MS SERVICE OF SMALL MOLECULES

phone lines: 302, 481 e-mail: brezinova@uochb.cas.cz

MS SERVICE WHICH WE PROVIDE

- a confirmation of a molecular structure by determination of exact mass and assigning a suitable formula
- MALDI of high molecular compounds such as peptides, proteins, oligonucleotides and some polymers
- a help with an identification of a structure according to fragmentation by EI or CI and measuring MS/MS analysis
- LC-MS analysis providing a previous agreement
- GC-MS and LC-MS self-service - after training and making a schedule

THE MASS SPECTROMETRY SERVICE

Where can you find us and your results ?

- the basement, number of the door 33.
- results and MS spectra on Novell I:/MISC/MS/DATA/your surname

What should you do ?

- enrol at the sample book and bring a complete form

SAMPLE BOOK

sequence number	date	name	team code	sample label	method (source)	date of measuring	measured by

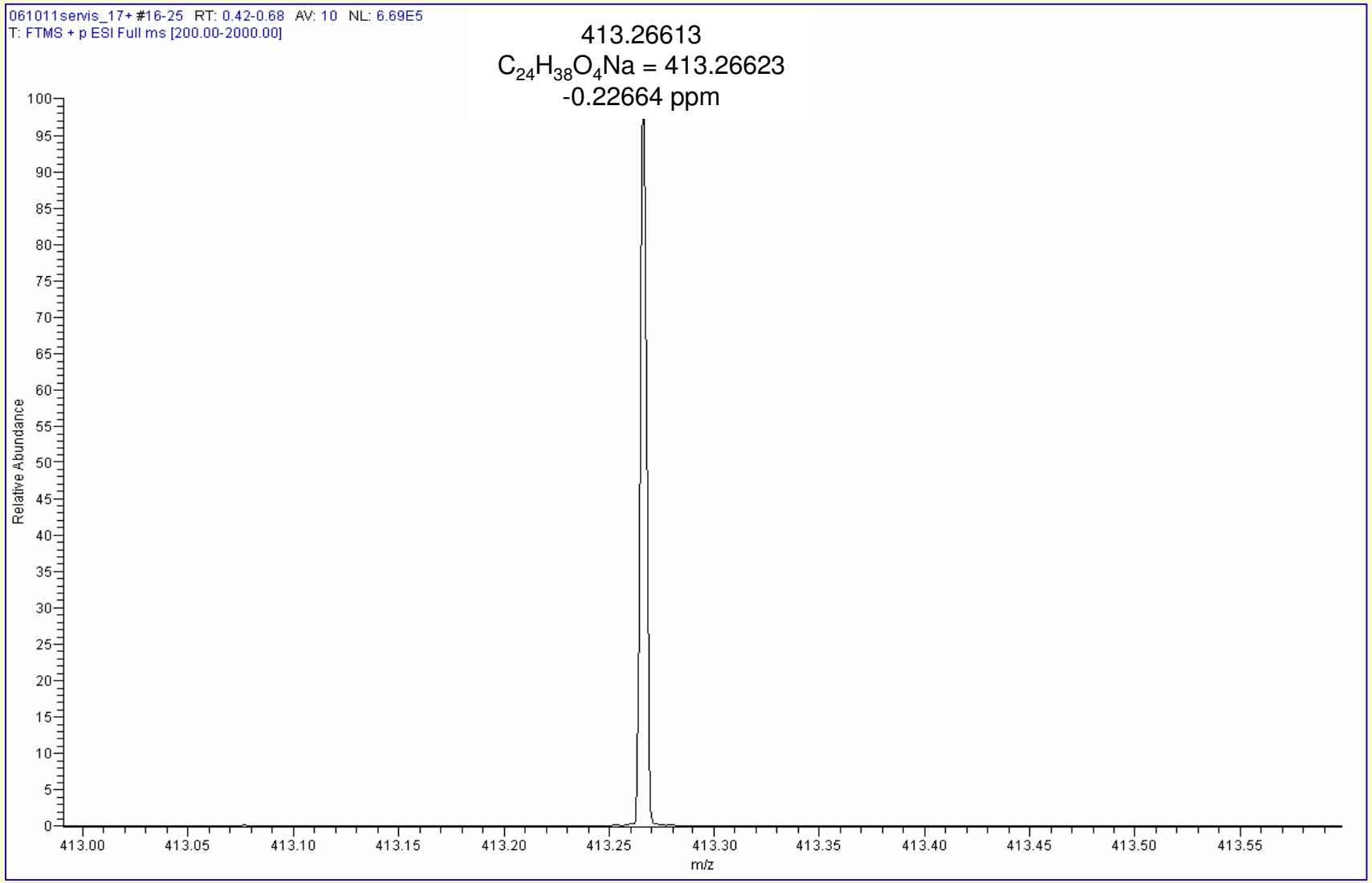
- each sample has to be written under its own sequence number separately

Skupina hmotnostní spektrometrie
Ústav organické chemie a biochemie AV ČR
Flemingovo nám. 2., 166 10 Praha 6
Tel.: 220183302, -481, -303 Fax: 220183583

ŽÁDANKA O MS ANALÝZU - SAMPLE SUBMISSION FORM

Jméno / Name:	Označení vzorku / Sample label:
Pracoviště / Department: Tel. linka / Phone:	**Pořadové číslo ! / Sequence number !:** Datum / Date:
Navrhovaný způsob ionizace / Suggested ionisation technique: ☐ **GC/EI or CI** ☐ **Direct probe EI** ☐ **ESI** ☐ **APCI** ☐ **MALDI**	
MS analýza / MS analysis: ☐ **LR - Hmotnostní spektrum - jednotkové rozlišení / Mass spectrum - unit resolution** ☐ **HR - Přesná hmotnost - vysoké rozlišení / Accurate mass - high resolution** ☐ **Peptidické mapování / Peptide mass fingerprinting** (Please contact MS staff)	
Předpokládaná struktura / Assumed structure:	Sumární vzorec / formula:
	Monoizotopická hmotnost / Monoisotopic mass:
	Rozpouštědla / Solvents: ☐ **H_2O** ☐ **MeOH** ☐ **ACN** ☐ **ACN/H_2O+FA** ☐ **$CHCl_3$** ☐ **Et_2O** ☐ **Hexan** ☐ **Aceton**
	Stabilita v kyselinách/ Stability in acids: ☐ **Ano / Yes** ☐ **Ne / No**
	Stabilita v bázích/ Stability in bases: ☐ **Ano / Yes** ☐ **Ne / No**
Poznámky / Notes:	

HOW DOES THE LR and HR ESI SPECTRA LOOK LIKE



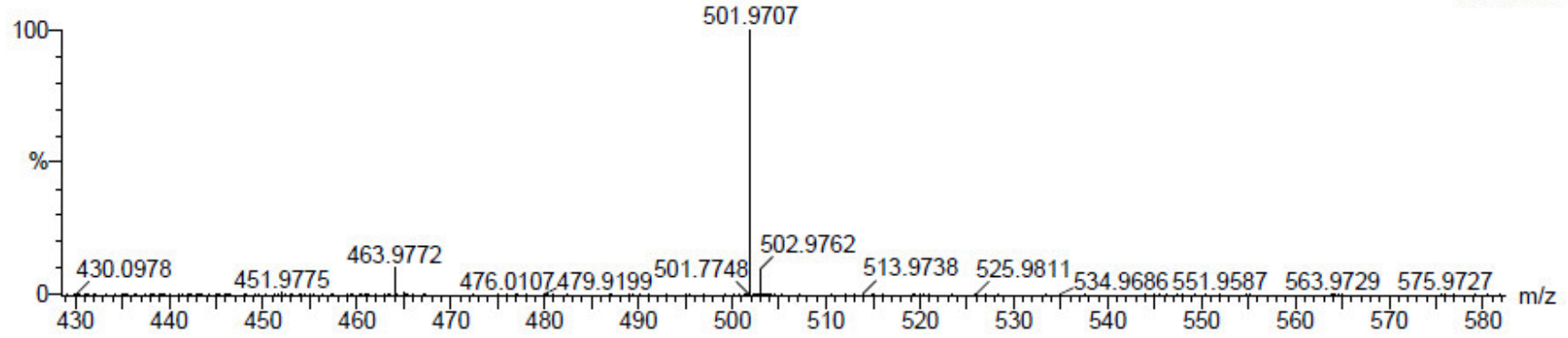
HOW DOES THE LR and HR EI SPECTRA LOOK LIKE

Elemental Composition Report **Page 1**

Single Mass Analysis (displaying only valid results)
Tolerance = 10.0 PPM / DBE: min = -100.0, max = 1000.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
17 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 3-15 H: 0-1 N: 1-1 F: 0-30
heptacosa
heptacosa 162 (2.701) Cm (160:191)

TOF MS EI+
8.82e+004



Minimum:				-100.0		
Maximum:		5.0	10.0	1000.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
501.9707	501.9711	-0.4	-0.8	0.5	3495.1	C9 N F20

SAMPLES

Notes

- if the samples are either toxic, unstable or moisture, light and high temperature sensitive you have to put it down in the form.

- if you have a small amount of sample, you can certainly get it back, providing it is written in the form.

Solid samples:

It is better to submit a solid sample (100 µg - 1.0 mg). Please do not forget mention a suitable solvent.

Desolved samples:

Please use only solvents for LC/MS or distilled solvents.

- samples should be fully dissolved.

All instruments are usually capable of detecting at least 1 µg/mL.

However, it is good habit to submit around 1 mg/mL.

RECOMMENDED SOLVENTS

Which solvent can I use? - it depends on the type of ionization and whether you want to use GC chromatography before MS or not.

- **ESI:** the most preferred solvents are **MeOH**, **H_2O** and **ACN** - a solution should be free from nonvolatile buffers and and another additives.

  $CHCl_3$ and **acetone** are also acceptable in mixture with MeOH or ACN.

- **APCI: MeOH, ACN, IPA, $CHCl_3$, EtAc, EtOH** and **acetone**
- **EI/CI with direct probe:** solid samples or dissolved in volatile solvents.
- **EI/CI coupled with GC:** hexan, ether, $CHCl_3$, CH_2Cl_2, MeOH, EtOH. The samples for GC/MS must be free from strong acid, bases and oxidizing compounds.
- **MALDI:** in a solution free from nonvolatile buffers, solvents and surfactants

! Please, avoid solvents with high boiling points (DMSO, DMF) !

USUAL CONTAMINANTS

- **Phthalates** - from plastics, contaminated solvents

  diisobutylphthalate (masses EI: 149, 205, 223, 278, ESI: 279, 301)

  diisooctylphthalate (masses EI: 149, 167, 279, 390, ESI: 391, 413)

- **Antioxidants** - from plastics : irganox, irgafos

  (the most common masses EI: 316 ,591, 647, 642, ESI: 663, 685)

- **Polysiloxans** - from silicone rubber, teflon lined caps from vials

  (the most common masses EI: 73, 147, 221, 295, 355, ESI: 297, 371, 445, 519)

- **PEGs** - extracted polymer from teflon/silicon septum (+44 series)

- **Amides** - from plastics - oleamide (ESI: 282), stearamide (ESI: 284), erucamide (ESI: 338)

- **Detergents** - Triton X-100

- **Fatty acids** - palmitic and oleic acid from skin (masses negESI : 255, 283)

SALTS, BUFFERS and ANOTHER ADDITIVES

- **volatile** - *TFA, FA, acetic acid, ammonium acetate, ammonium formate etc.*

- can be used in lower concentrations (up to 10mM)

- **nonvolatile** - *phosphate, sulfate buffers, SDS, CHAPS, TRIS, HEPES etc.*

- should not be used because of decreasing signal, salt clusters creation and unbearable contamination of mass spectrometers

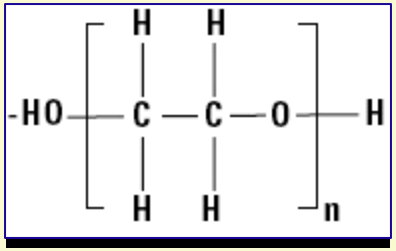
	MW	MALDI	MALDI	ESI	ESI
Surfactant/ buffer/ salt	(g/mol)	(mM)	(wt.%)	(mM)	(wt.%)
TRIS	121	100	1	n.a.	n.a.
HEPES	238	100	2,4	n.a.	n.a.
Urea	60	500	3	n.a.	n.a.
Dithiotreitol	154	500	7,7	n.a.	n.a.
Guanidine	96	250	2,4	n.a.	n.a.
Glycerol	92	130	1,2	n.a.	n.a.
Triton X-100	628	1,6	0,1	1,6	0,1
Tween20	1228	n.a.	n.a.	0,81	0,1
SDS	288	0,35	0,01	0,34	0,01
CHAPS	615	0,16	0,01	1,6	0,1
Sodium Azide	65	15	0,1	3,1	0,02
NaCl	58	50	0,29	n.a.	n.a.
Sodium Acetate	82	50	0,41	n.a.	n.a.
TFA	114	n.a.	n.a.	4,4	0,05
$NaHPO_4$	120	10	0,12	10	0,12

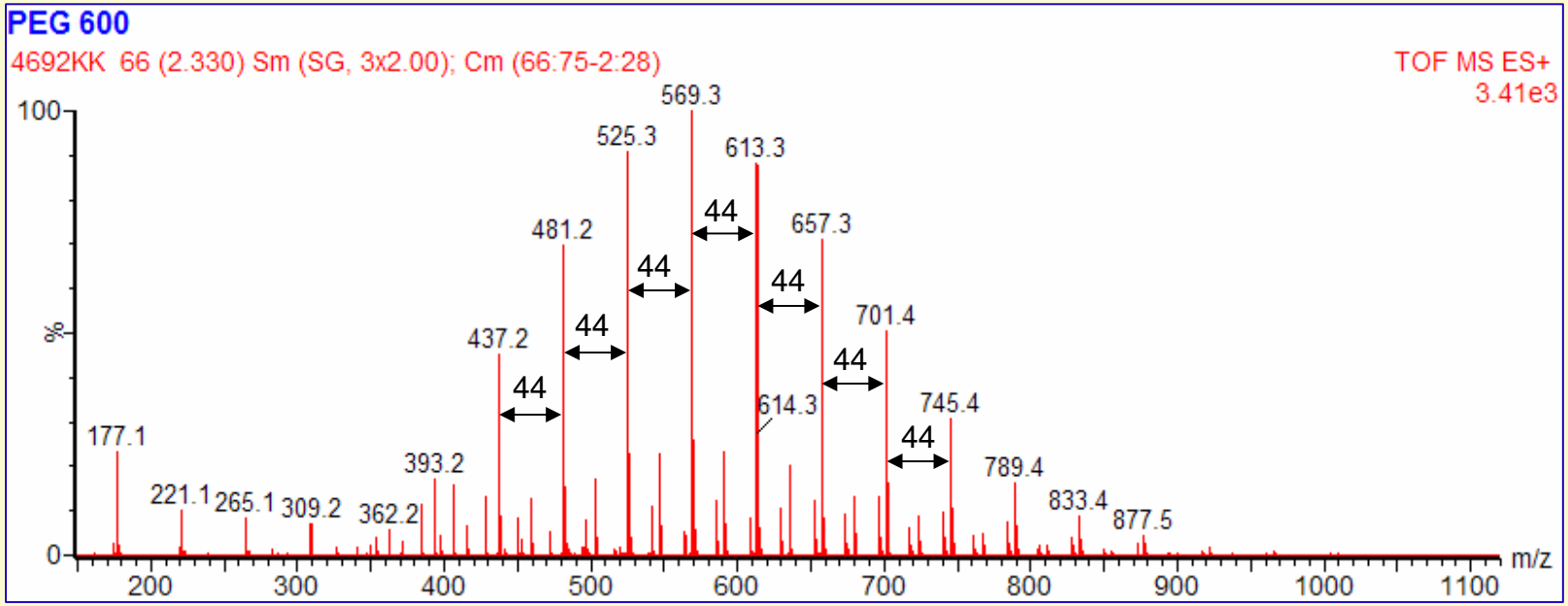
http://masspec.scripps.edu/services/proteomics/saltol.php

ADDUCTS AND CLUSTER PEAKS IN +/-ESI

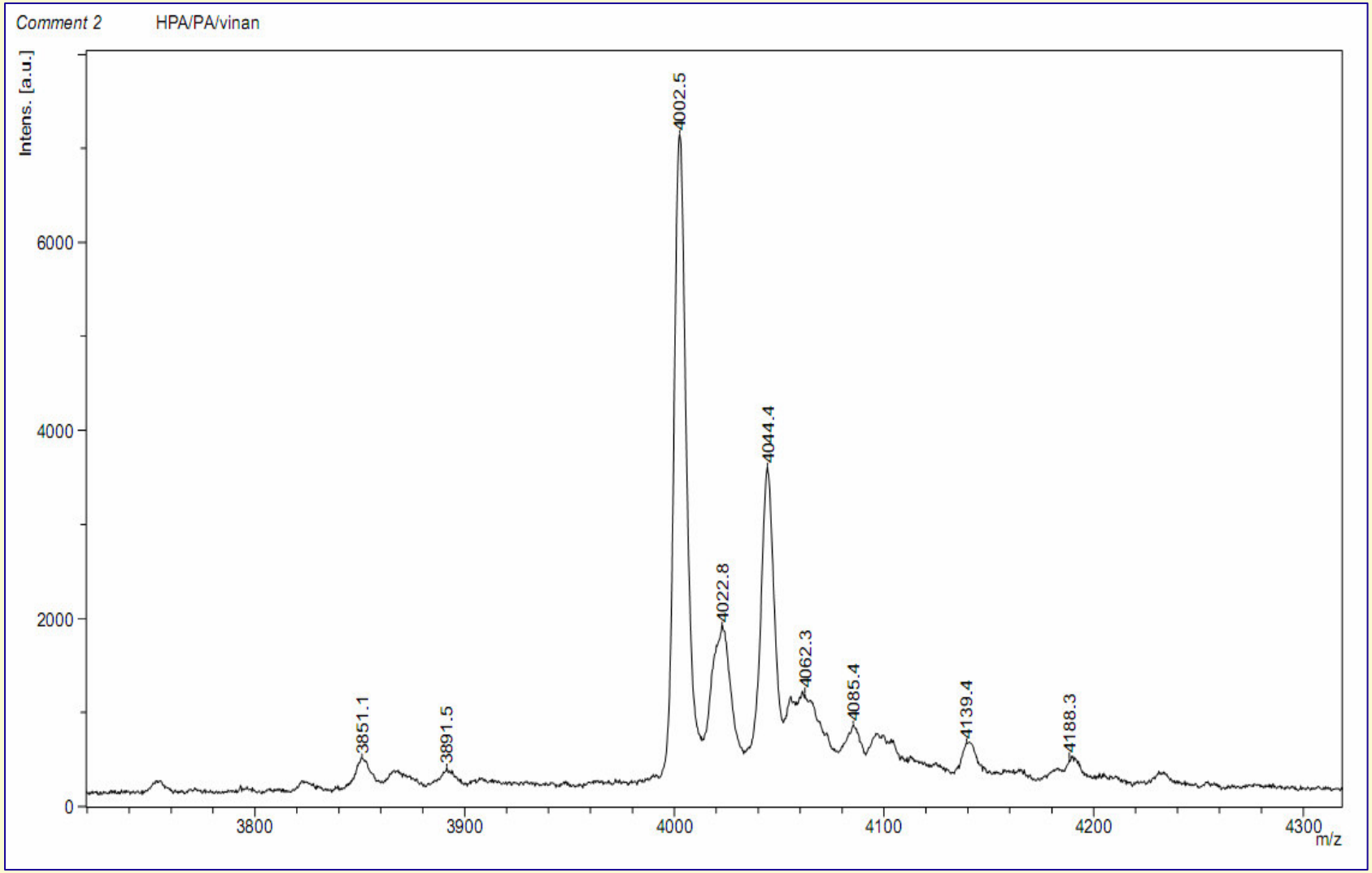
- +23n….sodium adducts….+ESI
- +32n….methanol adducts….+ESI
- +39n….potassium adducts….+ESI
- +41n….acetonitrile adducts….+ESI
- +44n…..polyethylene glycol related (-CH_2CH_2O-)n....+ESI
- +53n…..ammonium chloride adducts (NH_4Cl)….+ESI
- +63n…..ammonium formate adducts ($HCOONH_4$)….+ESI
- +68n…. sodium formate adducts (HCOONa)….+/-ESI
- +74n…..polysiloxanes ($Si(CH_3)_2O$)….+/- ESI
- +77n…..ammonium acetate salts (CH_3COONH_4)….+ESI
- +82n…..sodium acetate adducts (CH_3COONa)....+/-ESI
- +114n ...TFA (trifluoroacetic acid) adducts (CF_3COOH)….-ESI
- +136n….sodium TFA (trifluoroacetic acid) adducts (CF_3COONa)….+/-ESI
- +288n …SDS (sodium dodecylsulfate) adducts….-ESI

ESI SPECTRUM OF POLYETHYLENE GLYCOL 600

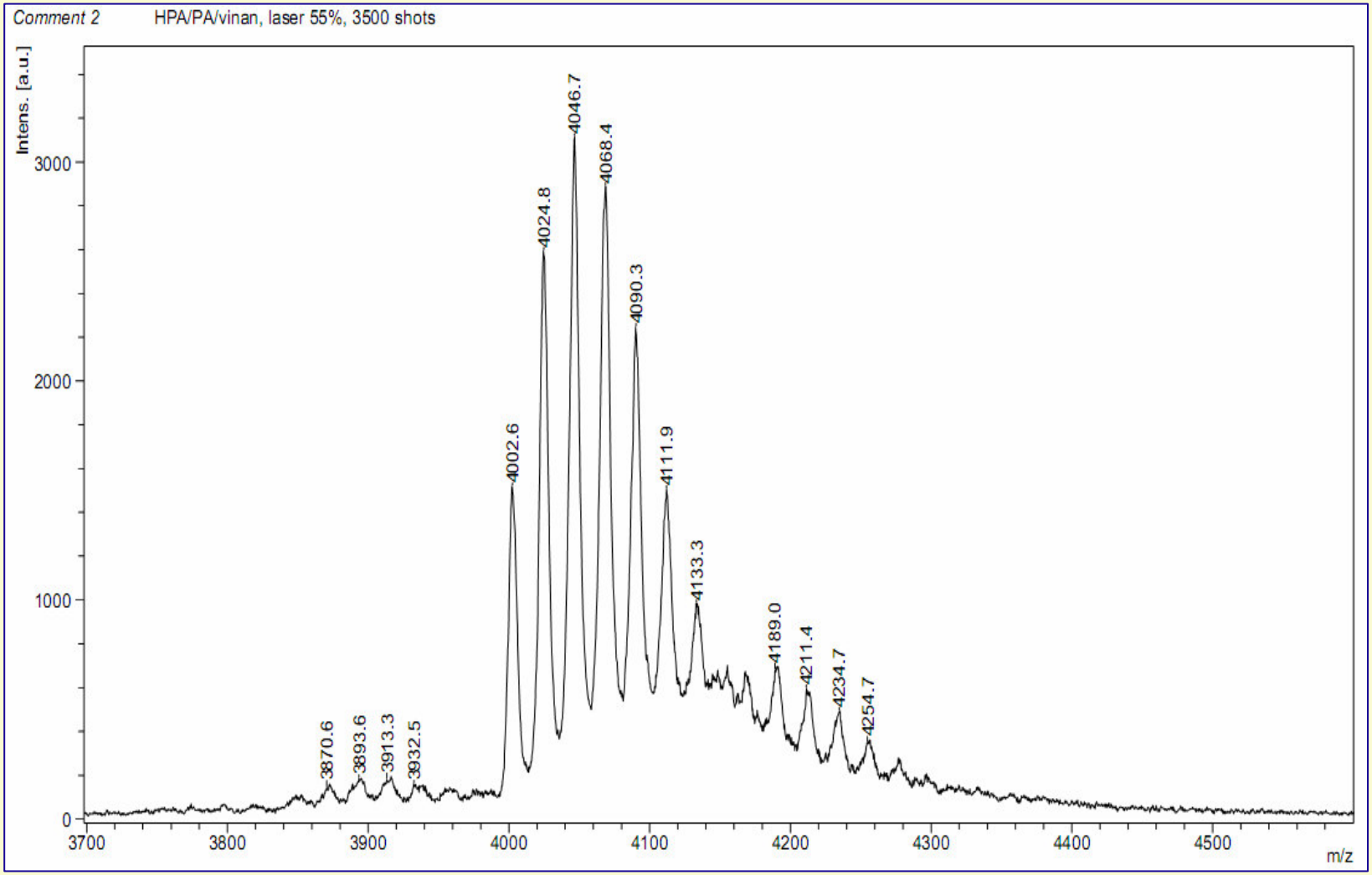




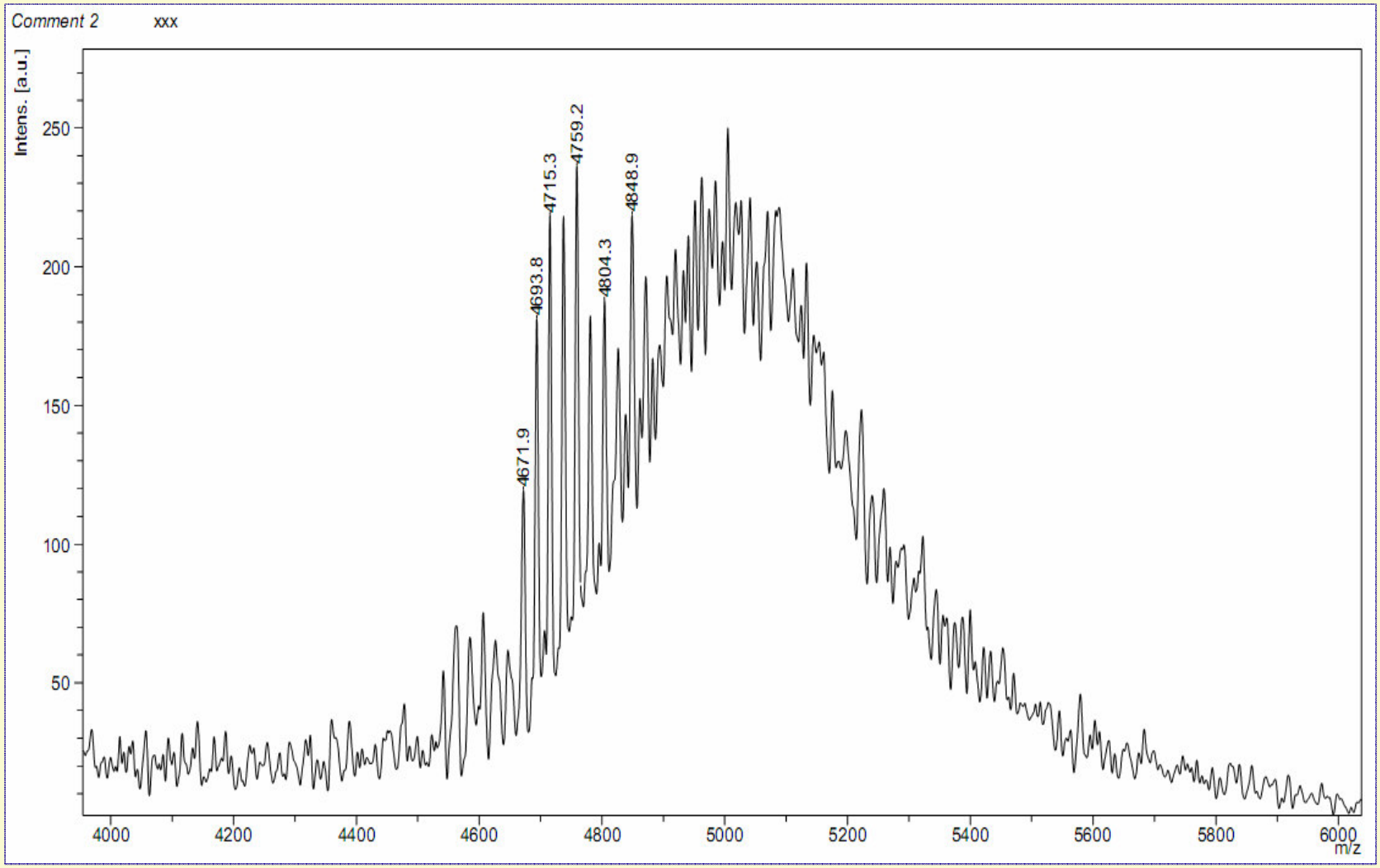
MALDI OF CLEAN SAMPLE



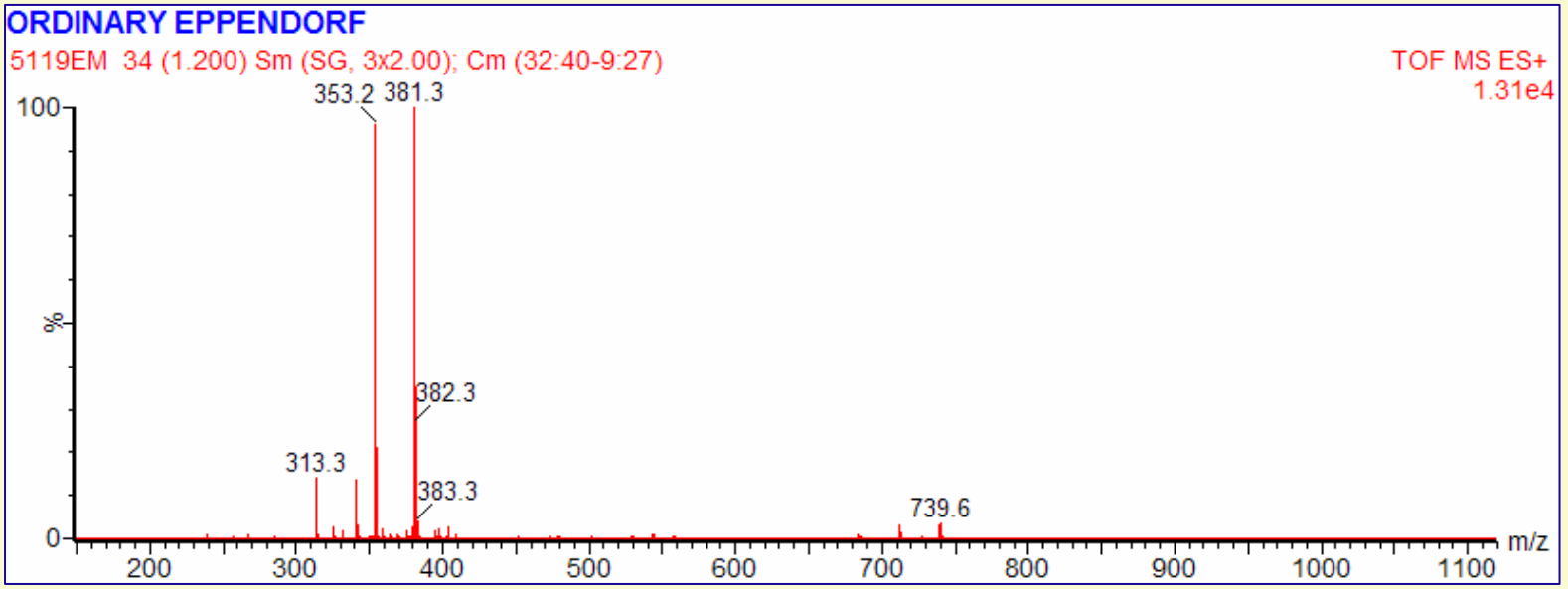
MALDI OF SAMPLE WHICH IS SLIGHTLY CONTAMINATED WITH SALTS

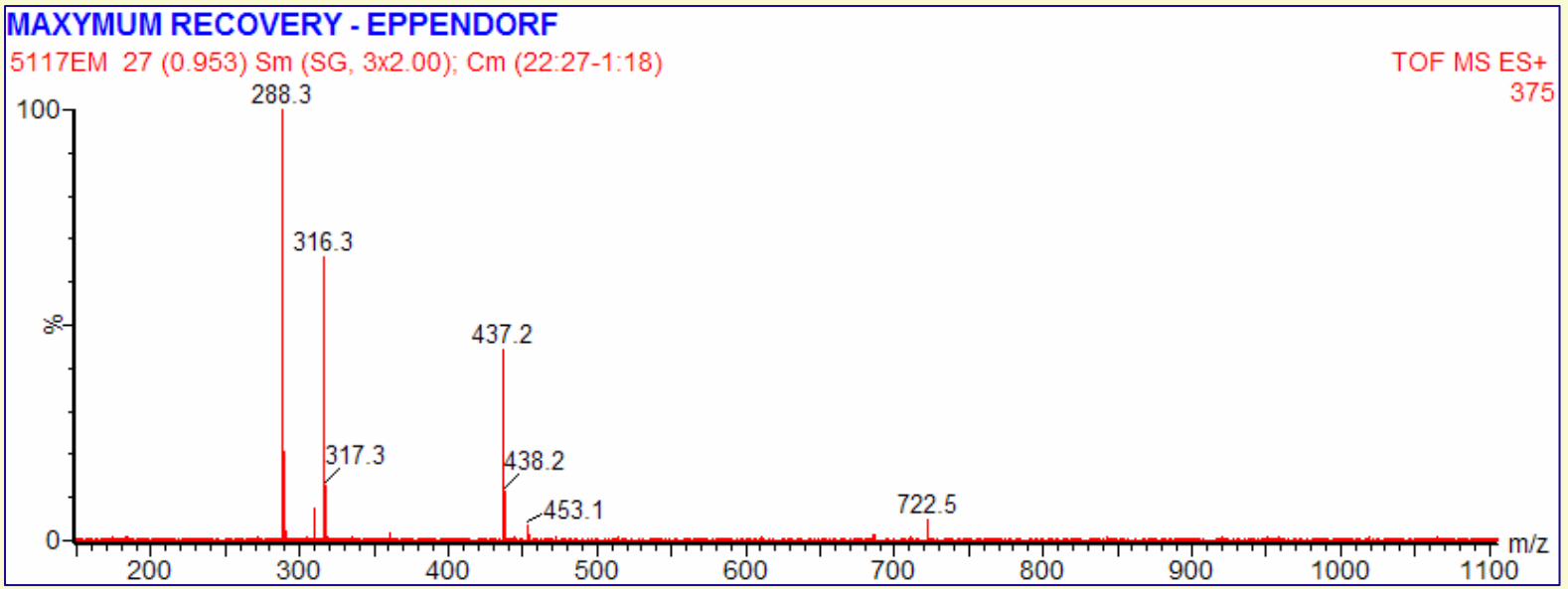


MALDI OF SAMPLE WHICH IS HIGHLY CONTAMINATED WITH SALTS

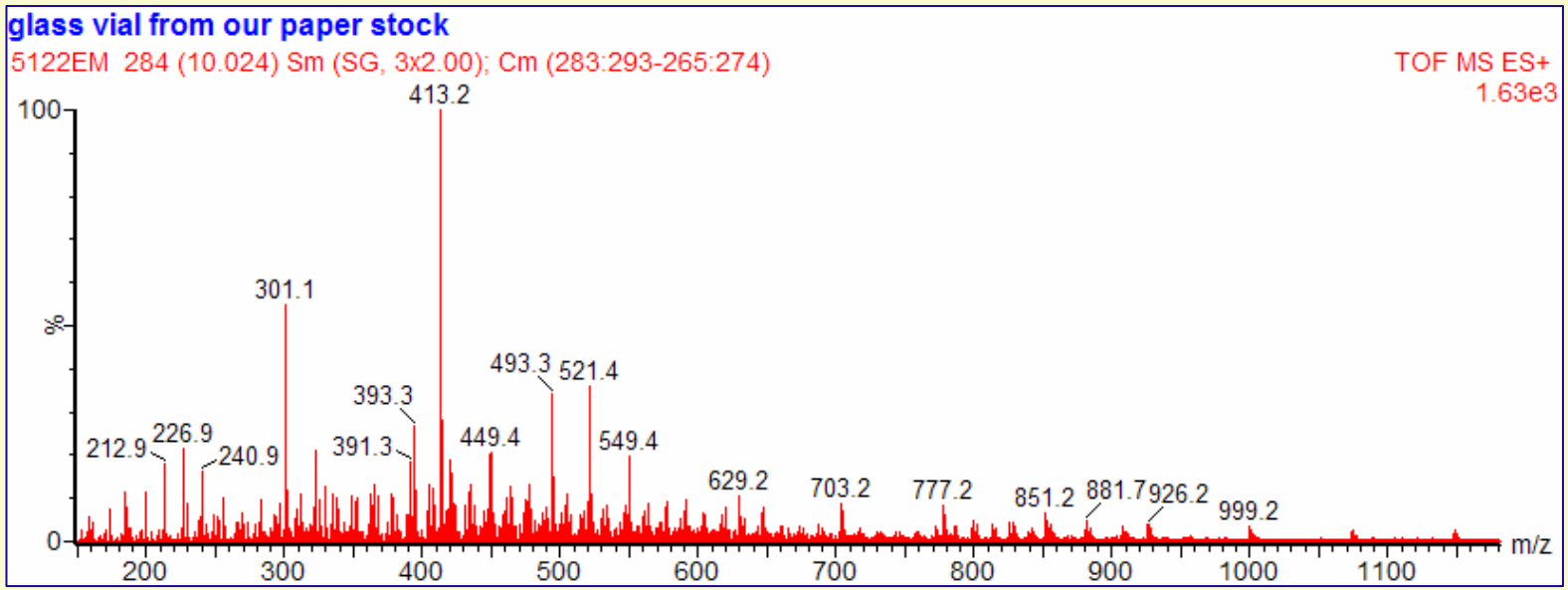


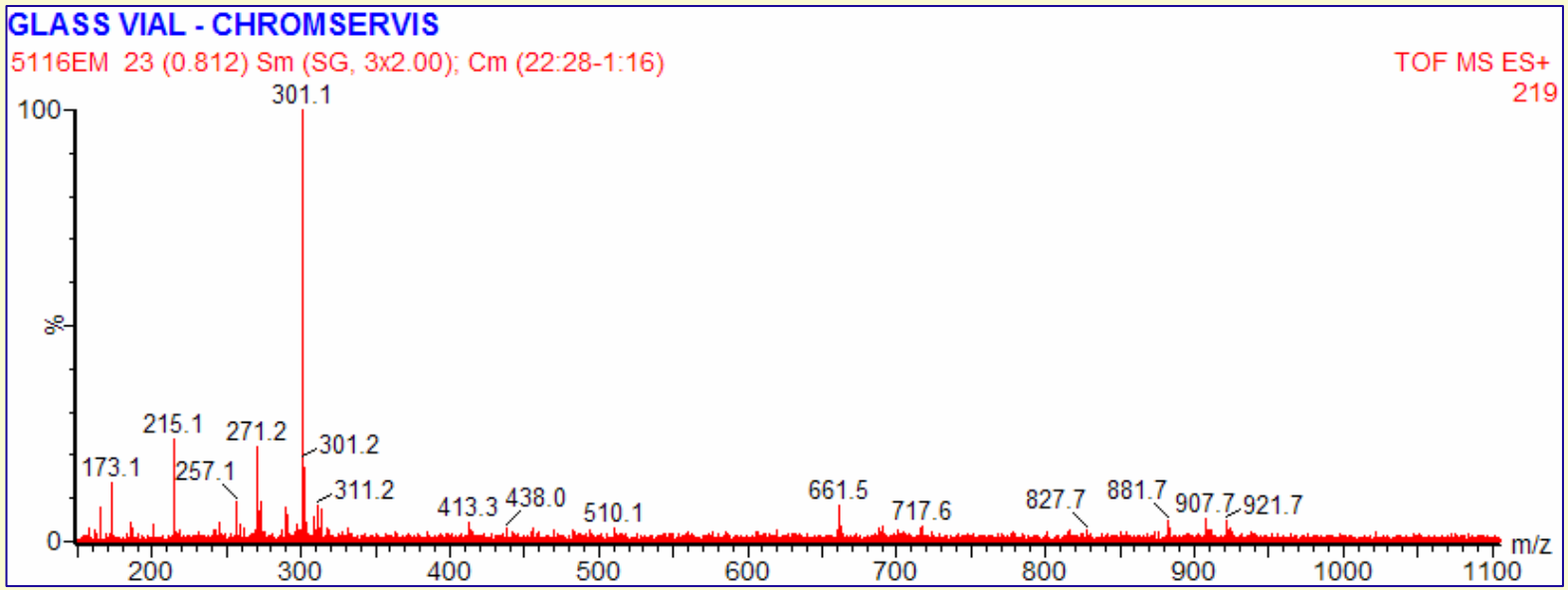
MeOH FROM EPPENDORFS





MeOH FROM GLASS VIALS





VIALS AND EPPENDORFS

Recommendation:

- glass vials + caps and septa PTFE/silicon, 2ml: *Chromservis s.r.o., P.N.C4000* for organic solvents
- eppendorfs MAXYMUM RECOVERY, 1,5 ml: *P-LAB a.s., P.N. U344701* for polar solvents

Thank you for your attention