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Application News

AIMsight™ Infrared Microscope

Micro-Contaminant Analysis Using AIMsight Infrared Microscope

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User Benefits

- The AIMsight infrared microscope can measure the infrared spectrum of micro regions as small as 10 × 10 µm.
- Highly accurate qualitative analysis is possible by using Shimadzu original Contaminant Library.
- "Spectrum Advisor (patent pending)" is an effective function for checking the quality of acquired infrared spectra.

■ Introduction

Recent years have seen heightened concern among consumers about contamination of products, and demand for analysis to respond to those concerns has also increased. Although reports that foreign matter has been found in some pharmaceuticals and food products have appeared in the news media from time to time, it would be difficult to eradicate this problem completely, since the causes of foreign matter are assumed to occur in various processes, for example, contamination of raw materials at the time of purchase, contamination of products due to deterioration of the component parts of production lines, and contamination of products by the customers themselves. The types of foreign matter are also diverse, including not only organic substances such as human hair, plastics, and rubber, but also oxides, metal fragments, and other types of inorganic materials. Thus, improved accuracy in qualitative analysis is required in order to identify the cause of contamination.

Since the AIMsight infrared microscope (Fig. 1) has a SN ratio of 30000 : 1, which is the highest in its class, it is possible to acquire satisfactory spectra in a short time even when analyzing microscopic contaminants. In addition, the AMsolution control software provides a sample length measurement function and the Spectrum Advisor function as standard equipment, which can check the quality of infrared spectra.

This article introduces an example of ATR (attenuated total reflection) measurement of a micro-contaminant adhering to the surface of a button cell with the AIMsight infrared microscope, and a contaminant analysis of the acquired infrared spectrum using Shimadzu Contaminant Library, which represents a compilation of this company's experience and analytical know-how.

Fig. 1 Appearance of IRTracer™-100 (Left) and AIMsight™ (Right)

■ Wide-Field Camera and Length Measurement Function

The work of setting a button cell on the stage of an infrared microscope and adjusting the microscope so that the measurement position of the foreign matter adhering to its surface is in the field of view (FOV) is one of the most time-consuming processes in contaminant analysis. In order to shorten the time required for this process, the standard equipment of the AIMsight includes a wide-field observation camera capable of observing a range as large as 10 × 13 mm, and also supports variable digital zooming with a maximum magnification of 5x (2.0 × 2.6 mm).

The microscope camera (FOV: 0.3 × 0.4 mm) used in actual measurements also has a variable digital zoom function with a maximum magnification of 10x (0.03 × 0.04 mm), making it possible to confirm foreign matter continuously from a wide FOV to a narrow FOV.

The wide-field camera and microscope camera also share positional information, so there is no shifting of the center of the FOV due to camera switching.

Fig. 2 shows the observation images of the micro-contaminant adhering to the button cell surface captured by the wide-field camera and the microscope camera. By using this combination of images, the position of the adhering foreign matter can be searched simply, while checking the overall image of the cell.

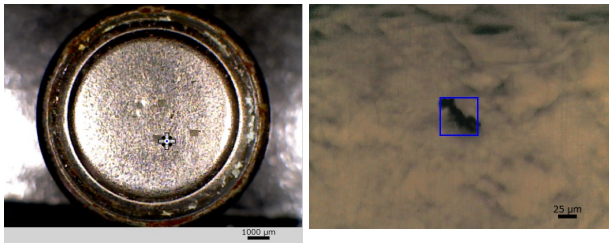


Fig. 2 Appearance of Contaminant on Surface of Button Cell
Left: Overall Observation Image of Button Cell by Wide-Field Camera,
Right: Observation Image of Contaminant on Cell Surface by Microscope Camera

The AMsolution software that controls AIMsight also has a length measurement function which makes it possible to measure the distance between two arbitrary points from the acquired images. Fig. 3 shows the length measurement function screen.

Fig. 3 Length Measurement Function Screen

The length between two points can be measured simply by deciding the starting and finishing points and clicking. Using this function, it was found that the lengths of the short side and long side of the micro-contaminant adhering to the button cell surface were 18 µm and 60 µm, respectively.

■ ATR Measurement of Micro-Contaminant Adhering to Button Cell Surface

ATR measurement of the micro-contaminant adhering to the surface of the button cell was carried out under the measurement conditions in Table 1. Fig. 4 shows the acquired infrared spectrum. The micro-contaminant could be captured accurately and measured with high sensitivity, and a satisfactory, strain-free infrared spectrum could be acquired.

Table 1 Measurement Conditions

Instruments	: IRTracer™-100, AIMsight
Resolution	: 8 cm^{-1}
Accumulation	: 200
Apodization function	: SqrTriangle
Detector	: T2SL

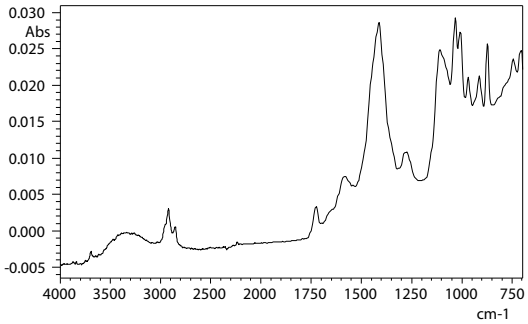


Fig. 4 Infrared Spectrum of Micro-Contaminant Adhering to Button Cell Surface

Here, it can be understood that the micro-contaminant was captured accurately, noise was slight, and measurement with high sensitivity was possible.

■ Library Search of Infrared Spectrum

A search for the infrared spectrum in Fig. 4 was conducted using the Shimadzu standard library. Fig. 5 shows the results obtained.

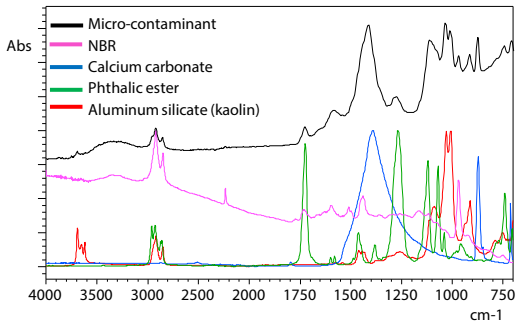
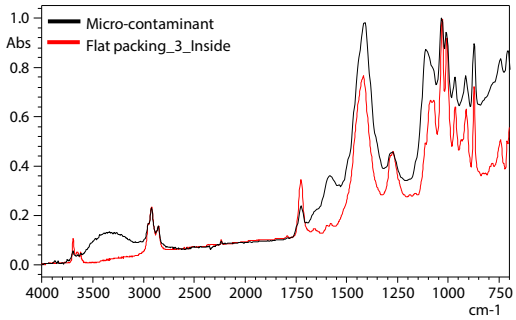


Fig. 5 Result of Search of Infrared Spectrum (Using Standard Library)

It can be estimated that this foreign matter consists of acrylonitrile butadiene rubber (NBR) as its main component, and also contains calcium carbonate ($CaCO_3$), phthalic ester, and aluminum silicate (kaolin) as other additives. However, it should be noted that a close investigation of each individual component is necessary when foreign matter consists of multiple components, and a certain level of skill in spectrum analysis is required.

Next, a search was carried out using Shimadzu original Contaminant Library. Fig. 6 shows the results obtained.



Infrared spectrum information for "Flat packing_3_Inside"
Materials; Acrylonitrile butadiene rubber (NBR), Calcium carbonate ($CaCO_3$), Phthalic ester, Aluminum silicate (KAOLIN, $Al_2Si_2O_5(OH)_4$)
Major elements; Ca,Si,Cl,Al,S Color; Black Shape; Rubber/Ring
Hardness; Soft Metallic luster; No Technique; ATR (Ge)

Fig. 6 Results of Search of Infrared Spectrum and Detailed Information on Library Data for Hit (Using Contaminant Library)

Unlike commercially-available libraries that record only data for single components, the Contaminant Library, which is an original library created by Shimadzu, contains data on foreign substances that were actually captured (data provided by water supply utilities and food product companies) and mixtures such as packing, and thus provides dramatically improved search accuracy. In addition, the Contaminant Library also includes various supplementary information such as the major elements, color, shape, hardness, and metallic luster of contaminant substances.

■ Spectrum Advisor Function

Although acquisition of a good spectrum is essential for conducting a smooth analysis, it is difficult for persons with little analysis experience to judge the quality of spectra. Spectrum Advisor is a function that checks the quality of acquired infrared spectra. It is also a convenient function, as it displays the results of spectrum checks and methods of solving problems when the analyst selects points of concern by interactive operation in an infrared spectrum, such as the baseline and peak. Since points of concern are selected by interactive operation using dialogue boxes, as shown in Fig. 7, the Spectrum Advisor function can be operated simply, even by persons with little analysis experience.

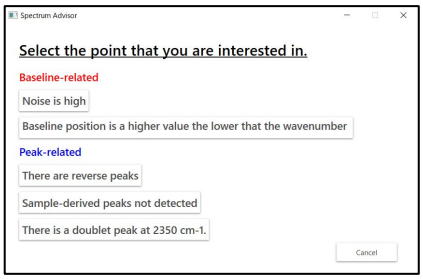


Fig. 7 Spectrum Advisor Screen

■ Conclusion

An analysis of micro-contamination adhering to the surface of a button cell using the AIMsight infrared microscope was introduced.

AIMsight is equipped with a rich range of features that support smooth contaminant analysis, including a wide-field observation camera and length measurement function and the Spectrum Advisor function. When conducting contamination analyses and defect analyses in which accurate qualitative analysis is demanded, use of Shimadzu Contaminant Library is recommended as a useful tool.

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