Challenge

Determination of Na, K, Fe, Ca, Cu, Zn, Mn and Mg in wine

Solution

Application of High-Resolution Continuum Source Flame AAS for determination of macro-, micro- and ultra-micro element contents using only one dilution factor in a single measurement run

Determination of Element Contents in Wine Using HR-CS Flame AAS

Introduction

Food and drinks are a hot topic of discussion and in the focus of interest with respect to the applicability of certain analytical techniques. Wine consists of a fairly complex matrix including water, sugar, alcohol as well as a great variety of organic and inorganic components. Ethanol especially influences the transport and nebulization properties of the sample due to changes in density and surface tension compared to aqueous standard solutions. The composition of wine is affected by many factors related to a large extent to its specific production area e.g. grape type, soil and climate, culture, wine production process, transport and storage. Wine contains macro-elements with concentrations above 10 mg/L (Na, K, Mg, Ca), micro-elements in the range between 10 mg/L and 10 µg/L (Fe, Cu, Zn, Mn, Pb) and ultramicro-elements with concentrations below 10 µg/L (Cr, As, Cd, Ni). Even though some of these metals such as copper and zinc are essential biometals and lack thereof can lead to serious illness, the majority of them have carcinogenic or toxic effects even at trace levels. Besides consumer health issues, some of these elements may cause precipitation of tartrates and other organic complexes and thus need to be monitored to ensure stability and storability of the wine. For these reasons, it is of great importance to constantly monitor the levels of certain elements.

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Atomic absorption spectrometry is the most commonly used technique for metal determination in wines. In fact, the AAS technique is generally little prone to interferences caused by organic compounds due to the high temperatures involved in the atomization step. This application note describes a straightforward and fast analysis of wine using the contrAA 800. Only one dilution factor needed to be applied to measure all of the above listed elements as all absorption lines in the spectral range of 185 to 900 nm can be accessed in the HR-CS AAS. Thus, some of the elements were determined by using secondary wavelengths, while others were evaluated using the primary wavelength.

Instrumentation

The analysis was carried out with the High-Resolution Continuum-Source AAS instrument, contrAA 800, equipped with a 100 mm burner head, the injection switch SFS (segmented flow star) and an autosampler with automatic dilution function.

Table 1: Configuration of the method and the instrument

Parameter	Specification
Instrument	contrAA 800 F
Burner width	100 mm
Fuel type	C_2H_2/air
Burner angle	0°
Spectral observation width	200 pixels
Delay / rinse time	18 s
Integration time	3 s (3 replicates)
Evaluation pixels	3
Baseline fit	IBC (iterative background correction)
Auto sampler	Yes (AS-FD)

Table 2: Method and evaluation parameters

Element	Wavelength [nm]	Fuel gas [L/h]	Burner height [L/h]	Spectral observation width [nm]
Na	330.2370	65	4	0.45
K	404.7201	70	7	0.50
Fe	248.3270	80	6	0.31
Ca	422.6728	75	8	0.57
Cu	324.7540	50	6	0.39
Zn	213.8570	70	5	0.27
Mn	279.4817	70	4	0.34
Mg	202.5820	70	4	0.27

Samples and Reagents

The samples were pre-diluted in a ratio of 1:10 with 1.5% HCl and 0.5% CsCl/$LaCl_3$. Samples were treated in an ultrasonic bath in order to remove the carbon dioxide.

Results and Discussion

Good method robustness for the analysis of diverse metals in wines was found for all elements of interest. By combination of sensitive and insensitive absorption lines within one method most elements can be measured out of the same pre-dilution. If the concentration range is still exceeded the sample can be diluted automatically by the autosampler and is re-measured for the respective element.

Before measuring the wine samples were treated in an ultrasonic bath in order to remove the carbon dioxide.

Na, K, Fe, Ca, Cu, Zn, Mn and Mg can be determined without interferences in the wine samples. It was found that the addition of 0.5% CsCl/$LaCl_3$ is necessary for determination of Ca in order to avoid atomization interferences. QC spike recovery rates in the range between 93.2 and 108% demonstrate the independence of the measurements from matrix effects.
By using the injection switch SFS (segmented flow star) the mixing chamber system and the burner head are permanently rinsed. Thus deposits and clogging, caused by the sample matrix (e.g., sugar) are avoided.

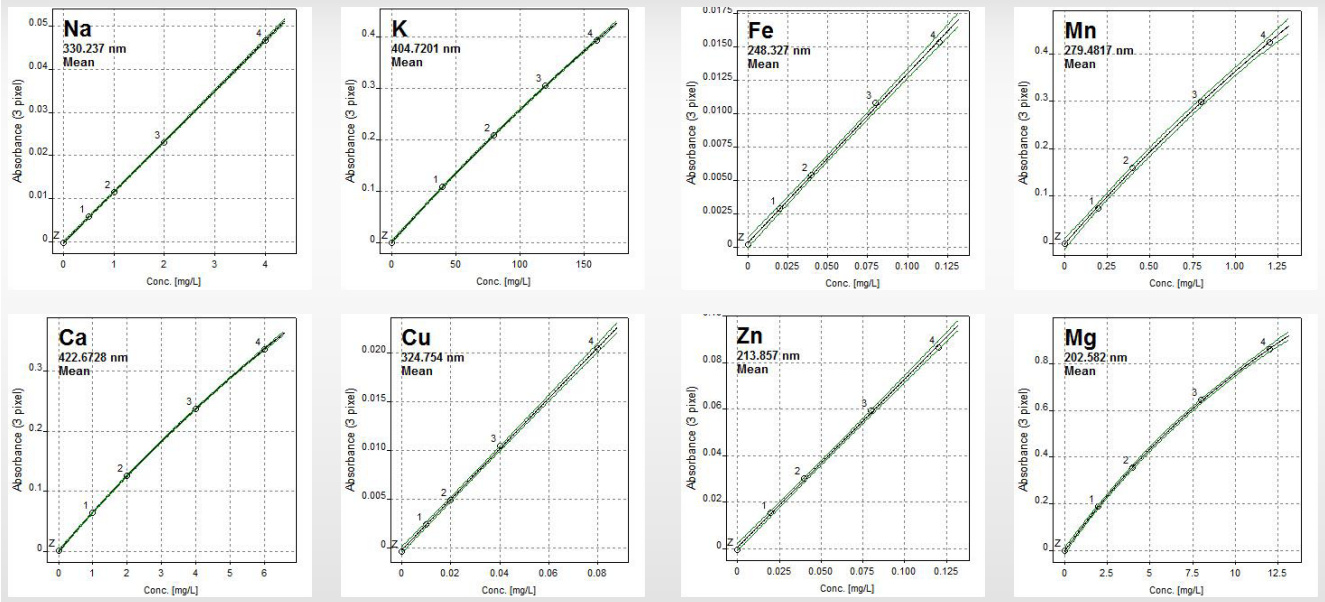

Figure 1: Calibration curves

Table 3: Results from measurement of wine samples on contrAA® 800

Sample	Element / Line	Pre-DF	Concentration [mg/L]	RSD [%]	Spike concentration [mg/L]	Spike Recovery [%]
V1	Na 330.237 nm	10	7.99 ± 0.25	0.3		
	K 404.7201 nm	10	826 ± 10.8	0.8		
	Fe 248.327 nm	10	0.71 ± 0.03	1.3		
	Ca 422.6728 nm	10	50.3 ± 0.35	1.1		
	Cu 324.754 nm	10	0.07 ± 0.01	4.8		
	Zn 213.857 nm	10	0.28 ± 0.02	0.7		
	Mn 279.4817 nm	10	2.32 ± 0.13	0.7		
	Mg 202.582 nm	10	130 ± 1.20	0.7		

Sample	Element / Line	Pre-DF	Concentration [mg/L]	RSD [%]	Spike concentration [mg/L]	Spike Recovery [%]
V2	Na 330.237 nm	10	7.53 ± 0.25	1.2		
	K 404.7201 nm	10	579 ± 10.6	0.7		
	Fe 248.327 nm	10	0.61 ± 0.03	1.5		
	Ca 422.6728 nm	10	62.8 ± 0.49	0.9		
	Cu 324.754 nm	10	0.18 ± 0.01	4.7		
	Zn 213.857 nm	10	0.58 ± 0.02	0.3		
	Mn 279.4817 nm	10	1.68 ± 0.13	1.0		
	Mg 202.582 nm	10	160 ± 5.11	1.4		
V3	Na 330.237 nm	10	23.9 ± 0.26	0.6		
	K 404.7201 nm	10	599 ± 10.7	1.0		
	Fe 248.327 nm	10	1.93 ± 0.18	0.5		
	Ca 422.6728 nm	10	51.3 ± 0.35	1.3		
	Cu 324.754 nm	10	0.61 ± 0.01	0.6		
	Zn 213.857 nm	10	1.38 ± 0.14	1.4		
	Mn 279.4817 nm	10	2.58 ± 0.13	0.9		
	Mg 202.582 nm	10	129 ± 4.54	0.9		
V4	Na 330.237 nm	10	12.6 ± 0.24	0.6	1	93.2
	K 404.7201 nm	10	469 ± 10.3	0.8	40	108
	Fe 248.327 nm	10	1.15 ± 0.04	1.3	0.04	103
	Ca 422.6728 nm	10	36.8 ± 0.36	1.5	1	103
	Cu 324.754 nm	10	0.63 ± 0.01	1.3	0.04	96.5
	Zn 213.857 nm	10	0.28 ± 0.02	1.2	0.04	101
	Mn 279.4817 nm	10	1.42 ± 0.13	0.1	0.2	101
	Mg 202.582 nm	10	77.6 ± 0.80	0.5	2	98.3

Conclusion

This study shows that contrAA 800 provides a fast and easy to use instrument solution for the analysis of wine samples. HR-CS Flame AAS offers a suitable technique for this kind of application, providing fast sequential determination of different elements in varying concentrations in a single measurement run only.

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