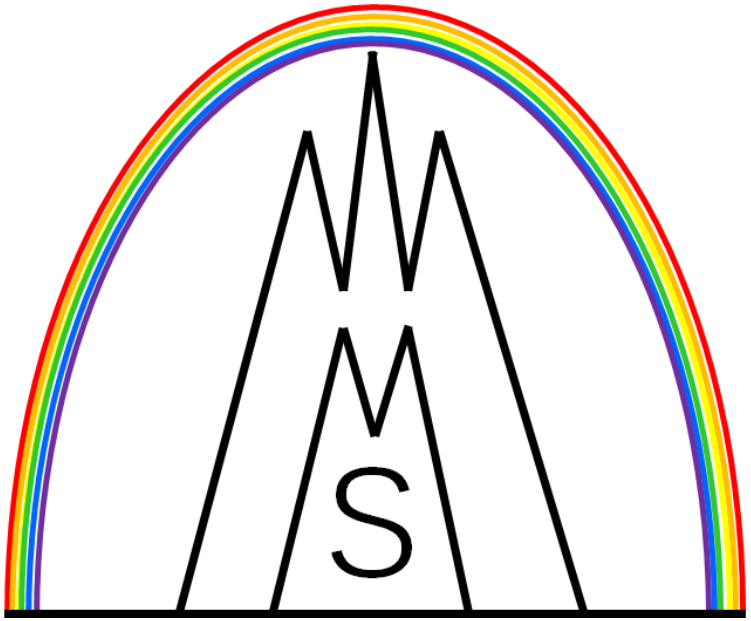
MS service of small molecules



Who we are

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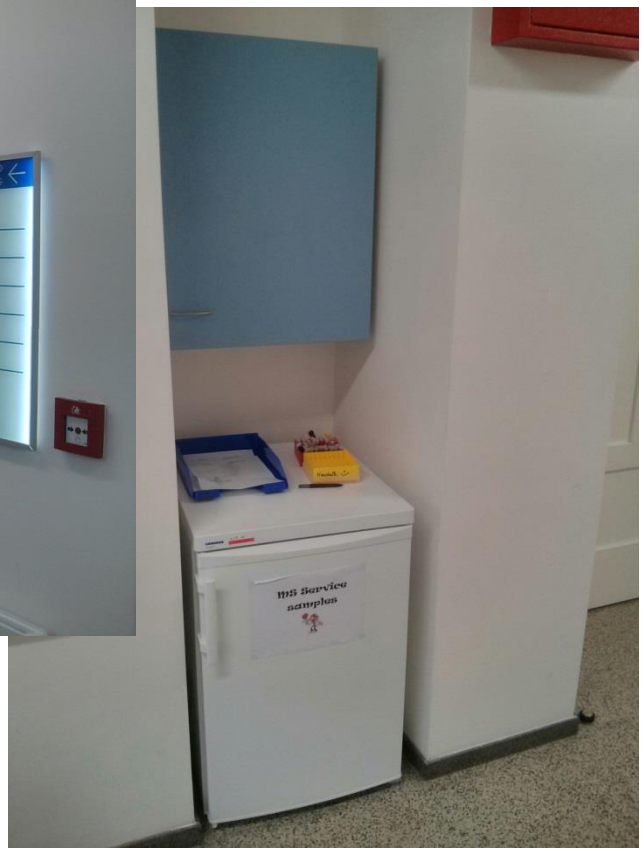
And where can you find us?

Building A, SE-wing, 1st floor



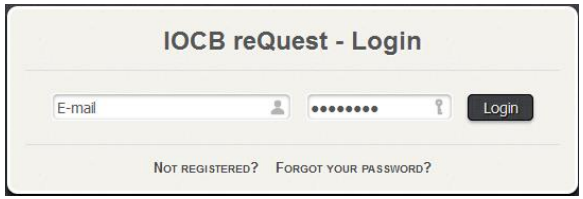
http://www.uochb.cz/web/structure/200.html

What can we offer?

- Mass spectra of small organic molecules – LRMS
- Accurate masses of small organic molecules – HRMS
- MALDI of high molecular structures (e.g. peptides, oligonucleotides, some polymers – up to 10kDa)
- GC-MS HR analysis and fragmentation of small organic molecules using EI/CI ionization technique
- A help to solve structural problem using MS/MS analysis
- LC-MS analysis of small organic molecules after previous consultation and agreement
- GC-MS self-service – after training and making schedule (Vrkoslav V., tel. 347)

IOCB reQuest

https://request.uochb.cas.cz - login (or registration first)



New request – what should I pay attention to (besides other things)

- name of the sample should correspond with the label on the vial
- correct formula
- correct mass (monoisotopic preferred – e.g. second number in the lower panel in ChemSketch)
- correct structure
- solubility of the sample, handling, sample return, notes and special requests
- the more we know the better and faster our work could be

Bring your sample with printed form to us. Results (pdf or msd form) are uploaded to the request page and you are notified via email.

Samples

- we prefer solid samples with the recommendation of suitable solvents
- glass vials if it’s possible
- we prefer clean samples, free of contaminants, salts, detergents, etc.
- visible sample in the vial (if not we would appreciate mark where it is)
- if your sample is temperature sensitive, please put it yourself into refrigerator or freezer which is located in our hall

ESI/APCI ionization technique

What can be seen in the spectrum most often.

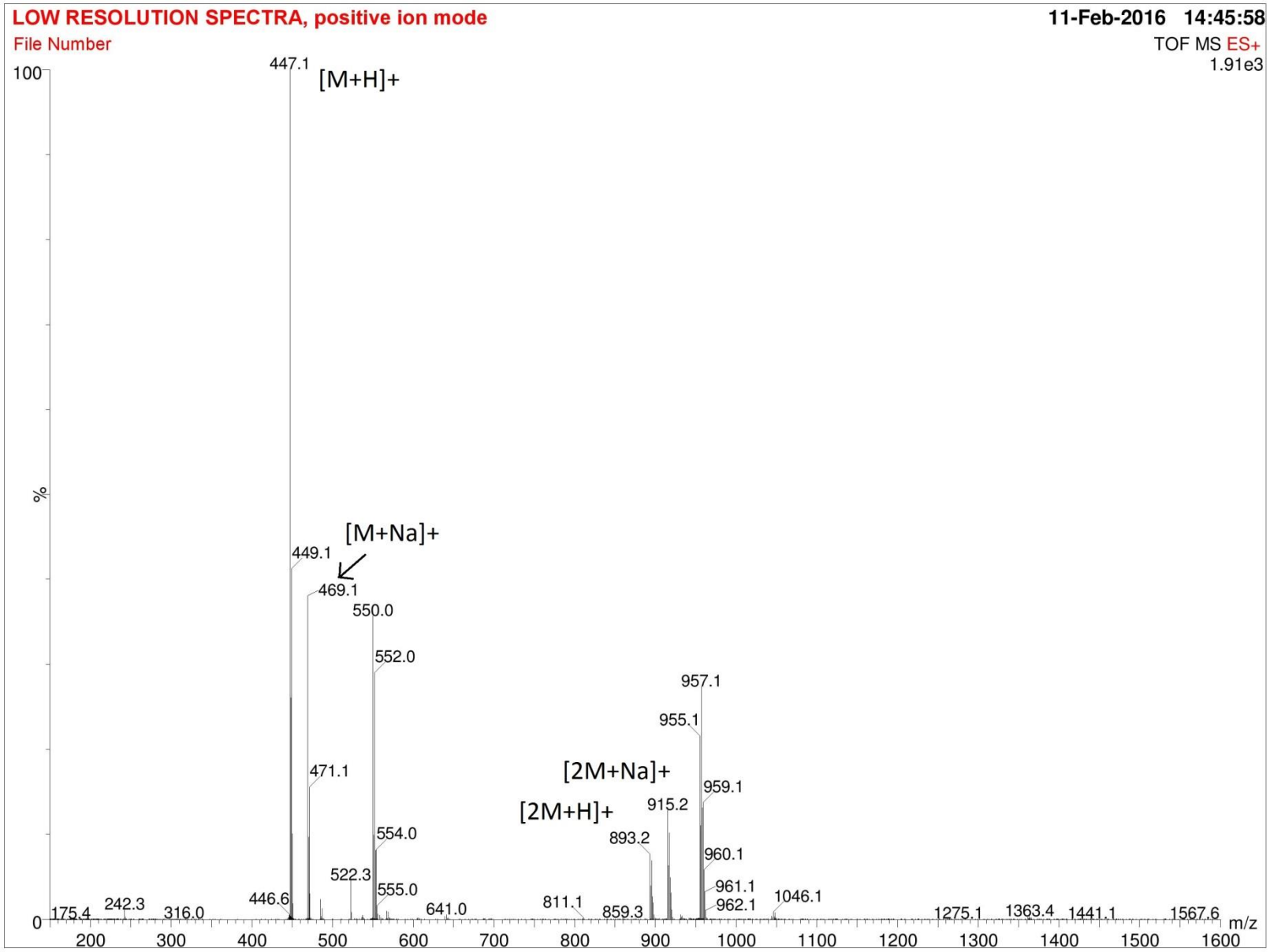
ESI

- ion [M+H]+ … mostly, usually accompanied by [M+Na]+ ion (+23 Da)
- ion [2M+H]+ or [2M+Na]+ …. dimer (there could be bigger multimer in some cases)
- multiple charge 2+, 3+, 4+ ions
- if there is -OH group in the molecule there could be loss of mentioned group = ion [M-17]+
- there also could be adduct with MeOH (M+H+32), MeCN (M+H+41) and/or other molecules of solvents

APCI

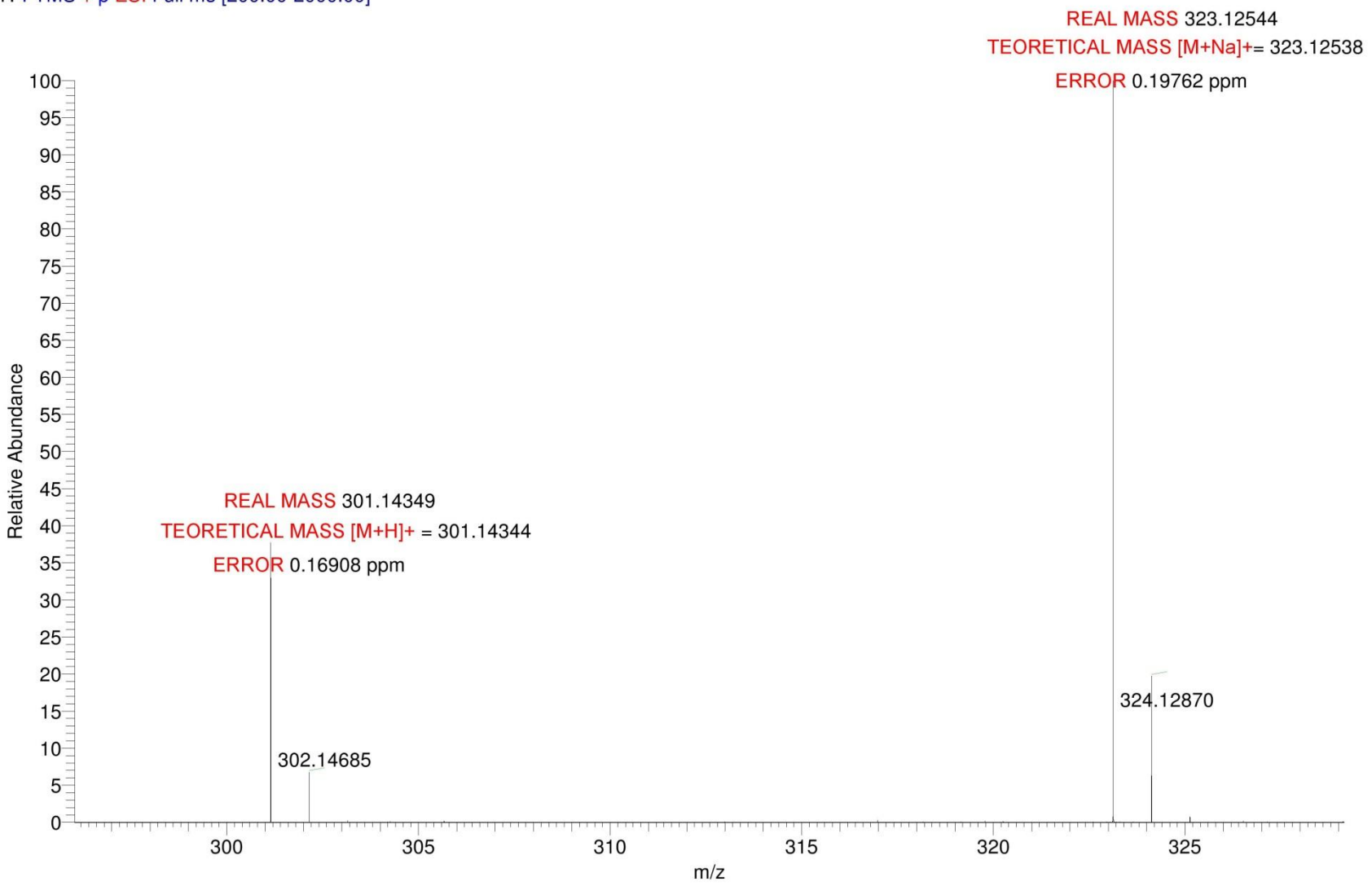
- ions previously mentioned but no salts adducts (they suppress ionization!)
- ion M+* in APCI, often MeCN (M+H+41)

- <u>interpretation of the ESI/APCI spectrum could by quite challenging and difficult</u>


LOW RESOLUTION SPECTRA, positive ion mode
File Number
11-Feb-2016 14:45:58
TOF MS ES+
1.91e3
100
%
0
447.1
[M+H]+
[M+Na]+
449.1
469.1
550.0
552.0
957.1
955.1
471.1
[2M+Na]+
959.1
[2M+H]+
915.2
893.2
554.0
960.1
522.3
961.1
555.0
962.1
1046.1
175.4
242.3
316.0
446.6
641.0
811.1
859.3
1275.1
1363.4
1441.1
1567.6
m/z
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400
1500
1600

Z:\SERVIS_HR\...\HR file number HIGH RESOLUTION SPECTRA Name, Sample label

100216servisHR_12+ #65-67 RT: 1.82-1.87 AV: 3 NL: 5.55E6
T: FTMS + p ESI Full ms [200.00-2000.00]



EI/CI ionization technique

What can be seen in the HR spectrum

EI … peak for ion M+* is usually the highest one but there are also peaks for [M+H]+ and [M-H]+ ions which are smaller

CI … the highest peak is usually for [M+H]+ ion, there are also peaks for [M-H]+ and M+* ions which are usually smaller

- this is not dogma, for some molecules the highest peak could be different then those previously mentioned

Elemental Composition Report

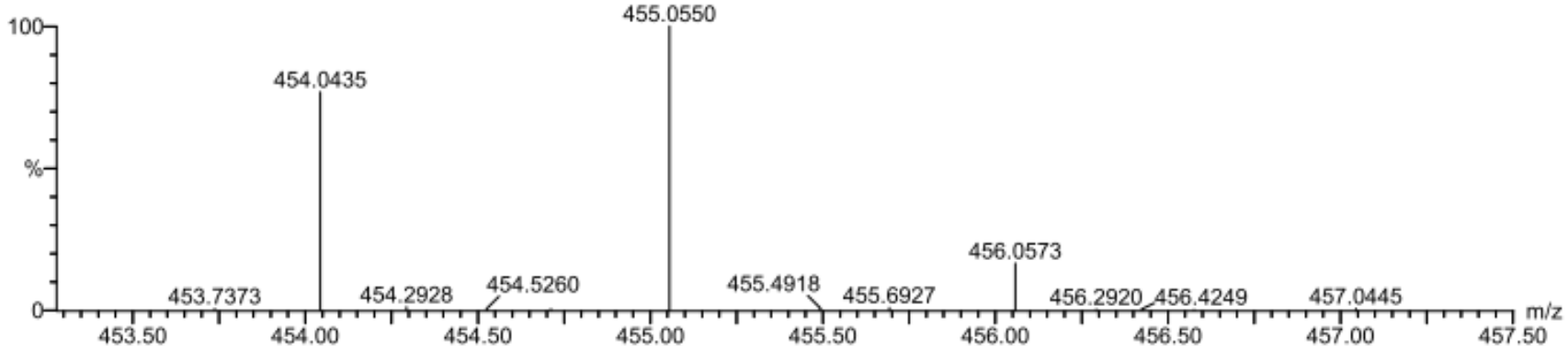
Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off

Monoisotopic Mass, Odd and Even Electron Ions
53 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 0-14 H: 0-10 N: 0-1 O: 0-1 F: 0-13

Kvicala, OS622x 592 (8.992) Cm (592-(609:614+606:613))

TOF MS EI+
1.73e+003

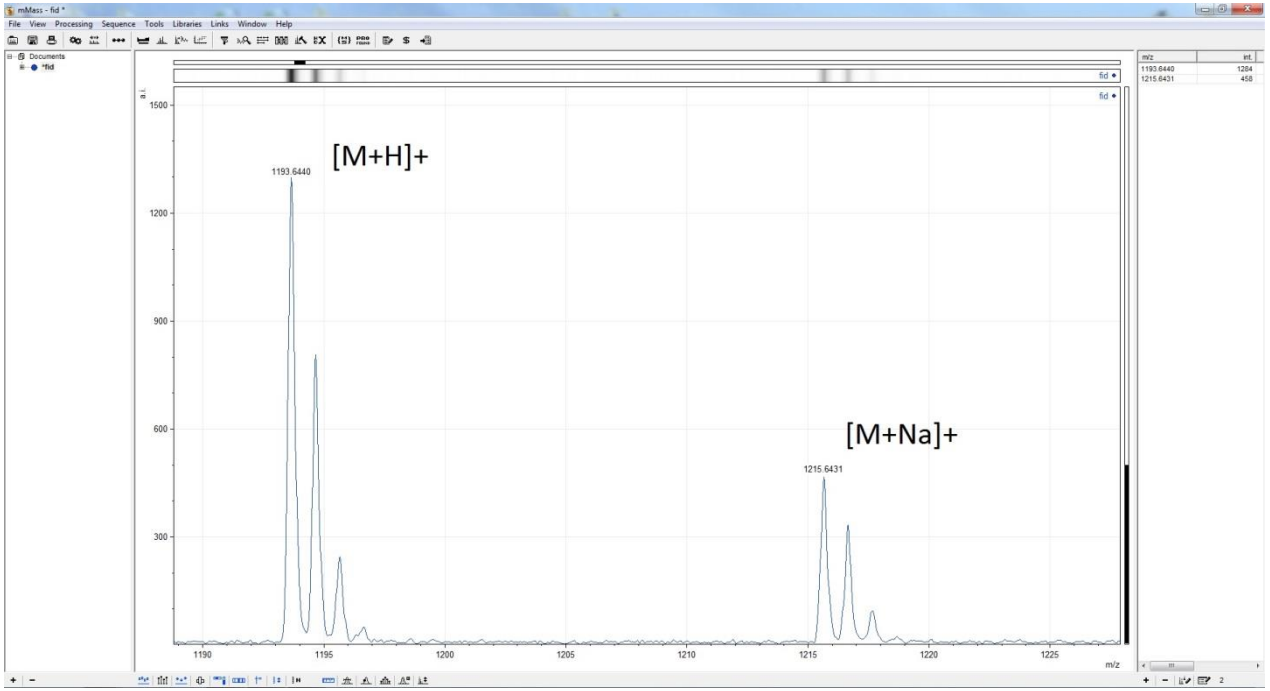


Minimum:				-1.5		
Maximum:		5.0	5.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
455.0550	455.0555	-0.5	-1.1	4.0	16.2	C14 H10 N O F13

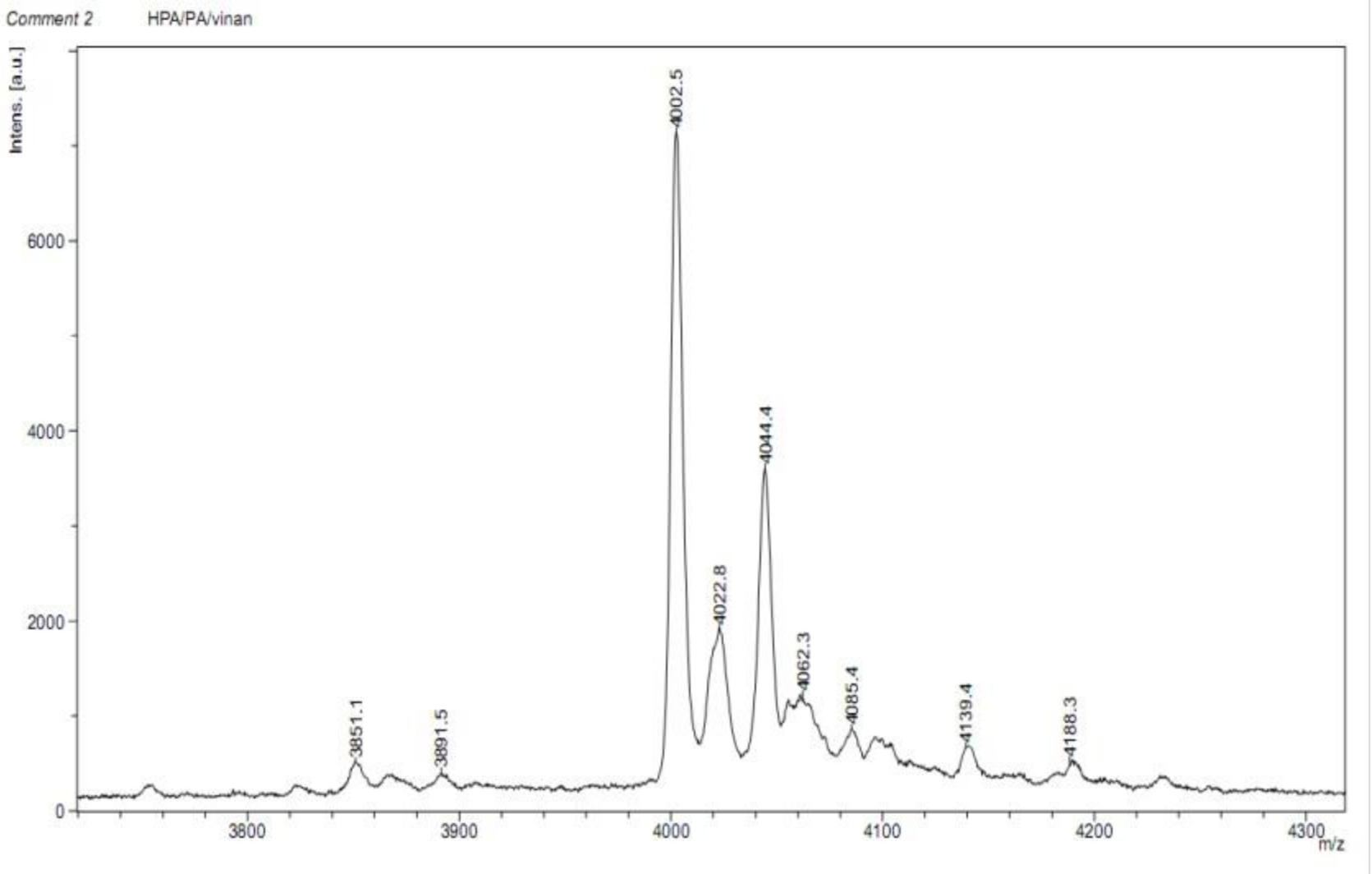
MALDI ionization technique

[M+H]+ … almost exclusively, <u>easier interpretation of the spectrum</u>

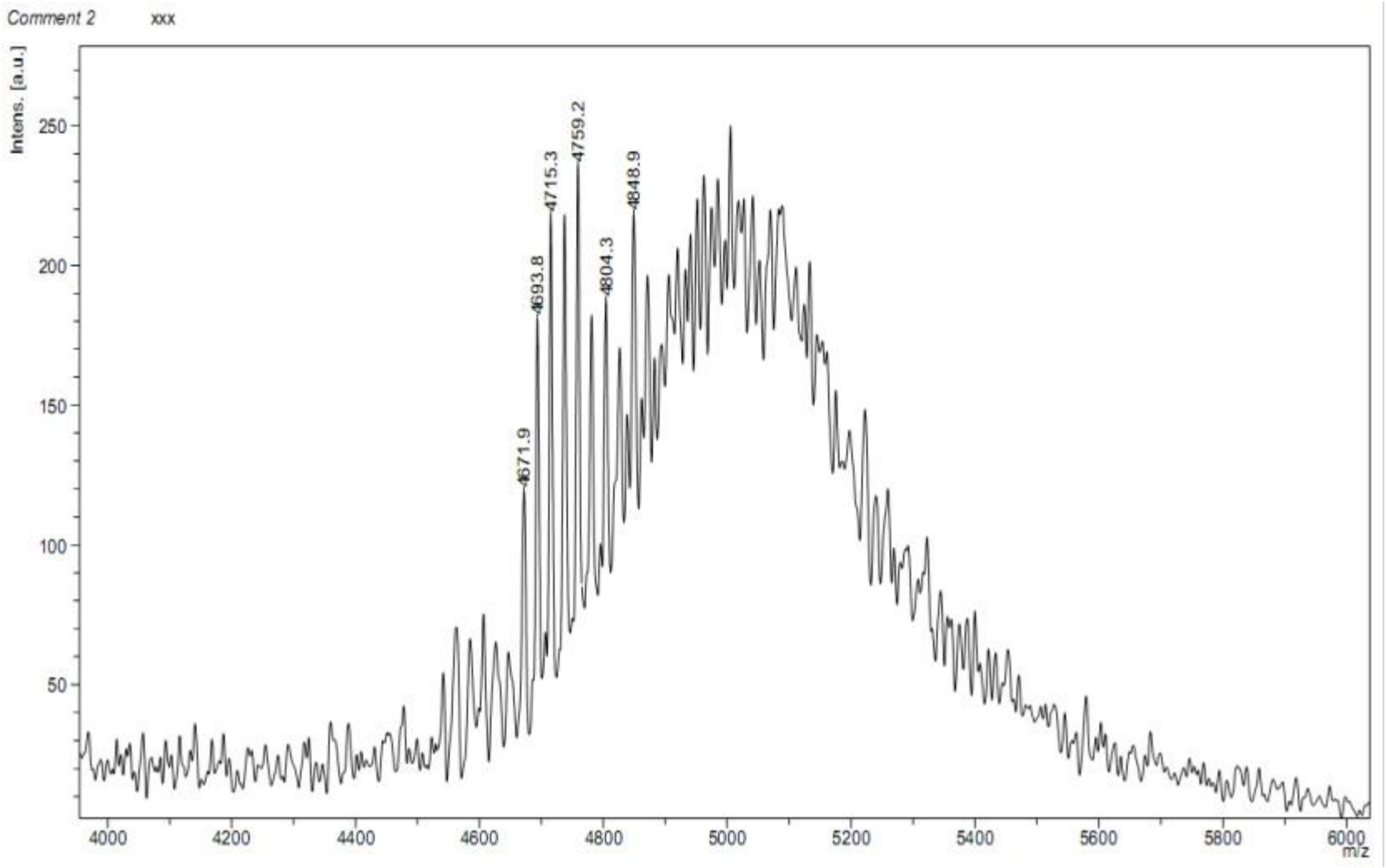
… sometimes accompanied by [M+Na]+ (+23) and [M+K]+ (+39) ions for smaller molecules



MALDI spectrum of clean sample



MALDI spectrum of sample which is contaminated wit salts



Usual contaminants in ESI

- Phthalates – from plastic, contaminated solvents
  peaks 279, 301, 391, 413
- Antioxidants – from plastics
  peaks 663, 685
- PEGs – from teflon/silicon spectrum
  peaks +44 series
- Fatty acids – from skin
  peaks negESI 255, 283

Thank you for your attention