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Application News

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Spectrophotometric Analysis

Contaminant Analysis Using EDXIR-Analysis Software for Combined EDX-FTIR Analysis

When contaminants are generated, it is important to quickly identify them, ascertain the source, and take measures against further occurrence. Energy dispersive X-ray spectroscopy (EDX) and Fourier transform infrared spectroscopy (FTIR) are very effective techniques for the identification of contaminants. Using EDX and FTIR together provides a simple method of obtaining information on both the elements and compounds present in a contaminant. However, making a comprehensive assessment of sets of EDX and FTIR data to identify a contaminant requires substantial experience and knowledge.

EDXIR-Analysis has been recently developed by Shimadzu as software specially designed to identify contaminants to a high degree of accuracy, and contains within it a database of actual contaminant samples and the cumulation of Shimadzu's expertise. In this article, we will describe an example contaminant analysis and in doing so demonstrate the functions and features of EDXIR-Analysis software.

■ EDXIR-Analysis Software

EDXIR-Analysis includes a contaminant library* with a wealth of data on 485 items, including both EDX and FTIR analysis results. Differing from other commercially available libraries that include data on pure materials, the EDXIR-Analysis database includes mixed materials such as packing materials and actual contaminant samples (provided by water supply agencies, food sector companies, etc.), vastly improving the accuracy of retrieved results. The database is registered with EDX qualitative profiles and quantitative analysis results, FTIR infrared spectra (single reflection ATR measurements), and qualitative results estimated based on both EDX and FTIR data. The database also includes detailed information on sample characteristics, such as images, color, shape, hardness, and metallic luster.

The home screen of EDXIR-Analysis is shown in Fig. 1. From the home screen, select (1) to perform a combined analysis of data obtained by EDX and FTIR, and select (2) or (3) to perform an analysis of data obtained by either EDX or FTIR. Data and sample analysis is performed simply by following the instructions on subsequent windows and uploading the analytical data.

A flow chart of this combined analysis is shown in Fig. 2. EDXIR-Analysis uses intensity of scattered X-rays to broadly divide materials of measured samples into one of three groups: inorganic, mixed (inorganic and organic), and organic. Then, a proprietary algorithm is used to perform combined analysis of EDX and FTIR data. If data from a sample does not match any library data, it can be added to the database as new data to build on the proprietary database and thereby increase the accuracy of retrieved results.

We used EDXIR-Analysis to perform an analysis of three contaminants. Instruments and analytical conditions are shown in Table 1.
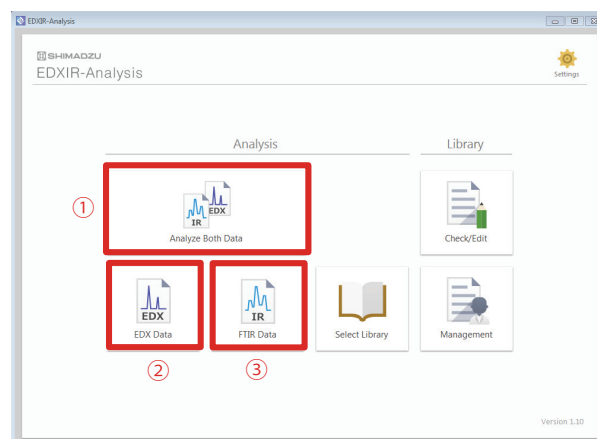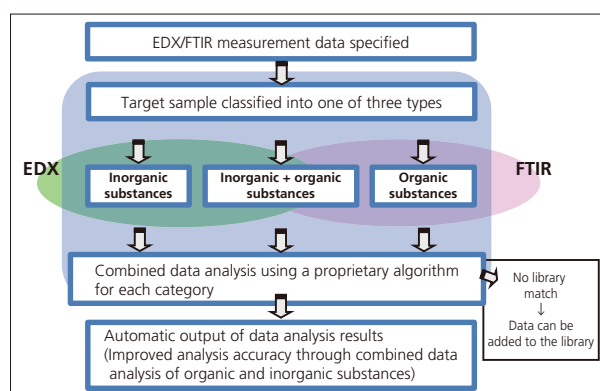

Fig. 1 EDXIR-Analysis Home Screen

Fig. 2 Flowchart of Combined Analysis

Table 1 Instruments and Analytical Conditions

[FTIR]

Instruments	: IRAffinity-1S, MIRacle10 (Germanium prism/Diamond prism)
Resolution	: 4 cm⁻¹
Accumulation	: 40
Apodization	: Happ-Genzel
Detector	: DLATGS

[EDX]

Instrument	: EDX-8000
X-Ray Tube	: Rh target
Voltage / Current	: 15 kV (C-Sc, S-K), 50 kV (Ti-U) / Auto
Atmosphere	: Vacuum
Measurement Diameter	: 1 mm ϕ / 3 mm ϕ
Integration Time	: 100 sec/ch

* For FTIR, the contaminant library can also be used with LabSolutionsIR, the FTIR analysis software. EDX data can be viewed using PDF files.

■ Contaminant Analysis: ① Combined Analysis

Contaminant A was collected from a tap water filter during periodic cleaning. An image of contaminant A is shown in Fig. 3.

Fig. 3 Contaminant A

Contaminant A was analyzed with both EDX and FTIR based on the possibility that it might contain both inorganic and organic constituents, and the results were analyzed using EDXIR-Analysis.

The sample profile and hit list from the EDX Data tab page are shown in Fig. 4. The window is structured as follows:
① Elements present in sample data and hit data (EDX)
② Profile of sample data and hit data (EDX)
③ Images of sample data and hit data
④ Hit list (EDX/FTIR)
⑤ Detailed hit data (EDX/FTIR)
⑥ Detailed sample data (EDX/FTIR)

Sample data refers to the analytical results obtained from the actual sample, and hit data refers to data matched from the library.
Selecting the EDX photo tab (Fig. 4, ③) reveals images of the sample, as shown in Fig. 5. Selecting the icon in the Details row for the hit No. 1 on the hit list (Fig. 4, ⑤) displays windows showing sample information, as shown in Fig. 6. These figures show that the sample images and data and the hit data are well matched. First on the hit list is an acrylonitrile butadiene rubber (NBR) that contains calcium carbonate and zinc stearate. Agreement between sample and hit data is represented numerically from 0 to 1, with a higher number showing a greater match between sample and hit data. The analysis gave a good match result of 0.9299.
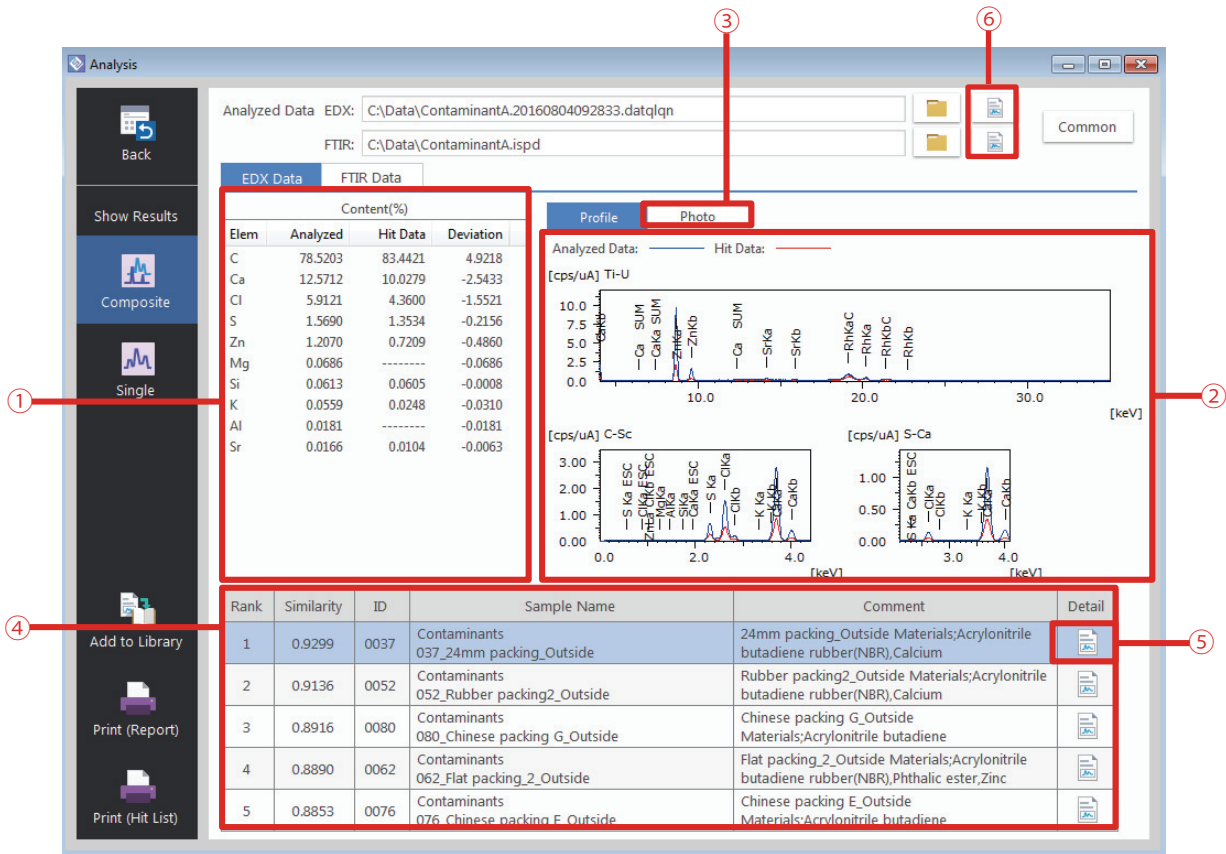

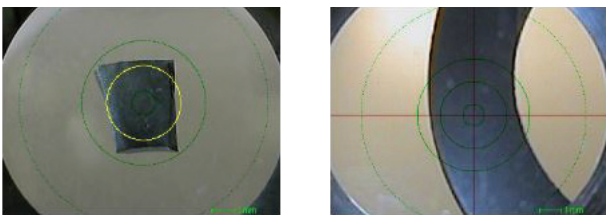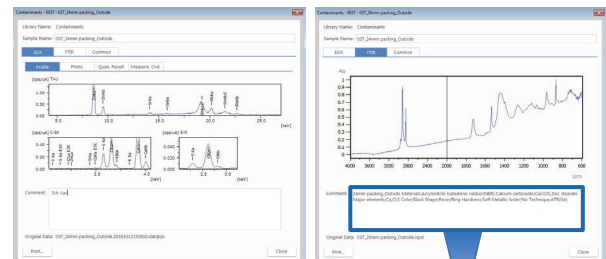**Fig. 4 EDX Profiles and Hit List**

Fig. 5 Images of Sample (left) and Hit Data (right)

Hit Data Information (Hit No.1)
24 mm packing_Outside Materials; Acrylonitrile butadiene rubber (NBR), Calcium carbonate (CaCO3), Zinc stearate
Major elements; Ca, Cl, S Color; Black Shape; Resin/Ring Hardness; Soft Metallic luster; No Technique; ATR (Ge)

Fig. 6 Detailed Hit List Data

The hit list and infrared spectrum from the FTIR Data tab page are shown in Fig. 7. The window is structured as follows:

① Spectra from sample data and hit data (FTIR)
② Add to Library button
③ Print button

So far, the results of this combined EDX and FTIR analysis show that contaminant A closely resembles the packing that appears highest on the hit list.

Sample data can be added to the library simply by selecting the ② Add to Library button, allowing the user to build a custom database (user library). Image files showing the sample and PDF format document files can also be added to the library along with data files. Adding to the library is also an effective means of grouping together different data formats and storing them electronically.

A report can also be printed simply by selecting the ③ Print button, as shown in Fig. 8. Reports can be stored in MS Word format as well as the standard format**.
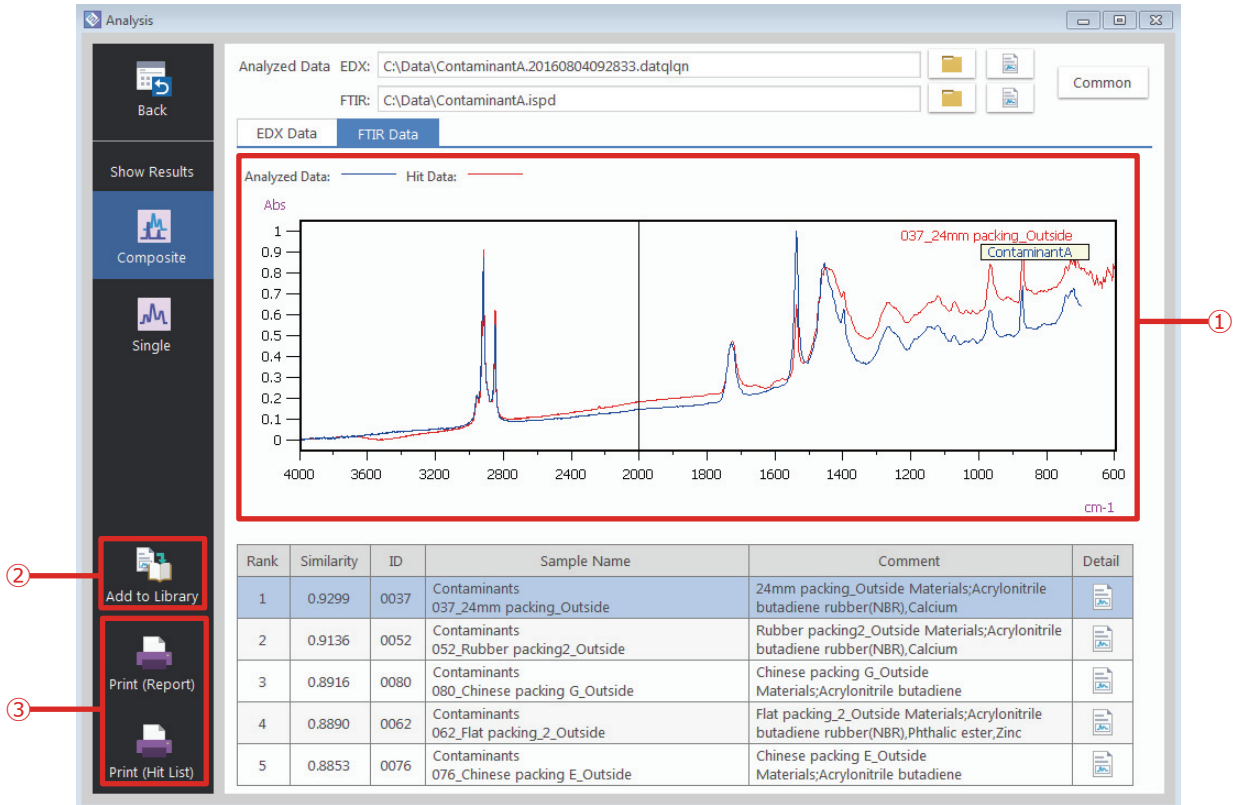** Requires installation of Microsoft Office.
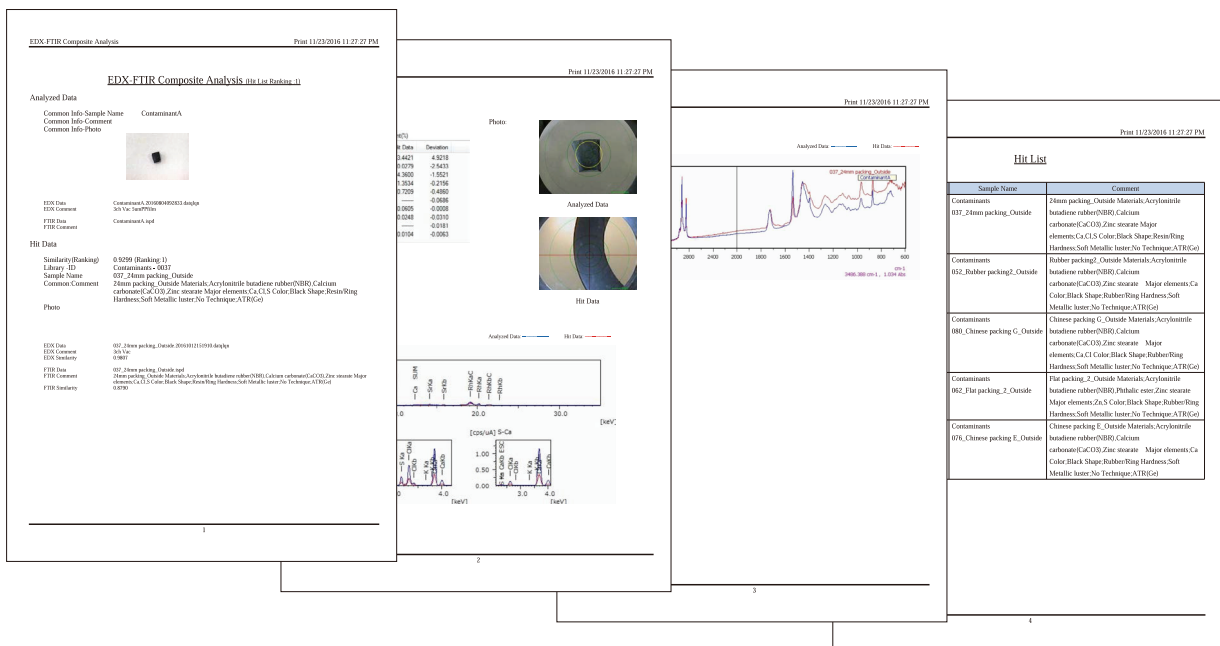

Fig. 7 Infrared Spectra and Hit List


Fig. 8 Example Printed Report

■ Contaminant Analysis: ② Single Analysis (EDX)

An image of contaminant B is shown in Fig. 9. Based on its metallic luster, the contaminant was presumed to be inorganic, and analysis was performed using EDX. Fig. 10 shows the EDX profile, hit list, and library sample image, while Fig. 11 shows the EDX qualitative profiles for contaminant B and hit data. The biggest hit is a metal pad (stainless steel).

Hit data includes EDX data in the form of qualitative profiles and quantitative analysis results, as well as infrared spectra from FTIR data. No significant peaks appeared in the infrared spectrum obtained from hit data for contaminant B. This shows that highly accurate results can also be obtained with just a single analysis (EDX).

Fig. 9 Contaminant B
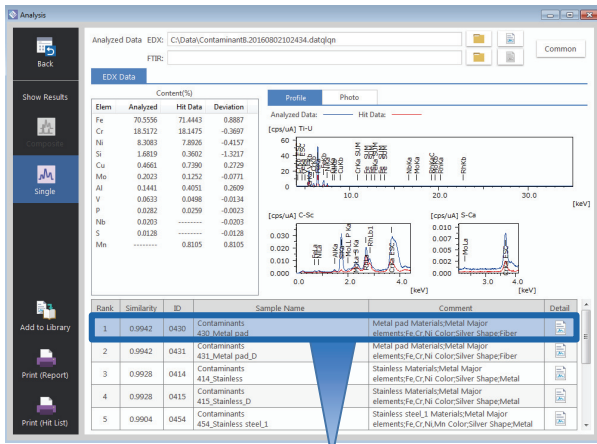
Hit Data Information
Metal pad Materials; Metal Major elements; Fe, Cr, Ni Color; Silver Shape; Fiber Hardness; Soft Metallic luster; Yes Technique; ATR (Ge)

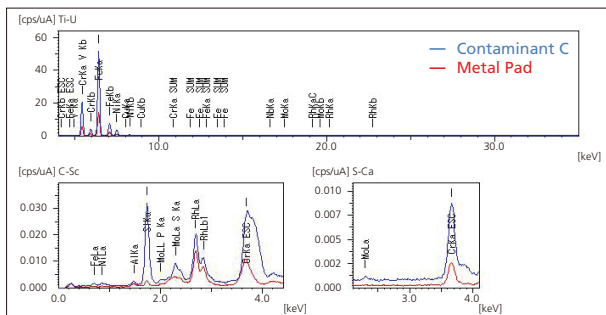**Fig. 10 EDX Profiles, Hit List (top) and Library Sample Image (bottom)**

Fig. 11 EDX Profiles

■ Contaminant Analysis: ③ Single Analysis (FTIR)

An image of contaminant C is shown in Fig. 12. The contaminant was presumed to be organic based on its external appearance, and analysis was performed using FTIR. Fig. 13 shows the infrared spectra, hit list, and library sample image. The biggest hit was polypropylene. The major element is present at no more than 1 % according to the hit data for contaminant C. Thus, based on the hit data, it is possible to view infrared spectra from FTIR data as well as element information obtained by EDX.

Fig. 12 Contaminant C
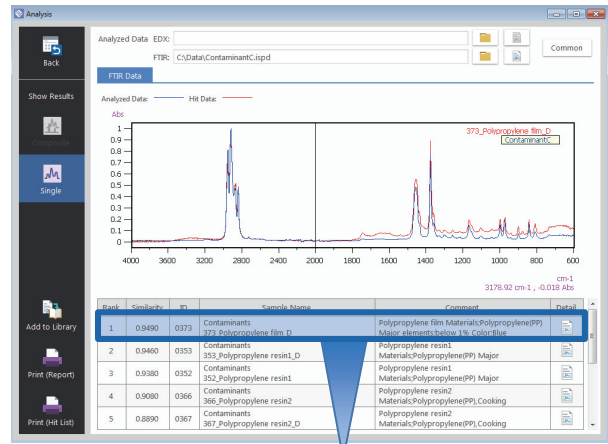
Hit Data Information
Polypropylene film Materials; Polypropylene (PP) Major elements; below 1% Color; Blue Shape; Fragment Hardness; Soft Metallic luster; No Technique; ATR (Diamond)

Fig. 13 Infrared Spectra, Hit List (top) and Library Sample Image (bottom)

■ Conclusion

In this study, we could successfully perform a combined analysis of analytical data obtained by EDX and FTIR both quickly and simply by using EDXIR-Analysis. EDXIR-Analysis includes Shimadzu's own original database that contains a wealth of data that can also be easily added to by the user to create a user library. Reports can also be created to summarize analytical results, and electronic files can be grouped and stored for each data group.

EDXIR-Analysis, which incorporates the expertise of Shimadzu, provides more powerful support for contaminant analysis.

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