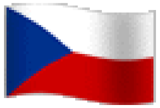
INSTITUTE OF
CHEMICAL TECHNOLOGY PRAGUE

Direct analysis in real time and high resolution mass spectrometry: a new tool for food authenticity

JANA HAJSLOVA
on behalf of her Ph.D. students & young scientists

17.5. 2013, Palermo, Italy

Prague, Czech Republic, EU



ICT Metrological and Testing laboratory

- research and education,
- collaboration with industry,
- collaboration with other research/testing laboratories (private, official),
- international co-operation within EU research projects aimed at detection techniques,
- expertise/testing, consultation
- laboratory audits,
- laboratory training and demonstration

L 1316.2

ilac-MRA

NÁRODNÍ AKREDITAČNÍ ORGÁN

Czech Accreditation Institute
Public Service Company
110 00 Praha 1 – Nové Město, Opletalova 41

issues this

CERTIFICATE OF ACCREDITATION

No. **319 / 2009**

to

Testing Laboratory No. **1316.2**

Vysoká škola chemicko-technologická v Praze
Metrologická a zkušební laboratoř
Technická 5, 166 28 Praha 6

Scope of accreditation:

Chemical analysis of food, beverages, basic materials for production of food and pharmaceuticals, animal feeding stuffs, chemical agents, biological materials (plant and animal tissues) and environment components to the extent as specified in the appendix to this Certificate which is attached.

Prof. Ing. Jana Hajšlová, CSc. and prof. Ing. Miloslav Suchánek, CSc. shall act on behalf of the accredited testing laboratory, and prof. Ing. Jana Hajšlová, CSc., prof. Ing. Miloslav Suchánek, CSc. and doc. Ing. Vladimír Kocourek, CSc. shall be responsible for the correctness of relevant test reports.

This Certificate of Accreditation was issued by the Czech Accreditation Institute, Public Service Company, on the basis of assessment of fulfilment of the accreditation criteria in accordance with

ČSN EN ISO/IEC 17025:2005

and after having found that the testing laboratory had been qualified for objective and independent testing to the extent of the scope of accreditation.

In its activities, performed within the scope and for the period of validity of this Certificate, the holder of this Certificate is entitled to use the identification "Accredited Testing Laboratory No. 1316.2" next to its name provided it observes all relevant regulations relating to the activity of accredited testing laboratory including regulations issued by the Czech Accreditation Institute, Public Service Company.

Should it be proved that the holder of this Certificate fails to meet the accreditation criteria decisive for the issue hereof and the obligations conditioning accreditation, the Czech Accreditation Institute, Public Service Company, may either suspend the validity of or withdraw or change this Certificate.

This Certificate is valid until: **30 April 2014**

Prague: 29 May 2009

Jiří Růžička
Director
Czech Accreditation Institute
Public Service Company

Instruction:
The holder can enter a written objection against this Certificate, provided it concerns the scope of accreditation, in 10 days from the receipt hereof. Timely submitted objection has no dilatory effect.

International projects

SIXTH FRAMEWORK PROGRAMME

SEVENTH FRAMEWORK PROGRAMME

CORE organic II

AUTHENTIC FOOD

theheatoxproject

CONFFIDENCE

COST

QualityLowInputFood

Perfood

SAFE FOOD

BioCop

NANO LYSE

EUREKA

TRUE food

TRADITIONAL UNITED EUROPE FOOD

PROMETHEUS

Br FIRE

TraceQ

EmerCon

Overview

- Introduction: food authenticity & fraud
- Metabolomics: what is its role in food analysis?
- Instrumental platforms for metabolomics
- DART ion source coupled with HRMS
- Case studies based on metabolomics

  - wine authenticity

  - oxidized fats detection

FOOD AUTHENTICITY AND FRAUD

- Important food quality parameter
- Most of valued food commodities are subject to fraud
- Substitution or extension by cheaper product / ingredient / raw material
- Geographic origin
- Use of undeclared technology / processing

ECONOMIC ASPECTS

CONSUMERS´ DECEPTION

THREAT TO CONSUMERS' HEALTH



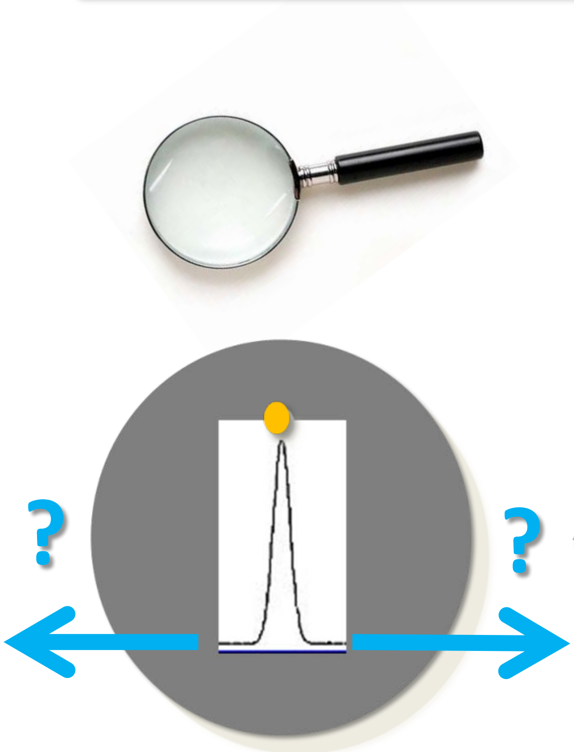




Authentication strategies: where to go….?

´Classic´ approach

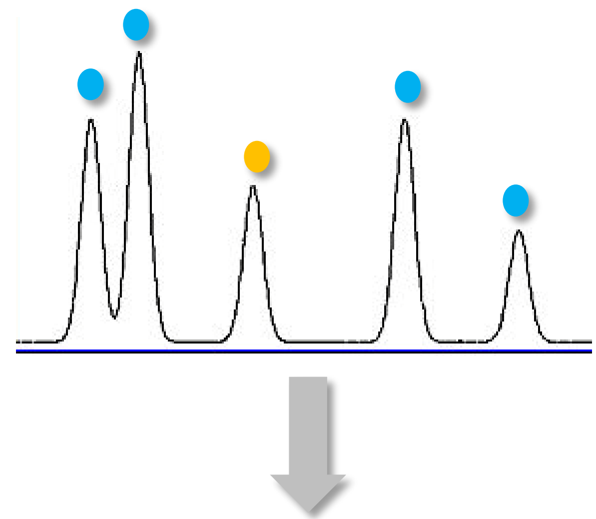
Analysis of one or few target markers



New strategy:
non-target screening / profiling

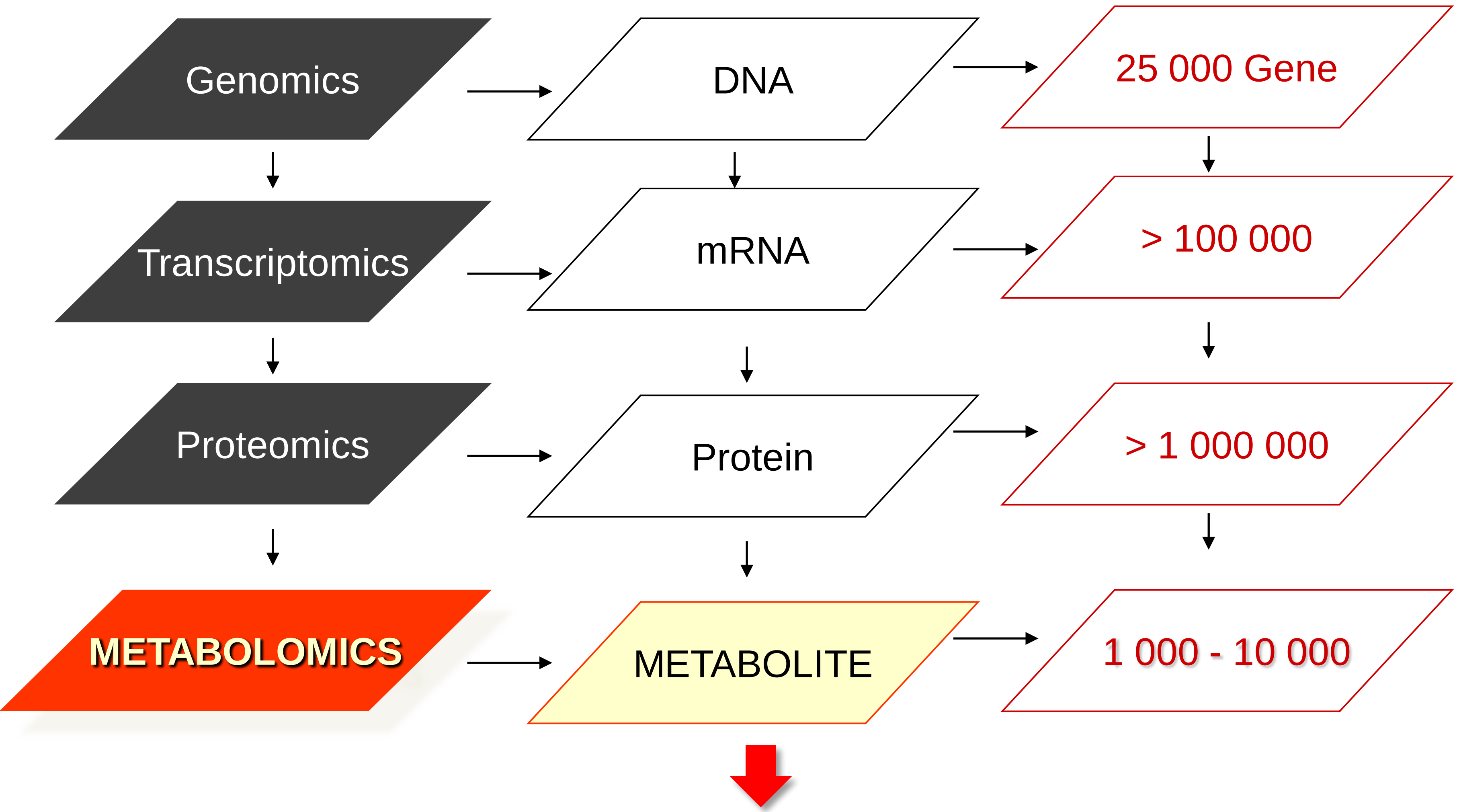
COMPREHENSIVE SAMPLE ANALYSIS

- detection / identification of ´unknown´ components
- Identification of set of composition markers



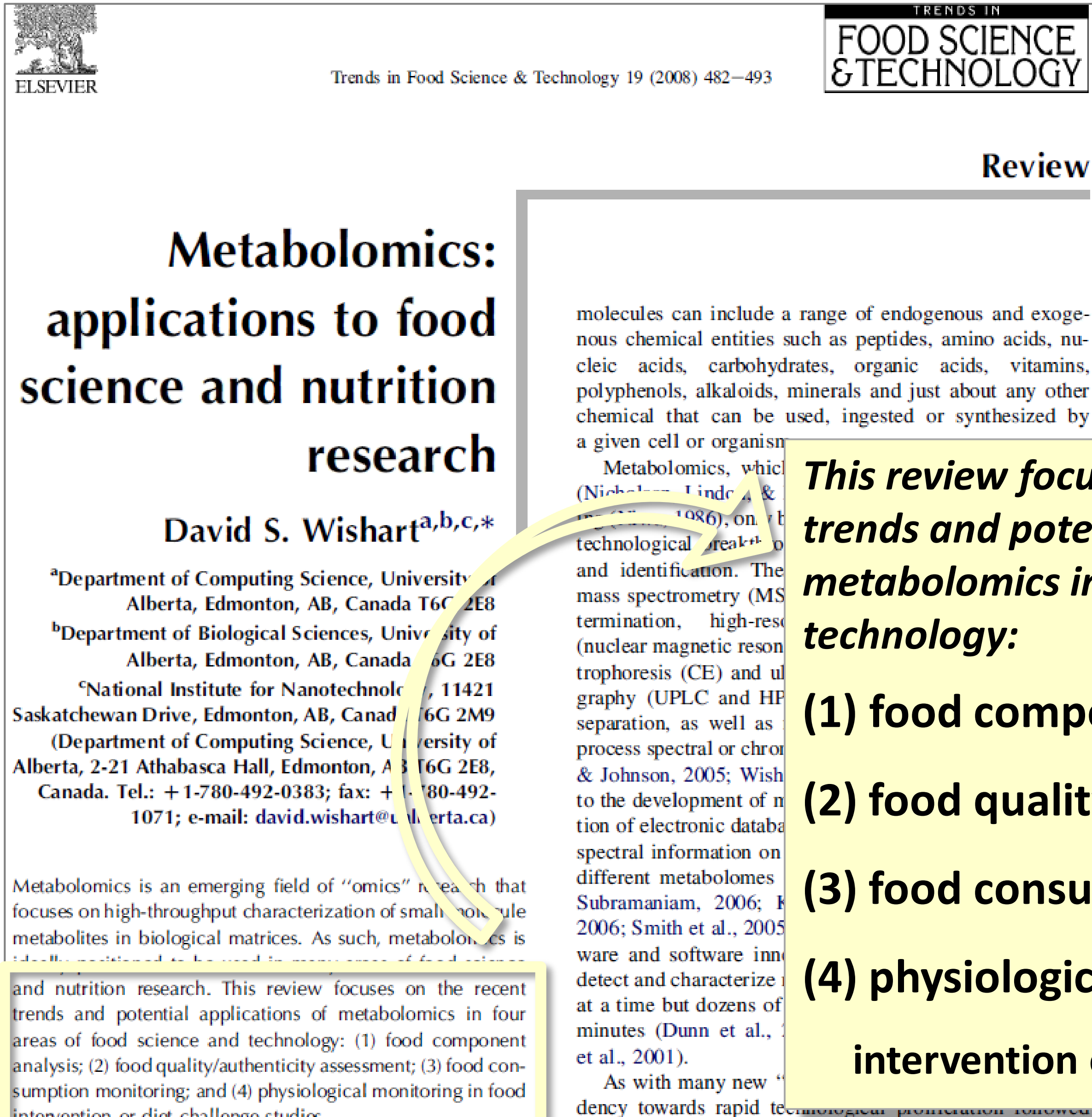
METABOLOMICS →

„OMICS“ hierarchy



Set of all small molecules (cca ≤1500 Da)

Application scope of metabolomics



ELSEVIER

Trends in Food Science & Technology 19 (2008) 482–493

TRENDS IN FOOD SCIENCE & TECHNOLOGY

Review

Metabolomics: applications to food science and nutrition research

David S. Wishart[a,b,c,*]

[a]Department of Computing Science, University of Alberta, Edmonton, AB, Canada T6G 2E8
[b]Department of Biological Sciences, University of Alberta, Edmonton, AB, Canada T6G 2E8
[c]National Institute for Nanotechnology, 11421 Saskatchewan Drive, Edmonton, AB, Canada T6G 2M9
(Department of Computing Science, University of Alberta, 2-21 Athabasca Hall, Edmonton, AB T6G 2E8, Canada. Tel.: +1-780-492-0383; fax: +1-780-492-1071; e-mail: david.wishart@ualberta.ca)

Metabolomics is an emerging field of "omics" research that focuses on high-throughput characterization of small molecule metabolites in biological matrices. As such, metabolomics is ideally positioned to be used in many areas of food science and nutrition research. This review focuses on the recent trends and potential applications of metabolomics in four areas of food science and technology: (1) food component analysis; (2) food quality/authenticity assessment; (3) food consumption monitoring; and (4) physiological monitoring in food intervention or diet challenge studies.

molecules can include a range of endogenous and exogenous chemical entities such as peptides, amino acids, nucleic acids, carbohydrates, organic acids, vitamins, polyphenols, alkaloids, minerals and just about any other chemical that can be used, ingested or synthesized by a given cell or organism...

2008

This review focuses on the recent trends and potential applications of metabolomics in four areas of food science and technology:

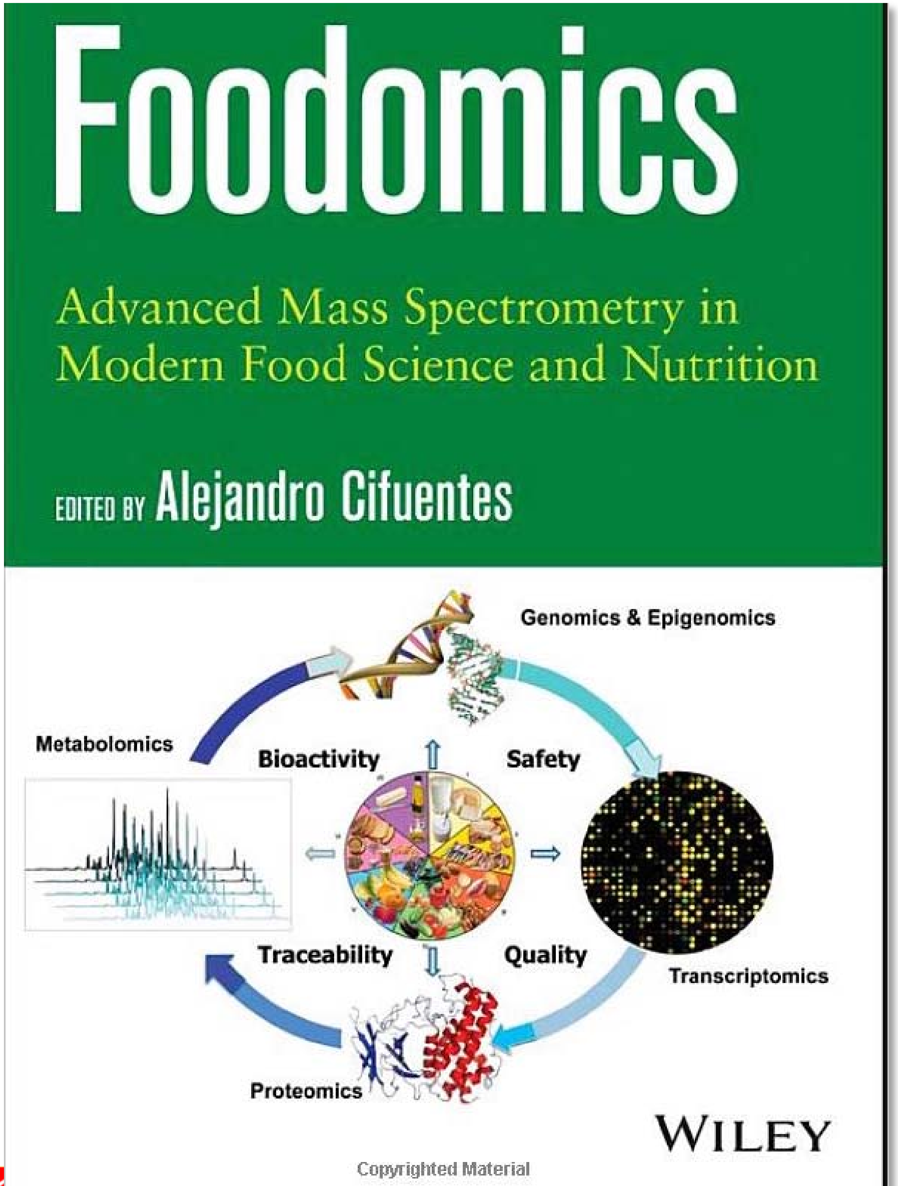
(1) food component analysis;

(2) food quality/authenticity assessment;

(3) food consumption monitoring

(4) physiological monitoring in food intervention or diet challenge studies

Foodomics: a new keyword in food analysis



2013

METABOLOMICS
(´FOODOMICS´?)

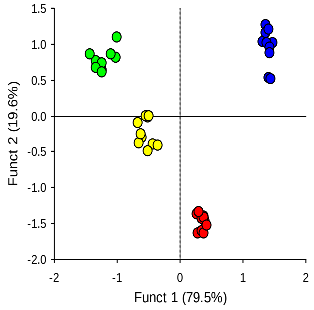
WORKFLOW: profiling / fingerprinting

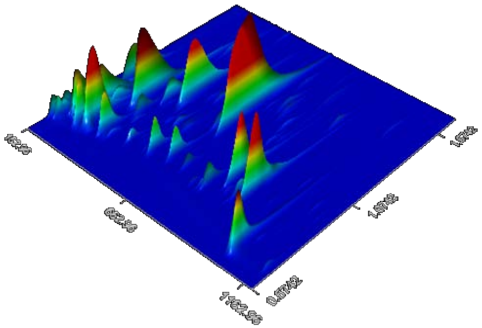
Samples

Output (*confirmation of rejection of claim/identity*)

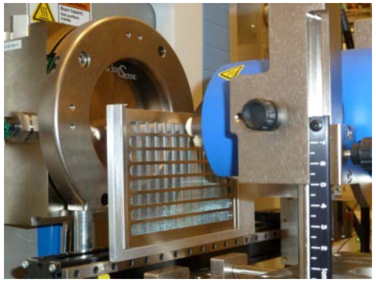
Act. Group	Pred. Group (Holdout) Group 1	Group 2	Group 3	Group 4	Correctly Classified
Group 1	10	0	0	0	1.000
Group 2	0	10	0	0	1.000
Group 3	0	0	10	0	1.000
Group 4	0	0	0	10	1.000
Overall Correct Classification Rate					1.000

Data analysis





Fingerprinting technique

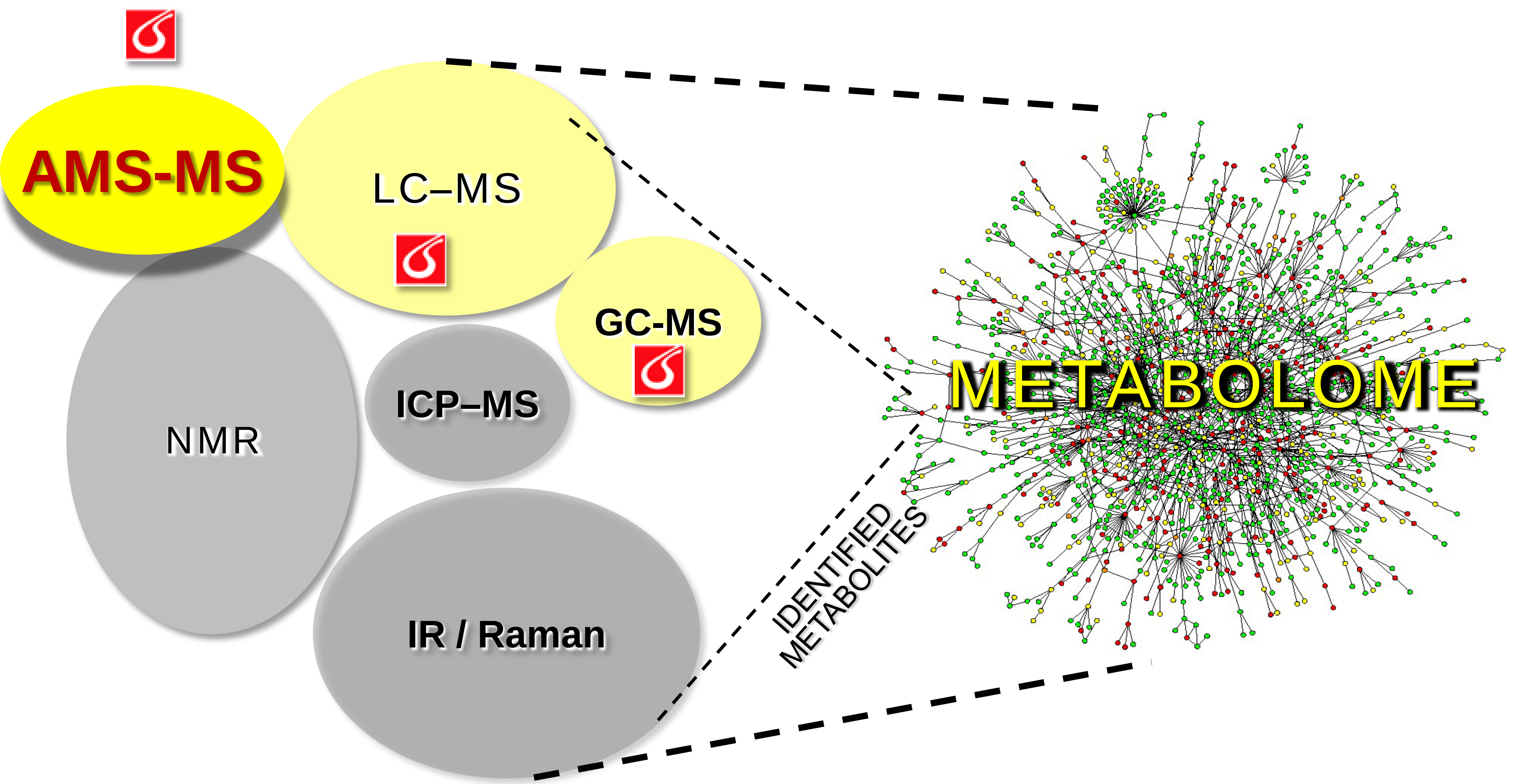


Recorded output

No.	Group	Var1	Var2	Var3	Var4	Var5	Var6	Var7	Var8	Var9	...	Var33
1	Group 1	0.488	0.158	0.217	0.564	0.069	0.242	0.115	0.064	0.019	...	0.005
2	Group 1	0.496	0.166	0.222	0.580	0.070	0.249	0.057	0.033	0.020	...	0.013
3	Group 1	0.083	0.043	0.032	0.001	0.022	0.140	0.001	0.001	0.008	...	0.002
...	...	...	...	...	...	...	...	...	...	...	...	...
11	Group 2	0.866	0.045	0.736	0.029	0.026	0.025	0.000	0.004	0.010	...	0.011
12	Group 2	0.853	0.043	0.703	0.028	0.026	0.024	0.000	0.004	0.009	...	0.000
13	Group 2	0.724	0.027	1.000	0.016	0.043	0.049	0.000	0.003	0.007	...	0.014
...	...	...	...	...	...	...	...	...	...	...	...	...
21	Group 3	0.264	0.098	0.060	0.015	0.028	0.153	0.000	0.001	0.015	...	0.006
22	Group 3	0.273	0.097	0.061	0.015	0.029	0.154	0.000	0.001	0.015	...	0.005
23	Group 3	0.435	0.016	0.010	0.000	0.012	0.165	0.000	0.001	0.002	...	0.000
...	...	...	...	...	...	...	...	...	...	...	...	...
38	Group 4	1.000	0.008	0.002	0.000	0.020	0.092	0.005	0.004	0.001	...	0.014
39	Group 4	0.783	0.007	0.000	0.005	0.045	0.036	0.000	0.002	0.000	...	0.022
40	Group 4	0.849	0.000	0.000	0.006	0.051	0.038	0.000	0.004	0.000	...	0.026

Data matrix

Instrumental platforms for metabolomics

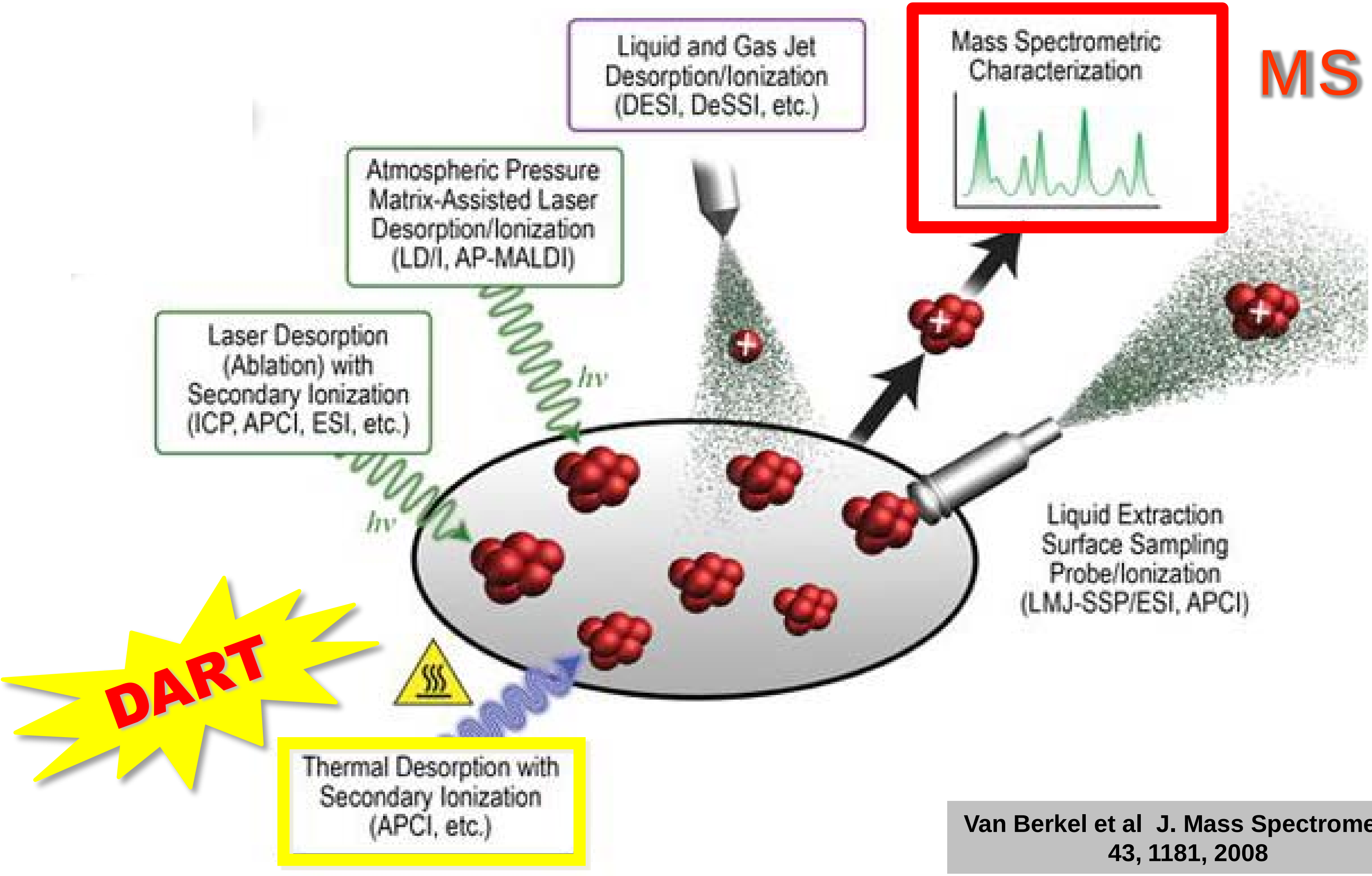


AMBIENT MASS SPECTROMETRY (AMS)

A new challenge for our labs?

ICT PRAGUE

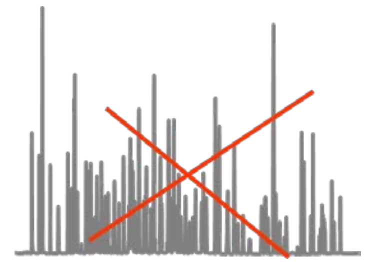
AMBIENT MASS SPECTROMETRY OPTIONS



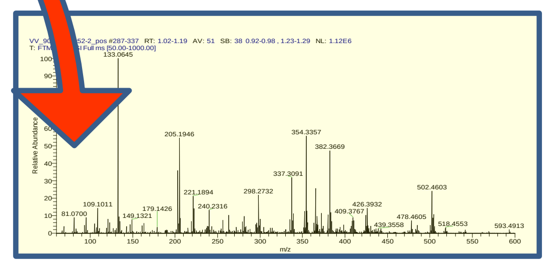
Van Berkel et al J. Mass Spectrometry, 43, 1181, 2008

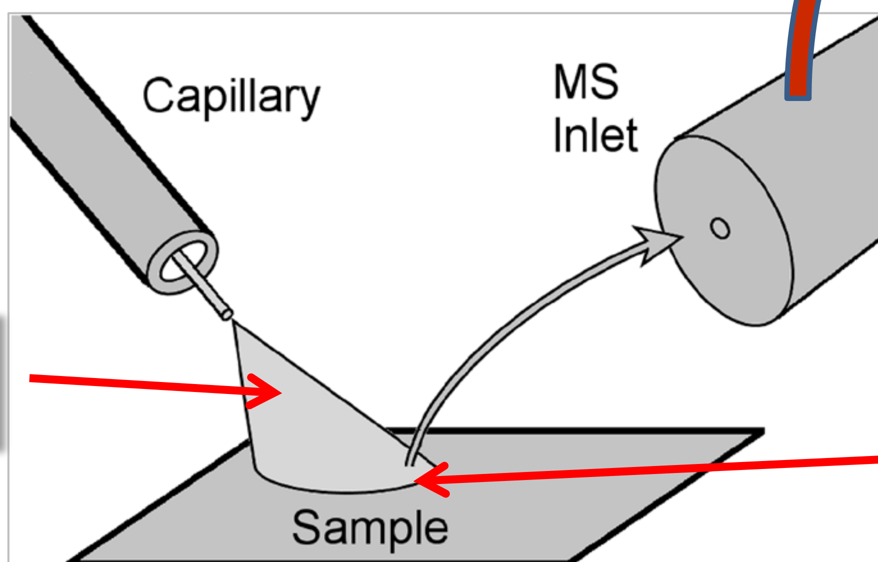
Novel fingerprinting technique:

AMBIENT MASS SPECTROMETRY



Mass spectrum



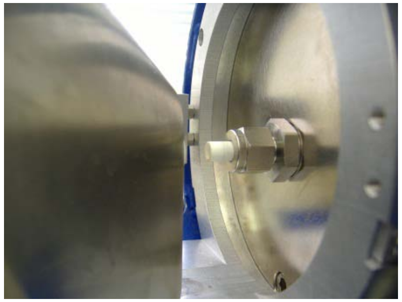


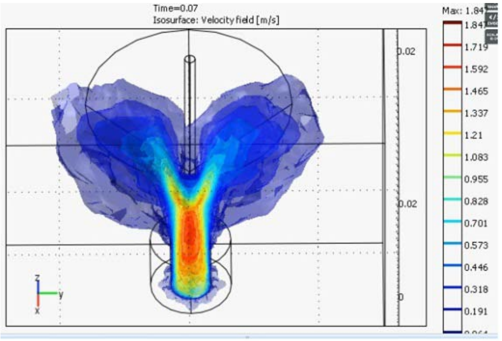
excited He
200- 400°C

THERMO DESORPTION,
APCI ionization

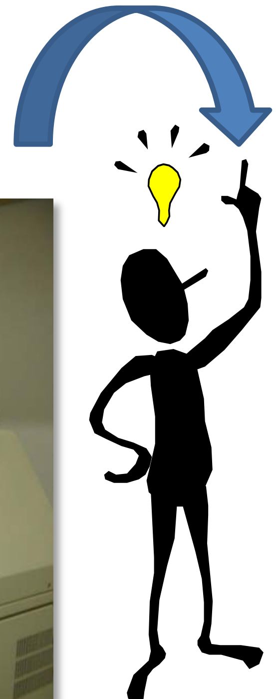
Steps occurring in DART together, or separately, under the influence of a particular agent:

- **DESORPTION** – a change in phase (e.g., solid to vapor)
- **IONIZATION** - an acquisition of charge by neutral analyte molecules





DART – HRMS has become an important tool in routine food analysis / food research



DART–TOFMS

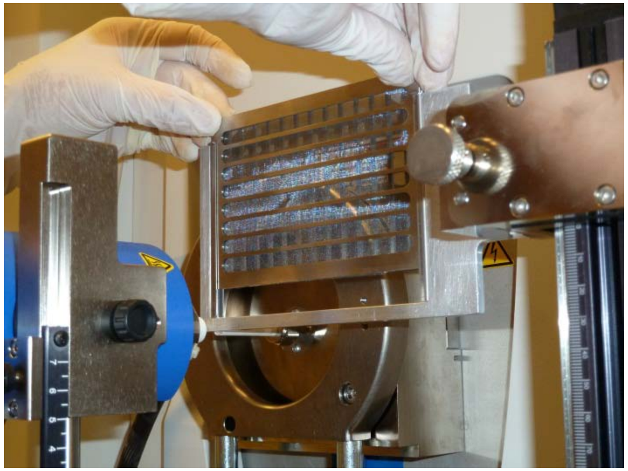
AccuTOF LP (Jeol)
Time-of-flight mass spectrometer
~ 5000 – 7000 fwhm

DART–orbitrapMS

Exactive (Thermo Scientific)
Orbitrap mass spectrometer
~ 10,000 – 100,000 fwhm

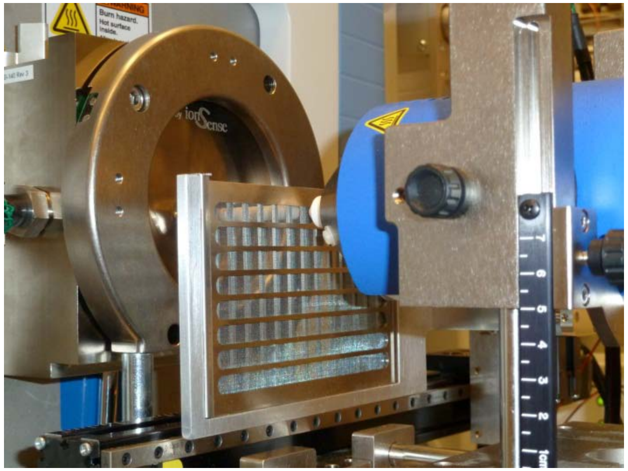
X-Z TRANSMISSION MODULE AUTOSAMPLER

(2)



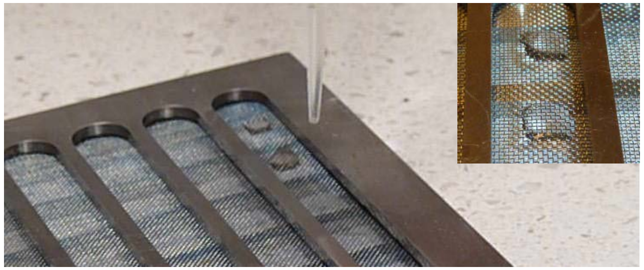
Metal mesh is placed into a holder

(3)

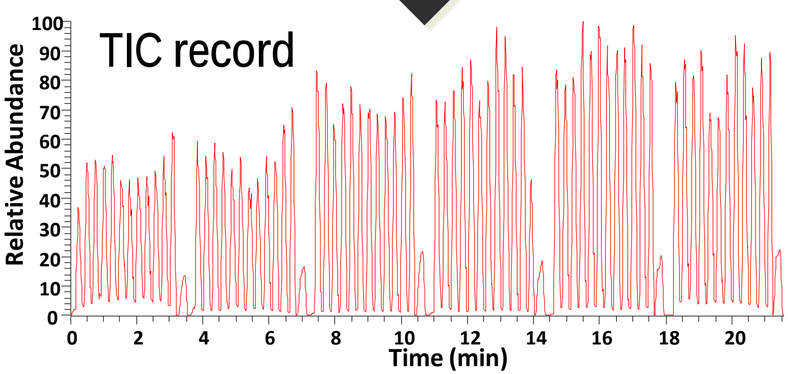


Gas flows through the metal mesh, desorbs and ionizes sample components.

(1)



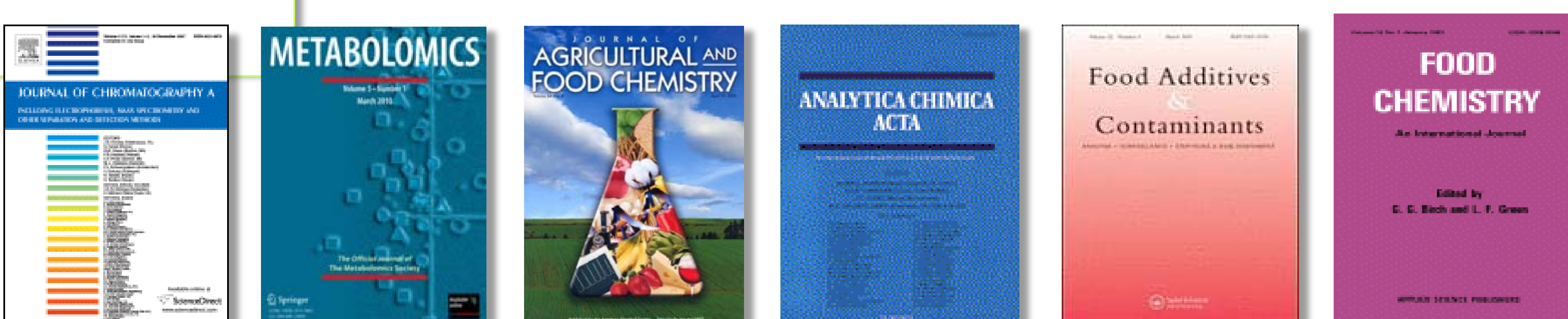
Sample (5 µl) is placed on metal mesh.



METABOLOMICS-BASED CASE STUDIES

Matebolomics based studies we have performed

- Adulteration of olive oil by hazelnut oil
- Fish feeding history authentication
- Classification of bear origin, traceability
- Identification of geografic origin of honey
- Authentication of fruit jjuice freshness
- Wines authentication
- Authentication of apple varienty and the way of farming



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NON- TARGET ANALYSIS:

Wine authentication

WINE AUTHENTICATION TOOLS

FORMER TRENDS

Physico-chemical and/or biochemical measurements

Single or only a few markers

CURRENT TRENDS

FINGERPRINTING / PROFILING

Comprehensive information on sample composition

METHODS FOR WINE AUTHENTICATION

HPLC–UV-VIS /–MS
(phenolic compounds)

Infrared spectroscopy

SPME–GC–FID
SPME–GC–MS
SPME–GC×GC–MS
(volatile compounds)

DNA (biomethods)

^{13}C NMR

ASAP–MS
DART–MS
(ionizable compounds)

AAS nebo ICP-MS
(minerals)

LC–MS
(polar compounds)

GC–FID
(alcohols)

AN OVERVIEW OF ANALYZED SAMPLES (WHITE WINES)

- **Chardonnay (*n* = 20)**
- **Müller Thurgau (*n* = 20)**
- **Rhine Riesling (*n* = 15)**

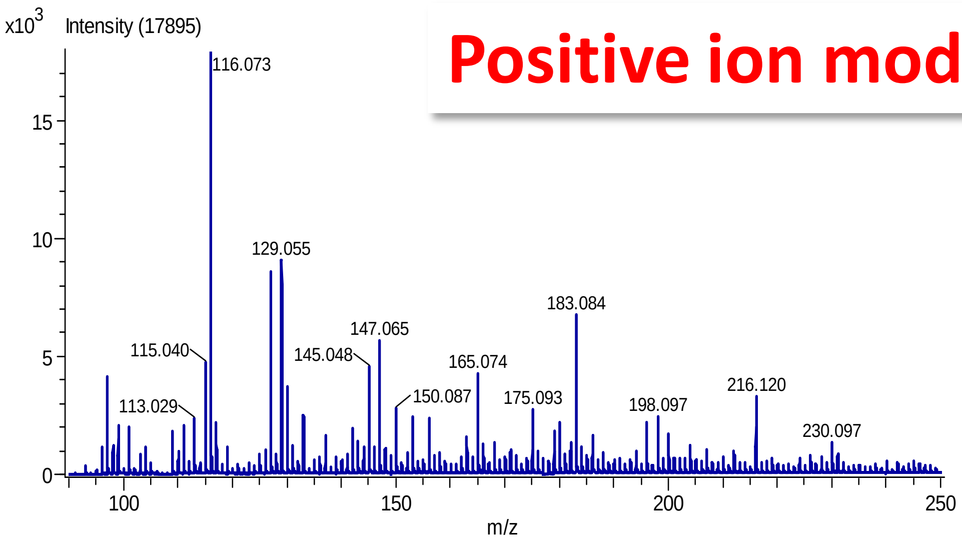
- **Origin of the samples: Australia, Austria, Bulgaria, Chile, Czech Republic, Hungary, Italy, Moldavia, South Africa, USA**
- **Years of the production: 2005–2008**

AN OVERVIEW OF ANALYZED SAMPLES (RED WINES)

- Cabernet Sauvignon (*n* = 15)
- Merlot (*n* = 16)
- Pinot Noir (*n* = 14)

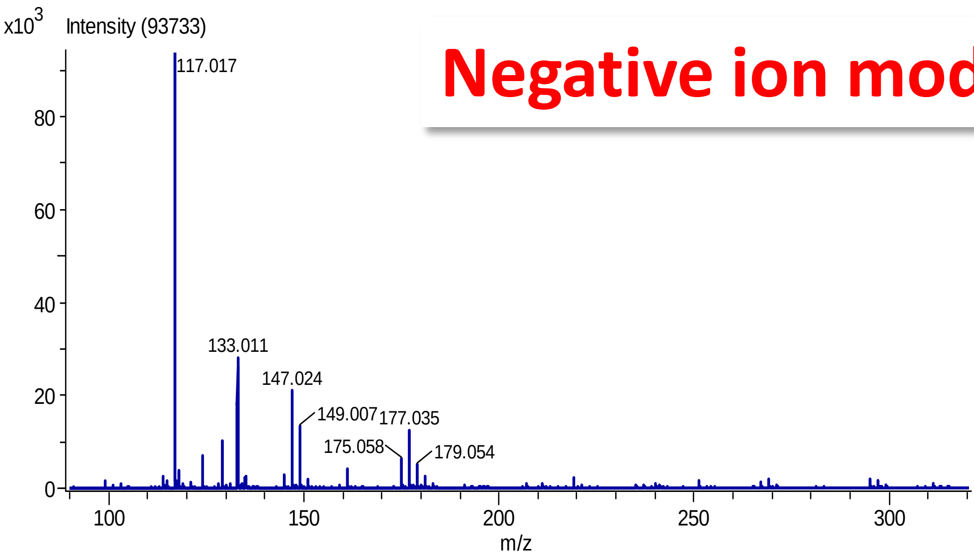
- Origin of the samples: Australia, Bulgaria, Czech Republic, France, Germany, Hungary, Chile, Makedonia, Slovakia, Spain, USA
- Years of the production: 2004–2008

DART–MS: Direct measurement of wine



Positive ion mode

Negative ion mode



Example: Cabernet Sauvignon

CHEMOMETRIC ANALYSIS (wine directly measured)

Model: Cabernet Sauvignon *vs.* Merlot

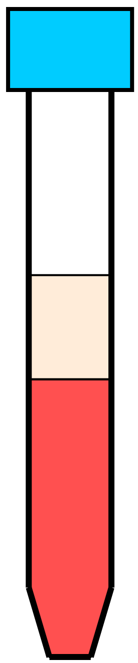
25 markers
(DART(+)–MS, dir. meas.)
(*m/z*)
↓
PLS-LDA + model validation (LOOCV)
↓
6 latent variables (LVs)
↓
Recognition ability: 100%
Prediction ability: 96.0%

14 markers (
(DART(-)–MS, dir. meas.)
(*m/z*)
↓
PLS-LDA + model validation (LOOCV)
↓
5 latent variables (LVs)
↓
Recognition ability: 88.0%
Prediction ability: 88.0%

DART: Direct measurement of phenolics fraction isolated by EtOAc extraction

Ethyl acetate (3 mL)

wine (3 mL) & added water (3 mL)



Shaking

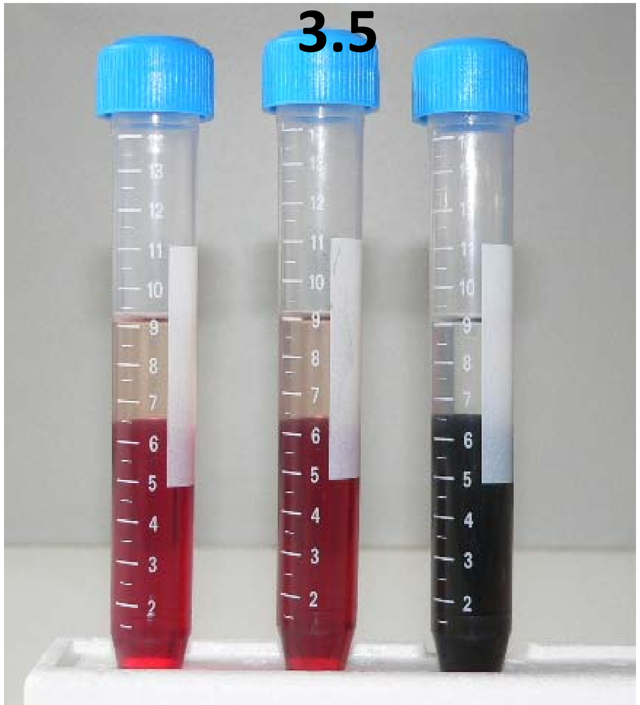
2 min

Centrifugation

11,000 rpm

5 min

pH = 2   pH = 3.5   pH = 7



DART analysis of EtOAc fraction

PHENOLIC COMPOUNDS

Phenolic acids

p-Hydroxybenzoic; *o*-hydroxybenzoic; salicylic; gallic; cinnamic; *p*-coumaroylartaric (= coutaric); caffeoyltartaric (= caftaric); feruloylartaric (= fertaric); *p*-coumaroyl glucose; feruloylglucose; glucose ester of coutaric acid

Anthocyanins

Cyanidin 3-glucoside; cy 3-acetylglucoside; cy 3-*p*-coumaryl-glucoside; peonidin 3-glucoside; pn 3-acetylglucoside; pn 3-*p*-coumarylglucoside; pn 3-caffeylglucoside (?); delphinidin 3-glucoside; dp 3-acetylglucoside; dp 3-*p*-coumarylglycoside; petunidin 3-glucoside; pt 3-*p*-coumarylglucoside; malvidin 3-glucoside; mv 3-acetylglucoside; mv 3-*p*-coumaryglucoside; mv 3-caffeylglucoside

Flavonols

Kaempferol 3-glucoside; k 3-glucuronide; k 3-glucosylarabinoside (?); k 3-galactoside; quercetin 3-glucoside; q 3-glucoronide; q 3-rutinoside; q 3-glucosylgalactoside (?); q 3-glucosylxyloside (?); iso-rhamnetic 3-glucoside

Flavan-3-ols and tannins

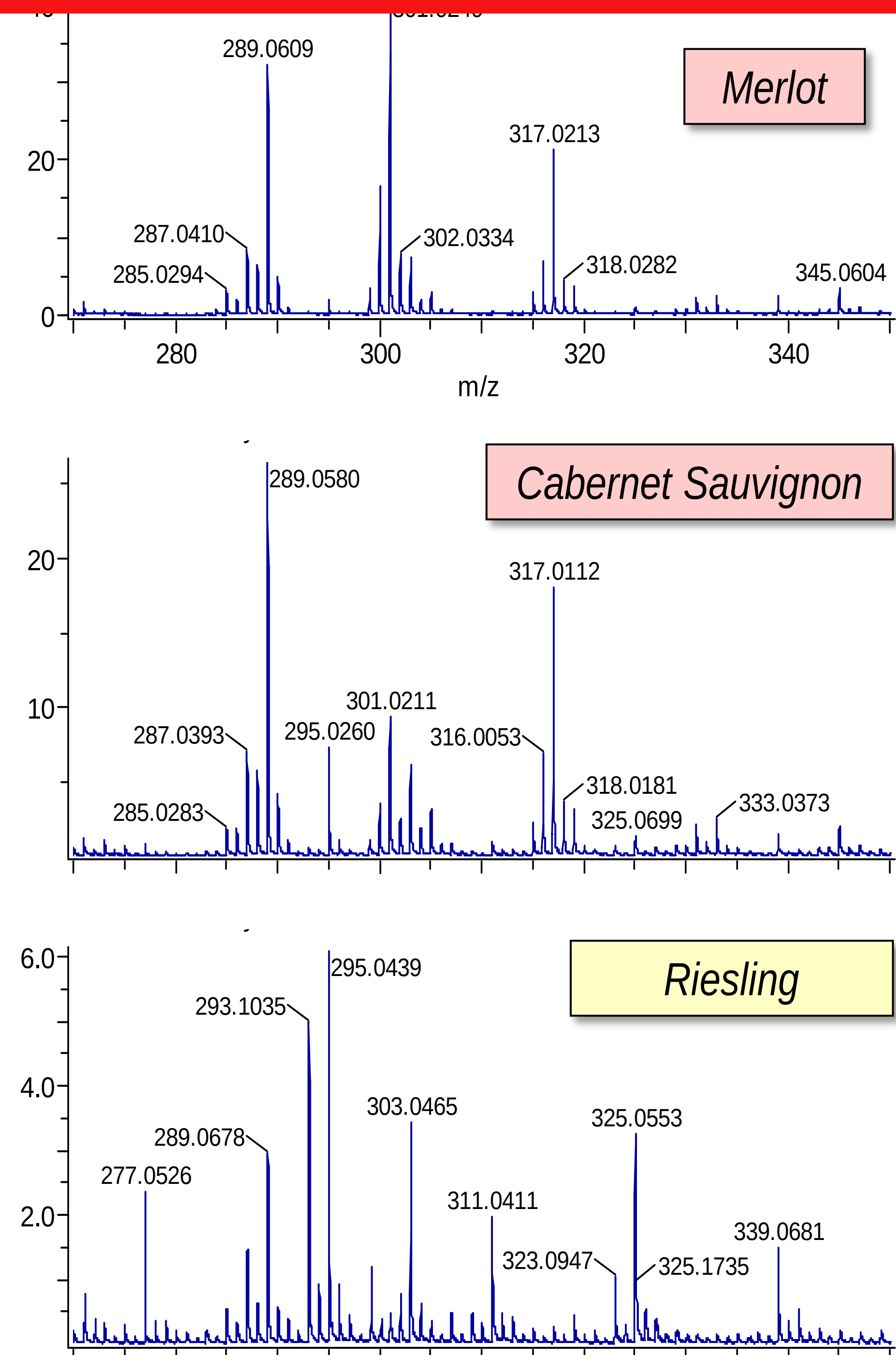
(+)Catechin; (–)epicatechin; (+)gallocatechin; (–)epigallocatechin; epicatechin-3-O-gallate; procyanidins B1, B2, B3, B4, C1, C2, polymeric forms of condensed tannins

Flavanonols

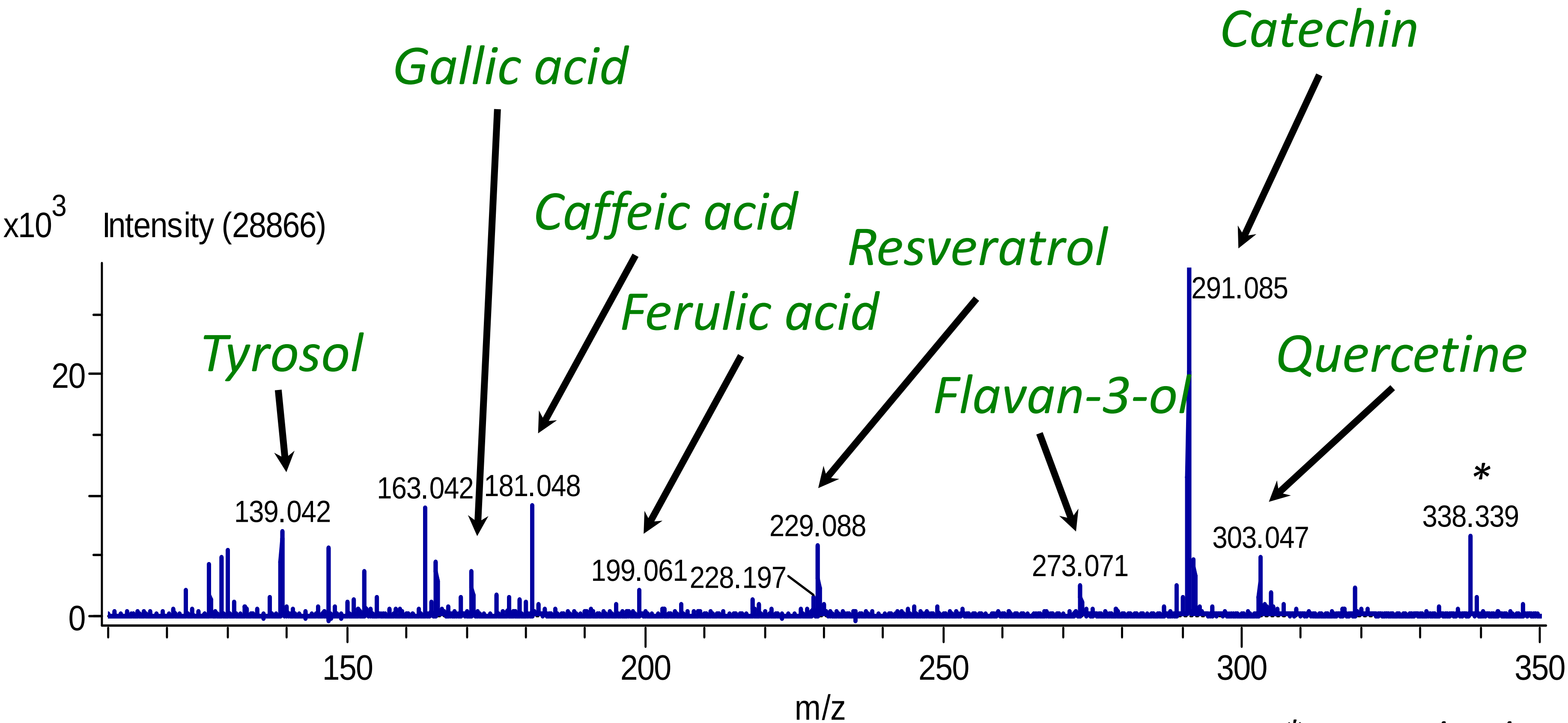
Dihydroquercetin 3-rhamnoside (= astilbin); dihydrokaempferol 3-rhamnoside (= engeletin)

DART [–]: PROFILING of phenolics fraction isolated by EtOAc extraction

m/z 260–350
↓
High-MW phenolics fraction



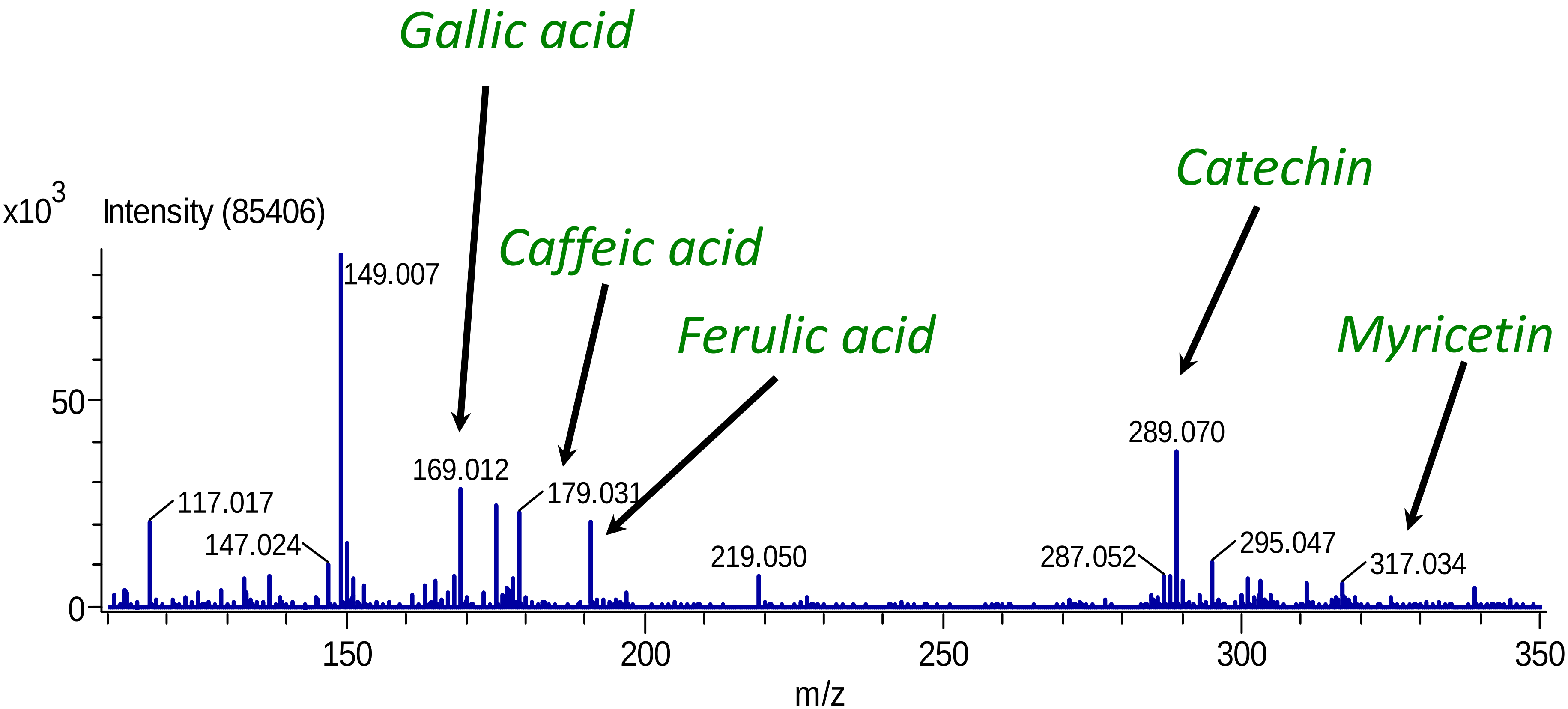
DART(+) measurement of phenolics fraction - identification based on exact mass



Example: Cabernet Sauvignon

** contamination*

DART(–) measurement of phenolics fraction - identification based on exact mass



Example: Cabernet Sauvignon

CHEMOMETRIC ANALYSIS (based on phenolics)

Model: Cabernet Sauvignon *vs.* Merlot

25 markers (DART(+)–MS, EtOAc fr.)
(*m/z*)
↓
PLS-LDA + model validation (LOOCV)
↓
5 latent variables (LVs)
↓
Recognition ability: 100%
Prediction ability: 100%

27 markers (DART(–)–MS, EtOAc fr.)
(*m/z*)
↓
PLS-LDA + model validation (LOOCV)
↓
5 latent variables (LVs)
↓
Recognition ability: 100%
Prediction ability: 100%

CHEMOMETRIC ANALYSIS

Model: Chardonnay *vs*. Müller Thurgau *vs*. Rhine Riesling

28 markers (DART(-)–MS, EtOAc fr.) **(*m/z*)**

↓

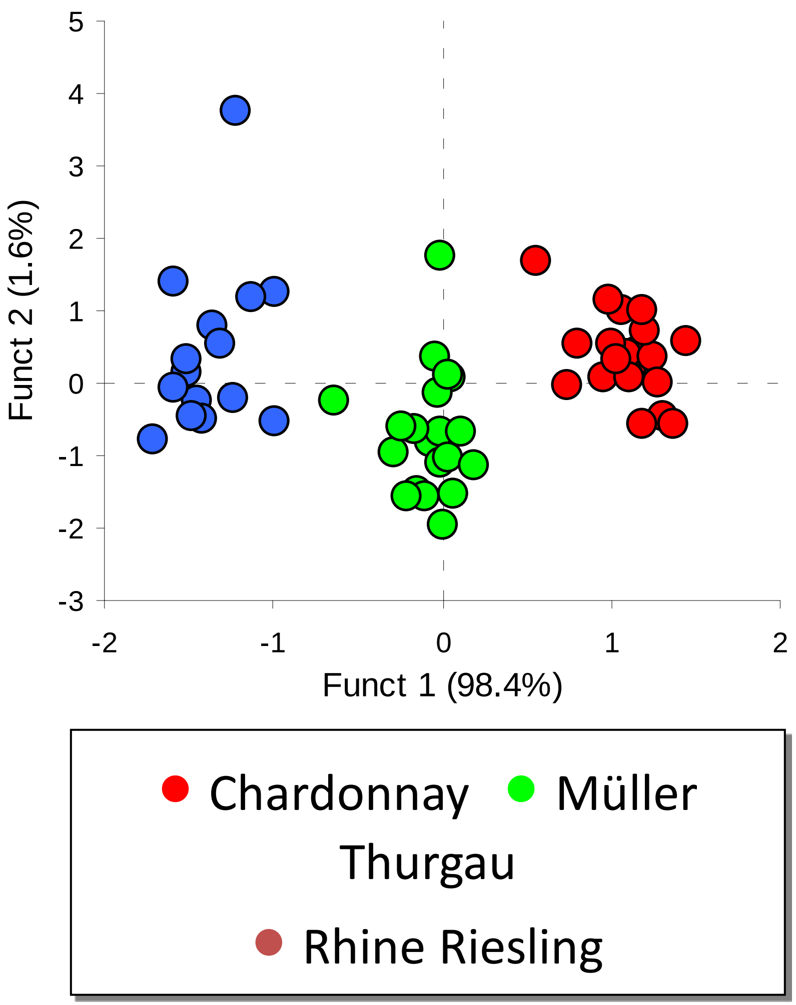
PLS-LDA + model validation (LOOCV)

↓

9 latent variables (LVs)

↓

Recognition ability: 98.9%
Prediction ability: 93.6%



RECENT REVIEW SUMMARIZING ICT EXPERIENCE

8 December 2010; Disk Used
Trends in Analytical Chemistry, Vol. xxx, No. x, 2010 Trends

Challenging applications offered by direct analysis in real time (DART) in food quality and safety analysis

Jana Hajslova, Tomas Cajka, Lukas Vaclavik

Direct analysis in real time (DART) is an ambient ionization technique undergoing a rapid development. With a minimal sample pre-treatment, ionization of analyte molecules outside the mass spectrometer (MS) in the ordinary atmosphere is feasible. This ionization approach relies upon fundamental principles of atmospheric pressure chemical ionization (APCI). The current review

ANOTHER CHALLENGE : LC-MS, direct injection

- To avoid any possible discrimination of metabolites a **direct injection** of wine samples was carried out.

Liquid Chromatography (UHPLC Ultimate 3000 Dionex-Thermo)

- Different mobile phases and modifiers were tested:
  - Formic acid
  - Ammonium acetate
  - Ammonium formate

Two aqueous mobile phase components were used with methanol for elution during acquisition in positive and negative mode.

- **ESI +** The lower pH (3.8) eluent containing **ammonium formate** was employed in positive ion mode to support protonation of eluted molecules and ammonium adducts $[M+NH_4]$.

- **ESI -** Higher pH (4.5) value was used in negative ion mode (**ammonium acetate**) and $[M+CH_3\ COO]^-$ could be detected.

Liquid Chromatography (UHPLC Ultimate 3000 Dionex-Thermo)

- Different columns were tested:
  - C18 (RPLC)
  - HILIC

1. The reversed phase chromatographic system enabled rapid separation of a wide range of compounds present in wine samples.

2. The use of stationary phase with sub-2 µm particles significantly increased column separation efficiency and chromatographic resolution.

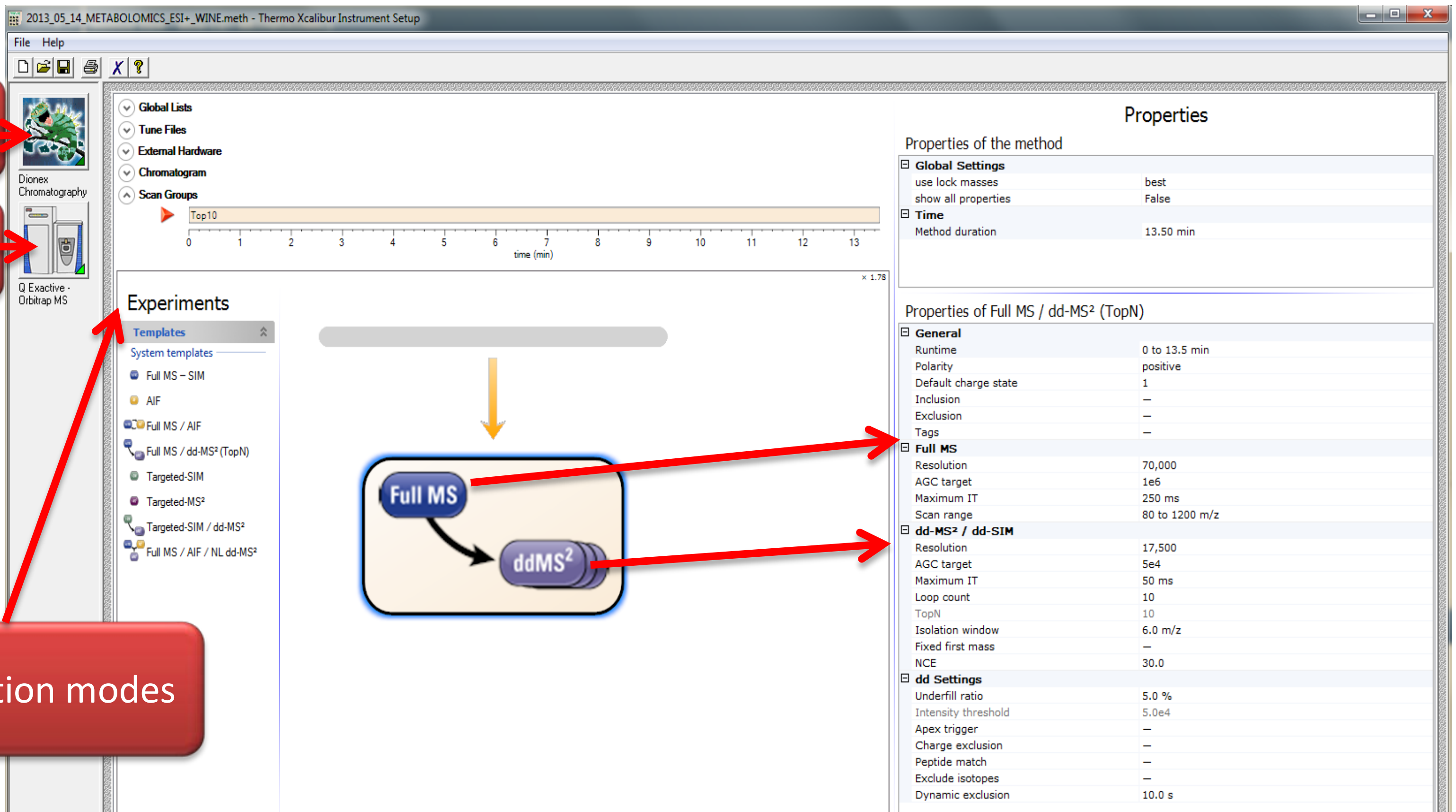
ICT PRAGUE

UHPLC-Q Exactive

The Thermo Scientific Q Exactive benchtop UHPLC-MS/MS combines high-performance quadrupole precursor selection with high-resolution, accurate-mass (HRAM) Orbitrap detection to deliver high performance and tremendous versatility.

- Resolving power up to 140,000
- Maximum scan speed 12Hz
- Intra-scan dynamic range > 5000:1
- Quadrupole mass filter
- Spectral multiplexing for enhanced duty cycle
- S-Lens ion source for increased sensitivity

UHPLC-Q Exactive: Method development



LC settings

MS settings

Acquisition modes

FULL MS-ddMS²

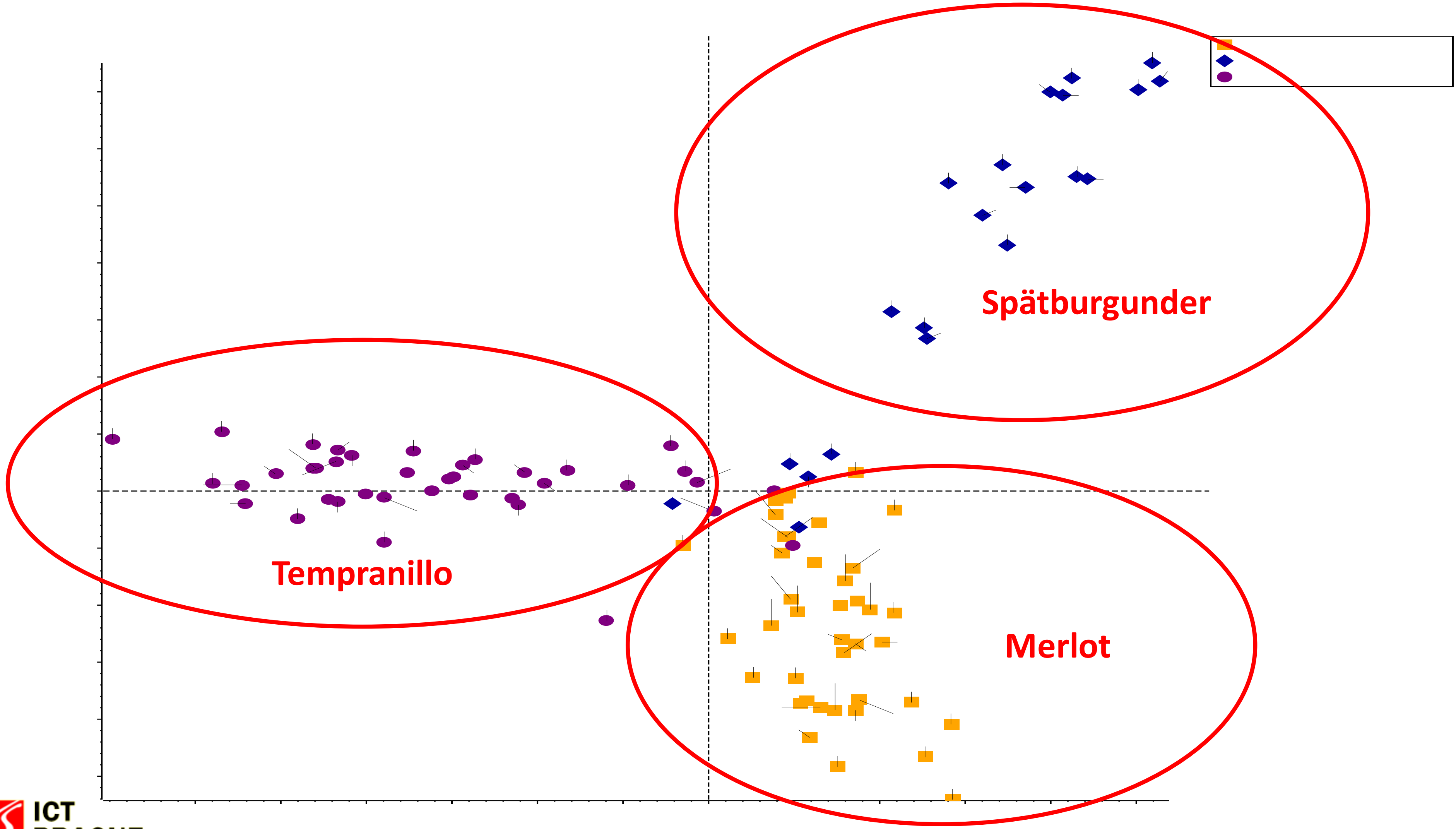
This acquisition method consists in a high resolution MS survey scan followed by MS/MS using a data depending settings.

Benefits of Q-Exactive: Metabolomics

- The superior quality of Q Exactive MS/MS data enables identification and quantitation of more compounds with greater confidence.
- Find the real differences in components across multiple sample groups with SIEVE 2.0

Chemometrics analysis: Red wine samples (PCA-DA ESI+)

Principal Component Analysis-Discriminant Analysis (PCA-DA). PCA calculates the best discriminating components without foreknowledge about groups, whereas discriminant analysis calculates the best discriminating components (= discriminants) for groups that are defined by the user.



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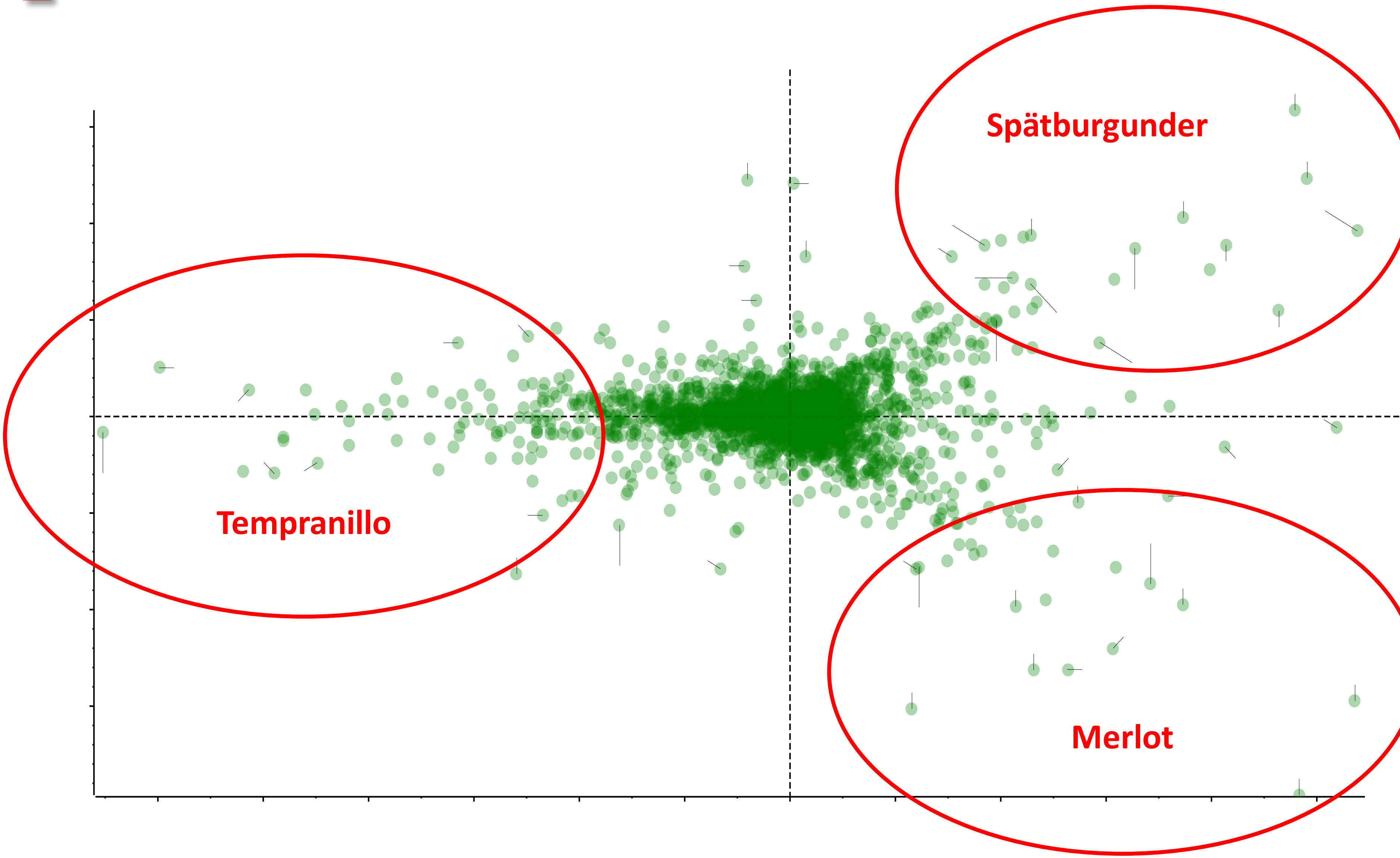
Chemometrics analysis: red wine samples (PCA-DA ESI+)



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Identification of markers: Workflow

- Identification of markers using PCA discriminant analysis.
- Xcalibur®:
  - **Full MS** extraction ion chromatogram and estimation of molecular formula.
  - **dd-MS²** data depending acquisition (Pathway)
- Libraries:

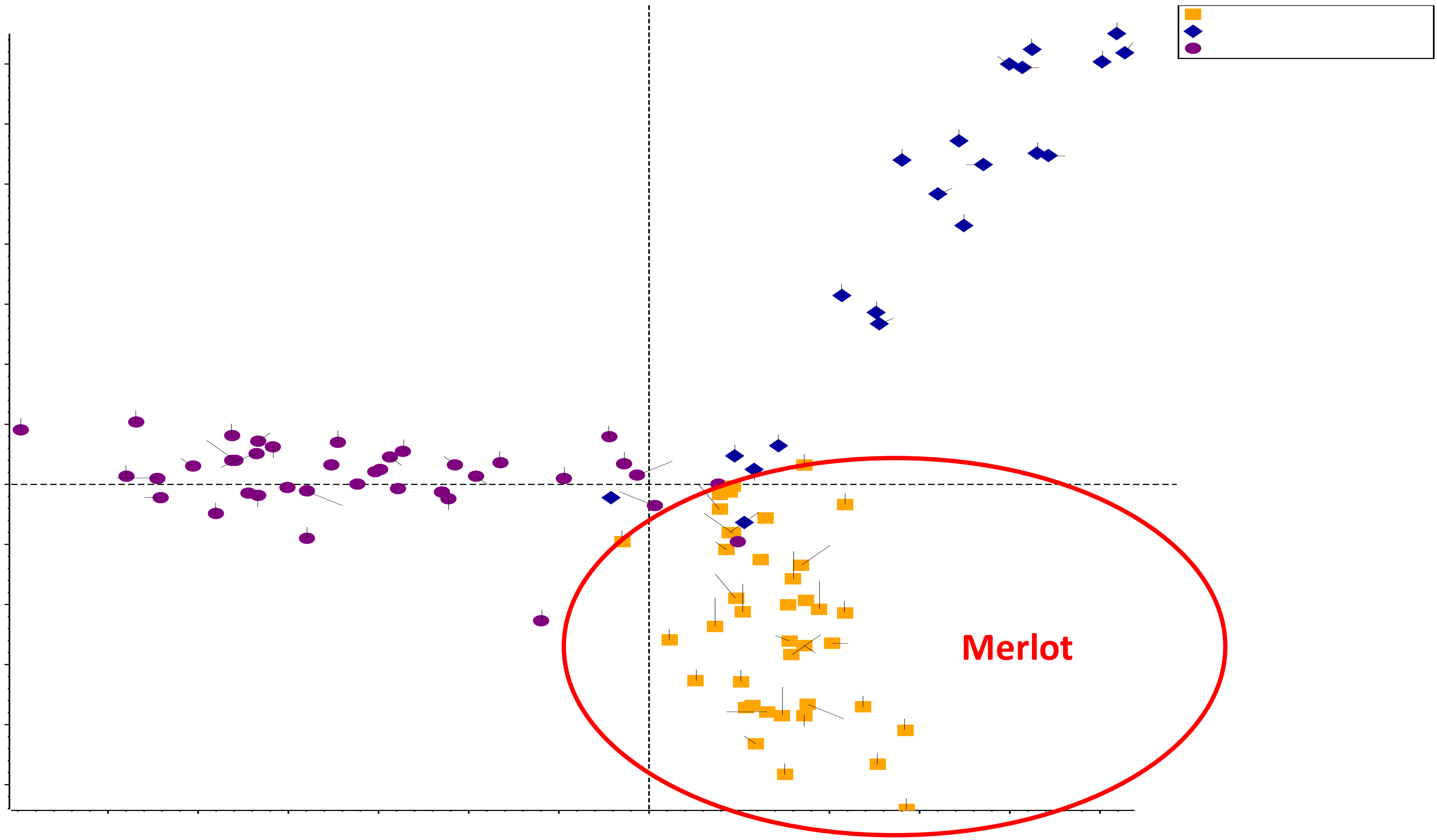
ChemSpider
The free chemical database

MassBank

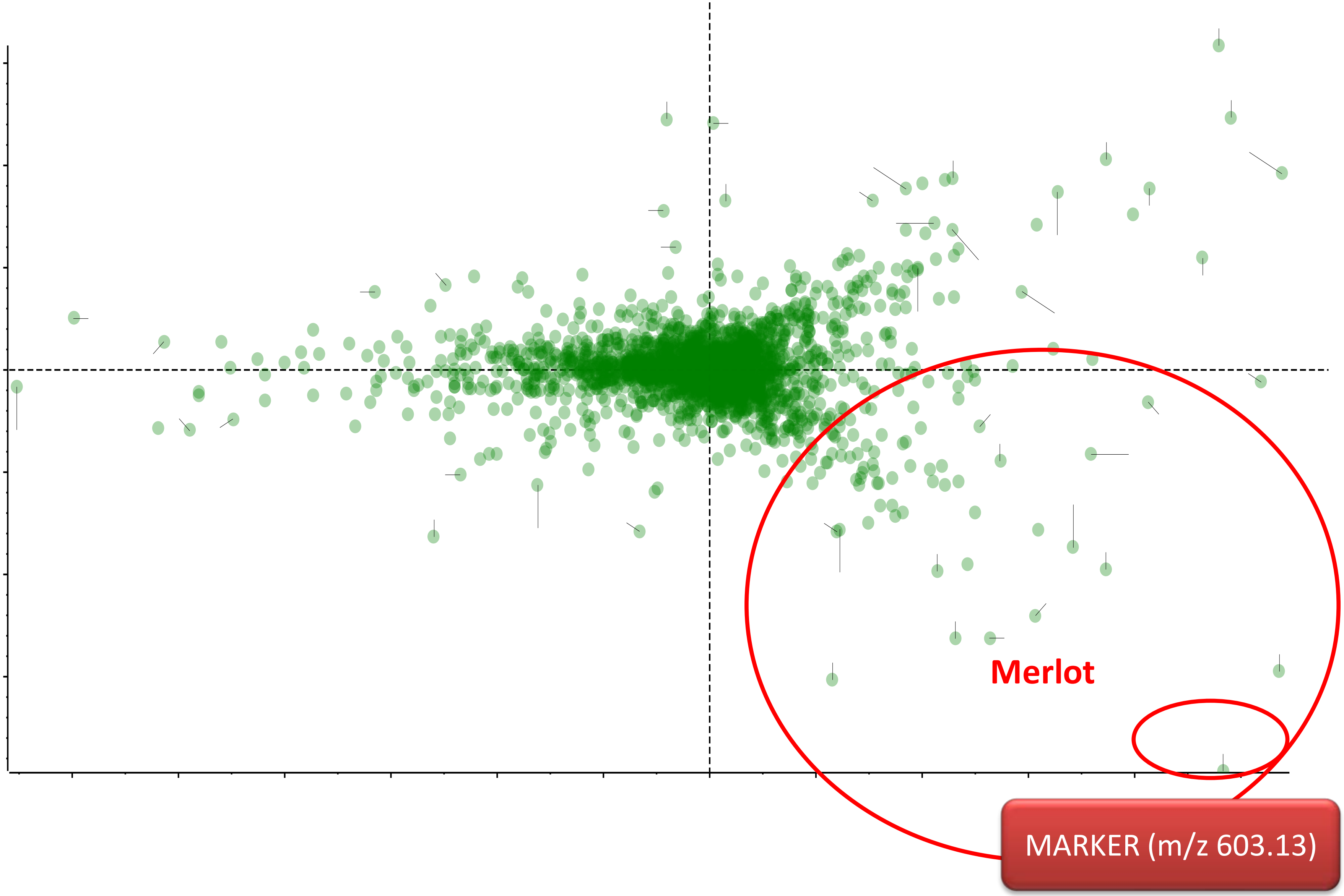
hmp
human metabolome project

Scripps Center For Metabolomics
METLIN: Metabolite and Tandem MS Database

Chemometrics analysis: Red wine samples (PCA-DA ESI+)

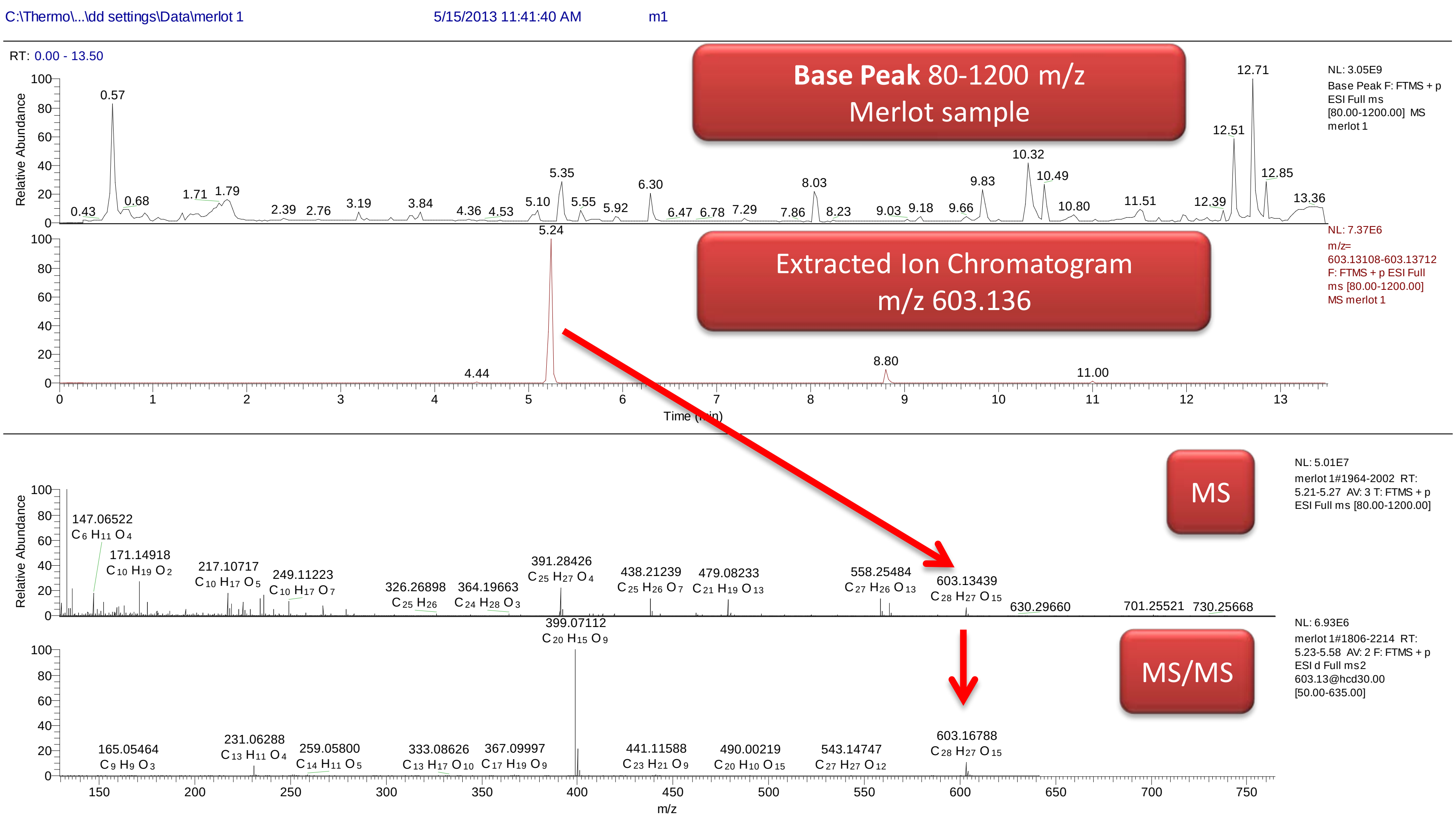


Chemometrics analysis: Merlot markers



ICT PRAGUE

Chemometrics analysis: Merlot markers (Workflow)

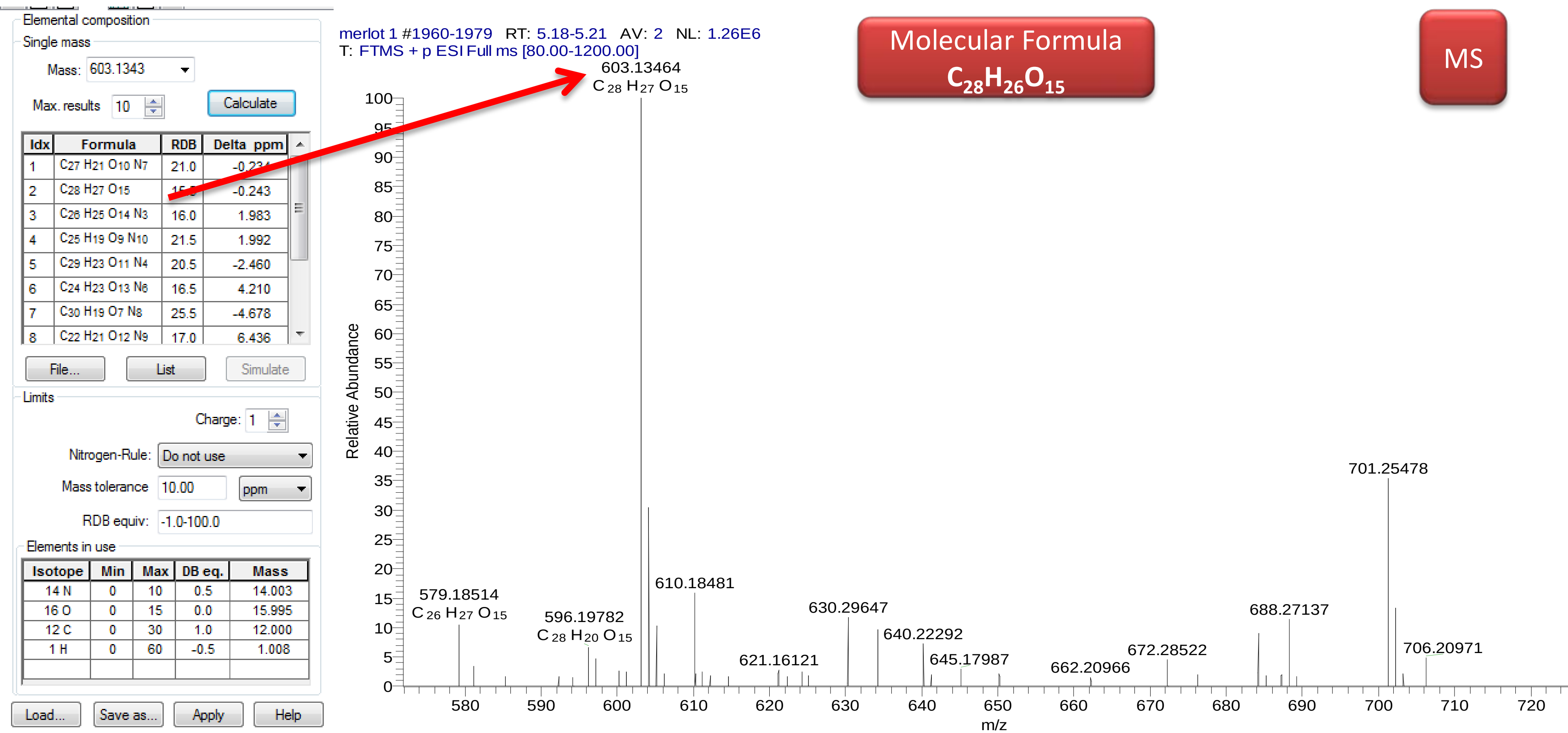


ICT PRAGUE

Chemometrics analysis: Merlot markers (Workflow)

Molecular Formula $C_{28}H_{26}O_{15}$

MS



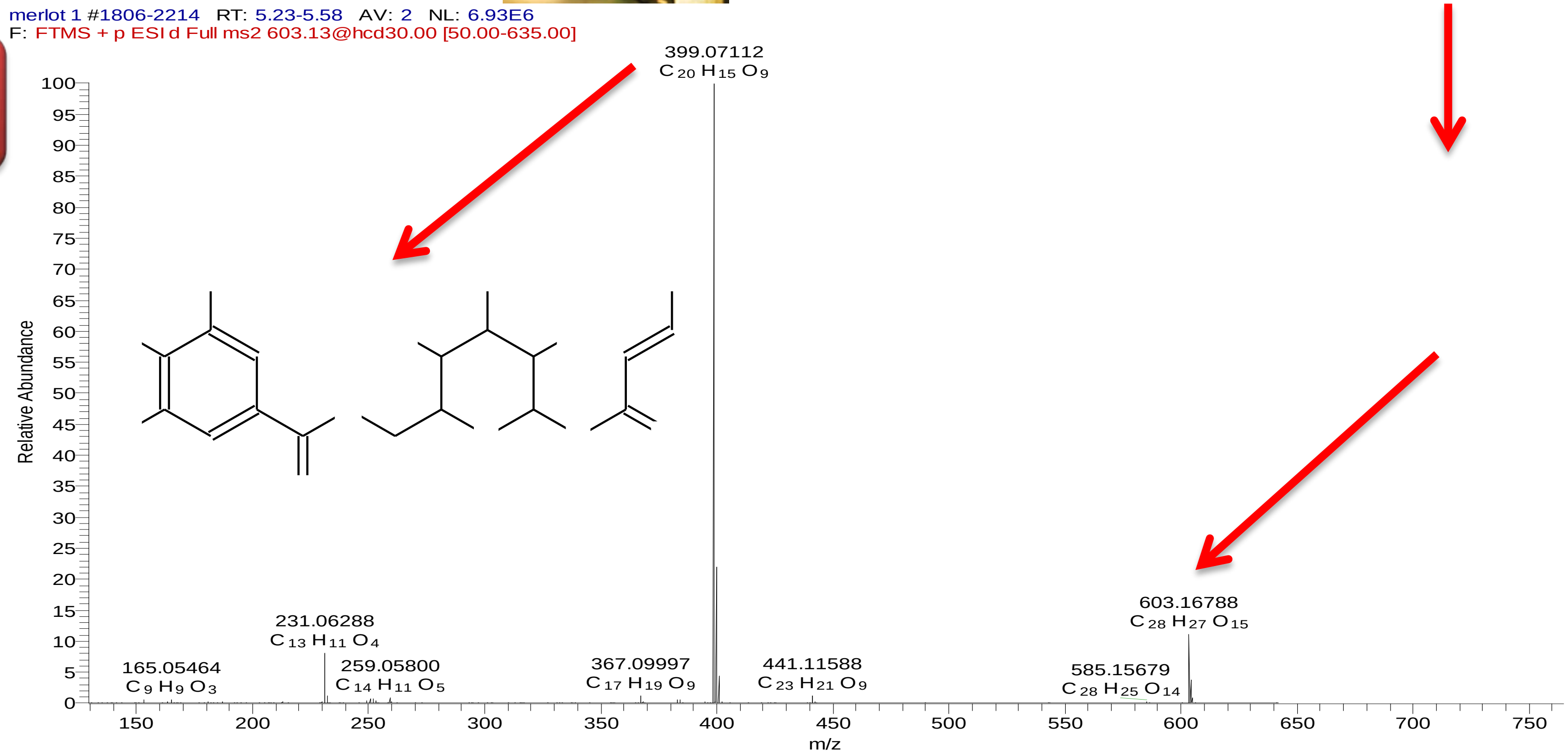
Chemometrics analysis: Merlot markers

Eriodictyol 7-(6-galloylglucoside) is found in fruits. Eriodictyol 7-(6-galloylglucoside) could be a constituent of the leaves and branches of *Vitis Vinifera.*

Scripps Center For Metabolomics
METLIN: Metabolite and Tandem MS Database

Eriodictyol 7-(6-galloylglucoside)
$C_{28}H_{26}O_{15}$
Flavonoid Glycosides

dd-MS[2]



Conclusions

- Q Exactive is well suited for routine in-depth metabolome analysis with fast high-resolution MS and MS/MS scan rate, and high quality HR/AM HCD spectrum.

- The applicability of UHPLC-Q Exactive for WINE (-omics) has been demonstrated, providing authentication of grape varieties.

Future trends

- Chemiometrics analysis:
  - ESI- data PCA and PCA-DA
  - ESI+ and ESI- data using SIMCA (PLS-DA, OPLS-DA and O2PLS-DA)
- Authentication:
  - Grape varieties
  - Origin and/or area
  - Quality

ASSESSMENT OF OILS AND FAT OXIDATION EXTENT




CHANGES OF FATS /OILS DURING PROCESSING

ALTERATION	CAUSATIVE AGENT	COMPOUNDS ORIGINATED
HYDROLYSIS	moisture	• Fatty acids • Diacylglycerols
OXIDATION	air	• Oxidized monomeric triacylglycerol • Oxidized dimeric and oligomeric triacylglycerols • Volatile compounds (aldehydes, ketones, alcohols, hydrocarbons, etc.)
THERMAL CHANGES	temperature	• Cyclic monomeric triacylglycerols • Isomeric monomeric triacylglycerols • Nonpolar dimeric and oligomeric triacylglycerols

AUTOOXIDATION OF FATS / OILS

- **Accumulation of oxidation products in oils / fats and fried foods**
- **Adverse health effects of oxidation products upon ingestion**

Initiation

$$RH \rightarrow R^{\cdot} + H^{\cdot}$$

$$R^{\cdot} + O_2 \rightarrow ROO^{\cdot}$$

Propagation

$$ROO^{\cdot} + R_1H \rightarrow ROOH + R_1^{\cdot}$$

Termination

$$R^{\cdot} + R^{\cdot} \rightarrow RR$$

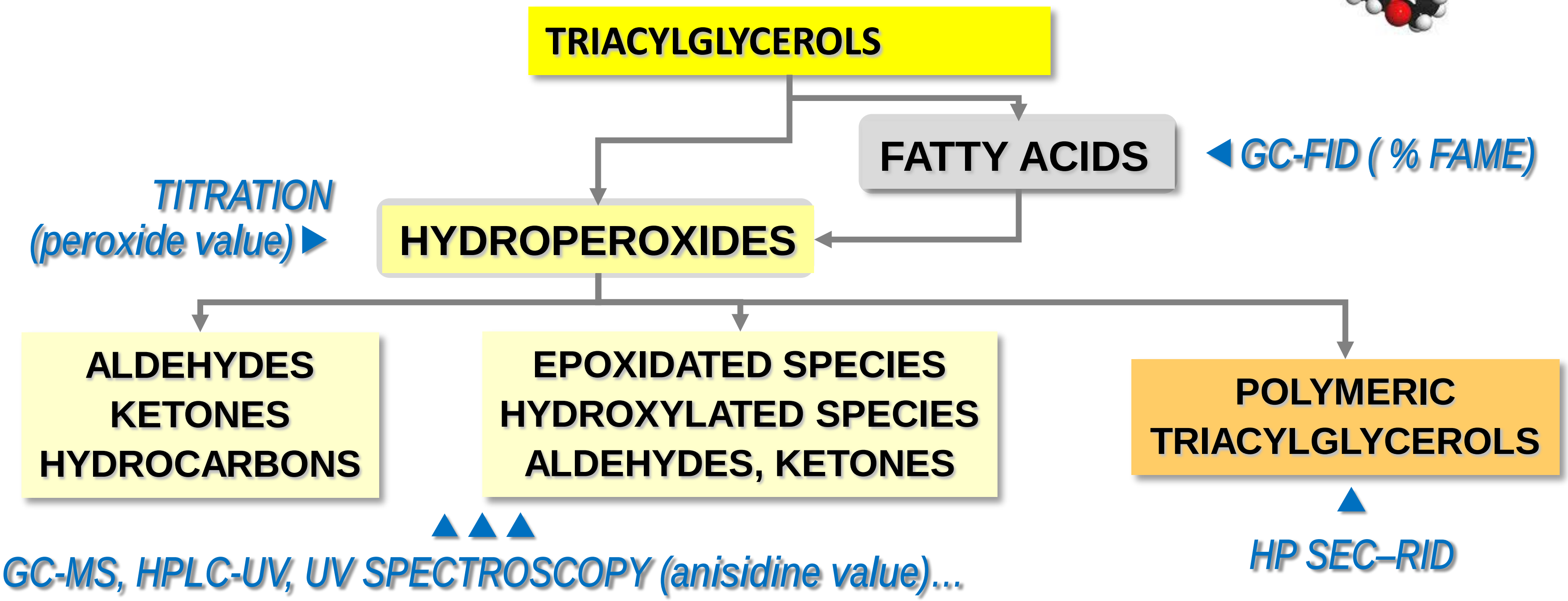
$$ROO^{\cdot} + ROO^{\cdot} \rightarrow ROOR + O_2$$

$$RO^{\cdot} + R^{\cdot} \rightarrow ROR$$

$$ROO^{\cdot} + R^{\cdot} \rightarrow ROOR$$

$$2RO^{\cdot} + 2ROO^{\cdot} \rightarrow 2ROOR + O_2$$

Conventional strategies in oils / fats quality assessment



High-throughput analytical methods for monitoring of oxidative degradation some innovattion needed….

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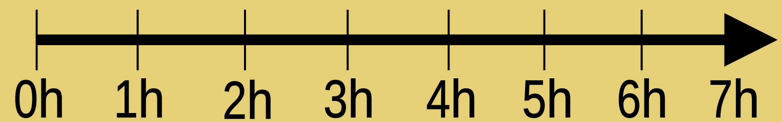
Samples

various fresh refined vegetable oils were sourced from retail market

- OLIVE OIL (PV 4.6)*
- RAPESEED OIL (PV 2.0)*
- SOYBEAN OIL (PV 2.2)*
- SUNFLOWER OIL (PV 2.3)*
- PALM OIL (PV 0.6)*

HEAT-TREATMENT

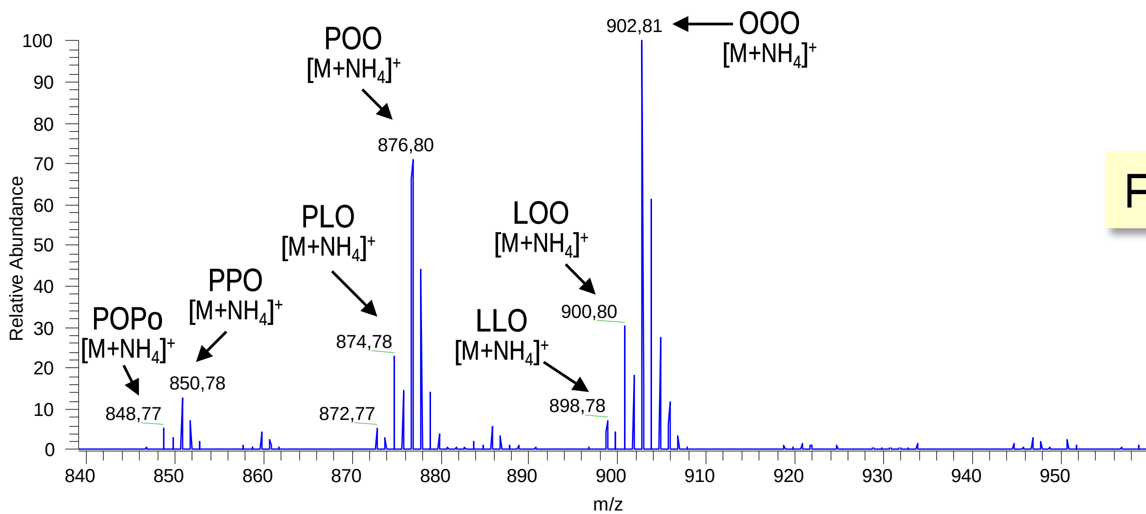
- Heating of 25g of oil in a beaker (*n*=3)
- Heating time 0 – 7 hours
- Controlled temperature 180 ± 2°C



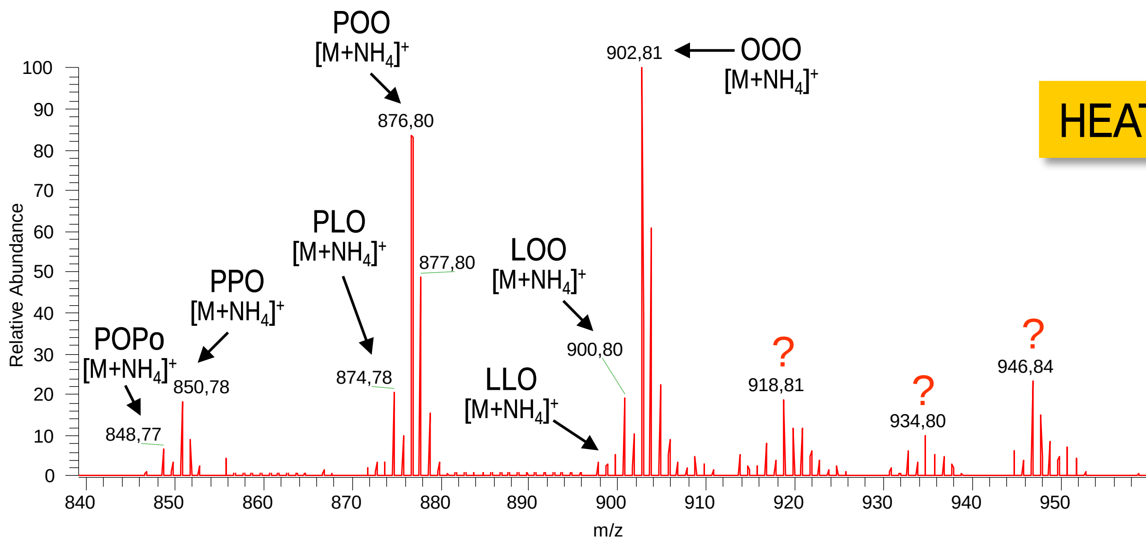
*PEROXIDE VALUE (mekv. of active oxygen per kg)

OXIDATION OF TRIACYLGLYCEROLS

DART–MS SPECTRA (POSITIVE ION MODE): DILUTED **OLIVE OIL**



FRESH OIL

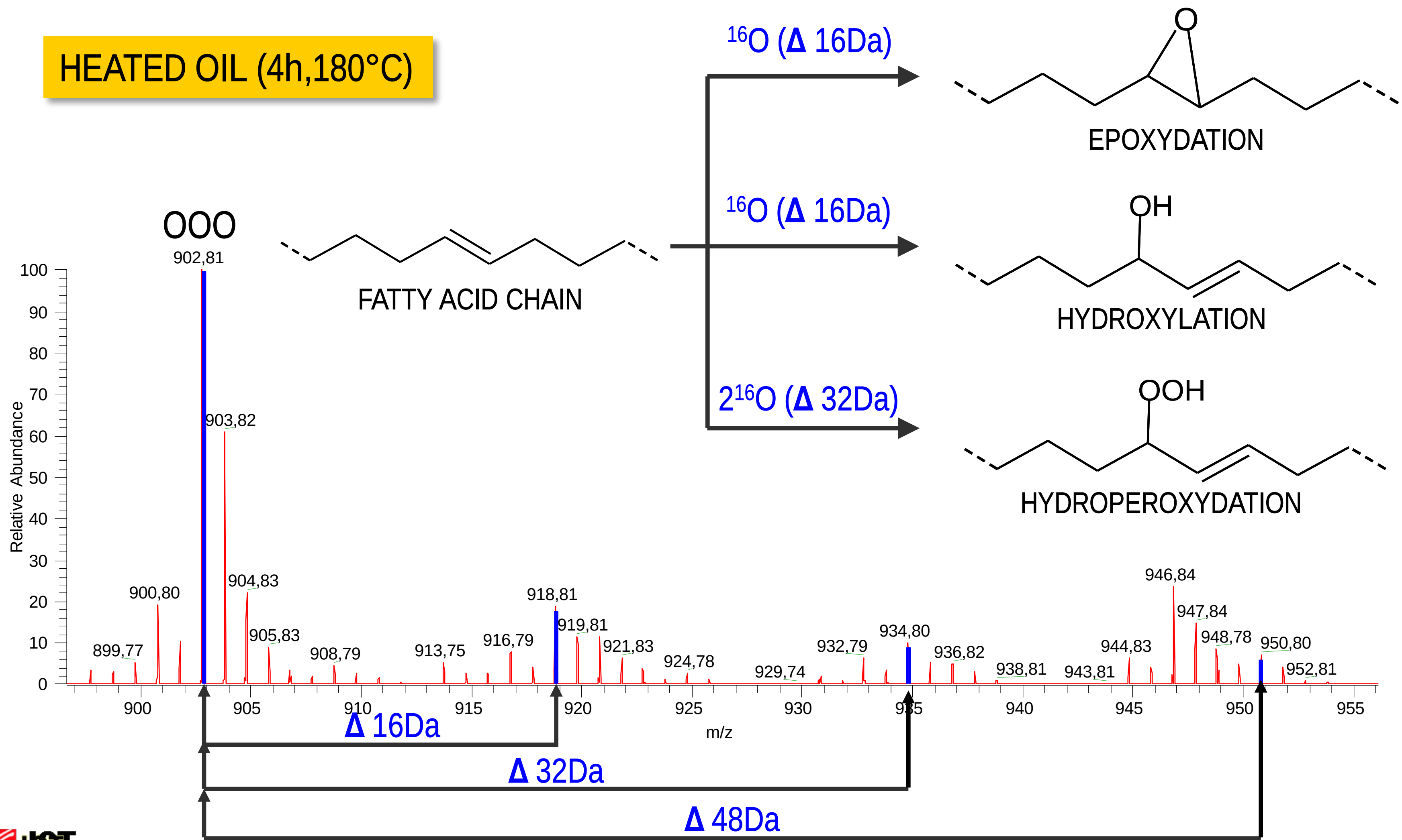


HEATED OIL (4h,180°C)

OXIDATION OF TRIACYLGLYCEROLS

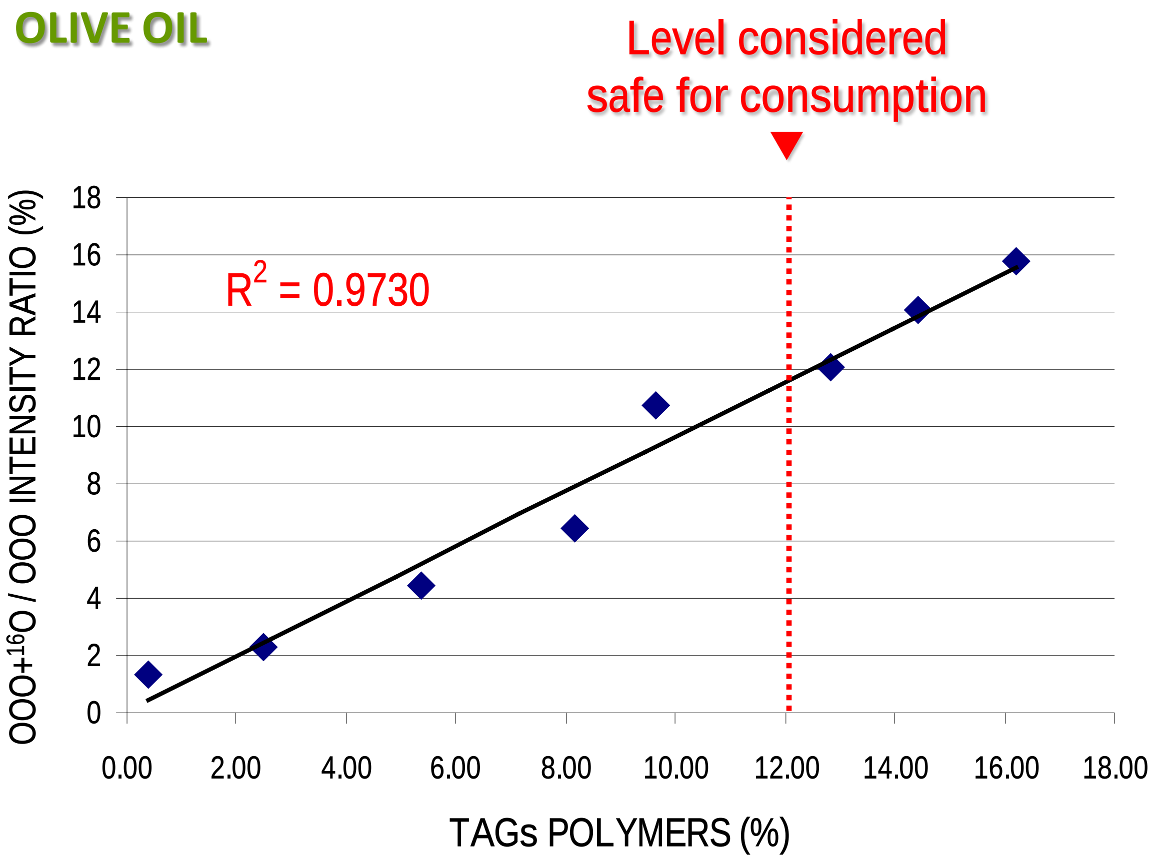
DART–MS SPECTRA (POSITIVE ION MODE): DILUTED **OLIVE OIL**

HEATED OIL (4h,180°C)



Oxidation of triacylglycerols

- CORRELATION WITH TAG POLYMERS CONTENT



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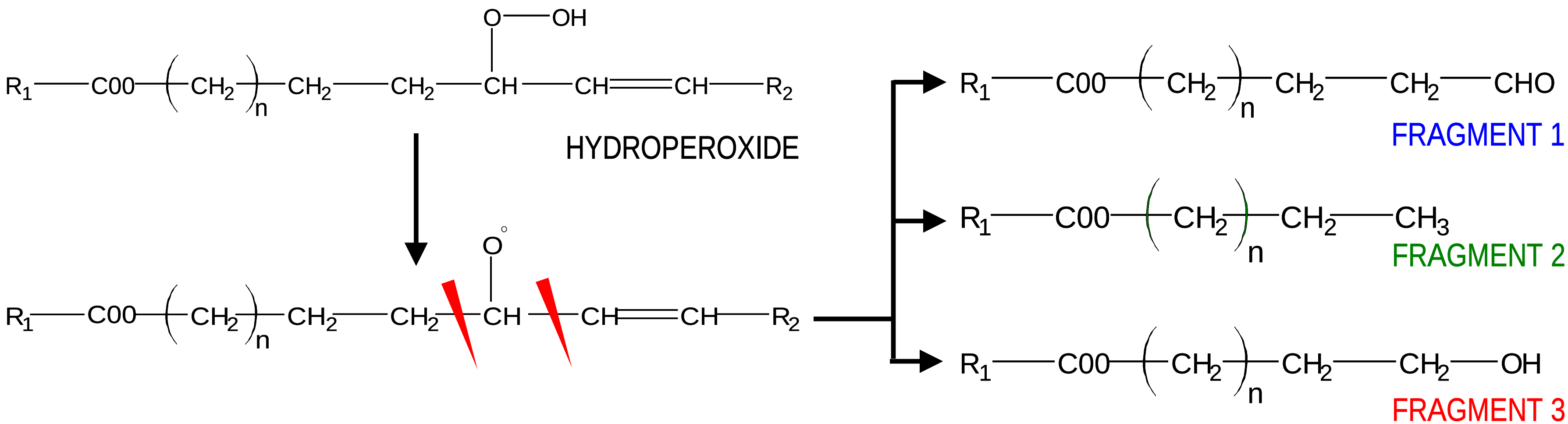
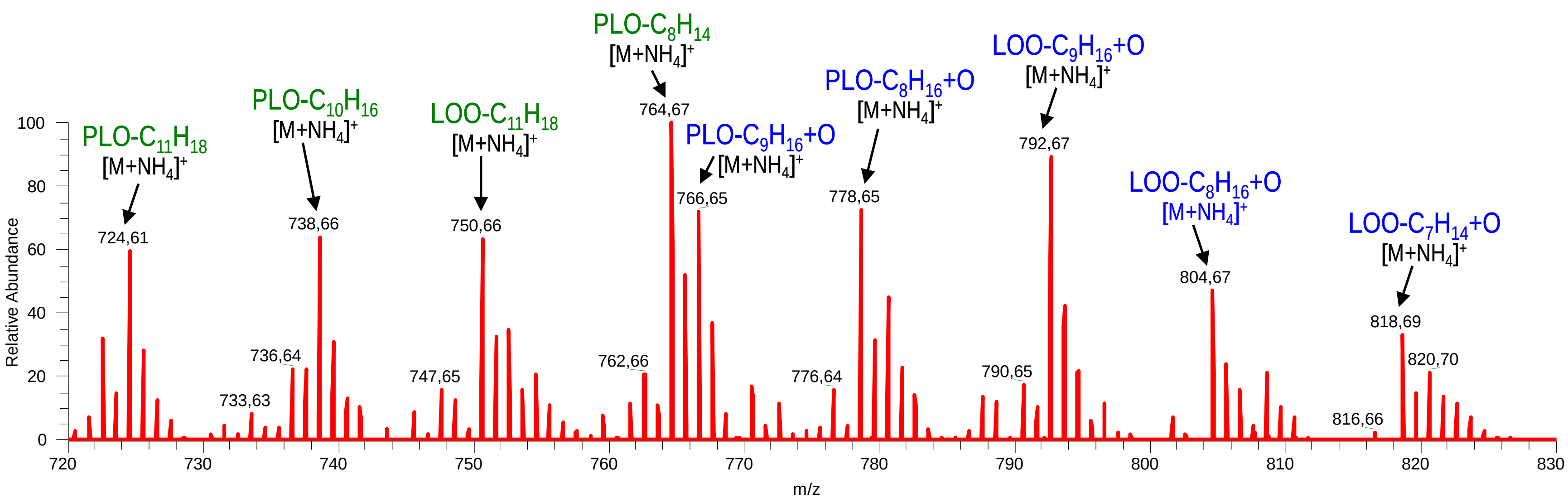
ß-SCISSION FRAGMENTS OF TAGs

DART–MS SPECTRA (POSITIVE ION MODE): DILUTED **OLIVE OIL**

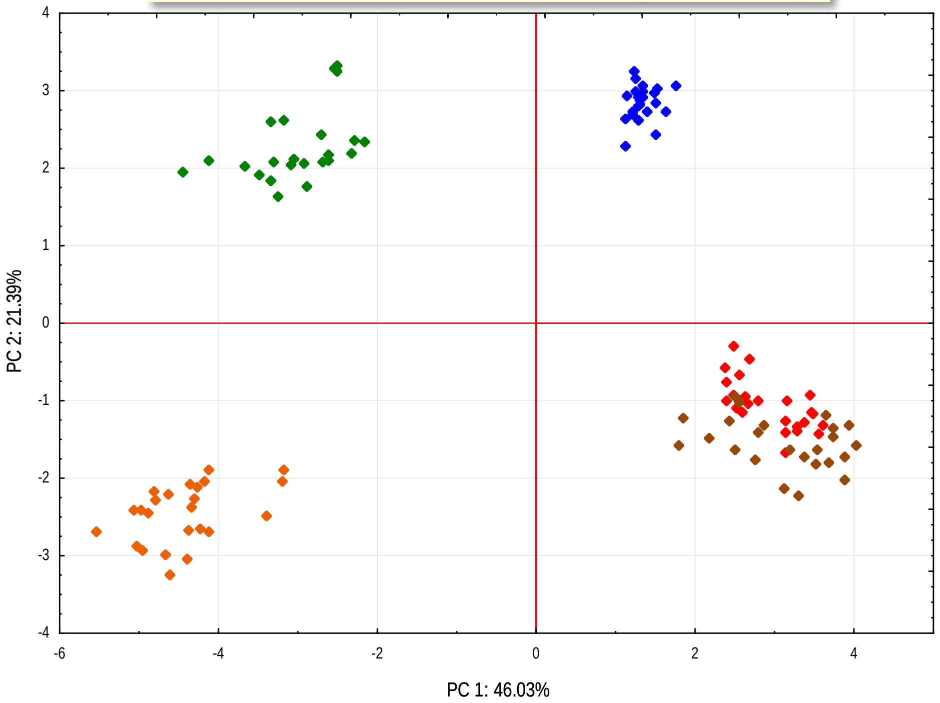




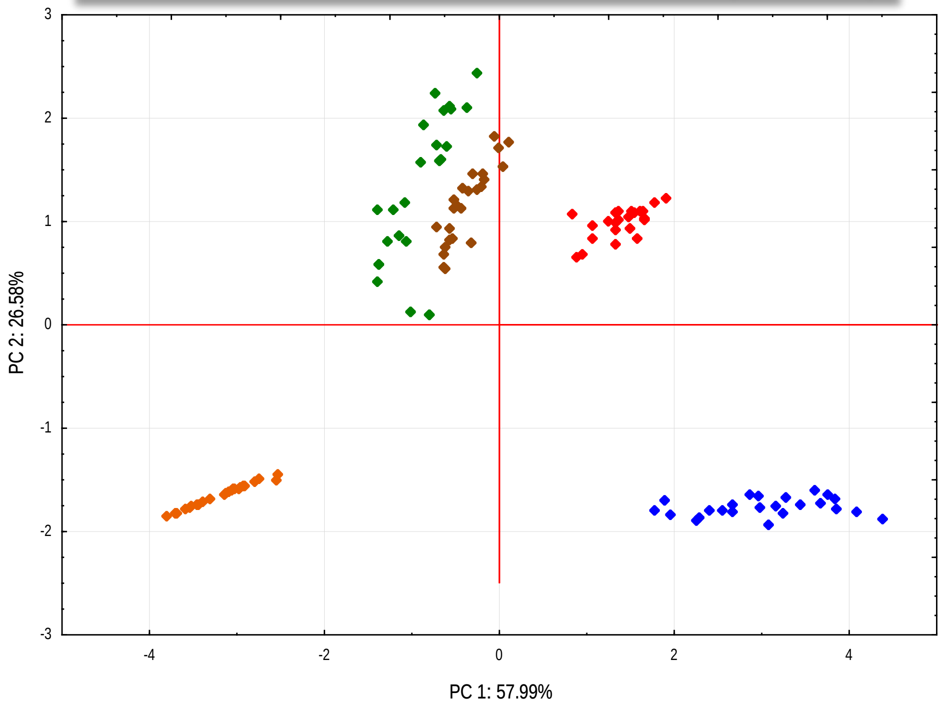
COMPREHENSIVE VIEW ON THE DATA

- PRINCIPAL COMPONENT ANALYSIS

TAGs & OXIDIZED TAGs (22 IONS)



STEROLS & OXIDIZED STEROLS (8 IONS)



- SOYBEAN OIL
- OLIVE OIL
- RAPESEED OIL
- SUNFLOWER OIL
- PALM OIL

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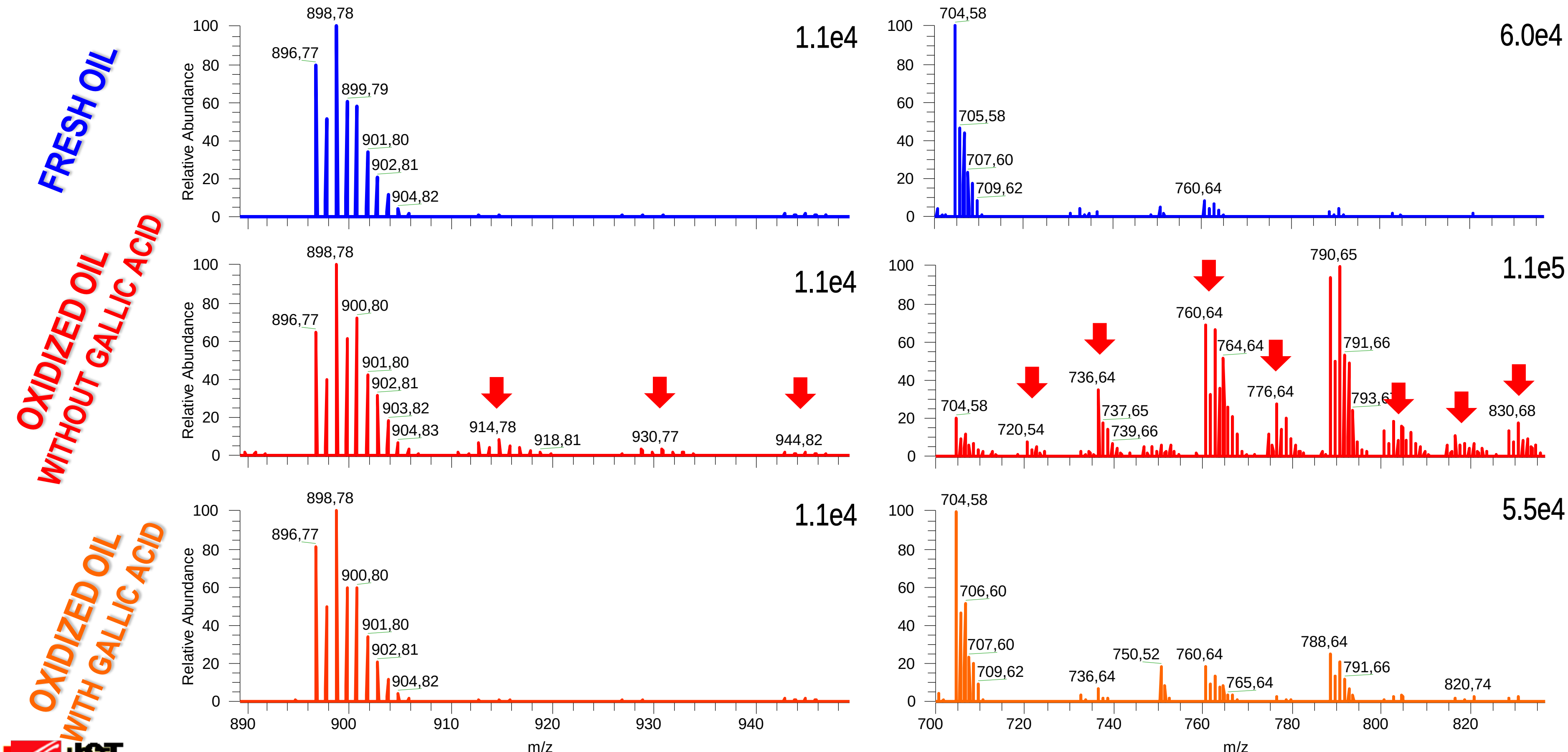
MONITORING OF ANTIOXIDANT ACTIVITY

- OXIDATION OF SUNFLOWER OIL (120°C, 2h) WITH AND WITHOUT ADDITION OF ANTIOXIDANT (GALLIC ACID, 100ppm)…

DART–MS SPECTRA (POSITIVE ION MODE)

NATIVE / OXIDATED TRIACYLGLYCEROLS

ß-SCISSION FRAGMENTS

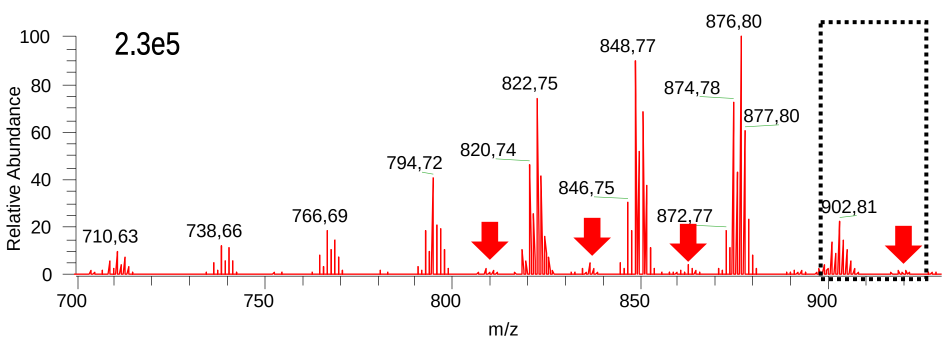


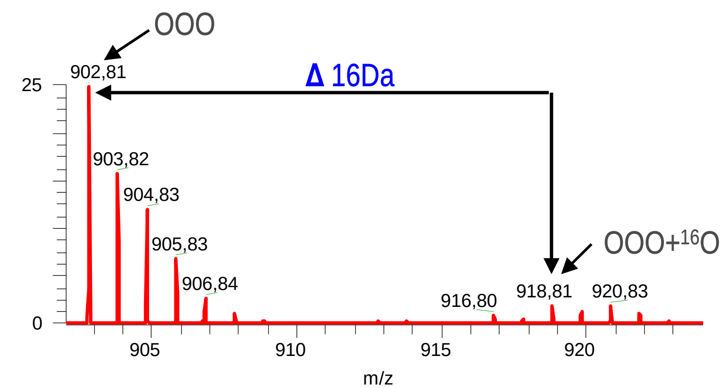
OXIDIZED TAGs IN HUMAN ADIPOSE TISSUE

- HUMAN ADIPOSE TISSUE SOURCED FROM LIPOSUCTION SURGERY (3 SAMPLES COLLECTED FROM 3 DIFFERENT PATIENTS)

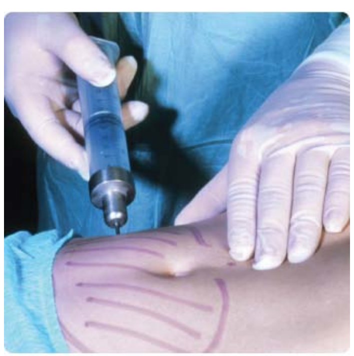
DART–MS SPECTRA (POSITIVE ION MODE)

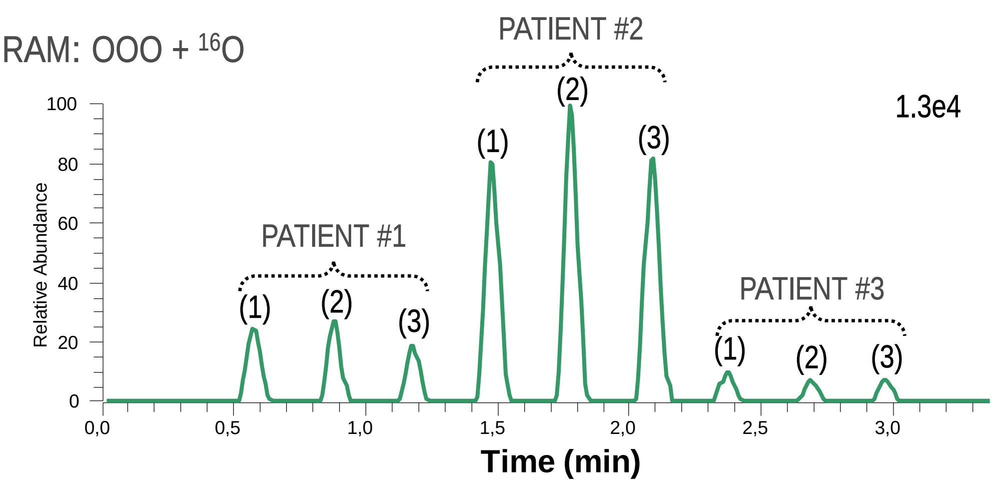
NATIVE / OXIDATED TRIACYLGLYCEROLS





EXTRACTED ION CHRONOGRAM: OOO + ^{16}O





CONCLUSIONS: FUTURE CHALLENGES

- **Databases *vs*. standardization**
- **Diversity/variation of metabolomic data**
- **More efficient ways of identifying markers**
- **More efficient ways of coupling:**

  Instruments → Data processing → Chemometric tools

- **New instrumentation platforms**

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- Veterinary drugs

- Authenticity, Fraud
- Agriforensics
- Food-Omics
- Novel foods & supplements
- Organic crops & foodstuffs
- QA/QC and chemometrics

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Prof. Franz Ulberth	JRC, Institute for Reference Materials and Measurements, Geel, B
Dr. Frans Verstraete	European Commission, DG Health and Consumers (DG SANCO), B
Dr. Zhihua Ye	Chinese Academy of Agricultural Sciences, Beijing, CN

KEYNOTE SPEAKERS INTRODUCING SYMPOSIUM TOPICS:

- Elke ANKLAM (European Commission, DG Joint Research Centre Director, Institute for Reference Materials and Measurements, Geel, B)

  NEW RESEARCH CHALLENGES IN FOOD QUALITY AND SAFETY CONTROL

- Paul BRERETON (The Food and Environment Research Agency, York, UK)

  ANALYTICAL CHALLENGES AND OPPORTUNITIES FOR ASSURING INTEGRITY OF THE FOOD SUPPLY

- Hermann BROLL (Federal Institute for Risk Assessment, Berlin, D)

  RECENT TRENDS IN APPLICATION OF MOLECULAR BIOLOGY BASED METHODS IN FOOD ANALYSIS

- Alejandro CIFUENTES (National Research Council of Spain, Institute of Food Science Research, Laboratory of Foodomics, Madrid, ES)

  FOOD-OMICS: PRESENT AND FUTURE CHALLENGES IN FOOD ANALYSIS

- Christopher ELLIOTT (Queen's University Belfast, Belfast, UK)

  BIOASSAYS FOR BIOMARKERS: TOOLS FOR DETERMINING HUMAN EXPOSURE TO FOOD TOXINS

- Jacob DE JONG (RIKILT Wageningen UR, Wageningen, NL)

  LESSONS LEARNT FROM THE CONffIDENCE PROJECT: CONTAMINANTS IN FOOD AND FEED – INEXPENSIVE DETECTION FOR CONTROL OF EXPOSURE

- Rudolf KRSKA (University of Natural Resources and Life Sciences, Vienna, IFA-Tulln, A)

  MYCOTOXINS AND THEIR METABOLITES: FROM TARGETED TO UNTARGETED ANALYSIS

- Erich LEITNER (Graz University of Technology, Graz, A)

  STRATEGIES FOR FLAVOUR AND OFF-FLAVOUR DETECTION

- Ryszard LOBINSKI (CNRS-University of Pau, Pau, FR)

  METALS AND METALLOIDS IN FOOD: SPECIATION AND –OMICS

RAFA 2013 keynote speakers, cont.

- Bert POPPING (Eurofins Scientific Group, Hamburg, D)

  THE EVOLUTION OF ALLERGEN METHODS: WILL TOMORROWS METHOD JUST BE BETTER OR ALSO FASTER?
- Peter SCHIEBERLE (Food Chemistry, Technical University of Munich, Garching, D)

  THE CHEMISTRY OF FOOD FLAVOURS: SIMPLY PLEASURE OR BEYOND?
- Yang SHUMING (Institute of Quality Standards & Testing Technology for Agro-Products, Chinese Academy of Agricultural Sciences, Beijing, CN)

  Title to be confirmed
- Michele SUMAN (Barilla Food Research Labs, Parma, I)

  SUMMARY & DISCUSSION PLATFORM: INDUSTRY PERSPECTIVES
- Bert VAN BAVEL (Örebro University, Örebro, S)

  THE IMPORTANCE OF QA/QC AND THE LATEST DEVELOPMENTS IN POP ANALYSIS REGULATED BY THE STOCKHOLM CONVENTION
- Frans VERSTRAETE (European Commission, DG Health and Consumers (DG SANCO), B)

  RECENT AND FUTURE DEVELOPMENTS IN THE REGULATION OF FOOD CONTAMINANTS
- Stefan WEIGEL (RIKILT Wageningen UR, Wageningen, NL)

  ANALYSIS OF NANOPARTICLES IN FOOD: FROM CHALLENGE TO ROUTINE?

Acknowledgement:

all the nice colleagues from Sicily

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