Tracing the origin of Lambrusco sparkling wines profiling the “aroma” by GC-QTOF

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Overview

Introduction to Food Profiling

Authenticity

Food Degradation

Process Development

Introduction to Food Profiling

Standard Food Testing Applications with Mass Spec:

- Contaminant Identification and Quantitation
  - Pesticides
  - Mycotoxins
  - Marine Toxins
  - Veterinary Drugs
  - Adulterants (Melamine)
  - And others

… but what are we missing?

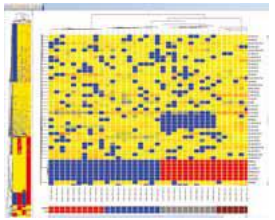
Introduction to Food Profiling

The questions contaminant testing won't answer:

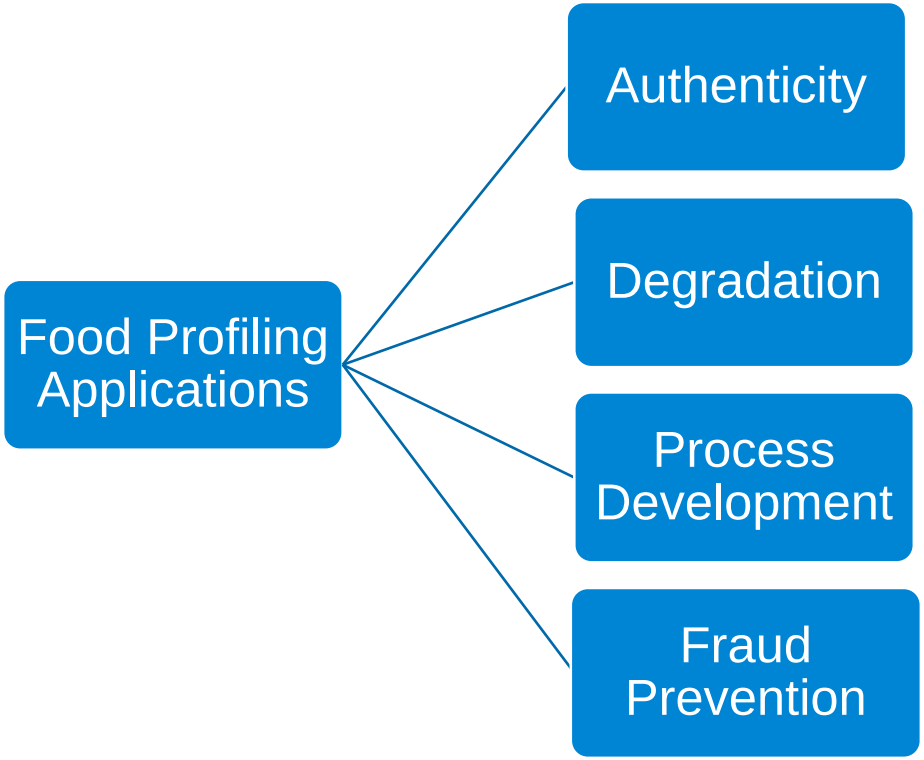
- Is this wine cabernet or pinot noir?
- Is this wine label true?
- Is this wine a real DOC wine?
- How did changing my growing or fermentation process affect my food product?

To answer these questions, we need
Food Profiling

Agilent Food Profiling Solution

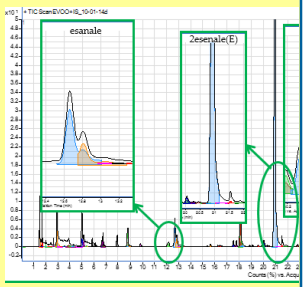
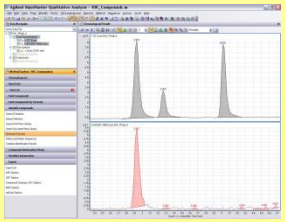


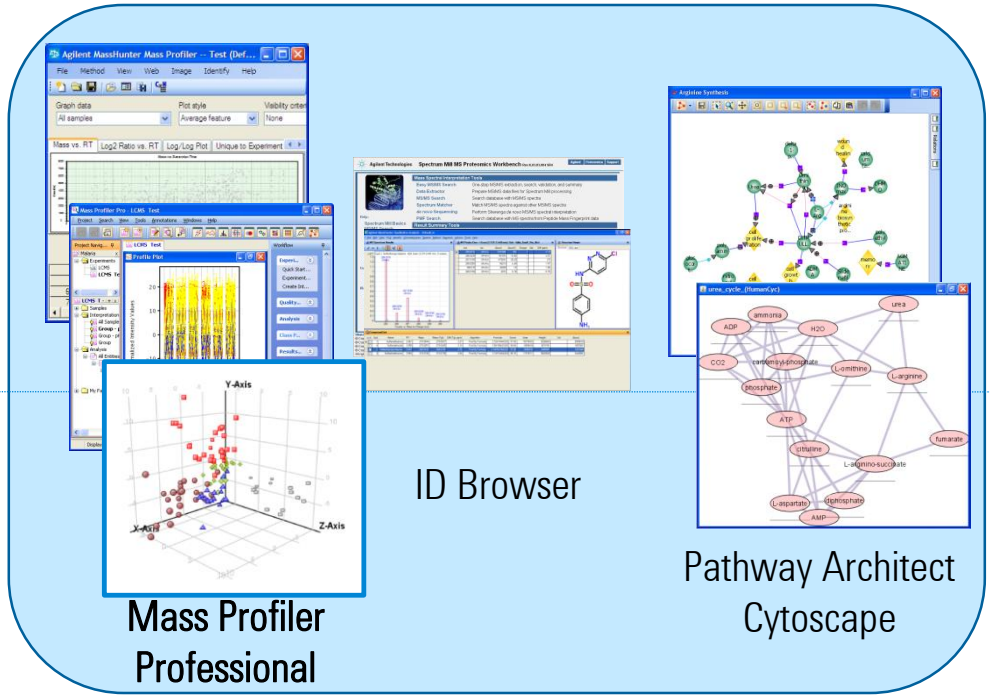
Q-TOFs and Mass Profiler Professional (MPP)



Food Profiling (def.) - *The processing of mass spectral data from two or more food samples in order to highlight the compounds which have a statistically different concentration.*

The Agilent Profiling: Chemometric Workflow

	Separate & Detect	Feature finding quantitate	Alignment & statistics	Identify	Pathways
GCMS	 GC-QTOF	 Deconvolution			
LCMS	 LC-QTOF	 Molecular Feature Extraction			



Agilent 7200 GC-QTOF

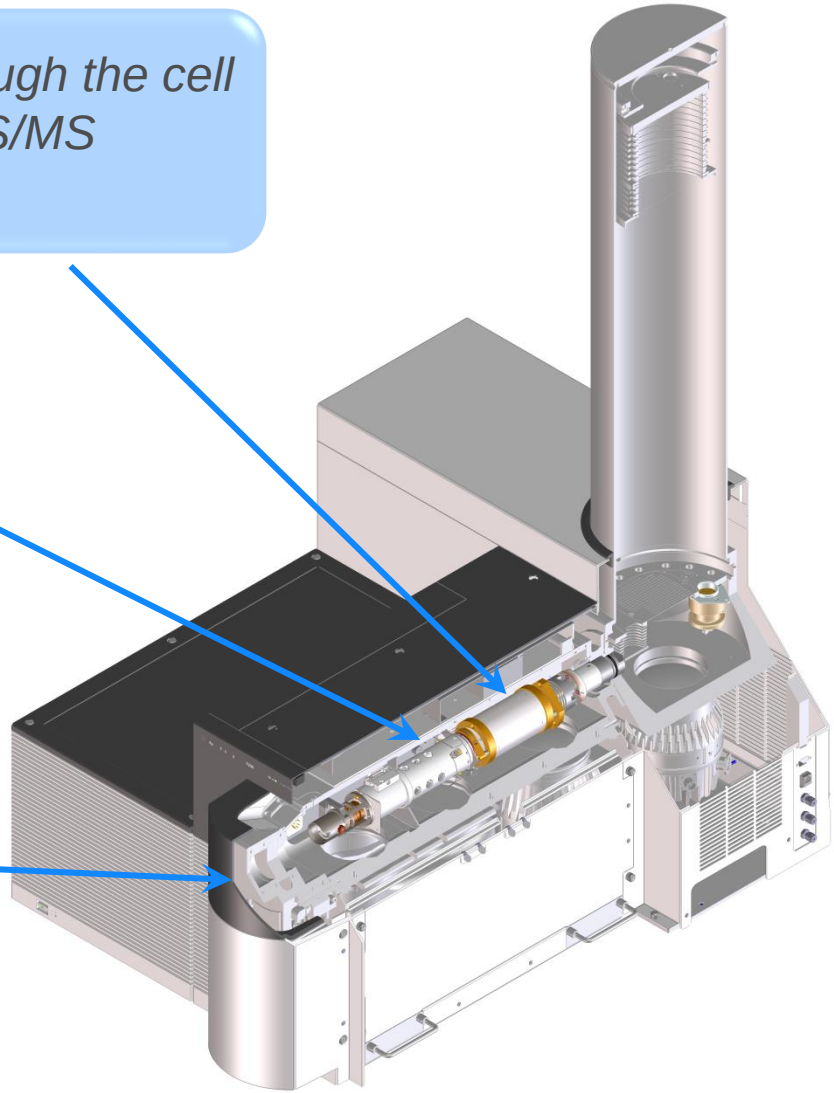
What is the GC-QTOF

7200 GC/Q-TOF

Hexapole collision cell *accelerates ion through the cell to enable faster generation of high-quality MS/MS spectra without cross-talk*

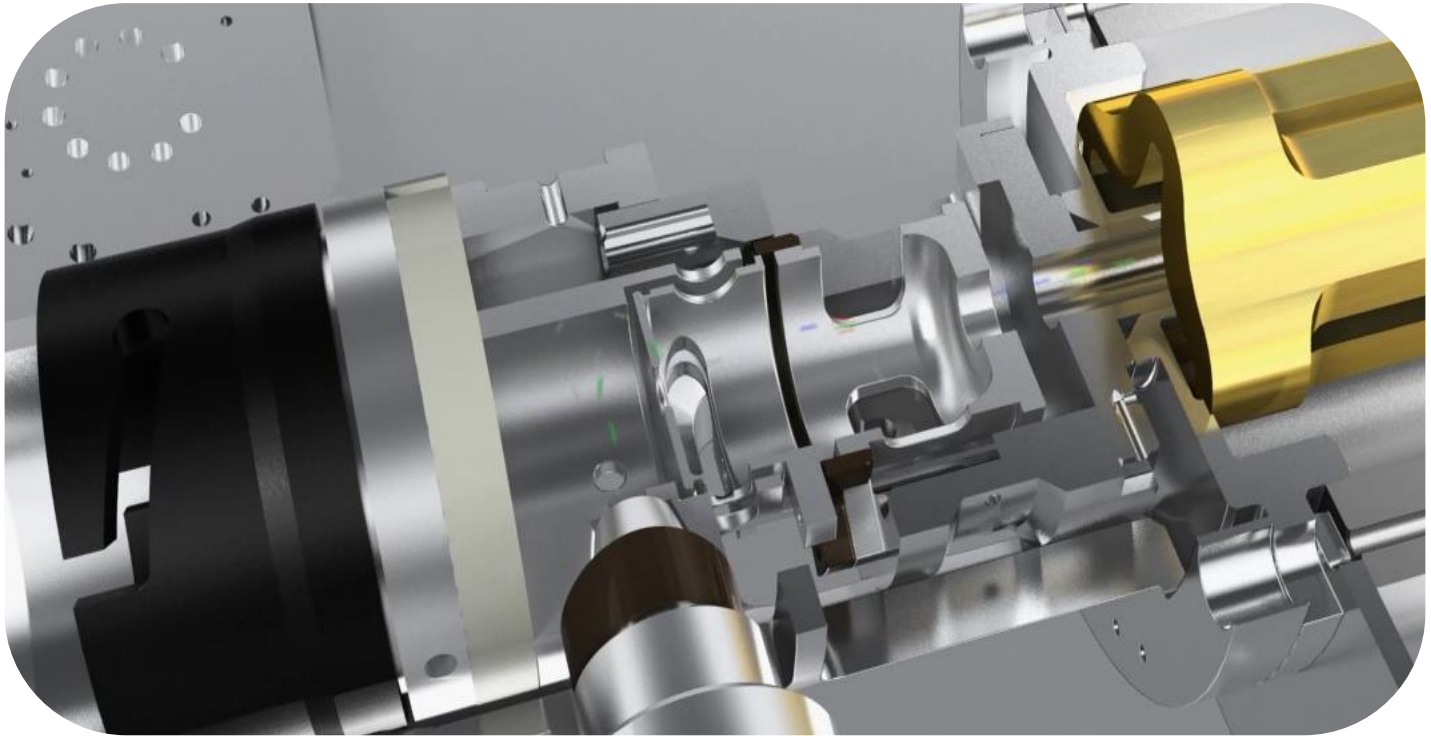
Hot, quartz monolithic quadrupole analyzer *identical to the 7000 Quadrupole MS/MS*

Removable Ion Source *includes repeller, ion volume, extraction lens and dual filaments*



New Removable Ion Source

includes repeller, ion volume, extraction lens and dual filaments



The Project “Lambrusco”

“New Analytical Methodologies for Geographical and Varietal Traceability of Oenological Products”

Red sparkling wine

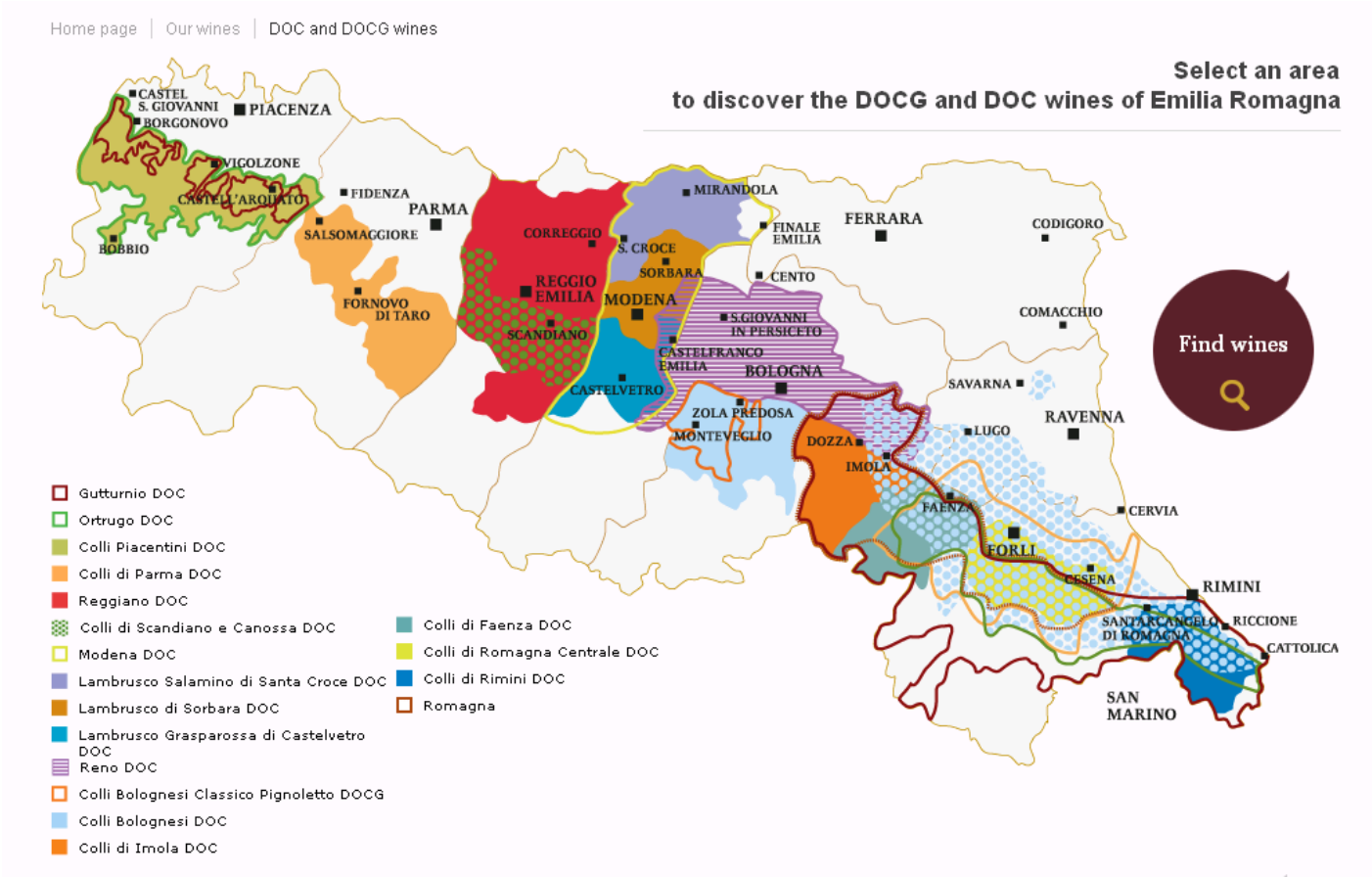
In Modena 4 DOC areas:

- Lambrusco Salamino di Santa Croce
- Lambrusco di Sorbara
- Lambrusco di Modena
- Lambrusco Grasparossa di Castelvetro

Lambrusco Project

New Analytical Methodologies for Geographical and Varietal Traceability of Oenological Products

Emilia-Romagna



GC-QTOF Analysis

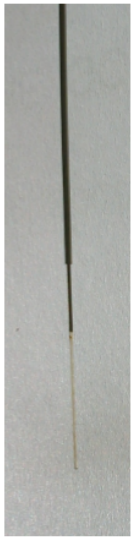
Solid Phase Microextraction SPME

Sample preparation

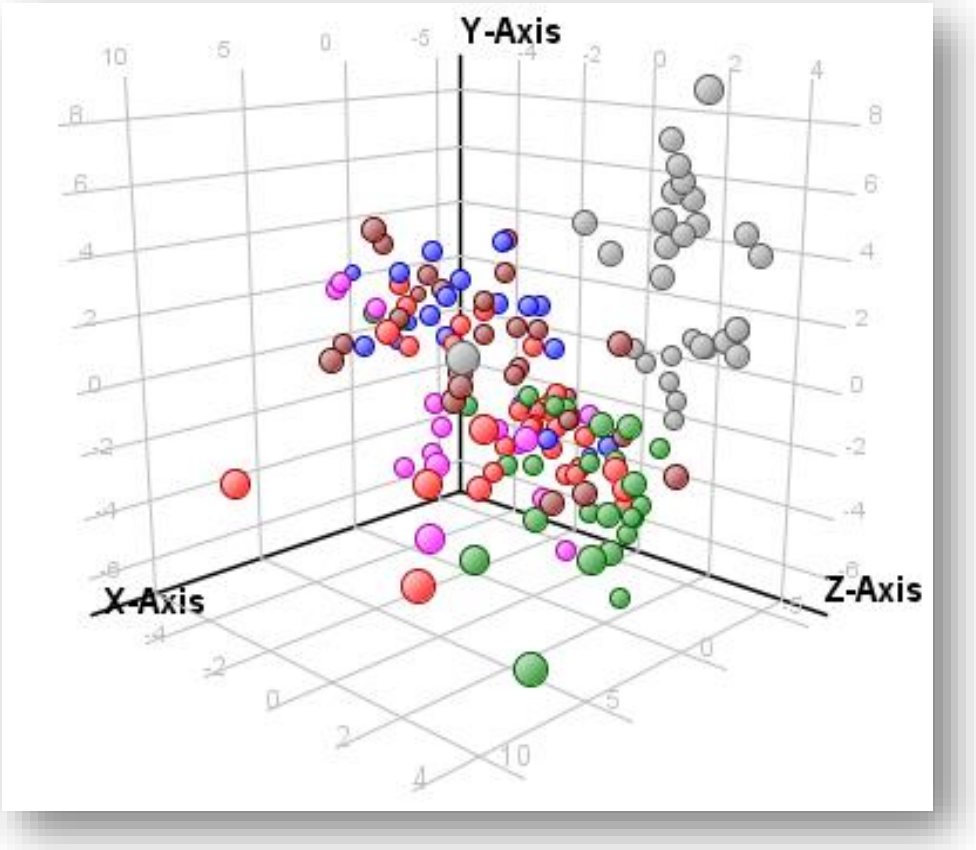
- ultrasonic bath to remove CO_2
- Internal standard: ethyl hexanoate d11
- Quality control QC: mix of all samples

Solid phase microextraction SPME

- 10 mL wine + 3 g NaCl in 20mL vial,
- incubation 10 min @ 35°C
- extraction 40 min @ 35°C
- DVB/CAR/PDMS 2cm fiber
- Agilent autosampler (like CTC combiPAL)
- Desorption 260°C in GC inlet 2 min splitless, narrow liner 0.75mm id
- Fibre cleaning 10min @ 265°C needle heater



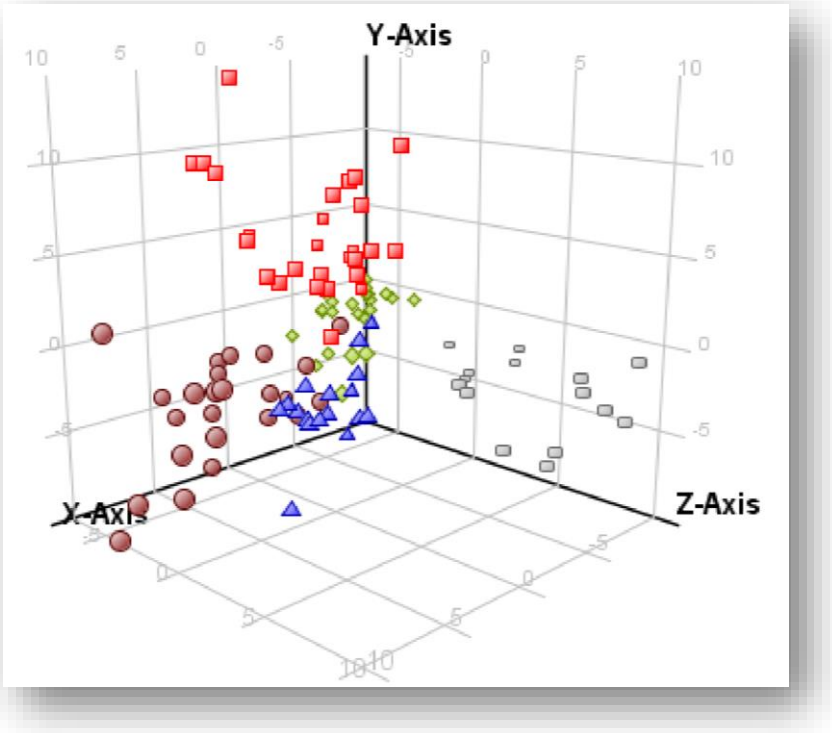
Mass Profiler Professional MPP



PCA: initial evaluation

Mass Profiler Professional MPP

Classification – PLS model

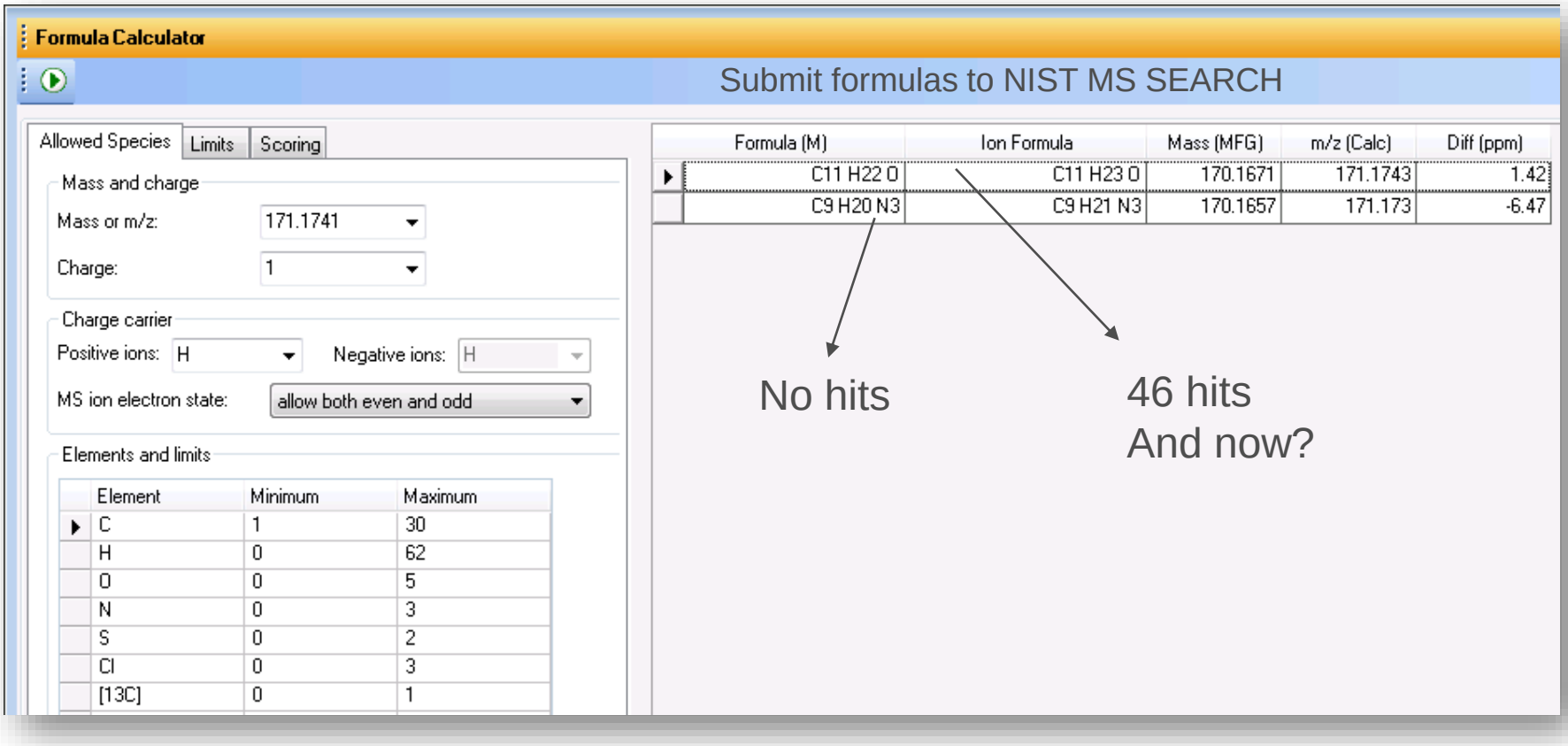


- Grasparossa
- Sorbara
- Salamino
- Modena
- outside

Overall model accuracy:97%

Identification of Compounds

Use accurate mass → formula calculator



Identification of Compounds

Chemical Ionization (CI):

- to define the Molecular Ion;

Electron Impact spectra (EI):

- Identification of fragments formulas, library search;

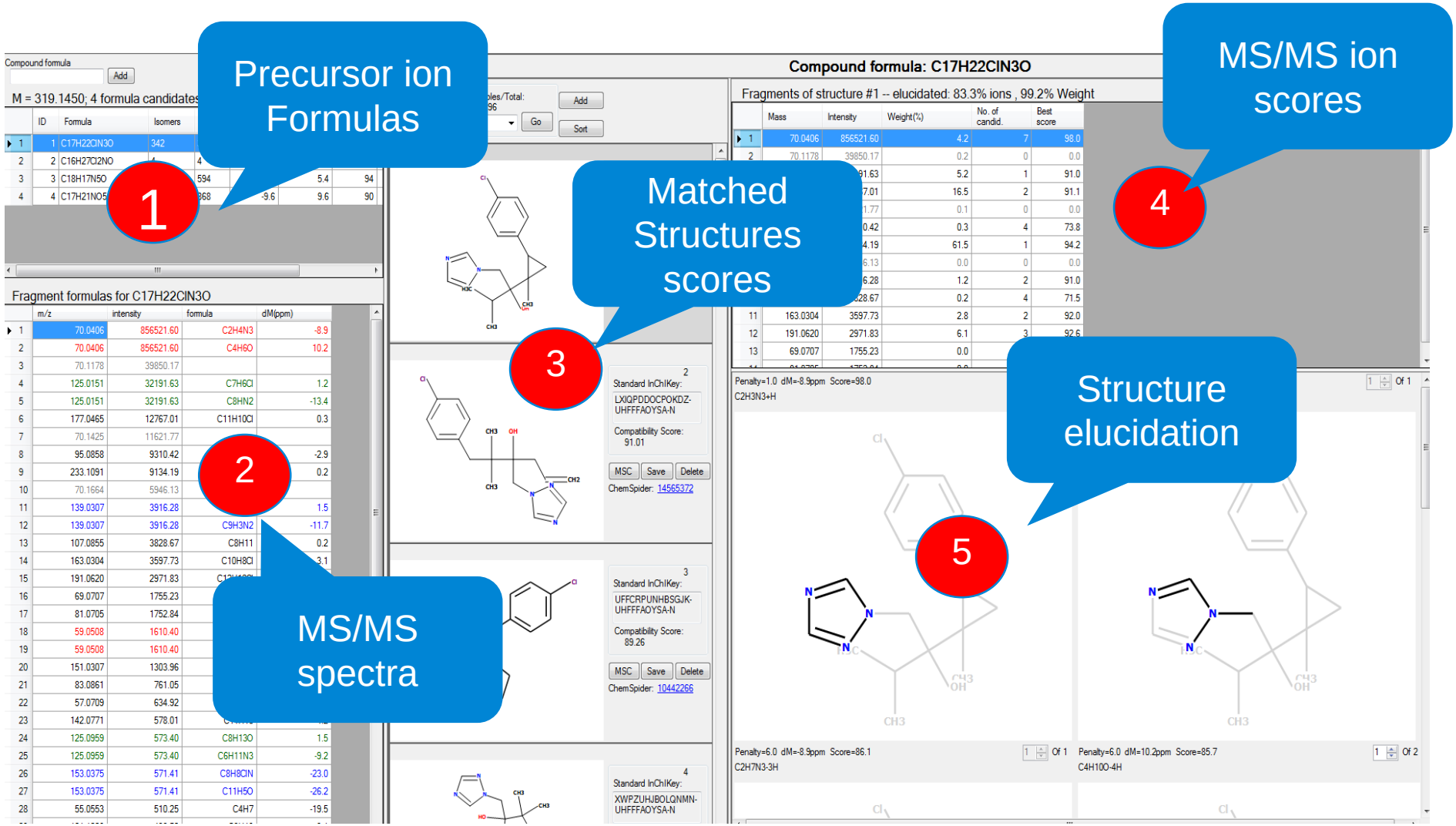
Using Accurate Ms/Ms data, 200 compounds are ready for identification.

Actual Results: 31 compounds identified (mf>60%) matching expected RT and RI

limonene
methylglyoxal
nonanal
p-cymene
undecanal
α-methylbutyric acid
1-hexanol
1-nonanol
E,E-2,4-hexadienal
2-nonanone
2-pentanone
3-ethoxy-1-propanol
4-ethylphenol
benzyl alcohol
decanal
epiglobulol
γ-octalactone
ethyl 2-methyl butyrate
2-methylbutyl acetate
ethyl hexanoate
ethyl heptanoate
ethyl isobutyrate
ethyl myristate
ethyl nonanoate
ethyl octanoate
ethyl phenylacetate
ethyl trans-4-decenoate
butyl benzoate
isobornyl acetate
ethyl heptanoate
ethyl sorbate*

* Degradation product from sorbic acid, wine additive, found in only 1 sample

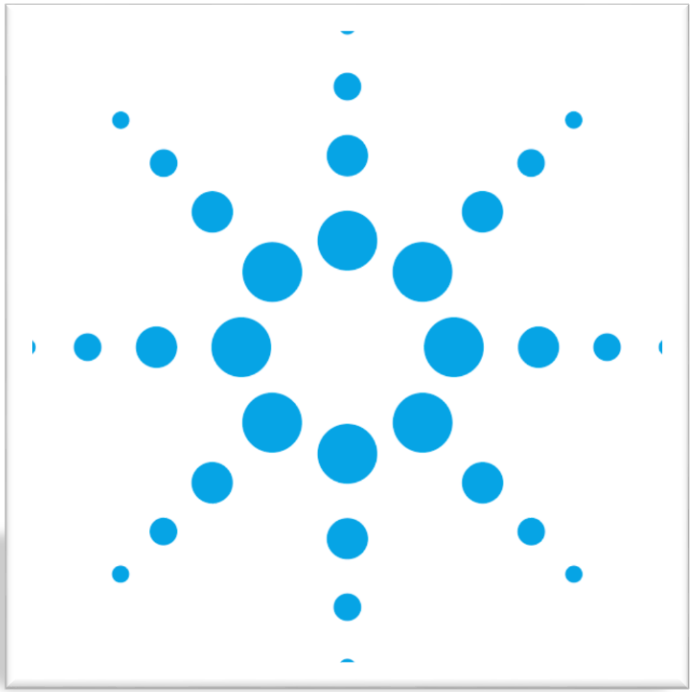
Molecular Structure Correlator Software (MSC)



Conclusions

- GCQTOF suitable for food profiling applications
- Volatile Wine fraction as biomarker
- Smooth statistical data processing needed
- Identification by Ci+EI data, Libraries and MSC

Thank you for attention



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