

OPTIMIZATION AND VALIDATION OF CAPILLARY ELECTROPHORESIS METHOD FOR SMALL-ANIONS MEASUREMENT IN RED WINES

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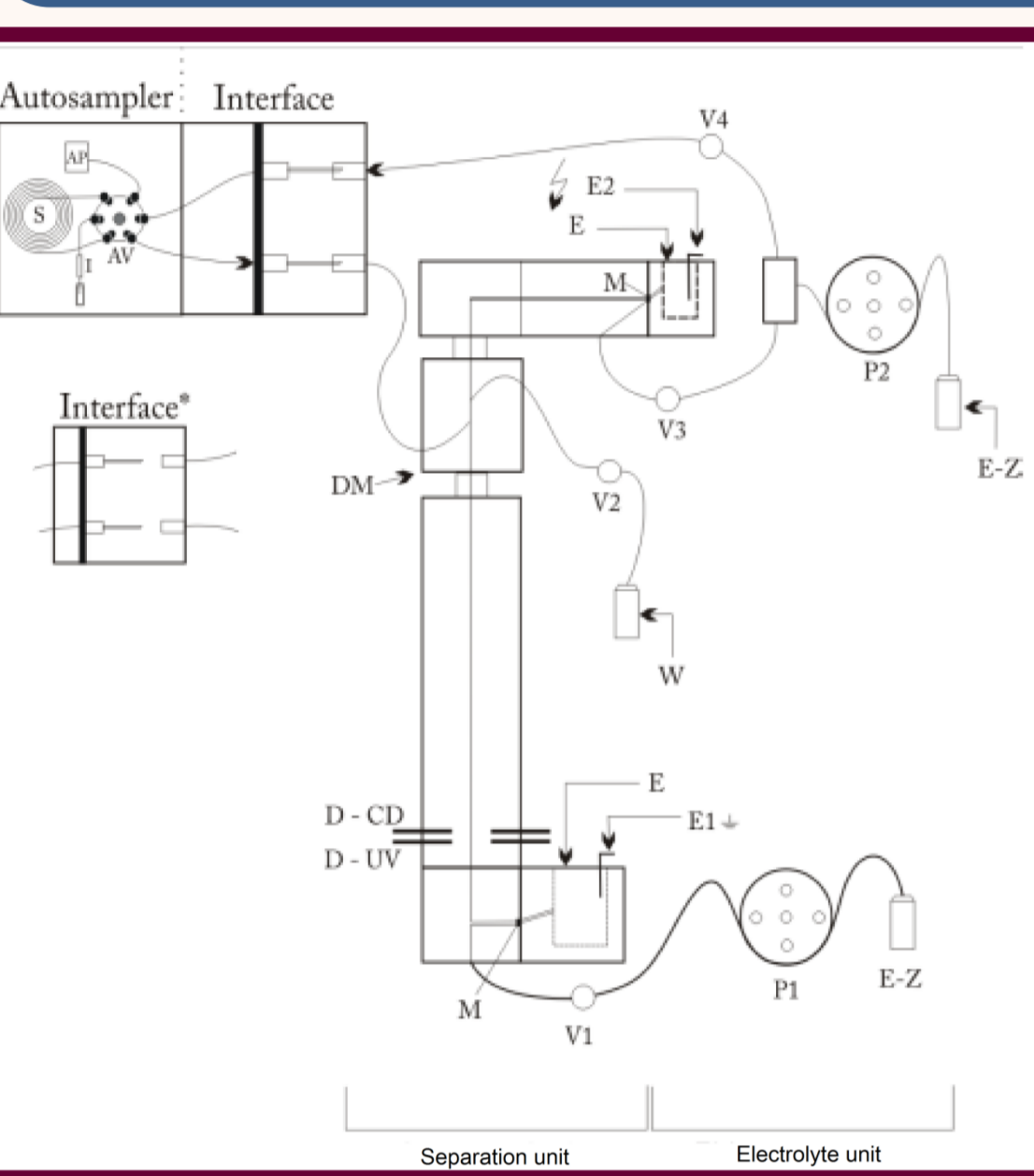
INTRODUCTION

Organic acids affect the chemical and microbiological stability, as well as sensorial characteristic of wine. α -hydroxy acids (tartaric, malic, lactic and citric acids) are the main organic acids in grapes and wine. The main purpose of this work was to determine the main organic acids in wine applying capillary electrophoresis. A capillary electrophoresis (CE) method has been developed and validated for analysis of organic acids (oxalate, tartrate, malate, malonate, pyruvate, succinate, acetate, citrate and lactate) and inorganic anions (sulfate and phosphate) in red wines.

MATERIALS AND METHODS

Capillary electrophoresis with UV-Vis/CD

Capillary electrophoresis conditions



Autosampler:
S - injection loop,
AP-sucking pump autosampler
I-needle aspiration specimens
AV - valve autosampler.
Electrolyte unit:
EZ - storage container with
supporting electrolytes,
P1-2-peristaltic pumps



Separation unit:
V1-V4 - shut-off solenoid valve,
E1-2-electrodes for a high voltage
source,
DM - dosing module
W - Waste,
D-UV - UV detector,
D-CD - contactless conductivity
detector
E - electrolyte container,
M - semi-permeable membrane,
*analytical capillary column of
fused silica (160 mm long)

Background electrolyte solutions

Parameter	ES1	ES2
Carrier ion	MES	MES
[mmol/L]	35	35
Counter-ion	Bis-tris	Bis-tris
[mmol/L]	3.4	3.4
Co-counter-ion	Bis-tris propane	Bis-tris propane
[mmol/L]	6	6
EOF suppressor	HEC	HEC
Concentration [%, m/v]	0.1	0.1
Complexing agent 1	α -cyclodextrin	α -cyclodextrin
[mmol/L]	-	20
Complexing agent 2	β -cyclodextrin	β -cyclodextrin
[mmol/L]	-	10
pH	6	5.95

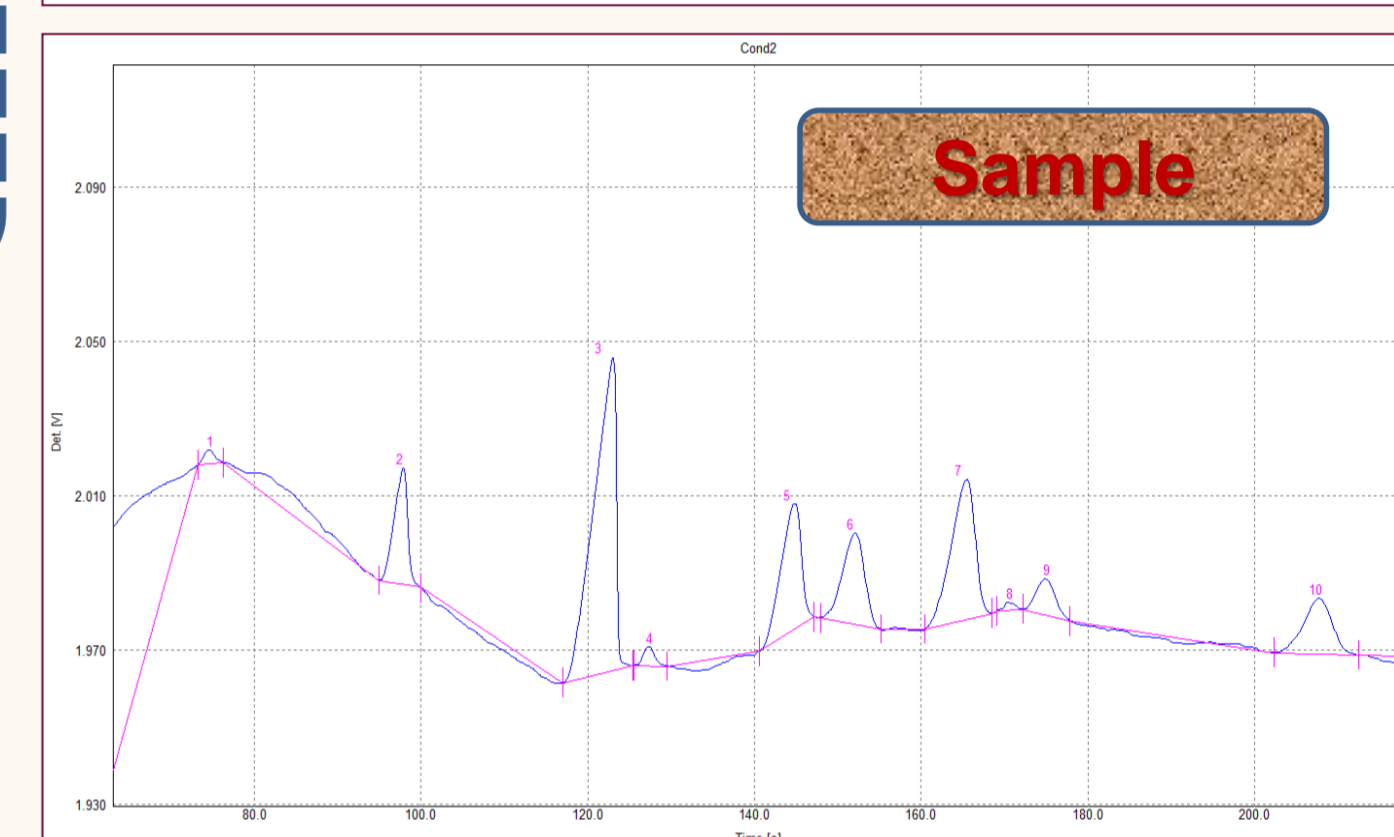
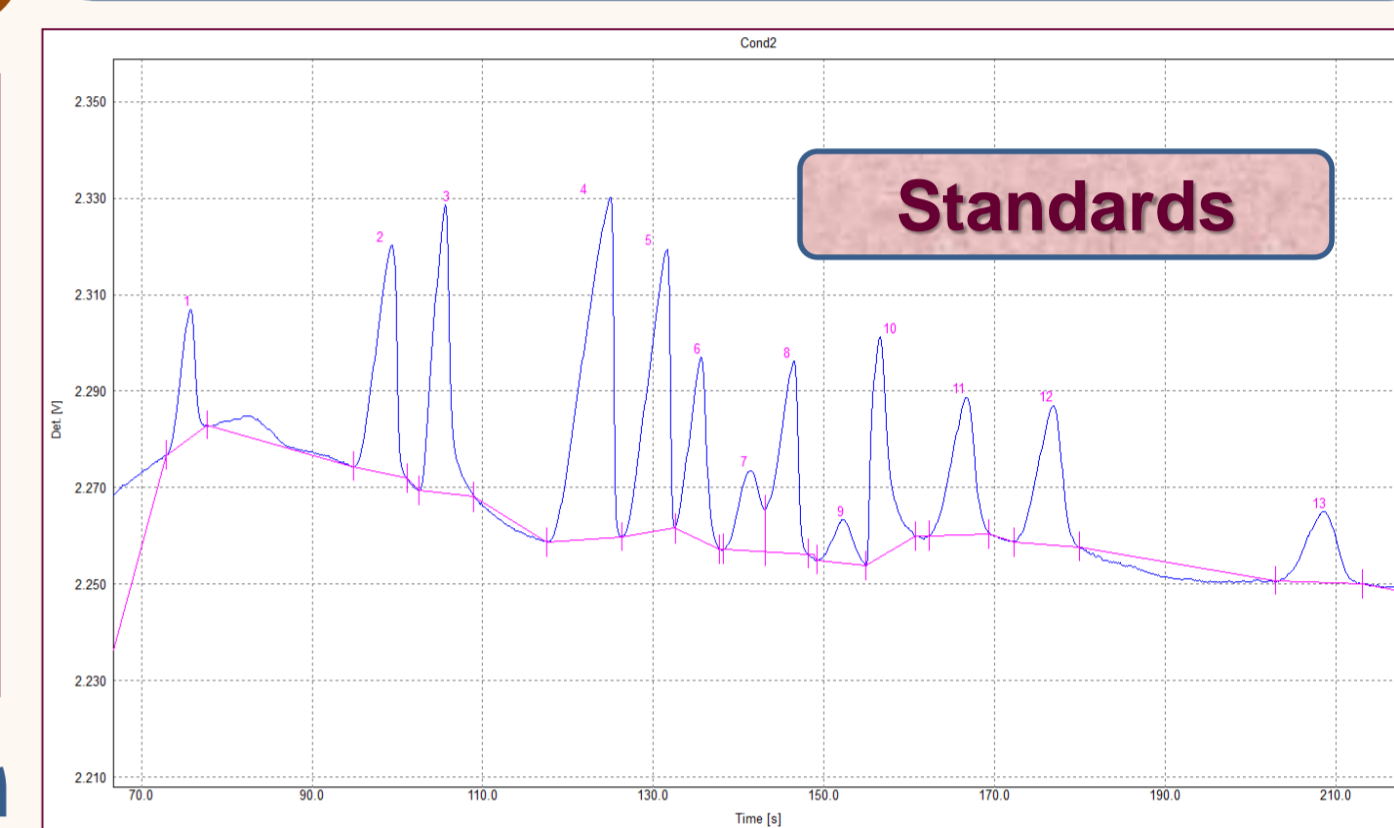
Wine sample preparation



15 μ l wine sample + 225 μ l IS+
150 μ l BGE+1110 μ l H₂O
=1500 μ l vial



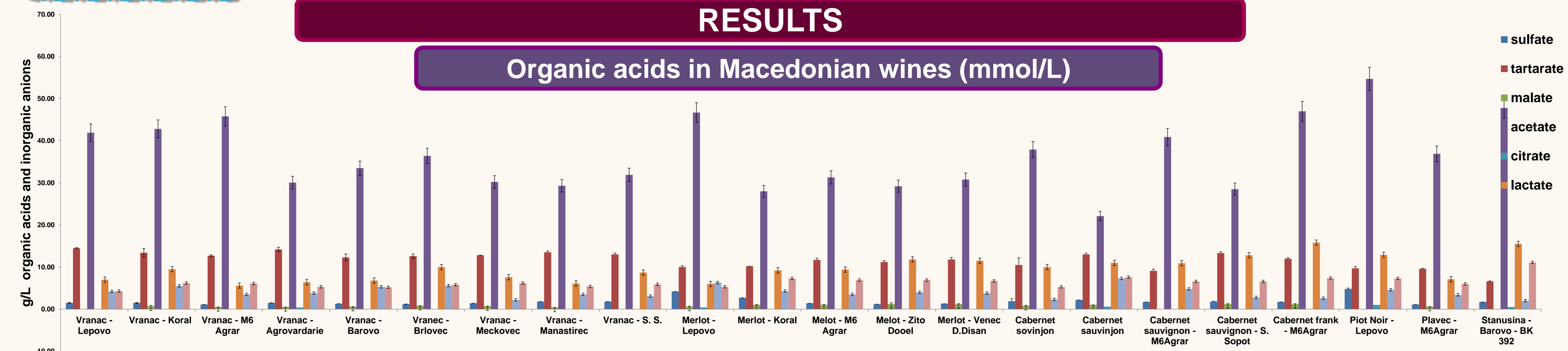
Electropherograms



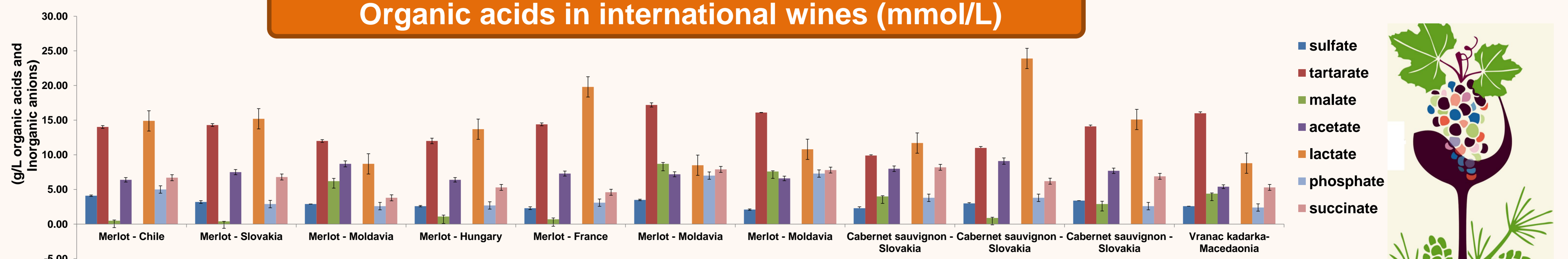
1-chloride, 2-sulfate, 3-oxalate,
4-tartrate, 5-malate, 6-malonate,
7-pyruvate, 8-succinate,
9-acetate, 10-citrate, 11-lactate,
12-phosphate, 13-NAS

RESULTS

Organic acids in Macedonian wines (mmol/L)



Organic acids in international wines (mmol/L)



Conclusion

Fact, accurate and simple method was optimized and developed for determination of organic acids in red wines applying capillary electrophoresis. In total, 33 red wines were analysed with the proposed method, including 22 Macedonian red wines produced in Tikves winery from various varieties and geographic areas as well as 11 commercial red wines from different countries. The main organic acid was the tartaric acid, present in the highest content in all wines, regardless the variety. Also, lactic acid was present in high concentration which means that malolactic fermentation was performed in most of the analyzed wines. For the first time, phosphate and sulfate were determined in Macedonian wines.

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