



● Potential of Trapped Ion Mobility combined with LC-HRMS in Food Authenticity Studies

Identification and Characterization of Secoiridoids Isomers found in Greek Extra Virgin Olive Oil (EVOO)

The work described in this application note results from a productive collaboration between Prof. Nikolaos Thomaidis' group at the National and Kapodistrian University of Athens and Bruker.

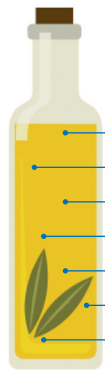
A special mention should go to Sofia Drakopoulou who undertook this study and was awarded 'Best Poster Award at RAFA 2019'. Olive oil has known health benefits and this study elucidates

the characterization and identification of stereochemical isomers associated with the health claim through the use of the powerful technique trapped ion mobility spectrometry.

Keywords:
olive oil authenticity,
characterization of
secoiridoids, trapped ion
mobility spectrometry,
timsTOF, PASEF

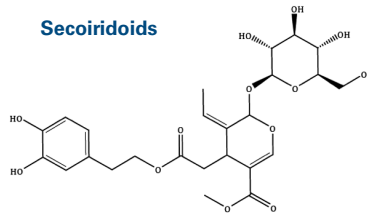
Introduction

Secoiridoids highly contribute to health claim enhancement.

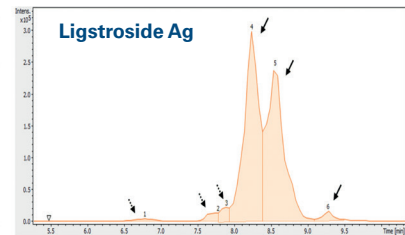
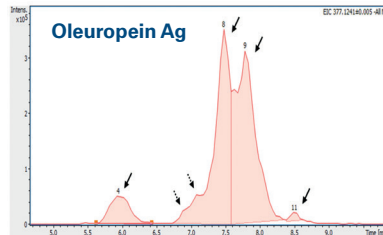


- Squalene
- Phytosterols
- Aliphatic fatty alcohols
- Triterpene alcohols
- Tocopherols
- Triterpene acids
- Phenolic Compounds (PCs)**

Secoiridoids



EVOOs, rich in PCs, (Hyd. and its derivatives) contribute to health protection (EU Reg. 432/2012).



Different isomeric peaks are detected

Scope

- Separation of isomeric compounds, crucial in olive oil health claim.
- Isomers identification.
- Discrimination of olive oil samples from different agricultural backgrounds (variety, geographical origin).

Samples

- 48 samples of Koroneiki variety from 3 different geographical origins in Greece (Peloponnese, Lesvos, Crete).
- 33 samples of 5 different Greek varieties: Koroneiki, Kolovi, Adramytiani (Lesvos), Chiotiki (Chios), Thrumba (Samos).



Materials and Methods

Sample treatment

Schematic workflow

0.5 g of olive oil

MeOH:H₂O 80:20% (v/v)

1 min Vortex

Centrifugation

Extraction

Filtration (0.22 μm)

UPLC-timsTOF-Pro
(Powered by PASEF)

Data treatment

MetaboScape 4.0

4D feature finder (T-ReX 4D)

Non-targeted data extraction

Data preprocessing (calibration, bucketing, filtering, scaling,...)

Creating bucket table

RT [min]	m/z meas.	M meas.	Mob. 1/X0	CCS (Å ²)	Include	Ions	MS/MS	Name	Molecular Formula	Annotations
8	6.49	285.11312	304.13096	0.781	164.0	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	Oleocanthal	C ₁₇ H ₃₂ O ₅	[Pub]
9	6.49	303.12046	304.12776	0.795	166.5	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	Oleocanthal	C ₁₇ H ₃₂ O ₅	[Pub]
10	6.48	303.12388	304.13115	1.195	250.1	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	Oleocanthal	C ₁₇ H ₃₂ O ₅	[Pub]
11	6.47	285.11293	304.13077	1.295	271.7	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	Oleocanthal	C ₁₇ H ₃₂ O ₅	[Pub]
12	6.48	285.11316	304.13100	1.195	250.8	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	Oleocanthal	C ₁₇ H ₃₂ O ₅	[Pub]
13	7.09	375.11097	376.11825	0.877	182.0	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	Dehydro oleuropein aglycone	C ₁₄ H ₂₀ O ₅	[Pub]
14	7.10	357.09663	376.11647	0.880	182.9	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>			
15	7.22	223.13373	224.14101	0.551	117.0	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	Methyl jasmonate	C ₁₁ H ₁₈ O ₂	[Pub]
16	7.21	205.12341	224.14125	0.728	155.3	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	Methyl jasmonate	C ₁₁ H ₁₈ O ₂	[Pub]
17	7.21	223.13389	224.14116	1.167	248.0	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	Methyl jasmonate	C ₁₁ H ₁₈ O ₂	[Pub]

UHPLC-timsTOF-MS Analysis

LC method

Bruker C18 column 2.1 × 100 mm, 1.8 μm

Mobile Phase and Flow Gradient

Temperature 40°C

timsTOF method

Ionization mode Negative & Positive

Scan Range of Mass 50-1000 m/z
1/k₀: 0.45-1.80 Vxs/cm²

ICC 5M

Ramp time 100 ms

TIMS Spectra rate 9.26 Hz

Accumulate

Trap

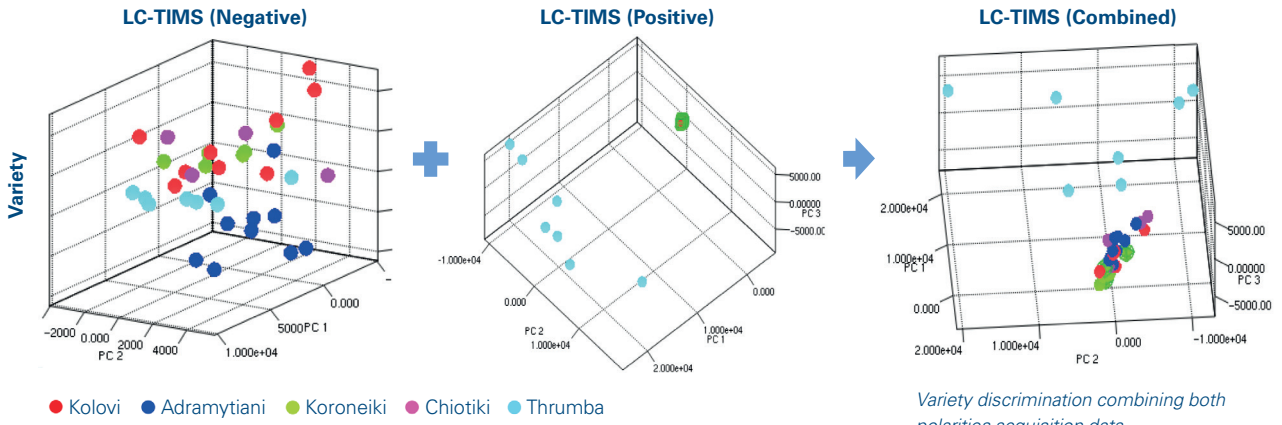
Elute

- Separation, based on the 3D size and charge of the compound
- Increased peak capacity by integrated separation dimension

Results and Discussion

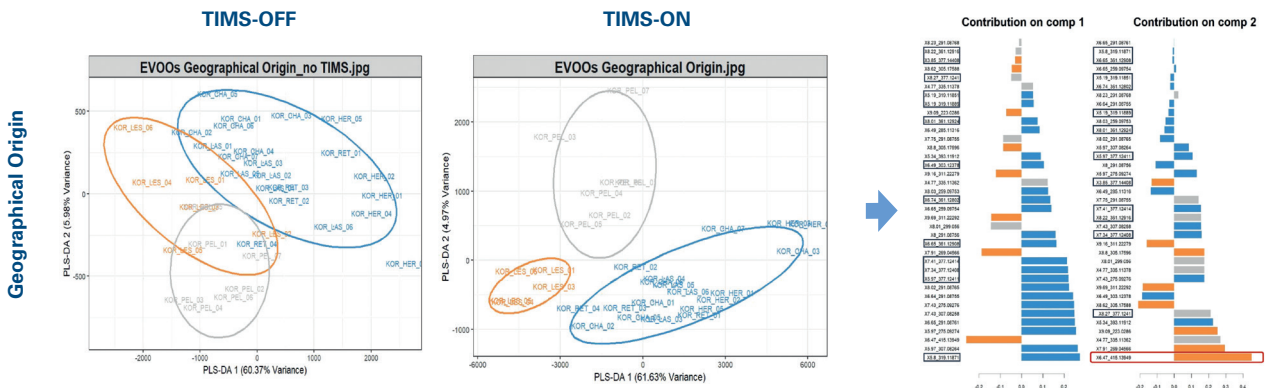
Discrimination - Untargeted Approach

PCA: Exploring Data Structure



Variety discrimination combining both polarities acquisition data

PLS-DA: Building discriminant models

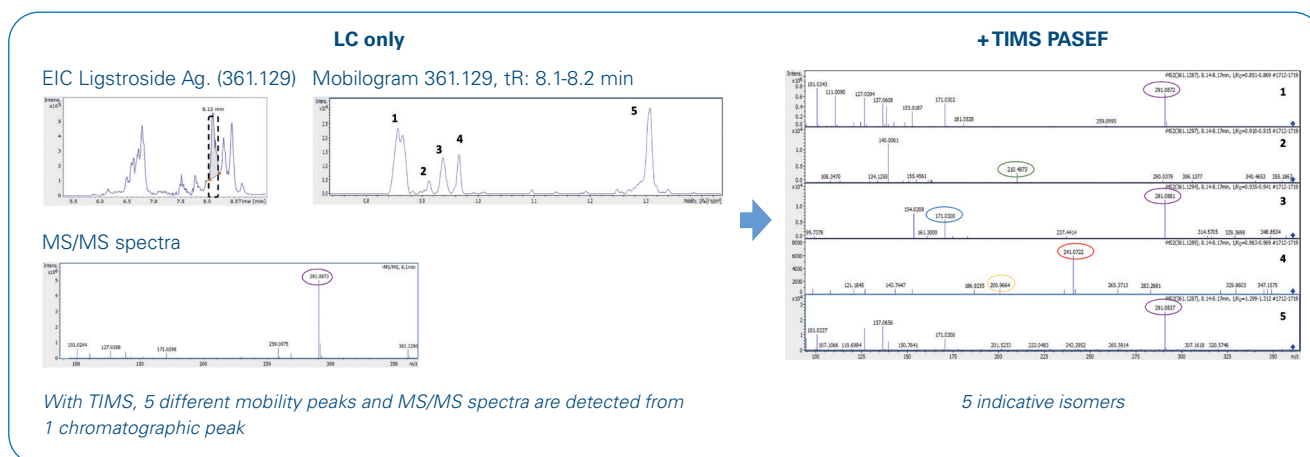


Using TIMS mobility dimension, geographical origin discrimination is clearly enhanced

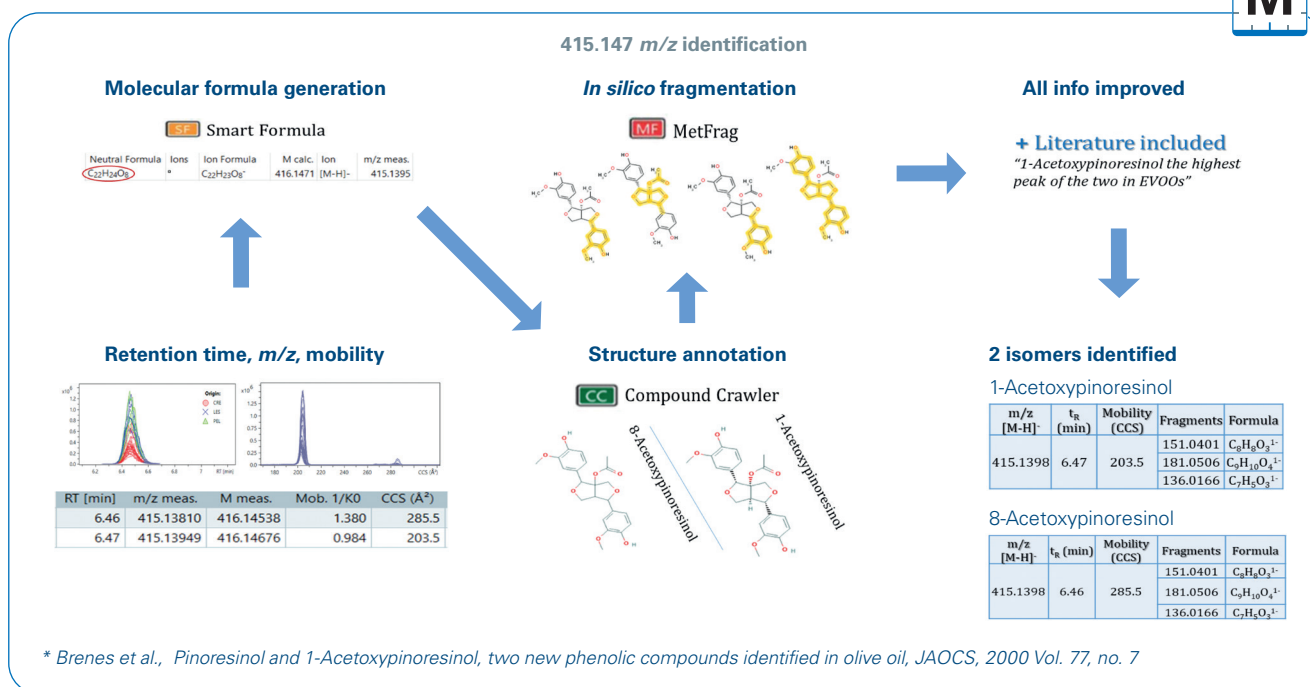
Isomers as potential authenticity markers

● Crete ● Lesvos ● Peloponnese

Separation of Isomeric Compounds



Isomers Identification



Acknowledgement

We acknowledge support for the olive oil sampling by the projects: "Emblematic Action, The Olive Road" and "Novel wide-scope research for the promotion of N. Aegean olive oil and olive products through the designation of their unique characteristics and bioactive content"

Conclusions

- The extra dimension of ion mobility enabled the separation of isomeric compounds
- Large amount of chemical information retrieved (t_R, m/z, CCS) using non-targeted data extraction
- Identification of unknown compounds was achieved, applying a structure elucidation-aimed workflow
- Application of chemometric tools (PCA, PLS-DA) for EVOOs variety and geographical origin discrimination



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