

METHOD FOR ANALYSIS OF ELEMENTS IN WINE USING ICP-MS

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Introduction

Red wine is a very complex mixture of ethanol and different organic compounds such as carbohydrates, organic acids, volatiles and bioactive compounds (anthocyanins, monomeric and polymeric flavan-3-ols, flavonols and phenolic acids). Therefore, sample pre-treatments are necessary for its multi-element analysis. Decomposition of organic matrix could be performed by wet digestion on a hot plate or in a microwave oven using concentrated HNO₃, HClO₄ and H₂SO₄ or mixtures of these acids.

In our study, the aim of the work was to develop, optimize and validate a microwave digestion method for wine sample pre-treatment, followed by ICP-MS determination of the elements.

Materials and Methods

Wine samples

Vranec wines from Tikveš region, R. Macedonia

Microwave digestion method

5 mL of wine was digested with 5 mL nitric acid (69.0%, w/w) and samples were made up to a final volume of 25 mL with ultrapure deionised water. Microwave digestion system, Model MARS, CEM Corporation, USA) [1].

ICP-MS analysis

ICP-MS, model 7500cx, Agilent technologies The concentration of 41 isotopes was measured in No-gas and Helium mode. The quality assurance of the method was approved with the standard addition method [1].

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Results

- Satisfactory linearity in all cases with correlation coefficients were obtained (R²>0.99) (Table 1).
Method is accurate and convenient for quantitative analysis of elements in red and white wines. Satisfactory results for the recovery ranged between 83-120% (Table 2 and Table 3).
Satisfactory inter- and intra- day reproducibility. RSD values ranged from 0.32% to 15.1% for red wines and from 0.19% to 8.73% for white wine.

Table 1. Linear regression data

Table with 7 columns: Element, Isotope, Unit, Slope, Intercept, R^2, LOD, LOQ. Lists various elements like Ag, Al, As, Ba, Be, Bi, Cd, Ce, Cr, Cu, Fe, Ga, Ge, In, Li, Mg, Mn, Mo, Ni, Pb, P, Sb, Se, Sn, Sr, Te, Ti, V, Zn.

Table 2. Standard additions for checking accuracy of the digestion procedure

Table with 6 columns: Element, Unit, Control, R(%), Mean, RSD(%). Shows recovery percentages for various elements.

Table 3. Repeatability data (5 measurements per day with 3 injections per measurement)

Table with 4 columns: Element, Unit, Mean concentration, RSD(%). Compares red wine and white wine measurements.

Table 4. Reproducibility for the analyzed elements in red and white wine (3 replicates x 3 injections x 3 days)

Table with 6 columns: Element, Unit, Day 1, Day 2, Day 3. Shows reproducibility data across multiple days.

Conclusion

Fast and accurate method for sample preparation followed with ICP-MS for multi-element analysis of wine was optimized and developed. The method presented satisfactory linearity, LOD, LOQ, accuracy, repeatability and reproducibility for total 38 elements (Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cs, Cu, Fe, Ga, Ge, In, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Pd, Rb, S, Sb, Se, Si, Sn, Sr, Te, Ti, Tl, V, Zn).

References

[1] Ivanova-Petropulos V., Balabanova B., Mitrev S., Nedelkovic D., Dimovska V., Gulaboski R. (2015). Optimization and validation of a microwave digestion method for multi-element characterization of Vranec wines. Food Analytical Methods, in press, doi:10.1007/s12161-015-0173-z.