Sample Name:
6421JPS85a
Data collected on:
Varian-NMR600-vnmrs600
Archive directory:
/home/walkup/vnmrsys/data
Sample directory:
6421JPS85a_20100726_01
FidFile: 6421JPS85a_1H

Pulse Sequence: PROTON (s2pul)
Solvent: cd3od
Data colected on: Jul 26 2010

Temp. 25.0 C / 298.1 K
Operator: walkup

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.704 sec
Width 9615.4 Hz
8 repetitions
OBSERVE H1, 599.8697736 MHz
DATA PROCESSING
FT size 32768
Total time 0 min 22 sec

Index	Frequency	PPM	HEIGHT
1	4347.5	7.247	7.1
2	4344.6	7.243	10.5
3	4338.7	7.233	7.4
4	4335.8	7.228	10.3
5	4329.9	7.218	20.3
6	4327.0	7.213	12.1
7	4052.9	6.756	18.0
8	4044.1	6.742	16.8
9	2980.3	4.835	62.0
10	1980.1	3.301	12.0
11	1978.3	3.298	27.5
12	1976.5	3.295	37.8
13	1974.8	3.292	20.2
14	1973.6	3.290	8.3
15	1926.1	3.211	185.3
16	1623.8	2.707	142.7

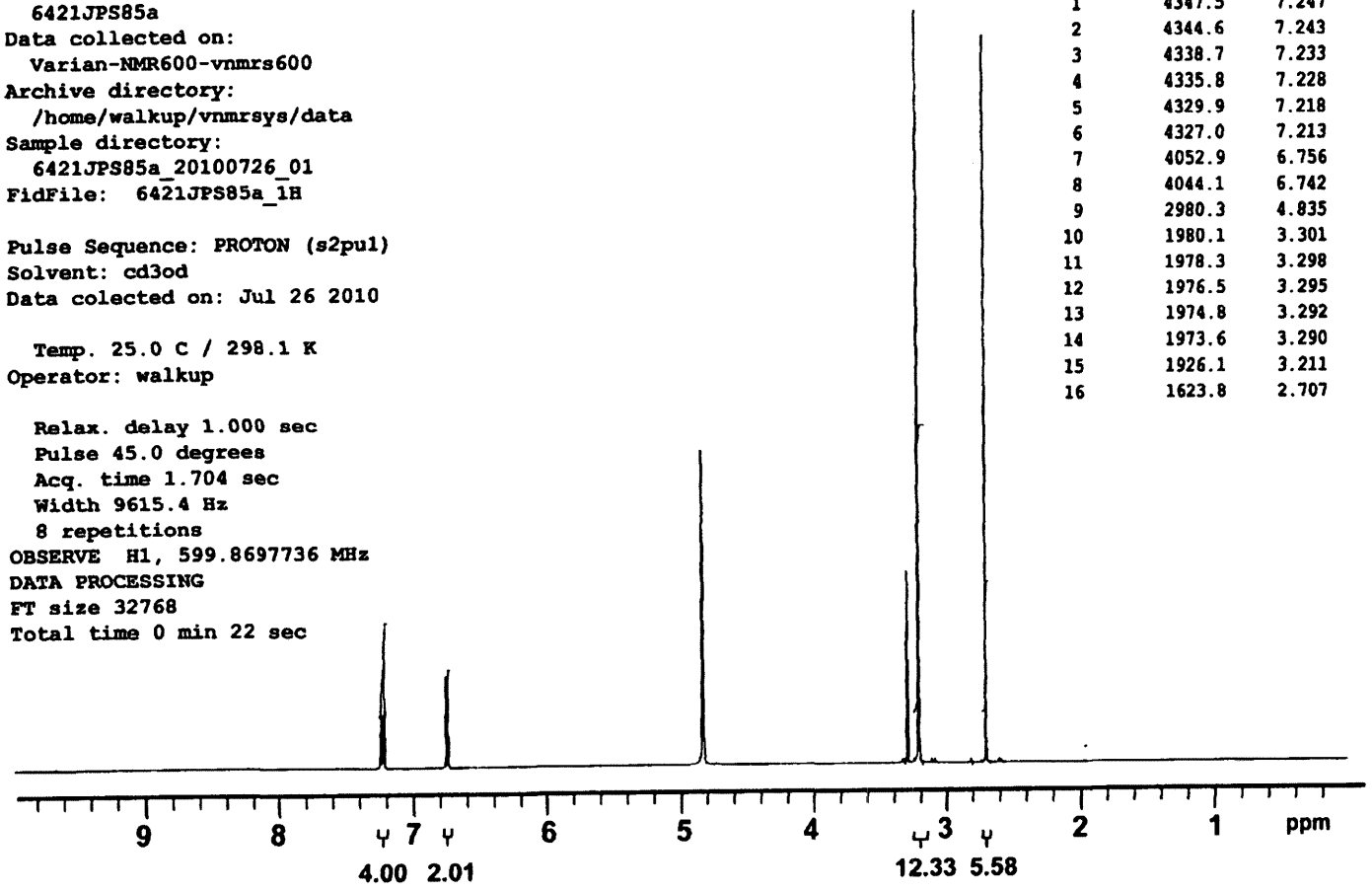


Figure 1:

Figure 2:

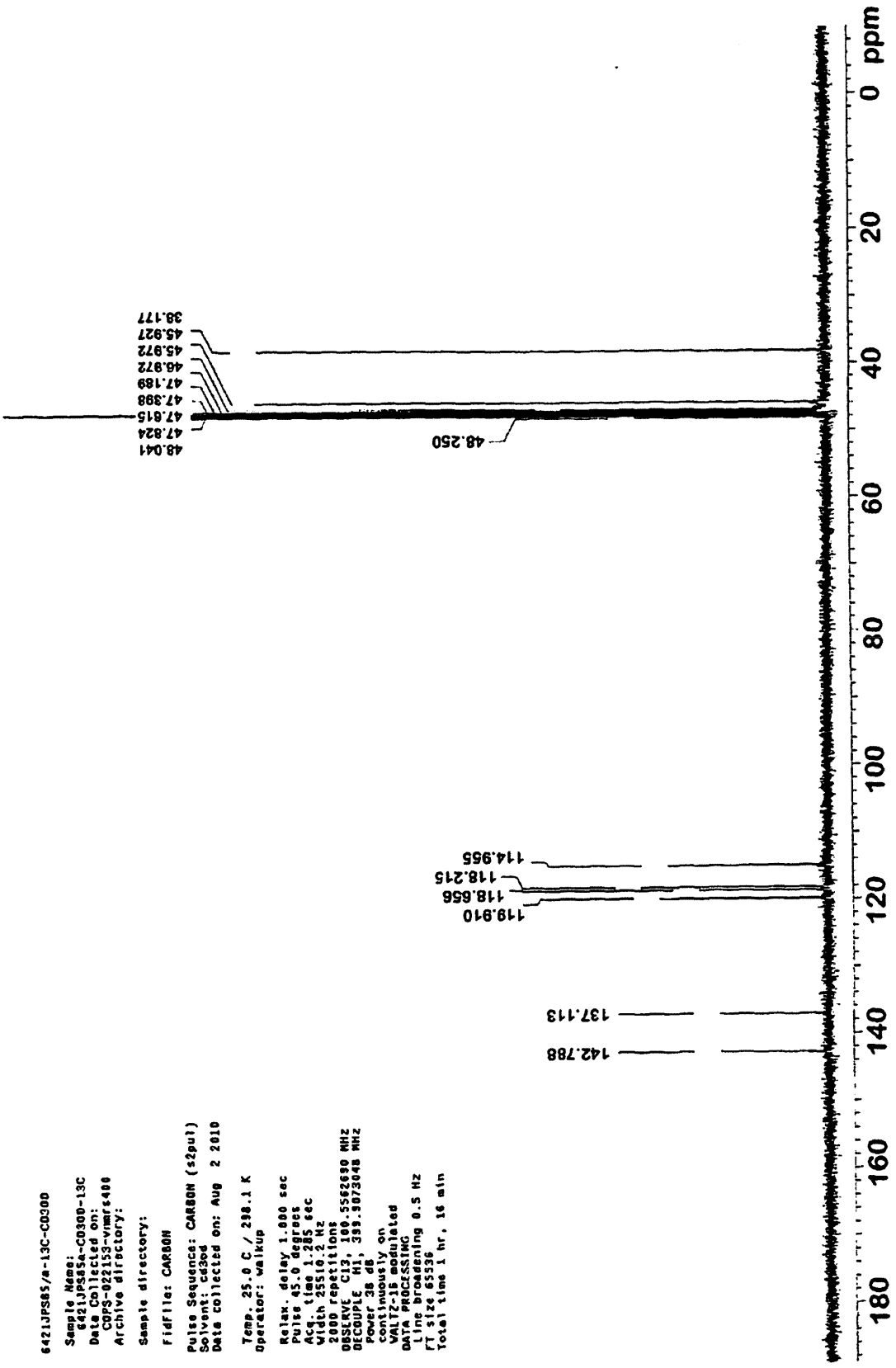

642iJPS85/a-13C-CD30D
Sample Name:
642iJPS85A-CD30D-13C
Data Collected on:
COPS-022153-vmrs400
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cd3od
Data collected on: Aug 2 2010
Temp. 25.0 C / 298.1 K
Operator: walkup
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
2000 repetitions
OBSERVE C13, 100.5562630 MHz
DECOUPLE H1, 399.3073048 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 16 min
38.177
45.927
45.972
46.972
47.189
47.398
47.615
47.824
48.041
48.250
114.955
118.215
118.656
119.910
137.113
142.788
0 ppm
20
40
60
80
100
120
140
160
180

Figure 3:

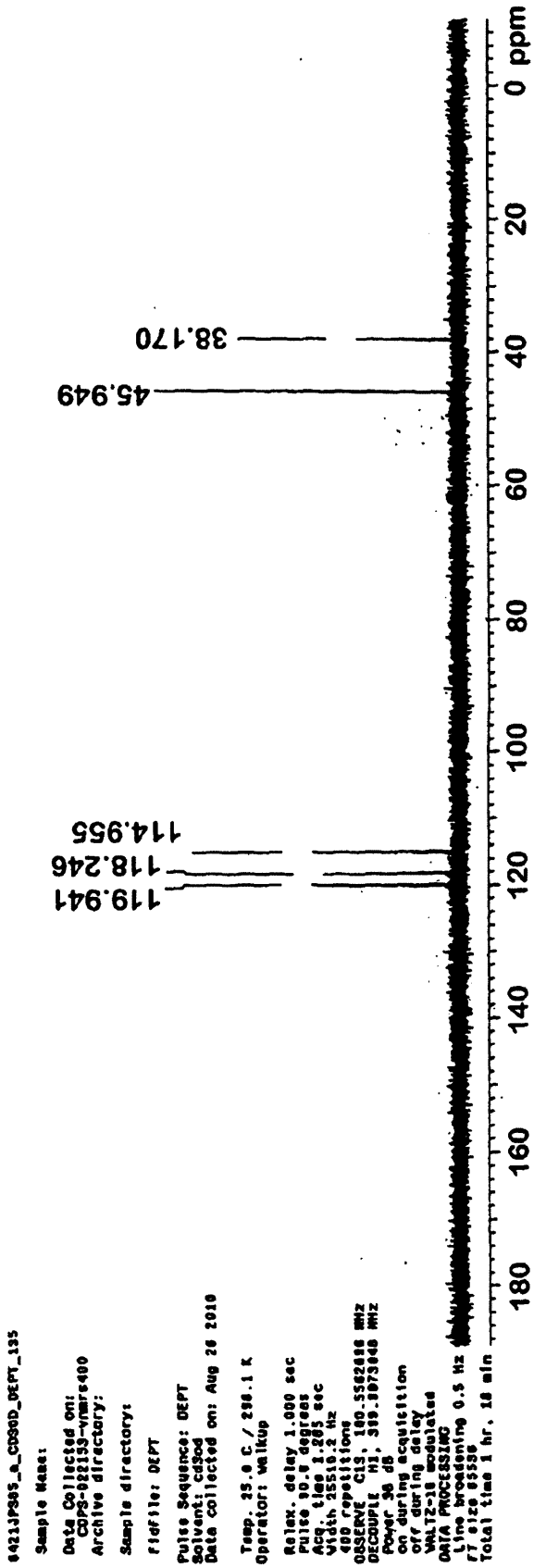

0421JP305_a_CD30D_DEPT_135
Sample Name:
Data Collected on:
CDPS-022133-vnmrs400
Archive directory:
Sample directory:
Fidfile: DEPT
Pulse Sequence: DEPT
Solvent: cd3od
Data collected on: Aug 26 2010
Temp. 25.0 C / 298.1 K
Operator: walkup
Relax. delay 1.000 sec
Pulse 10.8 degrees
Acq. time 1.285 sec
Width 25510.2 Hz
400 repetitions
OBSERVE C13, 100.5582486 MHz
DECOUPLE H1, 399.8873848 MHz
Power 36 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 hr, 18 min
38.170
45.949
114.955
118.246
119.941
180
160
140
120
100
80
60
40
20
0 ppm

Figure 4:

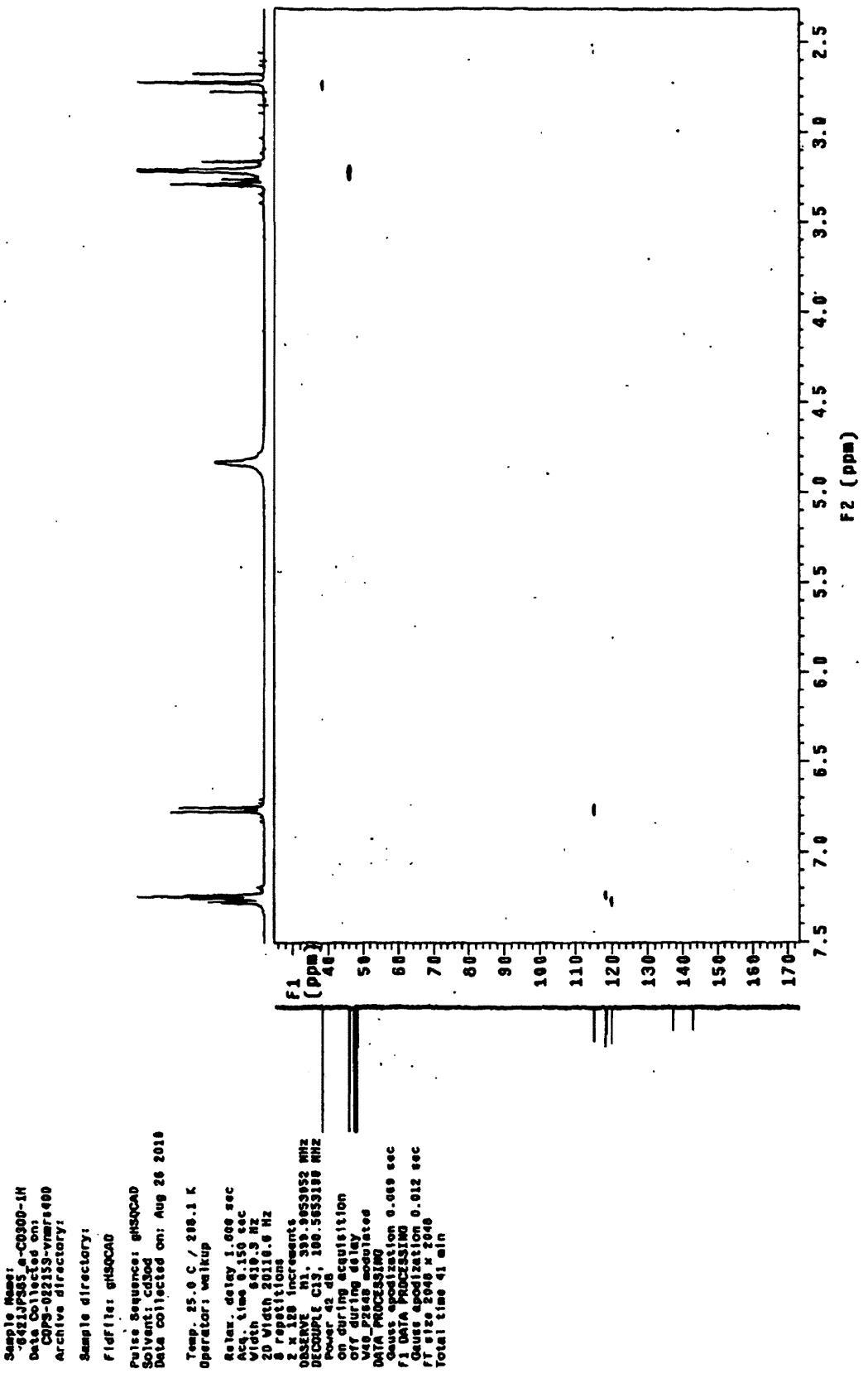

Sample Name:
-0421JP385_a-CD3OD-1H
Data Collected on:
CDP3-022153-vnmrs400
Archive directory:
Sample directory:
FidFile: gHSQCAD
Pulse Sequence: gHSQCAD
Solvent: cd3od
Data collected on: Aug 26 2010
Temp. 25.0 C / 298.1 K
Operator: walkup
Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 6410.3 Hz
2D Width 20118.6 Hz
8 repetitions
2 x 128 increments
OBSERVE H1, 399.9853052 MHz
DECOUPLE C13, 100.5653188 MHz
Power 42 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Gauss apodization 0.068 sec
F1 DATA PROCESSING
Gauss apodization 0.012 sec
FT size 2048 x 2048
Total time 41 min
F1 (ppm)
F2 (ppm)

Figure 5:

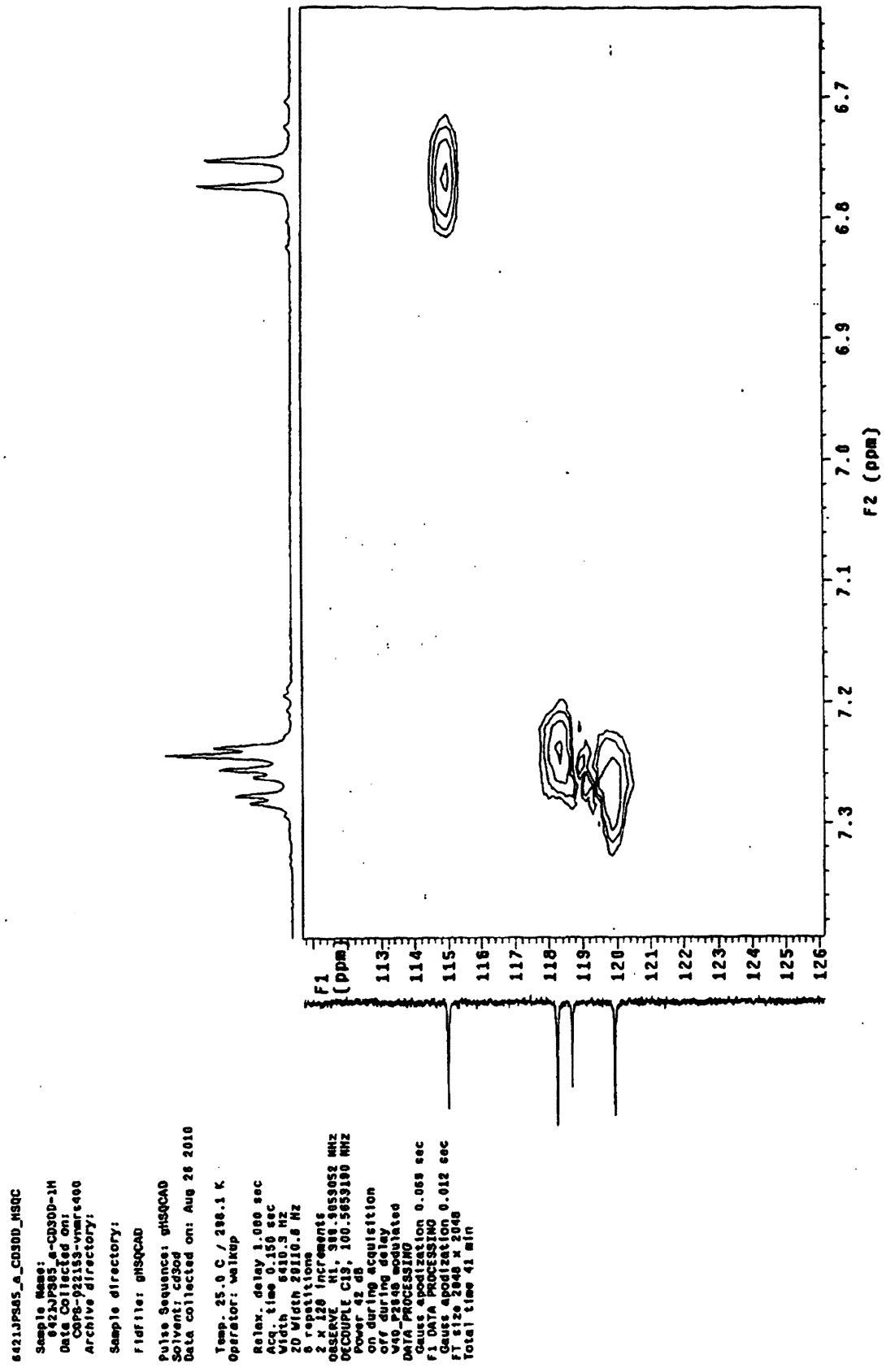
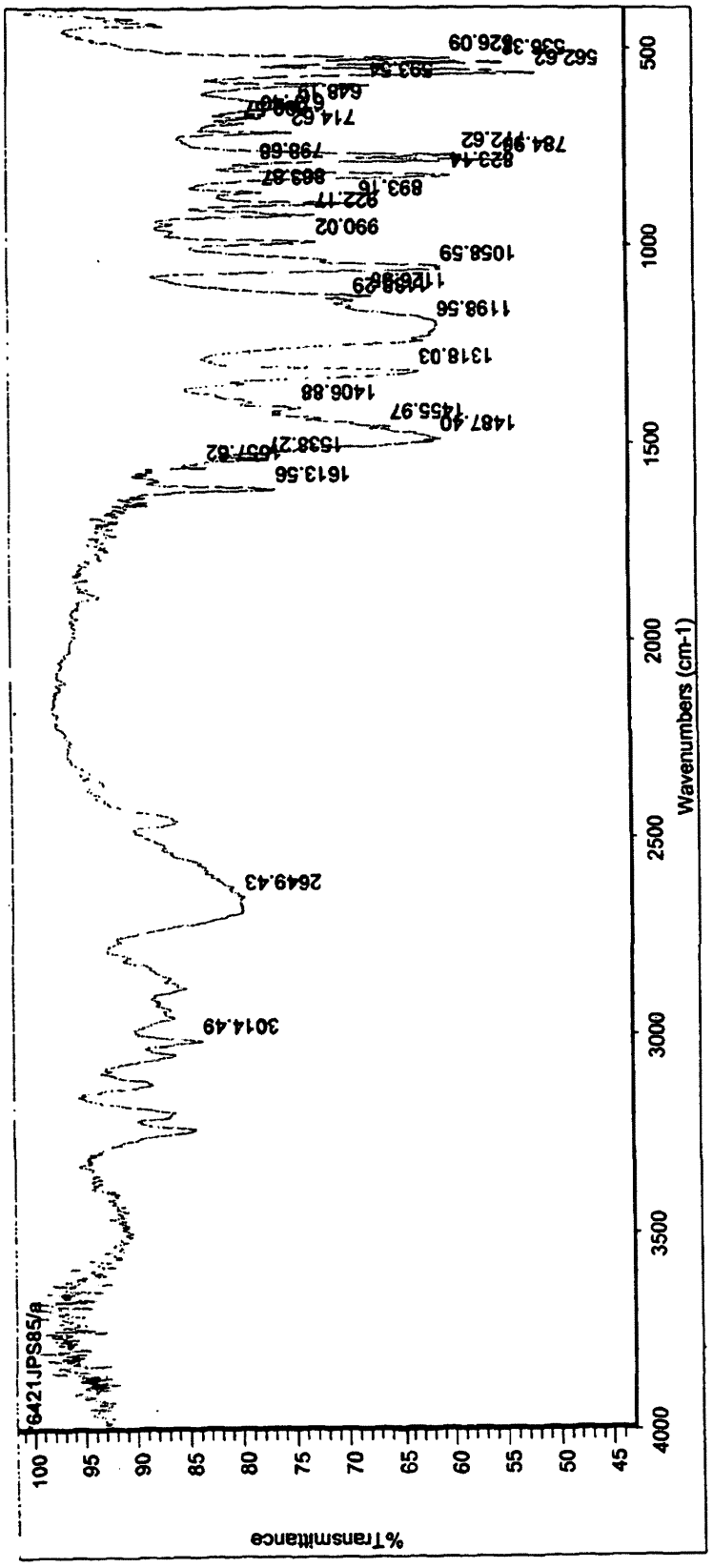

6421JPS65_a_CD30D_HSQC
Sample Name:
6421JPS65_a-CD30D-1H
Data Collected on:
COPS-02155.vnmrs400
Archive directory:
Sample directory:
Filefile: ghSQCAD
Pulse Sequence: ghSQCAD
Solvent: cd3od
Data collected on: Aug 26 2010
Temp. 25.0 C / 298.1 K
Operator: walkup
Relax. delay 1.000 sec
Acq. time 0.150 sec
Width 6410.3 Hz
2D Width 20110.8 Hz
8 repetitions
2 x 128 increments
OBSERVE H1, 399.9659052 MHz
DECOUPLE C13, 100.5653190 MHz
Power 42 dB
on during acquisition
off during delay
WALTZ16 modulated
DATA PROCESSING
Gauss apodization 0.059 sec
F1 DATA PROCESSING
Gauss apodization 0.012 sec
FT size 2048 x 2048
Total time 41 min
F1 (ppm)
F2 (ppm)

Figure 6:



Mon May 03 00:42:11 2010
FIND PEAKS:
Spectrum: '6421JPS85/a
Region: 4000.00 400.00
Absolute threshold: 84.166
Sensitivity: 50
Peak list:

Position:	526.09	Intensity:	59.505
Position:	536.32	Intensity:	54.578
Position:	562.62	Intensity:	51.500
Position:	593.54	Intensity:	67.112
Position:	648.19	Intensity:	73.812
Position:	677.40	Intensity:	77.342
Position:	690.57	Intensity:	78.882

[Nicolet IR100]

Figure 7:

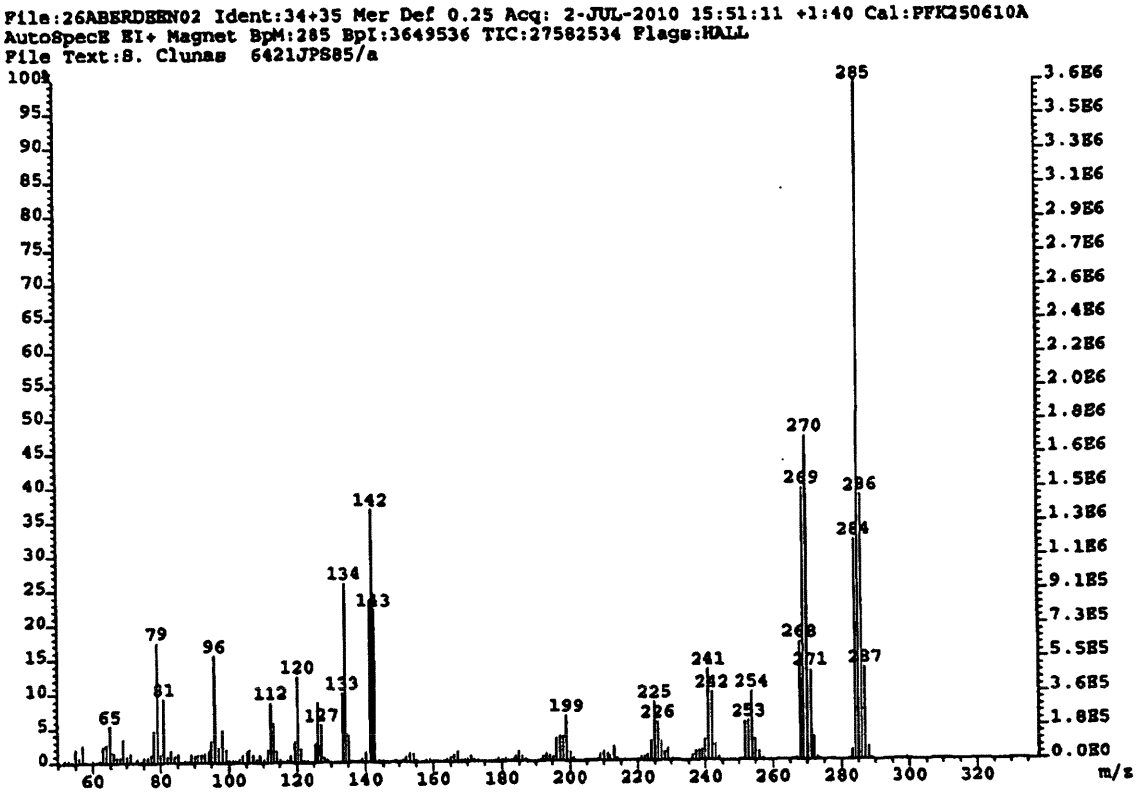

File:26ABERDEEN02 Ident:34+35 Mer Def 0.25 Acq: 2-JUL-2010 15:51:11 +1:40 Cal:PFK250610A
AutoSpecE EI+ Magnet BpM:285 BpI:3649536 TIC:27582534 Flags:HALL
File Text:8. Clunas 6421JPS85/a
285
270
269
286
284
142
134
143
79
96
120
133
112
127
81
65
199
225
226
241
242
254
253
268
271
287
m/z

Figure 8:

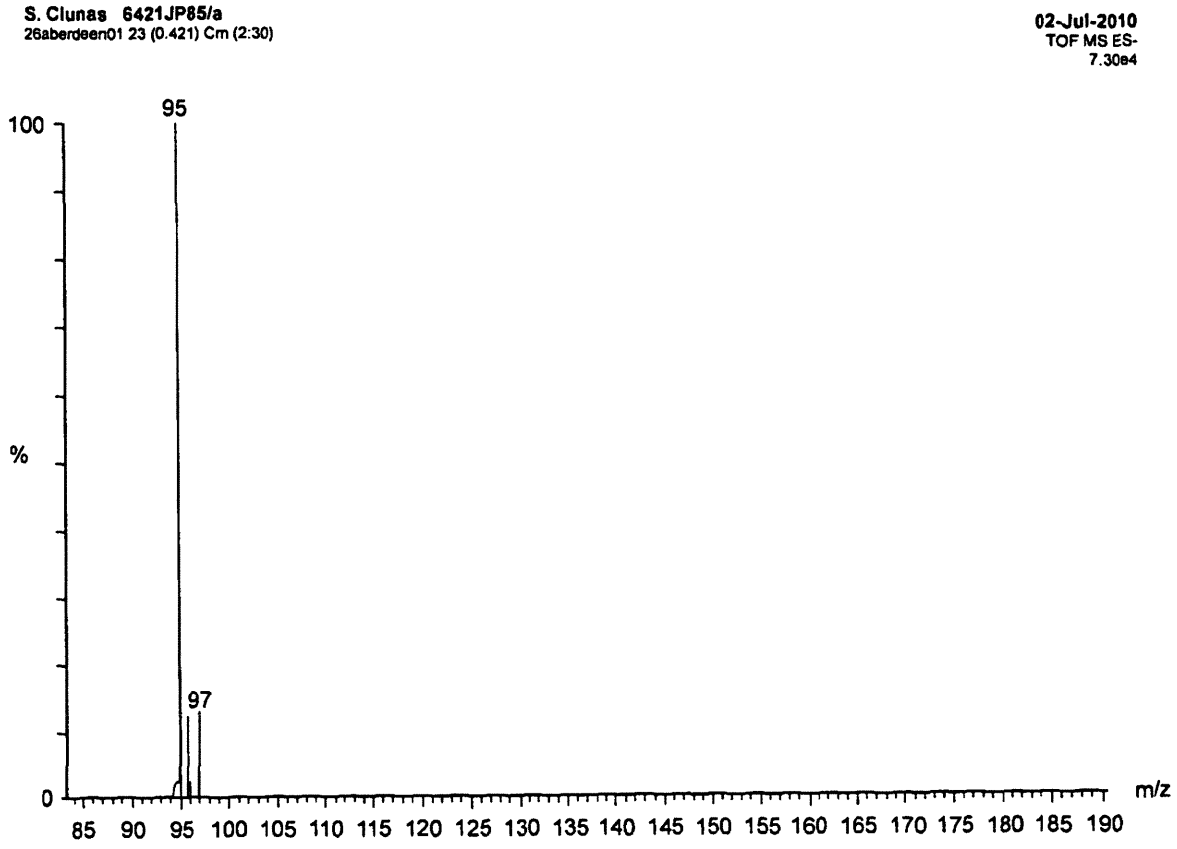

S. Clunas 6421JP85/a
26aberdeen01 23 (0.421) Cm (2:30)
02-Jul-2010
TOF MS ES-
7.30e4
100
%
0
95
97
m/z
85 90 95 100 105 110 115 120 125 130 135 140 145 150 155 160 165 170 175 180 185 190

Figure 9:

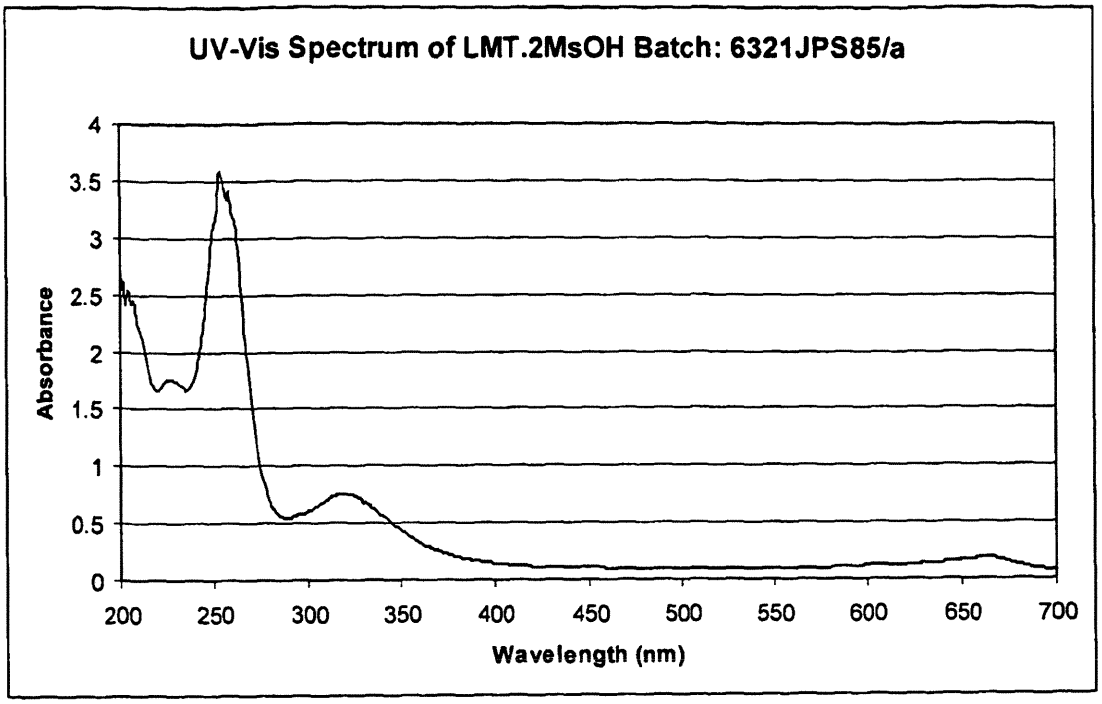

UV-Vis Spectrum of LMT.2MsOH Batch: 6321JPS85/a
Absorbance
4
3.5
3
2.5
2
1.5
1
0.5
0
200
250
300
350
400
450
500
550
600
650
700
Wavelength (nm)

Figure 10:

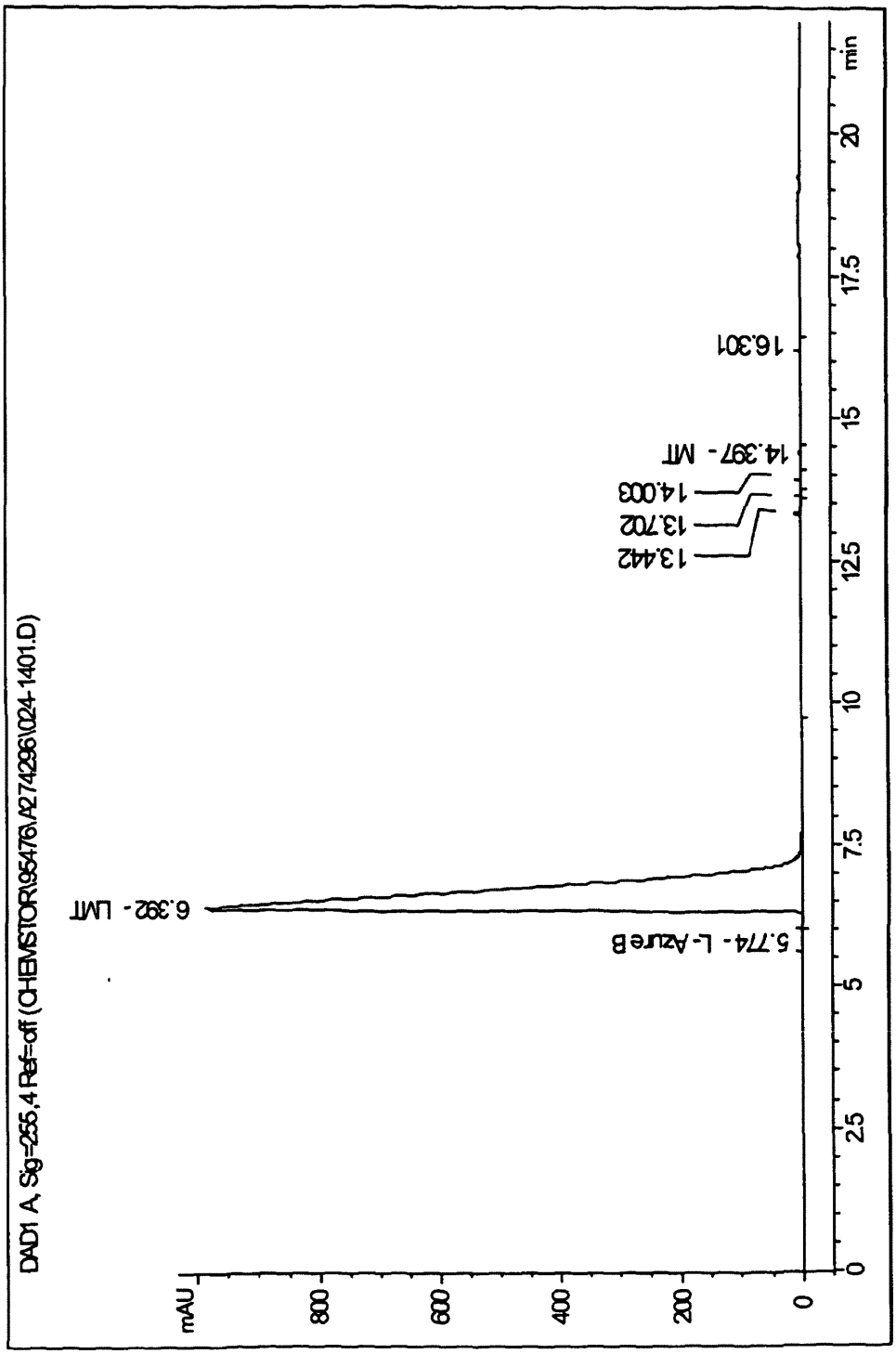

DAD1 A, Sig=255,4 Ref=off (CHEMSTOR\95476\A274296\024-1401.D)
mAU
800
600
400
200
0
0
2.5
5
7.5
10
12.5
15
17.5
20
min
6.392 - LMT
5.774 - L-Azure B
13.442
13.702
14.003
14.397 - MT
16.301

Figure 11:

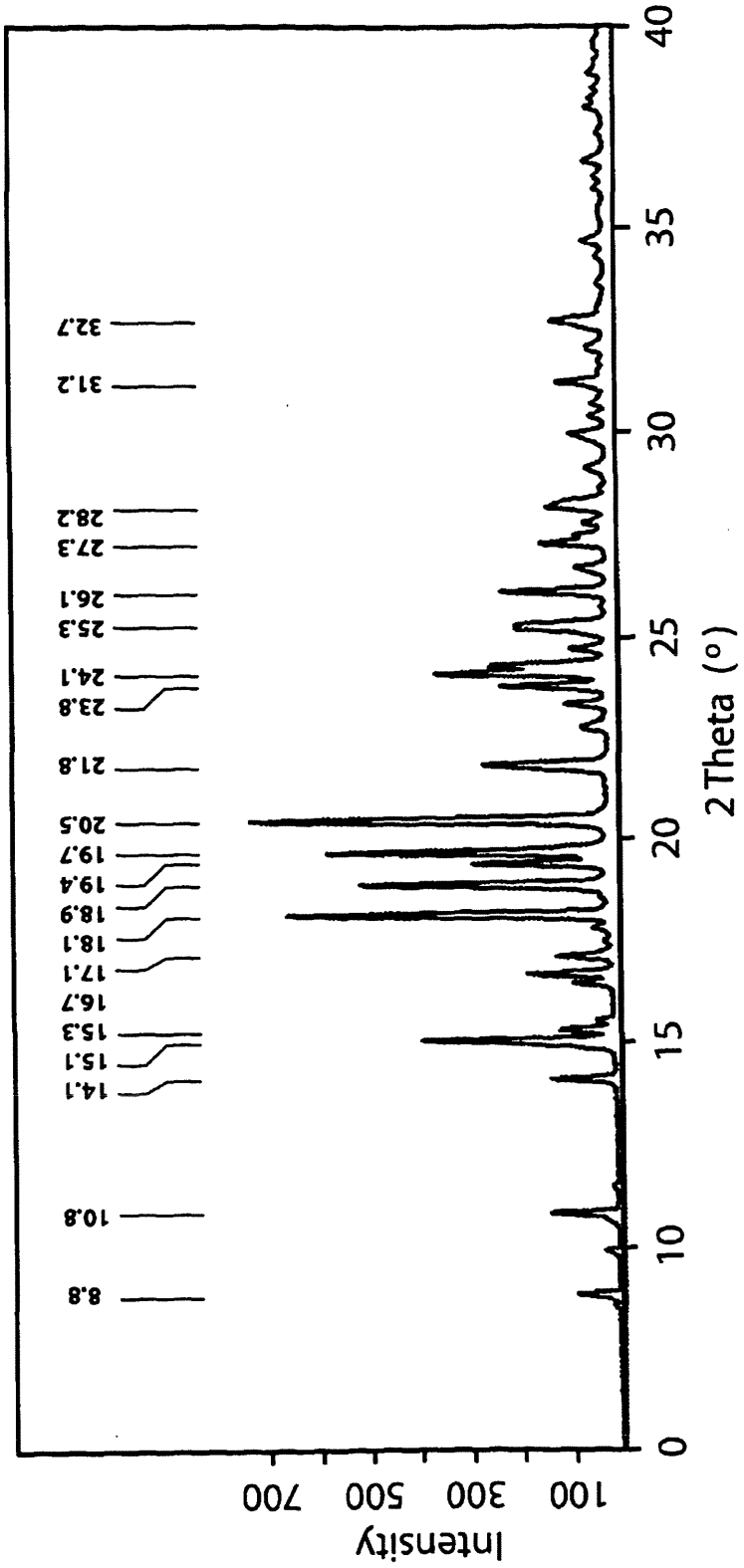

Intensity
100
300
500
700
0
10
15
20
25
30
35
40
2 Theta (°)
8.8
10.8
14.1
15.1
15.3
16.7
17.1
18.1
18.9
19.4
19.7
20.5
21.8
23.8
24.1
25.3
26.1
27.3
28.2
31.2
32.7

Figure 12:

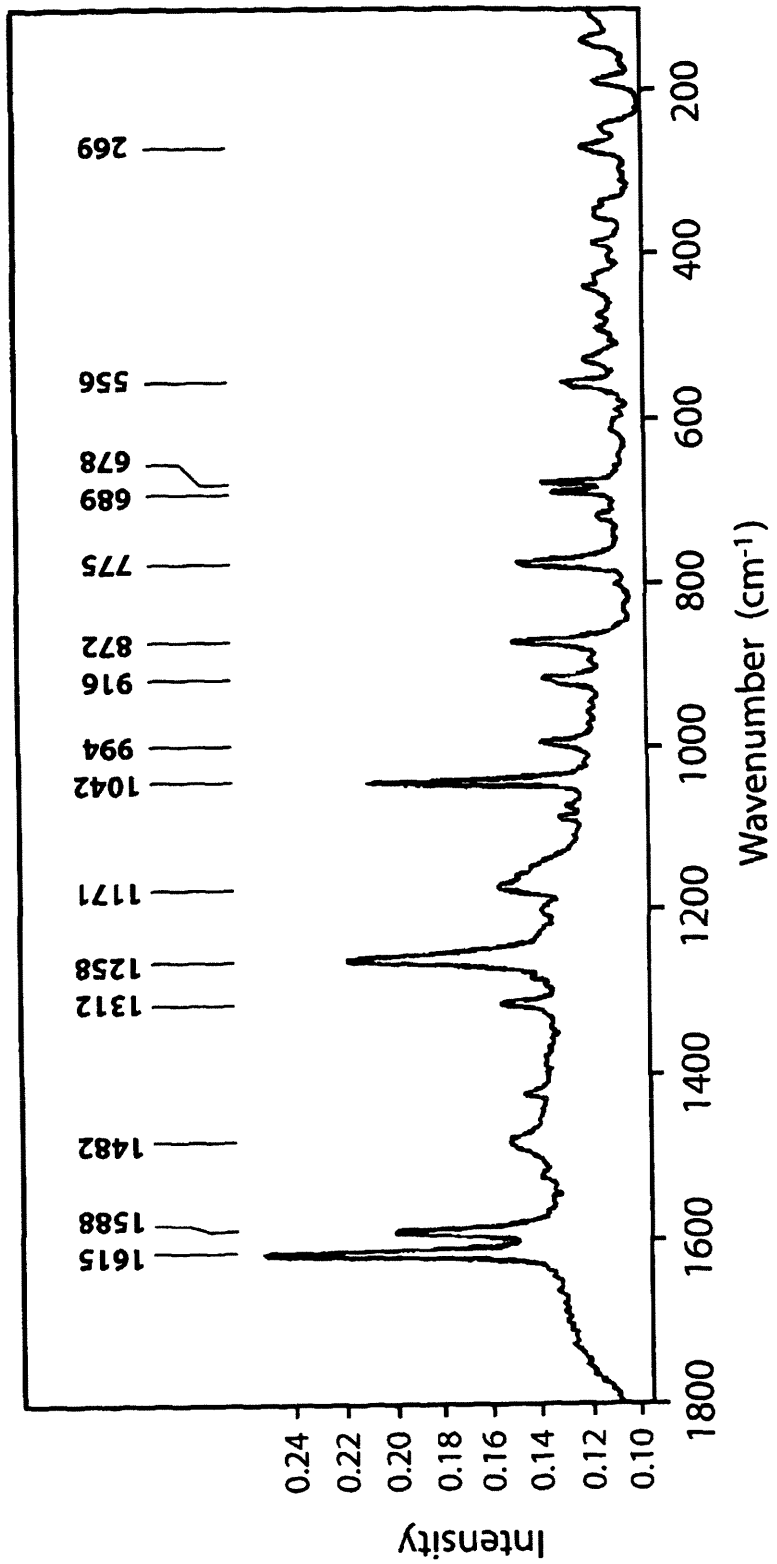

269
556
678
689
775
872
916
994
1042
1171
1258
1312
1482
1588
1615
Intensity
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
1800
1600
1400
1200
1000
800
600
400
200
Wavenumber (cm-1)

Figure 13:

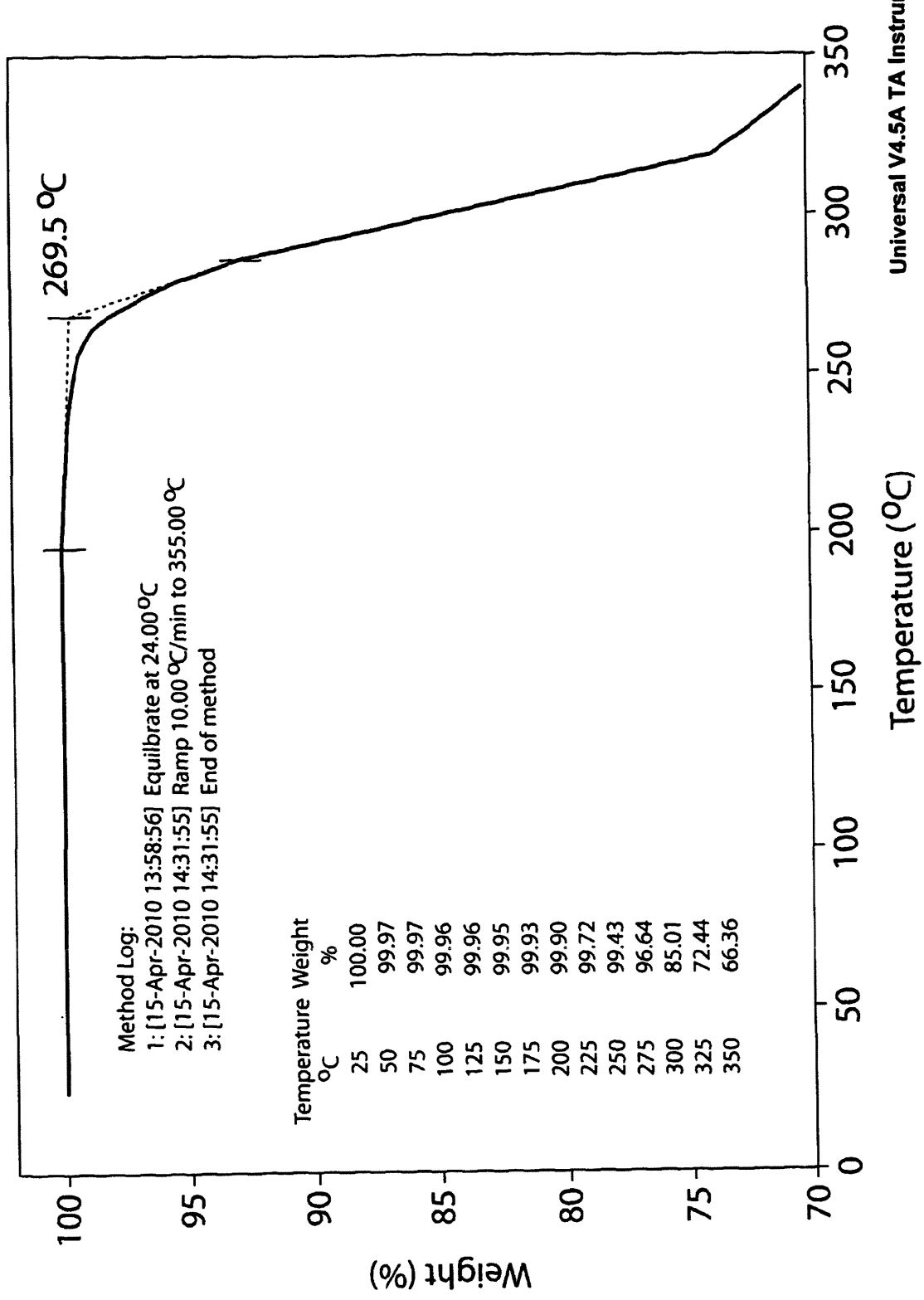

269.5 °C
Method Log:
1: [15-Apr-2010 13:58:56] Equilibrate at 24.00°C
2: [15-Apr-2010 14:31:55] Ramp 10.00°C/min to 355.00°C
3: [15-Apr-2010 14:31:55] End of method
Temperature °C
Weight %
25 100.00
50 99.97
75 99.97
100 99.96
125 99.96
150 99.95
175 99.93
200 99.90
225 99.72
250 99.43
275 96.64
300 85.01
325 72.44
350 66.36
Weight (%)
100
95
90
85
80
75
70
0
50
100
150
200
250
300
350
Temperature (°C)
Universal V4.5A TA Instruments

Figure 14:

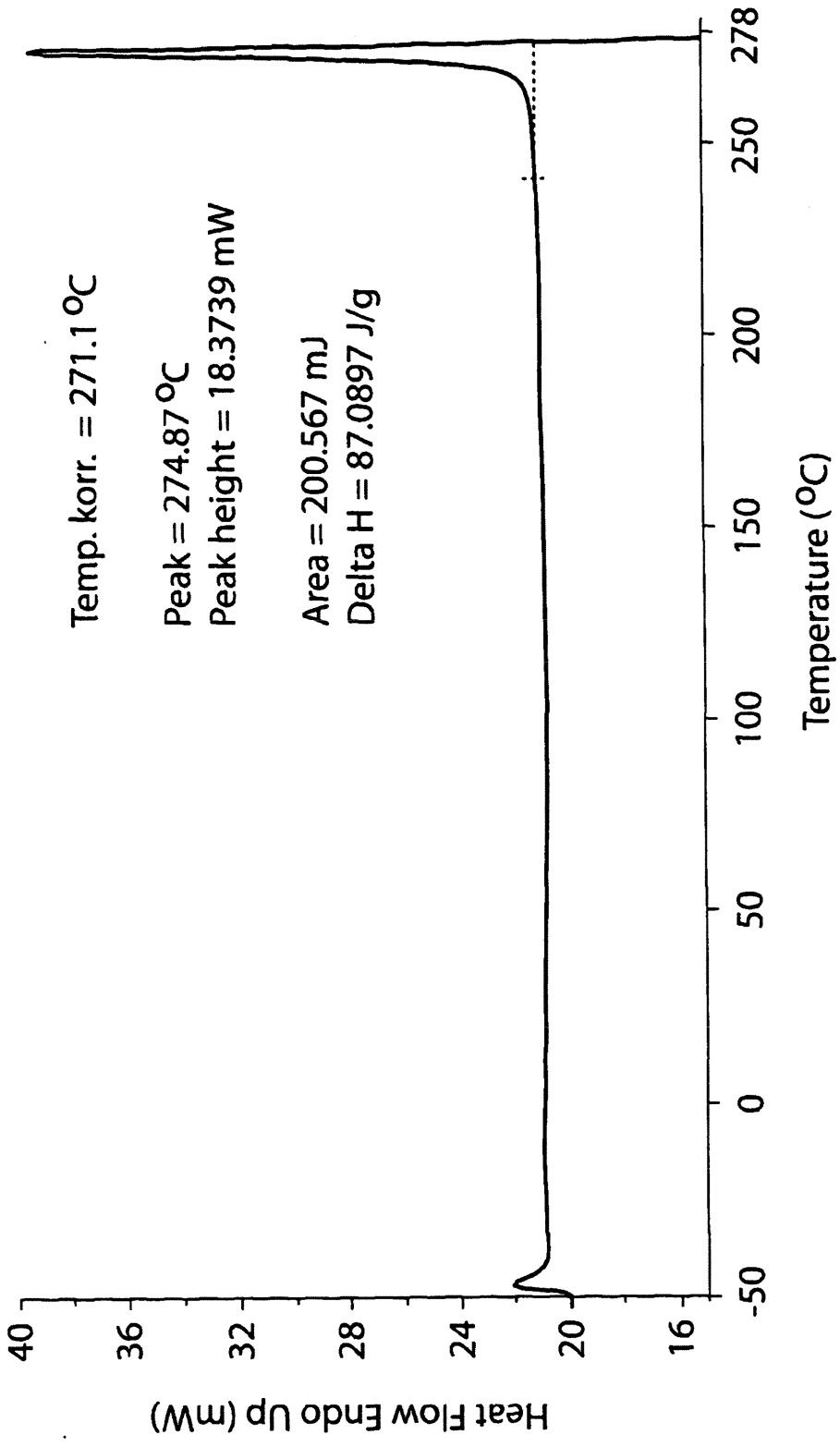

Temp. korr. = 271.1 °C
Peak = 274.87 °C
Peak height = 18.3739 mW
Area = 200.567 mJ
Delta H = 87.0897 J/g
Heat Flow Endo Up (mW)
40
36
32
28
24
20
16
-50
0
50
100
150
200
250
278
Temperature (°C)

Figure 15a:

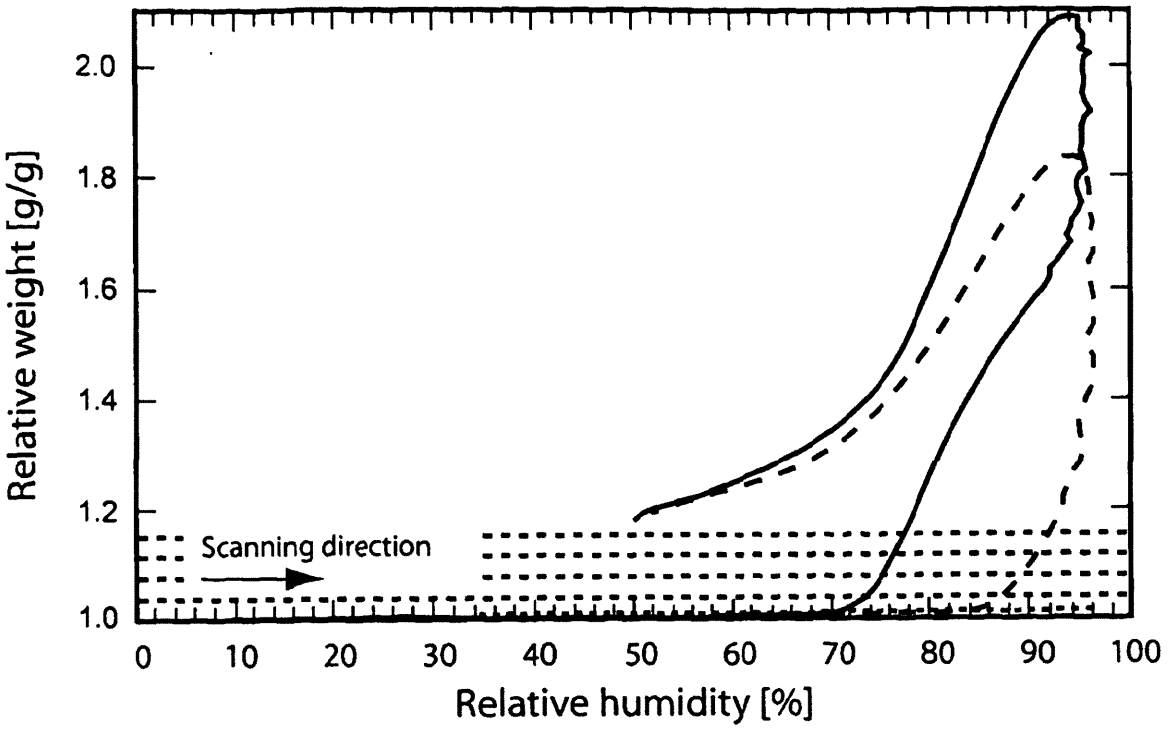


Figure 15b – expanded view:

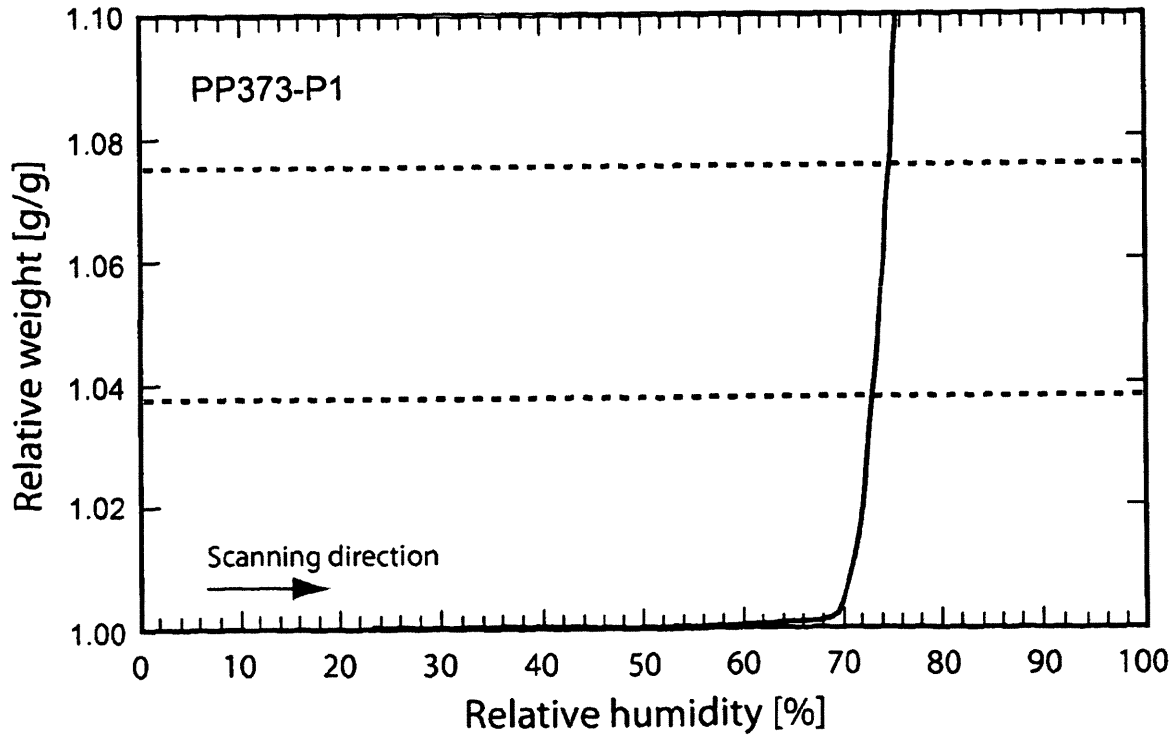


Figure 15c:

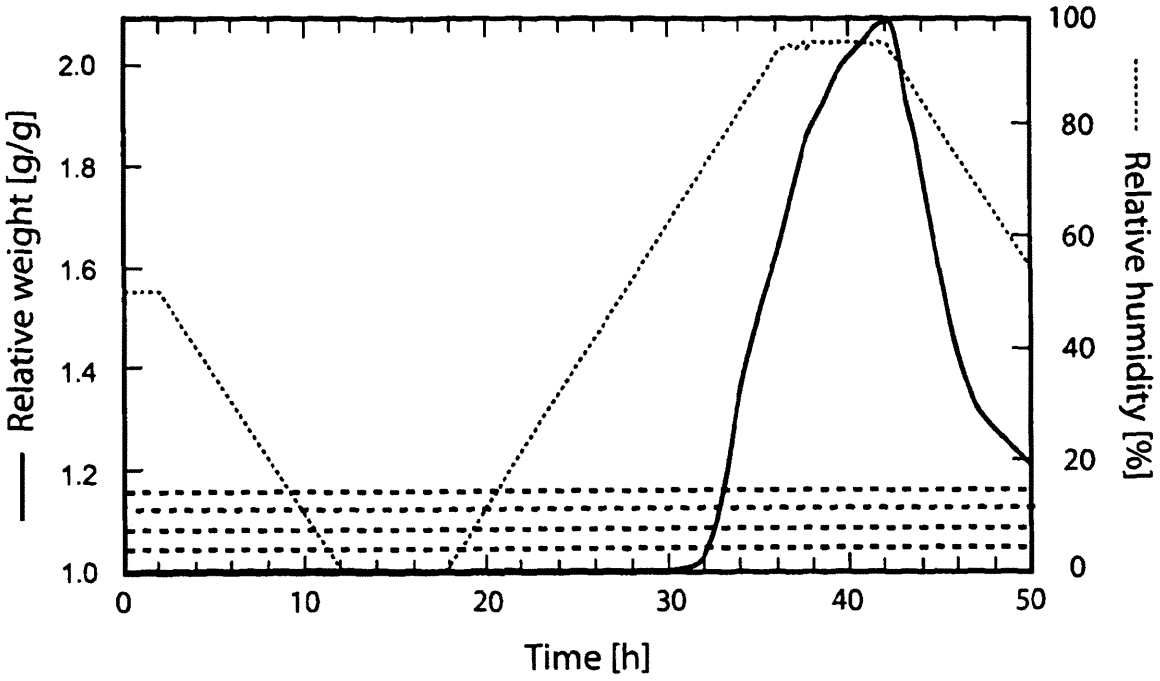

Relative weight [g/g]
2.0
1.8
1.6
1.4
1.2
1.0
0
10
20
30
40
50
Time [h]
100
80
60
40
20
0
Relative humidity [%]

Figure 16:

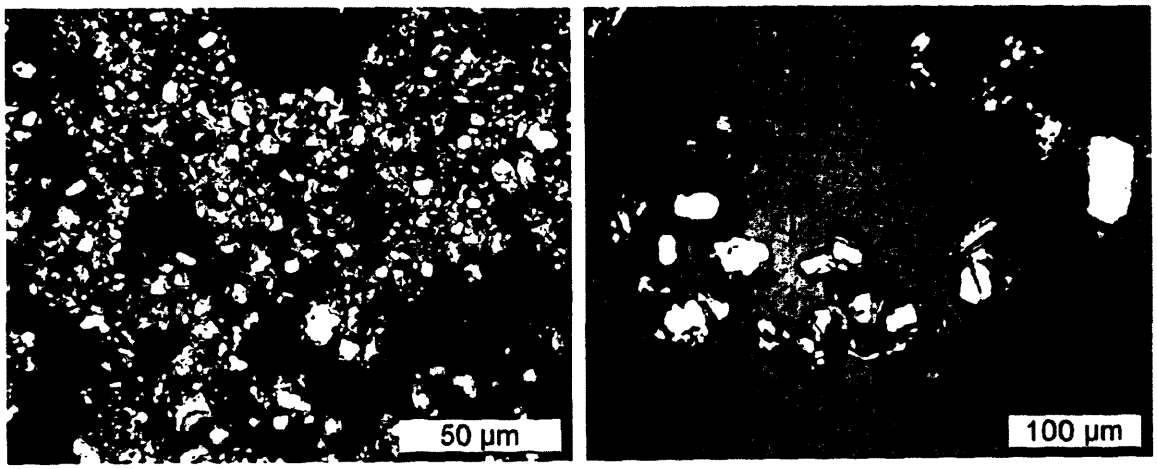
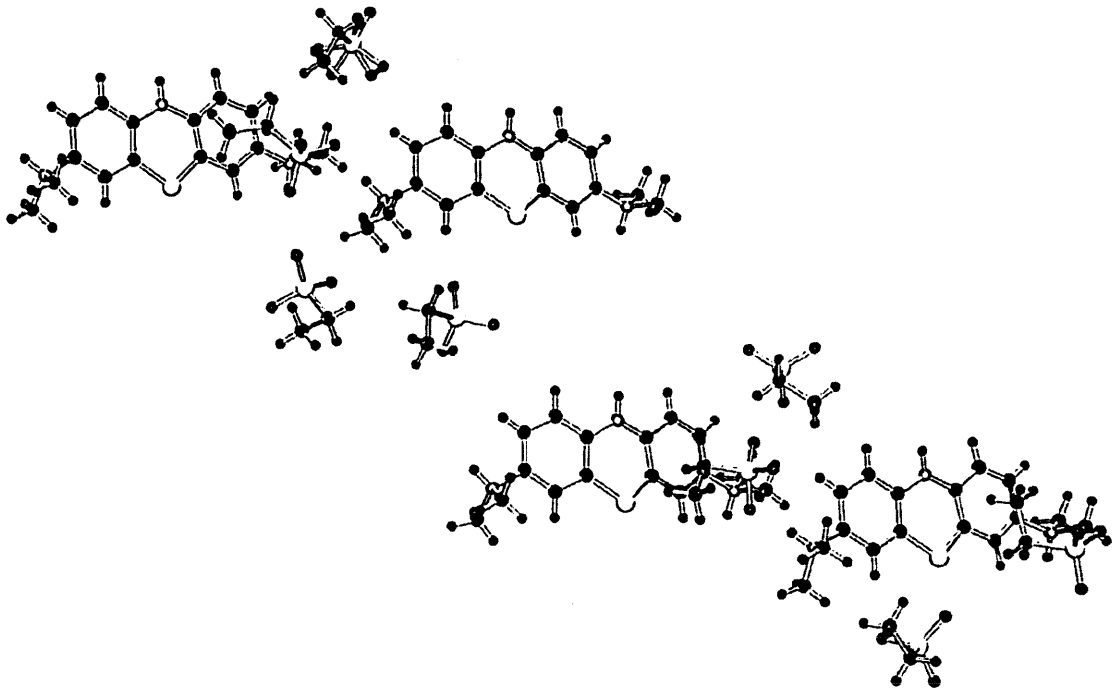
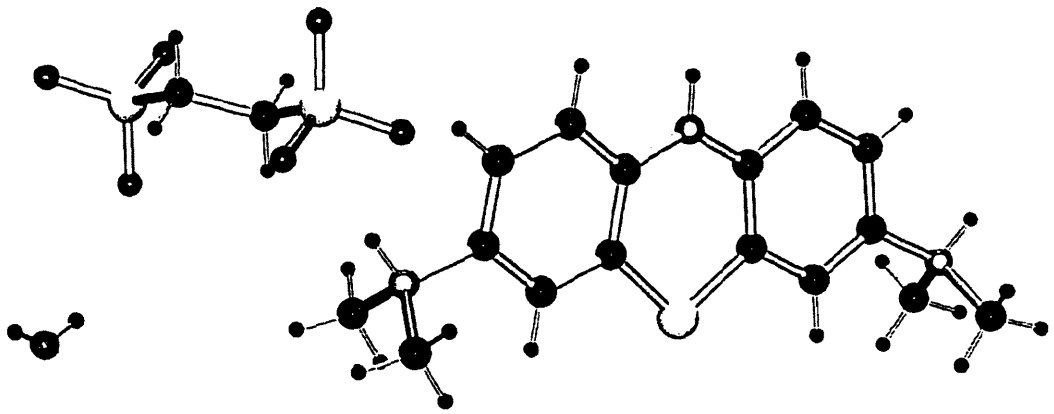


Figure 17A:



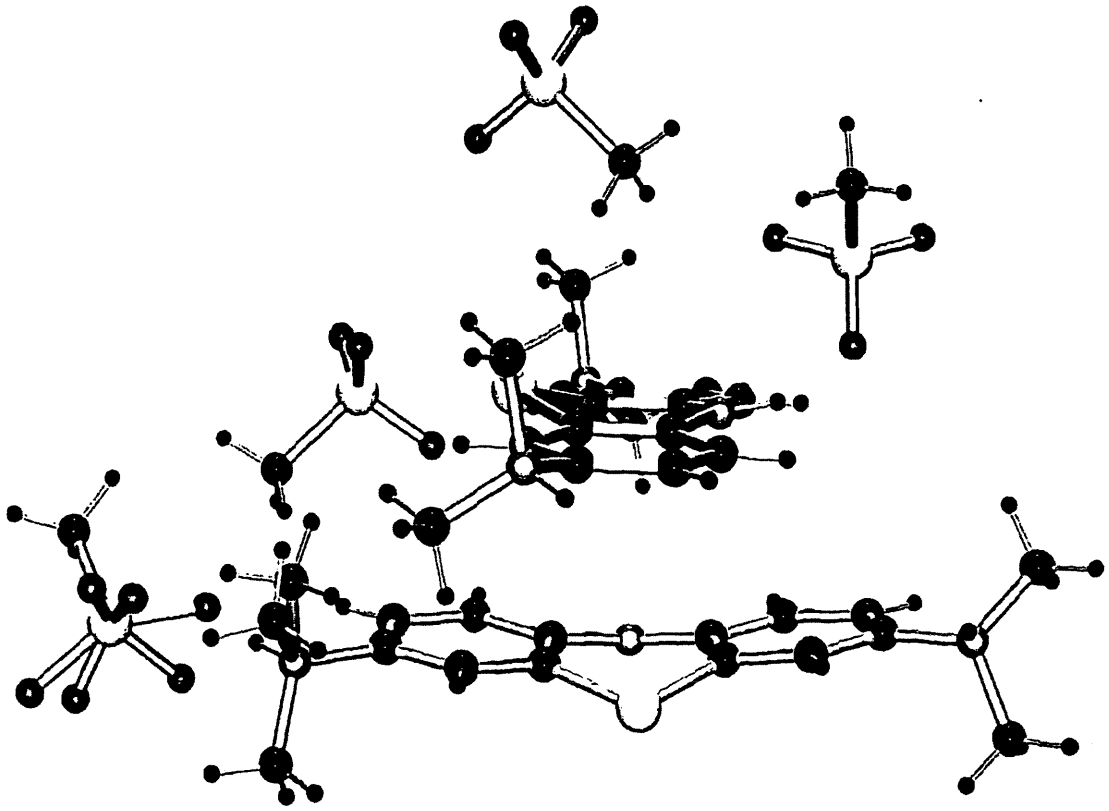
X-ray structure (unit cell) of LMTEs

Figure 17B:



X-ray structure (unit cell) of LMT.EDSA

Figure 17C:



X-ray structure (unit cell) of LMTM

Figure 18:

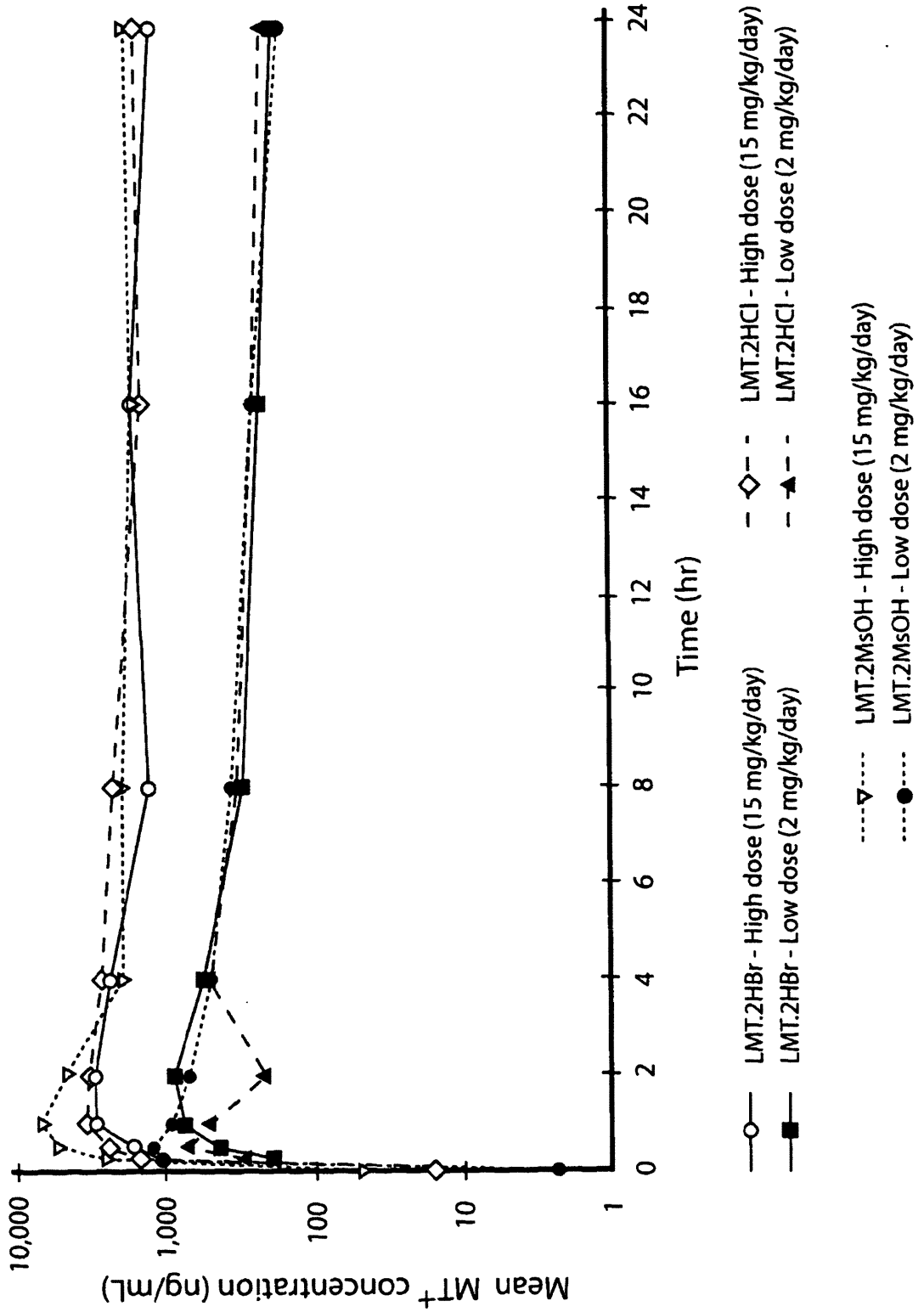

Mean MT+ concentration (ng/mL)
10,000
1,000
100
10
1
0
2
4
6
8
10
12
14
16
18
20
22
24
Time (hr)
LMT.2HBr - High dose (15 mg/kg/day)
LMT.2HBr - Low dose (2 mg/kg/day)
LMT.2HCl - High dose (15 mg/kg/day)
LMT.2HCl - Low dose (2 mg/kg/day)
LMT.2MsOH - High dose (15 mg/kg/day)
LMT.2MsOH - Low dose (2 mg/kg/day)

Figure 19:

