

Application News

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Spectrophotometric Analysis

Quantifying "Silent Change" Using EDXIR-Analysis Software: EDX-FTIR Contaminant Finder/Material Inspector

The act of changing raw materials without notifying business partners for the purpose of reducing costs is known as "silent change."

Since products manufactured with non-standard raw materials cannot be guaranteed in terms of quality and usage of such materials can lead to incidents, this has become a social issue. The management of safe and good quality raw materials is indispensable in the manufacture of high quality products.

The EDXIR-Analysis software features a data comparison function. EDX data or FTIR data, or both, can be used to quantify the differences between genuine products and test products in terms of similarity. This function facilitates verification of raw materials as standard materials and proves effective in acceptance inspections, sampling inspections, and primary screening.

In this article, we introduce an example analysis that utilizes the data comparison function.

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Analysis of Plastic Contaminated with Toxic Elements

In order to use the data comparison function, genuine product data is registered into the library first. Next, after a comparison is performed between the genuine product data and test product data, the similarity and comparisons of the respective element contents, profiles, and EDX images of the EDX analysis data and the similarity of FTIR spectra of the FTIR analysis data are displayed. The following data comparison was performed for a genuine product and test product of polyvinyl chloride (PVC) plastic. The results are indicated in Fig. 1 and 2.

The similarities determined using the data comparison function were 0.8332 with EDX data and 0.8680 with FTIR data, which resulted in a composite similarity of 0.8506. Similarity is displayed in a 0 to 1 range and higher values indicate greater similarity between data. In other words, a value close to 1 is obtained when the components in the samples for comparison are equivalent. However, we can conclude that the sample tested here may include different raw materials due to the resulting composite similarity of 0.8506.

The EDX profile and FTIR spectrum of the test product also indicate that it contains lead (Pb) and components derived from acrylic (indicated with stars), which are not detected in the genuine product.



Fig. 2 Comparison of FTIR Spectra



Fig. 1 Data Comparison Result and EDX Profiles

Analysis of Elemental Content Differences in Plastic Materials

RoHS compliant ERM-EC680 and ERM-EC681 polyethylene reference materials were selected for data comparison as a genuine product and test product respectively. Qualitative and quantitative analysis was performed with EDX and a single reflection ATR attachment was used to measure their infrared spectra. The results are indicated in Fig. 3 and 4 and the instruments and analysis conditions are listed in Table 1.

By overlapping the EDX profiles and FTIR spectra respectively of the genuine product and test product, we can see that while there are content differences for S, Cl, Cr, Zn, Br, Cd, Sn, and Sb, there is no difference with respect to plastic (main component).



Fig. 3 Comparison of EDX Qualitative Profiles



Fig. 4 Comparison of FTIR Spectra

The similarities determined using the data comparison function were 0.9616 with EDX data and 0.9830 with FTIR data, which resulted in a composite similarity of 0.9723 that suggests a difference in materials.

Table 2 indicates the result of determining whether there is a significant difference in similarity between genuine products and between a genuine product and test product, based on the repeatability accuracy observed through multiple comparisons. The EDX data results show a significant difference whereas the FTIR data results do not, which supports the assumption that the elemental content of the test product differs from the genuine product.

Table 1	Instruments	and Analysis	Conditions
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[EDX]				
Instrument	: EDX-8000			
X-ray Tube	: Rh target			
Voltage/Current	: 15 kV (C-Sc), 50 kV (Ti-U)/Auto			
Atmosphere	: Air			
Measurement Diameter	: 10 mm φ			
Primary Filter	: Without (Ti-U, C-Sc), #1 (Rh-Cd),			
	#2 (S-Ca), #3 (Cr-Fe), #4 (Zn-As, Pb)			
Integration Time	: 30 sec (without Primary Filter)			
	60 sec (with Primary Filter)			
	[FTIR]			
Instruments	: IRAffinity-1S, MIRacle10 (Diamond prism)			
Resolution	: 4 cm ⁻¹			
Accumulation	: 40			
Apodization	: Happ-Genzel			
Detector	: DLATGS			

Table 2	Similarity	Calculation	Result
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	EDX		FTIR	
n	Genuine product	Test product	Genuine product	Test product
1	0.9969	0.9616	0.9790	0.9830
2	0.9948	0.9613	0.9800	0.9800
3	0.9962	0.9613	0.9830	0.9810
4	0.9966	0.9612	0.9820	0.9830
5	0.9960	0.9613	0.9860	0.9790
Average	0.9961	0.9613	0.9820	0.9812
Standard deviation	0.0008	0.0002	0.0027	0.0018
Significant difference *	Yes		No	

* According to t-test (significant level: 5%) Using commercially-available spreadsheet software

Summary

This time we introduced an example in which genuine products and test products were analyzed by utilizing the data comparison function of the EDXIR-Analysis software. By not only checking data visually but also quantifying them, differences between samples were easily distinguished.

Use of both EDX and FTIR instruments allows a multifaceted approach by enabling analysis of both organic and inorganic substances and assists in the risk management of raw materials in relation to safety. This software enables linkage and storage of various data as electronic files and provides powerful support in developing measures against silent change.

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