

Application News

Energy Dispersive X-ray Fluorescence Spectrometer ALTRACE™

Elemental Analysis of Compost Using an Energy Dispersive X-Ray Fluorescence Spectrometer

Yuki Tamura

User Benefits

- ◆ Results consistent with those from ICP-AES can be obtained.
- ◆ No acid pretreatment is required, so samples can be analyzed after simply loading them into the sample container.
- ◆ ALTRACE systems can increase the efficiency of research and development operations for fertilizers that contain compost, as well as for incoming inspection of raw materials.

Introduction

The Japanese Ministry of Agriculture, Forestry and Fisheries recommends the active use of compost as fertilizer to reduce greenhouse gas emissions from livestock manure treatment and reduce the use of chemical fertilizers.

At the same time, compositional analysis is indispensable for the safe and effective use of compost as a regional resource. Quantification of the three primary fertilizer nutrients—nitrogen, phosphate, and potassium—as well as the carbon/nitrogen (C/N) ratio, pH, electrical conductivity (EC), and heavy metals content allows more precise fertilization planning and optimized use in combination with chemical fertilizers, which helps stabilize crop productivity and quality while reducing environmental impacts caused by excessive application.

Elemental analysis of compost is commonly performed by atomic absorption spectrophotometry (AAS) or inductively coupled plasma atomic emission spectrometry (ICP-AES), both of which generally require pretreatment such as acid digestion. In contrast, X-ray fluorescence analysis does not require such acid treatment. Irradiating the compost with X-rays enables measurement of the average elemental composition within the irradiated area, equivalent to bulk analysis.

For this Application News article, compost was qualitatively and quantitatively analyzed using an ALTRACE energy dispersive X-ray fluorescence (EDXRF) spectrometer. The validity and usefulness of the quantitative values obtained with the EDXRF system were also evaluated by comparison with ICP-AES results.

Although X-ray fluorescence spectrometry is not specified in the official fertilizer testing methods, its ease of use contributes to more efficient fertilizer R&D and incoming inspection of raw materials.

Sample

Air-Dried Compost

Main ingredients: Cattle manure and pig manure

Auxiliary ingredient: Rice husks

Elements

Target elements: ^{11}Na - ^{92}U

Sample Preparation

The sample was dried at 110 °C for 2 h, because high moisture content makes it difficult to evacuate the sample chamber during measurement. The moisture content calculated from the weight change before and after drying was 12.5 %.

The dried sample was placed in a sample cell lined with polypropylene film (thickness: 5 μm) for analysis.

A photo of the sample is shown in Fig. 1.



Fig. 1 Sample

Qualitative Analysis Results

Fig. 2 shows the qualitative analysis results obtained with the ALTRACE system. For the organic components (C to O), CH₂O was set as the balance component and the detected elements were quantified as oxides.

X-ray fluorescence analysis enables comprehensive analysis of inorganic elements in a sample after only simple sample preparation. The fundamental parameter (FP) method, a theoretical calculation-based quantitative method, does not require using standard samples.

The ALTRACE can also readily analyze poorly soluble components, such as silica and alumina.

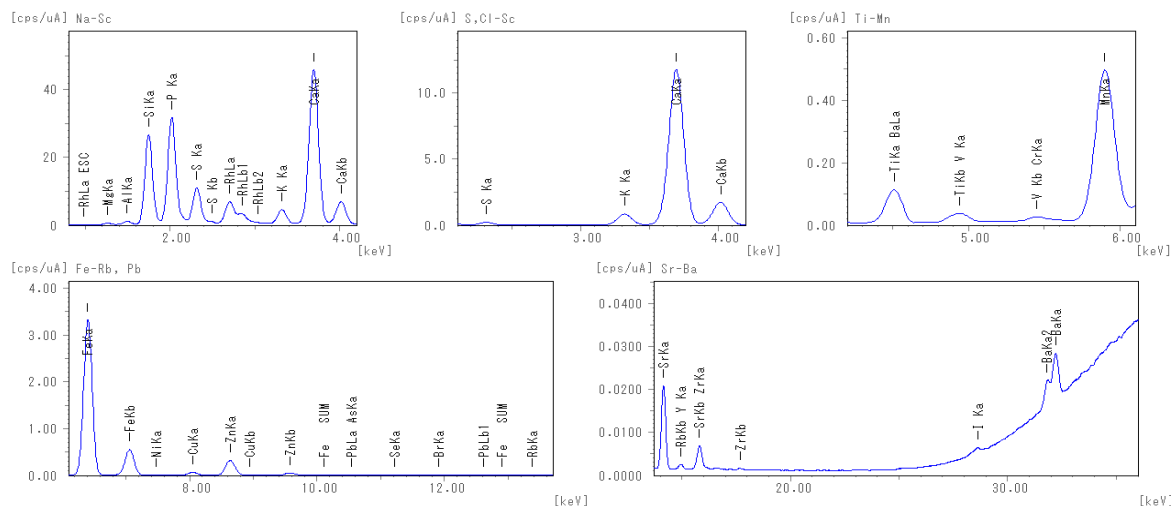


Fig. 2 Qualitative Analysis Results

Table 1 Quantitative Analysis Results (ALTRACE)

Unit	SiO ₂	P ₂ O ₅	CaO	Fe ₂ O ₃	MgO	Al ₂ O ₃	SO ₃	K ₂ O	ZnO	TiO ₂
%	14.6	9.62	7.80	7.06	2.19	0.93	0.79	0.78	0.23	0.22
	MnO	CuO	SrO	BaO	V ₂ O ₅	Cr ₂ O ₃	ZrO ₂	RbO ₂	NiO	Br
ppm*1	1215	650	182	96	92	34	19	18	12	10
	Y ₂ O ₃	I	As ₂ O ₃	PbO	SeO ₂	CH ₂ O (%)				
ppm	7	7	7	2	2	55.6				

*1 ppm = mg/kg

Table 2 Comparison of Quantitative Values Obtained by ALTRACE and ICP-AES

Item	Unit	ALTRACE (Quantitative Value ± 3σ*1)	ICP-AES (Average Value ± 2SE*2)	
			Digestion Method	Hydrochloric Acid Extraction Method
P ₂ O ₅	%	8.42 ± 0.02	9.76 ± 0.22	8.05 ± 0.90
K ₂ O	%	0.68 ± 0.004	0.66 ± 0.03	0.59 ± 0.04
CaO	%	6.82 ± 0.02	7.87 ± 0.30	7.67 ± 0.20
MgO	%	1.91 ± 0.04	2.19 ± 0.07	2.26 ± 0.14
SO ₃	%	0.69 ± 0.009	2.03	0.09 ± 0.04
Fe	mg/kg	43200 ± 65	46700 ± 2600	31000 ± 6400
Mn	mg/kg	823 ± 6	888 ± 129	966 ± 81.4
Zn	mg/kg	1620 ± 8	1570 ± 16	1260 ± 230
Cu	mg/kg	454 ± 5	436 ± 19	55.5 ± 3.2

*1 σ: Standard deviation *2 SE: Standard error

■ Quantitative Analysis Results

Table 1 shows the quantitative analysis results obtained with the ALTRACE system and Table 2 compares the quantitative values with those obtained by ICP-AES. The ICP-AES quantitative values were taken from the results of a round-robin test organized by Zenkoku Tsuchi no Kai. Depending on the analyte, 22 institutions throughout Japan participated in the analysis of compost.

In Table 1, the values were calculated on the assumption that the components in the dried sample were present as oxides. In Table 2, the values shown were converted to concentrations before drying. In addition, to match the notation used for the ICP-AES quantitative values, some elements were converted from oxide-based concentrations to elemental concentrations.

The quantitative values obtained with the ALTRACE system are based on an X-ray fluorescence technique that provides bulk analysis results. Those results were in good agreement with those obtained by the ICP-AES digestion method. Although the difference in the quantitative value for SO₃ is relatively large, that is presumably due to the fact that only one data point was available. Comparison with the values obtained by the ICP-AES hydrochloric acid extraction method also indicates that the difference from the quantitative values obtained with the ALTRACE system increases for heavy elements with low extraction recoveries.

Further improvement in the accuracy of the quantitative values can be expected by calibrating the sensitivity coefficients of the instrument (ALTRACE) using standard samples.

■ Conclusion

This Application News article compares the quantitative values obtained by an energy dispersive X-ray fluorescence (EDXRF) spectrometer with those obtained by inductively coupled plasma atomic emission spectrometry (ICP-AES). That confirmed that, when digestion was used as the pretreatment method for ICP-AES, the quantitative values obtained by the EDXRF spectrometer, which provides bulk analysis results, were in good agreement with the ICP-AES values.

Because analysis requires only simple sample preparation, EDXRF spectrometers contribute to improving the efficiency of researching and developing fertilizers that contain compost and conducting incoming inspections of raw materials.

■ Analysis Conditions

Table 2 summarizes the analysis conditions.

Table 3 Analysis Conditions

Instrument	ALTRACE
Target Elements	¹¹ Na- ⁹² U
Detector	SDD
X-Ray Tube	Rh target
Tube Voltage	65 kV: Sr-Ba, 50 kV: Fe-Rb and Pb, 30 kV: Ti-Mn, 15 kV: Na-Sc, and S and Cl-Sc
Tube Current	Auto (μA)
Primary Filter	#1: Sr-Ba, #2: Sr-Ba, #5: Fe-Rb and Pb, #6: Ti-Mn, None: Na-Sc
Atmosphere	Vac
Integration Time	100 s × 5 ch
Dead Time	Max. 15 %

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