Application Note

Wine analysis with the ACTIVA-M ICP-AES

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1 Introduction

There is an increasing diversity of wines on the worldwide market, leading to an increasing demand for elemental analysis. These analyses are performed for several purposes. One of these is to guarantee the quality of the wines by determining the amount of several elements that can indicate the origin of the wine and also influence parameters such as color. Another purpose is the control of heavy metals such as As, Cd, Pb, Se and Hg that is necessary to avoid consumer exposure to these toxic elements.
ICP-AES is well adapted to the analysis of wines due to its ability to determine many elements at low concentrations as well as major elements. The determination of trace heavy metals as well as major elements is possible in the same determination.
In this study the ACTIVA-M was used to determine several elements in both red and white wines, Cahors and Sauvignon, respectively.

2 Sample preparation

Due to the very robust torch of the ACTIVA-M, no sample preparation is required and the analysis was performed on the undiluted sample. To obtain accurate results, all standard solutions were prepared in 12% v/v Ethanol, using Ethanol 95% Normapur from VWR and Spex CertiPrep single element standard solutions. The calibration range for each element analyzed is given in Table 1.

Table 1. Concentration range

Elements	Low value (mg/L)	High value (mg/L)
Cu, Pb	0.25	1
Mn, Zn	1.25	5
Fe	2.5	10
Na	5	20
Mg	25	100
Ca	50	150
K	250	1000

3 Operating conditions

All characteristics of the ACTIVA-M used for this study are given in Table 2.

Table 2. Specifications of the ACTIVA-M ICP spectrometer

Parameter	Specification
Optical mounting	Czerny Turner
Focal length	0.64 m
Gratings (number of grooves/mm)	Dual back-to-back gratings 4343 g/mm 2400 g/mm
Resolution	< 10 pm 120-430 nm < 18 pm 430-800 nm
Thermoregulation	32 ± 0.1°C
Type of generator	40.68 MHz Solid state, water cooled
Torch	Vertical Radial viewing with Total Plasma View*

*Total Plasma View: Measurement of the whole Normal Analytical Zone of the plasma for enhanced sensitivity and freedom from interferences.

The ACTIVA-M is equipped with a unique fully demountable torch, 3 mm i.d. alumina injector and the HORIBA Jobin Yvon patented sheath gas. This configuration provides getting enhanced sensitivity along with reduced matrix and memory effects.
A concentric nebulizer and a double-pass cyclonic spray chamber were used to ensure sensitivity and stability with the undiluted wine matrix. All details on the introduction system are given in Table 3.

Table 3. Specifications of the sample introduction system

Parameter	Specification
Nebulizer	Concentric glass nebulizer
Spray chamber	Double-pass glass cyclonic
Sample uptake	1 mL/min
Pump tubing	Black – Black (sample) Grey – Grey (drain)

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All plasma parameters were optimized for sensitivity and robustness and are given in Table 4.

Table 4. Operating conditions

Parameter	Specification
Power	1200 W
Plasma gas	14 L/min
Auxiliary gas	0 L/min
Sheath gas	0.2 L/min
Nebulizer flow	0.80 L/min (3 bars)
Pump speed	15 rpm

The integration time used was 5 seconds for wavelengths in the 200 - 300 nm range and 3 seconds for wavelengths above 300 nm. Three replicates were used for each determination.

The whole sequence was run using the AS-500 autosampler for fully automated analysis.

4 Results

4.1 Detection limits

Detection limits were estimated using a 12% ethanol sample This sample was analyzed 5 times using 10 replicates for each determination. The detection limit was estimated using the following definition:

DL = 3 x sB, where DL is the detection limit in µg/L and s_B is the standard deviation, in µg/L of the 10 measurements of the blank.

To take full benefit of the CCD detection, many lines were used for several elements during the analysis. Detection limits are given on the basis of the most sensitive line and are compared to detection limits achieved in water for the same wavelengths (Table 5).

Table 5. Detection limits in 12% ethanol and in water

Element/Line	Detection limit in 12% Ethanol µg/L	Detection limit in Water µg/L
Cu 324.754	0.4	0.3
Fe 239.563	0.5	0.5
Mn 257.610	0.04	0.06
Pb 283.305	10	11
Zn 213.857	0.4	0.2

The detection limits obtained are statistically identical to those obtained in water using the same integration time, proving the sensitivity of the system and its high robustness.

4.2 Analysis

Analysis was performed on both the Cahors and the Sauvignon "samples" using the same calibration. A spike of 1 mg/L on low concentration elements was done to check for the ability to obtain good recoveries in such samples using a single calibration curve.

Results of the analysis of the unspiked and spiked samples are given in Tables 6 and 7 for Cahors and Sauvignon, respectively. All concentrations are given with 2 significant figures and recoveries were calculated with unrounded data.

Table 6. Results and recoveries on Cahors red wine

Element/Line	Cahors Conc mg/L	Cahors RSD	Spiked Cahors Conc mg/L	Spiked Cahors RSD	Recovery %
Ca 315.887	65	0.2			
Ca 317.933	64	0.5			
Ca 318.128	64	0.6			
Cu 324.754	0.074	0.3	1.1	0.6	102
Cu 327.395	0.071	0.6	1.1	0.6	103
Fe 236.491	4.6	0.3	5.6	0.2	100
Fe 239.563	4.6	0.2	5.5	0.5	99
Fe 239.924	4.7	0.2	5.5	0.4	98
Fe 240.489	4.5	0.3	5.6	0.3	101
Fe 240.666	4.6	0.2	5.7	0.3	102
K 404.721	564	0.4			
Mg 279.078	69	0.2			
Mg 279.800	69	0.2			
Mn 257.610	1.0	0.3	2.0	0.2	99
Mn 259.373	1.0	0.3	2.0	0.2	99
Mn 260.575	1.0	0.3	2.0	0.2	99
Na 589.592	12	0.2			
Pb 283.305	< LOD		1.0	0.8	102
Zn 206.200	0.98	0.1	2.0	0.3	101
Zn 213.857	0.98	0.4	2.0	0.3	103

< LOD = Less than the detection limit

Table 7. Results and recoveries on Sauvignon white wine

Element/ Line	Sauvignon Conc mg/L	RSD	Spiked Sauvignon Conc mg/L	RSD	Recovery %
Ca 315.887	82	0.4			
Ca 317.933	81	0.3			
Ca 318.128	81	0.4			
Cu 324.754	< LOD		1.0	0.34	103
Cu 327.395	< LOD		1.0	0.35	99
Fe 236.491	1.6	0.5	2.7	0.24	103
Fe 239.563	1.7	0.3	2.7	0.28	104
Fe 239.924	1.6	0.4	2.7	0.25	104
Fe 240.489	1.6	0.4	2.6	0.30	100
Fe 240.666	1.6	0.4	2.7	0.24	103
K 404.721	565	0.6			
Mg 279.078	50	0.05			
Mg 279.800	50	0.03			
Mn 257.610	1.1	0.5	2.1	0.30	99
Mn 259.373	1.1	0.5	2.1	0.29	99
Mn 260.575	1.1	0.5	2.1	0.32	100
Na 589.592	23	0.4			
Pb 283.305	< LOD		1.0	1.10	100
Zn 206.200	0.70	0.4	1.7	0.40	101
Zn 213.857	0.71	0.2	1.7	0.50	100

The results for an element according to all the lines used are very consistent. The multi-line analysis used here improves confidence in the final result. It allows a direct validation of the final result by highlighting any potential interference when results vary for different lines of an element. The reliability of the method is confirmed by the excellent recoveries obtained for all the elements determined, which within statistical variation is essentially 100%.

4.3 Stability

The stability was evaluated by performing 3 determinations on both the Cahors and the Sauvignon with a 1 hour delay between each determination. Results are given in Table 8 for the Cahors and in Table 9 for the Sauvignon. Stability is given on a single line for each element as all lines exhibited the same stability.

Table 8. Stability test on the Cahors red wine

Element Wavelength (nm)	Conc (mg/L)	Conc after 1 hour	Conc after 2 hours	RSD %
Ca 317.933	63.3	63.4	61.9	1.2
Cu 327.395	0.071	0.071	0.069	1.4
Fe 239.563	4.5	4.6	4.6	1.3
K 404.721	554	557	549	0.8
Mg 279.800	68.1	67.9	67.0	1.3
Mn 260.575	1.02	1.04	1.02	0.7
Na 589.592	12.2	12.2	12.1	0.6
Zn 206.200	0.98	1.0	1.0	1.6

Table 9. Stability test on the Sauvignon white wine

Element Wavelength (nm)	Conc (mg/L)	Conc after 1 hour	Conc after 2 hours	RSD %
Ca 317.933	81.1	80.9	79.9	0.6
Fe 239.563	1.6	1.6	1.6	1.2
K 404.721	558	563	555	0.7
Mg 279.800	49.2	49.1	47.8	1.7
Mn 260.575	1.09	1.08	1.07	0.8
Na 589.592	22.4	22.4	22.3	0.7
Zn 206.200	0.69	0.71	0.69	1.5

Excellent stability is achieved with RSDs below 2% for all elements, for high and low concentration, including alkali elements. Absolutely no drift was observed.

5. Conclusion

Analysis of wines for toxicological purposes as well as for quality control can easily be performed using the ACTIVA-M. The high level of robustness allows the analysis of undiluted wine using a standard configuration. Thanks to the vertical torch and the unique Total Plasma View feature, low detection limits are achieved along with reliability and stability. Thus, enhanced quality of the results are guaranteed.

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