Plant nutrient analysis using the Agilent 5100 Synchronous Vertical Dual View ICP OES

Application note

Food testing & agriculture

Authors

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Introduction

The determination of the elemental composition in plants is important for development, growth and maintenance of plant tissues. Elements, such as Al, B, Ba, Ca, Cu, Fe, K, Mg, Mn, S, Sr, P, and Zn, are important for plant nutrition, being vital nutrients required for tissue development, maintenance and plant metabolism [1]. The determination of macro, micronutrients and contaminants in plant samples is important to keep up with sources of nutrients and minerals. The chemical analysis of plant materials can be applied to assist in the remediation of contaminated soils or to solve mineral malnutrition, a problem that seriously affects the human population [2, 3]. Inductively coupled plasma optical emission spectrometry (ICP-OES) is an attractive technique for this analysis because it can accommodate the wide concentration ranges typical of macro and micronutrients in plants.

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Agronomical laboratories typically deal with large batches of samples. Several critical elements, in wide concentration ranges, must be determined on a routine basis for such samples. The Agilent 5100 Synchronous Vertical Dual View (SVDV) ICP-OES with Dichroic Spectral Combiner (DSC) technology, has the ability to keep up with these demands, performing axial and radial measurements in a single reading, leading to faster sample throughput times. With faster sample run times, the 5100 SVDV requires less argon per sample, meaning significant savings can be made for labs involved in high throughput analysis. The Vista Chip II detector used in the 5100 ICP-OES has the fastest processing speed (1 MHz) of any charge coupled device (CCD) detector used in ICP-OES. It delivers fast warm-up, high throughput, high sensitivity, and the largest dynamic range.

This application note describes the quantitation of Al, B, Ba, Ca, Cu, Fe, K, Mg, Mn, P, S, Sr and Zn in microwave acid digested alfalfa, corn and sugarcane samples and an apple leaves certified reference material (SRM NIST 1515), using the Agilent 5100 Synchronous Vertical Dual View (SVDV) ICP-OES.

Experimental

Instrumentation
All measurements were carried out using an Agilent 5100 SVDV ICP-OES with Dichroic Spectral Combiner (DSC) technology (Agilent Technologies) which offers simultaneous axial and radial view analysis. The sample introduction system consisted of a SeaSpray nebulizer, single-pass cyclonic spraychamber and a 1.8 mm DV i.d injector torch. Method and instrument operating conditions are presented in Table 1.

Table 1. Agilent 5100 SVDV ICP-OES method and instrument operation parameters

Parameter	Setting
Read time (s)	20
Replicates	3
Sample uptake delay (s)	15
Stabilization time (s)	15
Pump speed (rpm)	12
Sample tubing	White/White
Waste tubing	Blue/Blue
Background correction	Fitted
RF power (kW)	1.5
Aux flow (L/min)	1.0
Plasma flow (L/min)	12.0
Neb Flow (L/min)	0.60
Nebulizer	SeaSpray®
Viewing height (mm)	8
Spray chamber	Single-pass cyclonic

Standard and sample preparation
All glassware was decontaminated by immersion in 10% v/v HNO_3 for at least 24 h and then rinsed with distilled-deionized water (resistivity > 18.2 MΩ cm), obtained from a Milli-Q® Water System (Millipore, Billerica, MA, USA). All solutions and blanks were prepared with ultrapure water and nitric acid obtained from a sub-boiling distillation apparatus (Milestone). Working standards were prepared from 1000 ppm single element stock solutions of Al, B, Ba, Ca, Cu, Fe, K, Mg, Mn, P, S, Sr and Zn at concentrations of 0, 1, 5, 10, 20, 40, 60 and 100 ppm (Qhemis, São Paulo, SP, Brazil).

Embrapa Southeast Livestock (São Carlos, SP, Brazil) provided samples of corn roots, corn and alfalfa leaves. The two alfalfa samples were grown in soils previously fertilized with agricultural gypsum. Leaves of sugarcane were provided by the Sugar Cane Technology Center (Piracicaba, SP, Brazil). A Certified Reference Material (CRM) (SRM 1515, apple leaves) was obtained from the National Institute of Standards and Technology (NIST, Gaithersburg, MD, USA). All samples were microwave digested in triplicate. Approximately 200 mg of samples was digested with 8 mL of HNO_3 50% (v/v). Post digestion, distilled-deionized water and 2 mL H_2O_2 30% (v/v) was added to make up the final volume to 50 mL.

Results and discussion

Method Detection Limits and CRM recoveries
The performance of SVDV mode was assessed by calculating the Method Detection Limits (MDLs), considering background equivalent concentrations (BEC) and relative standard deviations (RSD) for 10 consecutive measurements of the digestion blanks (Table 2).

It is worth noting that low MDL values were obtained for important primary nutrients, such as P and K, secondary nutrients, such as Ca, Mg and S, and also for micronutrients, such as B, Cu, Fe, Mn and Zn.

Table 2. Method detection limit (MDL) and background equivalent concentrations (BEC) for Al, B, Ba, Ca, Cu, Fe, K, Mg, Mn, P, S, Sr and Zn using 5100 ICP-OES in SVDV mode.

Element and λ(nm)	MDL (µg/g)	BEC (µg/L)
Al I (396.152)	0.07	0.18
B I (249.772)	0.64	36.2
Ba II (455.673)	0.62	0.85
Ca I (422.673)	0.09	0.41
Cu I (324.754)	1.75	11.8
Fe II (238.204)	5.85	11.0
K I (766.491)	0.03	0.51
Mg I (285.213)	0.01	0.01
Mn II (257.610)	2.98	1.30
P I (178.222)	0.03	0.05
S II (181.972)	2.32	0.006
Sr I (460.733)	1.18	3.57
Zn I(213.857)	1.40	4.26

To verify the accuracy of the developed method, Al, B, Ba, Ca, Cu, Fe, K, Mg, Mn, P, S, Sr and Zn concentrations were determined in the apple leaves certified reference material (SRM NIST 1515, Table 3). Recoveries ranged from 94.2 to 108.6% for SVDV mode, all were within ±10 % of the target value. The excellent recoveries demonstrate the ability of the 5100 SVDV ICP-OES to accurately determine macro, micronutrients and contaminants in plant leaves.

SVDV mode collects data simultaneously for both axial and radial views. It does this in the same time it takes to measure only one viewing mode. The SVDV view mode was chosen to determine the analytes mentioned above in plant sample digests (see Table 4), in which low standard deviation values were achieved.

Table 3. Determination of Al, B, Ba, Ca, Cu, Fe, K, Mg, Mn, P, S, Sr and Zn (mean ± std. deviation × $n^{-0.5}$, n = 3) and analyte recoveries in apple leaves certified reference material (SRM NIST 1515) using 5100 ICP-OES in SVDV mode.

Analyte	Certified value (µg/g)	Found (µg/g)	Recovery (%)
Al	286.00±9.00	277.0±0.01	96.9±0.3
B	27.00±2.00	27.8±0.16	102.9±0.59
Ba	49.00±2.00	46.1±0.5	94.2±0.6
Ca	15260±0.015	16290±0.015	106.8±0.13
Cu	5.64±0.24	5.80±0.058	102.9±1.02
Fe	83.00±5.00	83.4±2.32	100.4±2.80
K	16100±0.02	15323±0.09	95.2±0.3
Mg	2710±0.008	2591±0.02	95.6±0.5
Mn	54.00±3.00	52.9±1.21	98±2.24
P	1.59±0.011	1.62±0.02	101.9±0.06
S	1800**	1703±0.01	94.6±0.4
Sr	25.00±2.00	27.2±0.2	108.8±0.96
Zn	12.50±0.30	12.52±0.36	100.2±2.86

**Non-certified concentration

Calibration Linearity
Figure 1 shows the calibration curves for Al, B, P and S, with calibration coefficients greater than 0.999 with less than 10% calibration error on each calibration point.

Table 4. Determination of Al, B, Ba, Ca, Cu, Fe, K, Mg, Mn, P, S, Sr and Zn (mean ± standard deviation, n = 3) in plant samples by ICP OES with SVDV mode.

| Sample	Determined concentrations (µg/g)						
	Al	**B**	**Ba**	**Ca**	**Cu**	**Fe**	**K**
Sugarcane leaves	230.0±41.9	4.54±0.10	31.7±0.10	5009±103	5.27±0.1	163.2±8.76	11821±8.60
Corn leaves	10.2±0.10	33.1±0.20	0.17±0.004	3313±188	2.91±0.1	49.93±1.22	25828±652.2
Corn roots	10.1±0.60	14.05±0.50	0.25±0.01	4525±108	2.70±0.1	527.3±55.5	30708±348.5
Alfalfa leaves (1)	292.0±30.2	27.52±0.31	58.9±0.70	13252±41.21	7.28±0.14	190.5±20.0	25948±192.7
Alfalfa leaves (2)	1154±3.39	33.61±0.31	31.9±0.20	10926±151.5	7.07±0.02	516.2±3.18	28466±223.4
	Determined concentrations (µg/g)						
	Mg	**Mn**	**P**	**S**	**Sr**	**Zn**	
Sugarcane leaves	2844±6.57	69.76±3.80	1482±28.7	1617±3.46	31.87±0.43	19.04±0.26	
Corn leaves	1313±46.7	30.75±0.90	4056±95.1	1228±57.2	4.43±0.04	12.37±0.45	
Corn roots	2023±28.0	17.06±1.33	3984±76.5	2150±56.0	0.81±0.06	17.12±1.76	
Alfalfa leaves (1)	1661±4.27	20.28±0.33	3941±81.1	4188±43.4	248.3±2.30	21.80±0.14	
Alfalfa leaves (2)	2368±30.8	34.45±0.23	3290±16.0	3544±39.6	67.39±0.92	17.99±0.19	
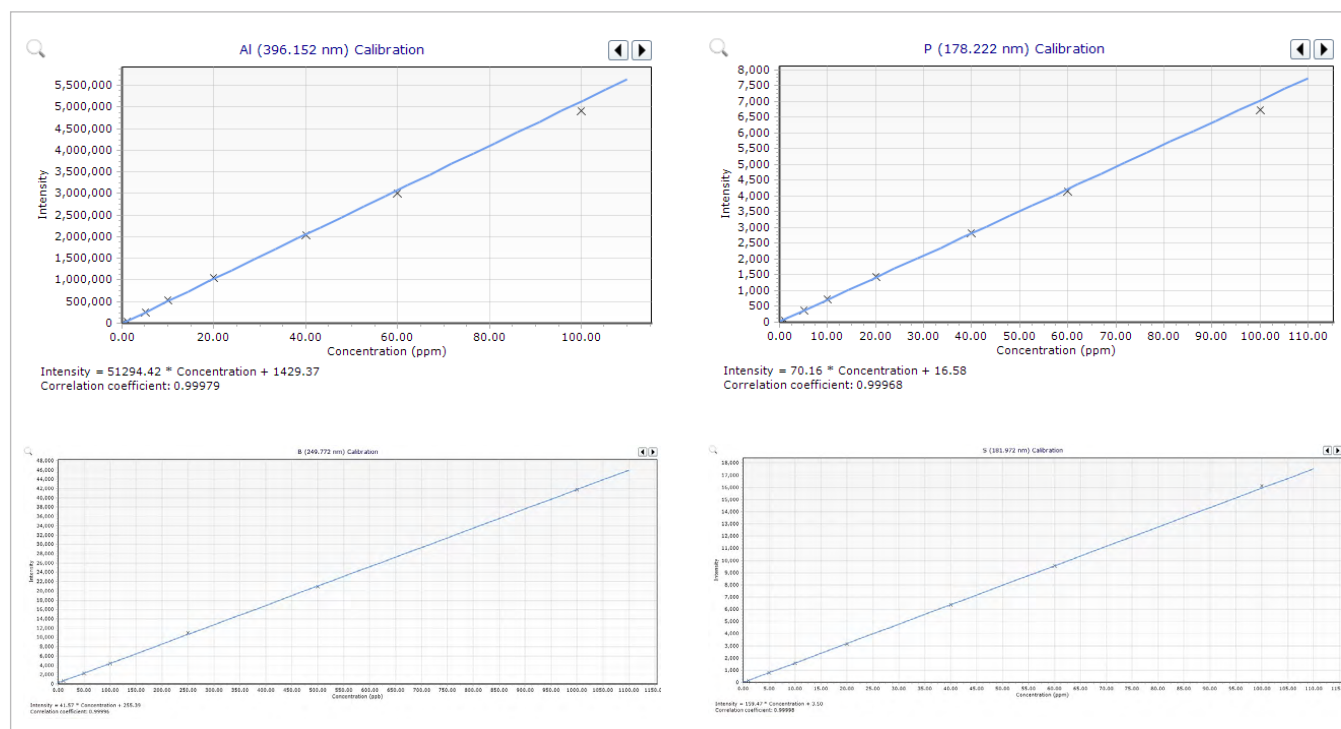
Figure 1. Calibrations obtained for Al, B, P and S using the 5100 SVDV ICP-OES.

Conclusions

This application note demonstrates the performance and suitability of the Agilent 5100 Synchronous Vertical Dual View (SVDV) ICP-OES for plant nutrient analysis. With its unique configuration and Vista Chip II detector, all wavelengths could be read in a single measurement over a wide concentration range. The 5100 SVDV ICP-OES produced good linearity and excellent method detection limits were obtained for both macro-nutrients, P, K, Ca, Mg and S, and micronutrients, B, Cu, Fe, Mn and Zn. The recoveries obtained for the certified reference material were within ±10% of the target value, demonstrating the accuracy of the developed method for SVDV mode. With successful analyte determinations in sample digests, using SVDV mode, low standard deviations values were obtained.

The Agilent 5100 SVDV ICP-OES offers suitable performance and flexibility to support the high throughput demands of agronomical labs. Measurements in SVDV mode take less time and use less argon gas per sample, due to the simultaneous data acquisition of both axial and radial views.

Acknowledgments

References

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