

# Application News

Energy Dispersive X-ray Fluorescence Spectrometer EDX-7200

## Quantitative Analysis of Antacid (Aluminum) in Drug by EDX

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### User Benefits

- ◆ The titration method and atomic absorption spectrophotometry require complex sample preparation, but with EDX, analysis is possible with simple sample preparation.
- ◆ The analysis time is only a few minutes, contributing to work efficiency.
- ◆ Repeatability is good, and highly accurate results can be obtained.

### ■ Introduction

Aluminum (Al) compounds such as aluminum hydroxide gel and synthetic hydrotalcite are widely used as antacids because they have the effect of neutralizing excess stomach acid and reducing irritation of the stomach wall. At pharmaceutical manufacturing sites, the titration method or atomic absorption spectrophotometry is generally adopted for control of antacids in drug products, but these analytical methods have the problems shown in the following Table 1.

Because the energy dispersive X-ray fluorescence spectrometer (EDX) enables analysis with only simple sample preparation, and the analysis time is also short, at a few minutes per sample, higher analysis efficiency can be expected. For this reason, EDX has attracted attention as an alternative analytical method to the titration method and atomic absorption spectrophotometry.

This article introduces an example of an analysis of an antacid (Al) used in commercially-available drugs.

Table 1 Problems of Titration Method and Atomic Absorption Spectrophotometry

Analysis technique	Problems
Titration method	(1) Necessary to prepare various samples.
	(2) Long analysis time.
	(3) Individual differences are likely to occur in recognizing color changes.
Atomic absorption spectrophotometry	(1) Sample preparation is comparatively complex.
	(2) Restrictions on where the equipment can be installed.

### ■ Calibration Curve Samples

An aluminum (Al) compound, which is the active ingredient, and admixtures were mixed so as to have substantially the same quality as the targeted formulation. The Al compound was added in proportions of 80 mg, 100 mg, and 120 mg by Al conversion in the maximum daily dosage of the target formulation (Table 2). Calibration curve samples were then prepared by pulverizing the samples with a mill, packing  $n = 3$  samples of each concentration in polyvinyl chloride (PVC) rings with an inner diameter of 25 mm $\varphi$ , and compacting the samples at 80 kN for 10 seconds.

Since this sample was brittle even after pressure molding, analysis was conducted with a 5  $\mu\text{m}$  polypropylene film placed underneath to prevent sample damage during measurement. Fig. 1 shows an image of a sample.

Table 2 Calibration Curve Samples

Calibration curve sample	Al content [mg]
(1)	80
(2)	100
(3)	120



Fig. 1 Image of Sample

### ■ Calibration Curve

Fig. 2 shows the calibration curve of Al which was prepared using the 3 calibration curve samples (average intensity of  $n = 3$  for each sample), together with the coefficient of correlation  $R$ . Fig. 3 shows the profile overlay of Al for the three concentration levels ( $n = 1$ ).

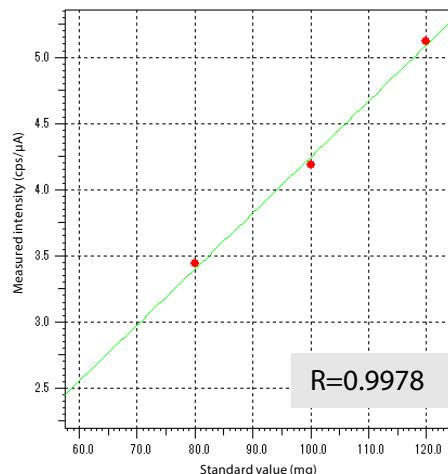


Fig. 2 Calibration Curve of Al

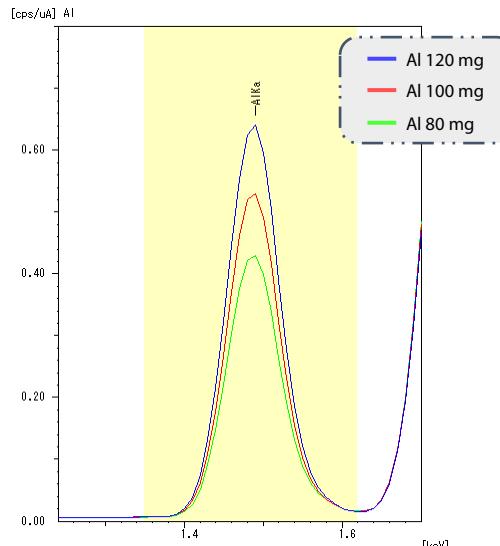


Fig. 3 Profile Overlay of Al

## ■ Repeatability

Table 3 shows the results of a simple 10-time repeatability test of calibration curve sample (2) Al 100 mg. The coefficient of variation is satisfactory, at 1 % or less.

Table 3 Results of Simple Repeatability Test [mg]

Average	100.7
Standard deviation	0.70
Coefficient of variation [%]	0.7

## ■ Comparison of Unknown Sample Analysis by EDX with Titration Method

Three unknown samples were prepared ( $n = 3$  for each sample material), and a quantitative analysis was conducted. Table 4 shows the results. The relative error of the average values obtained by the EDX method to the quantitative values obtained by the titration method showed a satisfactory correlation of 3.5 % or less.

A significance test of the difference in the quantitative values by the two methods was carried out assuming a significance level of 5 %. Because the null hypothesis "no difference" is not rejected, it cannot be concluded that there is a difference between the two methods.

EDX is a potential alternative to the titration method, and the evaluation of that possibility is based on a total judgment, including the number of unknown sample materials, the control range of the amount of addition, the variance between the two methods (homogeneity of variance test) and other factors.

Table 4 Comparison of Quantitative Analysis Results of Unknown Samples with Titration Method [mg]

	Unknown sample (1)	Unknown sample (2)	Unknown sample (3)
n=1	106.1	112.5	105.5
n=2	110.6	115.4	108.1
n=3	109.7	113.7	104.4
Average value by EDX	108.8	113.9	106.0
Quantitative value by titration method	110.8	113.8	109.8
Relative error [%]	1.8	0.1	3.5
Result of significance test*	Cannot be concluded that difference exists.		

\*By the t-test (assuming a significance level of 5 % and equal variance) using commercial spreadsheet software.

## ■ Analysis Conditions

Table 5 shows the analysis conditions.

Table 5 Analysis Conditions

Instrument	: EDX-7200
Element	: $^{13}\text{Al}$
Analysis group	: Quantitative analysis
Analysis method	: Calibration curve method
Detector	: SDD
X-ray tube	: Rh target
Tube voltage-current	: 15 [kV] – Auto [ $\mu\text{A}$ ]
Collimator	: 10 [mm $\phi$ ]
Primary filter	: None
Atmosphere	: Vacuum
Integration time	: 100 [s]
Dead time	: Max. 30 [%]

## ■ Conclusion

EDX has the following advantages:

(1) Analysis is possible with simple sample preparation.

(2) Short analysis with a few minutes.

(3) High repeatability.

(4) Easy analysis operation.

EDX is expected to be used as an alternative to the titration method in research and development and quality control work.



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### › EDX-7200

Energy Dispersive X-ray Fluorescence  
Spectrometer

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Pharmaceutical

› Biopharmaceutical

› Price Inquiry

› Product Inquiry

› Technical Service /  
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