Total metals analysis of digested plant tissue using an Agilent 4200 Microwave Plasma-AES

Application note

Agriculture

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Introduction

Plant growth and development largely depends on the composition and concentration of mineral nutrients available in the plant leaves and other tissues. These essential nutrients are divided into macronutrients (required in larger quantities because of their structural roles in the plant) and micronutrients (required in smaller quantities because they tend to be involved in regulatory roles in the plant). A deficiency or enrichment of nutrients may result in decreased plant productivity, crop yield or plant quality.

Analysis of the total metal content in plants is often carried out by Flame Atomic Absorption Spectrometry (FAAS) or Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES). More recently, agricultural testing labs looking to upgrade or replace their FAAS with a more powerful technique are looking to Microwave Plasma–Atomic Emission Spectrometry (MP-AES) with its many advantages. MP-AES is a multi-element technique that offers better detection limits over a wider working analytical range than FAAS and more elements are available for analysis by MP-AES, including

phosphorus, an expensive and widely used major nutrient in soil fertilization.

For laboratories that have difficulty in sourcing gases, are looking to reduce operating costs or are under pressure to improve safety by removing flammable gases, the MP-AES is ideal as it uses nitrogen gas, that can be generated from air.

This application note describes the sample preparation procedure and analytical method used to determine Cu, Fe, Mn, Zn, Na, K, Ca, Mg, B and P in a plant reference material using the Agilent 4200 MP-AES.

Experimental

Instrumentation

All measurements were performed using an Agilent 4200 MP-AES with nitrogen plasma gas supplied via an Agilent 4107 Nitrogen Generator. The generator alleviates the need and expense of sourcing analytical grade gases. The sample introduction system comprised a double-pass cyclonic spray chamber and the OneNeb nebulizer.

An Agilent SPS 3 autosampler was used to deliver samples to the instrument, allowing the system to be operated unattended. The instrument operated in a fast sequential mode and featured a Peltier-cooled CCD detector. Background and spectral interferences could be simutaneously corrected easily and accurately using Agilent's MP Expert software. Method parameters are given in Table 1.

Table 1. MP-AES method parameters

Parameter	Value
Replicates	3
Pump rate	15 rpm
Sample uptake delay	35 seconds
Rinse time	30 seconds
Stabilization time	15 seconds
Fast Pump during uptake and rinse	On (80 rpm)
Autosampler	Agilent SPS 3
Sample pump tubing	Orange/green
Waste pump tubing	Blue/blue

Samples

Botanical reference material (RM) ASPAC 80 Pasture was obtained from the Australasian Soil and Plant Analysis Council (ASPAC, Carapook, VIC, Australia).

Sample preparation

Microwave digestion was used to prepare the ASPAC 80 RM for total metals analysis of Cu, Fe, Mn, Zn, Na, K, Ca, Mg, B and P by MP-AES. 7 mL of HNO_3 and 1 mL H_2O_2 was added to 0.18 g of the sample. A preloaded method for the MARS (CEM, Corporation, USA) microwave was used to digest the sample. Once cooled, the solution was diluted to 50 mL using ultrapure water. No further sample preparation was required and no modifiers or ionization buffers were added.

Wavelength selection and calibration range

Details of wavelength selection and calibration range are given in Table 2. Continuous wavelength coverage allows lines to be chosen that have appropriate sensitivity for the concentration range, and avoid spectral interferences.

Table 2. Wavelength and working calibration concentration range

Element and wavelength (nm)	Calibration range (ppm)
Cu 324.754	1–5
Fe 259.940	5–25
Mn 257.610	5–25
Zn 213.857	1–5
Na 568.820	2–100
K 766.491	1–100
Ca 445.478	20–100
Mg 383.829	1–100
B 249.772	0.25–1.0
P 213.618	10–80

Results and discussion

Calibration

A typical calibration curve for phosphorus is displayed in Figure 1. The curve shows excellent linearity across the concentration range. The large linear dynamic range means that less sample dilutions are needed which improves productivity and reduces the risk of sample contamination.

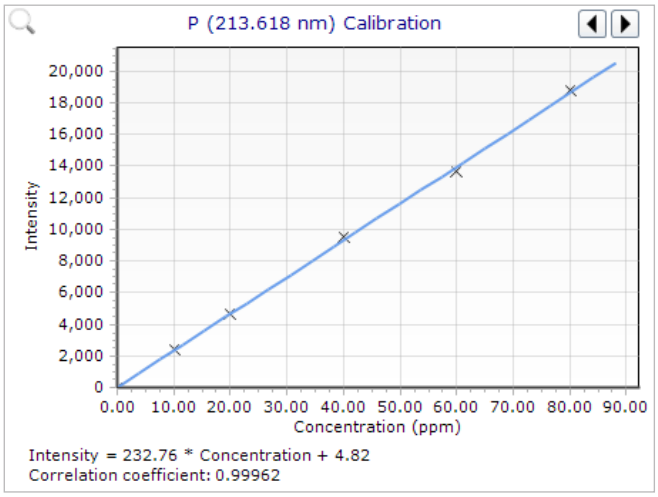

Figure 1. Calibration curve for phosphorus

Sample analysis

The plant RM sample was analyzed for all elements in a single measurement. The quality of the MP-AES results was evaluated by comparing them with the reference values for ASPAC 80. Table 3 shows good accuracy was achieved for all elements over a wide concentration range. The ability of the MP-AES to determine all elements in a single sample measurement greatly simplifies the workflow when compared to an FAAS, and eliminates the need for lamp changes, measurements in absorption and emission, and in the case of B and P, analysis of the samples by other techniques.

Table 3. MP-AES results for total metals content of plant reference material ASPAC 80.

Element	Wavelength nm	Measured value µg/g	Reference value µg/g	Accuracy %
Cu	324.754	13.6	14.7 ± 1.2	93
Fe	259.940	316.13	324 ± 32	98
Mn	257.610	125.8	138 ± 10	91
Zn	213.857	54.6	58.1 ± 5.3	94
Na	568.263	2512	2460 ± 210	102
K	766.491	27302	26700 ± 1850	102
Ca	445.478	10563	11100 ± 600	95
Mg	383.829	3239	3350 ± 220	97
B	249.772	21.65	23.7 ± 3.4	91
P	213.618	3223.35	2970 ± 250	109

Conclusions

The study shows the effectiveness of the Agilent 4200 MP-AES for the analysis of total metal content of a plant-based reference material following microwave digestion. Elements that are difficult to analyze by FAAS such as B and P were included, with all data acquired in a single run. Accurate determinations over a wide concentration range were obtained showing the suitability of MP-AES for the application. When compared to an FAAS, the workflow on the MP-AES is also simplified by eliminating the need for multiple sample preparations, lamp changes and measurements in absorption and emission modes.

Current trends in the market for lower detection limits, lower cost of analysis, improved ease of use and improved safety, are all met by the Agilent 4200 MP-AES. The instrument uses nitrogen, eliminating expensive and hazardous gases such as acetylene, increasing safety, and allowing for unattended operation of the instrument, even in remote locations. When the N_2 is supplied using the Agilent 4107 Nitrogen Generator that extracts N_2 from air, running costs are greatly reduced compared to an FAAS or ICP-OES that rely on a constant supply of analytical grade gases.

With greater sensitivity, linear dynamic range, and sample throughput compared to FAAS, the Agilent 4200 MP-AES is the ideal replacement for labs looking to extend their analytical capabilities.

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