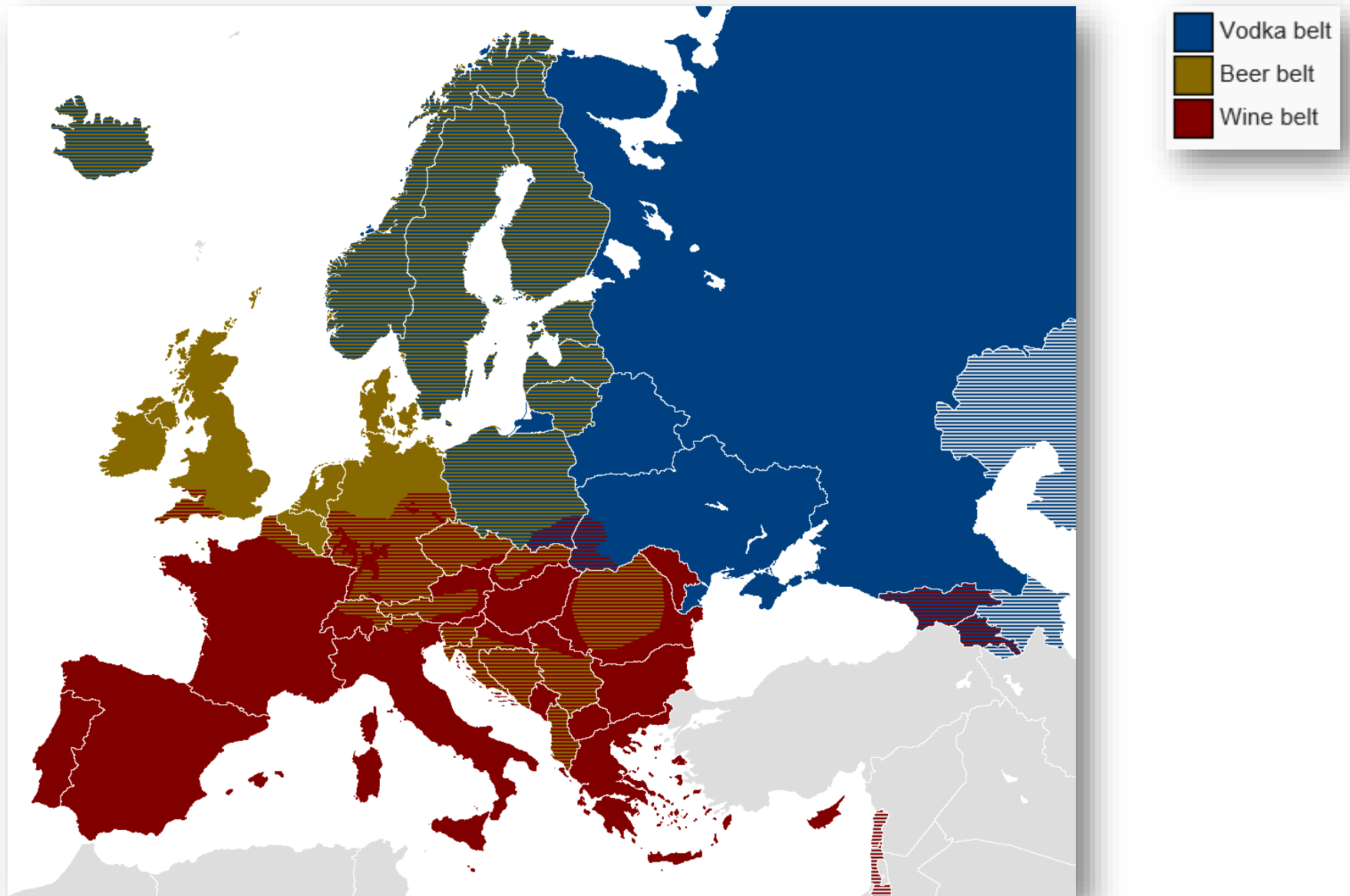


# Wine Analysis – Overview

**Uwe Oppermann**  
**Shimadzu Europa GmbH, Duisburg**

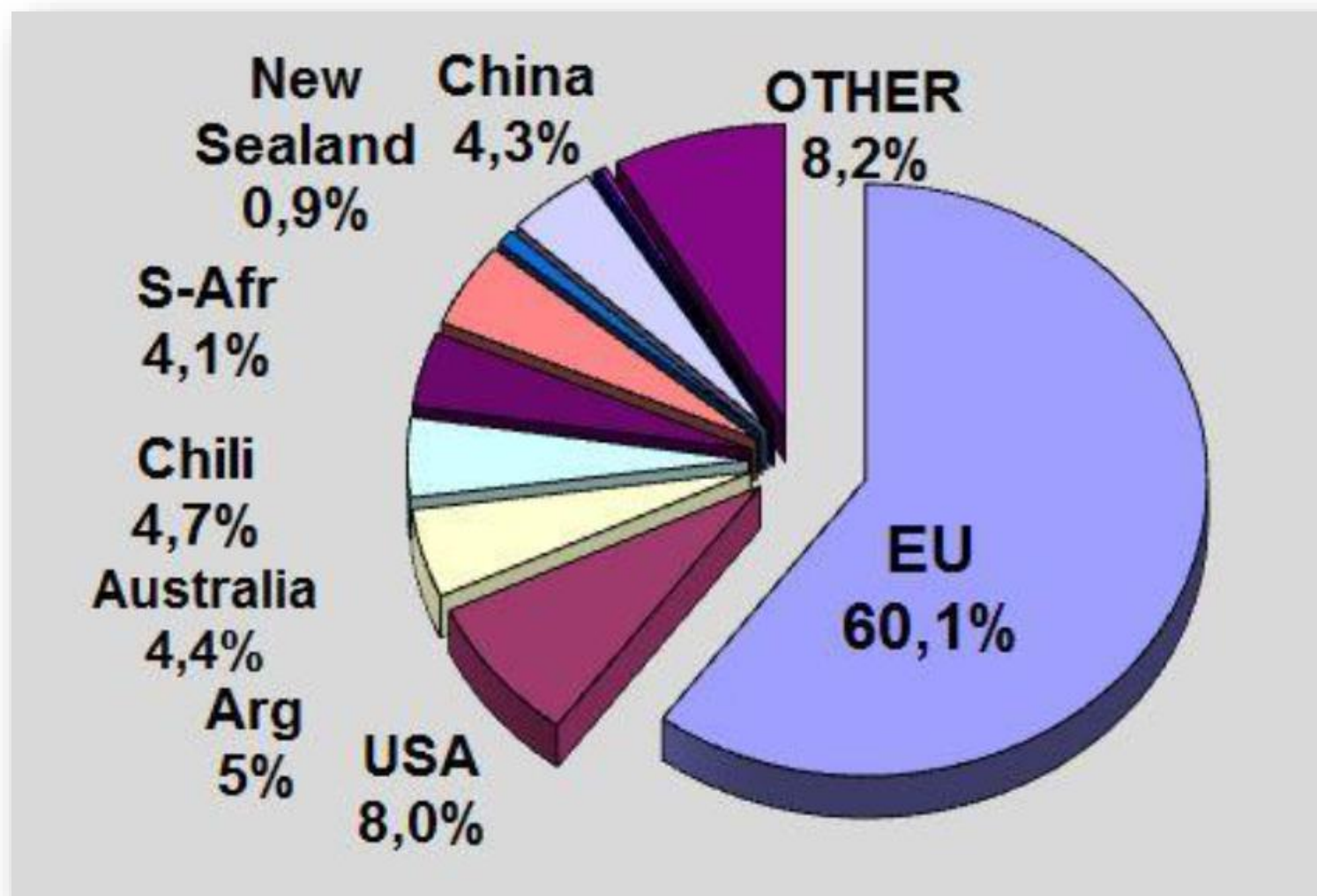


# The Alcohol Belts of Europe




[https://en.wikipedia.org/wiki/Alcohol\\_belts\\_of\\_Europe](https://en.wikipedia.org/wiki/Alcohol_belts_of_Europe)

# World Wine Production 2015



# Wine Production in Europe 2016

Organisation Commune des Marchés Règl. (CE) 1308/2013  EU-28 Production totale et vinification - CAMPAGNE 2016 – 2017 Situation au 15 Septembre 2016 _en 1.000 HL									
PAYS	Campagne 2011-12	Campagne 2012-13	Campagne 2013-14	Campagne 2014-15	Campagne 2015-16	Moyenne 5 ans (AVG5)	Production totale <sup>1)</sup> 2016-2017		
							1000 HL	Versus 2015/16 AVG5	Versus 2015/16 AVG5
Bulgarie	1 098	1 337	1 755	833	1 367	1 278	1 325	-3%	+4%
Tchéquie	650	487	501	536	819	599	631	-23%	+5%
Allemagne	9 132	9 012	8 409	9 202	8 819	8 915	8 423	-4%	-6%
Grèce	2 750	3 115	3 343	2 800	2 501	2 902	2 550	+2%	-12%
Espagne	37 199	34 241	53 233	44 080	42 001	42 151	42 500	+1%	+1%
France	51 090	41 365	41 491	47 094	47 857	45 780	42 906	-10%	-6%
Croatie	1 409	1 293	1 248	842	691	1 367	843	+22%	-38%
Italie	44 772	45 616	54 029	44 739	51 496	48 131	50 261	-2%	+4%
Chypre	85	112	108	94	79	96	69	-13%	-28%
Luxembourg	135	85	101	125	111	111	60	-46%	-46%
Hongrie	2 750	1 818	2 666	2 773	2 779	2 557	2 746	-1%	+7%
Autriche	2 756	2 155	2 392	1 999	2 268	2 314	1 750	-23%	-24%
Portugal	5 610	6 305	6 238	6 202	7 045	6 280	5 630	-20%	-10%
Roumanie	4 213	3 606	5 242	3 842	3 787	4 138	4 851	+28%	+17%
Slovénie	879	684	770		877	802	633	-28%	-21%
Slovaquie	341	324	371	286	375	339	300	-20%	-12%
United Kingdo	36	25	33	47	40	36	40	=	+11%
Other MS	21	27	29	31	41	30	40 <sup>4)</sup>	-4%	+34%
<b>Total EUR28</b>	<b>164 926</b>	<b>151 608</b>	<b>181 960</b>	<b>165 524</b>	<b>172 954</b>	<b>167 825</b>	<b>165 600</b>	<b>-4,3%</b>	<b>-1%</b>

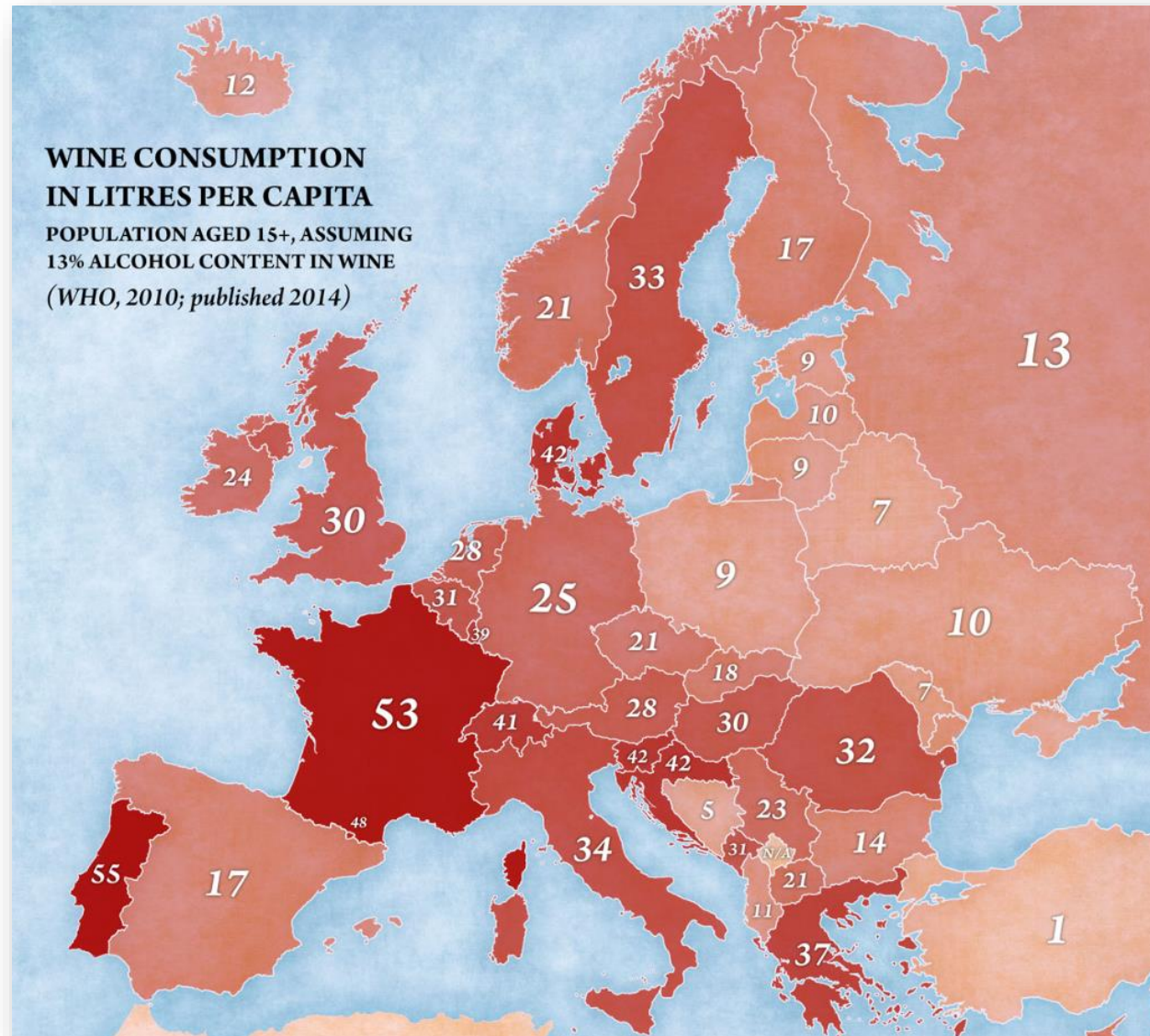
# World Wine Production

	Millionen Hektar	Millionen Hektoliter	Hektoliter pro Hektar
World	8	260	33
Europe	5	200	42
Asia	1.35	6	
America	0.75	47	62
Africa	0.31	10	
Ozeania	0.10	6	
Germany	0.11	9	81





# Wine Consumption in Europe



# Wine Analysis

## Is our wine safe????

- Nobody can answer this question
- but It has never been checked better than today
- **EFSA: Over 97% of foods in EU contain pesticide residues within legal limits**
  - 97.4% of the samples analysed fell within legal limits
  - 54.6% were free of detectable residues
  - 1.5% clearly exceeded the legal limits
  - multiple residues were found in 27.3% of samples.

# EC Approach to Food Safety \*

- Every European has the right to know how food is produced, processed, packaged, labelled and sold
- The goal of the EC's Food Safety policy is to ensure a high level of protection of human health regarding the food industry — Europe's largest manufacturing and employment sector
- The EC's guiding principle is to apply an integrated approach from farm to fork (white paper)
- [http://ec.europa.eu/food/index\\_en.htm](http://ec.europa.eu/food/index_en.htm)

\* dated 1<sup>st</sup> July 2016



# Food Safety/ Quality.....

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## Properties to determine:

- Composition
- Essential Elements
- Toxic Elements
- Taste, Color.....
- Contaminants

## Laws and Regulations:

- Drinking Water Regulation
- Beer Purity Law
- Foodstuff and Commodity Act
- European Food Law
- European Food Safety Authority

# More Regulations.....

- **European Union wine regulation**

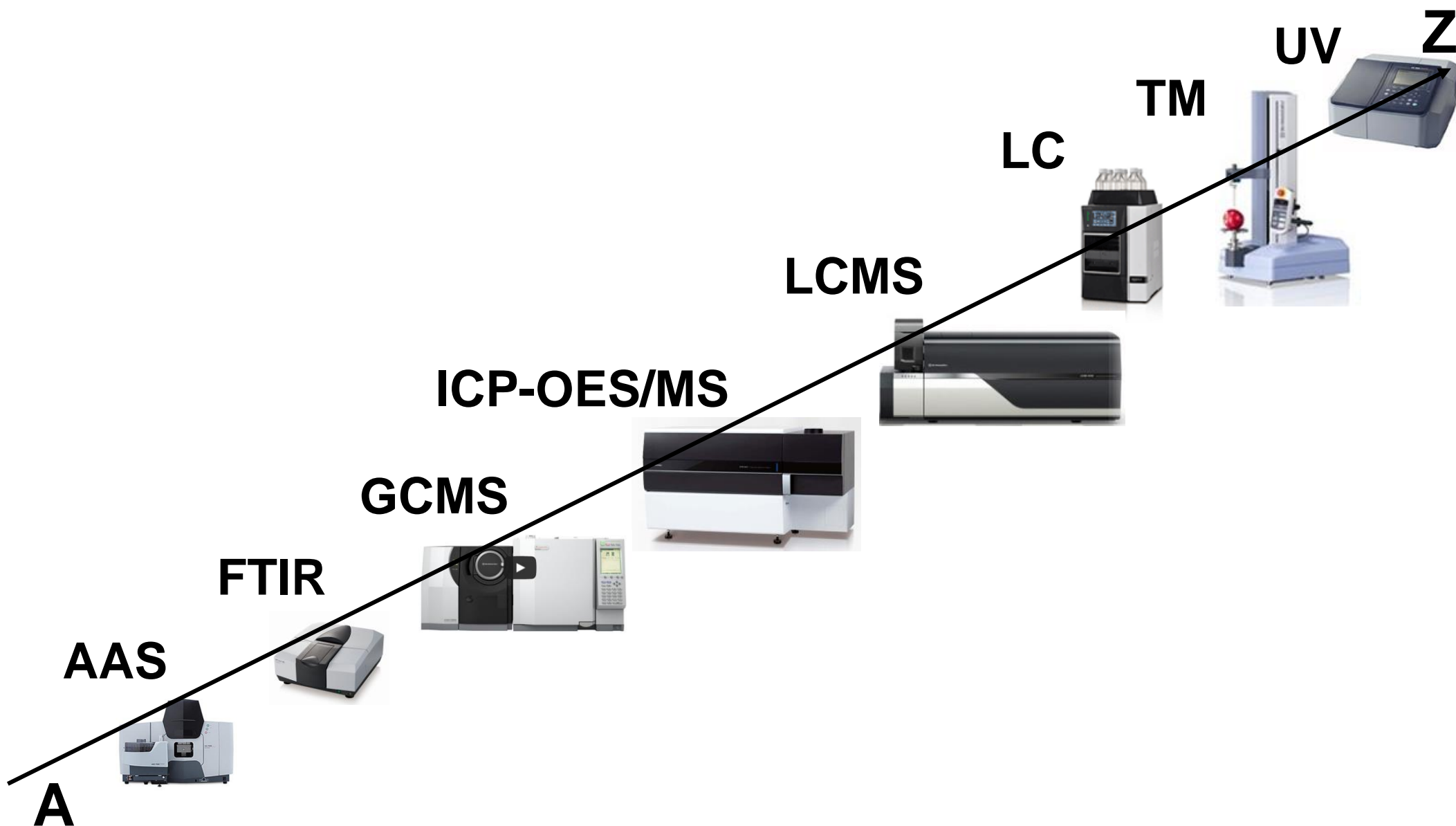
- Latest update from 9<sup>th</sup> March 2012

- **Testing physical parameters**

- Density
- Turbidity
- Color






# One Stop Vendor in Food Analysis



# European Wine Regulation

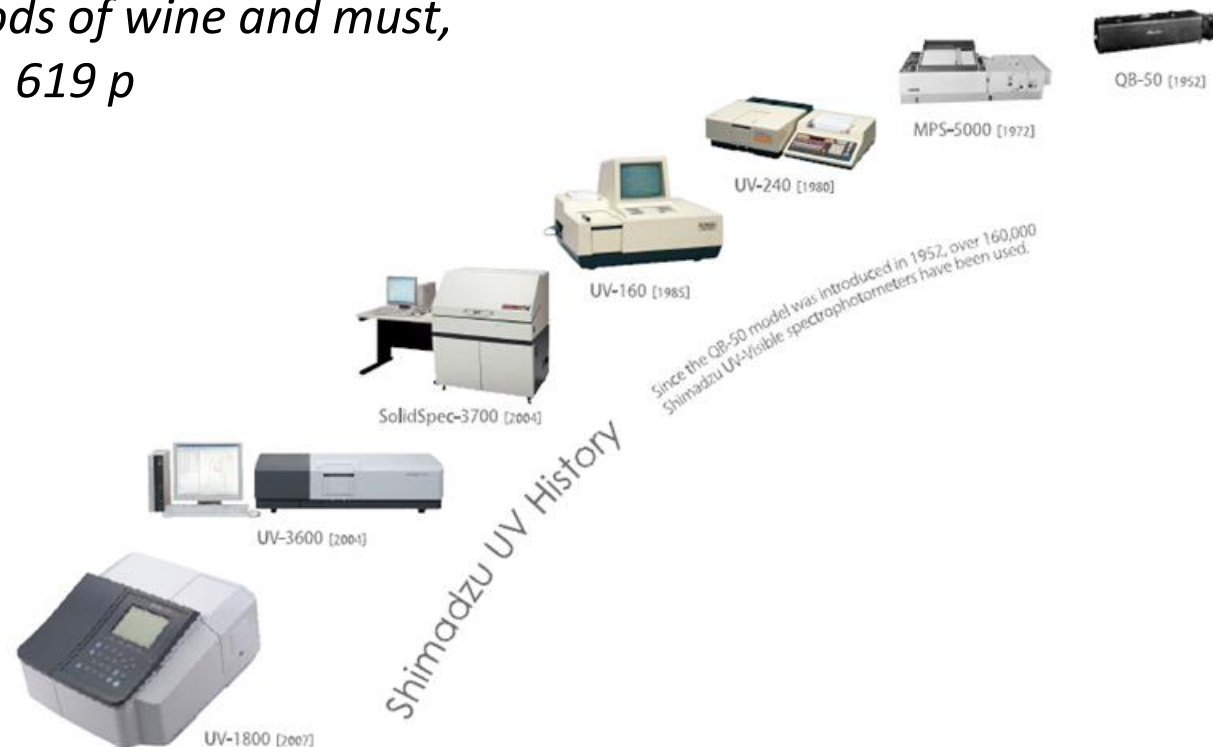
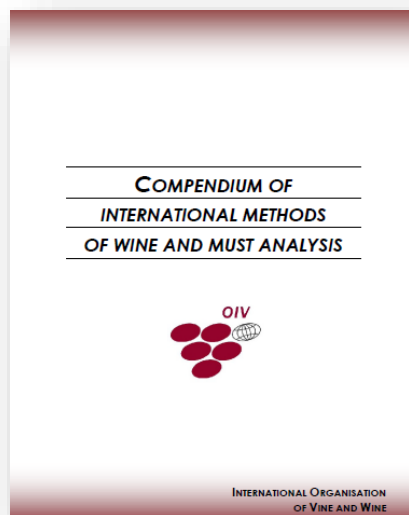
## Control Limits:

Aluminium		8,00 mg/l
Arsenic		0,10 mg/l
Lead		0,25 mg/l
Boron		80 mg/l
Cadmium		0,01 mg/l
Copper		2,00 mg/l
Zinc		5,00 mg/l
Tin		1,00 mg/l
Trichloromethane (Chloroform)		0,10 mg/l
Trichloroethene		0,10 mg/l
Tetrachloroethene		0,10 mg/l
Trichloromethane, Trichloroethene and Tetrachlorethene (total)		0,20 mg/l
Trichloroanisol (TCA)		

# Scientific Color Determination

- The International Organisation of Vine and Wine (OIV) provides methods to assess the color of a wine using spectrophotometers:

- *Compendium of international methods of wine and must, ANALYSIS, EDITION 2012 VOLUME 2, 619 p*



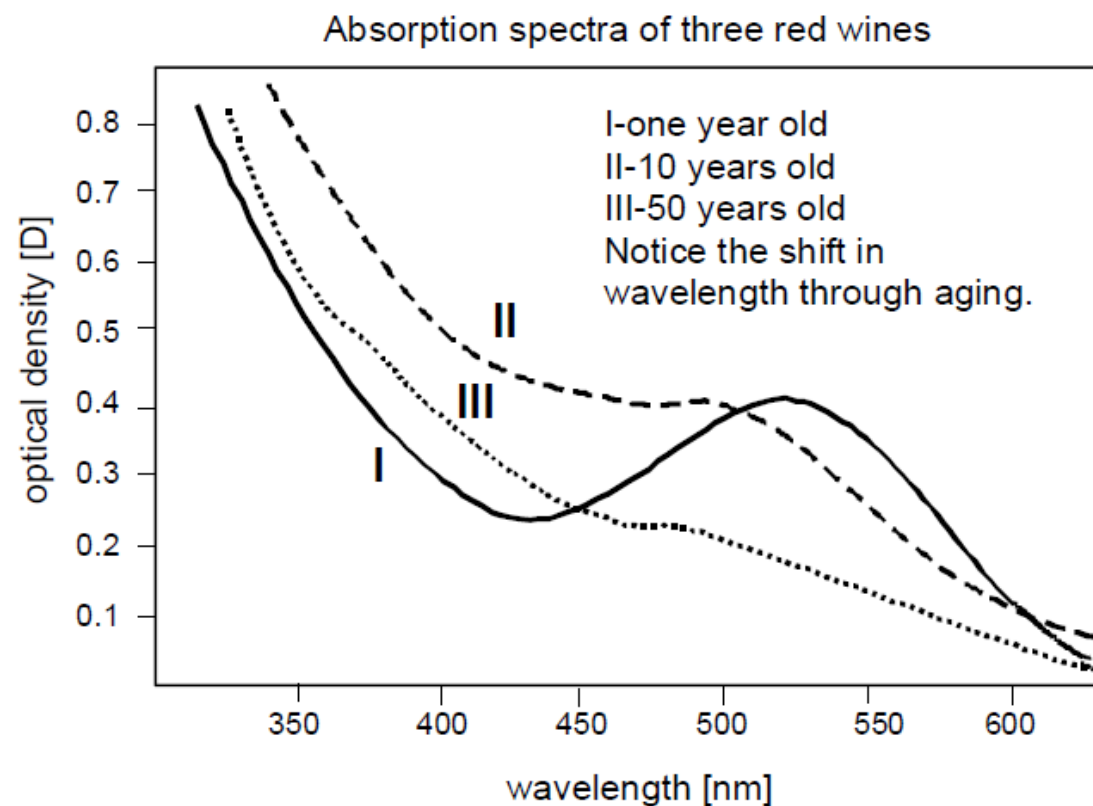
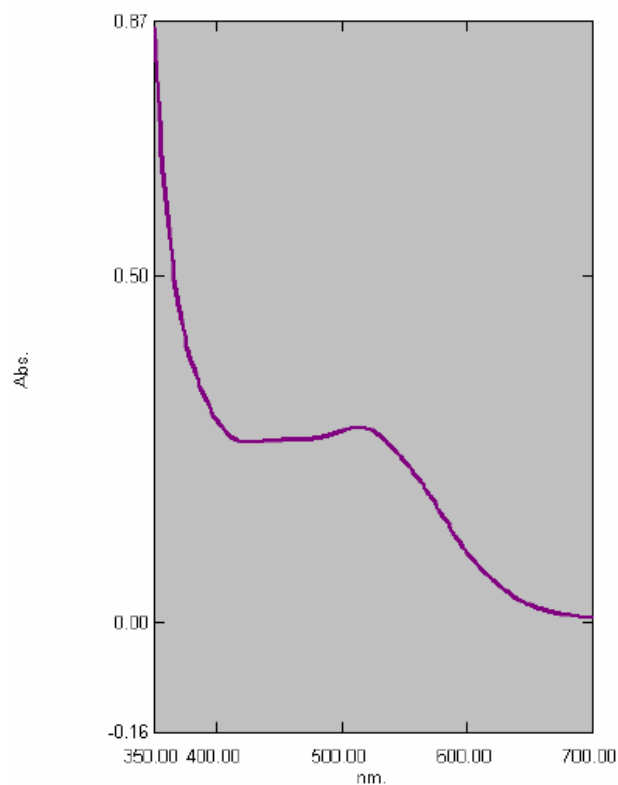


# Wine Color Analysis

- Sudraud Method:  $I = A_{420\text{nm}} + A_{520\text{nm}}$



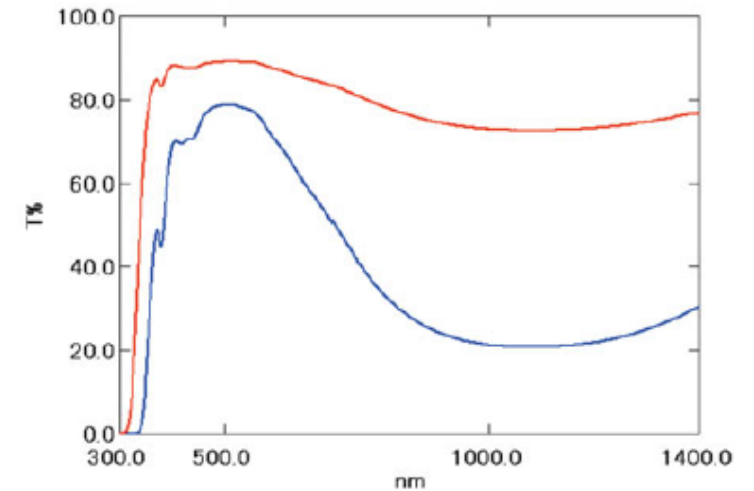
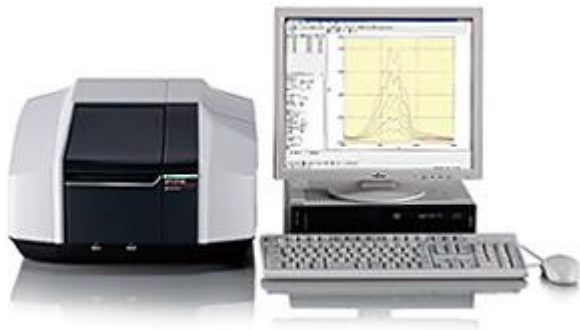
UV-1800



# Important Applications

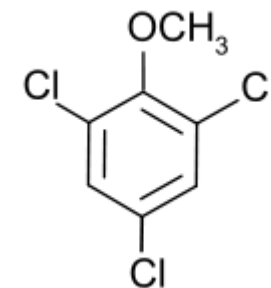
## ● Transmission/ Reflection of Glass

- Sun protective glass with Fe (II)/ (III), Cu (II) oxid
- UV-2600 with ISR-2600plus



# 2,4,6-Trichloranisole (TCA)

- Chlorinated aromatic hydrocarbon
- Metabolite of 2,4,6-Trichlorophenol (fungicide)
- Causing cork tainted wine
- Bad smell/ taste



- <http://www.ssi.shimadzu.com/products/literature/gcms/laan-j-ms-073.pdf>

# Analysis of TCA in Wine

....using GCMS-QP2010 Ultra with HS-20trap head space sampler

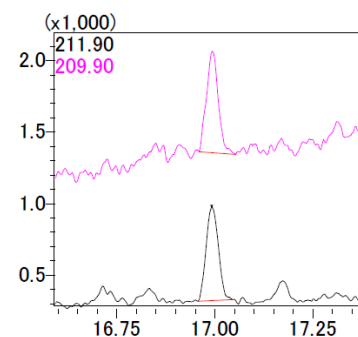


Figure 1: SIM Chromatogram of TCA in Wine using HS-Trap (wine spiked with 1 ng/L TCA)

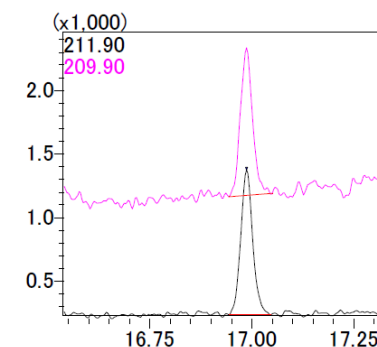


Figure 2: SIM Chromatogram of TCA in Wine using conventional headspace GCMS (wine spiked with 100 ng/L TCA)



# Flavor Analysis with GCMSMS

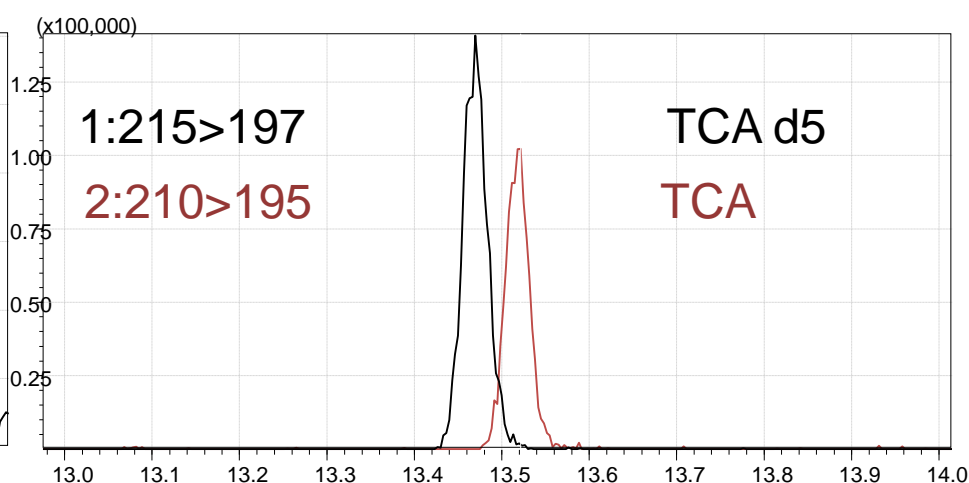
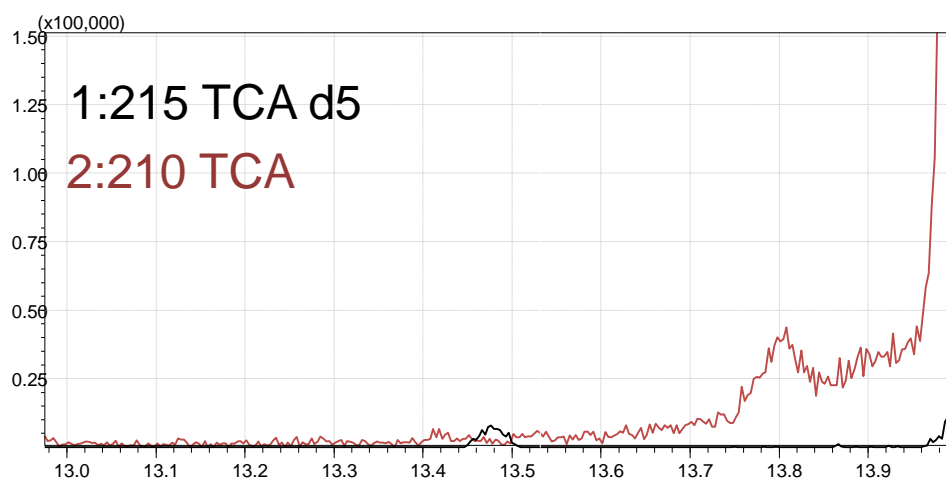
- Enhanced selectivity: Triple-quadrupole technology



GCMS-QP2020



GCMS-TQ8040





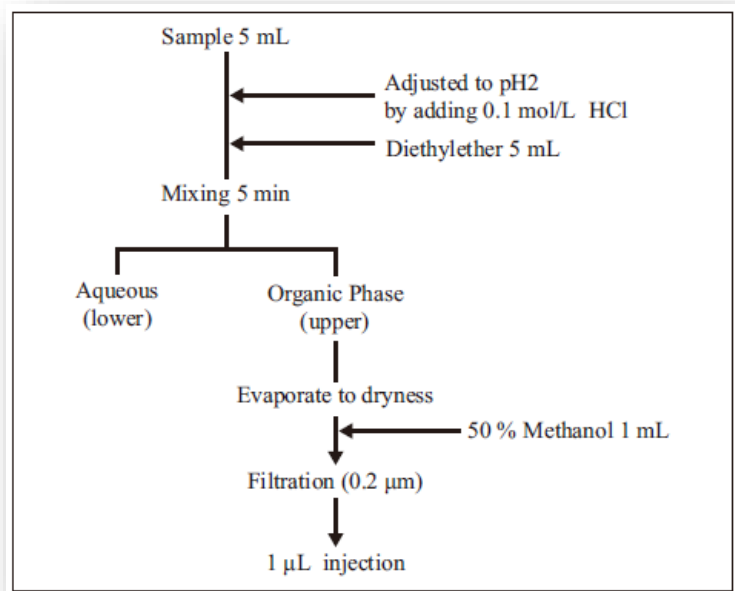
# Resveratrol Analysis

- Phytoalexin/ Polyphenol (Antioxidant)
- Present in the skin of red grapes
- Health benefits (cancer therapy)
- Dietary supplement for increased longevity
- .....using Nexera UHPLC/ RF-20Axs
- <http://www2.shimadzu.com/applications/LC/L413.pdf>

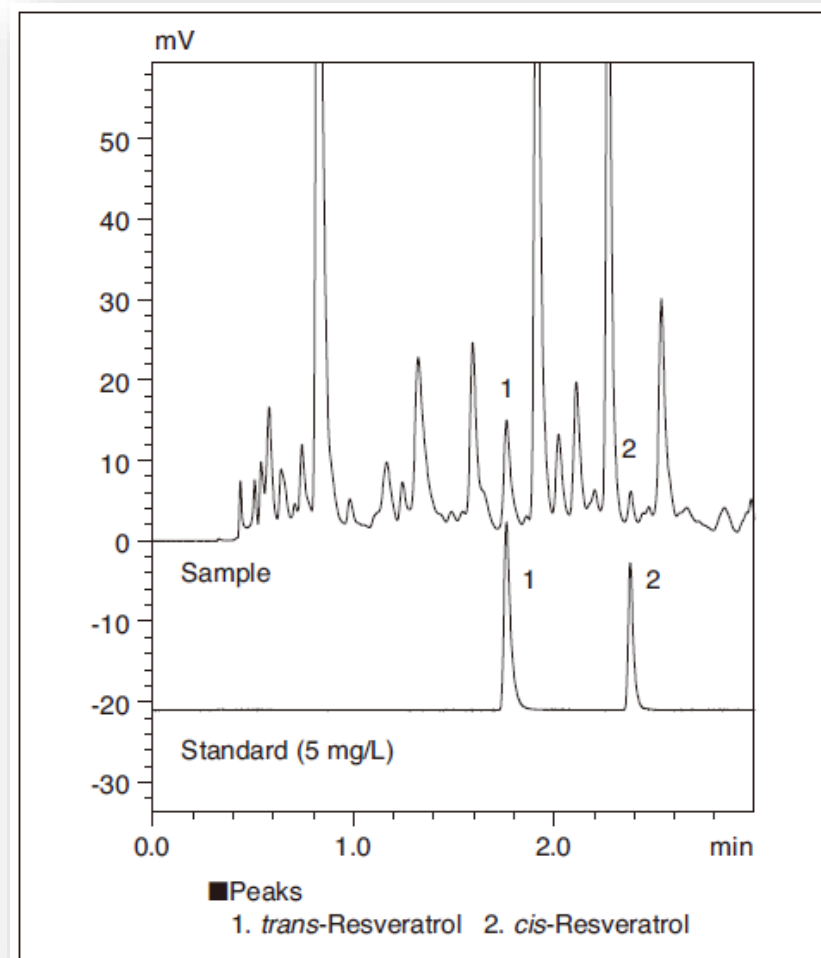


Column	: Shim-pack XR-ODS III (150 mm L. × 2.0 mm I.D., 2.2 μm)
Mobile Phase	: A : 0.2 % Formic acid - Water B : 0.2 % Formic acid - Acetonitrile
Time Program	: B Conc. 23 % (0.00 min) → 26 % (1.00 min) → 40 % (2.50 min) → 100 % (2.51-4.00 min) → 23 % (4.01 min)
Flow Rate	: 0.7 mL/min
Column Temp.	: 60 °C
Injection Volume	: 1 μL
Detection	: RF-20Axs Ex. at 300 nm, Em. at 386 nm
Cell Temp.	: 20 °C
Flow Cell	: Semi-micro cell

## Analytical Conditions



## Sample Preparation

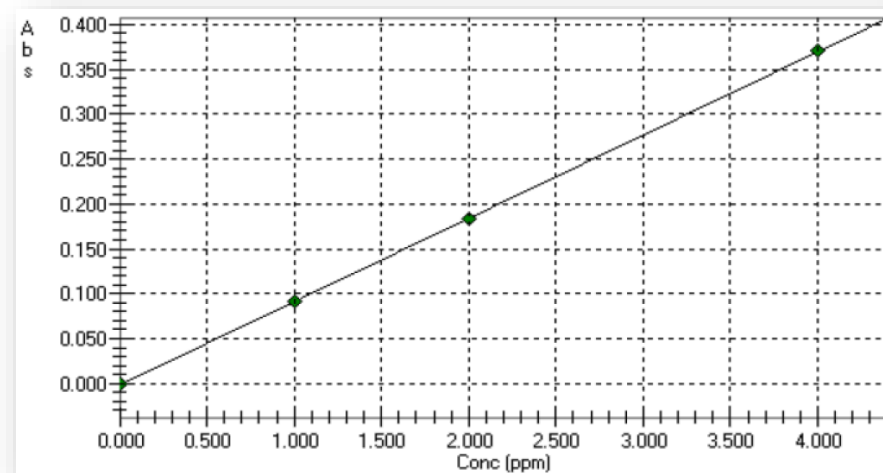


# Chromatogram of Red Wine

# Quantitation of Heavy Metals (1)

Sequential.....

...in Flame and furnace atomization using AA-7000  
Determination of Na, K, and Cu



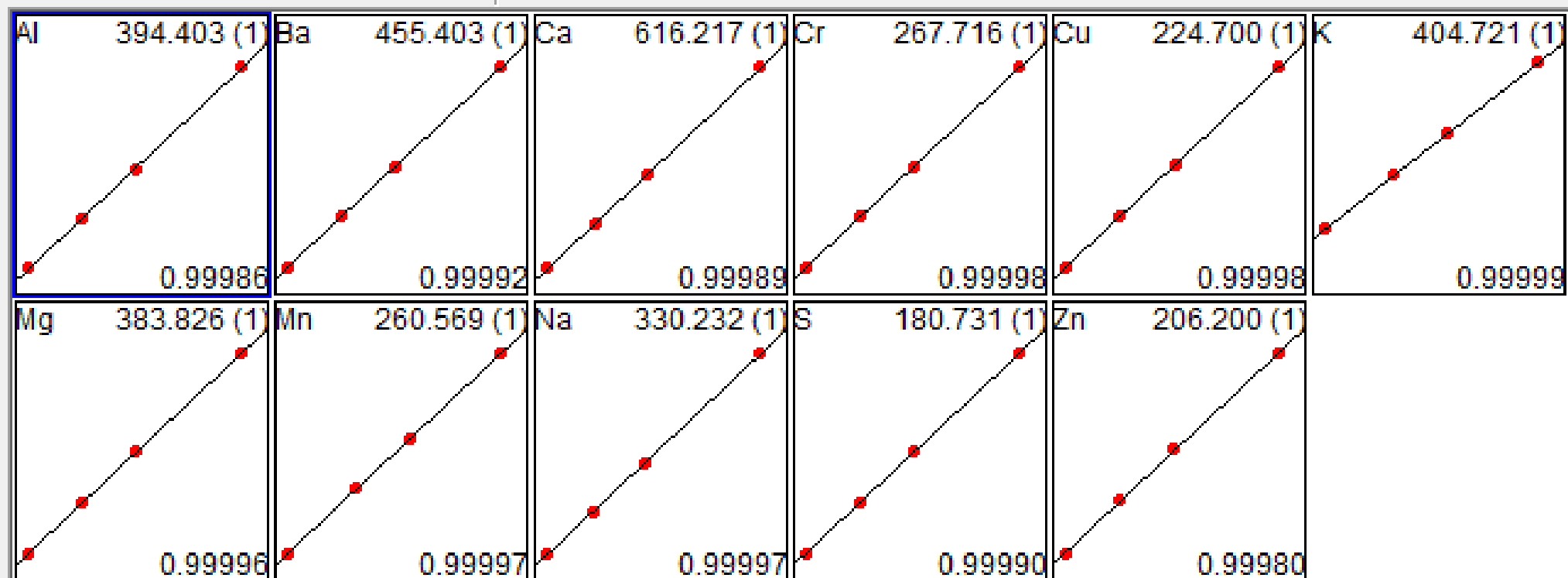
# Quantitation of Heavy Metals(2)

....using simultaneous ICP-OES: ICPE-9820



# Multielement Calibration

Selected Calibration range from: 0 - 1000 µg/L





# Quantitation of Heavy Metals(3)

....using high sensitivity ICPMS-2030



Parameter	Setting
RF generator power	1.2 kW
Plasma gas	8 l/min
Auxiliary gas	1,1 l/min
Carrier gas	0.7 l/min
Nebulizer type	MicroMist
Sampling depth	6 mm
Spray Chamber temperature	5°C
Coll. Cell gas flow (He)	4 ml/min (std) 8 ml/min for As <sup>75</sup> and Se <sup>78</sup>
Quantified Isotopes	V <sup>51</sup> , Cr <sup>52</sup> , Mn <sup>55</sup> , Fe <sup>56</sup> , Ni <sup>60</sup> , Cu <sup>63</sup> , Zn <sup>66</sup> , As <sup>75</sup> , Se <sup>78</sup> , Cd <sup>111</sup> , Sn <sup>118</sup> , Cs <sup>133</sup> , Tl <sup>205</sup> , Pb <sup>208</sup>
Internal Standards (ISTD)	Sc <sup>45</sup> , Ge <sup>72</sup> , Y <sup>89</sup> , In <sup>115</sup> , Tb <sup>159</sup> , Ho <sup>165</sup> , Lu <sup>175</sup> , Bi <sup>209</sup>



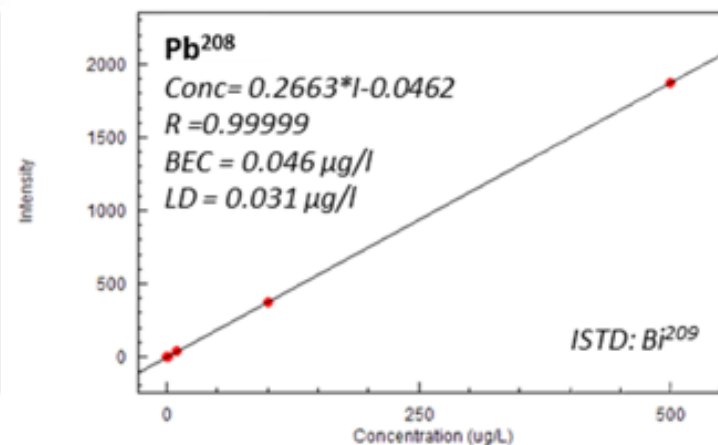
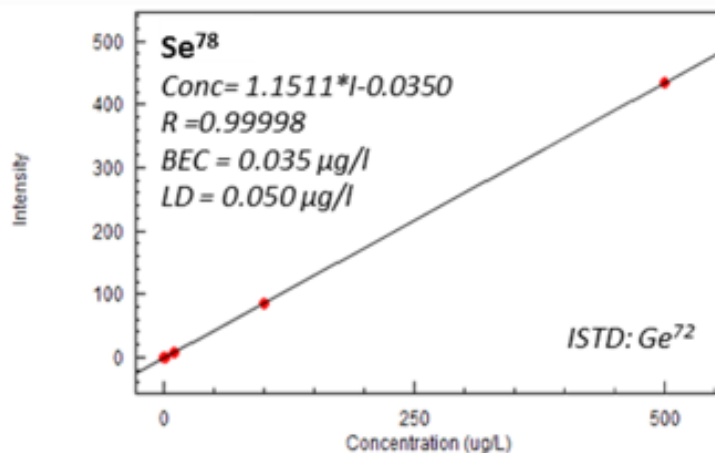
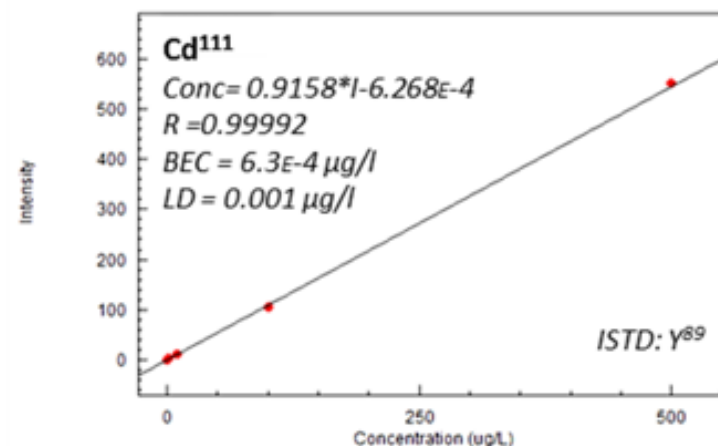
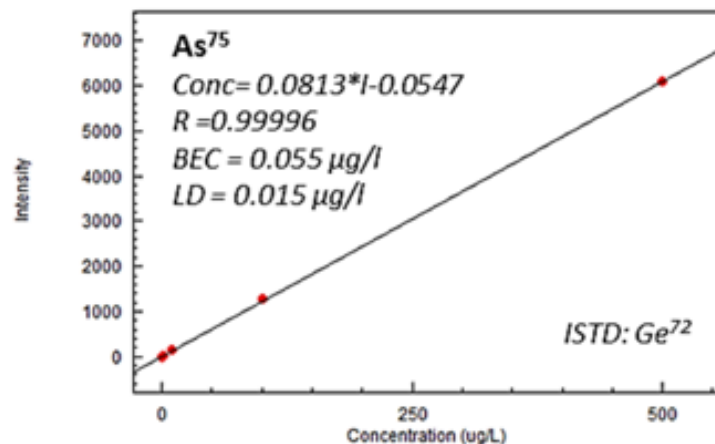
# ICP- Spectrometry coupled to Mass



## ● Analysis Technique:

- **Multi-element analysis** : simultaneous as in ICP-OES
- Extremely sensitive => analysis of ultra-traces (< ppb => ppt).

# Multielement Analysis



# Physical Analysis

- Cylindrical cork stoppers
  - Wine and sparkling wine bottles
- Determination of extraction force
- ISO 9727-5:2007
- Bottle height from 250 ml bis 750 ml



# Conclusion

Shimadzu is offering the full range of instruments  
for  
„state of the art“ solutions in wine analysis  
in order to maintain the highest level of food quality  
and food safety.



# Questions

● ???????



## Brand Statement "Excellence in Science"

We in the Shimadzu Group have delivered products and services to enable our customers around the world to develop a diverse range of new products, to protect and improve the environment, and to improve the health and lives of mankind. This brand statement represents our sense of pride in this endeavor. It is our commitment to society and ourselves that Shimadzu remains dedicated in our pursuit of technology and accumulation of knowledge, so that we can offer even more outstanding technologies, products, and services, so as to be recognized for excellence in the field of science.