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Synthesis of Triazole-Linked Morpholino Oligonucleotides *via* Cu^I Catalysed Cycloaddition

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1. Chemistry General details:

NMR spectra were recorded using either a Bruker DPX-400 (400 MHz), Bruker AV-400 (400 MHz), Bruker AV3-400 (400 MHz) or Bruker DRX 500 (500 MHz) spectrometer at ambient temperature, with samples being prepared as dilute solutions in the stated solvent. Chemical shifts are quoted in parts per million (ppm) downfield of tetramethylsilane, with the spectra being referenced to residual solvent. The multiplicity of each signal is designated by the following abbreviations: s (singlet), d (doublet), t (triplet), q (quartet), b (broad), m (multiplet) and app (apparent), and all coupling constants (*J*) are quoted in Hertz. Assignments were made on the basis of chemical shift, COSY, HMBC, HSQC and/or DEPT experiments. Mass spectra were recorded on a Bruker MicroTOF system, using electrospray (ESI) techniques.

Flash chromatography was carried on Merck silica gel 60 (230–400 mesh ASTM), and all of the solvents used were of analytical grade; petroleum ether refers to light petroleum, bp 40–60 °C. Reactions were monitored by thin layer chromatography (TLC) using aluminium plates precoated with Merck silica gel 60F254; they were visualised using ultraviolet light ($\lambda_{\text{max}} = 254 \text{ nm}$), and then stained using acid cerium molybdate solution or basic potassium permanganate solution.

ELECTRONIC SUPPORTING INFORMATION

Unless stated otherwise, reactions requiring anhydrous conditions were conducted in an inert atmosphere of argon in flame-dried or oven-dried apparatus. Dry organic solvents were routinely stored under a nitrogen or argon atmosphere. Dry and degassed Toluene, THF and MeCN were obtained by passing through anhydrous alumina columns using an Innovative Technology Inc. PS-400-7 solvent purification system. Dry DCM was distilled from CaH₂. All solvents were removed by concentration *in vacuo* at approx. 20 mm Hg using a Büchi rotary evaporator. Traces of solvent were then removed at approx. 0.2 mm Hg on a vacuum line

2. General procedures for oligonucleotides synthesis and analysis

Columns (SynBaseTM CPG), standard DNA and RNA-phosphoramidites and reagents for the synthesizer were purchased from Link Technologies Ltd (Scotland, UK). MeNH₂ solution (33 wt.% in ethanol) was obtained from Fluka, NEt₃.3HF, N-methylpyrrolidinone (NMP) and 3-hydroxypicolinic acid (HPA) were purchased from Aldrich, illustra NapTM5 columns were obtained from GE Healthcare Europe GmbH and Oligonucleotide Purification Cartridges (OPCTM) were purchased from Applied Biosystems UK. Water used for oligonucleotides analysis and purification was either purified by Milli-Q ultra pure water system (Millipore) or commercial nuclease-free water. MeCN HPLC grade was used for oligos synthesis and purification.

2.1. Synthesis of oligonucleotides

Oligonucleotides were synthesised on ABI 3400 DNA synthesiser (0.2 μ mol scale) following standard solid-phase chemistry. The following standard solutions were used;

- Deblock Mix (3% trichloroacetic acid in dichloromethane)
- BTT Activator (0.3M 5-benzylthio-1-H-tetrazole in acetonitrile)
- Oxidiser (0.02M iodine in THF/pyridine/water = 7:2:1)
- Cap Mix A (THF/pyridine/acetic anhydride = 8:1:1)

ELECTRONIC SUPPORTING INFORMATION

- Cap Mix B (10% methylimidazole in THF)

2.1.1 Synthesis and post-synthesis treatment of DNA oligonucleotides:

Unmodified parts of DNA oligonucleotides were synthesized using the standard (0.2 μ mol scale protocol provided by the manufacturer. Modified dinucleotides were coupled with a increased coupling time of 600 s and using 4 coupling cycles.

Once synthesised, the DNA oligonucleotides were transferred to DNase free falcons and cleaved manually from the resin and by adding 33% ammonia solution (2 mL). The falcons were placed in Aluminium heating block and kept at 60 °C for 18 h for the purpose of the deprotection of cyanoethyl and removal of base-protecting groups.

The oligonucleotides were purified using the standard protocol supplied with OPC[®] (Applied Biosystems, Foster City, CA, USA). Stock solutions used in purification protocol were: MeCN HPLC Grade, TEAA (2 M), NH₄OH (1.5 M), 3% TFA, Milli-Q water and 20% MeCN. Oligos samples were diluted with Milli-Q water (2 mL) prior to OPC[®] and loaded twice on the cartridge. Detritylation occurred using 3% TFA, DMTr-off samples were eluted upon addition of 20% MeCN and freeze-dried prior to further purification / analysis.

2.1.2 Synthesis and post-synthesis treatment of RNA oligonucleotides:

RNA oligonucleotides were synthesized using 0.2 μ M scale protocol with a 600 s coupling step for each nucleotide. The post-synthesis treatment can be categorized into the following steps:

- Cleavage and deprotection of cyanoethyl and base protecting groups:

The polymer-bound oligoribonucleotide was transferred from the synthesis column to a DNase free falcons and suspended in MeNH₂ solution (1 mL). The mixture was heated to 65°C for 10 min, cooled to room temperature (water/ice bath) and centrifuged for 1 min (10 000 g). The

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supernatant was separated from the CPG beads, the beads were washed with RNase free water (2 x 0.25 mL), all supernatants were combined and then freeze dried.

- Deprotection of 2'-O-TBDMS groups and Detritylation:

The oligoribonucleotide were resuspended in anhydrous $\text{NEt}_3 \cdot 3\text{HF} / \text{NEt}_3 / \text{NMP}$ solution (250 μL of a solution of 1.5 mL NMP, 750 μL NEt_3 and 1.0 mL $\text{NEt}_3 \cdot 3\text{HF}$), heated to 65°C for 1.5 h, cooled to room temperature and quenched with 3M NaOAc solution (25 μL).

- Butanol precipitation:

n-BuOH (1 mL) was added to the mixture, which was then thoroughly mixed, cooled to -78 °C for 2 h to encourage further precipitation and centrifuged for 30 min (4 °C, 13 000 g). The supernatant was removed, the pellet washed with 70% EtOH (2 × 500 μL) and then freeze dried.

2.1.3 Oligonucleotides desalting and determination of the concentrations

The dry RNA and DNA oligonucleotide precipitate was dissolved in RNase free water (1 mL) and desalted using a NapTM-5 column following the standard protocol supplied with the NapTM-5 column. The resulting solution were stored in the freezer and used as stock solutions for mass spec and T_m analysis. Determination of the concentrations of oligos was carried out using a Thermo Scientific NanoDropTM 1000 Spectrophotometer.

| Identifier | Sequence 5'-3' | Stock concentrations |
|---------------------------------|--------------------------|----------------------|
| DNA Target 18 | GCTGCAAACGTCG | 110 μM |
| RNA Target 19 | GCUGCAAACGUCC | 65 μM |
| Unmodified DNA 20 | CGACGTTTGCAGC | 135 μM |
| ^{T_L} MO 21 | CGACG T* TTGCAGC | 63 μM |
| ^{T_L} DNA 22 | CGACG T^A TTGCAGC | 105 μM |

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2.4. HPLC analysis of oligos.

DMTr-off oligos purity was assessed by analytical HPLC using an Agilent 1100 series HPLC and a Phenomenex Clarity 3 μ Oligo-RP 50 \times 4.6 mm column (Macclesfield, UK) with a flow rate of 1 mL/min following the elution gradient below

| Time (min) | Buffer A % | Buffer B % |
|------------|------------|------------|
| 0 | 95 | 5 |
| 5 | 95 | 5 |
| 20 | 80 | 20 |
| 30 | 80 | 20 |
| 32 | 95 | 5 |
| 35 | 95 | 5 |

Table S1. Elution method of HPLC for the purification of oligos

Buffer A: TEAA (100 mM in Water)

Buffer B: MeCN HPLC grade.

Data was collected and analysed using Agilent Technologies, Chemstation for LC software.

| Identifier | Sequence 5'-3' | Retention time (min) |
|----------------------|-----------------------------|----------------------|
| DNA Target 18 | GCTGCAAACGTCG | 7.76 |
| RNA Target 19 | GCUGCAAACGUCC | 17.08 |
| Unmodified DNA 20 | CGACGTTTGCAGC | 7.86 |
| ¹⁸ MO 21 | CGACG ^{T*} TTGCAGC | 8.10 |
| ¹⁸ DNA 22 | CGACG ^{T^} TTGCAGC | 8.32 |

Table S2 HPLC Retention times

2.5. Mass Spec analysis of oligonucleotides.

Masses of oligomers were obtained on a Thermo Scientific: LTQ FT Ultra Mass Spectrometer in negative mode. Mass ions were found for the M-6H adducts.

ELECTRONIC SUPPORTING INFORMATION

| Identifier | Sequence 5'-3' | MW | |
|----------------------|----------------|------------|---------|
| | | Calculated | Found |
| DNA Target 18 | GCTGCAAACGTCG | 3953.55 | 3953.73 |
| RNA Target 19 | GCUGCAAACGUCG | 4133.49 | 4133.63 |
| Unmodified DNA 20 | CGACGTTTGCAGC | 3944.53 | 3944.72 |
| ^{TL} MO 21 | CGACGT*TTGCAGC | 3944.64 | 3944.80 |
| ^{TL} DNA 22 | CGACGT^TTGCAGC | 3945.63 | 3945.78 |

Table S3. Mass spec data

2.6. UV-Melting experiments

The hybrids were formed at final concentration of 3 μ M by dissolving the lyophilised oligonucleotides in 10 mM phosphate buffer with 200 mM NaCl at the reported pH (7.0). The samples were annealed by placing the tubes in an aluminium heating block at 90 °C for 10 min, the samples were remove from the heating block and allow to cool to room temperature (approx. 45 min). The samples were stored at 4 °C prior to T_m analysis. The melting curve of the duplex was obtained by following the temperature dependencies of the absorption at 260 nm in a micro T_m cell with a 1 cm path length. The temperature was varied between 20 and 85 °C at 1 °C/min, both heating and cooling data were recorded.

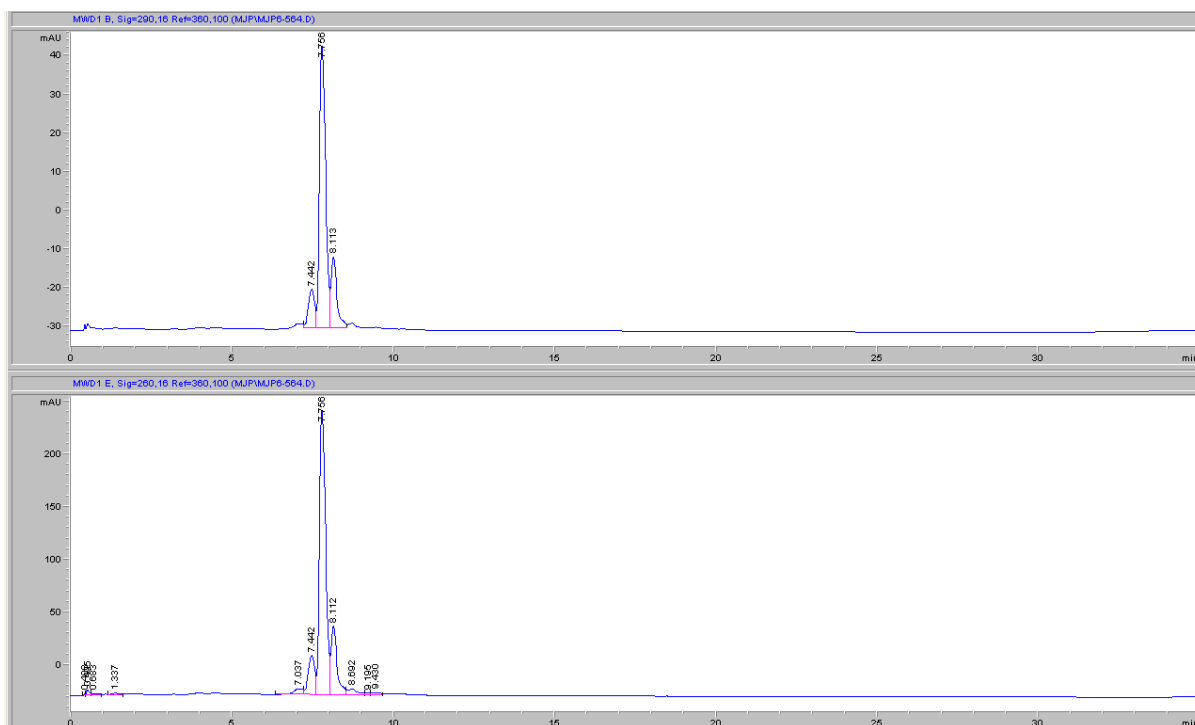
The data was normalised to % denaturation following [equation 4](#) and plotted against temperature to obtain the melt curves.^{15a,b}

$$\% \text{ Denaturation} = (A^0 - A^i / A^{\text{max}} - A^i) \times 100 \quad \text{equation (4)}$$

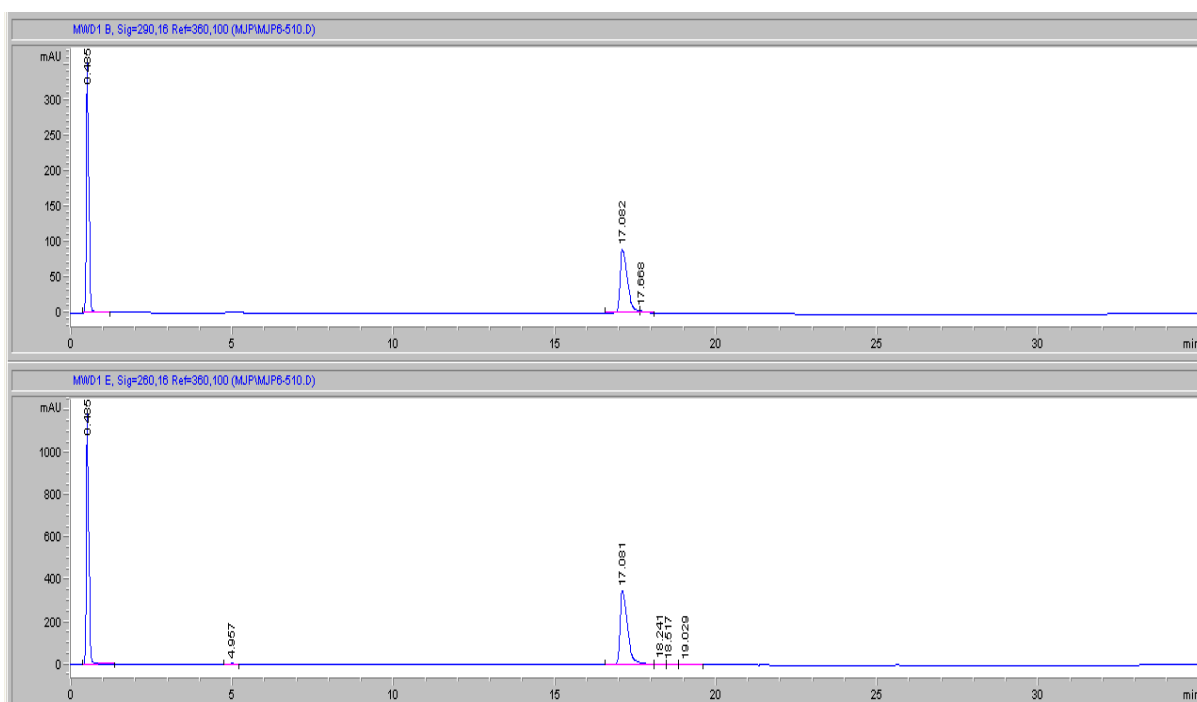
Where Abs⁰: observed absorbance, Absⁱ: initial absorbance at 25 °C and Abs^{max}: maximum absorbance at 85 °C. The values were combined to obtain a melting curve and the T_m was determined as the midpoint of the plot between the two states of the system.

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3. HPLC chromatograms of oligonucleotides

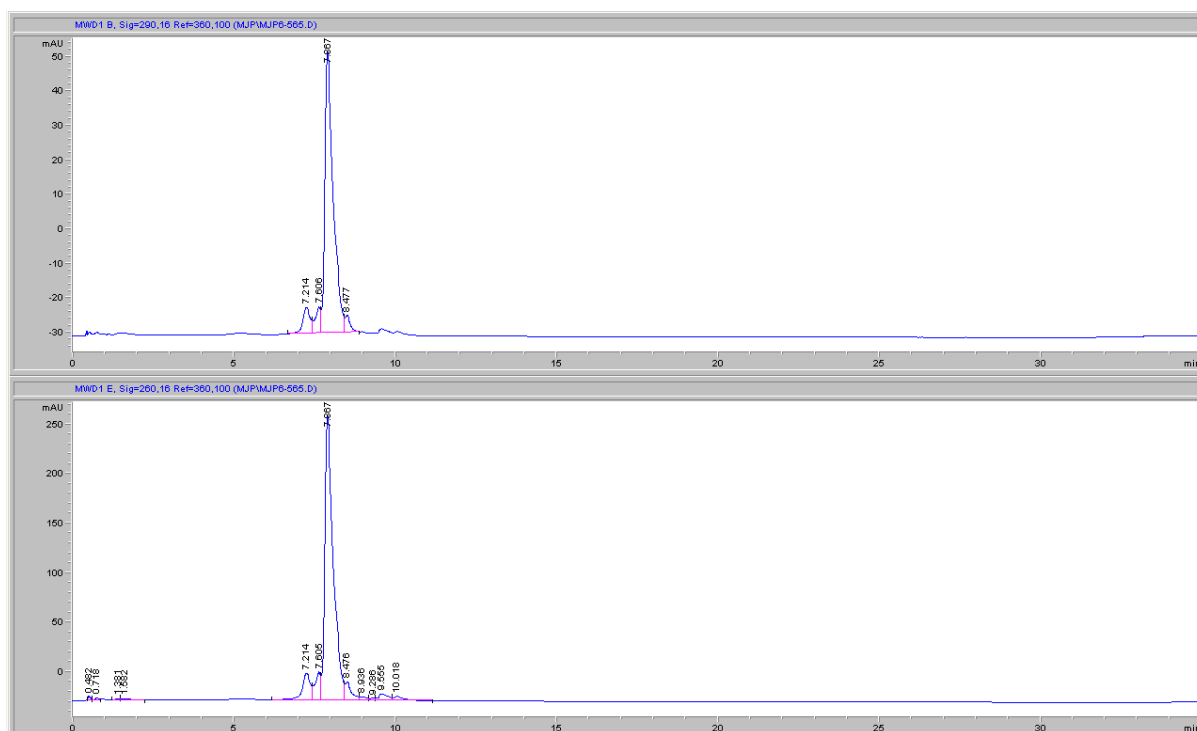


HPLC chromatogram of DNA Target 18 (GCTGCAAACGTCG)

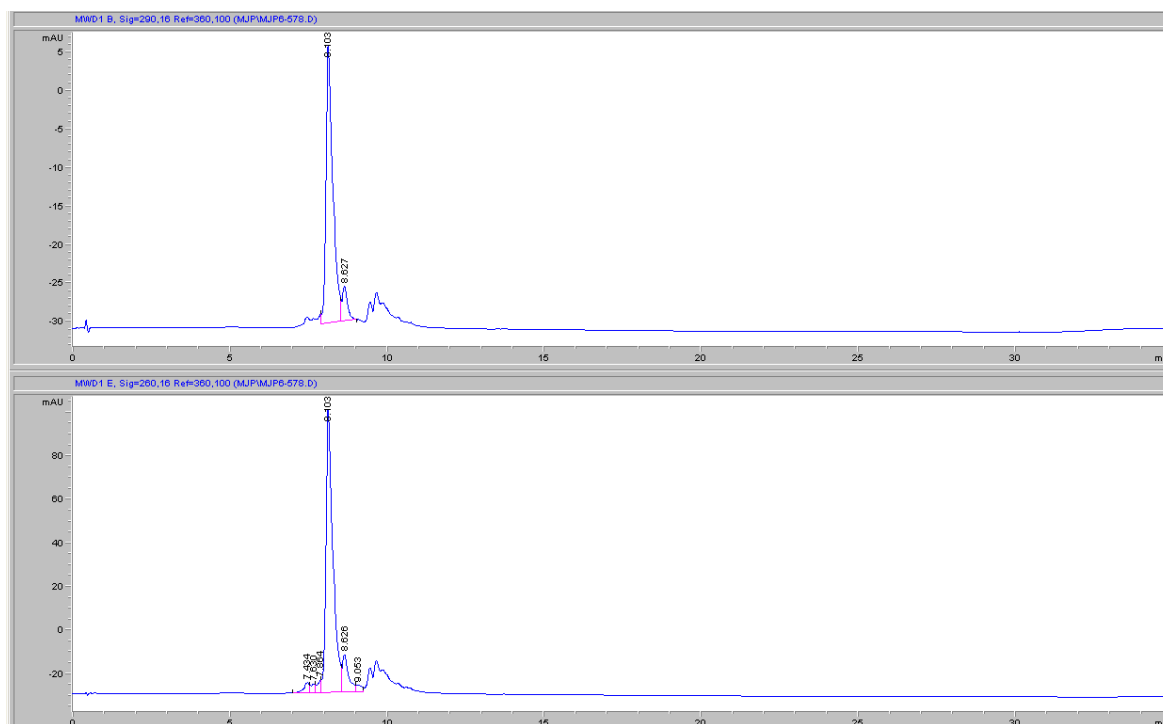


HPLC chromatogram of RNA Target 19 (GCUGCAAACGUCG)

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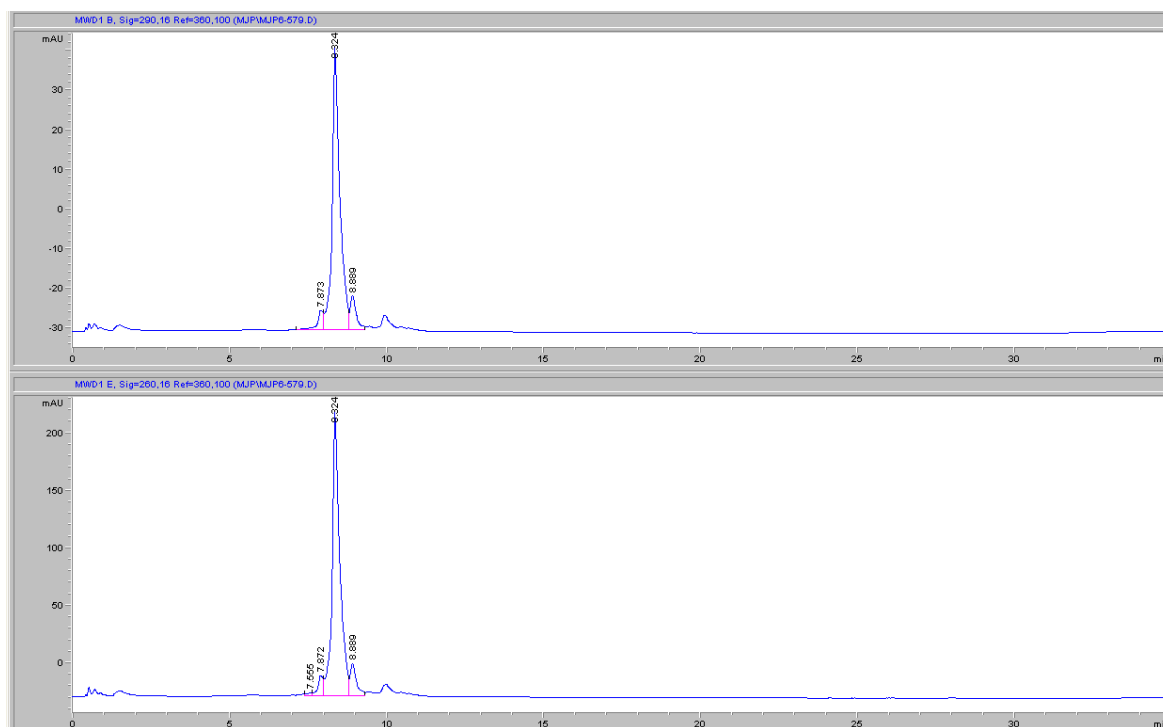


HPLC chromatogram of **Unmodified DNA 20 (CGACGTTTGCAGC)**



HPLC chromatogram of **^{TL}MO 21 (CGACG**T***TTGCAGC)**

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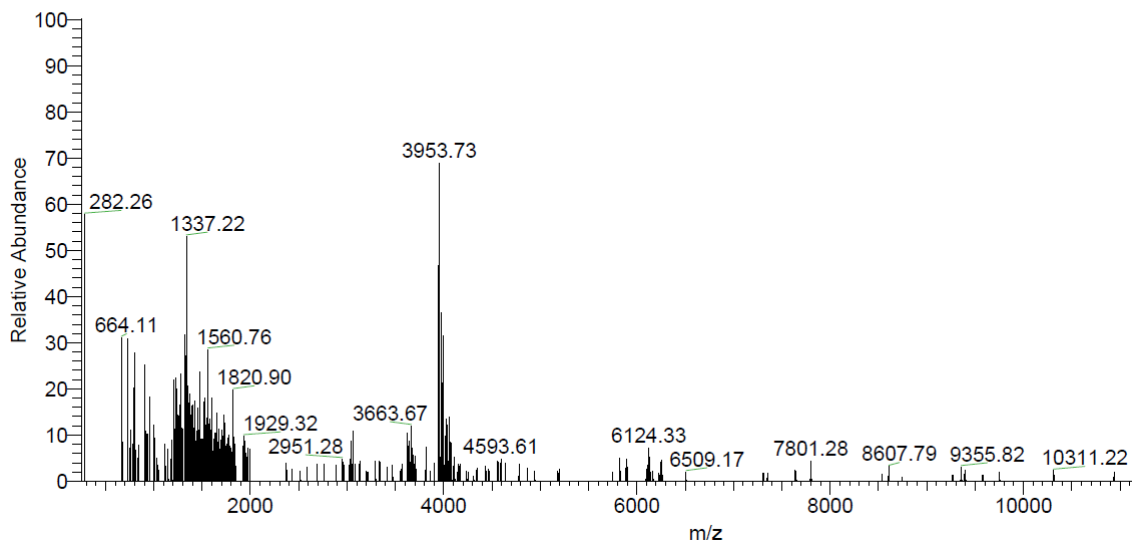


HPLC chromatogram of ^{TL}DNA 22 (CGACGT^{TL}TTGCAGC)

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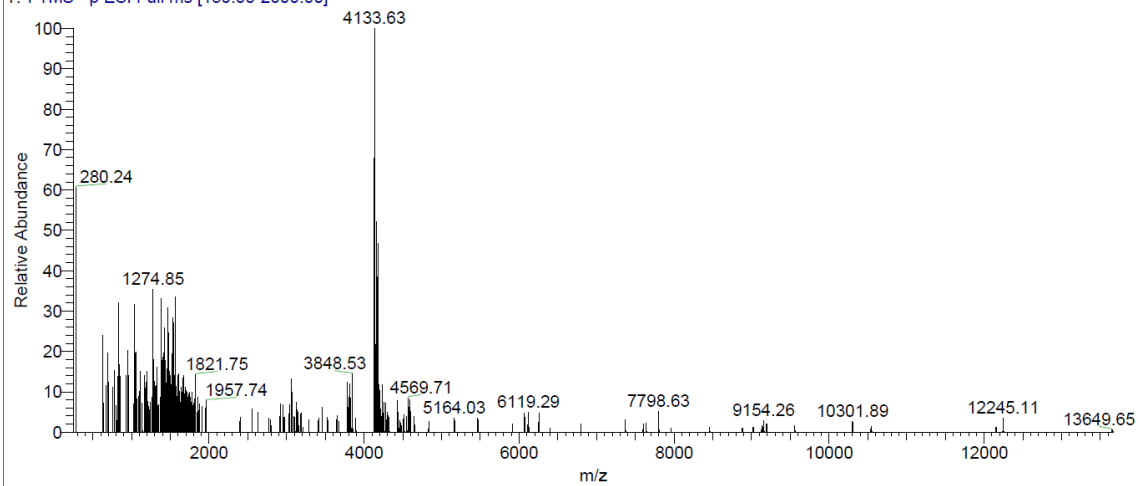
4. Mass Spectra of oligonucleotides

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T: FTMS - p ESI Full ms [150.00-2000.00]



DNA Target 18 (GCTGCAAACGTCG) – found 3953.73; ($M - 6H^+$) requires 3953.55

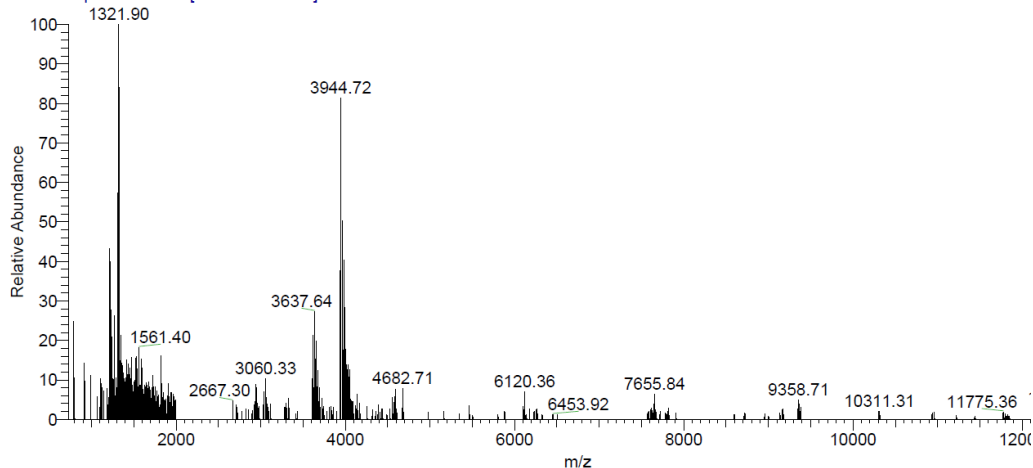
MJP6-510-RNA-r3_XT_00001_M_#1 RT: 1.00 AV: 1 NL: 2.21E4
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RNA Target 19 (GCUGCAAACGUCG) – found 4133.63; ($M - 6H^+$) requires 4133.49

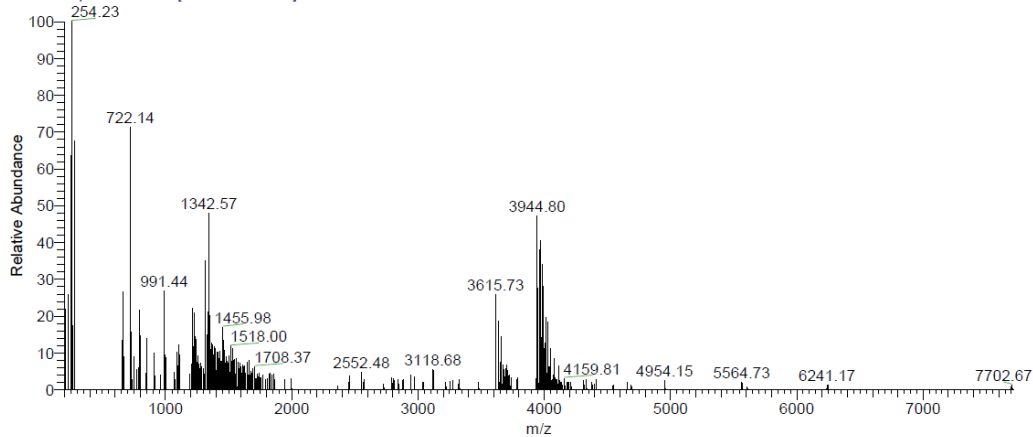
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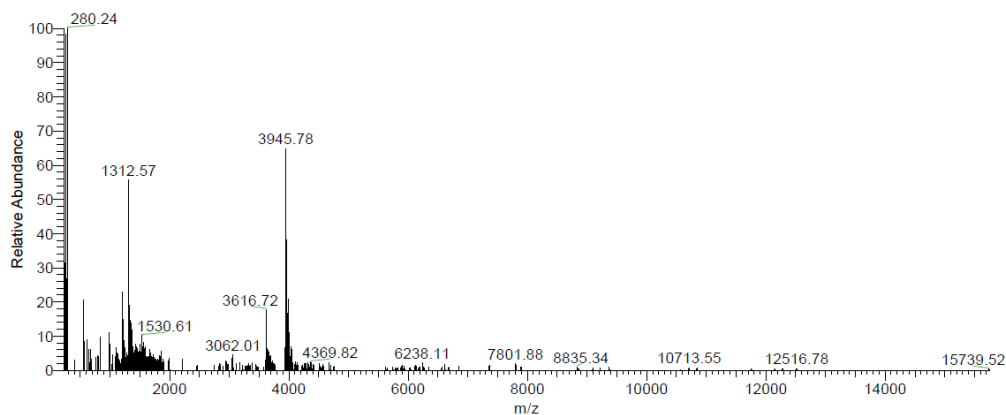
Unmodified DNA 20 (CGACGTTTGCAGC) – found 3944.72; $(M - 6H^+)$ requires 3944.53

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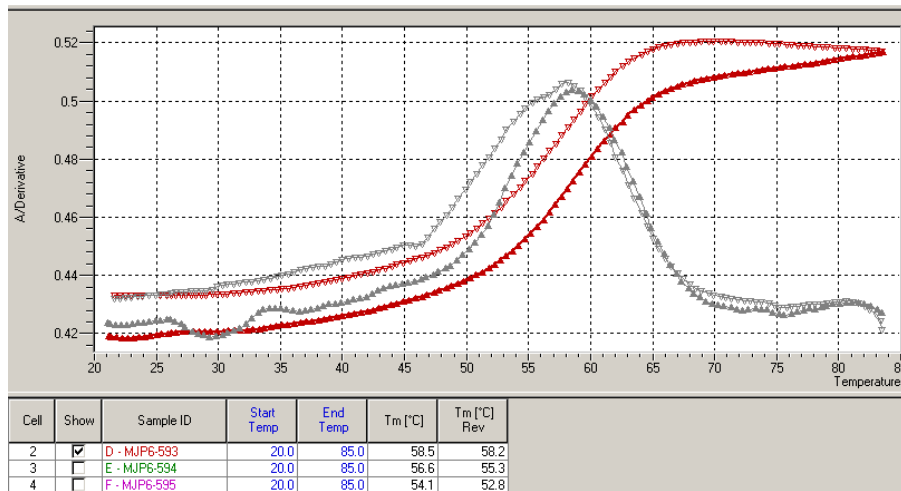
TLMO 21 (CGACGT*TTGCAGC) – found 3944.80; $(M - 6H^+)$ requires 3944.64

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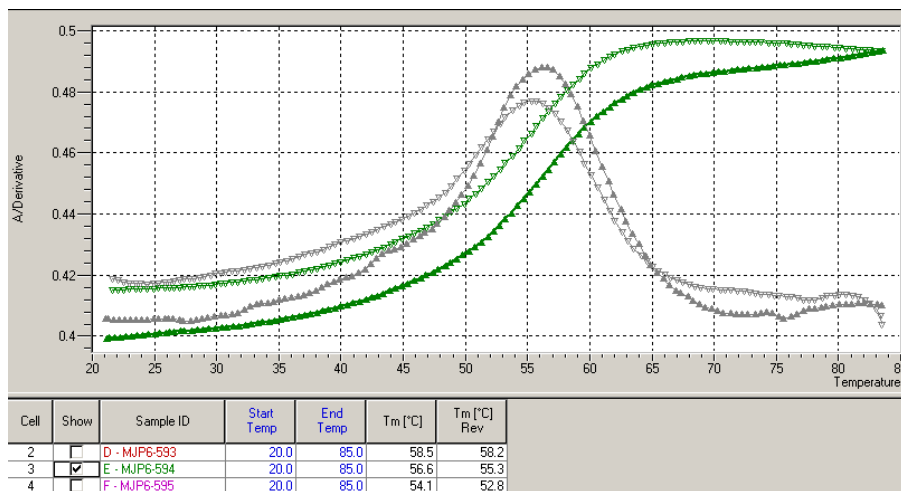


TLDNA 22 (CGACGT^ATTGCAGC) – found 3945.78; $(M - 6H^+)$ requires 3945.63

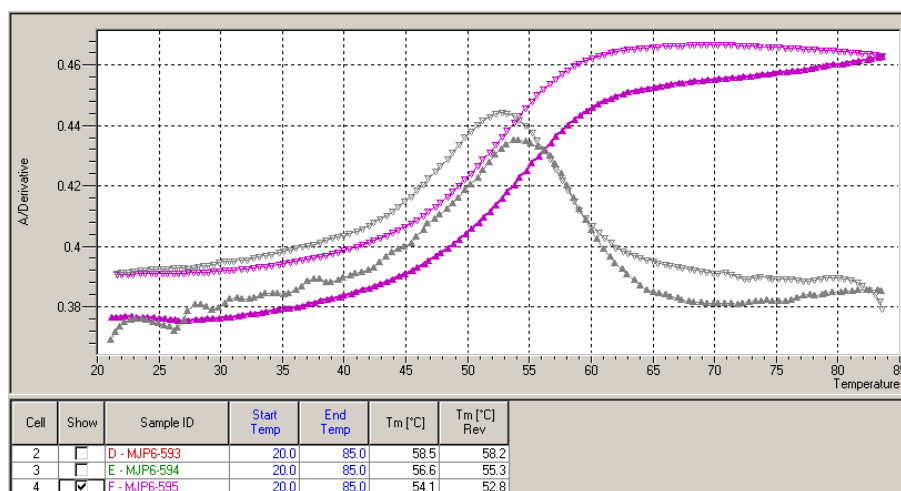
T_m data for RNA duplex at pH 7.0



Duplex of RNA 19 and unmodified DNA 20

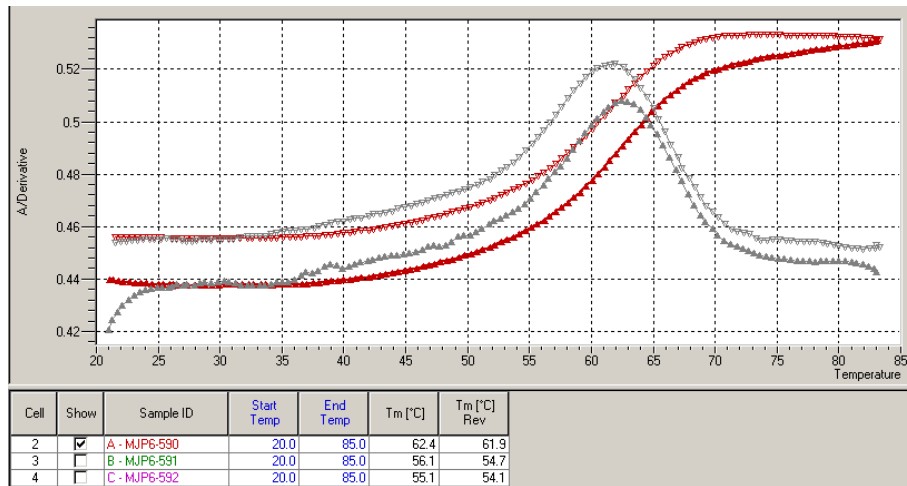


Duplex of RNA 19 and ^{TL}MO 21

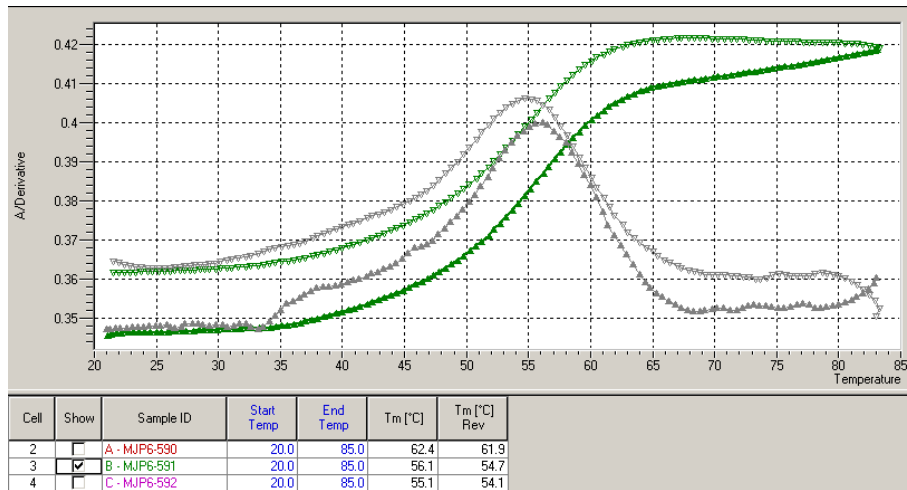


Duplex of RNA 19 and ^{TL}DNA 22

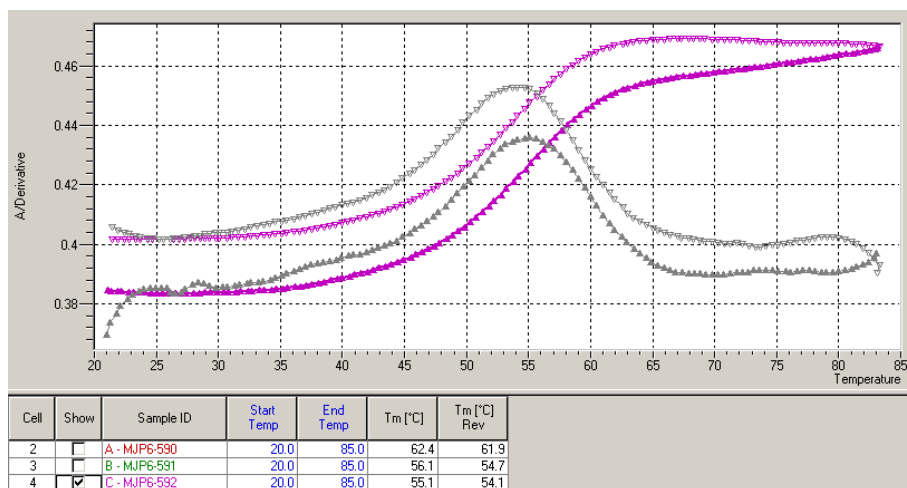
T_m data for DNA duplex at pH 7.0



Duplex of DNA 18 and unmodified DNA 20



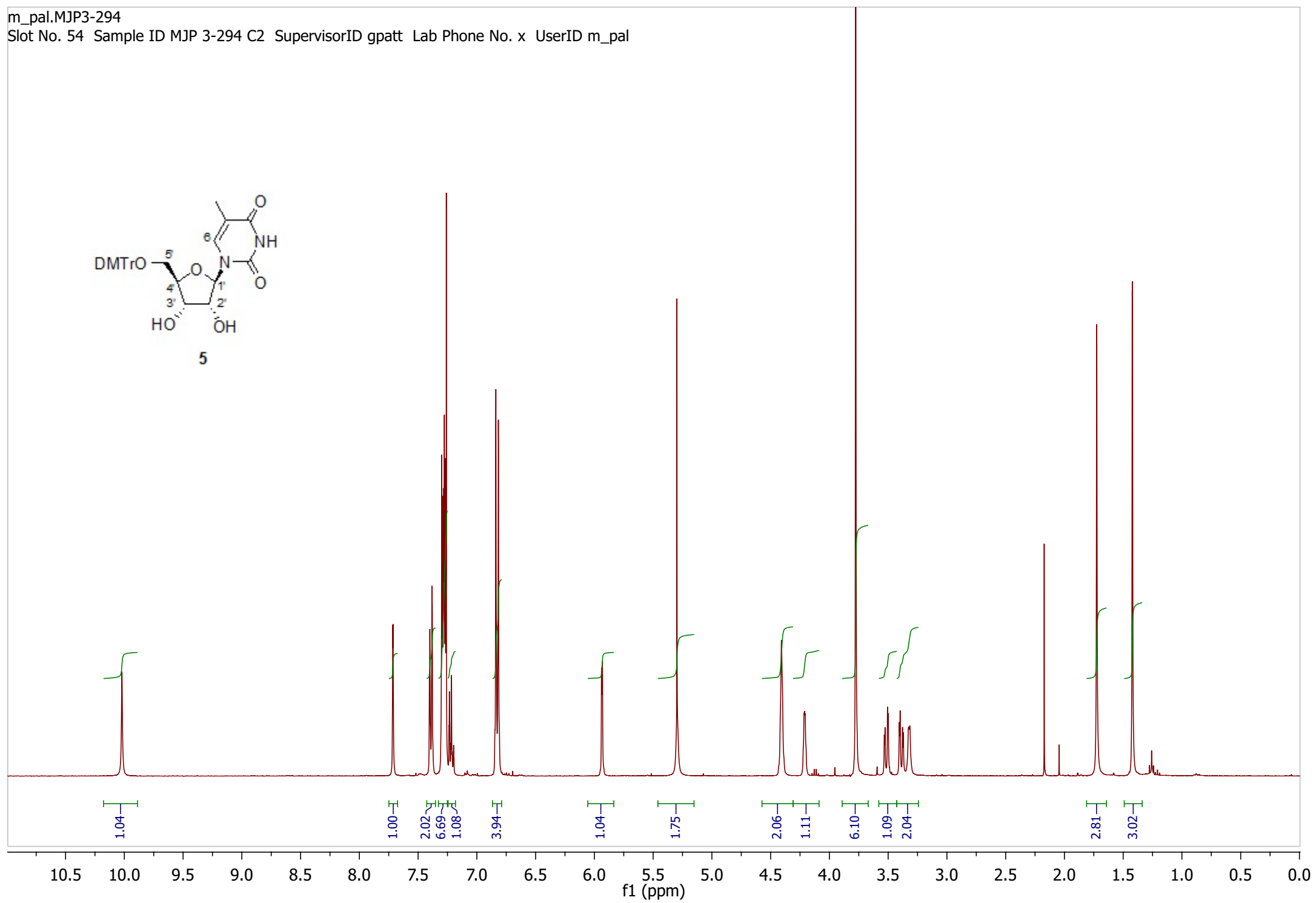
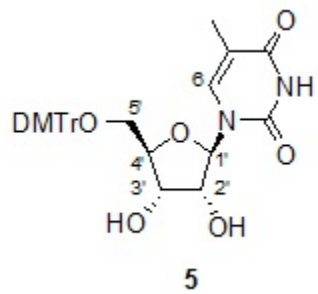
Duplex of DNA 18 and ^{TL}MO DNA 21



Duplex of DNA 19 and ^{TL}DNA 22

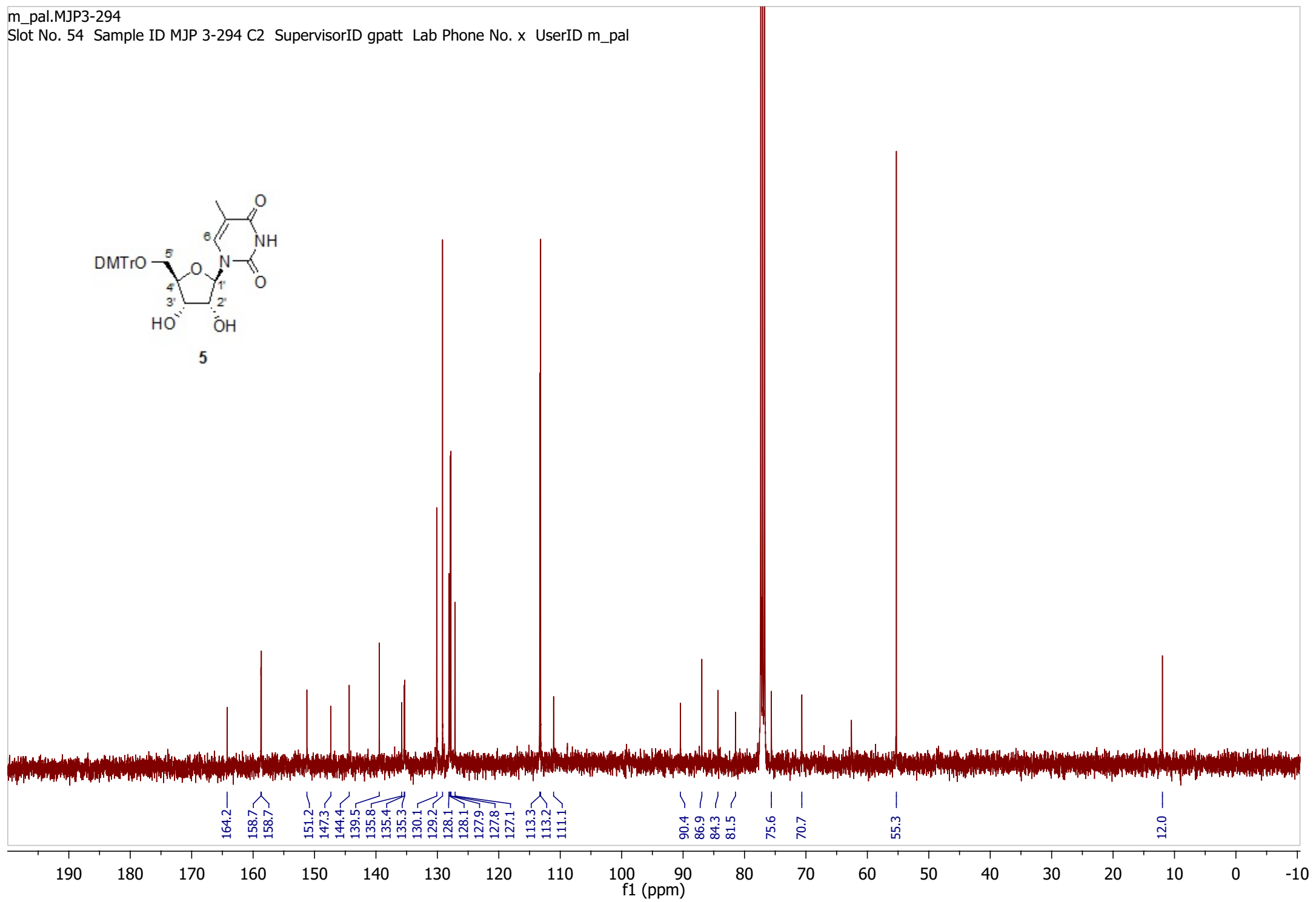
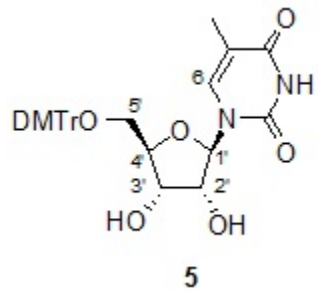
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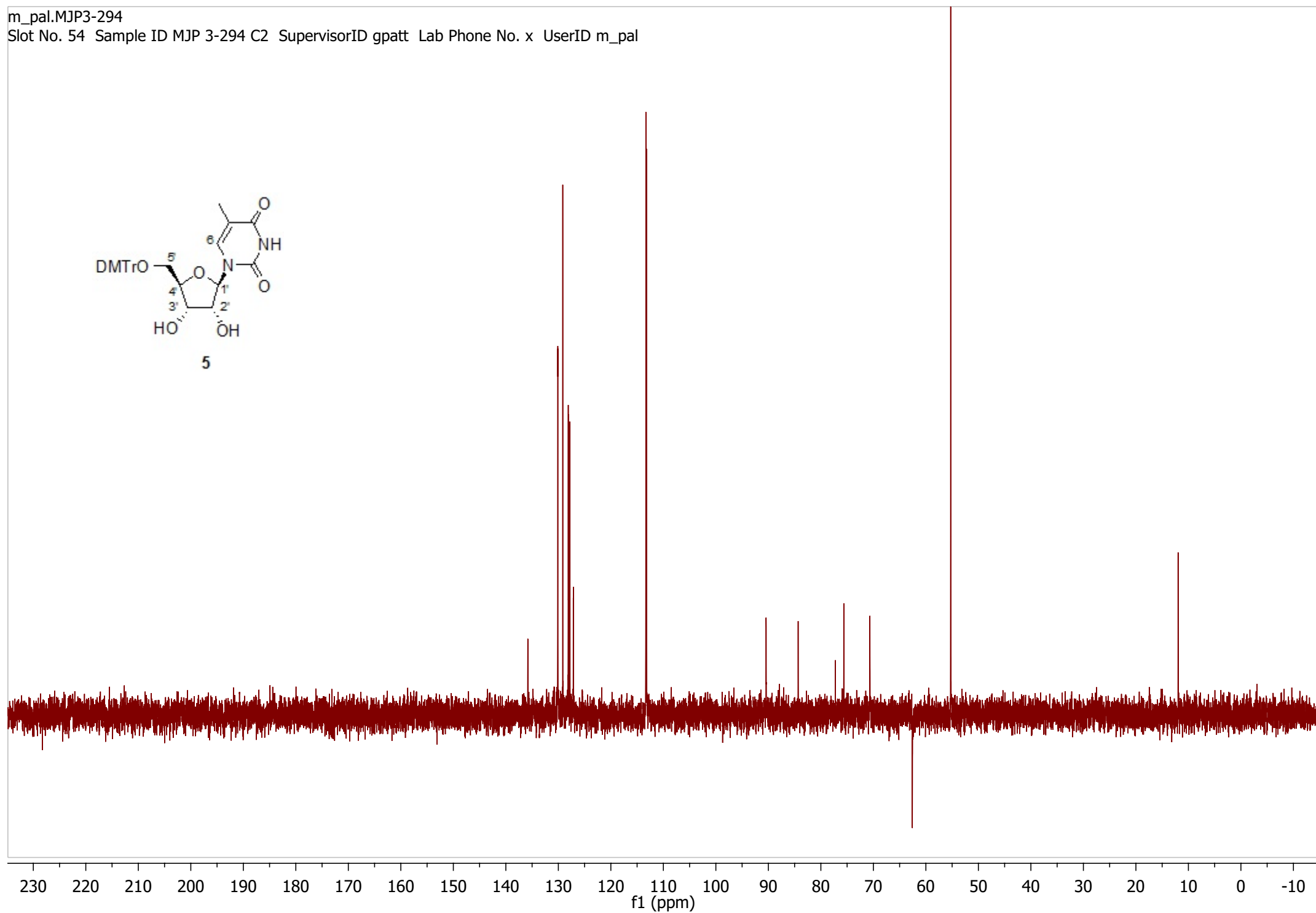
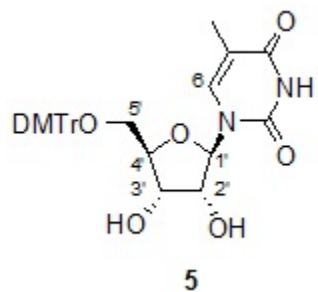
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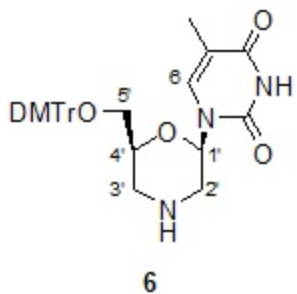
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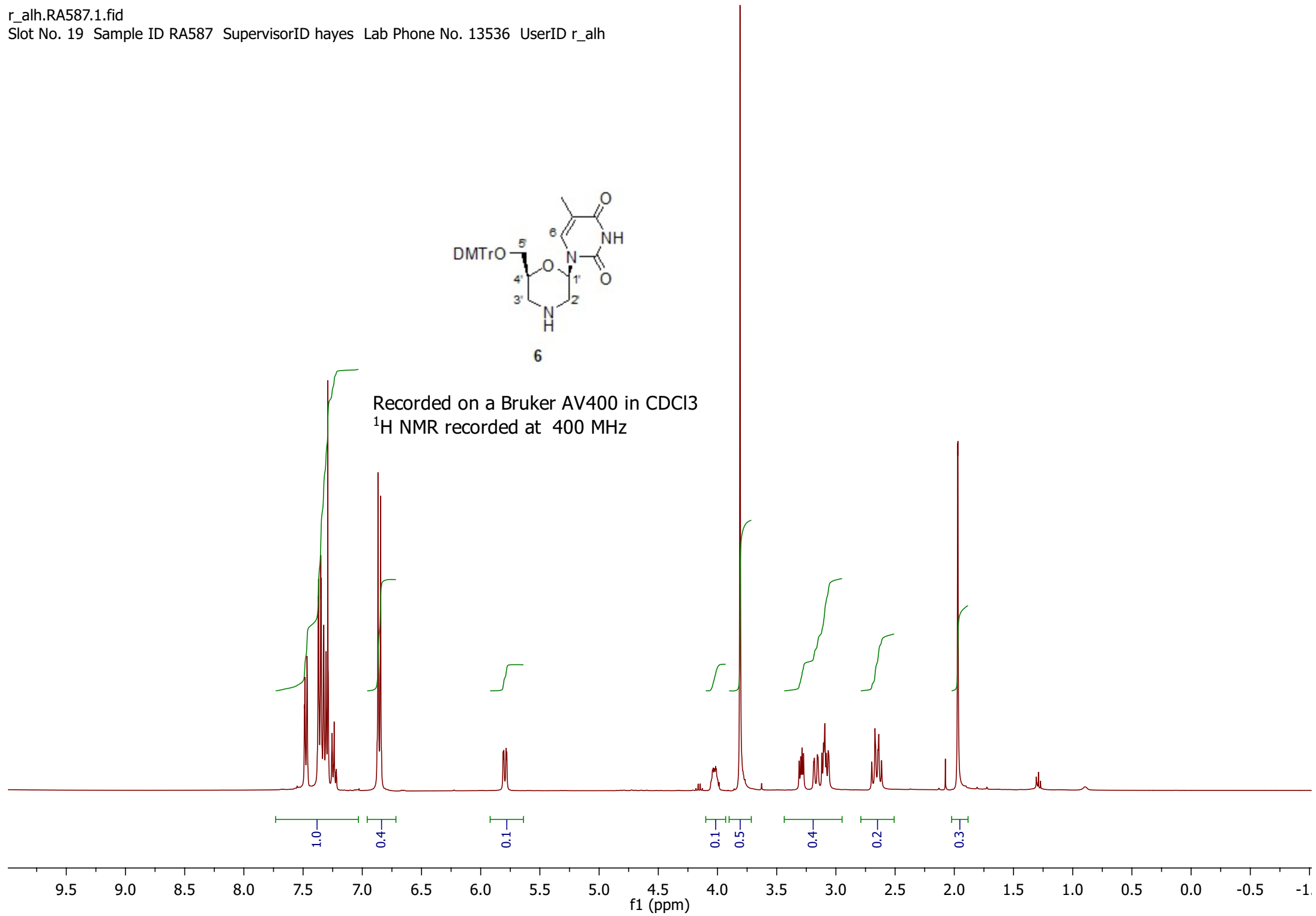
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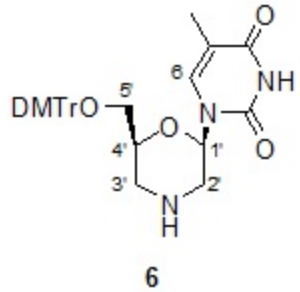
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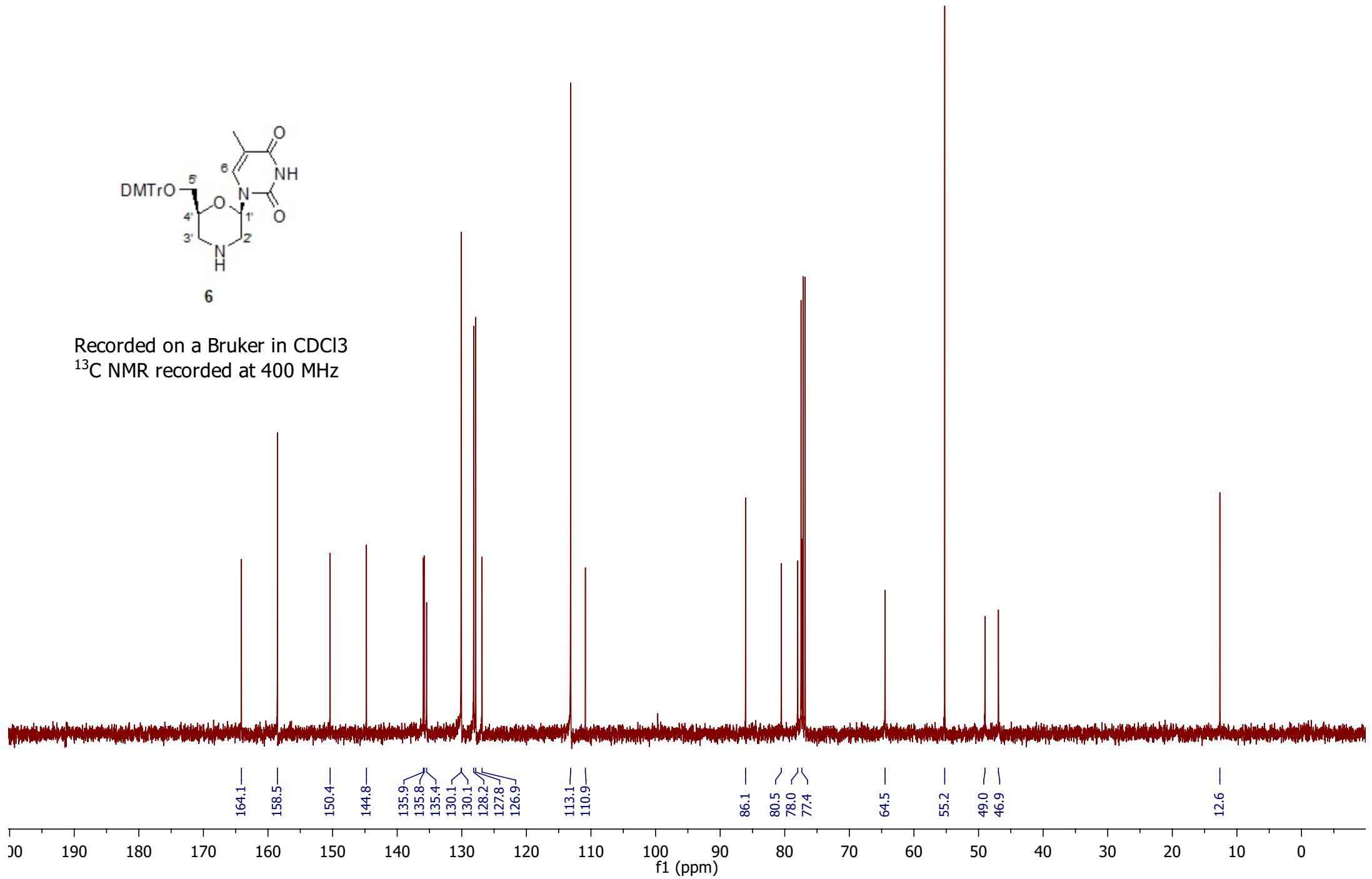


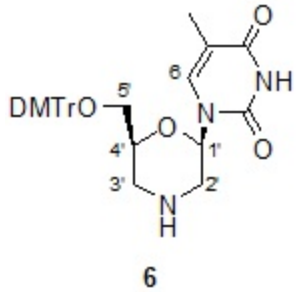
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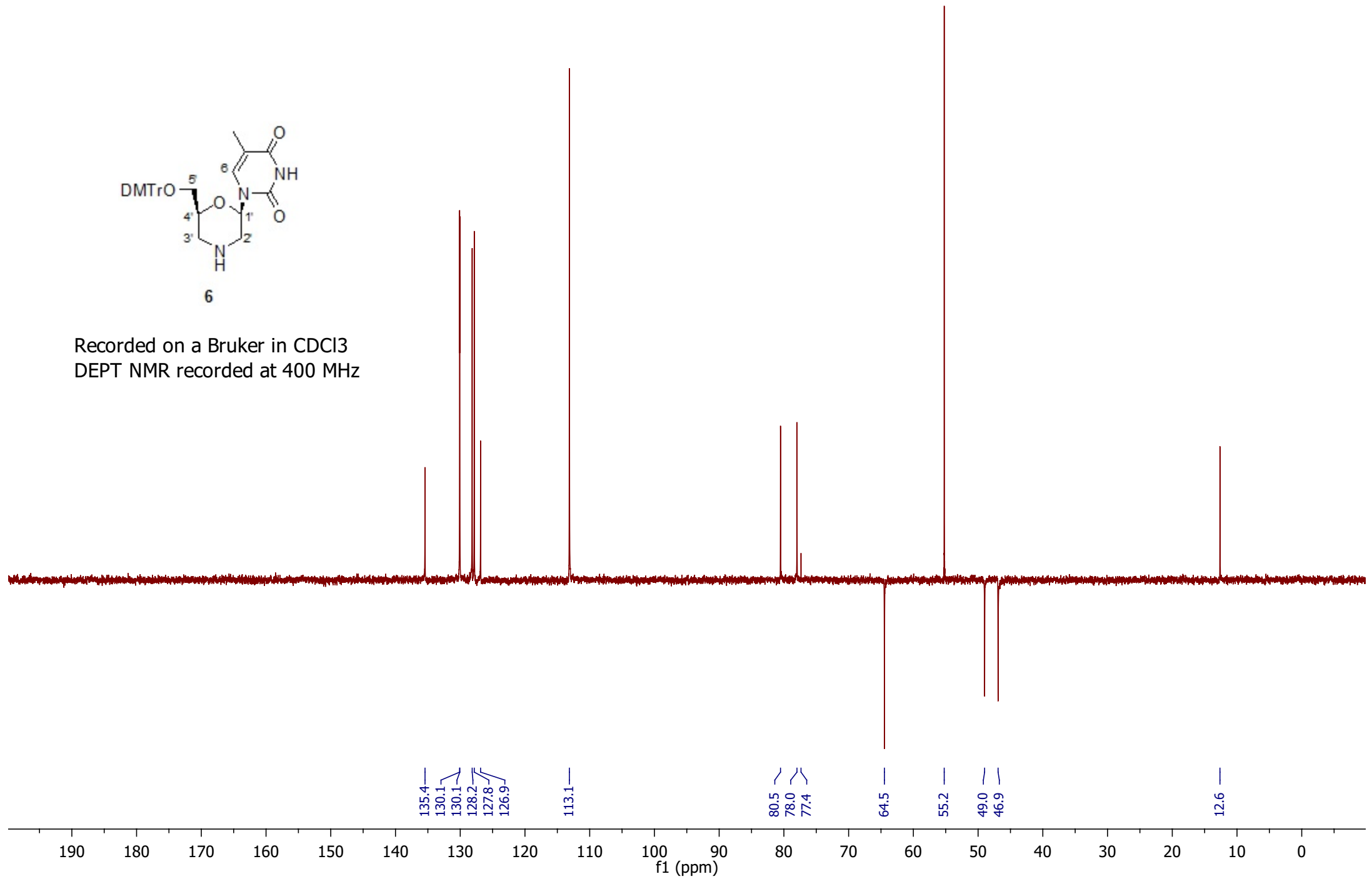


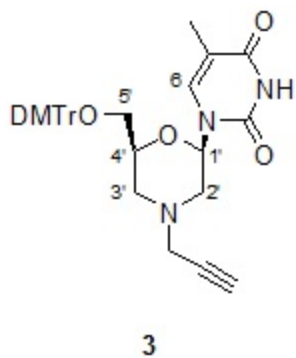
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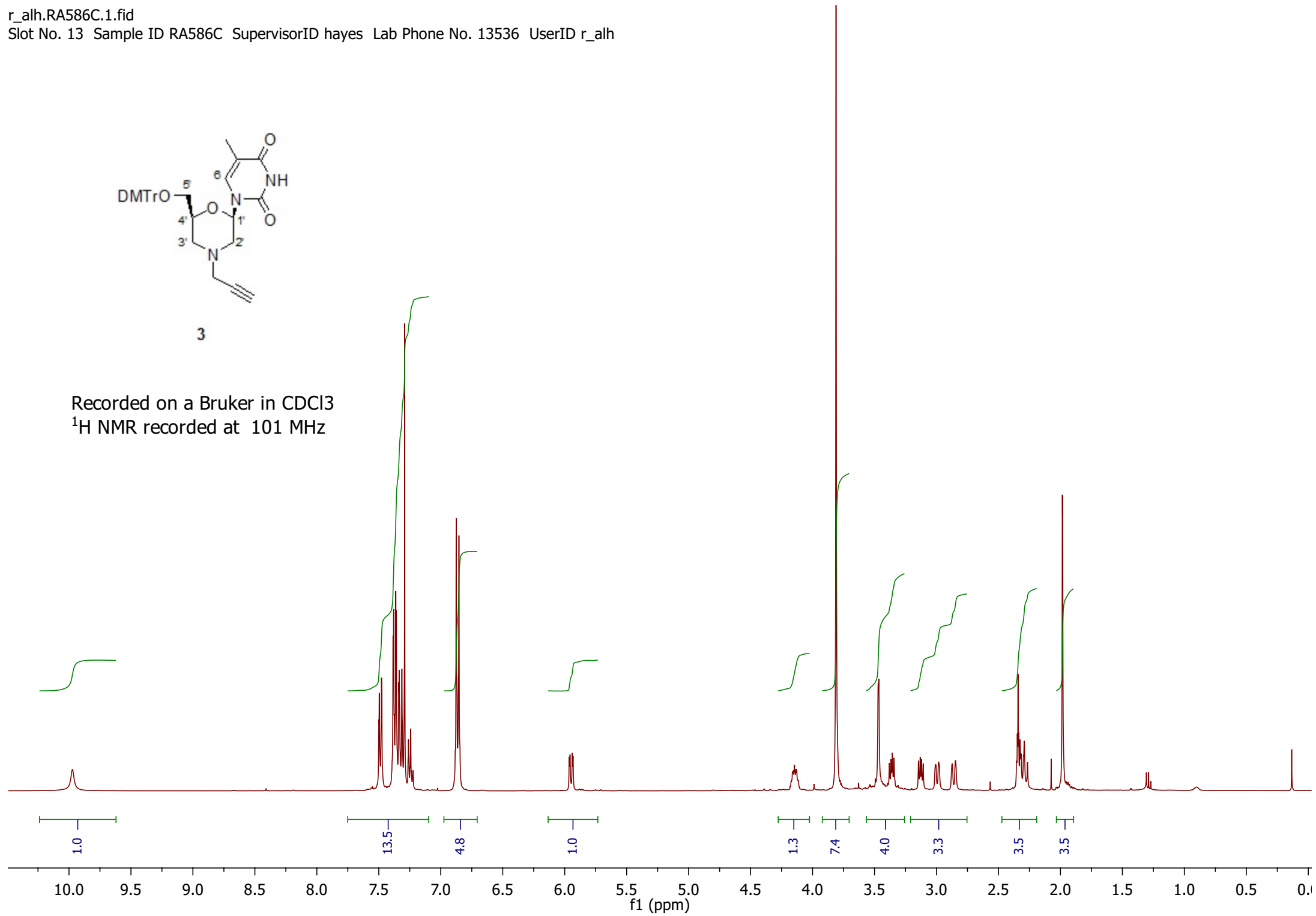


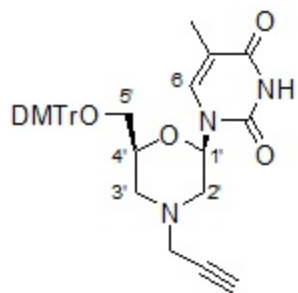
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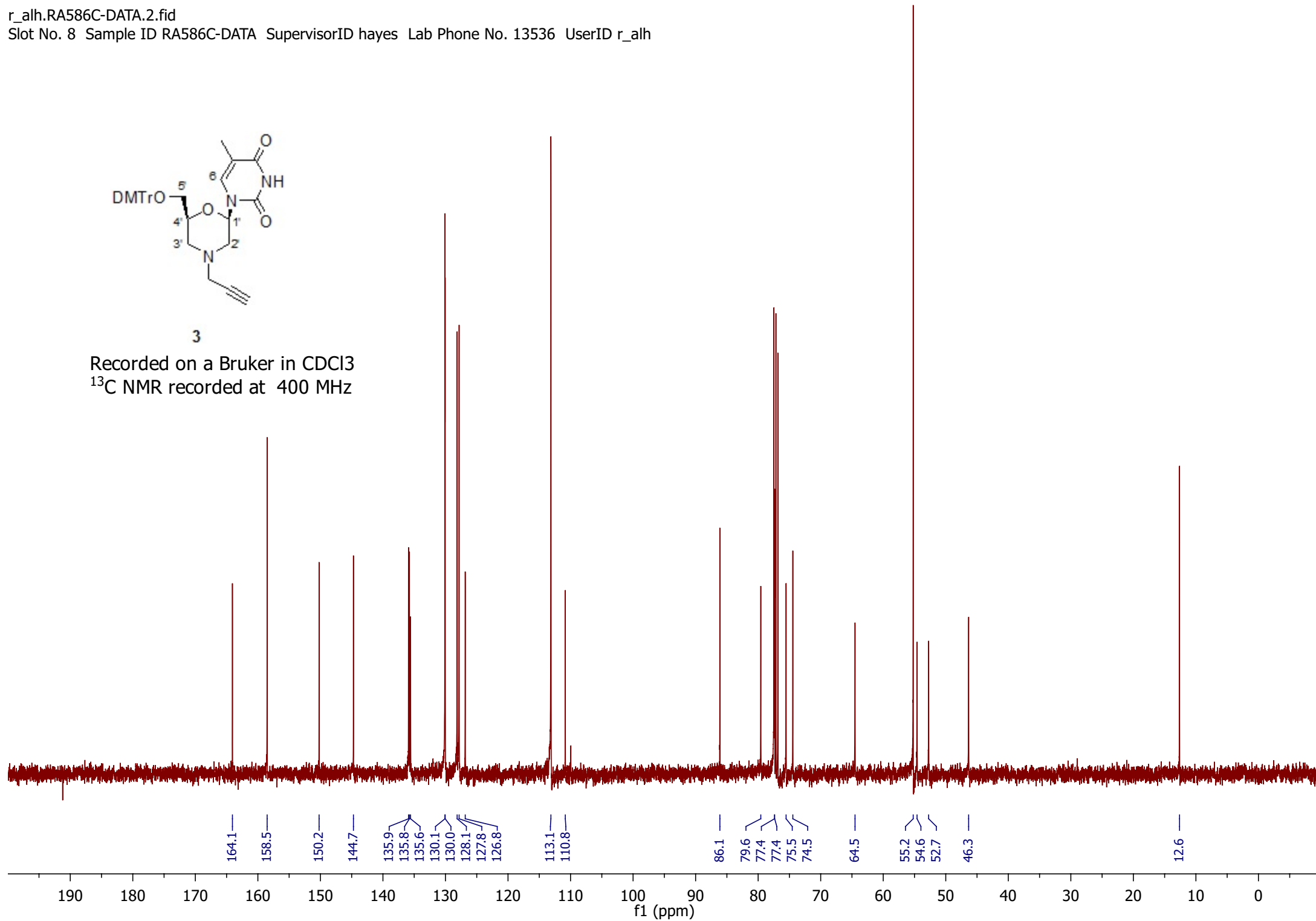
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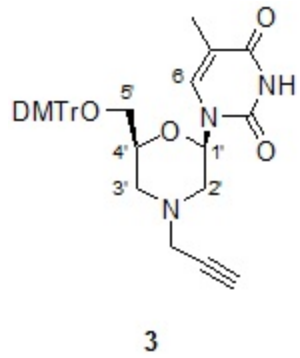




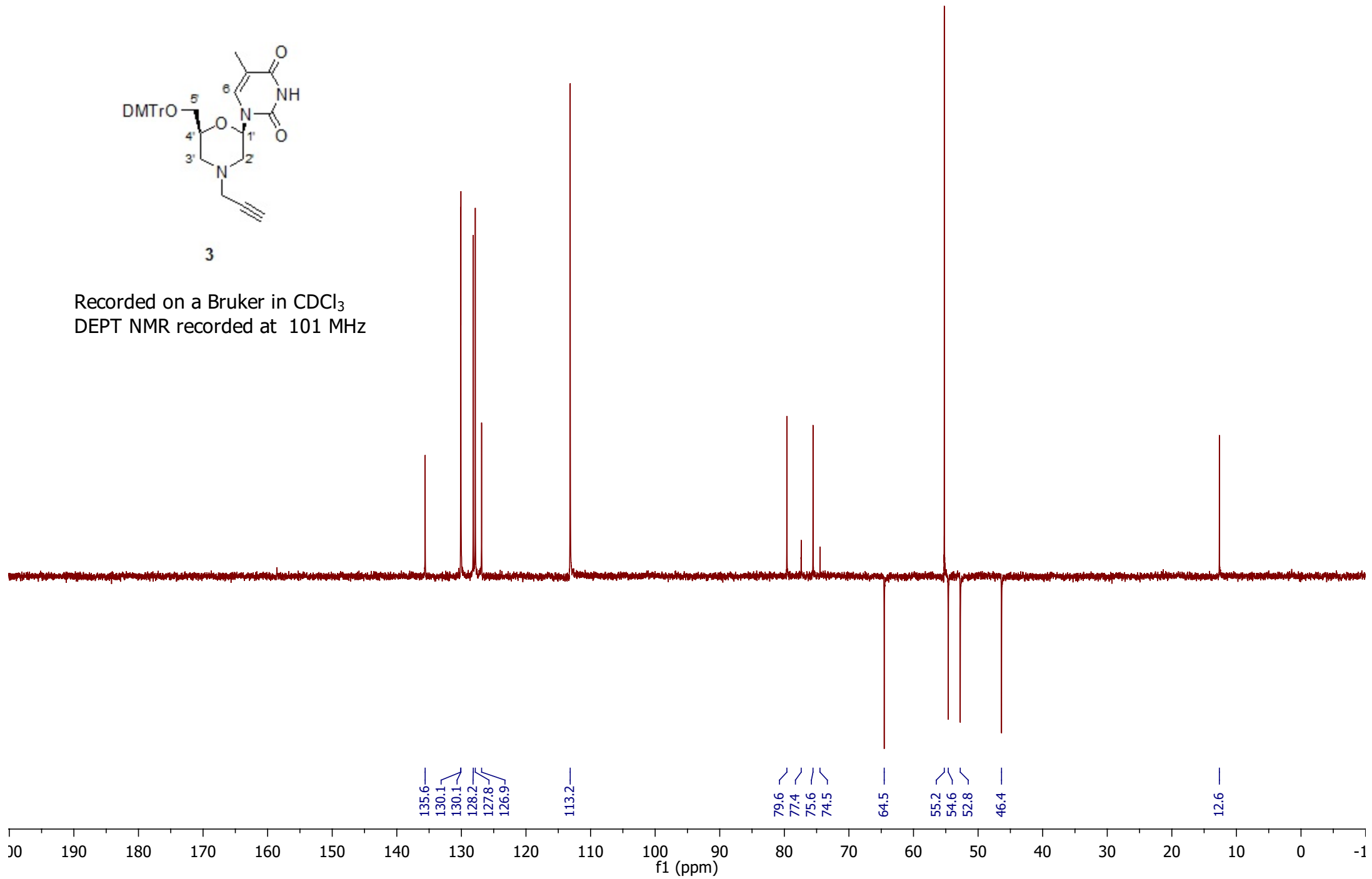
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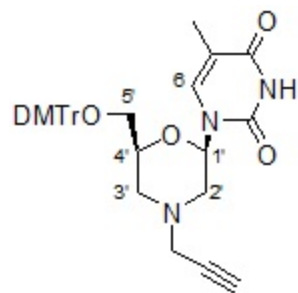


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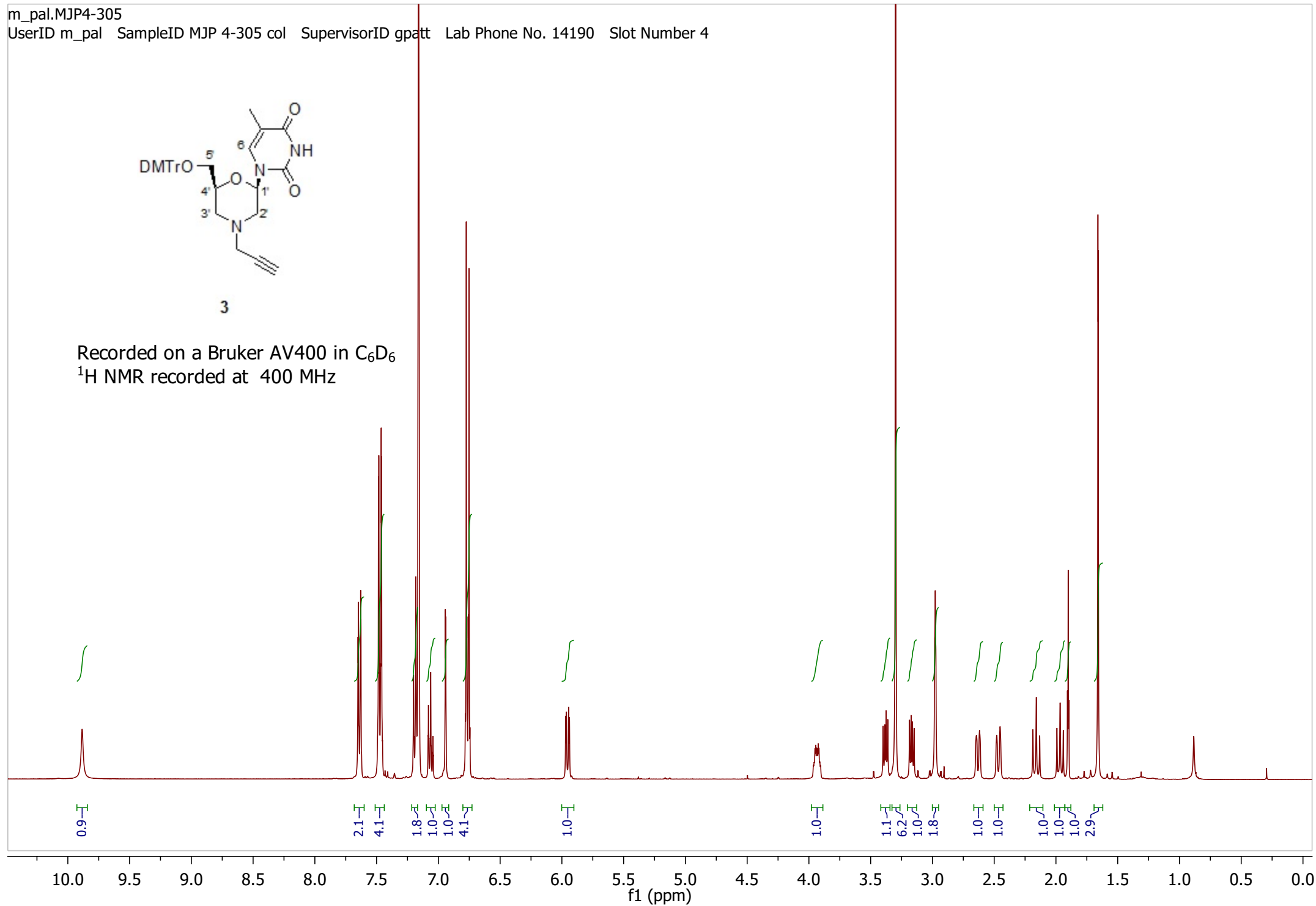
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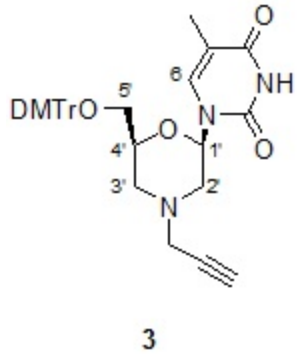
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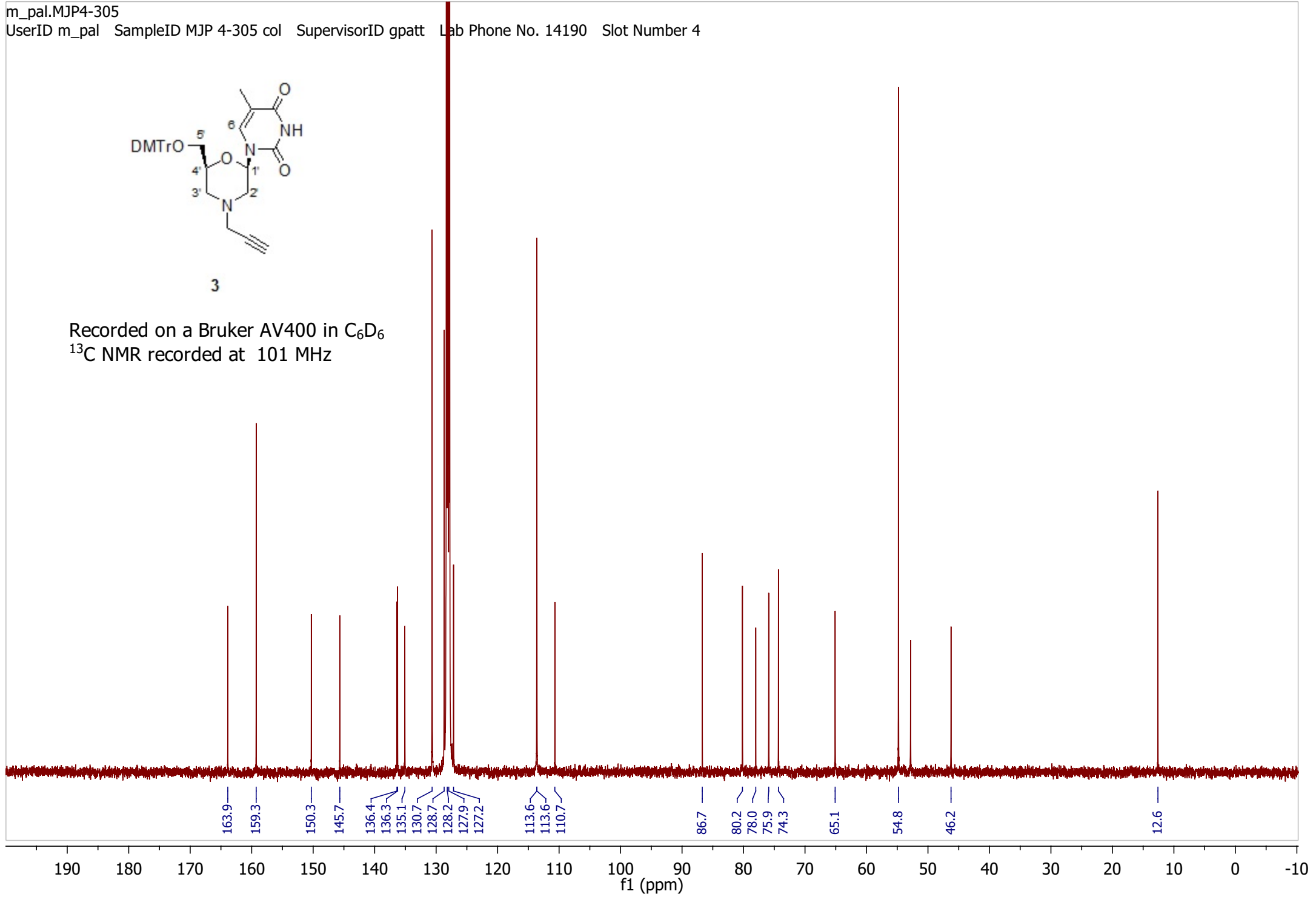


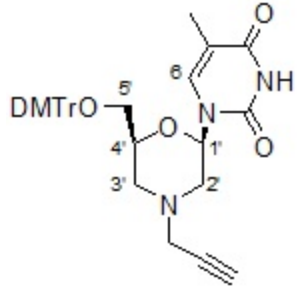
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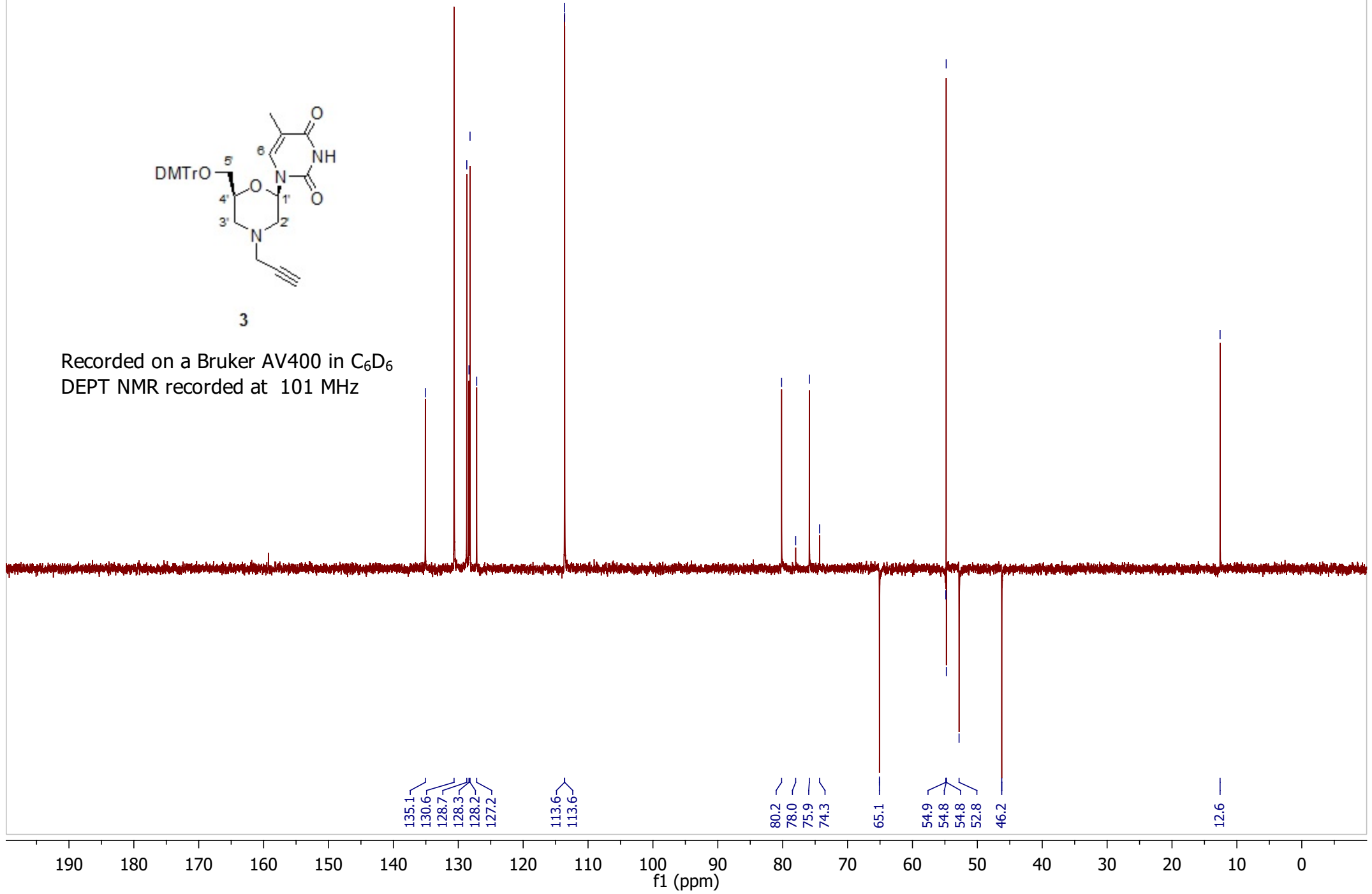
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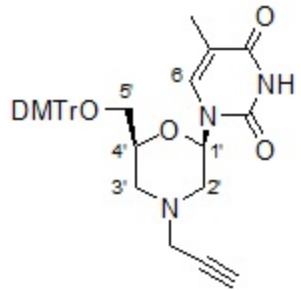


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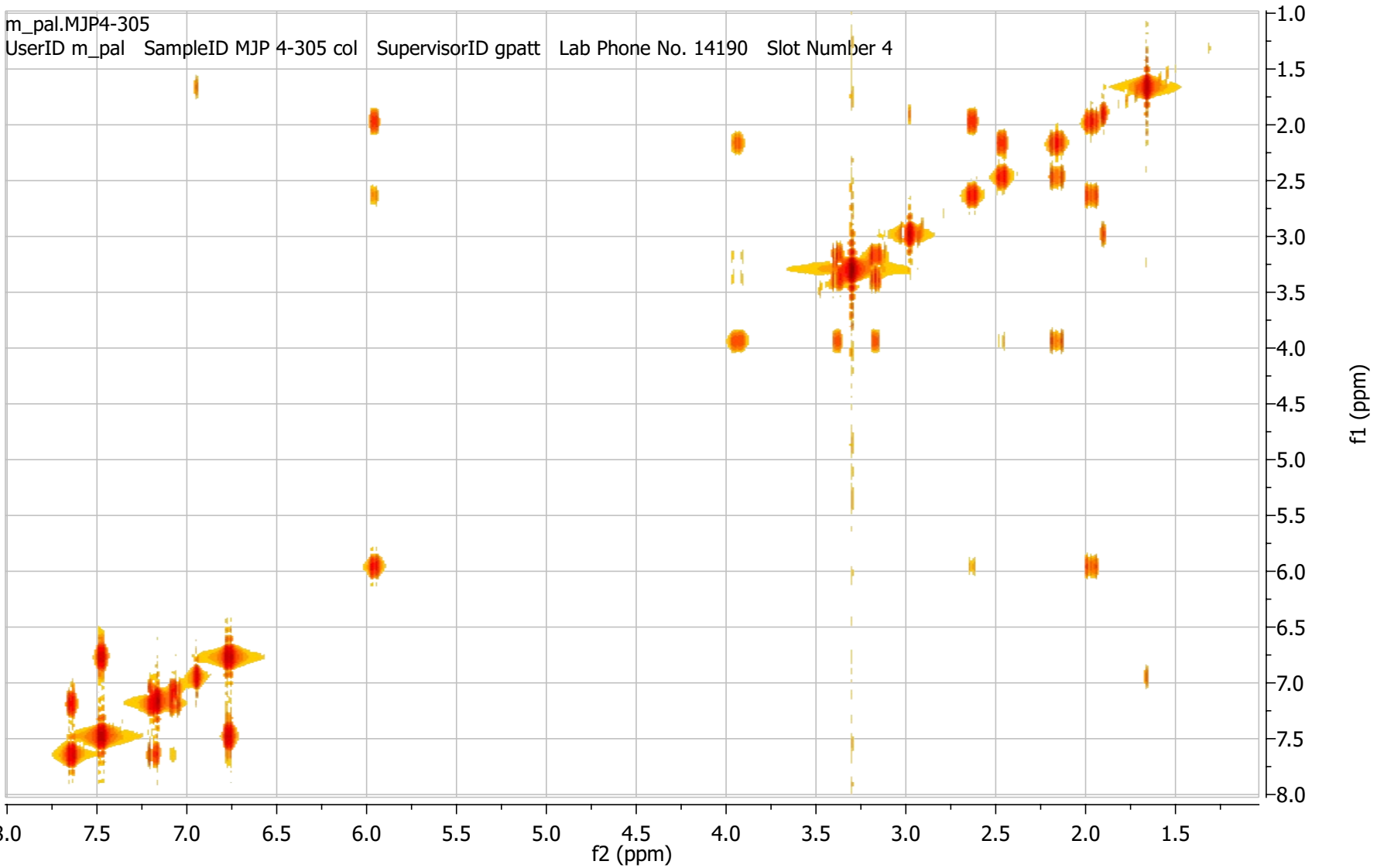
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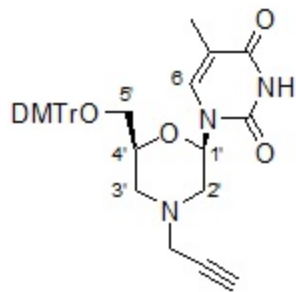
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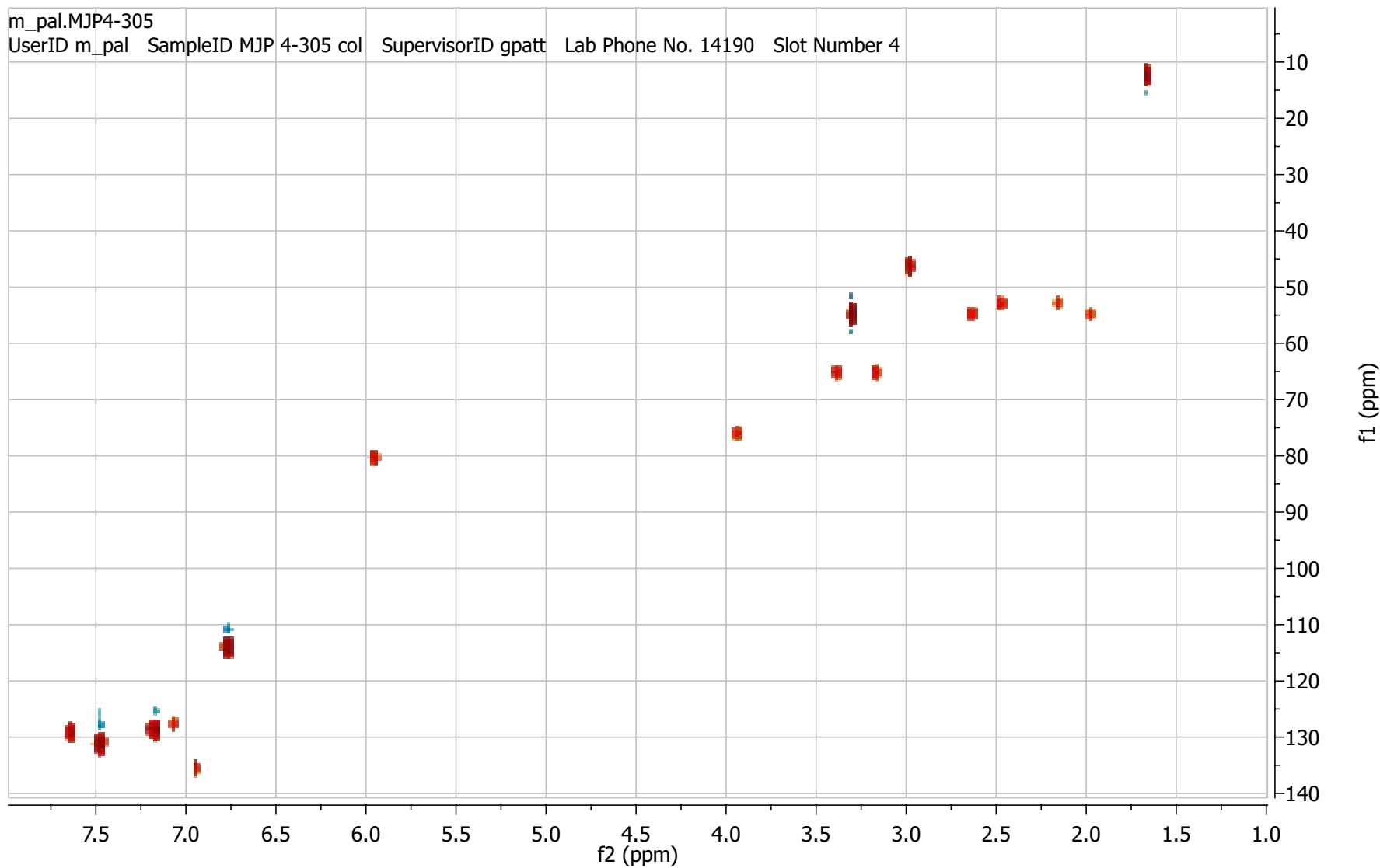
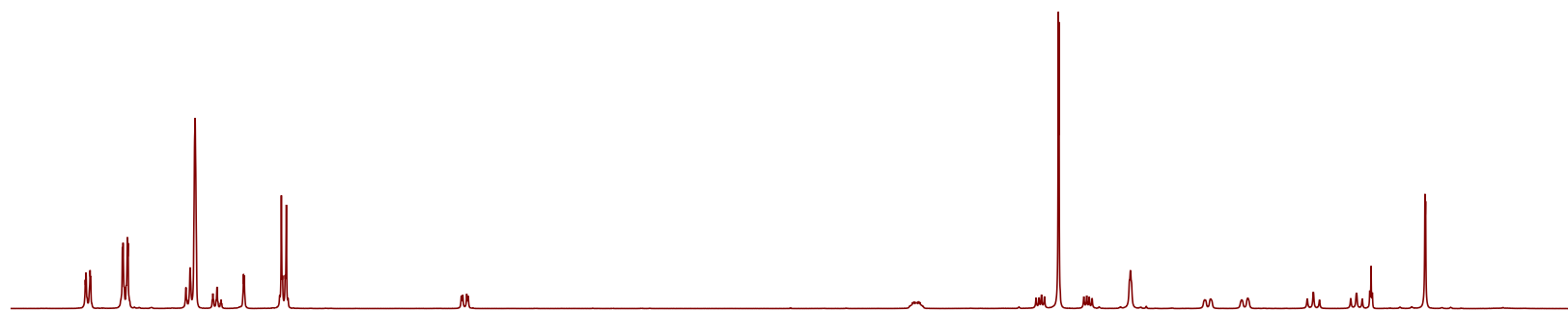
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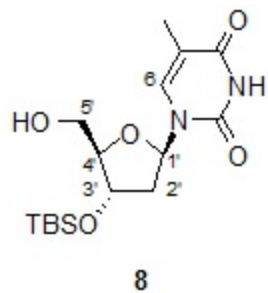


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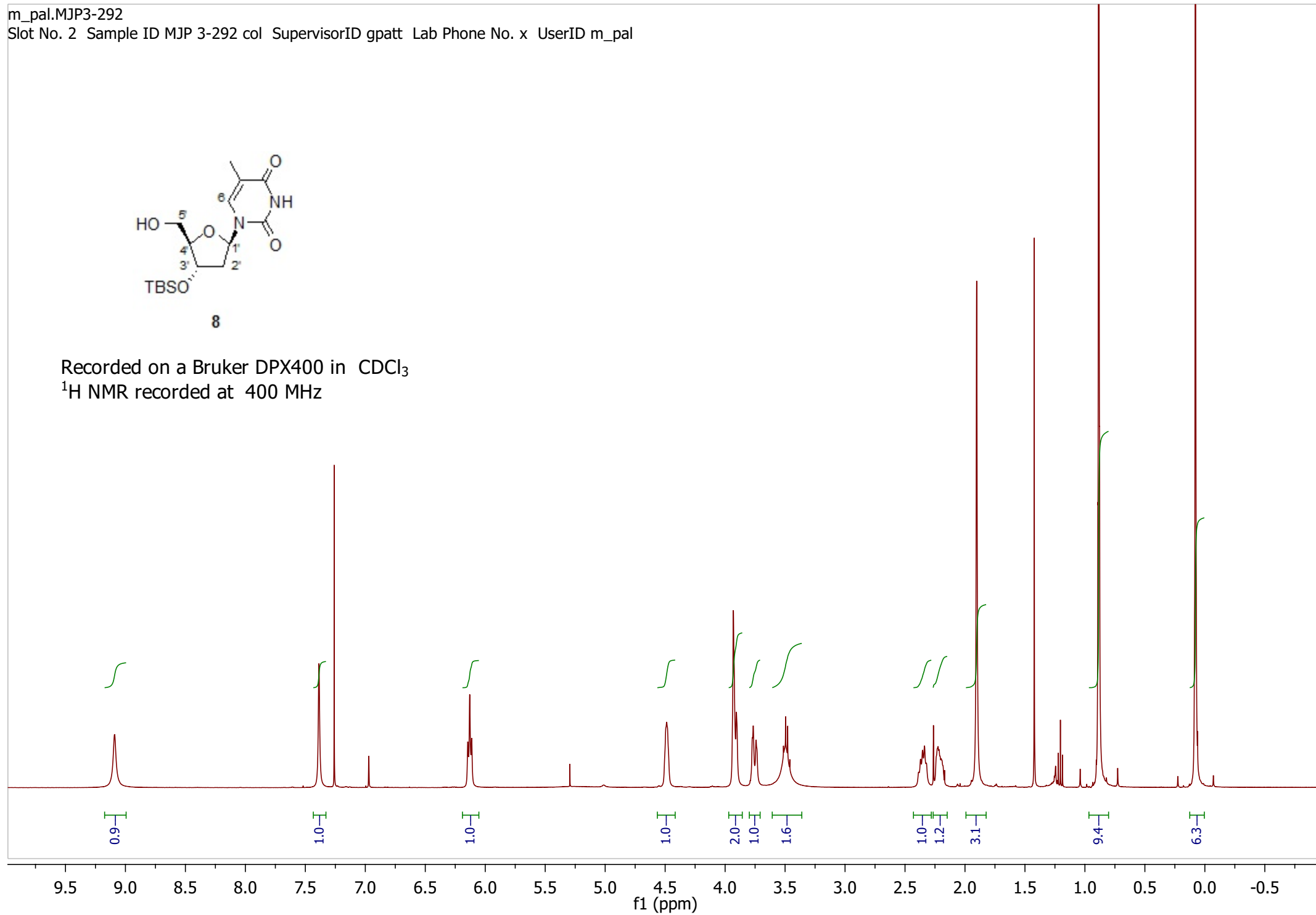


m_pal.MJP3-292

Slot No. 2 Sample ID MJP 3-292 col SupervisorID gpatt Lab Phone No. x UserID m_pal

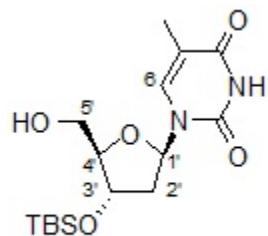


Recorded on a Bruker DPX400 in CDCl₃
¹H NMR recorded at 400 MHz



m_pal.MJP3-292

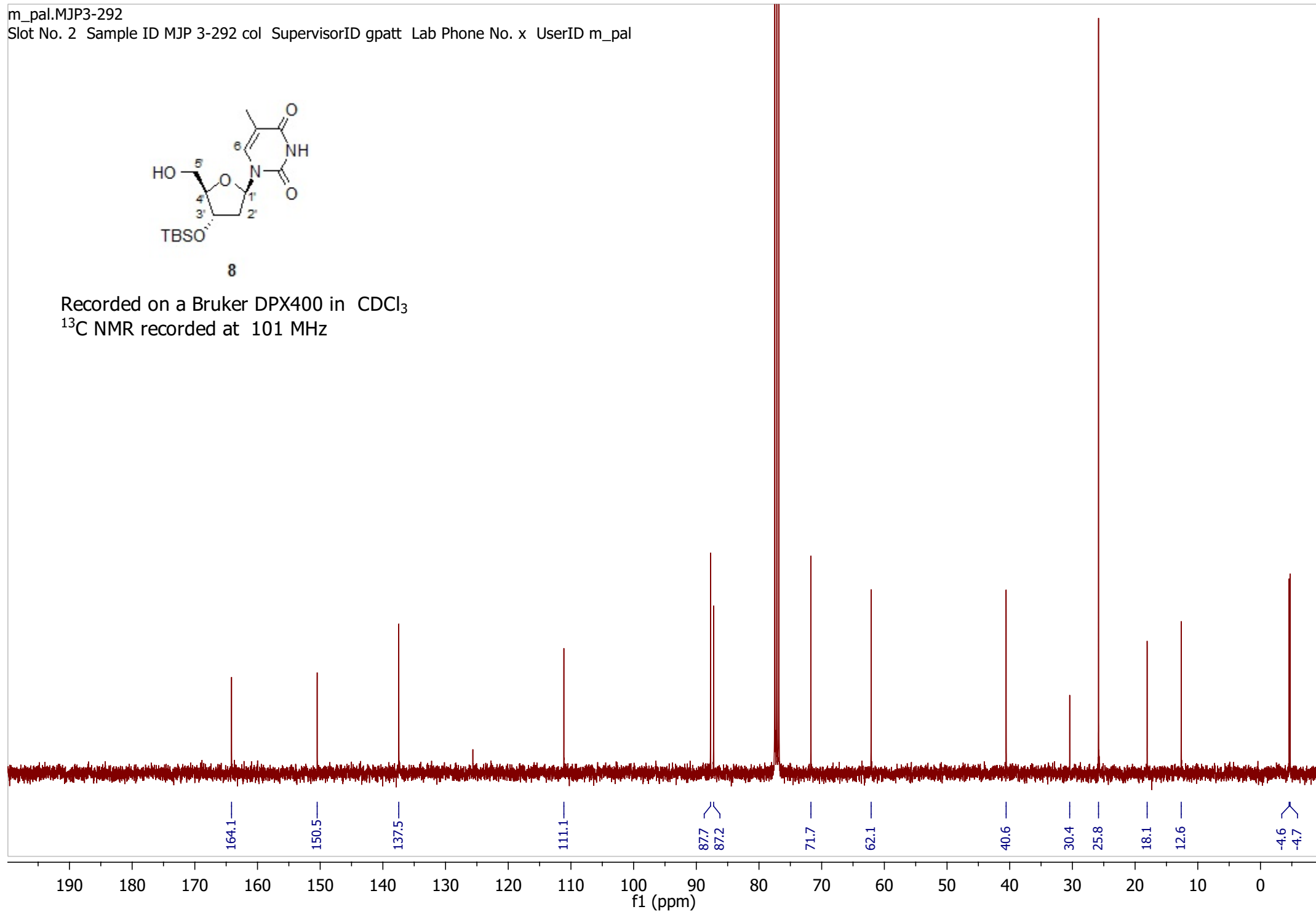
Slot No. 2 Sample ID MJP 3-292 col SupervisorID gpatt Lab Phone No. x UserID m_pal



8

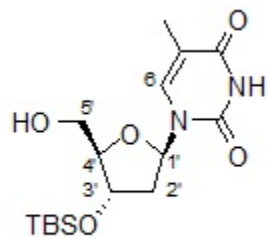
Recorded on a Bruker DPX400 in CDCl₃

¹³C NMR recorded at 101 MHz



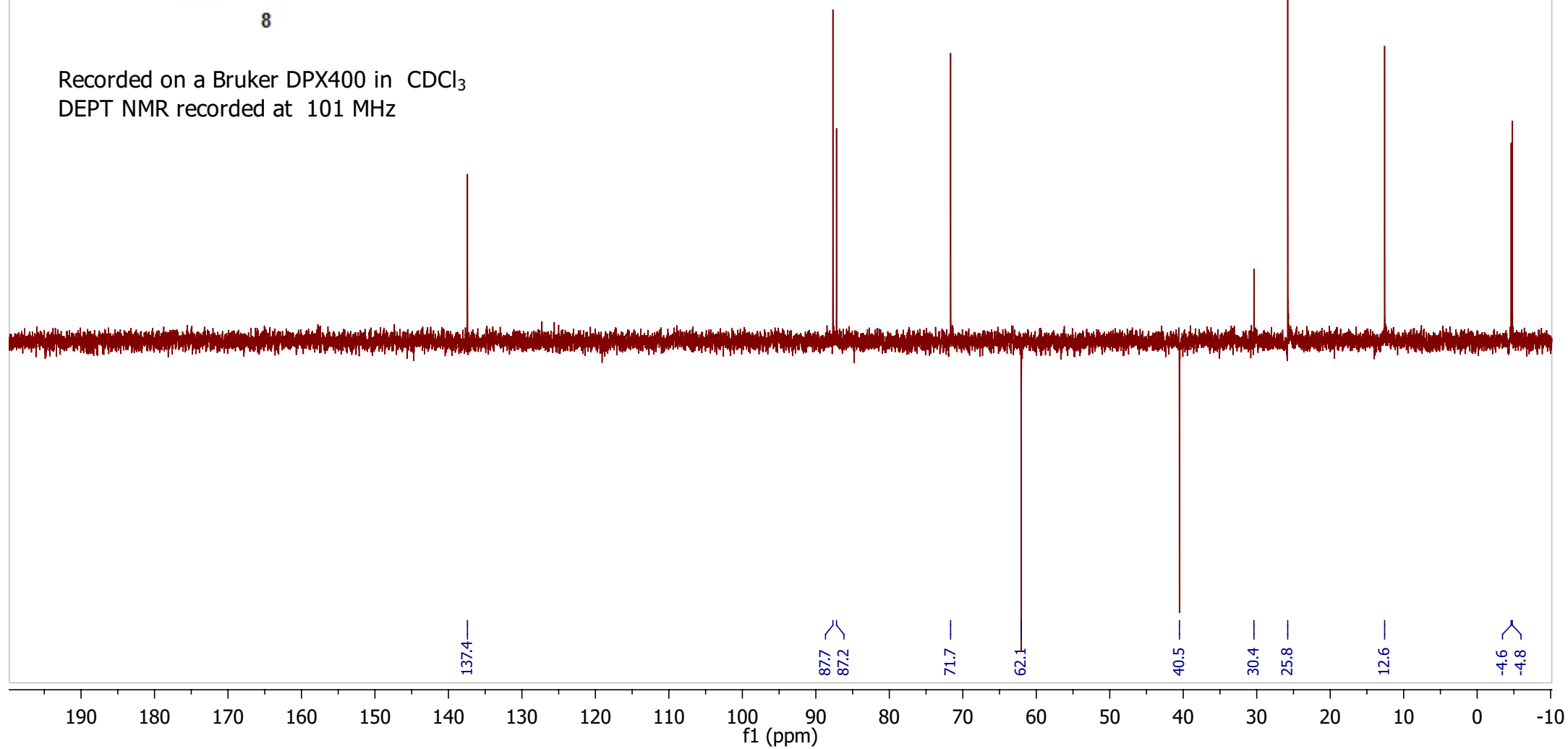
m_pal.MJP3-292

Slot No. 2 Sample ID MJP 3-292 col SupervisorID gpatt Lab Phone No. x UserID m_pal



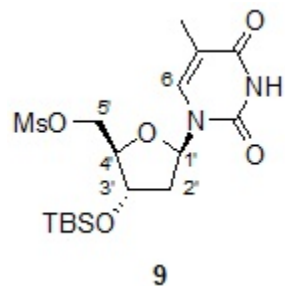
8

Recorded on a Bruker DPX400 in CDCl₃
DEPT NMR recorded at 101 MHz

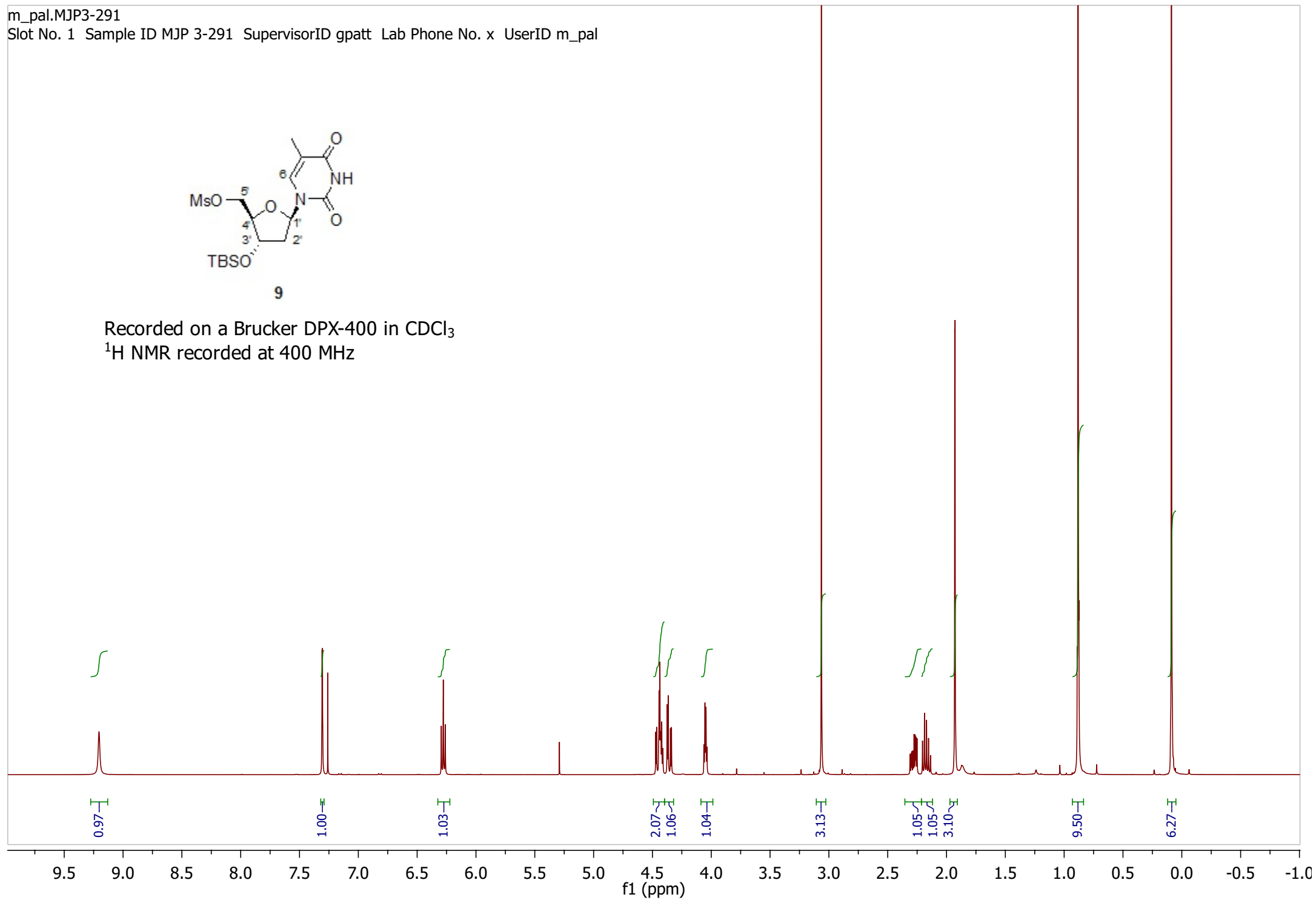


m_pal.MJP3-291

Slot No. 1 Sample ID MJP 3-291 SupervisorID gpatt Lab Phone No. x UserID m_pal

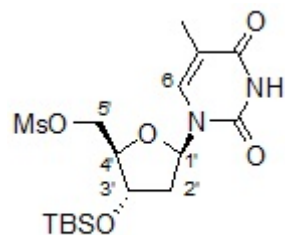


Recorded on a Bruker DPX-400 in CDCl₃
¹H NMR recorded at 400 MHz



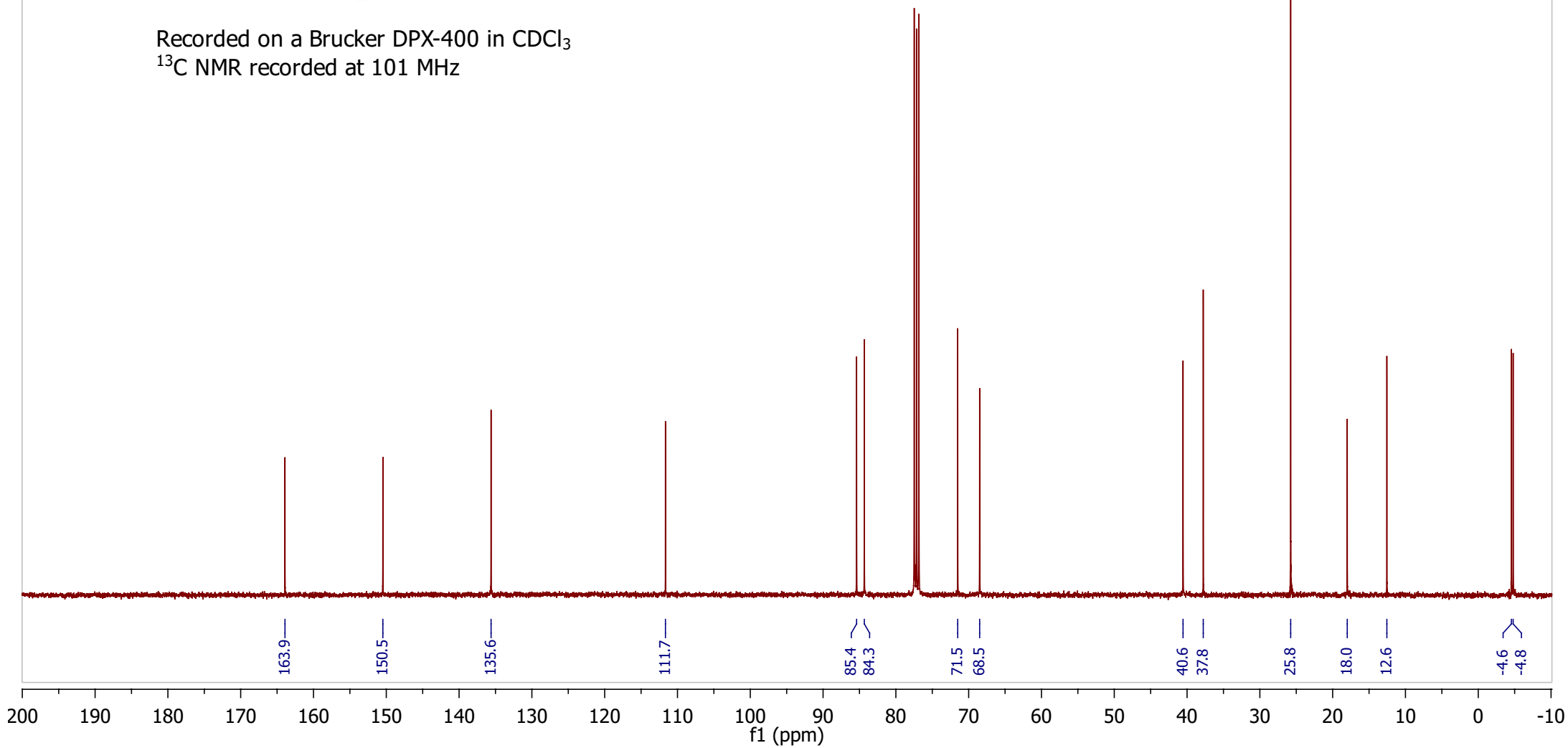
m_pal.MJP3-291

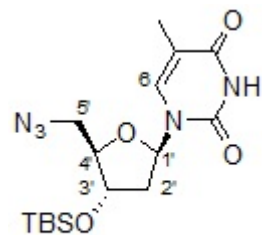
Slot No. 1 Sample ID MJP 3-291 SupervisorID gpatt Lab Phone No. x UserID m_pal



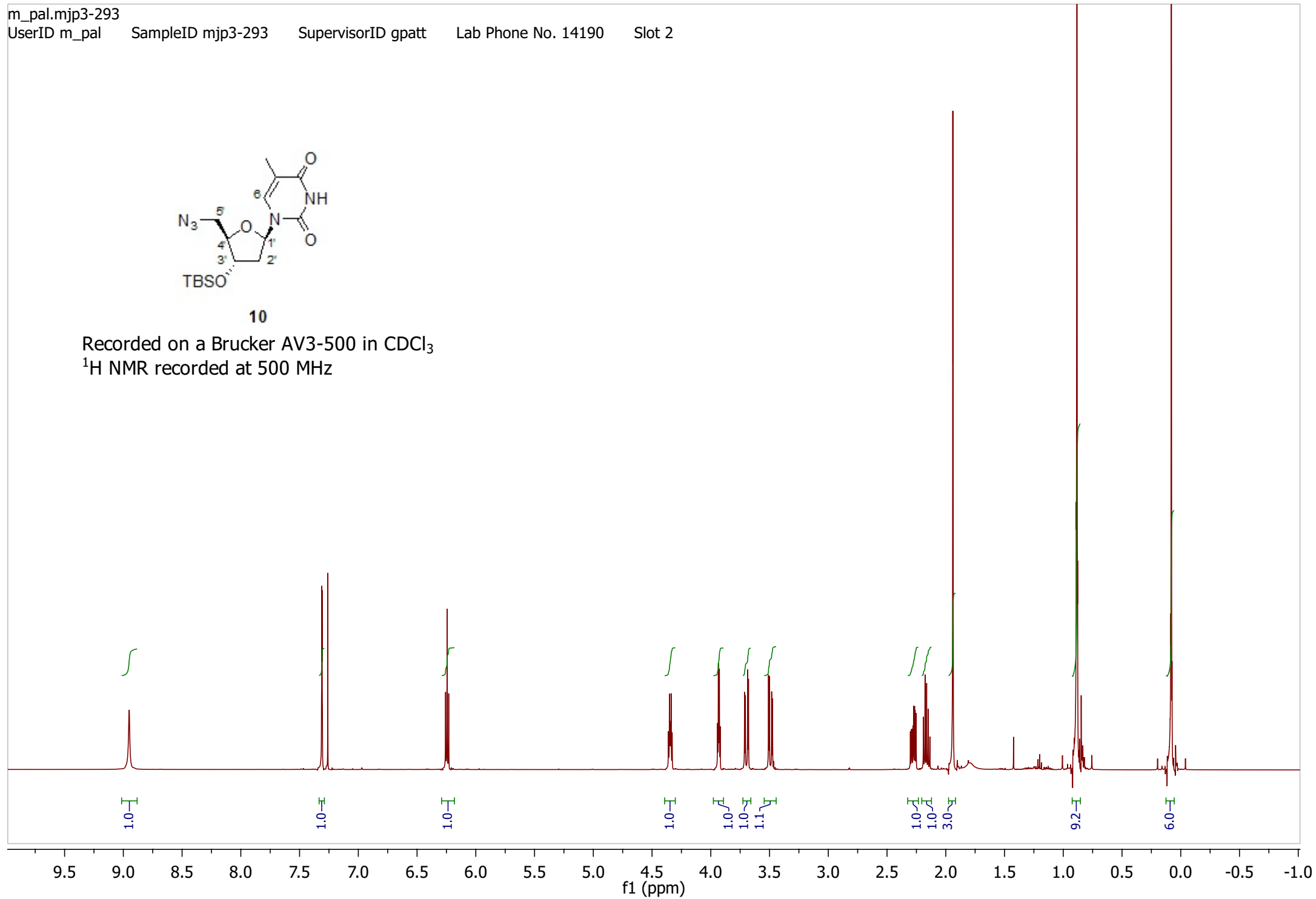
9

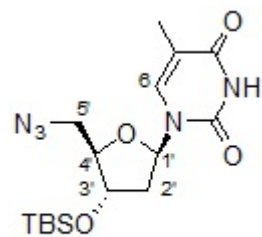
Recorded on a Bruker DPX-400 in CDCl₃
¹³C NMR recorded at 101 MHz



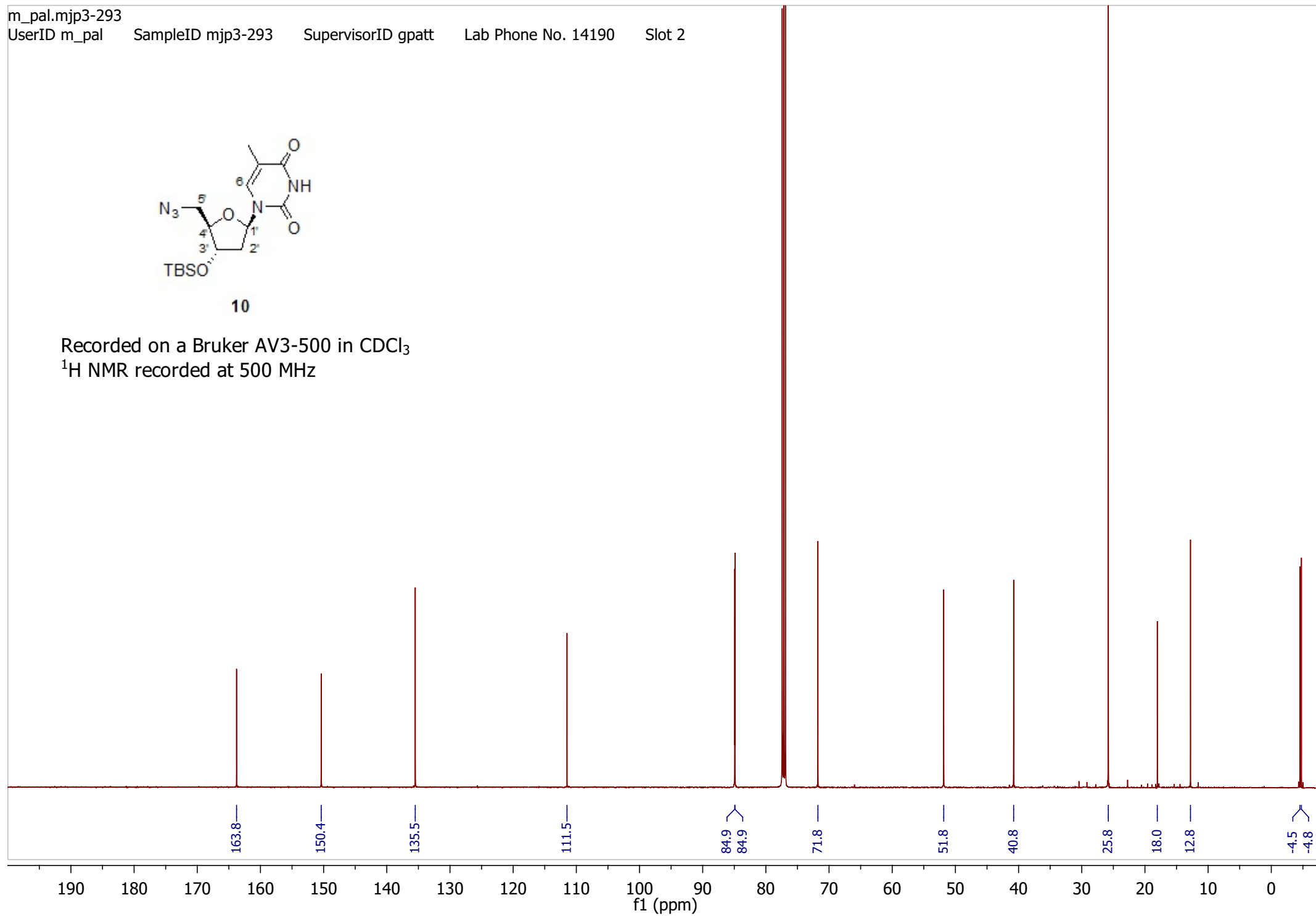


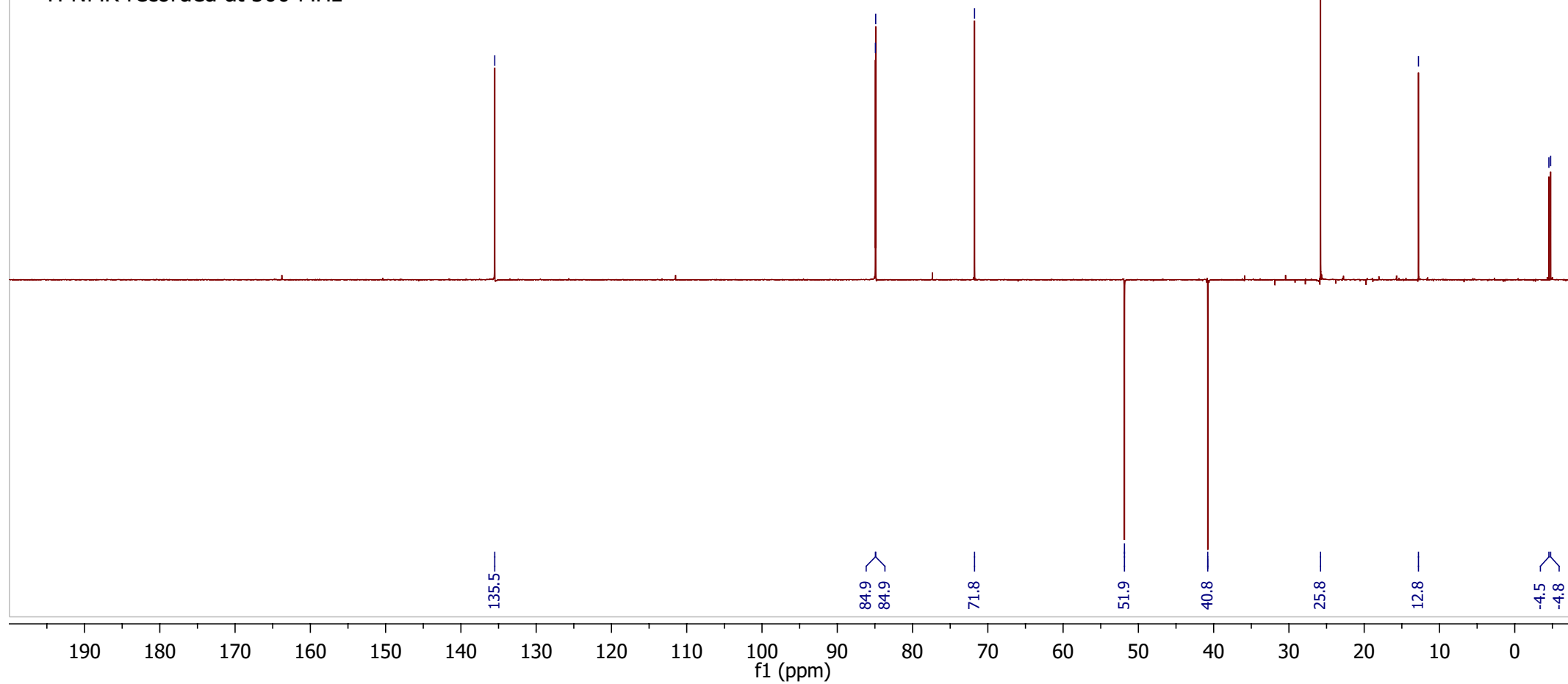
10

Recorded on a Bruker AV3-500 in $CDCl_3$ 1H NMR recorded at 500 MHz

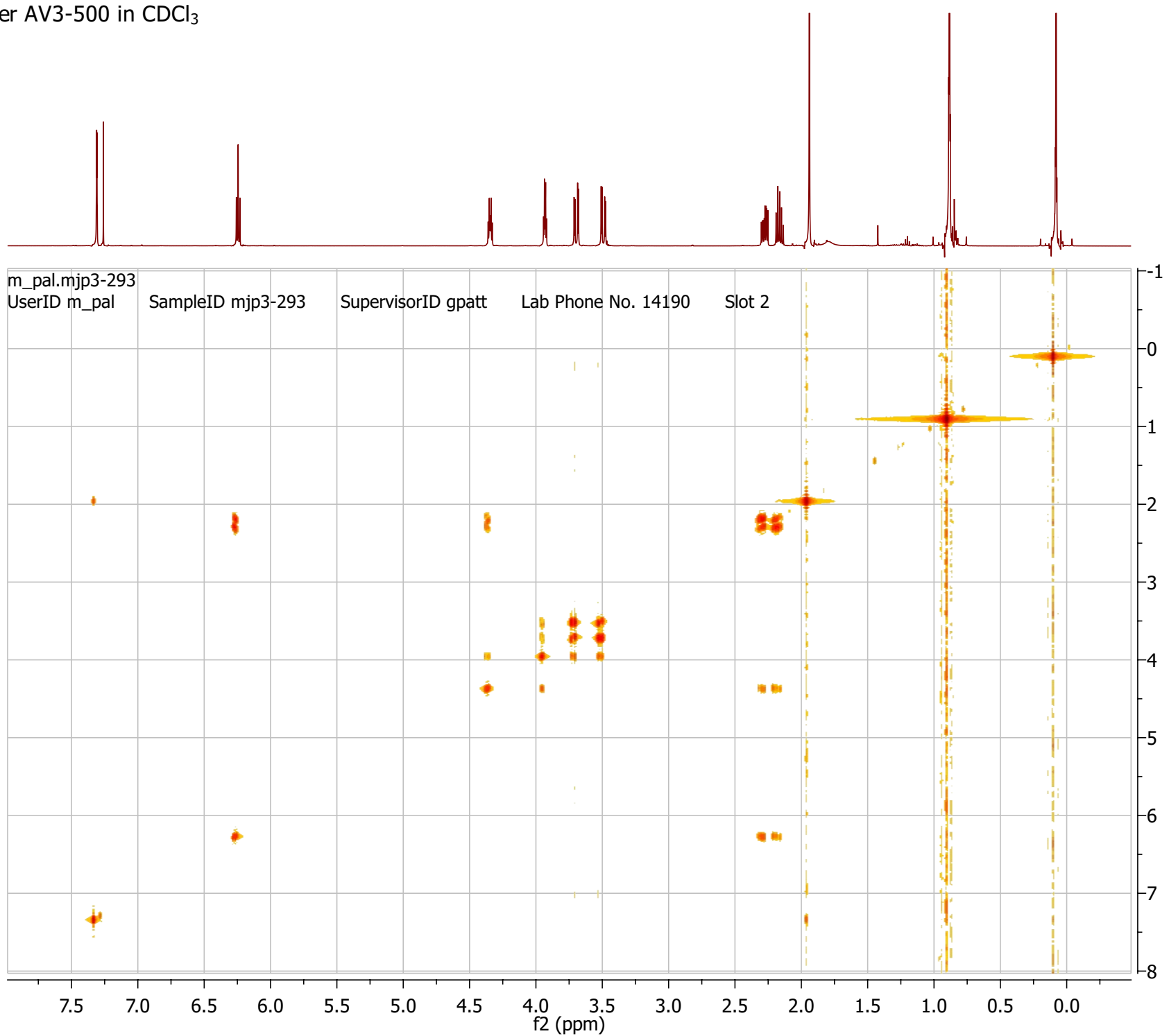
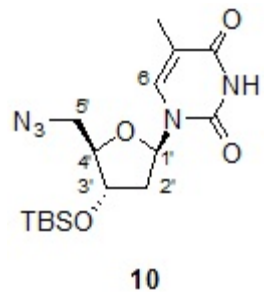
**10**

Recorded on a Bruker AV3-500 in CDCl₃
¹H NMR recorded at 500 MHz

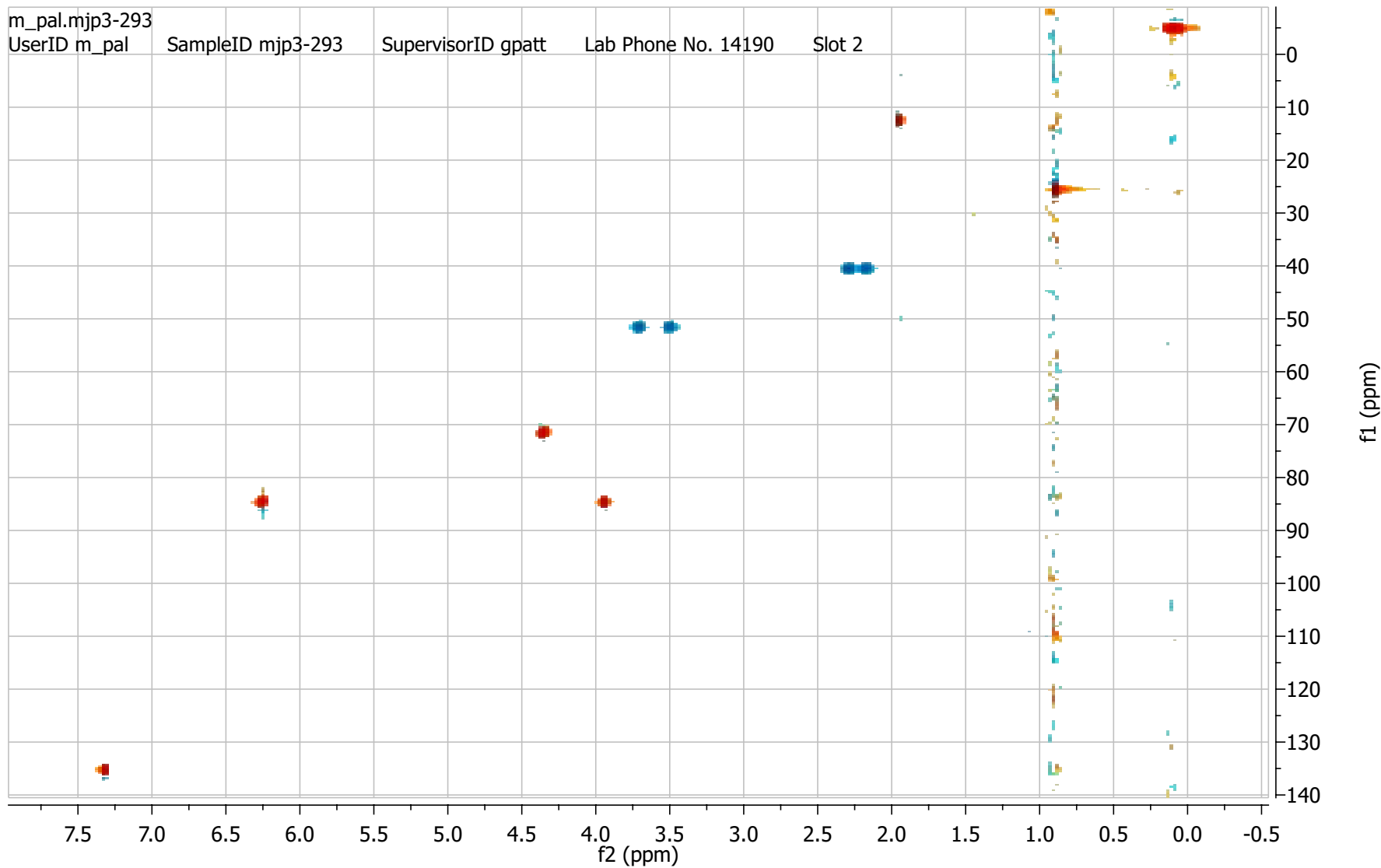
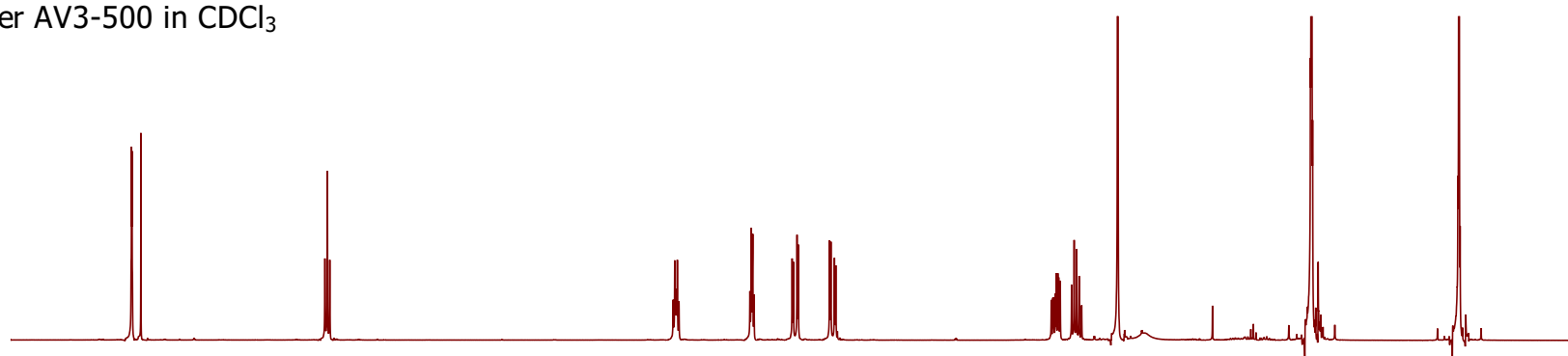
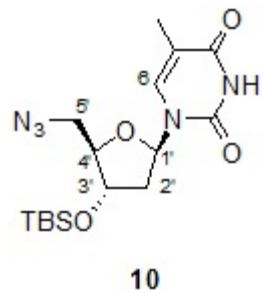


**10**Recorded on a Bruker AV3-500 in CDCl₃¹H NMR recorded at 500 MHz

HSQC Recorded on a Bruker AV3-500 in CDCl₃



HSQC Recorded on a Bruker AV3-500 in CDCl₃



m_pal.mjp4-308

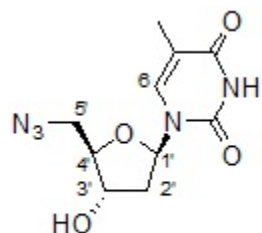
UserID m_pal

SampleID mjp4-308

SupervisorID gpatt

Lab Phone No. 14190

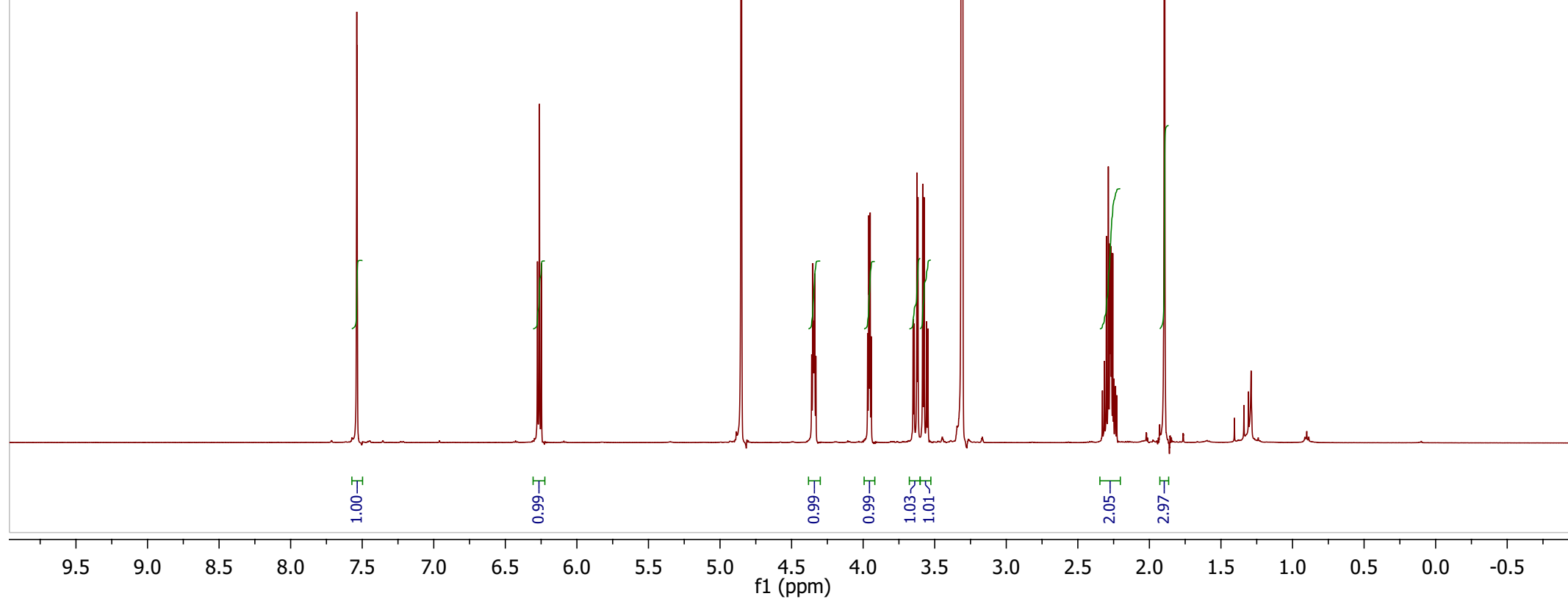
Slot 2

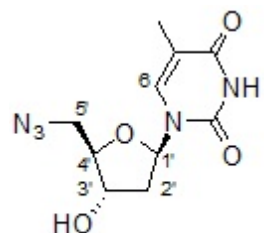


4

Recorded on a Bruker AV3-500 in D4-MeOD

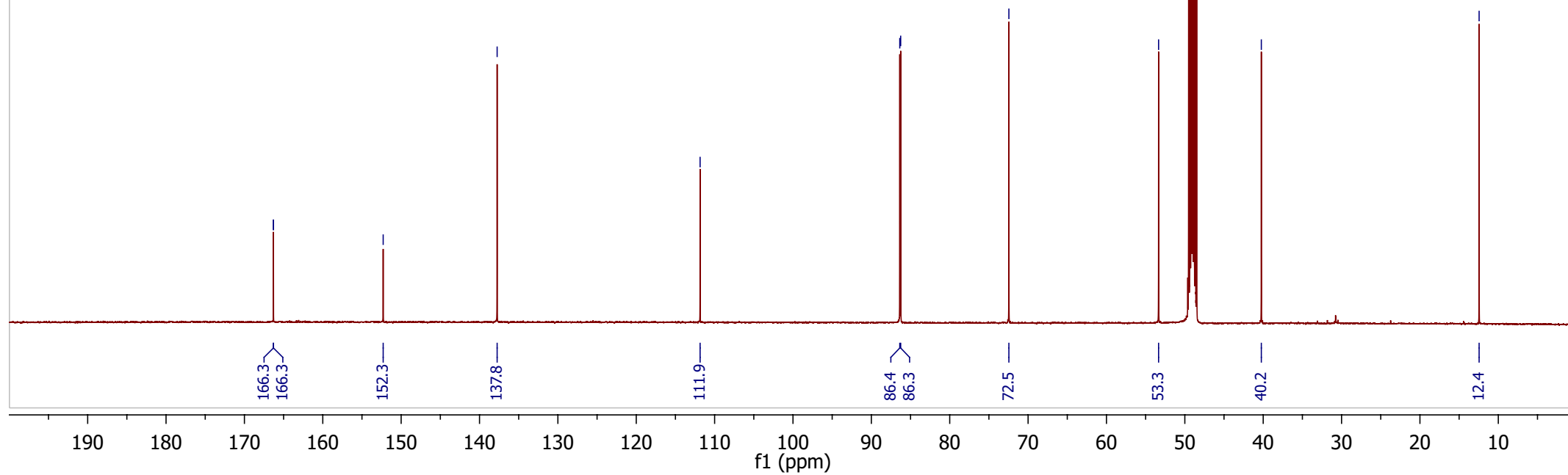
¹H NMR recorded at 500 MHz





4

Recorded on a Bruker AV3-500 in D4-MeOD

 ^1H NMR recorded at 126 MHz

m_pal.mjp4-308

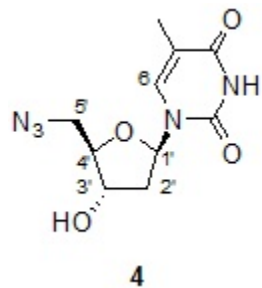
UserID m_pal

SampleID mjp4-308

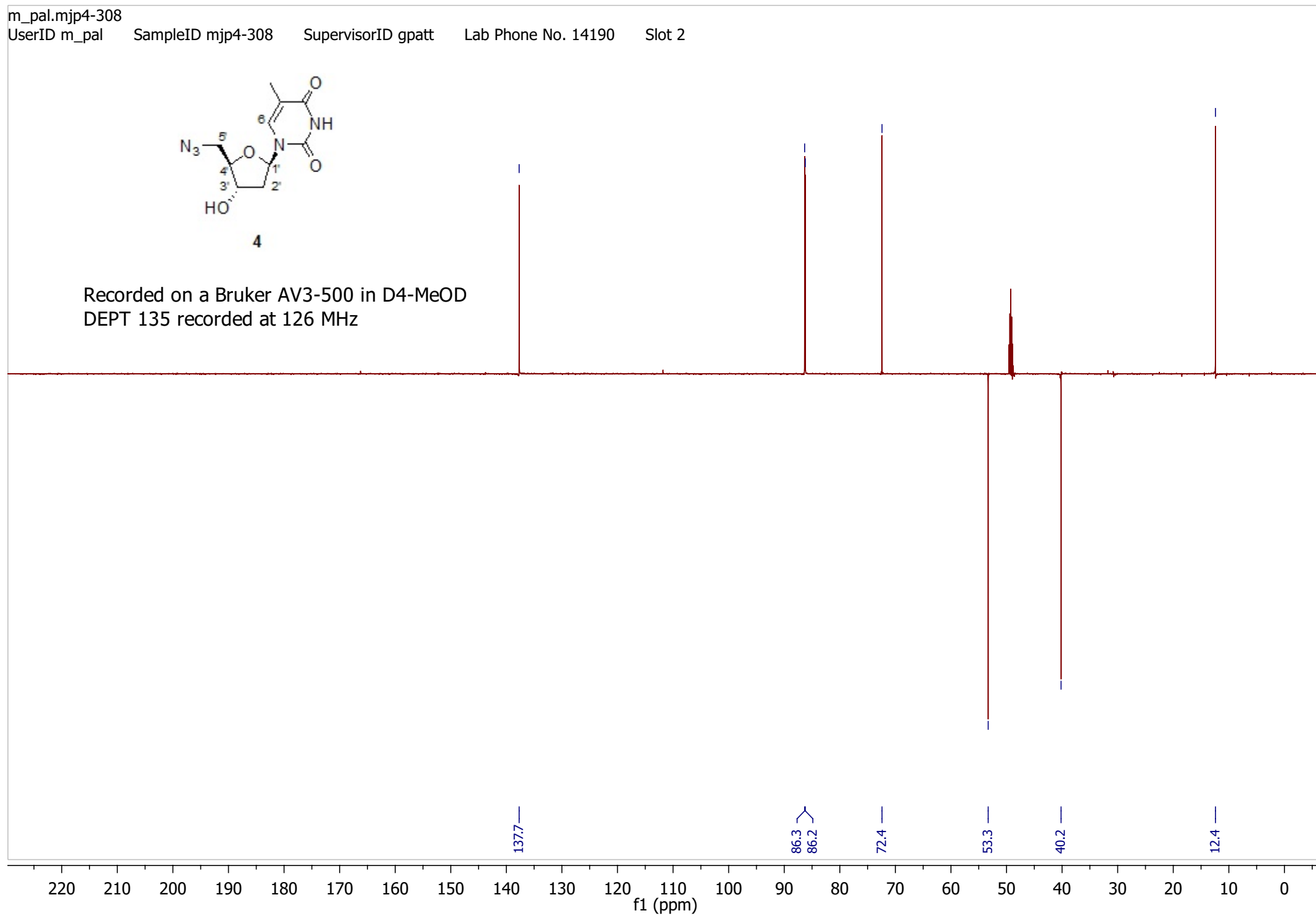
SupervisorID gpatt

Lab Phone No. 14190

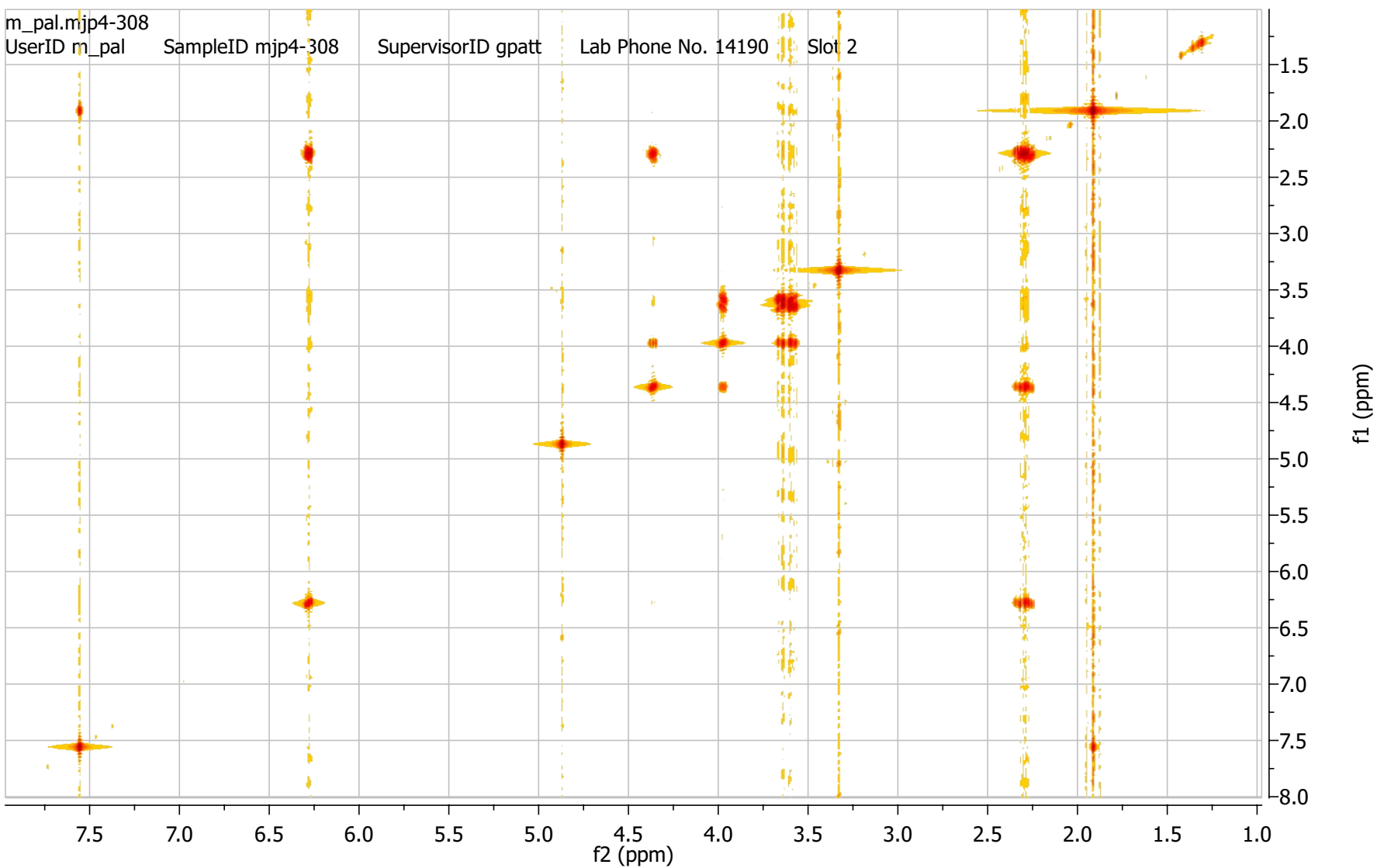
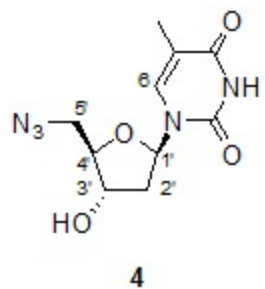
Slot 2



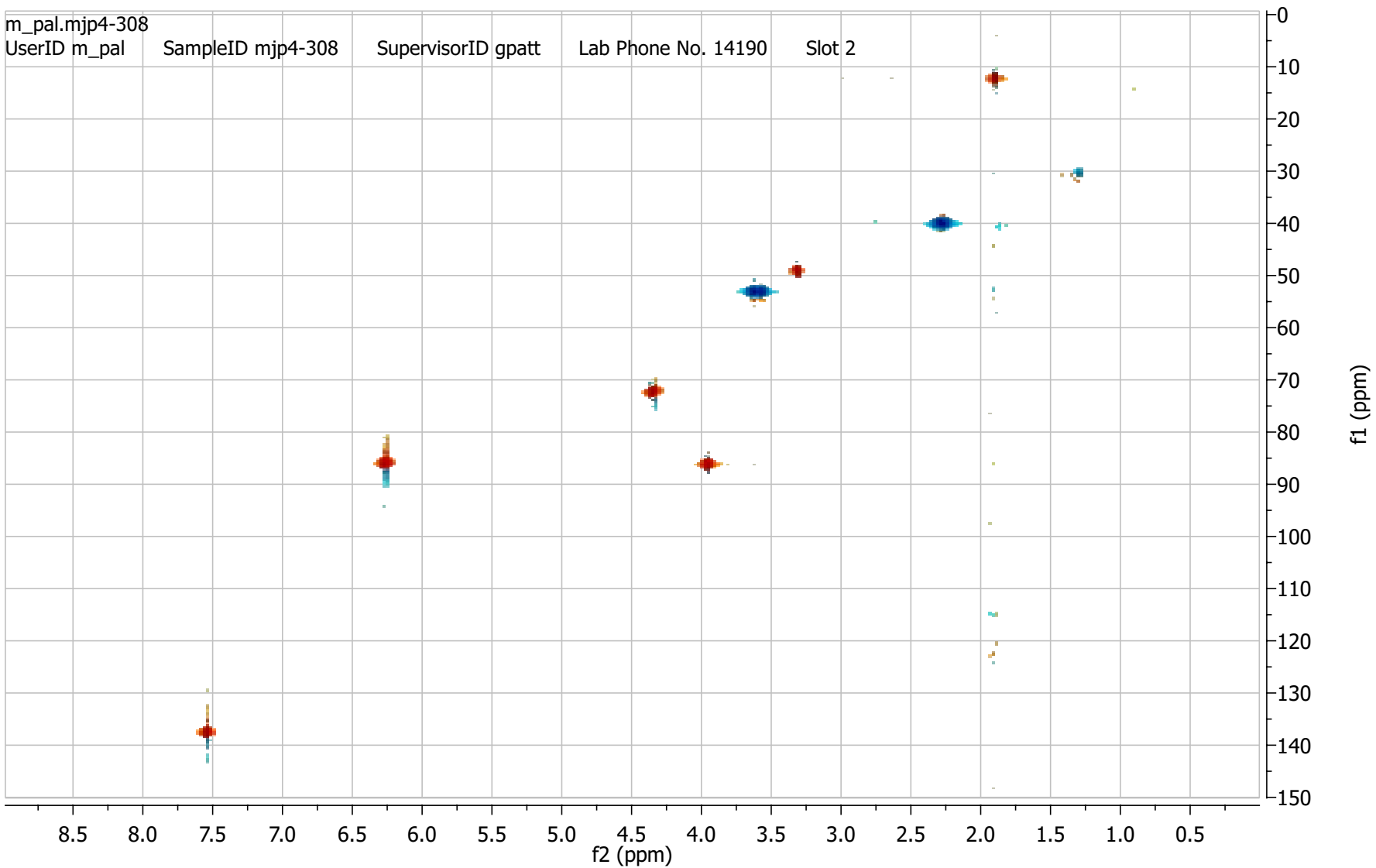
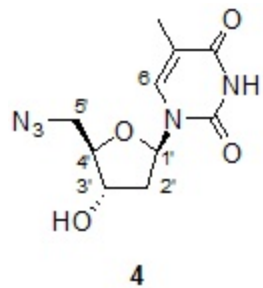
Recorded on a Bruker AV3-500 in D4-MeOD
DEPT 135 recorded at 126 MHz



COSY Recorded on a Bruker AV3-500 in D4-MeOD



¹H-¹³C HSQC recorded on Bruker AV3-500 in D₄-MeOD

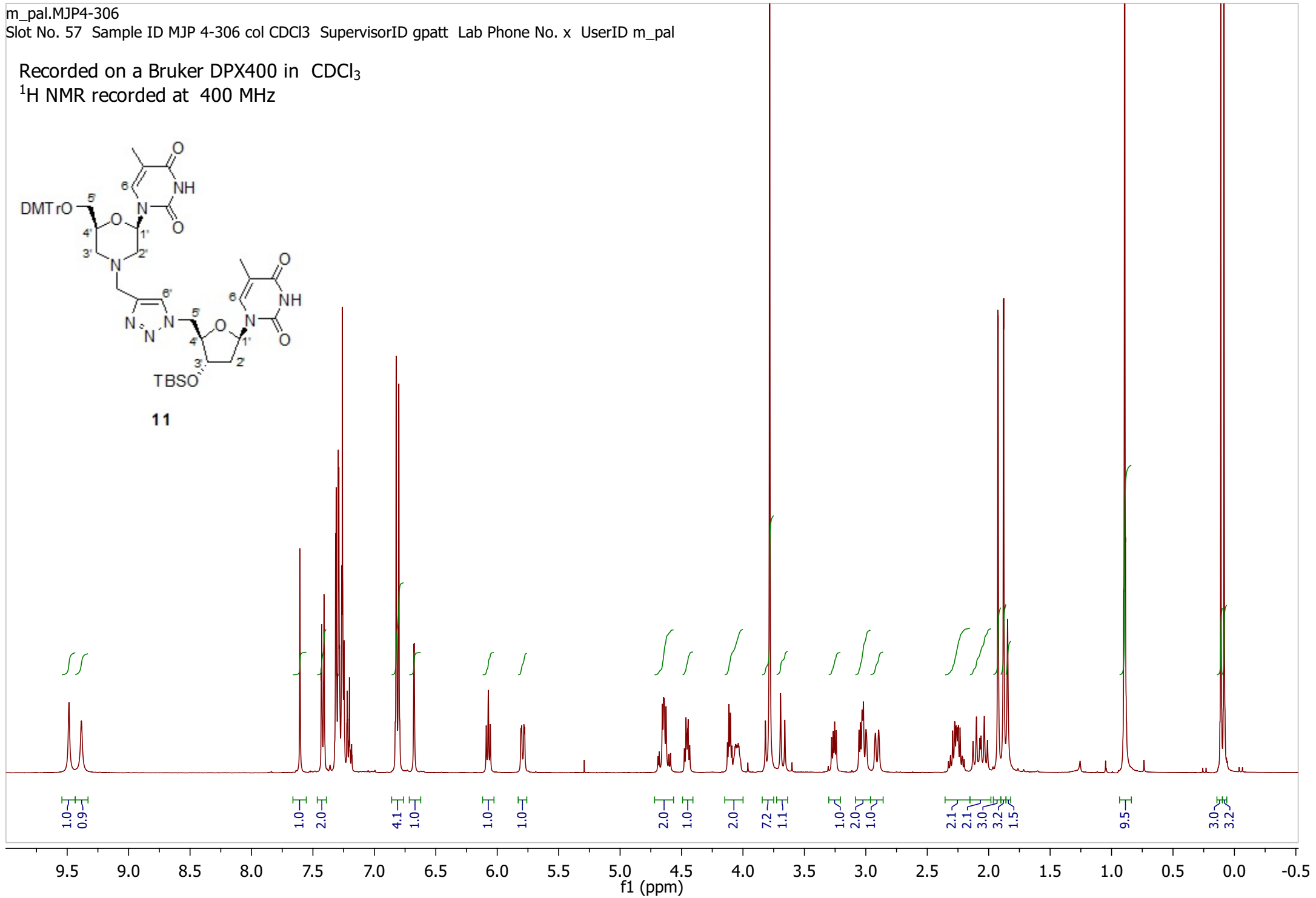
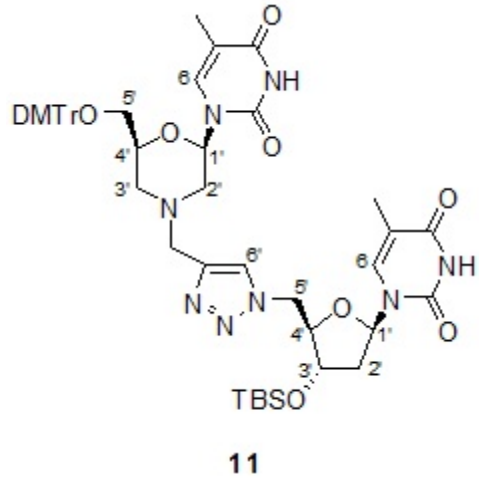


m_pal.MJP4-306

Slot No. 57 Sample ID MJP 4-306 col CDCl3 SupervisorID gpatt Lab Phone No. x UserID m_pal

Recorded on a Bruker DPX400 in CDCl₃

¹H NMR recorded at 400 MHz

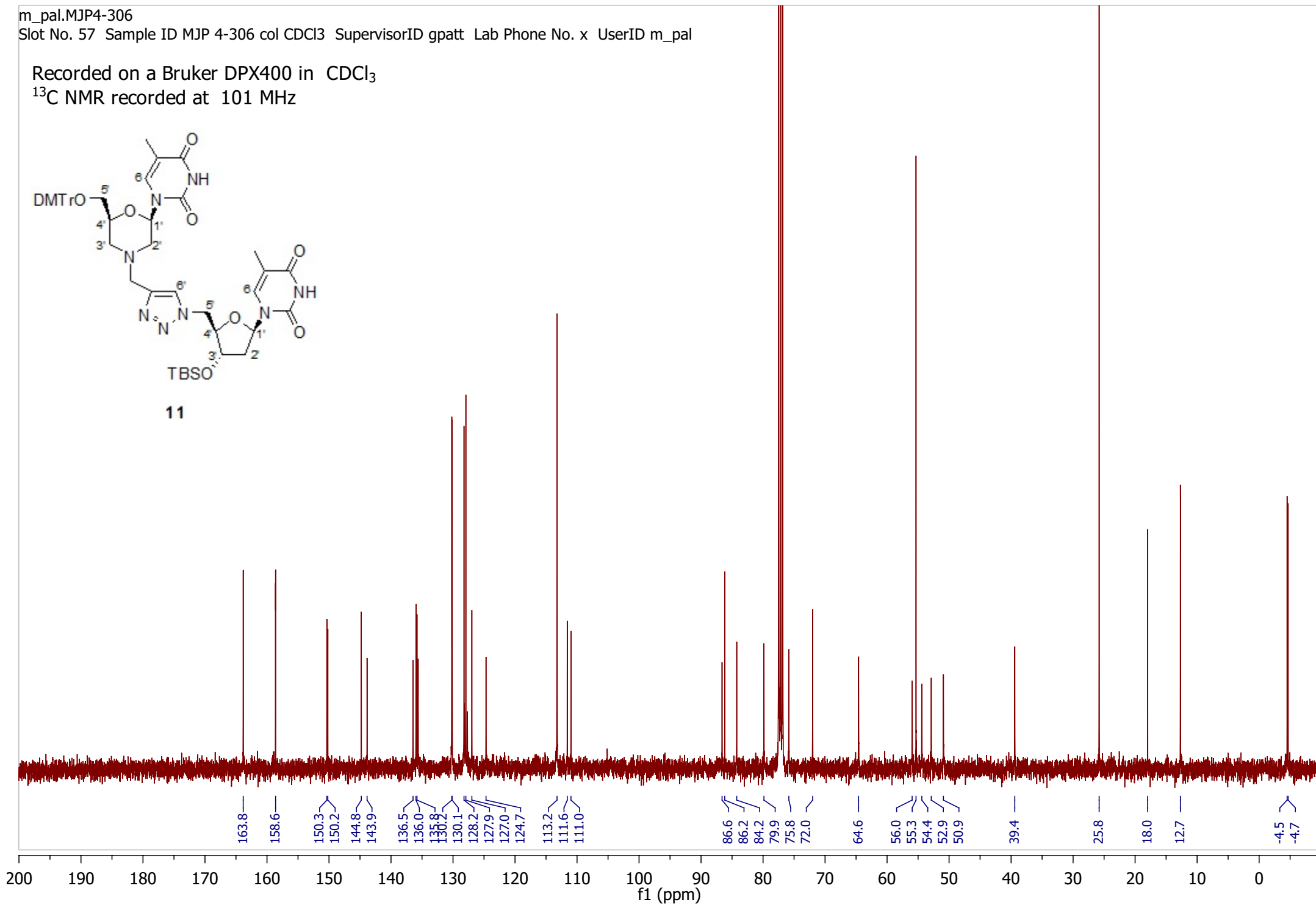
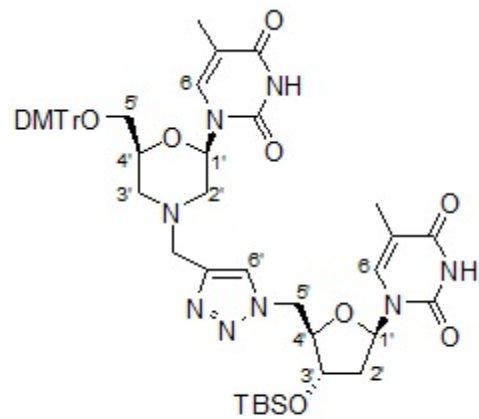


m_pal.MJP4-306

Slot No. 57 Sample ID MJP 4-306 col CDCl3 SupervisorID gpatt Lab Phone No. x UserID m_pal

Recorded on a Bruker DPX400 in CDCl₃

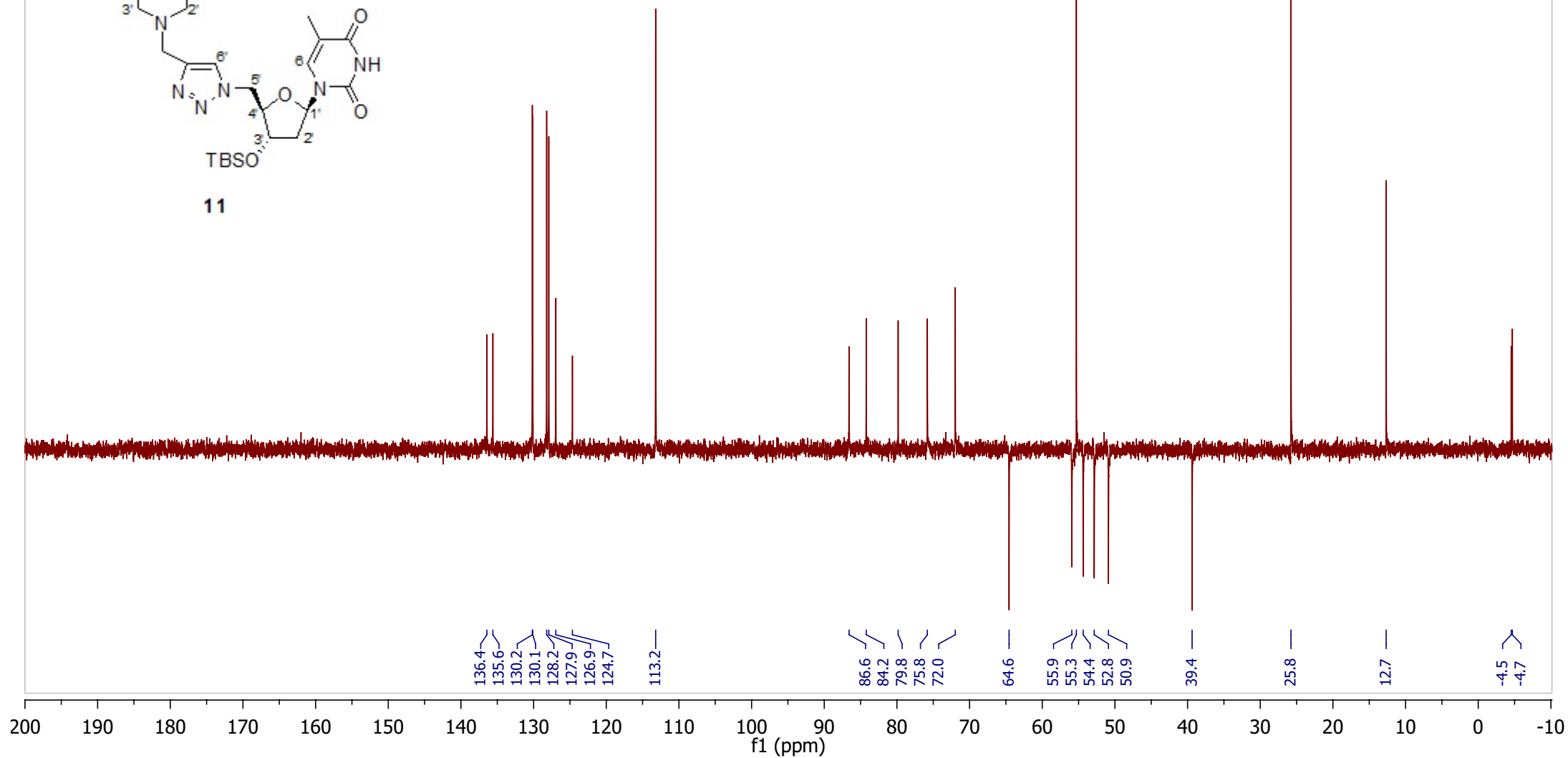
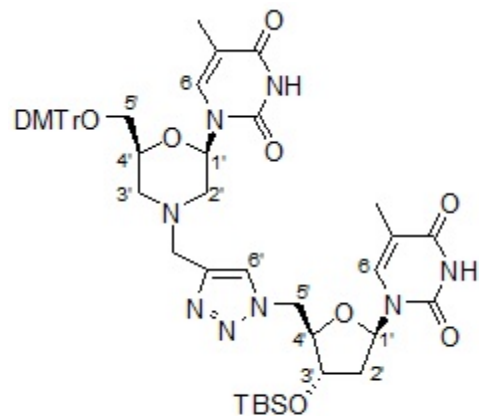
¹³C NMR recorded at 101 MHz

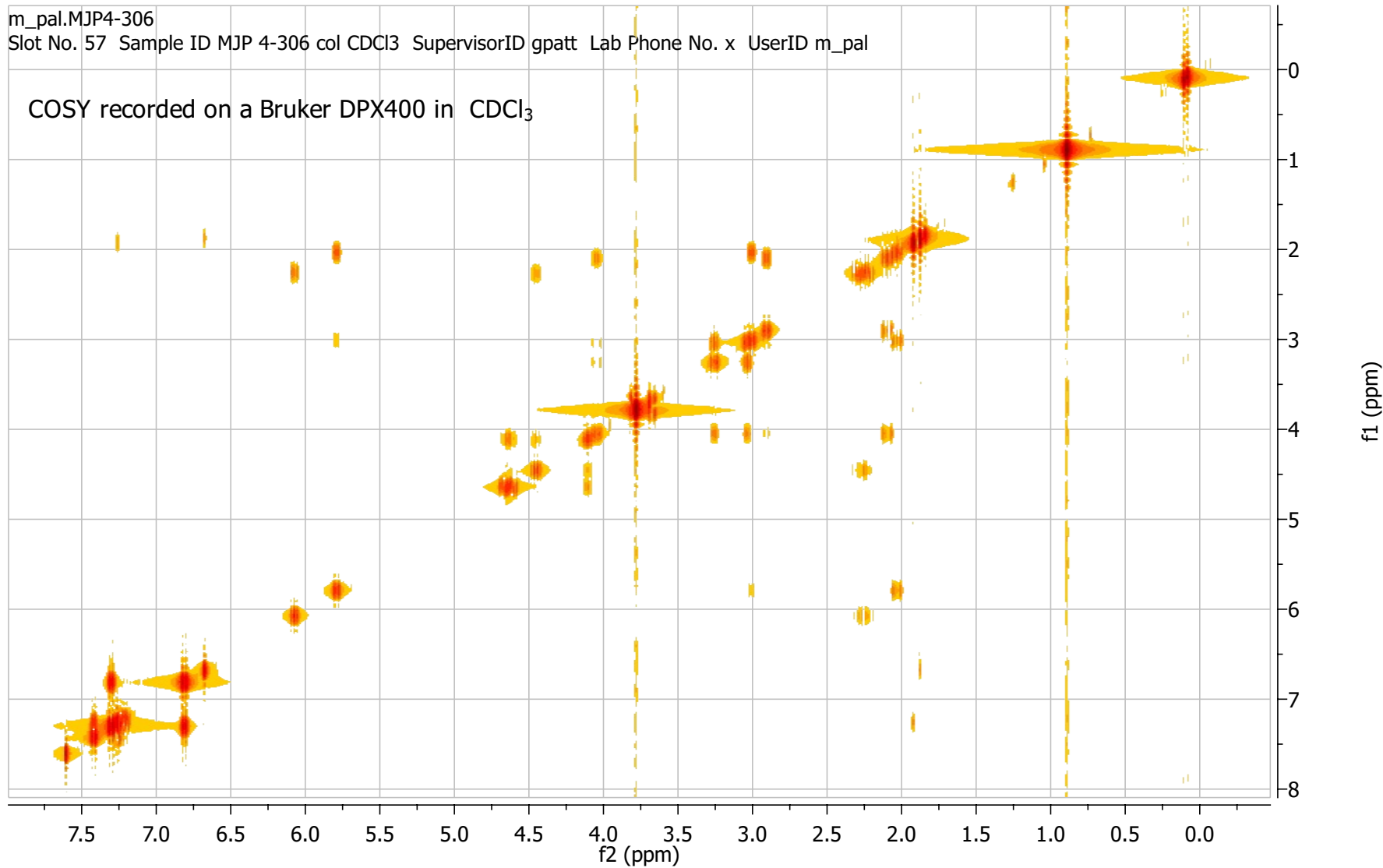
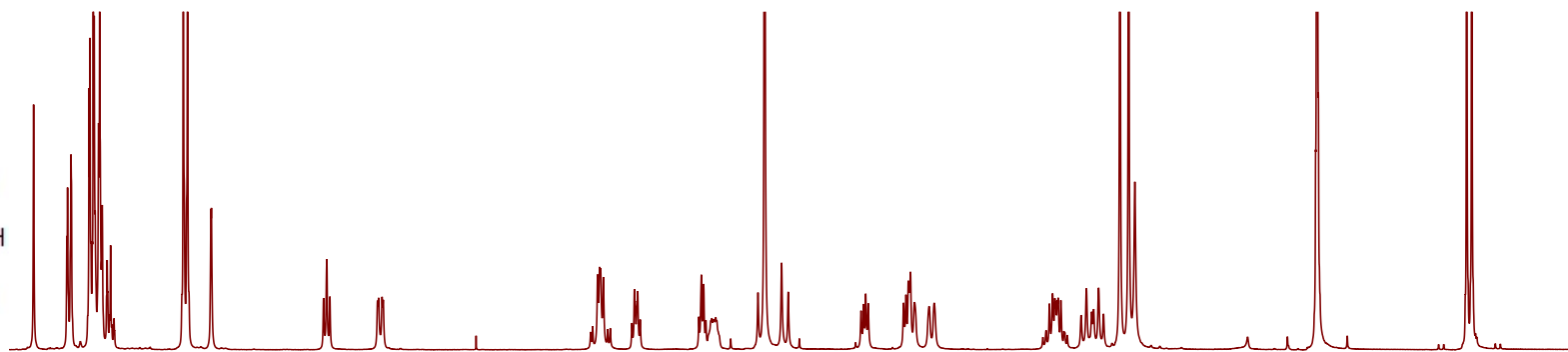
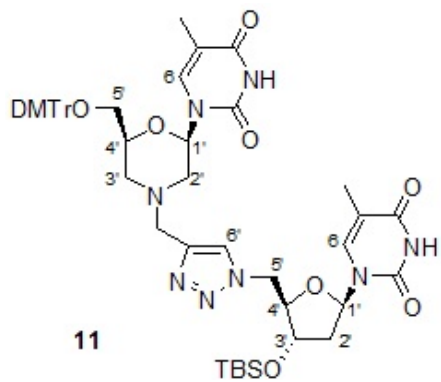


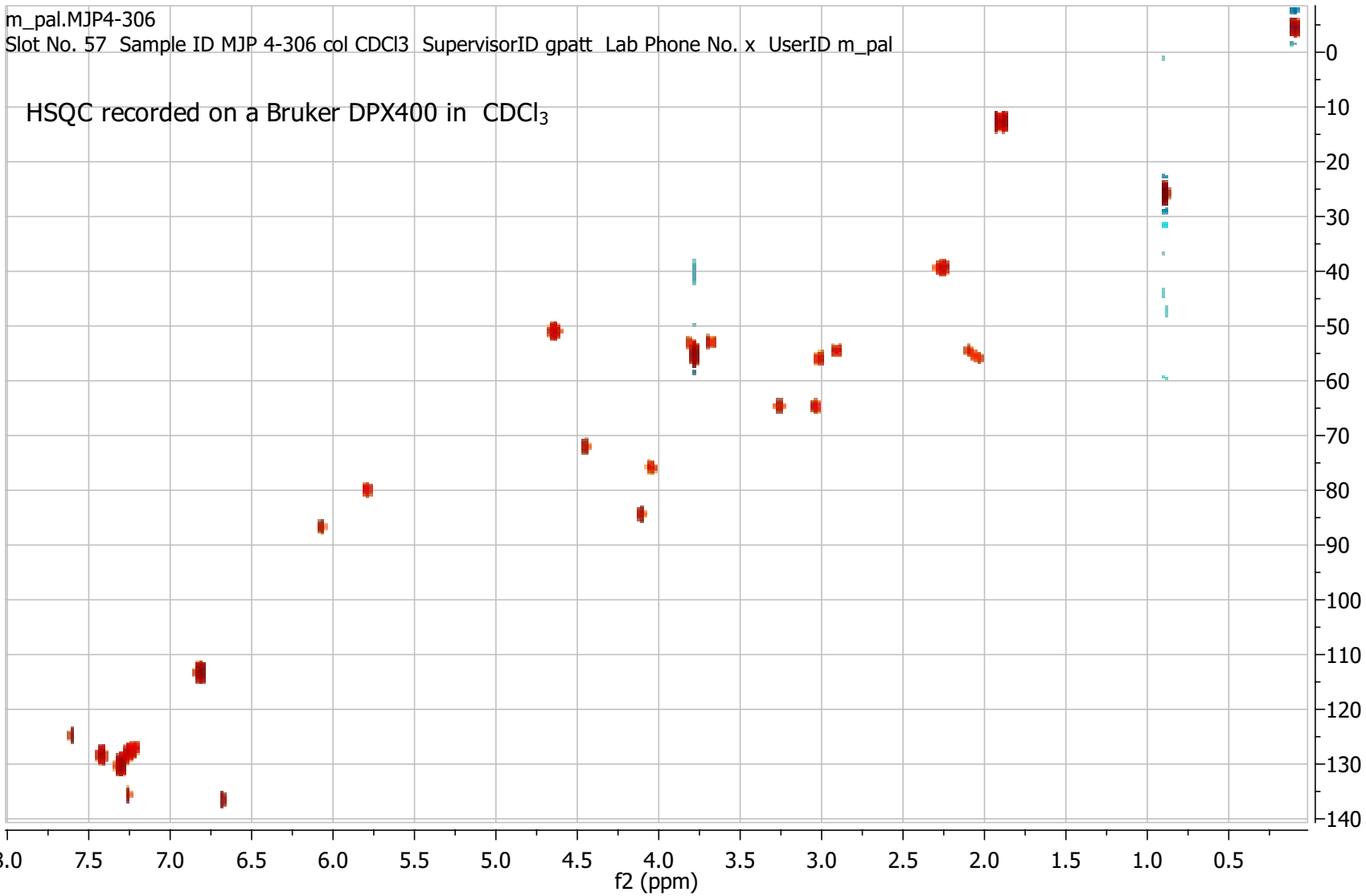
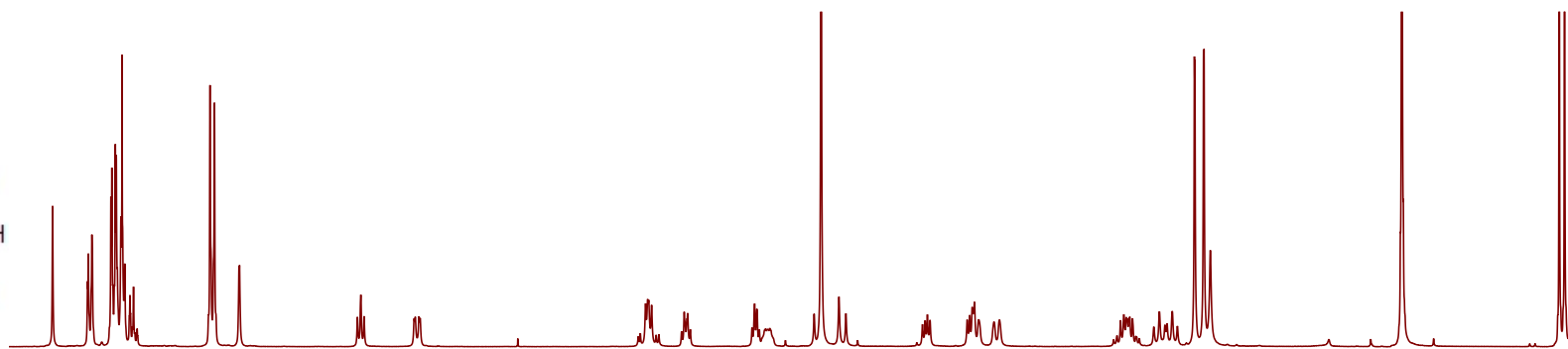
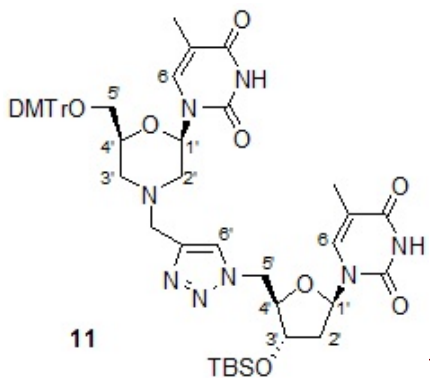
m_pal.MJP4-306

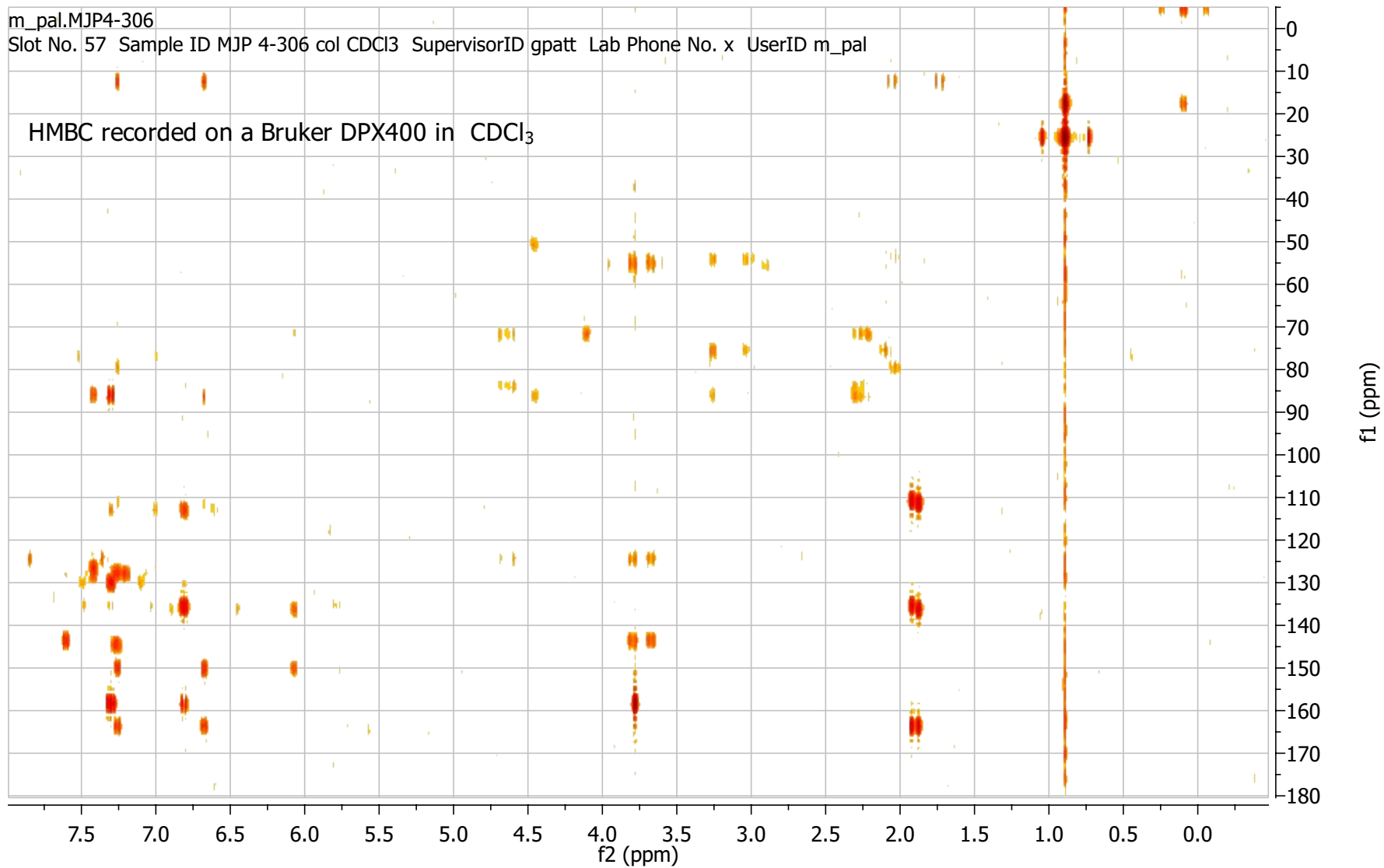
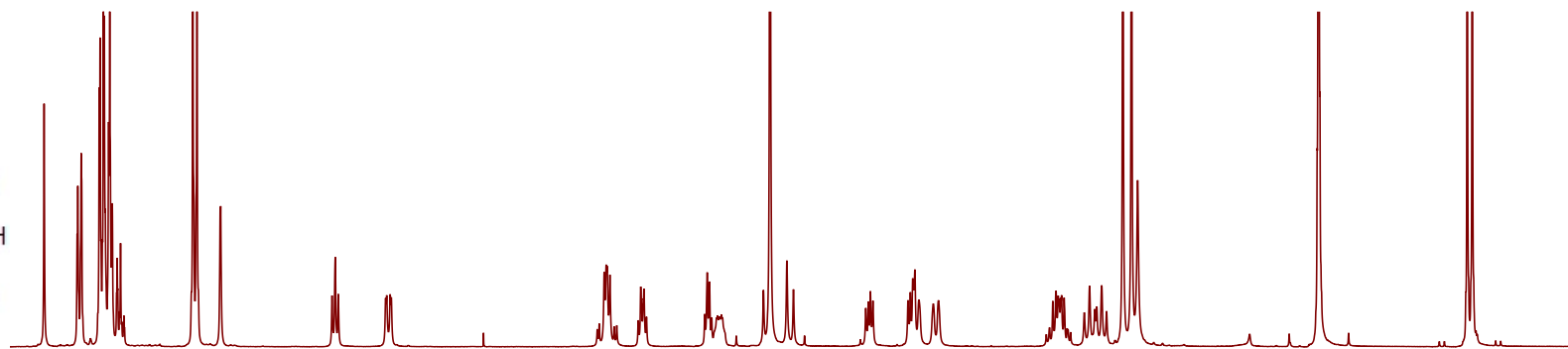
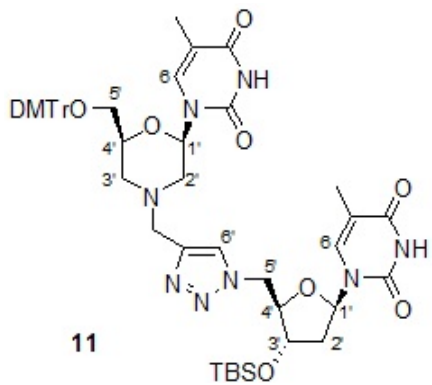
Slot No. 57 Sample ID MJP 4-306 col CDCl₃ SupervisorID gpatt Lab Phone No. x UserID m_pal

Recorded on a Bruker DPX400 in CDCl₃
DEPT135 NMR recorded at 101 MHz









m_pal.mjp4-309

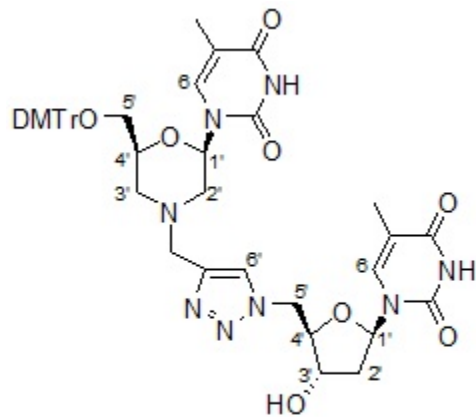
UserID m_pal

SampleID mjp4-309

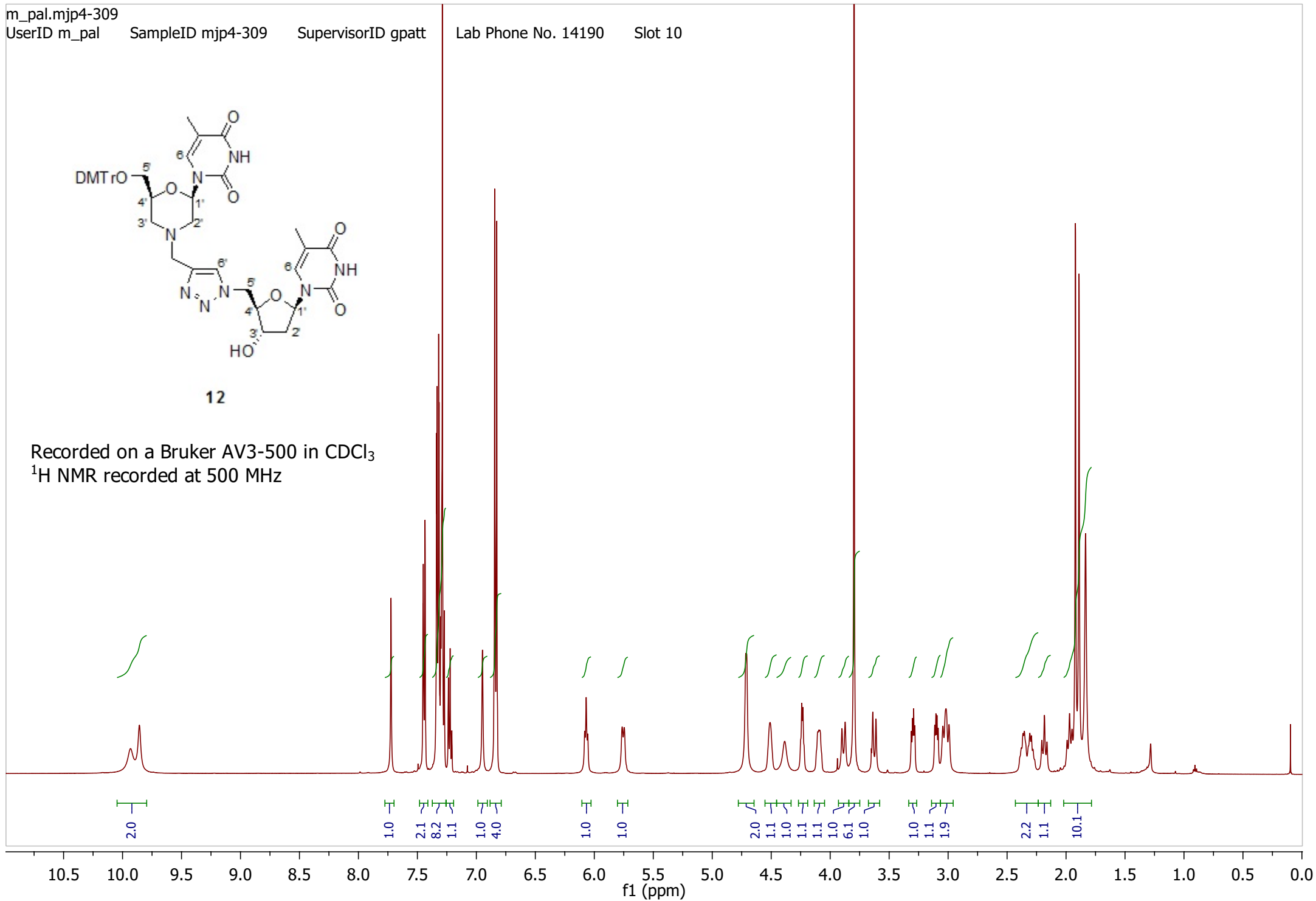
SupervisorID gpatt

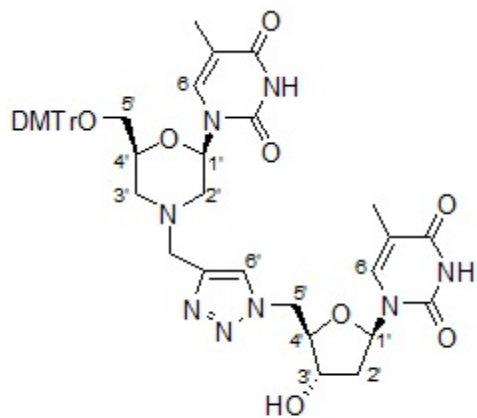
Lab Phone No. 14190

Slot 10

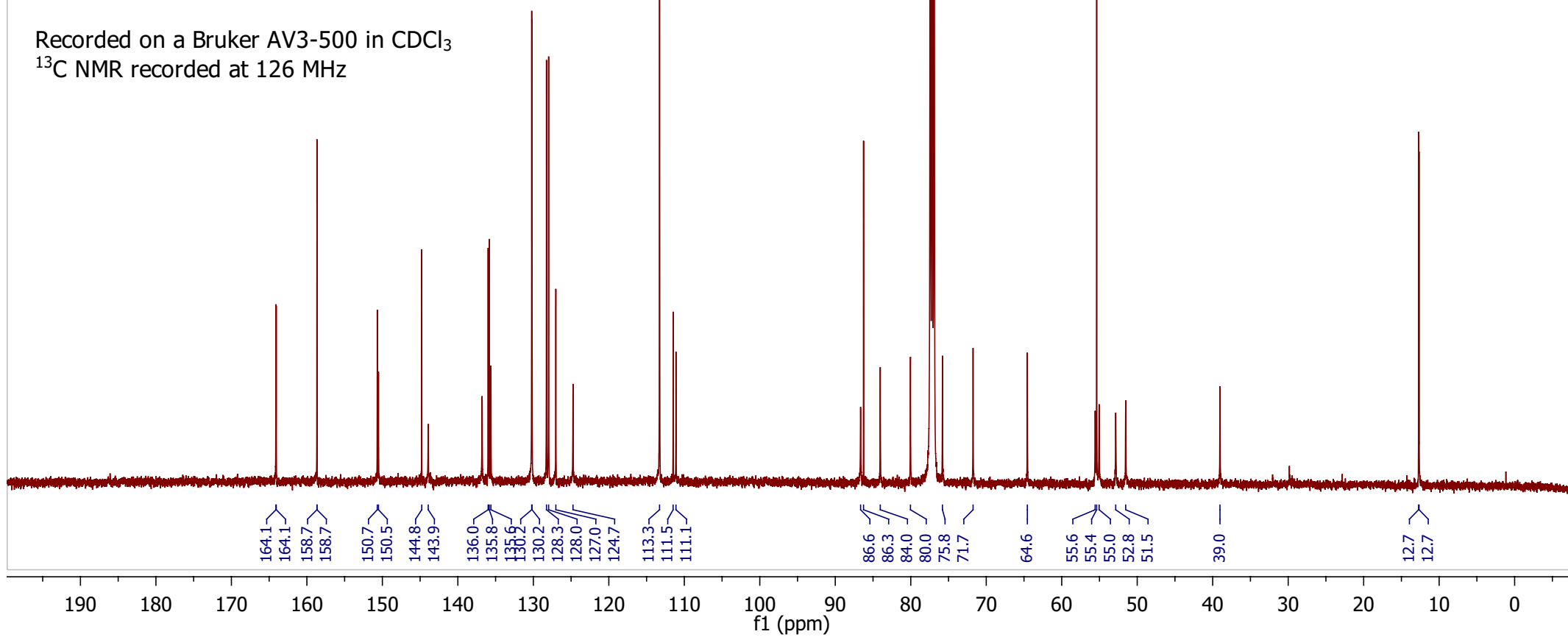


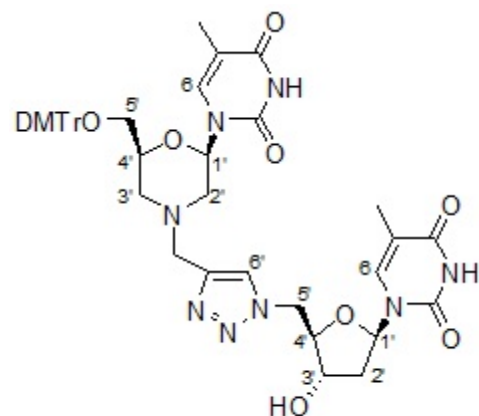
Recorded on a Bruker AV3-500 in CDCl₃
¹H NMR recorded at 500 MHz



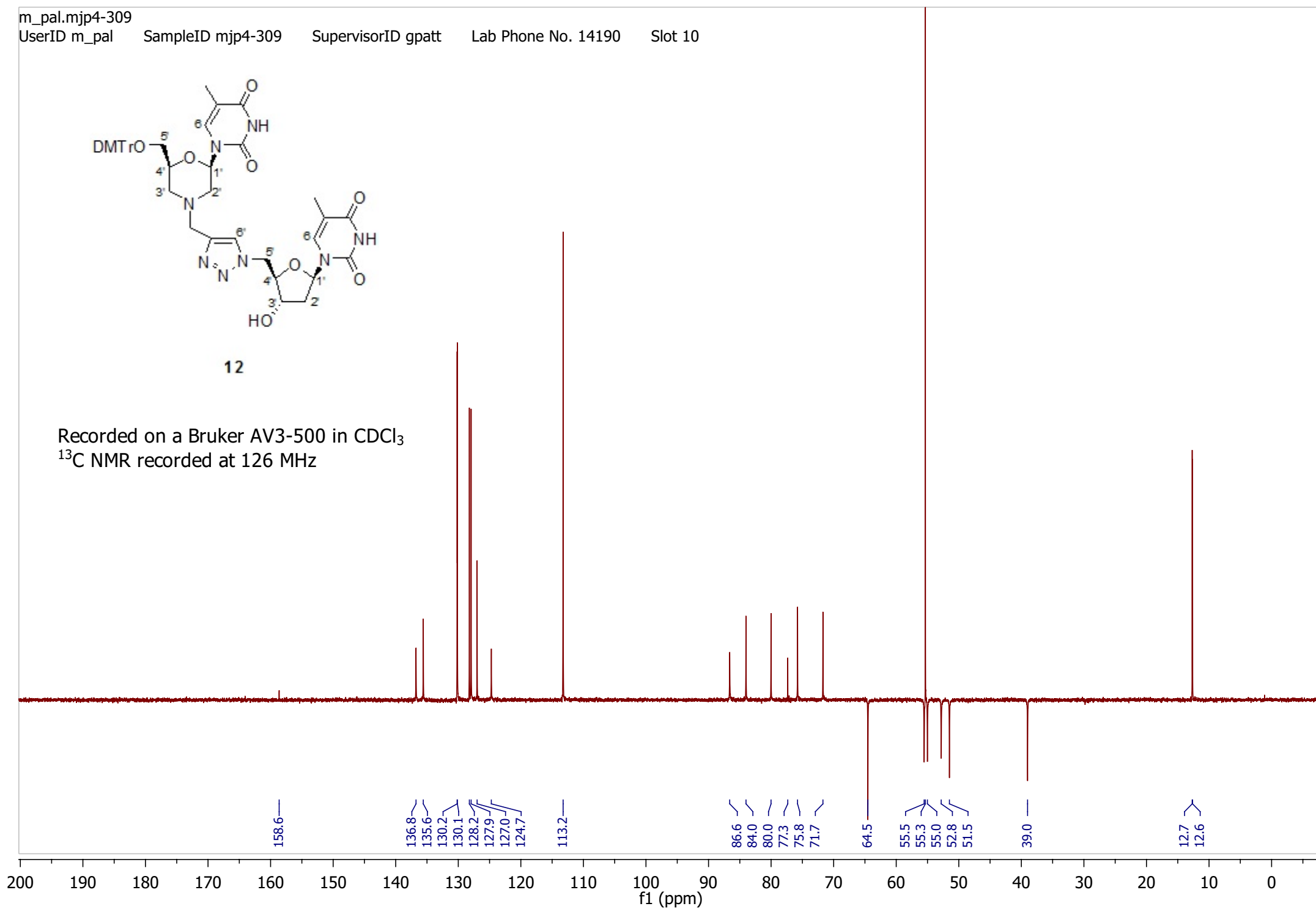


12

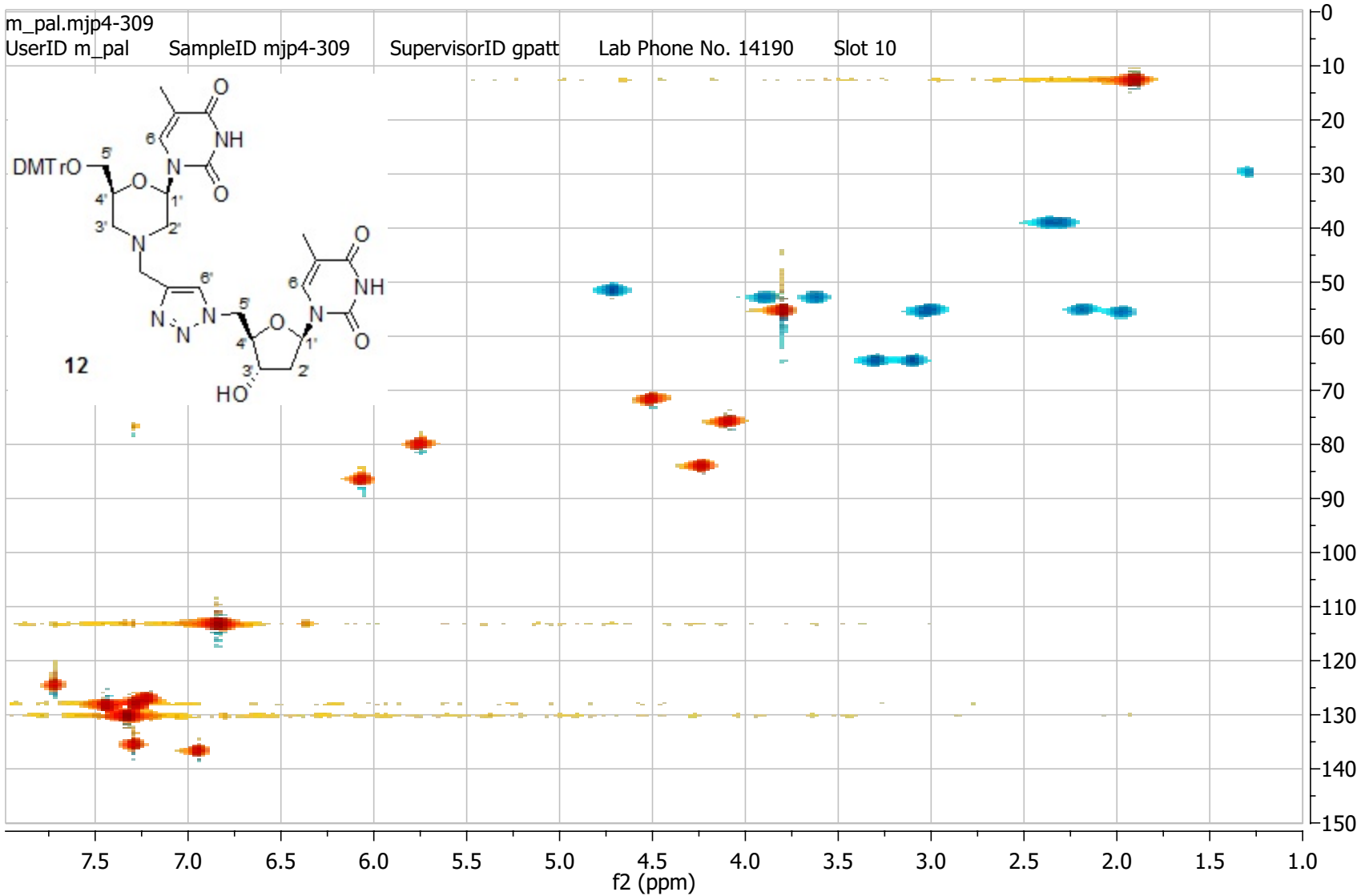
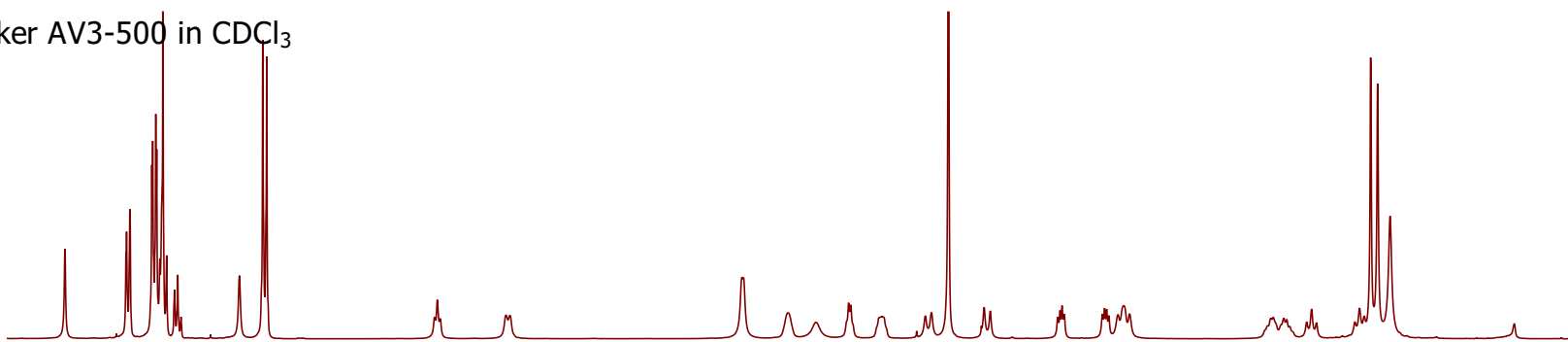
Recorded on a Bruker AV3-500 in CDCl₃¹³C NMR recorded at 126 MHz



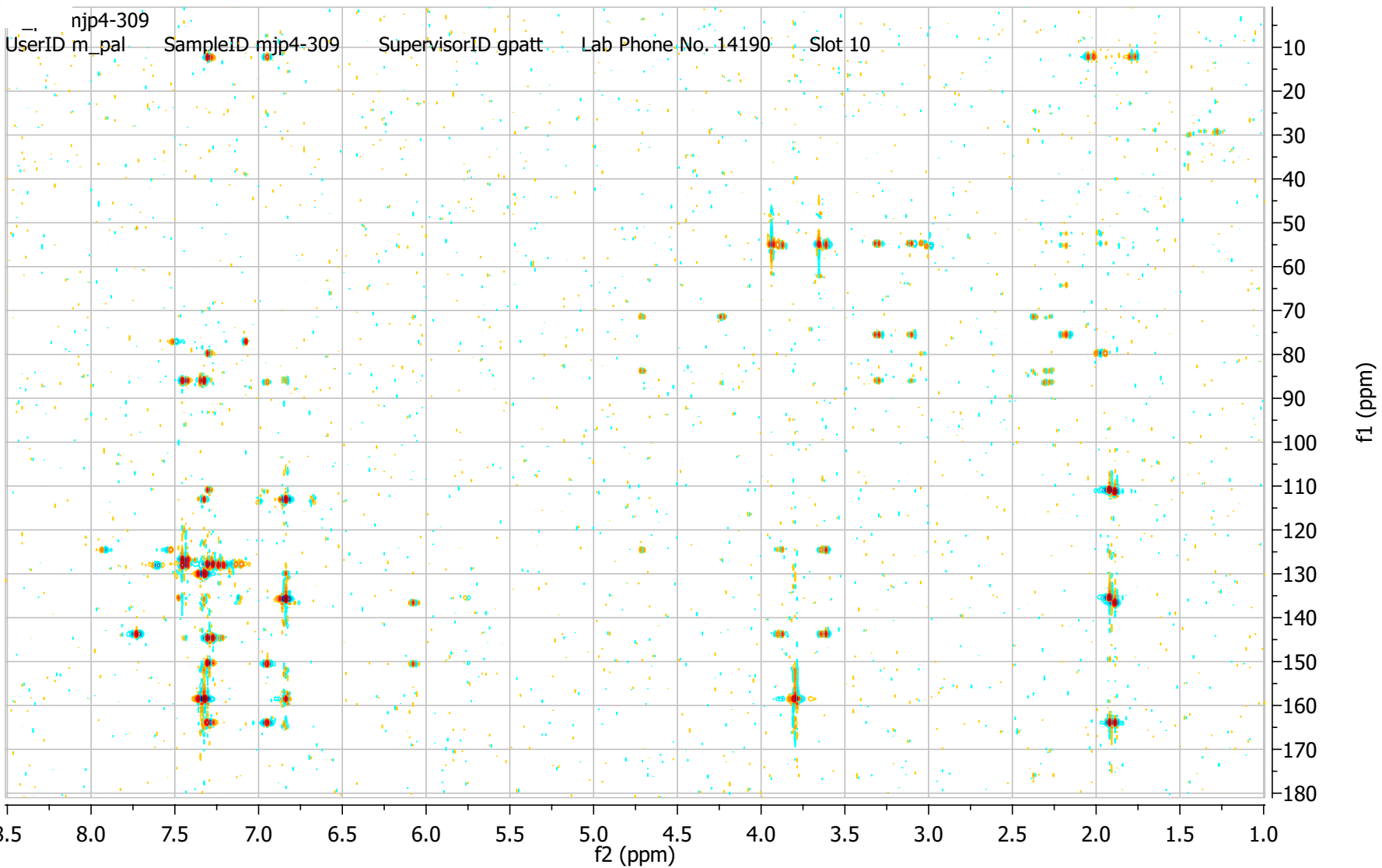
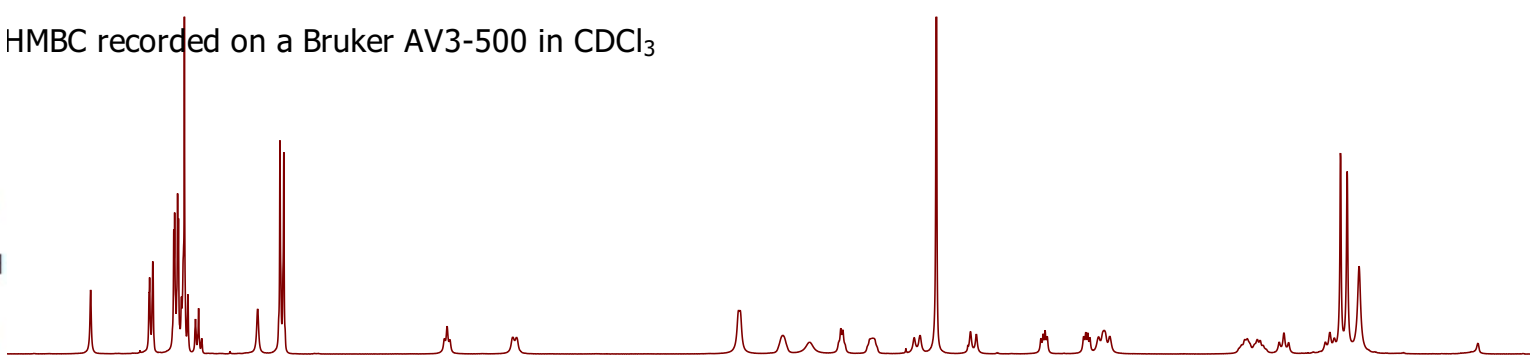
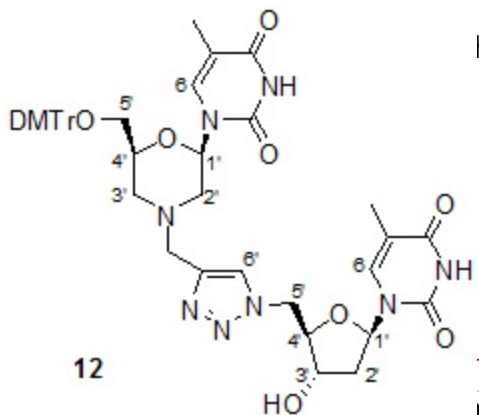
Recorded on a Bruker AV3-500 in CDCl₃
¹³C NMR recorded at 126 MHz



HSQC recorded on a Bruker AV3-500 in CDCl₃



HMBC recorded on a Bruker AV3-500 in CDCl₃



m_pal.4-334

UserID m_pal

SampleID 4-334 a3a5

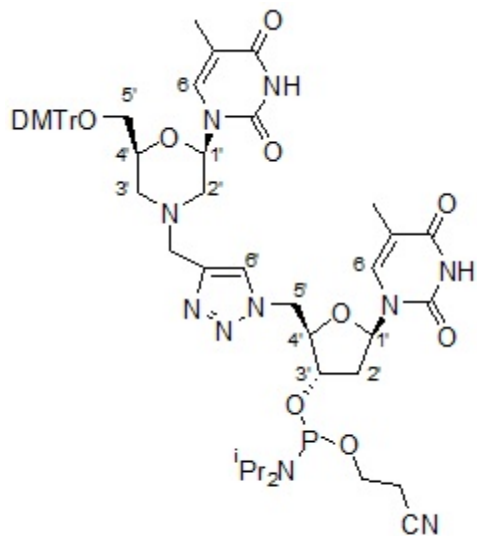
SupervisorID gpatt

Lab Phone No. 14190

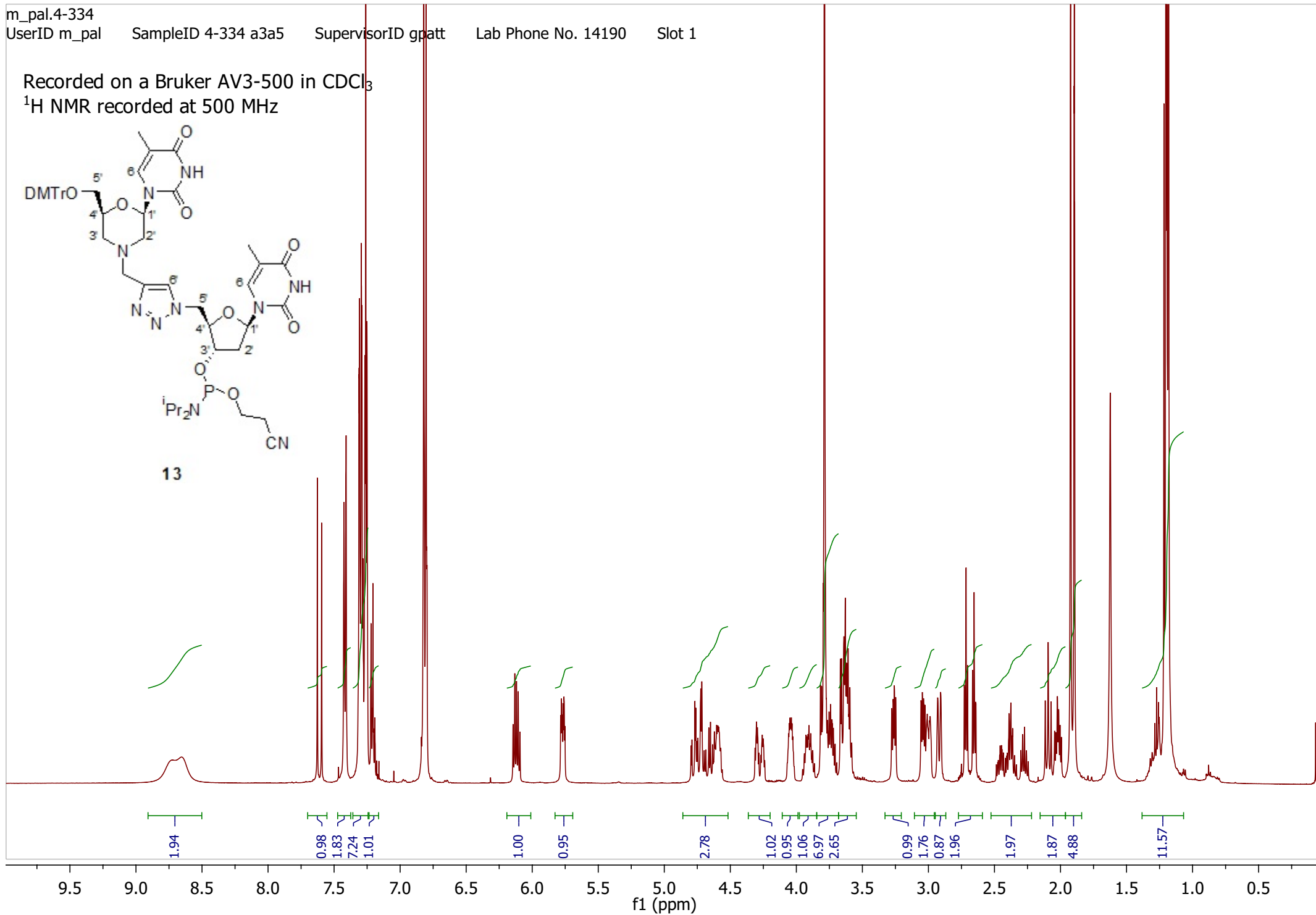
Slot 1

Recorded on a Bruker AV3-500 in CDCl₃

¹H NMR recorded at 500 MHz



13



m_pal.4-334

UserID m_pal

SampleID 4-334 a3a5

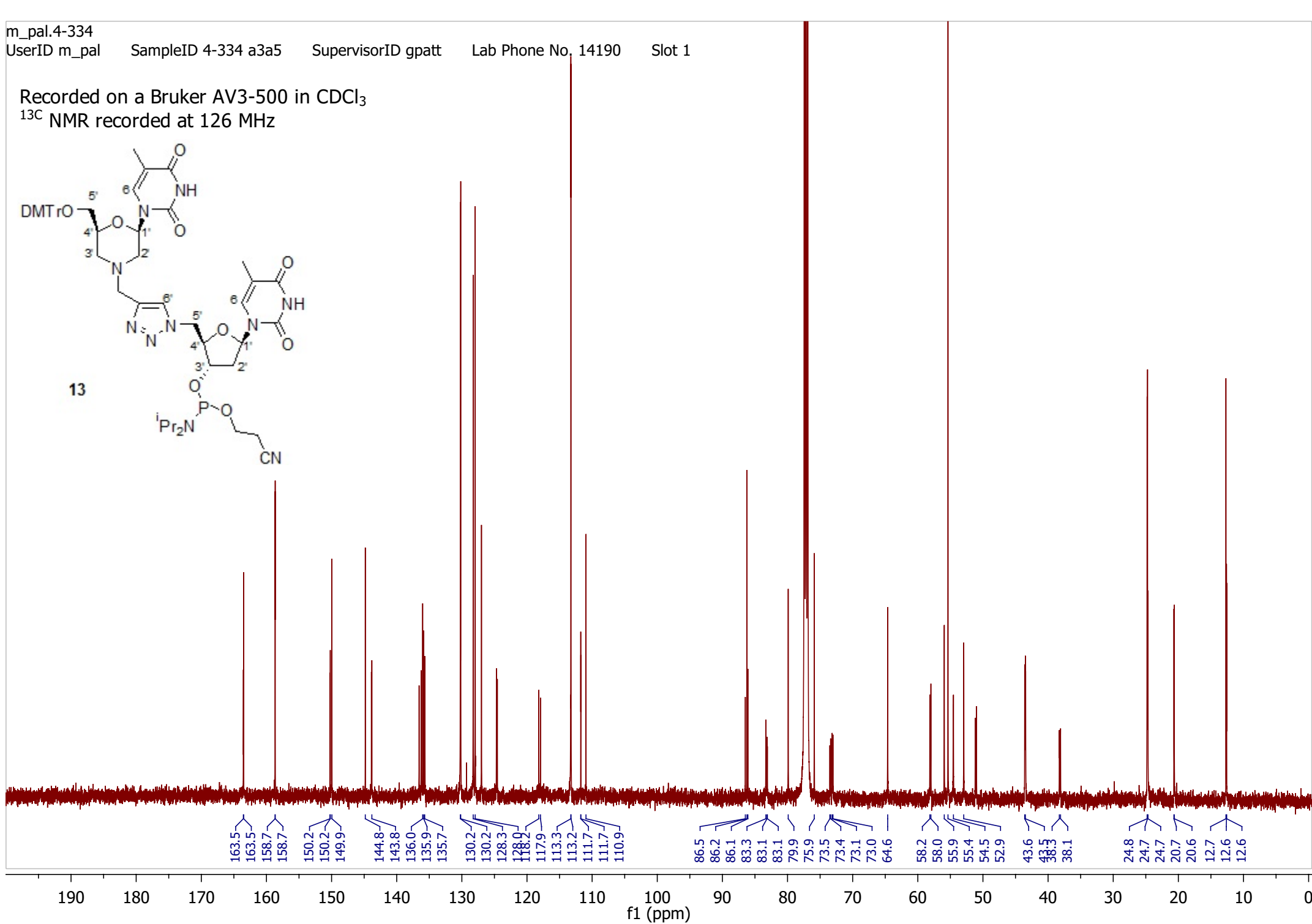
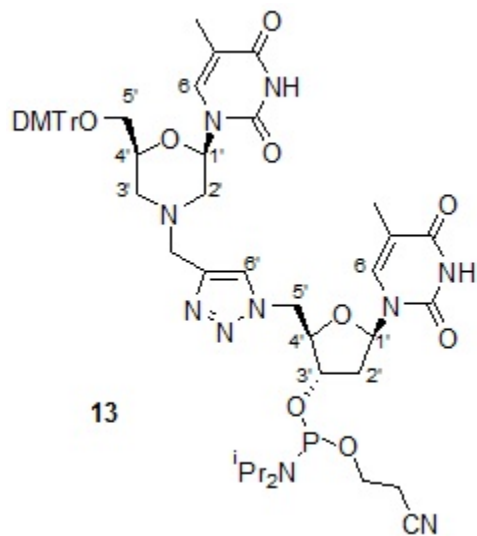
SupervisorID gpatt

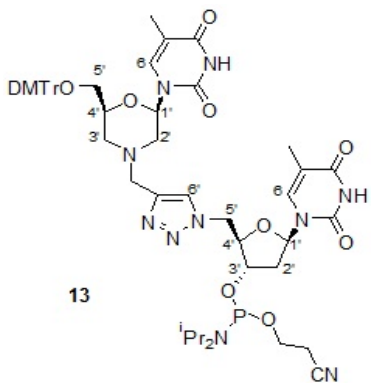
Lab Phone No. 14190

Slot 1

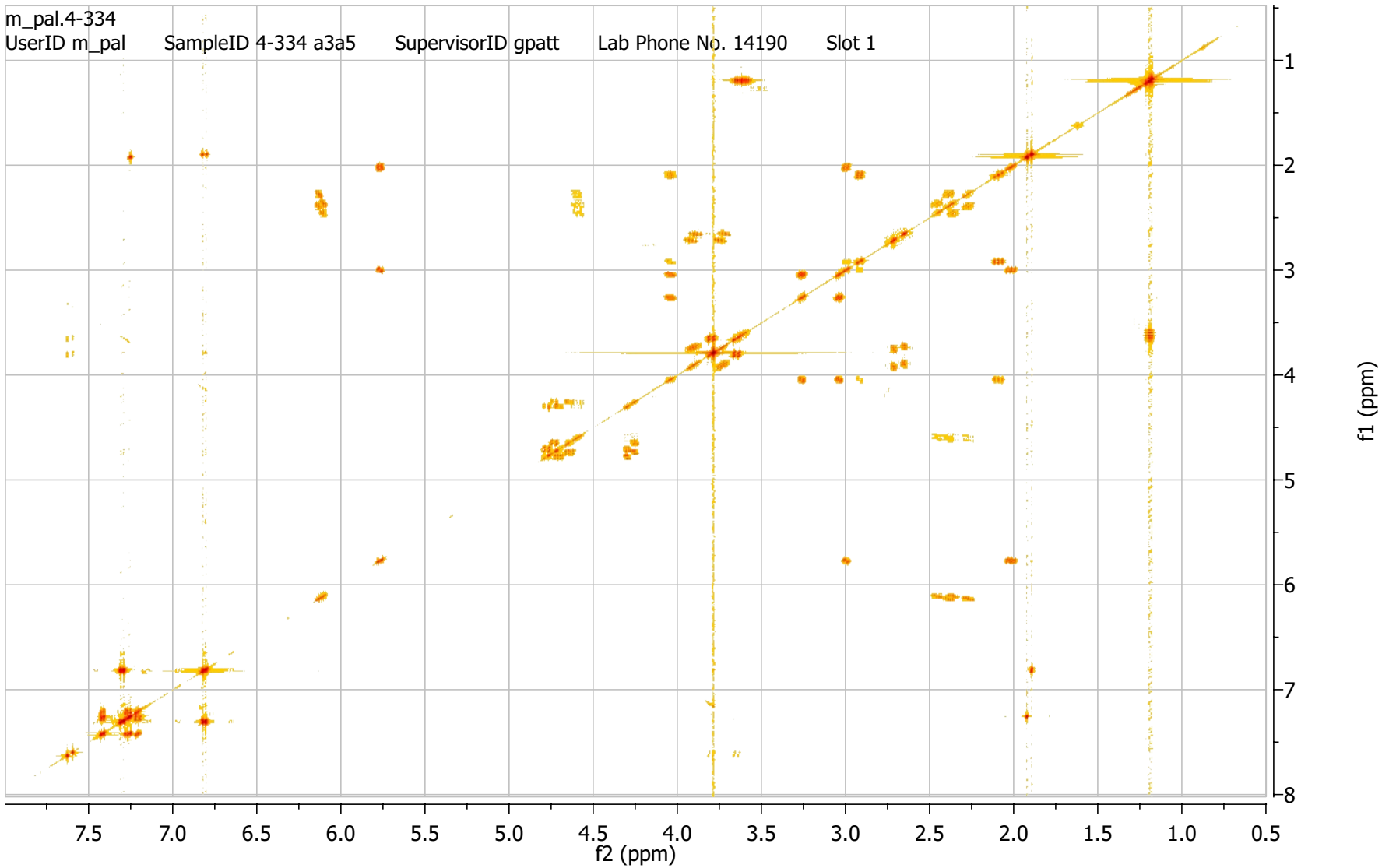
Recorded on a Bruker AV3-500 in CDCl₃

¹³C NMR recorded at 126 MHz

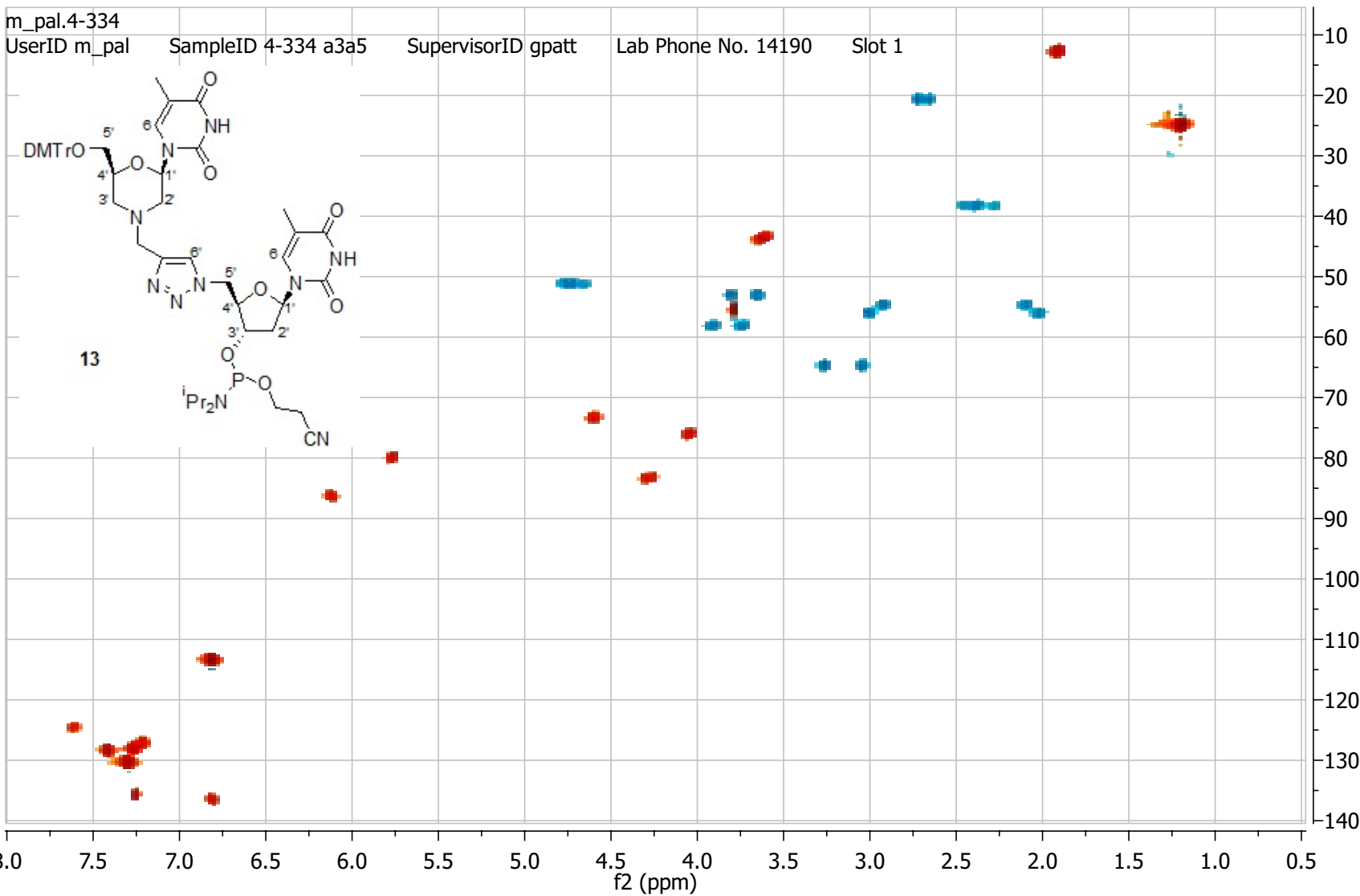
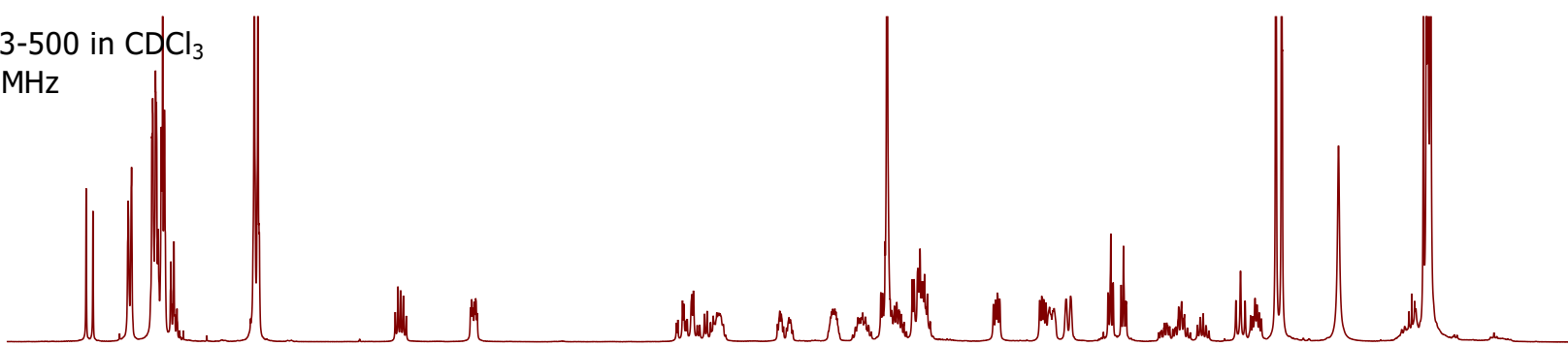


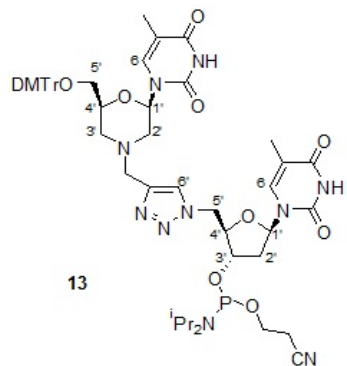


Recorded on a Bruker AV3-500 in $CDCl_3$
 ^{13}C NMR recorded at 126 MHz



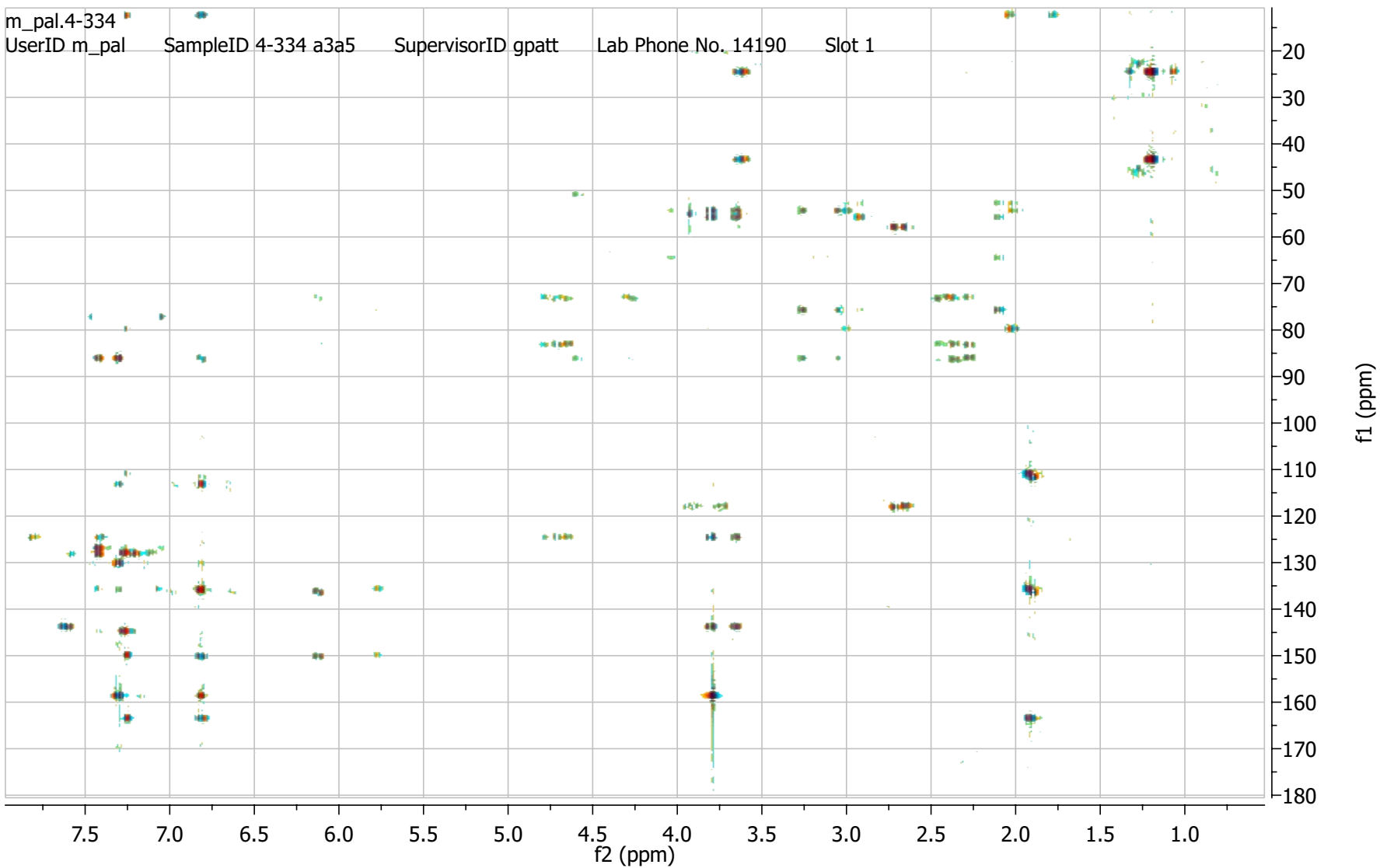
Recorded on a Bruker AV3-500 in CDCl₃
¹³C NMR recorded at 126 MHz

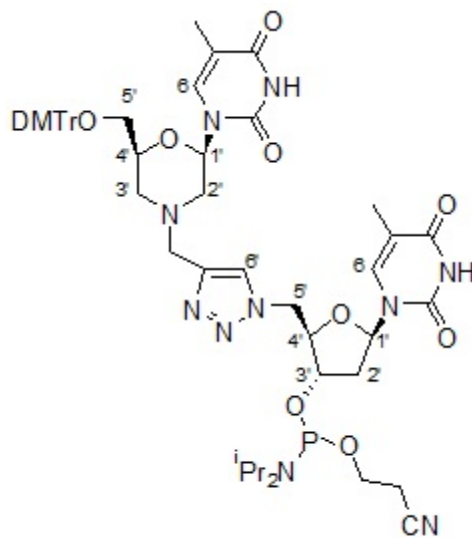




Recorded on a Bruker AV3-500 in CDCl₃

¹³C NMR recorded at 126 MHz



Recorded on a Bruker DPX400 in CDCl₃³¹P NMR recorded at 162 MHz**13**

149.2

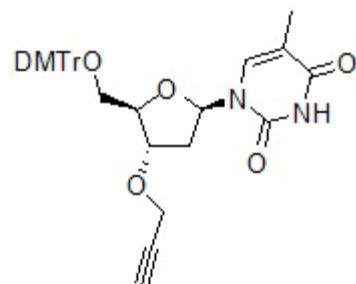
200 150 100 50 0 f1 (ppm) -50 -100 -150 -200

m_pal.MJP5-468

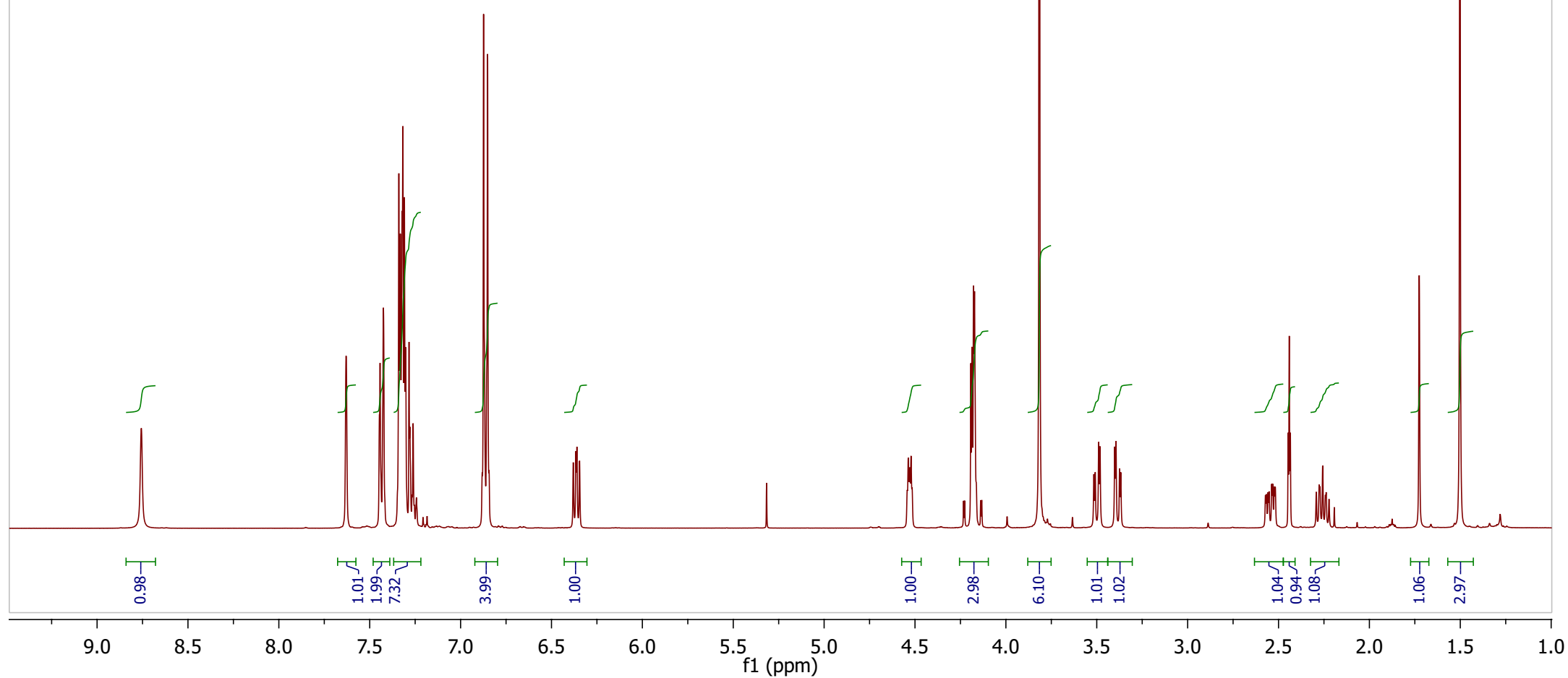
UserID m_pal SampleID MJP 5-469 col SupervisorID hayes Lab Phone No. 13536 Slot Number 38

Recorded on a Bruker AV-3-400 in CDCl₃

¹H NMR recorded at 400 MHz



15

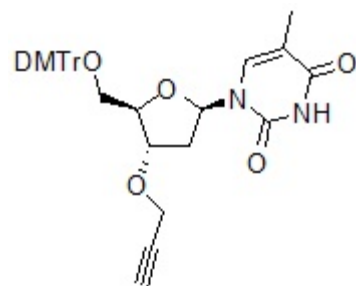


m_pal.MJP5-468

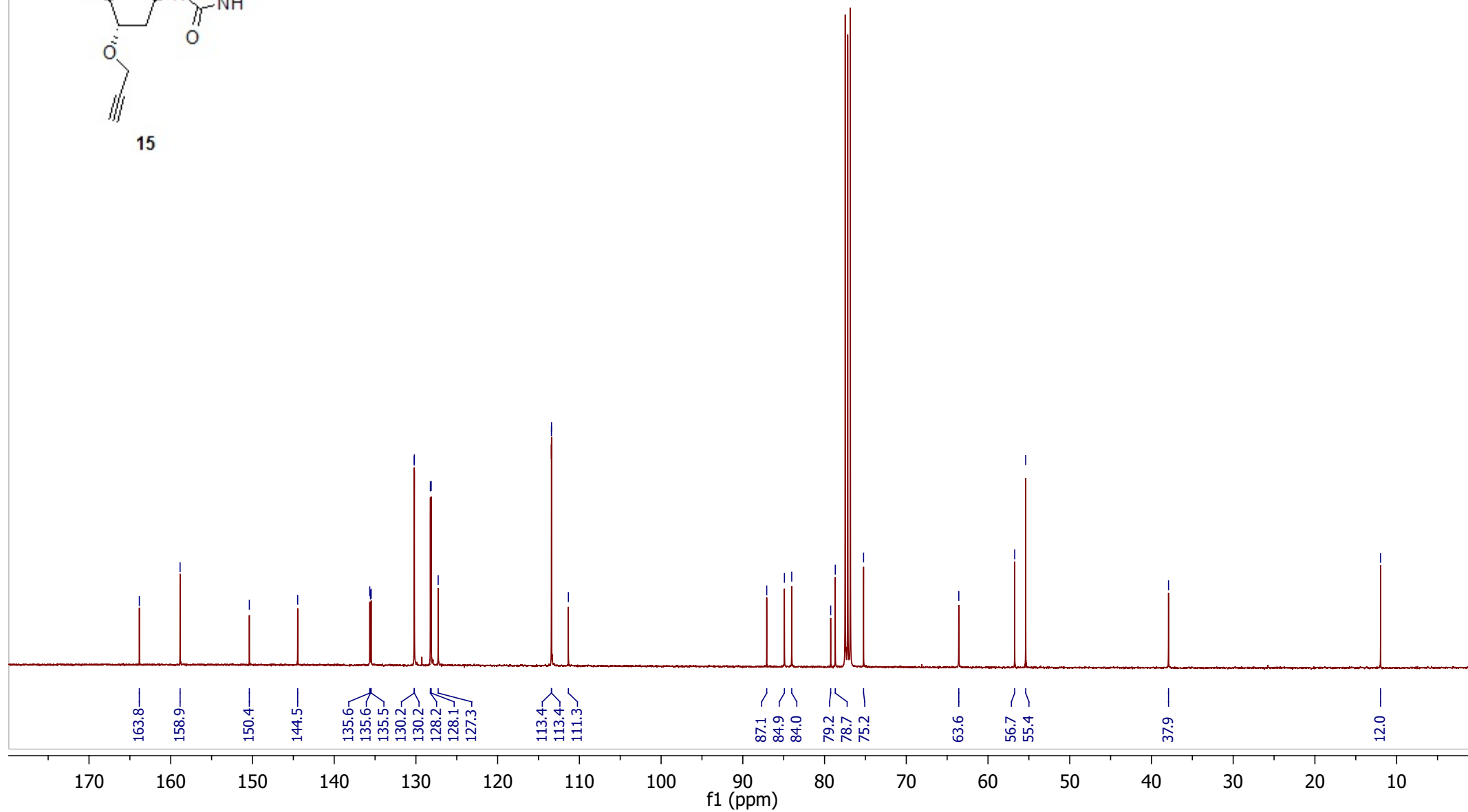
UserID m_pal SampleID MJP 5-469 col SupervisorID hayes Lab Phone No. 13536 Slot Number 38

Recorded on a Bruker AV-3-400 in CDCl₃

¹³C NMR recorded at 101 MHz



15

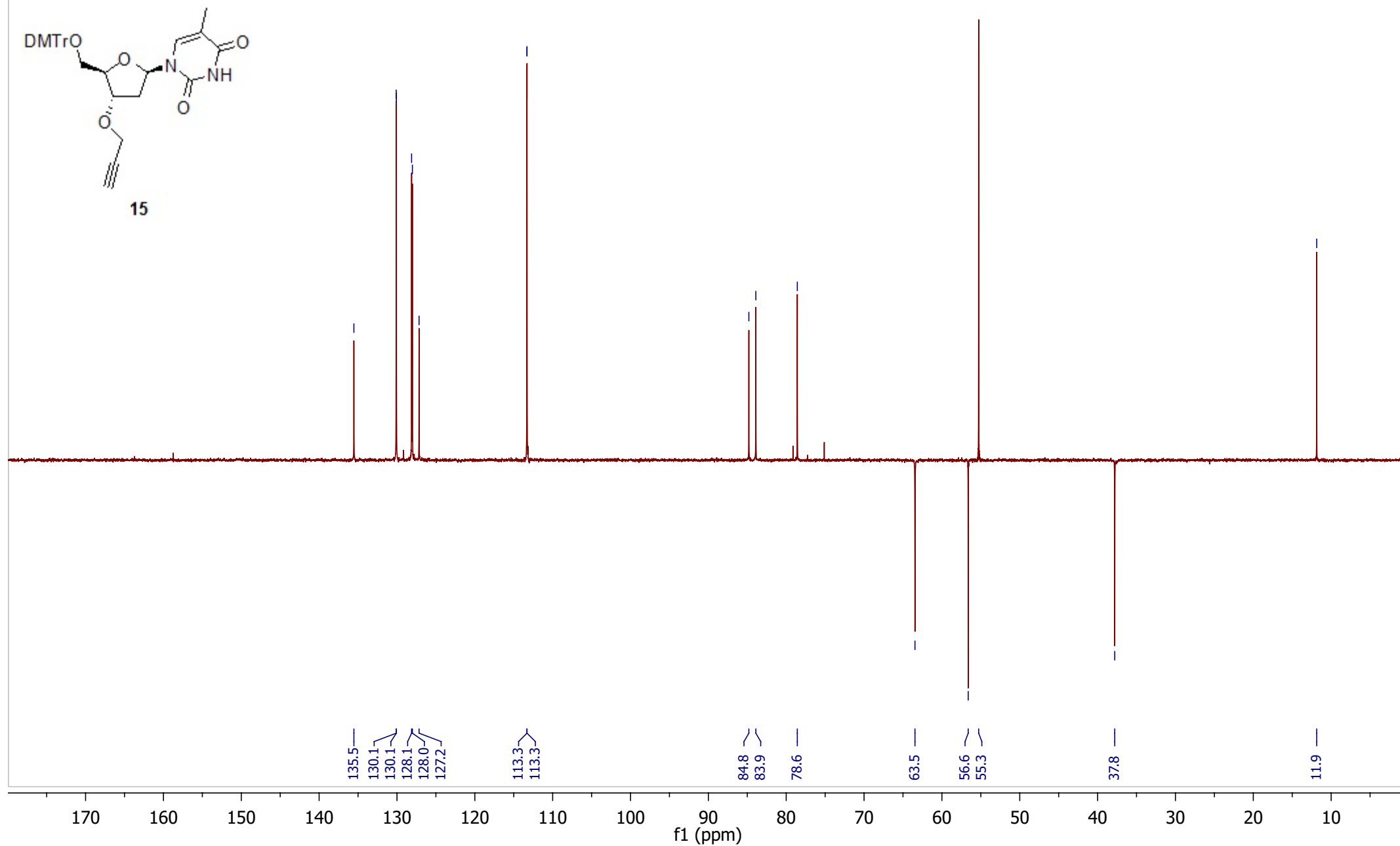


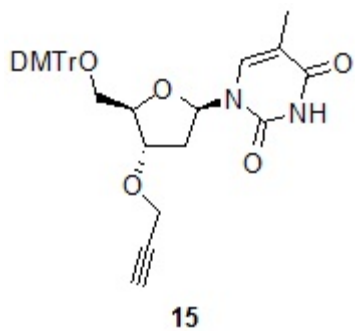
m_pal.MJP5-468

UserID m_pal SampleID MJP 5-469 col SupervisorID hayes Lab Phone No. 13536 Slot Number 38

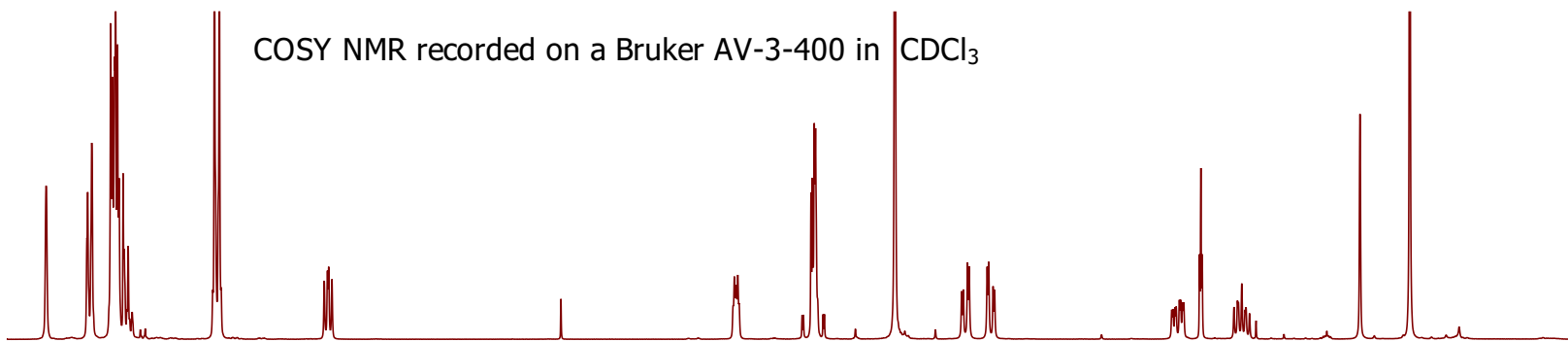
Recorded on a Bruker AV-3-400 in CDCl₃

DEPT 135 NMR recorded at 101 MHz





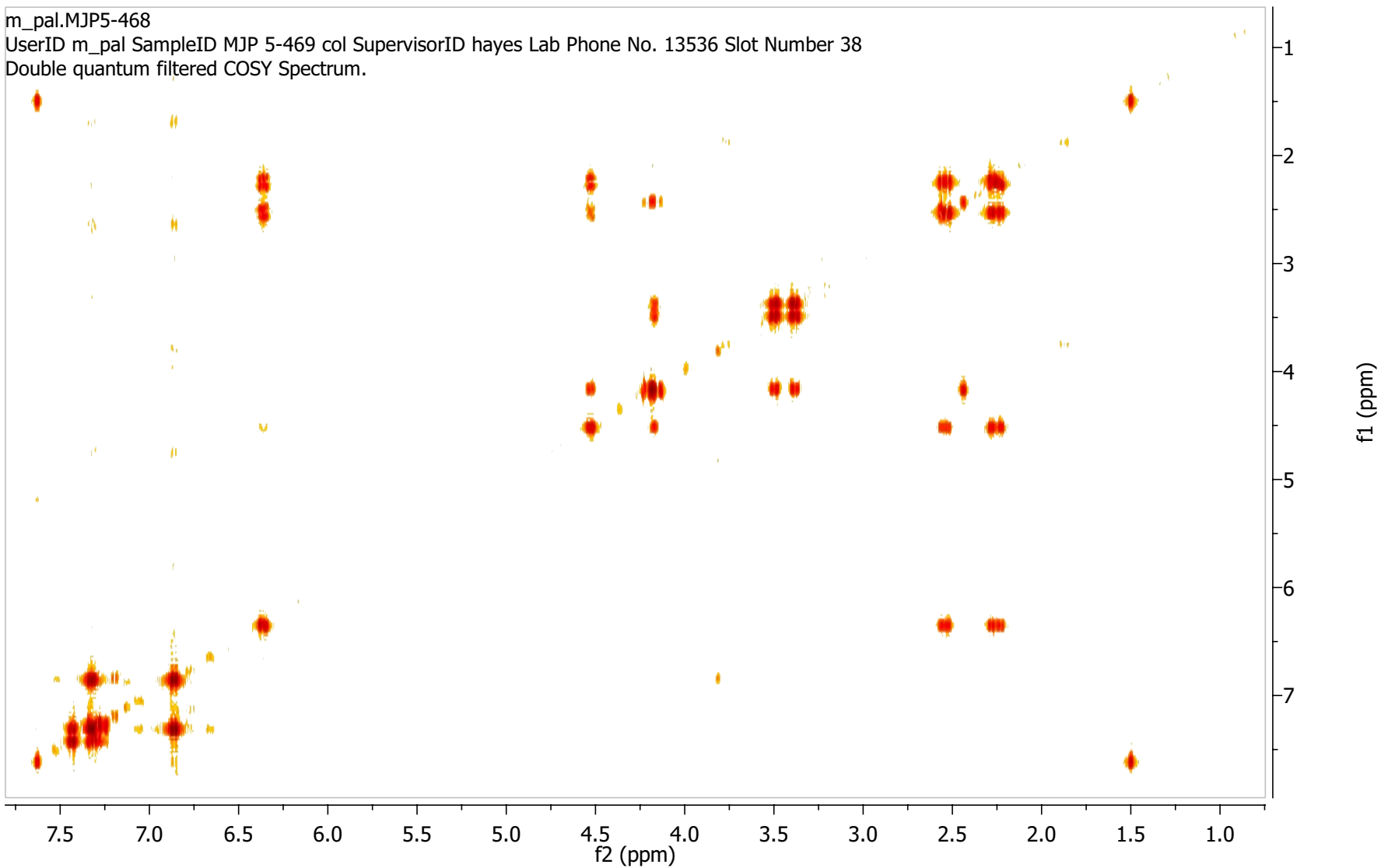
COSY NMR recorded on a Bruker AV-3-400 in CDCl₃

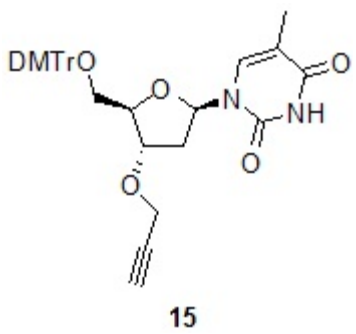


m_pal.MJP5-468

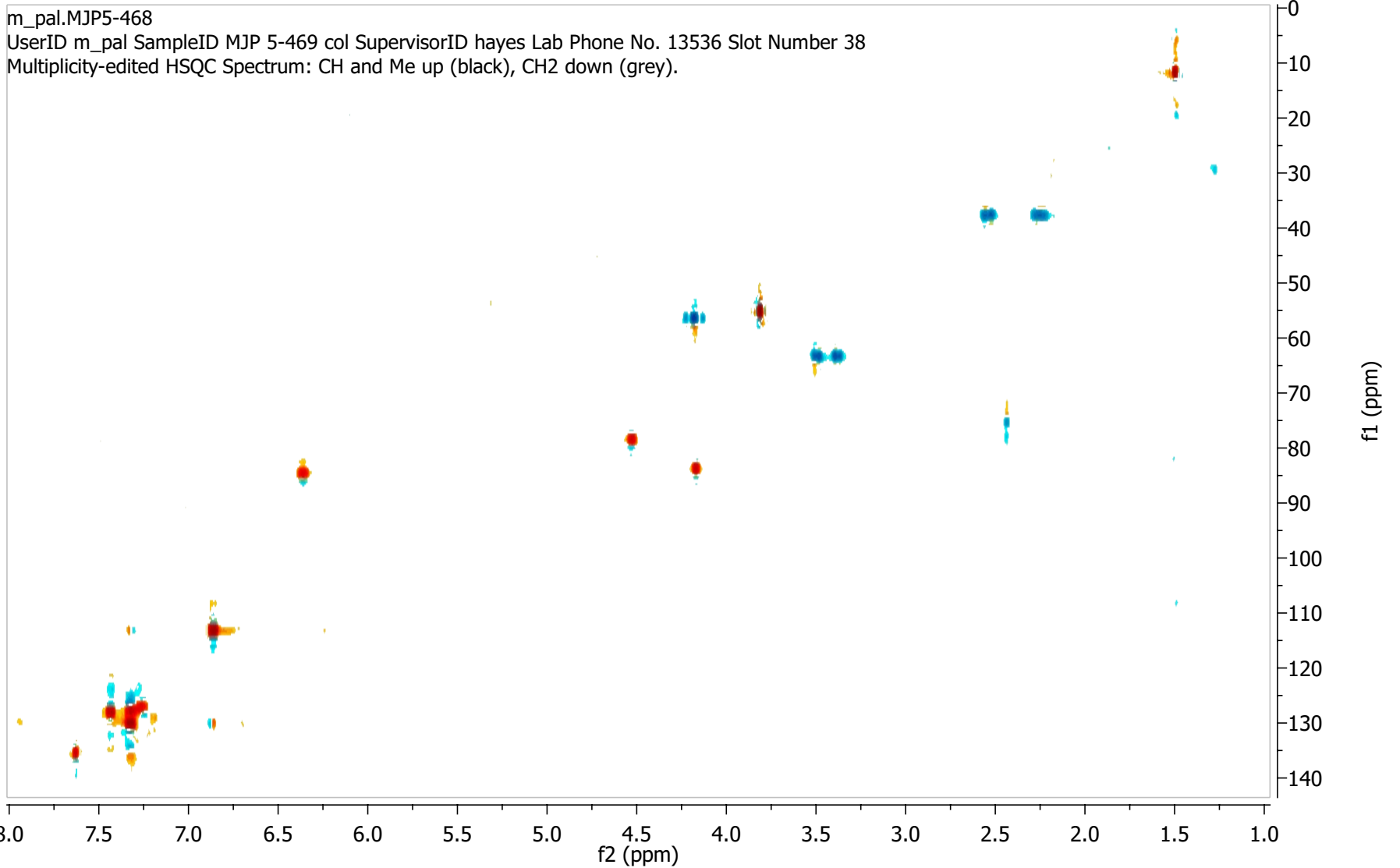
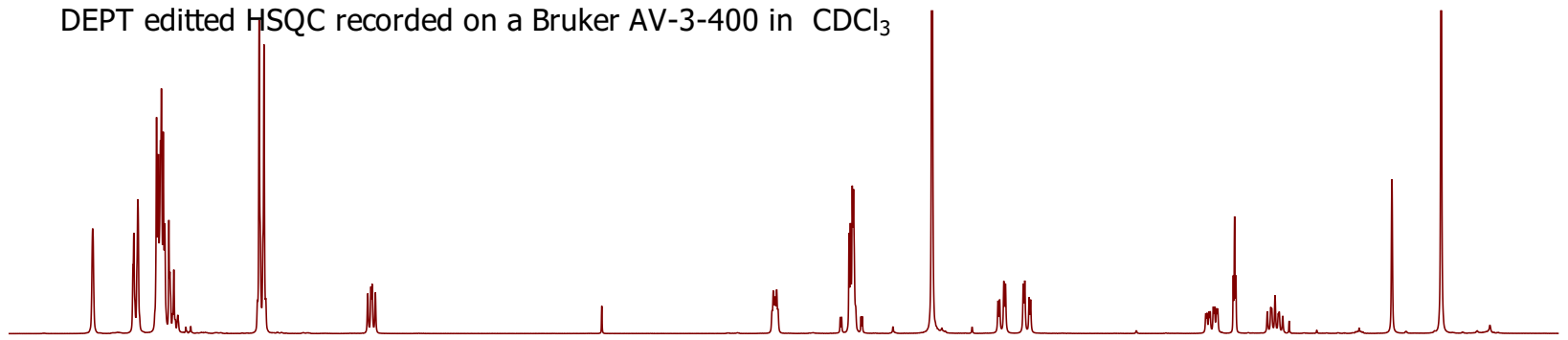
UserID m_pal SampleID MJP 5-469 col SupervisorID hayes Lab Phone No. 13536 Slot Number 38

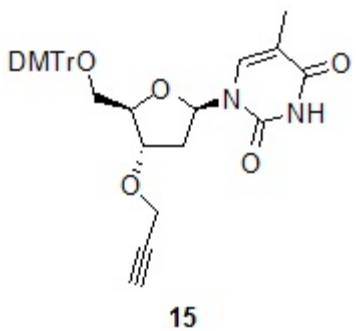
Double quantum filtered COSY Spectrum.



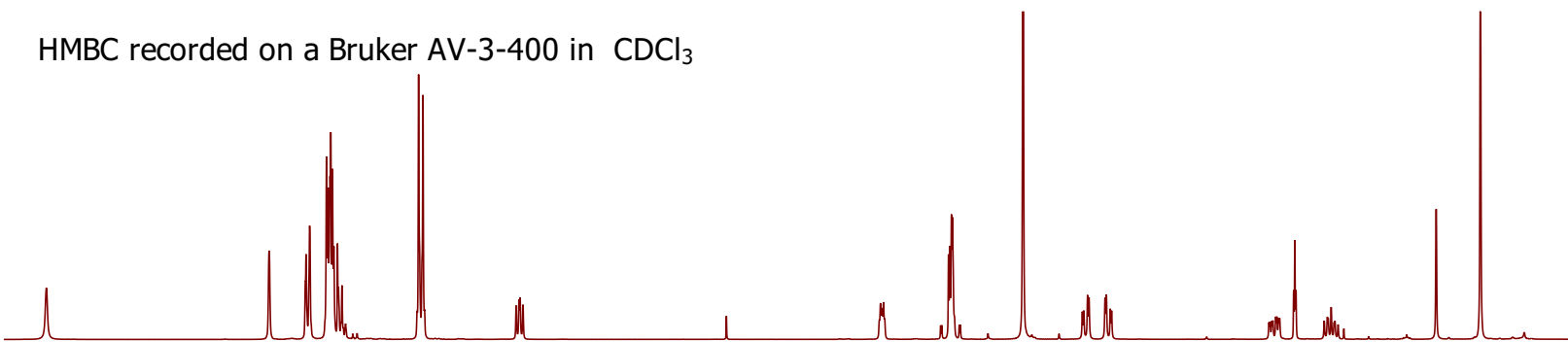


DEPT edited HSQC recorded on a Bruker AV-3-400 in CDCl₃





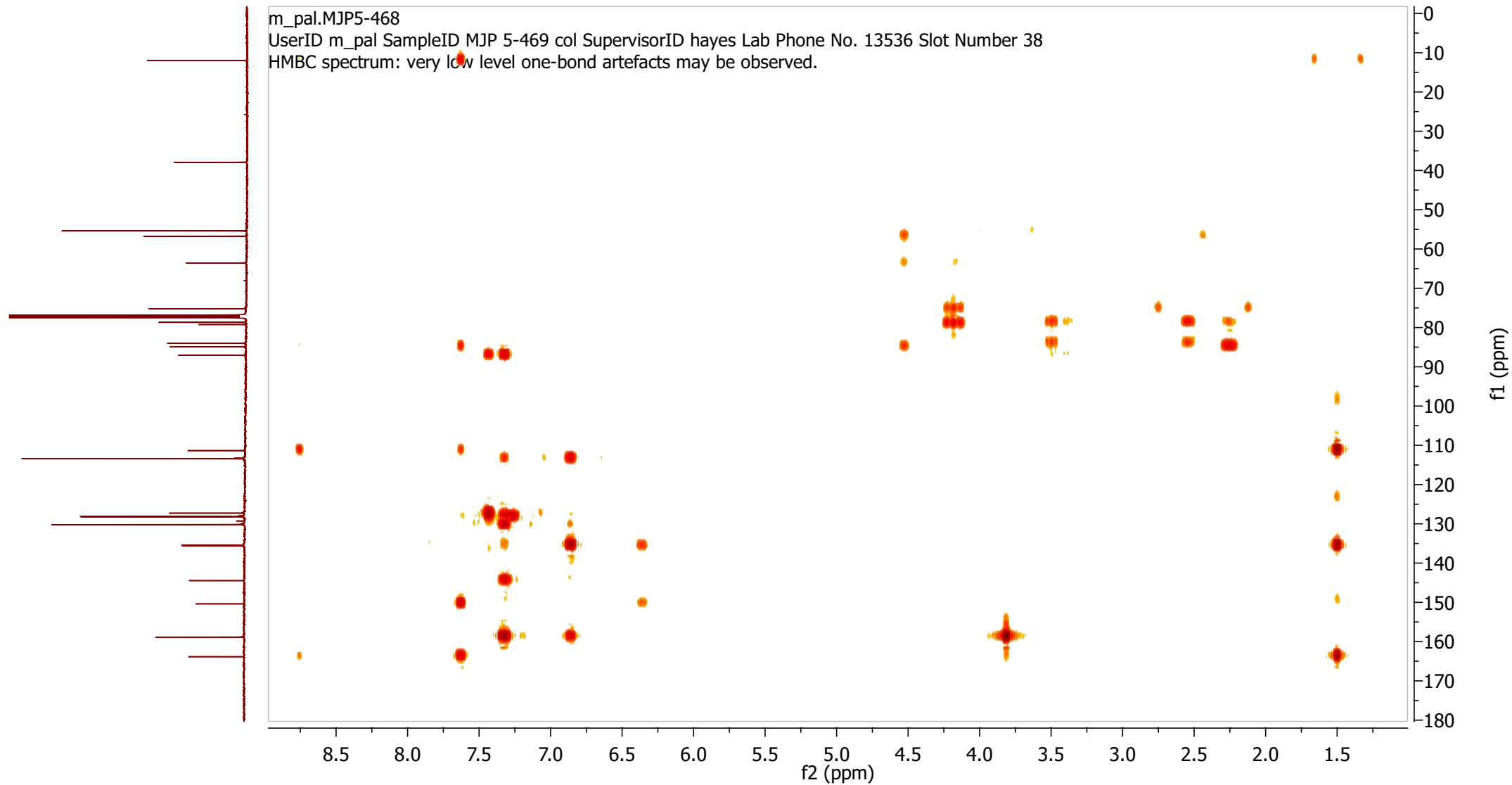
HMBC recorded on a Bruker AV-3-400 in CDCl₃



m_pal.MJP5-468

UserID m_pal SampleID MJP 5-469 col SupervisorID hayes Lab Phone No. 13536 Slot Number 38

HMBC spectrum: very low level one-bond artefacts may be observed.

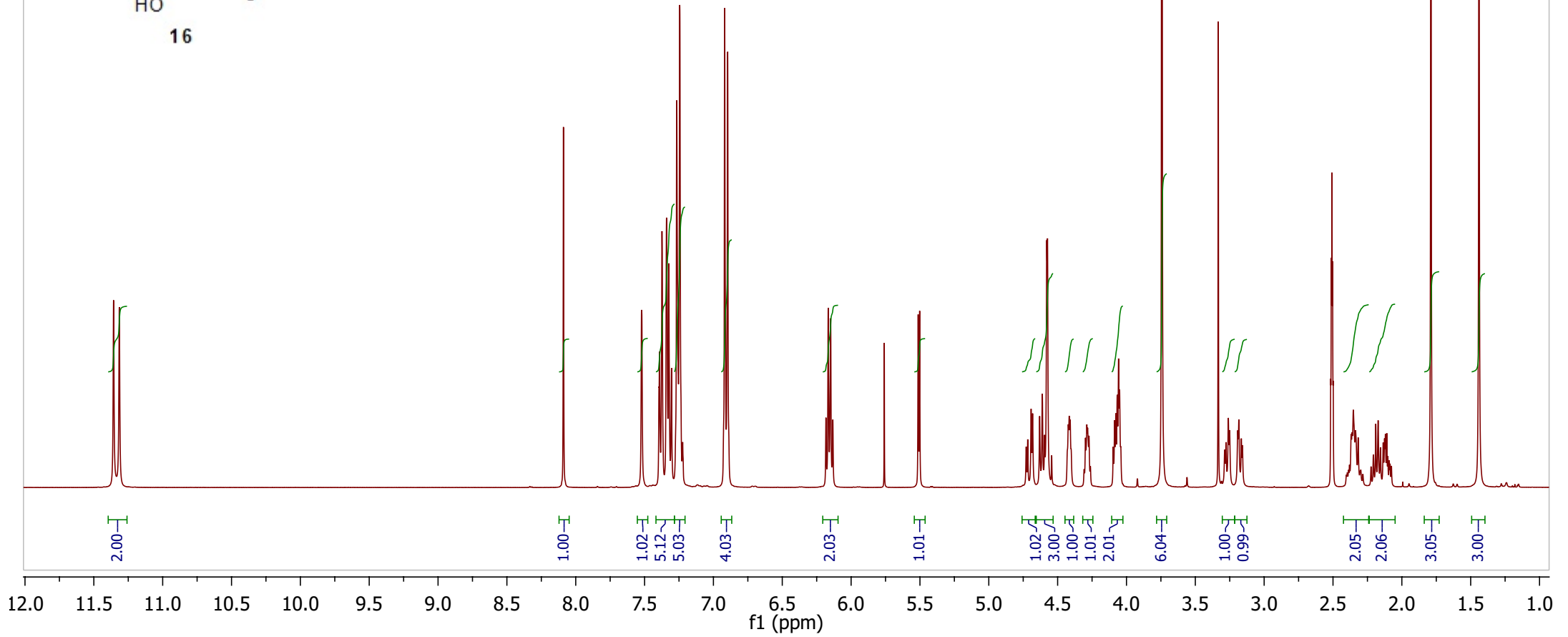
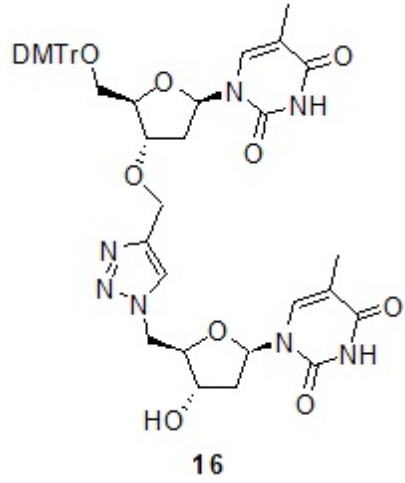


m_pal.MJP5-471

UserID m_pal SampleID MJP 5-471 - col - DMSO SupervisorID hayes Lab Phone No. 13536 Slot Number 13

Recorded on a Bruker AV-3-400 in d6-DMSO

¹H NMR recorded at 400 MHz

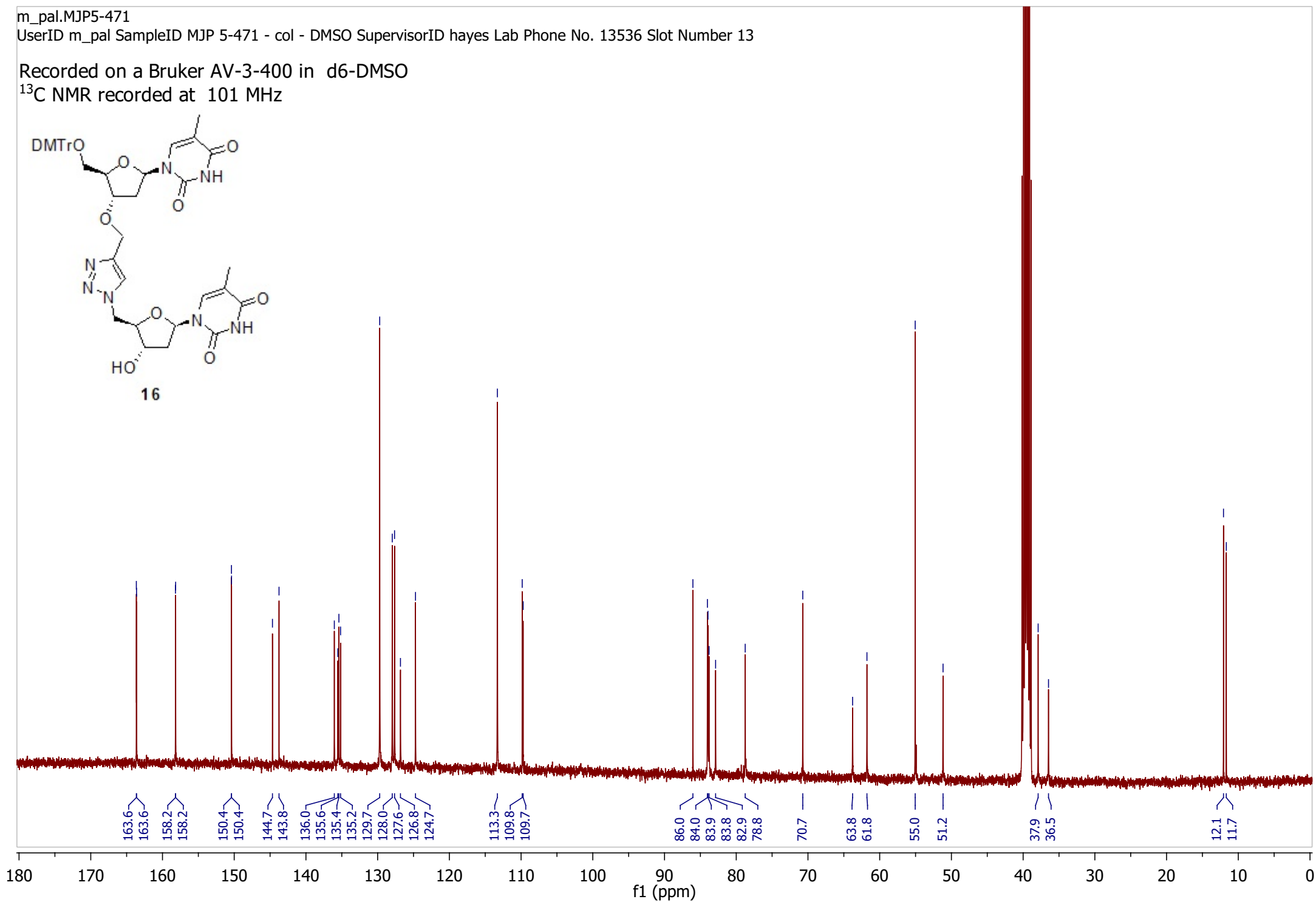
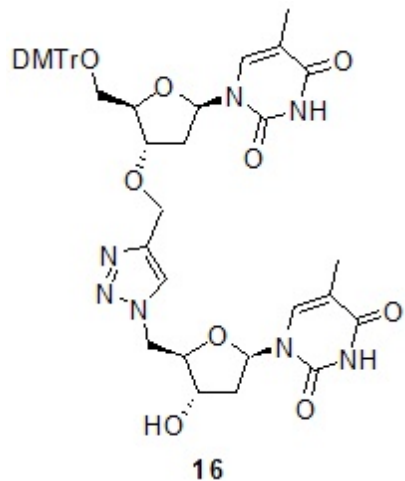


m_pal.MJP5-471

UserID m_pal SampleID MJP 5-471 - col - DMSO SupervisorID hayes Lab Phone No. 13536 Slot Number 13

Recorded on a Bruker AV-3-400 in d6-DMSO

¹³C NMR recorded at 101 MHz

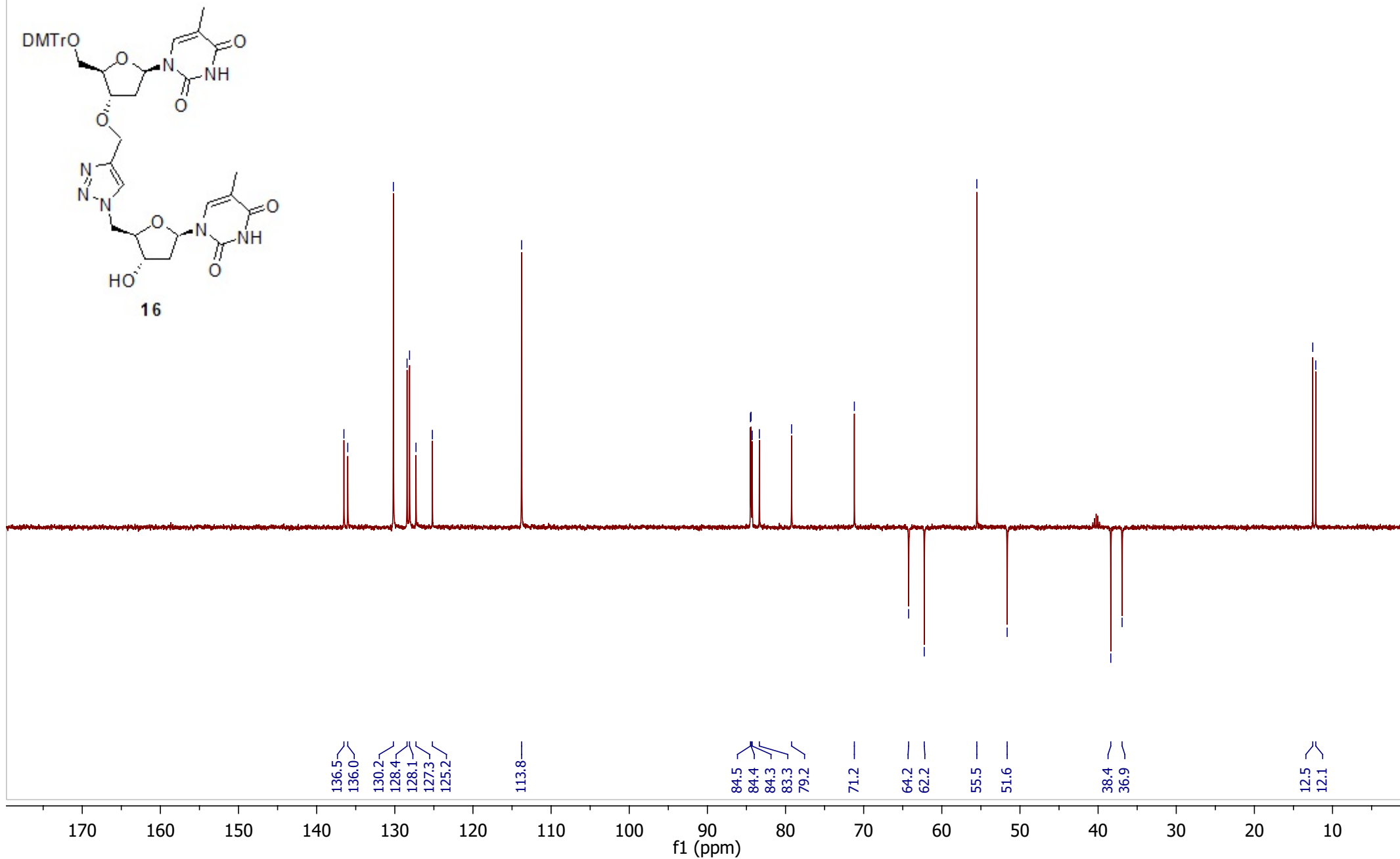


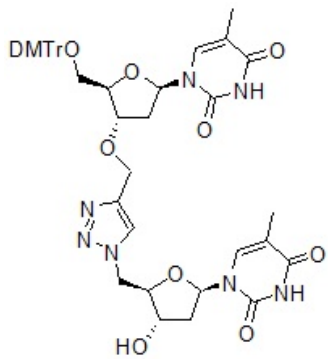
m_pal.MJP5-471

UserID m_pal SampleID MJP 5-471 - col - DMSO SupervisorID hayes Lab Phone No. 13536 Slot Number 13

Recorded on a Bruker AV-3-400 in d6-DMSO

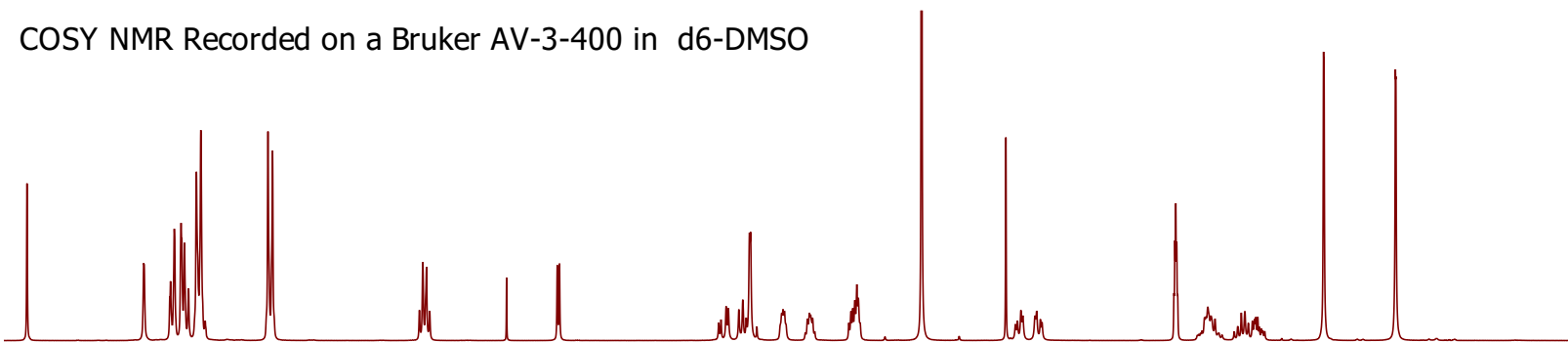
DEPT 135 NMR recorded at 101 MHz





16

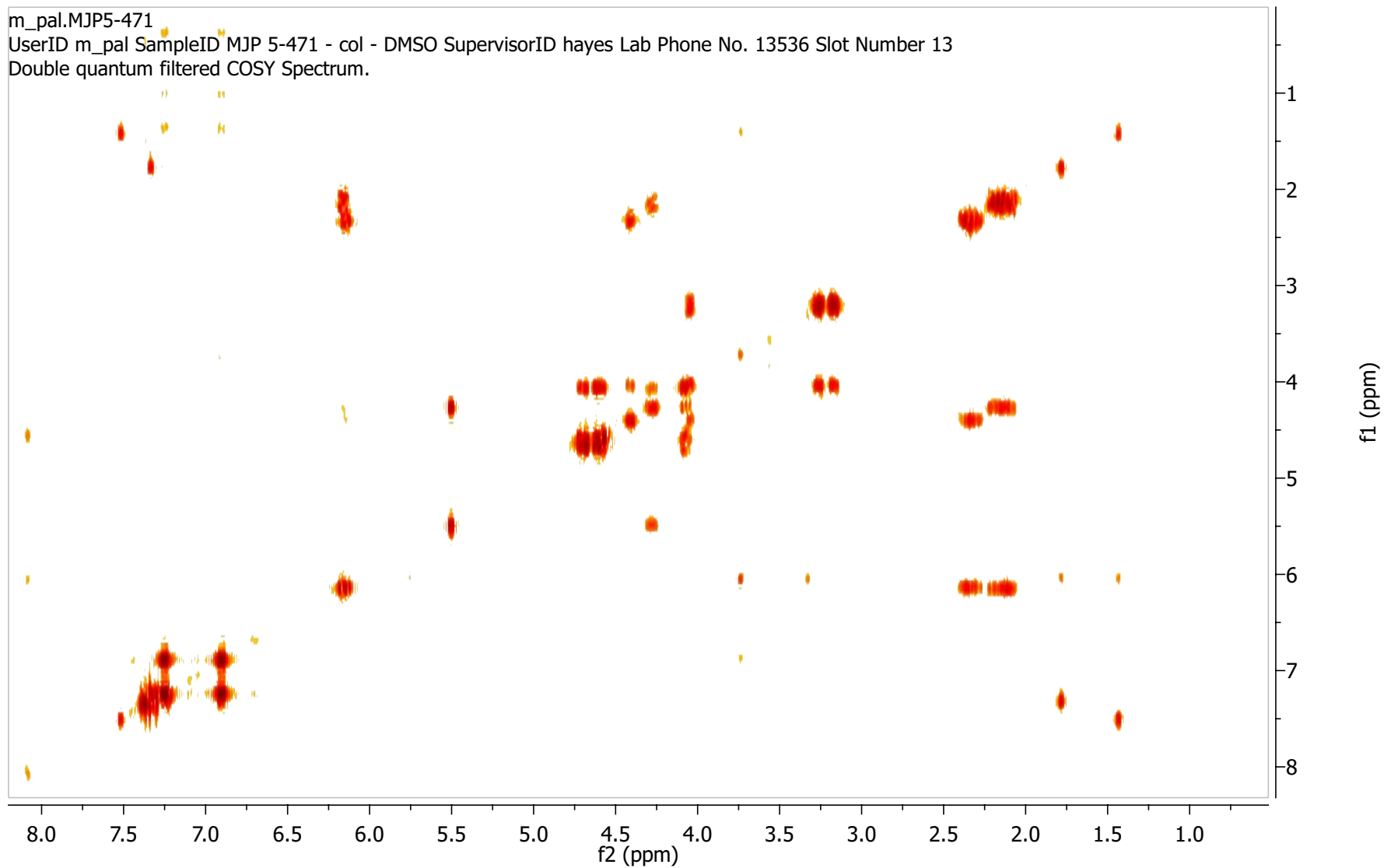
COSY NMR Recorded on a Bruker AV-3-400 in d6-DMSO

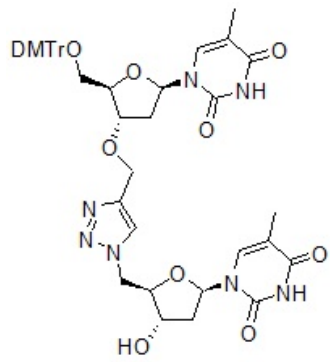


m_pal.MJP5-471

UserID m_pal SampleID MJP 5-471 - col - DMSO SupervisorID hayes Lab Phone No. 13536 Slot Number 13

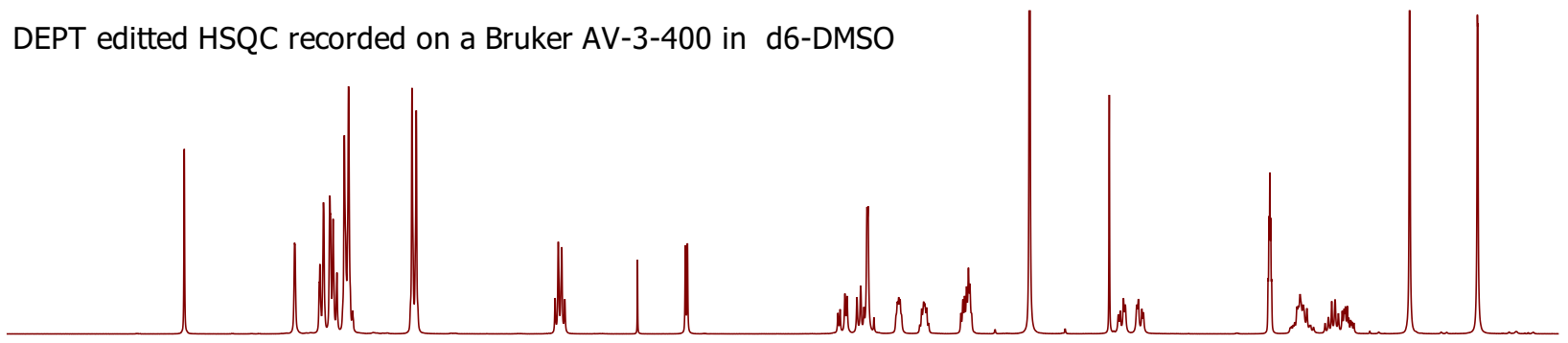
Double quantum filtered COSY Spectrum.





16

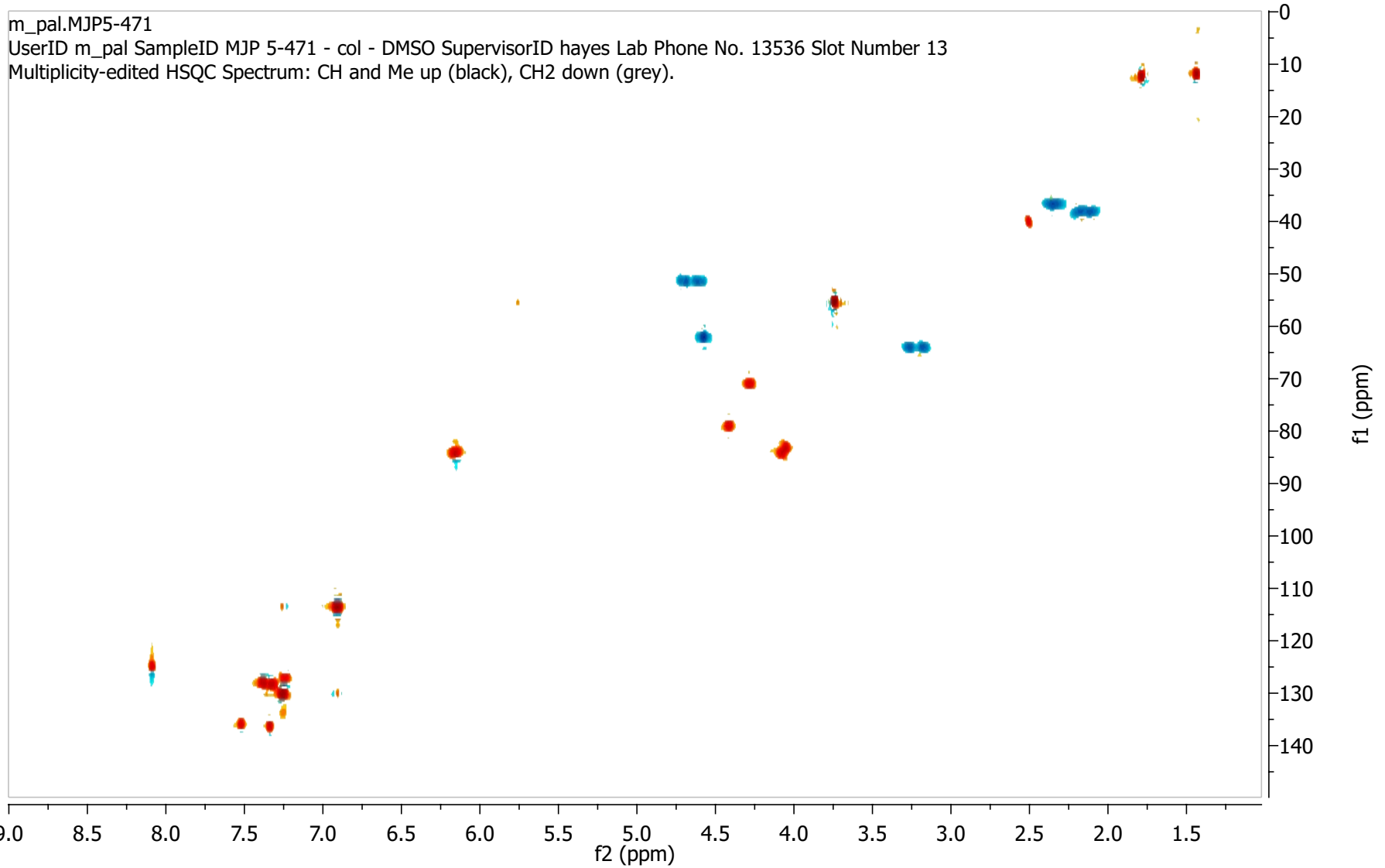
DEPT edited HSQC recorded on a Bruker AV-3-400 in d6-DMSO

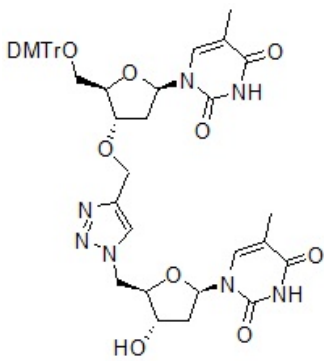


m_pal.MJP5-471

UserID m_pal SampleID MJP 5-471 - col - DMSO SupervisorID hayes Lab Phone No. 13536 Slot Number 13

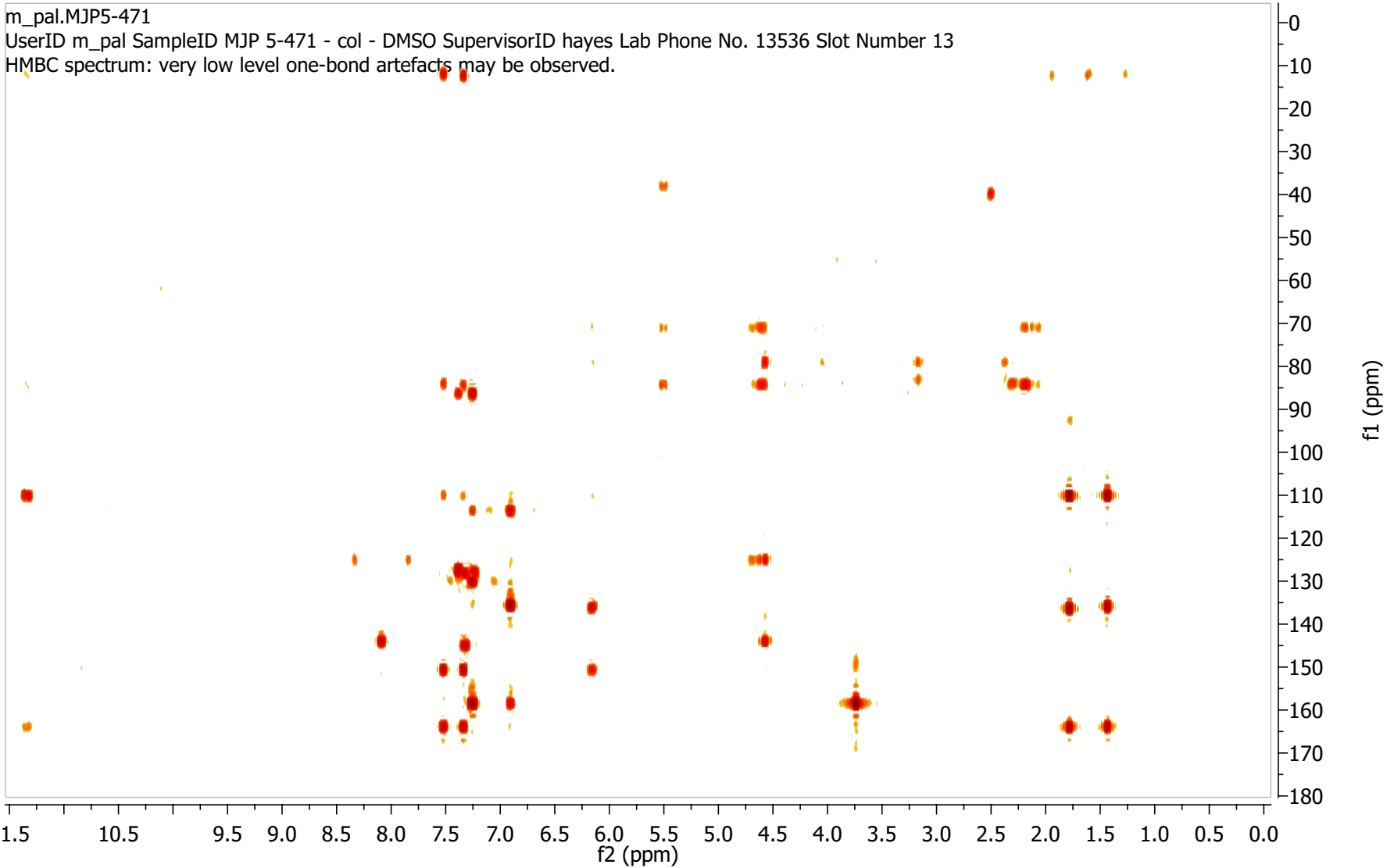
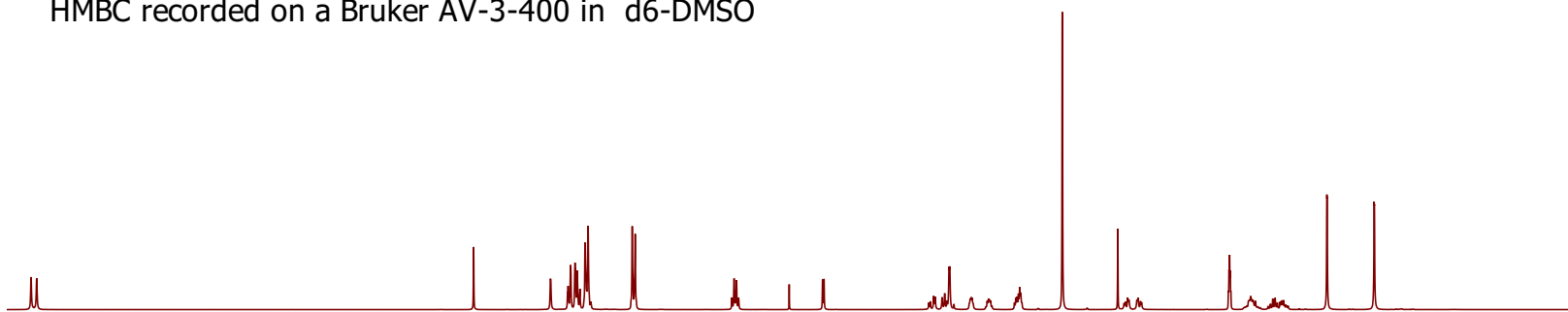
Multiplicity-edited HSQC Spectrum: CH and Me up (black), CH2 down (grey).





16

HMBC recorded on a Bruker AV-3-400 in d6-DMSO



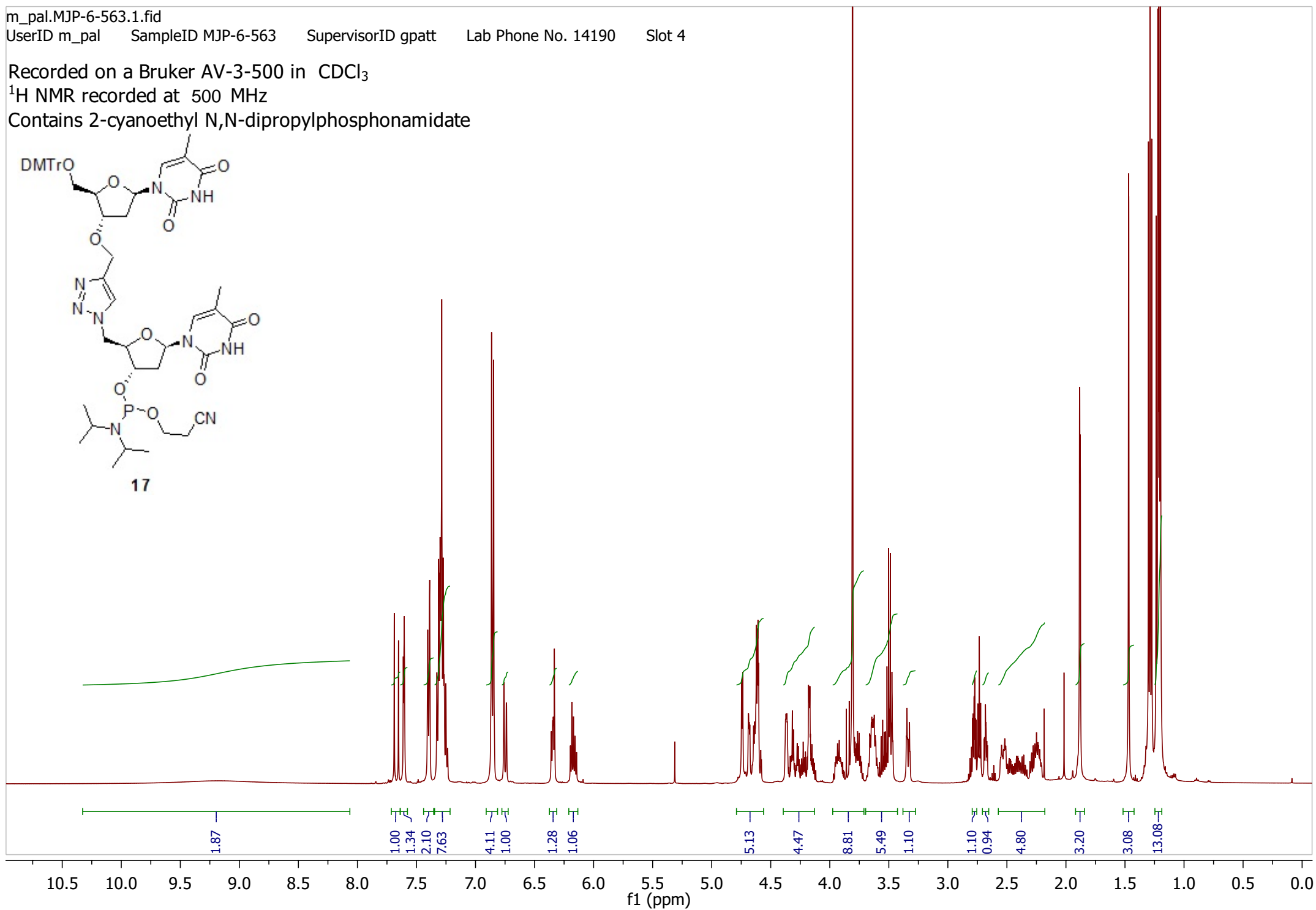
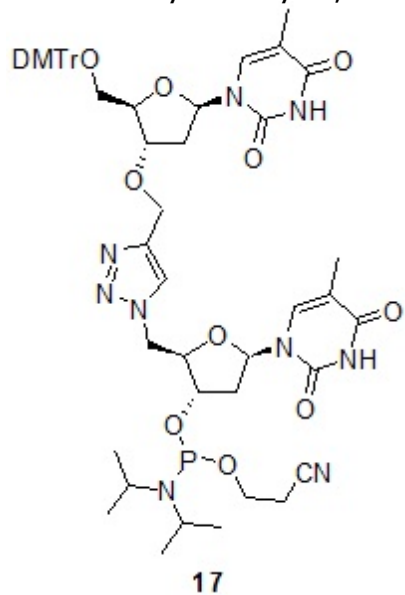
m_pal.MJP-6-563.1.fid

UserID m_pal SampleID MJP-6-563 SupervisorID gpatt Lab Phone No. 14190 Slot 4

Recorded on a Bruker AV-3-500 in CDCl₃

¹H NMR recorded at 500 MHz

Contains 2-cyanoethyl N,N-dipropylphosphoramidate



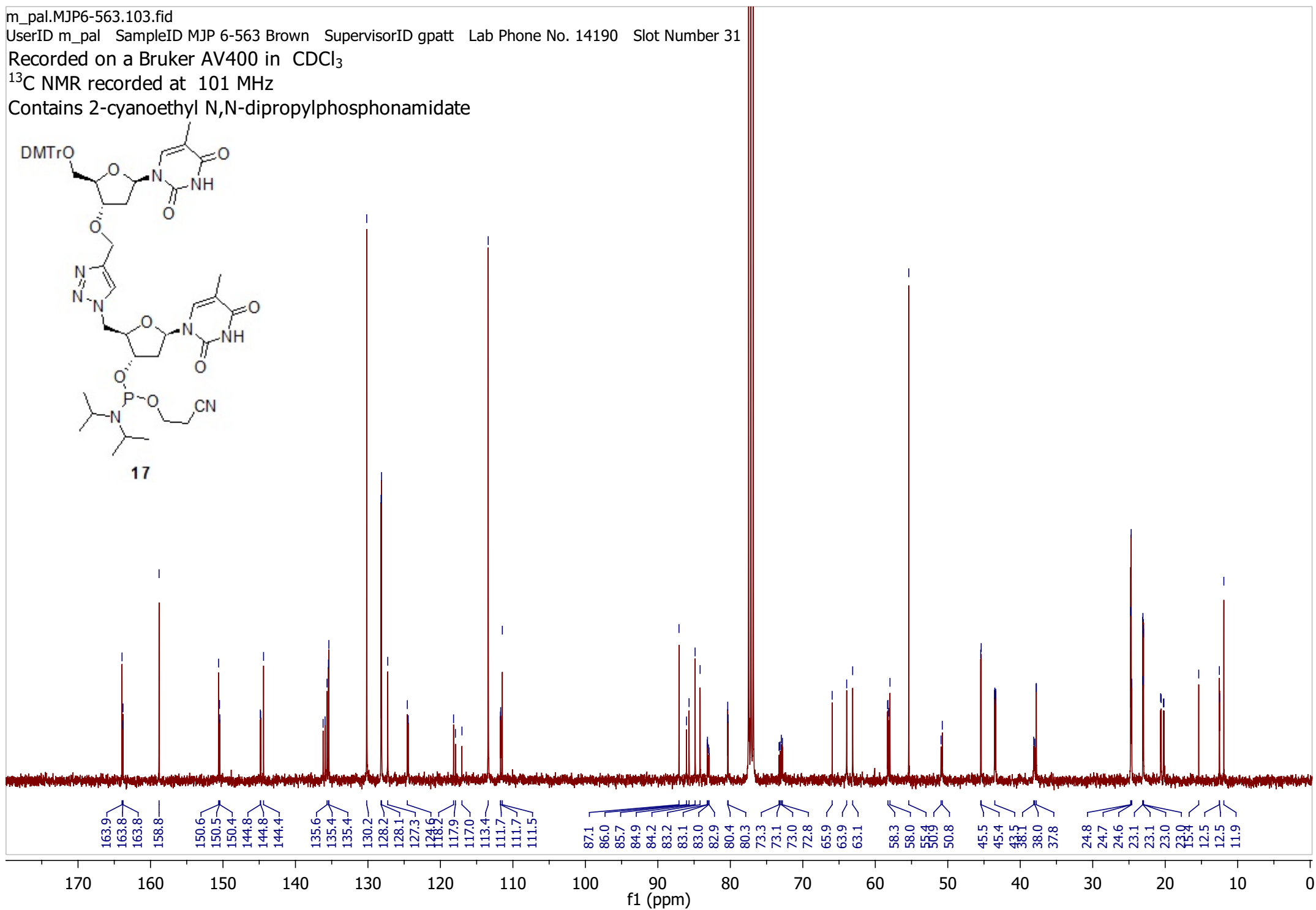
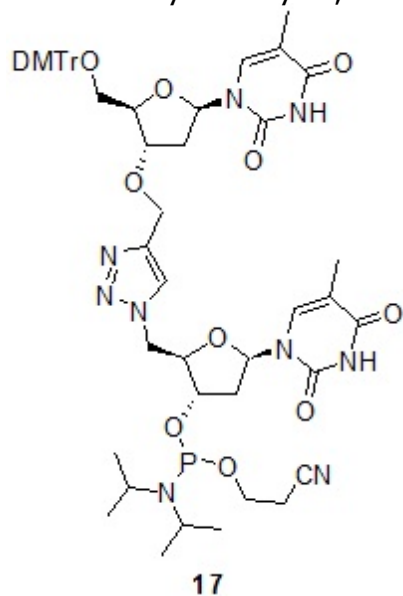
m_pal.MJP6-563.103.fid

UserID m_pal SampleID MJP 6-563 Brown SupervisorID gpatt Lab Phone No. 14190 Slot Number 31

Recorded on a Bruker AV400 in CDCl₃

¹³C NMR recorded at 101 MHz

Contains 2-cyanoethyl N,N-dipropylphosphonamidate



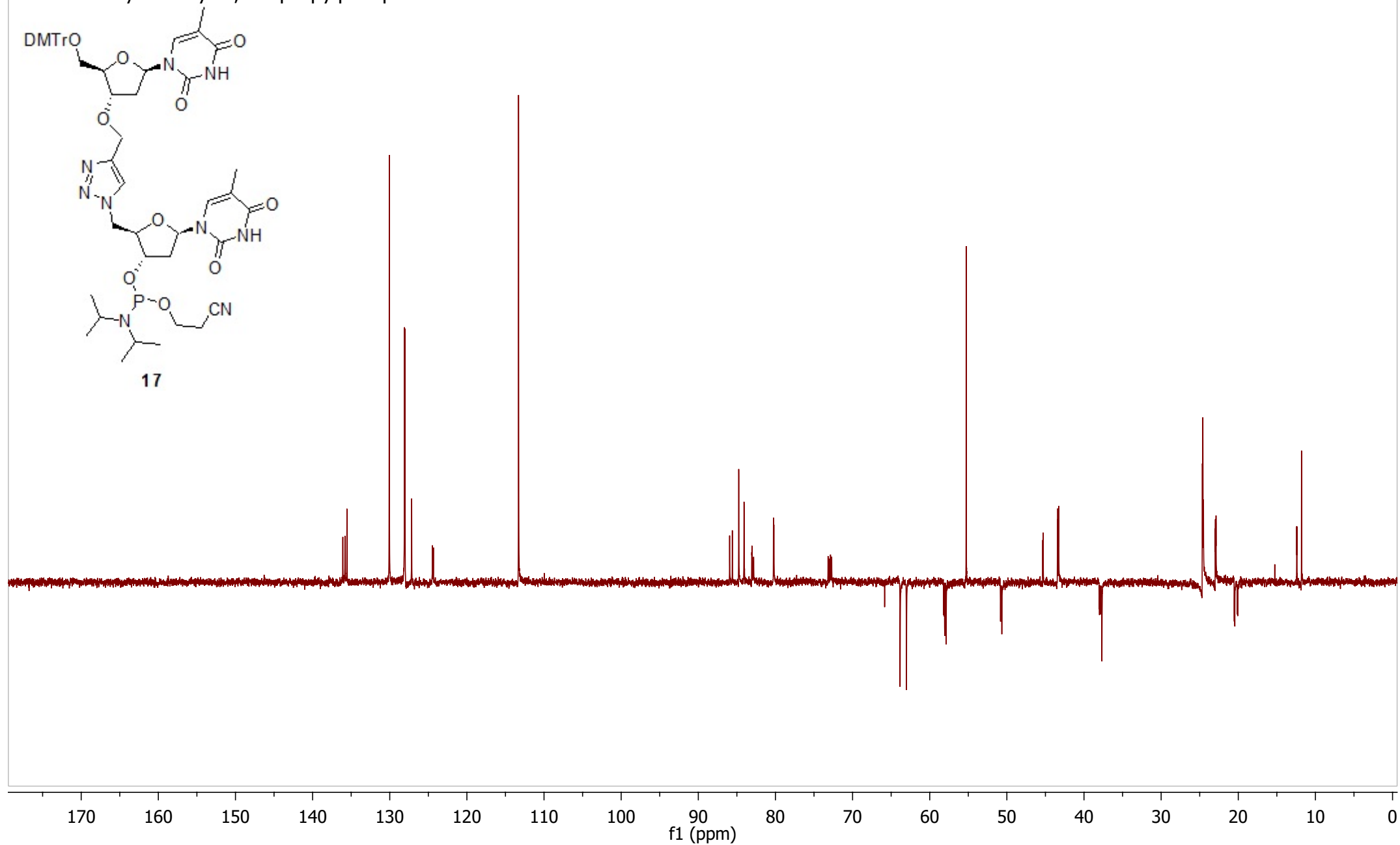
m_pal.MJP6-563.104.fid

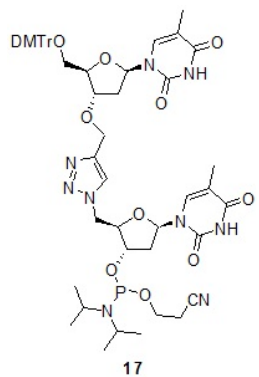
UserID m_pal SampleID MJP 6-563 Brown SupervisorID gpatt Lab Phone No. 14190 Slot Number 31

Recorded on a Bruker AV400 in CDCl₃

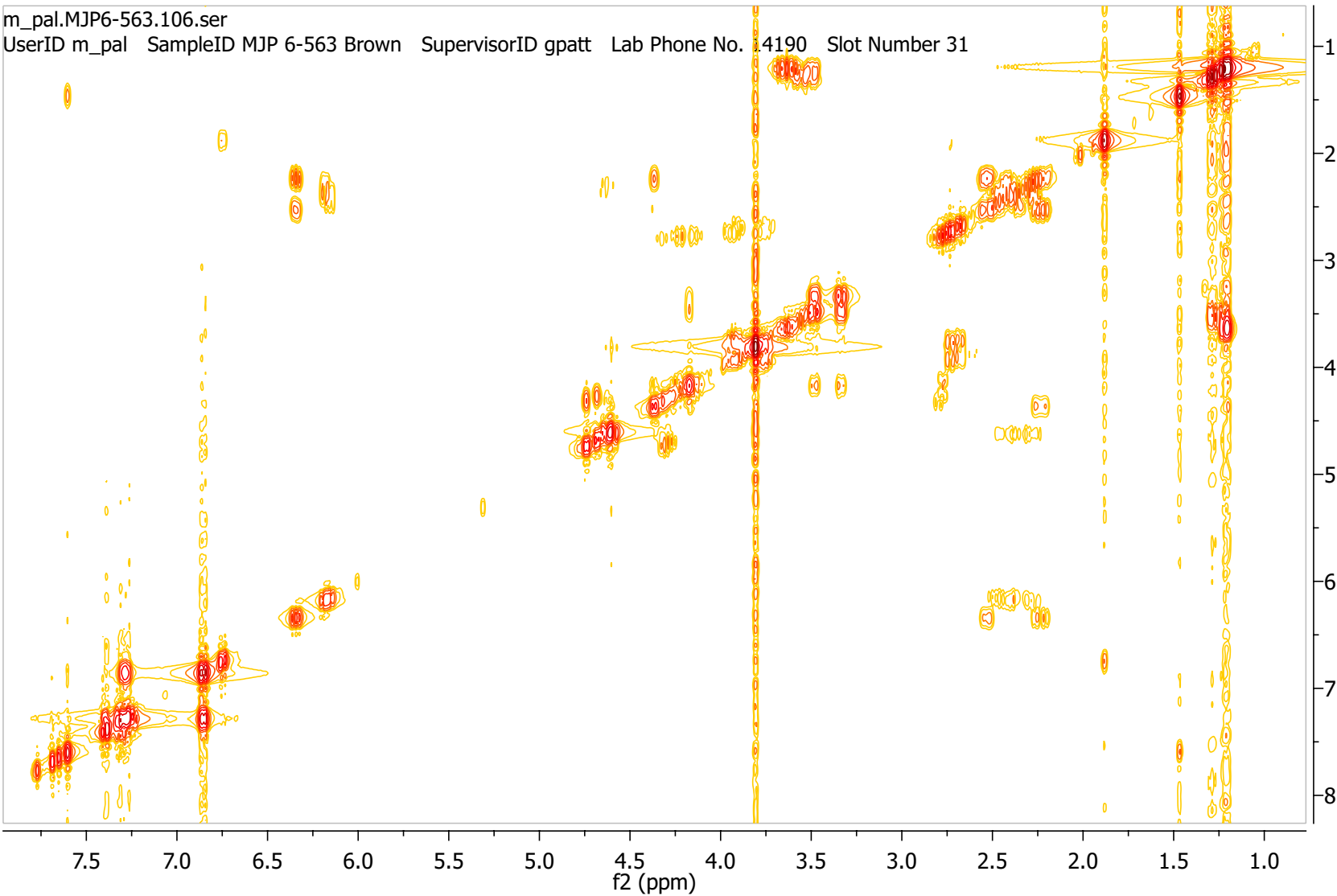
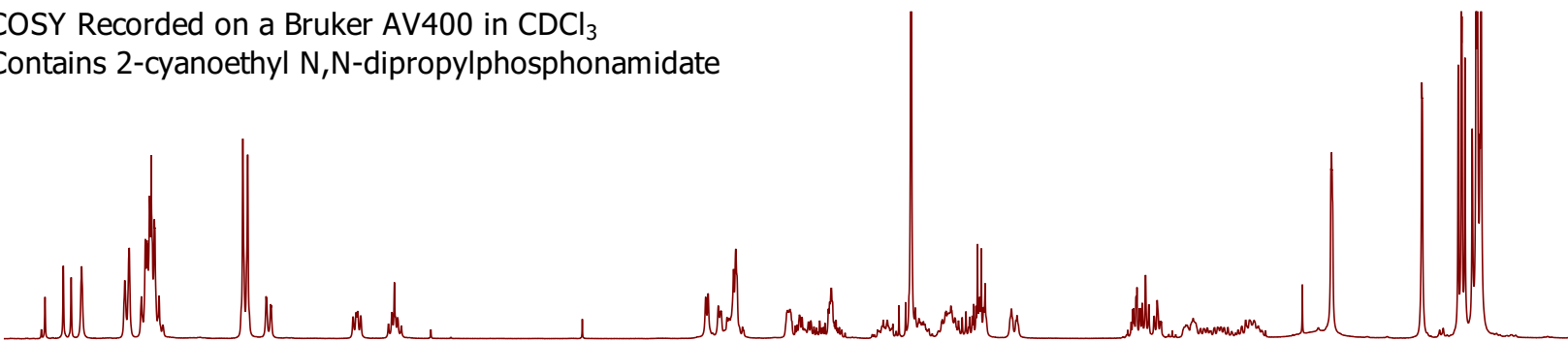
DEPT 135 recorded at 101 MHz

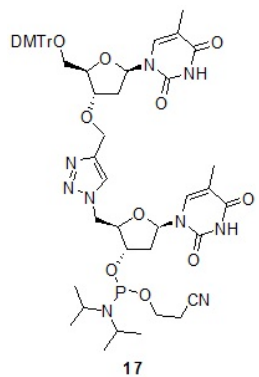
Contains 2-cyanoethyl N,N-dipropylphosphonamidate



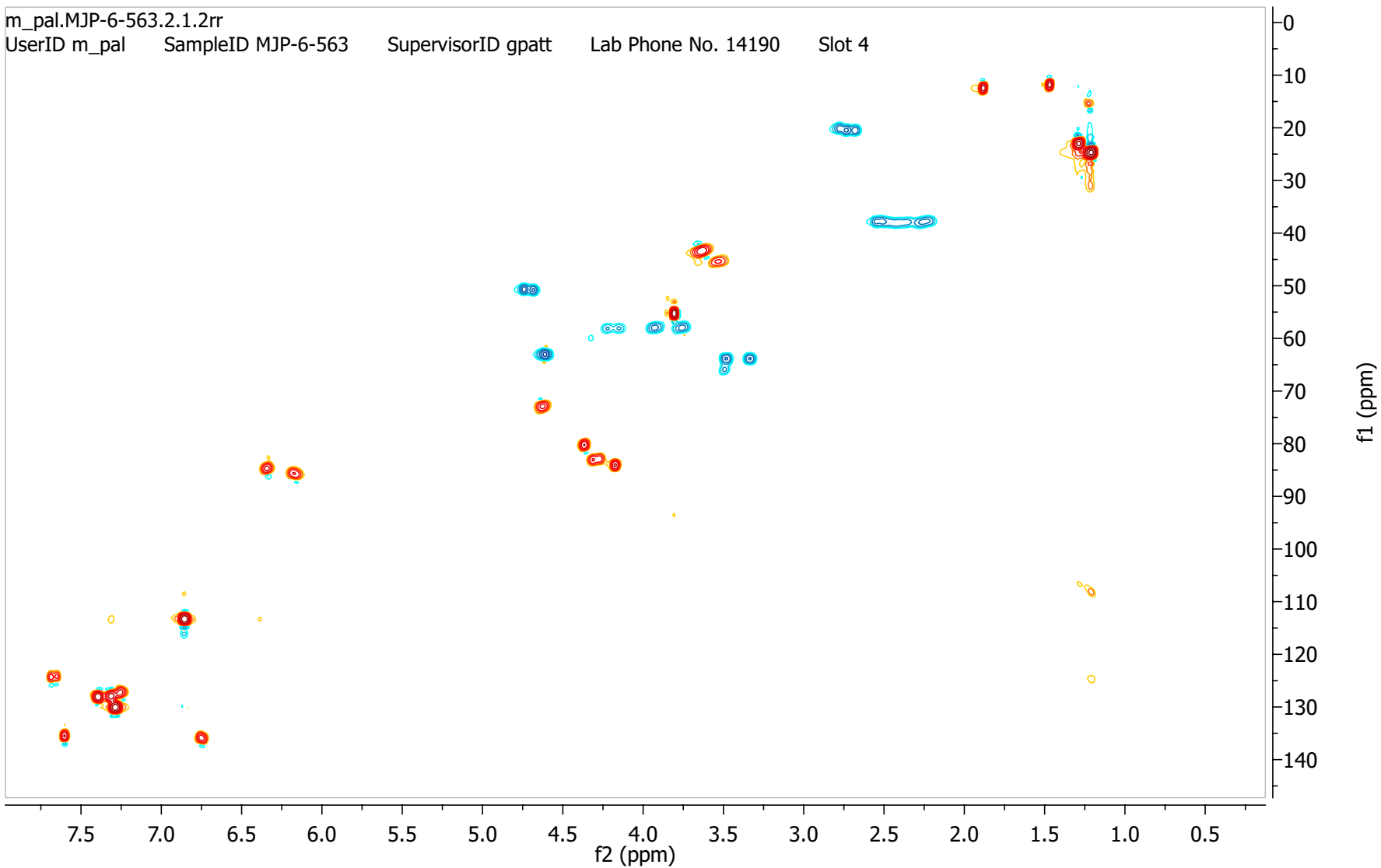


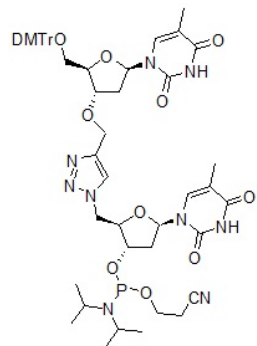
COSY Recorded on a Bruker AV400 in CDCl₃
Contains 2-cyanoethyl N,N-dipropylphosphoramidate





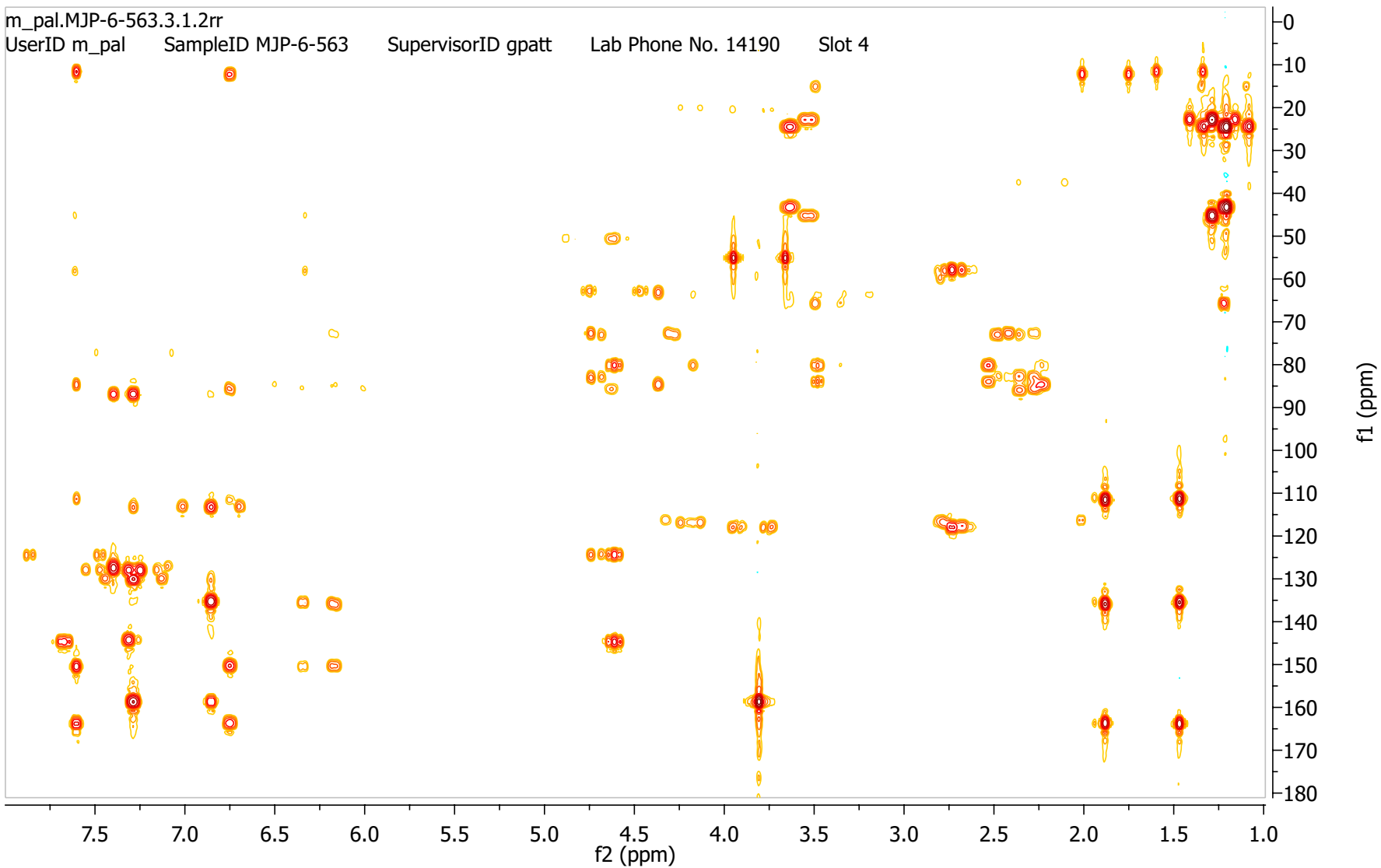
DEPT edited HSQC Recorded on a Bruker AV-3-500 in CDCl₃
Contains 2-cyanoethyl N,N-dipropylphosphonamidate





17

HMBC Recorded on a Bruker AV-3-500 in CDCl₃
Contains 2-cyanoethyl N,N-dipropylphosphonamidate



m_pal.MJP6-563.105.fid

UserID m_pal SampleID MJP 6-563 Brown SupervisorID gpatt Lab Phone No. 14190 Slot Number 31

Recorded on a Bruker AV400 in CDCl₃

³¹P NMR recorded at 162 MHz

Contains 2-cyanoethyl N,N-dipropylphosphonamidate

