

Application News

No. A466

Spectrophotometric Analysis

Efficient Analysis by Infrared Microscope Using Automated Measurement Features

As a method of contaminant analysis and defect analysis in various industries including the food, pharmaceutical, chemical, and electrical and electronic fields, infrared microspectroscopy is routinely used in many of these quality control departments. Not unexpectedly, there is a common desire throughout these fields for automation of the analysis process to enhance efficiency.

The AIMView software used for control of the infrared microscope permits automatic measurement at a maximum of 10 specified measurement sites using different aperture settings. Another feature is that images of the measurement sites are saved automatically in the spectrum file.

Here, we introduce the use of these features in combination with a new feature in LabSolutions IR called "Easy Macro," which can be used to easily automate a series of operations.

■ Preparation of the Macro Program

Easy Macro is a feature that permits original programs to be created by simply dragging and dropping macro items onto the screen. Please refer to Application News No. A465, which describes the use of Easy Macro to automate the Degree of Similarity calculation for possible use in verification testing. First, a macro program was created using the operation flow shown in Fig. 1. As a result, the entire operation from measurement to report output can be quickly completed, even by those unfamiliar with the software. Further, such macros can prevent mistakes in complicated operations associated with routine tasks, and can shorten the time required for repetitive tasks.

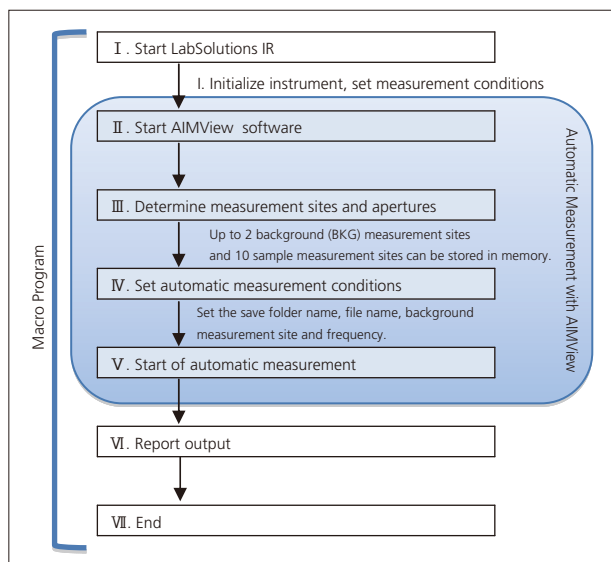


Fig. 1 Flowchart of Measurement Using Macro Program

■ Measurement of Contaminants on Surface of Tablet Using Infrared Microscope

First, the contaminant adhering to the tablet surface was transferred to the diamond cell using the tip of a thin needle. Fig. 2 shows a photograph after the sample was compressed using a diamond cell plate. In cases such as this where the contaminant may consist of a mixture of substances rather than a single one, it is necessary to determine whether or not the substance is uniform by taking measurements at different sites on the sample.

With that, the macro program was started to run the software (I and II in Fig. 1). Next, the apertures were set for background (BKG) measurement at two sites, and for sample measurement at four sites. The aperture size was 20 $\mu\text{m} \times 20 \mu\text{m}$ for sites (1) and (2), and 30 $\mu\text{m} \times 30 \mu\text{m}$ for sites (3) and (4) (Step III of Fig. 1). Next, the BKG measurement sites and file names were specified in the window shown in Fig. 3, where the automatic measurement conditions can be set (Step IV of Fig. 1).

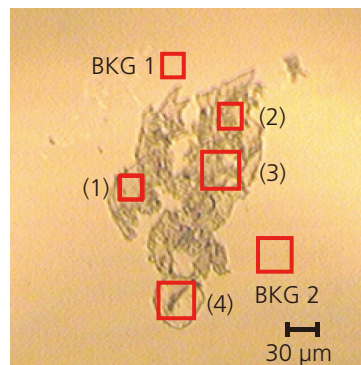


Fig. 2 Contaminants on Diamond Cell

Automeasure configuration window showing settings for data folder name, file name, and measurement sites.

No.	Measure	BKG pos	File name	Sample name	Sample ID	Option	Comment
1	<input checked="" type="checkbox"/>	B1	Contaminants_01.ispd				
2	<input checked="" type="checkbox"/>	B1	Contaminants_02.ispd				
3	<input checked="" type="checkbox"/>	B2	Contaminants_03.ispd				
4	<input checked="" type="checkbox"/>	B2	Contaminants_04.ispd				
5	<input type="checkbox"/>	B1	Contaminants_05.ispd				
6	<input type="checkbox"/>	B1	Contaminants_06.ispd				
7	<input type="checkbox"/>	B1	Contaminants_07.ispd				
8	<input type="checkbox"/>	B1	Contaminants_08.ispd				
9	<input type="checkbox"/>	B1	Contaminants_09.ispd				
10	<input type="checkbox"/>	B1	Contaminants_10.ispd				

Fig. 3 Setting of Automatic Measurement Conditions

Then, automatic measurement was conducted (Step V of Fig. 1). The instrument and analytical conditions that were used are shown in Table 1. The measurement results, shown in the report output example of Fig. 4 (Step VI of Fig. 1), were generated using the Easy Macro program.

Thus, the entire process from measurement to report output was conducted semi-automatically as described above using the Easy Macro features in combination with the automatic measurement features of the infrared microscope. Compared to the time that would be required to execute all these operations manually, the overall time required can be shortened by about 50 %.

The infrared spectra search results obtained in this analysis are shown in Fig. 5. The results indicate that contaminants 1, 2, and 3 consist mainly of a type of cellulose found in cotton, and that contaminant 4 contains an amide substance found in hair as its principal component.

Conclusion

Here, we conducted automatic measurement of an apparent contaminant adhering to the surface of a tablet using the automatic measurement features of the infrared microscope together with the Easy Macro feature. By conducting measurement at multiple sites on the sample, it was found that the contaminant consisted of a mixture of substances, rather than a single component.

When conducting an infrared microscope spectroscopic measurement, it is convenient to be able to conduct measurements automatically at multiple sites on the sample, for example (1) to compare spectra at different sites (verification of uniformity), (2) to conduct qualitative analysis of each layer in multilayer film, and (3) to conduct a target substance search.

In addition to microscope transmission measurement, this automated measurement feature can also be used for microscopic reflection measurement. And, by combining it with the Easy Macro feature, not only can it automate the process of measurement through report output, it can also greatly assist in automation of an infrared microscope spectroscopic measurement by automating routine tasks.

Table 1 Instrument and Analytical Conditions

Instrument	: IRAffinity-1, AIM-8800, Diamond cell
Resolution	: 8 cm ⁻¹
Accumulation	: 20
Apodization	: Sqr-Triangle
Detector	: MCT

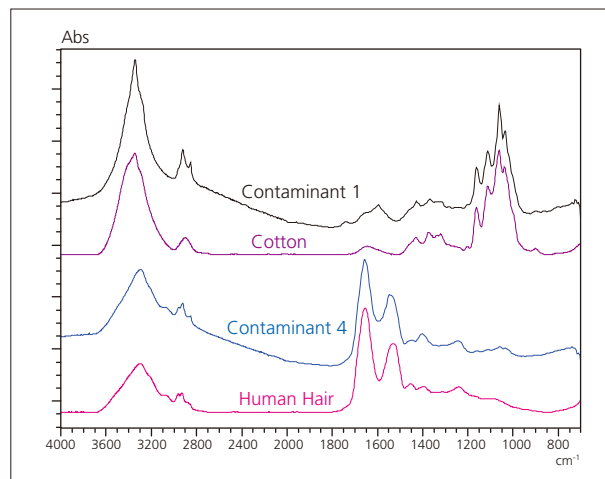


Fig. 5 Infrared Spectra Search Results

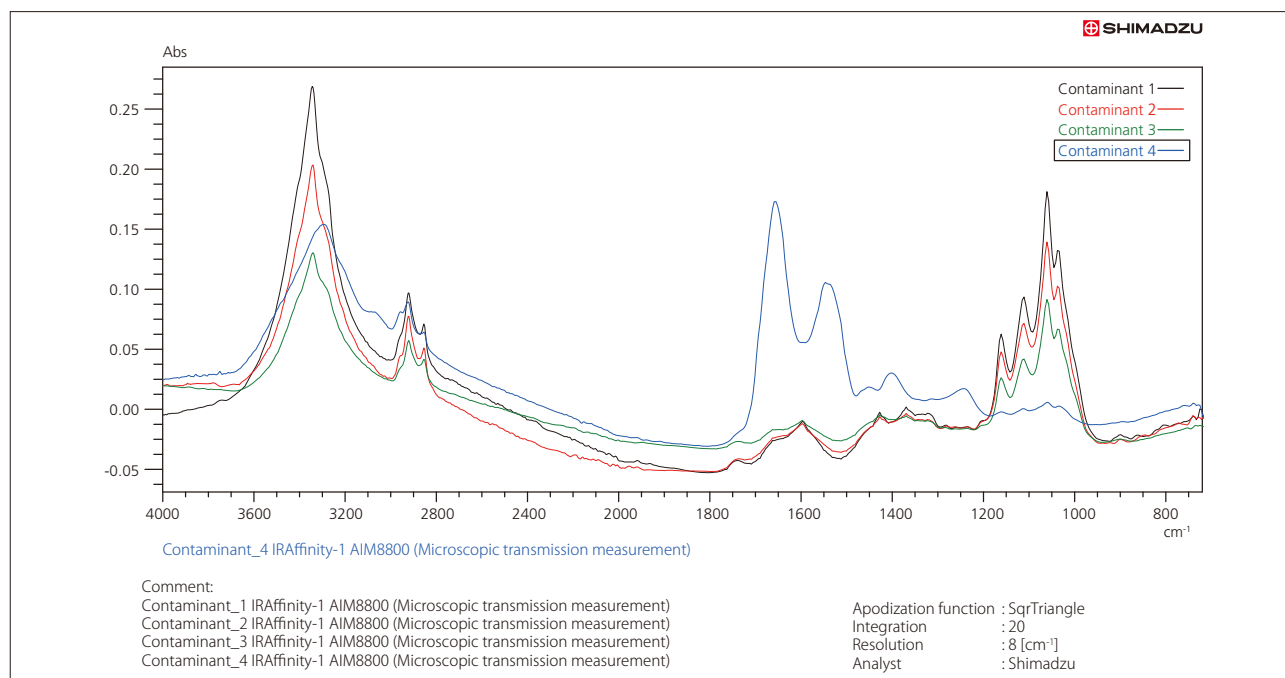


Fig. 4 Example of Report Output

First Edition: Aug. 2013



Shimadzu Corporation

www.shimadzu.com/an/

For Research Use Only. Not for use in diagnostic procedures.

The content of this publication shall not be reproduced, altered or sold for any commercial purpose without the written approval of Shimadzu. The information contained herein is provided to you "as is" without warranty of any kind including without limitation warranties as to its accuracy or completeness. Shimadzu does not assume any responsibility or liability for any damage, whether direct or indirect, relating to the use of this publication. This publication is based upon the information available to Shimadzu on or before the date of publication, and subject to change without notice.

© Shimadzu Corporation, 2013