Determination of Major Elements in Fertilizer Using the Teledyne Leeman Labs Dual-View Prodigy Plus ICP-OES

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Introduction

Global fertilizer consumption has increased substantially since 1950, while the world's population has grown from 2.5 to 7.7 billion. Because only approximately 3.5% of the earth's surface is suitable for agriculture, it is necessary to protect the fertility of the soil. The nutrients nitrogen (N), phosphorus (P) and potassium (K) in fertilizer are essential for healthy plants and larger crop yields. Presently, worldwide consumption of NPK fertilizer is approximately 200 million tons.

Fertilizer manufacturers and consumers require confidence in fertilizer elemental composition for both economic and environmental reasons. Manufacturers require strict quality control to maintain product performance, control costs and ensure fair market value. Consumers need to ensure that the composition of the product meets their needs and that the use of the product is consistent with specific crop and soil conditions. From an environmental perspective, awareness of a fertilizer's composition can reduce overuse, which has become an increasing concern. For example, nitrate can leach from the soil into ground water and phosphorus has been associated with eutrophication of bodies of water.

ICP-OES is a time-tested method of determining fertilizer elemental composition. This application note demonstrates the determination of major elements in fertilizer using a Teledyne Leeman Labs dual-view Prodigy Plus High Dispersion ICP.

Instrumentation

A Prodigy Plus High Dispersion ICP-OES system equipped with a dual-view torch and 120-position CETAC ASX-280 autosampler (Figure 1) was used to generate the data for this application note.

Figure 1 Prodigy Plus ICP-OES and ASX-280 Autosampler

The Prodigy Plus is a compact benchtop simultaneous ICP-OES system featuring an 800 mm focal length Echelle optical system coupled with a mega-pixel large format CMOS detector. At 28 x 28 mm, the active area of the CMOS detector is significantly larger than any other solid-state detector currently used for ICP-OES. This combination allows the Prodigy Plus to achieve higher optical resolution and dispersion than any other solid-state detector-based ICP systems. The detector also provides continuous wavelength coverage from 165 to 1100 nm, permitting measurement over the entire ICP spectrum in a single reading, without sacrificing wavelength range or resolution. The detector design is inherently anti-blooming and is capable of random access, non-destructive readout that results in a dynamic range of more than six orders of magnitude. The Prodigy Plus uses a 40.68 MHz rugged, free-running RF generator, allowing it to handle the most difficult sample matrices, as well as common organic solvents.

Sample Introduction

A high-sensitivity sample introduction system ensured that sufficient and steady emission signals were delivered to the spectrometer. The sample introduction system consisted of:

- Cyclonic Spray Chamber with Center Knockout Tube
- Seaspray™ High Solids AR30 Concentric Nebulizer
- Four-Channel Peristaltic Pump

The volume of the cyclonic spray chamber is low allowing for fast washout between samples, while its knockout tube efficiently reduces the amount of sample aerosol that reaches the plasma. The Prodigy Plus torch is mounted using an innovative twist-lock auto-aligning sample introduction system, shown in Figure 2. This design permits operators to remove and replace the torch to the exact same position, providing day-to-day reproducibility and simplified training. Additionally, the twist-lock design automatically connects the coolant and auxiliary gas flows, eliminating potential errors.

Figure 2 Radial Twist Lock Sample Introduction System

Method

The Prodigy Plus instrument operating parameters are shown in Table I.

Table I Instrument Operating Parameters

Parameter	Value	Part Number
RF Power	1.2 kW	-
Coolant Flow	15 L/min	-
Auxiliary Flow	0 L/min	-
Plasma Configuration	Radial	
Nebulizer Pressure	34 psi	-
Pump Rate	35 rpm	-
Torch	Demountable Quartz (Axial, Radial and Dual)	318-00167-1
Injector	2.5 mm Bore Quartz Demountable	318-00161-AQ1
Sample Uptake Time	35 s	-
Nebulizer	Seaspray™ High Solids AR30 Concentric	120-00474-1
Spray Chamber	Cyclonic with Knock-out	120-00393-1
Rinse	20 s	-
Integration Time	Radial 5 s	-
Optical Purge Rate	0.7 L/min	-

Wavelengths

The wavelengths used for the analysis are shown in Table II. Scandium, added at 10 ppm, was used as an internal standard. $LiNO_3$ was added at 0.4% as an ionization buffer.

Table II Wavelengths

Element	Wavelength	View
Fe	259.940	Radial
Al	309.271	Radial
Mg	279.078	Radial
Ca	315.887	Radial
Na	589.592	Radial
K	766.491	Radial
Sc	361.383	Radial

Sample Preparation

1. Weigh 0.5 g of sample into a 250 mL flask and add 10 mL of 50% HCl. Heat on a hot plate for 15 minutes then cool to ambient. For samples containing any solids, filter using Watman 541 paper.
2. Add 1 g of $LiNO_3$ (ionization buffer) and 2.5 mL of a 1000 ppm Sc standard (internal standard).
3. Dilute to 250 mL with deionized water.

Note: Alternatively, the Li buffer and Sc internal standard can be added using an inline tee system, rather than adding the reagent to each sample and standard separately.

Calibration Standard Preparation

Monoammonium phosphate (MAP, 99.999%) was used to create the P standards. An appropriate amount of MAP was weighed and placed in a 250 mL volumetric flask with 10 mL of 1:2 HCl. Single-element solutions were used to make the calibration standard for the remaining elements. Gravimetric factors were used to convert the standards concentration to % oxide.

Results

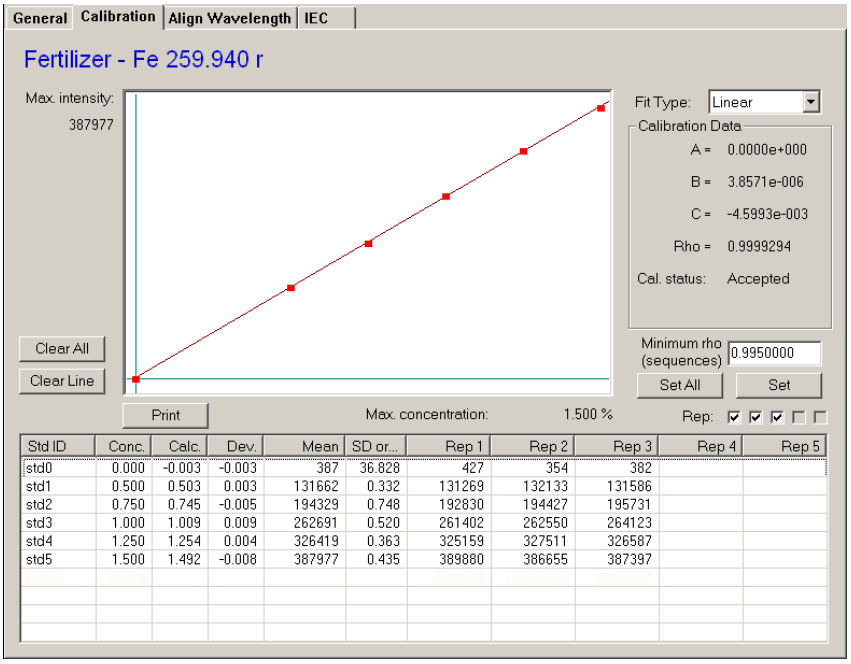
Calibration Results

After igniting the plasma and allowing 15 minutes for the instrument to warm up, the ICP was calibrated. Table III shows the calibration blank/standards and typical calibration data. A calibration curve is shown in Figure 3.

Table III Calibration Standard Concentrations

	P_2O_5	Fe_2O_3	Al_2O_3	MgO	CaO	Na_2O	K_2O
Std0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Std1	19.56	0.50	0.47	0.25	0.56	0.10	0.05
Std2	22.03	0.75	0.71	0.38	0.84	0.15	0.08
Std3	24.18	1.00	0.95	0.50	1.12	0.20	0.10
Std4	26.81	1.25	1.18	0.63	1.40	0.25	013
Std5	28.06	1.50	1.42	0.75	1.68	0.30	0.15

Figure 3 Fe 259.940 nm Calibration Data



Samples

Sample analysis results are shown in Table IV through Table VII. Agreement between the certified and measured values was very good.

Table IV NIST 120C Phosphate Rock - Results			
	Certificate	Measured	%Recovery
P_2O_5	33.3	33.0	99
Fe_2O_3	1.08	1.06	98
Al_2O_3	1.3	1.4	107
MgO	0.32	0.3	103
CaO	48.02	46.8	97
Na_2O	0.52	0.50	96
K_2O	0.15	0.14	96

Table V Diammonium Phosphate - Results			
	Certificate	Measured	%Recovery
P_2O_5	45.09	45.3	101
Fe_2O_3	1.13	1.2	103
Al_2O_3	1.85	1.8	98
MgO	0.89	0.85	96
CaO	0.25	0.27	107
Na_2O	0.17	0.16	95
K_2O	0.13	0.14	106

Table VI Monoammonium Phosphate - Results			
	Certificate	Measured	%Recovery
P_2O_5	47.6	47.1	99
Fe_2O_3	1.63	1.6	100
Al_2O_3	1.9	1.8	94
MgO	0.91	0.9	98
CaO	20.65	21.0	102
Na_2O	0.55	0.54	98
K_2O	0.2	0.20	102

Table VII Check 22 - Results			
	Certificate	Measured	%Recovery
P_2O_5	33.06	33.5	101
Fe_2O_3	1.1	1.1	101
Al_2O_3	1.42	1.5	104
MgO	0.35	0.3	99
CaO	47.74	46.7	98
Na_2O	0.4	0.40	99
K_2O	0.12	0.12	98

Conclusion

The analytical results demonstrate that the Prodigy Plus is well-suited for accurate and cost-effective determination of major elements in fertilizer. The sensitivity of the instrument using radial-view mode, combined with the instrument's broad linear dynamic range (due to the CMOS detector) allowed calibration over a wide concentration range. Consequently the Prodigy Plus High Dispersion ICP can easily accommodate fertilizers, as well as other related sample types.

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