

X-Ray Fluorescence Analysis of Crude Drugs

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User Benefits

- ◆ Energy-dispersive X-ray fluorescence (EDXRF) analysis enables rapid, non-destructive analysis of inorganic elements in crude drugs with only simple sample preparation.
- ◆ ALTRACE EDXRF spectrometers offer sufficient analytical sensitivity for screening samples with respect to the limits specified in the Japanese Pharmacopoeia for testing heavy metals and arsenic.
- ◆ Qualitative-quantitative analysis can easily determine the elemental composition of crude drugs, including not only heavy metals but also mineral components.

■ Introduction

Crude drugs are derived from natural sources, such as plants and animals. Qualitative and quantitative analysis of their elemental composition is important for ensuring their quality and safety. In particular, the content of mineral components and heavy metals in crude drugs can vary depending on the cultivation environment and processing steps. Therefore, they must be evaluated by appropriate analytical methods.

Methods for analyzing elemental composition include colorimetric methods, such as specified in the Japanese Pharmacopoeia (JP) for testing heavy metals and arsenic. Instrumental techniques, such as atomic absorption spectrophotometry (AAS), inductively coupled plasma atomic emission spectrometry (ICP-AES), and inductively coupled plasma mass spectrometry (ICP-MS), are also used in some cases.

The heavy metals test protocol in the JP adds a sodium sulfide reagent under acidic conditions and then evaluates resulting coloration as a lead (Pb)-equivalent value. That offers the advantage of being able to assess the total amount of heavy metals with a simple process. However, it has several drawbacks, including the need for skilled operation and reagent handling, as well as limited sensitivity and reproducibility.

In contrast, though AAS and ICP-AES, ICP-MS enable highly sensitive and highly accurate analysis, they require complete dissolution of samples in an acid, such as nitric acid or hydrochloric acid, as well as advanced expertise in chemical pretreatment. In addition, the instrument running costs and installation requirements can make their adoption difficult. Given such background circumstances, the adoption of analytical methods that enable rapid analysis with simpler sample preparation are increasingly expected.

This article describes examples of analyzing hazardous heavy metals in crude drugs using the calibration curve method and examples of qualitative-quantitative analysis using the Fundamental Parameter (FP) method, both performed with an energy dispersive X-ray fluorescence (EDXRF) spectrometer.

■ Features of EDXRF

A major feature of EDXRF (Fig. 1) is that samples can be analyzed as-is or with only simple sample preparation, such as crushing. It can be used to analyze samples in various forms, including solids, powders, and liquids, and elements with a wide range of concentrations, from the ppm level to 100 %.

Two quantitative analysis methods are available, which can be selected according to the given application. The calibration curve method requires standard samples and the FP (fundamental parameter) method does not require standard samples and derives quantitative values based on theoretical intensity calculations.

In addition, because EDXRF spectrometry is a non-destructive analytical technique, the same sample can be subsequently reanalyzed by other analytical methods, which is also a major advantage.



Fig. 1 ALTRACE EDXRF Spectrometer

■ Sample

Five crude drugs

(cinnamon bark, citrus unshiu peel, coptis rhizome, glycyrrhiza, and pueraria root)

■ Sample Preparation

The sample was placed in a sample cell lined with polypropylene film (thickness: 5 µm) and subjected to simple compression. Fig. 2 shows a photo of the sample.



Fig. 2 Sample (Glycyrrhiza)

1. Analysis of Hazardous Heavy Metals (Calibration Curve Method)

■ Elements

^{82}Pb , ^{33}As , ^{48}Cd , and ^{80}Hg

■ Standard Samples

Atomic absorption standard solutions (from FUJIFILM Wako Pure Chemical Corporation)

Five concentration levels were prepared: 0, 5, 10, 20, and 40 ppm (only 0, 1, 5, 10, and 20 ppm for Hg)

■ Calibration Curves

Fig. 3 shows the calibration curves. The As curve was corrected for overlap with Pb using coexisting element correction (dj method). In addition, internal standard correction using scattered X-rays was applied to compensate for matrix effects.

The calibration curves in Fig. 3 show good results for all elements, with accuracies within 0.5 ppm or less.

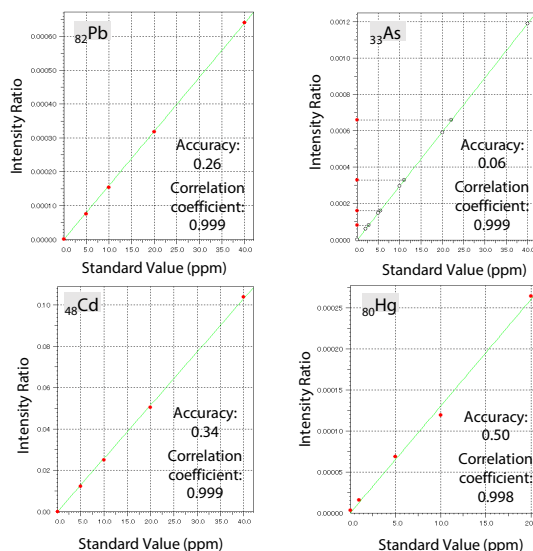


Fig. 3 Calibration Curve

■ Quantitative Analysis Results

Five commercially available crude drugs were quantitatively analyzed by the calibration curve method, with the results indicated in Table 1.

With an integration time of 300 s, the lower limits of quantitation were approximately 0.2 ppm for As, Hg, and Pb, and approximately 0.5 ppm for Cd (Table 1).

Since these values are lower than the limits specified in the Japanese Pharmacopoeia (JP), such as not more than 10 ppm for heavy metals and not more than 5 ppm for As, the system was confirmed to have performance useful for assessing compliance with the JP limits.

Table 1 Quantitative Analysis Results (Calibration Curve Method)

[ppm]

Sample	Pb	As	Cd	Hg	Heavy Metals Limit*	Arsenic Limit*
Cinnamon Bark	0.84	< 0.21	< 0.60**	< 0.15	Not stated	
Citrus Unshiu Peel	< 0.15	< 0.16	< 0.45	< 0.21	Not stated	
Coptis Rhizome	1.35	0.24	0.78	< 0.24	20 ppm or less	5 ppm or less
Glycyrrhiza	< 0.21	0.24	< 0.52	< 0.20	10 ppm or less	5 ppm or less
Pueraria Root	0.28	< 0.18	< 0.41	< 0.17	10 ppm or less	5 ppm or less

* Source: The Japanese Pharmacopoeia, Eighteenth Edition.

** The symbol "<" indicates that the result is below the lower limit of quantitation. The lower limit of quantitation is the theoretical 10 σ value.

■ X-Ray Fluorescence Spectra

Fig. 4 shows the superimposed profiles for each element. The Pb profile clearly shows differences in measured intensity for low concentrations below 1 ppm. Because the AsK α and PbL α peaks overlap, differences in As content cannot be determined from the peak shapes.

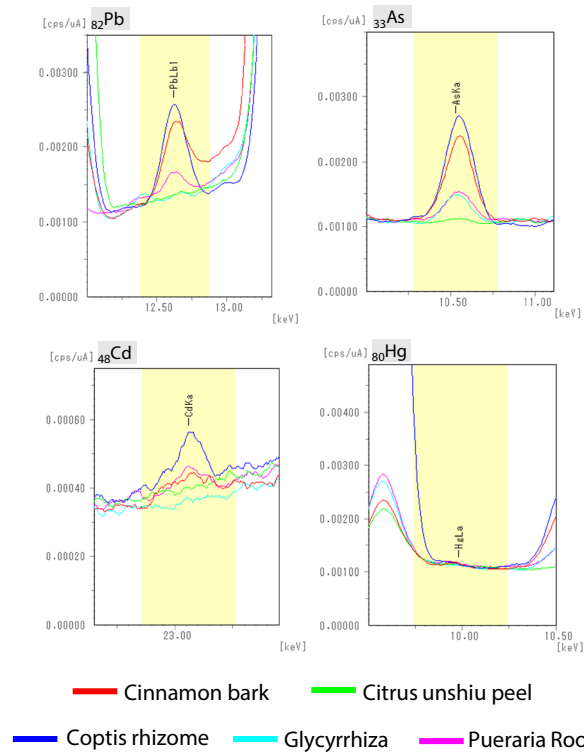


Fig. 4 Overlaid Profiles

■ Results of Repeatability Tests

The coptis rhizome sample was analyzed 10 consecutive times, with repeatability test results indicated in Table 2.

Despite Pb content of about 1 ppm, the coefficient of variation was 1.5 %, which indicates that the analysis was very accurate (Table 2). The As and Cd concentration levels close to the lower limit of quantitation resulted in coefficient of variation values of about 10 %.

Table 2 Results of Repeatability Test

[ppm]

Element	Pb	As	Cd	Hg
Mean	1.37	0.23	0.79	< 0.2*
Standard Deviation	0.020	0.017	0.060	—
Coefficient of Variation (%)	1.5	7.2	7.6	—

* Below the lower limit of quantitation

■ Analysis Conditions

The analysis conditions are summarized in Table 3.

Table 3 Analysis Conditions

Channel	Cd	Pb	As	Hg
Analytical Line	CdKα	PbLβ1	AsKα	HgLα
X-Ray Tube	Rh target			
Tube Voltage	65 kV	50 kV		
Tube Current	Auto [μA]			
Integration Time	300 s*	300 s*		
Dead Time	Max. 15 [%]			
Primary Filter	#1	#5		
Detector	SDD			
Atmosphere	Air			
Analysis Group	Calibration curve method			

* For internal standard correction, the background intensity was measured separately for 100 s.

2. Qualitative-Quantitative Analysis (FP Method)

■ Elements

$_{11}\text{Na}$ to $_{92}\text{U}$

Qualitative analysis combined with FP quantification allows comprehensive multi-element determination.

■ Qualitative-Quantitative Analysis Results

The resulting qualitative profiles are shown in Fig. 5 and the quantitative analysis results are indicated in Table 4. For the FP quantitative calculations, the organic components that cannot be detected by EDXRF were treated as $\text{C}_6\text{H}_{10}\text{O}_5$ (cellulose).

Qualitative-quantitative analysis makes it possible to easily determine the content of mineral components, such as Mg, P, K, and Ca, as well as trace hazardous elements, such as Pb.

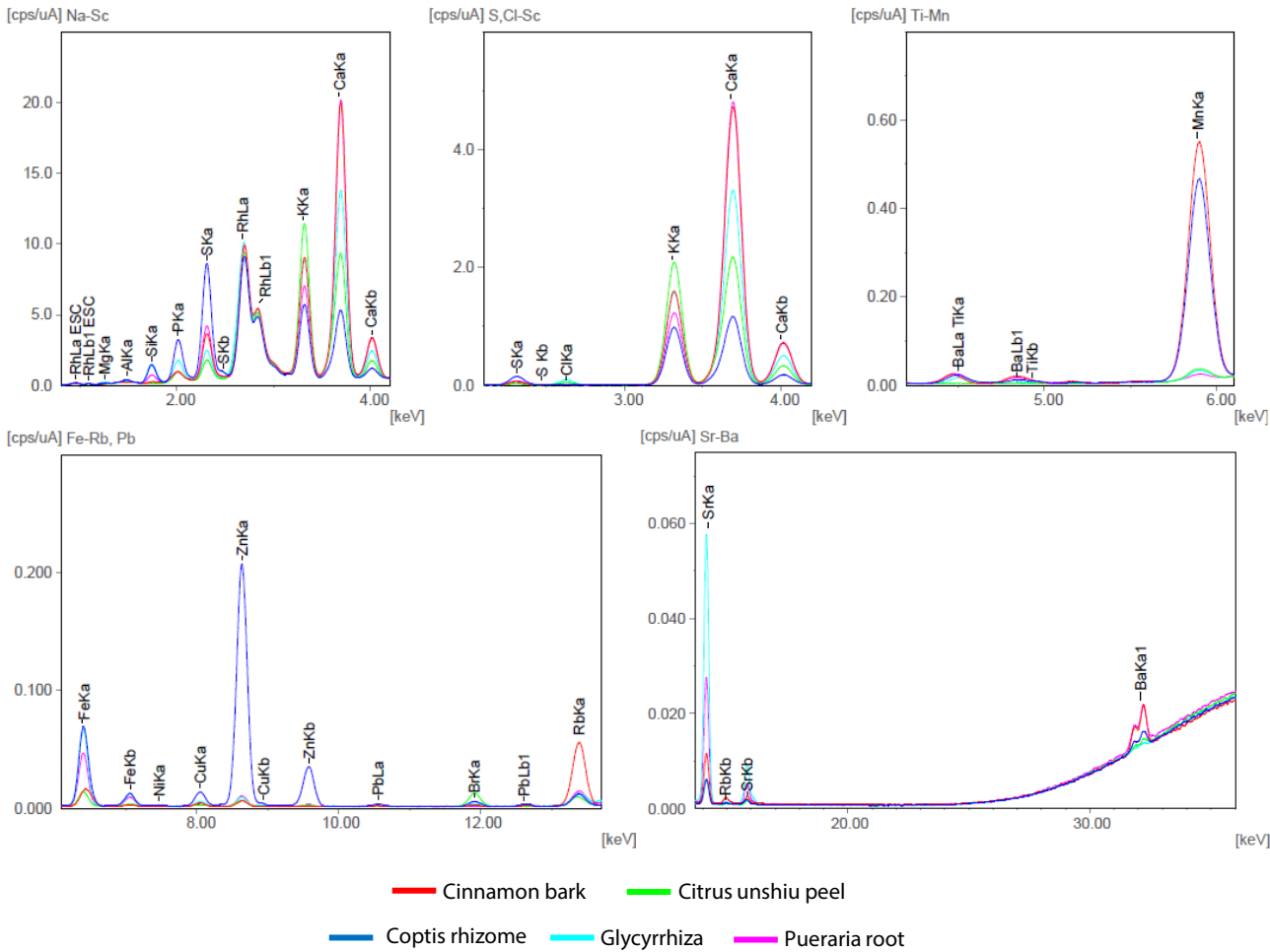


Fig. 5 Qualitative Profiles

Table 4 Quantitative Analysis Results (FP method)

Sample	Ca	K	S	P	Mg	Al	Mn	Si	Ba	Cl
Cinnamon Bark	9035	4627	1015	562	—*	—	345	124	97	71
Citrus Unshiu Peel	4537	5992	456	697	364	—	14	—	7.4	430
Coptis Rhizome	2187	2955	2491	2246	1117	1180	264	1878	43	137
Glycyrrhiza	6431	4706	636	1133	1951	866	12	1902	—	764
Pueraria Root	9268	3606	1238	580	2359	706	6.9	784	90	48
Sample	Fe	Sr	Rb	Zn	Cu	Ti	Br	Pb	Ni	$\text{C}_6\text{H}_{10}\text{O}_5$ (%)
Cinnamon Bark	41	37	36	7.5	5.6	—	1.1	1.1	—	98.4
Citrus Unshiu Peel	61	18	3.0	6.7	2.4	—	5.9	—	—	98.7
Coptis Rhizome	311	18	4.6	258	20	18	2.3	1.9	2.0	98.5
Glycyrrhiza	333	183	4.8	10	7.8	23	1.3	—	—	98.1
Pueraria Root	233	88	6.0	12	5.8	10	—	—	—	98.1

* "—" indicates not detected.

■ Analysis Conditions

The analysis conditions are summarized in Table 5.

Table 5 Analysis Conditions

Channel	Na-Sc	S, Cl-Sc	Ti-Mn	Fe-Rb, Pb	Sr-Ba
Analytical Line	K α , K β , L α , L β ...				
X-Ray Tube	Rh target				
Tube Voltage	15 kV		30 kV	50 kV	65 kV
Tube Current	Auto [μ A]				
Integration Time	60 s	60 s	60 s	60 s	60 s
Dead Time	Max. 15 [%]				
Primary Filter	None	#2	#6	#5	#1
Detector	SDD				
Atmosphere	Vac				
Analysis Group	FP method				

■ Conclusion

EDXRF spectrometry is an effective technique for simple, rapid, non-destructive analysis of mineral components and heavy metals in crude drugs. By using an ALTRACE EDXRF spectrometer, the burden on analysts associated with sample preparation can be reduced and large numbers of samples can be analyzed efficiently. As the quality control and monitoring of hazardous elements become increasingly important, X-ray fluorescence analysis holds considerable promise as a useful alternative technique in the field of crude drug analysis.

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