



Determination of boron in Turkish wines by microwave plasma atomic emission spectrometry



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ABSTRACT

Boron in various Turkish red wine and white wine samples was determined by microwave plasma-atomic emission spectrometry (MP-AES) at 249.677 nm. MP-AES is a novel instrument which provides a low-cost analysis based on a micro-wave plasma generated with nitrogen. Wine samples were not pre-treated prior to analysis. The recovery rate of boron added to the 1:1 diluted wine samples was around 80% which showed interference due to the matrix of the wine samples. Therefore standard addition method was used for quantifications. The limit of detection and limit of quantification were 0.08 and 0.28 $\mu\text{g mL}^{-1}$, respectively. A satisfactory linearity ($r^2 > 0.999$) was obtained up to 10 mg L^{-1} of boron. The range of boron concentrations in various wine samples was 4.2–10.8 mg L^{-1} .

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1. Introduction

Boron (B) is a non-metallic element which is a common constituent of foods since it accumulates to plants from boron-rich soils and also some boron compounds used as preservatives for foods. Boron deprivation effects bone development, brain functions, macromineral metabolism, energy substrate utilization, immune function and insulin secretion (Nielsen, 1997). According to World Health Organization, a tolerable daily intake (TDI) in human consumption is 0.16 mg B per kg body weight (WHO., 2003).

Boron can be determined using various analytical methods such as flame atomic absorption spectrometry (FAAS) (Mihaljevic, Sebek, Lukesova, & Bouzkova, 2001), electrothermal atomic absorption spectrometry (ETAAS) (Burguera, Burguera, Rondon, & Carrero, 2001), inductively coupled plasma mass spectroscopy (ICP-MS) (Hokura, Matsuura, Katsuki, & Haraguchi, 2000) and inductively coupled plasma atomic emission spectroscopy (ICP-OES) (Krejcova & Cernohorsky, 2003). Since boron is a thermally refractive element and forms stable carbides, its sensitivities in ETAAS and FAAS are low causing high limit of detection (LOD) values. Therefore, it can be determined by nitrous-oxide acetylene flame or by ETAAS only at high atomization temperature and prolonged times. Nevertheless, high LOD values restrict the determinations of low

concentrations. Boron can be determined by ICP-AES and ICP-MS at low concentrations however ICP techniques have their own drawbacks such as high price of ICP-MS as well as very high argon consumption of the both instrument. In literature there is only one paper about boron concentration in wine samples. Thiel and Danzer were determined boron in wine samples by ICP-OES and found concentrations between 2.0 and 5.8 mg L^{-1} (Thiel & Danzer, 1997).

Microwave Plasma Atomic Emission Spectrometry (MP-AES) is a recently developed plasma technique. This technique uses 2.45 GHz microwave magnetic field and nitrogen gas to generate stable plasma. It came out as a plasma technique with minimum (or no) cost for gas because it uses nitrogen for plasma, which is generated from air by a nitrogen generator or from a nitrogen cylinder. The plasma temperature is around 5000 K, which is suitable to excite many elements. However, the analysis by MP-AES is not free of problems. Spectral and non-spectral interferences are highly effective on the results as well as the total dissolved salt (TDS) content of the matrix is limited around 2%. Otherwise torch is damaged due to the accumulation of salt. The LOD values are not as low as ICP-MS or ETAAS but lower than FAAS and comparable with those of ICP-AES (Agilent Technologies, 2013).

Up to now, only a few papers have been published on the studies by MP-AES. Li et al. determined copper, iron, manganese and zinc in animal feed and fertilizer (Li, Simmons, Shrader, Herrman, & Dai, 2013). Balaram et al. designed and developed a method for quantitative determination of Au, Ag, Pt and Pd in several rock and ore reference samples (Balaram et al., 2013). Donati et al. determined

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Cr, Ni, Pb and V in gasoline and ethanol fuel (Donati, Amais, Schiavo, & Nobrega, 2013) and finally Hettipathirana determined B in high-temperature alloy steel (Hettipathirana, 2013).

In this study, a novel method was described for the determination of boron in wine samples using MP-AES. The calibration techniques were compared and the experimental/instrumental parameters were optimized. Finally, boron concentrations in different wine samples were determined.

2. Experimental

2.1. Instrumental

All experiments were carried out with Agilent 4100 MP-AES which is equipped with Inert One Neb nebulizer and double-pass glass cyclonic spray chamber (Agilent Technologies, Melbourne, Australia). Nitrogen was obtained from air using a F-DGSI, Thyster 8/1 LV, (Innovative Gas System Co., Evry, France), nitrogen generator. Prior to reading samples, 10 s for uptake time, 20 s for torch stabilization time is set. For all experiments, 5 s read time with 5 replicates was fixed. Before starting to study torch alignment and wavelength calibration were carried out automatically by the instrument using a wavelength calibration solution (Agilent Technologies, Melbourne, Australia, 2013).

2.2. Reagents and solutions

Water with 18.2 $\mu\Omega$ cm resistivity obtained by a TKA reverse osmosis and a TKA deionizer system (TKA Wasseraufbereitungsstandards, systeme GmbH, Niederelbert Germany) was used for all dilutions. Calibration standards were prepared from 1000 mg L⁻¹ of boron solution (Carlo Erba, Radona, Italy) daily. Red wine and white wine samples produced in different regions of Turkey from different grape varieties were bought from markets.

2.3. Procedure

The boron concentrations in Turkish wines were determined applying by standard addition technique. For this purpose, 5 mL aliquots of samples were completed to 10 mL with distilled water and standards. The External Gas Control Module (EGCM) was used to inject air into plasma to burn-off carbon. Results were given as the average of at least 5 replicate analyses. Blanks were prepared in 10% (v/v) ethanol in water to maintain the same plasma temperature and aspirating rate for the sample and the blank solutions.

3. Results and discussion

3.1. Optimization for boron determination by MP-AES

To obtain the highest sensitivity, the instrumental working parameters such as nebulizer pressure and viewing position were optimized by the instrument automatically and then fine adjustments were made. Nebulizer pressure was set to 120 kPa whereas viewing position set to 20. The calibration curves were formed at 249.772 nm, 208.889 nm, 249.677 nm, 208.957 nm and 330.244 nm. The highest slopes (i.e. sensitivity) were obtained at 249.772 and 249.677 nm with perfect linearity ($r^2 > 0.999$) in a wide range up to 10 mg L⁻¹ which includes the boron concentrations of samples even with standard addition technique (Fig. 1). Although the working range could be further extended providing non-linear calibration using rational fitting, the concentration of the analyte in the samples were low enough to work in the linear range.

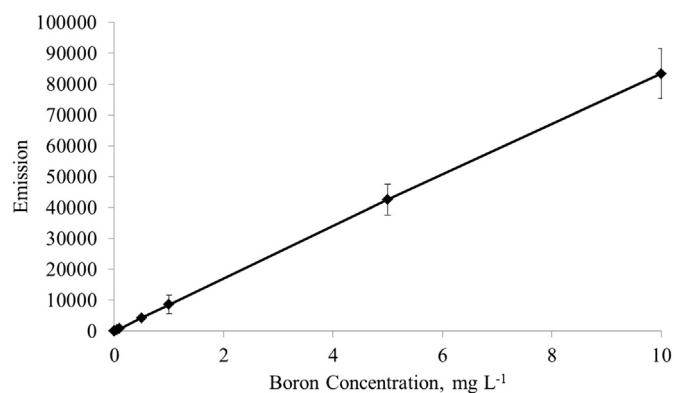


Fig. 1. Linearity range for boron at 249.677 nm.

It was reported that the secondary emission line of iron (249.698 nm) is very close to one of the analytical line of boron (249.772 nm) used in this study. Therefore, it is likely that overlapping of boron and iron lines may cause errors. Hettipathirana investigated the spectral interference of iron on boron determination and reported that for the determination of boron in steel samples, the high iron concentration caused significant spectral interference. Therefore, a correction with respect to iron was advised (Hettipathirana, 2013). However, in wine samples, it is expected that the relatively low iron concentrations did not cause any detectable spectral interference at 249.772 nm. Nevertheless, since the sensitivities and linearities for boron at 249.772 nm and 249.677 nm were not significantly different, to be on the safe side, all quantifications were performed at the latter wavelength.

3.2. Accuracy test and choice of calibration technique

The boron in matrix-free calibration standards is atomized and excited without being influenced from the matrix whereas in the sample may be influenced by sample constituents which results different emission sensitivities compared to standards. In this case, the quantification based on the comparison of emission intensities for matrix-free standards with sample causes wrong results. The non-spectral interferences due to sample matrix were tested by (i) the recovery of standards added to the samples, (ii) comparison of the results with those obtained by standard addition and linear calibration techniques. In the both cases, the analyte in the sample and standards are in the same matrix and exposed to the same effects.

When linear calibration technique was applied for 1:1 diluted wine samples, the boron concentrations added to some wine sample were recovered around 80% which may attributed to lower

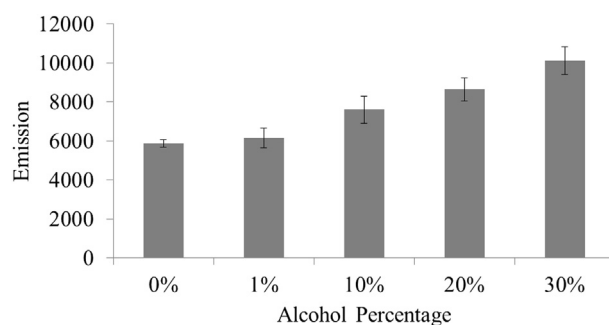


Fig. 2. Effects of ethanol content on the emission signal for 1 mg L⁻¹ of boron. Error bars present standard deviation of 5 replicates.

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