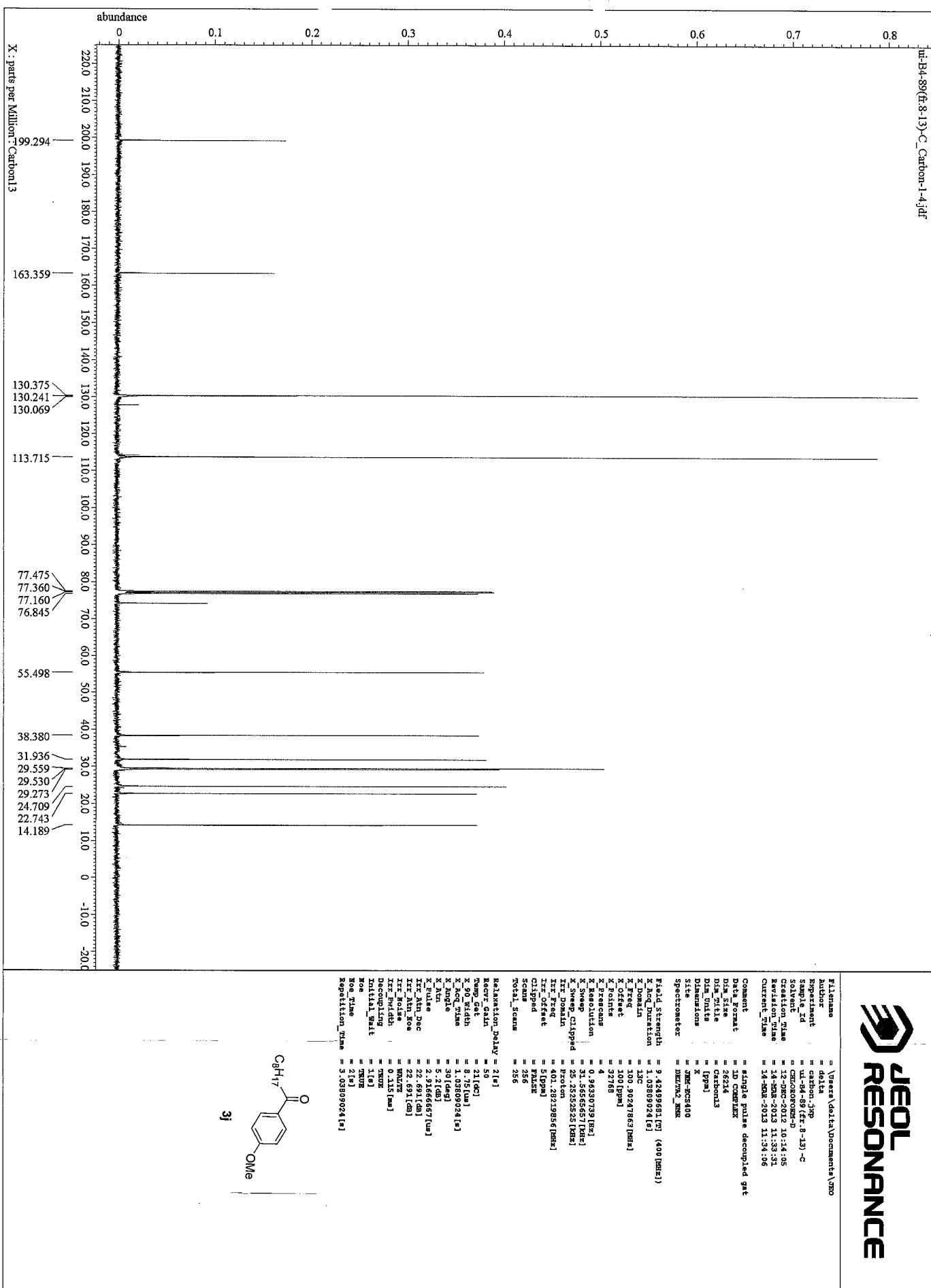


X : parts per Million: Carbon13
200.764

JEOL
RESONANCE



JEOL
RESONANCE

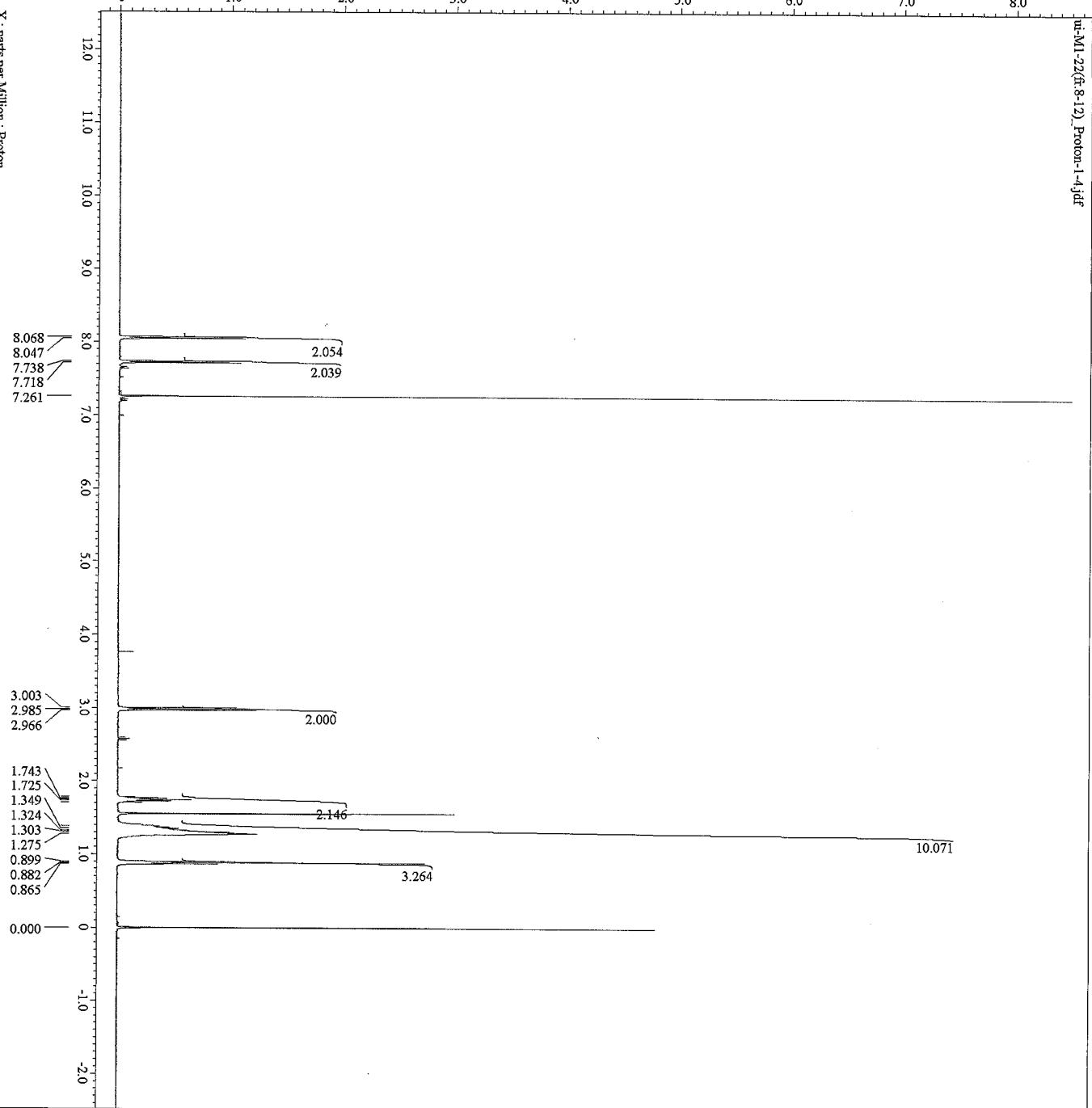
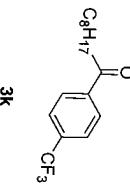


JEOL
RESONANCE

```

File_name = "Users\delta\Documents\JRC
Author = delta
Experiment = proton-JDP
Sample_id = ui-M1-22(f-8-12)
Solvent = CHLOROFORM-D
Creation_time = 10-MAY-2013 23:28:37
Revision_time = 11-MAY-2013 00:42:14
          = 11-MAY-2013 00:42:28
Comment = single_pulse
Data_Format = 1D_COMPLEX
Dim_Size = 13107
Dim_Ratio = Proton
Dim_Units = [PPM]
Dimensions = X
Site = JMS-EC3400
Spectrometer = INCOGNITO_NMR
Field_Strength = 9.42499651[MHz] (400[MHz])
X_Acq_Duration = 2.1757952[sec]
X_Domain = IH
X_Freq = 401.28219856[MHz]
X_Offset = 5[ppm]
X_Points = 16384
X_Precision = 1
X_Resolution = 0.4596208[Hz]
X_Sweep = 7.53012048[MHz]
X_Sweep_Clipped = 7.53012048[MHz]
Xr_Domain = proton
Xr_Freq = 401.28219856[MHz]
Xr_Offset = 5[ppm]
Xr_Domain = Proton
Xr_Freq = 401.28219856[MHz]
Xr_Offset = 5[ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5[sec]
Rever_Gain = 50
Temp_Get = 21.31[deg]
X_3D_Width = 9.25[un]
X_Acc_Runs = 2.1757952[sec]
X_Angle = 45[deg]
X_Btn = 0.8[deg]
X_Pulse = 4.625[us]
Tr1_Mode = off
Tr1_Offset = off
Dante_Preset = FALSE
Initial_Wait = 1[sec]
Repetition_Time = 7.1757952[sec]

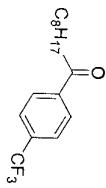
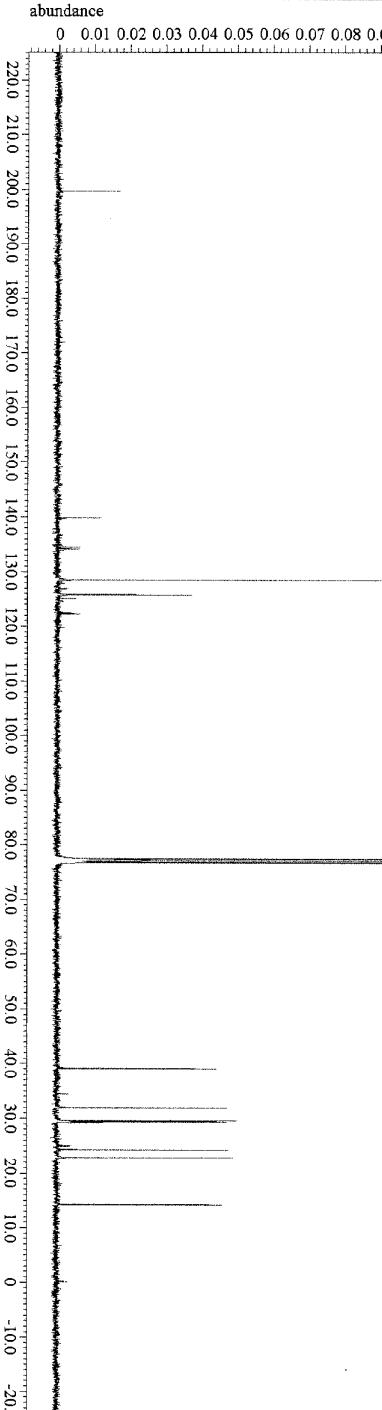
```



X : parts per Million : Proton

JEOL
RESONANCE

File name = \User\deltaelta\documents\jno
 Author = delta
 Experiment = carbon.jdp
 Sample_Id = 4-trifluoromethyl-phenyl_o
 Solvent = CHLOROFORM-D
 Creation Time = 15-MAY-2013 11:49:21
 Revision Time = 15-MAY-2013 14:25:56
 Current Time = 15-MAY-2013 14:26:26
 Comment = single pulse decoupled gat
 Data Format = 1D COMPLEX
 Dim Size = 26214
 Dim Title = carbon13
 Dim Units = [ppm]
 Dimensions = X
 Site = JMS-ECX400
 Spectrometer = DEPTA2_MM
 Field Strength = 9.42499561[MHz] (400 [MHz])
 X_Acc_Duration = 26214
 X_Domain = 1-3C
 X_Freq = 100.9024763 [MHz]
 X_Offset = 300 [ppm]
 X_Quants = 32768
 X_Tracers = 4
 X_Resolution = 0.96330739 [ppm]
 X_Sweep = 31.4656657 [ppm]
 X_Sweep_Clipped = 22.2555525 [ppm]
 Irr_Domain = Proton
 Irr_Freq = 401.20219856 [MHz]
 Irr_Offset = 5 [ppm]
 Clipped = FALSE
 Scans = 2048
 Total_Scans = 2048
 Relaxation_Delay = 2 [s]
 Preer_Gain = 50
 New_Get = 21.1 [deg]
 X_90_Width = 8.75 [us]
 X_Irr_Freq = 1.008059524 [Hz]
 X_Irr_Pulse = 30 [deg]
 X_Irr_Atn = 5.2 [deg]
 X_Irr_Atn_Dec = 2.94666676 [deg]
 X_Irr_Atn_Pow = 22.931 [dB]
 X_Irr_Noise = 0.002
 X_Irr_Pw0th = 0.115 [ns]
 Decoupling = 200K
 Initial_Wait = 1 [s]
 Noe_Mix = 2 [s]
 Noe_Mixing = 3.03809024 [s]



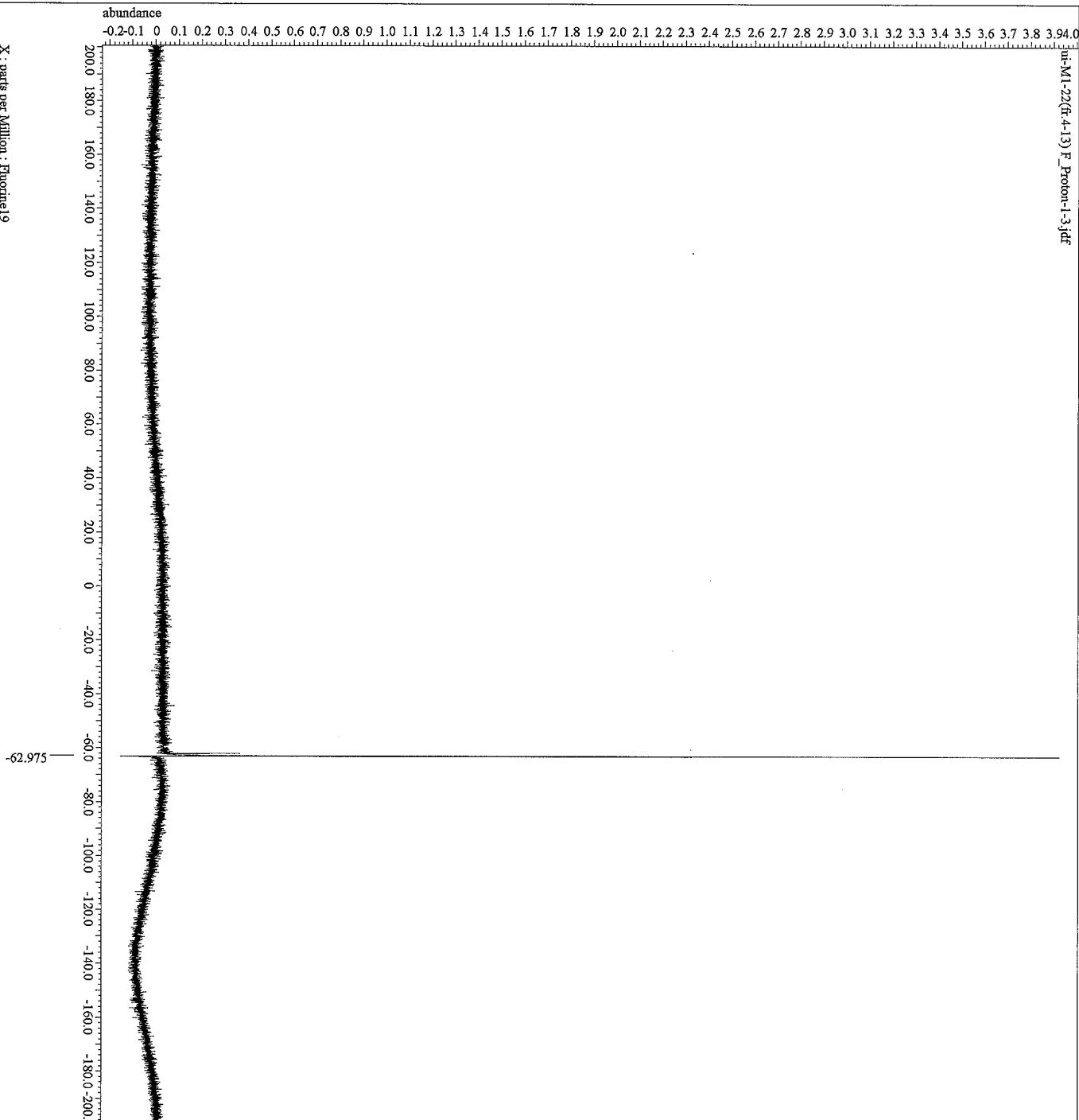
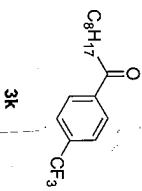
3k

JEOL
RESONANCE

```

filename = "Unes\datta\Documents\JED
Author = delta
Experiment = proton.JDP
Sample_Id = u1-M1-22(f,-13).F
Solvent = chloroform-D
Creation_Time = 13-May-2013 16:29:48
Revision_Time = 13-May-2013 16:26:56
Current_Time = 13-May-2013 16:26:56
Comment = single pulse
Data_Format = 3D_COMPLEX
Dim_Size = 13107
Dim_Tittle = Fluorine19
Dim_Units = [ppm]
Dimensions = X
Site = TMS-203400
Spectrometer = NMR2_MER
Field_Strength = 9.42499631[MHz] (400[ppm])
X_Avg_Duration = 86.50752[sec]
X_Domain = 139
X_Freq = 377.58246406[ppm]
X_Offset = 0 [ppm]
X_Points = 16384
X_Precision = 1
X_Resolution = 11.55968868[Hz]
X_Sweep = 159.39999999[Hz]
X_Sweep_Clipped = 151.5181812[Hz]
X_Domain = Fluorine19
X_Freq = 377.58246406[ppm]
X_Offset = 5 [ppm]
X_Domain = Fluorine19
X_Freq = 377.58246406[ppm]
X_Offset = FALSE
clipped = FALSE
scans = 8
Total_Scans = 8
Relaxation_Delay = 5[s]
Recv_Gain = 50
Tppg_Gain = 20.9[db]
X_SQ_With = 10.75[us]
X_Avg_Runs = 86.50752[sec]
X_Amplitude = 45[deg]
X_Am = 2[deg]
X_Pulse = 5.375[us]
IR_Mode = off
Pulse_Preset = FALSE
Initial_Wait = 1[s]
Repetition_Time = 5.0850752[s]

```

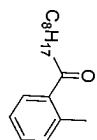


JEOL
RESONANCE

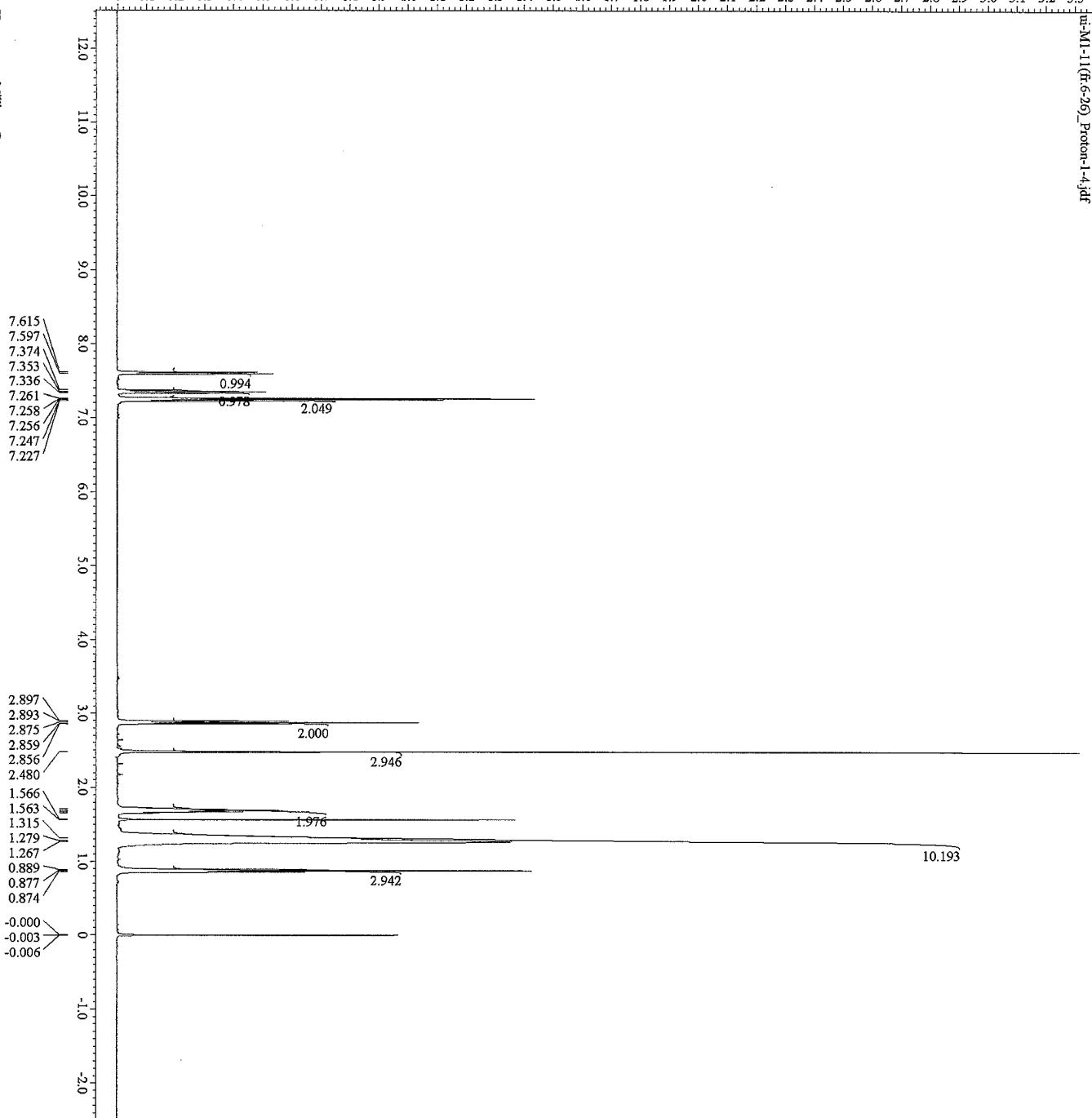
```

File Name = C:\Users\delta\Documents\J
Author = delta
Experiment = proton.jdp
Sample Id = u1-s1-11(Gr-6-6)
Solvent = CHLOROFORM-D
Creation Time = 19-APR-2013 20:53:26
Revision Time = 14-MAY-2013 20:22:55
Current Time = 14-MAY-2013 20:22:41
Comment = single_pulse
Data Format = 1D COMPLEX
Dim Size = 13107
Dim Nuclei = Proton
Dim Units = [ppm]
Dimensions = X
Site = JME-NCS400
Spectrometer = DEPTM2_NMR
Field Strength = 9.42499611[MHz] (400 MHz)
X-Accq_Direction = 1H
X-domain = 1H
X_Offset = 401.2821856[MHz]
X_Points = 51[ppm]
X_Prescans = 1
X_Resolution = 0.45960208[Hz]
X_Sweep = 7.53012048[Hz]
X_Sweep_Clipped = 6.02409659[Hz]
Xr_Domain = Proton
Xr_Freq = 401.2821856[MHz]
Xr_Offset = 5[ppm]
Tr1_Domain = Proton
Tr1_Freq = 401.2821856[MHz]
Tr1_Offset = 5[ppm]
Clipped = FALSE
Scans = 16
Total_scans = 16
Relaxation_Delay = 5[e]
Revol_Gain = 44
Temp_Get = 20.1[deg]
X_00_width = 9.25[us]
X_180q_trms = 2.1757952[e]
X_Angle = 45[deg]
X_Btn = 0.8[deg]
X_Pulse = 4.625[us]
IR_Mode = off
Tr1_Mode = off
Data_Preset = FALSE
Initial_Wait = 1[e]
Repetition_Time = 7.1757952[e]

```



31



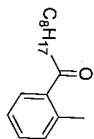
JEOL
RESONANCE

```

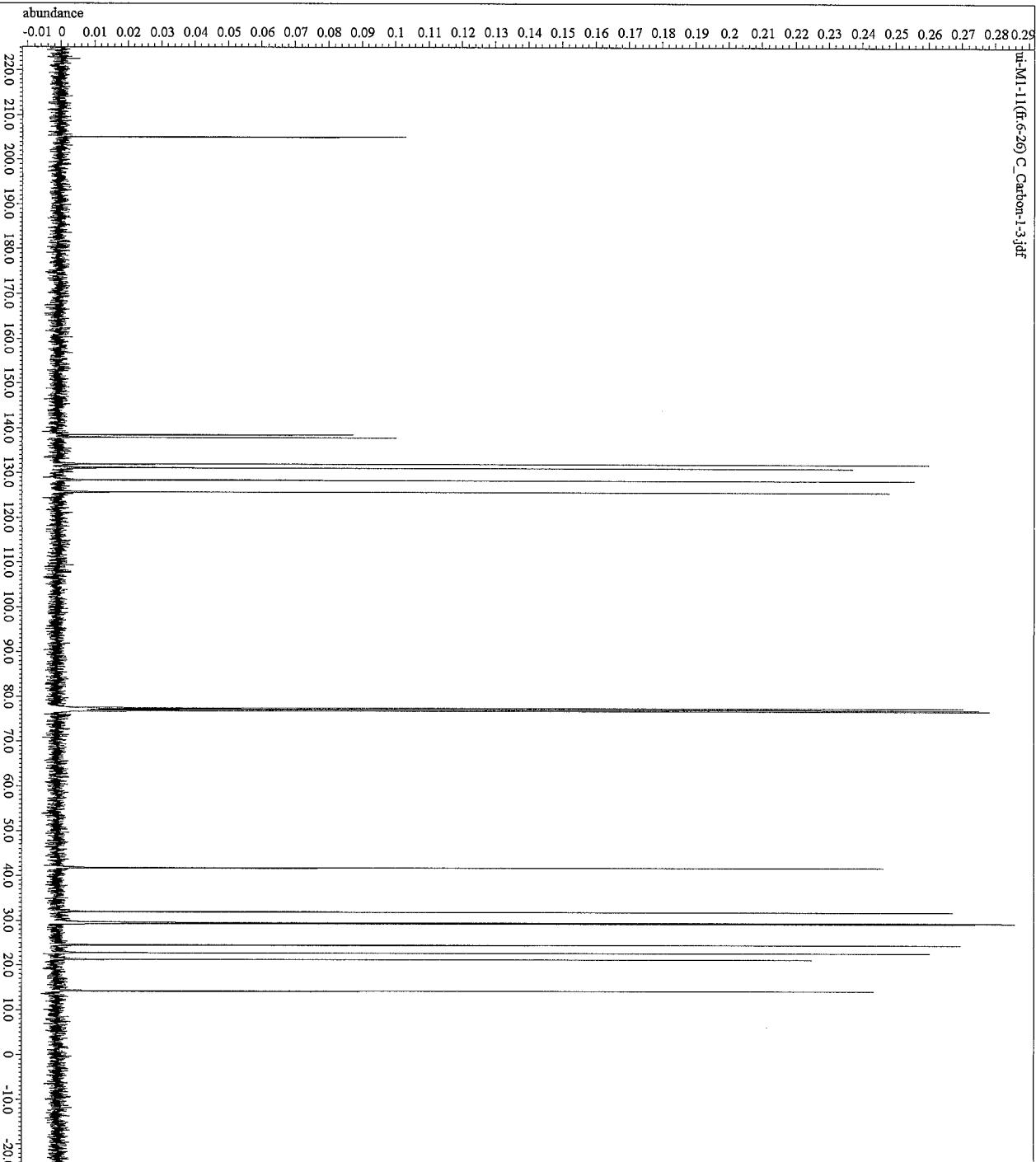
File Name          = C:\Users\delta\Documents\J
Author            = delta
Experiment        = carbon.jdp
Sample_Id         = ui-111-11fr-6-6) C
Solvent           = CHLOROFORM-D
Creation_Time    = 19-AUG-2013 21:06:14
Revision_Time    = 14-SEPT-2013 20:20:23
Current_Time     = 14-SEPT-2013 20:20:27

Comment          = single pulse decoupled gat
Data_Format      = 1D COMPLEX
Dim_Size          = 2614
Dim_Rule          = Carbon13
Dim_Units          = [ppm]
Dimensions        = X
Site              = JNM-ECX400
Spectrometer     = DEUTRON NMR
Field_Strength   = 9.42499601[T] (400[MHz])
XAccq_Direction = 1.03809024[us]
XDomain          = 13C
X_Treq            = 100 [ppm]
X_Offset          = 32768
X_Points          = 4
X_Prescans       = 0.96330739[us]
X_Resolution     = 31.5555657[us]
X_Sweep           = 25.25252525[us]
Xr_Domain         = proton
Xr_Freq           = 401.28239856[MHz]
Xr_Offset         = 5[ppm]
Clipped          = FALSE
Scans             = 256
Total_scans      = 256
Relaxation_Delay = 2[us]
Reevr_Gain        = 50
Temp_Get          = 20.7[deg]
X_90_Width        = 8.75[us]
X_Accq_Time       = 1.03809024[us]
X_Angle           = 30.0[deg]
X_Probe           = 5.2[db]
X_Rfume           = 2.93666667[us]
Xr_Atm_Dec        = 22.691[db]
Xr_Atm_Iso        = 22.691[db]
Xr_FdWidth        = 10000
Decoupling        = TRUE
Initial_Wait      = 1[us]
Nose_time         = 2[us]
Nose_time         = 2[us]
Repetition_Time   = 3.03809024[us]

```

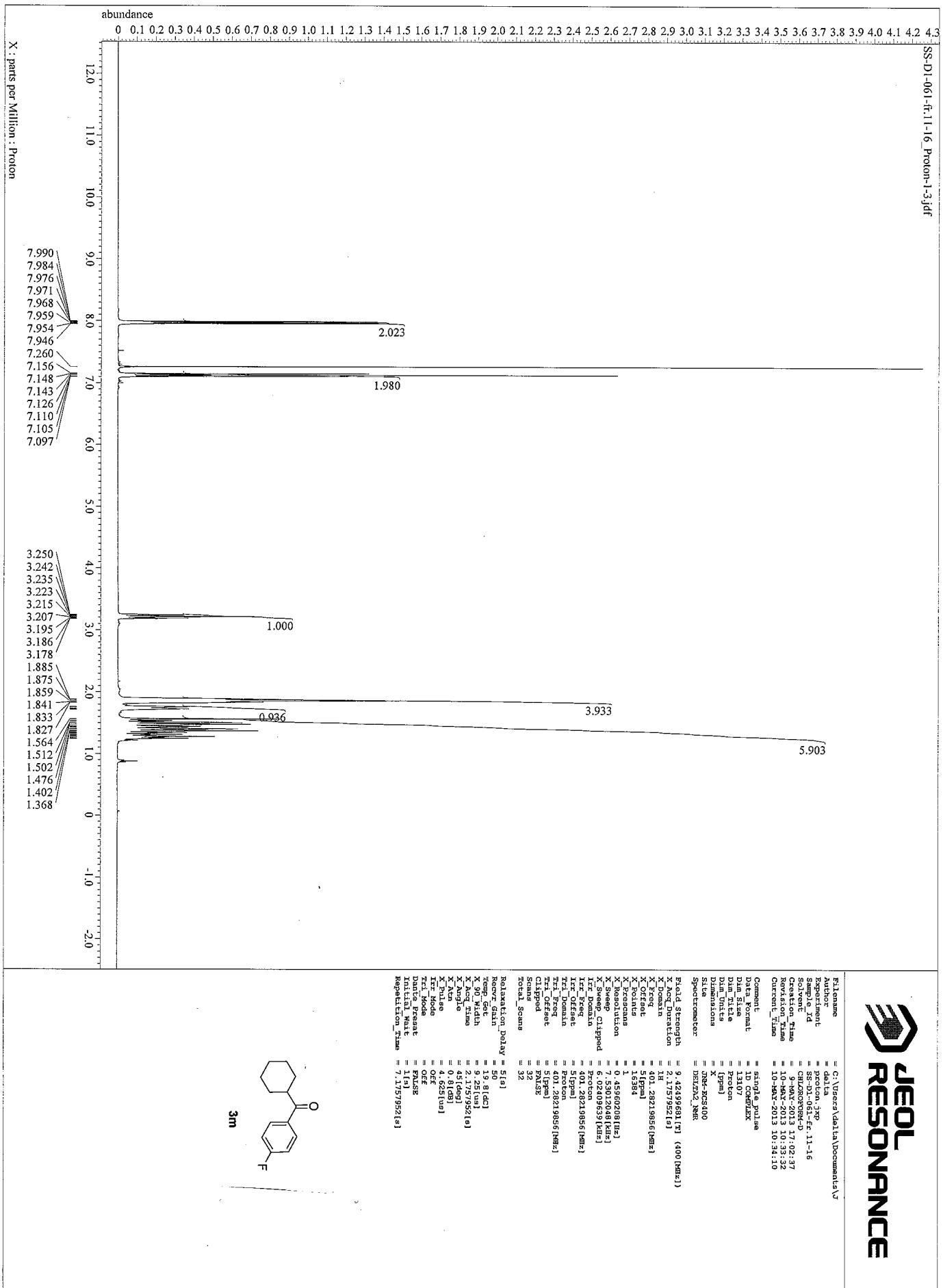


31

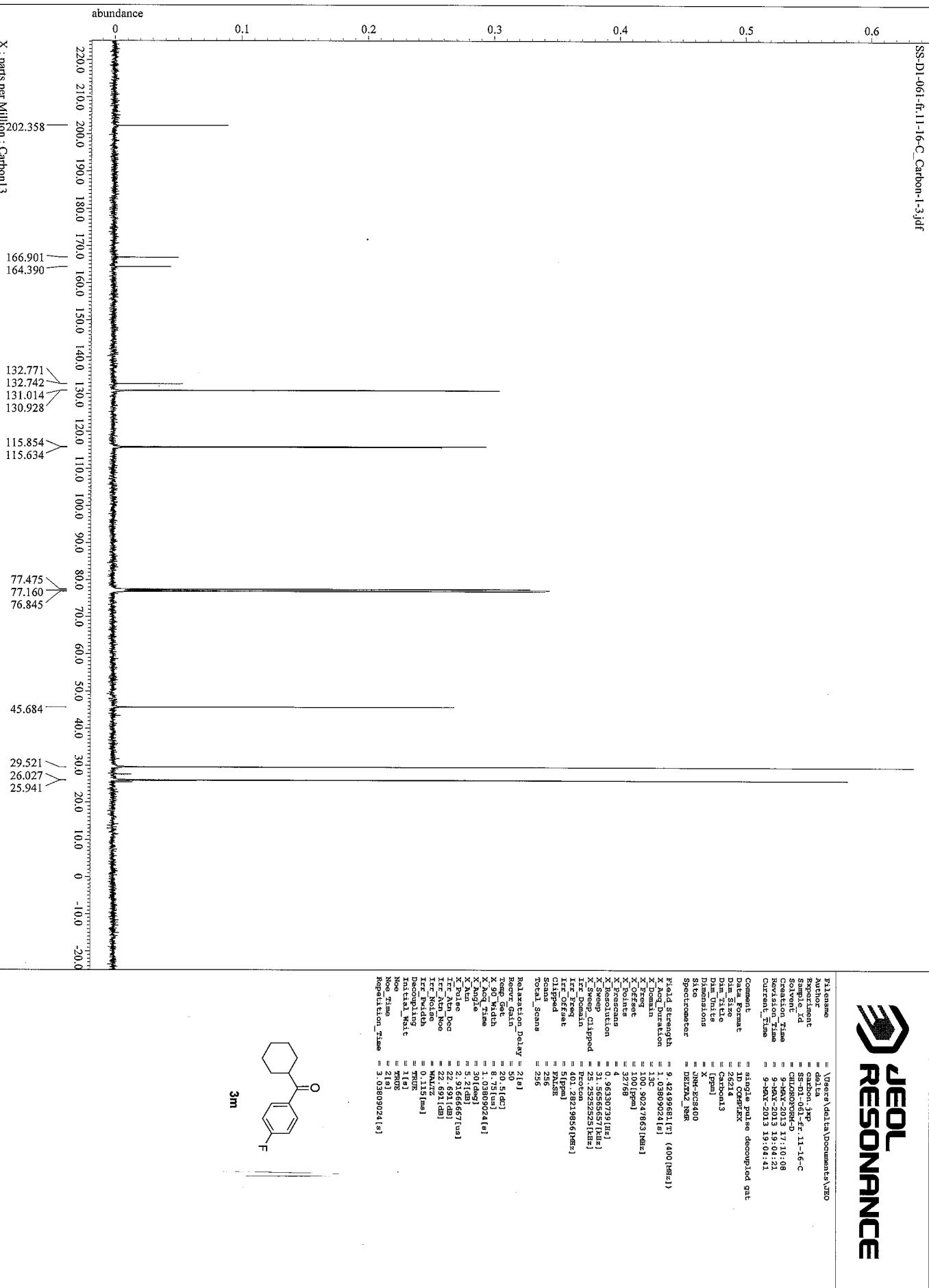


X : parts per Million : Carbon13

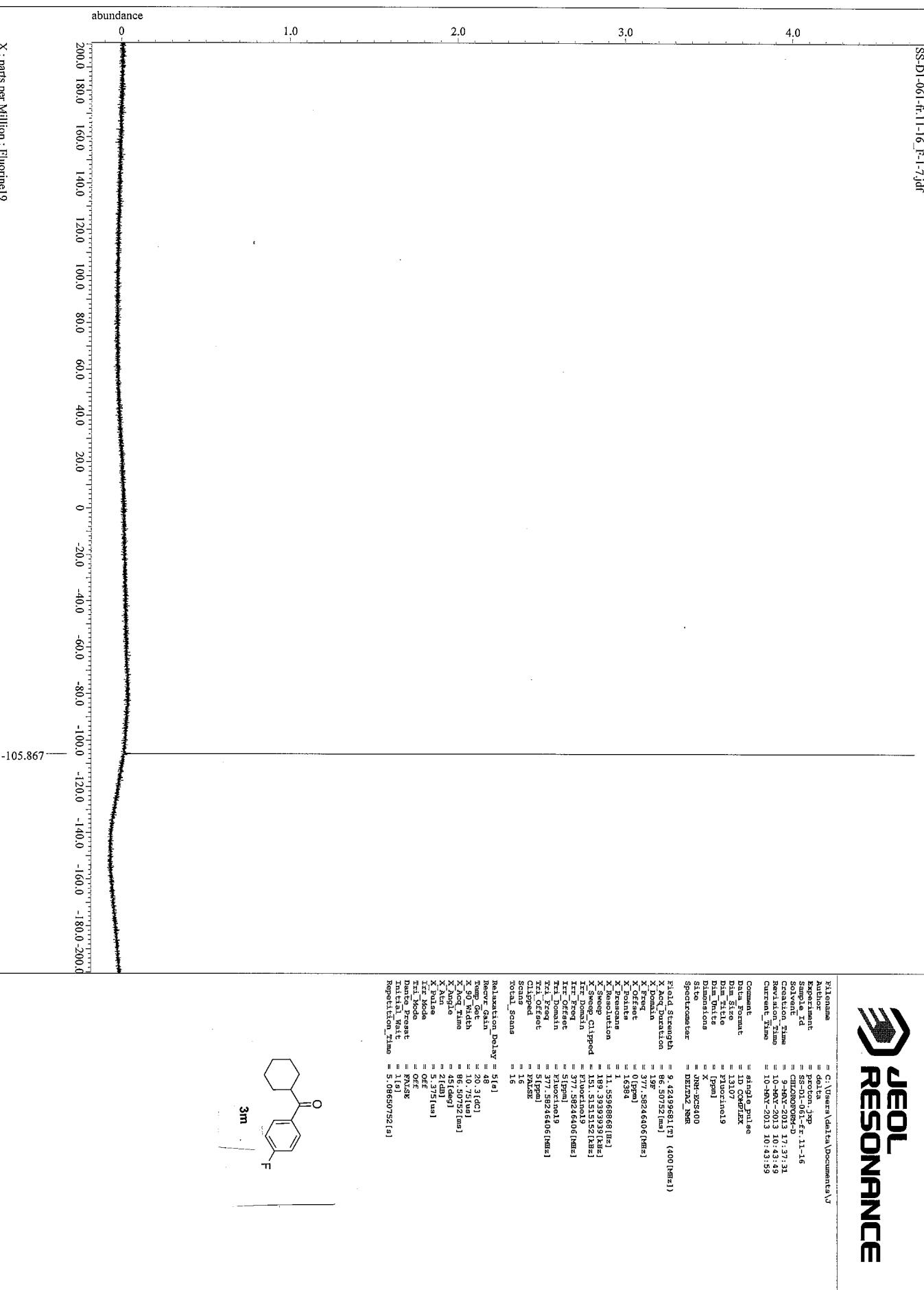
JEOL
RESONANCE



JEOL
RESONANCE



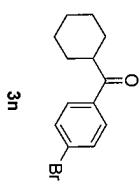
JEOL
RESONANCE



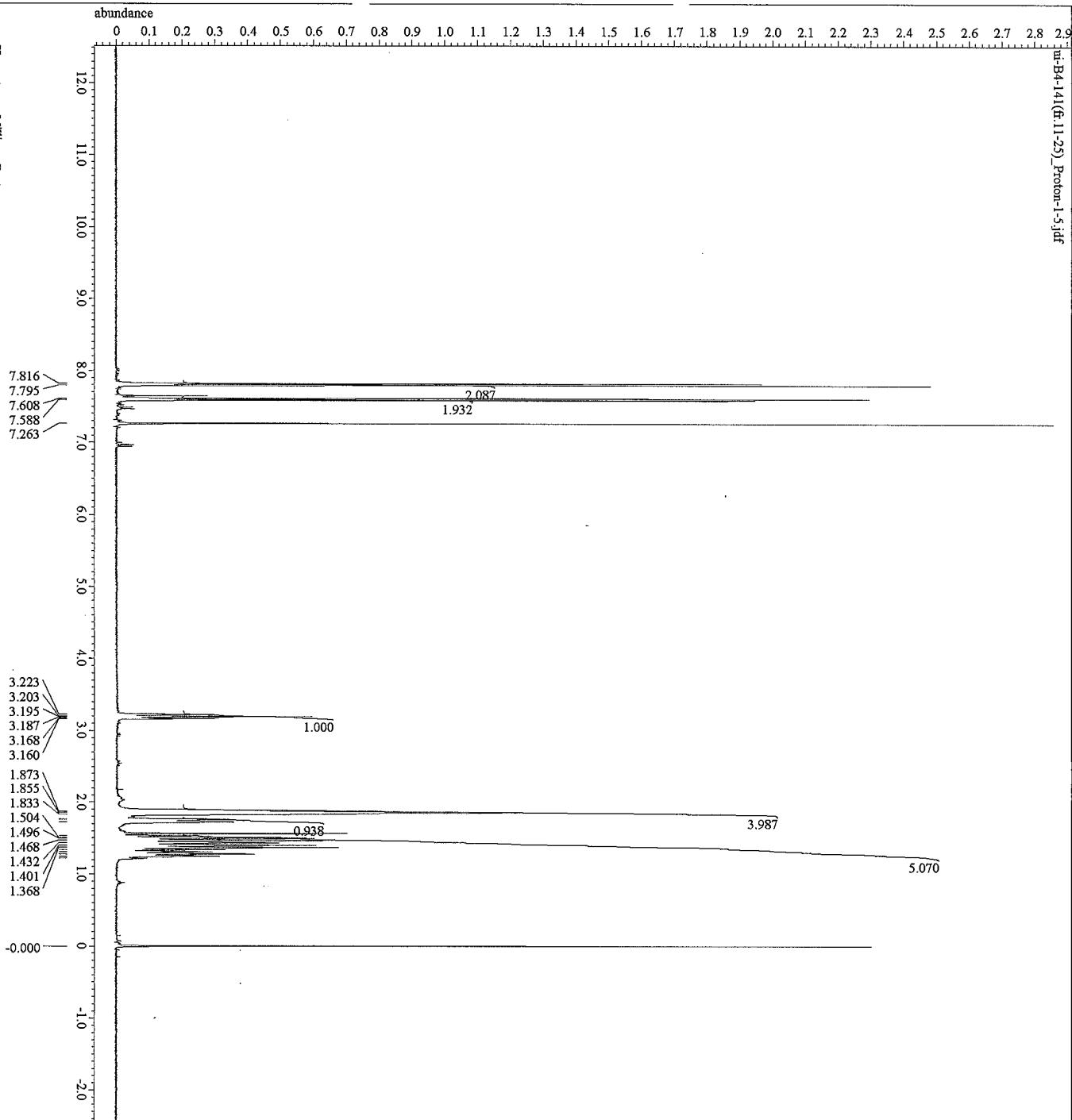
JEOL
RESONANCE

```
filename = "V:\era\delta\data\Documents\JDF"
abortion = "detra\delta\data\Documents\JDF"
experiment = "Proton_1-5"
sample_id = "U-94-44(Fr-11-25)"
solvent = "CHLOROFORM-D"
creation_time = "2-FEB-2013 17:07:55"
recreation_time = "14-MAR-2013 13:15:56"
current_time = "14-MAR-2013 13:15:56"
```

```
comment = "single_pulse"
data_format = "1D_NORMED"
data_size = "1M"
dim_z = "Fr"
dim_y = "PPM"
dim_x = "PPM"
dim_nucl = "X"
dim_t = "PPM"
dim_s = "DETECTOR_NUM"
spectrometer = "DETAQ2_NUM"
field_strength = "9.42499611 [T] (400 [MHz])"
k_2dq_duration = "2.1757951 [s]"
k_domain = "1H"
k_freq = "401.82119856 [MHz]"
k_offset = "51 [ppm]"
k_points = "1684"
k_precam = "1"
k_resolution = "0.45560208 [Hz]"
k_sweep = "7.3912018 [Hz]"
k_sweep_clipped = "6.02405659 [Hz]"
kx_domain = "Proton"
kx_freq = "401.82119856 [MHz]"
kx_offset = "5 [ppm]"
ky_domain = "Proton"
ky_freq = "401.82119856 [MHz]"
ky_offset = "5 [ppm]"
kz_domain = "Proton"
kz_freq = "401.82119856 [MHz]"
kz_offset = "PAUSE"
scans = "8"
total_scans = "8"
relaxation_delay = "5 [s]"
recv_gain = "48"
temp_get = "20.4 [degC]"
x_90_width = "9.35 [us]"
x_k2dq_name = "2.1757951 [s]"
x_angle = "45 [deg]"
x_ktrn = "0.8 [deg]"
x_pulse = "4.025 [us]"
x_trig = "off"
trig_mode = "PAUSE"
dwell_time = "1 [s]"
initial_wait = "7.1757952 [s]"
repetition_time = "7.1757952 [s]"
```



3n



X : parts per Million : Proton

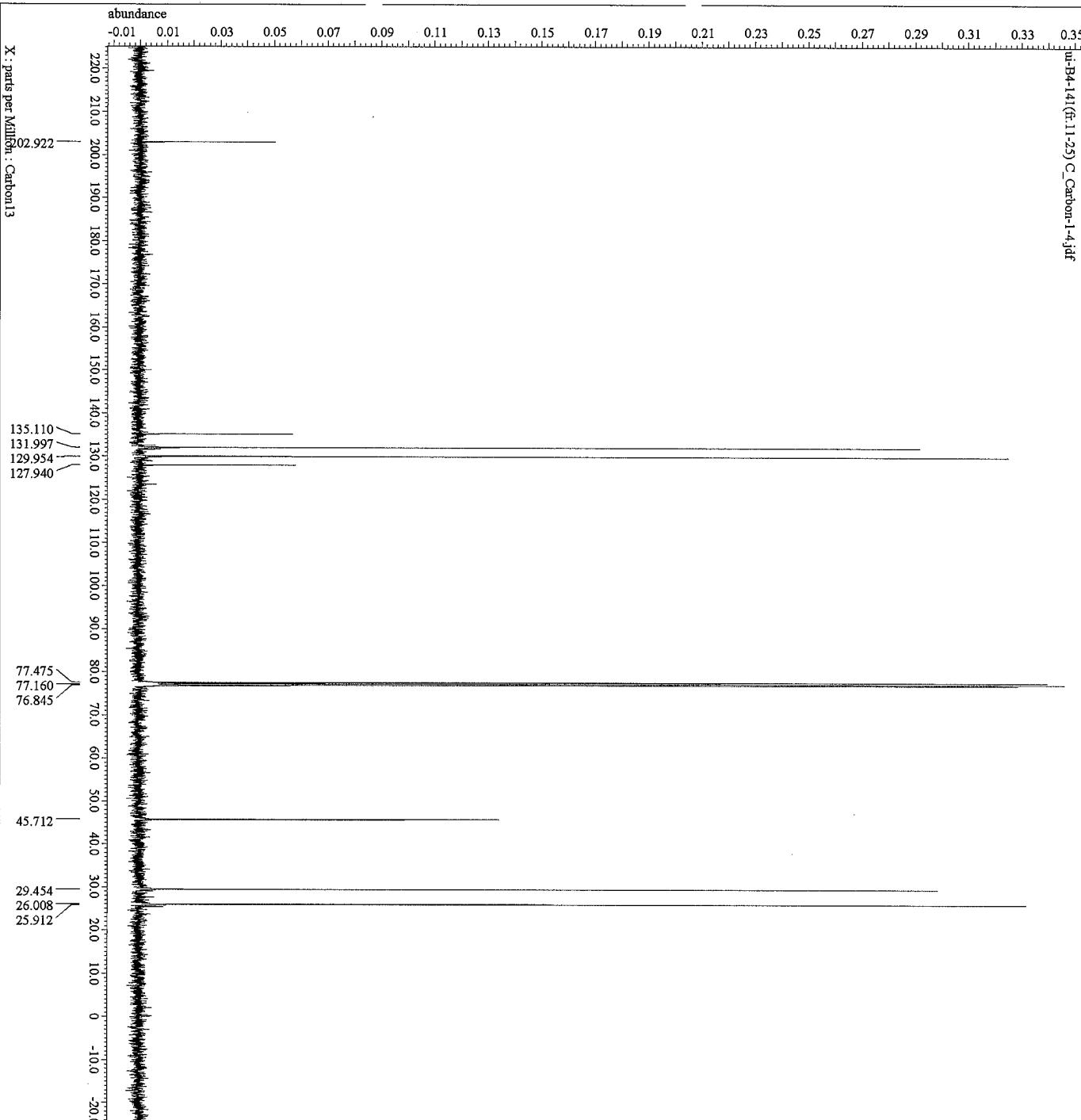
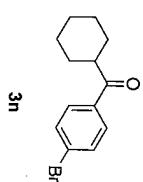
JEOL
RESONANCE

```

filename      = \Users\delta\Documents\JPG
author        = delta
experiment   = carbon_3D
sample_id    = ui-B4-141(f.11-25) C
solvent       = CLODORPHENOL
creation_time = 22-FEB-2013 17:22:28
revision_time = 14-SEP-2013 13:13:08
current_time  = 14-SEP-2013 13:13:21

comment      = single pulse decoupled gat
data_format  = 1D_COSYINE
dim_1_size   = 26113
dim_2_size   = Carbon13
dim_3_size   = 1[ppm]
dim_4_size   = 1[ppm]
size         = 1000000000
spectrometer = INNOVA_300
field_strength = 9.42499611 [T]
2D_qc_duration = 1.3500024 [s]
X_Domain    = 130
X_Pfreq     = 100.9049853 [MHz]
X_Offset    = 32768
X_Freq      = 4
X_Resonant  = 0.9639779 [Hz]
X_Sweep      = 31.3555555 [Hz]
X_Sweep_Clipped = 25.282828282 [Hz]
Xr_Domain   = proton
Xr_Freq     = 401.18219856 [MHz]
Xr_Offset   = 51ppm
Clipped     = FALSE
Scans        = 256
rot1, Scans = 256
relaxation_delay = 2 [s]
recv_gain    = 50
temp_get    = 20.3 [deg]
X_90_width  = 8.75 [us]
X_k90_gramw = 1.03000024 [s]
X_Angle      = 30.0 [deg]
X_Atn       = 5.2 [db]
X_Pulse      = 2.9166667 [us]
Xr_Atn_Dec = 22.691 [db]
Xr_Atn_Gee = 22.691 [db]
Xr_Bsize    = 10000
Xr_Fidtch   = 0.115 [us]
decoupling   = TRUE
Initial_Wait = 1 [s]
No_TIME     = 2 [s]
no_time     = 3.03609024 [s]
repetition_time = 3.03609024 [s]

```



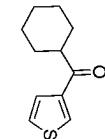
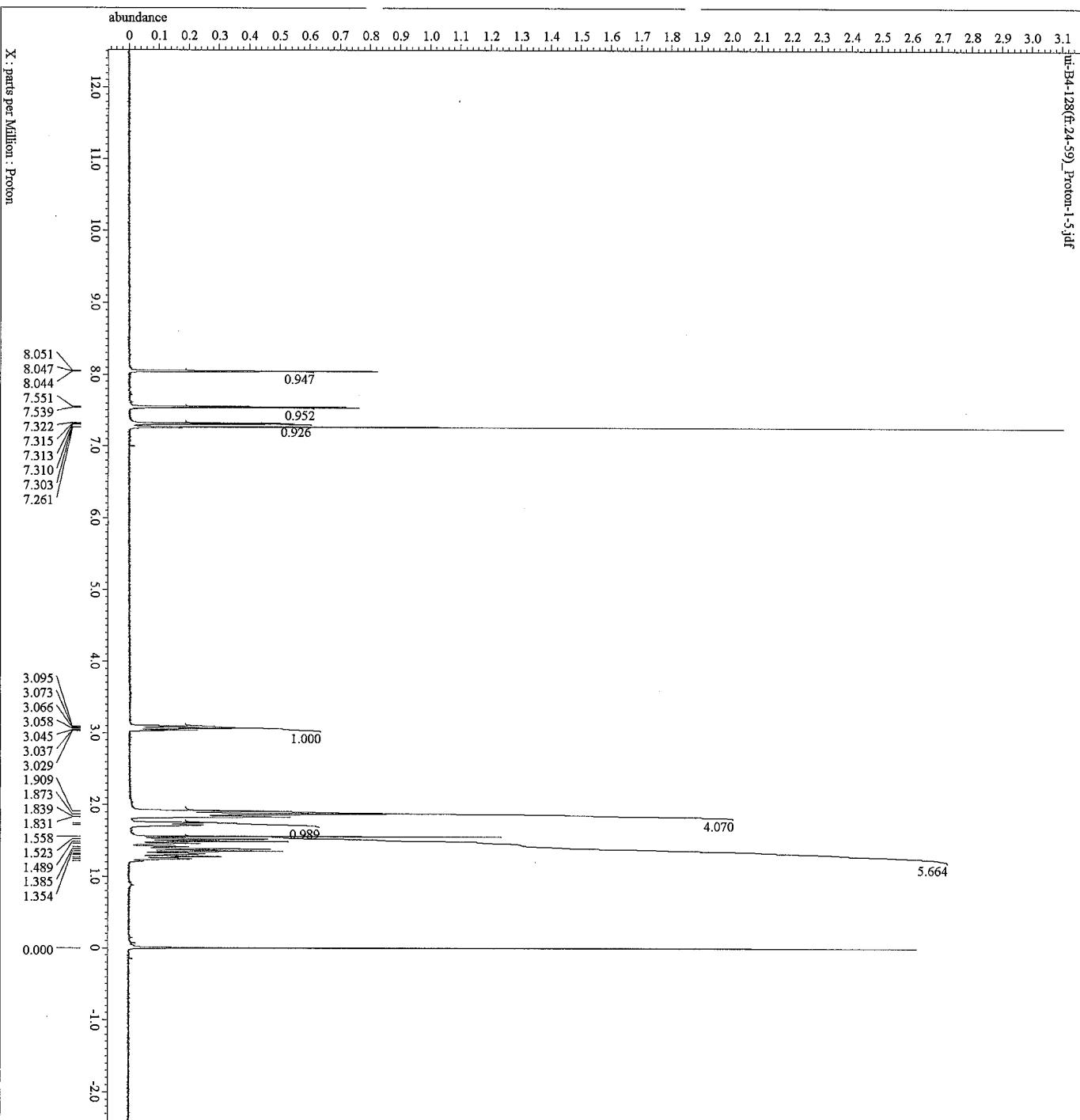
X : parts per Million : Carbon13

JEOL
RESONANCE

```

filename = \Users\delta\Documents\NMR
author = delta
Experiment = Proton JRD (Ex 24-59)
Sample_ID = ui-B-128(f2-24-59)
Solvent = CDCl3
Creation_Time = 13-FEB-2013 18:22:26
Revision_Time = 14-MAR-2013 13:30:27
Current_Time = 14-MAR-2013 13:30:42
Comment = single pulse
Date_Fomat = 1D_CHEM3D
Data_Size = 13.07MB
Data_Type = Proton
Data_Units = [ppm]
Dimensions = 1
SpectrumID = nmr-20130400
SpectrumID2 = nmr20130400
SpectrumID3 = nmr20130400
Pulse_Strength = 9.42499651[Hz] (400 [MHz])
X_Avg_Duration = 2.175775[ms]
X_Domain = 1H
X_Freq = 401.3831956 [MHz]
X_Offset = 5 [ppm]
X_Points = 16384
X_Precise = 1
X_Projection = 0.45560208 [Hz]
X_Sweep = 7.3312018 [Hz]
X_Sweep_Clipped = 6.02405659 [Hz]
Xr_Domain = Proton
Xr_Freq = 401.3831956 [MHz]
Xr_Offset = 5 [ppm]
Xr_Domainin = Proton
Xr_Freqin = 401.3831956 [MHz]
Xr_Offsetin = 5 [ppm]
Clipped = FALSE
Scans = 8
Total_Scans = 8
Relaxation_Delay = 5 [s]
Recv_Gain = 50
Recv_Weight = 20.3 [deg]
X90_Width = 9.25 [us]
X90Q_Phase = 2.1757952 [s]
X_Angle = 45 [deg]
X_Atn = 0.8 [dB]
X_Pulse = 4.625 [us]
Xr_Mode = OFF
TriMode = PAUSE
Dante_Preset = 1 [s]
Initial_Wait = 7.1757952 [s]
Repetition_Time =

```



3o

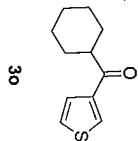
X : parts per Million : Proton

JEOL
RESONANCE

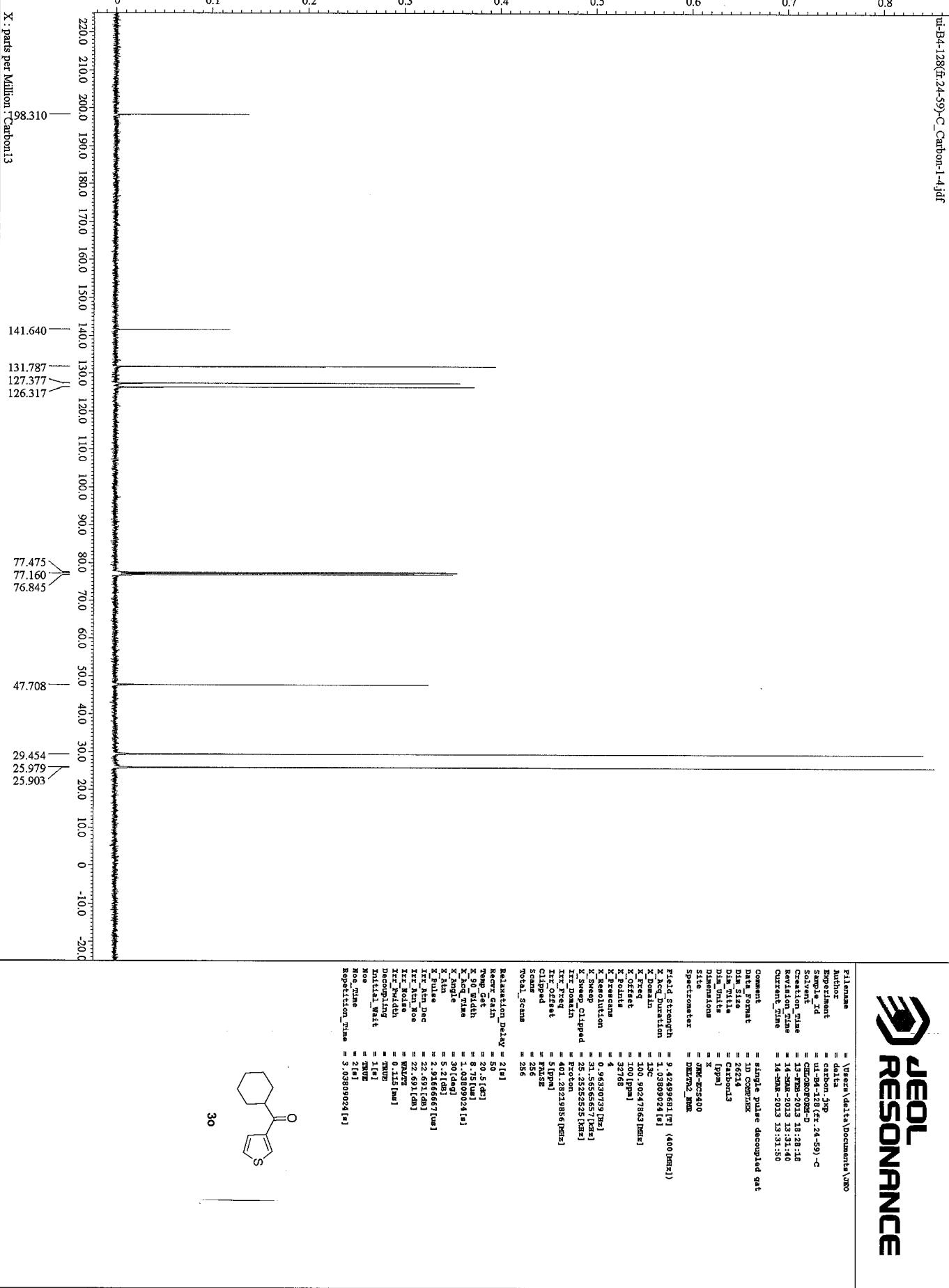
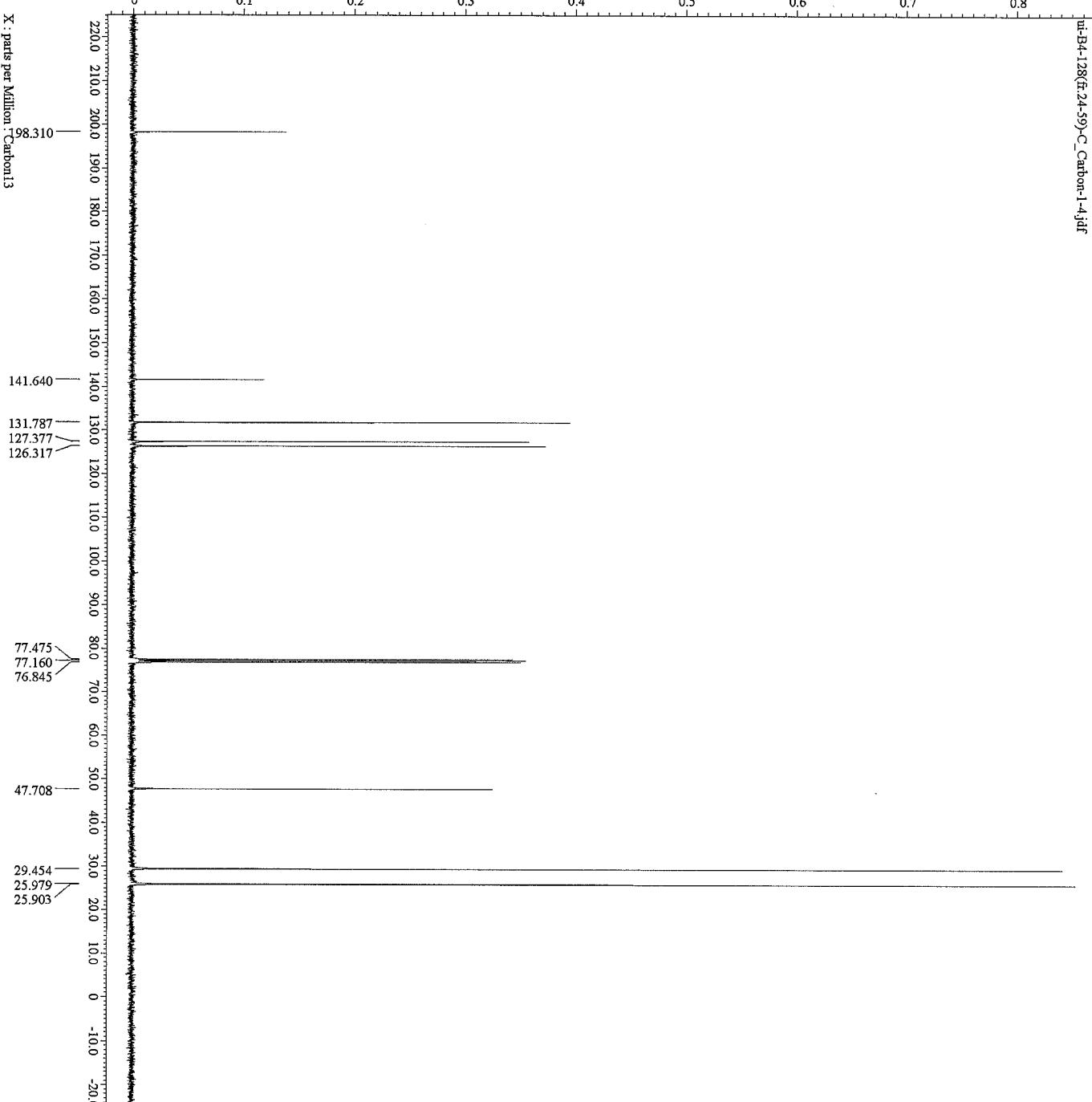
```

PulseSequence          = \User\deltap\documents\J00
Author                = data
Experiment           = carbon_13p
Sample_2d             = ui-B4-128(fr-24-59)-C
Solvent               = CHLOROFORM-D
Creation_Time         = 13-FEB-2013 18:12:18
Revision_Time         = 14-MAR-2013 13:31:40
Current_Time          = 14-MAR-2013 13:31:50
Comment               = single pulse decoupled gat
Pdppf_Fourier         = ID_CPMGEX
Dm_Size               = 26213
Dm_Tutte              = Carbon13
Dm_Units              = [ppm]
Dm_Amplitude          = X [ppm]
Spectrum              = JMR-500-400
Spectrum2             = JMR-200
Spectrum3             = JMR-200
X_Freq                = 100 [ppm]
X_Offset               = 32768
X_Pow                 = 4
X_AccTime              = 0.9639779 [sec]
X_Sweep               = 31.5959555 [sec]
X_Sweep_Direction     = 1.35000024 [e]
X_Domain              = Proton
X_Freq2               = 100 , 90.47863 [ppm]
X_Offset2              = 100 [ppm]
X_Pow2                = 32768
X_AccTime2             = 0.9639779 [sec]
X_Sweep2              = 31.5959555 [sec]
X_Sweep_Direction2    = 1.35000024 [e]
X_Domain2              = Proton
X_Freq3               = 401.78219856 [ppm]
X_Offset3              = 51 [ppm]
Clipped               = FALSE
Scans                 = 256
Total_Scans            = 256
Relaxation_Delay      = 2 [s]
Recv_Gain              = 50
T90_Get                = 20.5 [deg]
X90_Width              = 8.75 [deg]
X90_GoQ_Raw             = 1.03000024 [s]
X_Angle                = 30 [deg]
X_Alt                 = 5.2 [deg]
X_Full                = 2.9166667 [deg]
Xr_Alt_Pec              = 22.651 [deg]
Xr_Alt_Geo              = 22.651 [deg]
Xr_Role                = WALTZ
Xr_Pw1t                = 0.15 [ns]
Decoupling             = TRUE
Initial_Wait           = 1 [s]
None                  = TRUE
None_Rule              = 2 [s]
Repetition_Time        = 3.03809024 [s]

```



30



JEOL
RESONANCE

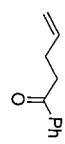
```

File Name: = \Users\deltaita\Documents\J00
Author: = delta
Experiment: = proton_1dp
Sample: = 11.m - Bif-6-01
Scanner: = corona
Creation_Time: = 9-MAY-2013 18:24:59
Revision_Time: = 9-MAY-2013 19:21:27
Current_Time: = 9-MAY-2013 19:21:39

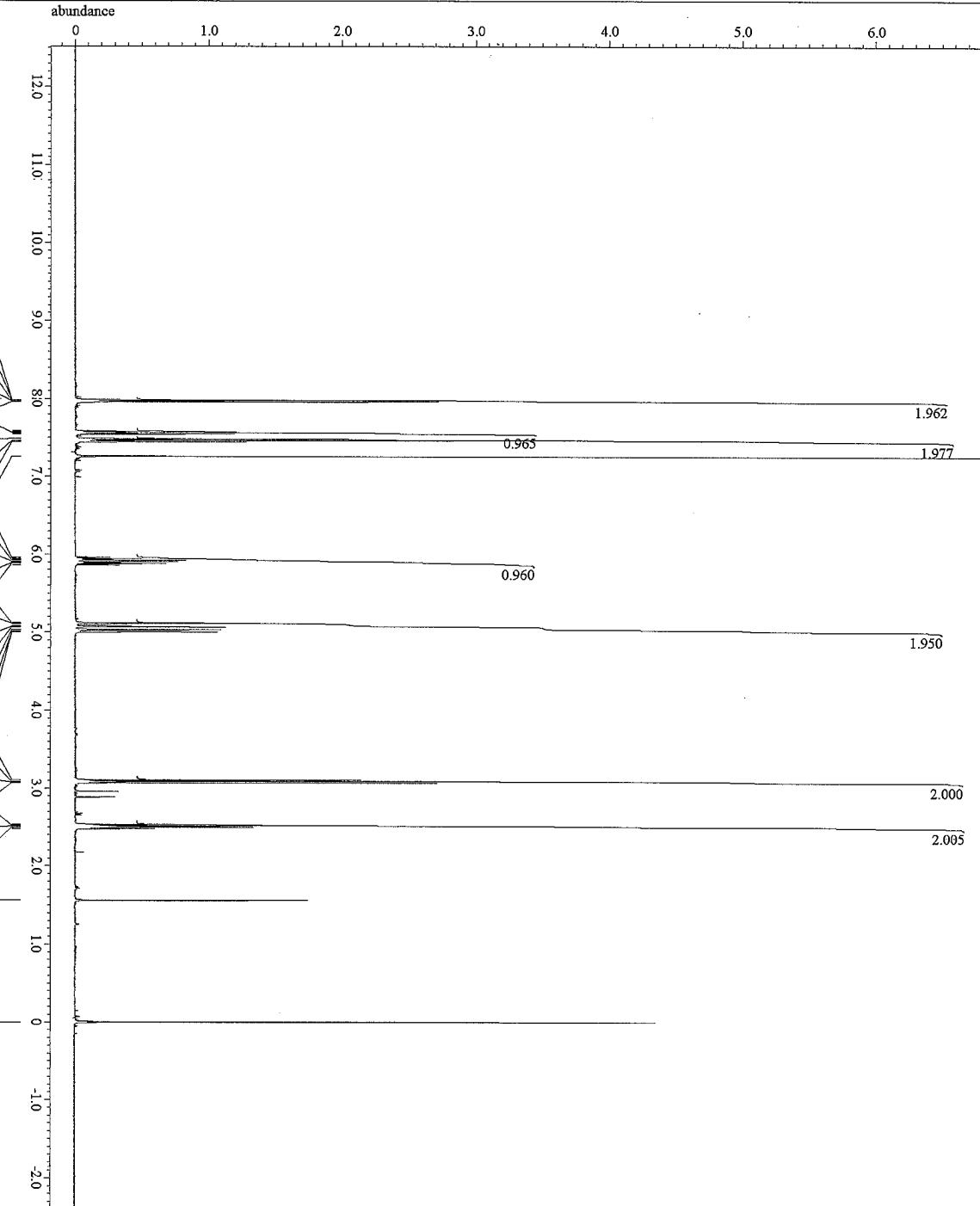
Comment: = simple_pulse
Pulse_Pattern: = 1D_COSINE
Acq_Scan: = 13.07
Acq_Tittle: = Proton
Acq_Units: = [ppm]
Acq_Avg: = X
Acq_JRES: = JRES-PRO-400
Spectrometer: = DEVAPO2_NMR

Field_Strength: = 9.42999611[pp] (400 [MHz])
Acq_Duration: = 2.1757952[ms]
L_DOMAIN: = 1H
L_FREQ: = 401.2819886[MHz]
L_OFFSET: = 5[ppm]
L_POINTS: = 16384
L_PPSIZE: = 1
L_RESOLUTION: = 0.4556208[Hz]
L_SWEEP: = 7.3011048[Hz]
L_SWEEP_CLIPPED: = 6.02409639[Hz]
L_DOMAIN: = Proton
L_FREQ: = 401.2819886[MHz]
L_OFFSET: = 5[ppm]
L_DOMAIN: = Proton
L_FREQ: = 401.2819886[MHz]
L_OFFSET: = 5[ppm]
CLIPGED: = FALSE
SCANS: = 8
TOTAL_SCANS: = 8
RELAXATION_DELAY: = 5 [s]
REVR_GAIN: = 50
TEMP_GEV: = 20.6 [deg]
X90_WIDTH: = 9.25 [us]
X90Q_TIME: = 2.1757952[ms]
X_ANGLE: = 45[deg]
X_RNM: = 0.8 [us]
X_FULME: = 4.625 [us]
IRR_MODE: = OFF
TR1_MODE: = OFF
DANTE_PREAM: = FALSE
INITIAL_WAIT: = 1[s]
REPETITION_TIME: = 7.1757952[ms]

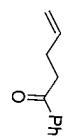
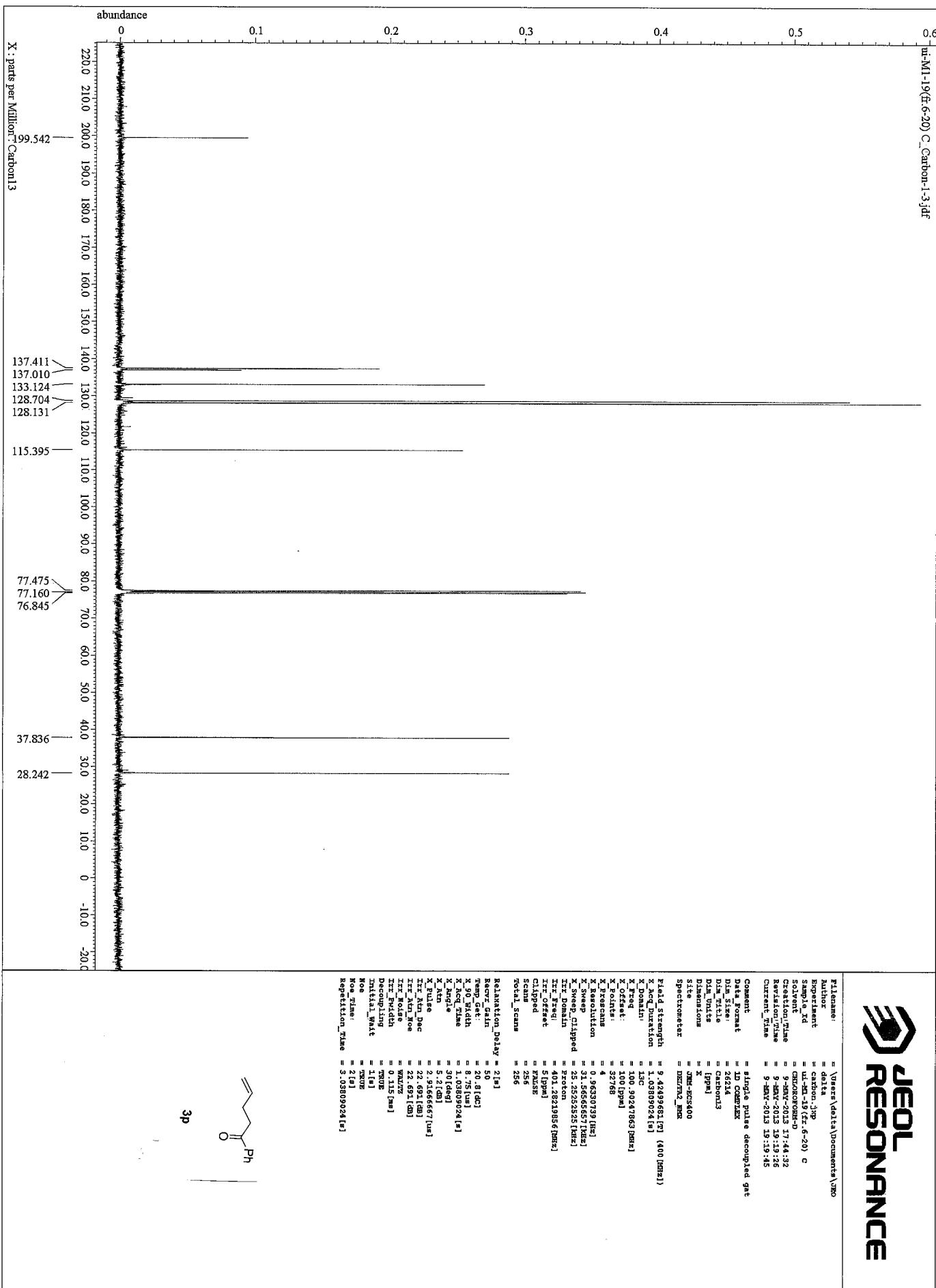
```



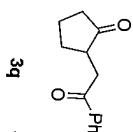
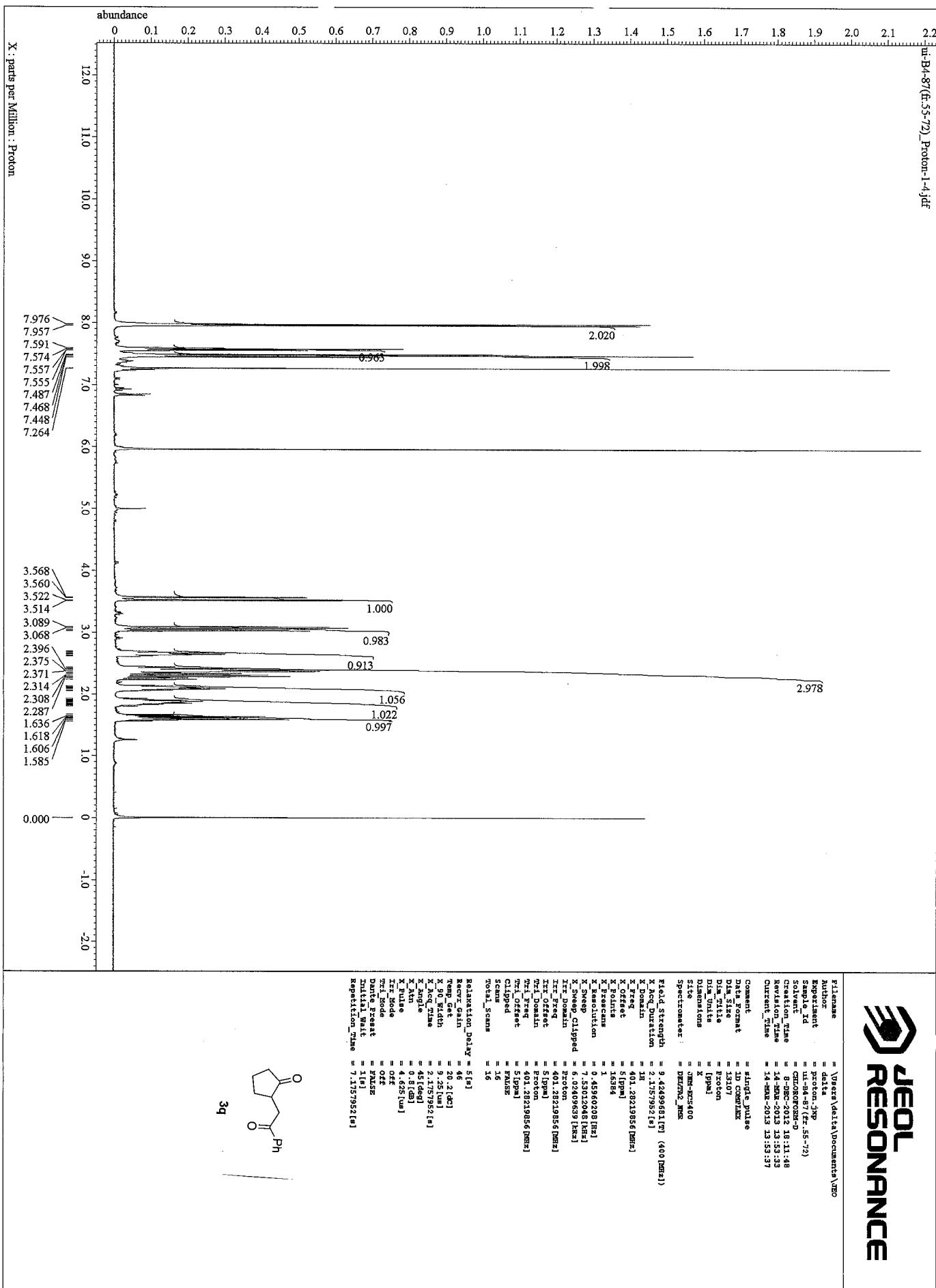
3p

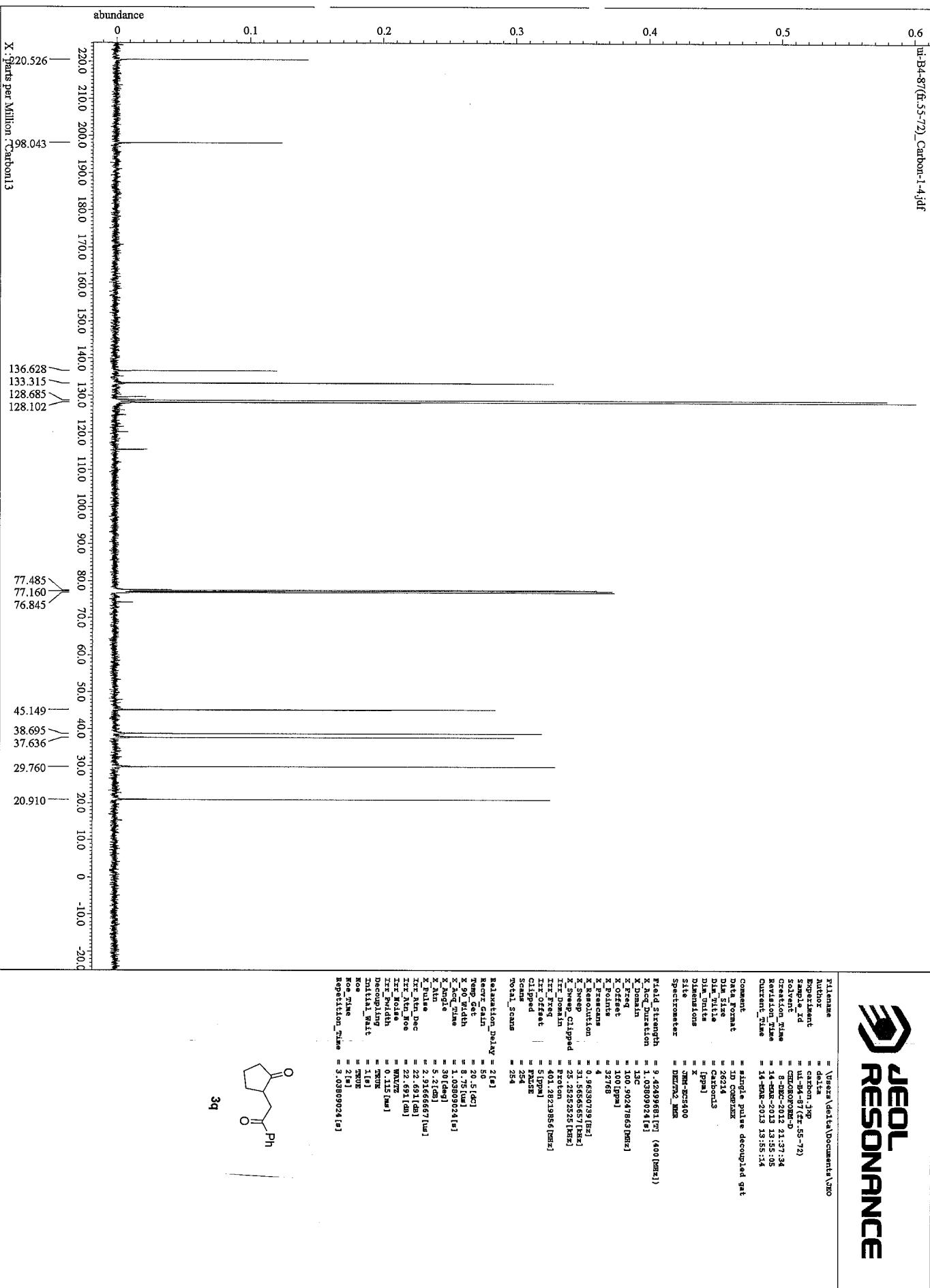


X : parts per Million : Proton



८





JEOL
RESONANCE

