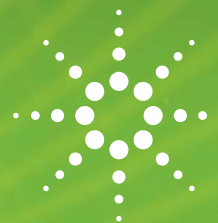


FOOD ANALYSIS

COGNAC ANALYSIS USING THE AGILENT 4200 MICROWAVE PLASMA-ATOMIC EMISSION SPECTROMETER (MP-AES)



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ABSTRACT

Cognac, famous all over the world for hundreds of years, is a brandy produced in the French region near the town of Cognac. Quality control is important throughout the wine making and preserving processes to ensure that the product is of the highest quality. For this purpose, elemental analysis of Cognac has been performed using the innovative Agilent 4200 Microwave Plasma-Atomic Emission Spectrometer (MP-AES). This technology runs on air, eliminating the need for flammable or expensive gases. The 4200 MP-AES is able to perform fast multi-element analyses with the lowest running costs and better analytical performance compared to flame atomic absorption spectroscopy (FAAS).

This application note presents the analytical performance of the Agilent 4200 MP-AES for the analysis of Cognac. Al, Ca, Cd, Cu, Fe, K, Na, Pb and Zn were measured in Cognac samples containing 40% ethanol (v/v) without any preparation prior to analysis. Detection limits down to ppb levels and excellent long term stability were achieved.

INTRODUCTION

Cognac, famous throughout the world for many years, is a brandy produced in the French region near the town of Cognac. Quality control is important throughout the wine making and preserving processes to ensure that the product is of the highest quality. For this purpose, elemental analysis in Cognac has been performed using the innovative Agilent 4200 Microwave Plasma-Atomic Emission Spectrometer (MP-AES).

The Agilent 4200 Microwave Plasma-Atomic Emission Spectrometer (MP-AES) uses magnetically-coupled microwave energy to generate a robust and stable nitrogen plasma capable even in challenging organic matrices. When compared to conventional flame AA, the 4200 MP-AES eliminates expensive and dangerous gases such as acetylene, resulting in lower running costs, unattended operation and improved productivity.

This application note describes the determination of Al, Ca, Cd, Cu, Fe, K, Na, Pb and Zn in Cognac samples containing 40% ethanol (v/v) without any preparation prior to analysis using an Agilent 4200 MP-AES, running with an Agilent 4107 Nitrogen Generator.



EXPERIMENTAL

Instrumentation

All measurements were performed using an Agilent 4200 MP-AES, with nitrogen supplied from an Agilent 4107 Nitrogen Generator. The sample introduction system consisted of a double pass spray chamber, a OneNeb nebulizer and a nebulization gas humidifier with semi-permeable capillary (no bubbling frit) to overcome any instability that may arise from the analysis of organic samples. The OneNeb nebulizer offers superior performance for this application over other comparable nebulizers, as it offers increased nebulization efficiency and a narrow distribution of small droplets as shown in Figure 1. Black-black tubing was used for samples to get the best sensitivity.

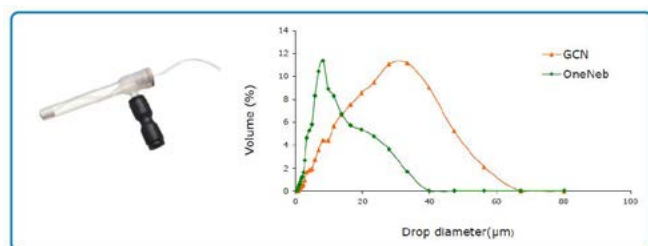


Figure 1: Droplet size distribution obtained from the OneNeb and a Glass Concentric Nebulizer (GCN).

The SPS 3 autosampler was used to deliver samples automatically to the instrument, allowing the system to be operated unattended. Fast pump was disabled to avoid plasma instability. The instrument was fitted with an External Gas Control Module (EGCM) allowing air-injection into the plasma to reduce background emissions, in order to optimize detection limits.

The instrument was controlled using Agilent's unique worksheet-based MP Expert software, which runs on the Microsoft® Windows® 7 operating system, and features automated optimization tools to accelerate method development by novice operators. For example, the software automatically adds the recommended wavelength and EGCM setting when elements are selected. Viewing position and nebulizer flow settings were optimized using the auto-optimization routines in MP Expert, by measuring the highest standard in 40% ethanol, in order to get the best sensitivity.

Also, the powerful Auto background correction mode easily and accurately corrects for the emission background arising from the organic matrix around the peak of interest. Each solution (standard/sample) was aspirated once. All elements, all wavelengths were read sequentially.

Sample and Standard Preparation

Cognac samples were obtained from the marketplace. Cognac contains 40% ethanol (v/v). The Cognac samples were directly analyzed, without any sample preparation.

Standard reference solutions used in the external calibration method were prepared at concentrations of 0.05 ppm, 0.1 ppm, 0.5 ppm and 1 ppm, from a 100 ppm multi-element calibration standard (S28). An Additional standard was prepared at 5 ppm for Ca, Cu, Na and K, from 1g/L mono-element standards. 40% ethanol (v/v) was used as the diluent. All standards were matrix matched with 40% ethanol (v/v).

For the spiked recovery test, Cognac samples were spiked with the standard solution S28 to give spiked concentrations of 0.05 ppm, 0.1 and 0.5 ppm.

Parameter Settings

Instrument operating conditions and analyte settings are listed in Tables 1a and 1b.

Instrument parameter	Setting
Nebulizer	Inert OneNeb + humidifier
Spray chamber	Double-pass glass concentric
Sample tubing	Black/black
Waste tubing	Blue/blue
Number of replicates	3
Uptake delay (s)	80
Stabilization time (s)	30
Rinse time (s)	80
Fast pump	Off
Background correction	Auto
Pump speed	15 rpm

Table 1a. Agilent 4200 MP-AES operating parameters

Element & wavelength (nm)	Reading time (s)	Nebulizer flow (L/min)	EGCM setting
Al 396.152 nm	3	0.85	High
Ca 422.673 nm	3	0.85	High
Cd 228.802 nm	10	0.4	Medium
Cu 324.754 nm	3	0.45	High
Fe 373.486 nm	10	0.4	High
K 766.491 nm	3	0.85	High
Na 589.592 nm	1	0.85	High
Pb 405.781 nm	10	0.6	High
Zn 213.857 nm	10	0.35	Medium

Table 1b. Analyte settings

RESULTS AND DISCUSSION

Calibration

The stable nitrogen plasma of the MP-AES can easily handle these Cognac samples and, as shown in Table 2, excellent correlation coefficients were obtained for the measured elements.

Element	Wavelength (nm)	Correlation coefficient
Al	396.152 nm	0.99999
Ca	422.673 nm	0.99999
Cd	228.802 nm	1
Cu	324.754 nm	1
Fe	373.486 nm	0.99995
K	766.491 nm	0.99996
Na	589.592 nm	1
Pb	405.781 nm	0.99999
Zn	213.857 nm	0.99999

Table 2. Calibration correlation coefficients

The 0.05 ppm standard spectrum for Pb 405.781 nm with automatic background correction and calibration curves for Pb 405.781 nm and Na 589.592 nm are shown in Figures 2, 3a and 3b.

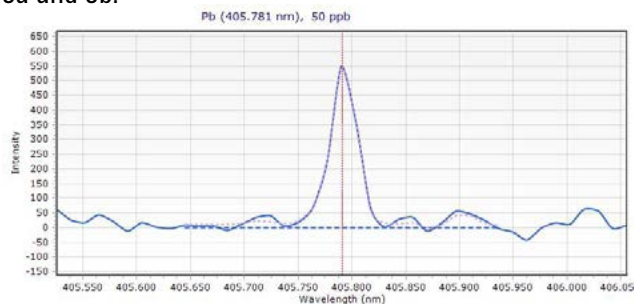


Figure 2. Representative spectrum for Pb 405.781 nm in the 0.05 ppm standard (40% ethanol)

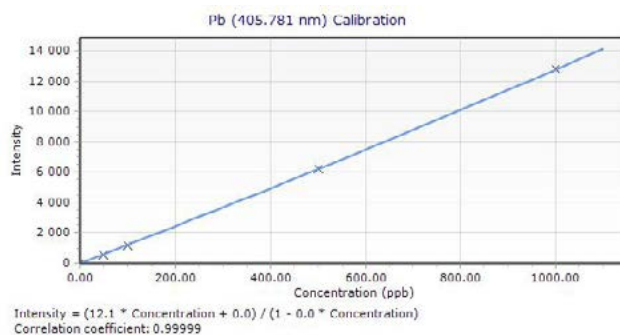


Figure 3a. Calibration curve for Pb 405.781 nm

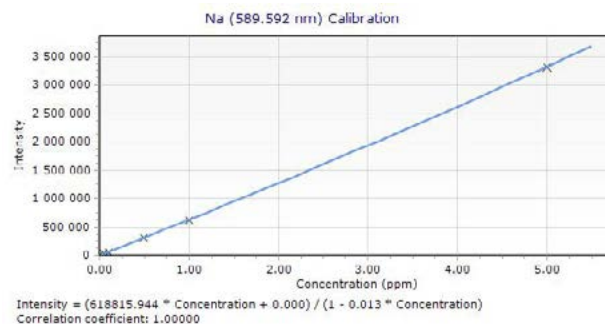


Figure 3b. Calibration curve for Na 589.592 nm

Spiked recoveries, precision and stability

Different spiked concentrations were tested at 0.05 ppm, 0.1 ppm and 0.5 ppm, according to the sensitivity of the element and its concentration in the Cognac sample. The spiked recovery results for the Cognac samples are shown in Table 3. The excellent recoveries demonstrate the ability of the 4200 MP-AES to accurately determine Al, Ca, Cd, Cu, Fe, K, Na, Pb and Zn in Cognac.

Samples and spiked samples were measured ten times over a period of 8 hours in order to study precision, accuracy and long term stability. Excellent precision was obtained for all elements analyzed, with less than 20% uncertainty and with a RSD of less than 4% over 8 hours, demonstrating the validity of the analytical method and the stability of the 4200 MP-AES.

The spectrum for the Cognac sample and the 0.05 ppm Pb spiked Cognac sample is shown in Figure 4.

Element & wavelength (nm)	Sample (ppm)	Theoretical Spike (ppm)	Spiked sample (ppm)	Recovery (%)	RSD (8 hours) (%)
Ca 422.673 nm	0.97	0.5	1.52	110	1.3
Cd 228.802 nm	0	0.05	0.052	103	1.5
Cu 324.754 nm	0.57	0.1	0.67	104	1.1
Fe 373.486 nm	0.023	0.1	0.118	95	1.5
K 766.491 nm	3.21	0.5	3.74	106	1.3
Na 589.592 nm	1.57	0.5	2.09	104	1.3
Pb 405.781 nm	0.013	0.05	0.059	92	1.9
Zn 213.857 nm	0.019	0.05	0.070	102	3.5

Table 3. Spiked recovery results in Cognac

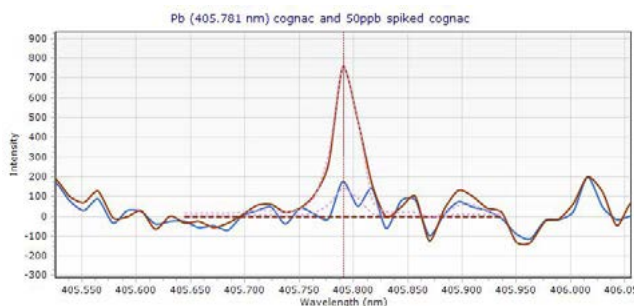


Figure 4. Spectrum for Pb 405.781 nm in the Cognac sample and the 0.05 ppm spiked Cognac sample

CONCLUSIONS

The Agilent 4200 MP-AES equipped with the Agilent 4107 Nitrogen Generator provides an ideal solution for the routine and direct analysis of Cognac. The nitrogen-based plasma excitation source exhibits a high tolerance level to organic solvent loading.

By injecting a controlled flow of air into the plasma via the EGCM and by using the OneNeb with the humidifier, excellent calibrations, detection limits, stability and recoveries were achieved in spiked Cognac samples at levels likely to be encountered in this analysis.

The 4200 MP-AES is the ideal instrument for those customers who are looking to transition away from FAAS and extend their laboratory's analytical capabilities. Recognized benefits of MP-AES include reduced running costs, enhanced productivity throughput thanks to unattended operation, improved safety, and improved analytical performance such as better detection limits and greater dynamic range.



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