Iron-Catalyzed Cross-Coupling of Imidoyl Chlorides with Grignard Reagents

Lars K. Ottesen, Fredrik Ek, and Roger Olsson*

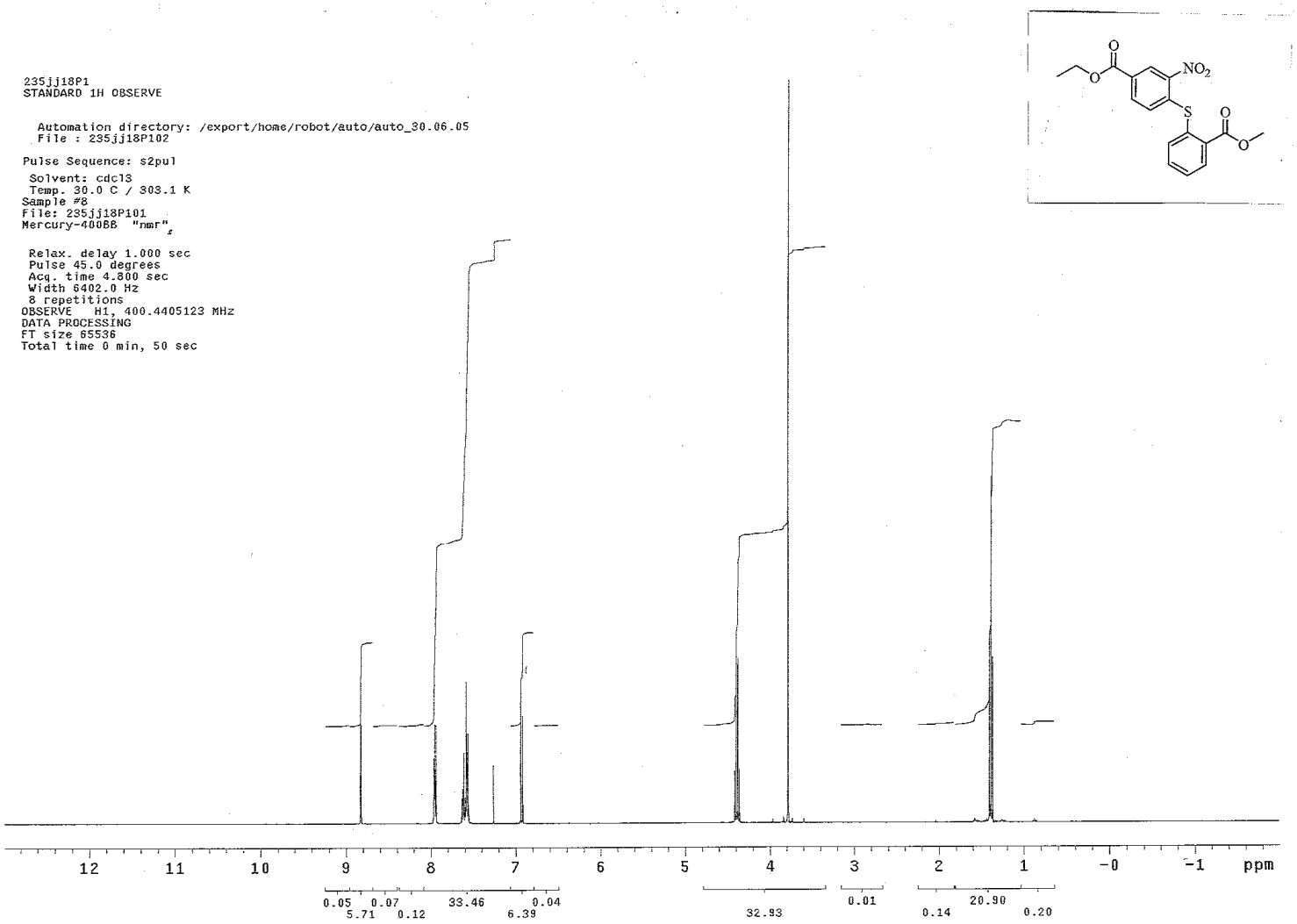
SUPPORTING INFORMATION

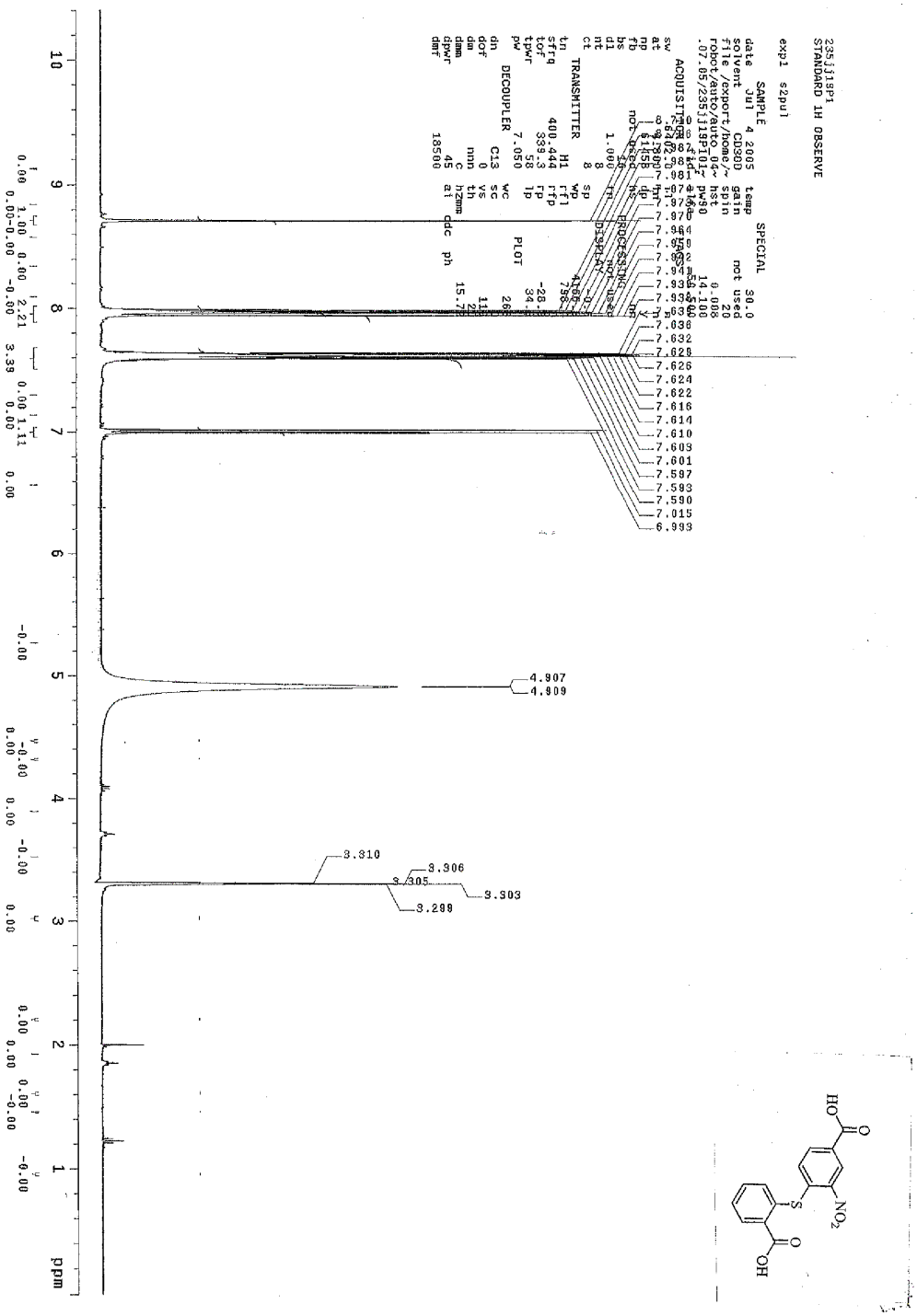
Table of Contents

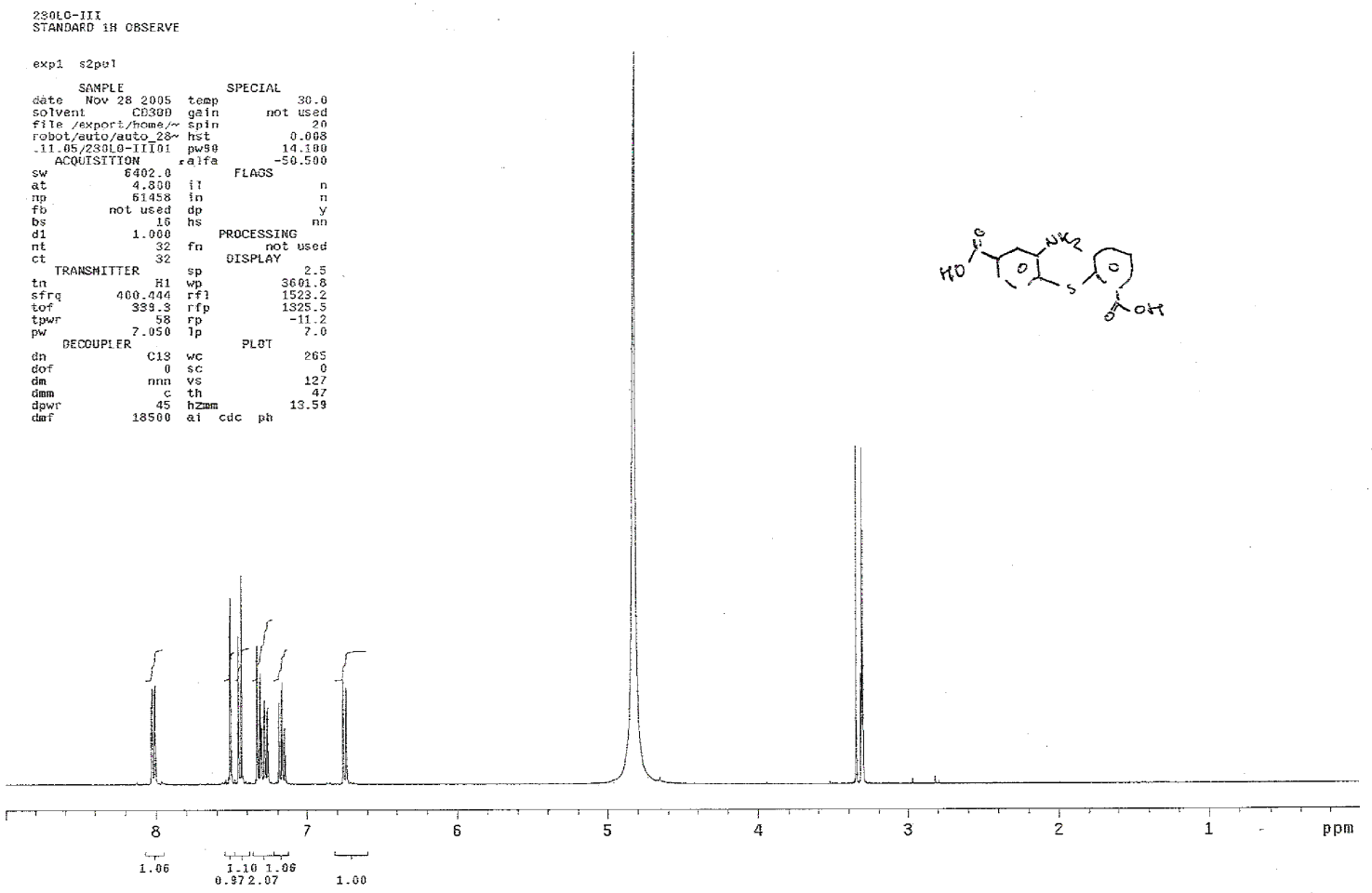
Supporting information file one

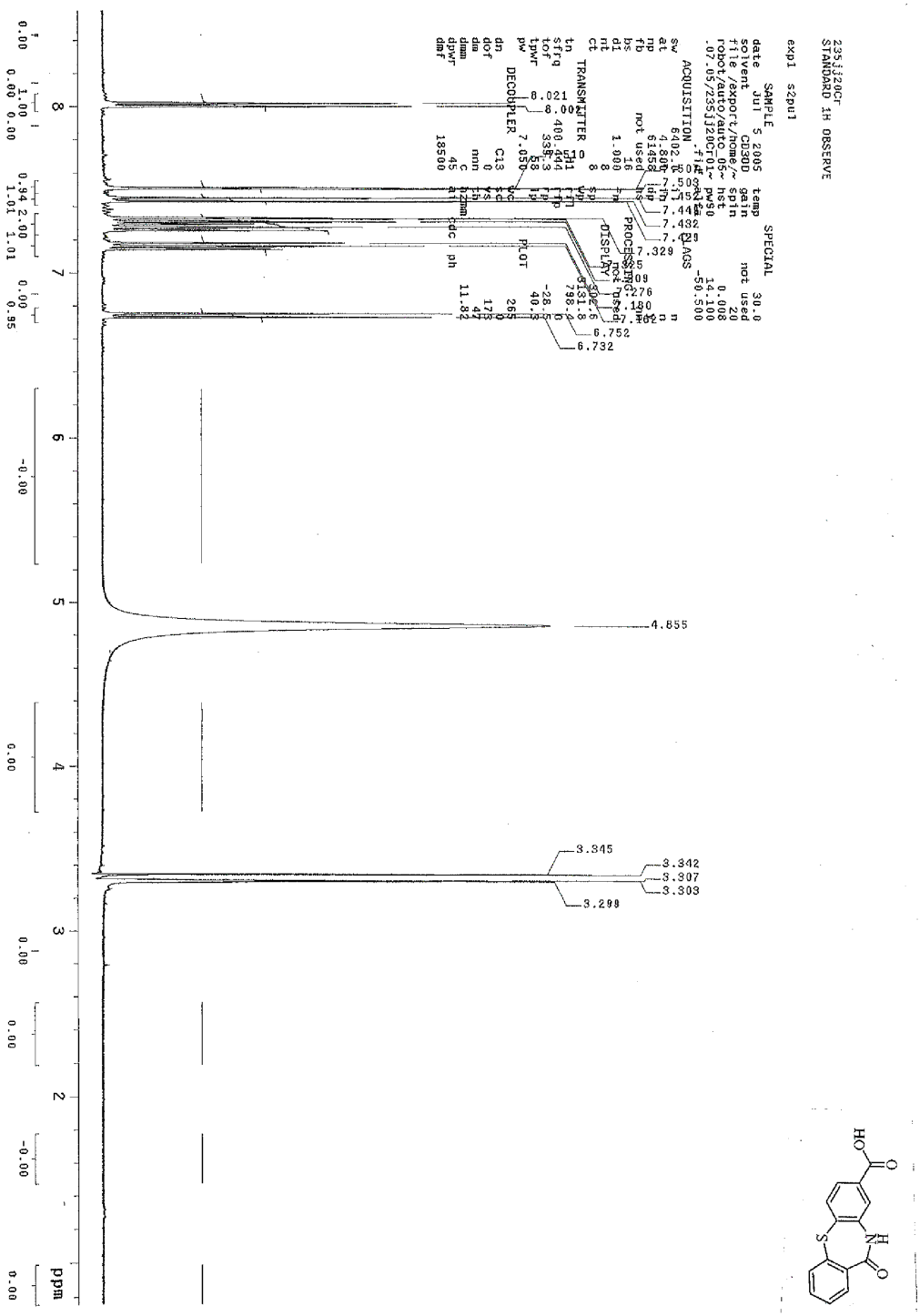
Supporting information file two

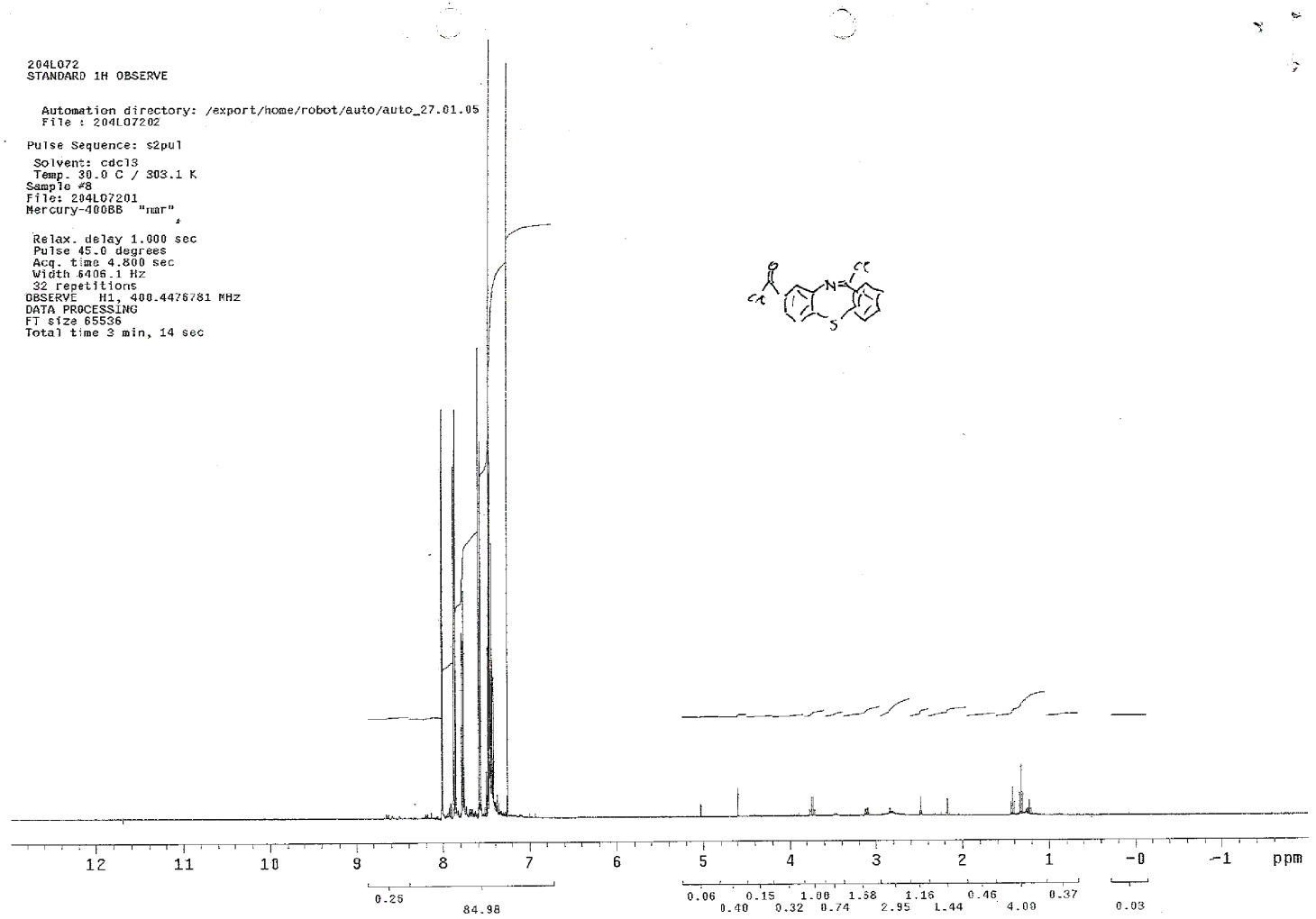
Supporting information file three (this file)


235jj18P1
STANDARD 1H OBSERVE
Automation directory: /export/home/robot/auto/auto_30.06.05
File : 235jj18P102
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 30.0 C / 303.1 K
Sample #8
File: 235jj18P101
Mercury-400BB "nmr"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 4.800 sec
Width 6402.0 Hz
8 repetitions
OBSERVE H1, 400.4405123 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 50 sec
O
O
NO2
S
O
O
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
0.05
0.07
33.46
0.04
5.71
0.12
6.39
32.93
0.01
20.90
0.14
0.20


235jj19P1
STANDARD 1H OBSERVE
4.907
4.909
3.310
3.306
3.305
3.303
3.298
ppm


280LG-III
STANDARD 1H OBSERVE
exp1 s2pul
SAMPLE
date Nov 28 2005
solvent CD3OD
file /export/home/~
robot/auto/auto_28~
.11.05/280LG-III01
ACQUISITION
sw 6402.0
at 4.800
np 61458
fb not used
bs 16
d1 1.000
nt 32
ct 32
TRANSMITTER
tn H1
sfrq 400.444
tof 339.3
tpwr 58
pw 7.050
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 45
daf 18500
SPECIAL
temp 30.0
gain not used
spin 20
hst 0.008
pw90 14.180
alfa -50.500
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp 2.5
wp 3601.8
rf1 1523.2
rfp 1325.5
rp -11.2
lp 7.0
PLOT
wc 265
sc 0
vs 127
th 47
hzmm 13.59
ai cdc ph
8
7
6
5
4
3
2
1
ppm
1.06
1.10
1.06
0.97
2.07
1.00

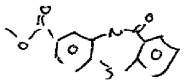

4.855
3.345
3.342
3.307
3.303
3.299
6.752
6.732
ppm

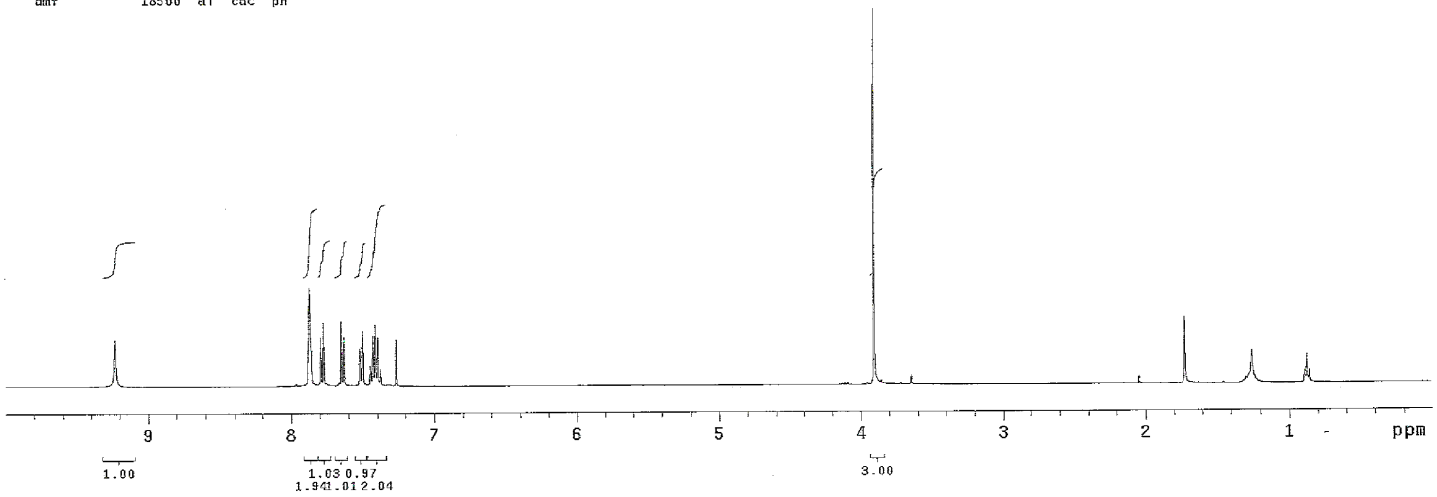

204L072
STANDARD 1H OBSERVE
Automation directory: /export/home/robot/auto/auto_27.01.05
File : 204L07202
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 30.0 C / 303.1 K
Sample #8
File: 204L07201
Mercury-400BB "nmr"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 4.800 sec
Width 6406.1 Hz
32 repetitions
OBSERVE H1, 400.4476781 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 14 sec
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
0.25
84.98
0.06
0.40
0.15
0.32
1.00
0.74
1.58
2.95
1.16
1.44
0.46
4.00
0.37
0.03

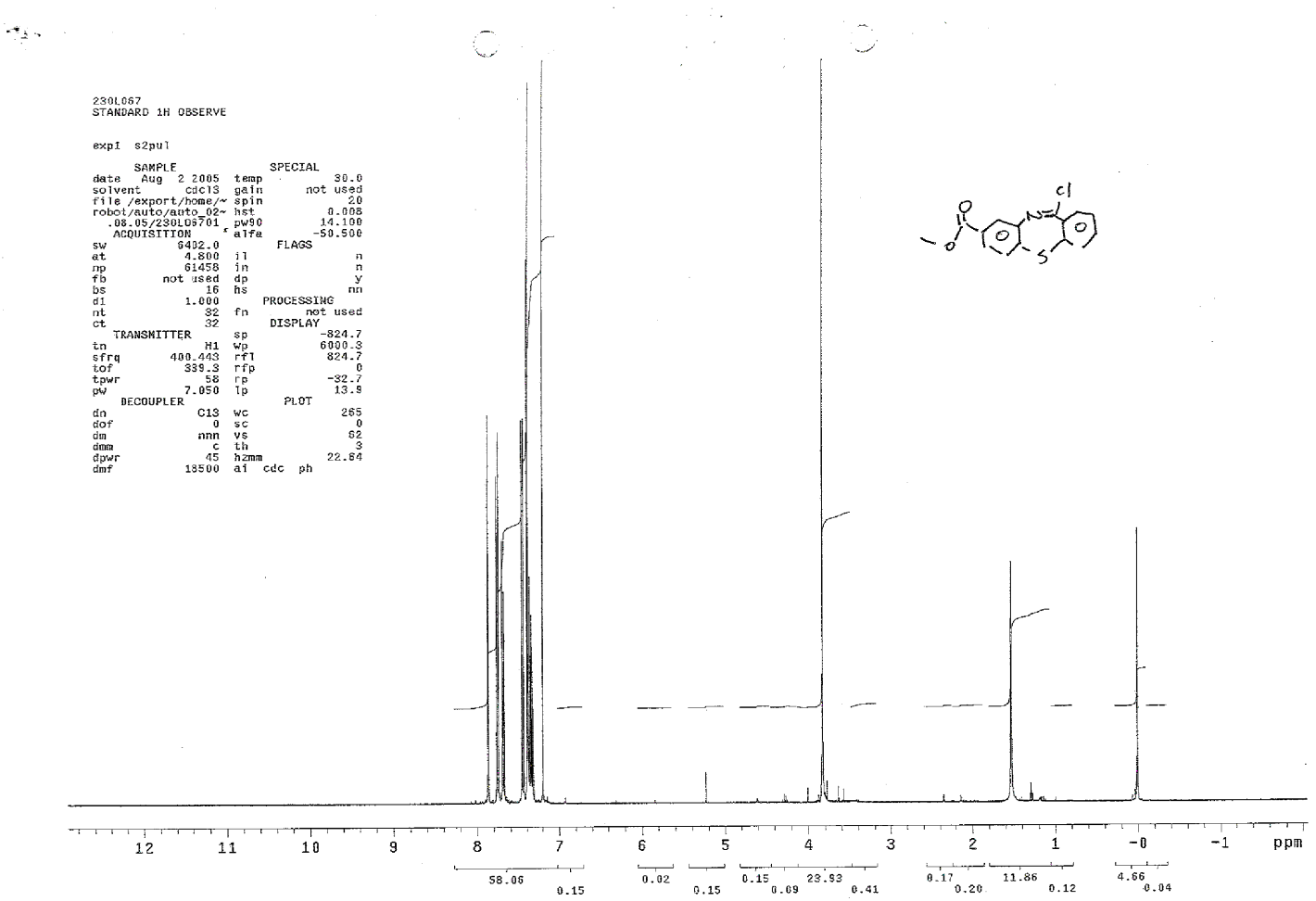
```
230LO50
STANDARD 1H OBSERVE

exp1  s2pul

      SAMPLE                SPECIAL
date   Aug  2 2005  temp          30.0
solvent      cdcl3  gain       not used
file /export/home/~ spin            20
robot/auto/auto_02~ hst          0.008
  .08.05/230LO6001  pw90         14.100
   ACQUISITION      alfa        -50.500
sw           6402.0          FLAGS
at            4.800  il              n
np          61458  in              n
fb        not used  dp              y
bs              16  hs             nn
dl           1.000      PROCESSING
nt              32  fn       not used
ct              32       DISPLAY
   TRANSMITTER          sp            2.1
tn              H1  wp         3999.4
sfrq       400.443  rfl         3703.6
tof          339.3  rfp         2907.2
tpwr            58  rp           -36.9
pw           7.050  lp            21.4
    DECOUPLER             PLOT
dn              C13  wc             265
dof               0  sc               0
dm              nnn  vs              41
dmm               c  th              32
dpwr             45  hzmm         15.09
dmf           18500  ai  cdc  ph
```



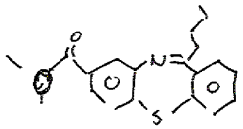


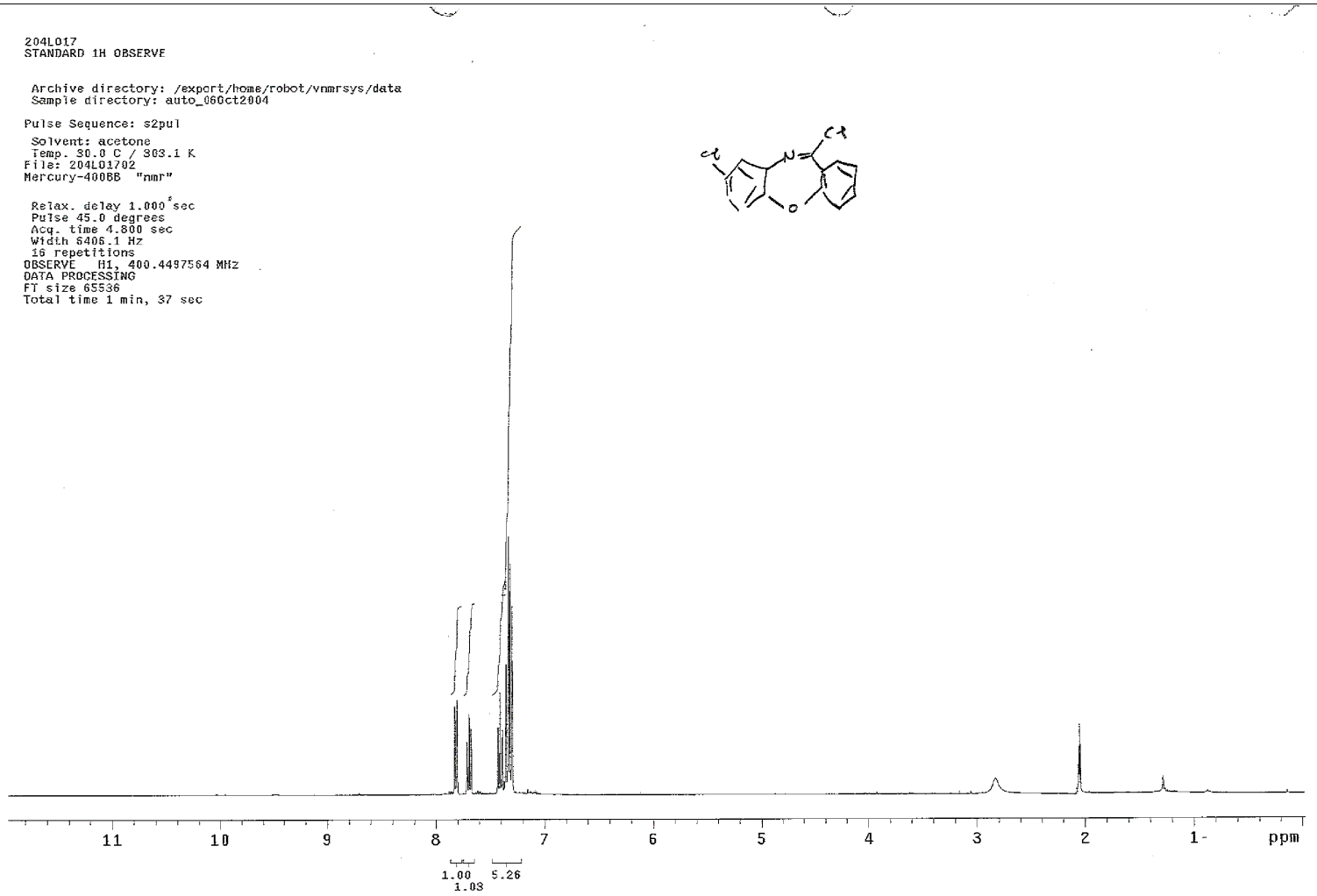

230L067
STANDARD 1H OBSERVE
expI s2pul
SAMPLE
date Aug 2 2005
solvent cdcl3
file /export/home/~
robot/auto/auto_02~
.08.05/230L06701
ACQUISITION
sw 6402.0
at 4.800
np 61458
fb not used
bs 16
d1 1.000
nt 32
ct 32
TRANSMITTER
tn H1
sfrq 400.443
tof 339.3
tpwr 58
pw 7.050
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 45
dmf 18500
SPECIAL
temp 30.0
gain not used
spin 20
hst 0.008
pw90 14.100
alfa -50.500
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -824.7
wp 6000.8
rfl 824.7
rfp 0
rp -32.7
lp 13.9
PLOT
wc 265
sc 0
vs 62
th 3
hzmm 22.84
ai cdc ph
Cl
S
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
58.06
0.15
0.02
0.15
0.15
0.09
23.93
0.41
0.17
0.20
11.86
0.12
4.66
0.04

230L052
STANDARD 1H OBSERVE

exp1 s2pul

SAMPLE		SPECIAL	
date	Jul 21 2005	temp	30.0
solvent	cdcl3	gain	not used
file	/export/home/~	spin	20
robot	/auto/auto_21~	hst	0.008
	.07_05/230L05201	pw90	14.100
ACQUISITION		alfa	-50.500
sw	6402.0	FLAGS	
at	4.800	il	n
np	61458	in	n
fb	not used	dp	y
bs	16	hs	nn
d1	1.000	PROCESSING	
nt	32	fn	not used
ct	32	DISPLAY	
TRANSMITTER		sp	-12.4
tn	H1	wp	3609.3
sfrq	400.443	rfl	788.4
tof	339.9	rfp	0
tpwr	58	rp	-27.5
pw	7.050	lp	5.0
DECOUPLER		PLOT	
dn	C13	wc	265
dof	0	sc	0
dm	nnn	vs	34
dmm	c	th	47
dpwr	45	hzmm	13.62
dmf	18500	ai cdc ph	



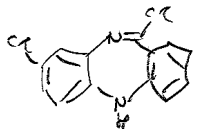

204L017
STANDARD 1H OBSERVE
Archive directory: /export/home/robot/vnmrsys/data
Sample directory: auto_060ct2004
Pulse Sequence: s2pul
Solvent: acetone
Temp. 30.0 C / 303.1 K
File: 204L01702
Mercury-400BB "nmr"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 4.800 sec
Width 5406.1 Hz
16 repetitions
OBSERVE H1, 400.4497564 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 37 sec
Cl
N
Cl
O
11
10
9
8
7
6
5
4
3
2
1
ppm
1.00
1.03
5.26

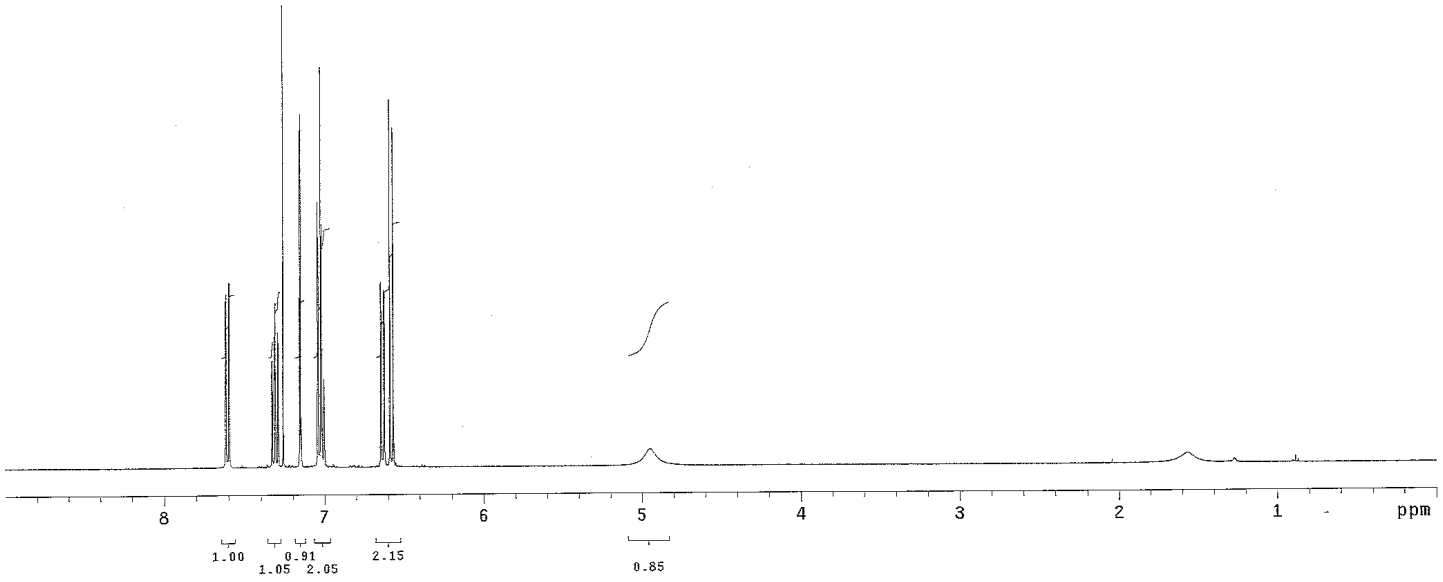
189JOImCl
STANDARD 1H OBSERVE

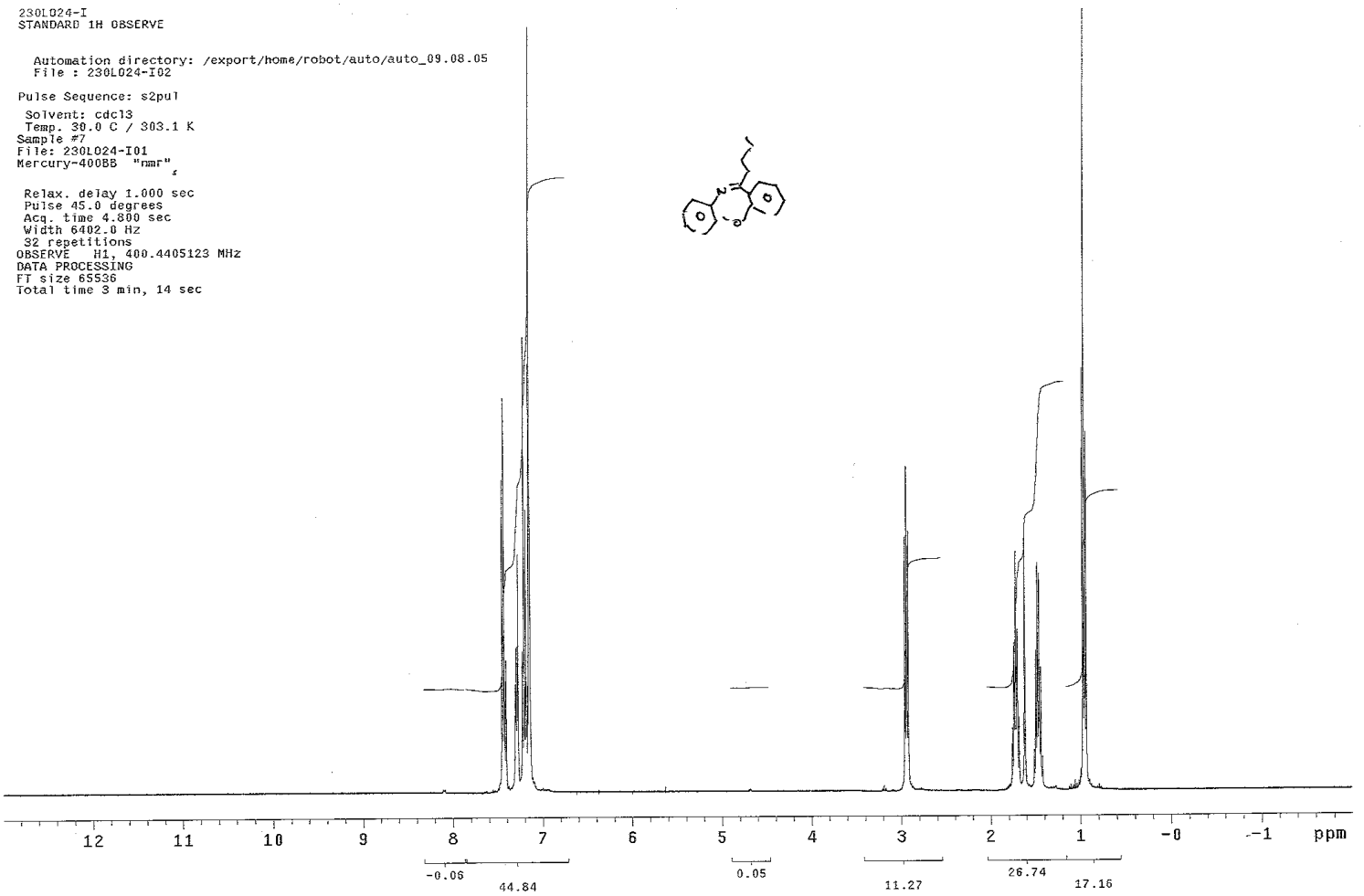
Archive directory: /export/home/robot/vnmrsys/data
Sample directory: auto_26Oct2004

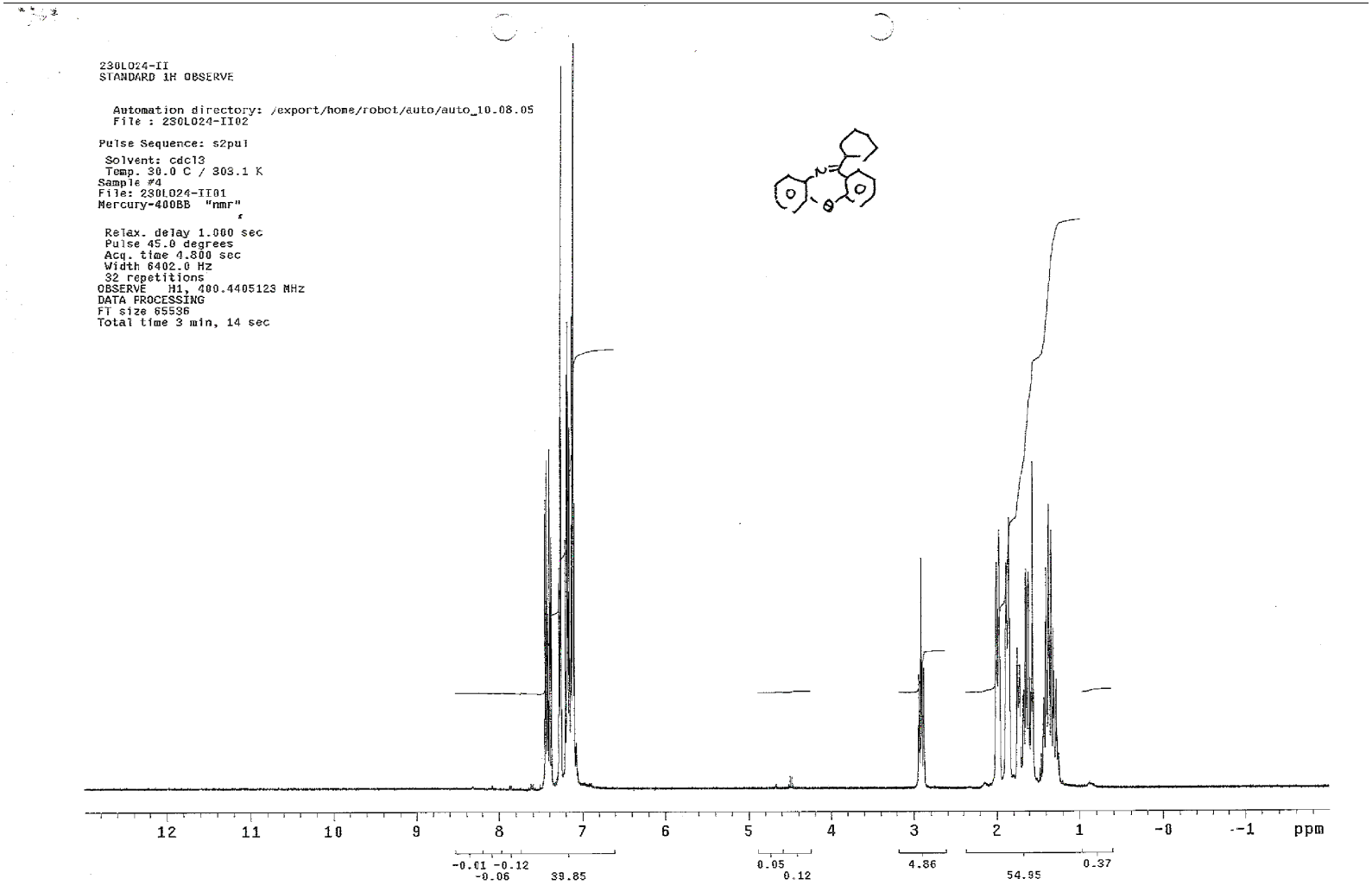
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 30.0 C / 303.1 K
File: 189JOImCl01
Mercury-400BB "nmr"

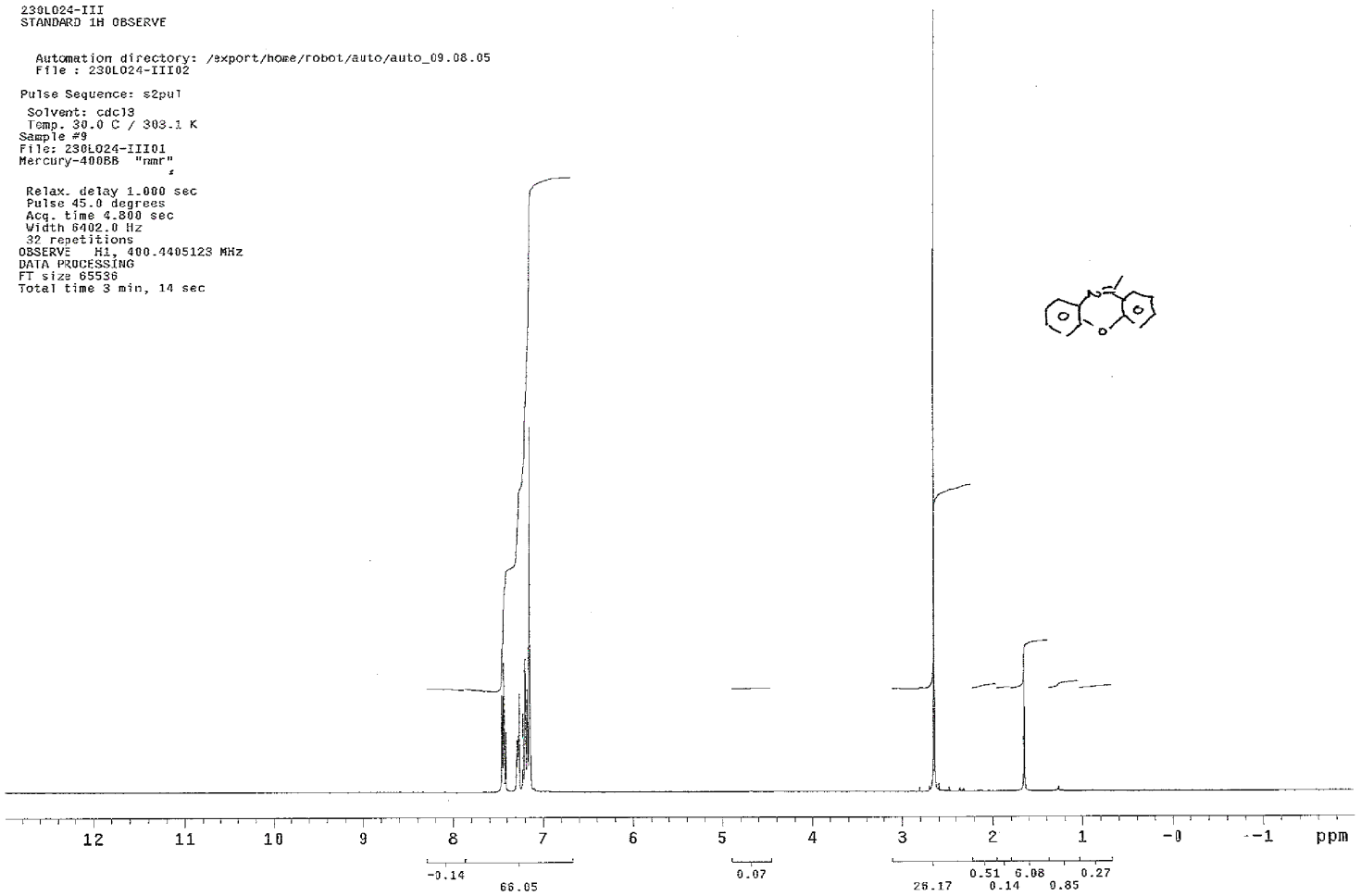
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 4.800 sec
Width 6406.1 Hz
16 repetitions
OBSERVE H1, 400.4476780 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 37 sec

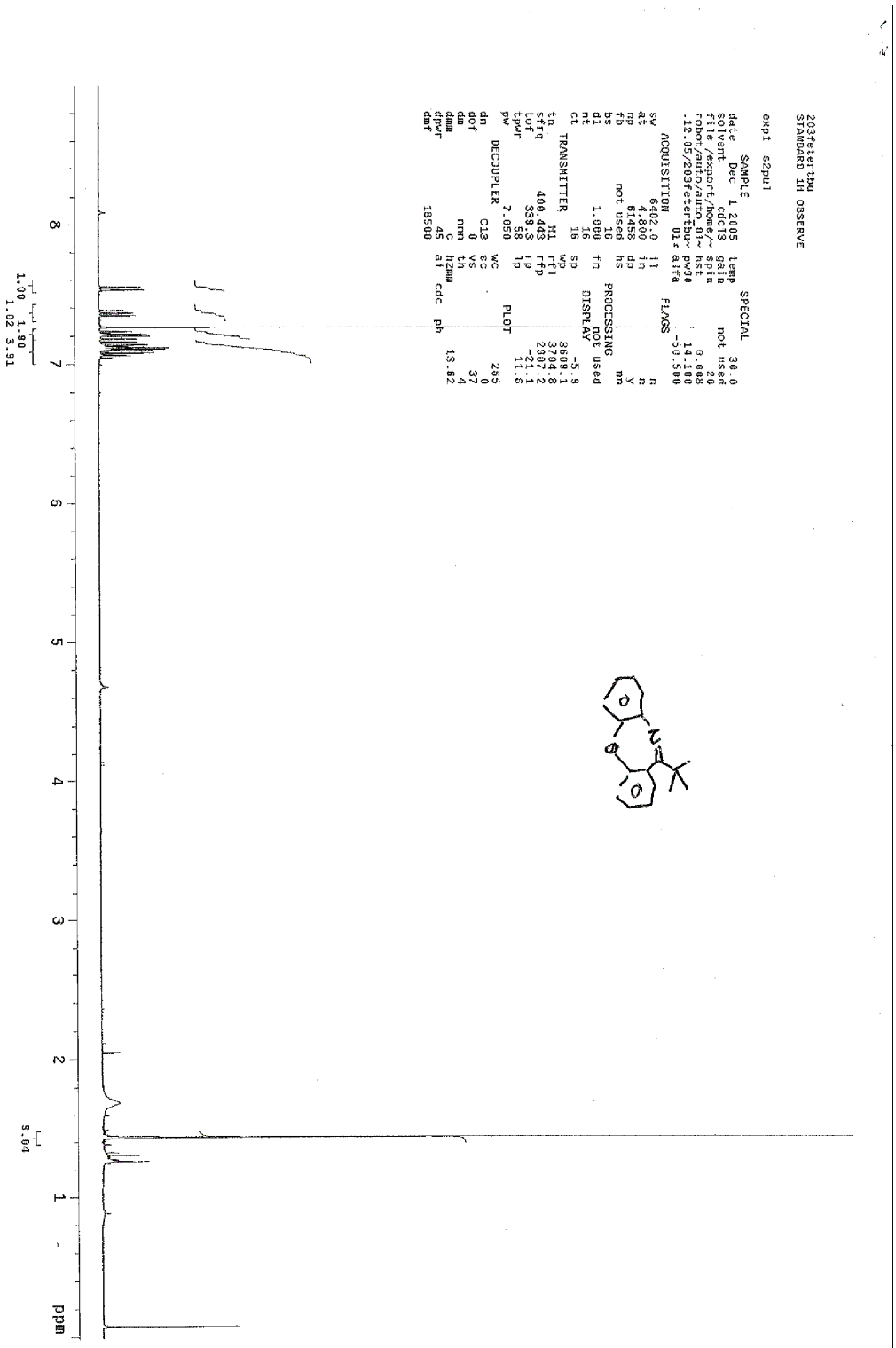


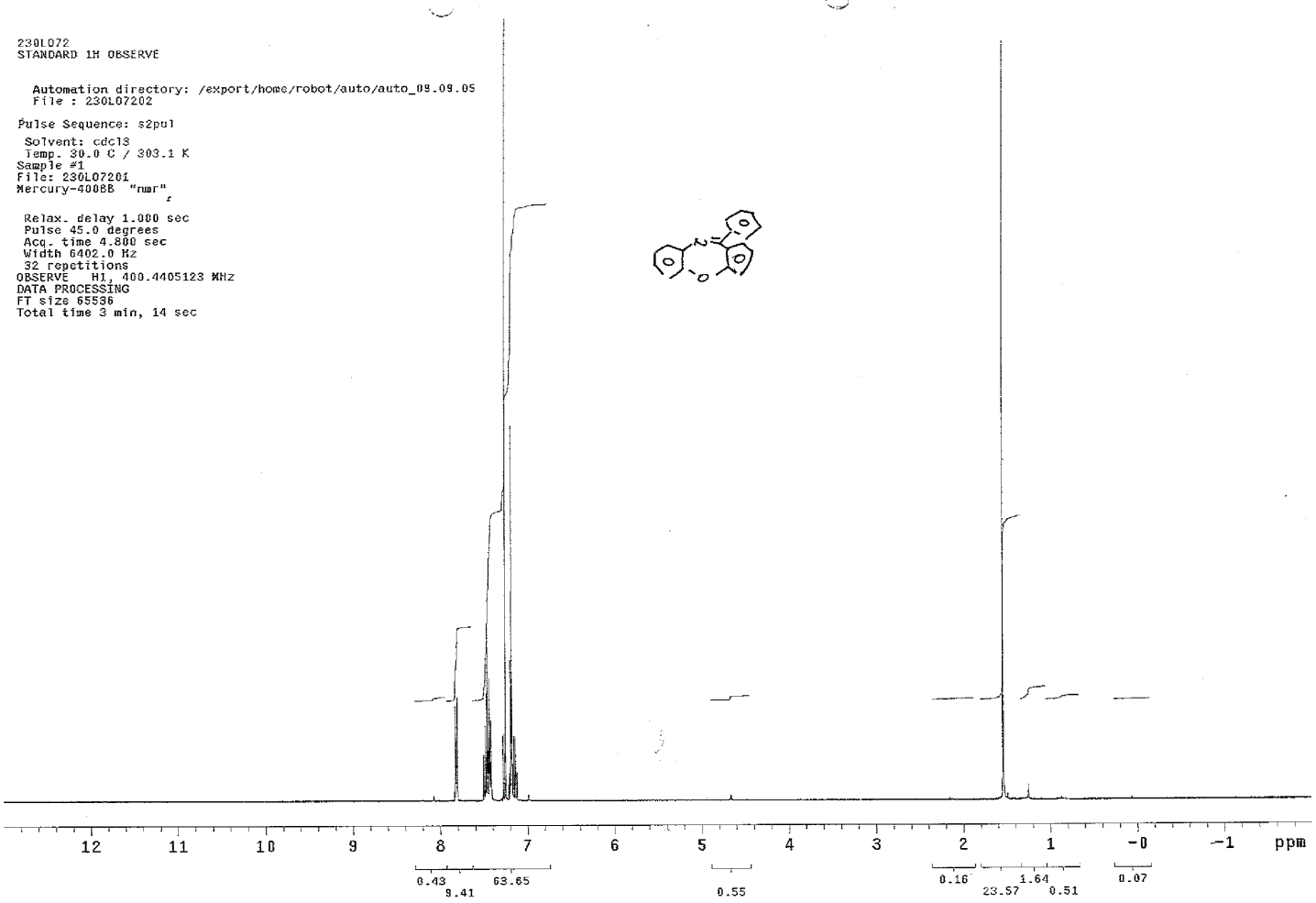


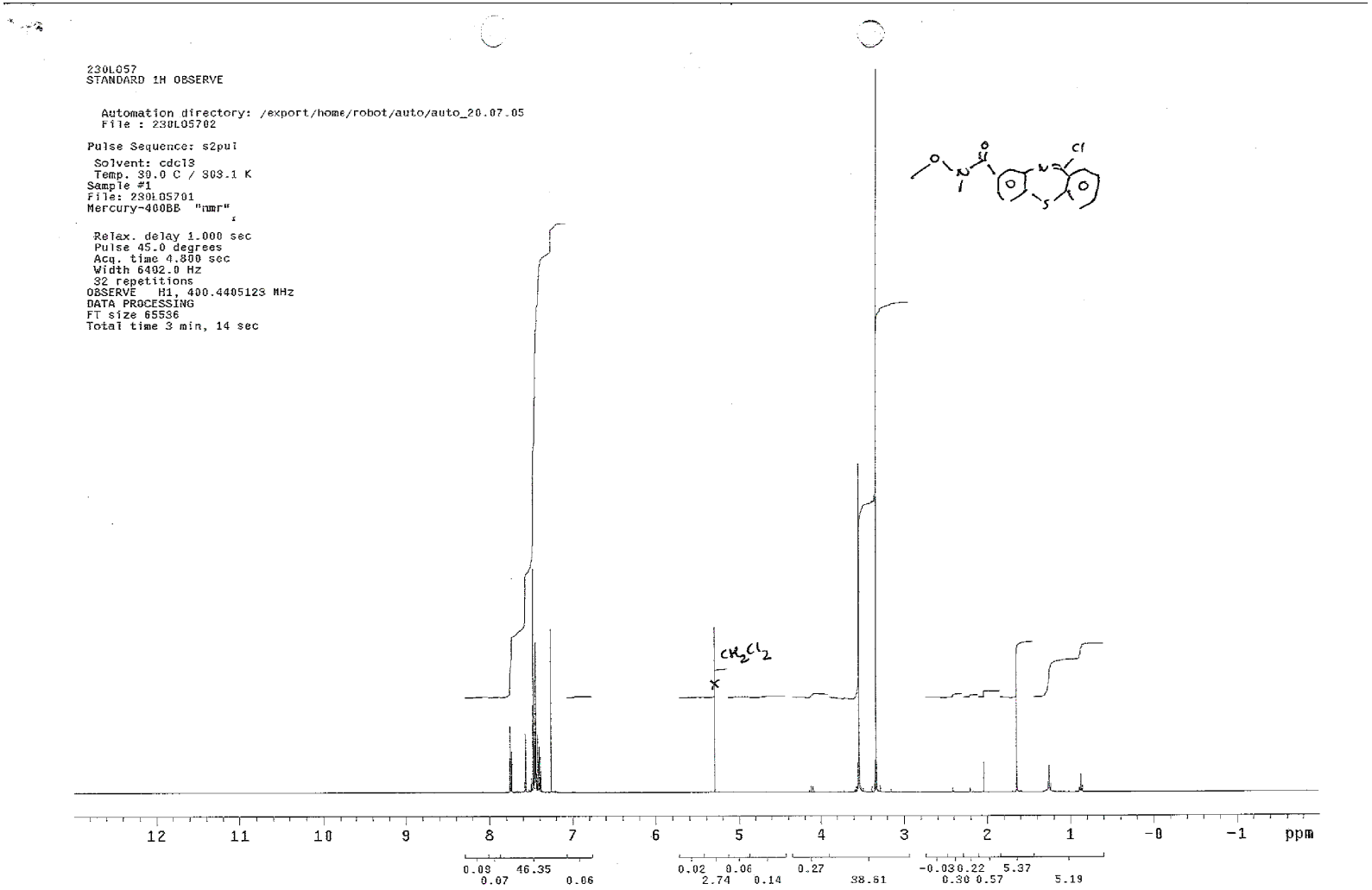

230L024-I
STANDARD 1H OBSERVE
Automation directory: /export/home/robot/auto/auto_09.08.05
File : 230L024-I02
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 30.0 C / 303.1 K
Sample #7
File: 230L024-I01
Mercury-400BB "nmr"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 4.800 sec
Width 6402.0 Hz
32 repetitions
OBSERVE H1, 400.4405123 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 14 sec
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
-0.06
44.84
0.05
11.27
26.74
17.16

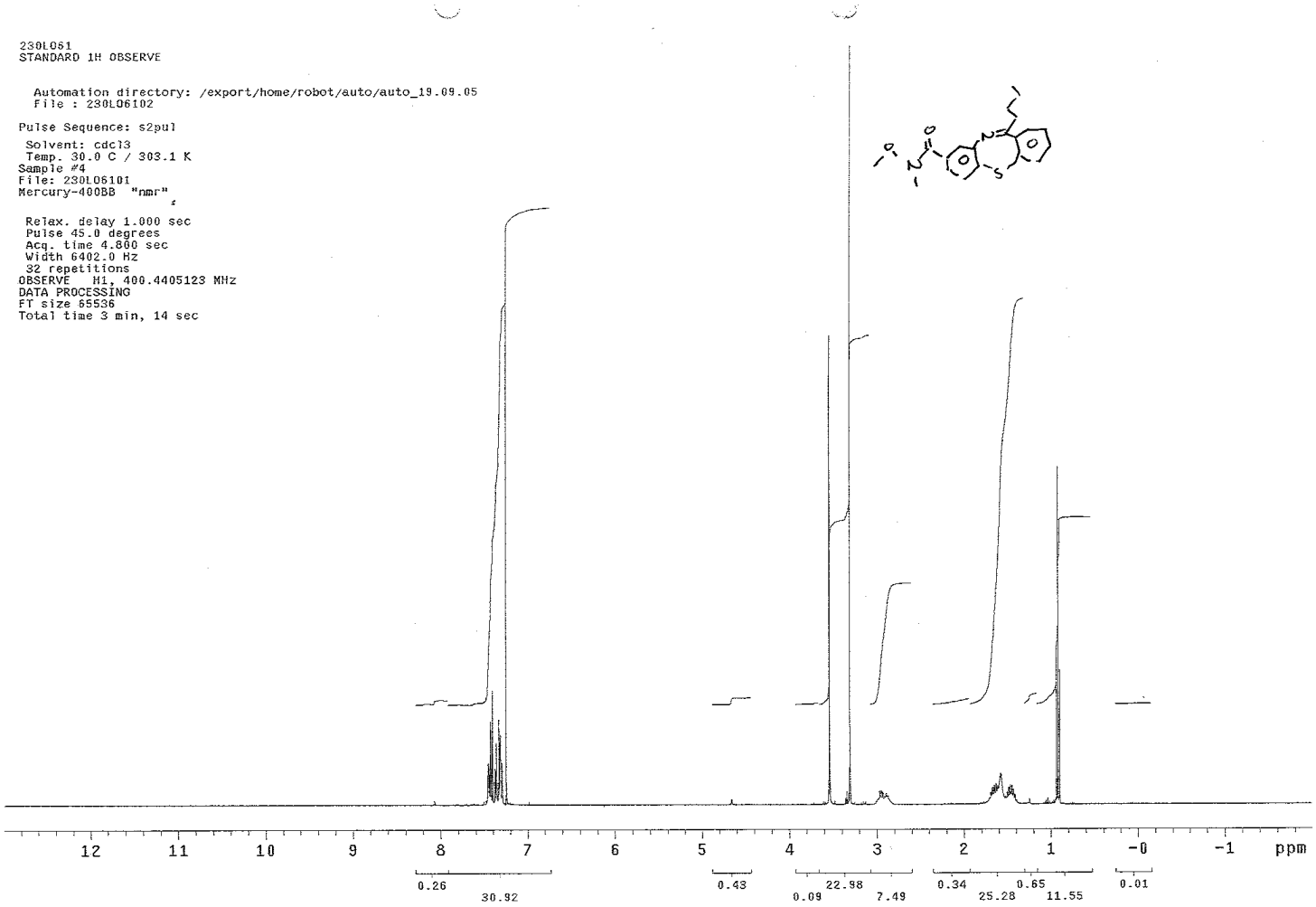

230L024-II
STANDARD 1H OBSERVE
Automation directory: /export/home/robot/auto/auto_10.08.05
File : 2S0L024-II02
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 30.0 C / 303.1 K
Sample #4
File: 230L024-II01
Mercury-400BB "nmr"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 4.800 sec
Width 6402.0 Hz
32 repetitions
OBSERVE H1, 400.4405123 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 14 sec
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
-0.01 -0.12
-0.06
38.85
0.05
0.12
4.86
54.95
0.37


230L024-III
STANDARD 1H OBSERVE
Automation directory: /export/home/robot/auto/auto_09.08.05
File : 230L024-III02
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 30.0 C / 303.1 K
Sample #9
File: 230L024-III01
Mercury-400BB "nmr"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 4.800 sec
Width 6402.0 Hz
32 repetitions
OBSERVE H1, 400.4405123 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 14 sec
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
-0.14
66.05
0.07
26.17
0.51
0.14
6.08
0.85
0.27


203fetertbu
STAMDARD 1H OBSERVE
expt s2pul
SAMPLE
date Dec 1 2005
solvent cdcl3
file /export/home/~
robot/auto/auto_01~
.12.05/203fetertbu~
ACQUISITION
sw 6402.0
at 4.800
np 61458
fb not used
bs
bs 16
d1 1.000
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 400.443
tof 339.3
tpwr 58
pw 7.050
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 45
dmf 18500
temp
gain
hst
pw90
01x alfa
SPECIAL
not used
FLAGS
il n
in n
dp y
hs nn
PROCESSING
not used
fn
DISPLAY
sp -5.9
wp 3609.1
rfl 3704.8
rfp 2907.2
rp -21.1
lp 11.6
PLOT
wc 285
sc 0
vs 37
th 4
h2pma
al cdc ph 13.62
8
7
6
5
4
3
2
1
ppm
1.00 1.90
1.02 3.91
5.04


230L072
STANDARD 1H OBSERVE
Automation directory: /export/home/robot/auto/auto_09.09.05
File : 230L07202
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 30.0 C / 303.1 K
Sample #1
File: 230L07201
Mercury-400BB "nmr"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 4.800 sec
Width 6402.0 Hz
32 repetitions
OBSERVE H1, 400.4405123 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 14 sec
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
0.43
9.41
63.65
0.55
0.16
23.57
1.64
0.51
0.07

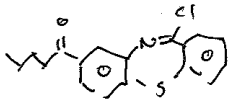

230L057
STANDARD 1H OBSERVE
Automation directory: /export/home/robot/auto/auto_20.07.05
File : 230L05702
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 30.0 C / 303.1 K
Sample #1
File: 230L05701
Mercury-400BB "nmr"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 4.800 sec
Width 6492.0 Hz
32 repetitions
OBSERVE H1, 400.4405123 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 14 sec
CH2Cl2
Cl
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
0.09 46.35
0.07 0.06
0.02 0.06
2.74 0.14
0.27
38.61
-0.03 0.22 5.37
0.30 0.57 5.19

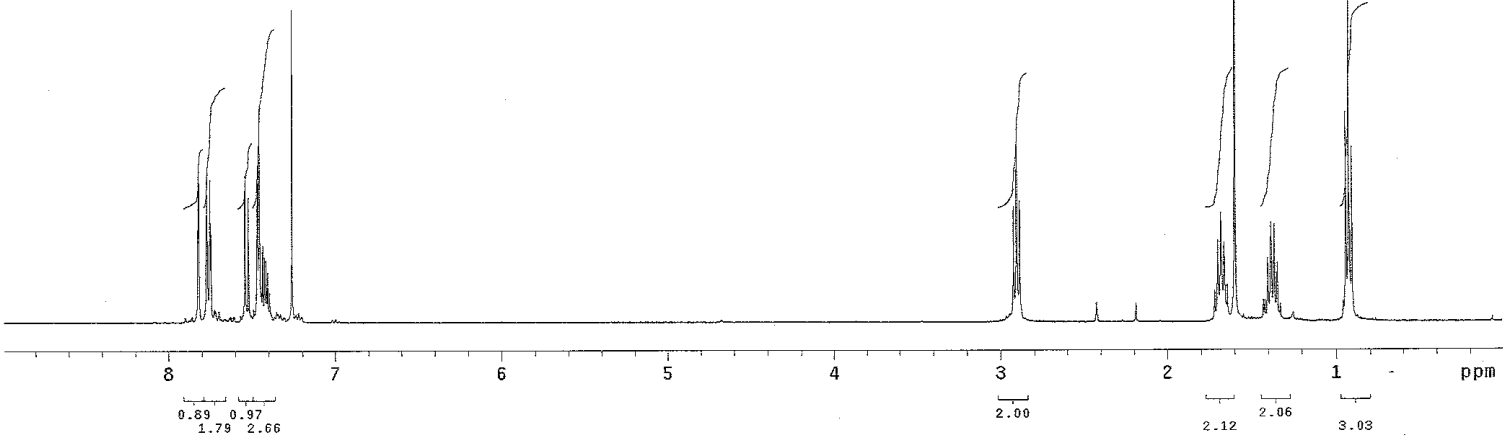

230L051
STANDARD 1H OBSERVE
Automation directory: /export/home/robot/auto/auto_19.09.05
File : 230L06102
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 30.0 C / 303.1 K
Sample #4
File: 230L06101
Mercury-400BB "nmr"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 4.800 sec
Width 6402.0 Hz
32 repetitions
OBSERVE H1, 400.4405123 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 14 sec
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
0.26
30.92
0.43
0.09
22.98
7.49
0.34
25.28
0.65
11.55
0.01

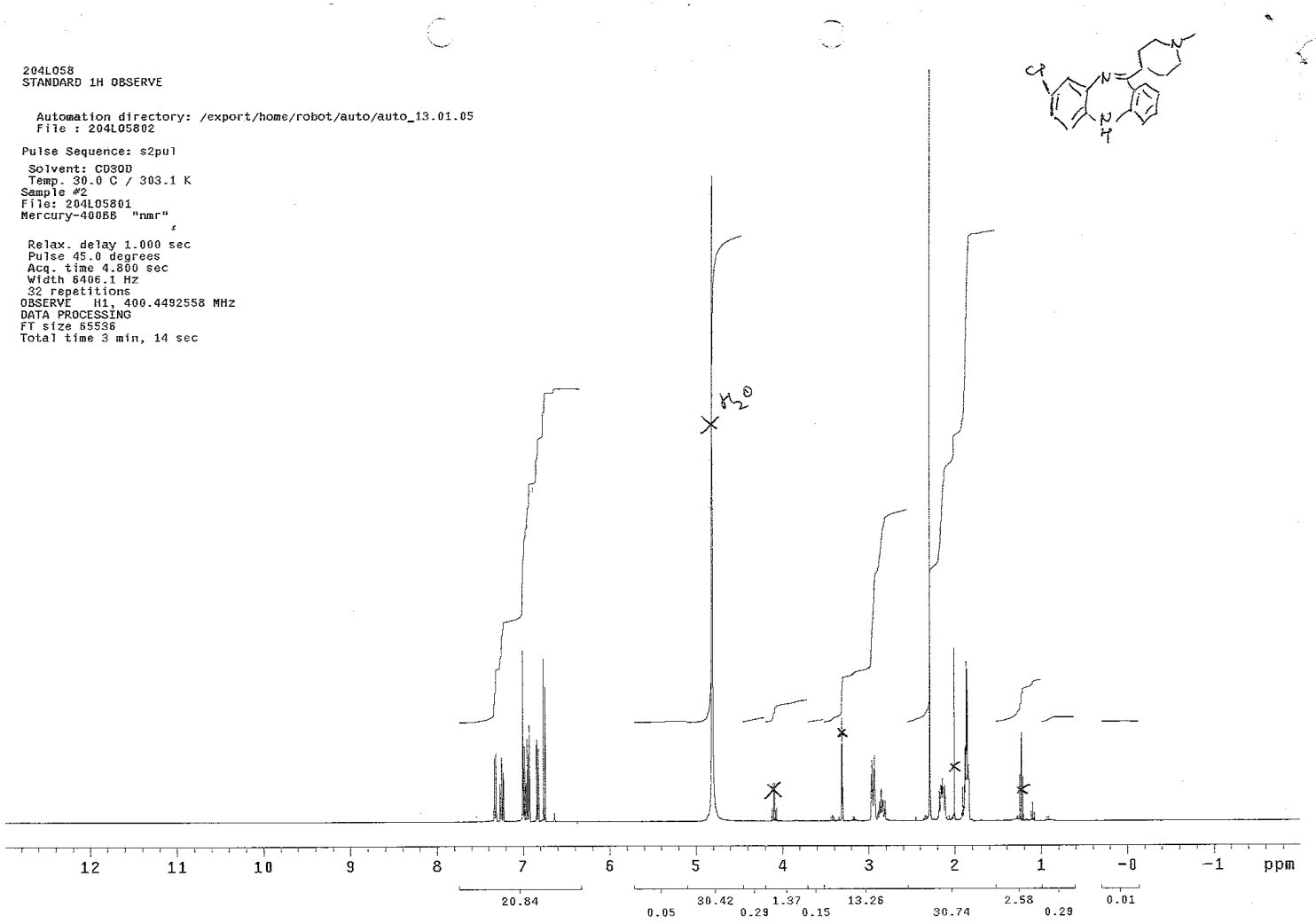
230L059
STANDARD 1H OBSERVE

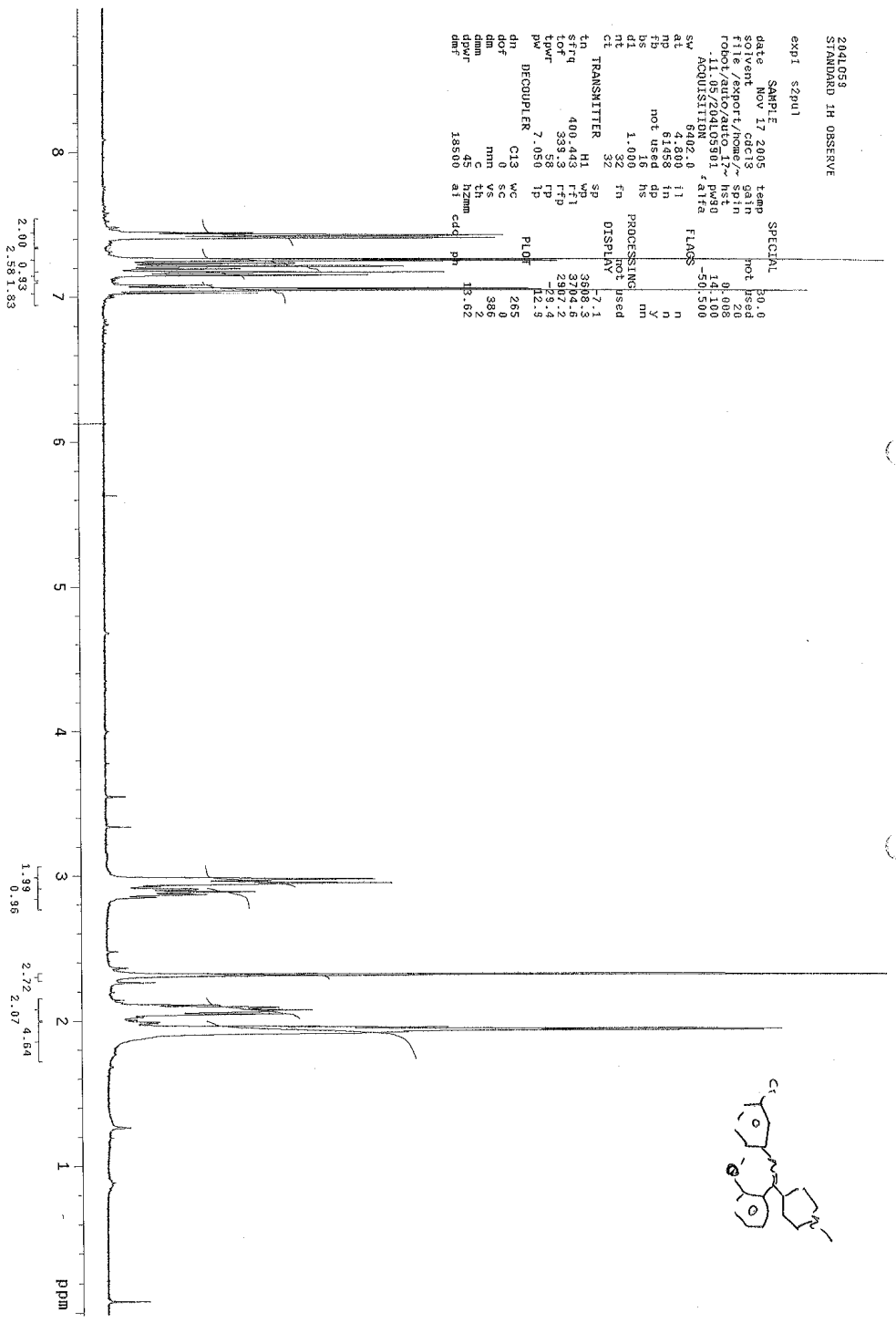
exp1 s2pul

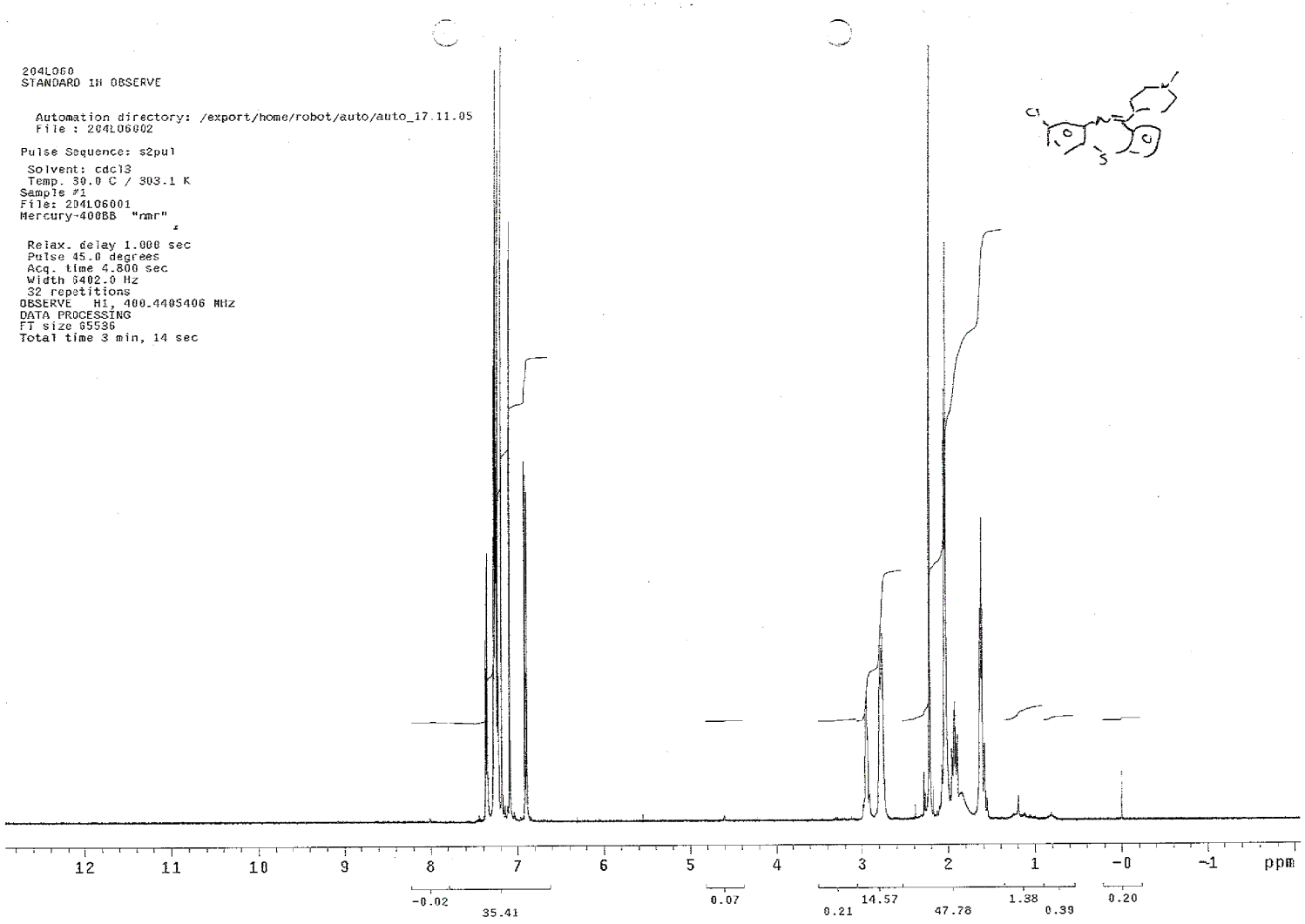
SAMPLE		SPECIAL	
date	Jul 29 2005	temp	30.0
solvent	cdcl3	gain	not used
file	/export/home/~	spin	20
robot/auto/auto_29~		hst	0.008
.07.05/230L059O1.f~		pw90	14.100
id	alfa		-50.560
ACQUISITION		**FLAGS**	
sw	6402.0	il	n
at	4.800	in	n
np	61458	dp	y
fb	not used	hs	nn
bs	16	**PROCESSING**	
d1	1.000	fn	not used
nt	32	**DISPLAY**	
ct	32	sp	2.5
TRANSMITTER		wp	3597.2
tn	H1	rfl	798.4
sfrq	400.443	rfp	0
tof	339.3	rp	-32.8
tpwr	58	lp	14.3
pw	7.050	**PLOT**	
DECOUPLER		wc	265
dn	C13	sc	0
dof	0	vs	50
dm	nnn	th	47
dmm	c	hzmm	13.57
dpwr	45	ai cdc ph	
dmf	18500		

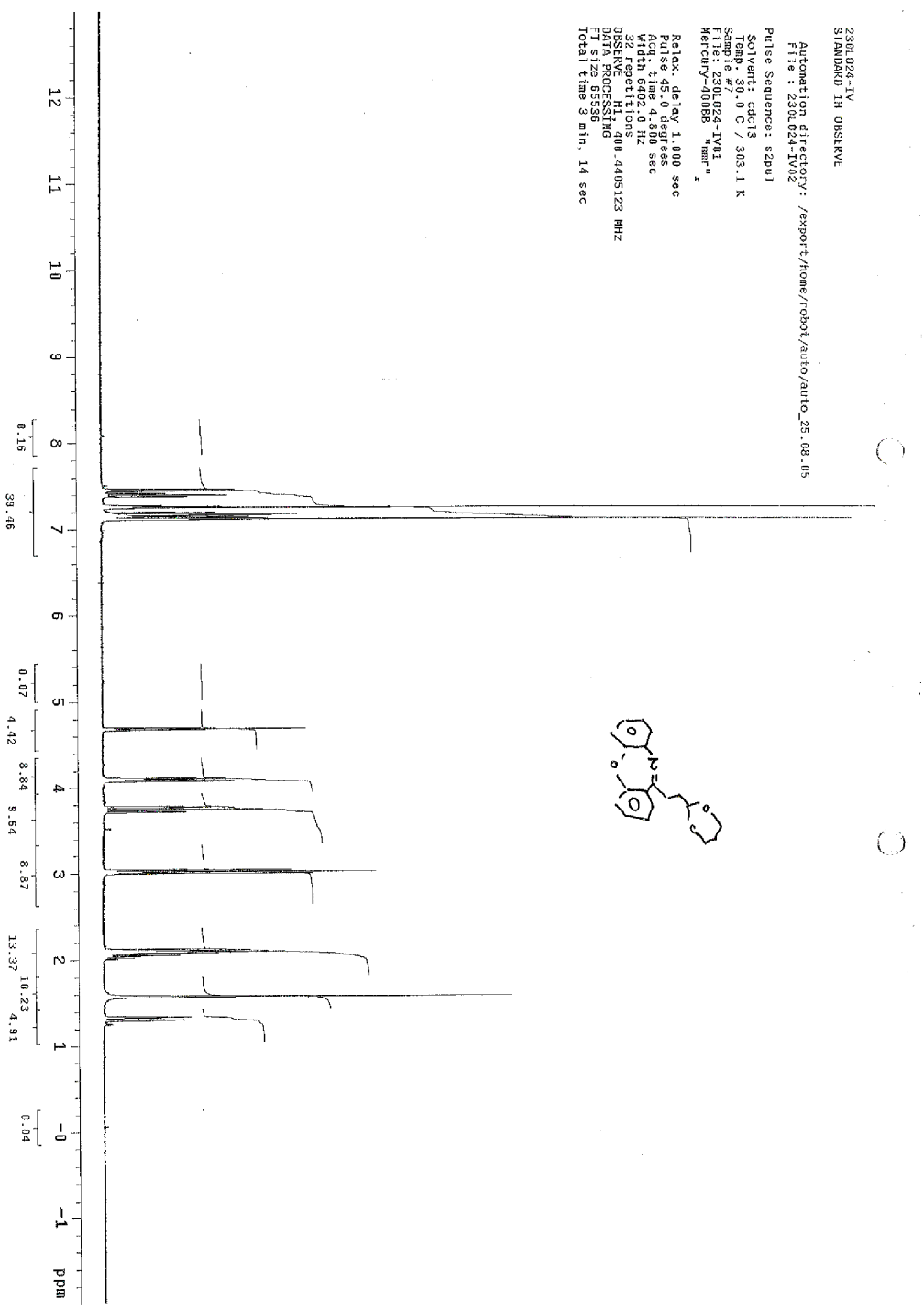





204L058
STANDARD 1H OBSERVE
Automation directory: /export/home/robot/auto/auto_13.01.05
File : 204L05802
Pulse Sequence: s2pul
Solvent: CD3OD
Temp. 30.0 C / 303.1 K
Sample #2
File: 204L05801
Mercury-400BB "nmr"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 4.800 sec
Width 6406.1 Hz
32 repetitions
OBSERVE H1, 400.4492558 MHz
DATA PROCESSING
FT size 55536
Total time 3 min, 14 sec
H2O
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
20.84
0.05
30.42
0.29
1.37
0.15
13.26
30.74
2.58
0.29
0.01


204LO59
STANDARD 1H OBSERVE
expl s2pul
SAMPLE
date Nov 17 2005
solvent cdcl3
file /export/home/~
robot/auto/auto_17~
ACQUISITION
sw 6402.0
at 4.800
np 61458
fb not used
bs 16
d1 1.000
nt 32
ct 32
TRANSMITTER
tn H1
sfrq 400.443
tof 339.3
tpwr 58
pw 7.050
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 45
dmf 18500
SPECIAL
temp 30.0
gain not used
spin 20
hst 0.008
pw90 14.100
alfa -50.500
FLAGS
il n
in n
dp y
hs nn
PROCESSING
not used
fn not used
DISPLAY
sp -7.1
wp 3608.3
rfl 3704.6
rfp 2907.2
rp -29.4
lp 12.9
PLOT
wc 265
sc 0
vs 386
th 2
hzmm 13.62
ai cdd ph
8
7
6
5
4
3
2
1
ppm
2.00 0.93
2.58 1.83
1.99
0.96
2.72
2.07
4.64


204L060
STANDARD IN OBSERVE
Automation directory: /export/home/robot/auto/auto_17.11.05
File : 204L06002
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 30.0 C / 303.1 K
Sample #1
File: 204L06001
Mercury-400BB "nmr"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 4.800 sec
Width 6402.0 Hz
32 repetitions
OBSERVE H1, 400.4405406 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 14 sec
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm
-0.02
35.41
0.07
0.21
14.57
47.78
1.38
0.39
0.20


230L024-IV
STANDARD 1H OBSERVE
Automation directory: /export/home/robot/auto/auto_25.08.05
File : 230L024-IV02
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. : 30.0 C / 303.1 K
Sample #7
File: 230L024-IV01
Mercury-400BB "nmr"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 4.580 sec
Width 8012.8 Hz
32 repetitions
OBSERVE H1, 400.4405123 MHz
DATA PROCESSING
FT size 65536
Total time 3 min, 14 sec
8.16
39.46
0.07
4.42
8.84
9.64
8.87
13.37
10.23
4.91
0.04
12
11
10
9
8
7
6
5
4
3
2
1
-0
-1
ppm