Synthesis of Glycosyl Formamides Through *N*-Glycosylation of Unprotected Carbohydrates

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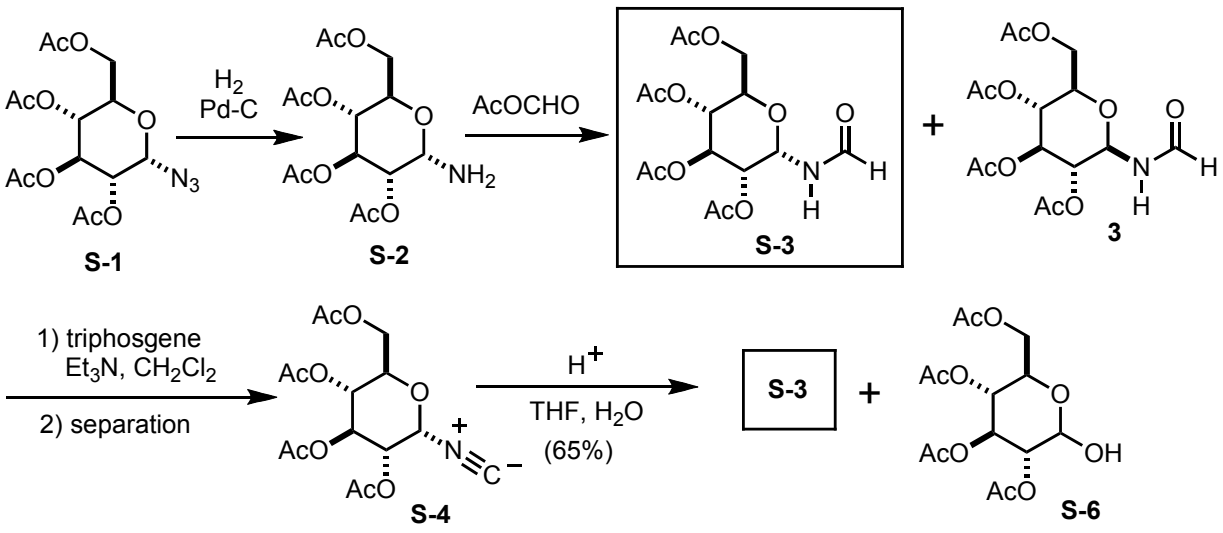
I. Synthesis, experimental procedures, characterization data, ^{1}H and ^{13}C NMR spectra for α-glucosyl and α-xylosyl formamides ‥ · · · ‥ ····· ‥ ··· · ‥ ····· ‥ ···· ‥ ····· ···· ··· ‥ ····· ····· ····· 1

II. ^{1}H and ^{13}C NMR spectra for all relevant compounds described in the manuscript ····· · ····· ···· · ‥17

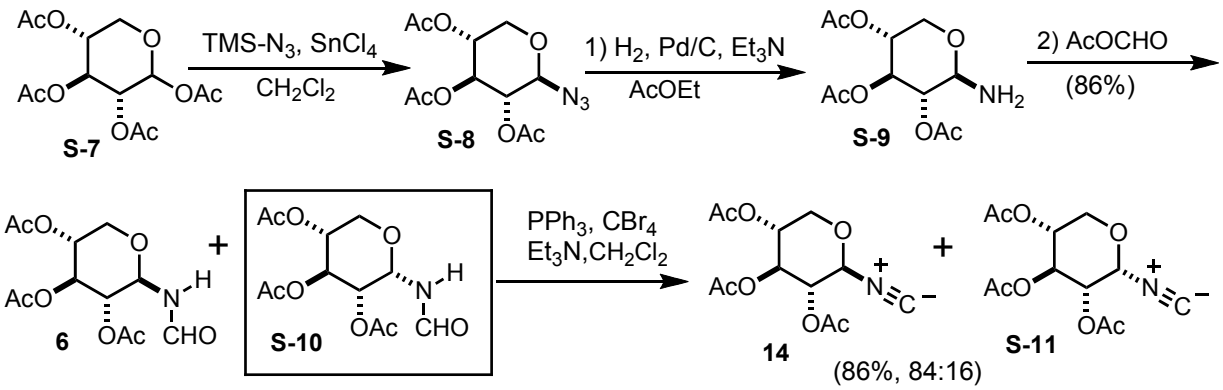
Previously, we reported the synthesis of α-glucosyl isocyanide **S-4** starting with α-glucosyl azide **S-1**[1] (Scheme 1). In this synthetic route, α-azide **S-1** was carefully hydrogenated to give unstable α-glucosylamine **S-2**, which was immediately treated with acetic formic anhydride. Due to the facile isomerization of α-glucosylamine **S-2** to the corresponding β-anomer, this process produced an inseparable mixture of α- and β-glucosyl formamides **S-3** and **3**. Dehydration of a mixture of **S-3** and **3** with triphosgene and triethylamine, produced a mixture of α- and β-glucosyl isocyanides, which was carefully separated to afford pure α-isocyanide **S-4**. In order to obtain pure α-glucosyl formamide **S-3**, we repeated this synthetic route to obtain α-glucosyl isocyanide **S-4**. Hydrolysis of α–isocyanide **S-4** afforded α-glucosyl formamide **S-3** in 65% yield. The moderate yield of this process is a result of the formation of lactol **S-6**. It is worthwhile to mention that ^{1}H NMR analysis of the β- and α- glucosyl formamides measured in $CDCl_3$ at room temperature showed that β-anomer **3** exists as a 5:1 mixture of (*Z*)- and (*E*)-rotamers, and that the α-counterpart **S-3** is a 1:1 mixture of rotamers.



Scheme 1 Synthesis of α-glucosyl formamide **S-3**

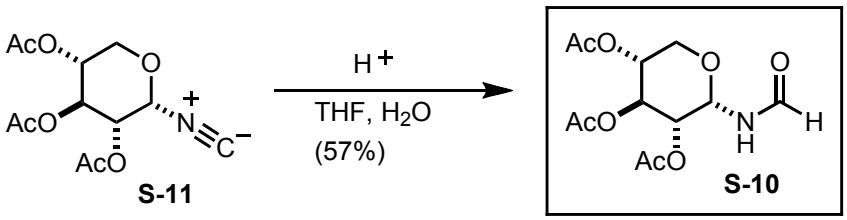
We have already reported the synthesis of β- and α-xylosyl isocyanides **14** and **S-11** starting with tetraacetyl xylopyranosyl acetate **S-7**[2] (Scheme 2). Reduction of β-azide **S-8** produced β-xylosylamine **S-9**, which was immediately formylated using acetic formic

anhydride to afford an inseparable mixture of xylosyl formamides **6** and **S-10**. Treatment of the mixture with triphenylphosphine, carbon tetrabromide and triethylamine afforded a mixture of isocyanides **14** and **S-11**, which were carefully separated by chromatography. In this synthesis, α-xylosyl isocyanide **S-11** was formed as an unexpected minor product. Repeating this synthetic route, we obtained a considerable amount of α-xylosyl isocyanide **S-11**.



Scheme 2 Synthsesis of β- and α-xylosyl isocyanides

Acid-catalyzed hydrolysis of α-xylosyl isocyanide **S-11** using similar reaction conditions as those depieted in Scheme 1 gave α-xylosyl formamide **S-10** in 57% yield (Scheme 3).



Scheme 3 Synthesis of α-xylosyl formamide **S-10**

References

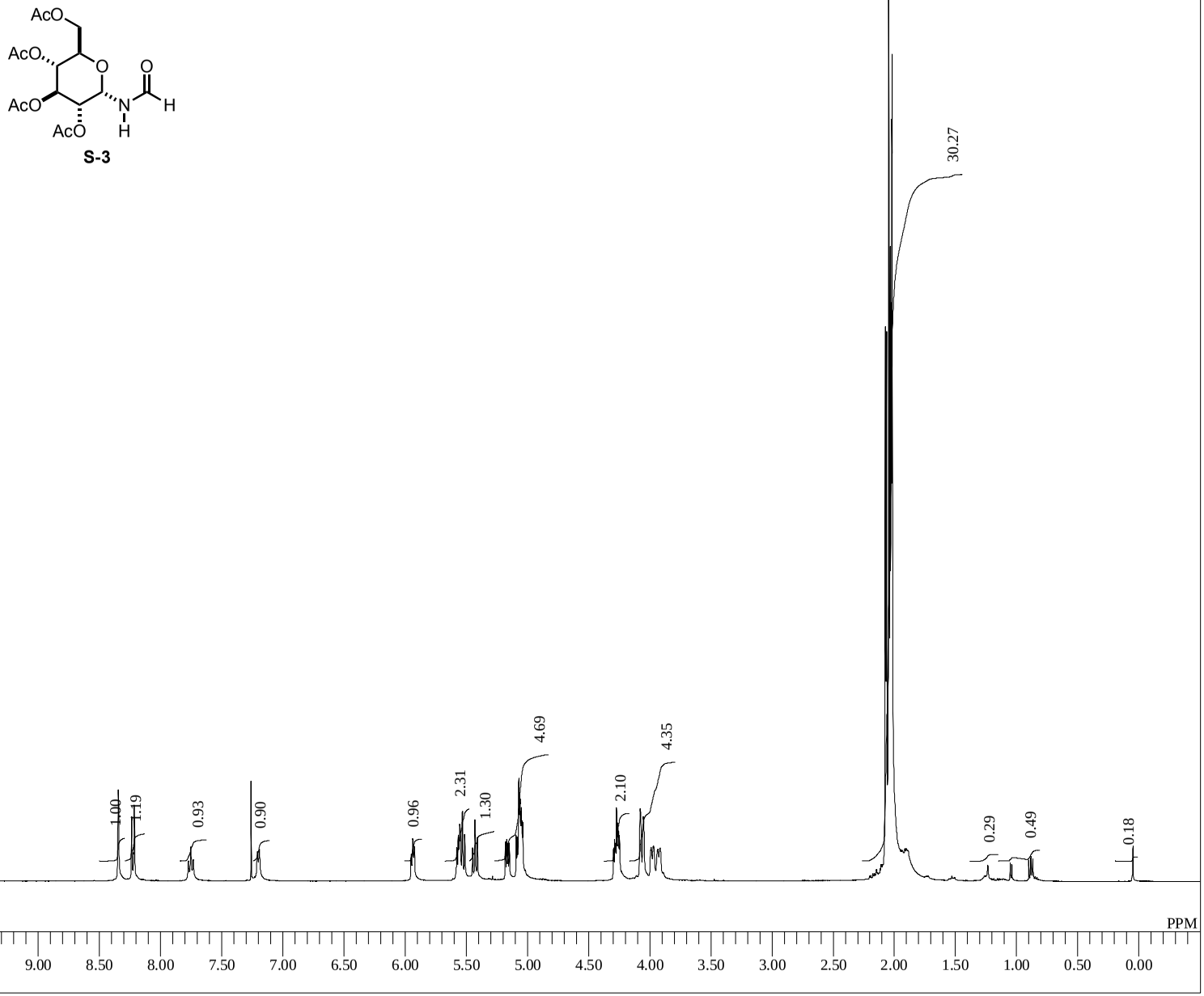
1. Y. Ichikawa, T. Nishiyama and M. Isobe, *J. Org. Chem.*, 2001, **66**, 4200-4205.

2. Y. Ichikawa, H. Watanabe, H. Kotsuki and K. Nakano, *Eur. J. Org. Chem.*, 2010, 6331-6337.

EXPERIMENTAL

α-D-Glucosyl formamide S-3. A solution of **S-4** (100 mg, 0.28 mmol) dissolved in a mixture of aqueous 0.1 N HCl (1.5 mL) and tetrahydrofuran (5.0 mL) was heated at 50 °C for 5.5 h. The reaction mixture was diluted wit ethyl acetate and brine, and the separated aqueous layer was extracted with ethyl acetate (×3). The combined organic extracts were dried (Na_2SO_4) and concentrated to afford the residue (132 mg), which was purified by silica gel chromatography (AcOEt/hexane 3:2) to furnish **S-3** (69 mg, 65%): m. p. 155–156 °C; $[\alpha]_D^{23}$ = +111.1 (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3345, 1757, 1737, 1717, 1699, 1531 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 2.015 (s, 3H), 2.020 (s, 3H), 2.025 (s, 3H), 2.05 (s, 6H), 2.06 (s, 3H), 2.07 (s, 3H), 3.93 (brd, *J* = 8.0 Hz, 1H), 3.98 (brd, *J* = 8.0 Hz, 1H), 4.05–4.09 (2H), 4.24–4.30 (2H), 5.03–5.10 (2H), 5.16 (dd, *J* = 10.0, 5.5 Hz, 1H), 5.43 (t, *J* = 10.0, 1H), 5.53 (t, *J* = 10.0, 1H), 5.56 (dd, *J* = 10.0, 5.5 Hz, 1H), 5.93 (dd, *J* = 7.5, 5.5 Hz, 1H), 7.20 (brd, *J* = 7.5 Hz, 1H), 7.75 (t, *J* = 10.0 Hz, 1H), 8.22 (d, *J* = 10.0 Hz, 1H), 8.34 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 20.37, 20.39, 20.46, 20.5, 60.3, 61.5, 61.7, 68.0, 68.18, 68.24, 68.4, 69.1, 69.5, 70.0, 72.8, 77.4, 161.9, 165.8, 169.1, 169.3, 169.3, 169.6, 170.2, 170.5, 170.7, 170.8. HRMS (ESI): m/z calcd for $C_{15}H_{22}NO_{10}$ $[M+H]^+$ 376.1238, found 376.1239.

α-D-Xylosyl formamide S-10. A solution of **S-11** (40 mg, 0.14 mmol) dissolved in a mixture of aqueous 0.1 N HCl (0.4 mL) and tetrahydrofuran (1.6 mL) was heated at 50 °C for 7.5 h, stirred at room temperature for 12.5 h, and heated at 50 °C for 1 h. The reaction mixture was diluted wit ethyl acetate and brine, and the separated aqueous layer was extracted with ethyl acetate (×3). The combined organic extracts were dried (Na_2SO_4) and concentrated to give the residue (42 mg), which was purified by silica gel chromatography (AcOEt/hexane 3:1) to furnish **S-10** (24 mg, 57%) as an oil: $[\alpha]_D^{22}$ = –13.2 (*c* 1.00, $CHCl_3$); IR (KBr) ν_{max} 3356, 1753, 1702, 1525, 1372, 1225 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ {2.07 (s)}, 2.08 (s, 3H), {2.10 (s)}, 2.12 (s, 3H), 2.14 (s, 3H), {3.81 (dd, *J* = 13.0, 5.5 Hz)}, 3.89 (dd, *J* = 12.0, 4.0 Hz, 1H), {3.91 (dd, *J* = 12.0, 4.0 Hz)}, 3.99 (dd, *J* = 13.0, 3.5 Hz, 1H), 4.75 (q, *J* = 3.5 Hz, 1H), 4.82 (dd, *J* = 5.0, 2.5 Hz, 1H), {4.87 (dd, *J* = 6.5, 3.5 Hz)}, 5.21 (t, *J* = 5.0 Hz, 1H), {5.24 (dd, *J* = 10.0, 3.5 Hz)}, {5.31 (t, *J* = 6.5 Hz)}, 5.69 (dd, *J* = 9.0, 2.5 Hz, 1H), 6.64–6.82 (br, N-H), 8.26 (s, CHO, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ {20.62}, {20.68}, 20.70, 20.71, {20.76}, 20.82, {62.2}, 63.8, 66.46, {66.90}, 67.1, {67.54}, 68.06, {68.44}, 72.8, {77.6}, 160.9, {165.0}, 169.02, {169.08}, 169.52, 169.55, {169.67}. HRMS (ESI): m/z calcd for $C_{12}H_{17}NO_8$ $[M+H]^+$ 304.1027, found 304.1027.



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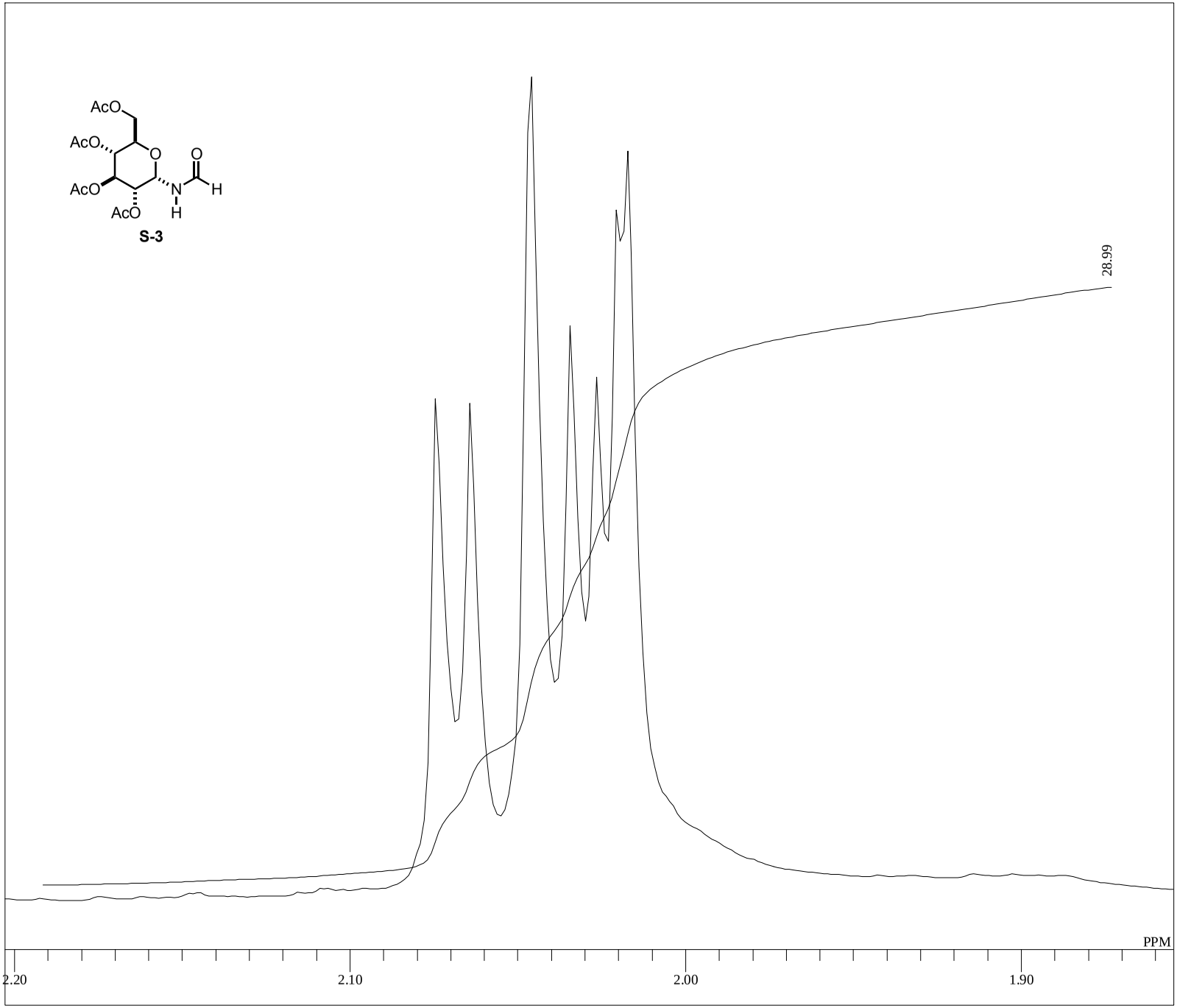

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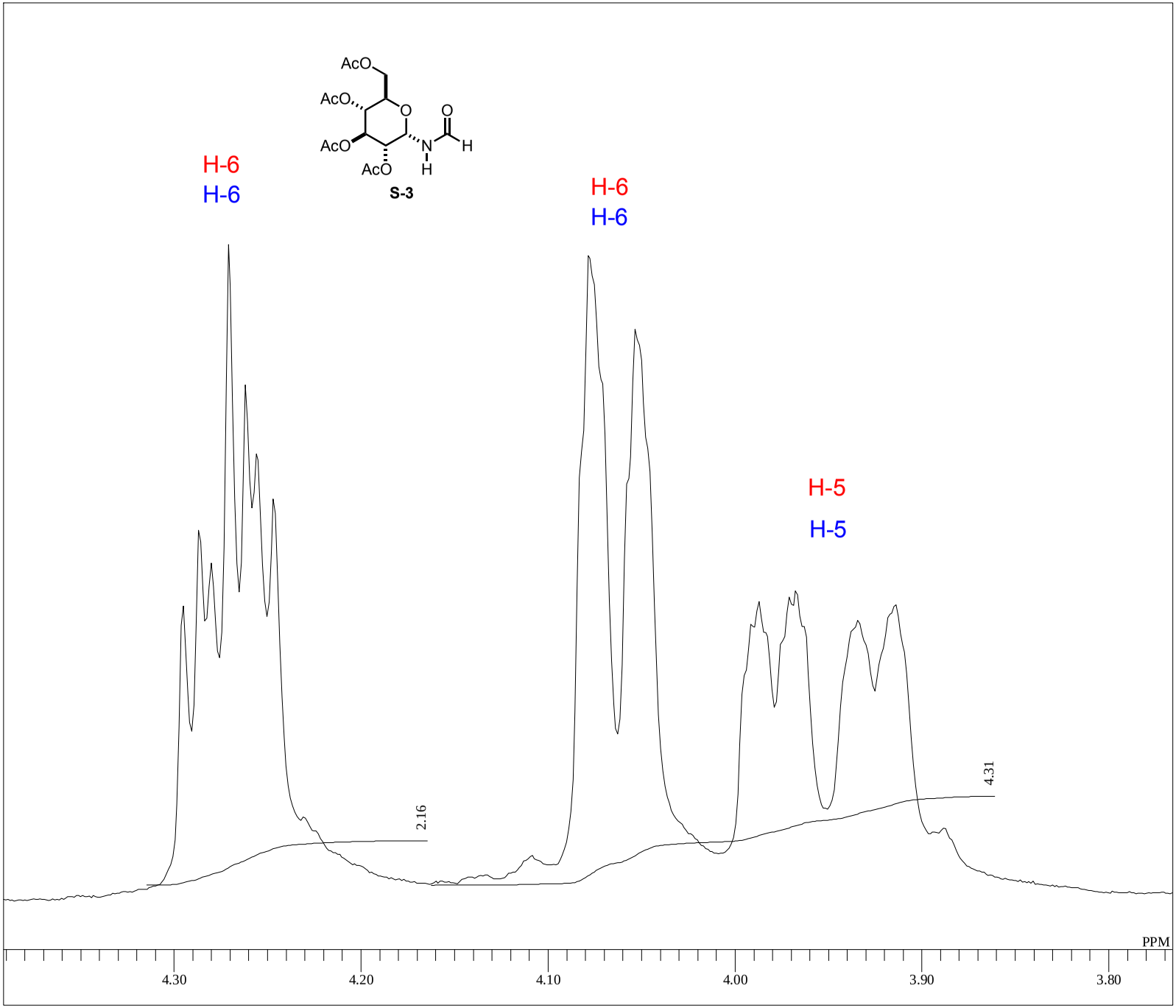

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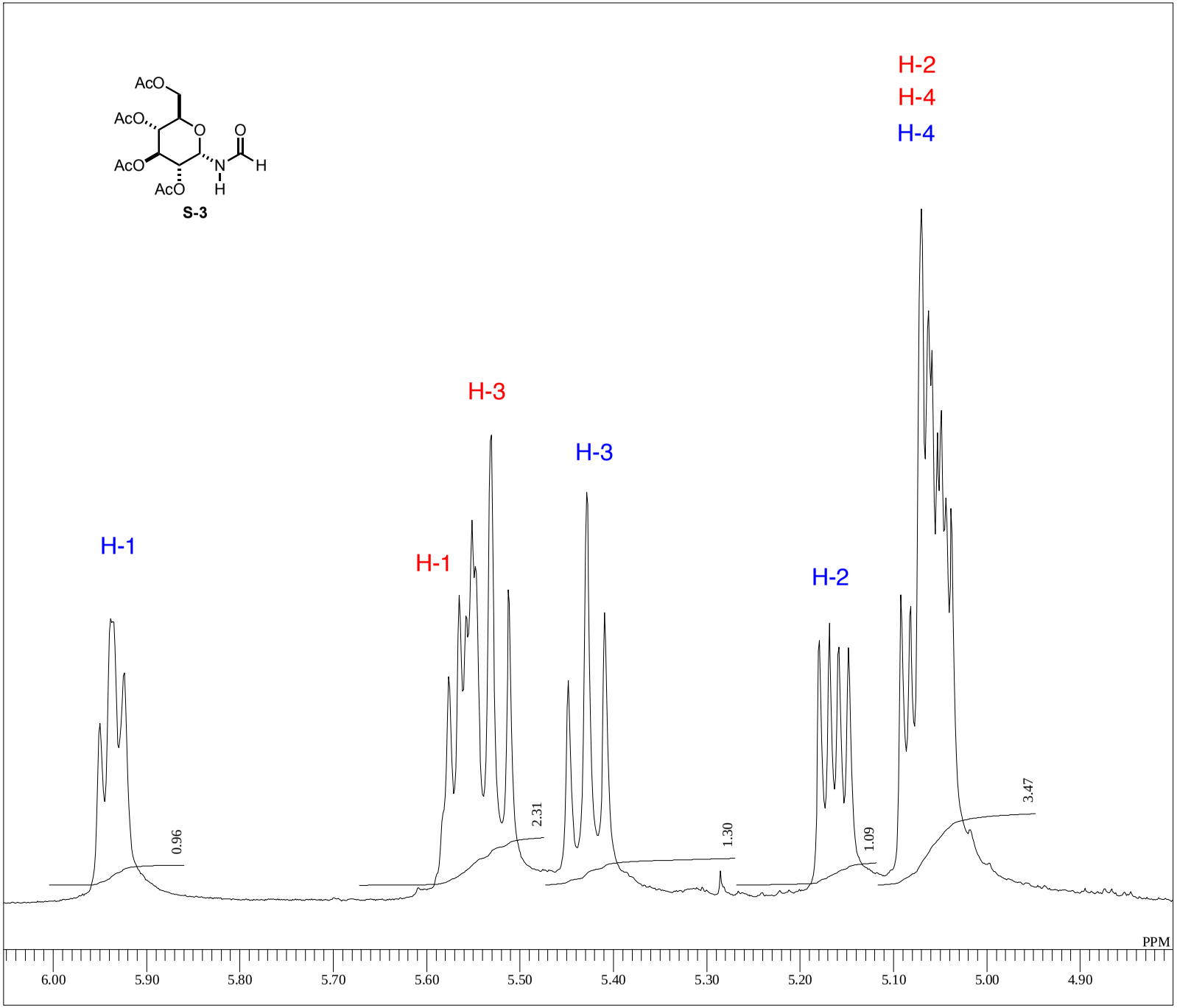

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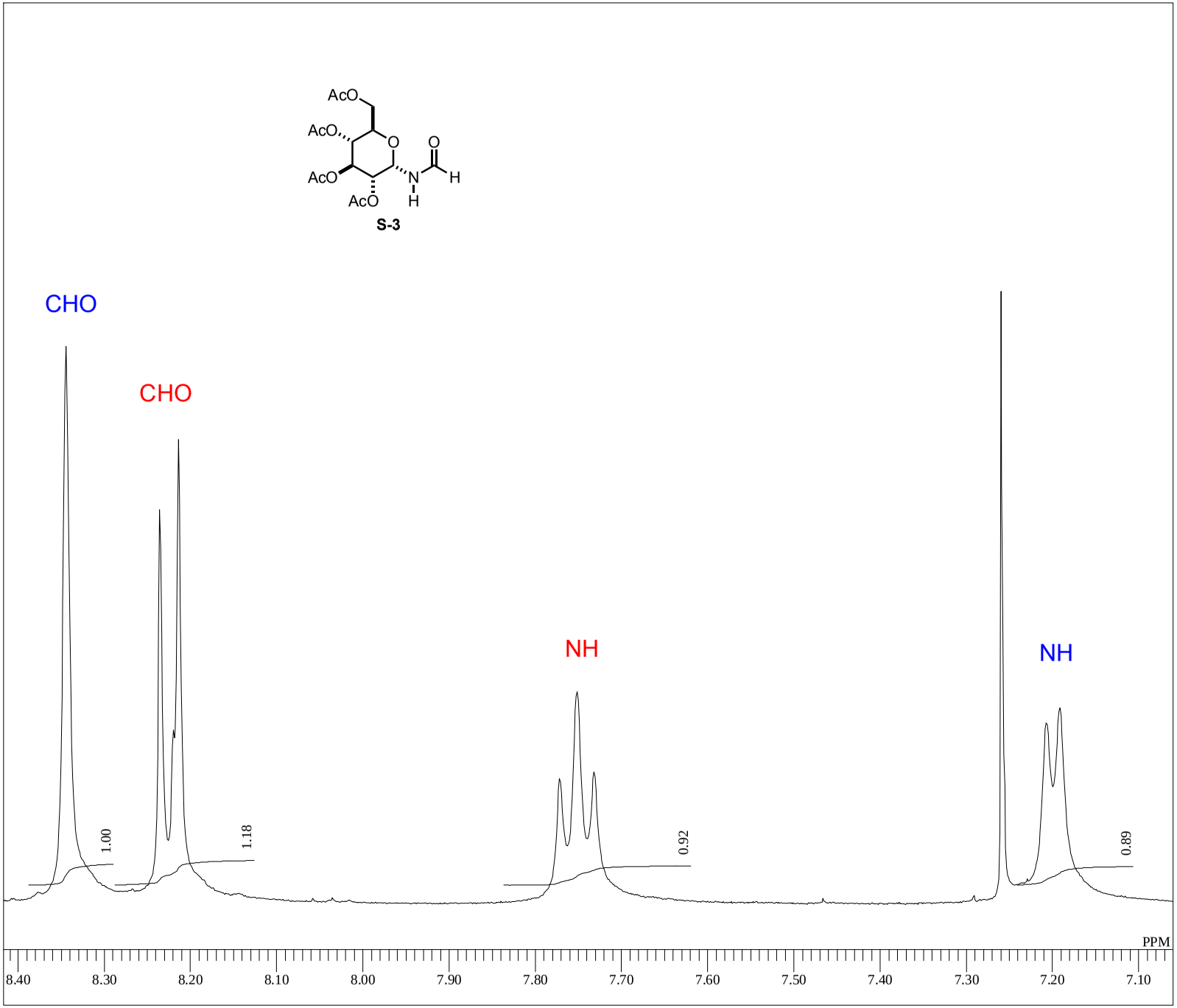

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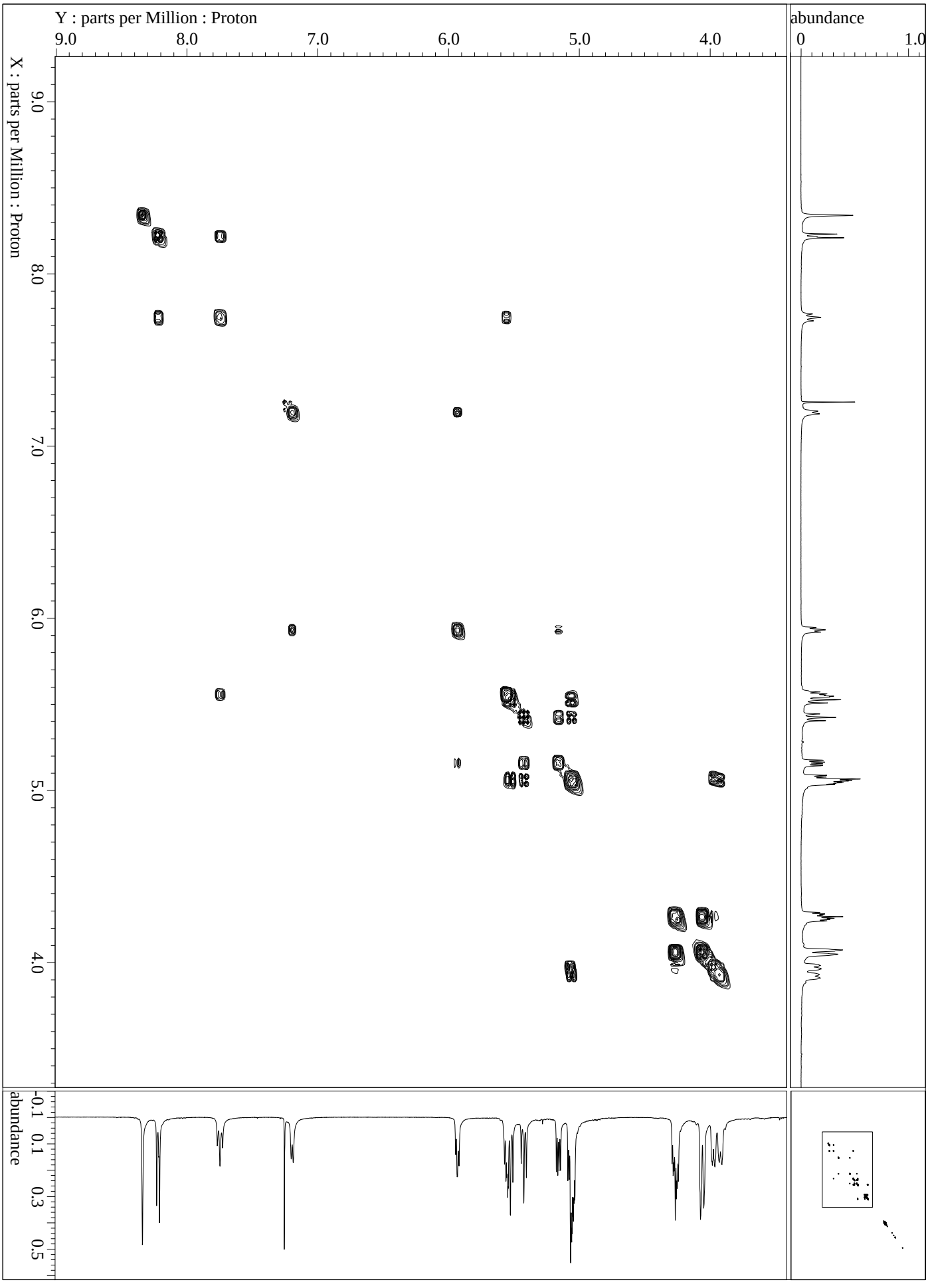
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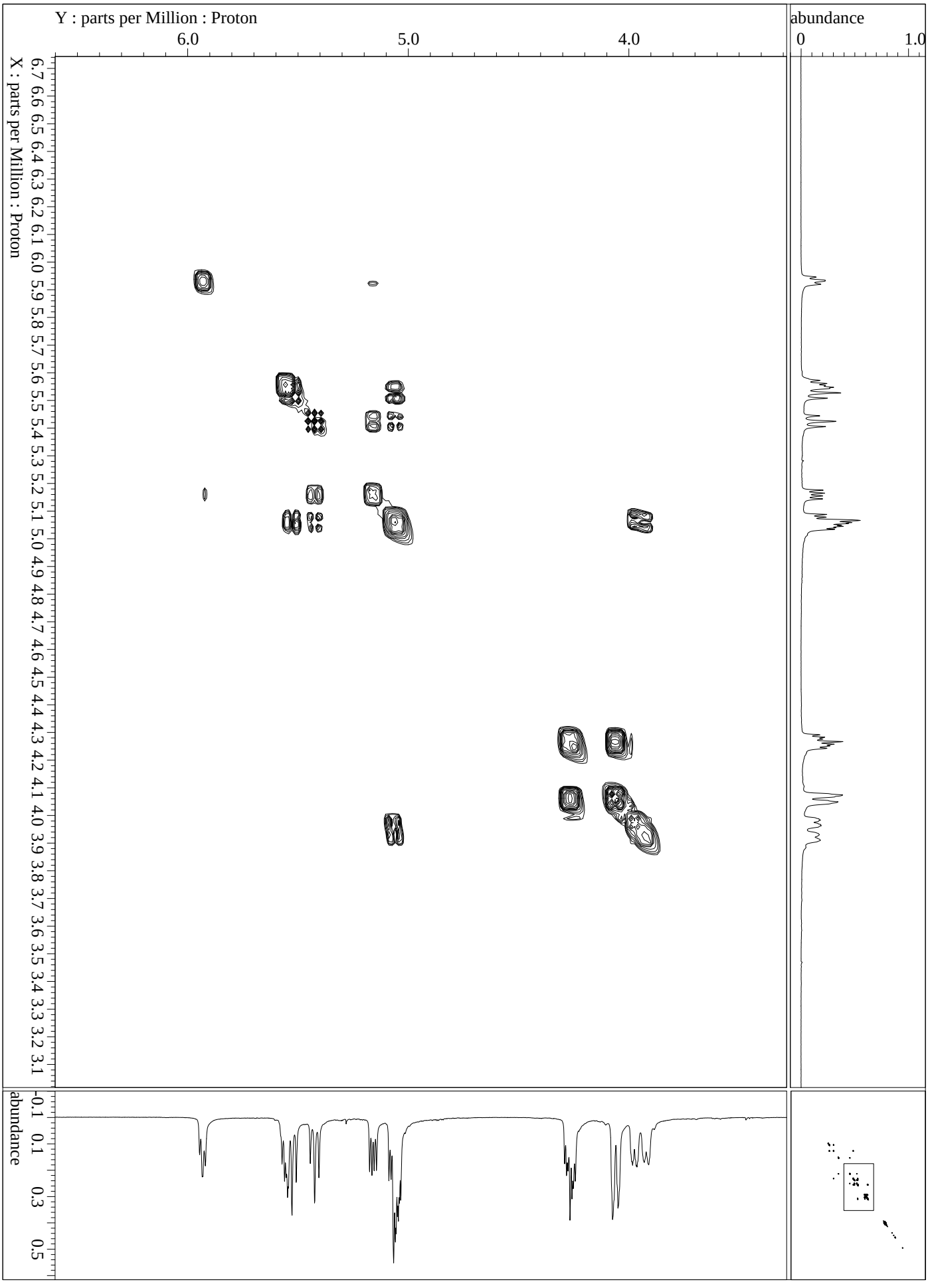
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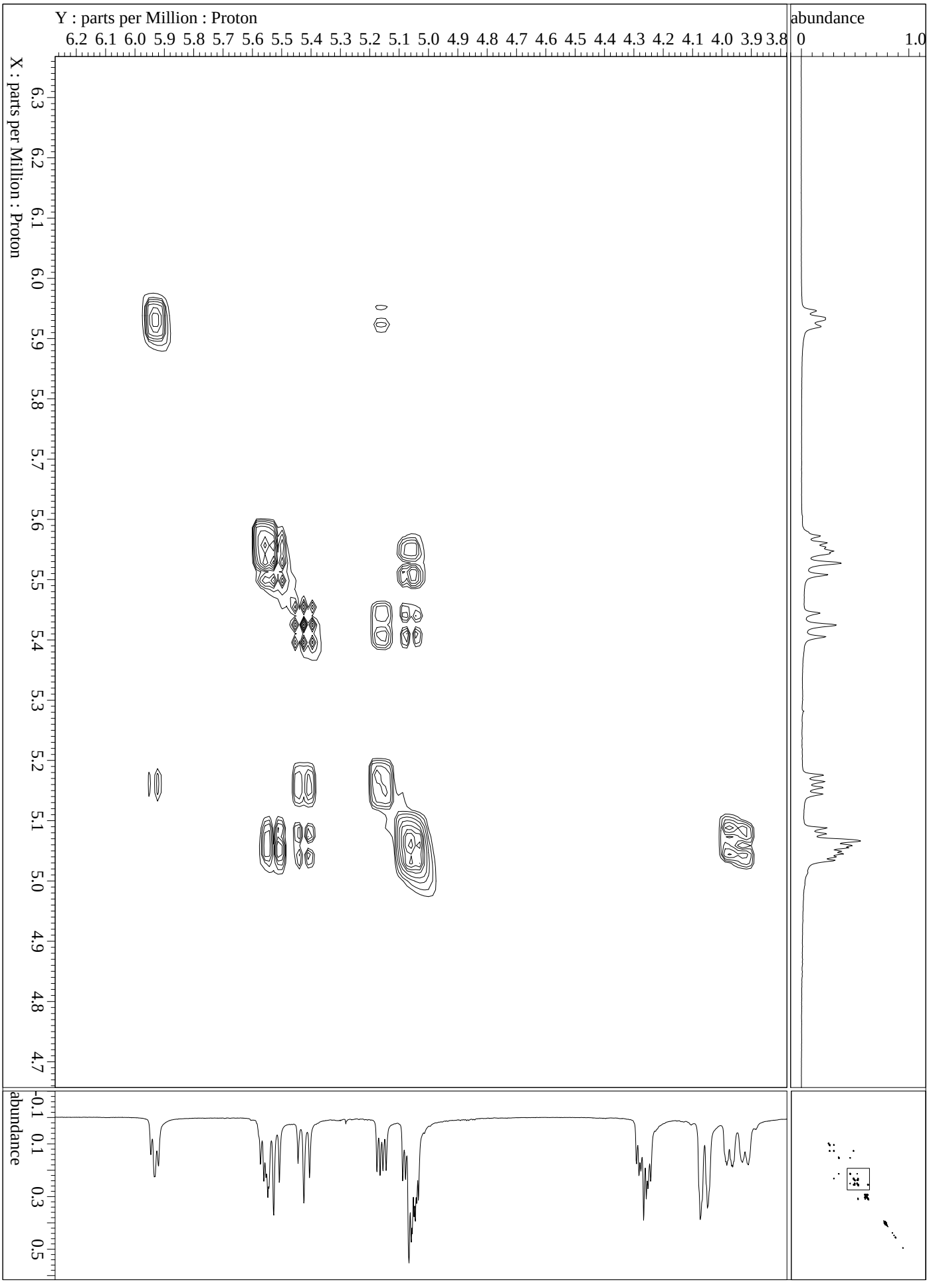
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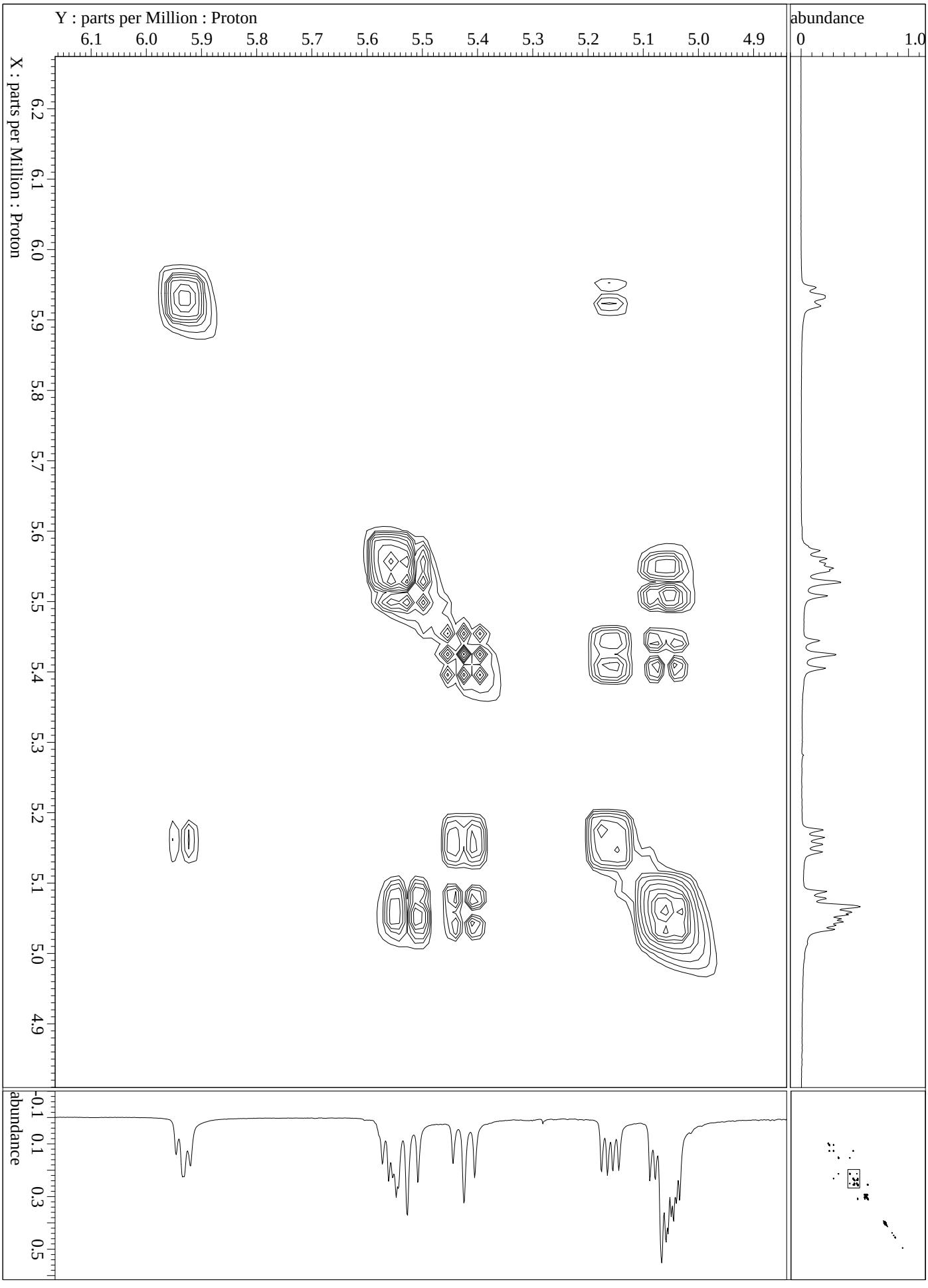
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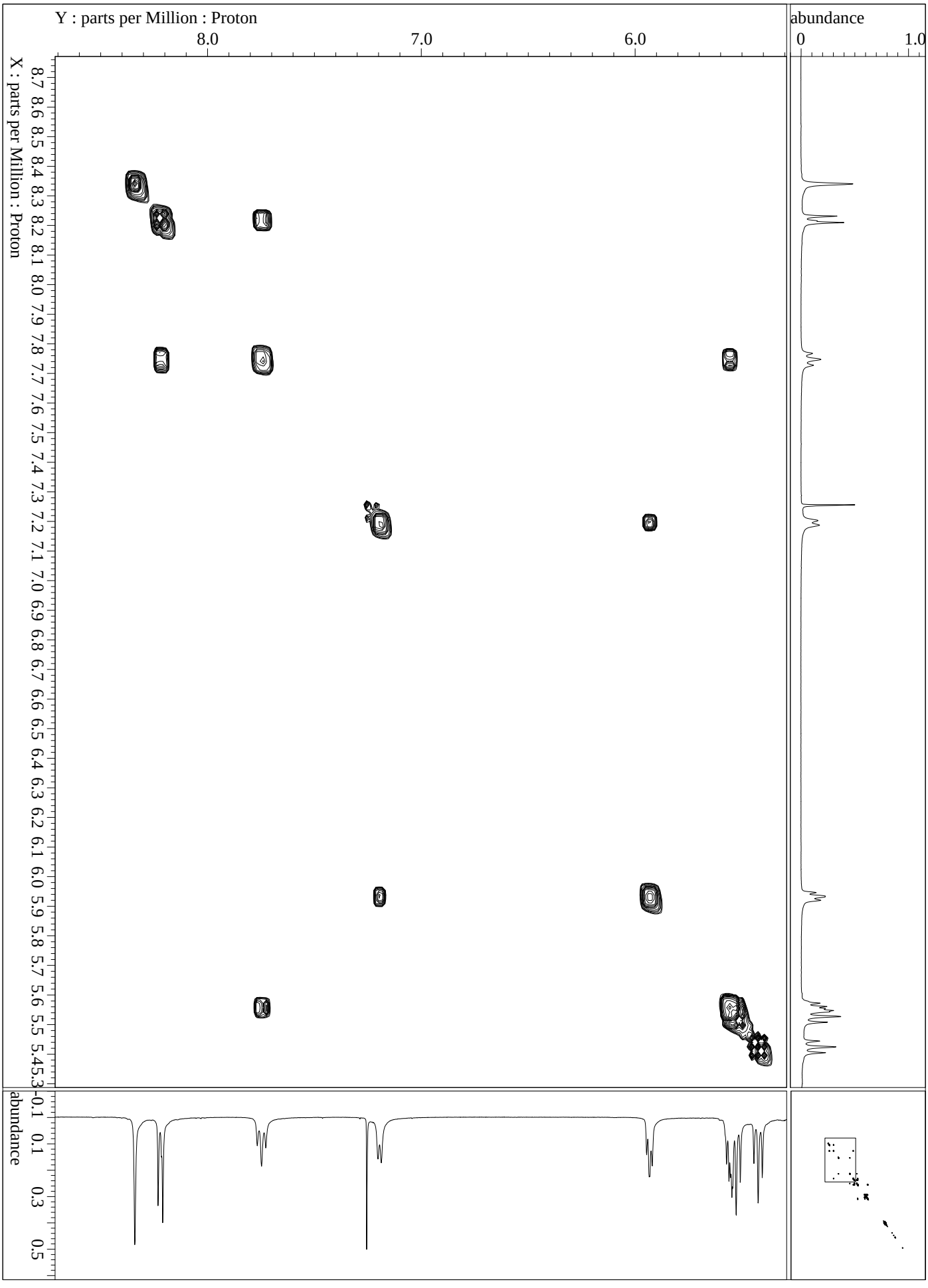
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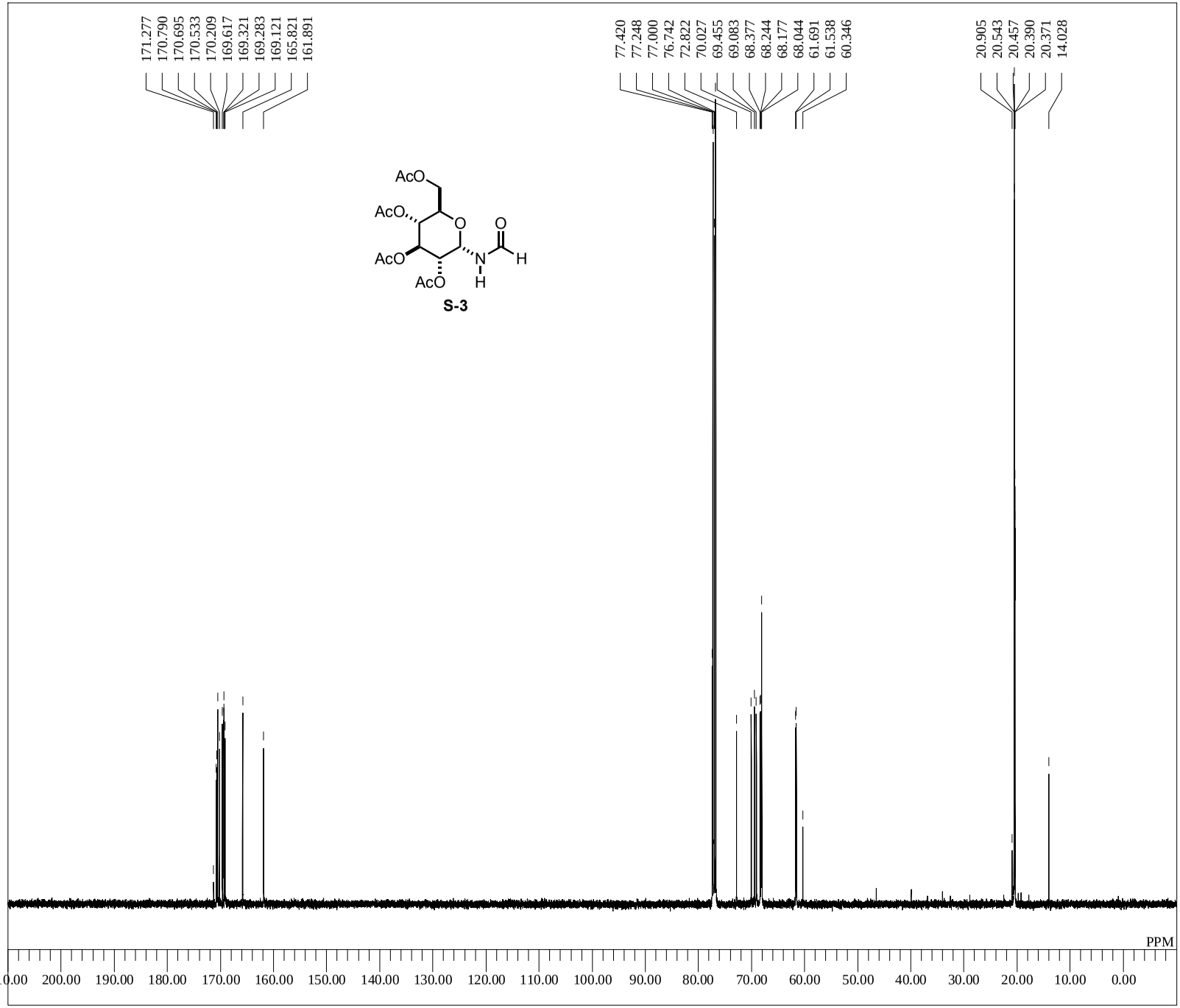

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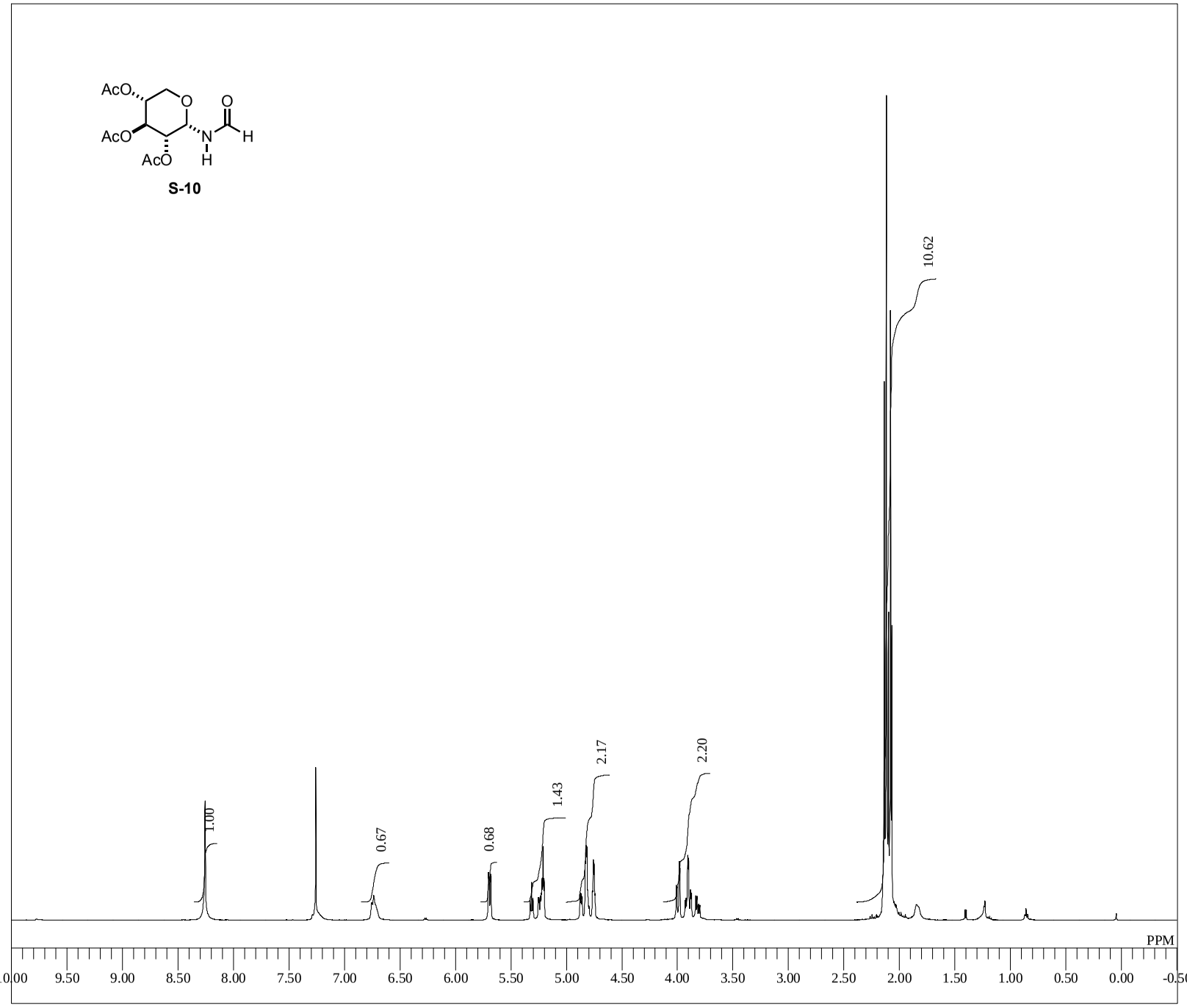

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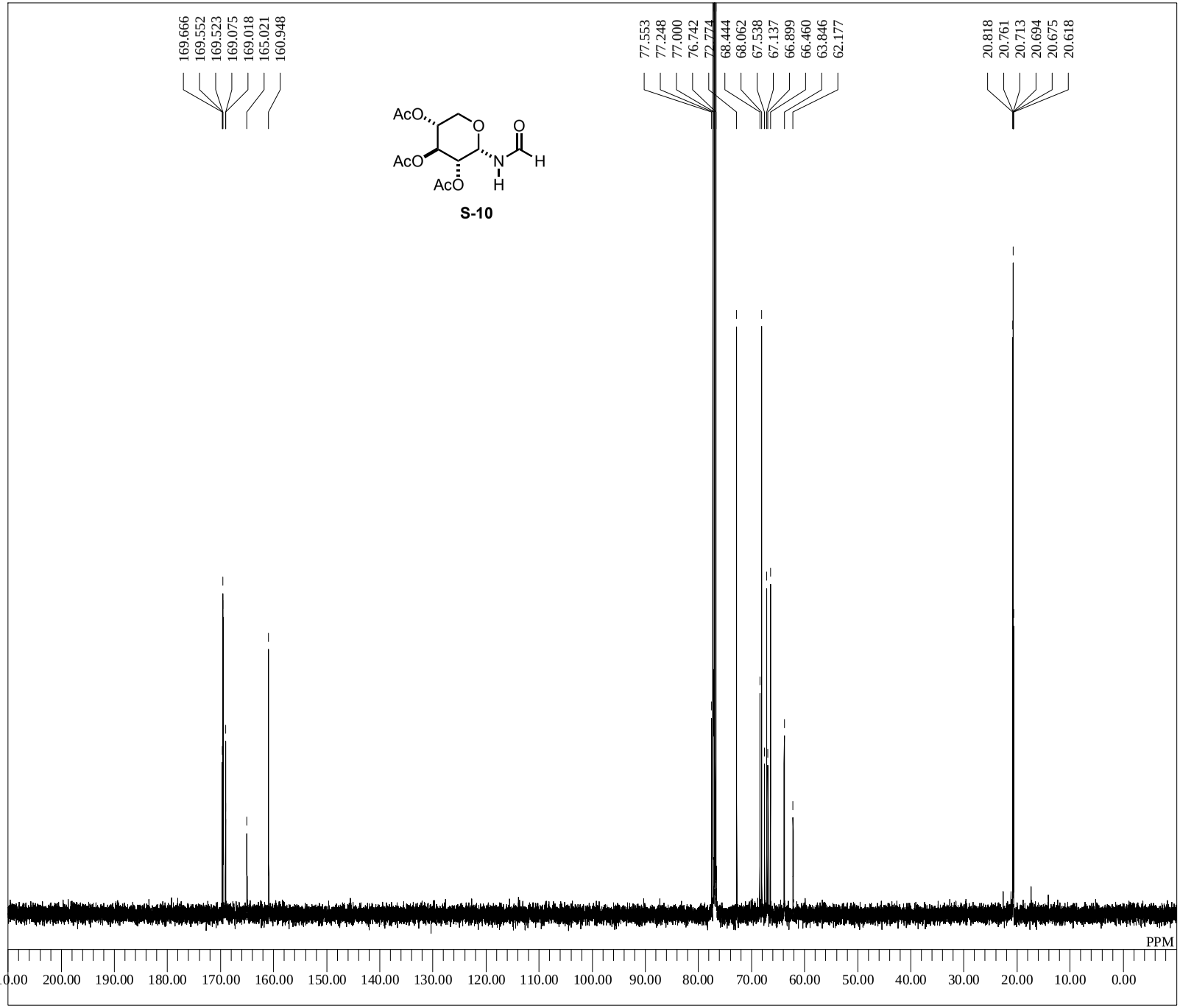

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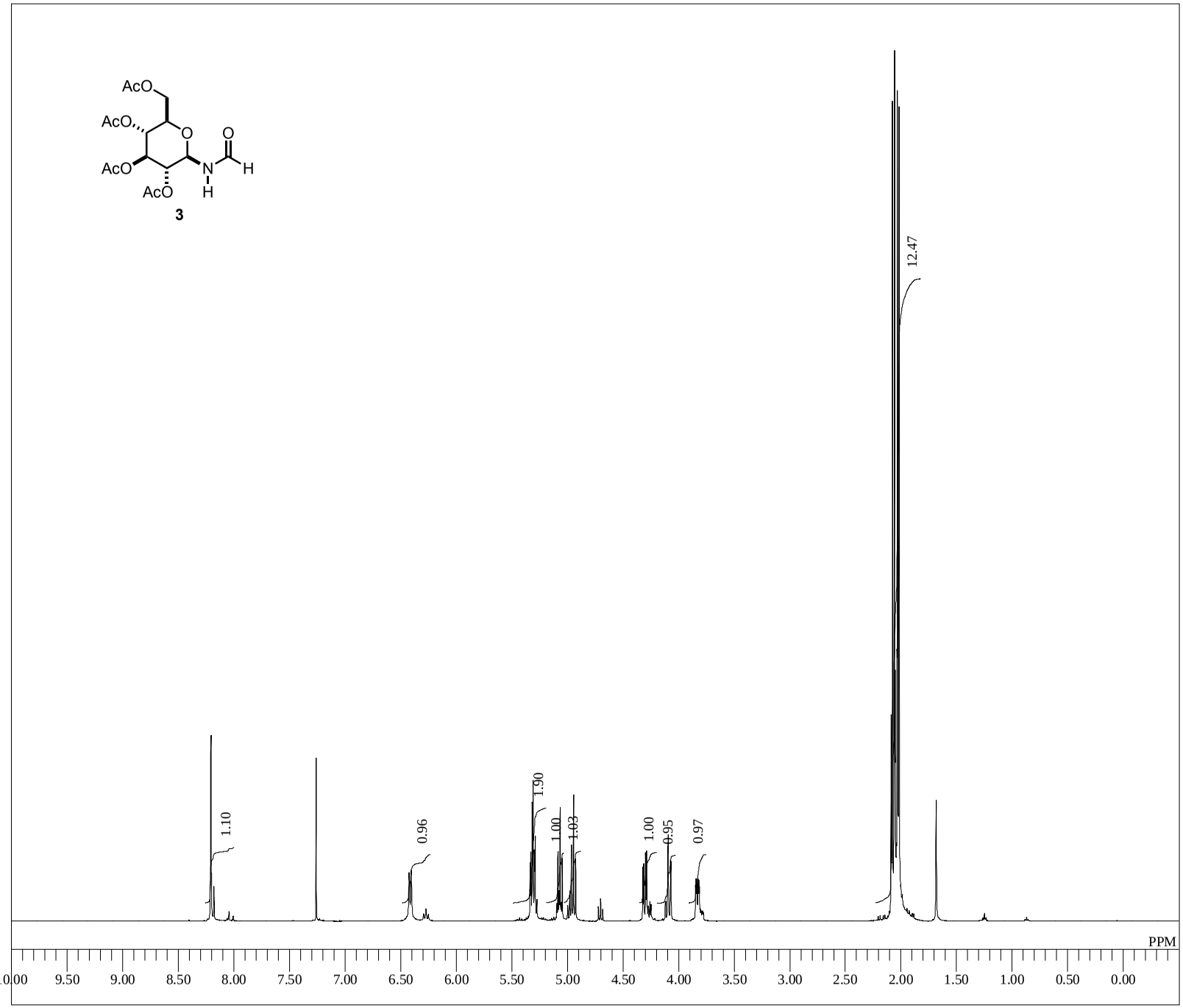

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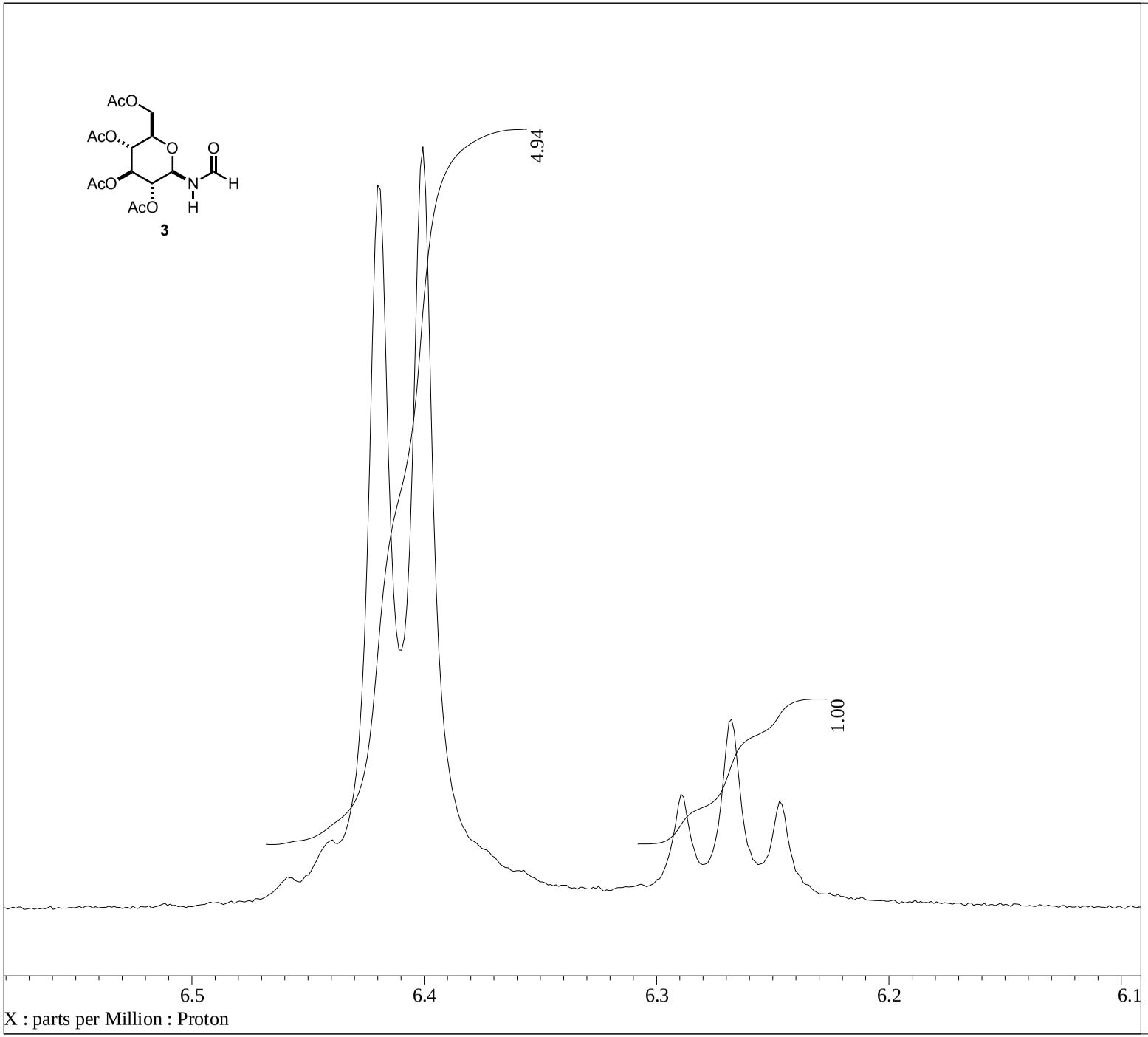


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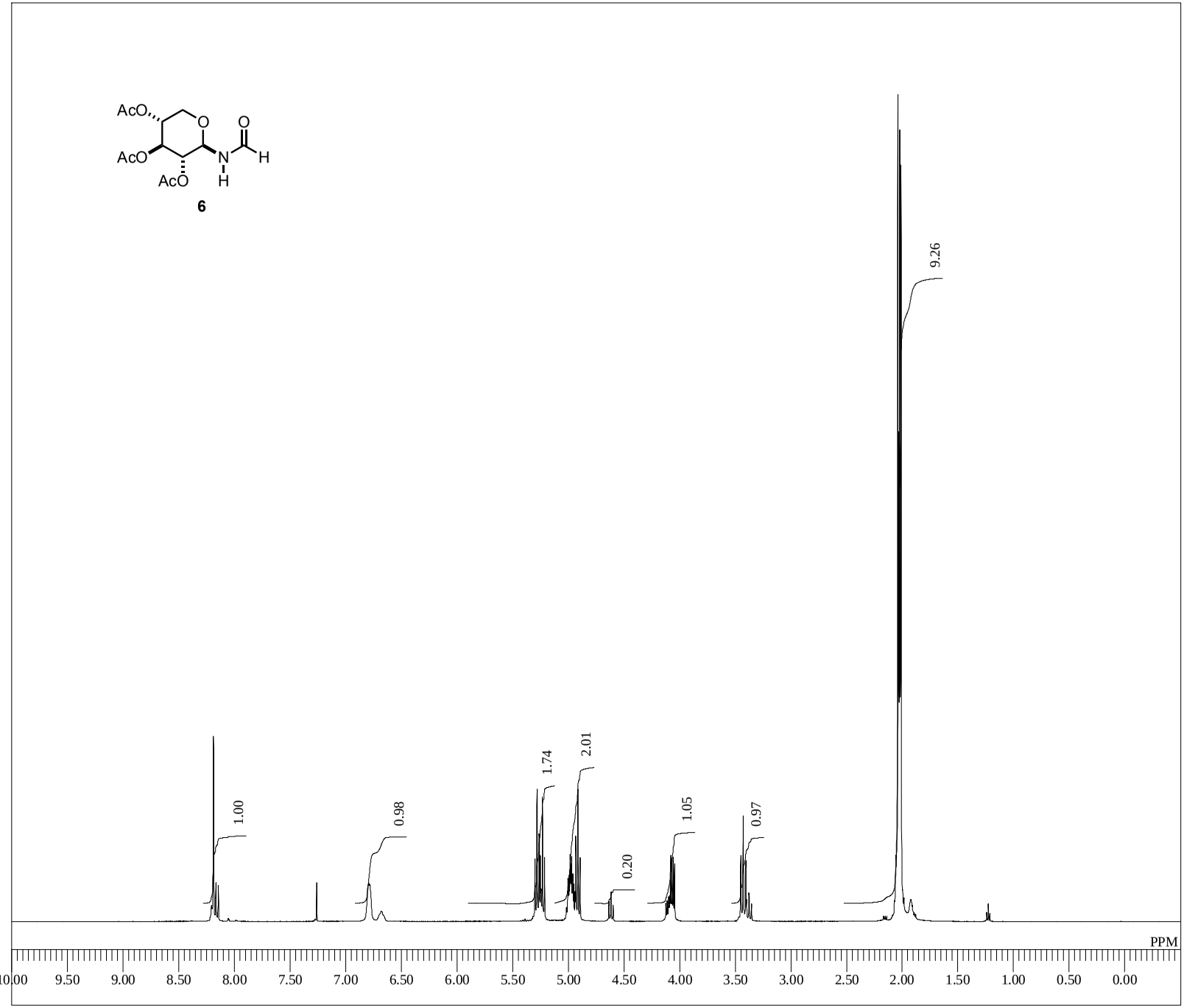
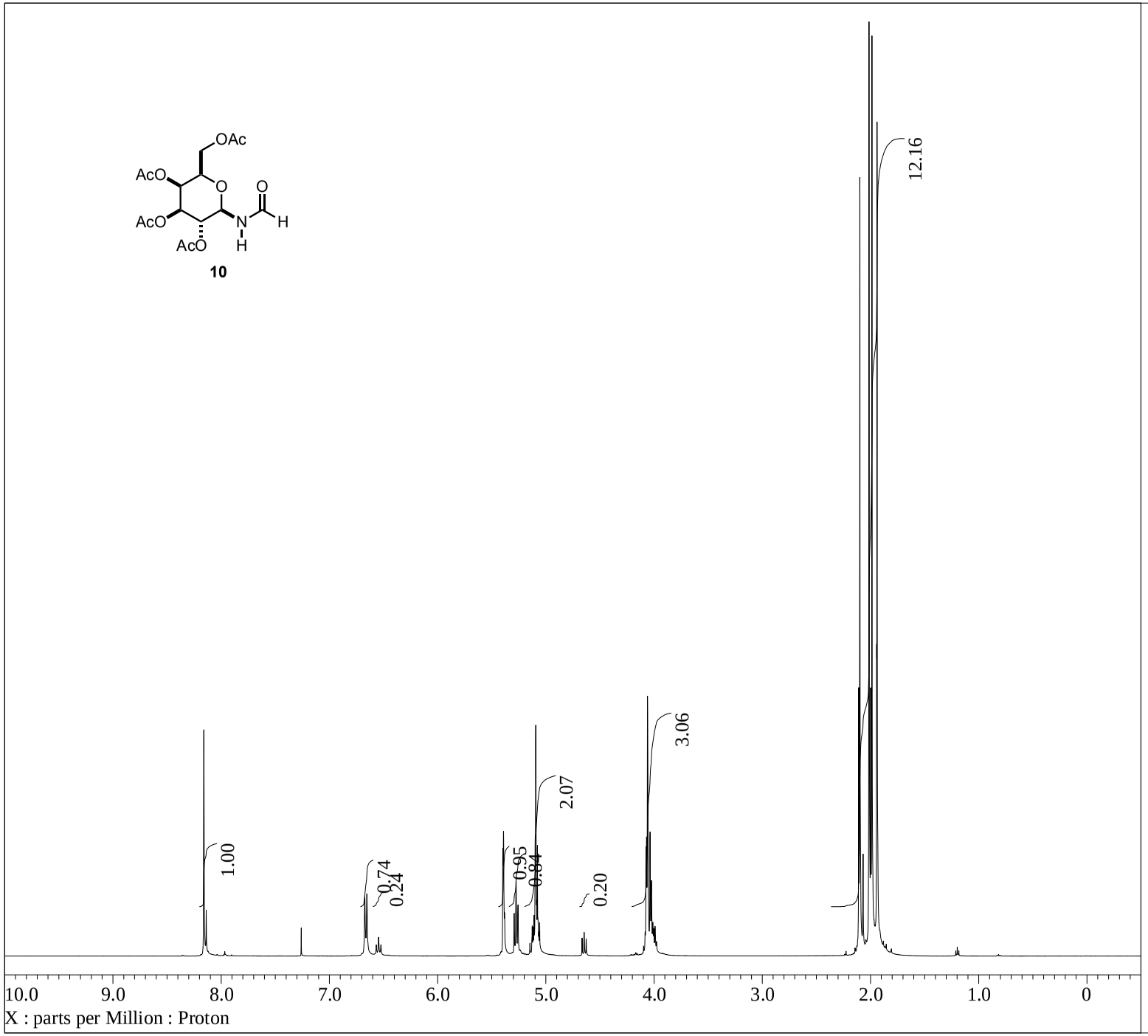
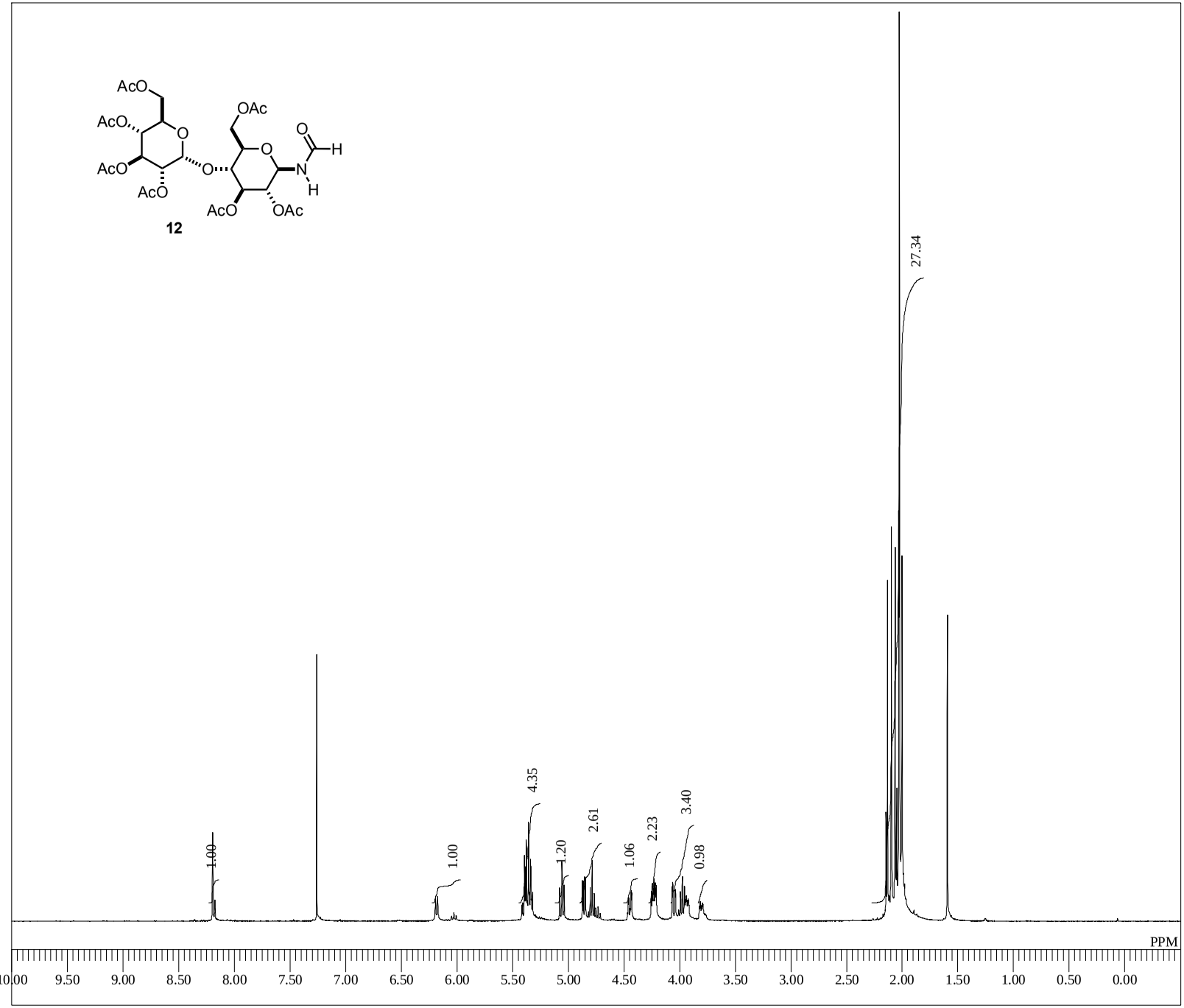


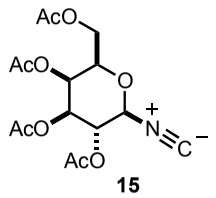
DFILE	ichikawa-CDCl3_proton-1-1.jdf
COMNT	single_pulse
DATIM	2016-06-04 11:14:12
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	21.8 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	34



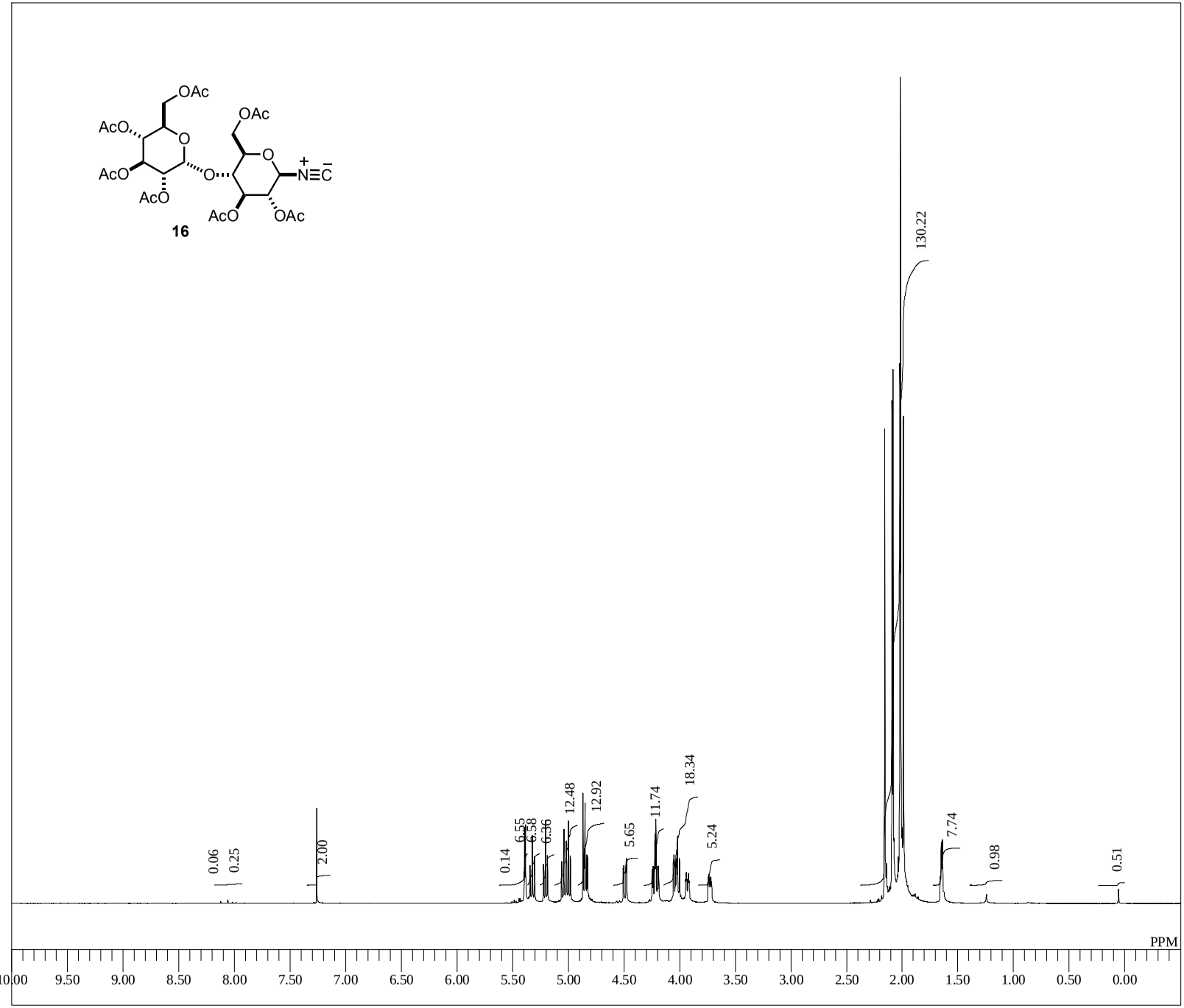

```
Filename          = MAT-ich-1040,1041_proton-2
Author            = delta
Experiment        = proton.jxp
Sample_Id         = MAT-ich-1040,1041
Solvent           = CHLOROFORM-D
Creation_Time     = 12-OCT-2016 11:40:47
Revision_Time     = 17-OCT-2016 15:36:44
Current_Time      = 17-OCT-2016 15:37:12

Comment           = single_pulse
Data_Format       = 1D COMPLEX
Dim_Size          = 13107
Dim_Title         = Proton
Dim_Units         = [ppm]
Dimensions        = X
Site              = JMN-ECA500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.7473579[T] (500[MHz])
X_Acq_Duration    = 1.74587904[s]
X_Domain          = 1H
X_Freq            = 500.15991521[MHz]
X_Offset          = 5.0[ppm]
X_Points          = 16384
X_Prescans        = 1
X_Resolution      = 0.57277737[Hz]
X_Sweep           = 9.38438438[kHz]
X_Sweep_Clipped   = 7.50750751[kHz]
Irr_Domain        = Proton
Irr_Freq          = 500.15991521[MHz]
Irr_Offset        = 5.0[ppm]
Tri_Domain        = Proton
Tri_Freq          = 500.15991521[MHz]
Tri_Offset        = 5.0[ppm]
Clipped           = FALSE
Scans             = 8
Total_Scans       = 8

Relaxation_Delay = 5[s]
Recvr_Gain        = 30
Temp_Get          = 21[dC]
X_90_Width        = 9.35[us]
X_Acq_Time        = 1.74587904[s]
X_Angle           = 45[deg]
X_Atn             = 2.8[dB]
X_Pulse           = 4.675[us]
Irr_Mode          = Off
Tri_Mode          = Off
Dante_Presat      = FALSE
Initial_Wait      = 1[s]
Repetition_Time   = 6.74587904[s]
```



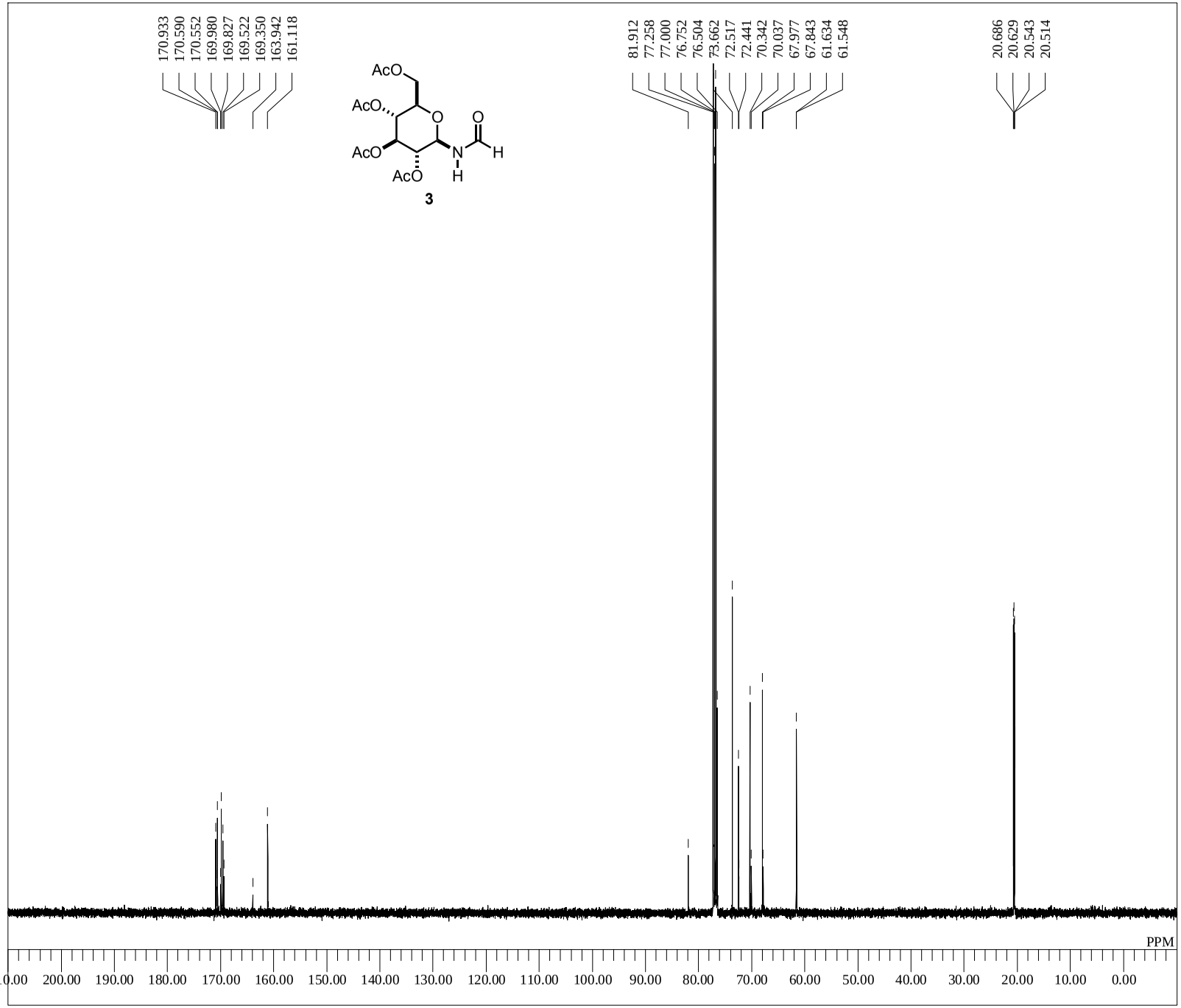
DFILE	MAT 2117_proton-1-1.jdf
COMNT	single_pulse
DATIM	2016-08-23 11:06:41
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	24.7 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	50



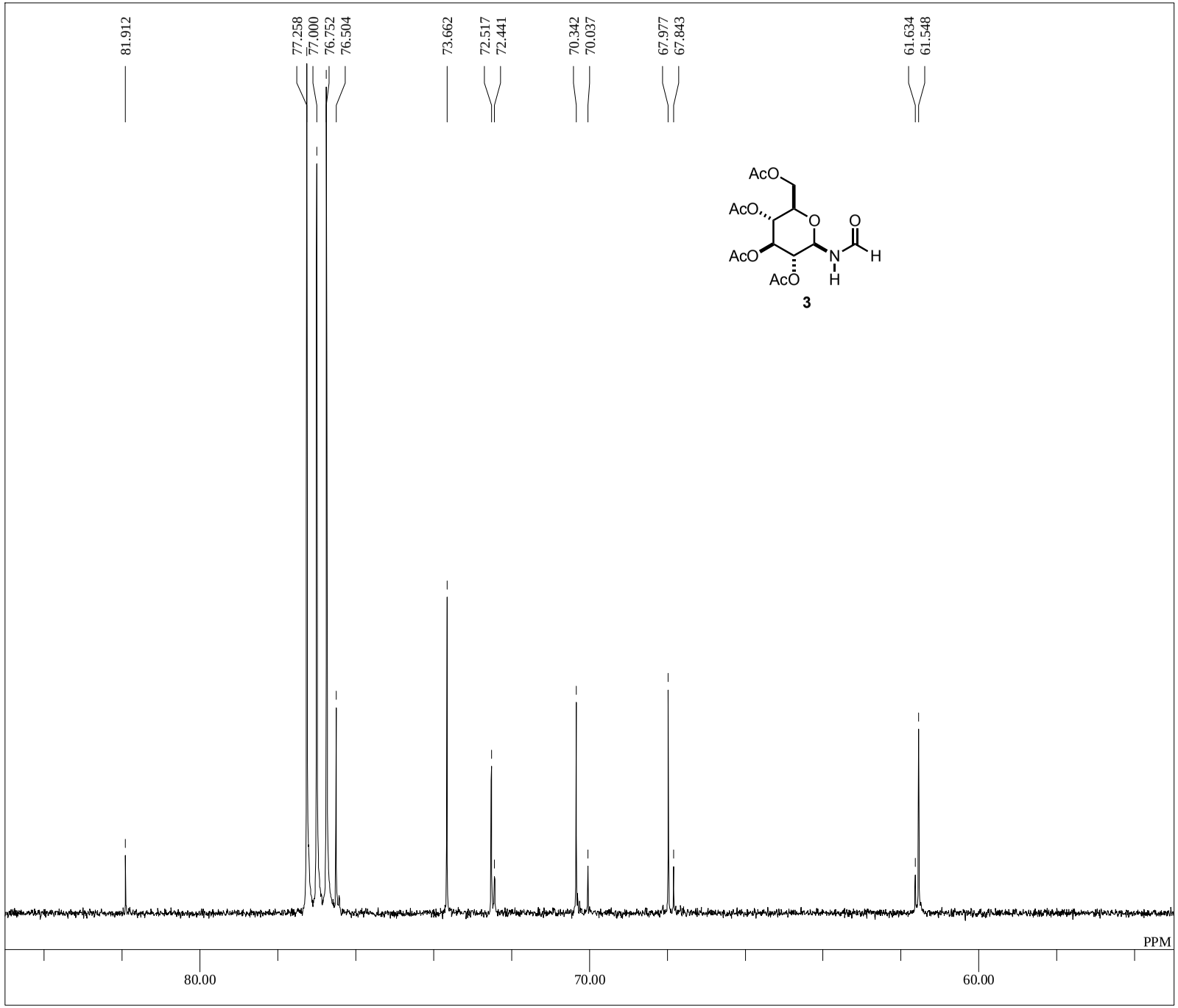
DFILE	MAE-185-recrystallized4_proton-1-1.jdf
COMNT	single_pulse
DATIM	2016-05-11 11:20:38
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	21.6 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40



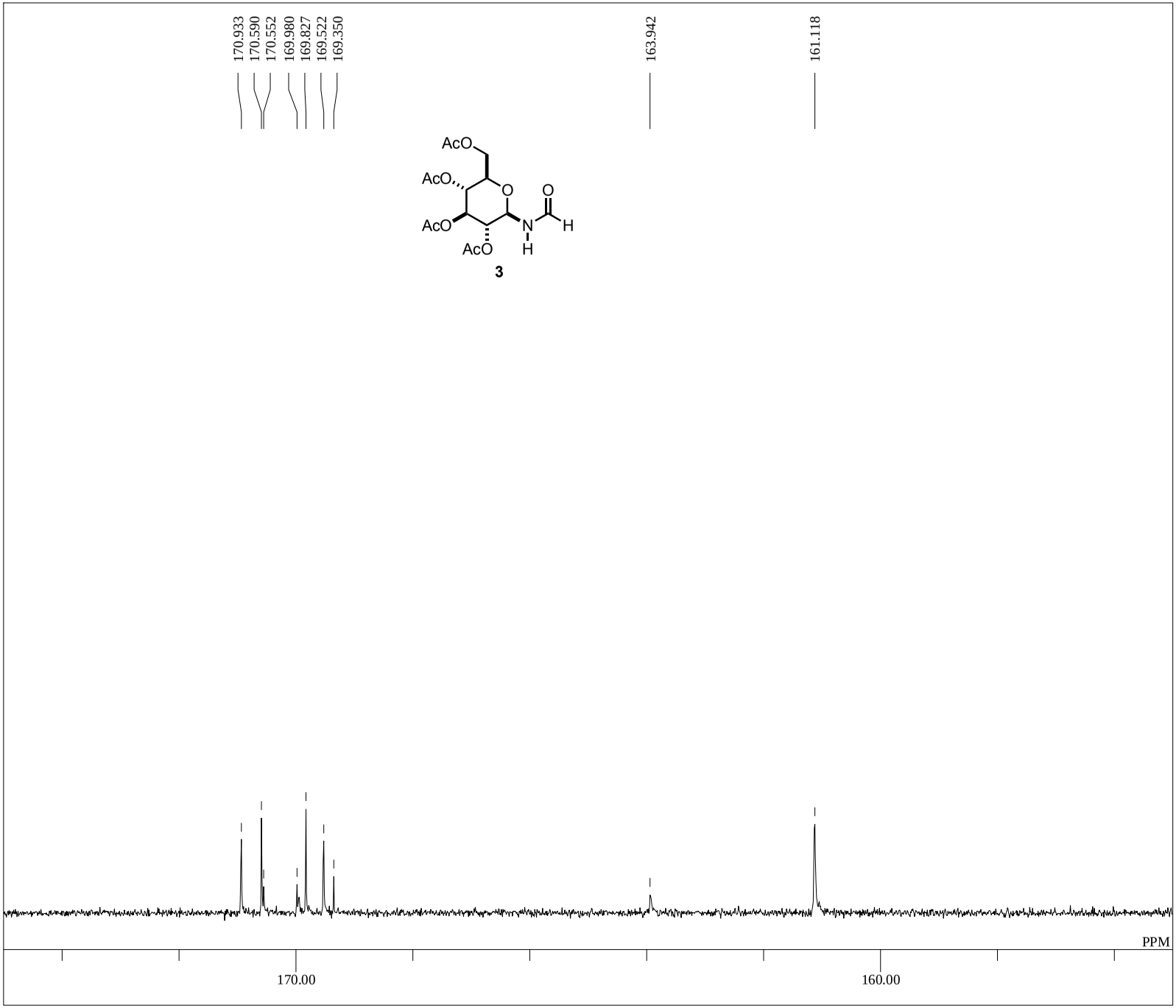
DFILE	MAT-2101-column_proton-2-1.jdf
COMNT	single_pulse
DATIM	2016-07-14 11:30:27
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	4.68 usec
IRNUC	1H
CTEMP	25.4 c
SLVNT	CDCL3
EXREF	7.26 ppm
BF	0.12 Hz
RGAIN	40



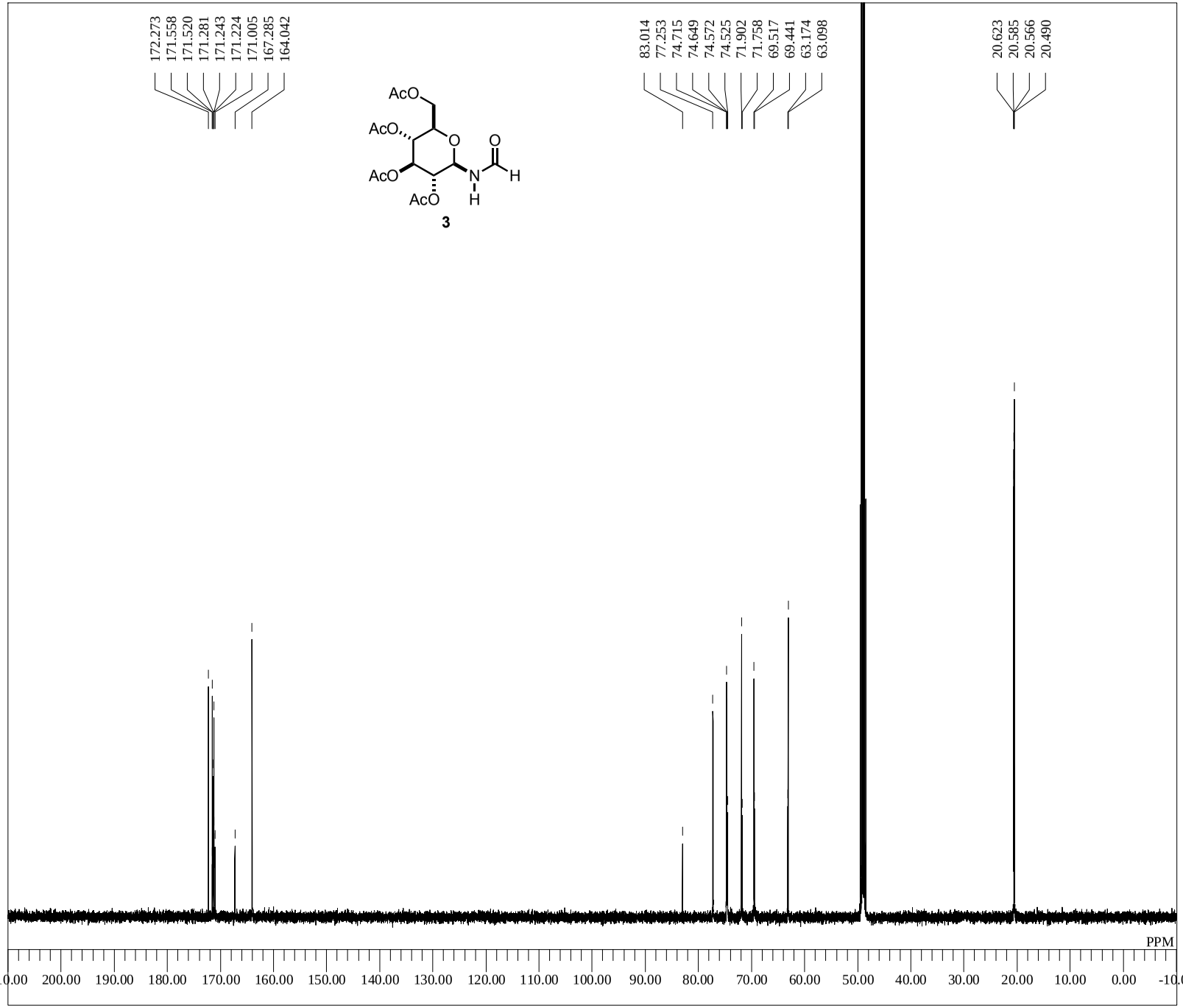
DFILE	mat glucose_carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2016-04-18 15:48:52
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	19.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



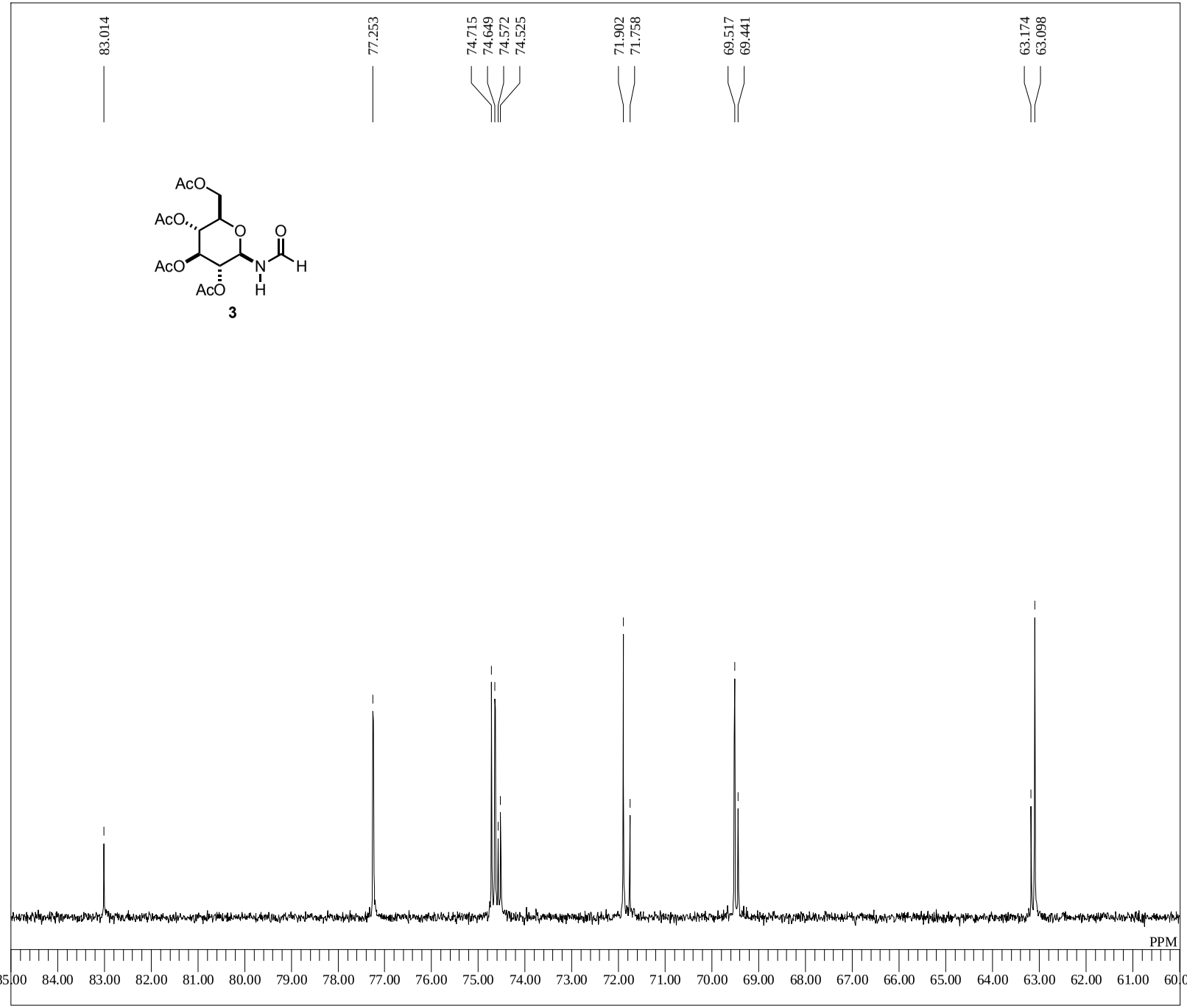
DFILE	mat glucose_carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2016-04-18 15:48:52
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	19.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



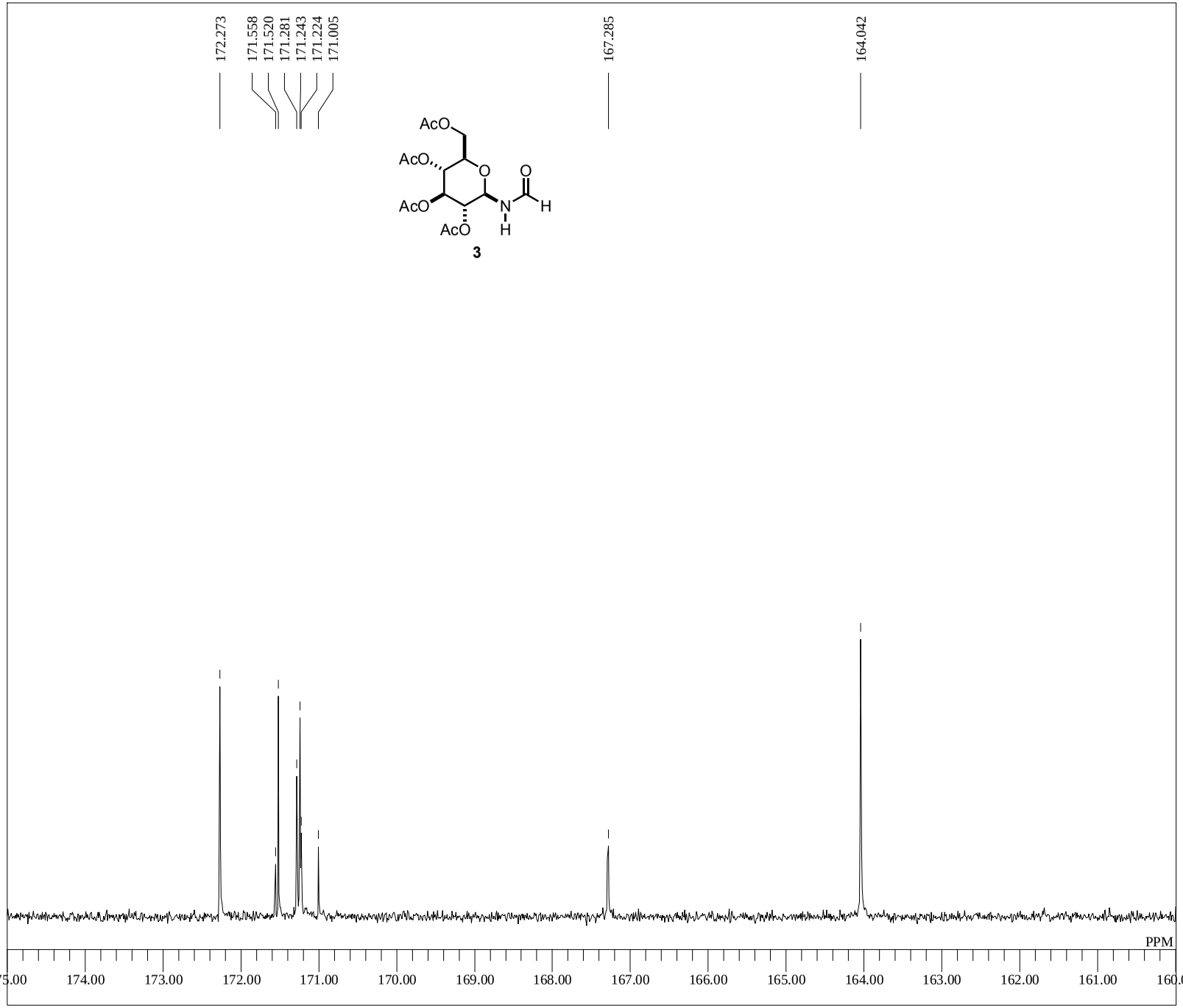
DFILE	mat glucose_carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2016-04-18 15:48:52
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	19.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	60



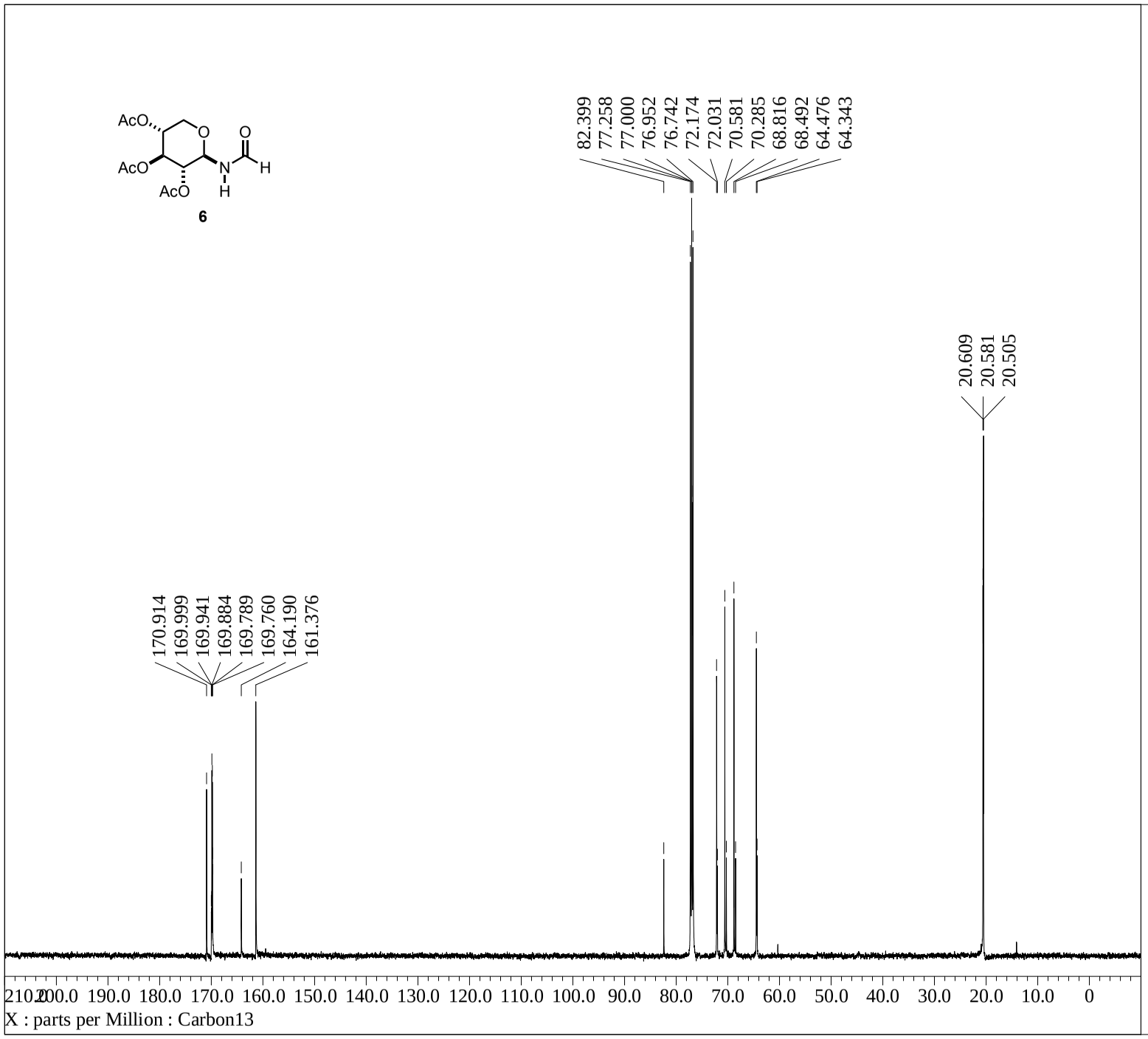
DFILE	MAT glucoseCD3OD_carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2016-04-18 21:02:09
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	19.3 c
SLVNT	CD3OD
EXREF	49.00 ppm
BF	0.12 Hz
RGAIN	60



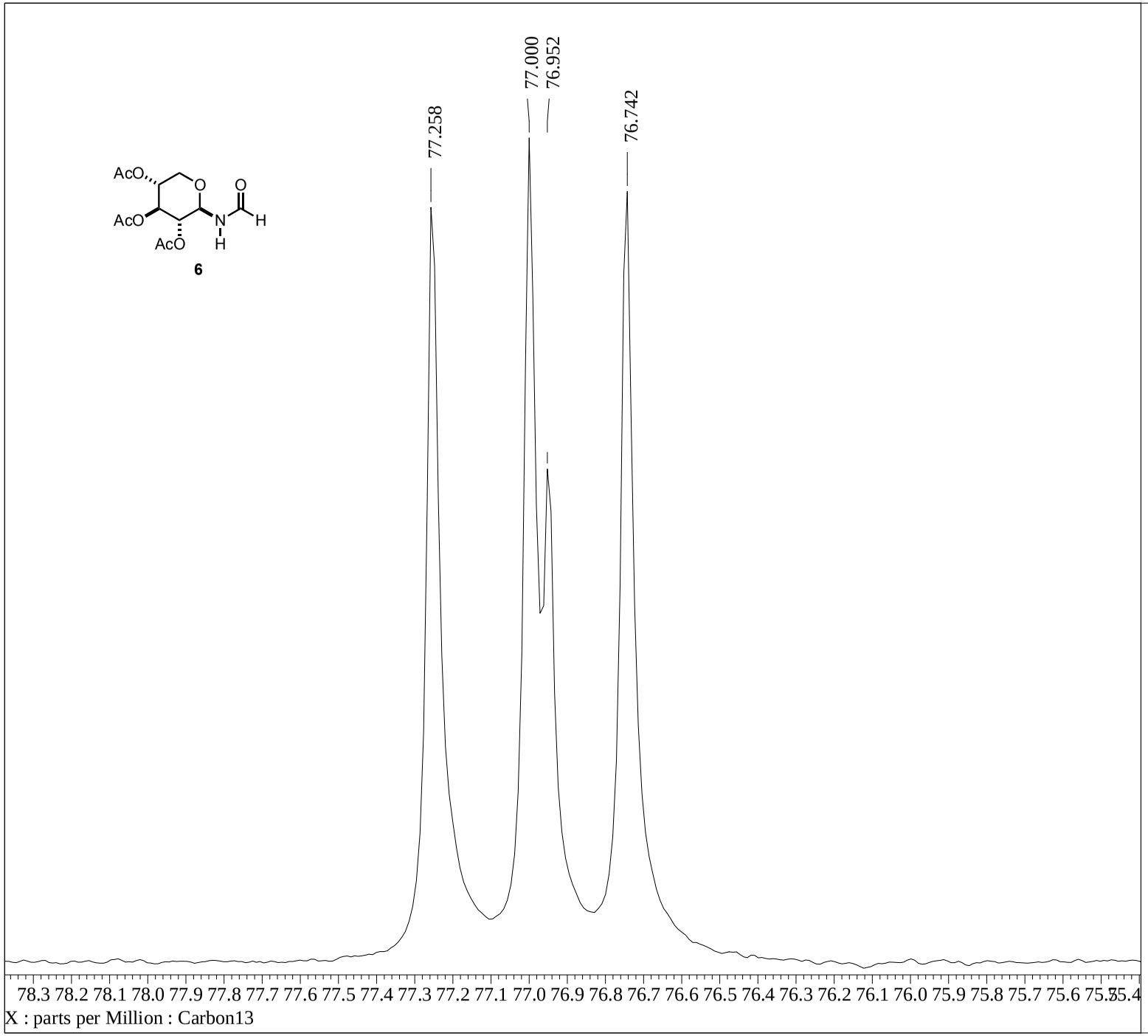
DFILE	MAT glucoseCD3OD_carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2016-04-18 21:02:09
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	19.3 c
SLVNT	CD3OD
EXREF	49.00 ppm
BF	0.12 Hz
RGAIN	60



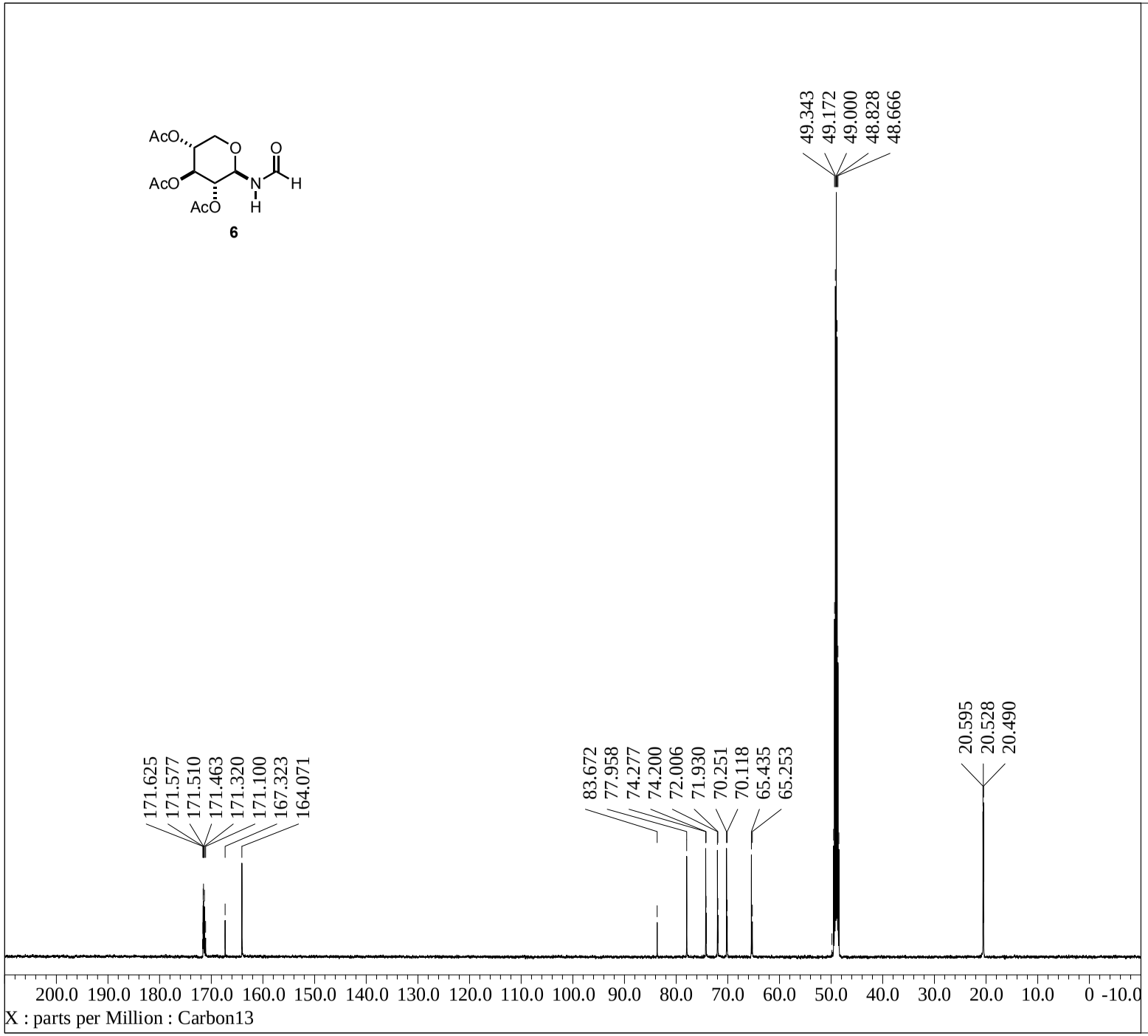
DFILE	MAT glucoseCD3OD_carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2016-04-18 21:02:09
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	19.3 c
SLVNT	CD3OD
EXREF	49.00 ppm
BF	0.12 Hz
RGAIN	60



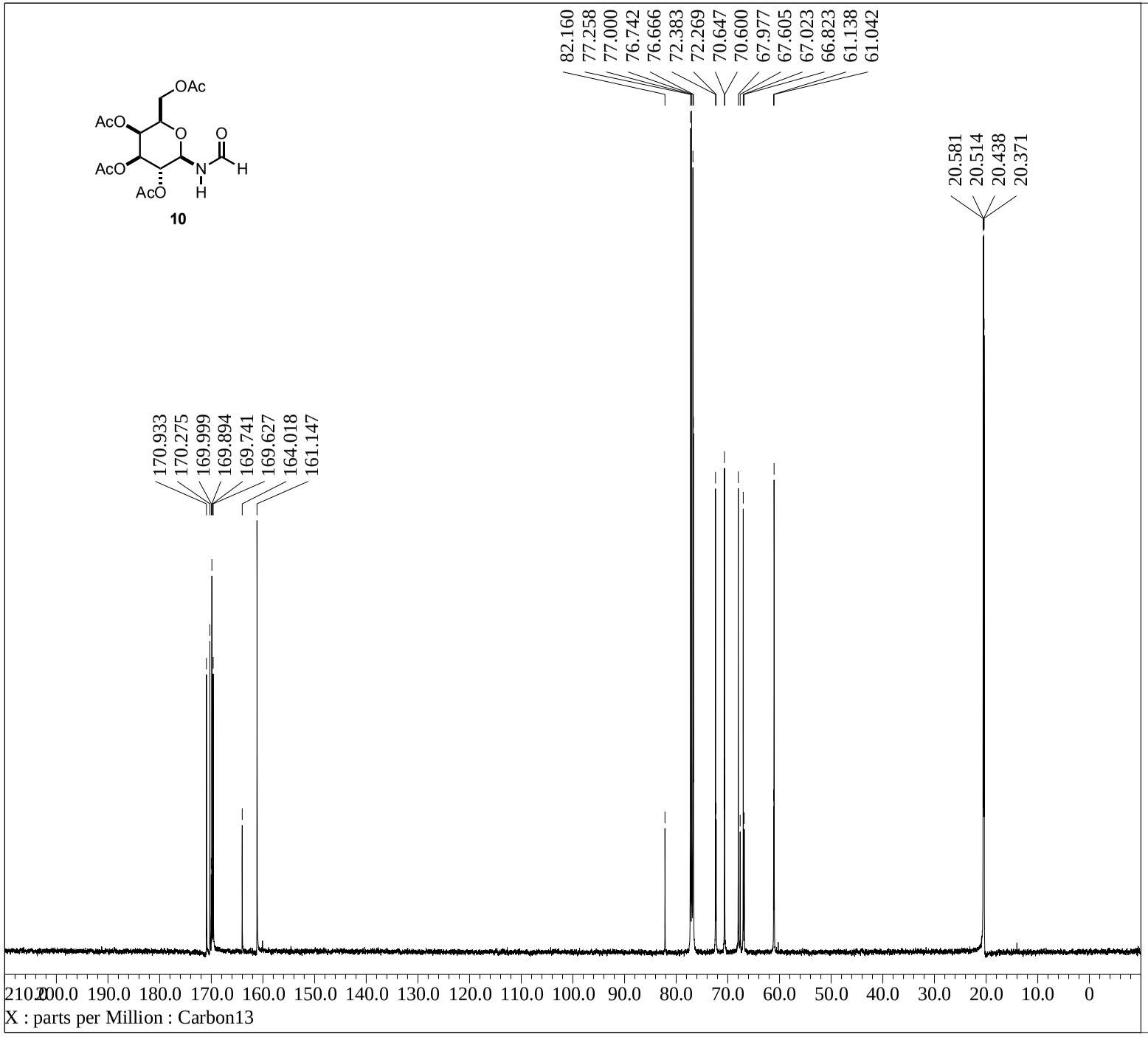

```
Filename          = ichikawa-1741-CDCl3_carbon
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = ichikawa-1741-CDCl3
Solvent           = CHLOROFORM-D
Creation_Time     =  4-JUN-2016 12:16:25
Revision_Time     = 13-OCT-2016 11:47:40
Current_Time      = 13-OCT-2016 11:50:30

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JMN-ECA500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.7473579[T] (500[MHz])
X_Acq_Duration    = 0.83361792[s]
X_Domain          = 13C
X_Freq            = 125.76529768[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.19959034[Hz]
X_Sweep           = 39.3081761[kHz]
X_Sweep_Clipped   = 31.44654088[kHz]
Irr_Domain        = Proton
Irr_Freq          = 500.15991521[MHz]
Irr_Offset        = 5.0[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay = 2[s]
Recvr_Gain        = 56
Temp_Get          = 22.1[dC]
X_90_Width        = 8.17[us]
X_Acq_Time        = 0.83361792[s]
X_Angle           = 30[deg]
X_Atn             = 6.5[dB]
X_Pulse           = 2.72333333[us]
Irr_Atn_Dec       = 22.66[dB]
Irr_Atn_Noe       = 22.66[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 92[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.83361792[s]
```



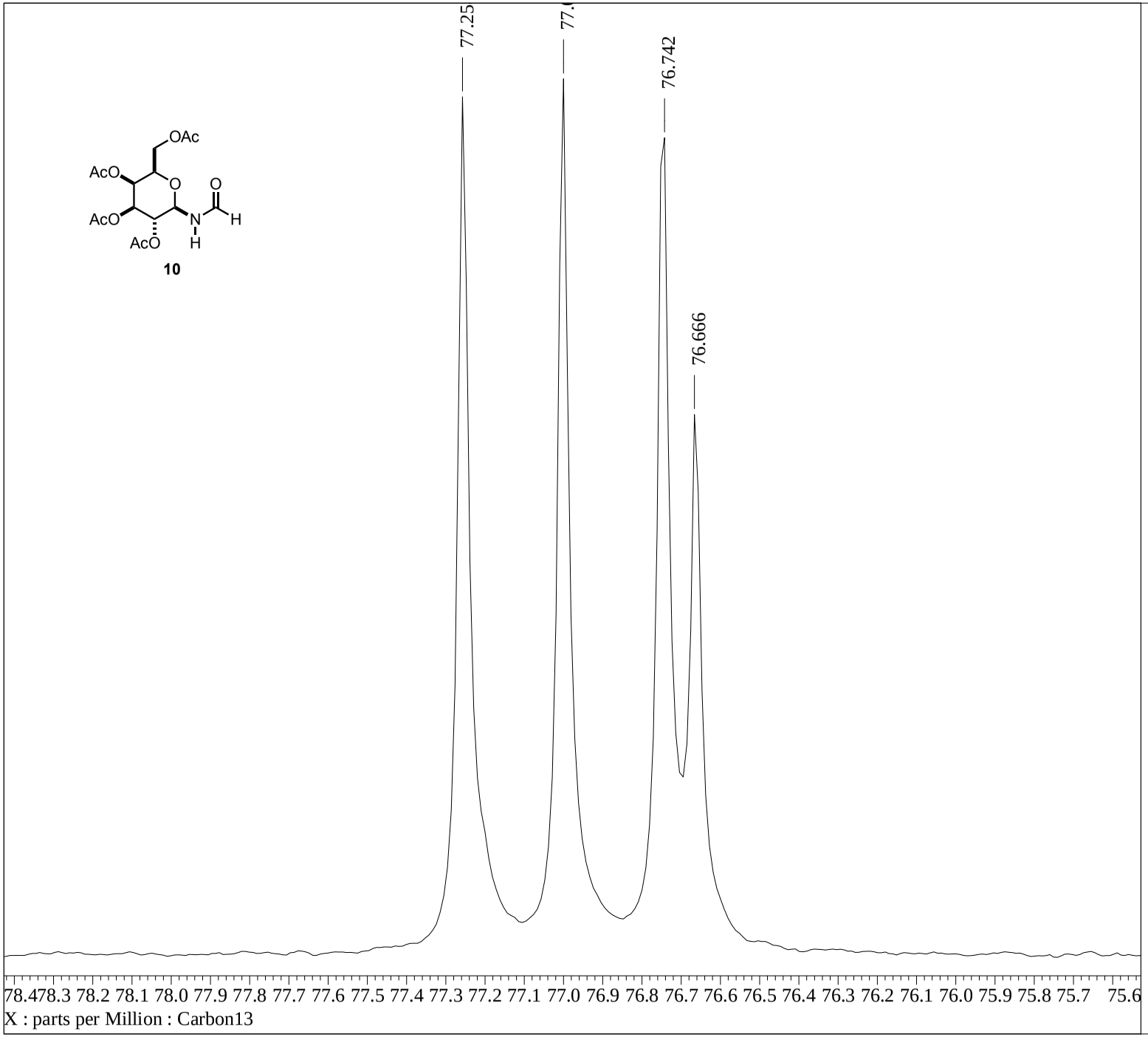

```
Filename          = ichikawa-1741-CDCl3_carbon
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = ichikawa-1741-CDCl3
Solvent           = CHLOROFORM-D
Creation_Time     =  4-JUN-2016 12:16:25
Revision_Time     = 13-OCT-2016 11:47:40
Current_Time      = 13-OCT-2016 11:51:28

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JMN-ECA500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.7473579[T] (500[MHz])
X_Acq_Duration    = 0.83361792[s]
X_Domain          = 13C
X_Freq            = 125.76529768[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.19959034[Hz]
X_Sweep           = 39.3081761[kHz]
X_Sweep_Clipped   = 31.44654088[kHz]
Irr_Domain        = Proton
Irr_Freq          = 500.15991521[MHz]
Irr_Offset        = 5.0[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay = 2[s]
Recvr_Gain        = 56
Temp_Get          = 22.1[dC]
X_90_Width        = 8.17[us]
X_Acq_Time        = 0.83361792[s]
X_Angle           = 30[deg]
X_Atn             = 6.5[dB]
X_Pulse           = 2.72333333[us]
Irr_Atn_Dec       = 22.66[dB]
Irr_Atn_Noe       = 22.66[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 92[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.83361792[s]
```



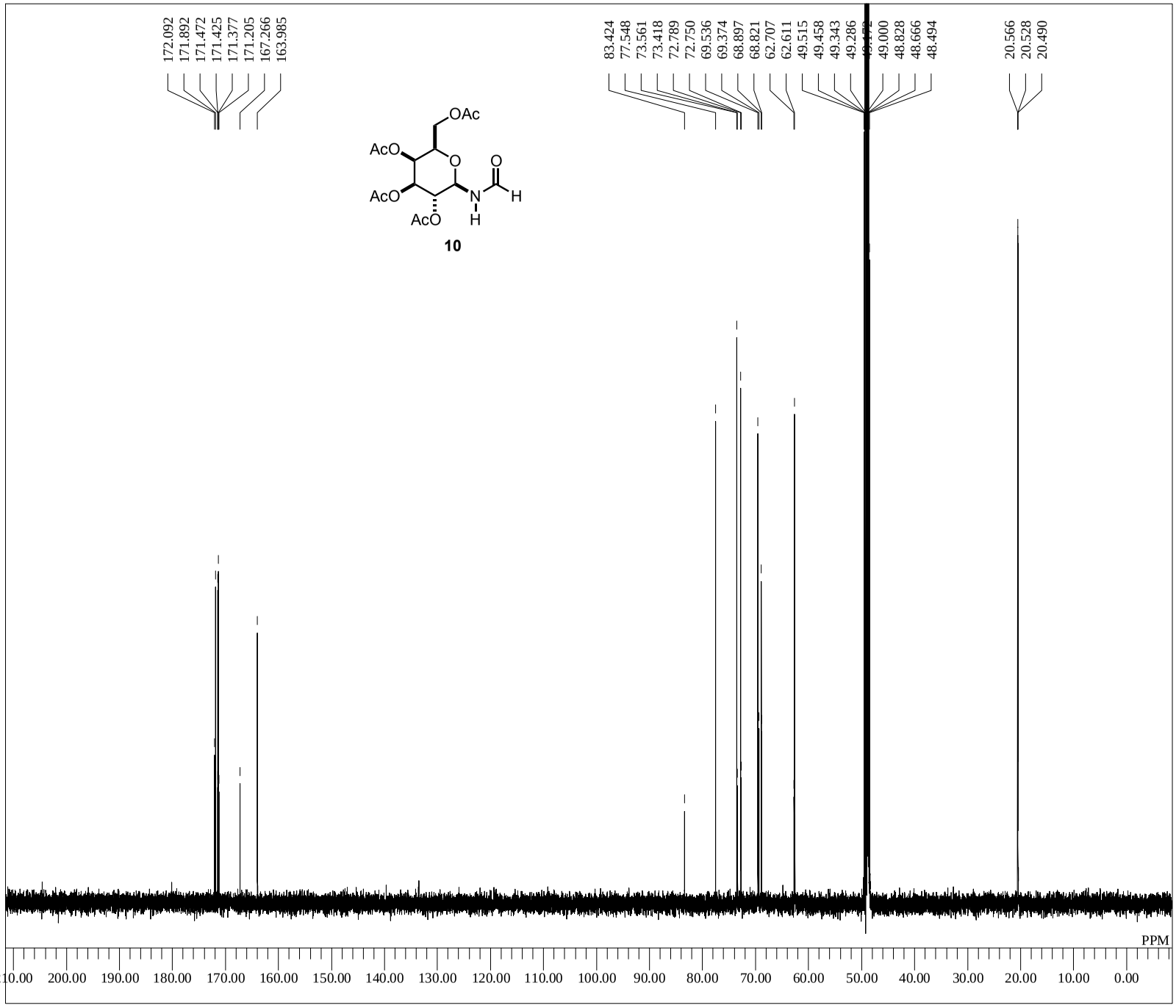

```
Filename          = ichikawa-1741-CD3OD_carbon
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = ichikawa-1741-CD3OD
Solvent           = METHANOL-D4
Creation_Time     =  4-JUN-2016 13:26:14
Revision_Time     = 13-OCT-2016 11:16:38
Current_Time      = 13-OCT-2016 11:17:51

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JMN-ECA500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.7473579[T] (500[MHz])
X_Acq_Duration    = 0.83361792[s]
X_Domain          = 13C
X_Freq            = 125.76529768[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.19959034[Hz]
X_Sweep           = 39.3081761[kHz]
X_Sweep_Clipped   = 31.44654088[kHz]
Irr_Domain        = Proton
Irr_Freq          = 500.15991521[MHz]
Irr_Offset        = 5.0[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay = 2[s]
Recvr_Gain        = 58
Temp_Get          = 22.1[dC]
X_90_Width        = 8.17[us]
X_Acq_Time        = 0.83361792[s]
X_Angle           = 30[deg]
X_Atn             = 6.5[dB]
X_Pulse           = 2.72333333[us]
Irr_Atn_Dec       = 22.66[dB]
Irr_Atn_Noe       = 22.66[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 92[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.83361792[s]
```



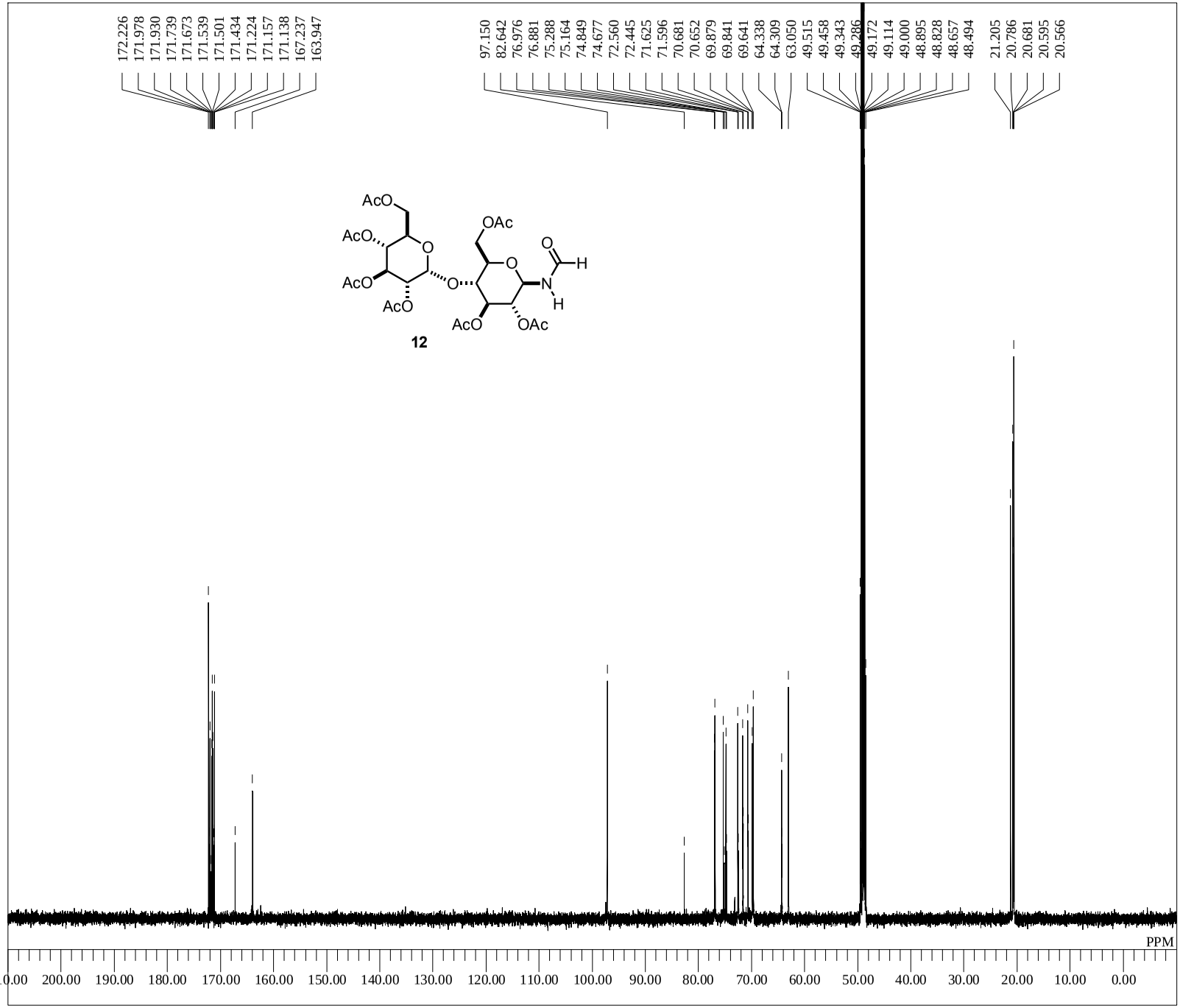

```
Filename          = MAT-ich-1040,1041_carbon-1
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = MAT-ich-1040,1041
Solvent           = CHLOROFORM-D
Creation_Time     = 12-OCT-2016 11:43:19
Revision_Time     = 17-OCT-2016 11:21:33
Current_Time      = 17-OCT-2016 11:35:39

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JMN-ECA500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.7473579[T] (500[MHz])
X_Acq_Duration    = 0.83361792[s]
X_Domain          = 13C
X_Freq            = 125.76529768[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.19959034[Hz]
X_Sweep           = 39.3081761[kHz]
X_Sweep_Clipped   = 31.44654088[kHz]
Irr_Domain        = Proton
Irr_Freq          = 500.15991521[MHz]
Irr_Offset        = 5.0[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 21.7[dC]
X_90_Width        = 8.17[us]
X_Acq_Time        = 0.83361792[s]
X_Angle           = 30[deg]
X_Atn             = 6.5[dB]
X_Pulse           = 2.72333333[us]
Irr_Atn_Dec       = 22.66[dB]
Irr_Atn_Noe       = 22.66[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 92[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.83361792[s]
```



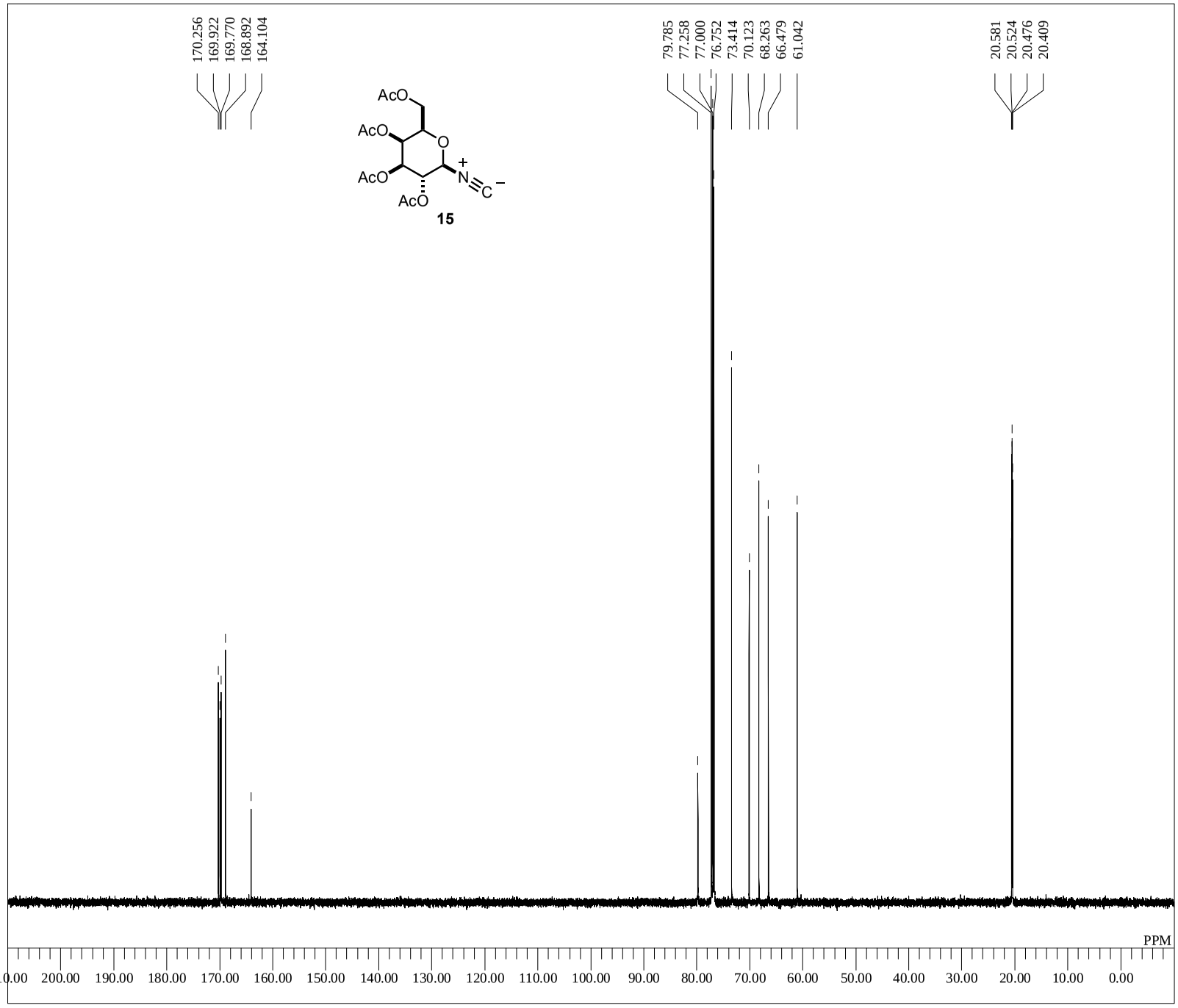

```
Filename          = MAT-ich-1040,1041_carbon-1
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = MAT-ich-1040,1041
Solvent           = CHLOROFORM-D
Creation_Time     = 12-OCT-2016 11:43:19
Revision_Time     = 17-OCT-2016 11:21:33
Current_Time      = 17-OCT-2016 11:38:14

Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JMN-ECA500
Spectrometer      = DELTA2_NMR

Field_Strength    = 11.7473579[T] (500[MHz])
X_Acq_Duration    = 0.83361792[s]
X_Domain          = 13C
X_Freq            = 125.76529768[MHz]
X_Offset          = 100[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.19959034[Hz]
X_Sweep           = 39.3081761[kHz]
X_Sweep_Clipped   = 31.44654088[kHz]
Irr_Domain        = Proton
Irr_Freq          = 500.15991521[MHz]
Irr_Offset        = 5.0[ppm]
Clipped           = FALSE
Scans             = 1024
Total_Scans       = 1024

Relaxation_Delay  = 2[s]
Recvr_Gain        = 50
Temp_Get          = 21.7[dC]
X_90_Width        = 8.17[us]
X_Acq_Time        = 0.83361792[s]
X_Angle           = 30[deg]
X_Atn             = 6.5[dB]
X_Pulse           = 2.72333333[us]
Irr_Atn_Dec       = 22.66[dB]
Irr_Atn_Noe       = 22.66[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 92[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.83361792[s]
```



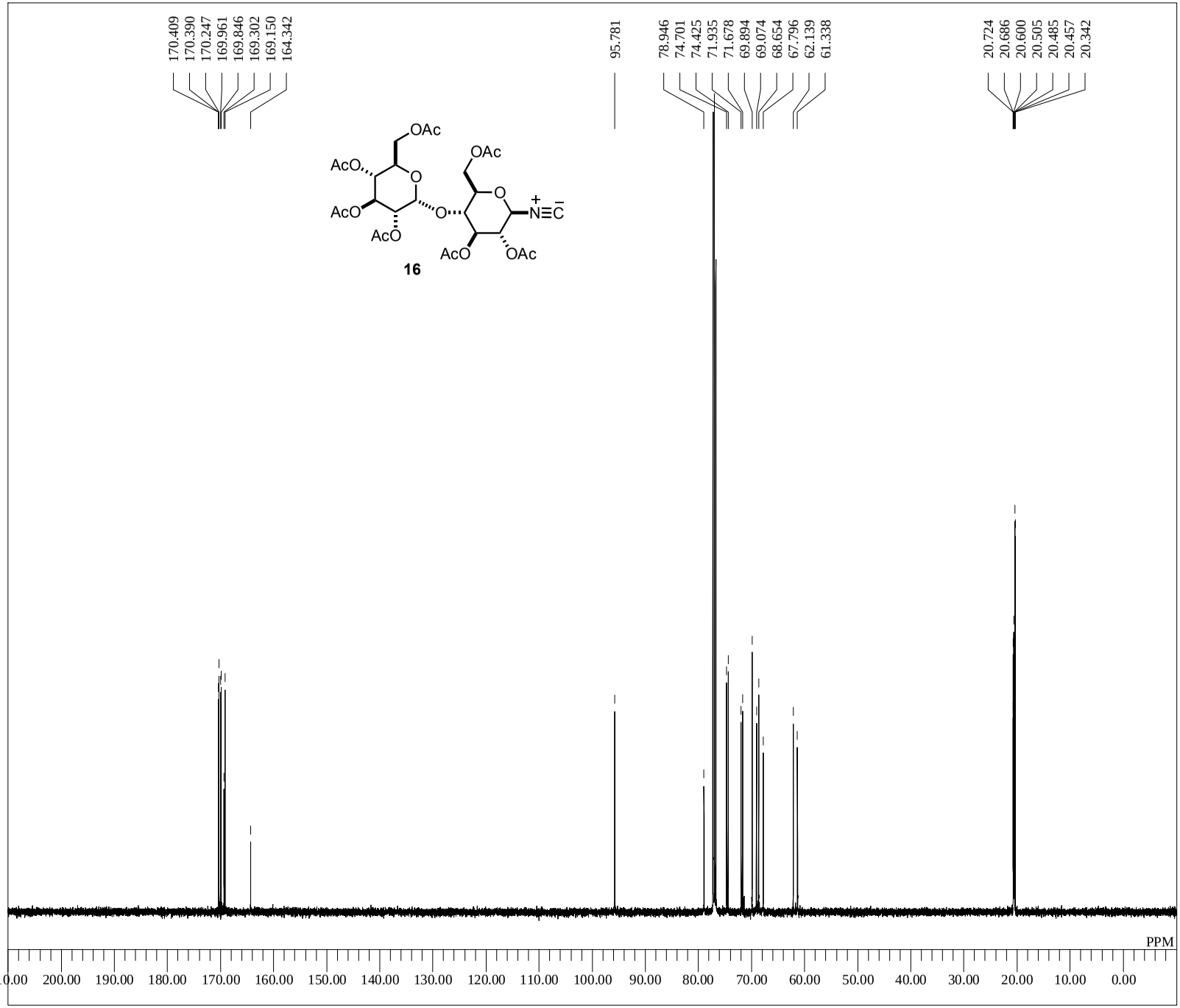
DFILE	ich-7-data2_carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2016-04-22 11:25:43
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	20.6 c
SLVNT	CD3OD
EXREF	49.00 ppm
BF	0.12 Hz
RGAIN	60



DFILE	MAT 2095 data CD3OD_carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2016-07-04 14:53:44
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	25.7 c
SLVNT	CD3OD
EXREF	49.00 ppm
BF	0.12 Hz
RGAIN	60



DFILE	MAE-1185-recrystallized4_carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2016-05-13 14:53:53
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	21.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	56



DFILE	MAT2101 data_carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2016-05-21 16:27:41
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32767
FREQU	39308.18 Hz
SCANS	1024
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	2.72 usec
IRNUC	1H
CTEMP	22.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.12 Hz
RGAIN	56