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Application News

No. J127

Inductively Coupled Plasma Atomic Emission Spectrometry

Analysis of Liquid Fertilizer by ICPE™-9820

Introduction

Fertilizers contain many elements which are useful in crop growth in addition to nitrogen, potassium, and phosphorus, which are the principal ingredients. As an analysis method for fertilizers, Japan's Ministry of Agriculture, Forestry and Fisheries (MAFF) established Official Methods of Analysis of Fertilizers in 1992. However, because much time had passed since that standard was established, Food and Agricultural Materials Inspection Center (FAMIC) issued Testing Methods for Fertilizers, which introduced new analysis conditions and analysis methods suited to the times. Testing Methods for Fertilizers includes inductively coupled plasma (ICP) atomic emission spectrometry as an analysis method for water-soluble metallic ingredients [1], [2].

This article introduces an example of analysis of the water-soluble ingredients in liquid fertilizers using a Shimadzu ICPE-9820 simultaneous ICP atomic emission spectrometer.

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Analysis Samples

Four commercially-available liquid fertilizers were used in the analysis.

Sample Pretreatment

Based on Testing Methods for Fertilizers, 1 g of the sample was weighed, introduced into a 100 mL volumetric flask and diluted to the marked line with pure water, after which the solution was filtered with filter paper. A set amount of the filtrate was sampled and introduced into a 100 mL volumetric flask, and the measurement solution was prepared by adding hydrochloric acid (HCl) and yttrium (Y) as an internal standard element and diluting the solution to the marked line with pure water.

Instrument and Measurement Conditions

A Shimadzu ICPE-9820 simultaneous ICP atomic emission spectrometer was used in the measurements.

Table 1 shows the measurement conditions.

Table 1 Measurement Conditions

Instrument	: ICPE-9820
High frequency output	: 1.2 (kW)
Plasma gas flow rate	: 10 (L/min)
Auxiliary gas flow rate	: 0.6 (L/min)
Carrier gas flow rate	: 0.7 (L/min)
Nebulizer	: Nebulizer 10
Chamber	: Cyclone chamber
Torch	: Mini-torch
View directions	: Radial (RD) / Axial (AX)

Analysis

Table 2 shows the view direction and measured wavelength for each element. The measured wavelengths were based on Testing Methods for Fertilizers.

Table 2 View Direction and Measured Wavelength for Each Element

Element	View direction	Measured wavelength (nm)
B	Axial	249.773
Ca	Radial	393.366
Co	Axial	228.616
Cu	Axial	327.396
Fe	Axial	259.940
K	Radial	766.490
Mg	Radial	279.553
Mn	Axial	257.610
Mo	Axial	202.030
P	Radial	178.287
Zn	Axial	213.856

Study of Conditions

Next, we studied the conditions for determining whether ionization interference occurs or not and conducting a simultaneous analysis of the wavelengths prescribed in Testing Methods for Fertilizers. When analyzing samples with high concentrations of alkali metal elements, ionization interference due to self-absorption requires attention, and it is known that ionization interference causes deflection of the calibration curve in axial view observation. Fig. 1 shows the calibration curves for radial (RD) view and axial (AX) view observation of K. Obvious deflection of the calibration curve can be seen in the result for AX observation. In RD view observation, ionization interference can be suppressed and the calibration curve displays satisfactory linearity. RD view observation also enables analysis at the prescribed wavelength in the case of Ca, which has good sensitivity. Thus, combined use of AX and RD view observation makes it possible to conduct simultaneous analysis of elements over the full range of concentrations from low to high without changing the dilution factor depending on elements.

Analysis Results

Table 3 shows the quantitation results for the respective samples. Table 4 shows the results of the dilution tests and spike and recovery tests of the samples. To check for interference, dilutions tests were conducted for samples with high concentrations of the contents, and spike and recovery tests were conducted for samples with low concentrations.

Satisfactory results were obtained for all samples, as the results of the dilution test and spike and recovery test were within 90% to 110% in all cases.

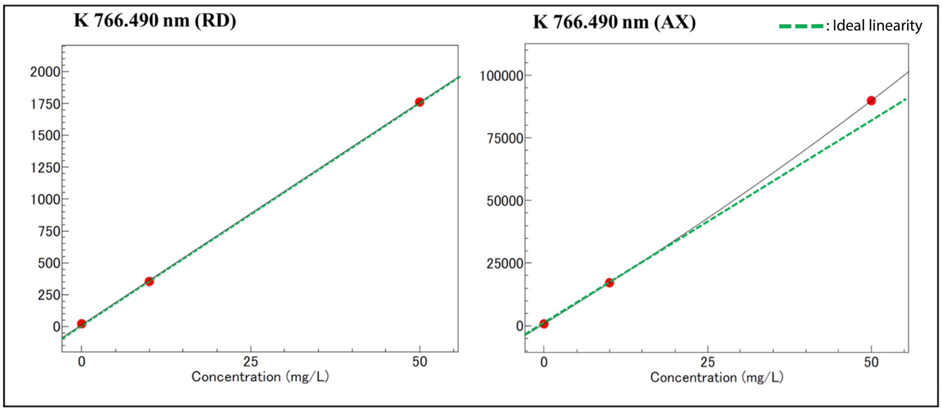

Fig. 1 Calibration Curves of K by Radial (RD) View and Axial (AX) View Observation

Conclusion

Eleven water-soluble elements in four liquid fertilizers were measured in accordance with Testing Methods for Fertilizers (2019). Use of the Shimadzu ICPE-9820 enabled simultaneous analysis over the full range of concentrations from low to high by combined use of high sensitivity axial (AX) view observation and radial (RD) view observation for analysis of high concentration ingredients. In addition, dilution tests and spike and recovery tests demonstrated that simultaneous analysis of low concentration ingredients is also possible without interference caused by high concentration ingredients.

<References>
(1) Testing Methods for Fertilizers (2019) (Food and Agricultural Materials Inspection Center (Incorporated Administrative Agency))
(2) Keisuke Aoyama, Simultaneous Determination of Water-Soluble Principal Ingredients (W-P_2O_5, W-K_2O, W-MgO, W-MnO and W-B_2O_3) in Liquid Fertilizer using Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) (FAMIC, Research Report of Fertilizer, No. 8 (2015))

Table 3 Analysis Results of Samples

Unit: mg/L

Element	Liquid fertilizer A	Liquid fertilizer B	Liquid fertilizer C	Liquid fertilizer D	Limit of Quantitation 10 σ
B	0.180	0.022	0.004	0.061	0.002
Ca	<	0.22	0.04	0.06	0.02
Co	<	<	<	<	0.002
Cu	0.059	<	<	<	0.003
Fe	0.141	0.485	0.007	0.015	0.001
K	46	47	69	48	1
Mg	0.073	0.390	0.253	0.654	0.001
Mn	0.054	0.013	0.002	0.051	0.0003
Mo	0.062	0.008	0.005	0.007	0.004
P	54	48	31	48	1
Zn	0.062	0.005	0.004	0.008	0.001

< : Below LOQ

Table 4 Results of Spike and Recovery Tests and Dilution Tests of Samples

Unit: %

Element	Liquid fertilizer A	Liquid fertilizer B	Liquid fertilizer C	Liquid fertilizer D
B	106	100	99	99
Ca	100	100	99	100
Co	104	106	102	102
Cu	104	102	99	100
Fe	108	98*	104	103
K	102*	101*	99*	102*
Mg	99	100	100	100
Mn	107	105	103	104
Mo	104	101	100	98
P	99*	95*	99*	99*
Zn	106	104	102	103

* : Result of dilution test

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First Edition: Feb. 2020