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FTIR TALK LETTER

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Why You Should Use Macro Programs
— To Simplify Routine Tasks —

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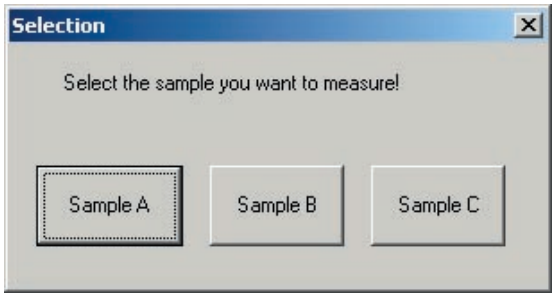
Each generation of Shimadzu FTIR workstation software offers powerful macro functions to simplify various tasks.
The latest IRsolution FTIR control and data processing software inherits this tradition of powerful macro functions. This FTIR Talk Letter introduces the possibilities offered by macro functions.

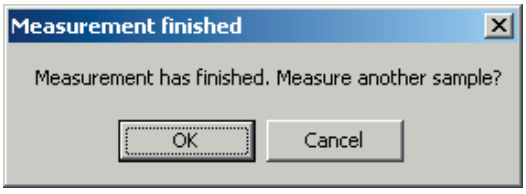
1. Automated Measurement

If measurements are required on two types of sample with the following different measuring conditions for each:
Sample A Resolution: 2 cm^{-1}; wavenumber range: 400 to 4000 cm^{-1}
Sample B Resolution: 4 cm^{-1}; wavenumber range: 750 to 3500 cm^{-1}
In this case, it is necessary to determine which sample is being measured and then ensure that the measurement parameters are correct. This process involves the following steps:
1) Set the resolution to 2 cm^{-1} (2 clicks);
2) Set the wavenumber range: 400 to 4000 cm^{-1}
(2 clicks + keyboard entry);
3) Measure the background (1 click);
4) Load sample A;
5) Measure sample A (1 click);
6) Unload sample A;
7) Set the resolution to 4 cm^{-1} (2 clicks);
8) Set the wavenumber range: 750 to 3500 cm^{-1}
(2 clicks + keyboard entry);
9) Measure the background (1 click);
10) Load sample B;
11) Measure sample B (1 click); and
12) Unload sample B.
If this process is handled by a macro, the following dialog box is displayed.



Click [Sample A] to set the measurement parameters for sample A and measure the background. The following dialog box is displayed when the background measurement is complete.

Load sample A in the sample compartment and click [OK] to start sample measurement.
When sample A measurement are completed, a dialog prompts whether to measure another sample. Click [Yes] to display the sample selection dialog box again. Click [Sample B] to measure sample B in the same way.

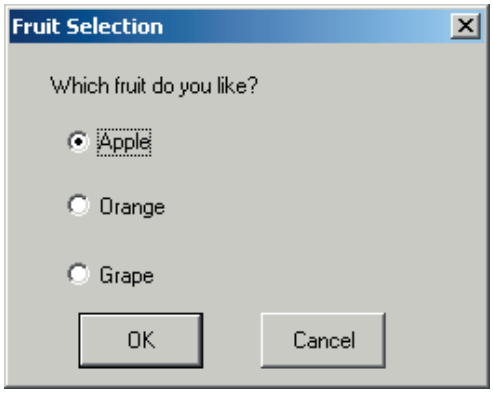


When handled by a macro, the process described above can be summarized as follows:
1) Run the macro;
2) Click the [Sample A] button;
3) When the background measurement is complete,
Load sample A, and click [OK];
4) Unload sample A;
5) Click [Yes] in the measurement complete dialog box;
6) Click the [Sample B] button;
7) When the background measurement is complete,
Load sample B;
8) Click [OK], and
9) Unload sample B to complete the measurements.

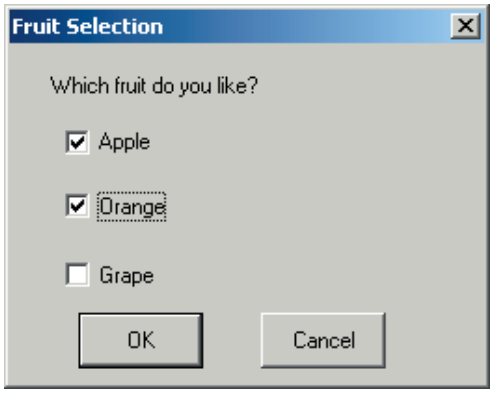
Comparing the required number of mouse clicks and keyboard entries shows:
Manual operation: 13 mouse clicks + several numeric entries from the keyboard
Macro operation: 6 mouse clicks
Using the macro reduces the number of mouse operations required. No numeric entry is required, which eliminates the chance of careless errors.

2. Simplification of input

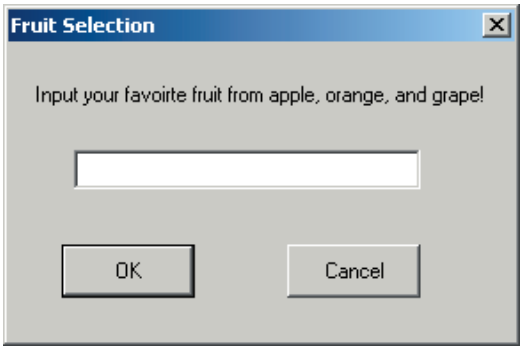
IRsolution macro has more freedom of designing user interface than previous software. Many of parts to receive user input (these are called "control")can be available on IRsolution. You can build in useful user interface bycombining these controls.
Available controls are shown below.



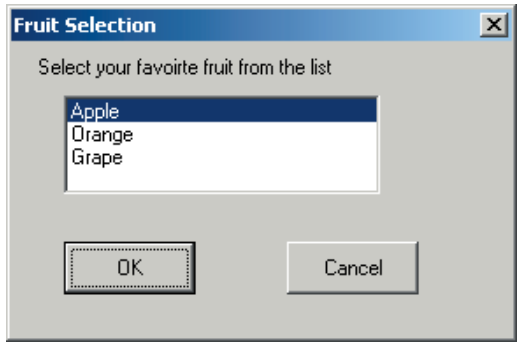
Radio buttons (to select a single item from multiple options)



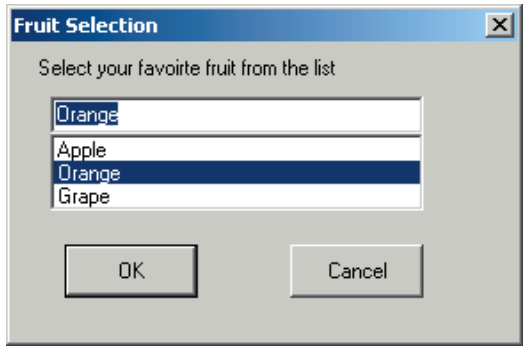
Check boxes (to select multiple items from multiple options)



Edit box (for keyboard entry)



List box (to select from preset character strings)



Combo box (to select from preset character strings or make a keyboard entry)

Dropdown list box (a list box to select from preset character strings – ▼ button displays the candidate items)

3. Automation of Complex Data Processing

Automating multiple data processing operations in a designated sequence with a macro ensures they are conducted reliably. The measured data is processed after measurements are complete and only the processed results are displayed.

4. Automation of Special Data Processing

Some special data processing, which is available by combining some standard data processing, can be done with easy operation by macro program. In case you calculate the corrected height of a peak by selecting peak top and peak bottom, you need many steps of operation to get the final result. You can decrease most of the steps by macro program which can process many steps of operation automatically.

5. Links to External Software

A macro program can call up external programs. This feature uses external programs to expand the functionality of IRsolution, such as accessing the RS-232C port.

6. Finally

Macro programs are introduced above. As macro programs offer higher functionality, it may become difficult for customers to create detailed macro programs. Therefore, Shimadzu handles all tasks from designing the specifications to writing the program and instruction manual. The standard procedure to create a macro program is shown at the right.

Consultation
(Consult your Shimadzu representative.)

↓

Shimadzu R&D contacts the customer
Communications to determine
the internal data processing procedures
and user interface design.

↓

Quotation based on results of meetings
Incorporating development costs

↓

Production starts when order is received

↓

Shimadzu checks operation
of completed program

↓

Installation

We hope that this information assists customers who are considering introducing macro programs to enhance working efficiency.

Microscope Measurement Techniques — Transmission Measurements —

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This FTIR Talk Letter Vol. 3 and 4 introduced the accessories used for IR microscope measurements and how to use them. This FTIR Talk Letter introduces analytical techniques using IR microscope transmission methods.

Diamond cells or a KBr or BaF_2 window material are often used for transmission measurements. However, great care is needed to obtain good quality spectra with such cells. Four important precautions when conducting measurements with a diamond cell are described below.

(1) Using a Diamond Cell

A diamond cell incorporates two cell plates, which compress the sample. Subsequently, measurements are conducted on the sample adhered on a single cell plate. If measurements are conducted with both cell plates, an interference pattern is superimposed over the sample spectrum. Fig. 1 shows the example of caffeine powder analyzed using a diamond cell. The red line shows the spectrum of the sample on a single cell plate; the blue line shows the spectrum measured with the sample sandwiched between two cell plates.

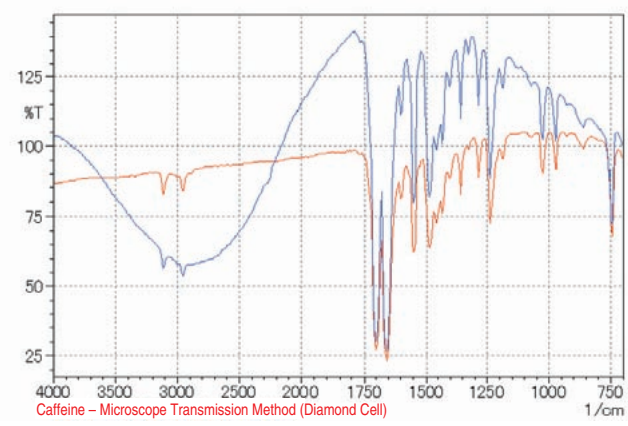


Fig. 1 Transmission Spectra of Caffeine Powder

Fig. 1 shows that an interference pattern is superimposed over the sample spectrum measured using two cell plates, such that the baseline exceeds 100%. The interference pattern occurs due to the parallel space between the two diamond disks. When measurements are conducted with two cell plates, the noise increases near the 2000 cm^{-1} absorption band of diamond.
For the reasons above, after a sample is crushed between the two cell plates, the sample adhered on a single cell plate is analyzed. As an exception, rubber and other elastic samples are measured while sandwiched between the two cell plates.

(2) Sample Quantity

Only a trace amount of sample is required for measurement. Too much sample lowers the baseline and results in a spectrum with saturated peaks. Fig. 2 shows an example of this using a caffeine power sample. The red line shows the spectrum obtained with an appropriate sample volume; the green line is the spectrum for an excess of sample.

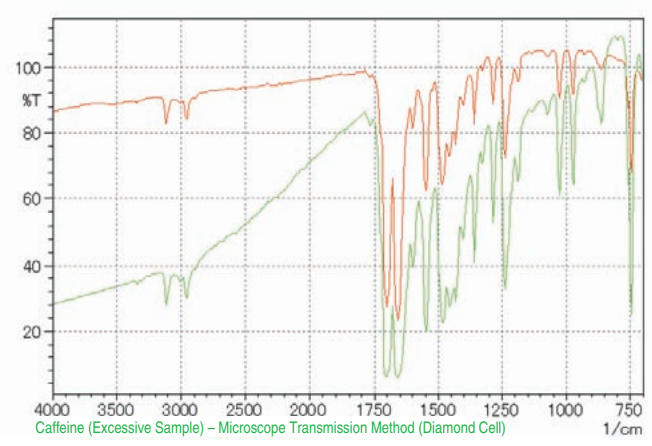


Fig. 2 Transmission Spectra of Caffeine Powder

As an excess of sample cannot be compressed thinly enough, measurements must be conducted on a thick film. This results in a spectrum that is difficult to analyze due to a lower or inclined baseline and saturated peaks, as shown by the green line in Fig. 2.
Therefore, the amount of sample must be adjusted to create a thin layer on the diamond cell.

(3) Background (BKG) Measurement and Sample Measurement Positions

The positions chosen for background measurement and sample measurement have an influence on the spectrum. Choosing a background measurement position as close as possible to the sample measurement position achieves a good spectrum with approximately 100% baseline.
Fig. 3 shows the spectra for cellulose fiber obtained with BKG measured in a corner of the diamond cell (BKG1) and with BKG measured near the sample (BKG2).

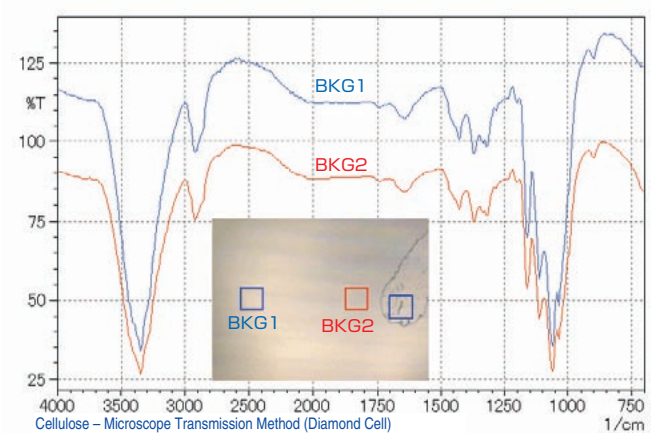


Fig. 3 Transmission Spectra of Fibrous Foreign Matter

The amount of transmitted IR light differs between the center and edges of the diamond cell. The amount of IR light is lower if BKG is measured away from the center of the cell. In this case, sample measurements result in a spectrum with a baseline exceeding 100%. Consequently, position the sample in the center of the diamond cell and measure the background as close to the sample as possible. If the sample is not in the center of the diamond cell, the background must also be measured near the actual sample position, away from the center of the cell.

An interference pattern may appear on the baseline if it is measured on a smooth part of the sample surface. Fig. 4 shows examples of polypropylene (PP) measurements. The insert shows a photograph of the actual measured positions.

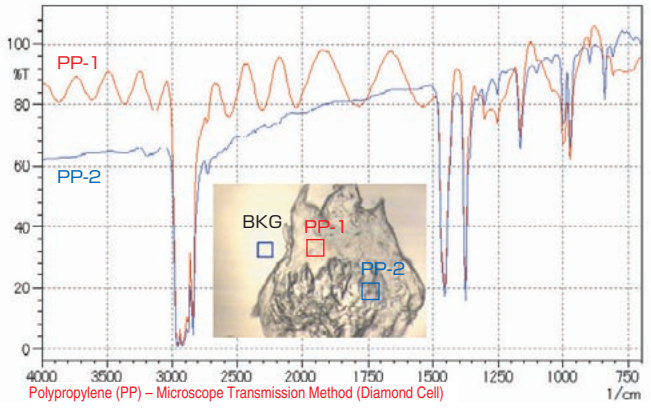


Fig. 4 Transmission Spectra of Polypropylene (PP)

Care is required if an interference pattern appears on the baseline, as the interference pattern may hide small peaks.

(4) Using the Aperture

If the foreign matter (sample) is relatively large, do not increase the aperture. It is more effective to set the aperture to approximately 50μm and measure the spectra at a variety of positions than to increase the aperture. Constant results at each measurement position indicate that the foreign matter is a single component or a uniform mixture. Sample identification and analysis by spectrum searching are possible on a spectrum not affected by peak saturation or an interference pattern.

However, differences apparent between the spectra indicate a non-uniform mixture of foreign matter. In this case, differential spectra are determined to obtain a spectrum for each component.

Fig. 5 shows the measurement of foreign matter on the surface of paper. The aperture is set to 30μm x 30μm.

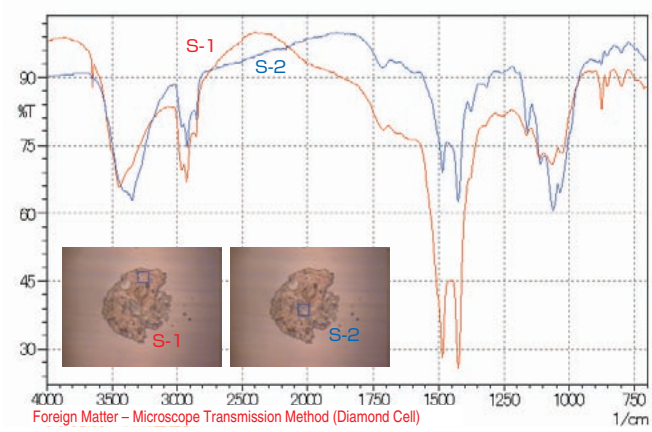


Fig. 5 Transmission Spectra of Foreign Matter on the Surface of Paper

The diagram shows a position of strong intensity near 1500 cm^{-1} (S-1) and a position of strong intensity near 1000 cm^{-1} (S-2). The position near 1000 cm^{-1} results from a normal component of paper (cellulose). Calculating the difference between the spectra from S-1 to S-2 in Fig. 5 gives the spectra for just the foreign matter. Fig. 6 shows the resulting differential spectrum.

Spectrum searching of the differential spectrum in Fig. 6 indicated the foreign matter to be magnesium carbonate.

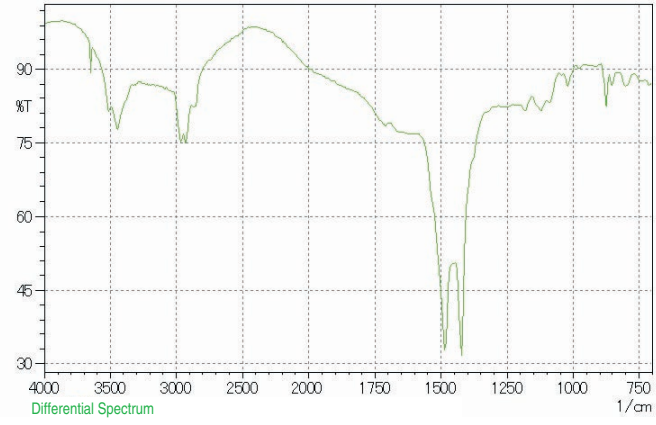


Fig. 6 Differential Spectrum

This FTIR Talk Letter introduced four important precautions when using a diamond cell for transmission methods with an IR microscope. Follow these precautions to more effectively utilize your IR microscope.

Question

Up to what size can I measure with an IR microscope?

Answer

Analysis up to 10μm x 10μm is generally possible, although this depends on the measurement method and type and form of the sample. The size is related to the instrument sensitivity (light intensity) and the wavelength (IR range: 2.5 to 25μm). For example, conducting measurements with the aperture set to 10μm x 10μm may result in the peak broadening or the appearance of ghost peaks in the region exceeding 10μm (up to 1000 cm^{-1}).
Fig. 1 shows an example of analyzing a novolac resin film on a metal plate with different aperture sizes.

To avoid influence of noise, measurements are conducted using different numbers of accumulations for each aperture setting:

30μm x 30μm	Resolution: 8 cm^{-1}; accumulations: 20 (approx. 8 s)
10μm x 10μm	Resolution: 8 cm^{-1}; accumulations: 100 (approx. 40 s)
5μm x 5μm	Resolution: 8 cm^{-1}; accumulations: 400 (approx. 160 s)

Fig. 2 shows a magnified view of the 1300 to 700 cm^{-1} range in Fig. 1. Even at 5μm x 5μm aperture, this view reveals the sample to be novolac resin. However, deformation of the spectrum at the low-wavenumber end is clearly apparent (Fig. 2).
This shows that care is required with spectral distortion at the low- wavenumber end if the aperture is set to 10μm x 10 μm, or less.

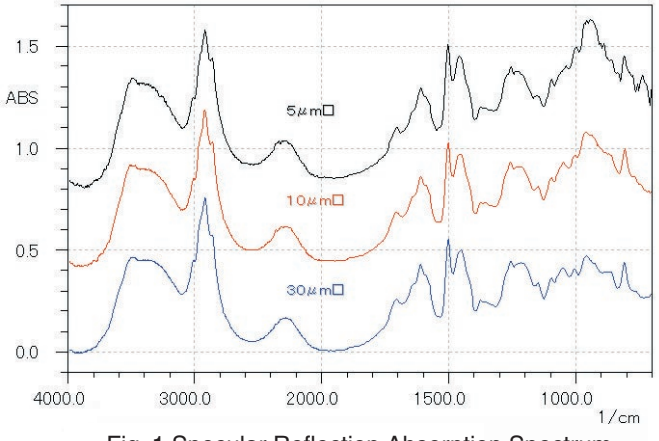


Fig. 1 Specular Reflection Absorption Spectrum of Novolac Resin at 5μm, 10μm, and 30μm Aperture

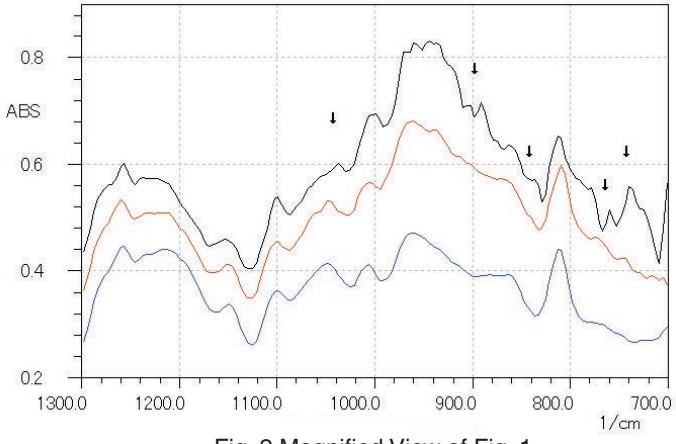
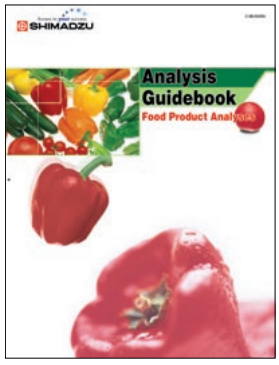
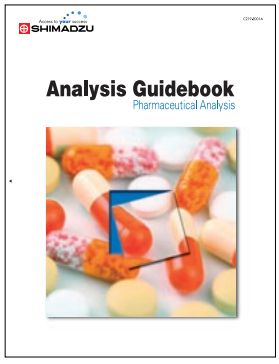


Fig. 2 Magnified View of Fig. 1

Application Guide Books available from Shimadzu



Analysis Guidebook
-Food-



Analysis Guidebook
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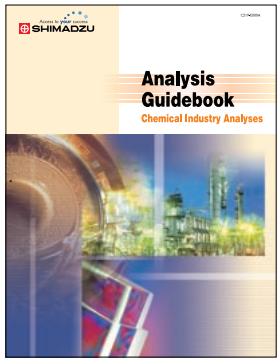

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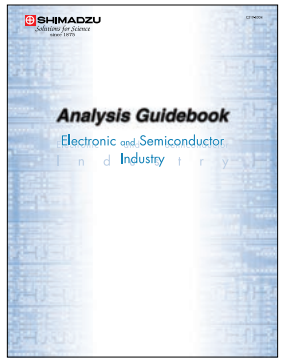

Analysis Guidebook
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Shimadzu Overseas Customer Support

To support customers engaged in the FTIR analysis, Shimadzu has established a global service network incorporating customer support, training and service centers in the USA, Germany, China and Singapore, as well as in Japan. Shimadzu provides comprehensive support services including instrument maintenance, training workshops and the provision of relevant information to meet customer needs regarding both software and hardware.

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