

Quantitative Detection of Residuals on Surfaces

by Vacuum Induced Desorption Mass Spectrometry

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ISO 19227 - Implants for surgery

Cleanliness of orthopedic implants

- Visual Inspection
- Bioburden
- Bacterial Endotoxins
- **Organic Contaminants**
 - Extraction
 - Detection (TOC, THC)
- Inorganic Contaminants
- Particulate Contamination
- Cytotoxicity

Cleanliness after Production

- Chemical, film-like Contamination
 - Production Aids
 - Oil, grease, detergents
 - Qualitative and/or quantitative detection
 - Few standards available
 - Requirements specification rather than acceptance criteria (ISO 19227)

Surgical Site Infections(Hip Prosthesis)

Country	No. of operations	No. of SSIs (1)	Percentage of SSIs per 100 operations [95% CI] (2)	No. of operations with a known discharge date
Patient-based data				
Austria	11 703	89	0.8 [0.6-0.9]	11 678
Finland	13 712	239	1.7 [1.5-2.0]	6 477
France	31 465	313	1.0 [0.9-1.1]	31 465
Germany	102 209	976	1.0 [0.9-1.0]	80 322
Hungary	2 909	66	2.3 [1.8-2.9]	2 909
Italy	5 176	76	1.5 [1.2-1.8]	4 238
Lithuania	960	4	0.4 [0.1-1.1]	960
Malta	573	22	3.8 [2.4-5.8]	573
Netherlands	13 893	291	2.1 [1.9-2.3]	13 893
Norway*	22 323	629	2.8 [2.6-3.0]	22 279
Portugal	3 031	27	0.9 [0.6-1.3]	3 031
UK–England	95 997	638	0.7 [0.6-0.7]	95 997

ECDC: Annual epidemiological report for 2014

Detection Methods

Wipe



Test ink



www.afs.biz

XPS Analysis



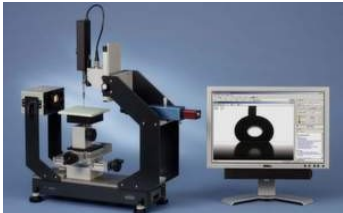
www.prevac.eu

ToF-SIMS



www.iontof.com

Contact angle measurement



<http://www.laborpraxis.vogel.de>

Fluorescence detection



www.sita-process.com

TD-GC-MS



www.cassen.ca

FTIR Spektr.



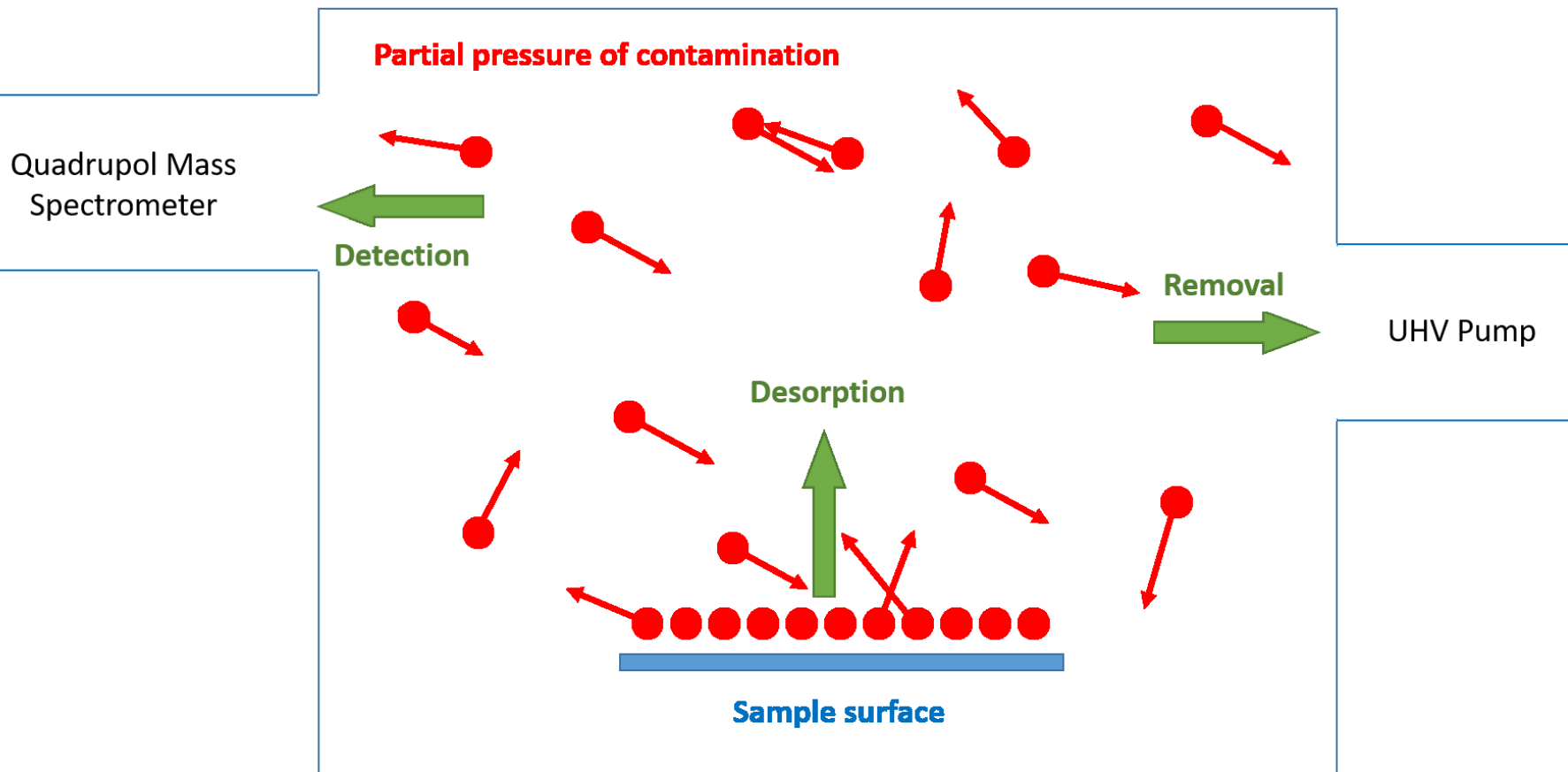
www.agilent.com

Simple and non destructive, low amount of information

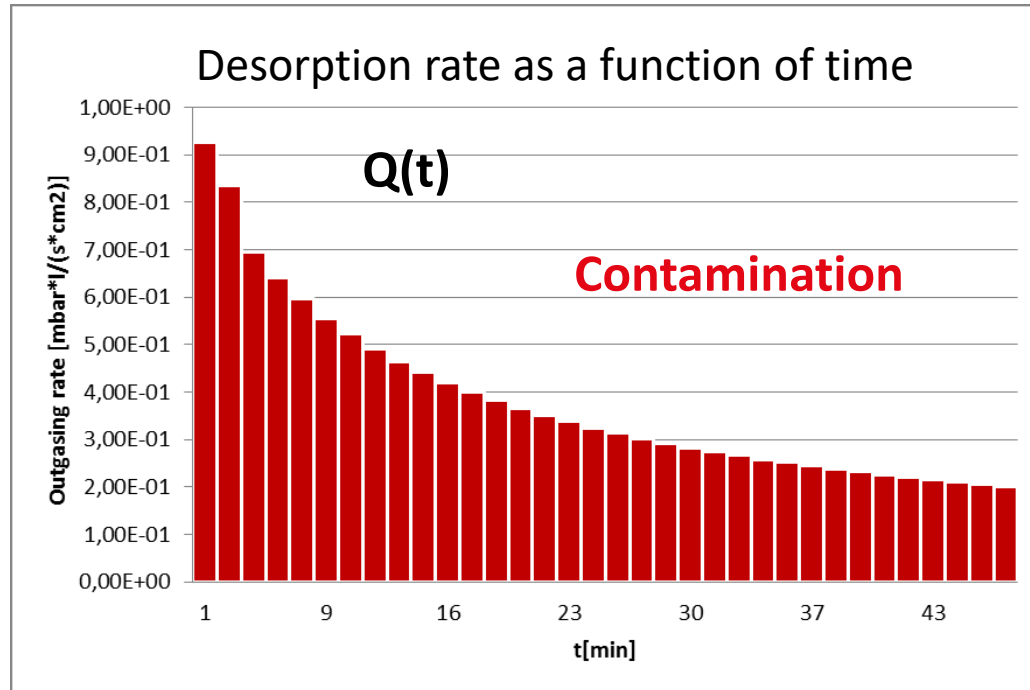
Complex & destructive, High amount of information

Basic Principle

Vacuum System



VIDAM



Desorption rate $Q(t)$

Residual contamination on the surface = $\int Q(t)dt$

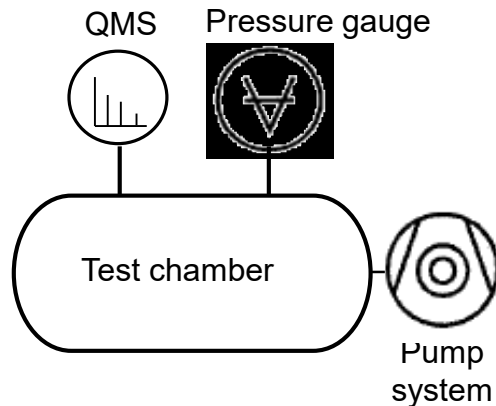
Each column shown represents the result of a complete mass spectrum ($m/z = 1-200$)

- Allows for non-destructive evaluation without prior preparation of the samples
- Allows for evaluation of any geometry of single samples as well as complete modules or production lots

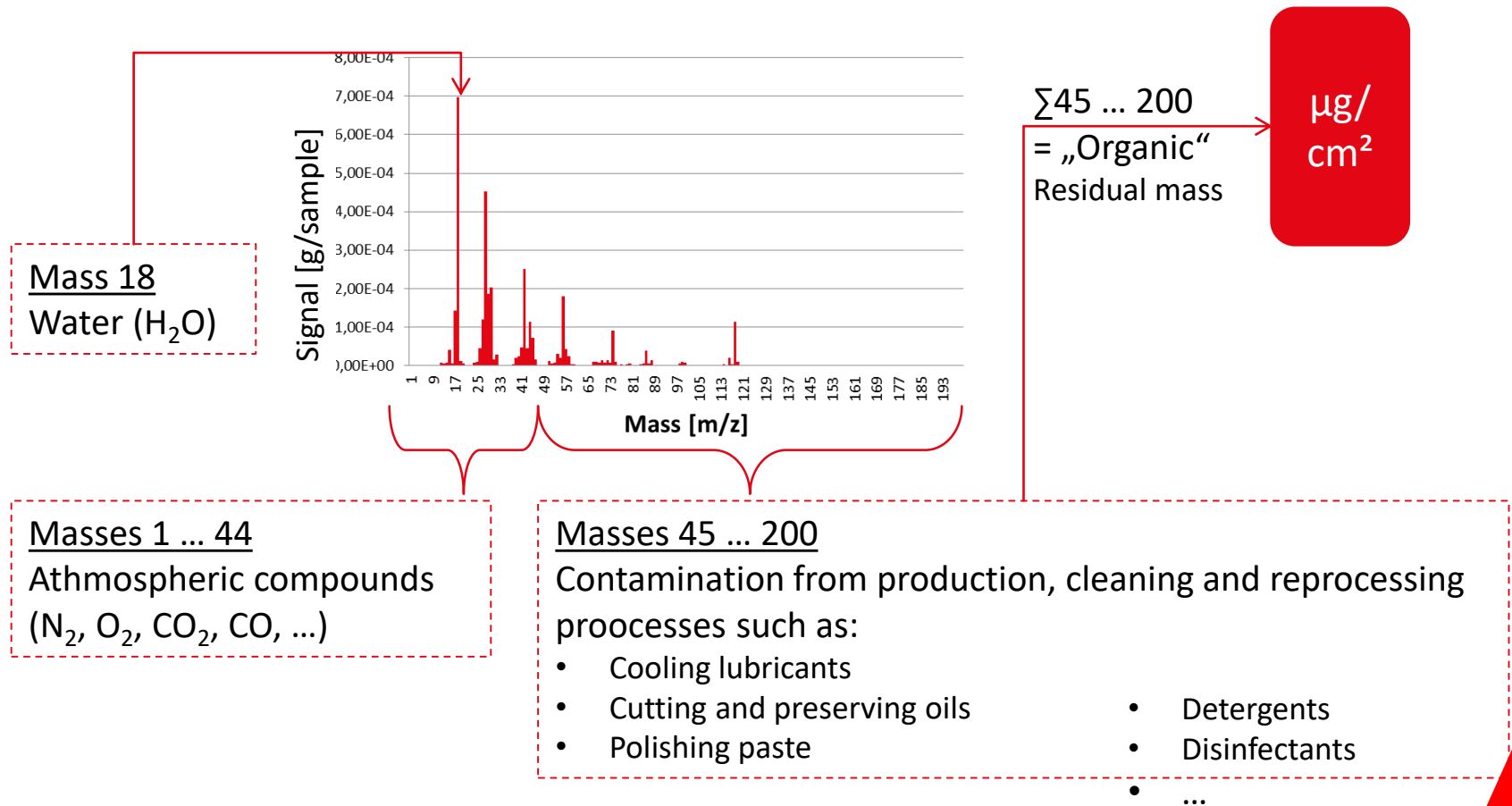
VIDAM



- VIDAM = Vacuum-induced desorption-analysis measurement system
= Vacuum-induced mass-spectrometry
- Developed by VACOM Systems

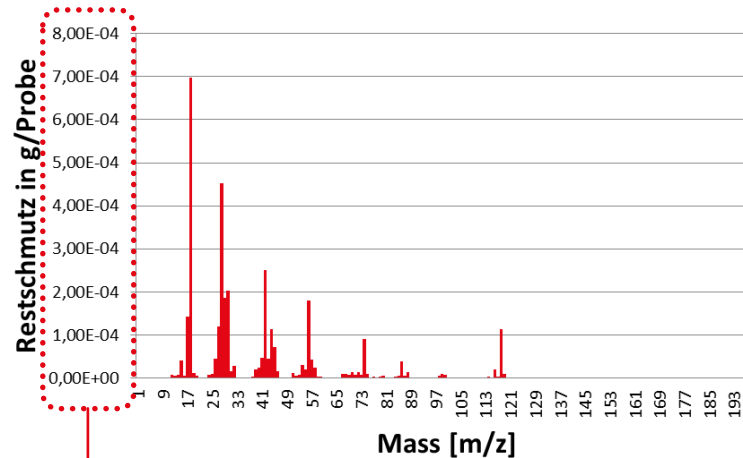


Interpretation



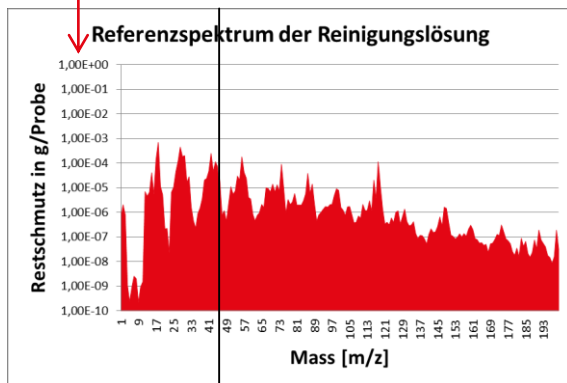
Interpretation

Practical:
logarithmic scale
easier to interpret



$\Sigma 45 \dots 200$
= „Organic“

$\mu\text{g}/\text{cm}^2$

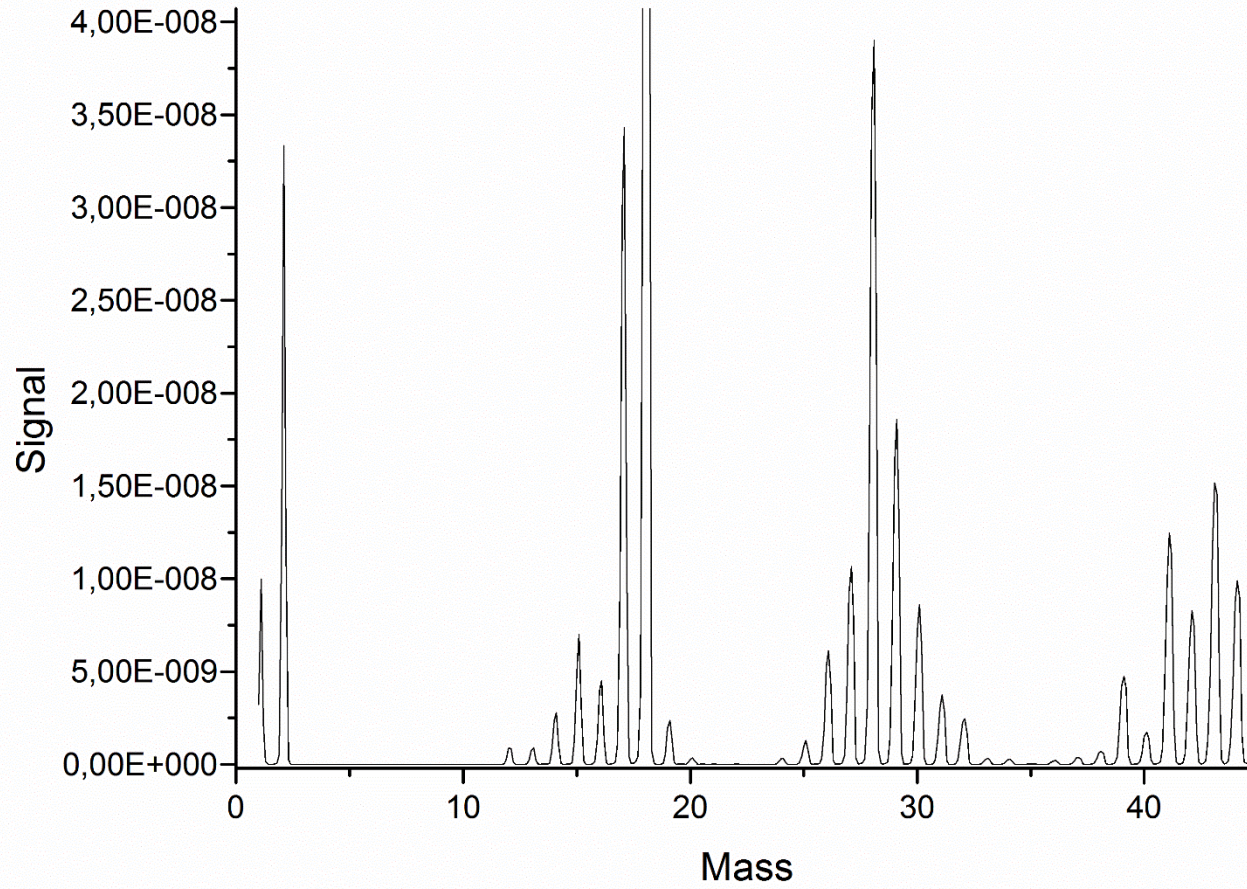


Masses 45 ... 200

Contamination from production, cleaning and reprocessing processes such as:

- Cooling lubricants
- Cutting and preserving oils
- Polishing paste
- Detergents
- Disinfectants
- ...

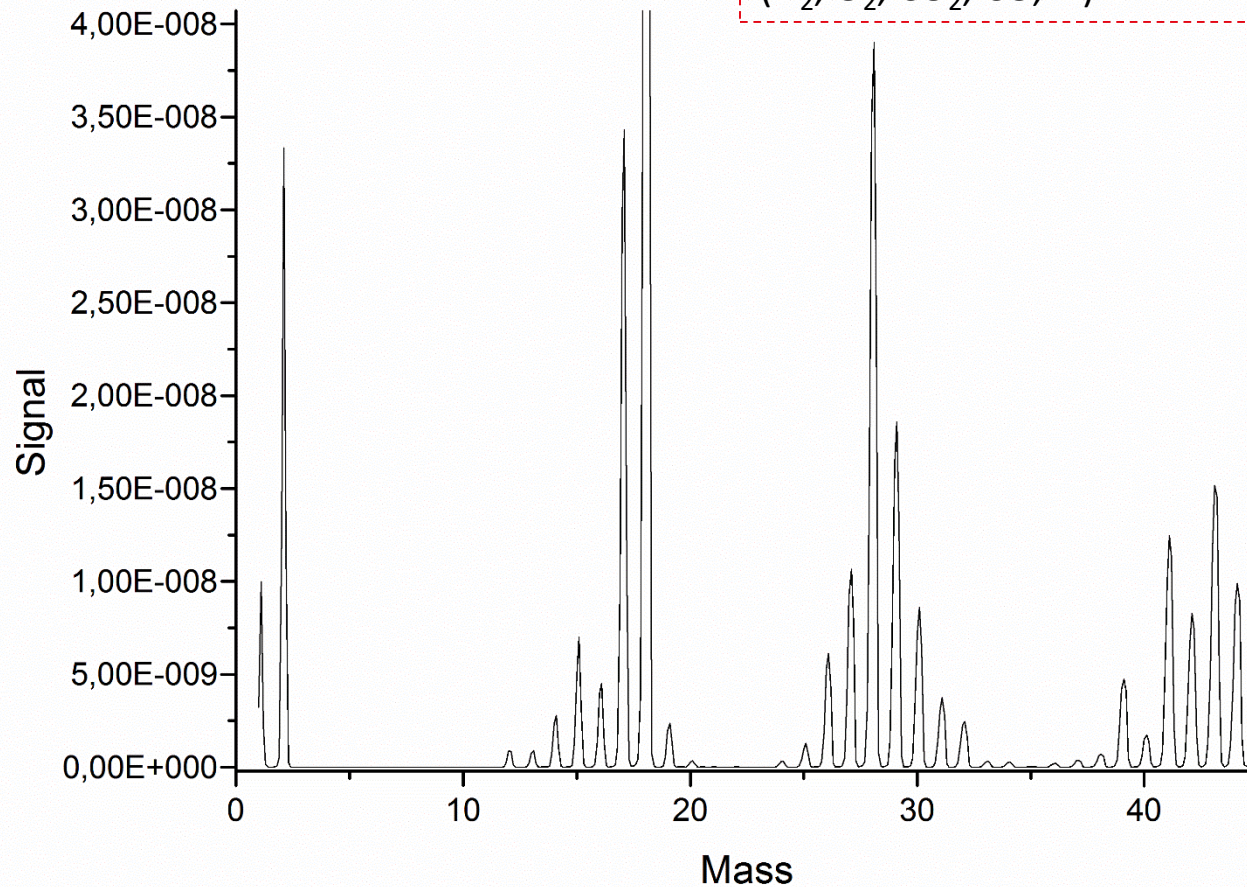
Example



Example

Mass 18
Water (H₂O)

Masses 1 ... 44
Atmospheric compounds
(N₂, O₂, CO₂, CO, ...)

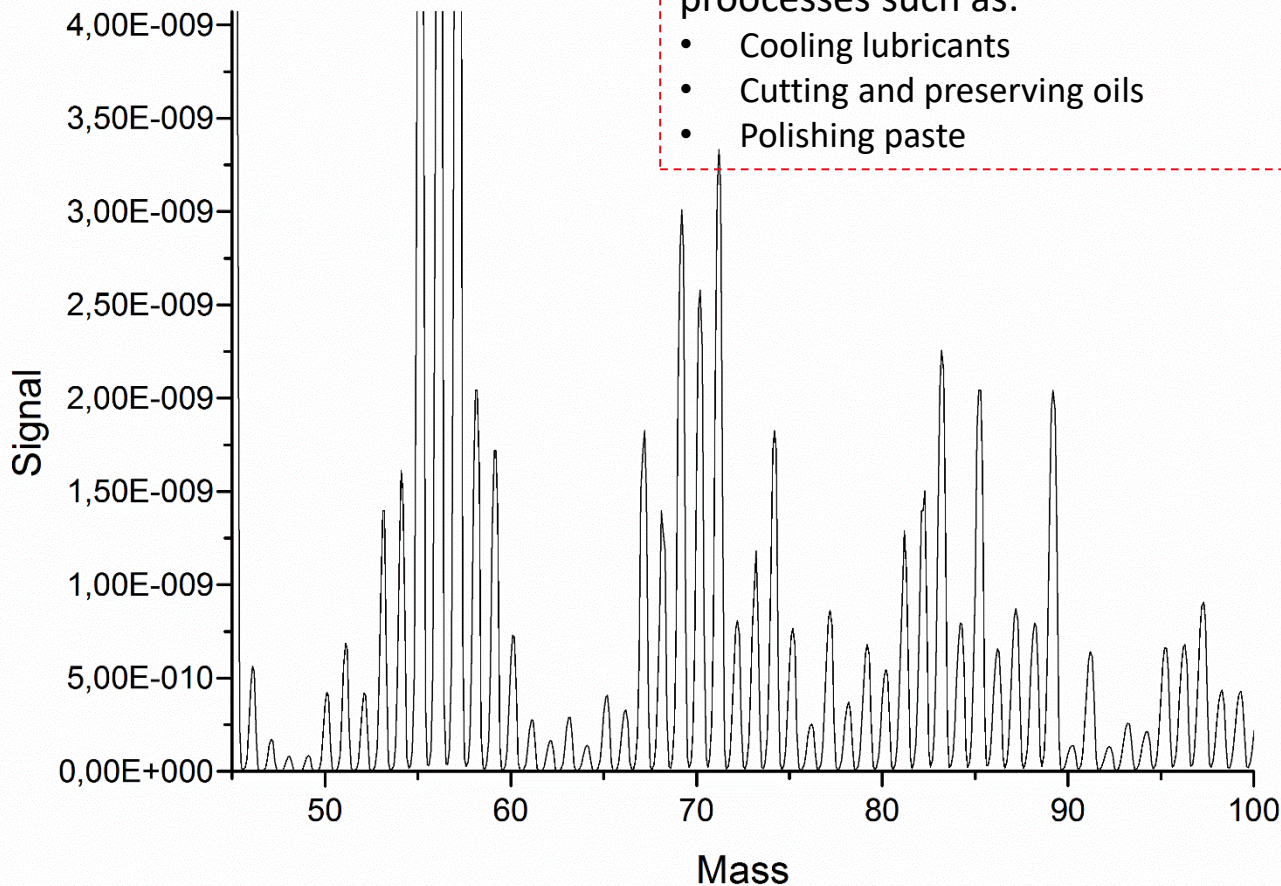


Example

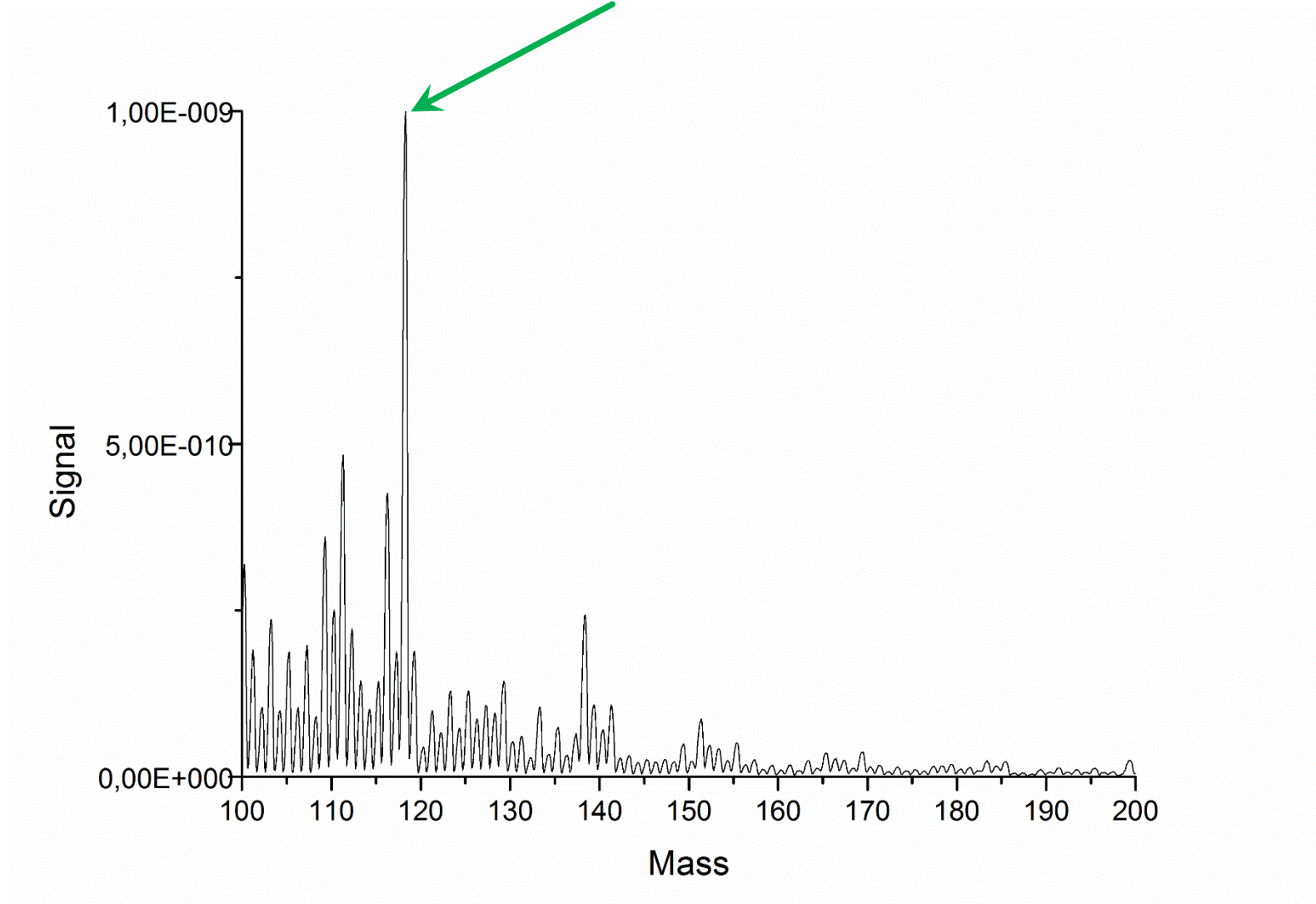
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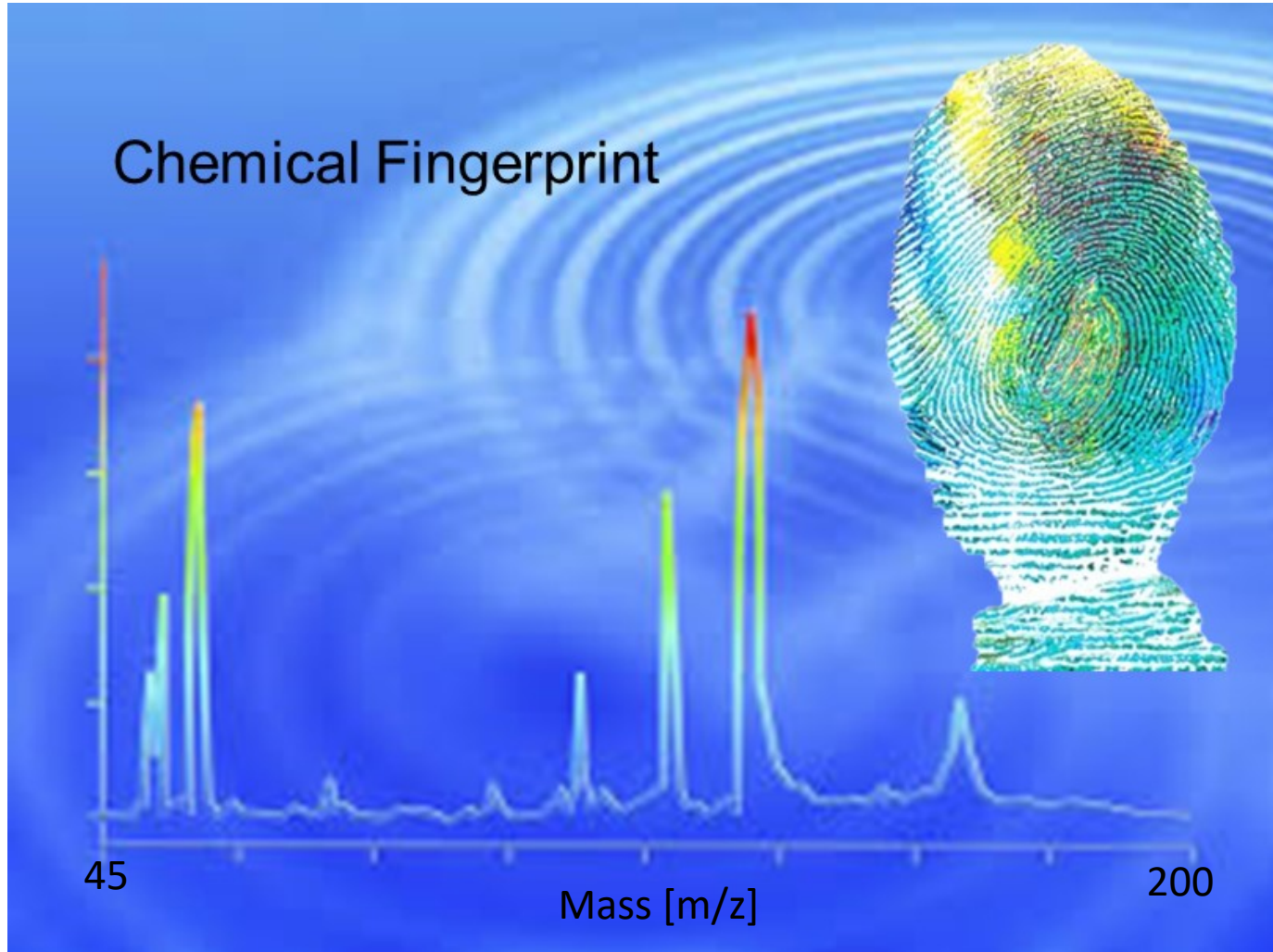
Example



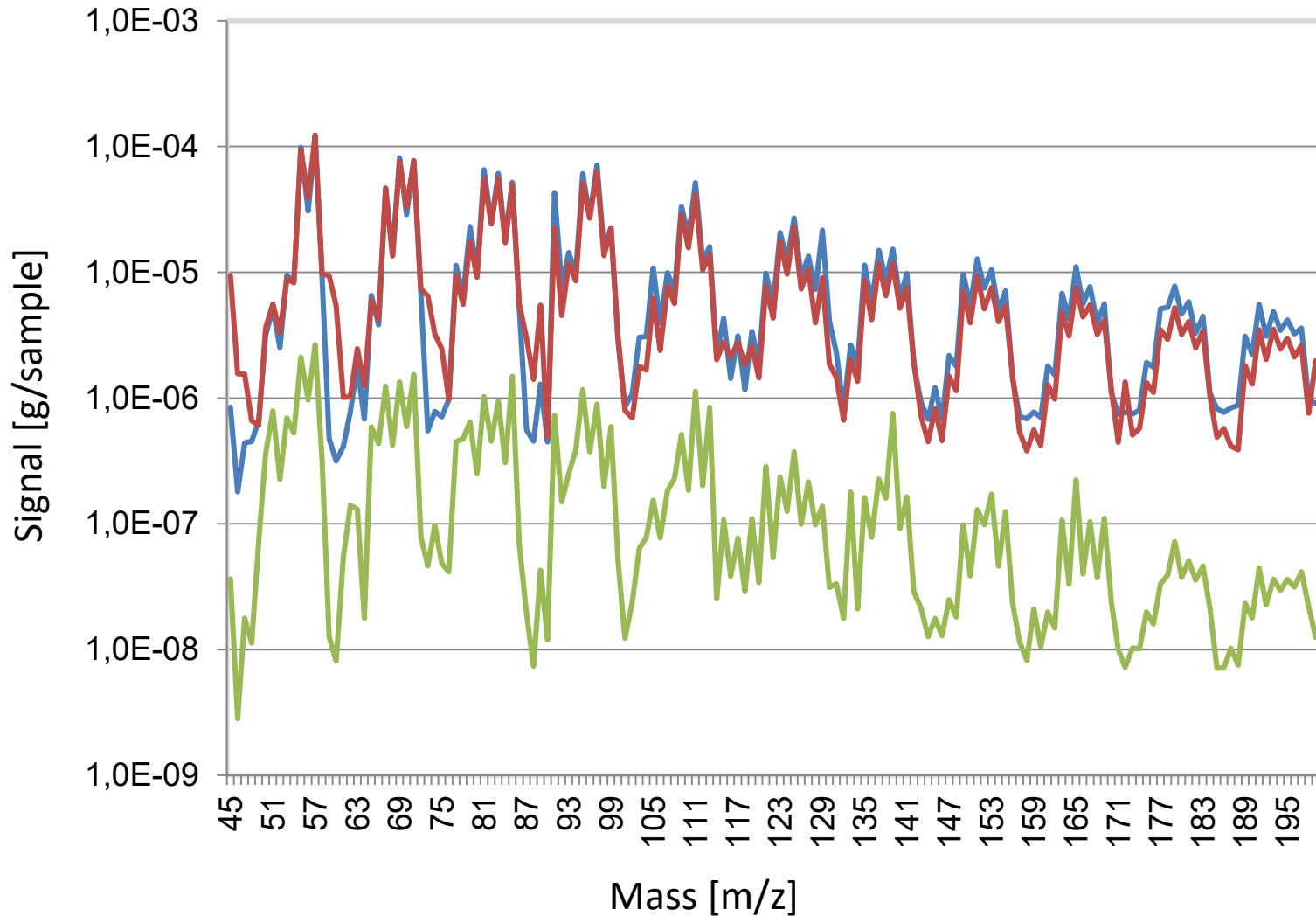
Reduction of Data

- Time profile for every mass is collected
- Times before and after the measurement are fitted
- Complete amount of material for every mass is computed by integration over time taking into account the physical process of pumping
- Results are given as spectrum of residual contamination pumped away
- For substances identified the total amount of contamination on the surface can be given using the specific detection factor (calibration)

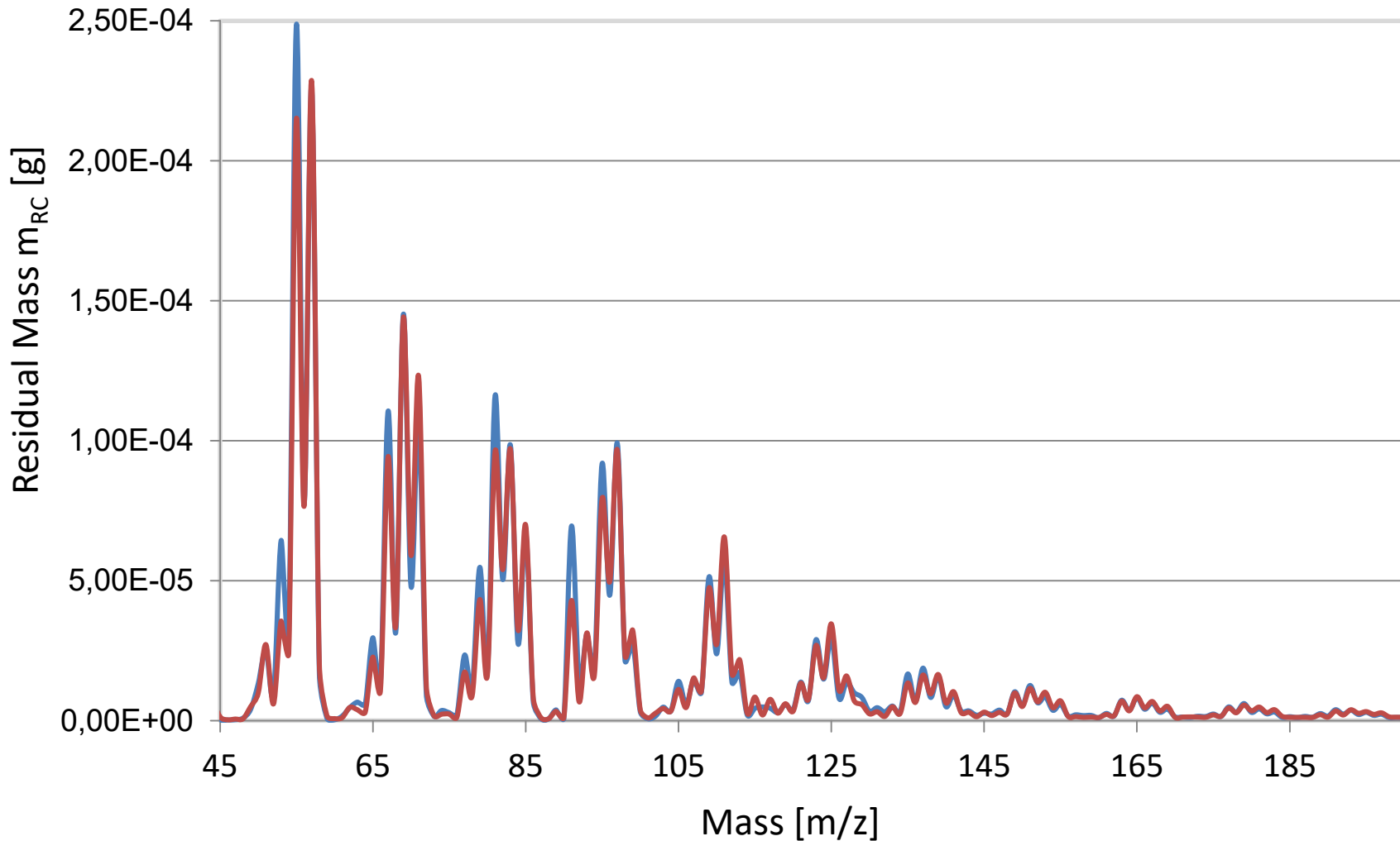
Identification of Contaminants



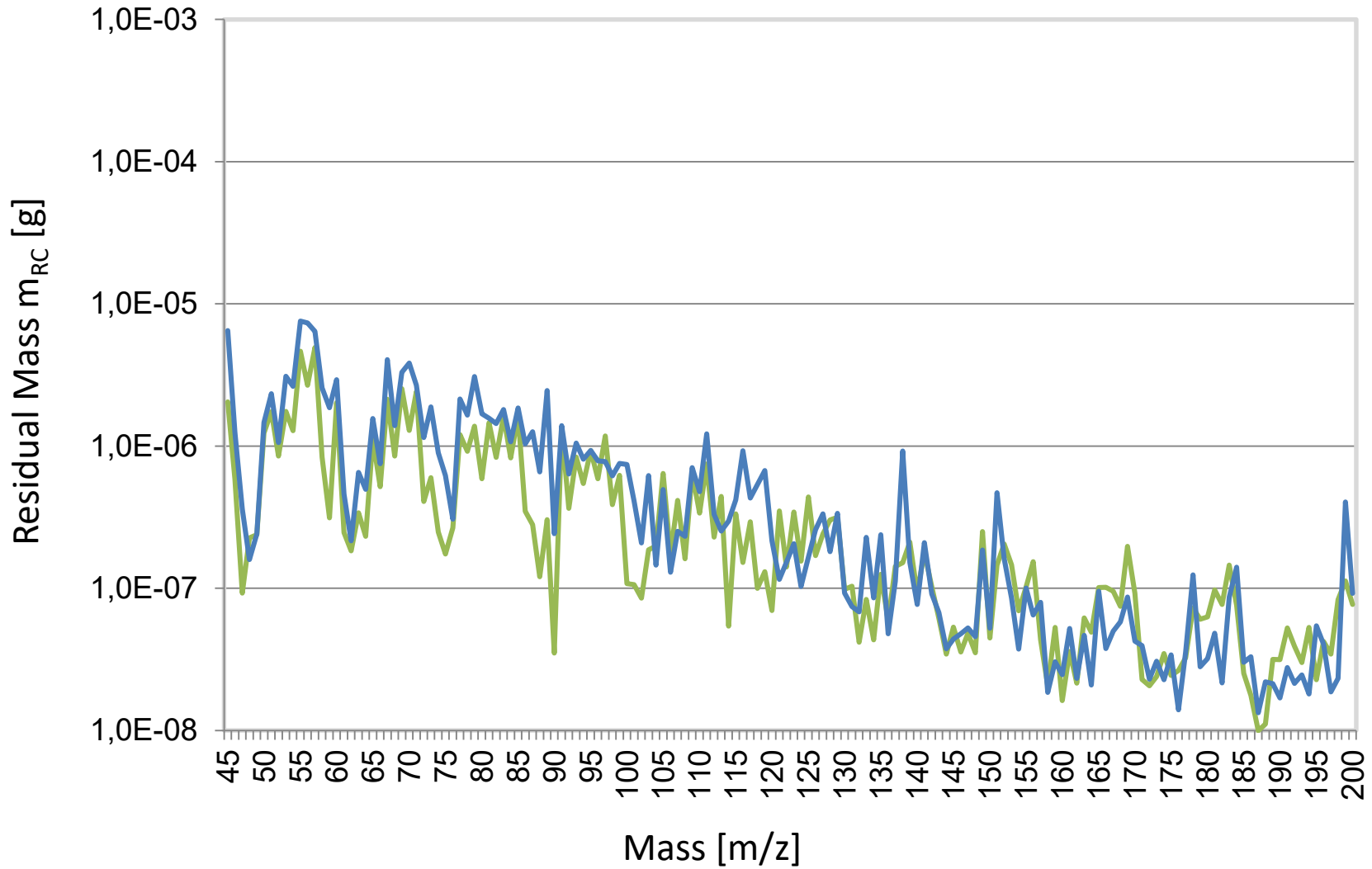
Oil in different amounts



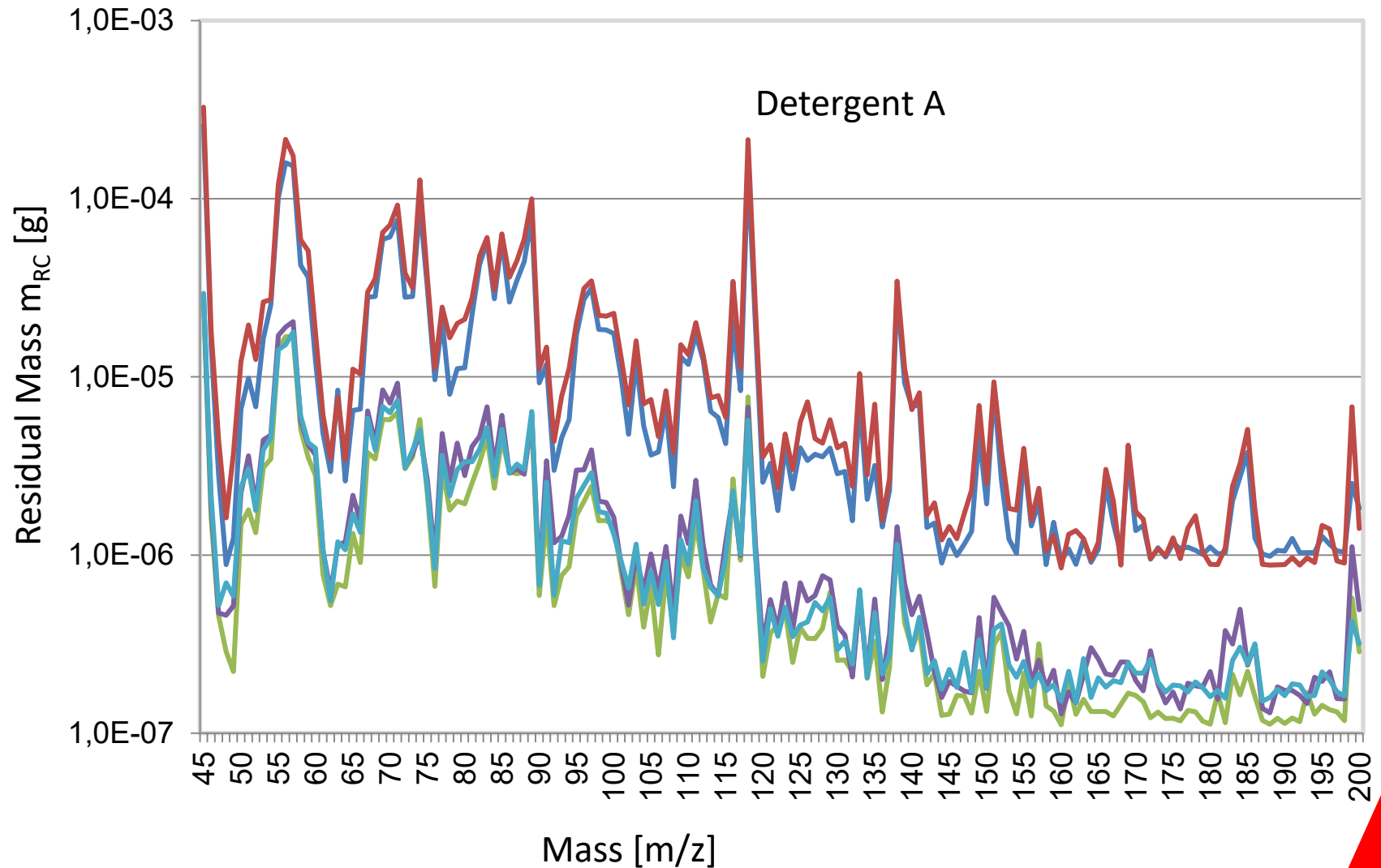
Two slightly different Oils (5 Years of Age)



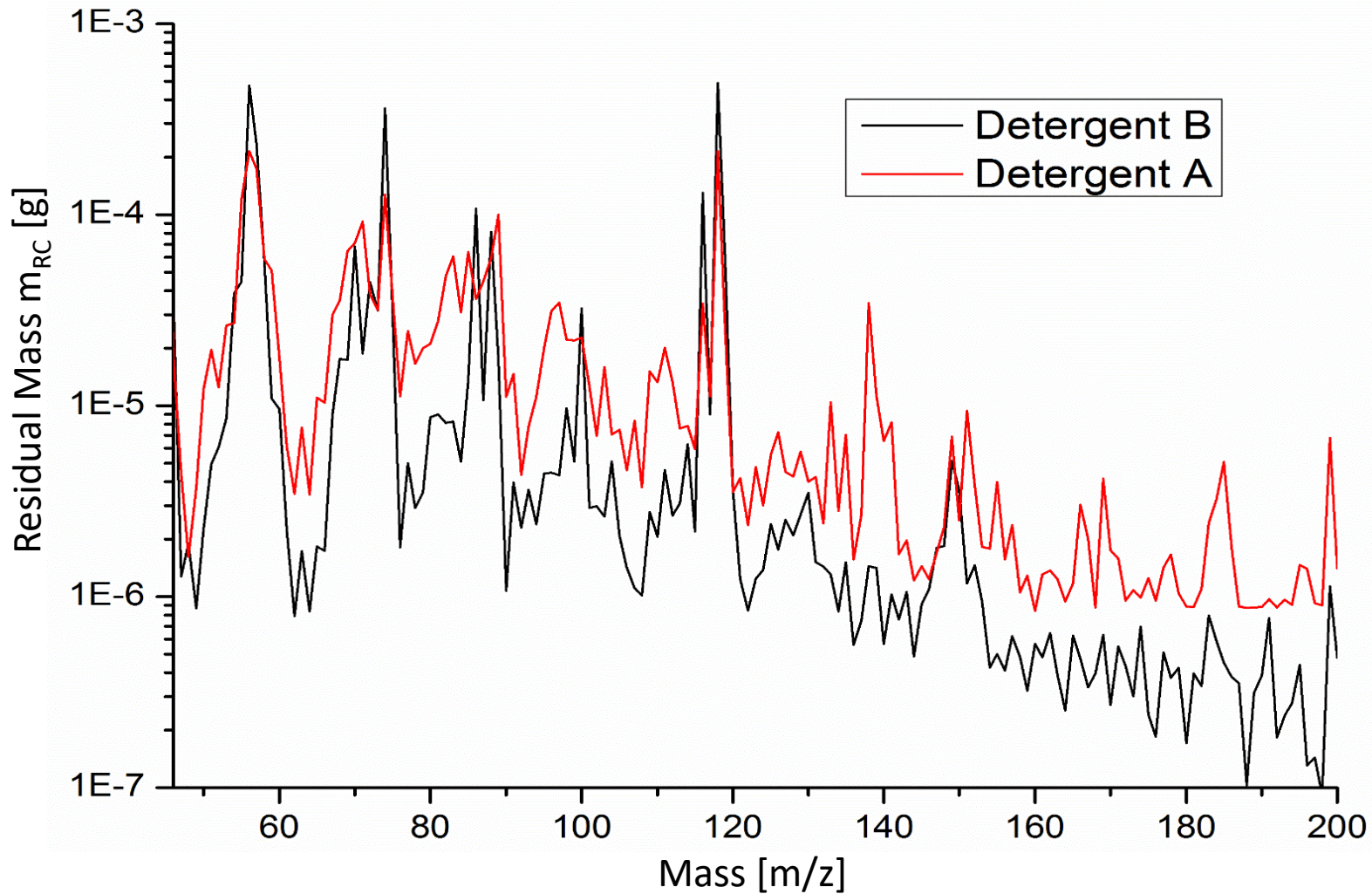
Grease



Detergent A in different amounts



Two different Detergents

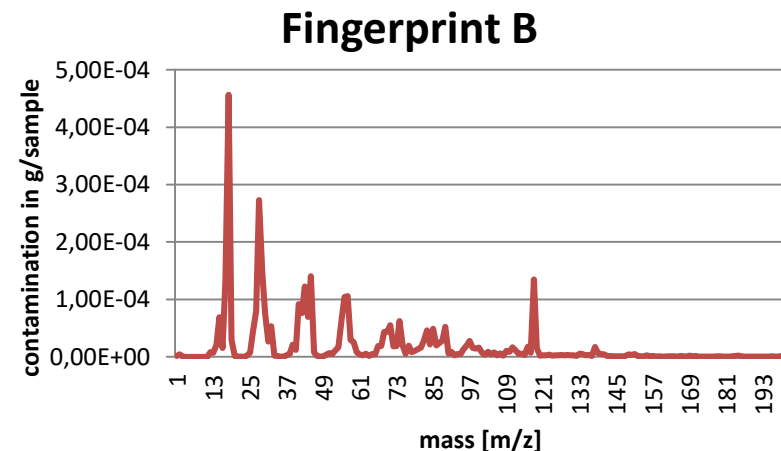
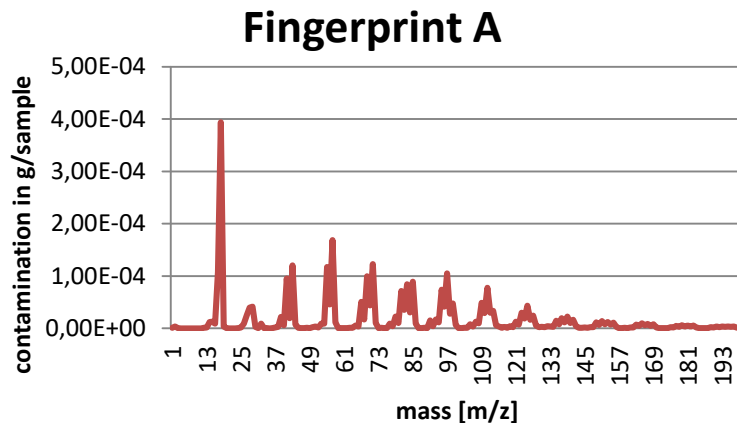


Creating Databases

Verification and Validation

- Efficiency of cleaning process
- Continuity of production aids and cleaning agents

In order to determine residues of production aids and thus to determine the source of contamination, reference measurements, so-called fingerprints, are made of production aids.



Measurements with the mass spectrometer can be made at any time during a production or cleaning process without negative impact on cleanliness of the product.

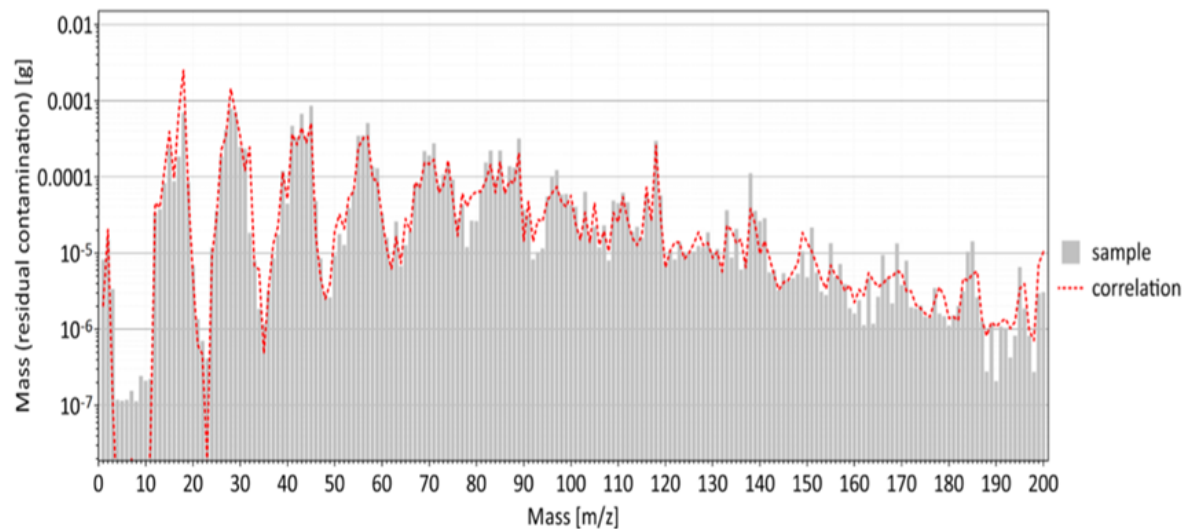
Interpretation of Acquired Records

Production aid

Identification of residues of production aids → source of contamination

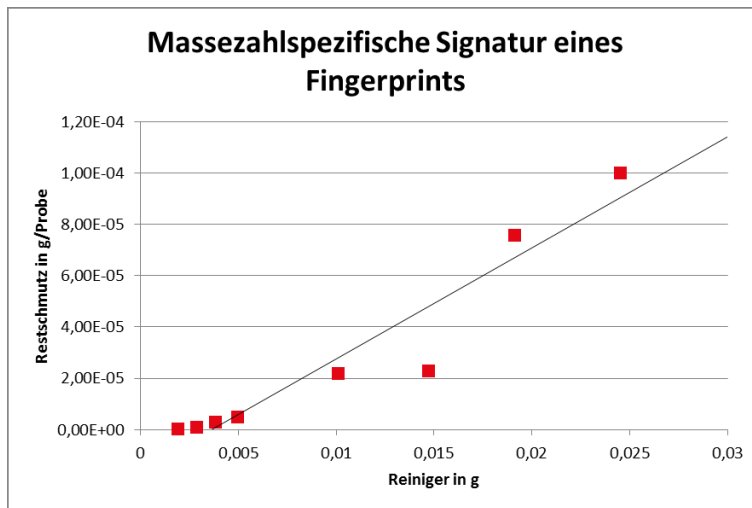
Database with fingerprint spectra allows the identification of sources of contamination.

Quantitative data are gained by optimising the correlation between the measured spectrum and a simulation based on a selection of fingerprint spectra.

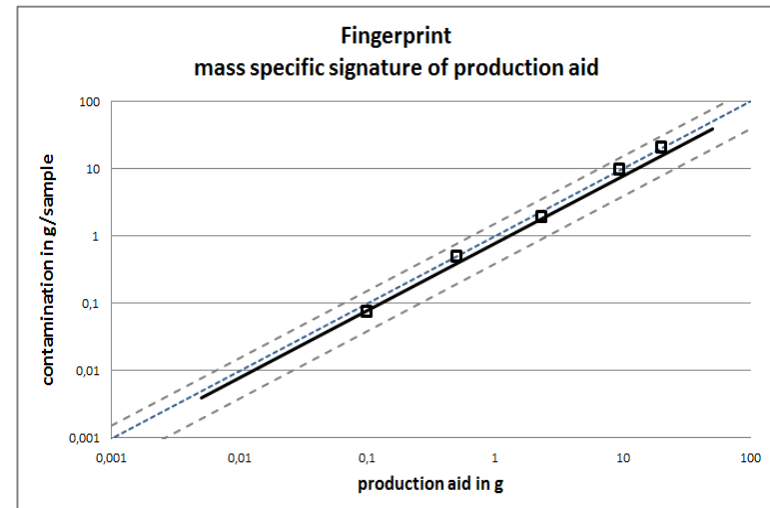


Quantification / Calibration

- Massspectrometric „fingerprint“
- Determination of specific detection factor for example for production aids or detergents by calibration with different amounts of contamination



Own data



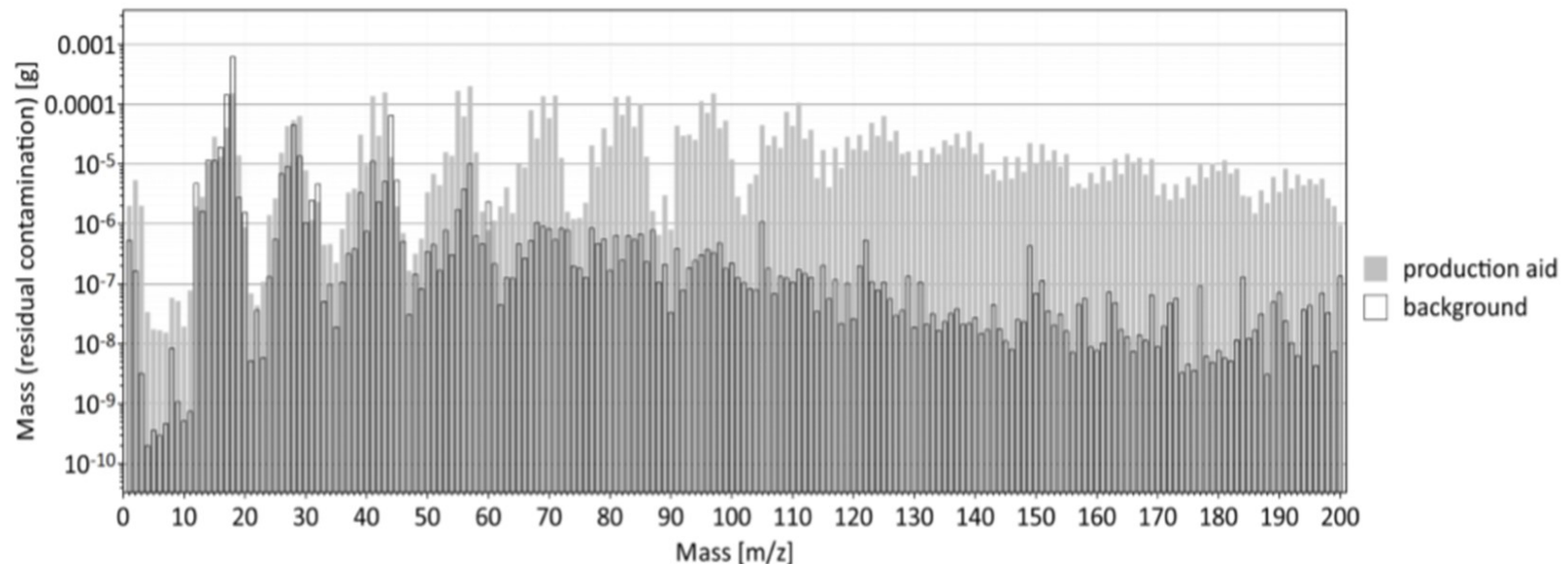
Example provided by VACOM

Specific detection factor is linear and characteristic for the substance

Potential

Quantitative measurement

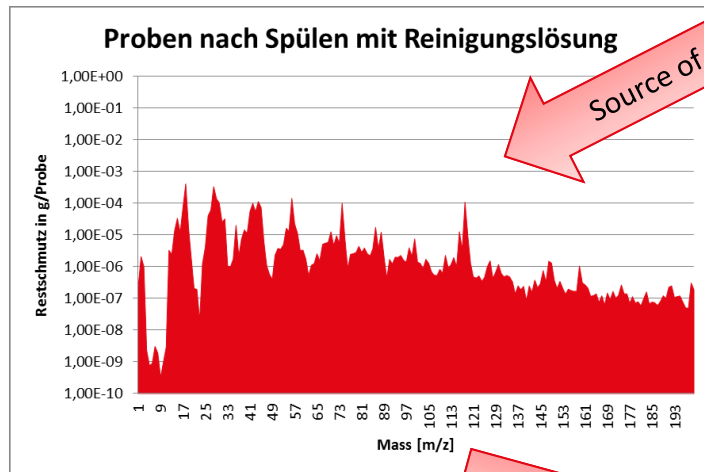
- Database with fingerprint spectra allows the identification of sources of contamination
- residual dirt [g/cm²] or [g]
→ Establishment of appropriate thresholds



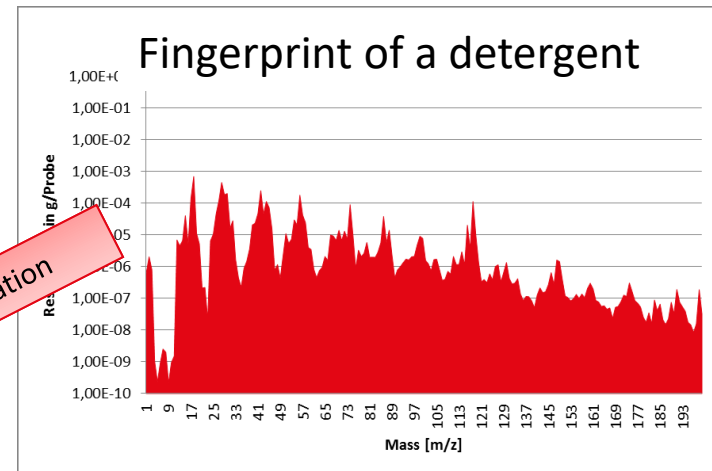
Applications

- Evaluation of efficiency of cleaning processes including analysis of single steps
- Release of manufacturing lots of devices (implants)
- Incoming inspection
- Analysis of residual sterilants
- Analysis of reprocessed devices
- Qualification of packaging materials and processes
- Cleaning of delicate devices
- Together with cytotoxicity testing definition of acceptance criteria for residual contamination

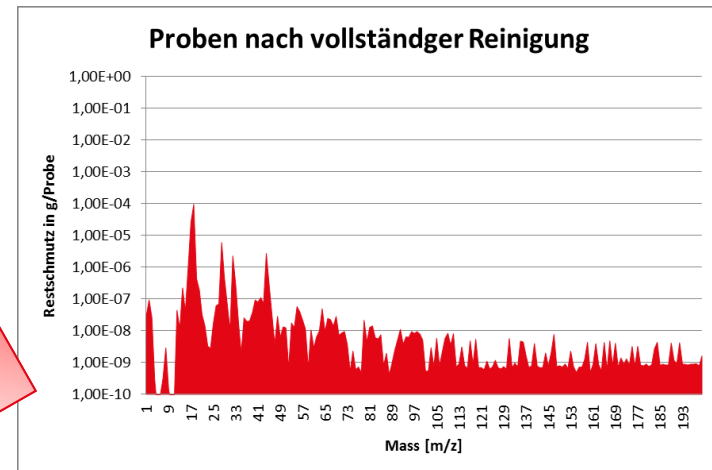
Detergent Removal



Source of Contamination

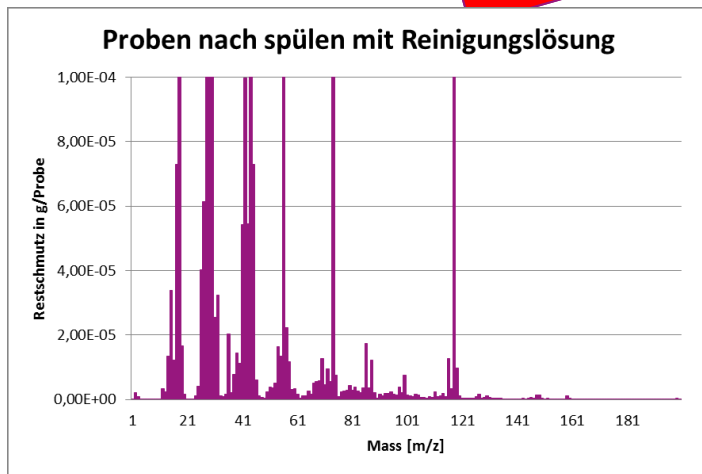
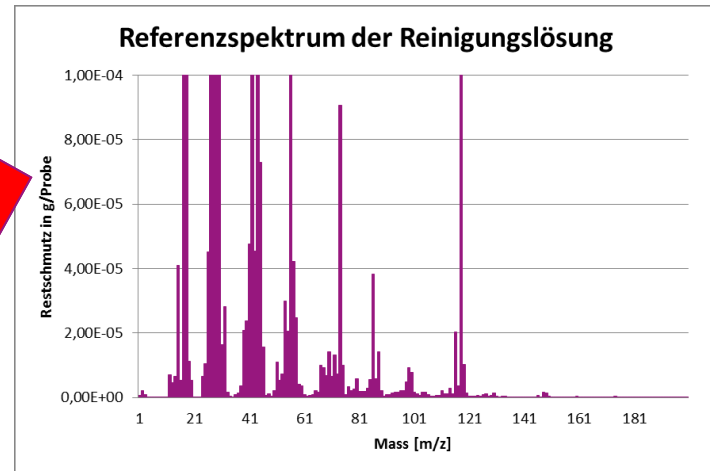


Optimized Rinsing

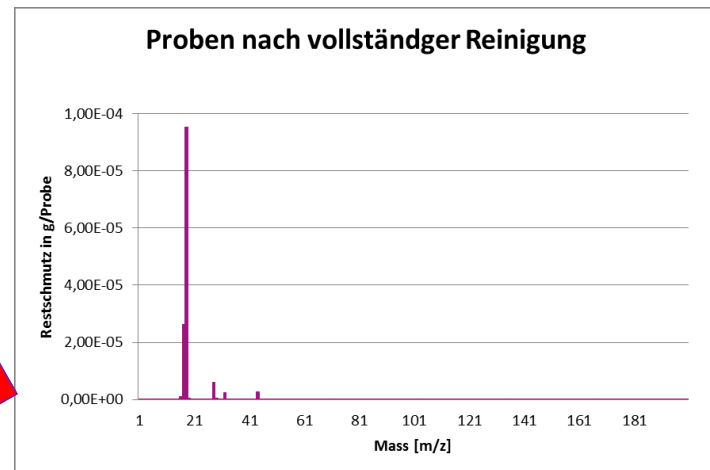


Detergent Removal

Source of Contamination

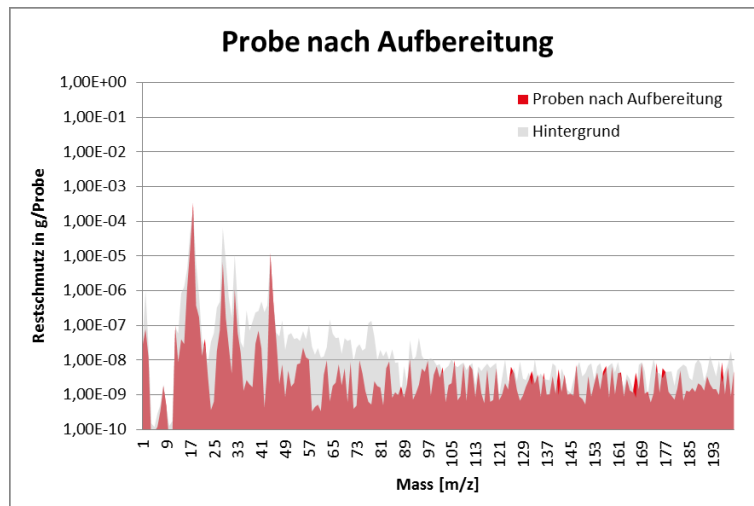


Optimized Cleaning

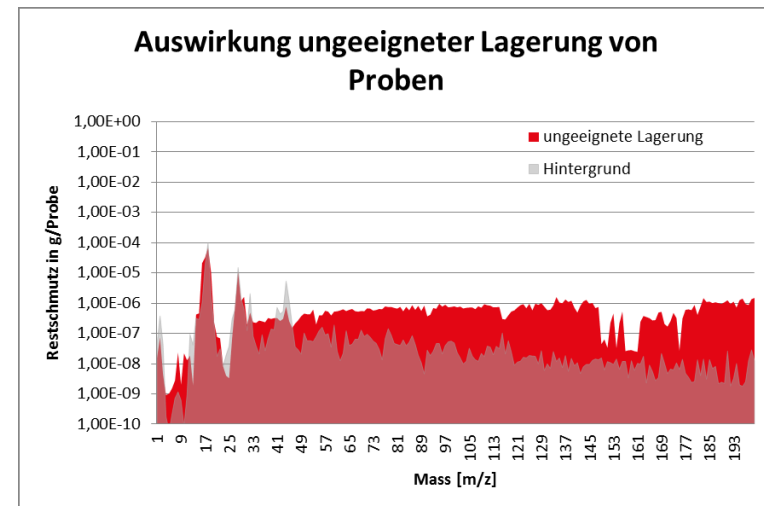


Packaging

- Useful methods of packaging can be evaluated
- Contamination by packaging materials of methods can be detected



Measurement after reprocessing of the sample

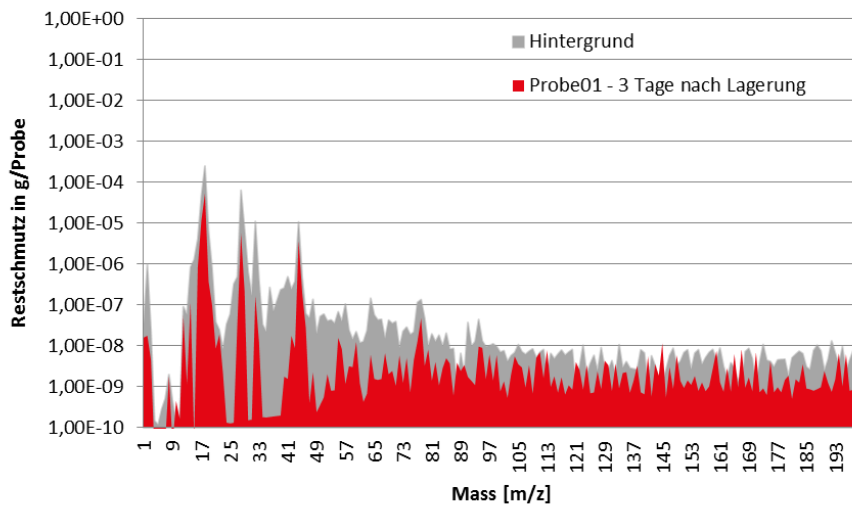


Measurement after 6 days of storage with not suitable packaging material

Packaging

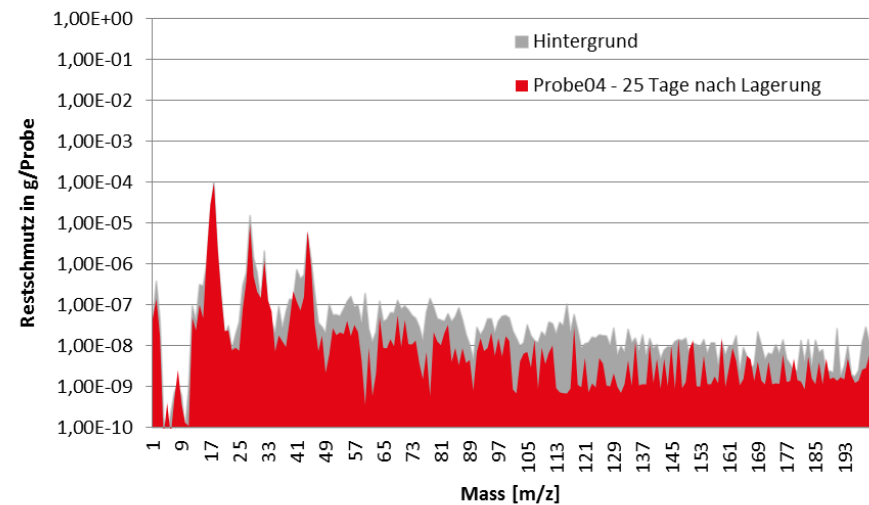
Positive Example

Auswirkungen der Probenlagerung



Measurement after 3 days of storage.

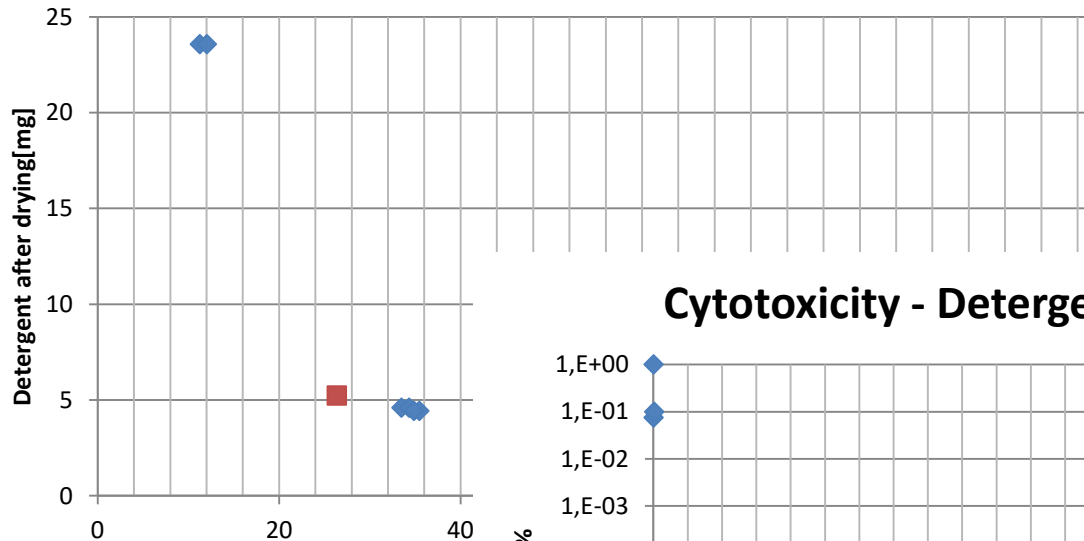
Auswirkungen der Probenlagerung



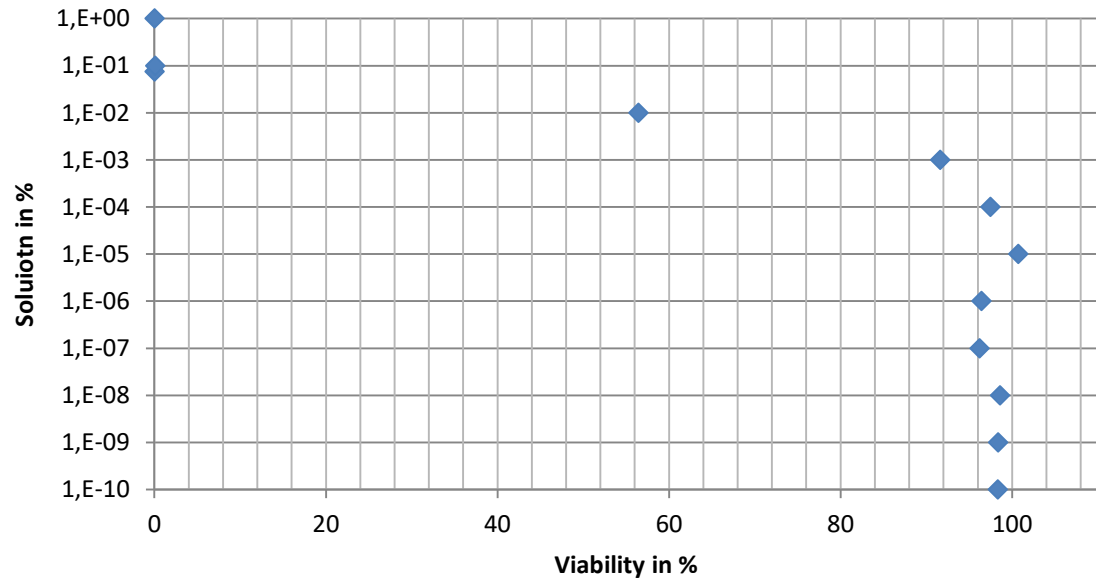
Measurement after 25 days of storage.

Cytotoxicity

Cytotoxicity - Detergent A after drying



Cytotoxicity - Detergent directly applied



Thank you for your Attention