SHIMADZU

Application News

No. SCA-115-023

Spectroscopy – ICP-OES

Determination of Heavy Metals in Wine using simultaneous ICP-OES

Figure 1: Grapes – the source of wine

Food and drinks are always a hot topic of discussion and in the focus of “state of the art” analytical techniques. Food scandals all around the world from eggs to horsemeat, tainted wine, oil, and milk force the European Community to establish an integrated approach to food control. The target is a high level of food safety, animal health, animal welfare and plant health within the European Union through so-called “farm-to-table” measures and monitoring, ensuring the effective functioning of the European market.

■ Wine Quality

The quality standards for wine analysis are defined in national wine regulations such as the “German Weinverordnung” (Bundes-gesetzblatt Teil 1 Nr. 32) from 22nd May 2002, with the latest revision in 2012. It includes the classification of wines from different locations, as well as the production process, alcohol concentrations and the maximum allowable concentrations of the elements listed in Table

Element	Max. concentration [mg/L
Aluminum	8.00
Arsenic	0.10
Boron	80.0
Cadmium	0.01
Copper	2.00
Lead	0.25
Tin	1.00
Zinc	5.00

Table 1: Maximum allowable concentrations of elements in wine

■ Quantitative Analysis

For quantitative determination of the elements in the required concentration range, ICP is the most preferable tool for quality control because of a high sensitivity, a wide dynamic range and a high sample throughput. Figure 2 shows the new simultaneous ICPE-9820 with CCD (charge-coupled device) detector, which has been used for all determinations. This system configuration is equipped with a unique optical system which sets new standards with respect to performance and speed and can be optimized for any type of complex samples such as wine analysis.

The vacuum system allows precise analysis of elements in the lower UV range under extremely stable conditions. The use of a mini torch allows a cooling gas flow rate of only 10 L/min. The system setup for determination of low concentration heavy metals in wine has been optimized using the dual view option in order to analyze low level elements at the same time with high matrix elements. The

Figure 2: ICPE-9820

wine samples have been diluted 4 times, and aspirated in the same way as aqueous solutions in the cyclone chamber. The standard solutions have been prepared including ethanol in order to match the matrix with an ethanol concentration of 3.5 % after dilution, for direct aspiration into the mini torch. Table 2 shows a summary of the system parameters.

Parameter	Setting
RF generator power	1.2 kW
Cooling gas	10 l/min
Plasma gas	0.6 l/min
Carrier gas	0.7 l/min
Nebulizer	Coaxial
Plasma observation	Axial/Radial
Sensitivity	Wide Range
Exposure time	15 sec.

Table 2: Analytical conditions for wine analysis

The concentration of copper in wine is limited to a maximum concentration level of 2 mg/L. In case of higher copper concentrations the wine may have a metallic, bitter taste and the fermentation process will also be influenced by higher copper concentrations. Copper in wine originates from the Bordeaux mixture fungicide, which is a mixture of copper (II) sulfate ($CuSO_4$) and calcium hydroxide ($Ca(OH)_2$) solution used in vineyards to protect against downy mildew and other fungi. Since the Bordeaux mixture is applied in large quantities, the copper accumulates in the soil and becomes a pollutant. This is why the Bordeaux mixture will most probably be banned in the European community as of 2016. The calibration curve in figure 3 shows the standards with concentrations starting at 250 µg/L up to the maximum concentration of 1000 µg/L. The limit of detection is calculated with < 0.02 µg/L (3 s). Furthermore the determination of arsenic and lead is important, as these elements still can be found in the environment generated from lead arsenate ($PbHAsO_4$) which has been used as an inorganic insecticide until 1988, after which it was officially banned.

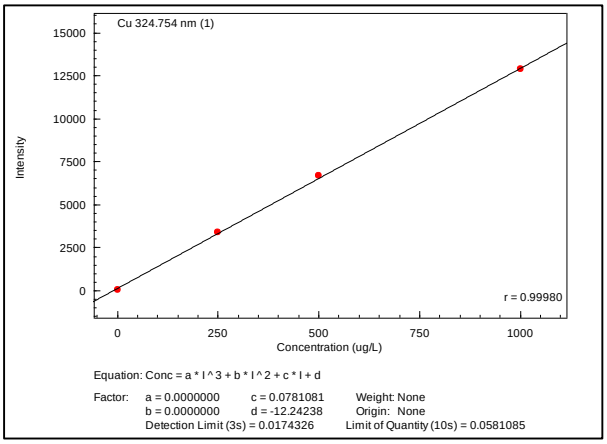

Figure 3: Copper Calibration

■ Conclusion

For the element analysis, ICP-OES Spectrometry using a simultaneous instrument is the “state of the art” tool for the daily routine in quality control of wine samples. The ICPE-9820 in dual view configuration is flexible enough to achieve calibration ranges in axial view in ppb level concentrations and at the same time high concentration levels of Boron, Potassium, Sodium and others in ppm scale in radial observation as well.

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