



Detection of the origin of Brazilian wines based on the determination of only four elements using high-resolution continuum source flame AAS

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ABSTRACT

A method has been developed to determine 10 elements in Brazilian red wines using high-resolution continuum source flame atomic absorption spectrometry, a technique that allows the fast sequential determination of an essentially unlimited number of elements per sample, each one under previously optimized conditions. All measurements were made without sample preparation, using aqueous standard solutions for calibration. The results were in agreement within 99% of confidence (*t*-test) with those obtained by inductively coupled plasma optical emission spectrometry. The same grape, *Cabernet sauvignon*, was used in all experiments, and the wines from each region were prepared especially for this investigation in order to avoid any confusion due to grapes from other regions, which are often used in commercial wines. The elements K, Mn, Rb and Sr were found to be the best indicators for the origin of the wines, based on a Principal Component Analysis.

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1. Introduction

Wine production and consumption have increased considerably over the last decade in Brazil [1]. To maintain the quality of wine, some properties have to be monitored regularly, such as pH, sugars, organic and inorganic compounds [2]. In this context, the determination of metals present in the wine is crucial; some elements are essential for our organism and wine is known as a good source for them [3]. The mineral composition of wine is based in the grape quality, which is directly related with the soil,

the climatic conditions and the production procedures [4]. The organoleptic properties are also significantly influenced by the presence of some elements [5]; the most important ones are Be, Ca, Co, Cu, K, Li, Mn, Na, Rb and Sr [6–9].

Wine is known to have a complex matrix and many studies in the literature report the determination of metals in wine [5]. Atomic and mass spectrometric techniques, such as flame atomic absorption spectrometry (F AAS), graphite furnace atomic absorption spectrometry (GF AAS), inductively coupled plasma optical emission spectrometry (ICP OES) and inductively coupled plasma mass spectrometry (ICP-MS) are most frequently used for the wine analysis. The principal advantages of the AAS techniques are the high sensitivity in the case of GF AAS and simplicity and low cost in the case of F AAS. The disadvantage of AAS techniques is that they are typically designed for the determination of just one element at-a-time [3–5,10]. The plasma techniques have the advantages of high sensitivity and multi-element determination, with the disadvantage that compromised conditions have to be used, which are not optimum for each element, and the higher cost, mostly in the case of ICP-MS [3–5,11].

Santos et al. [12] evaluated different pre-treatment procedures of wine for the determination of iron and manganese by F AAS because of the relatively high concentration of these elements. They found no significant difference between acid digestion and

Abbreviations: SGA, Serra Gaúcha; VVi, Vale dos Vinhedos; PBa, Pinto Bandeira; CCS, Campos de Cima da Serra; CGa, Campanha Gaúcha; SCa, Serra Catarinense; OPR, Oeste do Paraná; VSF, Vale do São Francisco; AAS, Atomic Absorption Spectrometry; CCD, Charge Coupled Device; F AAS, Flame Atomic Absorption Spectrometry; GF AAS, Graphite Furnace Atomic Absorption Spectrometry; HCA, Hierarchical Cluster Analysis; HR-CS F AAS, High-Resolution Continuum Source Flame Atomic Absorption Spectrometry; ICP-MS, Inductively Coupled Plasma Mass Spectrometry; ICP OES, Inductively Coupled Plasma Optical Emission Spectrometry; LOD, Limit of Detection; LOQ, Limit of Quantification; PCA, Principal Component Analysis; PC, Principal Component; RSD, Relative Standard Deviation.

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photo-oxidation of the wine samples. Sauvage et al. [13] used only the dilution of the wine samples to determine copper, iron, potassium, sodium, magnesium and calcium.

Moreno et al. [14] determined nickel and lead in commercial wine samples from Spain by GF AAS using dry ashing, followed by the addition of a 5% nitric acid solution. The results were used to classify Spanish wines according to their denomination of origin (DO). The same authors also determined Al, Ba, Cu, Fe, Mn, Sr, Zn, Ca, K, Na and Mg by ICP OES for the same purpose and using the same conditions. Dessuy et al. [15] developed a method to determine lead in wine via direct analysis using GF AAS, comparing different atomizers; the transversally heated filter atomizer showed better sensitivity and lower background absorption compared to a conventional transversely heated platform atomizer.

Bentlin et al. [16] evaluated wine samples from South America (Argentina, Brazil, Chile and Uruguay) by ICP OES and ICP-MS. They determined 45 elements after nitric acid digestion, and the results were used to classify each sample according to its country of origin using the Principal Component Analysis (PCA) and the Hierarchical Cluster Analysis (HCA). Šperková and Suchánek [17] determined 27 elements in Bohemian wines (Czech Republic) by ICP OES and ICP-MS. The samples were subjected to nitric acid digestion, after ethanol was eliminated by heating in open vessels.

High-resolution continuum source atomic absorption spectrometry (HR-CS AAS), which was introduced commercially less than a decade ago [18], appears to be an alternative technique for the determination of trace and minor elements in wine samples. The xenon short-arc lamp, used as a continuum source, provides the possibility to determine almost all known elements in a fast sequential manner when F AAS is used. The possibility of using the principal and secondary lines without loss in signal-to-noise ratio (SNR), the choice of the line wings to enhance sensitivity and/or to extend the linear working range are among the most important advantages brought about by this technique [10,18].

Some analytical techniques, such as ICP OES and ICP-MS provide a great number of results; however, the extraction of information might be difficult and time-consuming, even when using statistical tools, such as pattern recognition [19]. In wine samples, these tools can be used to determine their geographical origin, to verify frauds or to control the quality of the wine produced [20]. The most common pattern recognition techniques used in these cases are PCA and HCA [21].

The principal objective of this work was to develop a precise and accurate method for the determination of Be, Ca, Co, Cu, K, Li, Mn, Na, Rb and Sr in Brazilian red wine samples by HR-CS F AAS. With the results obtained by the developed method, techniques for pattern recognition were applied to verify some group formation. No commercially available wines were used in this study in order to avoid any confusion due to grapes from other regions, which are quite often added in this type of wines. Instead, only one kind of grape (*Cabernet sauvignon*) was used and the wine produced in small quantity directly

at the winery under strict control, but following the regular wine-making procedure.

2. Materials and methods

2.1. Instrumentation

A Model contraAA 700 high-resolution continuum source atomic absorption spectrometer (Analytik Jena, Jena, Germany), with a graphite furnace and a flame atomizer in two separate sample compartments, was used for most of the experiments. The spectrometer is equipped with a xenon short-arc lamp operating in a hot-spot mode, a prism pre-monochromator, an echelle grating monochromator for high resolution, and a charge-coupled device (CCD) array detector; the resolution is about 1.5 pm per pixel at 200 nm. An air-acetylene flame was used for the determination of Co, Cu, K, Li, Mn, Na and Rb, and a nitrous oxide-acetylene flame for Be, Ca and Sr under optimized conditions as specified in Table 1. High-purity acetylene and high-purity nitrous oxide (both 99.0% v/v, White Martins, Brazil) were used as fuel and oxidant gas, respectively. The aspiration rate used was 8 mL min⁻¹, and all measurements were carried out in triplicates. A Model SFS 6 injection valve (Analytik Jena) was used to reduce the sample consumption and to maintain the stability of the flame, as no air is aspirated when the standard or the sample solution is changed.

A Model MPX-CCD inductively coupled plasma optical emission spectrometer (Varian, Mulgrave, Australia) with radial configuration was used for the ICP OES measurements. Two different nebulization chambers were used in this equipment according to the concentration of the element to be determined. An ultrasonic nebulizer model U-5000 AT⁺ (CETAC Technologies, Omaha, NE) was used for the microelements Be, Co, Cu, Li, Mn, Rb and Sr, and a Sturman-Masters nebulization chamber (Varian) was applied for the macroelements Ca, K and Na. The operating conditions of the ICP are summarized in Table 2.

A microwave oven Model Microwave 3000 (Anton Paar, Graz, Austria), equipped with an eight-vessel capacity rotor and microwave-operated UV lamps was used for pre-treatment of the wine samples to evaluate if there is any need for sample digestion.

Table 2
Operational conditions for metals determination in wine samples by ICP OES.

Power (W)	1300
Auxiliary gas flow-rate (L min ⁻¹)	2.25
Plasma gas flow-rate (L min ⁻¹)	15
Measurement height (mm)	15
Pump rotation (rpm)	35
Nebulizer pressure (kPa)	240

Table 1
Instrumental parameters for HR-CS F AAS measurements.

Element	Wavelength (nm)	Relative sensitivity (%) ^a	Flame	C ₂ H ₂ flow-rate (L h ⁻¹)	Burner height (mm)	Pixels evaluated
Be	234.861	100	C ₂ H ₂ -N ₂ O	240	6	3
Ca	239.856	1.1	C ₂ H ₂ -N ₂ O	210	6	3
Sr	460.733	100	C ₂ H ₂ -N ₂ O	210	6	1
Co	240.725	100	C ₂ H ₂ -Air	80	7	3
Cu	324.754	100	C ₂ H ₂ -Air	40	5	3
K	404.720	0.24	C ₂ H ₂ -Air	50	5	1
Li	670.785	100	C ₂ H ₂ -Air	45	4	3
Mn	279.827	67	C ₂ H ₂ -Air	60	6	3
Na	330.237	0.48	C ₂ H ₂ -Air	50	6	3
Rb	780.027	100	C ₂ H ₂ -Air	45	5	1

^a Compared to the main resonance line for this element.

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