Vegetable oil analysis for Biodiesel production using the Agilent 4100 Microwave Plasma-Atomic Emission Spectrometer

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European Winter PlasmaConference
JP-4

Agilent Technologies

Introduction

The production and use of biodiesels have extensively increased over the past few years because of the multiple benefits of these products : biodegradability, non-toxicity and the significant reduction of pollutants.

Biodiesel is produced from vegetable oil by a transesterification reaction. The vegetable oil quality is very important in this process. The presence of elements such as phosphorus, calcium and magnesium in vegetable oil can impact the quality of the end product and must thereby be analyzed before transesterification.

The innovative Agilent 4100 Microwave Plasma-Atomic Emission Spectrometer (4100 MP-AES) has been used for this purpose.

This new technology runs on air, with no need for flammable or expensive gases anymore.

Ca, Mg and P were measured in vegetable oil.

Experimental

The Agilent 4100 MP-AES was used with an External Gas Control Module (EGCM) allowing air injection into the plasma to prevent carbon deposition in the torch, to overcome any plasma instability that may arise from the analysis of organic samples, and to reduce background emissions.

The instrument was set up with the Organics kit comprising of the EGCM, the inert OneNeb nebulizer and solvent resistant tubing, along with a double pass spray chamber. The OneNeb nebulizer offers increased nebulization efficiency and a narrow distribution of small droplets. This allows the analysis to be performed at lower flow rates, reducing the solvent loading on the plasma, whilst maintaining excellent sensitivity.

The samples were diluted 1:2 with xylene.

Each solution (standard/sample) is aspirated once. All elements, all wavelengths are read sequentially.

Experimental

Instrument operating conditions are listed in Table 1.

Table 1. MP4100 method parameters

Element Wavelength (nm)	Neb Pressure (kPa)	EGCM Setting	Read Time (s)
Ca 393.366	220	High	3
Mg 280.271	220	High	3
P 214.915	120	Medium	3

Calibration

Multi-elemental standards were prepared at concentrations (w/w) of 1 ppm, 5 ppm and 10 ppm using Conostan S-21. Xylene was used as the diluent. All standards were matrix-matched with 50% (w/w) Blank Oil 75. The calibration fit and correlation coefficients for the elements analyzed are shown in Figures 1a, 1b and 1c.

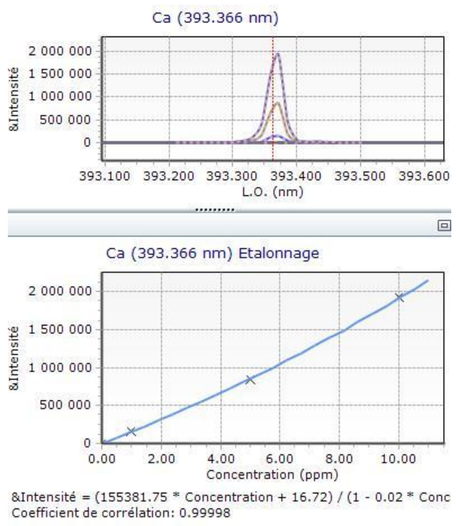

Figure 1a. Calibration curve for Ca 393.366 nm with a correlation coefficient of 0.99998

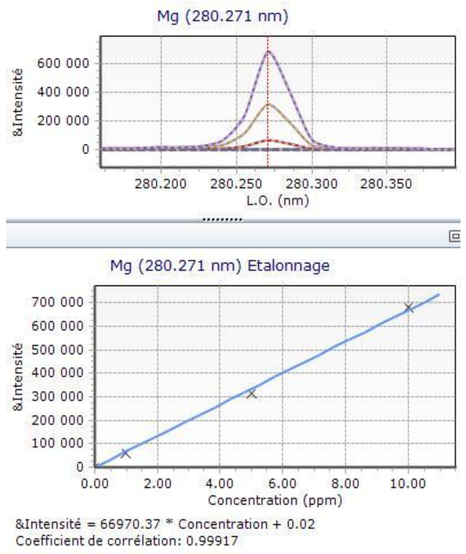

Figure 1b. Calibration curve for Mg 280.271 nm with a correlation coefficient of 0.99917

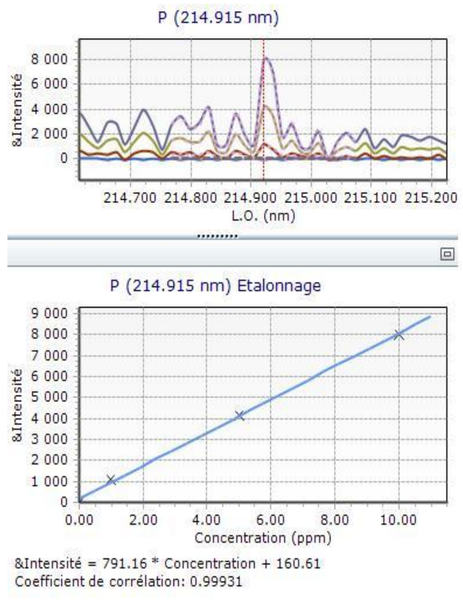

Figure 1c. Calibration curve for P 214.915 nm with a correlation coefficient of 0.99931

Results and Discussion

Recovery of unknown samples

A batch of unknown samples was analyzed using the 4100 MP-AES, and the results compared with the data obtained from analysis using the Agilent 725 ICP-OES. The comparison can be seen in Table 1.

Table 2. Ca, Mg, P measurements using the 4100 MP-AES and the 725 ICP-OES in soja oil samples.

	Ca (ppm)		Mg (ppm)		P (ppm)	
	MP	ICP	MP	ICP	MP	ICP
Soja 1h	15.3	14.5	0.22	0.32	0.12	0.15
Soja 13h	7.4	6.98	0.19	0.25	0.09	0.12

Precision

In order to test the precision of the 4100 MP-AES to analyze vegetable oil, one sample has been measured 25 times. The mean and standard deviation were calculated with precision > 95%. Shapiro Wilk and Dixon tests were applied and show a gaussian distribution for each element in Table 3.

Table 3. Statistical values calculated with 25 measurements of a sample.

Statistical Values	Ca	Mg	P
Mean	5.38	1.36	4.89
Standard deviation	0.124	0.036	0.171
Relative standard deviation %	2.312	2.659	3.491
Calculated critical value Shapiro Wilk	0.893	0.886	0.983
Theorical critical value Shapiro Wilk 95%, n=25	0.918	0.918	0.918
Lower Dixon value	0.103	0.182	0.185
Upper Dixon value	0.167	0	0.241
Table Dixon value at P=0.99	0.489	0.489	0.489

Long-term stability

Long term stability was investigated by continuously aspirating a standard over a 5 hour period. The resulting stability is shown below in Figure 2 with a standard deviation lower than 1%.

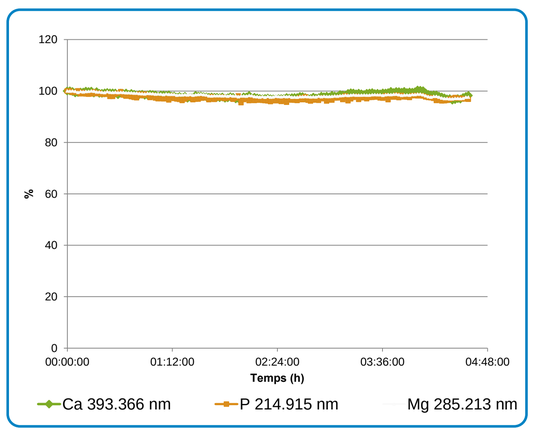

Figure 2. Normalized stability plot for a standard solution run repeatedly over a 5 hour period.

Conclusions

The 4100 MP-AES is able to perform fast multi-element organic analyses with the lowest running costs and high analytical performances. Moreover, the robustness of the plasma and the ease of use of the 4100 MP-AES make this technology best suited for routine analysis of complex matrices while ensuring maximum safety**.**

The 4100 MP-AES offers exceptional accuracy as demonstrated by the excellent agreement with results obtained on ICP-OES. Furthermore, the 4100 MP-AES has the lowest running costs of all of today's atomic spectroscopy techniques, due to reduced gas costs. Its use of non-combustible nitrogen.