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Infrared Microscope

—Dedicated AIMsolution Software

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AIMsolution software for AIM-9000 infrared microscopes offers many new and improved features and is easier to use than AIMView, Shimadzu's previous microscope software. Several of the more representative features are described below.

1. Introduction

As mentioned in the previous volume of FTIR Talk Letter, measurements using an infrared microscope involve the following steps.

(1) Pretreat the sample, as necessary.
(2) Place the item being measured on the infrared microscope stage and position it so that the target measurement location is within the field of view.
(3) Select the location to be measured and specify the aperture (measurement range).
(4) Measure the background and sample.

The previous article described the wide-view camera feature used to reduce the time required for step (2). This article describes the automatic contaminant recognition function and simultaneous visual observation and measurement function used to assist with steps (3) and (4), and also several other features provided to help support those performing measurements.

2. Automatic Contaminant Recognition Function

With AIMView software, specifying the aperture (measurement range) in step (3) required manually setting the aperture for each point in candidate measurement regions while viewing the visual observation image. The automatic contaminant recognition system included standard in AIMsolution software takes about one second to automatically specify appropriate aperture size and angle settings for each suggested measurement point in the image currently being observed. The following is a simplified description of how settings are automatically specified. First, the visible image is converted to a monochrome image (Fig. 1).

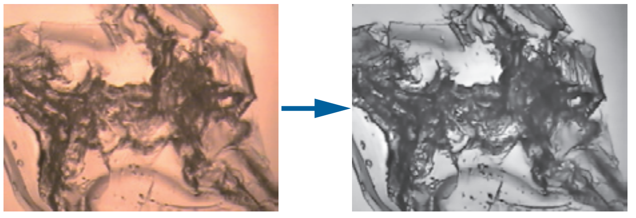
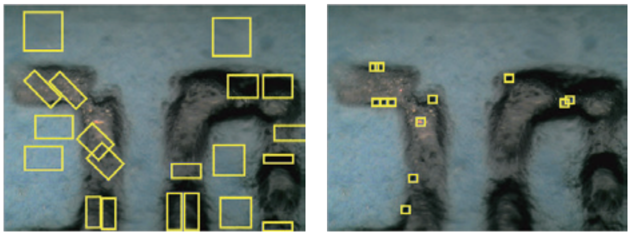


Fig. 1 Microscope Visible Image and Corresponding Monochrome Image

Then the monochrome image is used to perform contrast calculations. Different regions are separated based on differences in contrast and an aperture box is specified for the different regions.

Aperture setting results actually obtained are shown in Fig. 2.



Standard Settings   Micro Settings

Fig. 2 Aperture Setting Results from Automatic Contaminant Recognition Function

Two aperture setting modes are available. The micro setting mode detects targets 10 m or smaller and the standard setting mode specifies larger apertures. Setting results are also displayed at the same time.

That means the preferred setting mode can be selected based on the given application before starting measurements. If any automatically specified apertures are unnecessary, they can be deleted, or new apertures can be added.

3. Simultaneous Visual Observation and Measurement

It is sometimes desirable to confirm whether or not any significant peaks will appear from a specified measurement point before measuring the sample in step (4). The simultaneous visual observation and measurement function is useful in such cases.

With previous infrared microscopes, the optical path had to be switched to infrared after setting the apertures in the visual observation image. Therefore, it was not possible to check data while checking the observation image in real time. Consequently, if good data could not be acquired at a specified measurement point, for example, the measurement had to be aborted to reset the aperture settings in the visual observation image before repeating the measurement.

In contrast, the AIM-9000 allows sample or monitor measurements while viewing the visual image. That means data can be confirmed in advance by moving the stage or changing aperture settings.

Fig. 3 shows a cross-section image of a multilayer film viewed using an AIM-9000 microscope and infrared spectra obtained from each layer. The red box in the image indicates the aperture setting, where the sample position within the aperture box and the corresponding infrared spectrum can be changed by moving the stage. (An infrared spectrum for polyvinyl chloride was obtained from the position shown in the upper image of Fig. 3 and for polyethylene from the position in the lower image.)

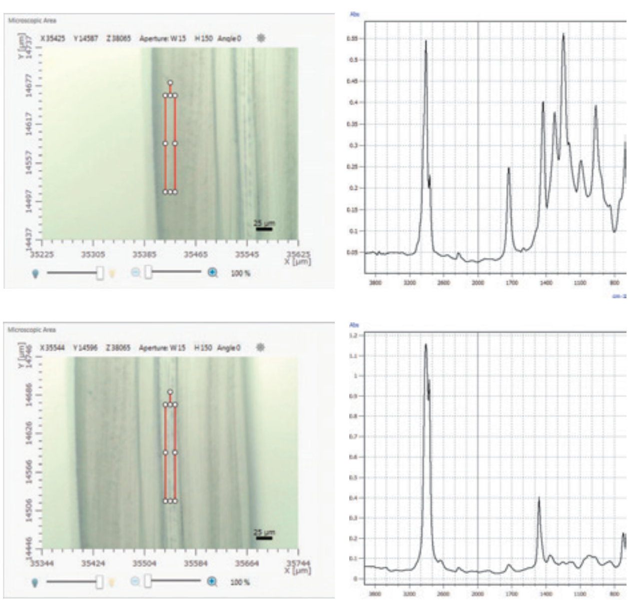


Fig. 3 Observation Image and Infrared Spectrum from Multilayer Film Cross Section
(Upper: Aperture box positioned on layer at left edge; Lower: Aperture box positioned on center layer)

To enable measurements at the same time as visual observation, the mirror referred to as a "hot mirror" is placed in the optical path. The hot mirror reflects infrared light, but transmits visible light. The hot mirror separates the light that is transmitted through and reflected from the sample into visible and infrared light. The visible light for observing the sample is sent to the camera, whereas the infrared light for measurement is sent to the detector. Apertures that limit the measurement region are only present in the infrared light path, so that the entire image can be observed, while only measuring the area inside the aperture box.

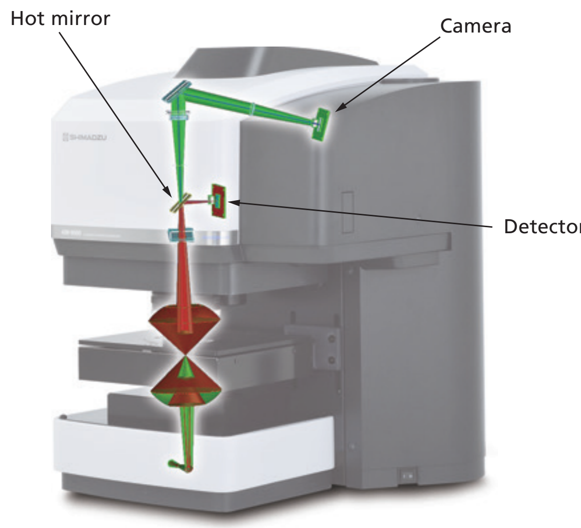


Fig. 4 Illustration of Visible and Infrared Light Paths
(Green: Path of visible light; Red: Path of infrared light)

4. Word Output Function

After spectra are measured and data is analyzed, results are output in a report.

The Word output function is especially useful for that process.

A Word output button is displayed in the print preview window (Fig. 5).

Clicking the Word button enables all the information displayed in the print preview window to be saved as a Microsoft Word file (requires that Microsoft Word software is installed).

Then the created Word file can be used to change the layout or add or delete text or graphs, as necessary (Fig. 6).

This function is especially convenient for creating reports.

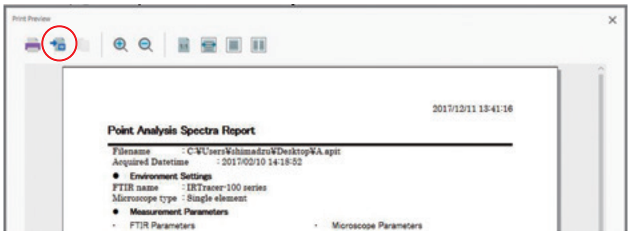


Fig. 5 Print Preview Window

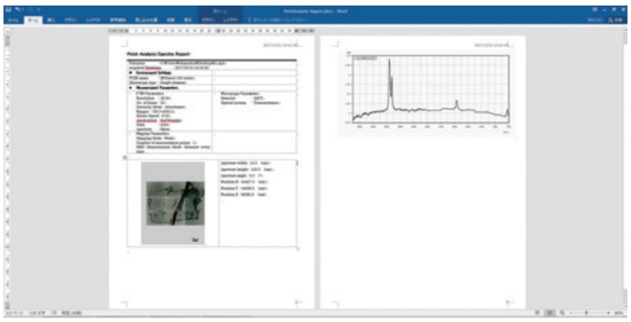


Fig. 6 Created Word File

5. Guidance Function

Guidance features display information for guiding operators through the measurement process from instrument initialization to sample measurement.

Clicking the guidance button displays a guidance bar along the left edge.

The example in Fig. 7 shows guidance information displayed for registering measurement points.

It indicates the upper limit values for registering measurement points and provides operating instructions for apertures, used to specify measurement points.

Registration, measurement, and other operations can be performed with the guidance information left displayed.

6. Summary

In addition to the functionality described above, AIMsolution software also includes many other features for supporting measurement operators.

We hope you have an opportunity to use the AIM-9000 infrared microscope and AIMsolution software.

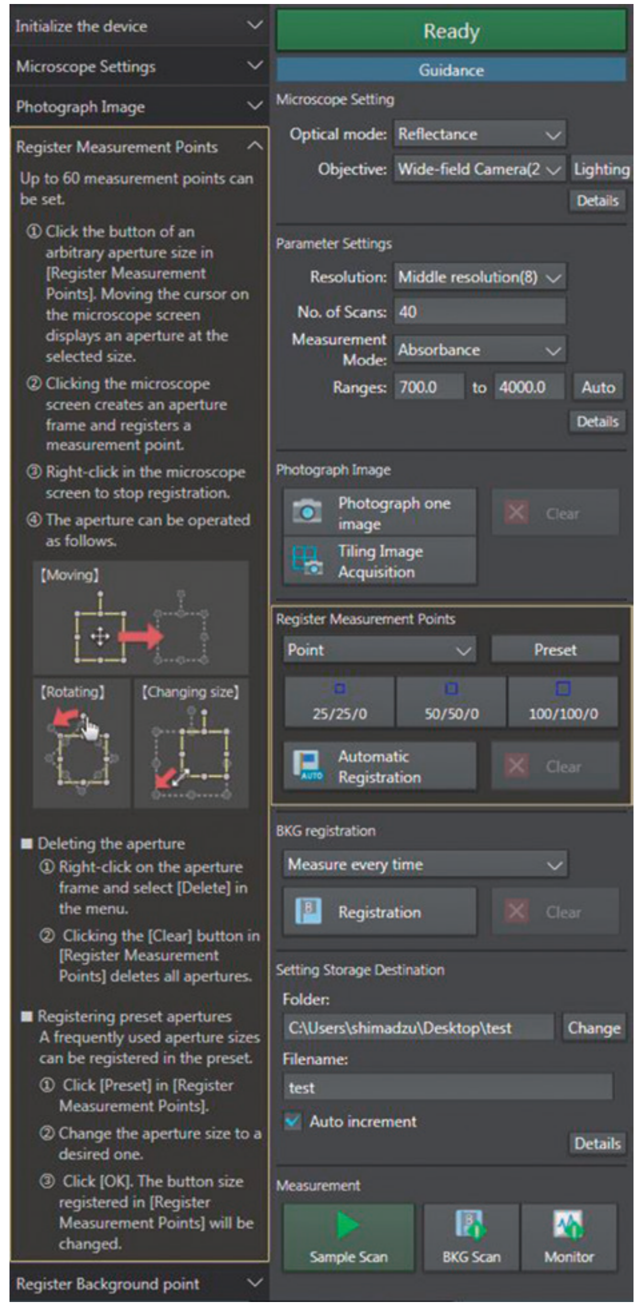


Fig. 7 Guidance Display Showing Instructions for Registering Measurement Points

Infrared Microscope
—Using Imaging Analysis

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In May 2016, Shimadzu released the AIM-9000 infrared microscope as an automatic failure analysis system. In FTIR Talk Letter Vol. 27, an article entitled "New Infrared Microscope—Convenience of a Wide-View Camera" described the convenience of using the wide-view camera to view large areas of a sample. This article describes the functionality of the optional mapping program available for the AIM-9000's AIMsolution control software.

1. Introduction

Imaging analysis refers to visualizing the distribution of components and spatial differences between chemical structures contained in measured target objects. Due to the large amount of information that can be obtained in a short time, imaging analysis is used for a wide variety of applications, such as analyzing contaminants, industrial materials, and biological samples.

The AIM-9000 infrared microscope offers mapping speeds that are about four times faster* than the AIM-8800 model. That means imaging analysis can be performed more smoothly than ever before. With a 30,000:1 signal-to-noise ratio (the highest sensitivity in the industry), the AIM-9000 offers both high speed and high sensitivity.

The mapping program can measure areas for analyzing in-plane distribution of components within samples or measure lines for analyzing data in the depth direction.

* Varies depending on measurement conditions (such as aperture size, step size, and number of scans).

2. Example of Contaminant Mixture Analysis

First, a portion of the sample is placed on the diamond cell and compressed to a thickness appropriate for transmittance measurements. After compression, the diamond cell is placed on the stage. The transmission method results in more light reaching the detector than the reflection or ATR methods, which offers the advantage of higher sensitivity measurements. On the other hand, a disadvantage is that absorption peaks can become saturated for samples that are unnecessarily thick, which can prevent obtaining accurate data. In such cases, saturation can be avoided by using the diamond cell to compress the sample to make it thinner. A thickness of 10 to 20 μm is generally considered appropriate.

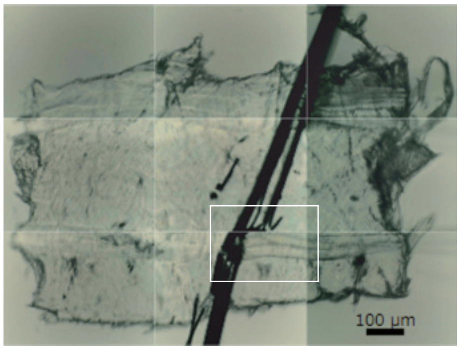

Fig. 1 Overview Image of Contaminant

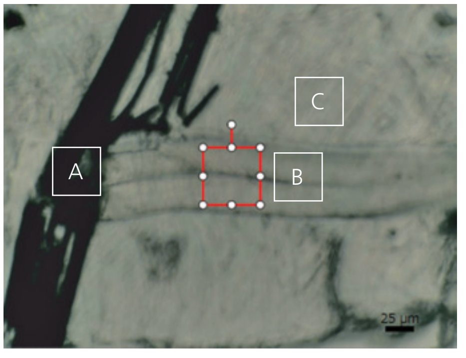

Fig. 2 Observation Image Obtained with the Microscope Camera

A visual observation of the contaminant confirmed that it contains a mixture of several types of substances. Fig. 1 shows an overview image of the contaminant, obtained by using the tiling function to stitch multiple images together. A microscope camera image of the area inside the white box in Fig. 1 is shown in Fig. 2. Based on Fig. 1, the contaminant size is determined to be about 600 µm tall by 900 µm wide.

In Fig. 2, characteristic features are labeled A, B, and C. Infrared spectra obtained at points A, B, and C are shown in Fig. 3. Polyethylene peaks were detected at points A and C, whereas peaks for aramid fiber were detected at point B.

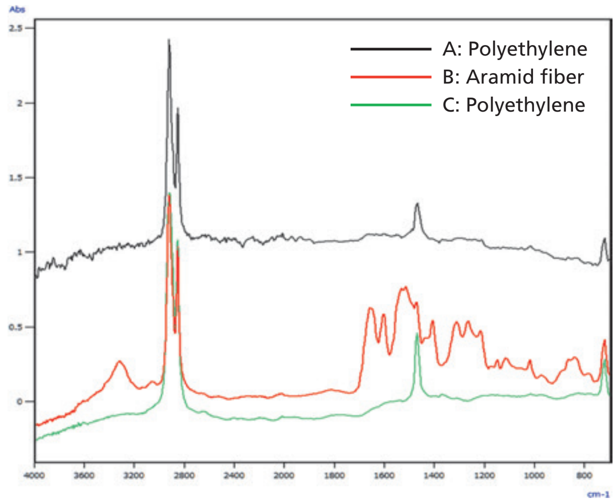

Fig. 3 Infrared Spectra of Contaminant Mixture (at Points A, B, and C)

To measure the distribution at each point, imaging analysis was performed using the microscope transmission method. A 175 µm tall by 250 m wide area of the sample was measured. Measurement conditions are indicated in Table 1 and the image obtained from the specified measurement range is shown in Fig. 4. A 25 µm × 25 µm aperture was specified for each measurement position. The blue grid indicates the aperture for each measurement position, which results in measuring the selected range without any gaps.

Table 1 Measurement Conditions

Instrument	: IRTracer-100 / AIM-9000	Apodization	: Sqr-Triangle
		Aperture size	: 25 µm×25 µm
Resolution	: 8 cm^{-1}	Step	: 25 µm
Accumulation	: 20	Detector	: MCT

Fig. 4 Image After Specifying Measurement Range Settings

Given default settings, imaging analytical results display a chemical image with areas with higher values in red and areas with lower values in blue, calculated based on either intensity at a specified position, intensity ratio at a specified position, peak height, peak height ratio, peak area, peak area ratio, or match score. Images can also be created based on multivariate analysis results, such as from principal component analysis (PCA) or multivariate curve resolution (MCR) analysis.

An imaging analysis image based on the peak height at 1651 cm^{-1} for the C=O stretching vibration of the amide bond (-CONH-) in aramid fiber is shown in Fig. 5 (a). An image based on the peak height at 1470 cm^{-1} for the bending vibration of CH_2 in polyethylene is shown in Fig. 5 (b). The images for the aramid fiber and polyethylene are inverted to show the respective distributions more clearly.

Imaging analysis enabled qualification of each component contained in the mixed contaminant mixture and visualization of their distribution.

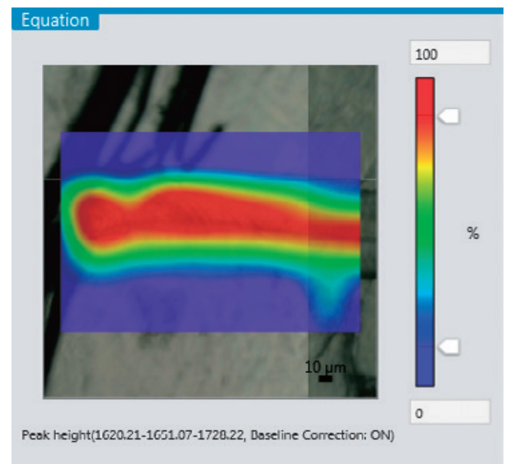

Fig. 5 (a) Distribution of Aramid Fibers (peak height at 1651 cm^{-1})

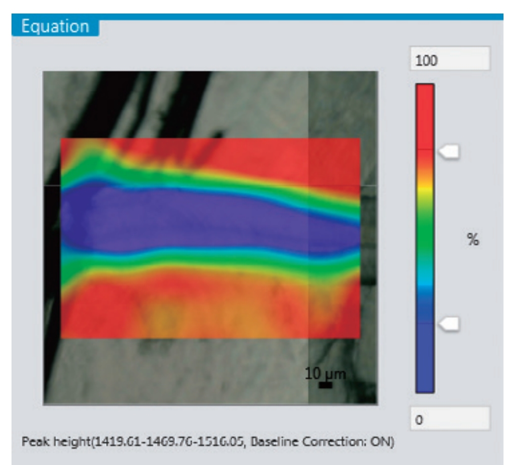

Fig. 5 (b) Distribution of Polyethylene (peak height at 1470 cm^{-1})

3. Example of Electronic Circuit Board Analysis

A tiled image of the electronic circuit board is shown in Fig. 6. Data from the area in the red box was analyzed. Because visual observation was not able to confirm differences with respect to the surrounding area, imaging analysis was performed using the microscope reflection method. An area 200 µm tall by 325 µm wide was measured. Other measurement parameters were the same as in Table 1.

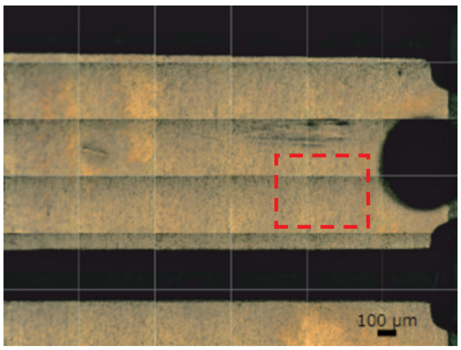

Fig. 6 Tiled Image of Electronic Circuit Board

The infrared spectra obtained from the area inside the red box on the electronic circuit board are shown in Fig. 7. Components judged to be paraffin oil and silicate were detected. Because peaks similar to paraffin oil were also detected from where the silicate is located, the location probably has a mixture of the two types of components.

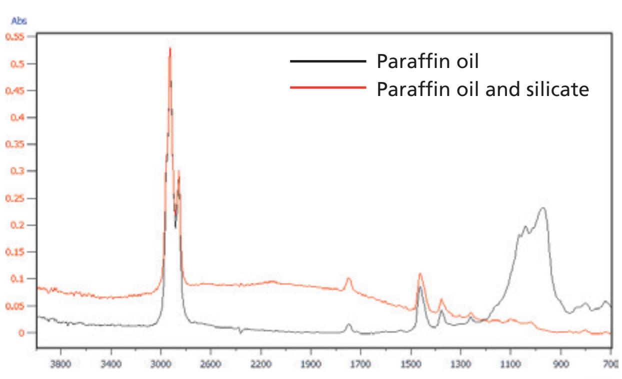

Fig. 7 Infrared Spectra Obtained from the Electronic Circuit Board

An imaging analysis image based on the peak height at 1377 cm^{-1} for the CH_3 bending vibration of the paraffin oil is shown in Fig. 8 (a). An image based on the peak height at 972 cm^{-1} for the silicate is shown in Fig. 8 (b). Fig. 8 (a) clearly shows the presence of paraffin oil extending out toward the left of the measurement range, whereas Fig. 8 (b) clearly shows that the presence of silicate is localized in the locations indicated in red. The figures also show normal areas where there is no adhesion of either component (indicated in blue).

Imaging analysis clearly showed the distribution of components, even in the case of this sample for which the distribution is not visually observable.

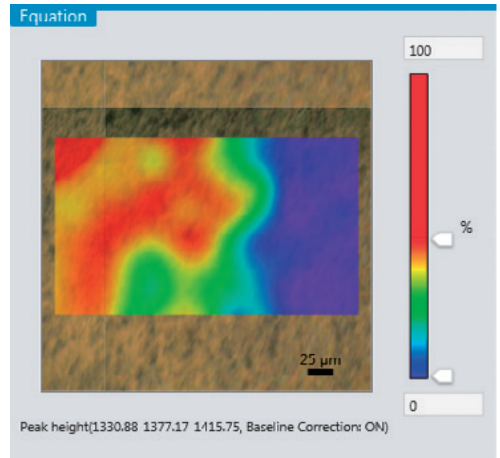

Fig. 8 (a) Distribution of Paraffin Oil (peak height at 1377 cm^{-1})

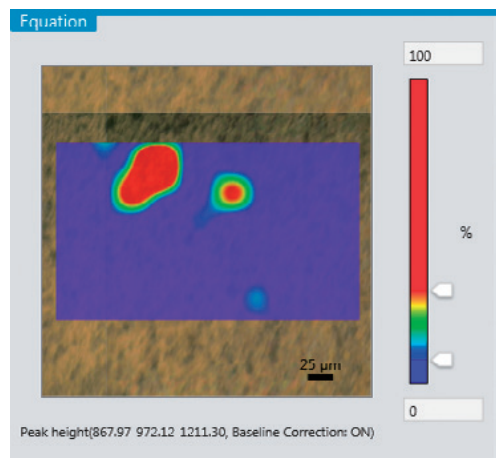

Fig. 8 (b) Distribution of Silicate (peak height at 972 cm^{-1})

4. Conclusion

The AIM-9000 infrared microscope not only enables both "high speed" and "high sensitivity" imaging analysis, but also offers excellent operability that was dramatically improved through Shimadzu's unique expertise. Though many customers have already experienced its convenience, we would be grateful if even more customers were given the opportunity to experience the outstanding features of the AIM-9000 infrared microscope as well.

Reference

Application News No. A514

Due to publication circumstances, the Q&A section has been omitted in this issue.

EDX-FTIR Contaminant Finder/Material Inspector

EDXIR-Analysis

EDXIR-Analysis software is specially designed to perform integrated analysis of data acquired from EDX, which is excellent at the elementary analysis of metals, inorganic compounds and other content, and from FTIR, which is excellent at the identification and qualification of organic compounds.

Contaminant Analysis

Performing qualitative analysis using data acquired by both EDX and FTIR enables high-precision automatic identification analysis. This heightens the efficiency of data analysis and provides strong support for contaminant analysis.

Data Comparisons for Confirmation Tests

With the data comparison function, which calculates the degree of matching between the actual measured data and the data registered in the library, the software can be used for countermeasures against "silent change"* and for other confirmation tests.

Data Registration and Storage

The data for the same sample acquired by other instruments (LC, GC, GCMS, SEM, etc.) can be converted into PDF format and then registered, enabling linked storage to the EDX/FTIR data.

*: A term used in Japan to indicate changes to materials by suppliers without the knowledge of the manufacturers.

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