Determination of major elements in fruit juices using the Agilent 4200 MP-AES with the Agilent 4107 Nitrogen Generator

Application note

Food testing

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Introduction

Major elements such as calcium, magnesium, sodium and potassium are essential nutrients in food and the routine monitoring of the levels of these elements in fruit juices is a common quality control process. Flame Atomic Absorption Spectroscopy (FAAS) is well suited to this application as it delivers the performance required for the analysis at a reasonable price. However, with the introduction of the Agilent Microwave Plasma-Atomic Emission Spectrometer (MP-AES), several of the analytical challenges of using FAAS for this application have been overcome, making it the ideal instrument for laboratories looking to transition away from FAAS to a more powerful and safer technique.

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The 4200 MP-AES main operating gas, nitrogen, is supplied from an Agilent 4107 Nitrogen Generator (with air supplied from an air compressor). This greatly reduces the running costs and eliminates the safety concerns associated with specialty gases required by FAAS such as acetylene and nitrous oxide. The nitrogen-based plasma source of the 4200 MP-AES operates at a higher temperature than the flame source of a FAAS, avoiding the chemical interferences present in FAAS (especially for elements such as Ca). This eliminates the time consuming, element specific sample preparation and burner head changeover that is required when analyzing Ca, Na, K and Mg in the same sample by FAAS. The analysis of this application by MP-AES also removes the need for costly and time consuming modifiers and ionization suppressants. The plasma source in the MP-AES also leads to improved performance with respect to detection limits and linear dynamic range when compared to FAAS, which is important in an analysis where the elements can be present over a wide range of concentrations. With no flammable gases required, the MP-AES is able to operate unattended which increases sample throughput.

This application note describes the analysis of fruit juice samples 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 and OneNeb nebulizer.

The instrument was controlled by the powerful and easy-to-use MP Expert software. The MP-AES features continuous wavelength coverage and MP Expert features an extensive wavelength database that allows the selection of wavelengths that are appropriate for the concentration range required for the analysis. For instance, in this application, the less sensitive Mg 518.360 nm line was preferred over the more sensitive Mg 285.213 line.

Table 1. Agilent 4200 MP-AES operating conditions

Parameter	Value			
Element	Ca	Mg	Na	K
Wavelength	422.673	518.360	589.592	769.897
Nebulizer	OneNeb			
Nebulizer flow rate	Default (0.75 L/min)			
Spray chamber	Double pass glass cyclonic			
Pump rate	15 rpm			
Sample pump tubing	Orange/green			
Waste pump tubing	Blue/blue			
Autosampler	Agilent SPS 3			
Read time	1 second			
Number of replicates	3			
Fast pump during uptake	On			
Sample uptake delay	30 seconds			
Rinse time	40 seconds			
Stabilization time	20 seconds			
Background correction	Auto			
Gas source	Agilent 4107 Nitrogen Generator			

Standard and sample preparation

Two quality control (QC) test materials were analyzed to validate the method:

- Apple Juice T1650QC (certified by FAPAS*)
- Grapefruit Juice T0842QC (certified by FAPAS*)

*FAPAS – The Food and Environmental Research Agency, York, UK.

Materials were purchased from Graham B Jackson (Aust) P/L.

Additionally, a commercially available apple juice was analyzed in the long term stability studies.

All fruit juice samples were diluted 20x with 5% HNO_3 (ACS Grade, Merck). No other modifiers or ionization suppressants were required.

Standards were prepared from a 10,000 mg/L multi element standard (Inorganic Ventures). All calibration blanks and standards were prepared in 5% HNO_3.

Results

Working range

The working concentration range of the standard solutions are summarized in Table 2. As the working range of MP-AES far exceeds that of FAAS (by up to 20 times in some instances), only one dilution of the sample is required to measure the complete set of elements.

Table 2. Working concentration range of the 4200 MP-AES standard solutions

Element	4200 MP-AES concentration range (mg/L)	Correlation coefficient
Ca 422.673	0–20	0.99990
Mg 518.360	0–100	0.99988
Na 589.592	0–20	0.99996
K 769.897	0–100	0.99968

Recoveries

Table 3 shows the concentration and recovery results of the four elements in the two fruit juices. The recovery results for Ca, Mg, Na, K in the fruit juices using this method were within +/- 10% of the assigned value. All results measured in this study were within the certified ranges of the two quality control test materials.

Table 3. Recovery results of 4 elements in the fruit juices using the 4200 MP-AES with the nitrogen generator

Apple Juice T0840QC	Certified value (mg/L)		Found (mg/L)	% Recovery
	Assigned value	Range		
Magnesium	49.0	40.3–57.8	49.9 ± 0.6	102
Sodium	21.2	16.9–25.4	22.2 ± 0.5	105
Potassium	1044	926–1161	1039 ± 29.7	100
Grapefruit Juice T0842QC	**Certified value (mg/L)**		**Found (mg/L)**	**% Recovery**
	Assigned value	**Range**		
Calcium	145.6	123.6–167.6	158.3 ± 3.2	109
Magnesium	92.5	77.5–107.4	91.1 ± 0.6	99
Potassium	1102	979–1225	1100 ± 14.7	100

Long term stability

A commercial apple juice solution (diluted 20x with 5% HNO_3) was repeatedly analyzed over a period of 6 hours. The resulting stability plot is shown in Figure 1. All elements have an RSD of less than 4% over 6 hours. With the OneNeb nebulizer and mass flow controlled nebulizer gas flow, excellent stability results were obtained for a sample with a complex high sugar matrix.

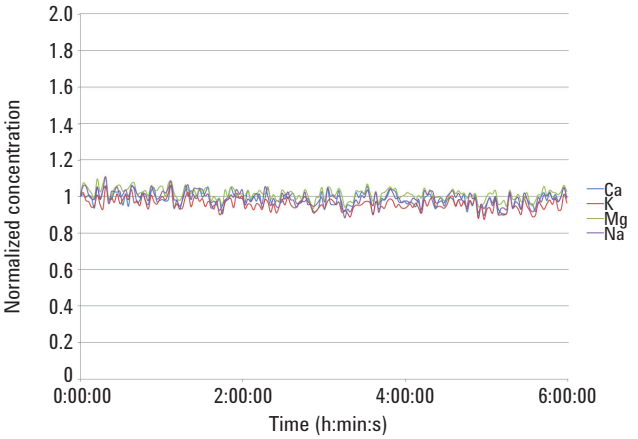
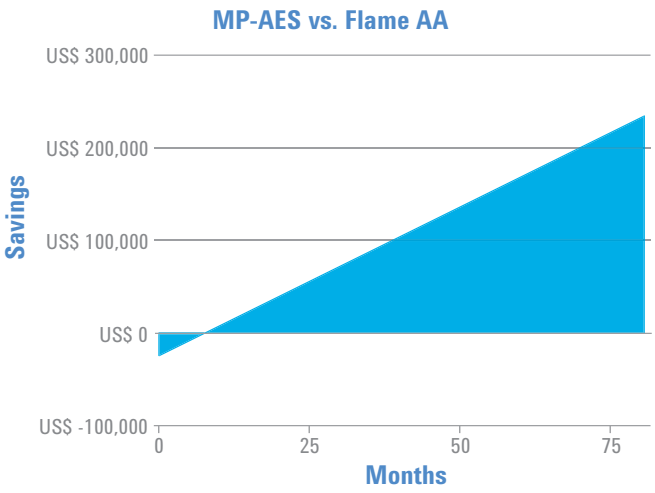


Figure 1. Normalized concentration of potassium in an apple juice sample over 6 hours

Cost savings with the 4200 MP-AES

The potential cost saving of using the 4200 MP-AES for this application was estimated by comparing an FAAS purchased with an air compressor and 1 year of consumables to an MP-AES purchased with air compressor, nitrogen generator, SPS 3, and 1 year of consumables. The analysis requirements were assumed to be 500 samples per week and 4 elements per sample. The calculation assumes that the FAAS is run without an autosampler and that 3 elements are analyzed with air/acetylene and 1 element with nitrous oxide/acetylene. In this example the results show an estimated cost saving of greater than US $220,000 over a 7 year evaluation period[1]. A global average gas cost was used in this calculation and results will vary from country to country.



[1]This example is intended to help you compare the running costs and savings of the MP-AES vs. flame AA. The applied formulas and parameters are correct to the best of our knowledge, but we cannot guarantee the results. Savings may vary depending on factors such as local gas and electricity costs, operator costs, number and types of elements. For this calculation operator labor costs were set to USD $25/hour and electricity costs were set to USD $0.18 per kW.

Conclusion

A simple and rapid method using MP-AES has been developed to analyze Ca, Mg, Na and K in fruit juice. The recoveries obtained from the analysis of the two QC test materials were within +/- 10% of the assigned values and within the certified concentration range. Using the standard sample introduction system supplied with the 4200 MP-AES, excellent long term stability was obtained over a 6 hour period.

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 through numerous ease-of-use features and simplified sample preparation, improved safety, and higher analytical performance such as better detection limits and greater linear dynamic range.

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