

## Application News

Energy Dispersive X-ray Fluorescence Spectrometer ALTRACE™

### Quantitative Analysis of Lead (Pb) and Arsenic (As) in Cosmetic Raw Material Powders

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#### User Benefits

- ◆ With simple sample preparation, lead and arsenic in cosmetic raw material powder can be quantified simultaneously.
- ◆ Increased X-ray tube output and optimized optical design have improved sensitivity for heavy elements.
- ◆ Continuous analysis of up to 48 samples is possible, improving analysis throughput.

#### ■ Introduction

The management of lead and arsenic contained in cosmetics raw materials is important for protecting people's health. Although colorimetric methods have been used for these analyses, there is a growing need for trace control due to tighter regulations in recent years, leading to a consideration of a shift to instrumental methods that enable quantification. Candidates for instrumental methods include ICP optical emission spectrometry/mass spectrometry and X-ray fluorescence spectrometry. While the former method is highly sensitive, powder and solid samples need to be dissolved in an acid such as nitric acid or hydrochloric acid. In contrast, X-ray fluorescence analysis is convenient because it can be analyzed without dissolving the sample.

This application news introduces the analysis of two inorganic oxide powders (talc and titanium oxide) using the energy-dispersive X-ray fluorescence Spectrometer ALTRACE (Fig. 1).



Fig. 1 ALTRACE™

#### ■ Calibration Curve

Two types of powders, talc ( $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$ ) and titanium oxide ( $\text{TiO}_2$ ), were prepared by adding and homogenizing lead (Pb) and arsenic (As) standard solutions for ICP at three levels (Table 1). The sample volume was 2 g per sample. Using these calibration curve samples, calibration curves for Pb and As were prepared for each of the two types of powders. Fig. 2 shows the calibration curves, Table 2 shows the accuracy and correlation coefficient of the calibration curves, and Fig. 3 shows the profiles. For As, overlap correction with Pb (coexisting element correction dj method) was applied.

Table 1 Calibration Curve Samples [ppm]

Sample	As	Pb
Blank	0	0
STD1	2	20
STD2	5	10
STD3	10	5

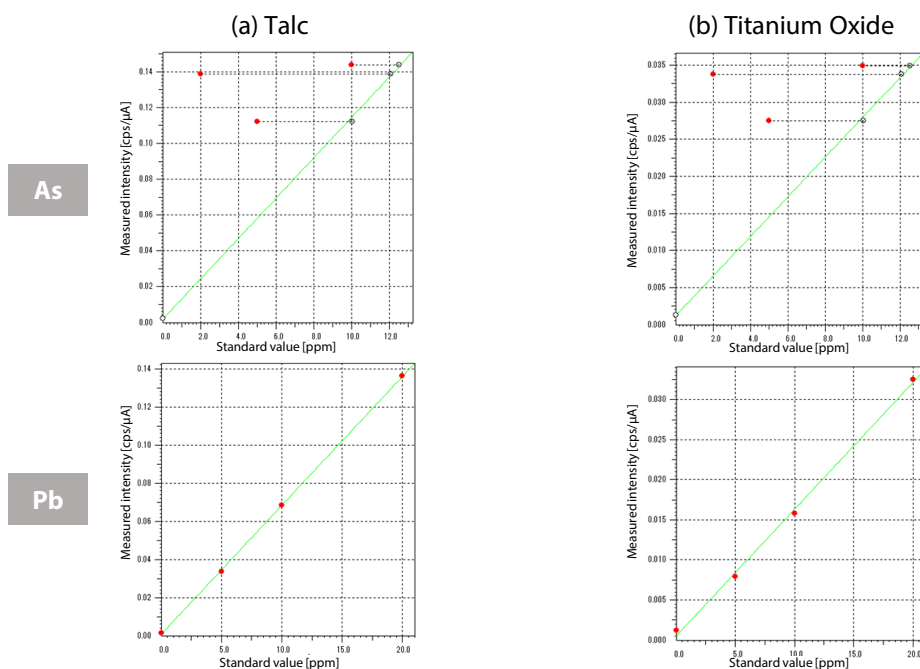
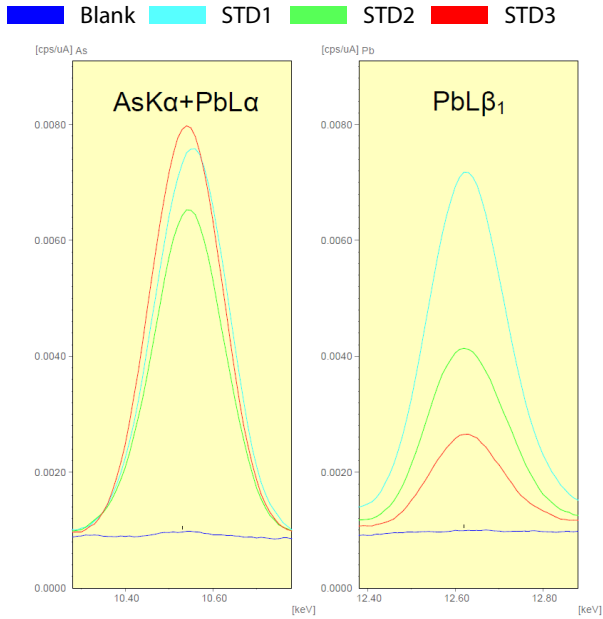


Fig. 2 Calibration Curves (a) Talc, (b) Titanium Oxide

Table 2 Accuracy [ppm] and Correlation Coefficients of the Calibration Curves

Sample	As		Pb	
	Acc.	Corr. Coef.	Acc.	Corr. Coef.
Talc	0.15	0.9997	0.15	0.9999
Titanium oxide	0.14	0.9997	0.40	0.9989

(a) Talc



(b) Titanium Oxide

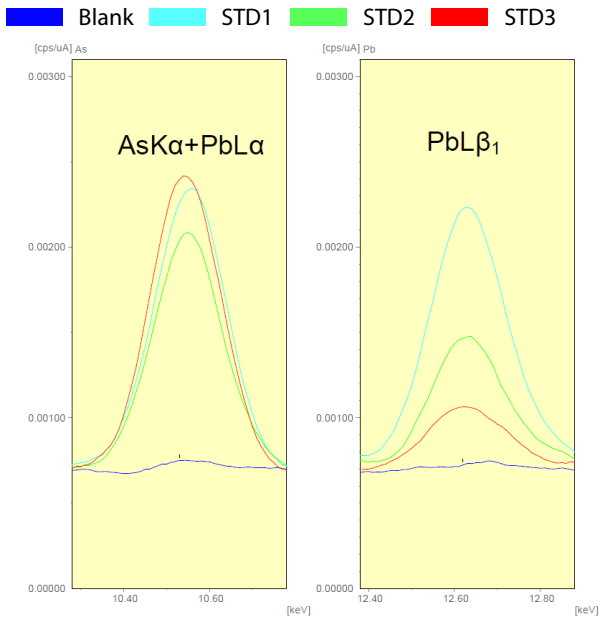


Fig. 3 The Profiles of the Calibration Curve Samples (As, Pb)  
(a) Talc, (b) Titanium Oxide

### ■ Lower Limit of Detection

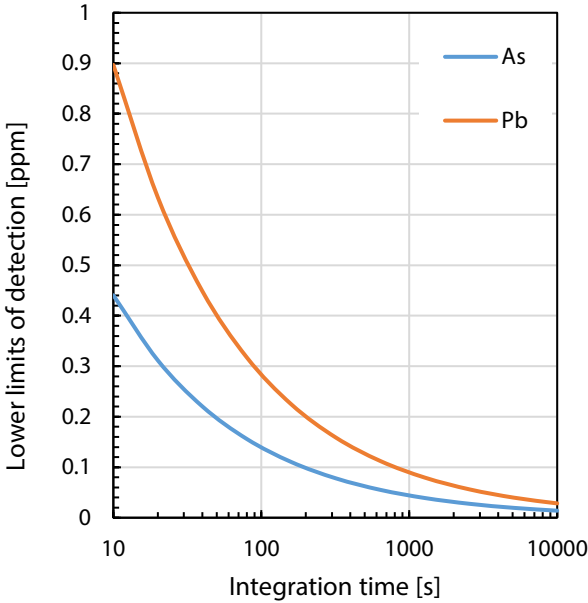
Table 3 shows the lower limits of detection calculated from the calibration curve. The integration time was set to 100 seconds for talc and 300 seconds for titanium oxide.

The lower limits of detection depend on the material, measured element, integration time, and other factors (Fig. 4). If the material is the same, the detection limit theoretically becomes  $1/\sqrt{N}$  times when the integration time is multiplied by  $N$ . The integration time is set according to the required detection limit.

Table 3 The Lower Limits of Detection (LLD) [ppm]

Sample	Element	Analysis line	LLD
Talc (100 s)	As	As K $\alpha$	0.14
	Pb	Pb L $\beta_1$	0.28
Titanium oxide (300 s)	As	As K $\alpha$	0.30
	Pb	Pb L $\beta_1$	0.59

(a) Talc



(b) Titanium Oxide

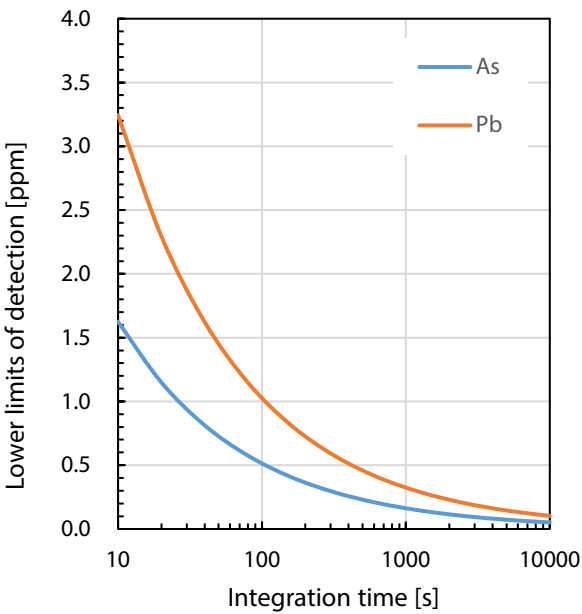


Fig. 4 Relationship between Integration Time and  
Lower Limits of Detection  
(a) Talc, (b) Titanium Oxide

## ■ Test Samples

- (1) Water-soluble titanium oxide
- (2) Oil-soluble titanium oxide
- (3) Water-soluble fine-particle titanium oxide
- (4) Oil-soluble fine-particle titanium oxide

## ■ Sample Preparation

Two grams of the sample was put into a sample container covered with a 5  $\mu\text{m}$  polypropylene film and simply compressed. Fig. 5 shows the sample image of (3) water-soluble fine-particle titanium oxide.



Fig. 5 (3) Water-Soluble Fine-Particle Titanium Oxide

## ■ Results of Quantitative Analysis

Table 4 shows the results of quantitative analysis. It is shown that this method can be used for the quantitative analysis of Pb and As in the raw powder of talc and titanium oxide.

Table 4 Results of Quantitative Analysis\*1 [ppm]

Sample	As	Pb
(1) Water-soluble titanium oxide	3.1	32.7
(2) Oil-soluble titanium oxide	3.2	5.1
(3) Water-soluble fine-particle titanium oxide	<0.5	5.6
(4) Oil-soluble fine-particle titanium oxide	<0.6	7.5

\*1 "<" is less than the lower limits of detection (theoretical  $3\sigma$ )

## ■ Profile

Fig. 6 shows the As and Pb profiles of the (1) water-soluble titanium oxide.

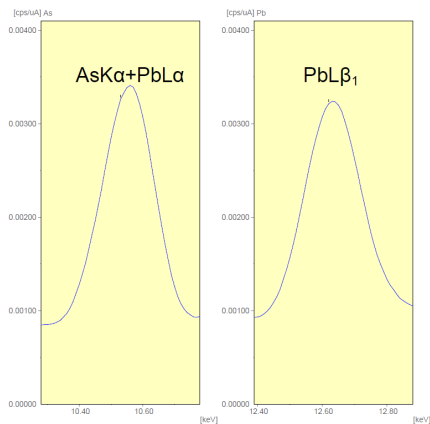


Fig. 6 The As and Pb Profiles of the (1) Water-Soluble Titanium Oxide

## ■ Results of Repeatability Test

Table 5 shows the results of simple 10 times repeatability test for (1) water-soluble titanium oxide.

Table 5 Results of Repeatability Test

Element	As	Pb
Mean value [ppm]	3.2	32.9
Standard deviation [ppm]	0.2	0.4
Coefficient of variation [%]	6.9	1.2

## ■ Conclusion

This application news reports on the quantitative analysis of lead (Pb) and arsenic (As) in two types of inorganic oxide powders: talc and titanium oxide. The analysis was performed using the energy-dispersive X-ray fluorescence spectrometer ALTRACE, which allows for the analysis of powders without dissolving them.

ALTRACE enables simultaneous analysis of Pb and As, unlike traditional colorimetric methods that test them separately. Additionally, ALTRACE can analyze 48 samples continuously, significantly improving the efficiency of the testing process.

## ■ Analysis Conditions

Table 6 Analysis Conditions

Instrument	:	ALTRACE
Elements	:	As (AsK $\alpha$ ), Pb (PbL $\beta_1$ )
Analysis group	:	Quantitative analysis
Analysis method	:	Calibration curve method
Detector	:	SDD
X-ray tube	:	Rh target
Tube voltage	:	50 [kV]
Tube current	:	Auto [ $\mu\text{A}$ ]
Primary filter	:	#5
Atmosphere	:	Air
Integration time*1	:	Talc : 100 [s] Titanium oxide : 300 [s]
Dead time	:	Max. 40 [%]

\*1 Since As and Pb are measured simultaneously, the integration time is the time per sample.

## <Related Applications>

- 1. [01-00775-EN](#) : Screening Analysis for Hazardous Heavy Metals in Foods and Food Additives

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### ➤ ALTRACE

Energy Dispersive X-ray Fluorescence Spectrometer

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