Supplementary Data for: A-Ring modified steroidal azoles retaining similar potent and slowly reversible CYP17A1 inhibition as abiraterone

Mariana Garrido, Hwei-Ming Peng, Francis K. Yoshimoto, Sunil K. Upadhyay, Eugene Bratoeff, and Richard J. Auchus

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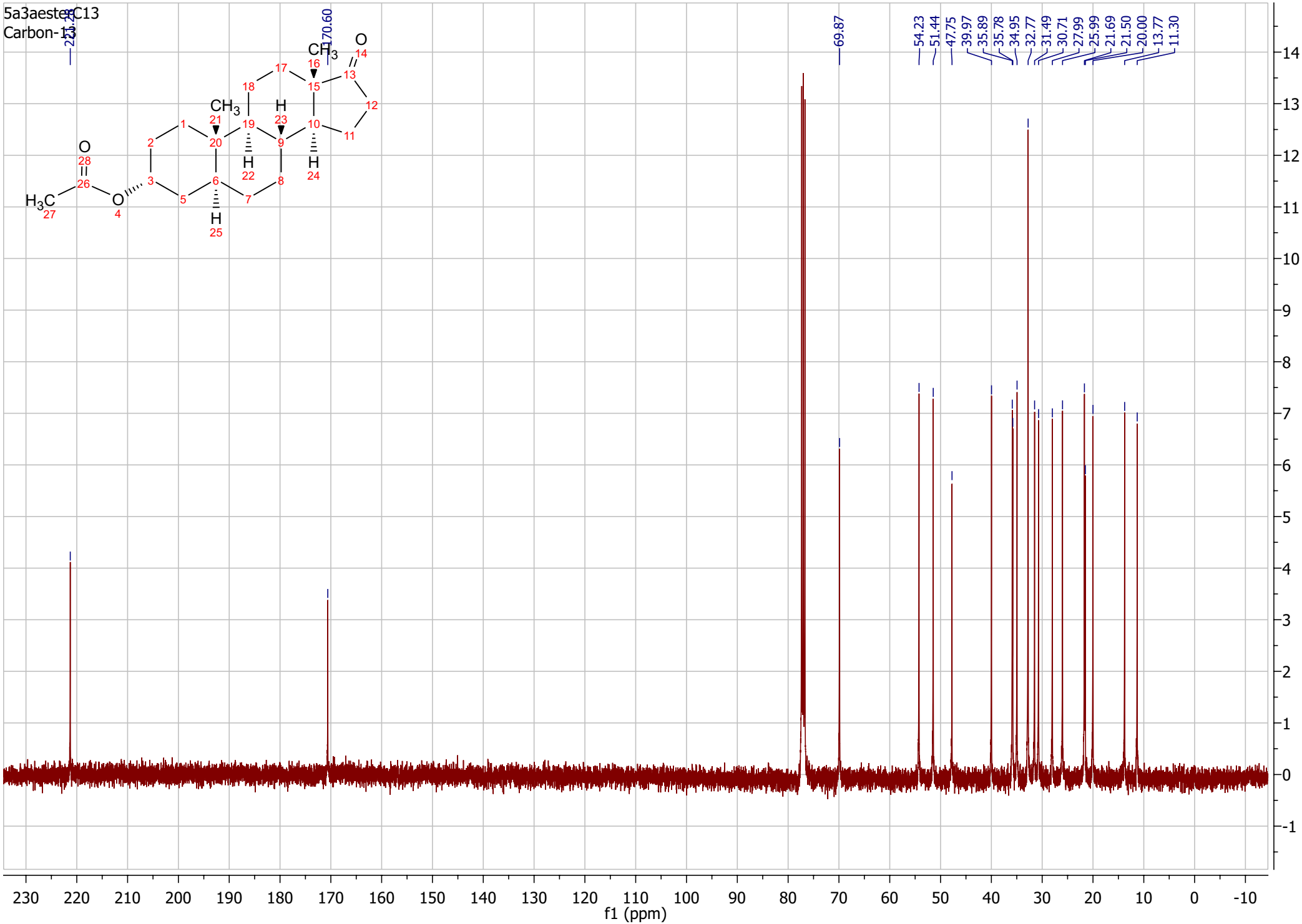
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51.44
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39.97
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21.50
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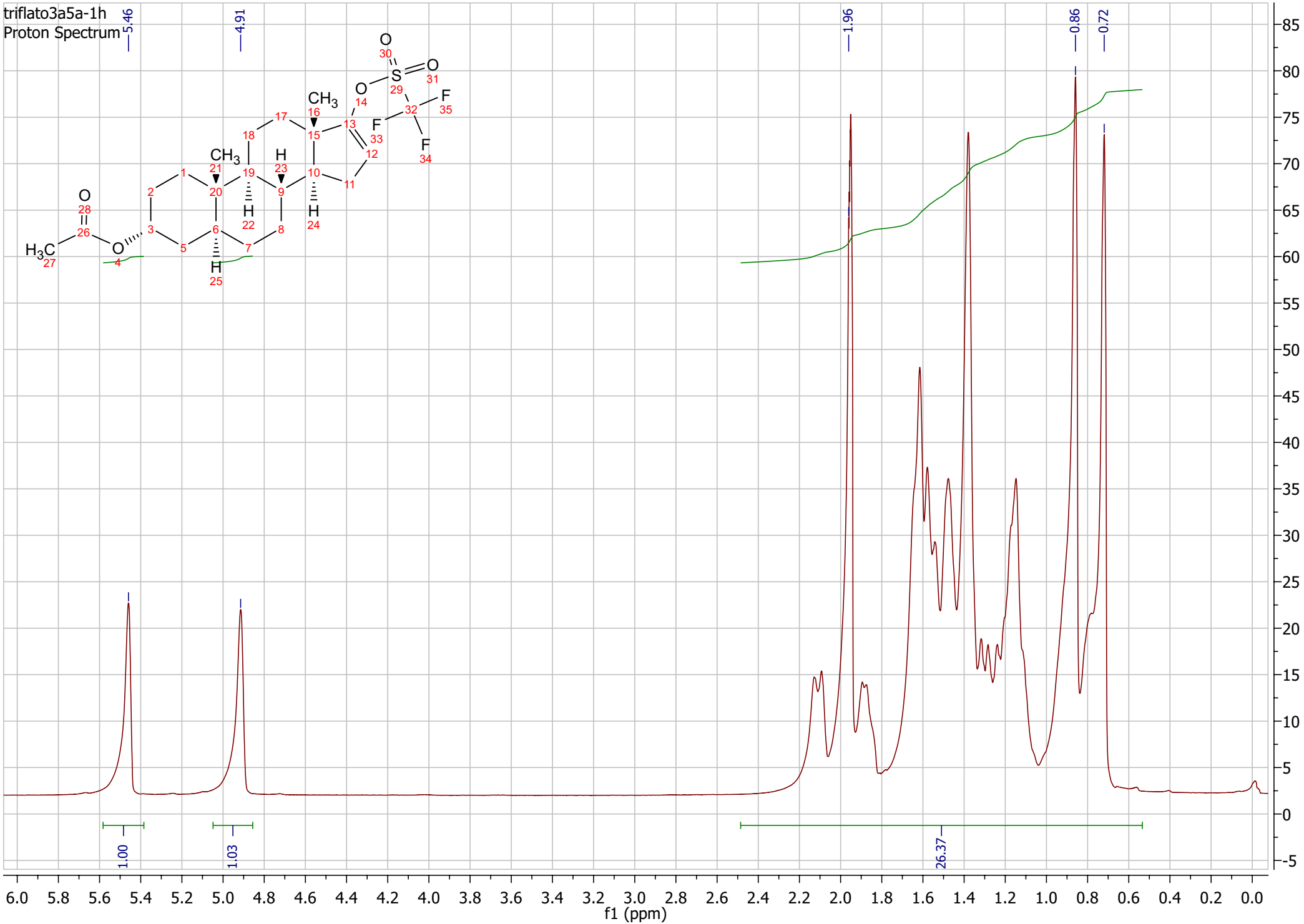
f1 (ppm)


triflato3a5a-1h
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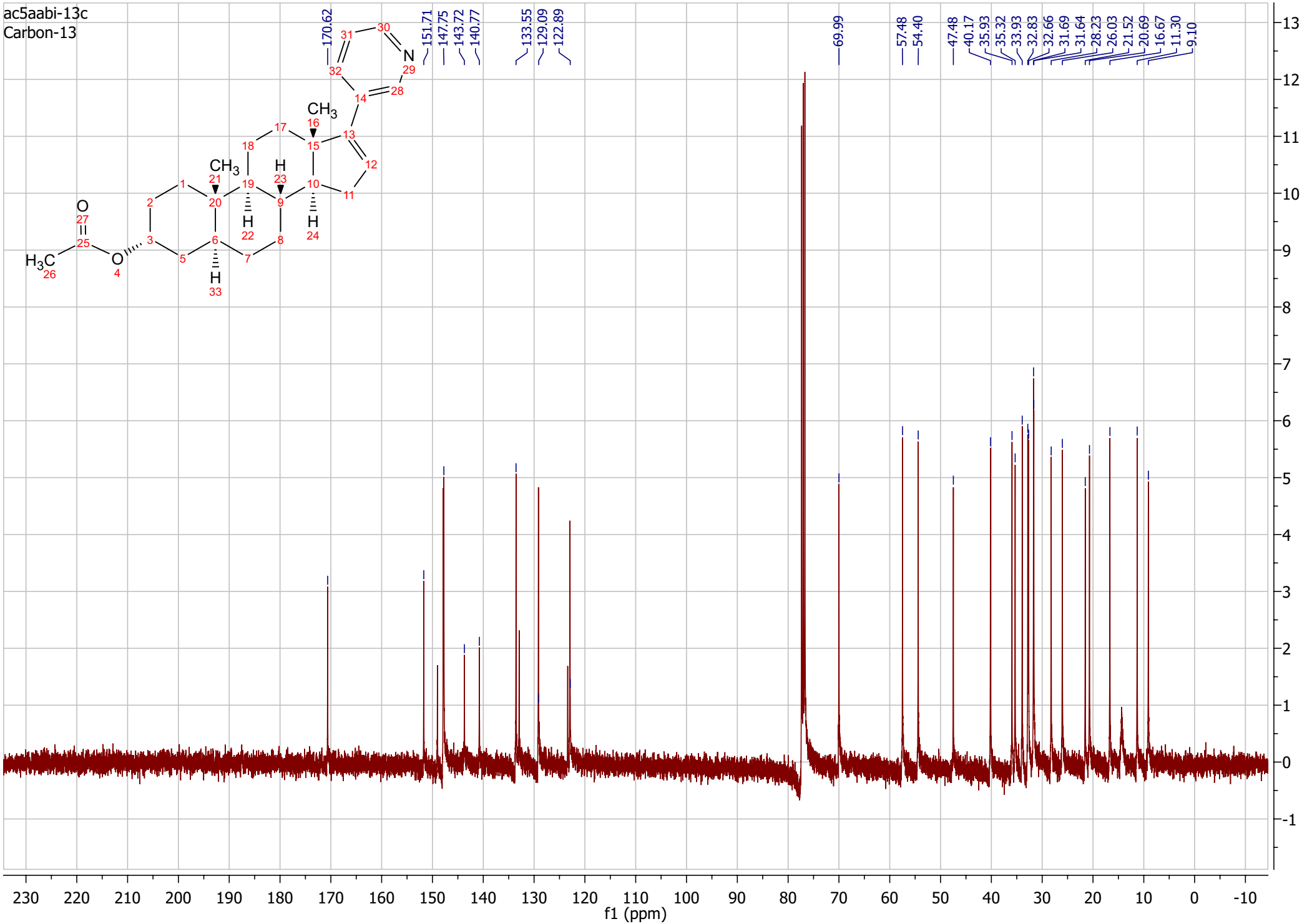
f1 (ppm)


ac5aabi-13c
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170.62
151.71
147.75
143.72
140.77
133.55
129.09
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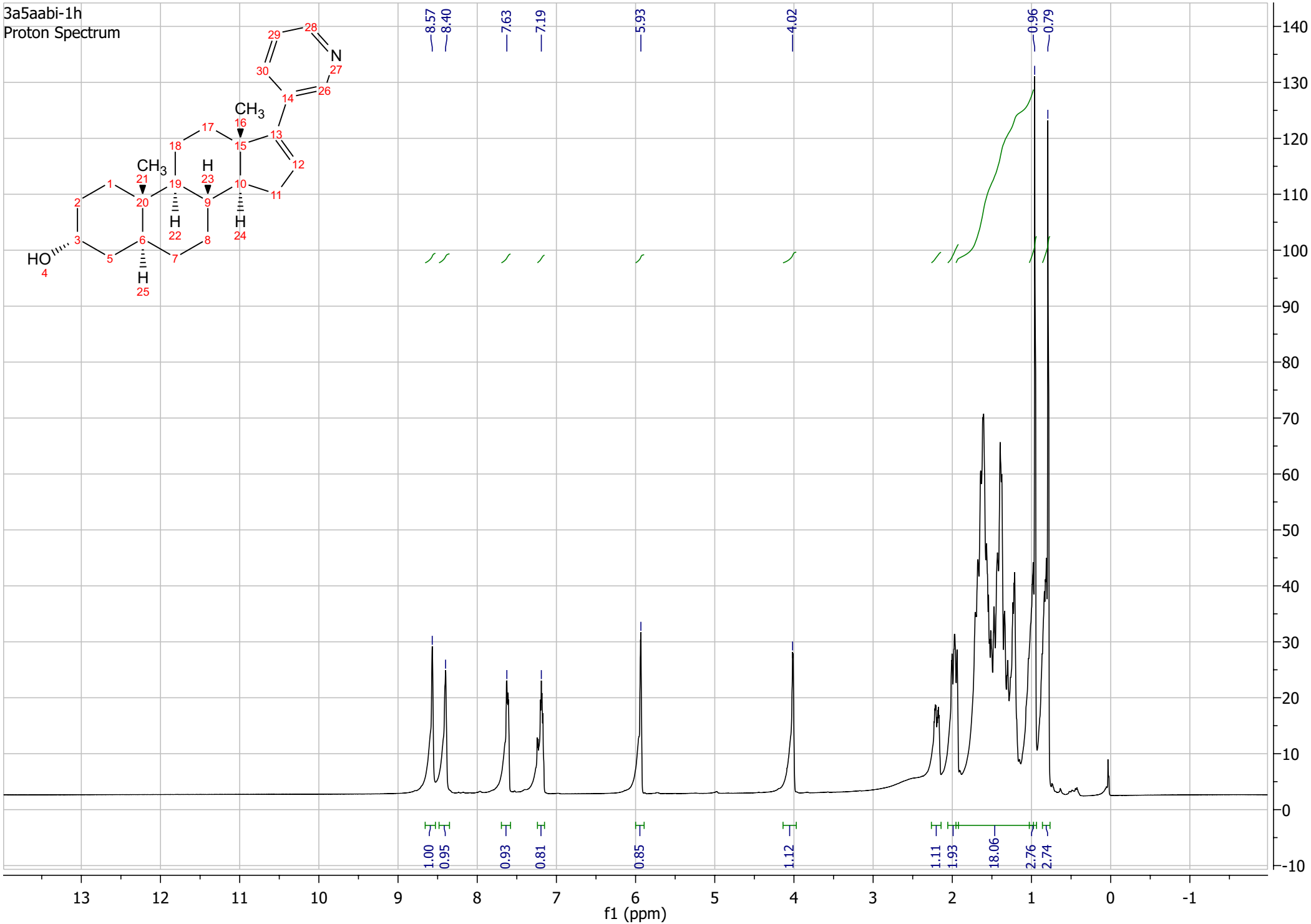
69.99
57.48
54.40
47.48
40.17
35.93
35.32
33.93
32.83
32.66
31.69
31.64
28.23
26.03
21.52
20.69
16.67
11.30
9.10
f1 (ppm)


3a5aabi-1h
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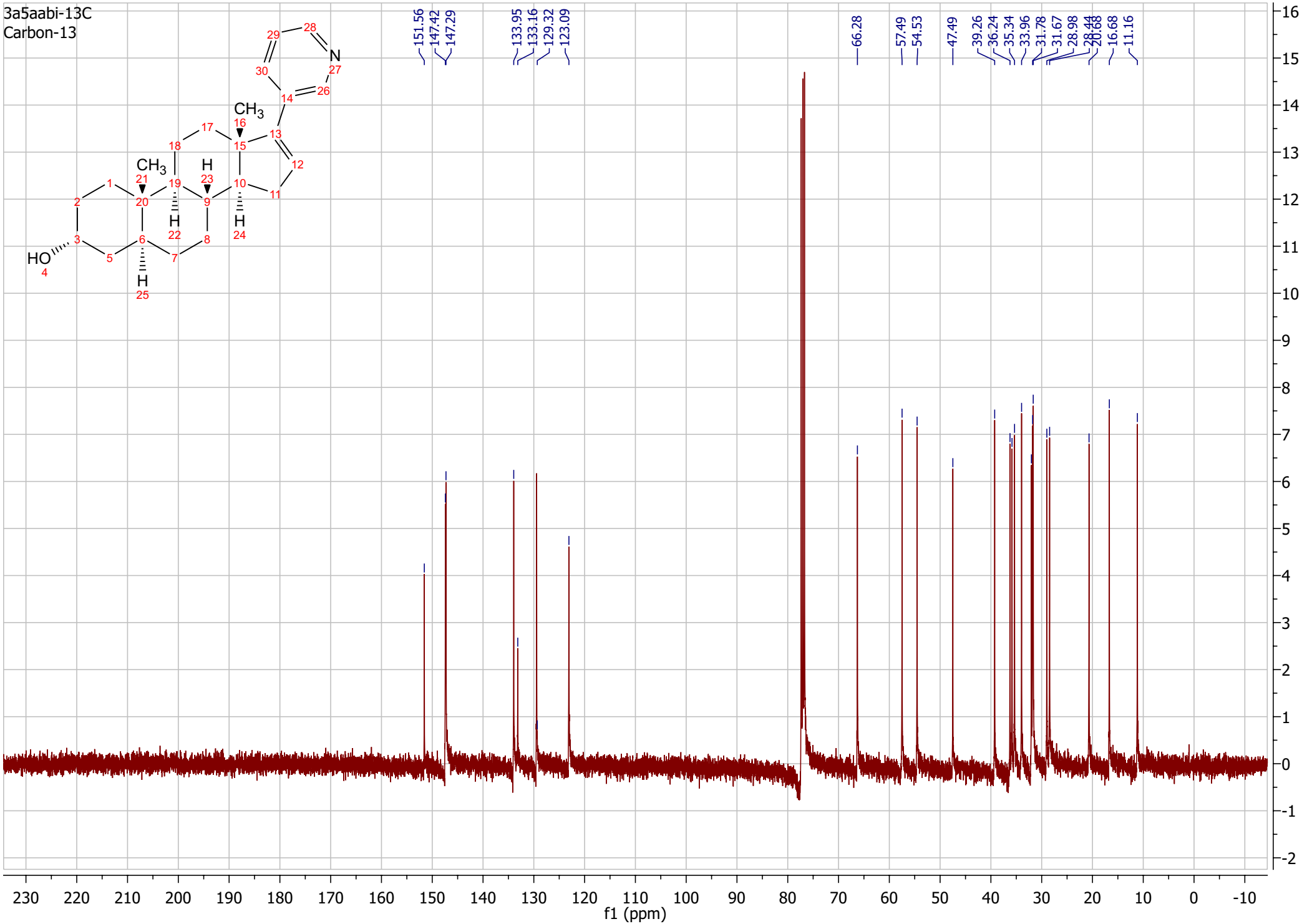
8.57
8.40
7.63
7.19
5.93
4.02
0.96
0.79
1.00
0.95
0.93
0.81
0.85
1.12
1.11
1.93
18.06
2.76
2.74
f1 (ppm)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Carbon-13
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147.42
147.29
133.95
133.16
129.32
123.09
66.28
57.49
54.53
47.49
39.26
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33.96
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31.67
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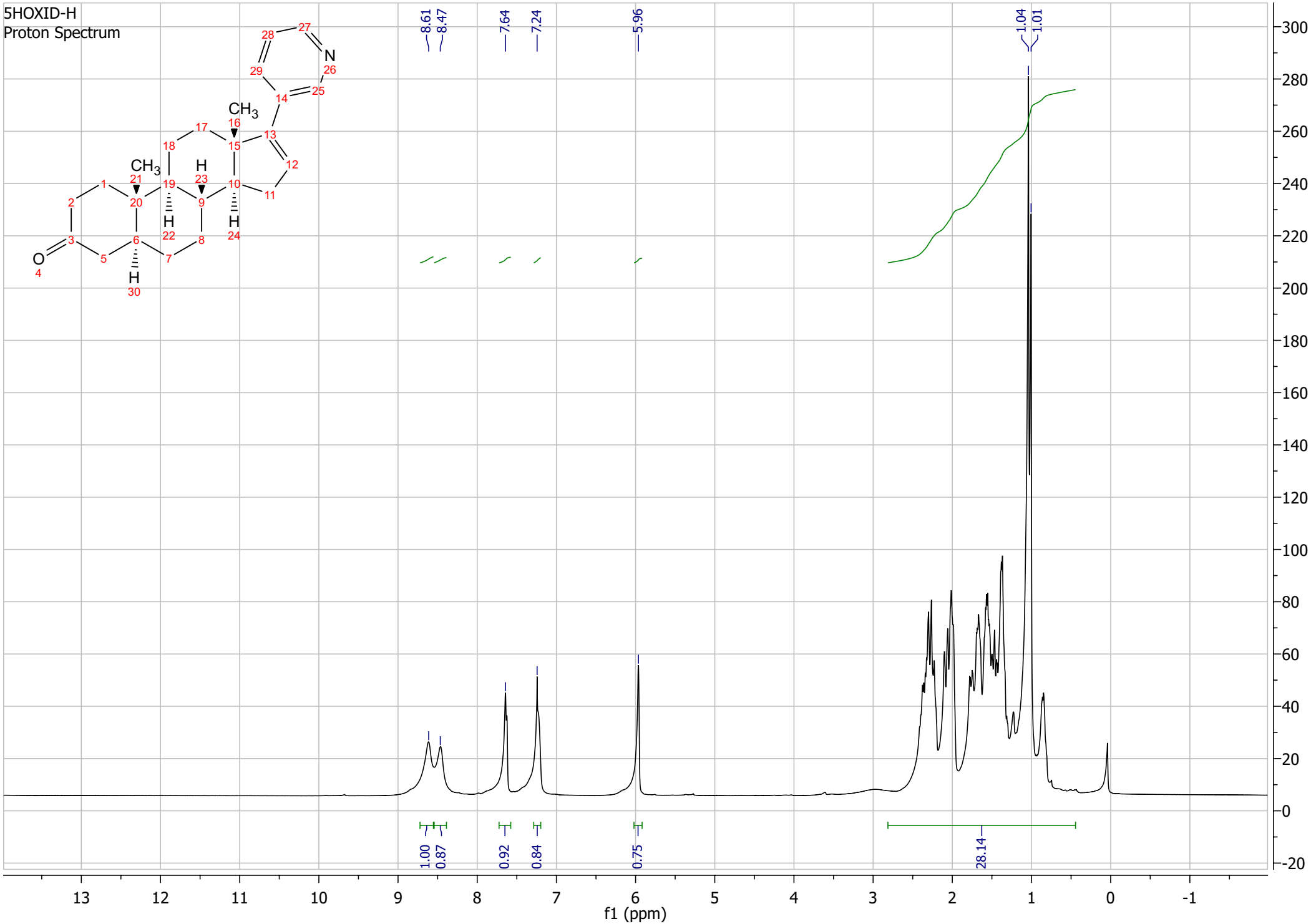
11.16
f1 (ppm)


5HOXID-H
Proton Spectrum
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8.47
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0.92
0.84
0.75
28.14
f1 (ppm)

Gradient Shimming

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3-keto-5a-Abi
Data Collected on:
ga.chem.lsa.umich.edu-vnmrs400
Archive directory:
/export/home/chempack/vnmrsys/data
Sample directory:

FidFile: 01022014-3-keto-5a-Abi-cdcl3-C13-NMR

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 2 2014

Operator: sunilku

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.621 sec
Width 25000.0 Hz
896 repetitions
OBSERVE C13, 100.4628324 MHz
DECOUPLE H1, 399.5357121 MHz
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WALTZ-16 modulated
DATA PROCESSING
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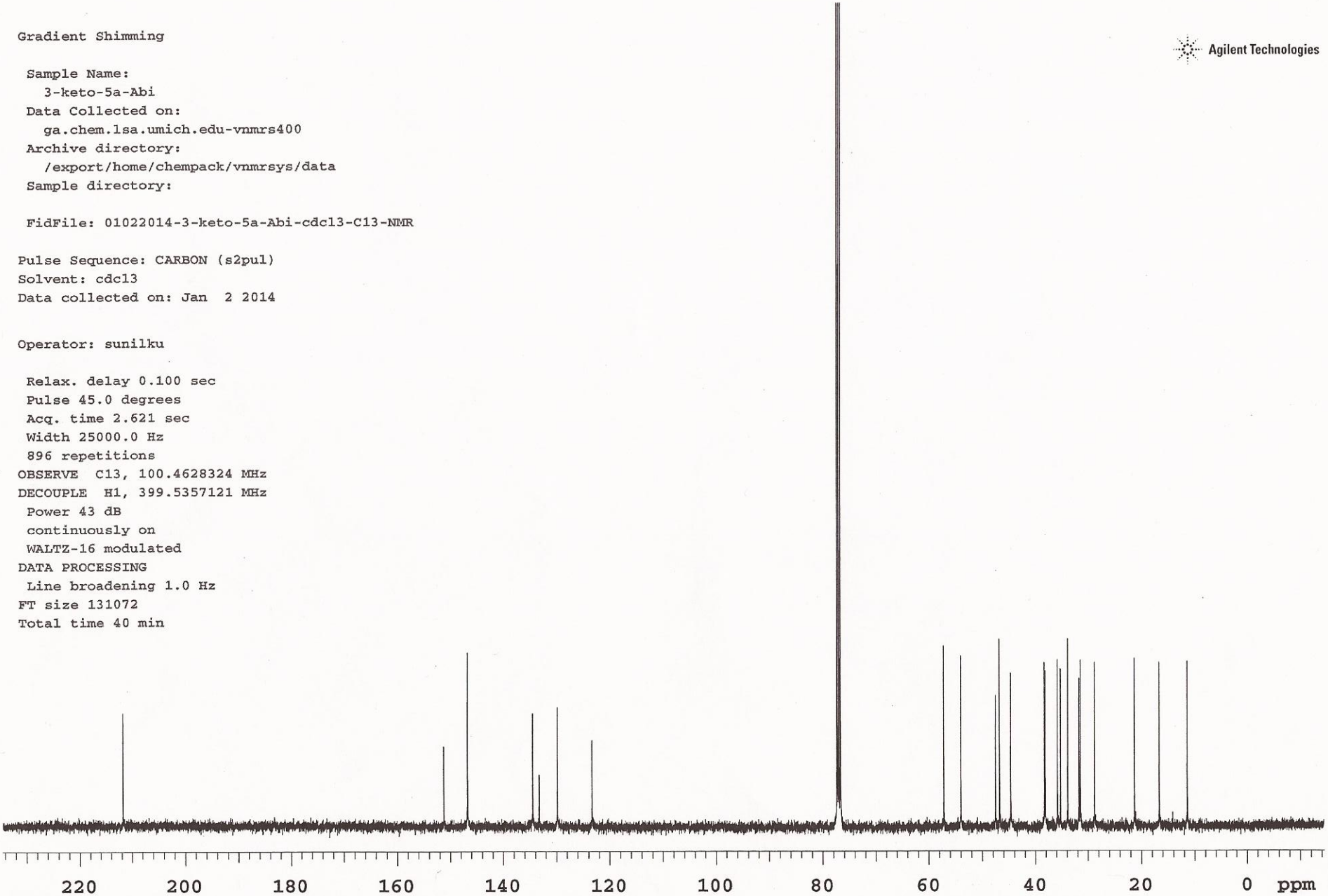

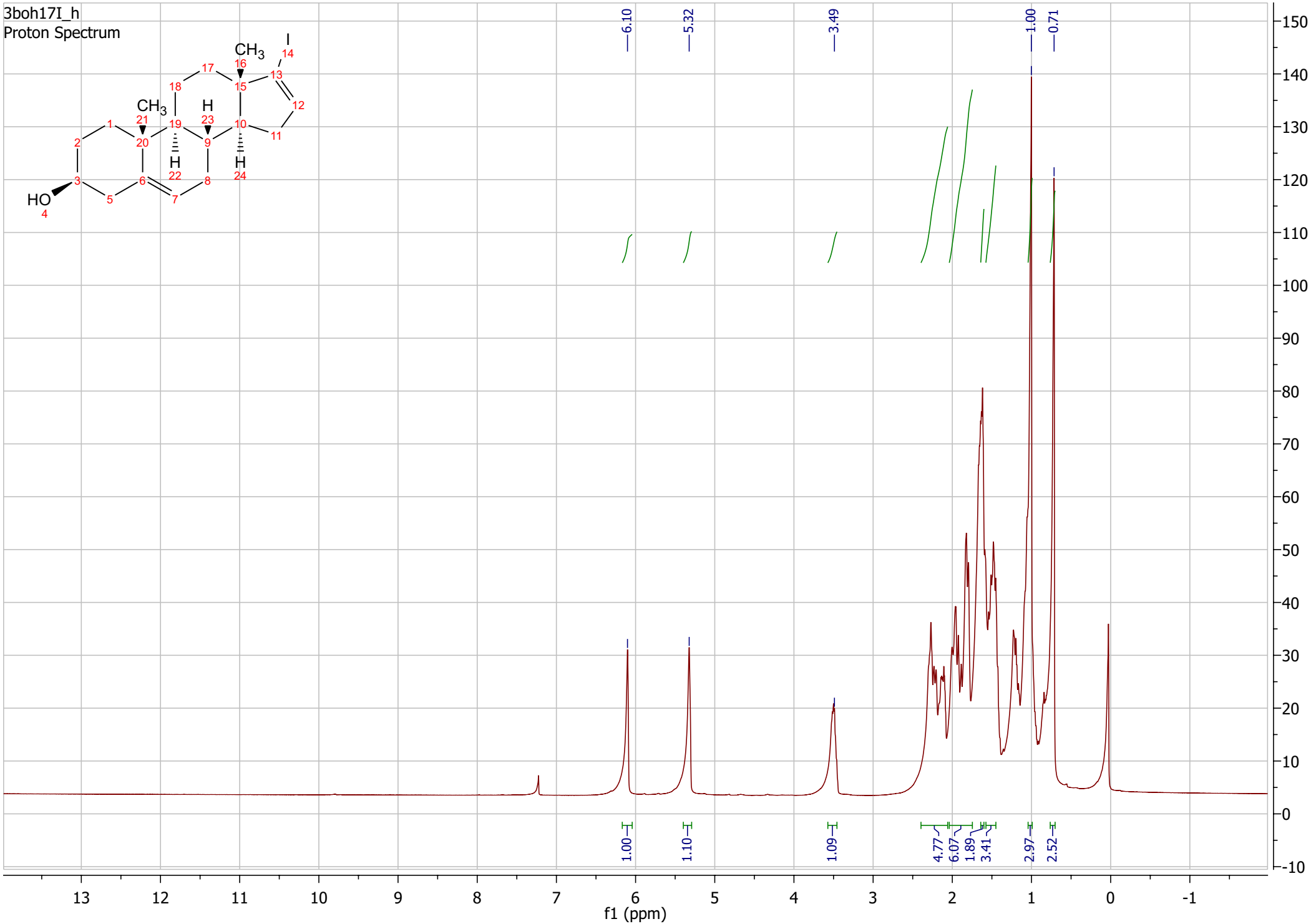
3boh17I_h
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0.71
1.00
1.10
1.09
4.77
6.07
1.89
3.41
2.97
2.52
f1 (ppm)

This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/

10/30/2013 9:33:20 AM

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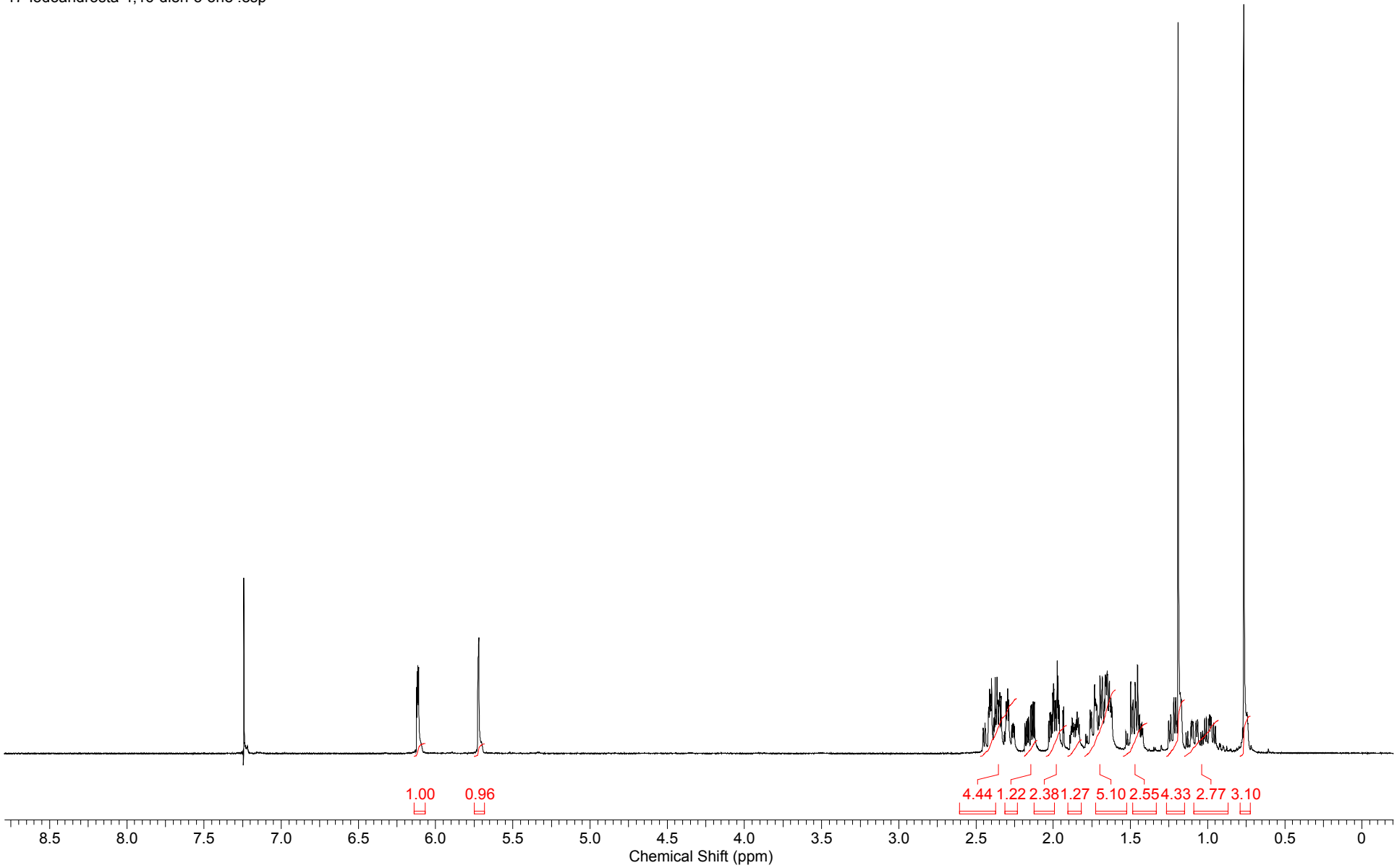
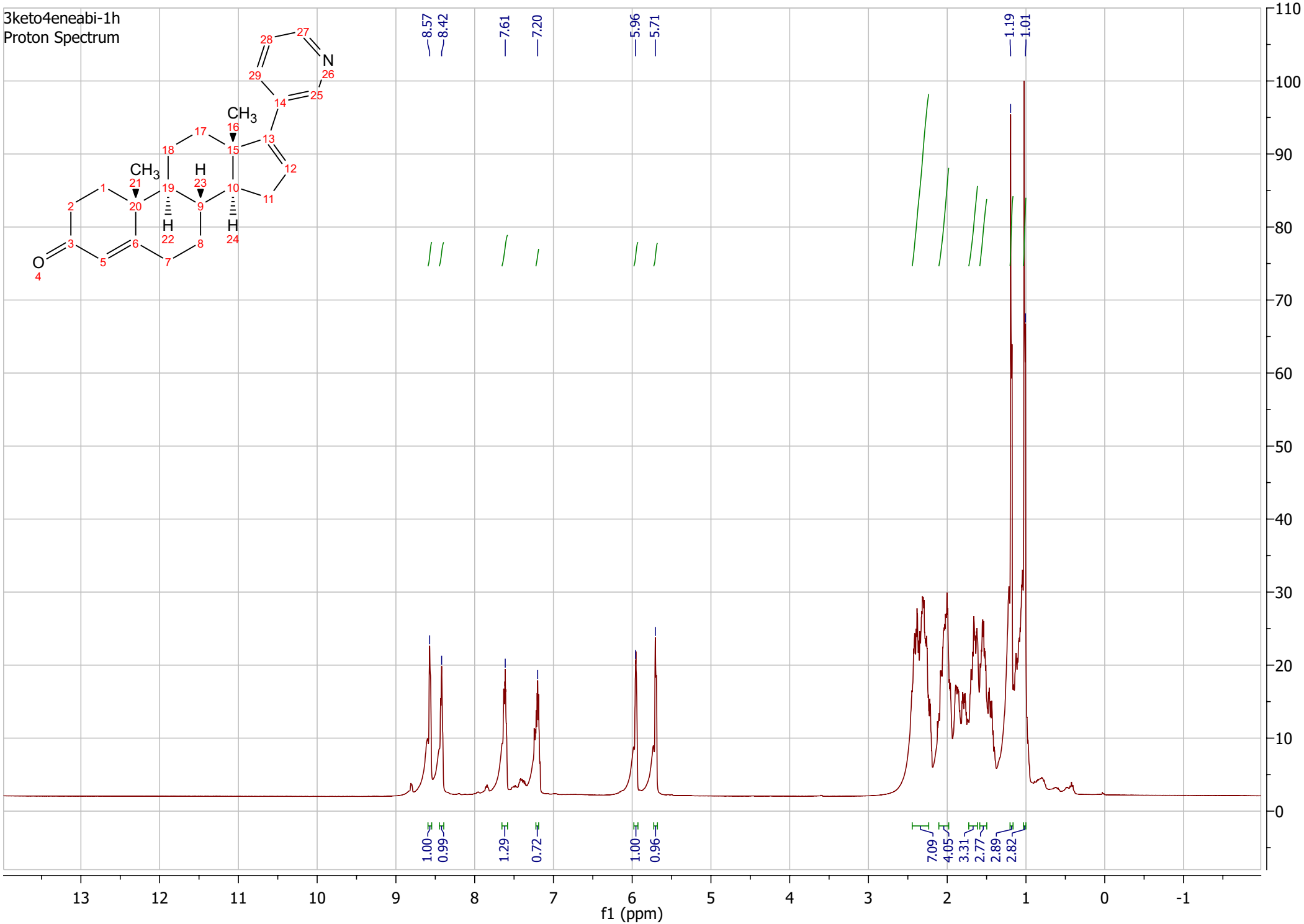
Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	44.00	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	2397.2000	***Spectrum Type***	STANDARD	***Sweep Width (Hz)***	6377.55	***Temperature (degree C)***	30.000

17-Iodoandrosta-4,16-dien-3-one .esp




3keto4eneabi-1h
Proton Spectrum
8.57
8.42
7.61
7.20
5.96
5.71
1.19
1.01
1.00
0.99
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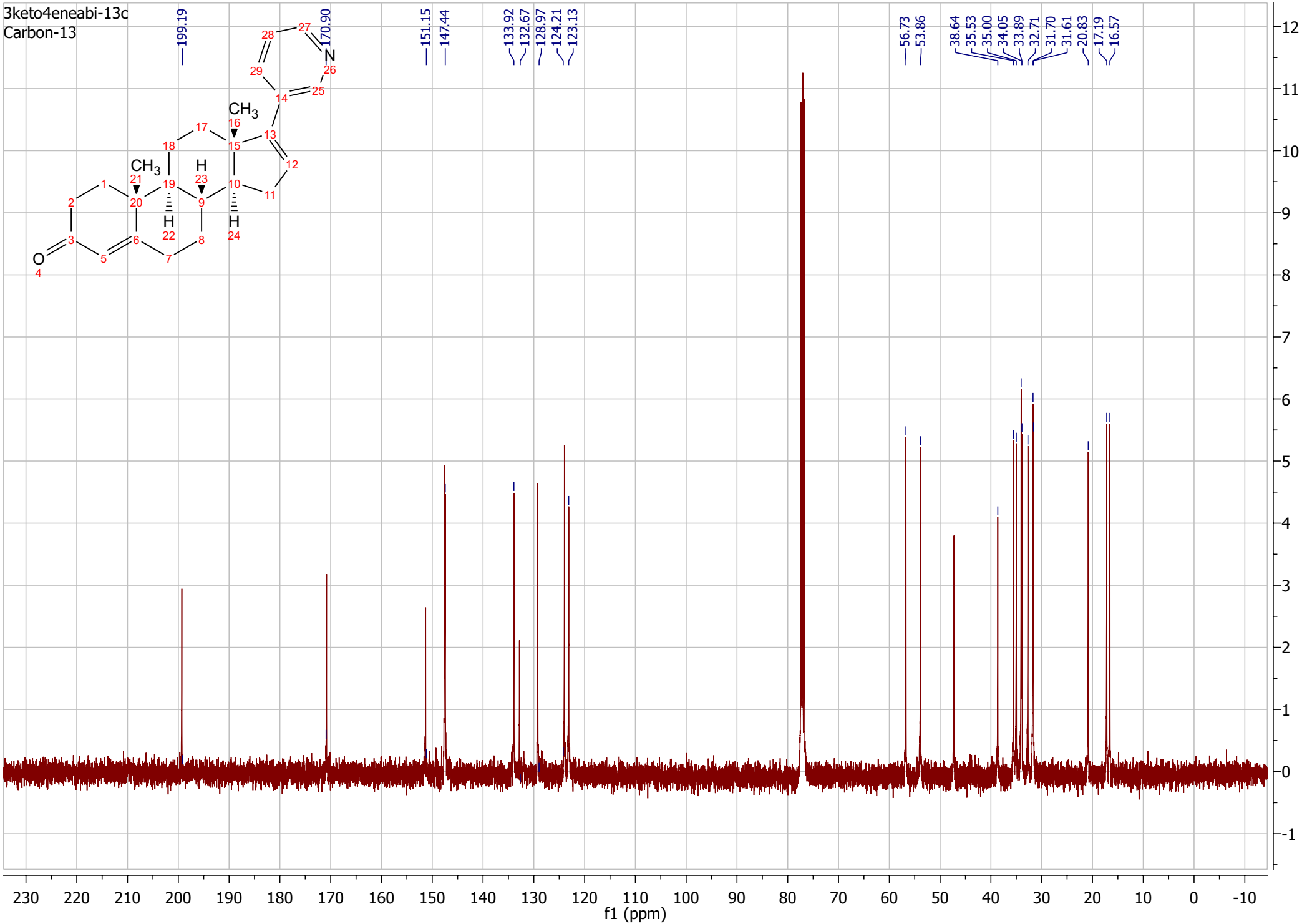
f1 (ppm)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Carbon-13
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123.13
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53.86
38.64
35.53
35.00
34.05
33.89
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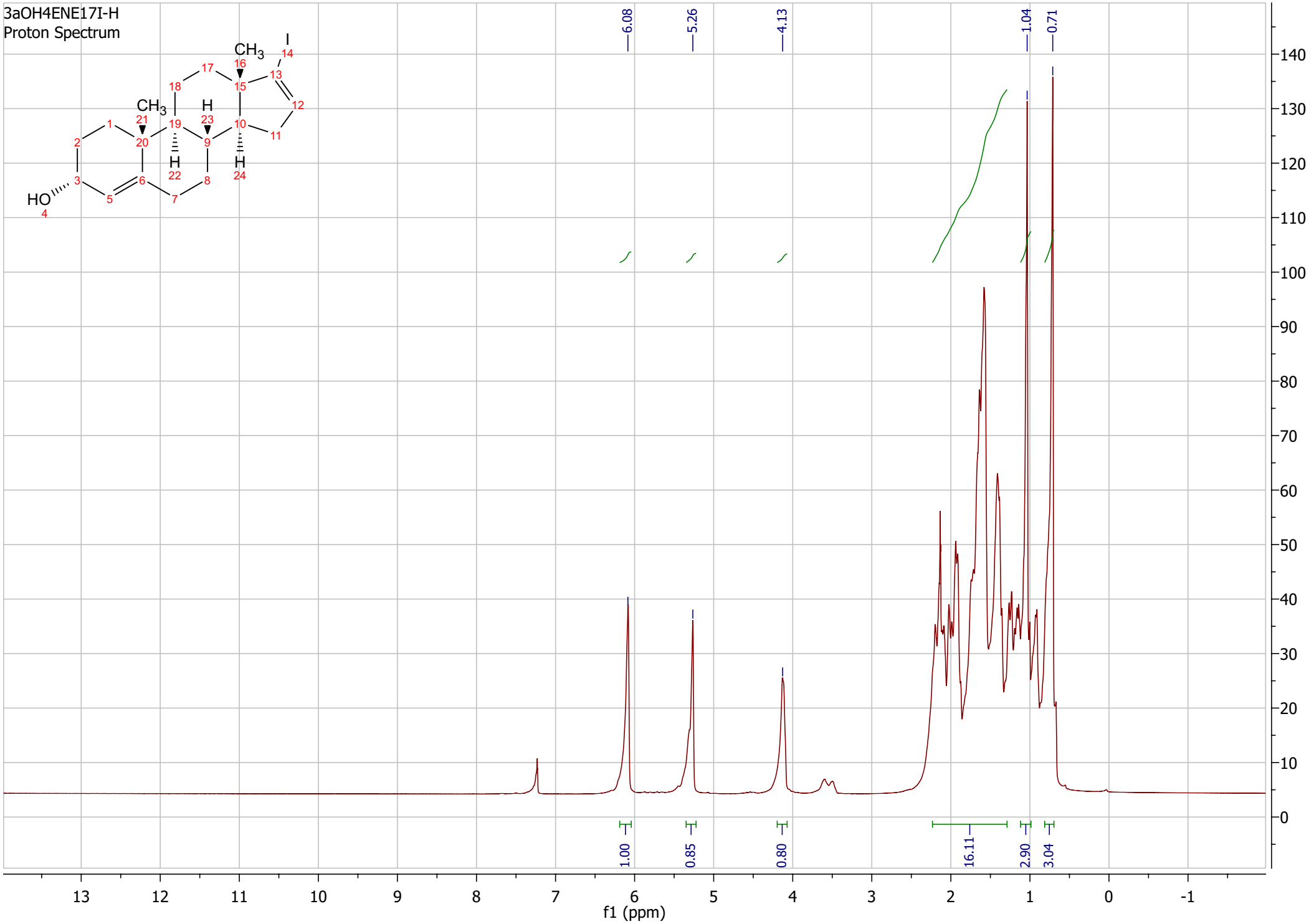
CH3
CH3
H
H
H
O
N
f1 (ppm)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Proton Spectrum
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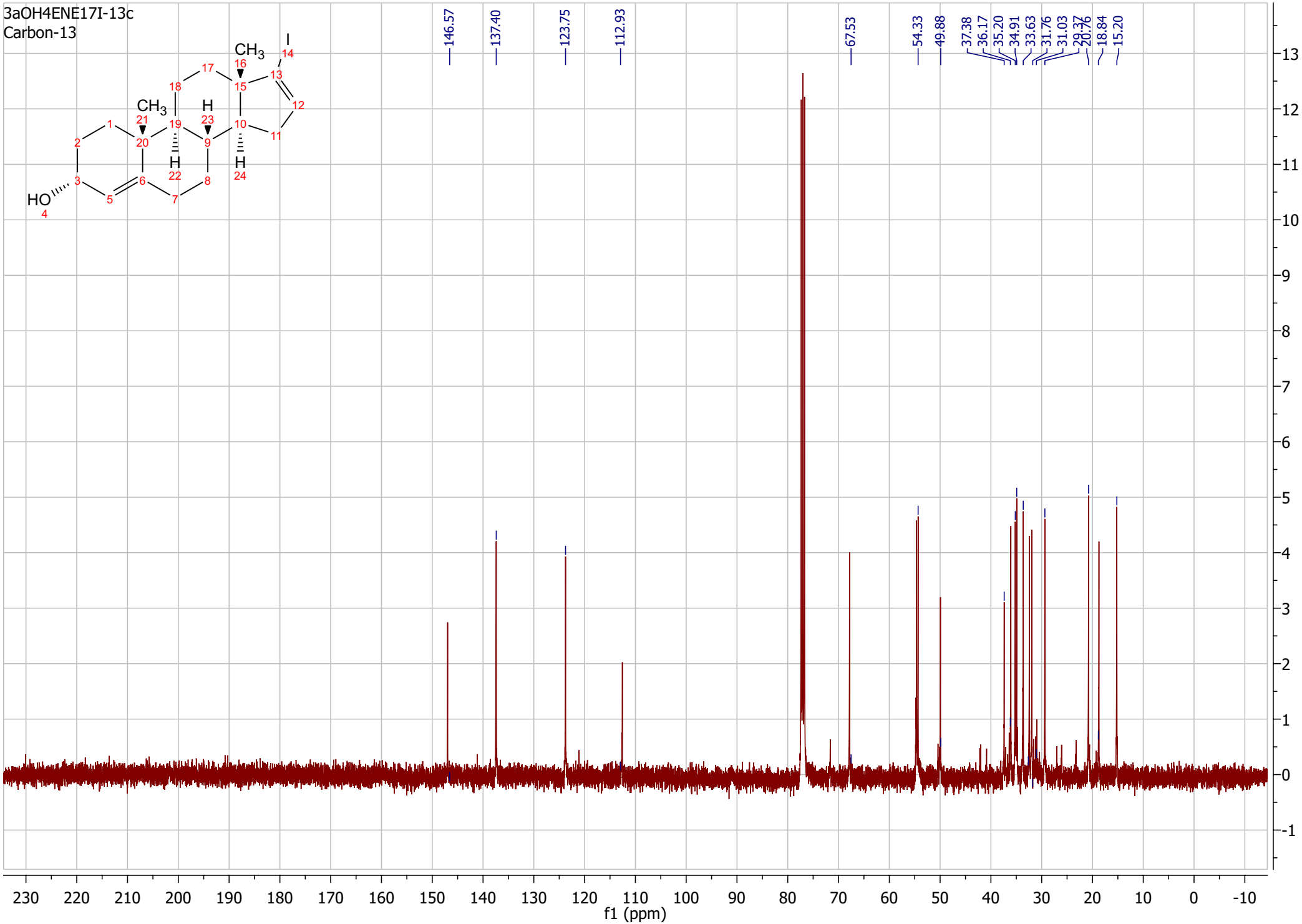
3.04
f1 (ppm)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Carbon-13
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67.53
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36.17
35.20
34.91
33.63
31.76
31.03
29.37
20.76
18.84
15.20
f1 (ppm)

This report was created by ACD/NMR Processor Academic Edition. For more information go to www.acdlabs.com/nmrproc/

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Points Count	32768	***Pulse Sequence***	s2pul	***Receiver Gain***	30.00	***Solvent***	CHLOROFORM-d
Spectrum Offset (Hz)	2397.2000	***Spectrum Type***	STANDARD	***Sweep Width (Hz)***	6377.55	***Temperature (degree C)***	AMBIENT TEMPERATURE

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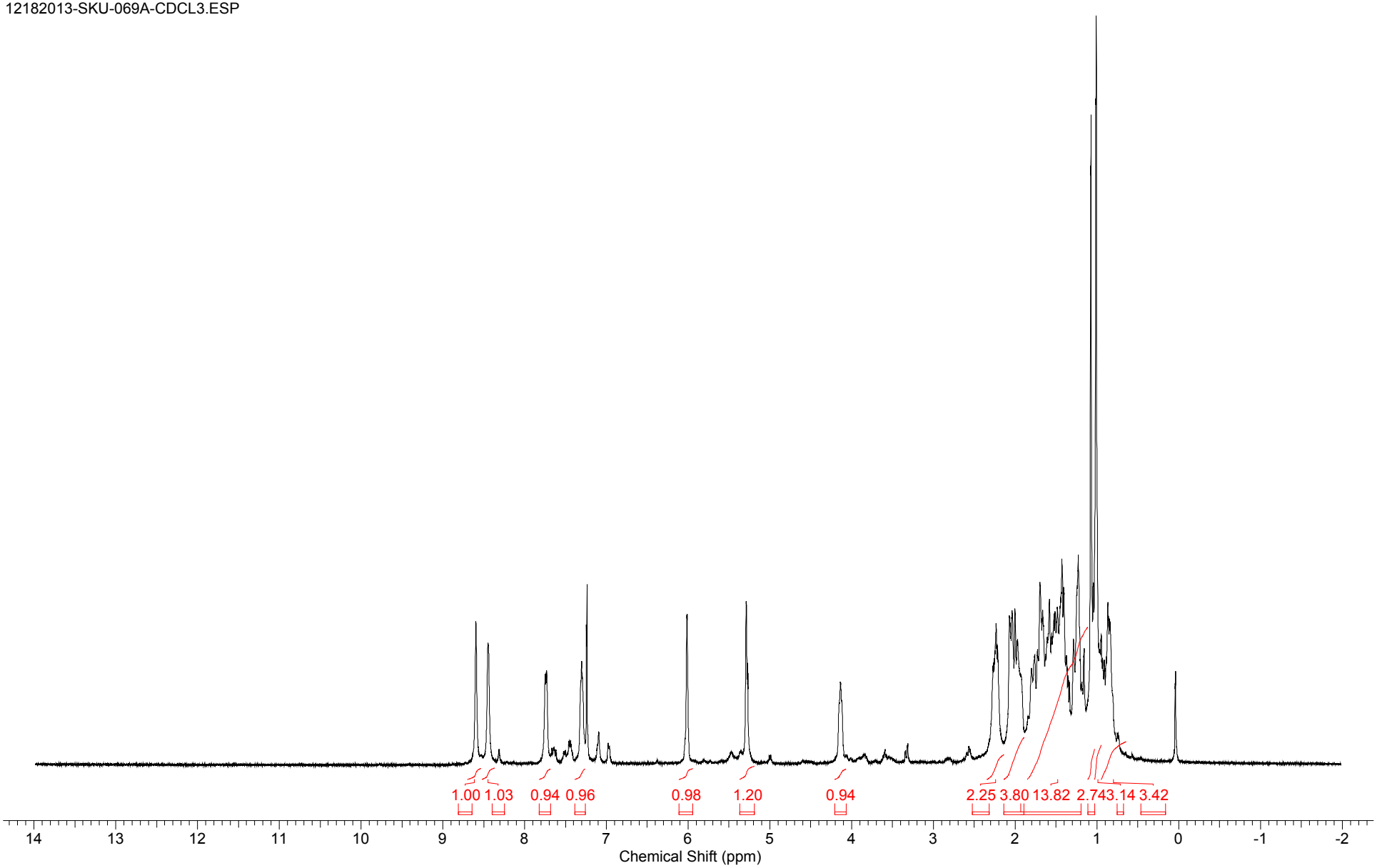

Gradient Shimming

Agilent Technologies

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ga.chem.lsa.umich.edu-vnmrs400
Archive directory:
/export/home/chempack/vnmrsys/data
Sample directory:

FidFile: 01072013-3a-OH-delta4-abi-1-C13

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Jan 7 2014

Operator: sunilku

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.621 sec
Width 25000.0 Hz
368 repetitions
OBSERVE C13, 100.4628324 MHz
DECOUPLE H1, 399.5357121 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
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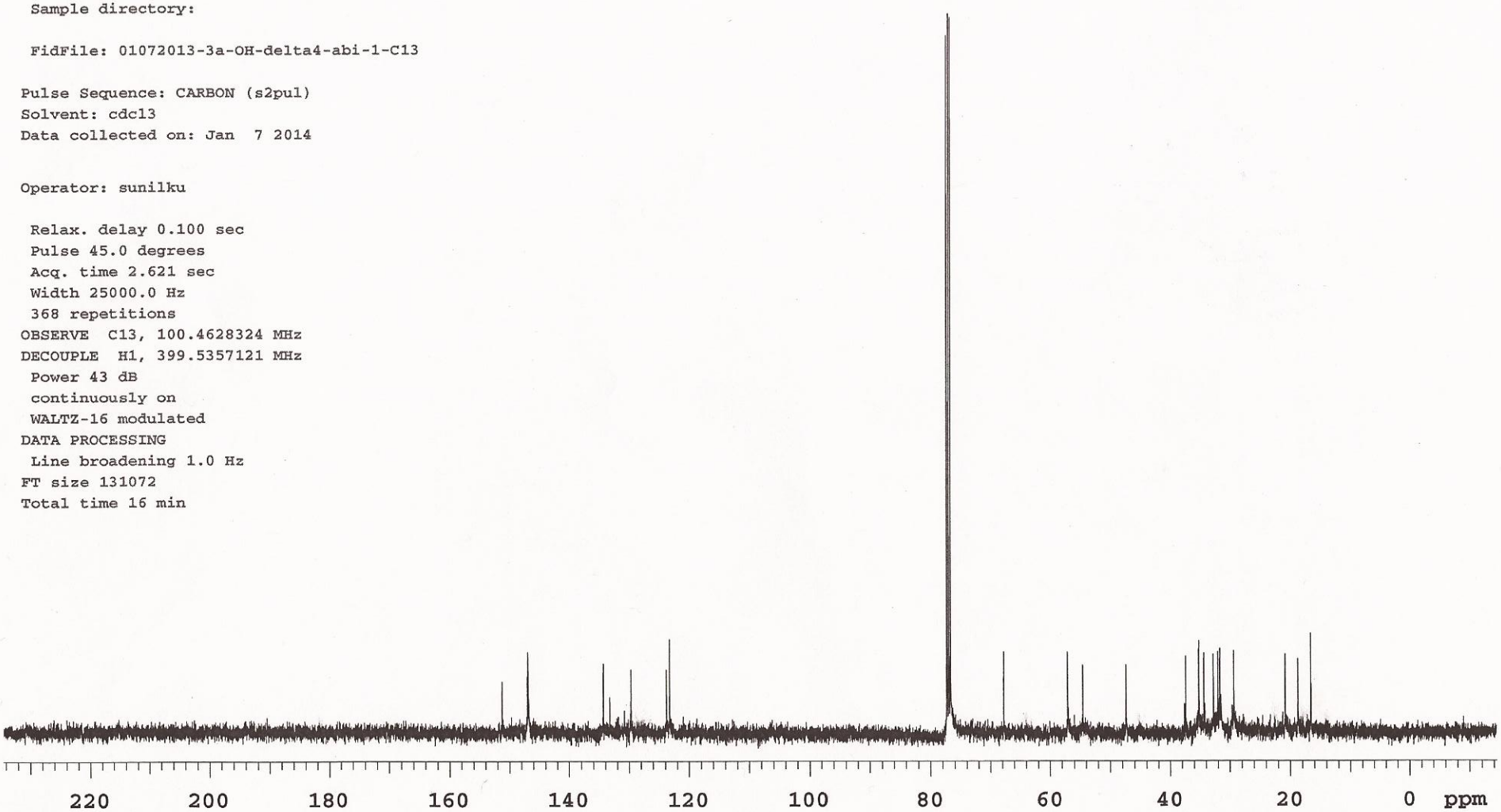
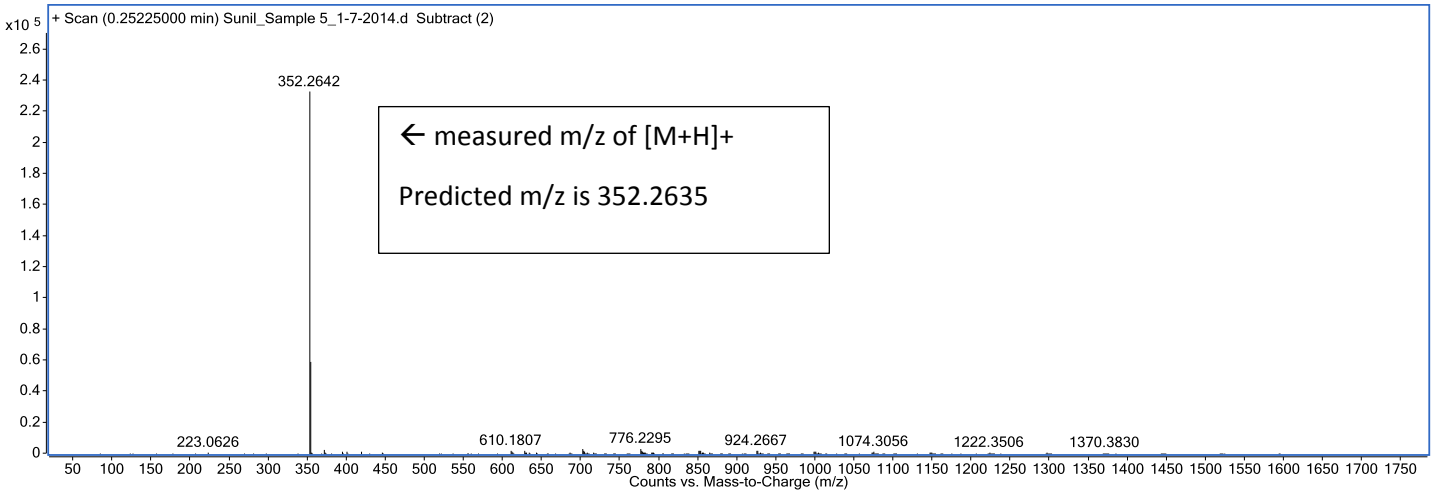
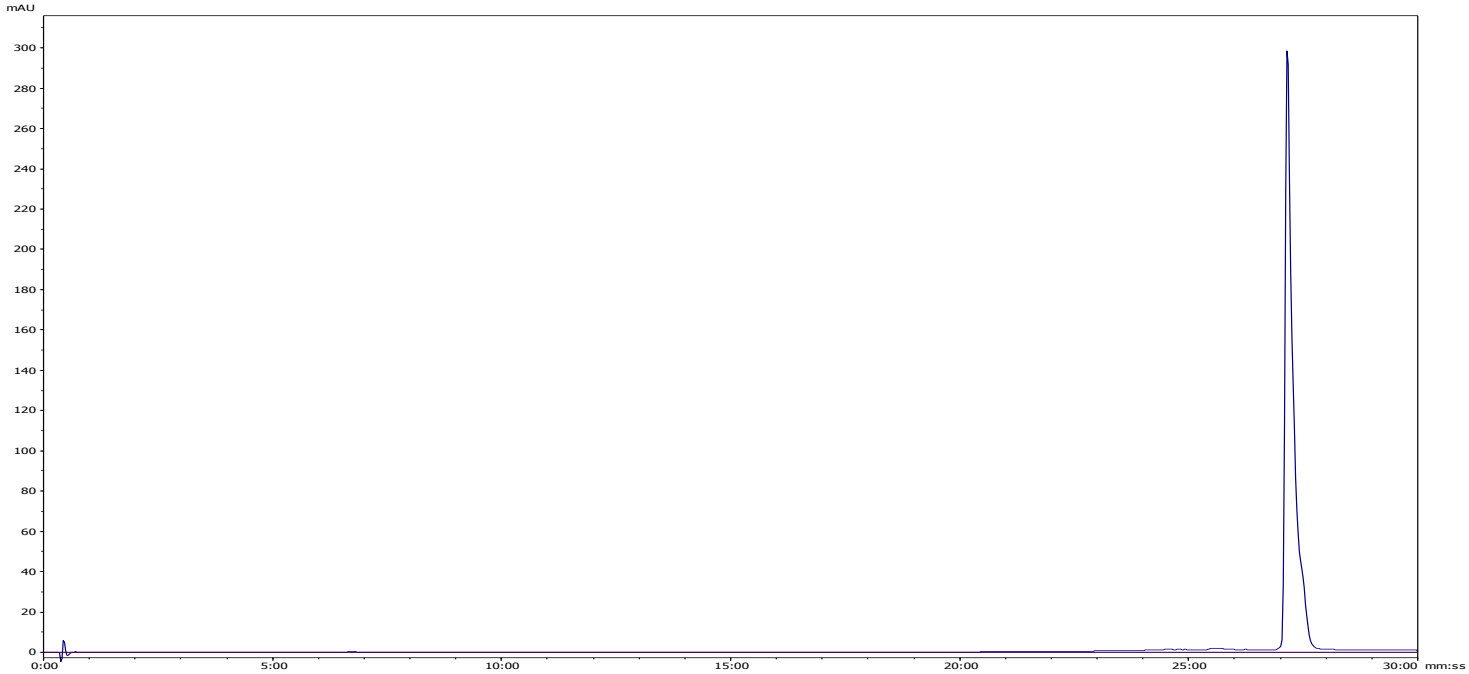
Total time 16 min



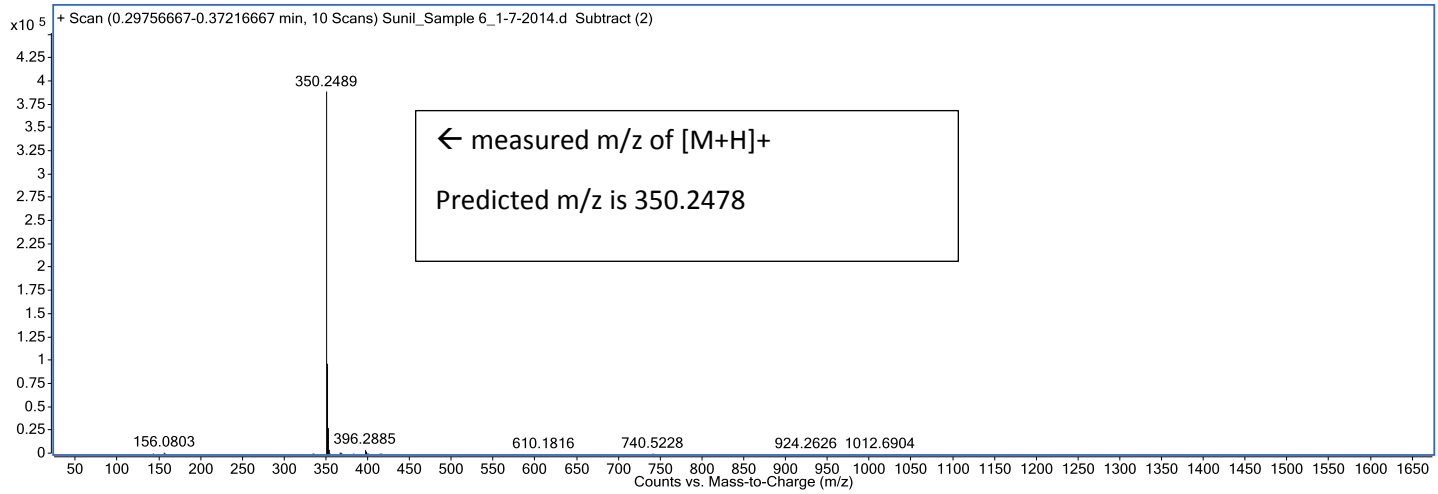
3α-OH-5 α-Abi (**5**) : by positive ion electrospray:



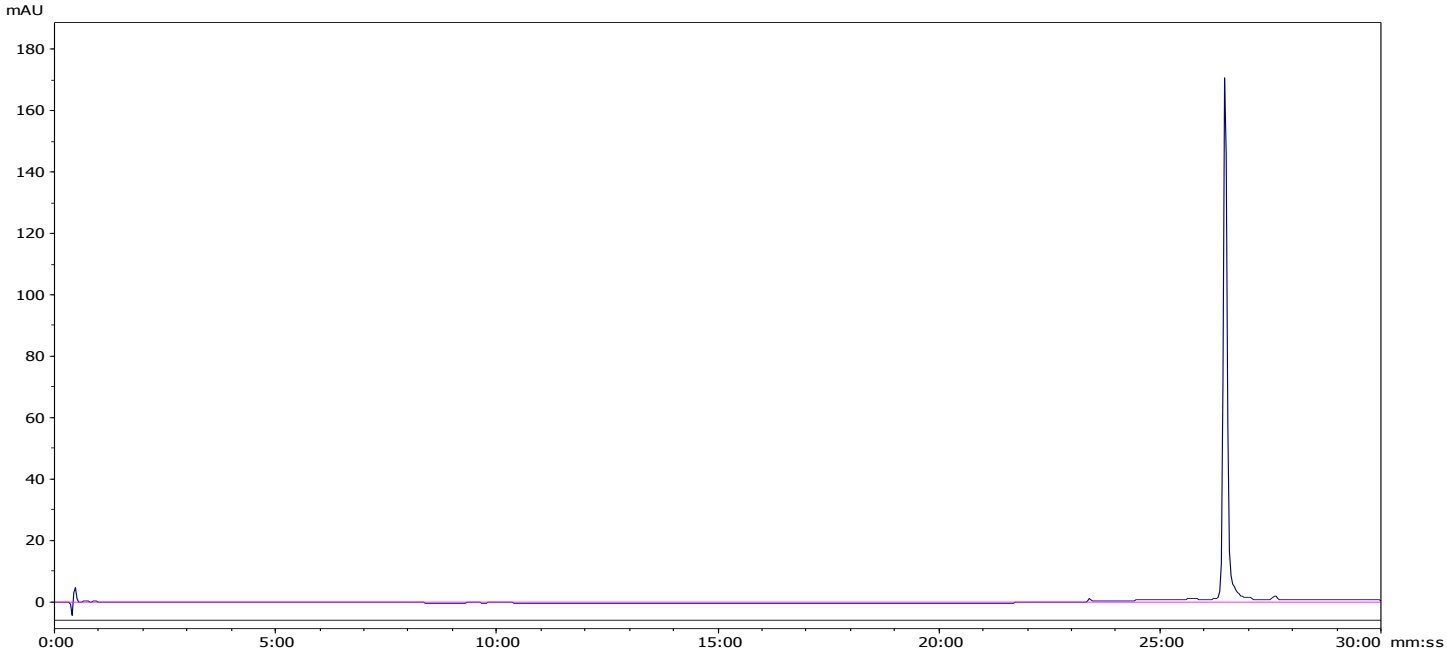
3α-OH-5 α-Abi (**5**) : HPLC-Profile:



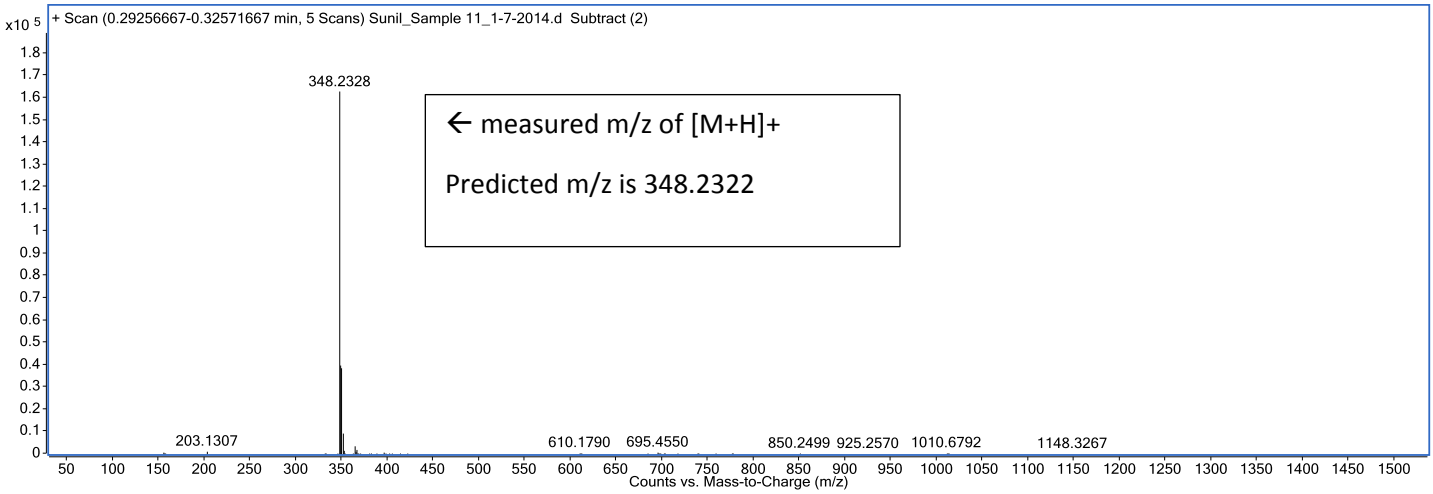
3-keto-5 α-Abi (**6**): by positive ion electrospray:



3-keto-5 α-Abi (**6**): HPLC-Profile:

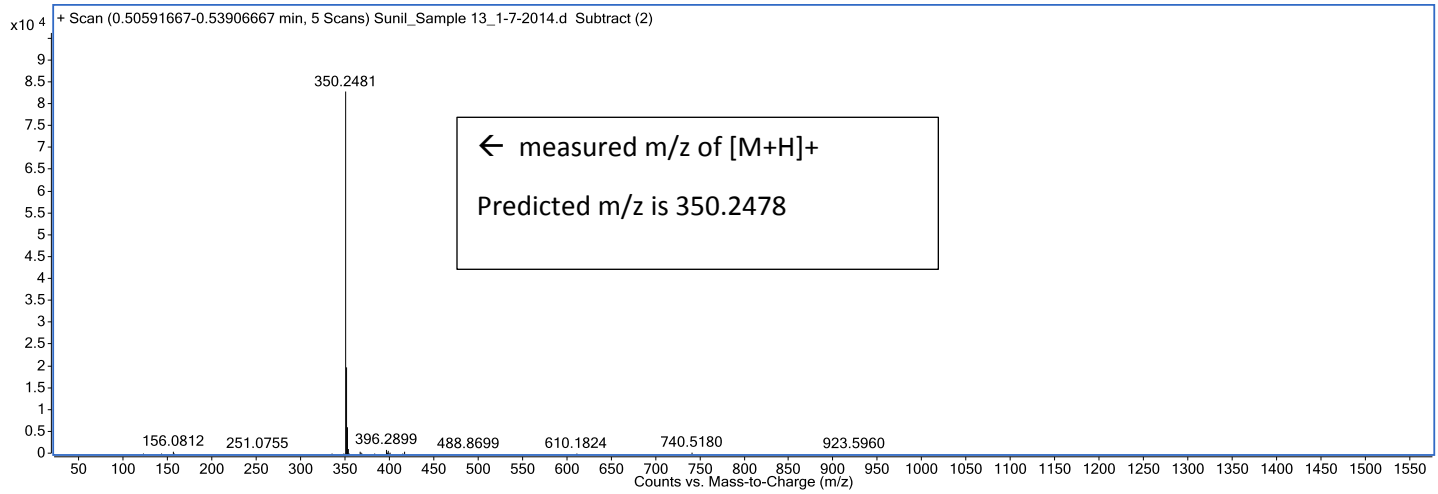
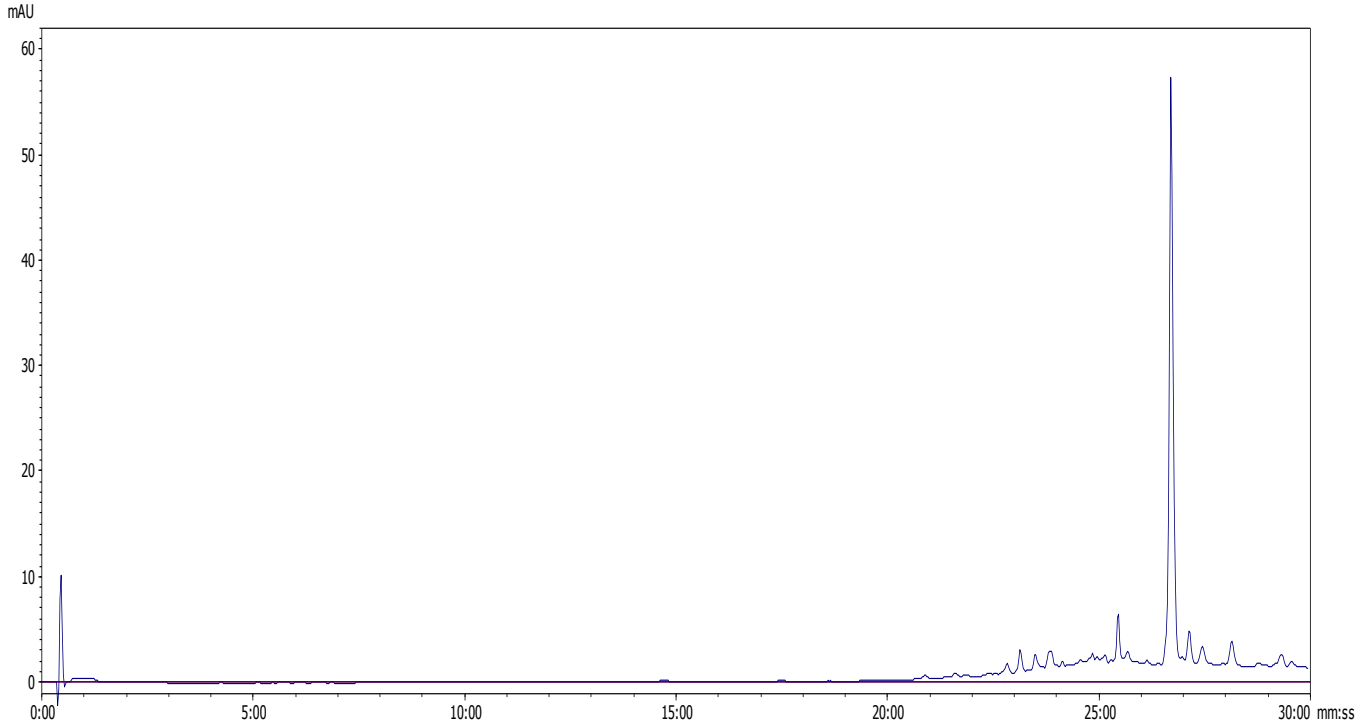


3-keto-Δ^4-Abi (**11**), by positive ion electrospray:



3-keto-Δ^4-Abi (**11**): HPLC-Profile:

3α-OH-Δ^4-Abi (**13**), by positive ion electrospray:



3α-OH-Δ^4-Abi (**13**), HPLC-Profile: