Анализ следовых элементов в удобрениях на приборе Avio 200 ICP-OES

Производство удобрений является ключевой отраслью для сельского хозяйства страны, главным преимуществом является повышение урожайности сельскохозяйственных культур. Основные элементы в удобрениях разделены на четыре класса: первичные питательные вещества, вторичные питательные вещества, питательные микроэлементы и следовые элементы.
В настоящее время химические удобрения широко применяются в сельском хозяйстве, что привело к росту спроса, но этот спрос также привел к тому, что некоторые удобрения производятся с плохим качеством.
Анализ и мониторинг первичных и вторичных питательных веществ, а также следовых элементов, необходим для обеспечения соответствия качества удобрений местным нормам. Хотя для измерения следовых элементов в удобрениях могут использоваться различные методы, ICP-OES предлагает наилучшее сочетание стоимости, простоты, надежности и точности, а также имеет динамический диапазон, подходящий для концентраций, которые ожидаются в образцах удобрения.

Эта работа демонстрирует способность прибора Avio® 200 ICP-OES соответствовать нормативным ограничениям для следовых элементов в удобрениях путем анализа стандартных образцов.

ССЫЛКА НА ИСТОЧНИК:

https://www.perkinelmer.com/lab-solutions/resources/docs/APP_013273_01-Avio-200-ICP-OES-Analysis-of-Trace-Elements-in-Fertilizer-Application-Note.pdf

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APPLICATION NOTE

ICP-Optical Emission Spectroscopy

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Analysis of Trace Elements in Fertilizer with Avio 200 ICP-OES

Introduction

The production of fertilizer is a key industry for a country's agricultural sector, with the main benefit of increasing crop productivity. The primary elements of interest in fertilizers are categorized into four classes: primary nutrients, secondary nutrients, micronutrients, and trace elements; Table 1 shows representative elements in each of these classes.

Currently chemical fertilizers are widely used in agriculture, which has led to increased demand, but this demand has also resulted in the production of some fertilizers of inferior quality. The analysis and monitoring of the primary and secondary nutrients, as well as trace elements, is necessary to ensure that the quality of fertilizers is compliant with local regulations. Table 2 lists the Thailand Agricultural Department regulatory levels of certain elements in fertilizers. Although a variety of techniques may be used for the measurement of trace elements in fertilizer, ICP-OES offers the best combination of cost, simplicity, ruggedness and accuracy, and also has a dynamic range which is appropriate for the concentrations expected in fertilizer samples.

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Table 1. Elemental Categories in Fertilizer.

Class	Elements
Primary Nutrients	N, P, K
Secondary Nutrients	Ca, Mg, Fe, Mn, Cu, Zn, Mo, B, S
Micronutrients	Al, Co, V, Se, Ni
Trace Elements	As, Cd, Cr, Pb

Table 2. Regulatory Limits of Elements in Fertilizers in Thailand.

Element	Regulatory Concentration (mg/kg)
As	50
Cd	5
Cr	300
Pb	500

This work demonstrates the ability of the Avio® 200 ICP-OES to meet Thai regulatory limits for trace elements in fertilizers through the analysis of reference materials.

Experimental Conditions

Samples and Sample Preparation

Two reference materials were used to validate the method: NIST 695 Trace Elements in Multi-Nutrient Fertilizer and BCR-032 Trace Elements in Moroccan Phosphate Rock. The second reference material was chosen because its high phosphate concentration is similar to the elevated phosphorus concentrations commonly found in fertilizers.

All samples were prepared by microwave digestion using the Titan® MPS Microwave digestion system. To each vessel, 0.15 g of sample was added, followed by 6 mL concentrated nitric acid and 2 mL concentrated hydrochloric acid. The vessels were allowed to sit uncapped for 10 minutes to allow any early reactions to occur safely. The vessels were then capped, placed in the Titan, and digested using the program in Table 3. After digestion, the samples were transferred to 100 mL volumetric flasks and diluted to 100 mL with deionized water for analysis.

Instrumental Parameters

All samples were analyzed with the Avio 200 using the parameters in Table 4, which includes the standard sample introduction system. The ability to use a total of only 9 L/min of argon is the result of Avio's Flat Plate™ plasma technology, which, in combination with the vertical torch, enhances the ability to analyze high-matrix samples. The analytes and wavelengths used are shown Table 5. All quantitative measurements were made against external calibration curves constructed from 0.05, 0.1, 1, 2, and 5 ppm standards.

Table 3. Titan MPS Temperature program.

Temperature Program					
Step	Target Temp (°C)	Pressure Limit (bar)	Ramp Time (min)	Hold Time (min)	Power (%)
1	160	30	5	5	90
2	180	35	2	30	100
3	50	35	1	15	0
4	-	-	-	-	-

Table 4. Instrumental parameters and sample introduction components for Avio 200 ICP-OES.

Parameter	Value
Nebulizer/spray chamber	Meinhard/Cyclonic
Injector	Alumina Injector 2.0
Read Time	1 sec (min) – 2 sec (max)
Plasma Gas	8 L/min
Auxiliary Gas	0.2 L/min
Nebulizer Gas	0.7 L/min
Power	1400 W
Plasma View	Axial

Table 5. Elements and Wavelengths.

Elements	Wavelength (nm)	Plasma View
Arsenic (As)	188.979	Axial
Lead (Pb)	220.353	Axial
Cobalt (Co)	228.616	Axial
Cadmium (Cd)	228.802	Axial
Nickel (Ni)	231.604	Axial
Chromium (Cr)	267.716	Axial

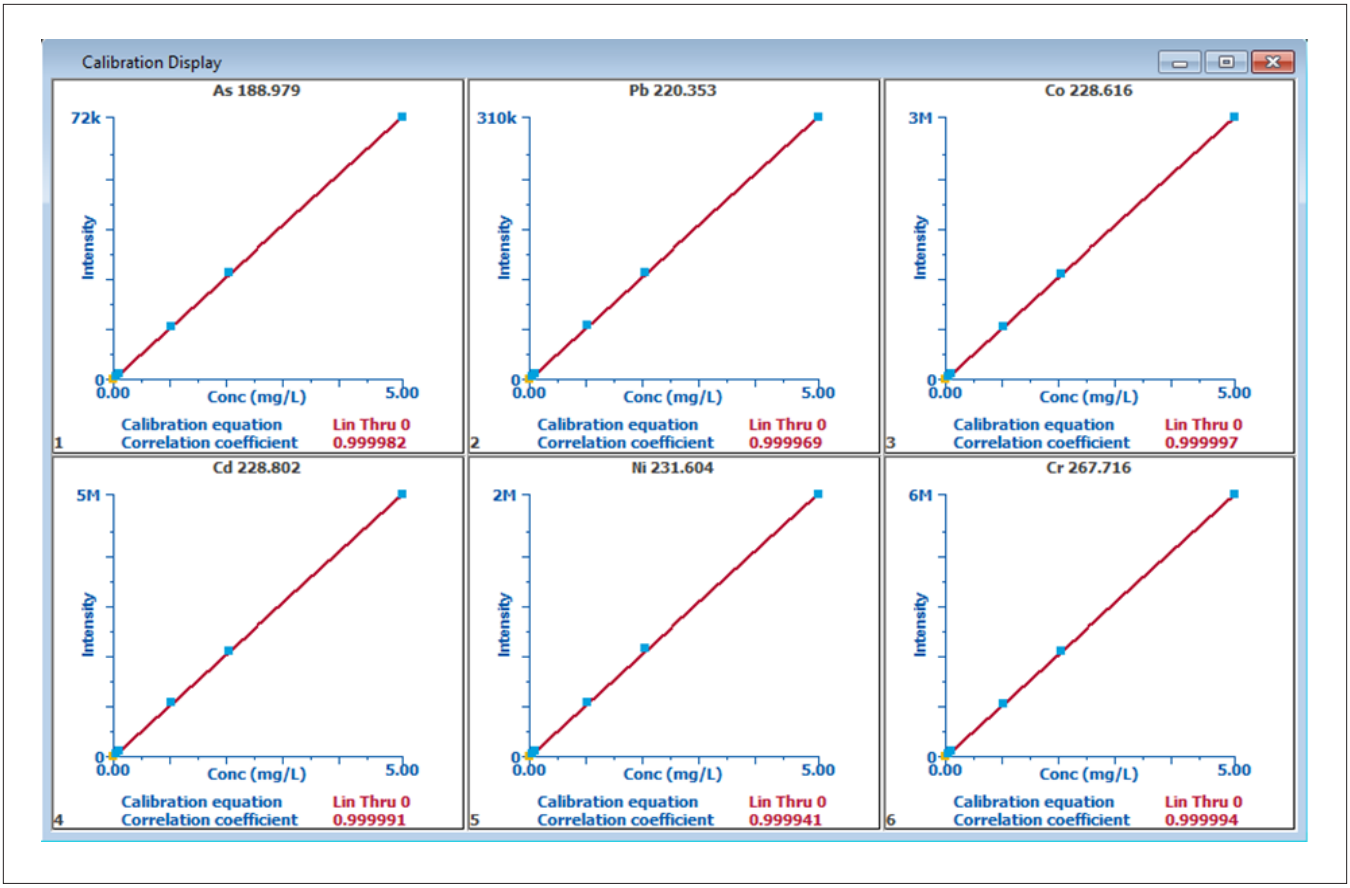

Figure 1. Calibration curve for all elements.

Results

The method accuracy was determined by measuring the reference materials. First, calibration curves were established and are shown in Figure 1, all with correlation coefficients greater than 0.9999. The results for the reference materials appear in Tables 6 and 7 for NIST 695 and BCR-032, respectively, and demonstrate the accuracy of the methodology with all recoveries being within 10% of the certified values.

The Detection Limits (DLs) of this methodology were determined by analyzing a matrix blank, which consisted of the same reagents and quantities as those used for sample preparation. The DLs were determined by multiplying the standard deviation of ten replicate measurements by three and are shown in Table 8.

Table 6. Result of SRM 695.

Analyte	Certified Values (mg/kg)	AVIO 200 Values (mg/kg)	% Recovery
As	200	211	107
Pb	273	295	108
Co	65.3	62.9	96
Cd	16.9	18.0	107
Ni	135	139	103
Cr	244	243	100

Table 7. Result CRM BCR -032.

Analyte	Certified Values (mg/kg)	Avio 200 Values (mg/kg)	% Recovery
As	9.5	< DL	-
Pb	5.4	<DL	-
Co	0.59	0.613	104
Cd	20.8	21.8	105
Ni	34.6	35.0	101
Cr	257	261	102

Table 8. Detection Limits in Fertilizer Samples.

Analyte	DLs (mg/kg)	Thai Regulated Levels (mg/kg)
As	3.01	50
Pb	3.37	500
Co	0.31	Not specified
Cd	0.21	5
Ni	0.59	Not specified
Cr	0.79	300

Conclusion

This work has demonstrated the ability of the Avio 200 ICP-OES to accurately measure secondary nutrients and trace elements in fertilizers at or below the regulated levels in Thailand.

Consumables Used

Avio 200 ICP-OES	
Component	Part Number
Red-Red PVC Pump Tubing	09908585
Black-Black PVC Pump Tubing	09908587
Autosampler Tubes	B0193233 (15 mL) B0193234 (50 mL)
PerkinElmer Pure XVI (QC 21 Elements)	N93002812 (125 mL)

Titan MPS Microwave	
Component	Part Number
Consumable Kit for Standard 75 mL Digestion Vessels	N3132000
Rupture Disks for Standard 75 mL Digestion Vessels (25 pieces)	N3132001
Pressure Seal for Standard 75 mL Digestion Vessels (10 pieces)	N3132002
End Cap Plug for Gas Containment Manifold	N3134004
Single Lip Seal Forming Tool for Standard 75 mL Digestion Vessels	N3132015
8-Position Lip Seal Forming Tool for Standard 75 mL Digestion Vessels	N3132014

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