APPLICATION NOTE

ICP-Optical Emission Spectroscopy

AUTHORS

Mohd Kamaruddin Jaffar

Nur Syafiqah Mohamad Sa'adan

Johor Plantations Group Berhad
R&D and Agricultural Services
Central Analytical Laboratory

Johor, Malaysia

Determination of Available Phosphorus in Soil Samples Using the Avio 550 Max ICP-OES

Introduction

Phosphorus (P) is a crucial nutrient for plants, and its scarcity in soils significantly constrains crop productivity. However, a surplus of total phosphorus in a specific soil type does not guarantee an abundance of available phosphorus, as the nutrient might be inaccessible. Therefore, assessing available phosphorus represents a vital agronomic parameter to ascertain its accessibility for plant absorption.[1]

To assess the available phosphorus in soils, laboratories frequently employ a UV/Vis spectrophotometric method, often referred to as the UV/Vis method. However, this UV/Vis method is recognized as a challenging technique for assessing available phosphorus since it involves several intricate steps, especially when working with samples that produce colored solutions during the preparation process. Such samples contain interfering species that absorb light within the same wavelength range as phosphorus. In such cases, the measured absorbance is inaccurate or unreliable, potentially leading to incorrect results.

The UV/Vis method for detecting available phosphorus is based on the determination of the blue phosphate-molybdate complex, where the intensity of the blue color is directly proportional to the phosphate-molybdate's concentration in solution. Some soil samples (such as peat) are dark-colored or rich in organic matter, which can interfere with the absorption of the phosphate-molybdate complex. Consequently, the UV/Vis method may not be able to distinguish between phosphorus and the soil itself, leading to inaccurate results.

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As an alternative, ICP-OES relies on the excitation of atoms in a sample using a high-temperature inductively coupled plasma (ICP) as the excitation source and is not affected by the color of the prepared solution. The emitted light (or optical emission) from these excited atoms is then measured to determine the elemental composition of the sample.

The Avio® 550 Max fully simultaneous ICP-OES is the ideal solution for this application. It is equipped with a unique optical system and SCD detector[2], delivering exceptional detection limits. Its dual-view plasma design[3] allows for the simultaneous analysis of trace and major elements within a single method. Furthermore, the Avio 550 Max ICP-OES offers the advantages of rapid start-up and cost-efficiency through the utilization of Flat Plate™ plasma technology.[4] Combining the Avio 550 Max ICP-OES with an autosampler enhances the efficiency of the measurement process.

Experimental

Sample Preparation

An in-house control mineral soil sample (control sample) and eight proficiency test samples from the Agricultural Laboratory Association Malaysia (AgLAM) were analyzed. The control sample was prepared by first air-drying and then crushing and passing through a 2 mm sieve. The sample then went through homogeneity and stability testing to ensure it was uniform with no significant variation in the properties or characteristics.

A 2 g quantity of dried soil sample was weighed into a suitable container, to which 20 mL of Bray No. 1 Extraction Solution (a mixture of 1N ammonium fluoride and 1N hydrochloric acid) was added. The mixture was then shaken by hand for 1 minute, and the solution was then filtered into a 50 mL autosampler tube.

Calibration standards were prepared from a 1000 mg/L phosphorus standard at 5, 25, and 50 mg/L; QC solutions were prepared at 15 and 25 mg/L. All measurements were made against an external calibration curve.

Instrumental Parameters and Conditions

All measurements were performed using an Avio 550 Max ICP-OES using the instrument conditions in Table 1. Radial viewing was chosen for higher sensitivity, and a SeaSpray® nebulizer was utilized due to its superior performance with soil extraction samples. The RF power was set to 1400 watts, and the plasma and nebulizer gas flows were set to 11 and 0.50 L/min, respectively, allowing all analyses to be completed with a total argon consumption of less than 12 L/min, a direct result of the Avio 550 Max's unique Flat Plate plasma technology.

Table 1. Avio 550 Max ICP-OES Instrumental Conditions.

Parameter	Value
Nebulizer	SeaSpray
Spray Chamber	Baffled Glass Cyclonic
Torch	1-Slot Quartz
Injector	2.0 mm i.d. Alumina
RF Power	1400 W
Plasma Gas Flow	11 L/min
Auxiliary Gas Flow	0.20 L/min
Nebulizer Gas Flow	0.50 L/min
Phosphorus Wavelength	213.617 nm
Plasma View	Radial
Read Delay	35 sec
Replicates	3
Sample Uptake Rate	1.50 mL/min

Results and Discussion

The variety of wavelengths available on the Avio 550 Max ICP-OES enables the optimum wavelength to be chosen for an analysis. In this work, P 213.617 nm was used for available phosphorus determination. The calibration plot in Figure 1 shows excellent linearity (correlation coefficients > 0.999), demonstrating the ability to measure P 213.617 nm accurately and consistently.

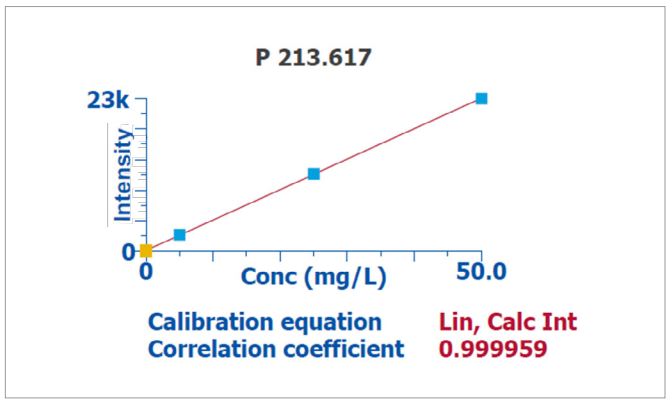

Figure 1. Calibration plot for phosphorus.

Precision and Repeatability

The precision between the UV/Vis and the ICP-OES methods was evaluated using the relative standard deviation (variance). Fifteen replicate measurements of the control sample were tested concurrently on both instruments, and the descriptive statistics were analyzed.

The variance obtained was 7.037% and 6.403% for both instruments, respectively, as shown in Table 2. According to the IUPAC Harmonized Guidelines for Single - Laboratory Validation of Method of Analysis[5] (2002), a variance of less than 10% indicates that the variability of the data is relatively low, indicating that both ICP-OES and UV/Vis yield equivalent results.

Table 2. Descriptive Statistics Comparing UV/Vis and ICP-OES Results.

Variable	Number of Measurements	Mean	Std. Error Mean	Standard Deviation	Variance
UV/Vis	15	32.767	0.685	2.653	7.037
ICP-OES	15	32.436	0.653	2.531	6.403

Next, a t-test was used to compare the means of the two groups of data to determine whether the difference between the means of the two groups is statistically significant. The data for both methods were analyzed using a t-test at a 95% confidence level, which shows that the t-statistic (the p-value) is 0.729. A p-value greater than 0.05 indicates that there is no significant difference between the data, further confirming that the ICP-OES produces equivalent results to the UV/Vis method.

Accuracy

The z-score is a measure that quantifies the proximity of a value to the average of a dataset. AgLAM's Proficiency Testing (AgLAM PT) encompasses 45 laboratories located across Malaysia. For the year 2021, eight samples from AgLAM's PT were meticulously examined to assess their accuracy. The analysis of these eight samples was carried out employing the ICP-OES method, yielding commendable results. Table 3 provides information on both the mean values and the z-scores for each of the AgLAM PT samples.

Table 3. Z Scores of Avio 500/550 Max Analyses of Eight Proficiency Samples.

Sample	Avio 550 Max Results (mg/kg)	AgLAM PT Means (mg/kg)	Z-Score	Satisfactory
S01-21	8.70	13.2	-0.80	Yes
S02-21	7.30	13.4	-1.33	Yes
S03-21	15.8	35.6	-1.47	Yes
S04-21	9.00	10.3	-0.43	Yes
S05-21	6.30	8.57	-0.63	Yes
S06-21	6.90	10.5	-0.97	Yes
S07-21	6.00	11.0	-1.12	Yes
S08-21	2.80	4.46	-0.71	Yes

Conclusion

This work demonstrates the ability of ICP-OES to determine the available phosphorus in soil samples using the Avio 550 Max fully simultaneous system. The ICP-OES method is comparable to the established UV/Vis method, with the advantage of offering multi-element capability to measure other elements simultaneously, thus increasing the laboratory productivity and capacity. Combined with Syngistix™ for ICP smart software which enhances the user experience, the multi-elemental capability of the Avio 550 Max provides a significant return on investment, especially when compared to dedicated phosphorus analyzers.

References

1. Ziadi, N., Whalen, J.K., Messiga, A.J., Morel, C., ***Advances in Agronomy,*** 2013, 122, 85-126.
2. "Avio 550/560 Max ICP-OES Optical System and SCD Detector", Technical Note, PerkinElmer, 2020.
3. "Vertical Dual View on the Avio Max Series ICP-OES", Technical Note, PerkinElmer, 2020.
4. "Flat Plate Plasma Technology on the Avio Max Series ICP-OES", Technical Note, PerkinElmer, 2020.
5. Harmonized Guidelines for Single-Laboratory Validation of Methods of Analysis. Pure Appl. Chem., Vol. 74, No. 5, pp. 835-855, 2002, IUPAC.

Consumables Used

Component / Description	Part Number
SeaSpray Nebulizer, 2 mL/min	N0775345
Single-Element Standard: Phosphorus, 1000 mg/L	N9303788 (125 mL) N9300139 (500 mL)
Sample Uptake Tubing, Black/Black (0.76 mm i.d.), PVC, Flared	N0777043
Drain Tubing, Red/Red (1.14 mm i.d.), PVC	09908585
Autosampler Tubes	B0193233 (15 mL) B0193234 (50 mL)

PerkinElmer U.S. LLC
710 Bridgeport Ave.
Shelton, CT 06484-4794 USA
(+1) 855-726-9377
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