

Supporting Information

A convenient solid-phase strategy for the synthesis of antimicrobial cyclic lipopeptides

Sílvia Vilà, Esther Badosa, Emilio Montesinos, Lidia Feliu,* and Marta Planas*

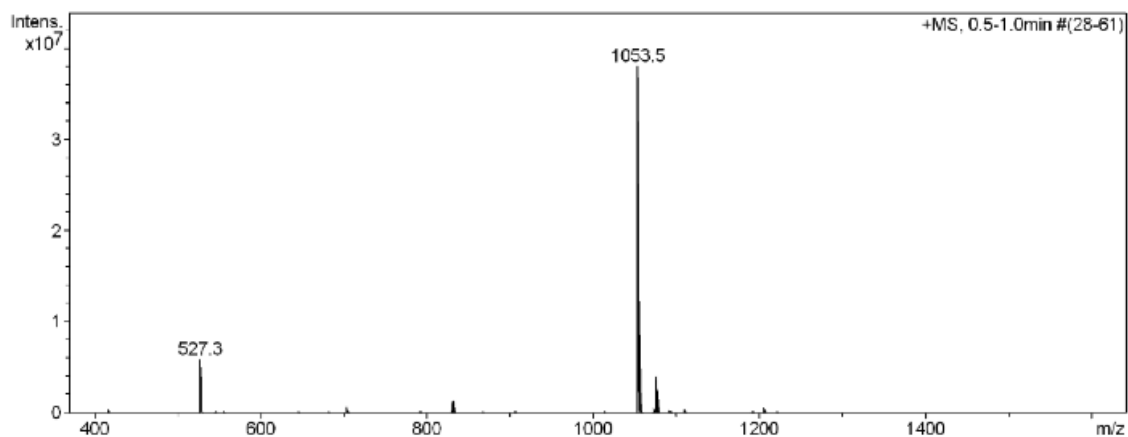
lidia.feliu@udg.edu, marta.planas@udg.edu

Table of contents

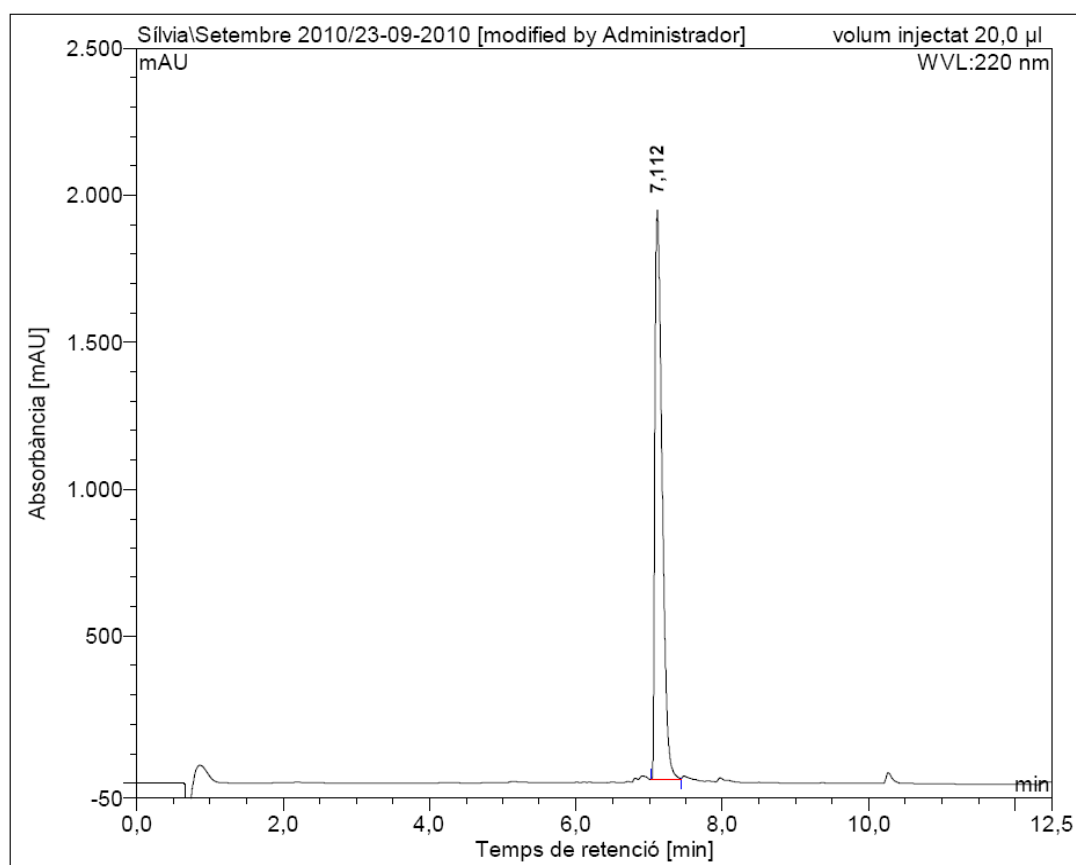
Fmoc-Lys-Phe-Lys-Lys-Leu-Gln-OAll	2
Fmoc-Lys-Lys-Leu-Lys-Lys-Phe-Lys-Lys-Leu-Gln-OAll	3
Fmoc-Lys-Lys-Leu-Lys-Lys(ivDde)-Phe-Lys-Lys-Leu-Gln-OAll.....	4
Fmoc-Lys(COCH ₃)-Phe-Lys-Lys-Leu-Gln-OAll (7a).....	5
Fmoc-Lys(COC ₃ H ₇)-Phe-Lys-Lys-Leu-Gln-OAll (7b)	6
Fmoc-Lys(COC ₇ H ₁₅)-Phe-Lys-Lys-Leu-Gln-OAll (7c)	7
Fmoc-Lys-Lys-Leu-Lys-Lys(COCH ₃)-Phe-Lys-Lys-Leu-Gln-OAll (11a)	8
Fmoc-Lys-Lys-Leu-Lys-Lys(COC ₃ H ₇)-Phe-Lys-Lys-Leu-Gln-OAll (11b)	9
Fmoc-Lys-Lys-Leu-Lys-Lys(COC ₇ H ₁₅)-Phe-Lys-Lys-Leu-Gln-OAll (11c).....	10
c(Lys-Lys-Leu-Lys-Lys-Phe-Lys-Lys-Leu-Gln) (BPC194)	11
c(Lys-Lys-Leu-Lys-Lys(ivDde)-Phe-Lys-Lys-Leu-Gln).....	11
c(Lys-Lys-Leu-Lys-Lys(COCH ₃)-Phe-Lys-Lys-Leu-Gln) (5).....	11
c(Lys-Lys-Leu-Lys-Lys(COC ₇ H ₁₅)-Phe-Lys-Lys-Leu-Gln) (BPC498).....	12
c(Lys-Lys-Leu-Lys-Lys(COC ₄ H ₇)-Phe-Lys-Lys-Leu-Gln) (BPC500)	14
c(Lys-Lys-Leu-Lys-Lys(COC ₁₅ H ₃₁)-Phe-Lys-Lys-Leu-Gln) (BPC502)	16
c(Lys-Lys-Leu-Lys-Lys(COC ₁₁ H ₂₂ OH)-Phe-Lys-Lys-Leu-Gln) (BPC524)	18
c(Lys-Lys-Leu-Lys-Lys(COC ₁₁ H ₂₃)-Phe-Lys-Lys-Leu-Gln) (BPC530)	20

Fmoc-Lys-Phe-Lys-Lys-Leu-Gln-OAll

ESI-MS (m/z)



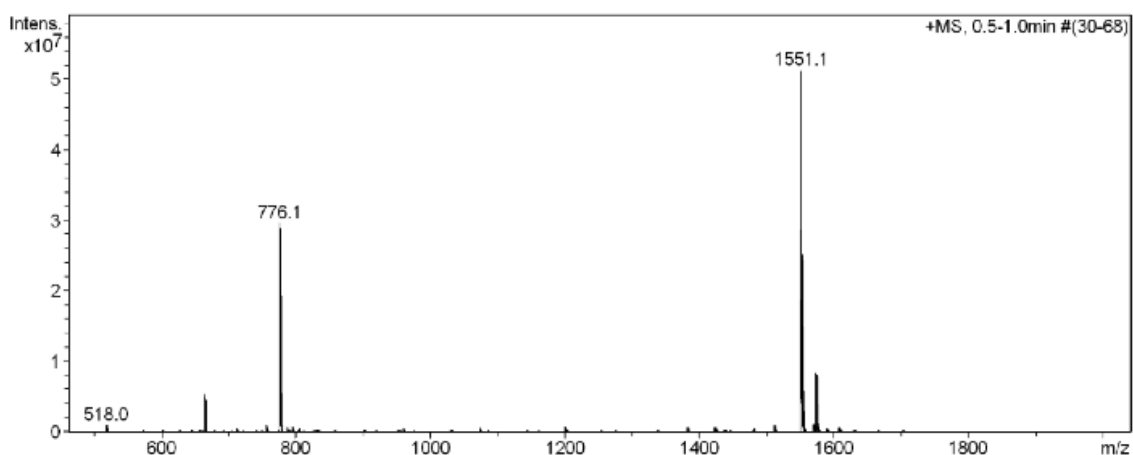
HPLC ($\lambda=220$ nm)



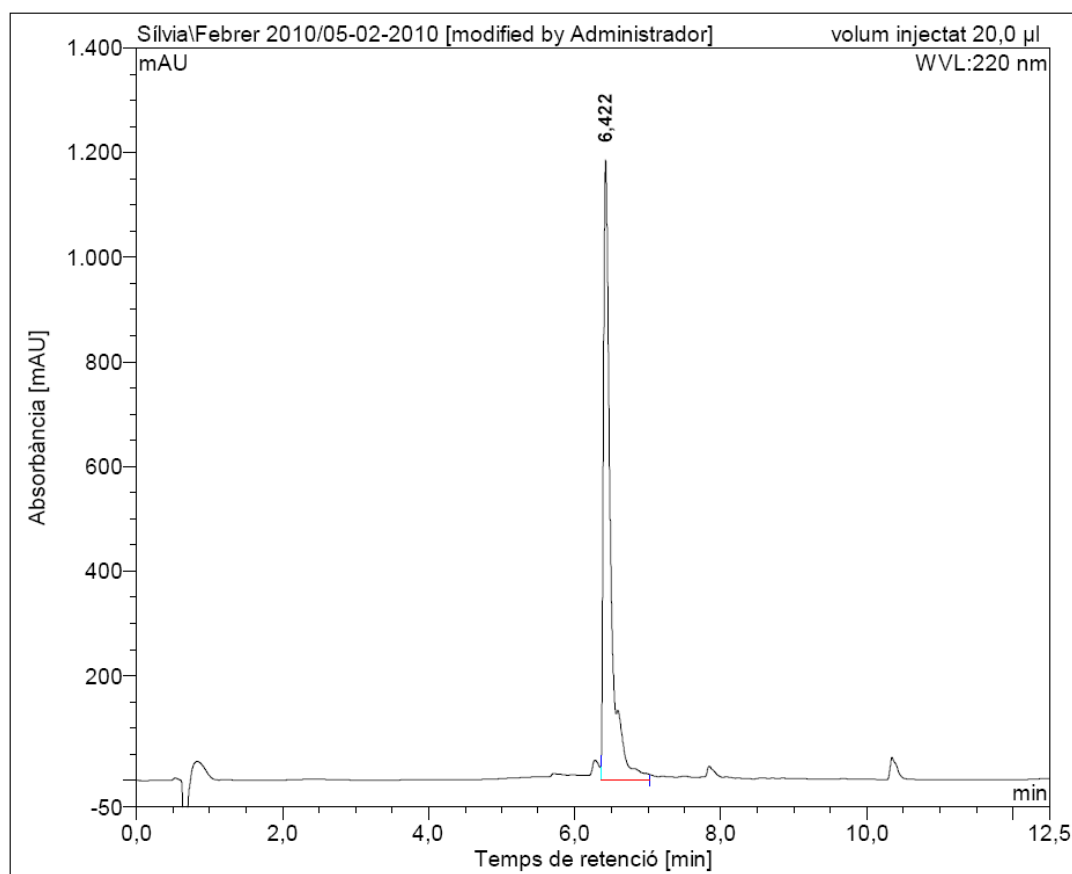
No.	mps retenc min	alçada mAU	Area mAU*min	Area relativa %
1	7,11	1937,784	223,207	100,00
Total:		1937,784	223,207	100,00

Fmoc-Lys-Lys-Leu-Lys-Lys-Phe-Lys-Lys-Leu-Gln-OAll

ESI-MS (m/z)



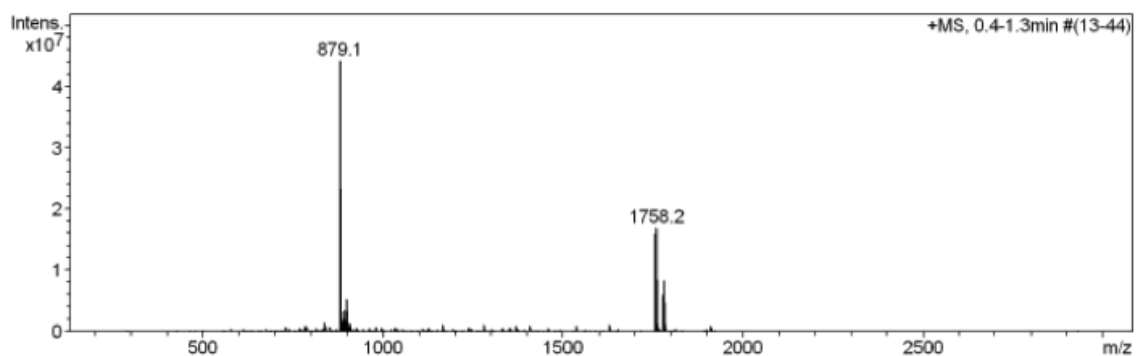
HPLC ($\lambda=220$ nm)



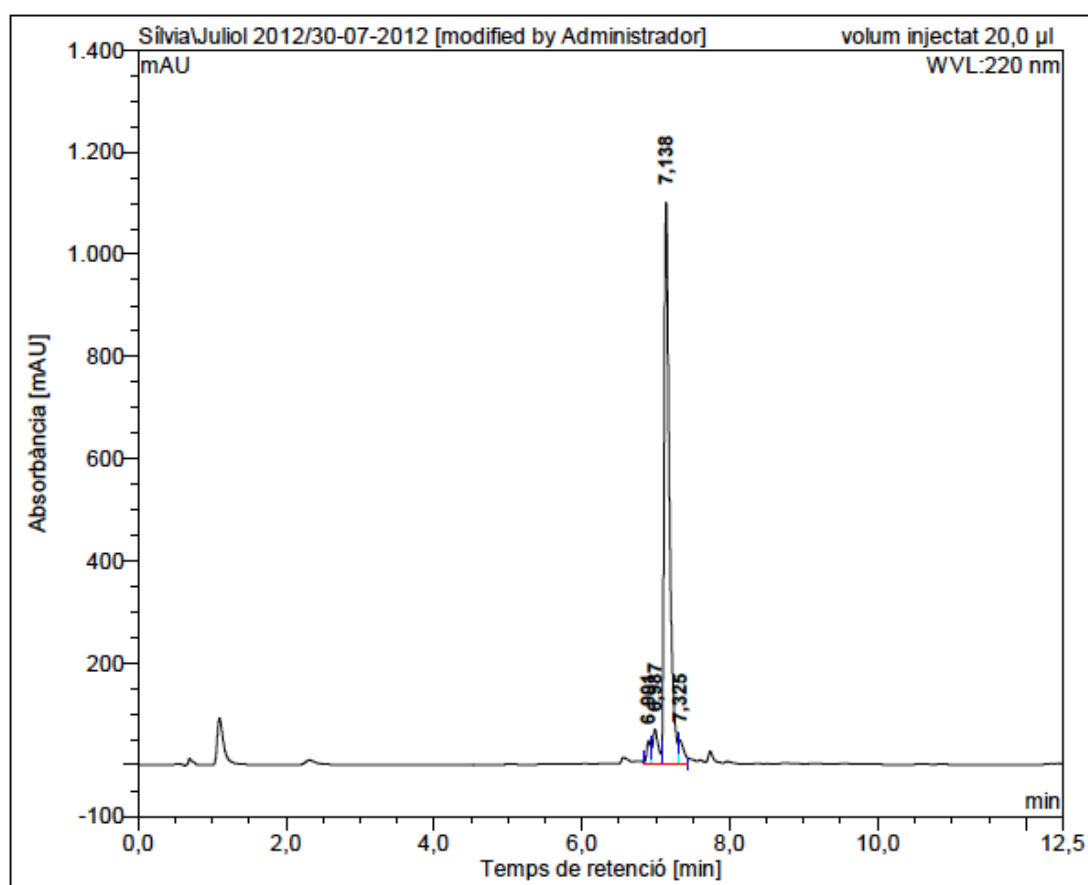
No.	mps retenc min	alçada mAU	Area mAU*min	Area relativa %
1	6,42	1186,004	134,786	100,00
Total:		1186,004	134,786	100,00

Fmoc-Lys-Lys-Leu-Lys-Lys(ivDde)-Phe-Lys-Lys-Leu-Gln-OAll

ESI-MS (m/z)



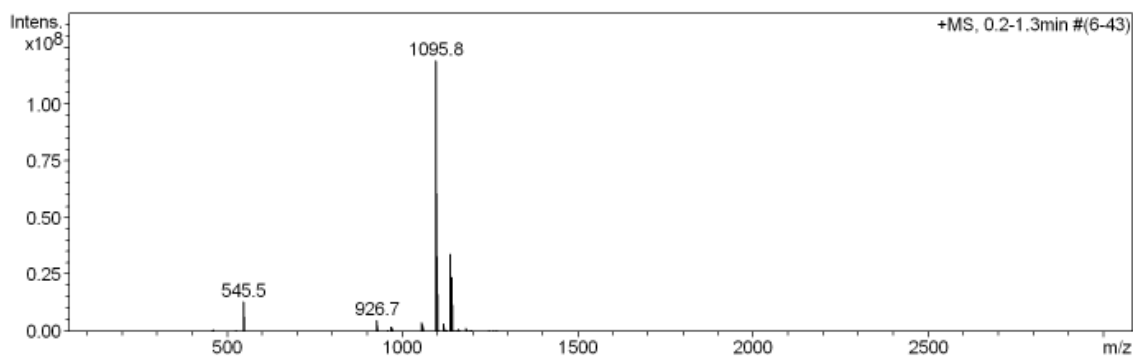
HPLC ($\lambda=220$ nm)



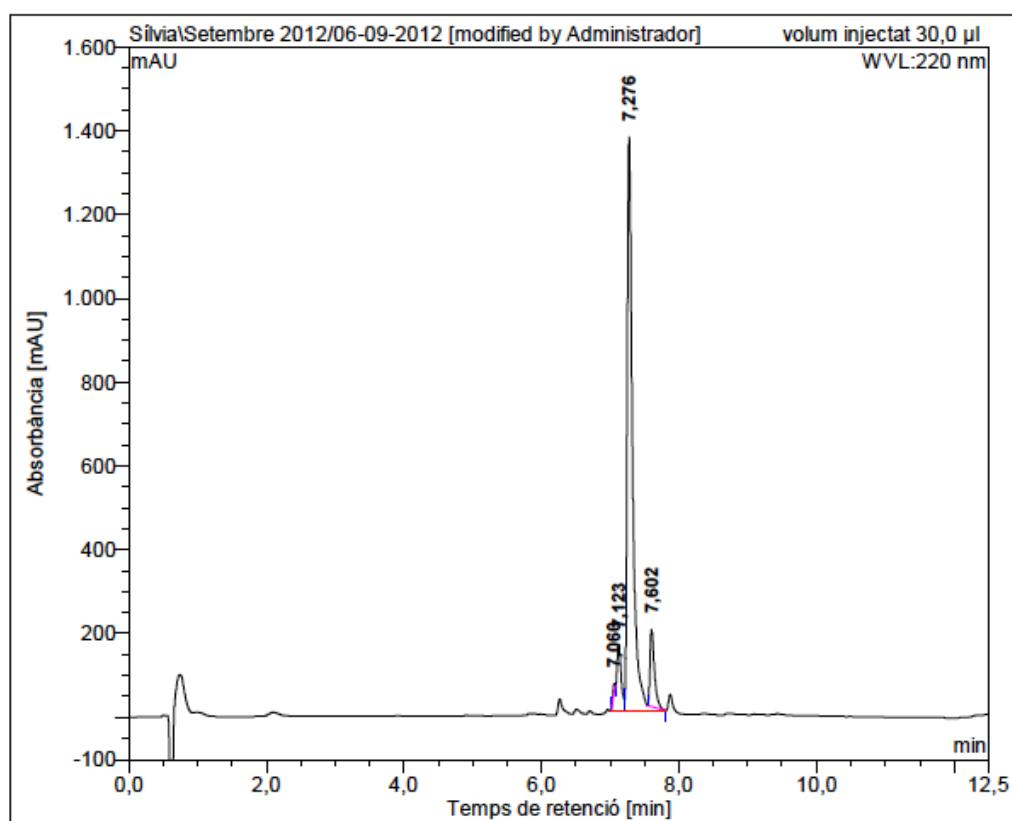
No.	mps retenc min	alçada mAU	Area mAU*min	Area relativa %
1	6,90	44,489	2,715	2,67
2	6,99	66,646	5,448	5,36
3	7,14	1098,328	89,672	88,22
4	7,33	45,526	3,814	3,75
Total:		1254,990	101,650	100,00

Fmoc-Lys(COCH₃)-Phe-Lys-Lys-Leu-Gln-OAll (7a)

ESI/MS (m/z)



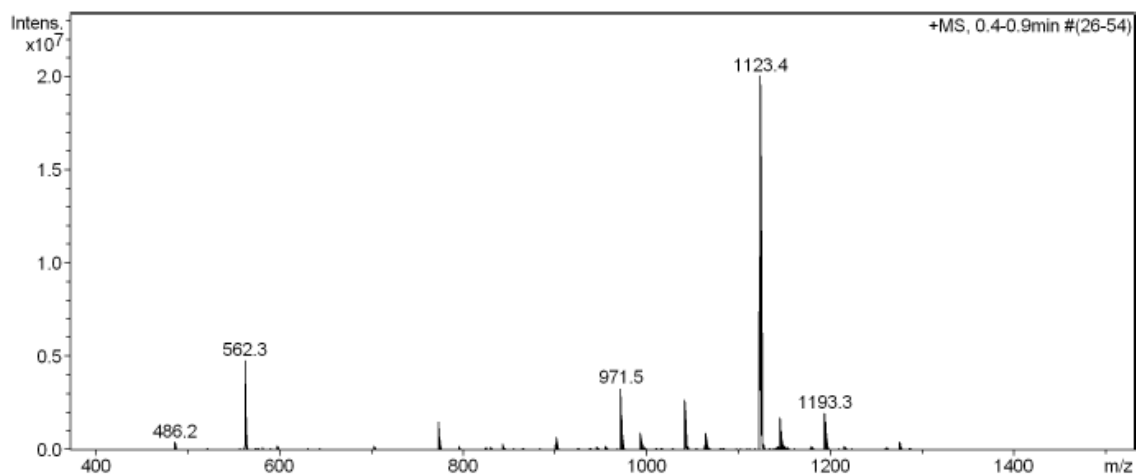
HPLC (220 nm)



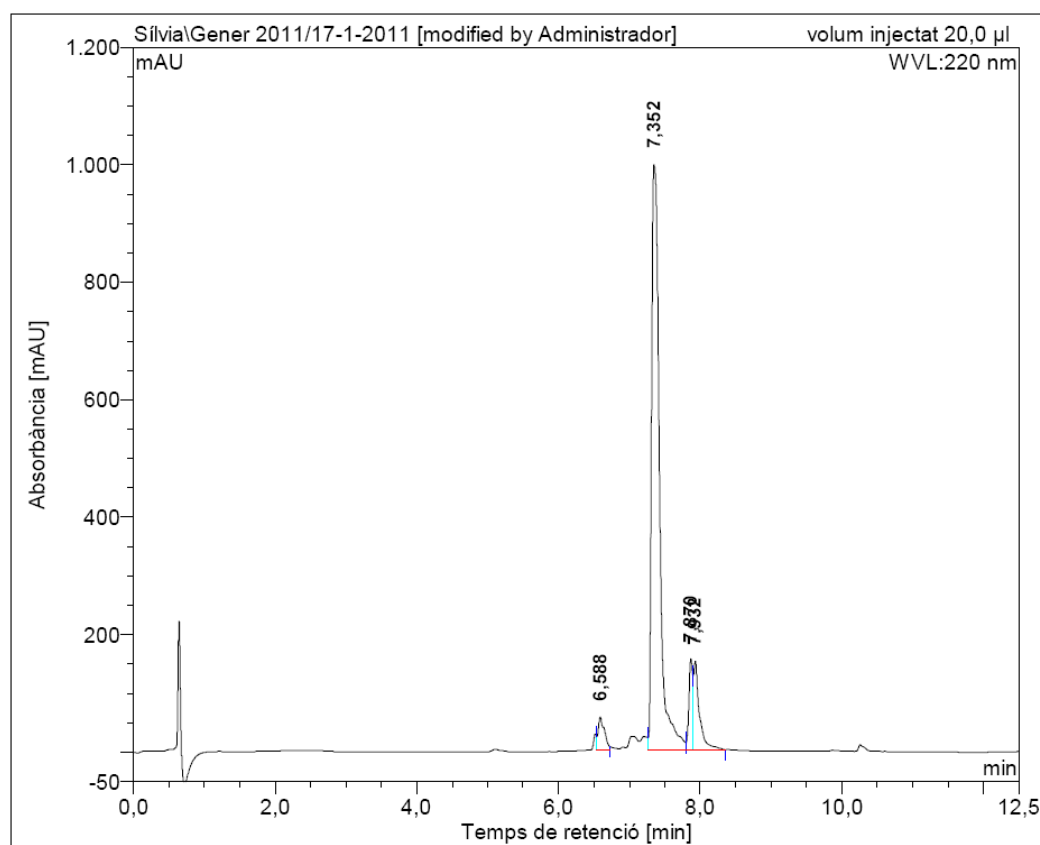
No.	mps retenc min	alçada mAU	Area mAU*min	Area relativa %
1	7,06	18,357	0,710	0,46
2	7,12	158,498	13,821	8,98
3	7,28	1370,242	125,530	81,53
4	7,60	183,745	13,901	9,03
Total:		1730,842	153,963	100,00

Fmoc-Lys(COC₃H₇)-Phe-Lys-Lys-Leu-Gln-OAll (7b)

ESI-MS (m/z)



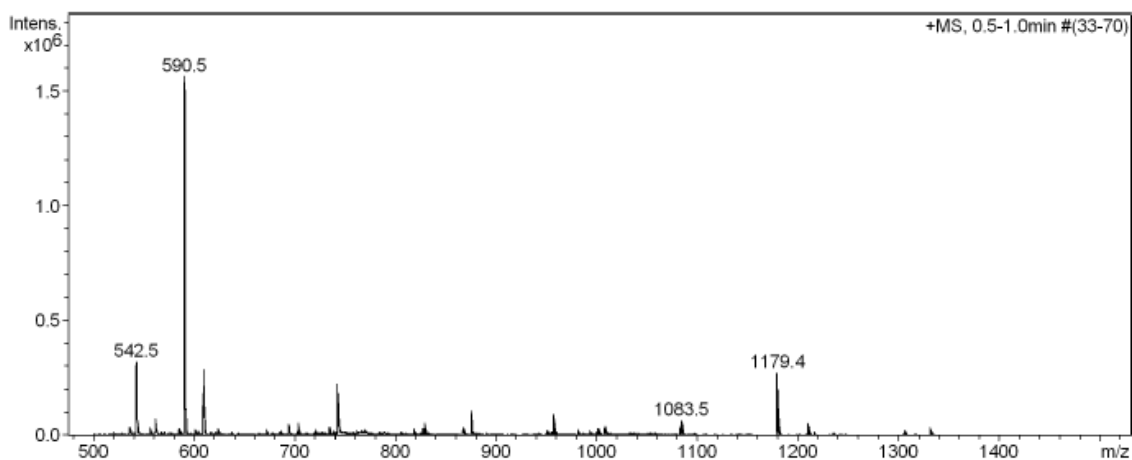
HPLC (220 nm)



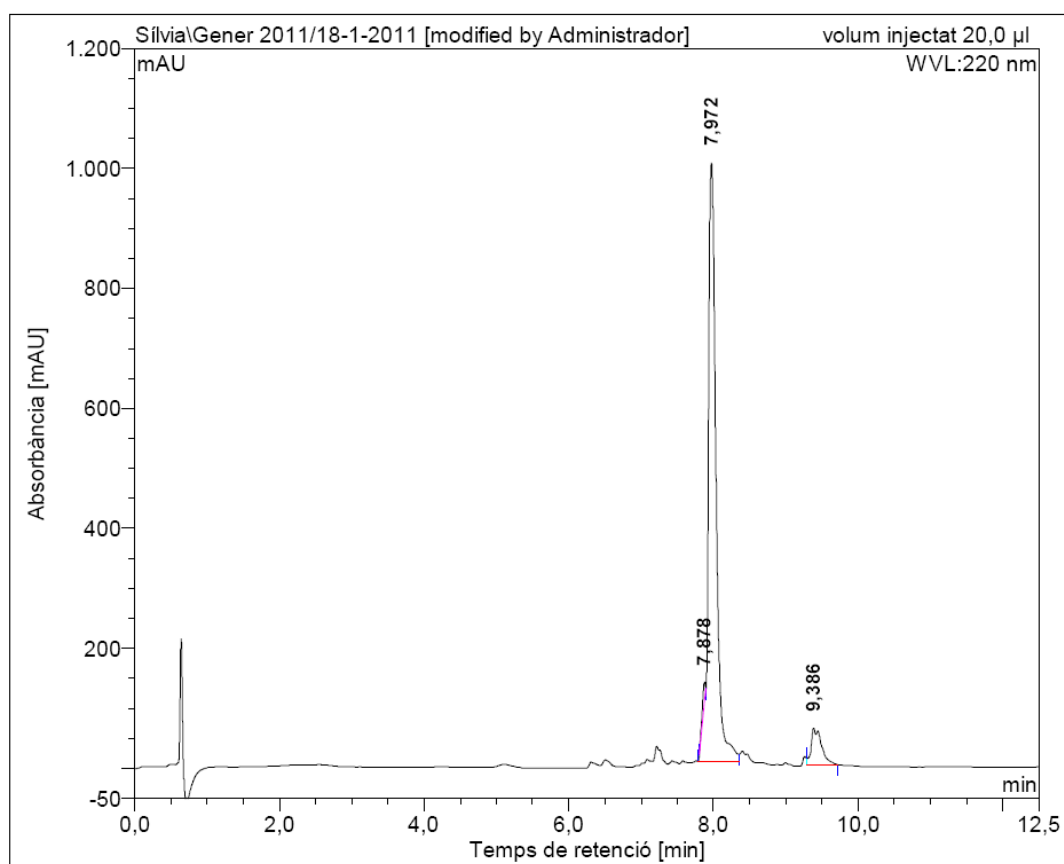
No.	mps retenc min	alçada mAU	Area mAU*min	Area relativa %
1	6,59	55,359	5,548	3,49
2	7,35	995,032	129,227	81,27
3	7,87	154,356	9,479	5,96
4	7,93	151,154	14,759	9,28
Total:		1355,902	159,013	100,00

Fmoc-Lys(COC₇H₁₅)-Phe-Lys-Lys-Leu-Gln-OAll (7c)

ESI-MS (m/z)



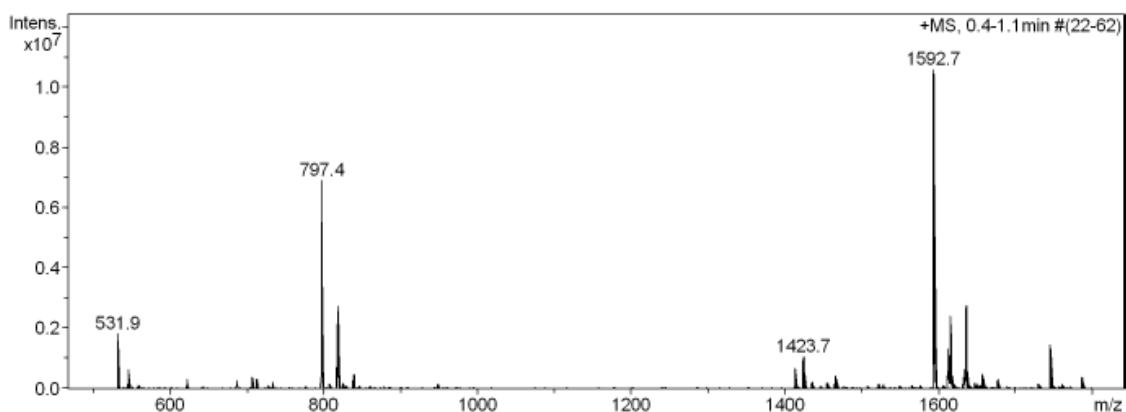
HPLC (220 nm)



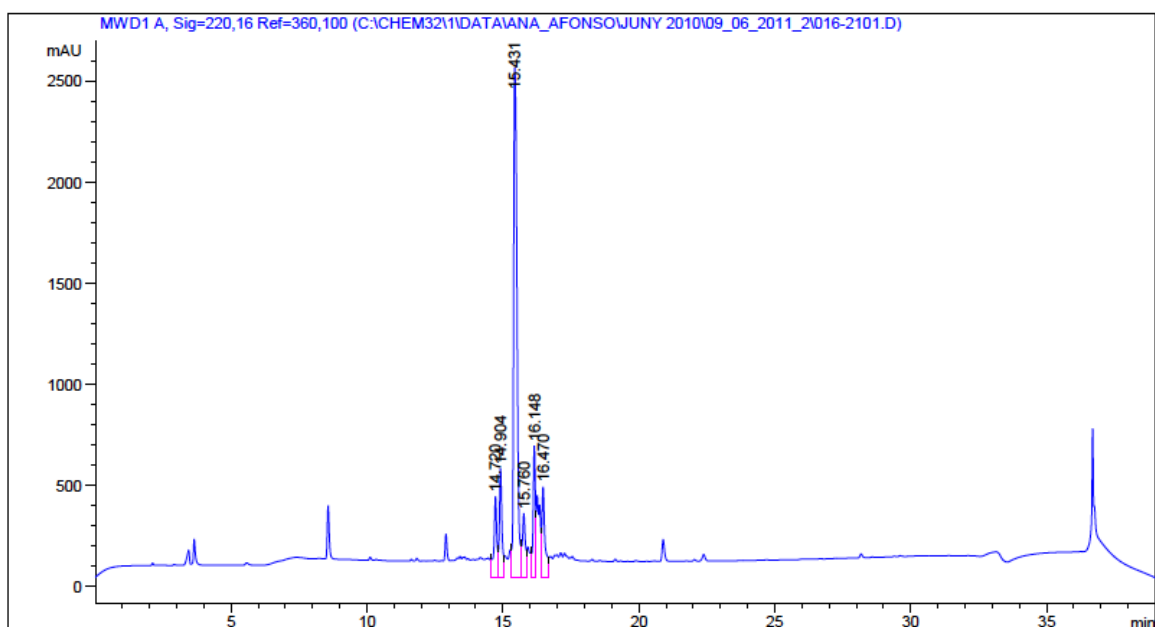
No.	mps retenc min	alçada mAU	Area mAU*min	Area relativa %
1	7,88	32,686	1,856	1,35
2	7,97	997,695	125,644	91,39
3	9,39	60,975	9,975	7,26
Total:		1091,356	137,476	100,00

Fmoc-Lys-Lys-Leu-Lys-Lys(COCH₃)-Phe-Lys-Lys-Leu-Gln-OAlI (11a)

ESI/MS (m/z)



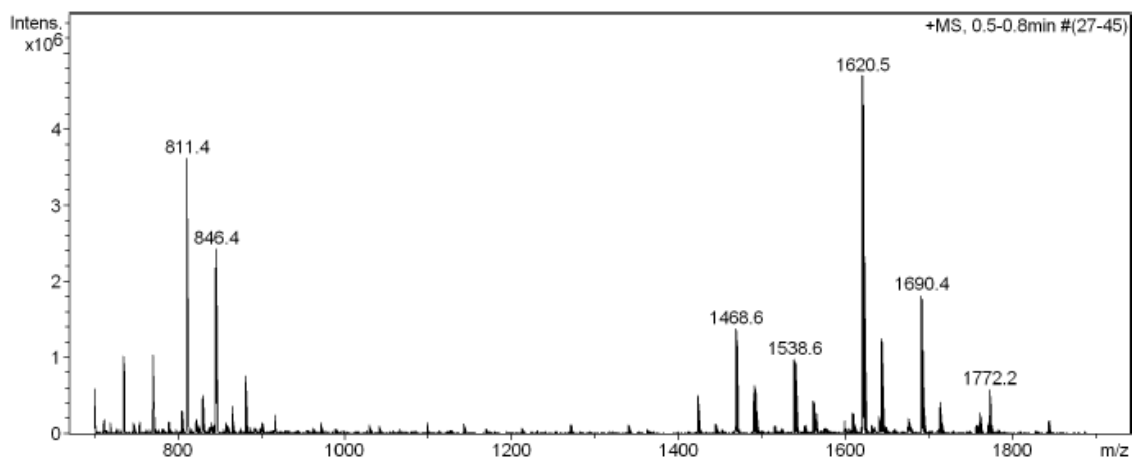
HPLC (220 nm)



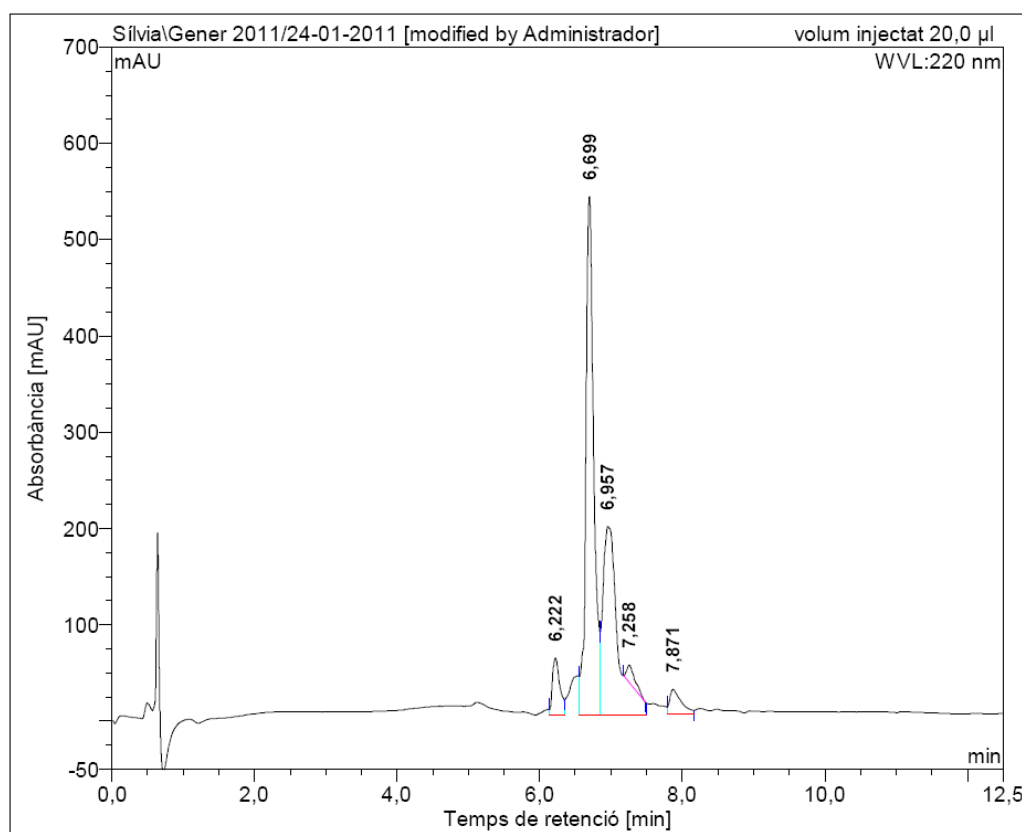
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.720	VV	0.1102	3174.79932	401.88586	8.1241
2	14.904	VV	0.0970	3654.04932	539.89490	9.3505
3	15.431	VV	0.1436	2.24210e4	2524.24414	57.3740
4	15.760	VV	0.1052	2374.83667	317.90781	6.0771
5	16.148	VV	0.0859	3801.68872	653.09937	9.7283
6	16.470	VV	0.1129	3652.27295	448.86636	9.3460

Fmoc-Lys-Lys-Leu-Lys-Lys(COC₃H₇)-Phe-Lys-Lys-Leu-Gln-OAll (11b)

ESI-MS (m/z)



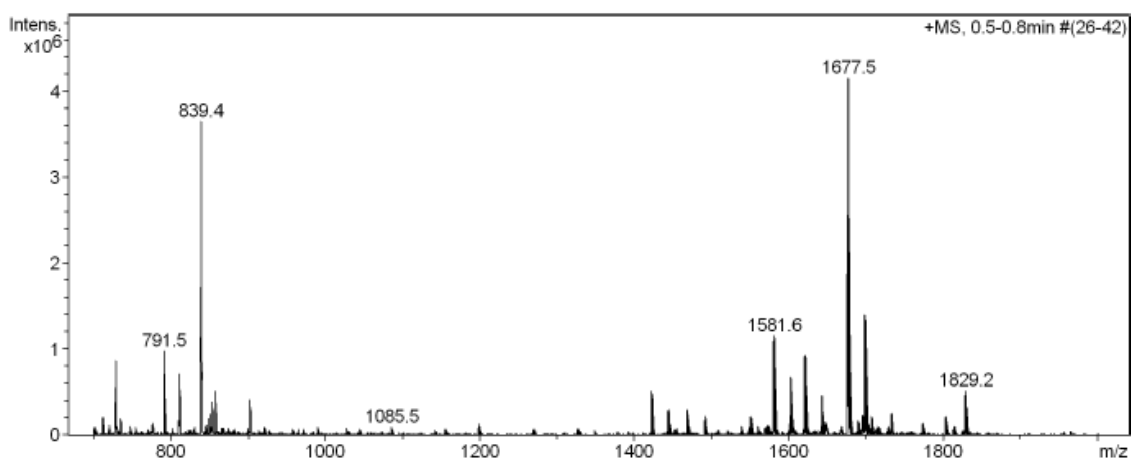
HPLC (220 nm)



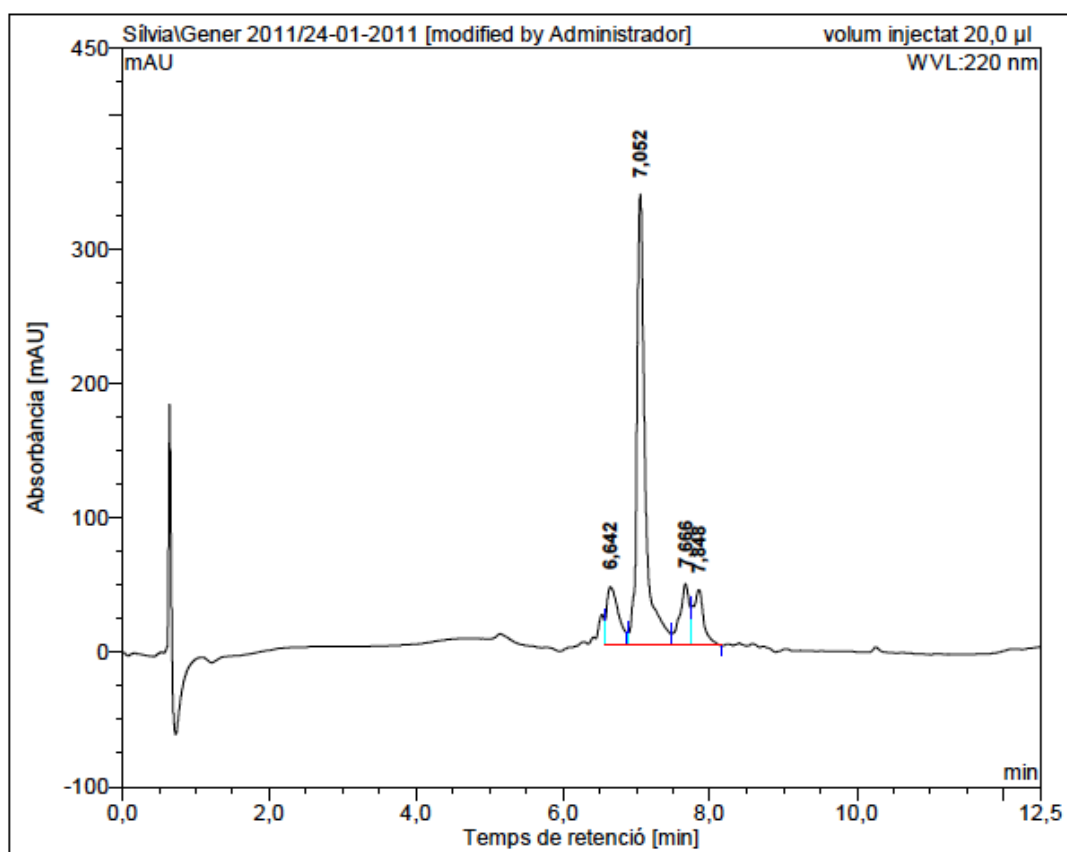
No.	mps retenc min	alçada mAU	Area mAU*min	Area relativa %
1	6,22	58,358	7,085	5,35
2	6,70	537,527	69,007	52,12
3	6,96	195,224	49,002	37,01
4	7,26	18,335	2,539	1,92
5	7,87	25,249	4,762	3,60
Total:		834,692	132,395	100,00

Fmoc-Lys-Lys-Leu-Lys-Lys(COC₇H₁₅)-Phe-Lys-Lys-Leu-Gln-OAll (11c)

ESI-MS (m/z)



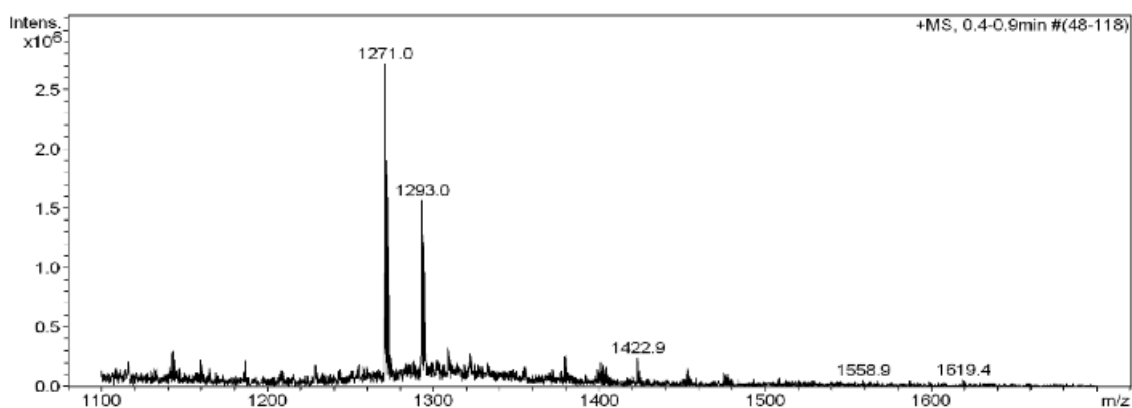
HPLC (220 nm)



No.	mps retenc min	alçada mAU	Area mAU*min	Area relativa %
1	6,64	43,326	7,995	12,01
2	7,05	335,685	44,668	67,09
3	7,67	46,050	6,733	10,11
4	7,85	41,628	7,189	10,80
Total:		466,689	66,584	100,00

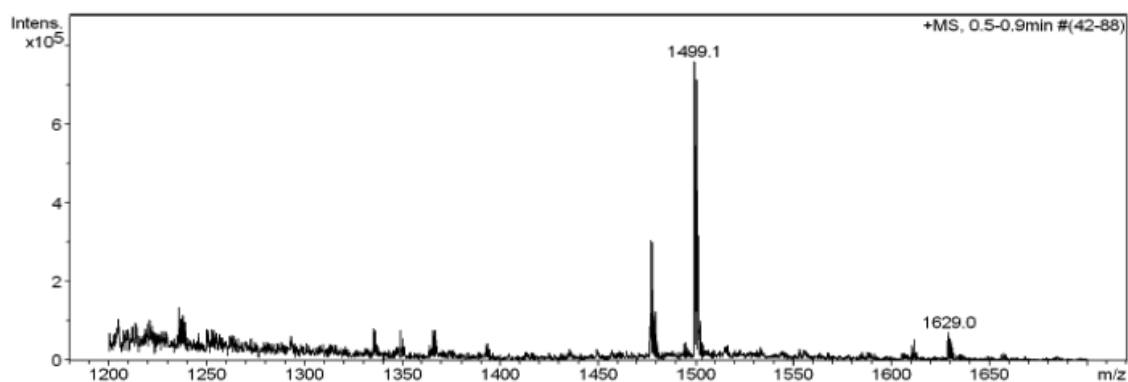
c(Lys-Lys-Leu-Lys-Lys-Phe-Lys-Lys-Leu-Gln) (BPC194)

ESI-MS (m/z)



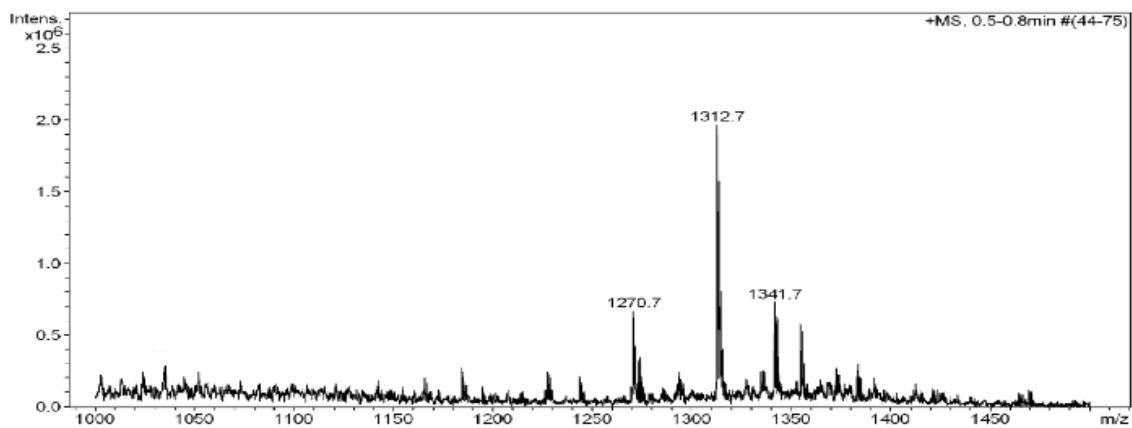
c(Lys-Lys-Leu-Lys-Lys(ivDde)-Phe-Lys-Lys-Leu-Gln)

ESI-MS (m/z)



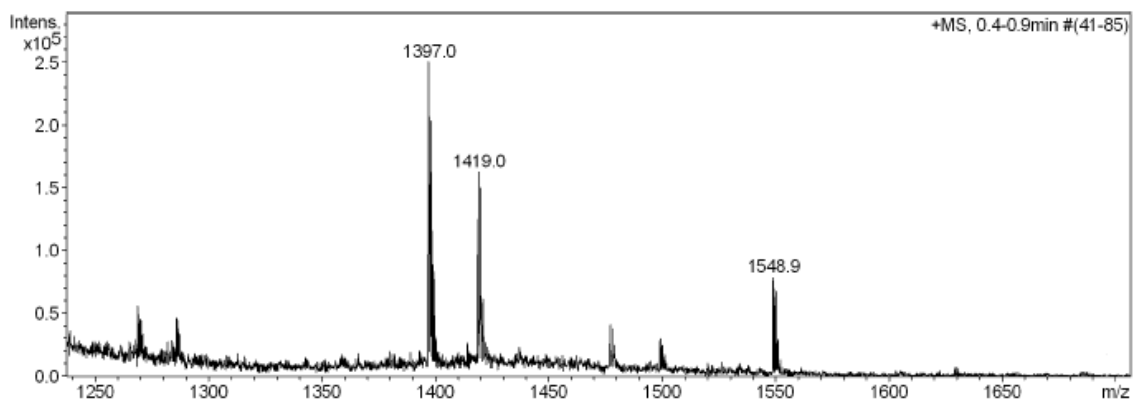
c(Lys-Lys-Leu-Lys-Lys(COCH₃)-Phe-Lys-Lys-Leu-Gln) (5)

ESI/MS (m/z)

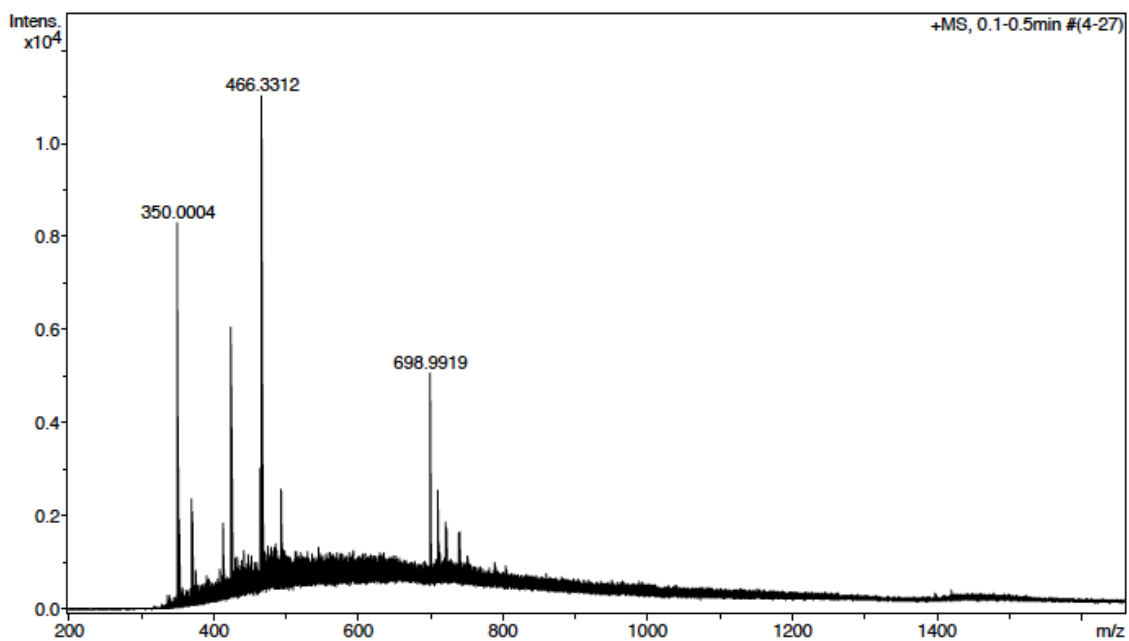


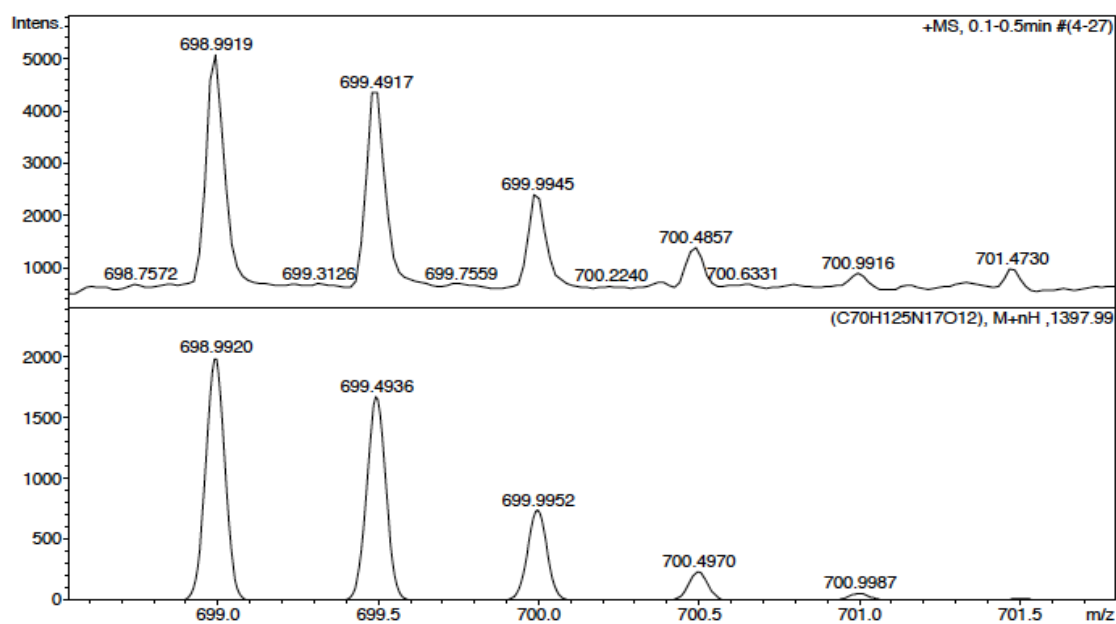
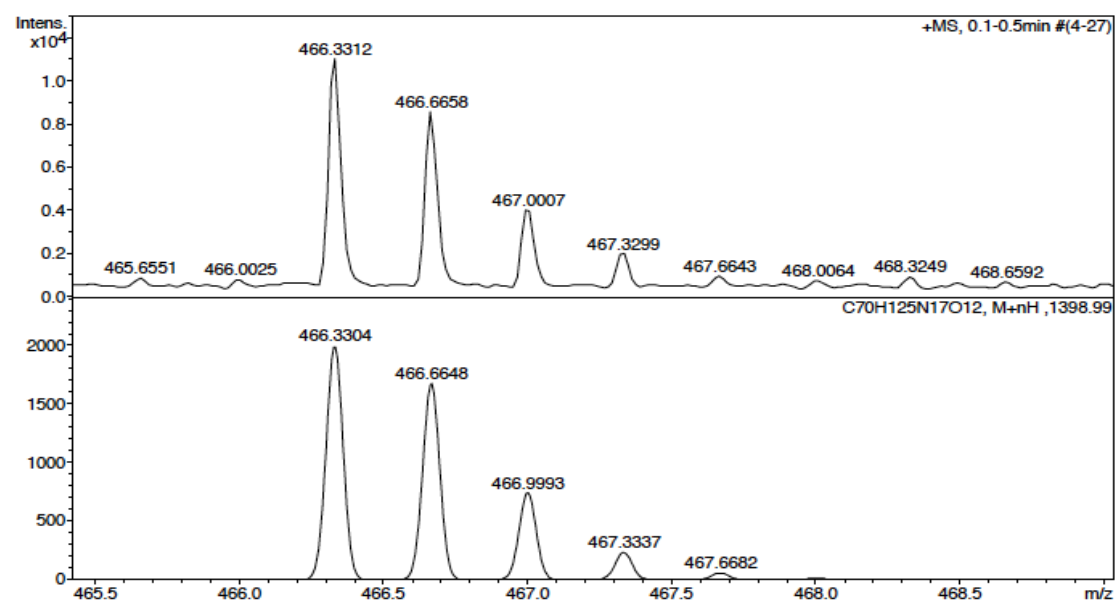
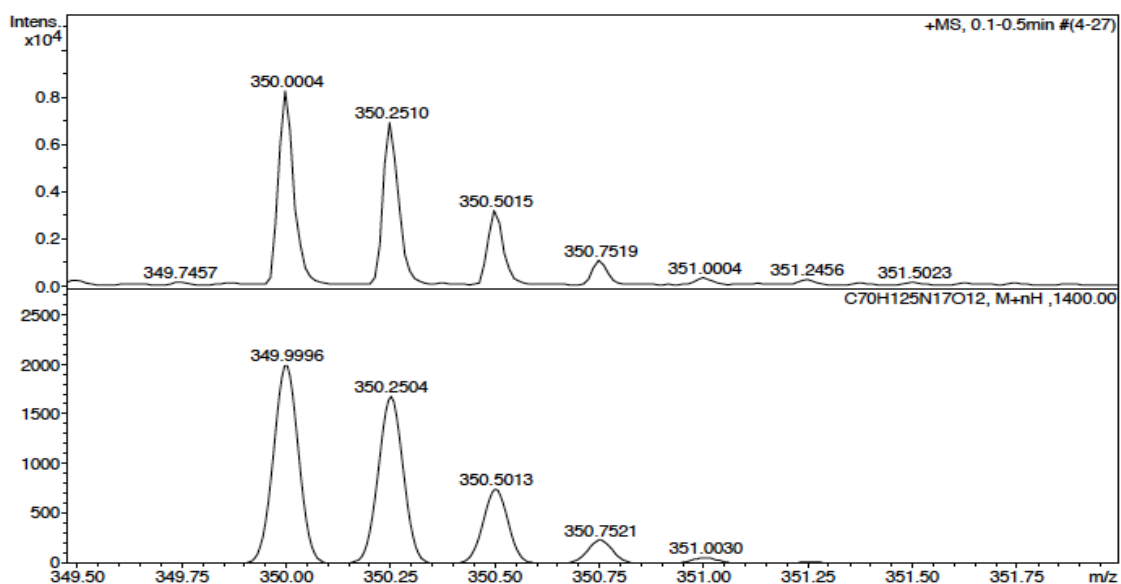
c(Lys-Lys-Leu-Lys-Lys(COC₇H₁₅)-Phe-Lys-Lys-Leu-Gln) (BPC498)

ESI-MS (m/z)



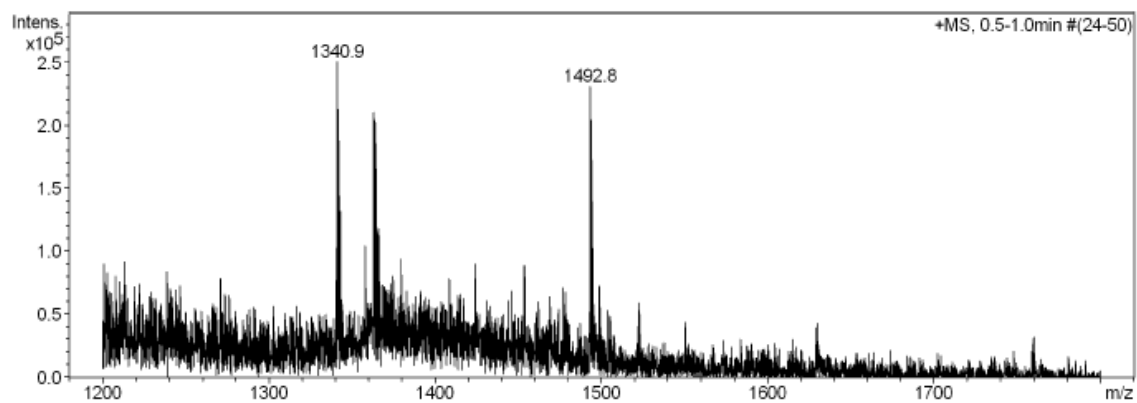
HRMS (ESI) m/z



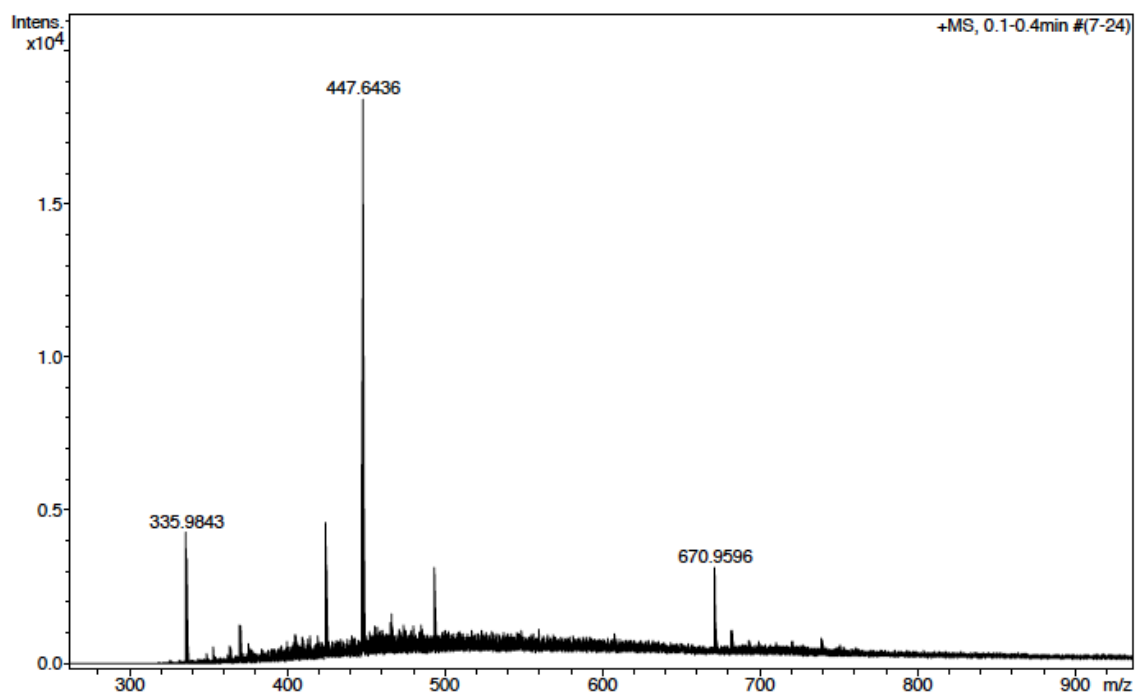


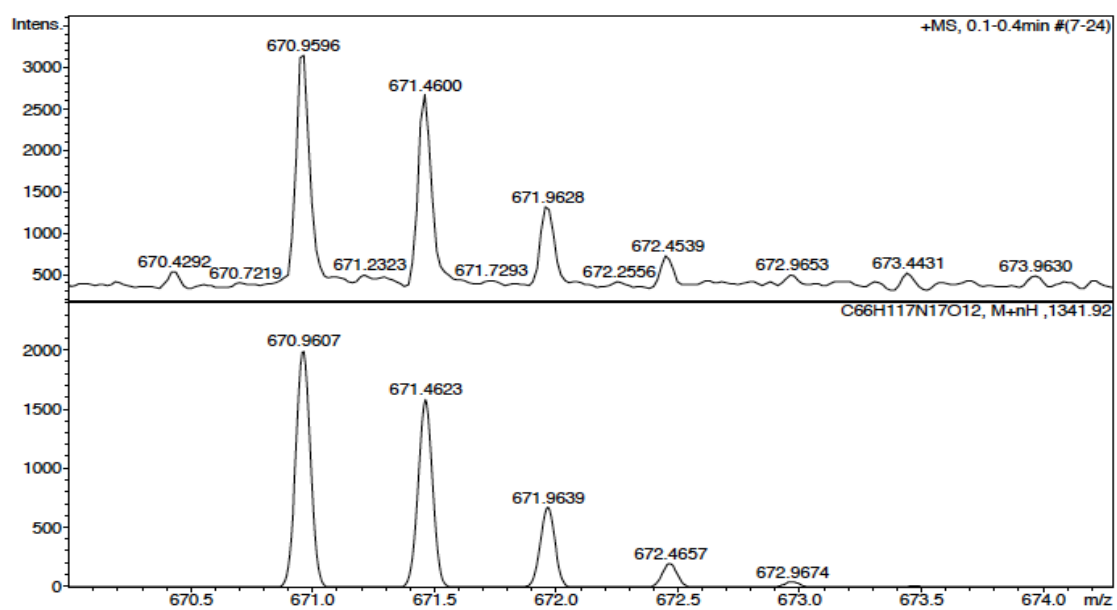
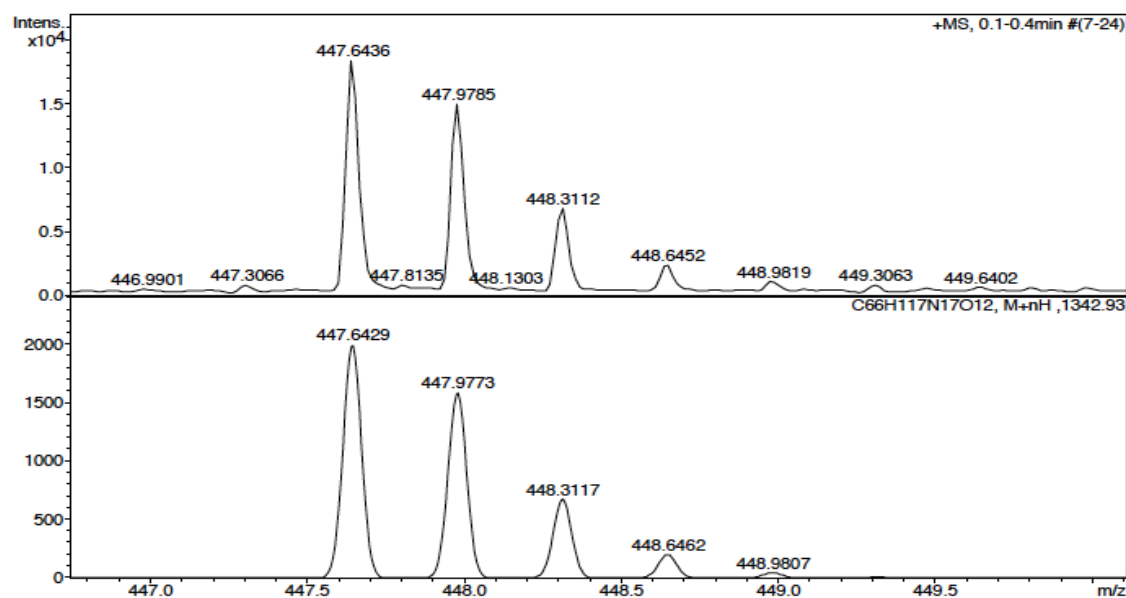
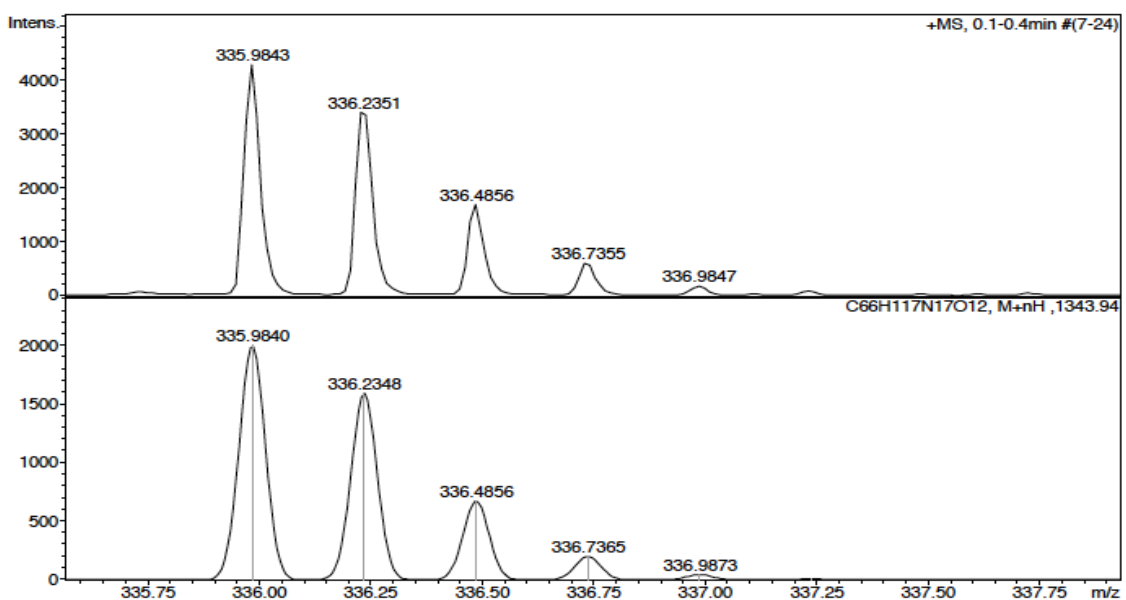
c(Lys-Lys-Leu-Lys-Lys(COC₄H₇)-Phe-Lys-Lys-Leu-Gln) (BPC500)

ESI-MS (m/z)



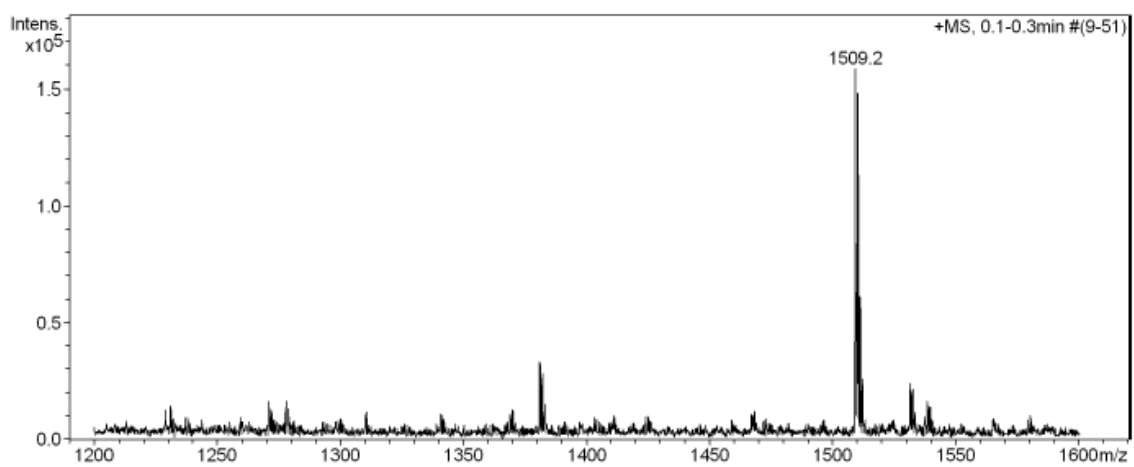
HRMS (ESI) m/z



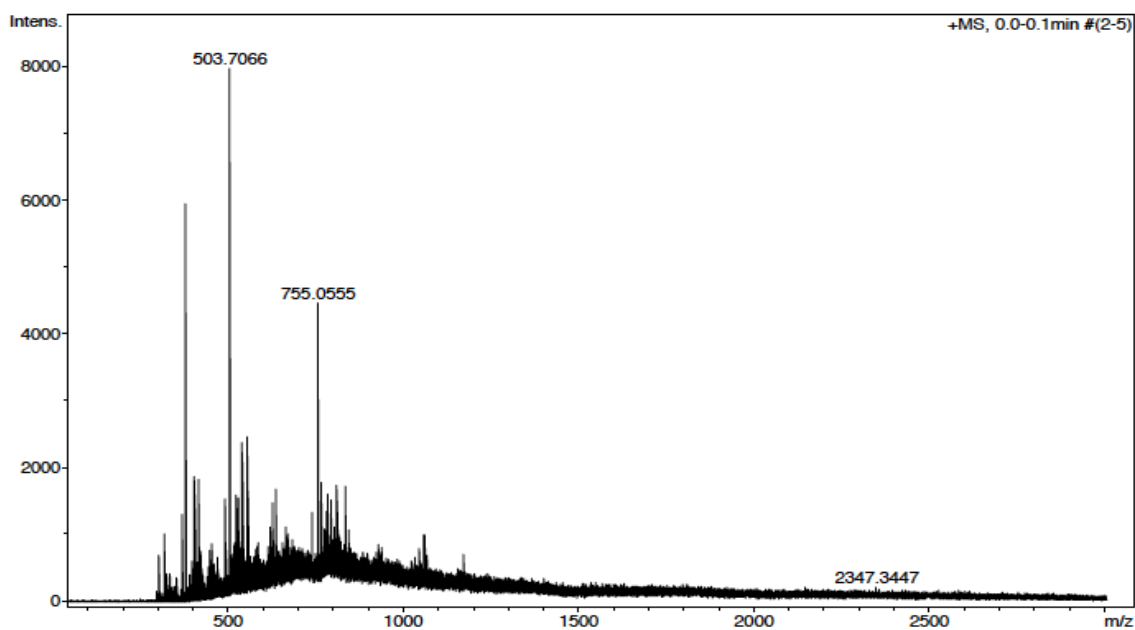


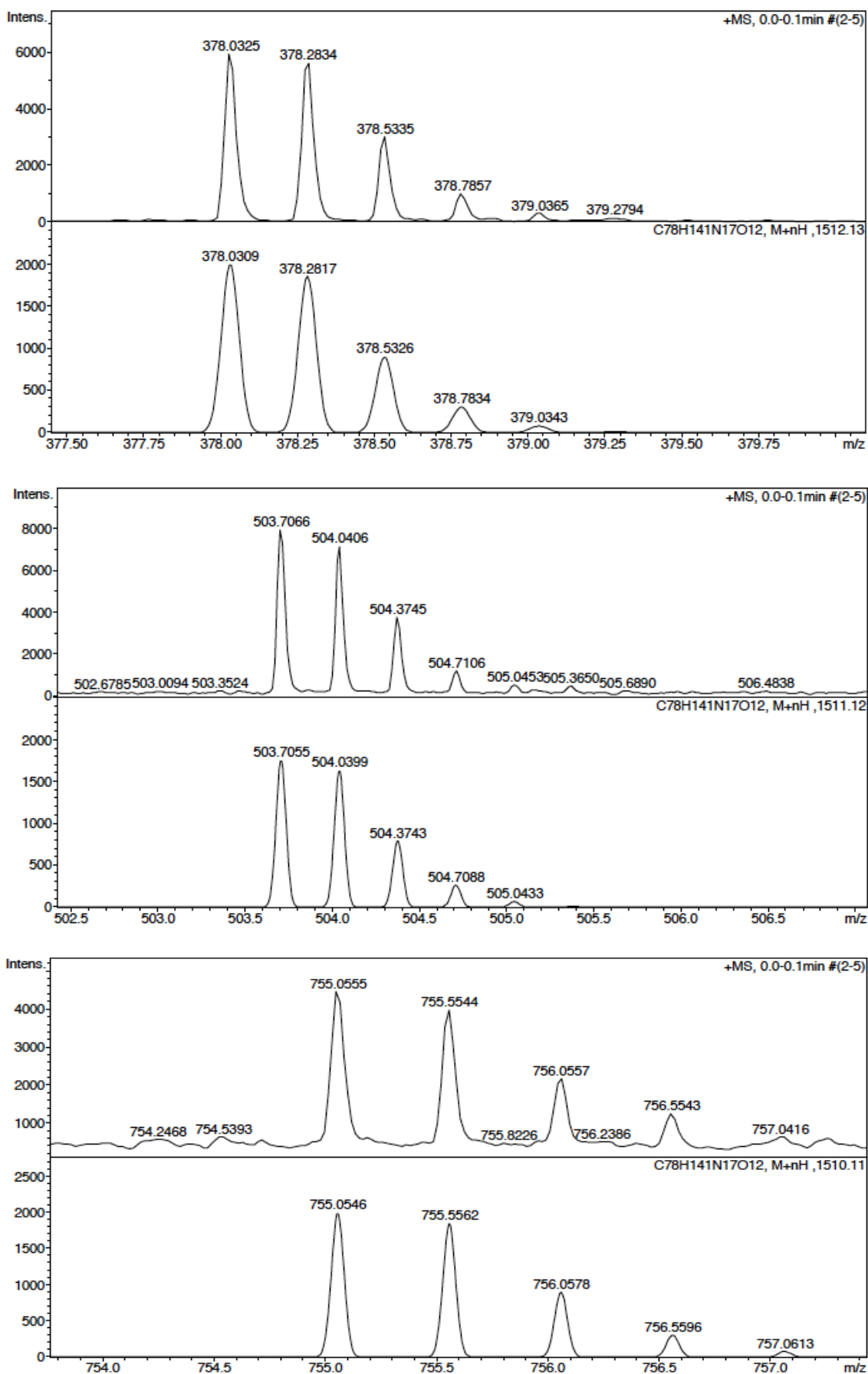
c(Lys-Lys-Leu-Lys-Lys(COC₁₅H₃₁))-Phe-Lys-Lys-Leu-Gln) (BPC502)

ESI-MS (m/z)



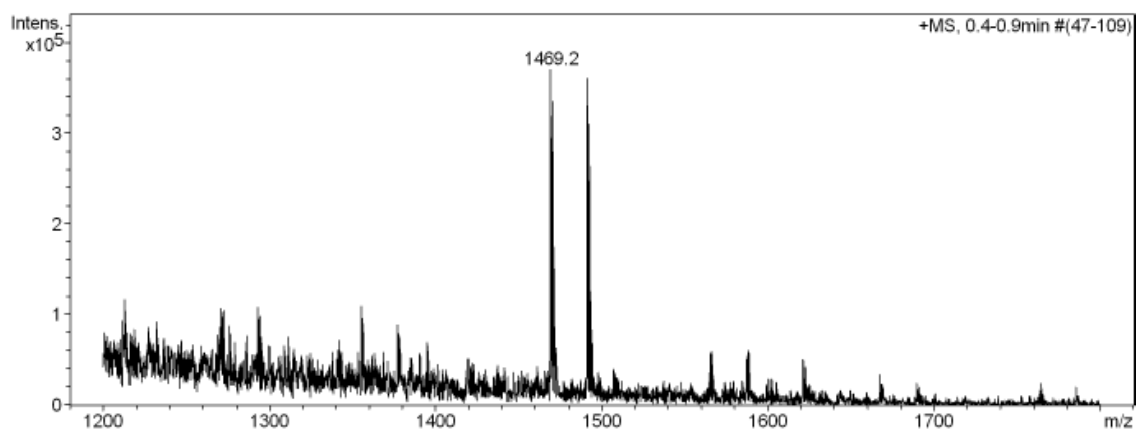
HRMS (ESI) m/z



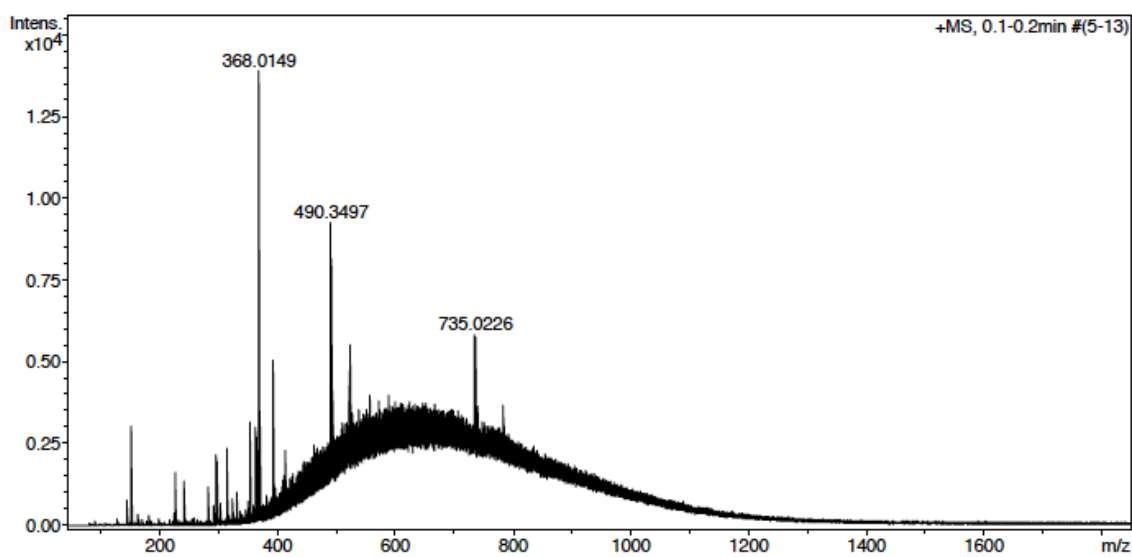


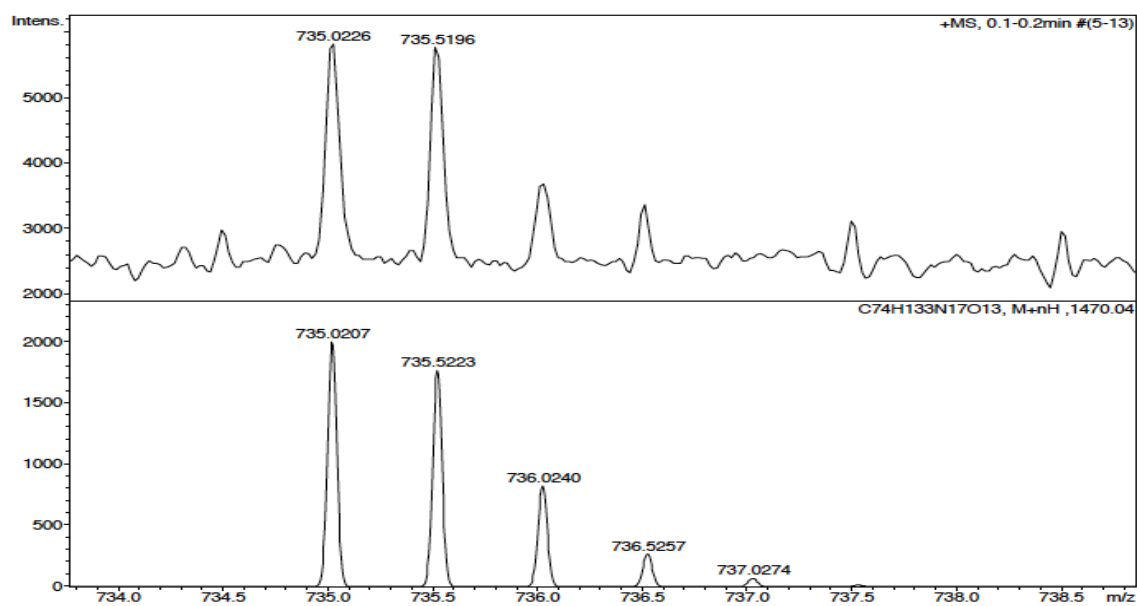
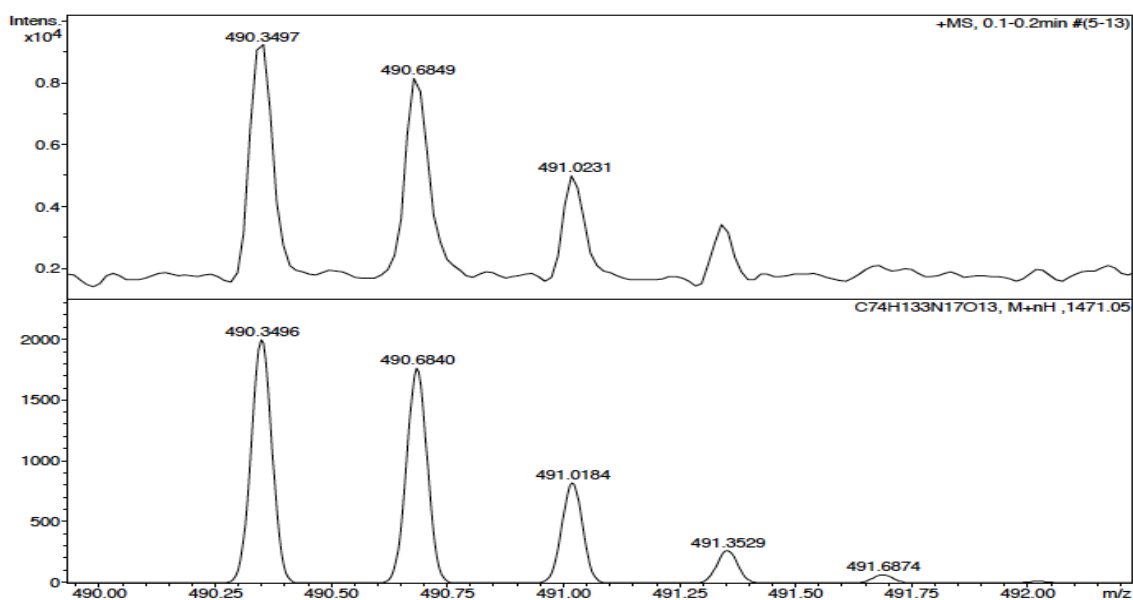
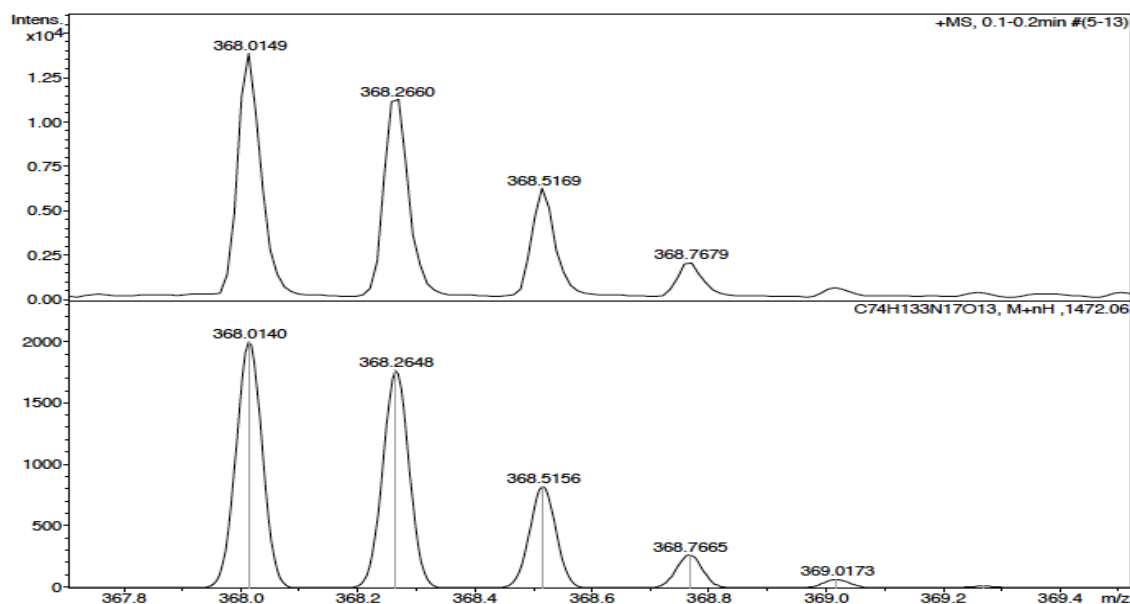
c(Lys-Lys-Leu-Lys-Lys(COC₁₁H₂₂OH)-Phe-Lys-Lys-Leu-Gln) (BPC524)

ESI-MS (m/z)



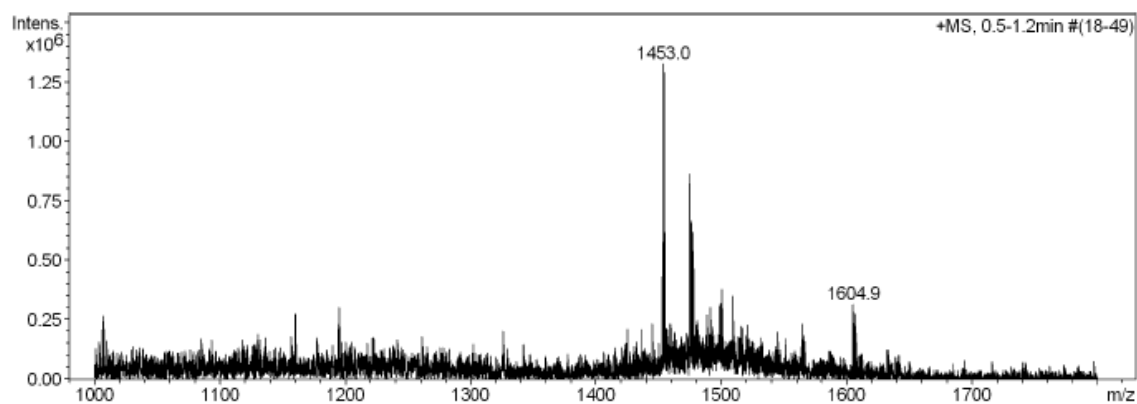
HRMS (ESI) m/z





c(Lys-Lys-Leu-Lys-Lys(COC₁₁H₂₃)-Phe-Lys-Lys-Leu-Gln) (BPC530)

ESI-MS (m/z)



HRMS (ESI) m/z

