Synthesis and evaluation of indole-based chalcones as inducers of methuosis, a novel type of non-apoptotic cell death

Michael W. Robinson[†], Jean H. Overmeyer[†], Ashley M. Young[†], Paul W. Erhardt[‡*], William A. Maltese[†*]

[†]Department of Biochemistry and Cancer Biology, University of Toledo College of Medicine, 3000 Arlington Ave., Toledo, OH 43614

‡Center for Drug Design and Development, Department of Medicinal and Biological Chemistry, University of Toledo College of Pharmacy, 2801 W. Bancroft Ave., Toledo, OH 43606

AUTHOR INFORMATION

*Corresponding Authors: For William A. Maltese: Phone, 419-383-4161; Fax, 419-383-6228; e-mail, william.maltese@utoledo.edu. For Paul W. Erhardt: Phone, 419-530-2167; Fax, 419-530-1994; e-mail, paul.erhardt@utoledo.edu.

SUPPORTING INFORMATION CONTENTS

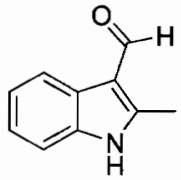
S2 – S113: NMR and IR spectra for all compounds

STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
File: 100204b
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8999.9 Hz
16 repetitions
OBSERVE H1, 599.8751442 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



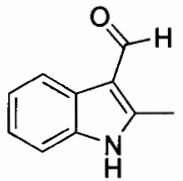
2a

13C OBSERVE

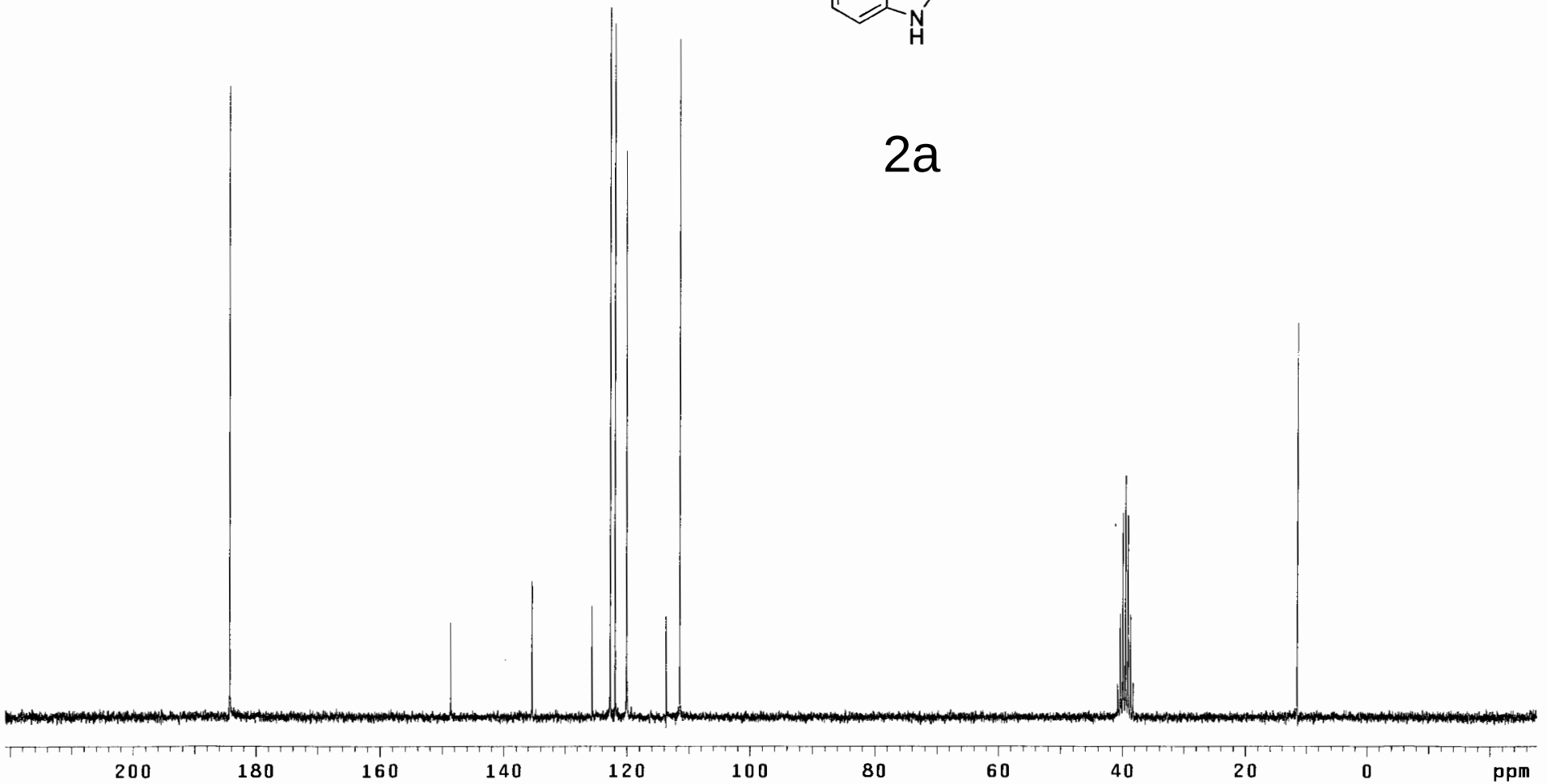
Jun 10 2011

Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
GEMINI-200BB "gem2000"

Relax. delay 0.800 sec
Pulse 48.2 degrees
Acq. time 0.600 sec
Width 12500.0 Hz
4832 repetitions
OBSERVE C13, 50.2840312 MHz
DECOUPLE H1, 199.9767234 MHz
Power 34 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.400 sec
center at 0.050 sec
FT size 131072
Total time 41 hr, 44 min, 49 sec



2a

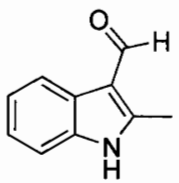


STANDARD 1H OBSERVE

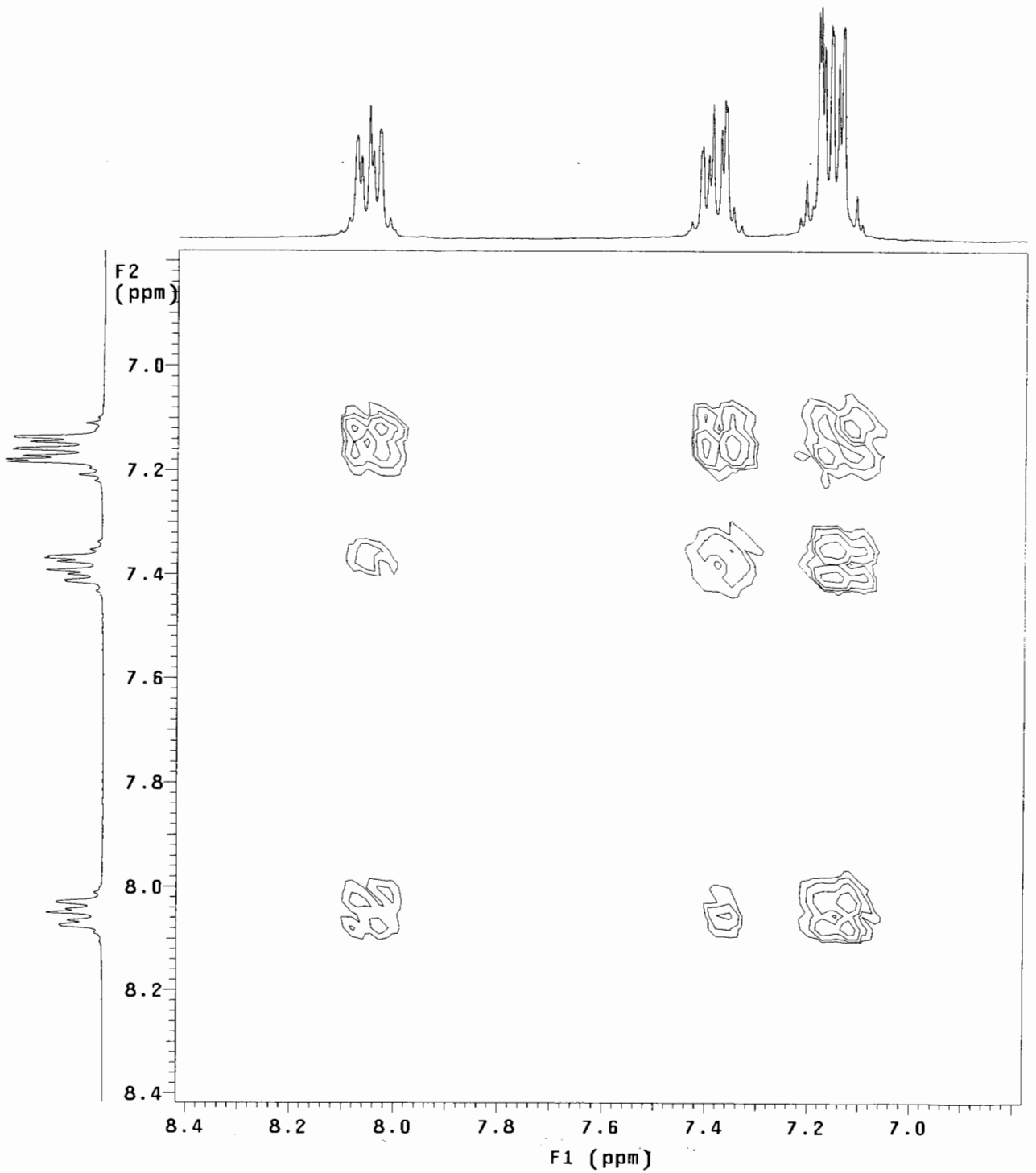
Pulse Sequence: relayh

Solvent: DMSO
Ambient temperature
GEMINI-200BB "gem2000"

Relax. delay 1.000 sec
COSY 90-90
Acq. time 0.196 sec
Width 327.2 Hz
2D Width 327.2 Hz
4 repetitions
64 increments
OBSERVE H1, 199.9760204 MHz
DATA PROCESSING
Sine bell 0.098 sec
F1 DATA PROCESSING
Sine bell 0.098 sec
FT size 128 x 128
Total time 5 min, 59 sec



2a

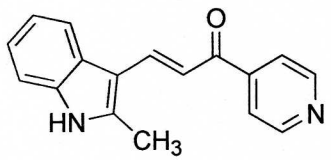


STANDARD PROTON PARAMETERS

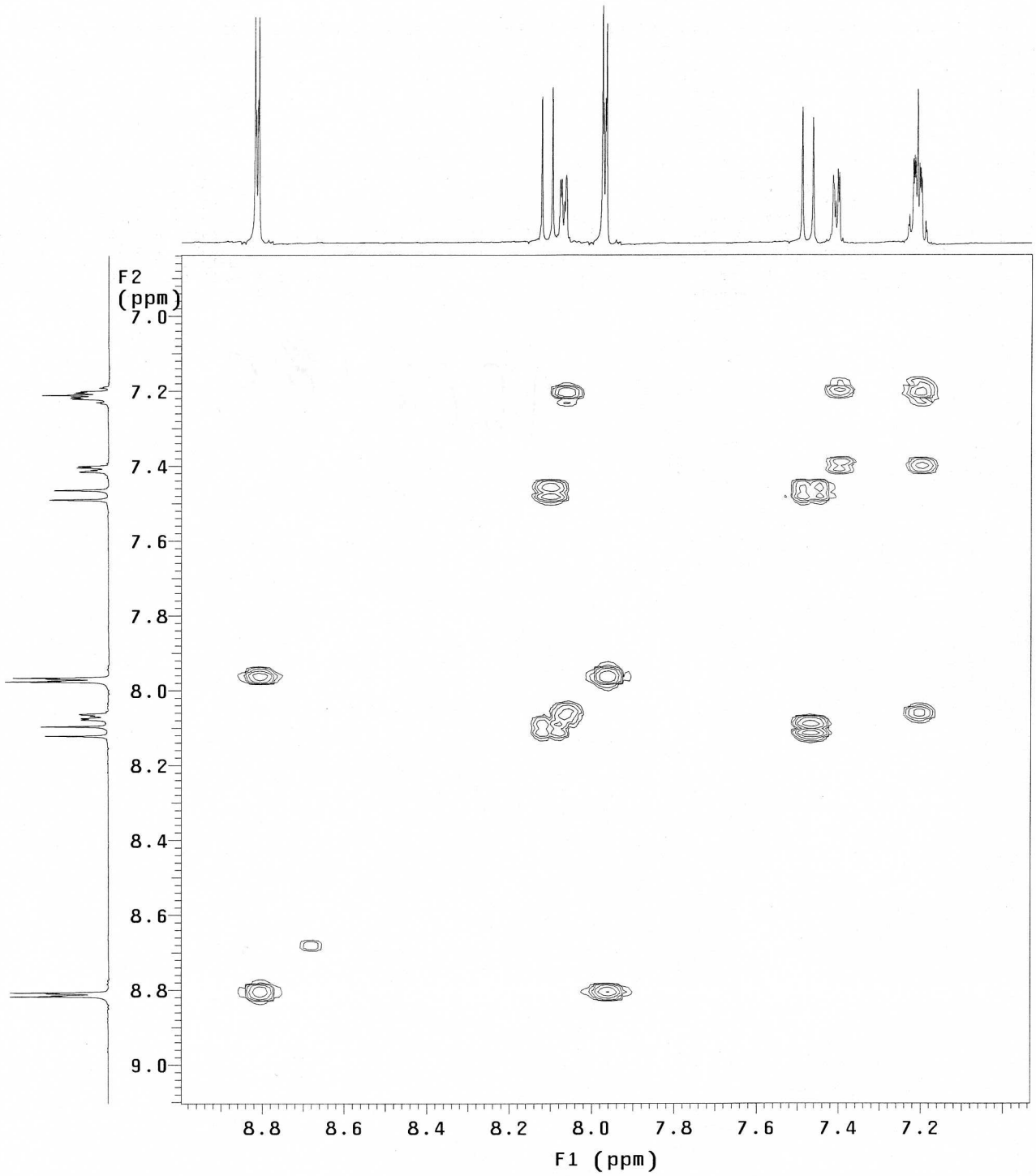
Pulse Sequence: gcosy

Solvent: DMSO
Ambient temperature
File: 100218c
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Acq. time 0.140 sec
Width 3654.3 Hz
2D Width 3654.3 Hz
Single scan
256 increments
OBSERVE H1, 599.8751457 MHz
DATA PROCESSING
Sine bell 0.070 sec
F1 DATA PROCESSING
Sine bell 0.035 sec
FT size 1024 x 1024
Total time 5 min, 6 sec



2

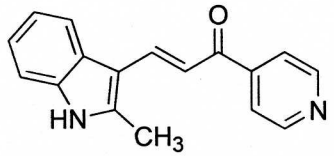


STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
File: 100218b
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8999.9 Hz
16 repetitions
OBSERVE H1, 599.8751457 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



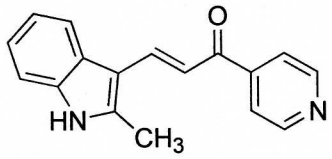
2

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
User: 1-14-87
File: 100218a
INOVA-600 "inova-wkst"

Pulse 51.7 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
6208 repetitions
OBSERVE C13, 150.8387920 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 2 hr, 48 min, 9 sec



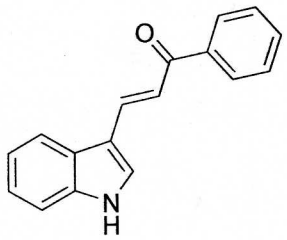
2

STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
File: 091006b
GEMINI-200BB "gem2000"

Relax. delay 1.000 sec
Pulse 30.9 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.8751449 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 10 sec



9

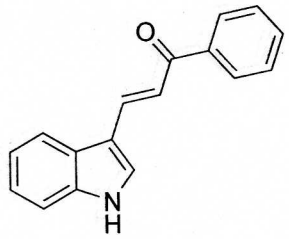
Oct 6 2009

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
User: 1-14-87
File: 091007a
GEMINI-200BB "gem2000"

Pulse 54.9 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
46384 repetitions
OBSERVE C13, 150.8387929 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 36 hr, 47 min, 1 sec

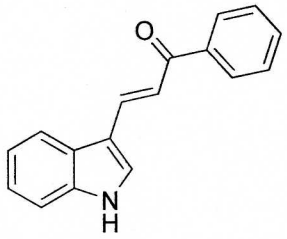


9

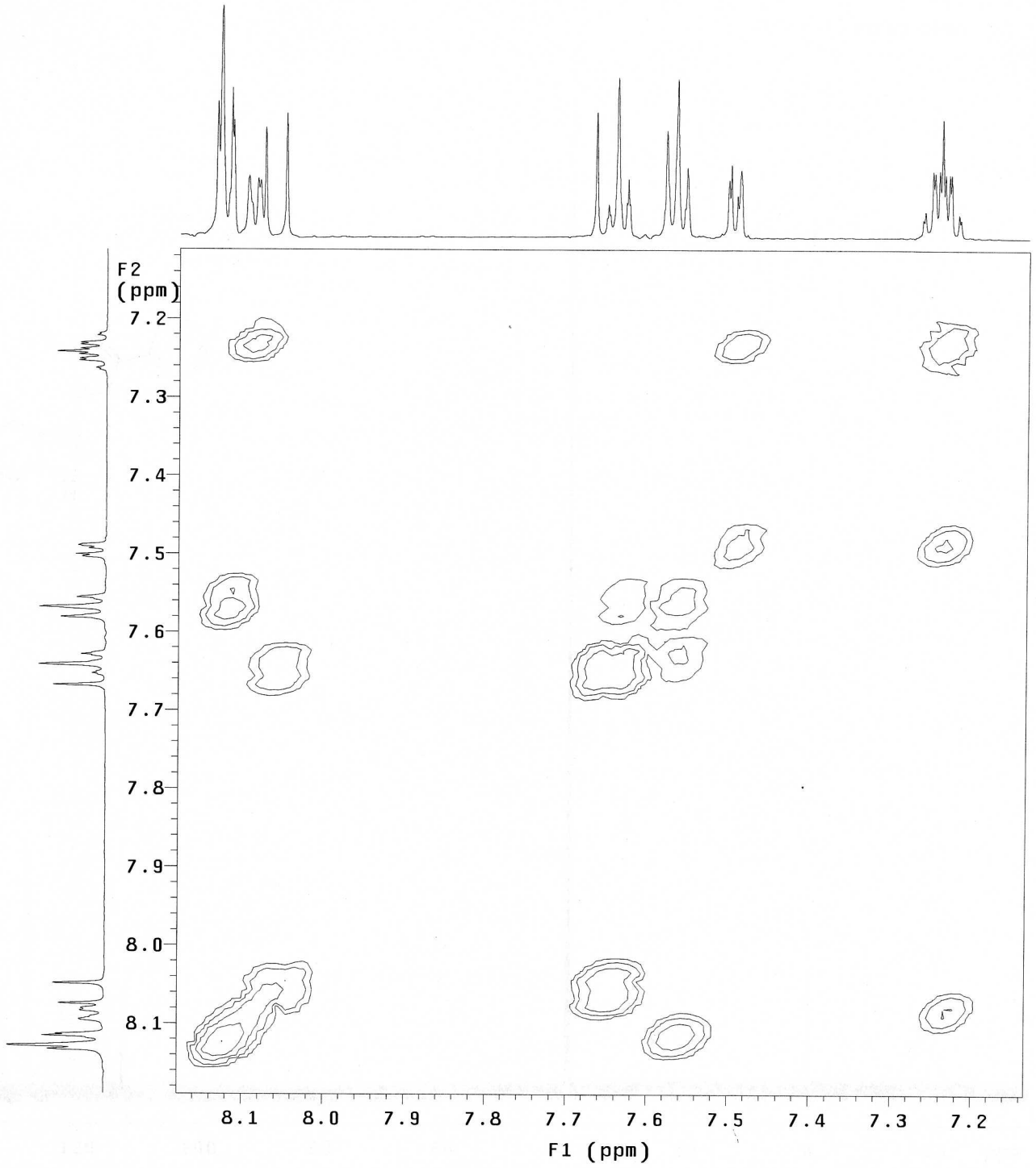
STANDARD PROTON PARAMETERS

Pulse Sequence: gcosy
Solvent: DMSO
Ambient temperature
File: 091006c
GEMINI-200BB "gem2000"

Relax. delay 1.000 sec
Acq. time 0.151 sec
Width 1700.2 Hz
2D Width 1700.2 Hz
Single scan
128 increments
OBSERVE H1, 599.8751449 MHz
DATA PROCESSING
Sine bell 0.075 sec
F1 DATA PROCESSING
Sine bell 0.038 sec
FT size 512 x 512
Total time 3 min, 14 sec



9

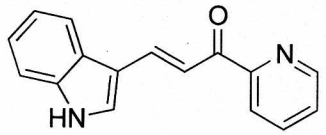


STANDARD PROTON PARAMETERS

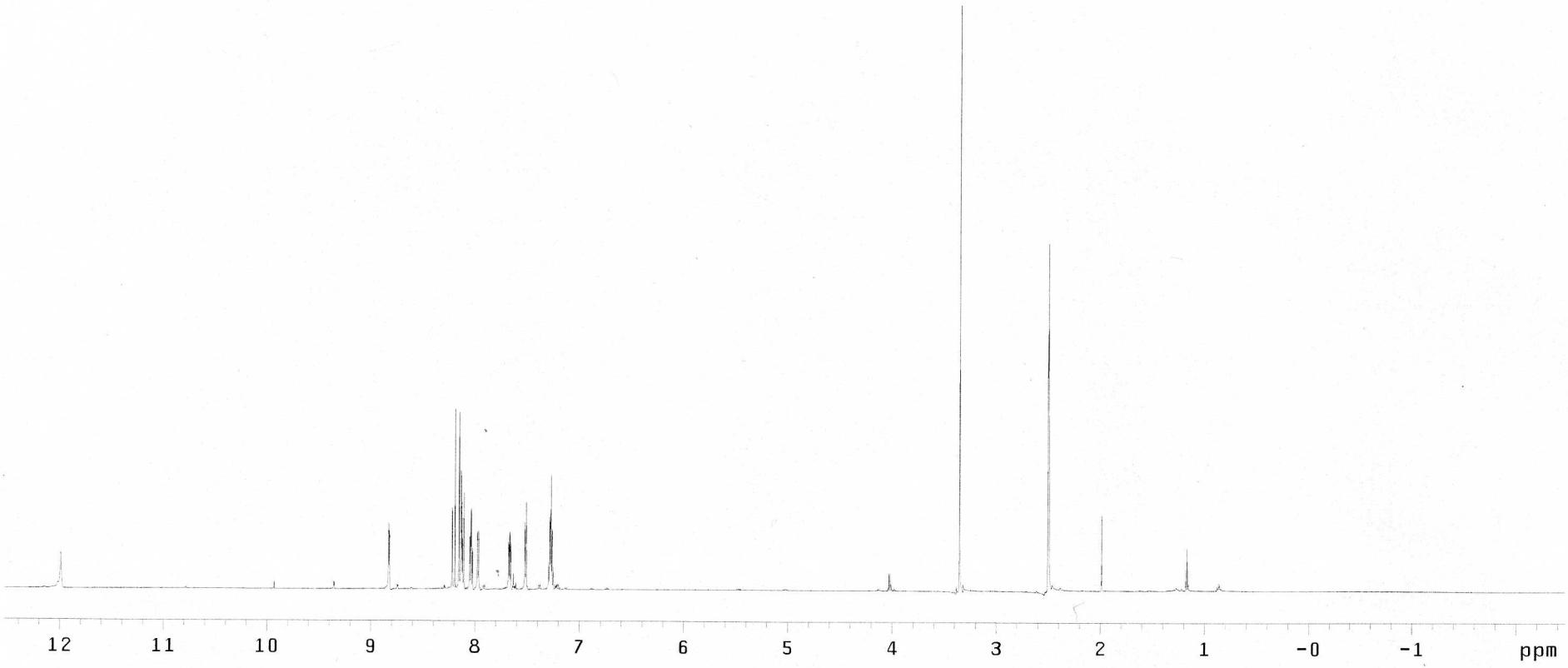
Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
File: 100729d
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8999.9 Hz
16 repetitions
OBSERVE H1, 599.8751449 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



10

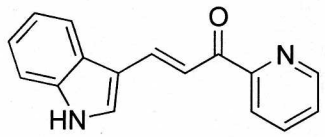


STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
User: 1-14-87
File: 100729f
INOVA-600 "inova600"

Pulse 46.4 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
37600 repetitions
OBSERVE C13, 150.8387929 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 28 hr, 1 min, 31 sec



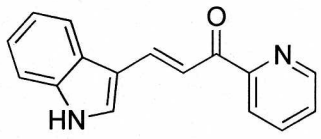
10

STANDARD PROTON PARAMETERS

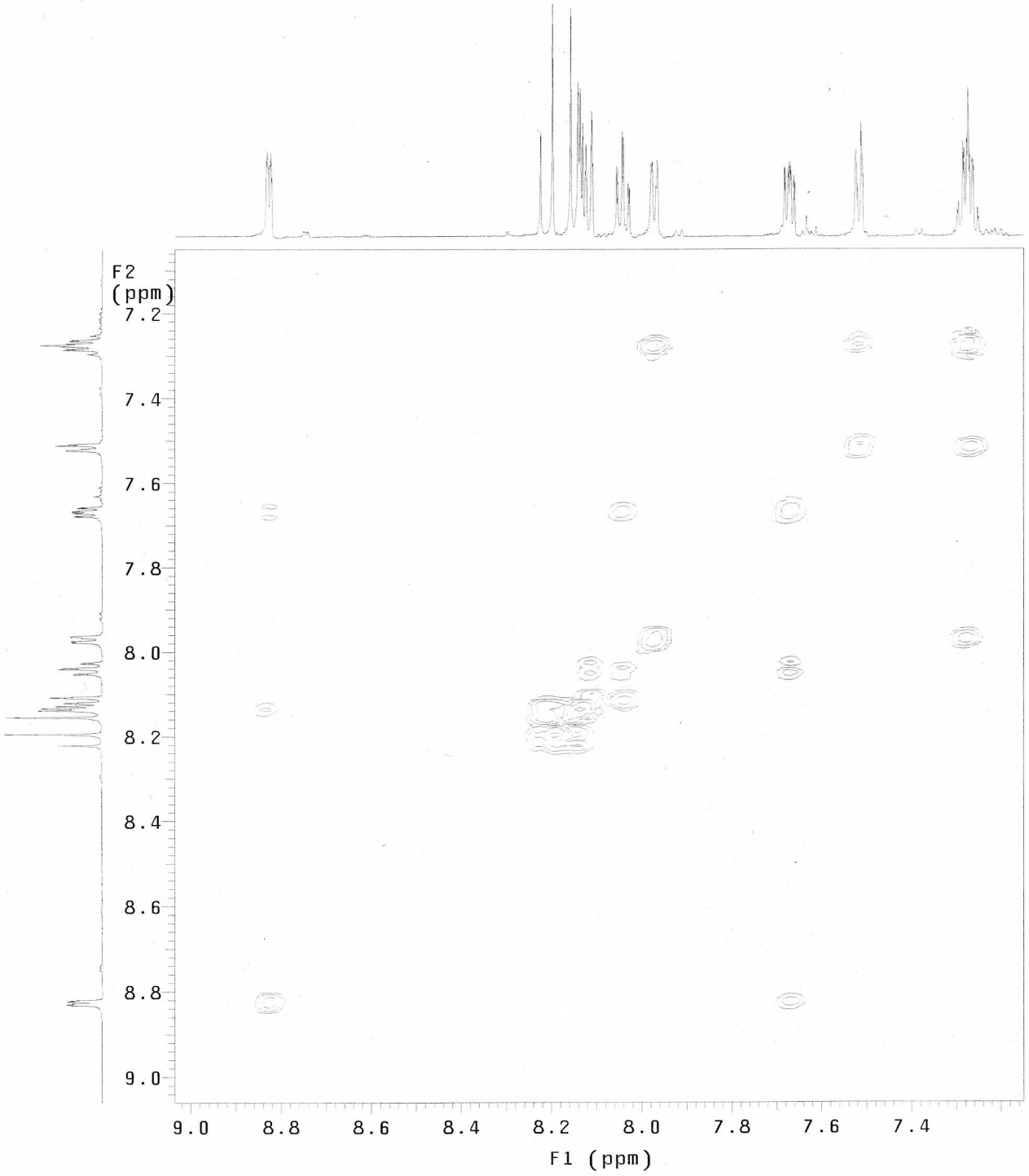
Pulse Sequence: gcosy

Solvent: DMSO
Ambient temperature
File: 100729e
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.135 sec
Width 1892.2 Hz
2D Width 1892.2 Hz
Single scan
128 increments
OBSERVE H1, 599.8751449 MHz
DATA PROCESSING
Sine bell 0.068 sec
F1 DATA PROCESSING
Sine bell 0.034 sec
FT size 512 x 512
Total time 2 min, 33 sec



10

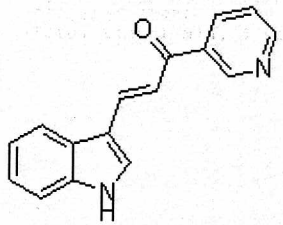


STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
File: 101028a
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8999.9 Hz
16 repetitions
OBSERVE H1, 599.8751455 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec

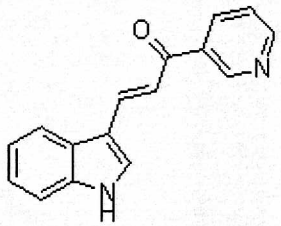


11

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
User: 1-14-87
File: 101028c
INOVA-600 "inova-wkst"

Pulse 46.4 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
7632 repetitions
OBSERVE C13, 150.8387919 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 28 hr, 1 min, 31 sec



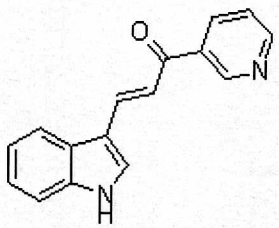
11

STANDARD PROTON PARAMETERS

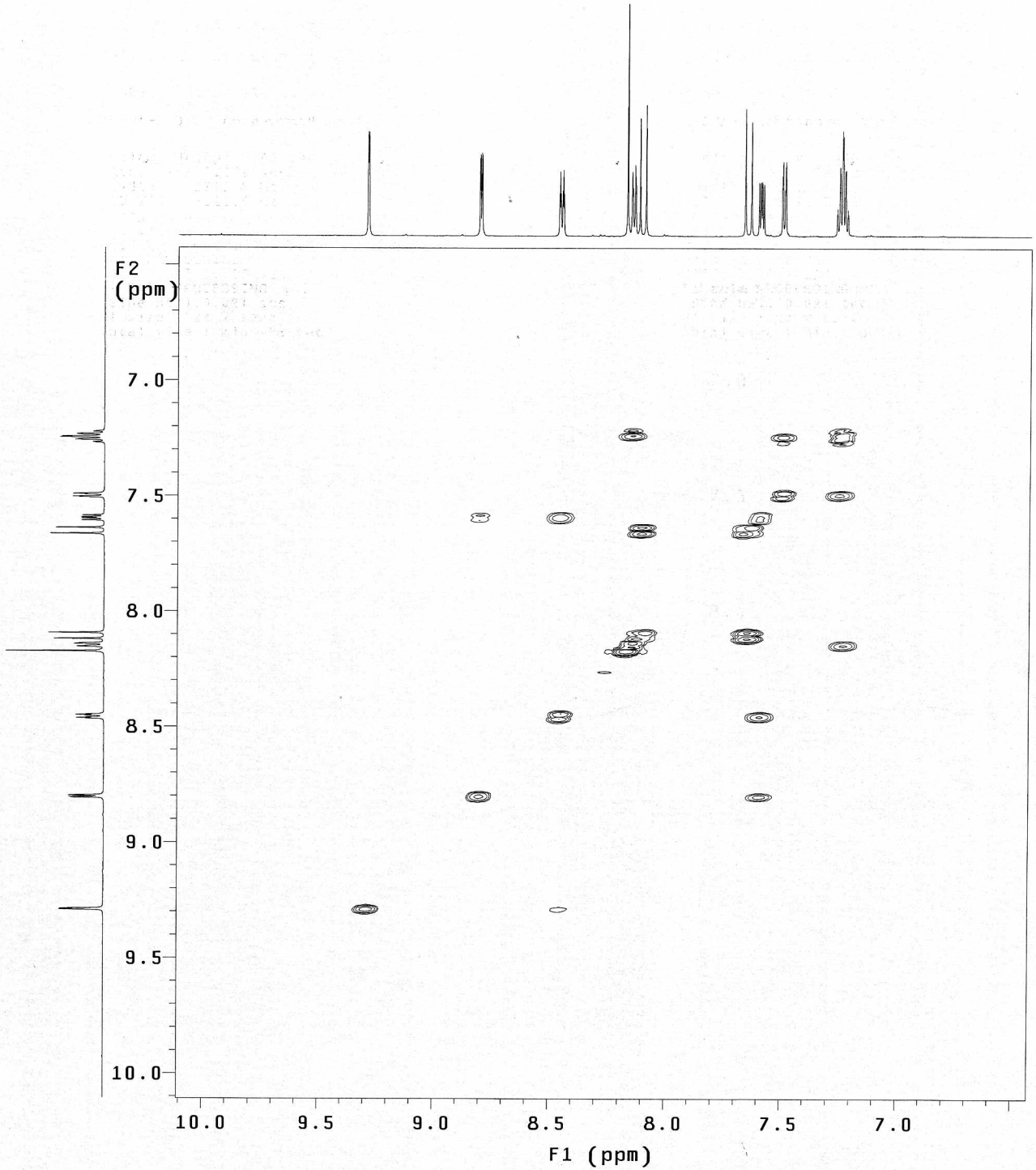
Pulse Sequence: gcosy

Solvent: DMSO
Ambient temperature
File: 101028b
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Acq. time 0.232 sec
Width 2211.5 Hz
2D Width 2211.5 Hz
Single scan
92 increments
OBSERVE H1, 599.8751455 MHz
DATA PROCESSING
Sine bell 0.116 sec
F1 DATA PROCESSING
Sine bell 0.021 sec
FT size 1024 x 1024
Total time 1 min, 58 sec



11



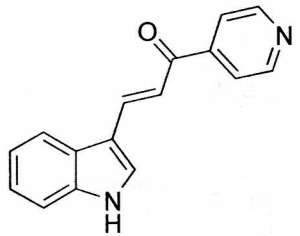
STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
File: 090930d
GEMINI-200BB "gem2000"

Relax. delay 1.000 sec
Pulse 30.9 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.8751452 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 10 sec

Sep 30 2009



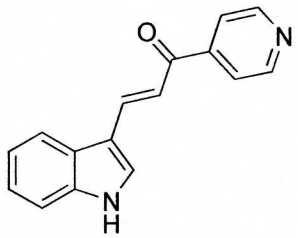
12

STANDARD CARBON PARAMETERS

Oct 3 2009

Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
User: 1-14-87
File: 091003a
GEMINI-200BB "gem2000"

Pulse 54.9 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
7264 repetitions
OBSERVE C13, 150.8387920 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 36 hr, 47 min, 1 sec



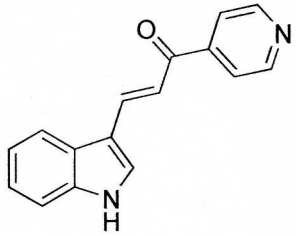
12

STANDARD PROTON PARAMETERS

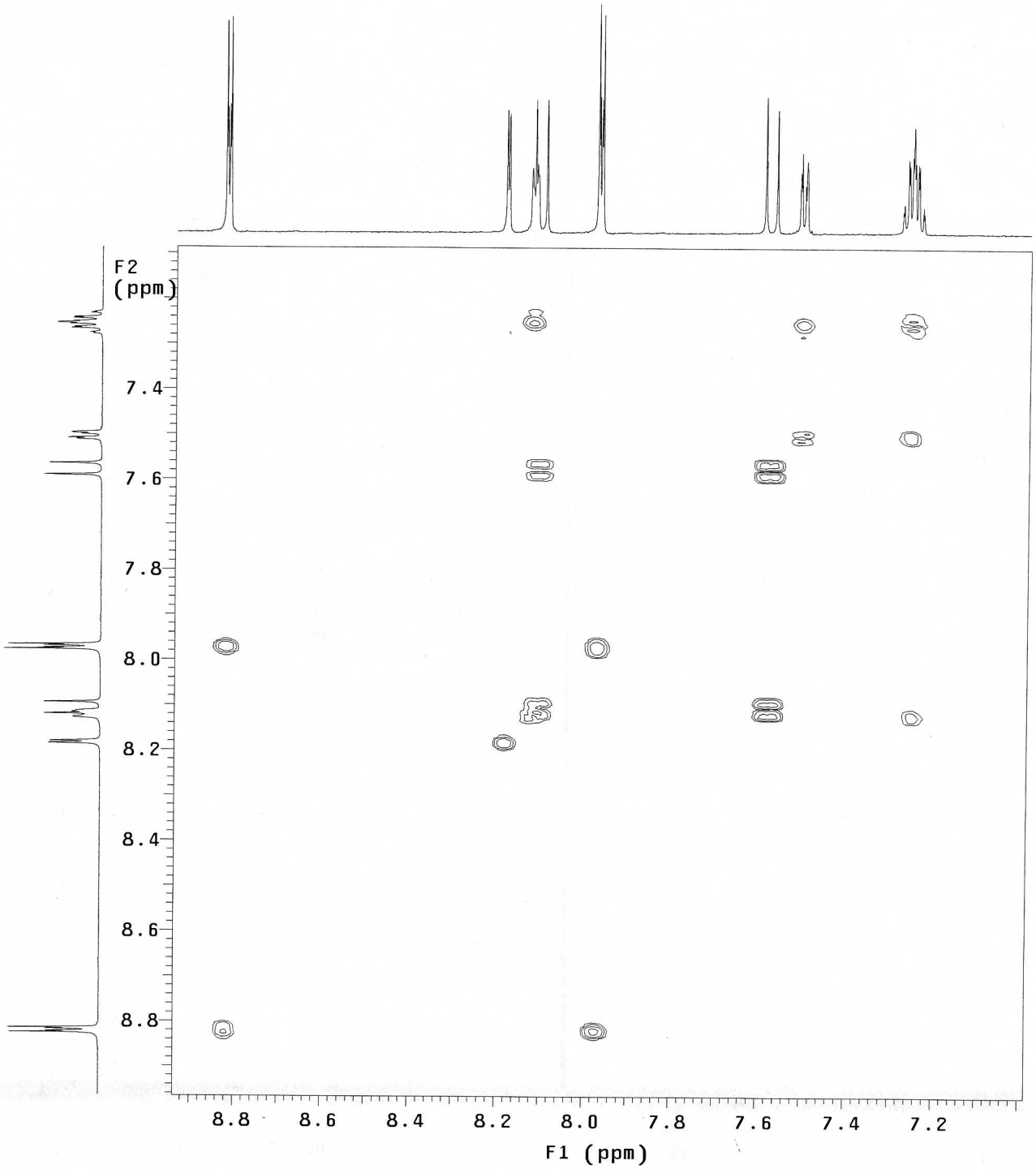
Pulse Sequence: gcosy

Solvent: DMSO
Ambient temperature
File: 090930c
GEMINI-200BB "gem2000"

Relax. delay 1.000 sec
Acq. time 0.180 sec
Width 5676.6 Hz
2D Width 5676.6 Hz
Single scan
512 increments
OBSERVE H1, 599.8751453 MHz
DATA PROCESSING
Sine bell 0.090 sec
F1 DATA PROCESSING
Sine bell 0.045 sec
FT size 2048 x 2048
Total time 13 min, 54 sec



12



STANDARD PROTON PARAMETERS

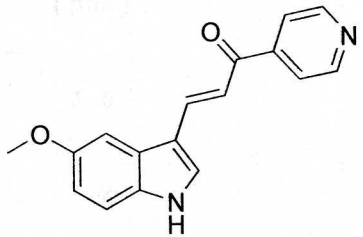
Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
File: 091004a
GEMINI-200BB "gem2000"

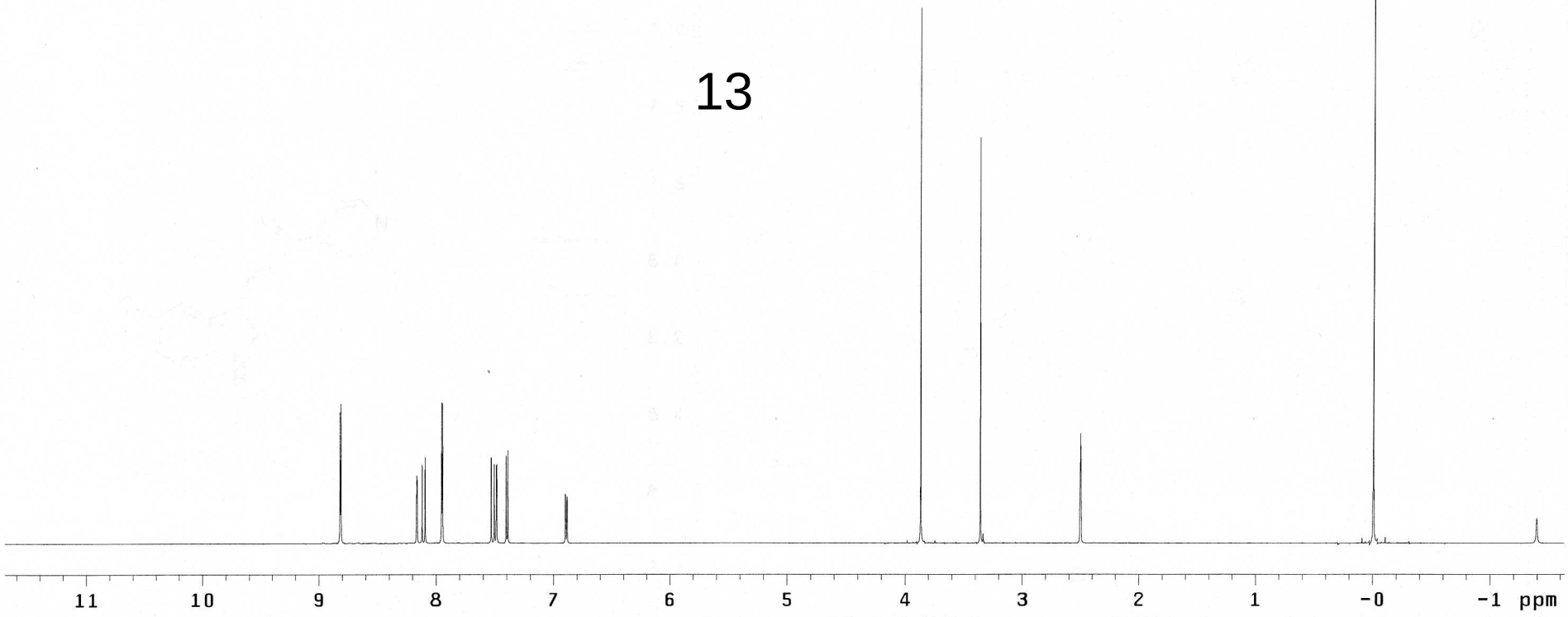
Relax. delay 1.000 sec
Pulse 30.9 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.8751449 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 10 sec

Oct 4 2009

-0.008



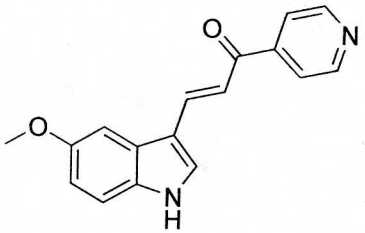
13



STANDARD CARBON PARAMETERS
S21
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
User: 1-14-87
File: 091004c
GEMINI-200BB "gem2000"

Pulse 54.9 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
6064 repetitions
OBSERVE C13, 150.8387908 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 36 hr, 47 min, 1 sec

Oct 4 2009

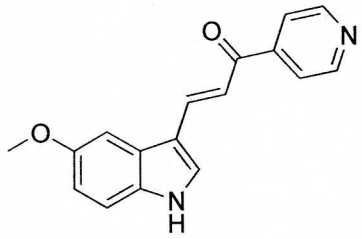


13

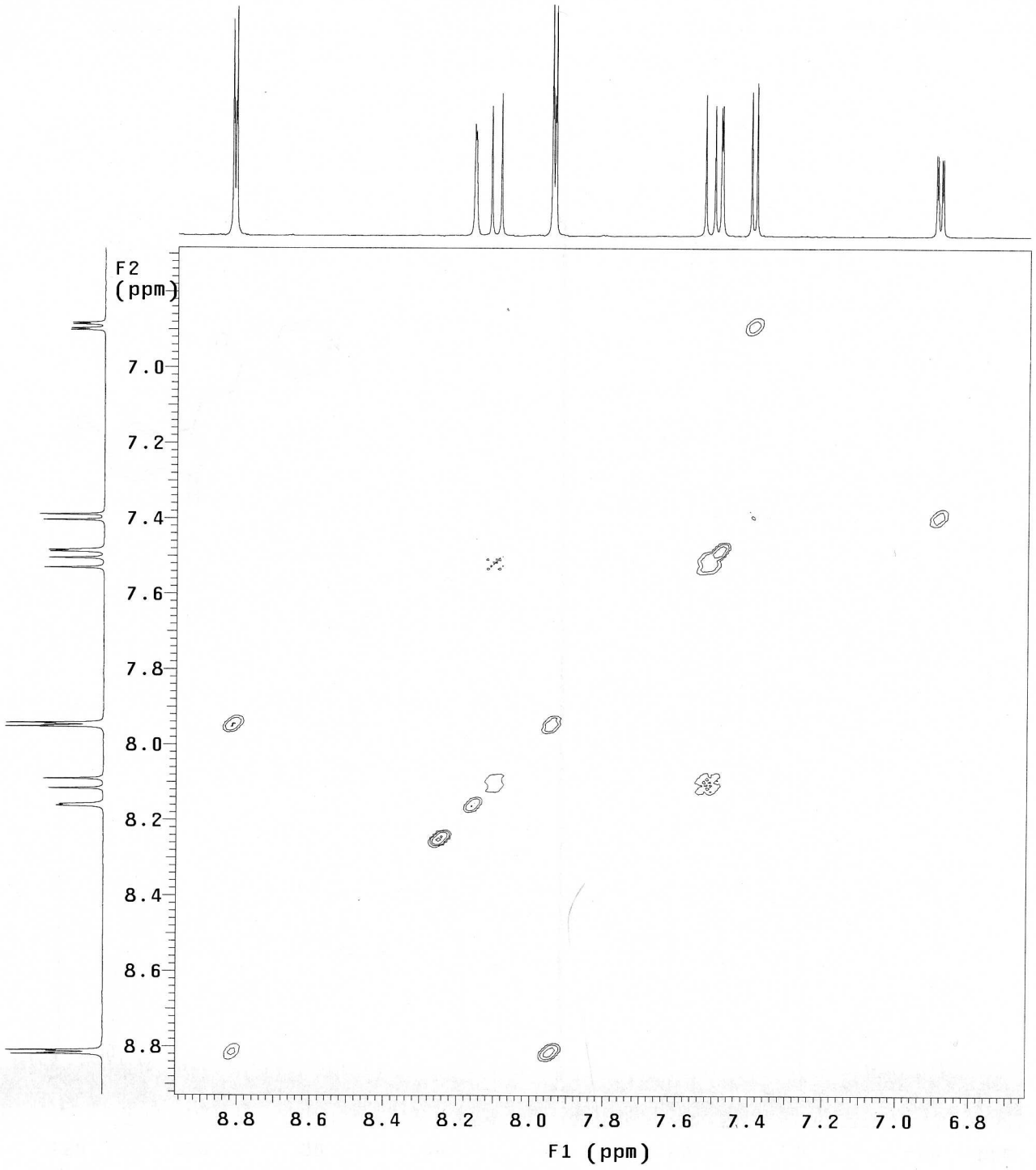
STANDARD PROTON PARAMETERS

Pulse Sequence: gcosy
Solvent: DMSO
Ambient temperature
File: 091004b
GEMINI-200BB "gem2000"

Relax. delay 1.000 sec
Acq. time 0.207 sec
Width 4953.9 Hz
2D Width 4953.9 Hz
Single scan
512 increments
OBSERVE H1, 599.8751449 MHz
DATA PROCESSING
Sine bell 0.103 sec
F1 DATA PROCESSING
Sine bell 0.052 sec
FT size 2048 x 2048
Total time 14 min, 11 sec



13

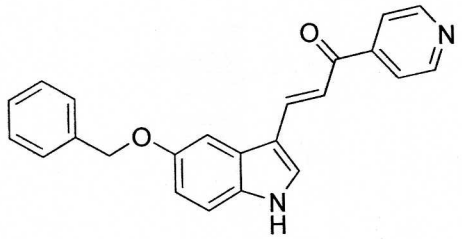


STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
File: 091005a
GEMINI-200BB "gem2000"

Relax. delay 1.000 sec
Pulse 30.9 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.8751449 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 10 sec



14

~~091006a~~

^{13}C NMR

mwk-1-45

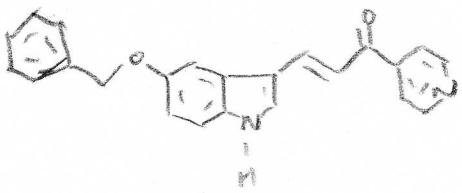
Oct 5 2009

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
User: 1-14-87
INOVA-600 "inova600"

Pulse 54.9 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
40912 repetitions
OBSERVE C13, 150.8387914 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 28 hr, 1 min, 31 sec

23 C
19 unique C



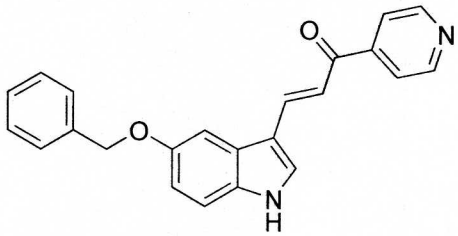
14

STANDARD PROTON PARAMETERS

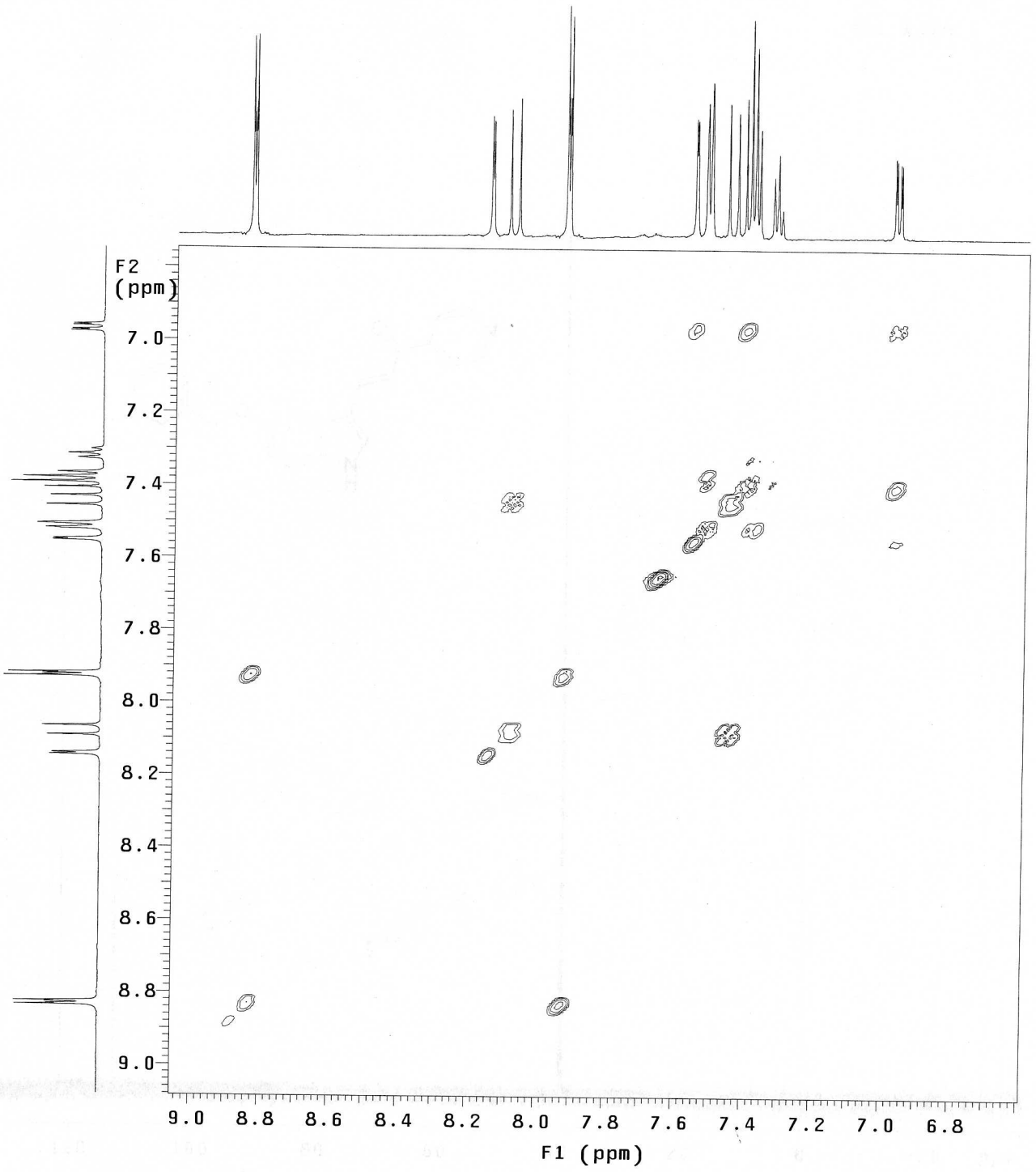
Pulse Sequence: gcosy

Solvent: DMSO
Ambient temperature
File: 091005b
GEMINI-200BB "gem2000"

Relax. delay 1.000 sec
Acq. time 0.223 sec
Width 4594.0 Hz
2D Width 4594.0 Hz
Single scan
512 increments
OBSERVE H1, 599.8751449 MHz
DATA PROCESSING
Sine bell 0.111 sec
F1 DATA PROCESSING
Sine bell 0.056 sec
FT size 2048 x 2048
Total time 14 min, 21 sec



14

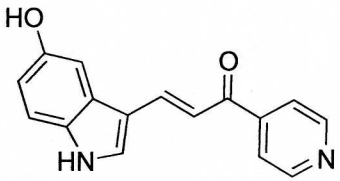


STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
File: 091222d
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8999.9 Hz
16 repetitions
OBSERVE H1, 599.8751465 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



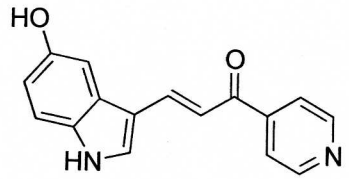
15

13C OBSERVE
S27

Feb 4 2010

Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
UNITY-400 "vxrs400"

Relax. delay 0.100 sec
Pulse 60.6 degrees
Acq. time 1.205 sec
Width 27192.4 Hz
4544 repetitions
OBSERVE C13, 100.5676993 MHz
DECOUPLE H1, 399.9530039 MHz
Low power 3 dB atten.
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
Gauss window 0.500 sec
center at 0.100 sec
FT size 131072
Total time 3 hr, 39 min, 18 sec



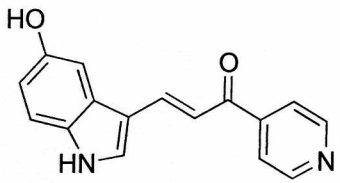
15

STANDARD PROTON PARAMETERS

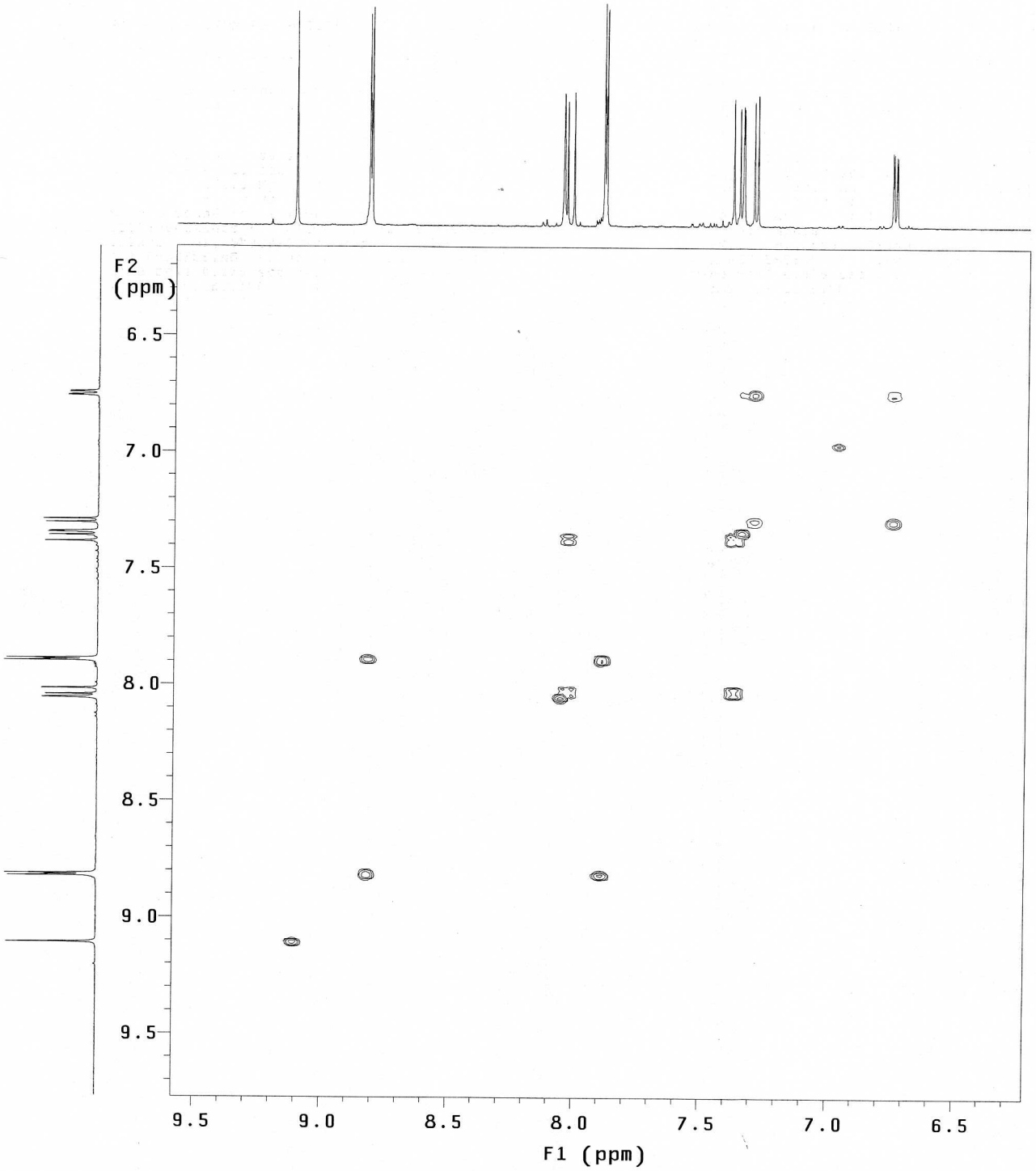
Pulse Sequence: gcosy

Solvent: DMSO
Ambient temperature
File: 091028b
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 6832.9 Hz
2D Width 6832.9 Hz
Single scan
512 increments
OBSERVE H1, 599.8751449 MHz
DATA PROCESSING
Sine bell 0.075 sec
F1 DATA PROCESSING
Sine bell 0.037 sec
FT size 2048 x 2048
Total time 10 min, 17 sec



15

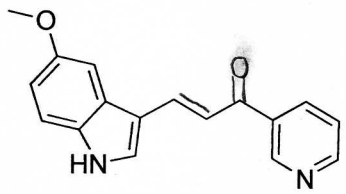


STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8999.9 Hz
16 repetitions
OBSERVE H1, 599.8751457 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



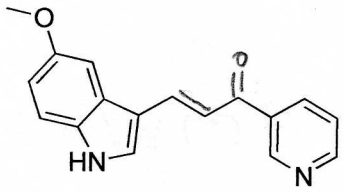
16

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
User: 1-14-87
INOVA-600 "inova600"

Pulse 51.7 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
41104 repetitions
OBSERVE C13, 150.8387879 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 28 hr, 1 min, 31 sec



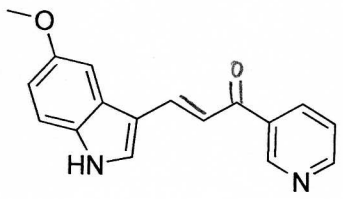
16

STANDARD PROTON PARAMETERS

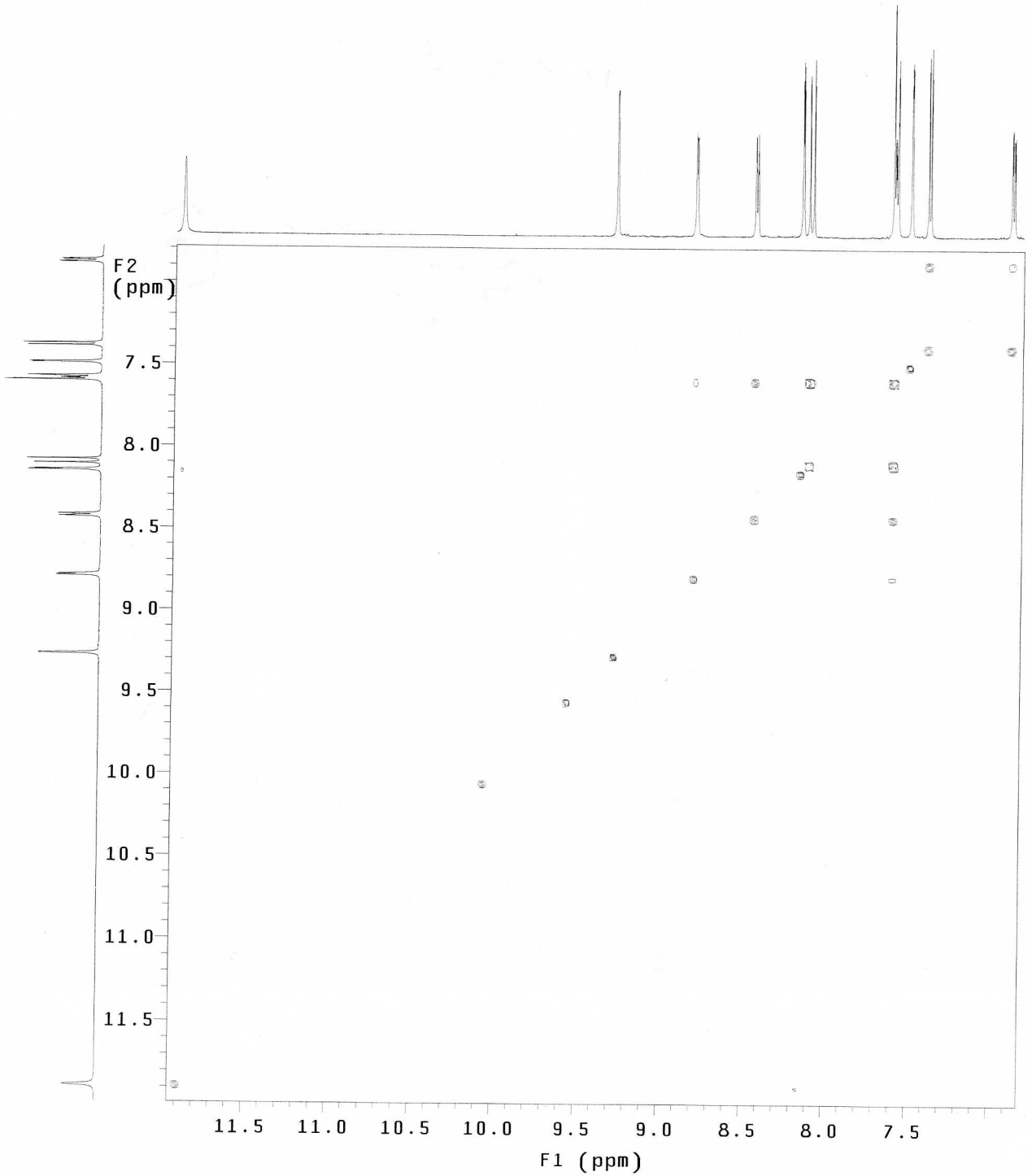
Pulse Sequence: gcosy

Solvent: DMSO
Ambient temperature
File: 100121d
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.138 sec
Width 3713.5 Hz
2D Width 3713.5 Hz
Single scan
256 increments
OBSERVE H1, 599.8751457 MHz
DATA PROCESSING
Sine bell 0.069 sec
F1 DATA PROCESSING
Sine bell 0.034 sec
FT size 1024 x 1024
Total time 5 min, 5 sec



16

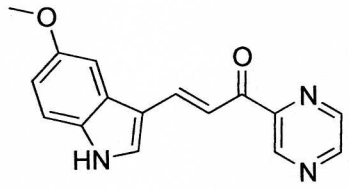


STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
File: 100125e
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8999.9 Hz
16 repetitions
OBSERVE H1, 599.8751461 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec

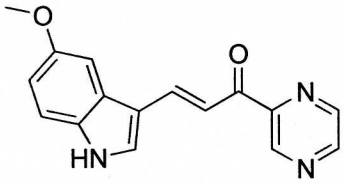


17

13C OBSERVE

Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
File: 100202a
UNITY-400 "vxrs400"

Relax. delay 0.100 sec
Pulse 60.6 degrees
Acq. time 1.205 sec
Width 27192.4 Hz
5408 repetitions
OBSERVE C13, 100.5676972 MHz
DECOUPLE H1, 399.9530039 MHz
Low power 3 dB atten.
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
Gauss window 0.500 sec
center at 0.100 sec
FT size 131072
Total time 36 hr, 33 min, 9 sec



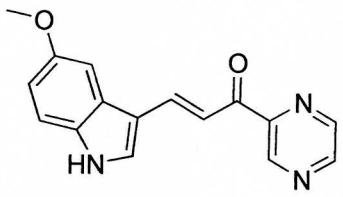
17

STANDARD PROTON PARAMETERS

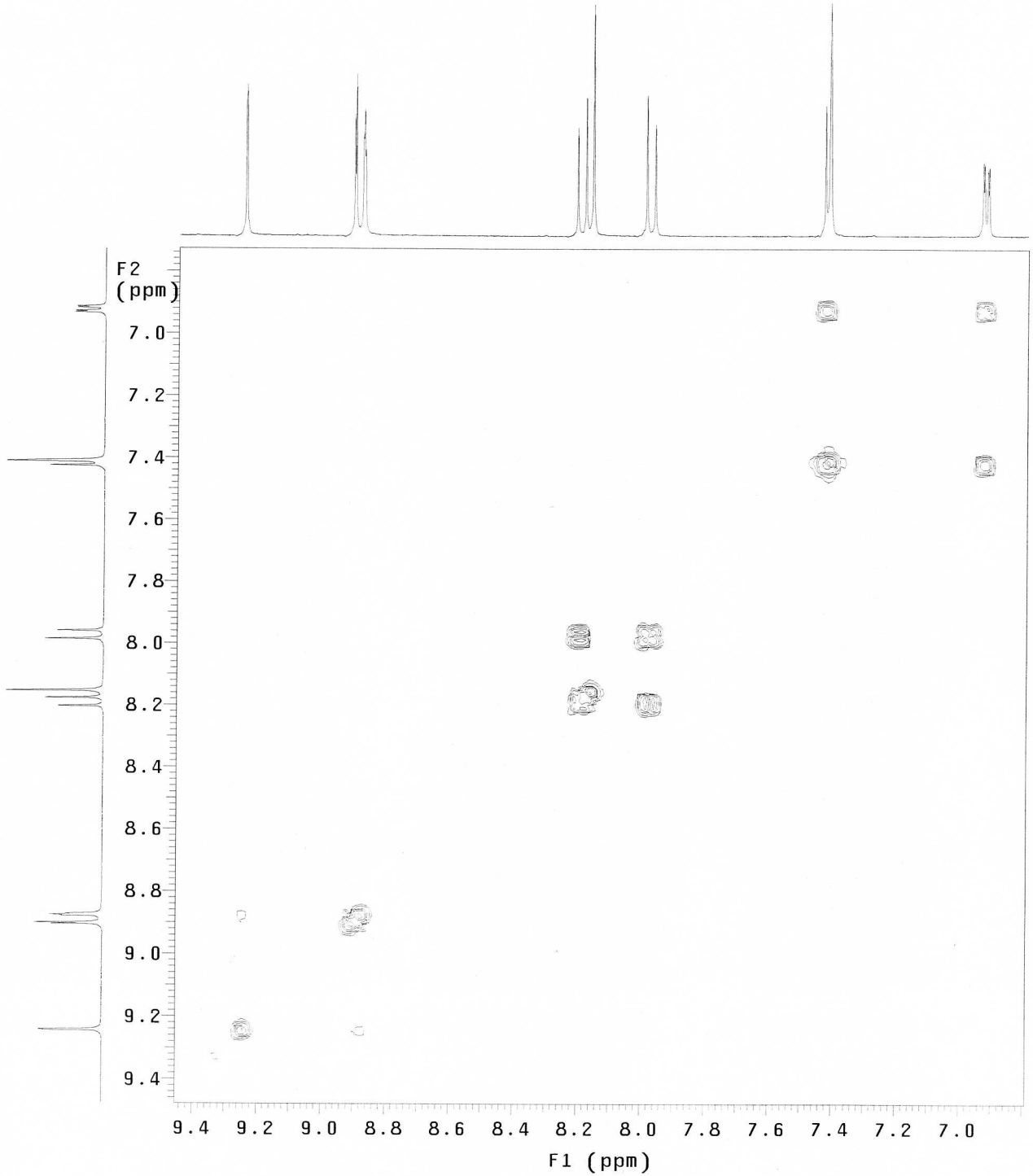
Pulse Sequence: gcosy

Solvent: DMSO
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.134 sec
Width 3819.9 Hz
2D Width 3819.9 Hz
Single scan
256 increments
OBSERVE H1, 599.8751461 MHz
DATA PROCESSING
Sine bell 0.067 sec
F1 DATA PROCESSING
Sine bell 0.034 sec
FT size 1024 x 1024
Total time 5 min, 4 sec



17

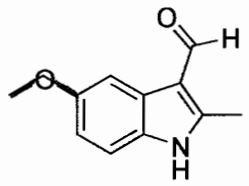


STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
File: 091222b
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.8723067 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



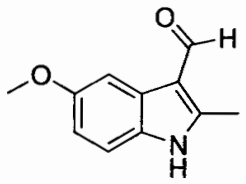
18

STANDARD CARBON PARAMETERS

Feb 2 2010

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 100202b
INOVA-600 "inova600"

Pulse 51.7 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
11408 repetitions
OBSERVE C13, 150.8379917 MHz
DECOUPLE H1, 599.8753144 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 28 hr, 1 min, 31 sec

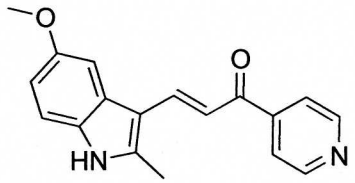


18

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
UNITY-400 "vxrs400"

Relax. delay 2.000 sec
Pulse 51.1 degrees
Acq. time 2.731 sec
Width 5999.7 Hz
32 repetitions
OBSERVE H1, 399.9505784 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 2 min, 31 sec



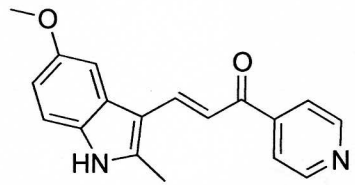
19

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
User: 1-14-87
INOVA-600 "inova600"

Pulse 51.7 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
2192 repetitions
OBSERVE C13, 150.8387920 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 2 hr, 48 min, 9 sec

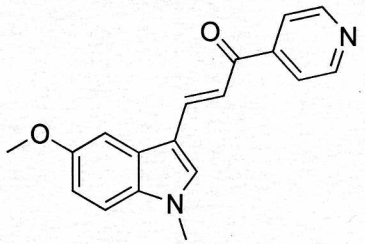


19

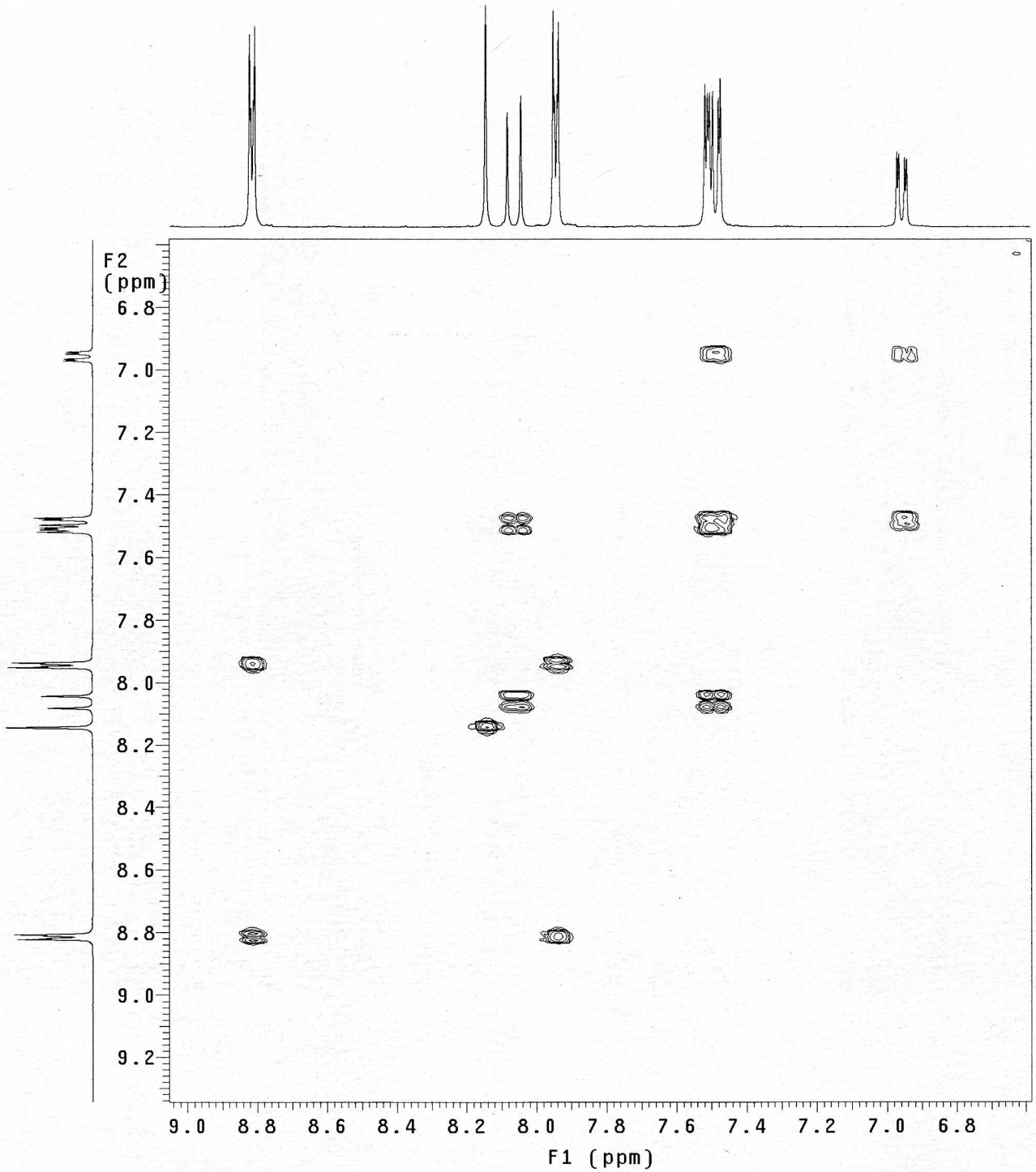
STANDARD 1H OBSERVE

Pulse Sequence: relayh
Solvent: DMSO
Ambient temperature
UNITY-400 "vxrs400"

Relax. delay 1.000 sec
COSY 90-90
Acq. time 0.232 sec
Width 1103.8 Hz
2D Width 1103.8 Hz
4 repetitions
128 increments
OBSERVE H1, 399.9505719 MHz
DATA PROCESSING
Sine bell 0.116 sec
F1 DATA PROCESSING
Sine bell 0.058 sec
FT size 512 x 512
Total time 11 min, 9 sec



20

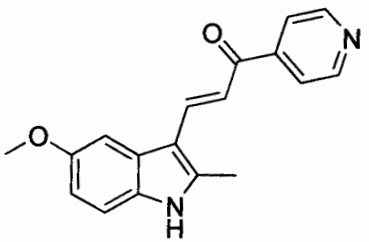


STANDARD PROTON PARAMETERS

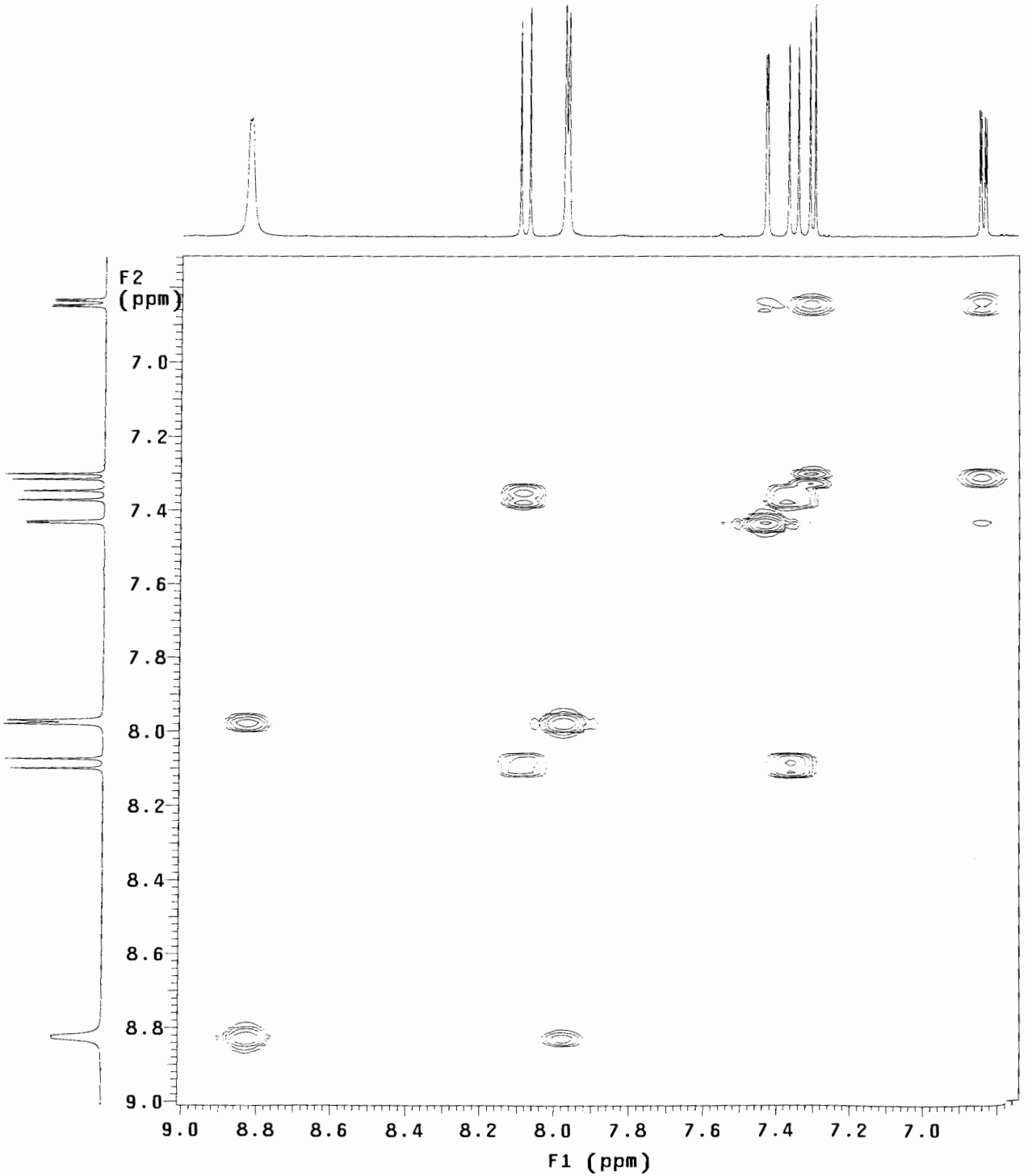
Pulse Sequence: gcosy

Solvent: DMSO
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.126 sec
Width 2035.4 Hz
2D Width 2035.4 Hz
Single scan
84 increments
OBSERVE H1, 599.8751470 MHz
DATA PROCESSING
Sine bell 0.063 sec
F1 DATA PROCESSING
Sine bell 0.021 sec
FT size 512 x 512
Total time 1 min, 39 sec



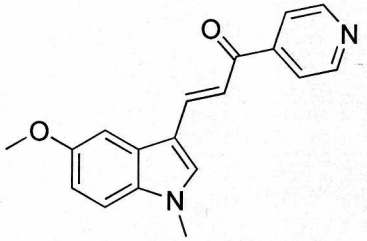
19



STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
UNITY-400 "vxrs400"

Relax. delay 2.000 sec
Pulse 47.4 degrees
Acq. time 2.731 sec
Width 5999.7 Hz
32 repetitions
OBSERVE H1, 399.9505719 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 2 min, 31 sec

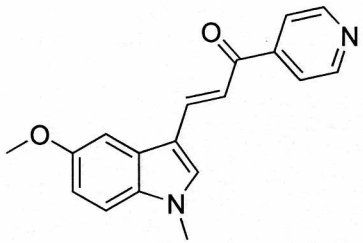


20

13C OBSERVE

Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
UNITY-400 "vxrs400"

Relax. delay 0.100 sec
Pulse 60.6 degrees
Acq. time 1.205 sec
Width 27192.4 Hz
4944 repetitions
OBSERVE C13, 100.5676951 MHz
DECOUPLE H1, 399.9530039 MHz
Low power 3 dB atten.
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
Gauss window 0.500 sec
center at 0.100 sec
FT size 131072
Total time 36 hr, 33 min, 9 sec



20

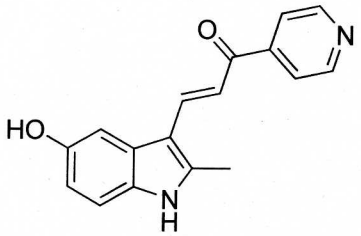
STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8999.9 Hz
16 repetitions
OBSERVE H1, 599.8751446 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec

Aug 12 2010



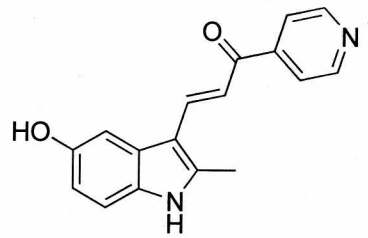
21

STANDARD CARBON PARAMETERS

Aug 12 2010

Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
User: 1-14-87
INOVA-600 "inova600"

Pulse 46.4 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
2576 repetitions
OBSERVE C13, 150.8387896 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 28 hr, 1 min, 31 sec



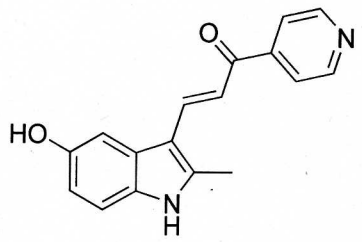
21

STANDARD PROTON PARAMETERS

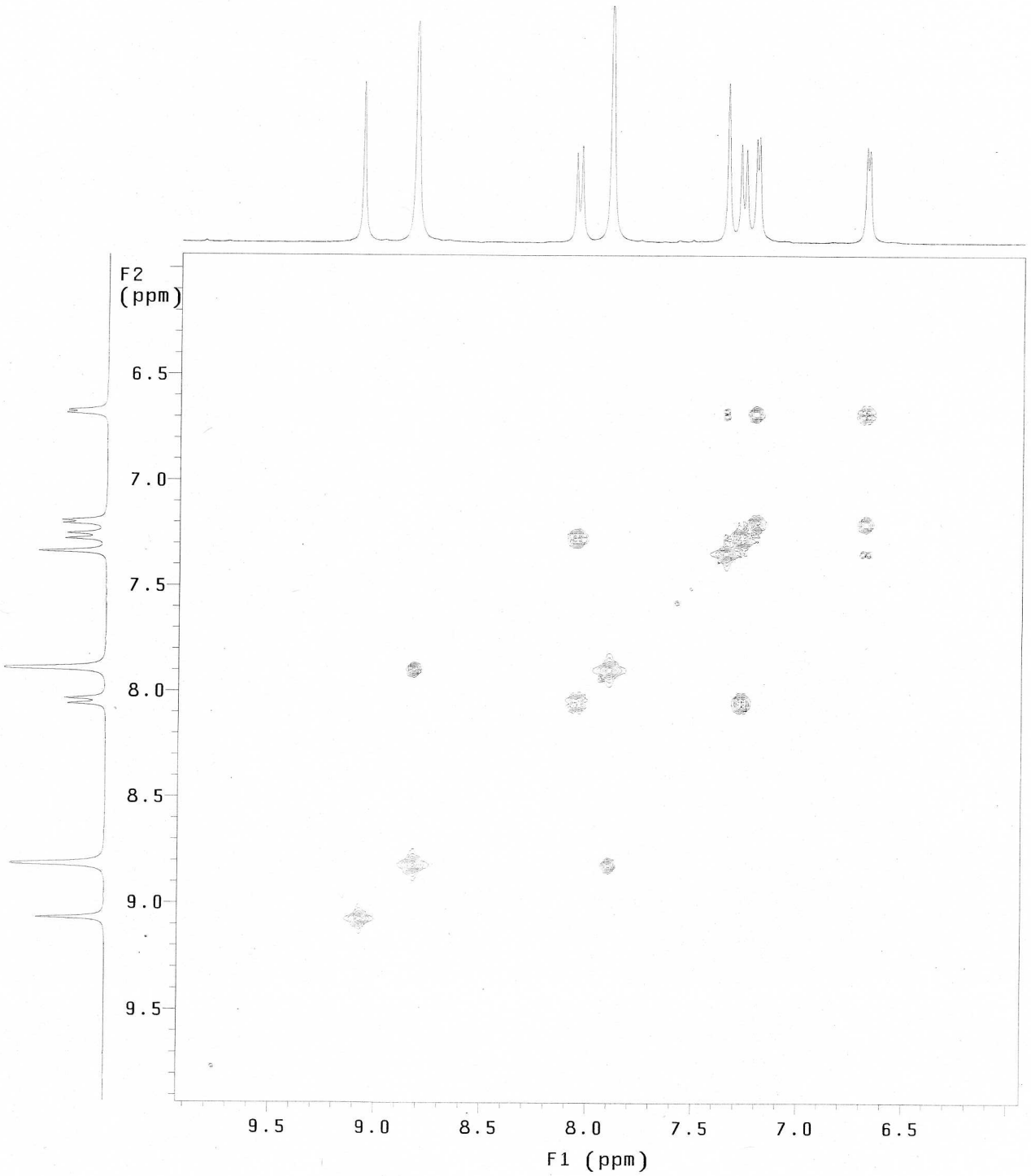
Pulse Sequence: gcosy

Solvent: DMSO
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.213 sec
Width 2400.7 Hz
2D Width 2400.7 Hz
Single scan
256 increments
OBSERVE H1, 599.8751446 MHz
DATA PROCESSING
Sine bell 0.107 sec
F1 DATA PROCESSING
Sine bell 0.053 sec
FT size 1024 x 1024
Total time 5 min, 30 sec



21



STANDARD PROTON PARAMETERS

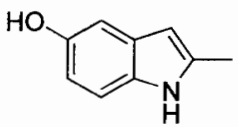
Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
File: 100823a
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8999.9 Hz
16 repetitions
OBSERVE H1, 599.8751449 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec

p173

Aug 23 2010



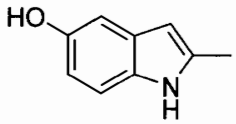
22

STANDARD CARBON PARAMETERS

Aug 23 2010

Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
User: 1-14-87
INOVA-600 "inova600"

Pulse 46.4 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
1440 repetitions
OBSERVE C13, 150.8387893 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 28 hr, 1 min, 31 sec



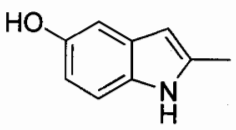
22

STANDARD PROTON PARAMETERS

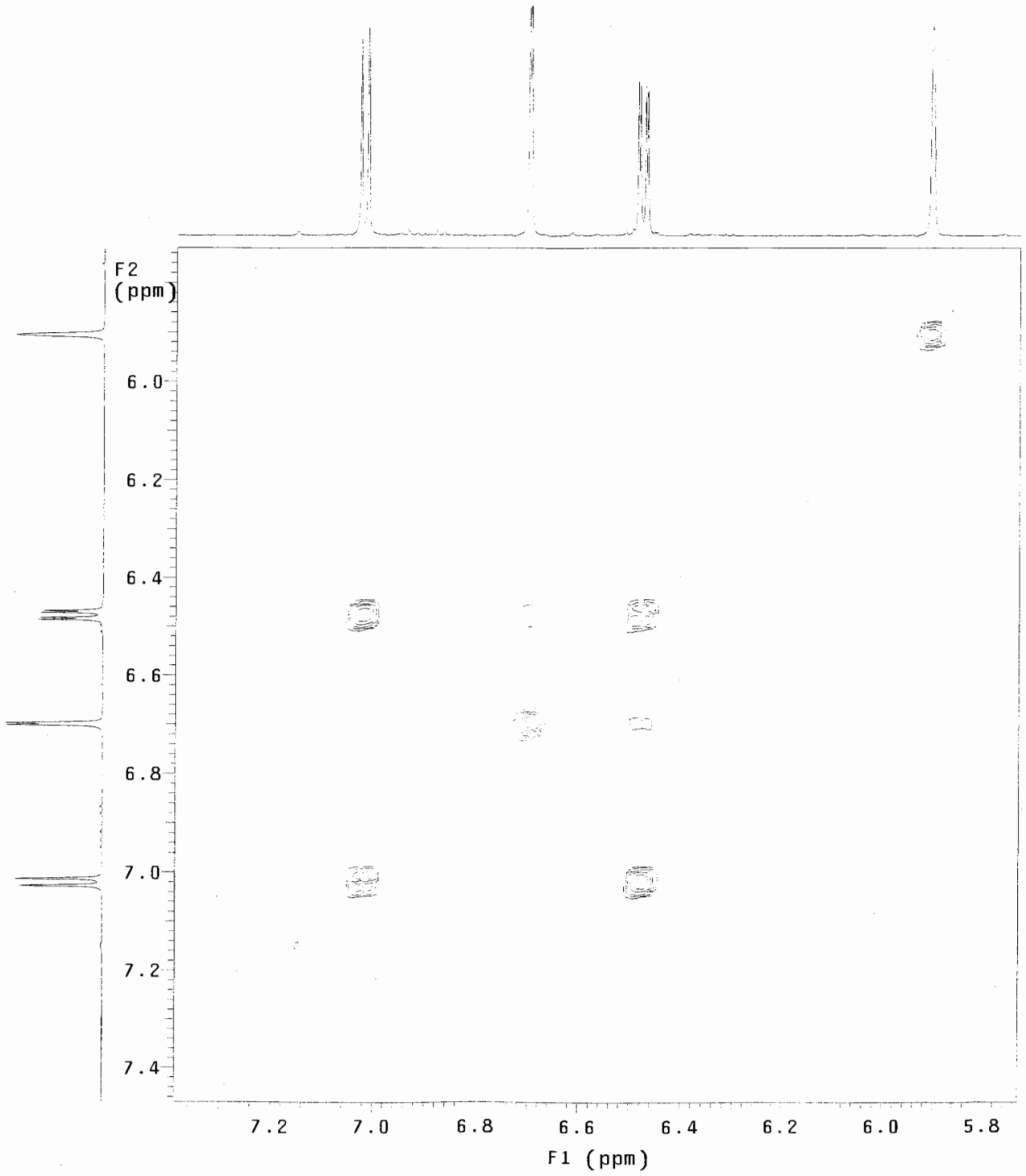
Pulse Sequence: gcosy

Solvent: DMSO
Ambient temperature
File: 100823b
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.159 sec
Width 1608.4 Hz
2D Width 1608.4 Hz
Single scan
128 increments
OBSERVE H1, 599.8751449 MHz
DATA PROCESSING
Sine bell 0.080 sec
F1 DATA PROCESSING
Sine bell 0.040 sec
FT size 512 x 512
Total time 2 min, 37 sec



22

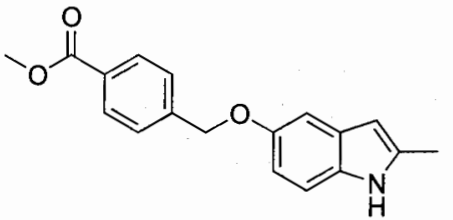


STANDARD 1H OBSERVE

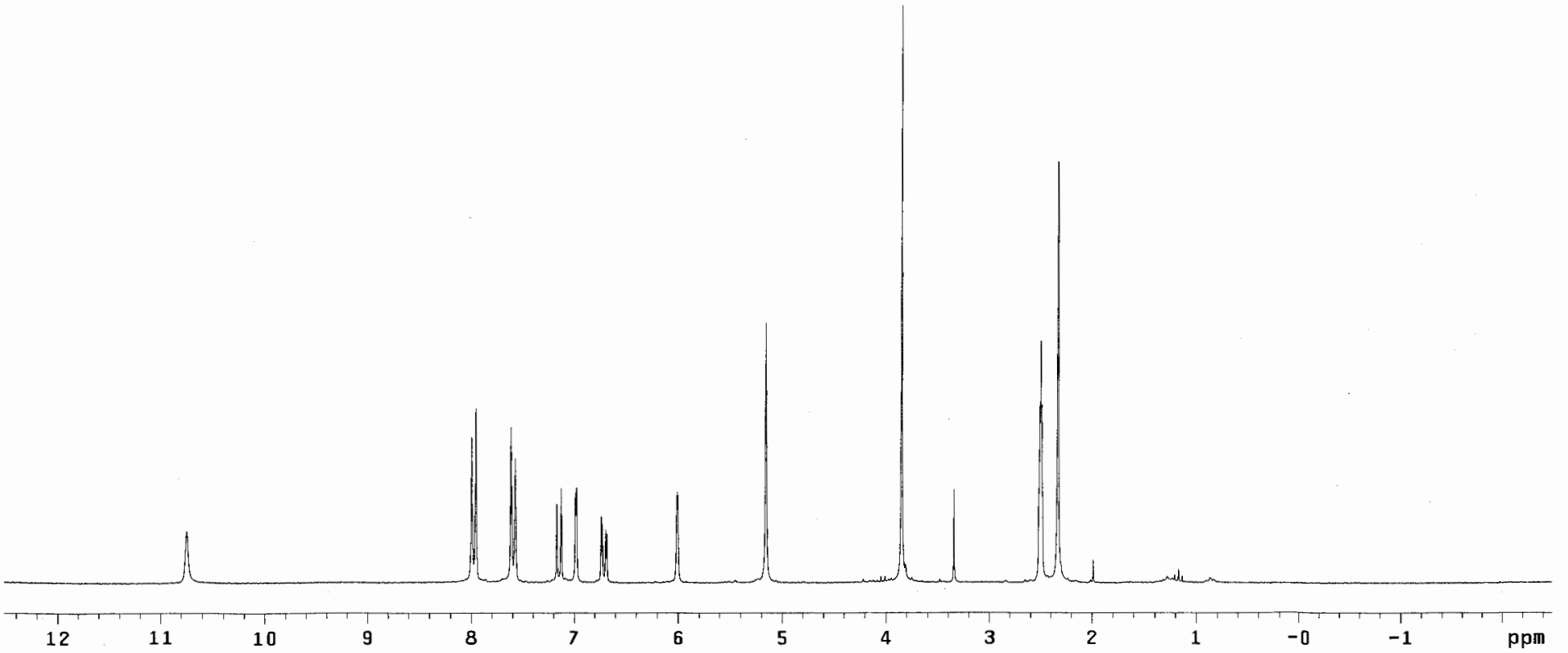
Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
File: 101022b
INOVA-600 "inova-wkst"

Relax. delay 2.000 sec
Pulse 31.9 degrees
Acq. time 2.500 sec
Width 3000.3 Hz
32 repetitions
OBSERVE H1, 199.9760204 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 2 min, 24 sec



23

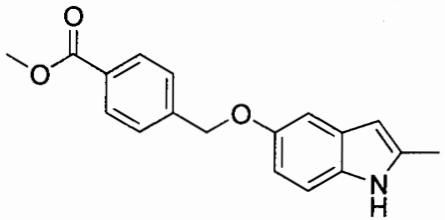


STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
User: 1-14-87
File: 101119a
INOVA-600 "inova-wkst"

Pulse 46.4 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
31264 repetitions
OBSERVE C13, 150.8387949 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 28 hr, 1 min, 31 sec



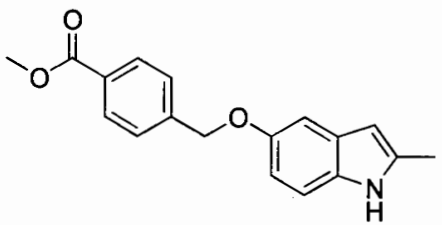
23

STANDARD 1H OBSERVE

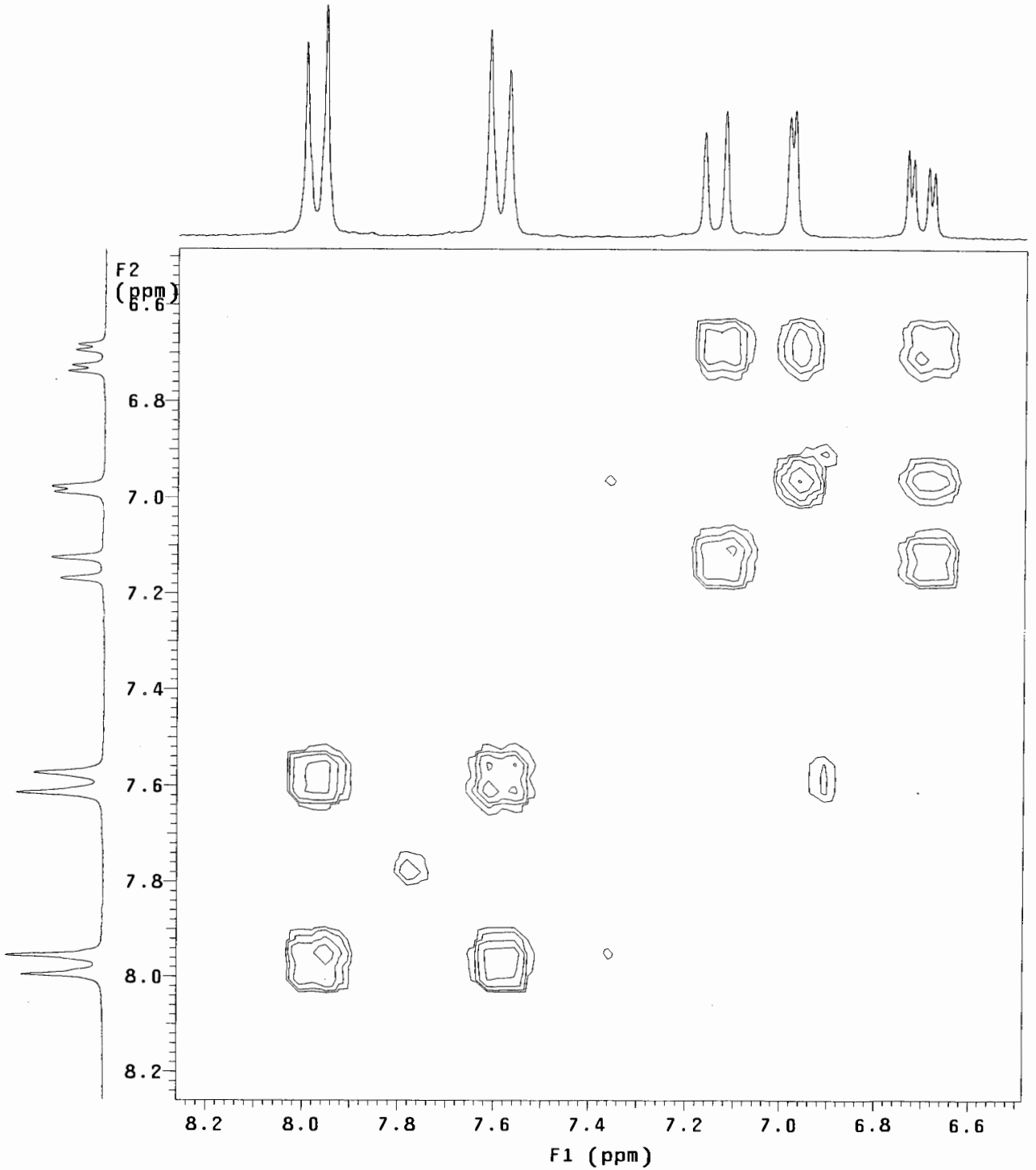
Pulse Sequence: relayh

Solvent: DMSO
Ambient temperature
File: 101022c
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
COSY 90-90
Acq. time 0.180 sec
Width 354.8 Hz
2D Width 354.8 Hz
4 repetitions
64 increments
OBSERVE H1, 199.9760204 MHz
DATA PROCESSING
Sine bell 0.090 sec
F1 DATA PROCESSING
Sine bell 0.090 sec
FT size 128 x 128
Total time 5 min, 29 sec



23

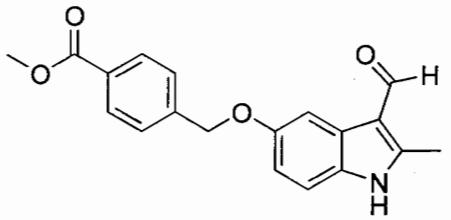


STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
File: 100921a
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8999.9 Hz
16 repetitions
OBSERVE H1, 599.8751455 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



24

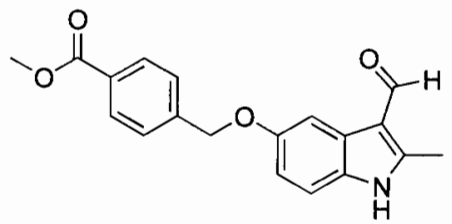
STANDARD CARBON PARAMETERS

Jun 11 2011

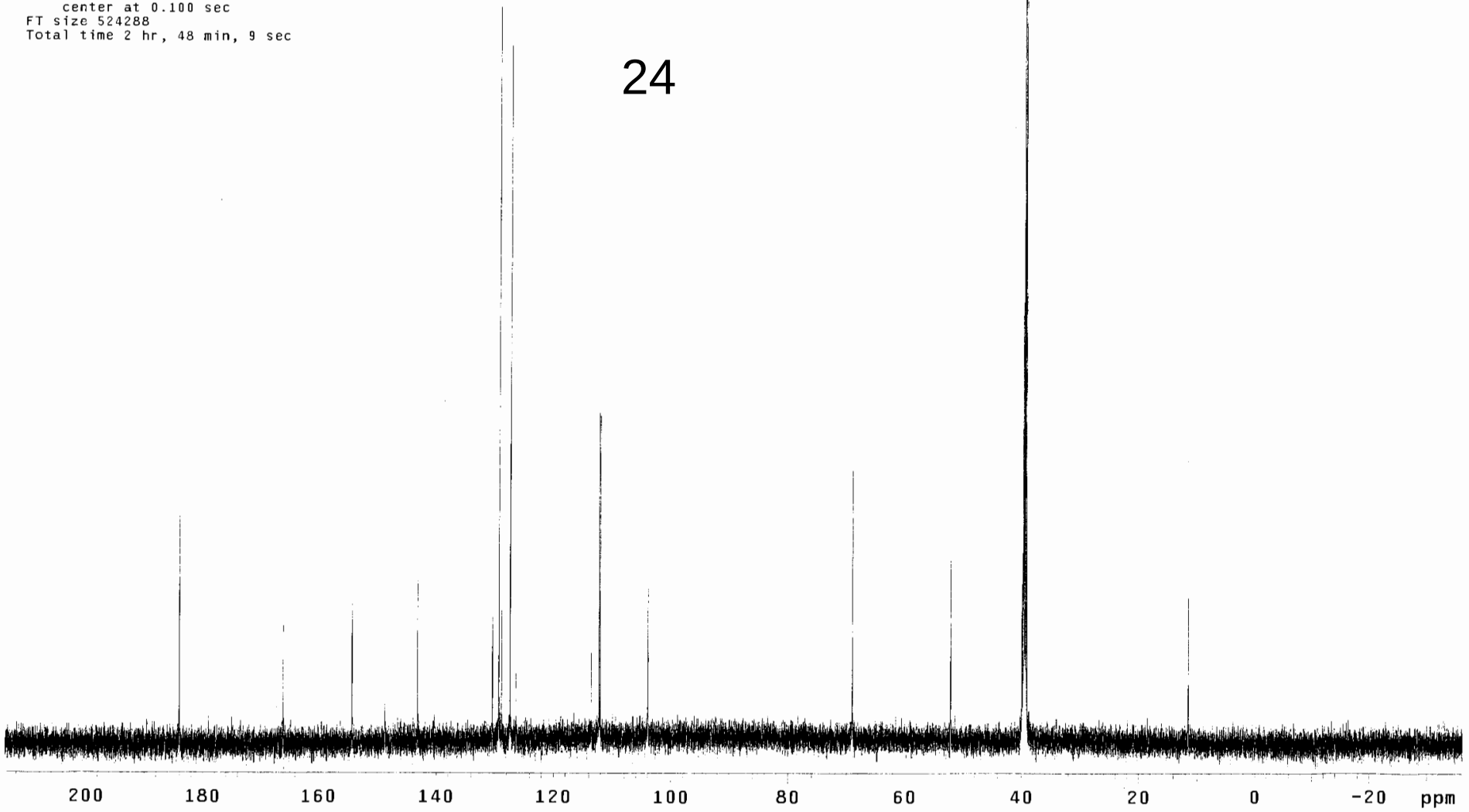
Pulse Sequence: s2pul

Solvent: DMSO
Temp. 23.0 C / 296.1 K
User: 1-14-87
File: 2-203_13C_6_11_2-11
INOVA-600 "inova600"

Pulse 46.4 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
7216 repetitions
OBSERVE C13, 150.8387946 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 2 hr, 48 min, 9 sec



24

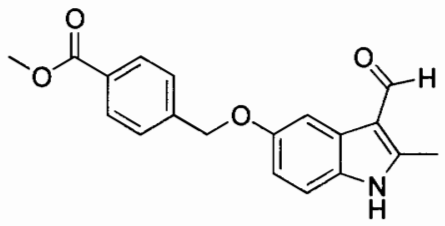


STANDARD PROTON PARAMETERS

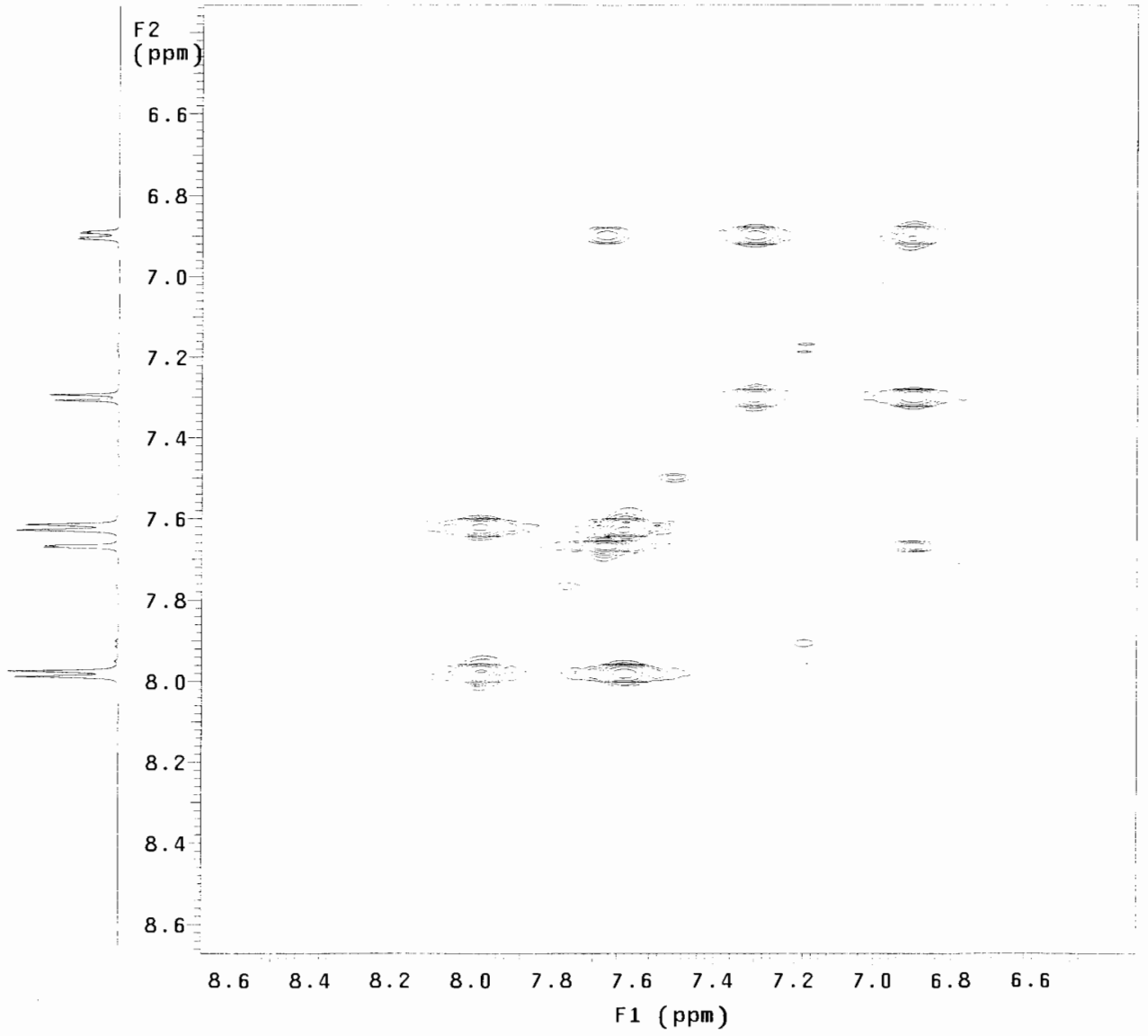
Pulse Sequence: gcosy

Solvent: DMSO
Temp. 23.0 C / 296.1 K
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.182 sec
Width 1407.3 Hz
2D Width 1407.3 Hz
Single scan
58 increments
OBSERVE H1, 599.8751444 MHz
DATA PROCESSING
Sine bell 0.091 sec
F1 DATA PROCESSING
Sine bell 0.021 sec
FT size 512 x 512
Total time 1 min, 12 sec



24

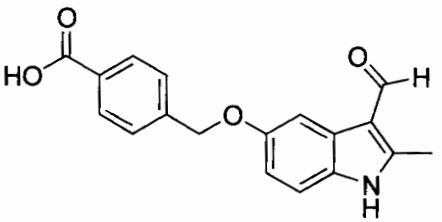


STANDARD PROTON PARAMETERS

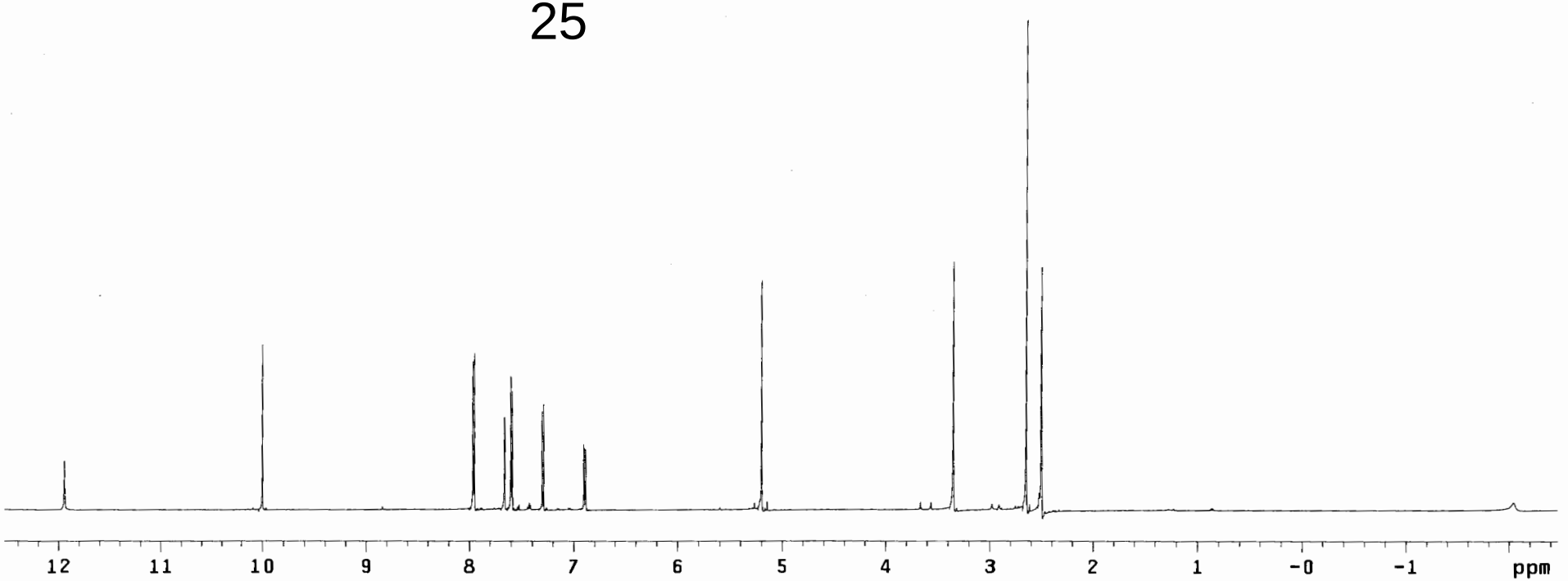
Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
File: 101013b
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8999.9 Hz
16 repetitions
OBSERVE H1, 599.8751451 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



25



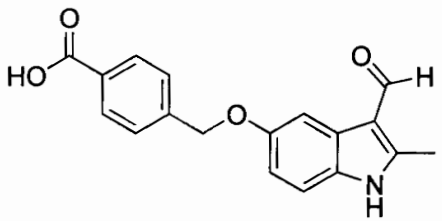
STANDARD CARBON PARAMETERS

Jun 11 2011

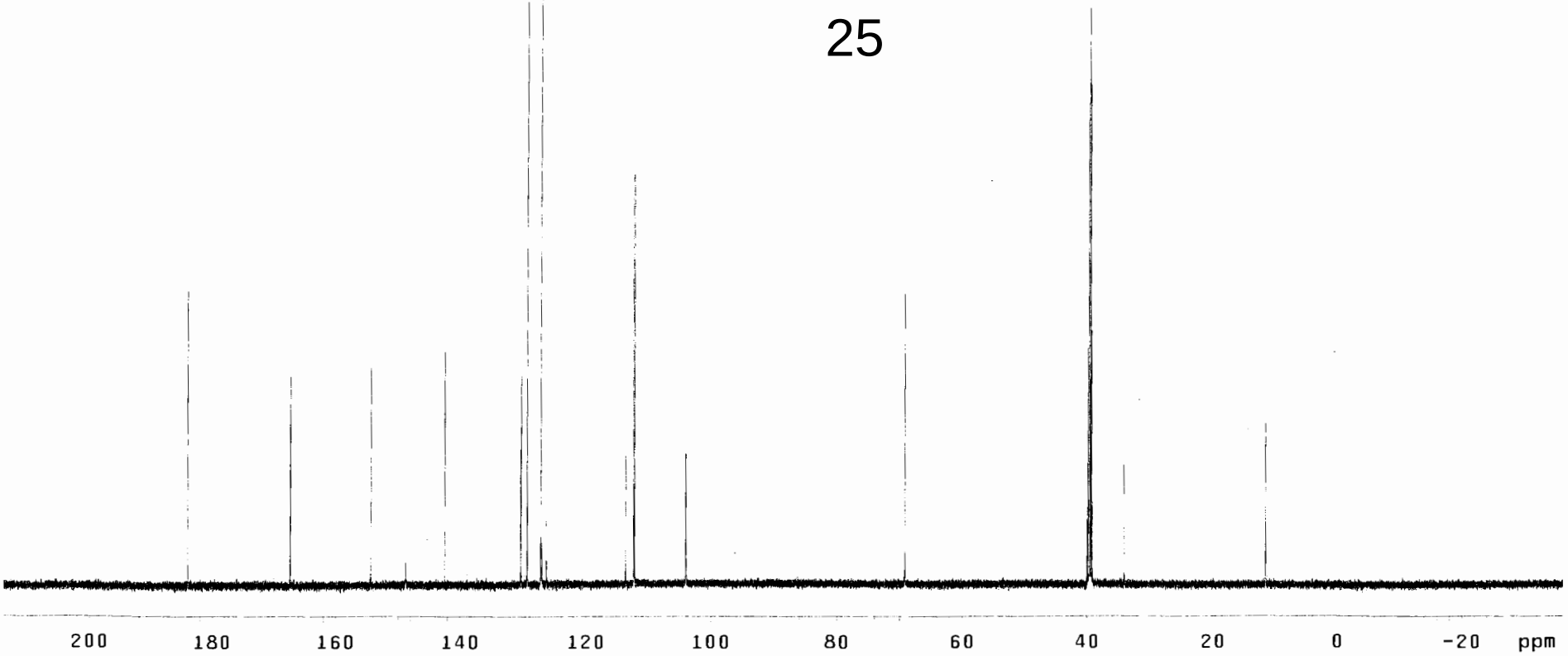
Pulse Sequence: s2pul

Solvent: DMSO
Temp. 23.0 C / 296.1 K
User: 1-14-87
INDVA-600 "inova600"

Pulse 46.4 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
4944 repetitions
DBSERVE C13, 150.8387904 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 28 hr, 1 min, 31 sec



25

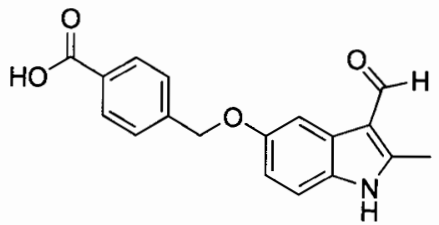


STANDARD PROTON PARAMETERS

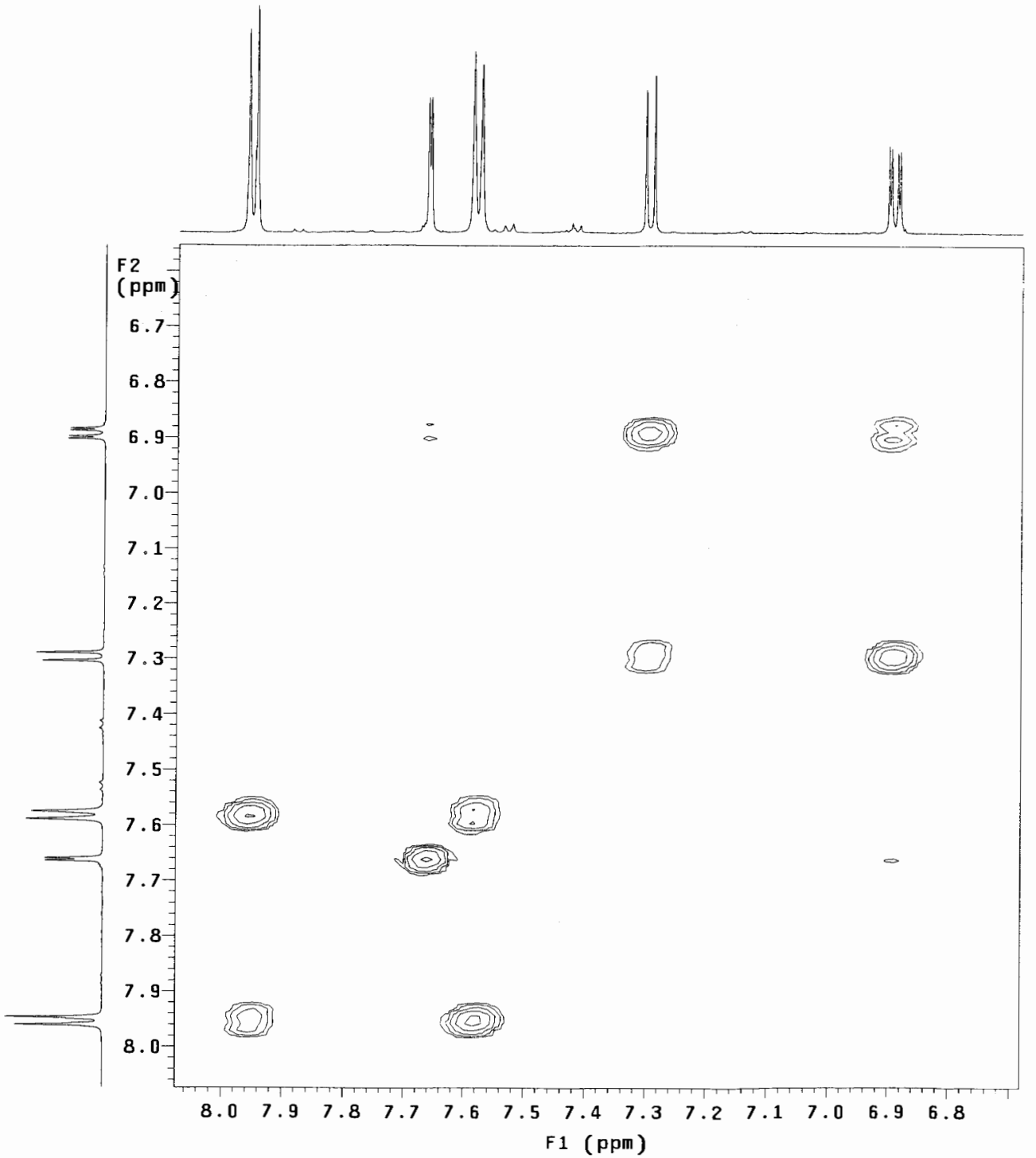
Pulse Sequence: gcosy

Solvent: DMSO
Ambient temperature
File: 100929b
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Acq. time 0.130 sec
Width 3950.0 Hz
2D Width 3950.0 Hz
Single scan
256 increments
OBSERVE H1, 599.8751454 MHz
DATA PROCESSING
Sine bell 0.065 sec
F1 DATA PROCESSING
Sine bell 0.032 sec
FT size 1024 x 1024
Total time 5 min, 3 sec



25

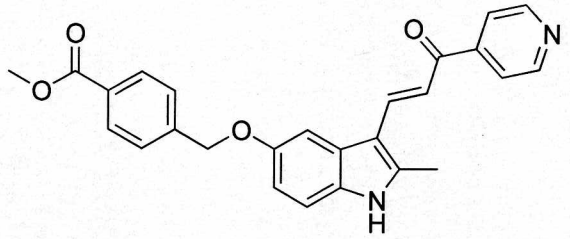


STANDARD PROTON PARAMETERS

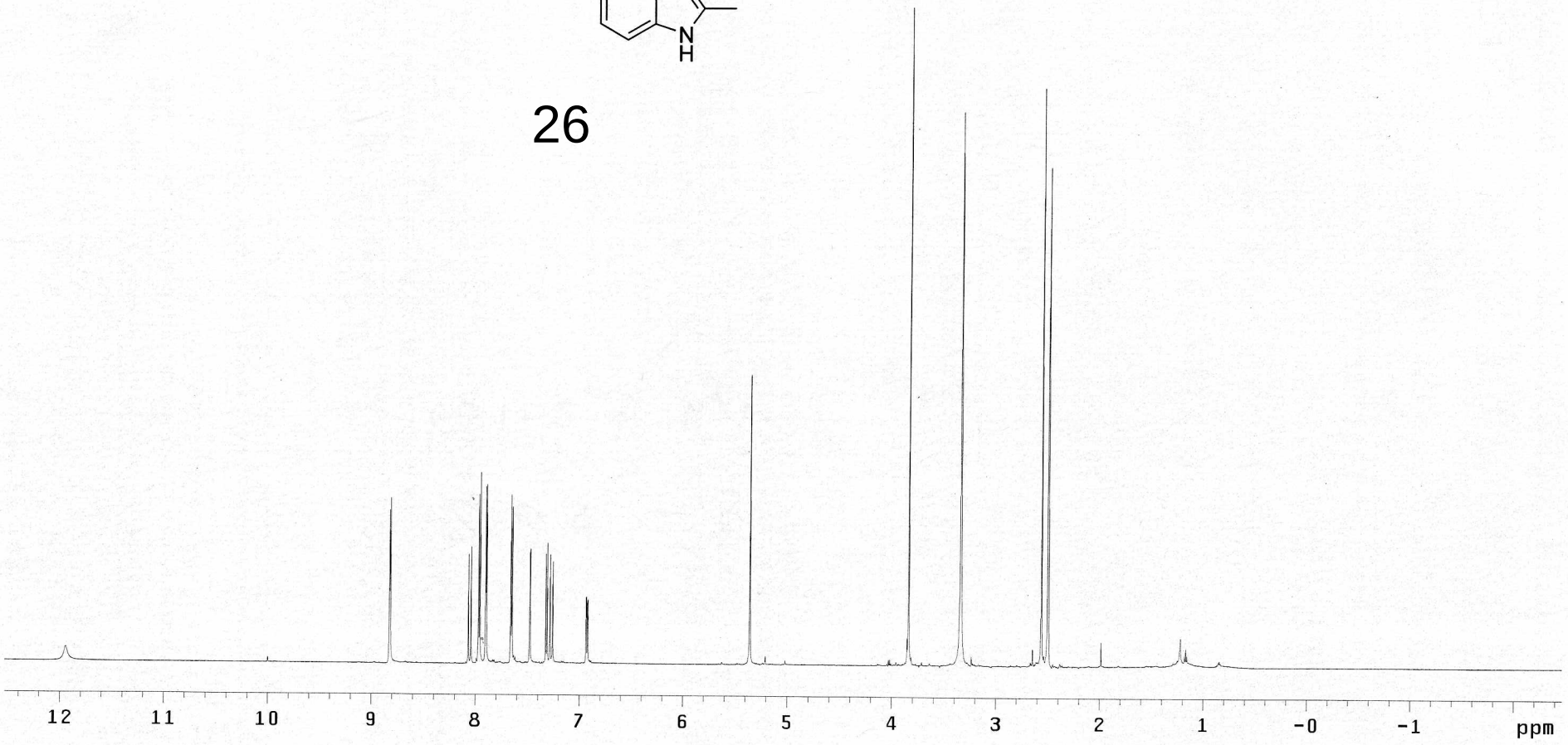
Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8999.9 Hz
16 repetitions
OBSERVE H1, 599.8751449 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



26

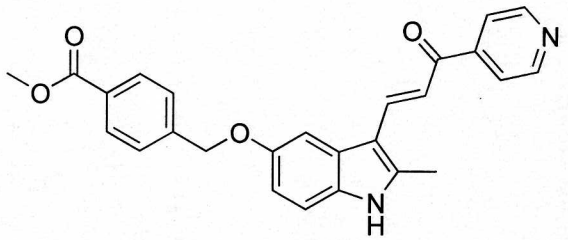


STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
User: 1-14-87
File: 101123j
INOVA-600 "inova600"

Pulse 46.4 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
42784 repetitions
OBSERVE C13, 150.8387924 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 28 hr, 1 min, 31 sec



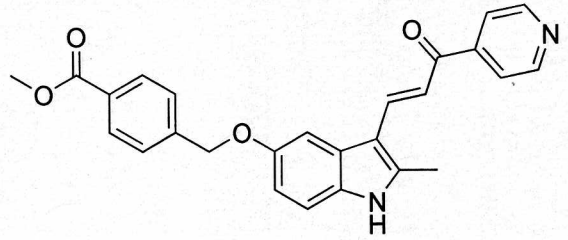
26

STANDARD PROTON PARAMETERS

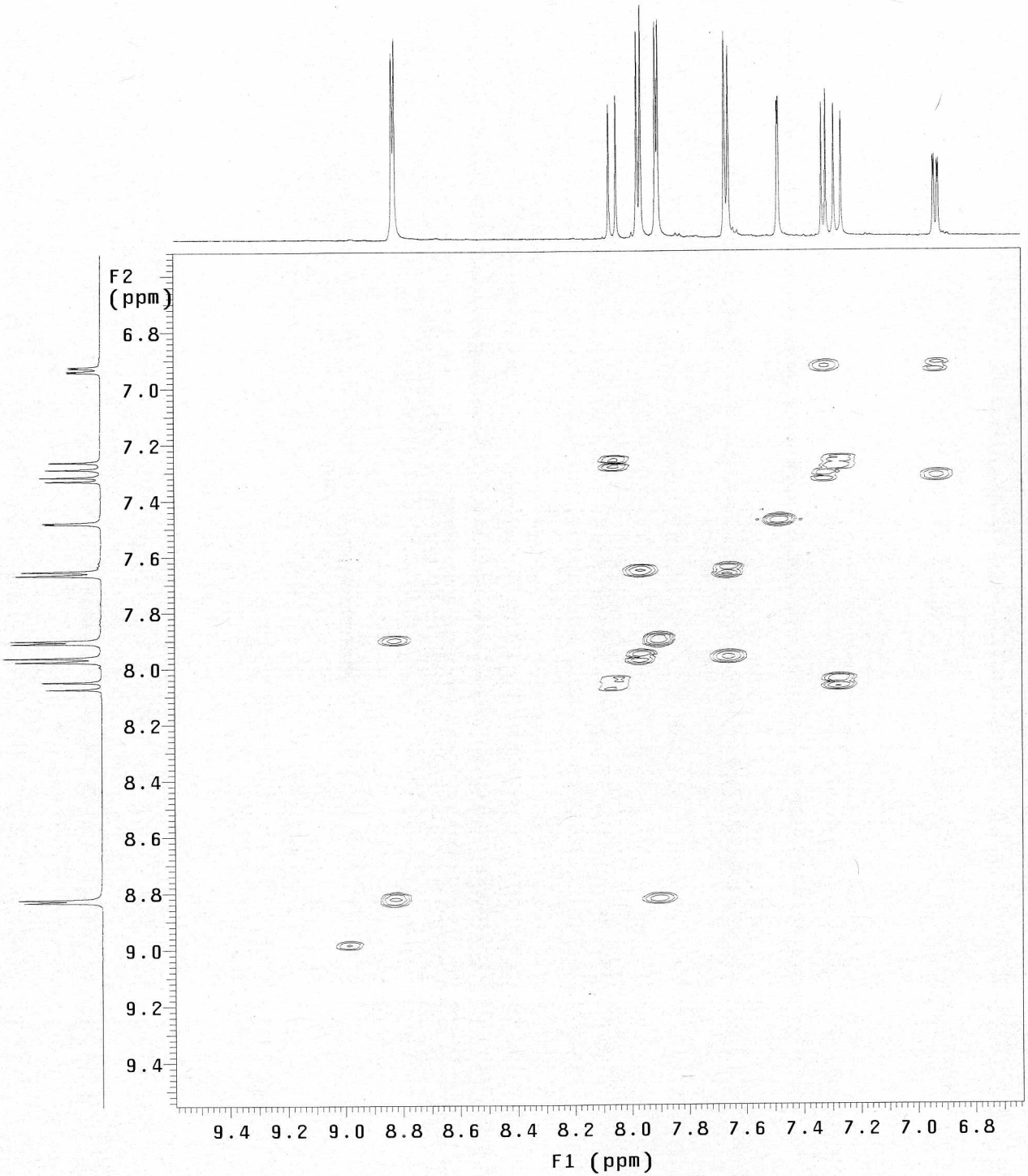
Pulse Sequence: gcosy

Solvent: DMSO
Ambient temperature
File: 101123i
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.235 sec
Width 2176.0 Hz
2D Width 2176.0 Hz
Single scan
90 increments
OBSERVE H1, 599.8751449 MHz
DATA PROCESSING
Sine bell 0.118 sec
F1 DATA PROCESSING
Sine bell 0.021 sec
FT size 1024 x 1024
Total time 1 min, 56 sec



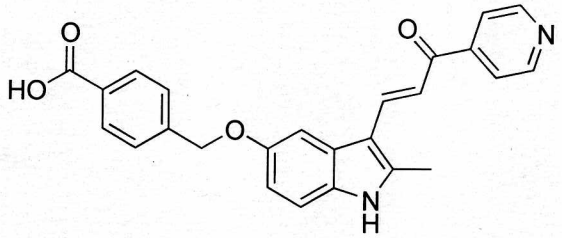
26



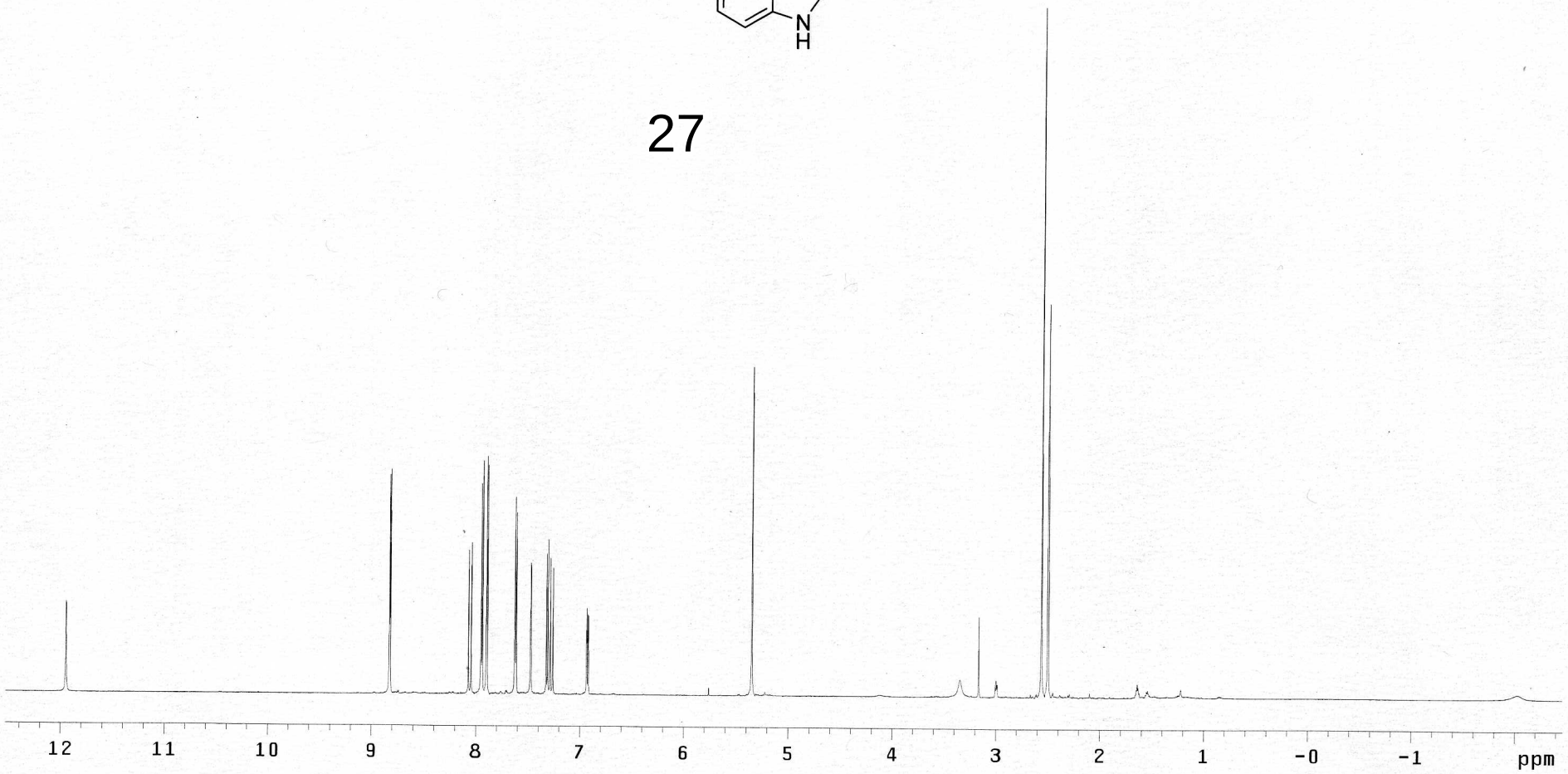
STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8999.9 Hz
16 repetitions
OBSERVE H1, 599.8751447 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



27

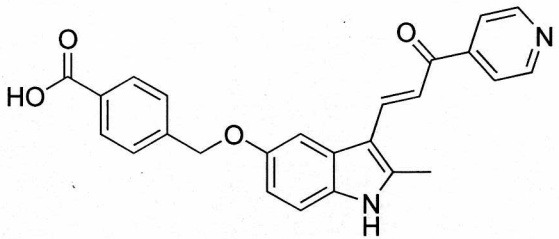


STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
User: 1-14-87
File: 101129b
INOVA-600 "inova600"

Pulse 93.8 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
41328 repetitions
OBSERVE C13, 150.8387926 MHz
DECOUPLE H1, 599.8781638 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 11 hr, 46 min, 14 sec



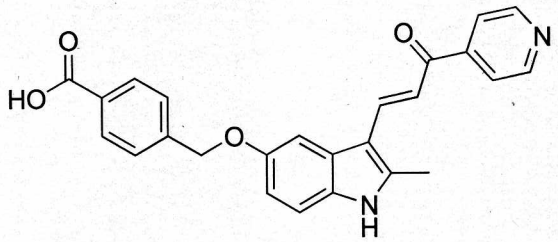
27

STANDARD PROTON PARAMETERS

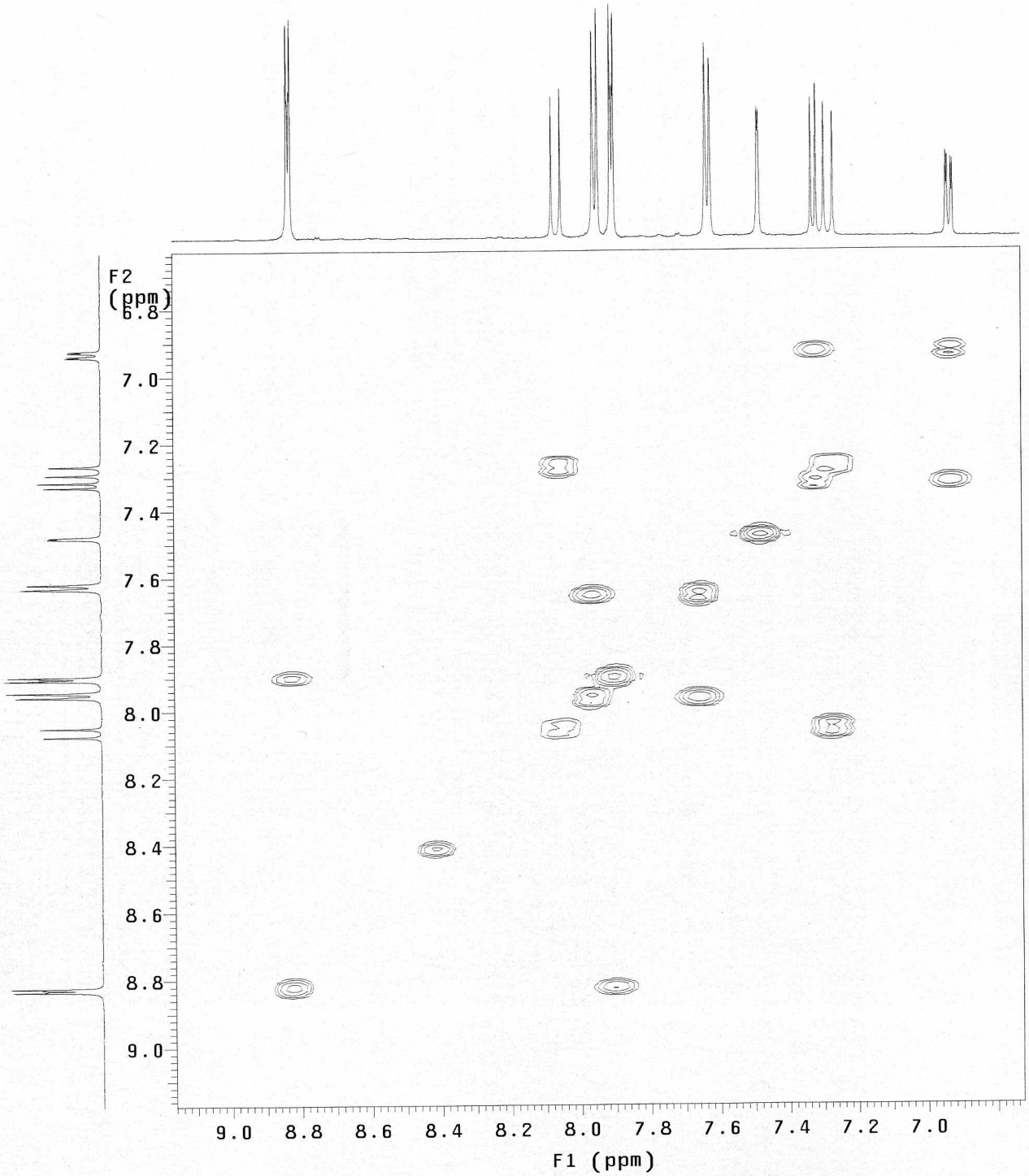
Pulse Sequence: gcosy

Solvent: DMSO
Ambient temperature
File: 101123g
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.140 sec
Width 1833.1 Hz
2D Width 1833.1 Hz
Single scan
76 increments
OBSERVE H1, 599.8751447 MHz
DATA PROCESSING
Sine bell 0.070 sec
F1 DATA PROCESSING
Sine bell 0.021 sec
FT size 512 x 512
Total time 1 min, 31 sec



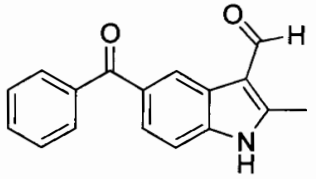
27



STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
File: 2-25B
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8999.9 Hz
16 repetitions
OBSERVE H1, 599.8751443 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



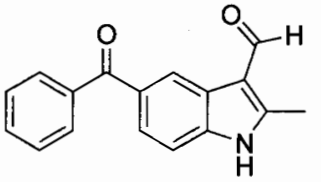
28

STANDARD CARBON PARAMETERS

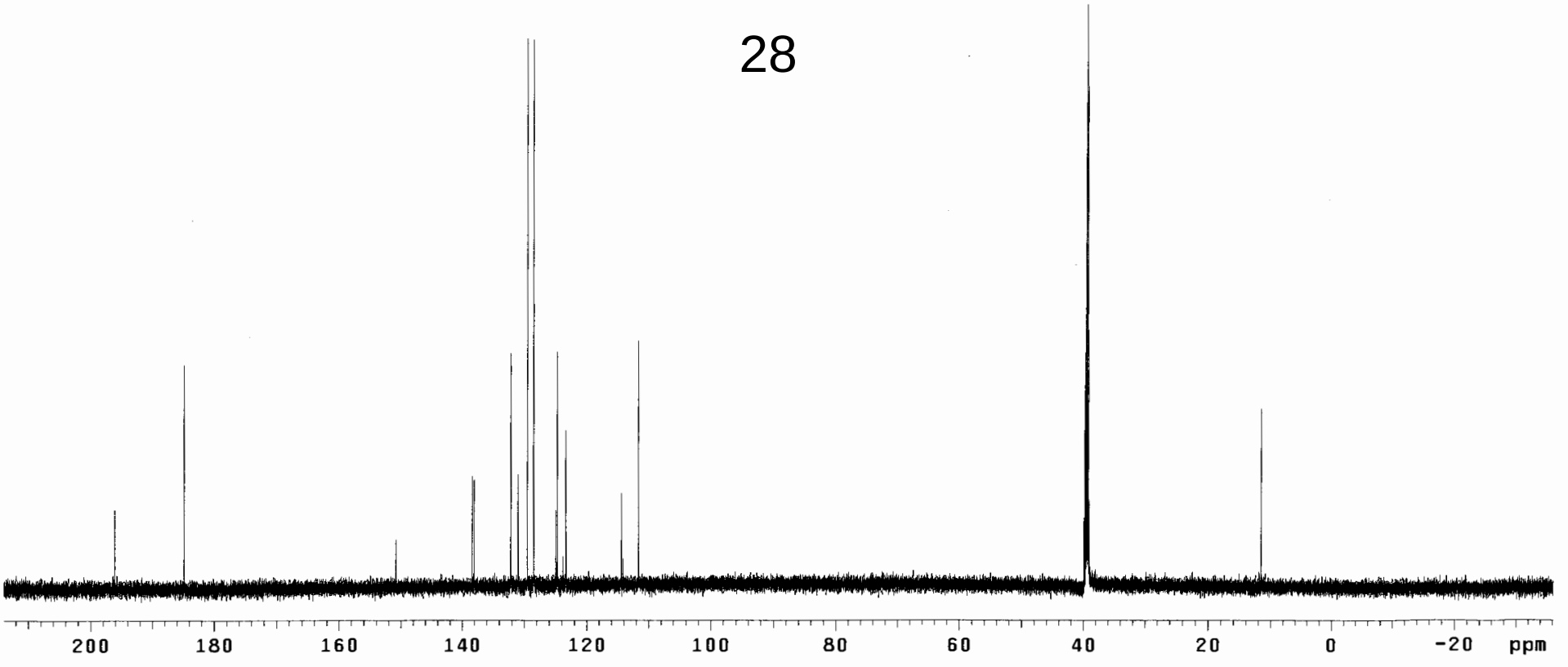
Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
User: 1-14-87
File: 2-21b_c13_6_6_2011
INOVA-600 "inova-wkst"

Pulse 46.4 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
10000 repetitions
OBSERVE C13, 150.8387860 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 2 hr, 48 min, 9 sec



28

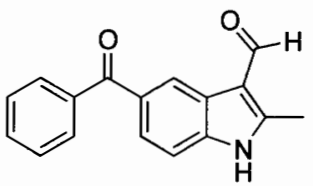


STANDARD PROTON PARAMETERS

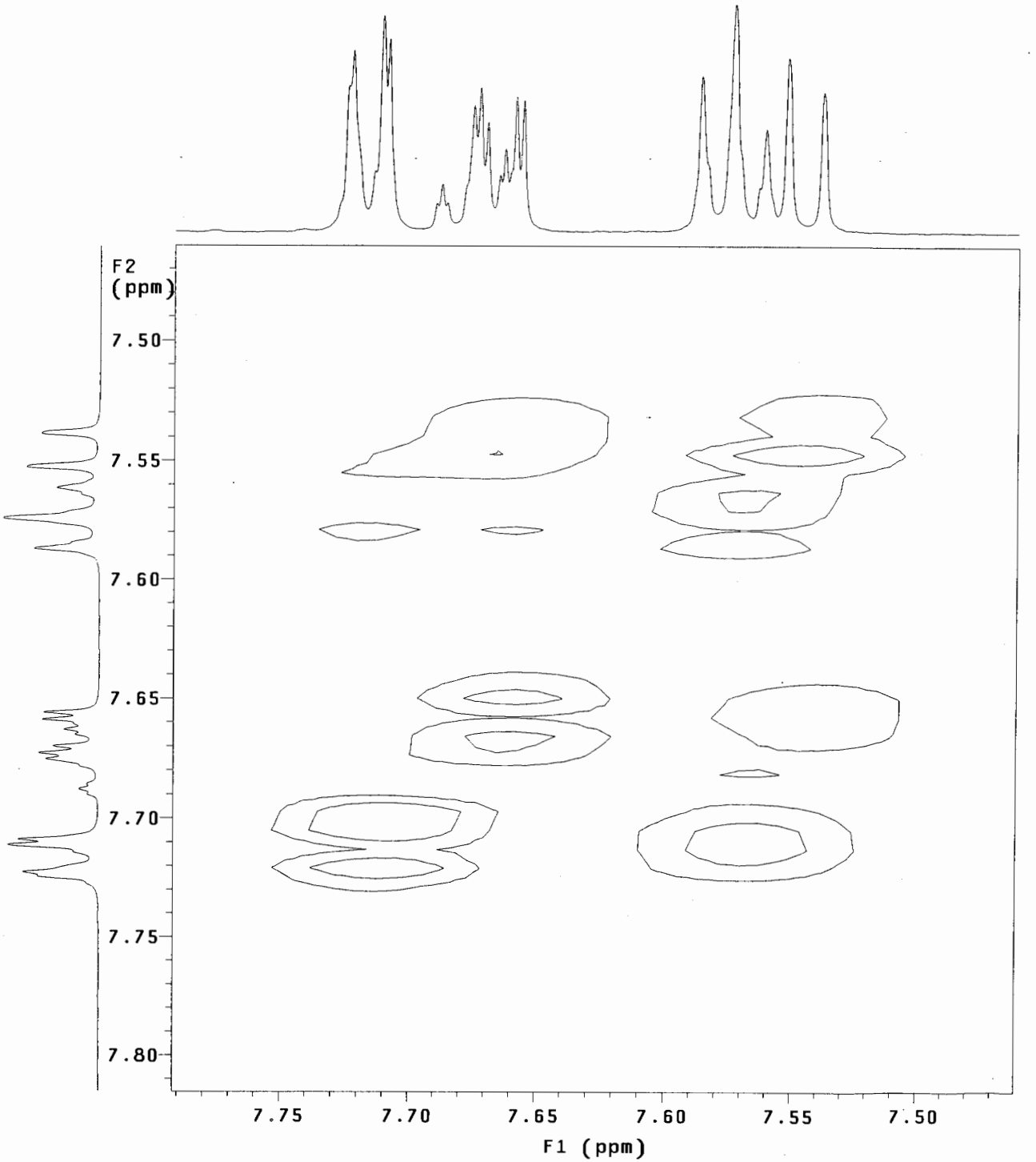
Pulse Sequence: gcosy

Solvent: DMSO
Ambient temperature
File: 2-258cosy
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Acq. time 0.212 sec
Width 1206.3 Hz
2D Width 1206.3 Hz
Single scan
50 increments
OBSERVE H1, 599.8751443 MHz
DATA PROCESSING
Sine bell 0.106 sec
F1 DATA PROCESSING
Sine bell 0.021 sec
FT size 512 x 512
Total time 1 min, 4 sec



28

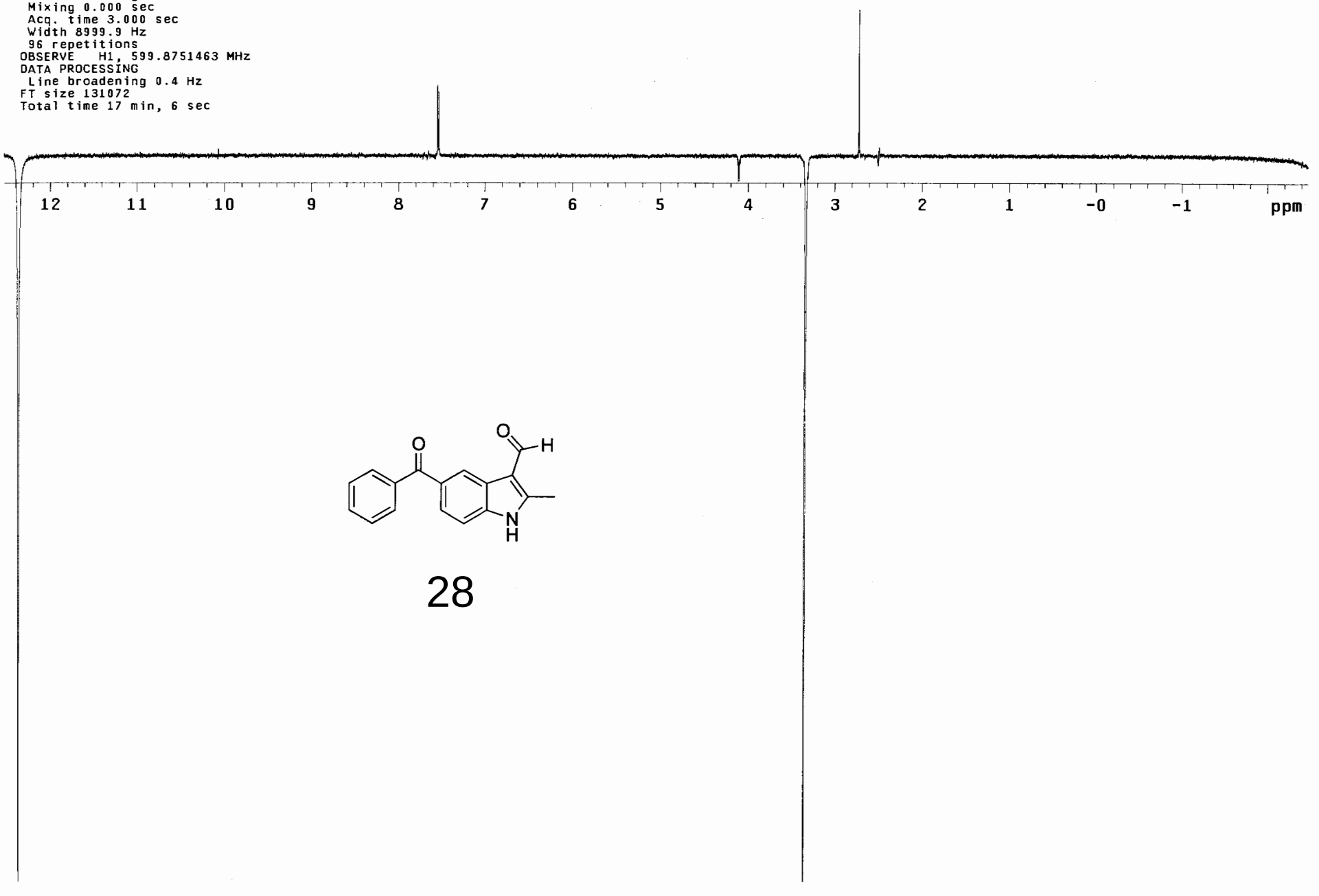


STANDARD PROTON PARAMETERS

Pulse Sequence: cyclenoe

Solvent: DMSO
Ambient temperature
File: 2-25bNOEnitrogen
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Pulse 90.0 degrees
Mixing 0.000 sec
Acq. time 3.000 sec
Width 8999.9 Hz
96 repetitions
OBSERVE H1, 599.8751463 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 17 min, 6 sec



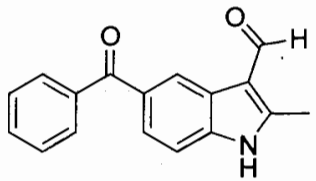
28

STANDARD PROTON PARAMETERS

Pulse Sequence: cyclenoe

Solvent: DMSO
Ambient temperature
File: 2-25bNOEch
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Pulse 90.0 degrees
Mixing 0.000 sec
Acq. time 3.000 sec
Width 8999.9 Hz
128 repetitions
OBSERVE H1, 599.8751464 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 17 min, 6 sec



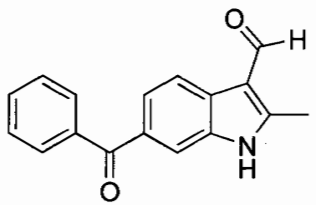
28

STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Temp. 20.0 C / 293.1 K
File: 2-25A
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8999.9 Hz
16 repetitions
OBSERVE H1, 599.8751444 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec

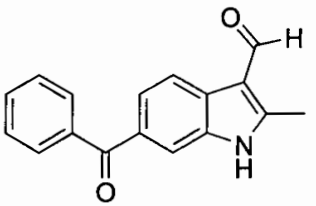


29

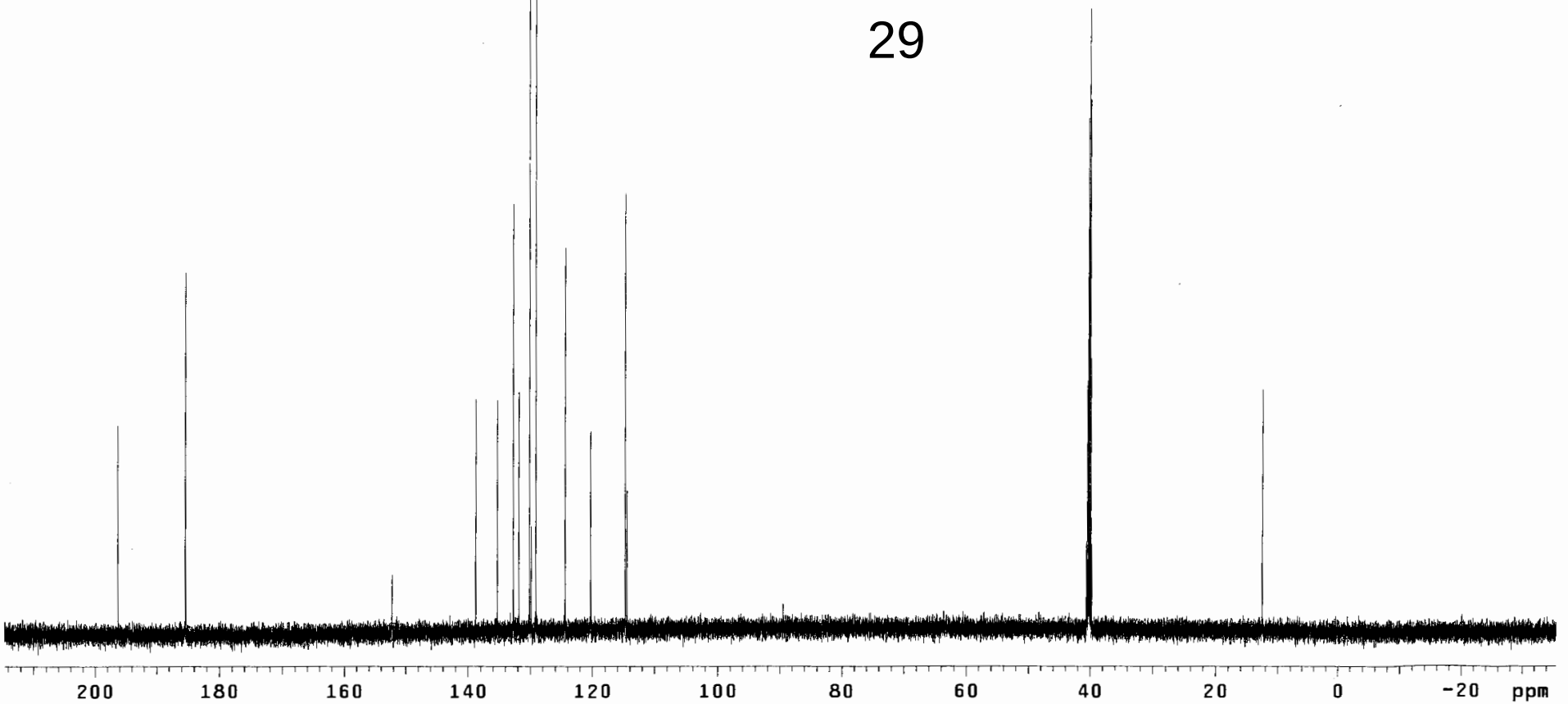
STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
User: 1-14-87
File: 2-21a_C13_6_6_2011
INOVA-600 "inova-wkst"

Pulse 46.4 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
10000 repetitions
OBSERVE C13, 150.8386940 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 2 hr, 48 min, 9 sec



29

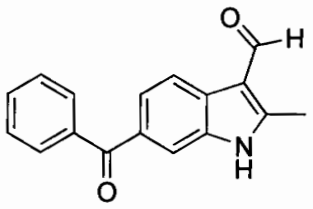


STANDARD PROTON PARAMETERS

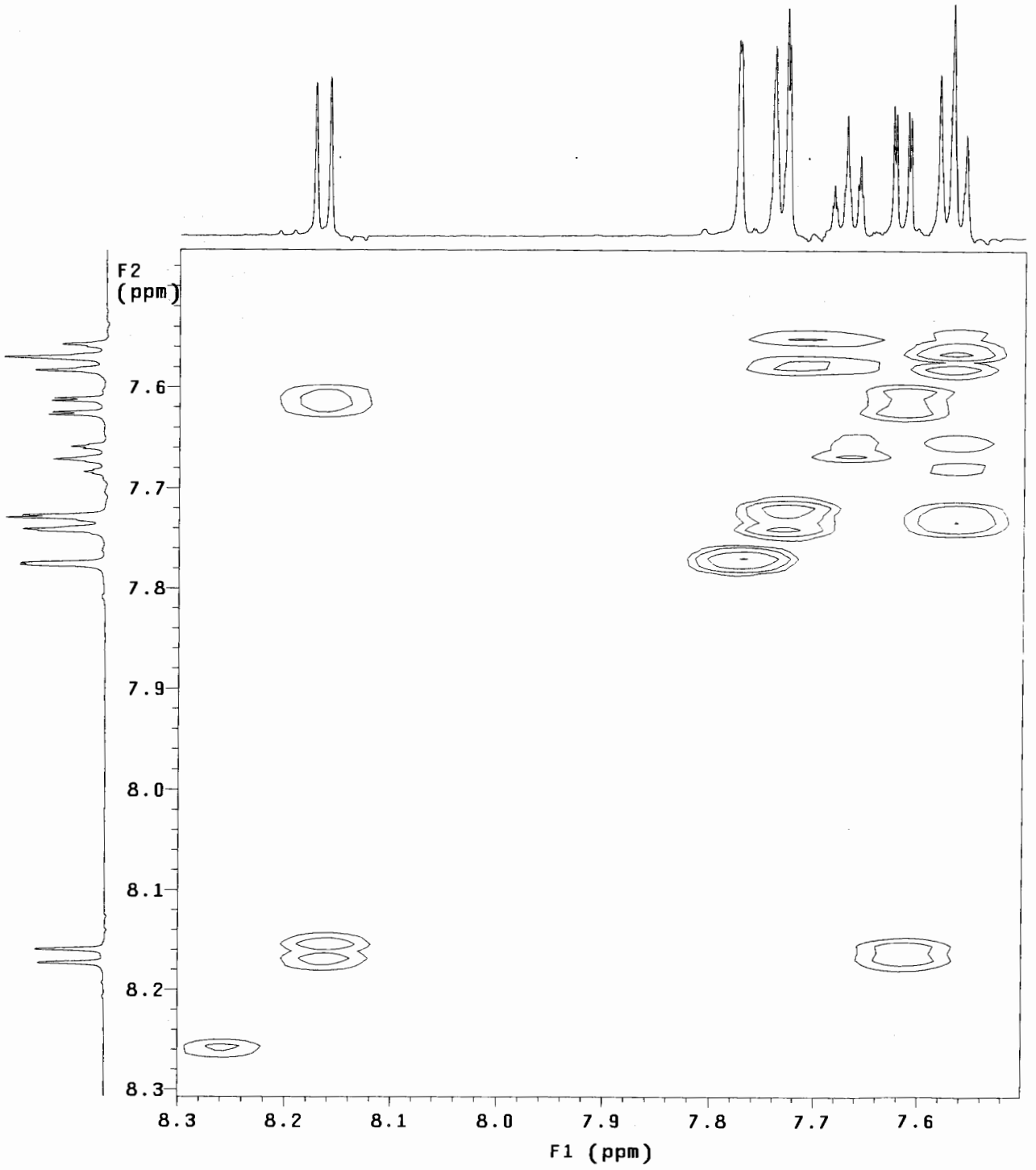
Pulse Sequence: gcosy

Solvent: DMSO
Temp. 20.0 C / 293.1 K
File: 2-25Acosy
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Acq. time 0.230 sec
Width 1111.7 Hz
2D Width 1111.7 Hz
Single scan
46 increments
OBSERVE H1, 599.8751444 MHz
DATA PROCESSING
Sine bell 0.115 sec
F1 DATA PROCESSING
Sine bell 0.021 sec
FT size 512 x 512
Total time 1 min, 0 sec



29

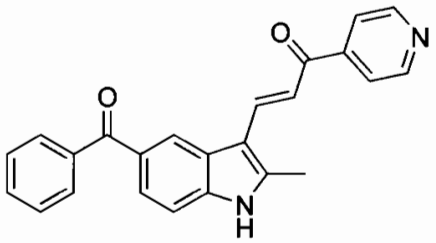


STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
File: 2-33.12-20-2010
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8999.9 Hz
16 repetitions
OBSERVE H1, 599.8751451 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



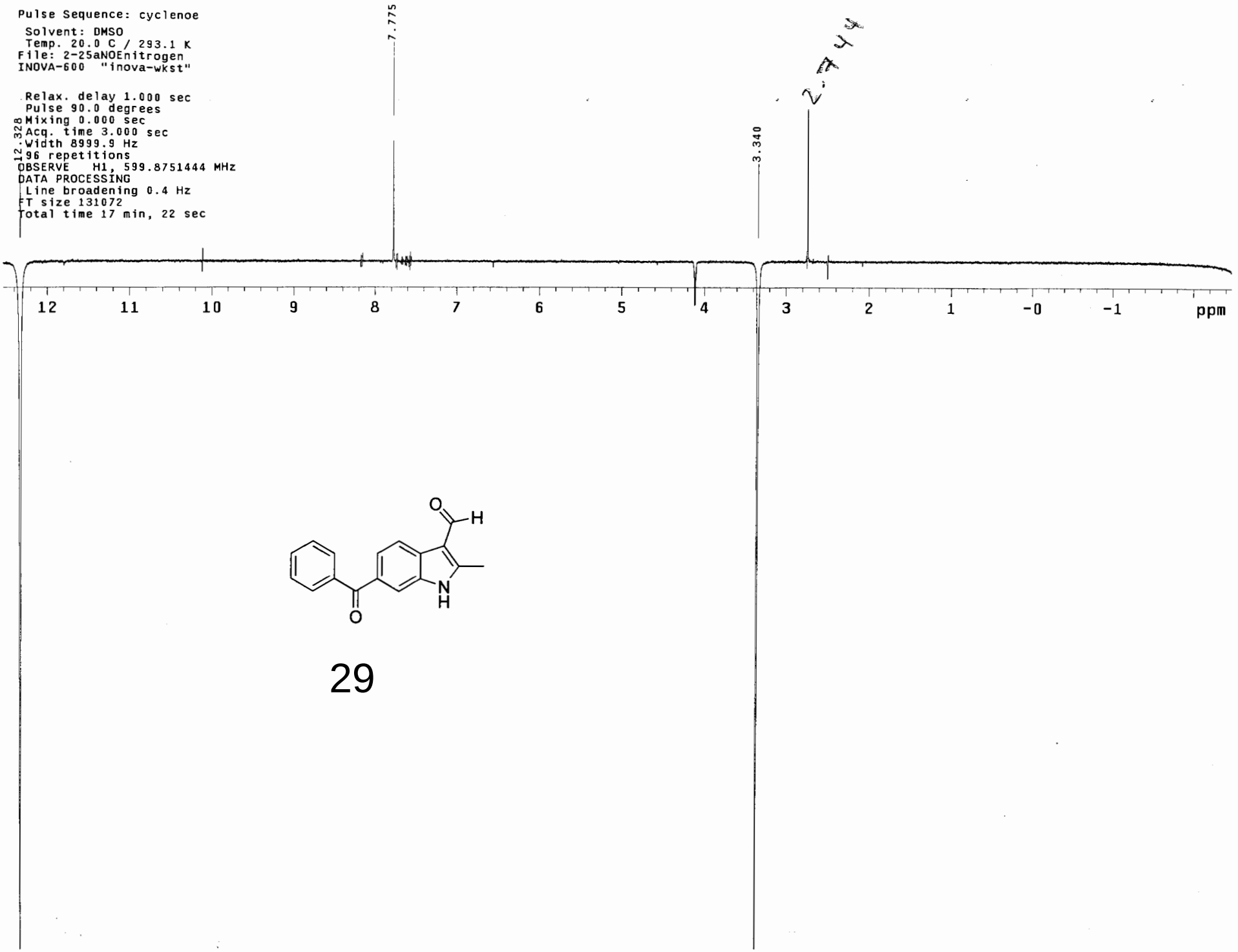
30

STANDARD PROTON PARAMETERS

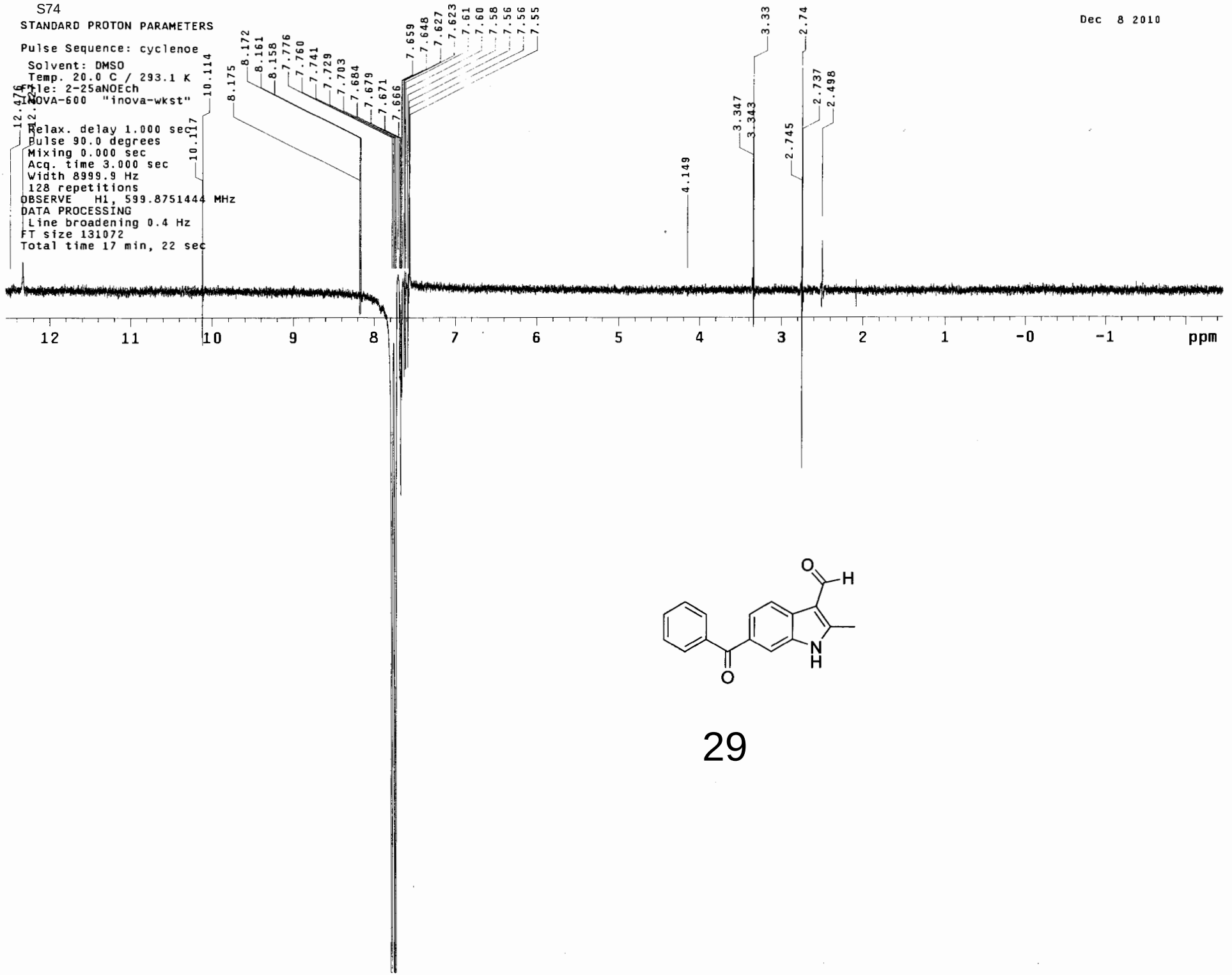
Pulse Sequence: cyclenoe
Solvent: DMSO
Temp. 20.0 C / 293.1 K
File: 2-25aNOEnitrogen
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Pulse 90.0 degrees
Mixing 0.000 sec
Acq. time 3.000 sec
Width 8999.9 Hz
96 repetitions
OBSERVE H1, 599.8751444 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 17 min, 22 sec

Dec 8 2010



29


STANDARD PROTON PARAMETERS
Pulse Sequence: cyclenoe
Solvent: DMSO
Temp. 20.0 C / 293.1 K
File: 2-25aNOEch
INOVA-600 "inova-wkst"
Relax. delay 1.000 sec
Pulse 90.0 degrees
Mixing 0.000 sec
Acq. time 3.000 sec
Width 8999.9 Hz
128 repetitions
OBSERVE H1, 599.8751444 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 17 min, 22 sec
Dec 8 2010
12.476
10.114
10.117
8.175
8.172
8.161
8.158
7.776
7.760
7.741
7.729
7.703
7.684
7.679
7.671
7.666
7.659
7.648
7.627
7.623
7.61
7.60
7.58
7.56
7.56
7.55
4.149
3.347
3.343
3.33
2.745
2.74
2.737
2.498
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm

O
H
N
H
O

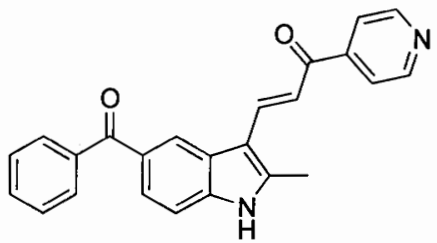
29

STANDARD PROTON PARAMETERS

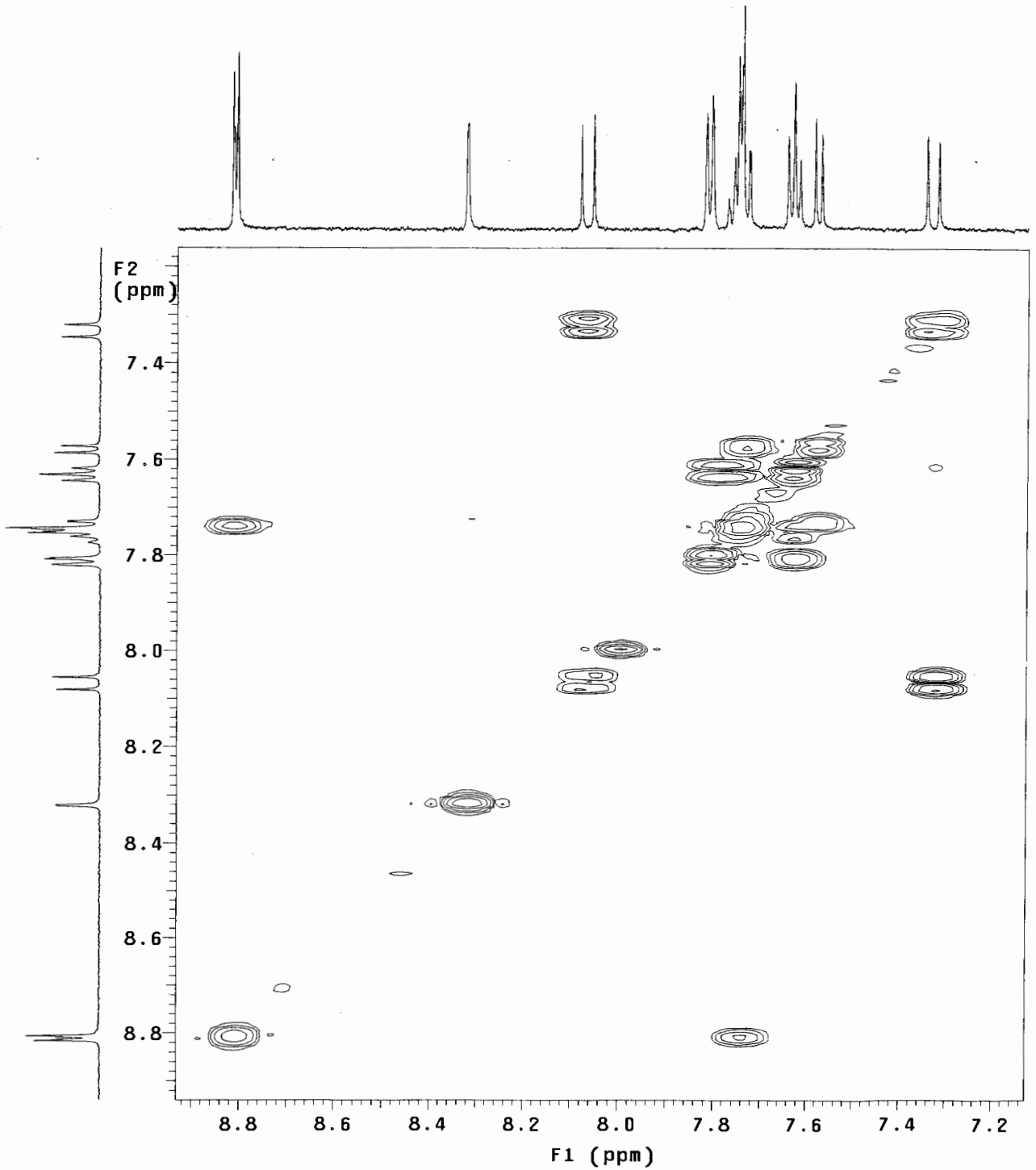
Pulse Sequence: gcosy

Solvent: DMSO
Temp. 26.0 C / 299.1 K
File: 2-33_cosy_4_29_2011
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Acq. time 0.197 sec
Width 1301.4 Hz
2D Width 1301.4 Hz
Single scan
54 increments
OBSERVE H1, 599.8751452 MHz
DATA PROCESSING
Sine bell 0.098 sec
F1 DATA PROCESSING
Sine bell 0.021 sec
FT size 512 x 512
Total time 1 min, 8 sec



30



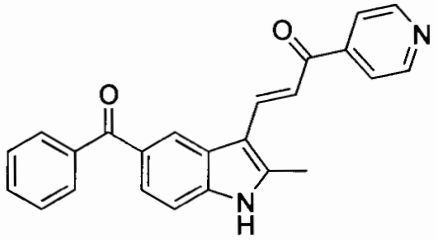
STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Temp. 23.0 C / 296.1 K
User: 1-14-87
File: 2-27b_13C_6_12_2011
INOVA-600 "inova600"

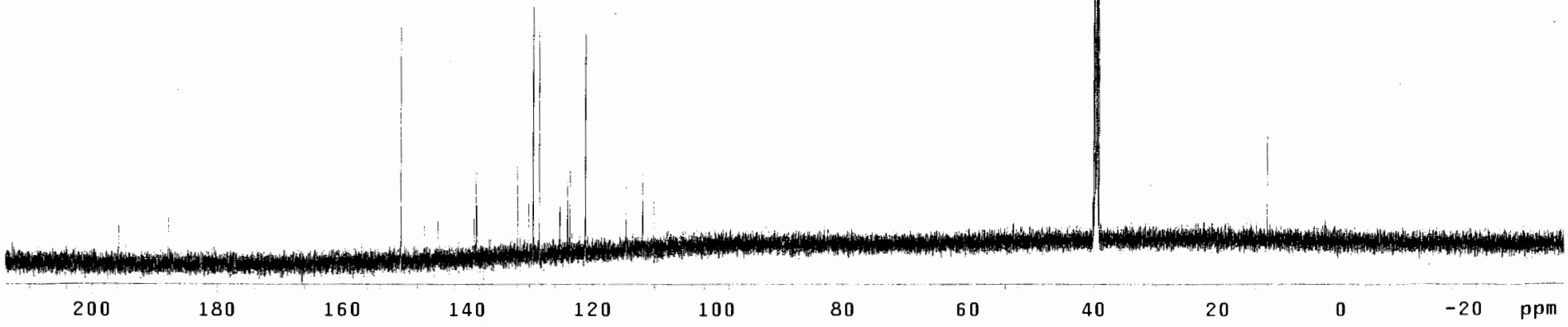
Pulse 46.4 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
50336 repetitions
OBSERVE C13, 150.8387978 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 28 hr, 1 min, 31 sec

Jun 12 2011



30

39.788
39.669
39.649
39.530
39.510
39.371
39.231

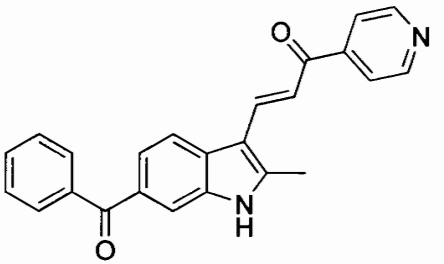


STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
File: 2-29h1
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8999.9 Hz
16 repetitions
OBSERVE H1, 599.8751457 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



31

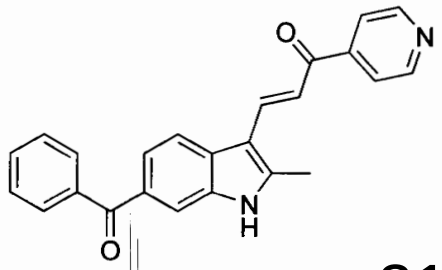
STANDARD CARBON PARAMETERS

Jun 12 2011

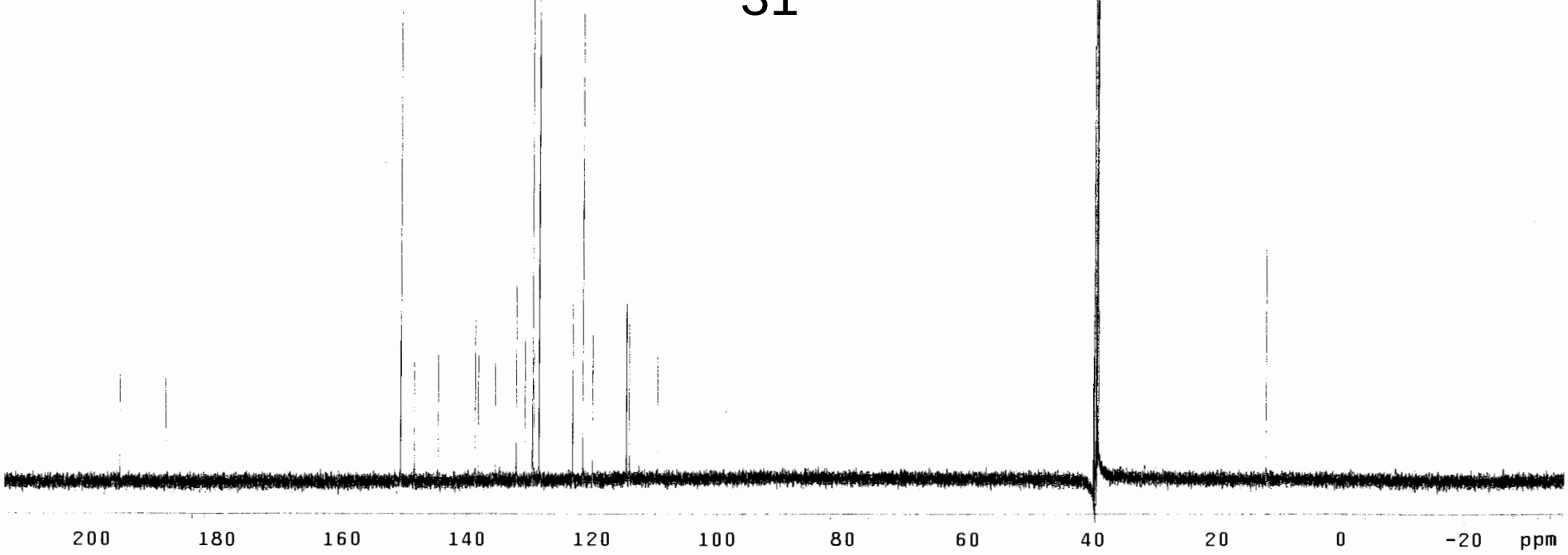
Pulse Sequence: s2pul

Solvent: DMSO
Temp. 23.0 C / 296.1 K
User: 1-14-87
INOVA-600 "inova600"

Pulse 46.4 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
2208 repetitions
OBSERVE C13, 150.8387986 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 2 hr, 48 min, 9 sec



31

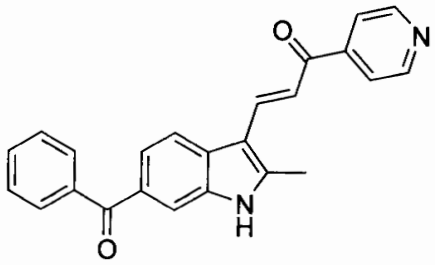


STANDARD PROTON PARAMETERS

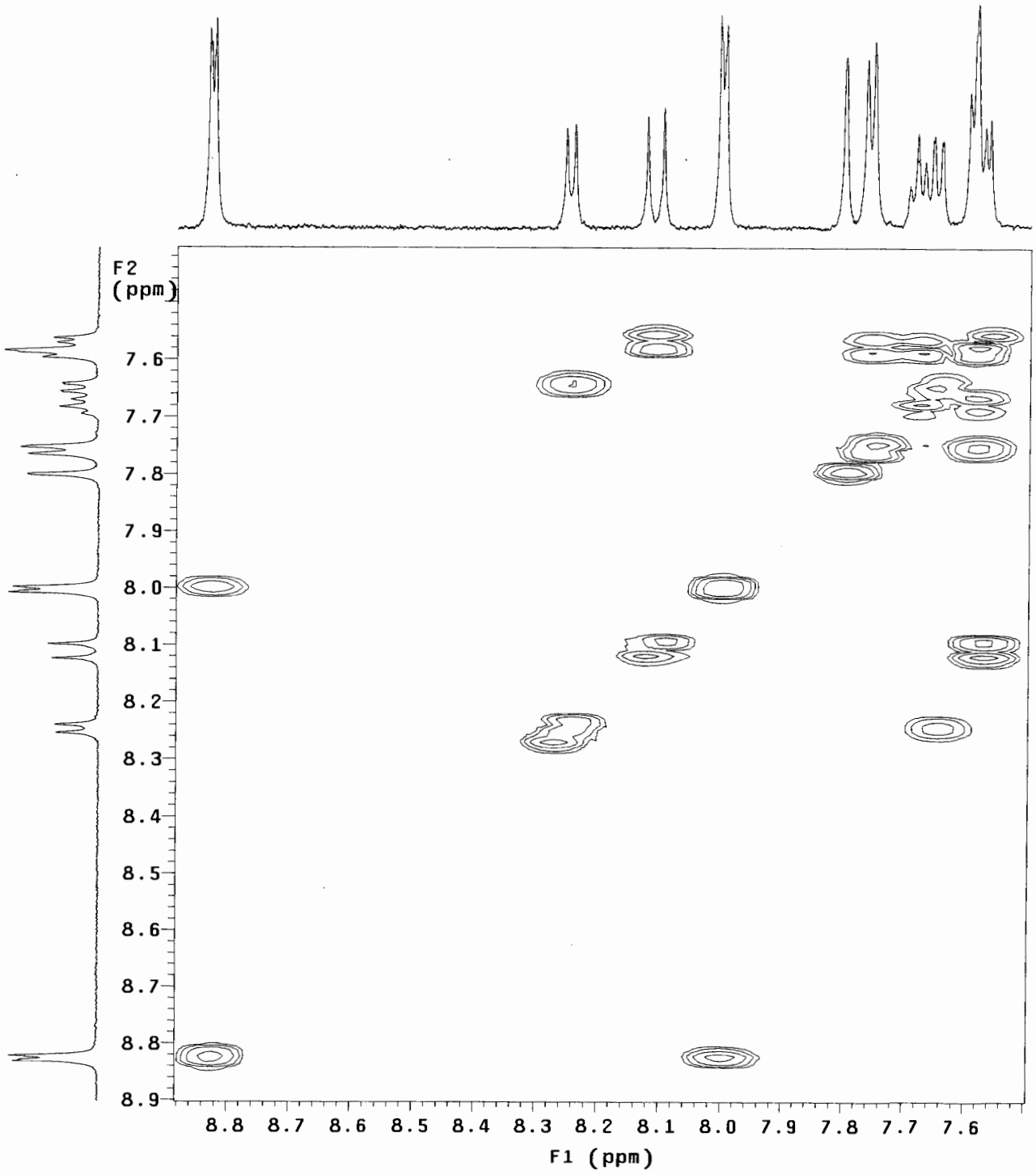
Pulse Sequence: gcosy

Solvent: DMSO
Ambient temperature
File: 2-29cosy
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Acq. time 0.167 sec
Width 1537.4 Hz
2D Width 1537.4 Hz
Single scan
64 increments
OBSERVE H1, 599.8751457 MHz
DATA PROCESSING
Sine bell 0.083 sec
F1 DATA PROCESSING
Sine bell 0.021 sec
FT size 512 x 512
Total time 1 min, 19 sec



31

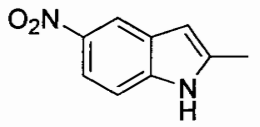


STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
File: 2_63_1h_3_14_2011
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8999.9 Hz
16 repetitions
OBSERVE H1, 599.8751436 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



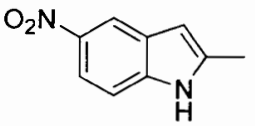
32

STANDARD CARBON PARAMETERS

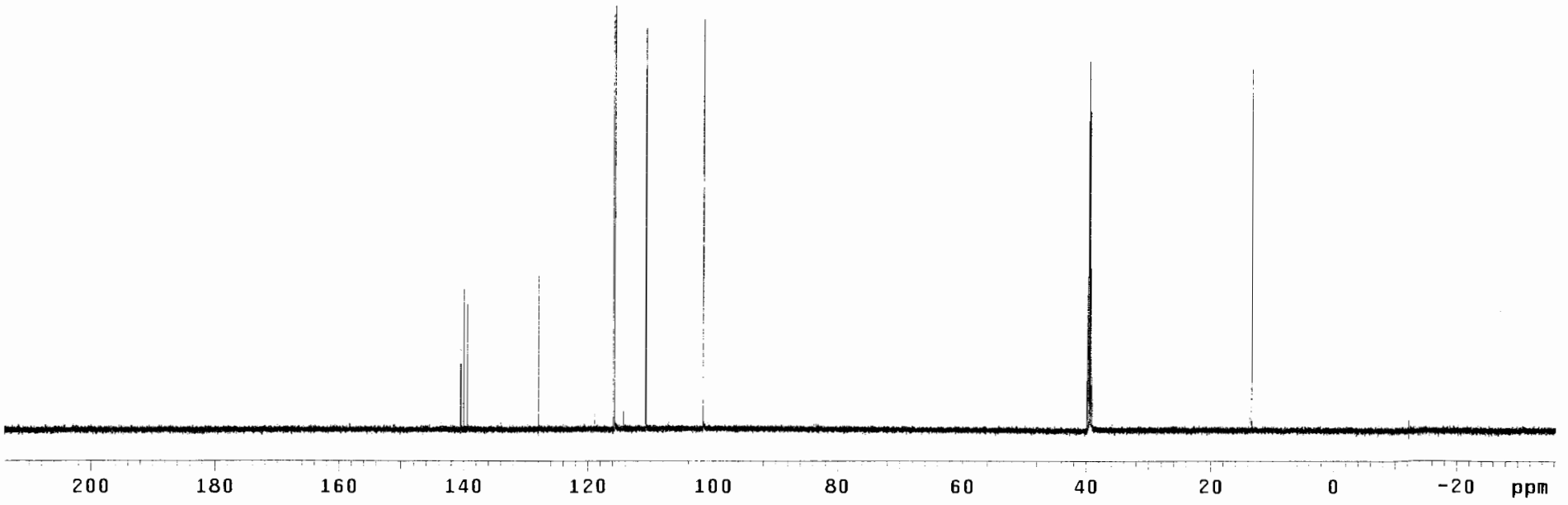
Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
User: 1-14-87
File: 2-63_13C_3_14_2011
INOVA-600 "inova600"

Pulse 46.4 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
5456 repetitions
OBSERVE C13, 150.8387910 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 28 hr, 1 min, 31 sec



32

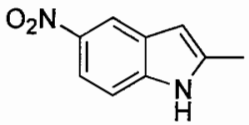


STANDARD PROTON PARAMETERS

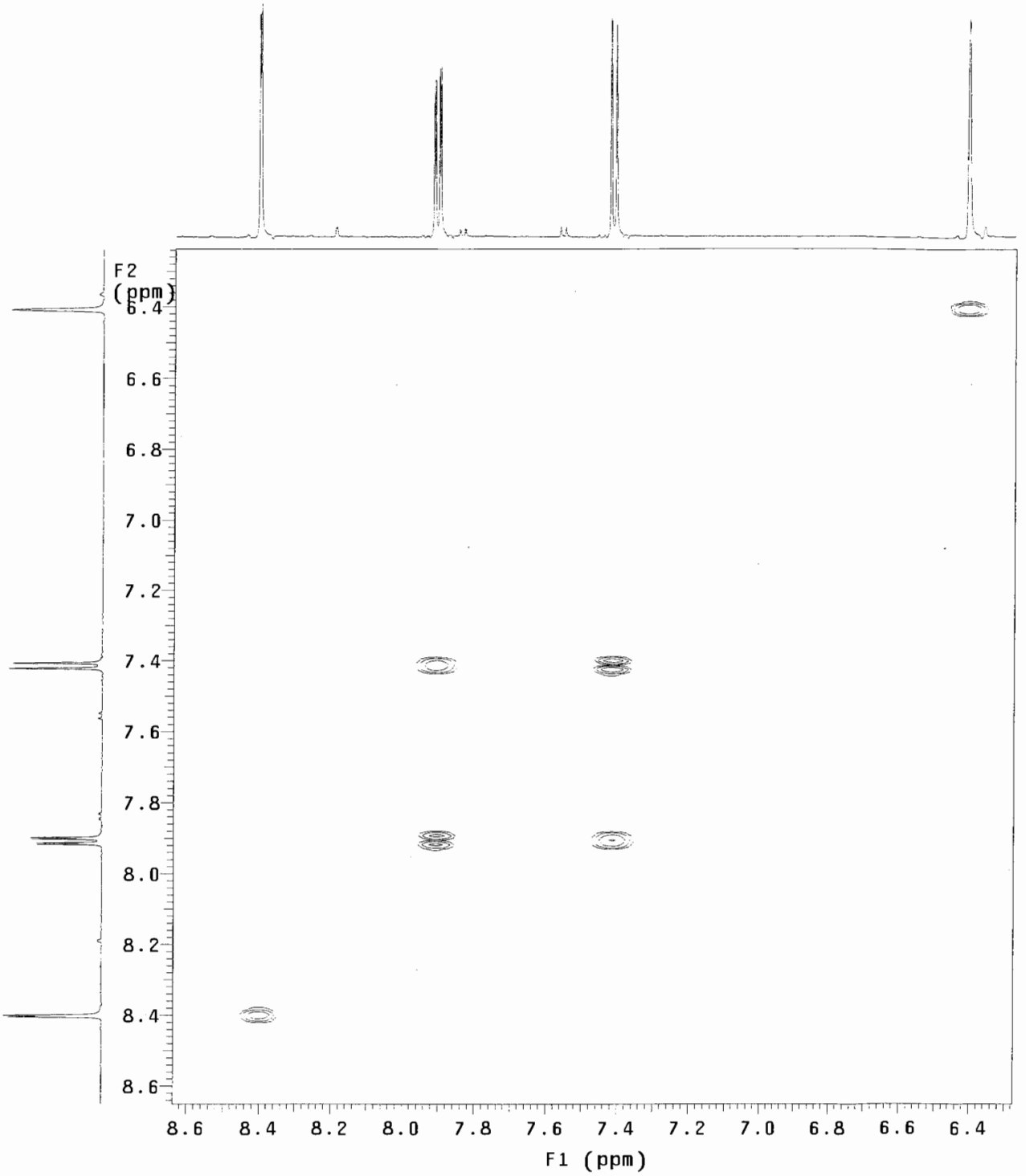
Pulse Sequence: gcosy

Solvent: DMSO
Ambient temperature
File: 2-63_cosy_3_14_2011
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.132 sec
Width 1939.5 Hz
2D Width 1939.5 Hz
Single scan
80 increments
OBSERVE H1, 599.8751436 MHz
DATA PROCESSING
Sine bell 0.066 sec
F1 DATA PROCESSING
Sine bell 0.021 sec
FT size 512 x 512
Total time 1 min, 35 sec



32

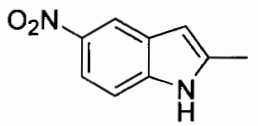


STANDARD PROTON PARAMETERS

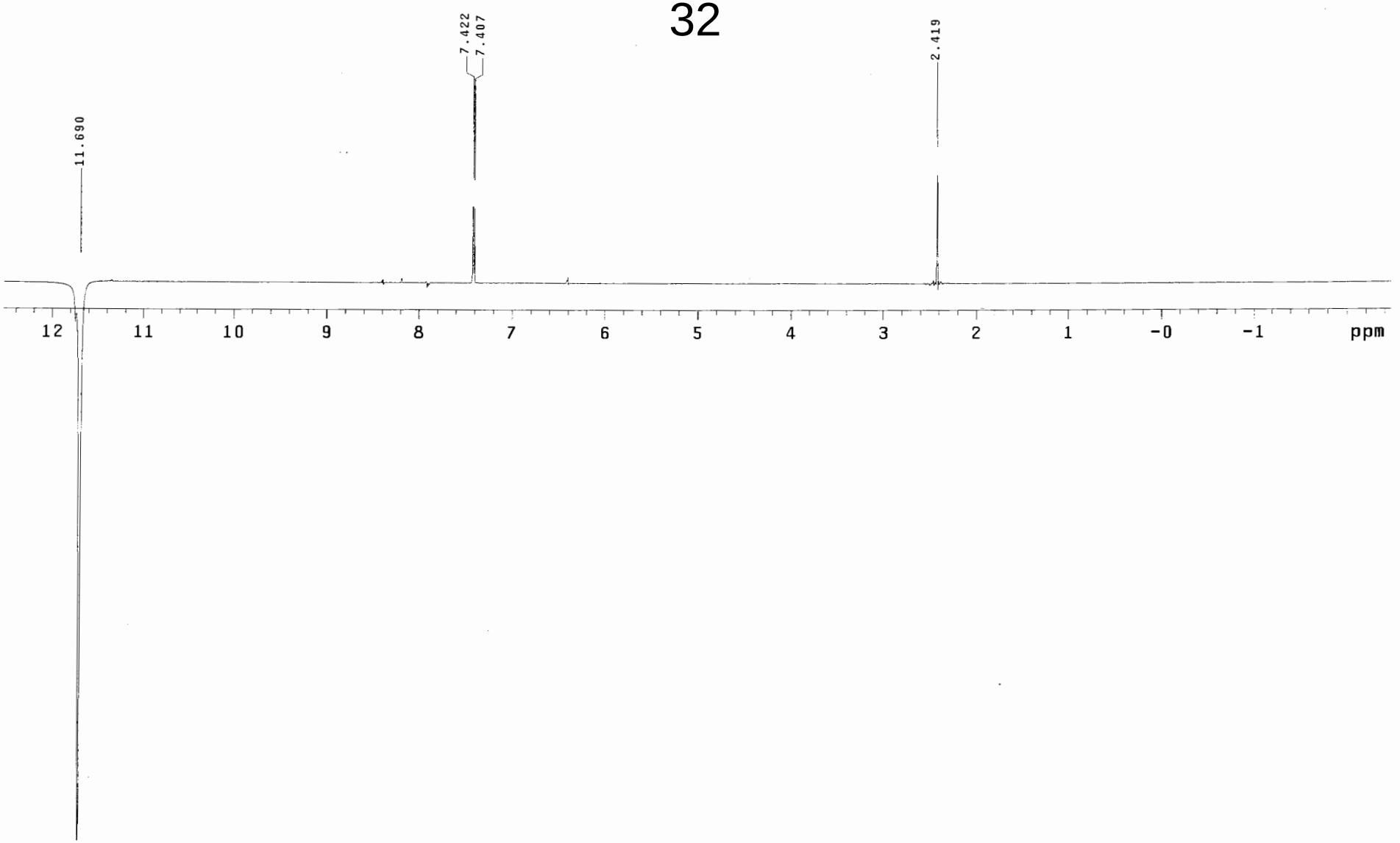
Mar 15 2011

Pulse Sequence: cyclenoe
Solvent: DMSO
Temp. 22.0 C / 295.1 K
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 90.0 degrees
Mixing 0.000 sec
Acq. time 1.000 sec
Width 9000.9 Hz
32 repetitions
OBSERVE H1, 599.8751438 MHz
DATA PROCESSING
Gauss apodization 0.200 sec
FT size 131072
Total time 3 min, 12 sec



32



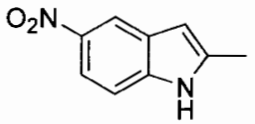
STANDARD PROTON PARAMETERS

Mar 15 2011

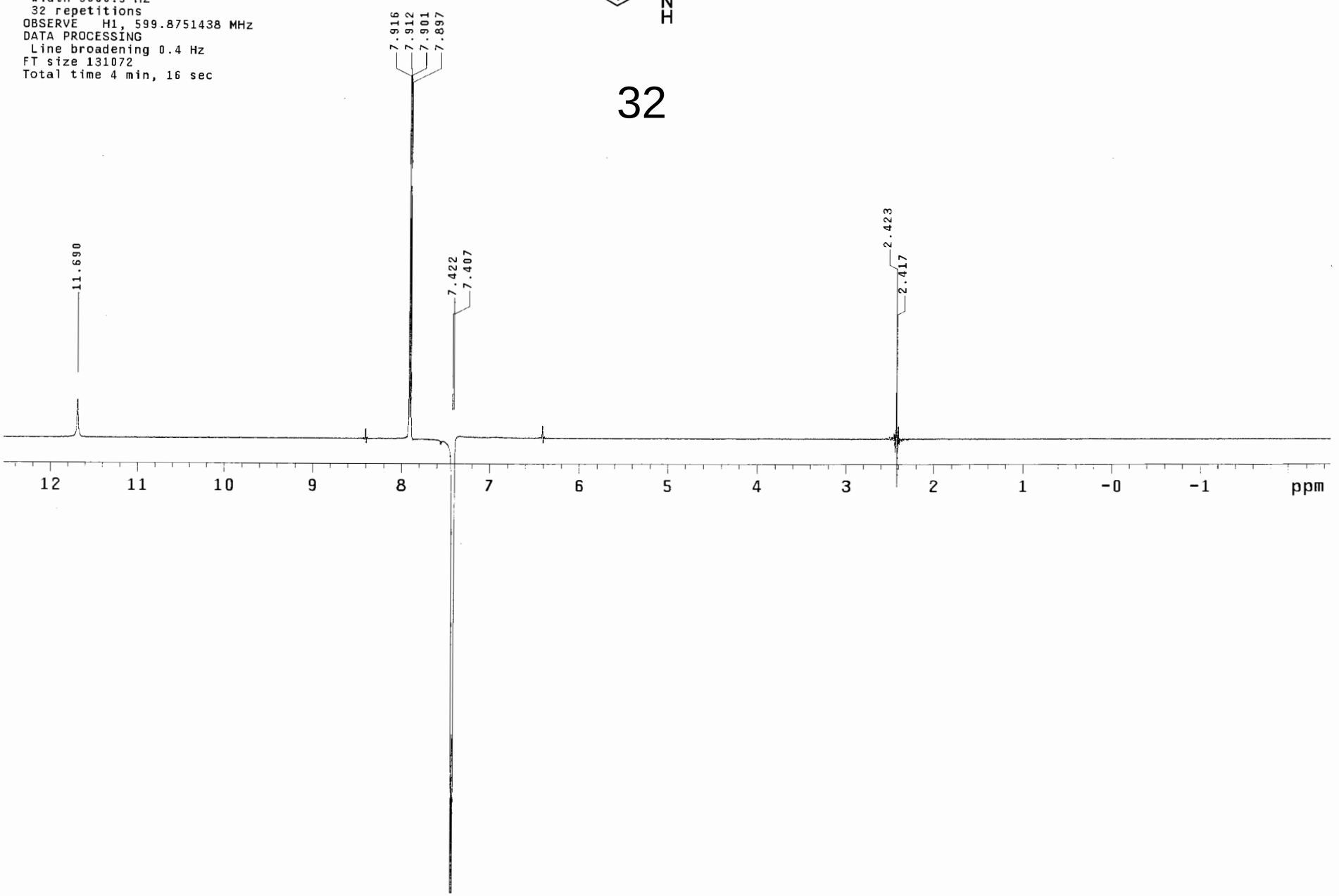
Pulse Sequence: cyclenoe

Solvent: DMSO
Temp. 22.0 C / 295.1 K
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 90.0 degrees
Mixing 0.000 sec
Acq. time 3.000 sec
Width 9000.9 Hz
32 repetitions
OBSERVE H1, 599.8751438 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 4 min, 16 sec



32

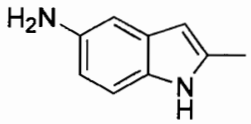


STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 9000.9 Hz
16 repetitions
OBSERVE H1, 599.8751436 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



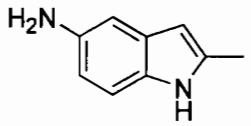
33

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
User: 1-14-87
INOVA-600 "inova600"

Pulse 46.4 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
992 repetitions
OBSERVE C13, 150.8387892 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 2 hr, 48 min, 9 sec



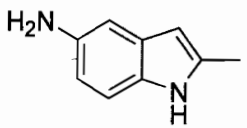
33

STANDARD PROTON PARAMETERS

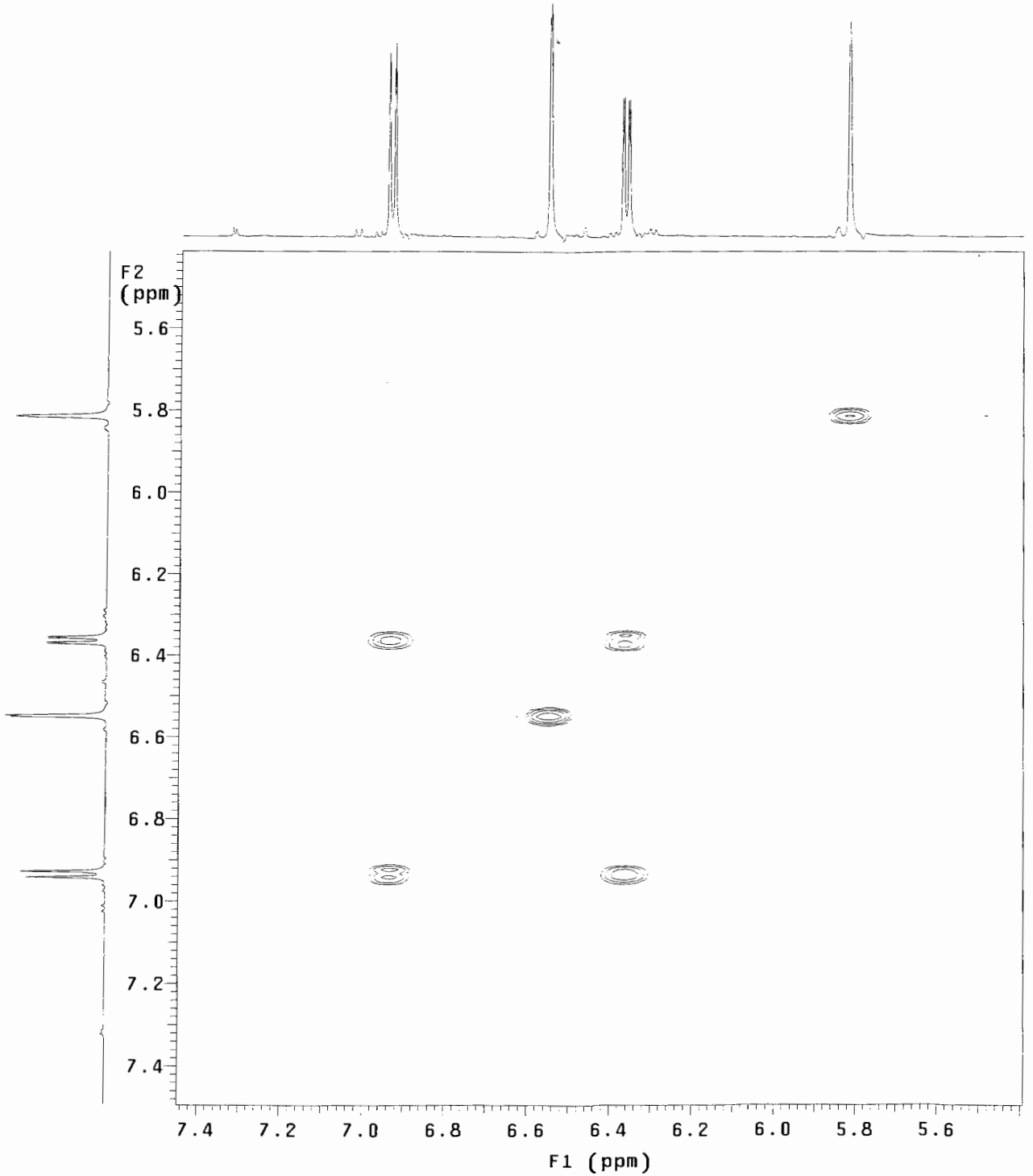
Pulse Sequence: gcosy

Solvent: DMSO
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.175 sec
Width 1466.9 Hz
2D Width 1466.9 Hz
Single scan
61 increments
OBSERVE H1, 599.8751436 MHz
DATA PROCESSING
Sine bell 0.087 sec
F1 DATA PROCESSING
Sine bell 0.021 sec
FT size 512 x 512
Total time 1 min, 16 sec



33

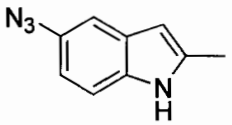


STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Temp. 25.0 C / 298.1 K
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 9000.9 Hz
16 repetitions
OBSERVE H1, 599.8751436 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



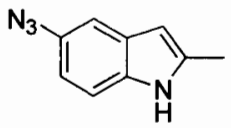
34

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: 2-87_13c
INOVA-600 "inova600"

Pulse 46.4 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
1824 repetitions
OBSERVE C13, 150.8387995 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 28 hr, 1 min, 31 sec



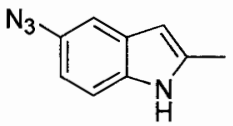
34

STANDARD PROTON PARAMETERS

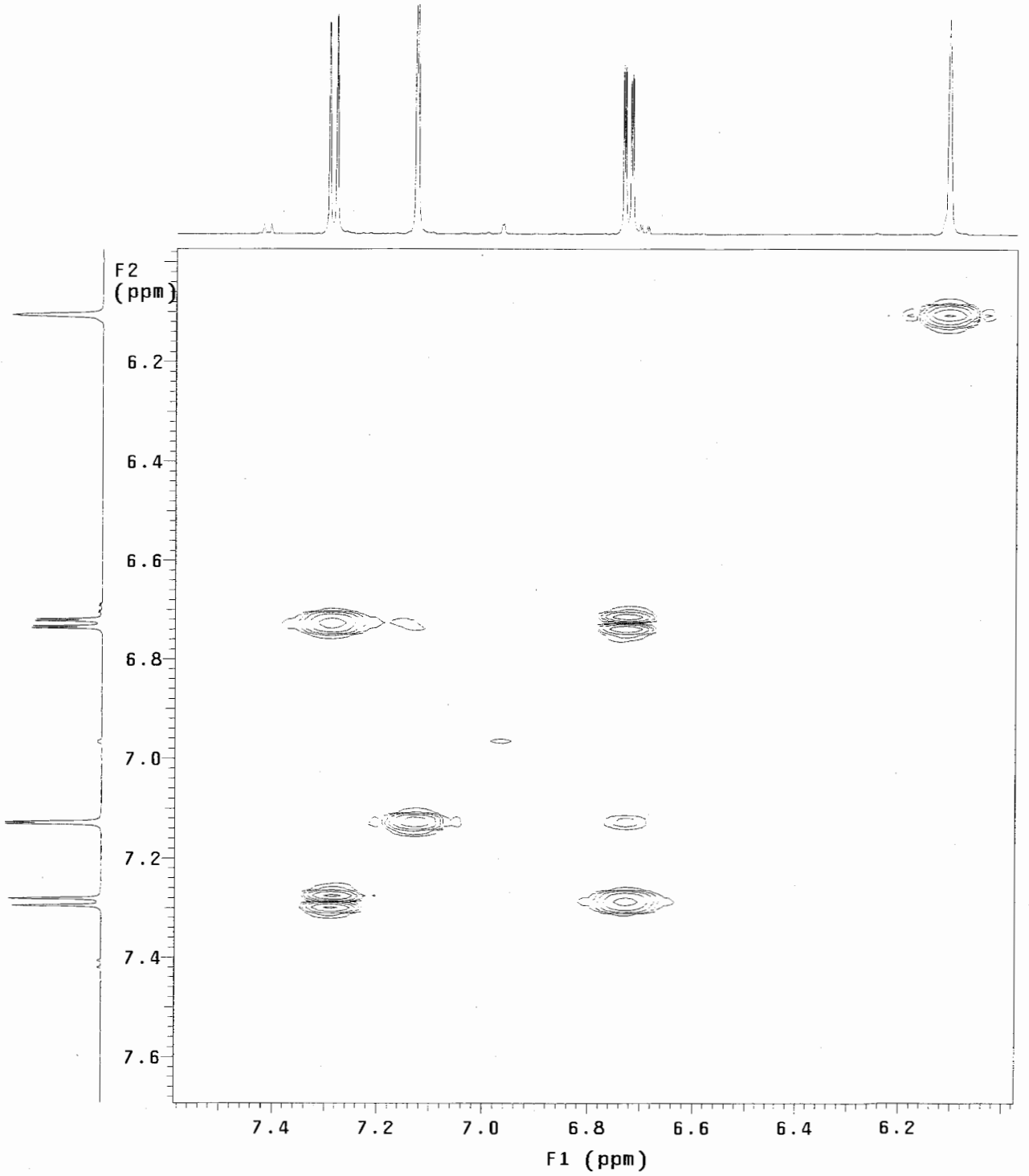
Pulse Sequence: gcosy

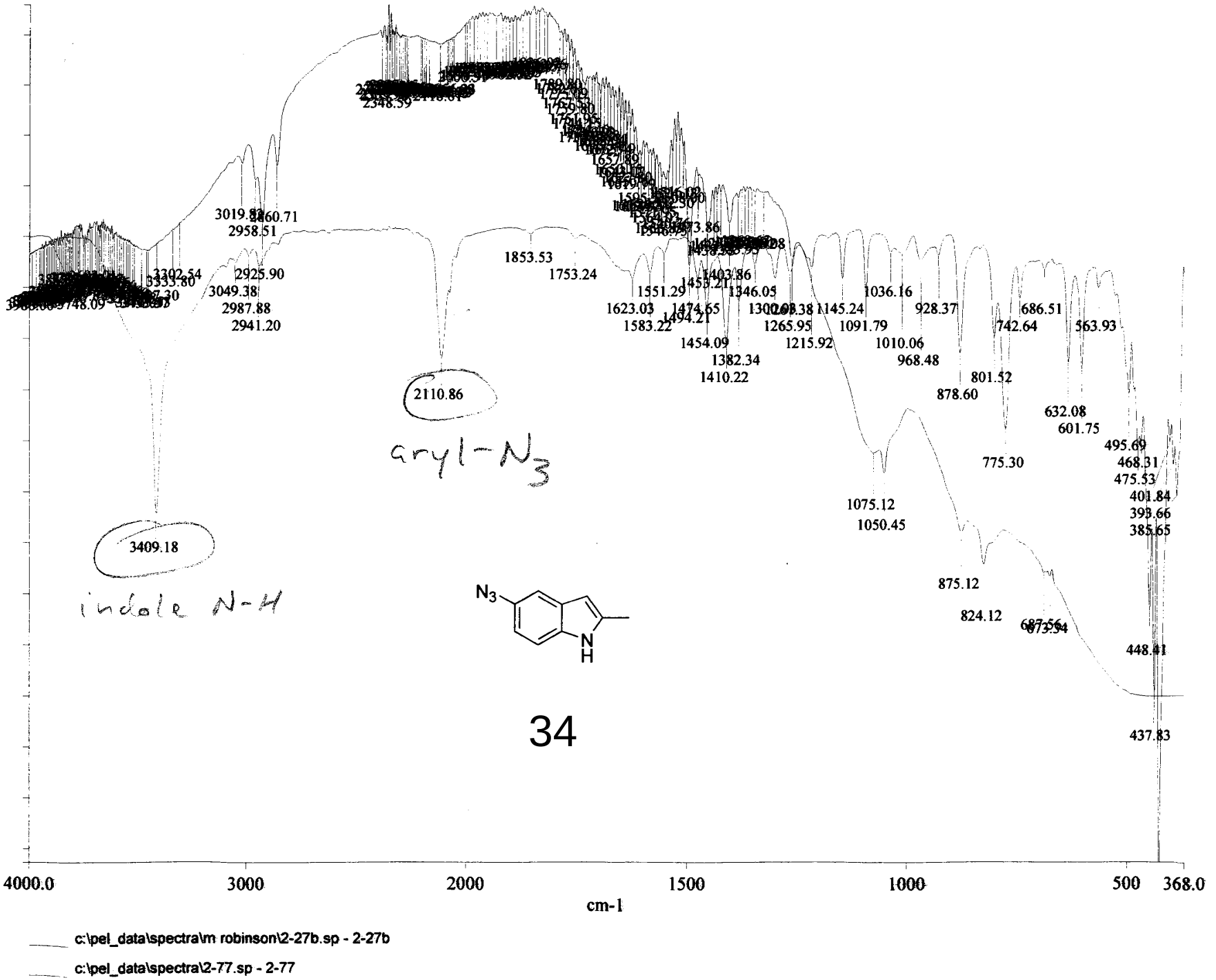
Solvent: DMSO
Temp. 25.0 C / 298.1 K
File: 2-87_cosy
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.140 sec
Width 1832.5 Hz
2D Width 1832.5 Hz
Single scan
76 increments
OBSERVE H1, 599.8751436 MHz
DATA PROCESSING
Sine bell 0.070 sec
F1 DATA PROCESSING
Sine bell 0.021 sec
FT size 512 x 512
Total time 1 min, 31 sec



34



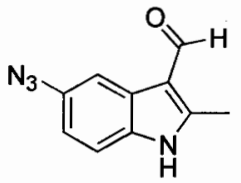

3409.18
indole N-H
2110.86
aryl-N3
3019.82
2960.71
2958.51
3392.54
3333.80
2925.90
3049.38
2987.88
2941.20
1853.53
1753.24
1623.03
1583.22
1551.29
1474.65
1494.21
1454.09
1410.22
1403.86
1346.05
1265.95
1215.92
1145.24
1091.79
1036.16
1010.06
968.48
928.37
878.60
801.52
775.30
742.64
686.51
632.08
601.75
563.93
495.69
468.31
475.53
401.84
393.66
385.65
1075.12
1050.45
875.12
824.12
687.56
673.54
448.41
437.83
N3
N
H
34
4000.0
3000
2000
1500
1000
500
368.0
cm-1
c:\pel_data\spectra\m robinson\2-27b.sp - 2-27b
c:\pel_data\spectra\2-77.sp - 2-77

STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Temp. 24.0 C / 297.1 K
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 9000.9 Hz
16 repetitions
OBSERVE H1, 599.8751437 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec

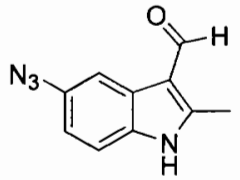


35

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul
Solvent: DMSO
Temp. 24.0 C / 297.1 K
User: 1-14-87
File: 2-89_c13
INOVA-600 "inova600"

Pulse 46.4 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
2752 repetitions
OBSERVE C13, 150.8387985 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 2 hr, 48 min, 9 sec



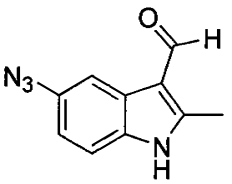
35

STANDARD PROTON PARAMETERS

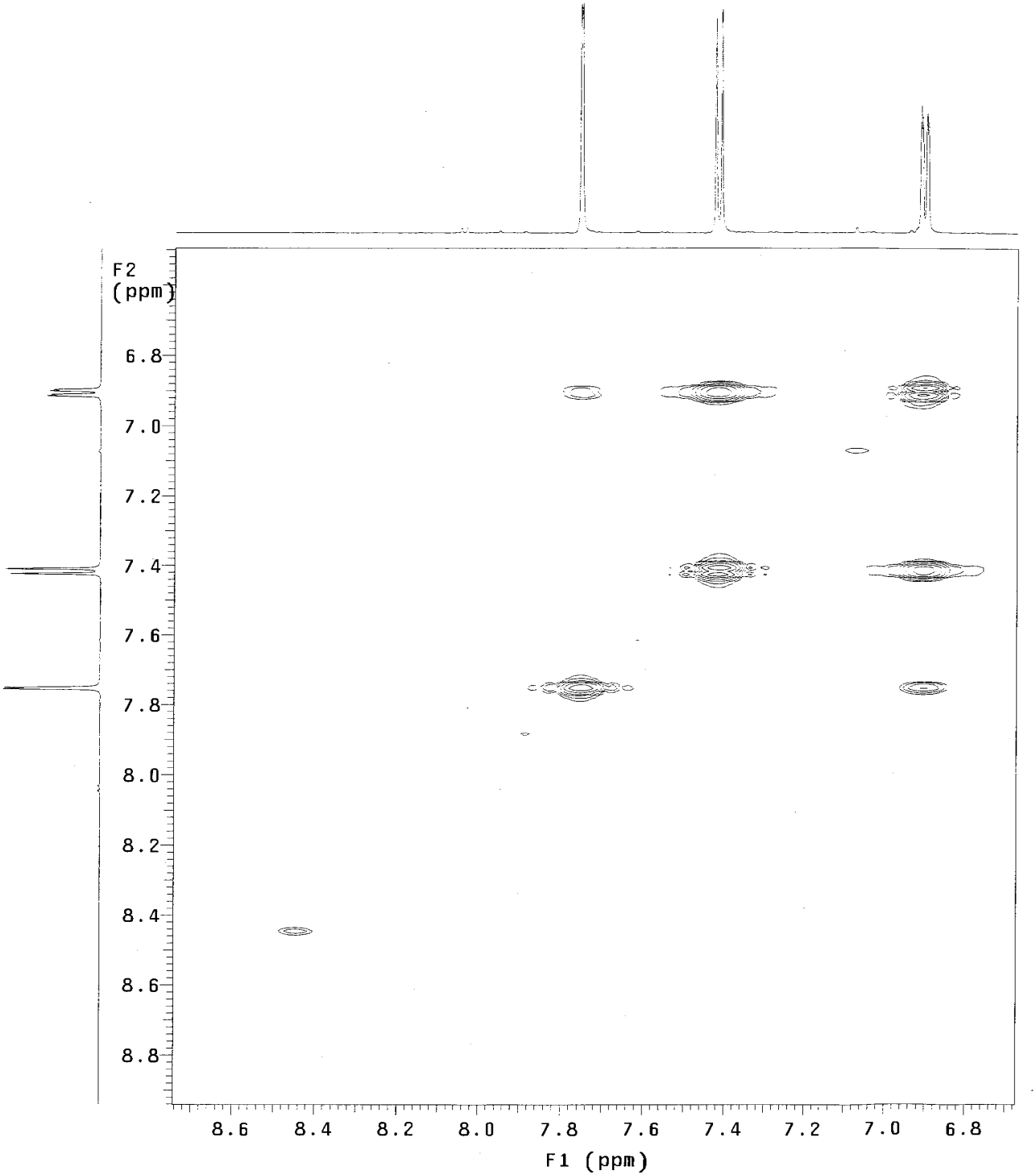
Pulse Sequence: gcosy

Solvent: DMSO
Temp. 24.0 C / 297.1 K
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.168 sec
Width 3040.0 Hz
2D Width 3040.0 Hz
Single scan
126 increments
OBSERVE H1, 599.8751437 MHz
DATA PROCESSING
Sine bell 0.084 sec
F1 DATA PROCESSING
Sine bell 0.021 sec
FT size 1024 x 1024
Total time 2 min, 33 sec



35

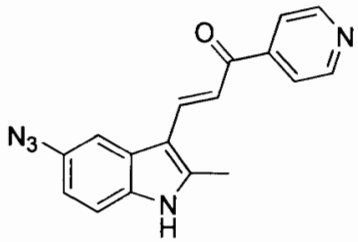


STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
File: 2-85_h1_dmso
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8999.9 Hz
16 repetitions
OBSERVE H1, 599.8751432 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



Mar 4 2011

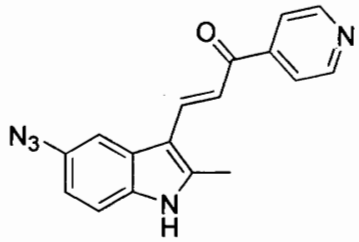
36

STANDARD CARBON PARAMETERS

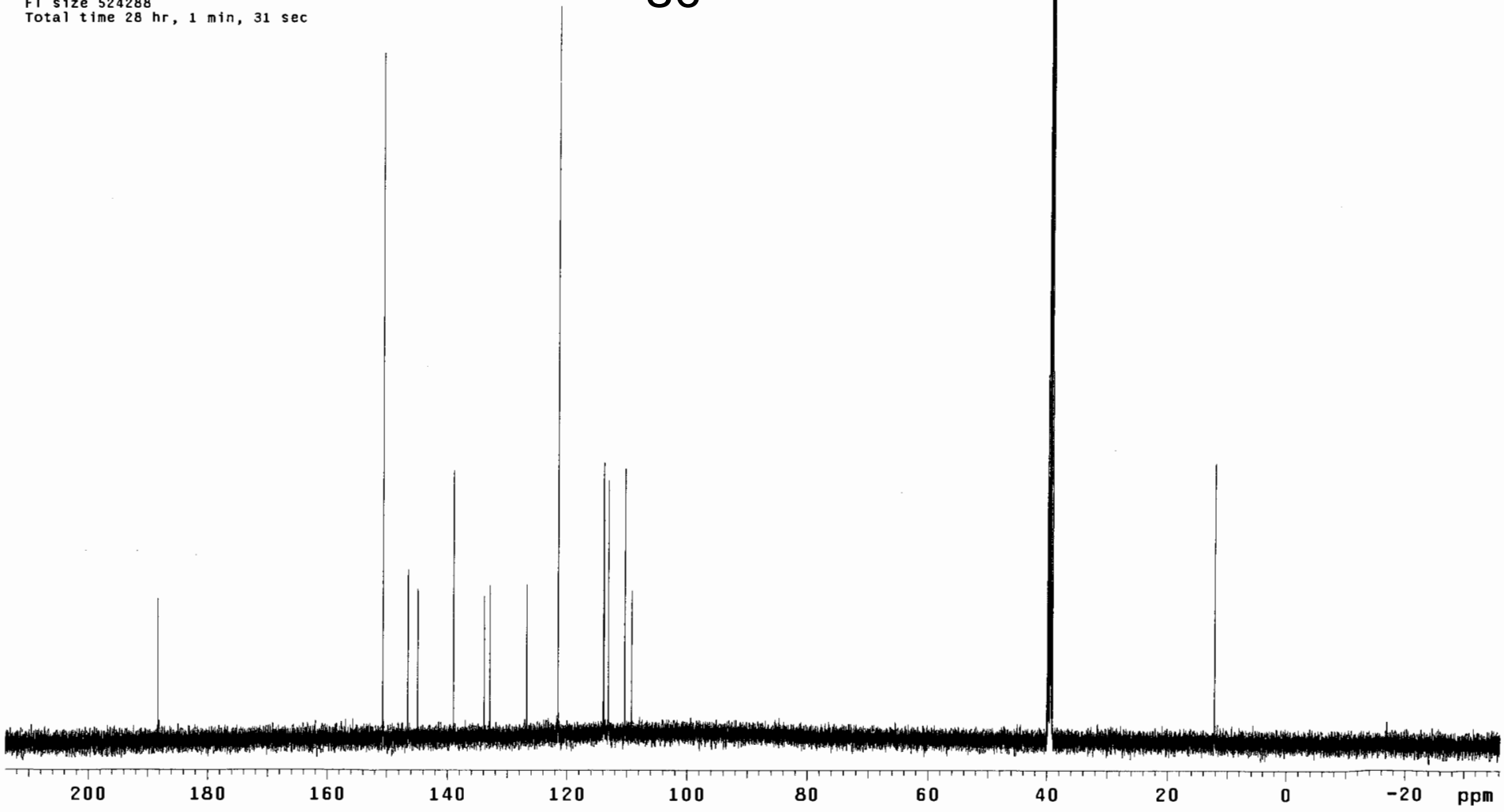
Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
User: 1-14-87
File: 2-85_c13_dmso
INOVA-600 "inova-wkst"

Pulse 46.4 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
24400 repetitions
OBSERVE C13, 150.8387904 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 28 hr, 1 min, 31 sec



36

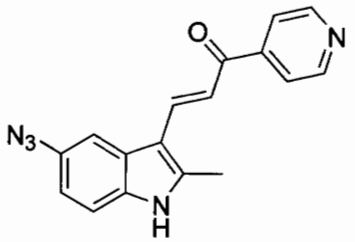


STANDARD PROTON PARAMETERS

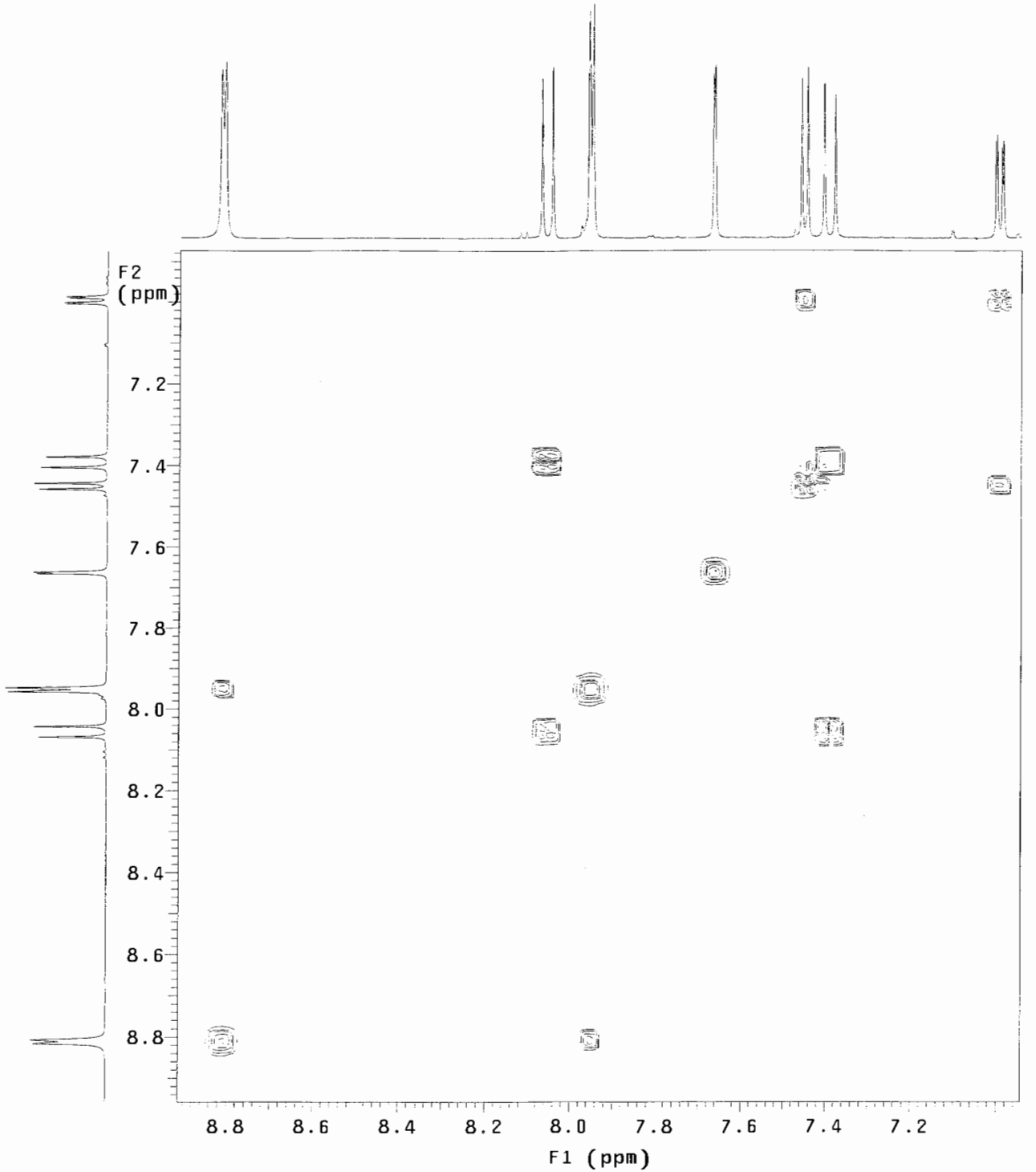
Pulse Sequence: gcosy

Solvent: DMSO
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.142 sec
Width 1797.6 Hz
2D Width 1797.6 Hz
Single scan
74 increments
OBSERVE H1, 599.8751433 MHz
DATA PROCESSING
Sine bell 0.071 sec
F1 DATA PROCESSING
Sine bell 0.021 sec
FT size 512 x 512
Total time 1 min, 29 sec



36

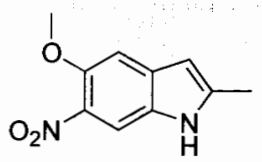


STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
File: 2-109_A
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.8723061 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



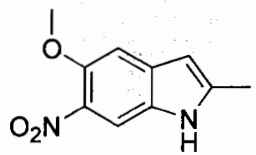
37

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 2-109_A_C13
INOVA-600 "inova-wkst"

Pulse 46.4 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
1760 repetitions
OBSERVE C13, 150.8379929 MHz
DECOUPLE H1, 599.8753144 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 2 hr, 48 min, 9 sec



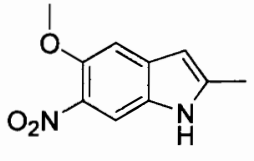
37

STANDARD PROTON PARAMETERS

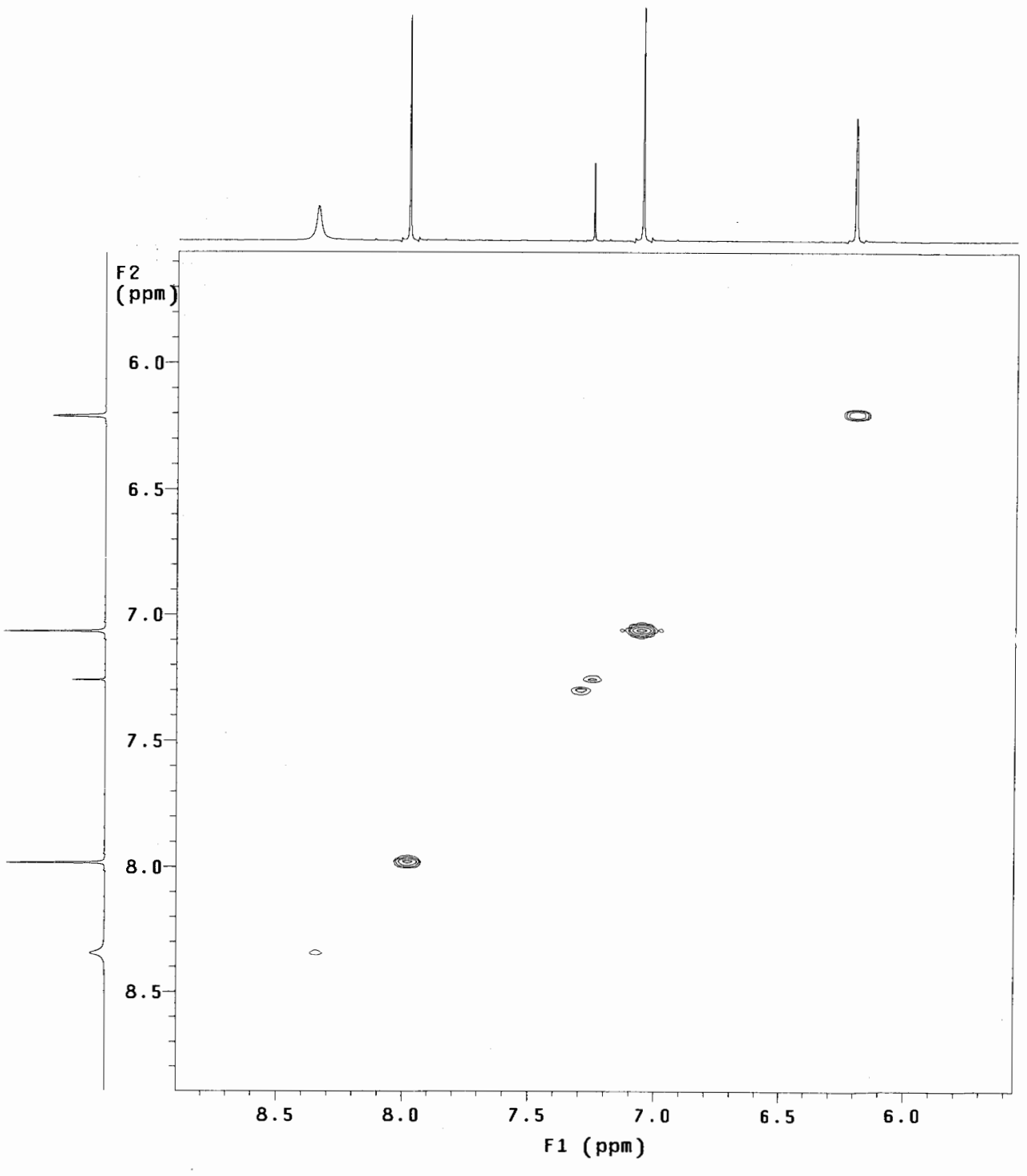
Pulse Sequence: gcosy

Solvent: CDCl3
Ambient temperature
File: 2-109_A_cosy
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Acq. time 0.128 sec
Width 2007.8 Hz
2D Width 2007.8 Hz
Single scan
83 increments
OBSERVE H1, 599.8723061 MHz
DATA PROCESSING
Sine bell 0.064 sec
F1 DATA PROCESSING
Sine bell 0.021 sec
FT size 512 x 512
Total time 1 min, 38 sec



37

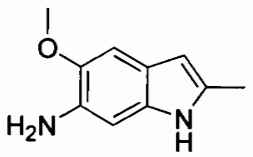


STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 9000.9 Hz
16 repetitions
OBSERVE H1, 599.8751458 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec

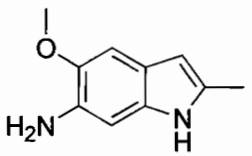


38

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 2-117
INOVA-600 "inova-wkst"

Relax. delay 2.000 sec
Pulse 47.4 degrees
Acq. time 2.731 sec
Width 5999.7 Hz
32 repetitions
OBSERVE H1, 399.9486779 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 2 min, 31 sec



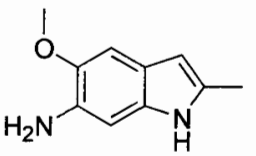
38

STANDARD CARBON PARAMETERS

Jun 9 2011

Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
User: 1-14-87
File: 2-99_c13_6_9_2011
GEMINI-2000B "gem2000"

Pulse 46.4 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
576 repetitions
OBSERVE C13, 150.8387896 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 36 hr, 47 min, 1 sec



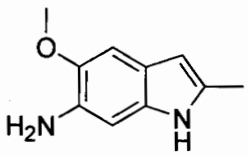
38

STANDARD PROTON PARAMETERS

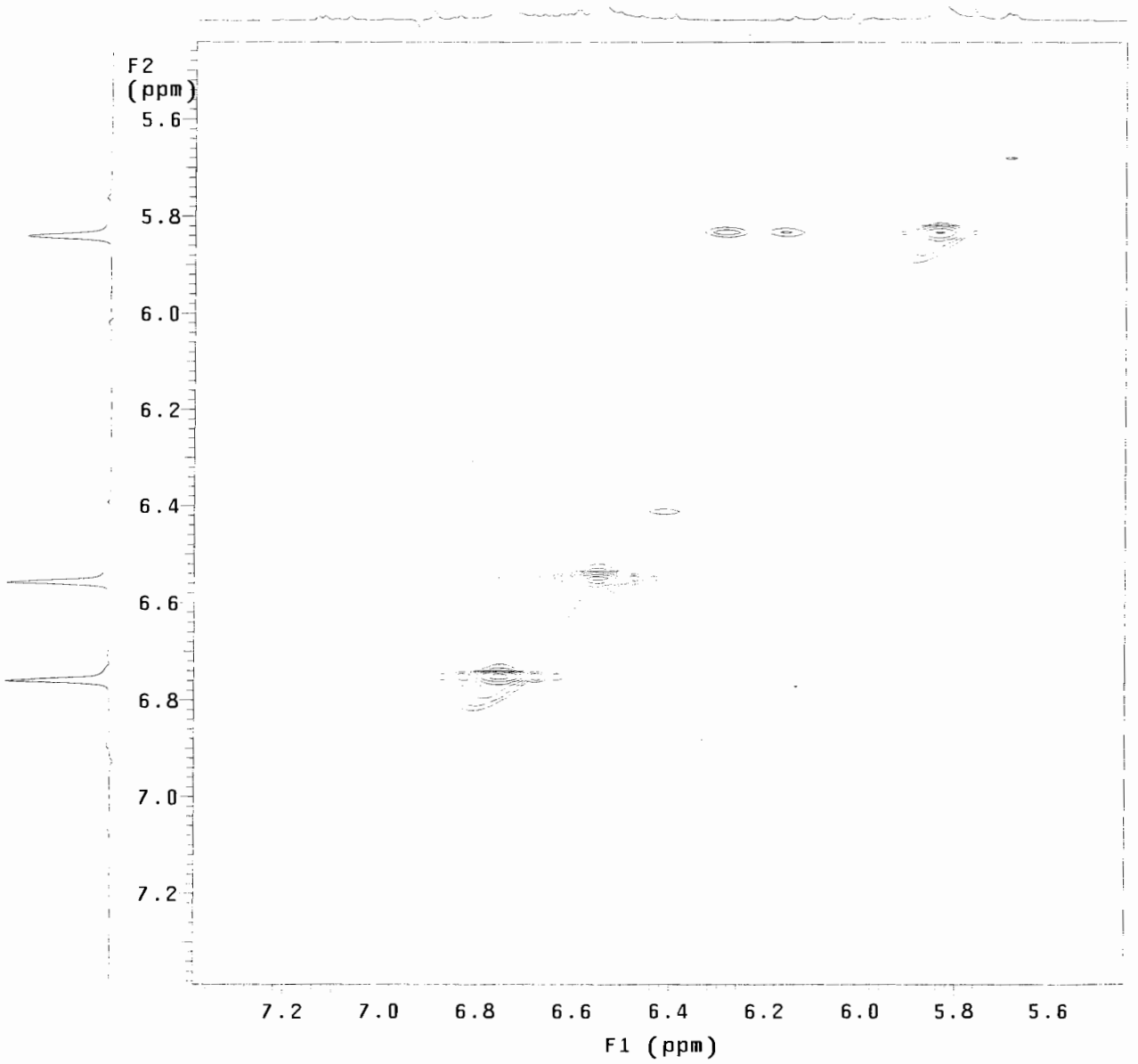
Pulse Sequence: gcosy

Solvent: DMSO
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.219 sec
Width 1170.6 Hz
2D Width 1170.6 Hz
Single scan
48 increments
DBSERVE H1, 599.8751458 MHz
DATA PROCESSING
Sine bell 0.109 sec
F1 DATA PROCESSING
Sine bell 0.021 sec
FT size 512 x 512
Total time 1 min, 2 sec



38

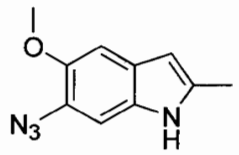


STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
File: 2-119
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 599.8723055 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



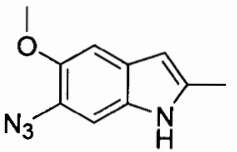
39

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 2-119_c13
INOVA-600 "inova-wkst"

Pulse 46.4 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
400 repetitions
OBSERVE C13, 150.8380121 MHz
DECOUPLE H1, 599.8753144 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 2 hr, 48 min, 9 sec



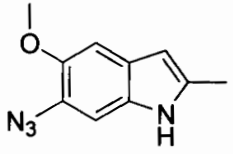
39

STANDARD PROTON PARAMETERS

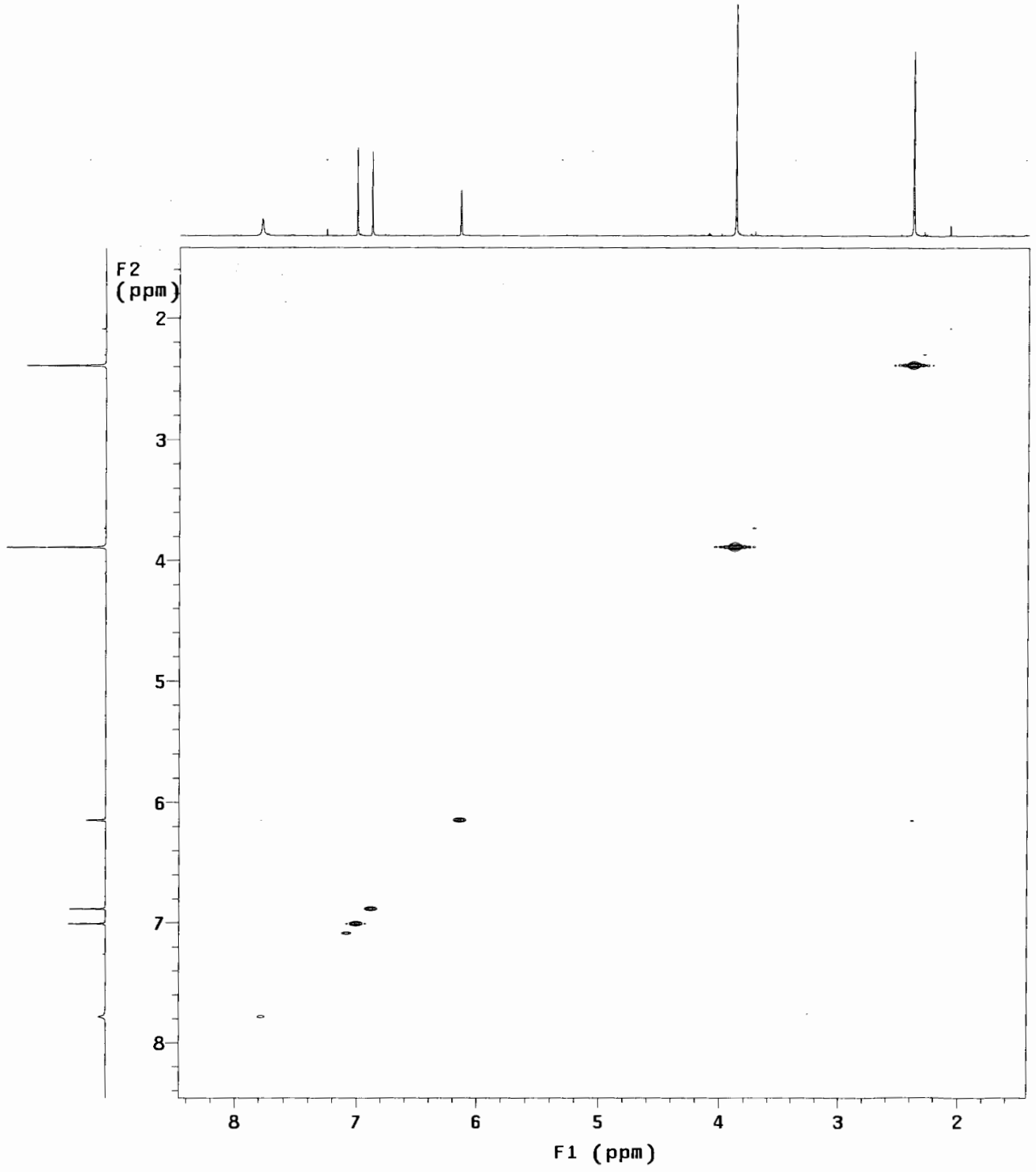
Pulse Sequence: gcosy

Solvent: CDCl3
Ambient temperature
File: 2-119_cosy
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Acq. time 0.242 sec
Width 4225.9 Hz
2D Width 4225.9 Hz
Single scan
176 increments
OBSERVE H1, 599.8723055 MHz
DATA PROCESSING
Sine bell 0.121 sec
F1 DATA PROCESSING
Sine bell 0.021 sec
FT size 2048 x 2048
Total time 3 min, 47 sec



39

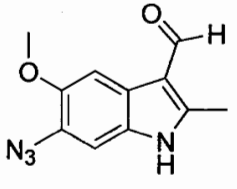


STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
INOVA-600 "inova600"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 9000.9 Hz
16 repetitions
OBSERVE H1, 599.8751433 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



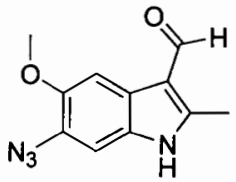
40

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: OMSO
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-600 "inova600"

Pulse 46.4 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
41392 repetitions
OBSERVE C13, 150.8386940 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 14 hr, 45 sec



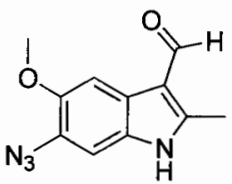
40

STANDARD PROTON PARAMETERS

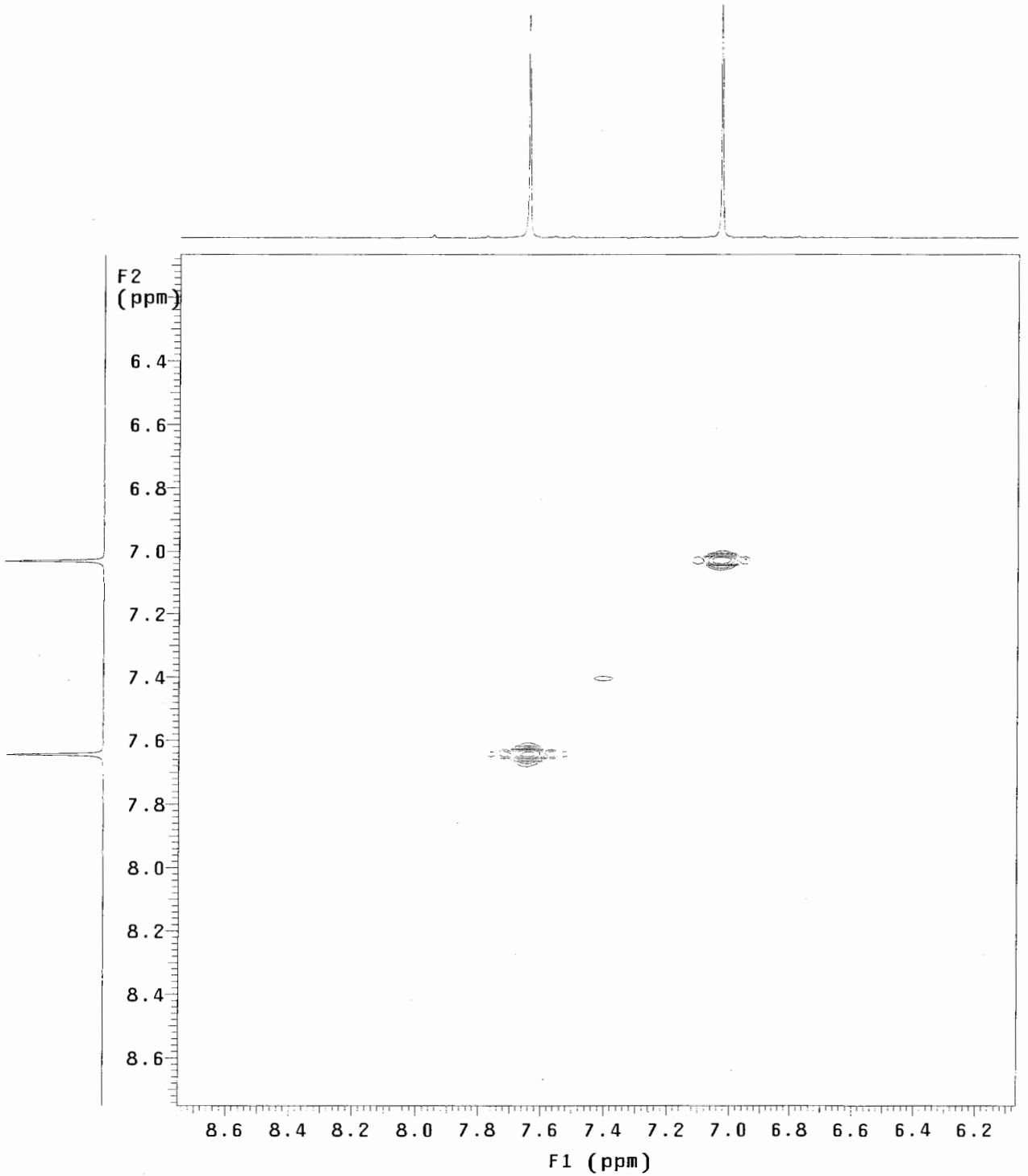
Pulse Sequence: gcosy

Solvent: DMSO
Temp. 25.0 C / 298.1 K
File: 2-107_cosy_b
INDVA-600 "inova600"

Relax. delay 1.000 sec
Acq. time 0.158 sec
Width 1616.0 Hz
2D Width 1616.0 Hz
Single scan
67 increments
OBSERVE H1, 599.8751439 MHz
DATA PROCESSING
Sine bell 0.079 sec
F1 DATA PROCESSING
Sine bell 0.021 sec
FT size 512 x 512
Total time 1 min, 22 sec



40

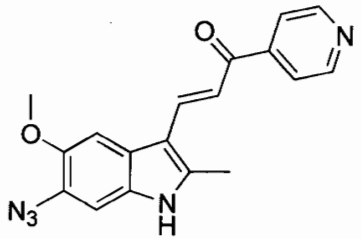


STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
File: 2-113_H1_b
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Pulse 31.8 degrees
Acq. time 3.000 sec
Width 8999.9 Hz
16 repetitions
OBSERVE H1, 599.8751432 MHz
DATA PROCESSING
Line broadening 0.4 Hz
FT size 131072
Total time 1 min, 4 sec



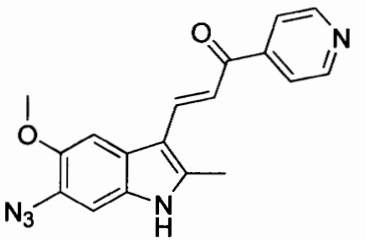
41

STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul

Solvent: DMSO
Ambient temperature
User: 1-14-87
File: 2-113_C13
INOVA-600 "inova-wkst"

Pulse 46.4 degrees
Acq. time 1.000 sec
Width 37735.8 Hz
39968 repetitions
OBSERVE C13, 150.8387910 MHz
DECOUPLE H1, 599.8781638 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss window 0.600 sec
center at 0.100 sec
FT size 524288
Total time 28 hr, 1 min, 31 sec



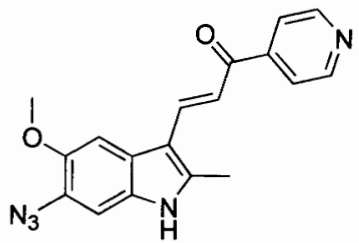
41

STANDARD PROTON PARAMETERS

Pulse Sequence: gcosy

Solvent: DMSO
Ambient temperature
File: 2-113_cosy
INOVA-600 "inova-wkst"

Relax. delay 1.000 sec
Acq. time 0.228 sec
Width 2247.0 Hz
2D Width 2247.0 Hz
Single scan
93 increments
OBSERVE H1, 599.8751442 MHz
DATA PROCESSING
Sine bell 0.114 sec
F1 DATA PROCESSING
Sine bell 0.021 sec
FT size 1024 x 1024
Total time 1 min, 59 sec



41