Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Synthesis of 2-monosubstituted indolin-3-ones by *cine*-substitution of 3-azido-2-methoxyindolines

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SUPPORTING INFORMATION

Contents

1. General Experimental

Melting points were recorded with a METTLER TOLEDO MP50 Melting Point System and are uncorrected. High-resolution MS spectra were recorded with a Brucker micrOTOF mass spectrometers (ESI-TOF-MS). IR spectra were measured with a Shimadzu IR Affinity-1 spectrometer. The NMR experiments were performed with JEOL JNM-ECZ600R (^{1}H NMR: 600 MHz, ^{13}C NMR: 151 MHz) spectrometer, and chemical shifts are expressed in ppm (δ) using residual undeuterated solvent as an internal reference ($CDCl_3$, ^{1}H NMR: δ 7.25, ^{13}C NMR: δ 77.1). The following abbreviations were used to explain NMR peak multiplicities: s = singlet, d = doublet, t = triplet, sep = septet, m = multiplet, dd = doublet of doublets, ddd = doublet of doublet of doublets, tt = triplet of triplets; coupling constants in Hz; integration.

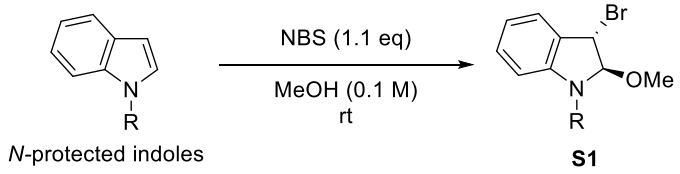
Reactions were monitored by thin layer chromatography (TLC) carried out on a silica gel plates (60F-254) and visualized under UV illumination at 254 or 365 nm depending on the compounds. Flash column chromatography was performed on silica gel (WAKO Gel 75−150 mesh, WAKO Co., Ltd.).

2. Experimental Procedure

■Synthesis of *N*-protected indoles

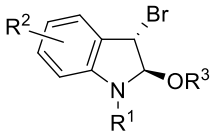
The *N*-protected indoles as *N*-tosylindoles, *N*-benzenesulfonylindole, *N*-(4-methoxybenzenesulfonyl)indole and *N*-mesylindole were prepared by reported methods.[S1, 2] All substrates were used as received from commercial suppliers (Sigma-Aldrich, Kanto Chemical, TCI and Wako) and all reagents were weighed and handled in air at room temperature.

■General procedure for the bromoalkoxylation of *N*-protected indoles

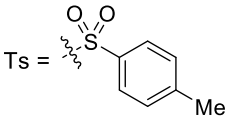


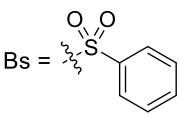
N-protected indoles　　**S1**

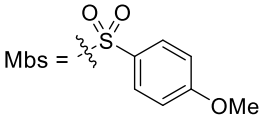
2-Alkoxy-3-bromo-*N*-protected indolines

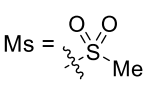


S1





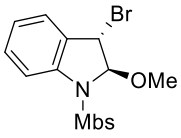




entry	R^1 =	R^2 =	R^3 =
S1a	Ts	H	Me
S1b	Bs	H	Me
S1c	Mbs	H	Me
S1d	Ms	H	Me
S1e	Ts	5-OMe	Me
S1f	Ts	5-Cl	Me
S1g	Ts	5-Br	Me
S1h	Ts	4-Cl	Me
S1i	Ts	6-Cl	Me
S1j	Ts	7-Cl	Me
S1k	Ts	H	*i*Pr
S1l	Ts	H	*t*Bu
S1m	Ts	H	Bn

To a solution of *N*-protected indoles (2.0 mmol) in MeOH (20 mL, 0.1 M) was added NBS (392 mg, 1.1 mmol). The mixture was stirred at room temperature until the complete disappearance of starting material as indicated by TLC. After H_2O (20 mL) was added to the mixture, the whole was extracted with AcOEt (3 x 50 mL), washed with brine (25 mL). The combined organic layer was dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by recrystallization from MeOH and/or silica gel column chromatography (AcOEt/hexane = 1/8–1/2 and/or $CHCl_3$/hexane = 1/3–1/1) to give 2-alkoxy-3-bromo-*N*-protectedindolines **S1c** and **S1d**. The other compounds were prepared by our reported method.[S3]

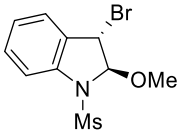
trans-3-Bromo-2-methoxy-1-(4-methoxybenzenesulfonyl)indoline (S1c)



S1c

708 mg, 89% yield. colorless solid; mp 121–123 °C; IR (KBr): 1595, 1499, 1352, 1155, 974 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.73 (ddd, *J* = 8.4, 1.8, 1.8 Hz, 2H), 7.64 (d, *J* = 7.8 Hz, 1H), 7.31 (ddd, *J* = 8.4, 7.8, 0.6 Hz, 1H), 7.27 (d, *J* = 6.6 Hz, 1H), 7.09 (ddd, *J* = 8.4, 7.8, 0.6 Hz, 1H), 6.84 (ddd, *J* = 8.4, 1.8, 1.8 Hz, 2H), 5.57 (s, 1H), 4.93 (s, 1H), 3.78 (s, 3H), 3.59 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 163.7, 140.7, 131.5, 130.6, 130.0, 129.9, 126.2, 125.4, 117.1, 114.1, 99.9, 56.4, 55.6, 47.3; HRMS (ESI) *m/z*: 419.9882, 421.9860 (Calcd for $C_{16}H_{16}BrNO_4SNa$ $[M+Na]^+$: 419.9881, 421.9861).

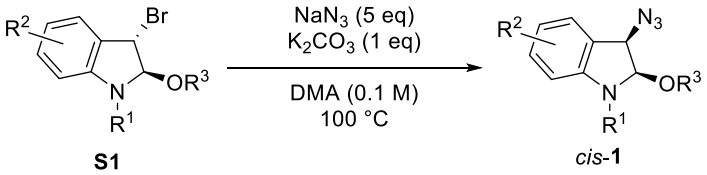
trans-3-Bromo-1-mesyl-2-methoxyindoline (S1d)



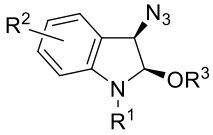
S1d

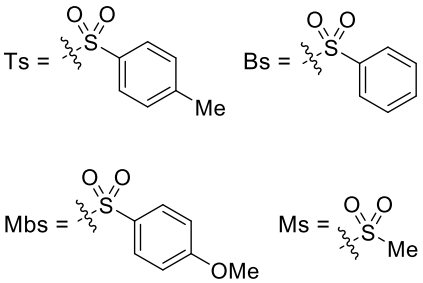
557 mg, 79% yield. colorless solid; mp 114–115 °C; IR (KBr): 1605, 1462, 1342, 1155, 995 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.50 (d, *J* = 8.4 Hz, 1H), 7.43 (d, *J* = 8.4 Hz, 1H), 7.36 (t, *J* = 8.4 Hz, 1H), 7.18 (t, *J* = 8.4 Hz, 1H), 5.59 (s, 1H), 5.10 (s, 1H), 3.52 (s, 3H), 3.05 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 140.5, 131.4, 131.1, 126.6, 125.7, 116.2, 100.1, 56.3, 47.9, 39.0; HRMS (ESI) *m/z*: 327.9619, 329.9599 (Calcd for $C_{10}H_{12}BrNO_3SNa$ $[M+Na]^+$: 327.9619, 329.9599).

■General procedure for the synthesis of *cis*-3-azido-2-methoxyindolines (AZINs) 1



2-Alkoxy-3-azido-*N*-protected indolines





entry	R^1 =	R^2 =	R^3 =
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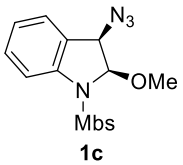
1b	Bs	H	Me
1c	Mbs	H	Me
1d	Ms	H	Me
1e	Ts	5-OMe	Me
1f	Ts	5-Cl	Me
1g	Ts	5-Br	Me
1h	Ts	4-Cl	Me
1i	Ts	6-Cl	Me
1j	Ts	7-Cl	Me
1k	Ts	H	*i*Pr
1l	Ts	H	*t*Bu
1m	Ts	H	Bn

To a solution of K_2CO_3 (138 mg, 1 mmol) and NaN_3 (325 mg, 5 mmol) in DMA (dimethylacetamide) (10 mL, 0.1 M) was added **S1** (1 mmol) and the mixture was stirred at 100 °C in oil bath until the complete disappearance of starting material as indicated by TLC. After the reaction mixture was cooled down to room temperature, H_2O (10 mL) was added to the mixture. Then, the whole was extracted with AcOEt/hexane = 1/5 (3 x 25 mL), washed with H_2O (25 mL) and brine (25 mL). The combined organic layer was dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by recrystallization from $CHCl_3$/hexane and/or silica gel column chromatography (AcOEt/hexane = 1/10–1/3) to give *cis*-**1c**, **1d**, **1l**. The other compounds were prepared by our reported method.[S4]

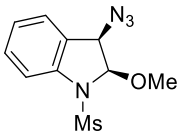
cis-3-Azido-2-methoxy-1-(4-methoxybenzenesulfonyl)indoline (1c)



1c

320 mg, 89% yield. colorless solid; mp 109–111 °C; IR (KBr): 2114, 1593, 1462, 1356, 1167 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.61 (ddd, *J* = 9.0, 2.4, 2.4 Hz, 2H), 7.55 (d, *J* = 7.8 Hz, 1H), 7.32 (ddd, *J* = 9.0, 7.8, 1.2 Hz, 1H), 7.21 (d, *J* = 7.2 Hz, 1H), 7.15 (ddd, *J* = 7.2, 6.0, 1.2 Hz, 1H), 6.84 (ddd, *J* = 9.0, 2.4, 2.4 Hz, 2H), 5.39 (d, *J* = 5.4 Hz, 1H), 4.09 (d, *J* = 5.4 Hz, 1H), 3.80 (s, 3H), 3.63 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 163.6, 139.9, 130.3, 130.0, 129.6, 129.1, 125.5, 124.4, 118.0, 114.5, 94.5, 63.0, 56.8, 55.7; HRMS (ESI) *m/z*: 383.0790 (Calcd for $C_{16}H_{16}N_4O_4SNa$ $[M+Na]^+$: 383.0790).

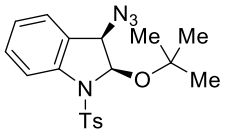
cis-3-Azido-1-mesyl-2-methoxyindoline (1d)



1d

231 mg, 86% yield. colorless solid; mp 91–93 °C; IR (KBr): 2110, 1605, 1460, 1348, 1161 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.39 (d, *J* = 8.4 Hz, 1H), 7.37 (d, *J* = 8.4 Hz, 1H), 7.35 (t, *J* = 8.4 Hz, 1H), 7.16 (t, *J* = 8.4 Hz, 1H), 5.43 (d, *J* = 6.0 Hz, 1H), 4.61 (d, *J* = 6.0 Hz, 1H), 3.69 (s, 3H), 3.08 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 140.2, 130.4, 126.5, 125.2, 124.2, 113.4, 95.2, 62.4, 58.8, 40.2; HRMS (ESI) *m/z*: 291.0527 (Calcd for $C_{10}H_{12}N_4O_3SNa$ $[M+Na]^+$: 291.0528).

cis-3-Azido-2-*tert*-butoxy-1-tosylindoline (1l)

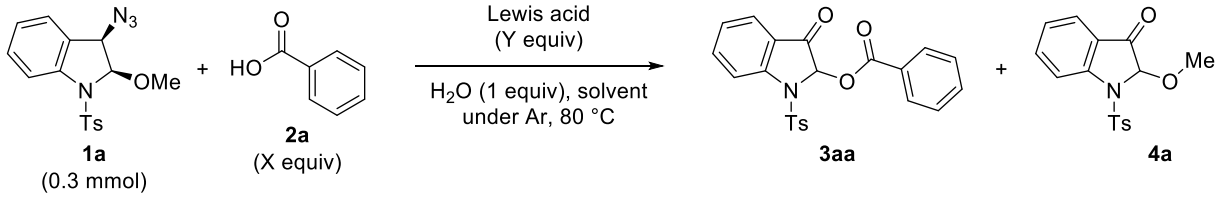


1l

308 mg, 80% yield. colorless solid; mp 149–150 °C; IR (KBr): 2110, 1597, 1460, 1348, 1169 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.56 (d, *J* = 8.4 Hz, 1H), 7.45 (ddd, *J* = 8.4, 1.8, 1.8 Hz, 2H), 7.35–7.32 (m, 1H), 7.19–7.16 (m, 2H), 7.13 (d, *J* = 8.4 Hz, 2H), 5.77 (d, *J* = 5.4 Hz, 1H), 3.69 (d, *J* = 5.4 Hz, 1H), 2.34 (s, 3H), 1.41 (s, 9H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 144.4, 140.4, 135.9, 131.3, 129.8, 129.7, 126.8, 126.1, 124.2, 119.9, 89.7, 78.1, 63.2, 28.6, 21.6; HRMS (ESI) *m/z*: 409.1310 (Calcd for $C_{19}H_{22}N_4O_3SNa$ $[M+Na]^+$: 409.1310).

■Optimization of Reaction Conditions

Table S1 Optimization of reaction conditions using 1a and 2a

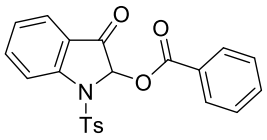


run	Lewis acid	X	Y	solvent	time (h)	yield (%)	
						3aa[a]	**4a**[a]
1	$InCl_3•4H_2O$	5	1	DCE	0.1	27	9
2	$InCl_3•4H_2O$	5	2	DCE	0.1	37	32
3	$In(OTf)_3$	5	1	DCE	0.1	37	32
4	$Cu(OTf)_2$	5	1	DCE	0.1	17	40
5	$Zn(OTf)_2$	5	1	DCE	0.1	1	0
6	**$Zn(OTf)_2$**	**5**	**2**	**DCE**	**16**	**72**	**8**
7	$BF_3•OEt_2$	5	1	DCE	0.1	38	15
8	**$BF_3•OEt_2$**	**5**	**2**	**DCE**	**0.1**	**74**	**16**
9	$BF_3•OEt_2$	2	2	DCE	0.1	59	21
10	$BF_3•OEt_2$	5	3	DCE	0.1	80	11
11	$BF_3•OEt_2$	3	3	DCE	0.1	67	12
12	**$BF_3•OEt_2$**	**5**	**5**	**DCE**	**0.1**	**85**	**8**
13	$BF_3•OEt_2$	5	10	DCE	0.1	87	5
14[b]	$BF_3•OEt_2$	5	5	DCE	0.1	54	23
15[c]	$BF_3•OEt_2$	5	5	DCE	0.1	59	20
16	$BF_3•OEt_2$	5	5	MeCN	0.1	73	7
17	$BF_3•OEt_2$	5	5	toluene	0.1	62	6
18	$BF_3•OEt_2$	5	5	HFIP	1.5	77	7
19	$BF_3•OEt_2$	5	5	AcOEt	0.1	74	7
20	—	5	—	DCE	24	nr	

[a] Isolated yields. [b] Using H_2O (10 equiv). [c] Without using H_2O.

To a solution of **1a** (103 mg, 0.3 mmol) and benzoic acid **2a** (0.3 x X mmol, X equiv) in solvent (1.5 mL, 0.2 M) was added lewis acid (0.3 x Y mmol, Y equiv) under Ar. The mixture was stirred at 80 °C in oil bath until the complete disappearance of starting material and 3-azidoindole as indicated by TLC or for 16 h. The reaction mixture was quenched by H_2O (3 mL) and cooled to room temperature. Then, the whole was extracted with $CHCl_3$ (3 x 20 mL), washed with brine (20 mL). The organic layer was dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/10–1/5) to give **3aa** and **4a**.

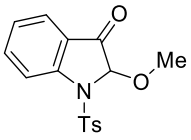
1-Tosylindolin-3-on-2-yl benzoate (3aa)



3aa

Table S1, run 12: 103 mg, 85% yield. colorless solid; mp 138–140 °C; IR (KBr): 1740, 1605, 1462, 1375, 1177 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 8.02–7.99 (m, 3H), 7.78 (d, *J* = 7.8 Hz, 2H), 7.70 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 7.70 (d, *J* = 7.8 Hz, 1H), 7.59 (d, *J* = 7.8 Hz, 1H), 7.43 (t, *J* = 8.4 Hz, 2H), 7.22 (t, *J* = 8.4 Hz, 1H), 7.19 (d, *J* = 7.8 Hz, 2H), 6.38 (s, 1H), 2.31 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 191.3, 164.7, 152.1, 145.5, 138.1, 134.3, 134.0, 130.4, 130.2, 128.5, 128.4, 127.5, 125.2, 124.8, 122.5, 115.8, 80.5, 21.7; HRMS (ESI) *m/z*: 430.0725 (Calcd for $C_{22}H_{17}NO_5SNa$ $[M+Na]^+$: 430.0725).

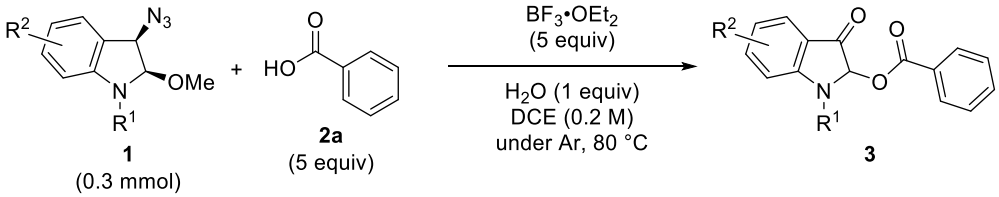
2-Methoxy-1-tosylindolin-3-one (4a)



4a

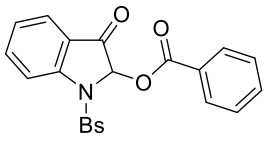
Table S1, run 12: 7.8 mg, 8% yield. colorless solid; mp 131–133 °C; IR (KBr): 1726, 1605, 1460, 1357, 1173 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.82 (d, *J* = 7.8 Hz, 1H), 7.75 (d, *J* = 7.8 Hz, 2H), 7.64 (ddd, *J* = 8.4, 1.2, 1.2 Hz, 1H), 7.61 (d, *J* = 7.8 Hz, 1H), 7.25 (d, *J* = 7.8 Hz, 2H), 7.15 (t, *J* = 7.8 Hz, 1H) 5.12 (s, 1H), 3.51 (s, 3H), 2.37 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 194.4, 152.5, 145.2, 138.1, 134.9, 130.1, 127.4, 125.1, 124.6, 122.9, 116.2, 88.9, 55.7, 21.7; HRMS (ESI) *m/z*: 340.0620 (Calcd for $C_{16}H_{15}NO_4SNa$ $[M+Na]^+$: 340.0620).

■General Procedure for the reaction of 1 with 2a (Scheme 3: AZINs)



To a solution of **1** (0.3 mmol) and **2a** (183 mg, 1.5 mmol) in 1,2-dichloroethane (DCE) (1.5 mL, 0.2 M) was added BF_3•OEt_2 (0.19 mL, 1.5 mmol) under Ar. The mixture was stirred at 80 °C in oil bath until the complete disappearance of starting material and 3-azidoindole as indicated by TLC. The reaction mixture was quenched by H_2O (3 mL) and cooled to room temperature. Then, the whole was extracted with $CHCl_3$ (3 x 20 mL), washed with brine (20 mL). The organic layer was dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/10–1/5) to give **3aa–3ja**.

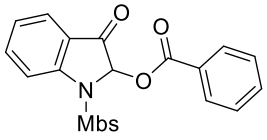
1-Benzenesulfonylindolin-3-on-2-yl benzoate (3ba)



3ba

108 mg, 92% yield. colorless solid; mp 155–157; IR (KBr): 1744, 1732, 1605, 1464, 1371, 1175 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 8.02–7.99 (m, 3H), 7.91 (d, *J* = 7.2 Hz, 2H), 7.72 (t, *J* = 7.8 Hz, 1H), 7.71 (d, *J* = 7.8 Hz, 1H), 7.59 (t, *J* = 7.2 Hz, 1H), 7.54 (t, *J* = 7.2 Hz, 1H), 7.44–7.40 (m, 4H), 7.23 (d, *J* = 6.6 Hz, 1H), 6.41 (s, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 191.2, 164.7, 152.0, 138.1, 137.3, 134.3, 134.0, 130.4, 129.6, 128.6, 128.4, 127.5, 125.2, 124.9, 122.5, 115.7, 80.5; HRMS (ESI) *m/z*: 416.0569 (Calcd for $C_{21}H_{15}NO_5SNa$ $[M+Na]^+$: 416.0569).

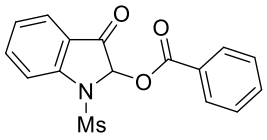
1-(4-Methoxybenzenesulfonyl)indolin-3-on-2-yl benzoate (3ca)



3ca

114 mg, 90% yield. colorless solid; mp 158–160 °C; IR (KBr): 1726, 1605, 1460, 1358, 1173 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 8.02 (dd, *J* = 7.8, 1.2 Hz, 2H), 7.99 (d, *J* = 8.4 Hz, 1H), 7.83 (ddd, *J* = 8.4, 1.8, 1.8 Hz, 2H), 7.72–7.69 (m, 2H), 7.59 (t, *J* = 7.8 Hz, 1H), 7.43 (t, *J* = 7.8 Hz, 2H), 7.23 (t, *J* = 7.8 Hz, 1H), 6.85 (ddd, *J* = 8.4, 1.8, 1.8 Hz, 2H), 6.38 (s, 1H), 3.77 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 191.4, 164.8, 164.2, 152.2, 138.1, 134.0, 130.4, 129.8, 128.7, 128.6, 128.5, 125.2, 124.7, 122.5, 115.8, 114.8, 80.5, 55.7; HRMS (ESI) *m/z*: 446.0675 (Calcd for $C_{22}H_{17}NO_6SNa$ $[M+Na]^+$: 446.0674).

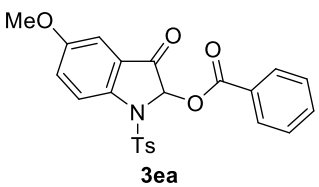
1-Mesylindolin-3-on-2-yl benzoate (3da)



3da

88.2 mg, 89% yield. colorless solid; mp 161–163 °C; IR (KBr): 1746, 1607, 1466, 1356, 1163 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 8.06 (dd, *J* = 7.8, 1.2 Hz, 2H), 7.81 (d, *J* = 7.8 Hz, 1H), 7.73–7.70 (m, 2H), 7.60 (tt, *J* = 7.8, 1.2 Hz, 1H), 7.45 (t, *J* = 7.8 Hz, 2H), 7.28 (ddd, *J* = 7.8, 6.6, 1.2 Hz, 1H), 6.50 (s, 1H), 3.11 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 191.0, 164.5, 151.6, 138.2, 134.2, 130.3, 128.8, 128.2, 125.5, 124.8, 122.3, 114.8, 80.8, 40.2; HRMS (ESI) *m/z*: 354.0412 (Calcd for $C_{16}H_{13}NO_5SNa$ $[M+Na]^+$: 354.0412).

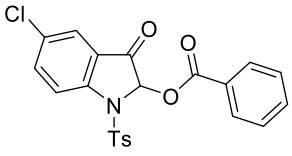
5-Methoxy-1-tosylindolin-3-on-2-yl benzoate (3ea)



3ea

108 mg, 82% yield. colorless solid; mp 154–156 °C; IR (KBr): 1743, 1732, 1597, 1498, 1366, 1171 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 8.03 (dd, *J* = 8.4, 1.2 Hz, 2H), 7.94 (d, *J* = 9.0 Hz, 1H), 7.75 (ddd, *J* = 8.4, 1.2, 1.2 Hz, 2H), 7.59 (tt, *J* = 8.4, 1.2 Hz, 1H), 7.43 (t, *J* = 7.8 Hz, 2H), 7.30 (dd, *J* = 8.4, 2.4 Hz, 1H), 7.21 (d, *J* = 7.8 Hz, 2H), 7.10 (d, *J* = 2.4 Hz, 1H), 6.30 (s, 1H), 3.80 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 191.4, 164.8, 157.3, 146.6, 145.4, 134.0, 133.8, 130.4, 130.2, 128.6, 128.4, 127.6, 126.9, 123.5, 117.5, 106.0, 81.1, 55.9, 21.7; HRMS (ESI) *m/z*: 460.0830 (Calcd for $C_{23}H_{19}NO_6SNa$ $[M+Na]^+$: 460.0831).

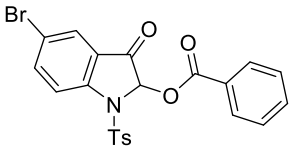
5-Chloro-1-tosylindolin-3-on-2-yl benzoate (3fa)



3fa

107 mg, 81% yield. colorless solid; mp 165–167 °C; IR (KBr): 1753, 1736, 1601, 1460, 1369, 1179, 1024 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.98 (dd, *J* = 8.4, 1.2 Hz, 2H), 7.95 (d, *J* = 8.4 Hz, 1H), 7.75 (ddd, *J* = 8.4, 1.8, 1.8 Hz, 2H), 7.66–7.63 (m, 2H), 7.60 (tt, *J* = 7.8, 1.2 Hz, 1H), 7.43 (t, *J* = 7.8 Hz, 2H), 7.20 (d, *J* = 7.8 Hz, 2H), 6.26 (s, 1H), 2.32 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 190.2, 164.6, 150.3, 145.7, 137.6, 134.1, 134.1, 130.7, 130.4, 130.3, 128.6, 128.1, 127.5, 124.6, 123.9, 117.1, 81.0, 21.7; HRMS (ESI) *m/z*: 464.0333, 466.0307 (Calcd for $C_{22}H_{16}ClNO_5SNa$ $[M+Na]^+$: 464.0335, 466.0306).

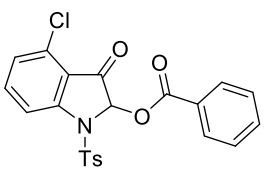
5-Bromo-1-tosylindolin-3-on-2-yl benzoate (3ga)



3ga

126 mg, 86% yield. colorless solid; mp 158–160 °C; IR (KBr): 1751, 1736, 1599, 1452, 1369, 1179, 955 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.98 (dd, *J* = 7.8, 1.2 Hz, 2H), 7.90 (d, *J* = 8.4 Hz, 1H), 7.81 (d, *J* = 1.8 Hz, 1H), 7.78 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.75 (ddd, *J* = 8.4, 1.8, 1.8 Hz, 2H), 7.60 (tt, *J* = 8.4, 1.2 Hz, 1H), 7.43 (t, *J* = 7.8 Hz, 2H), 7.20 (d, *J* = 7.8 Hz, 2H), 6.26 (s, 1H), 2.32 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 190.0, 164.6, 150.8, 145.7, 140.4, 134.1, 134.1, 130.4, 130.3, 128.6, 128.1, 127.7, 127.5, 124.2, 118.0, 117.4, 80.8, 21.7; HRMS (ESI) *m/z*: 507.9832, 509.9810 (Calcd for $C_{22}H_{16}BrNO_5SNa$ $[M+Na]^+$: 507.9830, 509.9810).

4-Chloro-1-tosylindolin-3-on-2-yl benzoate (3ha)

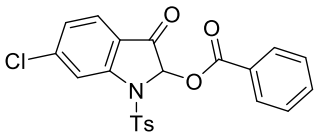


3ha

114 mg, 86% yield. colorless solid; mp 168–170 °C; IR (KBr): 1749, 1724, 1597, 1470, 1375, 1177, 1024 cm^{-1}; ^1H

NMR (600 MHz, $CDCl_3$) δ: 8.00 (dd, *J* = 8.4, 1.2 Hz, 2H), 7.91 (d, *J* = 7.8 Hz, 1H), 7.76 (ddd, *J* = 7.8, 1.8, 1.8 Hz, 2H), 7.61–7.57 (m, 2H), 7.43 (t, *J* = 7.8 Hz, 2H), 7.20 (d, *J* = 8.4 Hz, 2H), 7.16 (d, *J* = 7.8 Hz, 1H), 6.34 (s, 1H), 2.33 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 188.4, 164.6, 153.3, 145.8, 137.9, 134.1, 134.0, 133.3, 130.4, 130.3, 128.6, 128.3, 127.5, 126.1, 119.4, 113.8, 80.5, 21.7; HRMS (ESI) *m/z*: 464.0335, 466.0306 (Calcd for $C_{22}H_{16}ClNO_5SNa$ $[M+Na]^+$: 464.0335, 466.0306).

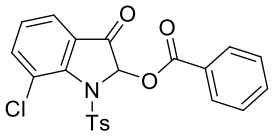
6-Chloro-1-tosylindolin-3-on-2-yl benzoate (3ia)



3ia

117 mg, 88% yield. colorless solid; mp 177–179 °C; IR (KBr): 1749, 1740, 1603, 1452, 1371, 1171, 1026 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 8.02 (d, *J* = 1.2 Hz, 1H), 7.97 (dd, *J* = 8.4, 1.2 Hz, 2H), 7.77 (ddd, *J* = 7.8, 1.8, 1.8 Hz, 2H), 7.63 (d, *J* = 8.4 Hz, 1H), 7.59 (tt, *J* = 8.4, 1.2 Hz, 1H), 7.43 (t, *J* = 7.8 Hz, 2H), 7.21–7.19 (m, 3H), 6.34 (s, 1H), 2.33 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 190.0, 164.6, 152.6, 145.7, 144.7, 134.3, 134.1, 130.4, 130.3, 128.6, 128.2, 127.5, 125.9, 125.4, 121.0, 116.0, 80.9, 21.7; HRMS (ESI) *m/z*: 464.0335, 466.0306 (Calcd for $C_{22}H_{16}ClNO_5SNa$ $[M+Na]^+$: 464.0335, 466.0306).

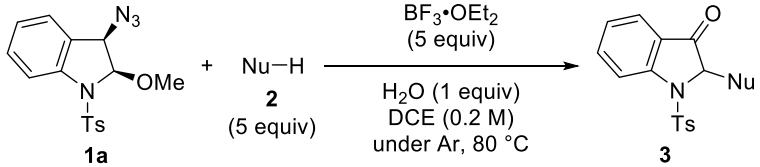
7-Chloro-1-tosylindolin-3-on-2-yl benzoate (3ja)



3ja

98.3 mg, 74% yield. purple solid; mp 156–158 °C; IR (KBr): 1748, 1726, 1601, 1470, 1356, 1169, 1018 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 8.05 (dd, *J* = 8.4, 1.2 Hz, 2H), 7.79 (ddd, *J* = 8.4, 1.8, 1.8 Hz, 2H), 7.71 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.64–7.60 (m, 2H), 7.46 (t, *J* = 7.8 Hz, 2H), 7.25 (t, *J* = 7.8 Hz, 1H), 7.18 (d, *J* = 8.4 Hz, 2H), 6.93 (s, 1H), 2.35 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 191.5, 164.6, 149.6, 144.6, 139.3, 137.2, 134.2, 130.3, 129.6, 128.8, 128.1, 127.9, 127.4, 126.8, 124.1, 123.4, 82.2, 21.7; HRMS (ESI) *m/z*: 464.0335, 466.0308 (Calcd for $C_{22}H_{16}ClNO_5SNa$ $[M+Na]^+$: 464.0335, 466.0306).

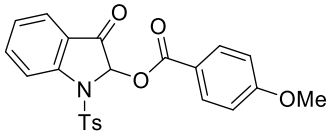
■General Procedure for the reaction of 1a with 2 (Scheme 3: carboxylic acids, alcohols, pharmaceuticals)



To a solution of **1a** (344 mg, 1 mmol) and **2** (5 mmol, 5 equiv) in DCE (5 mL, 0.2 M) was added BF_3•OEt_2 (0.63 mL, 5 mmol) or $Zn(OTf)_2$ (727 mg, 2 mmol) under Ar. The mixture was stirred at 80 °C in oil bath until the complete

disappearance of starting material and 3-azidoindole as indicated by TLC. The reaction mixture was quenched by H_2O (10 mL) and cooled to room temperature. Then, the whole was extracted with $CHCl_3$ (3 x 25 mL), washed with brine (25 mL). The organic layer was dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/10–1/5 and/or $CHCl_3$/hexane = 1/3–1/1) and/or to give **3aa–3an** and **4b–4f**. Nucleophiles **2i** and **2j** were prepared by reported methods.[S5] The other nucleophiles were used as received from commercial suppliers (Sigma-Aldrich, Kanto Chemical, TCI and Wako).

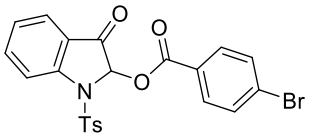
1-Tosylindolin-3-on-2-yl 4-methoxybenzoate (3ab)



3ab

371 mg, 85% yield. colorless solid; mp 178–180 °C; IR (KBr): 1736, 1719, 1609, 1458, 1368, 1175 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.98 (d, *J* = 9.0 Hz, 1H), 7.96 (d, *J* = 9.0 Hz, 2H), 7.78 (d, *J* = 7.8 Hz, 2H), 7.71–7.68 (m, 2H), 7.22 (t, *J* = 7.8 Hz, 1H), 7.20 (d, *J* = 7.8 Hz, 2H), 6.90 (d, *J* = 8.4 Hz, 2H), 6.32 (s, 1H), 3.86 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 191.6, 164.4, 164.2, 152.0, 145.4, 138.0, 134.3, 132.6, 130.2, 127.5, 125.1, 124.7, 122.6, 120.6, 115.7, 113.8, 80.5, 55.6, 21.7; HRMS (ESI) *m/z*: 460.0830 (Calcd for $C_{23}H_{19}NO_6SNa$ $[M+Na]^+$: 460.0831).

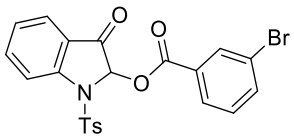
1-Tosylindolin-3-on-2-yl 4-bromobenzoate (3ac)



3ac

282 mg, 58% yield. colorless solid; mp 165–167 °C; IR (KBr): 1736, 1724, 1605, 1464, 1364, 1173, 1036 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 8.00 (d, *J* = 9.0 Hz, 1H), 7.87 (ddd, *J* = 8.4, 1.8, 1.8 Hz, 2H), 7.77 (d, *J* = 8.4 Hz, 2H), 7.73–7.69 (m, 2H), 7.58 (ddd, *J* = 8.4, 1.8, 1.8 Hz, 2H), 7.24–7.21 (m, 3H), 6.36 (s, 1H), 2.34 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 191.1, 164.1, 152.1, 145.6, 138.2, 134.1, 132.0, 131.8, 130.3, 129.3, 127.5, 127.3, 125.2, 124.9, 122.4, 115.8, 80.5, 21.7; HRMS (ESI) *m/z*: 507.9831, 509.9809 (Calcd for $C_{22}H_{16}BrNO_5SNa$ $[M+Na]^+$: 507.9830, 509.9810).

1-Tosylindolin-3-on-2-yl 3-bromobenzoate (3ad)

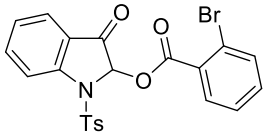


3ad

418 mg, 86% yield. colorless solid; mp 140–142 °C; IR (KBr): 1739, 1728, 1603, 1460, 1375, 1175, 1049 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 8.09 (t, *J* = 1.2 Hz, 1H), 8.01 (d, *J* = 8.4 Hz, 1H), 7.96 (d, *J* = 7.8 Hz, 1H), 7.77 (d, *J* =

8.4 Hz, 2H), 7.73–7.69 (m, 3H), 7.32 (t, J = 8.4 Hz, 1H), 7.24–7.21 (m, 3H), 6.41 (s, 1H), 2.34 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 191.0, 163.5, 152.2, 145.7, 138.3, 136.9, 134.2, 133.2, 130.3, 130.3, 130.2, 129.0, 127.5, 125.3, 124.9, 122.6, 122.3, 115.8, 80.5, 21.7; HRMS (ESI) *m/z*: 507.9832, 509.9810 (Calcd for $C_{22}H_{16}BrNO_5SNa$ $[M+Na]^+$: 507.9830, 509.9810).

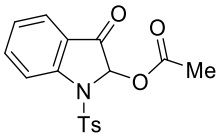
1-Tosylindolin-3-on-2-yl 2-bromobenzoate (3ae)



3ae

404 mg, 83% yield. colorless solid; mp 170–172 °C; IR (KBr): 1749, 1605, 1462, 1362, 1175, 1022 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ: 8.01 (d, J = 8.4 Hz, 1H), 7.96–7.94 (m, 1H), 7.80 (d, J = 8.4 Hz, 2H), 7.72–7.67 (m, 3H), 7.38–7.36 (m, 2H), 7.25 (d, J = 8.4 Hz, 2H), 7.22 (t, J = 7.8 Hz, 1H), 6.39 (s, 1H), 2.35 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 190.9, 163.7, 152.1, 145.7, 138.2, 134.7, 133.8, 133.6, 132.5, 130.3, 129.7, 127.6, 127.3, 125.3, 124.9, 122.9, 122.3, 115.8, 80.3, 21.7; HRMS (ESI) *m/z*: 507.9830, 509.9811 (Calcd for $C_{22}H_{16}BrNO_5SNa$ $[M+Na]^+$: 507.9830, 509.9810).

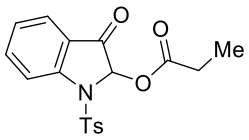
1-Tosylindolin-3-on-2-yl acetate (3af)



3af

321 mg, 93% yield. colorless solid; mp 159–161 °C; IR (KBr): 1761, 1742, 1607, 1462, 1373, 1175 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.97 (d, J = 8.4 Hz, 1H), 7.77 (d, J = 8.4 Hz, 2H), 7.68 (ddd, J = 7.8, 6.6, 1.2 Hz, 1H), 7.64 (d, J = 7.8 Hz, 1H), 7.28 (d, J = 8.4 Hz, 2H), 7.20 (t, J = 7.8 Hz, 1H), 6.19 (s, 1H), 2.38 (s, 3H), 2.18 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 191.4, 169.2, 152.1, 145.7, 138.2, 133.8, 130.3, 127.6, 125.2, 124.9, 122.3, 115.9, 79.6, 21.7, 20.7; HRMS (ESI) *m/z*: 368.0567 (Calcd for $C_{17}H_{15}NO_5SNa$ $[M+Na]^+$: 368.0569).

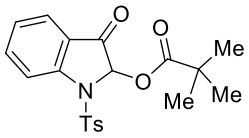
1-Tosylindolin-3-on-2-yl propionate (3ag)



3ag

331 mg, 92% yield. colorless solid; mp 129–130 °C; IR (KBr): 1736, 1605, 1462, 1371, 1173 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.97 (d, J = 7.8 Hz, 1H), 7.77 (d, J = 8.4 Hz, 2H), 7.68 (ddd, J = 8.4, 6.6, 1.8 Hz, 1H), 7.64 (d, J = 8.4 Hz, 1H), 7.28 (d, J = 8.4 Hz, 2H), 7.19 (t, J = 8.4 Hz, 1H), 6.16 (s, 1H), 2.52–2.40 (m, 2H), 2.38 (s, 3H), 1.17 (t, J = 7.2 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 191.5, 172.7, 152.1, 145.6, 138.1, 133.8, 130.3, 127.6, 125.1, 124.9, 122.3, 115.9, 79.7, 27.3, 21.7, 8.7; HRMS (ESI) *m/z*: 382.0725 (Calcd for $C_{18}H_{17}NO_5SNa$ $[M+Na]^+$: 382.0725).

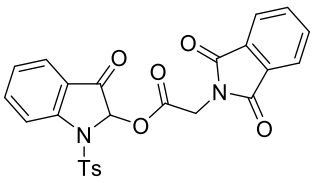
1-Tosylindolin-3-on-2-yl pivalate (3ah)



3ah

311 mg, 80% yield. colorless oil; IR (KBr): 1740, 1605, 1462, 1371, 1175 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.98 (d, *J* = 7.8 Hz, 1H), 7.77 (ddd, *J* = 8.4, 1.8, 1.8 Hz, 2H), 7.68 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 7.64 (d, *J* = 7.8 Hz, 1H), 7.27 (d, *J* = 8.4 Hz, 2H), 7.20 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 6.11 (s, 1H), 2.37 (s, 3H), 1.27 (s, 9H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 191.5, 176.7, 152.1, 145.6, 138.0, 133.7, 130.3, 127.6, 125.0, 124.9, 122.5, 116.0, 79.9, 38.9, 27.0, 21.7; HRMS (ESI) *m/z*: 410.1039 (Calcd for $C_{20}H_{21}NO_5SNa$ $[M+Na]^+$: 410.1038).

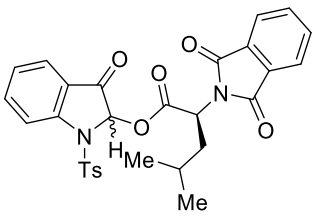
1-Tosylindolin-3-on-2-yl 2-(*N*,*N*-phthaloylamino)acetate (3ai)



3ai

0.3 mmol scale: 91.0 mg, 62% yield. colorless oil; IR (KBr): 1771, 1724, 1605, 1464, 1369, 1175 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.91 (d, *J* = 8.4 Hz, 1H), 7.89–7.88 (m, 2H), 7.78 (ddd, *J* = 8.4, 1.8, 1.8 Hz, 2H), 7.74–7.73 (m, 2H), 7.67 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 7.64 (d, *J* = 8.4 Hz, 1H), 7.30 (d, *J* = 8.4 Hz, 2H), 7.19 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 6.23 (s, 1H), 4.57 (d, *J* = 18.0 Hz, 1H), 4.52 (d, *J* = 18.0 Hz, 1H), 2.39 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 190.2, 167.1, 166.1, 152.1, 145.8, 138.2, 134.4, 134.0, 132.0, 130.4, 127.6, 125.3, 124.9, 123.8, 122.1, 115.8, 80.3, 38.7, 21.7; HRMS (ESI) *m/z*: 513.0732 (Calcd for $C_{25}H_{18}N_2O_7SNa$ $[M+Na]^+$: 513.0732).

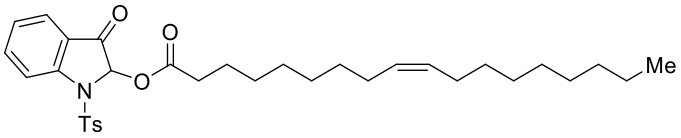
1-Tosylindolin-3-on-2-yl (2*S*)-2-(*N*,*N*-phthaloylamino)-4-methylpentanoate (3aj, mixture of diastereomer)



3aj: mixture of diastereomer (dr = 1:1)

0.3 mmol scale: 118 mg, 72% yield. colorless oil; IR (KBr): 1763, 1740, 1717, 1605, 1464, 1387, 1175 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.89–7.88 (m, 1H), 7.86–7.82 (m, 2H), 7.77–7.73 (m, 2H), 7.72–7.68 (m, 2H), 7.66–7.58 (m, 2H), 7.28–7.26 (m, 2H), 7.19–7.13 (m, 1H), 6.19 and 6.16 (s, 1H), 5.04–5.00 (m, 1H), 2.46–2.40 (m, 1H), 2.36 (s, 3H), 1.96–1.81 (m, 1H), 1.50–1.43 (m, 1H), 0.93–0.85 (m, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 190.4, 190.3, 168.4, 167.4, 167.3, 152.0, 145.7, 145.7, 138.1, 134.3, 134.3, 134.1, 134.0, 131.8, 130.4, 130.4, 127.6, 127.5, 125.2, 125.2, 124.9, 123.7, 123.7, 122.3, 122.2, 115.8, 80.6, 80.4, 50.7, 50.5, 37.0, 36.8, 25.1, 23.2, 23.2, 21.7, 21.1, 21.0; HRMS (ESI) *m/z*: 569.1358 (Calcd for $C_{29}H_{26}N_2O_7SNa$ $[M+Na]^+$: 569.1358).

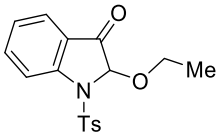
1-Tosylindolin-3-on-2-yl oleate (3ak)



3ak

0.3 mmol scale: 66.9 mg, 39% yield. colorless oil; IR (KBr): 1740, 1605, 1462, 1373, 1177 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.97 (d, *J* = 8.4 Hz, 1H), 7.77 (d, *J* = 8.4 Hz, 2H), 7.68 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 7.64 (d, *J* = 7.8 Hz, 1H), 7.28 (d, *J* = 8.4 Hz, 2H), 7.21 (t, *J* = 7.8 Hz, 1H), 6.16 (s, 1H), 5.37–5.32 (m, 2H), 2.43–2.37 (m, 2H), 2.38 (s, 3H), 2.01–1.94 (m, 4H), 1.69–1.62 (m, 2H), 1.35–1.25 (m, 20H), 0.86 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 191.5, 172.0, 152.1, 145.6, 138.0, 133.9, 130.3, 130.1, 129.9, 127.6, 125.1, 124.8, 122.4, 115.9, 79.7, 33.9, 32.0, 29.9, 29.8, 29.6, 29.4, 29.2, 29.2, 29.0, 27.3, 27.3, 24.6, 22.8, 21.7, 14.2; HRMS (ESI) *m/z*: 590.2916 (Calcd for $C_{33}H_{45}NO_5SNa$ $[M+Na]^+$: 590.2916).

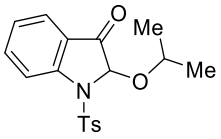
2-Ethoxy-1-tosylindolin-3-one (4b)



4b

199 mg, 60% yield. colorless solid; mp 94–96 °C; IR (KBr): 1734, 1605, 1460, 1364, 1173 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.78 (d, *J* = 8.4 Hz, 1H), 7.75 (ddd, *J* = 8.4, 1.8, 1.8 Hz, 2H), 7.62 (ddd, *J* = 7.8, 6.6, 1.2 Hz, 1H), 7.60 (d, *J* = 7.8 Hz, 1H), 7.24 (d, *J* = 7.8 Hz, 2H), 7.14 (ddd, *J* = 8.4, 7.8, 0.6 Hz, 1H), 5.17 (s, 1H), 3.86–3.81 (m, 2H), 2.36 (s, 3H), 1.23 (t, *J* = 6.6 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 194.6, 152.4, 145.1, 138.0, 135.1, 130.0, 127.4, 125.1, 124.5, 122.9, 116.1, 88.2, 64.8, 21.7, 15.1; HRMS (ESI) *m/z*: 354.0776 (Calcd for $C_{17}H_{17}NO_4SNa$ $[M+Na]^+$: 354.0776).

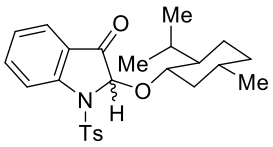
2-*iso*-Propoxy-1-tosylindolin-3-one (4c)



4c

214 mg, 62% yield. colorless oil; IR (KBr): 1734, 1605, 1464, 1362, 1173 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.75 (ddd, *J* = 7.8, 1.8, 1.8 Hz, 2H), 7.69 (d, *J* = 7.8 Hz, 1H), 7.61–7.58 (m, 2H), 7.24 (d, *J* = 7.8 Hz, 2H), 7.13 (ddd, *J* = 7.8, 6.6, 1.2 Hz, 1H), 5.20 (s, 1H), 4.36 (sep, *J* = 6.0 Hz, 1H), 2.37 (s, 3H), 1.33 (d, *J* = 6.0 Hz, 3H), 1.38 (d, *J* = 6.0 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 194.8, 152.2, 144.9, 137.8, 135.3, 130.0, 127.4, 125.2, 124.4, 122.8, 116.1, 87.0, 73.3, 23.0, 22.7, 21.7; HRMS (ESI) *m/z*: 368.0933 (Calcd for $C_{18}H_{19}NO_4SNa$ $[M+Na]^+$: 368.0933).

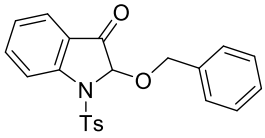
2-(2-Isopropyl-5-methylcyclohexan-1-yl)oxy-1-tosylindolin-3-one (4d, mixture of diastereomer)



4d: mixture of diastereomer (dr = 3:1)

142 mg, 32% yield. colorless oil; IR (KBr): 1736, 1605, 1464, 1369, 1173 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.80–7.55 (m, 5H), 7.23–7.13 (m, 3H), 5.25 and 5.23 (s, 1H), 4.03–3.95 (m, 1H), 2.36 and 2.34 (s, 3H), 2.29–2.25 (m, 1H), 2.14–2.08 (m, 1H), 1.68–1.61 (m, 2H), 1.55–1.45 (m, 2H), 1.28–1.23 (m, 1H), 1.09–0.74 (m, 11H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 195.1, 194.6, 152.5, 152.4, 145.0, 144.8, 137.7, 137.6, 135.4, 134.4, 130.0, 129.9, 127.6, 127.3, 125.3, 125.2, 124.5, 124.1, 123.1, 118.1, 116.54, 86.2, 85.6, 78.6, 78.3, 48.8, 48.1, 40.8, 40.4, 34.4, 31.7, 31.5, 25.3, 25.0, 23.2, 22.5, 22.4, 21.6, 21.2, 21.1, 16.0, 16.0; HRMS (ESI) *m/z*: 464.1872 (Calcd for $C_{25}H_{31}NO_4SNa$ $[M+Na]^+$: 464.1872).

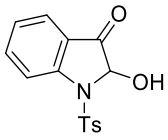
2-Benzyloxy-1-tosylindolin-3-one (4f)



4f

237 mg, 59% yield. colorless oil; IR (KBr): 1732, 1605, 1462, 1364, 1173 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.81 (d, J = 8.4 Hz, 1H), 7.69 (d, J = 8.4 Hz, 2H), 7.65–7.62 (m, 2H), 7.41–7.40 (m, 2H), 7.36–7.31 (m, 3H), 7.18–7.15 (m, 3H), 5.28 (s, 1H), 4.90 (d, J = 10.2 Hz, 1H), 4.84 (d, J = 10.2 Hz, 1H), 2.34 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 194.3, 152.4, 145.1, 138.0, 136.5, 134.9, 130.0, 128.6, 128.5, 128.2, 127.5, 125.2, 124.6, 122.9, 116.3, 87.6, 71.0, 21.7; HRMS (ESI) *m/z*: 416.0933 (Calcd for $C_{22}H_{19}NO_4SNa$ $[M+Na]^+$: 416.0933).

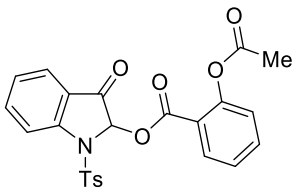
2-Hydroxy-1-tosylindolin-3-one (4g)



4g

107 mg, 32% yield. colorless needle; mp 165–167 °C; IR (KBr): 3480, 1724, 1605, 1462, 1358, 1161 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.81 (ddd, J = 7.8, 1.8, 1.8 Hz, 2H), 7.72 (d, J = 8.4 Hz, 1H), 7.67 (d, J = 8.4 Hz, 1H), 7.64 (ddd, J = 8.4, 7.2, 1.2 Hz, 1H), 7.29 (d, J = 7.8 Hz, 2H), 7.81 (t, J = 8.4 Hz, 1H), 5.36 (s, 1H), 3.94 (s, 1H), 2.39 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 192.9, 151.3, 145.5, 138.3, 134.4, 130.3, 127.5, 125.5, 124.5, 122.1, 115.0, 82.7, 21.7; HRMS (ESI) *m/z*: 326.0462 (Calcd for $C_{13}H_{13}NO_4SNa$ $[M+Na]^+$: 326.0463).

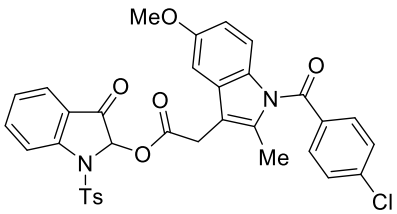
1-Tosylindolin-3-on-2-yl 2-acetoxybenzoate (3al)



3al

146 mg, 31% yield. colorless solid; mp 137–139 °C; IR (KBr): 1771, 1744, 1736, 1605, 1464, 1369, 1175 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.96 (d, *J* = 8.4 Hz, 1H), 7.95 (d, *J* = 8.4 Hz, 1H), 7.77 (d, *J* = 8.4 Hz, 2H), 7.69 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 7.68 (d, *J* = 8.4 Hz, 1H), 7.58 (t, *J* = 7.8 Hz, 1H), 7.28 (t, *J* = 7.8 Hz, 1H), 7.22 (d, *J* = 7.8 Hz, 2H), 7.21 (t, *J* = 7.2 Hz, 1H), 7.13 (d, *J* = 7.8 Hz, 1H), 6.41 (s, 1H), 2.33 (s, 3H), 2.30 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 190.9, 169.5, 162.6, 152.2, 151.1, 145.6, 138.1, 134.6, 134.1, 132.3, 130.3, 127.5, 126.1, 125.2, 124.9, 124.2, 122.4, 122.0, 115.8, 80.1, 21.7, 21.0; HRMS (ESI) *m/z*: 488.0781 (Calcd for $C_{24}H_{19}NO_7SNa$ $[M+Na]^+$: 488.0780).

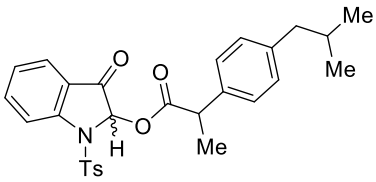
1-Tosylindolin-3-on-2-yl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methylindol-3-yl)acetate (3am)



3am

0.3 mmol scale, reaction with $Zn(OTf)_2$: 55.4 mg, 29% yield. colorless oil; IR (KBr): 1761, 1738, 1605, 1462, 1364, 1175, 1089 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.93 (d, *J* = 8.4 Hz, 1H), 7.73 (d, *J* = 8.4 Hz, 2H), 7.69–7.64 (m, 4H), 7.45 (d, *J* = 8.4 Hz, 2H), 7.23–7.19 (m, 3H), 6.99 (d, *J* = 2.4 Hz, 1H), 6.95 (d, *J* = 8.4 Hz, 1H), 6.68 (dd, *J* = 8.4, 2.4 Hz, 1H), 6.24 (s, 1H), 3.85 (d, *J* = 17.4 Hz, 1H), 3.85 (s, 3H), 3.83 (d, *J* = 17.4 Hz, 1H), 2.36 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 191.1, 169.3, 168.4, 156.3, 152.1, 145.7, 139.3, 138.2, 136.2, 134.0, 133.6, 131.4, 130.9, 130.6, 130.4, 129.2, 127.6, 125.2, 125.0, 122.2, 115.8, 115.1, 112.5, 111.6, 100.8, 79.9, 55.8, 29.8, 21.7, 13.6; HRMS (ESI) *m/z*: 665.1124, 667.1097 (Calcd for $C_{34}H_{27}ClN_2O_7SNa$ $[M+Na]^+$: 665.1125, 667.1096).

1-Tosylindolin-3-on-2-yl 2-(4-isobutylphenyl)propanoate (3an, mixture of diastereomer)



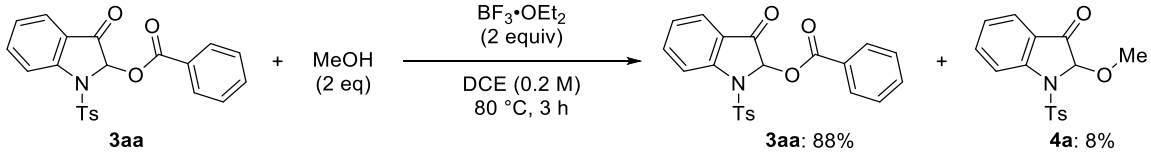
3an: mixture of diastereomer (dr = 1:1)

0.3 mmol scale: 116 mg, 78% yield. colorless oil; IR (KBr): 1757, 1740, 1605, 1464, 1373, 1175 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ: 7.91–7.85 (m, 1H), 7.74–7.59 (m, 4H), 7.25–7.10 (m, 7H), 6.21–6.12 (s, 1H), 3.86–3.78 (m, 1H), 2.44–2.36 (m, 6H), 1.86–1.81 (m, 1H), 1.59–1.50 (m, 2H), 0.89–0.87 (m, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 191.2, 191.1, 173.2, 172.9, 152.1, 152.0, 145.5, 145.4, 140.8, 140.8, 138.0, 137.9, 136.7, 134.0, 133.8, 130.32, 130.2,

129.5, 129.5, 127.6, 127.5, 127.5, 125.1, 125.0, 124.8, 124.7, 122.5, 122.4, 115.9, 115.8, 80.2, 79.8, 45.2, 44.9, 44.8, 30.2, 22.5, 21.7, 19.0, 18.8; HRMS (ESI) *m/z*: 514.1665 (Calcd for $C_{28}H_{29}NO_5SNa$ $[M+Na]^+$: 514.1664).

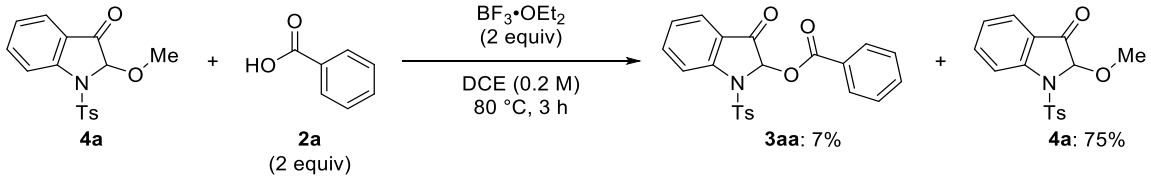
■Scrambling experiments (Scheme 4a)

Reaction of 3aa with MeOH



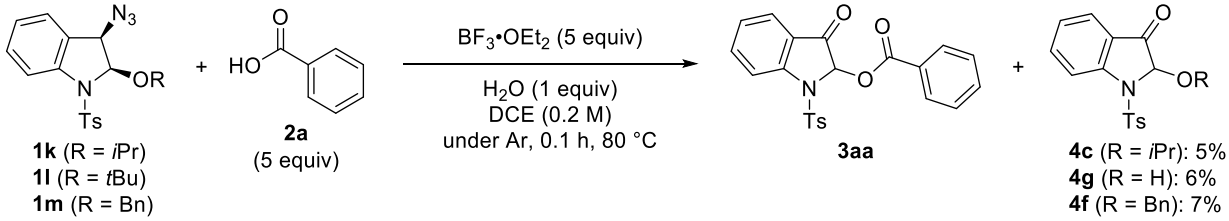
To a solution of **3aa** (40.7 mg, 0.1 mmol) and MeOH (8 μL, 0.2 mmol) in DCE (0.5 mL, 0.2 M) was added BF_3•OEt_2 (25 μL, 0.2 mmol) under Ar. The reaction mixture was quenched by H_2O (2 mL) and cooled to room temperature. Then, the whole was extracted with $CHCl_3$ (3 x 10 mL), washed with brine (10 mL). The organic layer was dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/10–1/5) to give **3aa** (35.8 mg, 88%) and **4a** (2.5 mg, 8%).

Reaction of 4a with benzoic acid (2a)



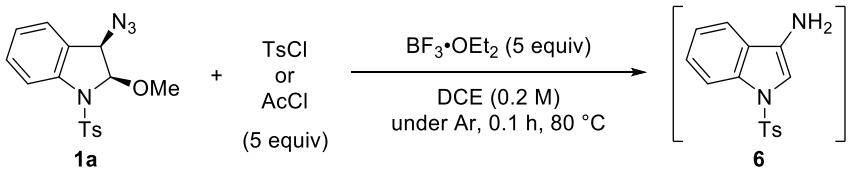
To a solution of **4a** (31.7 mg, 0.1 mmol) and **2a** (24.4 mg, 0.2 mmol) in DCE (0.5 mL, 0.2 M) was added BF_3•OEt_2 (25 μL, 0.2 mmol) under Ar. The reaction mixture was quenched by H_2O (2 mL) and cooled to room temperature. Then, the whole was extracted with $CHCl_3$ (3 x 10 mL), washed with brine (10 mL). The organic layer was dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/10–1/5) to give **3aa** (2.7 mg, 7%) and **4a** (23.7 mg, 75%).

■Investigation of alkoxy substituents of AZINs (Scheme 4b)



To a solution of **1** (0.3 mmol) and **2a** (183 mg, 1.5 mmol) in DCE (1.5 mL, 0.2 M) was added BF_3•OEt_2 (0.19 mL, 1.5 mmol) under Ar. The mixture was stirred at 80 °C in oil bath until the complete disappearance of starting material as indicated by TLC. The reaction mixture was quenched by H_2O (3 mL) and cooled to room temperature. Then, the whole was extracted with $CHCl_3$ (3 x 20 mL), washed with brine (20 mL). The organic layer was dried over Na_2SO_4 and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/10–1/2) to give **3aa** and **4c–4f**.

■Attempt of capturing the compounds derived from plausible reaction intermediates



To a solution of **1** (0.1 mmol) and TsCl or AcCl (0.5 mmol) in DCE (0.5 mL, 0.2 M) was added BF_3•OEt_2 (63 μL, 0.5 mmol) under Ar. The mixture was stirred at 80 °C in oil bath for 0.1 h. Then, the samples for mass spectrometry measurement were carefully prepared under anhydrous conditions and measured HRMS to find a peak of *m/z*: 287.0854 (use of TsCl) and 287.0849 (use of AcCl) (Calcd for $C_{15}H_{15}N_2O_2S$ $[M+H]^+$: 287.0854).

4. Supplementary References

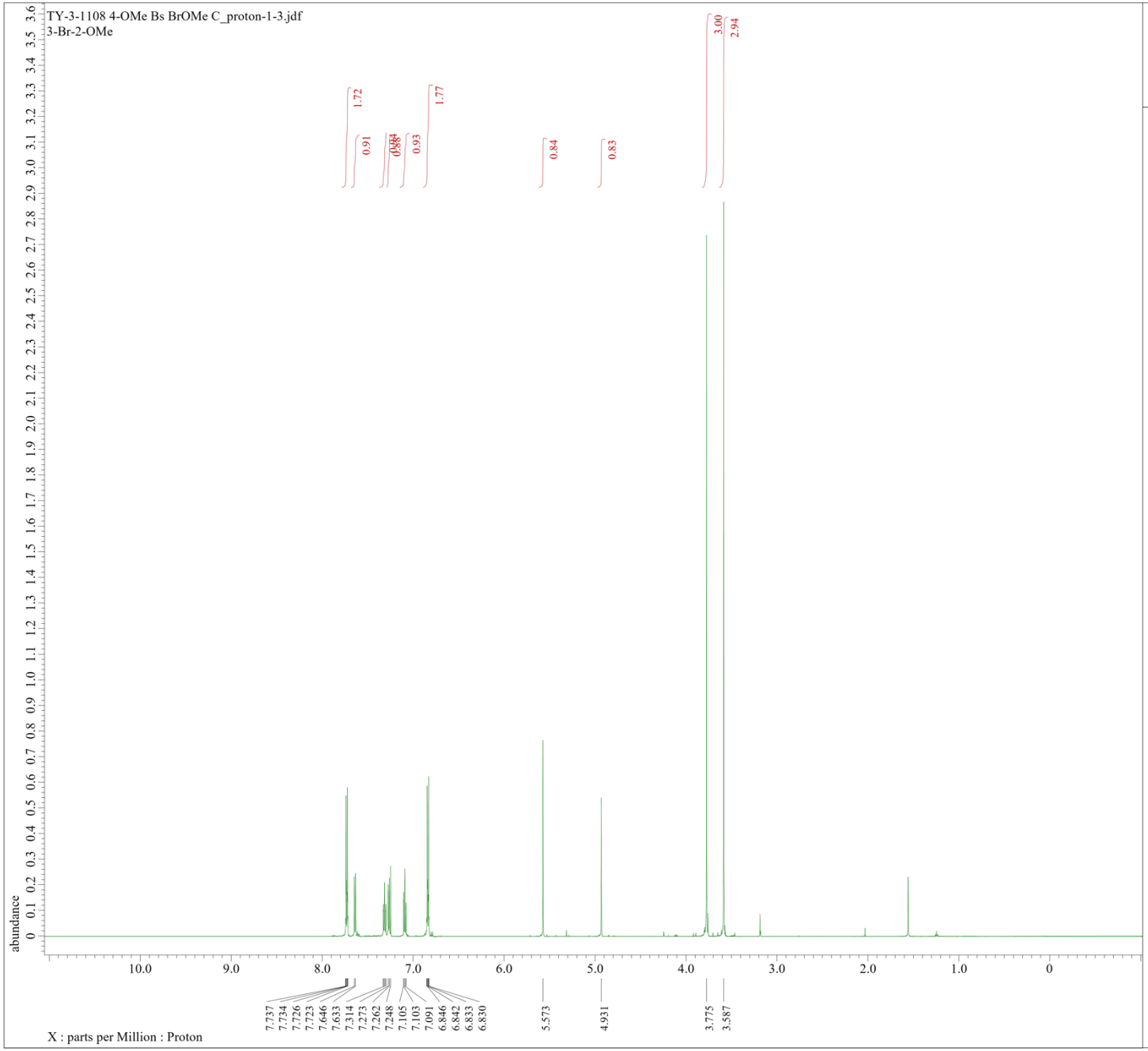
[S1] *N*-tosylation, *N*-benzenesulfonylation, and *N*-2-nosylation: Hodson, H. F.; Madge, D. J.; Slawin, A. N. Z.; Widdowson, D. A.; Williams, D. J. *Tetrahedron* **1994**, *50*, 1899–1906.

[S2] *N*-mesylation: Laha, J. K.; Dayal, N. *Org., Lett.* **2015**, *17*, 4742–4745.

[S3] Synthesis of 3-bromo-2-alkoxyindolines: Abe, T.; Kosaka, Y.; Kawasaki, T.; Ohata, Y.; Yamashiro, T.; Yamada, K. *Chem. Pharm. Bull.* **2020**, *68*, 555–558.

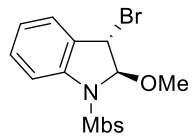
[S4] Synthesis of 3-azido-2-alkoxyindolines: Yamashiro, T.; Abe, T.; Tanioka, M.; Kamino, S.; Sawada, D. *Chem. Commun.* **2021**, *57*, 13381–13384.

[S5] *N*-Phthaloylation of amino acids: Kanyiva, K. S.; Makino, S.; Shibata, T. *Chem. Asian J.* **2018**, *13*, 496–499.

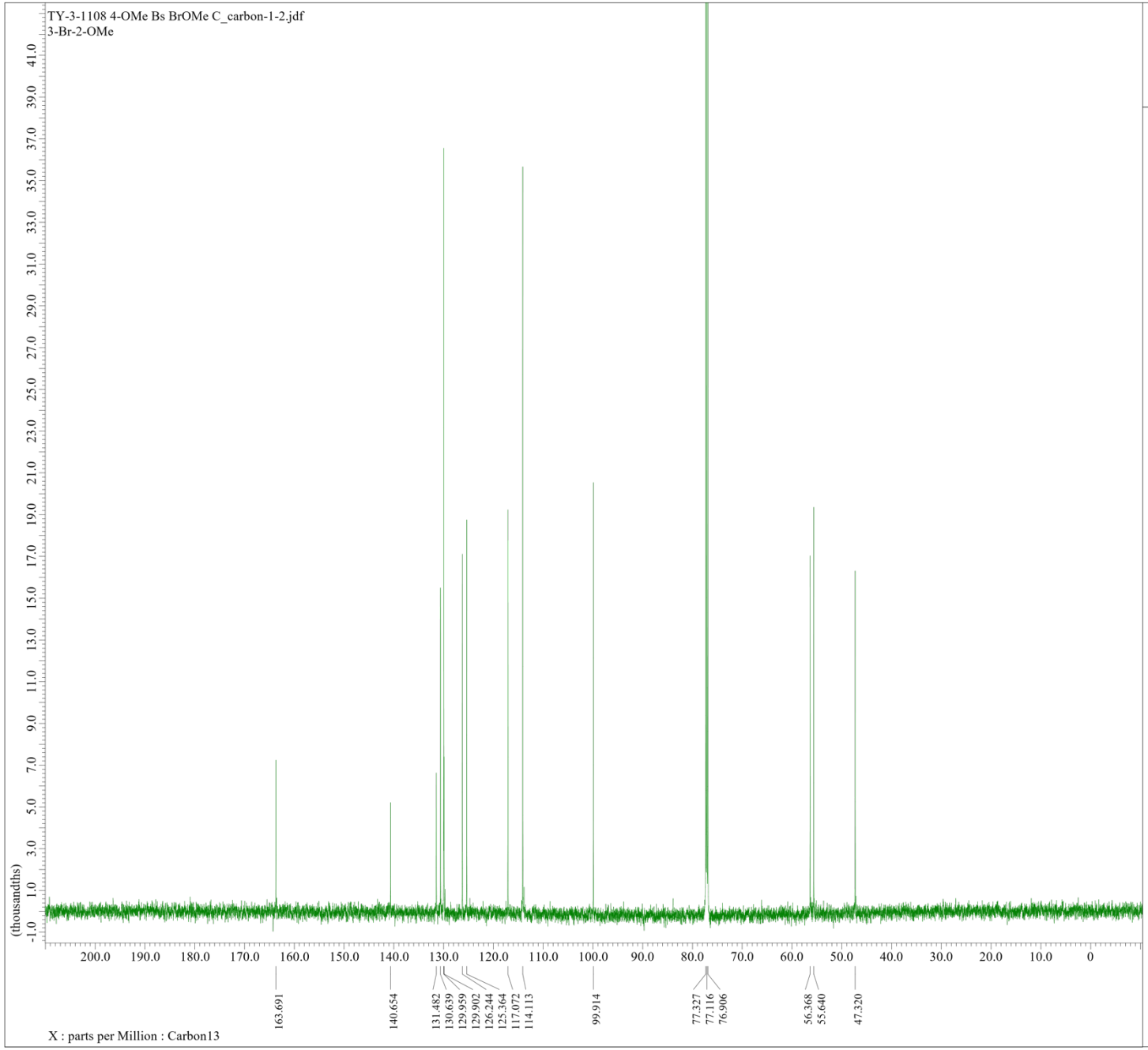


JEOL

Filename	= TY-3-1108 4-OMe Bs Br
Author	= delta
Experiment	= proton.jxp
Sample_Id	= TY-3-1108 4-OMe Bs Br
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 8-NOV-2021 16:53:57
Revision_Time	= 8-NOV-2021 18:06:58
Comment	= 3-Br-2-OMe
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (600[M
X_Acq_Duration	= 2.18103808[s]
X_Domain	= Proton
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.45849727[Hz]
X_Sweep	= 15.02403846[kHz]
X_Sweep_Clipped	= 12.01923077[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 5[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 21.7[dC]
X_90_Width	= 9.4[us]
X_Acq_Time	= 2.18103808[s]
X_Angle	= 45[deg]
X_Atn	= 8.1[dB]
X_Pulse	= 4.7[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 100
Dante_Presat	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\JEOL
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180
Presat_Time	= 1[s]
Presat_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 3.18103808[s]

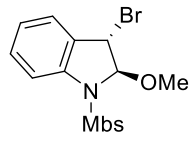


S1c

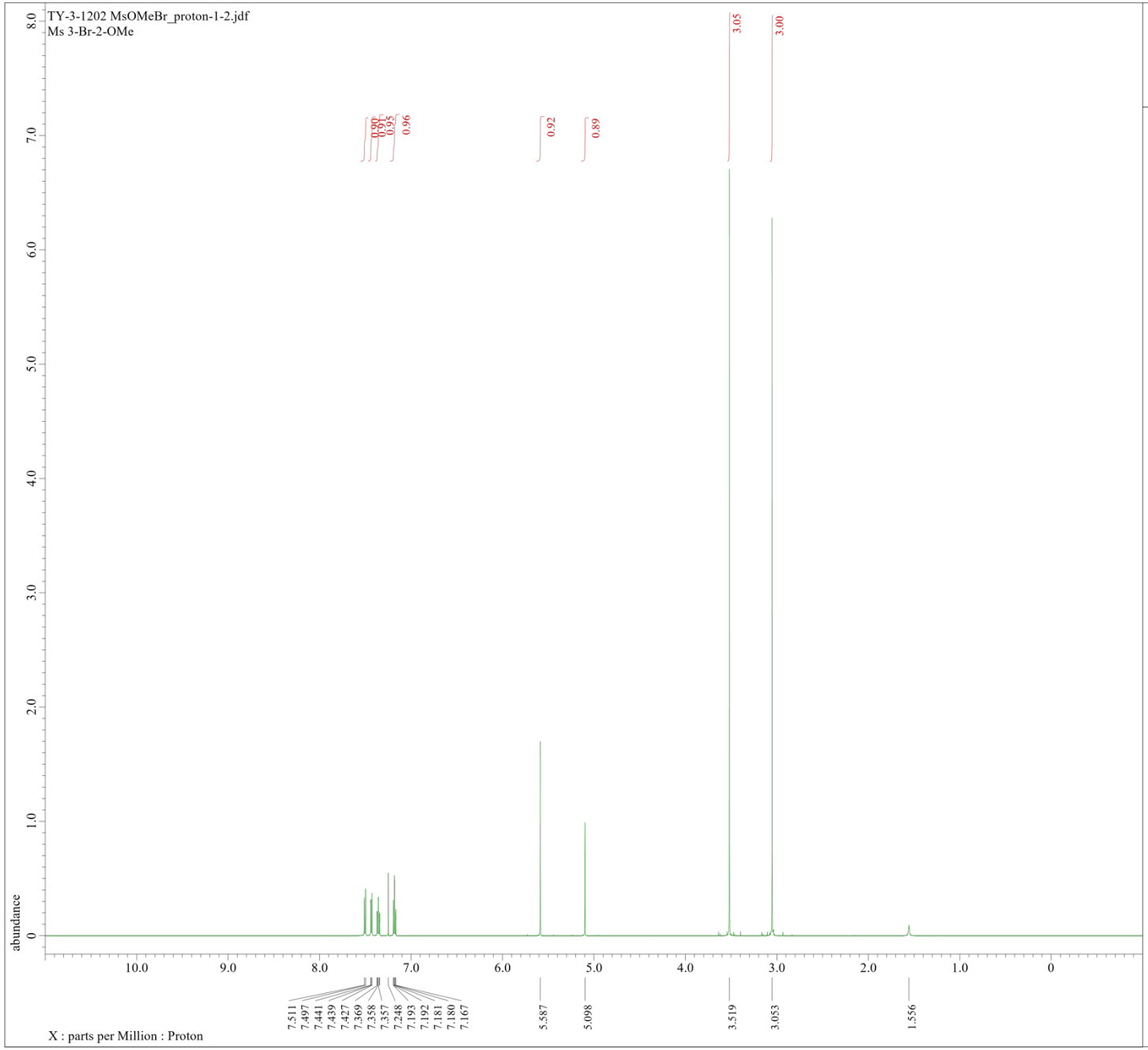


JEOL

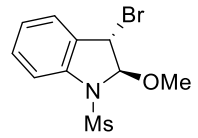
Filename	= TY-3-1108 4-OMe Bs
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1108 4-OMe Bs
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 8-NOV-2021 16:54:
Revision_Time	= 8-NOV-2021 18:06:
Comment	= 3-Br-2-OMe
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 15[us]
Clipped	= FALSE
Scans	= 99
Total_Scans	= 99
Relaxation_Delay	= 1[s]
Recvr_Gain	= 56
Temp_Get	= 21.7[dC]
X_90_Width	= 9.6[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 11[dB]
X_Pulse	= 3.2[us]
Irr_Atn_Dec	= 26.254[dB]
Irr_Atn_Dec_Calc	= 26.254[dB]
Irr_Atn_Dec_Default_Calc	= 26.254[dB]
Irr_Atn_Noe	= 26.254[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]



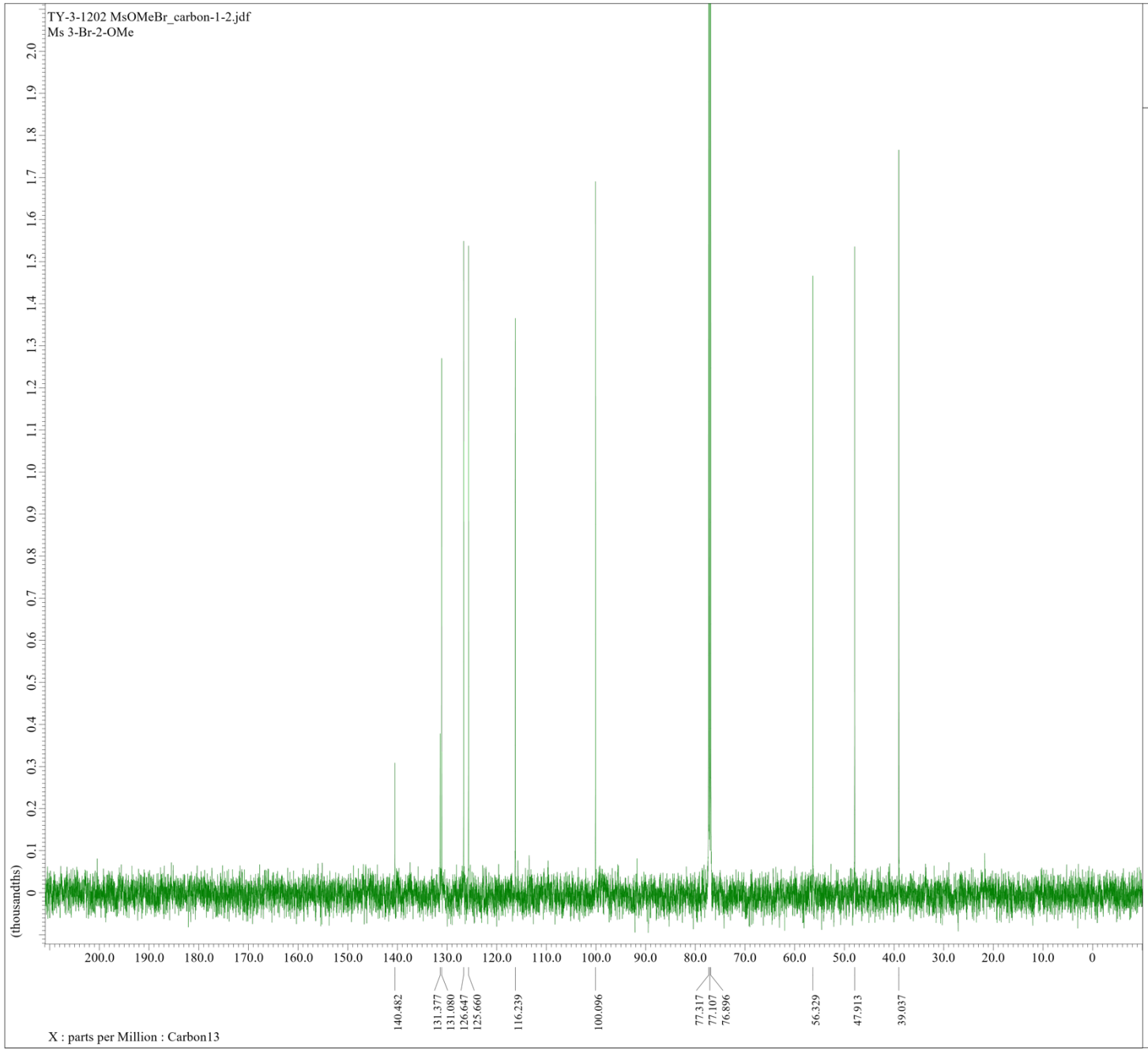
S1c




```
Filename              = TY-3-1202 MsOMeBr_pro
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-3-1202 MsOMeBr
Solvent               = CHLOROFORM-D
Actual_Start_Time     =  2-DEC-2021 13:31:03
Revision_Time         =  2-DEC-2021 13:55:13

Comment               = Ms 3-Br-2-OMe
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8

Relaxation_Delay      = 1[s]
Recvr_Gain            = 46
Temp_Get              = 21[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```

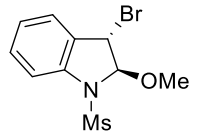


S1d

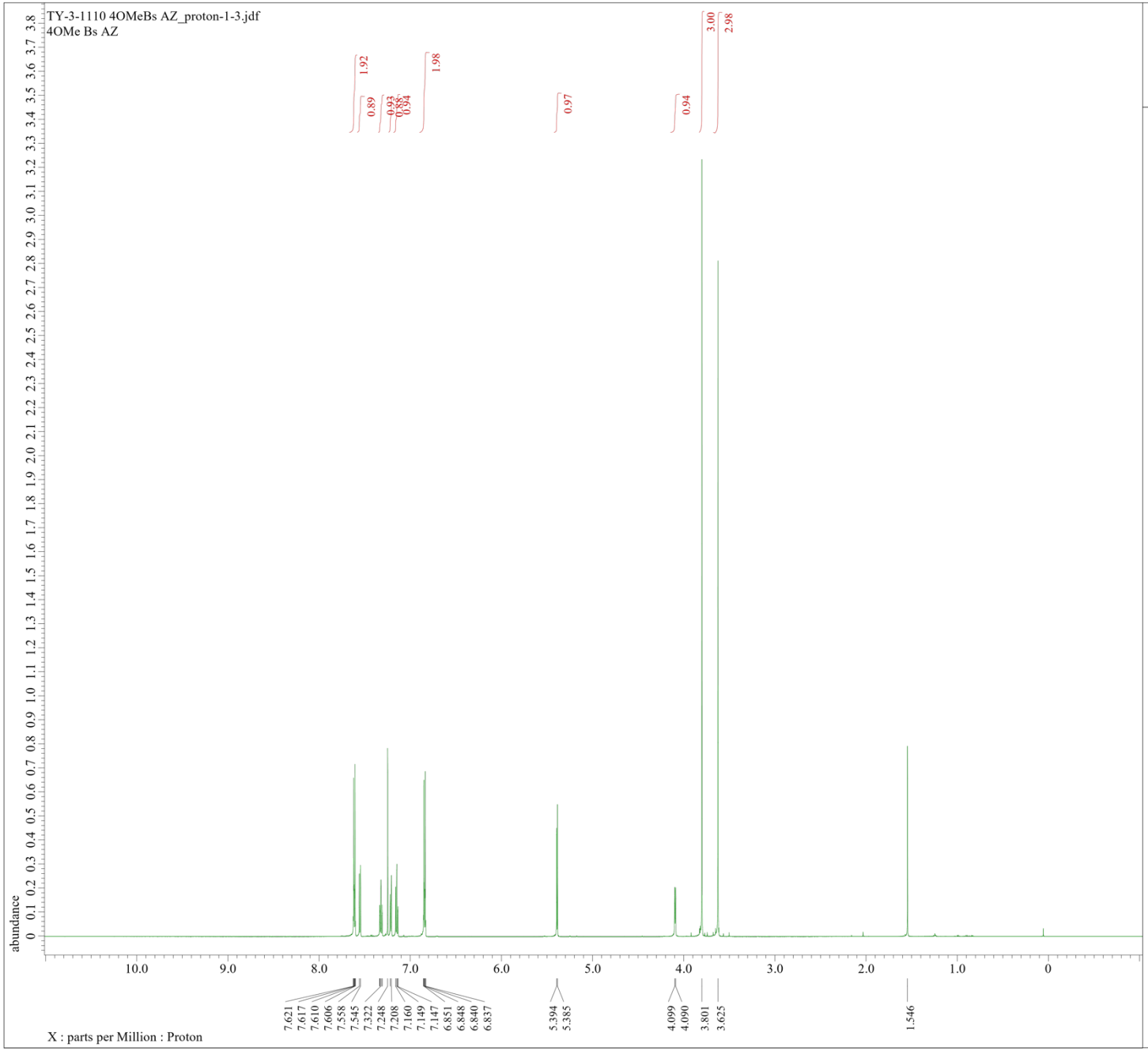


JEOL

Filename	= TY-3-1202 MsOMeBr_
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1202 MsOMeBr
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 2-DEC-2021 13:33:
Revision_Time	= 2-DEC-2021 13:56:
Comment	= Ms 3-Br-2-OMe
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 237
Total_Scans	= 237
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 21[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]



S1d



JEOL

```
Filename              = TY-3-1110 4OMeBs AZ_p
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-3-1110 4OMeBs AZ
Solvent               = CHLOROFORM-D
Actual_Start_Time     = 10-NOV-2021 15:45:43
Revision_Time         = 10-NOV-2021 16:05:44

Comment               = 4OMe Bs AZ
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 5[us]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8

Relaxation_Delay      = 1[s]
Recvr_Gain            = 46
Temp_Get              = 21.7[dC]
X_90_Width            = 9.4[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 4.7[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
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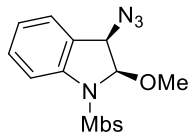

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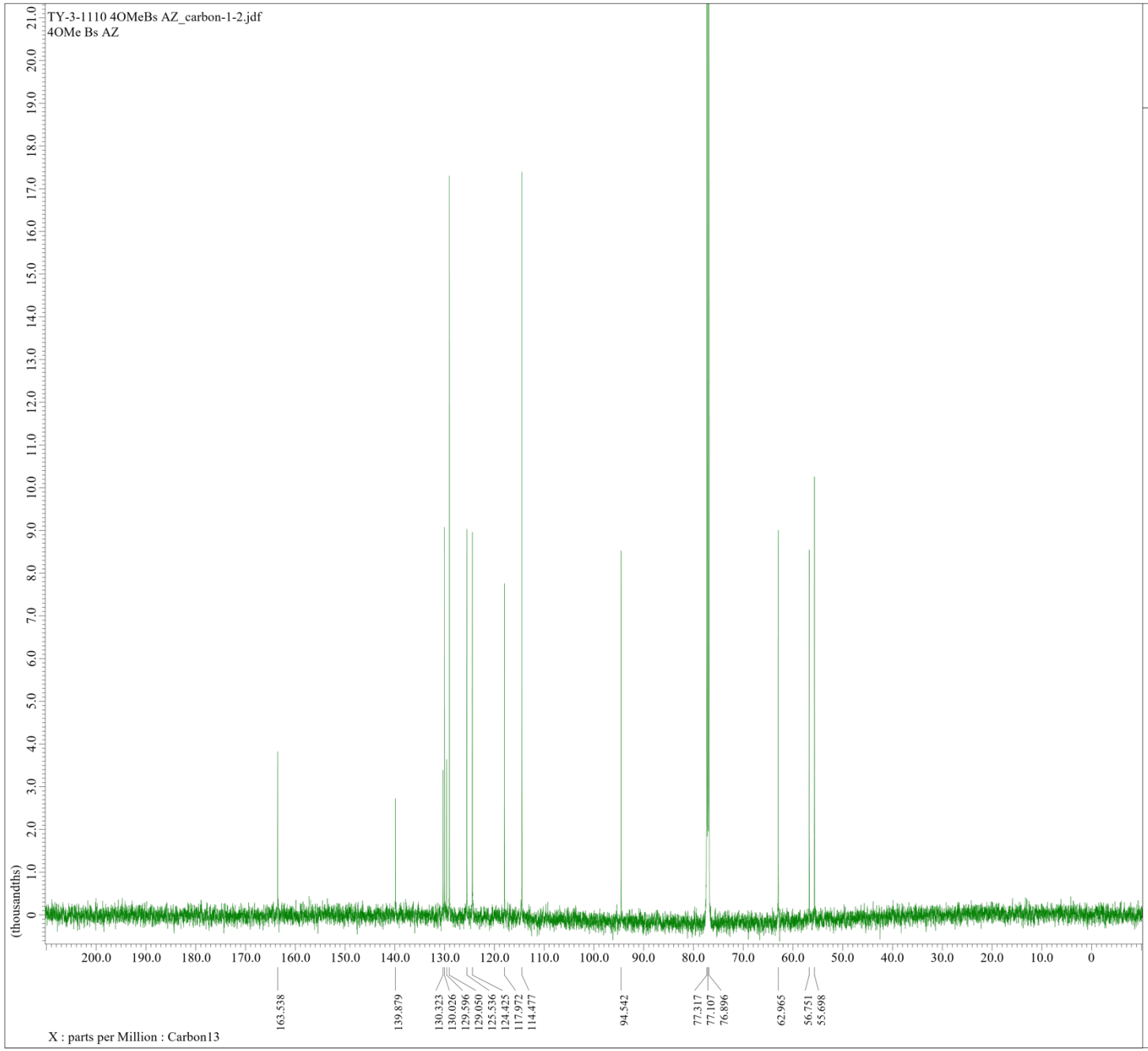
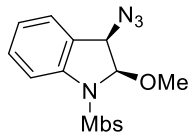


JEOL

Filename	= TY-3-1110 4OMeBs A
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1110 4OMeBs A
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 10-NOV-2021 16:05:
Revision_Time	= 10-NOV-2021 16:12:
Comment	= 4OMe Bs AZ
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 15[us]
Clipped	= FALSE
Scans	= 256
Total_Scans	= 256
Relaxation_Delay	= 1[s]
Recvr_Gain	= 56
Temp_Get	= 21.6[dC]
X_90_Width	= 9.6[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 11[dB]
X_Pulse	= 3.2[us]
Irr_Atn_Dec	= 26.254[dB]
Irr_Atn_Dec_Calc	= 26.254[dB]
Irr_Atn_Dec_Default_Calc	= 26.254[dB]
Irr_Atn_Noe	= 26.254[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]



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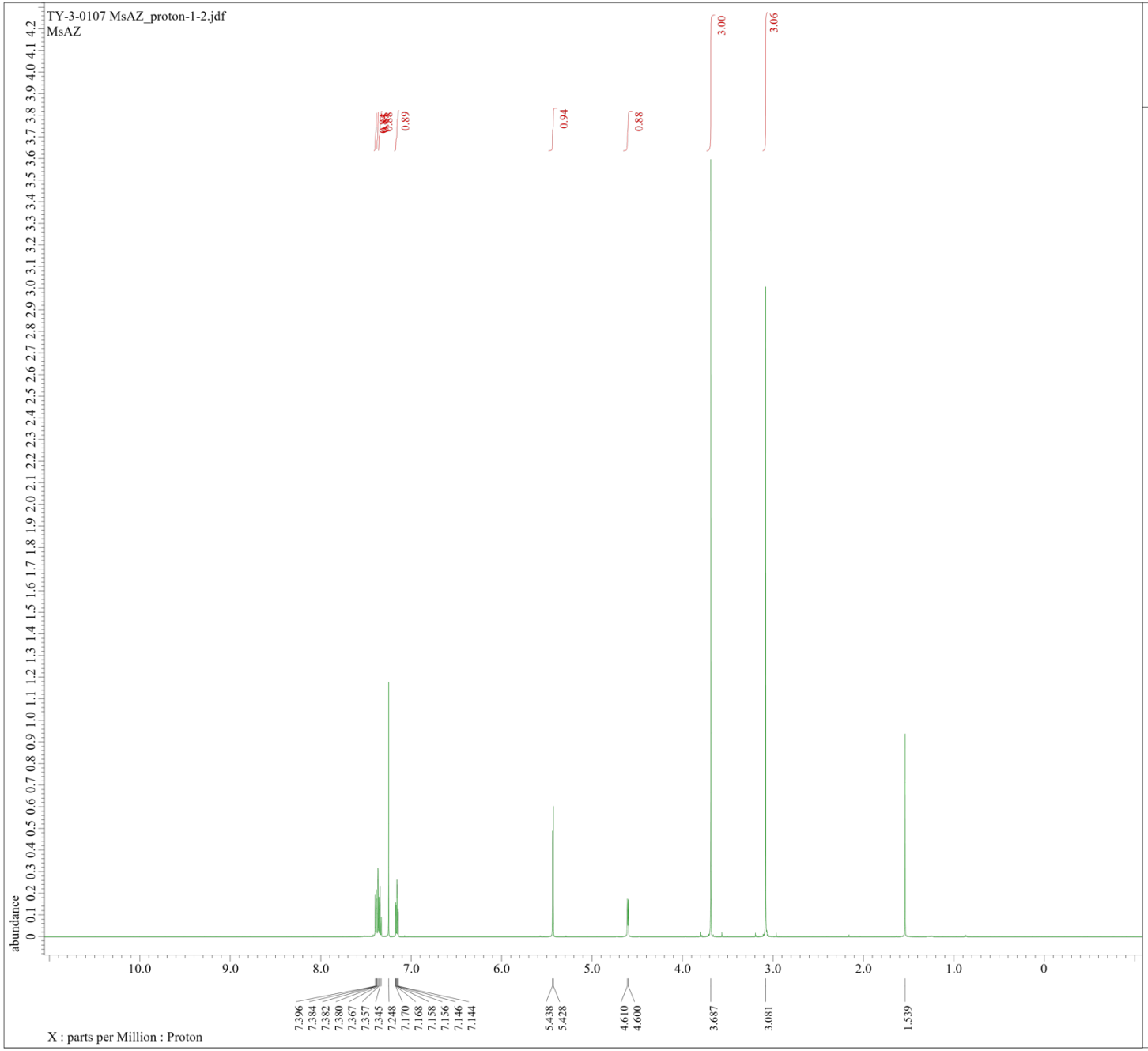

JEOL

```
Filename              = TY-3-0107 MsAZ_proton
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-3-0107 MsAZ
Solvent               = CHLOROFORM-D
Actual_Start_Time     =  7-JAN-2022 13:30:28
Revision_Time         =  7-JAN-2022 13:55:01

Comment               = MsAZ
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8

Relaxation_Delay      = 1[s]
Recvr_Gain            = 56
Temp_Get              = 20.9[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
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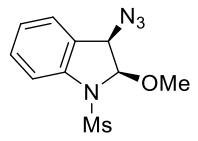
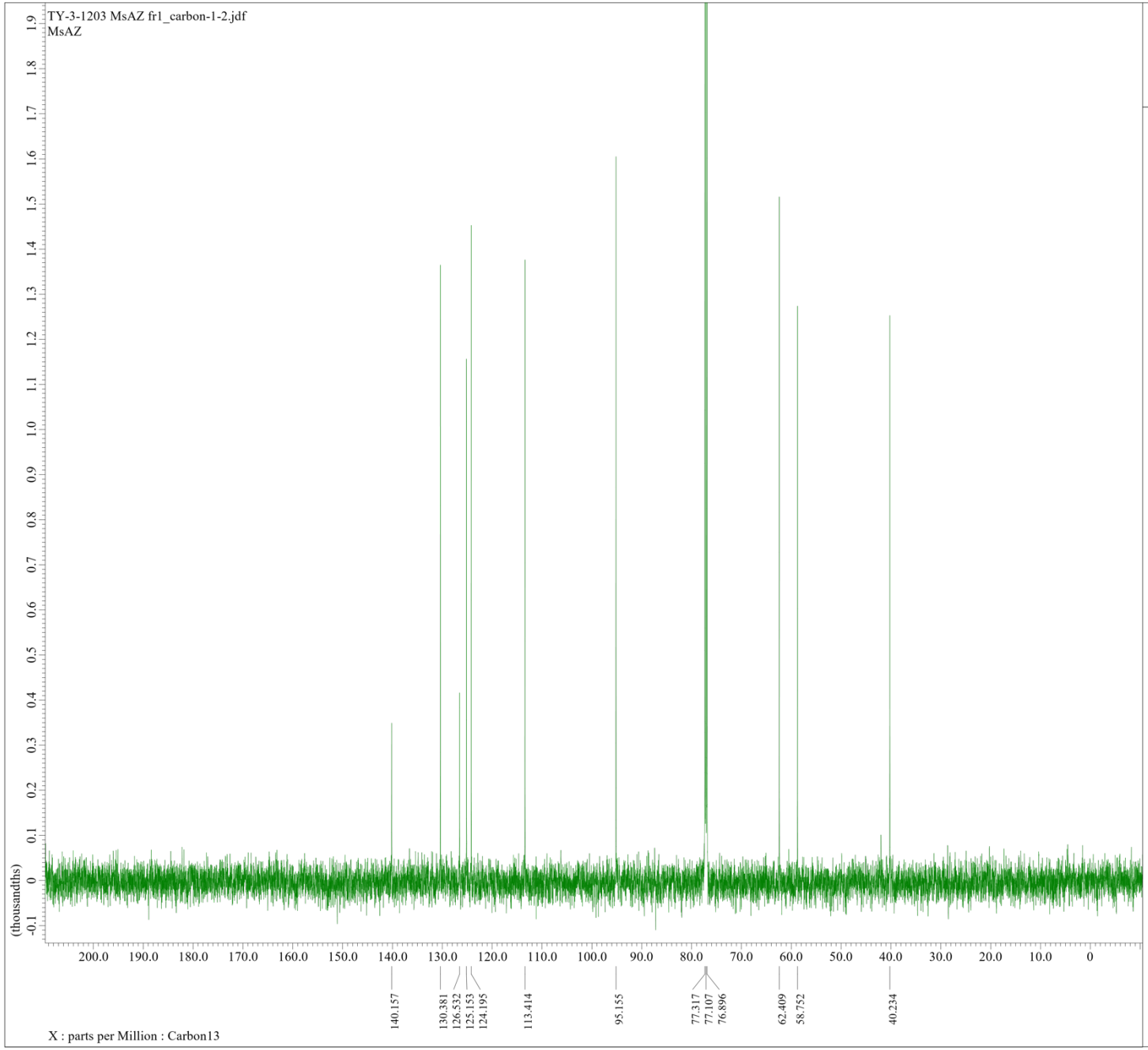
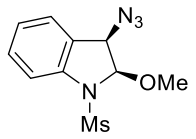


1d

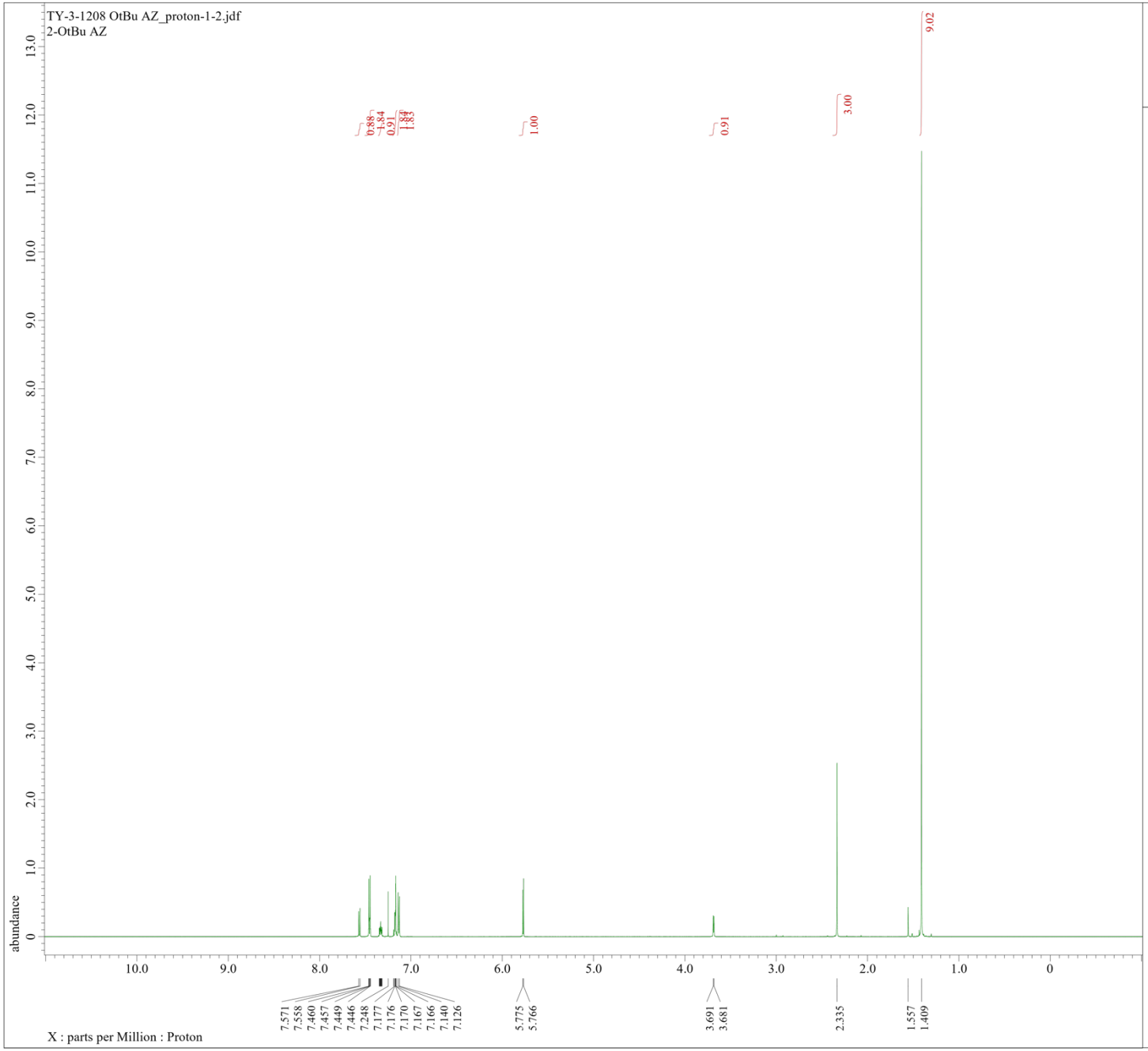


JEOL

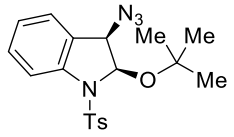
Filename	= TY-3-1203 MsAZ fr1
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1203 MsAZ fr1
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 3-DEC-2021 21:54:
Revision_Time	= 3-DEC-2021 22:08:
Comment	= MsAZ
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 257
Total_Scans	= 257
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 21.2[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]



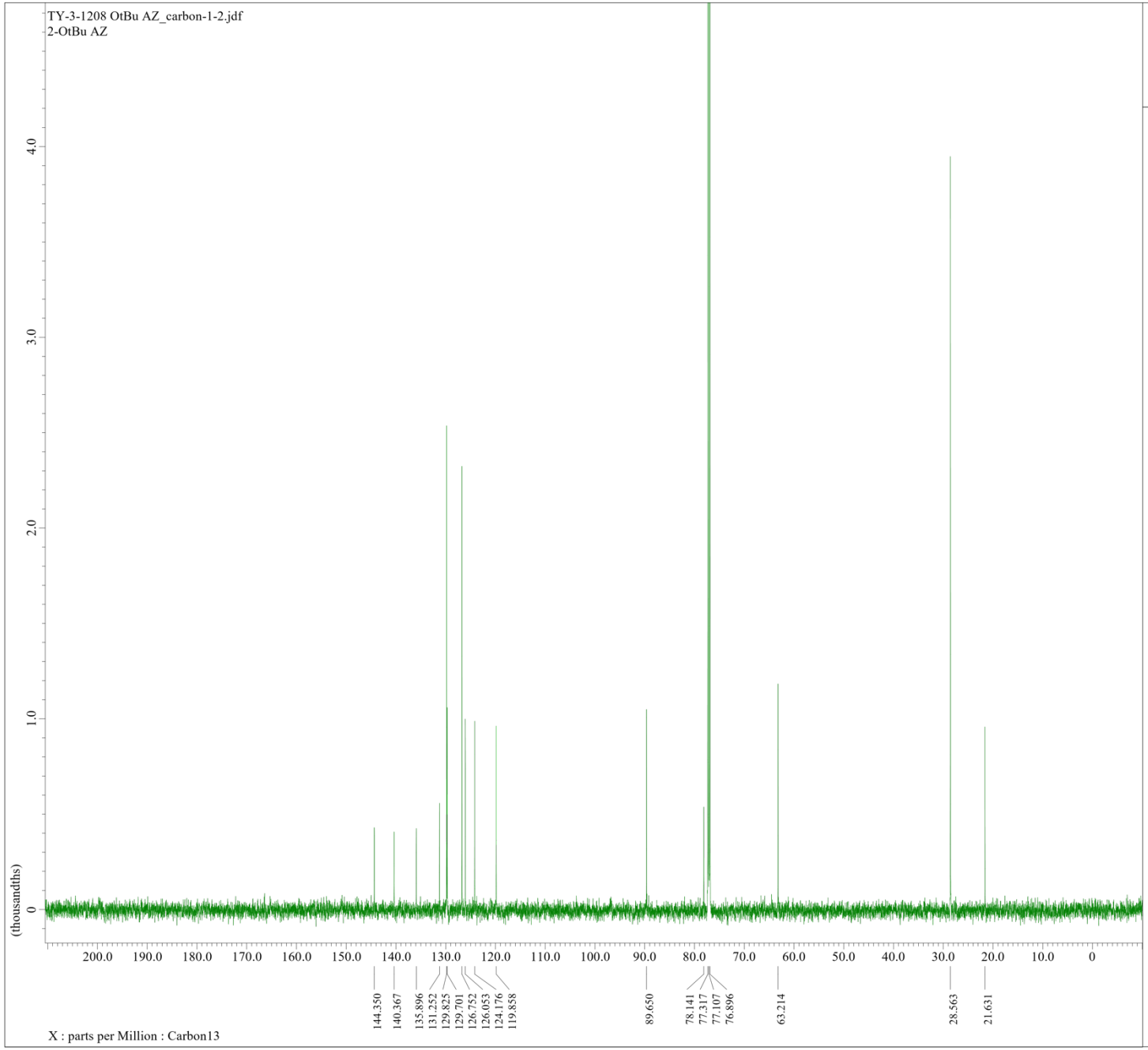
1d




```
Filename              = TY-3-1208 OtBu AZ_pro
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-3-1208 OtBu AZ
Solvent               = CHLOROFORM-D
Actual_Start_Time     =  8-DEC-2021 15:53:05
Revision_Time         =  8-DEC-2021 15:51:15

Comment               = 2-OtBu AZ
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8

Relaxation_Delay      = 1[s]
Recvr_Gain            = 46
Temp_Get              = 21[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```

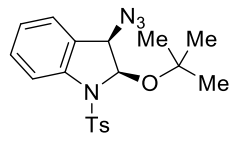


1l

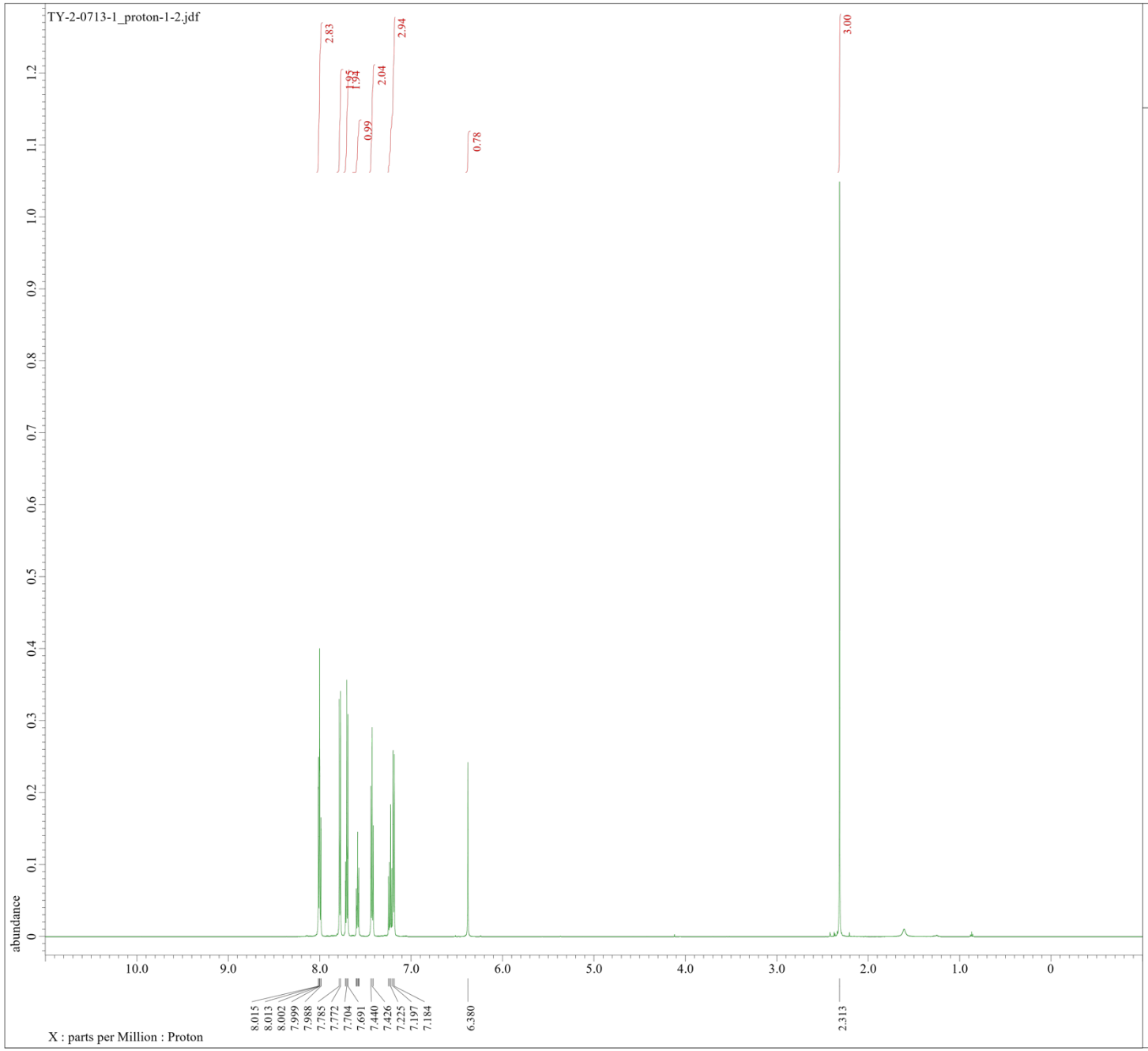


JEOL

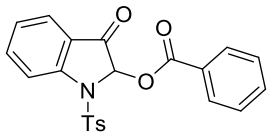
Filename	= TY-3-1208 OtBu AZ_
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1208 OtBu AZ
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 8-DEC-2021 15:55:
Revision_Time	= 8-DEC-2021 15:57:
Comment	= 2-OtBu AZ
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 256
Total_Scans	= 256
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 21[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]



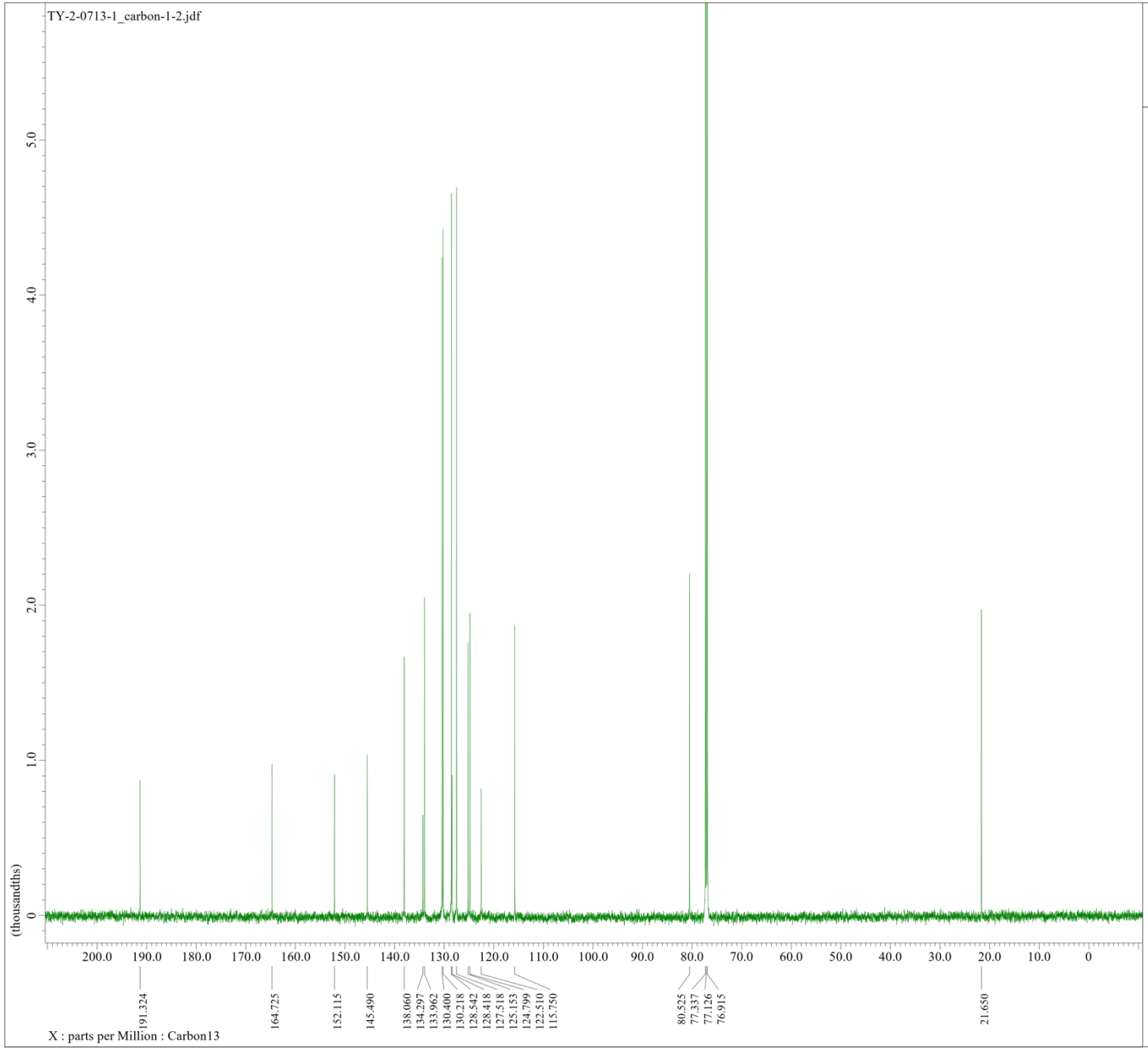
1l




```
Filename              = TY-2-0713-1_proton-1-
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-2-0713-1
Solvent               = CHLOROFORM-D
Actual_Start_Time     = 13-JUL-2021 20:13:06
Revision_Time         = 13-JUL-2021 20:37:23

Comment               = PhCOOH AZ
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 16
Total_Scans           = 16

Relaxation_Delay      = 1[s]
Recvr_Gain            = 36
Temp_Get              = 20.7[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```

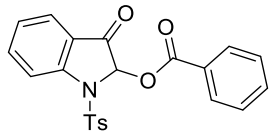


3aa

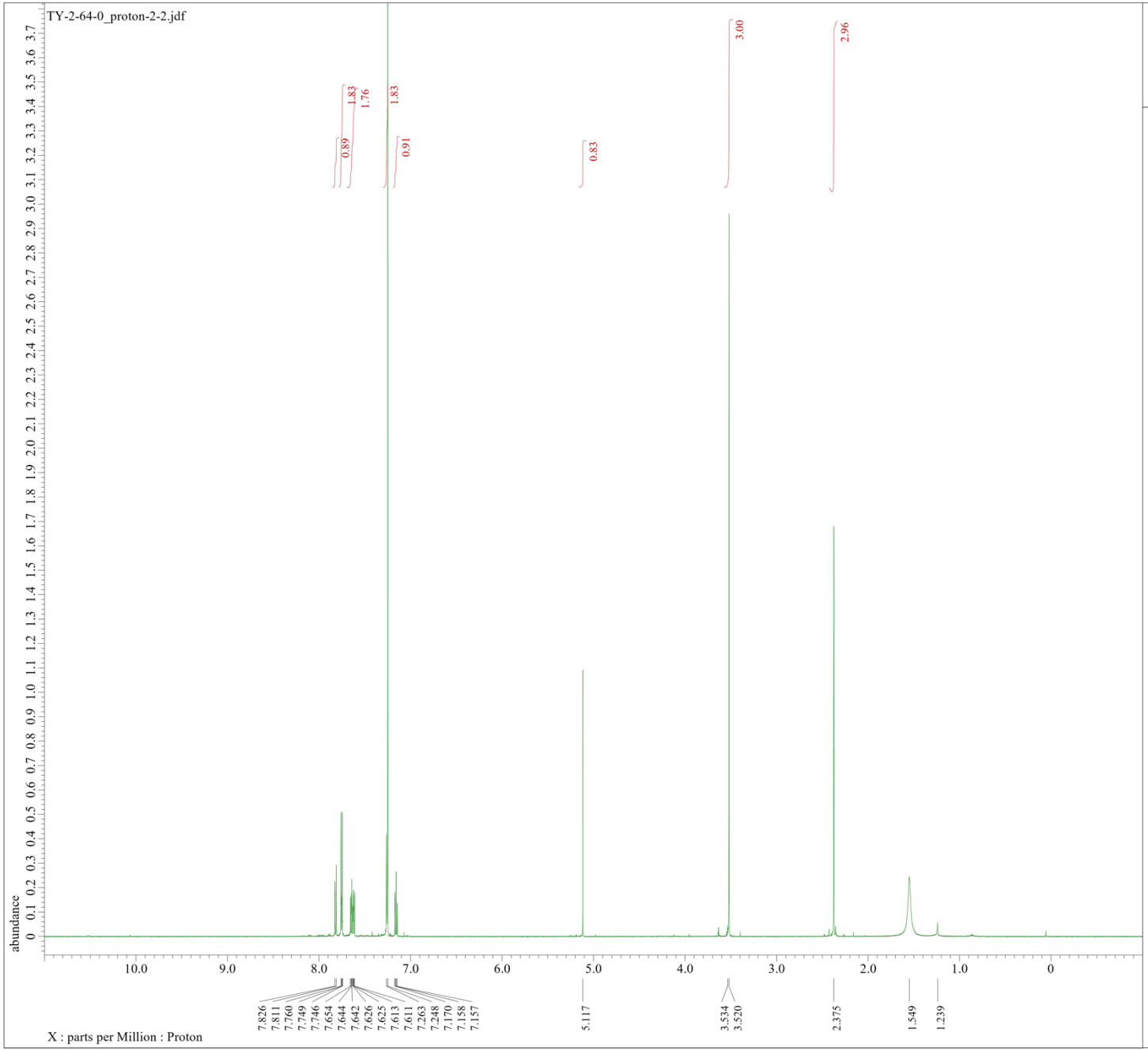


JEOL

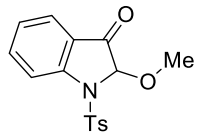
Filename	= TY-2-0713-1_carbon
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-2-0713-1
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 13-JUL-2021 20:15:
Revision_Time	= 13-JUL-2021 20:59:
Comment	= PhCOOH AZ
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= TRUE
Scans	= 512
Total_Scans	= 512
Relaxation_Delay	= 2[s]
Recvr_Gain	= 36
Temp_Get	= 21[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 2[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 2[s]
Repetition_Time	= 2.69206016[s]



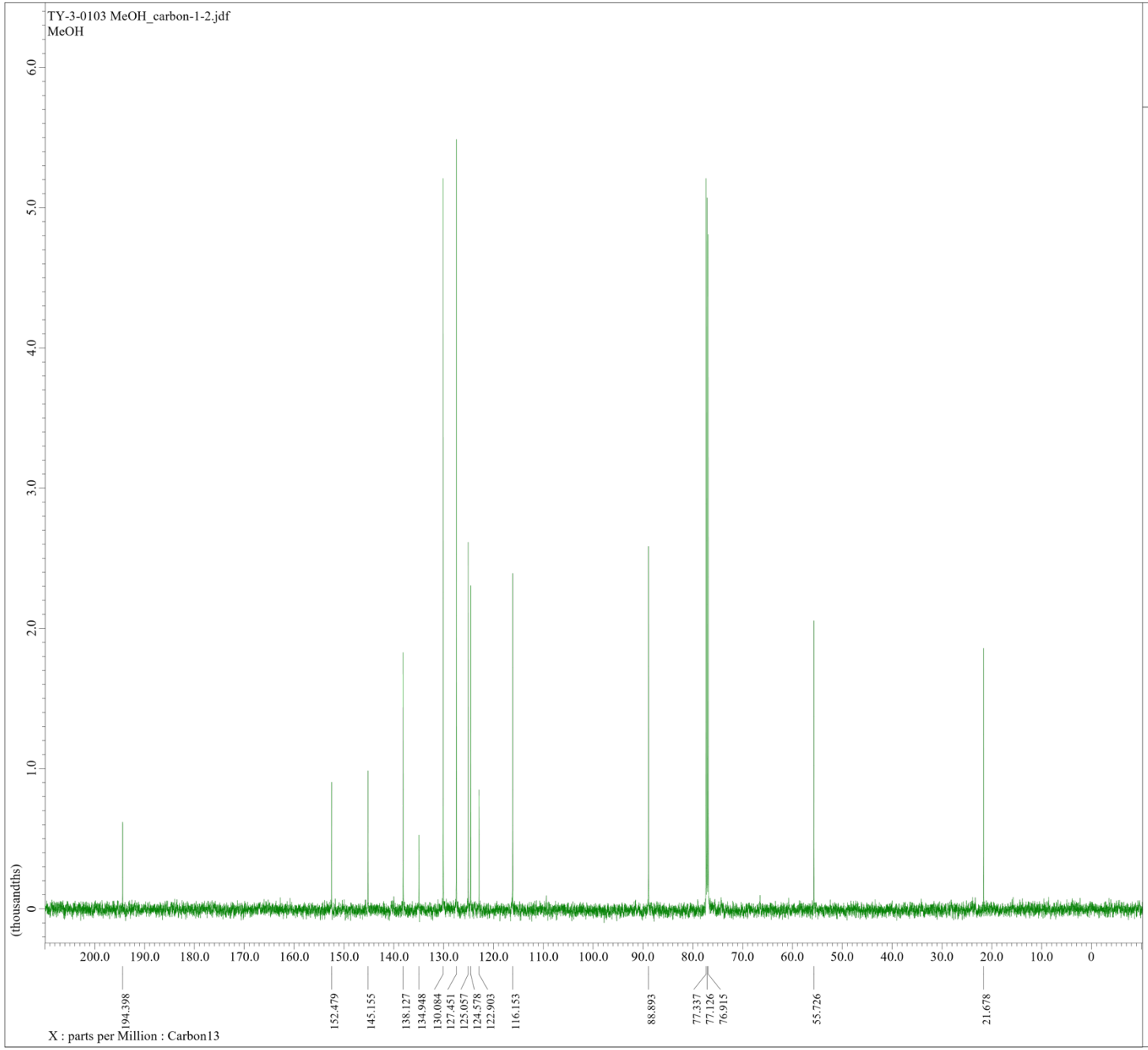
3aa




```
Filename              = TY-2-64-0_proton-2-2.
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-2-64-0
Solvent               = CHLOROFORM-D
Actual_Start_Time     = 12-JUL-2021 15:50:46
Revision_Time         = 12-JUL-2021 16:23:43

Comment               = 2-OMe
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8

Relaxation_Delay      = 1[s]
Recvr_Gain            = 66
Temp_Get              = 20.8[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```

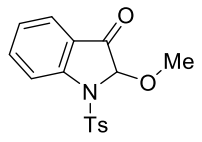


4a

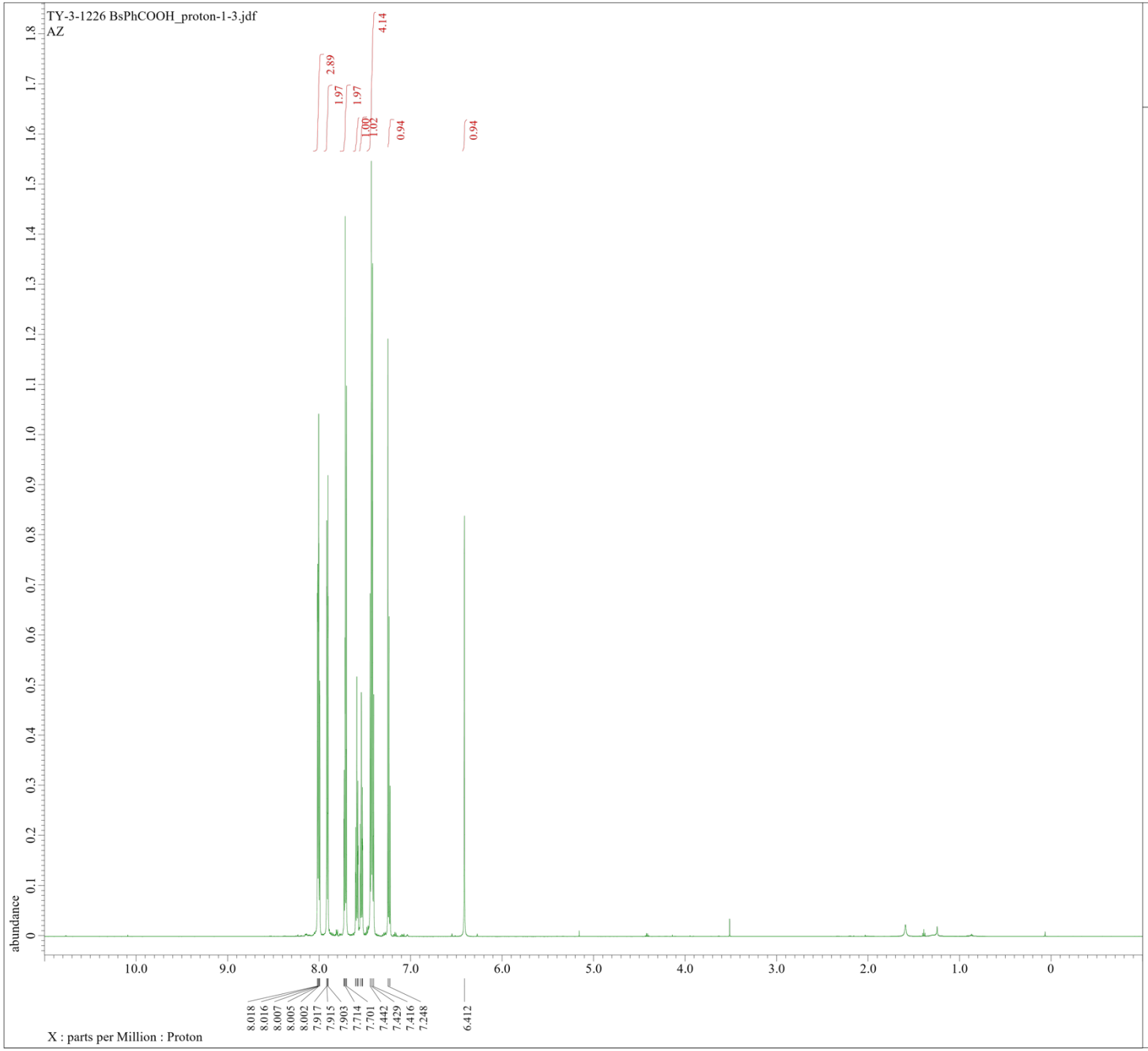


JEOL

Filename	= TY-3-0103 MeOH_car
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-0103 MeOH
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 3-JAN-2022 21:02:
Revision_Time	= 3-JAN-2022 21:15:
Comment	= MeOH
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= TRUE
Scans	= 227
Total_Scans	= 227
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 20.5[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]

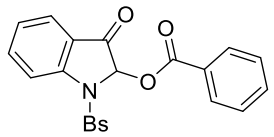


4a

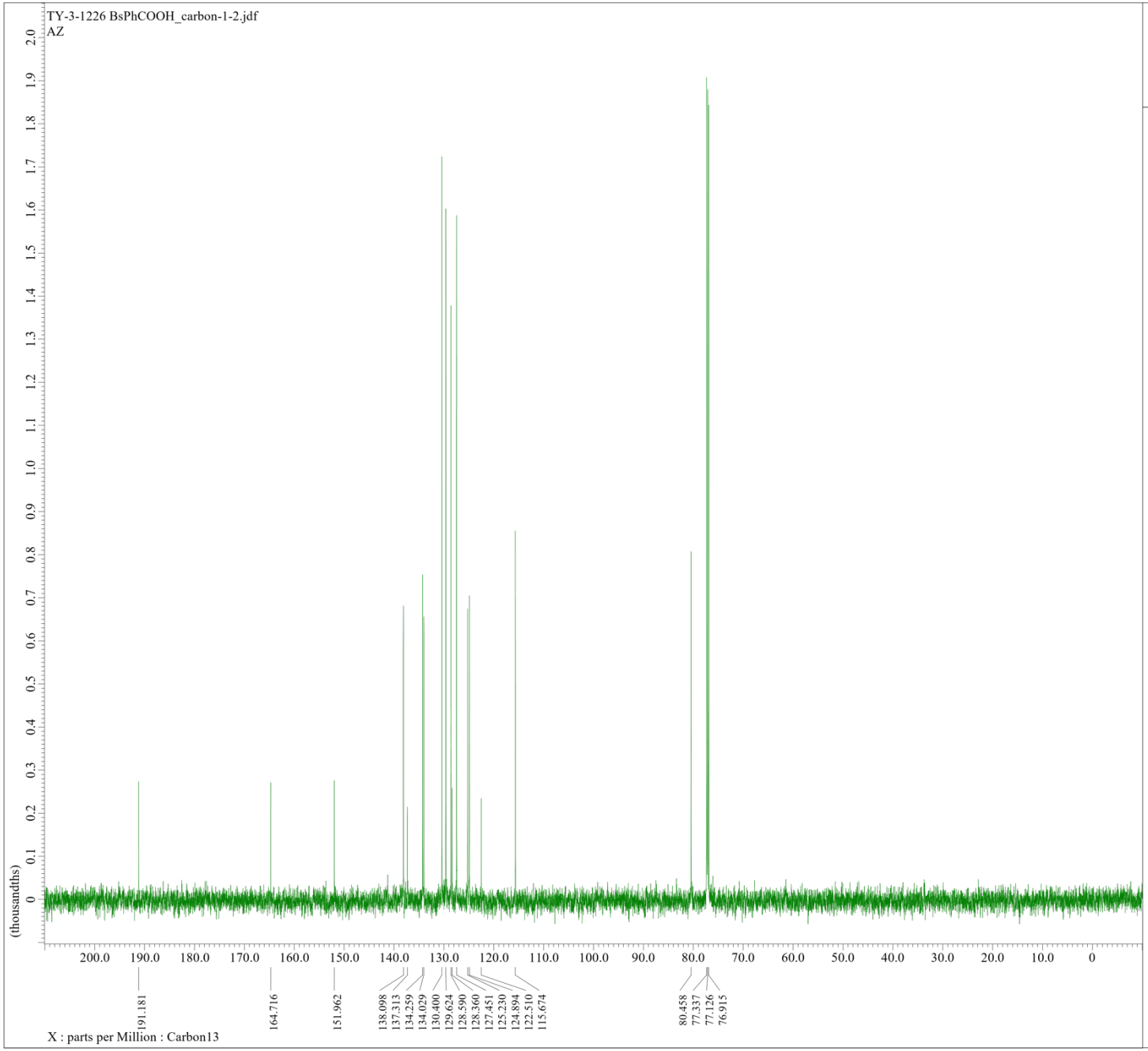


JEOL

Filename	= TY-3-1226 BsPhCOOH_pr
Author	= delta
Experiment	= proton.jxp
Sample_Id	= TY-3-1226 BsPhCOOH
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 26-DEC-2021 17:29:48
Revision_Time	= 26-DEC-2021 17:46:16
Comment	= AZ
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (600[M
X_Acq_Duration	= 2.18103808[s]
X_Domain	= Proton
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.45849727[Hz]
X_Sweep	= 15.02403846[kHz]
X_Sweep_Clipped	= 12.01923077[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 1[s]
Recvr_Gain	= 46
Temp_Get	= 21.1[dC]
X_90_Width	= 7.7[us]
X_Acq_Time	= 2.18103808[s]
X_Angle	= 45[deg]
X_Atn	= 8.1[dB]
X_Pulse	= 3.85[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 100
Dante_Presat	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\JEOL
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180
Presat_Time	= 1[s]
Presat_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 3.18103808[s]

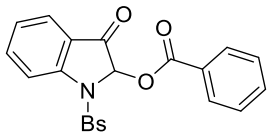


3ba

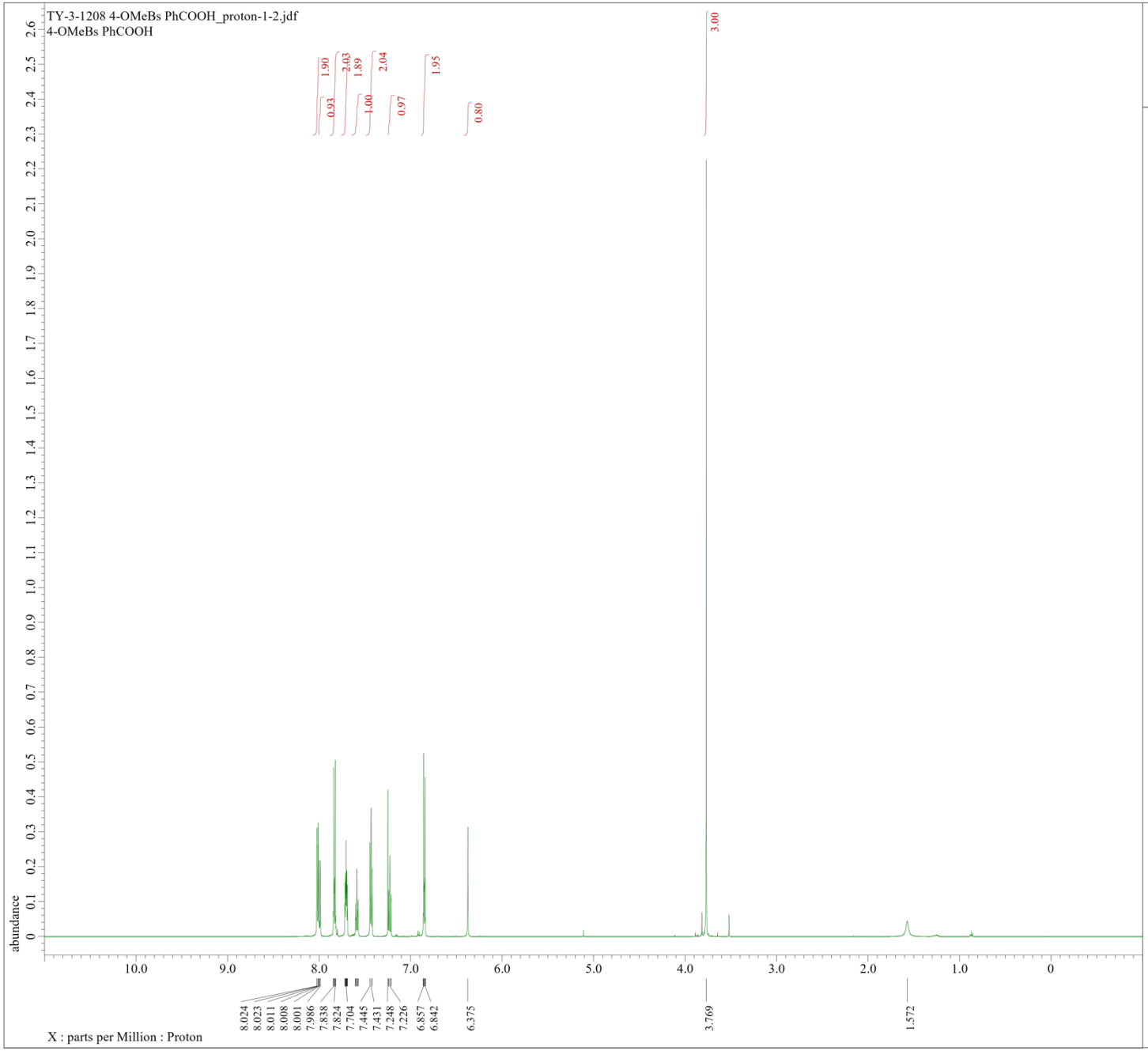


JEOL

Filename	= TY-3-1226 BsPhCOOH
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1226 BsPhCOOH
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 26-DEC-2021 17:31:
Revision_Time	= 26-DEC-2021 17:46:
Comment	= AZ
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 95
Total_Scans	= 95
Relaxation_Delay	= 1[s]
Recvr_Gain	= 26
Temp_Get	= 21.2[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]

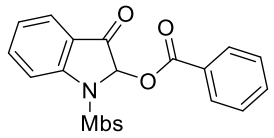


3ba

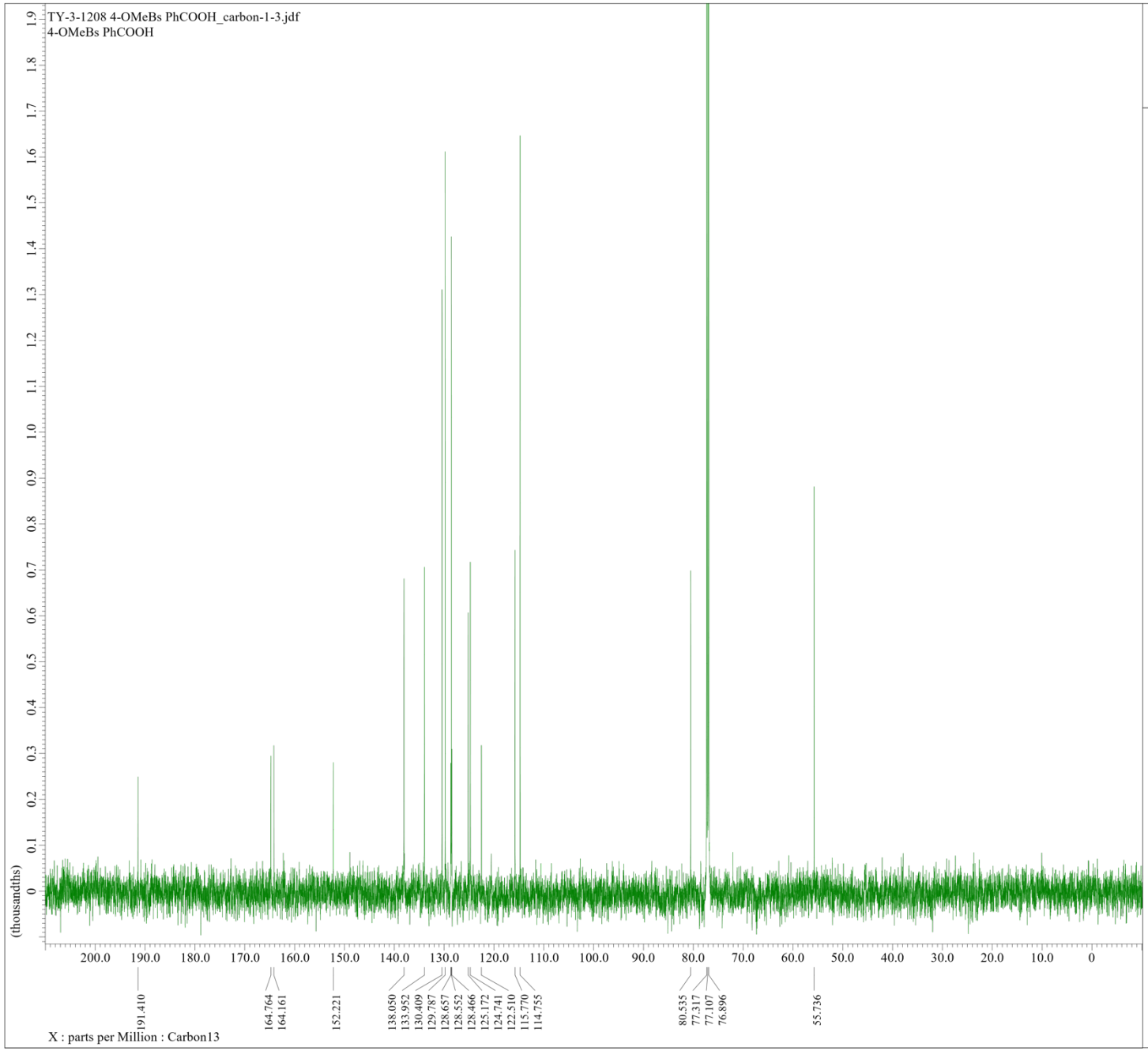


JEOL

Filename	= TY-3-1208 4-OMeBs PhC
Author	= delta
Experiment	= proton.jxp
Sample_Id	= TY-3-1208 4-OMeBs PhC
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 8-DEC-2021 15:38:44
Revision_Time	= 8-DEC-2021 15:46:17
Comment	= 4-OMeBs PhCOOH
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (600[M
X_Acq_Duration	= 2.18103808[s]
X_Domain	= Proton
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.45849727[Hz]
X_Sweep	= 15.02403846[kHz]
X_Sweep_Clipped	= 12.01923077[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 1[s]
Recvr_Gain	= 46
Temp_Get	= 21.2[dC]
X_90_Width	= 7.7[us]
X_Acq_Time	= 2.18103808[s]
X_Angle	= 45[deg]
X_Atn	= 8.1[dB]
X_Pulse	= 3.85[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 100
Dante_Presat	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\JEOL
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180
Presat_Time	= 1[s]
Presat_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 3.18103808[s]

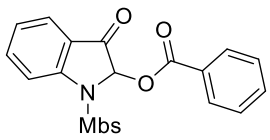


3ca

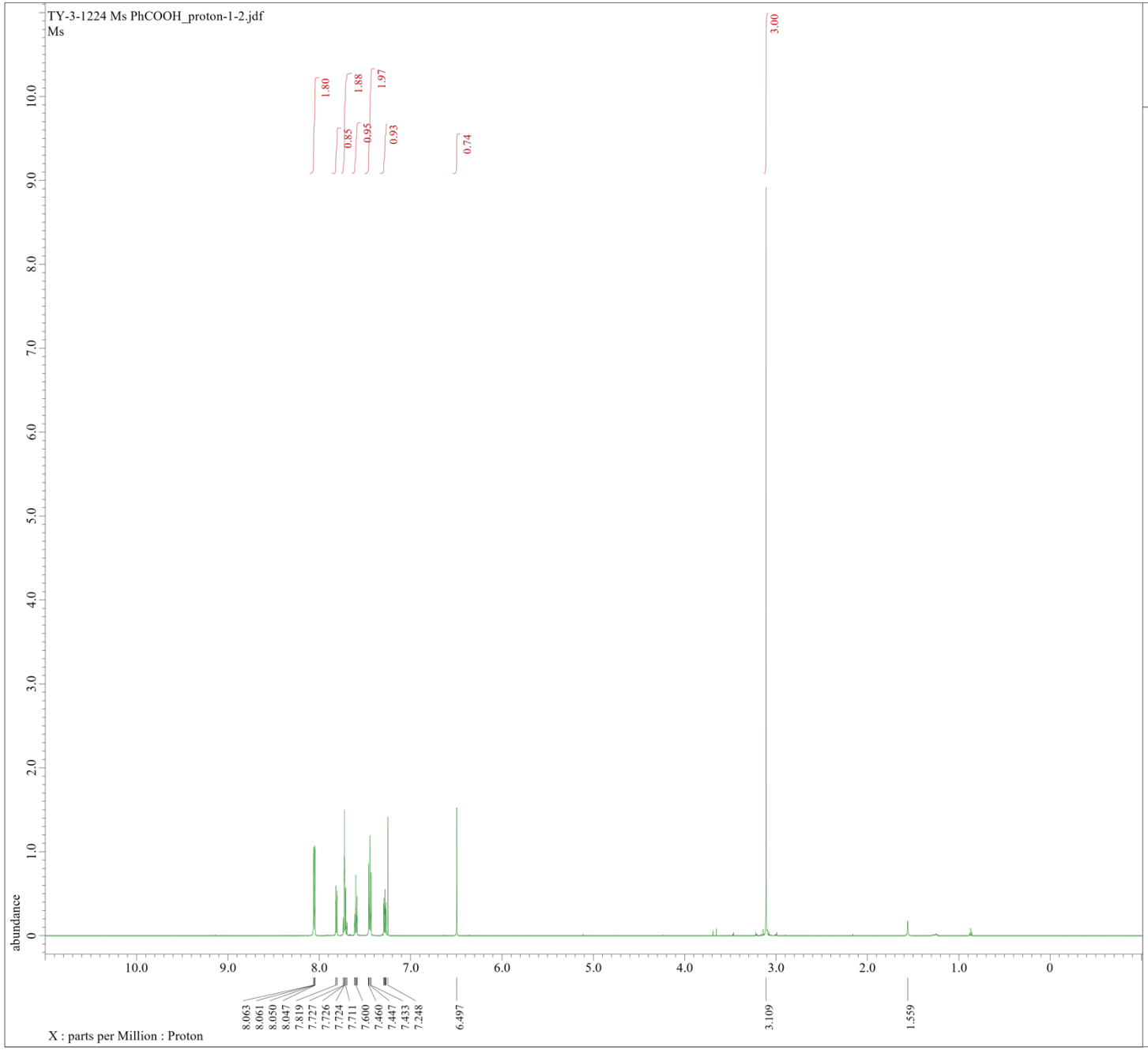


JEOL

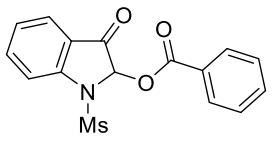
Filename	= TY-3-1208 4-OMeBs
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1208 4-OMeBs
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 8-DEC-2021 15:40:
Revision_Time	= 8-DEC-2021 16:07:
Comment	= 4-OMeBs PhCOOH
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 256
Total_Scans	= 256
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 21.1[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]



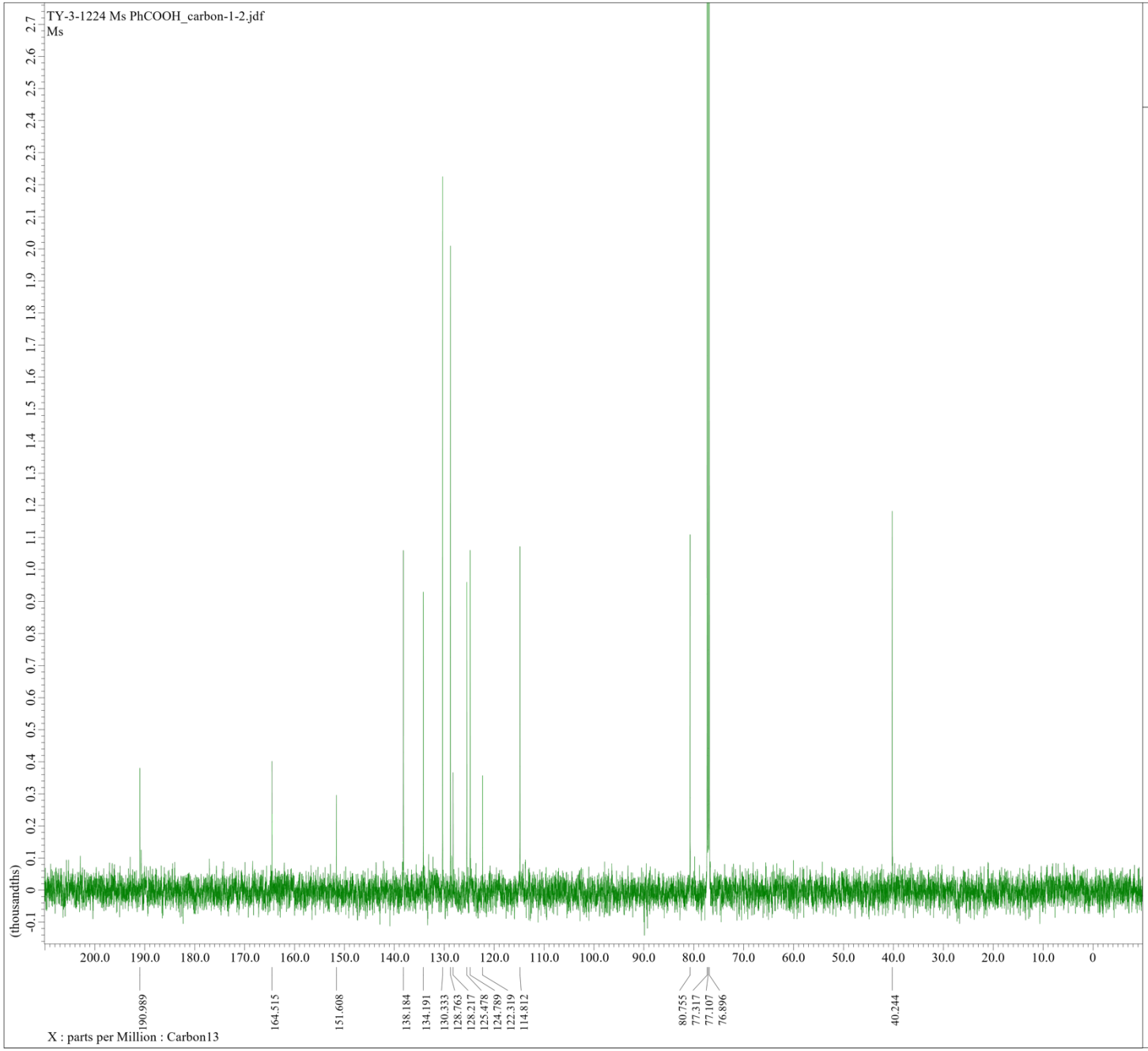
3ca



```
Filename              = TY-3-1224 Ms PhCOOH_p
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-3-1224 Ms PhCOOH
Solvent               = CHLOROFORM-D
Actual_Start_Time     = 24-DEC-2021 19:40:43
Revision_Time         = 24-DEC-2021 19:45:57

Comment               = Ms
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8

Relaxation_Delay      = 1[s]
Recvr_Gain            = 56
Temp_Get              = 21.1[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```

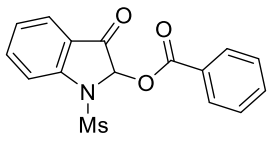


3da

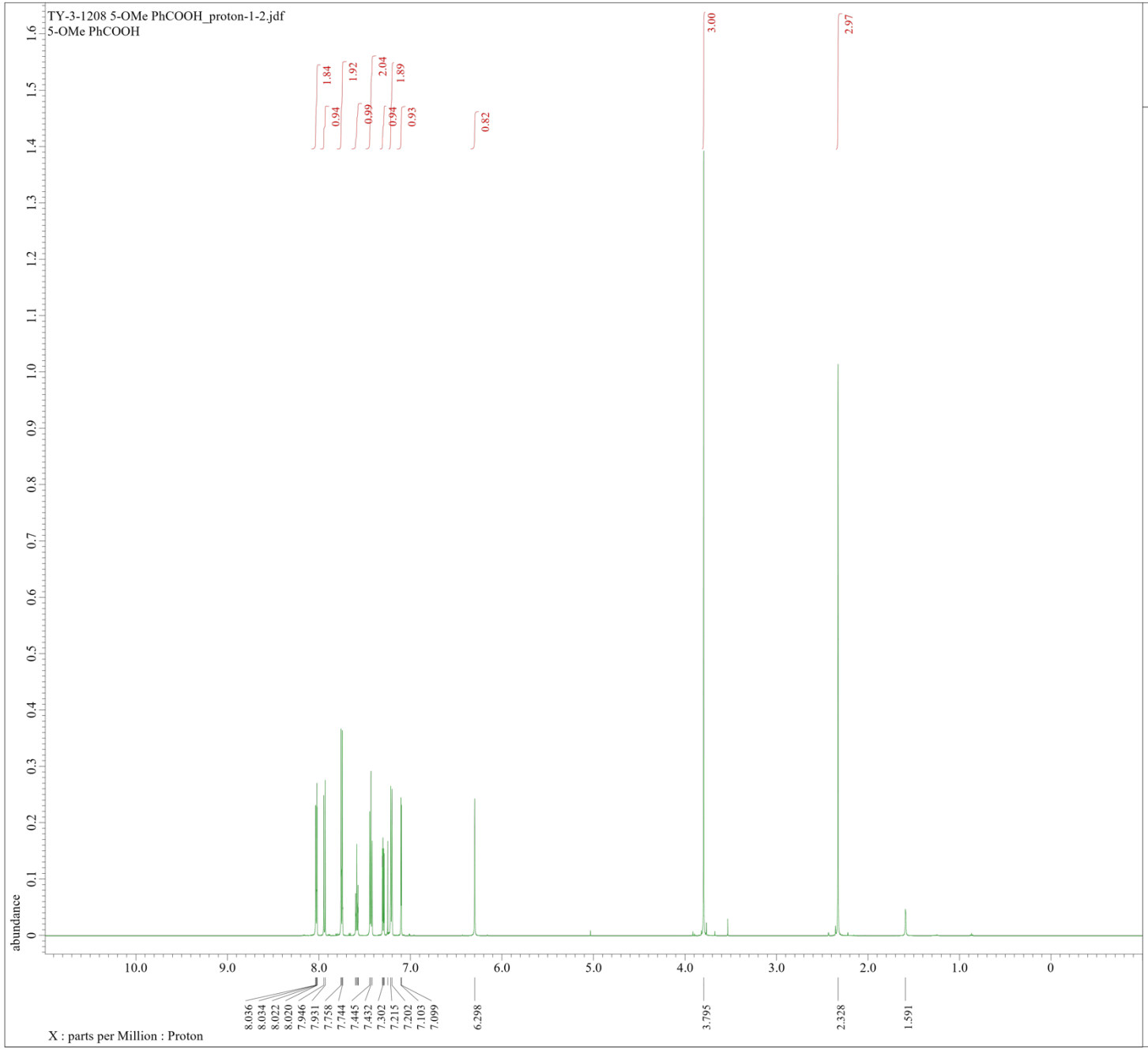


JEOL

Filename	= TY-3-1224 Ms PhCOO
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1224 Ms PhCOO
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 24-DEC-2021 19:42:
Revision_Time	= 24-DEC-2021 19:48:
Comment	= Ms
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 178
Total_Scans	= 178
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 21.2[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]

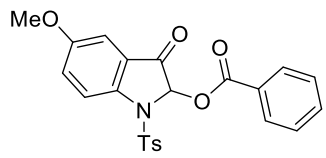


3da

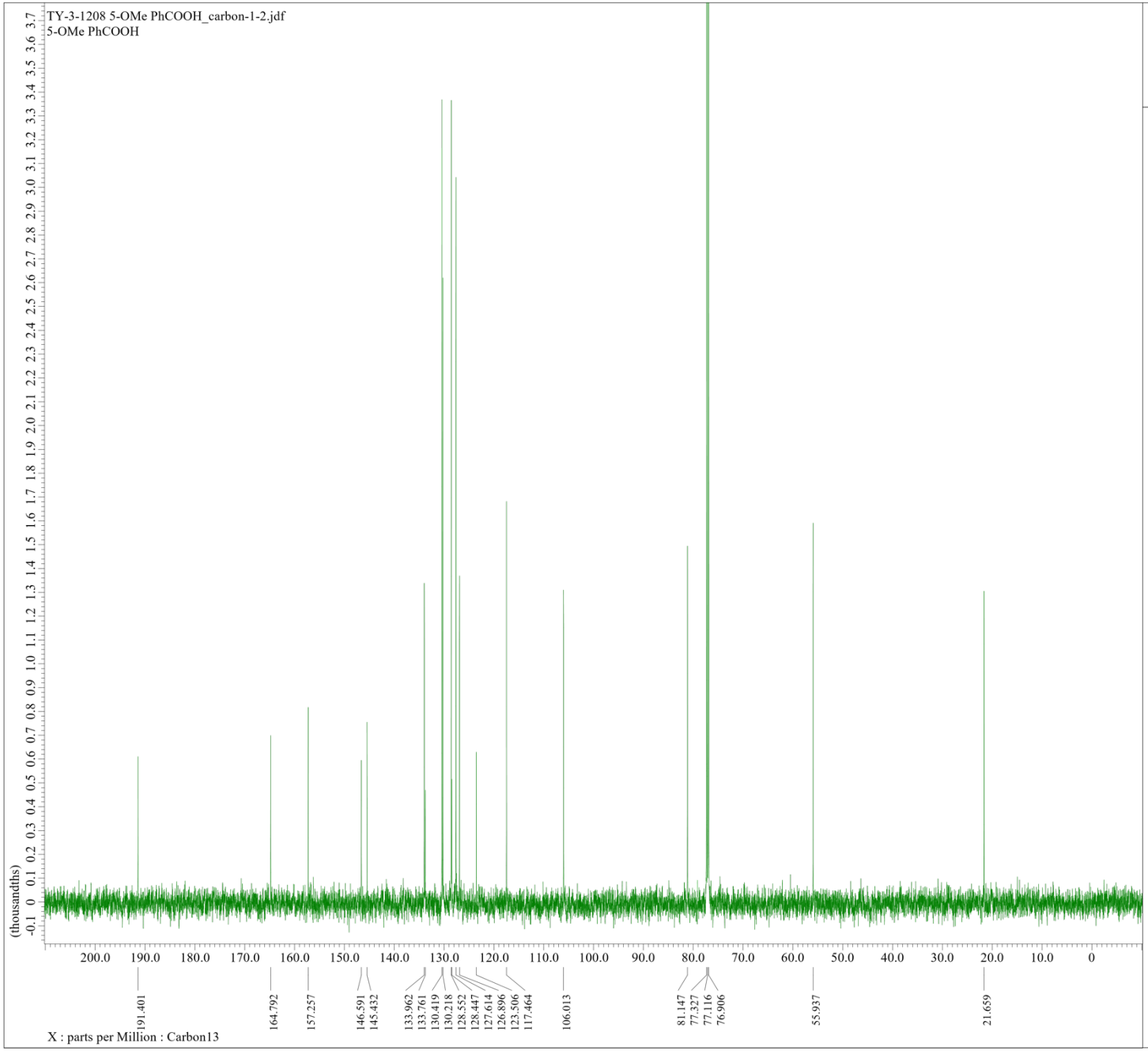


JEOL

Filename	= TY-3-1208 5-OMe PhCOO
Author	= delta
Experiment	= proton.jxp
Sample_Id	= TY-3-1208 5-OMe PhCOO
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 8-DEC-2021 15:27:20
Revision_Time	= 8-DEC-2021 15:42:36
Comment	= 5-OMe PhCOOH
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (600[M
X_Acq_Duration	= 2.18103808[s]
X_Domain	= Proton
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.45849727[Hz]
X_Sweep	= 15.02403846[kHz]
X_Sweep_Clipped	= 12.01923077[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 21.1[dC]
X_90_Width	= 7.7[us]
X_Acq_Time	= 2.18103808[s]
X_Angle	= 45[deg]
X_Atn	= 8.1[dB]
X_Pulse	= 3.85[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 100
Dante_Presat	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\JEOL
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180
Presat_Time	= 1[s]
Presat_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 3.18103808[s]

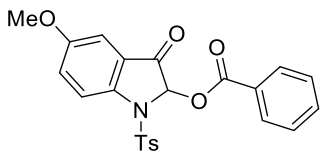


3ea

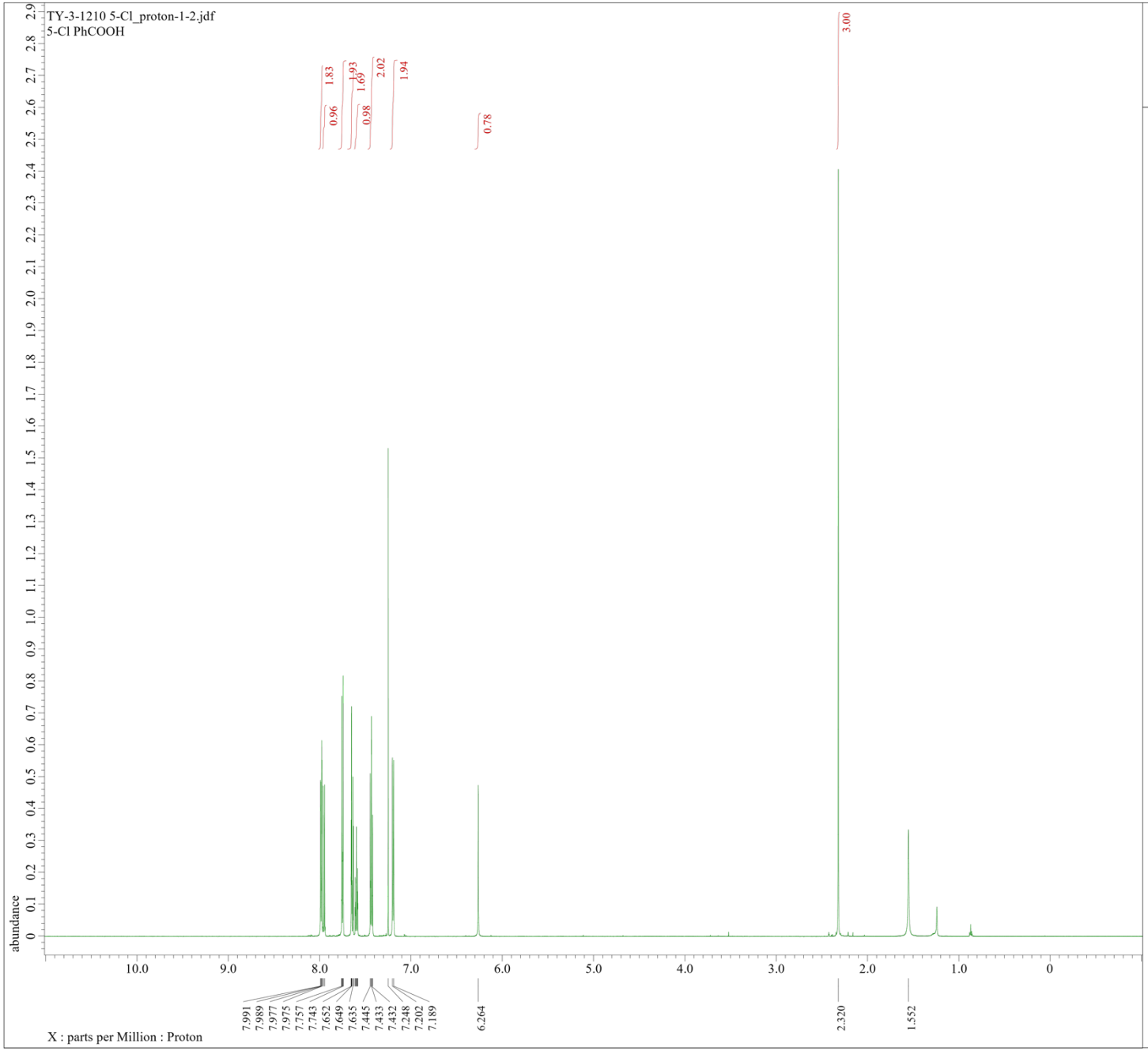


JEOL

Filename	= TY-3-1208 5-OMe Ph
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1208 5-OMe Ph
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 8-DEC-2021 15:29:
Revision_Time	= 8-DEC-2021 15:43:
Comment	= 5-OMe PhCOOH
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 152
Total_Scans	= 152
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 21.3[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]

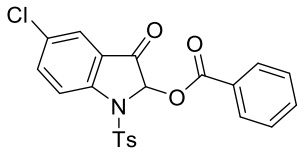


3ea



JEOL

Filename	= TY-3-1210 5-Cl_proton
Author	= delta
Experiment	= proton.jxp
Sample_Id	= TY-3-1210 5-Cl
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 10-DEC-2021 12:22:46
Revision_Time	= 10-DEC-2021 12:26:08
Comment	= 5-Cl PhCOOH
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (600[M
X_Acq_Duration	= 2.18103808[s]
X_Domain	= Proton
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.45849727[Hz]
X_Sweep	= 15.02403846[kHz]
X_Sweep_Clipped	= 12.01923077[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 1[s]
Recvr_Gain	= 56
Temp_Get	= 21.3[dC]
X_90_Width	= 7.7[us]
X_Acq_Time	= 2.18103808[s]
X_Angle	= 45[deg]
X_Atn	= 8.1[dB]
X_Pulse	= 3.85[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 100
Dante_Presat	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\JEOL
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180
Presat_Time	= 1[s]
Presat_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 3.18103808[s]



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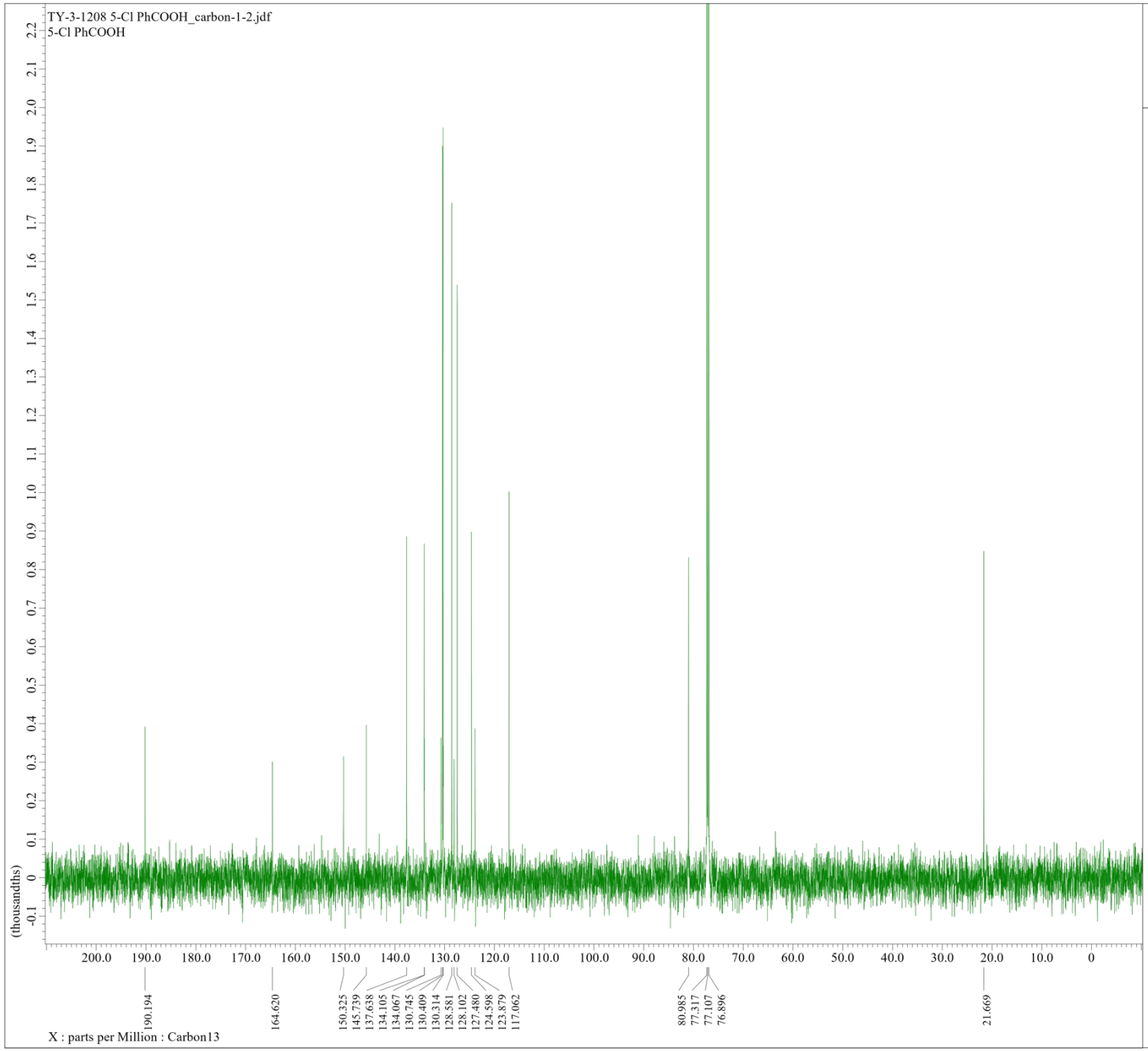
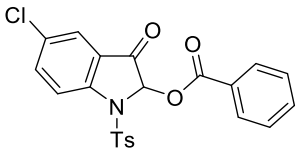


JEOL

Filename	= TY-3-1208 5-Cl PhC
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1208 5-Cl PhC
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 8-DEC-2021 22:19:
Revision_Time	= 9-DEC-2021 22:13:
Comment	= 5-Cl PhCOOH
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= TRUE
Scans	= 144
Total_Scans	= 144
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 21.3[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]



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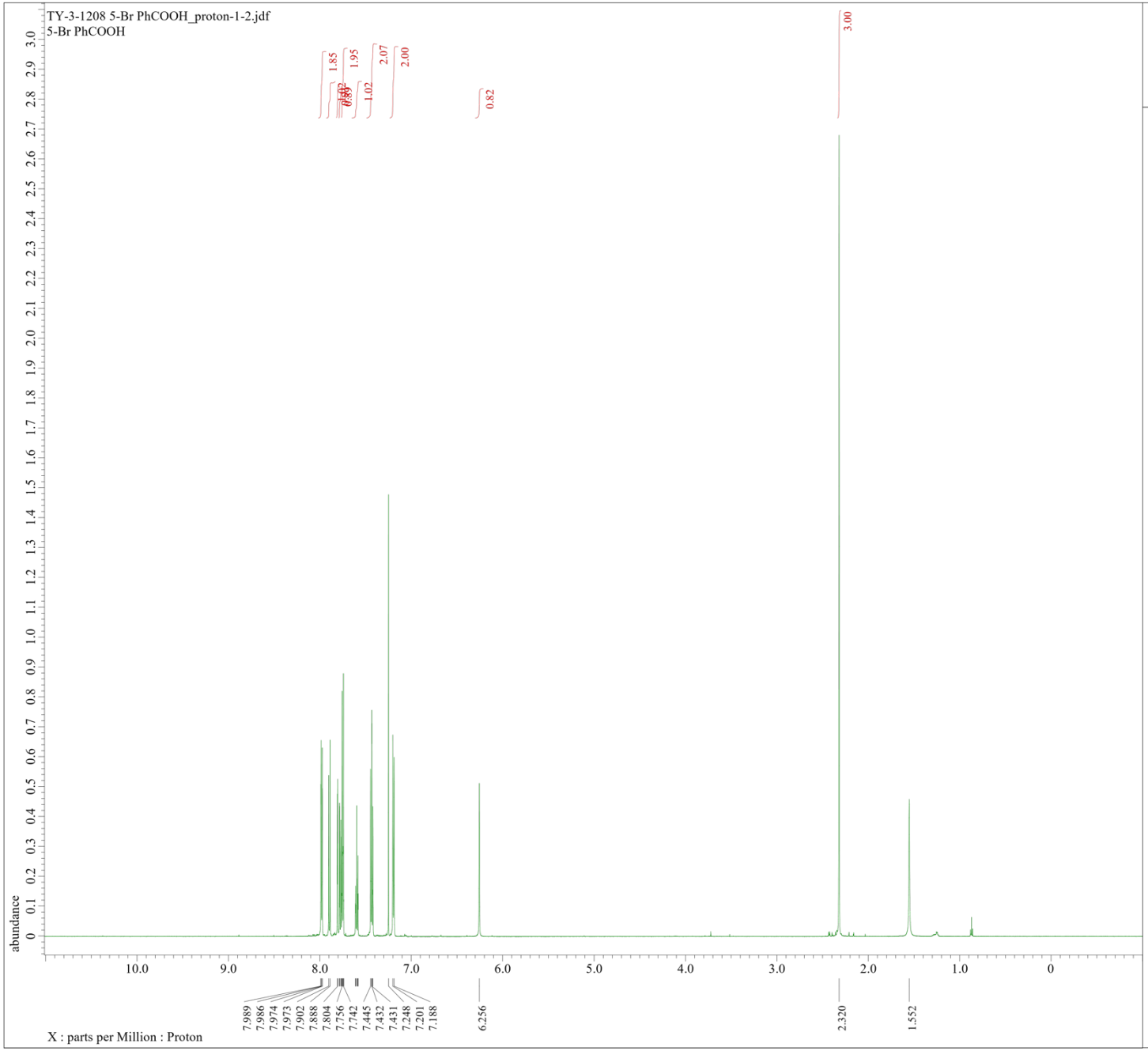
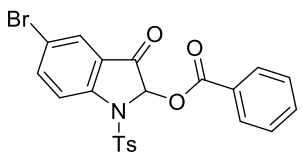


JEOL

Filename	= TY-3-1208 5-Br PhCOOH
Author	= delta
Experiment	= proton.jxp
Sample_Id	= TY-3-1208 5-Br PhCOOH
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 8-DEC-2021 22:28:22
Revision_Time	= 8-DEC-2021 22:24:34
Comment	= 5-Br PhCOOH
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (600[M
X_Acq_Duration	= 2.18103808[s]
X_Domain	= Proton
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.45849727[Hz]
X_Sweep	= 15.02403846[kHz]
X_Sweep_Clipped	= 12.01923077[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 1[s]
Recvr_Gain	= 56
Temp_Get	= 21.2[dC]
X_90_Width	= 7.7[us]
X_Acq_Time	= 2.18103808[s]
X_Angle	= 45[deg]
X_Atn	= 8.1[dB]
X_Pulse	= 3.85[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 100
Dante_Presat	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\JEOL
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180
Presat_Time	= 1[s]
Presat_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 3.18103808[s]



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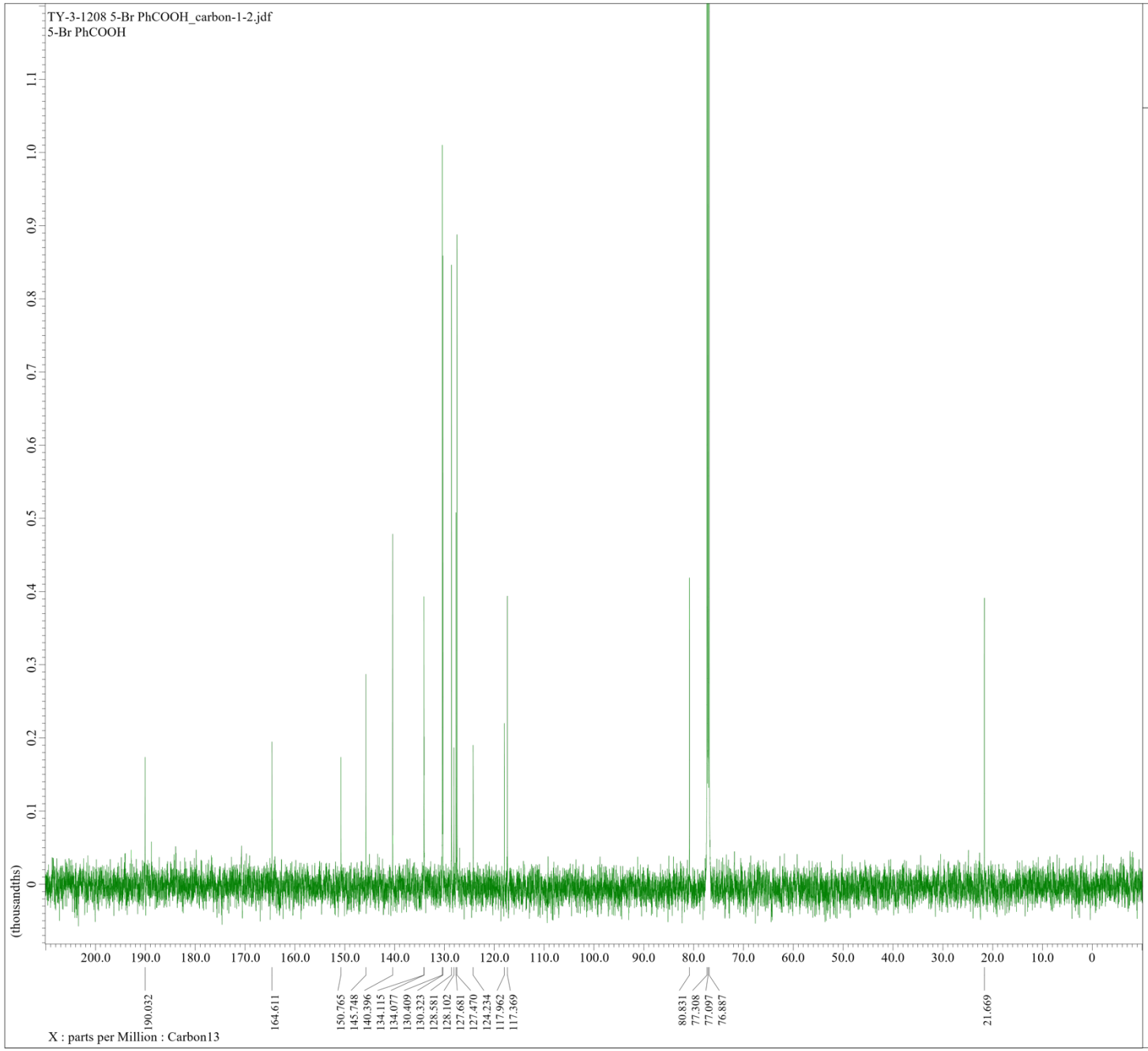
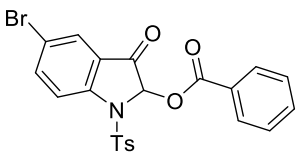
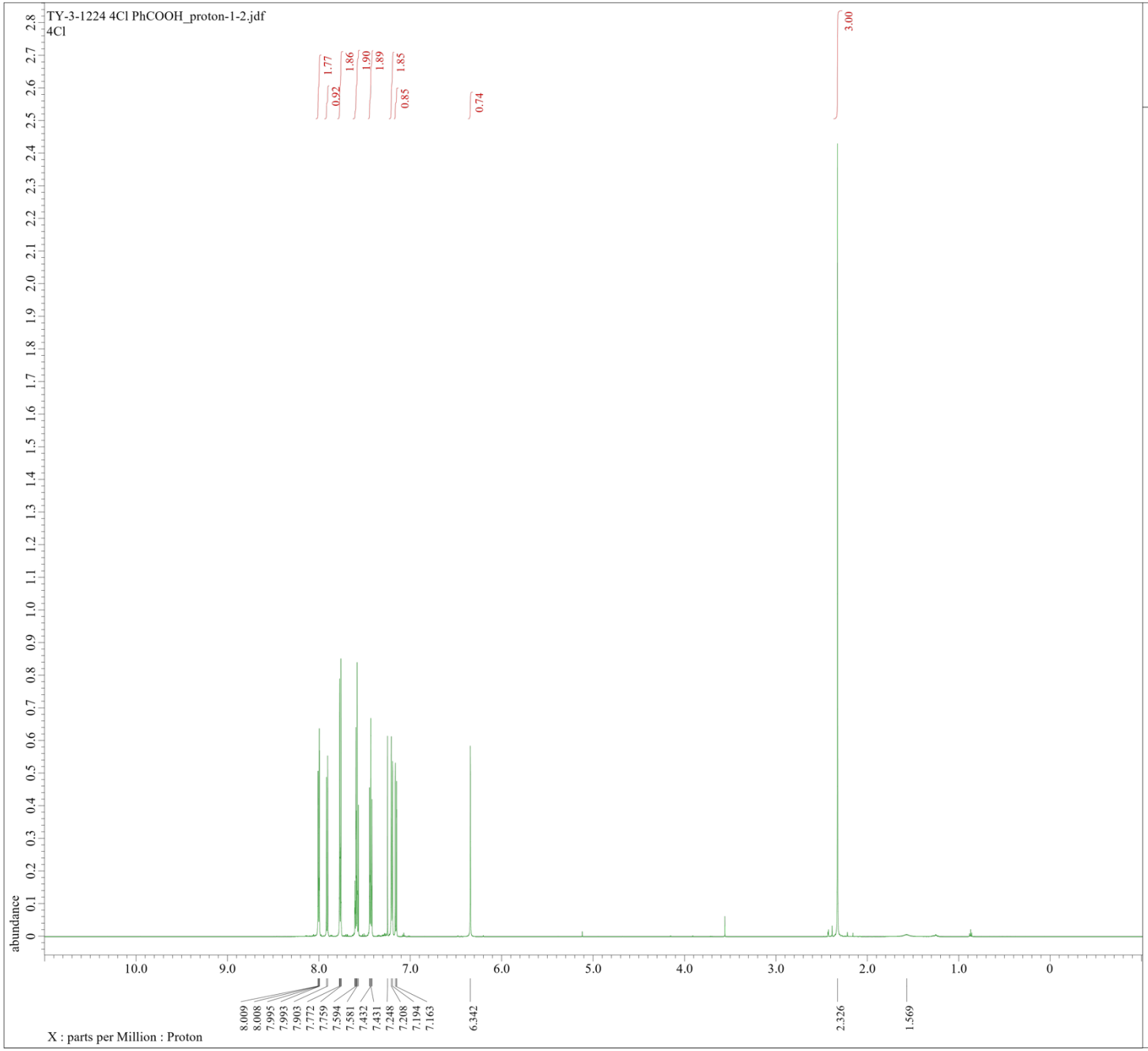


JEOL

Filename	= TY-3-1208 5-Br PhC
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1208 5-Br PhC
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 8-DEC-2021 22:30:
Revision_Time	= 9-DEC-2021 22:10:
Comment	= 5-Br PhCOOH
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 668
Total_Scans	= 668
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 21.3[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]

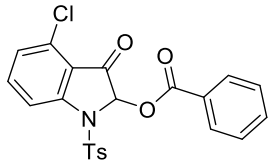


3ga

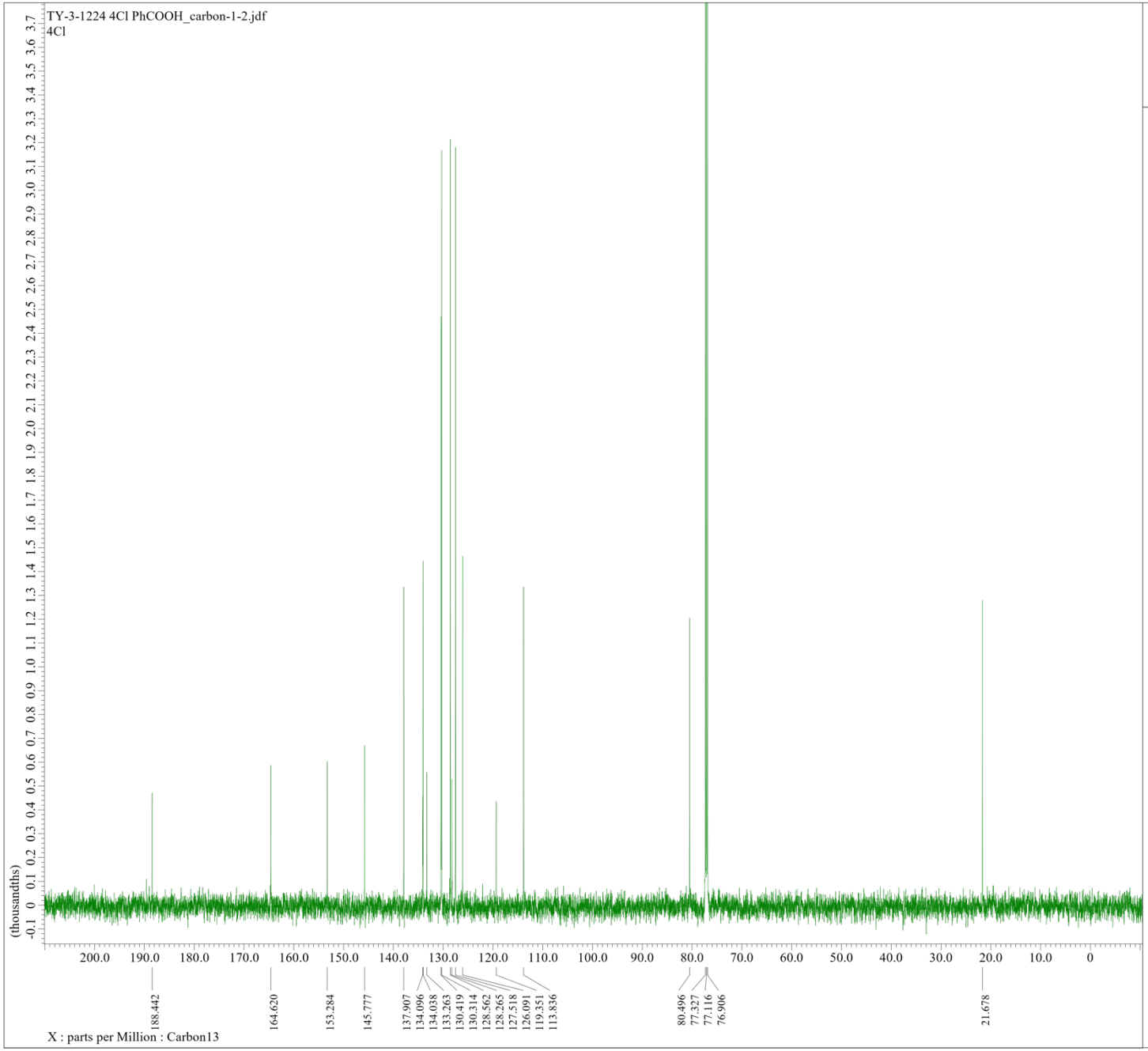


JEOL

```
Filename              = TY-3-1224 4Cl PhCOOH_
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-3-1224 4Cl PhCOOH
Solvent               = CHLOROFORM-D
Actual_Start_Time     = 24-DEC-2021 18:51:32
Revision_Time         = 24-DEC-2021 18:55:31

Comment               = 4Cl
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8

Relaxation_Delay      = 1[s]
Recvr_Gain            = 46
Temp_Get              = 20.9[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```

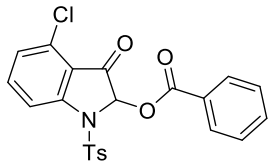


3ha



JEOL

Filename	= TY-3-1224 4Cl PhCO
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1224 4Cl PhCO
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 24-DEC-2021 18:53:
Revision_Time	= 24-DEC-2021 19:09:
Comment	= 4Cl
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 221
Total_Scans	= 221
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 21.1[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]



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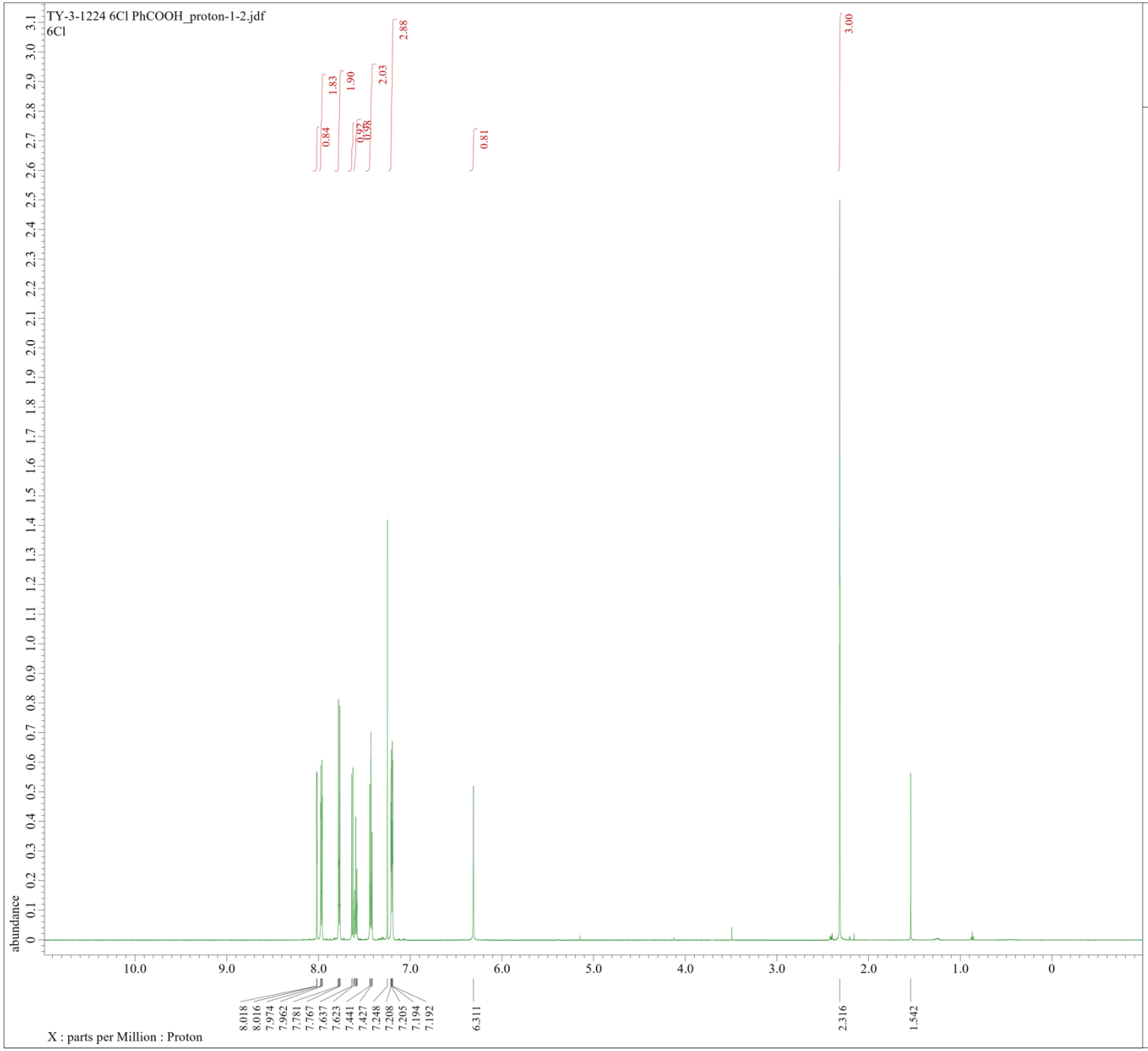
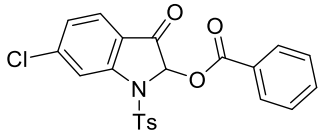
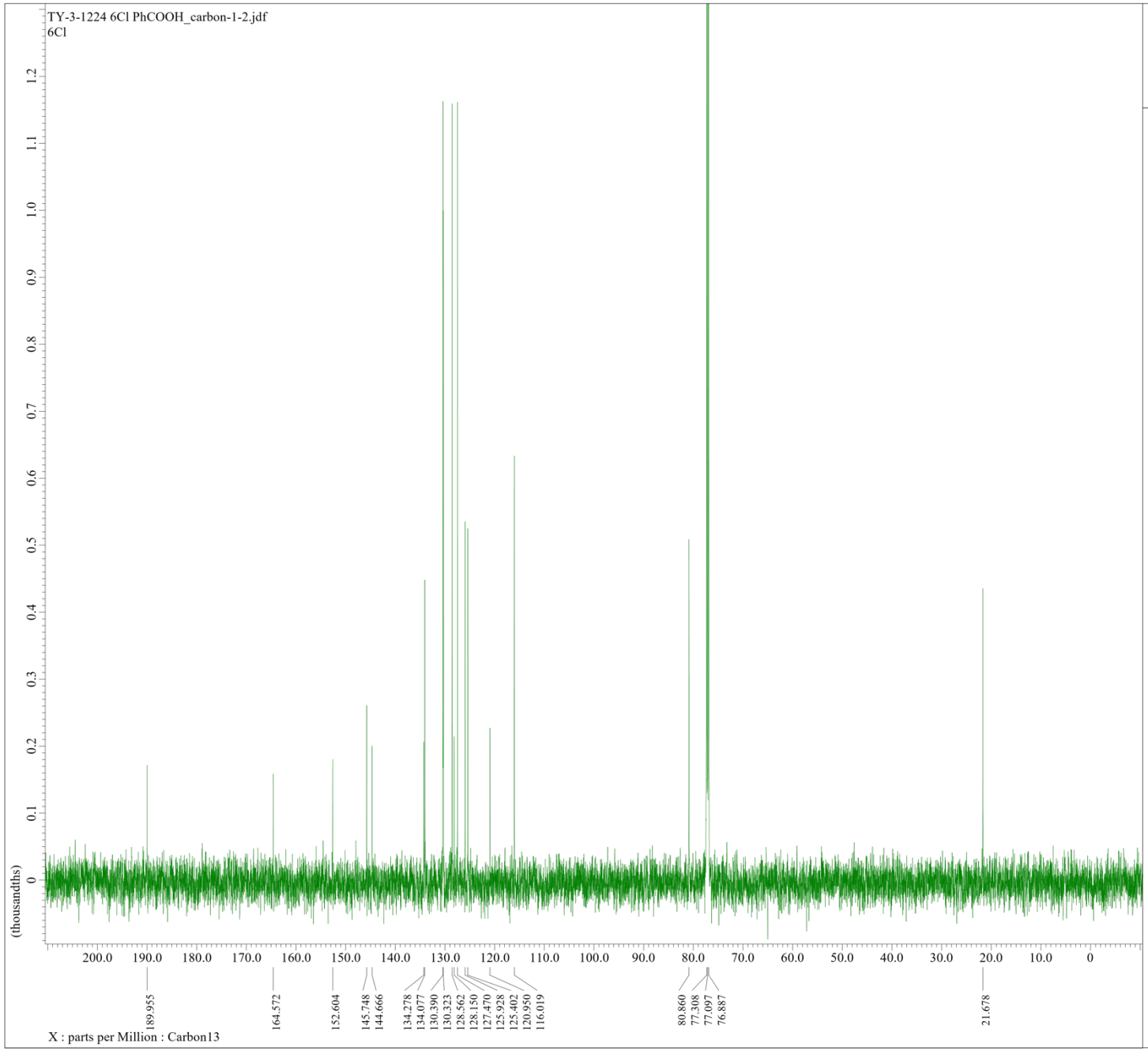


JEOL

```
Filename              = TY-3-1224 6Cl PhCOOH_
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-3-1224 6Cl PhCOOH
Solvent               = CHLOROFORM-D
Actual_Start_Time     = 24-DEC-2021 19:04:36
Revision_Time         = 24-DEC-2021 19:06:07

Comment               = 6Cl
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8

Relaxation_Delay      = 1[s]
Recvr_Gain            = 56
Temp_Get              = 21[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```

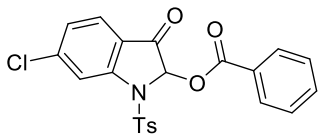


3ia

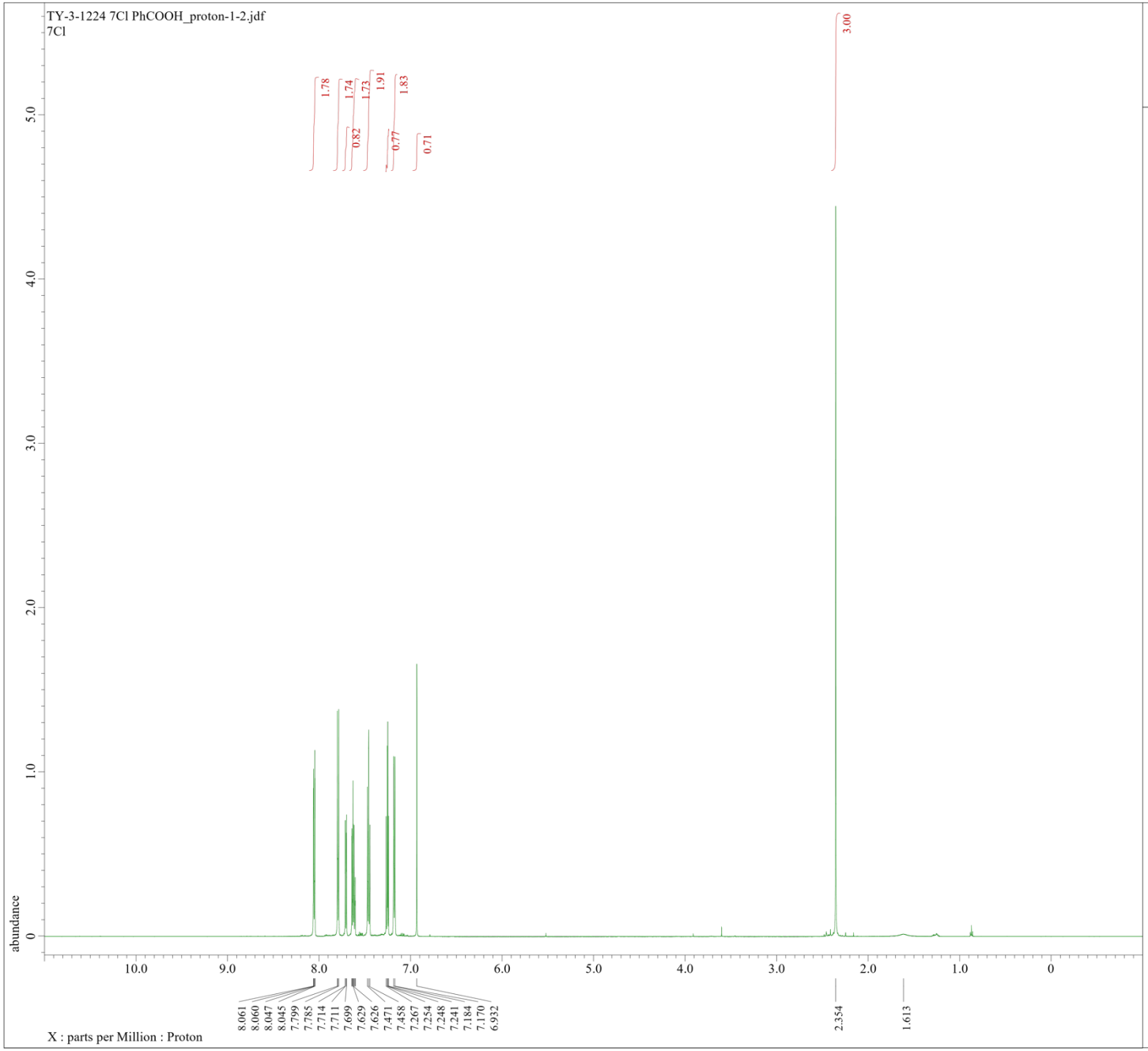


JEOL

Filename	= TY-3-1224 6Cl PhCO
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1224 6Cl PhCO
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 24-DEC-2021 19:07:
Revision_Time	= 24-DEC-2021 19:26:
Comment	= 6Cl
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 512
Total_Scans	= 512
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 21.1[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]

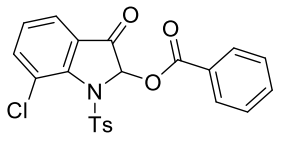


3ia

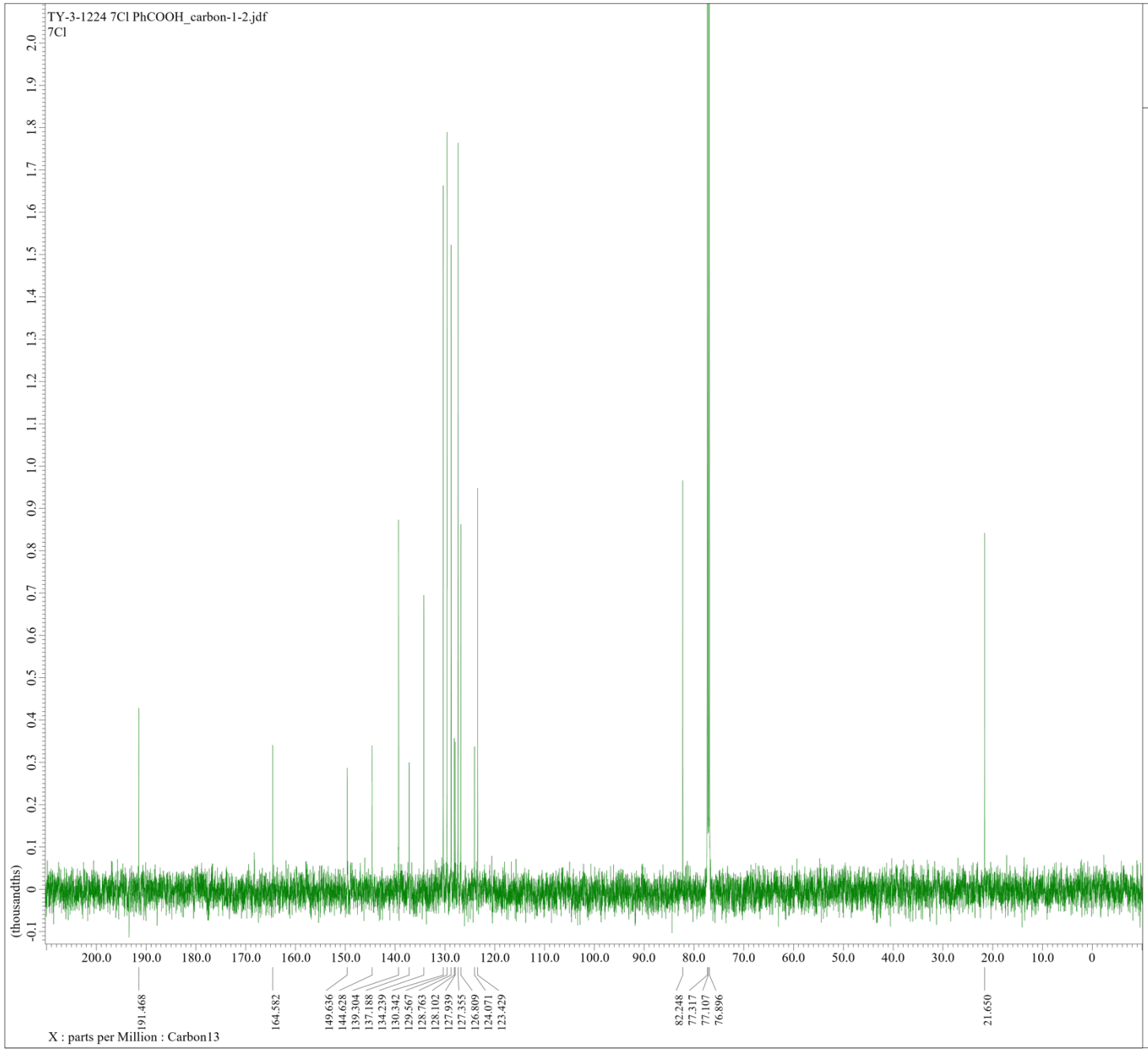


JEOL

```
Filename              = TY-3-1224 7Cl PhCOOH_
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-3-1224 7Cl PhCOOH
Solvent               = CHLOROFORM-D
Actual_Start_Time     = 24-DEC-2021 19:27:52
Revision_Time         = 24-DEC-2021 19:29:45

Comment               = 7Cl
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8

Relaxation_Delay      = 1[s]
Recvr_Gain            = 56
Temp_Get              = 21.1[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```

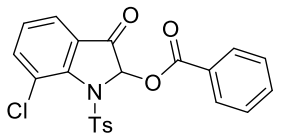


3ja

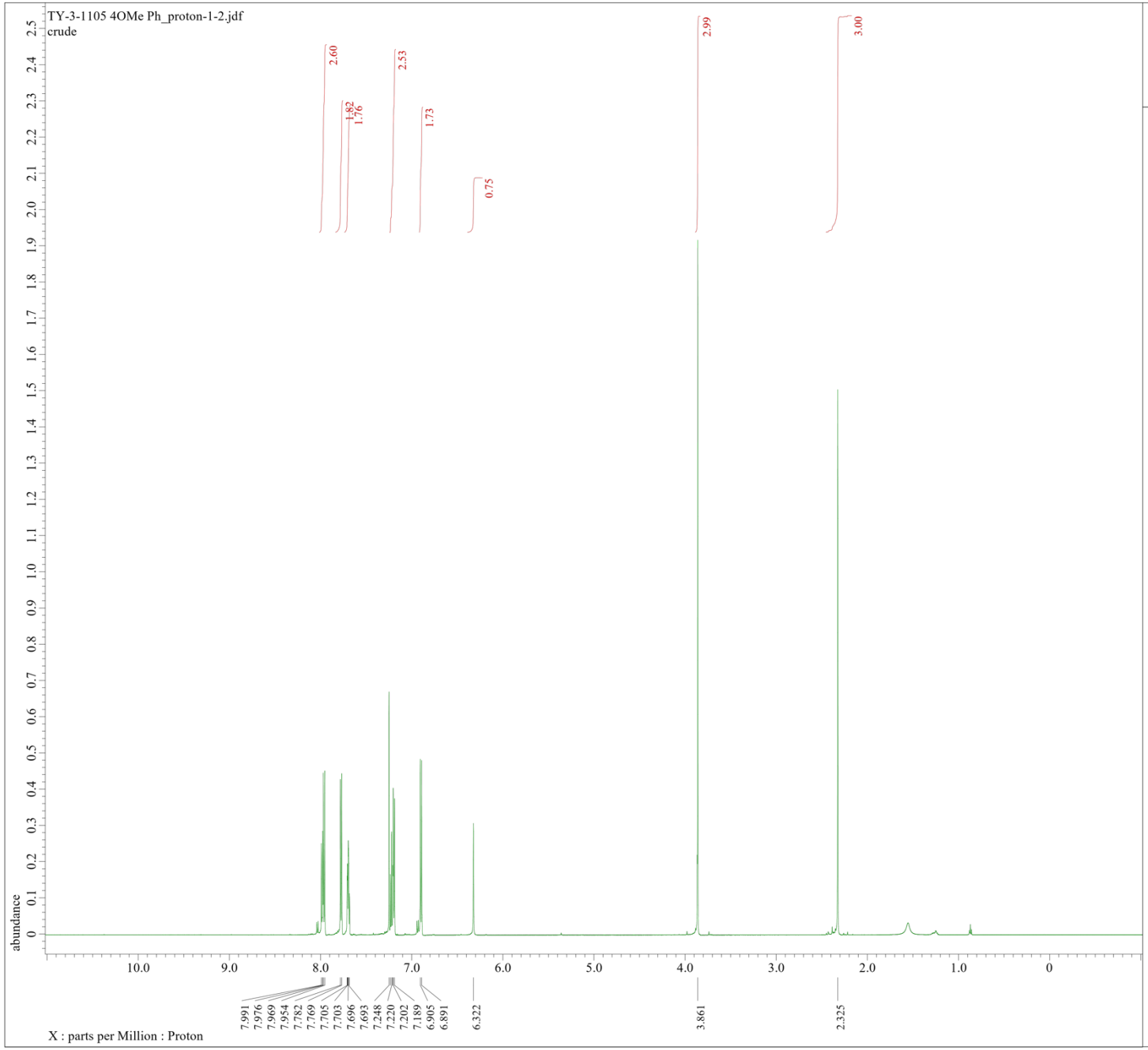


JEOL

Filename	= TY-3-1224 7Cl PhCO
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1224 7Cl PhCO
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 24-DEC-2021 19:30:
Revision_Time	= 24-DEC-2021 19:44:
Comment	= 7Cl
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 221
Total_Scans	= 221
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 21.1[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]

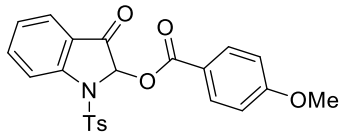


3ja

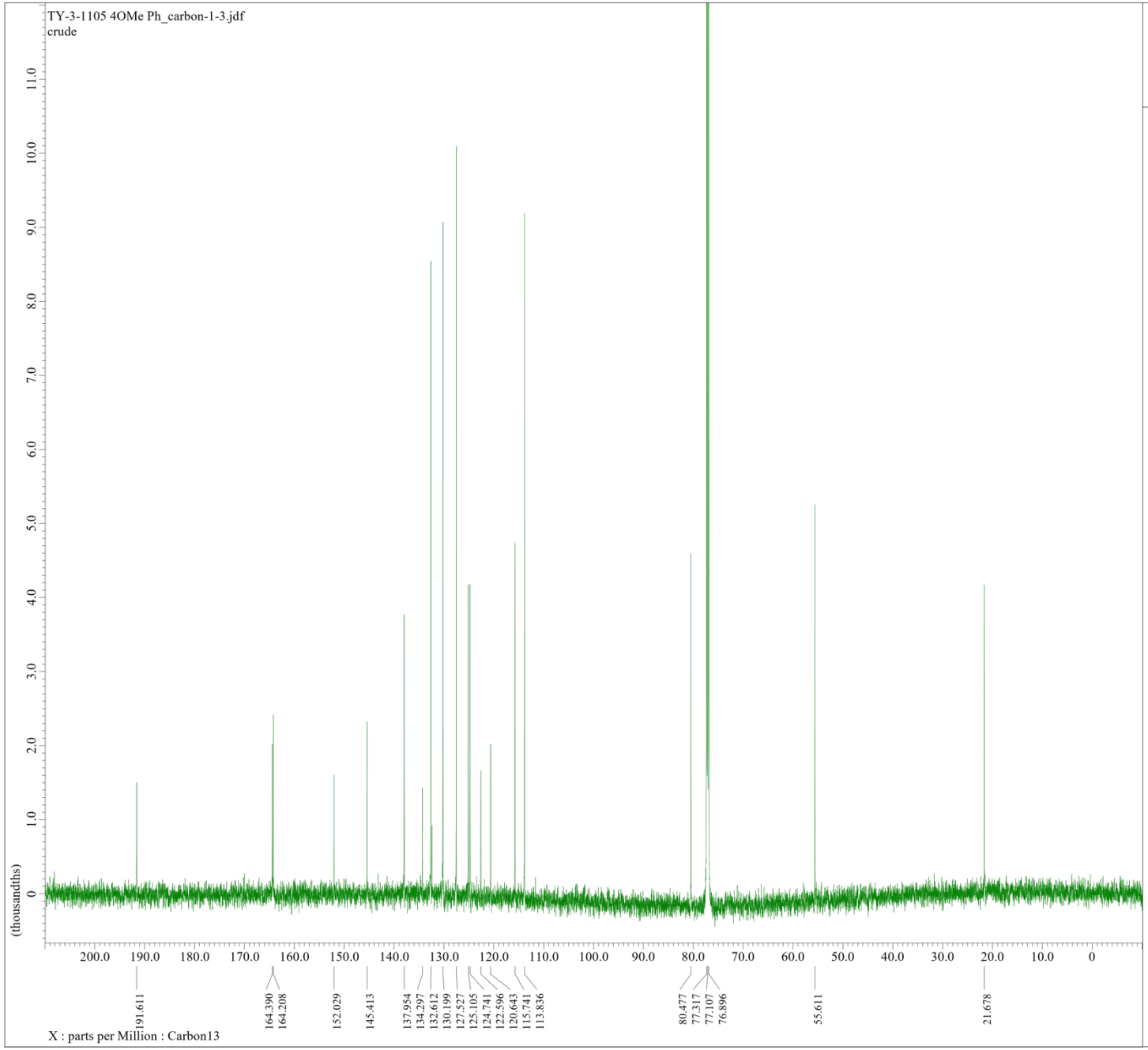


JEOL

```
Filename              = TY-3-1105 4OMe Ph_pro
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-3-1105 4OMe Ph
Solvent               = CHLOROFORM-D
Actual_Start_Time     =  6-NOV-2021 00:14:03
Revision_Time         =  6-NOV-2021 10:03:27

Comment               = crude
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 5[us]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8

Relaxation_Delay      = 1[s]
Recvr_Gain            = 46
Temp_Get              = 20.9[dC]
X_90_Width            = 9.4[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 4.7[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```

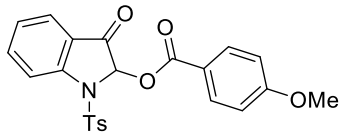


3ab

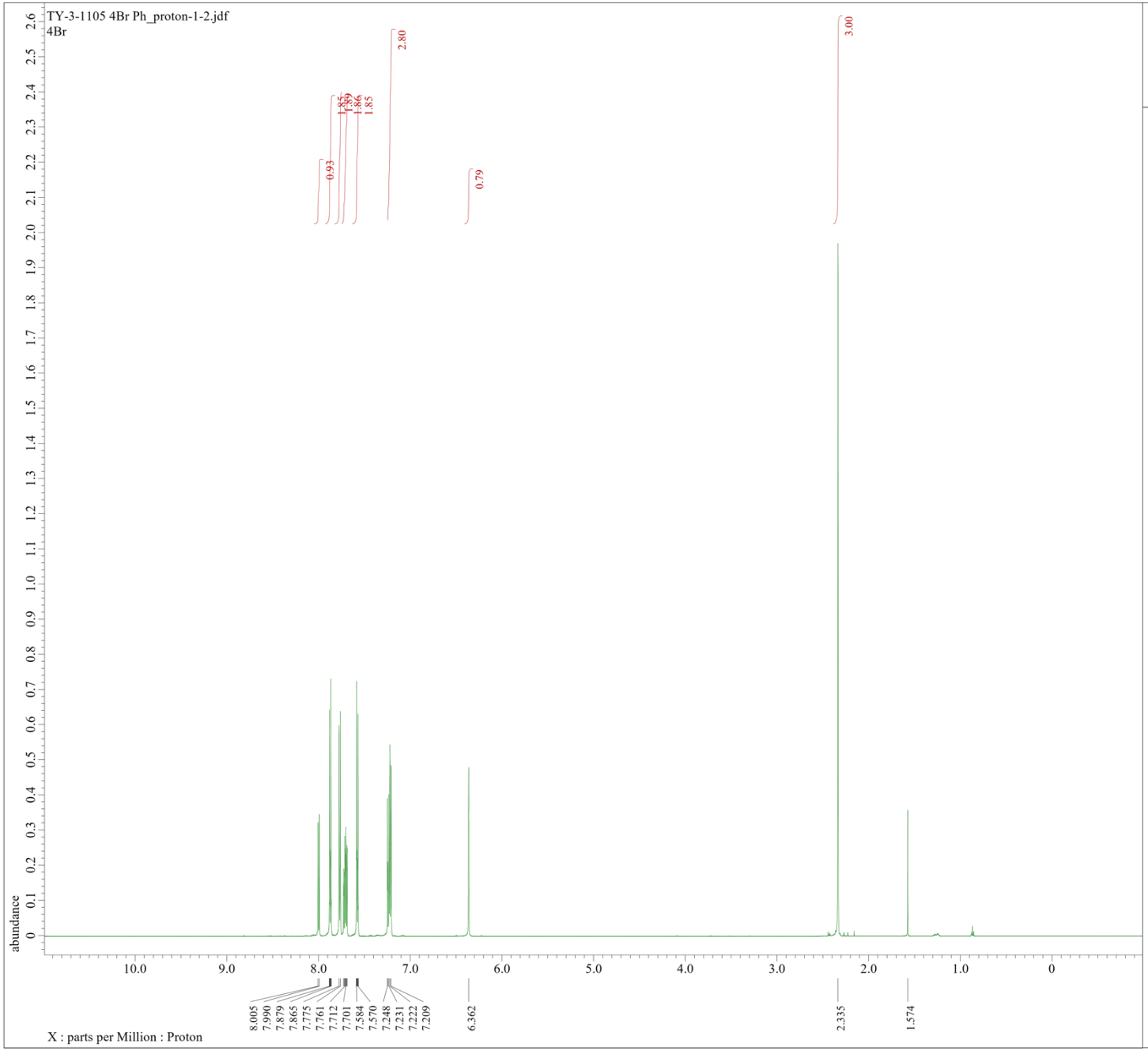


JEOL

Filename	= TY-3-1105 4OMe Ph_
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1105 4OMe Ph
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 6-NOV-2021 00:15:
Revision_Time	= 2-DEC-2021 14:11:
Comment	= crude
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 15[us]
Clipped	= FALSE
Scans	= 512
Total_Scans	= 512
Relaxation_Delay	= 1[s]
Recvr_Gain	= 56
Temp_Get	= 19.6[dC]
X_90_Width	= 9.6[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 11[dB]
X_Pulse	= 3.2[us]
Irr_Atn_Dec	= 26.254[dB]
Irr_Atn_Dec_Calc	= 26.254[dB]
Irr_Atn_Dec_Default_Calc	= 26.254[dB]
Irr_Atn_Noe	= 26.254[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]

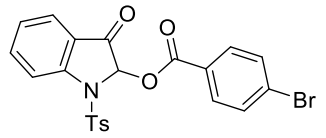


3ab

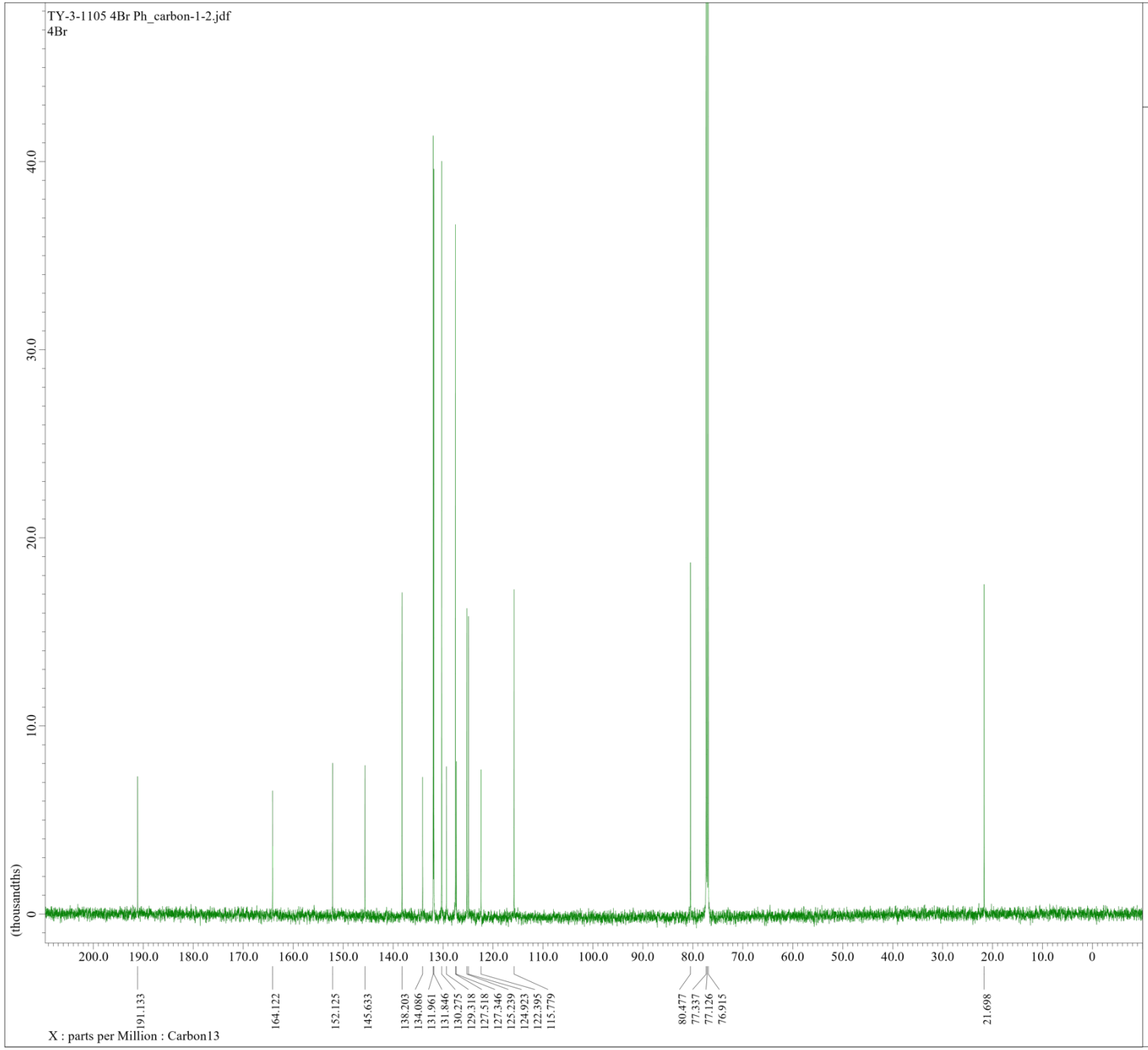


JEOL

```
Filename              = TY-3-1105 4Br Ph_prot
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-3-1105 4Br Ph
Solvent               = CHLOROFORM-D
Actual_Start_Time     =  6-NOV-2021 00:36:00
Revision_Time         =  6-NOV-2021 10:06:29

Comment               = 4Br
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 5[us]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8

Relaxation_Delay      = 1[s]
Recvr_Gain            = 36
Temp_Get              = 19.6[dC]
X_90_Width            = 9.4[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 4.7[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```

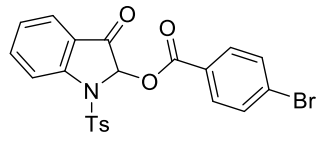


3ac

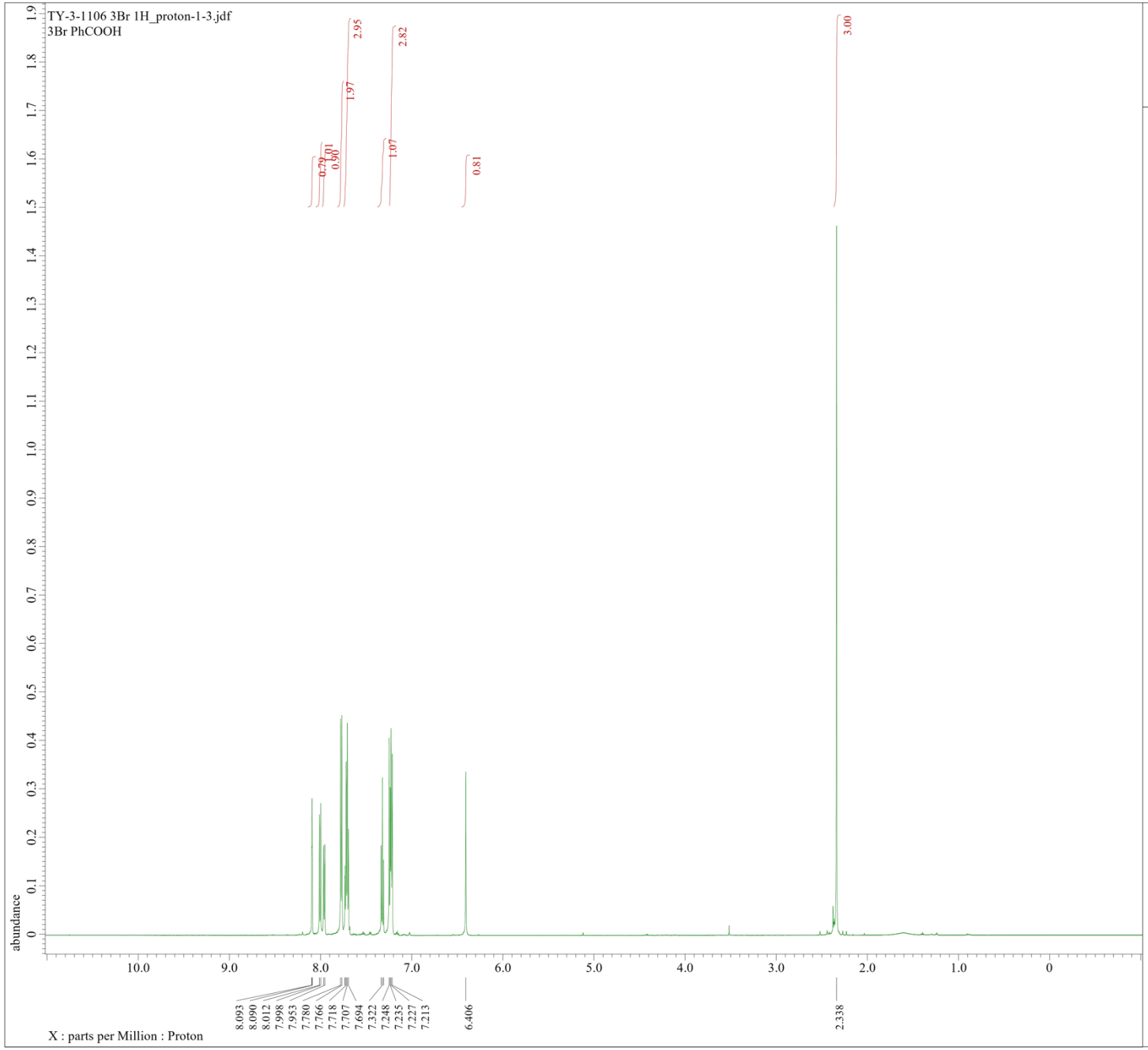


JEOL

Filename	= TY-3-1105 4Br Ph_c
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1105 4Br Ph
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 6-NOV-2021 00:37:
Revision_Time	= 6-NOV-2021 10:07:
Comment	= 4Br
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 15[us]
Clipped	= FALSE
Scans	= 128
Total_Scans	= 128
Relaxation_Delay	= 1[s]
Recvr_Gain	= 56
Temp_Get	= 20.4[dC]
X_90_Width	= 9.6[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 11[dB]
X_Pulse	= 3.2[us]
Irr_Atn_Dec	= 26.254[dB]
Irr_Atn_Dec_Calc	= 26.254[dB]
Irr_Atn_Dec_Default_Calc	= 26.254[dB]
Irr_Atn_Noe	= 26.254[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]

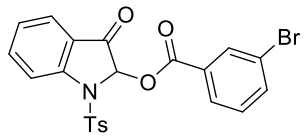


3ac

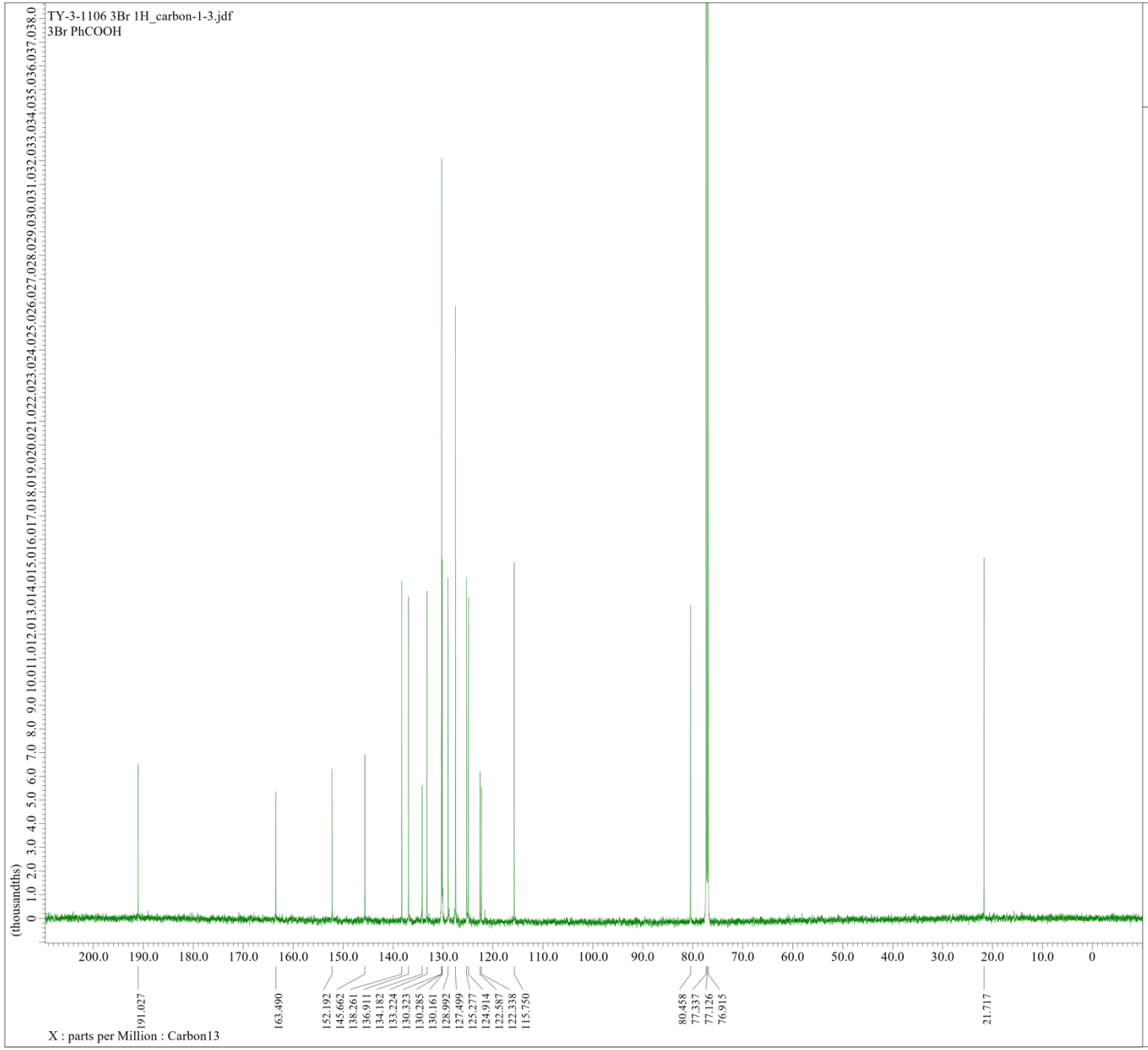


JEOL

```
Filename              = TY-3-1106 3Br 1H_prot
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-3-1106 3Br 1H
Solvent               = CHLOROFORM-D
Actual_Start_Time     =  6-NOV-2021 09:41:30
Revision_Time         =  6-NOV-2021 10:18:36

Comment               = 3Br PhCOOH
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 5[us]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8

Relaxation_Delay      = 1[s]
Recvr_Gain            = 36
Temp_Get              = 19.5[dC]
X_90_Width            = 9.4[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 4.7[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```

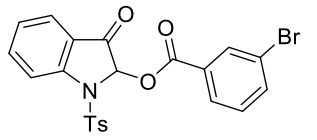


3ad



JEOL

Filename	= TY-3-1106 3Br 1H_c
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1106 3Br 1H
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 6-NOV-2021 09:42:
Revision_Time	= 6-NOV-2021 10:20:
Comment	= 3Br PhCOOH
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 15[us]
Clipped	= FALSE
Scans	= 512
Total_Scans	= 512
Relaxation_Delay	= 1[s]
Recvr_Gain	= 56
Temp_Get	= 19.9[dC]
X_90_Width	= 9.6[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 11[dB]
X_Pulse	= 3.2[us]
Irr_Atn_Dec	= 26.254[dB]
Irr_Atn_Dec_Calc	= 26.254[dB]
Irr_Atn_Dec_Default_Calc	= 26.254[dB]
Irr_Atn_Noe	= 26.254[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]



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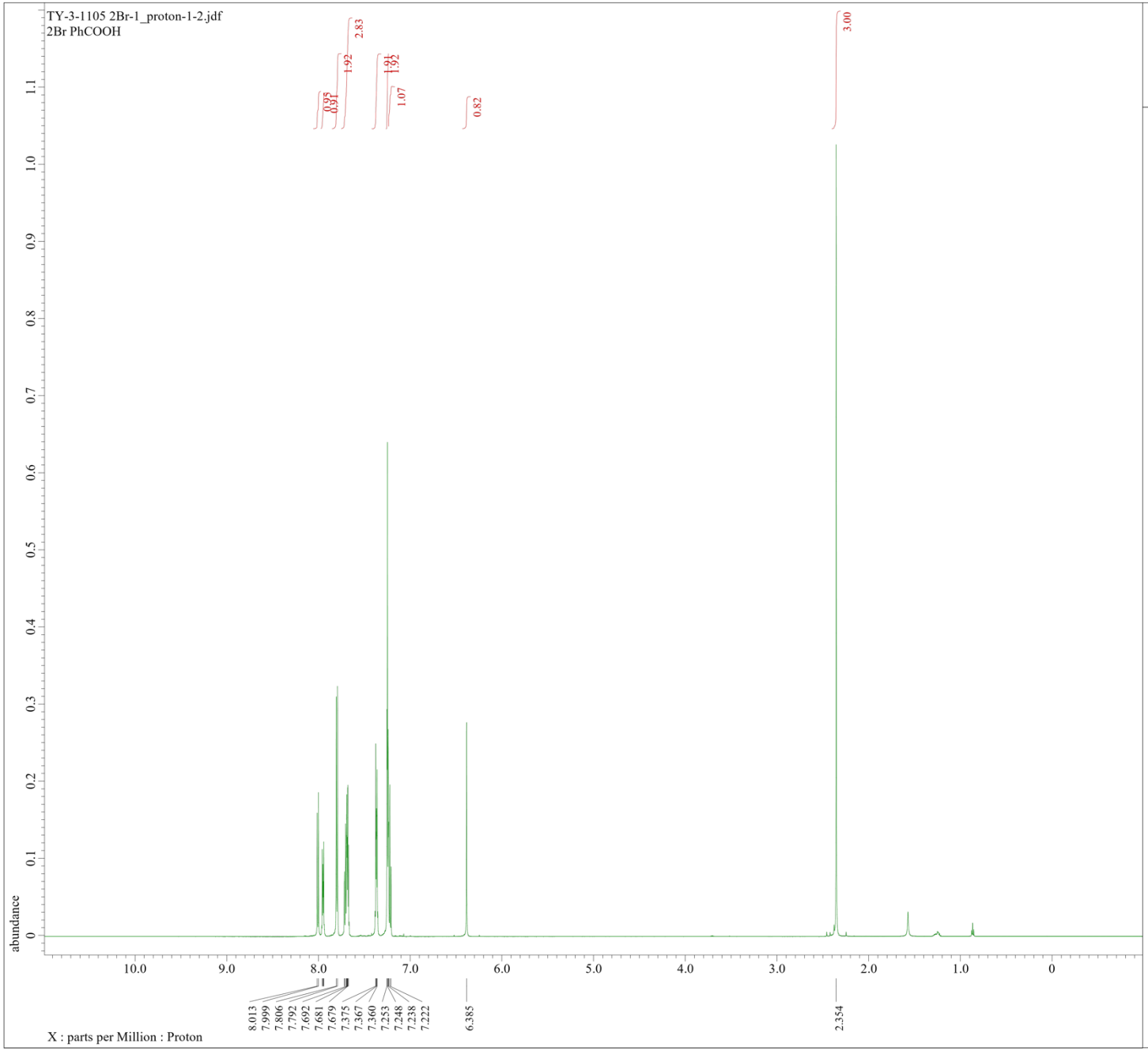
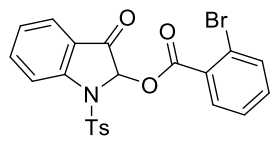


JEOL

Filename	= TY-3-1105 2Br-1_proto
Author	= delta
Experiment	= proton.jxp
Sample_Id	= TY-3-1105 2Br-1
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 5-NOV-2021 16:06:29
Revision_Time	= 5-NOV-2021 16:09:37
Comment	= 2Br PhCOOH
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (600[M
X_Acq_Duration	= 2.18103808[s]
X_Domain	= Proton
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.45849727[Hz]
X_Sweep	= 15.02403846[kHz]
X_Sweep_Clipped	= 12.01923077[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 5[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 19.8[dC]
X_90_Width	= 9.4[us]
X_Acq_Time	= 2.18103808[s]
X_Angle	= 45[deg]
X_Atn	= 8.1[dB]
X_Pulse	= 4.7[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 100
Dante_Presat	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\JEOL
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180
Presat_Time	= 1[s]
Presat_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 3.18103808[s]



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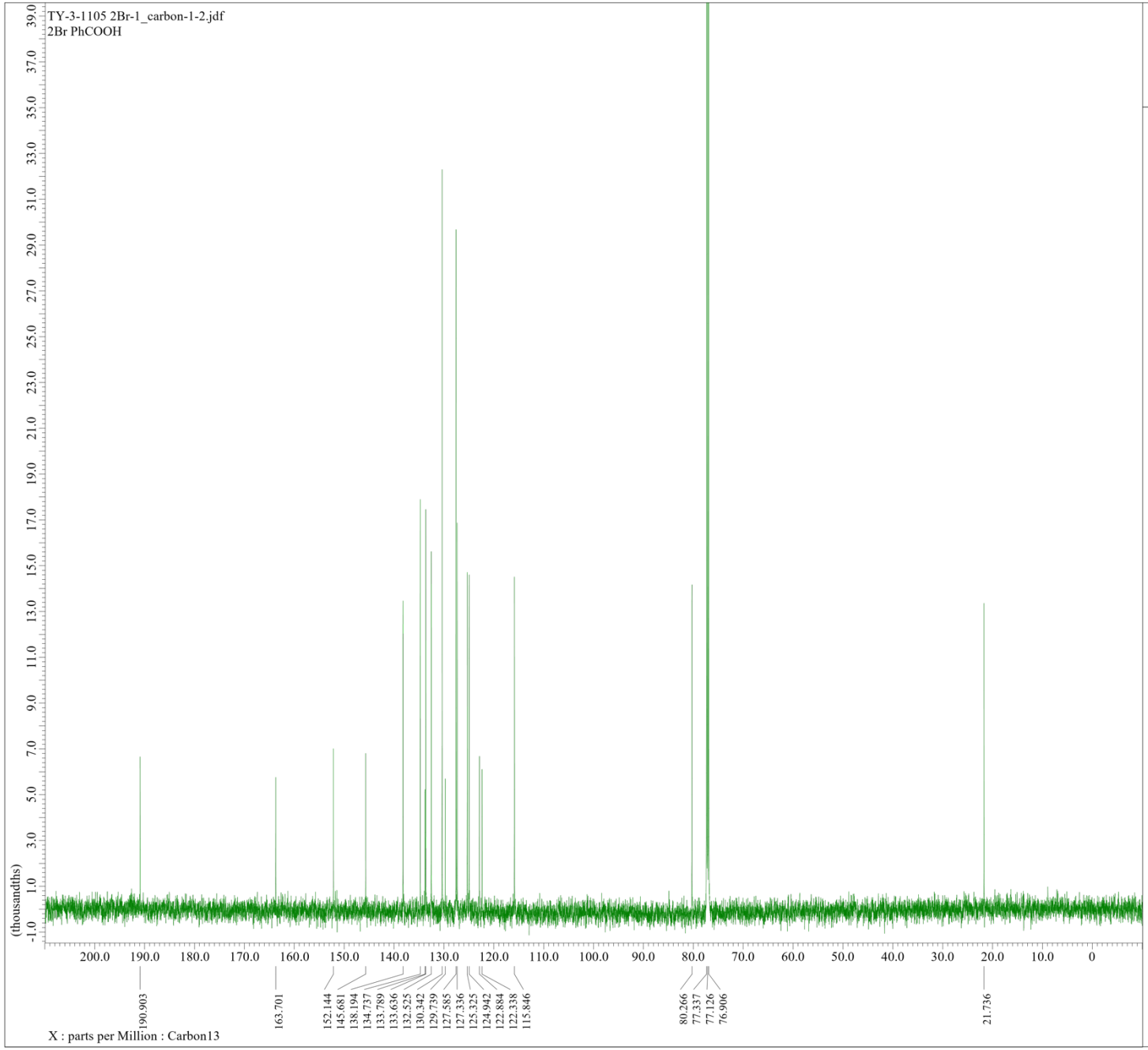
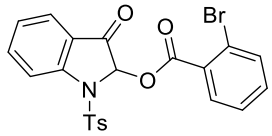


JEOL

Filename	= TY-3-1105 2Br-1_ca
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1105 2Br-1
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 5-NOV-2021 16:07:
Revision_Time	= 5-NOV-2021 16:10:
Comment	= 2Br PhCOOH
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 15[us]
Clipped	= FALSE
Scans	= 47
Total_Scans	= 47
Relaxation_Delay	= 1[s]
Recvr_Gain	= 56
Temp_Get	= 20[dC]
X_90_Width	= 9.6[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 11[dB]
X_Pulse	= 3.2[us]
Irr_Atn_Dec	= 26.254[dB]
Irr_Atn_Dec_Calc	= 26.254[dB]
Irr_Atn_Dec_Default_Calc	= 26.254[dB]
Irr_Atn_Noe	= 26.254[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]



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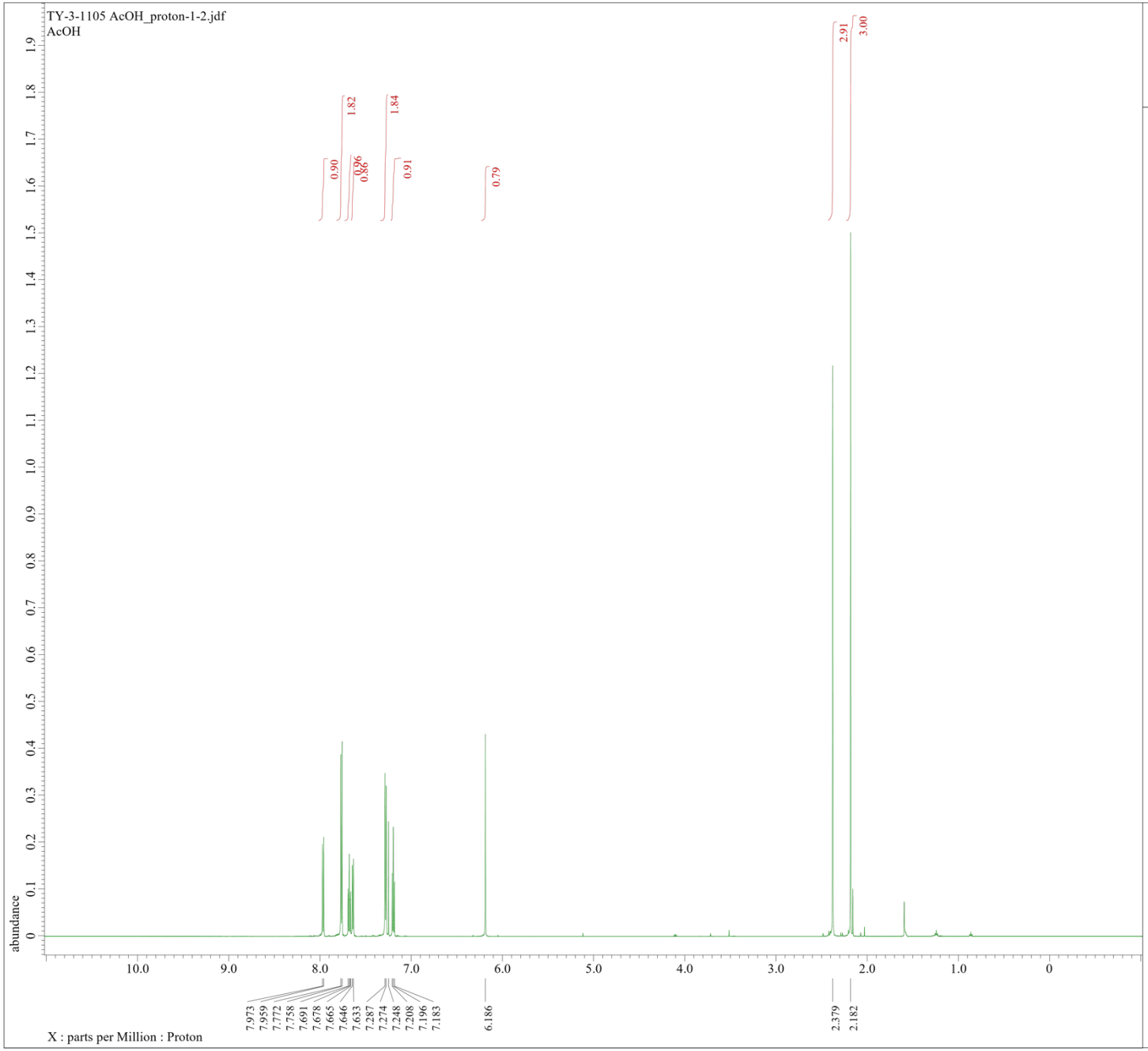
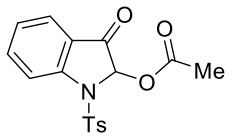


JEOL

Filename	= TY-3-1105 AcOH_proton
Author	= delta
Experiment	= proton.jxp
Sample_Id	= TY-3-1105 AcOH
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 6-NOV-2021 00:59:13
Revision_Time	= 6-NOV-2021 10:12:38
Comment	= AcOH
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (600[M
X_Acq_Duration	= 2.18103808[s]
X_Domain	= Proton
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.45849727[Hz]
X_Sweep	= 15.02403846[kHz]
X_Sweep_Clipped	= 12.01923077[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 5[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 19.3[dC]
X_90_Width	= 9.4[us]
X_Acq_Time	= 2.18103808[s]
X_Angle	= 45[deg]
X_Atn	= 8.1[dB]
X_Pulse	= 4.7[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 100
Dante_Presat	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\JEOL
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180
Presat_Time	= 1[s]
Presat_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 3.18103808[s]



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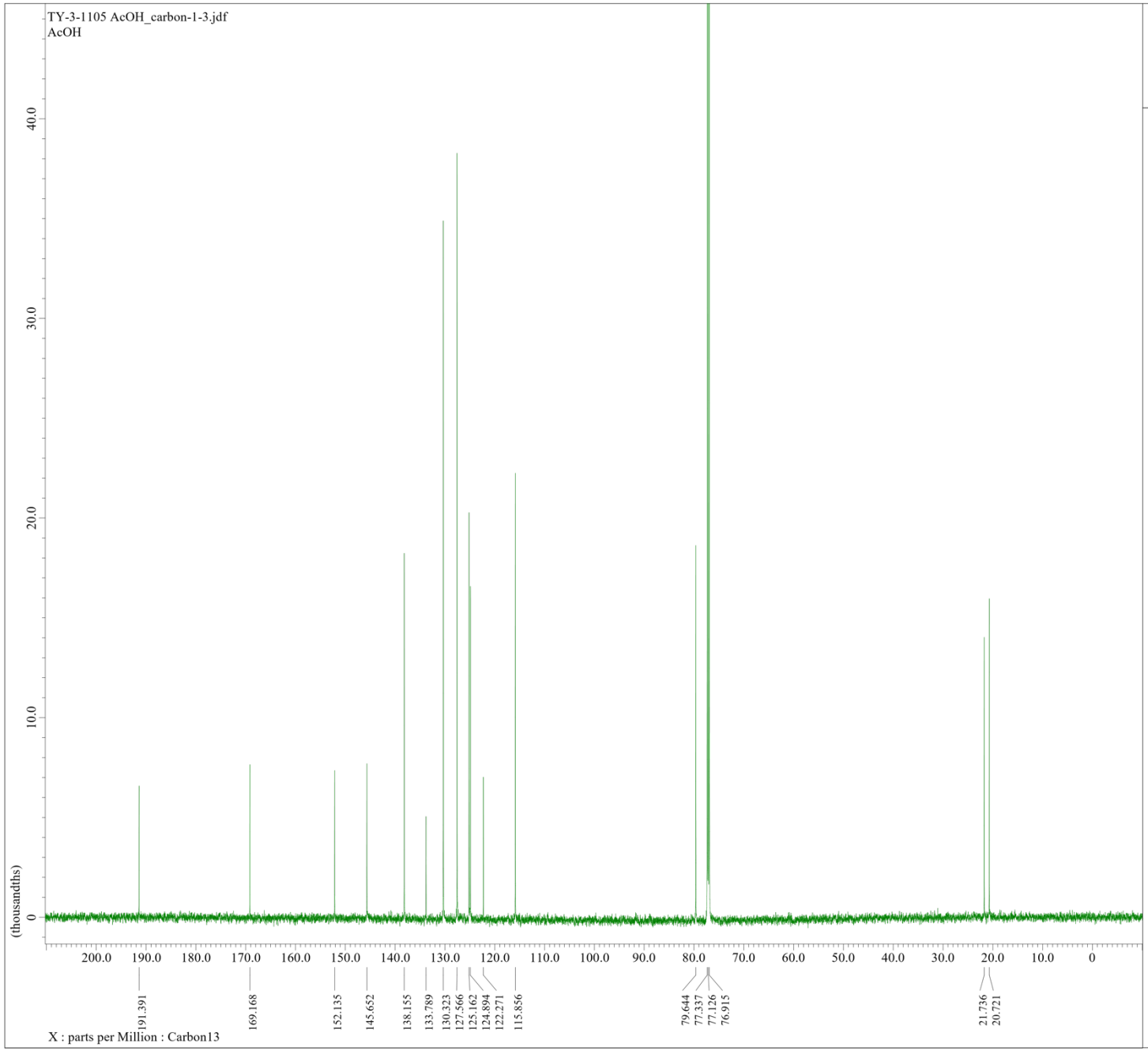

JEOL

```
Filename                = TY-3-1105 AcOH_car
Author                  = delta
Experiment              = carbon.jxp
Sample_Id               = TY-3-1105 AcOH
Solvent                 = CHLOROFORM-D
Actual_Start_Time       =  6-NOV-2021 01:00:
Revision_Time           =  8-NOV-2021 14:17:

Comment                 = AcOH
Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Carbon13
Dim_Title               = Carbon13
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ600R/S3

Field_Strength          = 14.09636928[T] (60
X_Acq_Duration          = 0.69206016[s]
X_Domain                = Carbon13
X_Freq                  = 150.91343039[MHz]
X_Offset                = 100[ppm]
X_Points                = 32768
X_Prescans              = 4
X_Resolution            = 1.44496109[Hz]
X_Sweep                 = 47.34848485[kHz]
X_Sweep_Clipped         = 37.87878788[kHz]
Irr_Domain              = Proton
Irr_Freq                = 600.1723046[MHz]
Irr_Offset              = 5[ppm]
Blanking                = 15[us]
Clipped                 = FALSE
Scans                   = 256
Total_Scans             = 256

Relaxation_Delay        = 1[s]
Recvr_Gain              = 56
Temp_Get                = 20.9[dC]
X_90_Width              = 9.6[us]
X_Acq_Time              = 0.69206016[s]
X_Angle                 = 30[deg]
X_Atn                   = 11[dB]
X_Pulse                 = 3.2[us]
Irr_Atn_Dec             = 26.254[dB]
Irr_Atn_Dec_Calc        = 26.254[dB]
Irr_Atn_Dec_Default_Calc = 26.254[dB]
Irr_Atn_Noe             = 26.254[dB]
Irr_Dec_Bandwidth_Hz    = 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm   = 12.05794078[ppm]
Irr_Dec_Freq            = 600.1723046[MHz]
Irr_Dec_Merit_Factor    = 2.2
Irr_Decoupling          = TRUE
Irr_Noe                 = TRUE
Irr_Noise               = WALTZ
Irr_Offset_Default      = 5[ppm]
Irr_Pwidth              = 76[us]
Irr_Pwidth_Default      = 76[us]
Irr_Pwidth_Default_Calc = 76[us]
Irr_Pwidth_Temp1        = 76[us]
Irr_Wurst               = FALSE
Decimation_Rate         = 0
Experiment_Path         = c:\Program Files\J
Initial_Wait            = 1[s]
Noe_Time                = 1[s]
Noe_Time_Flag           = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 1[s]
Repetition_Time         = 1.69206016[s]
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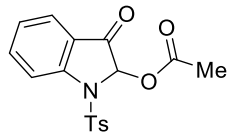

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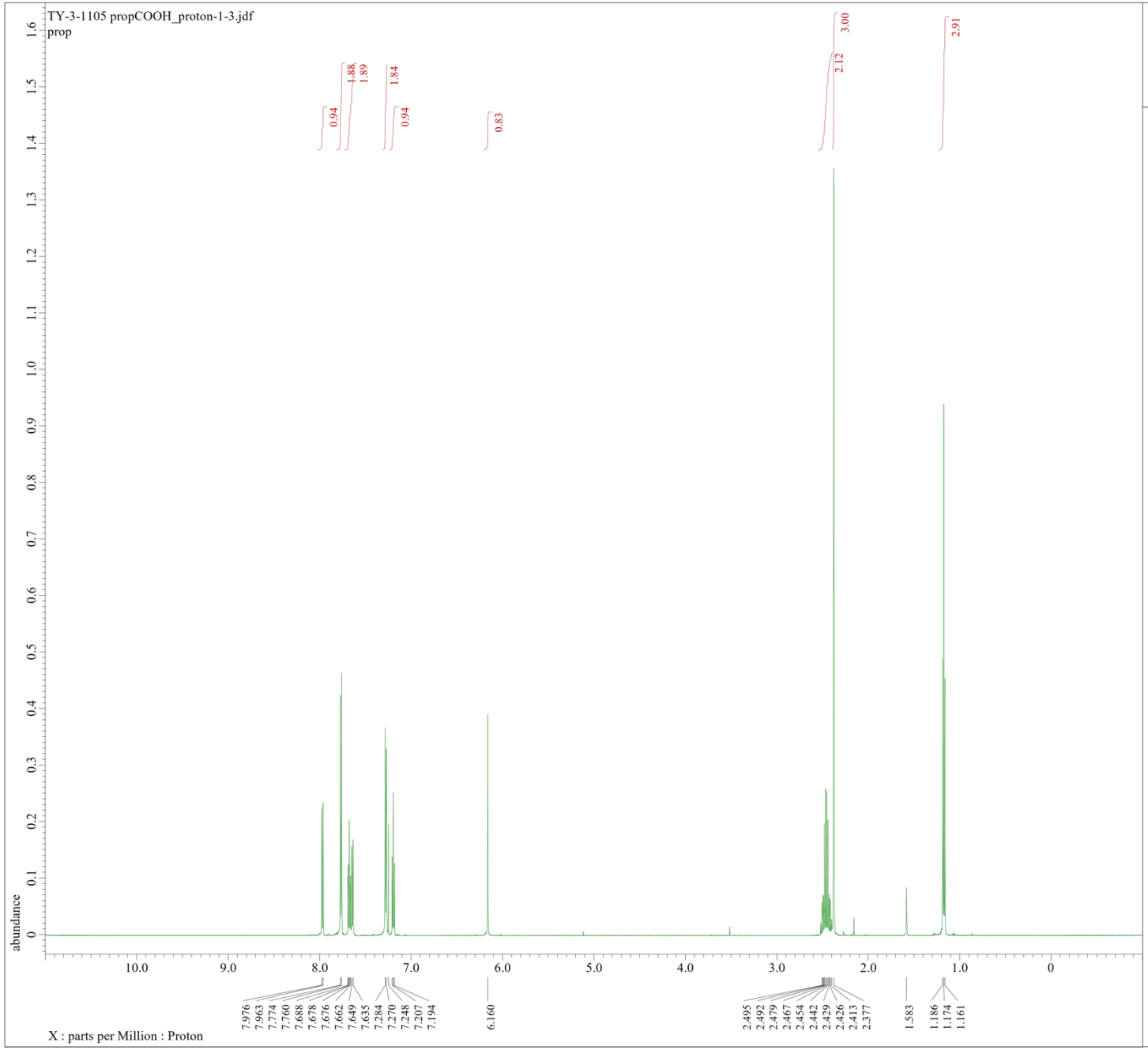

JEOL

```
Filename                = TY-3-1105 propCOOH_pr
Author                  = delta
Experiment              = proton.jxp
Sample_Id               = TY-3-1105 propCOOH
Solvent                 = CHLOROFORM-D
Actual_Start_Time       =  6-NOV-2021 00:45:45
Revision_Time           =  8-NOV-2021 14:15:51

Comment                 = prop
Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Proton
Dim_Title               = Proton
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ600R/S3

Field_Strength          = 14.09636928[T] (600[M
X_Acq_Duration          = 2.18103808[s]
X_Domain                = Proton
X_Freq                  = 600.1723046[MHz]
X_Offset                = 5[ppm]
X_Points                = 32768
X_Prescans              = 1
X_Resolution            = 0.45849727[Hz]
X_Sweep                 = 15.02403846[kHz]
X_Sweep_Clipped         = 12.01923077[kHz]
Irr_Domain              = Proton
Irr_Freq                = 600.1723046[MHz]
Irr_Offset              = 5[ppm]
Tri_Domain              = Proton
Tri_Freq                = 600.1723046[MHz]
Tri_Offset              = 5[ppm]
Blanking                = 5[us]
Clipped                 = FALSE
Scans                   = 8
Total_Scans             = 8

Relaxation_Delay        = 1[s]
Recvr_Gain              = 36
Temp_Get                = 21[dC]
X_90_Width              = 9.4[us]
X_Acq_Time              = 2.18103808[s]
X_Angle                 = 45[deg]
X_Atn                   = 8.1[dB]
X_Pulse                 = 4.7[us]
Irr_Mode                = Off
Tri_Mode                = Off
Dante_Loop              = 100
Dante_Presat            = FALSE
Decimation_Rate         = 0
Experiment_Path         = c:\Program Files\JEOL
Initial_Wait            = 1[s]
Phase                   = {0, 90, 270, 180, 180
Presat_Time             = 1[s]
Presat_Time_Flag        = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 1[s]
Repetition_Time         = 3.18103808[s]
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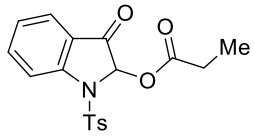

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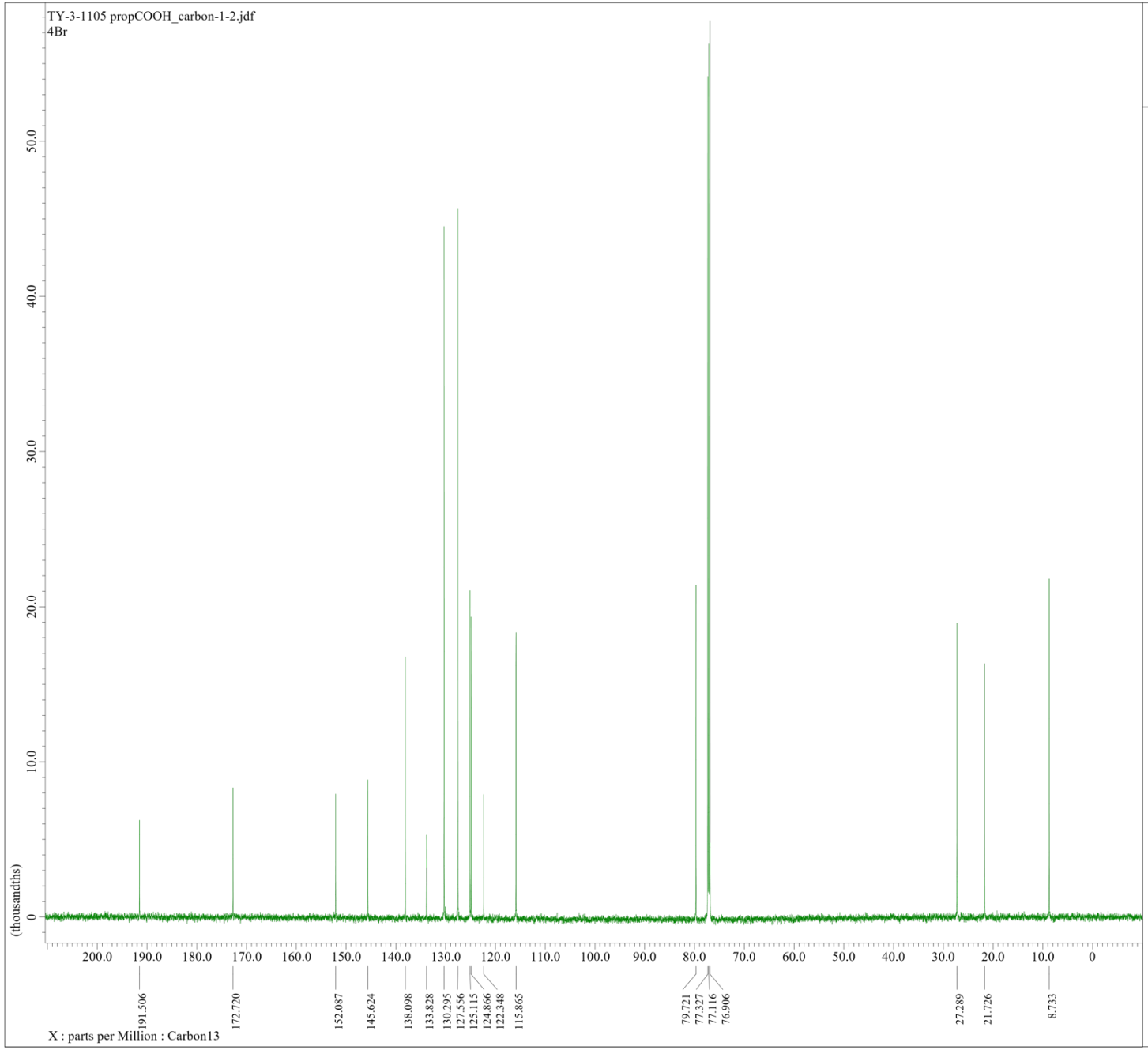
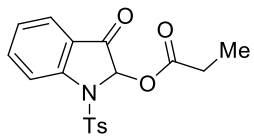


JEOL

Filename	= TY-3-1105 propCOOH
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1105 propCOOH
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 6-NOV-2021 00:46:
Revision_Time	= 6-NOV-2021 10:11:
Comment	= 4Br
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 15[us]
Clipped	= FALSE
Scans	= 256
Total_Scans	= 256
Relaxation_Delay	= 1[s]
Recvr_Gain	= 56
Temp_Get	= 19.6[dC]
X_90_Width	= 9.6[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 11[dB]
X_Pulse	= 3.2[us]
Irr_Atn_Dec	= 26.254[dB]
Irr_Atn_Dec_Calc	= 26.254[dB]
Irr_Atn_Dec_Default_Calc	= 26.254[dB]
Irr_Atn_Noe	= 26.254[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]



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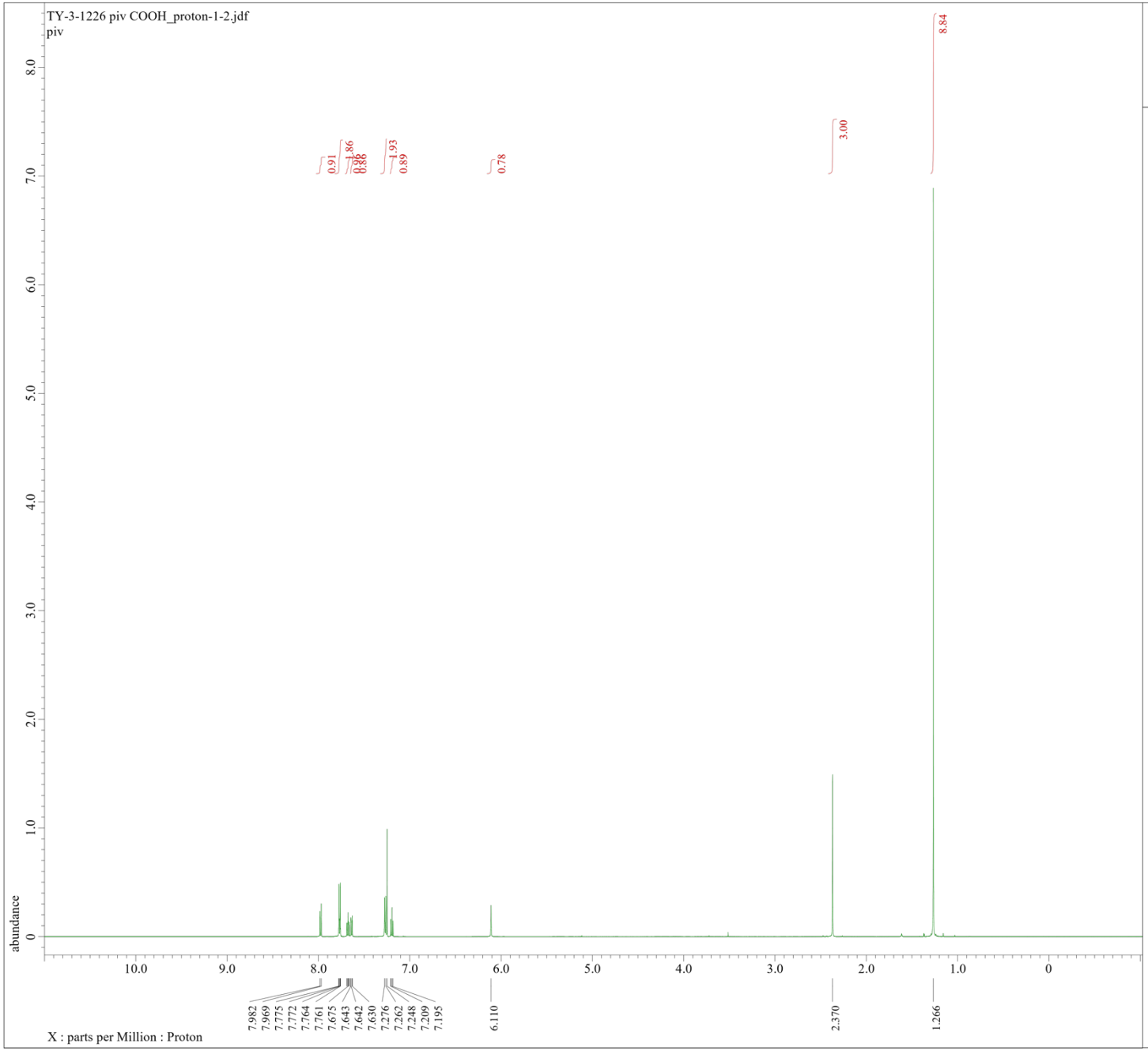
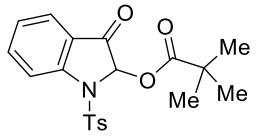


JEOL

Filename	= TY-3-1226 piv COOH_pr
Author	= delta
Experiment	= proton.jxp
Sample_Id	= TY-3-1226 piv COOH
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 26-DEC-2021 17:06:39
Revision_Time	= 26-DEC-2021 17:54:34
Comment	= piv
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (600[M
X_Acq_Duration	= 2.18103808[s]
X_Domain	= Proton
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.45849727[Hz]
X_Sweep	= 15.02403846[kHz]
X_Sweep_Clipped	= 12.01923077[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 21.1[dC]
X_90_Width	= 7.7[us]
X_Acq_Time	= 2.18103808[s]
X_Angle	= 45[deg]
X_Atn	= 8.1[dB]
X_Pulse	= 3.85[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 100
Dante_Presat	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\JEOL
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180
Presat_Time	= 1[s]
Presat_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 3.18103808[s]



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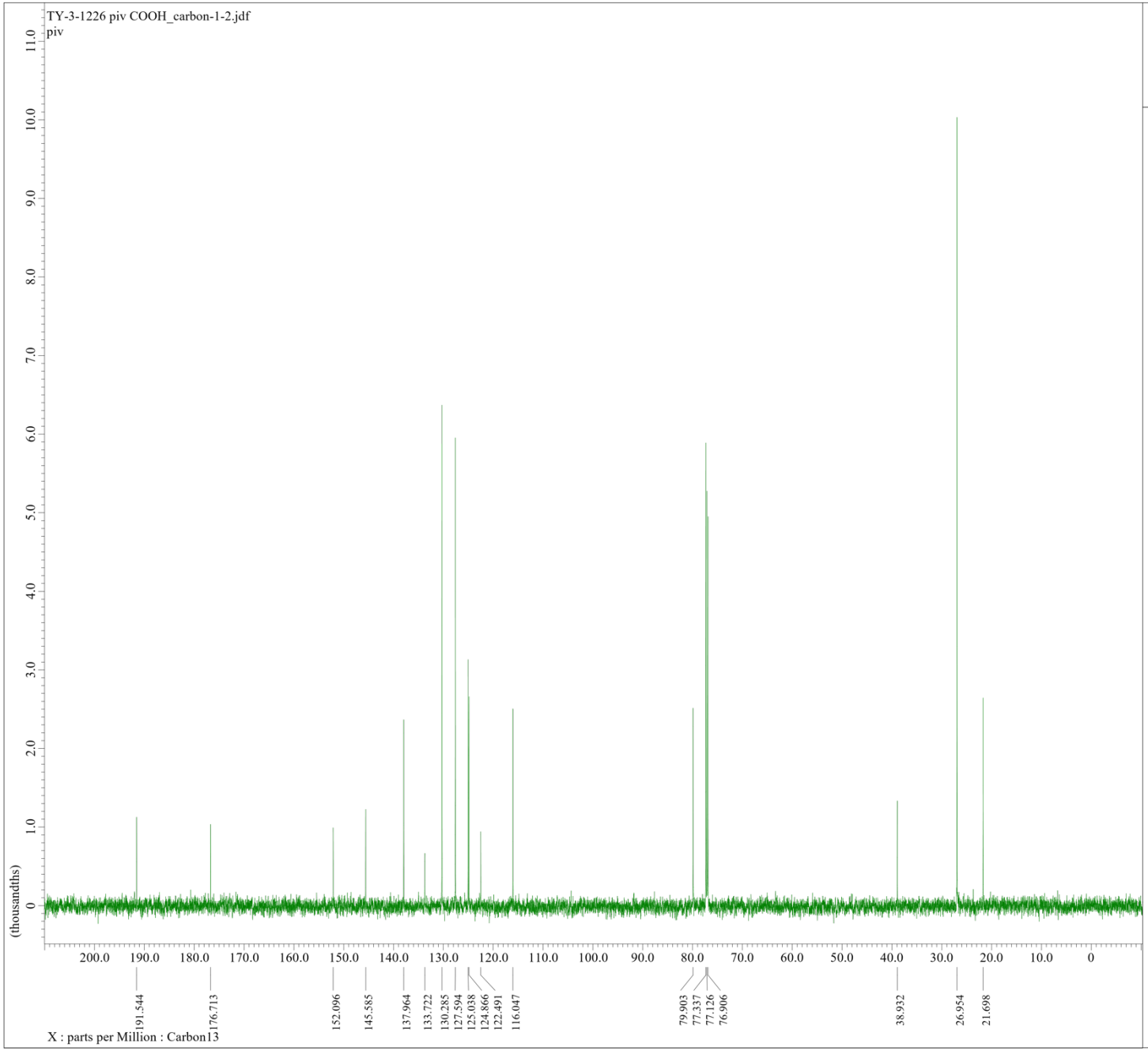
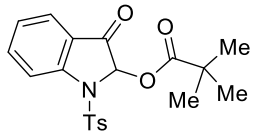
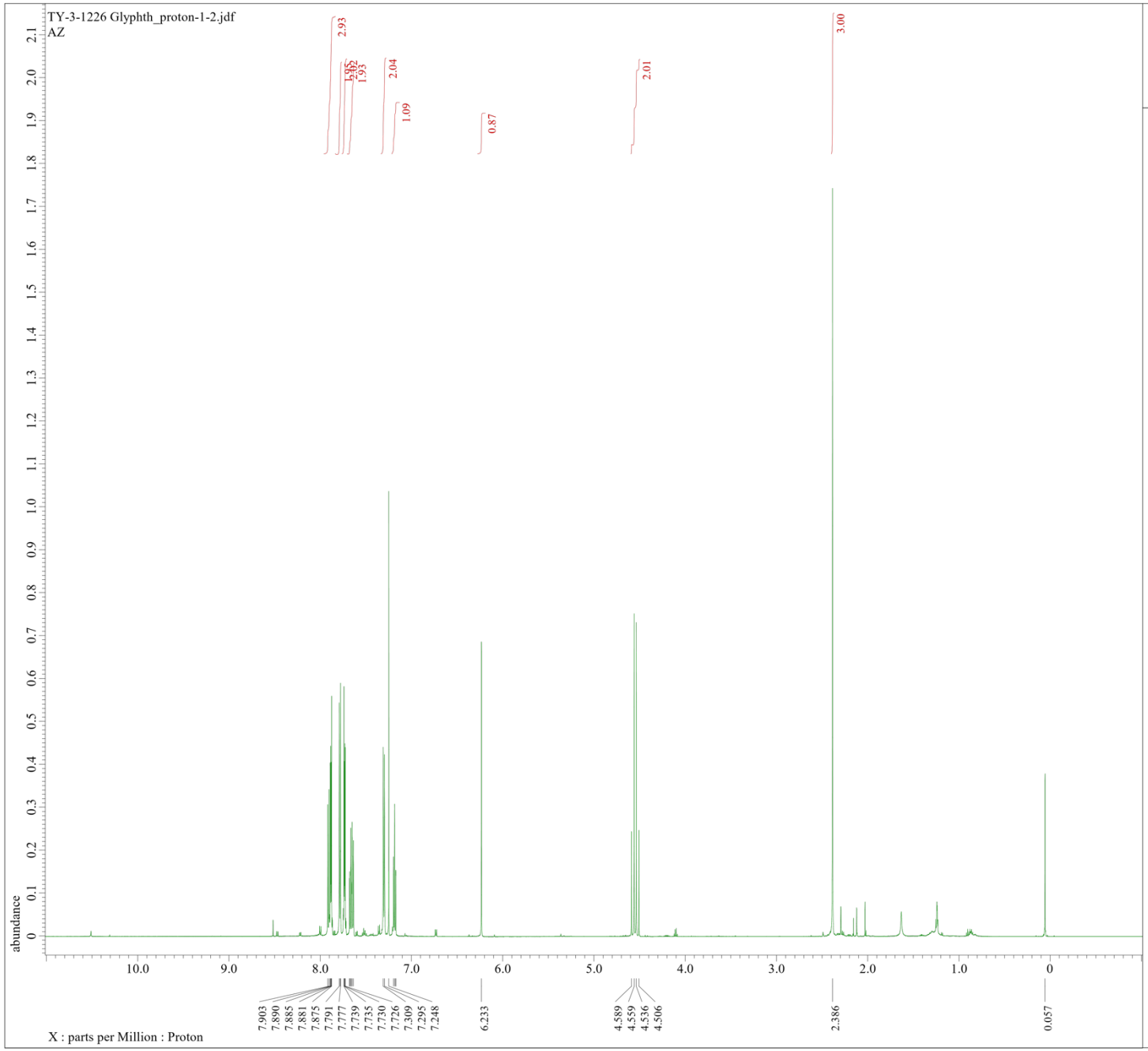


JEOL

Filename	= TY-3-1226 piv COOH
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1226 piv COO
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 26-DEC-2021 17:08:
Revision_Time	= 26-DEC-2021 17:13:
Comment	= piv
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 56
Total_Scans	= 56
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 21.2[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]

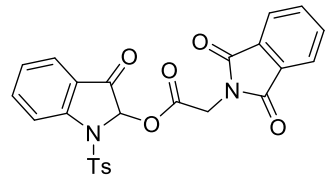


3ah

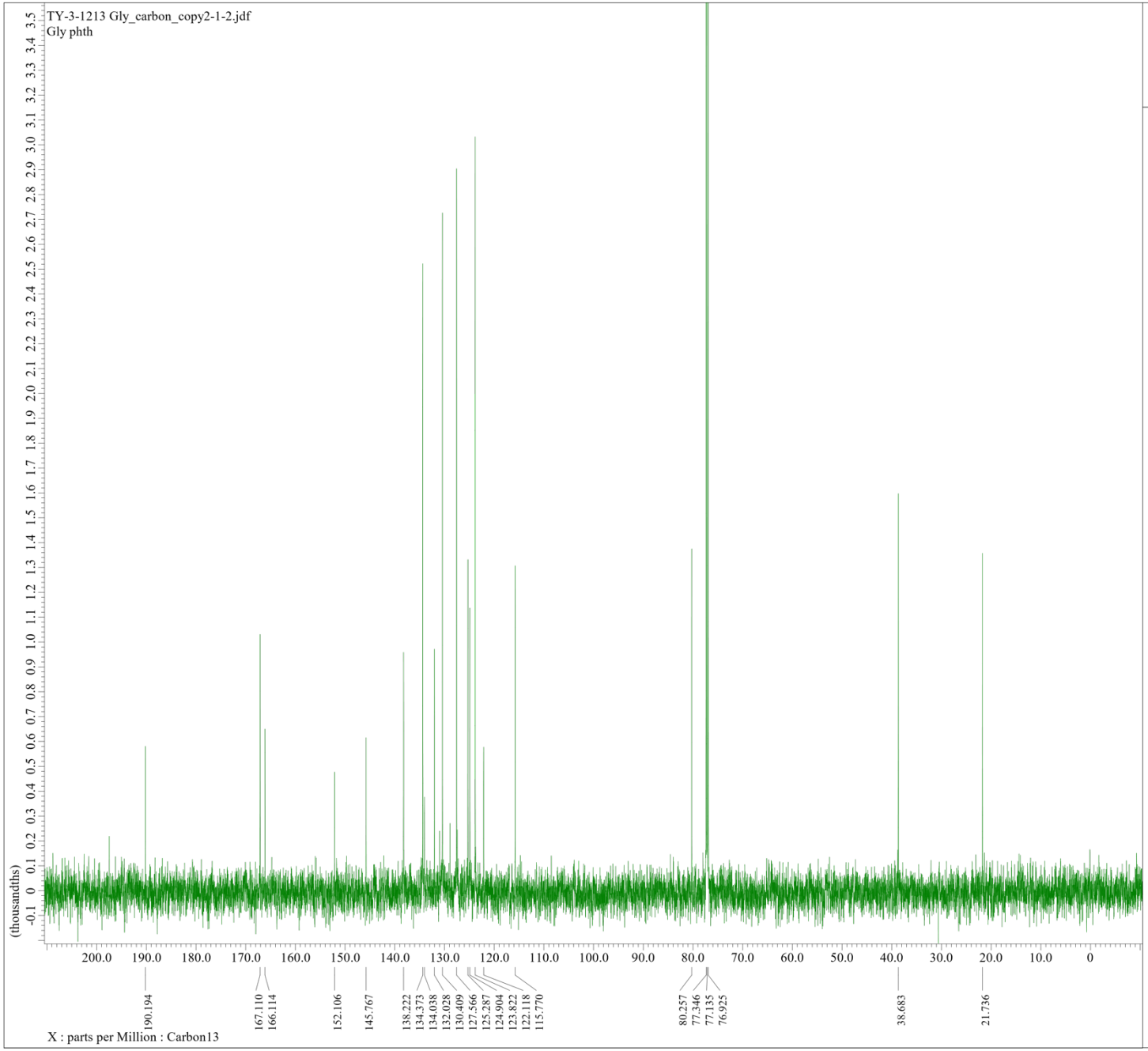


JEOL

Filename	= TY-3-1226 Glyphth_pro
Author	= delta
Experiment	= proton.jxp
Sample_Id	= TY-3-1226 Glyphth
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 26-DEC-2021 17:38:47
Revision_Time	= 26-DEC-2021 17:52:26
Comment	= AZ
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Proton
Dim_Title	= Proton
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (600[M
X_Acq_Duration	= 2.18103808[s]
X_Domain	= Proton
X_Freq	= 600.1723046[MHz]
X_Offset	= 5[ppm]
X_Points	= 32768
X_Prescans	= 1
X_Resolution	= 0.45849727[Hz]
X_Sweep	= 15.02403846[kHz]
X_Sweep_Clipped	= 12.01923077[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Tri_Domain	= Proton
Tri_Freq	= 600.1723046[MHz]
Tri_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 8
Total_Scans	= 8
Relaxation_Delay	= 1[s]
Recvr_Gain	= 46
Temp_Get	= 21.1[dC]
X_90_Width	= 7.7[us]
X_Acq_Time	= 2.18103808[s]
X_Angle	= 45[deg]
X_Atn	= 8.1[dB]
X_Pulse	= 3.85[us]
Irr_Mode	= Off
Tri_Mode	= Off
Dante_Loop	= 100
Dante_Presat	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\JEOL
Initial_Wait	= 1[s]
Phase	= {0, 90, 270, 180, 180
Presat_Time	= 1[s]
Presat_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 3.18103808[s]

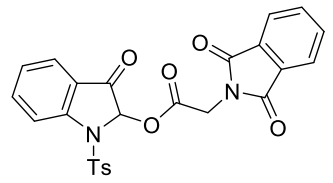


3ai

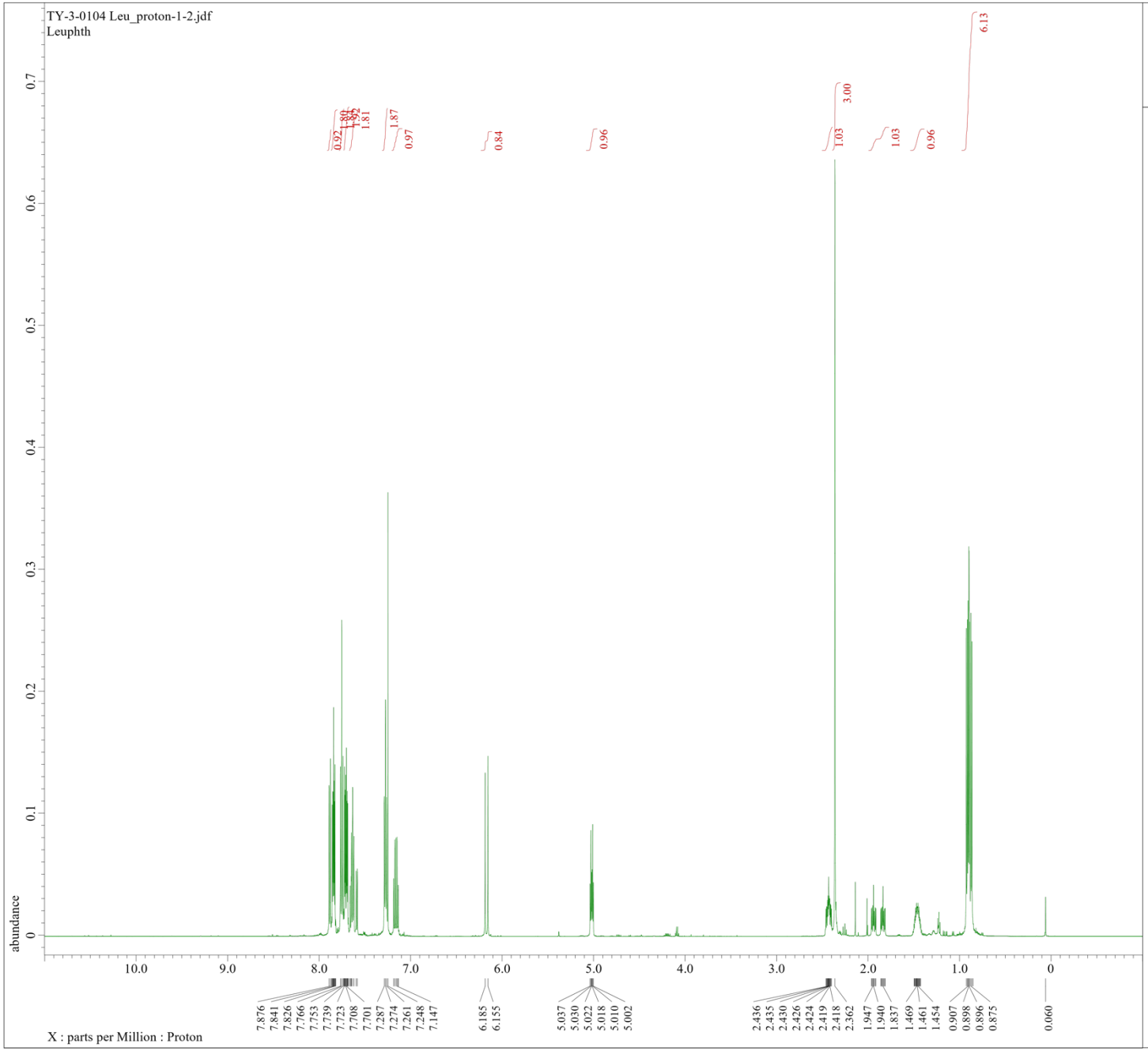


JEOL

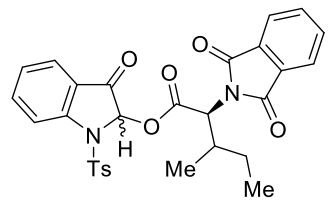
Filename	= TY-3-1213 Gly_carb
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1213 Gly
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 14-DEC-2021 12:23:
Revision_Time	= 14-DEC-2021 12:25:
Comment	= Gly phth
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Incomplete_Copy	= TRUE
Scans	= 65
Total_Scans	= 65
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 20.9[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]



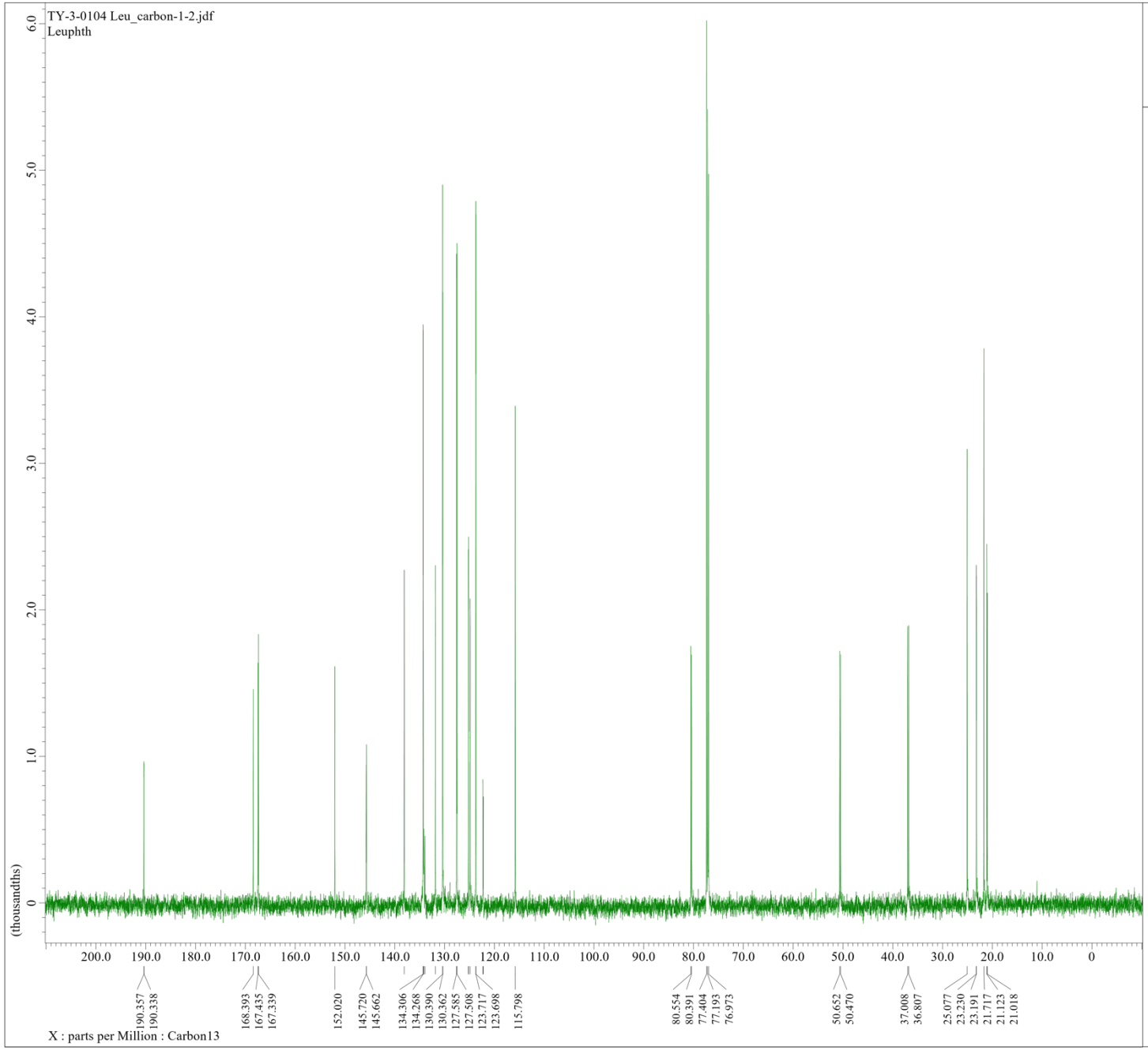
3ai




```
Filename              = TY-3-0104 Leu_proton-
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-3-0104 Leu
Solvent               = CHLOROFORM-D
Actual_Start_Time     =  4-JAN-2022 23:16:50
Revision_Time         =  4-JAN-2022 23:21:14

Comment               = Leuphth
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8

Relaxation_Delay      = 1[s]
Recvr_Gain            = 26
Temp_Get              = 20.5[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```

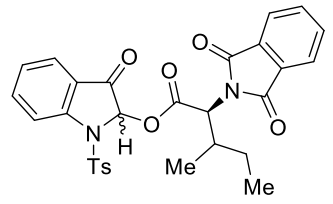


3aj

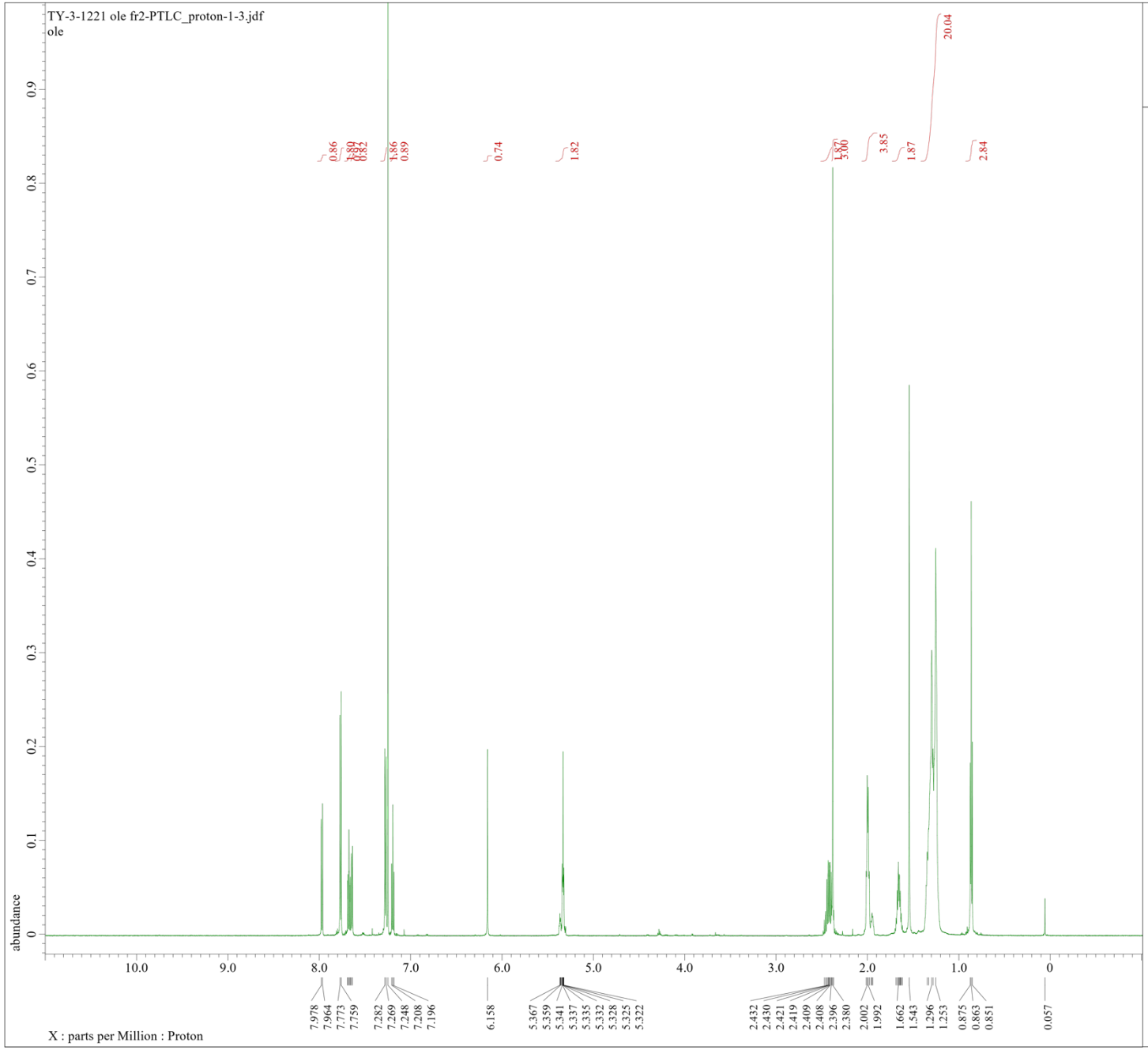


JEOL

Filename	= TY-3-0104 Leu_carb
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-0104 Leu
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 4-JAN-2022 23:19:
Revision_Time	= 4-JAN-2022 23:25:
Comment	= Leuphth
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 128
Total_Scans	= 128
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 20.6[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]

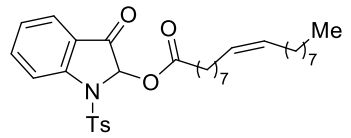


3aj

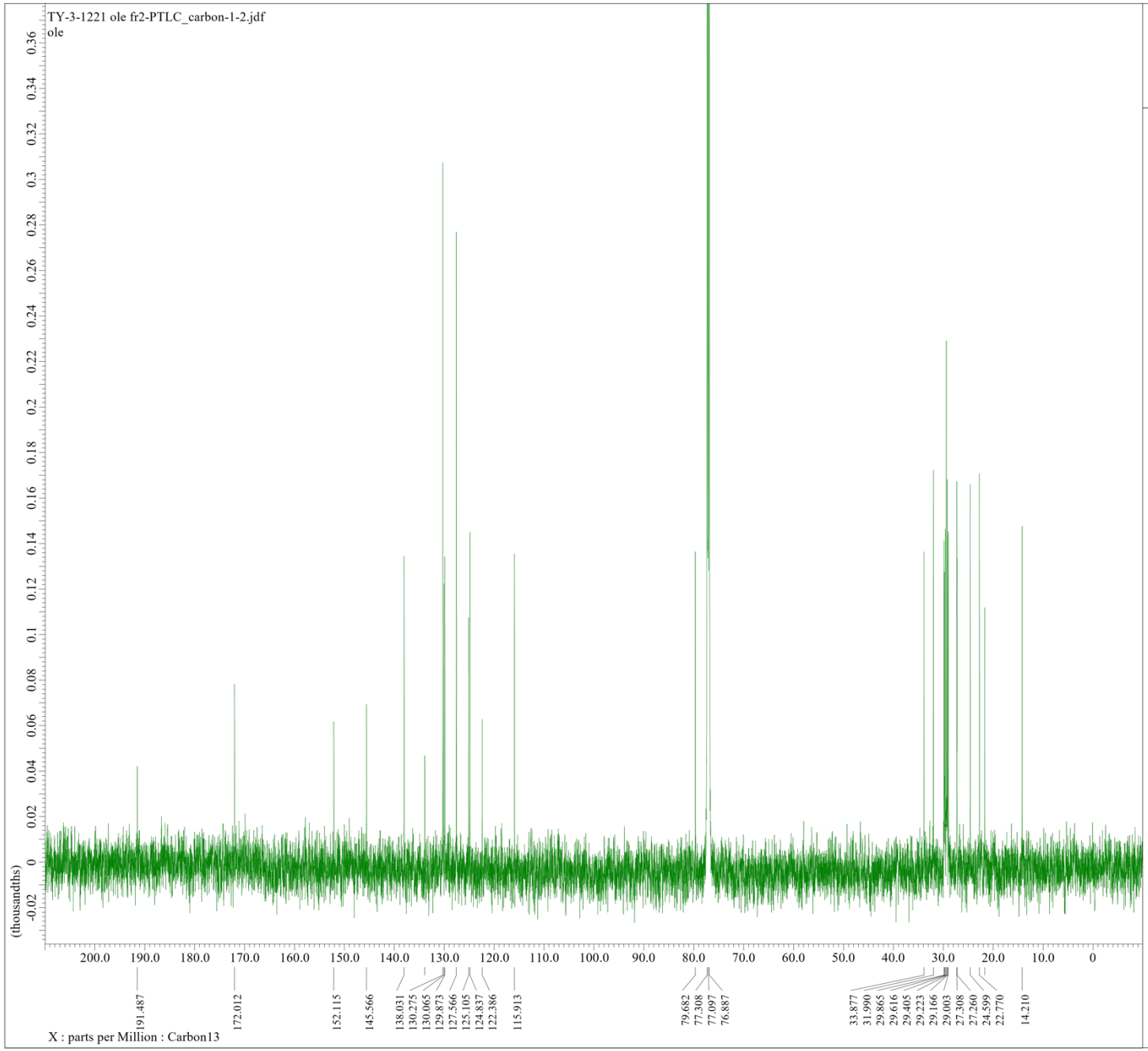


JEOL

```
Filename              = TY-3-1221 ole fr2-PTL
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-3-1221 ole fr2-PTL
Solvent               = CHLOROFORM-D
Actual_Start_Time     = 21-DEC-2021 20:53:37
Revision_Time         = 22-DEC-2021 08:41:25

Comment               = ole
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8

Relaxation_Delay      = 1[s]
Recvr_Gain            = 56
Temp_Get              = 21[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```

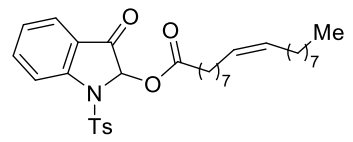


3ak

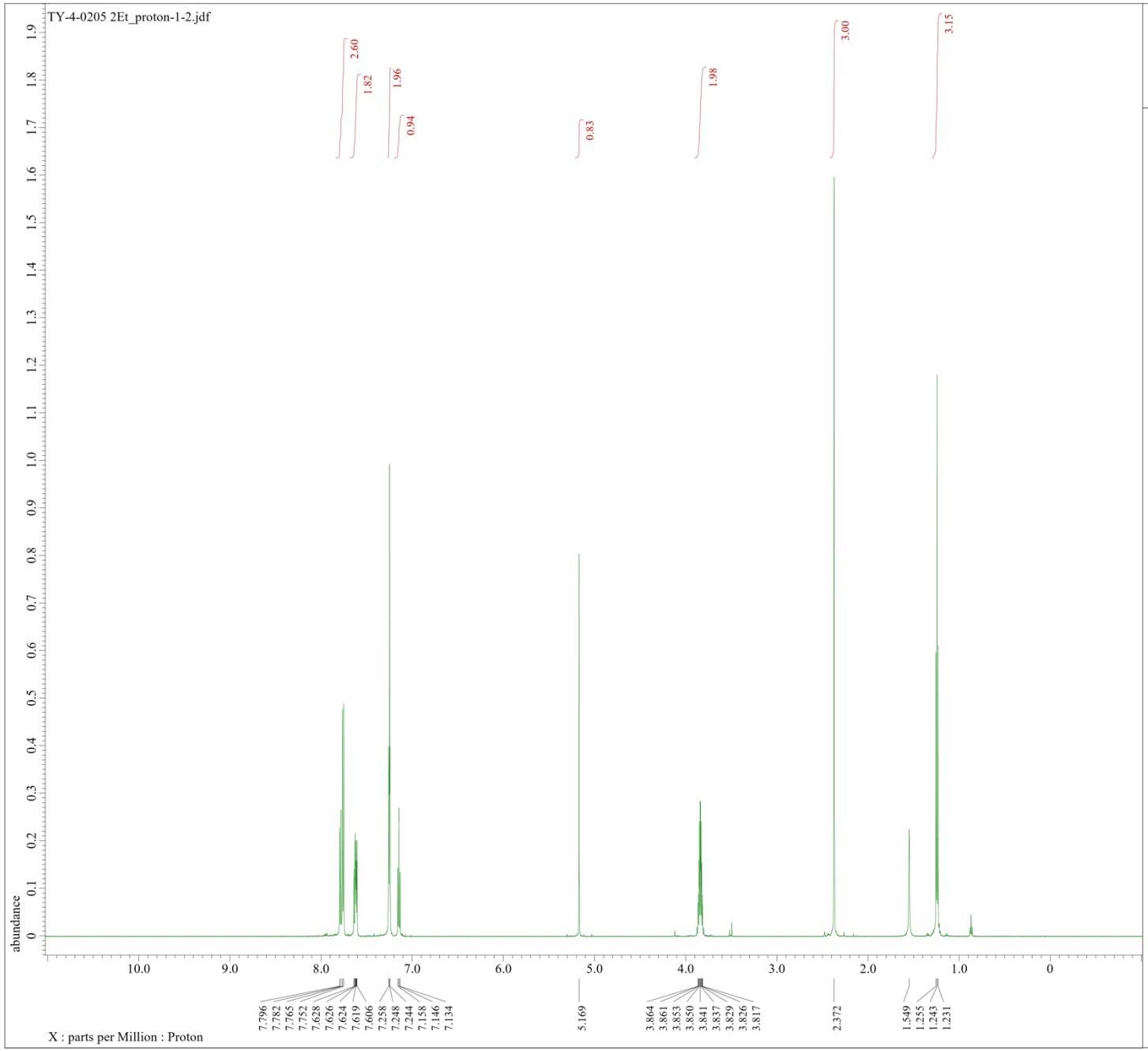


JEOL

Filename	= TY-3-1221 ole fr2-
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1221 ole fr2-
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 21-DEC-2021 20:55:
Revision_Time	= 22-DEC-2021 08:42:
Comment	= ole
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 4096
Total_Scans	= 4096
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 21.2[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]

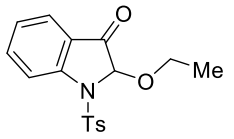


3ak

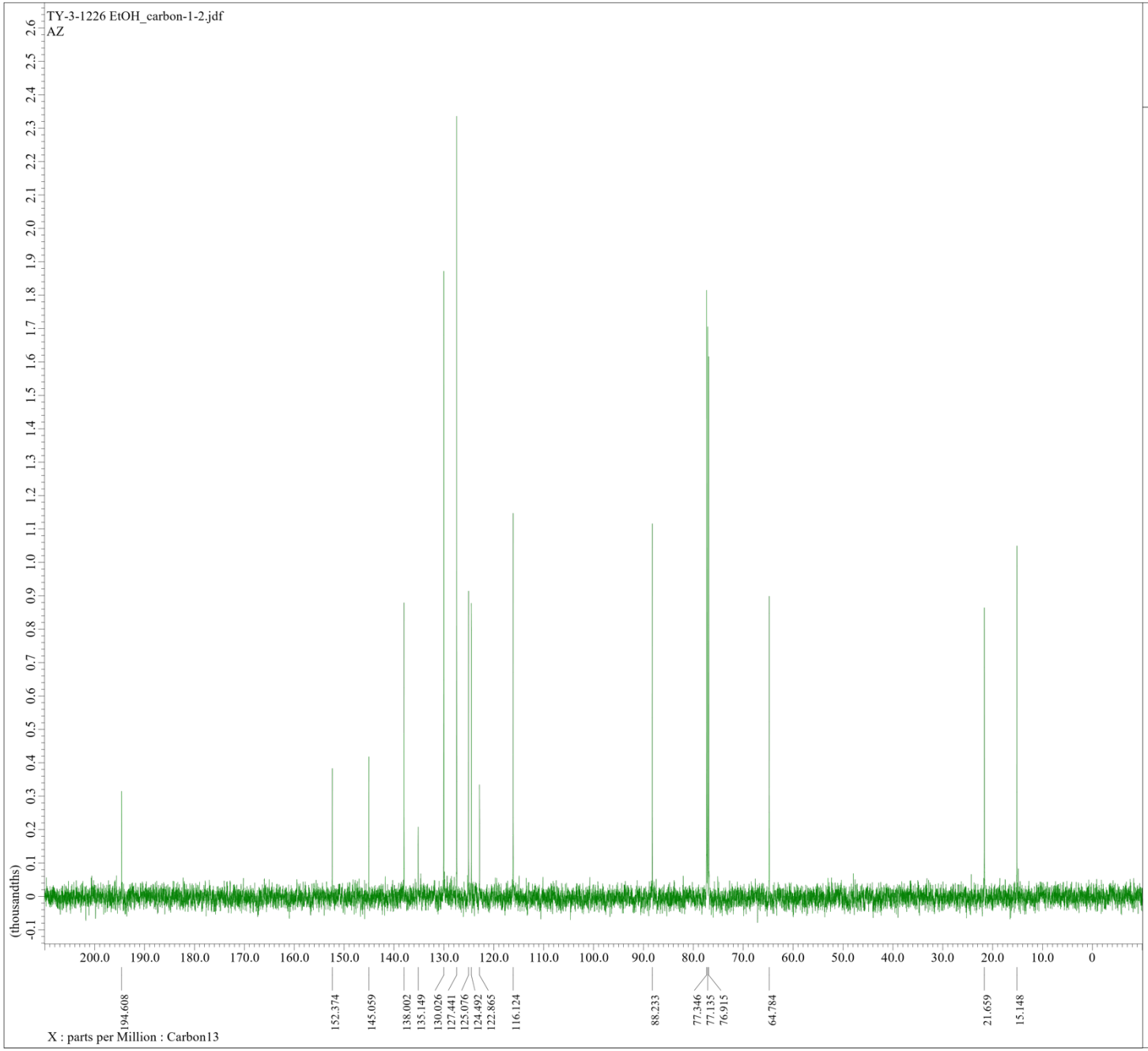


JEOL

```
Filename              = TY-4-0205 2Et_proton-
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-4-0205 2Et
Solvent               = CHLOROFORM-D
Actual_Start_Time     =  5-FEB-2022 17:55:29
Revision_Time         =  5-FEB-2022 18:11:46

Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8

Relaxation_Delay      = 1[s]
Recvr_Gain            = 56
Temp_Get              = 20.6[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```

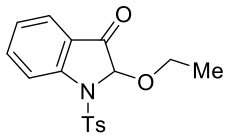


4b

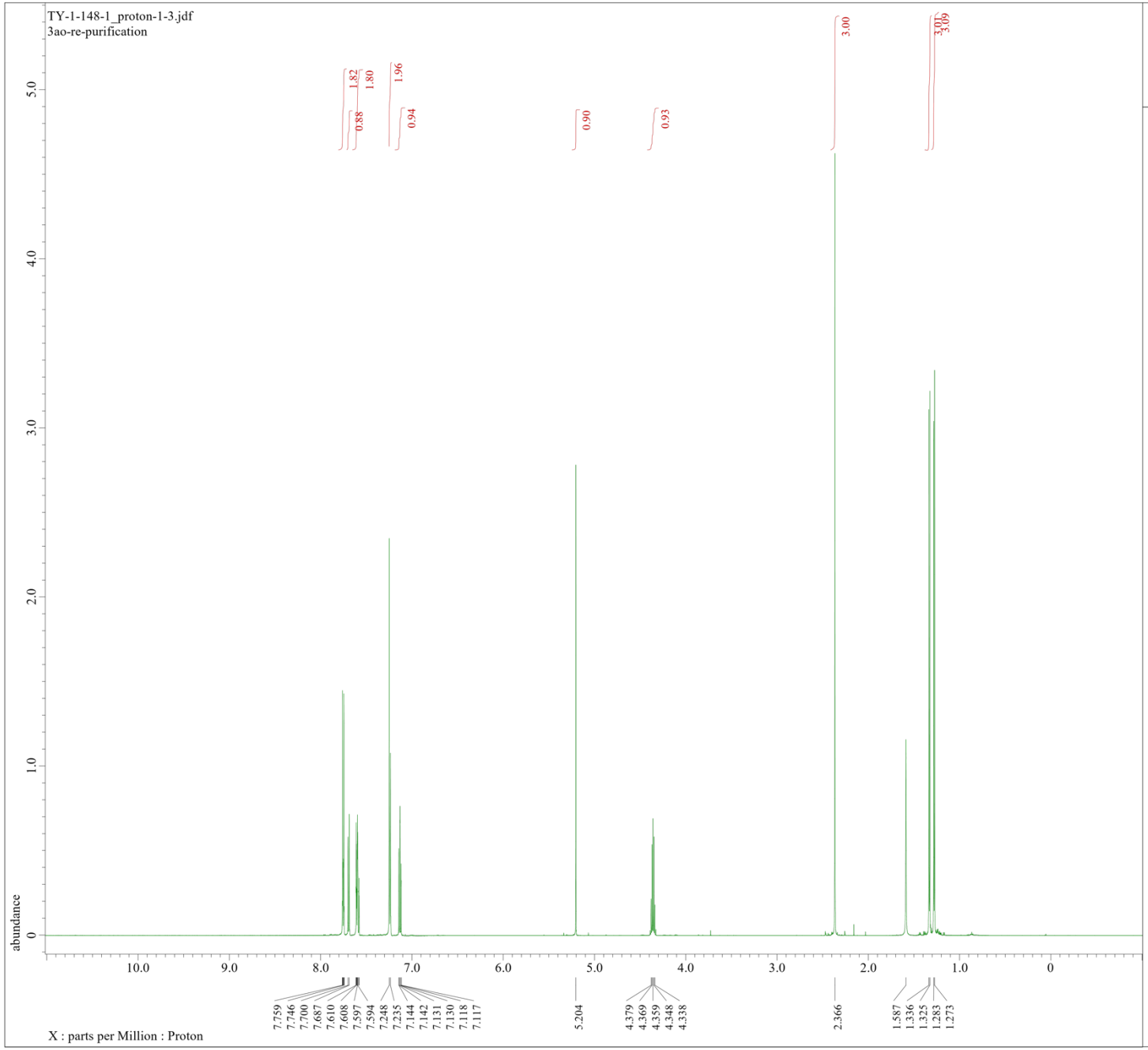


JEOL

Filename	= TY-3-1226 EtOH_car
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1226 EtOH
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 26-DEC-2021 17:24:
Revision_Time	= 26-DEC-2021 17:48:
Comment	= AZ
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 48
Total_Scans	= 48
Relaxation_Delay	= 1[s]
Recvr_Gain	= 26
Temp_Get	= 21.2[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]

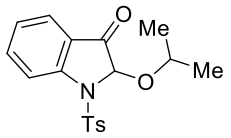


4b

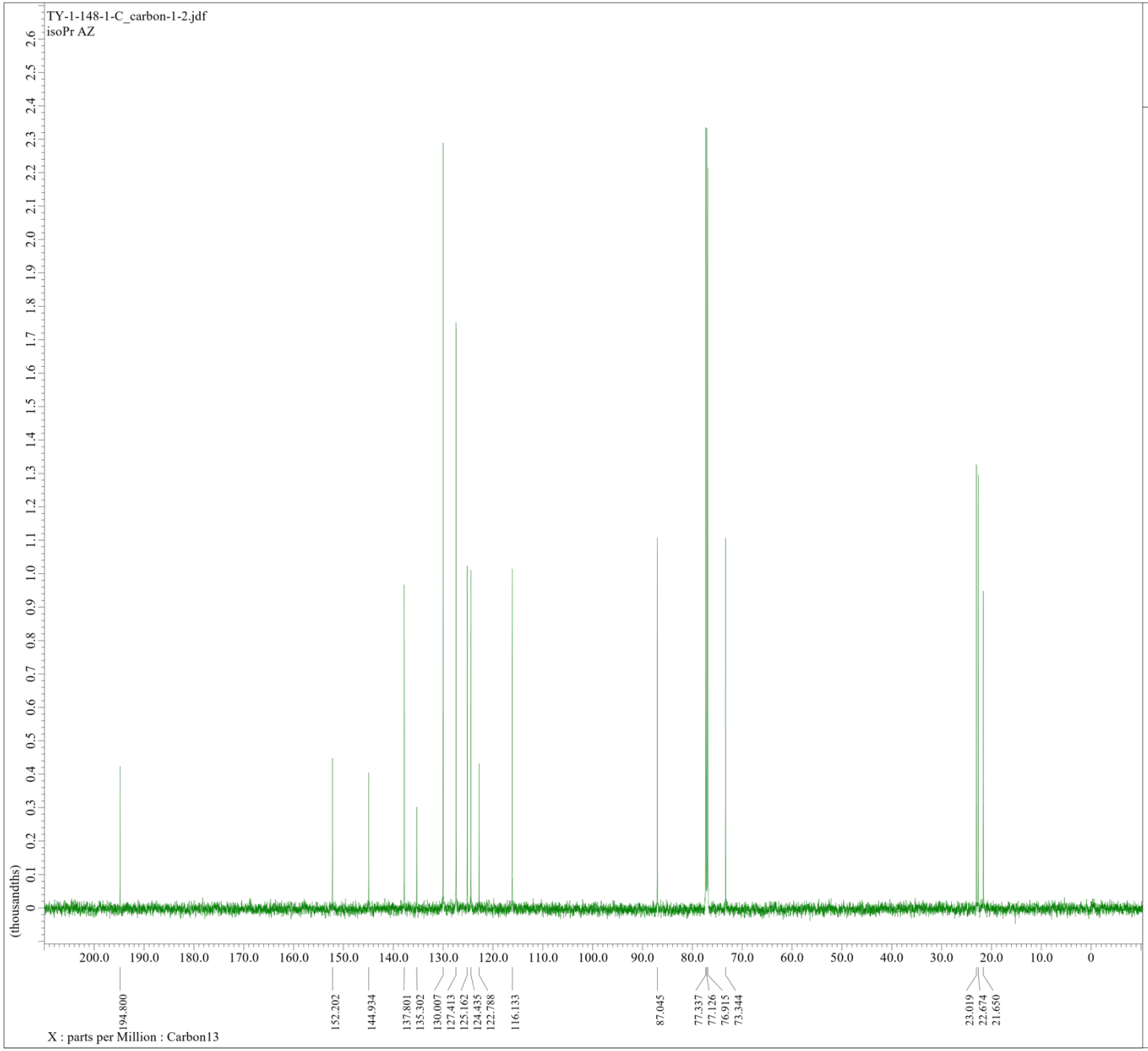


JEOL

```
Filename              = TY-1-148-1_proton-1-3
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-1-148-1
Solvent               = CHLOROFORM-D
Actual_Start_Time     = 16-JUN-2021 11:53:27
Revision_Time         =  3-DEC-2021 22:01:34

Comment               = 3ao-re-purification
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8

Relaxation_Delay      = 1[s]
Recvr_Gain            = 56
Temp_Get              = 20.6[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```

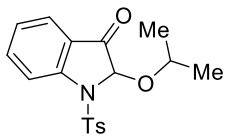


4c

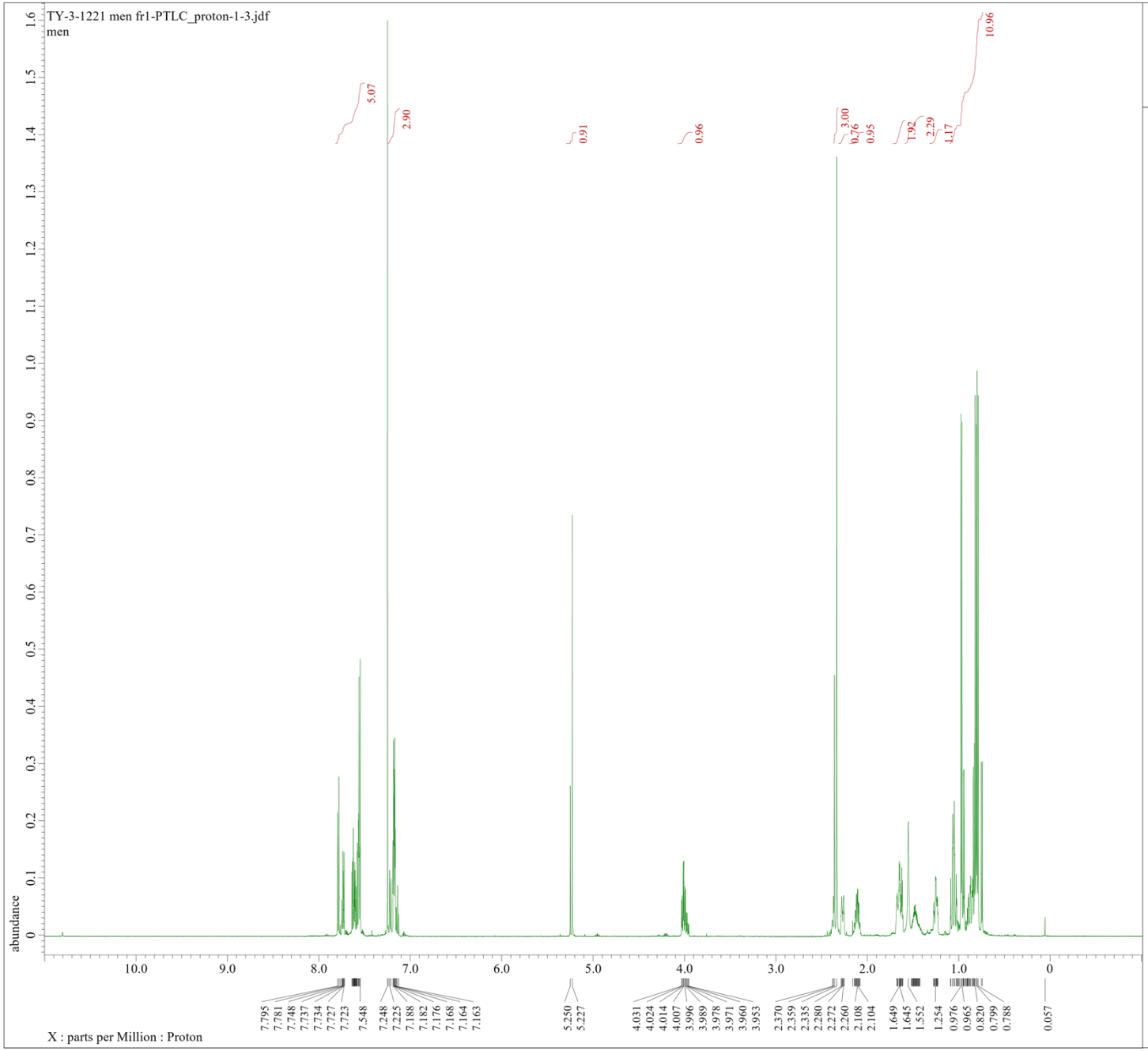


JEOL

Filename	= TY-1-148-1-C_carbo
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-1-148-1-C
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 17-JUN-2021 20:44:
Revision_Time	= 17-JUN-2021 20:50:
Comment	= isoPr AZ
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 201
Total_Scans	= 201
Relaxation_Delay	= 2[s]
Recvr_Gain	= 26
Temp_Get	= 20.9[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 2[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 2[s]
Repetition_Time	= 2.69206016[s]

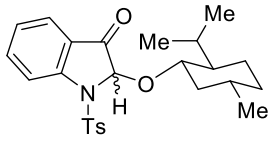


4c

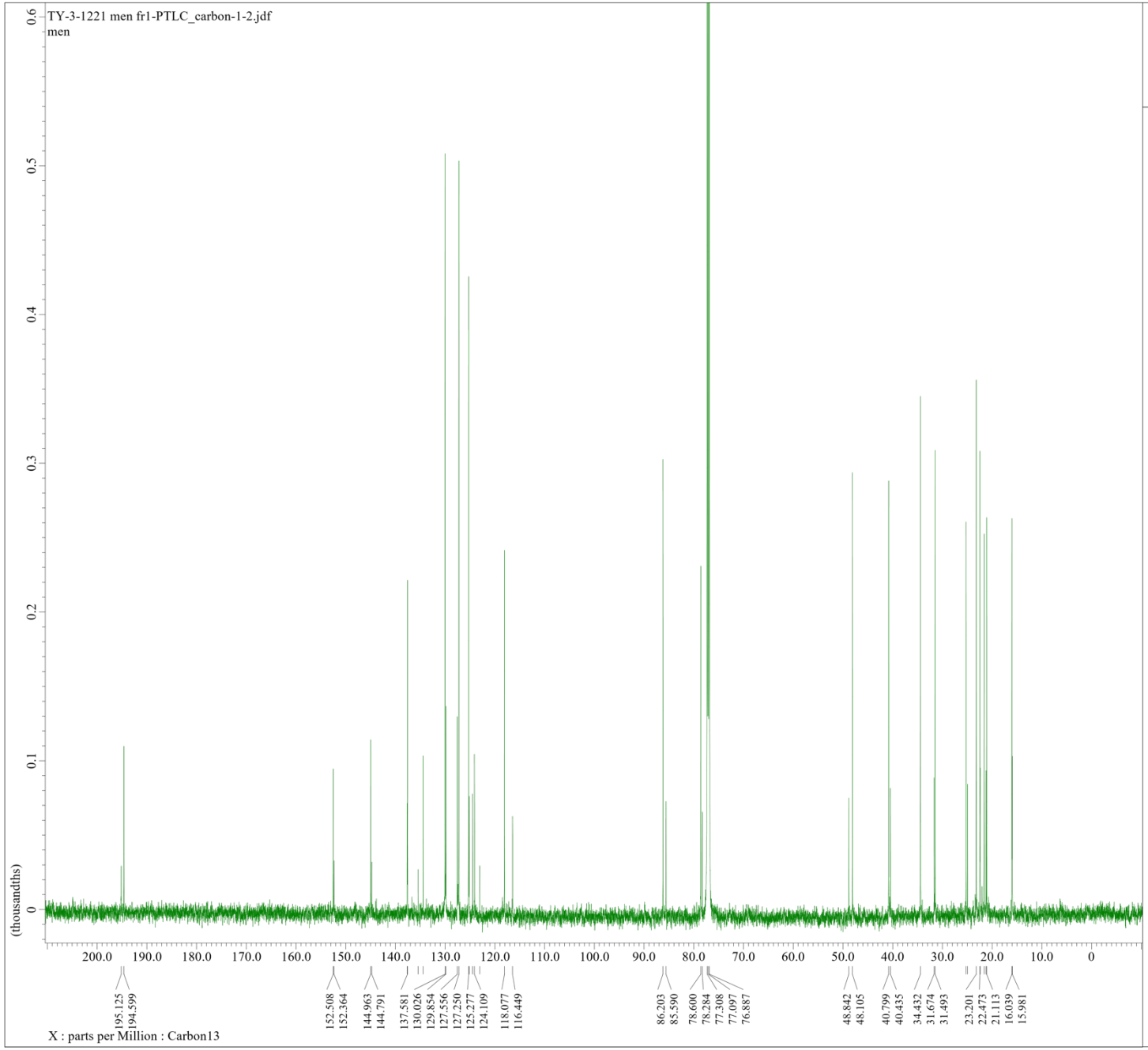


JEOL

```
Filename              = TY-3-1221 men fr1-PTL
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-3-1221 men fr1-PTL
Solvent               = CHLOROFORM-D
Actual_Start_Time     = 21-DEC-2021 23:29:08
Revision_Time         =  3-JAN-2022 21:04:22

Comment               = men
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8

Relaxation_Delay      = 1[s]
Recvr_Gain            = 56
Temp_Get              = 21[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```

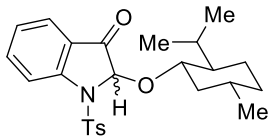


4d

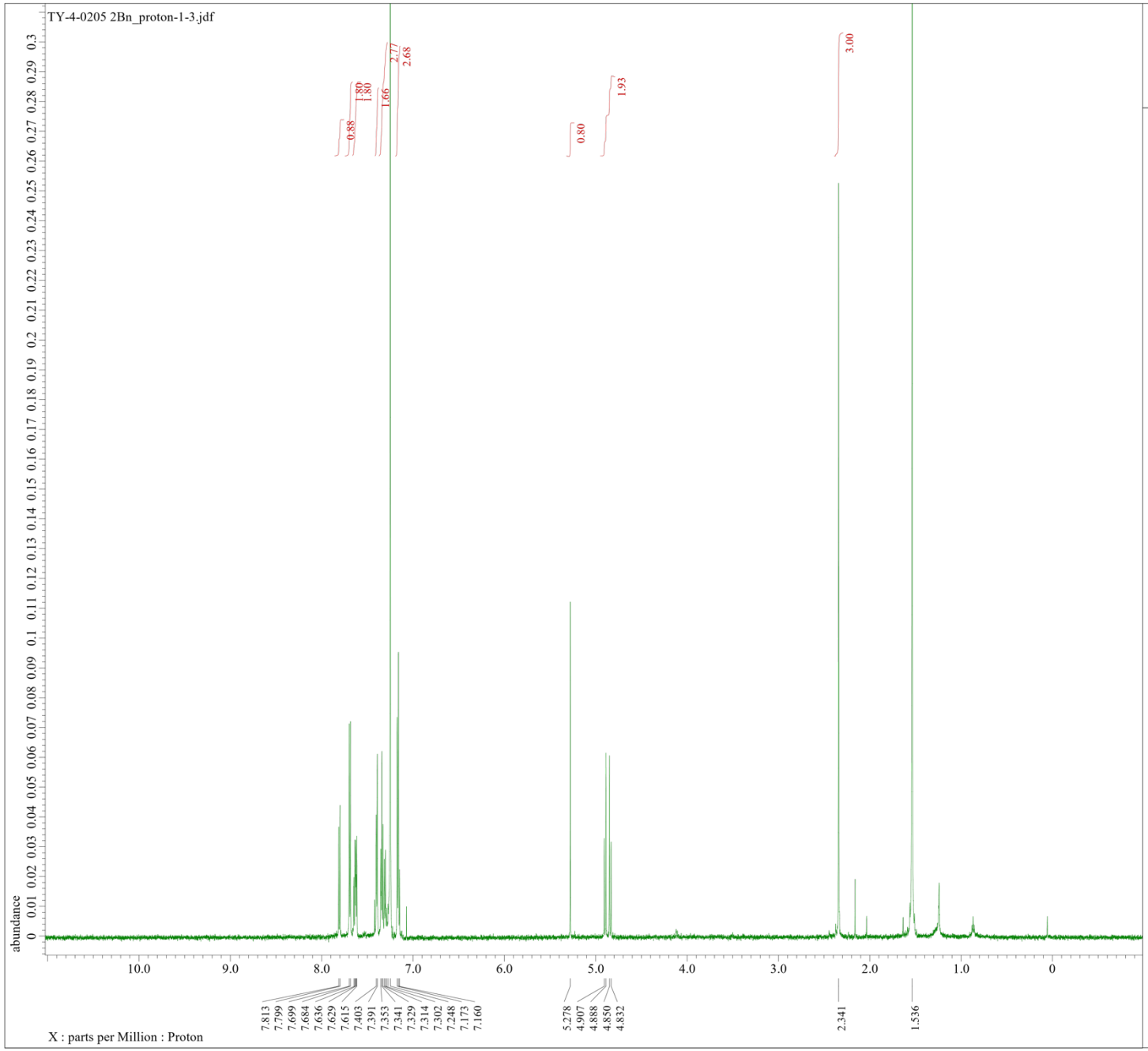


JEOL

Filename	= TY-3-1221 men fr1-
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1221 men fr1-
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 21-DEC-2021 23:31:
Revision_Time	= 22-DEC-2021 08:38:
Comment	= men
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 16114
Total_Scans	= 16114
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 21.2[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]

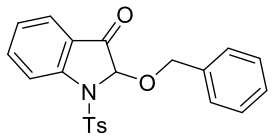


4d

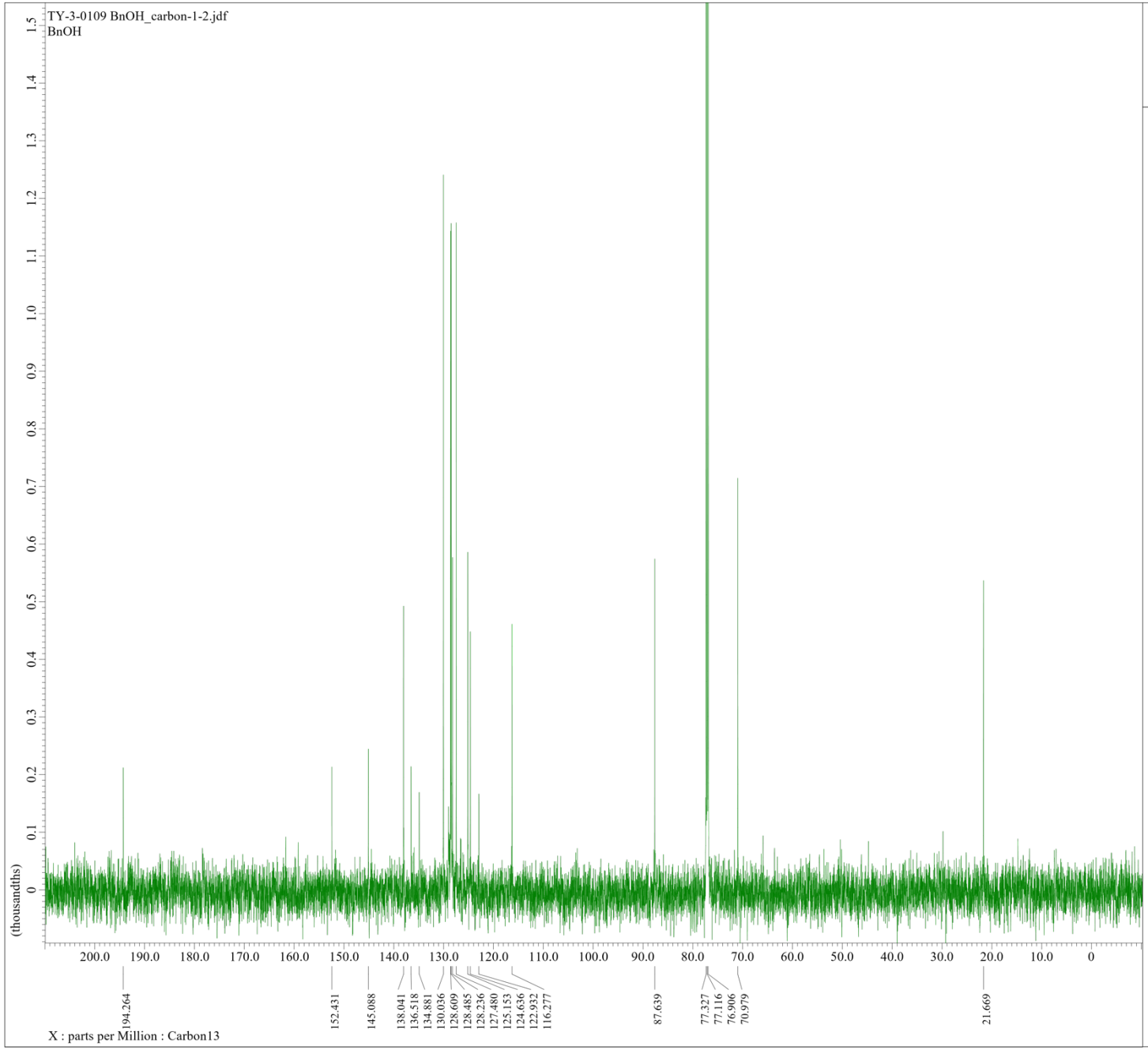


JEOL

```
Filename              = TY-4-0205 2Bn_proton-
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-4-0205 2Bn
Solvent               = CHLOROFORM-D
Actual_Start_Time     =  5-FEB-2022 15:02:26
Revision_Time         =  5-FEB-2022 15:14:36

Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8

Relaxation_Delay      = 1[s]
Recvr_Gain            = 66
Temp_Get              = 20.6[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```

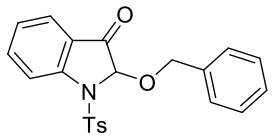


4f

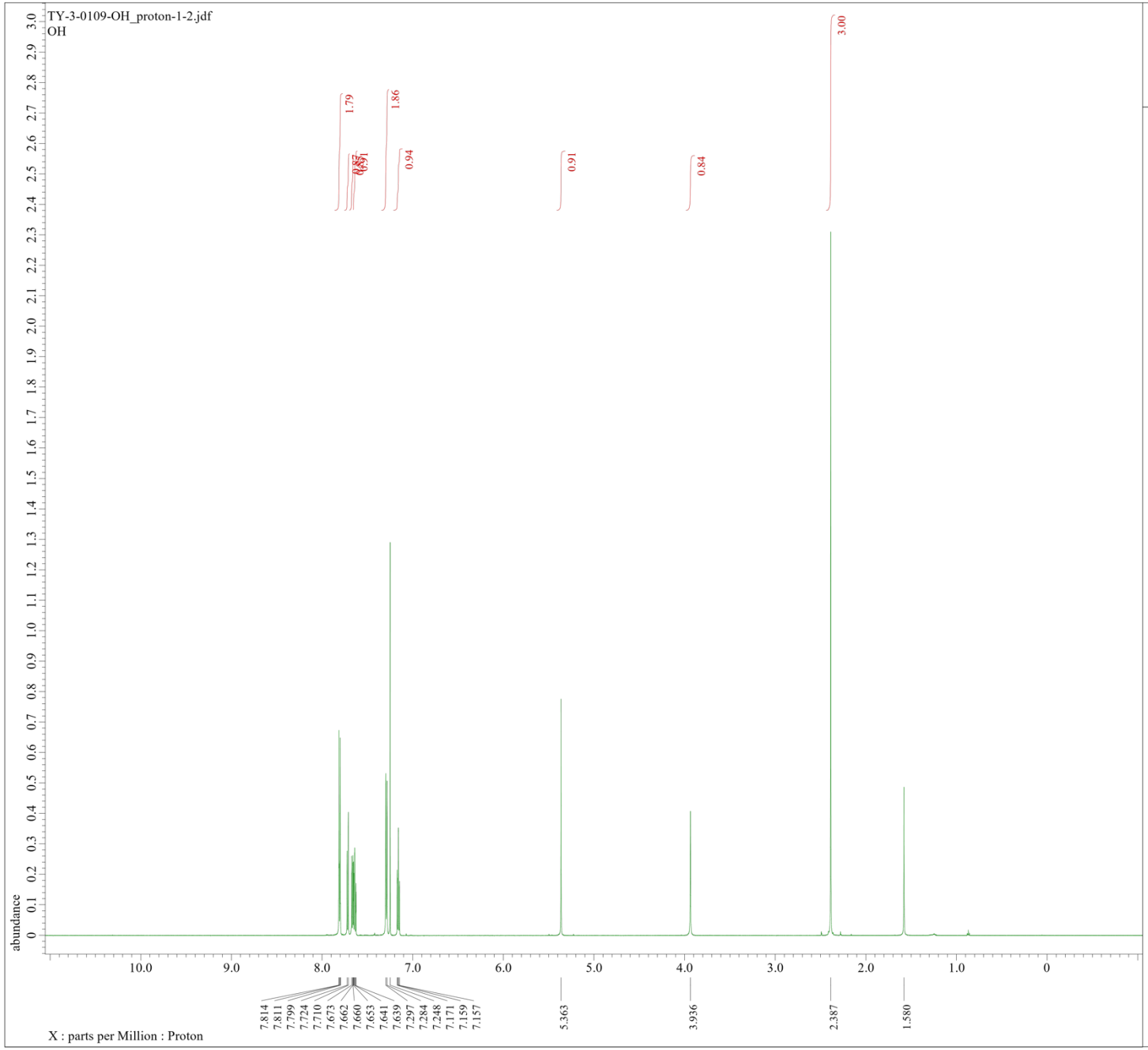


JEOL

Filename	= TY-3-0109 BnOH_car
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-0109 BnOH
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 9-JAN-2022 09:30:
Revision_Time	= 9-JAN-2022 09:42:
Comment	= BnOH
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 257
Total_Scans	= 257
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 18.2[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]

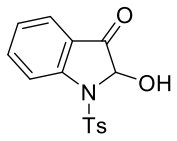


4f

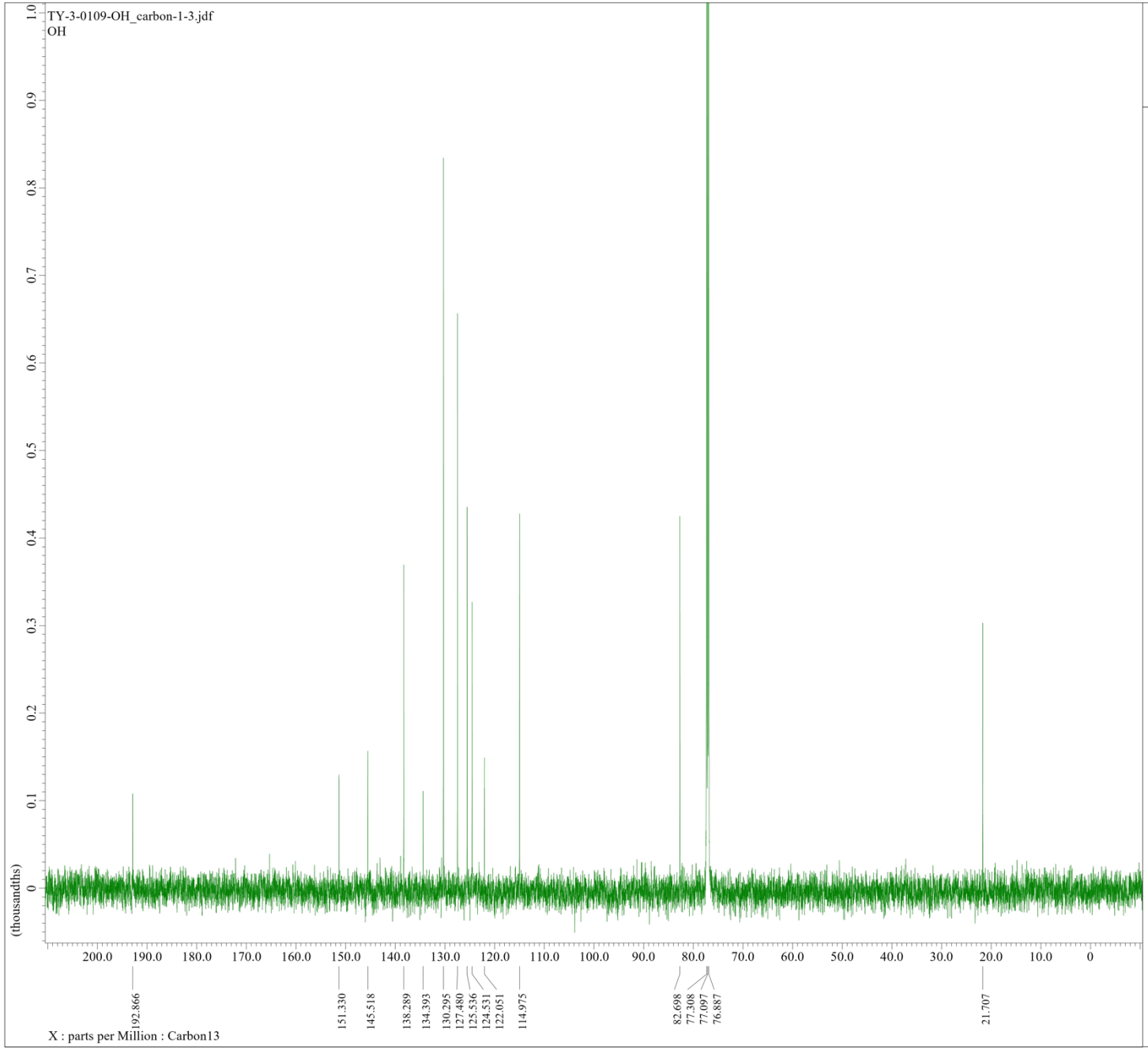


JEOL

```
Filename              = TY-3-0109-OH_proton-1
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-3-0109-OH
Solvent               = CHLOROFORM-D
Actual_Start_Time     =  9-JAN-2022 20:40:27
Revision_Time         =  9-JAN-2022 21:31:24

Comment               = OH
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8

Relaxation_Delay      = 1[s]
Recvr_Gain            = 56
Temp_Get              = 20.6[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```

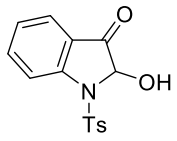


4g

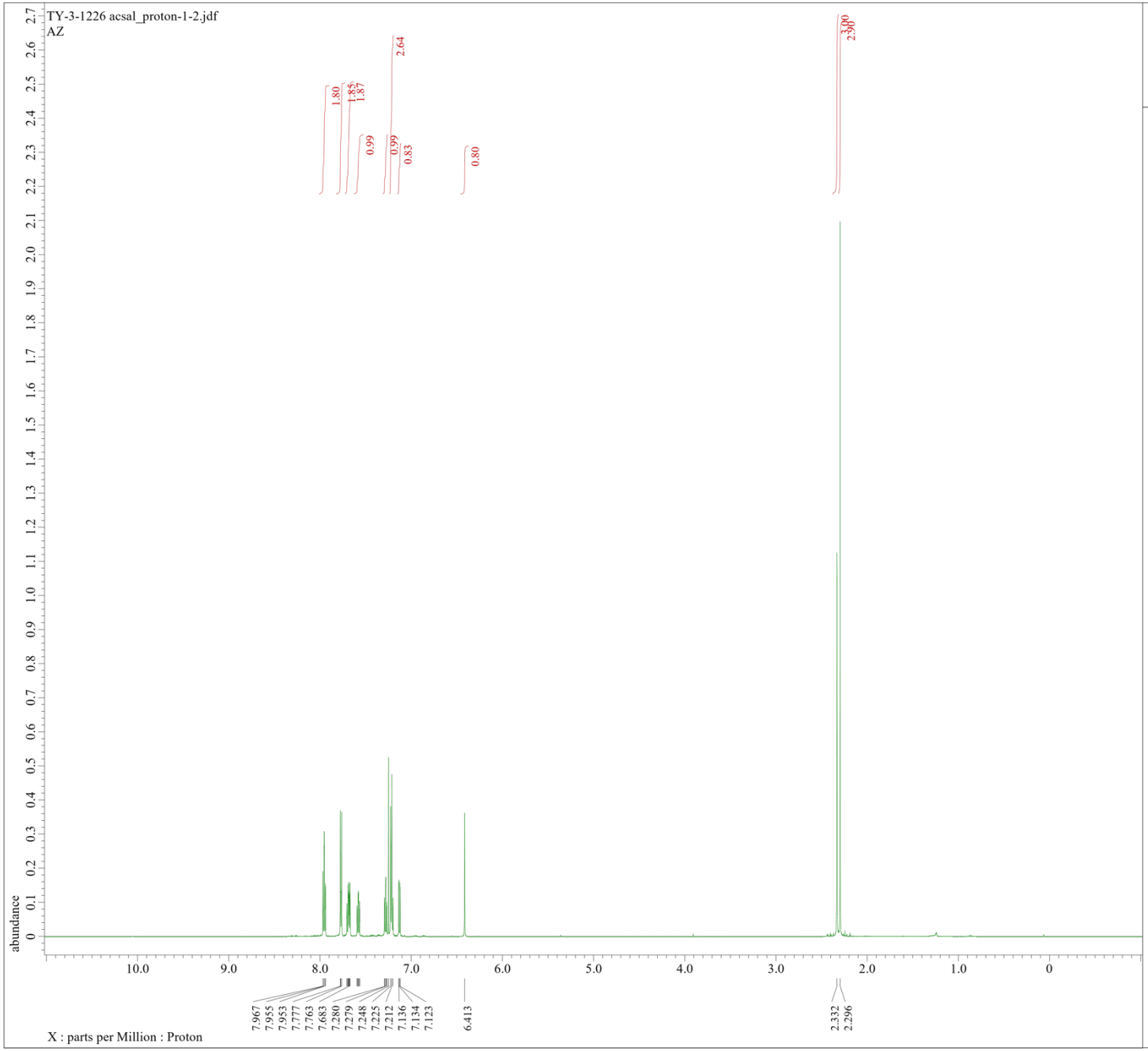


JEOL

Filename	= TY-3-0109-OH_carbo
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-0109-OH
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 9-JAN-2022 20:43:
Revision_Time	= 9-JAN-2022 21:33:
Comment	= OH
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= TRUE
Scans	= 1296
Total_Scans	= 1296
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 20.9[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]

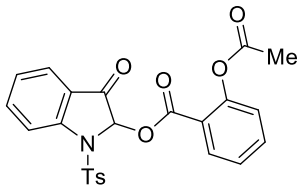


4g

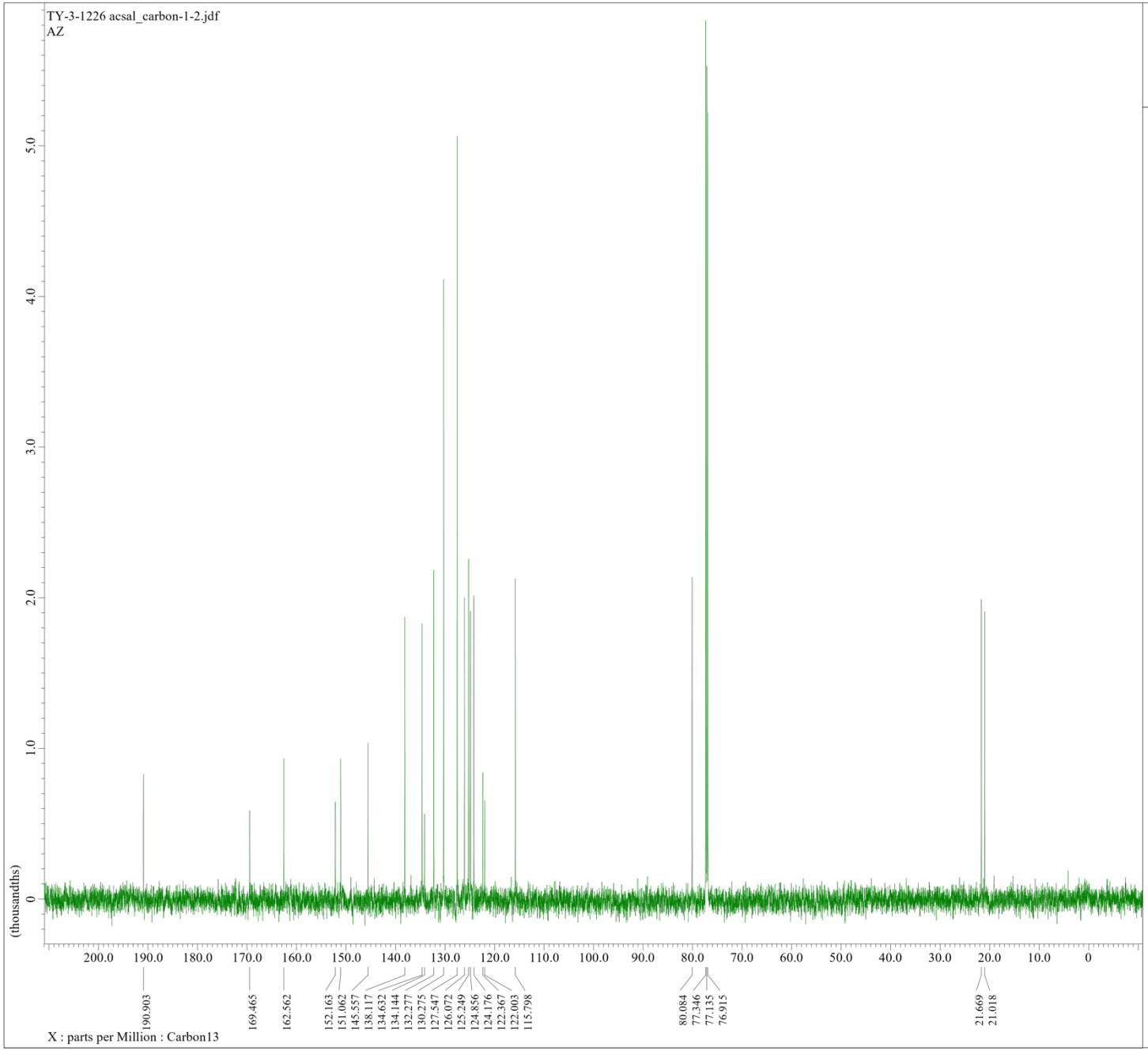


JEOL

```
Filename              = TY-3-1226 acsal_proto
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-3-1226 acsal
Solvent               = CHLOROFORM-D
Actual_Start_Time     = 26-DEC-2021 17:14:10
Revision_Time         = 26-DEC-2021 17:44:55

Comment               = AZ
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8

Relaxation_Delay      = 1[s]
Recvr_Gain            = 36
Temp_Get              = 21.2[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```

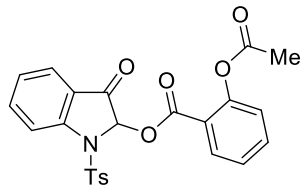


3al

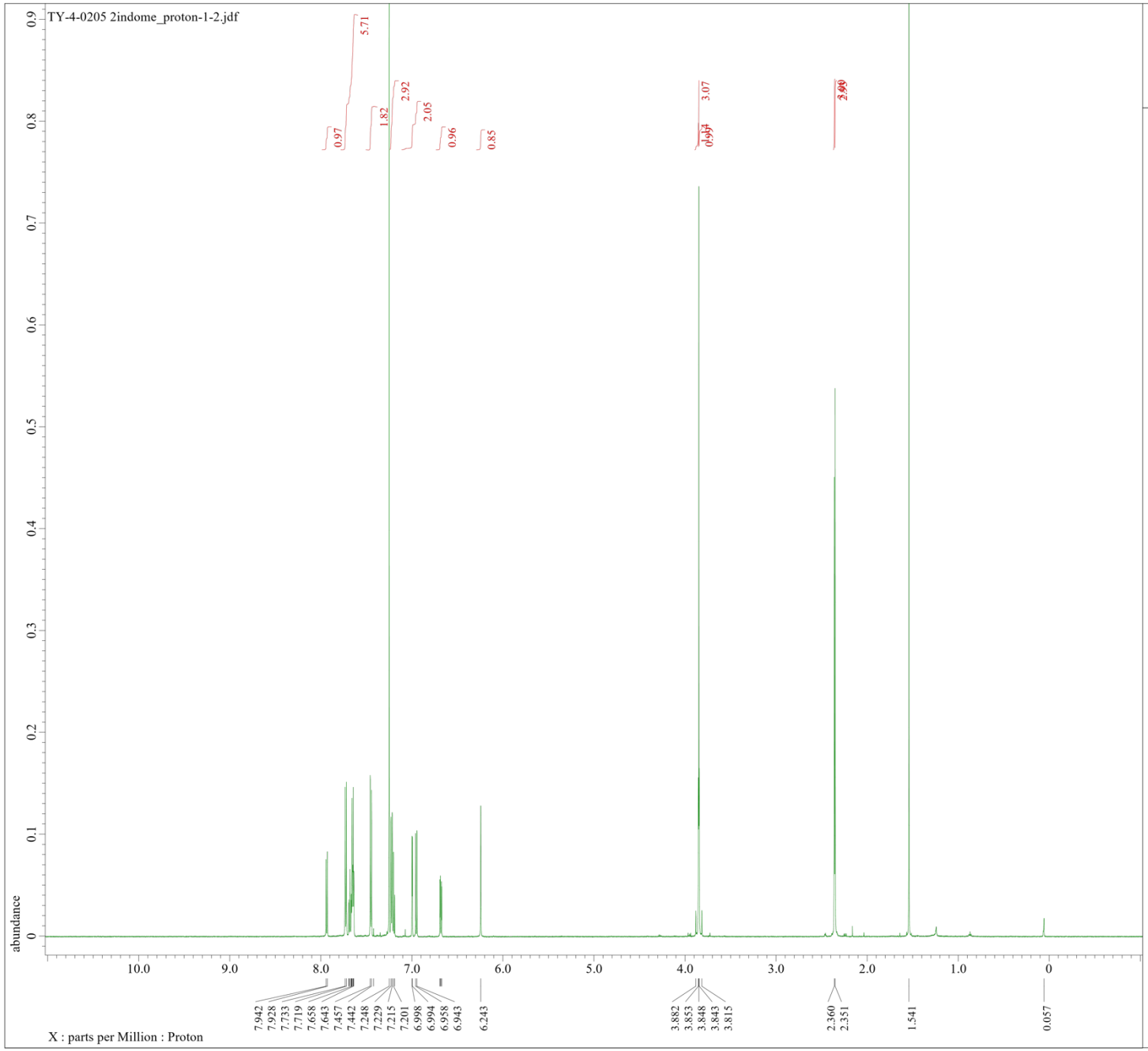


JEOL

Filename	= TY-3-1226 acsal_ca
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-1226 acsal
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 26-DEC-2021 17:16:
Revision_Time	= 26-DEC-2021 17:42:
Comment	= AZ
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 81
Total_Scans	= 81
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 21.2[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]

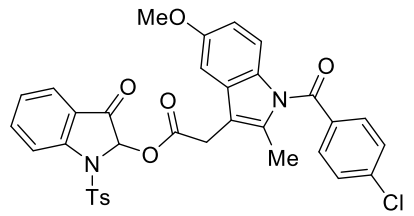


3al

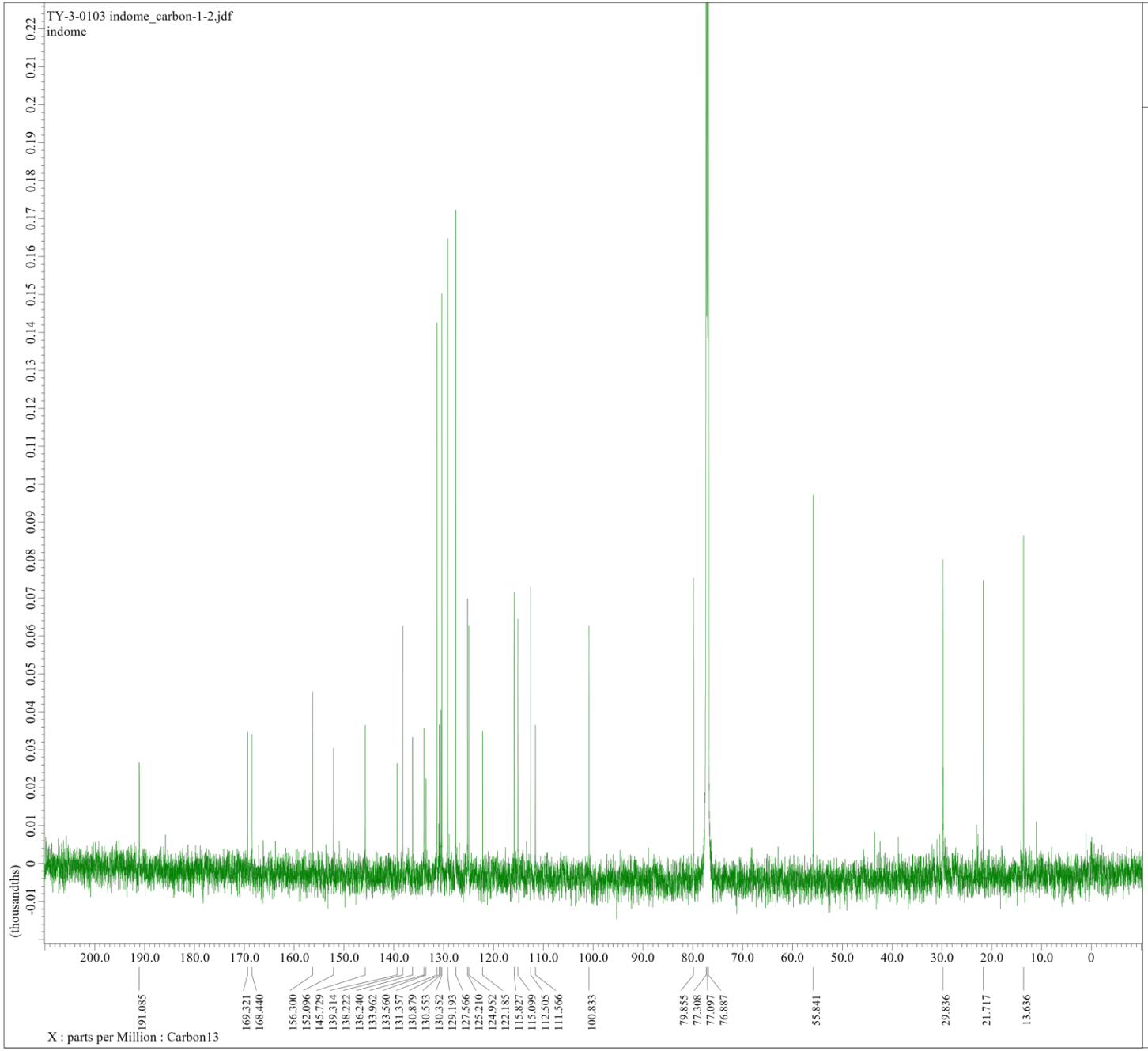


JEOL

```
Filename                = TY-4-0205 2indome_pro
Author                  = delta
Experiment              = proton.jxp
Sample_Id               = TY-4-0205 2indome
Solvent                 = CHLOROFORM-D
Actual_Start_Time       =  5-FEB-2022 17:58:51
Revision_Time           =  5-FEB-2022 18:14:03

Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Proton
Dim_Title               = Proton
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ600R/S3

Field_Strength          = 14.09636928[T] (600[M
X_Acq_Duration          = 2.18103808[s]
X_Domain                = Proton
X_Freq                  = 600.1723046[MHz]
X_Offset                = 5[ppm]
X_Points                = 32768
X_Prescans              = 1
X_Resolution            = 0.45849727[Hz]
X_Sweep                 = 15.02403846[kHz]
X_Sweep_Clipped         = 12.01923077[kHz]
Irr_Domain              = Proton
Irr_Freq                = 600.1723046[MHz]
Irr_Offset              = 5[ppm]
Tri_Domain              = Proton
Tri_Freq                = 600.1723046[MHz]
Tri_Offset              = 5[ppm]
Blanking                = 2[us]
Clipped                 = FALSE
Scans                   = 8
Total_Scans             = 8

Relaxation_Delay        = 1[s]
Recvr_Gain              = 56
Temp_Get                = 20.6[dC]
X_90_Width              = 7.7[us]
X_Acq_Time              = 2.18103808[s]
X_Angle                 = 45[deg]
X_Atn                   = 8.1[dB]
X_Pulse                 = 3.85[us]
Irr_Mode                = Off
Tri_Mode                = Off
Dante_Loop              = 100
Dante_Presat            = FALSE
Decimation_Rate         = 0
Experiment_Path         = c:\Program Files\JEOL
Initial_Wait            = 1[s]
Phase                   = {0, 90, 270, 180, 180
Presat_Time             = 1[s]
Presat_Time_Flag        = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 1[s]
Repetition_Time         = 3.18103808[s]
```

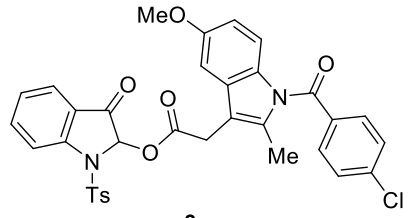


3am

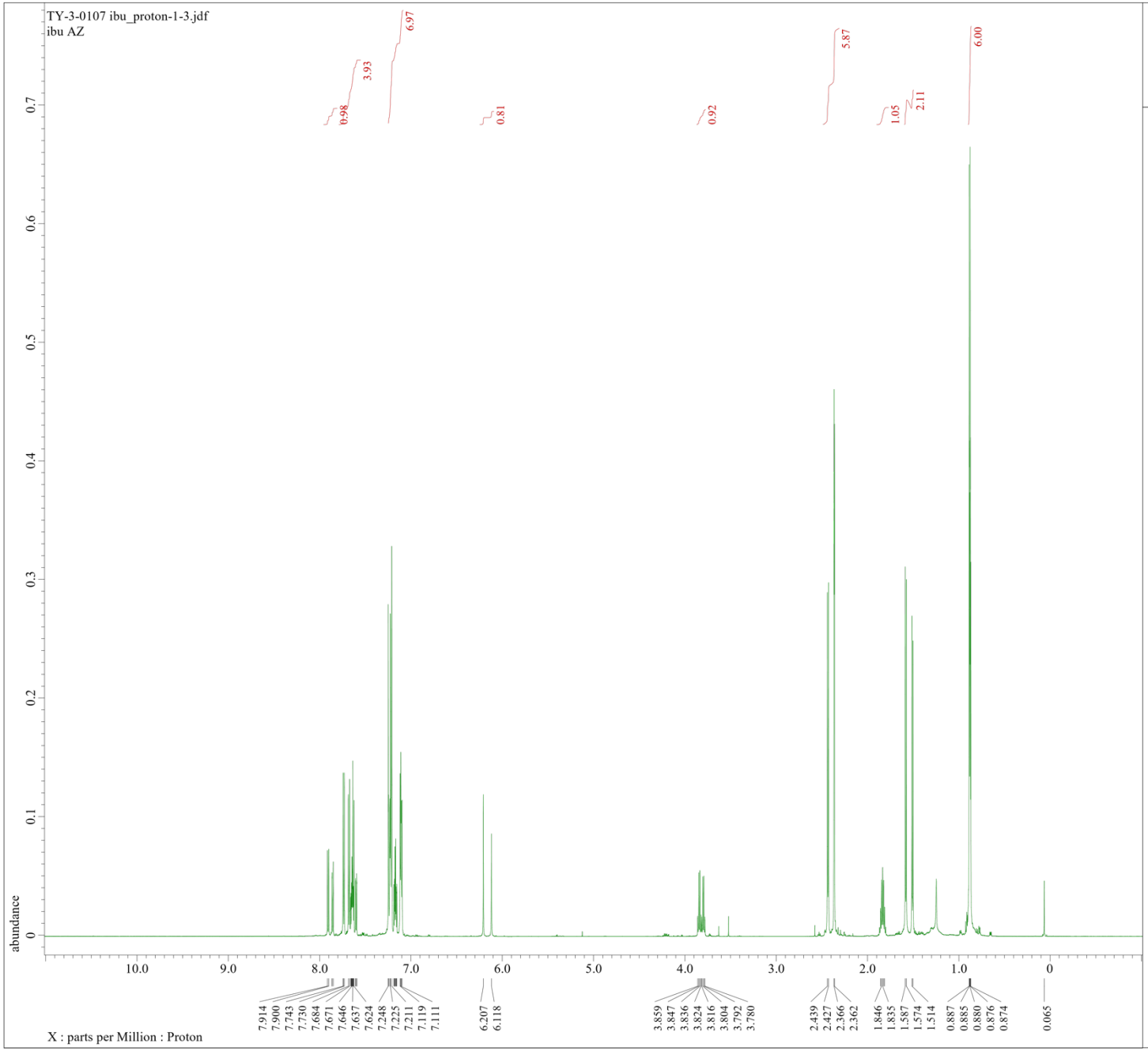


JEOL

Filename	= TY-3-0103 indome_c
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-0103 indome
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 3-JAN-2022 21:17:
Revision_Time	= 4-JAN-2022 09:09:
Comment	= indome
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= TRUE
Scans	= 21183
Total_Scans	= 21183
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 20.1[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]

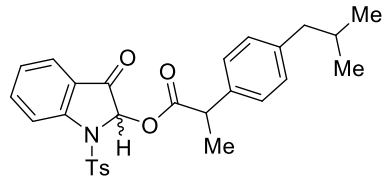


3am

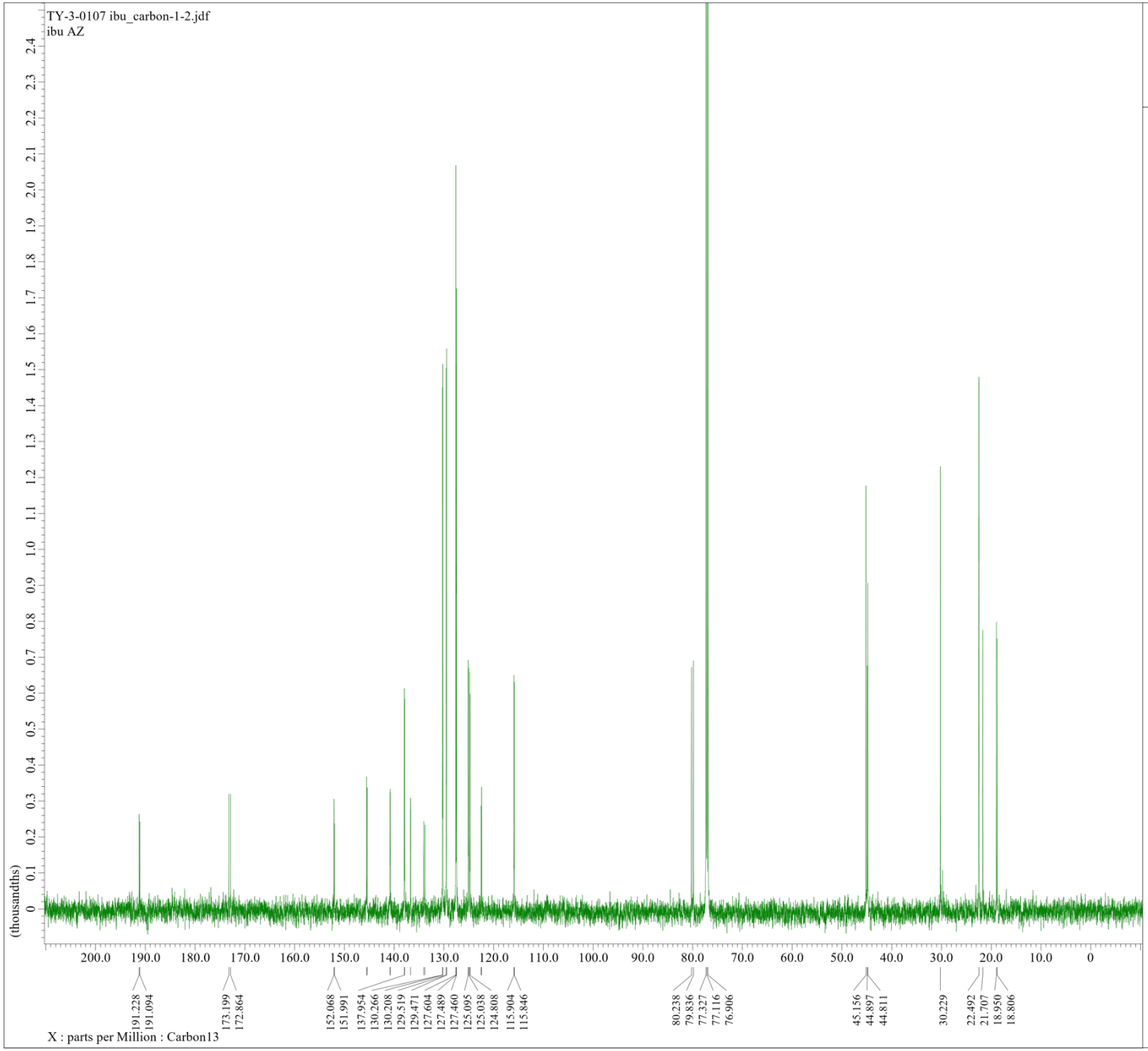


JEOL

```
Filename              = TY-3-0107 ibu_proton-
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-3-0107 ibu
Solvent               = CHLOROFORM-D
Actual_Start_Time     =  7-JAN-2022 13:41:03
Revision_Time         =  7-JAN-2022 14:18:01

Comment               = ibu AZ
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 8
Total_Scans           = 8

Relaxation_Delay      = 1[s]
Recvr_Gain            = 36
Temp_Get              = 20.8[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```

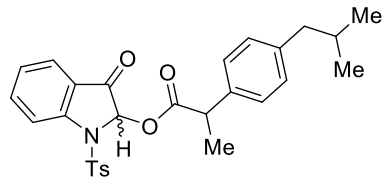


3an



JEOL

Filename	= TY-3-0107 ibu_carb
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-3-0107 ibu
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 7-JAN-2022 13:43:
Revision_Time	= 7-JAN-2022 14:19:
Comment	= ibu AZ
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 2[us]
Clipped	= FALSE
Scans	= 501
Total_Scans	= 501
Relaxation_Delay	= 1[s]
Recvr_Gain	= 36
Temp_Get	= 20.9[dC]
X_90_Width	= 11.8[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 9.6[dB]
X_Pulse	= 3.93333333[us]
Irr_Atn_Dec	= 27.986[dB]
Irr_Atn_Dec_Calc	= 27.986[dB]
Irr_Atn_Dec_Default_Calc	= 27.986[dB]
Irr_Atn_Noe	= 27.986[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Temp1	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]



3an