

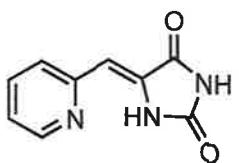
Supplementary Material for

Synthesis and Evaluation of 5-(Heteroaryl)methylene)hydantoins as Glycogen Synthase Kinase-3 β Inhibitors

Nicholas O. Schneider, Kendra Gilreath, Daniel J. Burkett, Martin St. Maurice and William A. Donaldson

This document includes:

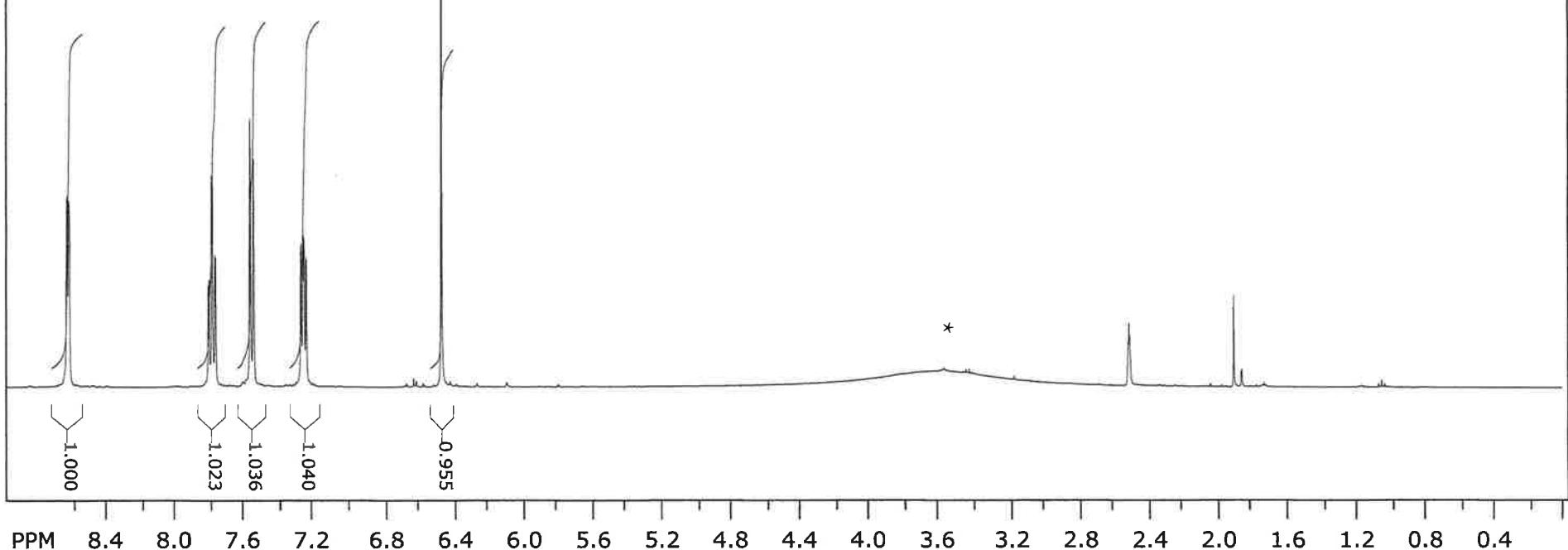
^1H NMR spectrum of 17 (d_6 -DMSO)	S2
^{13}C NMR spectrum of 17 (d_6 -DMSO)	S3
^1H NMR spectrum of 18 (d_6 -DMSO)	S4
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^{13}C NMR spectrum of 19 (d_6 -DMSO)	S6
^1H NMR spectrum of 15 (d_6 -DMSO)	S7
^{13}C NMR spectrum of 15 (d_6 -DMSO)	S8
^1H NMR spectrum of 20 (d_6 -DMSO)	S9
^{13}C NMR spectrum of 20 (d_6 -DMSO)	S10
^1H NMR spectrum of 21 (d_6 -DMSO)	S11
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^1H NMR spectrum of 22 (d_6 -DMSO)	S13
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^1H NMR spectrum of 25 (d_6 -DMSO)	S19
^{13}C NMR spectrum of 25 (d_6 -DMSO)	S20
^1H NMR spectrum of 26 (d_6 -DMSO)	S21
^{13}C NMR spectrum of 26 (d_6 -DMSO)	S22
^1H NMR spectrum of 27 (d_6 -DMSO)	S23
^{13}C NMR spectrum of 27 (d_6 -DMSO)	S24
^1H NMR spectrum of 28 (d_6 -DMSO)	S25
^{13}C NMR spectrum of 28 (d_6 -DMSO)	S26
^1H NMR spectrum of 29 (d_6 -DMSO)	S27
^{13}C NMR spectrum of 29 (d_6 -DMSO)	S28



¹H NMR
(400 MHz, d₆-DMSO)

*moisture in DMSO

17

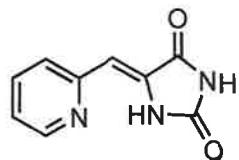


file: ...oton-DMSO-reflux-overnight.fid\fid block# 1 expt: "s2pul"
transmitter freq.: 399.732139 MHz
time domain size: 32768 points
width: 6410.26 Hz = 16.0364 ppm = 0.195626 Hz/pt
number of scans: 8

freq. of 0 ppm: 399.729732 MHz
processed size: 32768 complex points
LB: 0.500 GF: 0.0000
Hz/cm: 143.875 ppm/cm: 0.35993

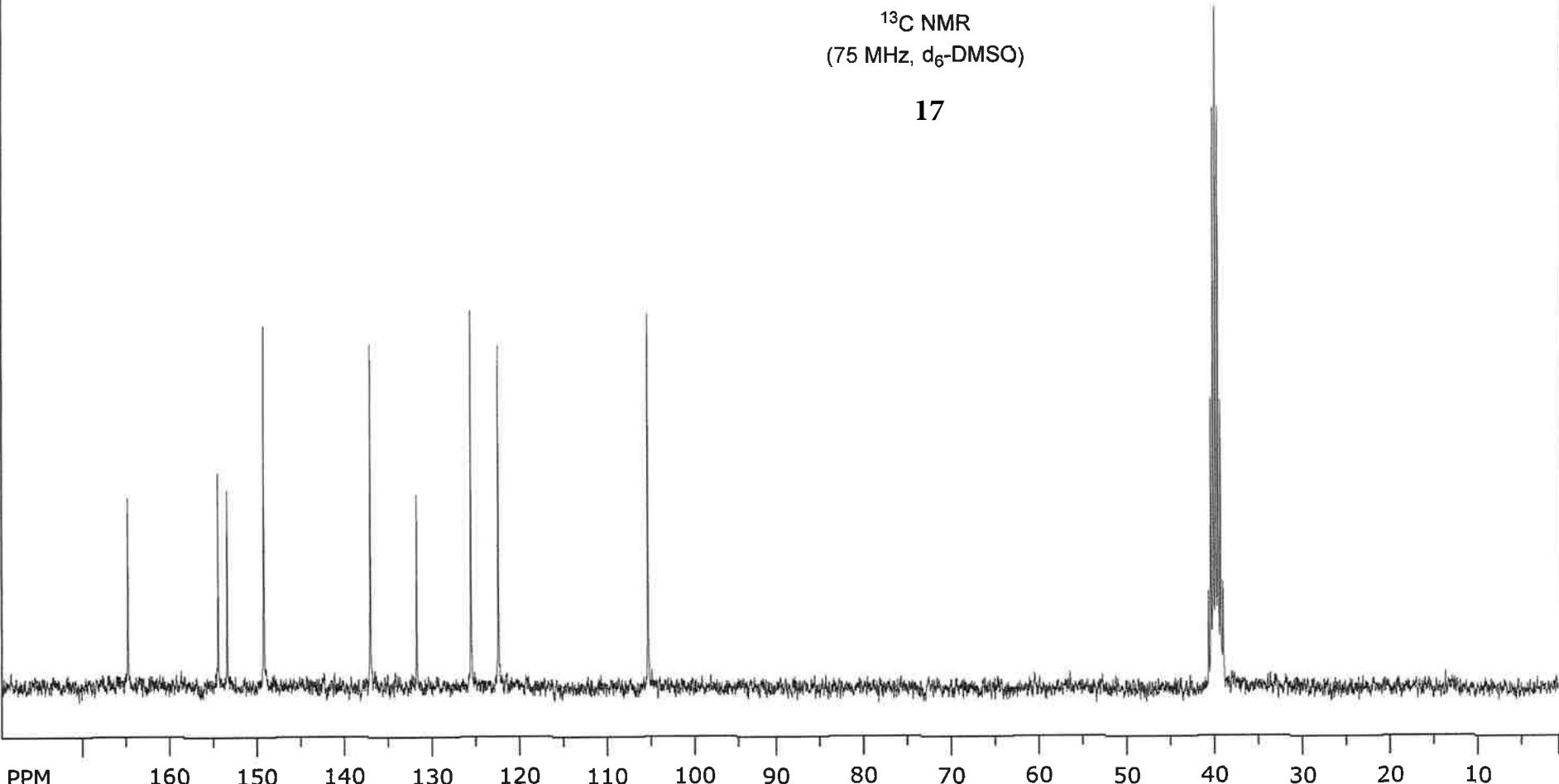
SpinWorks 4: 13C OBSERVE

165.269 —
149.518 —
153.757 —
154.808 —
137.189 —
131.836 —
125.552 —
122.392 —
105.099 —



13C NMR
(75 MHz, d₆-DMSO)

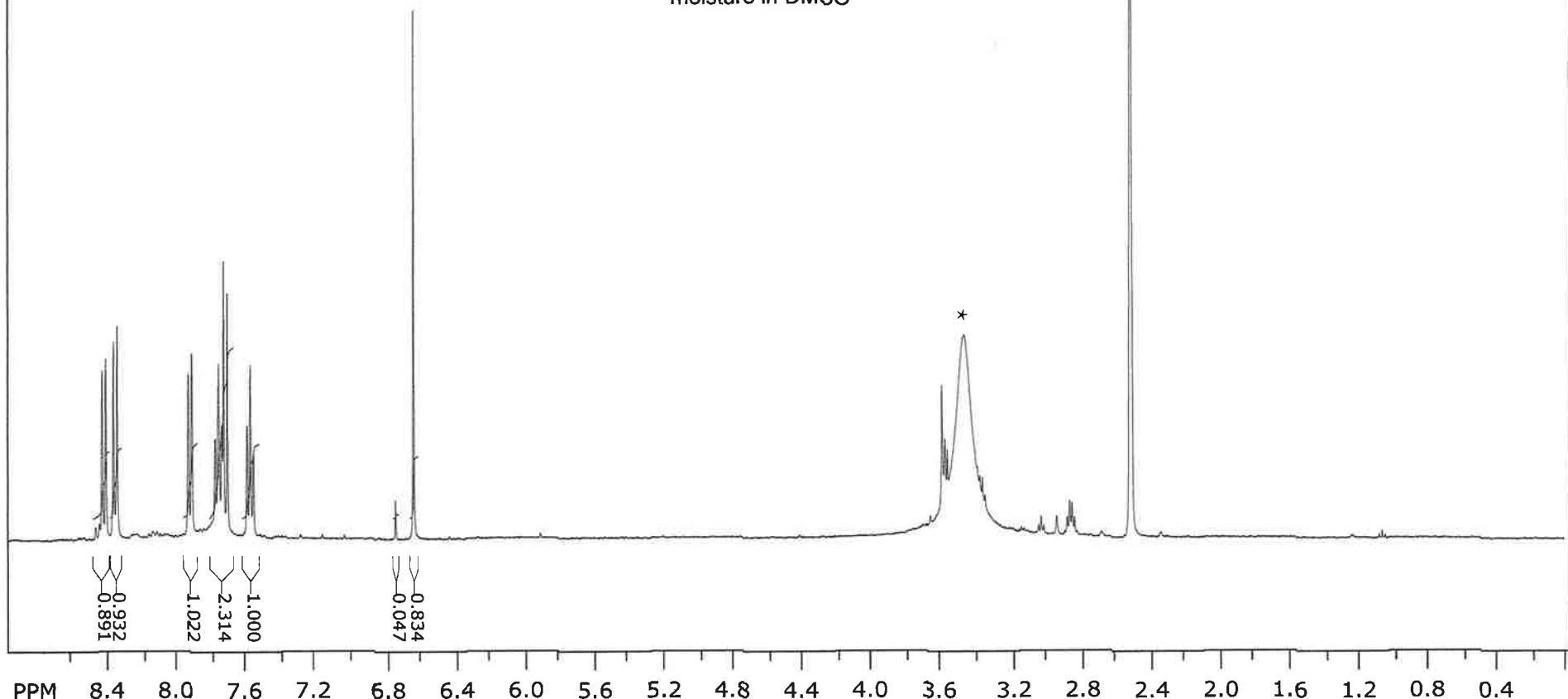
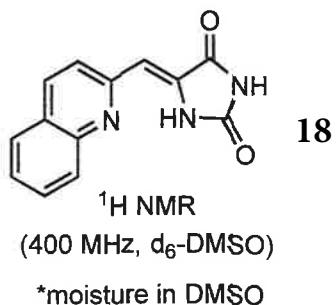
17



file: ...ldsonw\Desktop\DJB-234-13C.fid\fid block# 1 expt: "s2pul"
transmitter freq.: 75.476694 MHz
time domain size: 68492 points
width: 18867.92 Hz = 249.9835 ppm = 0.275476 Hz/pt
number of scans: 256

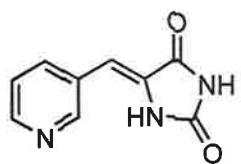
freq. of 0 ppm: 75.468435 MHz
processed size: 131072 complex points
LB: 2.500 GF: 0.0000
Hz/cm: 543.396 ppm/cm: 7.19952

SpinWorks 4: DJB-331-proton



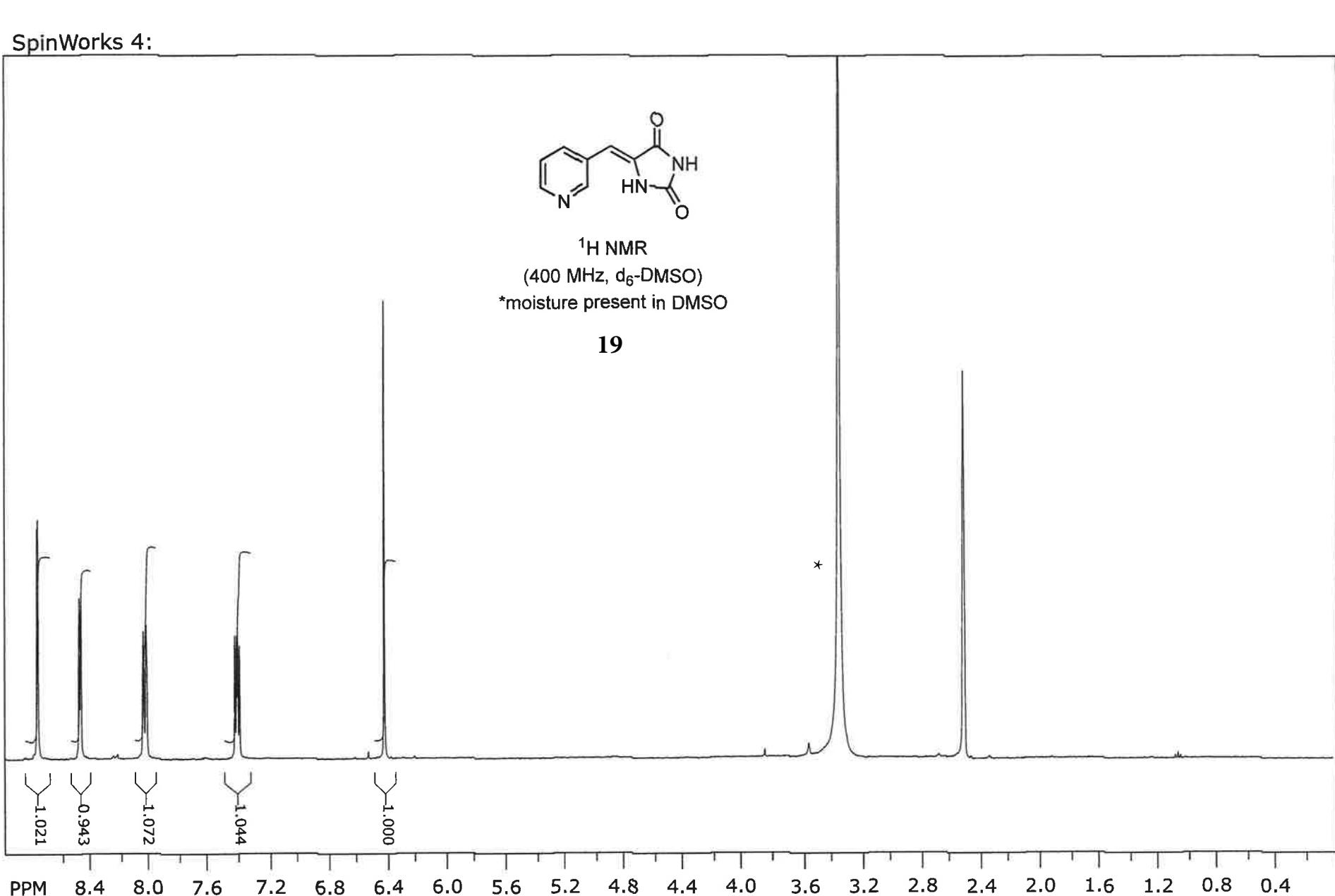
file: ...DJB spectra\DJB-334-proton.fid\fid block# 1 expt: "s2pul"
transmitter freq.: 399.736048 MHz
time domain size: 32768 points
width: 6410.26 Hz = 16.0362 ppm = 0.195626 Hz/pt
number of scans: 8

freq. of 0 ppm: 399.733634 MHz
processed size: 32768 complex points
LB: 0.500 GF: 0.0000
Hz/cm: 143.875 ppm/cm: 0.35992



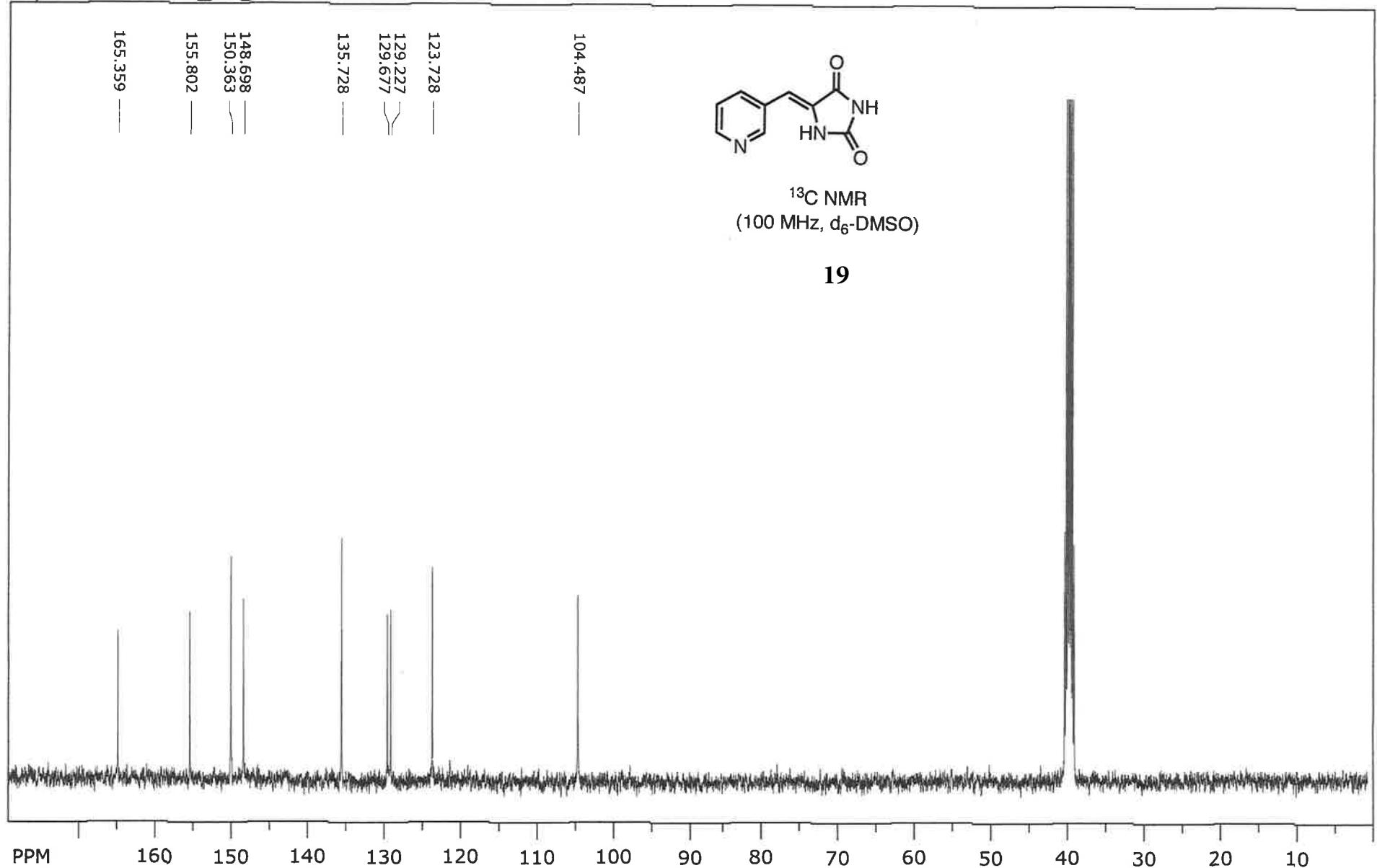
¹H NMR
(400 MHz, d₆-DMSO)
*moisture present in DMSO

19



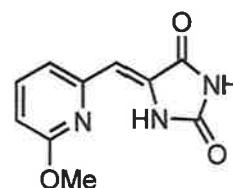
file: ...DJB spectra\DJB-235-proton.fid\fid block# 1 expt: "s2pul"
transmitter freq.: 399.738002 MHz
time domain size: 26264 points
width: 6410.26 Hz = 16.0361 ppm = 0.244070 Hz/pt
number of scans: 8

freq. of 0 ppm: 399.735598 MHz
processed size: 65536 complex points
LB: 0.500 GF: 0.0000
Hz/cm: 143.875 ppm/cm: 0.35992



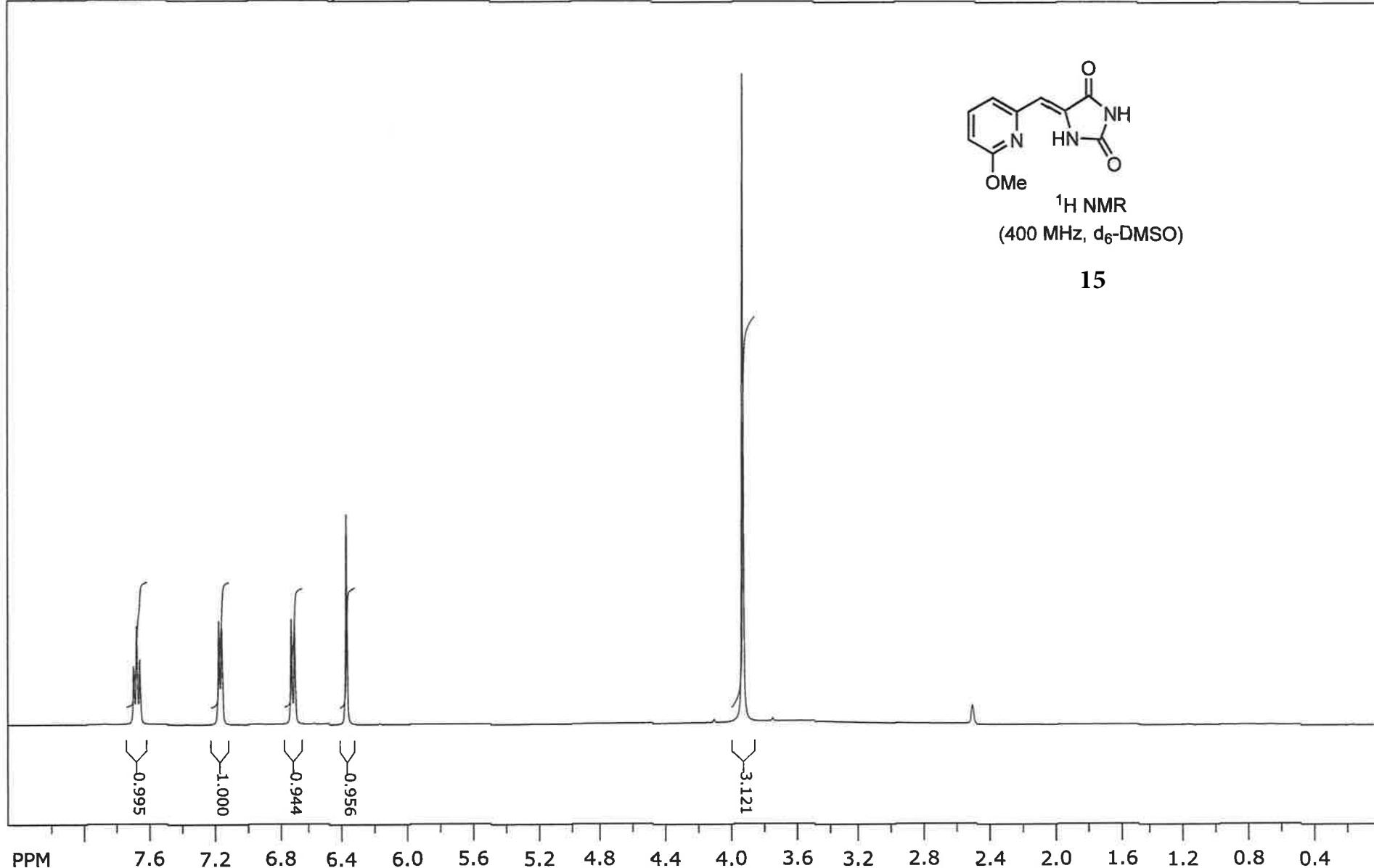
file: ...on\WD325_13C_DMSO_20240116.fid fid block# 1 expt: "s2pul"
transmitter freq.: 100.523180 MHz
time domain size: 65536 points
width: 25000.00 Hz = 248.6989 ppm = 0.381470 Hz/pt
number of scans: 2048

freq. of 0 ppm: 100.512160 MHz
processed size: 65536 complex points
LB: 2.500 GF: 0.0000
Hz/cm: 723.333 ppm/cm: 7.19569



¹H NMR
(400 MHz, d₆-DMSO)

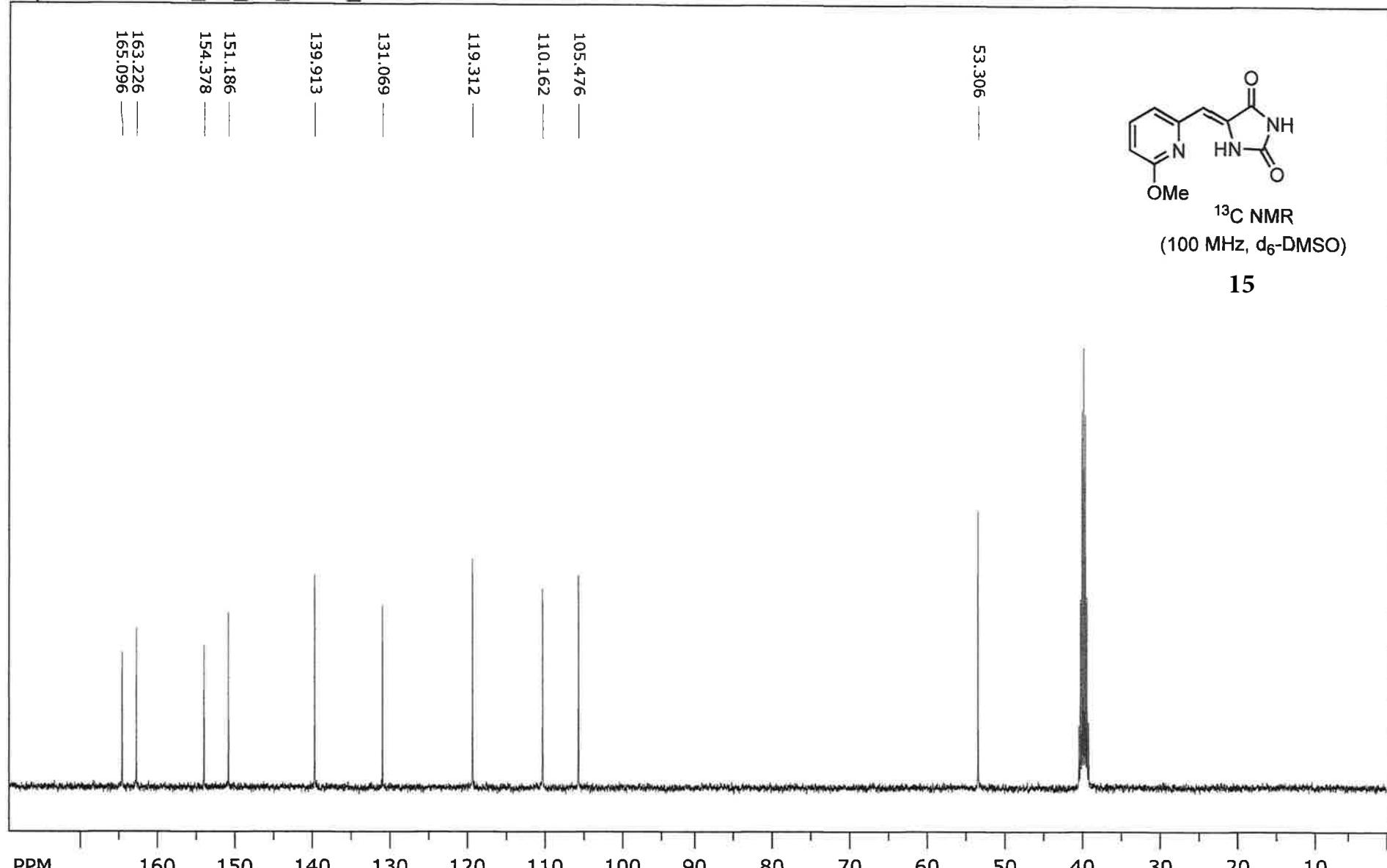
15



file: ...spectra\WD340-349\WD340-1H.fid fid block# 1 expt: "s2pul"
transmitter freq.: 399.732139 MHz
time domain size: 32768 points
width: 6410.26 Hz = 16.0364 ppm = 0.195626 Hz/pt
number of scans: 8

freq. of 0 ppm: 399.729734 MHz
processed size: 32768 complex points
LB: 0.500 GF: 0.0000
Hz/cm: 135.897 ppm/cm: 0.33997

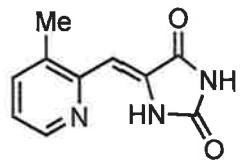
SpinWorks 4: 7_27_20_Ester_carbon



file: ...ectra\WD spectra\WD340-13C.fid\fid block# 1 expt: "s2pul"
transmitter freq.: 100.523180 MHz
time domain size: 65536 points
width: 25000.00 Hz = 248.6989 ppm = 0.381470 Hz/pt
number of scans: 256

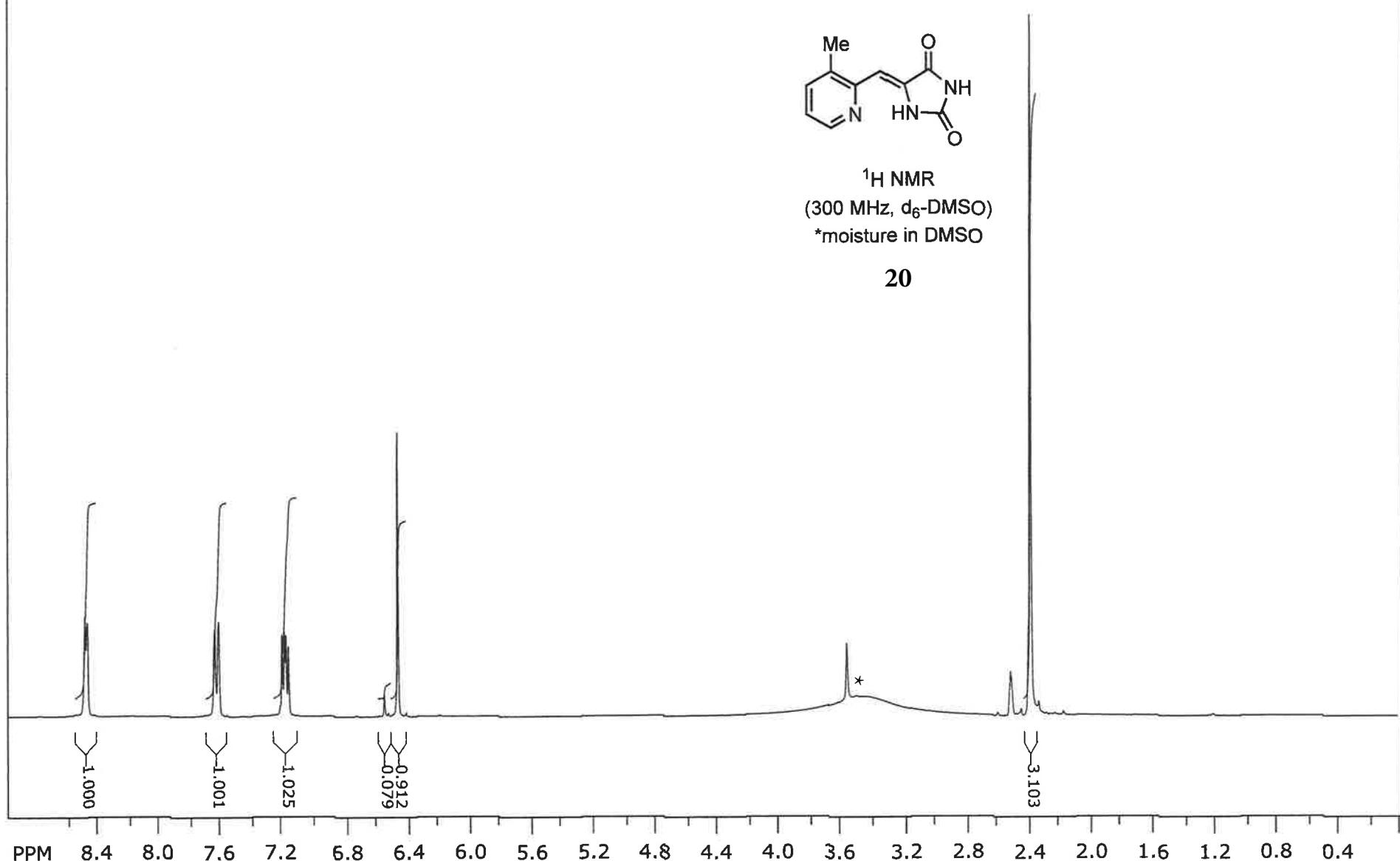
freq. of 0 ppm: 100.512156 MHz
processed size: 65536 complex points
LB: 1.500 GF: 0.0000
Hz/cm: 723.889 ppm/cm: 7.20121

SpinWorks 4: STANDARD 1H OBSERVE



¹H NMR
(300 MHz, d₆-DMSO)
*moisture in DMSO

20

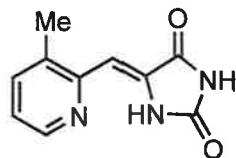


file: ...spectra\WD320-329\WD-328_1H.fid fid block# 1 expt: "s2pu"
transmitter freq.: 300.134434 MHz
time domain size: 19192 points
width: 4803.07 Hz = 16.0031 ppm = 0.250264 Hz/pt
number of scans: 8

freq. of 0 ppm: 300.132628 MHz
processed size: 32768 complex points
LB: 0.500 GF: 0.0000
Hz/cm: 108.016 ppm/cm: 0.35989

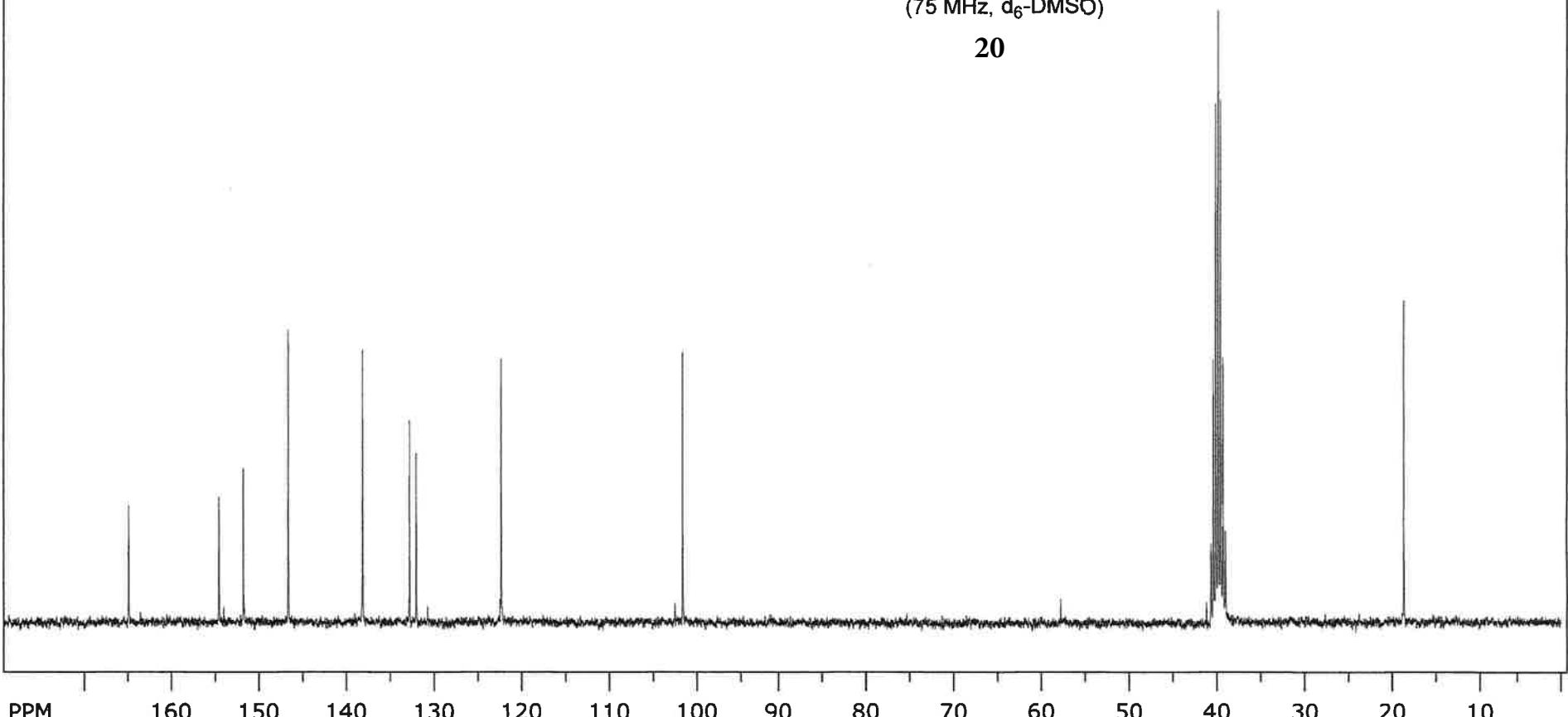
SpinWorks 4: 13C OBSERVE

165.395 —
154.924 —
146.902 —
152.101 —
138.308 —
132.902 —
132.131 —
122.311 —
101.358 —
18.117 —



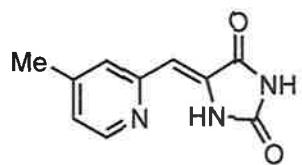
13C NMR
(75 MHz, d₆-DMSO)

20



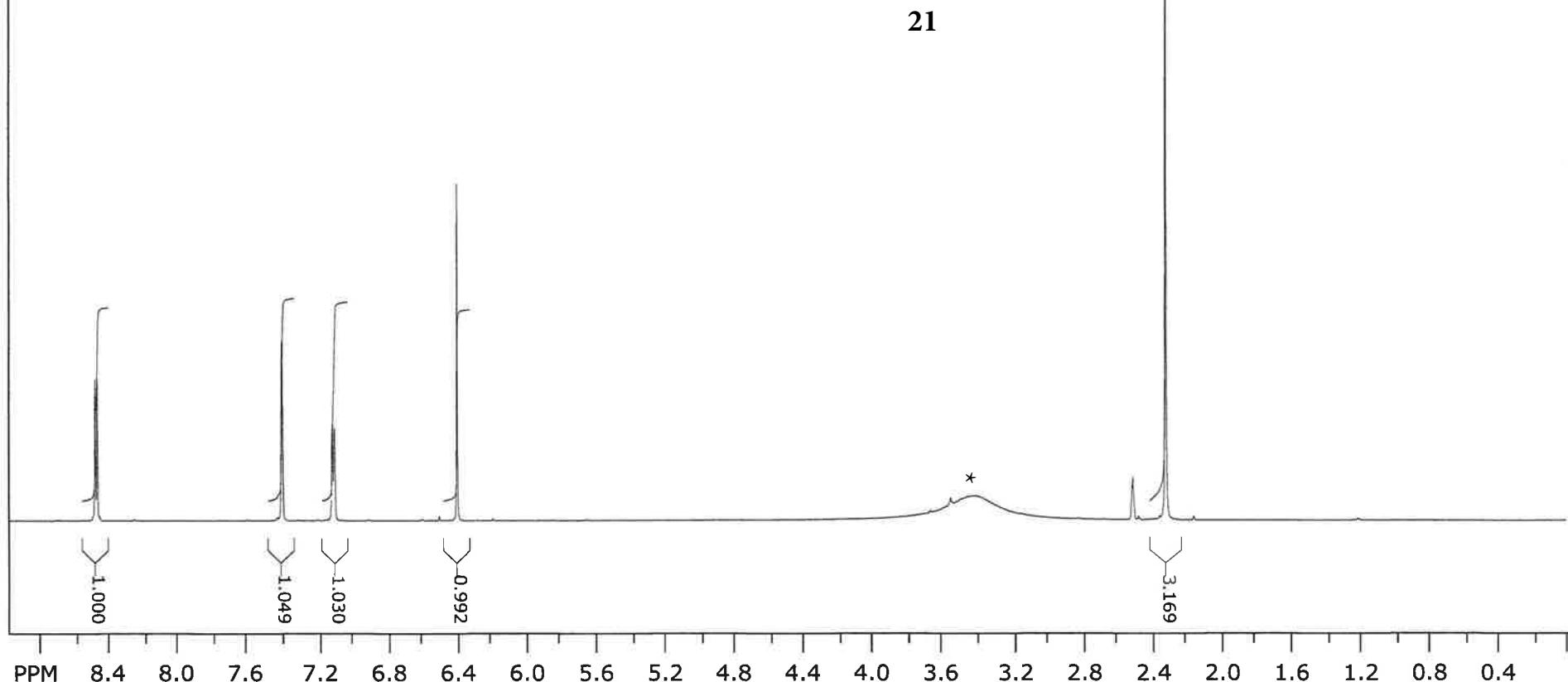
file: ...ctra\WD spectra\WD-328_13C.fid\fld block# 1 expt: "s2pul"
transmitter freq.: 75.476694 MHz
time domain size: 68492 points
width: 18867.92 Hz = 249.9835 ppm = 0.275476 Hz/pt
number of scans: 384

freq. of 0 ppm: 75.468436 MHz
processed size: 131072 complex points
LB: 1.500 GF: 0.0000
Hz/cm: 543.396 ppm/cm: 7.19952



¹H NMR
(400 MHz, d₆-DMSO)
*moisture in DMSO

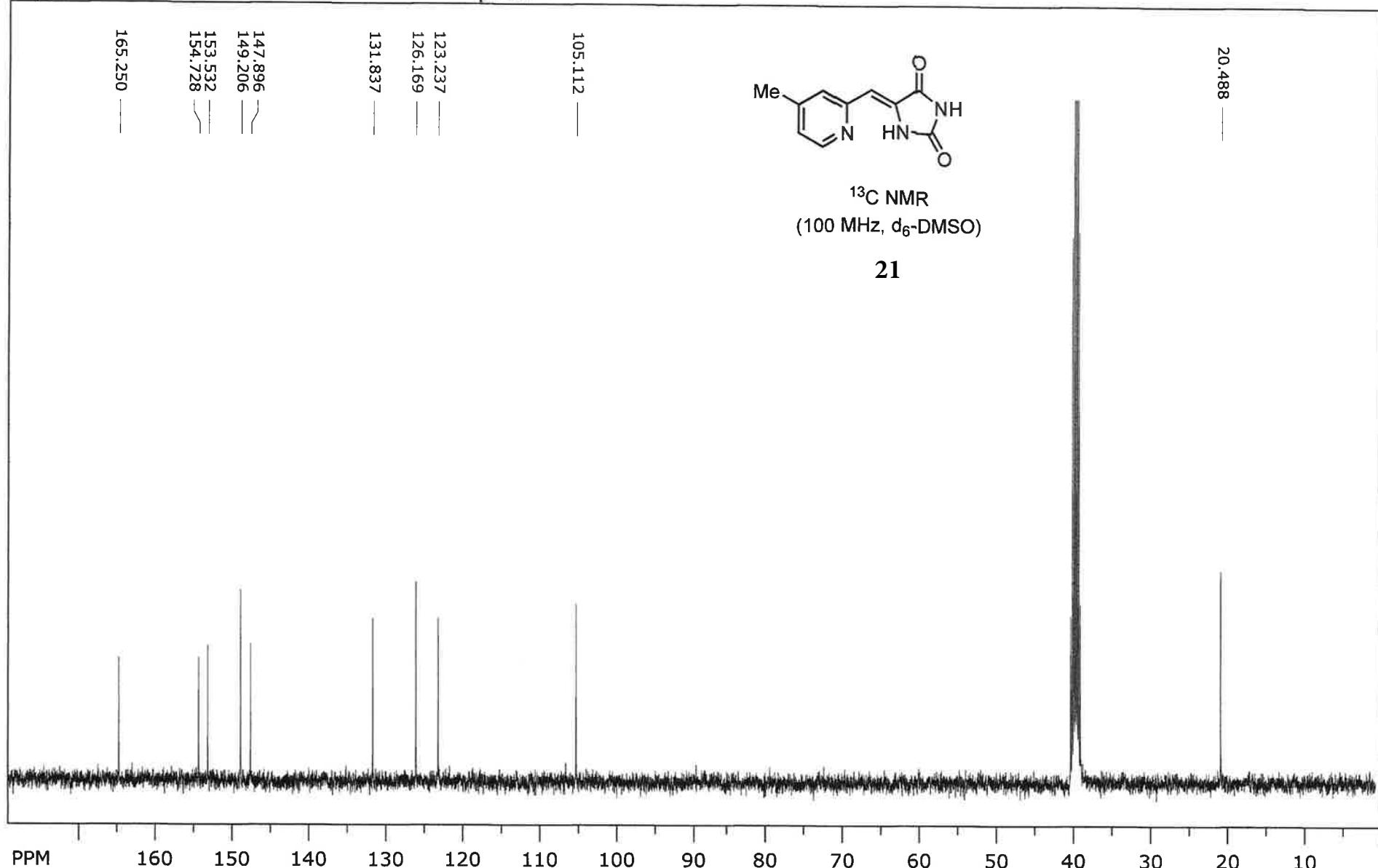
21



file: ...pectra\WD spectra\WD342_1H.fid fid block# 1 expt: "s2pu!"
transmitter freq.: 399.732139 MHz
time domain size: 32768 points
width: 6410.26 Hz = 16.0364 ppm = 0.195626 Hz/pt
number of scans: 8

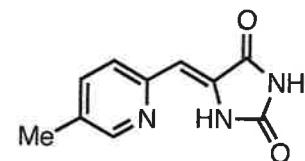
freq. of 0 ppm: 399.729733 MHz
processed size: 32768 complex points
LB: 0.051 GF: 0.0000
Hz/cm: 144.017 ppm/cm: 0.36028

SpinWorks 4: STANDARD 1H OBSERVE - profile



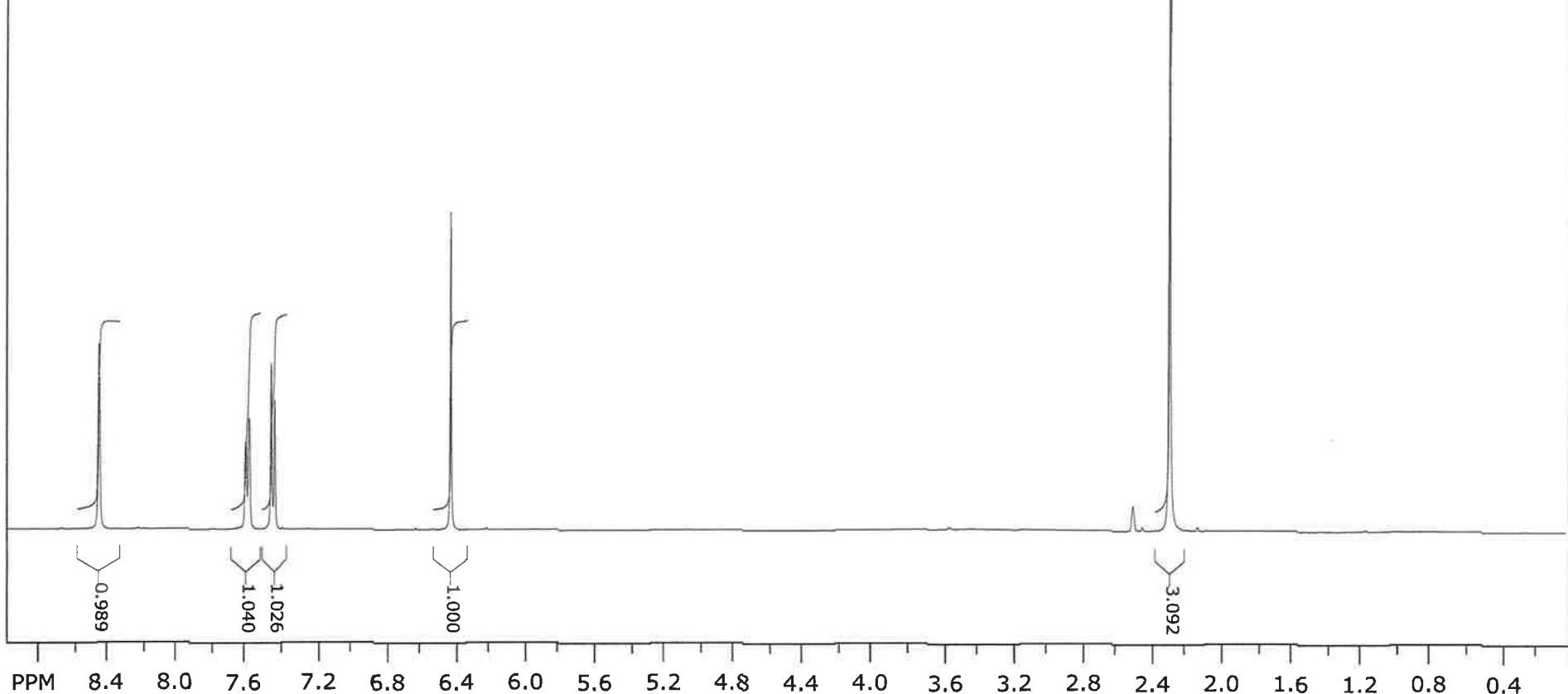
file: ...ectra\WD spectra\WD342_13C.fid fid block# 1 expt: "s2pul"
transmitter freq.: 100.523180 MHz
time domain size: 65536 points
width: 25000.00 Hz = 248.6989 ppm = 0.381470 Hz/pt
number of scans: 512

freq. of 0 ppm: 100.512160 MHz
processed size: 65536 complex points
LB: 1.500 GF: 0.0000
Hz/cm: 723.333 ppm/cm: 7.19569



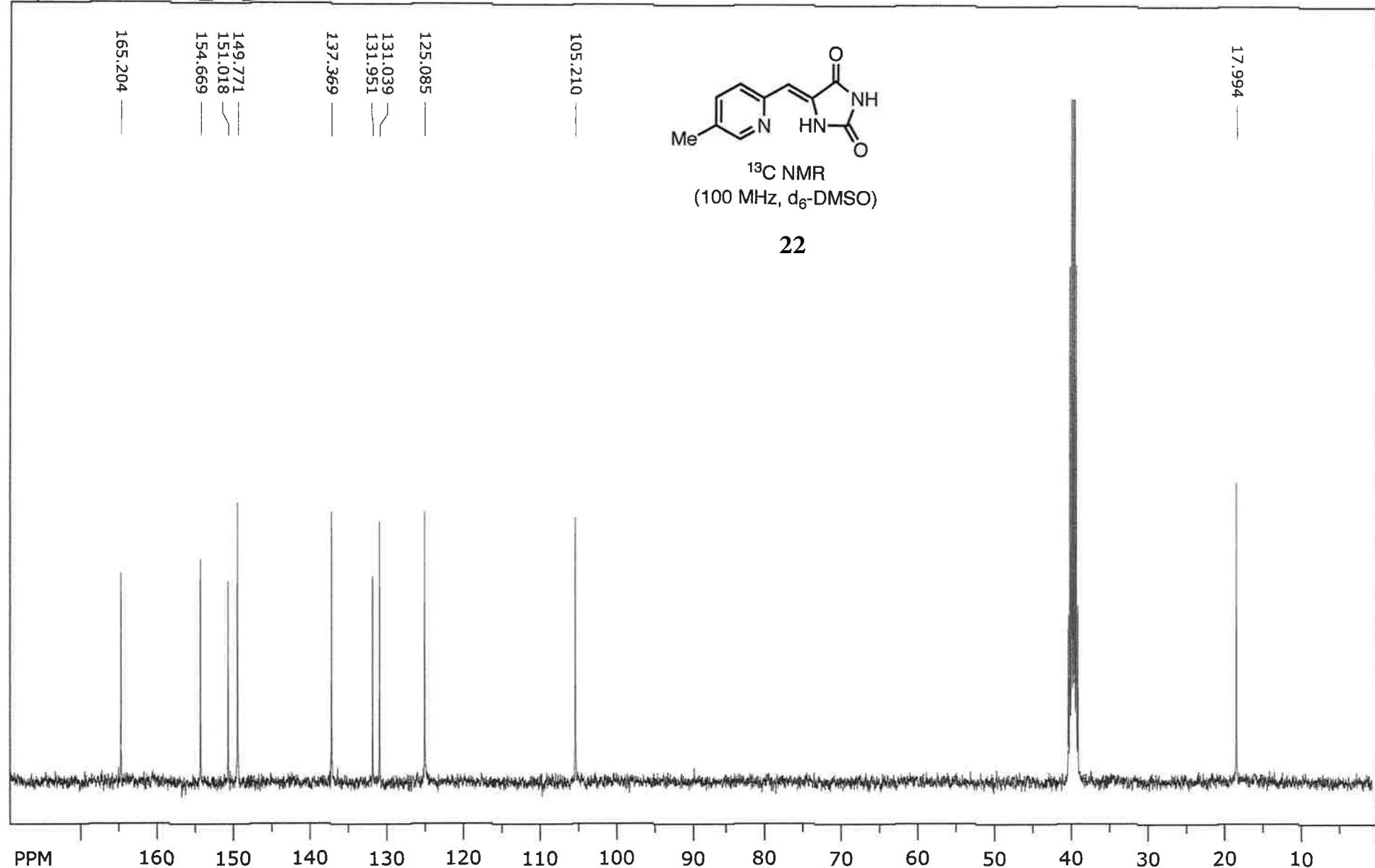
¹H NMR
(400 MHz, d₆-DMSO)

22



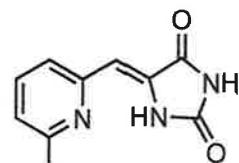
file: ...son\WD348_1H_DMSO_20240118.fid\fid block# 1 expt: "s2pul"
transmitter freq.: 399.732139 MHz
time domain size: 32768 points
width: 6410.26 Hz = 16.0364 ppm = 0.195626 Hz/pt
number of scans: 16

freq. of 0 ppm: 399.729733 MHz
processed size: 32768 complex points
LB: 0.500 GF: 0.0000
Hz/cm: 143.875 ppm/cm: 0.35993



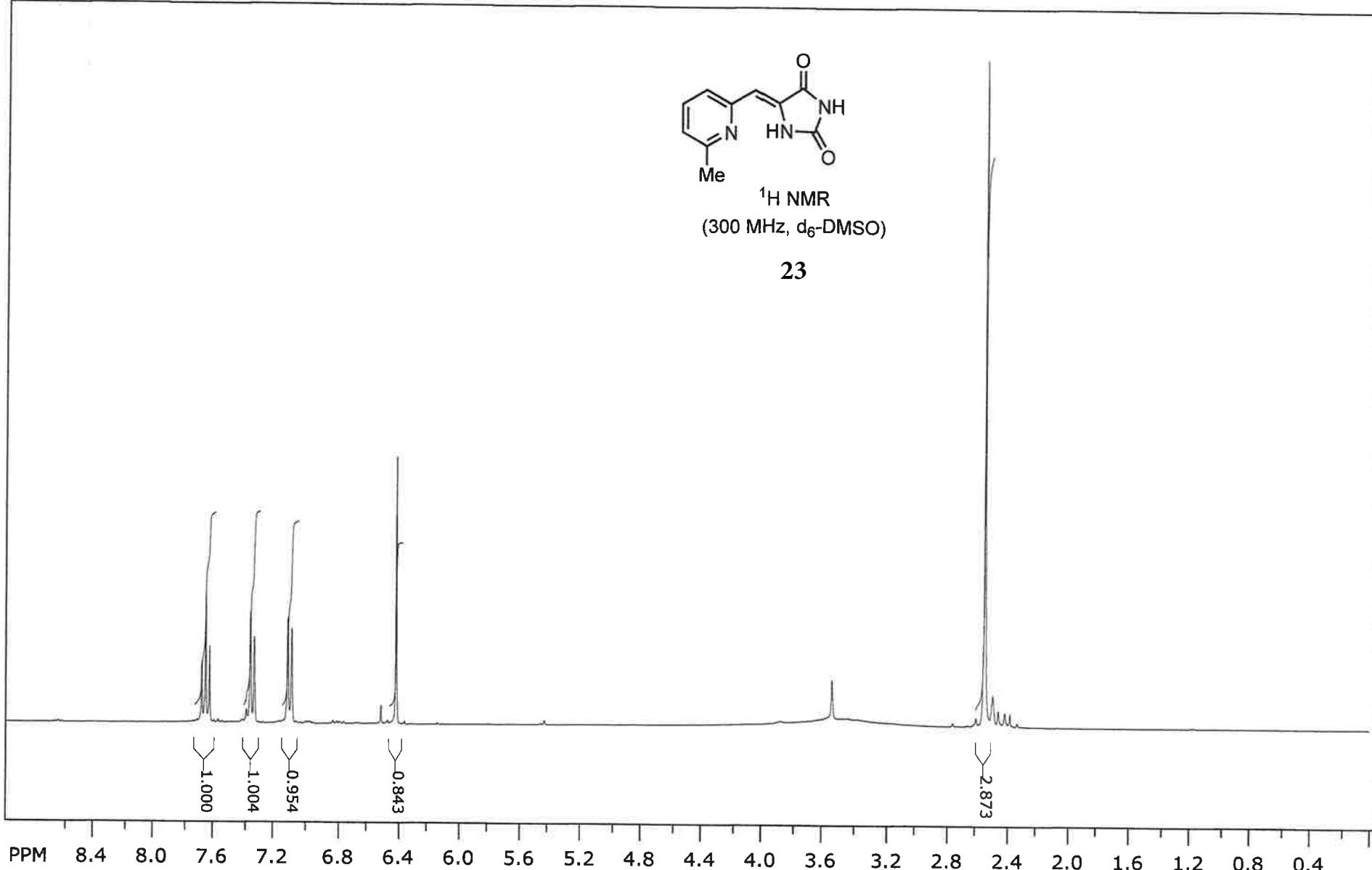
file: ...on\WD348_13C_DMSO_20240118.fid fid block# 1 expt: "s2pul"
transmitter freq.: 100.523180 MHz
time domain size: 65536 points
width: 25000.00 Hz = 248.6989 ppm = 0.381470 Hz/pt
number of scans: 2048

freq. of 0 ppm: 100.512160 MHz
processed size: 65536 complex points
LB: 2.500 GF: 0.0000
Hz/cm: 723.333 ppm/cm: 7.19569



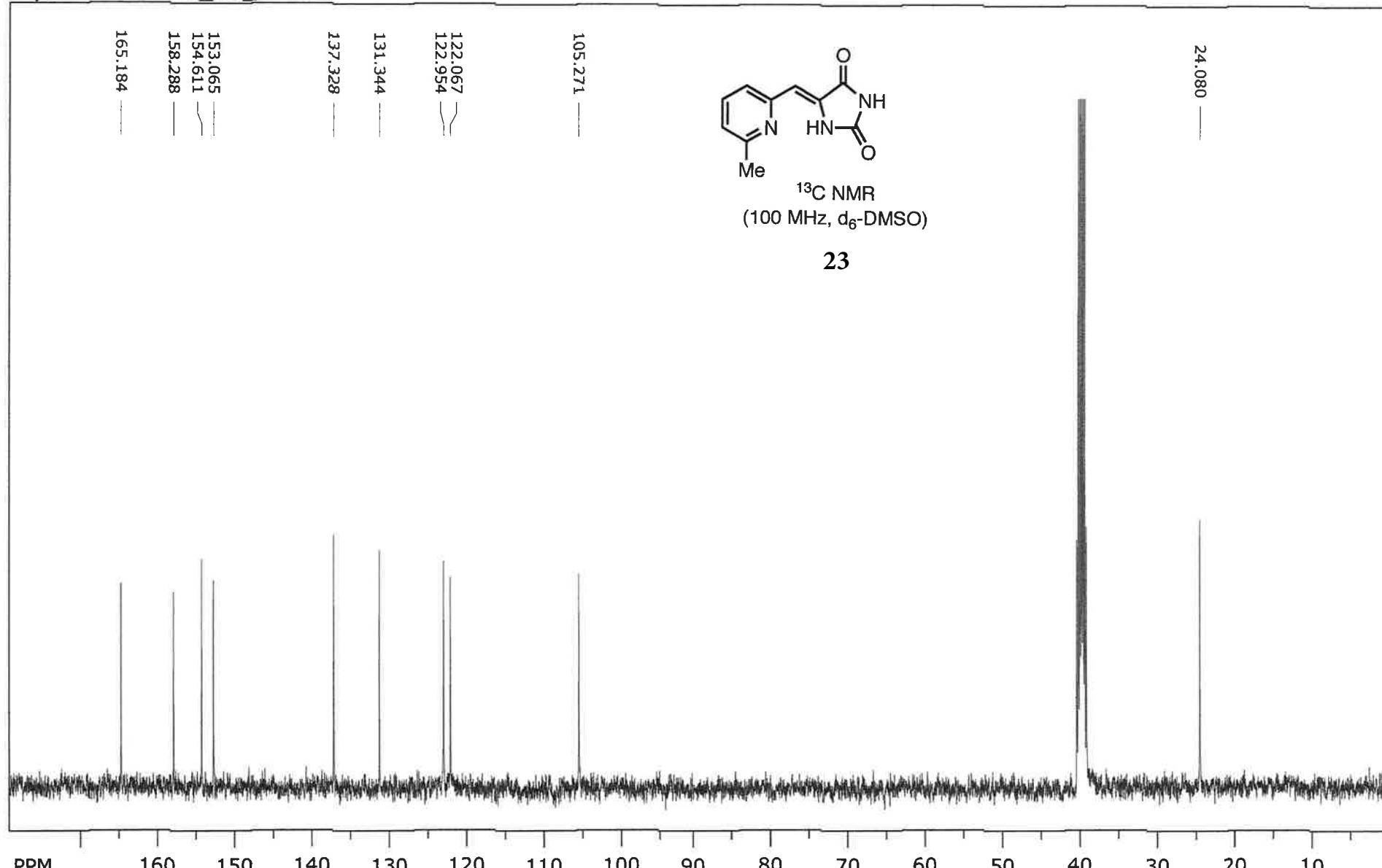
¹H NMR
(300 MHz, d₆-DMSO)

23



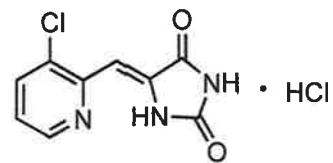
file: ...pectra\WD spectra\WD329_1H.fid\fid block# 1 expt: "s2pul"
transmitter freq.: 300.134434 MHz
time domain size: 19192 points
width: 4803.07 Hz = 16.0031 ppm = 0.250264 Hz/pt
number of scans: 8

freq. of 0 ppm: 300.132634 MHz
processed size: 32768 complex points
LB: 0.500 GF: 0.0000
Hz/cm: 108.016 ppm/cm: 0.35989



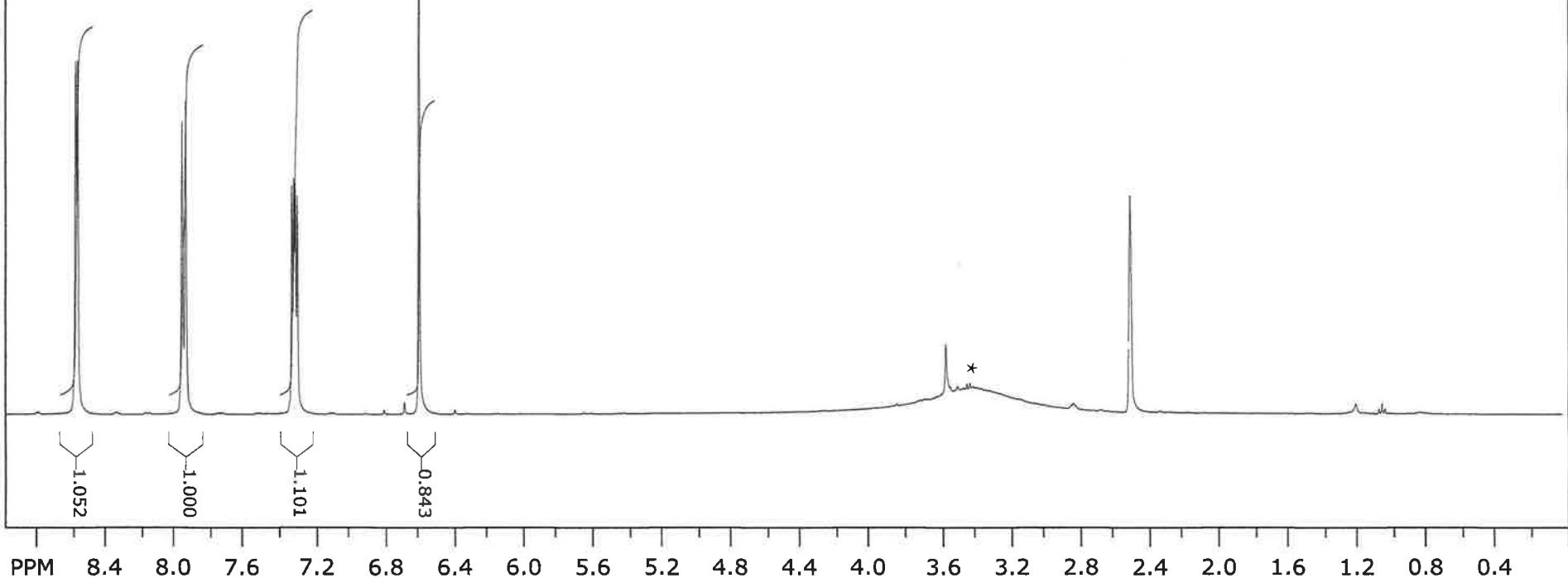
file: ...on\WD329_13C_DMSO_20240116.fid\fid block# 1 expt: "s2pul"
transmitter freq.: 100.523180 MHz
time domain size: 65536 points
width: 25000.00 Hz = 248.6989 ppm = 0.381470 Hz/pt
number of scans: 1088

freq. of 0 ppm: 100.512161 MHz
processed size: 65536 complex points
LB: 2.500 GF: 0.0000
Hz/cm: 723.333 ppm/cm: 7.19569



^1H NMR
(400 MHz, d_6 -DMSO)
*moisture in DMSO

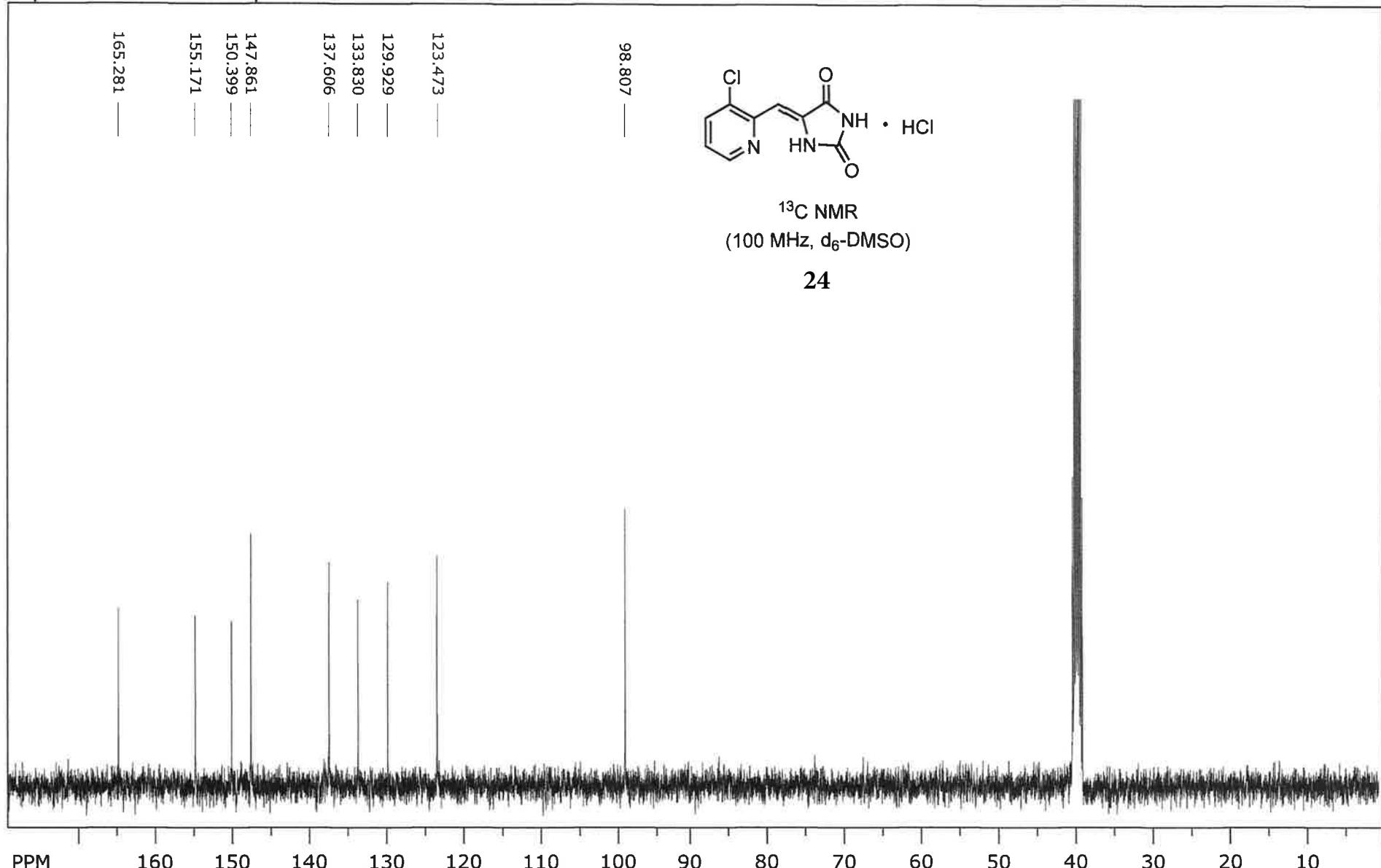
24



file: ...spectra\WD340-349\WD346-1H.fid\fid block# 1 expt: "s2pul"
transmitter freq.: 399.732139 MHz
time domain size: 32768 points
width: 6410.26 Hz = 16.0364 ppm = 0.195626 Hz/pt
number of scans: 8

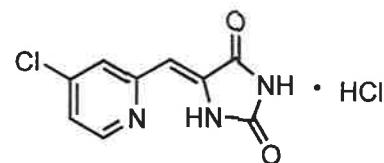
freq. of 0 ppm: 399.729734 MHz
processed size: 32768 complex points
LB: 0.510 GF: 0.0000
Hz/cm: 143.875 ppm/cm: 0.35993

SpinWorks 4: new experiment



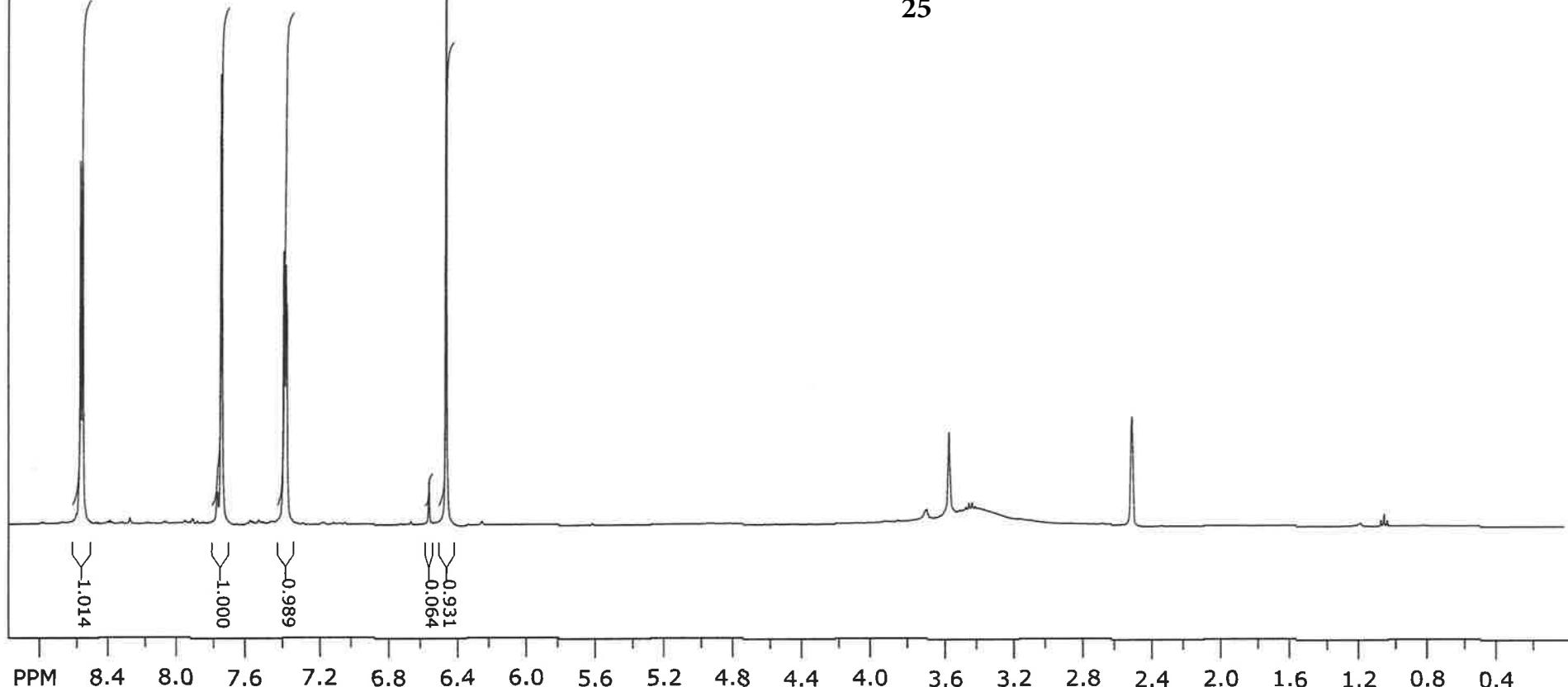
file: ...pectra\WD340-349\WD346-13C.fid\fid block# 1 expt: "s2pul"
transmitter freq.: 100.523180 MHz
time domain size: 65536 points
width: 25000.00 Hz = 248.6989 ppm = 0.381470 Hz/pt
number of scans: 512

freq. of 0 ppm: 100.512163 MHz
processed size: 65536 complex points
LB: 1.500 GF: 0.0000
Hz/cm: 723.333 ppm/cm: 7.19569



^1H NMR
(400 MHz, $\text{d}_6\text{-DMSO}$)

25

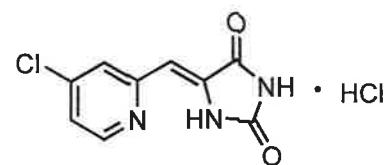


file: ...spectra\WD340-349\WD347-1H.fid fid block# 1 expt: "s2pul"
transmitter freq.: 399.732139 MHz
time domain size: 32768 points
width: 6410.26 Hz = 16.0364 ppm = 0.195626 Hz/pt
number of scans: 8

freq. of 0 ppm: 399.729734 MHz
processed size: 32768 complex points
LB: 0.610 GF: 0.0000
Hz/cm: 143.875 ppm/cm: 0.35993

SpinWorks 4: new experiment

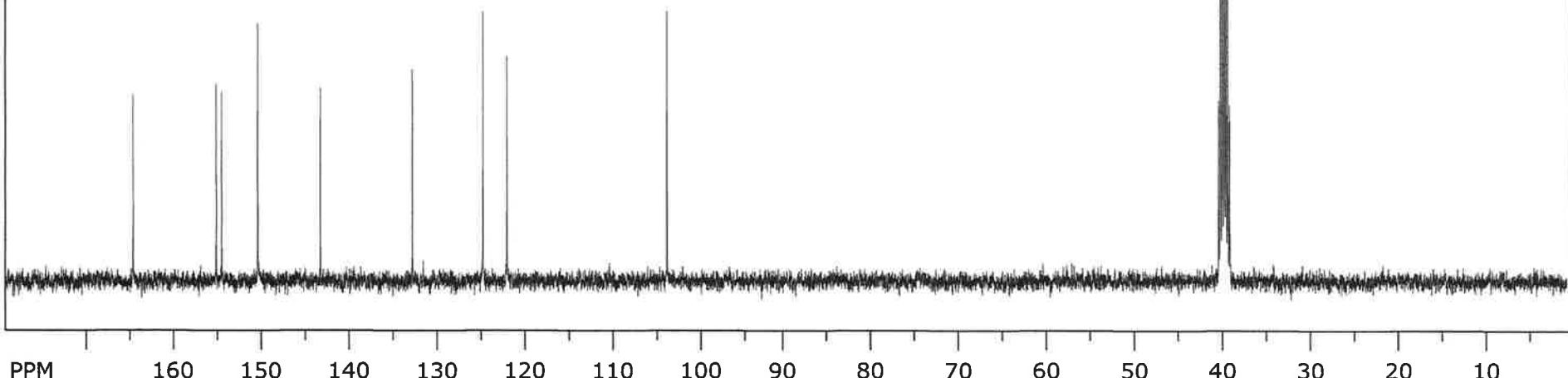
165.101 —
150.702 —
154.869 —
155.496 —
143.489 —
132.915 —
122.034 —
124.792 —
103.629 —



^{13}C NMR

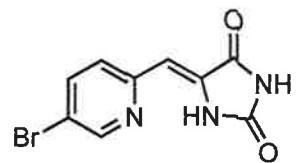
(100 MHz, $\text{d}_6\text{-DMSO}$)

25



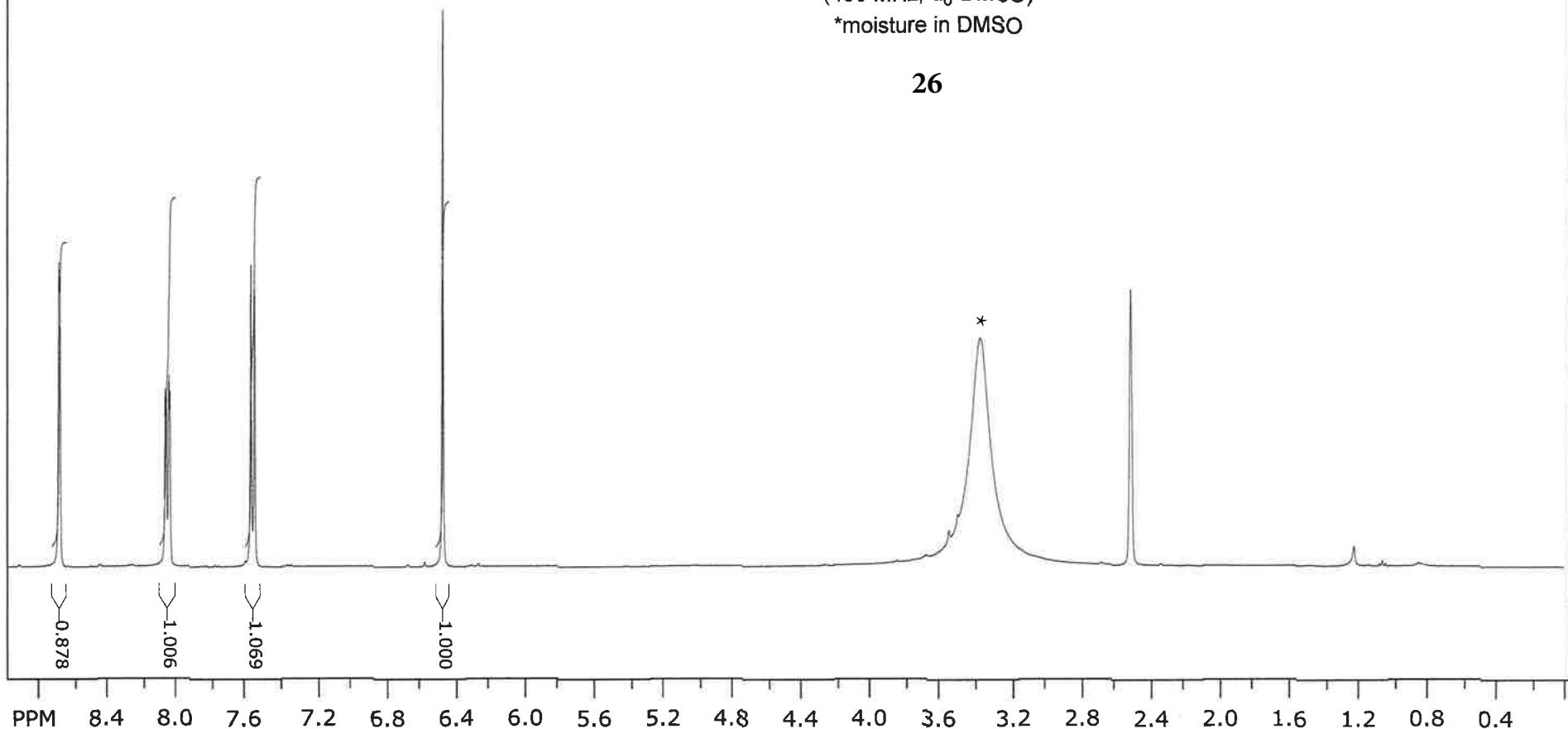
file: ...pectra\WD340-349\WD347-13C.fid fid block# 1 expt: "s2pul"
transmitter freq.: 100.523180 MHz
time domain size: 65536 points
width: 25000.00 Hz = 248.6989 ppm = 0.381470 Hz/pt
number of scans: 512

freq. of 0 ppm: 100.512162 MHz
processed size: 65536 complex points
LB: 1.500 GF: 0.0000
Hz/cm: 723.333 ppm/cm: 7.19569



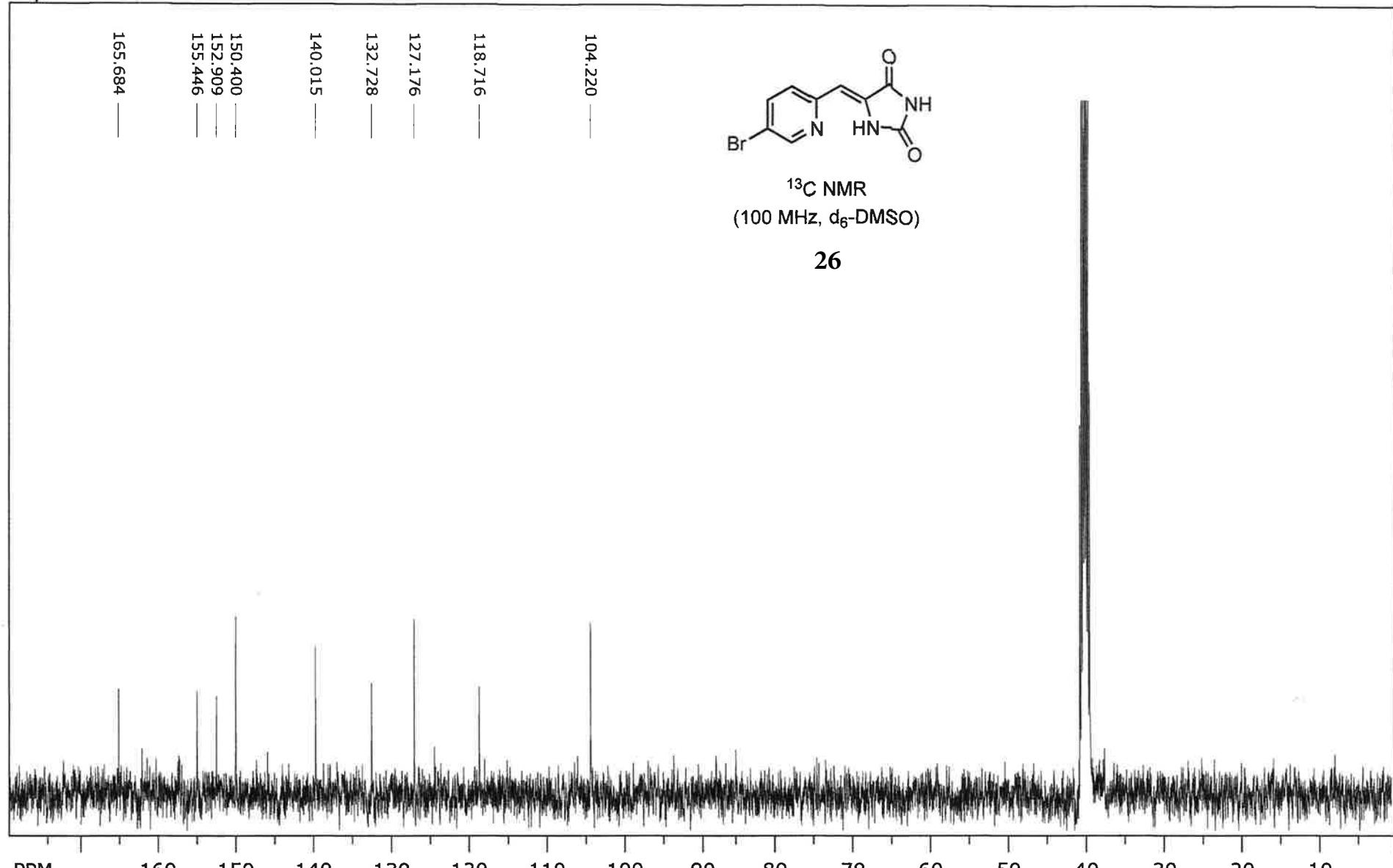
¹H NMR
(400 MHz, d₆-DMSO)
*moisture in DMSO

26



file: ...spectra\WD330-339\WD333-H1.fid fid block# 1 expt: "s2pul"
transmitter freq.: 399.732139 MHz
time domain size: 32768 points
width: 6410.26 Hz = 16.0364 ppm = 0.195626 Hz/pt
number of scans: 8

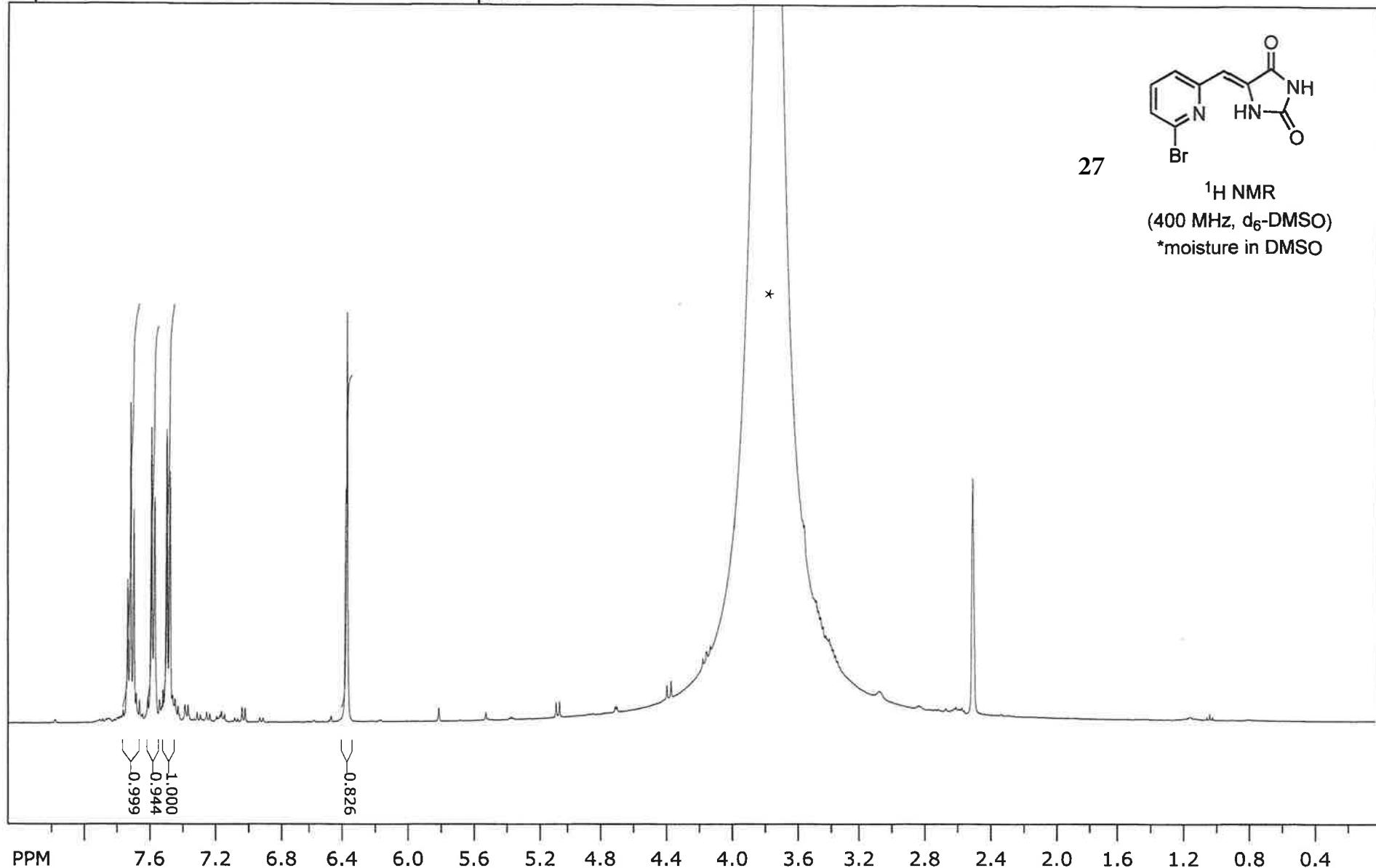
freq. of 0 ppm: 399.729733 MHz
processed size: 32768 complex points
LB: 0.610 GF: 0.0000
Hz/cm: 144.017 ppm/cm: 0.36028



file: ...pectra\WD330-339\WD333-C13.fid fid block# 1 expt: "s2pul"
transmitter freq.: 100.523180 MHz
time domain size: 65536 points
width: 25000.00 Hz = 248.6989 ppm = 0.381470 Hz/pt
number of scans: 256

freq. of 0 ppm: 100.512123 MHz
processed size: 65536 complex points
LB: 2.500 GF: 0.0000
Hz/cm: 723.889 ppm/cm: 7.20121

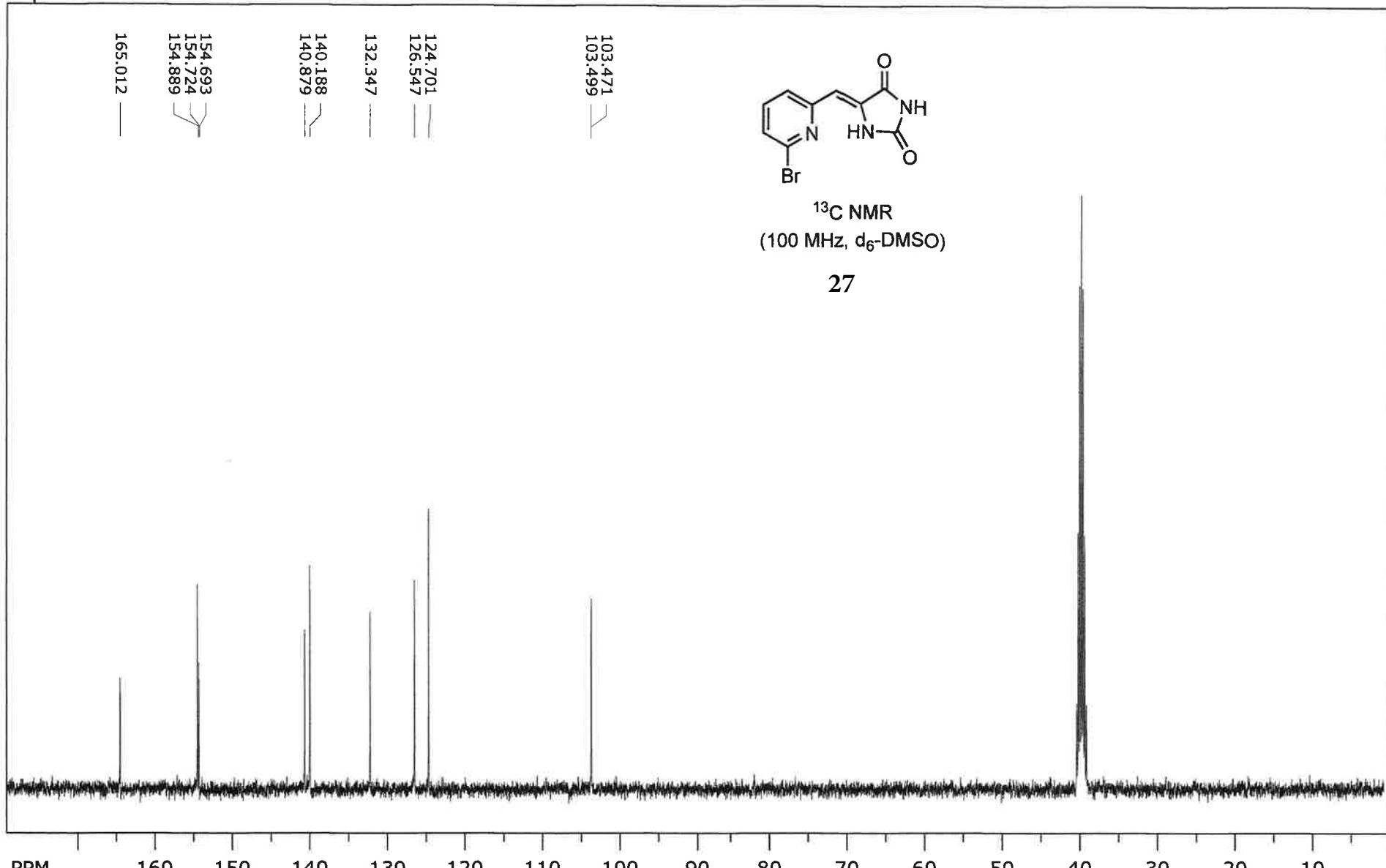
SpinWorks 4: STANDARD 1H OBSERVE - profile



file: ...pectra\WD spectra\WD326_1H.fid\fid block# 1 expt: "s2pul"
transmitter freq.: 399.732139 MHz
time domain size: 32768 points
width: 6410.26 Hz = 16.0364 ppm = 0.195626 Hz/pt
number of scans: 8

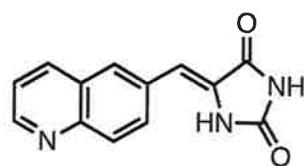
freq. of 0 ppm: 399.729734 MHz
processed size: 32768 complex points
LB: 0.506 GF: 0.0000
Hz/cm: 135.897 ppm/cm: 0.33997

SpinWorks 4: wefarv



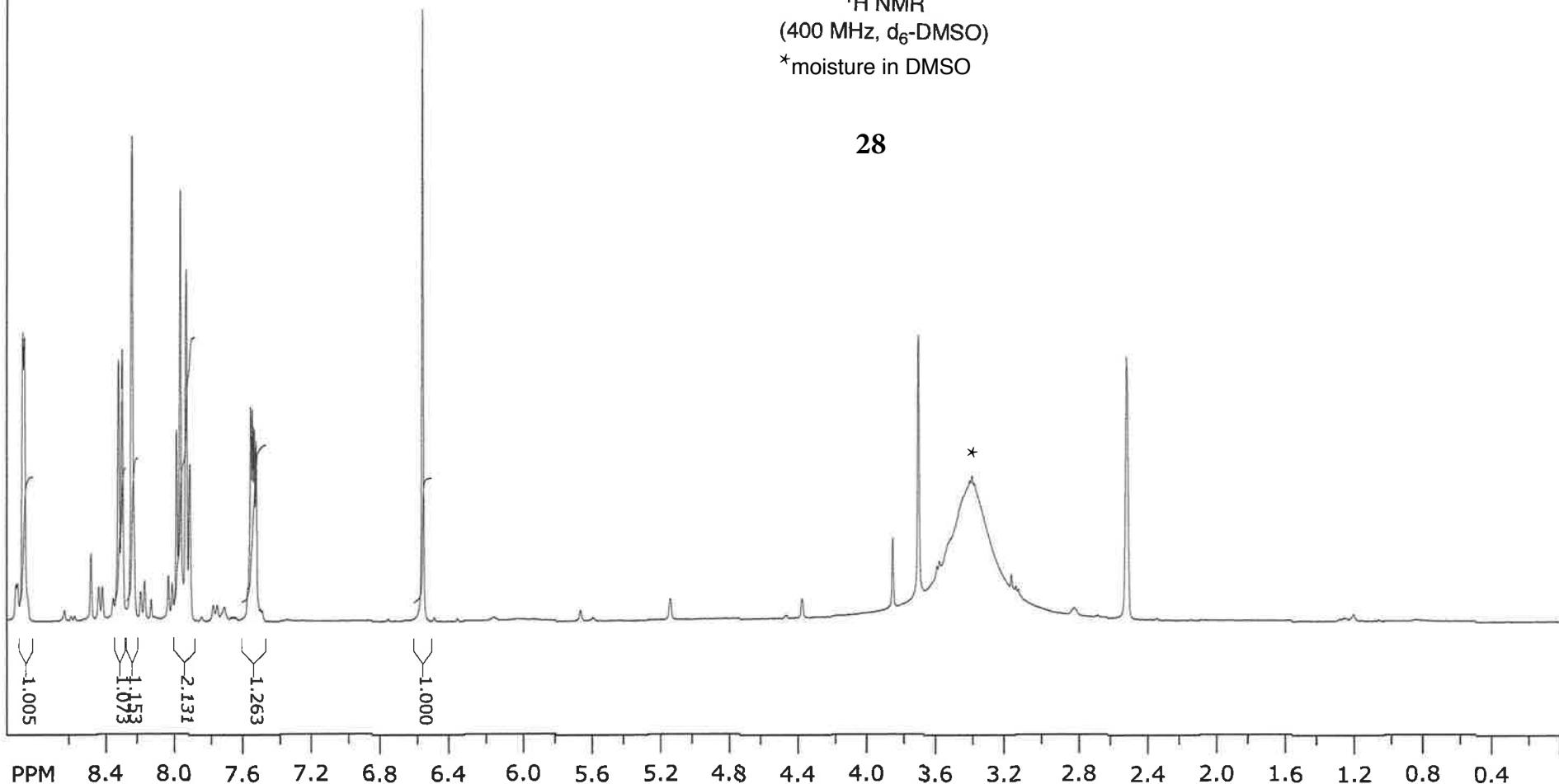
file: ...pectra\WD320-329\WD327-C13.fid\fid block# 1 expt: "s2pul"
transmitter freq.: 100.523180 MHz
time domain size: 65536 points
width: 25000.00 Hz = 248.6989 ppm = 0.381470 Hz/pt
number of scans: 256

freq. of 0 ppm: 100.512160 MHz
processed size: 65536 complex points
LB: 1.500 GF: 0.0000
Hz/cm: 723.333 ppm/cm: 7.19569



¹H NMR
(400 MHz, d₆-DMSO)
*moisture in DMSO

28



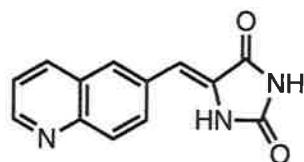
file: ...pectra\KG01-10\KG10-proton.fid\fid block# 1 expt: "s2pul"
transmitter freq.: 399.732139 MHz
time domain size: 32768 points
width: 6410.26 Hz = 16.0364 ppm = 0.195626 Hz/pt
number of scans: 8

freq. of 0 ppm: 399.729733 MHz
processed size: 32768 complex points
LB: 0.500 GF: 0.0000
Hz/cm: 143.732 ppm/cm: 0.35957

SpinWorks 4: JC_SS_1

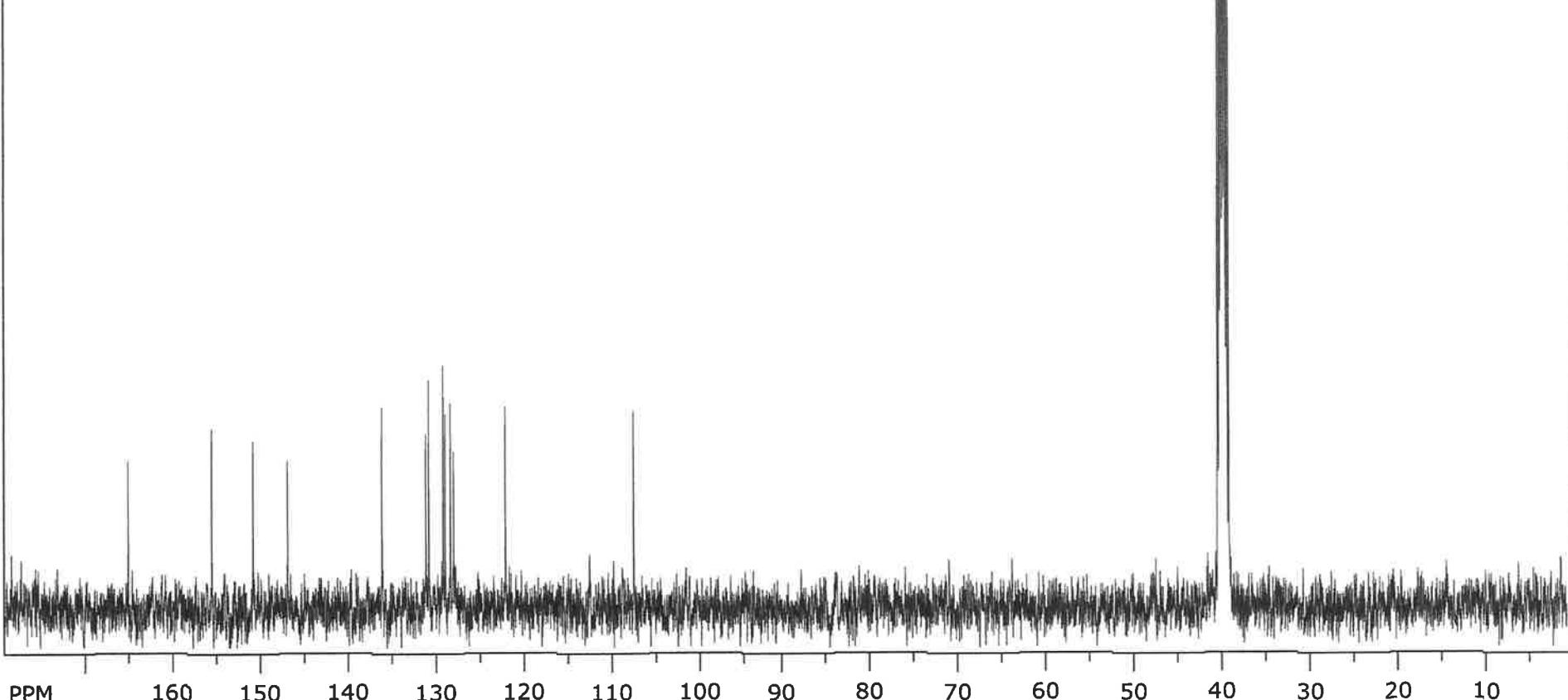
165.527 —
147.138 —
151.114 —
155.874 —
122.075
128.019
128.390
128.987
129.216
130.878
131.223
136.264

107.316 —



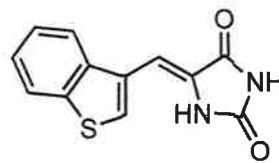
¹³C NMR
(100 MHz, d₆-DMSO)

28



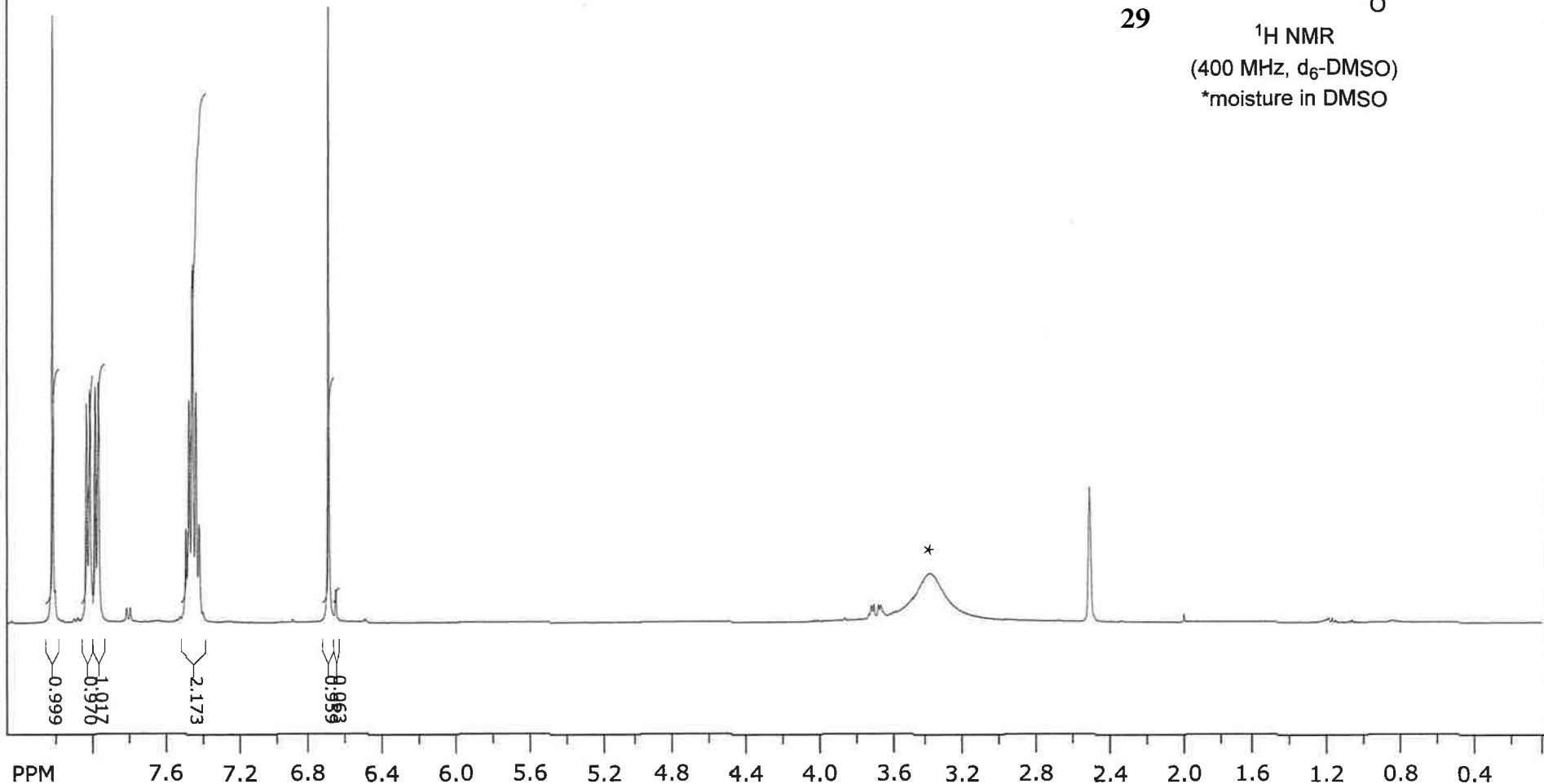
file: ...son\KG10_13C_DMSO_20240118.fid\fid block# 1 expt: "s2pul"
transmitter freq.: 100.523180 MHz
time domain size: 65536 points
width: 25000.00 Hz = 248.6989 ppm = 0.381470 Hz/pt
number of scans: 2048

freq. of 0 ppm: 100.512162 MHz
processed size: 65536 complex points
LB: 2.500 GF: 0.0000
Hz/cm: 723.333 ppm/cm: 7.19569



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¹H NMR
(400 MHz, d₆-DMSO)
*moisture in DMSO



file: ...pectra\KG11-20\KG18-proton.fid\fid block# 1 expt: "s2pu"
transmitter freq.: 399.732139 MHz
time domain size: 32768 points
width: 6410.26 Hz = 16.0364 ppm = 0.195626 Hz/pt
number of scans: 8

freq. of 0 ppm: 399.729735 MHz
processed size: 32768 complex points
LB: 0.500 GF: 0.0000
Hz/cm: 135.897 ppm/cm: 0.33997

SpinWorks 4: EAW1050_carbon

165.488 —
155.746 —
138.045
138.718 —
121.561
123.054
124.964
125.168
127.019
127.983
128.984

98.876 —



^{13}C NMR
(100 MHz, $\text{d}_6\text{-DMSO}$)

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PPM 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

file: ...ldsonw\Desktop\KG18-carbon.fid\fid block# 1 expt: "s2pul"
transmitter freq.: 100.523180 MHz
time domain size: 65536 points
width: 25000.00 Hz = 248.6989 ppm = 0.381470 Hz/pt
number of scans: 256

freq. of 0 ppm: 100.512160 MHz
processed size: 65536 complex points
LB: 1.500 GF: 0.0000
Hz/cm: 723.333 ppm/cm: 7.19569