



## Wine and must analysis with ICP-OES

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**There is an increasing demand for quality control on wines worldwide, and consequently more and more needs for elemental analysis.**

**Thanks to its multi-element analysis capability, its high sensitivity and its ease-of-use, the ICP-OES technique is well adapted for wine elemental characterization.**

**From this characterization, the origin of wines can be identified as well as the technological processes applied during manufacturing. The identification of geographical origin as well as the processes used provides increased guarantee in the quality of the final product.**

**In this study the Ultima 2 was used to analyze in white, red, rosé wines and must samples.**

### Sample preparation

To facilitate the analysis, it was decided to use a single calibration curve for white, red and rosé wine. Thus, the wine samples were diluted twice in water prior analysis to minimize matrix effects due to variations in alcohol content and in organic compounds. To improve accuracy, all standard solutions were prepared in 6% v/v Ethanol, using Ethanol 95% Normapur from VWR and Spex CertiPrep single element standard solutions.

The must samples were analyzed with a two times dilution using external calibration in water. The standard solutions were prepared using Spex CertiPrep single element standard solutions.

### Operating conditions

The characteristics of the ULTIMA 2 High Resolution spectrometer used for this study are given in Table 1.

Table 1. Characteristics of ULTIMA 2 ICP-OES Spectrometer

|                  |   |
|------------------|---|
| Optical mounting | Czerny-Turner                                       |
| Focal length     | 1 meter   |
| Grating          | 2400 g/mm   |
| Resolution       | 5 pm for 120 - 320 nm<br>10 pm for 320 - 800 nm     |
| Thermoregulation | 32 ± 0.1°C  |
| RF Generator     | 40.68 MHz solid state, water cooled                 |
| Torch            | Vertical with Radial Viewing and Total plasma View* |

\* Total Plasma View: Measurement of the whole Normal Analytical Zone for enhanced sensitivity and reduced matrix effects

A parallel flow nebulizer and a double pass cyclonic spray chamber were used to ensure sensitivity and stability with wine and must matrix. All details of the introduction system are given in Table 2.



Table 2. Specification of the sample introduction system

|                              |   |
|------------------------------|---|
| Nebulizer                    | Parallel flow   |
| Spray chamber                | Glass cyclonic double pass  |
| Sample uptake                | 1 mL/min  |
| Injector tube inner diameter | 3 mm  |
| Pump tubing                  | Black-black pump tubing for sample<br>Grey-grey pump tubing for drain |

All plasma parameters were optimized for sensitivity and robustness and are given in Table 3.

Table 3. Operating conditions

|                |           |
|----------------|-----------|
| Power          | 1200 W    |
| Plasma gas     | 12 L/min  |
| Auxiliary gas  | 0 L/min   |
| Sheath gas     | 0.2 L/min |
| Nebulizer flow | 0.8 L/min |
| Pump speed     | 15 rpm    |

Acquisition was done using max mode with 4s integration time and 2s integration time for background correction. All calibrations and analyses steps were ran with 3 replicates.

## Results

Lines used for analysis of both wines and must are given in Table 4.

Table 4: List of lines used for the analysis

| Element | Wavelength (nm) | Element | Wavelength (nm) |
|---------|-----------------|---------|-----------------|
| Ca      | 422.673         | Li      | 670.784         |
| Cd      | 214.438         | Mg      | 279.079         |
| Cu      | 327.754         | Na      | 588.995         |
| Fe      | 240.488         | Pb      | 220.353         |
| K       | 404.721         | Zn      | 213.856         |

All results were compared to those obtained in a reference laboratory. All concentrations are given with two significant figures and for each value; the bias is calculated with respect to the value of the reference laboratory.

All results are given in Table 5 to 8 below and are corrected from the dilution factor.

Table 5 to 8: Results obtained on wine samples and comparison with reference laboratory

|           | White wine     |                      |          |
|-----------|----------------|----------------------|----------|
|           | Conc. Measured | Reference laboratory | Bias (%) |
| Ca (mg/L) | 69             | 75                   | 8        |
| Cd (µg/L) | < 0.6*         | 0.18                 |          |
| Cu (mg/L) | 0.34           | 0.35                 | 3        |
| Fe (mg/L) | 3              | 2.9                  | 3        |
| K (mg/L)  | 657            | 670                  | 2        |
| Li (µg/L) | 13             |                      |          |
| Mg (mg/L) | 72             | 70                   | 3        |
| Na (mg/L) | 23             | 25                   | 8        |
| Pb (µg/L) | < 6            | 11                   |          |
| Zn (mg/L) | 0.51           | 0.53                 | 4        |

\*< LOQ – lower than quantification limit.

|           | Rosé wine      |                      |          |
|-----------|----------------|----------------------|----------|
|           | Conc. Measured | Reference laboratory | Bias (%) |
| Ca (mg/L) | 73             | 80                   | 9        |
| Cd (µg/L) | < 0.6*         | 0.17                 |          |
| Cu (mg/L) | 0.32           | 0.32                 | 0        |
| Fe (mg/L) | 2.1            | 2.2                  | 4        |
| K (mg/L)  | 759            | 739                  | 3        |
| Li (µg/L) | 18             |                      |          |
| Mg (mg/L) | 88             | 85                   | 4        |
| Na (mg/L) | 29             | 33                   | 12       |
| Pb (µg/L) | 12             | 13                   | 8        |
| Zn (mg/L) | 0.77           | 0.79                 | 2        |

\*< LOQ – lower than quantification limit.

|           | Red wine       |                      |          |
|-----------|----------------|----------------------|----------|
|           | Conc. Measured | Reference laboratory | Bias (%) |
| Ca (mg/L) | 72             | 78                   | 8        |
| Cd (µg/L) | < 0.6*         | 0.18                 |          |
| Cu (mg/L) | 0.23           | 0.25                 | 8        |
| Fe (mg/L) | 2.5            | 2.5                  | 0        |
| K (mg/L)  | 1056           | 1135                 | 7        |
| Li (µg/L) | 18             |                      |          |
| Mg (mg/L) | 103            | 106                  | 3        |
| Na (mg/L) | 19             | 20                   | 5        |
| Pb (µg/L) | 12             | 11                   | 9        |
| Zn (mg/L) | 0.74           | 0.69                 | 7        |

\*< LOQ – lower than quantification limit.

|           | Must sample    |                      |          |
|-----------|----------------|----------------------|----------|
|           | Conc. Measured | Reference laboratory | Bias (%) |
| Ca (mg/L) | 177            | 187                  | 5        |
| Cd (µg/L) | < 1*           | 0.77                 |          |
| Cu (mg/L) | 1.7            | 1.8                  | 6        |
| Fe (mg/L) | 24             | 25                   | 3        |
| K (mg/L)  | 1473           | 1591                 | 7        |
| Li (µg/L) | 22             |                      |          |
| Mg (mg/L) | 95             | 87                   | 9        |
| Na (mg/L) | 31             | 33                   | 7        |
| Pb (µg/L) | 163            | 154                  | 6        |
| Zn (mg/L) | 2.2            | 2.4                  | 8        |

\*< LOQ – lower than quantification limit.

## Conclusion

Thanks to reduced matrix effects of the ULTIMA 2, a good agreement between concentrations measured and concentrations from the reference laboratory is obtained with the use of a single calibration curve for all wines.

This is mainly due to the 3 mm i.d. injector that reduces matrix effects and increases the residence time of the sample in the plasma, thus enhances sensitivity.

The use of a single calibration curve increases throughput and facilitates analysis for the user when the robustness of the plasma facilitates the method optimization by allowing the use of standard plasma conditions.

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