



## ARIMNet2 Mid-Term Project Evaluation Meeting 12 October 2017, Montpellier, France

### MedOOmics

# Mediterranean Extra Virgin Olive Oil Omics: profiling and fingerprinting

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# CONSORTIUM



**Project start date:** 01/09/2016 - **Project end date:** 31/08/2019

- **Universidade de Évora (Portugal) (Coordinator):** stable isotopes, mineral elements and NMR Spectroscopy
- **Universidade Nova de Lisboa - Portugal:** comprehensive two-dimensional gas chromatography (if needed)
- **Adana Science and Technology University- Turkey:** minor compounds of olive oil
- **Cukurova University - Turkey:** minor compounds of olive oil
- **Aix-Marseille Université, France:** chemometric and statistical techniques, Raman and Infrared Spectroscopy
- **Centre Technique de l'Olivier, France:** sampling French olive oils
- **Institut Supérieur de Biotechnologie de Sfax, Université de Sfax, Tunisie:** major compounds of olive oils



# PROJECT OBJECTIVES

## Objective 1

- A full characterization of varietal EVOOs from Portugal, France, Turkey and Tunisia, targeting some less studied and typical varieties of each country.

## Objective 2

- Establish tools that enable to ascertain the geographical origin of EVOOs, that can be further used for Geographical Indication of Origin (IGP) and Protected Designation of Origin (PDO) certification

# PROJECT OBJECTIVES

## Objective 3

- Establish chemical markers to be used for EVOOs authenticity

## Objective 4

- Transfer of some methodologies developed within this proposal to industries

# SCIENTIFIC APPROACH

## Profiling

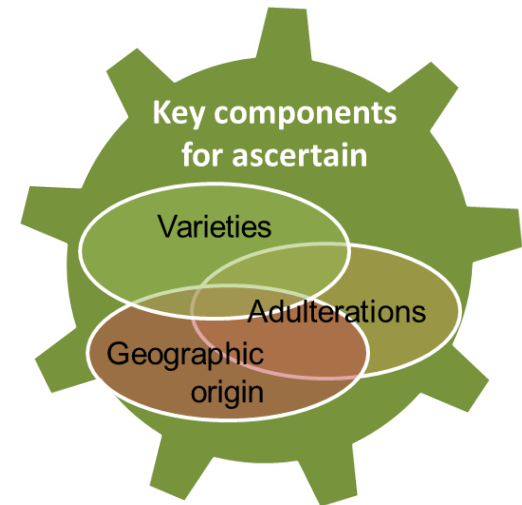
- ICP/MS
- SIRMS
- HPLC-DAD; HPLC-FLD; HPLC/MS
- GC/MS; GC x GC

## Fingerprinting

- NMR
- MIR
- NIR
- Raman

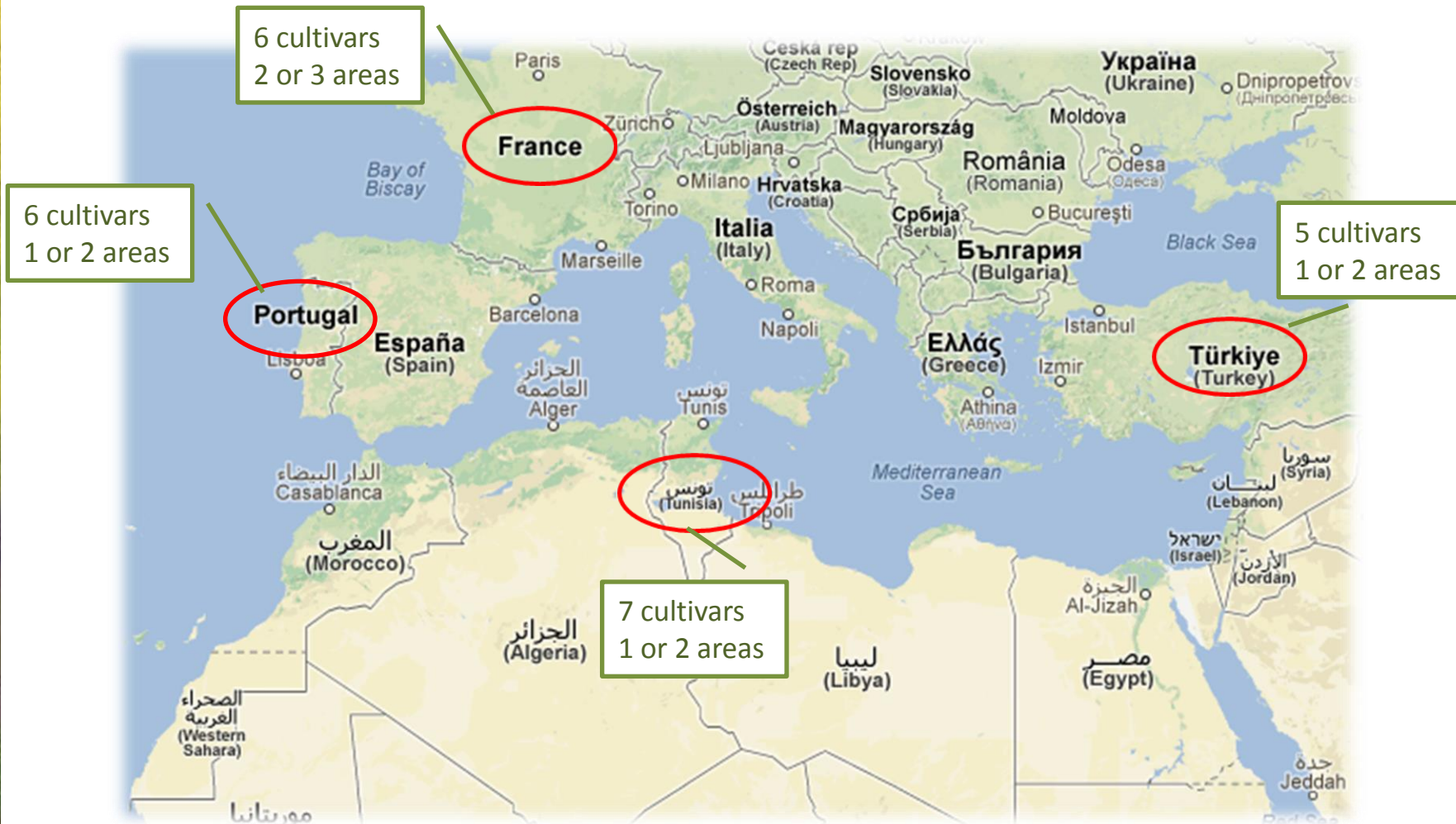
Chemometrics  
and statistics

## Olive oil metabolomics

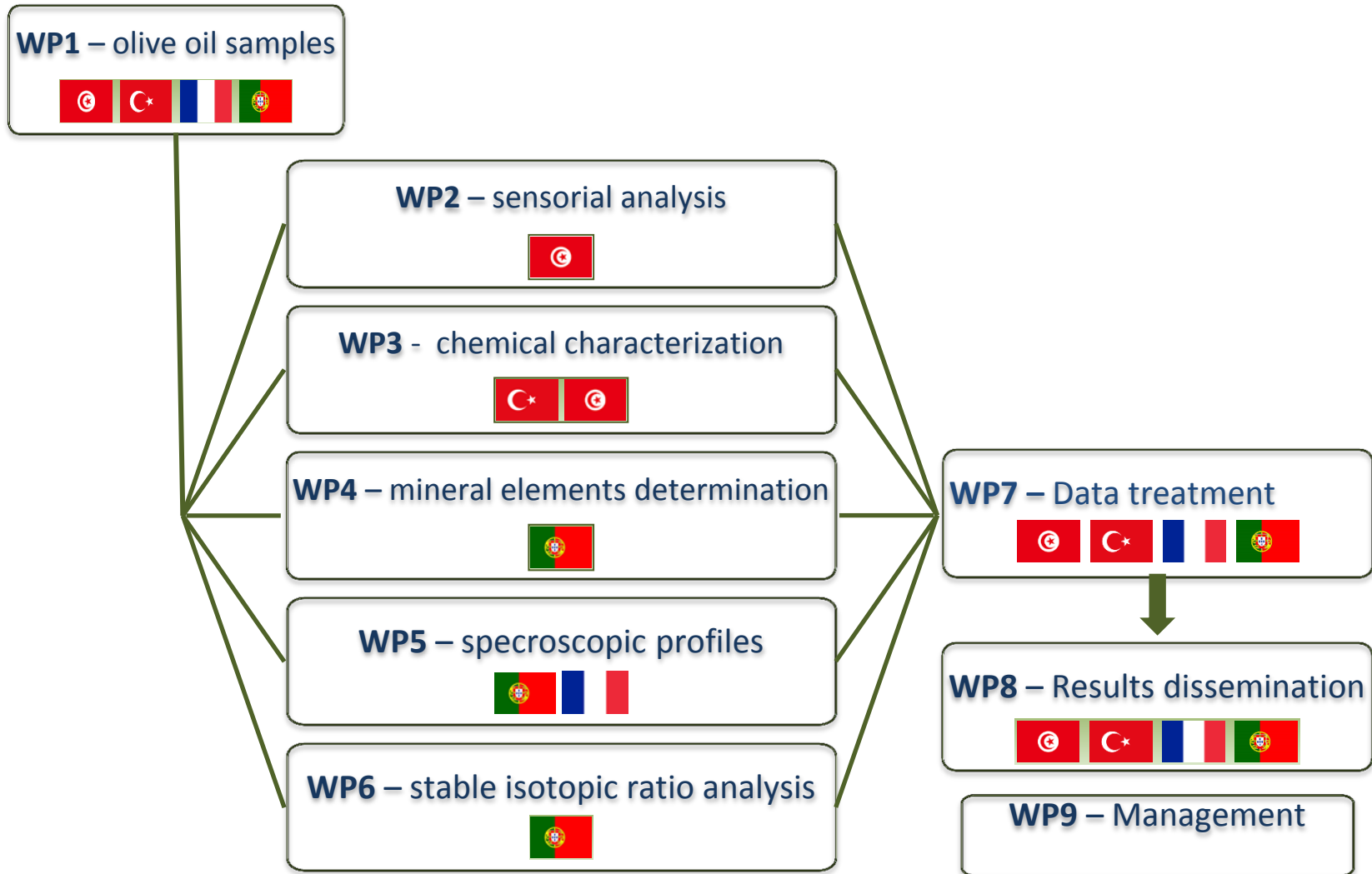




# STUDY AREA / SITES



# ACTIVITIES / WORK PACKAGES



# RESULTS SO FAR

## Olive Oil samples (W1)

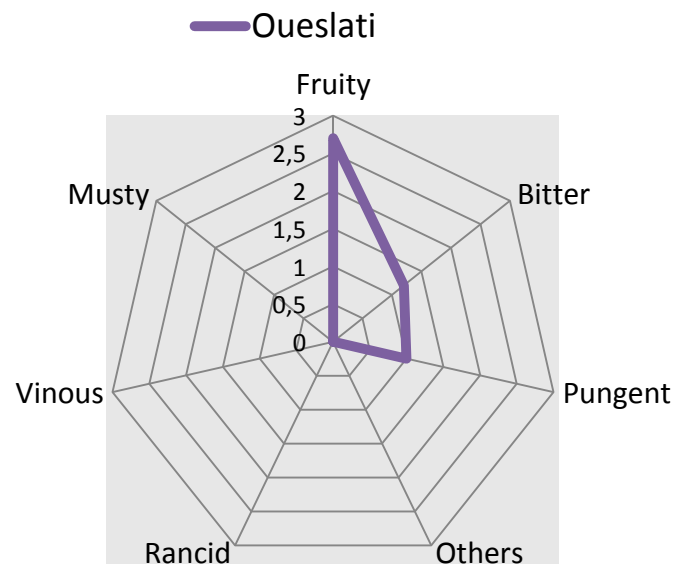
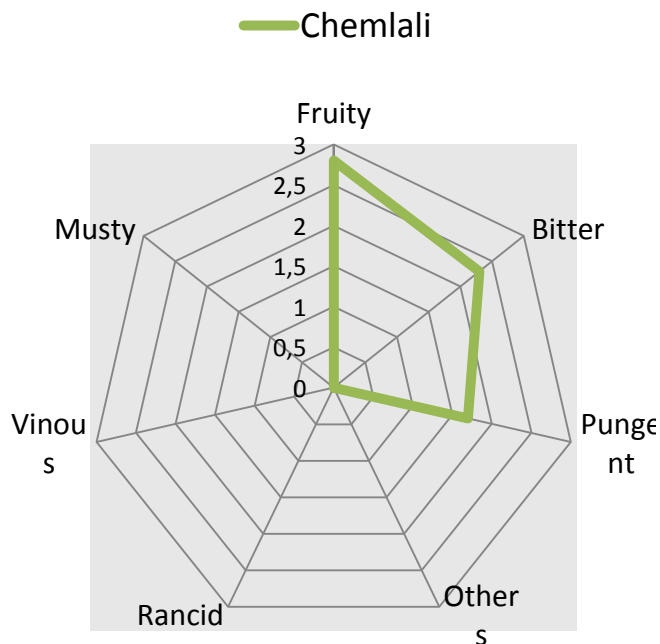
- **France** — Aglandau, Picholine, Salonenque, Cailletier, Tanche, Olivière
- **Portugal** — Cobrançosa, Galega Vulgar, Cordovil de Serpa, Blanqueta, Verdeal e Madural
- **Tunisia** — Oueslati, Zarrazi, Jemri, Zalmati, Neb Jmal, Chemlali, Chétoui
- **Turkey** — Halhali, Hasebi, Kilis, Nizip, Sariulak



# RESULTS SO FAR

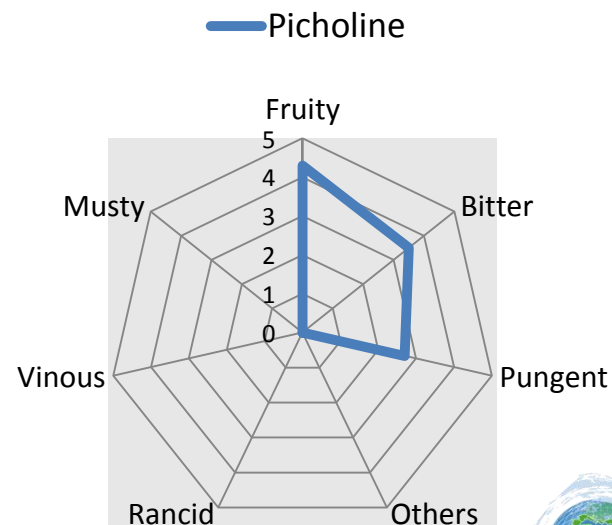
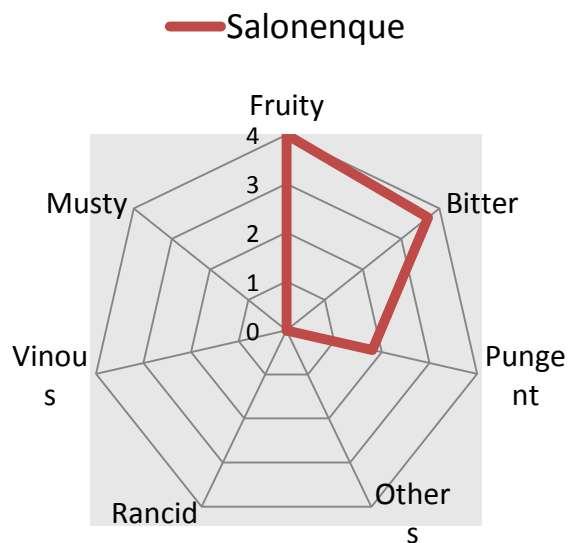
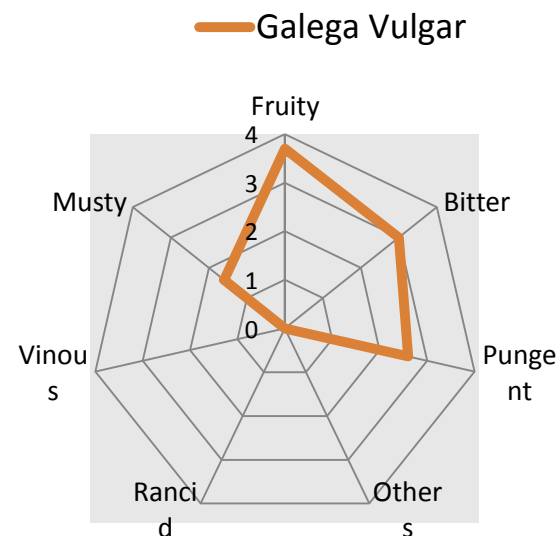
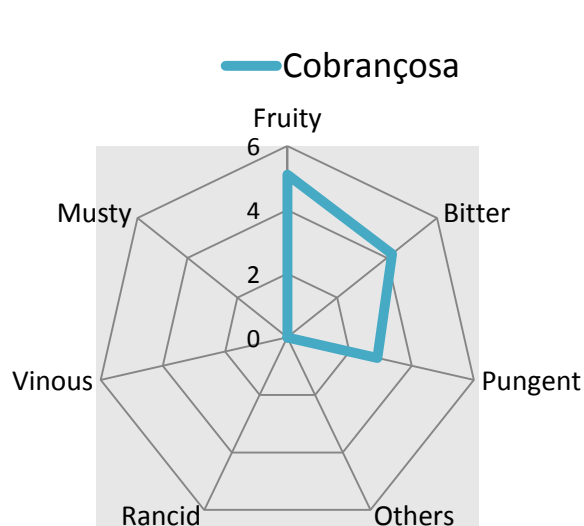
## OLIVE OIL TASTING PANELS (W2 -Tunisia)

- A panel is established (following IOC standards) for organoleptic assessment of virgin olive oil from Tunisia, Portugal, France Turkey



# RESULTS SO FAR

## OLIVE OIL TASTING PANELS (W2 -Tunisia)

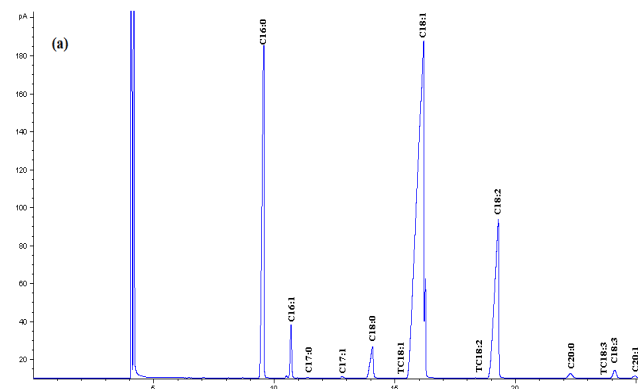
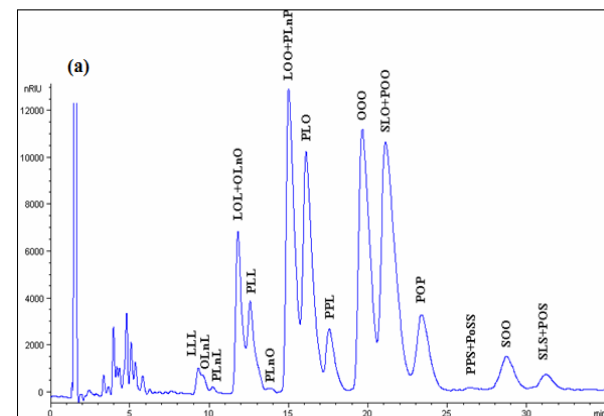


# RESULTS SO FAR

## Chemical characterization (W3-Tunisia)

- Liquid and Gas Chromatography methodology for analysis of Triglycerides and fatty acid composition were established

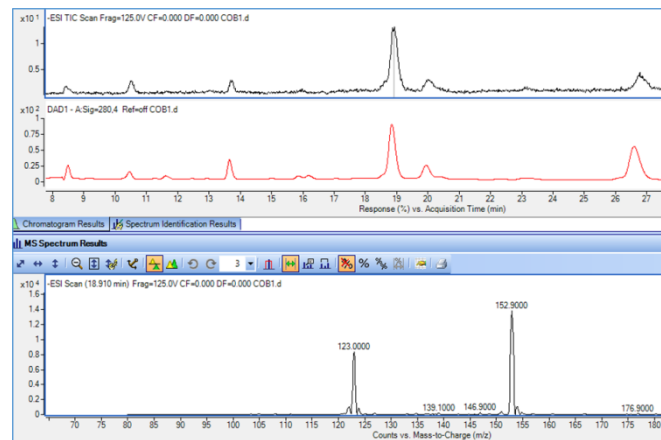
Fatty Acids (%)	France		Tunisia		Portugal	
	Picholine	Salonen-que	Chemlali	Oueslati	Cobranço-sa	Galega Vulgar
C14 :0	0.01	0.01	0.01	0.01	0.01	0.01
C16 :0	11.84	15.95	20.35	11.43	12.68	14.84
C16 :1w7	0.78	1.36	2.56	0.66	1.04	2.69
C17 :0	0.07	0.08	0.06	0.08	0.15	0.12
C17 :1w8	0.11	0.14	0.03	0.05	0.23	0.30
C18 :0	2.43	2.51	2.50	3.18	4.18	1.67
C18 :1w9	75.13	65.74	59.30	71.89	71.51	73.69
C18 :2w6	8.19	12.93	20.50	11.15	8.65	5.27
C18 :3w3	0.79	0.62	0.80	0.71	0.86	0.82
C20 :0	0.36	0.42	0.43	0.50	0.48	0.32
C20 :1w9	0.29	0.24	0.20	0.35	0.21	0.27
Σ AGS	14.71	18.97	23.52	15.2	17.5	16.96
Σ AGIS	85.29	81.03	86.86	84.81	82.5	83.04
C18:1/C18:2	9.19	5.08	2.89	6.44	8.26	13.98



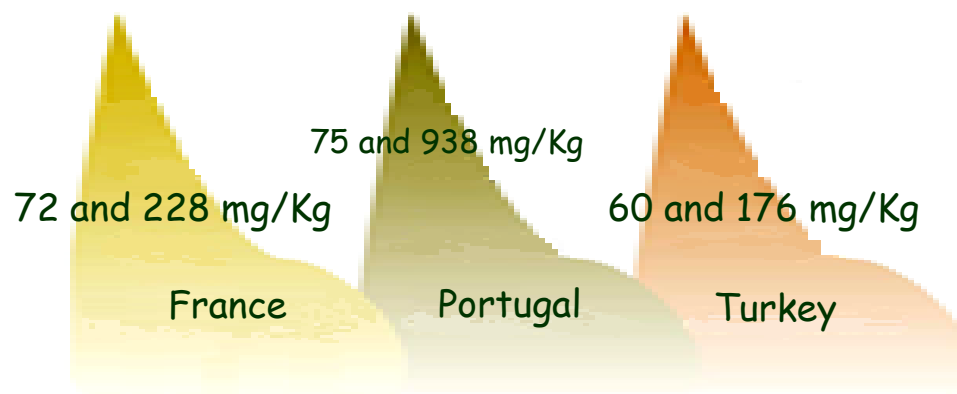
# RESULTS SO FAR

## Chemical characterization (W3-Turkey)

- LC-DAD-ESI-MS/MS methodology for analysis of phenolic compounds is established



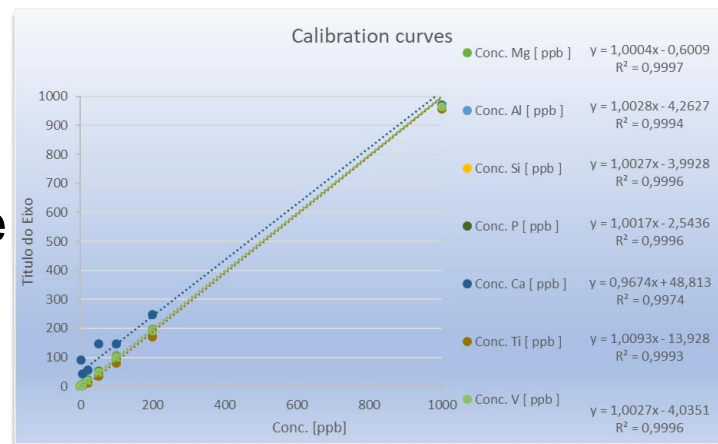
identification of 14 phenolic compounds: 3 phenolic alcohols, 4 phenolic acids, 1 phenolic aldehyde, 3 secoiridoids, and 3 flavones



# RESULTS SO FAR

## Mineral elements (W4)

- Optimization of sample preparation of olive oils:
  - Wet digestion in hot plate – results in uncomplete digested samples.
  - Micro-wave digestion – on going experiments
- Analytical methodology:
  - Ready to go
  - Calibration curves are done

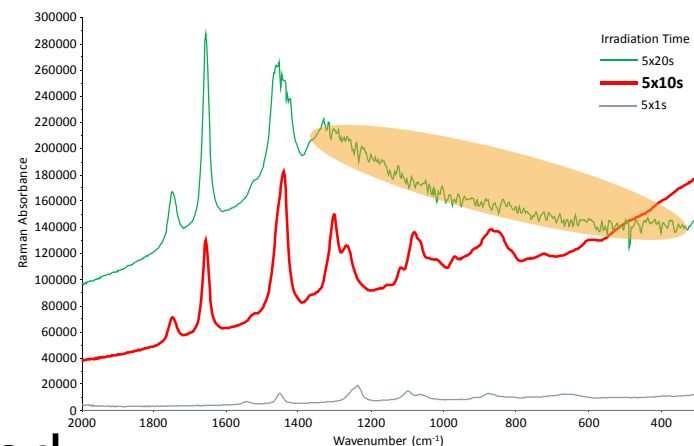




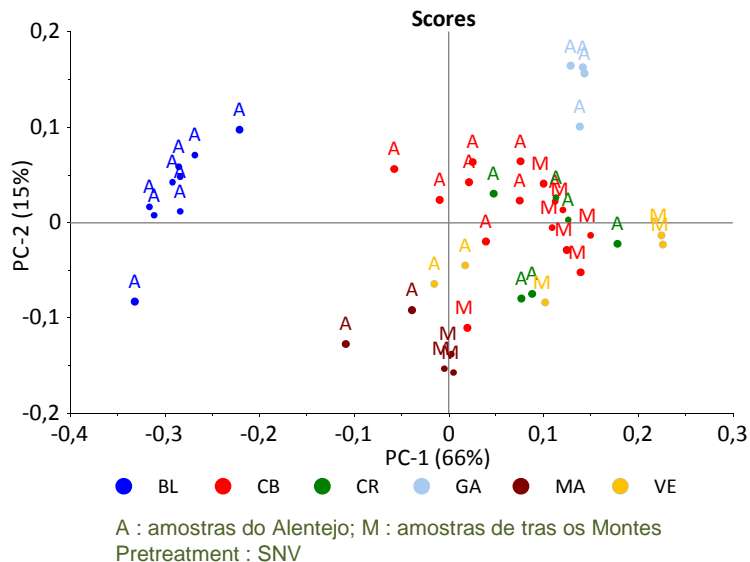
# RESULTS SO FAR

## Spectroscopic profiles (W5)

- Raman acquisition conditions optimised
- MIR and NIR data bases obtained



PCA on MIR Spectra of Portuguese samples



PLS1-DA predictions on MIR Spectra

Samples	% correct	% uncertain [0.4-0.6]	Pre-treatment	Parameters
French, Cultivars prediction (Cal = 62, Val = 31)	87.1%	9.7%	none	3 to 10 factors RMSEP = 0.20 to 0.39
	96.2%	2.2%	SG 1 <sup>st</sup> derivative (7 points, order 2)	2 to 10 factors, RMSEP = 0.16 à 0.27
Portuguese, Cultivars prediction (Cal = 29, Val = 15)	74.4%	15.6%	none	1 to 7 factors, RMSEP = 0.09 to 0.51
	85.6%	8.9%	SG 1 <sup>st</sup> derivative (7 points, order 2)	2 to 6 factors, RMSEP = 0.14 to 0.37
French + Portuguese, Cultivars prediction (Cal = 90, Val = 47)	91.5%	6.6%	none	7 to 10 factors RMSEP = 0.10 to 0.31
	96.3%	2.7%	SG 1 <sup>st</sup> derivative (7 points, order 2)	6 to 10 factors RMSEP = 0.10 to 0.29
Portuguese, Regions prediction (Cal = 16, Val = 9)	100.0%	0.0%	none	3 factors, RMSEP = 0.28

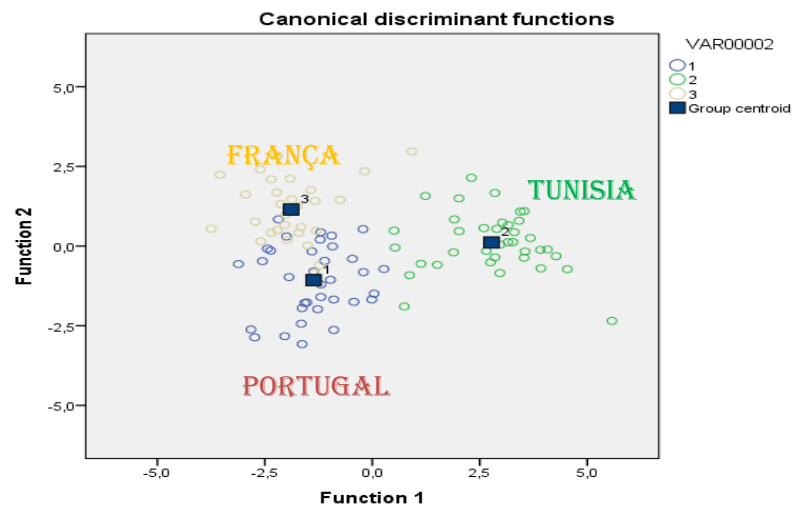
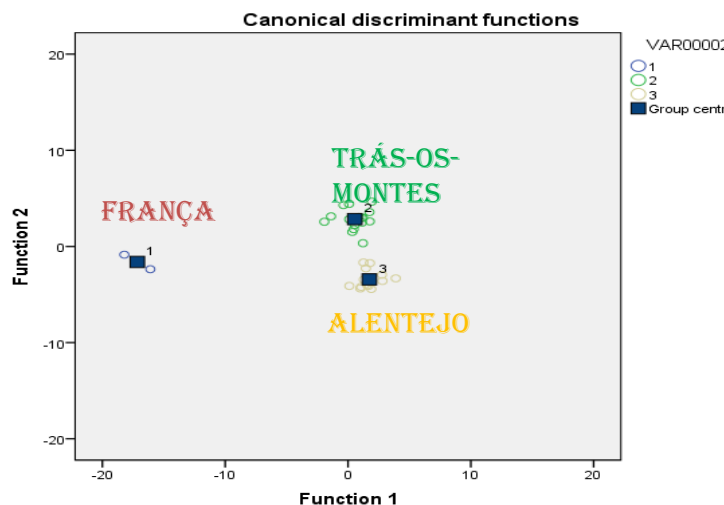
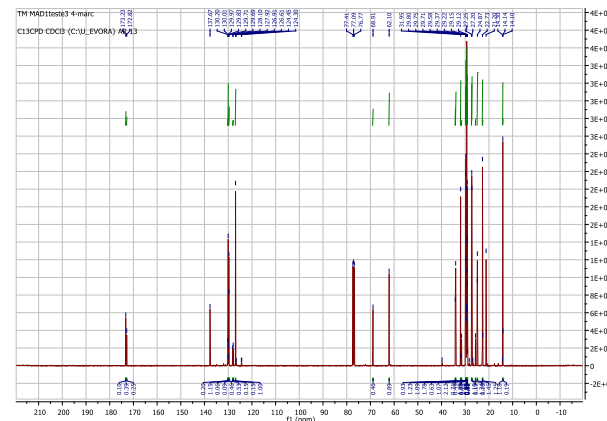
$$\% \text{ correct} = \frac{\text{true positive} + \text{true negative}}{\text{true positive} + \text{true negative} + \text{false positive} + \text{false negative} + \text{uncertain}}$$



# RESULTS SO FAR

## Spectroscopic profiles (W5)

- NMR acquisition conditions optimised
- NMR data base being built



# RESULTS SO FAR

## Stable isotopes (W6)

- Olive oil bulk analysis is being developed for oxygen isotopes
- Carbon isotopes almost complete for all samples. Data analysis is being processed.



# DISSEMINATION ACTIVITIES

**Project webpage** - available soon, under construction

## **Conferences and workshops:**

- Project presentation in “Science Day” at Évora University - Portugal
- Discrimination variétale des huiles d'olive vierges extra - France
- Authentication of Tunisian virgin olive oils using volatile compounds and chemometric analysis - Tunisia
- Evaluation of phenolic composition and antioxidant activity changes in olive flowers during development using HPLC/DAD and LC-MS/MS. - Tunisia

# TRAINING & MOBILITY

## Mobility

- Members of Tunisia Team have been in Portugal
  - PhD student Ons Rekik, GC/MS techniques
  - Post-Doc Boutheina Gargouri, GC/MS techniques

## Training

- Master, PhD and Post-Doc students:
  - Nuno Martins (post-Doc), Arona Pires (PhD student)
  - Boutheina Gargouri (Post-Doc)
  - Astrid MALECHAUX (PhD student)



# NEXT STEPS / PERSPECTIVES

**Olive oil sampling from 2017**

**Obtaining results from all tasks, data treatment,  
publication**

# PROJECT FUNDERS

**FCT** Fundação para a Ciência e a Tecnologia  
MINISTÉRIO DA CIÊNCIA, TECNOLOGIA E ENSINO SUPERIOR



République Tunisienne  
Ministère de l'Enseignement Supérieur,  
de la Recherche Scientifique





**Thank you for your attention!**

