

Simple and efficient solid-phase preparation of azido-peptides

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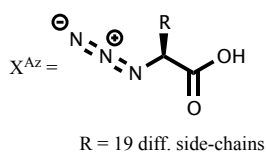
Supporting Information

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For the XLYKAG peptides the following analytical data is available and presented in the order stated here – all for 30 min. reactions:

HPLC (XLYKAG), ESI-MS (XLYKAG), HPLC (X^{Az}LYKAG), LCMS (X^{Az}LYKAG)



X = Arg	s17
Asn	s21
Asp	s25
Cys	s29
Gln	s34
Glu	s38
Gly	s42
His	s46
Ile	s50
Leu	s54
Lys	s58
Met	s62
Phe	s66
Ser	s70
Thr	s74
Trp	s78
Tyr	s82
Val	s86

1.0 Additional figures

1.1 Double diazotransfer reaction cycle

It was speculated that the diazotransfer reagent might be rather unstable and might break down before all ALYKAG peptides have had time to react (though no breakdown of the diazotransfer reagent in solution was observed over time according to NMR, result not shown). Therefore, a reaction with three equivalents of diazotransfer reagent was run for 30 min after which the diazotransfer reagent was replaced with a fresh solution and samples taken after 1, 5 and 30 min (Figure S1). Unfortunately, this ‘double diazotransfer reaction’ cycle did not lead to full conversion either, as ‘only’ 98% conversion was observed.

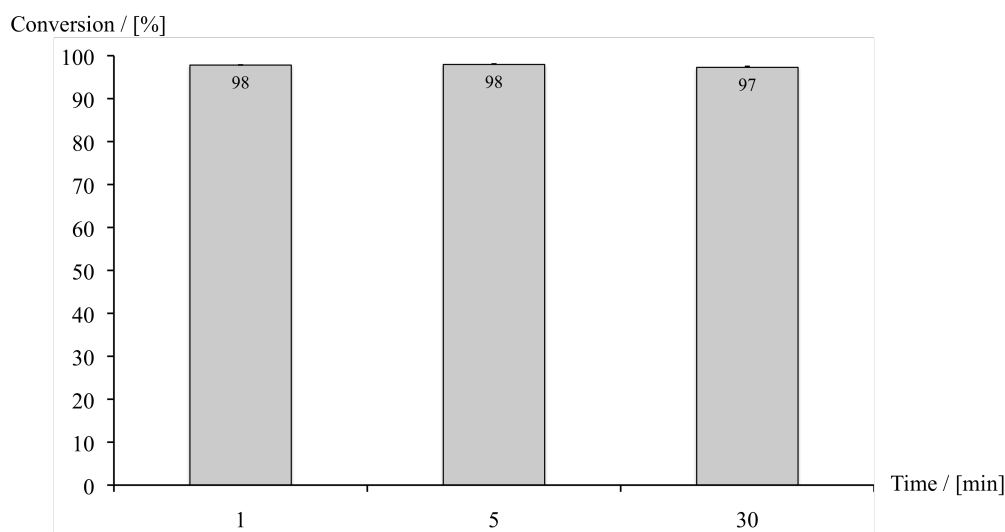


Figure S1. Two cycles of diazotization. A reaction with 3 equiv of imidazole-1-sulfonyl azide hydrochloride and 4.5 equiv K_2CO_3 was run for 30 min, after which the reaction mixture was replaced with a fresh solution of imidazole-1-sulfonyl azide hydrochloride and K_2CO_3 . Samples were then taken after 1, 5 and 30 min. Each bar (value shown inside the bars) represents the relative conversion of amino-ALYKAG to azido-ALYKAG (mean $\pm$ SD; $n = 3$). Error bars are within the bars when not visible.

1.2 Diazotransfer in different solvents

In dichloromethane azido-ALYKAG was barely detectable after 5 min and after 30 min only 5% conversion was observed (Fig, S2). In methanol the diazotization worked better, though still substandard, achieving a conversion of only 40% after 30 min. By comparison of all four solvents a clear trend seems to manifest: increasing solvent polarity leads to increased conversion efficiency. Solvent proticity on the other hand, seems to be less important for high conversions as DMF (non-protic) was better than methanol (protic solvent).

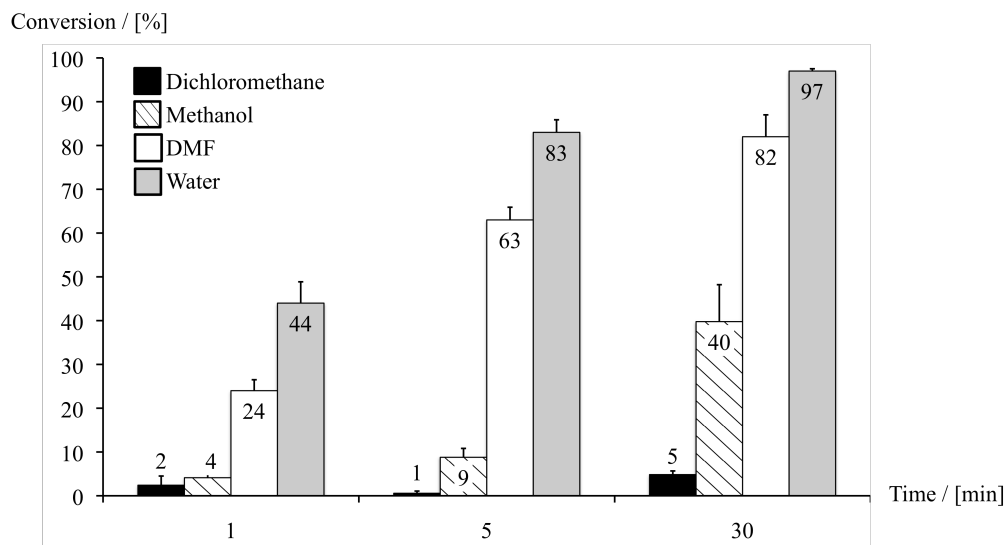


Figure S2. Diazotization of ALYKAG on NovaPEG in different solvents. A reaction with 3 equiv of imidazole-1-sulfonyl azide hydrochloride and 4.5 equiv DIPEA (K_2CO_3 was used in the case of water) was set up and samples were taken after 1, 5 and 30 min. Each bar (value shown inside the bars) represents the relative conversion of amino-ALYKAG to azido-ALYKAG (mean $\pm$ SD; $n = 3$). Error bars are within the bars when not visible.

2.0 Experimental procedures

2.1 General

NovaPEG Rink amide resin and polystyrene Rink amide resin were purchased from Novabiochem and Fmoc-*L*-amino acids from Bachem (Bubendorf, Switzerland) or Novabiochem (EMD Chemicals, Gibbstown, USA). All other chemicals were purchased from Baker, Fluka or Sigma Aldrich and used as received. Mass spectra were acquired on a Thermo Finnigan LCQ-Advantage MAX ESI-ion trap.

2.2 Reverse phase HPLC analysis and LCMS

Analytical HPLC was performed on a Shimadzu LC-20A Prominence system (Shimadzu, 's-Hertogenbosch, The Netherlands) equipped with a C18 ReproSil column, 150x3 mm, particle size 3 μ m (Screening Devices, Amersfoort, The Netherlands). Elution of the peptides was achieved with MeCN/water gradient containing 0.1% trifluoroacetic acid (5-100%, 1-50 min, flow 0.4 mL/min). The desired peaks were integrated manually using a LabSolutions software package (Shimadzu, 's-Hertogenbosch, The Netherlands). LCMS was performed on a Thermo Finnigan LCQ-Fleet ESI-ion trap (Thermo Fischer, Breda, The Netherlands) equipped with a Alltima C18 column, 2.1x150 mm, particle size 3 μ m (Alltech Applied Sciences BV, Breda, Netherlands) using the same gradient as used for HPLC, only trifluoroacetic acid was replaced with formic acid.

2.3 Peptide synthesis

The peptides were synthesized on NovaPEG Rink amide or polystyrene Rink amide resins using a Labortec640 peptide synthesizer (Labortec, Bubendorf, Switzerland) and employing a standard Fmoc solid-phase peptide synthesis (SPPS) protocol.^[14] Briefly, the resin was swollen in DMF for 20 min prior to use. The first and subsequent Fmoc groups were removed by washing the resin with piperidine in DMF (20%, v/v) and then shaking for 25 min with another portion of piperidine in DMF. The desired sequence of amino acids was coupled to the resin using Fmoc-*L*-amino acids (3.0 equiv), diisopropylcarbodiimide (DIPCDI, 3.3 equiv) and *N*-hydroxy benzotriazole (HOBt, 3.6 equiv). Peptide couplings were followed to completion using the Kaiser test.^[20] After the final Fmoc removal the resin was washed with DMF, CH₂Cl₂, *i*-PrOH, CH₂Cl₂, Et₂O and air-dried for at least 2 h.

2.4 Synthesis of ALYKAG

The peptide was synthesized as describe above and a small amount of ALYKAG on resin (approx. 20 mg) was suspended in cleavage mixture (500 μ L, trifluoroacetic acid/water/triisopropylsilane/thioanisole 90:5:2.5:2.5) for 3h. The free peptide was precipitated in Et₂O, redissolved in water and analyzed by analytical HPLC. Retention time 3.88 min (99% pure). ESI-ion trap *m/z*: [M+H]⁺ = 621.3 (calcd. 621.4), [M+2H]²⁺ = 311.3 (calcd. 311.2).

2.5 Synthesis of XLYKAG (X = 20 diff. natural amino acids excl. Pro, Ala)

A common batch of LYKAG (2.24 g resin, loading 0.23 mmol/g) was synthesized as described above and split into 18 portions of 100 mg each after the final Fmoc removal, solvent washes and air-drying. To each portion was coupled one of the 18 different amino acids using standard coupling conditions. After Fmoc removal a small amount of the XLYKAG on resin (approx. 20 mg each) was suspended in cleavage mixture (500 μ L, trifluoroacetic acid/water/triisopropylsilane/thioanisole 90:5:2.5:2.5) for 3h. The free peptide was precipitated from Et₂O, redissolved in water and analyzed by analytical HPLC (typically 99% pure). Analytical data (table S1):

Table S1. XLYKAG MS and HPLC data

X =	Caculated	Measured	Retention time (min)
Arg	[M+H] ⁺ = 706.5, [M+2H] ²⁺ = 353.8	[M+H] ⁺ = 706.4, [M+2H] ²⁺ = 353.7	3.2
Asn	[M+H] ⁺ = 664.4, [M+2H] ²⁺ = 332.7	[M+H] ⁺ = 664.3, [M+2H] ²⁺ = 332.7	3.8
Asp	[M+H] ⁺ = 665.4	[M+H] ⁺ = 665.3	3.8
Cys (SS-dimer)	[M+2H] ²⁺ = 651.4, [M+3H] ³⁺ = 434.6	[M+2H] ²⁺ = 652.4, [M+3H] ³⁺ = 435.3	12.5
Gln	[M+H] ⁺ = 678.4, [M+2H] ²⁺ = 339.7	[M+H] ²⁺ = 678.3, [M+2H] ²⁺ = 339.7	3.7
Glu	[M+H] ⁺ = 679.4, [M+2H] ²⁺ = 340.2	[M+H] ⁺ = 679.3, [M+2H] ²⁺ = 340.2	3.8
Gly	[M+H] ⁺ = 607.4, [M+2H] ²⁺ = 304.2	[M+H] ⁺ = 607.3, [M+2H] ²⁺ = 304.2	3.8
His	[M+H] ⁺ = 687.4, [M+2H] ²⁺ = 344.2	[M+H] ⁺ = 687.3, [M+2H] ²⁺ = 344.3	3.1
Ile	[M+H] ⁺ = 663.4, [M+2H] ²⁺ = 332.2	[M+H] ⁺ = 663.4, [M+2H] ²⁺ = 332.3	5.6
Leu	[M+H] ⁺ = 663.4, [M+2H] ²⁺ = 332.2	[M+H] ⁺ = 663.4, [M+2H] ²⁺ = 332.3	7.1
Lys	[M+H] ⁺ = 678.4, [M+2H] ²⁺ = 339.7	[M+H] ⁺ = 678.4, [M+2H] ²⁺ = 339.7	3.0
Met	[M+H] ⁺ = 681.4, [M+2H] ²⁺ = 341.2	[M+H] ⁺ = 681.3, [M+2H] ²⁺ = 341.2	5.6
Phe	[M+H] ⁺ = 697.4, [M+2H] ²⁺ = 349.2	[M+H] ⁺ = 697.3, [M+2H] ²⁺ = 349.2	11.6
Ser	[M+H] ⁺ = 637.4, [M+2H] ²⁺ = 319.2	[M+H] ⁺ = 637.4, [M+2H] ²⁺ = 319.3	3.9
Thr	[M+H] ⁺ = 651.4, [M+2H] ²⁺ = 326.2	[M+H] ⁺ = 651.3, [M+2H] ²⁺ = 326.2	3.7
Trp	[M+H] ⁺ = 736.4, [M+2H] ²⁺ = 368.7	[M+H] ⁺ = 736.3, [M+2H] ²⁺ = 368.7	12.5
Tyr	[M+H] ⁺ = 713.4, [M+2H] ²⁺ = 357.2	[M+H] ⁺ = 713.4, [M+2H] ²⁺ = 357.3	6.3
Val	[M+H] ⁺ = 649.4, [M+2H] ²⁺ = 325.2	[M+H] ⁺ = 649.3, [M+2H] ²⁺ = 325.3	4.2

2.6 General procedure for on-resin diazotization

The ALYKAG on NovaPEG resin was swollen in DMF for 5 min and then incubated in HOBt (1 M in DMF) for 30 min. subsequently, the resin was washed with 20% piperidine in DMF, DMF, CH₂Cl₂, *i*-PrOH, CH₂Cl₂, *i*-PrOH, water (in case of reactions in MeOH or CH₂Cl₂, water was replaced with MeOH or CH₂Cl₂, respectively). In all experiments the ratio between resin and solvent (water) was kept constant at 12 μ L solvent/mg resin. In most experiments (indicated if otherwise) imidazole-1-sulfonyl azide hydrochloride (3.0 equiv) and K₂CO₃ (4.5 equiv) were prepared as two separate aqueous solutions each corresponding to 12.5% of the total volume (freshly made prior to each experiment[†]). The remaining water (75% of the total volume) was added to the resin and the mixture was shaken for 5 min. The reaction was initiated by addition of the imidazole-1-sulfonyl azide hydrochloride and K₂CO₃ solutions to the resin and the resulting suspension was shaken. After a desired amount of time, a sample was removed with a pipette, added to a filter tube and washed extensively with water. After removal of the last sample, all samples were washed with *i*-PrOH, CH₂Cl₂, *i*-PrOH, CH₂Cl₂, Et₂O and air-dried for at least 2 h. The dry resins were then suspended in cleavage mixture (500 μ L, trifluoroacetic acid/water/triisopropylsilane/thioanisole 90:5:2.5:2.5) for 3 h. The free peptide was precipitated from Et₂O, redissolved in water and analyzed by analytical HPLC. Analytical data for azido-ALYKAG: Retention time 13.72 min (99% pure). ESI-ion trap *m/z*: [M+H]⁺ = 648.28 (calcd. 647.34).

All diazotization reactions described herein follow this general protocol. Only deviations from this protocol are described below.

When amounts of resin are stated below this represents the total weight of resin, peptide and protecting groups, whereas the molar amounts refers to the molar amount of peptide.

2.7 Kinetics of diazotization

ALYKAG on NovaPEG resin (150 mg, loading 0.23 mmol/g, 29.4 μ mol) was suspended in water (1.8 mL) and K₂CO₃ (132 μ mol, 24.3 mg) and imidazole-1-sulfonyl azide hydrochloride (87.9 μ mol, 18.4 mg) were added in this order. Samples (150 μ L) were taken after 15 sec., 30 sec., 1 min, 2 min, 3 min, 4 min, 5 min, 7 min, 10 min, 30 min and 60 min. A similar reaction was carried out in which CuSO₄ (2.9 μ mol, 0.73 mg) was added as well. All experiments were performed in quadruple.

2.8 Variation of the amount of diazotransfer reagent

Three experiments with 1, 3 or 5 equivalents of imidazole-1-sulfonyl azide hydrochloride (*n* = 1, 3 or 5) were performed. ALYKAG on NovaPEG resin (50 mg, loading 0.23 mmol/g, 9.76 μ mol) was suspended in water (600 – [20 + 22.2] $\times$ *n*) μ L. Then an aqueous solution (0.88 M) of K₂CO₃ (22.2 $\times$ *n*) μ L was added followed by an aqueous solution (0.49 M) of imidazole-1-sulfonyl azide hydrochloride (20 $\times$ *n*) μ L. Samples (200 μ L) were taken after 1, 5 and 30 min. All experiments were performed in quadruple.

2.9 Two cycles of diazotiation

ALYKAG on NovaPEG resin (40 mg, loading 0.23 mmol/g, 7.81 μ mol) was suspended in water (392 μ L). Then an aqueous solution of K₂CO₃ (0.88 M, 35.2 μ mol, 40 μ L) was added followed by an aqueous solution of imidazole-1-sulfonyl azide hydrochloride (0.49 M, 23.5 μ mol, 48 μ L). The reaction was shaken for 30 min after which the reaction solution was removed and the resin washed extensively with water. Fresh amounts of water, imidazole-1-sulfonyl azide hydrochloride and K₂CO₃ were added and samples (160 μ L) were taken after 1, 5 and 30 min. All experiments were performed in triplicate.

2.10 Diazotization on NovaPEG or polystyrene resin in DMF using K₂CO₃ as base

ALYKAG on polystyrene resin (50 mg, loading 0.39 mmol/g, 15.0 μ mol) was suspended in DMF (417 μ L). Then an aqueous solution of K₂CO₃ (0.88 M, 67.4 μ mol, 76.6 μ L) was added followed by a

solution of imidazole-1-sulfonyl azide hydrochloride in DMF (0.49 M, 45.0 μ mol, 91.9 μ L). ALYKAG on NovaPEG (50 mg, loading 0.23 mmol/g, 9.76 μ mol) was treated similarly; only less reagents were added as the loading is lower, hence water and DMF were added to compensate for this. Samples (200 μ L) were taken after 1, 5 and 30 min. All experiments were performed in triplicate.

2.11 Diazotization on NovaPEG or polystyrene resin in DMF using DIPEA as base

ALYKAG on polystyrene resin (50 mg, loading 0.39 mmol/g, 15.0 μ mol) was suspended in DMF (496 μ L). Then DIPEA (67.4 μ mol, 11.7 μ L) was added followed by a solution (0.49 M) of imidazole-1-sulfonyl azide hydrochloride in DMF (0.49 M, 45.0 μ mol, 91.9 μ L). ALYKAG on NovaPEG (50 mg, loading 0.23 mmol/g, 9.76 μ mol) was treated similarly; only less reagents were added as the resin loading was lower, hence DMF were added to compensate for this. Samples (200 μ L) were taken after 1, 5 and 30 min. All experiments were performed in triplicate.

2.12 Diazotization on NovaPEG resin in MeOH using DIPEA as base

Performed as described above for NovaPEG resin in DMF.

2.13 Diazotization on NovaPEG resin in CH₂Cl₂ using DIPEA as base

Imidazole-1-sulfonyl azide hydrochloride only dissolves sparingly in CH₂Cl₂ so to circumvent this DIPEA (366 μ mol, 63.8 μ L) was added to a suspension of imidazole-1-sulfonyl azide hydrochloride (51.2 mg, 244 μ mol) in CH₂Cl₂ (436 μ L) leading to a clear solution. The required amount of this solution (60 μ L) was added immediately to ALYKAG on NovaPEG resin (50 mg, loading 0.23 mmol/g, 9.76 μ mol) suspended in CH₂Cl₂ (540 μ L) and the remainder disposed. Samples (200 μ L) were taken after 1, 5 and 30 min. All experiments were performed in triplicate.

2.14 Diazotization of XLYKAG – variation of the N-terminal amino acid

XLYKAG (20 mg, loading 0.23 μ mol/g) was suspended in water (196 μ L). Then an aqueous solution of K₂CO₃ (0.88 M, 17.5 μ mol, 19.9 μ L) was added followed by an aqueous solution of imidazole-1-sulfonyl azide hydrochloride (0.49 M, 11.7 μ mol, 23.9 μ L). Samples (80 μ L) were taken after 1, 5 and 30 min. All experiments were performed in triplicate.

Table S2. X^{Az}LYKAG MS and HPLC data

X =	Calculated	Measured	Retention time (min)
Arg	[M+H] ⁺ = 732.42,	[M+H] ⁺ = 732.28	11.64
	[M+2H] ²⁺ = 366.71	[M+2H] ²⁺ = 366.68	
Asn	[M+H] ⁺ = 690.36,	[M+H] ⁺ = 690.32,	11.88
	[M+2H] ²⁺ = 345.68	[M+2H] ²⁺ = 345.52	
Asp	[M+H] ⁺ = 691.35	[M+H] ⁺ = 691.32	11.83
Cys* (SS-dimer)	[M+H] ⁺ = 678.33	[M+H] ⁺ = 678.36	22.18
Gln	[M+H] ⁺ = 704.38,	[M+H] ⁺ = 704.32,	11.60
	[M+2H] ²⁺ = 352.69	[M+2H] ²⁺ = 352.48	
Glu	[M+H] ⁺ = 705.36	[M+H] ⁺ = 705.32	13.42
Gly	[M+H] ⁺ = 633.34	[M+H] ⁺ = 633.28	11.92
His	[M+H] ⁺ = 713.38,	[M+H] ⁺ = 713.20,	7.32
	[M+2H] ²⁺ = 357.19	[M+2H] ²⁺ = 357.12	

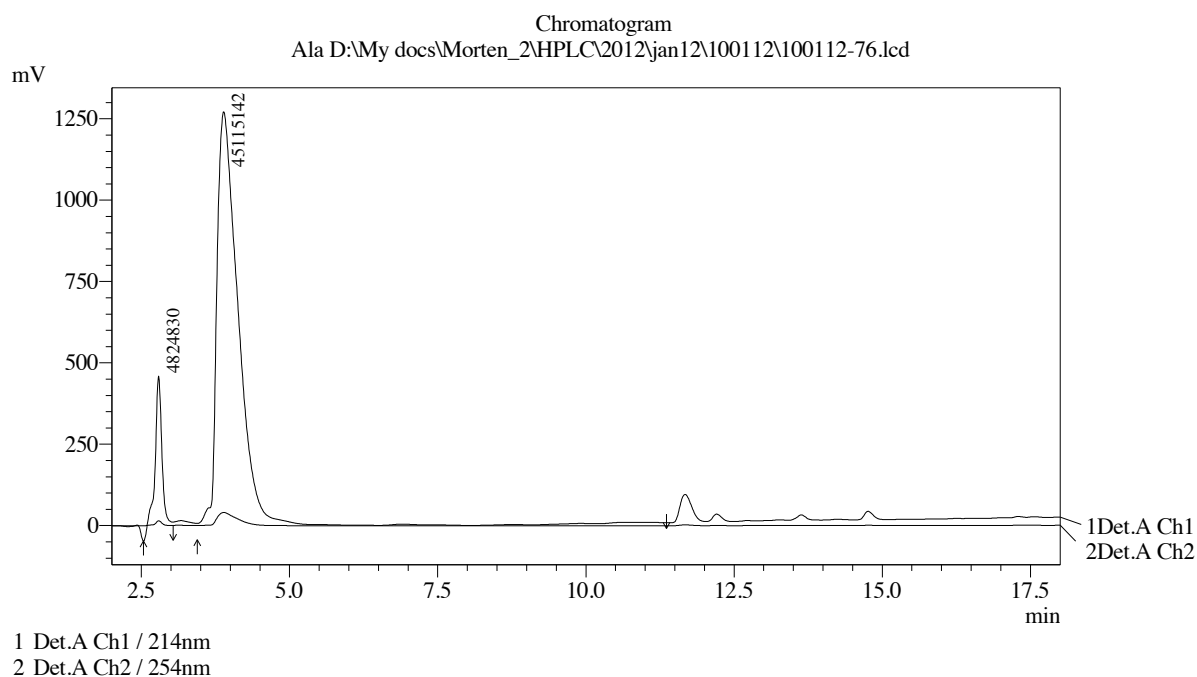
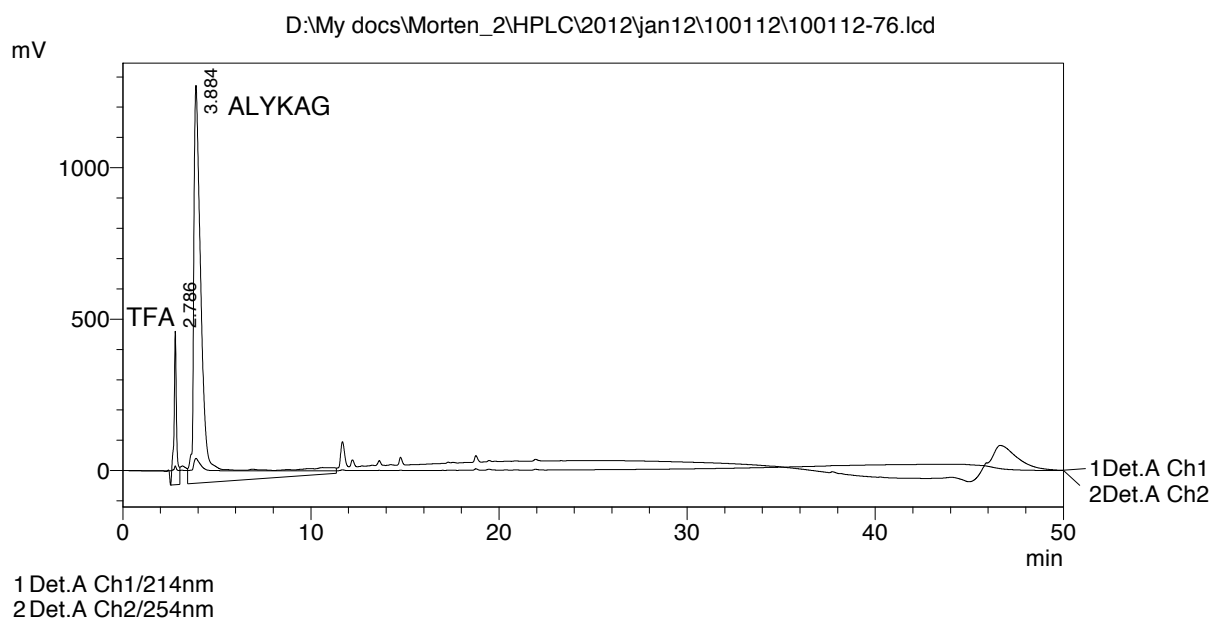
Ile	[M+H] ⁺ = 689.40	[M+H] ⁺ = 689.36	20.99
Leu	[M+H] ⁺ = 689.40	[M+H] ⁺ = 689.36	16.99
Lys	[M+H] ⁺ = 704.41, [M+2H] ²⁺ = 352.71	[M+H] ⁺ = 352.64, [M+2H] ²⁺ = 704.24	11.22
MetO and MetO ₂	[M+H] ⁺ = 723.35, [M+H] ⁺ = 739.35	[M+H] ⁺ = 723.28, [M+H] ⁺ = 739.28	MetO ₂ : 12.43 MetO: 13.10
Phe	[M+H] ⁺ = 723.39	[M+H] ⁺ = 723.32	17.21
Ser	[M+H] ⁺ = 663.35	[M+H] ⁺ = 663.28	11.16
Thr	[M+H] ⁺ = 677.37	[M+H] ⁺ = 677.32	10.98
Trp	[M+H] ⁺ = 762.40	[M+H] ⁺ = 762.36	17.34
Tyr	[M+H] ⁺ = 739.38	[M+H] ⁺ = 739.32	15.33
Val	[M+H] ⁺ = 675.39	[M+H] ⁺ = 675.32	15.90

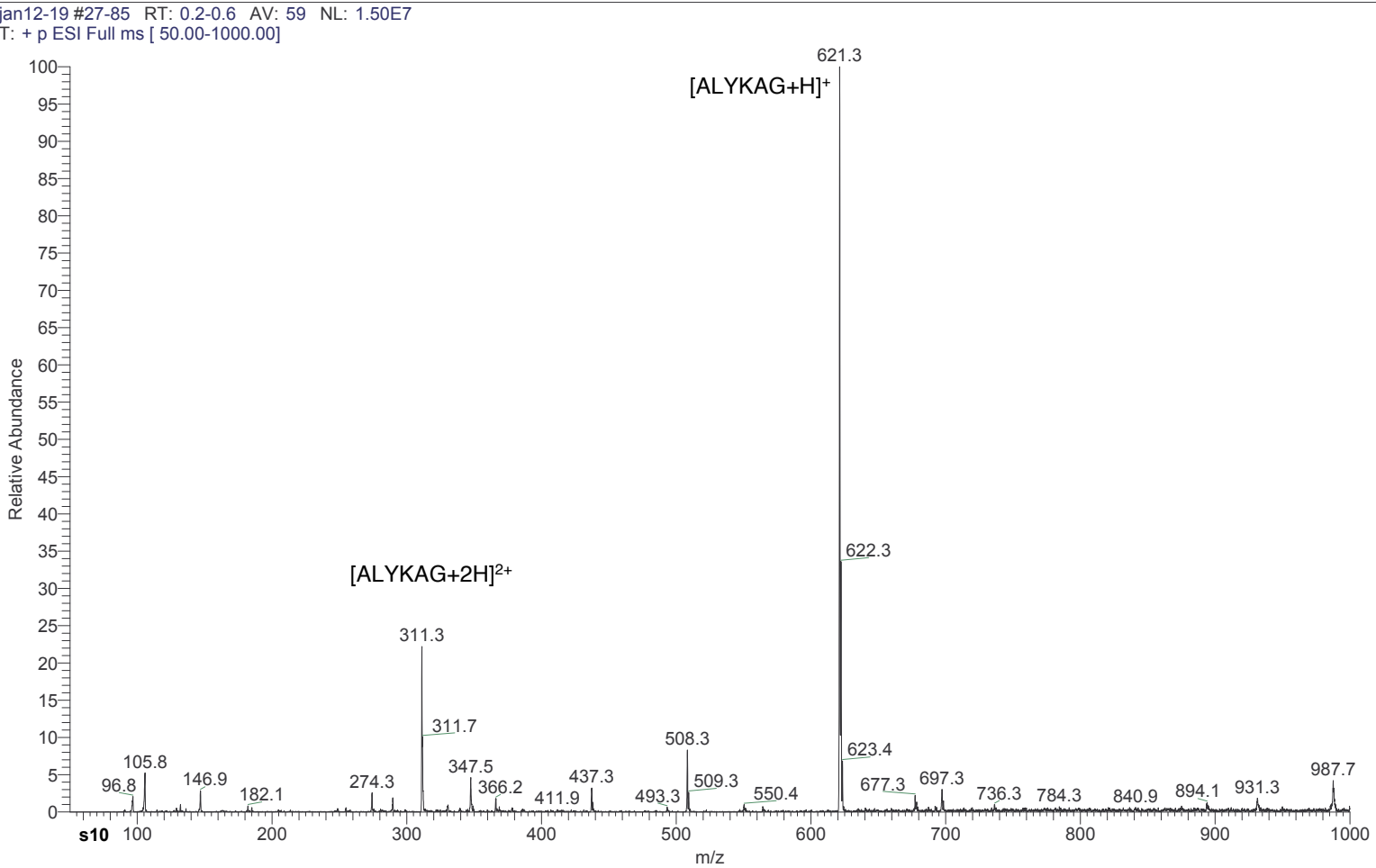
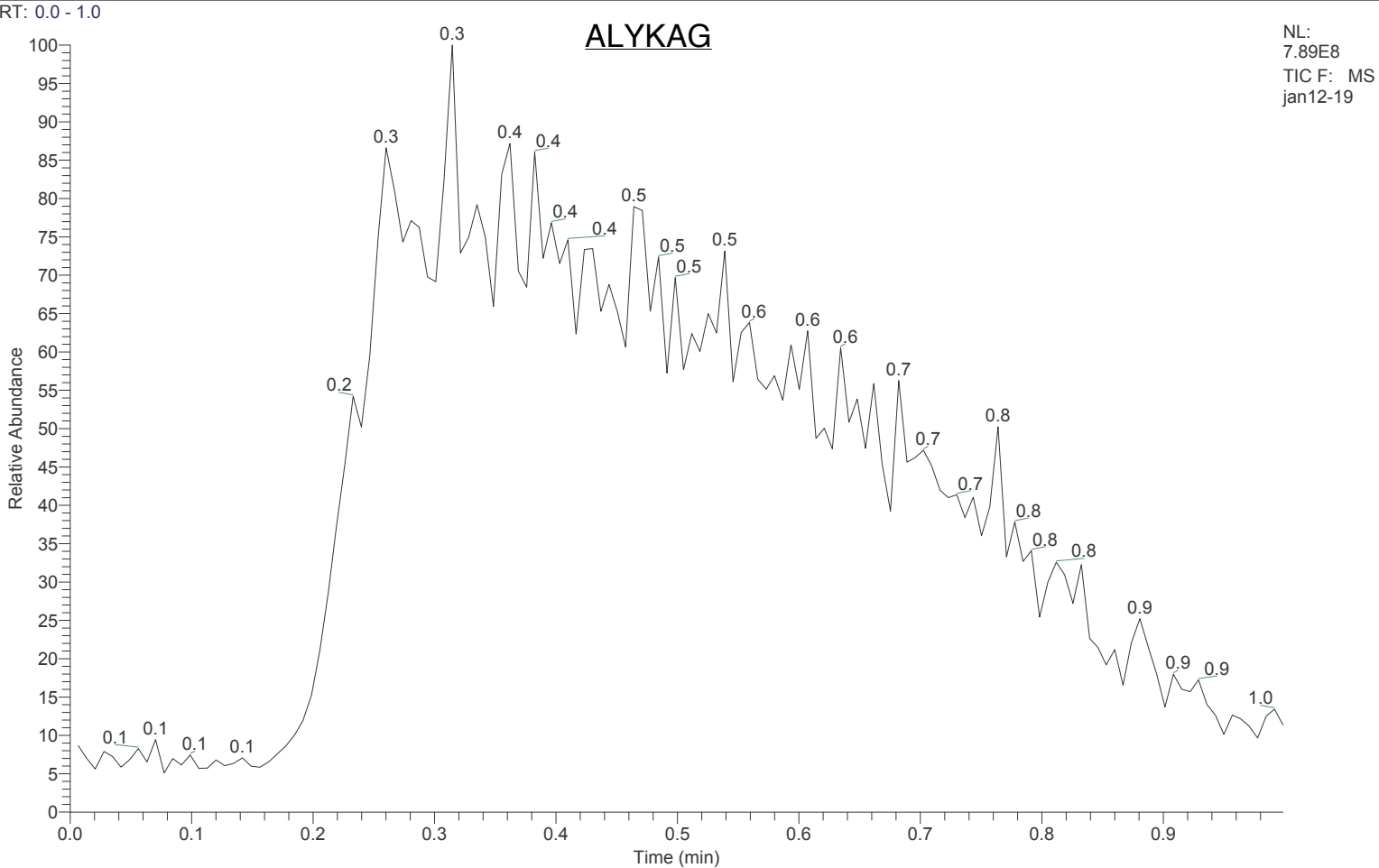
* Contains mixtures of disulfide-linked peptides

ⁱ It should be noted that is shelf-stable, but should not be kept as a concentrated aqueous solution for prolonged time due to risk of decomposition (Goddard-Borger, E. D.; Stick, R. V. *Org. Lett.* **2011**, *13*, 2514).

ALYKAG**==== Shimadzu LCsolution Analysis Report ====**

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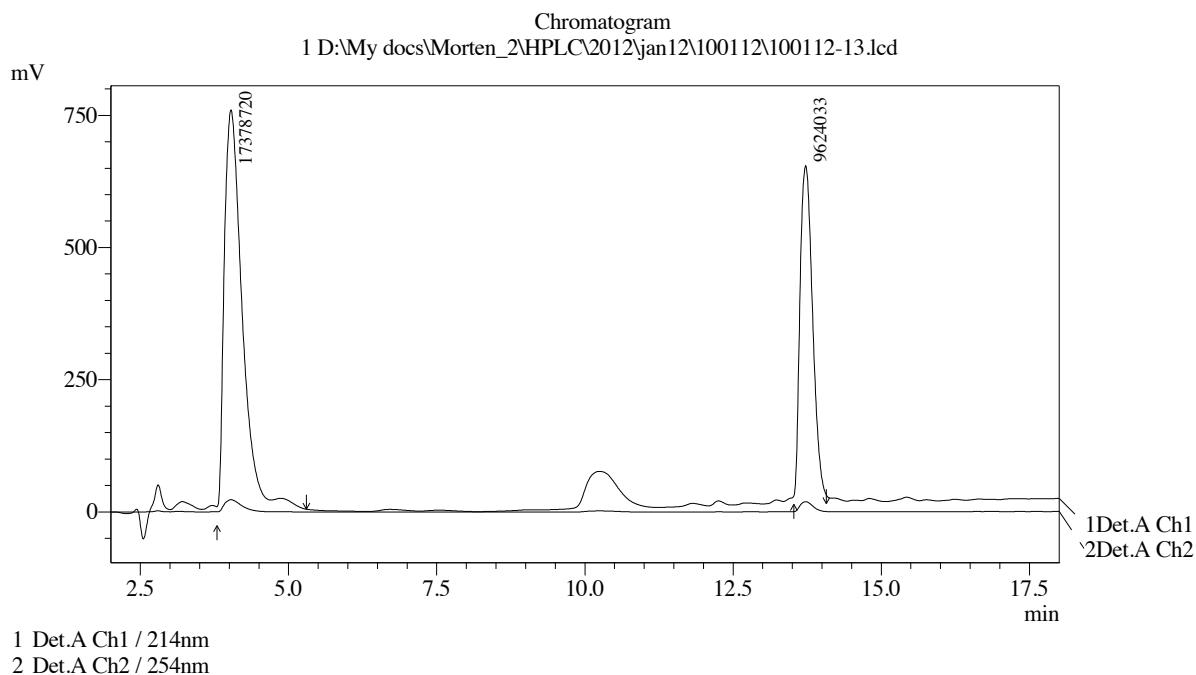
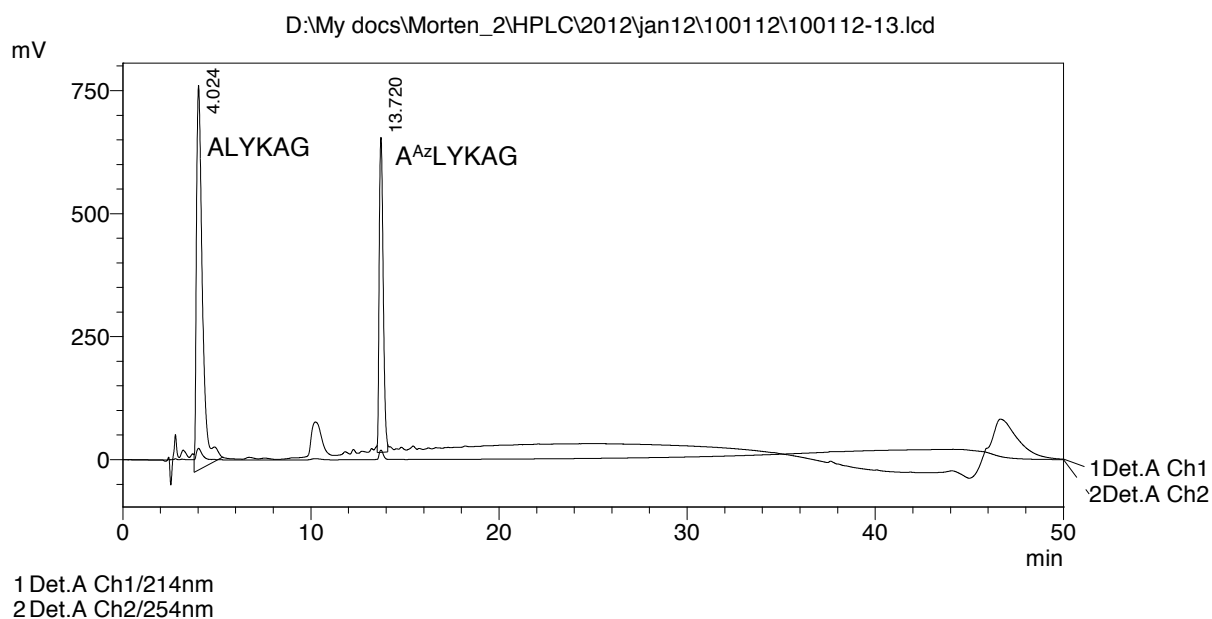


ALYKAG
NovaPEG resin, 1 min.

==== Shimadzu LCsolution Analysis Report ====

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Sample ID	: mbh421B	3 eq. imidazole-1-sulfonyl azide,
Tray#	: 1	4.5 eq. K ₂ CO ₃ , water, 1 min,
Vial #	: 14	NovaPEG resin
Injection Volume	: 10 uL	
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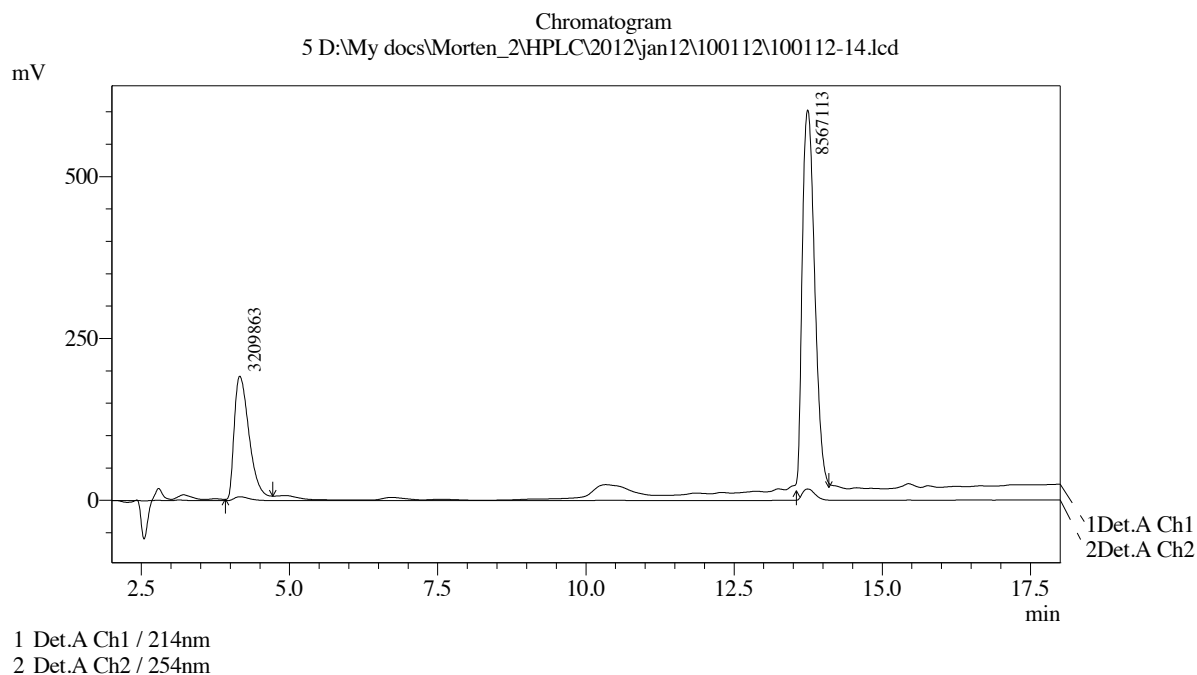
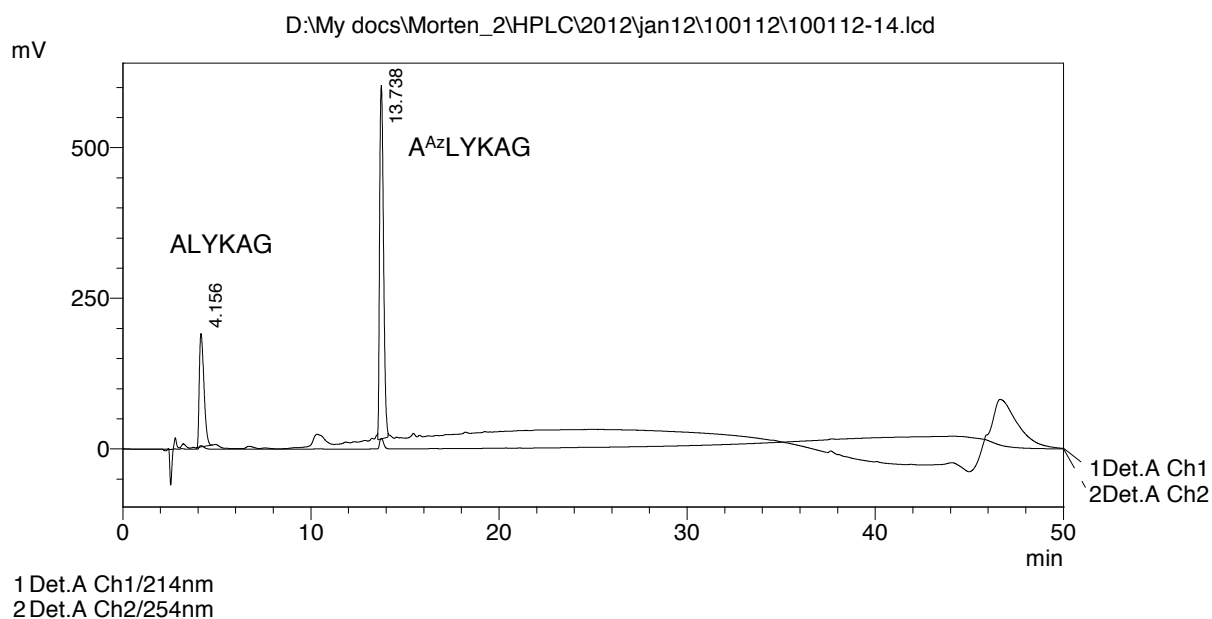


ALYKAG
NovaPEG resin, 5 min.

==== Shimadzu LCsolution Analysis Report ====

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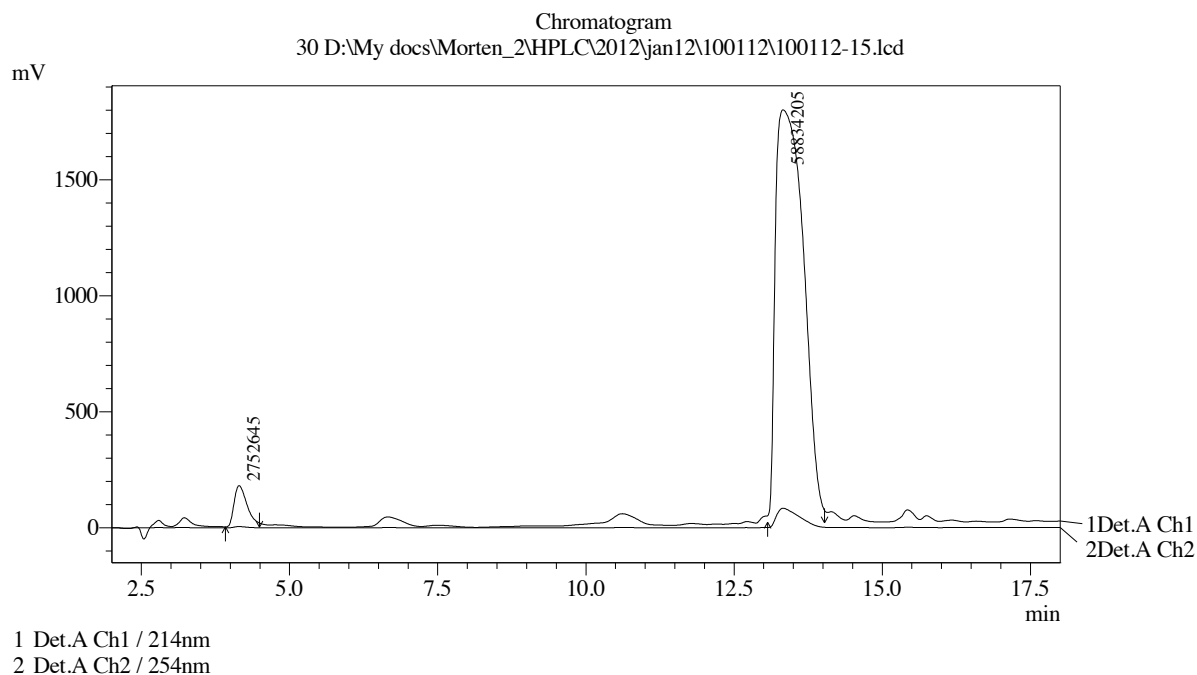
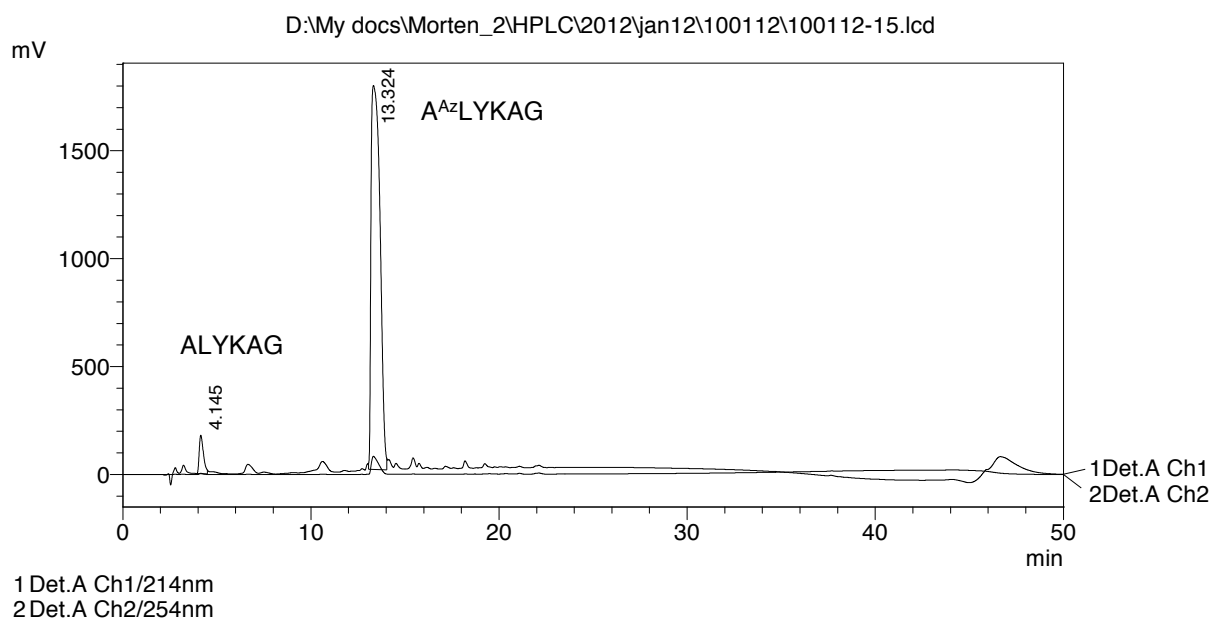
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4.5 eq. K_2CO_3 , water, 5 min,
NovaPEG resin



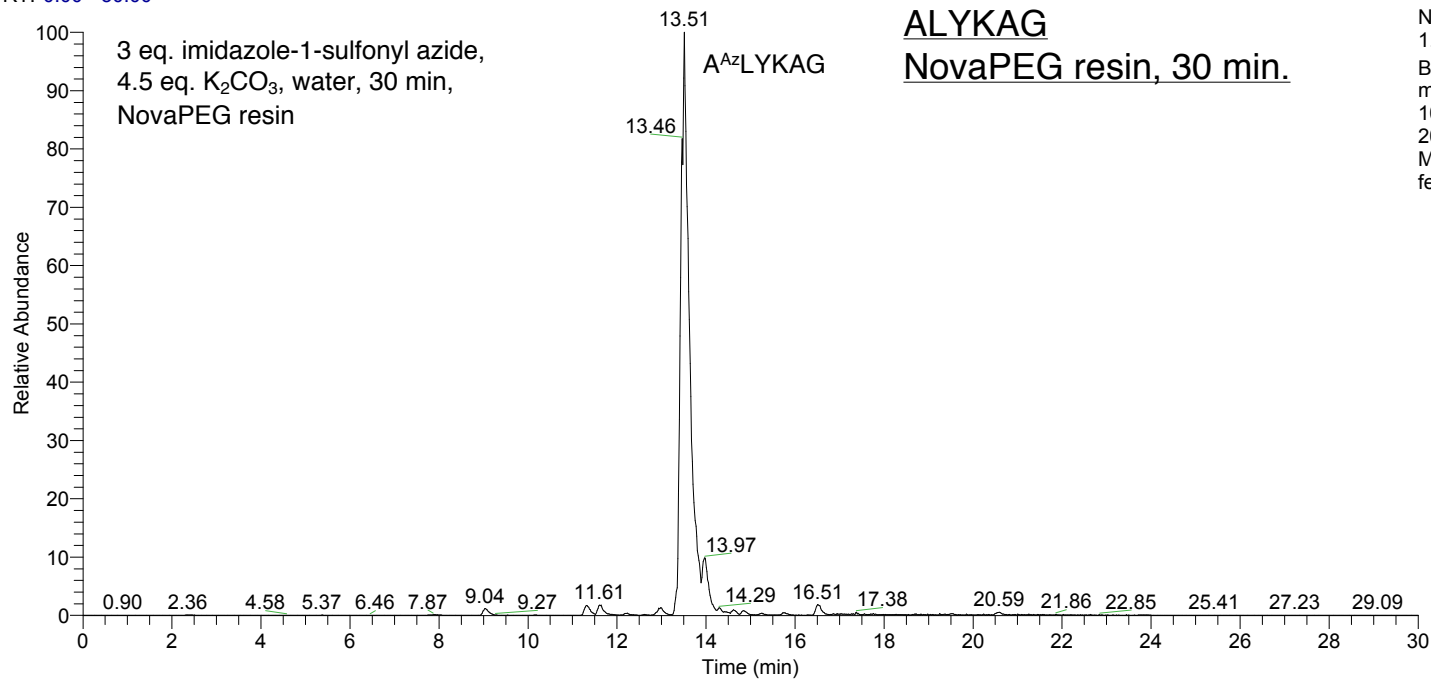
ALYKAGNovaPEG resin, 30 min.**==== Shimadzu LCsolution Analysis Report ====**

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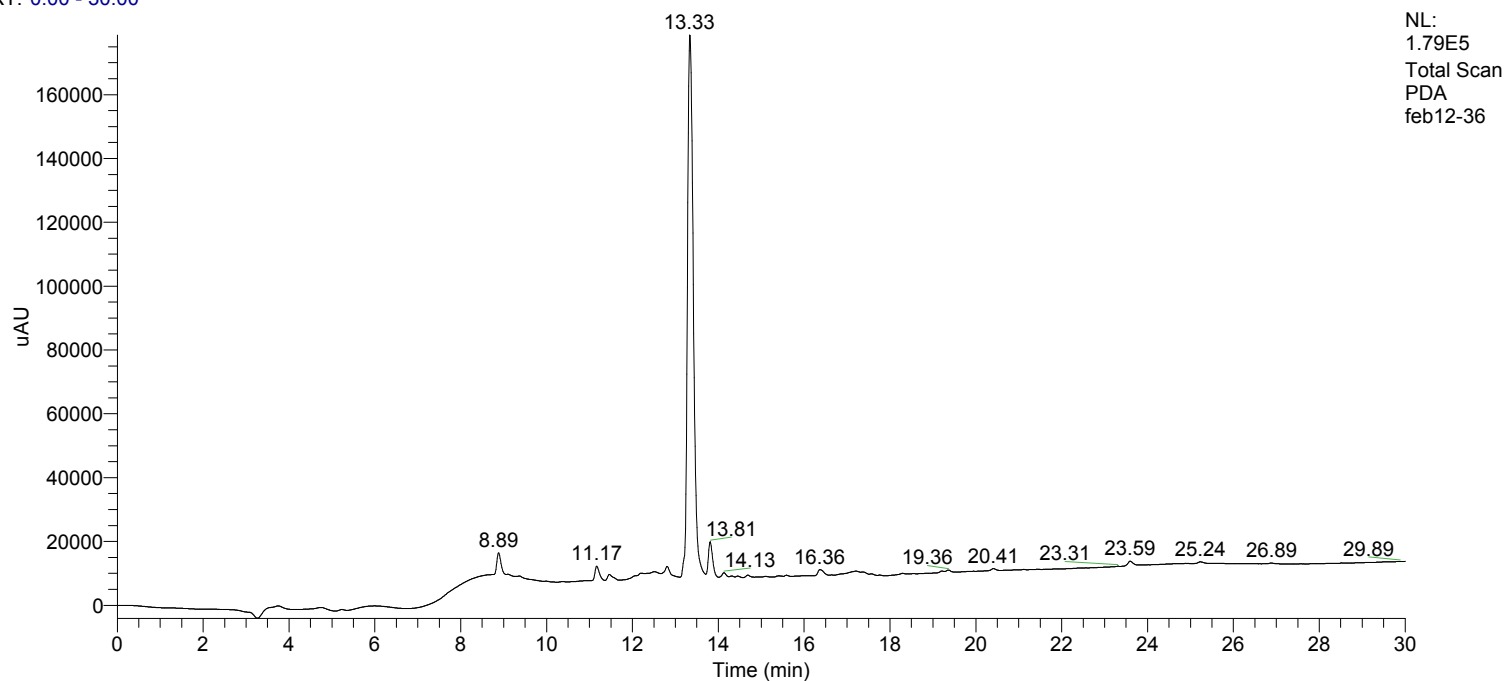
3 eq. imidazole-1-sulfonyl azide,
 4.5 eq. K₂CO₃, water, 30 min,
 NovaPEG resin



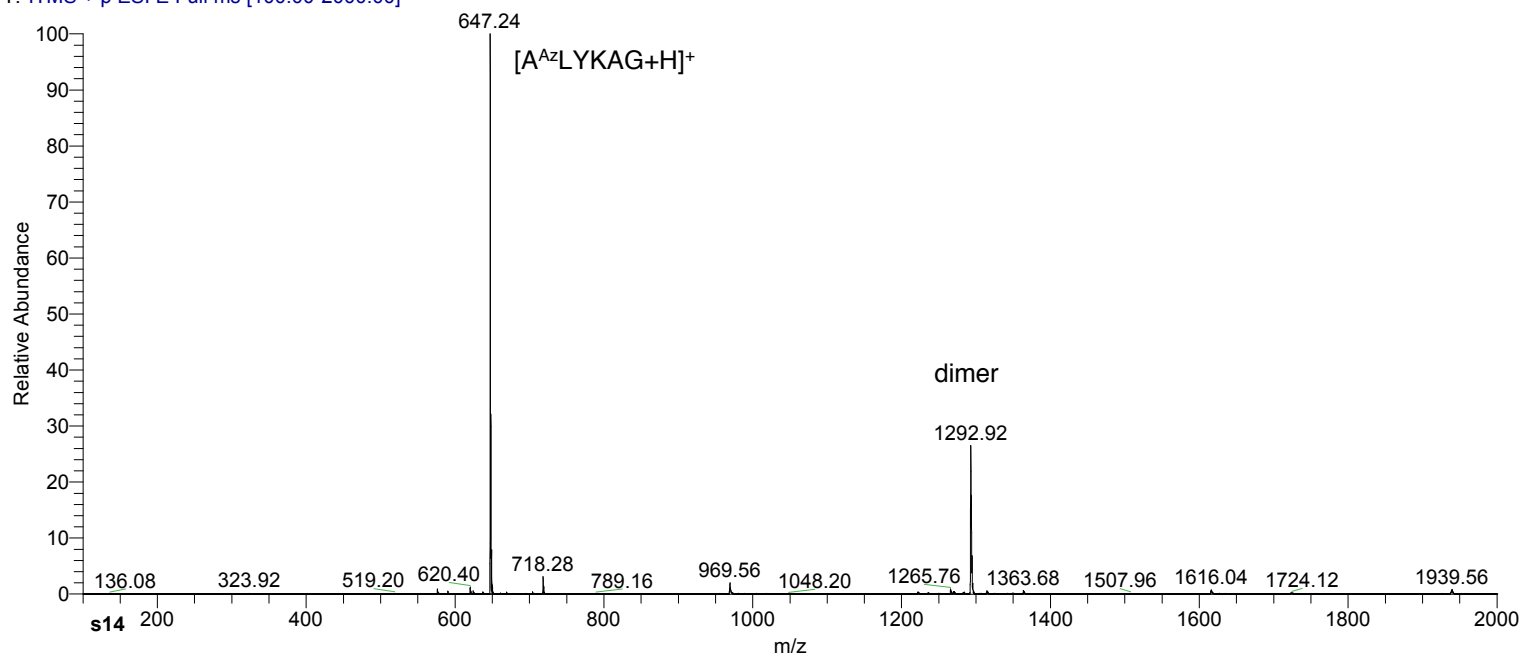
RT: 0.00 - 30.00



RT: 0.00 - 30.00

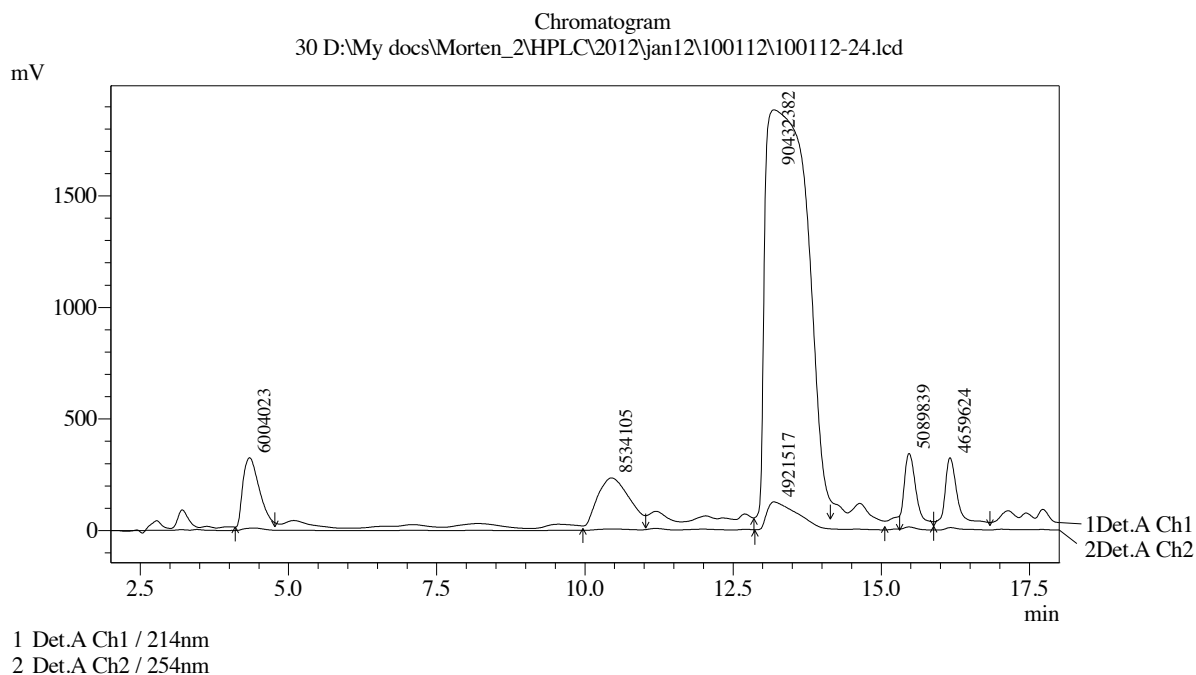
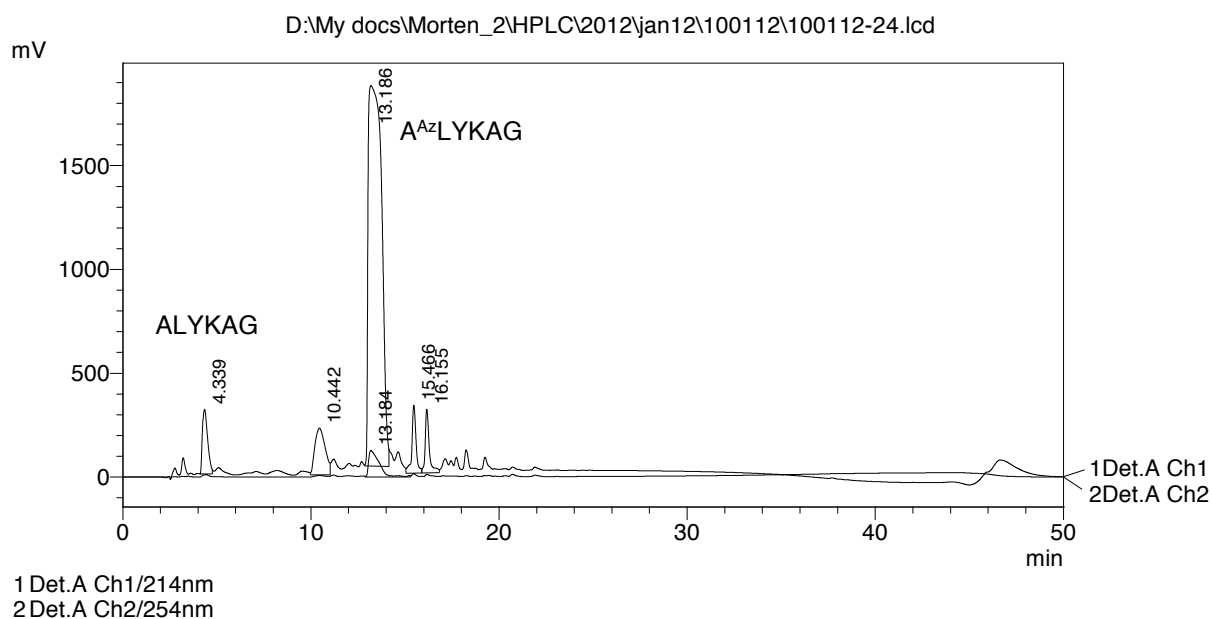


feb12-36 #707-756 RT: 13.27-14.07 AV: 50 NL: 4.46E4
T: ITMS + p ESI E Full ms [100.00-2000.00]

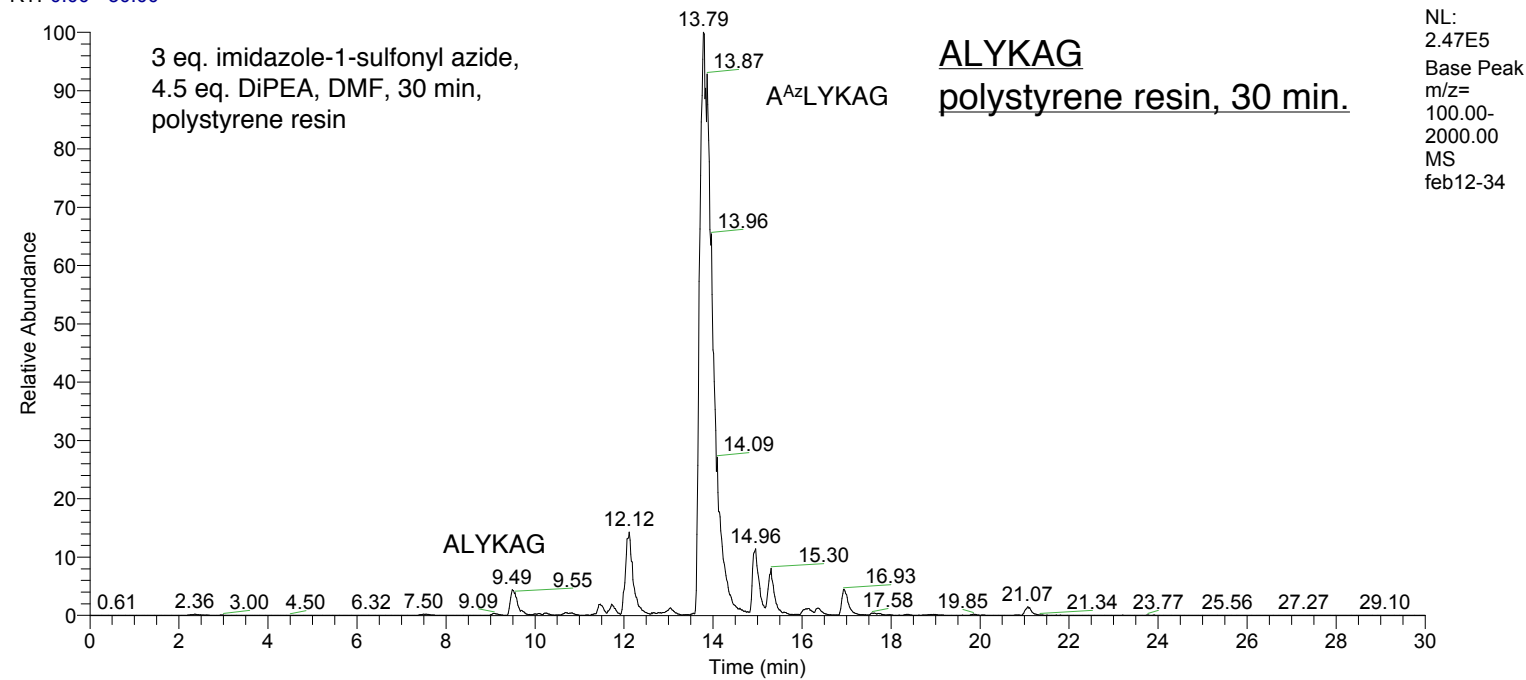


ALYKAG**polystyrene resin, 30 min.****==== Shimadzu LCsolution Analysis Report ====**

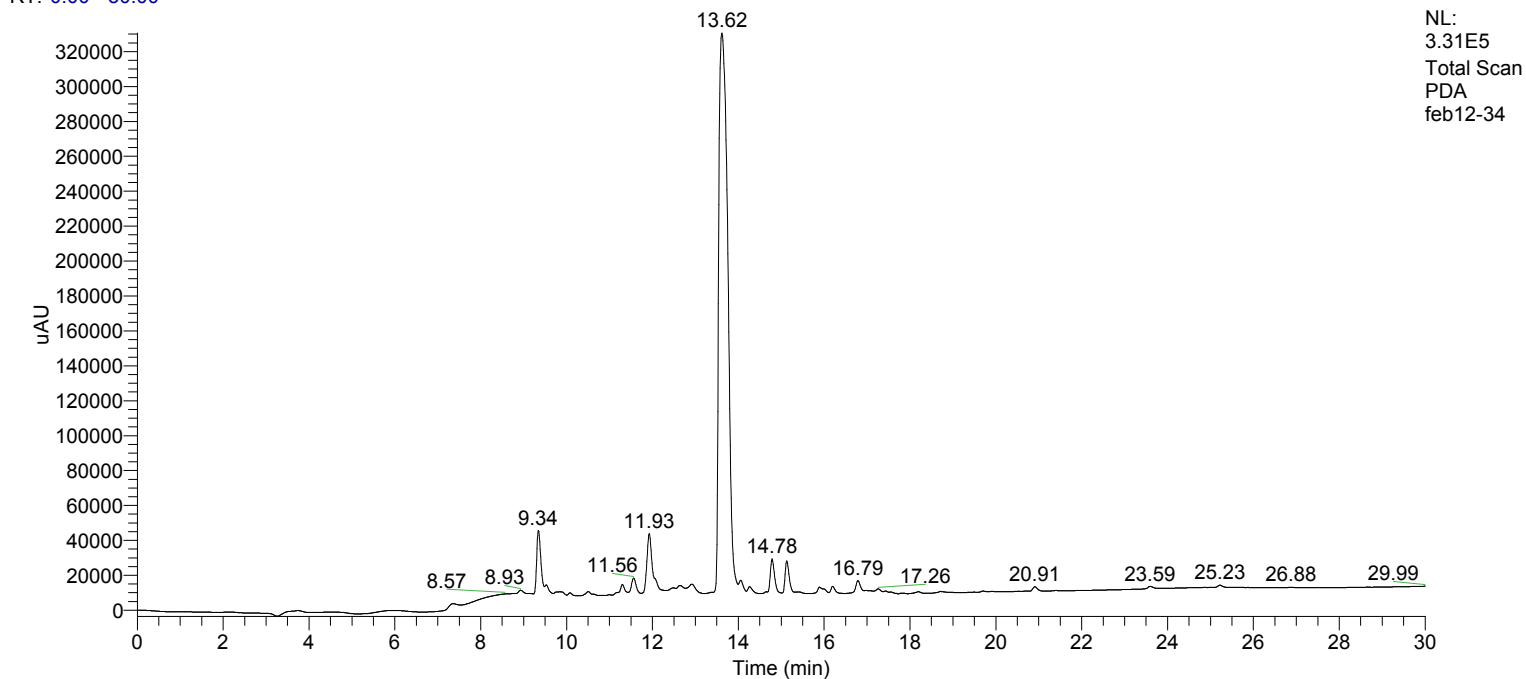
D:\My docs\Morten_2\HPLC\2012\jan12\100112\100112-24.lcd
 Acquired by : user
 Sample Name : 30 3 eq. imidazole-1-sulfonyl azide,
 Sample ID : mbh422B 4.5 eq. DiPEA, DMF, 30 min,
 Tray# : 1 polystyrene resin
 Vial # : 25
 Injection Volume : 10 uL
 Data File Name : 100112-24.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 100112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 11-Jan-12 15:27:14
 Data Processed : 17-Jan-12 15:52:59



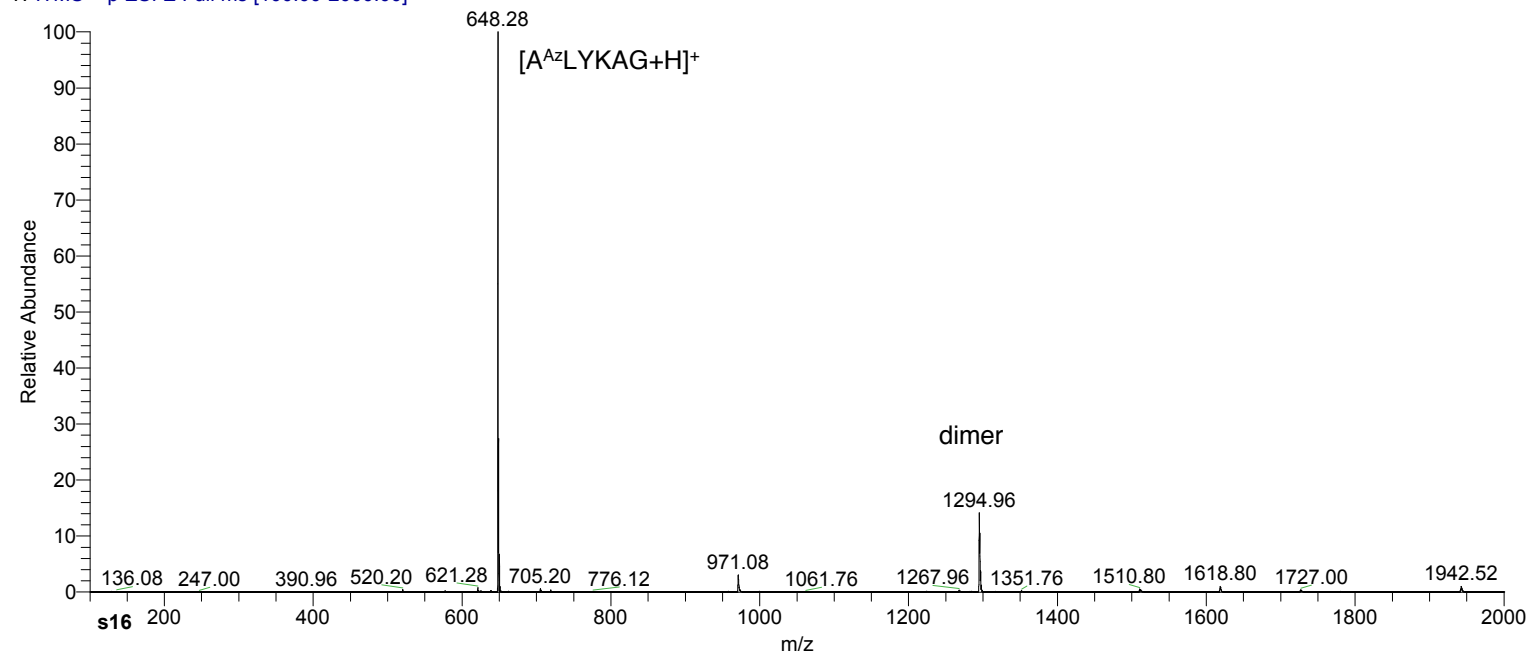
RT: 0.00 - 30.00



RT: 0.00 - 30.00

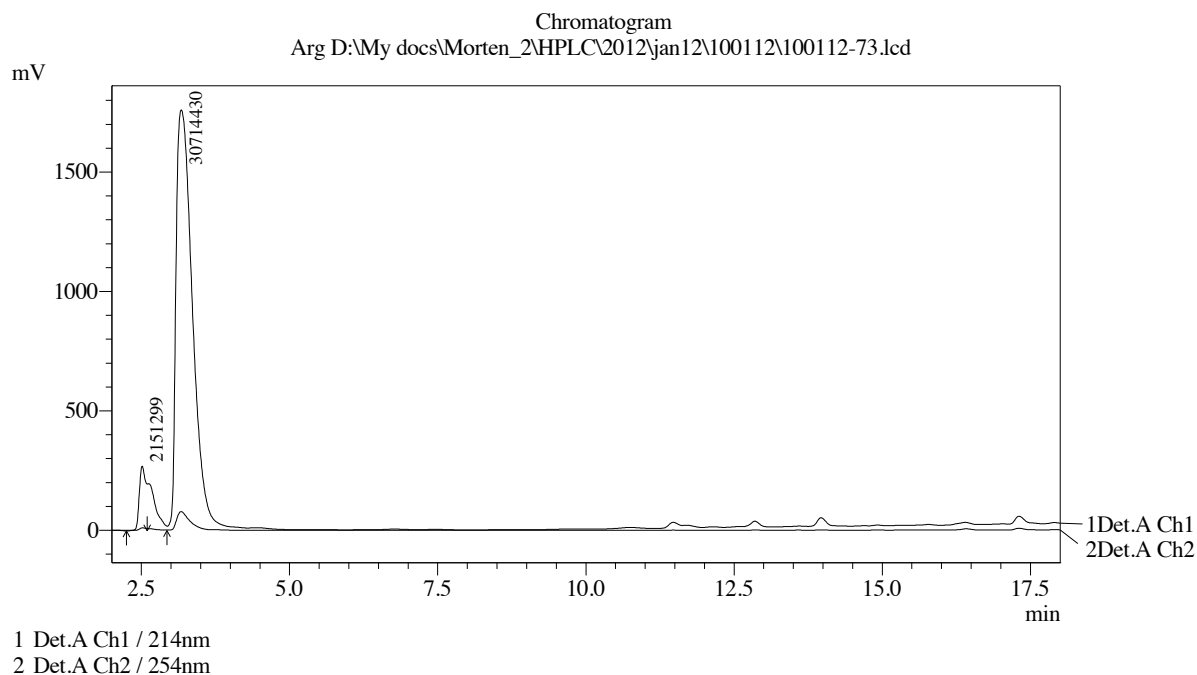
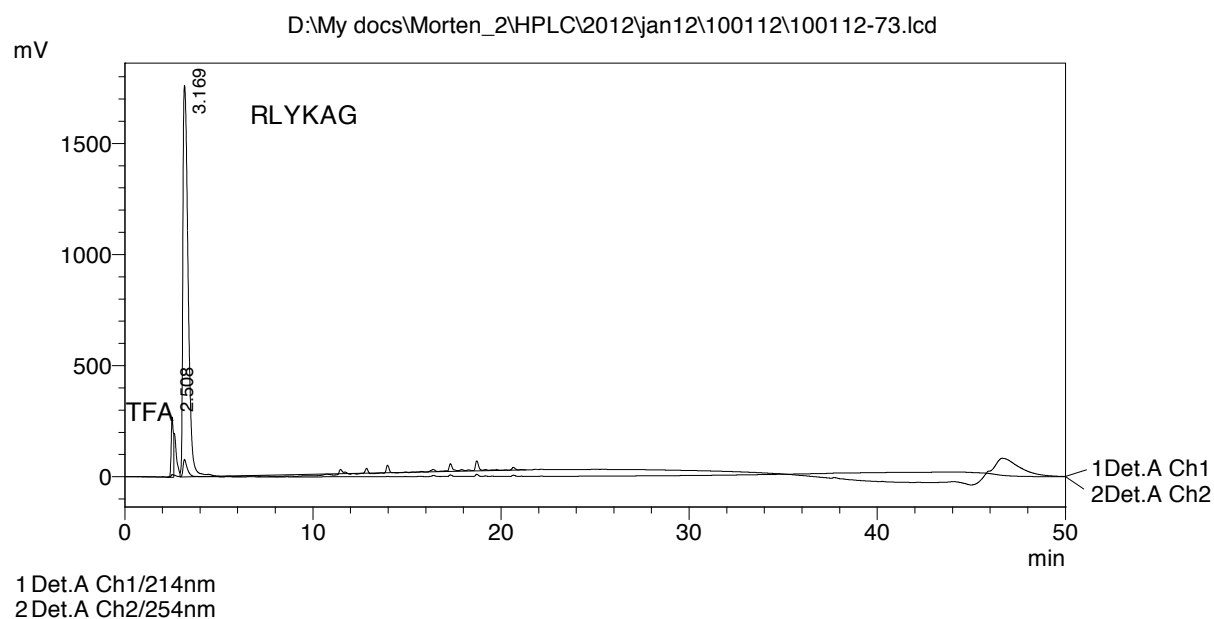


feb12-34 #753-777 RT: 13.70-14.09 AV: 25 NL: 1.68E5
T: ITMS + p ESI E Full ms [100.00-2000.00]

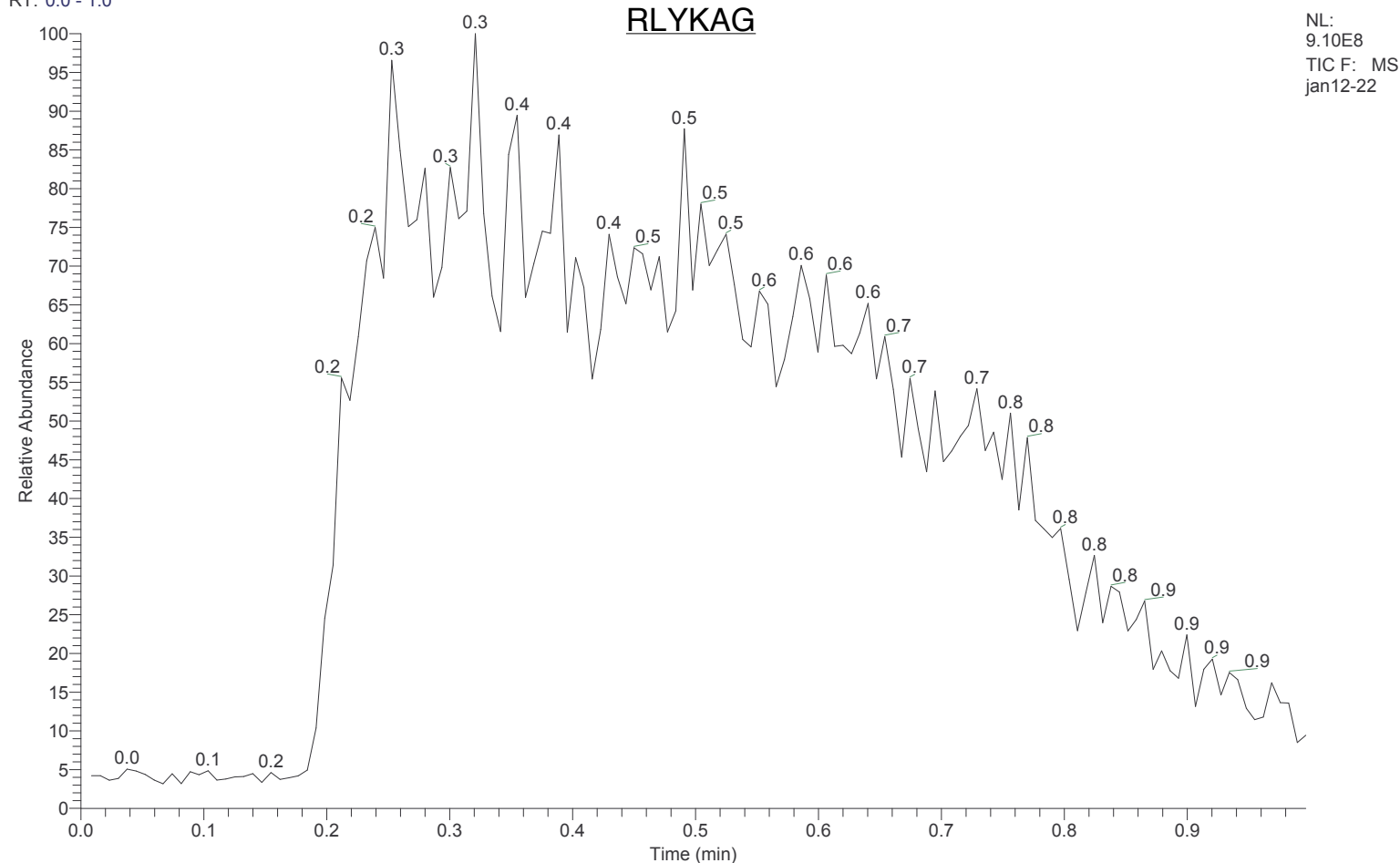
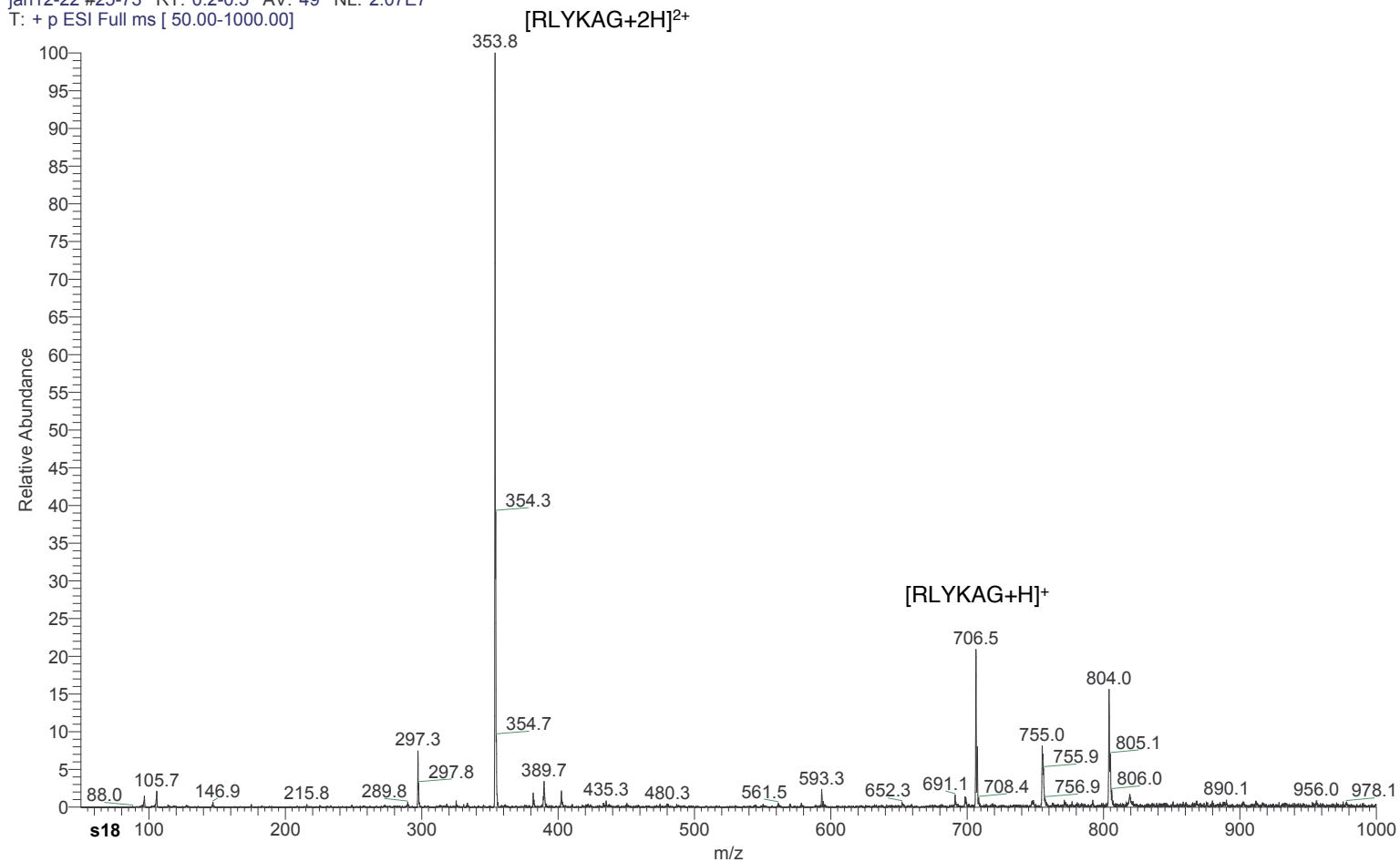


RLYKAG**==== Shimadzu LCsolution Analysis Report ====**

D:\My docs\Morten_2\HPLC\2012\jan12\100112\100112-73.lcd
 Acquired by : user
 Sample Name : Arg
 Sample ID : bef. diazotization
 Tray# : 1
 Vial # : 91
 Injection Volume : 10 uL
 Data File Name : 100112-73.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 100112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 13-Jan-12 10:56:41
 Data Processed : 13-Jan-12 11:46:51

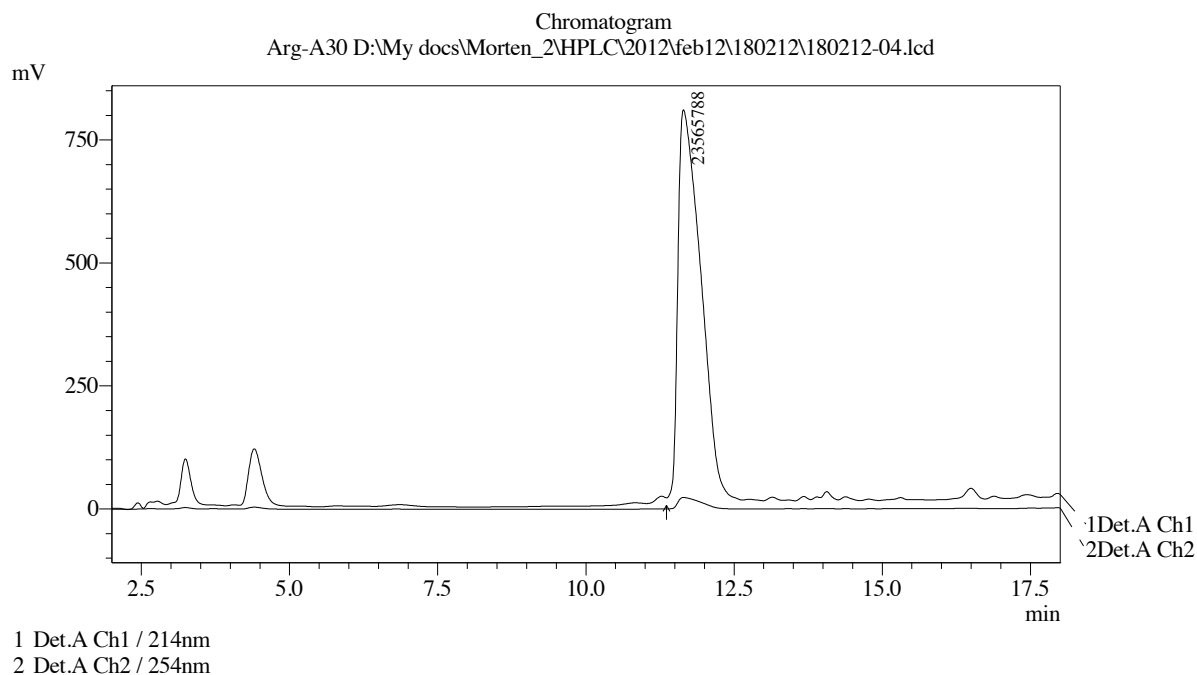
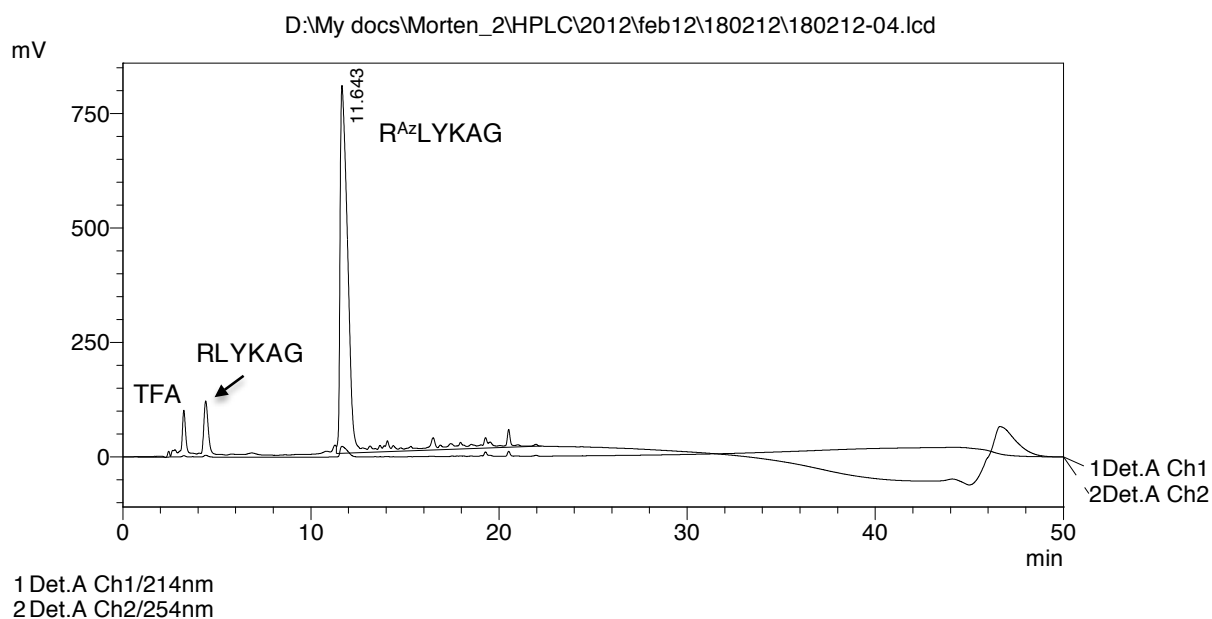


RT: 0.0 - 1.0

jan12-22 #25-73 RT: 0.2-0.5 AV: 49 NL: 2.07E7
T: + p ESI Full ms [50.00-1000.00]

RLYKAG**==== Shimadzu LCsolution Analysis Report ====**

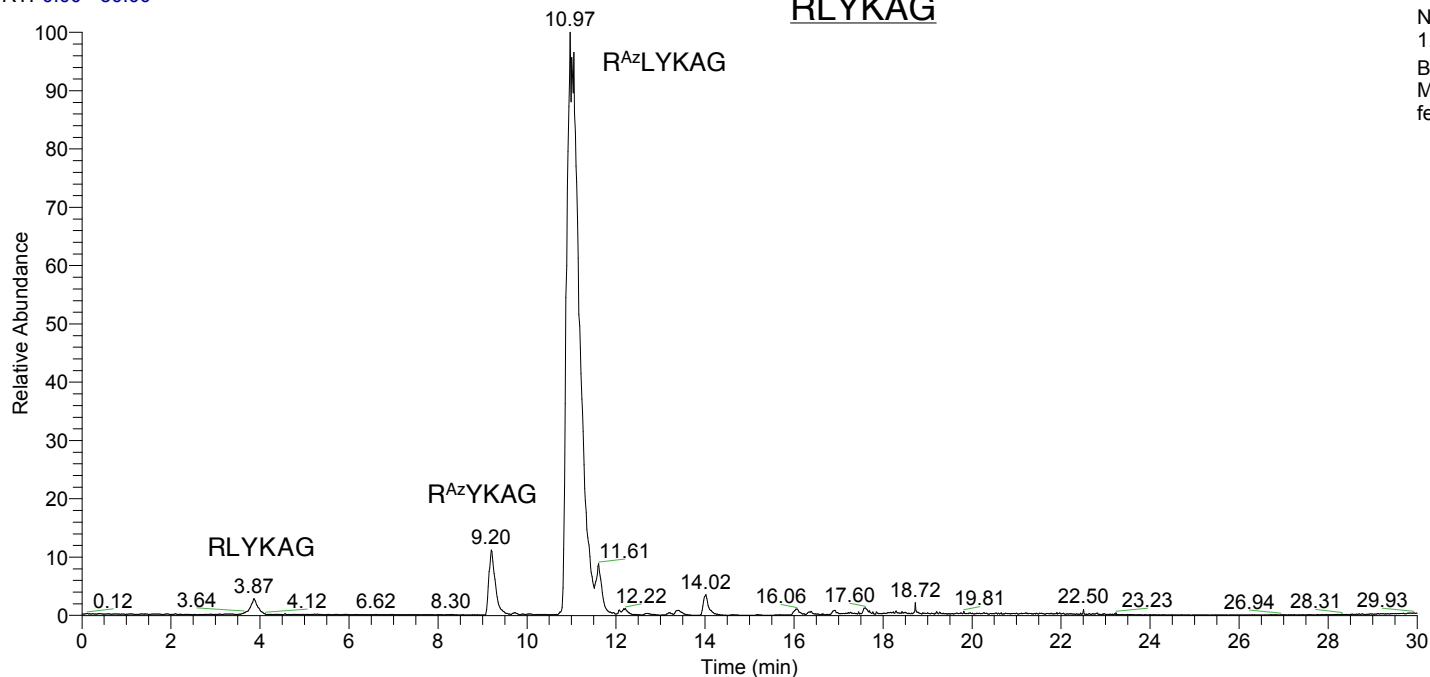
D:\My docs\Morten_2\HPLC\2012\feb12\180212\180212-04.lcd
 Acquired by : guest user
 Sample Name : Arg-A30
 Sample ID :
 Tray# : 1
 Vial # : 71
 Injection Volume : 3 uL
 Data File Name : 180212-04.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 20120218.lcb
 Report File Name : Default.lcr
 Data Acquired : 19-Feb-12 0:37:19
 Data Processed : 19-Feb-12 1:27:24



RT: 0.00 - 30.00

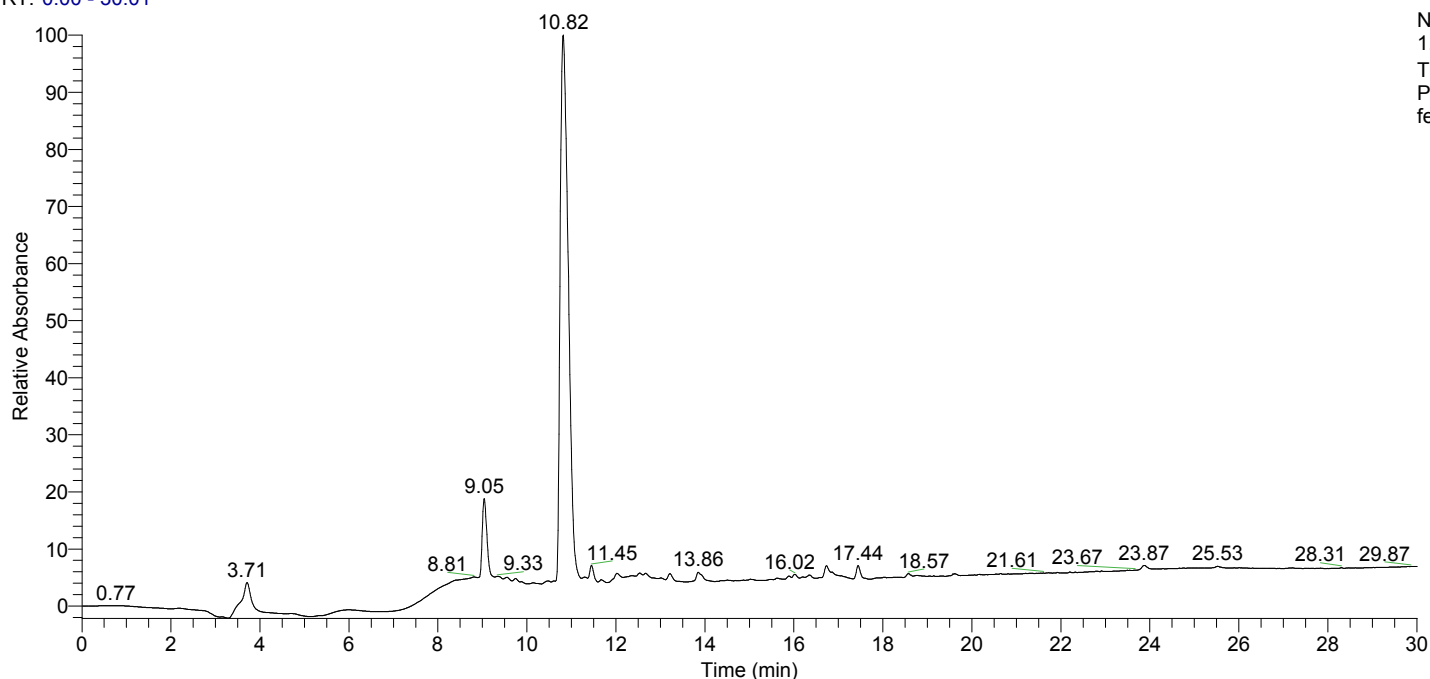
RLYKAG

NL:
1.60E5
Base Peak
MS
feb12-03

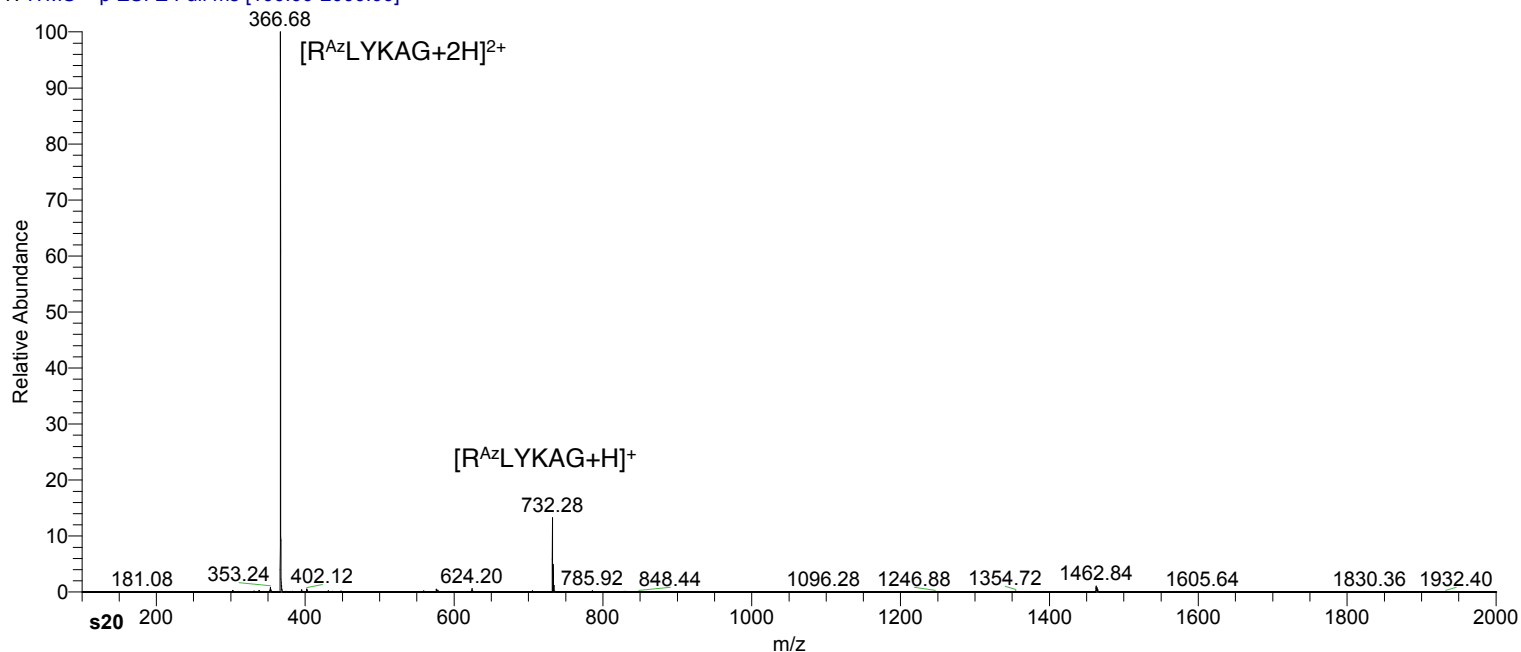


RT: 0.00 - 30.01

NL:
1.83E5
Total Scan
PDA
feb12-03



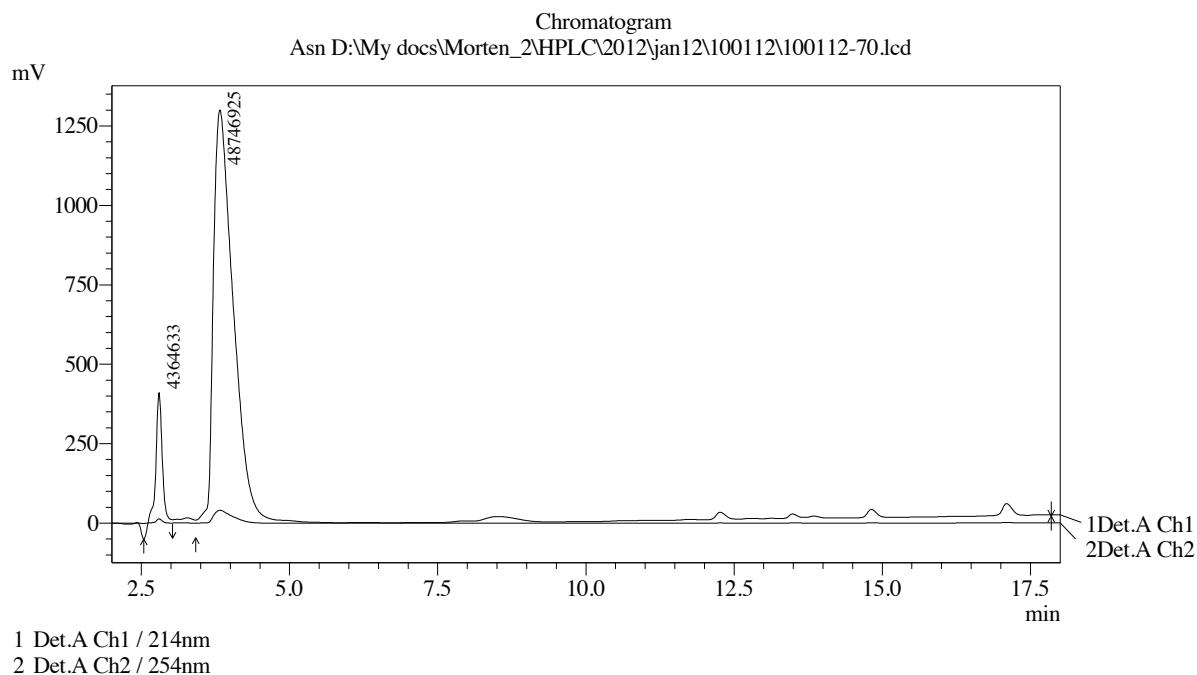
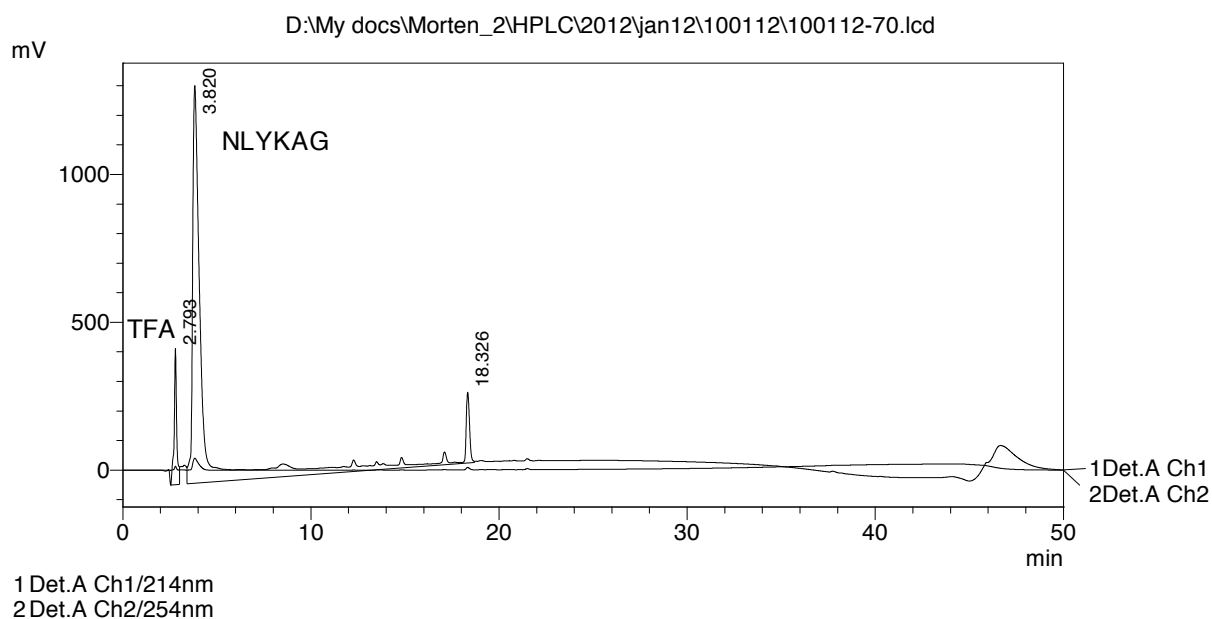
feb12-03 #577-635 RT: 10.76-11.71 AV: 59 NL: 5.33E4
T: ITMS + p ESI E Full ms [100.00-2000.00]



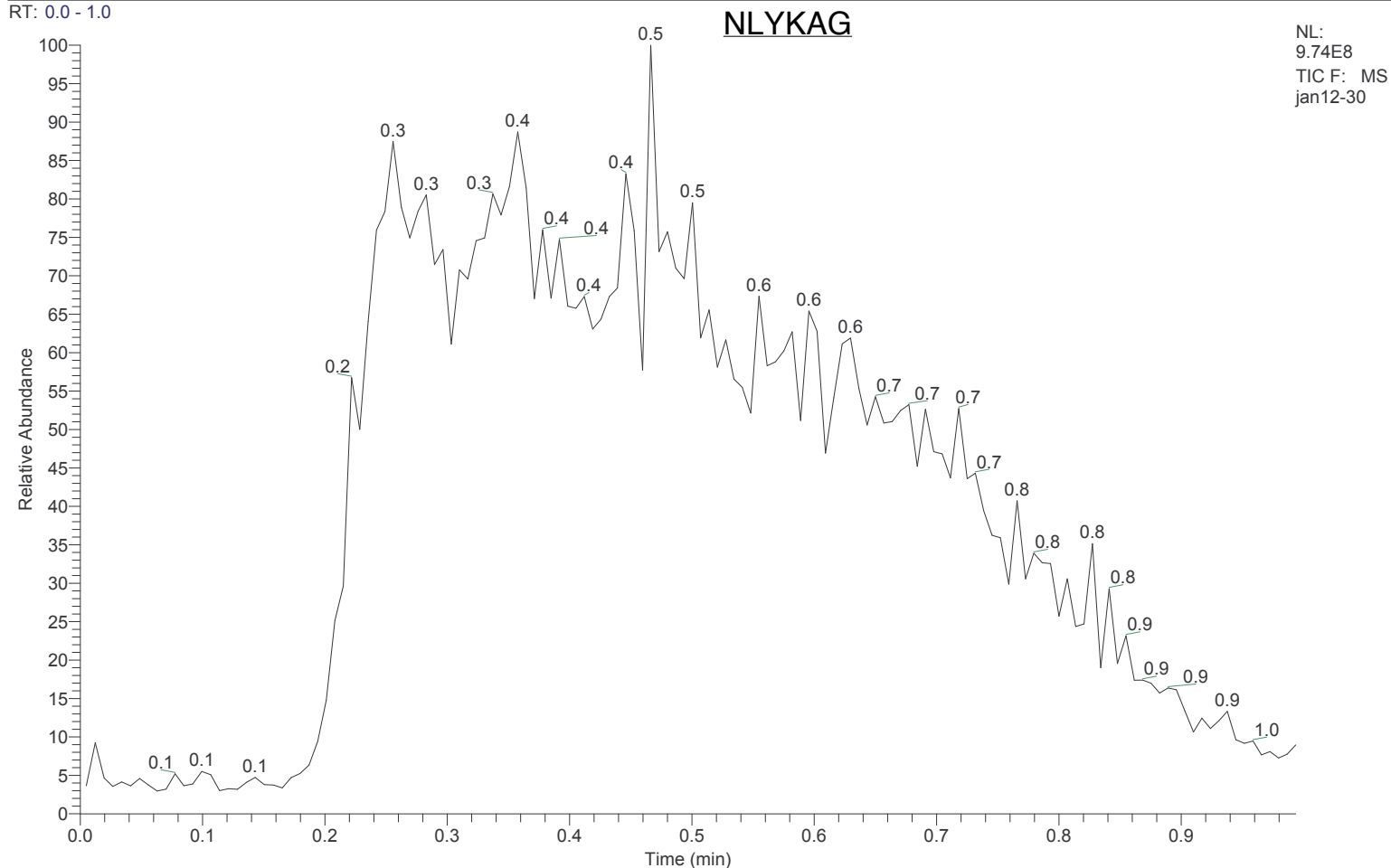
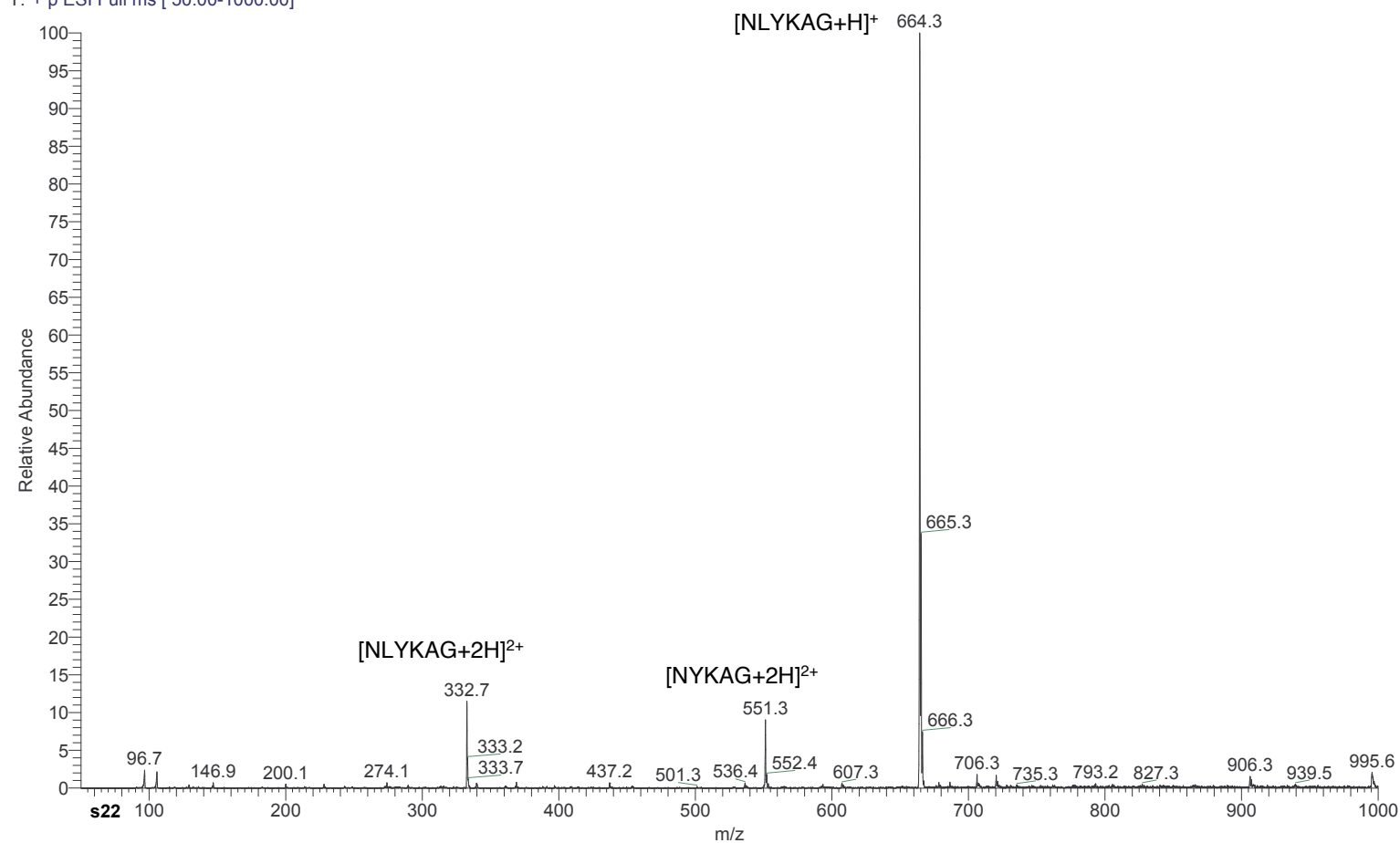
NLYKAG

==== Shimadzu LCsolution Analysis Report ====

D:\My docs\Morten_2\HPLC\2012\jan12\100112\100112-70.lcd
 Acquired by : user
 Sample Name : Asn
 Sample ID : bef. diazotization
 Tray# : 1
 Vial # : 88
 Injection Volume : 10 uL
 Data File Name : 100112-70.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 100112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 13-Jan-12 8:25:18
 Data Processed : 13-Jan-12 9:15:22



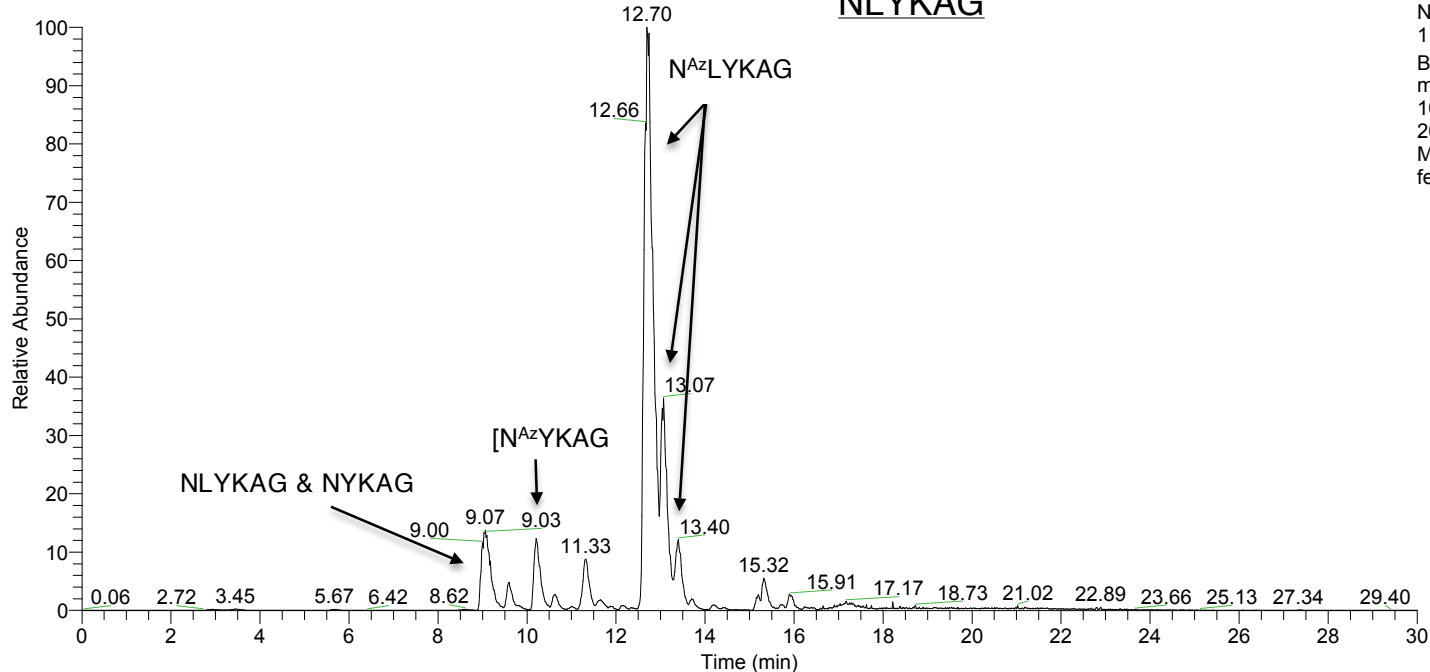
RT: 0.0 - 1.0

jan12-30 #25-88 RT: 0.2-0.6 AV: 64 NL: 2.59E7
T: + p ESI Full ms [50.00-1000.00]

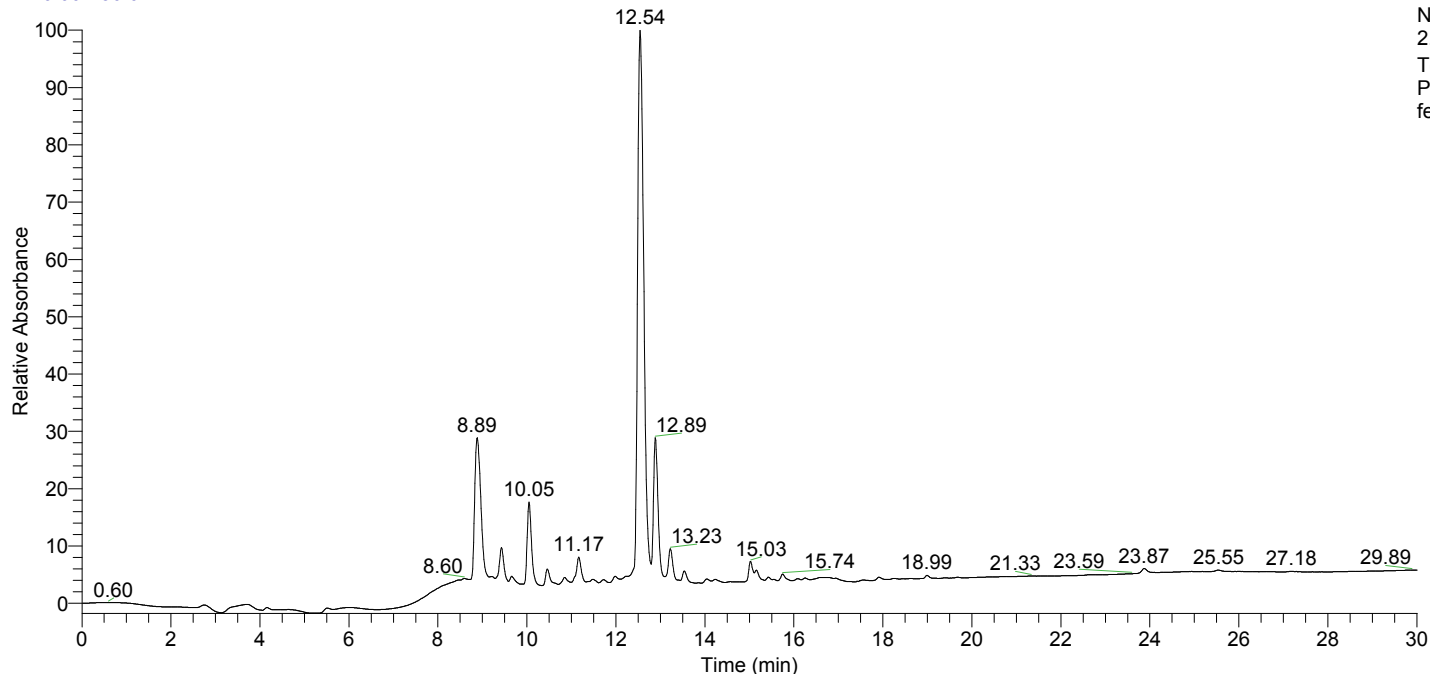
RT: 0.00 - 30.00

NLYKAG

NL:
1.50E5
Base Peak
m/z=
100.00-
2000.00
MS
feb12-15

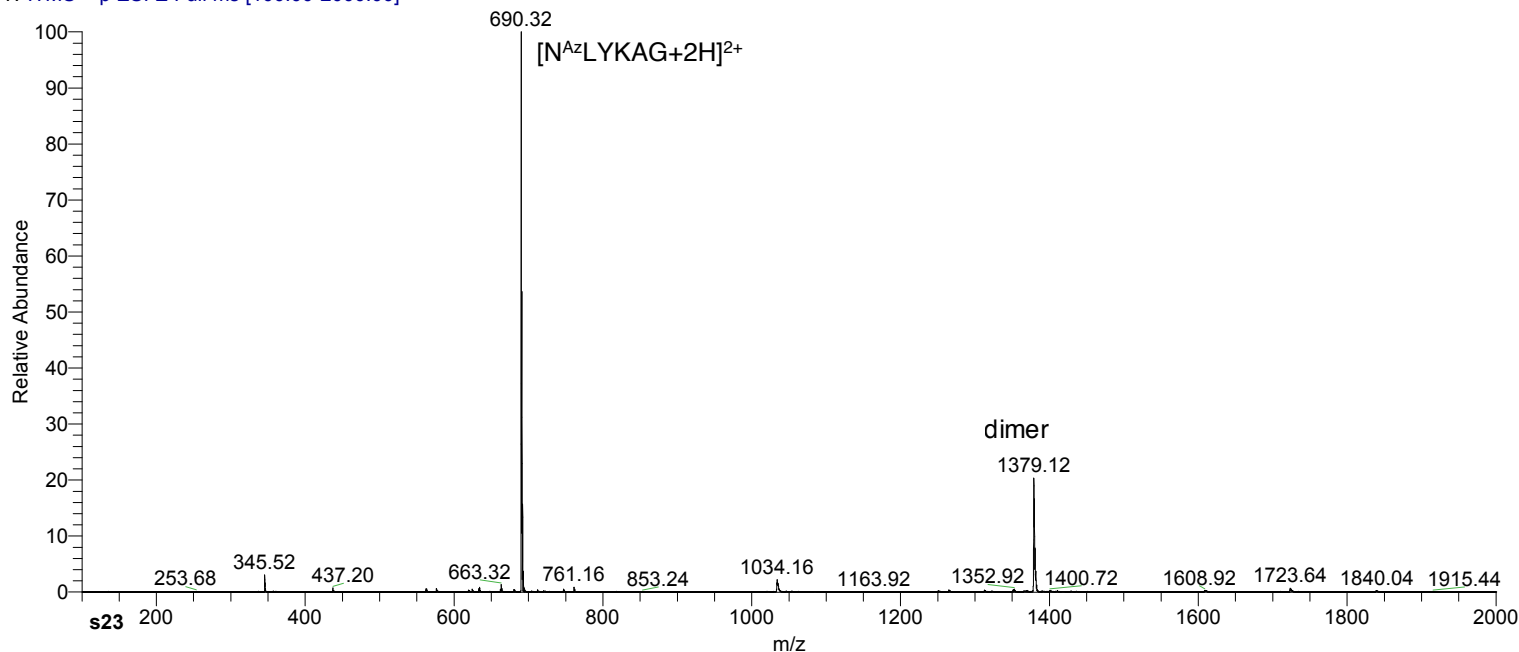


RT: 0.00 - 30.01



NL:
2.15E5
Total Scan
PDA
feb12-15

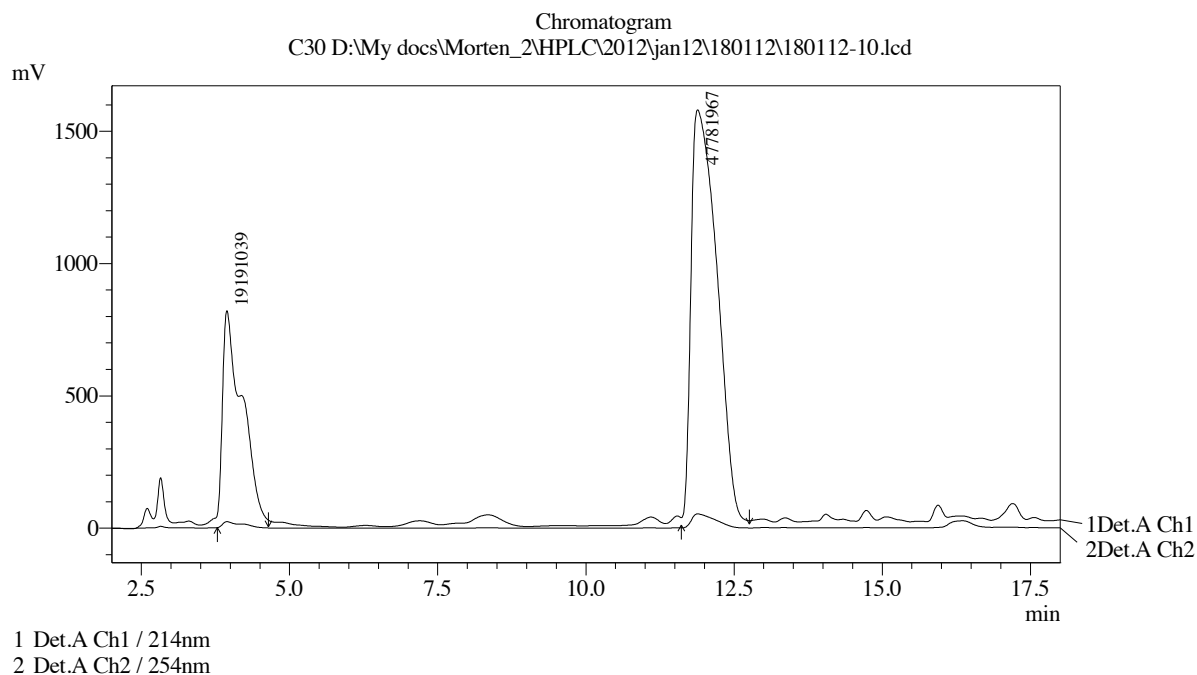
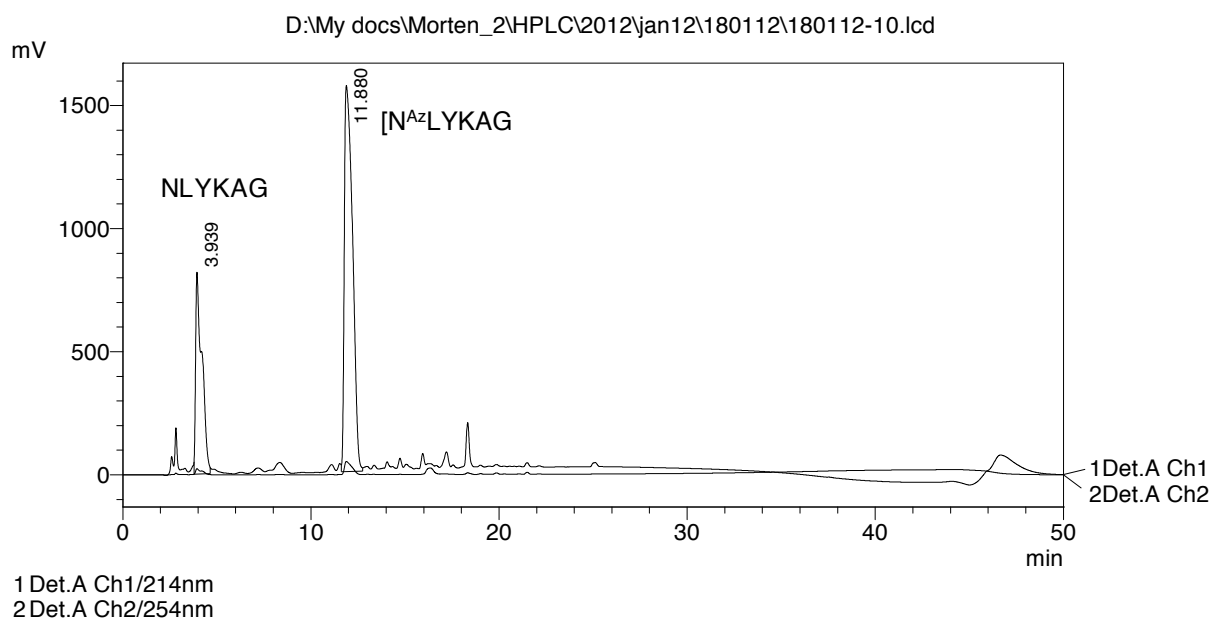
feb12-15 #673-741 RT: 12.41-13.53 AV: 69 NL: 3.23E4
T: ITMS + p ESI E Full ms [100.00-2000.00]



NLYKAG

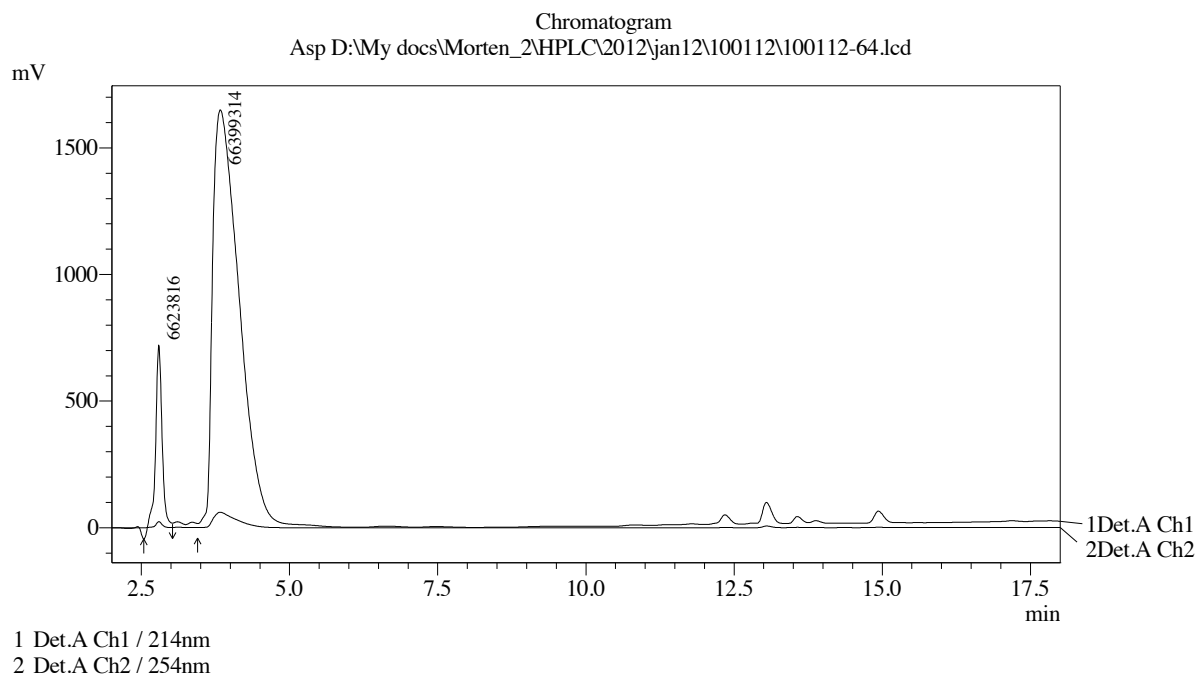
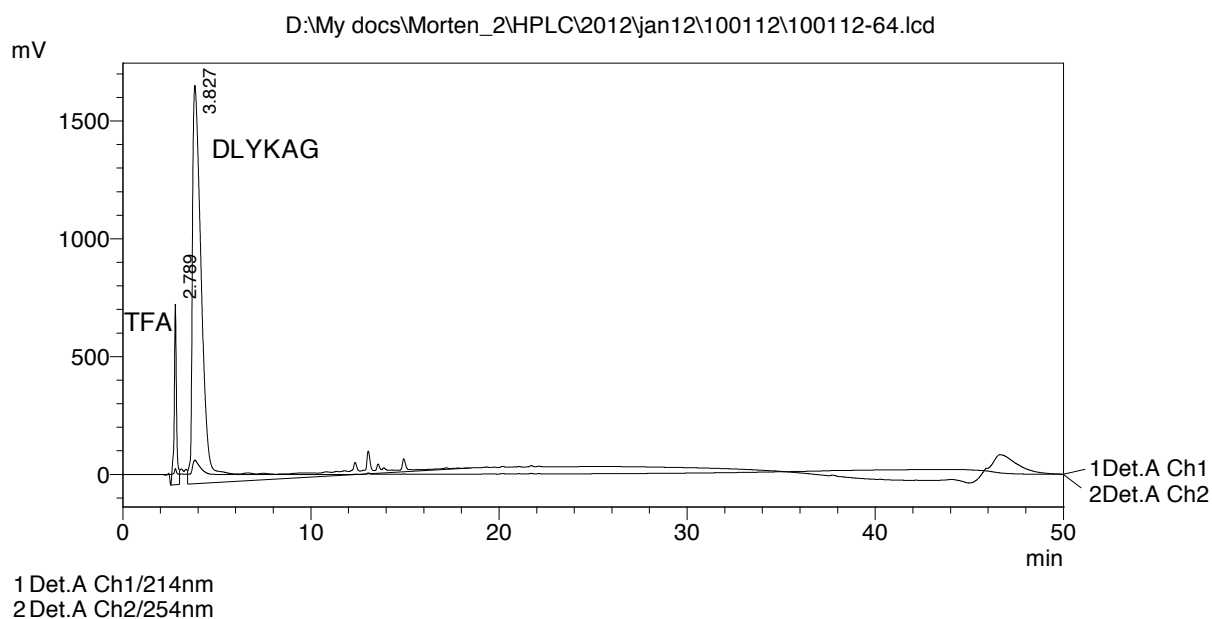
==== Shimadzu LCsolution Analysis Report ====

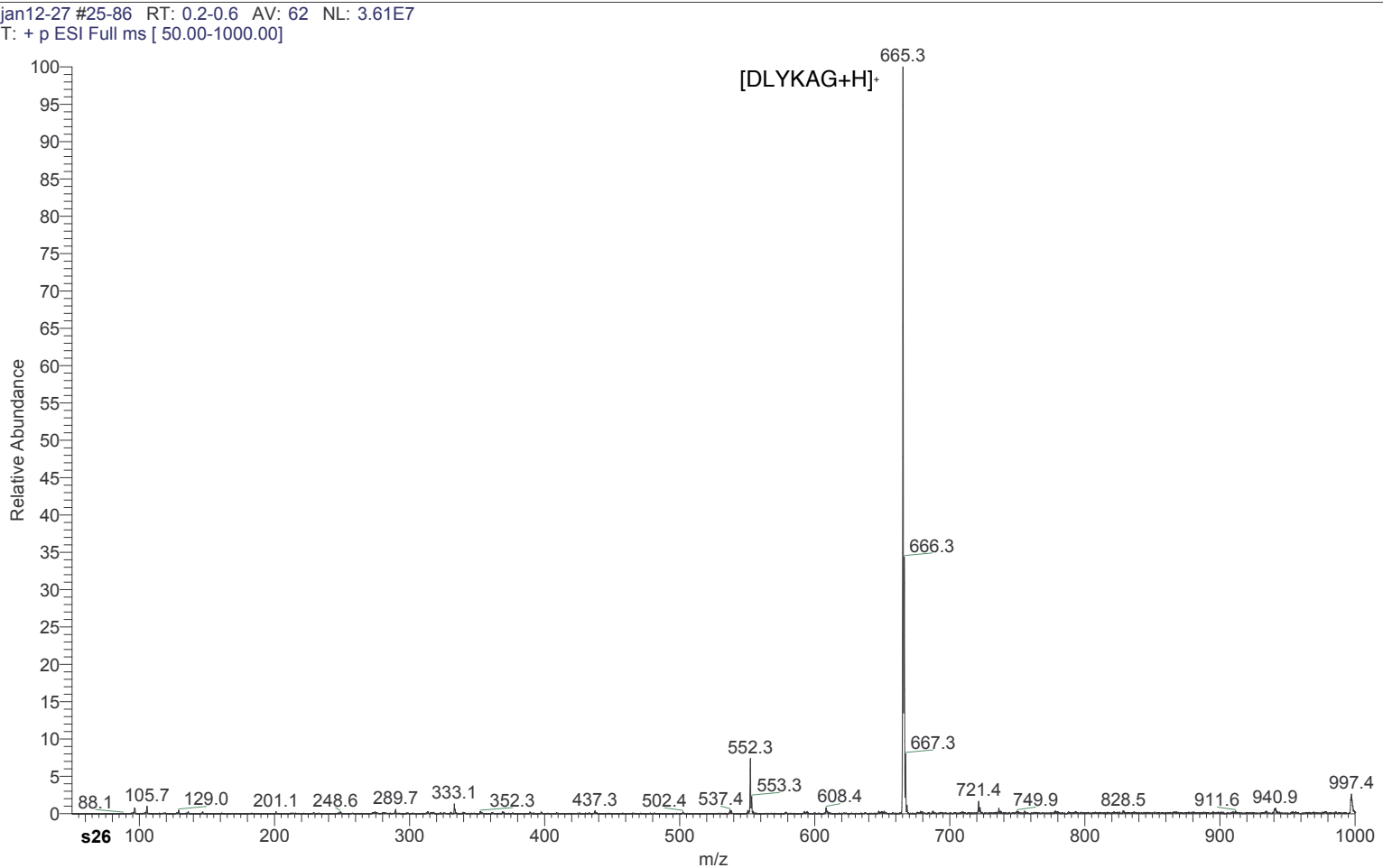
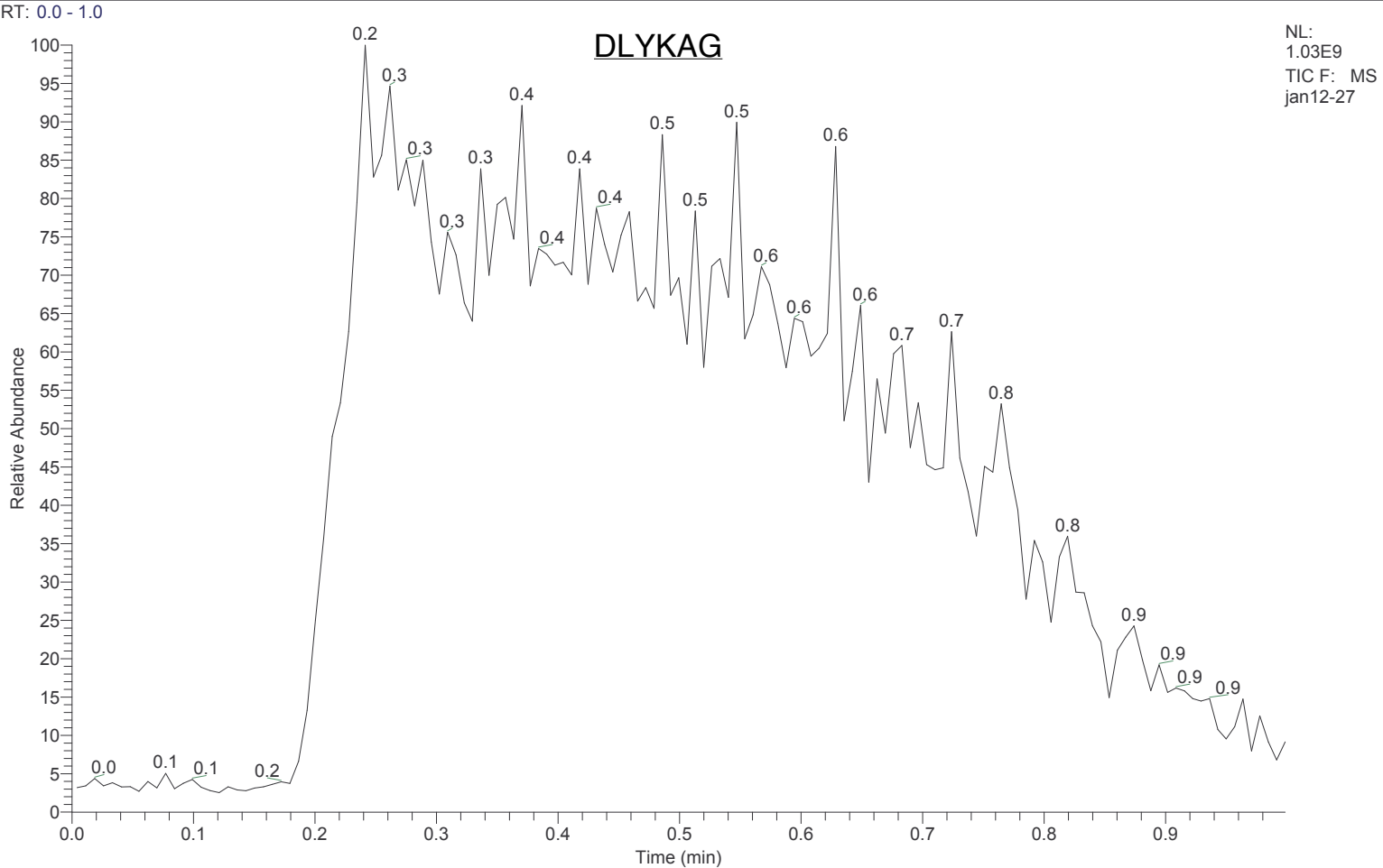
D:\My docs\Morten_2\HPLC\2012\jan12\180112\180112-10.lcd
 Acquired by : user
 Sample Name : C30
 Sample ID : Asn - diazotized
 Tray# : 1
 Vail # : 29
 Injection Volume : 20 uL
 Data File Name : 180112-10.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 180112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 18-Jan-12 22:06:36
 Data Processed : 25-Jan-12 13:14:08



DLYKAG**==== Shimadzu LCsolution Analysis Report ====**

D:\My docs\Morten_2\HPLC\2012\jan12\100112\100112-64.lcd
 Acquired by : user
 Sample Name : Asp
 Sample ID : bef. diazotization
 Tray# : 1
 Vial # : 82
 Injection Volume : 10 uL
 Data File Name : 100112-64.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 100112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 13-Jan-12 3:22:37
 Data Processed : 13-Jan-12 4:12:41

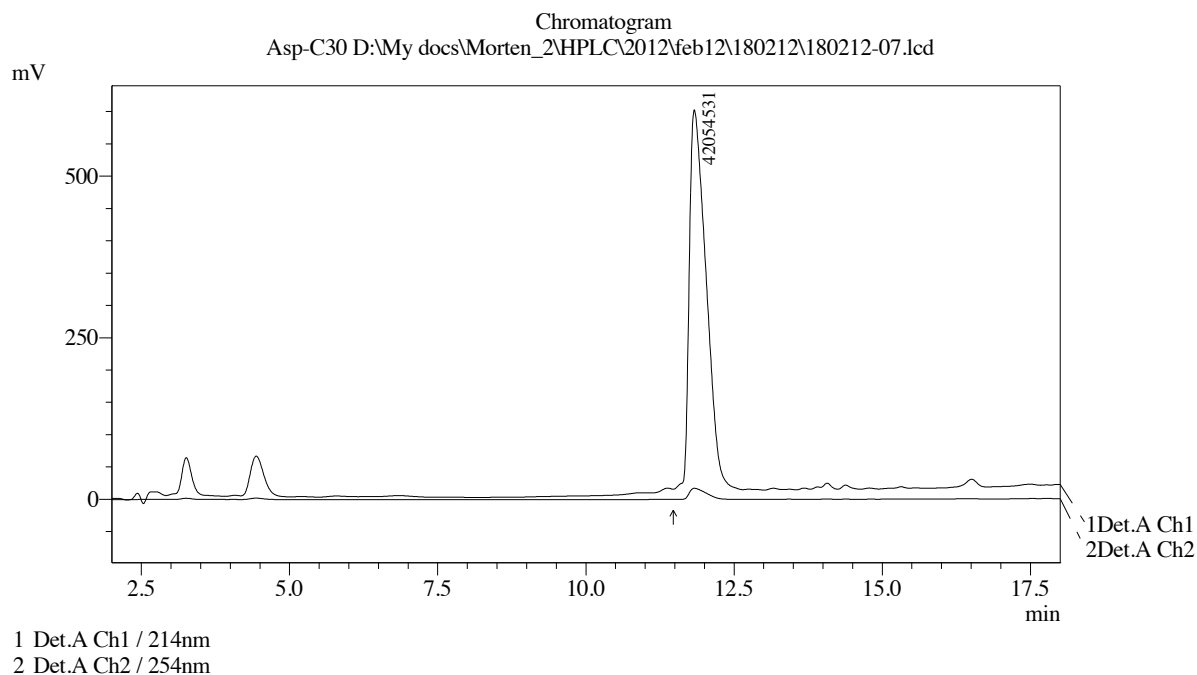
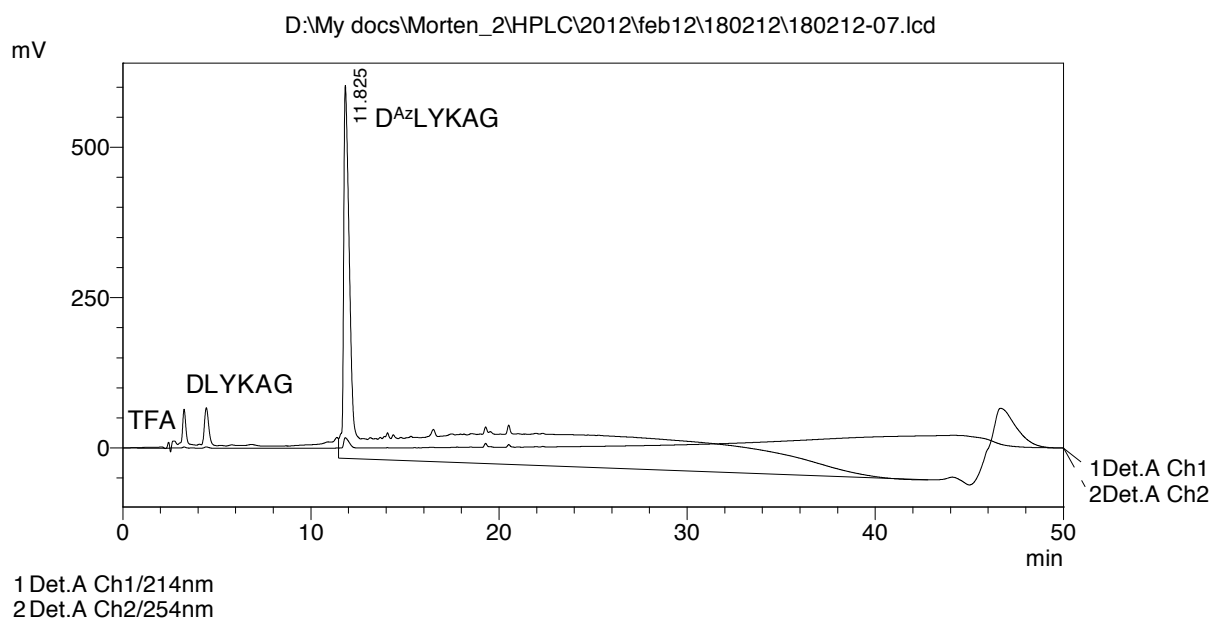




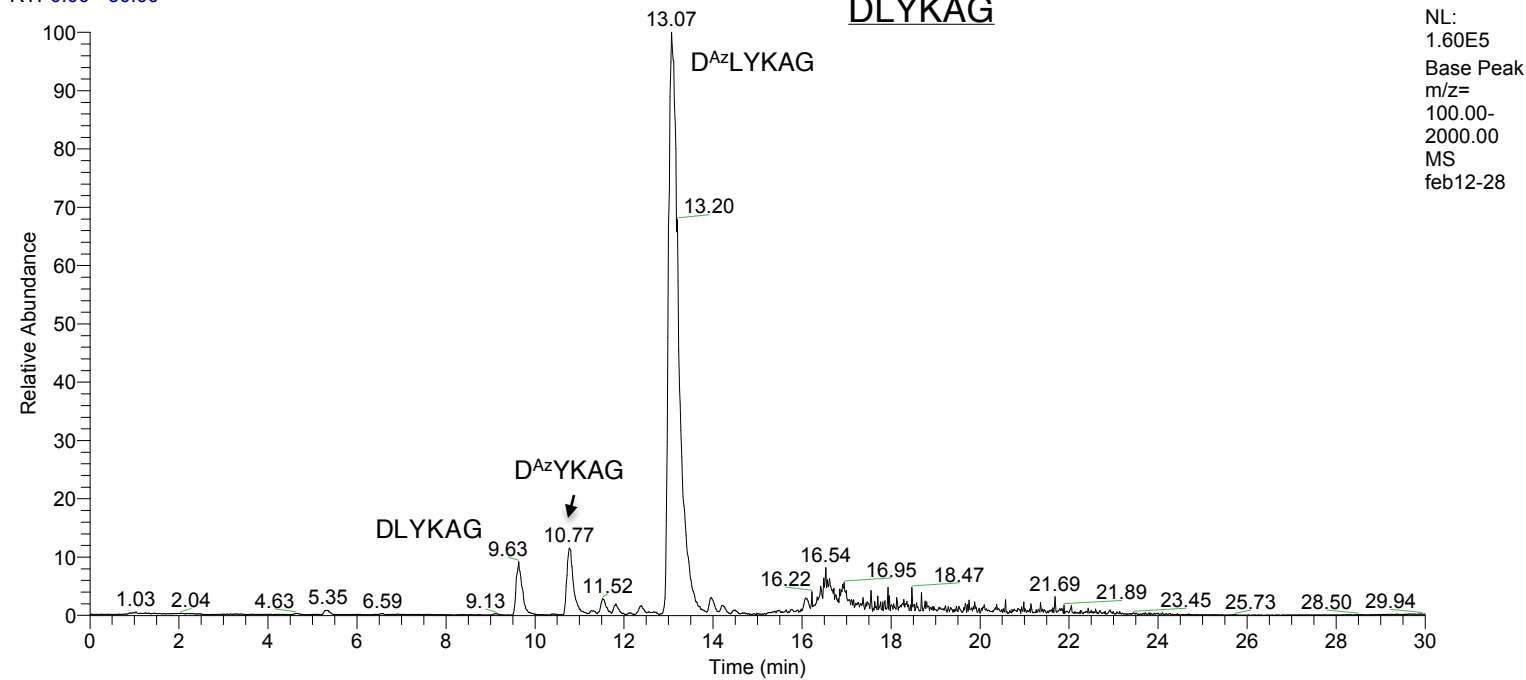
DLYKAG

==== Shimadzu LCsolution Analysis Report ====

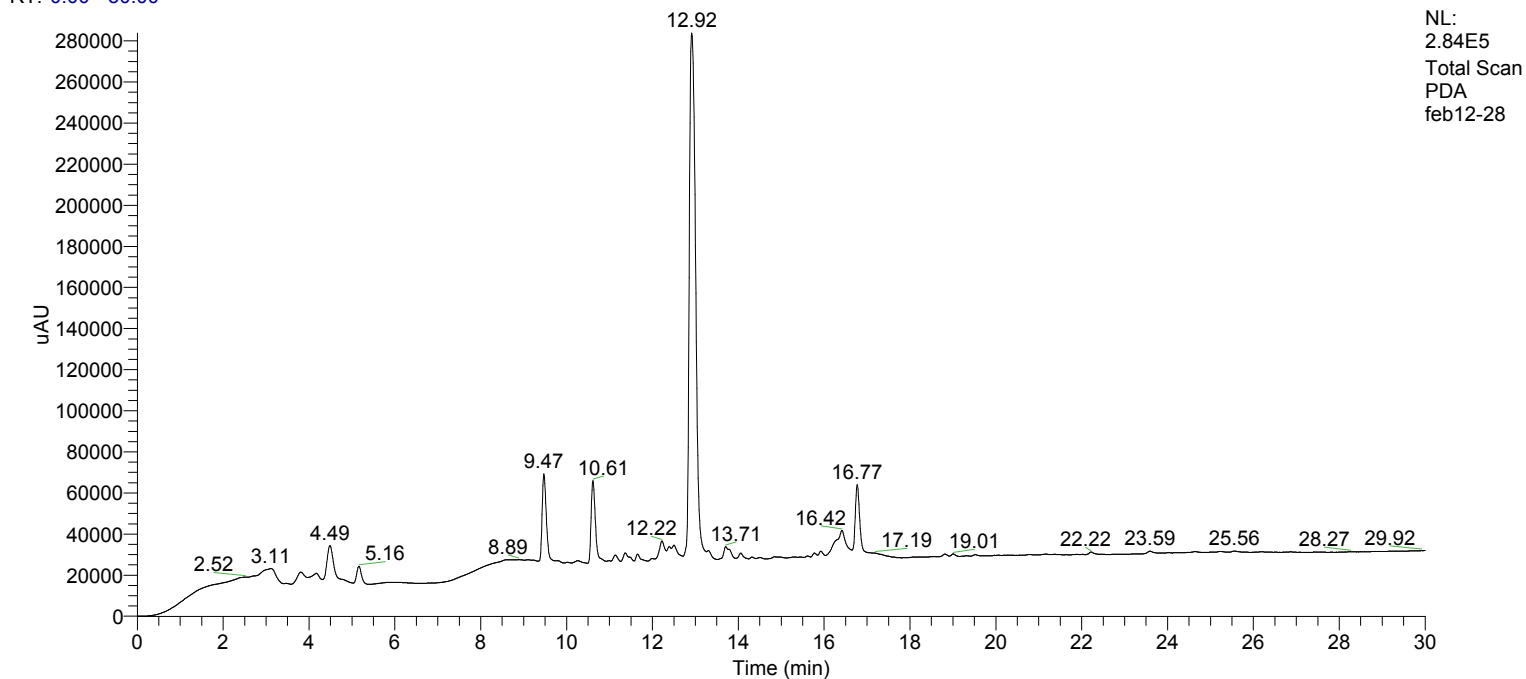
D:\My docs\Morten_2\HPLC\2012\feb12\180212\180212-07.lcd
 Acquired by : guest user
 Sample Name : Asp-C30
 Sample ID :
 Tray# : 1
 Vial # : 74
 Injection Volume : 3 uL
 Data File Name : 180212-07.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 20120218.lcb
 Report File Name : Default.lcr
 Data Acquired : 19-Feb-12 3:08:40
 Data Processed : 19-Feb-12 3:58:46



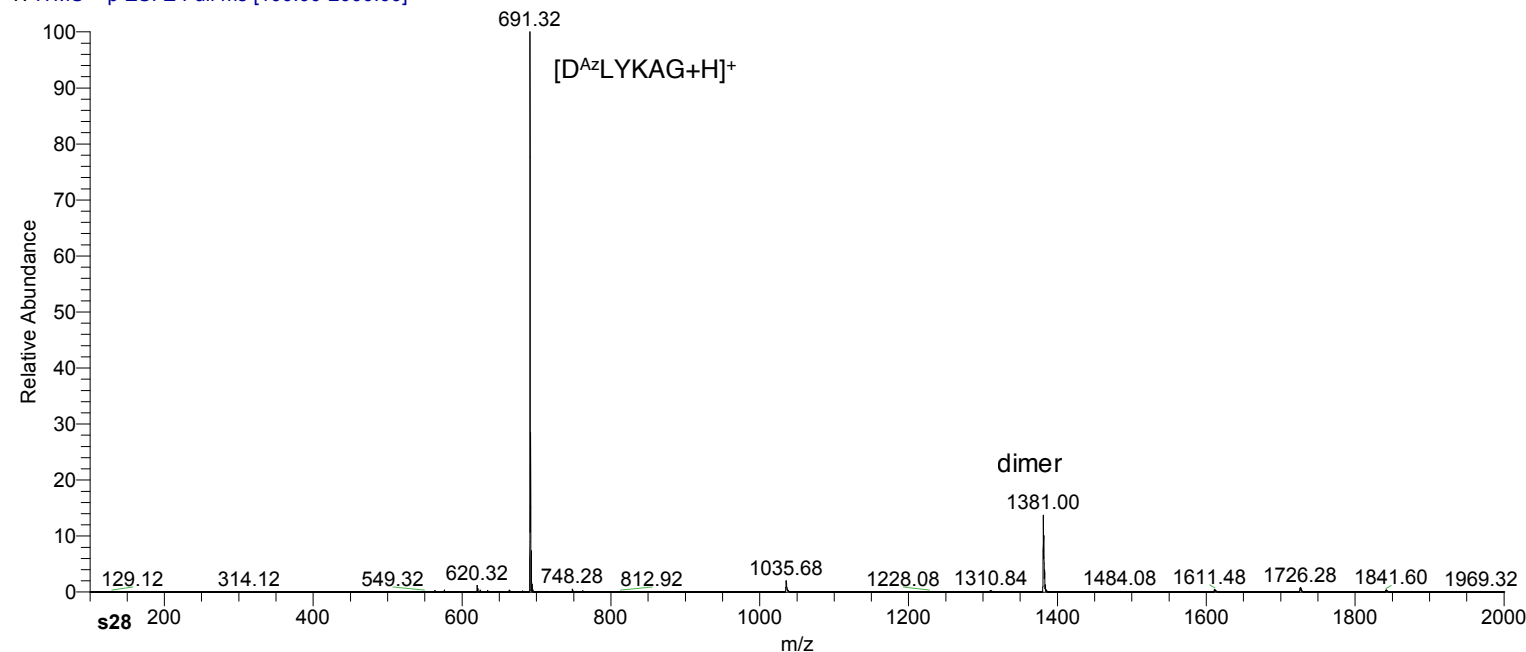
RT: 0.00 - 30.00



RT: 0.00 - 30.00

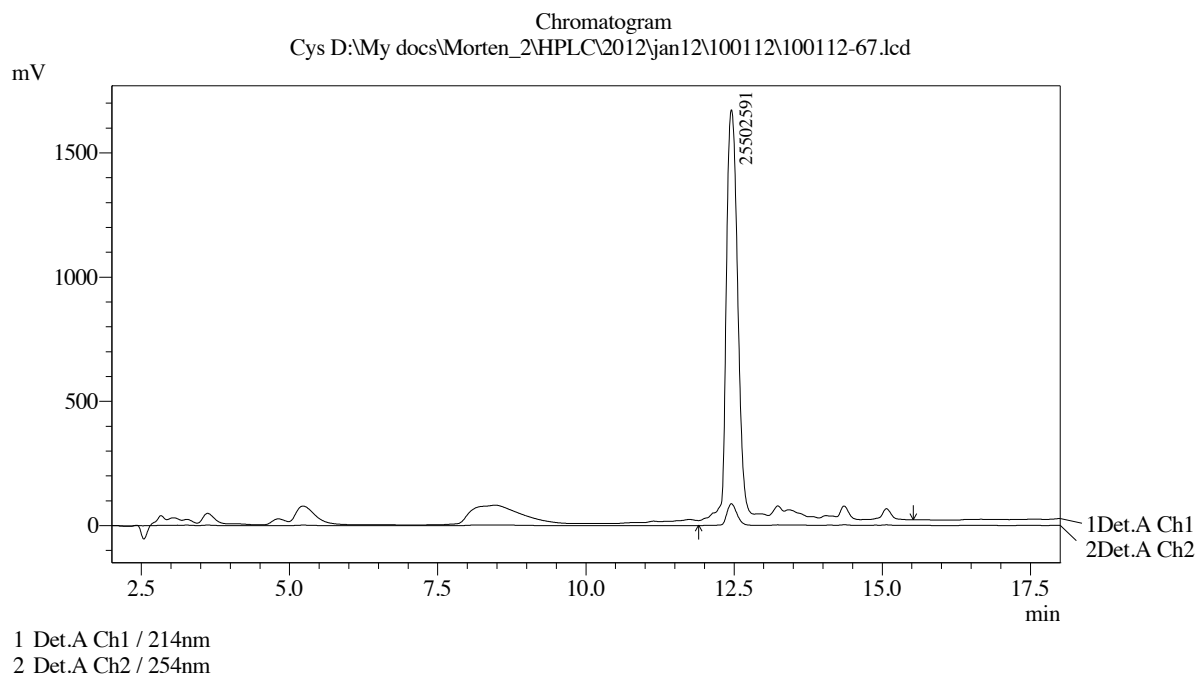
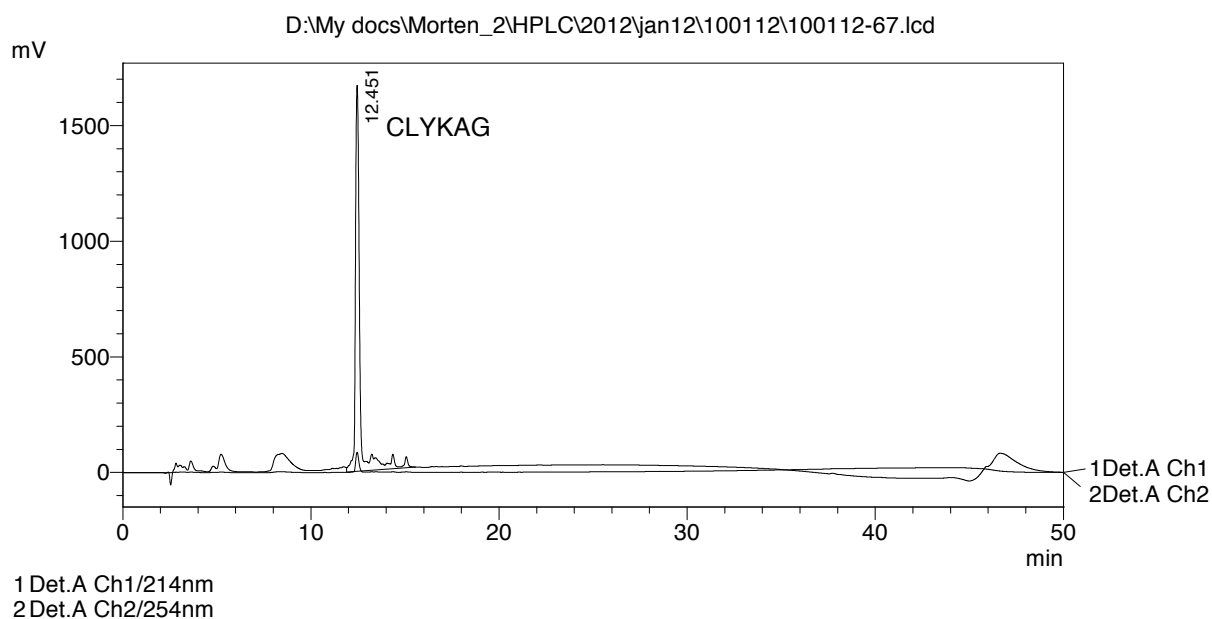


feb12-28 #718-746 RT: 12.94-13.39 AV: 29 NL: 8.42E4
T: ITMS + p ESI E Full ms [100.00-2000.00]

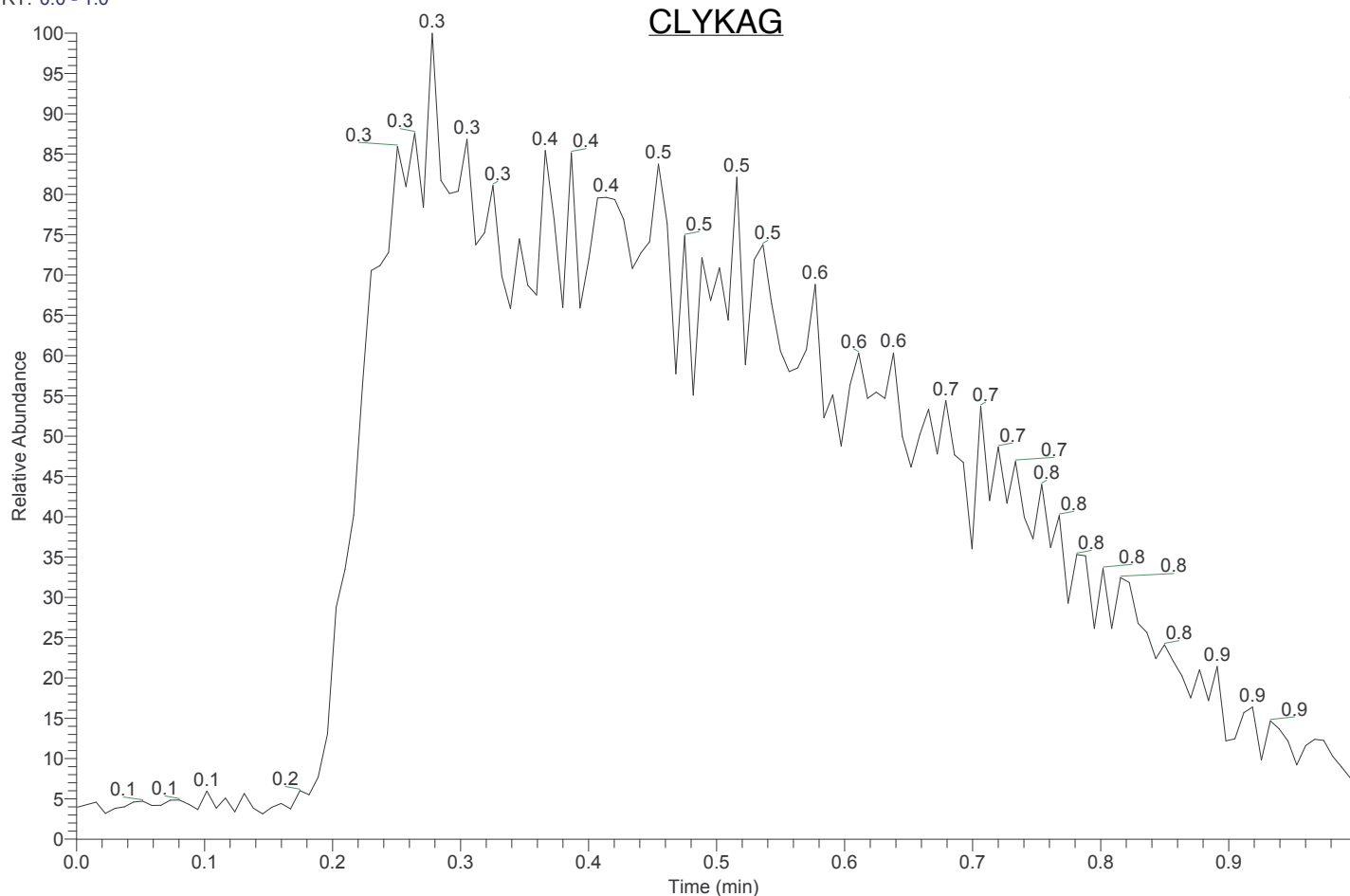
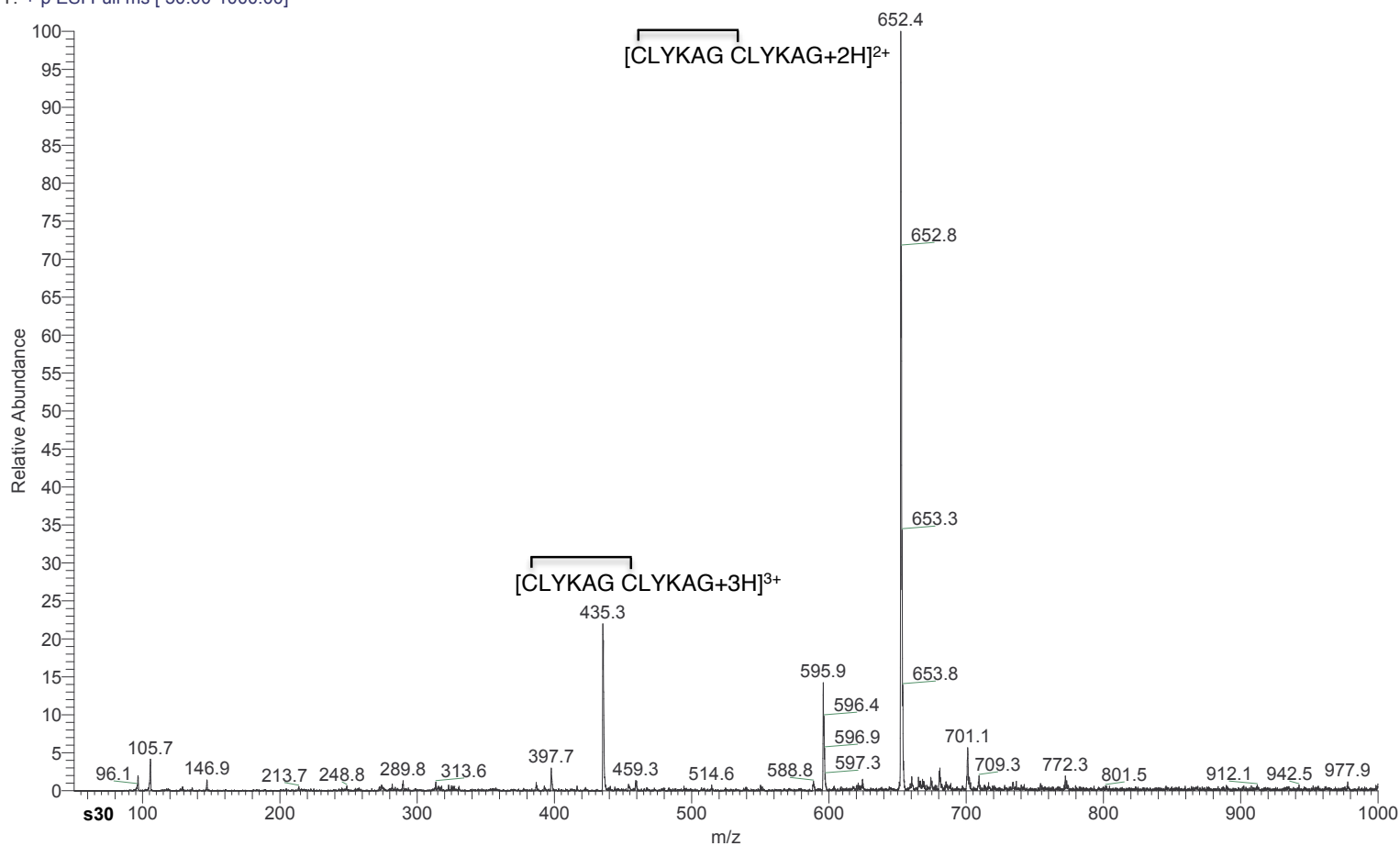


==== Shimadzu LCsolution Analysis Report ====

Acquired by : user
Sample Name : Cys
Sample ID : bef. diazotization
Tray# : 1
Vial # : 85
Injection Volume : 10 uL
Data File Name : 100112-67.lcd
Method File Name : gradient-anal.lcm
Batch File Name : 100112.lcb
Report File Name : ALYKAG-report.lcr
Data Acquired : 13-Jan-12 5:53:57
Data Processed : 13-Jan-12 6:44:01



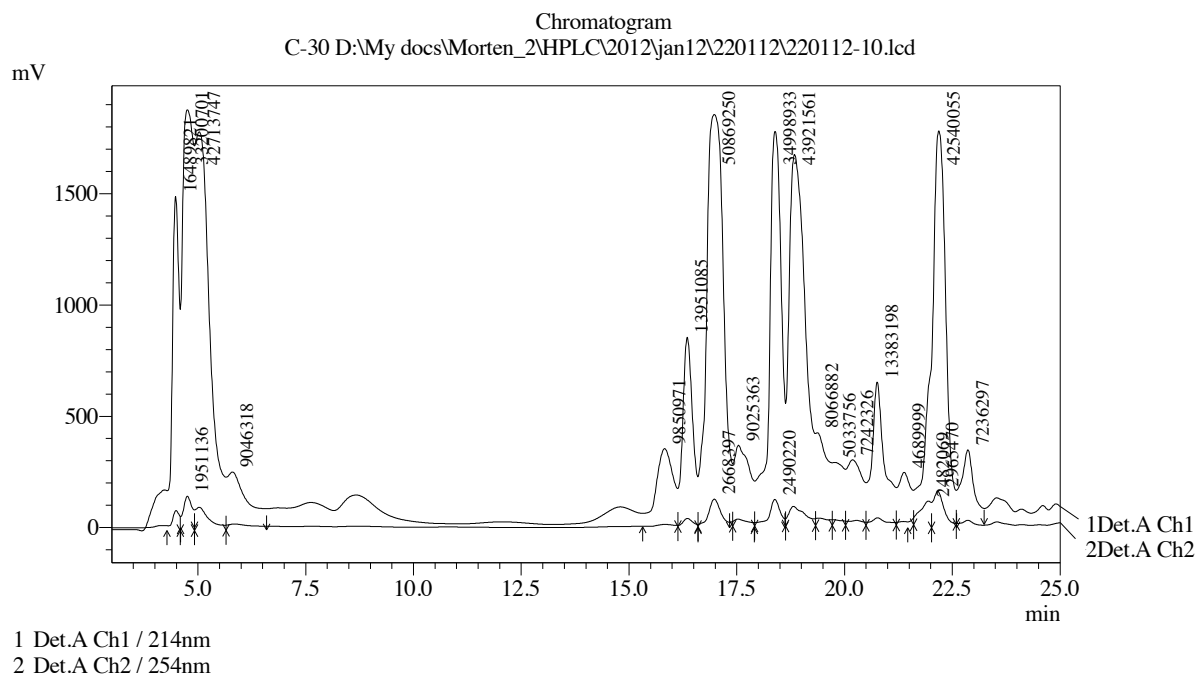
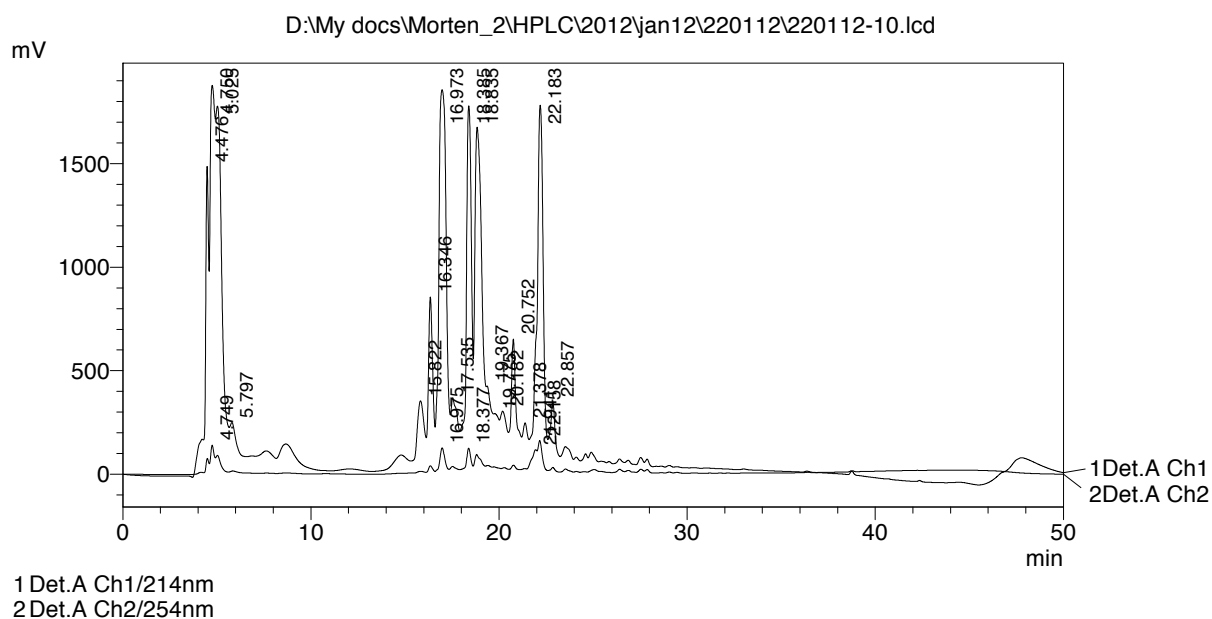
RT: 0.0 - 1.0

jan12-21 #28-76 RT: 0.2-0.5 AV: 49 NL: 1.64E7
T: + p ESI Full ms [50.00-1000.00]

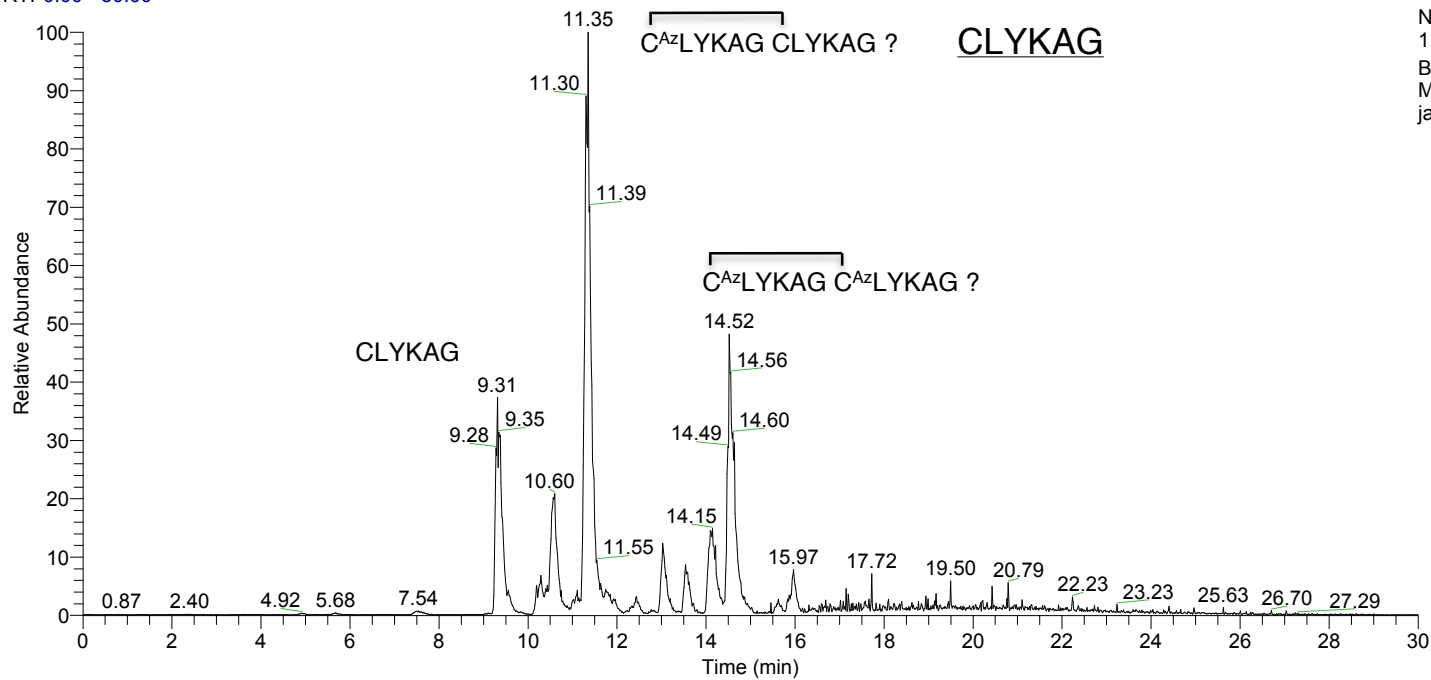
CLYKAG

==== Shimadzu LCsolution Analysis Report ====

D:\My docs\Morten_2\HPLC\2012\jan12\220112\220112-10.lcd
 Acquired by : user
 Sample Name : C-30
 Sample ID : Cys - diazotized
 Tray# : 1
 Vail # : 69
 Injection Volume : 20 uL
 Data File Name : 220112-10.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 220112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 23-Jan-12 1:21:57
 Data Processed : 23-Jan-12 2:12:01

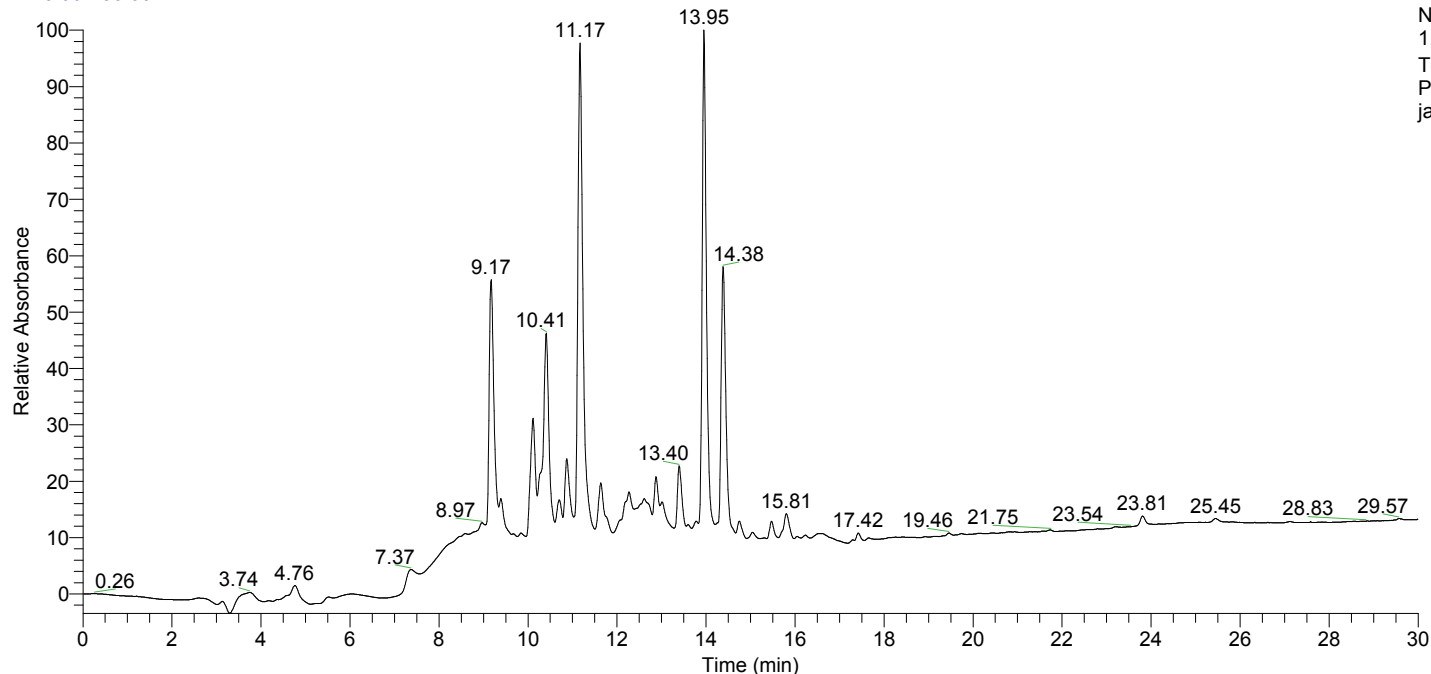


RT: 0.00 - 30.00



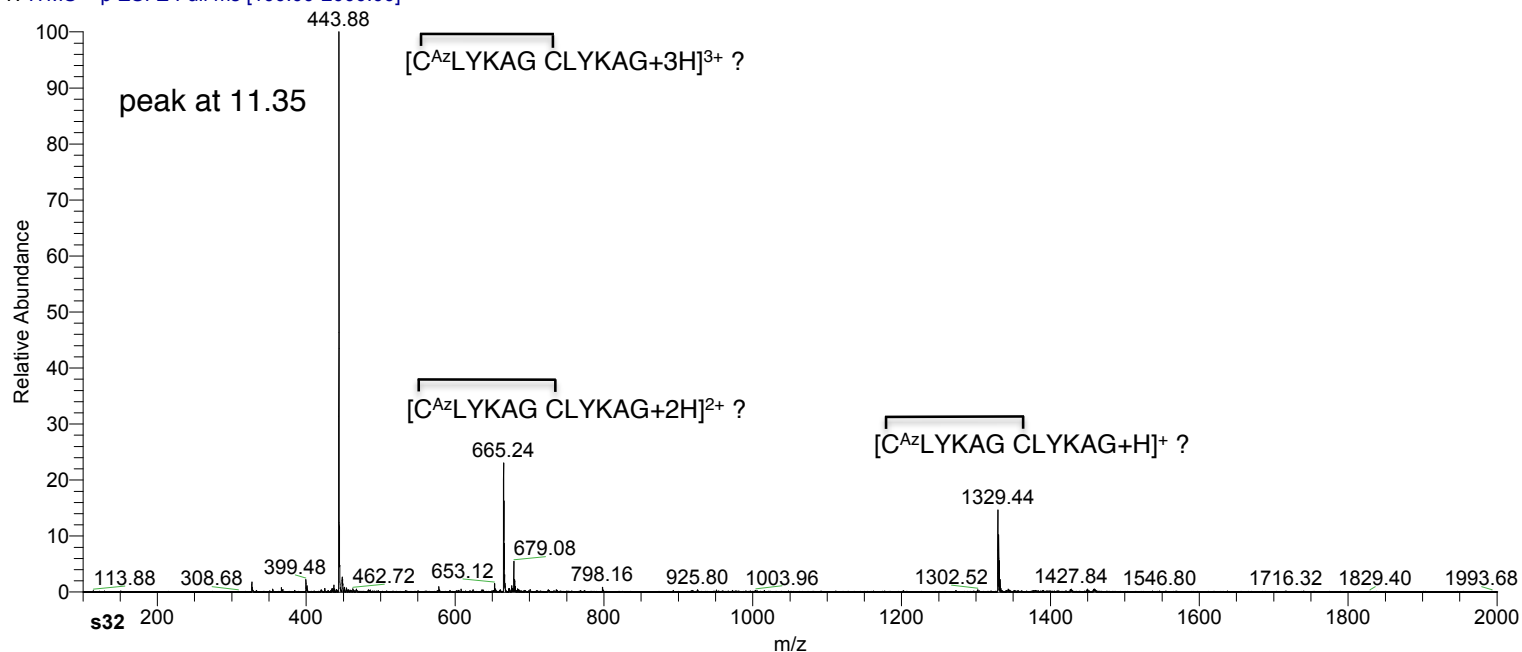
NL:
1.24E5
Base Peak
MS
jan12-21

RT: 0.00 - 30.00



NL:
1.01E5
Total Scan
PDA
jan12-21

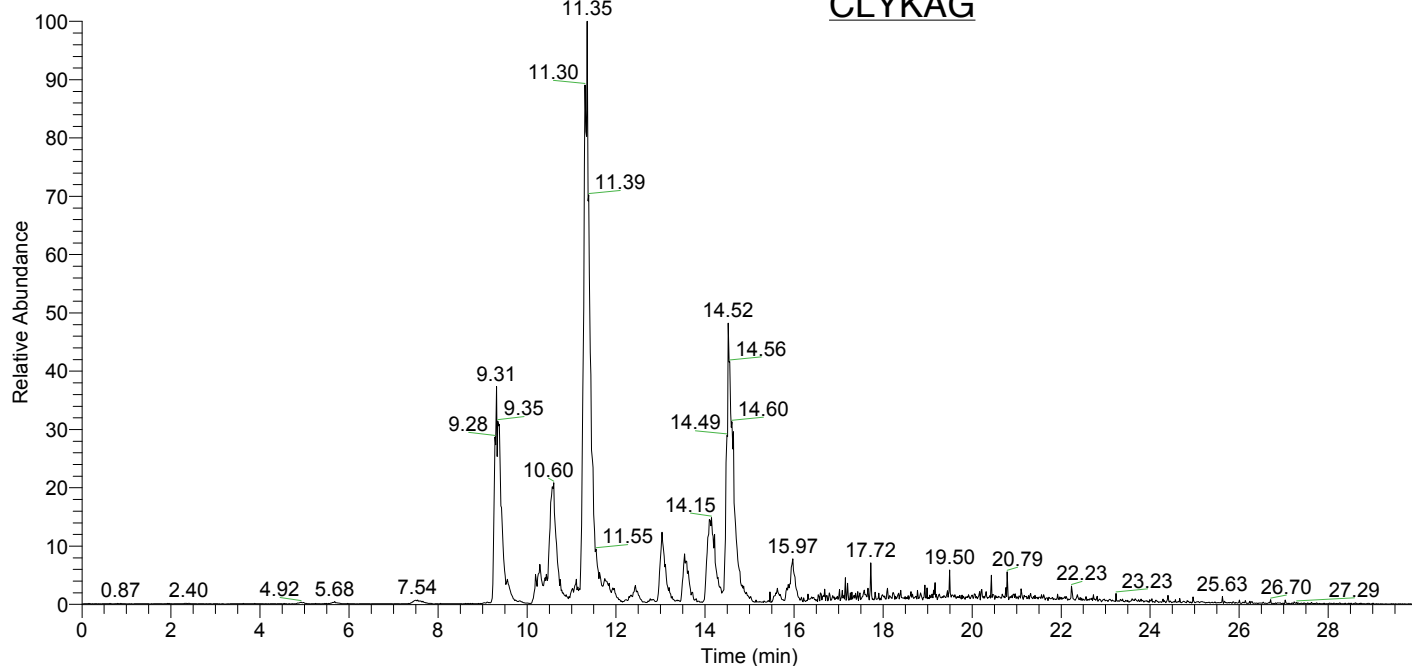
jan12-21 #655-678 RT: 11.19-11.57 AV: 24 NL: 4.84E4
T: ITMS + p ESI E Full ms [100.00-2000.00]



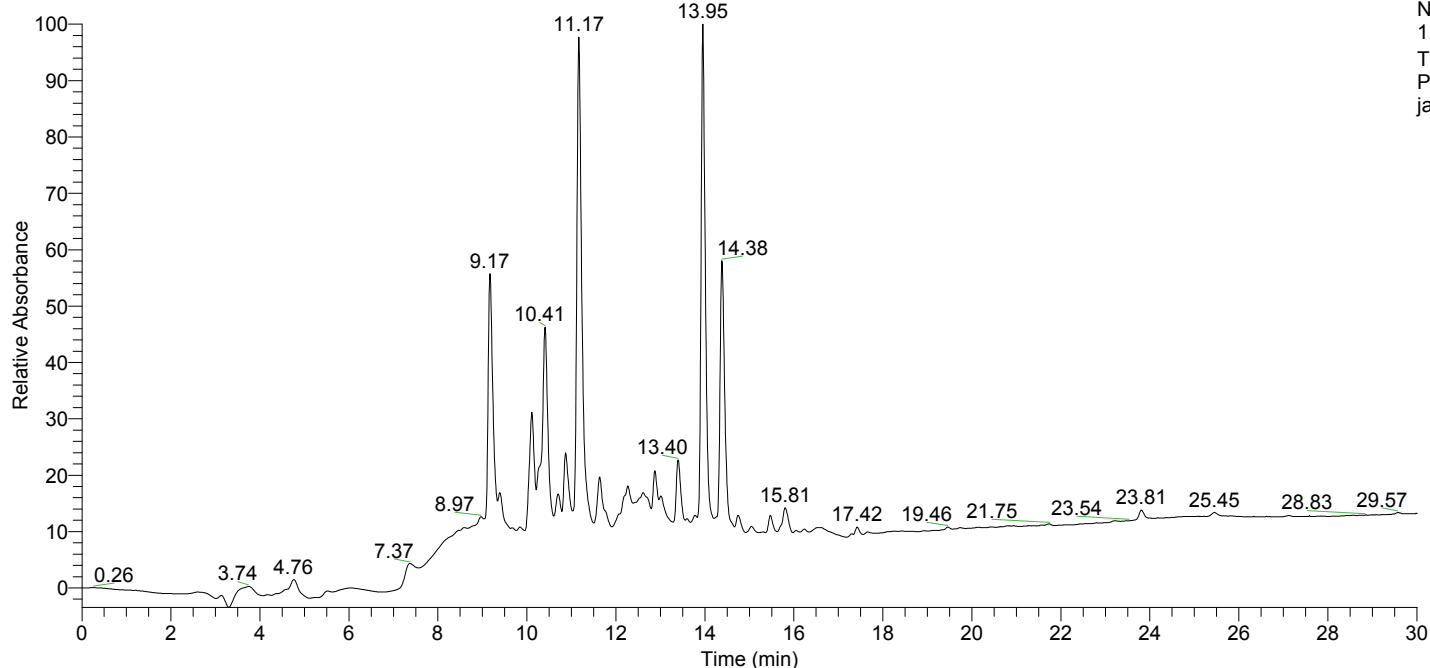
RT: 0.00 - 30.00

CLYKAG

NL:
1.24E5
Base Peak
MS
jan12-21

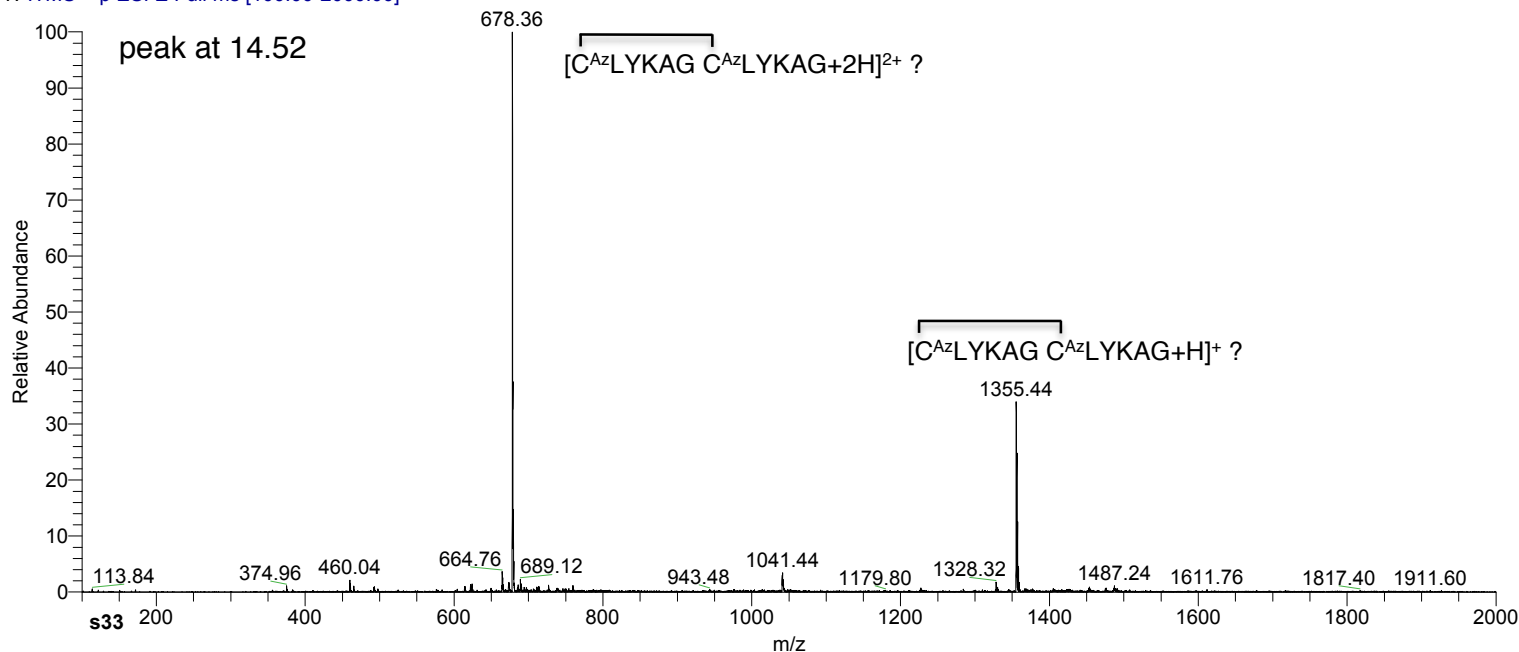


RT: 0.00 - 30.01



NL:
1.01E5
Total Scan
PDA
jan12-21

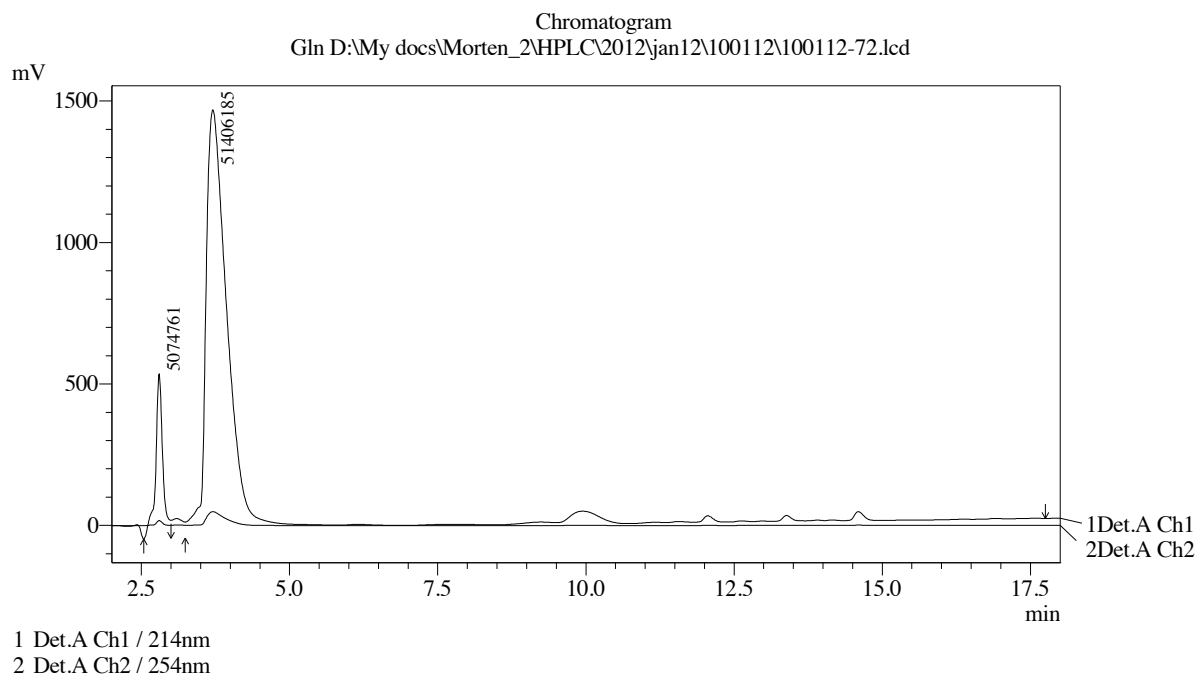
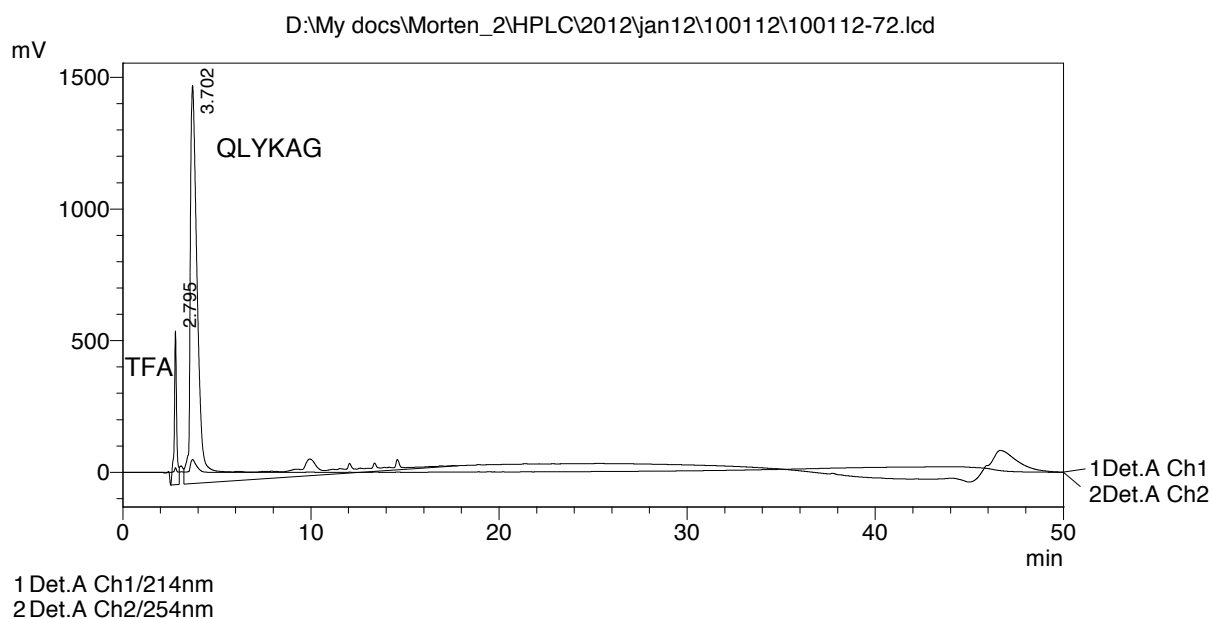
jan12-21 #854-876 RT: 14.46-14.82 AV: 23 NL: 2.42E4
T: ITMS + p ESI E Full ms [100.00-2000.00]

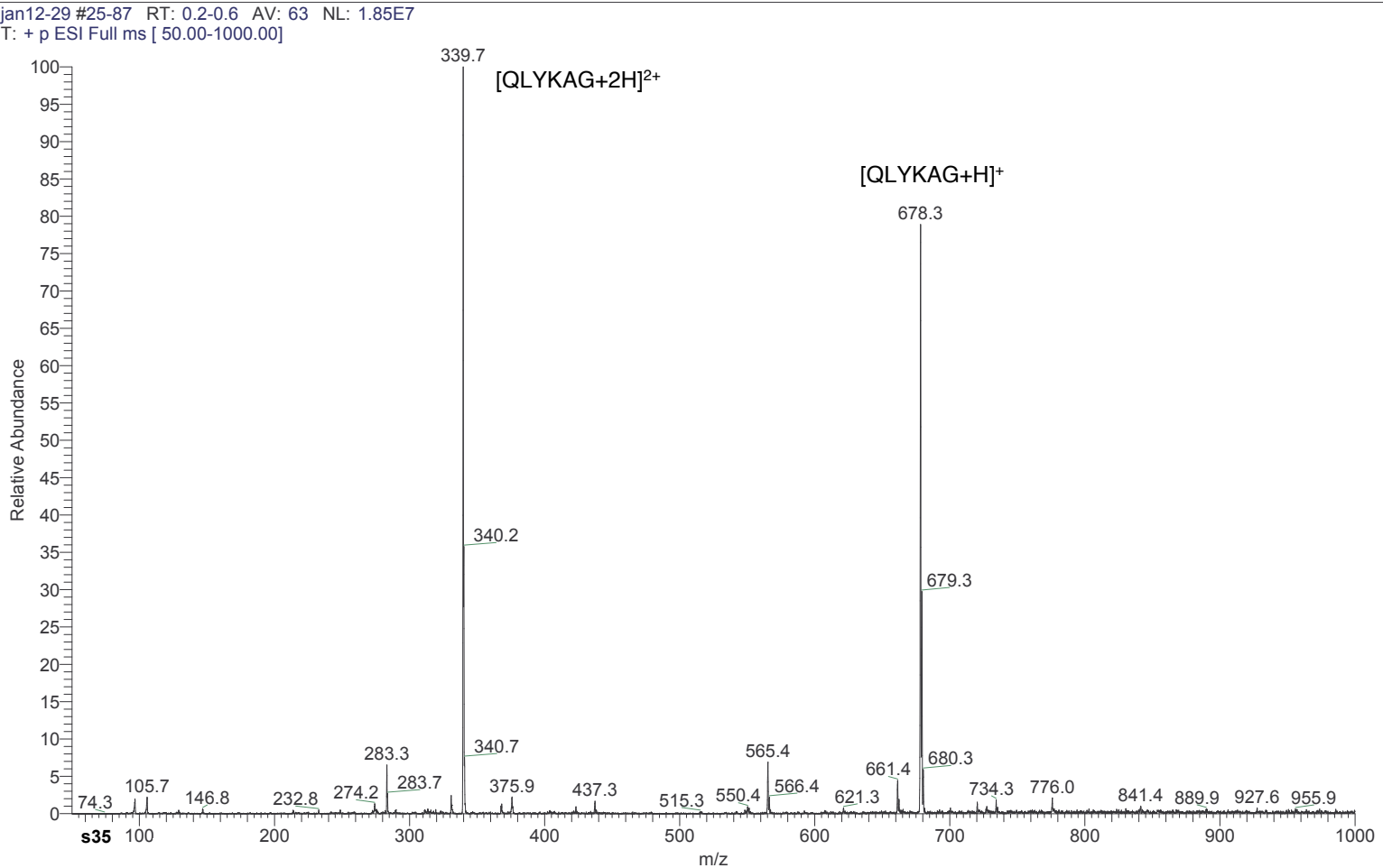
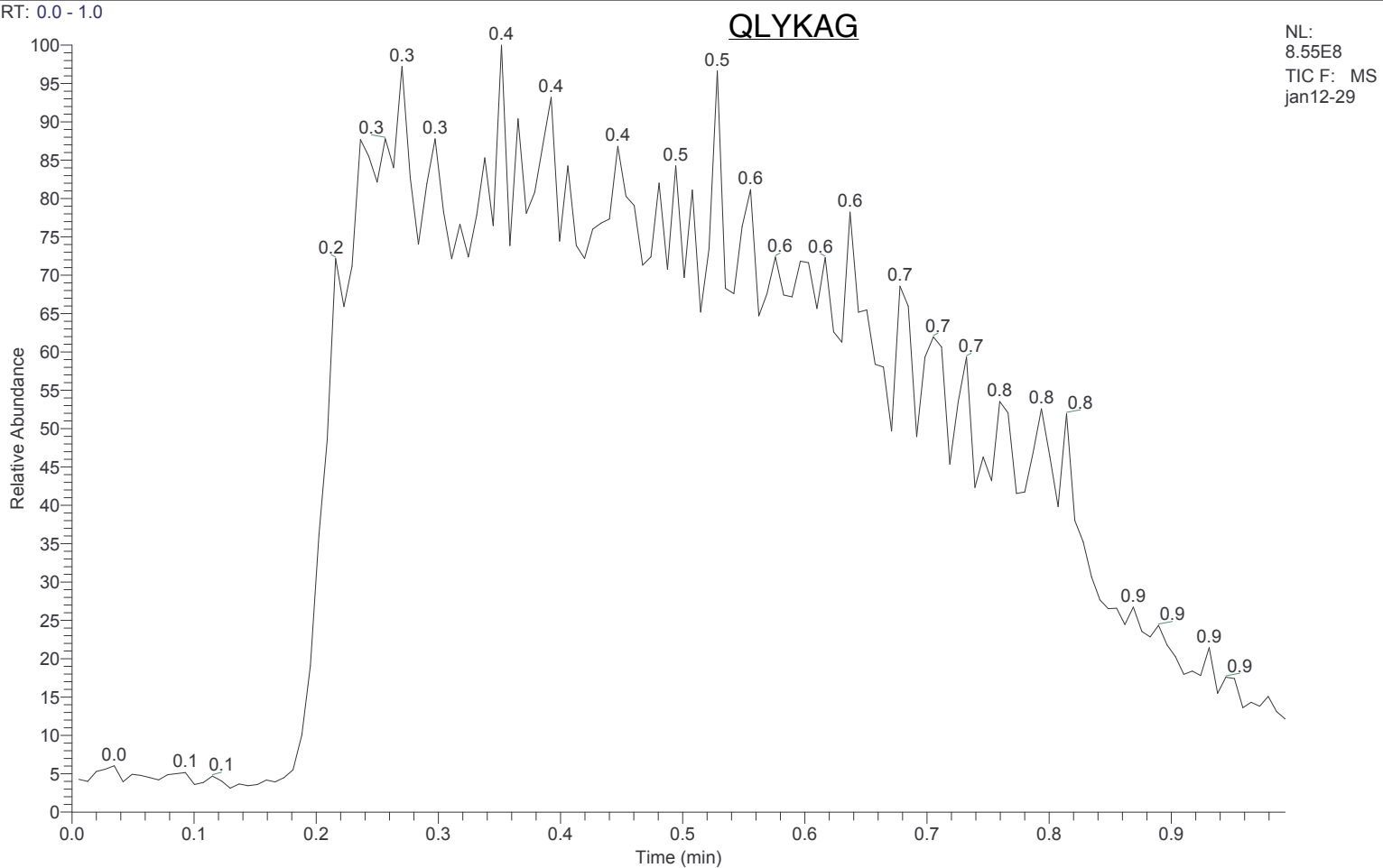


QLYKAG

==== Shimadzu LCsolution Analysis Report ====

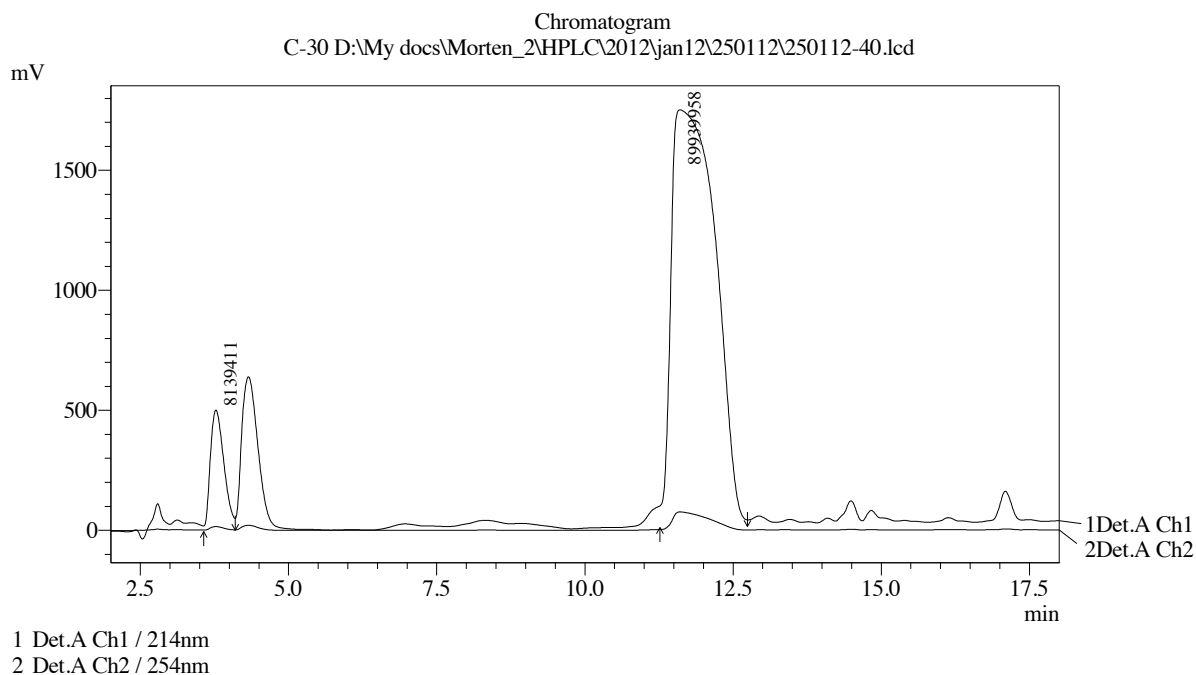
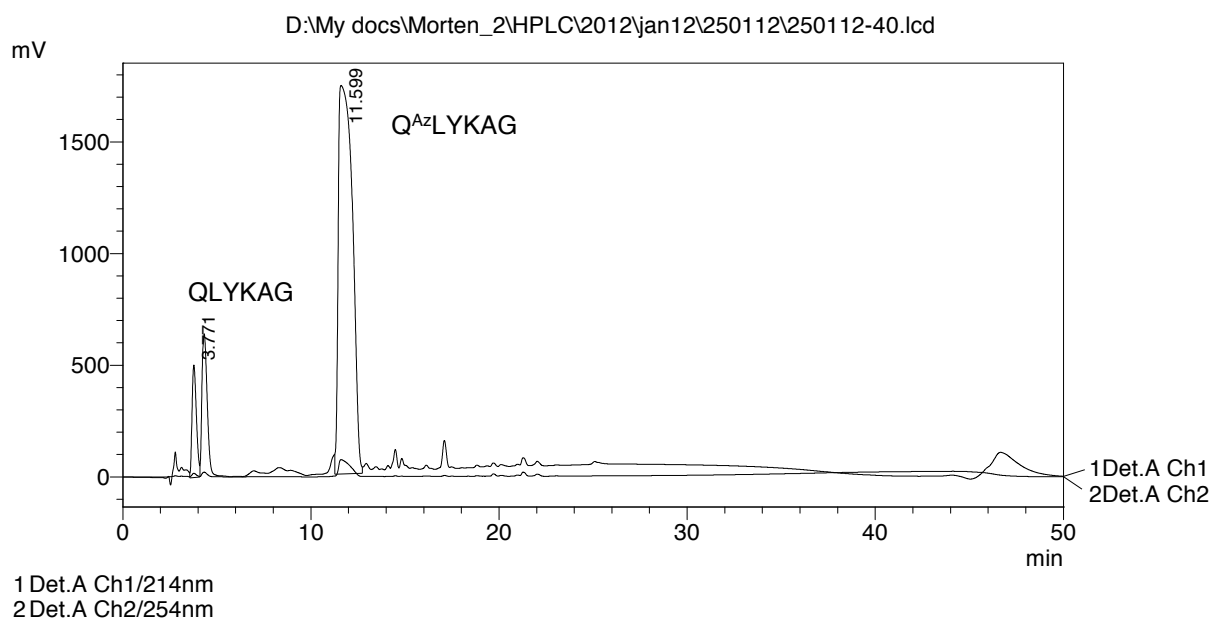
D:\My docs\Morten_2\HPLC\2012\jan12\100112\100112-72.lcd
 Acquired by : user
 Sample Name : Gln
 Sample ID : bef. diazotization
 Tray# : 1
 Vial # : 90
 Injection Volume : 10 uL
 Data File Name : 100112-72.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 100112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 13-Jan-12 10:06:12
 Data Processed : 13-Jan-12 10:56:18



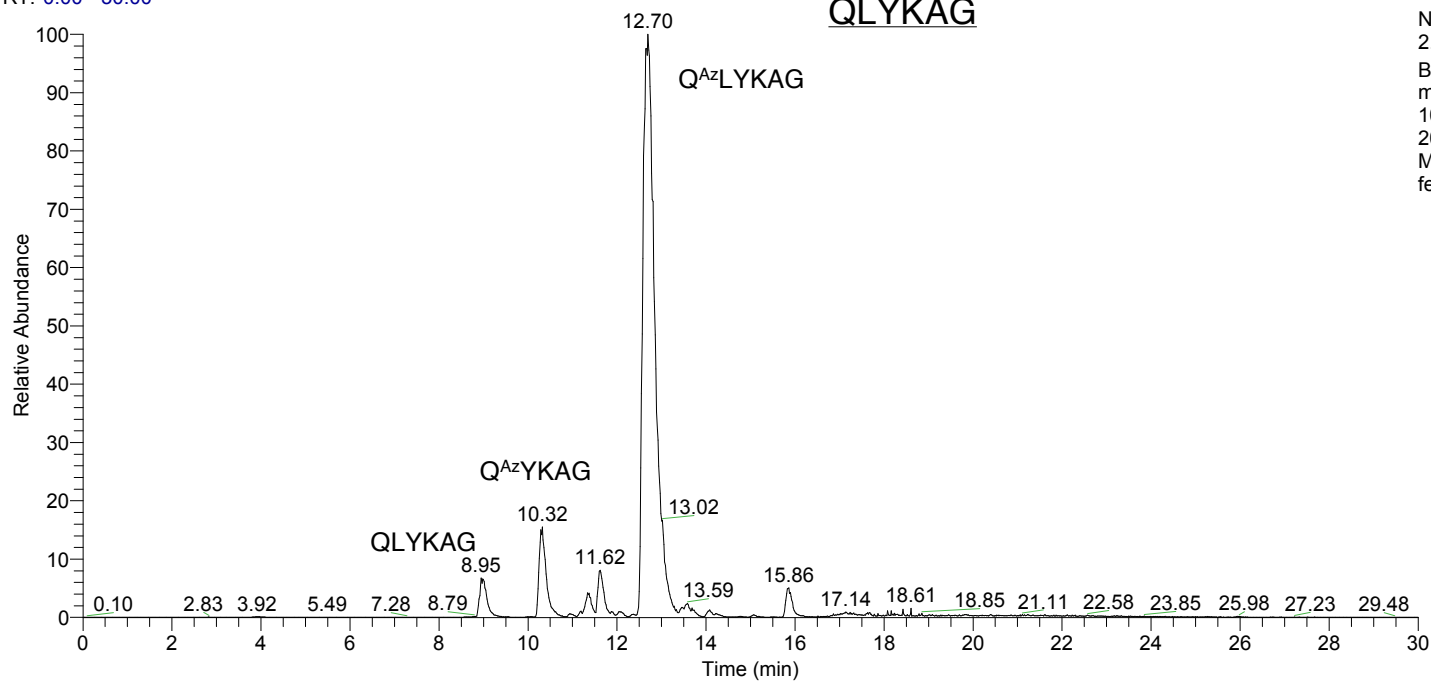


QLYKAG**==== Shimadzu LCsolution Analysis Report ====**

D:\My docs\Morten_2\HPLC\2012\jan12\250112\250112-40.lcd
 Acquired by : user
 Sample Name : C-30
 Sample ID : Gln - diazotized
 Tray# : 1
 Vial # : 33
 Injection Volume : 10 uL
 Data File Name : 250112-40.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 250112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 27-Jan-12 7:57:34
 Data Processed : 05-Feb-12 17:45:29

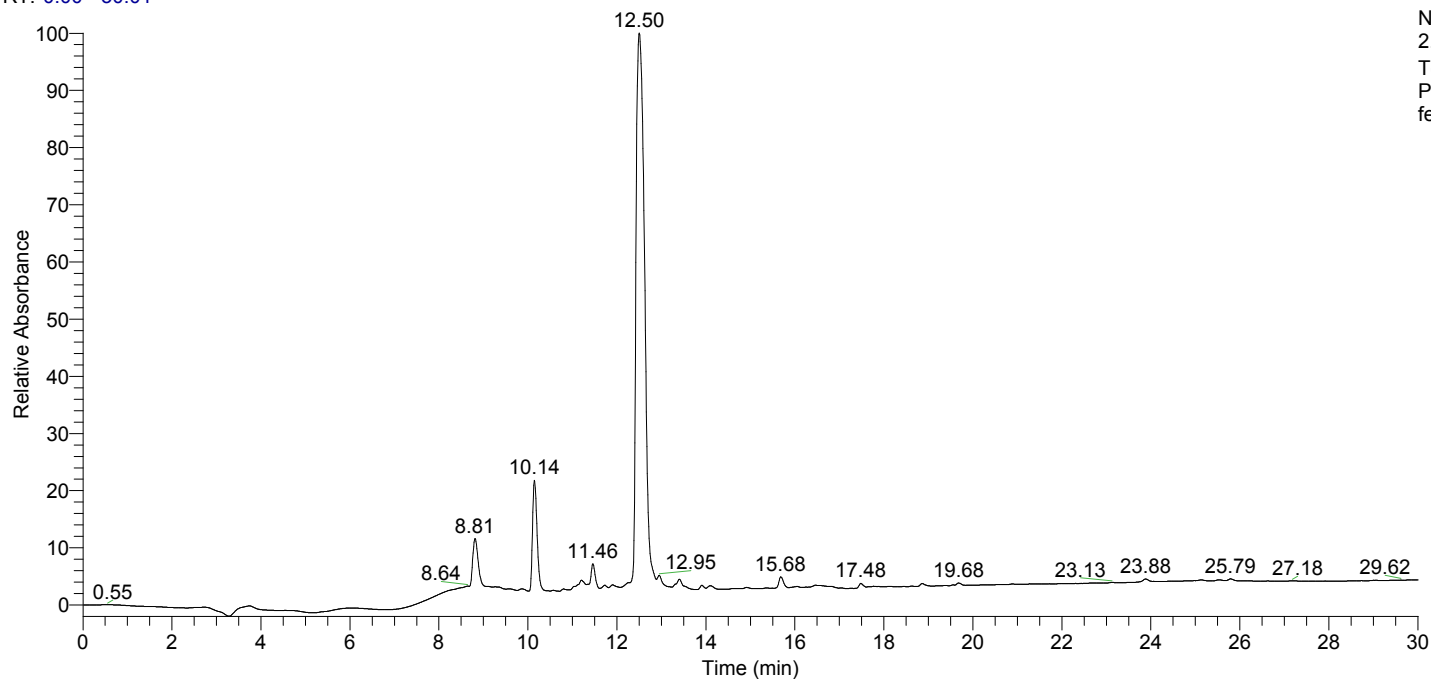


RT: 0.00 - 30.00



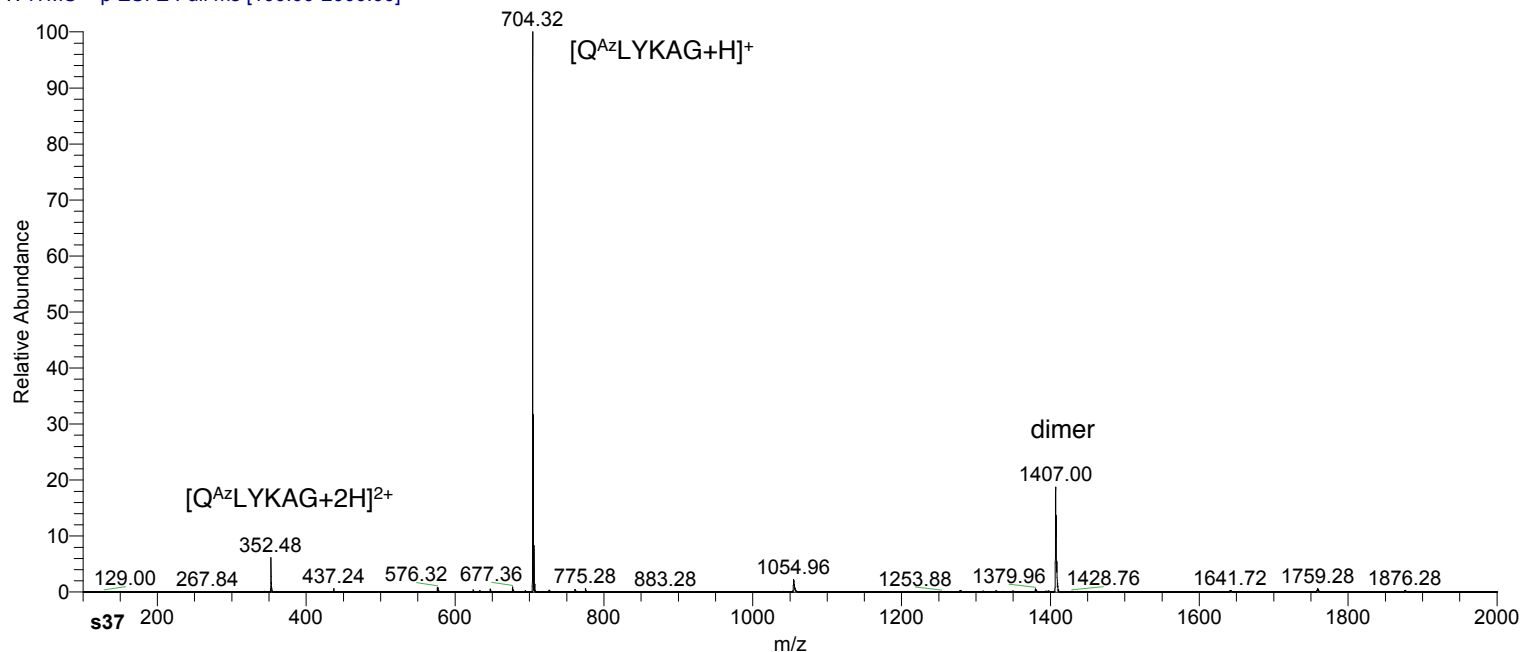
NL:
2.00E5
Base Peak
m/z=
100.00-
2000.00
MS
feb12-14

RT: 0.00 - 30.01



NL:
2.82E5
Total Scan
PDA
feb12-14

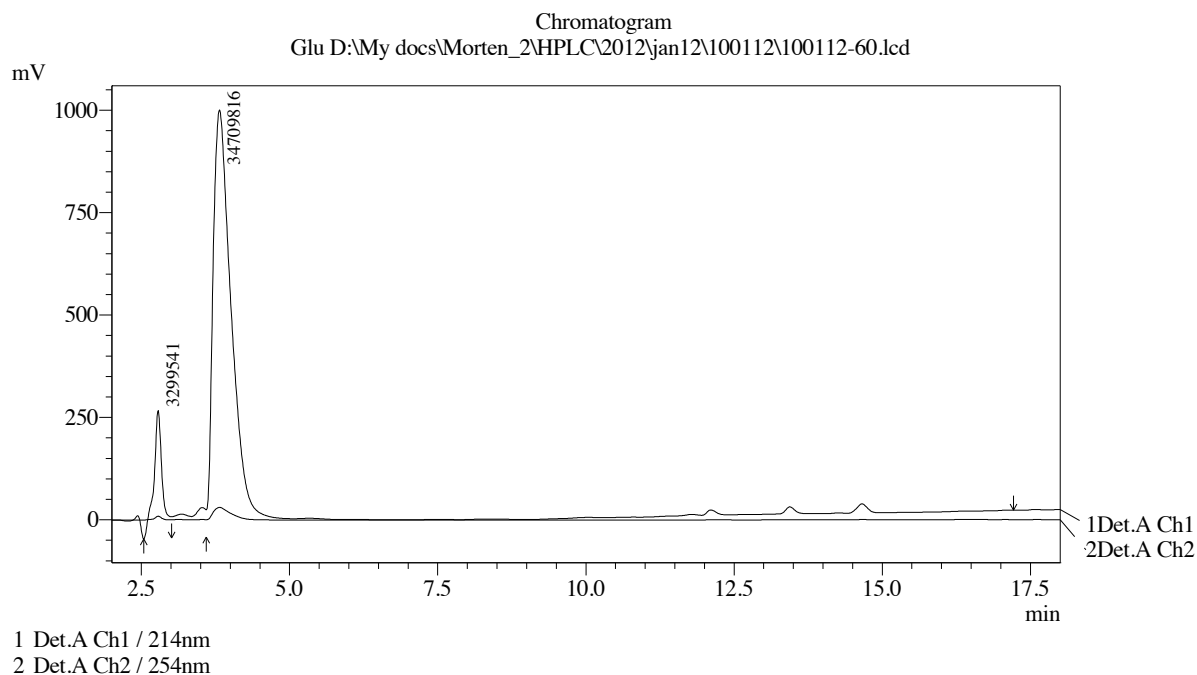
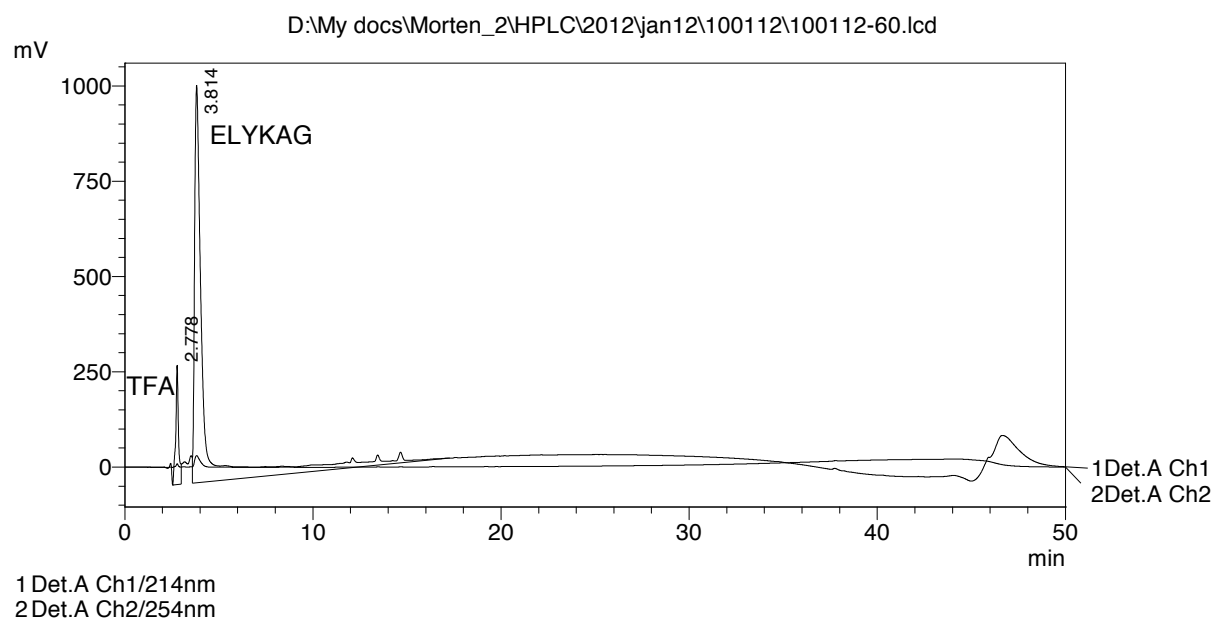
feb12-14 #675-713 RT: 12.52-13.14 AV: 39 NL: 9.63E4
T: ITMS + p ESI E Full ms [100.00-2000.00]



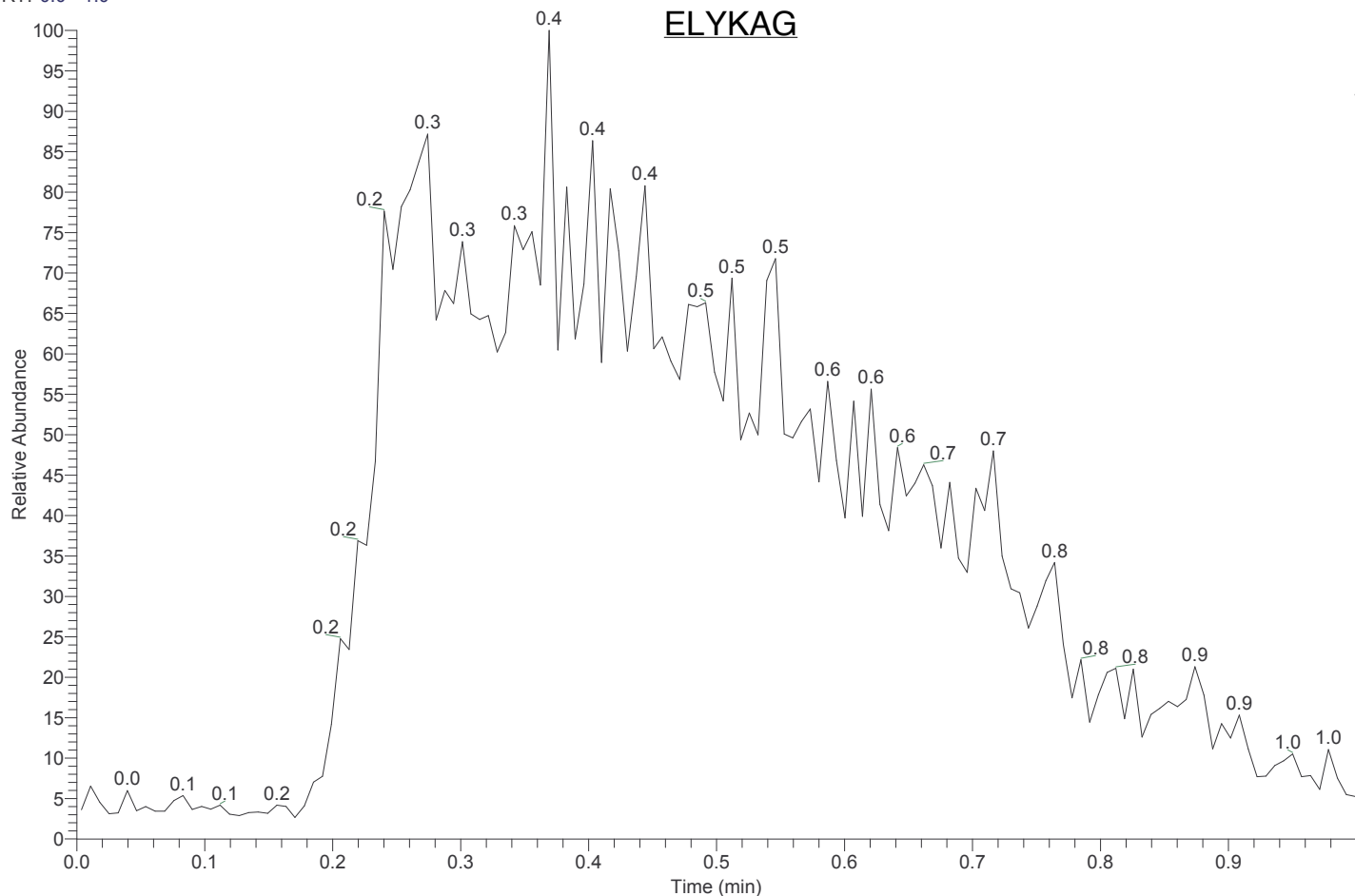
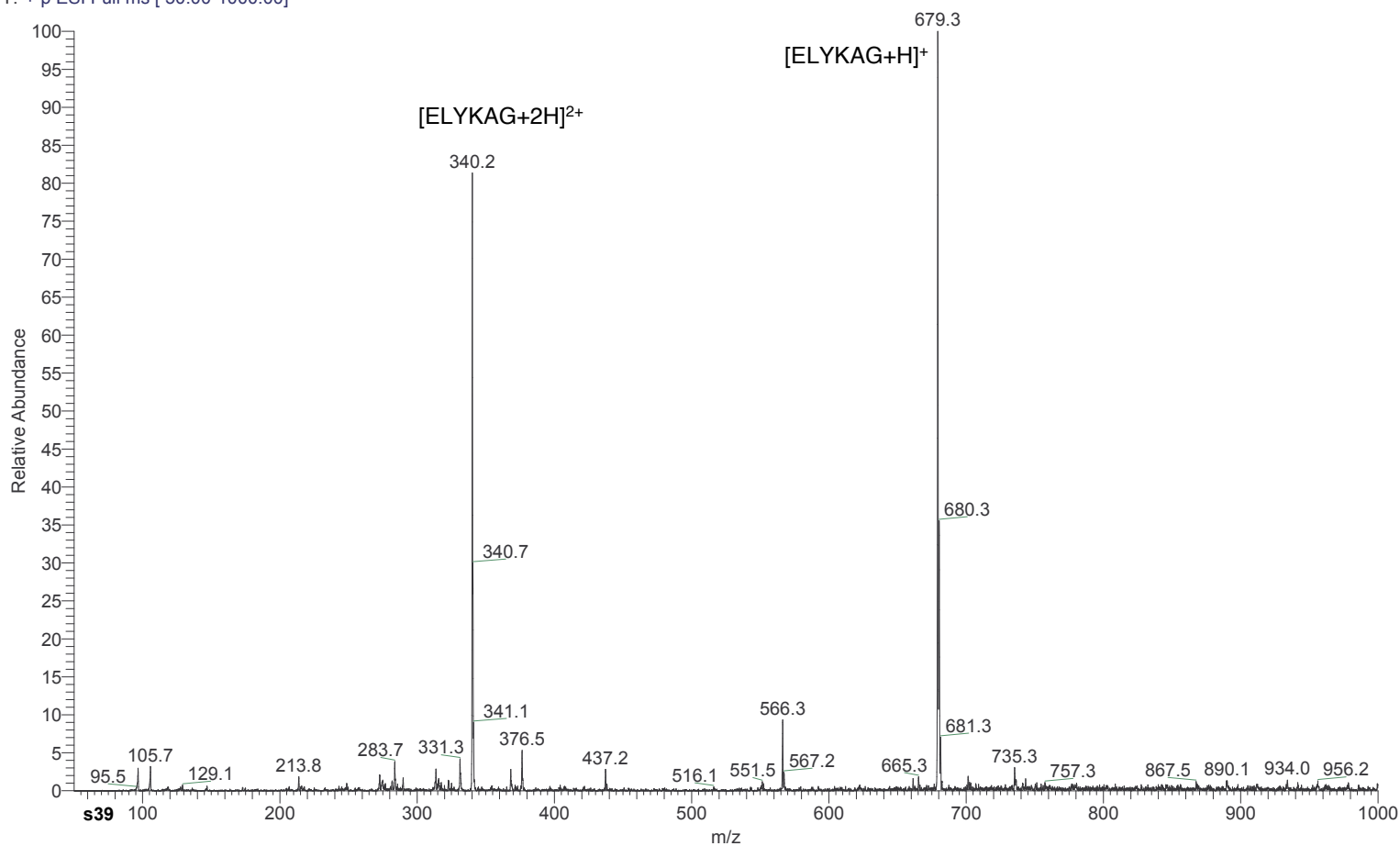
ELYKAG

==== Shimadzu LCsolution Analysis Report ====

D:\My docs\Morten_2\HPLC\2012\jan12\100112\100112-60.lcd
 Acquired by : user
 Sample Name : Glu
 Sample ID : bef. diazotization
 Tray# : 1
 Vail # : 78
 Injection Volume : 10 uL
 Data File Name : 100112-60.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 100112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 13-Jan-12 0:00:53
 Data Processed : 13-Jan-12 0:50:57

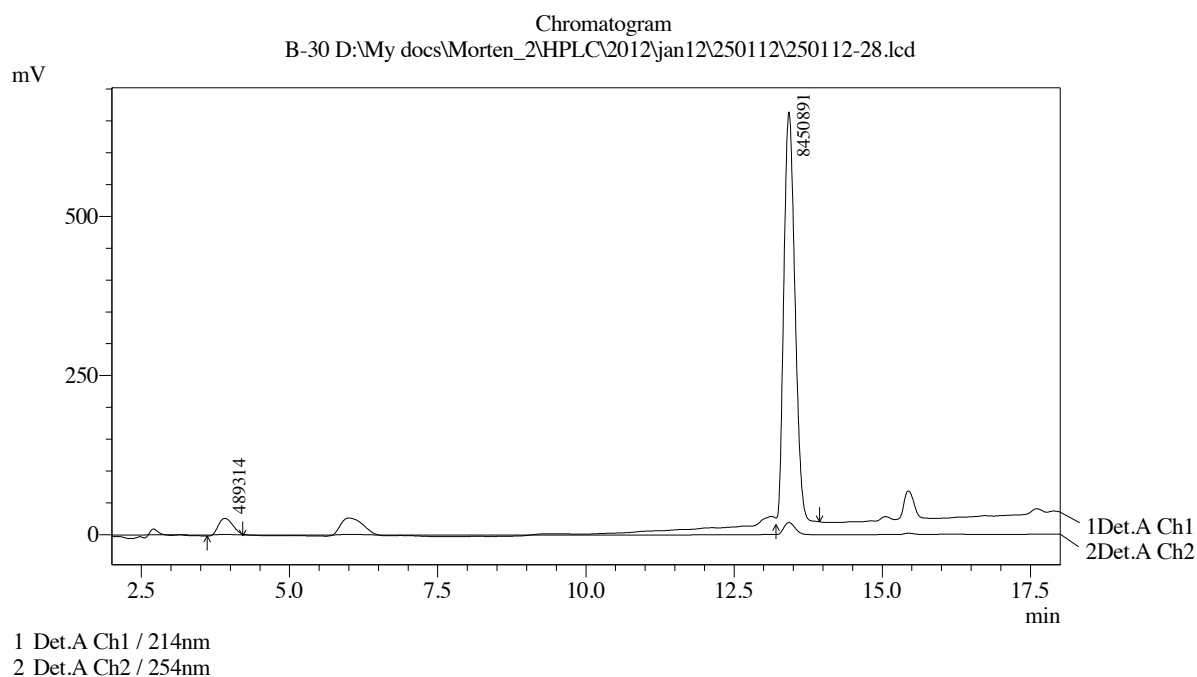
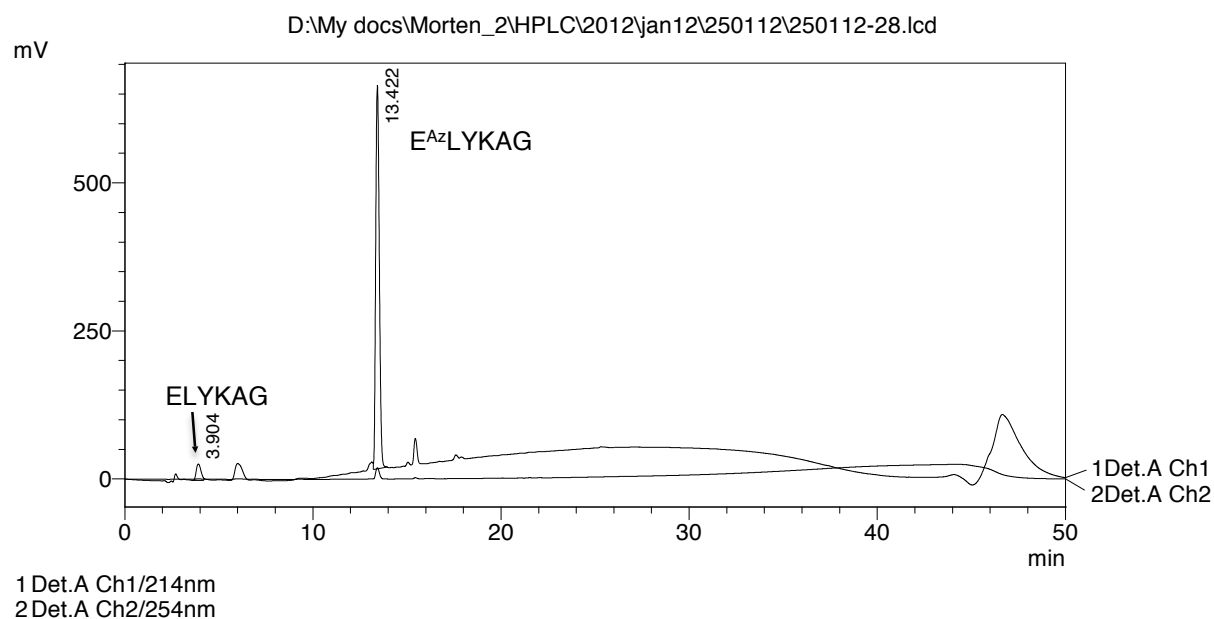


RT: 0.0 - 1.0

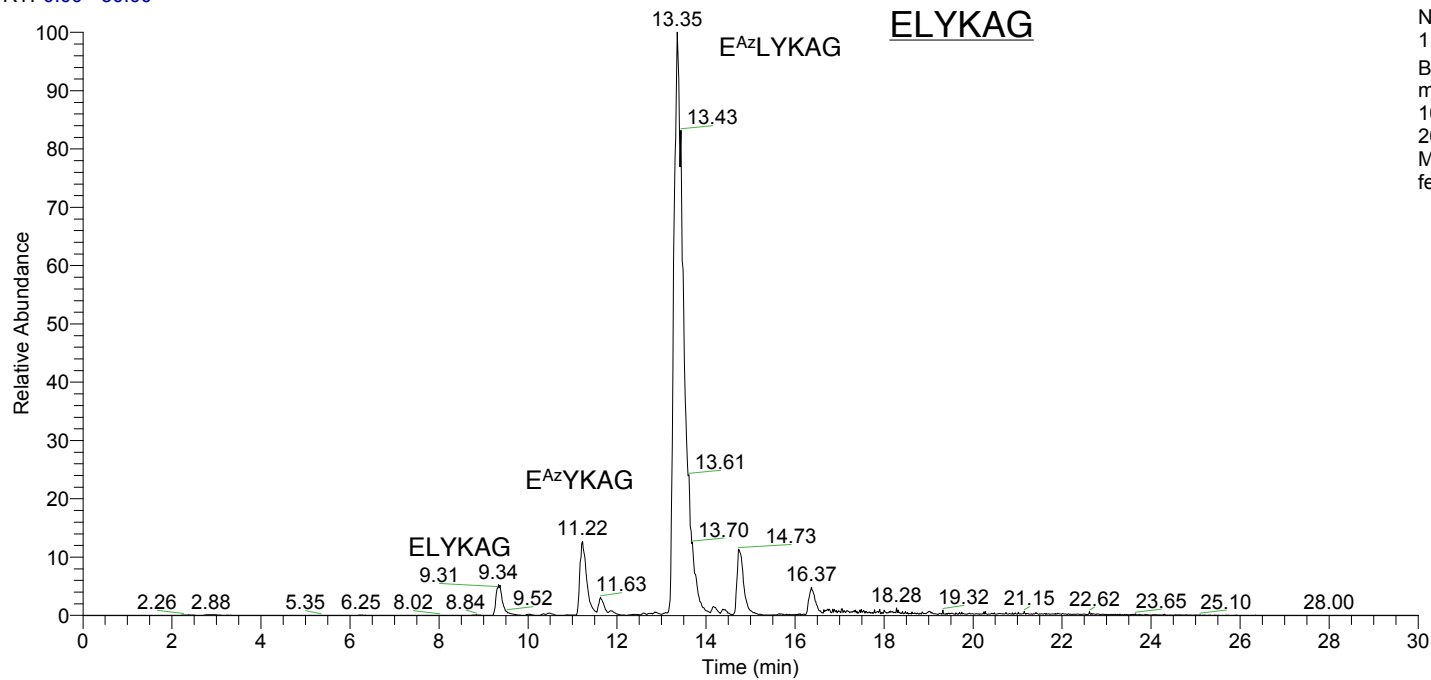
NL:
1.03E9
TIC F: MS
jan12-28jan12-28 #28-70 RT: 0.2-0.5 AV: 43 NL: 1.55E7
T: + p ESI Full ms [50.00-1000.00]

ELYKAG**==== Shimadzu LCsolution Analysis Report ====**

D:\My docs\Morten_2\HPLC\2012\jan12\250112\250112-28.lcd
 Acquired by : user
 Sample Name : B-30
 Sample ID : Glu - diazotized
 Tray# : 1
 Vial # : 21
 Injection Volume : 10 uL
 Data File Name : 250112-28.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 250112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 26-Jan-12 21:51:55
 Data Processed : 31-Jan-12 22:42:20

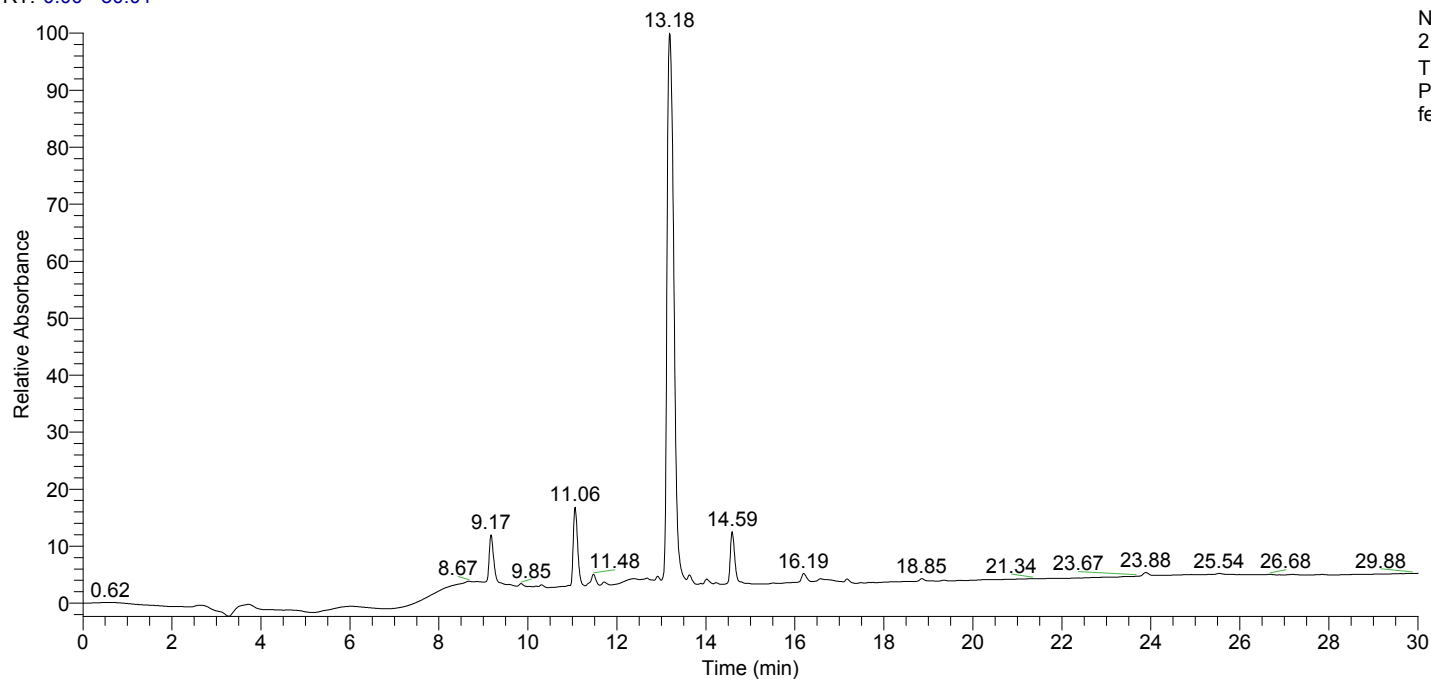


RT: 0.00 - 30.00



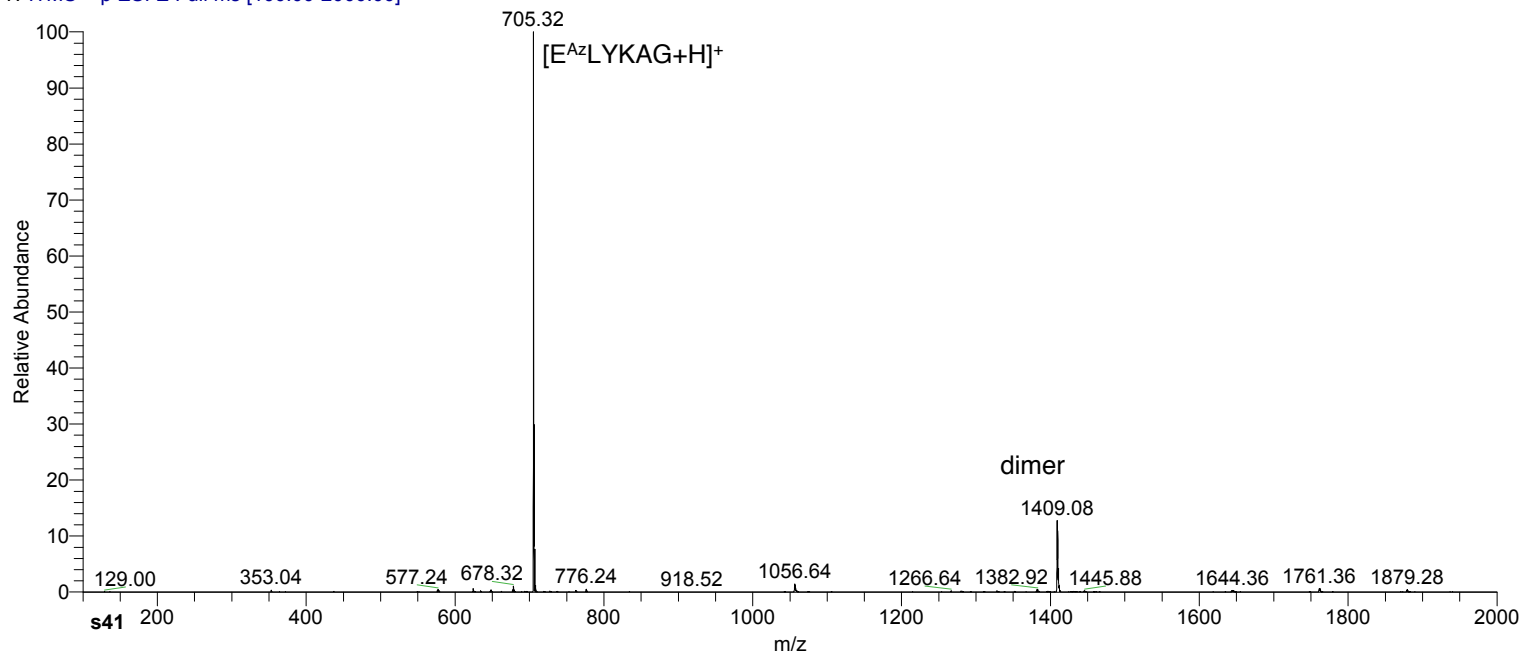
NL:
1.84E5
Base Peak
m/z=
100.00-
2000.00
MS
feb12-19

RT: 0.00 - 30.01



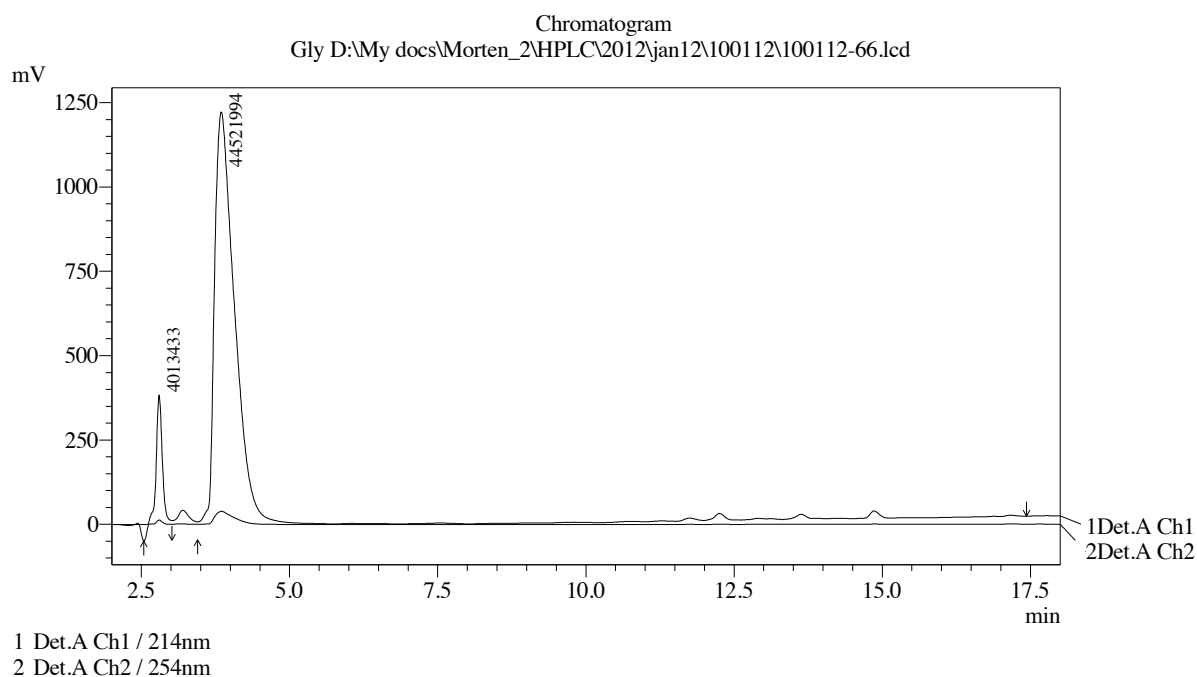
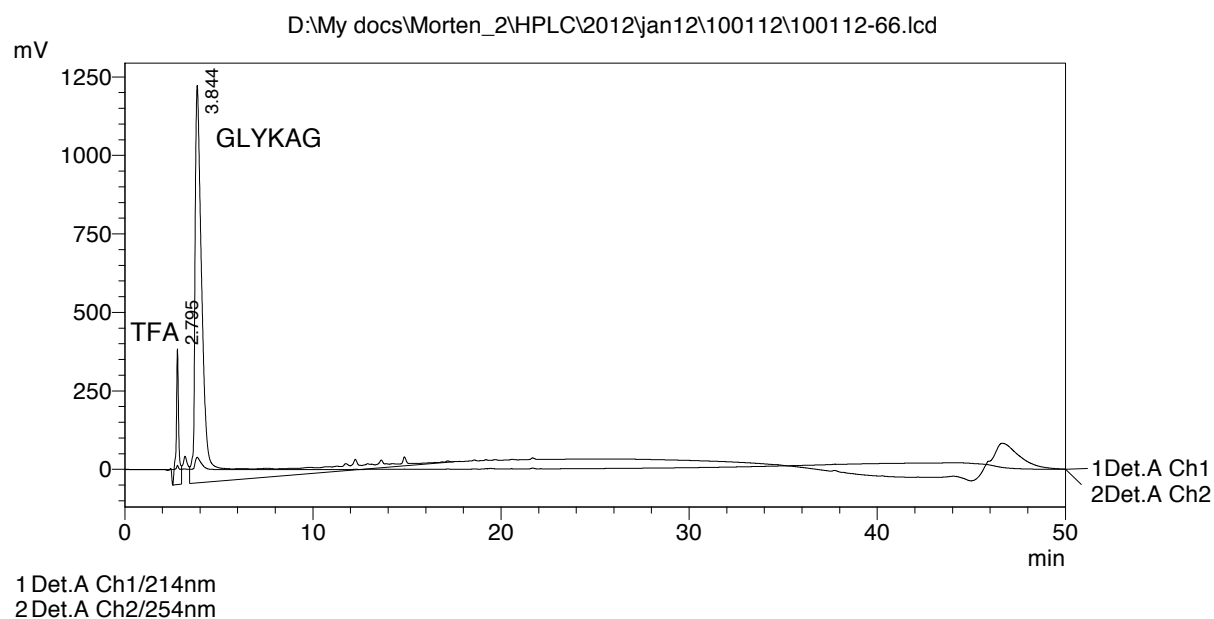
NL:
2.37E5
Total Scan
PDA
feb12-19

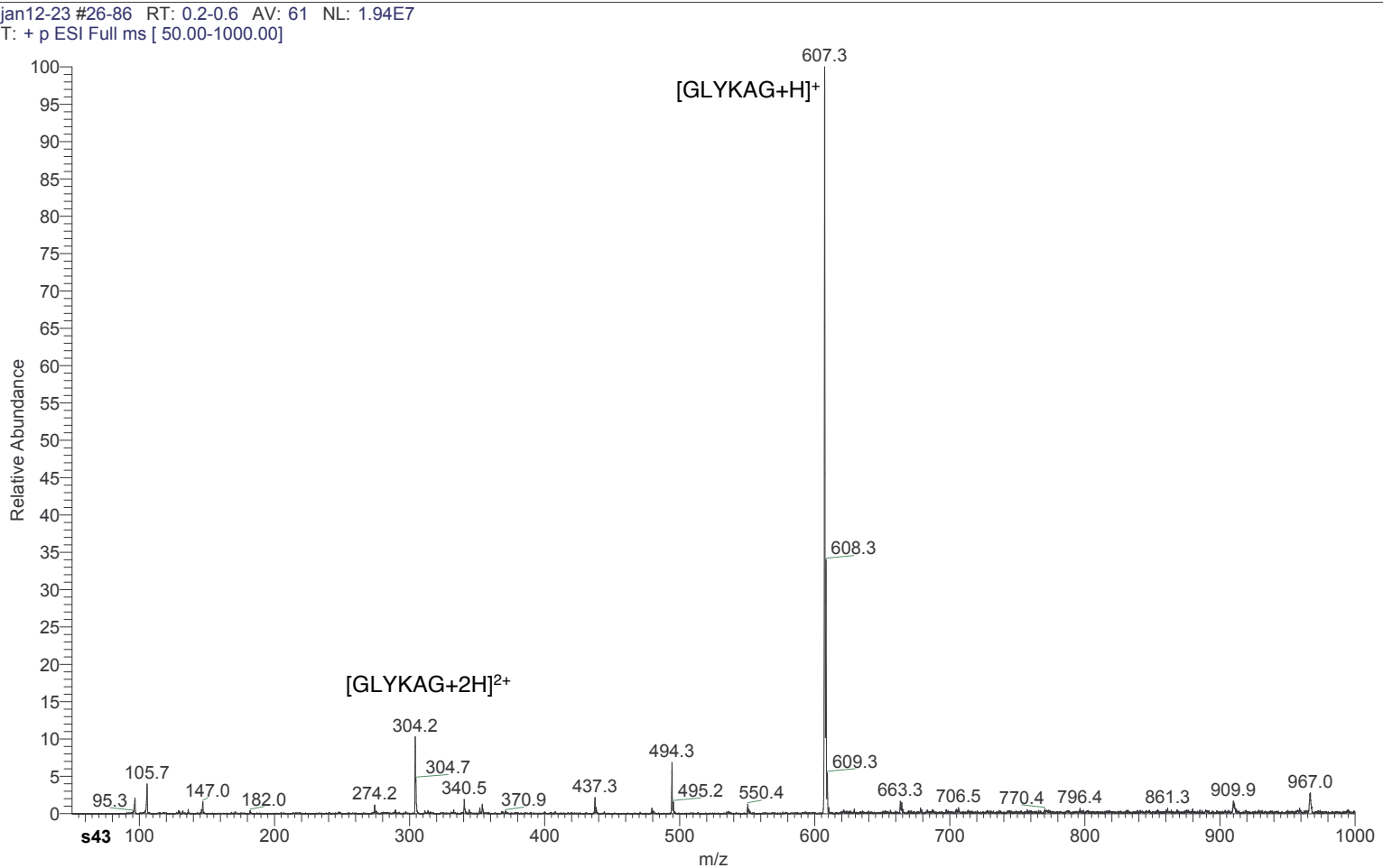
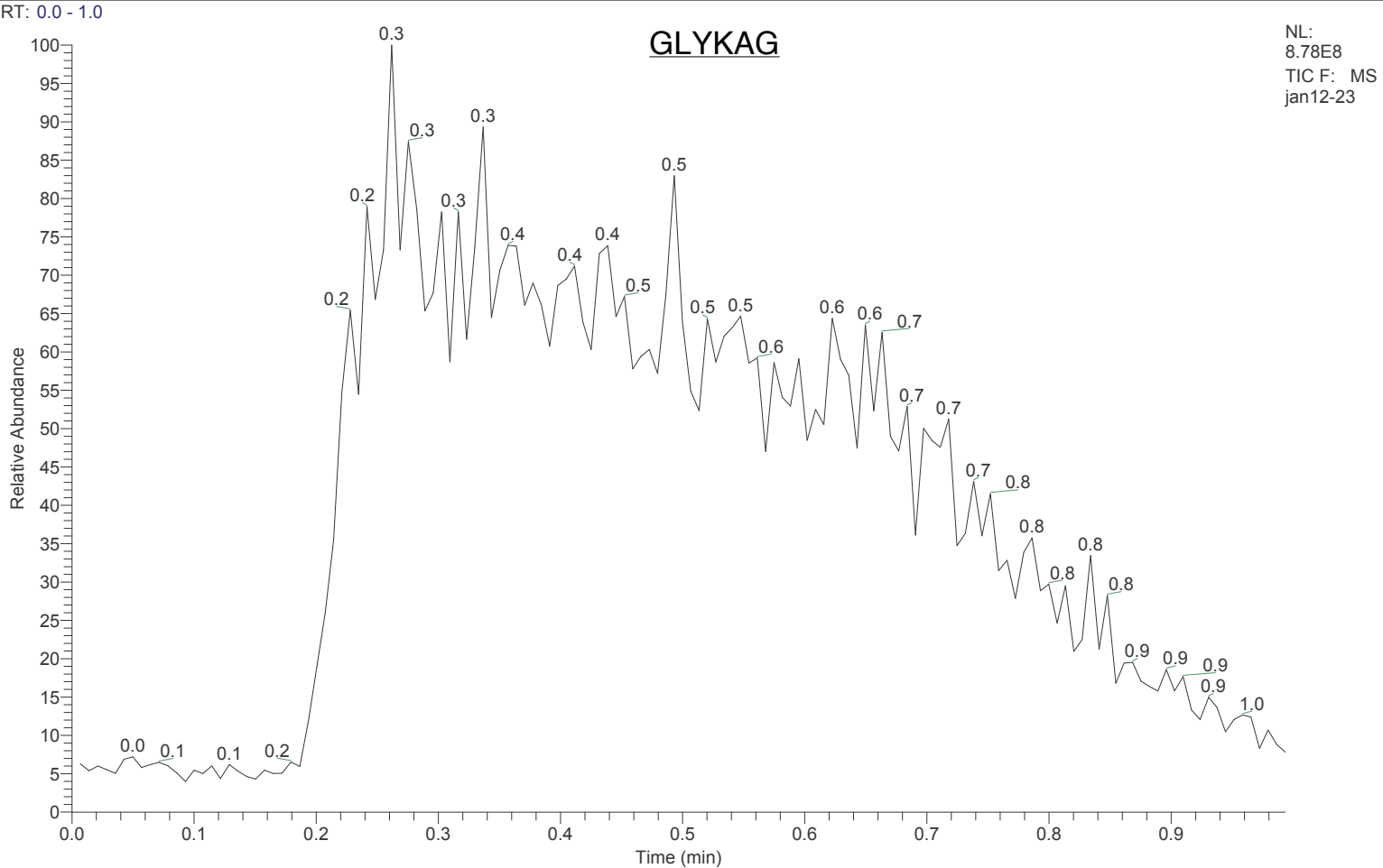
feb12-19 #709-748 RT: 13.14-13.78 AV: 40 NL: 7.29E4
T: ITMS + p ESI E Full ms [100.00-2000.00]



GLYKAG**==== Shimadzu LCsolution Analysis Report ====**

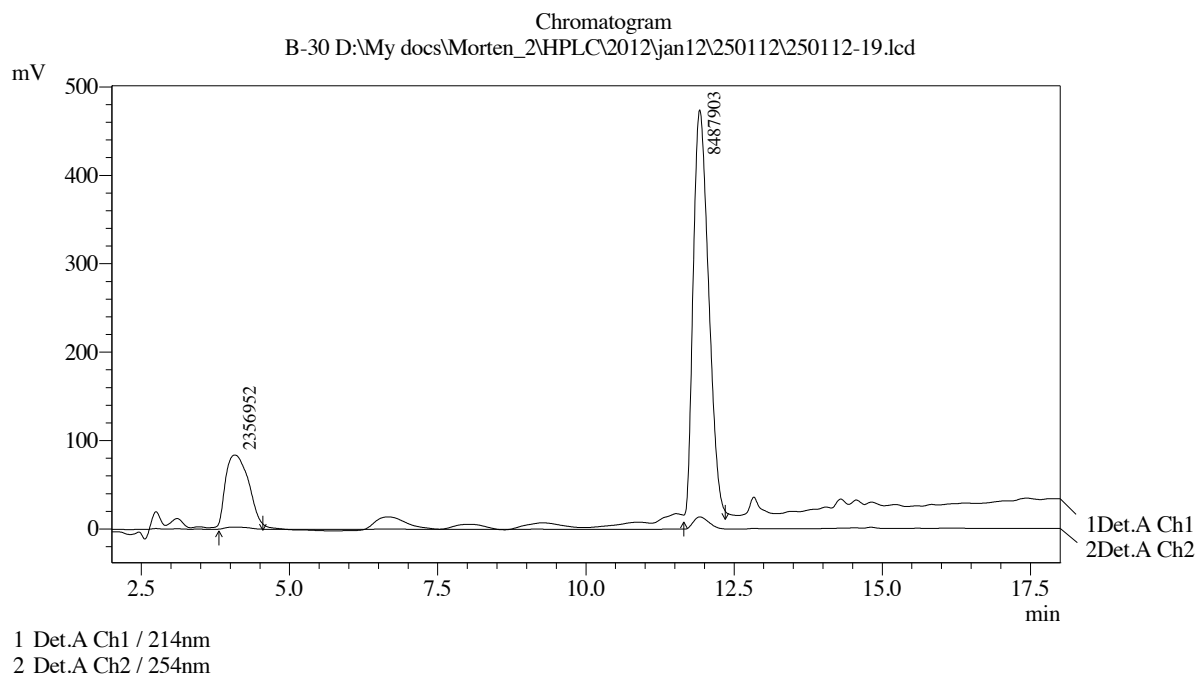
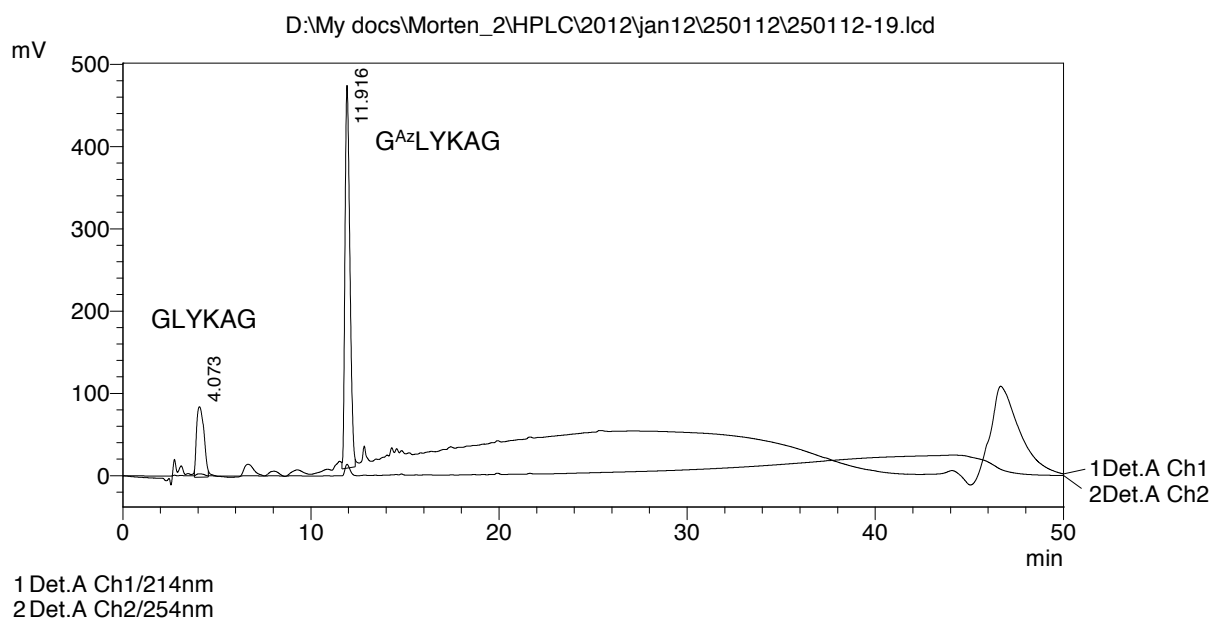
D:\My docs\Morten_2\HPLC\2012\jan12\100112\100112-66.lcd
 Acquired by : user
 Sample Name : Gly
 Sample ID : bef. diazotization
 Tray# : 1
 Vial # : 84
 Injection Volume : 10 uL
 Data File Name : 100112-66.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 100112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 13-Jan-12 5:03:29
 Data Processed : 13-Jan-12 5:53:35



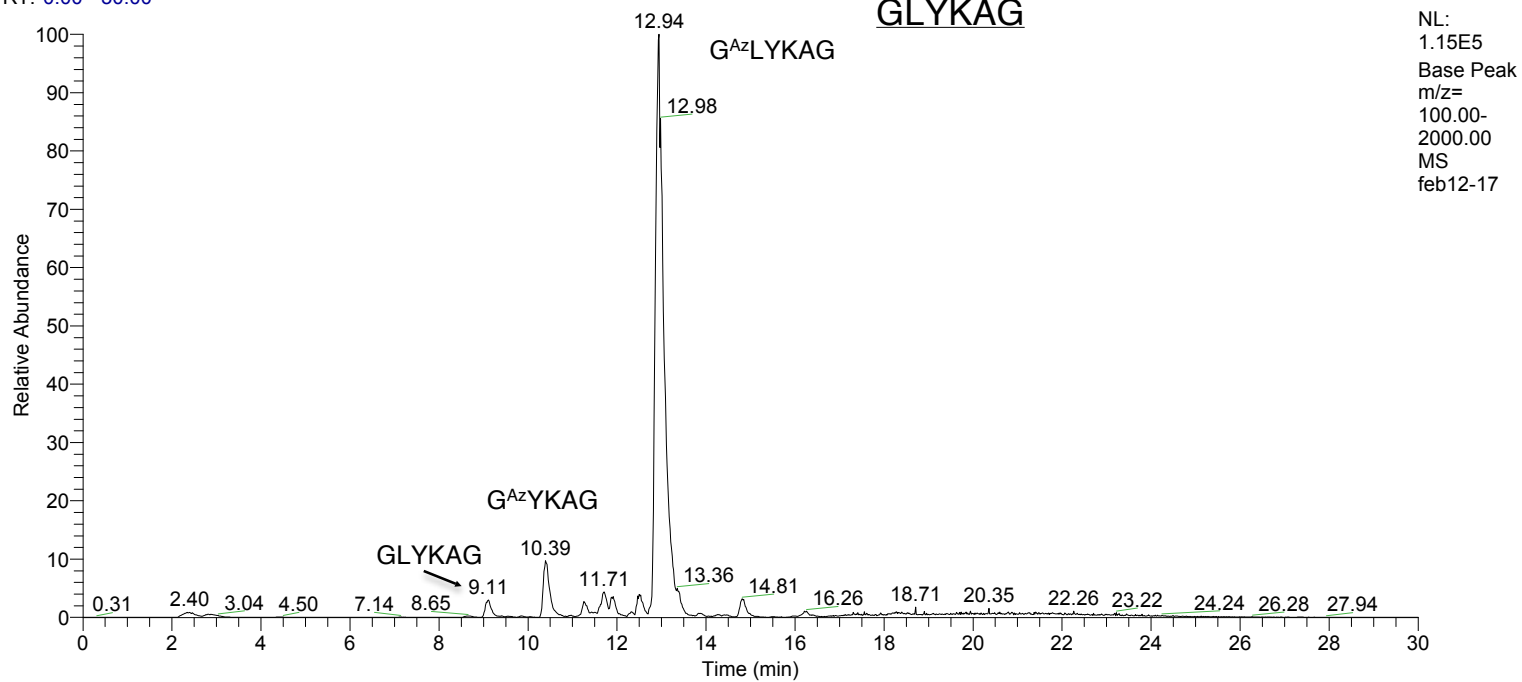


GLYKAG**==== Shimadzu LCsolution Analysis Report ====**

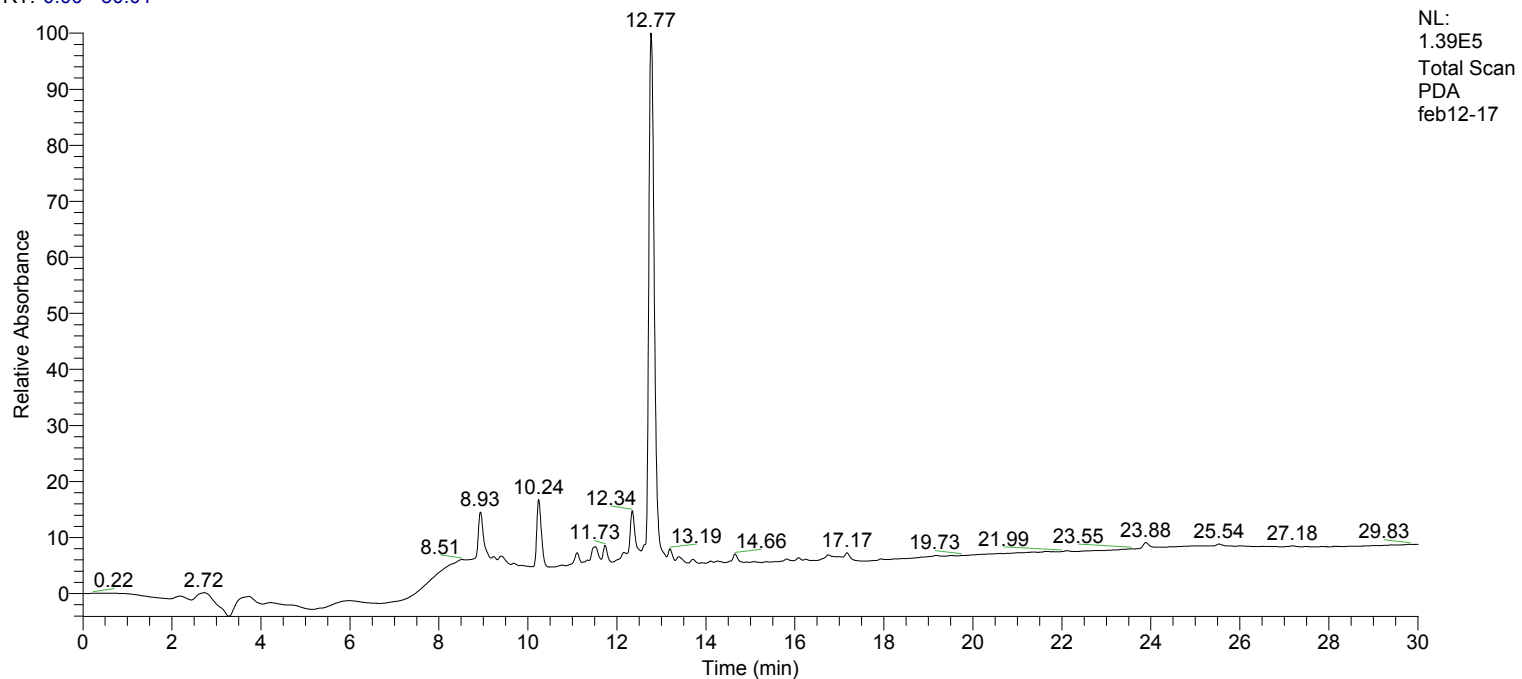
D:\My docs\Morten_2\HPLC\2012\jan12\250112\250112-19.lcd
 Acquired by : user
 Sample Name : B-30
 Sample ID : Gly - diazotized
 Tray# : 1
 Vial # : 94
 Injection Volume : 10 uL
 Data File Name : 250112-19.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 250112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 26-Jan-12 14:17:49
 Data Processed : 31-Jan-12 22:18:34



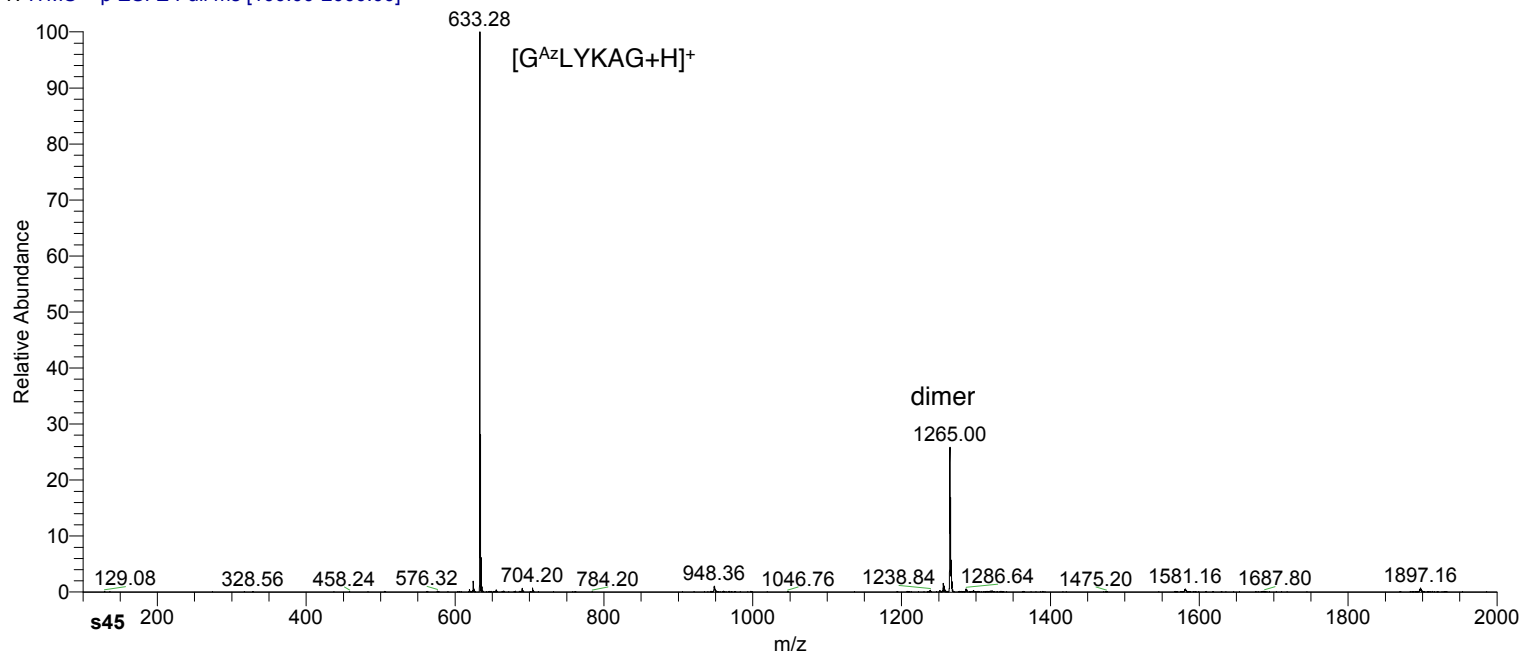
RT: 0.00 - 30.00



RT: 0.00 - 30.01

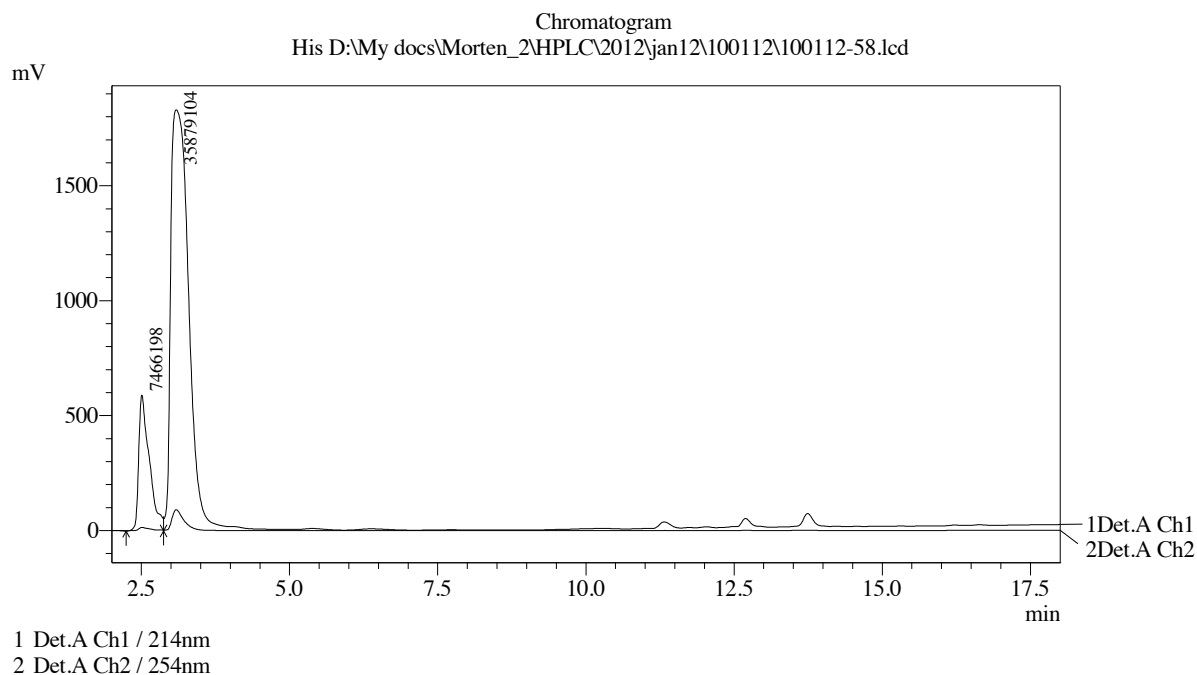
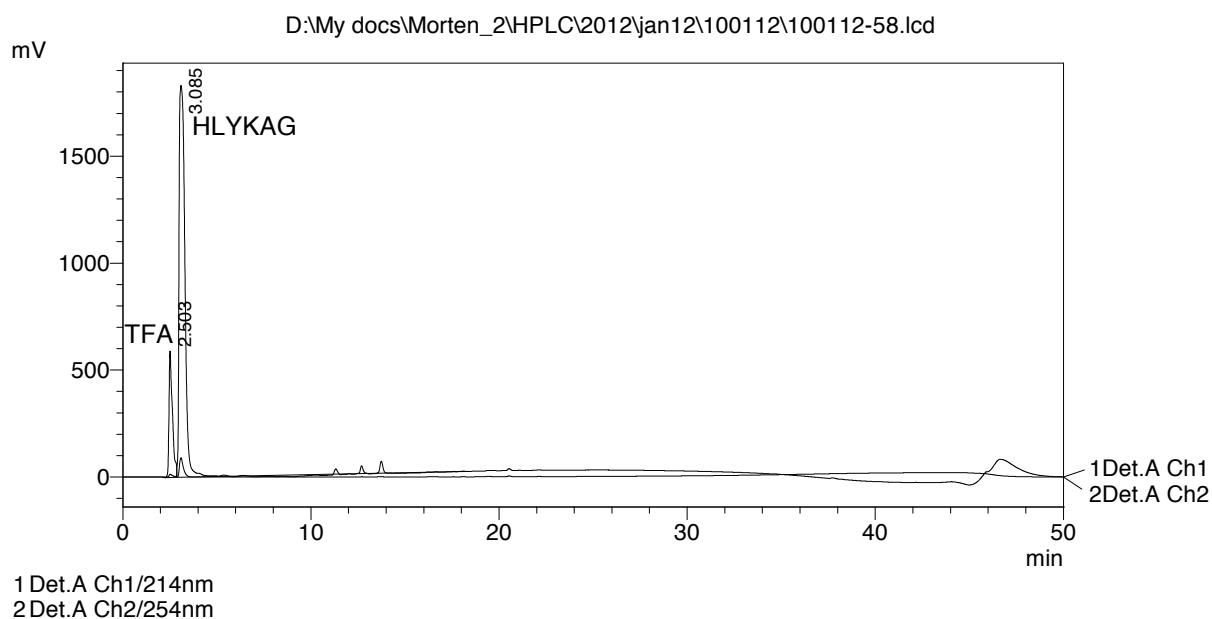


feb12-17 #687-722 RT: 12.73-13.31 AV: 36 NL: 4.32E4
T: ITMS + p ESI E Full ms [100.00-2000.00]

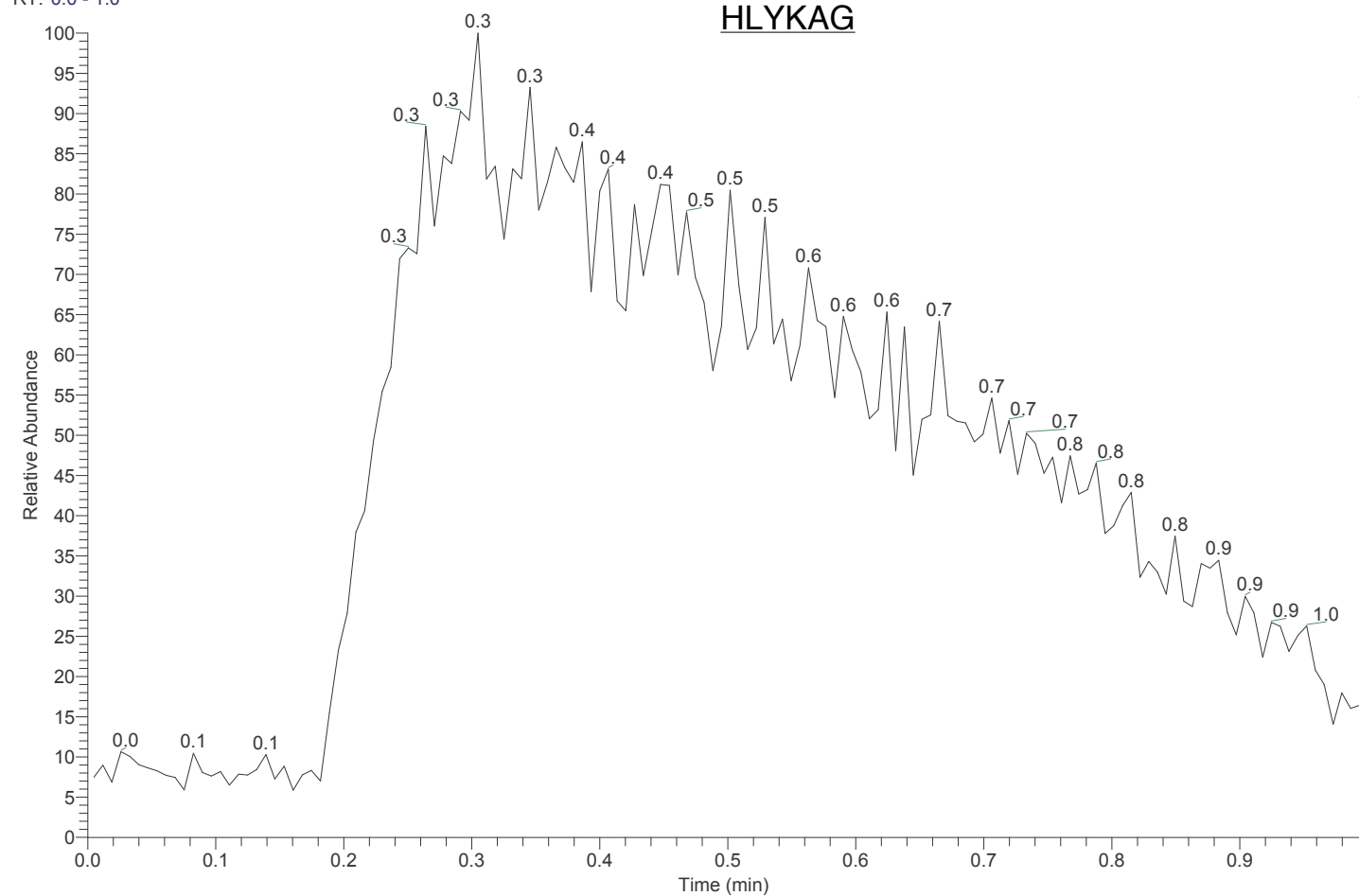


HLYKAG**==== Shimadzu LCsolution Analysis Report ====**

D:\My docs\Morten_2\HPLC\2012\jan12\100112\100112-58.lcd
 Acquired by : user
 Sample Name : His
 Sample ID : bef. diazotization
 Tray# : 1
 Vail # : 76
 Injection Volume : 10 uL
 Data File Name : 100112-58.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 100112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 12-Jan-12 22:20:01
 Data Processed : 12-Jan-12 23:10:05

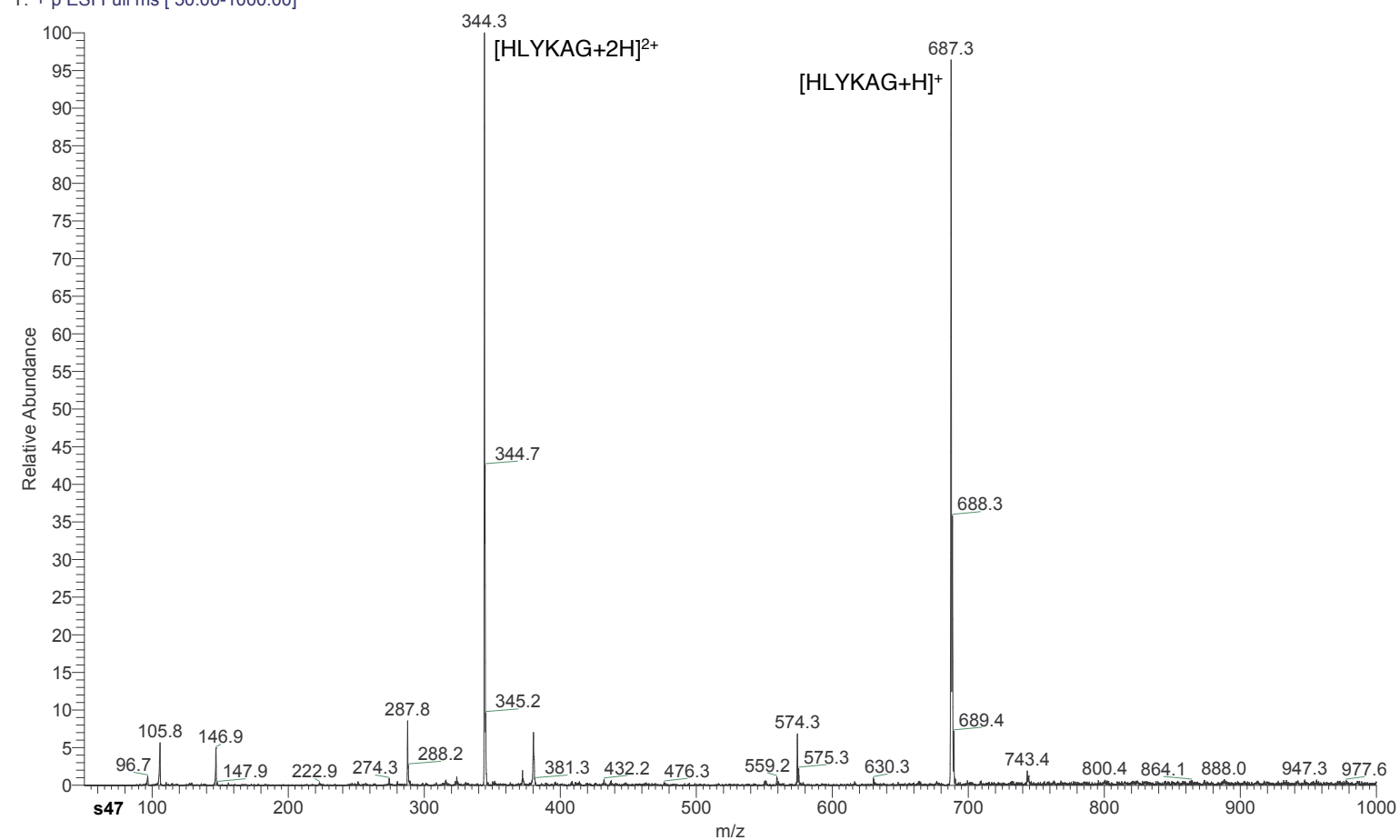


RT: 0.0 - 1.0



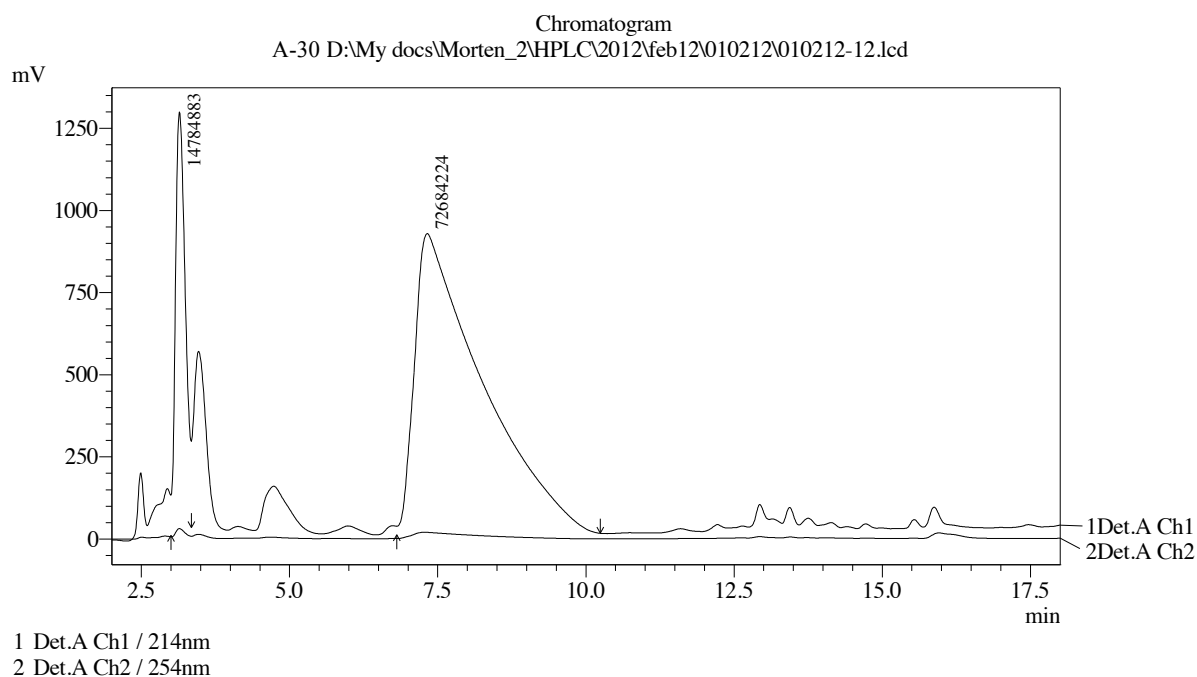
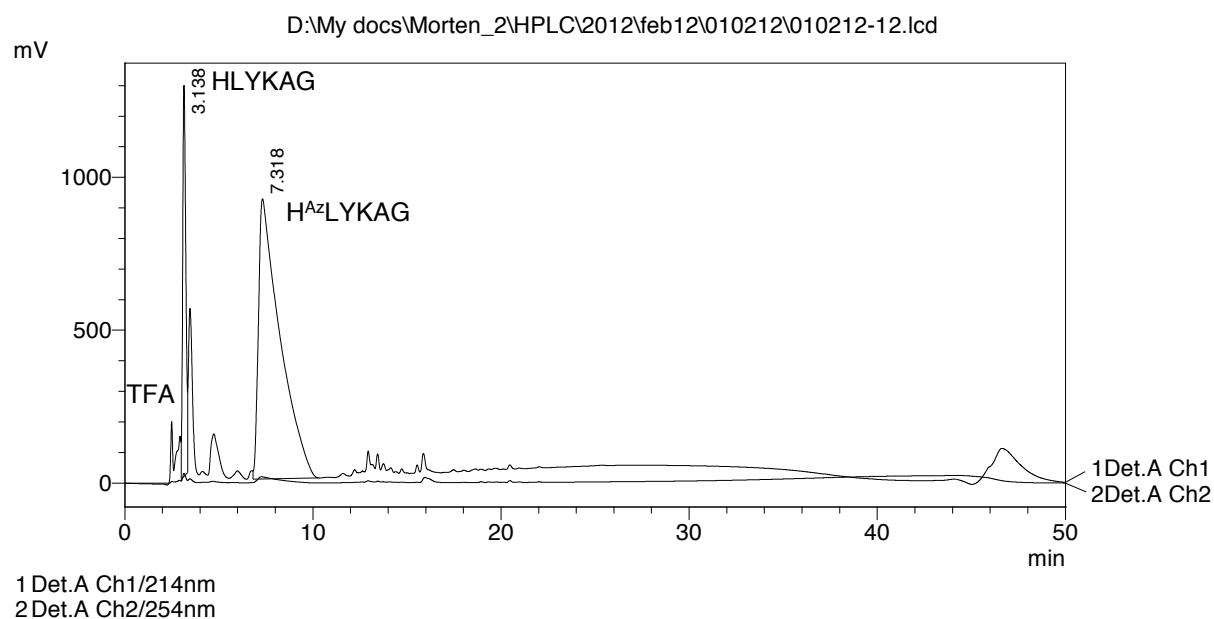
NL:
7.71E8
TIC F: MS
jan12-15

jan12-15 #25-85 RT: 0.2-0.6 AV: 61 NL: 1.41E7
T: + p ESI Full ms [50.00-1000.00]

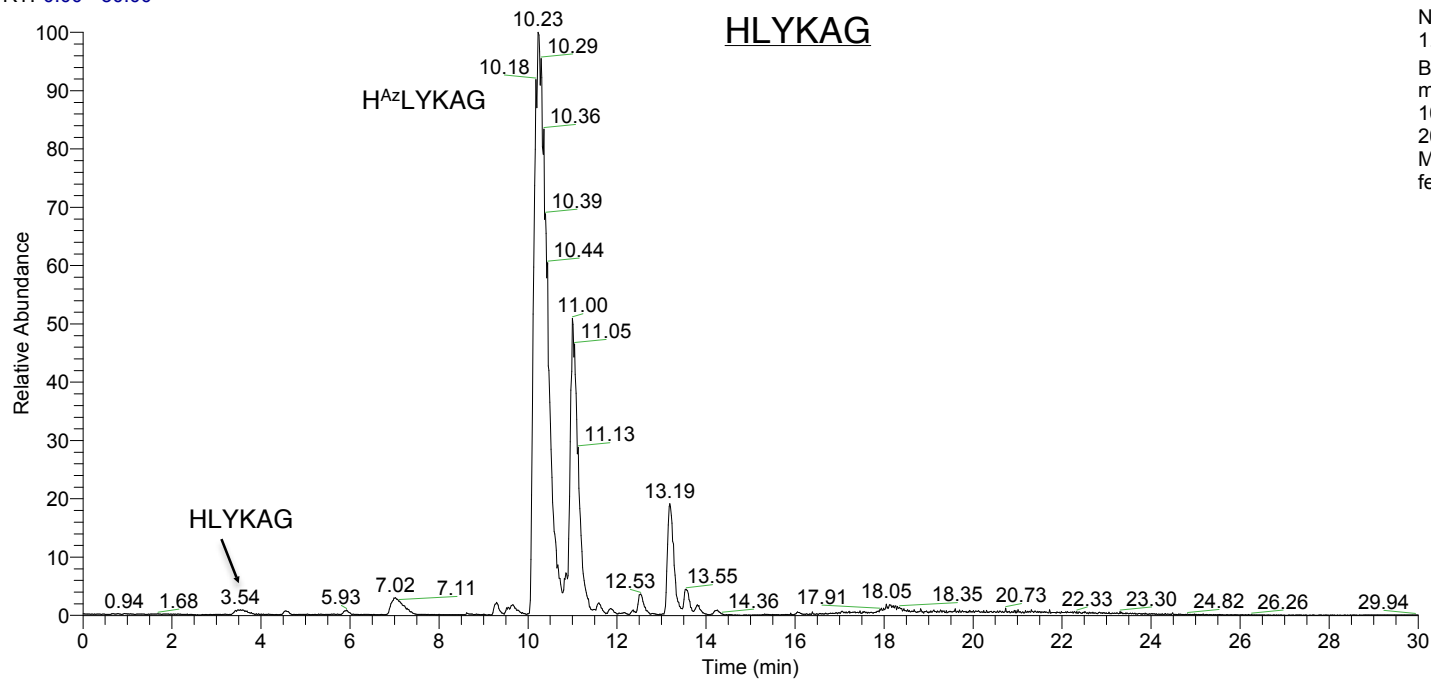


HLYKAG**==== Shimadzu LCsolution Analysis Report ====**

D:\My docs\Morten_2\HPLC\2012\feb12\010212\010212-12.lcd
 Acquired by : guest user
 Sample Name : A-30
 Sample ID : His - diazotized
 Tray# : 1
 Vail # : 12
 Injection Volume : 20 uL
 Data File Name : 010212-12.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 010212.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 02-Feb-12 4:46:07
 Data Processed : 05-Feb-12 20:00:17

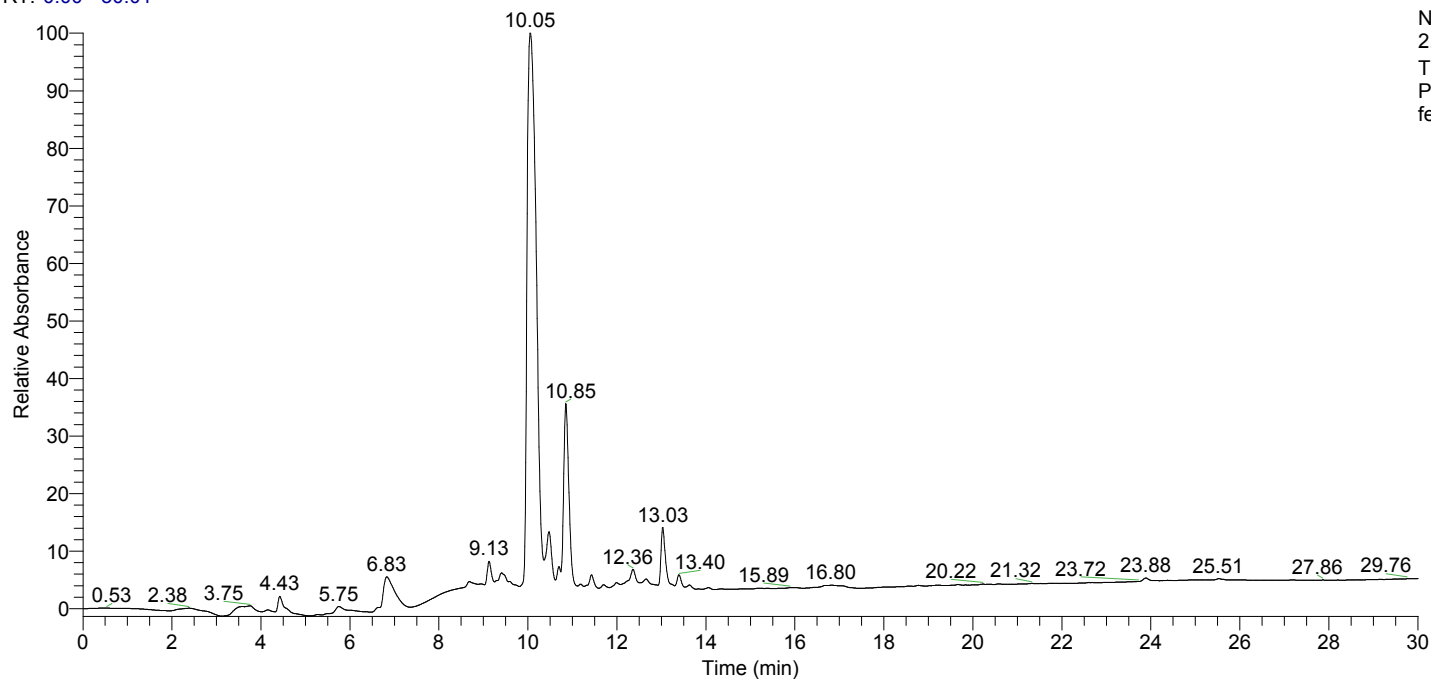


RT: 0.00 - 30.00



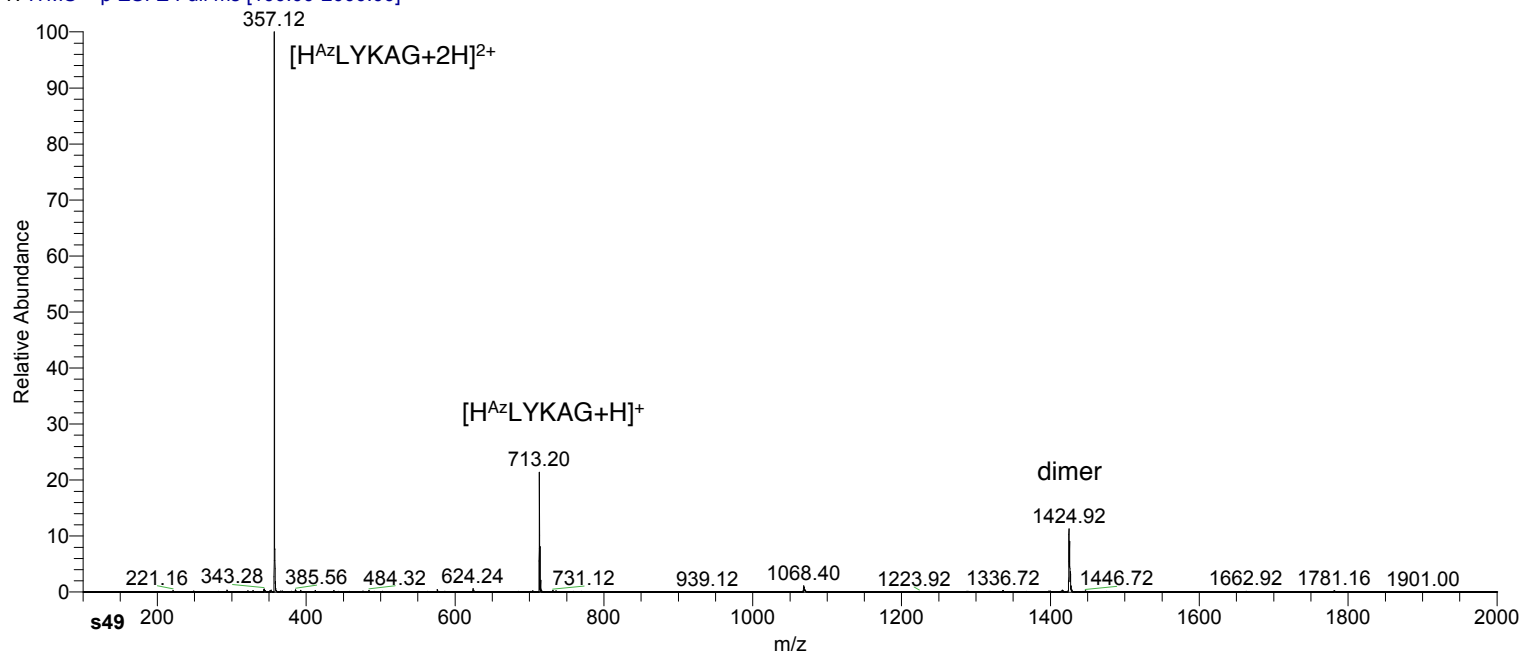
NL:
1.14E5
Base Peak
m/z=
100.00-
2000.00
MS
feb12-06

RT: 0.00 - 30.01



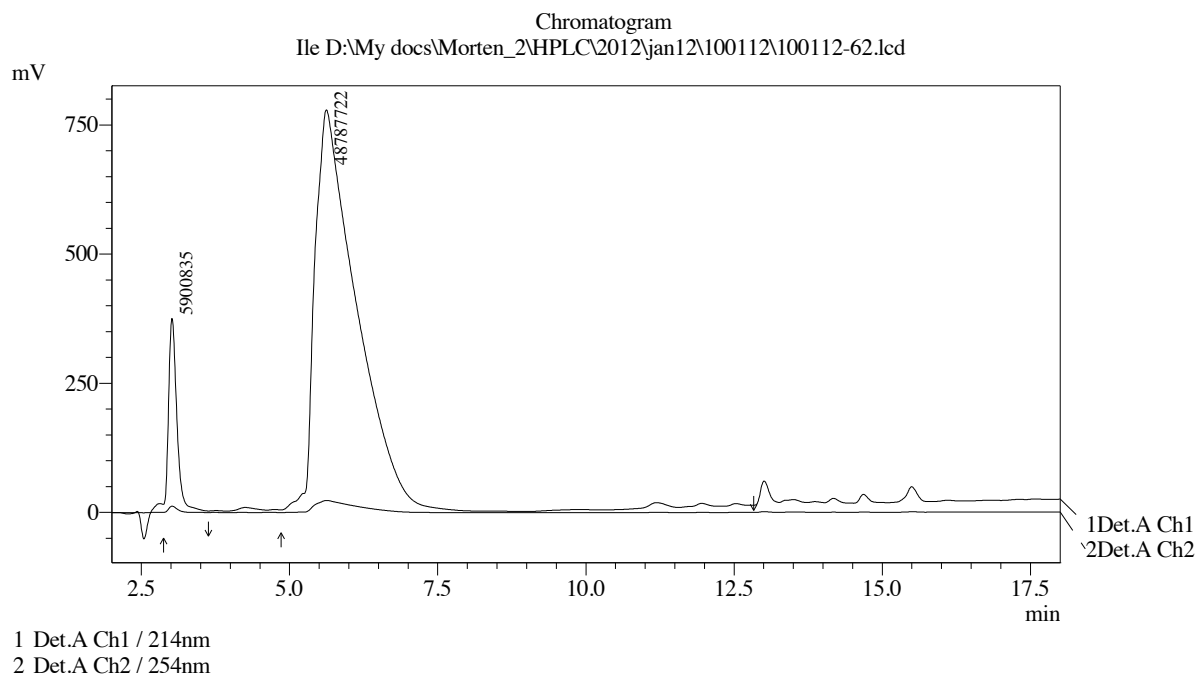
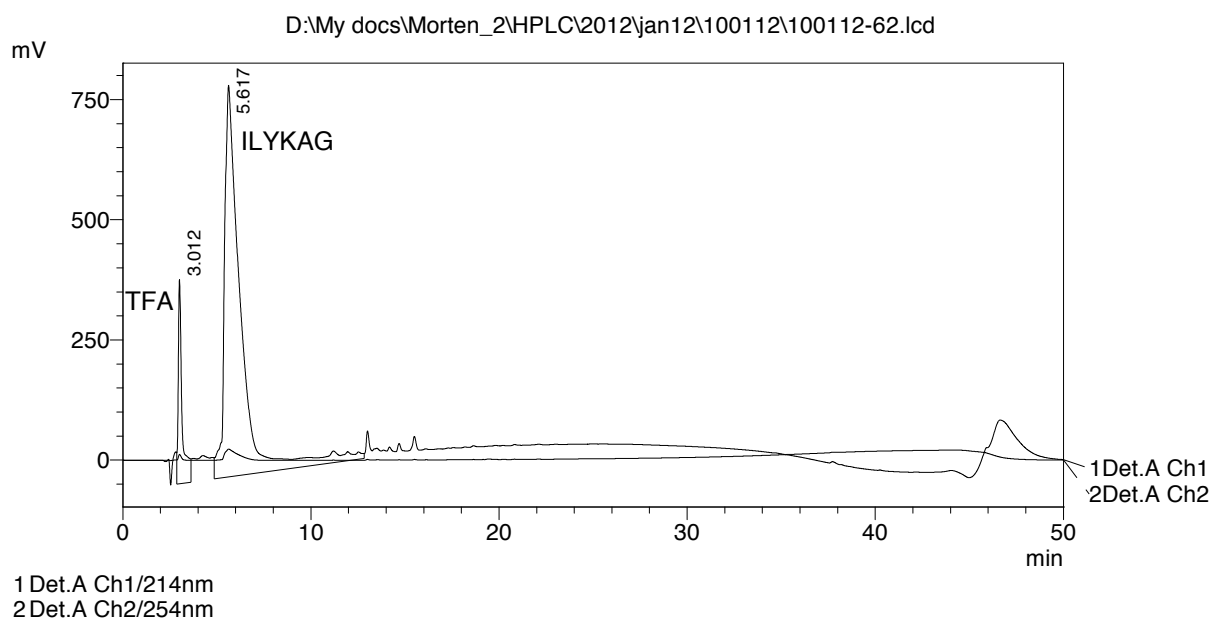
NL:
2.46E5
Total Scan
PDA
feb12-06

feb12-06 #547-575 RT: 10.11-10.57 AV: 29 NL: 7.49E4
T: ITMS + p ESI E Full ms [100.00-2000.00]

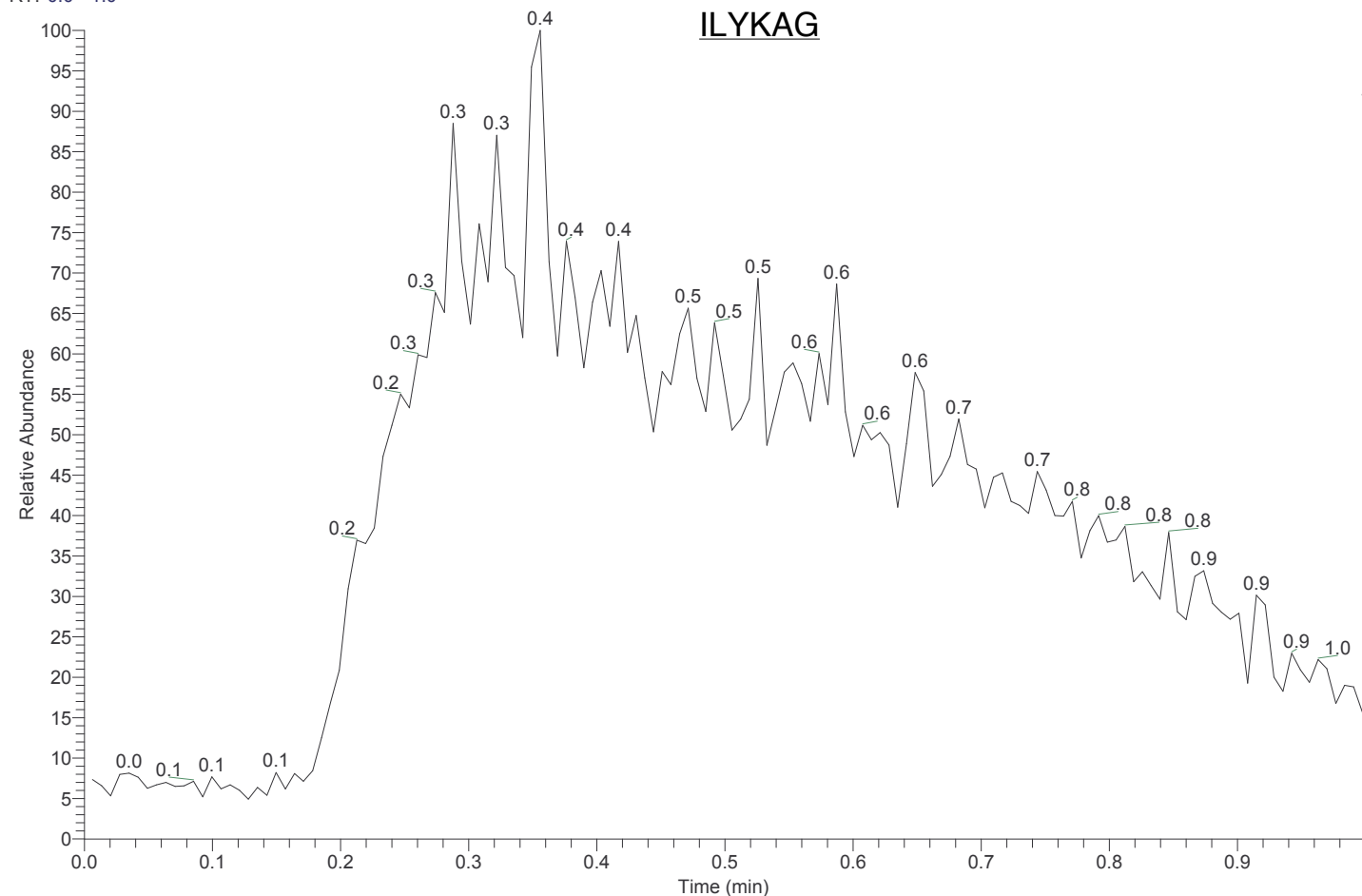
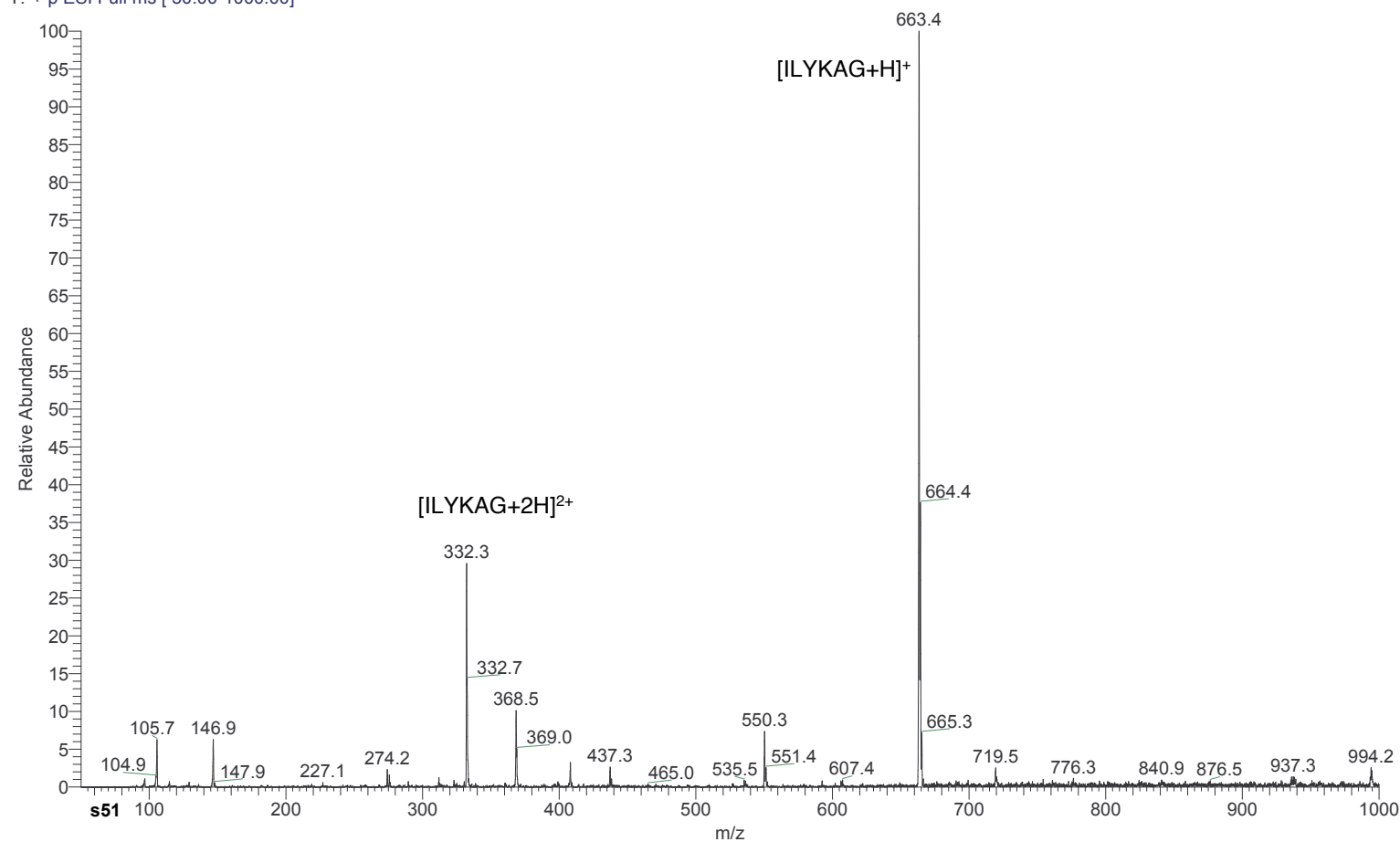


ILYKAG**==== Shimadzu LCsolution Analysis Report ====**

D:\My docs\Morten_2\HPLC\2012\jan12\100112\100112-62.lcd
 Acquired by : user
 Sample Name : Ile
 Sample ID : bef. diazotization
 Tray# : 1
 Vail # : 80
 Injection Volume : 10 uL
 Data File Name : 100112-62.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 100112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 13-Jan-12 1:41:45
 Data Processed : 13-Jan-12 2:31:49



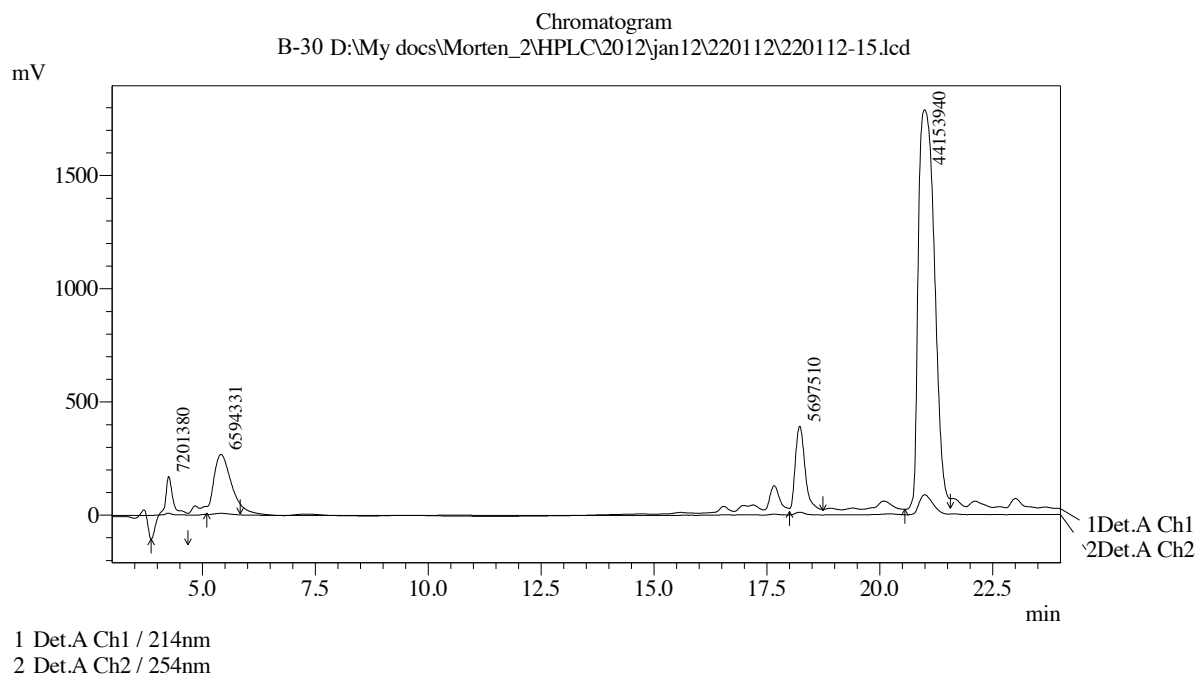
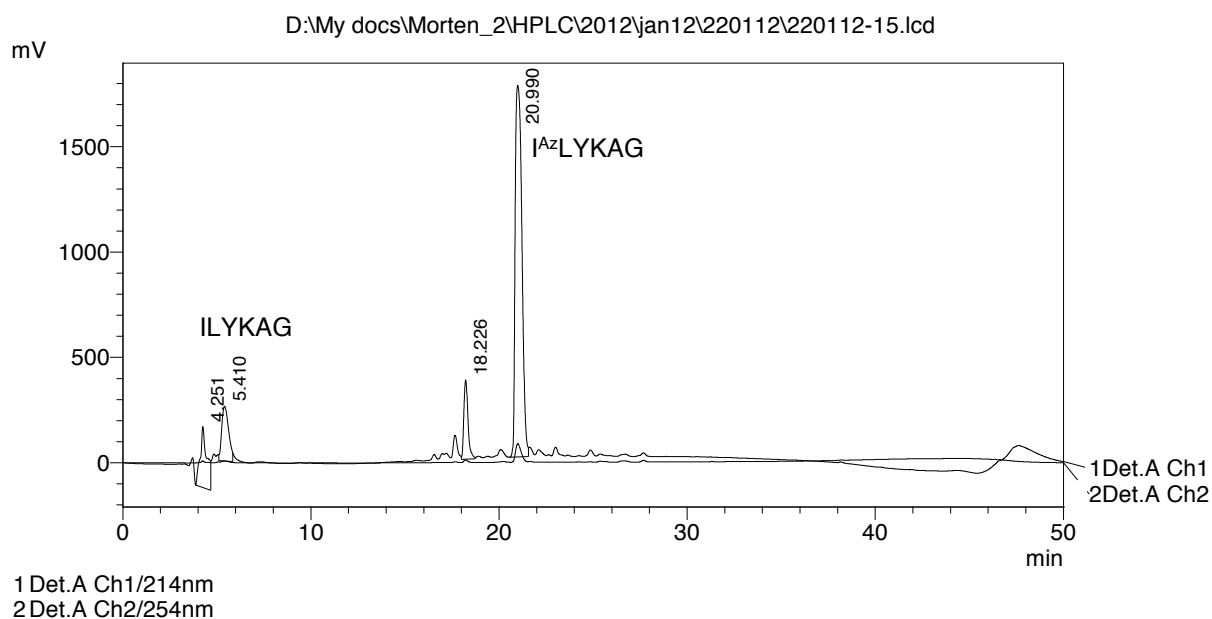
RT: 0.0 - 1.0

jan12-14 #26-70 RT: 0.2-0.5 AV: 45 NL: 1.28E7
T: + p ESI Full ms [50.00-1000.00]

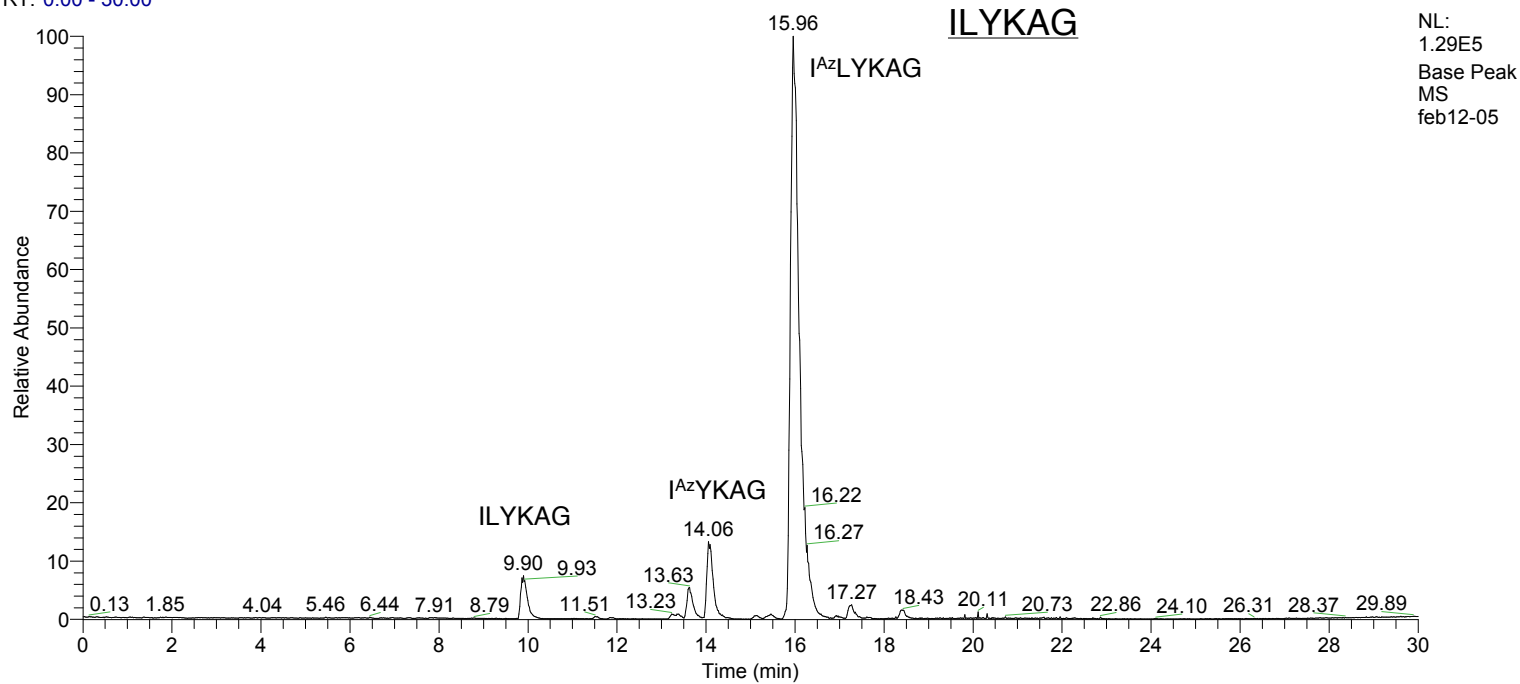
ILYKAG

==== Shimadzu LCsolution Analysis Report ====

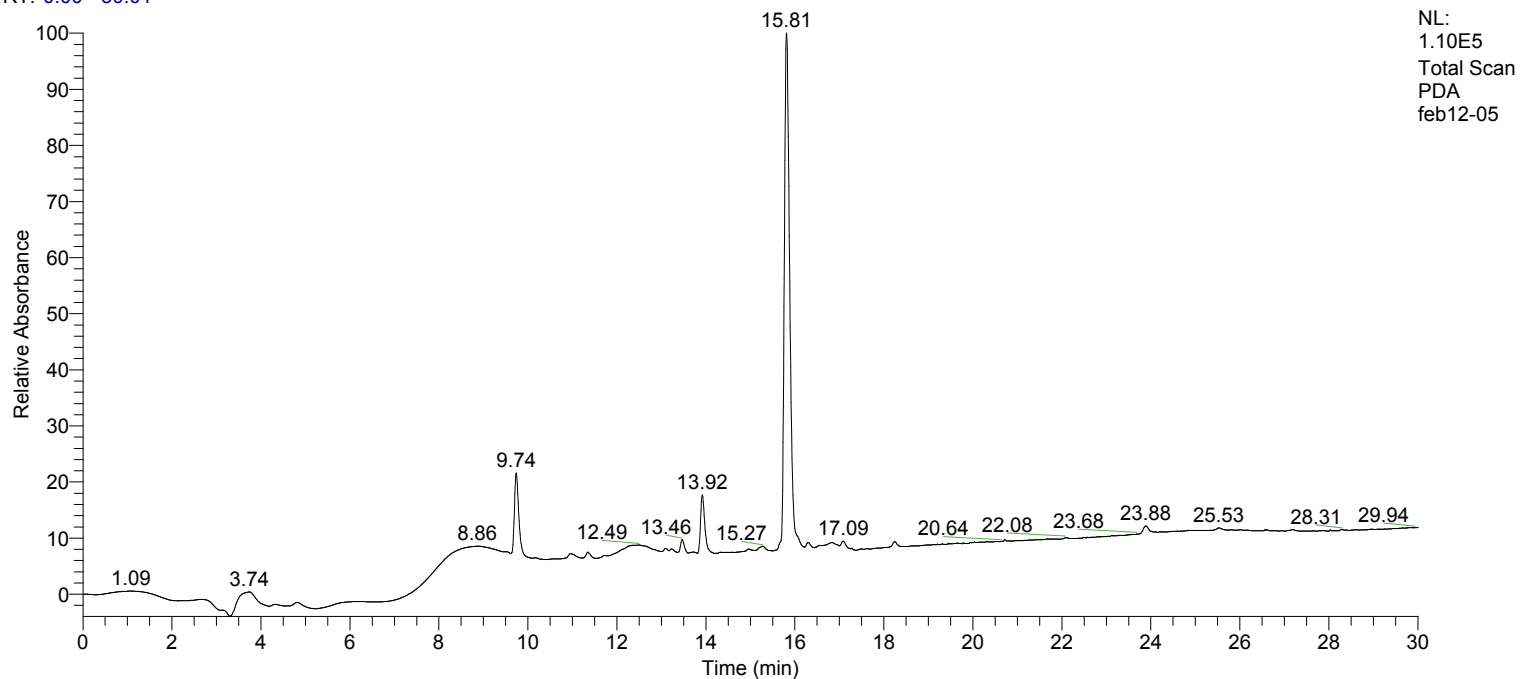
D:\My docs\Morten_2\HPLC\2012\jan12\220112\220112-15.lcd
 Acquired by : user
 Sample Name : B-30
 Sample ID : Ile - diazotized
 Tray# : 1
 Vial # : 74
 Injection Volume : 20 uL
 Data File Name : 220112-15.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 220112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 23-Jan-12 5:34:16
 Data Processed : 25-Jan-12 17:29:31



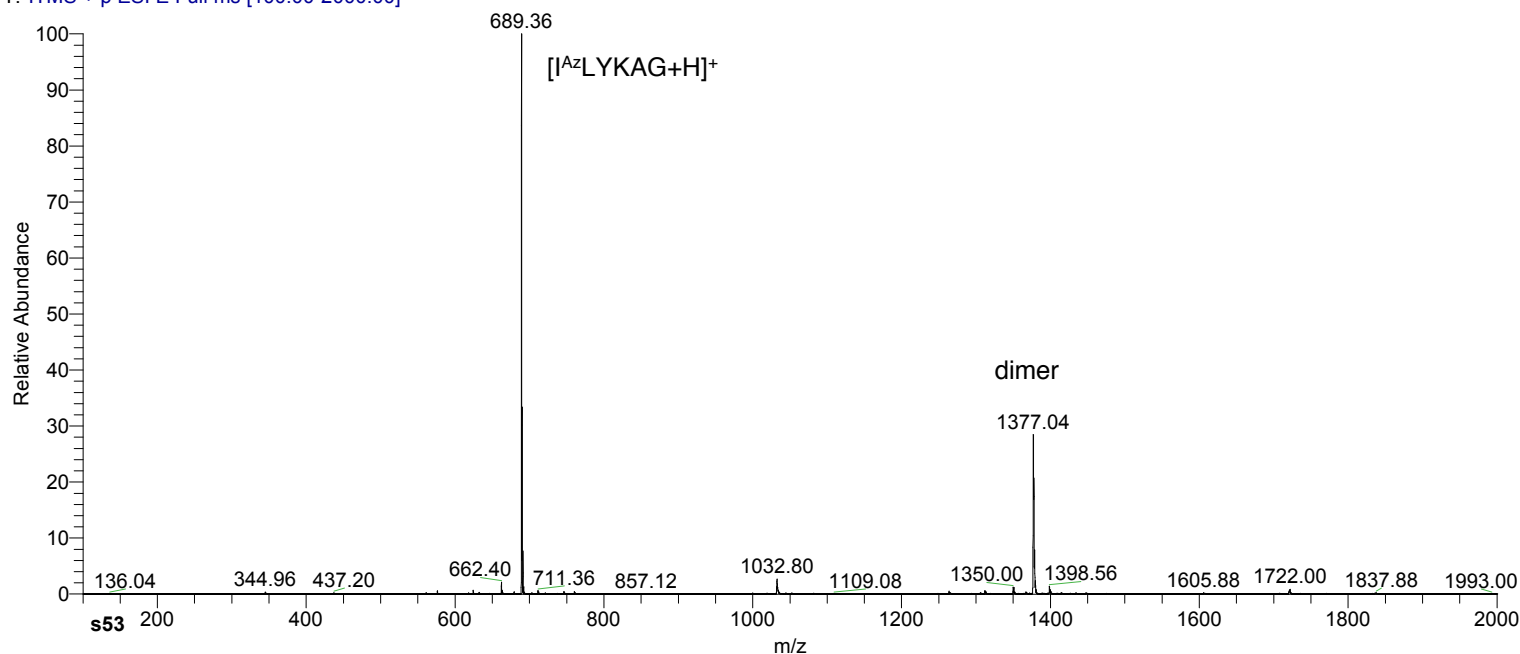
RT: 0.00 - 30.00



RT: 0.00 - 30.01

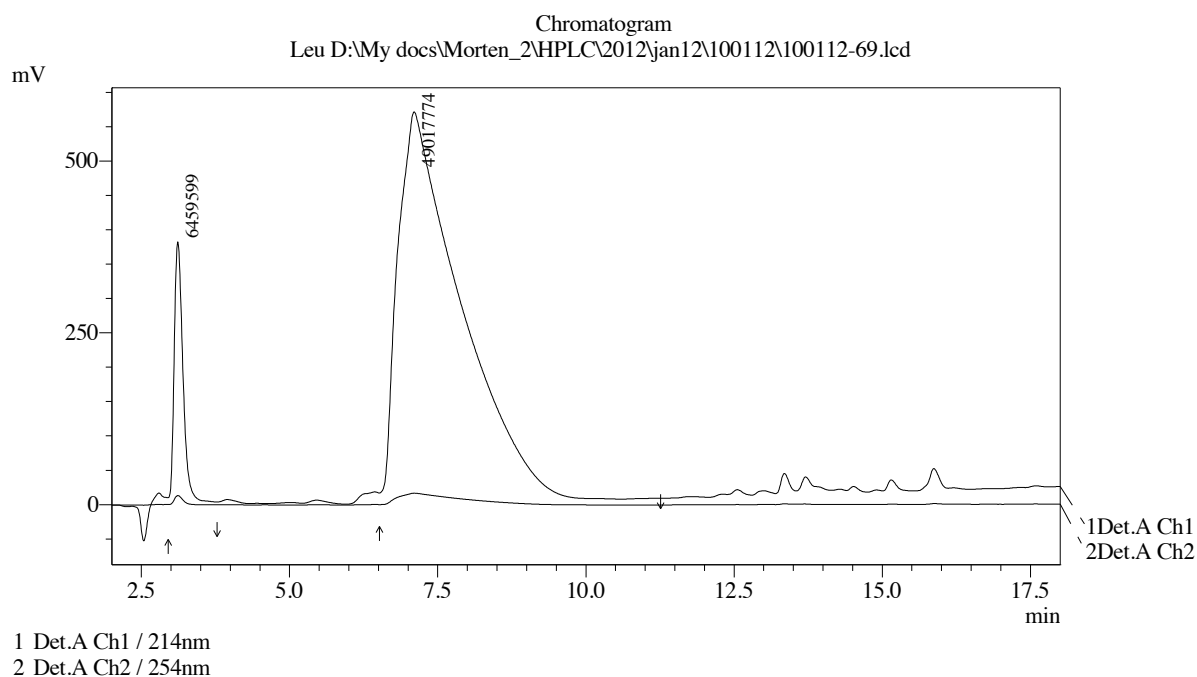
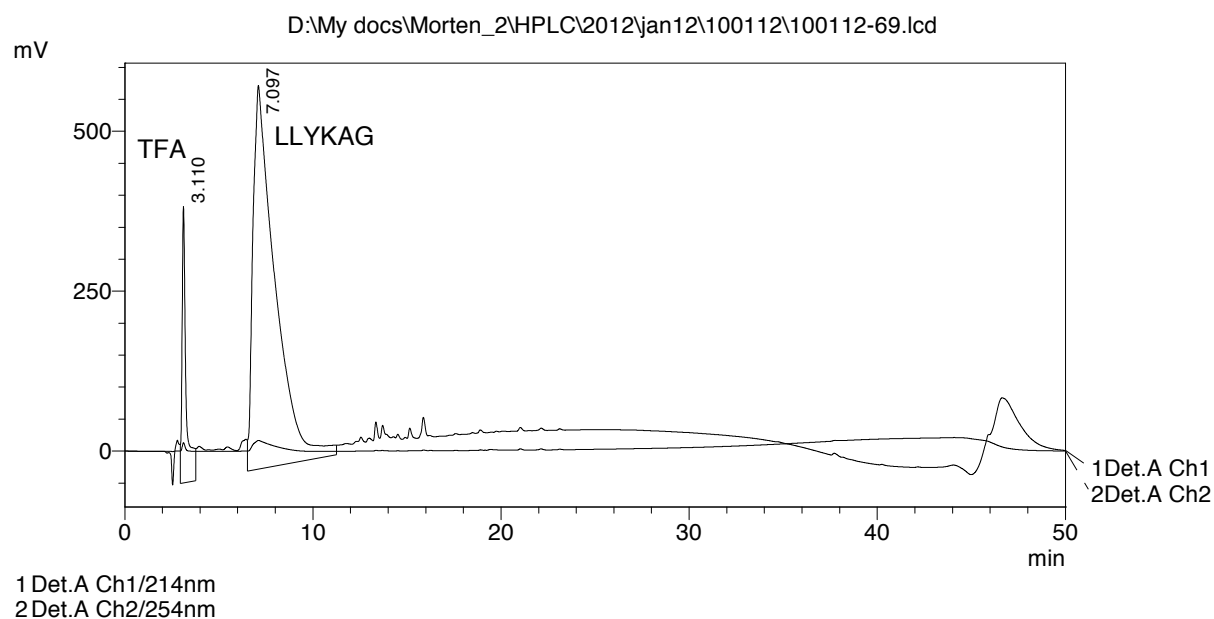


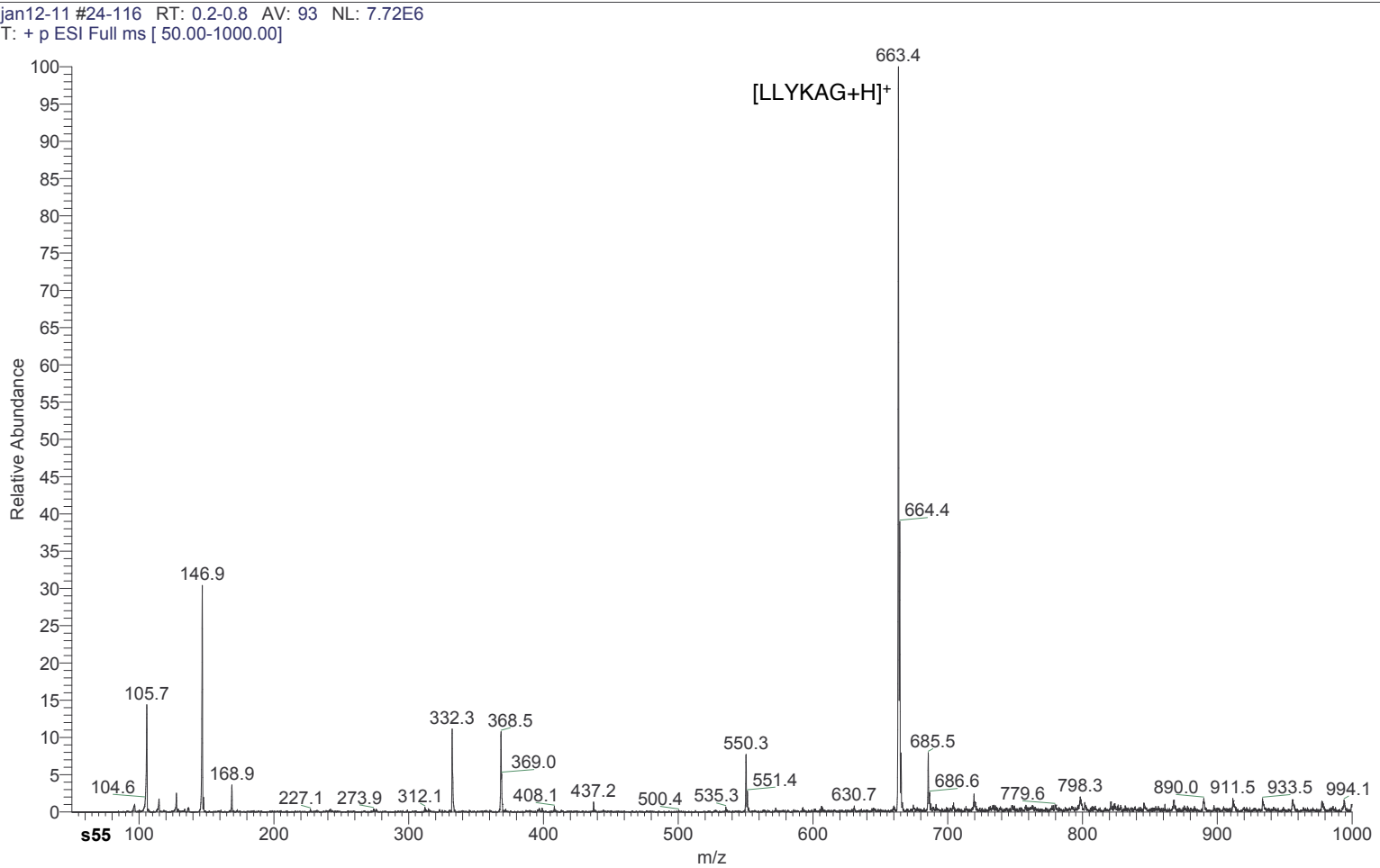
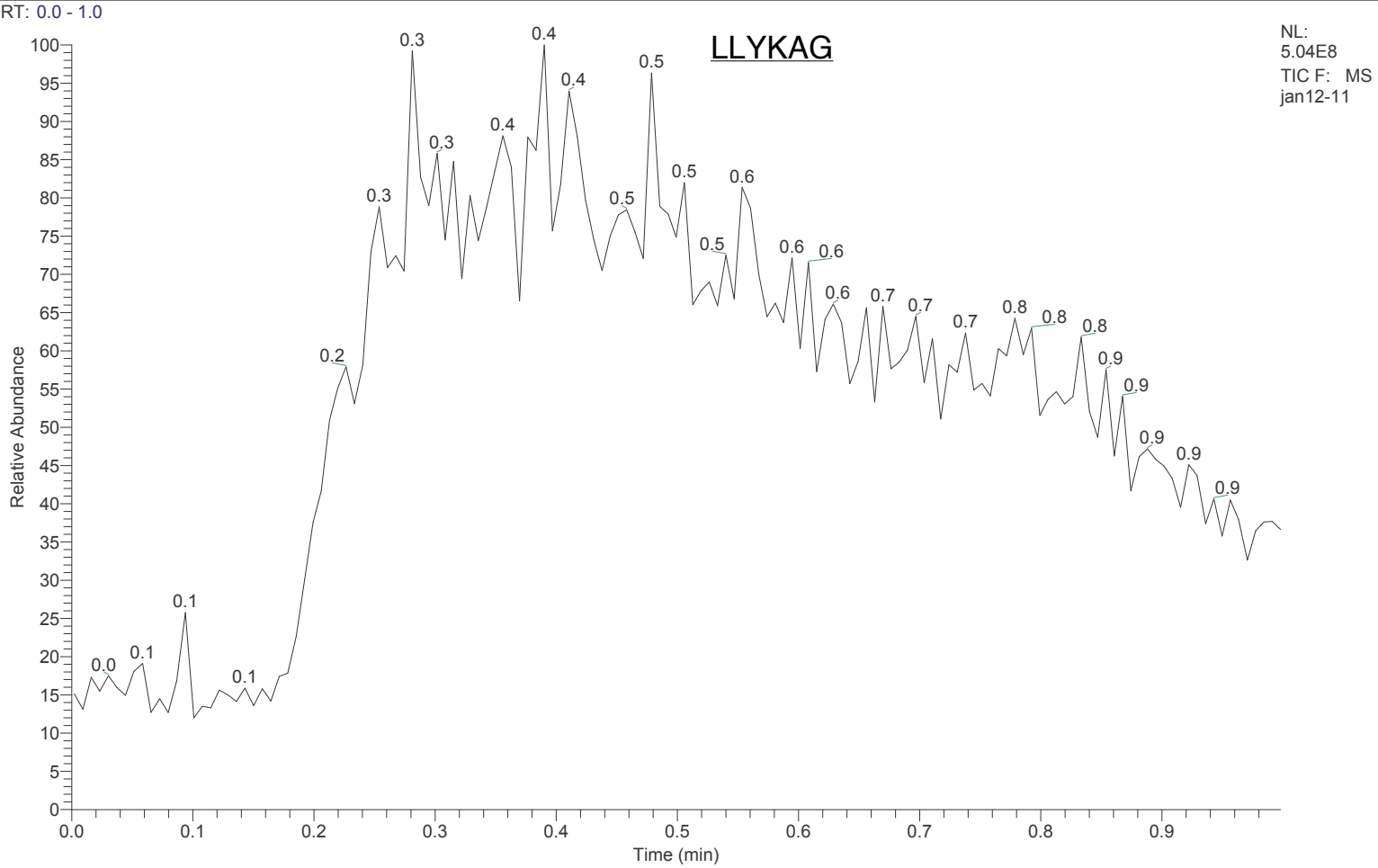
feb12-05 #850-877 RT: 15.85-16.29 AV: 28 NL: 6.30E4
T: ITMS + p ESI E Full ms [100.00-2000.00]



LLYKAG**==== Shimadzu LCsolution Analysis Report ====**

D:\My docs\Morten_2\HPLC\2012\jan12\100112\100112-69.lcd
 Acquired by : user
 Sample Name : Leu
 Sample ID : bef. diazotization
 Tray# : 1
 Vial # : 87
 Injection Volume : 10 uL
 Data File Name : 100112-69.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 100112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 13-Jan-12 7:34:50
 Data Processed : 13-Jan-12 8:24:55

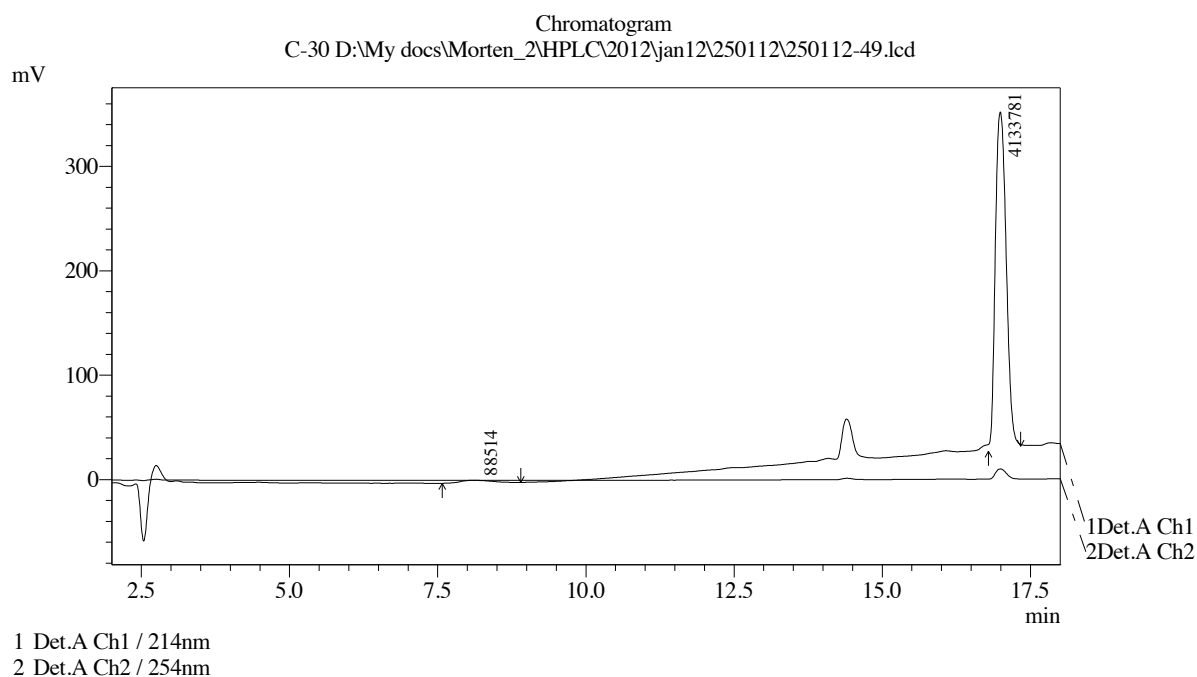
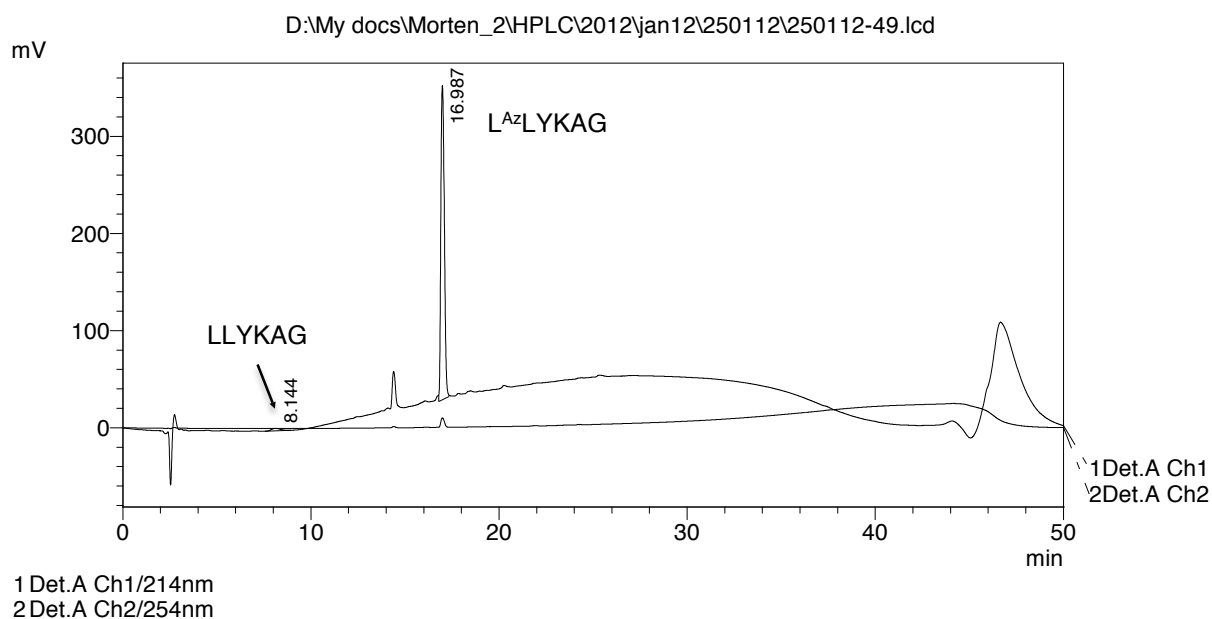




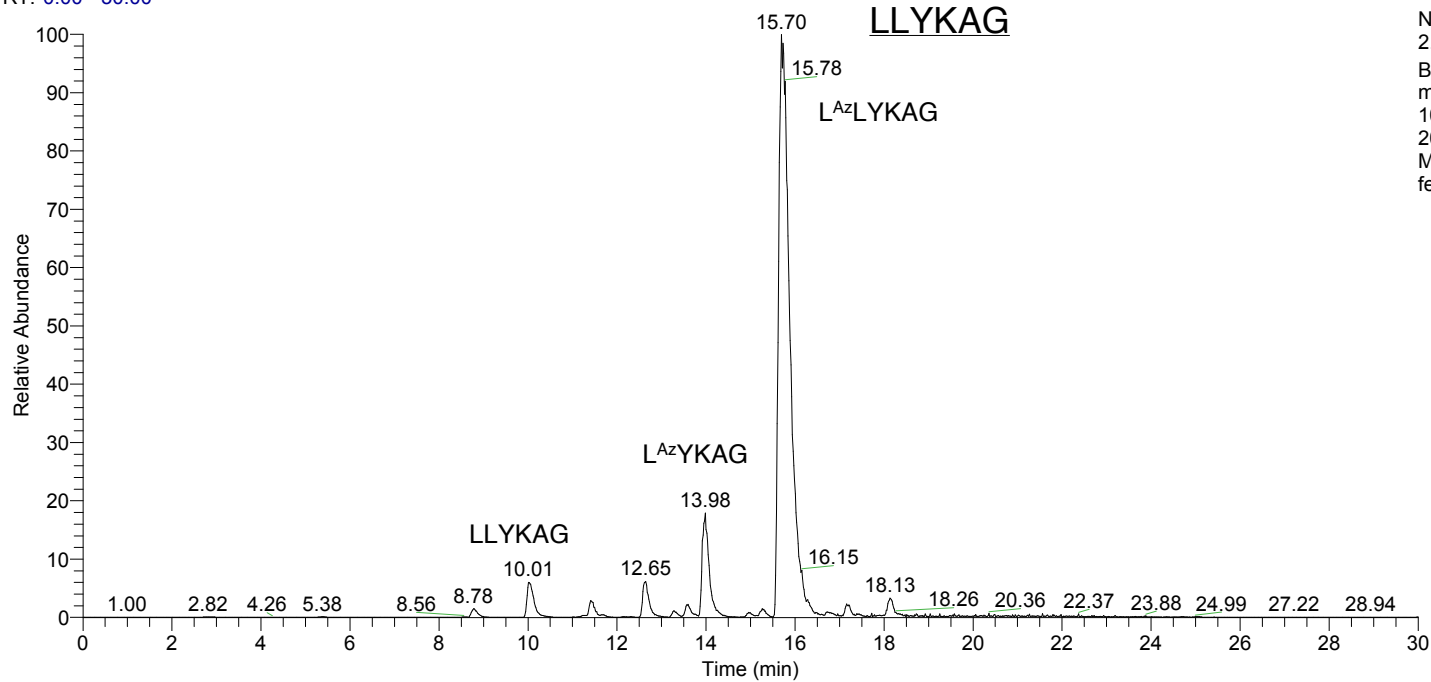
LLYKAG

==== Shimadzu LCsolution Analysis Report ====

D:\My docs\Morten_2\HPLC\2012\jan12\250112\250112-49.lcd
 Acquired by : user
 Sample Name : C-30
 Sample ID : Leu - diazotized
 Tray# : 1
 Vial # : 42
 Injection Volume : 10 uL
 Data File Name : 250112-49.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 250112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 27-Jan-12 15:31:42
 Data Processed : 05-Feb-12 18:15:53

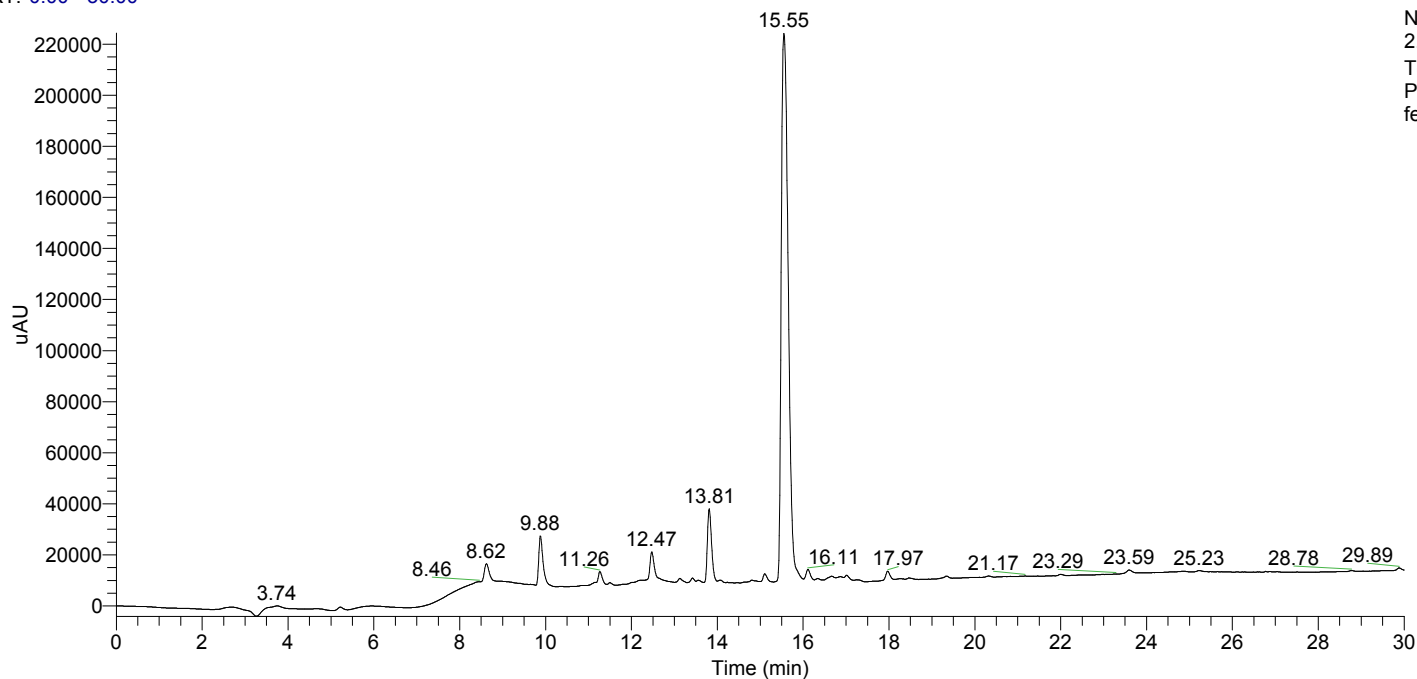


RT: 0.00 - 30.00



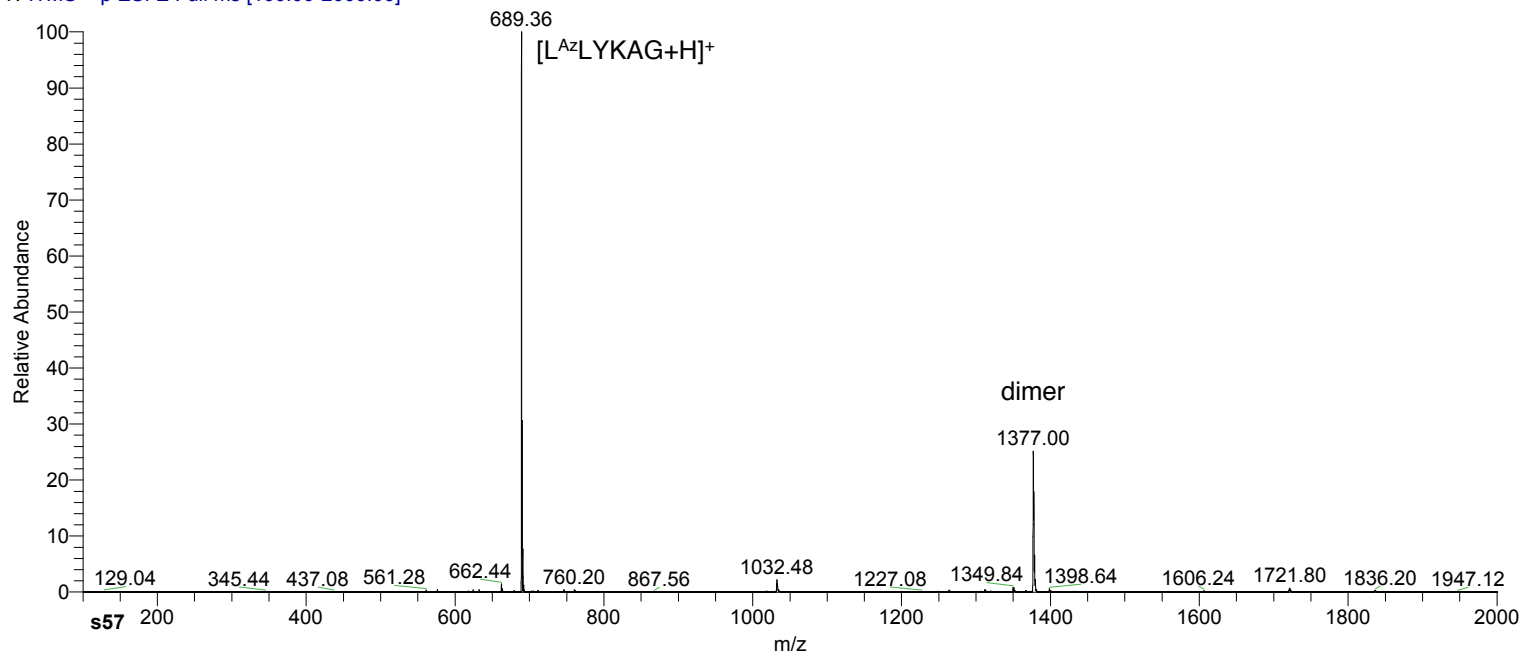
NL:
2.17E5
Base Peak
m/z=
100.00-
2000.00
MS
feb12-31

RT: 0.00 - 30.00



NL:
2.24E5
Total Scan
PDA
feb12-31

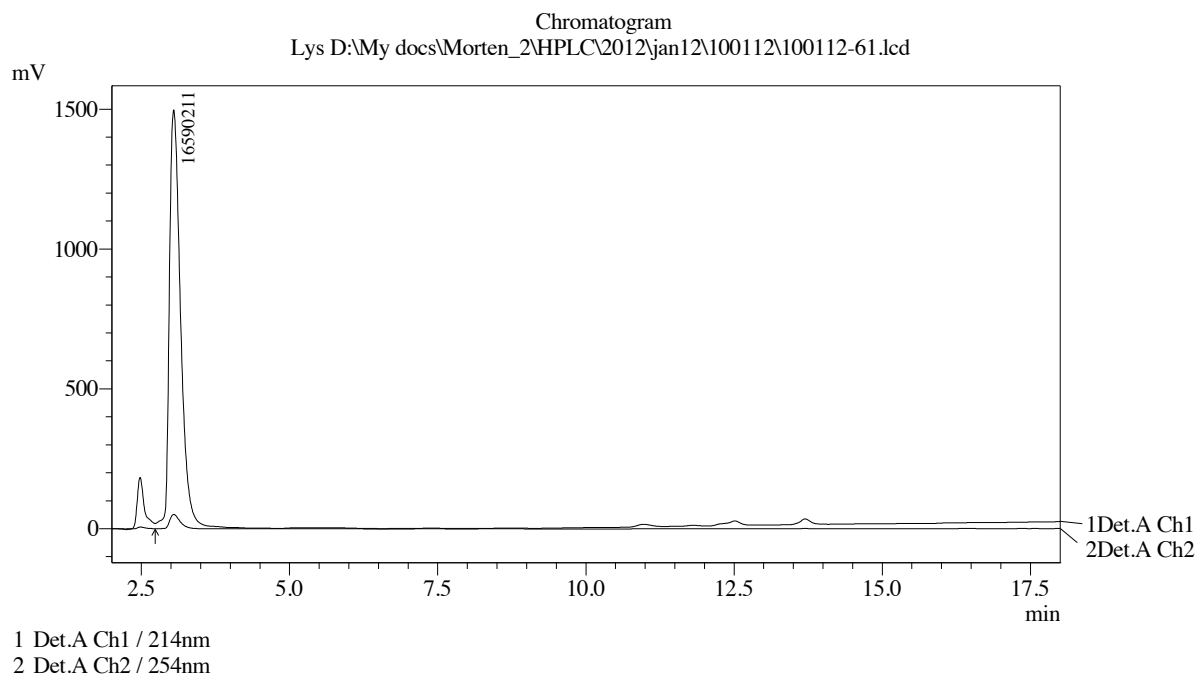
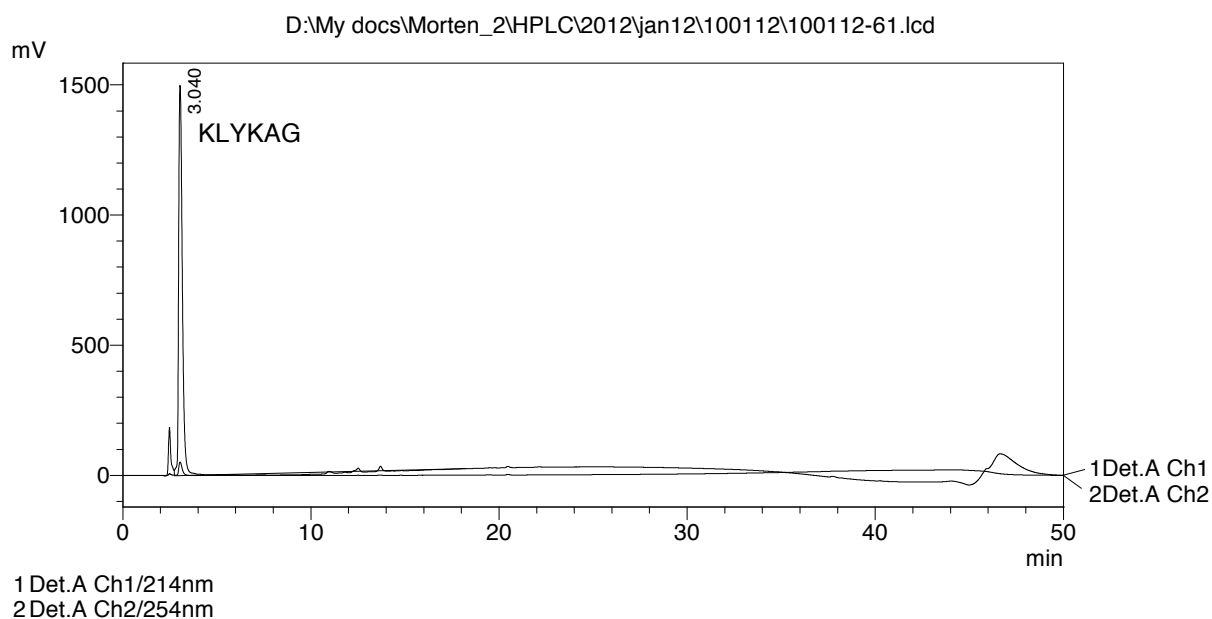
feb12-31 #848-884 RT: 15.55-16.14 AV: 37 NL: 1.01E5
T: ITMS + p ESI E Full ms [100.00-2000.00]



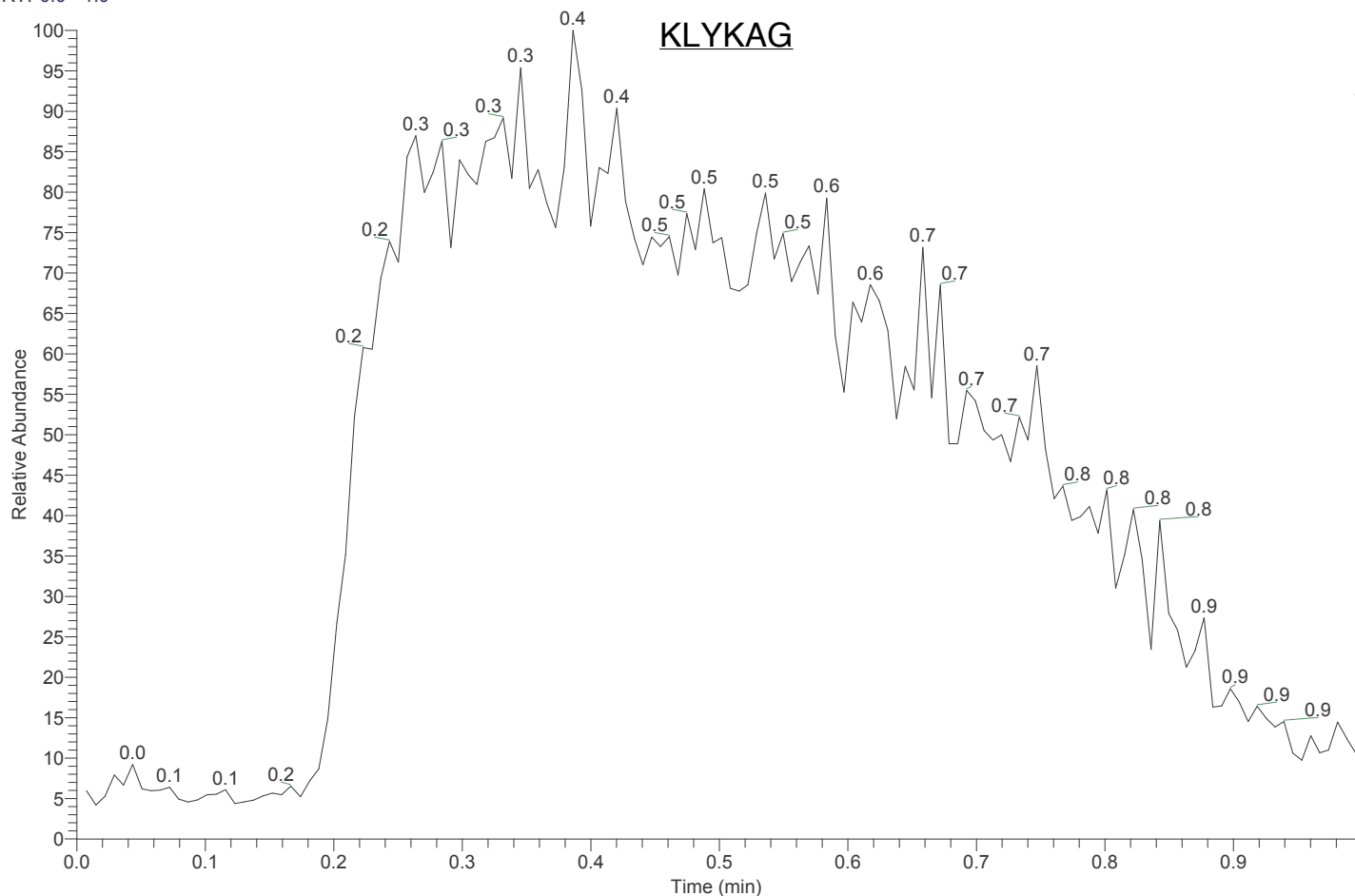
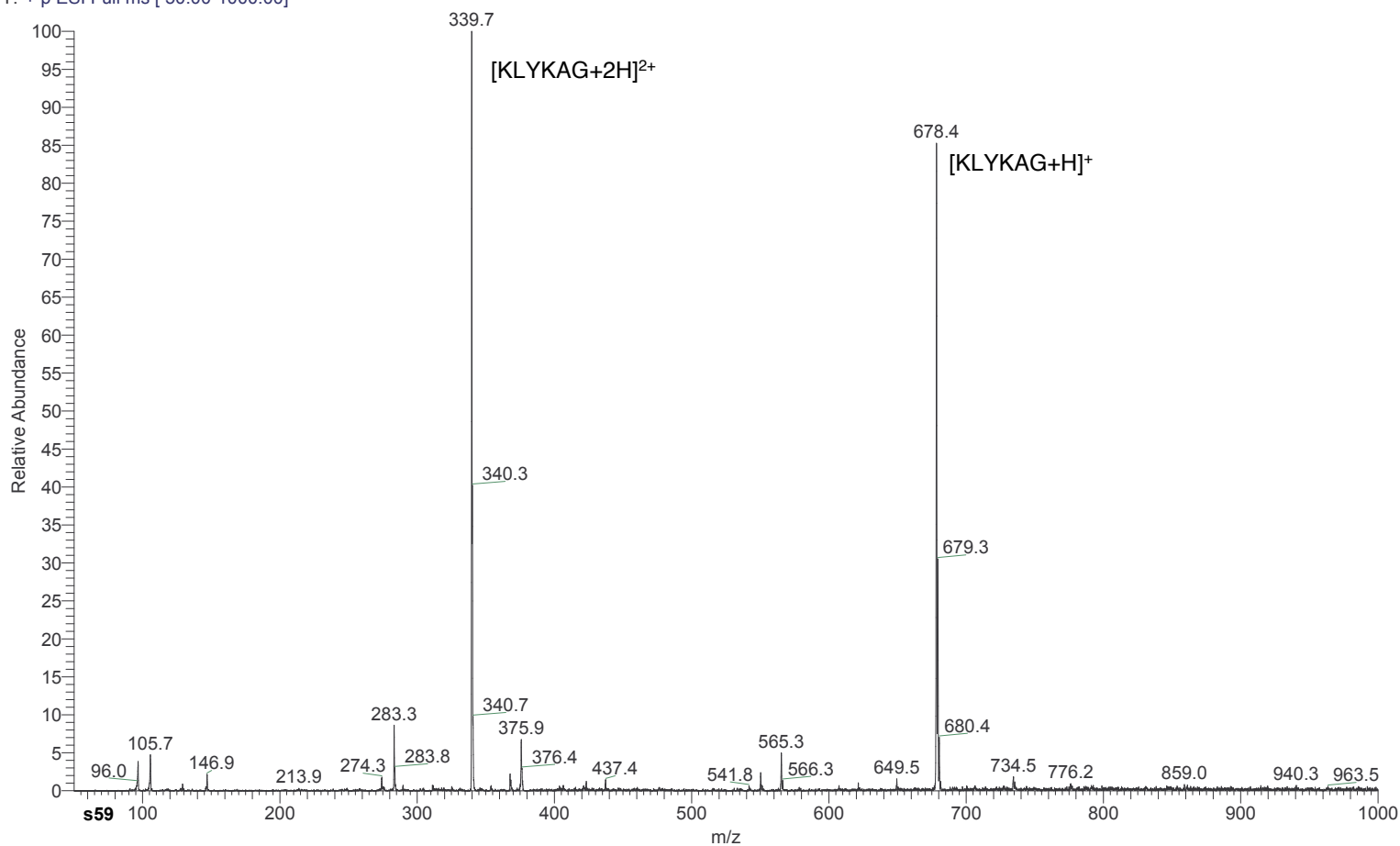
KLYKAG

==== Shimadzu LCsolution Analysis Report ====

D:\My docs\Morten_2\HPLC\2012\jan12\100112\100112-61.lcd
 Acquired by : user
 Sample Name : Lys
 Sample ID : bef. diazotization
 Tray# : 1
 Vial # : 79
 Injection Volume : 10 uL
 Data File Name : 100112-61.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 100112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 13-Jan-12 0:51:19
 Data Processed : 13-Jan-12 1:41:23



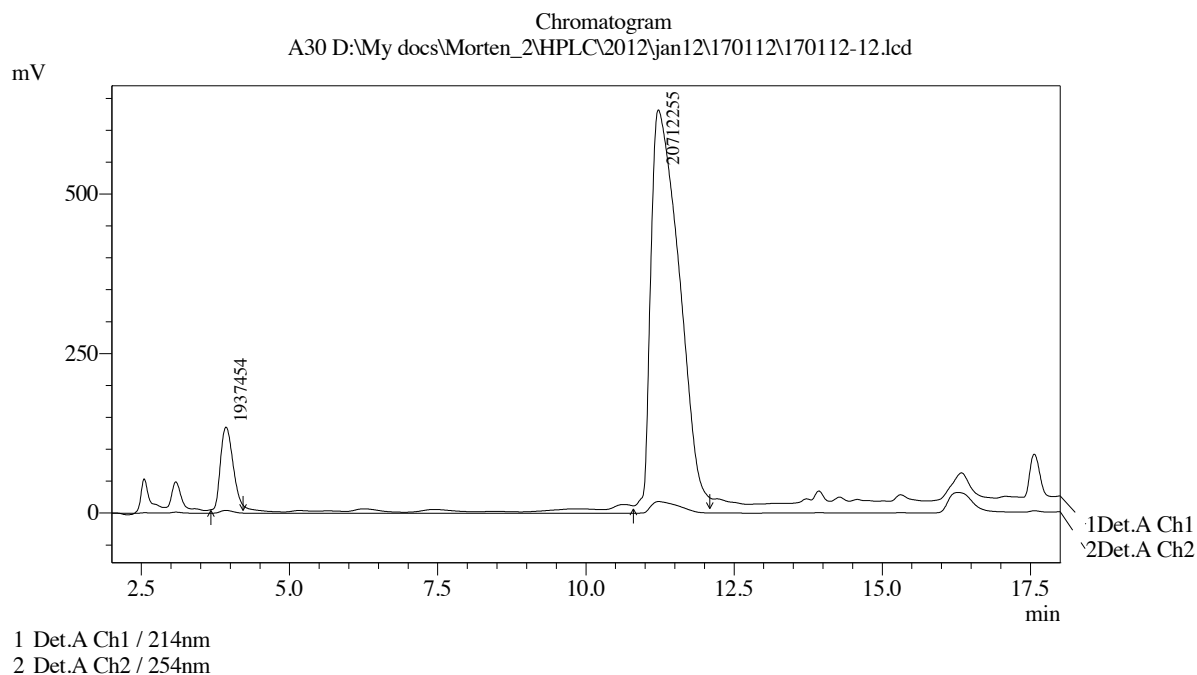
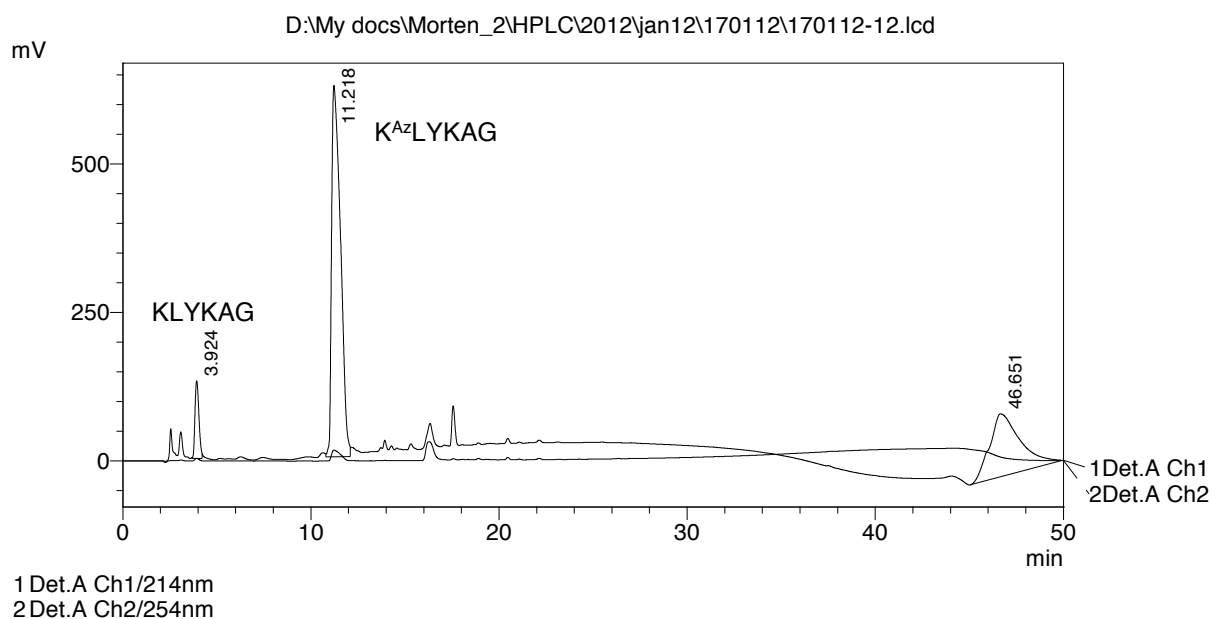
RT: 0.0 - 1.0

NL:
7.66E8
TIC F: MS
jan12-25jan12-25 #27-79 RT: 0.2-0.5 AV: 53 NL: 1.56E7
T: + p ESI Full ms [50.00-1000.00]

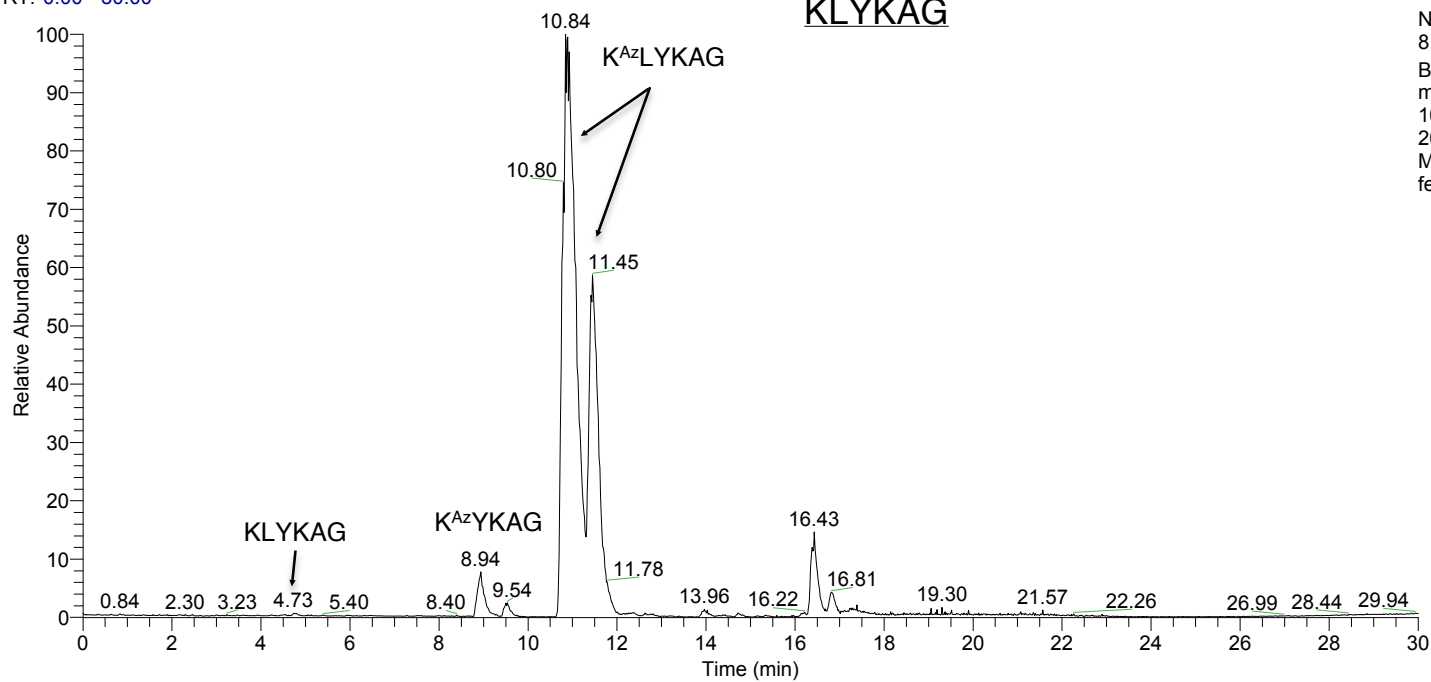
KLYKAG

==== Shimadzu LCsolution Analysis Report ====

D:\My docs\Morten_2\HPLC\2012\jan12\170112\170112-12.lcd
 Acquired by : user
 Sample Name : A30
 Sample ID : Lys - diazotized
 Tray# : 1
 Vial # : 14
 Injection Volume : 10 uL
 Data File Name : 170112-12.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 170112.lcb
 Report File Name : Default.lcr
 Data Acquired : 18-Jan-12 0:59:05
 Data Processed : 18-Jan-12 11:35:10

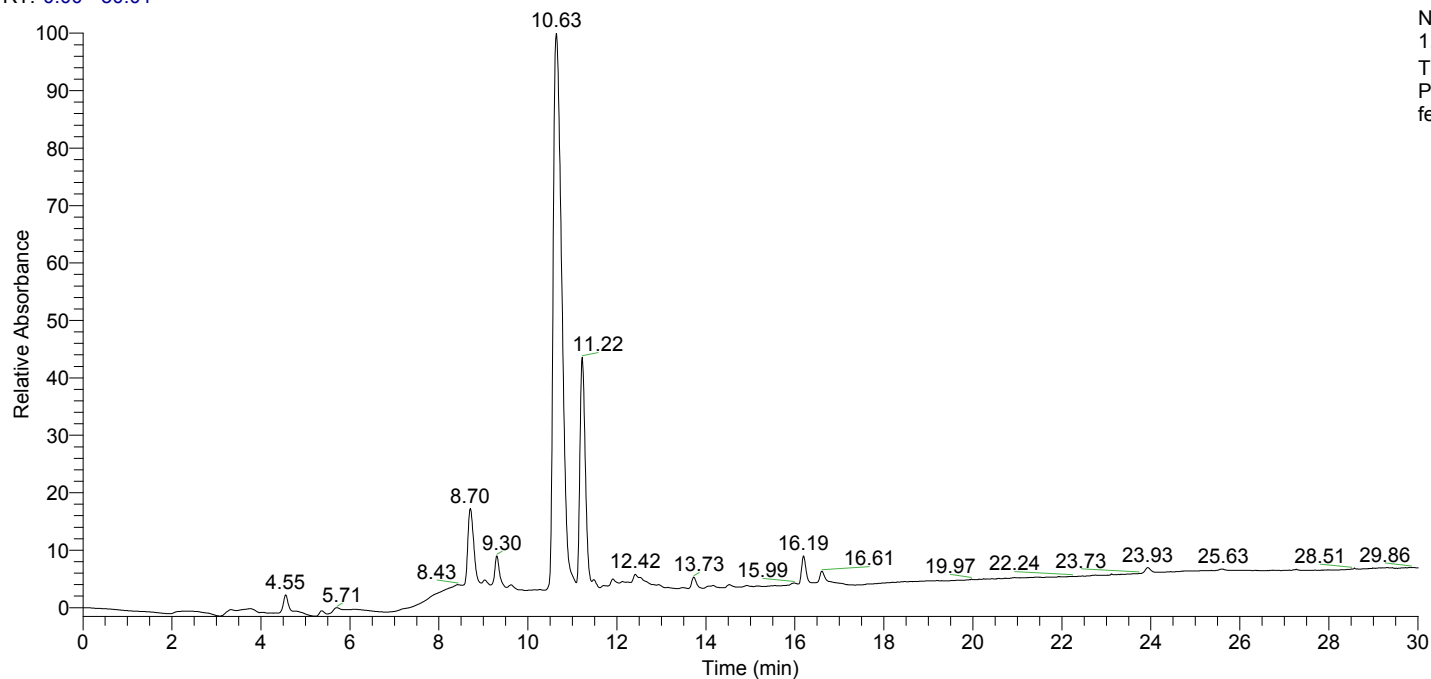


RT: 0.00 - 30.00



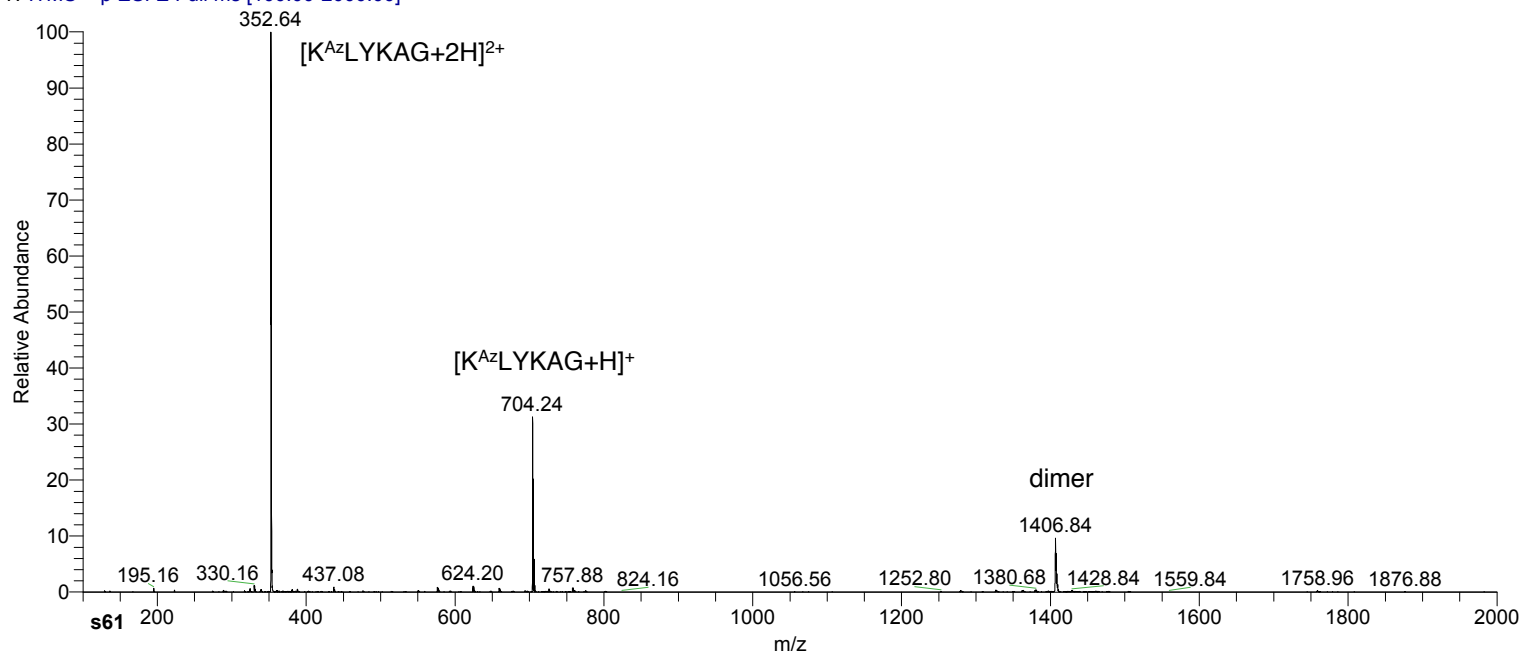
NL:
8.78E4
Base Peak
m/z=
100.00-
2000.00
MS
feb12-25

RT: 0.00 - 30.01



NL:
1.93E5
Total Scan
PDA
feb12-25

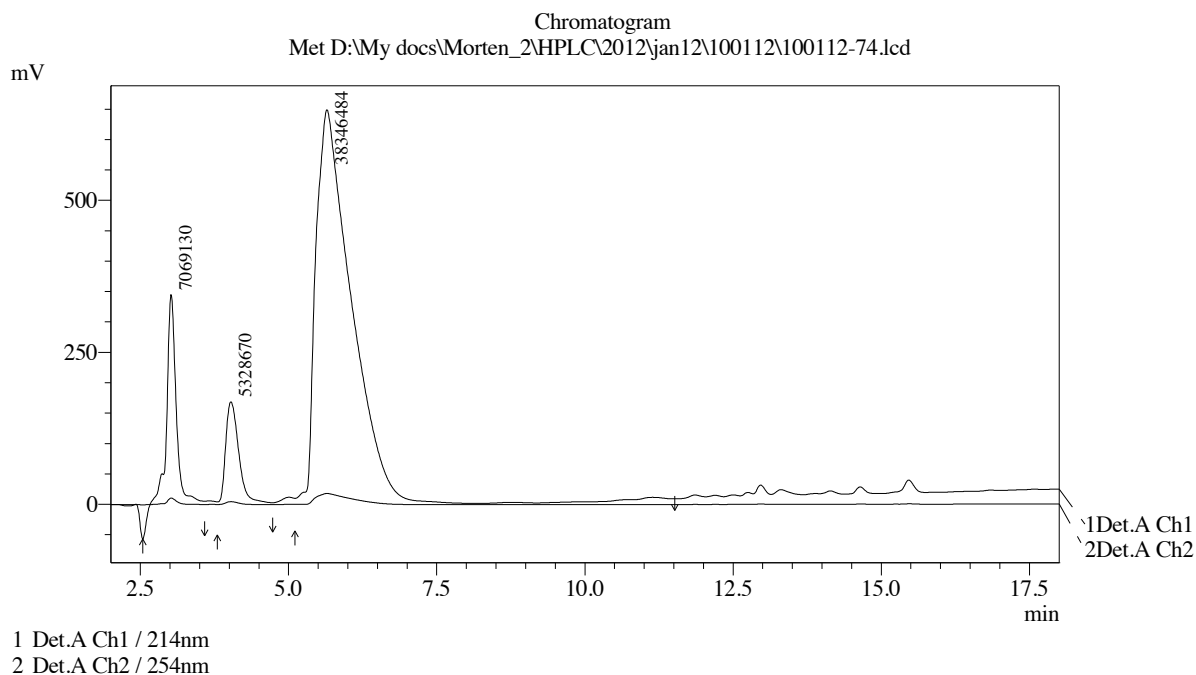
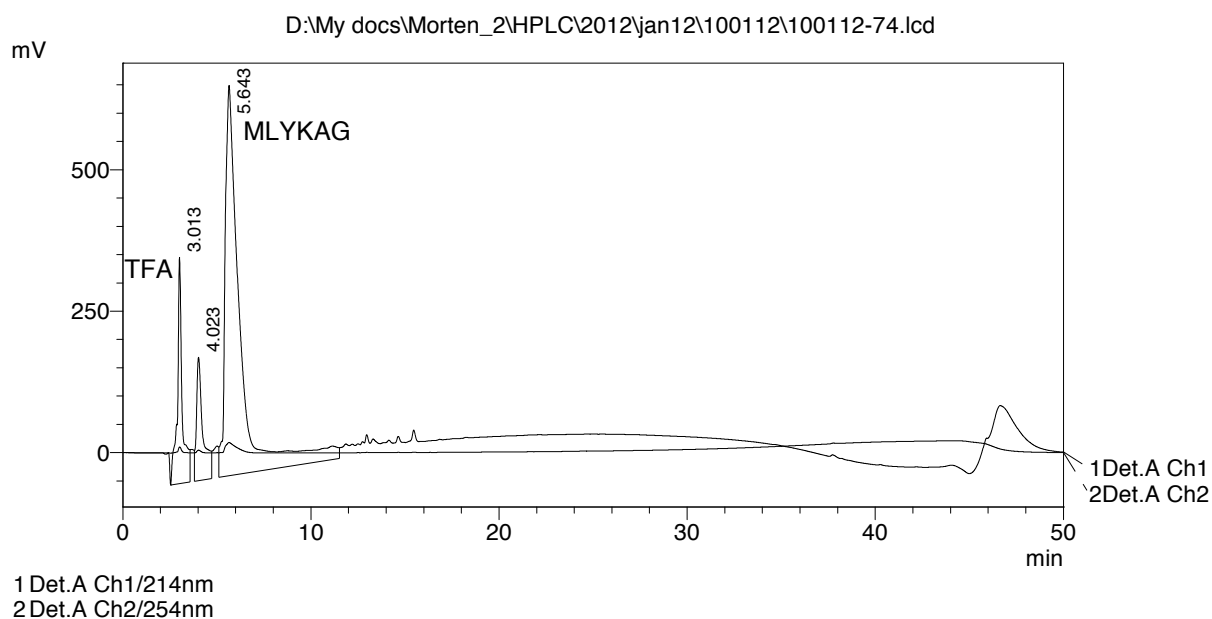
feb12-25 #568-646 RT: 10.63-11.91 AV: 79 NL: 2.33E4
T: ITMS + p ESI E Full ms [100.00-2000.00]



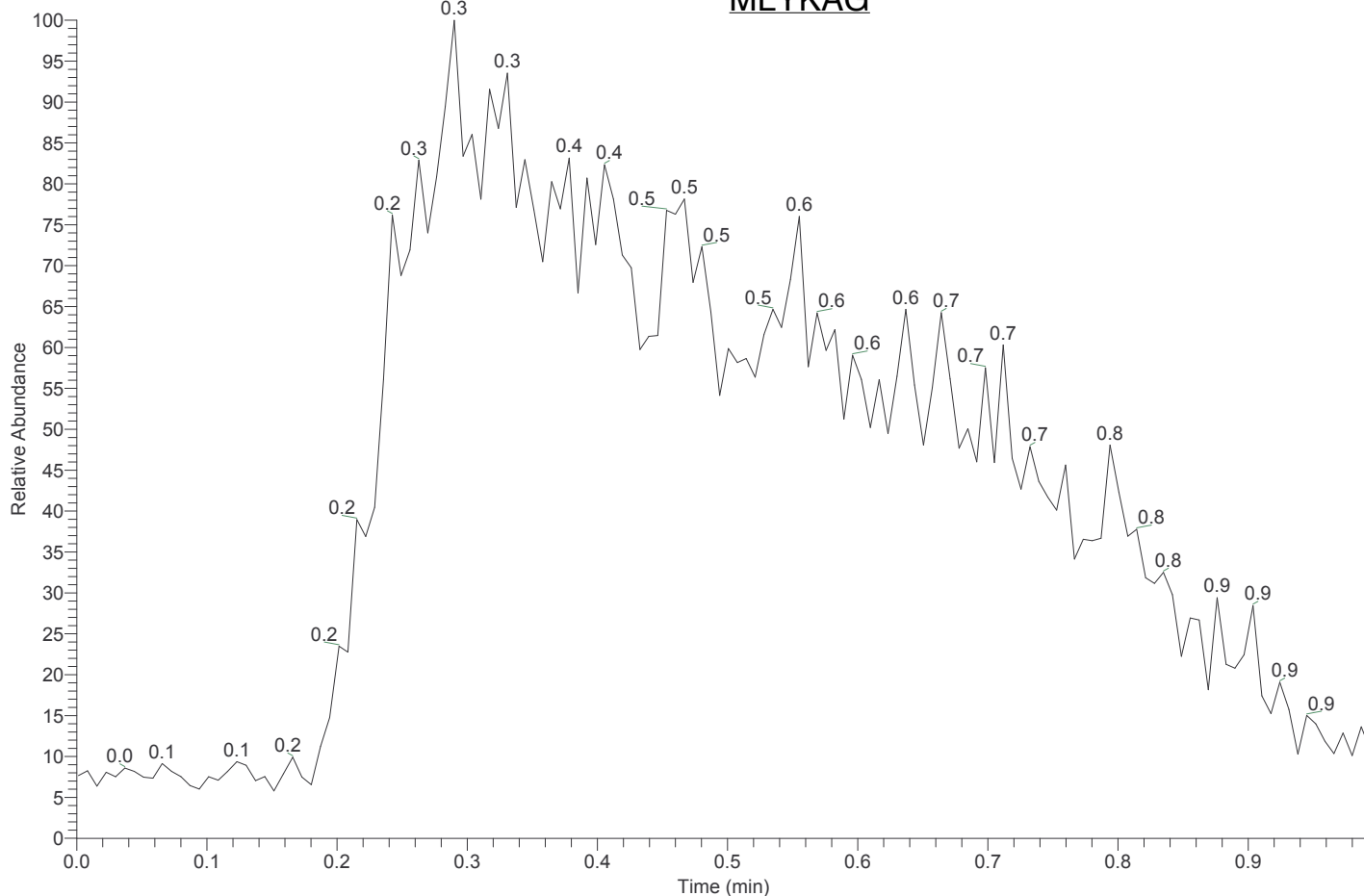
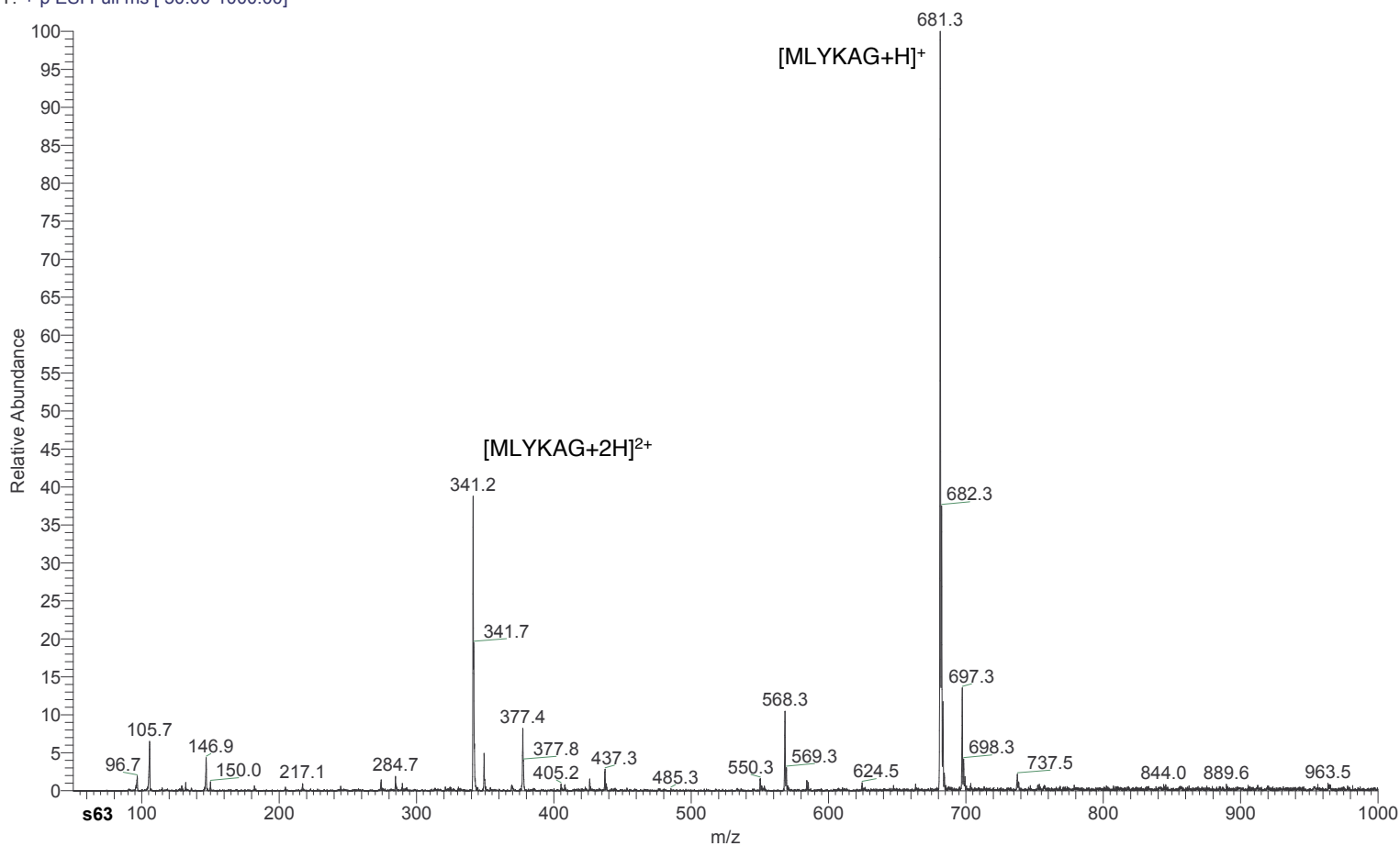
MLYKAG

==== Shimadzu LCsolution Analysis Report ====

D:\My docs\Morten_2\HPLC\2012\jan12\100112\100112-74.lcd
 Acquired by : user
 Sample Name : Met
 Sample ID : bef. diazotization
 Tray# : 1
 Vail # : 92
 Injection Volume : 10 uL
 Data File Name : 100112-74.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 100112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 13-Jan-12 11:47:13
 Data Processed : 13-Jan-12 12:37:18



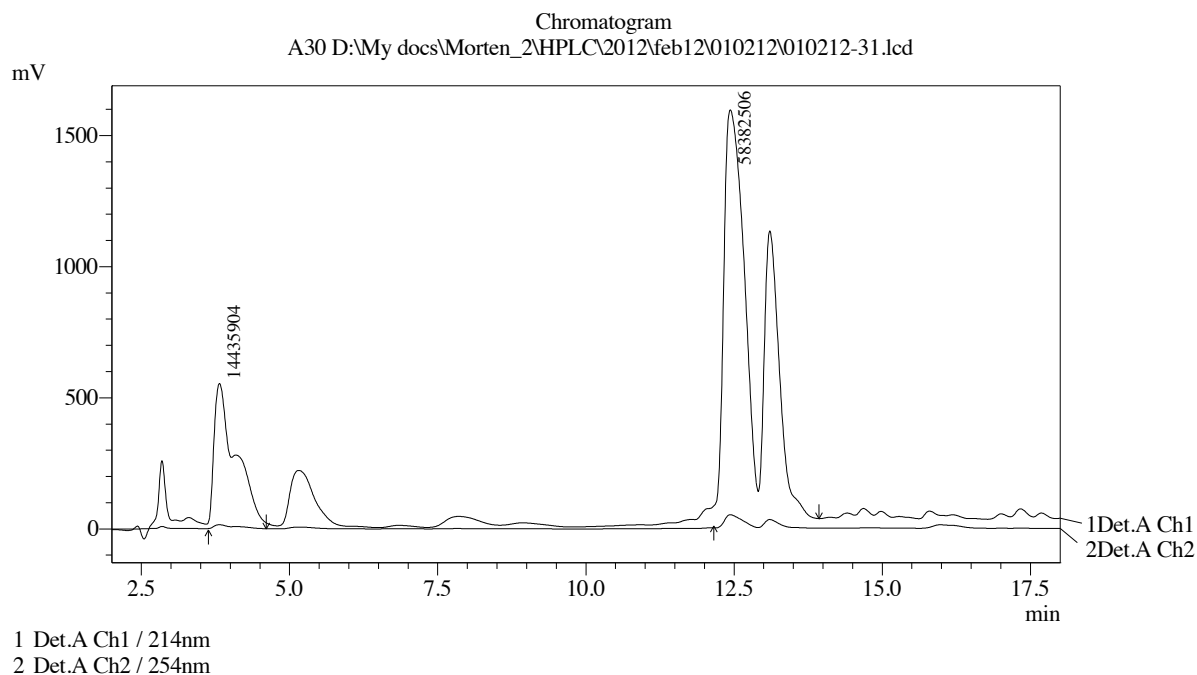
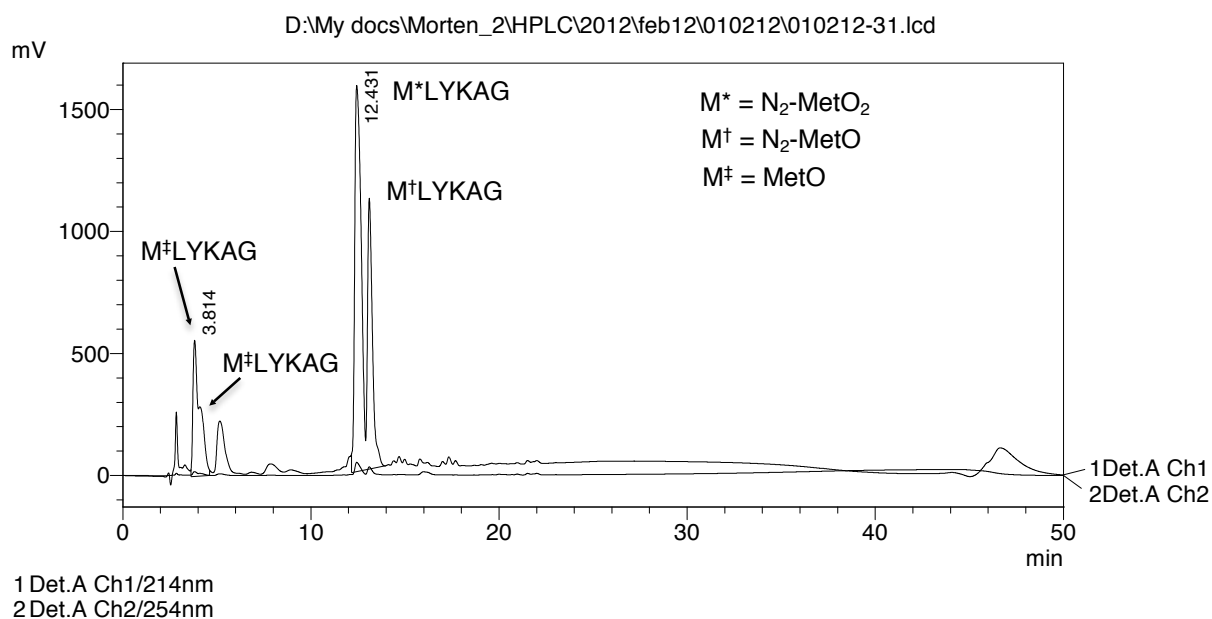
RT: 0.0 - 1.0

MLYKAGNL:
6.99E8
TIC F: MS
jan12-17jan12-17 #27-77 RT: 0.2-0.5 AV: 51 NL: 1.26E7
T: + p ESI Full ms [50.00-1000.00]

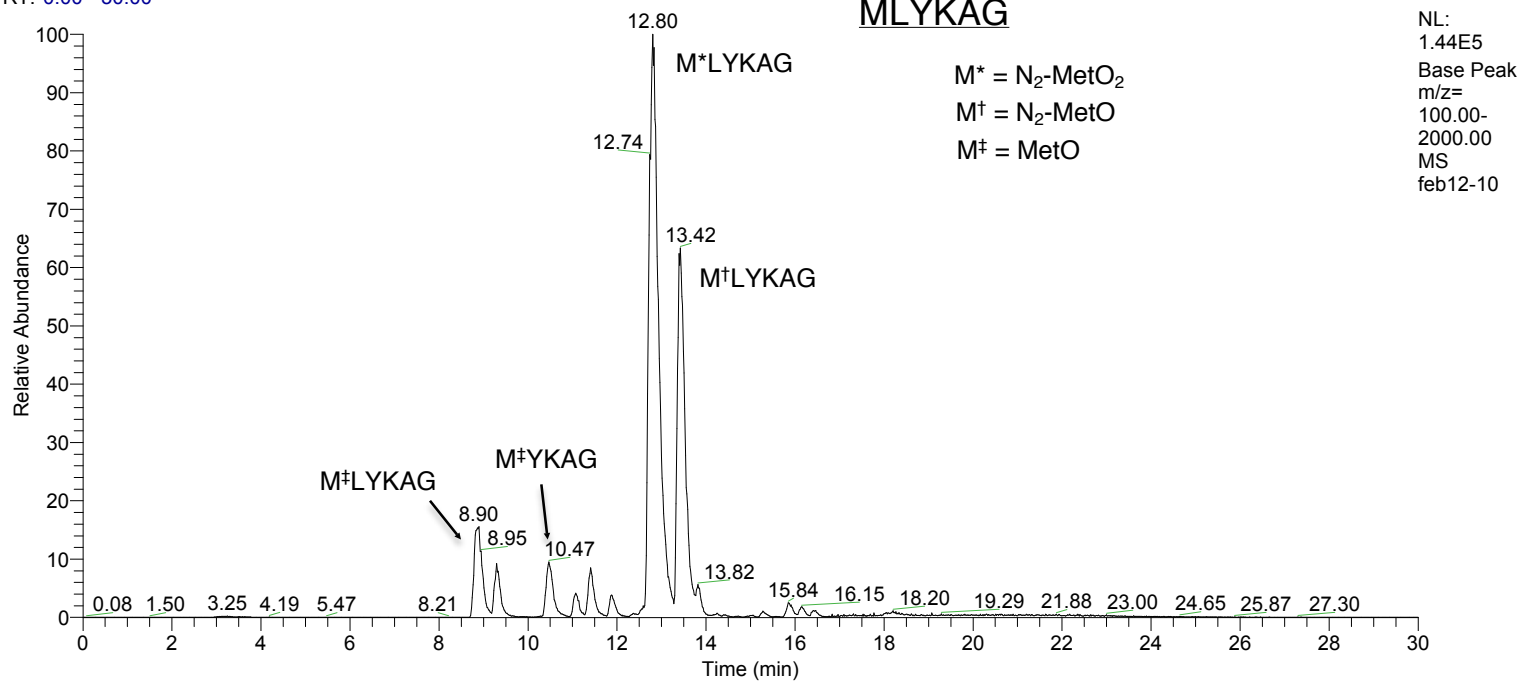
MLYKAG

==== Shimadzu LCsolution Analysis Report ====

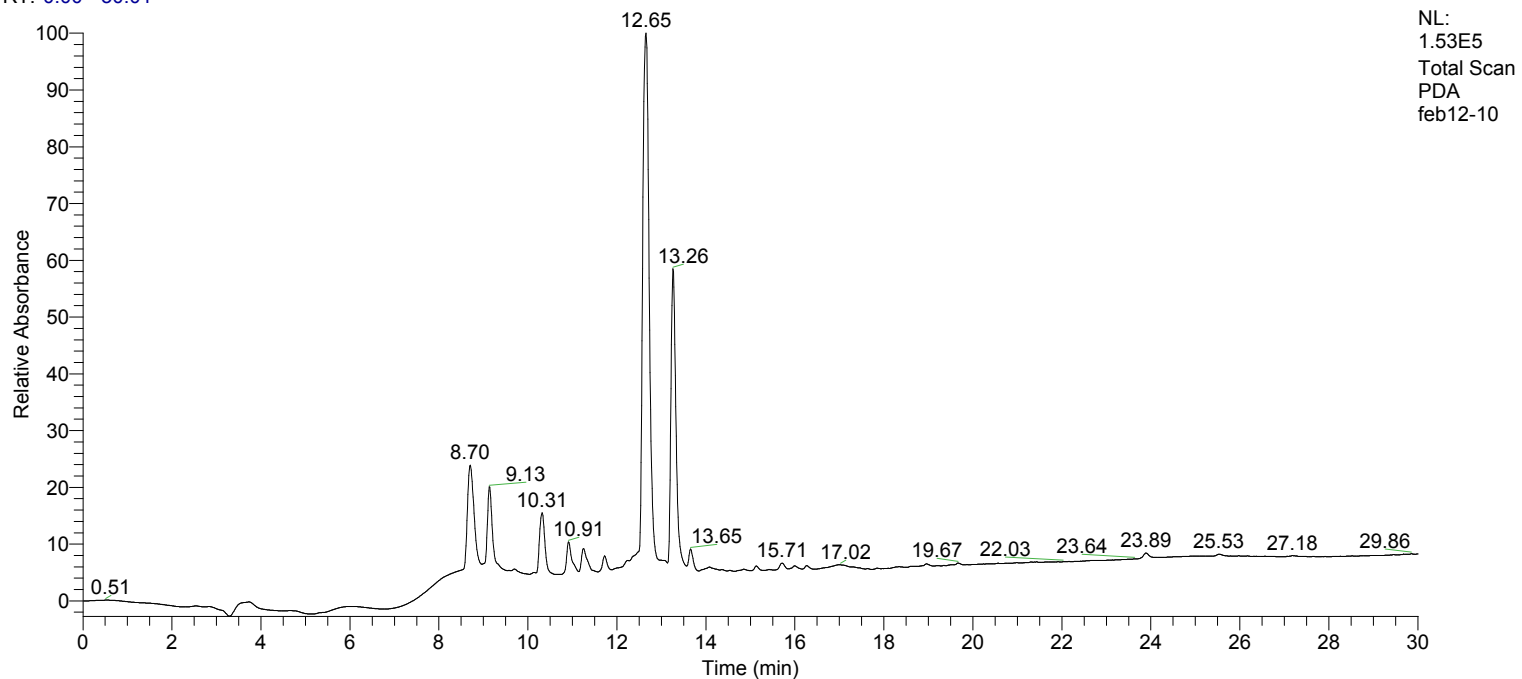
D:\My docs\Morten_2\HPLC\2012\feb12\010212\010212-31.lcd
 Acquired by : guest user
 Sample Name : A30
 Sample ID : Met - diazotized
 Tray# : 1
 Vial # : 31
 Injection Volume : 10 uL
 Data File Name : 010212-31.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 010212.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 02-Feb-12 20:44:53
 Data Processed : 06-Feb-12 9:10:57



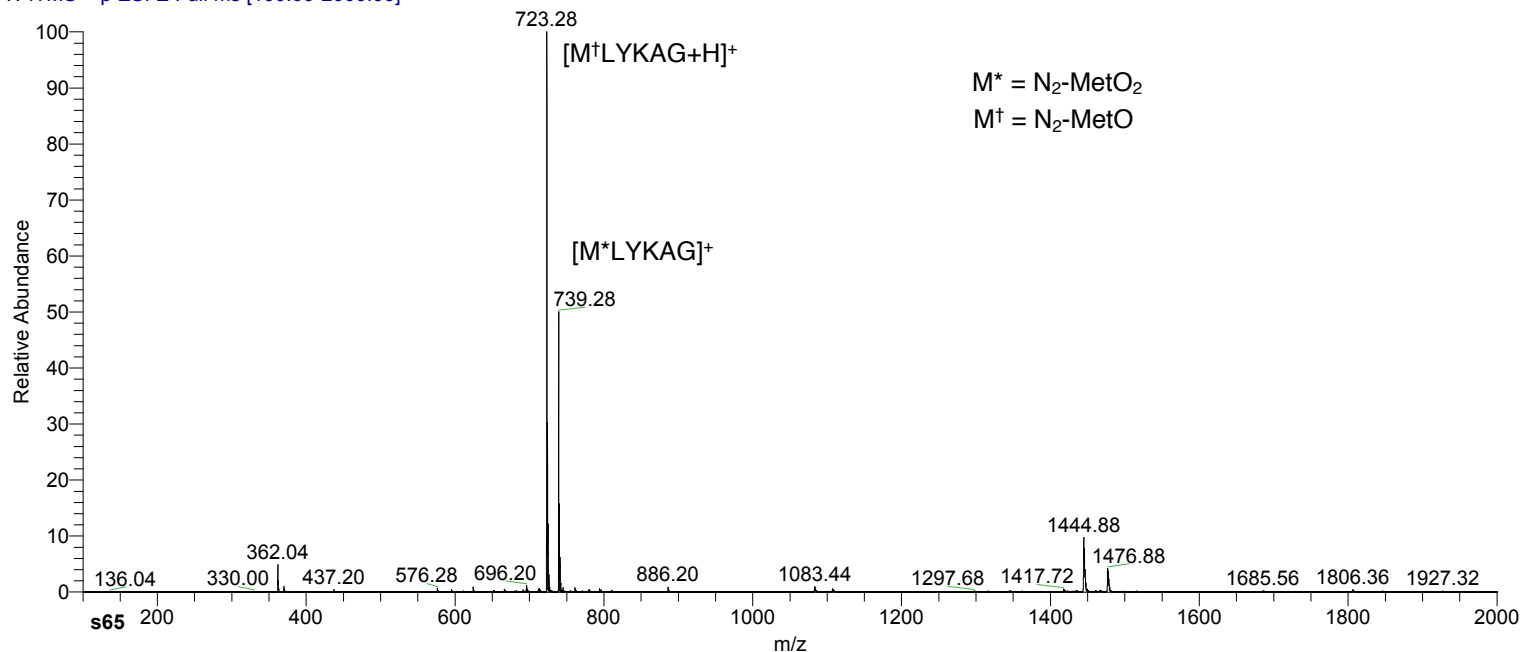
RT: 0.00 - 30.00



RT: 0.00 - 30.01



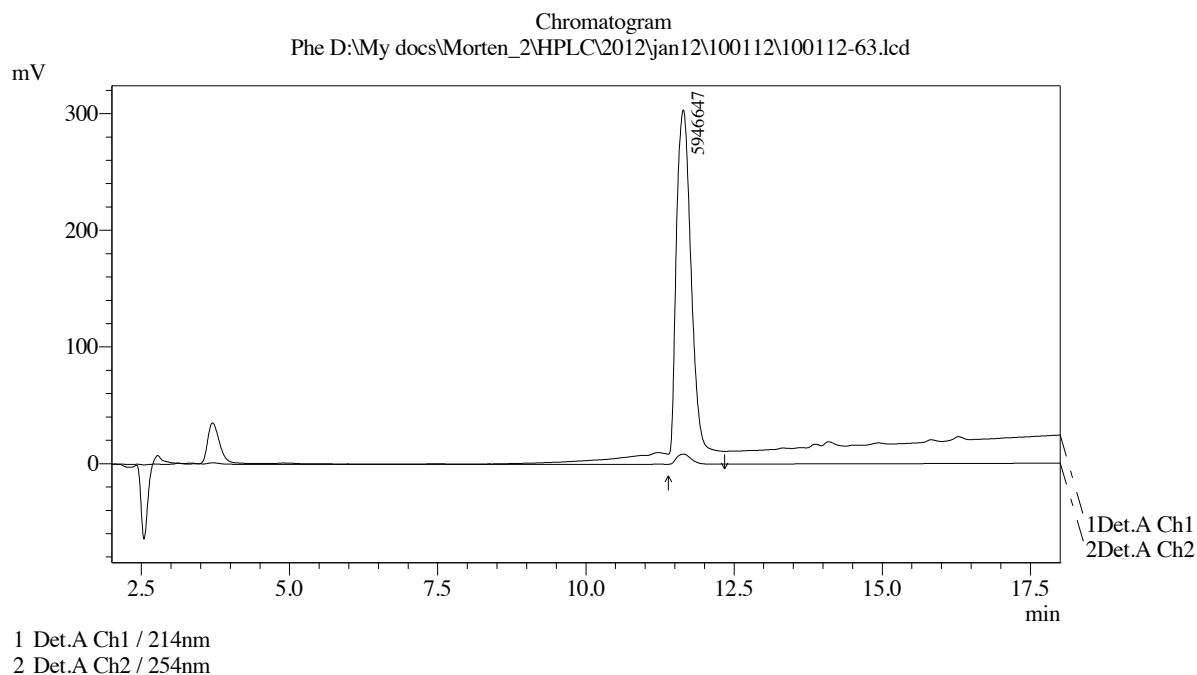
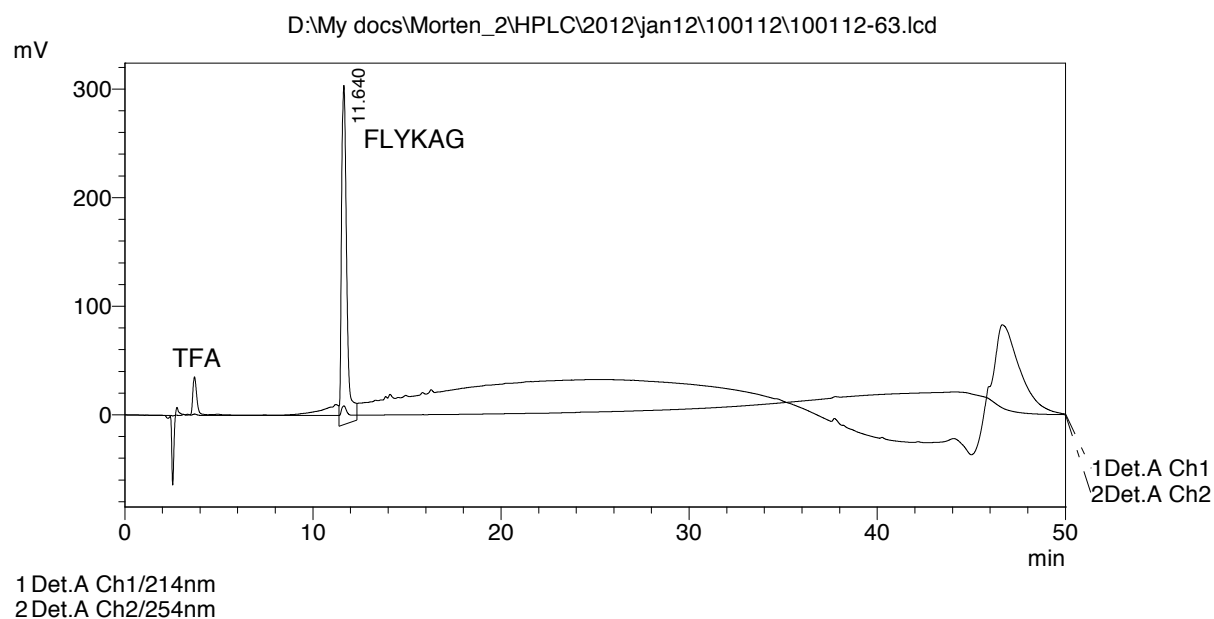
feb12-10 #686-752 RT: 12.62-13.70 AV: 67 NL: 3.37E4
T: ITMS + p ESI E Full ms [100.00-2000.00]



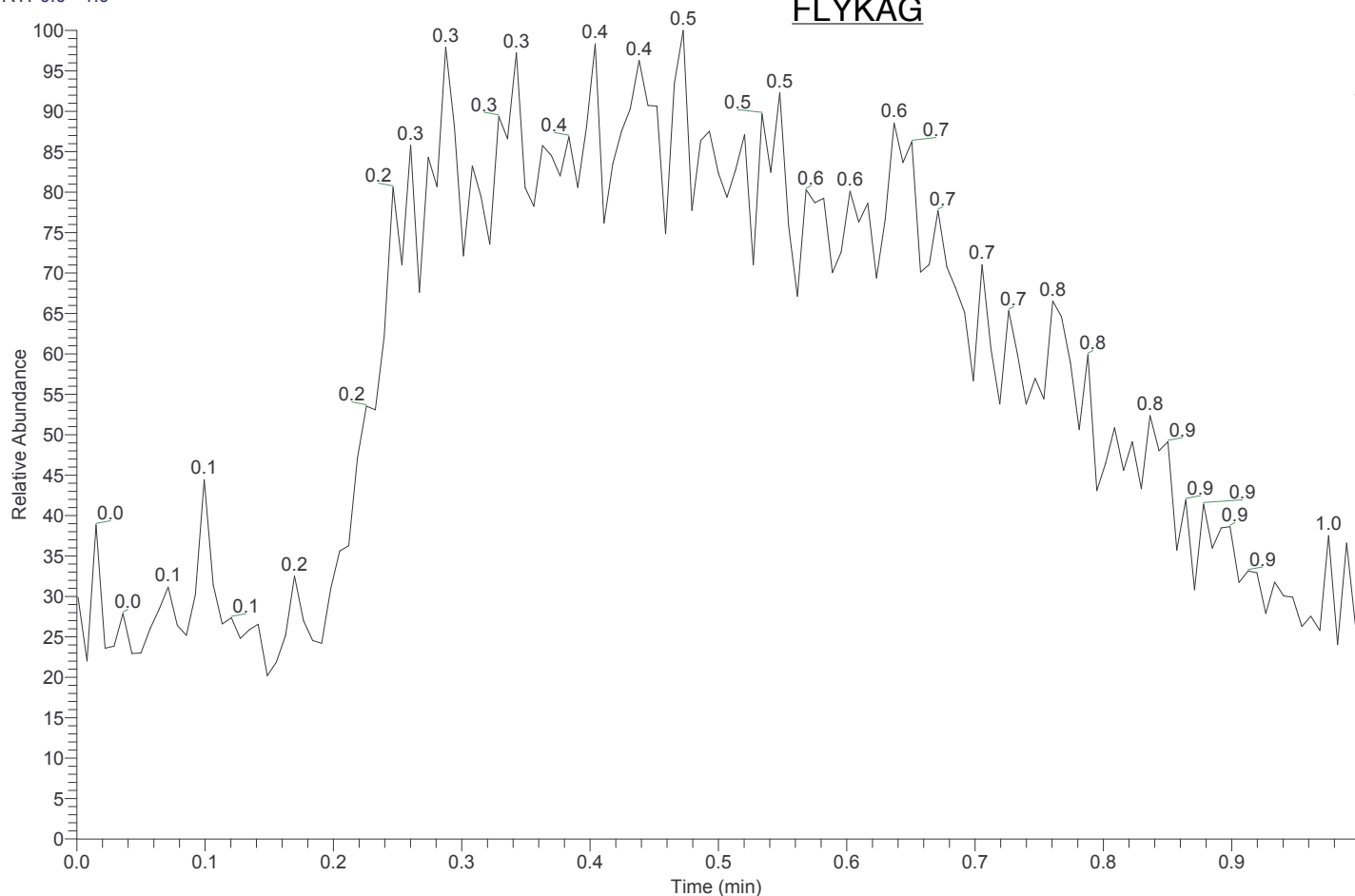
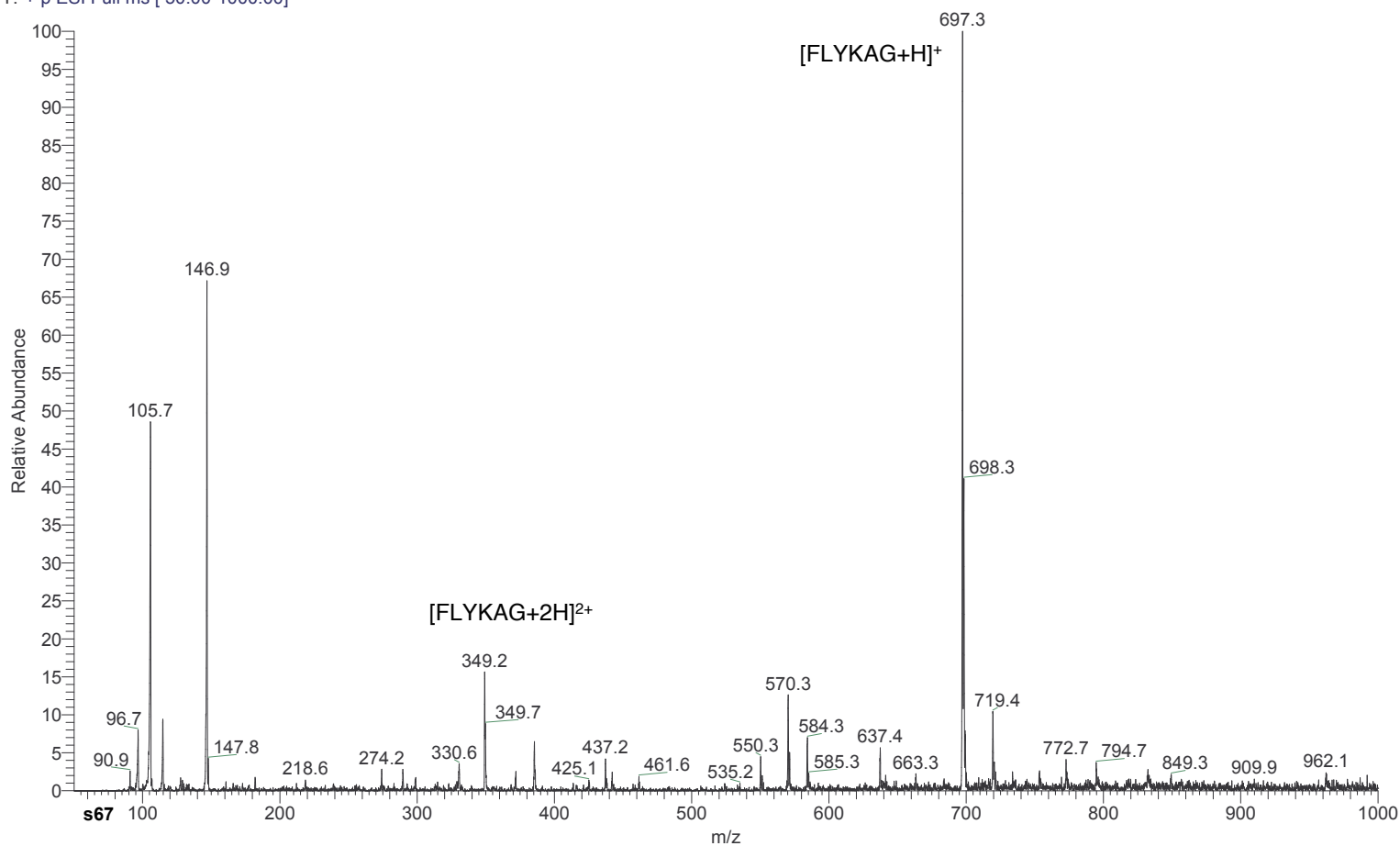
FLYKAG

==== Shimadzu LCsolution Analysis Report ====

Acquired by : user
Sample Name : Phe
Sample ID : bef. diazotization
Tray# : 1
Vial # : 81
Injection Volume : 10 uL
Data File Name : 100112-63.lcd
Method File Name : gradient-anal.lcm
Batch File Name : 100112.lcb
Report File Name : ALYKAG-report.lcr
Data Acquired : 13-Jan-12 2:32:10
Data Processed : 13-Jan-12 3:22:15



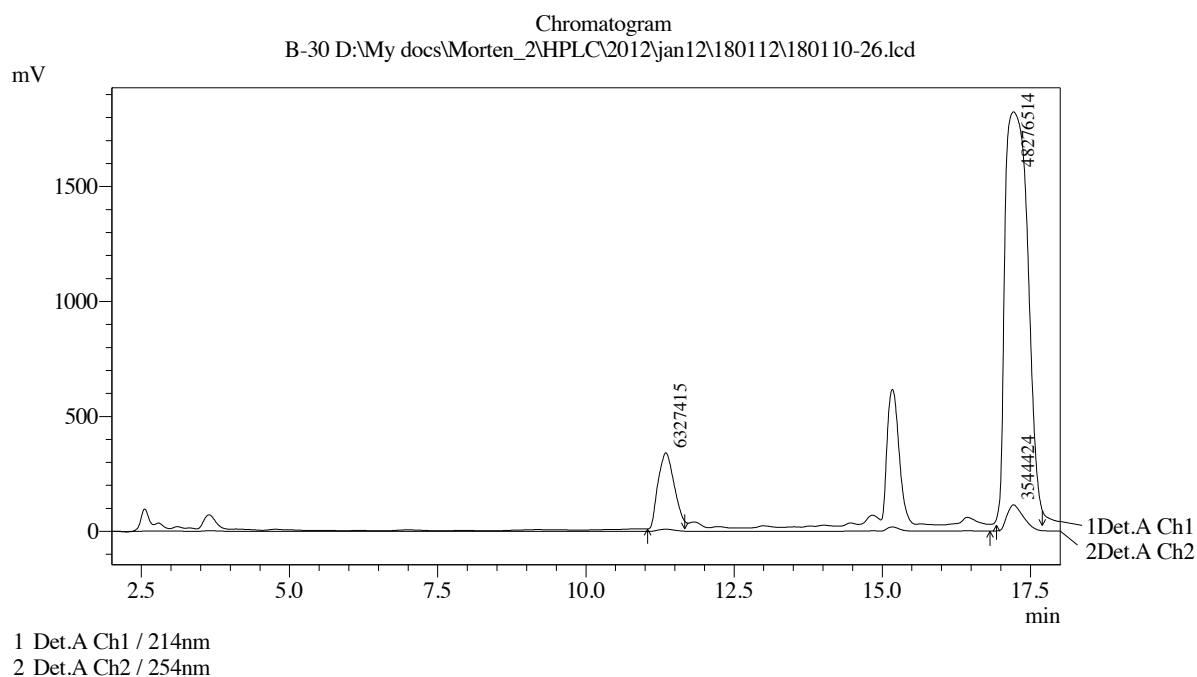
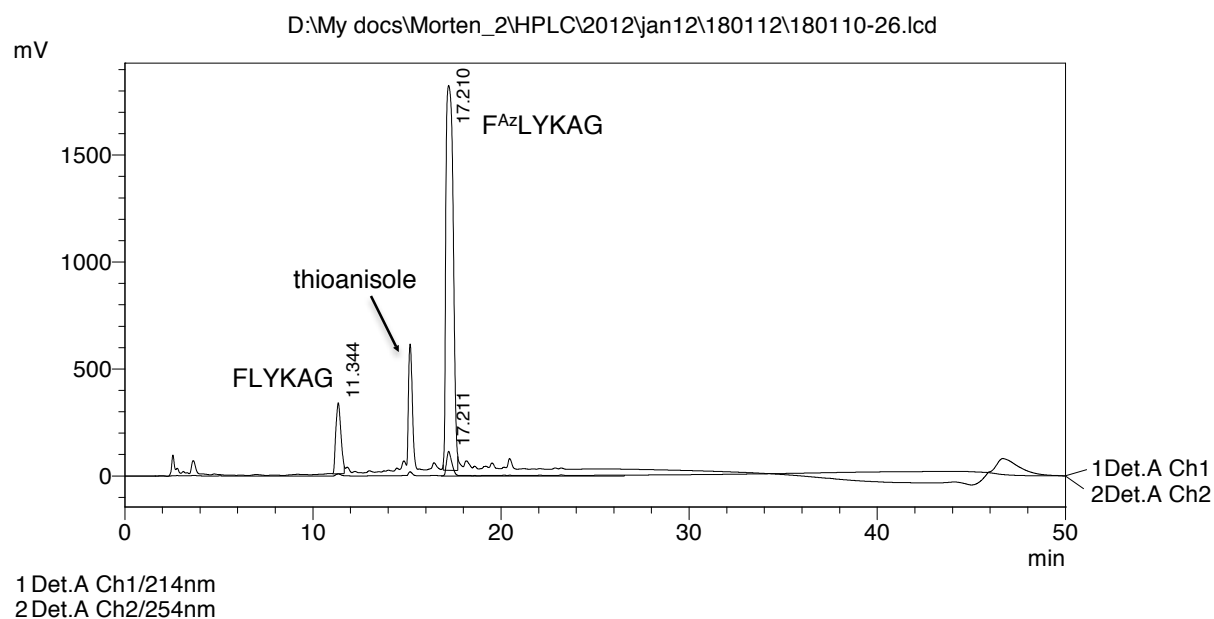
RT: 0.0 - 1.0

NL:
2.94E8
TIC F: MS
jan12-13jan12-13 #29-68 RT: 0.2-0.5 AV: 40 NL: 3.21E6
T: + p ESI Full ms [50.00-1000.00]

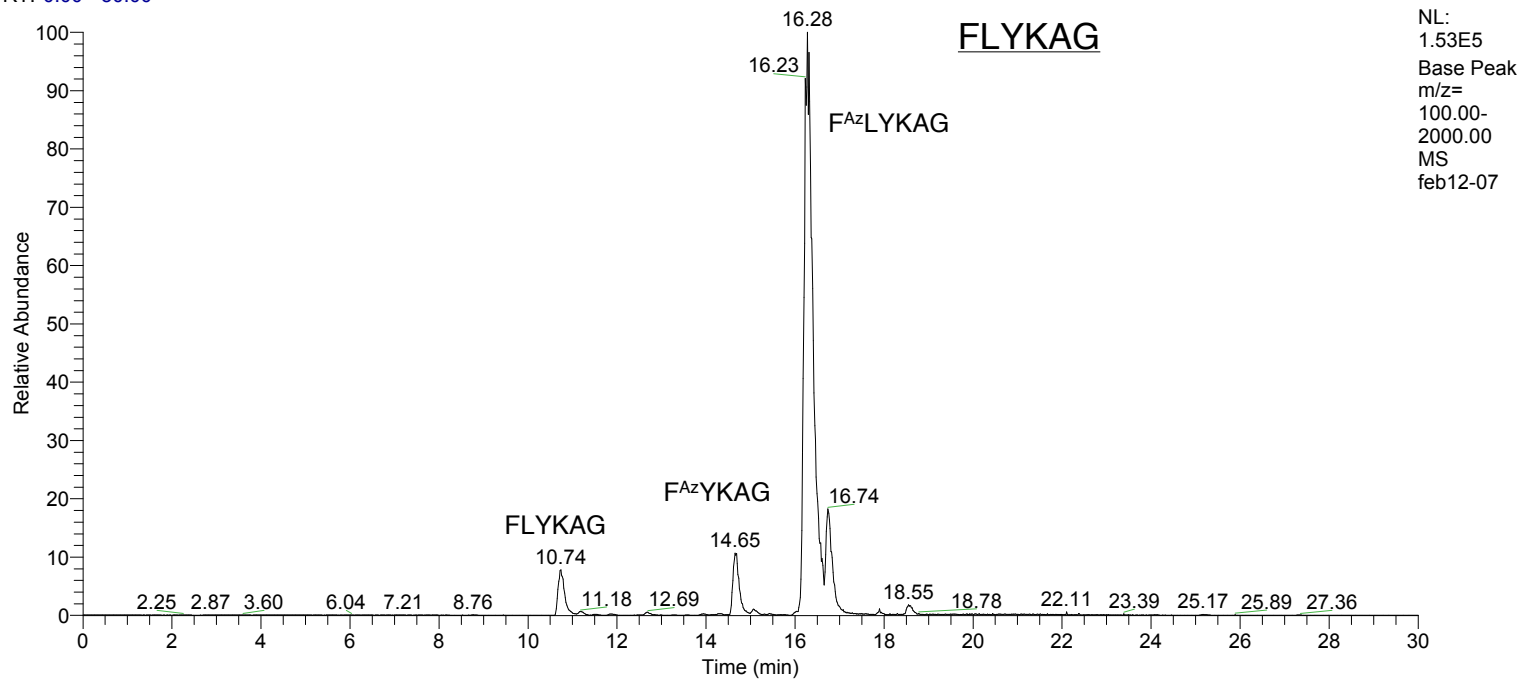
FLYKAG

==== Shimadzu LCsolution Analysis Report ====

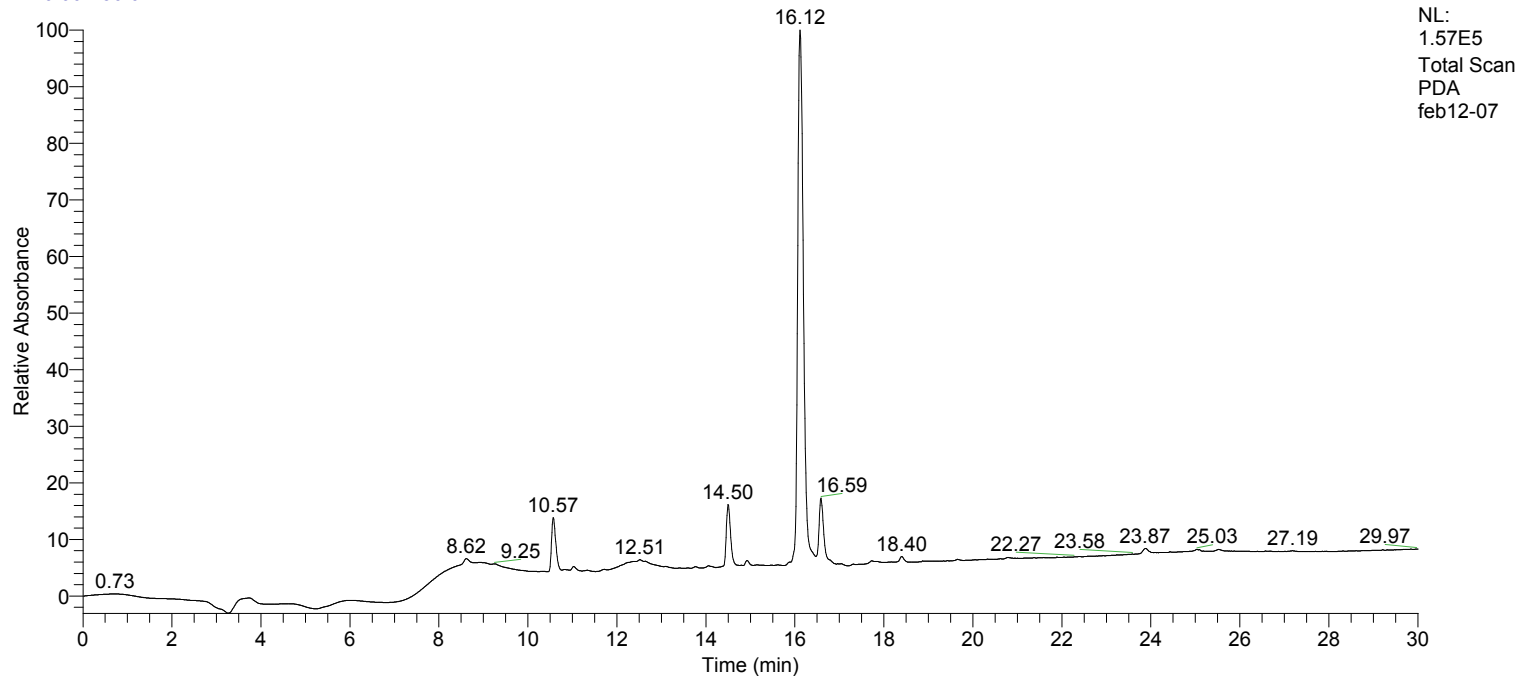
D:\My docs\Morten_2\HPLC\2012\jan12\180112\180110-26.lcd
 Acquired by : user
 Sample Name : B-30
 Sample ID : Phe - diazotized
 Tray# : 1
 Vial # : 41
 Injection Volume : 10 uL
 Data File Name : 180110-26.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : Batch file_2012.lcb
 Report File Name : Default.lcr
 Data Acquired : 25-Jan-12 18:06:55
 Data Processed : 31-Jan-12 21:33:11



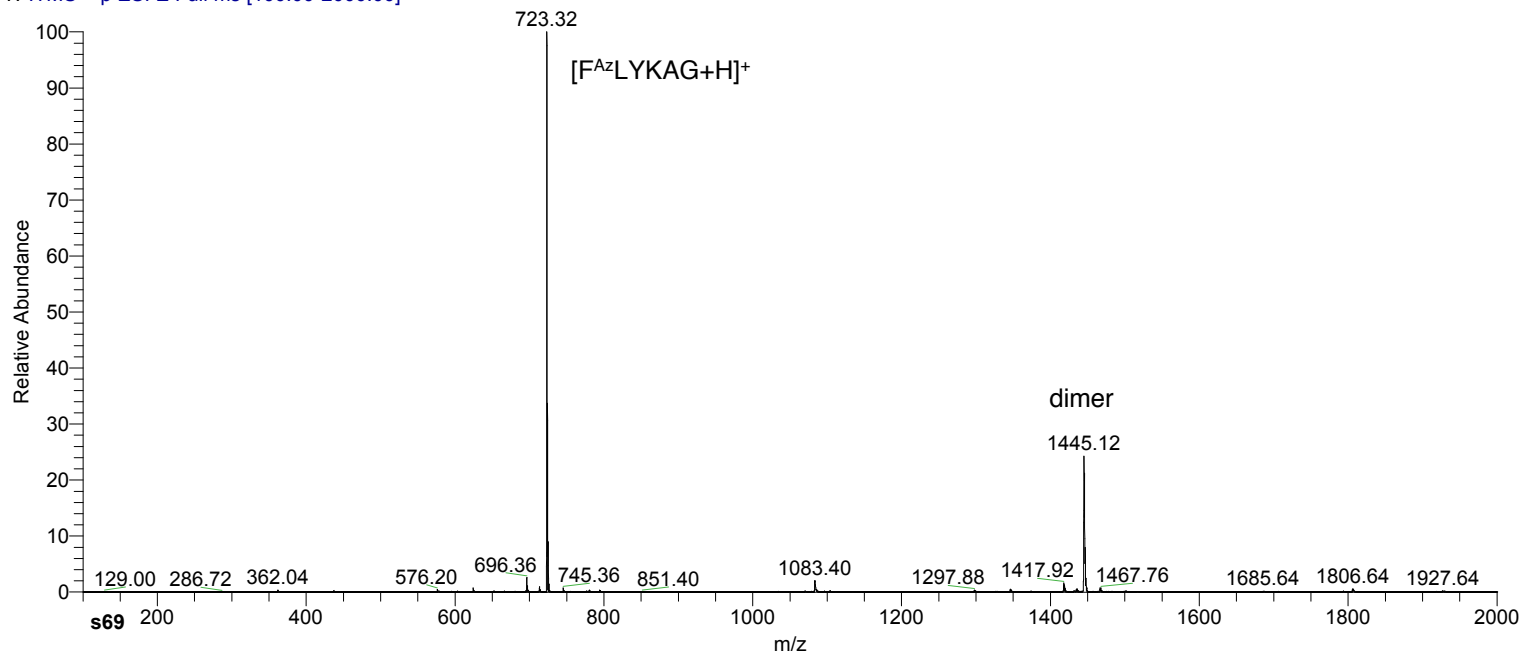
RT: 0.00 - 30.00



RT: 0.00 - 30.01

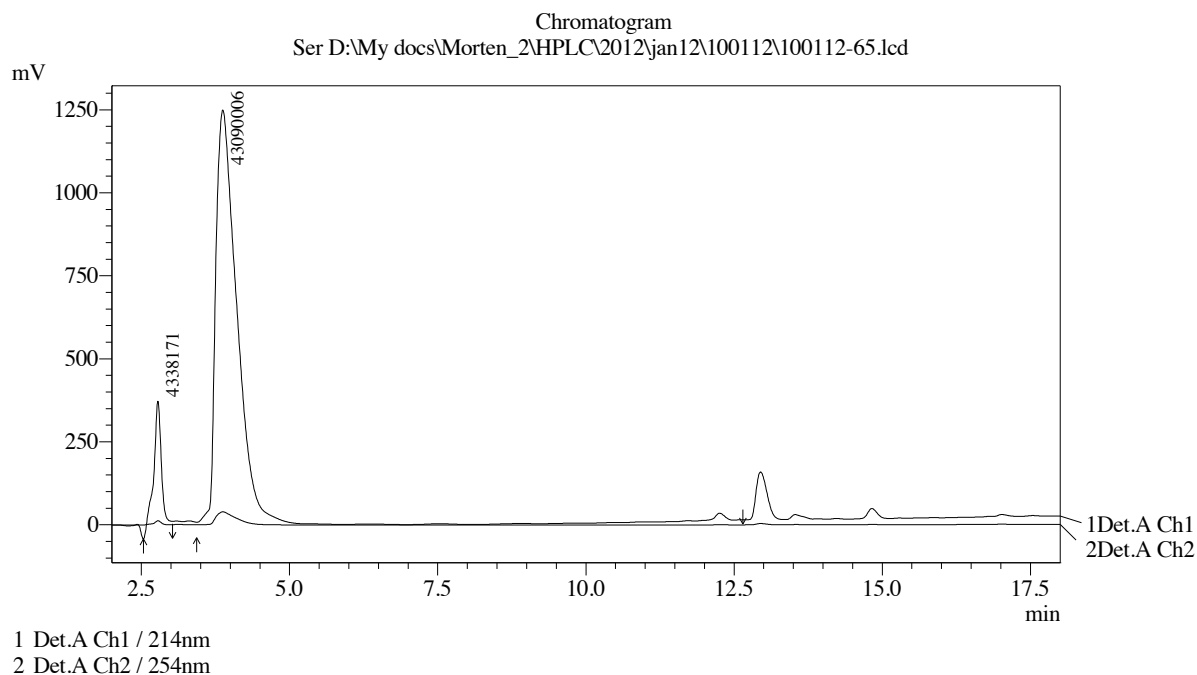
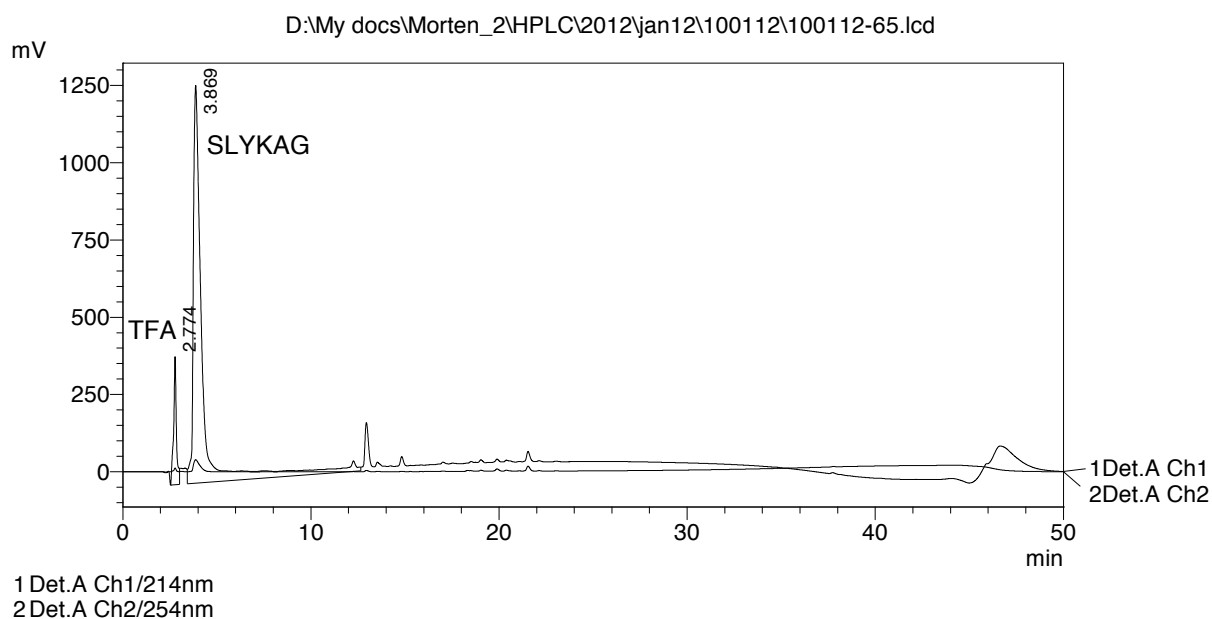


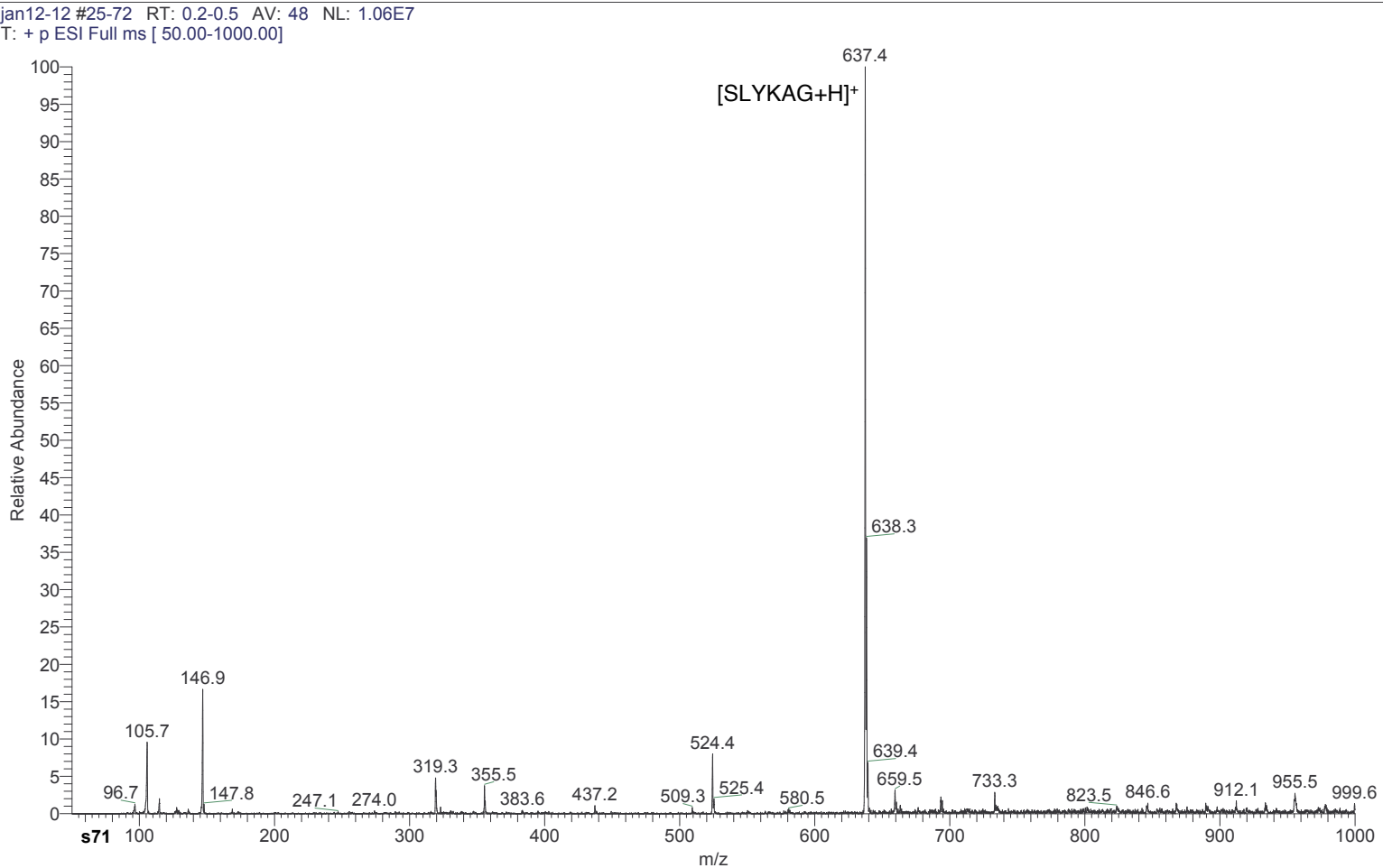
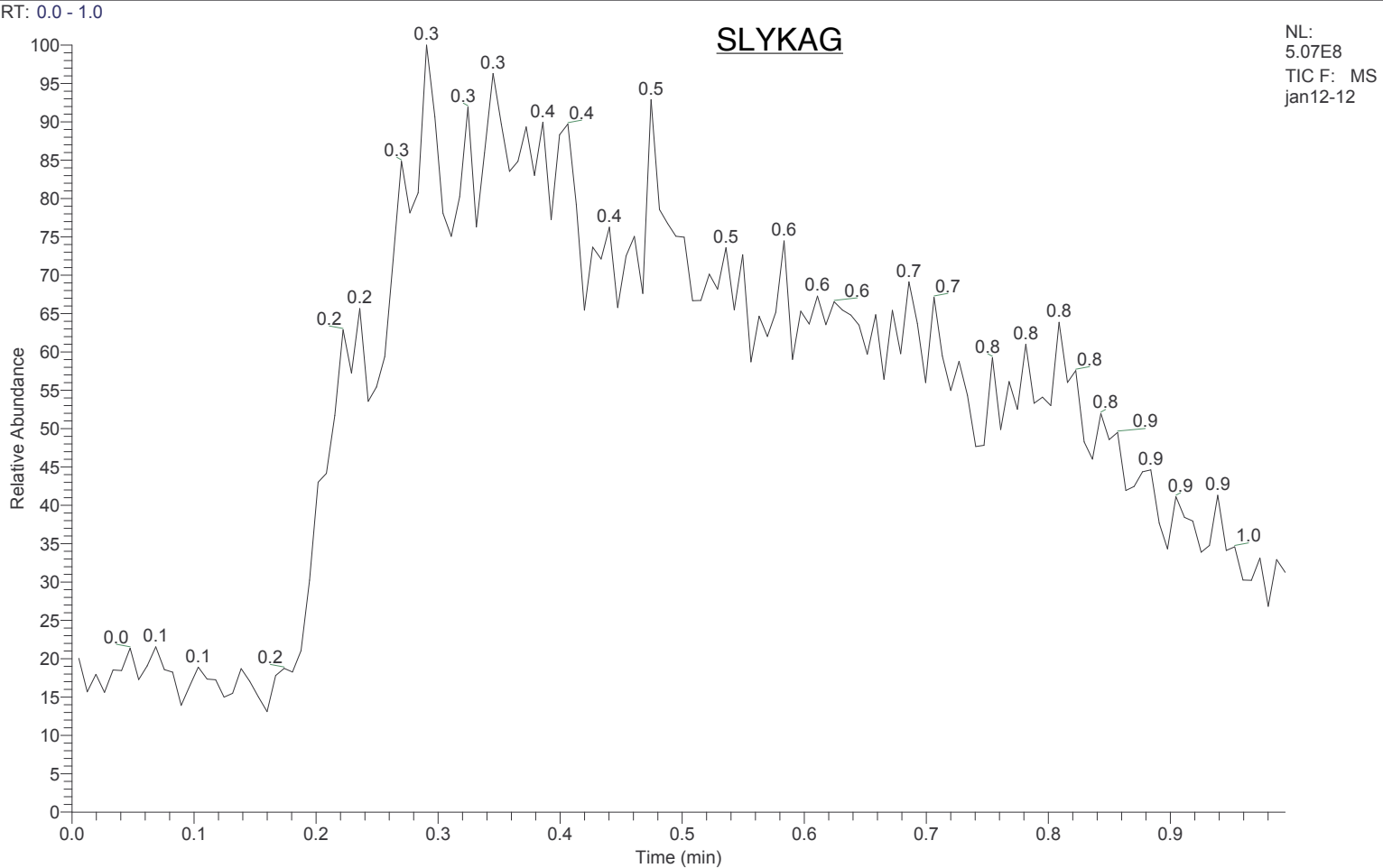
feb12-07 #852-880 RT: 16.07-16.53 AV: 29 NL: 7.00E4
T: ITMS + p ESI E Full ms [100.00-2000.00]



SLYKAG**==== Shimadzu LCsolution Analysis Report ====**

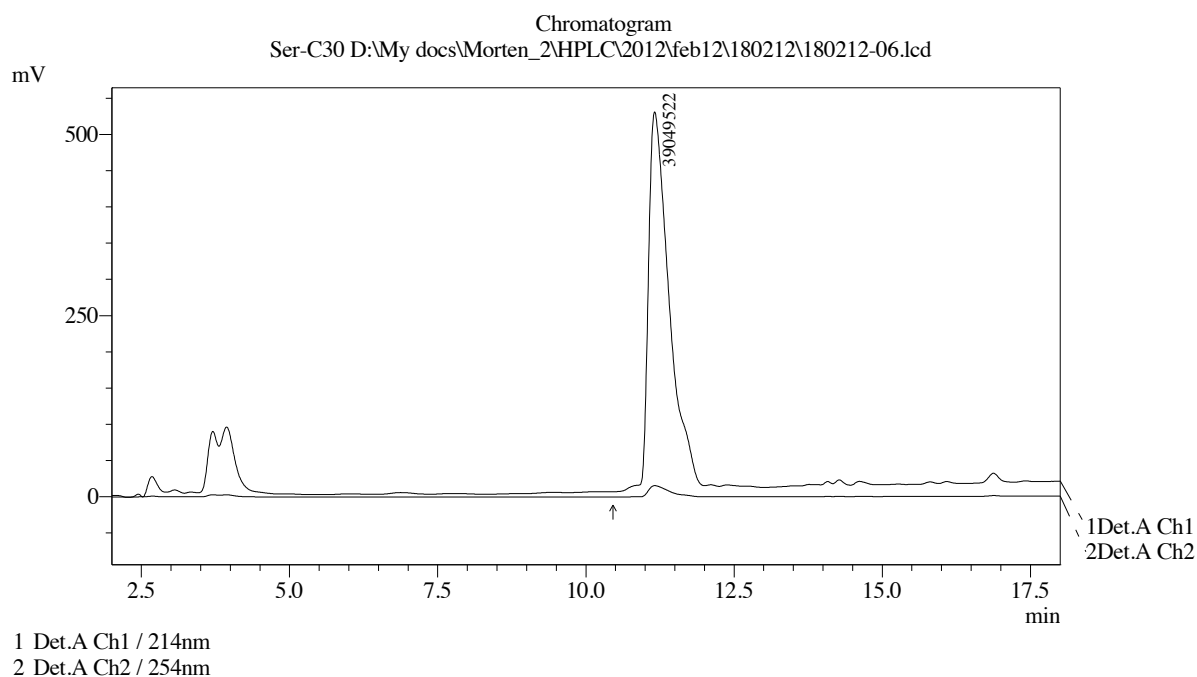
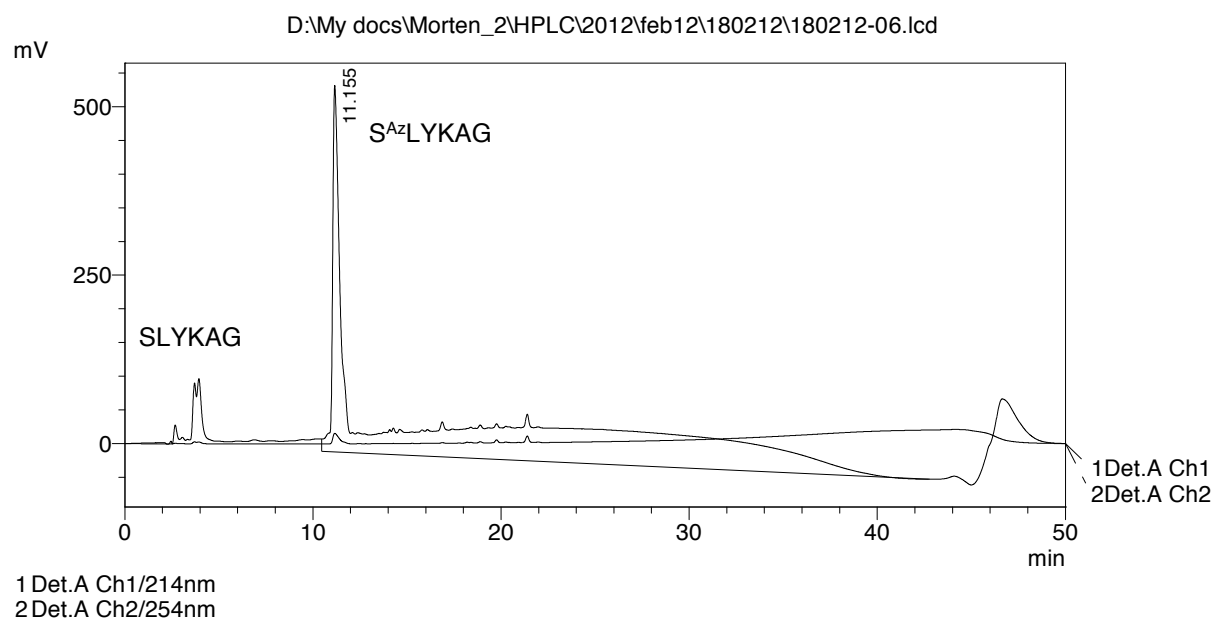
D:\My docs\Morten_2\HPLC\2012\jan12\100112\100112-65.lcd
 Acquired by : user
 Sample Name : Ser
 Sample ID : bef. diazotization
 Tray# : 1
 Vail # : 83
 Injection Volume : 10 uL
 Data File Name : 100112-65.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 100112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 13-Jan-12 4:13:04
 Data Processed : 13-Jan-12 5:03:08



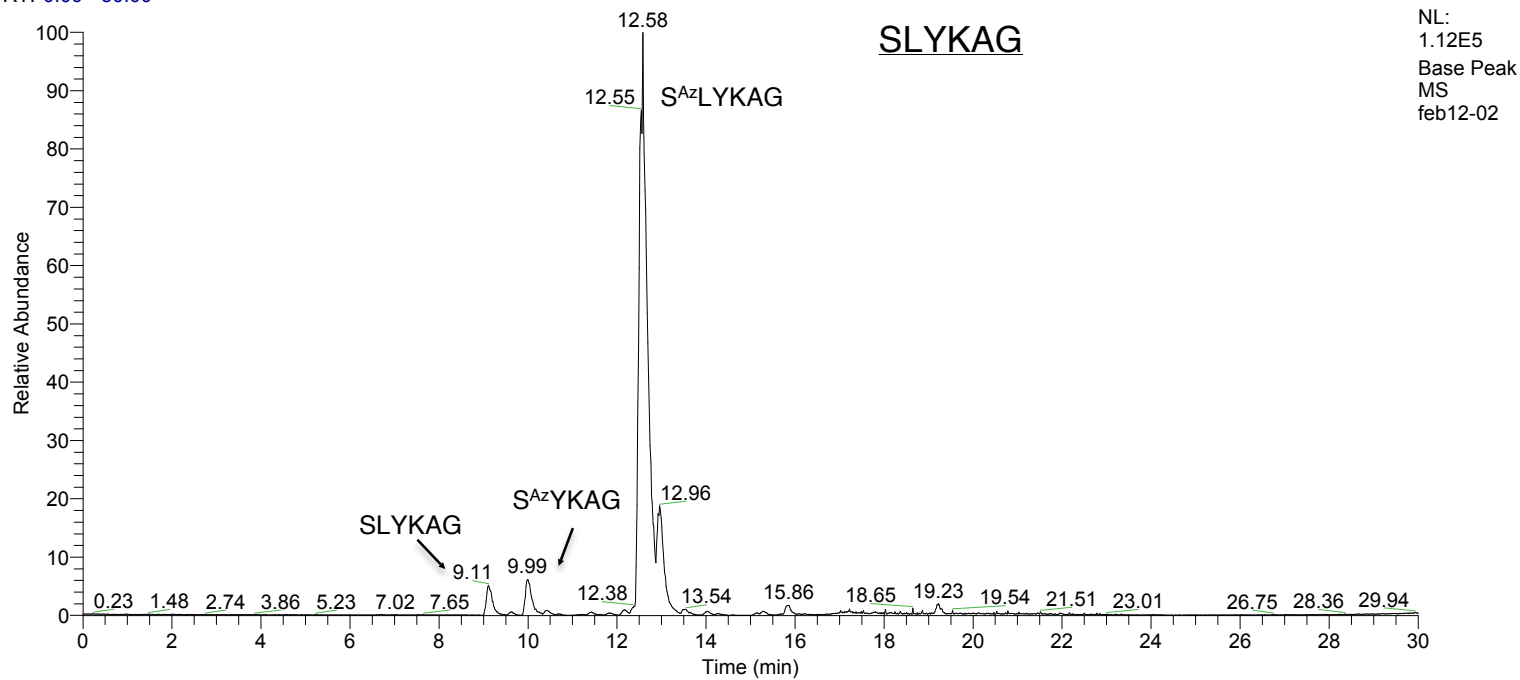


SLYKAG**==== Shimadzu LCsolution Analysis Report ====**

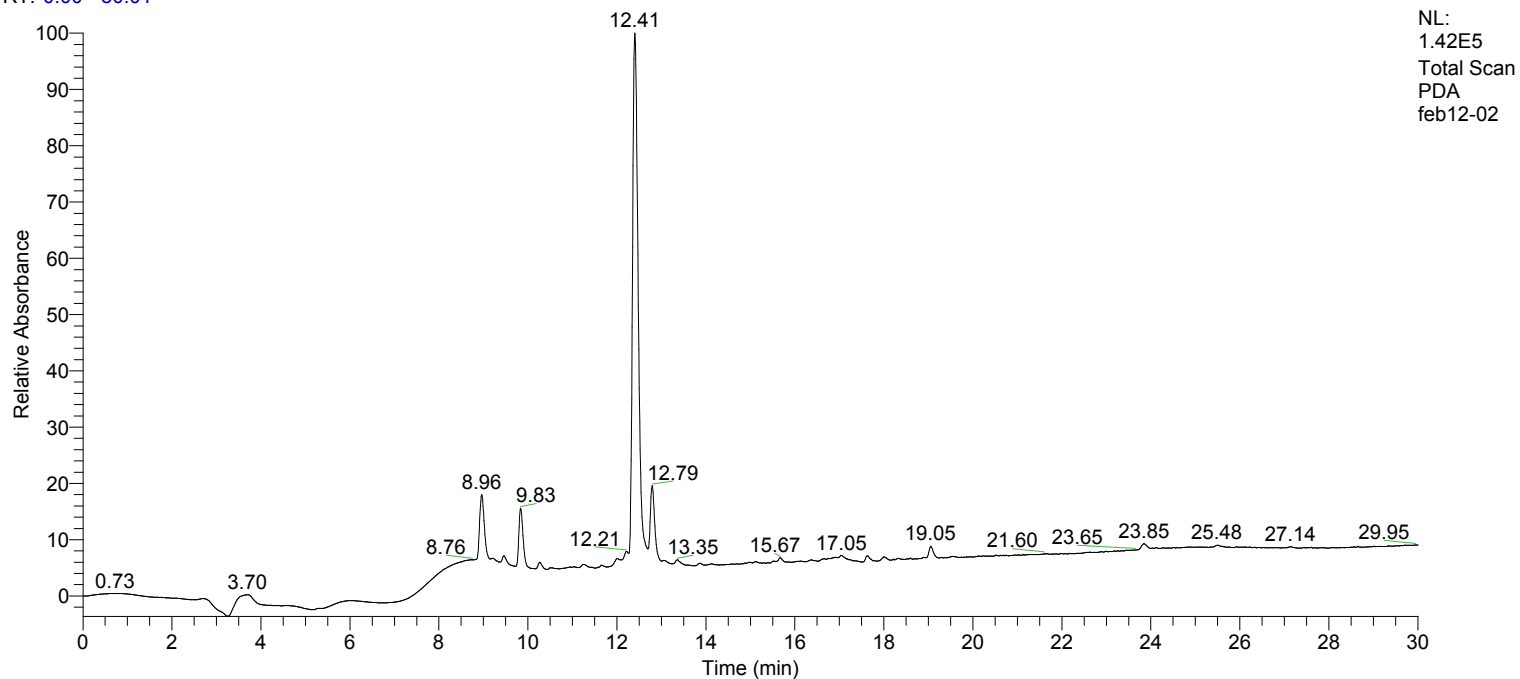
D:\My docs\Morten_2\HPLC\2012\feb12\180212\180212-06.lcd
Acquired by : guest user
Sample Name : Ser-C30
Sample ID :
Tray# : 1
Vial # : 73
Injection Volume : 3 uL
Data File Name : 180212-06.lcd
Method File Name : gradient-anal.lcm
Batch File Name : 20120218.lcb
Report File Name : Default.lcr
Data Acquired : 19-Feb-12 2:18:13
Data Processed : 19-Feb-12 3:08:18



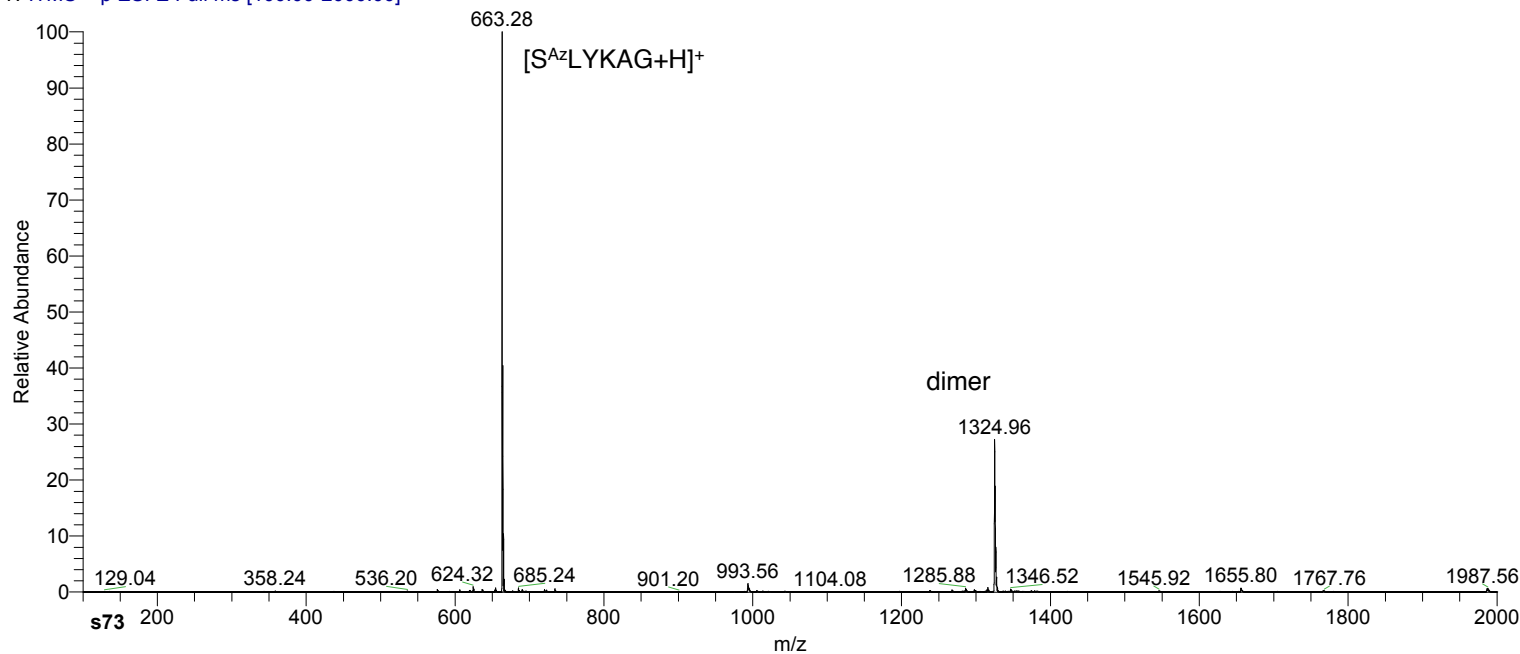
RT: 0.00 - 30.00



RT: 0.00 - 30.01

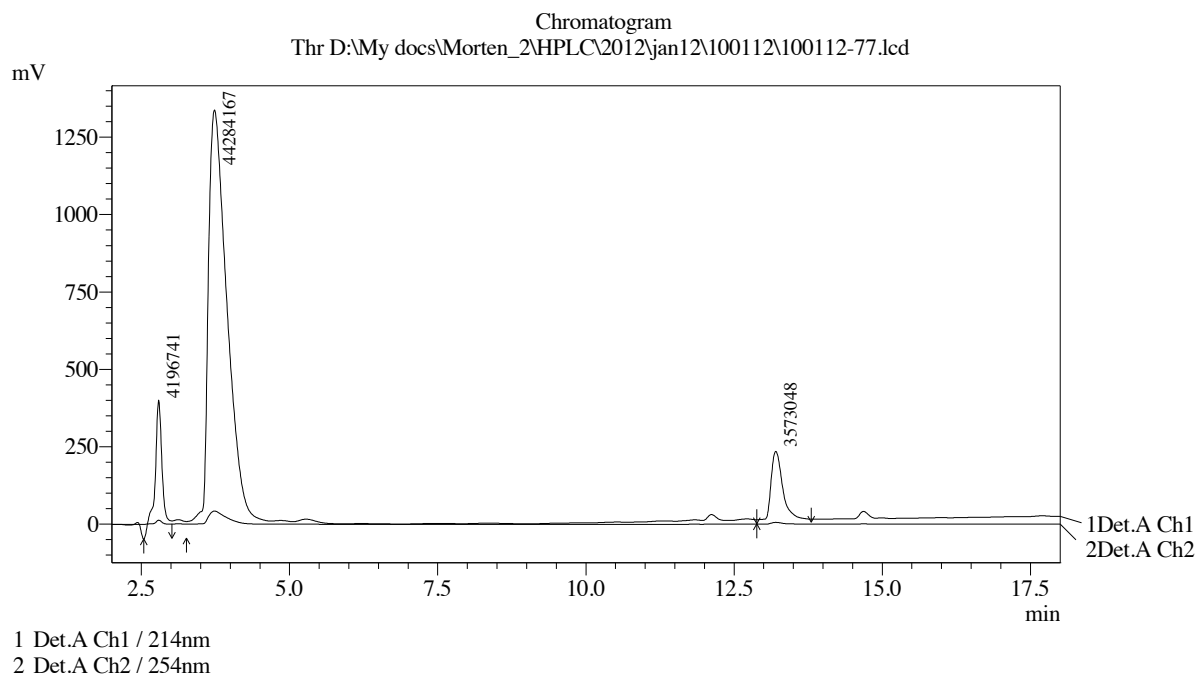
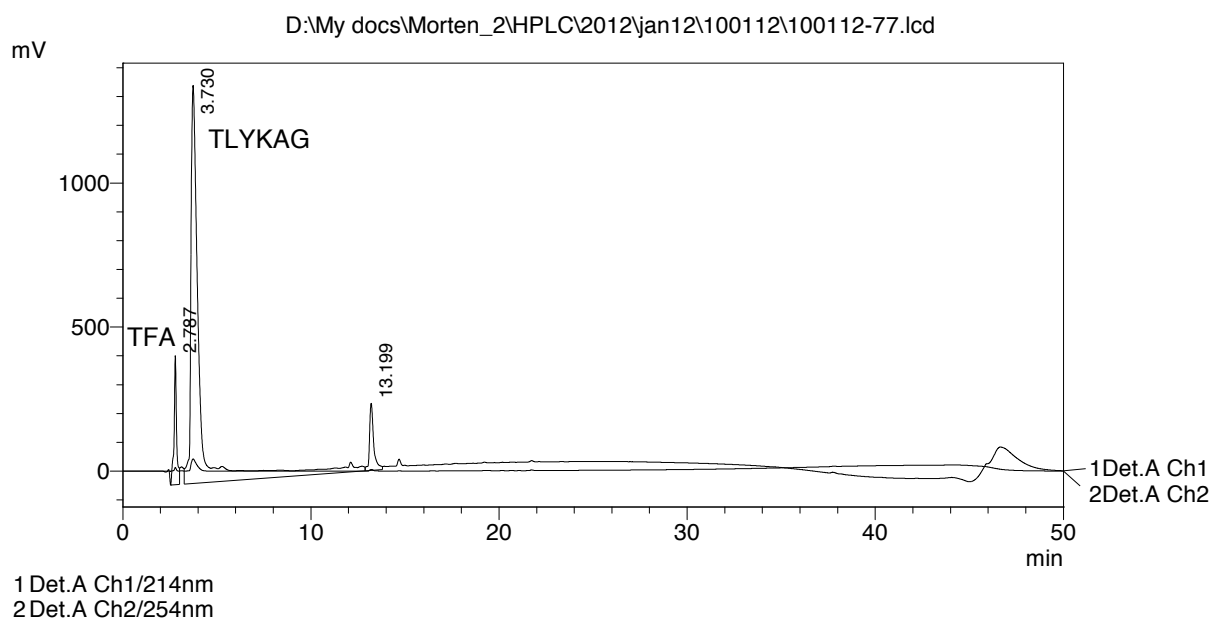


feb12-02 #661-707 RT: 12.38-13.14 AV: 47 NL: 3.15E4
T: ITMS + p ESI E Full ms [100.00-2000.00]

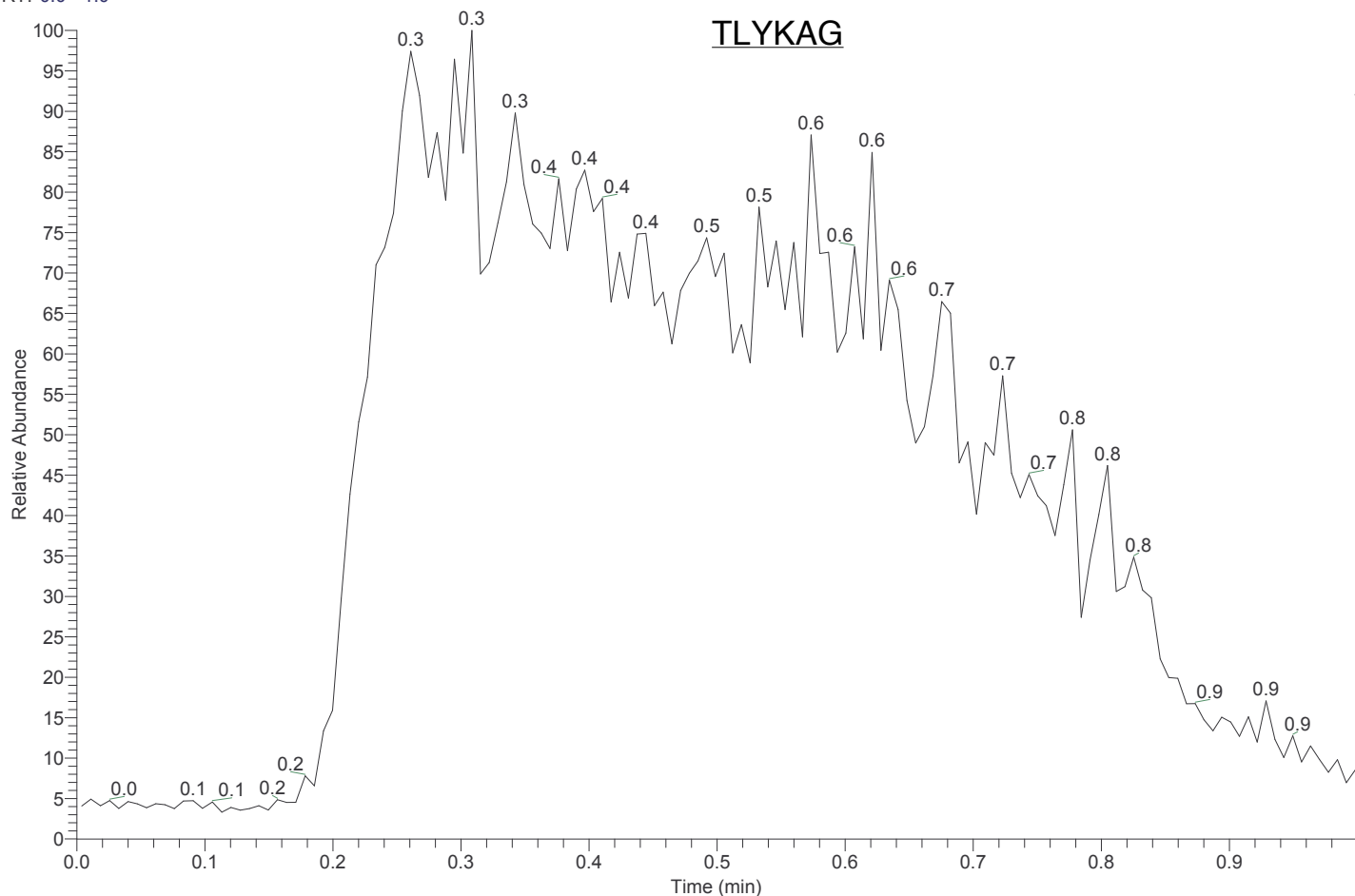
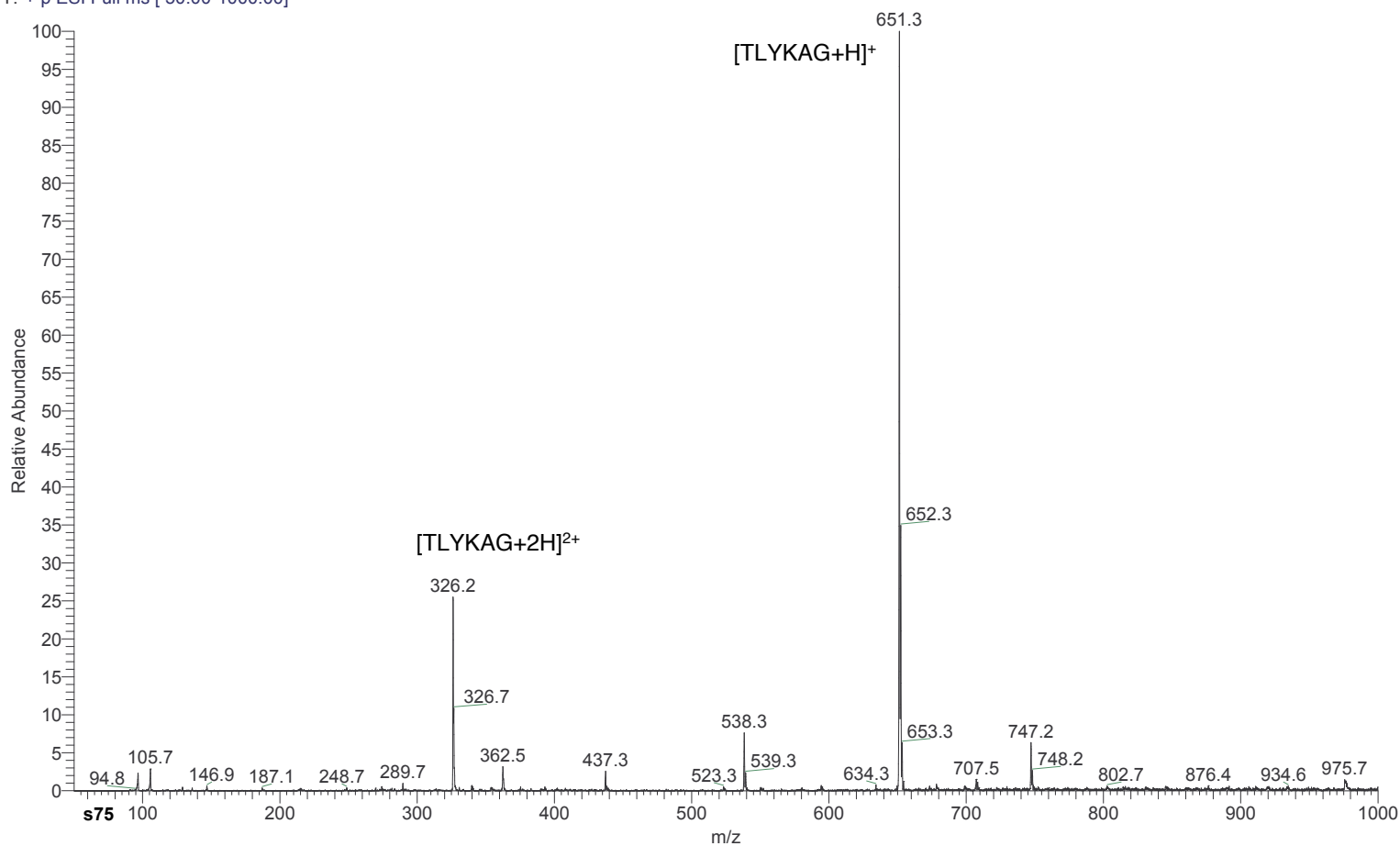


TLYKAG**==== Shimadzu LCsolution Analysis Report ====**

D:\My docs\Morten_2\HPLC\2012\jan12\100112\100112-77.lcd
 Acquired by : user
 Sample Name : Thr
 Sample ID : bef. diazotization
 Tray# : 1
 Vail # : 95
 Injection Volume : 10 uL
 Data File Name : 100112-77.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 100112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 13-Jan-12 14:18:35
 Data Processed : 13-Jan-12 15:08:38

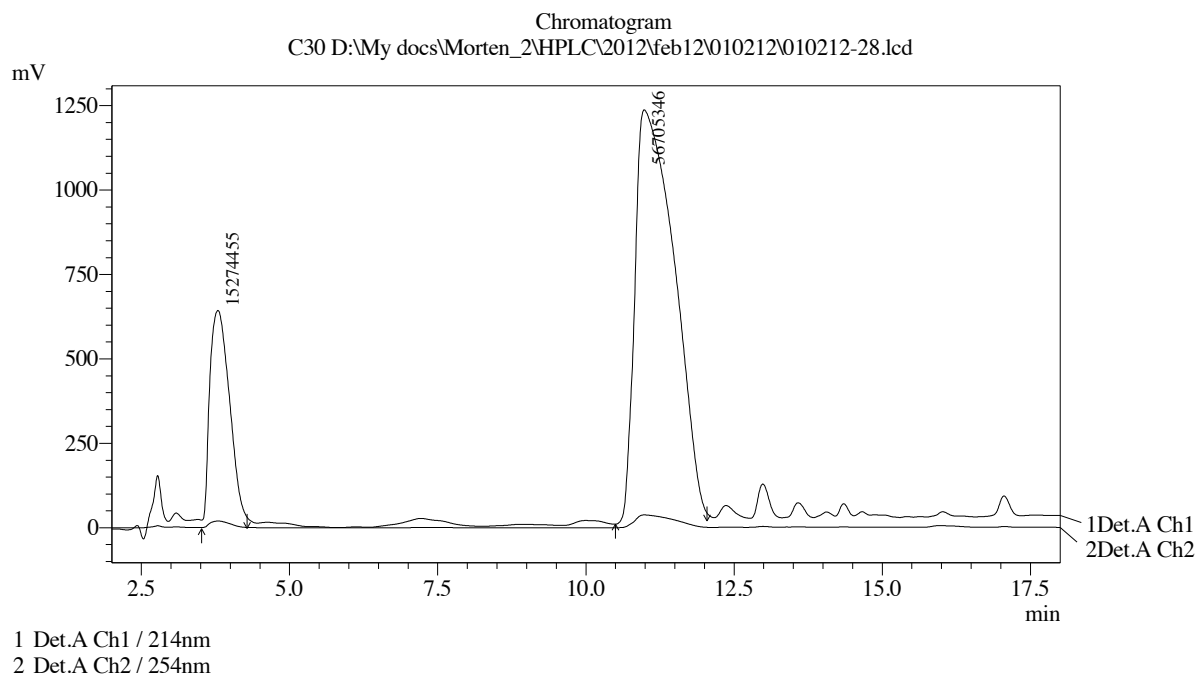
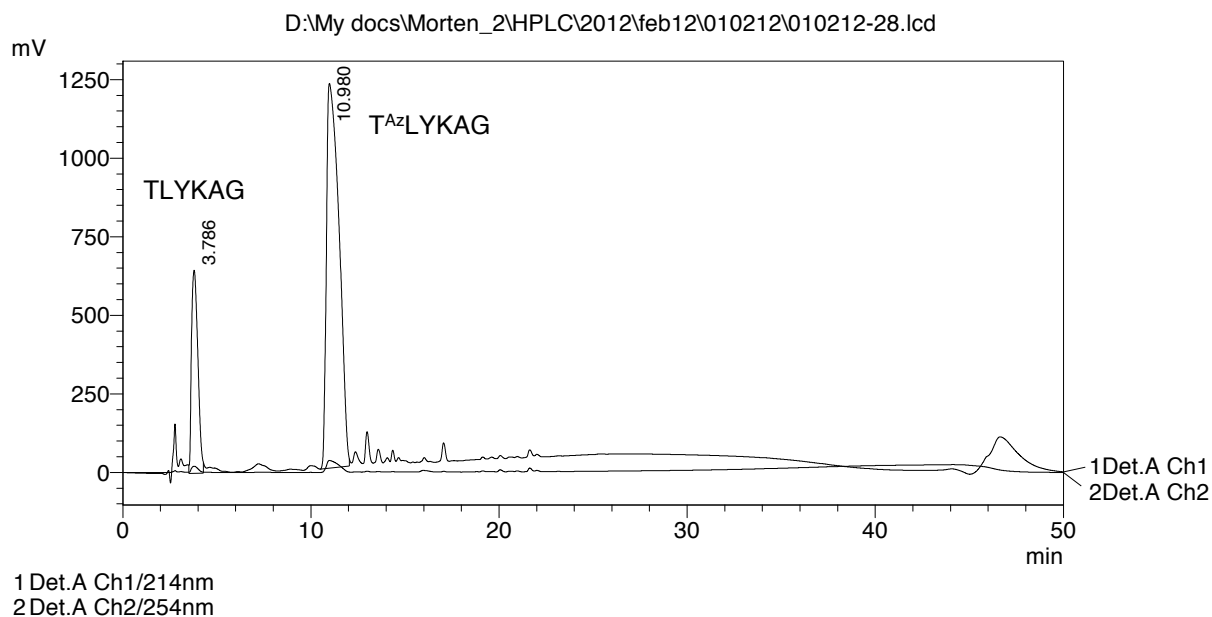


RT: 0.0 - 1.0

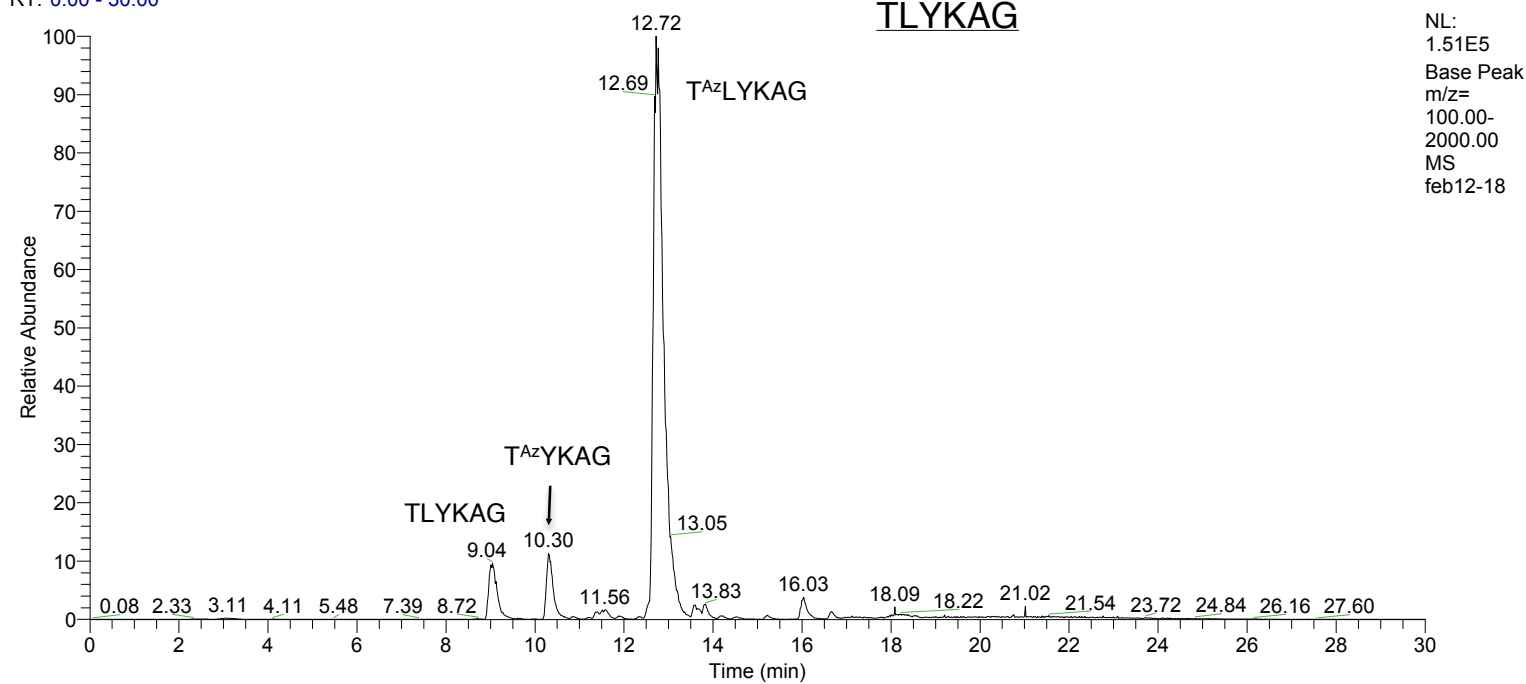
NL:
8.67E8
TIC F: MS
jan12-26jan12-26 #25-83 RT: 0.2-0.6 AV: 59 NL: 2.14E7
T: + p ESI Full ms [50.00-1000.00]

TLYKAG**==== Shimadzu LCsolution Analysis Report ====**

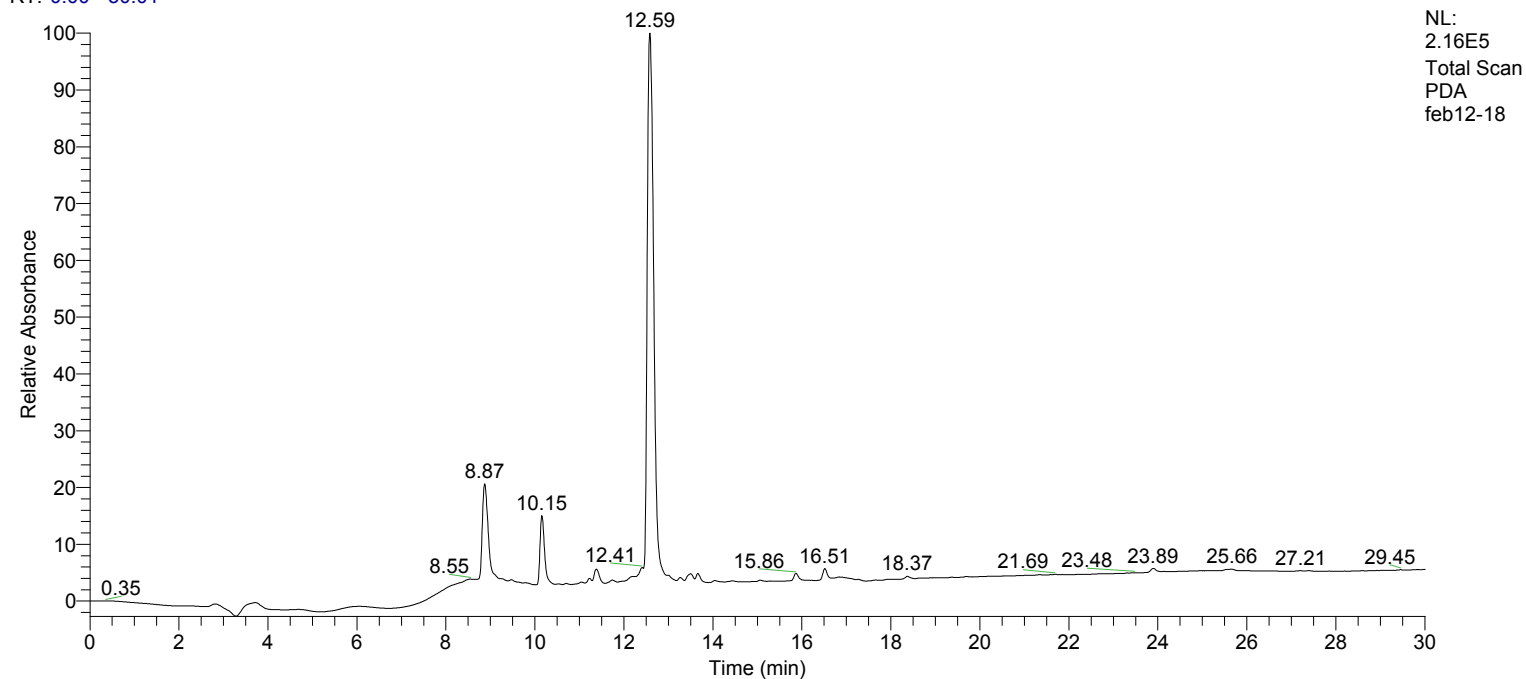
D:\My docs\Morten_2\HPLC\2012\feb12\010212\010212-28.lcd
 Acquired by : guest user
 Sample Name : C30
 Sample ID : Thr - diazotized
 Tray# : 1
 Vail # : 28
 Injection Volume : 10 uL
 Data File Name : 010212-28.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 010212.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 02-Feb-12 18:13:27
 Data Processed : 06-Feb-12 9:01:34



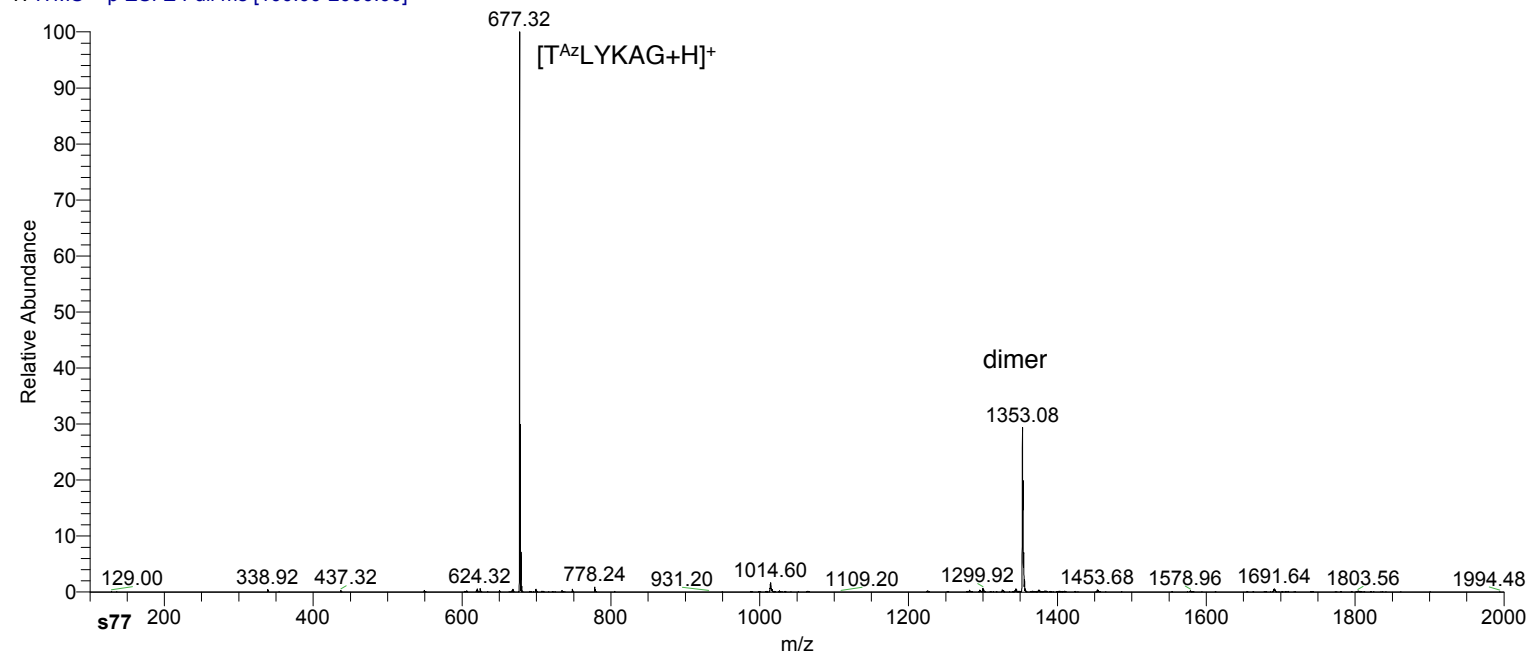
RT: 0.00 - 30.00



RT: 0.00 - 30.01



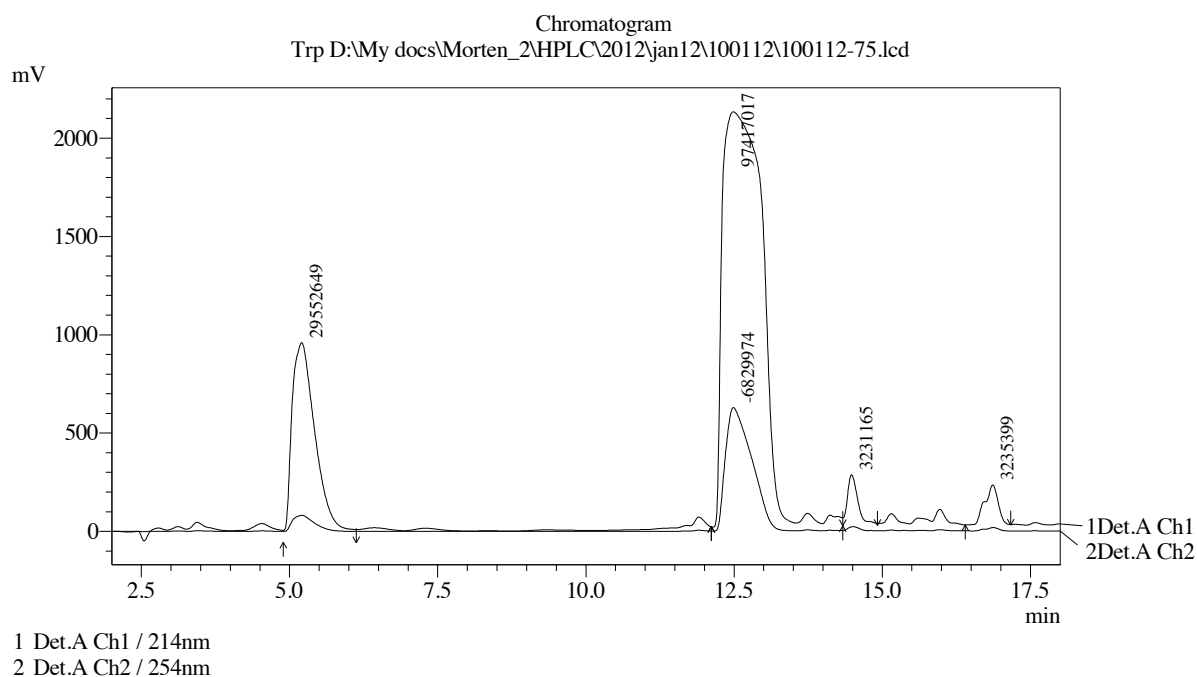
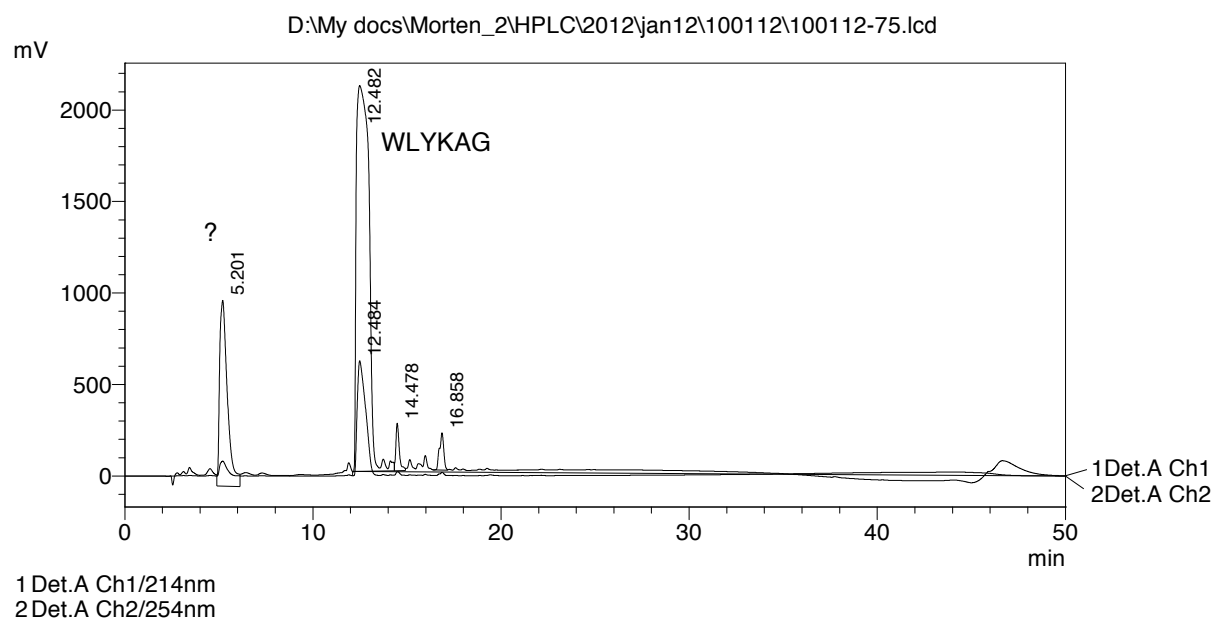
feb12-18 #677-708 RT: 12.57-13.08 AV: 32 NL: 7.41E4
T: ITMS + p ESI E Full ms [100.00-2000.00]



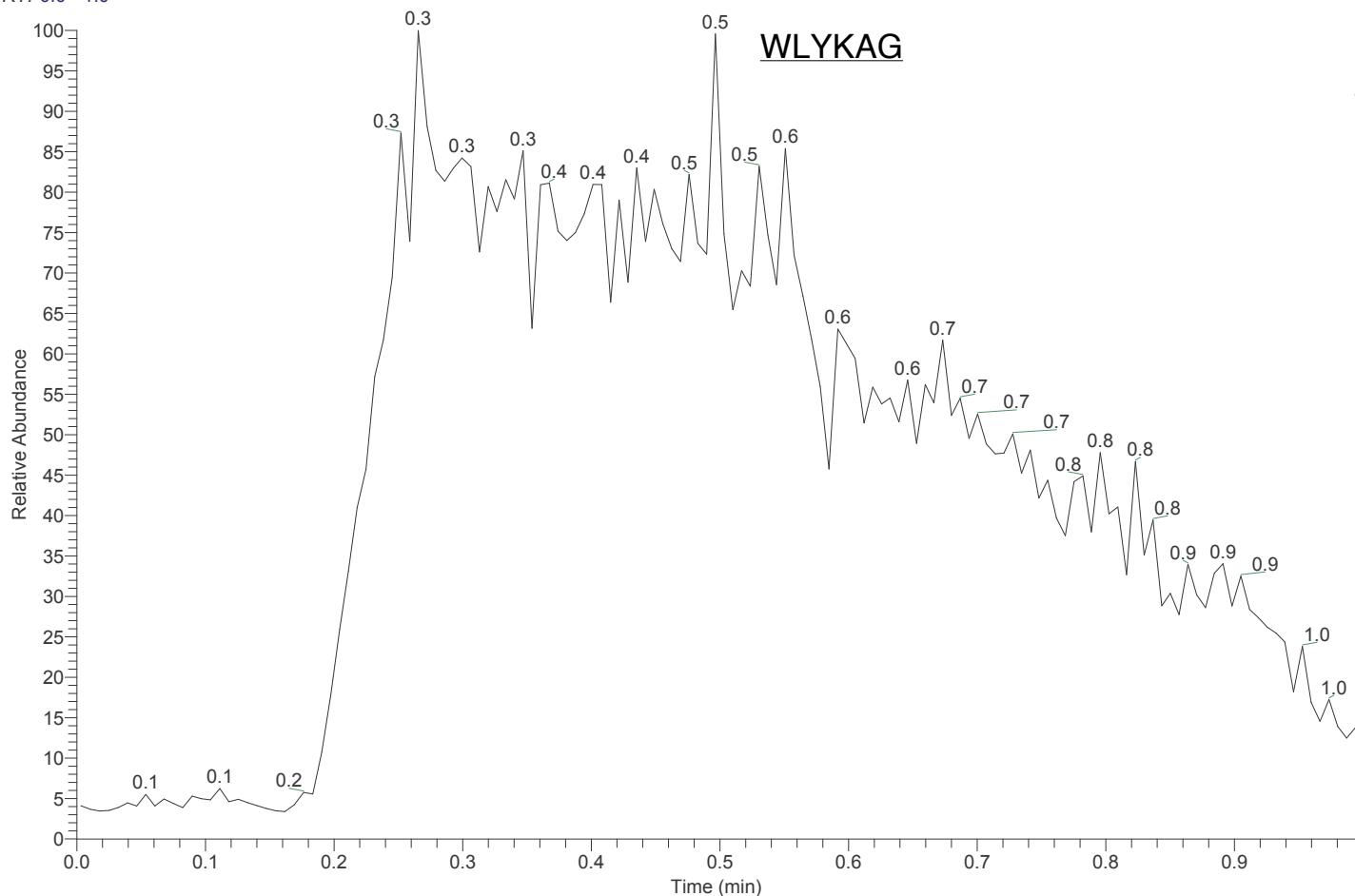
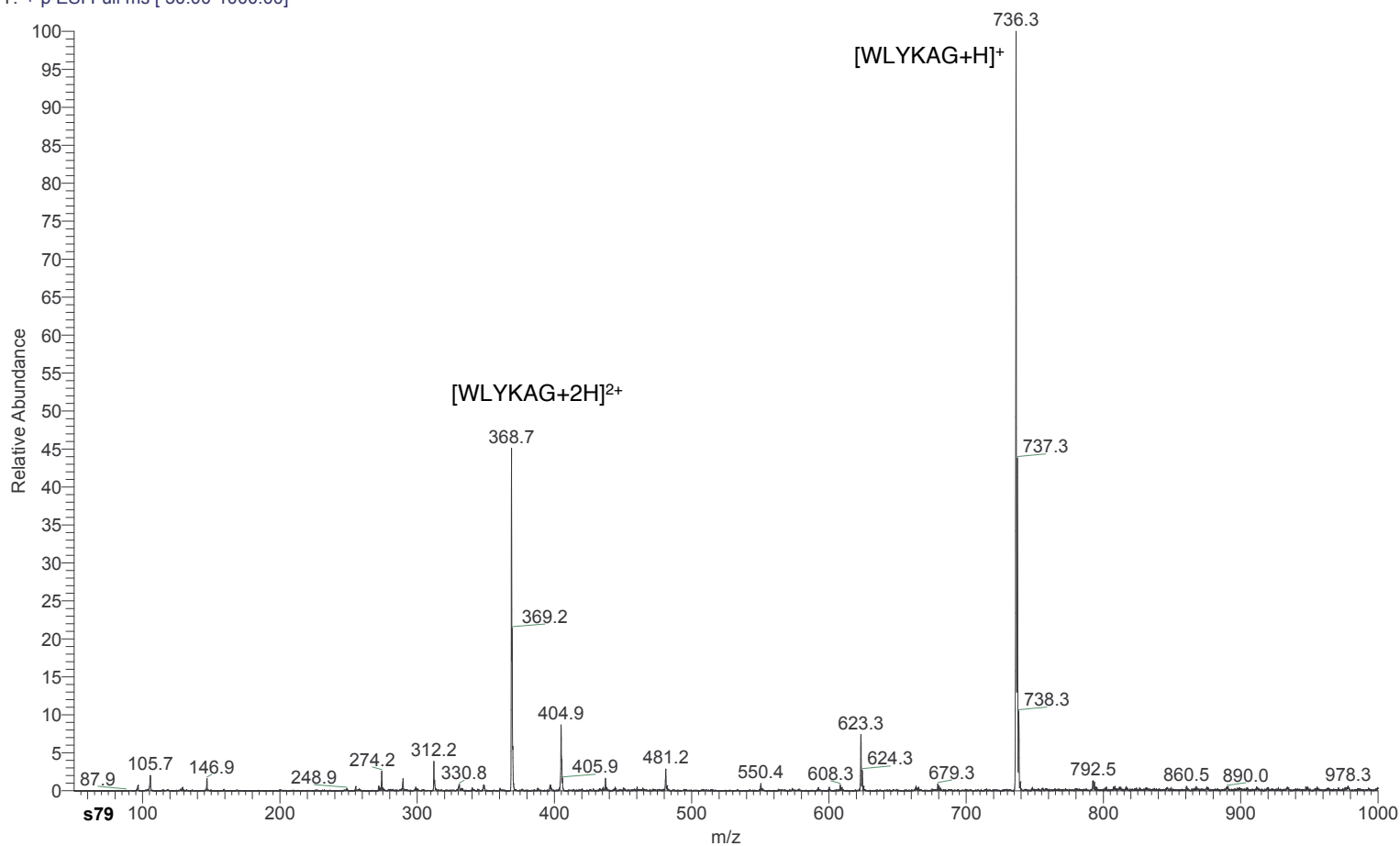
WLYKAG

==== Shimadzu LCsolution Analysis Report ====

D:\My docs\Morten_2\HPLC\2012\jan12\100112\100112-75.lcd
 Acquired by : user
 Sample Name : Trp
 Sample ID : bef. diazotization
 Tray# : 1
 Vail # : 93
 Injection Volume : 10 uL
 Data File Name : 100112-75.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 100112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 13-Jan-12 12:37:40
 Data Processed : 13-Jan-12 13:27:45

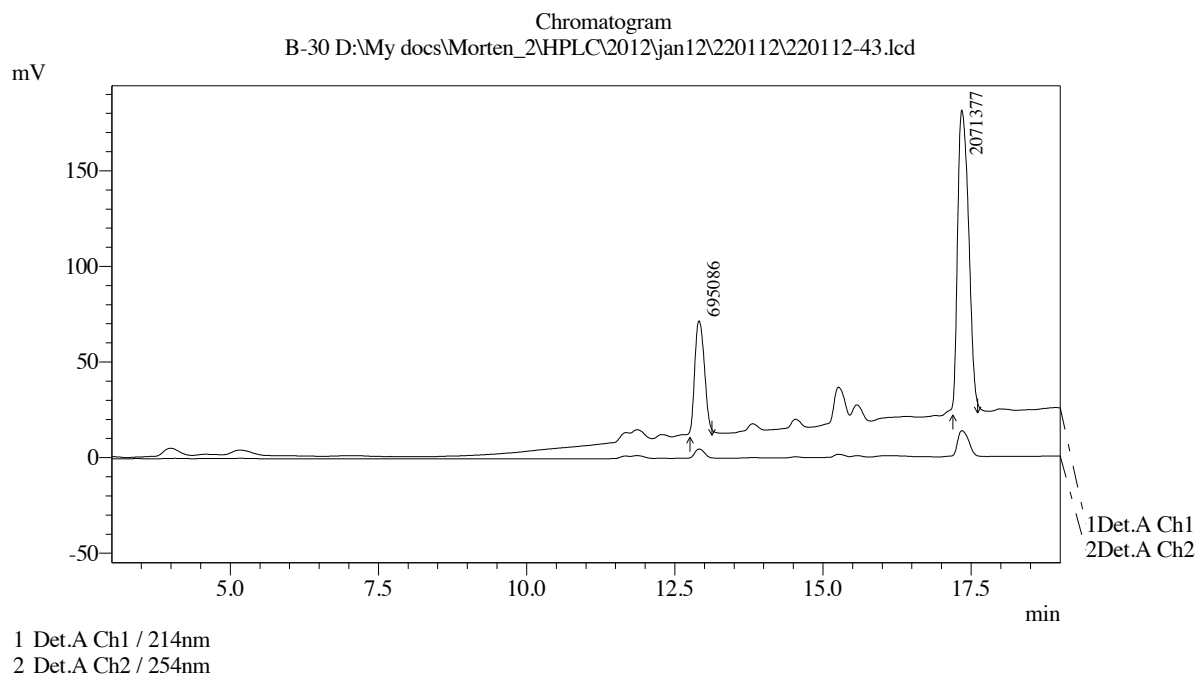
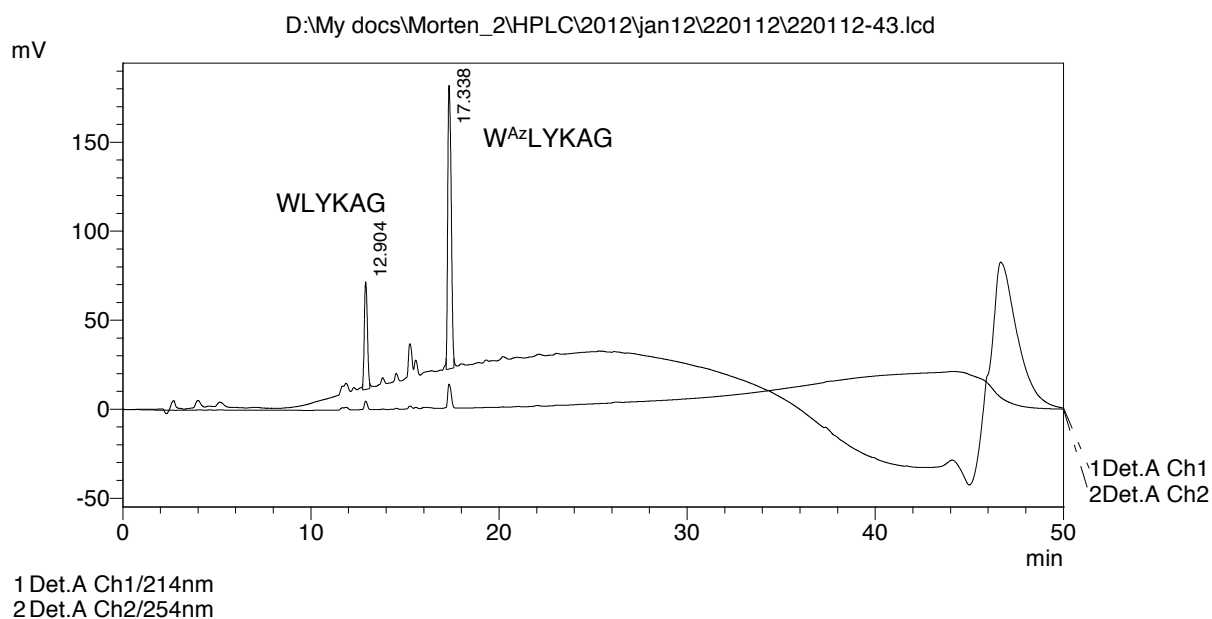


RT: 0.0 - 1.0

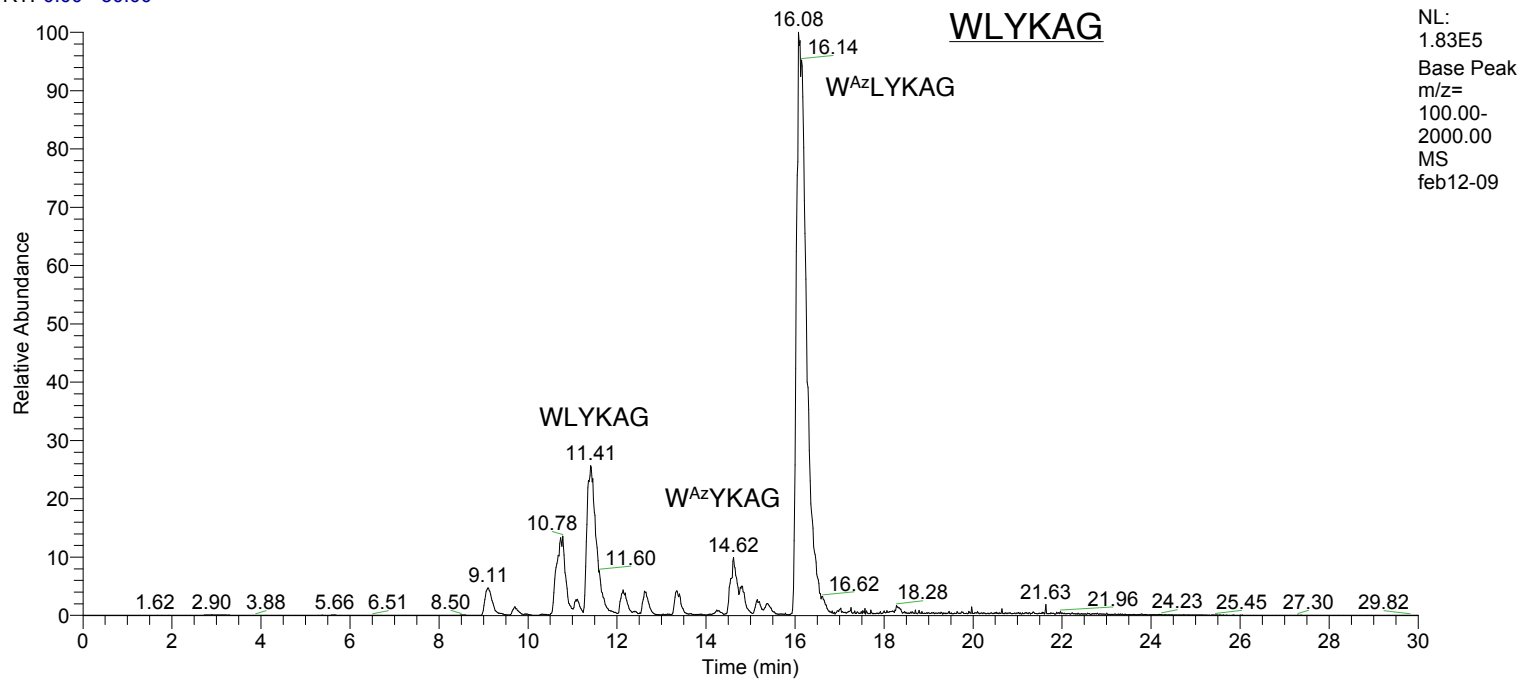
NL:
1.01E9
TIC F: MS
jan12-18jan12-18 #25-95 RT: 0.2-0.7 AV: 71 NL: 2.27E7
T: + p ESI Full ms [50.00-1000.00]

WLYKAG**==== Shimadzu LCsolution Analysis Report ====**

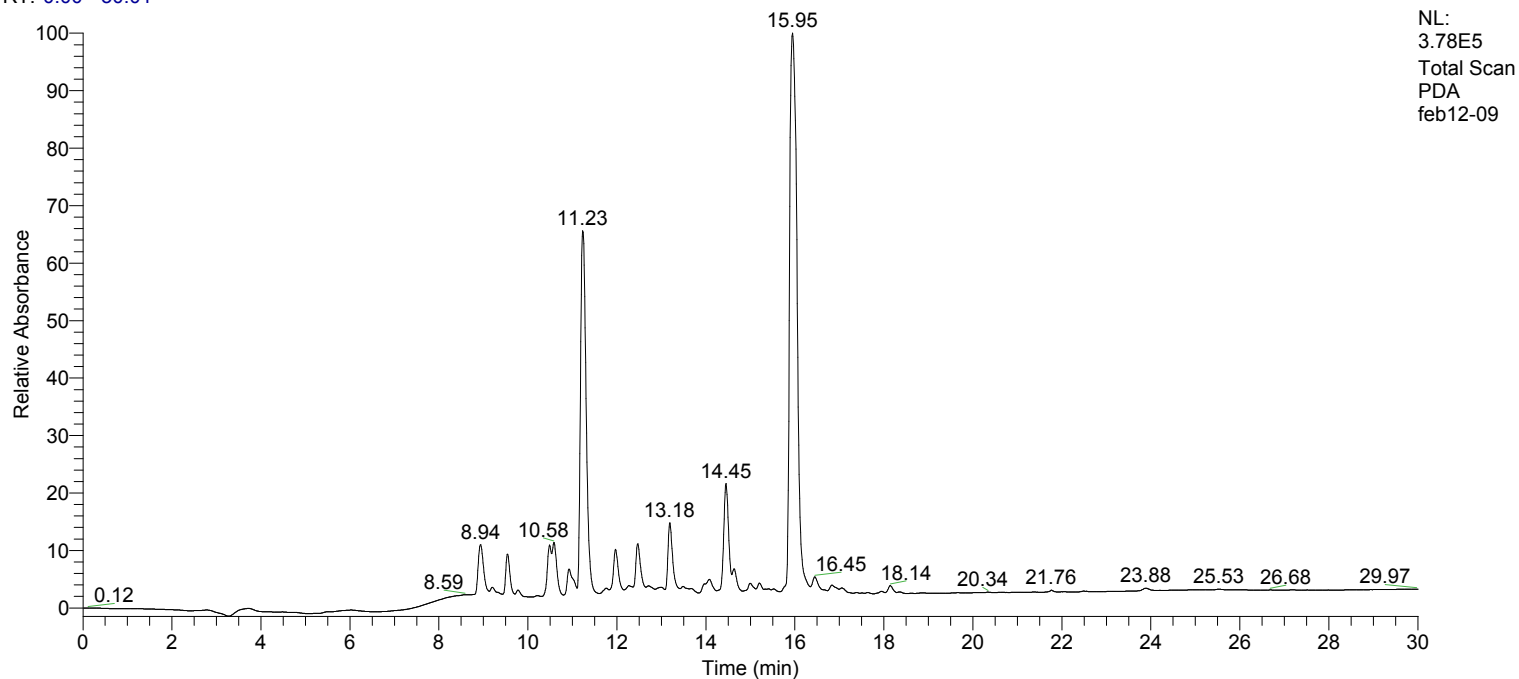
D:\My docs\Morten_2\HPLC\2012\jan12\220112\220112-43.lcd
 Acquired by : user
 Sample Name : B-30
 Sample ID : Trp - diazotized
 Tray# : 1
 Vial # : 102
 Injection Volume : 20 uL
 Data File Name : 220112-43.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 220112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 24-Jan-12 20:15:39
 Data Processed : 31-Jan-12 22:01:29



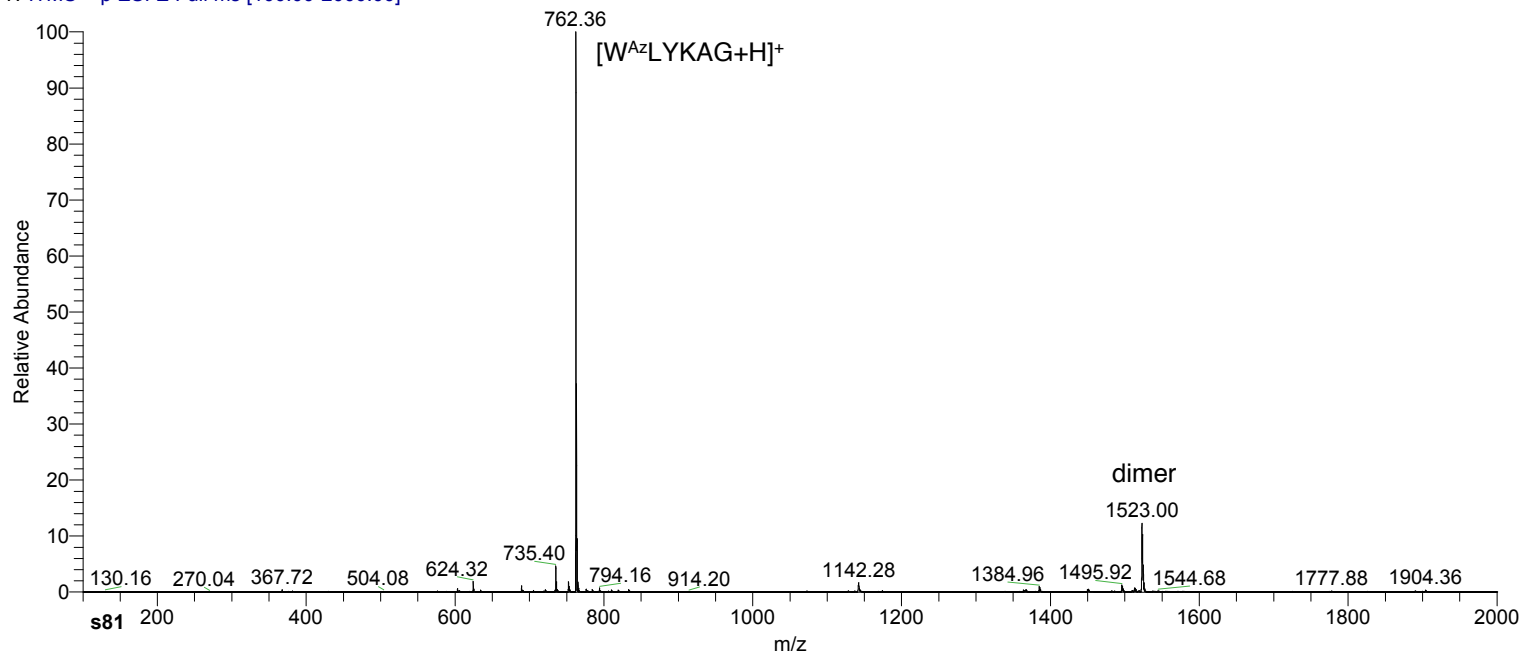
RT: 0.00 - 30.00



RT: 0.00 - 30.01

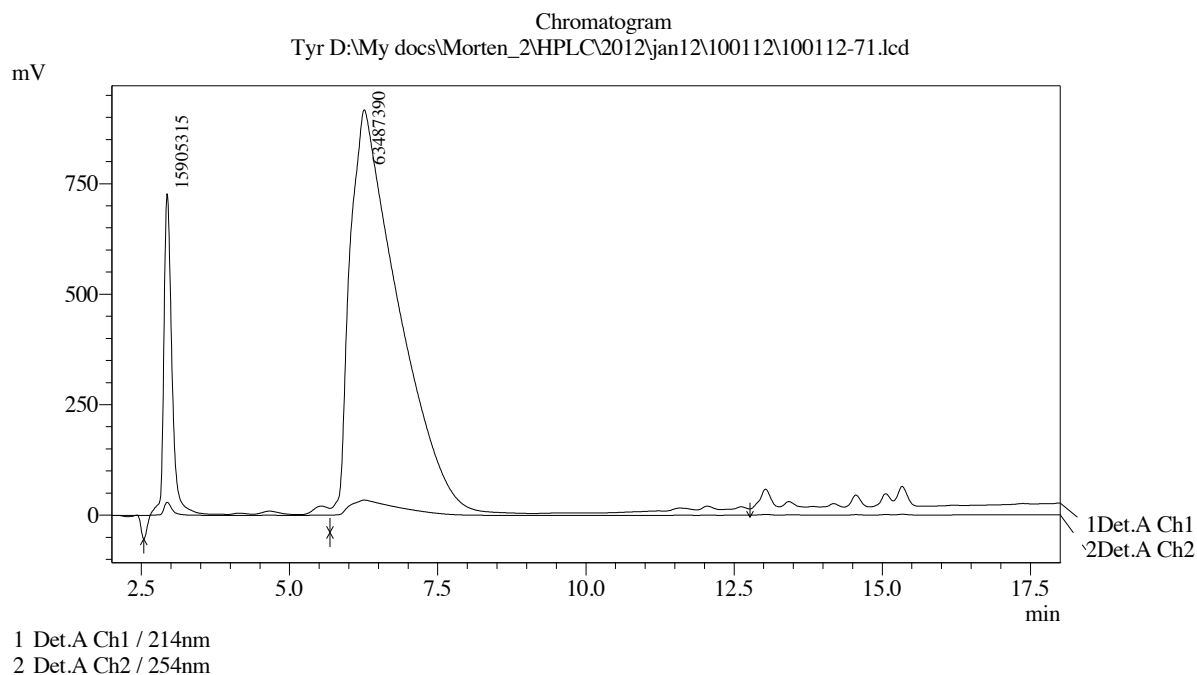
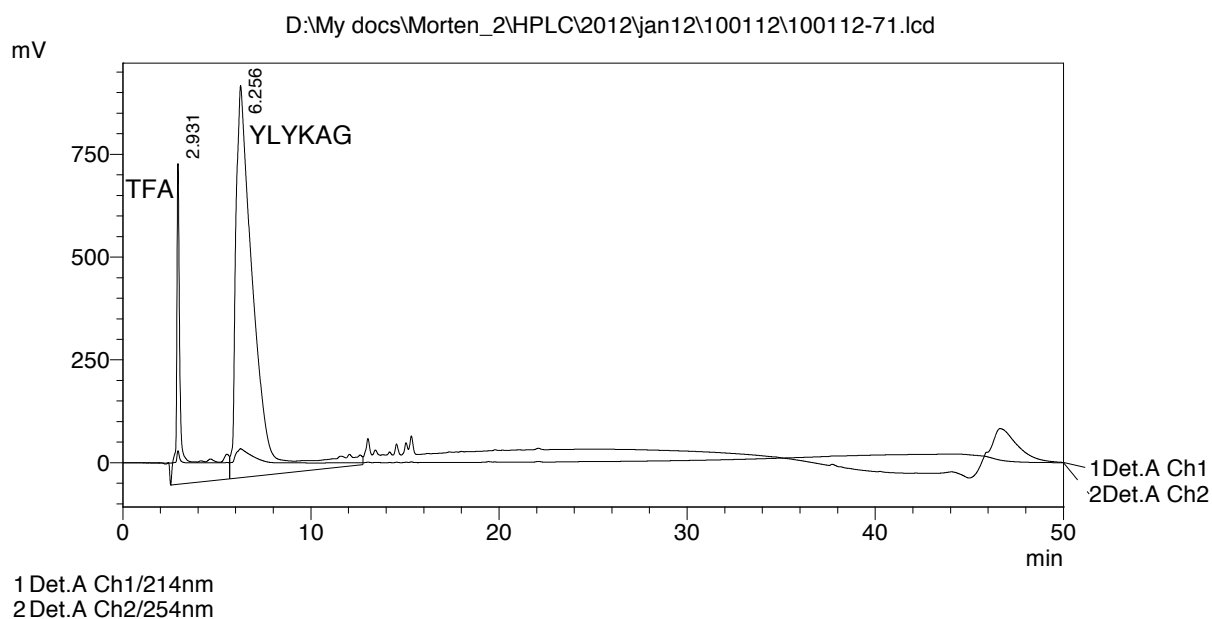


feb12-09 #889-919 RT: 16.01-16.50 AV: 31 NL: 9.03E4
T: ITMS + p ESI E Full ms [100.00-2000.00]

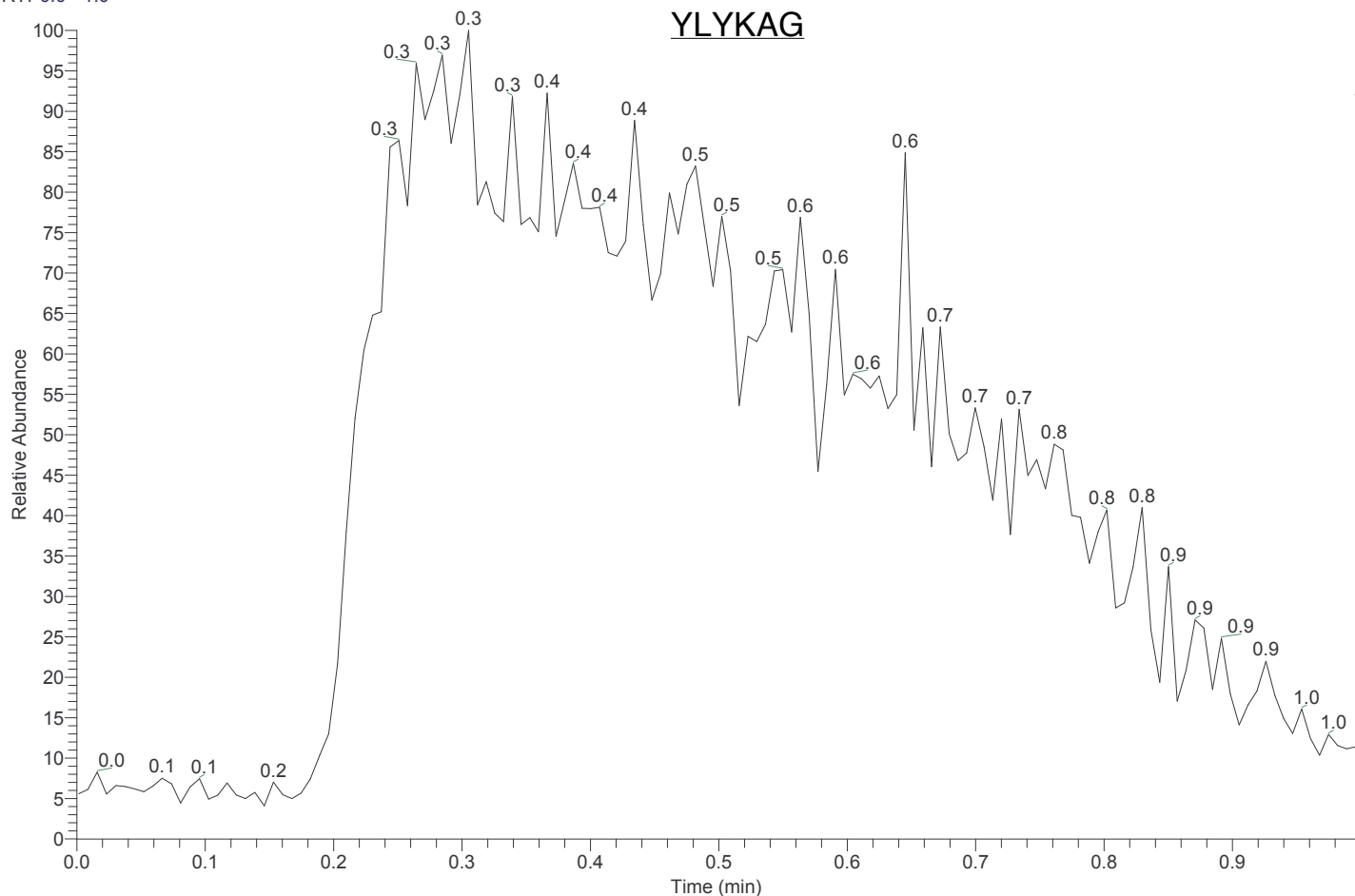
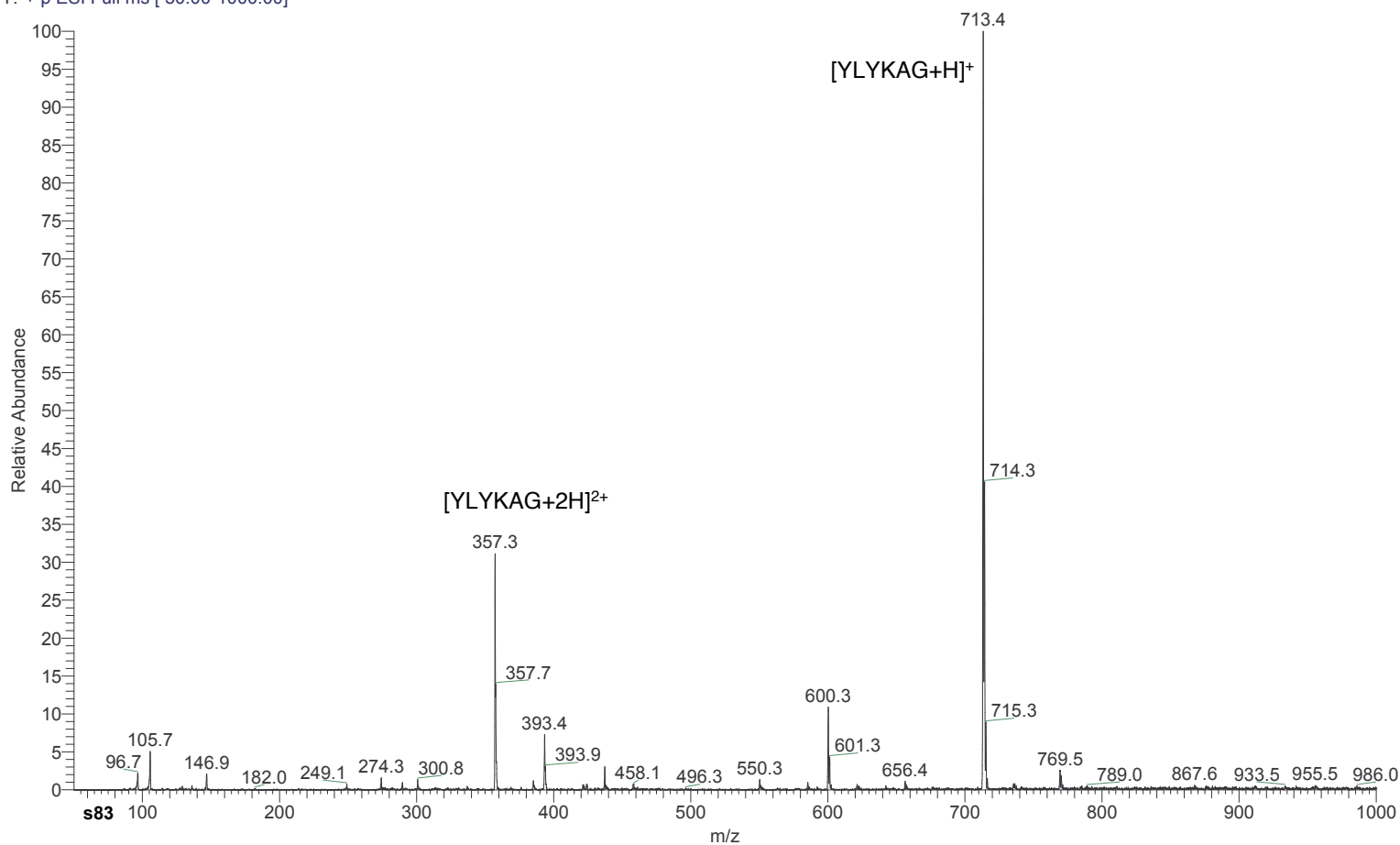


YLYKAG**==== Shimadzu LCsolution Analysis Report ====**

D:\My docs\Morten_2\HPLC\2012\jan12\100112\100112-71.lcd
 Acquired by : user
 Sample Name : Tyr
 Sample ID : bef. diazotization
 Tray# : 1
 Vial # : 89
 Injection Volume : 10 uL
 Data File Name : 100112-71.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 100112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 13-Jan-12 9:15:45
 Data Processed : 13-Jan-12 10:05:49

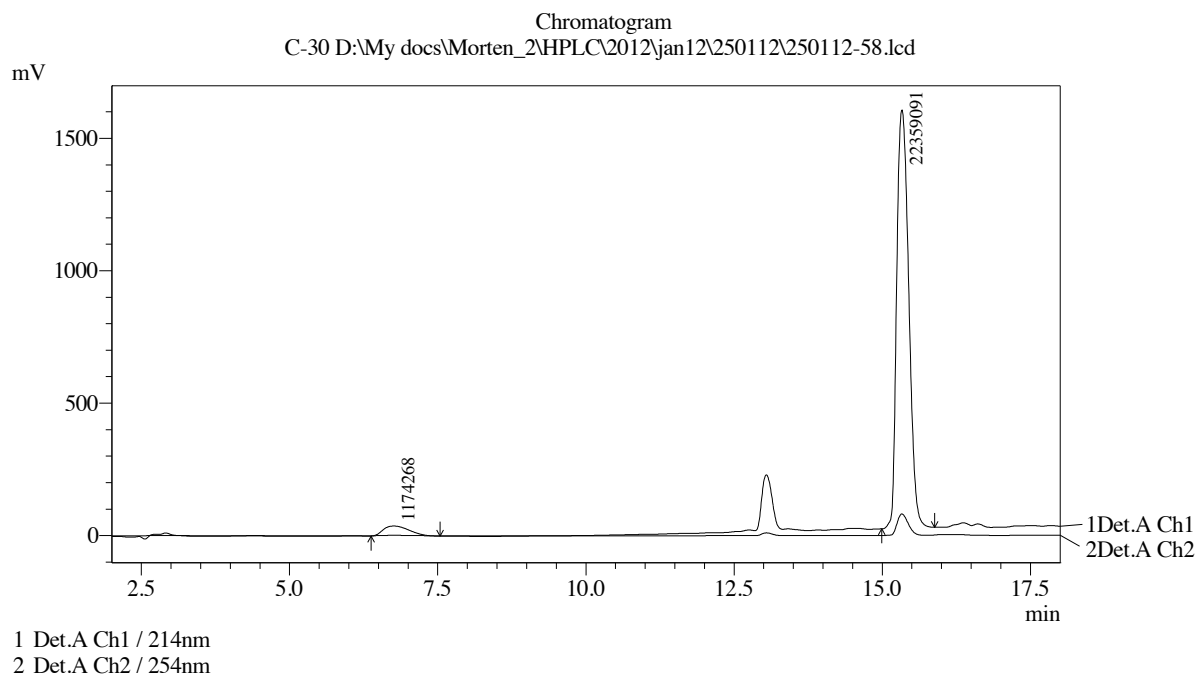
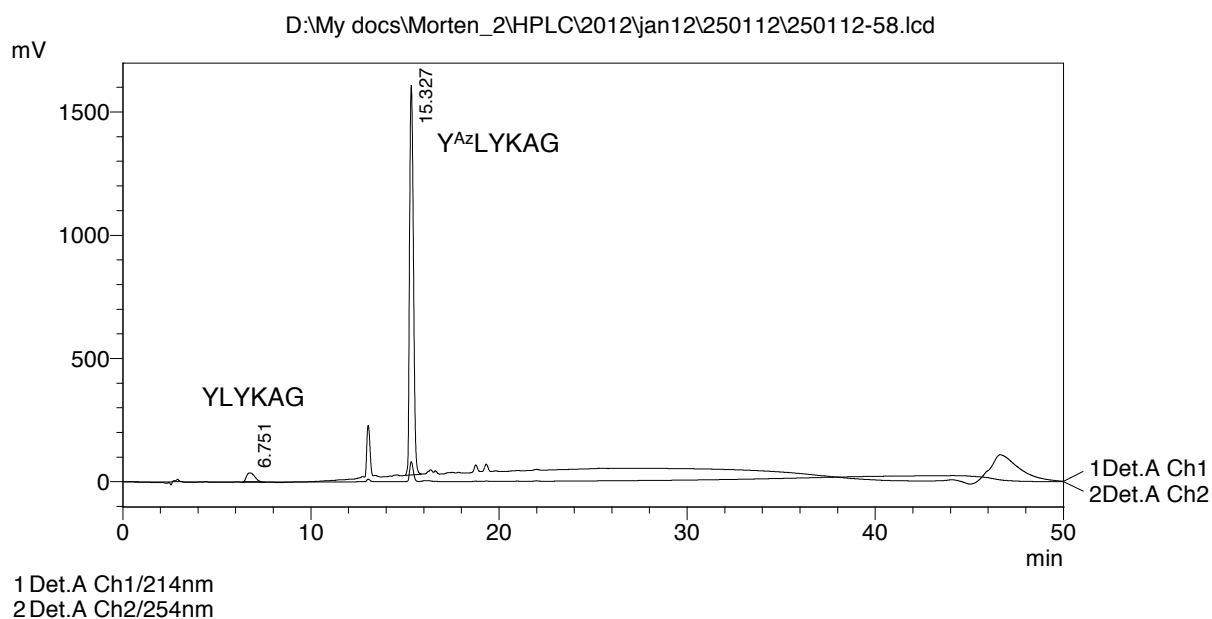


RT: 0.0 - 1.0

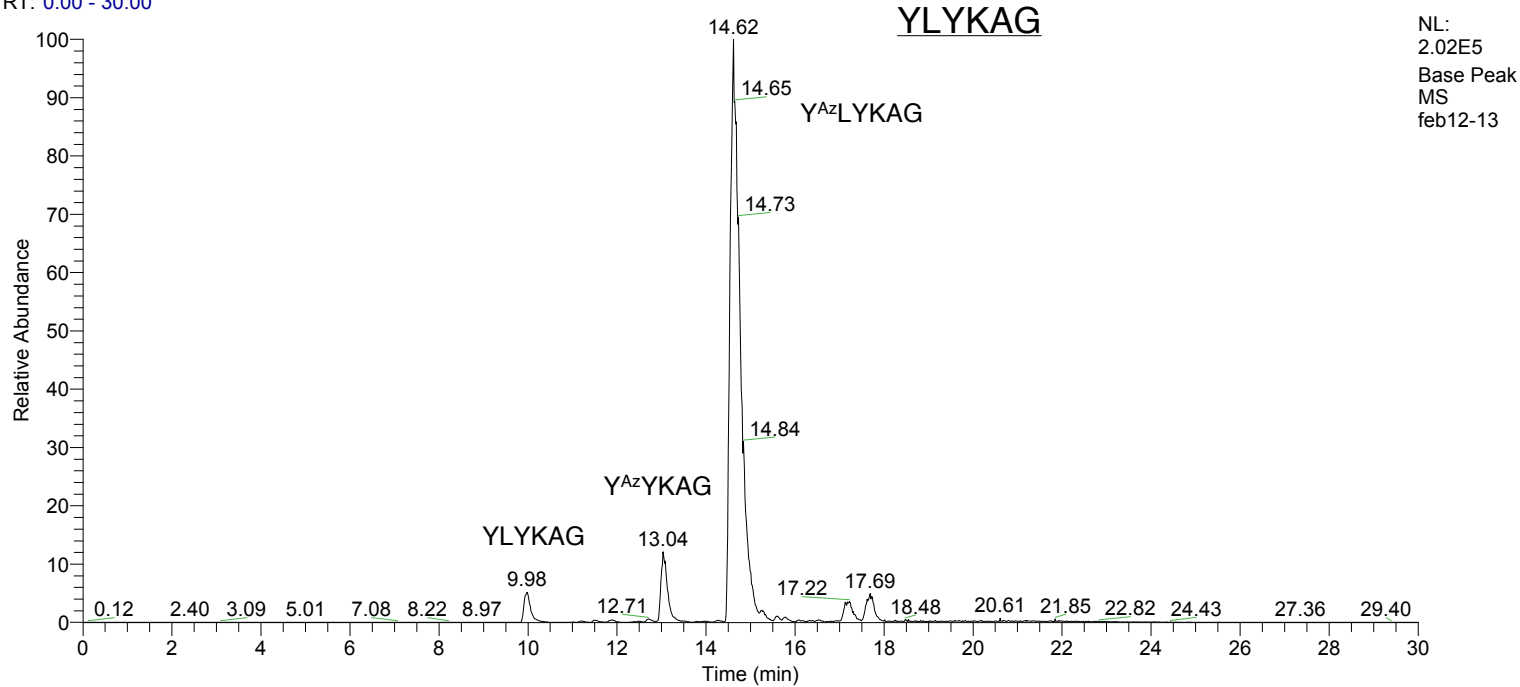
NL:
7.44E8
TIC F: MS
jan12-20jan12-20 #25-91 RT: 0.2-0.6 AV: 67 NL: 1.69E7
T: + p ESI Full ms [50.00-1000.00]

YLYKAG**==== Shimadzu LCsolution Analysis Report ====**

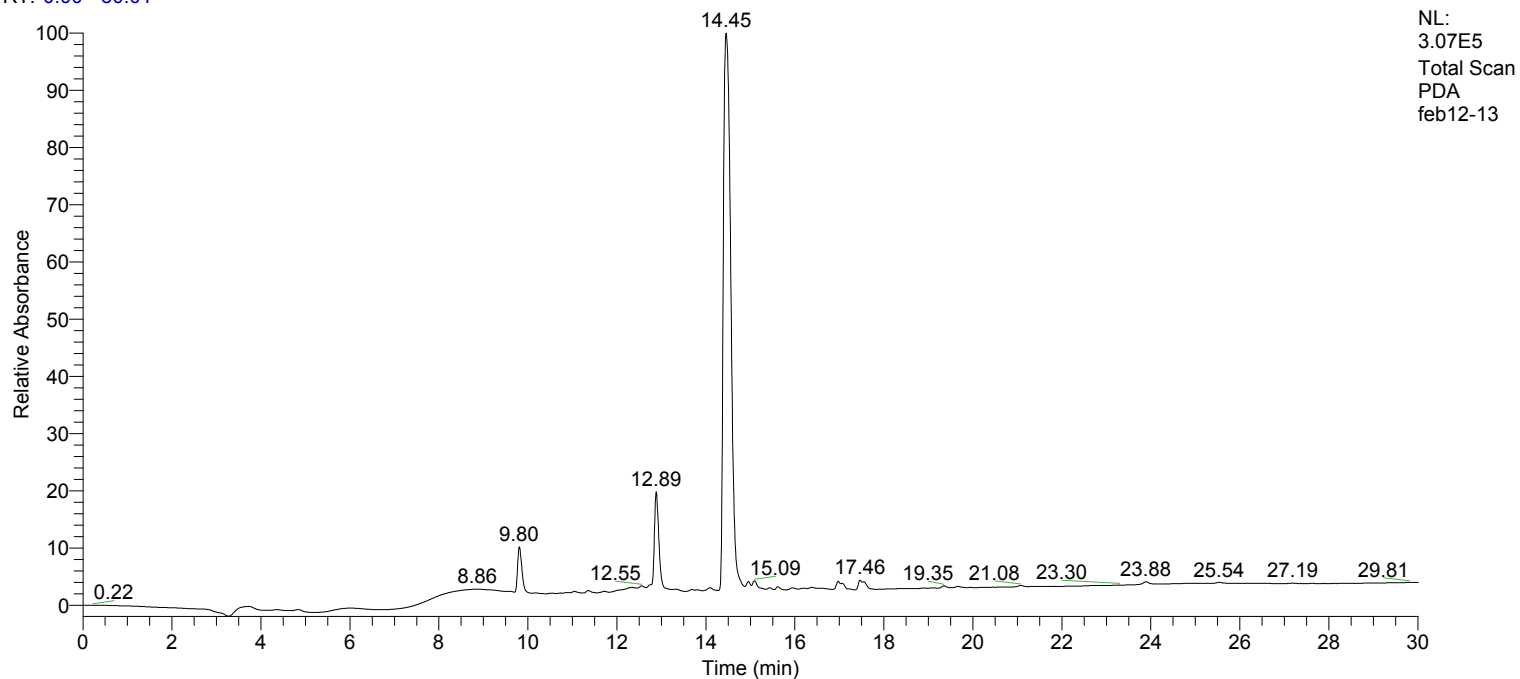
D:\My docs\Morten_2\HPLC\2012\jan12\250112\250112-58.lcd
 Acquired by : user
 Sample Name : C-30
 Sample ID : Tyr - diazotized
 Tray# : 1
 Vial # : 51
 Injection Volume : 10 uL
 Data File Name : 250112-58.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 250112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 27-Jan-12 23:05:51
 Data Processed : 05-Feb-12 19:16:32



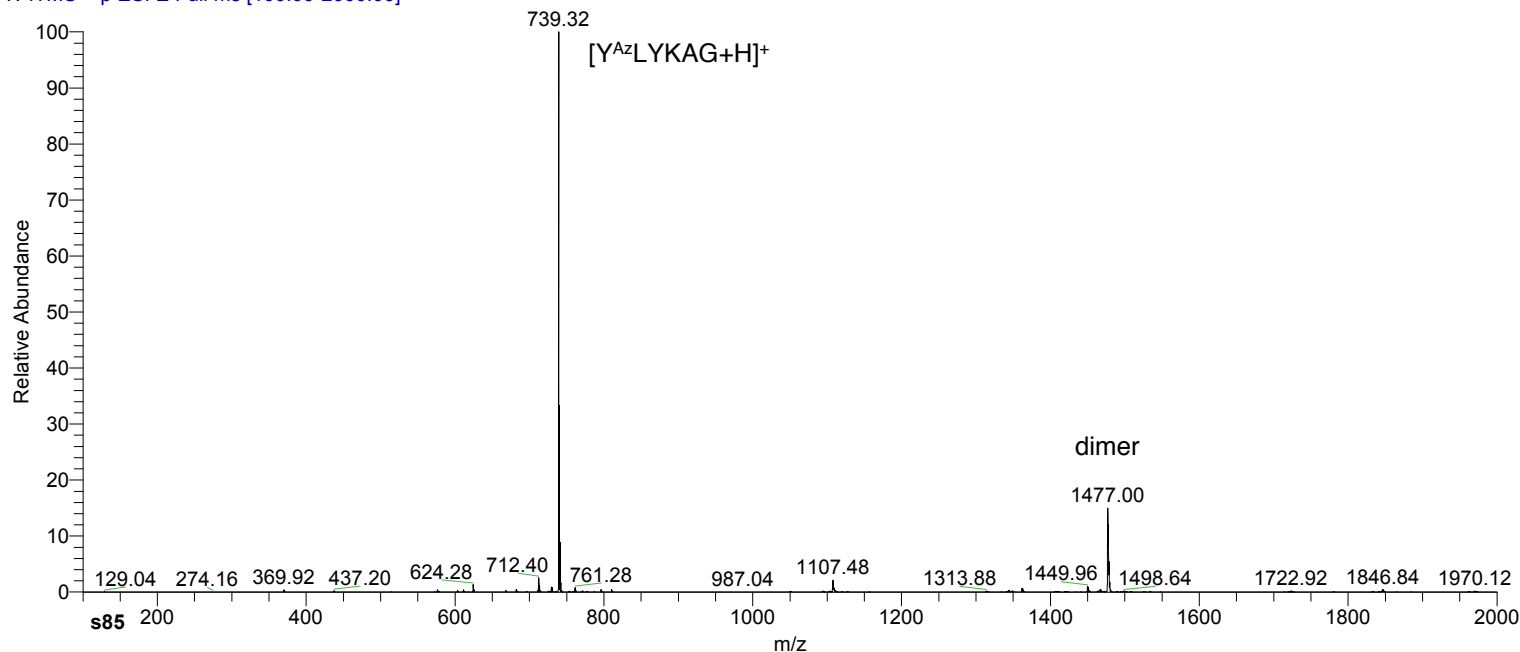
RT: 0.00 - 30.00



RT: 0.00 - 30.01



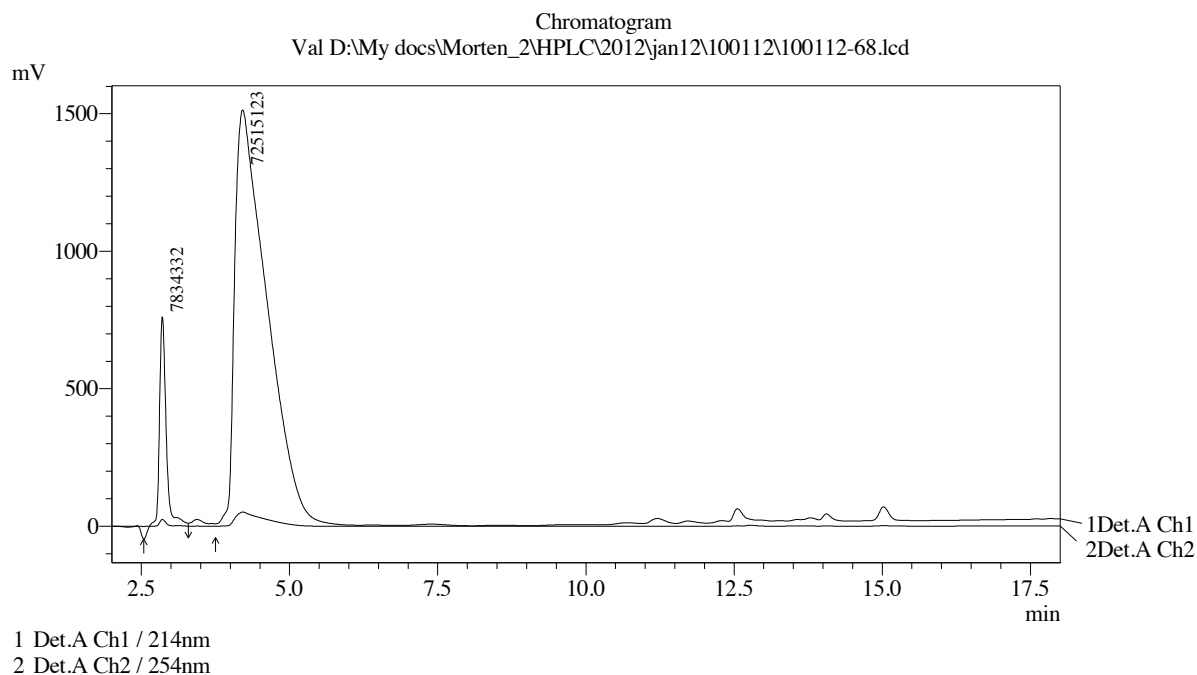
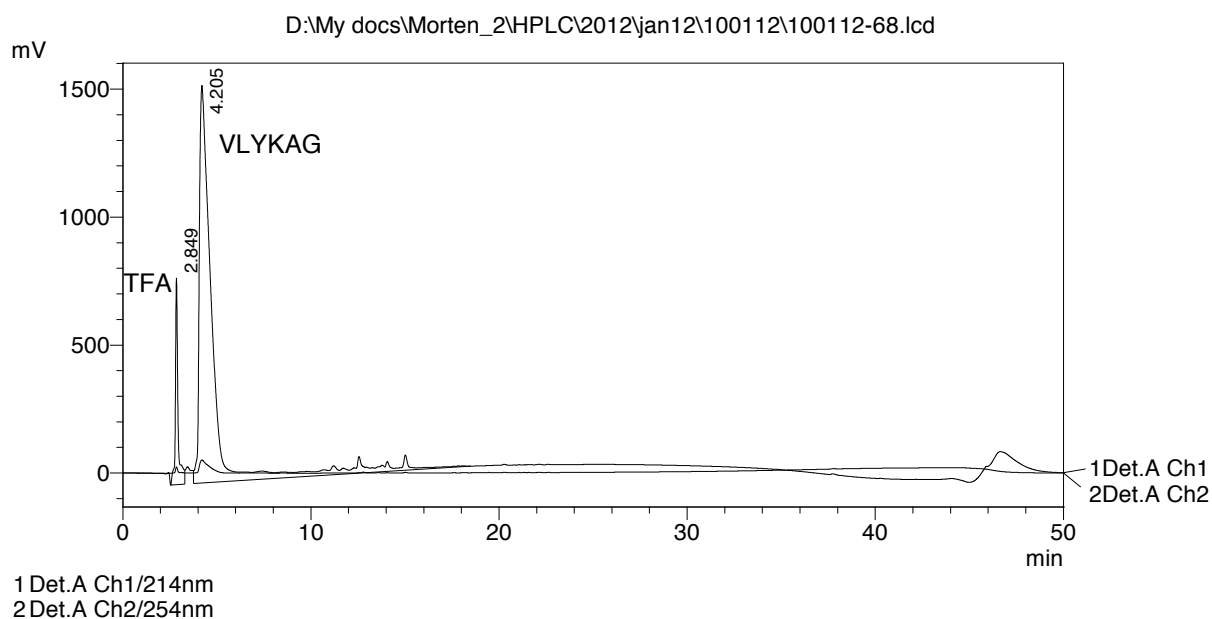
feb12-13 #769-810 RT: 14.43-15.11 AV: 42 NL: 7.72E4
T: ITMS + p ESI E Full ms [100.00-2000.00]



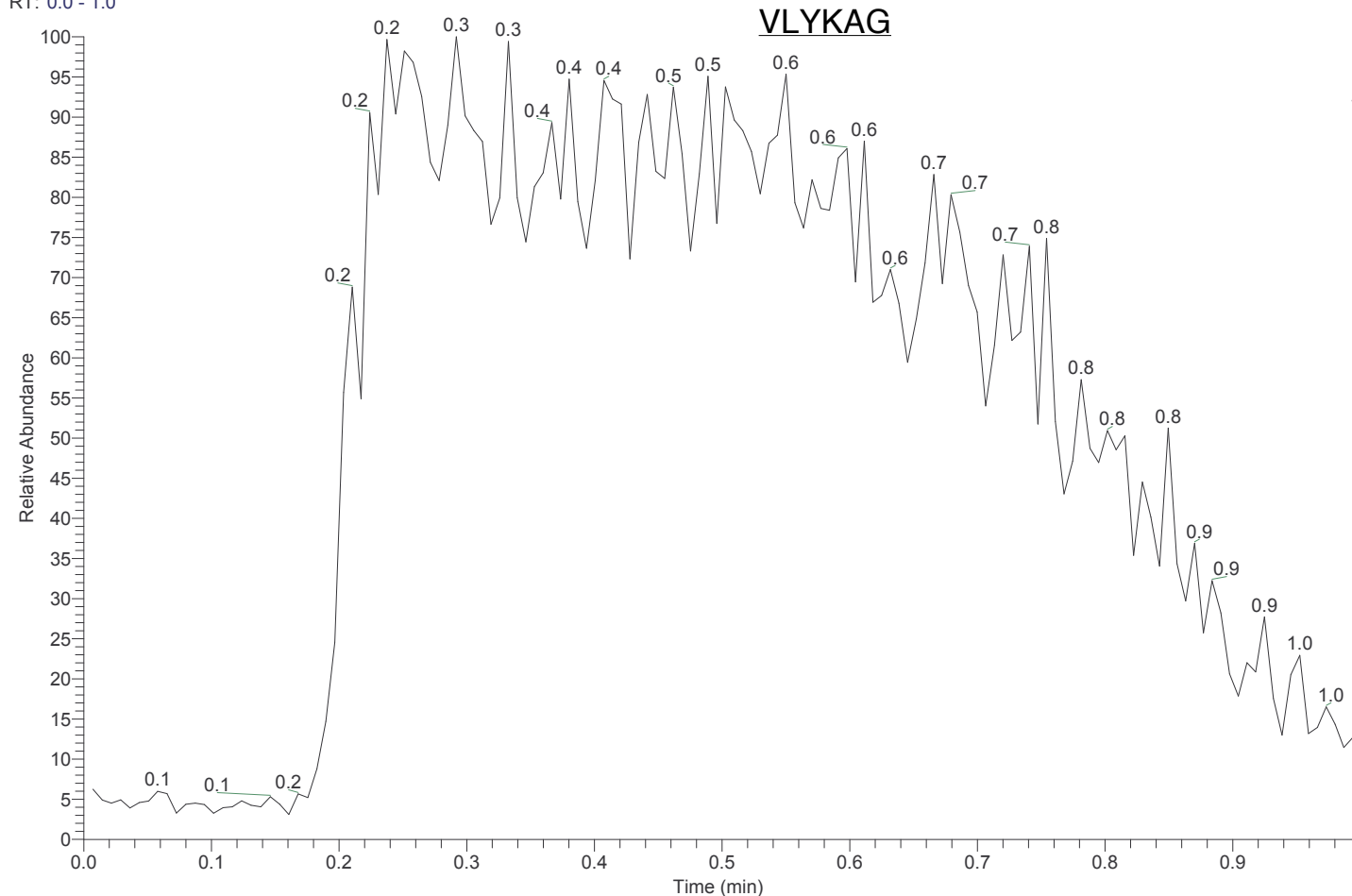
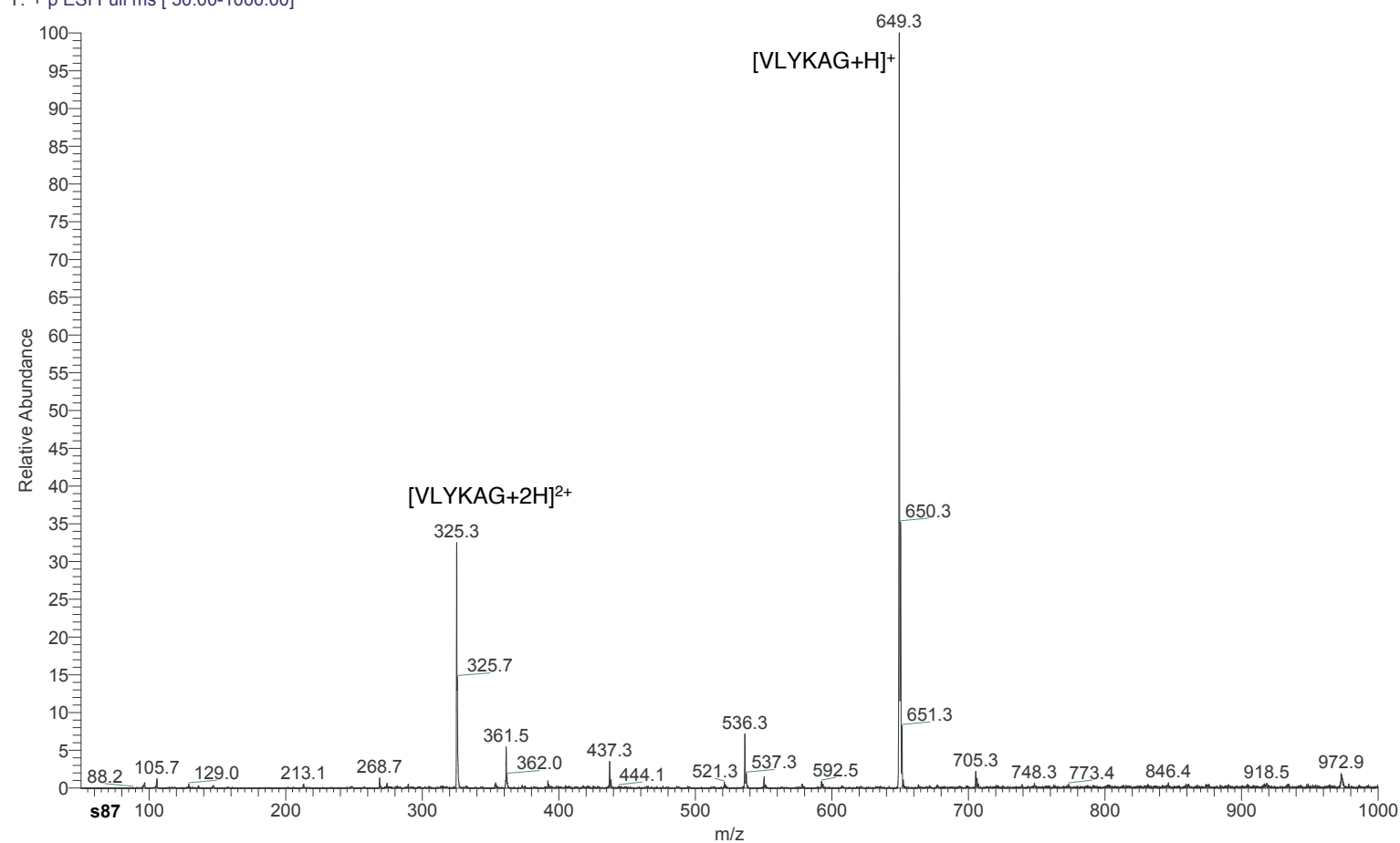
VLYKAG

==== Shimadzu LCsolution Analysis Report ====

D:\My docs\Morten_2\HPLC\2012\jan12\100112\100112-68.lcd
 Acquired by : user
 Sample Name : Val
 Sample ID : bef. diazotization
 Tray# : 1
 Vail # : 86
 Injection Volume : 10 uL
 Data File Name : 100112-68.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 100112.lcb
 Report File Name : ALYKAG-report.lcr
 Data Acquired : 13-Jan-12 6:44:23
 Data Processed : 13-Jan-12 7:34:27



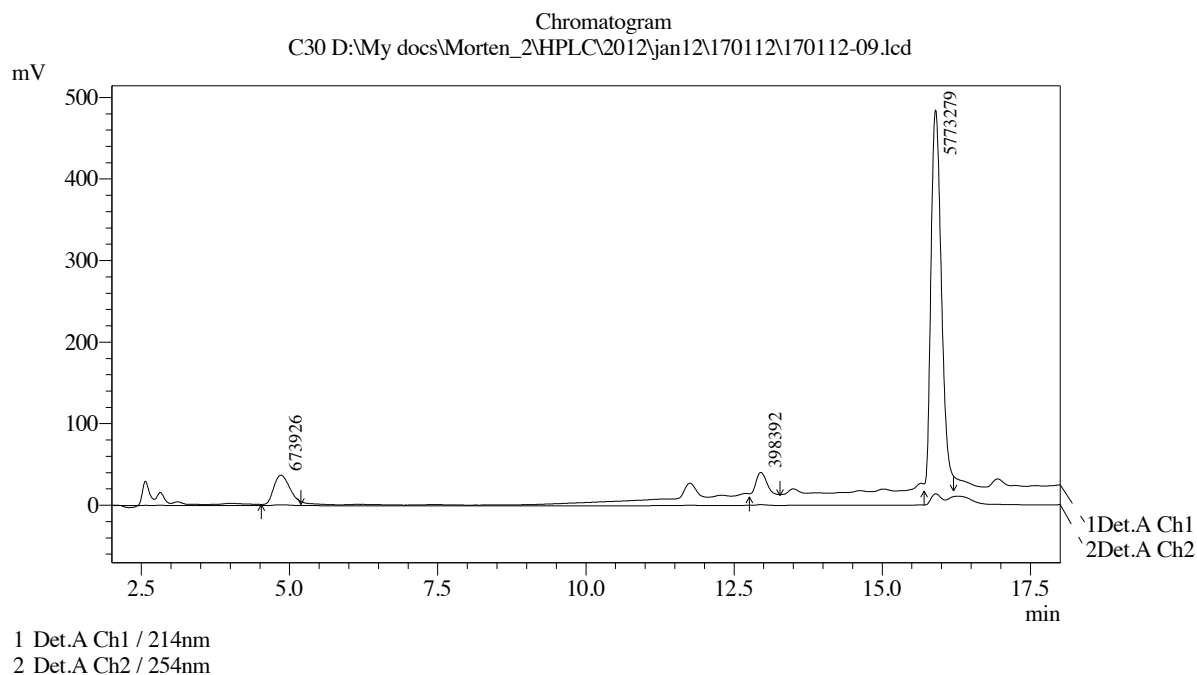
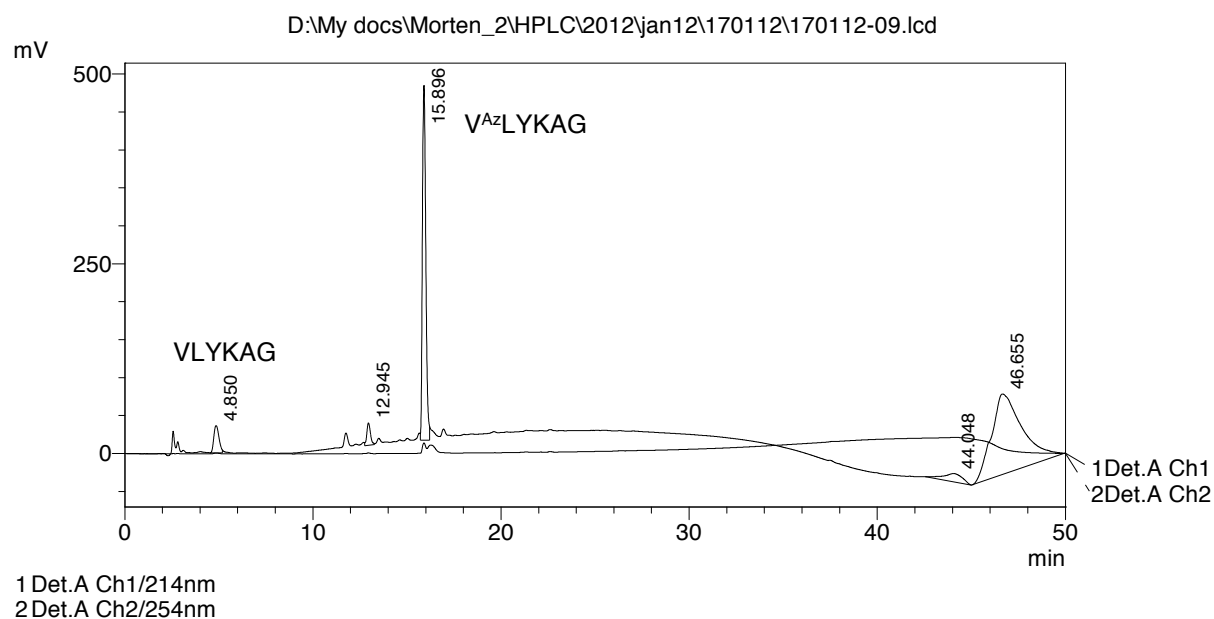
RT: 0.0 - 1.0

NL:
8.30E8
TIC F: MS
jan12-24jan12-24 #26-63 RT: 0.2-0.4 AV: 38 NL: 2.59E7
T: + p ESI Full ms [50.00-1000.00]

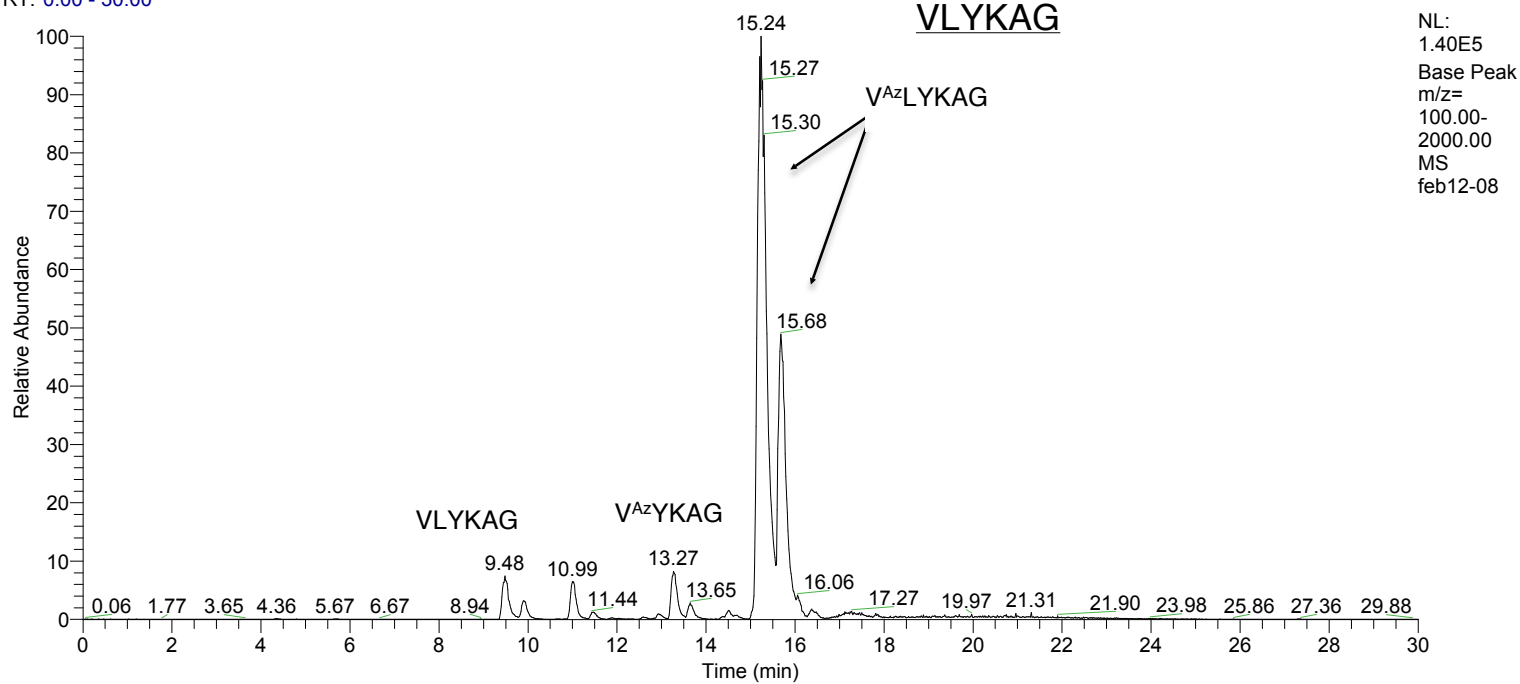
VLYKAG

==== Shimadzu LCsolution Analysis Report ====

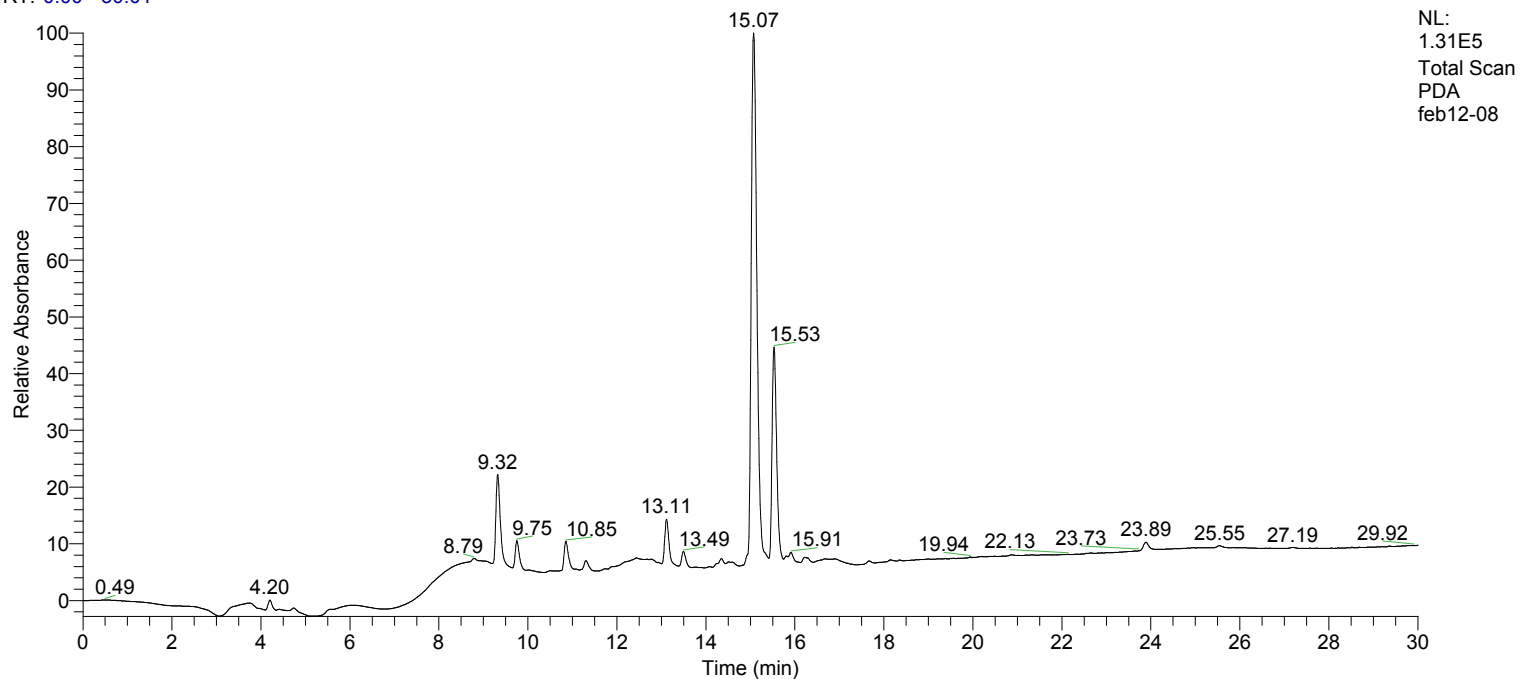
D:\My docs\Morten_2\HPLC\2012\jan12\170112\170112-09.lcd
 Acquired by : user
 Sample Name : C30
 Sample ID : Val - diazotized
 Tray# : 1
 Vail # : 11
 Injection Volume : 10 uL
 Data File Name : 170112-09.lcd
 Method File Name : gradient-anal.lcm
 Batch File Name : 170112.lcb
 Report File Name : Default.lcr
 Data Acquired : 17-Jan-12 22:27:43
 Data Processed : 18-Jan-12 11:29:34



RT: 0.00 - 30.00



RT: 0.00 - 30.01



feb12-08 #807-862 RT: 15.06-15.96 AV: 56 NL: 3.68E4
T: ITMS + p ESI E Full ms [100.00-2000.00]

