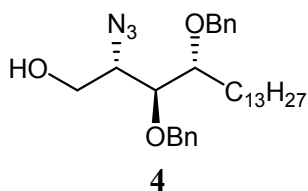
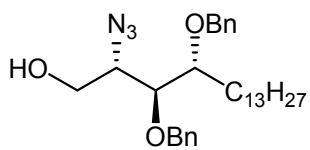
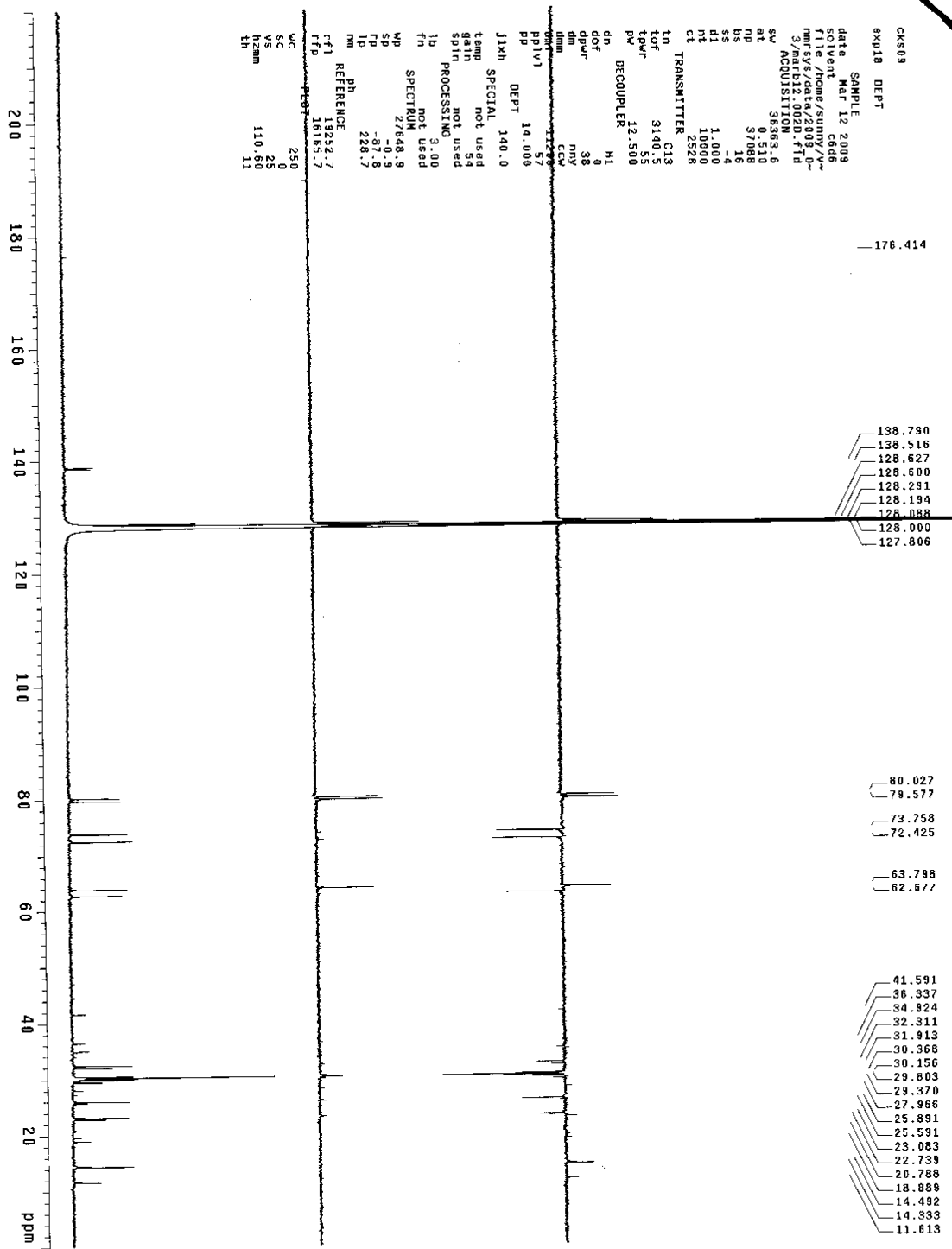
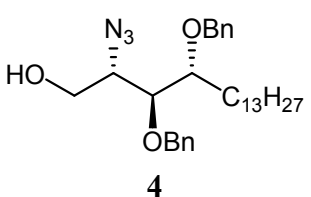
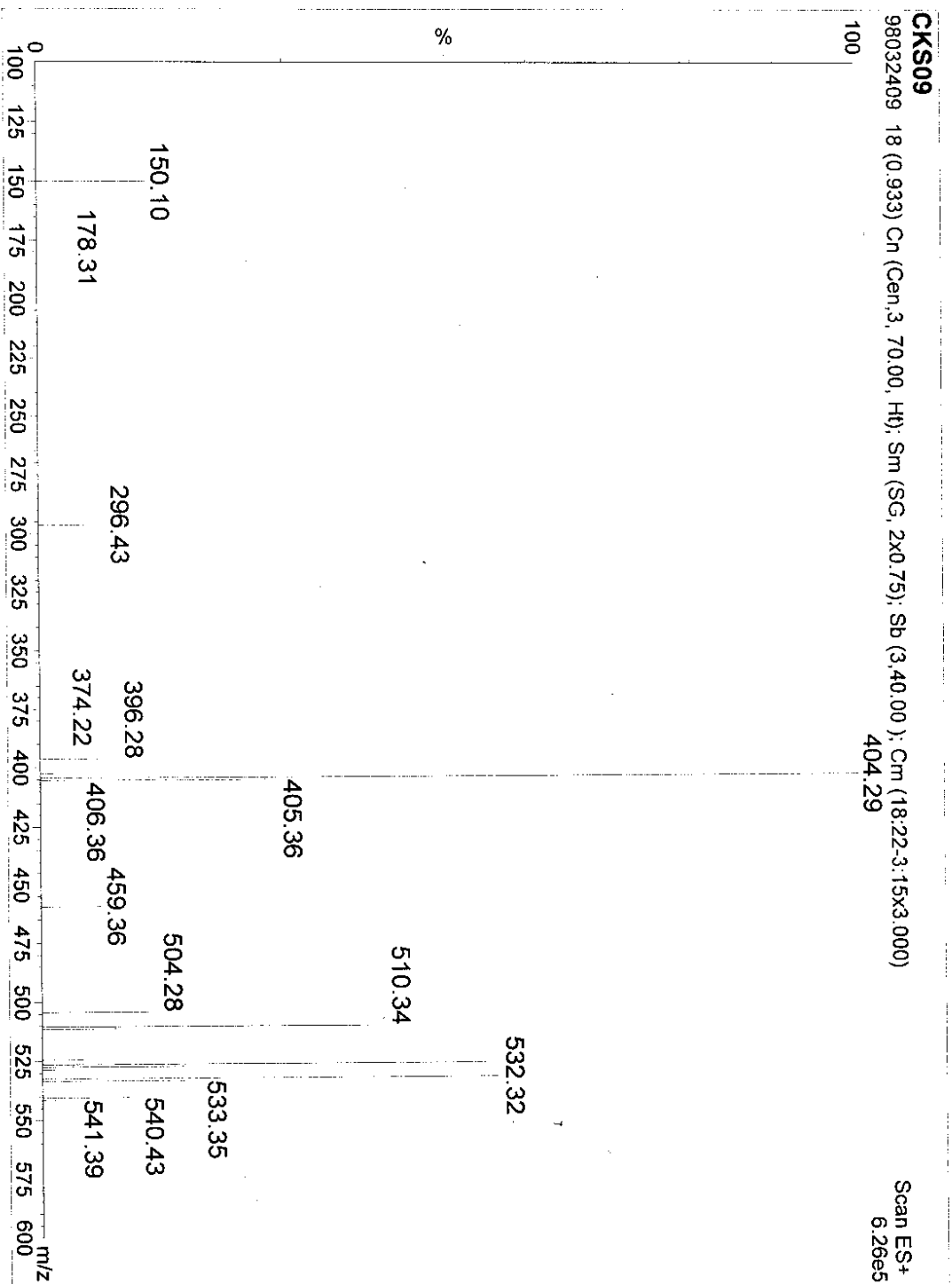


¹H- and ¹³C-NMR, ESI-MS spectra and HPLC chromatogram







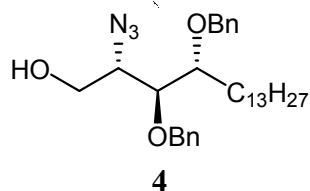
國立交通大學應用化學系

元素分析儀 Heraeus CHN-O Rapid 服務報告書

使用者姓名：張凱翔		中心編號：980357	
服務單位：清大原科 俞鐘山實驗室 樣品名稱或代號：CKS09			
收件日期： 98 年 3 月 24 日 完成日期： 98 年 3 月 26 日			
分析結果：			
實驗值：	N%	C%	H%
1.	8.10	72.85	8.49
2.	8.21	72.74	9.09
3.			
4.			
推測值：	8.24	73.05	9.29
本日所使用之 Standard：A			
(A)Acetanilide	(B)Atropin	(C)N-Anilin	
	N%	C%	H%
理論值：	10.36	71.09	6.71
測出值：	10.42	70.86	6.55
備註：			
費用核算：NCH：800 元			
報告日期： 98 年 3 月 27 日			

儀器負責人簽章：謝有容

技術員簽章：技士李蘊明



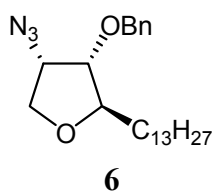
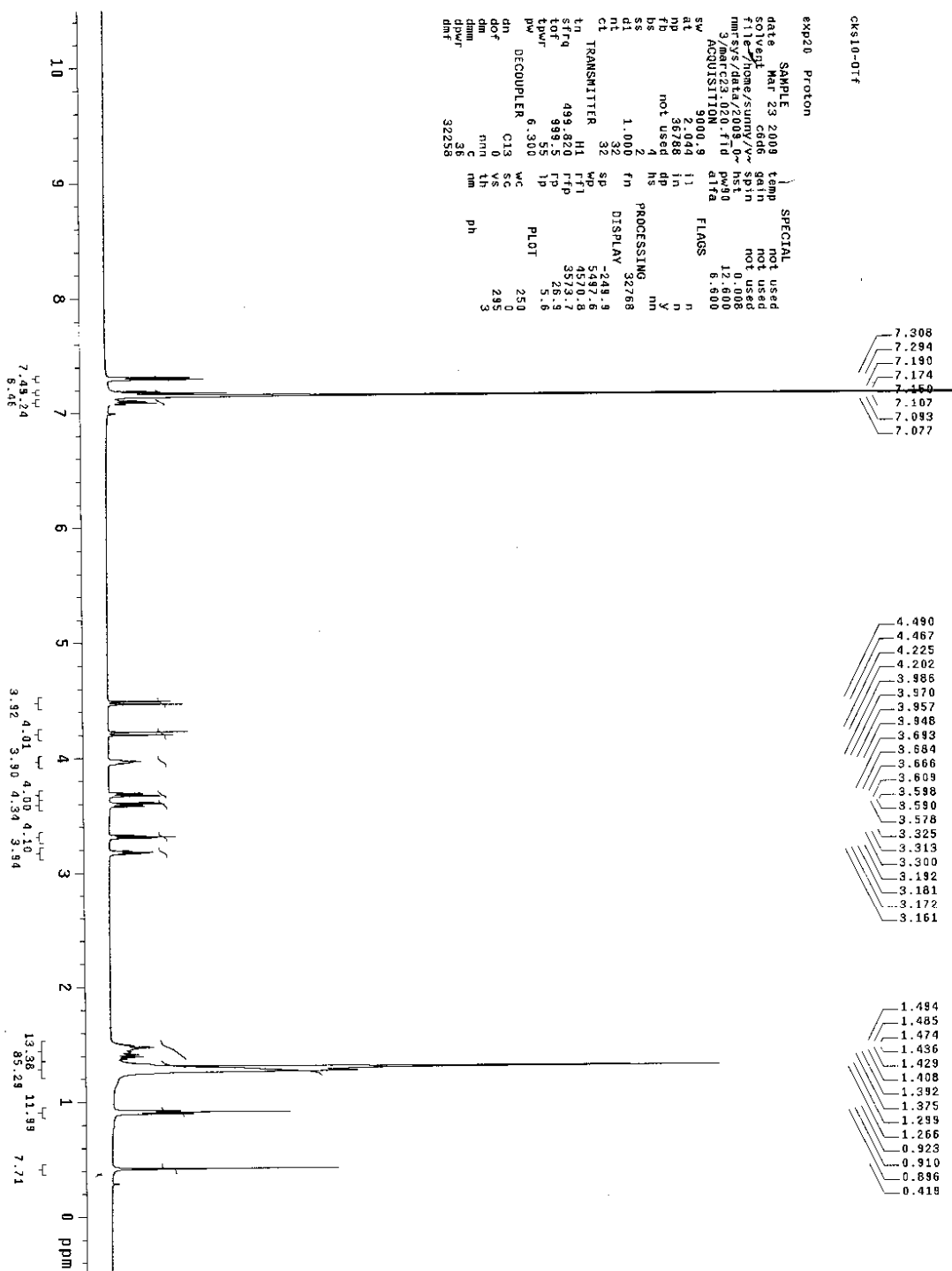
7.308
7.294
7.190
7.174
7.150
7.107
7.093
7.077

4.490
4.467
4.225
4.202
3.986
3.970
3.957
3.948
3.693
3.684
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3.325
3.313
3.300
3.192
3.181
3.172
3.161

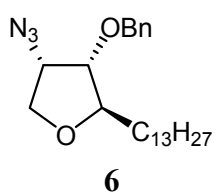
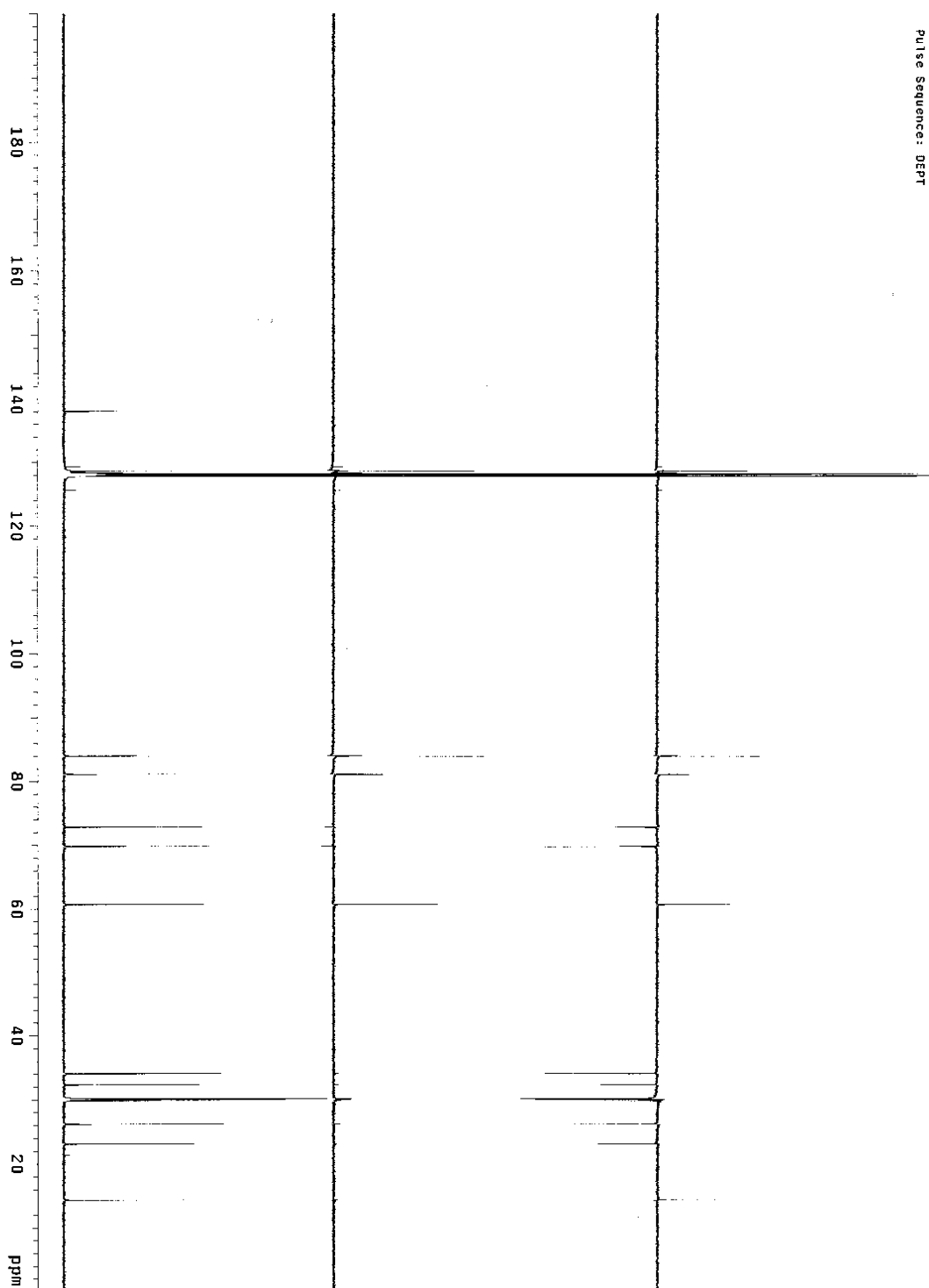
1.494
1.485
1.474
1.436
1.429
1.408
1.392
1.375
1.299
1.266
0.923
0.910
0.896
0.418

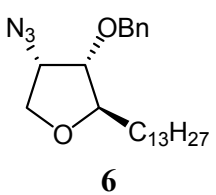
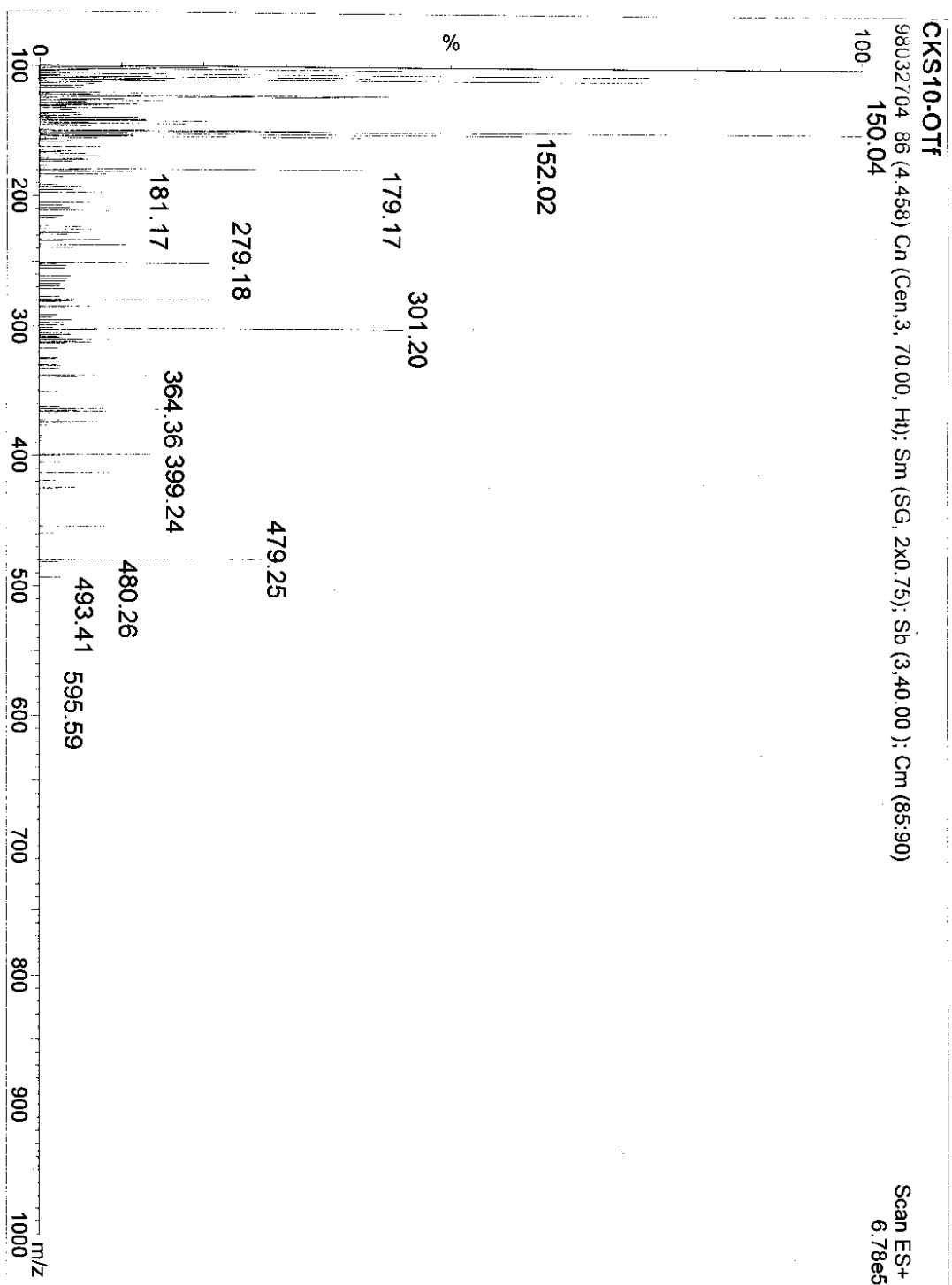
SAMPLE 1 SPECIAL

date	Mar 23 2009	temp	not used	not used
solvent	C6H6	gain	not used	not used
file	/home/sunny/v- mmsys/data/2008-06-hst	spin	0.008	not used
3dmc23.0	fid	pw	6.500	6.500
3dmc23.1	fid	attd		
sw	ACQUS1170	900.9		FLAGS
at	2.044	1	n	
np	36788	dp	n	
fb	not used	dp	n	
bs	2	n	n	
dt	7	n	n	
nt	1.000	fn		PROCESSING
cl	32	sp		32768
TRANSMITTER	32	sp		DISPLAY
h	559.6	wf1		-243.9
h1	499.820	rfp		352.7
h2	489.5	rfp		28.5
tpwr	6.300	55	1p	5.6
pw	6.300			
DECOUPLER	C13	wc		PLOT
dn	0	vc		250
dm	nm	vc		250
dmm	nm	nm		3
d1	nm	nm		
d2	nm	nm		
d3	nm	nm		
d4	nm	nm		
d5	nm	nm		
d6	nm	nm		
d7	nm	nm		
d8	nm	nm		
d9	nm	nm		
d10	nm	nm		
d11	nm	nm		
d12	nm	nm		
d13	nm	nm		
d14	nm	nm		
d15	nm	nm		
d16	nm	nm		
d17	nm	nm		
d18	nm	nm		
d19	nm	nm		
d20	nm	nm		
d21	nm	nm		
d22	nm	nm		
d23	nm	nm		
d24	nm	nm		
d25	nm	nm		
d26	nm	nm		
d27	nm	nm		
d28	nm	nm		
d29	nm	nm		
d30	nm	nm		
d31	nm	nm		
d32	nm	nm		
d33	nm	nm		
d34	nm	nm		
d35	nm	nm		
d36	nm	nm		
d37	nm	nm		
d38	nm	nm		
d39	nm	nm		
d40	nm	nm		
d41	nm	nm		
d42	nm	nm		
d43	nm	nm		
d44	nm	nm		
d45	nm	nm		
d46	nm	nm		
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d68	nm	nm		
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d74	nm	nm		
d75	nm	nm		
d76	nm	nm		
d77	nm	nm		
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d80	nm	nm		
d81	nm	nm		
d82	nm	nm		
d83	nm	nm		
d84	nm	nm		
d85	nm	nm		
d86	nm	nm		
d87	nm	nm		
d88	nm	nm		
d89	nm	nm		
d90	nm	nm		
d91	nm	nm		
d92	nm	nm		
d93	nm	nm		
d94	nm	nm		
d95	nm	nm		
d96	nm	nm		
d9				



CS-10-OTF/d-benzene
300024apt
Pulse Sequence: DEPT





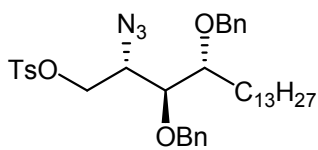
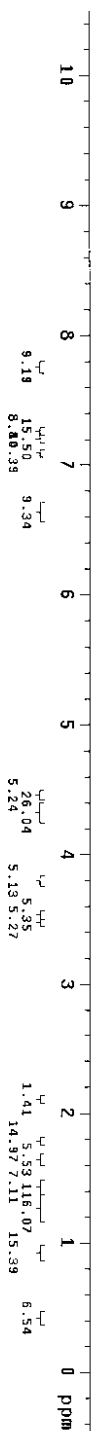
expl9 Proton

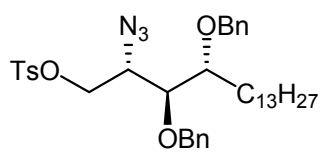
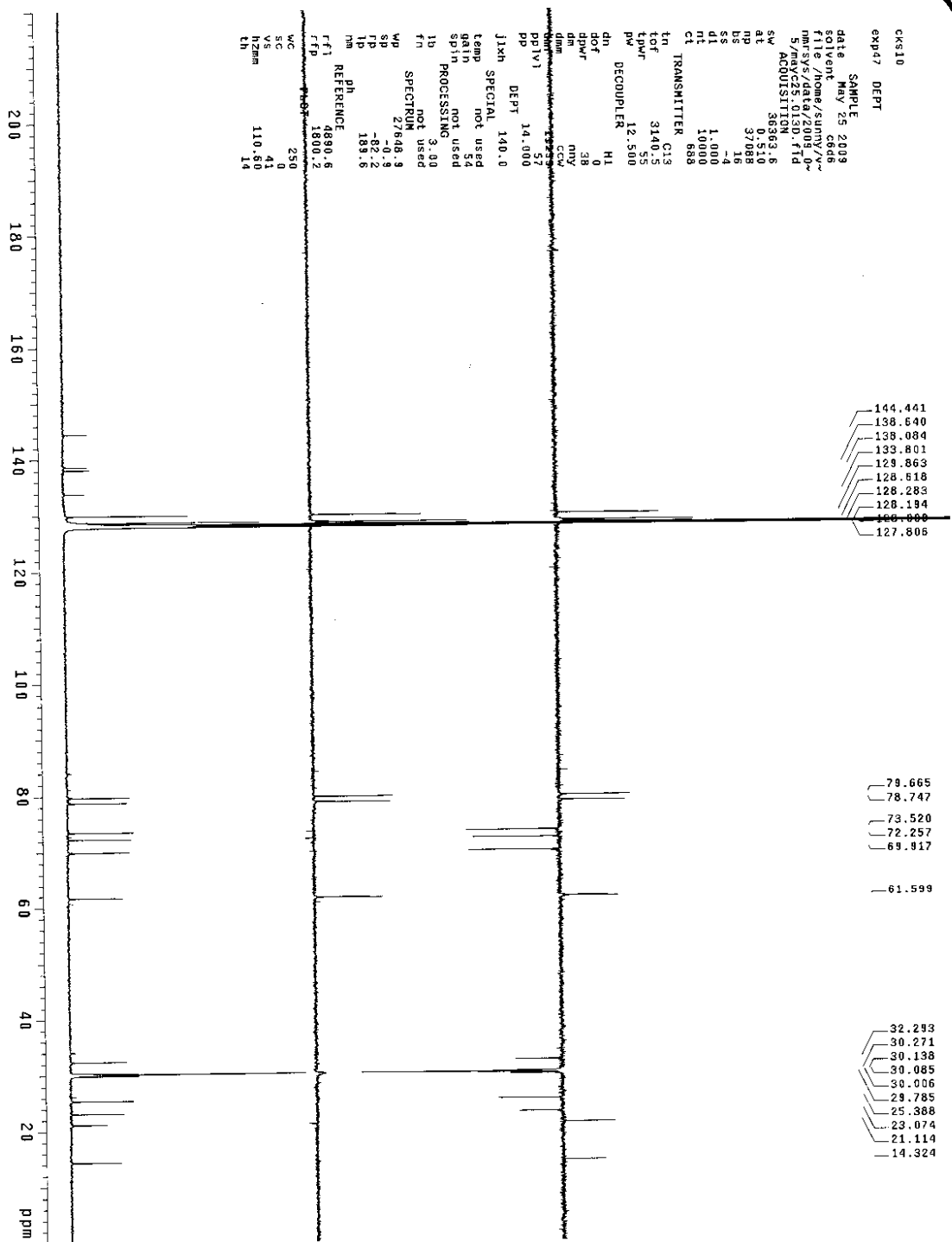
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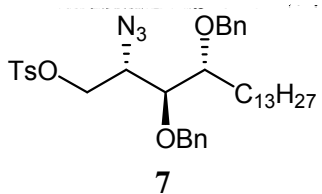
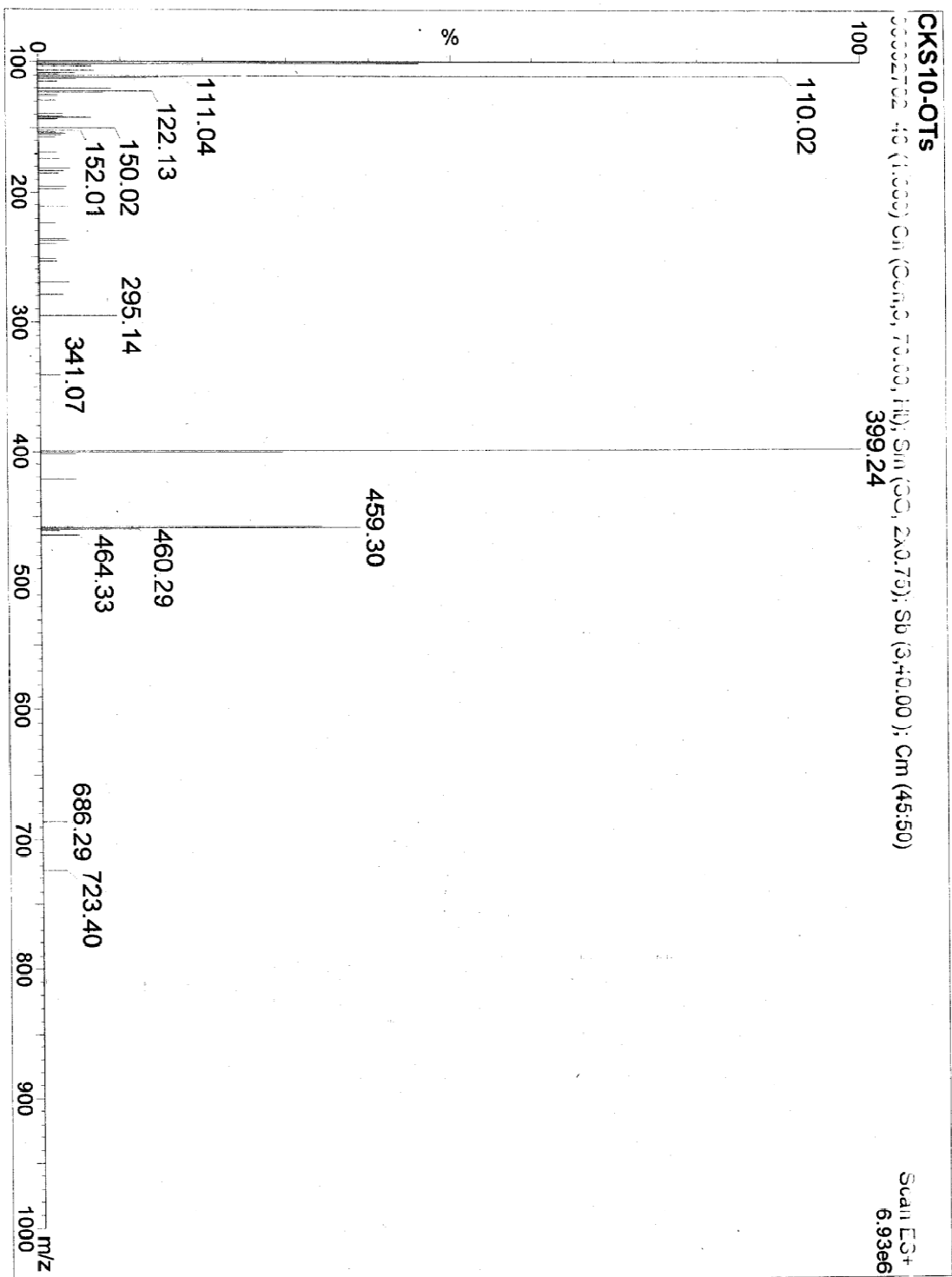
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7.212
7.198
7.183
7.168
7.160
7.150
7.130
7.124
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7.097
7.094
7.083
7.080
7.064
6.641
6.626

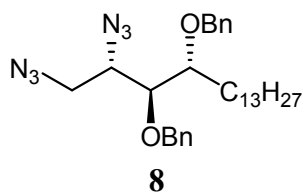
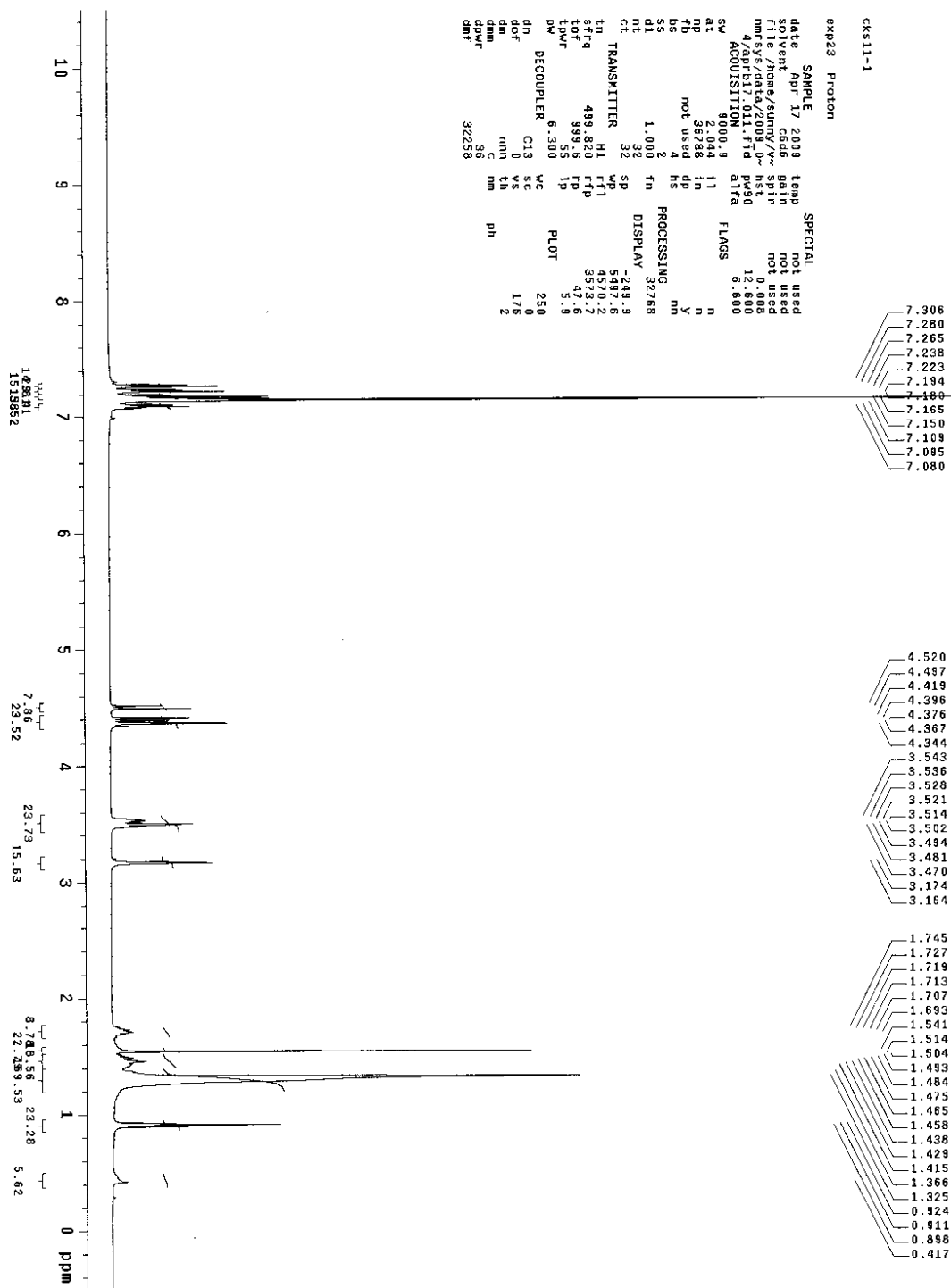
4.466
4.450
4.378
4.363
4.339
4.316
4.301
4.293
4.286
4.265
3.813
3.798
3.788
3.547
3.538
3.524
3.516
3.485
3.476
3.466

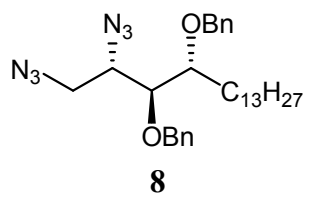
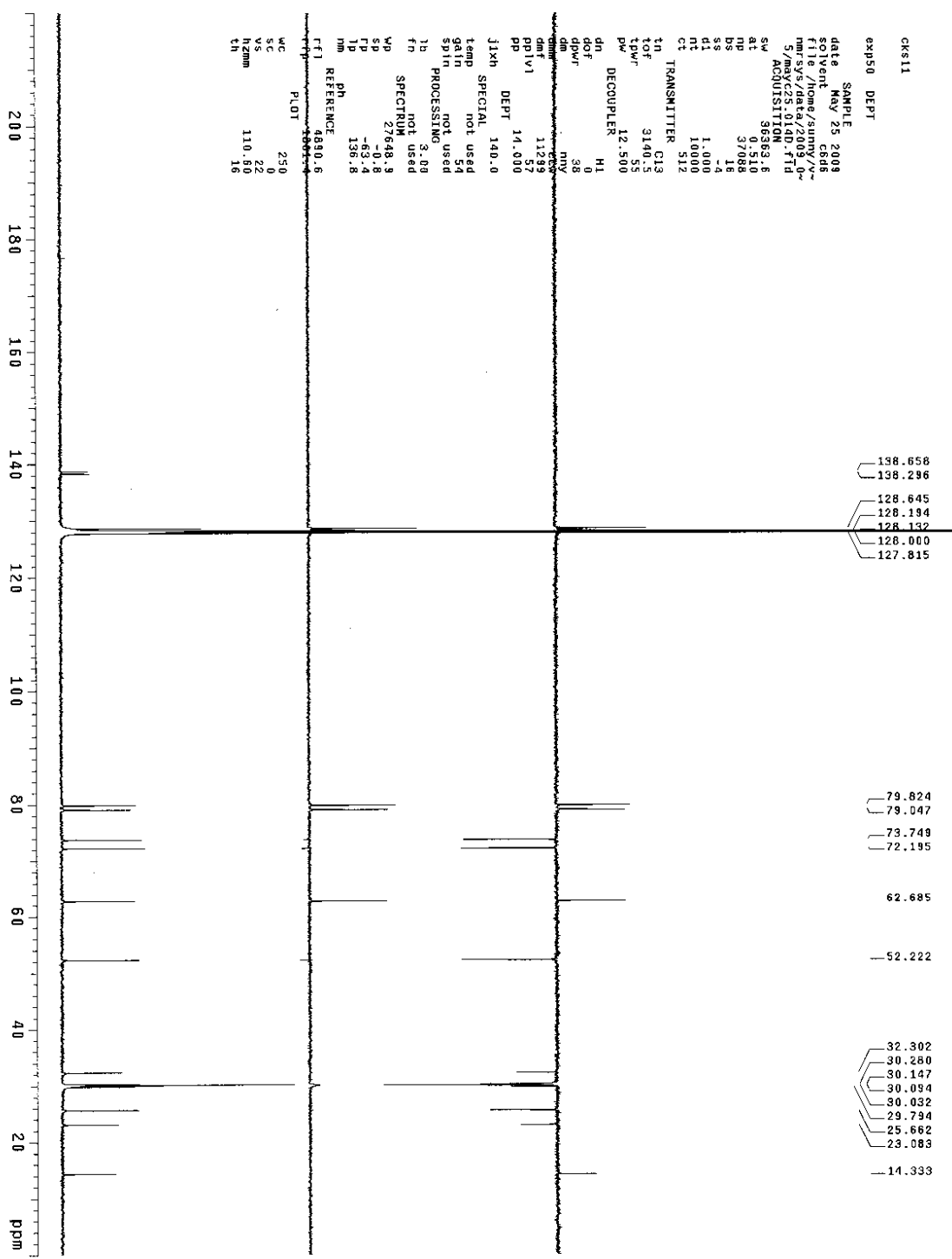
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1.654
1.644
1.639
1.634
1.439
1.430
1.419
1.410
1.399
1.356
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1.208
1.194
0.922
0.909
0.895
0.423

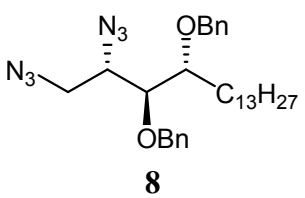
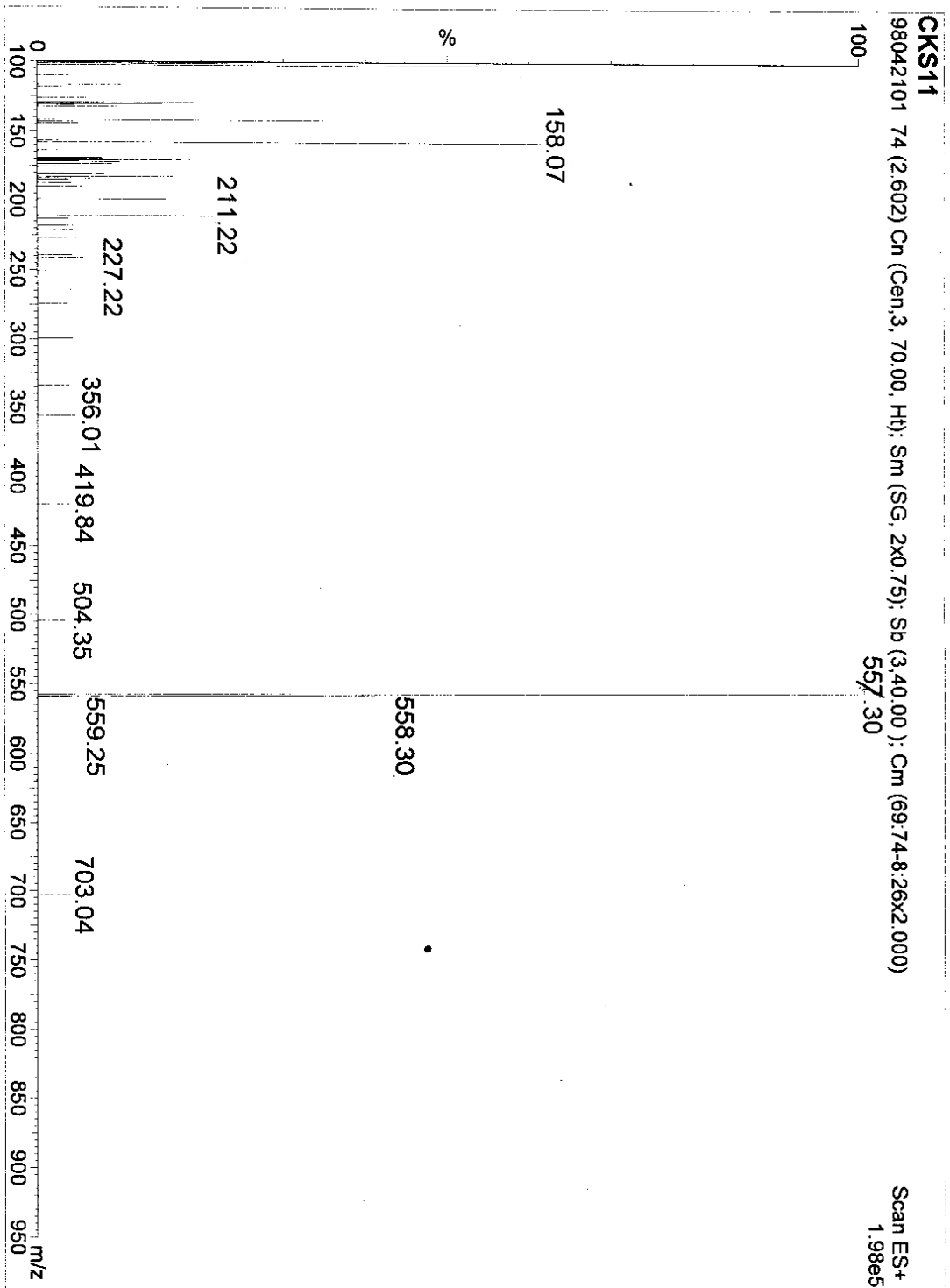












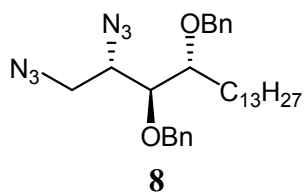
國立交通大學應用化學系

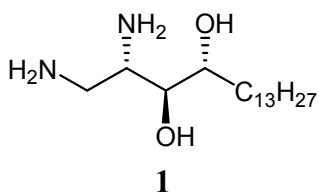
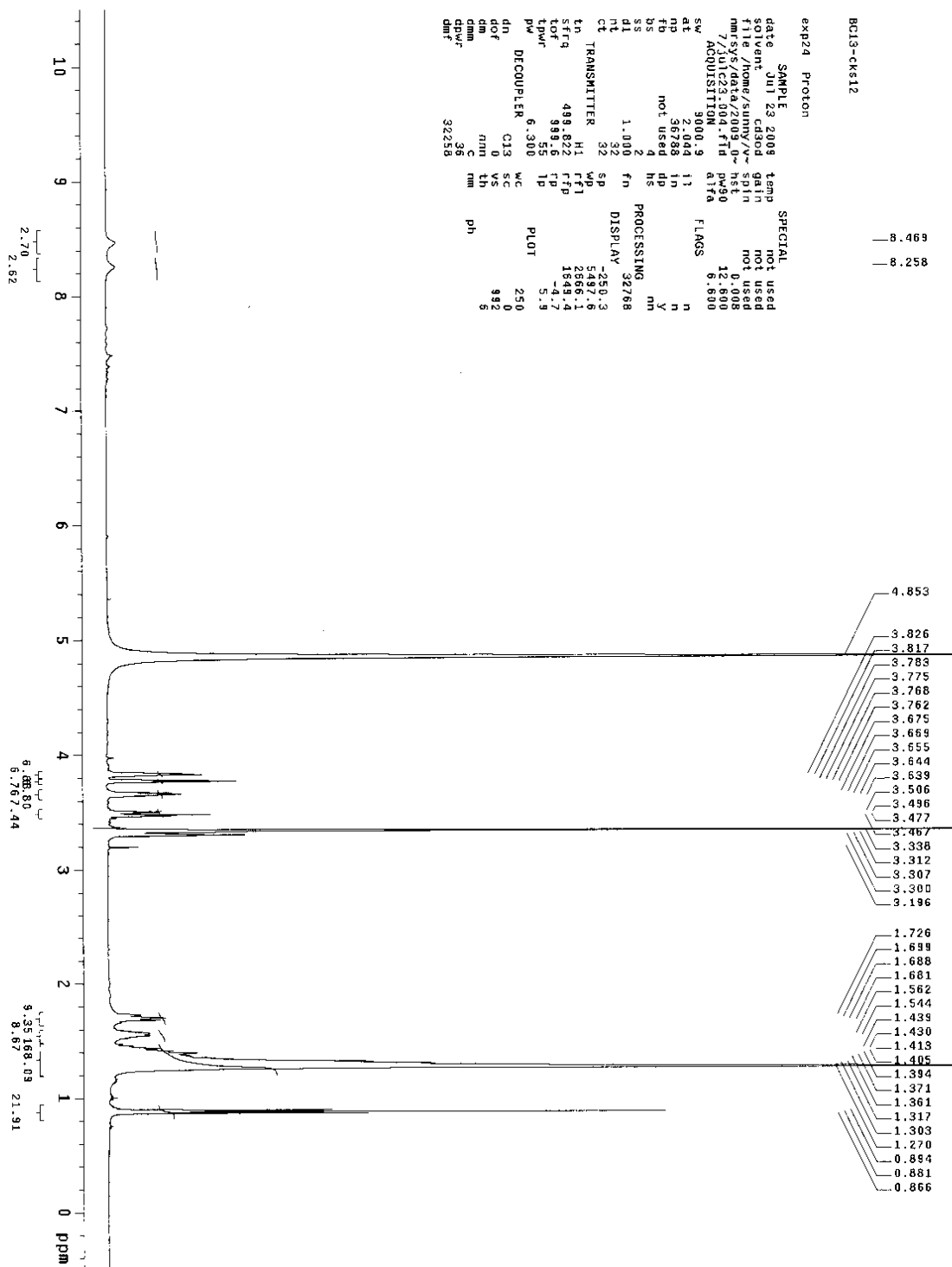
元素分析儀 Heraeus CHN-O Rapid 服務報告書

使用者姓名：張凱翔		中心編號：980530	
服務單位：清大原科 俞鐘山實驗室 樣品名稱或代號：CKS11			
收件日期： 98 年 5 月 14 日 完成日期： 98 年 5 月 18 日			
分析結果：			
實驗值：	N%	C%	H%
1.	16.03	69.96	8.24
2.	15.83	69.40	8.53
3.			
4.			
推測值：	15.72	69.63	8.67
本日所使用之 Standard：A			
(A)Acetanilide	(B)Atropin	(C)N-Anilin	
	N%	C%	H%
理論值：	10.36	71.09	6.71
測出值：	10.34	71.01	7.09
備註：			
費用核算：NCH：800 元			
報告日期： 98 年 5 月 19 日			

儀器負責人簽章：謝育仁

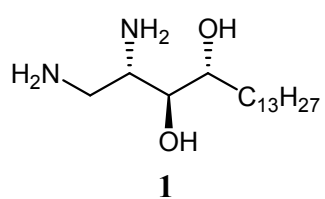
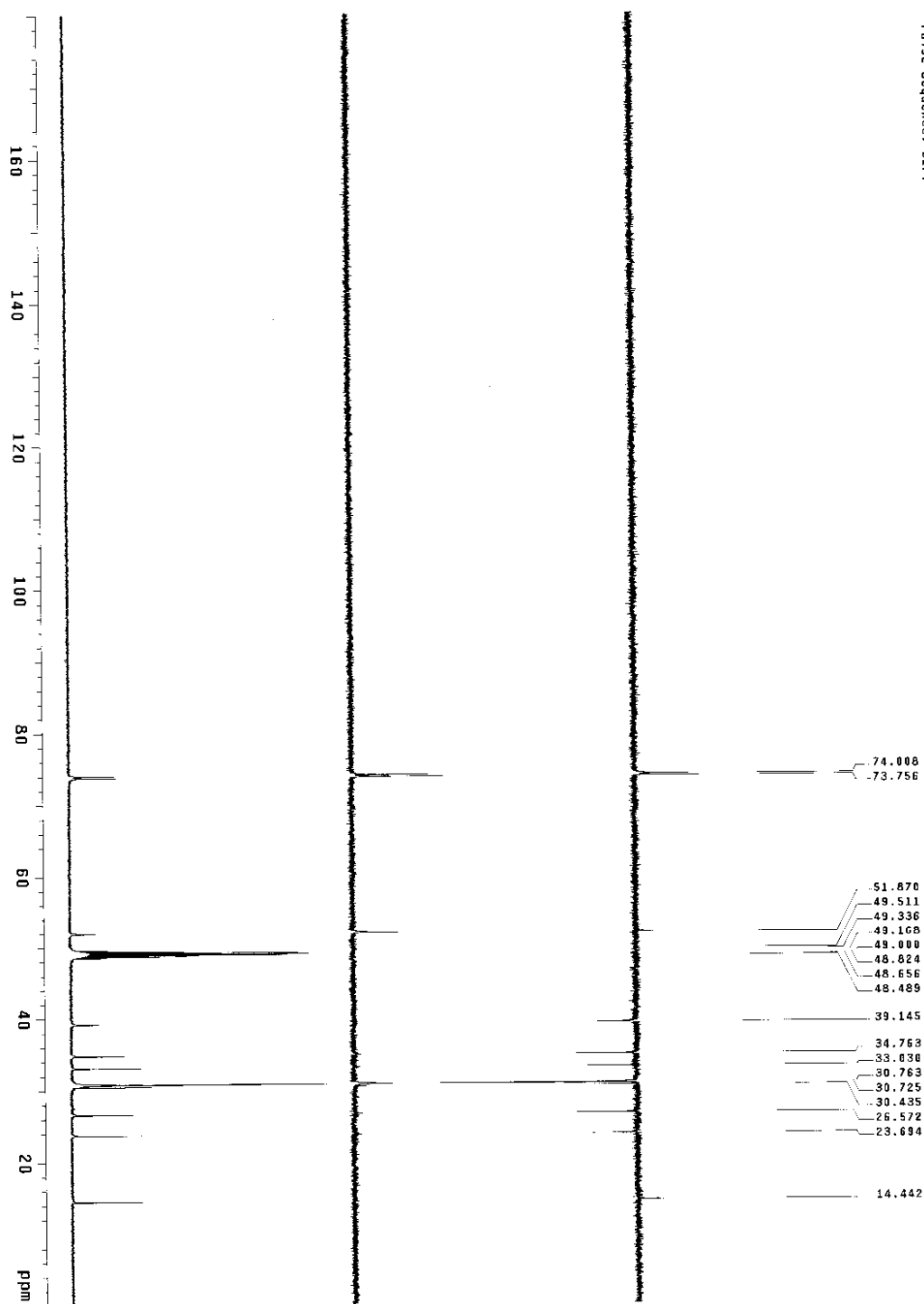
技術員簽章：技士李蘊明

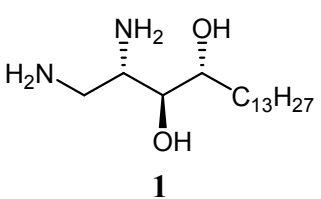
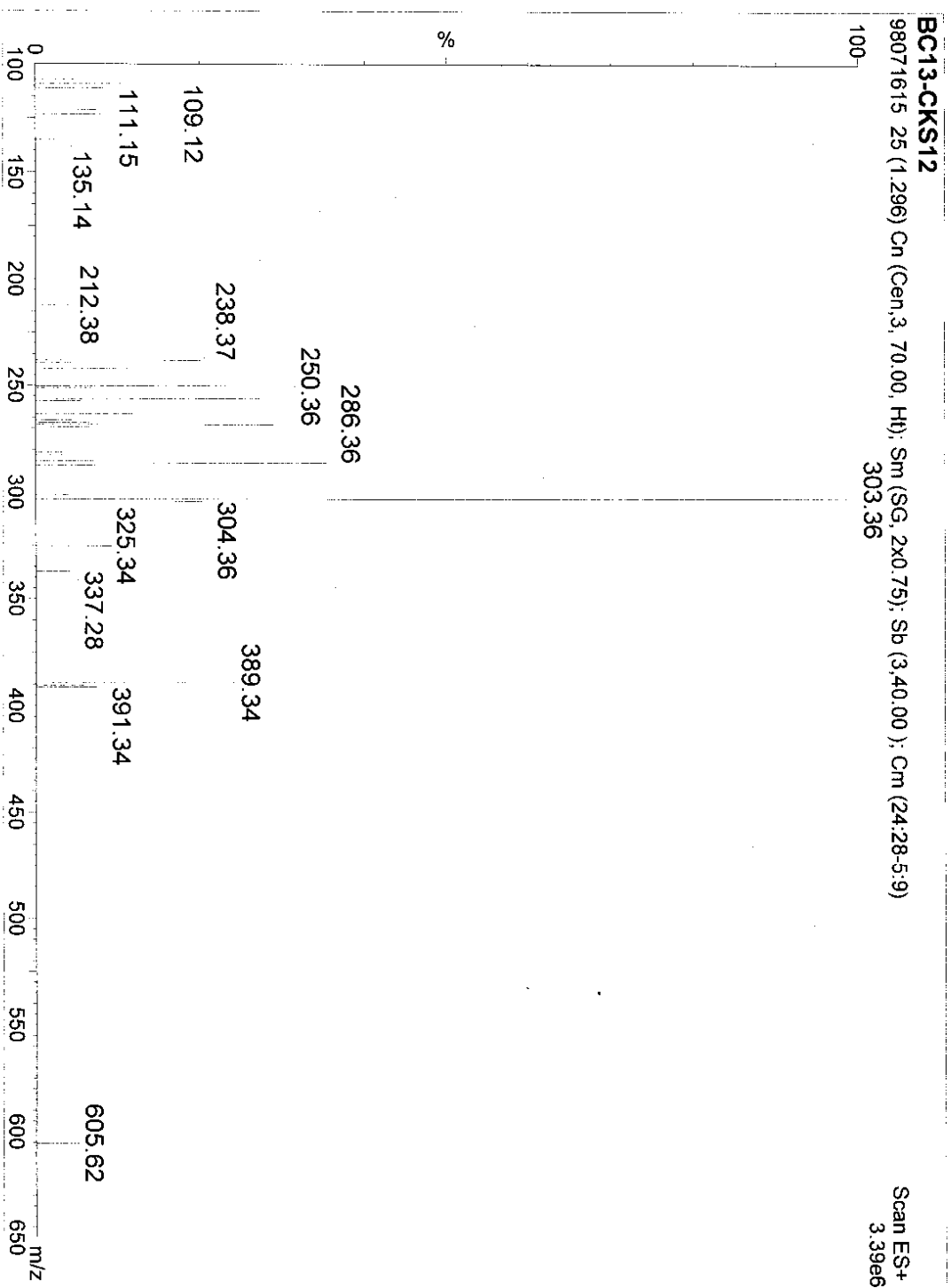


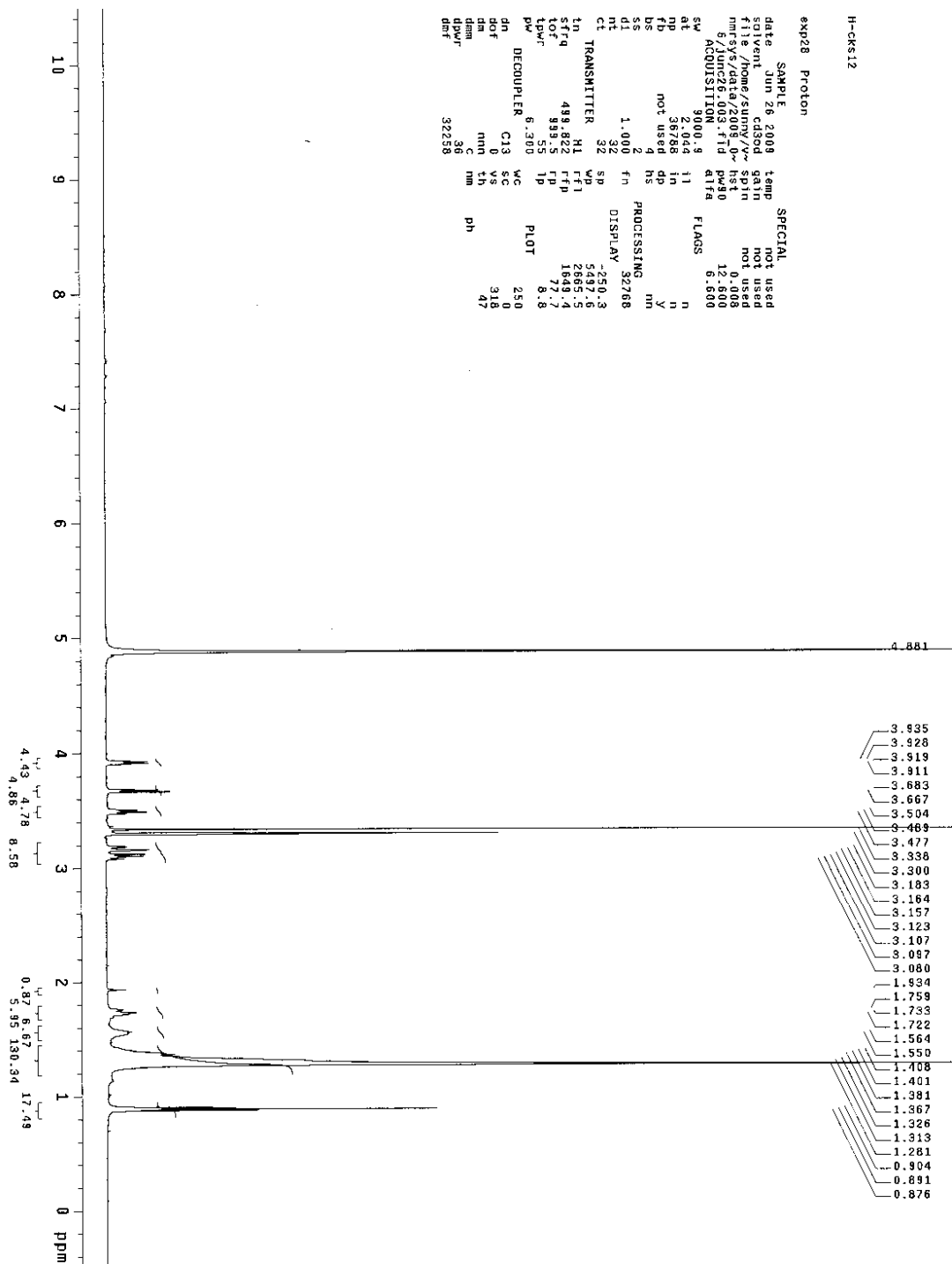
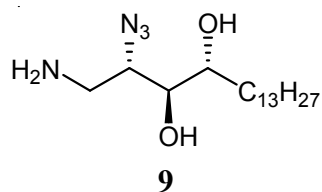


CRS12/MeOD
500000dept

Pulse Sequence: DEPT







exp28 Proton

SAMPLE SPECIAL

date Jun 26 2009 temp not used

solvent cd3od gain not used

file /home/sunny/v- spin not used

nmf 5/June26.003.fid pw40 12.600

ACQUISITION 3000.9 atfa 6.600

SW 2.044 11 n

at 36786 1n n

np not used ns y

bs 2 n

ss 1.000 fn PROCESSING nm

nt 32 DISPLAY 32768

ct 32 sp -250.3

in 11 wp 5497.6

to 488.82 ffl 2685.5

td 488.82 fd 1883.5

td 55 1p 72.7

pw 6.300 PLOT 6.8

DECOUPLER C13 wc 250

dn 0 sc 0

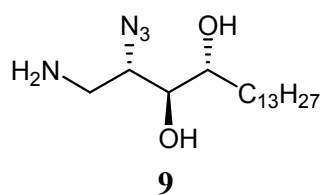
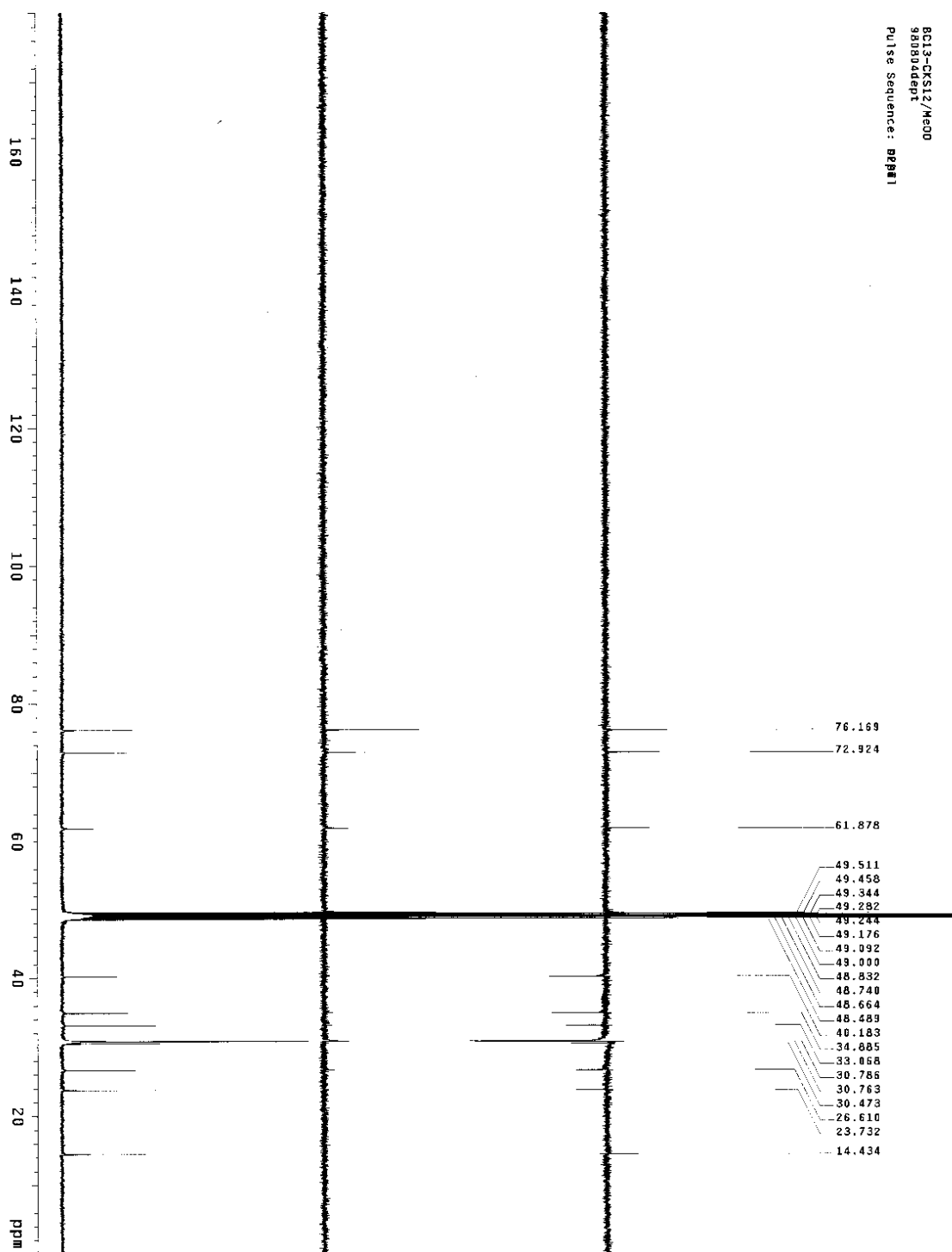
dnf 0 vs 318

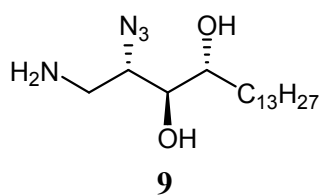
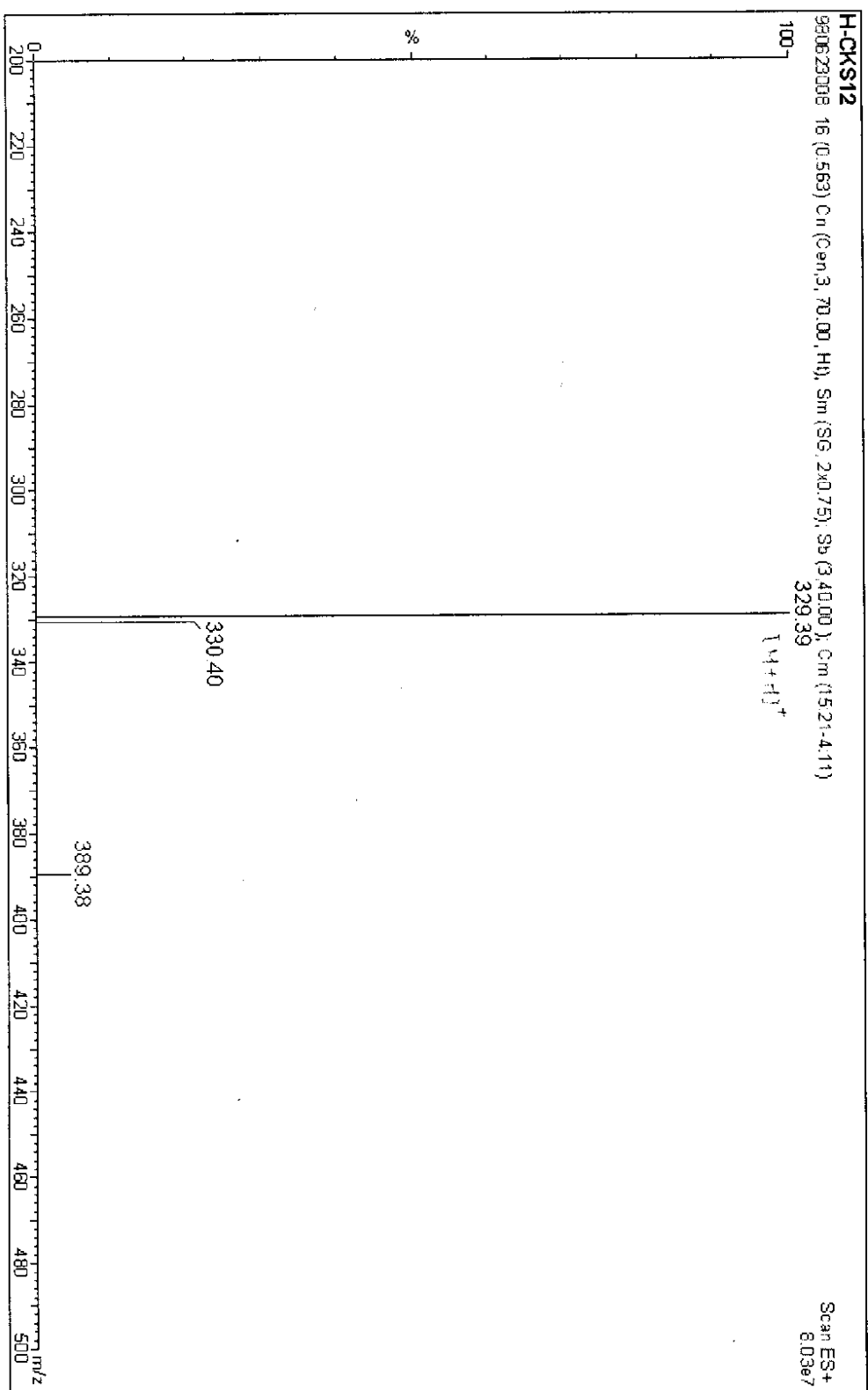
dmu 47

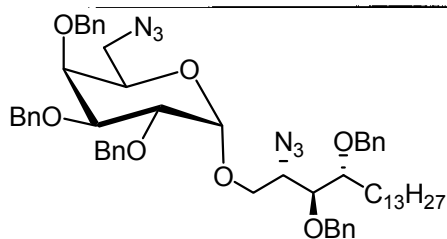
dmn 36 mm

dmf 32258 ph 47

BC1
980

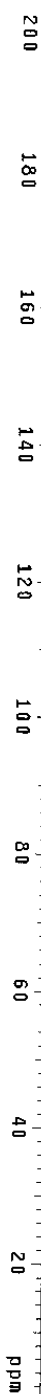


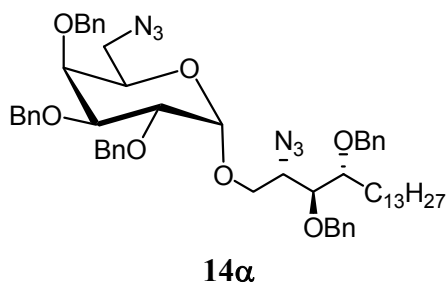
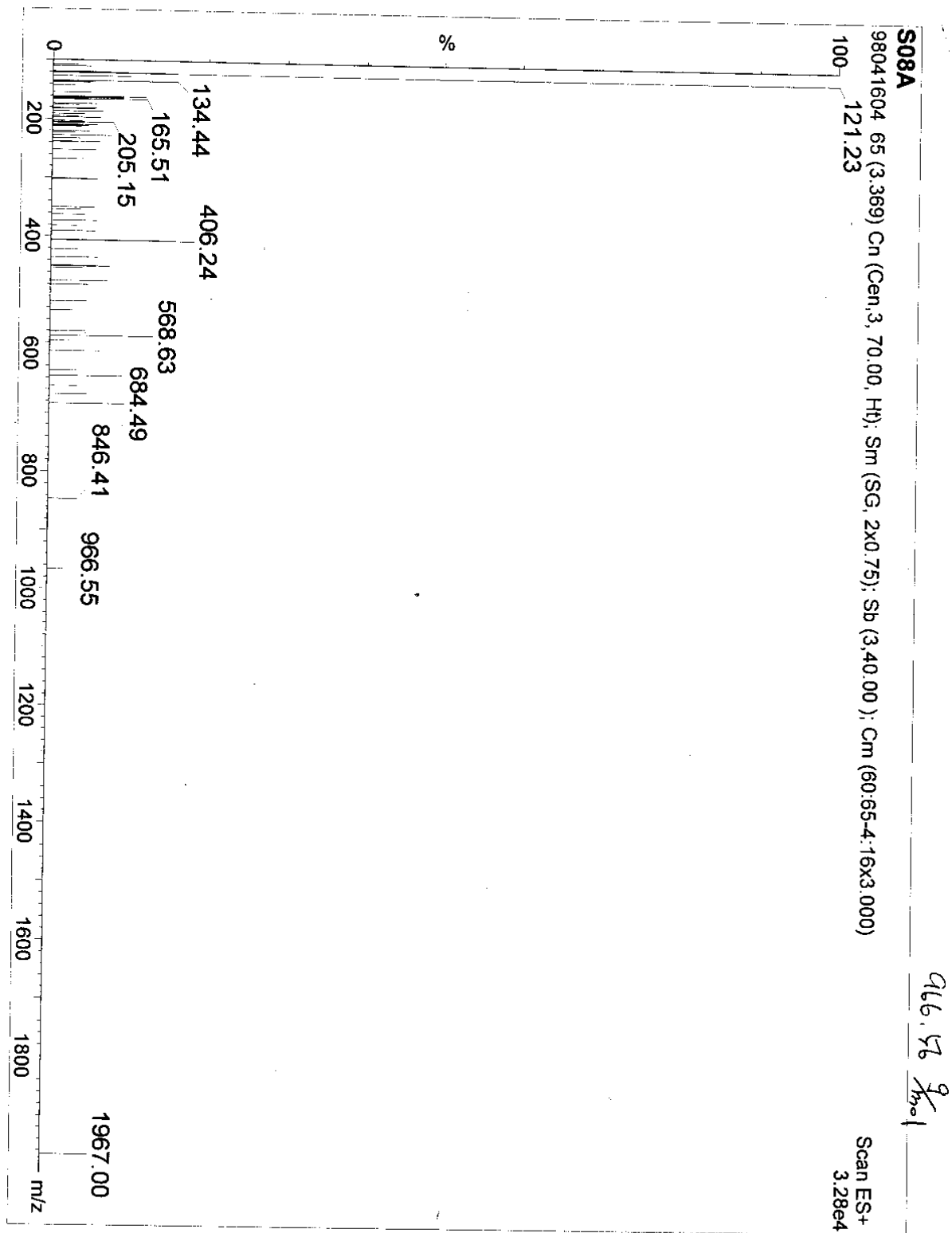




14α

CE
4891.7
1801.4
250
0
3297
110.60
12





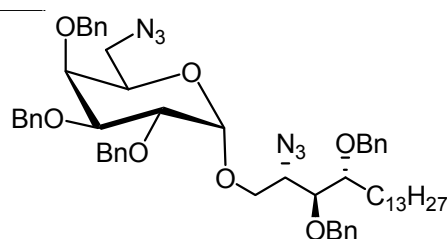
國立交通大學應用化學系

元素分析儀 Heraeus CHN-O Rapid 服務報告書

使用者姓名：廖國延		中心編號：980529	
服務單位：清大原科 俞鐘山實驗室 樣品名稱或代號：SO8A			
收件日期：98 年 5 月 14 日 完成日期：98 年 5 月 18 日			
分析結果：			
實驗值：	N%	C%	H%
1.	8.66	72.11	7.42
2.	8.46	71.79	7.27
3.			
4.			
推測值：	8.69	72.02	7.71
本日所使用之 Standard：A			
(A)Acetanilide	(B)Atropin	(C)N-Anilin	
	N%	C%	H%
理論值：	10.36	71.09	6.71
測出值：	10.34	71.01	7.09
備註：			
費用核算：NCH：800 元			
報告日期：98 年 5 月 19 日			

儀器負責人簽章：謝育宏

技術員簽章：技士李蘊明



14α

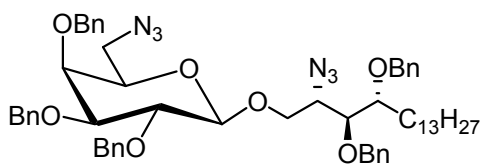
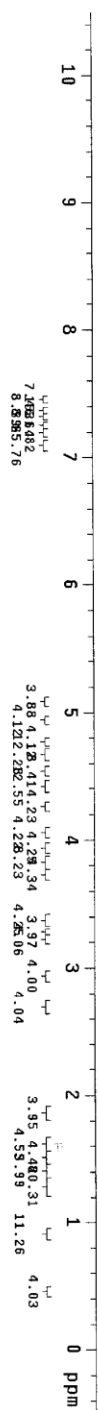
exp7 Proton

SAMPLE		SPECIAL	
date	May 13 2003	temp	not used
solvent	CH2Cl2/CHCl3	gain	not used
nmr	500/300/200	h1	not used
nmr	500/300/200	h2	not used
nmr	500/300/200	h3	not used
nmr	500/300/200	h4	not used
nmr	500/300/200	h5	not used
nmr	500/300/200	h6	not used
nmr	500/300/200	h7	not used
nmr	500/300/200	h8	not used
nmr	500/300/200	h9	not used
nmr	500/300/200	h10	not used
nmr	500/300/200	h11	not used
nmr	500/300/200	h12	not used
nmr	500/300/200	h13	not used
nmr	500/300/200	h14	not used
nmr	500/300/200	h15	not used
nmr	500/300/200	h16	not used
nmr	500/300/200	h17	not used
nmr	500/300/200	h18	not used
nmr	500/300/200	h19	not used
nmr	500/300/200	h20	not used
nmr	500/300/200	h21	not used
nmr	500/300/200	h22	not used
nmr	500/300/200	h23	not used
nmr	500/300/200	h24	not used
nmr	500/300/200	h25	not used
nmr	500/300/200	h26	not used
nmr	500/300/200	h27	not used
nmr	500/300/200	h28	not used
nmr	500/300/200	h29	not used
nmr	500/300/200	h30	not used
nmr	500/300/200	h31	not used
nmr	500/300/200	h32	not used
nmr	500/300/200	h33	not used
nmr	500/300/200	h34	not used
nmr	500/300/200	h35	not used
nmr	500/300/200	h36	not used
nmr	500/300/200	h37	not used
nmr	500/300/200	h38	not used
nmr	500/300/200	h39	not used
nmr	500/300/200	h40	not used
nmr	500/300/200	h41	not used
nmr	500/300/200	h42	not used
nmr	500/300/200	h43	not used
nmr	500/300/200	h44	not used
nmr	500/300/200	h45	not used
nmr	500/300/200	h46	not used
nmr	500/300/200	h47	not used
nmr	500/300/200	h48	not used
nmr	500/300/200	h49	not used
nmr	500/300/200	h50	not used
nmr	500/300/200	h51	not used
nmr	500/300/200	h52	not used
nmr	500/300/200	h53	not used
nmr	500/300/200	h54	not used
nmr	500/300/200	h55	not used
nmr	500/300/200	h56	not used
nmr	500/300/200	h57	not used
nmr	500/300/200	h58	not used
nmr	500/300/200	h59	not used
nmr	500/300/200	h60	not used
nmr	500/300/200	h61	not used
nmr	500/300/200	h62	not used
nmr	500/300/200	h63	not used
nmr	500/300/200	h64	not used
nmr	500/300/200	h65	not used
nmr	500/300/200	h66	not used
nmr	500/300/200	h67	not used
nmr	500/300/200	h68	not used
nmr	500/300/200	h69	not used
nmr	500/300/200	h70	not used
nmr	500/300/200	h71	not used
nmr	500/300/200	h72	not used
nmr	500/300/200	h73	not used
nmr	500/300/200	h74	not used
nmr	500/300/200	h75	not used
nmr	500/300/200	h76	not used
nmr	500/300/200	h77	not used
nmr	500/300/200	h78	not used
nmr	500/300/200	h79	not used
nmr	500/300/200	h80	not used
nmr	500/300/200	h81	not used

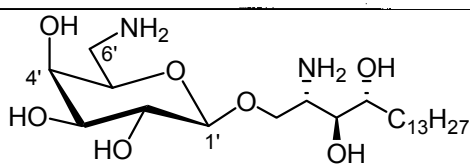
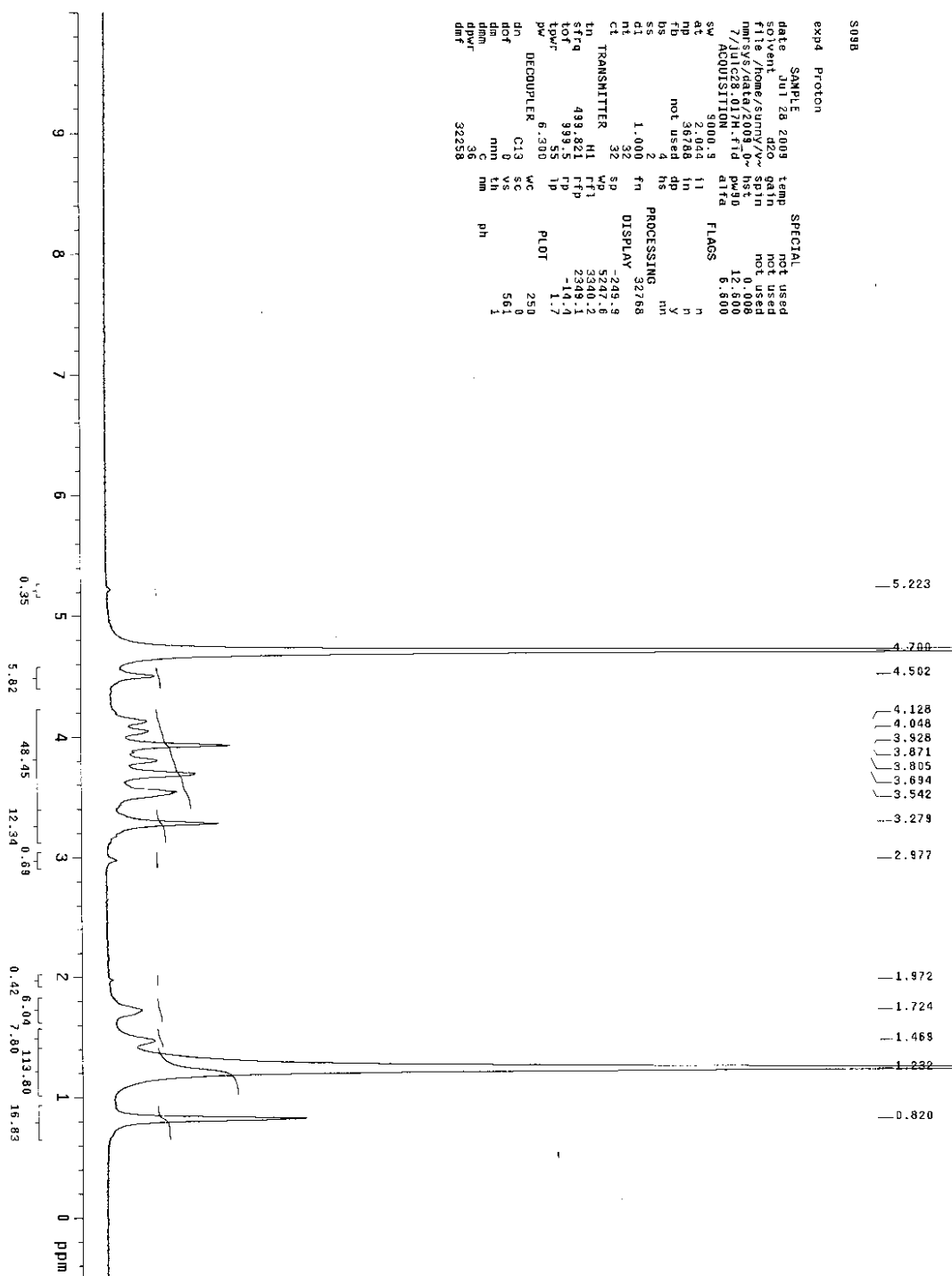
7.462
7.447
7.372
7.357
7.337
7.321
7.260
7.246
7.207
7.193
7.180
7.164
7.150
7.129
7.117
7.104
7.088
7.073
7.059

5.099
5.076
4.948
4.925
4.775
4.753
4.699
4.677
4.653
4.556
4.521
4.448
4.425
4.255
4.057
3.951
3.836
3.805
3.727
3.395
3.355
3.277
3.241
2.940
2.709
2.684

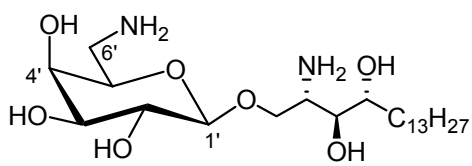
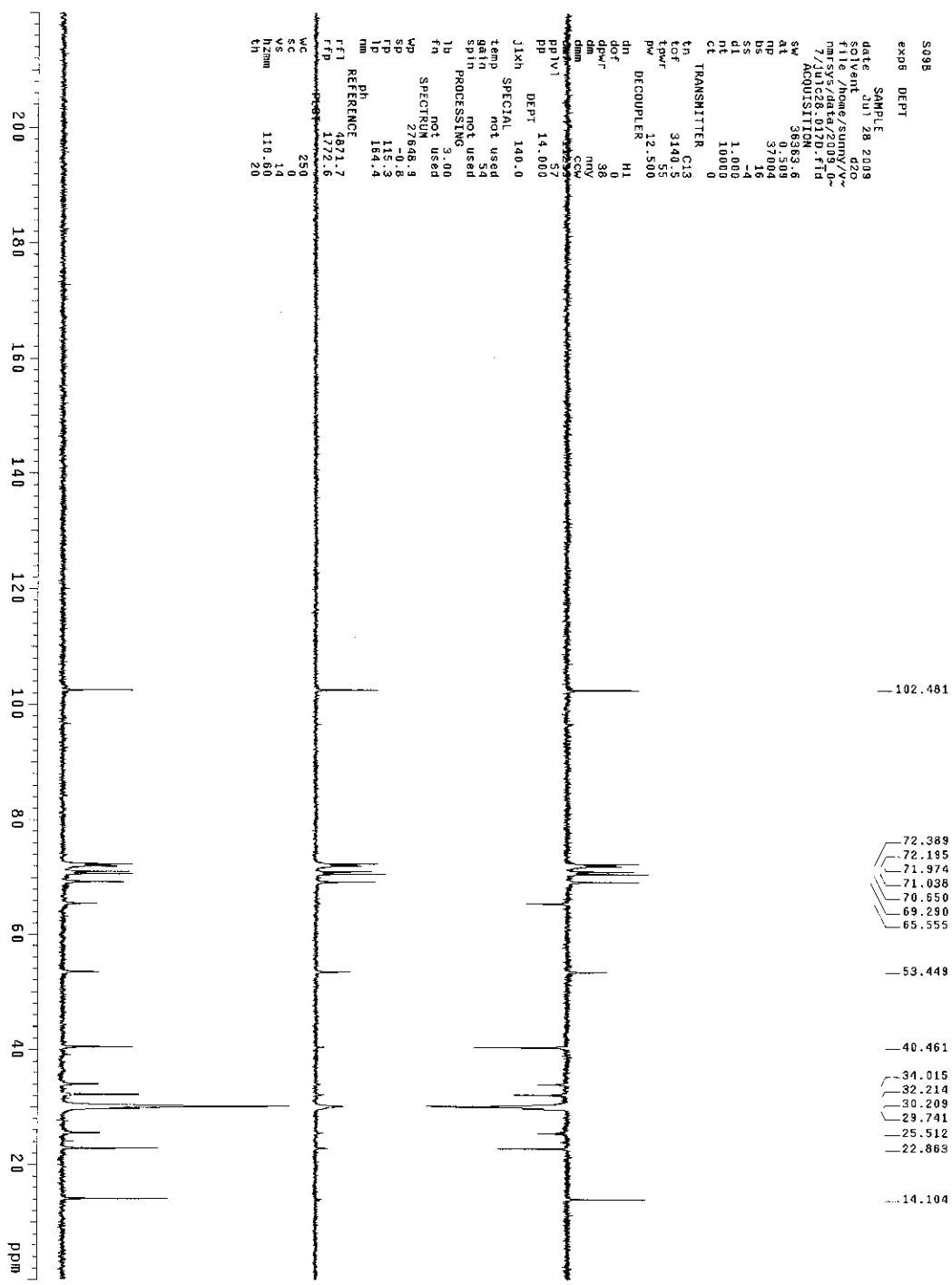
1.870
1.861
1.850
1.622
1.614
1.603
1.594
1.583
1.576
1.523
1.409
1.398
1.308
0.921
0.909
0.895
0.459



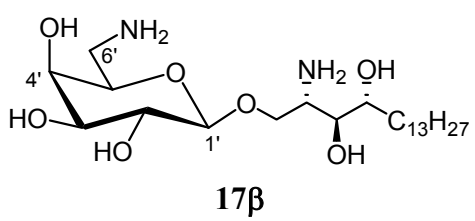
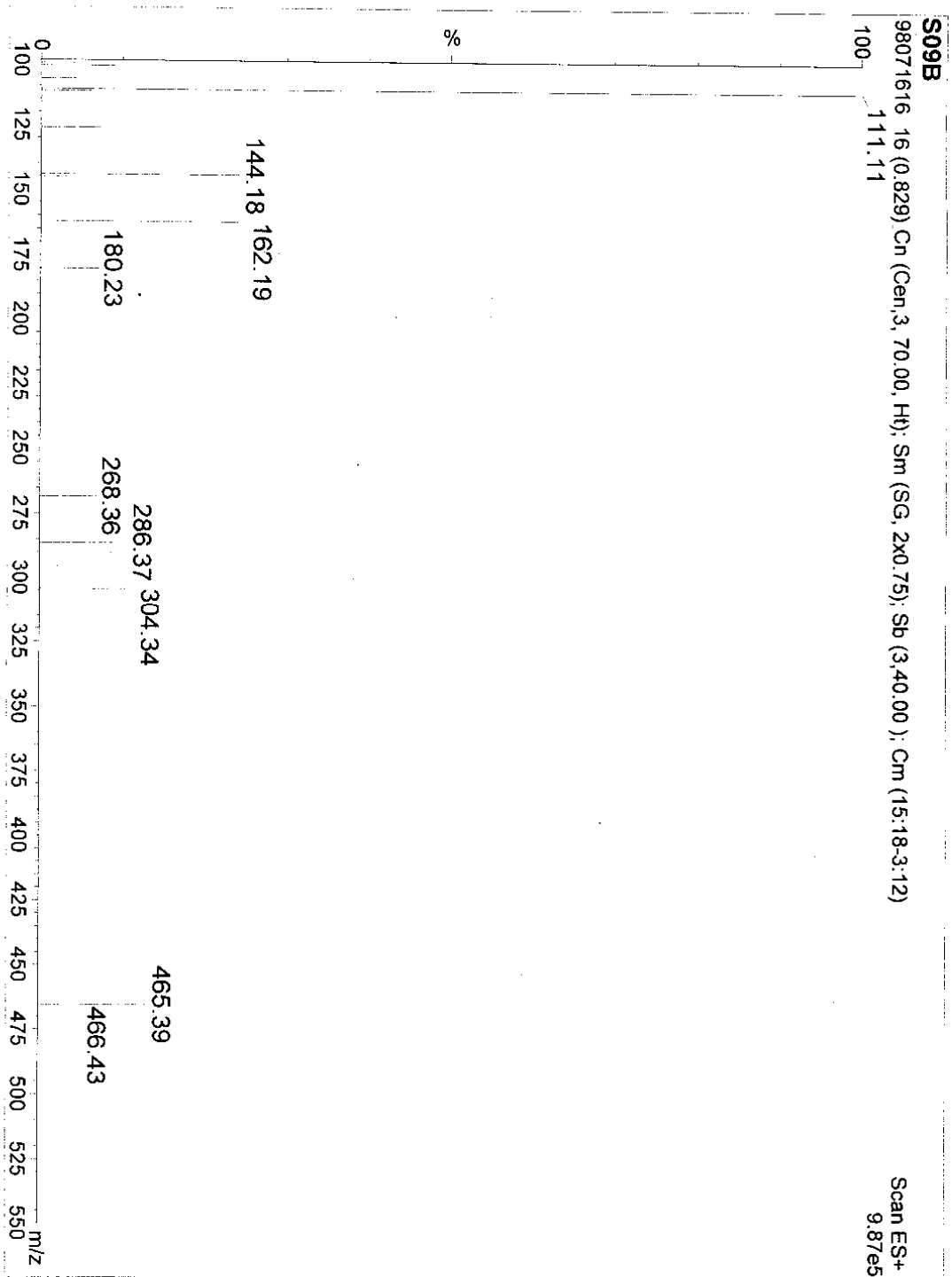
14β

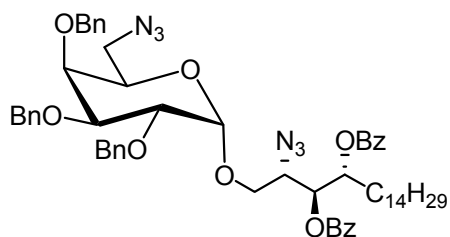
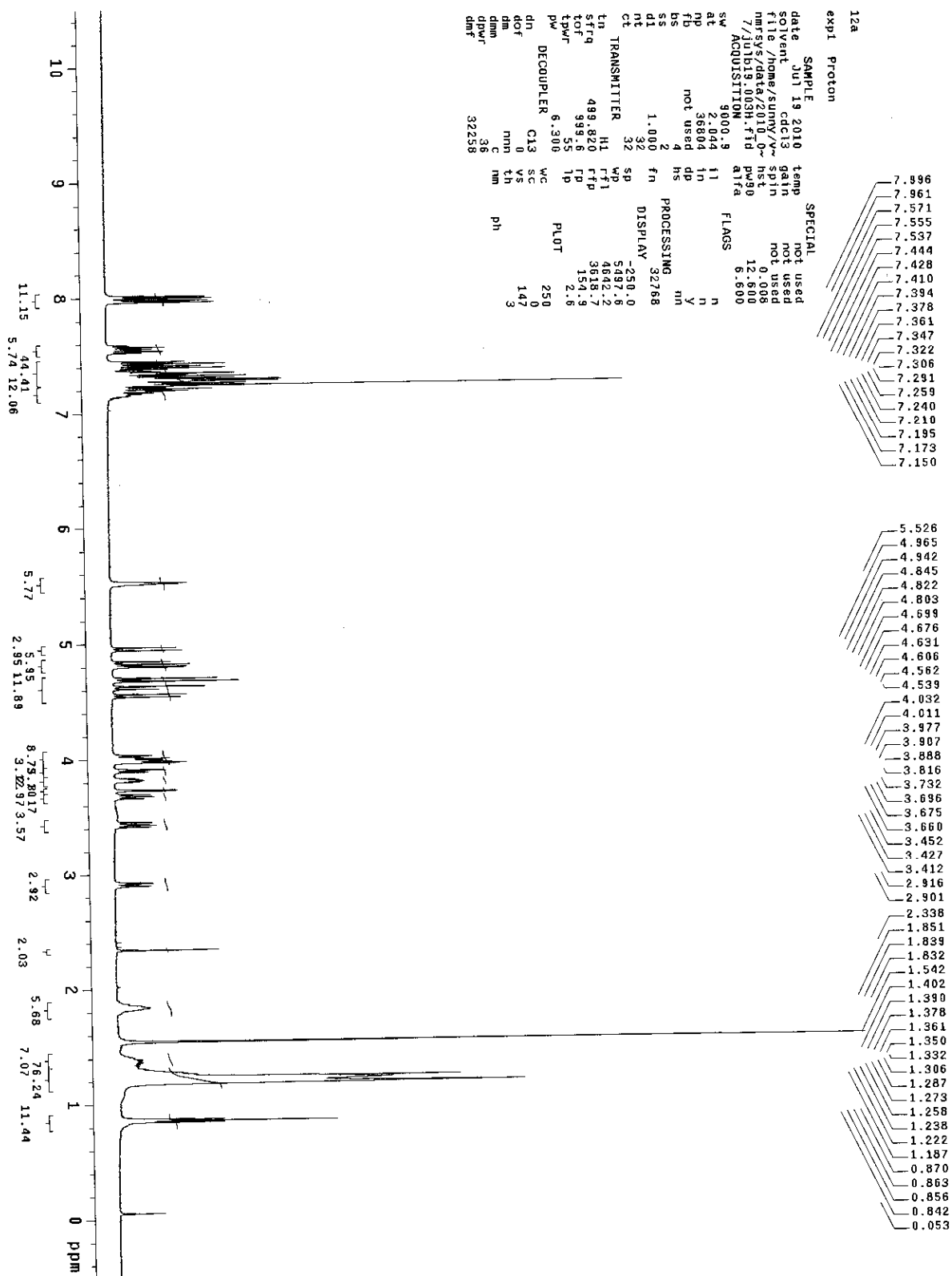


17β



17β





15α

12a
 exp3 DEPT
 SAMPLE
 date JUL 19 2010
 solvent cdcl3
 f1 name/home/500mhz/1
 f2 name/home/500mhz/1
 7/19/19.0030.f1d
 ACQUISITION
 sw 36363.6
 at 0.510
 np 37088
 bs 20
 ss -4
 d1 1.000
 nt 10000
 ct TRANSMITTER C13
 tn 3140.5
 tof 3140.5
 tpuw 12.500
 pw DECOUPLER H1
 dn 0
 dof 0
 dpwr 38
 dm nny
 dnm CCW
 ppiw 57
 pp 14.000
 DEPT 140.0
 J1kh SPECIAL
 temp not used
 gain 5.4
 spin not used
 PROCESSING
 lb 3.00
 fn not used
 SPECTRUM 27648.9
 wp -0.6
 sp 5.5
 tp 158.0
 nm
 ph
 REFERENCE
 rft 4908.3
 rfd 1773.8
 wc 250
 sc 0
 vs 25
 hzmm 110.60
 th 35

200 180 160 140 120 100 80 60 40 20 ppm

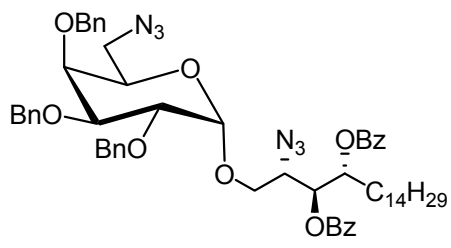
165.731
165.149

138.703
 138.509
 138.109
 133.476
 133.158
 129.865
 129.732
 129.317
 128.558
 128.469
 128.434
 128.381
 128.266
 127.887
 127.825
 127.586
 127.551

98.801

78.422
 77.256
 77.000
 76.753
 76.214
 75.199
 74.581
 73.653
 73.124
 72.903
 72.859
 70.316
 68.541
 61.362
 51.402

31.915
 29.866
 29.637
 29.584
 29.504
 29.407
 29.380
 25.310
 22.679
 14.114

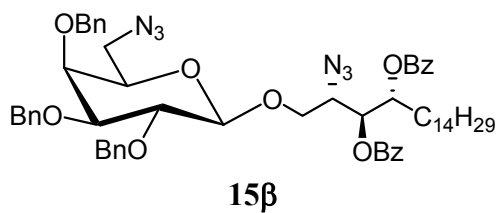
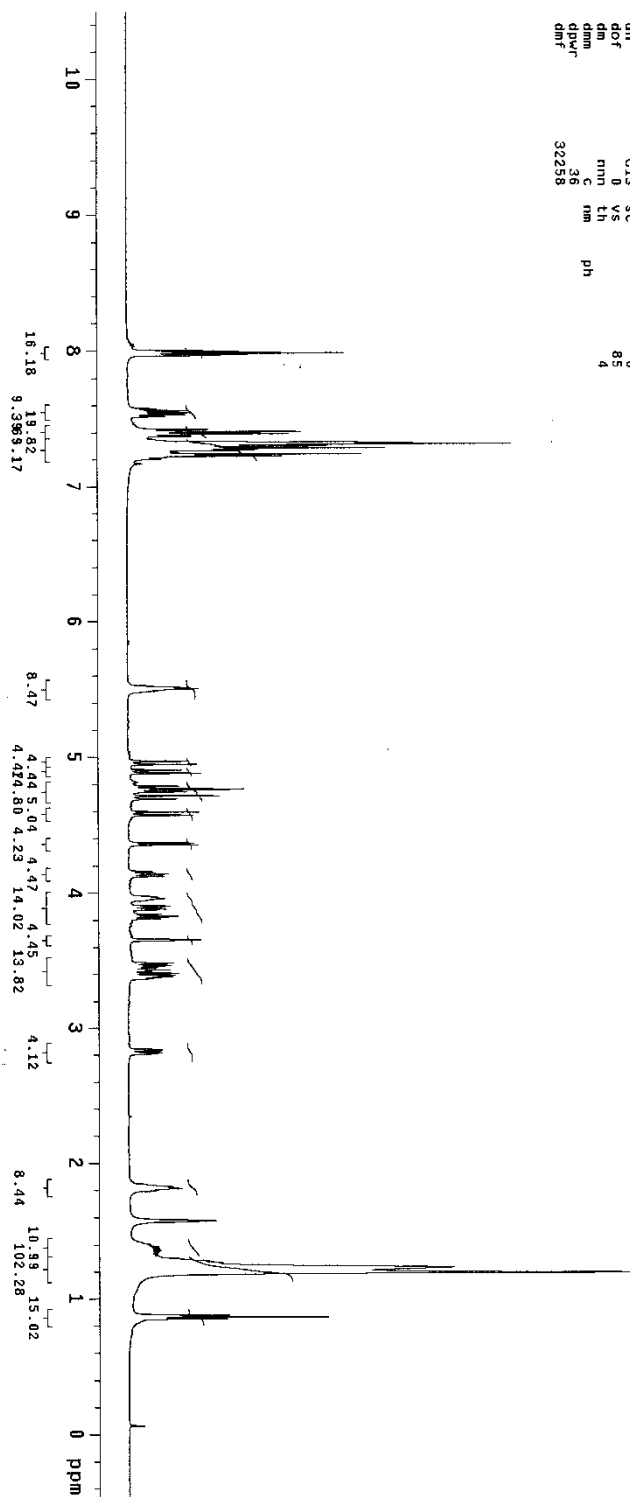


15a

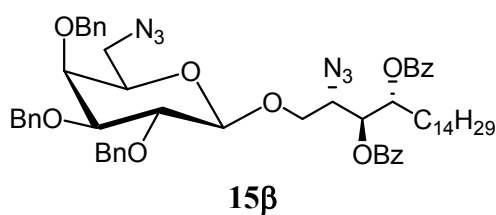
1.825
1.812
1.797
1.574
1.384
1.369
1.355
1.341
1.323
1.280
1.266
1.230
1.191
0.877
0.863
0.848

SAMPLE	SPECIAL
JUL 19 2010	temp not used

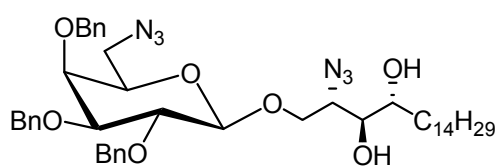
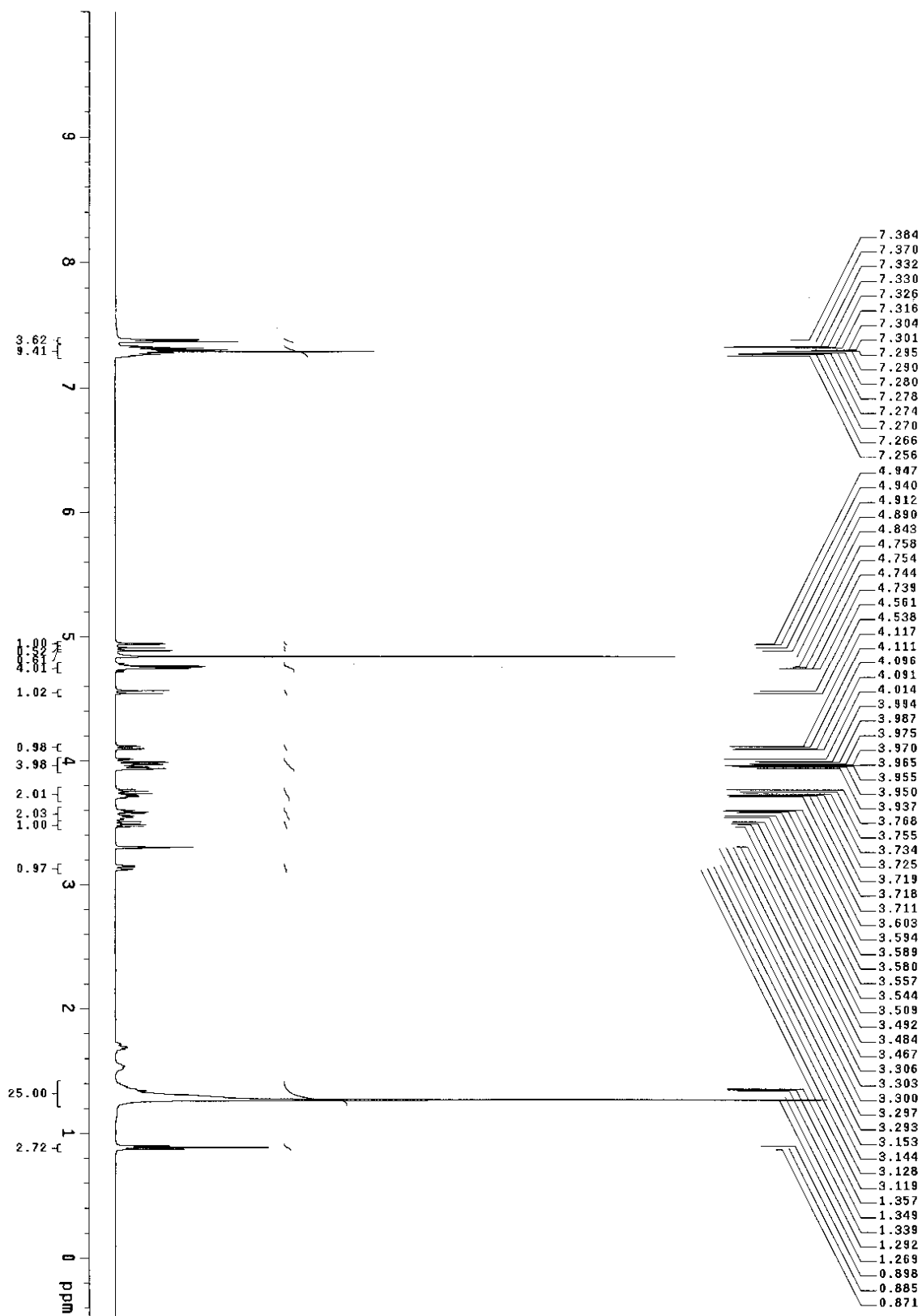
SAMPLE	date	JUL 19 2010	temp
solvnt	CdCl ₃	gain	not used
file /home/sunny/v-	spin	not used	not used
mmsys\data\2010_0	pst	0.0008	0.0008
/JMODSIN	nm-ftd	PW0	1.6800
ACQUISITION	afta	FLAAS	
sw	9000.9		n
at	2.0-04	11	n
np	not	36804	in
fb	used	pd	y
bs	4	hs	n
ss	2		
cs	1.002		
ct	1.92		
TRANSMITTER	32	fn	PROCESSING
tn	H1	f1	DISPLAY
tfreq	499.820	rfp	-250.0-0.0
torf	999.6	tp	5497.6-6.6
twpw	35	wc	4641.6-6.6
DECOUPLER	6.300	lp	3618.7-7.7
din	G13	ph	8.3-
dof	d	vs	250.0
dmm	mn	ns	85.0
c	nm		
dmf	36		
320258			



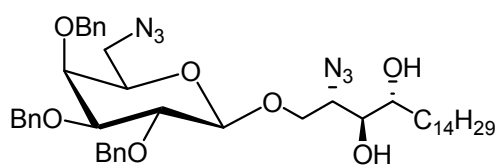
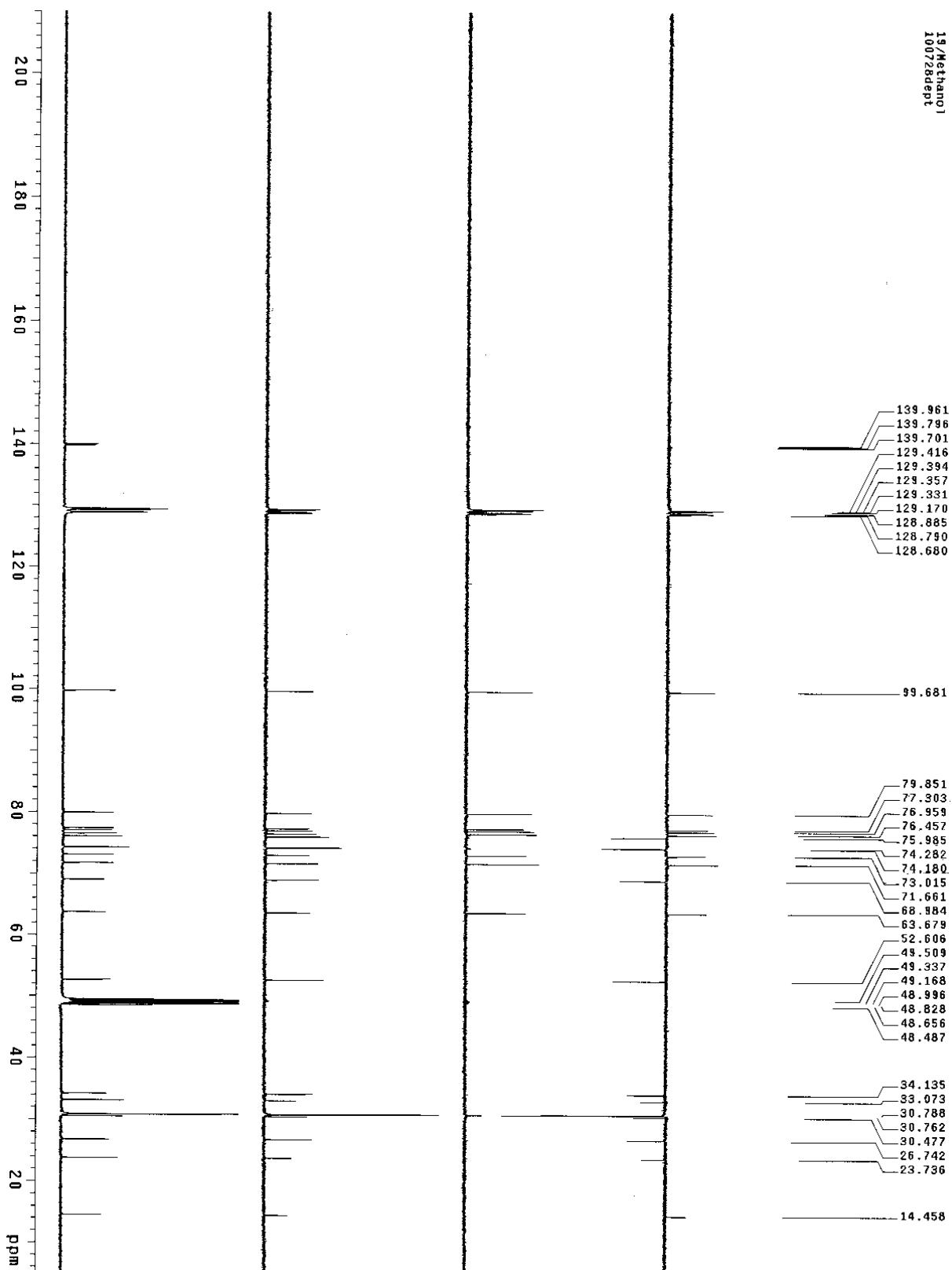
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29.619
29.566
29.486
29.389
29.336
25.275
22.670
14.096



100728H

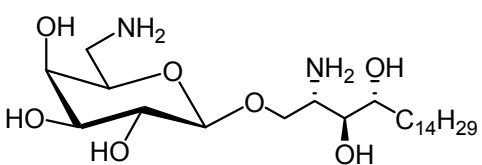
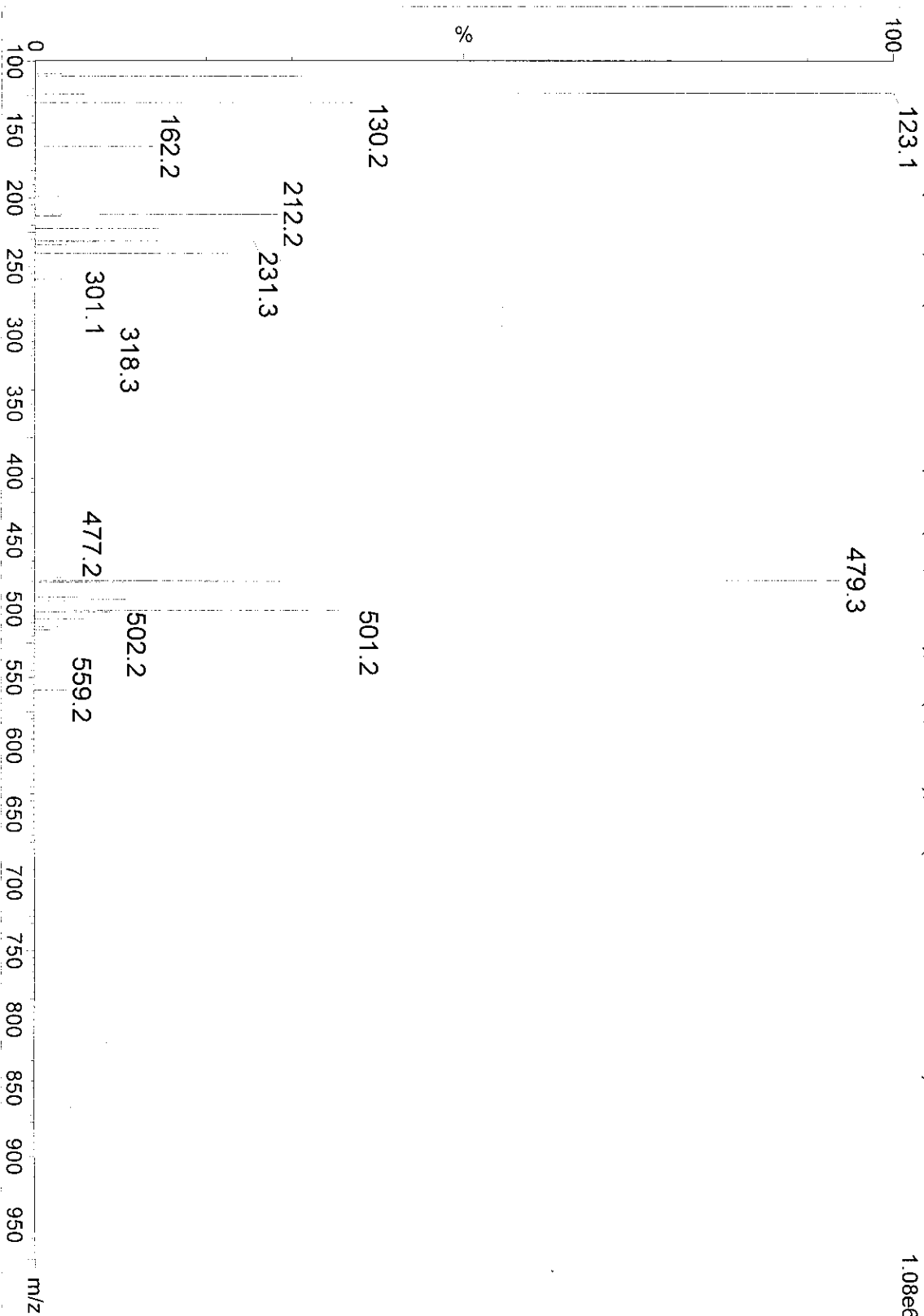


13/Methanol
100728dept

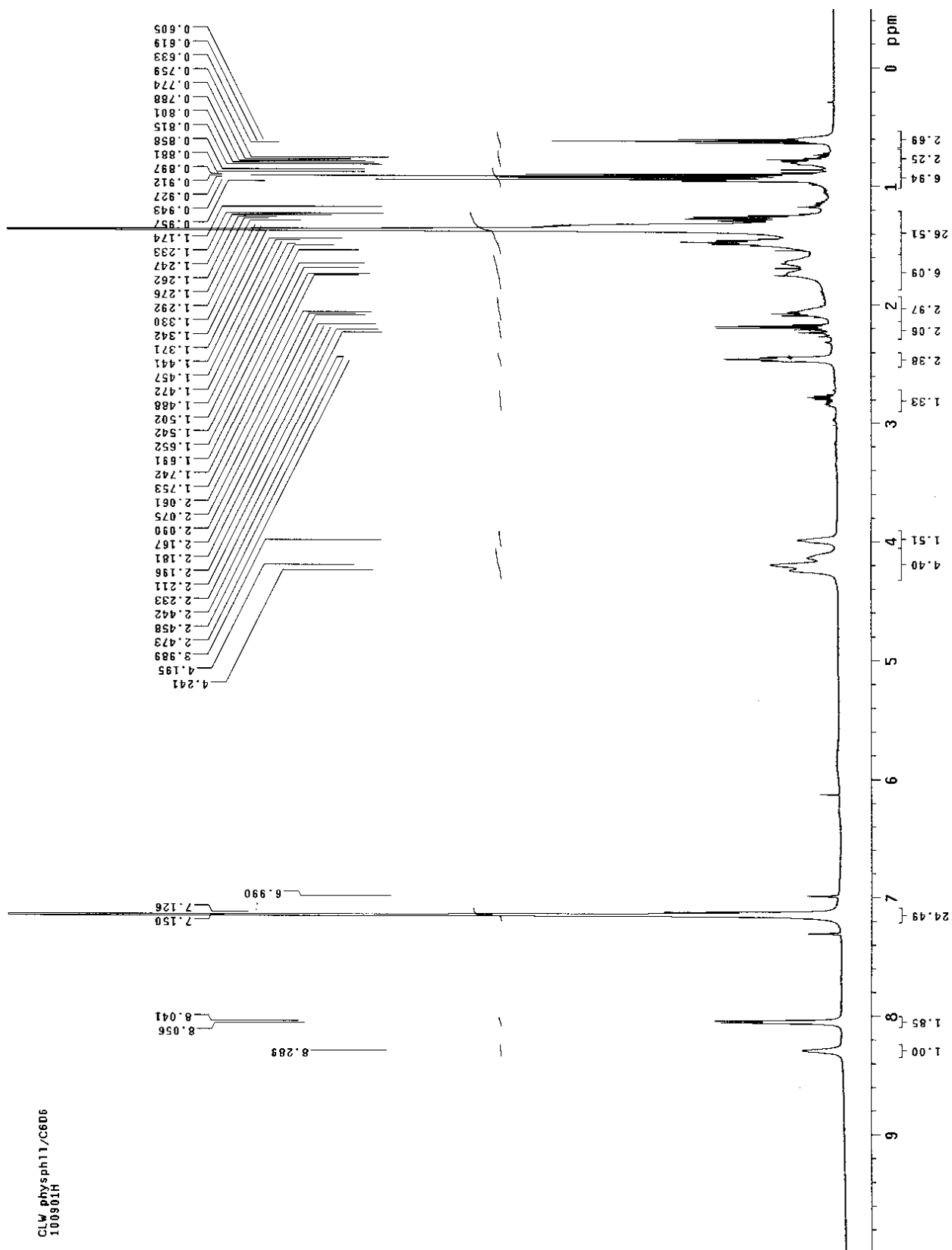
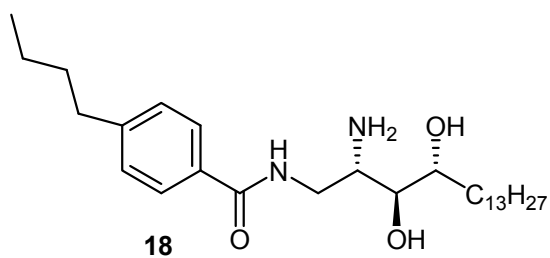


20

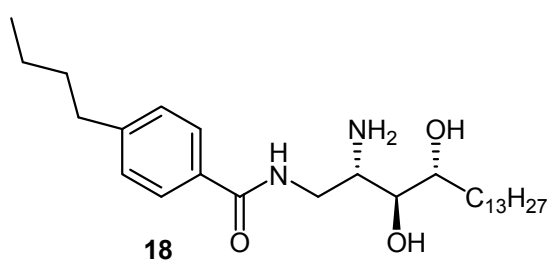
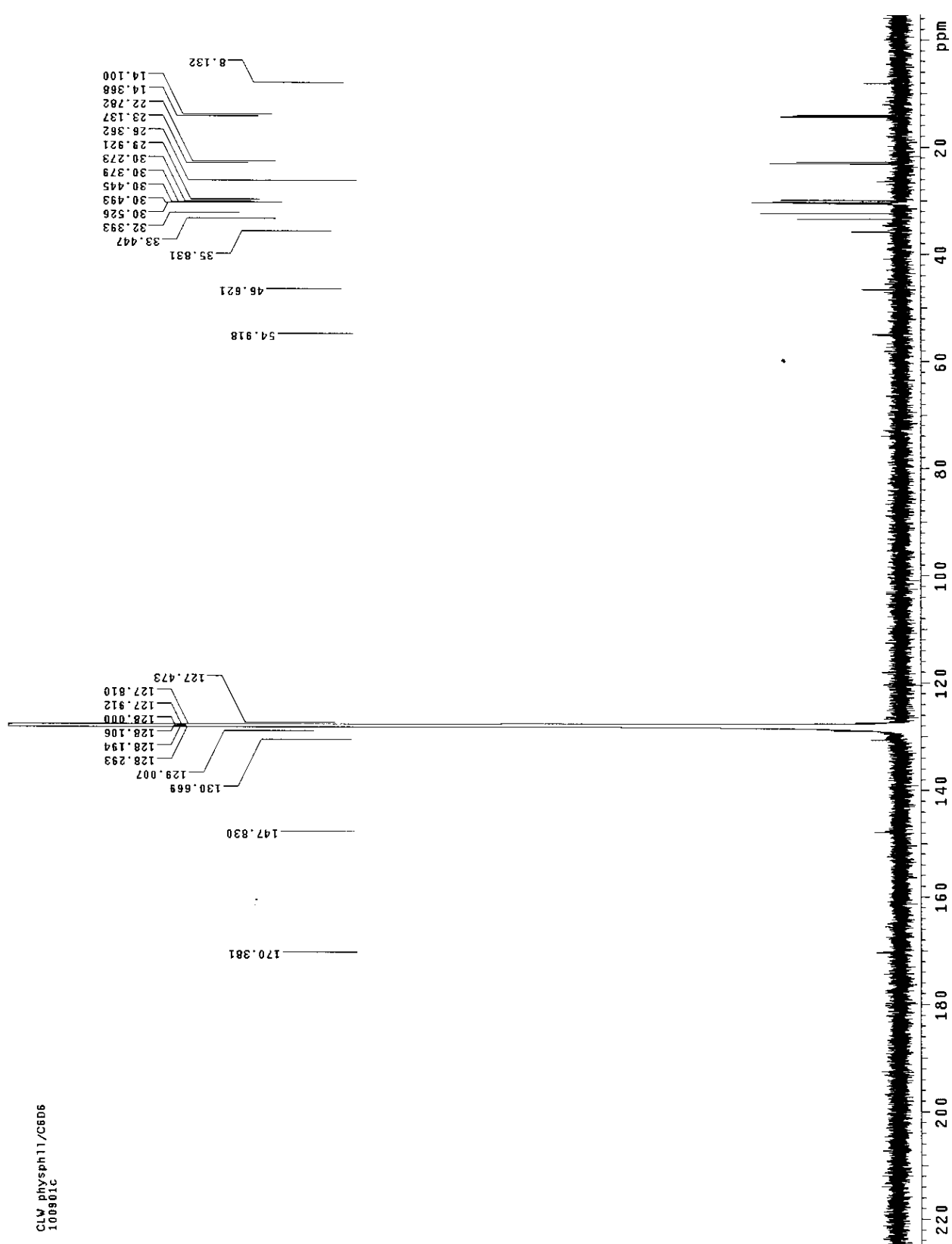
2010072705_110 (5.702) Cn (Cen,3, 80.00, Hb); Sm (SG, 2x0.75); Sb (3,40.00); Cm (107.112-72:104x2.000)

Scan ES+
1.08e6

2



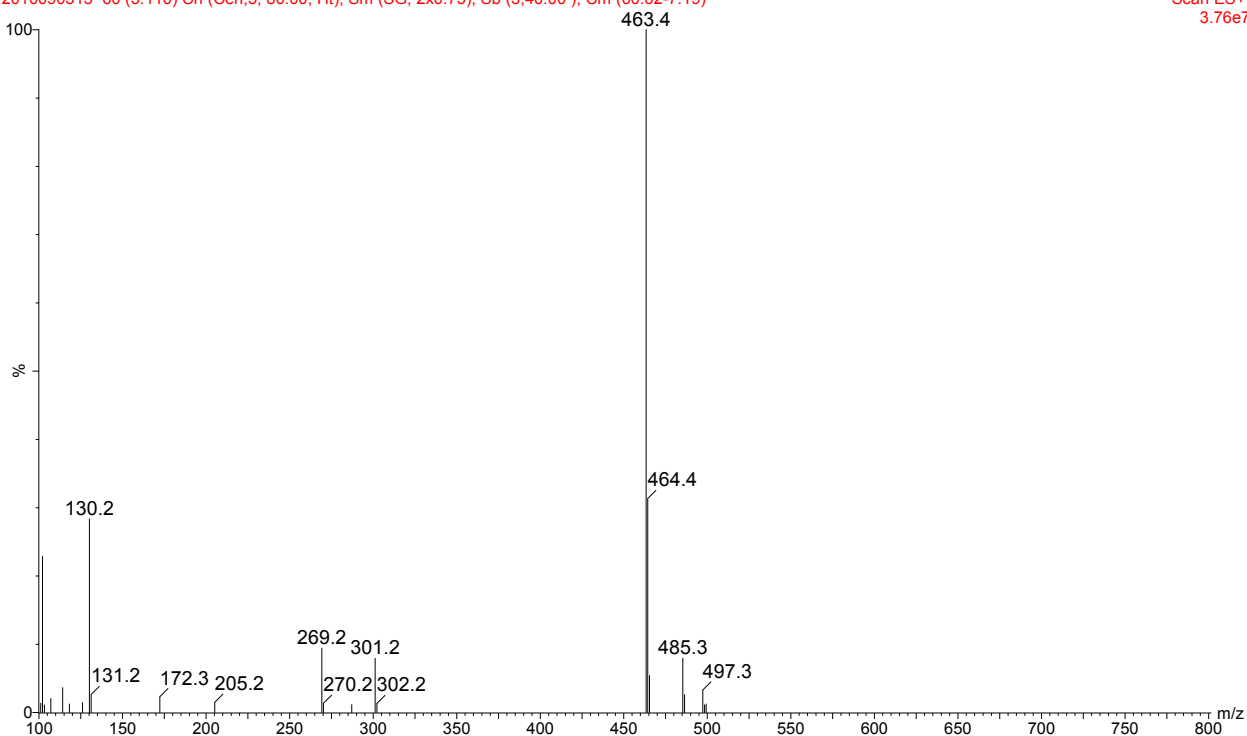
CLW physph11/C606
100901H



CLWphysph11

2010090313 60 (3.110) Cn (Cen,3, 80.00, Ht); Sm (SG, 2x0.75); Sb (3,40.00); Cm (60:62-7:19)

Scan ES+
3.76e7



101.0 4.995e5

102.1 8.586e6

103.2 4.086e5

107.0 7.572e5

114.1 1.361e6

118.1 4.668e5

126.0 5.419e5

130.2 1.065e7

131.2 9.776e5

172.3 8.613e5

205.2 5.447e5

269.2 3.542e6

270.2 4.965e5

287.2 4.321e5

301.2 2.977e6

302.2 4.835e5

463.4 3.758e7

464.4 1.177e7

465.4 2.025e6

485.3 2.978e6

486.4 9.812e5

497.3 1.221e6

498.3 4.184e5

499.3 4.683e5

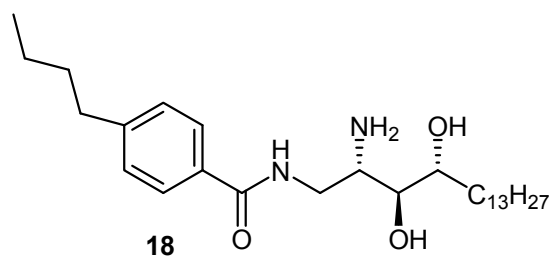
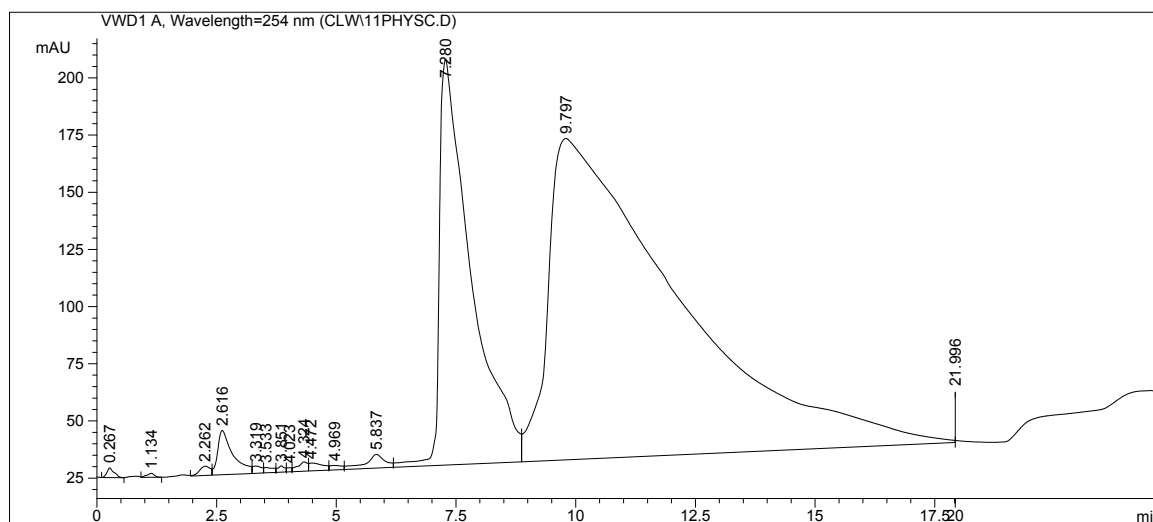
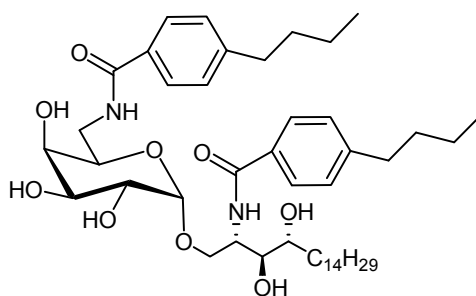
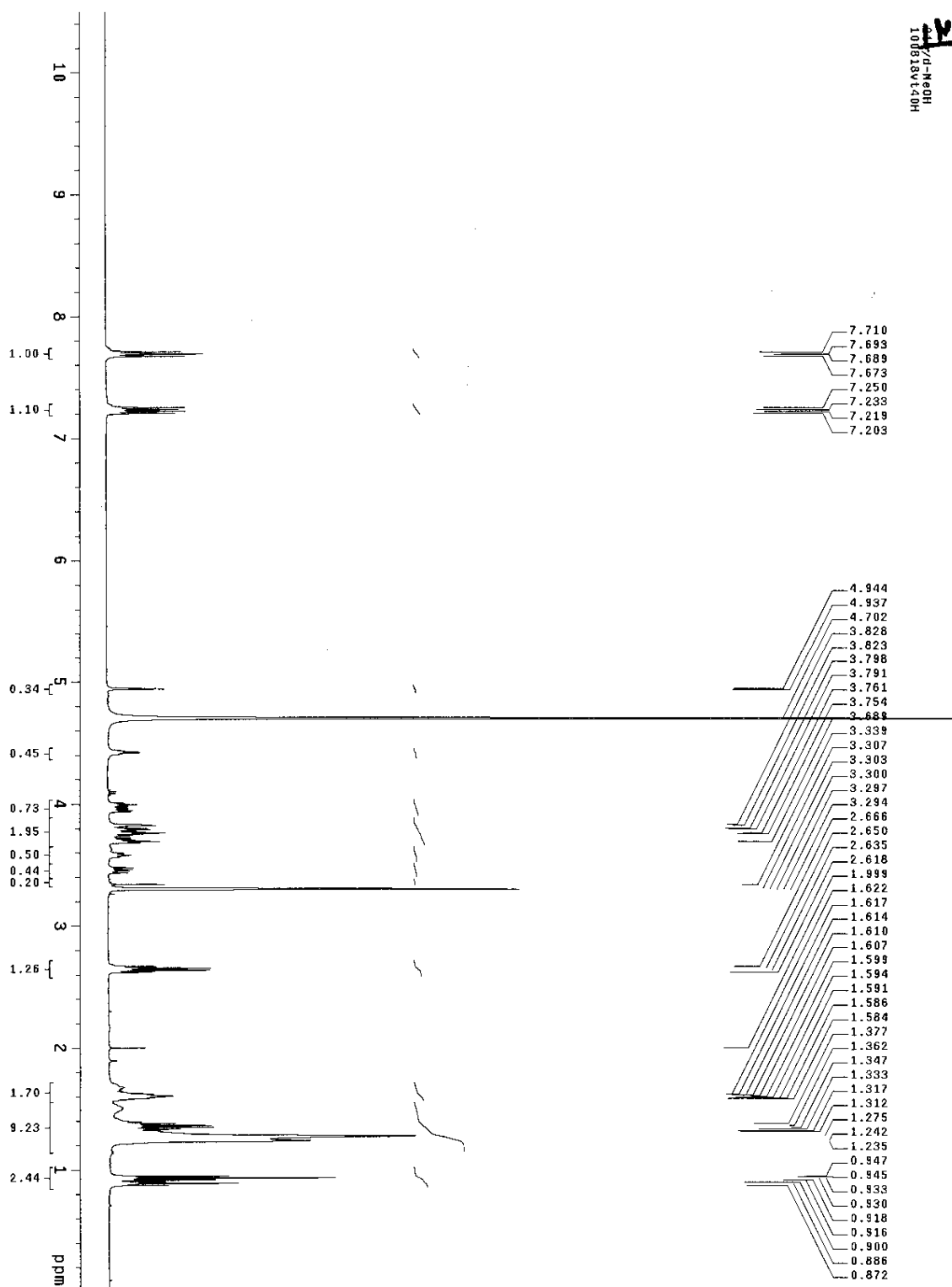


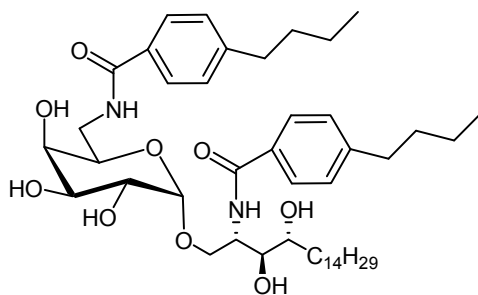
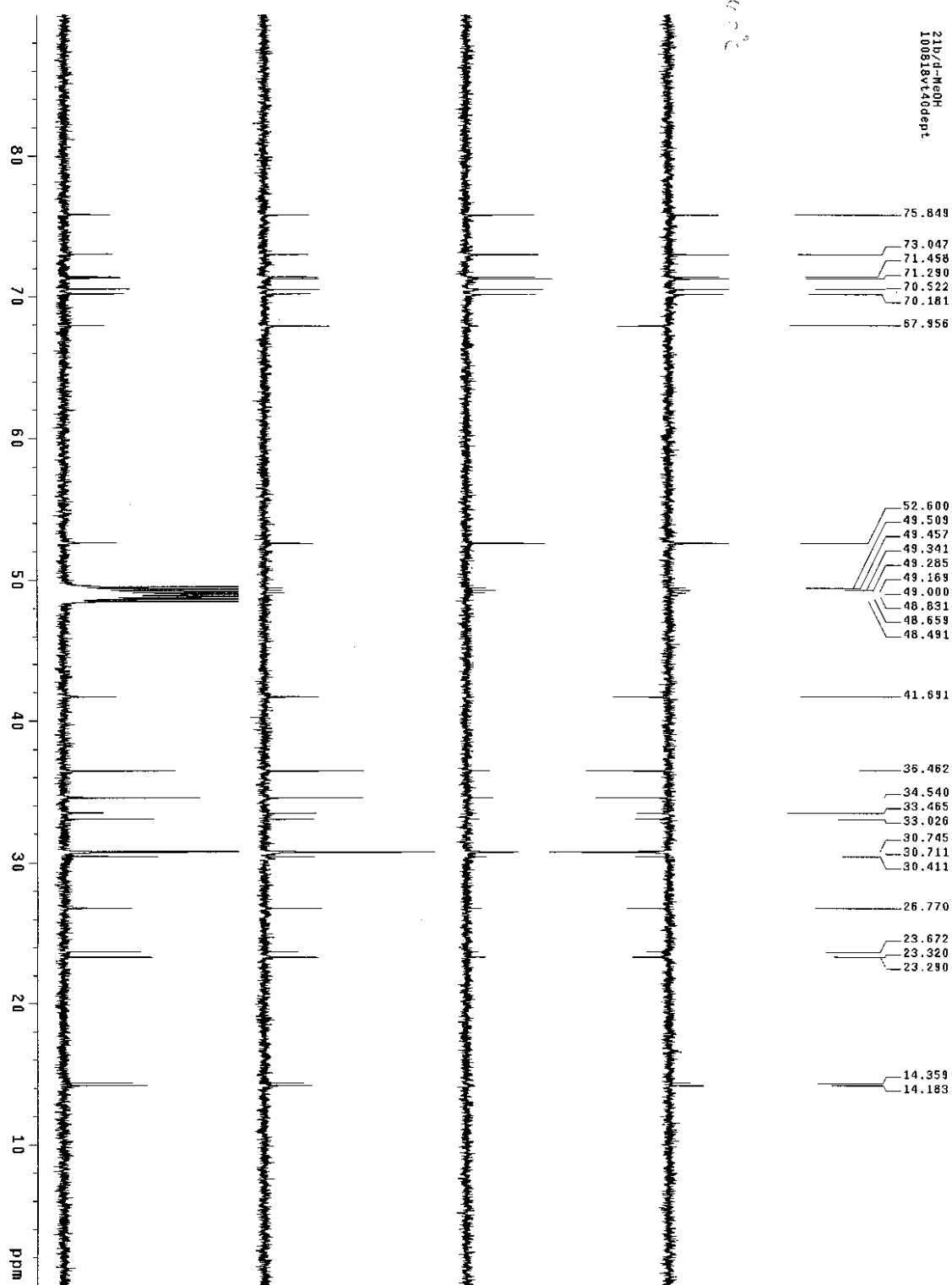
Figure 2. Chromatogram for the mixtures of amide compound **18** after purification with column chromatography.

Figure 3. HPLC chromatogram derived from the reinjection of the collected fraction shown in Fig. 1.





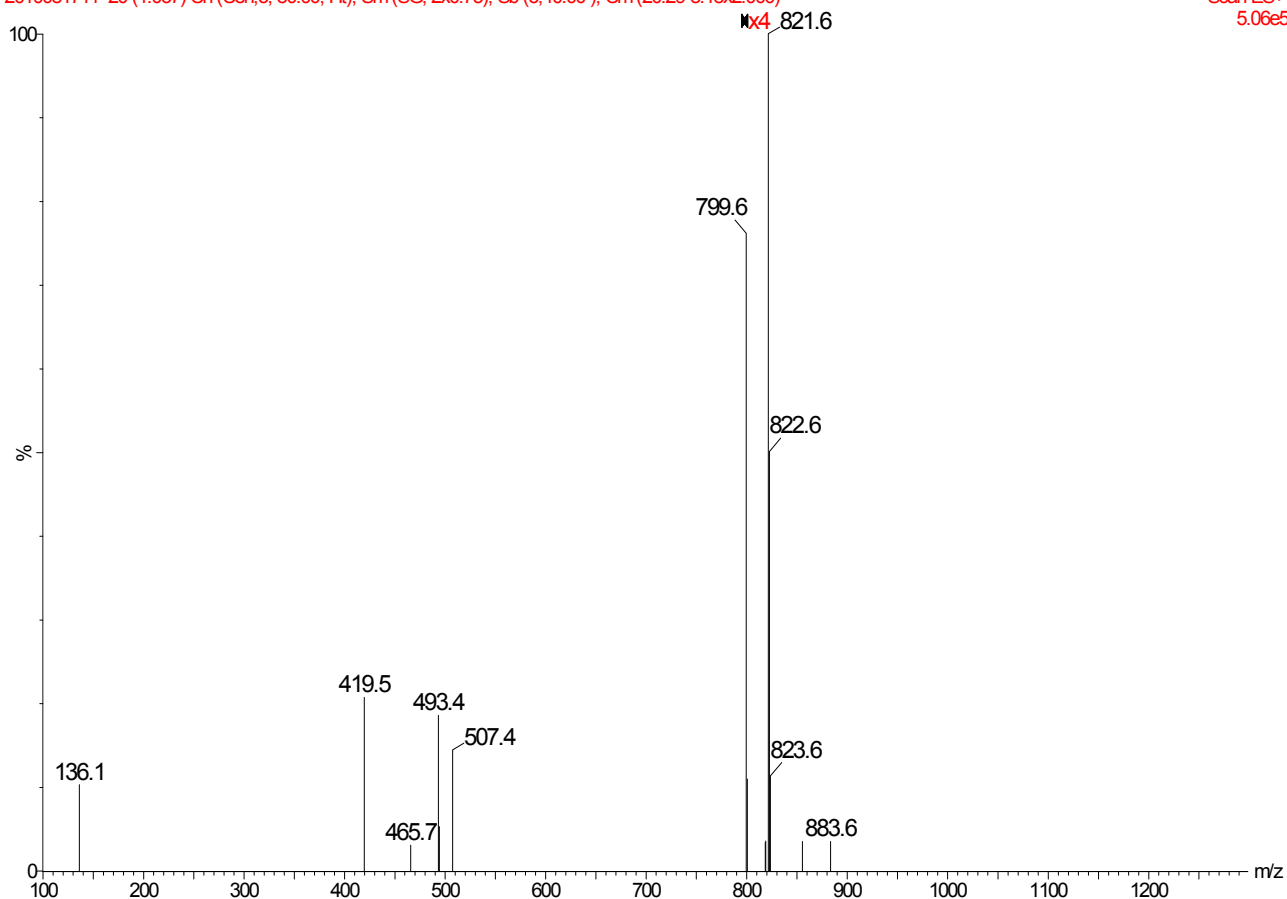
19



19

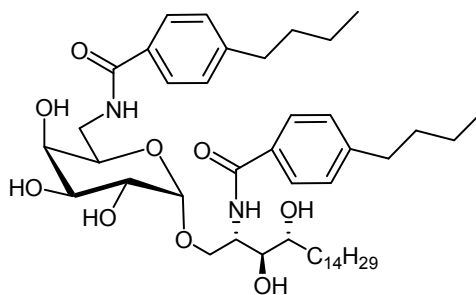
2010081711 20 (1.037) Cn (Cen,3, 80.00, Ht); Sm (SG, 2x0.75); Sb (3,40.00); Cm (20:29-8:18x2.000)

Scan ES+
5.06e5



2010081711 20 (1.037) Cn (C

No	Mass	Inten	%BPI	%TIC
1:	136.1	5.21e4	10.30	2.04
2:	419.5	1.05e5	20.73	4.10
3:	465.7	1.56e4	3.09	0.61
4:	493.4	9.40e4	18.59	3.68
5:	494.3	2.68e4	5.30	1.05
6:	507.4	7.31e4	14.44	2.86
7:	508.4	1.22e4	2.41	0.48
8:	799.6	9.63e4	19.04	3.77
9:	800.6	5.56e4	10.99	2.18
10:	818.6	1.72e4	3.40	0.67
11:	819.0	1.80e4	3.56	0.71
12:	819.7	1.31e4	2.60	0.51
13:	821.6	5.06e5	100.00	19.79
14:	822.6	2.53e5	50.09	9.91
15:	823.6	5.73e4	11.33	2.24
16:	835.5	1.02e4	2.03	0.40
17:	855.5	1.78e4	3.51	0.70
18:	883.6	1.78e4	3.51	0.70
19:	889.6	1.18e4	2.34	0.46



19

Figure 4. HPLC Chromatogram for the mixtures of amide compound **19** obtained from purification through column chromatography.

