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ALTRACE™ Energy Dispersive X-ray Fluorescence (EDXRF) Spectrometer

Application News

Measurement of Residual Metal Catalysts by X-ray Fluorescence

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User Benefits

- ALTRACE can determine levels of heavy metals present in organic compounds at around 1 mg/kg in under 10 minutes per sample.
- Analyzing and managing levels of residual metal catalysts can be performed by simply placing samples in the sample vessel and analyzing them without the need for complex sample pretreatment.
- By increasing the X-ray tube power and optimizing the optical design, the sensitivity of ALTRACE for heavy metals has been dramatically improved compared to the previous model.

■ Introduction

Many industrial products we encounter every day are produced from organic compounds by manufacturing processes that utilize a range of synthesis reactions and metal catalysts. Catalysis can be classified as either homogeneous or heterogeneous (although drug and chemical production processes typically use homogeneous catalytic systems). While homogeneous catalysis allows for precise control over catalytic reactions, recovering the catalyst after the reaction can be difficult.

Nevertheless, managing the levels of residual catalysts in industrial products is critical to ensure safety and because of their high costs. For example, the ICH Q3D Guideline for Elemental Impurities (adopted in April 2017) requires risk assessments when materials such as metal catalysts are added intentionally during a production process.

Energy dispersive X-ray fluorescence (EDXRF) spectrometry offers a simple but effective technique for assessing the levels of residual metal catalysts. Normally, the lower limit of quantification for EDXRF spectrometric analysis of heavy metals is above 1 mg/kg, which limits its use in quantitative analysis. However, the ALTRACE is equipped with a high-power X-ray tube that dramatically improves its sensitivity for heavy metals, enabling their determination at 1 mg/kg and below in less than 10 minutes.

This Application News describes using ALTRACE to determine residual levels of a homogeneous catalyst after a synthesis reaction. The catalyst used in this analysis is palladium (Pd), which is a widely applied heavy metal catalyst, and the reaction catalyzed by the Pd catalyst is a cross-coupling reaction.

A metal scavenger and activated carbon were each used to remove the Pd catalyst from the reaction products. Metal scavengers are frequently used in this role, and activated carbon offers a relatively cheap method for removing catalysts.

■ Samples

(1) Samples for Analysis

1. Suzuki-Miyaura Cross Coupling Reaction Experiment Kit 2
2. SiliaMetS DMT metal scavenger (SiliCycle)
3. Activated Carbon

This analysis used an experiment kit containing reagents and catalysts that synthesize two fluorophores (fluorophore 1 and 2) in a Suzuki-Miyaura cross-coupling reaction. Palladium acetate was used as the catalyst, and the catalyst was removed using activated carbon and a metal scavenger that binds palladium.
Fig. 1 shows the sample preparation process and images of the samples at each stage of preparation.

A: Mix the catalyst with the reaction solution for each fluorophore (1 and 2)

Fluorophore	1	2
		
	Sample 1	Sample 2

B: Add the metal scavenger or activated carbon to each solution, leave for a given period, and then centrifuge, filter, and analyze the filtrate.

	Metal scavenger		Activated carbon	
Fluorophore	1	2	1	2
				
	Sample 3	Sample 4	Sample 5	Sample 6

C: Recover and analyze the powder already isolated by centrifugation.

	Metal scavenger		Activated carbon	
Fluorophore	1	2	1	2
				
	Sample 7	Sample 8	Sample 9	Sample 10

Fig. 1 Sample Preparation Process and Images of Samples

(2) Calibration Curve Samples
Reference solution for atomic absorption spectrometry (FUJIFILM Wako Pure Chemical Corporation)
Six calibration curve samples were prepared at 0 (pure water blank), 1, 5, 10, 20, and 100 mg/kg.

■ Target Element

$_{46}$Pd

■ EDXRF Spectrometry

Samples were prepared for analysis by placing them in a sample vessel covered with a 5-µm thick polypropylene film.

The calibration curve obtained is shown in Fig. 2, and its correlation coefficient and accuracy are shown in Table 1. Its accuracy was good at 0.2 mg/kg or lower (Table 1). A scattering X-ray internal standard was used to correct for material properties.

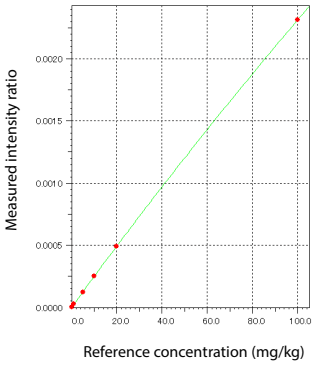


Fig. 2 Calibration Curve

Table 1 Calibration Curve Data

Coefficient of Correlation	0.9999
Accuracy (mg/kg)	0.12

■ Quantitative Results

The calibration curve was used to determine levels of Pd at each step in the following sample preparation process: in the reaction mixture before removing the catalyst (A: samples 1 and 2), in the filtrate recovered after the catalyst was removed (B: samples 3 to 6), and in the powder recovered after the catalyst was removed (C: samples 7 to 10). The results of this analysis show that the metal scavenger and the activated carbon were effective in trapping and removing the palladium catalyst (Table 2).

Table 2 Results of Quantitative Analysis

[mg/kg]

(Catalyst Removal)	Fluorophore 1		Fluorophore 2	
	Metal scavenger	Activated carbon	Metal scavenger	Activated carbon
A: Reaction Mixture before Catalyst Removal	56.4 (Sample 1)		56.2 (Sample 2)	
B: Filtrate Recovered after Catalyst Removal	0.80 (Sample 3)	0.49 (Sample 5)	0.60 (Sample 4)	1.96 (Sample 6)
C: Powder Recovered after Catalyst Removal*1 (Reference)	437 (Sample 7)	1444 (Sample 9)	499 (Sample 8)	1312 (Sample 10)
Lower Limit of Quantitation*2	0.27			

*1 The Pd concentrations in (C) are higher than in (A) because the total volume of (A) is larger, so a smaller amount of metal scavenger and activated carbon was used in (C) to capture the Pd.

*2 Lower limit of quantitation: Calculated by measuring a blank sample (pure water) 10 times in a repeatability test.

■ Repeatability Test Results

Table 3 shows the results of a repeatability test that measured a 1 mg/kg Pd sample 10 times.

Despite a Pd concentration of just 1 mg/kg, the coefficient of variation was only 2.3 %, which shows the analysis was highly accurate (Table 3).

Table 3 Repeatability Test Results

[mg/kg]

Element	Pd
Mean	1.009
Standard Deviation	0.023
Coefficient of Variation (%)	2.3

■ Analysis Conditions

The analysis conditions are shown in Table 4.

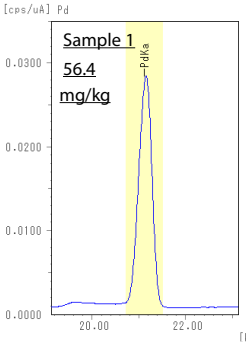
Table 4 Analysis Conditions

Equipment	ALTRACE	
Element	Pd (NET)	Pd (BG)
Analysis Group	Calibration curve method	
Detector	SDD	
X-ray Tube	Rh target	
Tube Voltage	65 kV	
Tube Current	Auto (μA)	
Primary Filter	#1	None
Atmosphere	Air	
Integration Time	300 sec	100 sec
Dead Time	Max. 40 (%)	

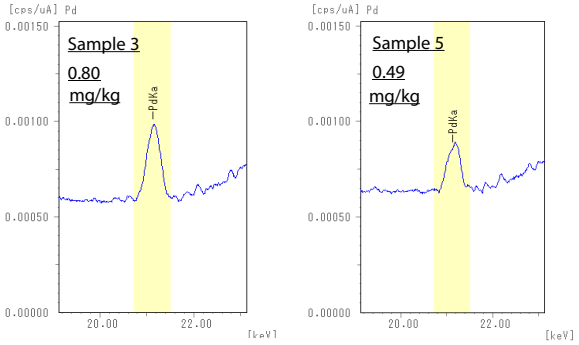
■ Qualitative Signal Profile

Fig. 3 shows the qualitative signal profiles for fluorophore 1 at each step of the process (A to C). Clearly recognizable peaks are visible even at concentrations below 1 mg/kg (Fig. 3, samples 3 and 5).

A: Reaction mixture before catalyst removal



B: Filtrate recovered after catalyst removal



C: Powder recovered after catalyst removal

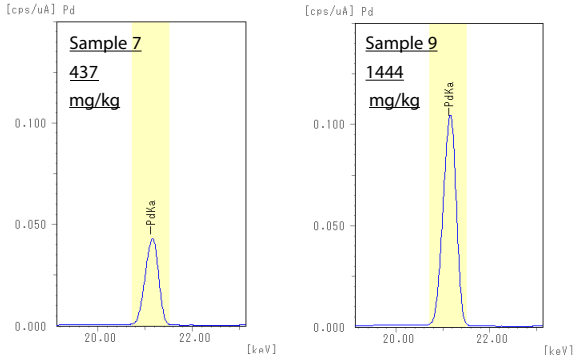


Fig. 3 Qualitative Signal Profiles

■ Conclusion

Common methods of elemental impurity analysis, such as atomic absorption spectrometry (AAS), ICP atomic emission spectroscopy (ICP-AES), and ICP mass spectrometry (ICP-MS), require liquid samples, so solid and powder samples must be prepared into a solution prior to analysis. However, EDXRF spectrometry can determine levels of elemental impurities regardless of the sample phase or form (solution, powder, etc.), provided the target element is distributed uniformly throughout the sample.

The sensitivity of ALTRACE for heavy metals has been substantially improved compared to previous models, allowing for rapid quantitative determination of heavy metals at 1 mg/kg and below. Based on the performance presented in this article, ALTRACE is an effective tool for managing residual levels of metal catalysts.

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1. X-Ray Fluorescence Analysis of Residual Catalysts
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