

Supporting Information

Synthesis of Tetramic Acid Derivatives via Intramolecular sp^3 C–H Amination Mediated by Hypervalent Iodine(III) Reagents/Brønsted Acids

Lujia Mao, Yan Li*, Tao Xiong, Kai Sun and Qian Zhang*

Department of Chemistry, Northeast Normal University, Changchun, 130024, China

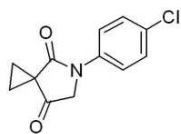
Email: liy078@nenu.edu.cn; zhangq651@nenu.edu.cn

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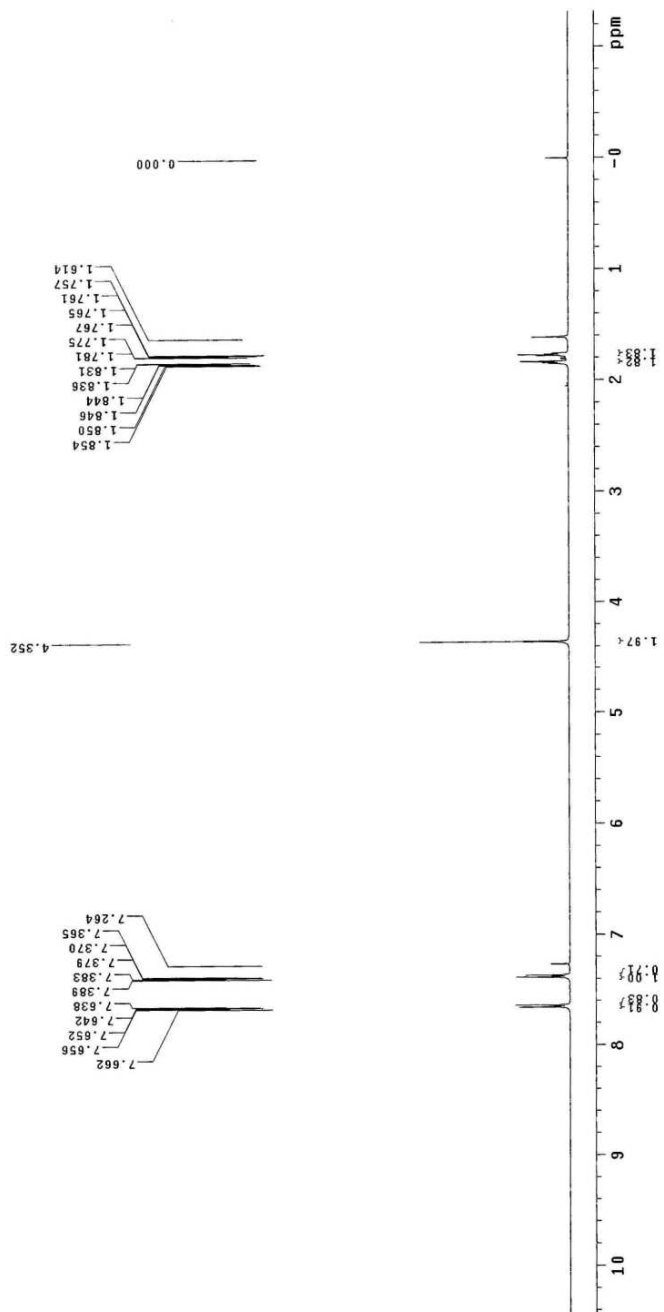
^1H NMR and ^{13}C NMR spectra copies for compounds 2	S2–35
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¹H NMR and ¹³C NMR spectra copies for compounds 2

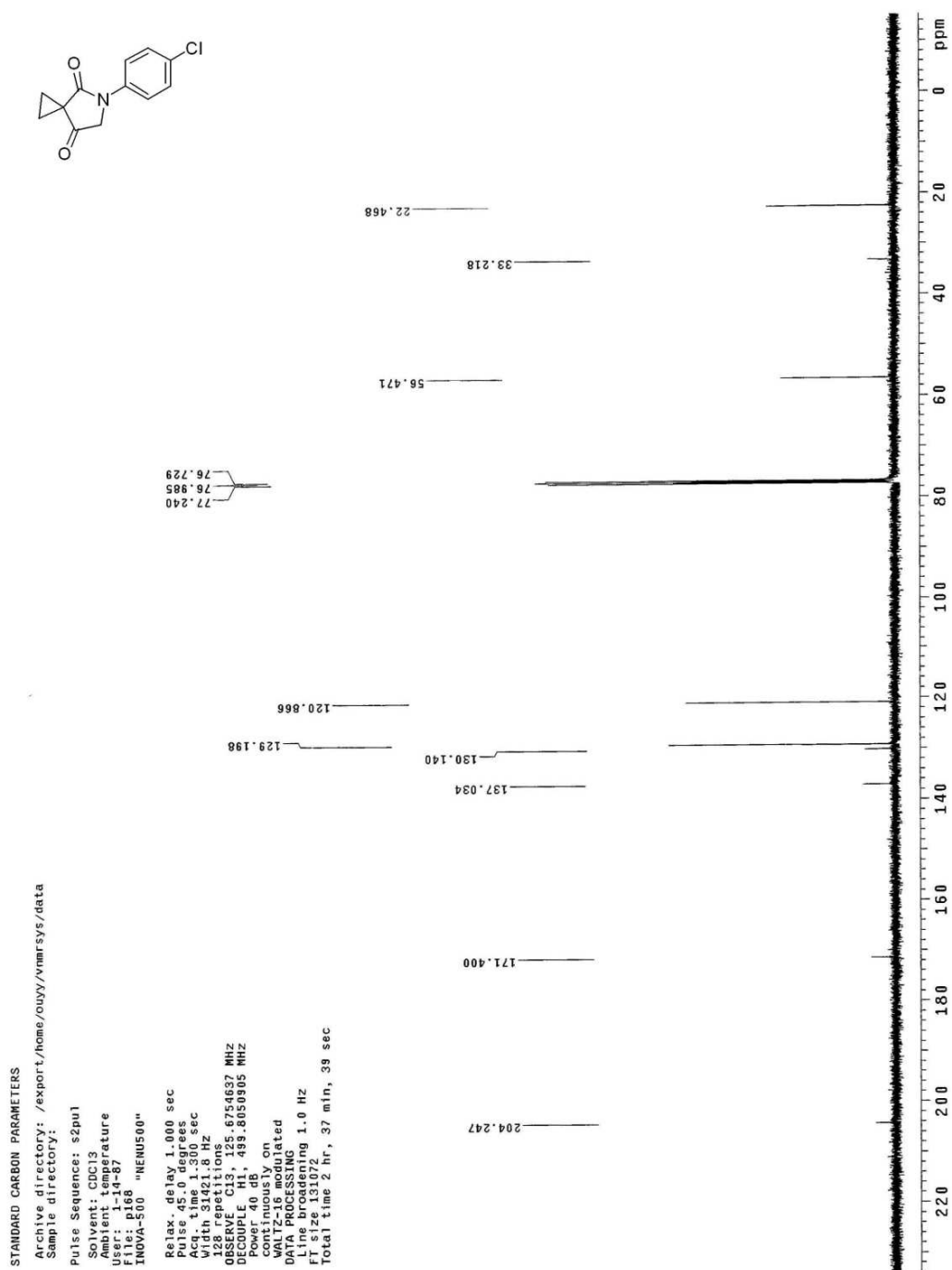
Compound 2a



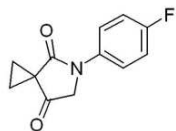
STANDARD PROTON PARAMETERS
 Archive directory: /export/home/ouyy/vnmrsws/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 File: p167
 INOVA-500 "HREUS00"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.832 sec
 Width 899.8 Hz
 Observed F1 (MHz)
 OBSERVE H1 499.8025895 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 23 sec



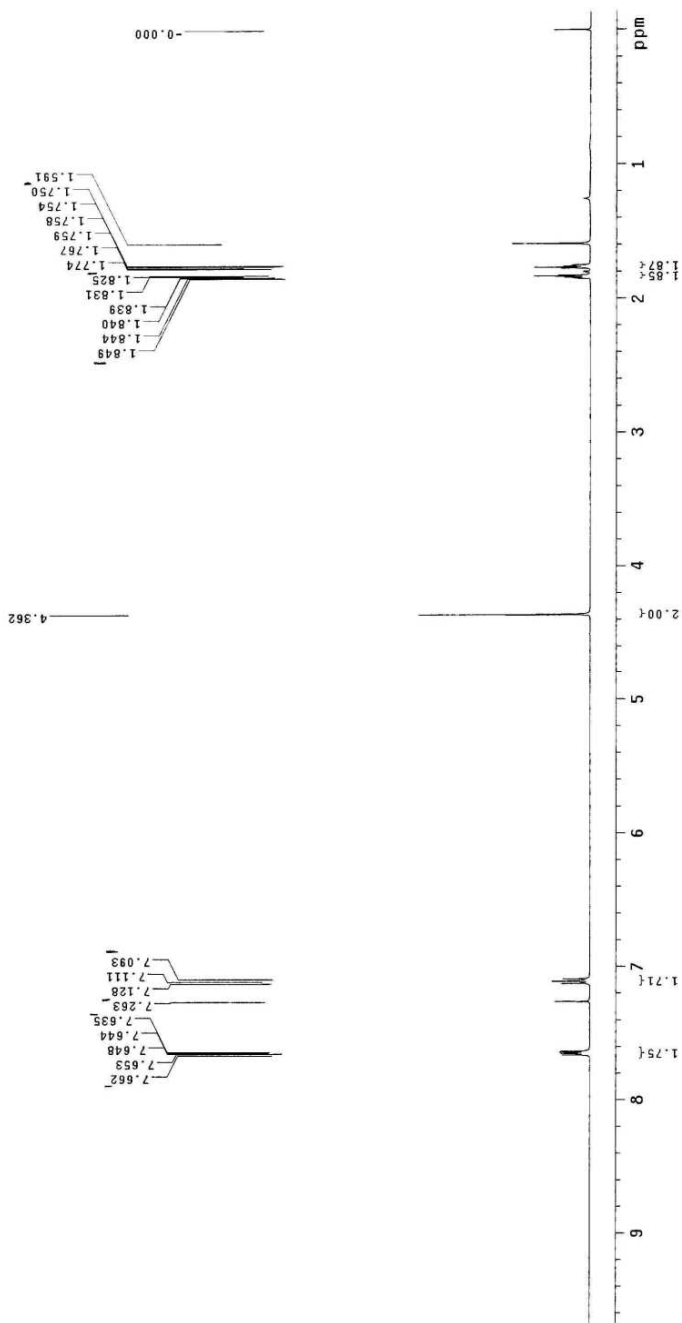
Compound 2a



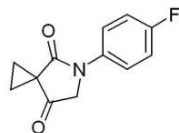
Compound 2b



STANDARD PROTON PARAMETERS
 Archive directory: /export/home/ouys/vnmr/sys/data
 Sample directory:
 Pulse Sequence: szpu1
 Solvent: CDCl3
 Ambient temperature
 F2 size 65536
 INOVA-500 "MNU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.882 sec
 F1 size 65536
 F2 size 65536
 8 F2 data files
 OBSERVE H1, 499.8025903 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 23 sec



Compound 2b



STANDARD CARBON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul1

Solvent: CDCl₃

Ambient temperature

User: 1-14-87

File: p182

INOVA-500 "MENU500"

Relax. delay 0.500 sec

Power 40 dB

Acq. time 1.300 sec

Width 31421.8 Hz

5632 repetitions

Observed C13, 125.6754608 MHz

Observed H1, 499.8050305 MHz

Power 40 dB

continuously on

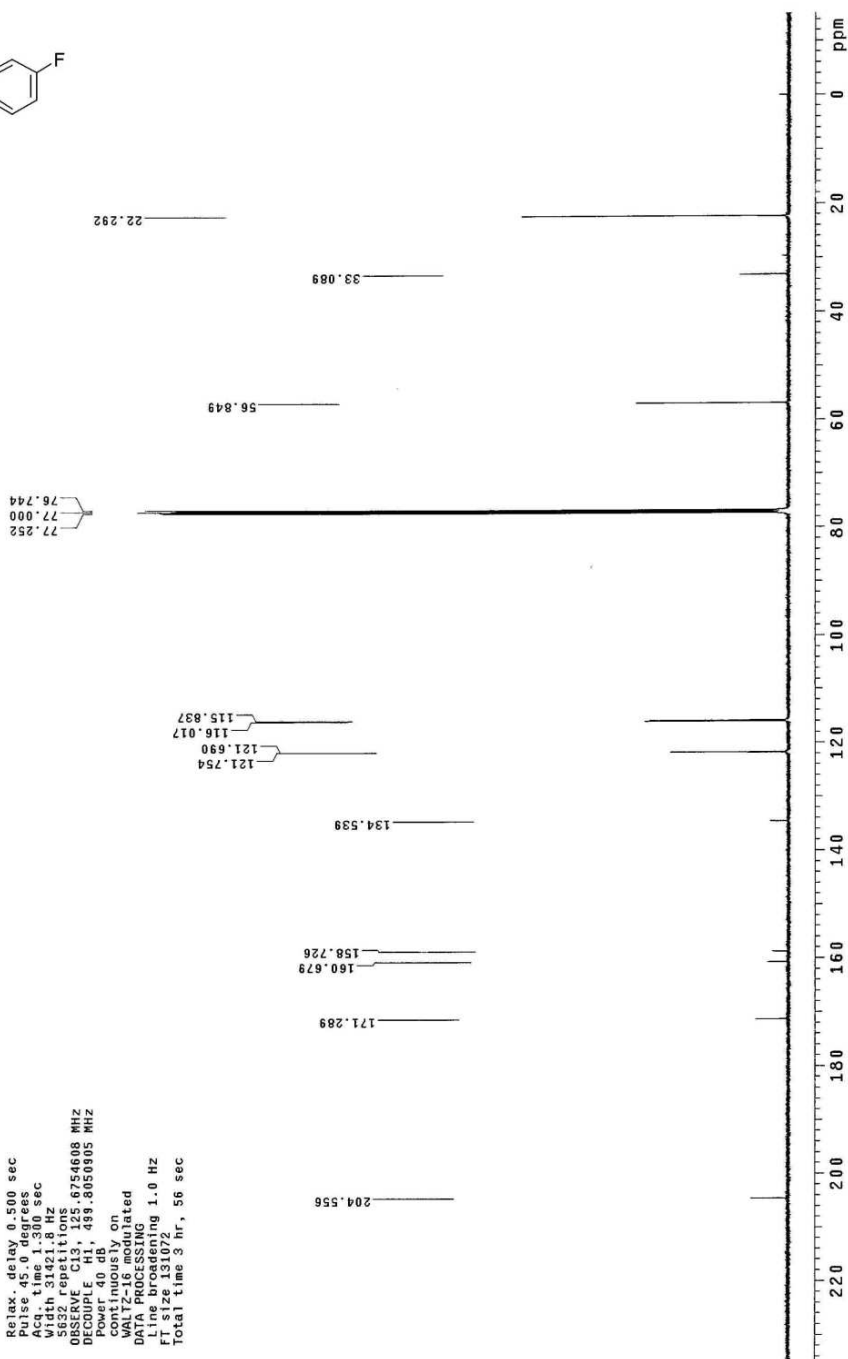
WALTZ-16 modulated

DATA PROCESSING

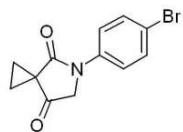
Time acquisition 1.0 Hz

FT size 131072

Total time 3 hr, 56 sec



Compound 2c



STANDARD PROTON PARAMETERS

Archive directory: /export/home/ouy/vnmr/sys/data
Sample directory:

Pulse Sequence: s2pu1

Solvent: CDCl₃

Ambient temperature

File: k323

INOVA-500 "NMRUS00"

Relax. delay 1.000 sec

Pulse 45.0 deg

Acq. time 1.892 sec

Width 8440.6 Hz

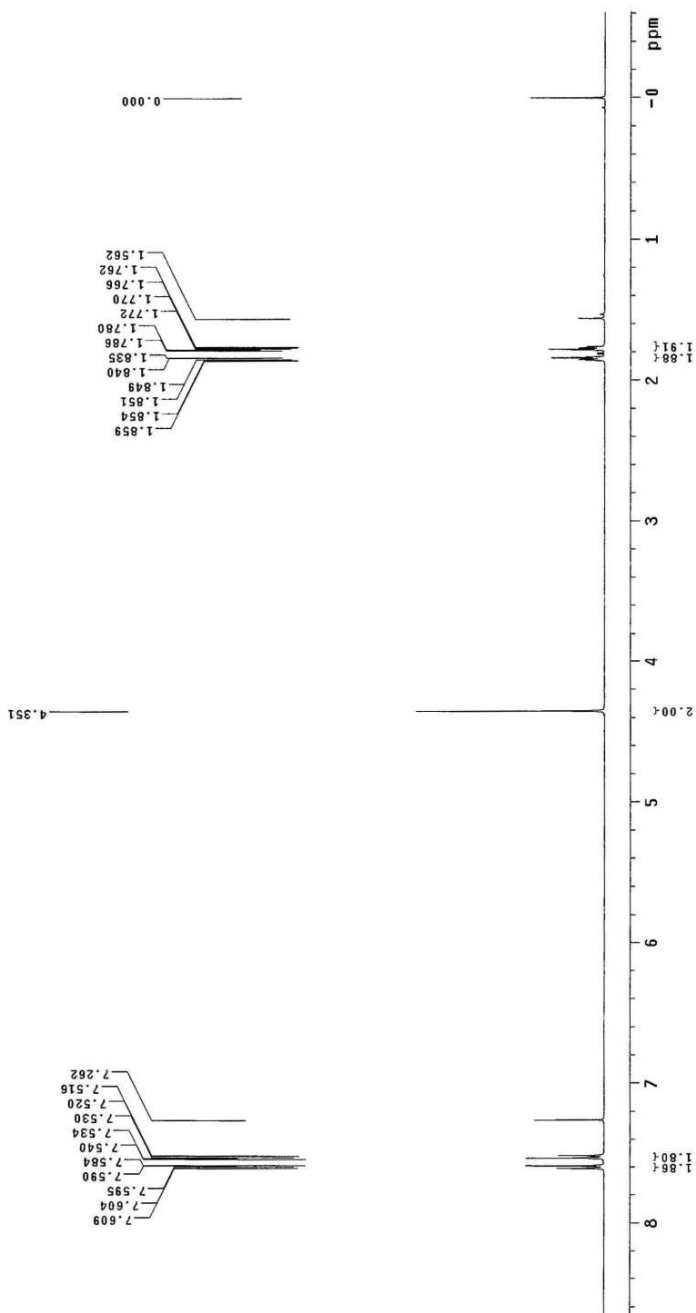
Frequency 500.136 MHz

Observed 499.5025914 MHz

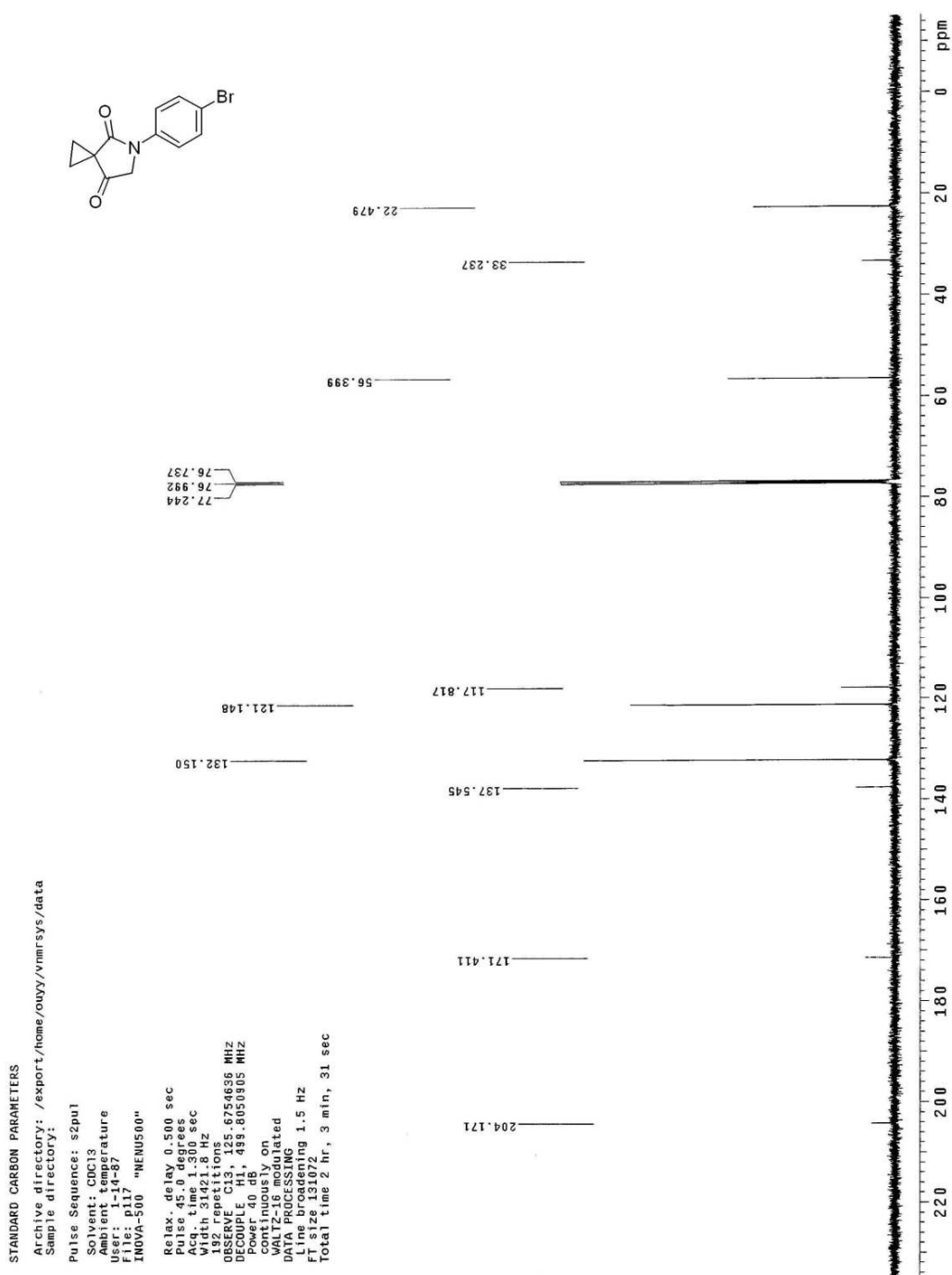
DATA PROCESSING

FT size 65536

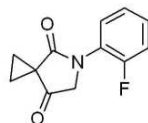
Total time 0 min, 23 sec



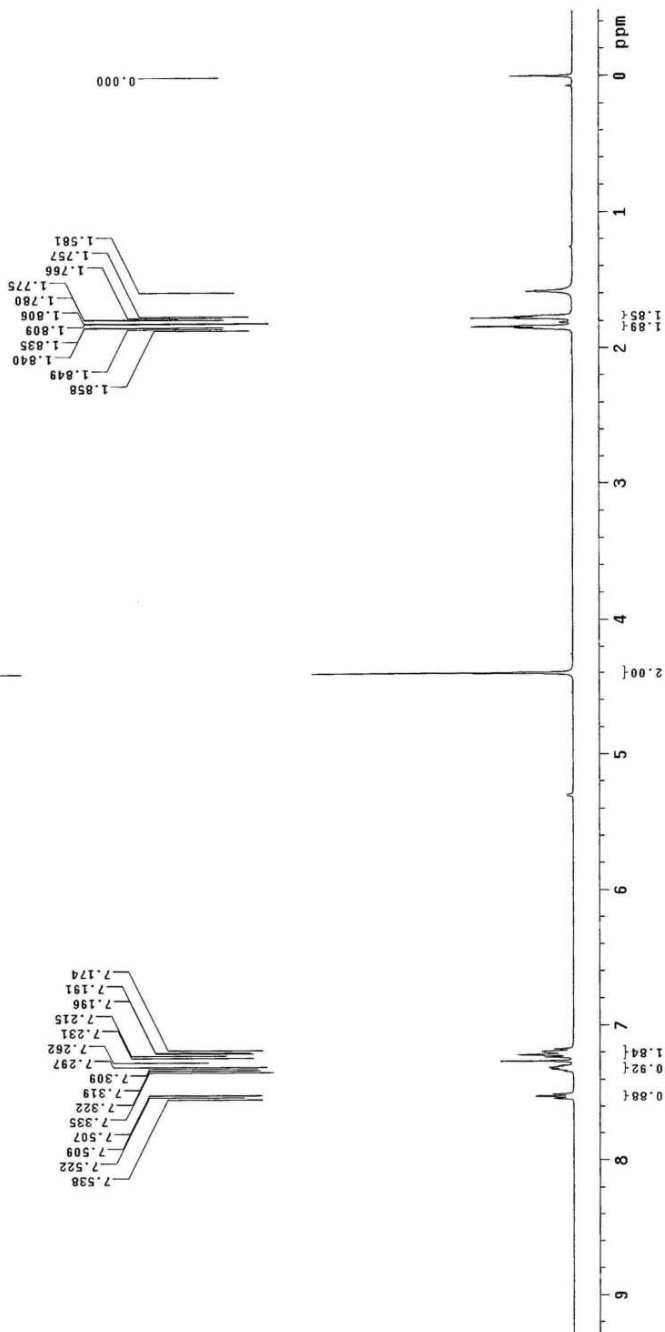
Compound 2c



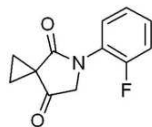
Compound 2d



STANDARD PROTON PARAMETERS
 Archive directory: /export/home/duffy/vnmr/sys/data
 Sample directory:
 Pulse Sequence: s2pul1
 Solvent: CDCl3
 Ambient temperature
 F1 size 65536
 INOVA-500 "HMENU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 Width 31500 Hz
 SFO 500.136 MHz
 8 repetitions
 OBSERVE H1, 499.8025888 MHz
 DATA PROCESSING
 F1 size 65536
 Total time 0 min, 23 sec



Compound 2d



STANDARD CARBON PARAMETERS

Archive directory: /export/home/ouy/vnmr/sys/data

Sample directory:

Pulse Sequence: s2pu1

Solvent: cdcl3

Acq. temperature

File: e326

INOVA-500 "HENU500"

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 0.500 sec

Width 31421.8 Hz

4928 repetitions

OBSERVE C13, 125.6754642 MHz

DECOUPLE H1, 499.8050905 MHz

Power 22.00 dB

Continuously on

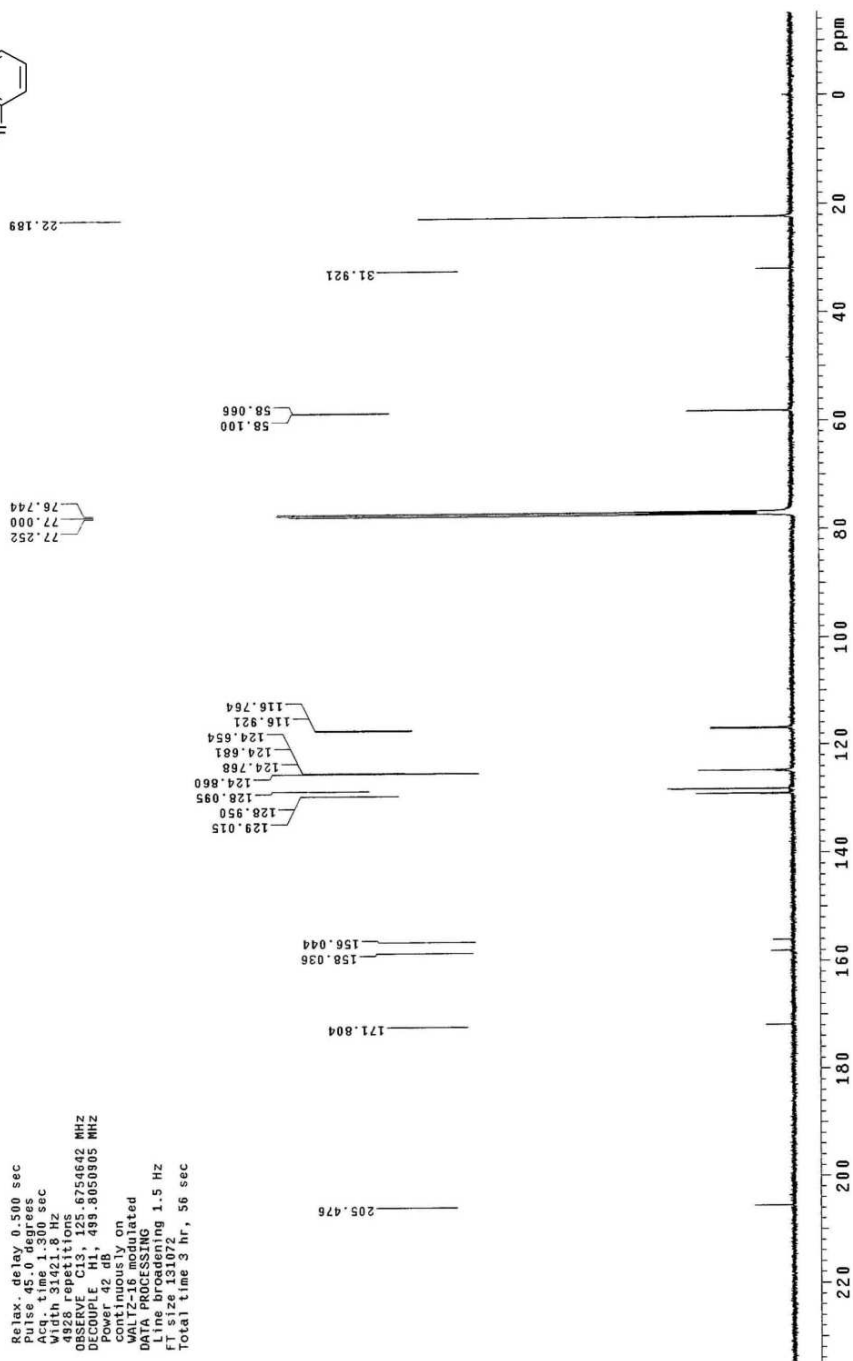
WALTZ-16 modulated

DATA PROCESSING

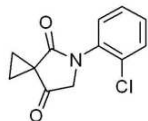
Line broadening 1.5 Hz

File size 131072

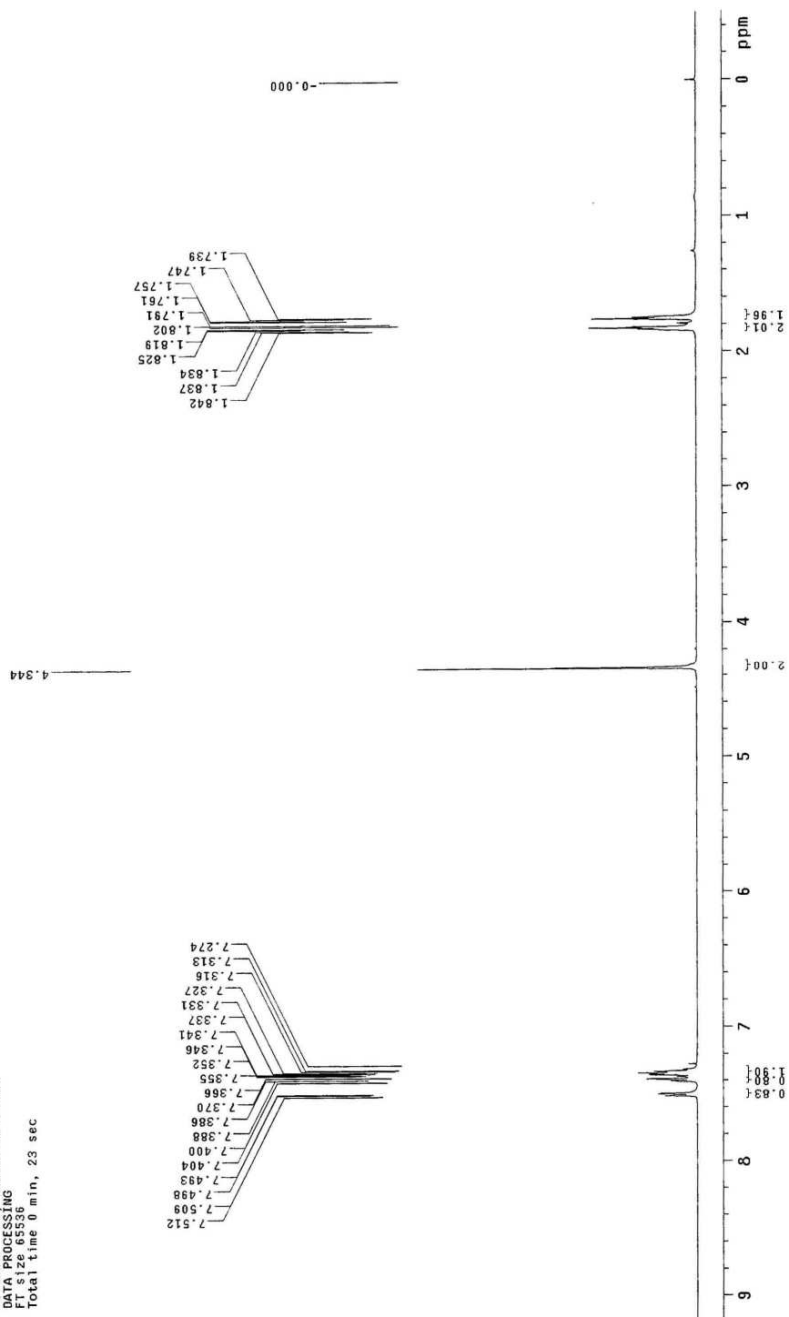
Total time 3 hr, 56 sec



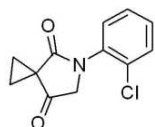
Compound 2e



STANDARD PROTON PARAMETERS
 Archive directory: /export/home/ouy/vnmr/sy/data
 Sample directory:
 Pulse Sequence: szpu1
 Solvent: CDCl3
 Ambient temperature
 F1 size 65536
 INOVA-500 "NMR500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 F1 size 65536 Hz
 8 repetitions
 OBSERVE H1, 499.8025846 MHz
 DATA PROCESSING
 F1 size 65536
 Total time 0 min, 23 sec



Compound 2e



STANDARD CARBON PARAMETERS

Archive directory: /export/home/ouyy/vnmr/sys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

User: 1-14-87

File: 1-14-87

INOVA-500 "MENV500"

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 31421.8 Hz

15 Spectral tone

OBSERVE C13, 125.6754737 MHz

DECOUPLE H1, 499.8050805 MHz

Power 40 dB

continuously on

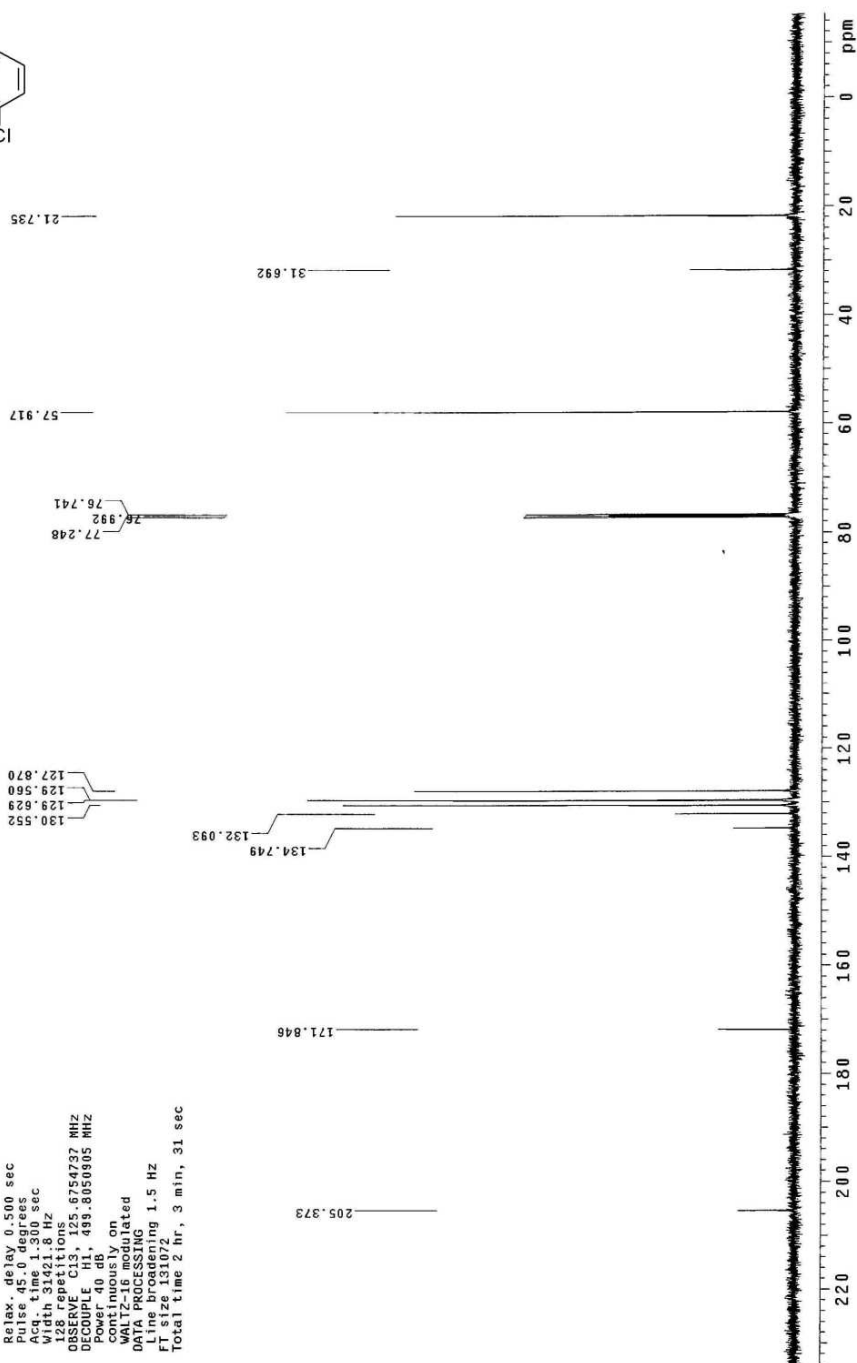
Antenna cooled

DATA PROCESSING

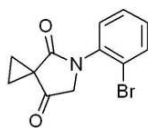
Line broadening 1.5 Hz

FT size 131072

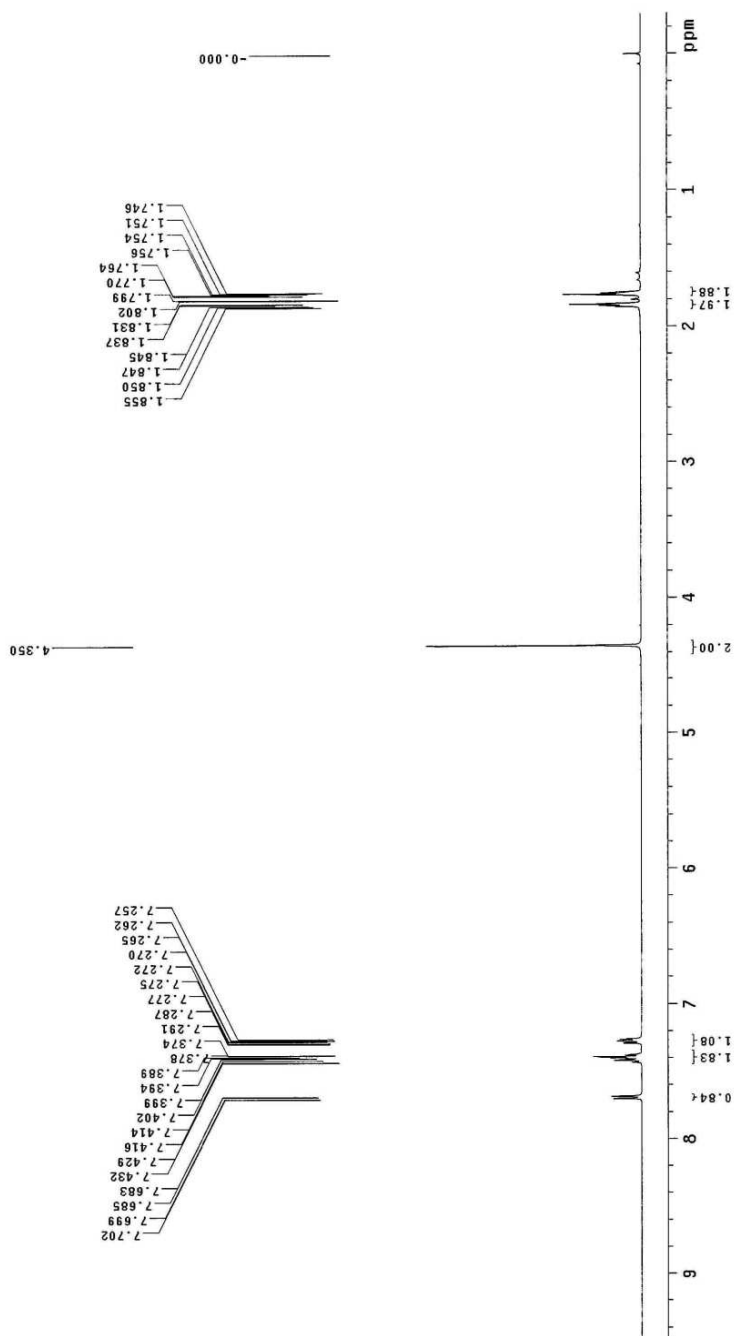
Total time 2 hr, 3 min, 31 sec



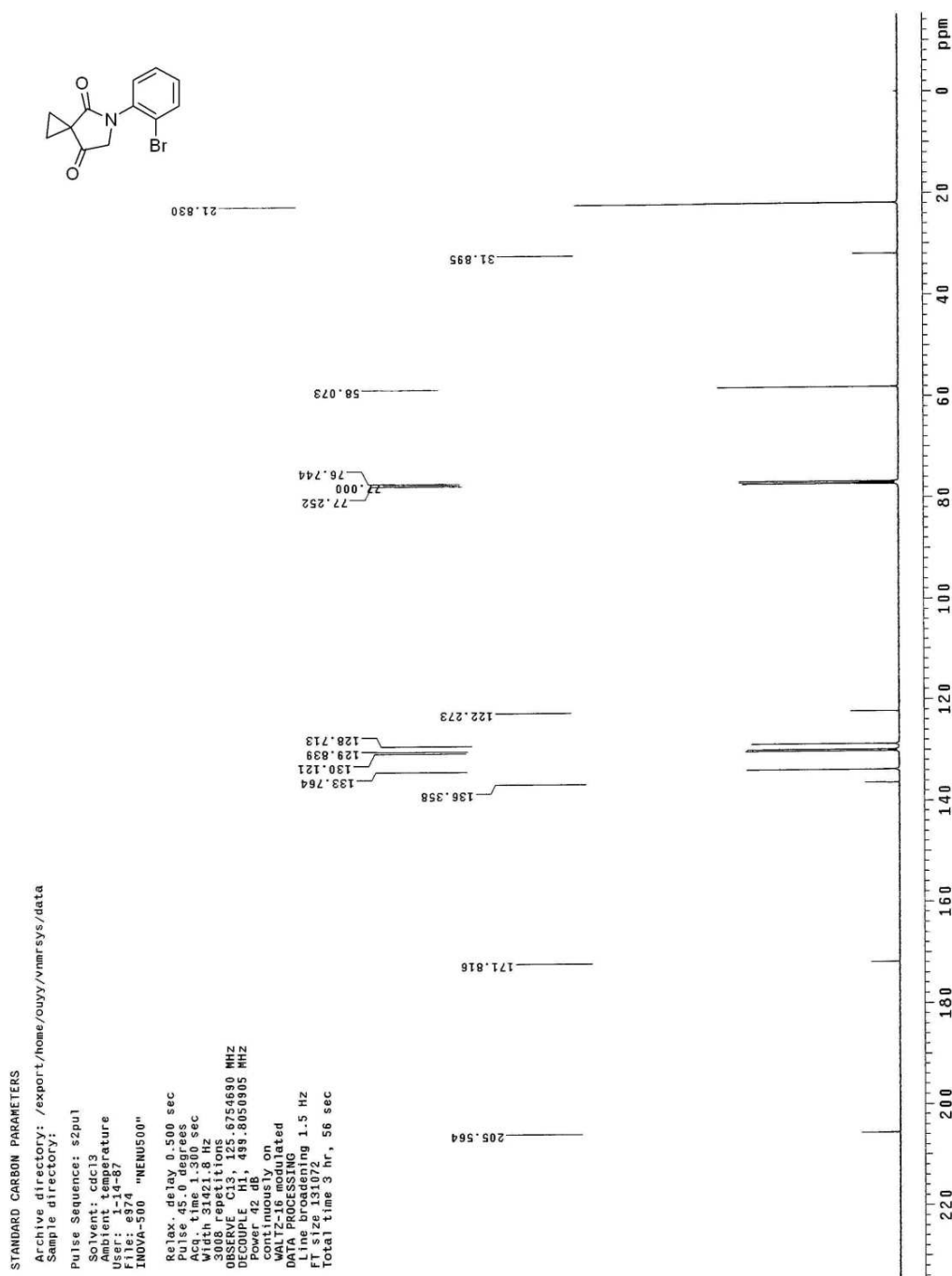
Compound 2f



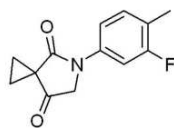
STANDARD PROTON PARAMETERS
 Archive directory: /export/home/ouvy/vnmr/sys/data
 Sample directory:
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Acquisition temperature
 File: K330 "NENU500"
 INOVA-500 "NENU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acc. time 2.000 sec
 Width 8040.6 Hz
 8 repetitions
 OBSERVE H1, 499.8025907 MHz
 DATA PROCESSING
 Processing time 0.00 sec
 Total time 0 min, 23 sec



Compound 2f



Compound 2g



STANDARD PROTON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsvs/data

Sample directory:

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

File: C336

INQA-S00 "NMRUS00"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.852 sec

Wdth 8102.4 Hz

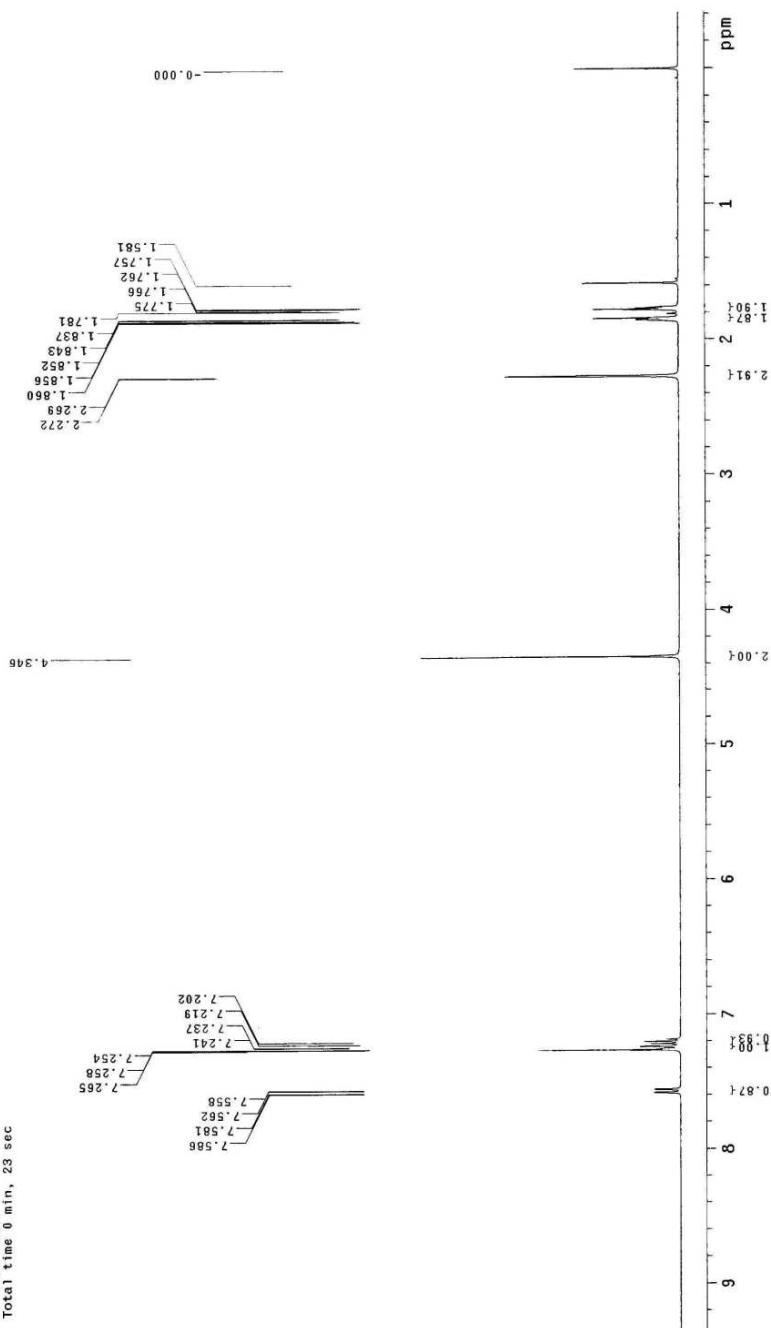
Wdth 8102.4 Hz

OBSERVE H1 499.3025906 MHz

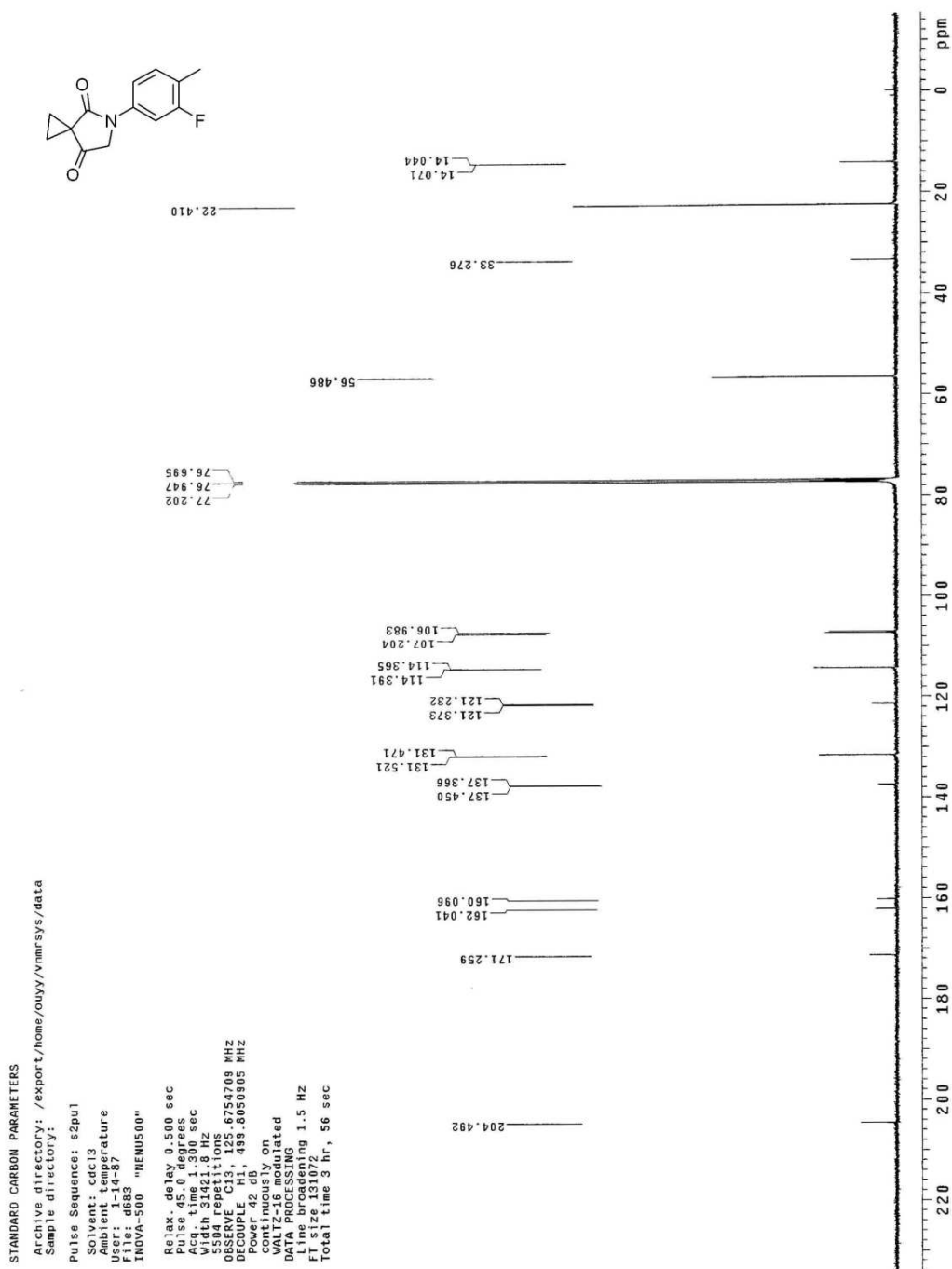
DATA PROCESSING

FT size 65536

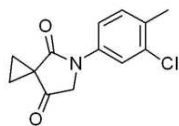
Total time 0 min, 23 sec



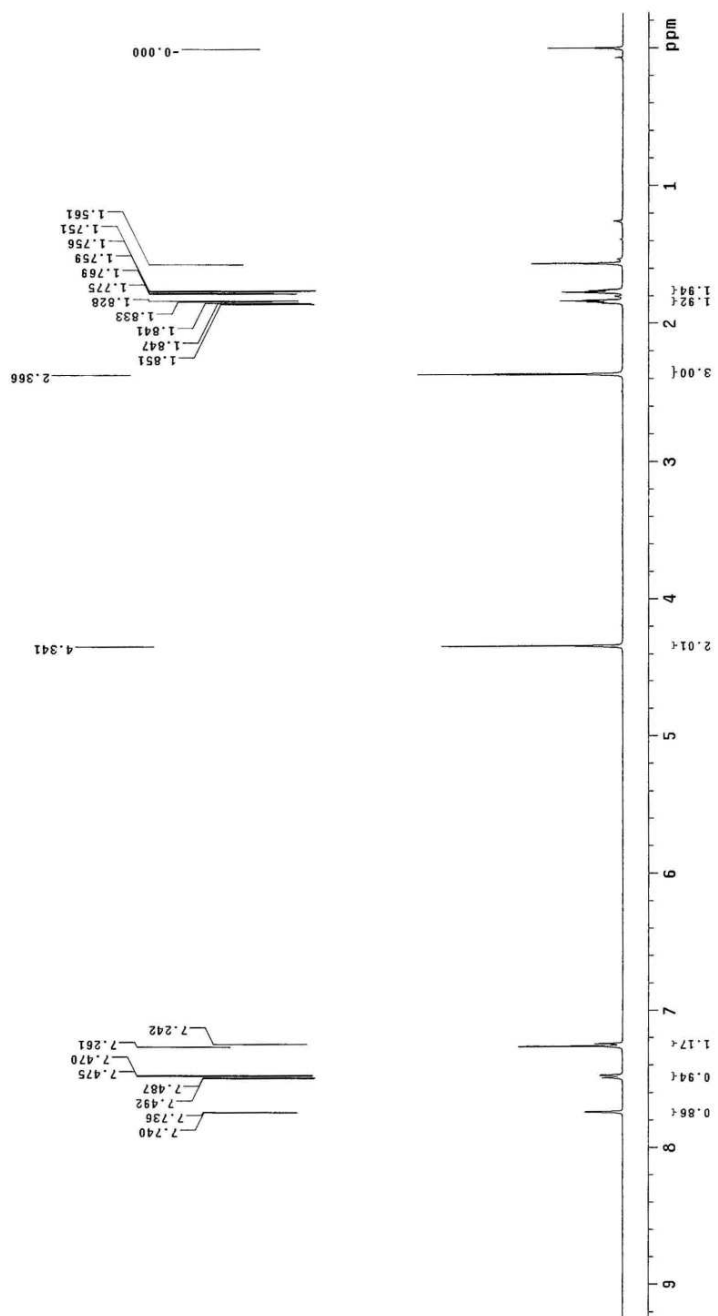
Compound 2g



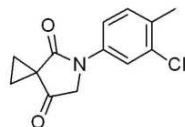
Compound 2h



STANDARD PROTON PARAMETERS
 Archive directory: /export/home/ouyy/vmarsys/data
 Sample directory:
 Pulse Sequence: szpul
 Solvent: CDCl3
 Ambient temperature
 File: k328
 INOVA-500 "HENU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 Width 8440.6 Hz
 Spectral width 10000.0 Hz
 OBSERVE H 499.8025919 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 23 sec



Compound 2h



STANDARD CARBON PARAMETERS

Archive directory: /export/home/ouy/vnmrsys/data

Sample directory:

Pulse Sequence: s2pu1

Solvent: cdcl3

Ambient temperature

User: i-14-87

File: d684

INOVA-500 "NENU500"

Relax. delay 0.500 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 31421.8 Hz

4352 repetitions

DECUPLE H1, 489.8050905 MHz

Power 42 dB

continuously on

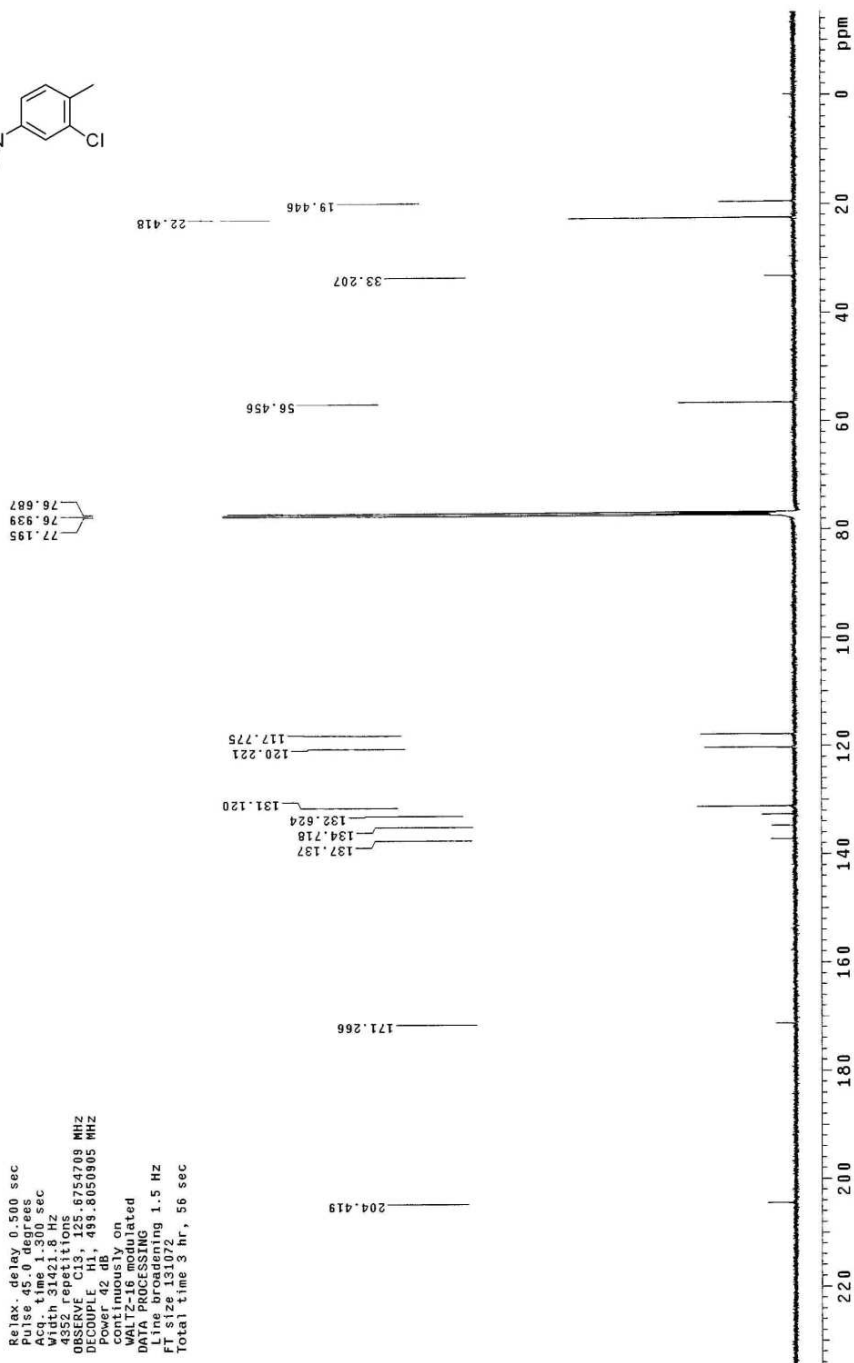
WALTZ-16 modulated

DMF-PCSS100

Time broadening 1.5 Hz

FT size 131072

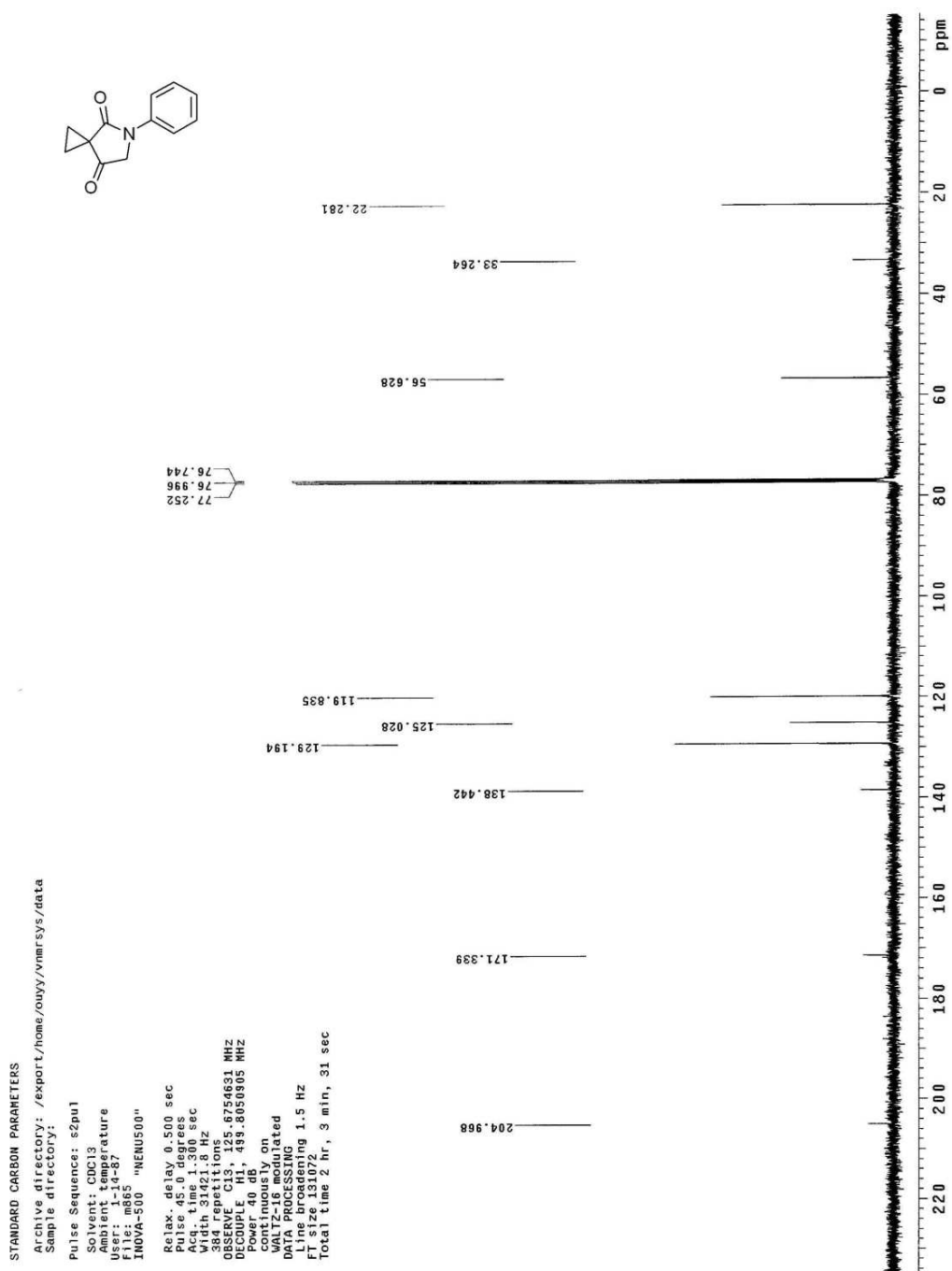
Total time 3 hr, 56 sec



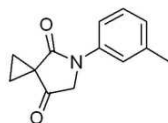
¹H NMR spectrum of 1,2,3,4,5-pentachlorobenzene in CDCl₃. The spectrum shows two main multiplets. The aromatic region (7.200–7.693 ppm) has an integration of 2.004. The aliphatic region (1.605–1.855 ppm) has an integration of 9.386. Solvent peaks for CDCl₃ are visible at 7.26 and 1.76 ppm.

Chemical Shift (ppm)	Integration
7.200 – 7.693	2.004
1.605 – 1.855	9.386

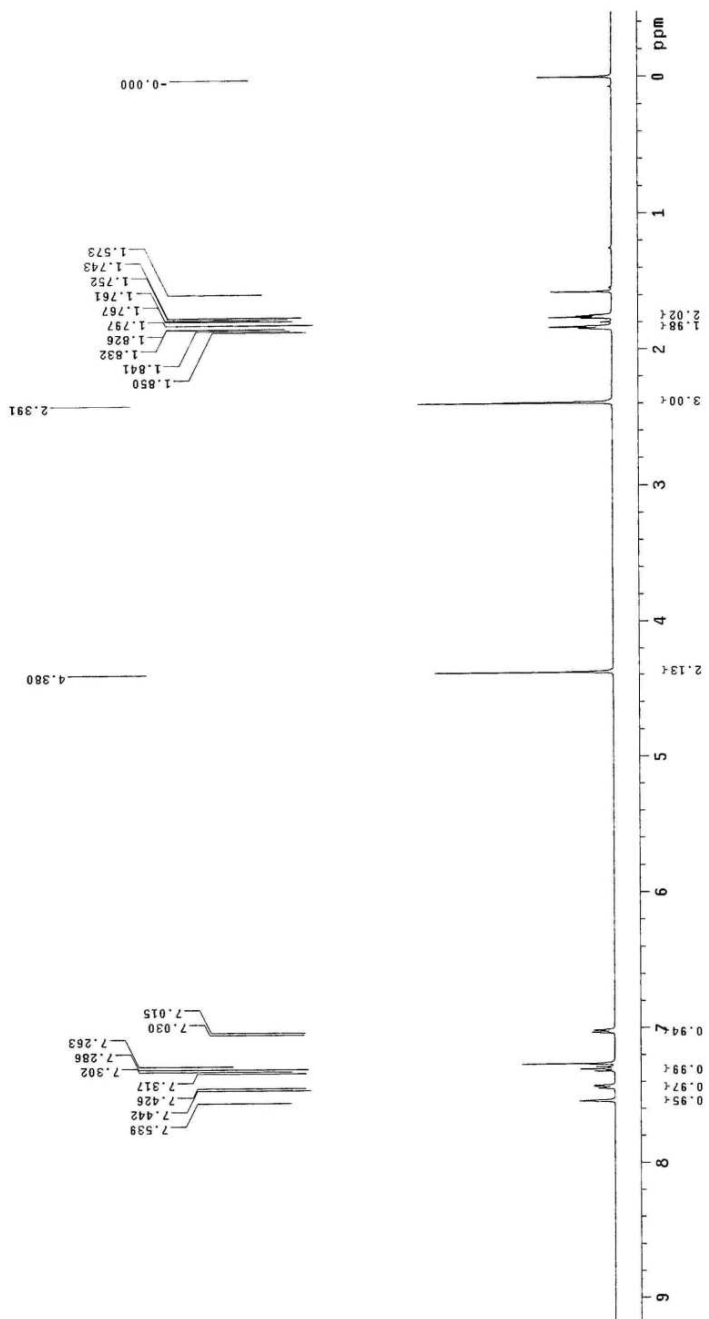
Compound 2i



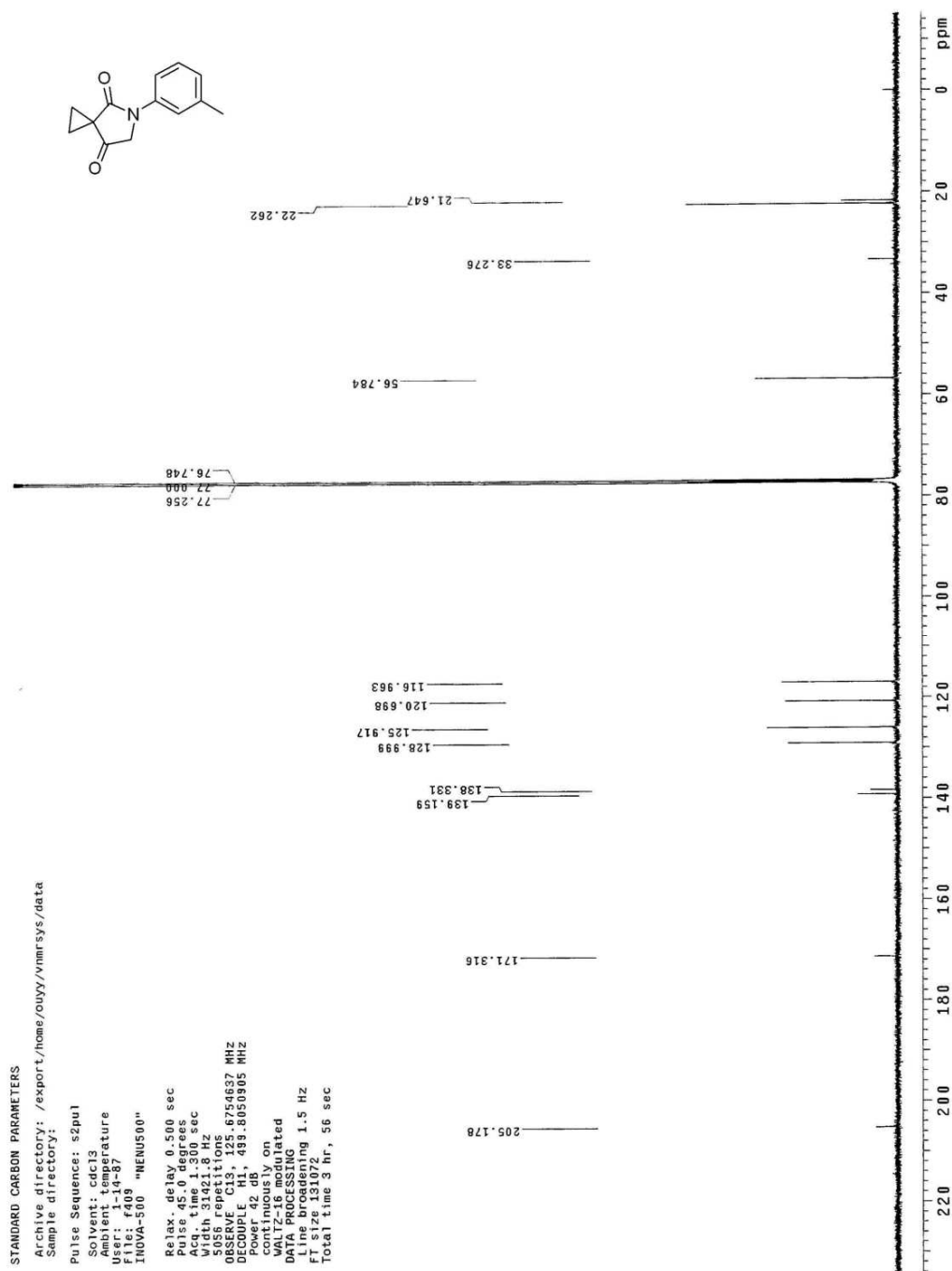
Compound 2j



STANDARD PROTON PARAMETERS
 Archive directory: /export/home/ouyy/vnmrsys/data
 Sample directory:
 Pulse Sequence: s2pul1
 Solvent: CDCl3
 Ambient temperature
 File: f450
 INOVA-500 "NEWUS00"
 Relax. delay: 1.000 sec
 Pulse: 45.0 degrees
 Acq. time: 1.892 sec
 Width: 3201.7 Hz
 Spectral width: 3201.7 Hz
 Observed: 400.136 MHz
 Reference: TMS
 DATA PROCESSING
 FT size: 65536
 Total time: 0 min, 23 sec



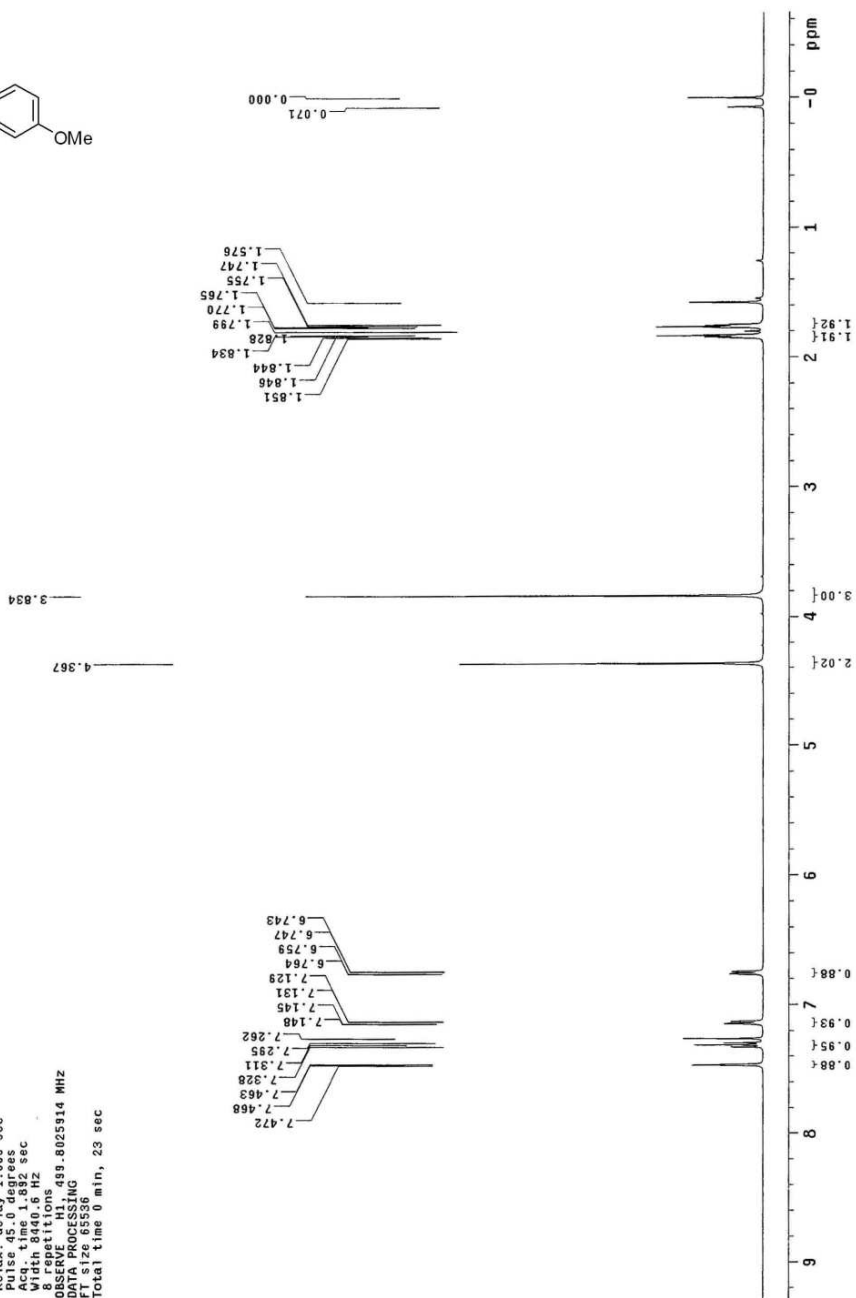
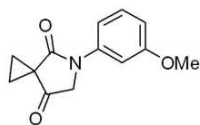
Compound 2j



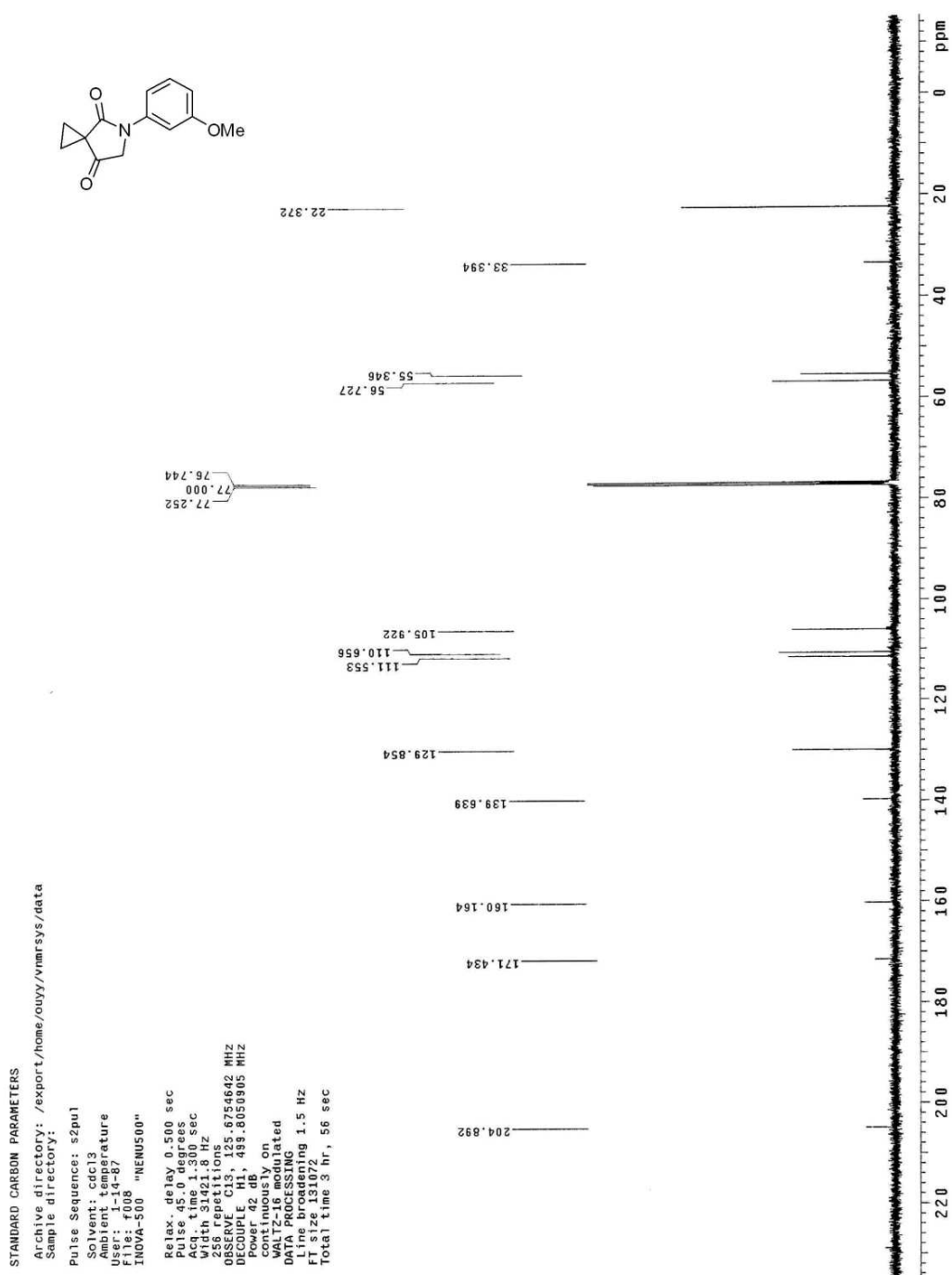
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STANDARD PROTON PARAMETERS
Archive directory: /export
Sample directory:
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature:
File: k331
INVOA=500 "NMR500"
Relax delay 1.000 sec
Pulse width 0.000 sec
Acq. time 1.892 sec
Width 8400.6 Hz
Sweep rate 100.000 Hz
Repetitions 89.802514
DATA PROCESSING
File size 85536
Total time 0 min, 23 sec

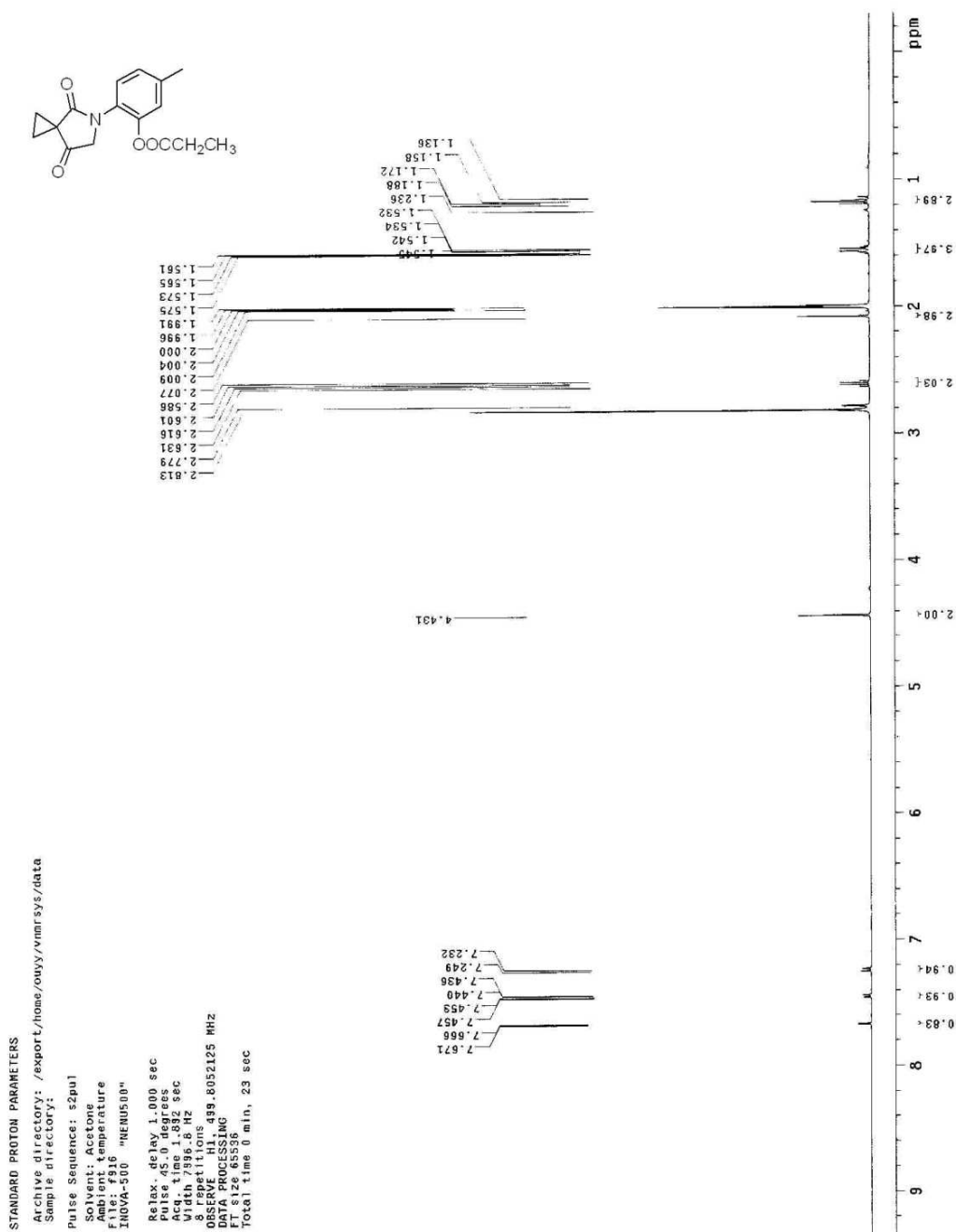
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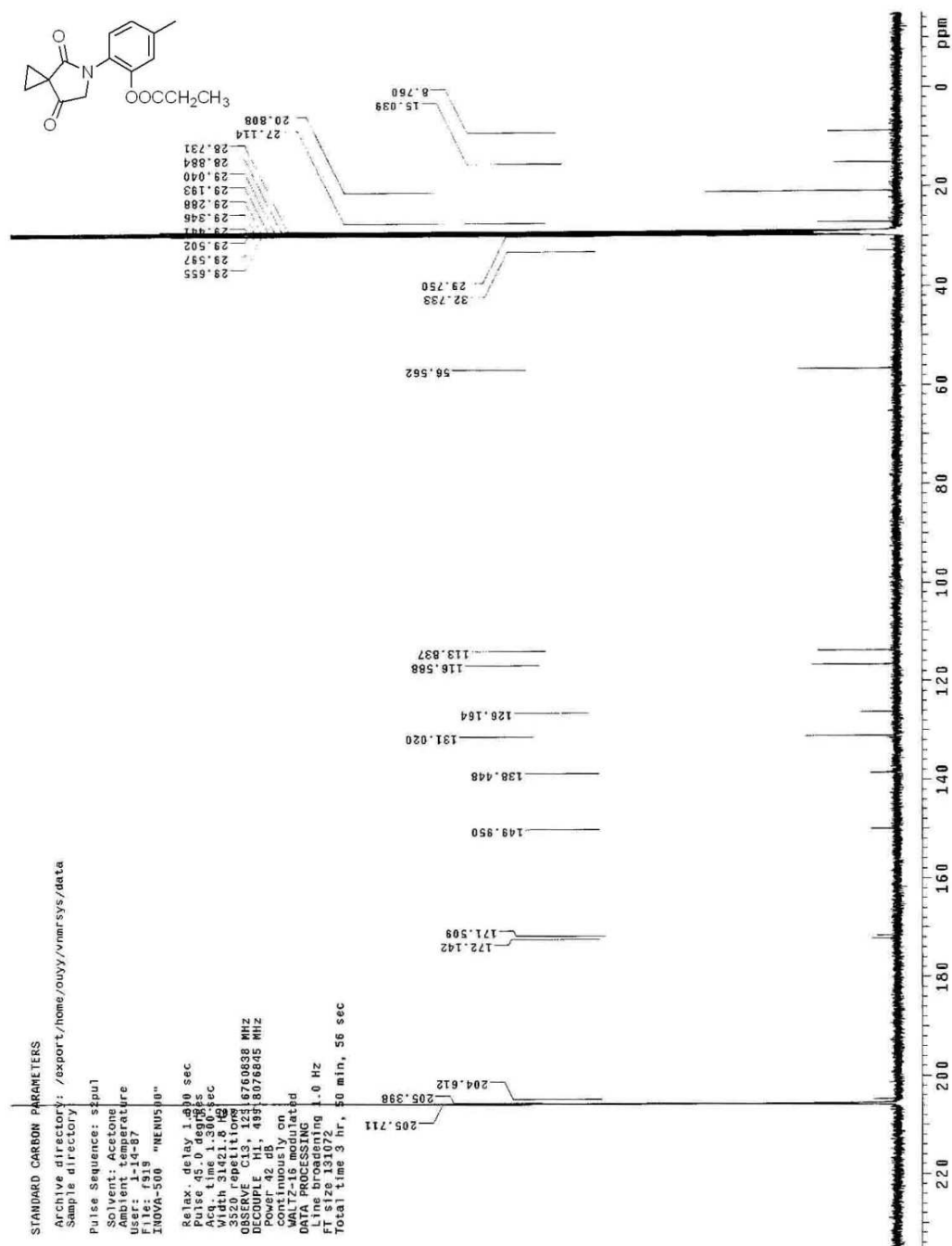


Compound 2k

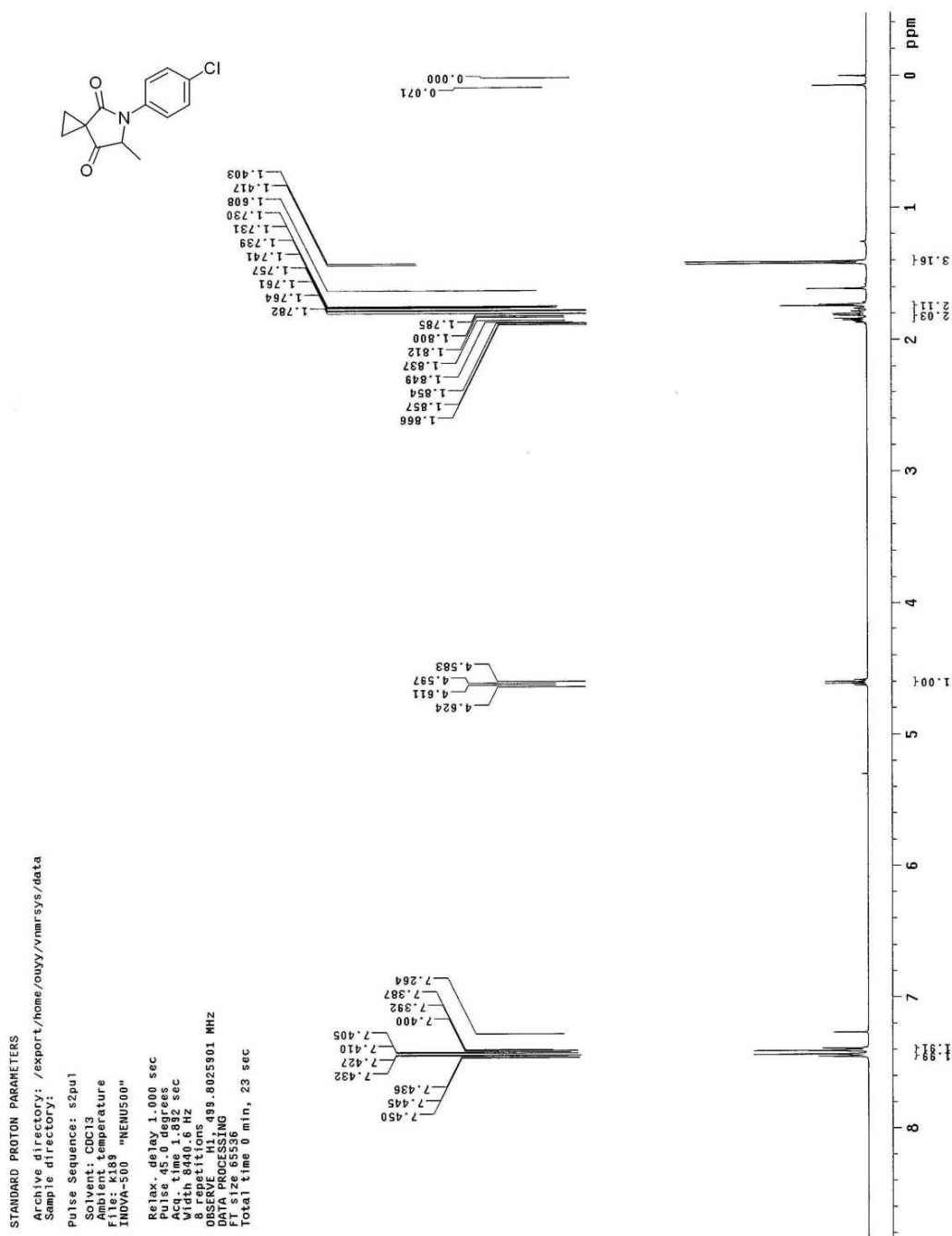


Compound 21

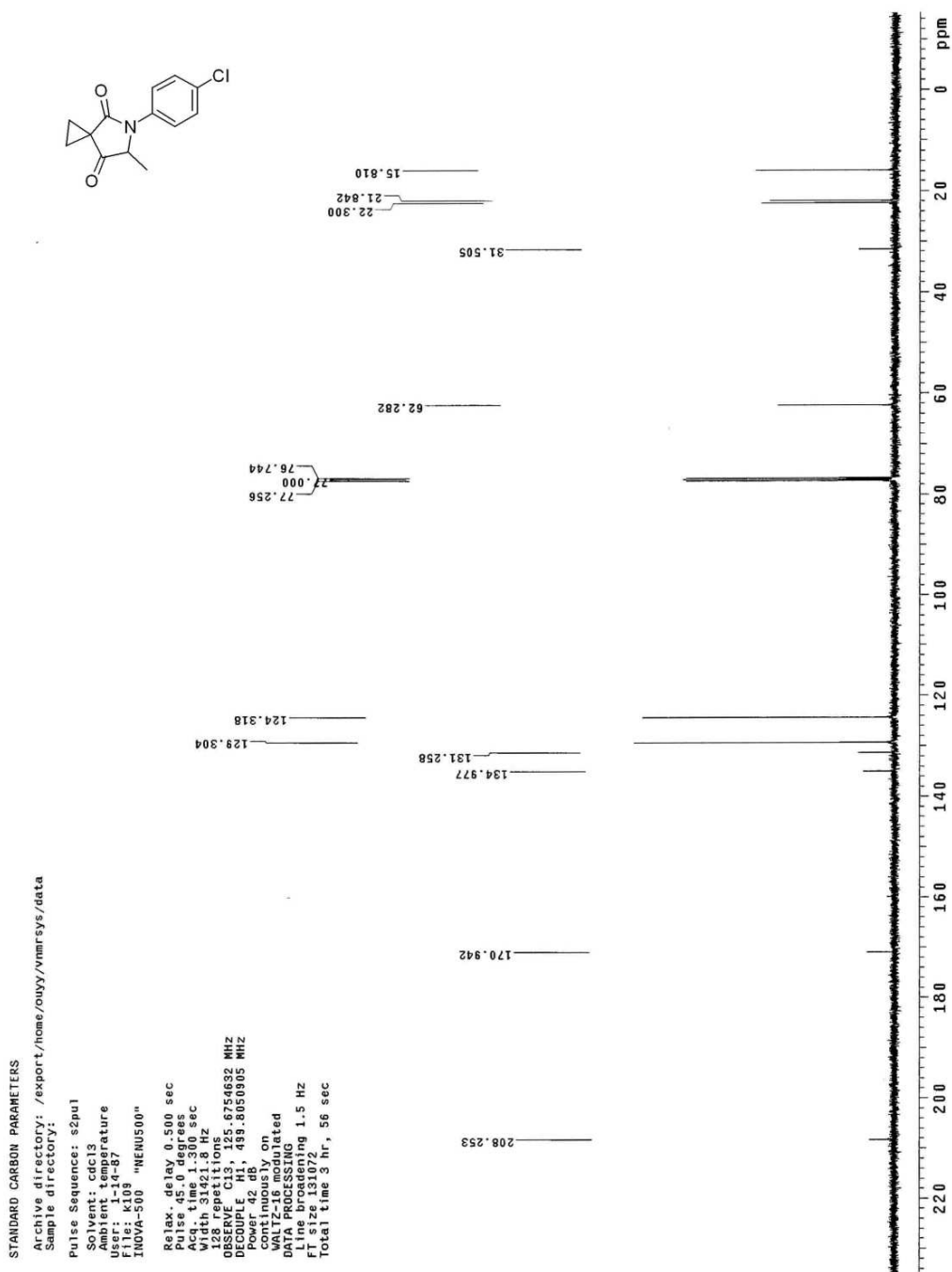


CCOC(=O)c1ccc(cc1)N2C(=O)C3CC3C(=O)N2

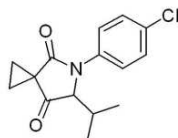
Compound 2o



Compound 2o



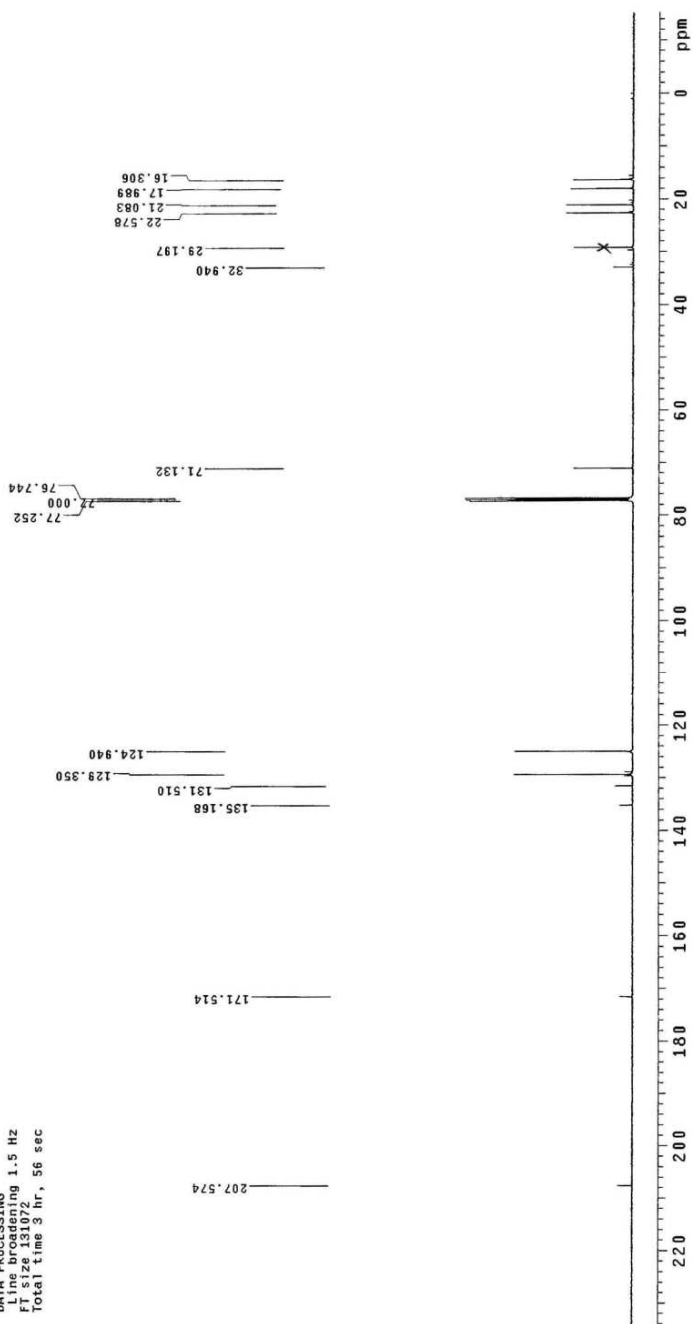
Compound 2p



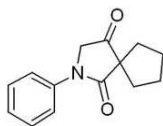
STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouss/vnmr/sys/data
Sample directory:

Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: k832
File: k832
INOVA-500 "MENU500"

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 0.500 sec
Width 31421.8 Hz
4928 repetitions
OBSERVE C13, 125.6754618 MHz
DECOUPLE H1, 499.8050305 MHz
Pwr 42 db
continuously on
WALTZ-16 modulated
DATA PROCESSING
1.5 Hz
FT size 131072
Total time 3 hr, 56 sec



Compound 2s



STANDARD PROTON PARAMETERS

Archive directory: /export/home/ouyy/vmrsys/data

Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

File: v856

INOVA-500 "MNU500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 8291.0 Hz

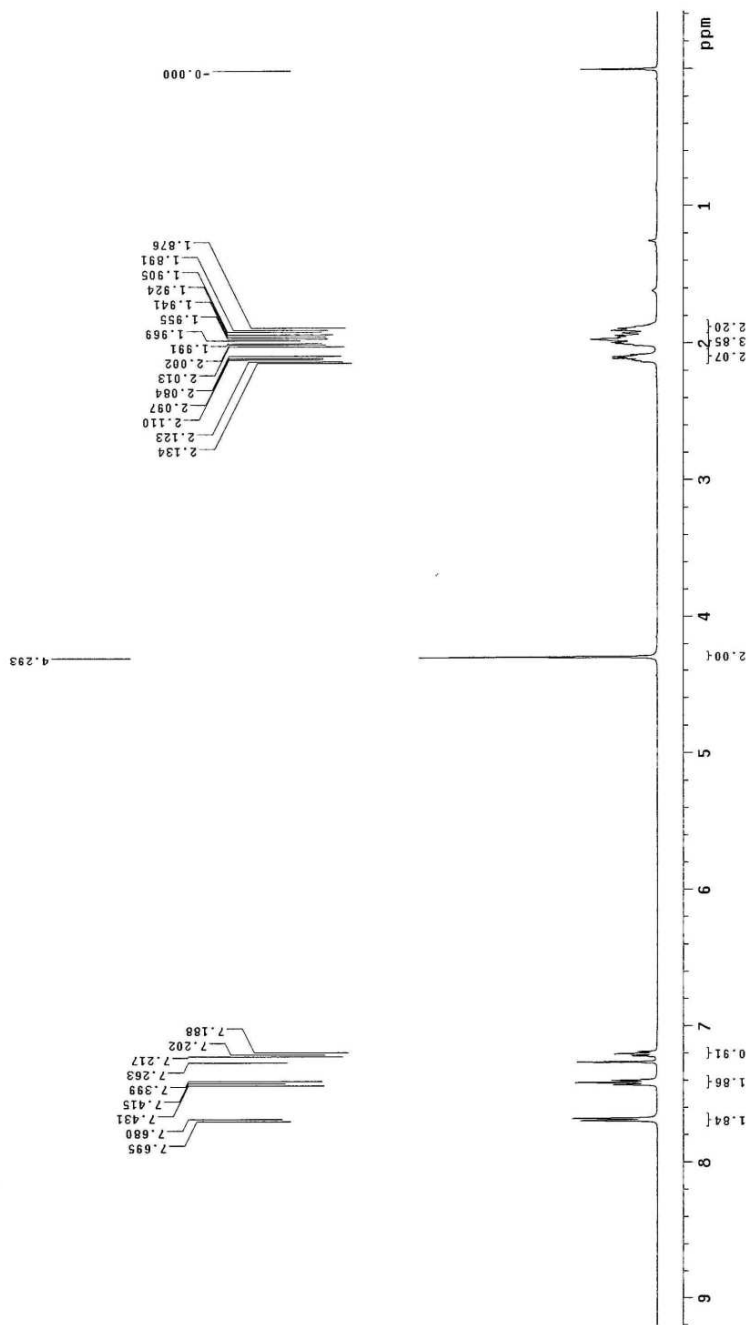
Observed time 1.892 sec

OBSERVED F1 499.8025902 MHZ

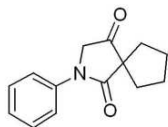
DATA PROCESSING

FT size 65536

Total time 0 min, 23 sec



Compound 2s

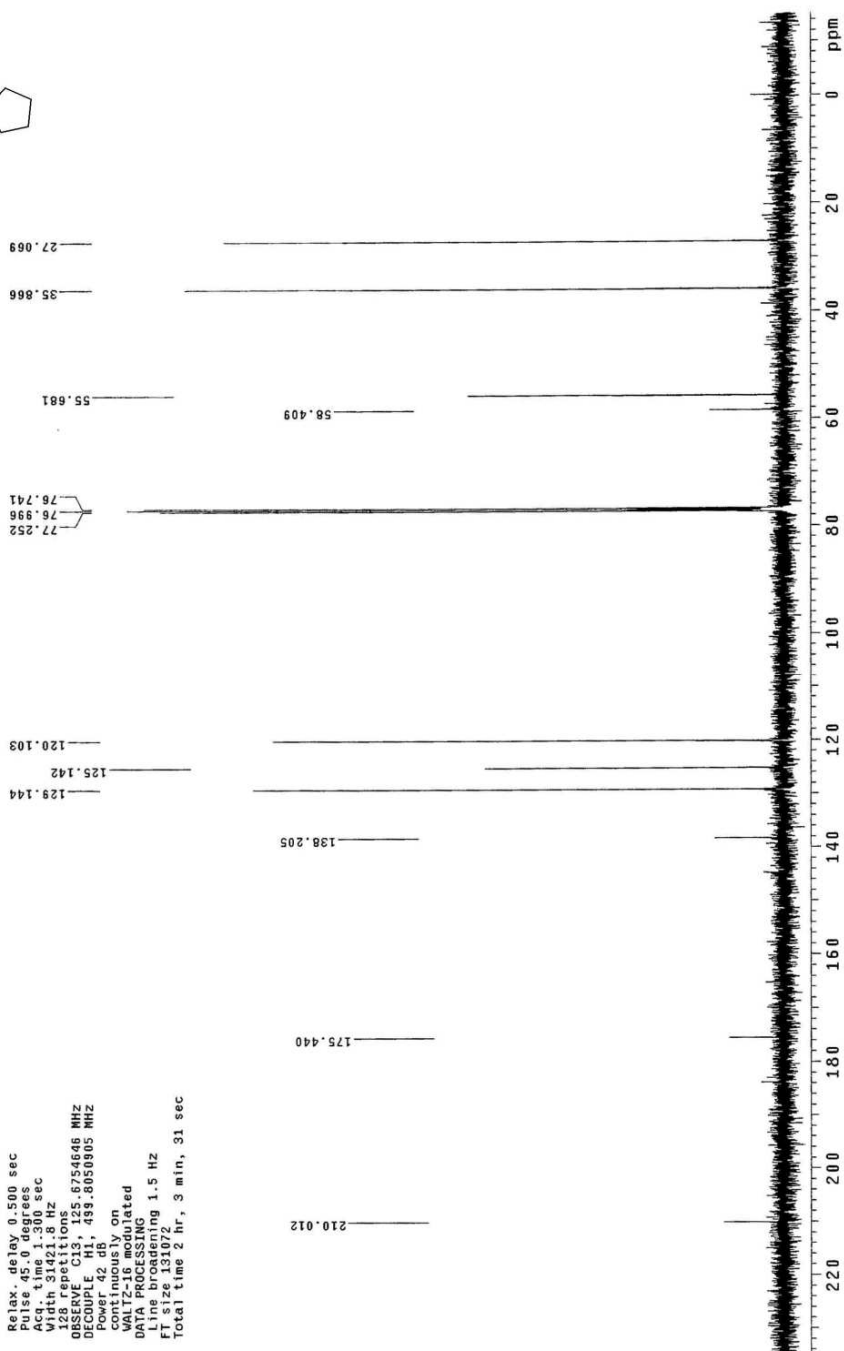


STANDARD CARBON PARAMETERS

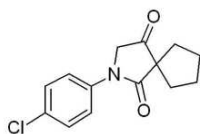
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Sample directory:

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Date: 02-14-07
File: v857
INOVA-500 "NMR500"

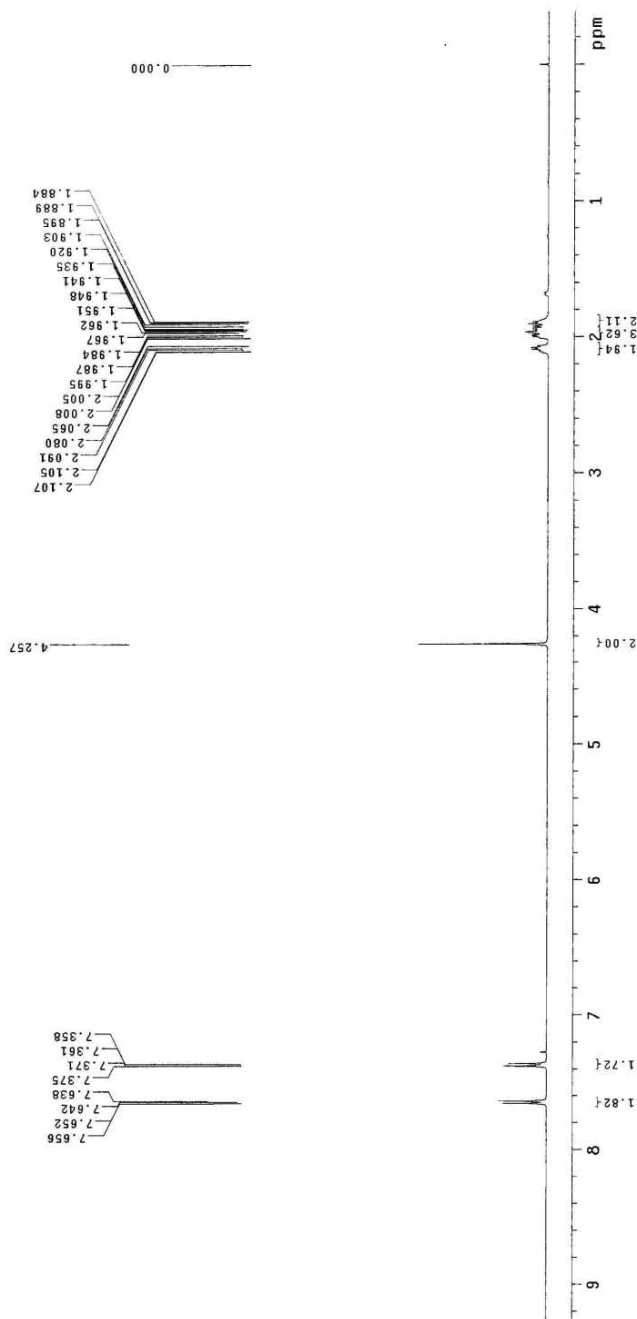
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.500 hr
Width 31921.8 Hz
128 repetitions
OBSERVE C13, 125.6754646 MHz
DECOUPLE H1, 499.8050905 MHz
Power 12.0 dB
P1 12.0 dB
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
F1 size 131072
Total time 2 hr, 3 min, 31 sec



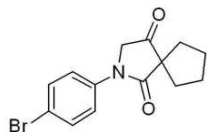
Compound 2t



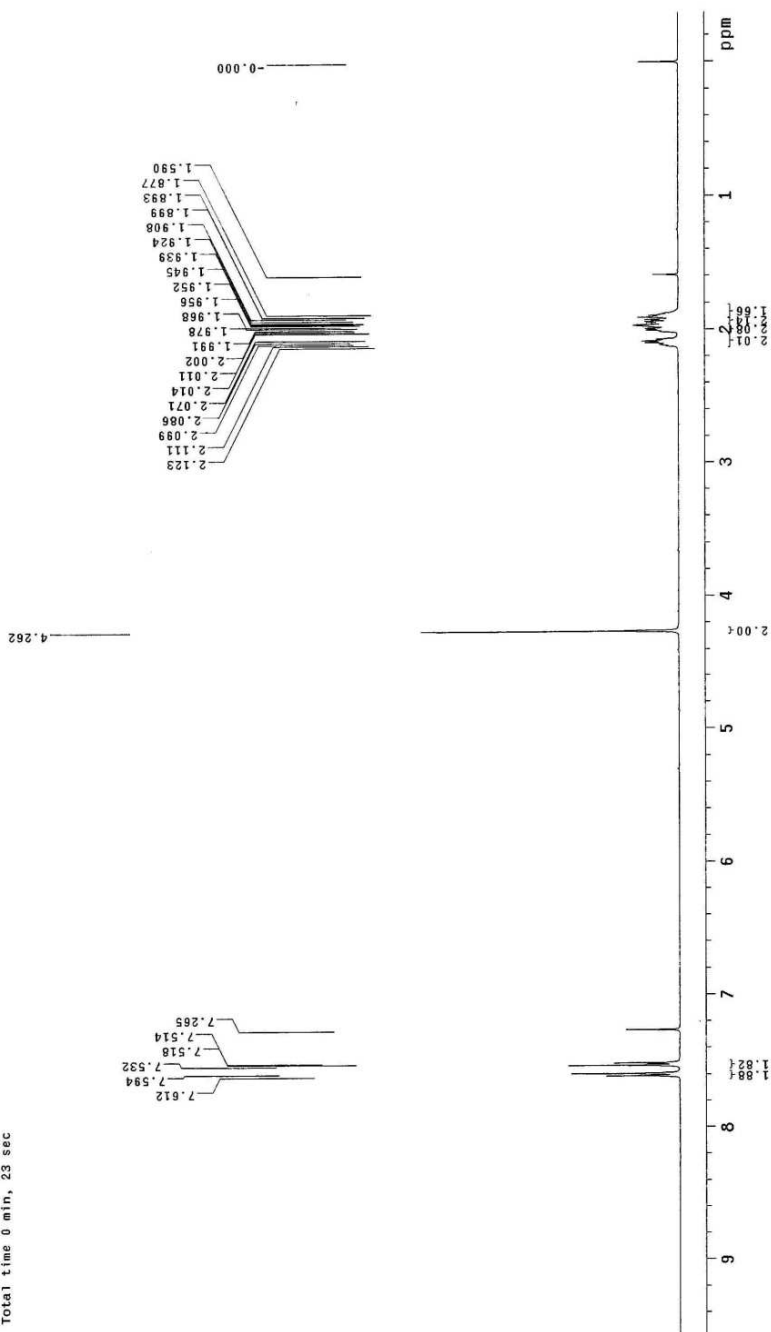
STANDARD PROTON PARAMETERS
 Archive directory: /export/home/ouyy/vnmrSYS/data
 Sample directory:
 Pulse Sequence: s2p01
 Solvent: CDCl3
 Ambient temperature
 File: p108
 INOVA-500 "MENU500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 Width 8889.8 Hz
 Spectroscopy
 Frequency 499.8025862 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 23 sec



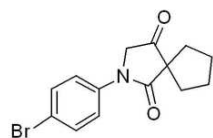
Compound 2u



STANDARD PROTON PARAMETERS
 Archive directory: /export/home/ouyy/vnmrsws/data
 Sample directory:
 Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 File: d893
 INOVA-500 "NMR500"
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 1.892 sec
 Width 8578.2 Hz
 Spectrometer
 OBSERVE H1 499.8025891 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 23 sec



Compound 2u



STANDARD CARBON PARAMETERS

Archive directory: /export/home/ouyy/vnmrfsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

User: 1-14-87

File: d834

INOVA-500 "MNU500"

Relax. delay 0.500 sec

Pulse delay 0.000 sec

Acq time 1.300 sec

Width 31421.8 Hz

3392 repetitions

Observed C13, 125.6754656 MHz

Observed H1, 400.1463400 MHz

Power 42 dB, 433.8058905 MHz

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.5 Hz

FT size 131072

Total time 3 hr, 56 sec

