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Comprehensive analysis of wine —from test to taste

Live Webcast: Thursday, October 18, 2012, 9:00 am EDT; 15:00 CEST

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Comprehensive analysis of Wine – from test to taste

By Roberta Danzi

*Technical Manager
and Responsible for
Quality Management of Unione
Italiana Vini Labs*



Unione Italiana Vini
since 1895

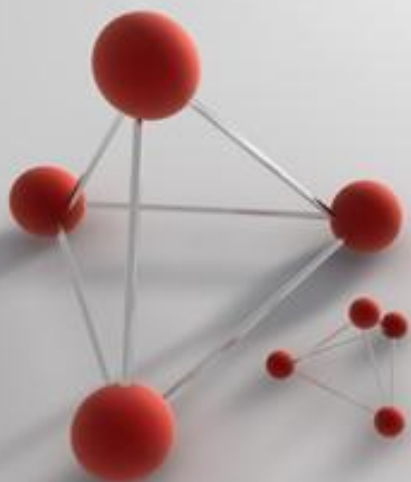


Italy, along with France, is the world's largest producer of wine. Wine has always been one of the traditional Italian products, and even though domestic consumption has been reduced to a great extent over the past 50 years, wine remains one of the most important products of the Italian agri-food sector. With regard to the export it is one of the leading products made in Italy in the world.

Some numbers

- Vineyard surface 664,000 ha
- Italy turnover 10.7 billions €
- Share of food (Italy) 9%
- Export turnover 4.4 billions €
- Share of food (export) 13%
- Induced value 2 billions €
- Supply chain employee 1.2 millions

Source: Istat, Ismea, Ministero del Lavoro, data 2010/11



Agenda

Unione Italiana Vini

Product Control

Law Compliance

Hygiene and Health

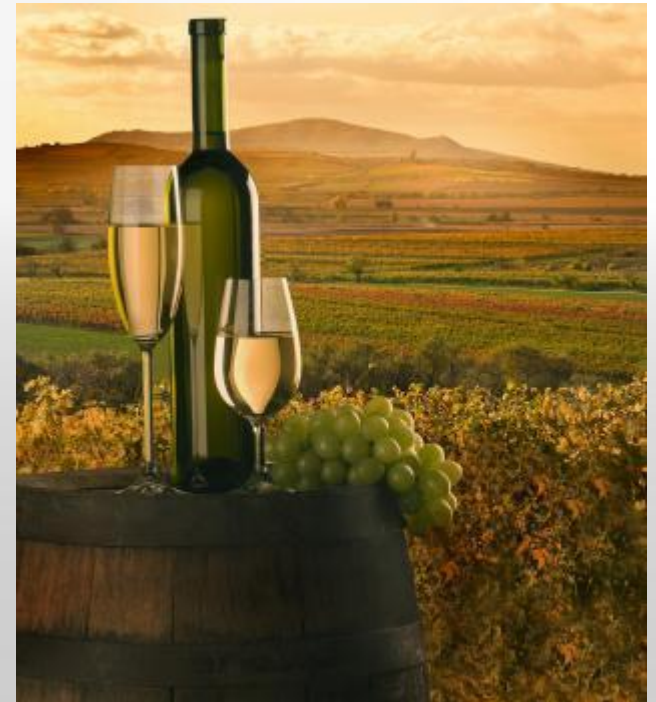
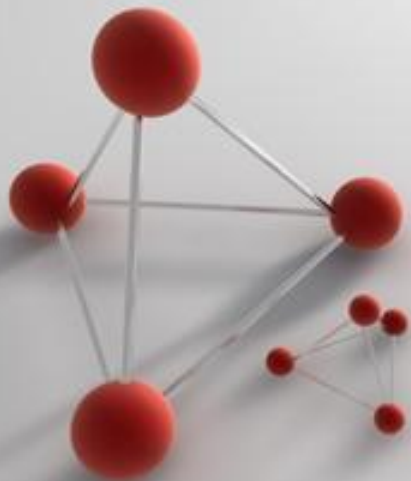
Authenticity and Origin

Technological Quality

Analytical Techniques

Applications

Needs of a Lab



Unione Italiana Vini is...

Association Founded in Milan 1895

Members: about 500, with a turnover of 5 billions euros (equal to 50% of Italian domestic turnover and 60% of exports)

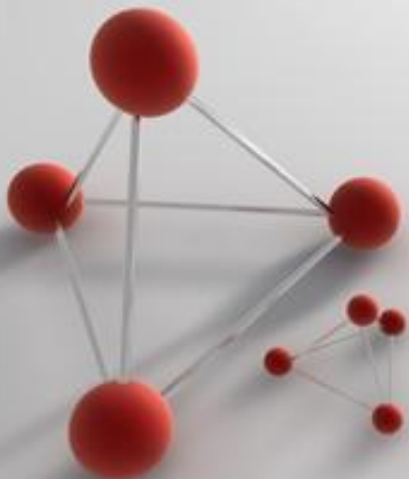


Offices:

- Verona
- Milan
- Rome

Labs:

- Verona
- Siena
- Asti
- Reggio Emilia
- Lecce



The role of Unione Italiana Vini

In the last years, the Food and Beverage industry underwent very important changes affecting the whole production chain.

The market and the mass-media are pressing stakeholders to invest in and improve:

- Quality of products;
- Guarantee of healthiness;
- Typical features;

Legislators translate those expectations, with the objective to protect health and the interest of citizens, with:

- Compulsory rules;
- Voluntary rules;
- International Standards;

The need to standardize processes, including distribution, induces the producer to review its corporate structure, to improve communication with the Market.

Market means all the stakeholders:

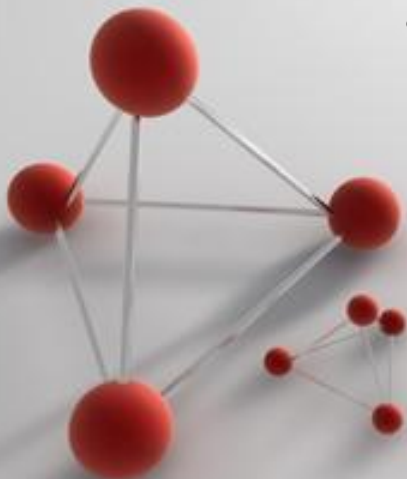
- Control bodies
- Institutions
- Large-scale retail traders
- Consumers



Unione Italiana Vini developed its own resources, laboratories and organizational advising, in order to supply to the enterprises some services and to reach some target, working in a self-control logic in synergy with the control agencies (ICQRF -Central Inspectorate for the Protection of the Quality and Fraud Repression of Agri-Food Products, NAS -Anti-Adulteration Group, etc).

The core business of our laboratories

- Analysis for **economic transactions**. We aim to be a third part that guarantees and favors the exchange trades;
(~17.000 samples/year for national trade, ~ 12.000 samples/year for export trade)
- Monitoring of the **compliance to PDO (Protected Designation of Origin) wines**;
(~10.000 samples/year)
- Great effort is spent in terms of supporting producers as a guarantee of the **self-control on bulk wine** (HACCP and others). Controls regarding healthiness of wine (hygiene), quality and genuinity;
(~26.000 samples/year)
- **Monitoring of wine on shelf** for large-scale trade distributors
(~2.000 samples/year)

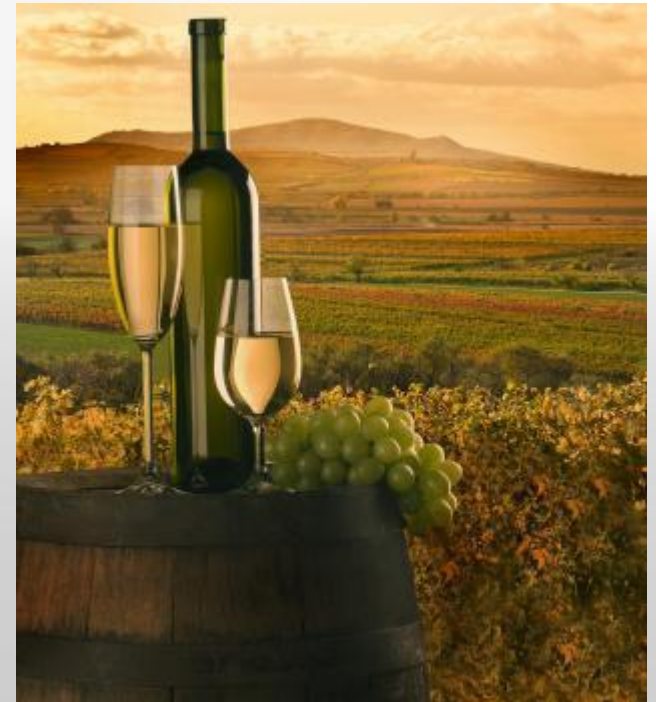
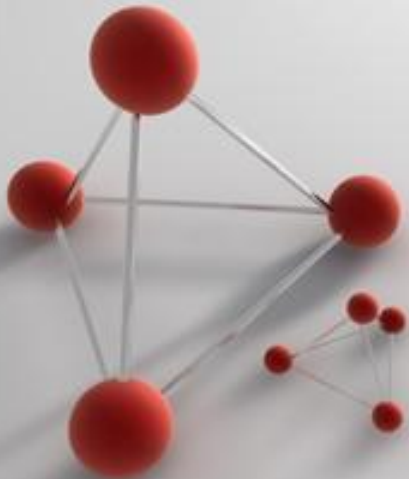


Agenda

Unione Italiana Vini **Product Control**

Law Compliance
Hygiene and Health
Authenticity and Origin
Technological Quality

Analytical Techniques
Applications
Needs of a Lab



The analytical protocols are designed to satisfy different requirements

■ Compliance to Laws

The control aims to check if the product complies to the National Laws and those of the European Community (characteristics needed to identify the product group).

(OCM regulation: REG CE 491/2009, 606/2009, 607/2009)

■ Hygiene and Health

Analysis of organic and inorganic pollutants (exogen or endogen origin).

■ Authenticity and origin

includes chemical and isotopic analysis to check for presence of forbidden ingredients."

■ Technological quality

includes analysis made to check the quality characteristics of the product and of the production process.



Analysis

- Alcoholic strength
- Total Acidity
- Sugars
- Dry extract deduced sugars
- Volatile acidity
- Ashes

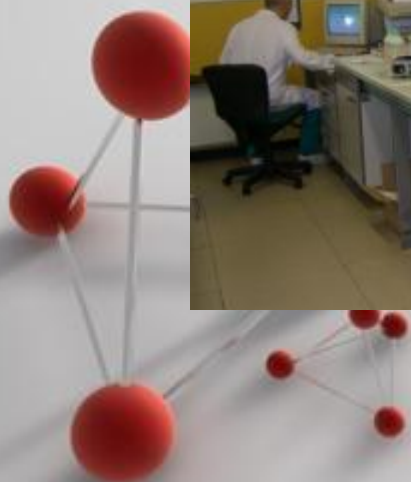


"Classic" Analytical Techniques

- Densitometry*
- Volumetric analysis (Titration)*
- Gravimetric analysis*

- FTIR

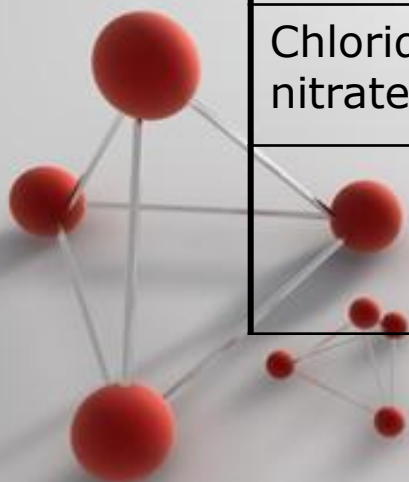
*official methods



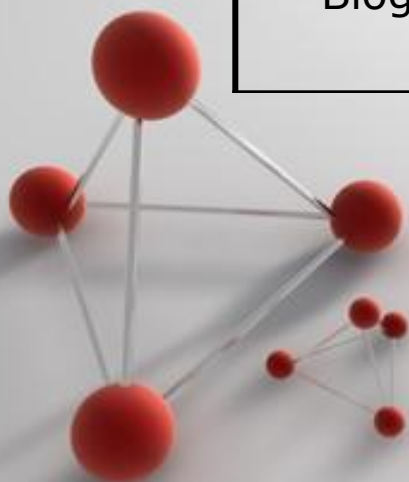
Exogenous pollutants

Preservatives and additives (sorbic acid, ascorbic acid)	They can be used and there is a law limit (200 mg/l sorbic, 250 mg/ ascorbic) *
Cyanide Derivatives	Use of potassium ferrocyanide to lower heavy metal content.
Ochratoxin A	mycotoxin produced by molds Content in wine <2ppb
Glycols	Used in production to refrigerate. Pollution in case of breakage of the plants
Residual pesticides	Vineyard treatment
Chlorides, sulfates, phosphates, nitrates	Partly endogenous, partly from oenological practice*
Heavy metals (Lead, Copper, Zinc, Cadmium, Chromium)	Pollution from vineyard treatment (Cu) Pollution from production equipment (Pb, Cd, Cr) Partly endogenous (Zn)

*oenological practice: see Reg CE 606/2009

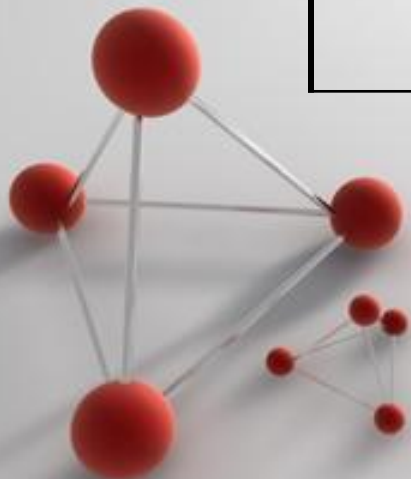


Endogenous pollutants	
Methanol	Methanol is formed from pectins (mostly present on fruit skin), during alcoholic fermentation. In Italy law limit: 0,20 ml/100mlTA for white wine 0,25 ml/100mlTA for red wine
Ethyl carbamate	Urea can spontaneously react with the alcohol in wine to form EC. Urea come from metabolized amino acids (arginine)#
Biogenic amines	Biogenic amines are important because they contain a health risk for sensitive individuals. They can be an alert of not hygienic condition in production chain



see more about EC in food : *The EFSA Journal* (2007) 551, 1-44

Allergenic compounds Dir. 2003/89/CE and Reg. 2012/579/CE	
Sulphur dioxide	Commonly used as preservative Content in wine limited (reg CE 606/2009)
Milk derivatives (casein)	Commonly used as fining agent, mostly in white wine.
Egg derivatives (ovalbumin, lysozyme)	Albumin is commonly used as fining agent, mostly in red wine. Lysozyme is used to delay or prevent malolactic fermentation.



oenological practice: see Reg CE 606/2009

Illegal practices



Search of exogenous glycerol markers:

3-Methoxypropane-1,2-diol and Cyclic Diglycerols

Search of exogenous water:

Determination of isotopic Ratio $^{18}\text{O}/^{16}\text{O}$

Search of exogenous sugars:

Determination of isotopic ratio $^{13}\text{C}/^{12}\text{C}$ of ethanol

Determination of isotopic ratio D/H of ethanol



Technological quality

Chemical-physical analysis

Organic acids

Sugars

Chromatic characteristics and polyphenolic profiles

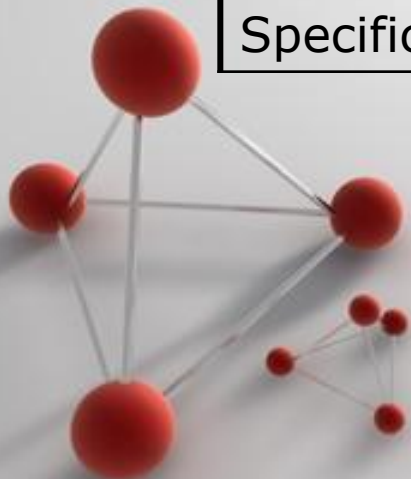
Secondary fermentation compounds

Profiles of volatile compounds (flavors profiles)

Microbiology

Molds, total bacteria counts, yeasts

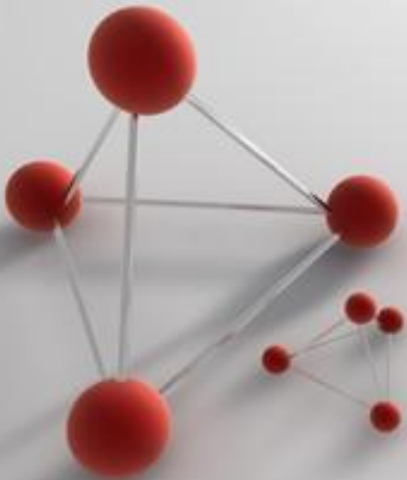
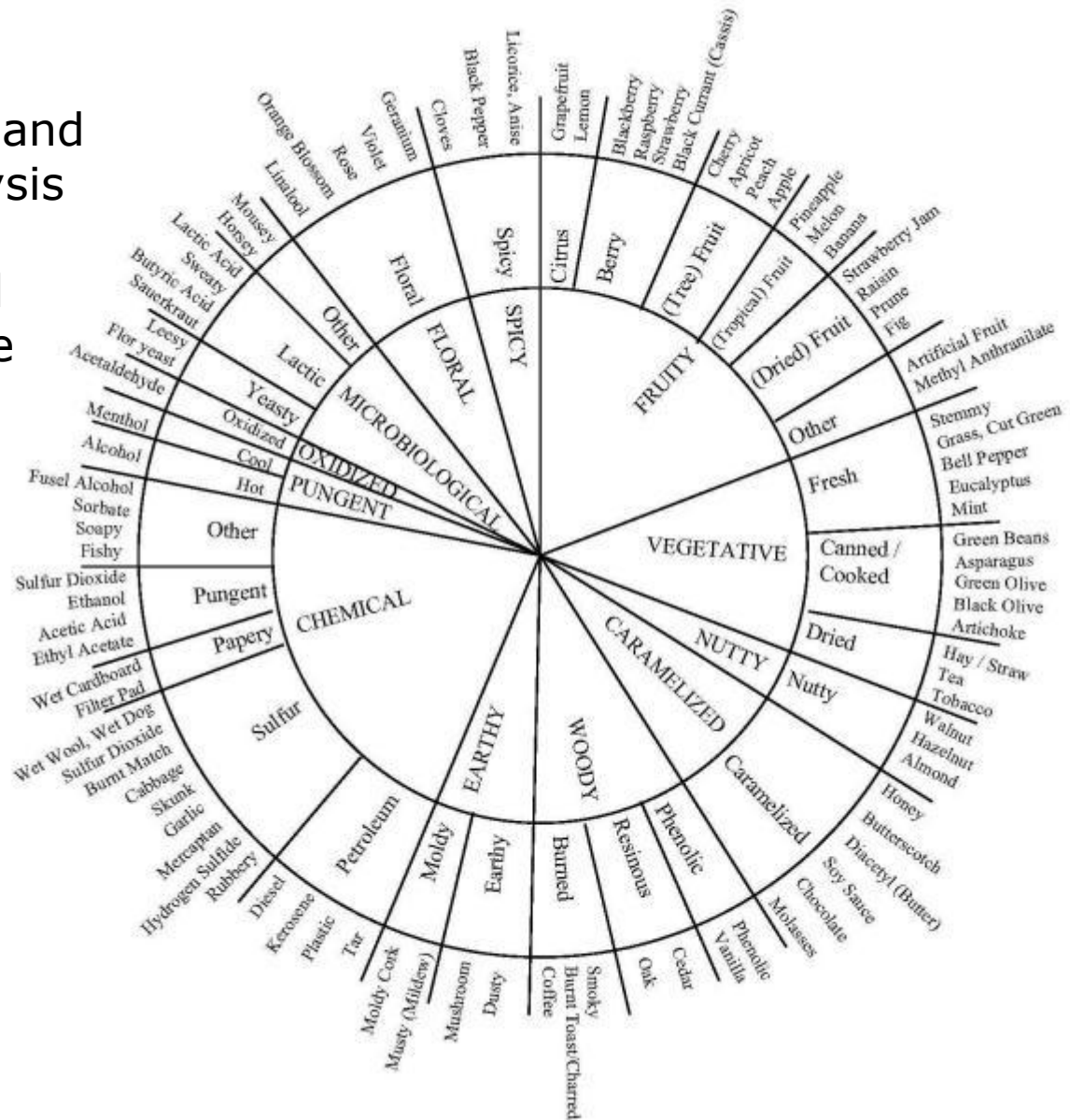
Specific searches: brettanomyces, lactic bacteria, acetic bacteria



Technological quality

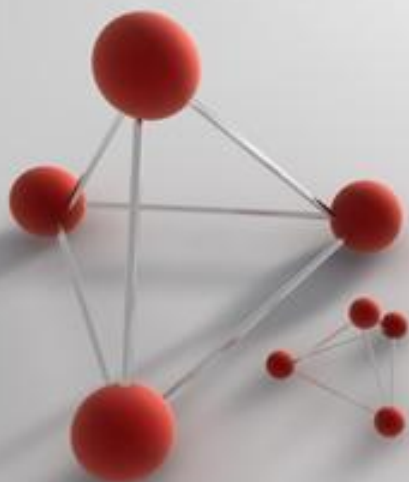
Sensorial analysis

Complementing and supporting chemical and microbiological analysis of wine-growing and production, sensorial analysis assesses the quality level of productions.



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Analytical Techniques

SPECTROSCOPY

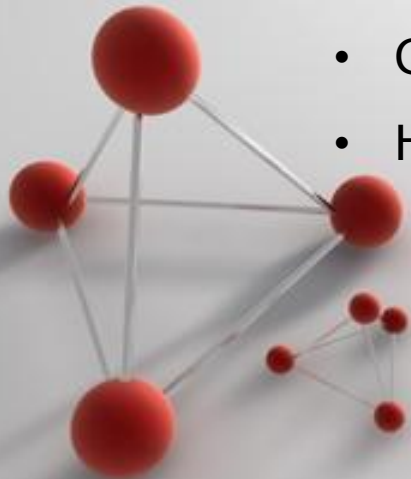
- UV-VIS spectroscopy
- Atomic Absorption spectroscopy
- ICP-MS

CHROMATOGRAPHY (and hybrid techniques)

- HPLC (UV-DAD-RI-FLD)
- GC - FID
- GC - MS
- GC - MS/MS
- HPLC - MS/MS

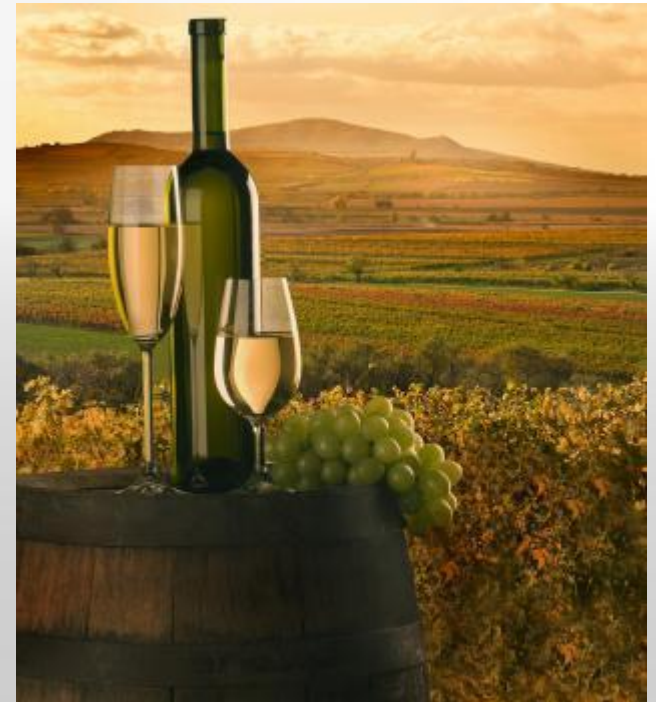
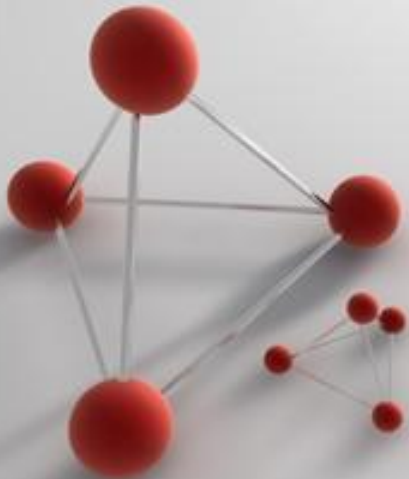
ISOTOPIC TECHNIQUES

- IRMS
- RMN



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Applications

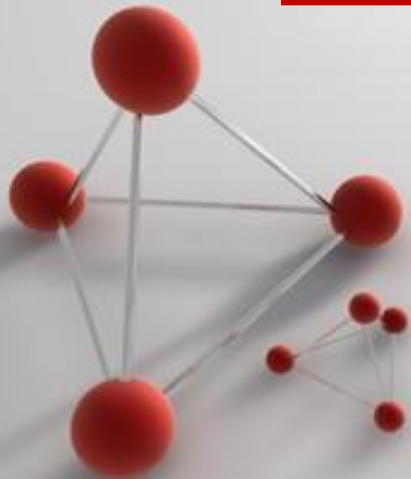
SPECTROSCOPY

UV-Vis

- Cyanide derivatives
- Chromatic characteristics

ELISA

- Casein
- Albumin



Applications

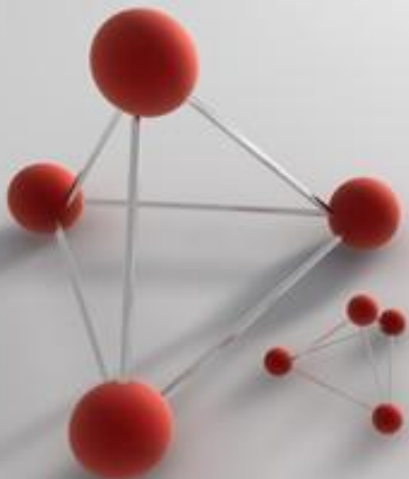
SPECTROSCOPY

AAS

- Metals (Na, K, Ca, Mg, Cu, Zn, Fe, Li)

ICP-MS

- Metals as AA
- Pb, Cr, Cd, Ni, Mn, Ni, Co, Au, Ag, As, Hg, Al and more



CHROMATOGRAPHY

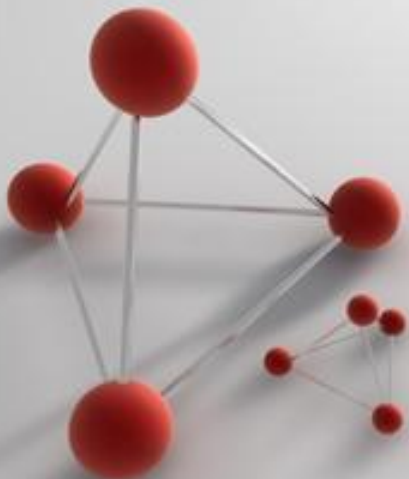
HPLC

- sorbic acid, ascorbic acid (UV)
- Organic acids (UV+RID)
- Polyphenolic profiles (DAD)
- Sugars (RID)
- Ochratoxin A (FLD)
- Biogenic Amines (UV)
- Lysozyme (UV+FLD)



Ionic Chromatography

- Chlorides, sulfates, phosphates, nitrates



Applications

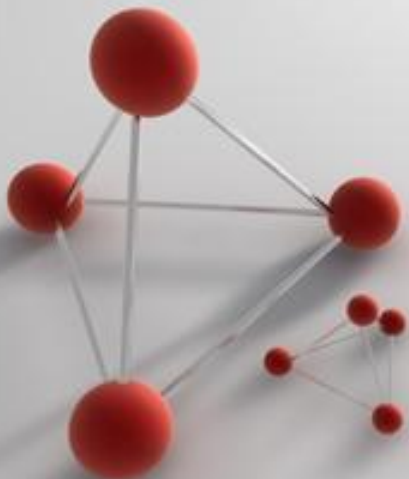
CHROMATOGRAPHY

GC - FID

- Methanol and secondary fermentation compounds
- Minor sugars in must (sucrose-maltose)

GC - MS

- Ethyl carbamate
- Glycols
- Exogenous glycerol markers
- Profiles of volatile compounds (flavors profiles)

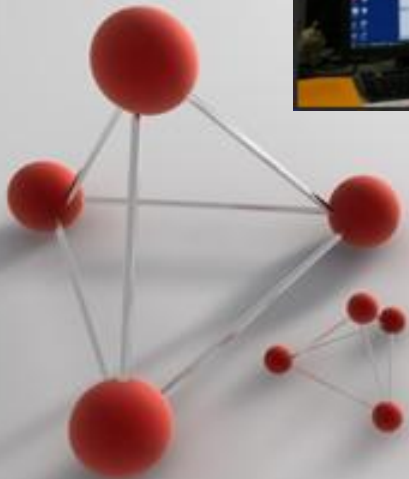
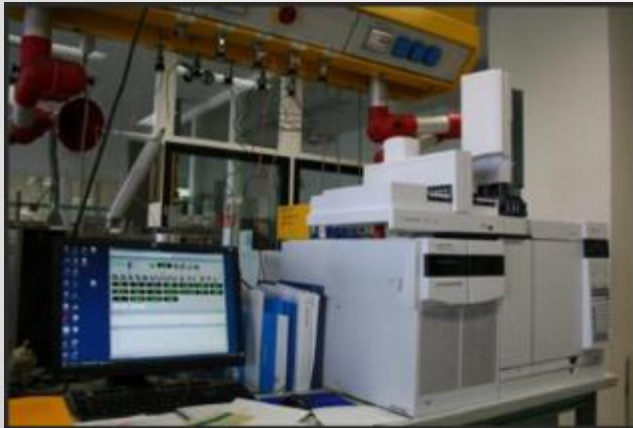


Applications

CHROMATOGRAPHY

GC – MS/MS
HPLC – MS/MS

- Residual pesticides



Applications

ISOTOPIC TECHNIQUES



IRMS

Search of exogenous water:

Determination of isotopic Ratio $^{18}\text{O}/^{16}\text{O}$

Search of exogenous sugars:

Determination of isotopic ratio $^{13}\text{C}/^{12}\text{C}$ of ethanol



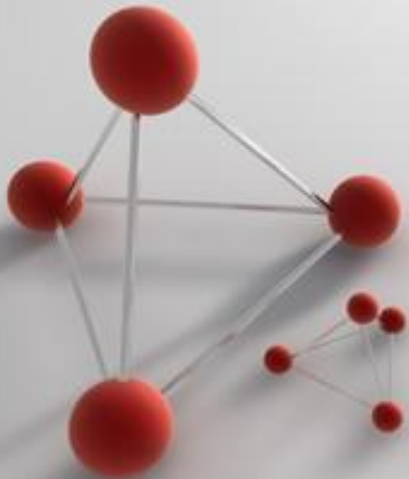
NMR

Search of exogenous sugars:

Determination of isotopic ratio D/H of ethanol

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A high throughput laboratory, such as UIV lab, needs to find methods for highest efficiency.

When we choose a method our goals are:

- Compliance to the OIV* official method requirements (if they exists)
- Sample preparation: time consumption, recovery performance, automation suitability;
- Instrumental analysis: time consumption, sensitivity, automation suitability;

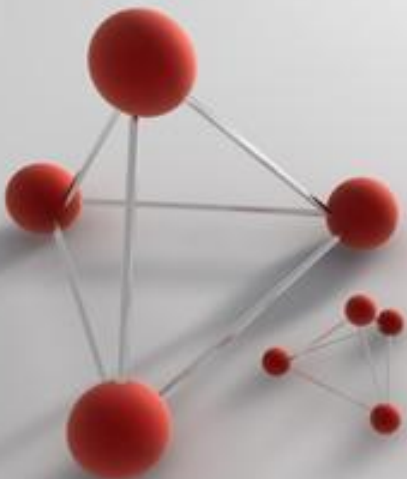
Official methods of analysis for wine

For EU they are established by:

The International Organisation of Vine and Wine (OIV)

And collected in the publication: **COMPENDIUM OF INTERNATIONAL METHODS OF ANALYSIS – OIV** (www.oiv.int)

* The OIV is an intergovernmental organisation of a scientific and technical nature of recognised competence for its works concerning vines, wine, wine-based beverages, table grapes, raisins and other vine-based products.



The evolution of the method to detect residual pesticides

Sample preparation		Instrumental equipment	
Liquid-liquid extraction	<ul style="list-style-type: none"> • Low recovery factor • Time consuming (2-h or more) • Matrix effect • NO automation 	GC ECD-FPD- NPD	<ul style="list-style-type: none"> • LOQ medium-high (50 ppb or more) • Many analysis to check different families of compound • Non volatile compounds must be derivatized
SPE extraction	<ul style="list-style-type: none"> • Good recovery factor • Time consuming* • Low repeatability* • Automation possible <p>*with automation this is better</p>	GC-MS	<ul style="list-style-type: none"> • LOQ decreased (10 ppb or more) • Detection of many families with 1 analysis • Qualitative identification improved • Non volatile compounds must be derivatized
QuEChERS	<ul style="list-style-type: none"> • Good recovery factor • Good repeatability • Quite fast • NO Automation 	LC-MS/MS GC – MS/MS	<ul style="list-style-type: none"> • LOQ decreased a lot (1 ppb) • Numbers of amenable compounds increases significantly • LC allows the analysis of non volatile compounds • Qualitative identification improved

Conclusion

Many different analytical techniques are applied in a modern, high throughput oenological laboratory like Unione Italiana Vini.

Wine is a very complex matrix, that can be a real challenge for the analytical chemist and offering ideas for continuous development.

Thank you for your attention



Unione Italiana Vini

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