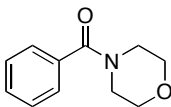


**SUPPORTING INFORMATION**

**Title:** Simple Synthesis of Amides and Weinreb Amides Using PPh<sub>3</sub> or Polymer-Supported PPh<sub>3</sub> and Iodine

**Author(s):** Amit Kumar, Hari Kiran Akula, Mahesh K. Lakshman\*

**Ref. No.:** O200901420

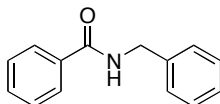
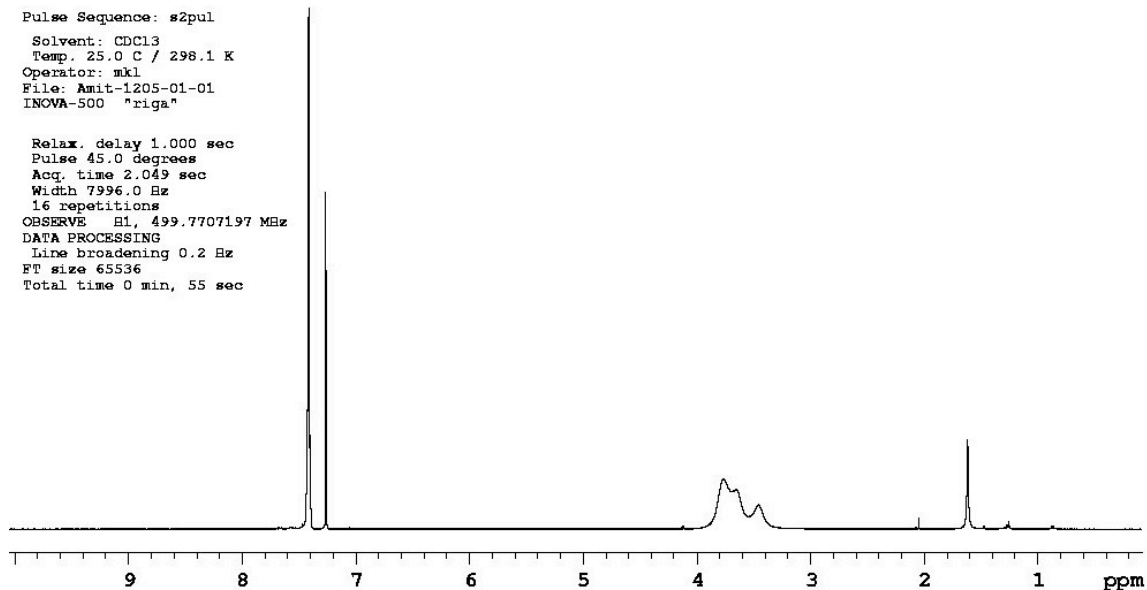


Std proton  
Amit-1205-01-01

Archive directory:  
Sample directory:

Pulse Sequence: s2pul  
Solvent: CDCl3  
Temp: 25.0 C / 298.1 K  
Operator: mkl  
File: Amit-1205-01-01  
INOVA-500 "riga"

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 2.049 sec  
Width 7996.0 Hz  
16 repetitions  
OBSERVE H1, 499.7707197 MHz  
DATA PROCESSING  
Line broadening 0.2 Hz  
FT size 65536  
Total time 0 min, 55 sec

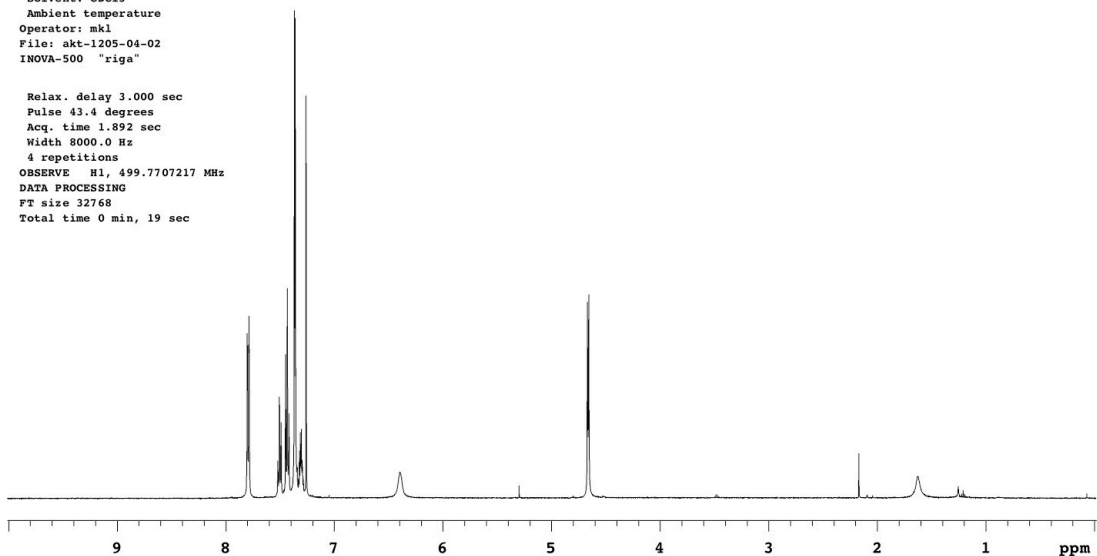


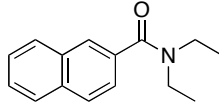
akt-1205-04-02

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

Pulse Sequence: s2pul  
Solvent: CDCl3  
Ambient temperature  
Operator: mkl  
File: akt-1205-04-02  
INOVA-500 "riga"

Relax. delay 3.000 sec  
Pulse 43.4 degrees  
Acq. time 1.892 sec  
Width 8000.0 Hz  
4 repetitions  
OBSERVE H1, 499.7707217 MHz  
DATA PROCESSING  
FT size 32768  
Total time 0 min, 19 sec



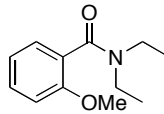
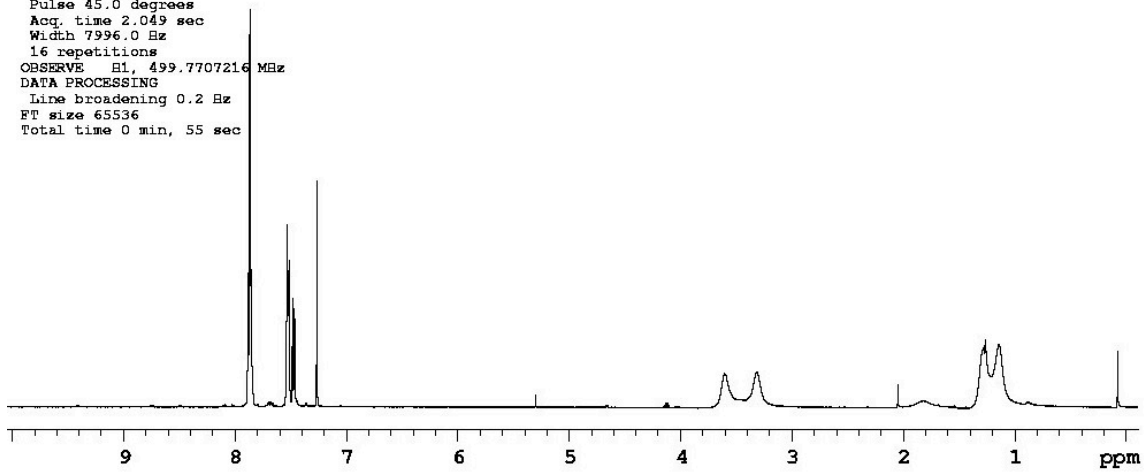


akt1205-01-04

Archive directory:  
Sample directory:

Pulse Sequence: s2pul  
Solvent: CDCl3  
Temp. 25.0 C / 298.1 K  
Operator: mkl  
File: akt1205-01-04  
INOVA-500 "riga"

Relax. delay 1.000 sec  
Pulse 45.0 degrees  
Acq. time 2.049 sec  
Width 7996.0 Hz  
16 repetitions  
OBSERVE E1, 499.7707216 MHz  
DATA PROCESSING  
Line broadening 0.2 Hz  
FT size 65536  
Total time 0 min, 55 sec

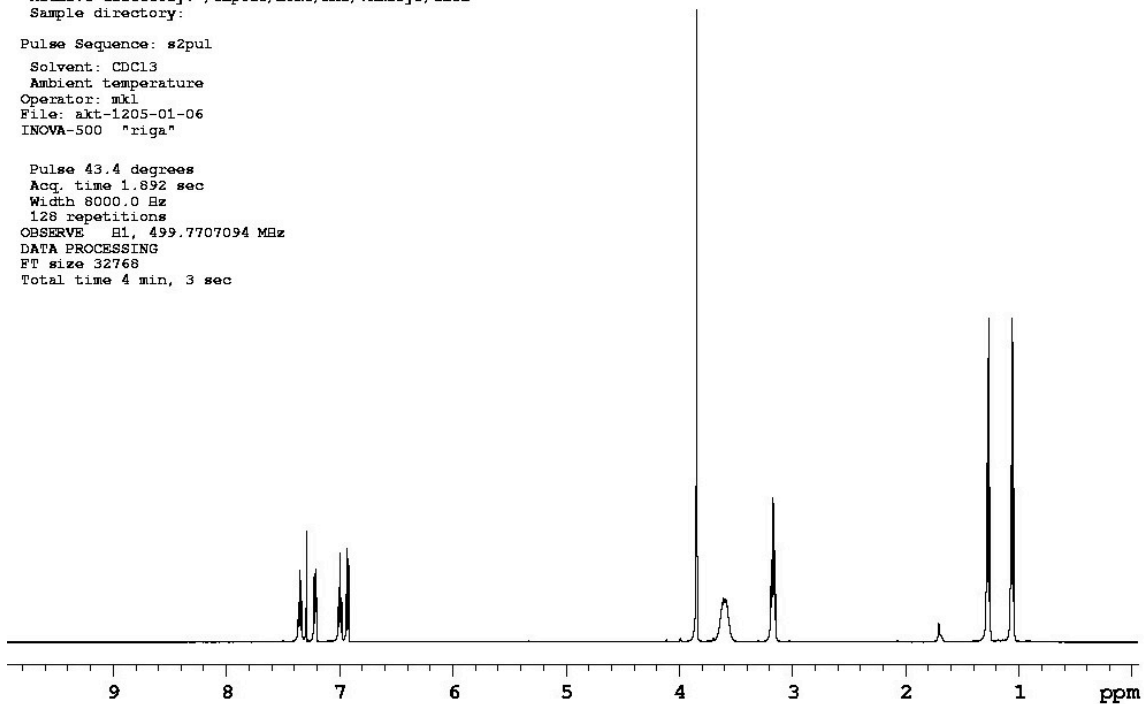


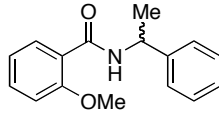
akt-1205-01-06

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

Pulse Sequence: s2pul  
Solvent: CDCl3  
Ambient temperature  
Operator: mkl  
File: akt-1205-01-06  
INOVA-500 "riga"

Pulse 43.4 degrees  
Acq. time 1.892 sec  
Width 8000.0 Hz  
128 repetitions  
OBSERVE E1, 499.7707094 MHz  
DATA PROCESSING  
FT size 32768  
Total time 4 min, 3 sec





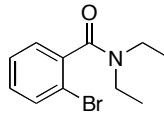
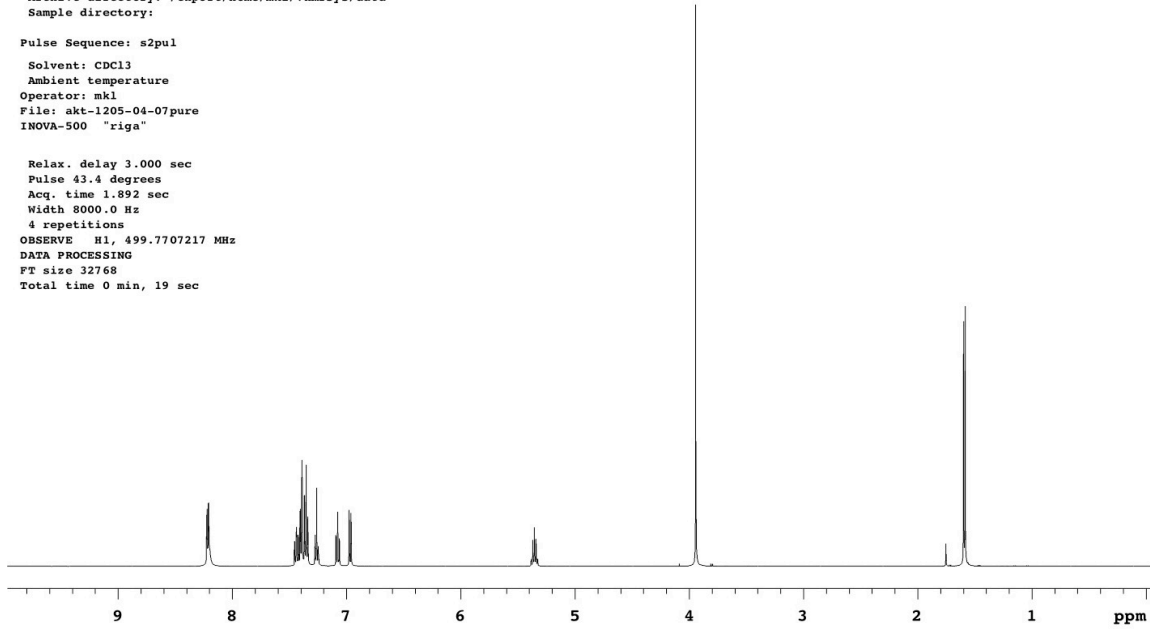
akt-1205-04-07pure

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

Pulse Sequence: s2pul

Solvent: CDCl3  
Ambient temperature  
Operator: mkl  
File: akt-1205-04-07pure  
INOVA-500 "riga"

Relax. delay 3.000 sec  
Pulse 43.4 degrees  
Acq. time 1.892 sec  
Width 8000.0 Hz  
4 repetitions  
OBSERVE H1, 499.7707217 MHz  
DATA PROCESSING  
FT size 32768  
Total time 0 min, 19 sec



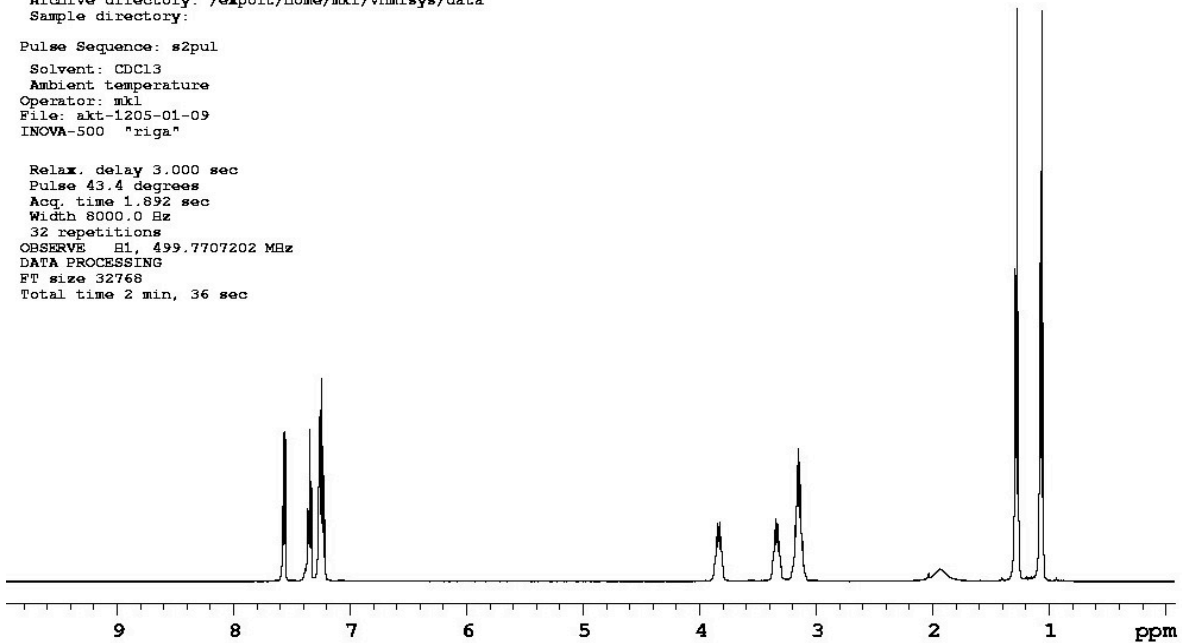
akt-1205-01-09

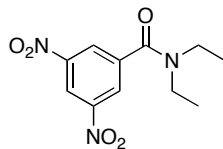
Archive directory: /export/home/mkl/vnmrsys/data  
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl3  
Ambient temperature  
Operator: mkl  
File: akt-1205-01-09  
INOVA-500 "riga"

Relax. delay 3.000 sec  
Pulse 43.4 degrees  
Acq. time 1.892 sec  
Width 8000.0 Hz  
32 repetitions  
OBSERVE H1, 499.7707202 MHz  
DATA PROCESSING  
FT size 32768  
Total time 2 min, 36 sec





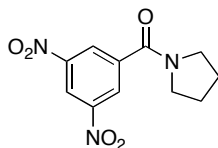
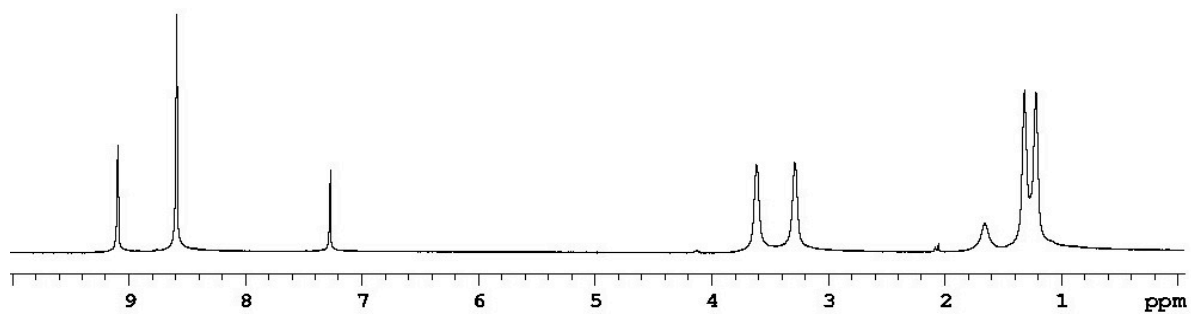
akt-1205-01-10

Archive directory: /export/home/mkl/vnmrSYS/data  
 Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl3  
 Ambient temperature  
 Operator: mkl  
 File: akt-1205-01-10  
 INOVA-500 "riga"

Relax. delay 3.000 sec  
 Pulse 43.4 degrees  
 Acq. time 1.892 sec  
 Width 8000.0 Hz  
 32 repetitions  
 OBSERVE H1, 499.7707202 MHz  
 DATA PROCESSING  
 FT size 32768  
 Total time 2 min, 36 sec



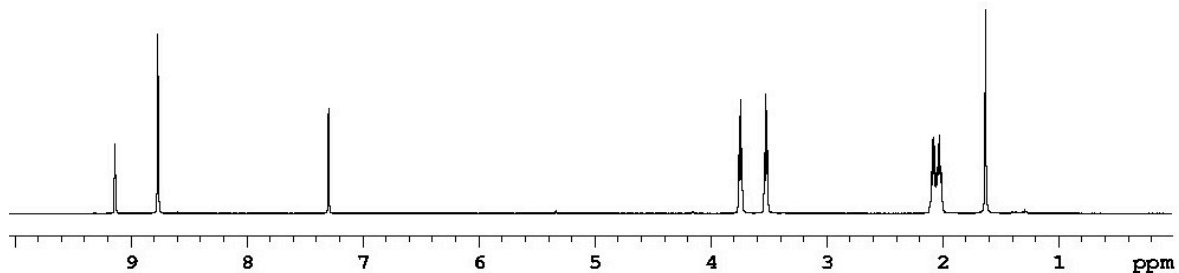
akt-1205-01-13

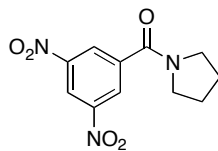
Archive directory:  
 Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl3  
 Temp. 25.0 C / 298.1 K  
 Operator: mkl  
 File: akt-1205-01-13  
 INOVA-500 "riga"

Relax. delay 1.000 sec  
 Pulse 45.0 degrees  
 Acq. time 2.049 sec  
 Width 7996.0 Hz  
 16 repetitions  
 OBSERVE H1, 499.7707096 MHz  
 DATA PROCESSING  
 Line broadening 0.2 Hz  
 FT size 65536  
 Total time 0 min, 55 sec



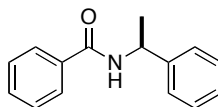
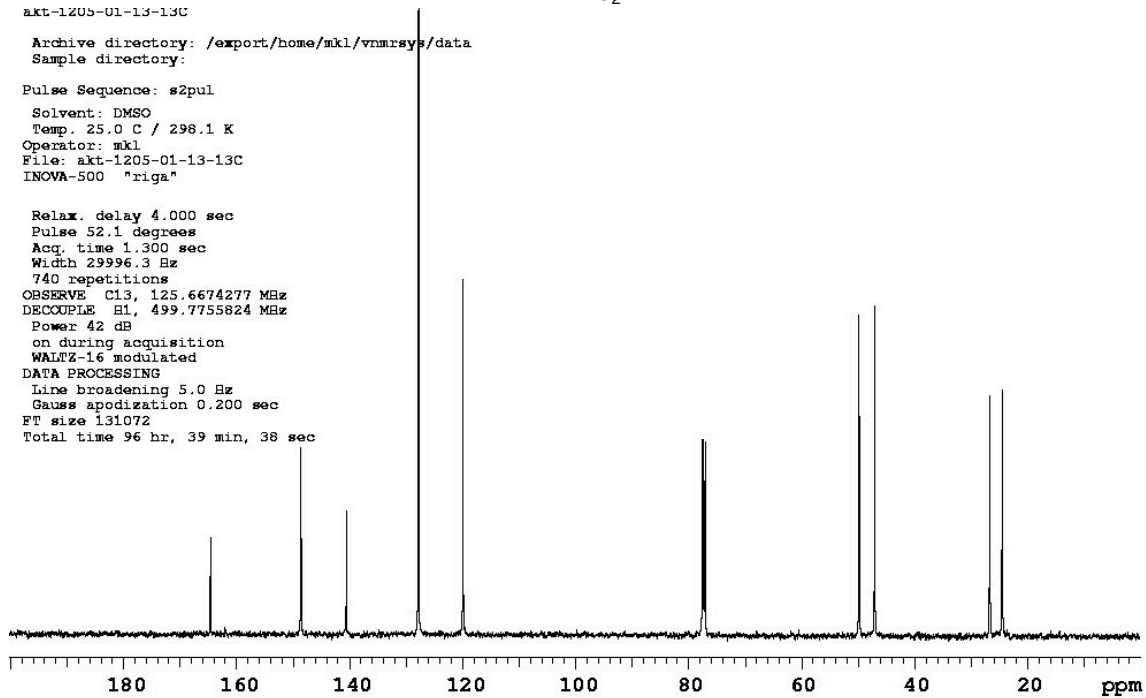


akt-1205-01-13-13C

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

Pulse Sequence: s2pul  
 Solvent: DMSO  
 Temp. 25.0 C / 298.1 K  
 Operator: mkl  
 File: akt-1205-01-13-13C  
 INOVA-500 "riga"

Relax. delay 4.000 sec  
 Pulse 52.1 degrees  
 Acq. time 1.300 sec  
 Width 29996.3 Hz  
 740 repetitions  
 OBSERVE C13, 125.6674277 MHz  
 DECOUPLE H1, 499.7755824 MHz  
 Power 42 dB  
 on during acquisition  
 WALTZ-16 modulated  
 DATA PROCESSING  
 Line broadening 5.0 Hz  
 Gauss apodization 0.200 sec  
 FT size 131072  
 Total time 96 hr, 39 min, 38 sec

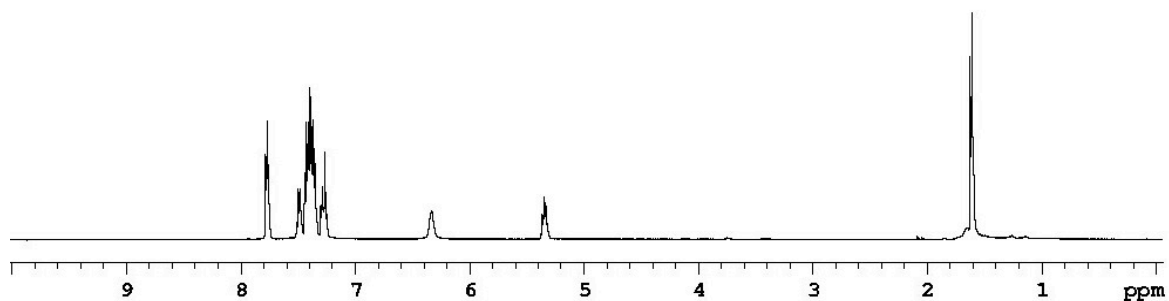


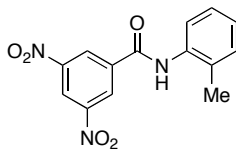
akt-1205-01-16

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

Pulse Sequence: s2pul  
 Solvent: CDCl3  
 Ambient temperature  
 Operator: mkl  
 File: akt-1205-01-16  
 INOVA-500 "riga"

Relax. delay 3.000 sec  
 Pulse 43.4 degrees  
 Acq. time 1.892 sec  
 Width 8000.0 Hz  
 32 repetitions  
 OBSERVE H1, 499.7707212 MHz  
 DATA PROCESSING  
 FT size 32768  
 Total time 2 min, 36 sec





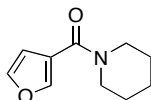
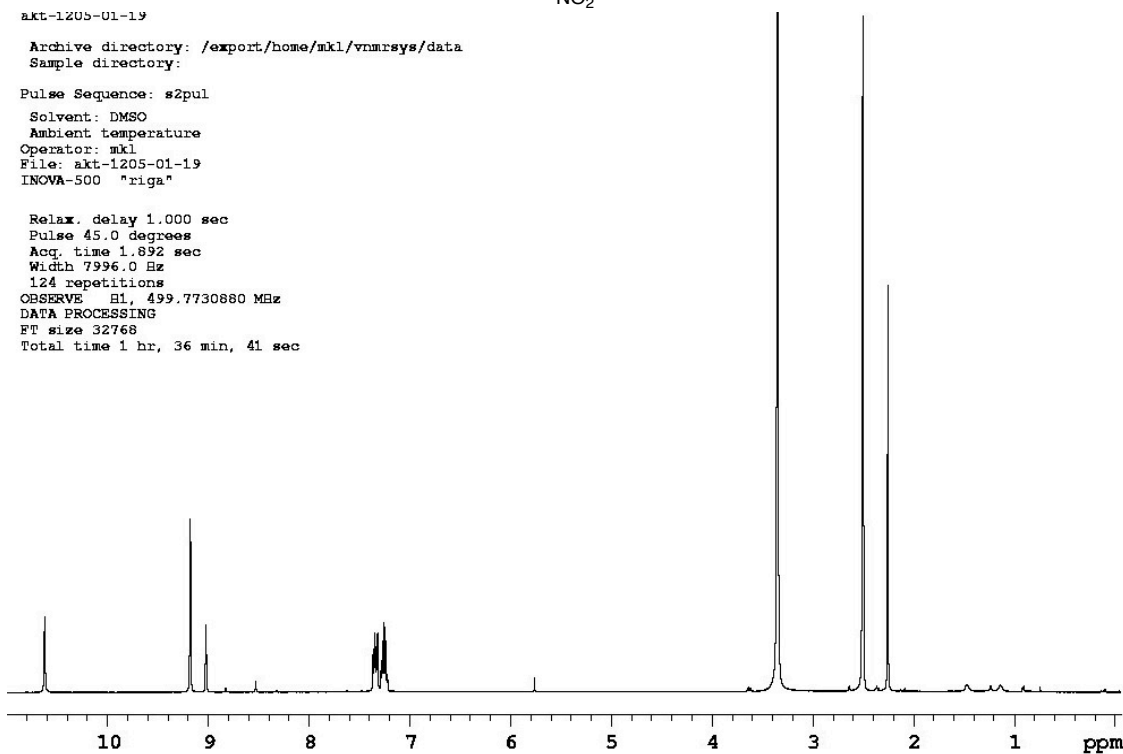
akt-1205-01-19

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

Pulse Sequence: s2pul

Solvent: DMSO  
 Ambient temperature  
 Operator: mkl  
 File: akt-1205-01-19  
 INOVA-500 "riga"

Relax. delay 1.000 sec  
 Pulse 45.0 Degrees  
 Acq. time 1.892 sec  
 Width 7996.0 Hz  
 124 repetitions  
 OBSERVE H1, 499.7730880 MHz  
 DATA PROCESSING  
 FT size 32768  
 Total time 1 hr, 36 min, 41 sec



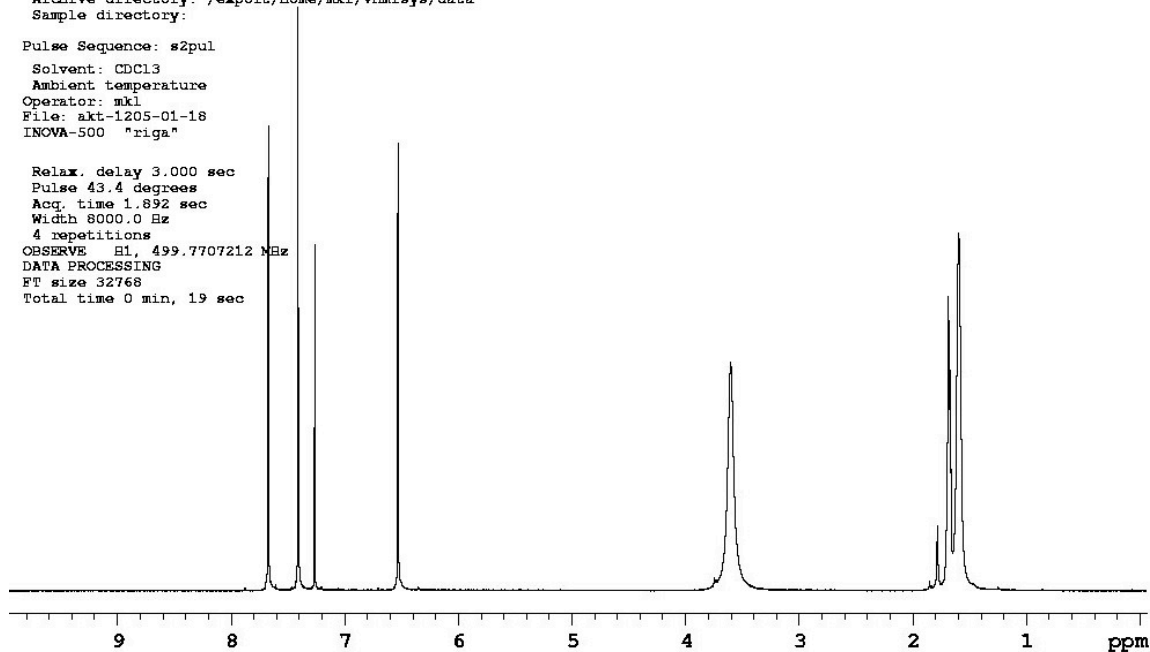
akt-1205-01-18

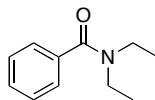
Archive directory: /export/home/mkl/vnmrsys/data  
 Sample directory:

Pulse Sequence: s2pul

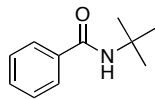
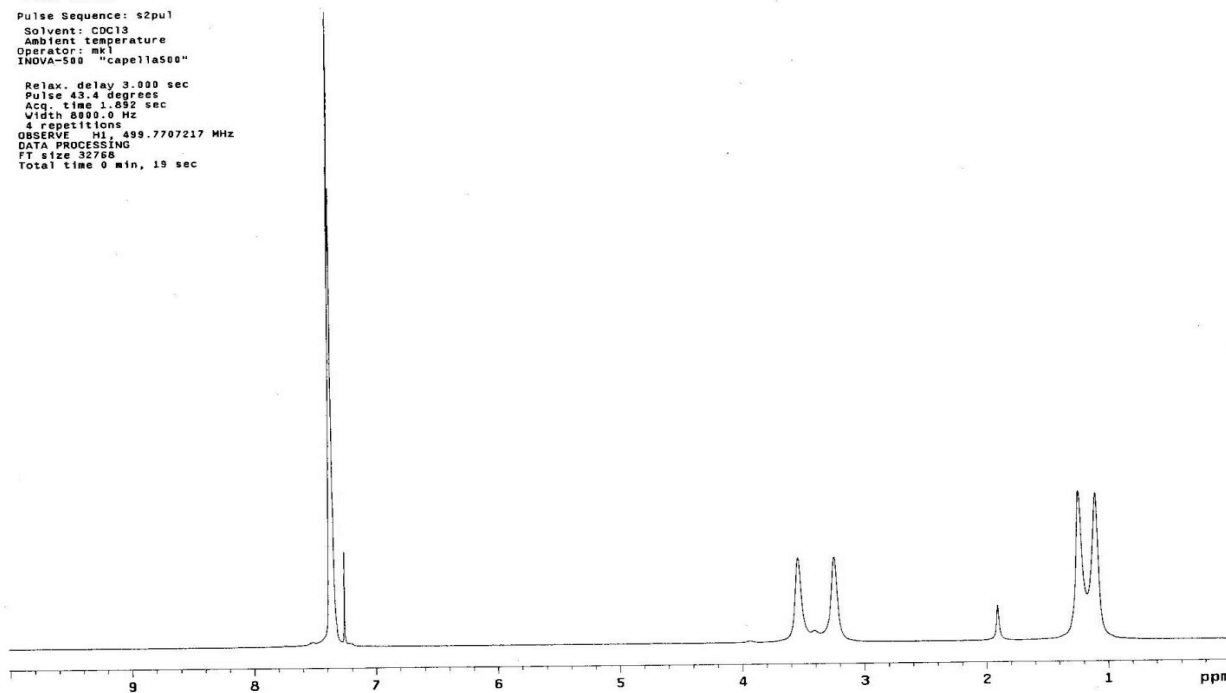
Solvent: CDCl3  
 Ambient temperature  
 Operator: mkl  
 File: akt-1205-01-18  
 INOVA-500 "riga"

Relax. delay 3.000 sec  
 Pulse 43.4 degrees  
 Acq. time 1.892 sec  
 Width 8000.0 Hz  
 4 repetitions  
 OBSERVE H1, 499.7707212 MHz  
 DATA PROCESSING  
 FT size 32768  
 Total time 0 min, 19 sec

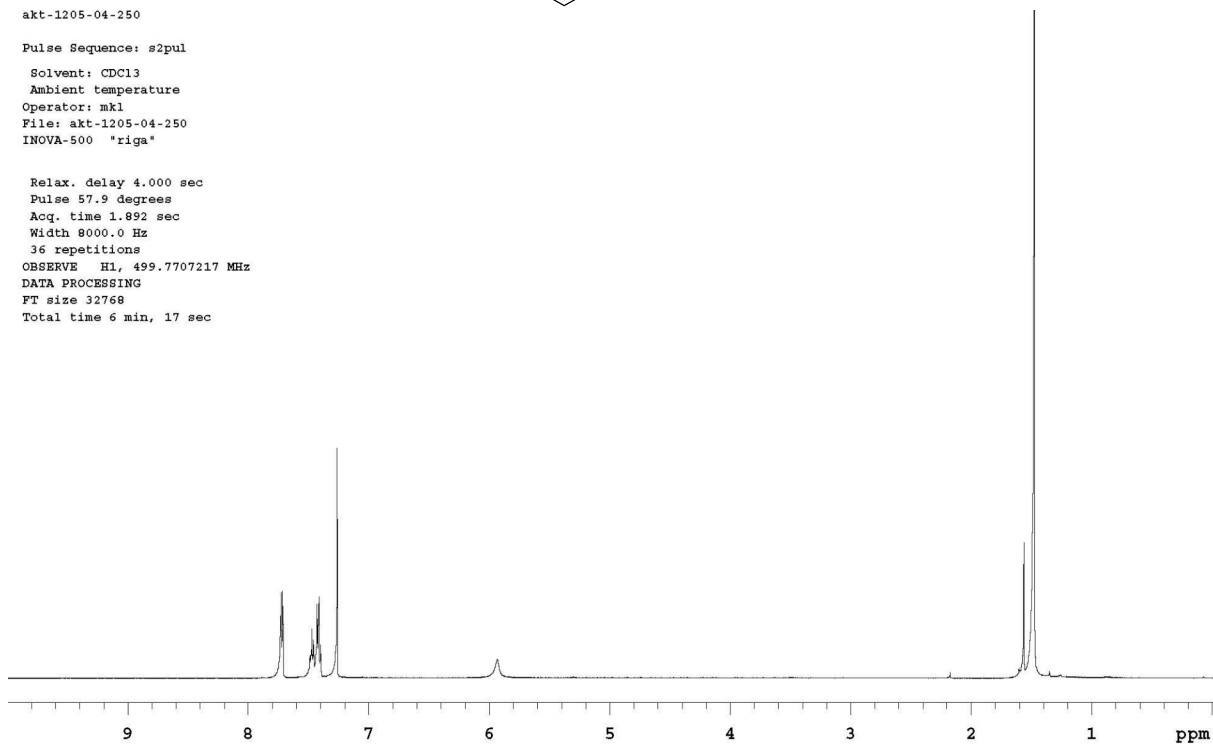




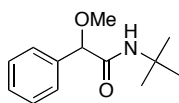
akt-1205-01-38  
Archive directory: /export/home/mkl/vnarsys/data  
File: PROTON  
Pulse Sequence: s2pu1  
Solvent: CDCl3  
Ambient temperature  
Operator: mkl  
INOVA-500 "capella500"  
Relax. delay 3.000 sec  
Pulse 43.4 degrees  
Acq. time 1.892 sec  
Width 8000.0 Hz  
4 repetitions  
OBSERVE H1, 499.7707217 MHz  
DATA PROCESSING  
FT size 32768  
Total time 0 min, 19 sec



akt-1205-04-250  
Pulse Sequence: s2pul  
Solvent: CDCl3  
Ambient temperature  
Operator: mkl  
File: akt-1205-04-250  
INOVA-500 "riga"  
Relax. delay 4.000 sec  
Pulse 57.9 degrees  
Acq. time 1.892 sec  
Width 8000.0 Hz  
36 repetitions  
OBSERVE H1, 499.7707217 MHz  
DATA PROCESSING  
FT size 32768  
Total time 6 min, 17 sec







AKT-1205-04-246-pure

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

Operator: mkl

File: AKT-1205-04-246-pure

INOVA-500 "riga"

Relax. delay 4.000 sec

Pulse 57.9 degrees

Acq. time 1.892 sec

Width 8000.0 Hz

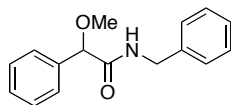
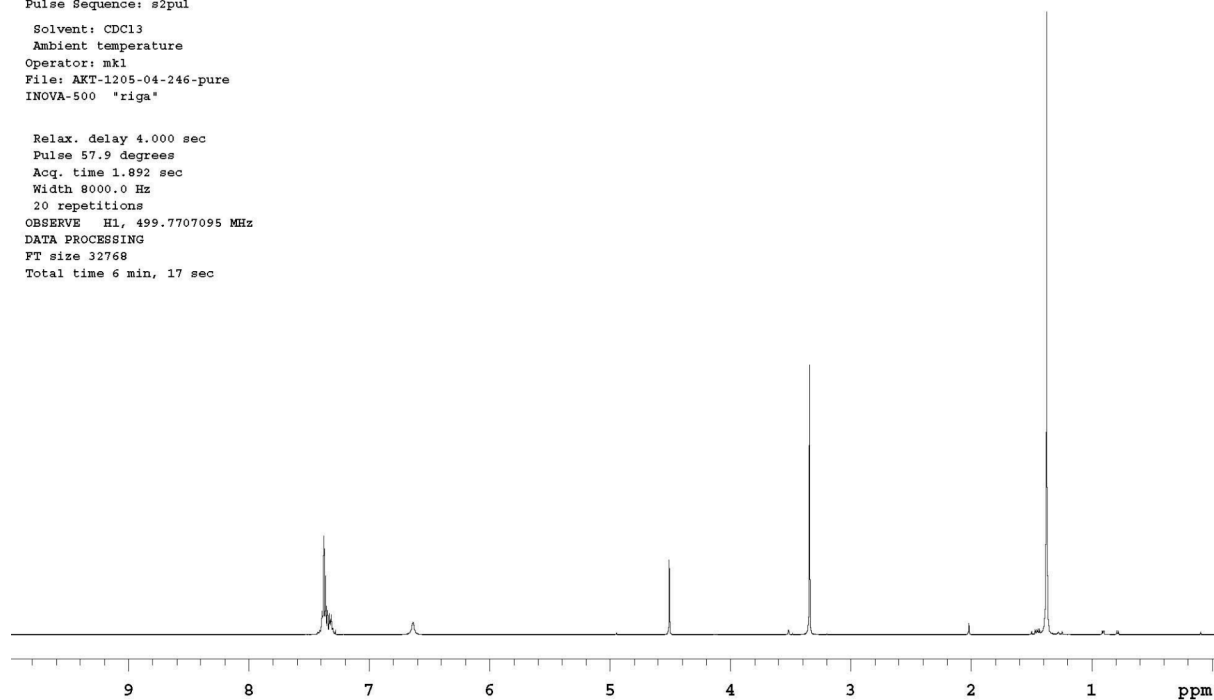
20 repetitions

OBSERVE H1, 499.7707095 MHz

DATA PROCESSING

FT size 32768

Total time 6 min, 17 sec



AKT-1205-04-247

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

Operator: mkl

File: AKT-1205-04-247

INOVA-500 "riga"

Relax. delay 4.000 sec

Pulse 57.9 degrees

Acq. time 1.892 sec

Width 8000.0 Hz

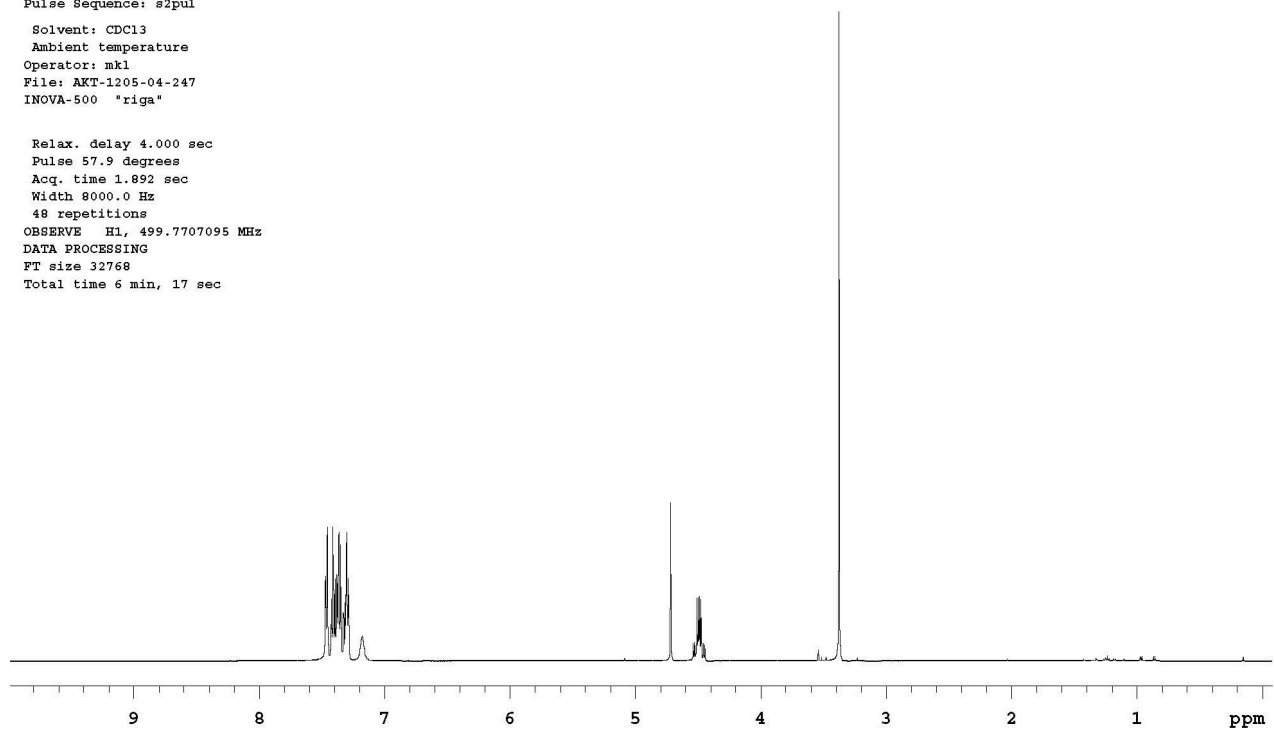
48 repetitions

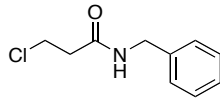
OBSERVE H1, 499.7707095 MHz

DATA PROCESSING

FT size 32768

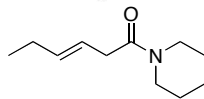
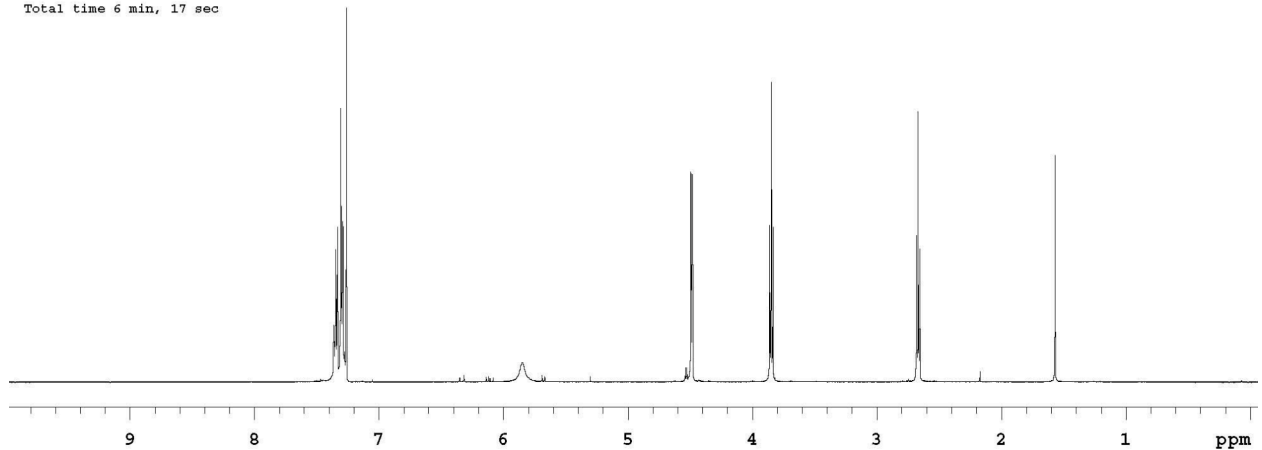
Total time 6 min, 17 sec





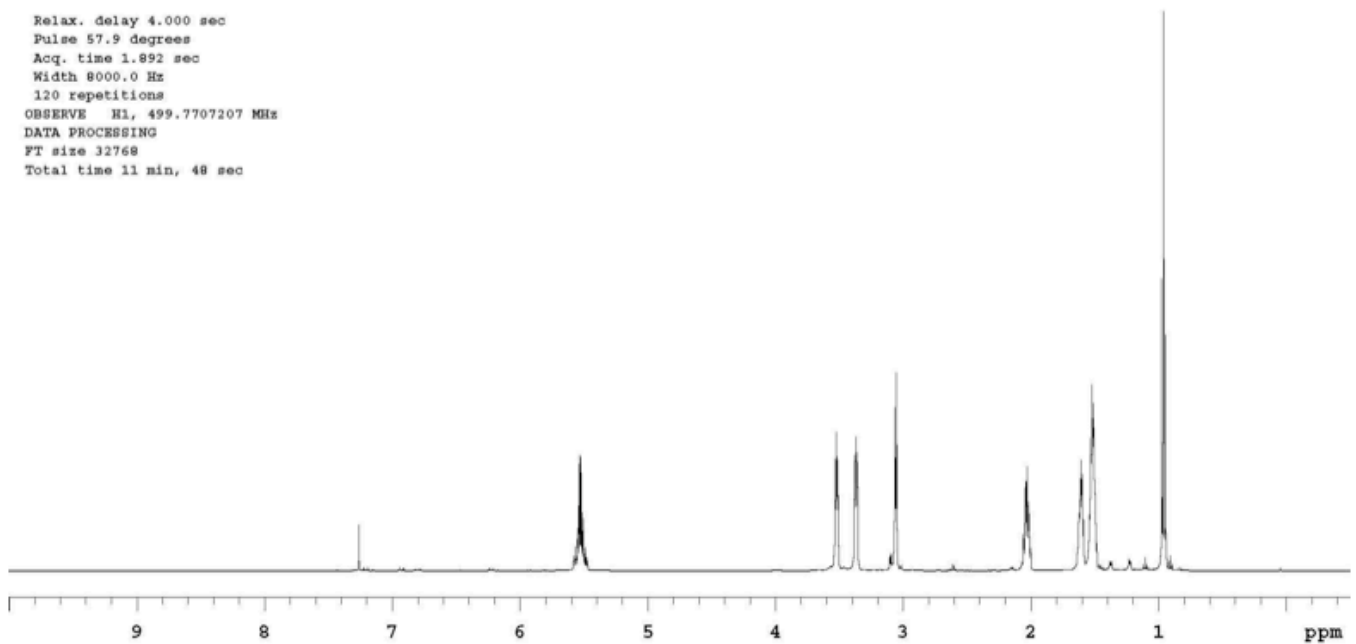
akt-1205-04-245purefinal  
Pulse Sequence: s2pul  
Solvent: CDCl3  
Ambient temperature  
Operator: mk1  
File: akt-1205-04-245purefinal  
INOVA-500 "riga"

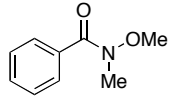
Relax. delay 4.000 sec  
Pulse 57.9 degrees  
Acq. time 1.892 sec  
Width 8000.0 Hz  
36 repetitions  
OBSERVE H1, 499.7707222 MHz  
DATA PROCESSING  
FT size 32768  
Total time 6 min, 17 sec



1203-HK-02-18  
Pulse Sequence: s2pul  
Solvent: CDCl3  
Ambient temperature  
Operator: mk1  
File: 1203-HK-02-18  
INOVA-500 "riga"

Relax. delay 4.000 sec  
Pulse 57.9 degrees  
Acq. time 1.892 sec  
Width 8000.0 Hz  
120 repetitions  
OBSERVE H1, 499.7707207 MHz  
DATA PROCESSING  
FT size 32768  
Total time 11 min, 48 sec



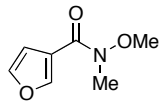
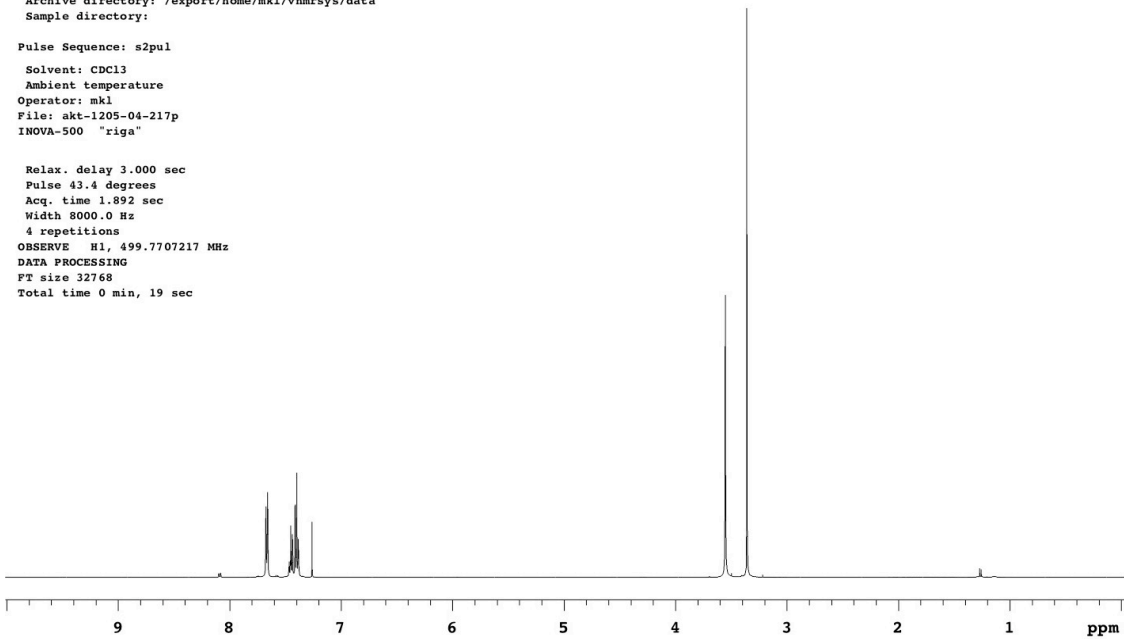


akt-1205-04-217p

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

Pulse Sequence: s2pul  
Solvent: CDCl3  
Ambient temperature  
Operator: mkl  
File: akt-1205-04-217p  
INOVA-500 "riga"

Relax. delay 3.000 sec  
Pulse 43.4 degrees  
Acq. time 1.892 sec  
Width 8000.0 Hz  
4 repetitions  
OBSERVE H1, 499.7707217 MHz  
DATA PROCESSING  
FT size 32768  
Total time 0 min, 19 sec

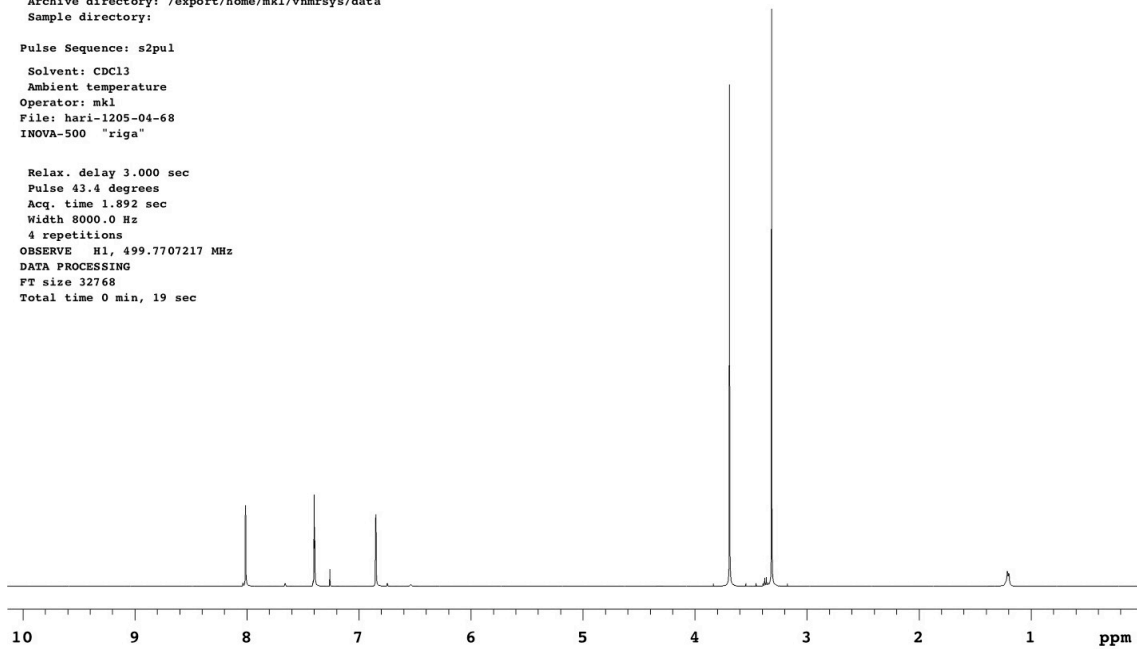


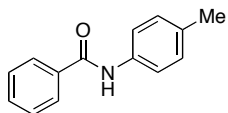
hari-1205-04-68

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

Pulse Sequence: s2pul  
Solvent: CDCl3  
Ambient temperature  
Operator: mkl  
File: hari-1205-04-68  
INOVA-500 "riga"

Relax. delay 3.000 sec  
Pulse 43.4 degrees  
Acq. time 1.892 sec  
Width 8000.0 Hz  
4 repetitions  
OBSERVE H1, 499.7707217 MHz  
DATA PROCESSING  
FT size 32768  
Total time 0 min, 19 sec





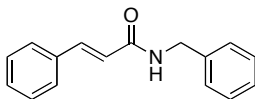
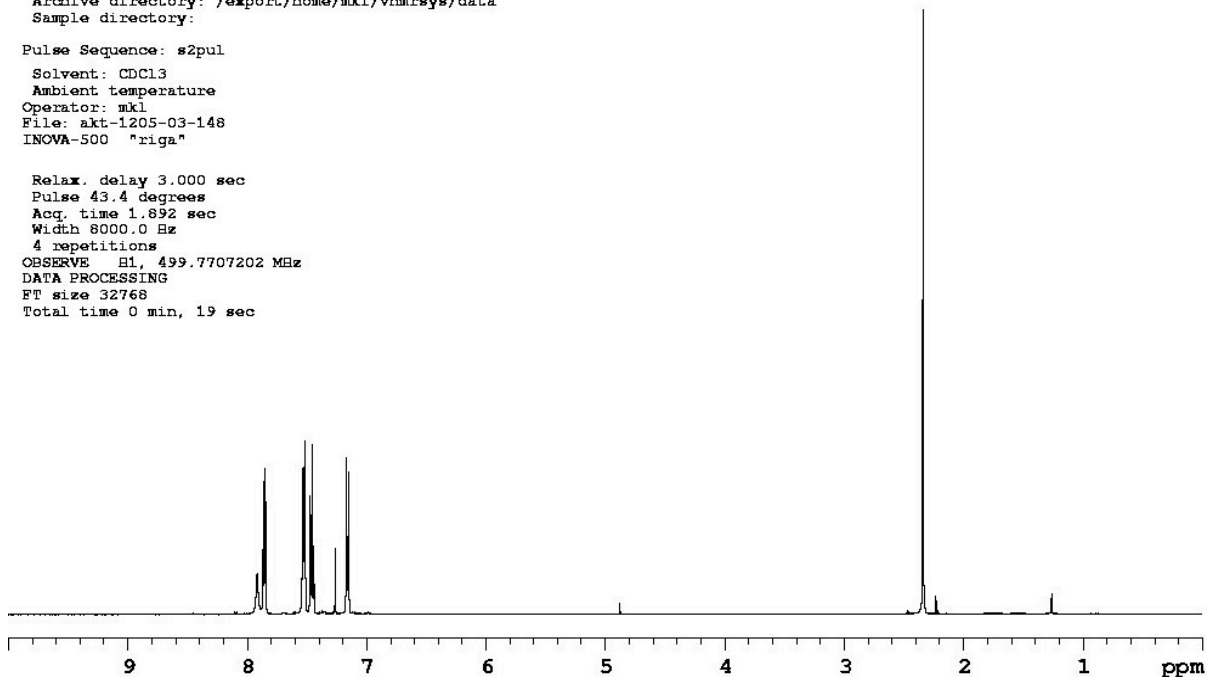
akt-1205-03-148

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

Pulse Sequence: s2pul

Solvent: CDCl3  
Ambient temperature  
Operator: mkl  
File: akt-1205-03-148  
INOVA-500 "riga"

Relax. delay 3.000 sec  
Pulse 43.4 degrees  
Acq. time 1.892 sec  
Width 8000.0 Hz  
4 repetitions  
OBSERVE H1, 499.7707202 MHz  
DATA PROCESSING  
FT size 32768  
Total time 0 min, 19 sec



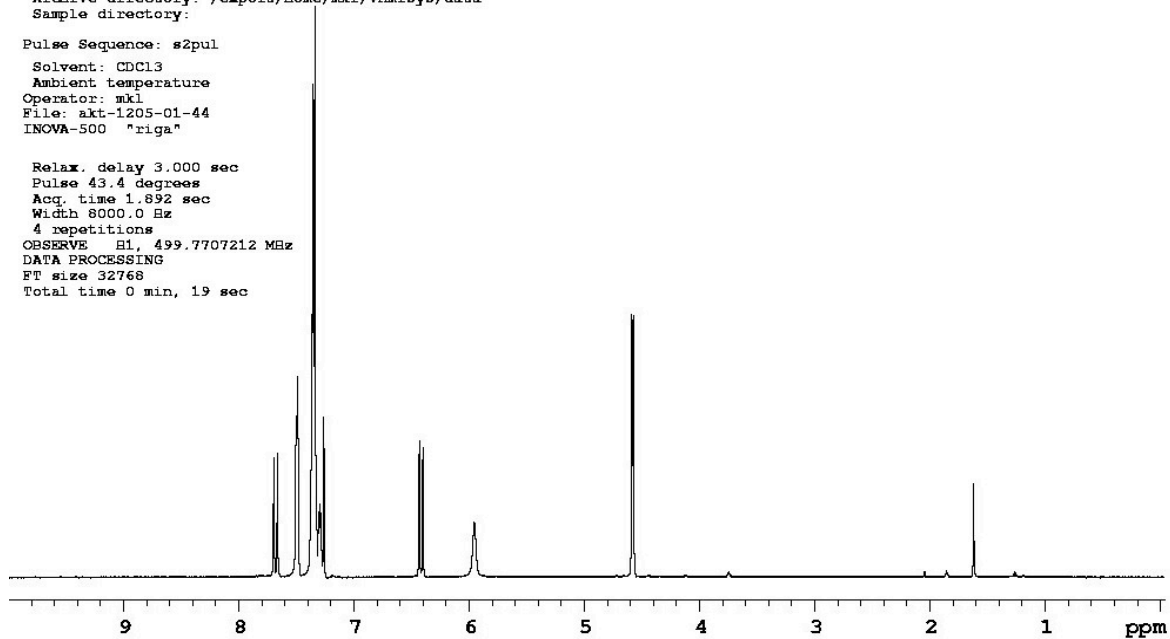
akt-1205-01-44

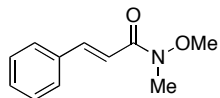
Archive directory: /export/home/mkl/vnmrsys/data  
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl3  
Ambient temperature  
Operator: mkl  
File: akt-1205-01-44  
INOVA-500 "riga"

Relax. delay 3.000 sec  
Pulse 43.4 degrees  
Acq. time 1.892 sec  
Width 8000.0 Hz  
4 repetitions  
OBSERVE H1, 499.7707212 MHz  
DATA PROCESSING  
FT size 32768  
Total time 0 min, 19 sec



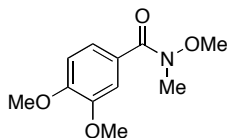
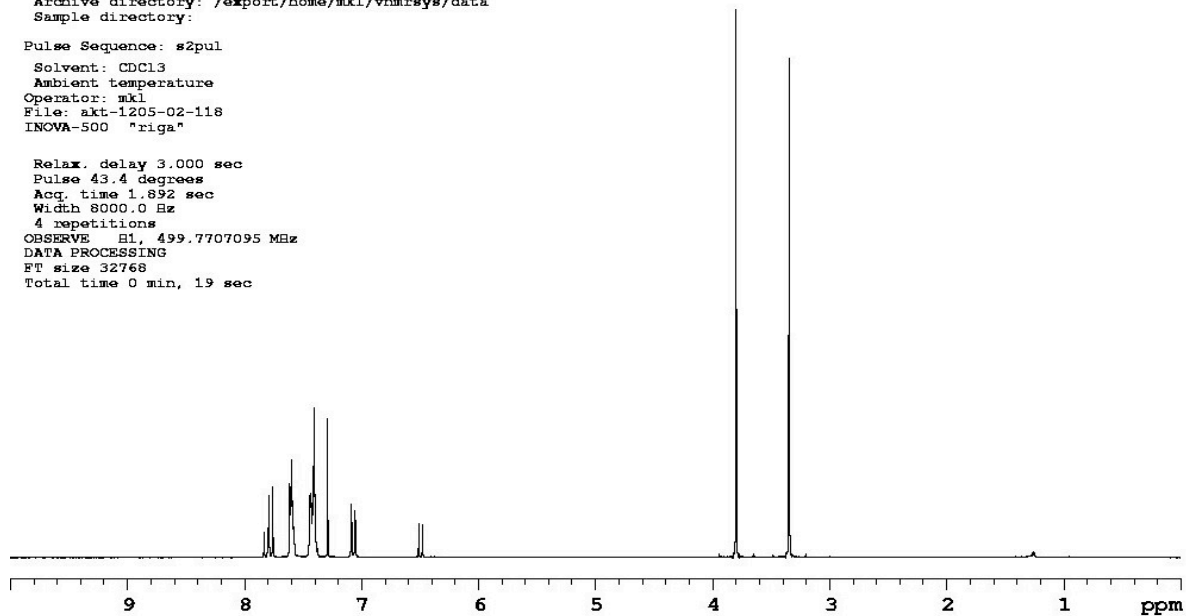


akt-1205-02-118

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

Pulse Sequence: s2pul  
 Solvent: CDCl3  
 Ambient temperature  
 Operator: mkl  
 File: akt-1205-02-118  
 INOVA-500 "riga"

Relax. delay 3.000 sec  
 Pulse 43.4 degrees  
 Acq. time 1.892 sec  
 Width 8000.0 Hz  
 4 repetitions  
 OBSERVE H1, 499.7707095 MHz  
 DATA PROCESSING  
 FT size 32768  
 Total time 0 min, 19 sec

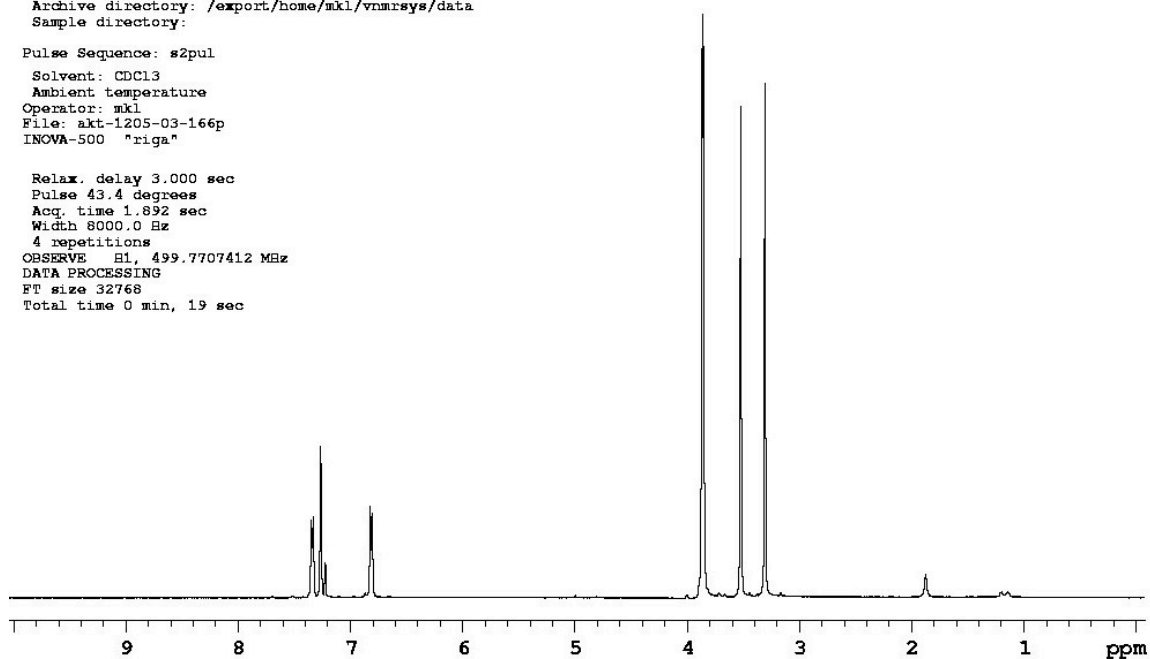


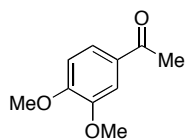
akt-1205-03-166p

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

Pulse Sequence: s2pul  
 Solvent: CDCl3  
 Ambient temperature  
 Operator: mkl  
 File: akt-1205-03-166p  
 INOVA-500 "riga"

Relax. delay 3.000 sec  
 Pulse 43.4 degrees  
 Acq. time 1.892 sec  
 Width 8000.0 Hz  
 4 repetitions  
 OBSERVE H1, 499.7707412 MHz  
 DATA PROCESSING  
 FT size 32768  
 Total time 0 min, 19 sec



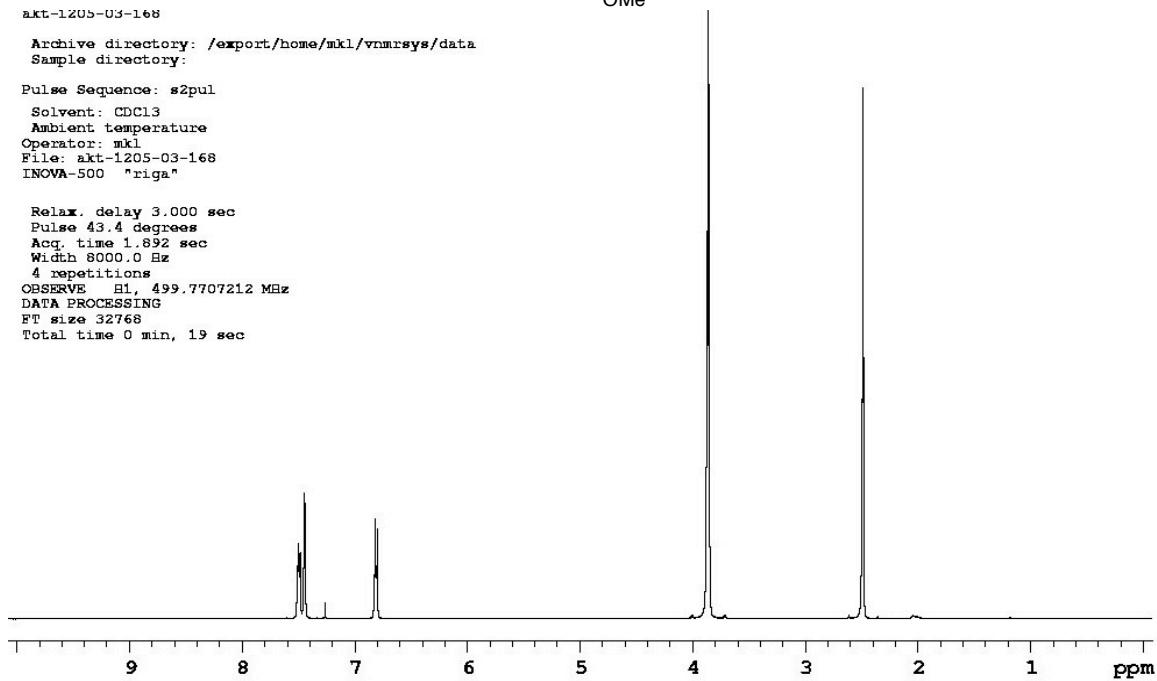


akt-1205-03-168

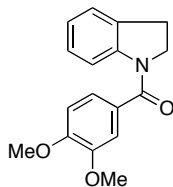
Archive directory: /export/home/mkl/vnmrsys/data  
Sample directory:

Pulse Sequence: s2pul  
Solvent: CDCl3  
Ambient temperature  
Operator: mkl  
File: akt-1205-03-168  
INOVA-500 "riga"

Relax. delay 3.000 sec  
Pulse 43.4 degrees  
Acq. time 1.892 sec  
Width 8000.0 Hz  
4 repetitions  
OBSERVE H1, 499.7707212 MHz  
DATA PROCESSING  
F1 size 32768  
Total time 0 min, 19 sec



The following pages contain NMR spectra of intermediates *en route* to pratosine and hippadine, as well as NMR spectra of anhydrolychorinine and hippadine



In CDCl<sub>3</sub> at room temperature

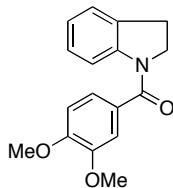
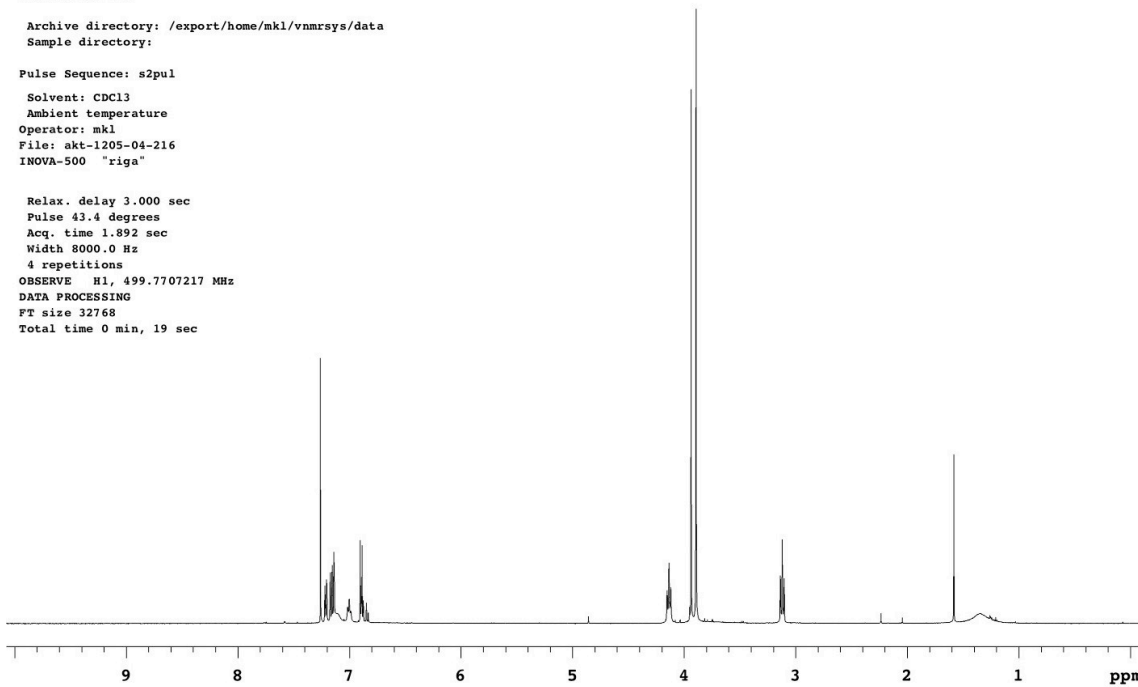
akt-1205-04-216

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

Pulse Sequence: s2pul

Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Operator: mkl  
File: akt-1205-04-216  
INOVA-500 "riga"

Relax. delay 3.000 sec  
Pulse 43.4 degrees  
Acq. time 1.892 sec  
Width 8000.0 Hz  
4 repetitions  
OBSERVE H1, 499.7707217 MHz  
DATA PROCESSING  
FT size 32768  
Total time 0 min, 19 sec



In C<sub>6</sub>D<sub>6</sub> at room temperature

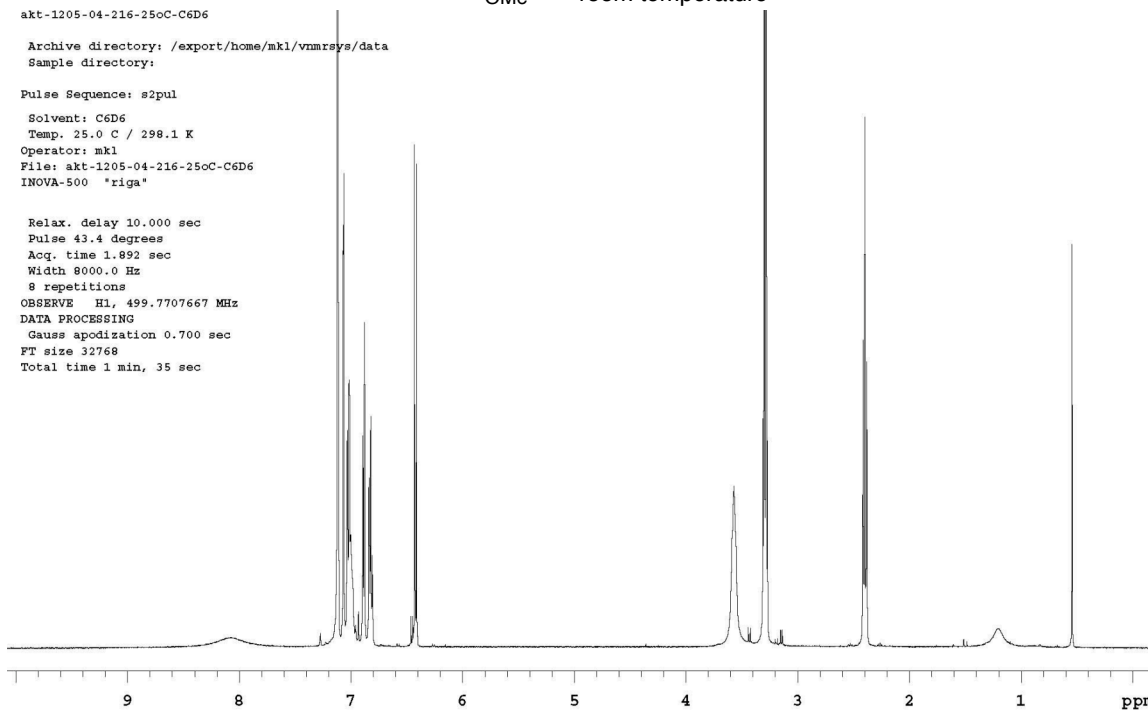
akt-1205-04-216-25oC-C6D6

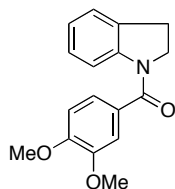
Archive directory: /export/home/mkl/vnmrsys/data  
Sample directory:

Pulse Sequence: s2pul

Solvent: C6D6  
Temp. 25.0 C / 298.1 K  
Operator: mkl  
File: akt-1205-04-216-25oC-C6D6  
INOVA-500 "riga"

Relax. delay 10.000 sec  
Pulse 43.4 degrees  
Acq. time 1.892 sec  
Width 8000.0 Hz  
8 repetitions  
OBSERVE H1, 499.7707667 MHz  
DATA PROCESSING  
Gauss apodization 0.700 sec  
FT size 32768  
Total time 1 min, 35 sec





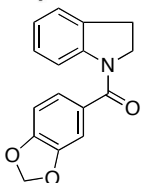
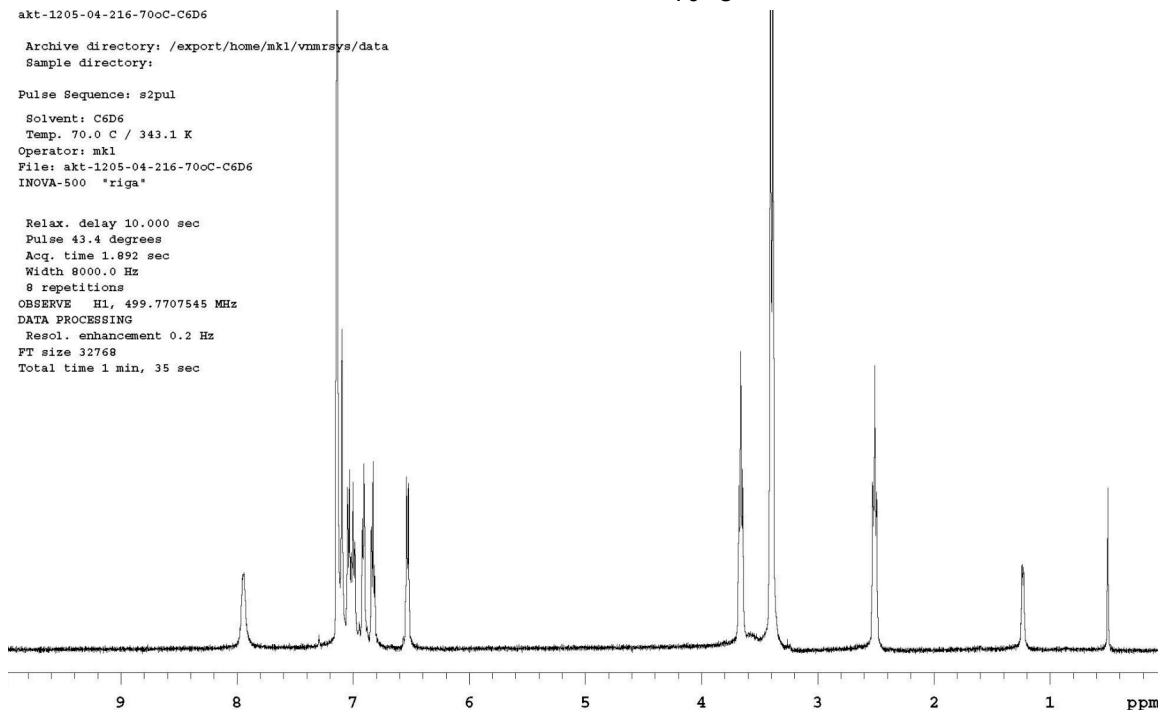
In  $C_6D_6$  at  
70 °C

akt-1205-04-216-70oC-C6D6

Archive directory: /export/home/mkl/vnmrSYS/data  
Sample directory:

Pulse Sequence: s2pul  
Solvent: C6D6  
Temp. 70.0 C / 343.1 K  
Operator: mkl  
File: akt-1205-04-216-70oC-C6D6  
INOVA-500 "riga"

Relax. delay 10.000 sec  
Pulse 43.4 degrees  
Acq. time 1.892 sec  
Width 8000.0 Hz  
8 repetitions  
OBSERVE H1, 499.7707545 MHz  
DATA PROCESSING  
Resol. enhancement 0.2 Hz  
FT size 32768  
Total time 1 min, 35 sec



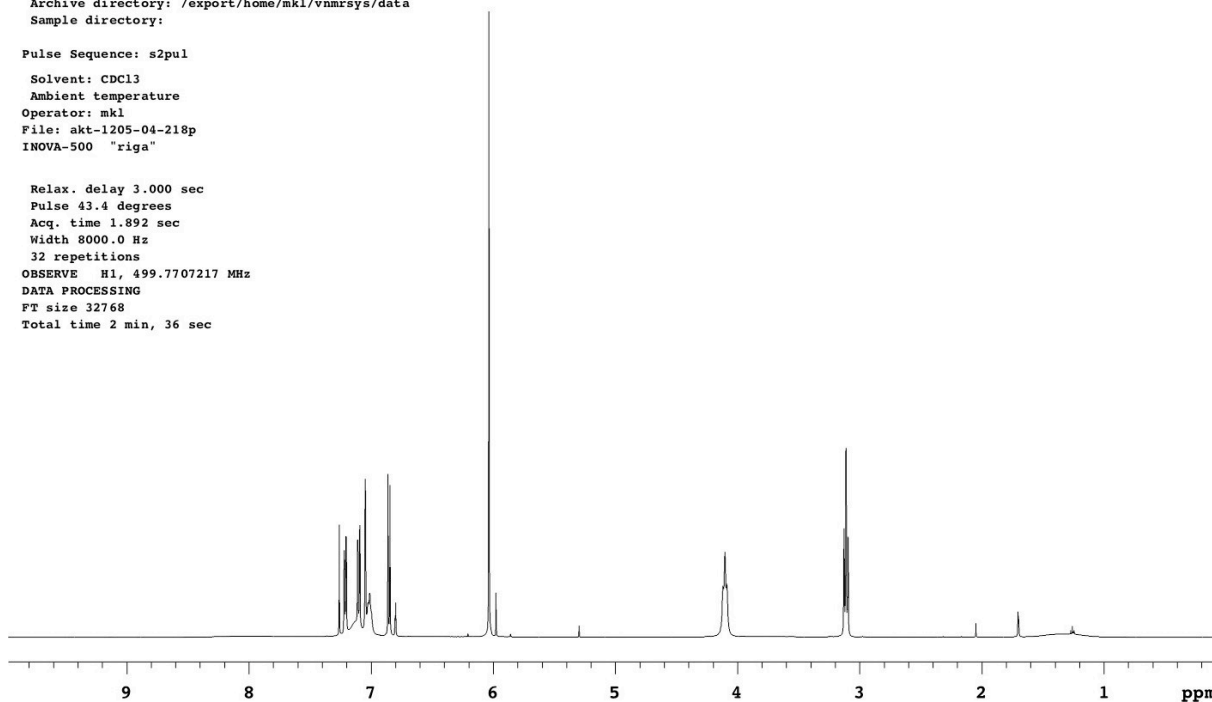
In  $CDCl_3$  at  
room temperature

akt-1205-04-218p

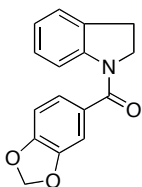
Archive directory: /export/home/mkl/vnmrSYS/data  
Sample directory:

Pulse Sequence: s2pul  
Solvent: CDCl3  
Ambient temperature  
Operator: mkl  
File: akt-1205-04-218p  
INOVA-500 "riga"

Relax. delay 3.000 sec  
Pulse 43.4 degrees  
Acq. time 1.892 sec  
Width 8000.0 Hz  
32 repetitions  
OBSERVE H1, 499.7707217 MHz  
DATA PROCESSING  
FT size 32768  
Total time 2 min, 36 sec







In C<sub>6</sub>D<sub>6</sub> at  
room temperature

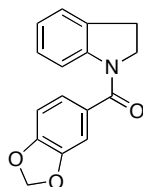
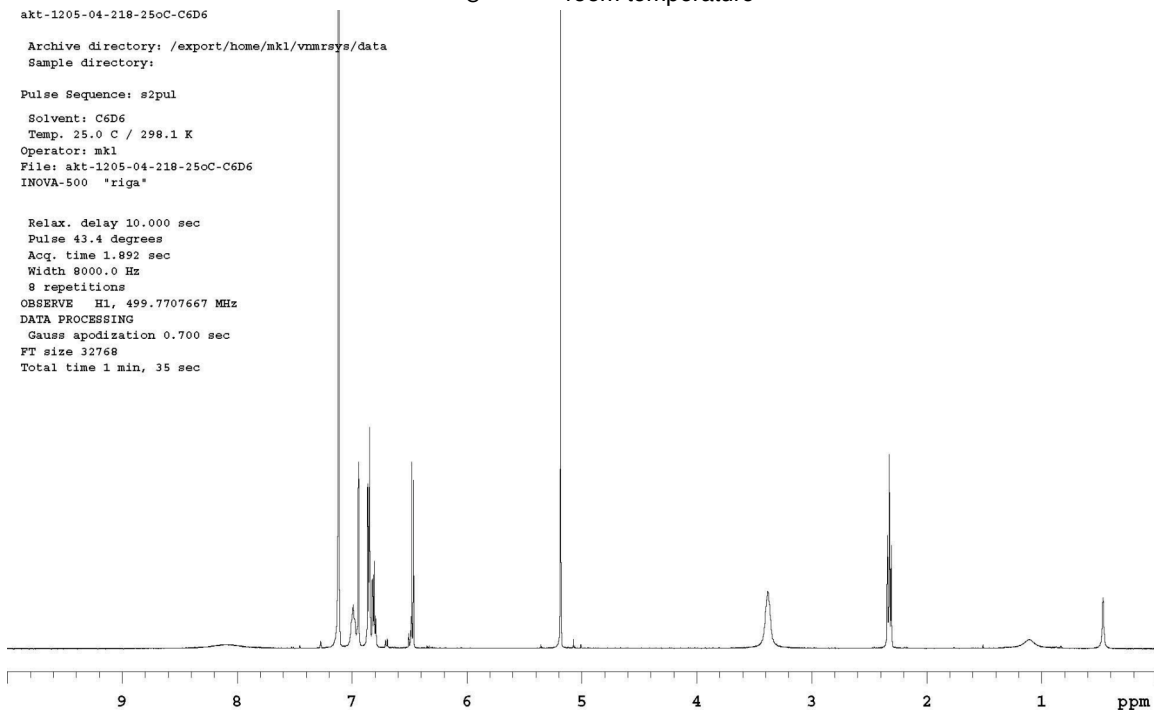
akt-1205-04-218-250C-C6D6

Archive directory: /export/home/mkl/vnmrsvs/data  
Sample directory:

Pulse Sequence: s2pul

Solvent: C6D6  
Temp. 25.0 C / 298.1 K  
Operator: mkl  
File: akt-1205-04-218-250C-C6D6  
INOVA-500 "riga"

Relax. delay 10.000 sec  
Pulse 43.4 degrees  
Acq. time 1.892 sec  
Width 8000.0 Hz  
8 repetitions  
OBSERVE H1, 499.7707667 MHz  
DATA PROCESSING  
Gauss apodization 0.700 sec  
FT size 32768  
Total time 1 min, 35 sec



In C<sub>6</sub>D<sub>6</sub> at  
70 °C

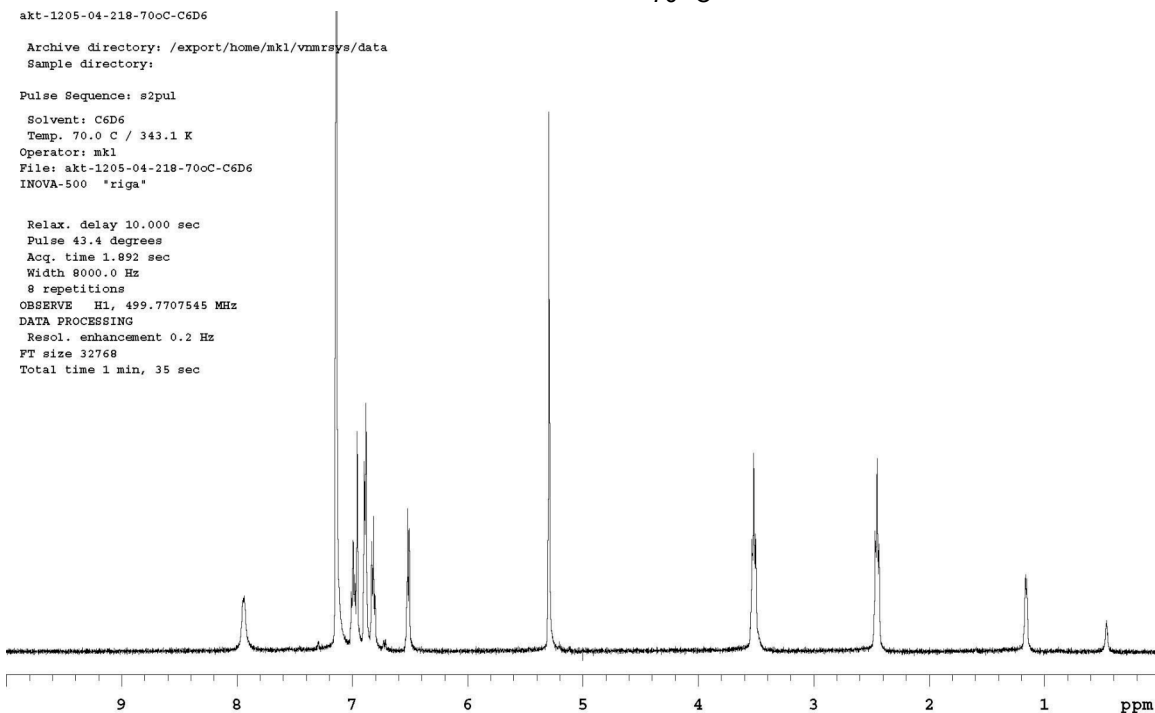
akt-1205-04-218-700C-C6D6

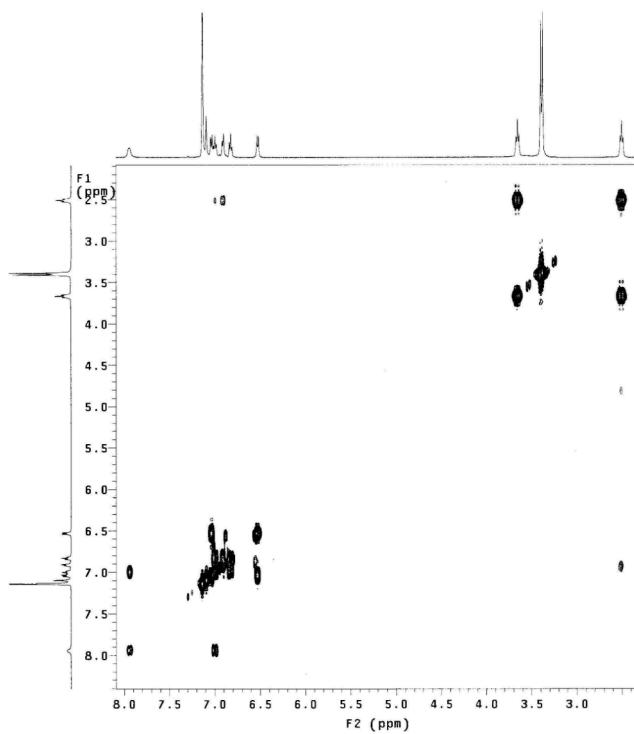
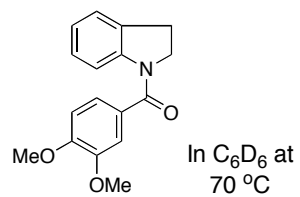
Archive directory: /export/home/mkl/vnmrsvs/data  
Sample directory:

Pulse Sequence: s2pul

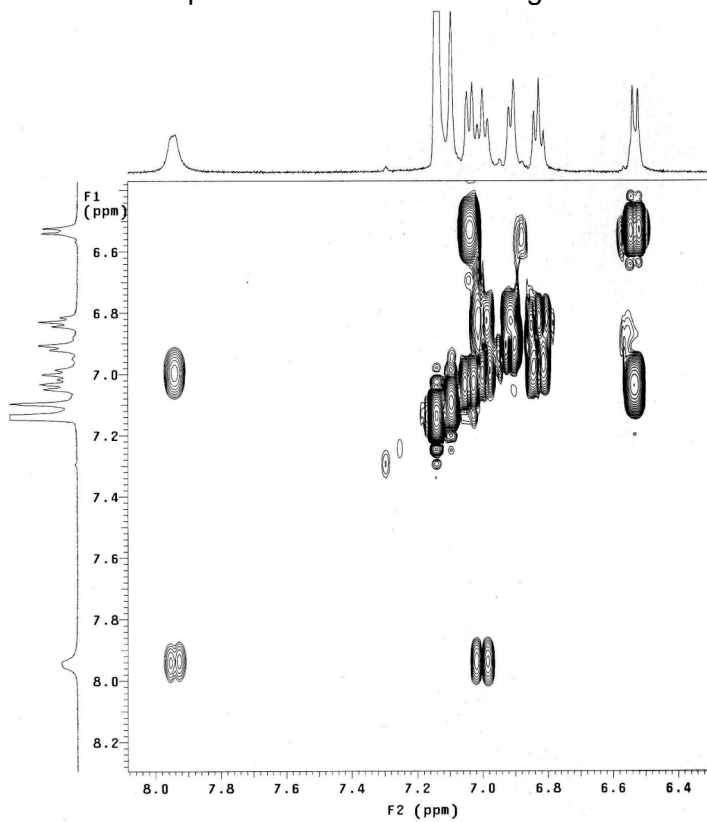
Solvent: C6D6  
Temp. 70.0 C / 343.1 K  
Operator: mkl  
File: akt-1205-04-218-700C-C6D6  
INOVA-500 "riga"

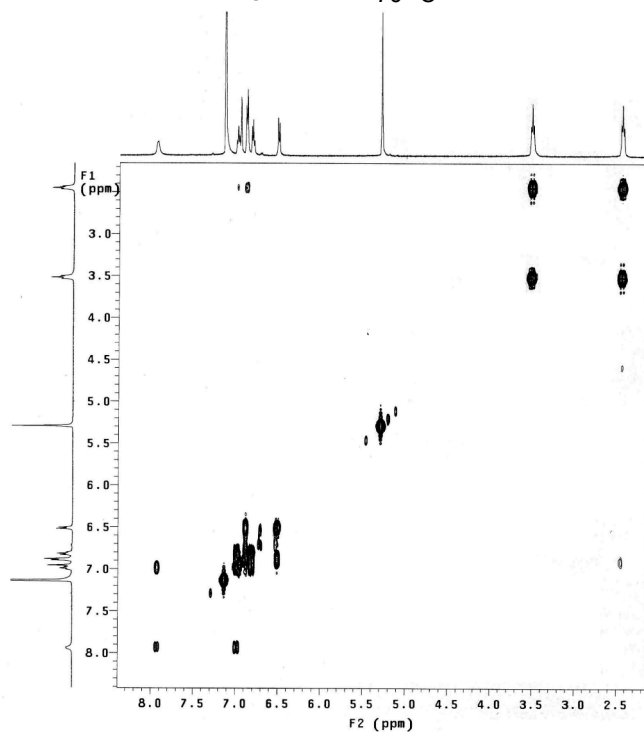
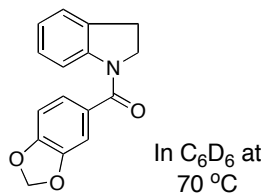
Relax. delay 10.000 sec  
Pulse 43.4 degrees  
Acq. time 1.892 sec  
Width 8000.0 Hz  
8 repetitions  
OBSERVE H1, 499.7707545 MHz  
DATA PROCESSING  
Resol. enhancement 0.2 Hz  
FT size 32768  
Total time 1 min, 35 sec



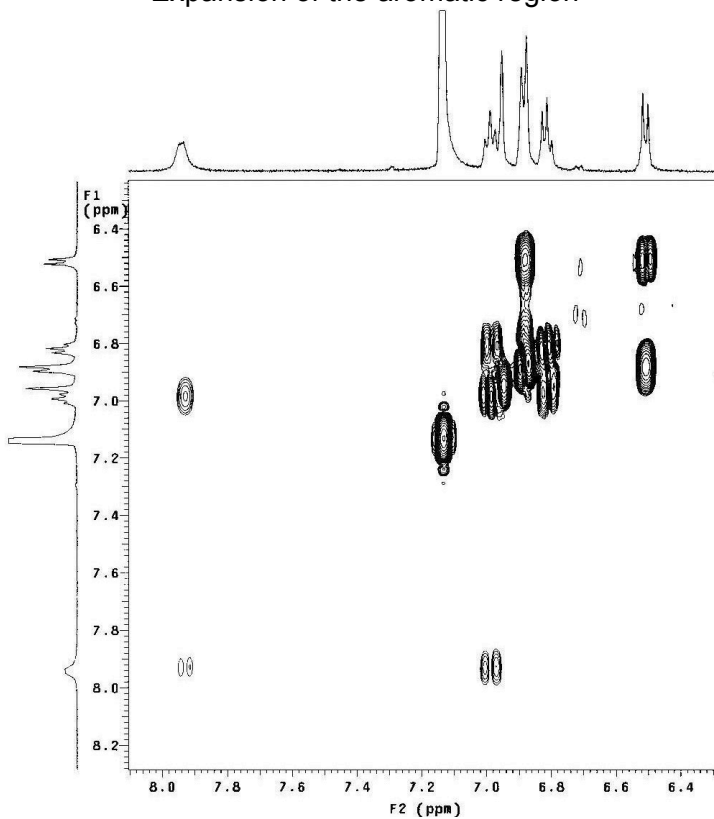


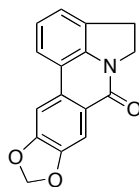
Expansion of the aromatic region





Expansion of the aromatic region





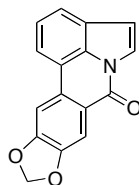
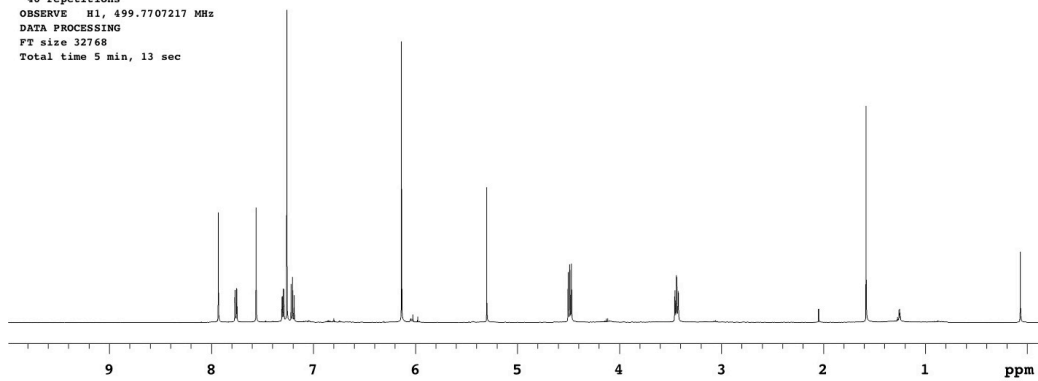
akt-1205-04-221p

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

Pulse Sequence: s2pul

Solvent: CDCl3  
Ambient temperature  
Operator: mkl  
File: akt-1205-04-221p  
INOVA-500 "riga"

Relax. delay 3.000 sec  
Pulse 43.4 degrees  
Acq. time 1.892 sec  
Width 8000.0 Hz  
40 repetitions  
OBSERVE H1, 499.7707217 MHz  
DATA PROCESSING  
FT size 32768  
Total time 5 min, 13 sec



hari-1205-04-69

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

Pulse Sequence: s2pul

Solvent: CDCl3  
Ambient temperature  
Operator: mkl  
File: hari-1205-04-69  
INOVA-500 "riga"

Relax. delay 3.000 sec  
Pulse 43.4 degrees  
Acq. time 1.892 sec  
Width 8000.0 Hz  
4 repetitions  
OBSERVE H1, 499.7707217 MHz  
DATA PROCESSING  
FT size 32768  
Total time 0 min, 19 sec

