

Recherche et Développement

SampleName LCC-232-90

HPLC 3285-301098

Vial 3

Injection 1

Injection Volume 20.00 ul

Sample Type Unknown

Run Time 45.0 Minutes

Project Name EM_5

Date Acquired 30/Oct/1998 12:53:30

Acq Method Set EM2

Processing Method EM1

Date Processed 02/Nov/1998 16:46:16

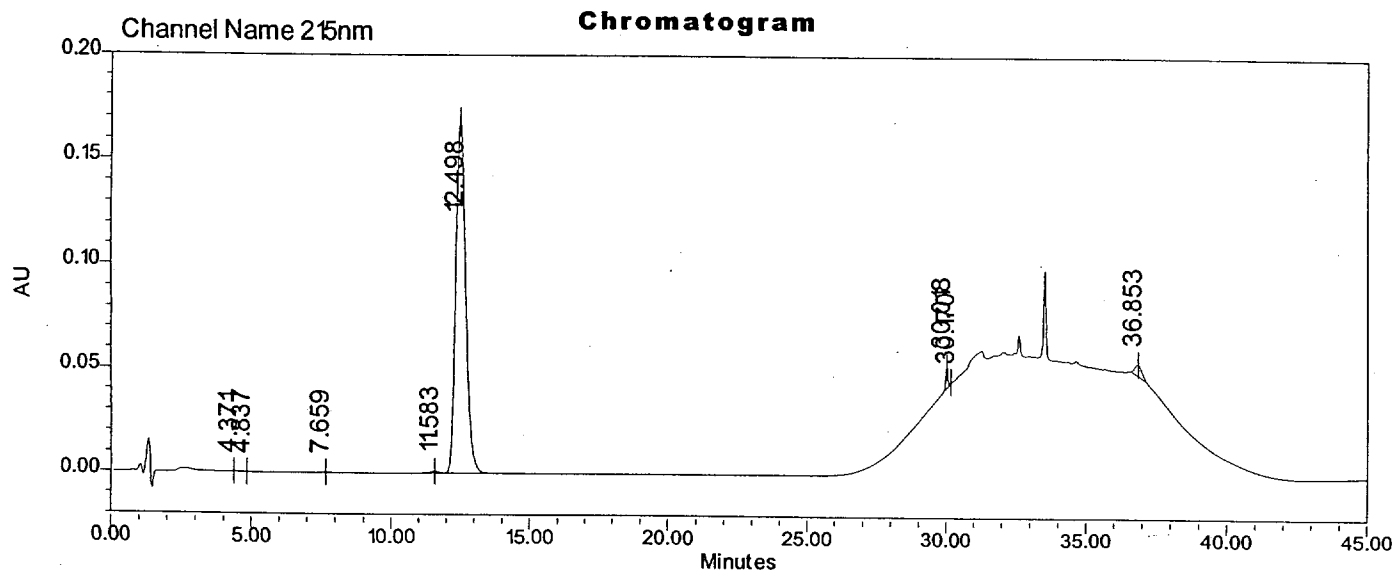
Column: Nova Pak C18, #Lot: T80791P 220

Column T': 25°C

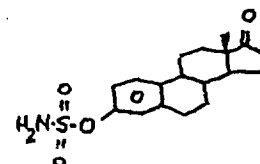
Dimension: 150mm x 3.9 mm id

Flow: 1 mL/min

HPLC conditions: Time	ACN	H2O	MeOH
0	25	55	20
20	25	55	20
25	0	0	100
30	0	0	100
35	25	55	20
45	25	55	20

**Peak Results**

RT	Area	%Area	Purity1 Angle	Purity1 Threshold
1 4.37	5345	0.12	24.021	23.532
2 4.84	2233	0.05	34.720	51.146
3 7.66	7078	0.16	10.634	12.894
4 11.58	18336	0.42	3.141	3.697
5 12.50	4223018	95.75	0.133	0.300
6 30.02	54522	1.24	1.707	1.236
7 30.17	4197	0.10		
8 36.85	95592	2.17	2.158	1.386



Vérifié aussi à 205 nm

Selon les normes HPLC: Accepté____ Refusé____ # Lot du LREM:____

Analyste

Signature: N. Lafontaine

Date: 02 nov. 98

Responsable du HPLC

Signature:

Date: 03 nov. 98

Recherche et Développement

SampleName LCC-232-103

HPLC 3300-051198

Vial 21

Injection 1

Injection Volume 20.00 ul

Run Time 45.0 Minutes

Project Name EM_5

Date Acquired 28/Nov/1998 08:46:27

Acq Method Set EM_4

Processing Method EM

Date Processed 30/Nov/1998 10:13:24

Column: Nova-Pak, C18 #Lot: T80791P 220

Column T°: 25°C

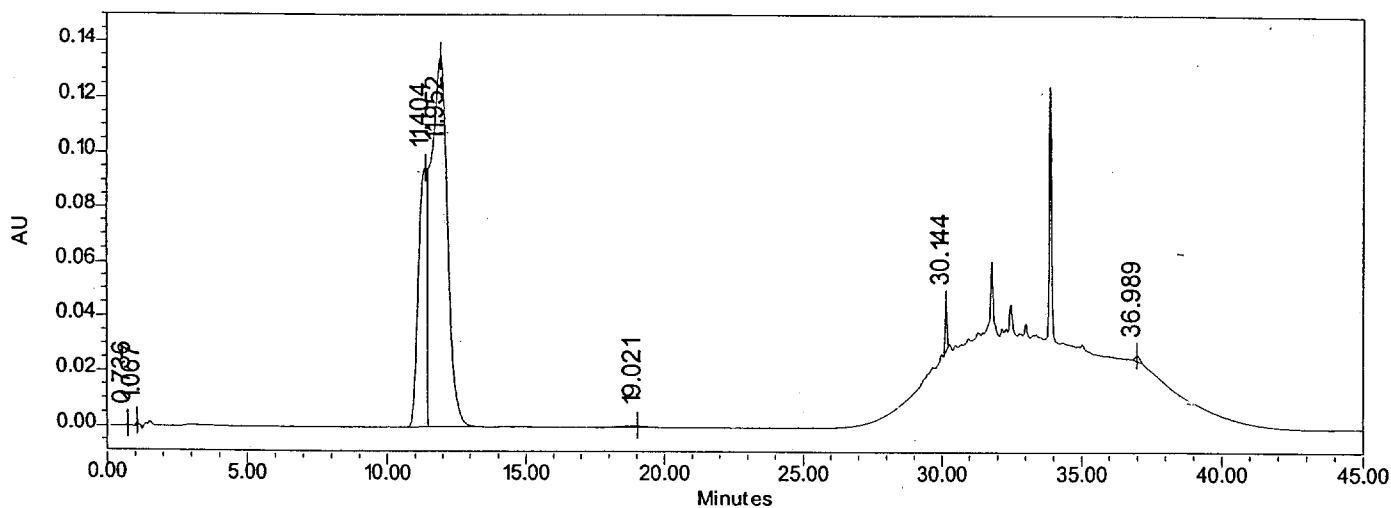
Dimension: 150mm x 3.9 mm id

HPLC #2

Flow: 1 ml/min

HPLC conditions:	Time	ACN	H2O	MeOH
	0	10	70	20
	20	10	70	20
	25	0	0	100
	30	0	0	100
	35	10	70	20
	45	10	70	20

Channel Name 220nm

Chromatogram**Peak Results**

RT	Area	%Area	Purity1 Angle	Purity1 Threshold
1 0.74	1432	0.02		
2 1.07	7512	0.10		
3 11.40	2051866	26.49	0.113	0.321
4 11.95	5551043	71.67	0.089	0.294
5 19.02	30574	0.39	14.516	18.728
6 30.14	77960	1.01		
7 36.99	24540	0.32	6.147	4.949



98%

Vérifier aussi à $\lambda = 275 \text{ nm}$

Selon les normes CLHP: Accepté _____ Refusé _____

#Lot de LREM: _____

Analyste

Signature: *N. Lafontaine*

Date: 30 nov. 98

Responsable CLHP

Signature: *J. Madou*

Date: 30 nov. 98

Recherche et Développement

SampleName RM-240-127
 HPLC 3301-051198
 Vial 5
 Injection 1
 Injection Volume 20.00 ul
 Sample Type Unknown
 Run Time 45.0 Minutes
 Project Name EM_6
 Date Acquired 16/Nov/1998 20:54:40
 Acq Method Set EM3
 Processing Method EM1
 Date Processed 17/Nov/1998 08:4126

Column: Nova Pak C18, #Lot: T80791P 220

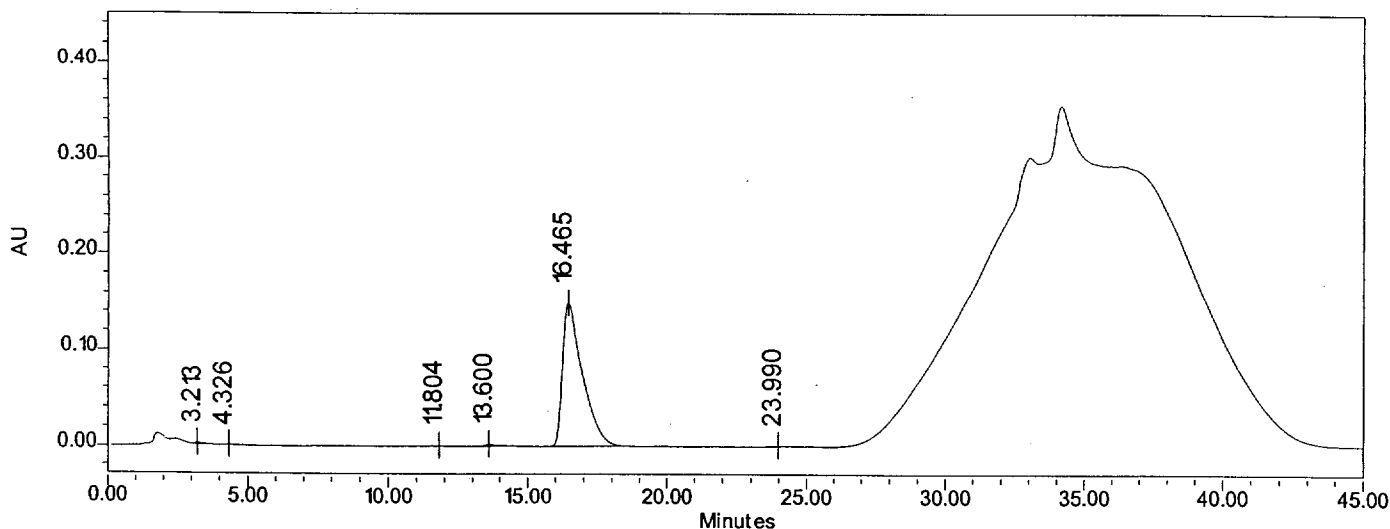
Column T°: 25°C

Dimension: 150mm x 3.9 mm id

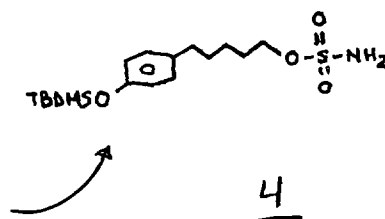
Flow: 1 mL/min

HPLC conditions: Time	ACN	H2O	MeOH
0	40	30	40
20	40	30	40
25	0	0	100
30	0	0	100
35	40	30	40
45	40	30	40

Channel Name 205nm

Chromatogram**Peak Results**

RT	Area	%Area	Purity1 Angle	Purity1 Threshold
1 3.21	27128	0.37	3.093	2.961
2 4.33	7333	0.10	24.273	35.349
3 11.80	6973	0.09	16.866	21.125
4 13.60	57840	0.78	4.875	5.441
5 16.46	7253273	98.40	0.042	0.255
6 23.99	18486	0.25	22.697	29.139



Selon les normes CLHP: Accepté _____ Refusé _____ # Lot de LREM: _____

Analyste

Signature: *J. Kader*

Date: 17 nov. 98

Responsable CLHP:

Signature: *[Signature]*

Date: 17 nov. 98

APC 06 5

Recherche et Développement

SampleName LCC-232-99
HPLC 3299-05198

Vial 25
Injection 1
Injection Volume 20.00 ul

Sample Type Unknown

Run Time 45.0 Minutes

Project Name EM_5

Date Acquired 07/Nov/1998 09:23:35

Acq Method Set EM6

Processing Method EM_1

Date Processed 09/Nov/1998 14:37:13

Column: Nova Pak C18, #Lot: T80791P 017

Column T°: 25°C

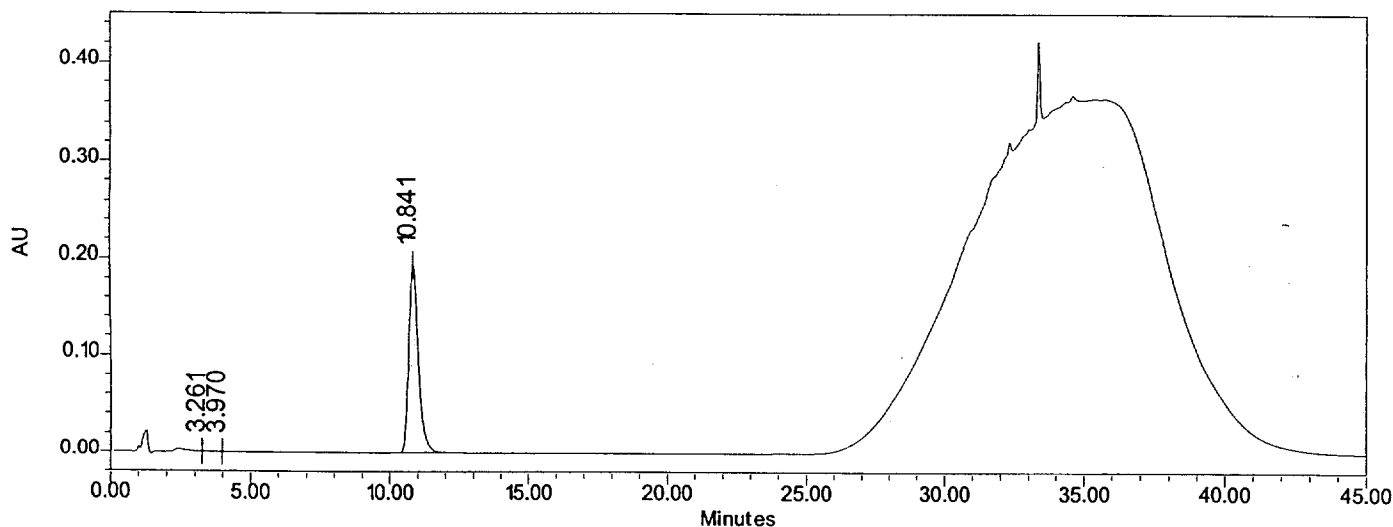
Dimension: 150mm x 3.9 mm id

Flow: 1 mL/min

HPLC conditions: Time	ACN	H2O	MeOH
0	25	55	20
20	25	55	20
25	0	0	100
30	0	0	100
35	25	55	20
45	25	55	20

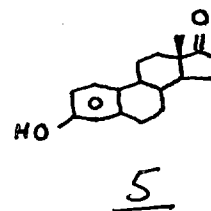
Channel Name 205nm

Chromatogram



Peak Results

RT	Area	%Area	Purity1 Angle	Purity1 Threshold
1 3.26	1526	0.03		
2 3.97	2887	0.06	6.142	10.098
3 10.84	4598729	99.90	0.107	1.547



Selon les normes CLHP: Accepté _____ Refusé _____ #Lot de LREM: _____

Analyste

Signature: *C. Bouchar*

Date: 09 nov. 98

Responsable CLHP

Signature: *[Signature]*

Date: 10 nov. 98

Recherche et Développement

SampleName LCC-232-107
HPLC 3316-101198

Vial 7
Injection 1
Injection Volume 20.00 ul

Sample Type Unknown
Run Time 45.0 Minutes
Project Name EM_7
Date Acquired 27/Nov/1998 19:39:50
Acq Method Set EM3
Processing Method EM1
Date Processed 30/Nov/1998 14:37:55

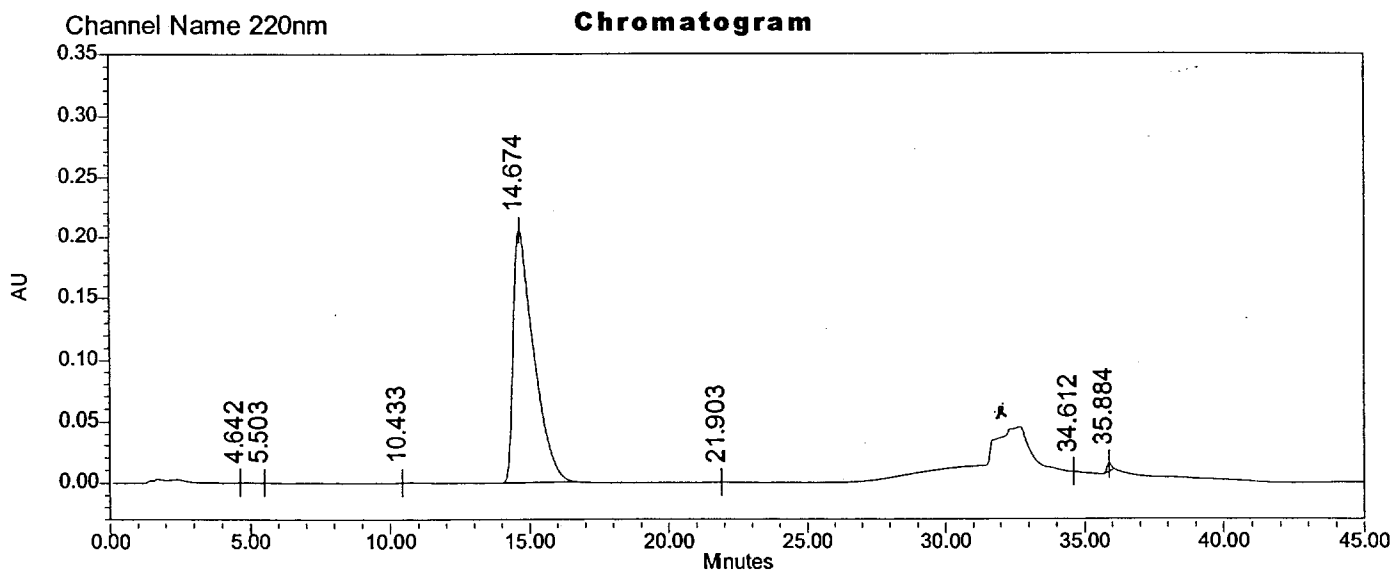
Column: Nova Pak C18, #Lot: T80791P 222

Column T': 25°C

Dimension: 150mm x 3.9 mm id

Flow: 1 mL/min

HPLC conditions: Time	ACN	H2O	MeOH
0	40	25	35
20	40	25	35
25	0	0	100
30	0	0	100
35	40	25	35
45	40	25	35

**Peak Results**

RT	Area	% Area	Purity1 Angle	Purity1 Threshold
1 4.64	5133	0.05	11.123	12.813
2 5.50	2258	0.02	24.611	39.427
3 10.43	6305	0.06	16.625	22.255
4 14.67	10399078	99.13	0.149	0.259
5 21.90	4738	0.05	25.699	37.111
6 34.61	4858	0.05	6.587	10.784
7 35.88	67497	0.64	0.811	1.337



7

Note: également accepté à 280 nm.

*: ce n'est pas une impureté.

Selon les normes CLHP: Accepté _____ Refusé _____

Lot de LREM: _____

Analyse

Signature: *Madu*

Date: 02 dec. 98

Responsable CLHP:

Signature: *Madu*

Date: 02 dec. 98

HPLC of 17

Recherche et Développement

SampleName LCC-232-135

HPLC 3423-151298

Vial 1

Injection 1

Injection Volume 20.00 ul

Sample Type Unknown

Run Time 45.0 Minutes

Project Name EM_7

Date Acquired 15/Dec/1998 09:37:56

Acq Method Set EM1

Processing Method EM1

Date Processed 15/Dec/1998 11:20:49

Column: Nova Pak C18, #Lot: T80791P 220

Column T: 25°C

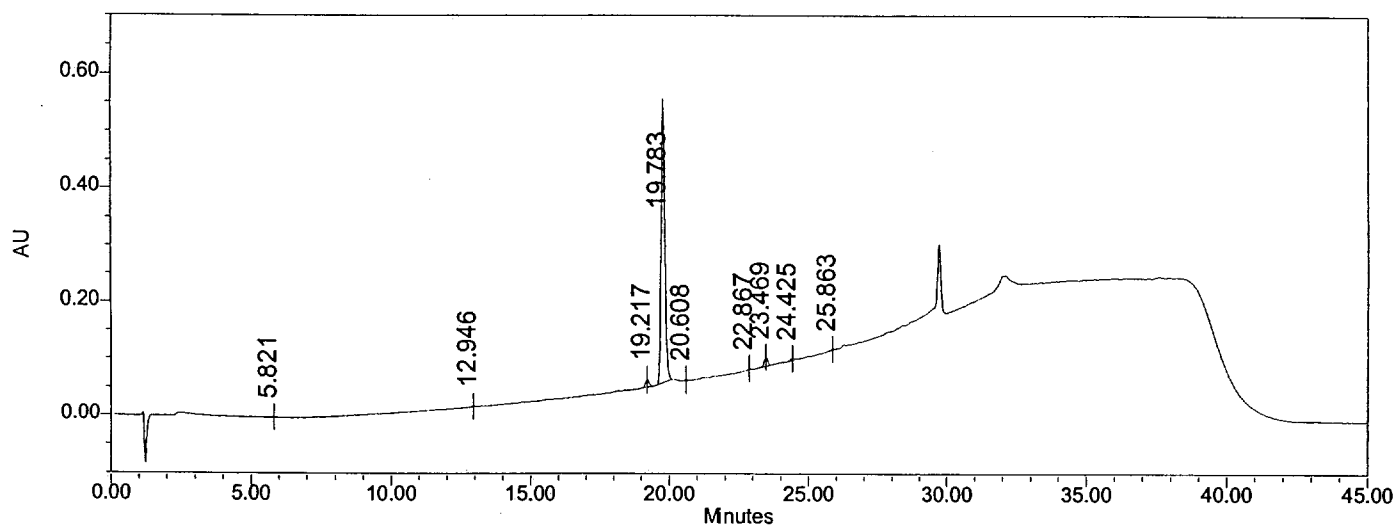
Dimension: 150mm x 3.9 mm id

Flow: 1 mL/min

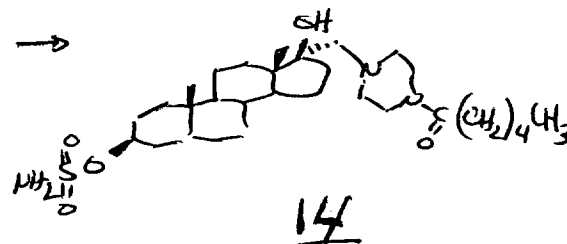
HPLC conditions: Time	A	B
0	50	50
25	100	0
32	100	0
35	50	50
45	50	50

A: H₂O + AcNH₄ 10mMB: MeOH + AcNH₄ 10mM

Channel Name 205nm

Chromatogram**Peak Results**

RT	Area	% Area	Purity1 Angle	Purity1 Threshold
1 5.82	9446	0.19	25.993	48.556
2 12.95	11279	0.23	8.235	12.532
3 19.22	114497	2.33	2.769	3.179
4 19.78	4542300	92.58	0.351	0.297
5 20.61	7653	0.16		
6 22.87	21468	0.44	14.817	15.391
7 23.47	133446	2.72	1.541	1.974
8 24.43	42049	0.86	5.107	6.681
9 25.86	23984	0.49	10.030	10.285



Selon les normes CLHP: Accepté _____ Refusé _____

Lot de LREM: _____

Analyse

Signature: N. Lafontaine

Date: 15 dec. 98

Responsable CLHP:

Signature: _____

Date: 15 dec. 98

Recherche et Développement

SampleName LCC-232-180-DC
HPLC 3671080399

Vial 1
Injection 1
Injection Volume 20.00 ul

Sample Type Unknown

Run Time 45.0 Minutes

Project Name EM_10

Date Acquired 08/Mar/1999 10:24:19

Acq Method Set EM2

Processing Method EM_1

Date Processed 09/Mar/1999 09:08:56

Column: Nova Pak C18, #Lot: T80791P 220

Column T: 25°C

Dimension: 150mm x 3.9 mm id

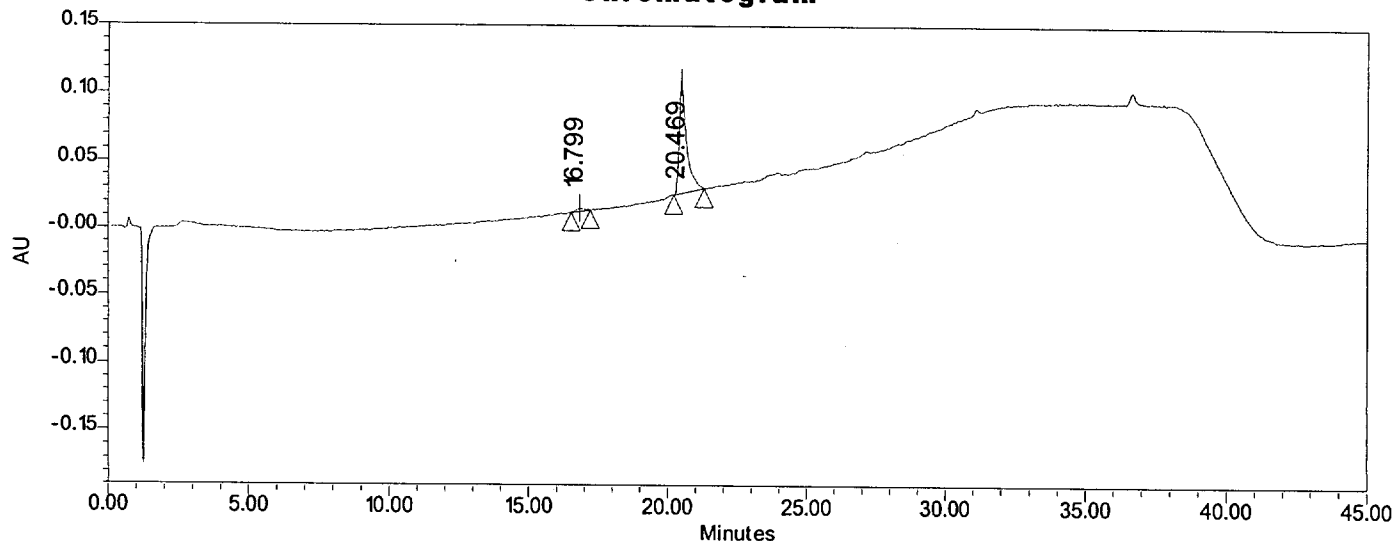
Flow: 1 mL/min

HPLC conditions:	Time	A	B
	0	50	50
	25	100	0
	32	100	0
	35	50	50
	45	50	50

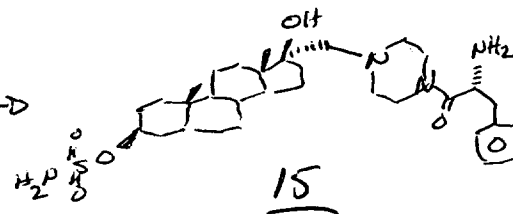
A: MeOH:H2O (9:1) + AcNH4 20mM

B: H2O + AcNH4 20mM

Channel Name 205nm

Chromatogram**Peak Results**

	RT	Area	%Area	Purity1 Angle	Purity1 Threshold
1	16.80	49638	3.18	10.584	19.402
2	20.47	1510452	96.82	0.782	1.971



Selon les normes HPLC: Accepté: _____ Refusé: _____ #Lot du LREM _____

Analyste

Signature: *Nha/ontaine*

Date: 09 mars 99

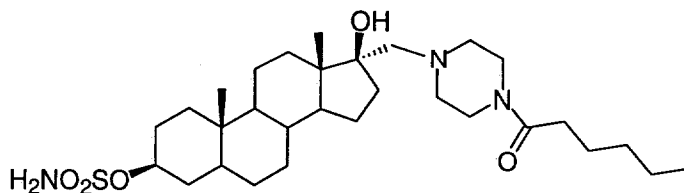
Responsable HPLC

Signature: *[Signature]*

Date: 09 mars 99

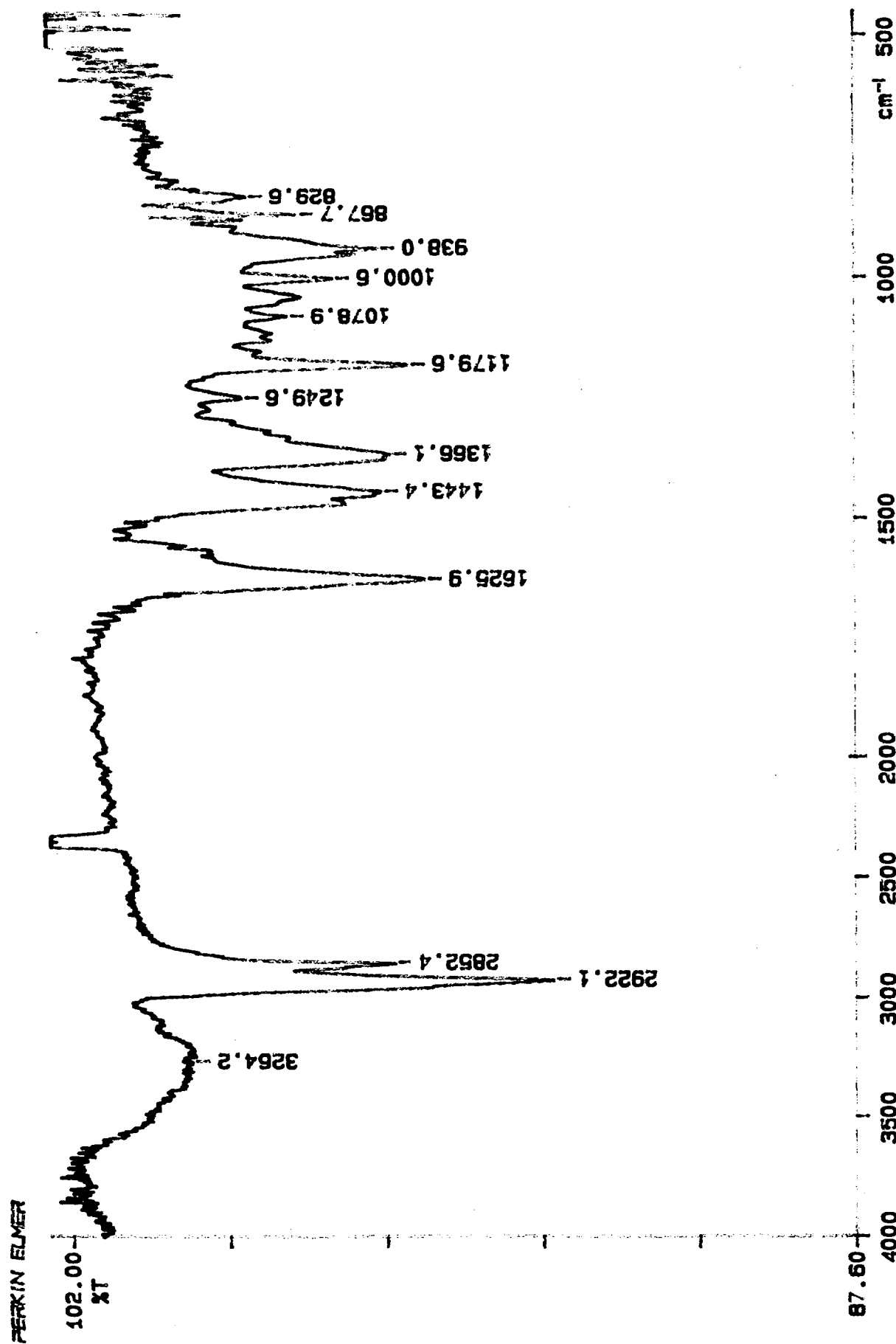
Spectroscopic data of

3 β -Sulfamoyloxy-17 α -[(4'-hexanoyl-piperazine)methyl]-androstan-17 β -ol (14)



White solid; IR ν (film) 3264 (broad, NH_2 and OH), 1626 (CON), 1366 and 1180 (S=O); LR-MS for $\text{C}_{30}\text{H}_{54}\text{N}_3\text{O}_5\text{S}$ (MH^+) 568.6; HPLC purity of 93% (C18-NovaPak column) with a mixture of $\text{H}_2\text{O}:\text{CH}_3\text{OH}$ (50:50) containing AcNH_4 10 mM; ^1H NMR δ ($\text{MeOH}-d_4$) 0.85 and 0.87 (2s, CH_3 -18 and CH_3 -19), 0.92 (t, $J = 6.7$ Hz, CH_2CH_3), 2.37 (t, $J = 7.6$ Hz, CH_2CO), 2.40 (4H), 2.70 (1H) and 2.80 (1H) (3m, 3 x CH_2N), 3.55 (m, 2 x CH_2NCO), 4.40 (m, CH-3 α); ^{13}C NMR δ ($\text{MeOH}-d_4$) 12.57, 14.29, 14.97, 21.91, 23.50, 24.65, 26.35, 29.49, 29.68, 32.05, 32.67, 32.98, 33.42, 33.98, 35.99, 36.50, 37.71, 38.08, 43.02, 46.22, 47.14, 47.72, 52.06, 55.59, 56.08, 56.56, 64.78, 82.32, 84.80, 174.11

IR of 14 (free amine)



99/03/25 19:05 test
Y: 16 scans, 4.0cm-1, flat
LCC232-135, NaCl

14

Résultats d'Analyse en Spectrométrie de Masse à l'UCA

Date d'analyse: 31 mai 99 #Échantillon: LCC-232-135
Chimiste: Ciobanu Liviu #ASM: 056-310399

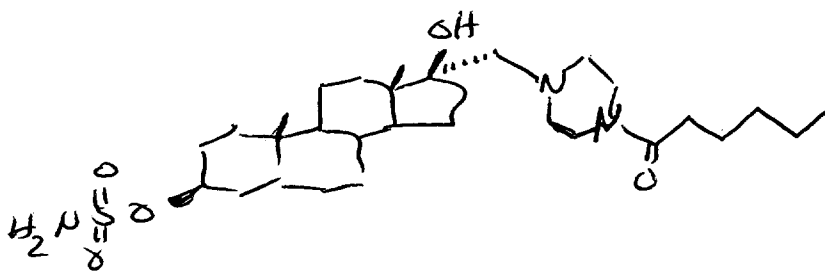
Solvants : MeOH + NH₄CO₂H

Observations particulières:

En positif = $[M+H]^+ \rightarrow 568$

En négatif = $[M-H]^- \rightarrow 566$

$[M+H(CO_2)]^- \rightarrow 612$



14 (free amine)

Effectué par: [Signature]

Date: 31 mai 99

Belong 31 mai 99

page 1 of 1

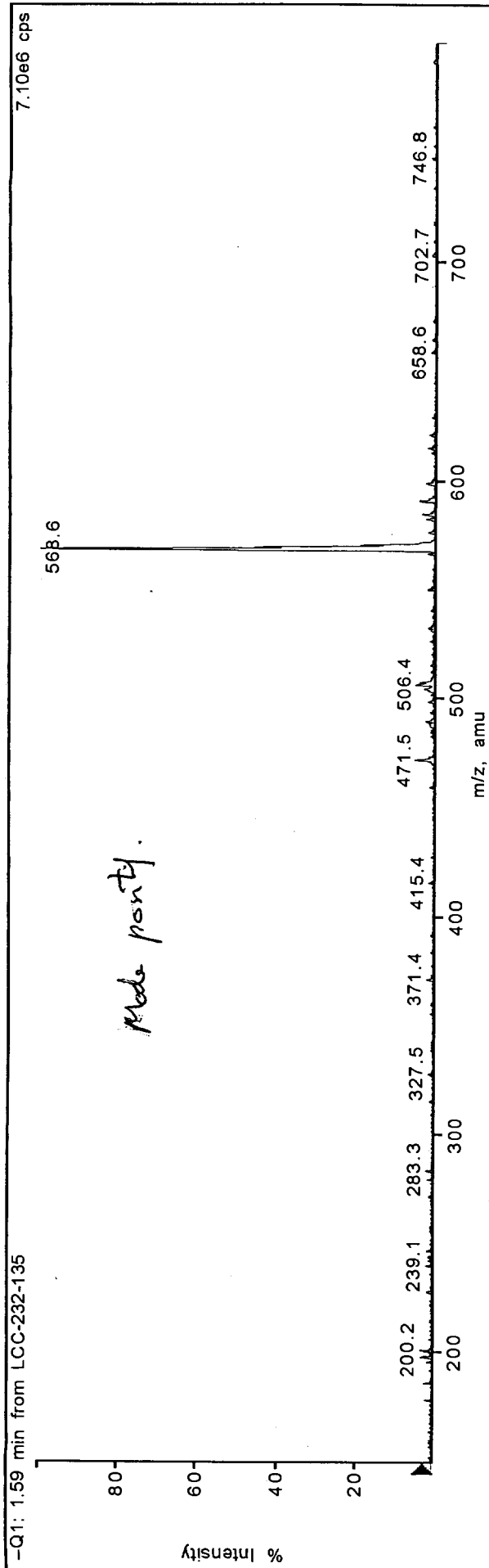
March 31, 1999 10:31

MultiView 1.3

Info for pane 2: LCC-232-135 (No Title)

Period 1, Expt. 1; Mass range: 150.0 to 800.0 by 0.2 amu; Dwell: 0.4 ms; Pause: 5.0 ms

Acq. Time: Mar 31, 1999 at 10:27:44; Exp. Comment: EM Methode



-Q1: 1.59 min from LCC-232-135, centroided

Point num.	m/z (amu)	Intensity (cps)	% Max. Int.	Width (amu)
397	568.6	3.852e7	100.00	1.80
398	569.7	1.136e7	29.48	0.80
399	570.7	4.290e6	11.14	0.80
315	471.5	1.608e6	4.17	1.20
412	590.4	1.398e6	3.63	1.80
348	506.4	1.065e6	2.76	0.60
400	571.7	1.062e6	2.76	1.00
349	507.7	9.825e5	2.55	0.80
347	504.6	9.525e5	2.47	1.20
408	584.5	9.275e5	2.41	1.20
419	598.4	8.525e5	2.21	1.40
44	197.3	8.400e5	2.18	2.00
47	200.3	6.975e5	1.81	1.00
333	489.8	6.900e5	1.79	1.00
407	582.3	6.775e5	1.76	1.60
403	576.4	6.475e5	1.68	2.00
215	371.3	6.000e5	1.56	1.60
204	550.5	5.550e5	1.44	1.40

3.85e7 cps

page 1 of 1

7.78e5 cps

MS of 14

March 31, 1999 10:30

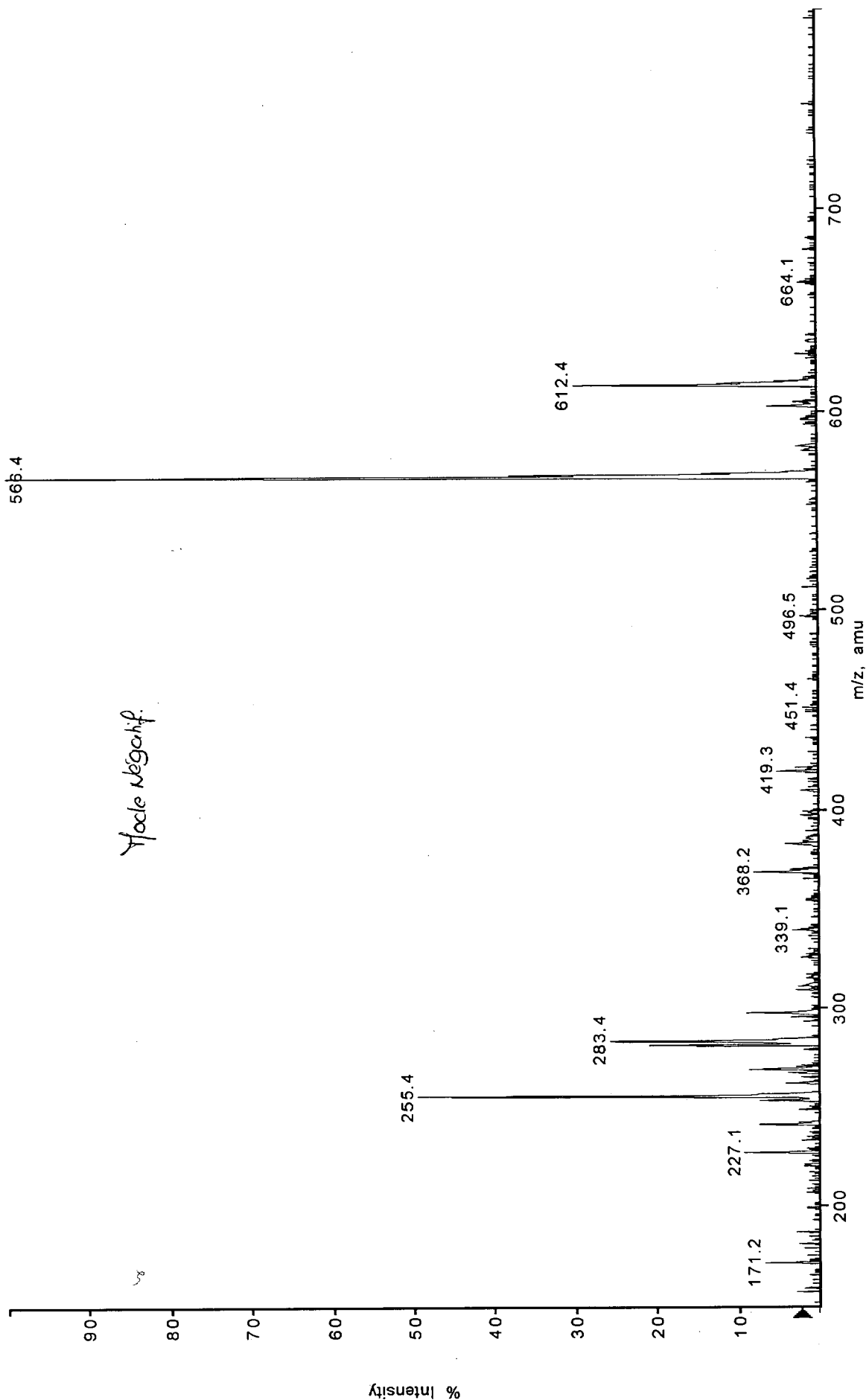
MultiView 1.3

LCC-232-135 (No Title)

Period 1, Expt 1; Mass range: 150.0 to 800.0 by 0.2 amu; Dwell: 0.4 ms; Pause: 5.0 ms

Acq. Time: Mar 31, 1999 at 10:27:44; Exp. Comment: EM Methode

-Q1: 2.42 min from LCC-232-135



31mar99

MIN. INTENSITY = 2.541 MAXY = 20.00000
INTENS. LEVEL = 2.541 NOISE = .01349
F1 = 2761.41 HZ = 9.2006 PPM F2 = -239

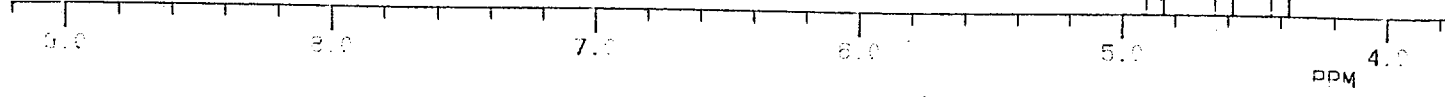
Compd 14

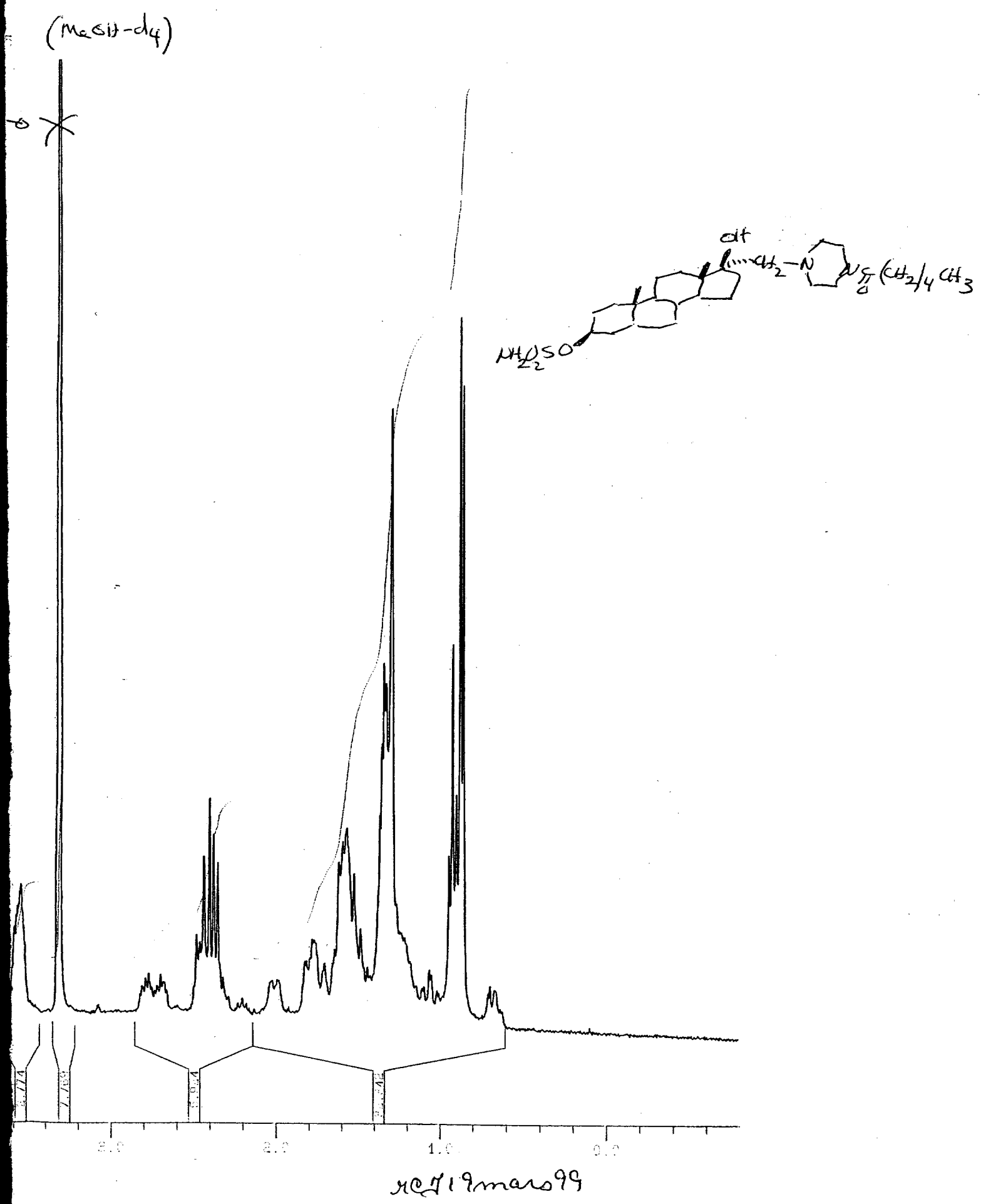
#	CURSOR	FREQUENCY	PPM	INTENSITY
1	4698	1467.586	4.8898	148.360
2	5435	1062.225	3.5392	2.592
3	5557	994.995	3.3152	14.727
4	5560	993.465	3.3101	26.989
5	5563	991.912	3.3049	36.123
6	5566	990.360	3.2997	26.026
7	5569	988.833	3.2946	13.694
8	6043	728.282	2.4265	3.238
9	6061	718.241	2.3931	4.460
10	6074	710.856	2.3685	3.681
11	6088	703.155	2.3428	3.068
12	6489	482.944	1.6091	3.096
13	6501	476.055	1.5861	3.523
14	6515	468.578	1.5612	3.804
15	6539	455.370	1.5172	2.839
16	6625	407.928	1.3592	4.079
17	6632	404.296	1.3471	5.608
18	6638	400.815	1.3355	7.281
19	6645	396.914	1.3225	6.854
20	6665	386.194	1.2867	12.797
21	6853	282.482	.9412	3.246
22	6866	275.687	.9185	7.938
23	6878	268.990	.8962	4.542
24	6891	261.749	.8721	14.617
25	6901	256.368	.8542	13.350

X solvent

2.

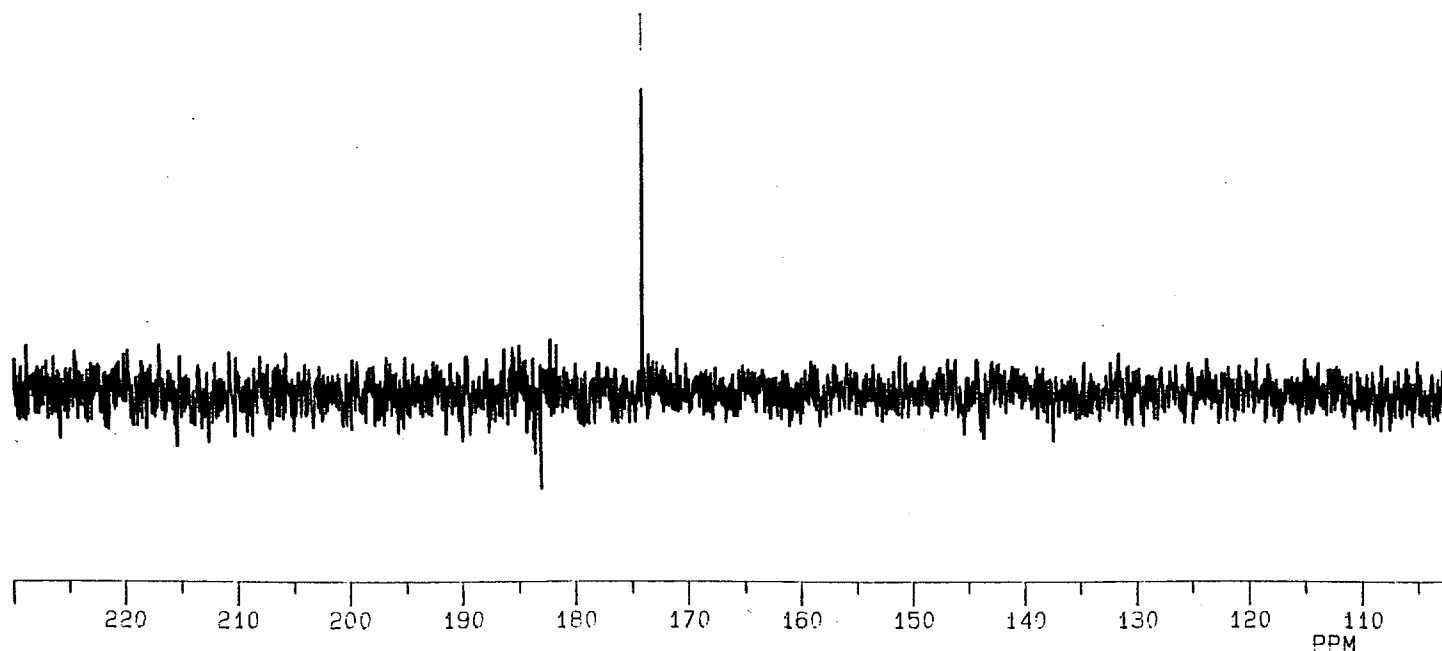
INTEGRAL





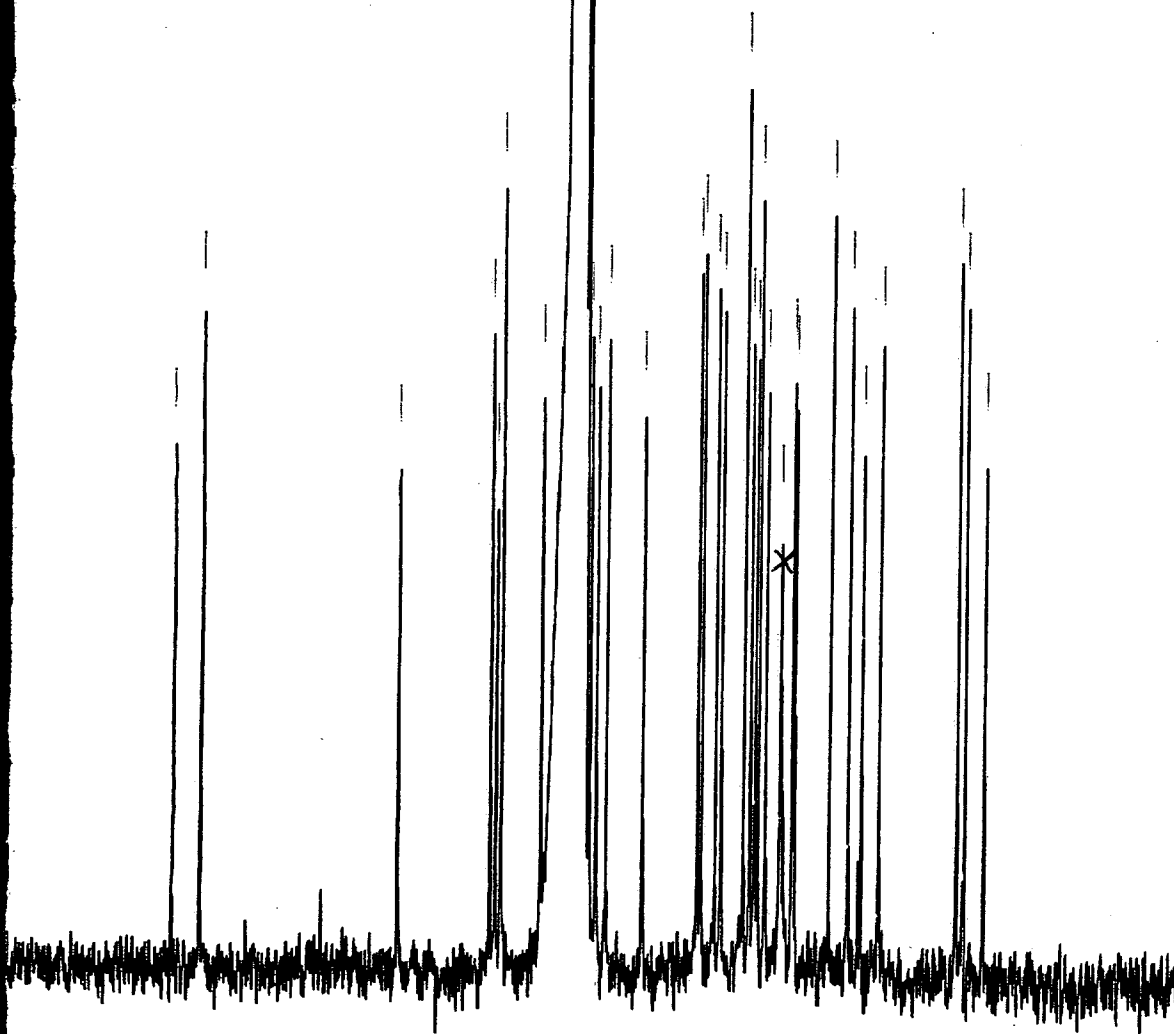
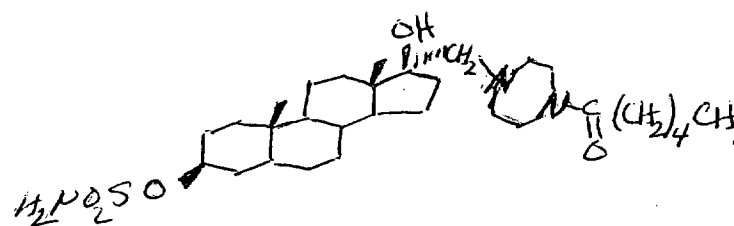
1	2056	13139.642	174.1070	4.055
2	4927	6399.890	84.8018	6.930
3	5007	6212.417	82.3177	8.751
4	5571	4888.598	64.7764	6.764
5	5835	4268.424	56.5588	8.441
6	5851	4231.910	56.0750	6.537
7	5866	4195.678	55.5949	10.387
8	5980	3928.640	52.0565	7.850
9	6051	3761.407	49.8406	119.065
10	6060	3740.247	49.5602	344.118
11	6069	3719.049	49.2793	638.914
12	6078	3697.762	48.9972	636.384
13	6087	3676.273	48.7125	449.249
14	6097	3654.198	48.4200	215.840
15	6106	3632.590	48.1337	87.622
16	6119	3601.686	47.7242	8.425
17	6138	3557.442	47.1379	7.847
18	6167	3488.466	46.2240	8.641
19	6270	3246.806	43.0218	7.502
20	6429	2873.931	38.0811	9.290
21	6441	2846.064	37.7118	9.609
22	6480	2754.291	36.4958	9.083
23	6496	2716.470	35.9946	8.846
24	6561	2564.156	33.9764	11.741
25	6579	2521.964	33.4173	8.366
26	6593	2488.758	32.9773	8.203
27	6603	2465.477	32.6688	10.264
28	6623	2418.939	32.0522	7.825
29	6664	2321.240	30.7576	6.032
30	6699	2239.744	29.6778	7.978
31	6705	2225.386	29.4875	7.742
32	6806	1988.890	26.3538	10.095
33	6861	1860.018	24.6462	8.887
34	6898	1773.492	23.4997	7.090
35	6949	1653.530	21.9101	8.411
36	7172	1129.768	14.9700	9.477
37	7194	1078.272	14.2877	8.891
38	7249	948.366	12.5663	7.040

Compd 14



84.502	64.176
82.318	56.555
	56.073
	55.591
	52.056
	49.841
	49.569
	48.279
	48.097
	48.712
	48.420
	47.724
	47.138
	46.224
	43.022
	39.031
	37.712
	36.496
	35.995
	33.977
	33.417
	33.977
	32.663
	32.032
	30.733
	29.673
	29.485
	26.554
	24.646
	23.502
	21.916
	14.971
	14.583
	12.566

Solvent →

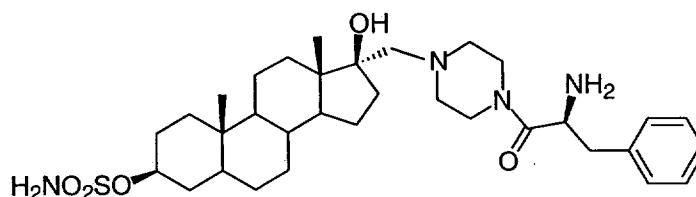


0 50 80 70 60 50 40 30 20 10 0

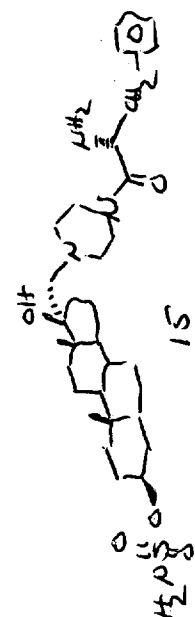
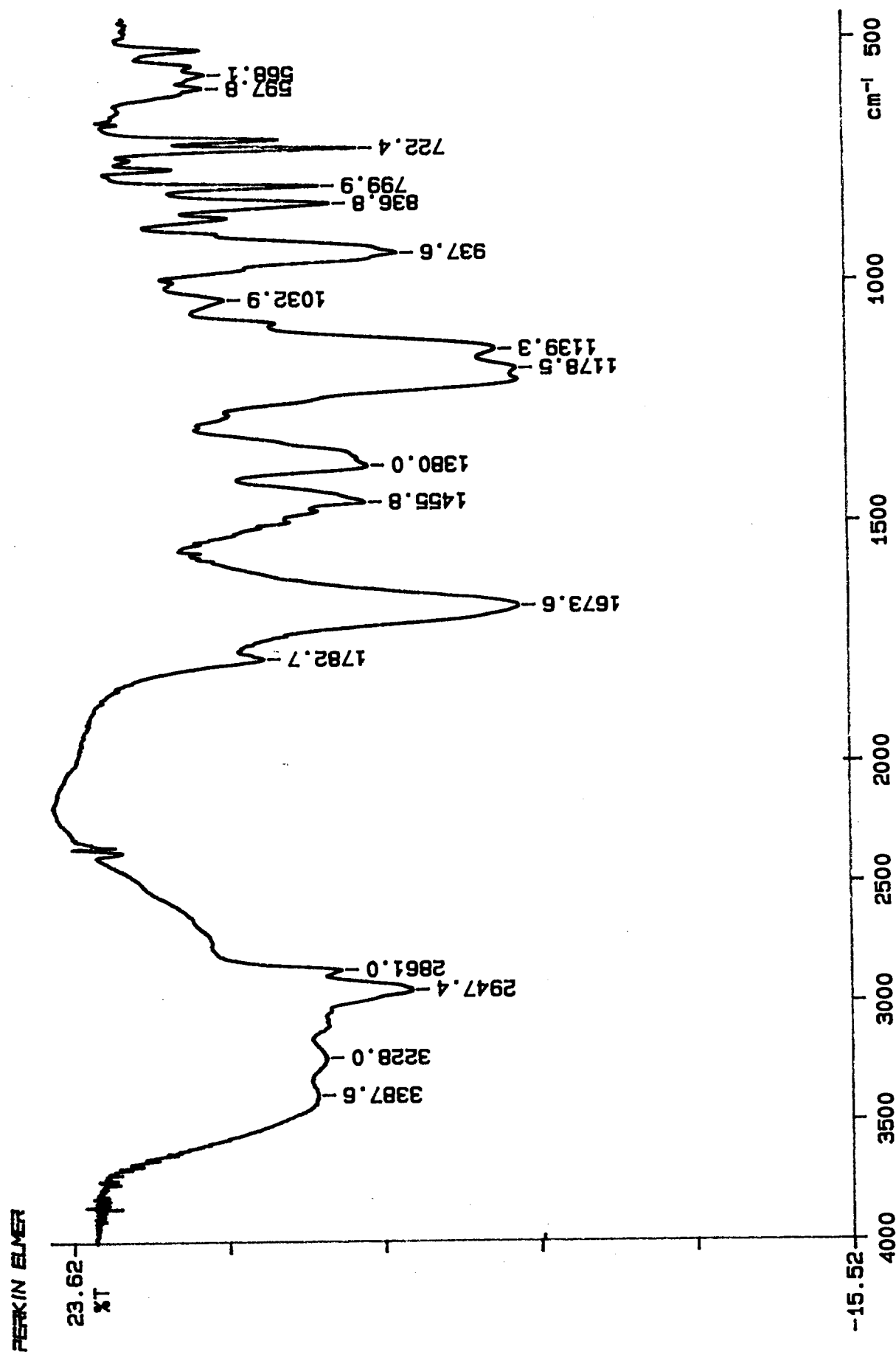
XC722 mars99

Spectroscopic data of

3 β -Sulfamoyloxy-17 α -{[4'-(2''-(*S*)-benzyl-2''-ammonium-acetoxy)piperazine] methyl}-androstan-17 β -ol (15)



White solid; IR ν (KBr) 3388, 3228 (broad, NH_2 and OH), 1674 (CON), 1366 and 1180 ($\text{S}=\text{O}$); LR-MS for $\text{C}_{33}\text{H}_{53}\text{N}_4\text{O}_5\text{S}$ (MH^+) 617.4; HPLC purity of 97% (C18-NovaPak column) with a mixture of $\text{H}_2\text{O}:\text{CH}_3\text{OH}$ (50:50) containing AcNH_4 20 mM; ^1H NMR δ ($\text{MeOH}-d_4$) 0.83 and 0.86 (2s, CH_3 -18 and CH_3 -19), 2.1-3.1 (multiple signals, CH_2N and CH_2Ph), 3.50 (m, CH_2N), 4.06 (t, $J = 7.0$ Hz, CHN), 4.40 (m, CH-3 α), 7.1-7.3 (m, 5H, Phenyl); ^{13}C NMR δ ($\text{MeOH}-d_4$) 12.58, 14.94, 21.91, 24.62, 29.52, 29.68, 32.05, 32.96, 33.48, 36.03, 36.49, 37.71, 38.09, 43.32, 43.62, 46.22, 46.67, 47.64, 52.05, 52.55, 55.60, 55.74, 55.88, 64.62, 82.16, 84.74, 127.95, 129.66 (2x), 130.56 (2x), 138.46, 174.57.



99/03/10 11:24 test
X: 16 scans, 4.0cm⁻¹, flat
LCC232-180DC, KBr

IR of 15

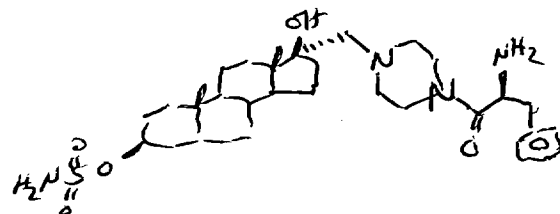
[1413] 4 73

Vérification de la masse moléculaire par infusion (ESI)

Name of Compound: _____

Reference No : _____

LREM Lot Number : _____

Supplier Batch No. : 1CC-232-1808CNo. ASM : 819-191199

Les spectres de masse ont été obtenus à l'aide d'un appareil API-150ex muni d'une source « turbo ionspray ». Le composé a été dissous dans le méthanol avec une petite quantité de formate d'ammonium pour favoriser l'ionisation.

Masse exacte théorique de la molécule « **M** » proposée: 616,37Masse de la molécule « **A** » supposée : _____

Masse(s) obtenue (s):

Mode positif : $m/z =$ 617,4 $\Rightarrow$ $[M+H]^+$ $m/z =$ _____ $\Rightarrow$ _____Mode négatif : $m/z =$ 615,3 $\Rightarrow$ $[M-H]^-$ $m/z =$ _____ $\Rightarrow$ _____

Confirmation de la masse :

Oui ☒Non ☐

Commentaires : _____

Date d'analyse : 19 Nov 99

Analyste : _____

Date : 19 Nov 99

Vérifié par : _____

Date : 19 nov. 99

MS of 15

page 1 of 1

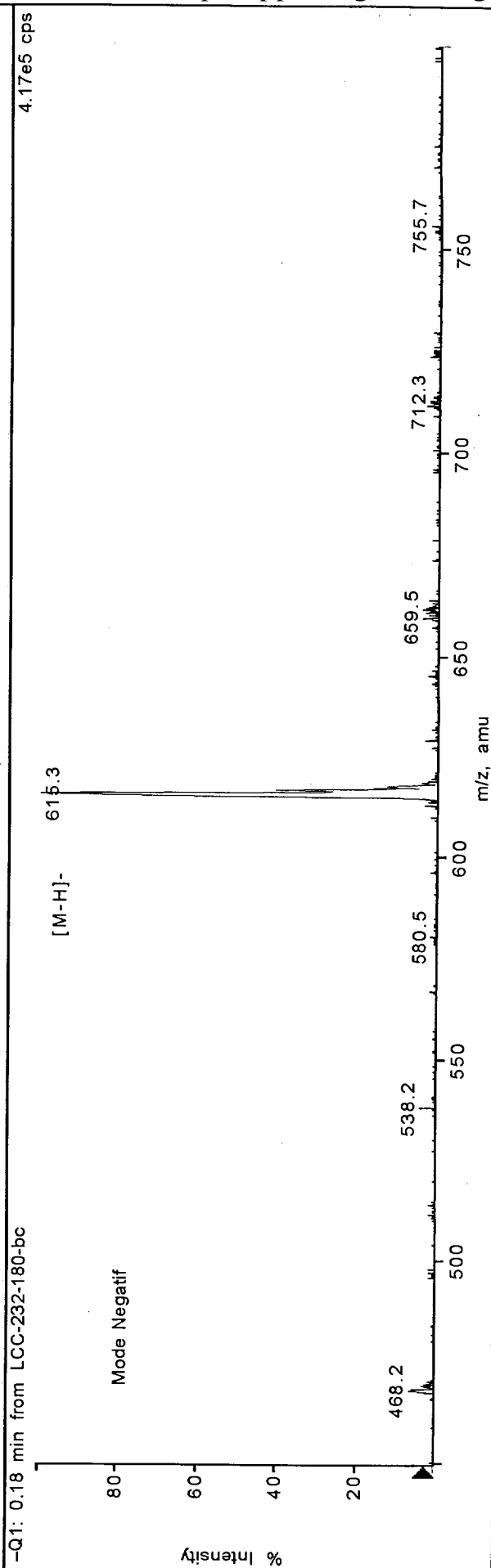
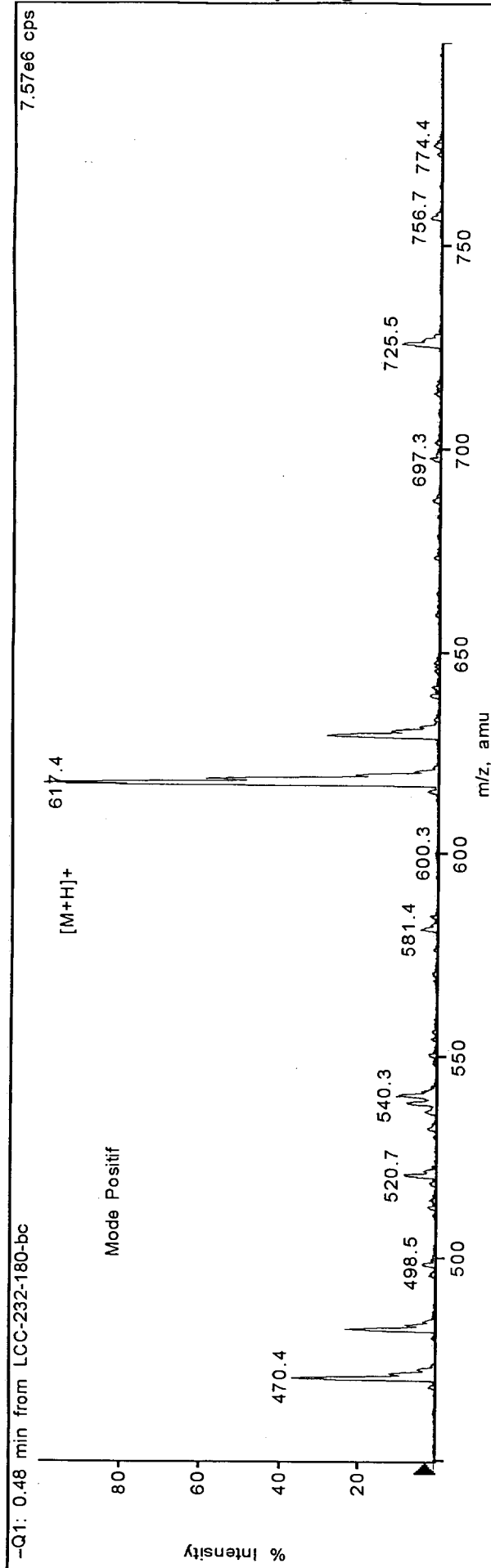
November 19, 1999 11:23

MultiView 1.3

Info for pane 2: LCC-232-180-bc (No Title)

Period 1, Expt. 1; Mass range: 450.0 to 800.0 by 0.1 amu; Dwell: 0.3 ms; Pause: 5.0 ms

Acq. Time: Nov 19, 1999 at 11:20:03; Exp. Comment: EM Methode



TA 19 Nov 99

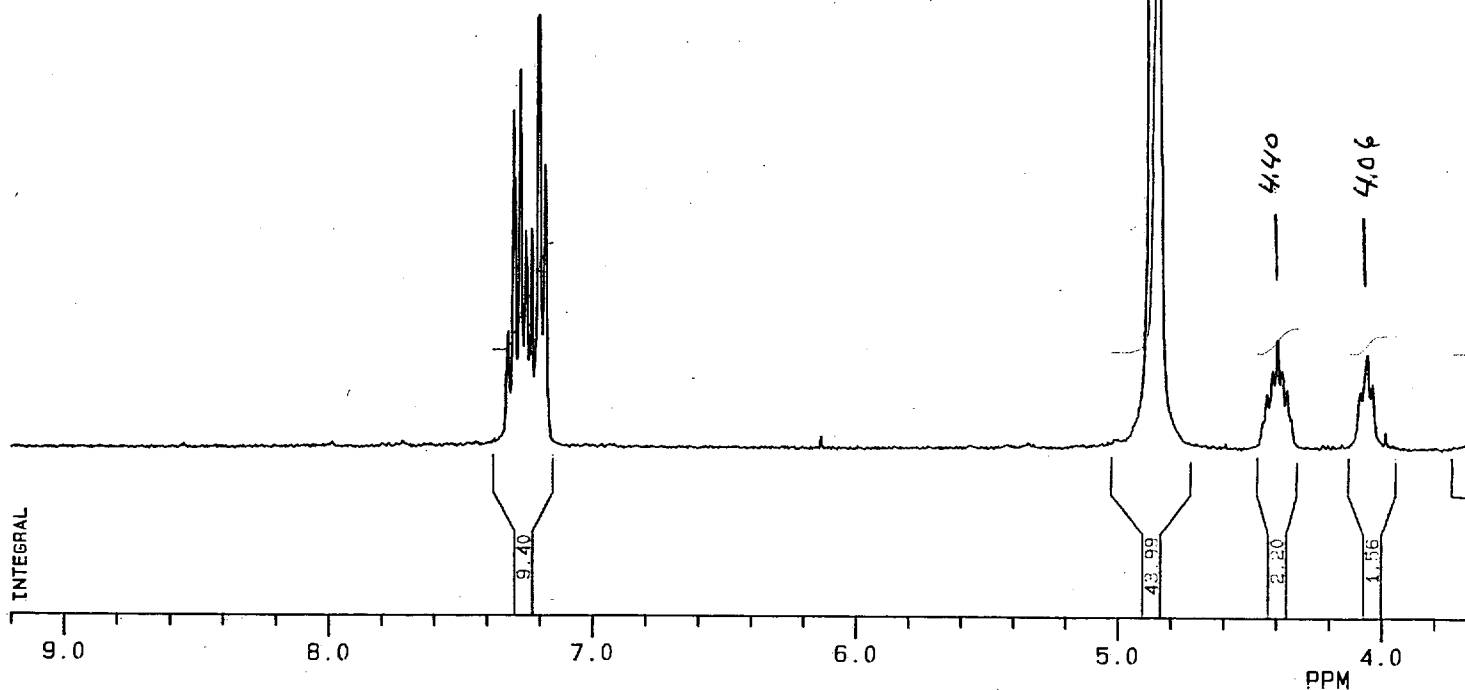
Compd 15

LCC-232-180

LCC23218.0DH=F1

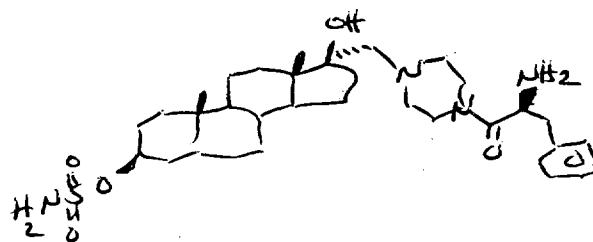
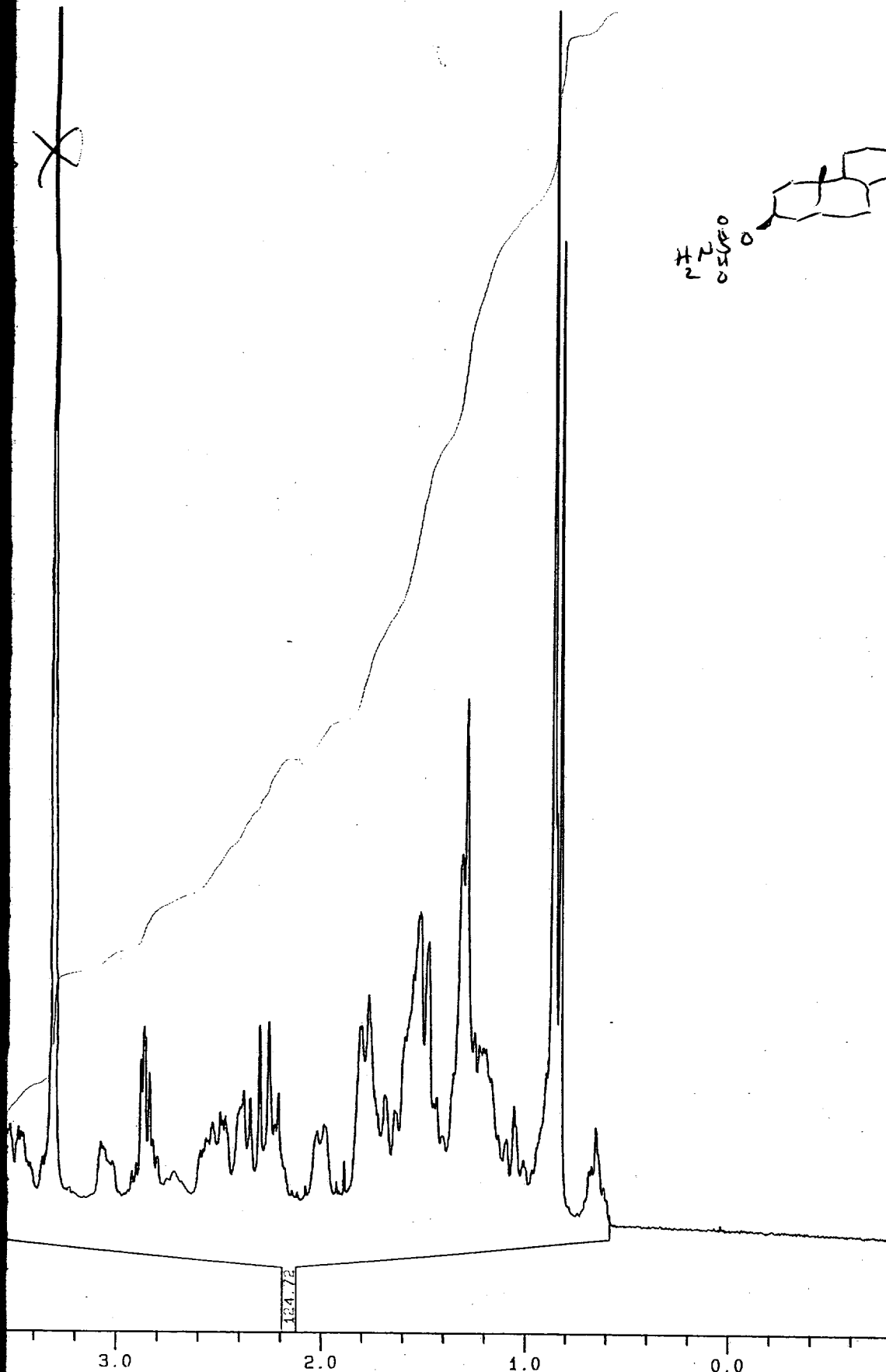
MIN. INTENSITY = 2.574 MAXY = 20.00000 PP
INTENS. LEVEL = 2.574 NOISE = .00771 SE
F1 = 2761.41 HZ = 9.2006 PPM F2 = -239.7

#	CURSOR	FREQUENCY	PPM	INTENSITY
1	3385	2189.450	7.2949	4.208
2	3398	2182.132	7.2705	4.903
3	3410	2175.842	7.2496	2.576
4	3422	2169.086	7.2270	2.631
5	3435	2161.870	7.2030	5.504
6	3438	2160.514	7.1985	5.468
7	3450	2153.834	7.1762	3.470
8	4708	1462.293	4.8721	55.463
9	5557	995.136	3.3156	10.832
10	5560	993.526	3.3103	20.895
11	5563	991.915	3.3049	29.455
12	5566	990.304	3.2995	21.424
13	5569	988.704	3.2942	11.809
14	5804	859.457	2.8636	2.693
15	6112	690.296	2.3000	2.771
16	6136	676.923	2.2554	2.799
17	6385	539.875	1.7988	2.736
18	6403	529.826	1.7653	3.264
19	6523	464.087	1.5463	3.624
20	6541	454.110	1.5130	4.679
21	6564	441.690	1.4716	4.187
22	6653	392.752	1.3086	5.679
23	6665	385.999	1.2861	8.329
24	6686	374.265	1.2470	2.641
25	6897	258.547	.8614	20.250
26	6904	254.791	.8489	6.489
27	6916	248.216	.8270	16.615



$^1\text{H NMR}$ of 15

(in $\text{Me}_2\text{SO}-d_6$)



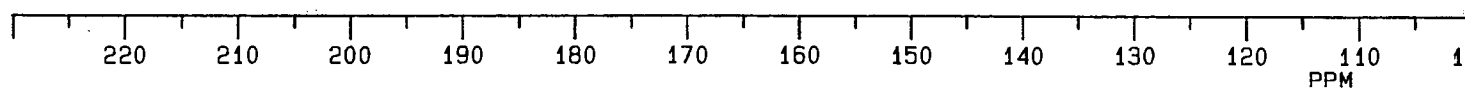
10720 April 99

INTENS. LEVEL = 1.366 NOISE = .30247
F1 = 17358.11 HZ = 230.0038 PPM F2 = -31

LCC-232-180

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2	3202	10449.472	138.4608	2.649
3	3456	9853.319	130.5615	10.138
4	3485	9785.242	129.6595	9.625
5	3540	9656.281	127.9507	4.284
6	4929	6395.378	84.7420	3.810
7	5012	6200.183	82.1556	5.695
8	5576	4876.506	64.6162	3.956
9	5857	4217.207	55.8802	3.729
10	5861	4206.637	55.7401	3.783
11	5866	4195.660	55.5946	6.313
12	5964	3965.955	52.5509	3.149
13	5980	3927.810	52.0455	4.861
14	6051	3761.772	49.8454	22.224
15	6060	3740.413	49.5624	77.804
16	6069	3719.106	49.2801	182.002
17	6078	3697.908	48.9992	208.795
18	6087	3676.767	48.7190	164.747
19	6096	3655.628	48.4389	76.784
20	6105	3634.375	48.1573	23.664
21	6122	3595.267	47.6391	3.453
22	6153	3522.408	46.6737	3.511
23	6167	3488.209	46.2205	4.959
24	6251	3291.834	43.6185	2.774
25	6261	3268.904	43.3146	3.319
26	6429	2874.360	38.0867	4.878
27	6441	2845.595	37.7056	5.848
28	6480	2754.051	36.4926	5.217
29	6495	2718.837	36.0260	5.473
30	6577	2526.352	33.4755	3.562
31	6594	2487.727	32.9637	4.492
32	6623	2418.771	32.0500	4.745
33	6699	2239.973	29.6808	5.691
34	6704	2227.442	29.5147	4.425
35	6862	1857.769	24.6164	4.460
36	6949	1653.391	21.9083	4.911
37	7173	1127.573	14.9409	5.979
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138.461

130.562
129.659
127.951Cmpd
15

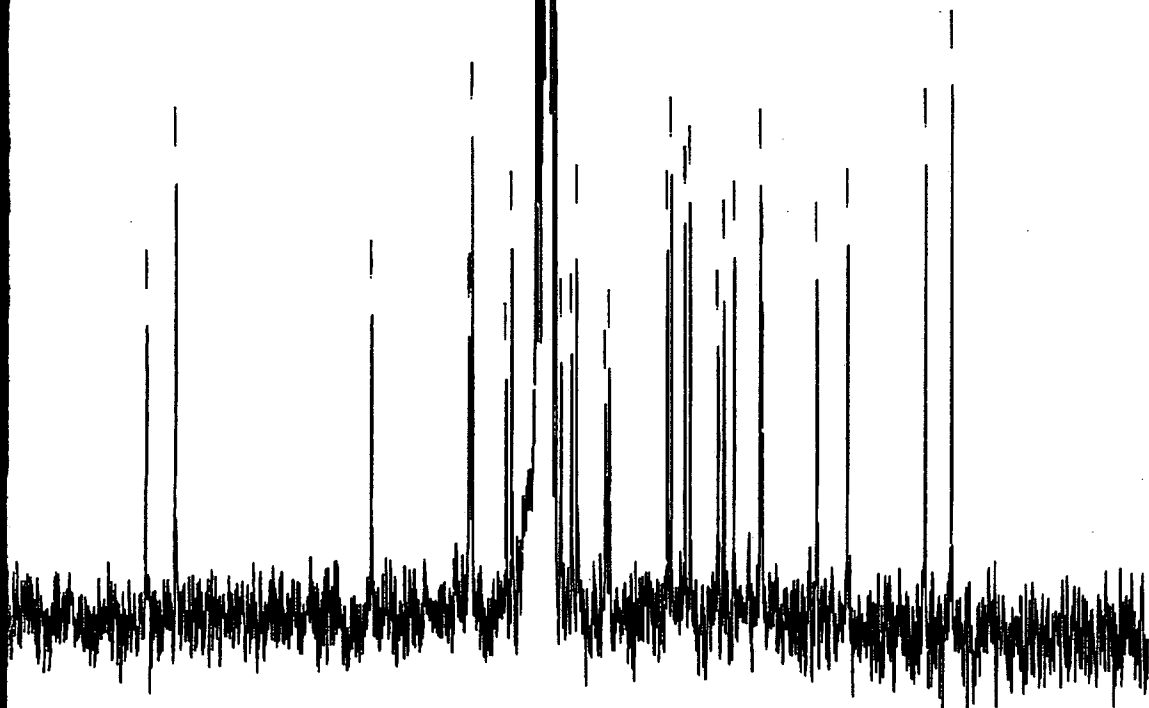
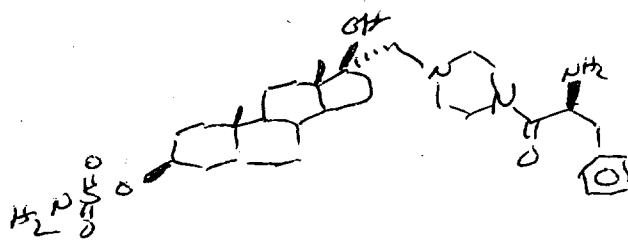
¹³C NMR 67 15

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37.706
36.793
36.028
33.473
32.964
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29.681
29.515
24.616
21.908

14.941
12.575

~~Solvent~~



90 80 70 60 50 40 30 20 10 0

12720 april 99