Application Note 43189

Iron, Copper and Zinc Determination in Wine using Flame Atomic Absorption Spectroscopy

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Key Words

Copper, heavy metal testing, iron, wine, zinc

Goal

This application note describes the analysis of heavy metals in wine by Flame AAS following a simple dilution of the sample.

Introduction

Heavy metals occur naturally in the ecosystem through anthropogenic sources such as pollution. Living organisms require varying amounts of "heavy metals" such as iron, cobalt, copper, manganese, molybdenum, and zinc. However, exposure to excessive levels can be damaging to organisms. [1] This exposure can easily occur through dietary intake, through the consumption of wine for example. There are many contributing factors that determine the metal content in wine, including; soil, type of vineyard, various steps of the wine production cycle (from grape to the finished wine) and from wine processing equipment, conservation and bottling. [2]

Some metals can affect the quality of the wine, in particular zinc, copper and iron which can lead to haze formation in bottled wine. It is therefore recommended that winemakers screen for these metals prior to bottling. In addition, due to the increased use of copper sulfate as a fining agent, copper levels in wine are rising worldwide. [3] It is essential to ensure that levels are below the maximum recommended of 0.5 mg/L for copper and 30 mg/L for iron and zinc that is stipulated by EU directive EC 606/2009. The prescribed method of analysis is Flame Atomic Absorption Spectroscopy (FAAS). [4]

Method

Instrumentation

A Thermo Scientific™ iCE™ 3300 AA was used for the FAAS measurements of iron, copper and zinc in different wine samples. The Thermo Scientific™ SOLAAR™ software contains pre-set spectrometer parameters for iron, copper and zinc and these were used to measure the samples (Figure 1). This in turn has greatly simplified the method development. Each measurement was performed in triplicate and the fast resamples option was selected to speed up the analysis. The final set of spectrometer parameters used is shown in Table 1.

Table 1. Instrument settings for the iCE 3300 AAS.

Parameter	Iron	Copper	Zinc
Wavelength	248.3 nm	324.8 nm	213.9 nm
Band pass	0.2 nm	0.5 nm	0.2 nm
Background Correction	D2	D2	D2
Lamp Current	75%	75%	75%
Signal	Continuous	Continuous	Continuous
Flame type	Air-Acetylene	Air-Acetylene	Air-Acetylene
Fuel flow rate	0.9 L/min	1.1 L/min	1.2 L/min
Measurement Time	4s	4s	4s
Replicates	3	3	3

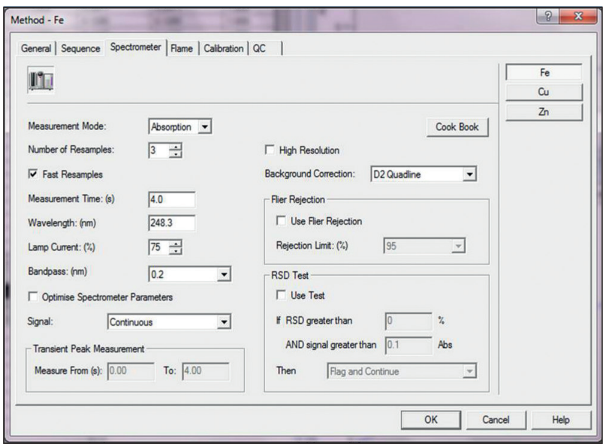

Figure 1: An example of a Flame parameters set-up for Iron.

Sample Preparation

Standards

Iron, copper and zinc stock standard solutions containing 1000 mg/L of these metals were diluted with a pre-mixed solution of deionised water and analytical grade concentrated nitric acid to provide working standards of various concentrations in 2% (w/v) HNO_3. The calibration blank solution used throughout was a 2% w/v HNO_3 solution. For iron and copper the working standards were 1, 5, and 10 mg/L. For zinc, standards were 0.5, 1 and 2 mg/L.

Samples

A total of 3 different brands of products were analyzed, representing the three types of wine that are commercially available. A 10 ml sample of each wine was accurately measured and transferred into a 40 mL flask. 0.8 mL of analytical grade HNO_3 was added and then made up to a final volume of 40 mL with Ultra Pure DI water. Spikes were similarly prepared with 100 uL of 1000 mg/L of iron and copper and 40 uL of 1000 mg/L zinc standard solution. This resulted in a concentration of 2.5 mg/L iron and copper and 1 mg/L zinc spike in the final 40 mL sample volume. Differing spike levels are due to the different sensitivities of FAAS for these heavy metals.

Calibration

The calibration curve was obtained using the calibration standards that were manually prepared. The method of quadratic least squares fit was used. An example of a standard calibration curve is given in Figure 2.

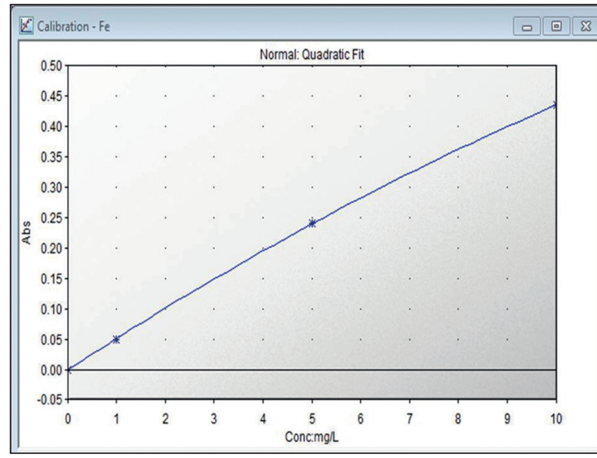

Figure 2: Calibration curve for Iron.

Results

The results indicate that these wine samples are within the recommended range of 0.5 mg/L for copper and less than 30 mg/L for iron and zinc. The heavy metal levels found in these types of wine are low and comply with the guidelines. Table 2 shows the results for measured iron, copper and zinc concentrations in the wine samples.

Table 2. The measured concentration of iron, copper and zinc measured in the wine samples analyzed.

*All data was calculated from 3 replicate readings for each solution, the results are calculated with a view of 1:4 dilution.

Sample ID	Iron (mg/L)	Copper (mg/L)	Zinc (mg/L)
White wine*	1.165	0.027	0.988
Rosé wine*	1.835	0.028	0.514
Red wine*	2.437	0.037	0.575

Table 3 displays the results for the spike recoveries, showing the recovery range of 93.5 – 101.0 %. These results indicate the reliability of the data and the instrument.

Table 3. Results to show the expected and measured concentrations with percentage spike recovery for three in the wine samples

Sample ID	Expected concentration spike. (mg/L)			Measured concentration spike. (mg/L)			Spike Recovery (%)		
	Iron	Copper	Zinc	Iron	Copper	Zinc	Iron	Copper	Zinc
White wine	2.789	2.507	1.247	2.616	2.429	1.202	93.8	96.9	96.4
Rose wine	2.965	2.507	1.129	2.995	2.463	1.056	101	98.3	93.5
Red wine	3.122	2.509	1.144	3.131	2.435	1.097	100.3	97.1	95.9

Conclusion

The Thermo Scientific iCE 3300 AA demonstrates an ideal solution for iron, copper and zinc determination in wine samples following dilution. The optimization wizards within the Thermo Scientific SOLAAR software make method development simple and ensure optimum analytical conditions.

References

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