

Application News

No. A537

Spectrophotometric Analysis

Introducing the EDXIR-Holder: Sample Holder/Stocker for Contaminant Measurement

In contaminant analysis, an effective technique is to combine EDX, which is suited to the elemental analysis of metals and inorganic compounds, and FTIR, which is suited to analysis of polymeric materials and organic substances. Both of these methods are ideal for contaminant analysis since they allow nondestructive analysis and can be performed very quickly and easily. This article introduces the EDXIR-Holder, which can be used for holding and storing contaminant measurement samples, and facilitates efficient EDX analysis and FTIR analysis. Use of the EDXIR-Holder simplifies handling and storage of samples that have been troublesome to manage until now.

S. Iwasaki

Overview of EDXIR-Holder

As shown in Fig. 1 (a), the EDXIR-Holder is an openable case that combines an adhesive layer of film (35 μm thick) for adhering samples and a polypropylene film (5 μm thick) for fluorescent X-rays (EDX).

With the sample adhered to the adhesive layer of film, FTIR analysis is performed with the case opened and the ATR prism pressed directly onto the sample (Fig. 1 (b)). When performing EDX analysis, the case is closed and set with the polypropylene film side facing down toward the X-ray irradiation source (Fig. 1 (c)). The EDXIR-Holder saves labor and leads to efficiency in analysis tasks because the sample exchange process is kept to a minimum.

Furthermore, samples can be stored in the case after measurement. With a size of only about 5 cm \times 5 cm when closed, the case is compact and the sample name can be written on the white frame. Since there is no need to move samples to other containers or bags, there is little risk of losing samples.

The recommended contaminant sample sizes are from 1.5 mm to 5.0 mm, sample thicknesses from 0.5 mm to 1.5 mm for EDX and 0.1 mm to 4.0 mm for FTIR, and sample weights of 1 g or less. Samples can be held very easily especially for EDX analysis, as long as the sample size and weight is within the above stated ranges, simply by placing the sample in the holder. However, be careful of samples with protrusions as they may puncture the film.

Example of Contaminant Analysis

A contaminant detected on a production line was held using the EDXIR-Holder and then FTIR and EDX analyses were performed.

Fig. 2 shows the ATR spectrum obtained by FTIR analysis and the library search results. The contaminant is presumed to be a polyvinylchloride (PVC) mixture that contains phthalate ester and calcium carbonate.

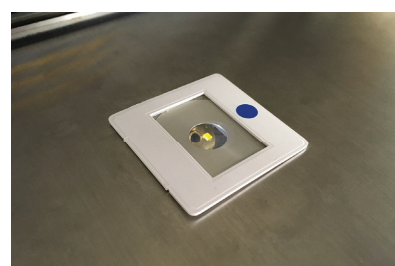
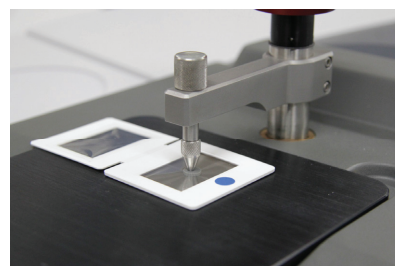
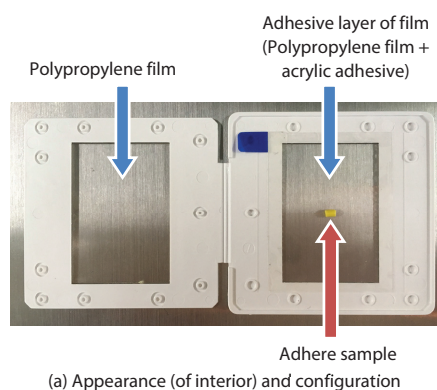


Fig. 1 Overview of EDXIR-Holder

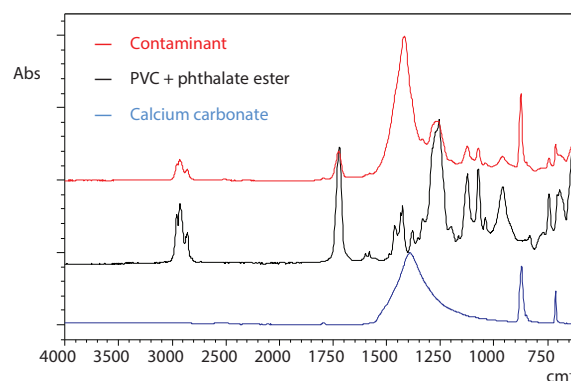
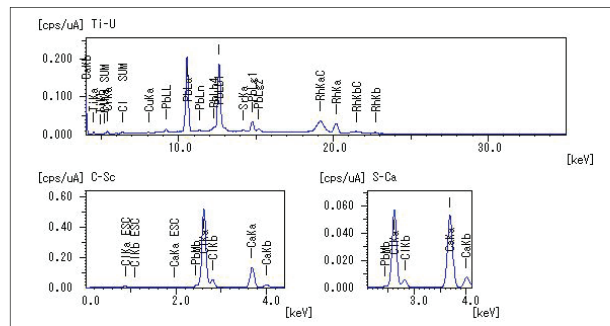


Fig. 2 ATR Spectrum of Contaminant and Search Results: FTIR

Fig. 3 shows the ${}^{6}\text{C}-{}^{92}\text{U}$ qualitative and quantitative analysis results obtained by EDX analysis. Based on the FTIR results, the main component PVC (C_2H_3) was quantified as the balance (residue) ^{*1} component. The contaminant contains Cl and Ca, and as presumed by FTIR analysis, presence of PVC and calcium carbonate was confirmed. We found that the contaminant also contained Pb, which could not be detected by FTIR analysis.



Component	Cl	Ca	Pb	Ti	Cr
Quantitative value (%)	25.14	11.43	0.60	0.062	0.040

Component	Sr	Cu	C_2H_3
Quantitative value (%)	0.005	0.003	62.73

Fig. 3 Qualitative and Quantitative Analysis Results of the Contaminant: EDX

Precautions for Measurement

The EDXIR-Holder is capable of holding samples from 1.5 mm to 5.0 mm in size and weighing 1 g or less. Since the diameter of ATR prisms used in FTIR analysis is commonly around 1.5 mm, using the holder for analysis of any smaller, finely fibrous, or thin samples is likely to result in overlapping of the ATR spectrum of the film's acrylic adhesive layer with the ATR spectrum of the measurement target.

Fig. 4 shows the ATR spectrum measured by holding a piece of polyethylene (PE) film with a diameter of about 0.3 mm using the EDXIR-Holder and the library search result. The top search result was ethylene acrylic acid (EAA), which is incorrect. Then we calculated the difference spectrum between the film piece and acrylic adhesive layer and performed a library search on the difference spectrum. As shown in Fig. 5, the search result was PE, which is the material of the film piece. This demonstrates that care must be taken when analyzing minute samples that are easily affected by the acrylic adhesive layer.

In order to avoid such erroneous results, Shimadzu recommends setting samples so that they cover the entire surface of the ATR prism. For minute samples, either perform analysis without using the EDXIR-Holder or obtain the ATR spectrum of the acrylic adhesive layer and use the difference spectrum to perform qualitative analysis, as presented here. However, please note that identification using difference spectra may not be possible when peaks originating from the sample are miniscule in the obtained ATR spectrum.

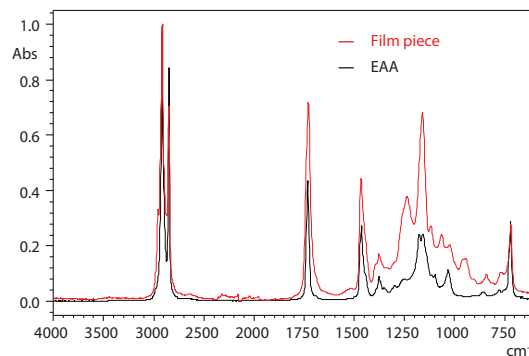


Fig. 4 ATR Spectrum of Film Piece and Search Result: FTIR

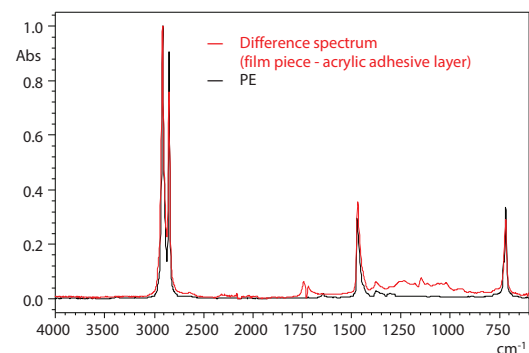


Fig. 5 Difference Spectrum and Search Result: FTIR

Conclusion

The EDXIR-Holder introduced in this article is an original Shimadzu product that incorporates our extensive experience regarding contaminant analysis and serves as a very effective tool when performing both EDX analysis and FTIR analysis. Even when used for these analyses individually, it proves to be simple to use and saves labor in analysis tasks.

Regarding data analysis, the EDX-FTIR contaminant finder/material inspector EDXIR-Analysis software is capable of analyzing EDX and FTIR data either together or separately ^{*2} ^{*3}. Shimadzu recommends utilizing EDXIR-Holder together with this software for effective and efficient analysis.

Table 1 Instrument and Analysis Conditions

Instrument	: EDX-8000
X-ray Tube	: Rh target
Voltage/Current	: 15 kV (C-Sc, S-Ca), 50 kV (Ti-U)/Auto
Atmosphere	: Vacuum
Measurement Diameter	: 1 mm φ
Primary Filter	: Without (Ti-U, C-Sc), #2 (S-Ca),
Integration Time	: 30 sec (without Primary Filter) 60 sec (with Primary Filter)
Instruments	: IRAffinity-1S, MIRacle10 (Diamond prism)
Resolution	: 4 cm^{-1}
Accumulation	: 40
Apodization	: Happ-Genzel
Detector	: DLATGS

References

- *1 Shimadzu Application News No. X255
- *2 Shimadzu Application News No. A522A
- *3 Shimadzu Application News No. A527