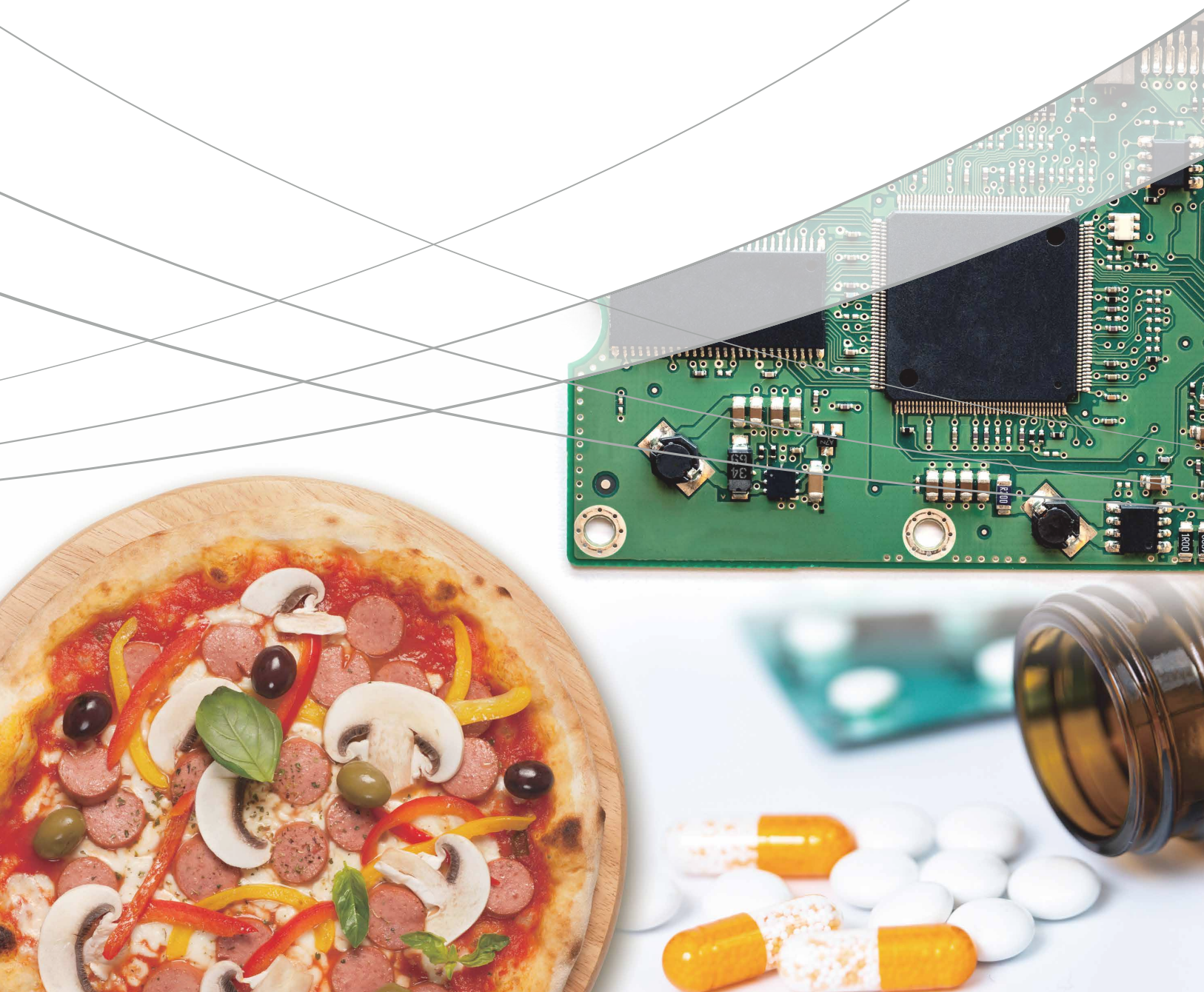
C10G-E099

SHIMADZU
Excellence in Science

Solutions for Contaminant Analysis

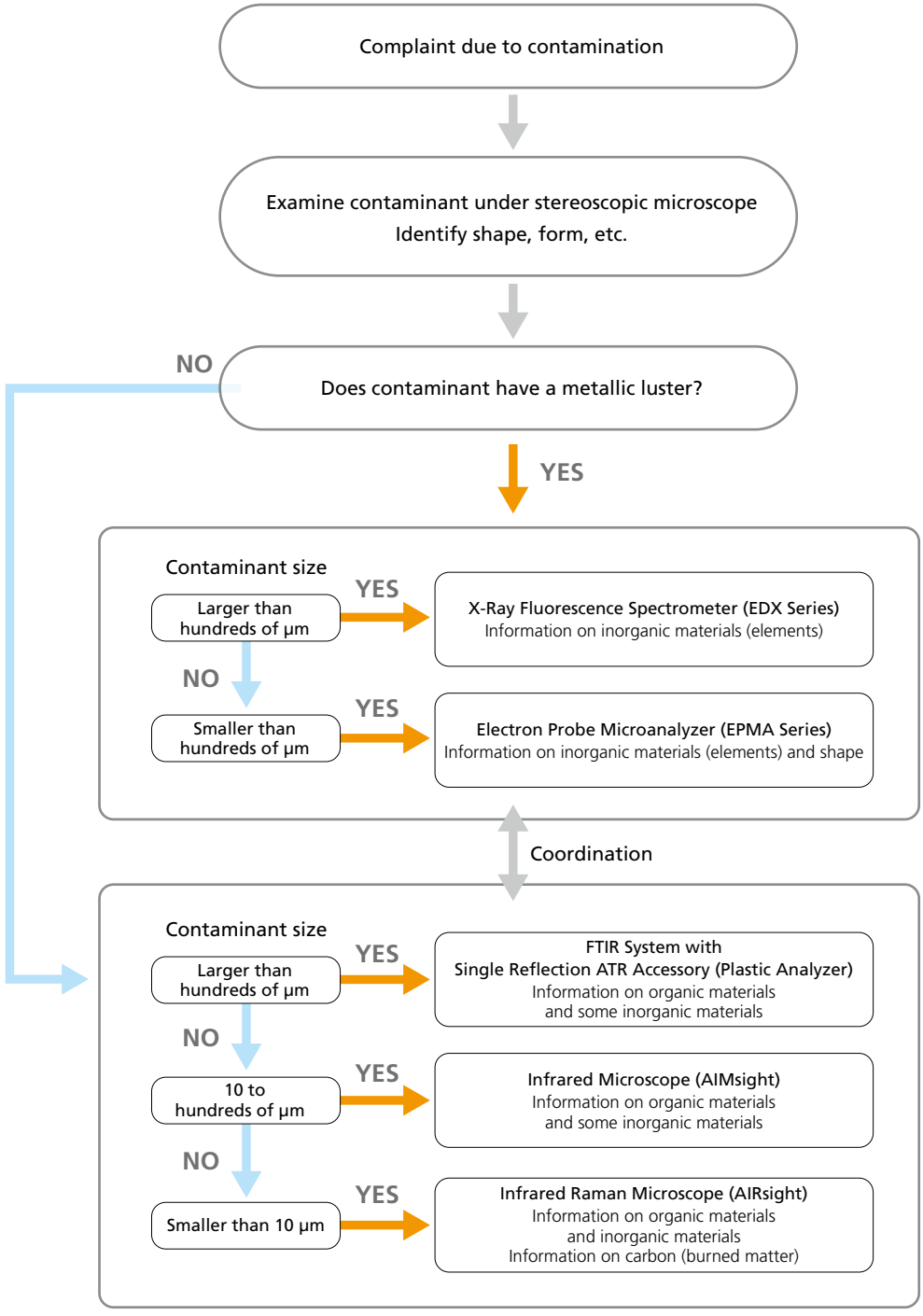


Total Support for Contaminant Analysis and Failure Identification

The adulteration of products by contaminants causes a wide range of problems in every sector of industry. These problems can only be resolved by analyzing the contaminating material and identifying its source. Shimadzu offers a powerful range of products and tools for contaminant analysis and failure analysis.

Contaminant Analysis Flowchart

Contaminant analysis can be undertaken by a variety of methods, and it is vital to select an analytical method best suited to the shape and size of the contaminant. The flowchart below summarizes this process of identifying the contaminant and selecting the appropriate analytical system. Follow this flowchart to select the optimum system for your contaminant analysis.



Energy Dispersive X-Ray Fluorescence Spectrometers (EDX)

When a material is irradiated with X-rays, each element in the material generates unique X-rays (X-ray fluorescence). The elements and the concentrations of those elements in the material can be determined based on the energies of these X-rays and their intensity. EDX systems can analyze samples in various forms with no chemical pretreatment, including solid, liquid, and powder samples, and are ideal for contaminant analysis when preserving the sample is a priority.

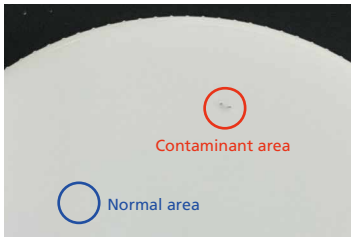
EDX-7200 / 8100

These flagship models in the Shimadzu EDX series are equipped with a liquid nitrogen-free, high-performance semiconductor detector, a collimator, and sample camera effective for the analysis of very small samples and very small sample quantities, and also support helium purge analysis and vacuum analysis (optional) for highly sensitive measurement of light elements.

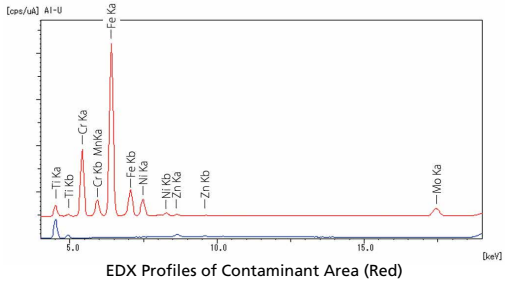
EDX-7200 / 8100

Analysis of a Microscopic Metal Contaminant on a Molded Plastic Product

EDX systems are non-destructive and can perform elemental analysis, thus are effective for analyzing contaminants that are mixed with or adhered to foods, pharmaceuticals, and other products. The sample camera and collimator allow for easy characterization of very small contaminants, and the irradiation field diameter can be adjusted to match the sample size, reducing the influence of the surrounding material. In the example below, these system features enabled quantitative data to be accurately matched with reference data and the contaminant to be identified as SUS316 stainless steel.



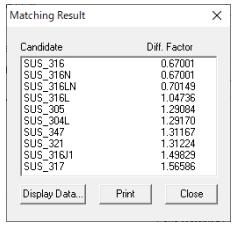
View of Sample



EDX Profiles of Contaminant Area (Red) and Normal Area (Blue) Superimposed

Analyte	Result	
Fe	66.443	%
Cr	17.865	%
Ni	11.254	%
Mo	2.433	%
Mn	2.005	%

Results from Quantitative Analysis of Contaminant by FP Method
Titanium (Ti) and Zinc (Zn) were detected in the material around the contaminant and excluded from quantitative calculations.



Results of Data Matching
(Collected data was compared against an internal library, identifying the contaminant as SUS316)

A Metal Particle (Approx. 0.1 mm Diameter) Attached to a Piece of Confectionery

A metal particle approx. 0.1 mm in diameter was attached to a piece of confectionery and analyzed with irradiation field diameters of 1 mm and 0.3 mm. The 1 mm field diameter resulted in a larger overall background effect due to irradiation scatter from the area surrounding the metal particle (from the confectionery) and a poorer S/N ratio, while the 0.3 mm field diameter reduced the X-rays scattered by the surrounding area and produced in an EDX profile with a good S/N ratio. Copper (Cu) and zinc (Zn) were the main components detected at both irradiation field diameters and the metal particle was identified as brass at both irradiation field sizes, but the 0.3 mm field size also detected a lead (Pb) peak, which suggested the metal particle was free cutting brass. This shows the smaller irradiation field diameter of 0.3 mm offers a more accurate analysis of small contaminants surrounded by organic and other materials that generate large amounts of scattered X-rays.

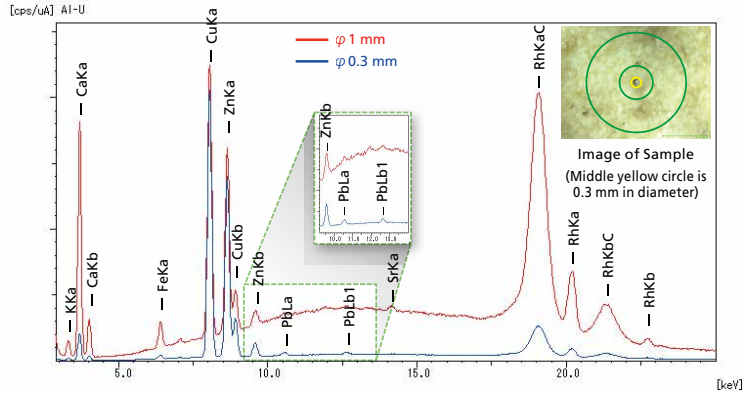


Image of Sample
(Middle yellow circle is 0.3 mm in diameter)

Fourier Transform Infrared Spectrophotometers (FTIR)

Every material has a unique infrared absorption spectrum, hence contaminants can be identified and characterized by measuring their infrared absorption spectrum with an FTIR system and comparing it against library data. This process can take several seconds to several minutes and is a powerful tool in the analysis of organic substances.

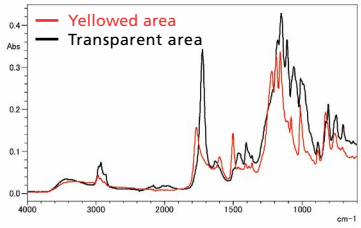
FTIR System with Single Reflection ATR Accessory

The single reflection ATR accessory almost entirely removes the need for sample pretreatment before FTIR analysis. Measurements can be taken while simply holding the object of interest in contact with the approx. 1.5-mm diameter prism.
Measurements can be collected directly from samples in a variety of forms, including powders, films, bulk samples, liquids, and surface deposits.

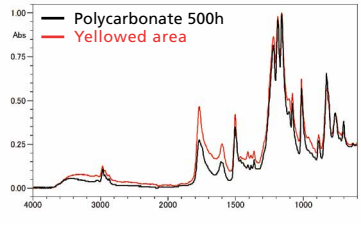
Plastic Analyzer (Uses IRSpirit FTIR Unit)

Analysis of a Degraded Automobile Headlight Cover

The Plastic Analyzer system was used to analyze an automobile headlight cover with yellow discoloring. The yellow-discolored area was near the middle of the cover where the cover was exposed to the outdoor environment. Comparing the absorption spectrum of the yellowed area and a transparent area of the cover was expected to reveal additional infrared absorption in the transparent area compared to the yellowed area. A UV-Damaged Plastics Library is included as standard with the Plastic Analyzer, and a search of this library identified the yellowed area of the cover as degraded polycarbonate (PC). Although headlight covers are often made from PC because it is a mechanically strong material that tends not to shatter when broken, PC is also vulnerable to ultraviolet radiation.



Headlight Cover Infrared Spectra
(Black line: transparent area, red line: yellowed area)



Library Search Result for Yellowed Area

AIMsight Infrared Microscope

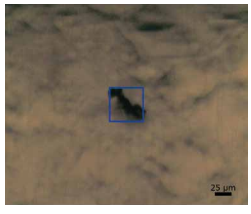
Analyzes samples as small as approx. 10 µm in size. Supports transmission, specular reflection, ATR, and other sampling techniques for analysis of a wide variety of samples. The AIMsight infrared microscope also comes with a range of features as standard that assist contaminant analysis, such as a wide-field camera (maximum field of view: 10 × 13 mm), the Contaminant Analysis program that automatically identifies contaminants in the field of view, and the Spectrum Advisor function that helps the user to determine the quality of their data.

IRXross+AIMsight

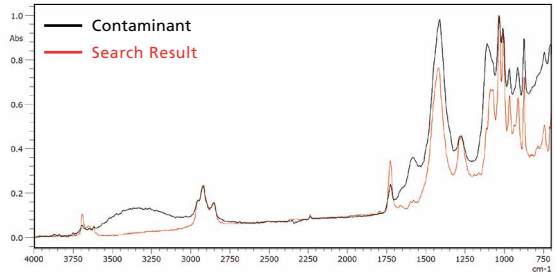
Analysis of a Contaminant Attached to a Button Cell Battery

AIMsight was used to analyze a contaminant attached to a button cell battery. The wide-field camera simplified the process of examining the button cell battery and determining the specific area for analysis. The sampling technique used in this analysis was direct ATR. A library search of an original Shimadzu contaminant library showed the contaminant was mainly composed of acrylonitrile butadiene rubber (NBR) with CaCO3, aluminum silicate (KAOLIN), and phthalate ester as additives.

Wide-Field Camera Image



Microscope Camera Image



ATR Spectrum of Contaminant Attached to Button Cell Battery Overlaid with Spectral Search Result

Raman Spectrophotometers

Similar to infrared spectra, every material has a unique Raman spectrum that can be used to identify and characterize contaminants by comparing them against library data.
Raman analysis also requires almost no sample pretreatment. Raman analysis can also use visible light lasers to analyze samples across transparent glass slides and containers.

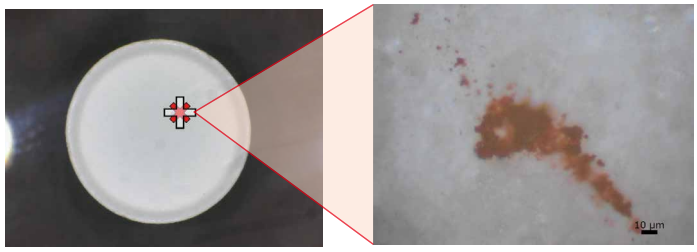
AIRsight Infrared/Raman Microscope

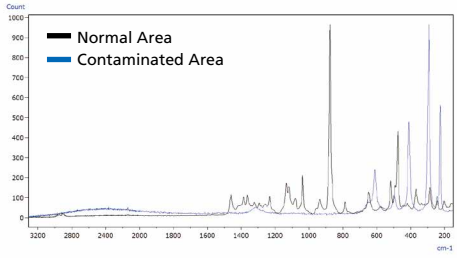
Raman mode on the AIRsight infrared/Raman microscope can analyze very small contaminants 10 µm and smaller in size not easily analyzed in infrared mode. Raman mode can also be used to characterize inorganic materials not easily characterizable in infrared mode (for metal inorganic materials, use the EDX systems mentioned above or EMPA systems mentioned below).

IRTracer-100+AIRsight

Analysis of a Reddish-Brown Contaminant on the Surface of a Medicinal Tablet

A reddish-brown contaminant was found scattered over an area around 100 µm in size on the surface of a medicinal tablet. Infrared mode with ATR sampling was initially used to analyze the normal area and contaminated area of the tablet, but this approach did not provide useful peaks from the contaminated area. Switching to Raman mode and performing a similar analysis of the normal area and contaminated area of the tablet revealed peaks that seemed to show the contaminant. A spectral search then revealed the adhered contaminant to be iron oxide.





Raman Spectrum of Contaminant Adhered to Surface of Medicinal Tablet Overlaid with Raman Spectrum of Normal Area

Electron Probe Microanalyzer

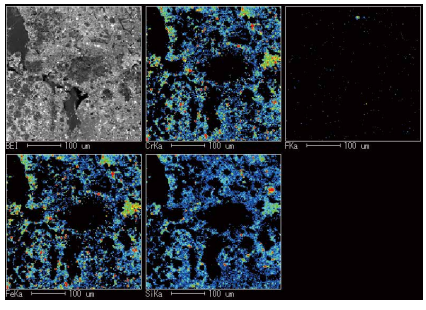
Irradiates the sample with an electron beam to perform elemental analysis and obtain geometric data on micrometer-order contaminants at high magnifications.
Electron probe microanalyzers can perform highly accurate and precise elemental analysis across very small and large areas.

EPMA-1720 Series/8050G

Analysis of a Contaminant on a Frozen Pizza

A black contaminant attached to a frozen pizza was sampled and subjected to elemental mapping analysis. Iron (Fe) and chrome (Cr) were detected across a wide area and fluorine (F) was detected in localized areas of the sample. FTIR analysis revealed starch, linseed oil, and fluoropolymer material, and EDX analysis revealed nickel (Ni) in addition to Fe and Cr. The black contaminant is presumed to be burned vegetable oil mixed with fluorine compounds and stainless steel powder originating from the cooking equipment and manufacturing machinery, though this requires verification at the manufacturing site.

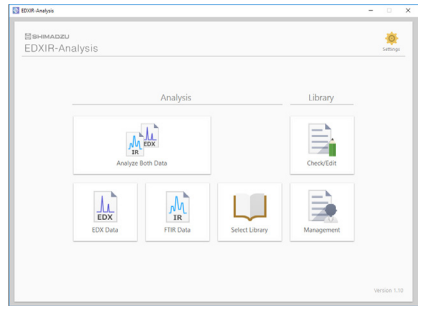
EPMA-1720 Series



EPMA-8050G

EDXIR-Analysis: Integrated EDX-FTIR Analysis Software (Optional)

This integrated EDX-FTIR analysis software (EDXIR-Analysis) is designed to perform a qualitative analysis with the data obtained by an energy dispersive X-ray fluorescence spectrometer (EDX) and a Fourier transform infrared spectrophotometer (FTIR). The EDXIR-Analysis software integrates the analysis of data obtained by FTIR analysis (suited to identification and characterization of organic materials) and EDX analysis (suited to elemental analysis such as of metals and inorganic materials) to identify target materials and calculate degrees of similarity against reference data. EDXIR-Analysis can also perform an analysis of data from EDX analysis alone or FTIR analysis alone. The spectral library used by EDXIR-Analysis is an original Shimadzu library (485 data sets included as standard) created with help from water utility companies and food producers. New data, images, and PDF format document files can also be added to the library, offering an effective means of linking together and storing different types of data electronically.



Integrated Analysis of Contaminant Data and Identification Testing by Data Comparison

An analyst simply clicks on "Analysis Using Both EDX and FTIR Data," selects the EDX and FTIR data, and the EDXIR-Analysis software automatically performs the qualitative analysis*1. The software streamlines a time-consuming process that is typically performed by the analyst, offering a powerful tool for contaminant analysis.

The integrated analysis produces a hit list and displays the EDX profile and FTIR spectra of each library hit.

Results from a non-integrated analysis of data collected from each analytical technique can also be viewed by clicking the "Single" button.

A "Data Comparison" feature also calculates the degree of similarity between library data and collected data for applications in identification testing, such as to protect against silent changes.

Click the "Print" button to print out files in a standard format and save files in Word format*2.

*1: Categorizes samples as inorganic/organic/mixed based on the EDX profile. Applies a weighting to each category for integrated analysis. [Patent pending]
*2: Requires Microsoft® Word.

An example integrated analysis was performed on data collected from a black rubber-like contaminant and, in a separate analysis, data collected from a legitimate product made of polyvinyl chloride (PVC) was compared with data collected from a product for inspection.

The integrated analysis showed the black rubber-like contaminant was acrylonitrile butadiene rubber (NBR) with calcium carbonate and zinc stearate.

The data comparison showed the degree of similarity between the PVC product for inspection and the legitimate PVC product was 0.8506. According to the EDX profile and FTIR spectrum, lead (Pb) and acrylic not present in the legitimate product were detected in the product for inspection. Based on this finding, the components not present in the legitimate product are probably contaminants in the product for inspection.

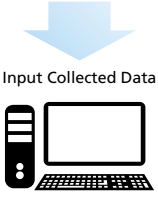

EDX

EDX Profile

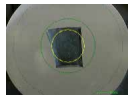
FTIR Spectrum

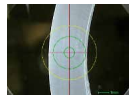

Plastic Analyzer


IRXross+AIMsight

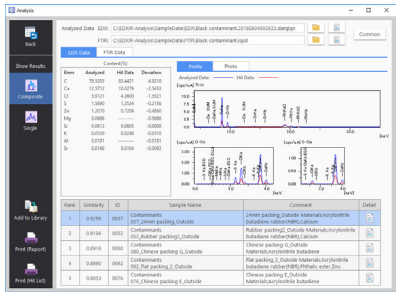

Input Collected Data

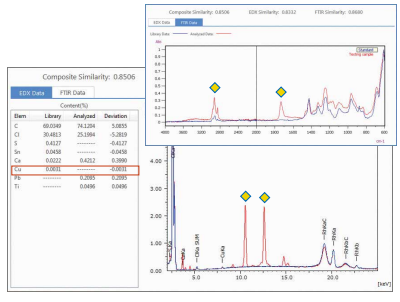
Integrated Analysis





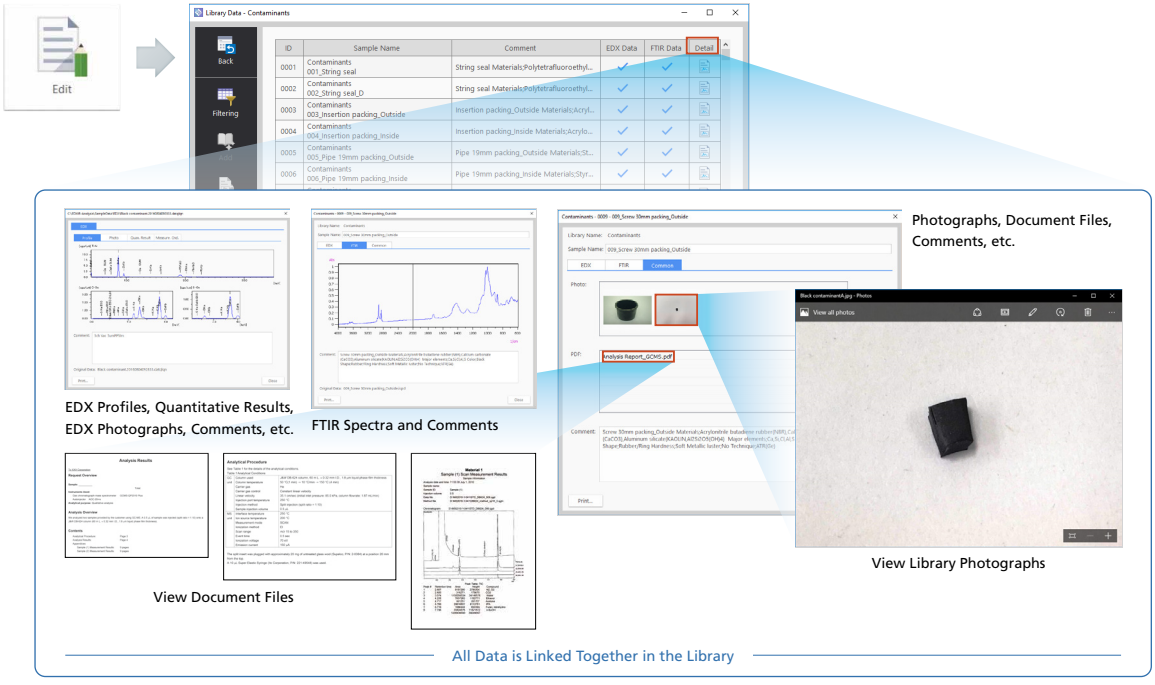
Data Comparison


Results from Integrated Analysis of Data on Black Rubber-Like Contaminant


Comparing Data from Legitimate PVC Product and Product for Inspection

Viewing Library Data and Adding, Editing, and Deleting Data, Images, and Document Files

Click on "View and Edit Data" and select an existing library to access data, images, and document files in the selected library and to add, edit, and delete data in the library. Users can also create new libraries with EDXIR-Analysis. Data collected with analytical systems other than EDX and FTIR systems (chromatography systems, mass spectrometers, surface analyzers, etc.) can also be saved alongside EDX and FTIR system data by adding them to the library in PDF format.



Products for EDX and FTIR Systems

EDXIR-Holder Contaminant Retainer/Storage Container (Optional)

Samples placed in an EDXIR-Holder can be analyzed on both an EDX system and an FTIR system. After analysis, the sample can also be stored in the EDXIR-Holder with no further processing.

Reducing Analytical Work

The EDXIR-Holder contains an adhesive film for sample adhesion and a polypropylene film for X-ray fluorescence analysis and can be opened and closed. To perform EDX analysis, the EDXIR-Holder is closed and positioned with the polypropylene film facing the irradiated side in an EDX system (facing down). To perform FTIR analysis, the EDXIR-Holder is opened and the sample adhered to the adhesive film is pushed directly against the ATR prism. EDXIR-Holder minimizes the work involved in sample preparation, thus reducing analytical tasks and streamlining the analysis process.

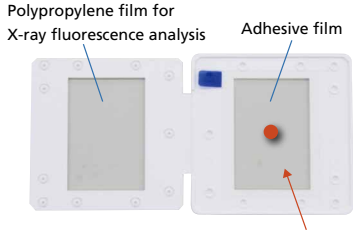
Preventing Lost Samples

After analysis, the EDXIR-Holder can be closed and the sample stored inside the EDXIR-Holder with no further processing. The sample does not need to be transferred to a separate storage container, thus lowering the risk of sample loss.

For EDX Analysis

Close the EDXIR-Holder and face the polypropylene film towards the irradiated side (facing down)

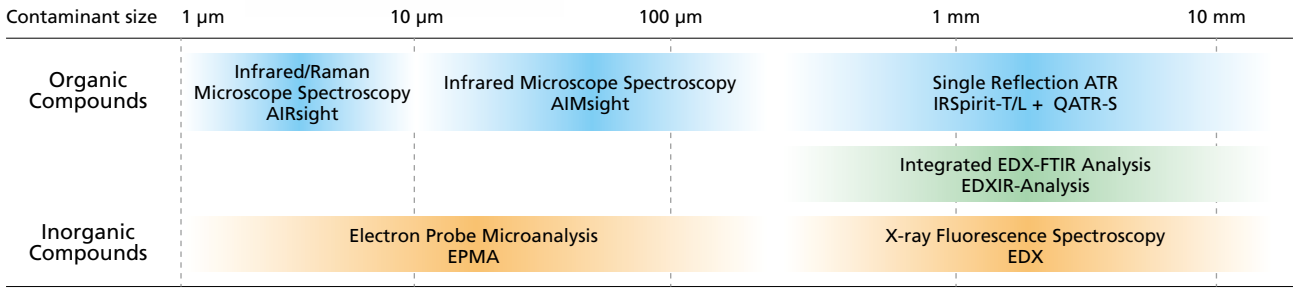
An open EDXIR-Holder (inside)



For FTIR Analysis

Open the EDXIR-Holder and push the sample adhered to the adhesive film up against the prism.

Shimadzu Recommended Analytical Techniques by Sample Size



Original Shimadzu FTIR Databases for Contaminant Characterization

Contaminant Library for LabSolutions IR (P/N 206-33179-91) P/N 206-33179-91

This original Shimadzu library contains information on actual contaminants in the water supply and foods, as well as information on commercially available products used in water supply maintenance. The library also includes X-ray fluorescence profiles (in PDF file format) for significantly more accurate contaminant characterization. Unlike previous libraries, this library includes data on mixed materials and accommodates the wide-ranging knowledge and extensive experience needed for qualitative assessment.

Thermally-Damaged Plastics Library*1 P/N 206-33039-91 and UV-Damaged Plastics Library*2 P/N 206-31808-41

Unlike previous libraries, these libraries compile data on plastics that have undergone oxidative degradation by heat and ultraviolet irradiation. Degraded materials are a common object of analysis, and these libraries prove extremely effective in such cases.

*1: Library compiled by Shimadzu Corporation from spectra recorded by Hamamatsu Technical Support Center, Industrial Research Institute of Shizuoka Prefecture.
*2: Library compiled from infrared spectra of plastics degraded by approx. 10 years of ultraviolet irradiation using an ultra-accelerated weathering test machine (Iwasaki Electric Co., Ltd.).

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