

## Application News

# No. A546

### Spectrophotometric Analysis

## Identification of Contaminant Found in Resin Production Process Using IR Pilot

The Shimadzu IRSpirit launched recently is a compact Fourier transform infrared spectrophotometer characterized by the highest signal to noise ratio and the highest maximum resolution in its class (Fig. 1). The dimensions of the body are W390 × D250 × H210 mm and less than A3 in size. The IRSpirit adopts a unique design which allows not only lateral installation but also longitudinal installation so that it can be installed in a narrow space in width. The IRSpirit also has another feature that it can mount the existing accessories such as a single reflection type attenuated total reflection attachment and a diffuse reflection attachment and the commercially available accessories in addition to the accessories for the transmission measurement such as a demountable cell and a KBr pellet holder.

A dedicated program named IR Pilot to assist the analysis, which allows four measurement items - identification test, contaminant analysis, quantitative analysis and film thickness determination - to be easily operated, is equipped in the LabSolutions IR software which controls the IRSpirit and executes data manipulations. By using the IR Pilot, successive operations of measurement, analysis and print can be orderly performed following the on-screen instructions. The IR Pilot is comprised of twenty-three kinds of the dedicated program of which the often-used program up to four kinds can be registered in its main menu.

This article introduces the contaminant analysis using the IR Pilot.

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Fig. 1 External View of Shimadzu IRSpirit compact FTIR

### Procedures of Contaminant Analysis

The contaminant analysis using FTIR is conducted in a variety of industries and essential to identify the pathway and the source of the contamination.

In general, the contaminant analysis is performed according to the procedures written below.

1. Select a measurement technique suitable to a sample form.
2. Set the FTIR measurement parameters.
3. Measure a background spectrum.
4. Set a contaminant to an instrument.
5. Measure it.
6. Execute data manipulations if needed.
7. Execute spectrum search.
8. Print the results.

There are some settings and selections to note for such successive operations. The IR Pilot supports them. The IR Plot allows the selection of a method and the setting of parameters to be completed only following the on-screen instructions and enables the next operation to be understood. Table 1 shows the conditions used for the measurement.

Table 1 Measurement Conditions

|              |                                                                |
|--------------|----------------------------------------------------------------|
| Instrument   | : IRSpirit-L (KBr window),<br>QuestATR (Wideband diamond disk) |
| Resolution   | : 4 cm <sup>-1</sup>                                           |
| Accumulation | : 45                                                           |
| Apodization  | : Sqr Triangle                                                 |
| Detector     | : LiTaO <sub>3</sub>                                           |

### Contaminant Analysis Using the IR Pilot

The contaminant analysis was performed using the IR Plot. A white contaminant found in a resin production process was measured. After starting the program, "Spectrum" program in the LabSolutions IR is displayed on the screen and the IRSpirit is automatically initialized.

"Main Menu" is displayed as the initial screen of the IR Pilot. Then "Contaminant Analysis" in it is selected (Fig. 2).

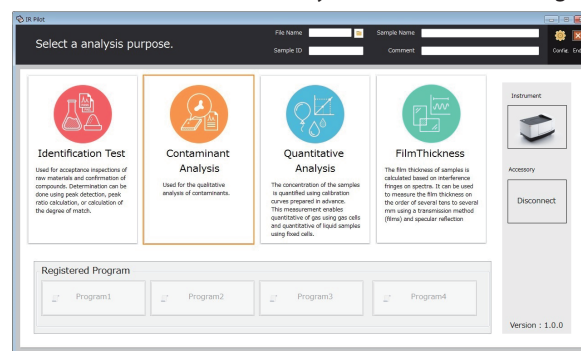


Fig. 2 Main Menu

After executing "Contaminant Analysis", a screen to select a measurement technique is displayed (Fig. 3). When the ATR measurement is performed, "Yes" is chosen. If another technique is used, "No" is selected. If the ATR measurement instrument is automatically recognized, ATR spectroscopy will be selected automatically, and Fig. 3 screen will not be displayed.

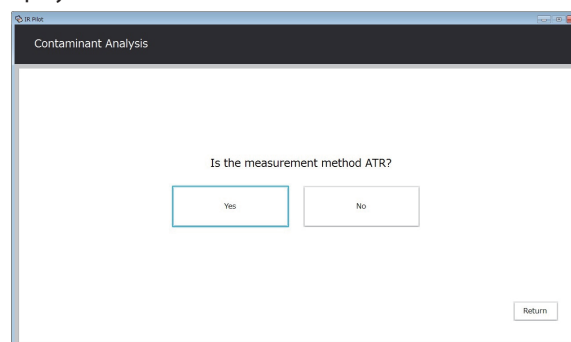
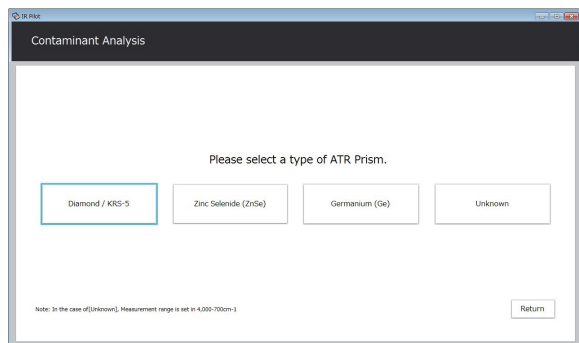


Fig. 3 Screen to Select Measurement Technique

Next, a prism used for the measurement is selected. A diamond prism is often used for ATR spectroscopy. However, when a substance with high refractive index such as black rubber is measured with a diamond prism, its peaks are often distorted by anomalous dispersion of refractive index. In such a case, a germanium prism is used to avoid the distortion. The operable wavenumber range depends on a using prim. The IR Plot automatically set the wavenumber range suitable to a selected prism (Fig. 4). In this article, a diamond prism was used for the measurement.

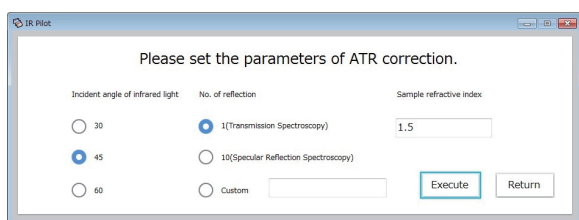


**Fig. 4 Screen to Select Prism**

After selecting a prism, a screen to select the execution of the background measurement is displayed. After confirming that a sample is not on a prism, the background measurement is performed.

When the background measurement is completed, a screen to indicate the sample setting is displayed. After tightly holding a sample on a prism, the sample measurement is performed by pressing "OK" button.

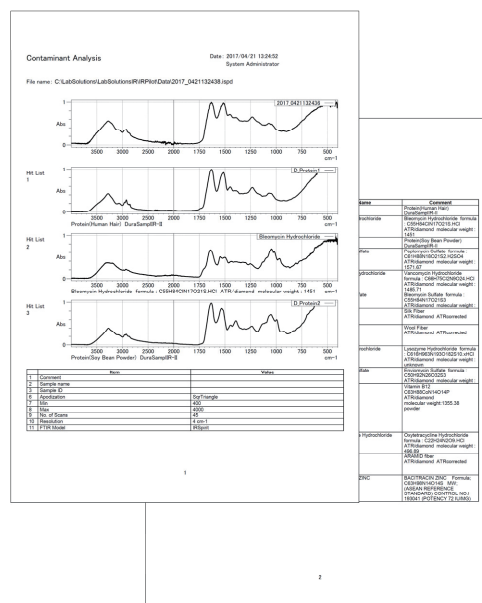
After measuring the sample, a screen to select the execution of ATR correction is displayed. If needed, "Yes" is chosen to execute it. ATR correction requires the input of some parameters such as the incident angle and the number of reflection which are written in an instruction manual of an ATR attachment (Fig. 5). The input of the refractive index of a sample is also necessary. In general, since the refractive index of an organic compound is in the range from about 1.4 to 1.7, 1.5 is input if the refractive index of the sample is unknown. Although the refractive index of the prism is also needed for ATR correction, it is automatically set when the prism is selected. "Execute" button is pressed to execute ATR correction.



**Fig. 5 Screen to Input Parameters for ATR Correction**

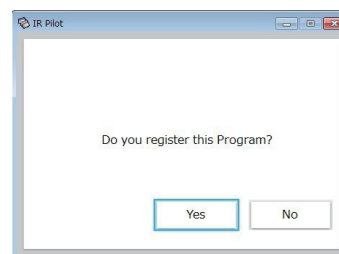
Then a screen to select the execution of spectrum search for a measured spectrum is displayed. If needed, "Yes" is chosen to execute it. The setting of the search parameters is not necessary because it is automatically done. After the spectrum search, the result can be printed. If the print of the result is needed, "Yes" is chosen in the screen to

execute the print. Fig. 6 shows an example of the print. An infrared spectrum of a sample is printed with infrared spectra of the first to third match in the search result to compare it with them. The list up to 20th match is also printed. The search result indicated that the measured contaminant was a kind of protein originating from a human being.



**Fig. 6 Example of Print**

Finally, a screen to register the program shown in Fig. 7 is displayed. The program following a series of operations for the contaminant analysis written in this article can be registered in the main menu. The registration allows the next measurement to be more easily performed because the number of displayed screen is reduced.



**Fig. 7 Screen to Register Program**

## Conclusion

The contaminant analysis was performed using the IR Pilot. The IR Pilot can execute successive operations of the setting of measurement parameters, the background measurement, the sample measurement, the data manipulation, the spectrum search and the print automatically and facilitates the contaminant analysis. The IR Pilot allows a beginner of FTIR and a user conducting the routine measurement to use the Shimadzu IRSpirit more comfortably according to the proper procedures. Please try to use it.