# **Development of method for multielement analysis of olive oil by ICPMS**

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### Introduction

Multielemental analysis of olive oil can be important tool in evaluation of its geographical traceability, as the elemental composition of soil is reflected in olives and in olive oil. However, whereas determination of elements in soil and plant is an easy task, it is not a case for olive oil, as only minor fraction of elements are transferred to the oil, and concentrations of trace elements are usually very low. An ideal instrumental technique to achieve this goal is inductively coupled plasma mass spectrometry technique (ICPMS), which enable multi-elemental analysis and very low detection limits.

In the present work microwave digestion and the recently proposed ultrasonic extraction method (Camin et al., 2010) for determination of elements in olive oil are compared.

# Material and Methods

**Preparation of olive oil:** Centrifugal oil extraction from olive fruits by Abencor oil mill (mc2, Ingenierias y Sistemas) within 24 h after the harvesting. Six Croatian olive oils were used in the experiments.

*Microwave digestion:* Digestion of ~0.5 g of olive oil by 6 ml HNO<sub>3</sub> (Trace select, Fluka) in Microwave oven Multiwave GO (Anton Paar) and dilution to 50 ml by Milli-Q<sup>®</sup> water.

*Ultrasonic extraction:* Extraction of ~15 g of olive oil with 15 ml of (1 % HNO<sub>3</sub>+0.2 % HCl) in the ulrasonic bath; centrifugation, isolation of water layer. *Measurements:* HR ICPMS (Element 2, Thermo), Measured elements (47): LR: <sup>7</sup>Li, <sup>9</sup>Be, <sup>85</sup>Rb, <sup>95</sup>Mo, <sup>111</sup>Cd, <sup>129</sup>Sn, <sup>133</sup>Cs, <sup>165</sup>Ho, <sup>205</sup>Tl, <sup>208</sup>Pb, <sup>209</sup>Bi, <sup>238</sup>U

### **Results and Discussion**

Digestion of oil samples in the Multivave GO microwave oven is fast and efficient, but limited to 0.5 g of olive oil due to risk of over-pressure. Recoveries for elements present in oil standard were satisfactory (Tab. 1). However, due to the very low concentrations of elements in analysed olive oils, and small quantity of samples, all measured elements, except K and Rb, were below detection limit.

The ultrasonic bath extraction, in which elements are extracted from 15 g of oil, had much lower detection limits, although the concentrations of elements in blank solutions for both methods were similar (Fig. 1). Nevertheless, ultrasonic extraction had lower recoveries for some elements than microwave digestion (Tab. 1), which may indicate their partial extraction from oil. Due to higher quantity of oil, 34 elements were detectable in analysed oil samples (Tab. 2).

Concentrations of K and Rb in olive oils obtained by the two techniques were comparable proving that ultrasonic bath extraction is efficient for these two elements.

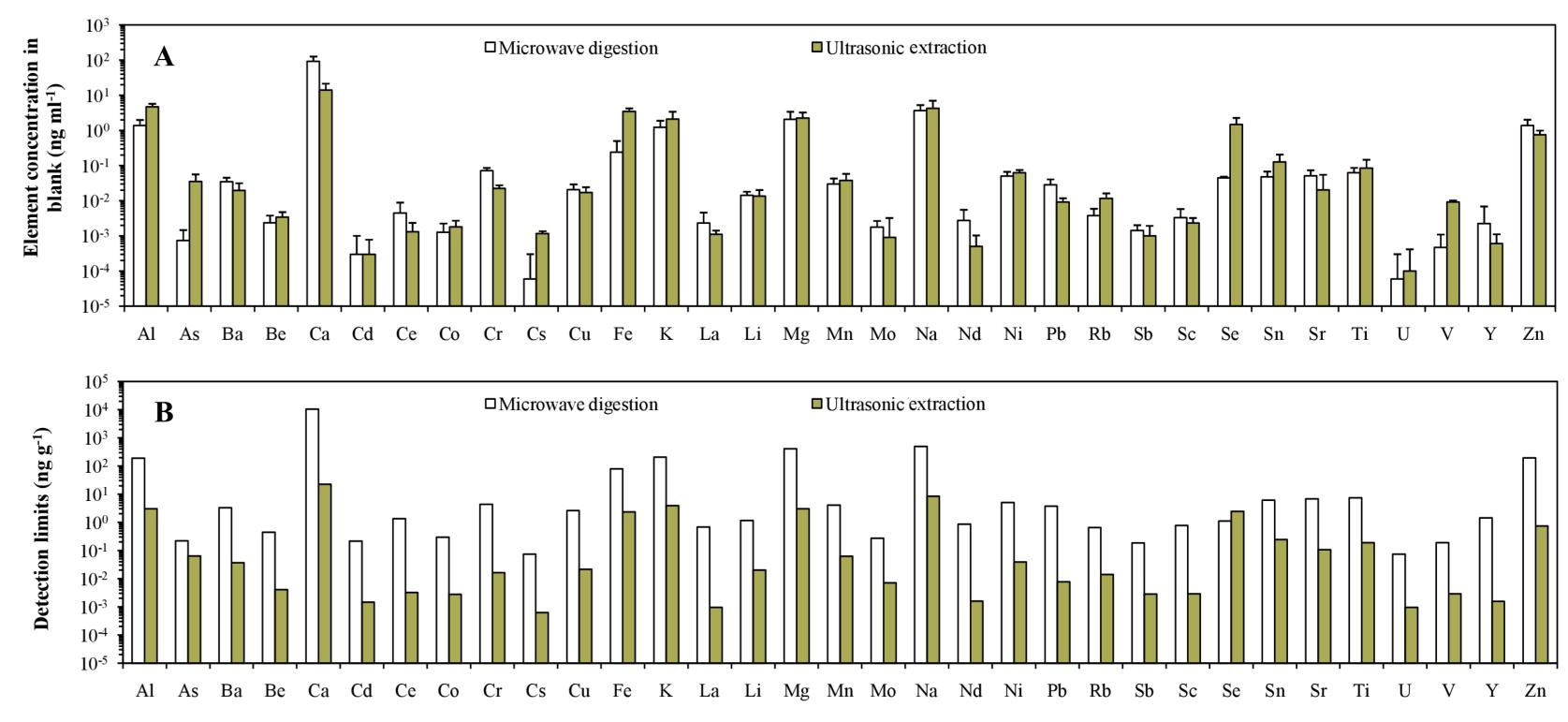
Tab. 2 Range of elements concentration in six olive oils analysed by ultrasonic extraction method.

**Tab. 1** Elements recoveries (%) for two methods at 2 ng  $g^{-1}$  spiked oil standard.

MR: <sup>23</sup>Na, <sup>24</sup>Mg, <sup>27</sup>Al, <sup>42</sup>Ca, <sup>45</sup>Sc, <sup>47</sup>Ti, <sup>51</sup>V, <sup>52</sup>Cr, <sup>55</sup>Mn, <sup>56</sup>Fe, <sup>59</sup>Co, <sup>60</sup>Ni, <sup>63</sup>Cu, <sup>66</sup>Zn, <sup>86</sup>Sr, <sup>89</sup>Y, <sup>90</sup>Zr, <sup>121</sup>Sb, <sup>138</sup>Ba, <sup>139</sup>La, <sup>140</sup>Ce, <sup>141</sup>Pr, <sup>145</sup>Nd, <sup>147</sup>Sm, <sup>151</sup>Eu, <sup>159</sup>Tb, <sup>163</sup>Dy, <sup>167</sup>Er, <sup>169</sup>Tm, <sup>171</sup>Yb, <sup>175</sup>Lu

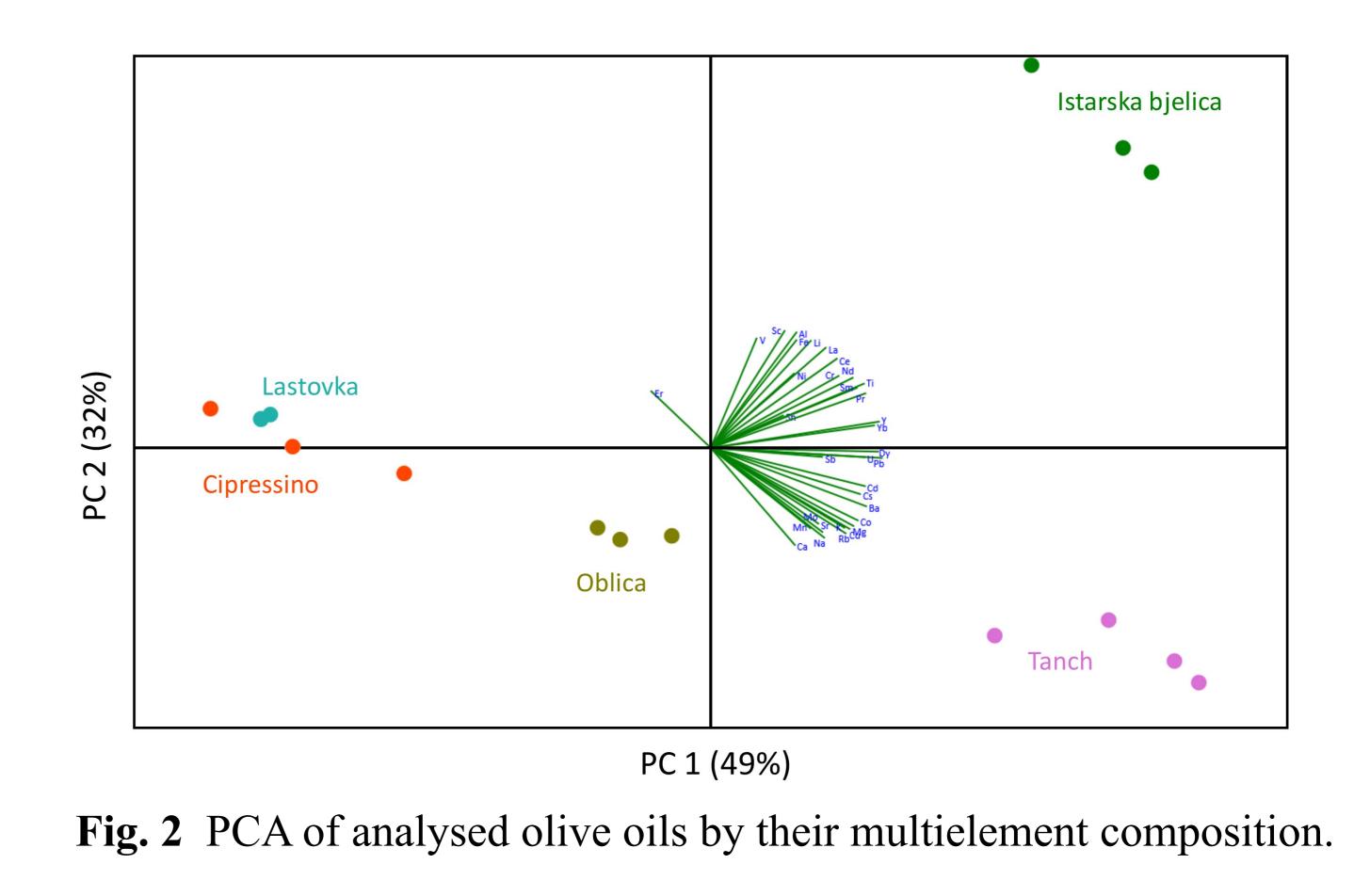
## HR: <sup>39</sup>K, <sup>75</sup>As, <sup>77</sup>Se, <sup>157</sup>Gd

Internal standard: <sup>115</sup>In; standard used for recovery experiments: Multi-element II mixture dissolved in oil (100 ppm), Merck, Certipur®



Pr $< 0.001$ $0.005$ Rb $< 0.014$ $8.47$ Sb $< 0.003$ $0.011$ Sc $< 0.003$ $0.011$ Sc $< 0.003$ $0.048$ Sm $< 0.001$ $0.005$ Sn $< 0.05$ $0.128$ Sr $0.02$ $0.505$ Ti $< 0.2$ $0.766$ U $< 0.001$ $0.002$ V $0.001$ $0.021$ This research was funded by UKE or							
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Pr $< 0.001$ $0.005$ Rb $< 0.014$ $8.47$ Sb $< 0.003$ $0.011$ Sc $< 0.003$ $0.011$ Sc $< 0.003$ $0.048$ Sm $< 0.001$ $0.005$ Sn $< 0.05$ $0.128$ Sr $0.02$ $0.505$ Ti $< 0.2$ $0.766$ U $< 0.001$ $0.002$ V $0.001$ $0.021$ This research was funded by UKE or	Ni	< 0.04	1.27	<ul> <li>oil due to much lower detection limits elements obtained recoveries are not s time ect.) is required.</li> <li>Nevertheless, the 34 elements four Component Analysis (Fig. 2) suggests</li> </ul>			
Rb $<0.001$ $0.005$ Rb $<0.014$ $8.47$ Sb $<0.003$ $0.011$ Sc $<0.003$ $0.011$ Sc $<0.003$ $0.048$ Sm $<0.001$ $0.005$ Sn $<0.05$ $0.128$ Sr $0.02$ $0.505$ Ti $<0.2$ $0.766$ U $<0.001$ $0.002$ V $0.001$ $0.022$ This research was funded by UKE or	Pb	0.095	0.527				
Red $(0.014$ $(0.47)$ Sb $<0.003$ $0.011$ Sc $<0.003$ $0.048$ Sm $<0.001$ $0.005$ Sn $<0.05$ $0.128$ Sr $0.02$ $0.505$ Ti $<0.2$ $0.766$ U $<0.001$ $0.002$ V $0.001$ $0.021$ This research was funded by LIKE graves	Pr	< 0.001	0.005				
So $(0.003)$ $(0.011)$ Sc $<0.003$ $0.048$ Sm $<0.001$ $0.005$ Sn $<0.05$ $0.128$ Sr $0.02$ $0.505$ Ti $<0.2$ $0.766$ U $<0.001$ $0.002$ V $0.001$ $0.221$ This research was funded by LIKE or	Rb	< 0.014	8.47				
Set $(0.003)$ $(0.016)$ traceability of their origin.Sm $<0.001$ $0.005$ traceability of their origin.Sn $<0.05$ $0.128$ ReferenceSr $0.02$ $0.505$ Camin et al. (2010) Food ChemistTi $<0.2$ $0.766$ AcknowledgmentsU $<0.001$ $0.021$ This research was funded by LIKE or	Sb	< 0.003	0.011				
Sin       <0.001       0.003         Sin       <0.05       0.128         Sr       0.02       0.505       Camin et al. (2010) Food Chemist         Ti       <0.2       0.766       Camin et al. (2010) Food Chemist         U       <0.001       0.002       Acknowledgments         V       0.001       0.221       This research was funded by LIKE or	Sc	< 0.003	0.048				
Sr       0.02       0.505       Camin et al. (2010) Food Chemist         Ti       <0.2	Sm	< 0.001	0.005				
Ti       <0.2	Sn	< 0.05	0.128	Refere	nce		
Ti       <0.2	Sr	0.02	0.505	Cam	in et al. (2010	)) Food Chemist	
V 0.001 0.221 This research was funded by UKE or	Ti	< 0.2	0.766				
V 0.001 0.221 This research was funded by UKE or	U	< 0.001	0.002	Ackno	wladamante	2	
V <0.001 0.009 This research was funded by UKF gr	V	0.001	0.221	This research was funded by UKF gra			
1 < 0.001 = 0.008	Y	< 0.001	0.008				
Yb <0.001 0.007 We would like to thank Anton Paar C	Yb	< 0.001	0.007	We wou	We would like to thank Anton Paar G		

**Fig. 1** Mean concentrations of elements in blank (n = 10, bars are SD)(A) and detection limits calculated as 3 SD of the blank (**B**) for the two methods.



ed by Camin et al. (2010) is a promising method for determination of elements in olive its compared to microwave digestion and good reproducibility. However, for some satisfactory and further research on the extraction conditions (acid mitxure, extraction

and in the six olive oils were sufficient to discriminate the samples through Principal sting that multielement composition of olive oils is different enough to be used for

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