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# FTIR
## TALK LETTER
Vol. 29

# Objective Lenses for Infrared Microscope

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## 1. Introduction

It is said that camera performance is determined by lens performance, and objective lenses are no different in being an important and necessary component that determines the performance of microscopes.

Increasing the Numerical Aperture (NA) of the AIM-9000 Infrared Microscope from 0.5 to 0.7 improved performance compared to the previous AIM-8800 model.

This article explains the structure of the AIM-9000 Infrared Microscope's objective lens and the reasons for the improved performance while comparing the AIM-9000 to the AIM-8800.

Fig. 1 AIM-9000 Automatic Failure Analysis System

## 2. Objective Lens for Infrared Microscope (Cassegrain Mirror)

Instead of a lens system, infrared microscopes use reflecting objective mirrors called Cassegrain mirrors due to (1) limited choices in optical materials that transmit infrared light, and (2) the wide wavelength range of infrared light that is prone to chromatic aberration (Fig. 2).

Fig. 3 compares the objective lens structure of the AIM-9000 to the AIM-8800. Cassegrain mirrors are comprised of a convex mirror and a concave mirror. The Cassegrain mirror in the AIM-9000 has a magnifying power of ×15 and produces an image of a sample on the sample stage at the aperture position. A microscope visible light camera and infrared detector are used to observe the sample image generated at the aperture position (Fig. 3). Camera lens brightness is expressed by FNO (f-number, equation (1)), but the brightness of an objective lens is expressed by its Numerical Aperture (NA, equation (2)).

As shown in Fig. 3, the larger the NA the larger the angle from which light is gathered and the brighter the image.

The AIM-8800 objective lens (mirror) has an NA of sin 30° = 0.5, whereas the AIM-9000 objective lens has an NA of sin 45° = 0.7.

$$\mathrm{FNO} = f/D \quad (1)$$

$$\mathrm{NA} = 1/(2\cdot\mathrm{FNO}) = \sin\theta \quad (2)$$

f : Objective Lens Focal Length

D : Objective Lens Effective Diameter

Fig. 2 Cassegrain Mirror (AIM-9000)

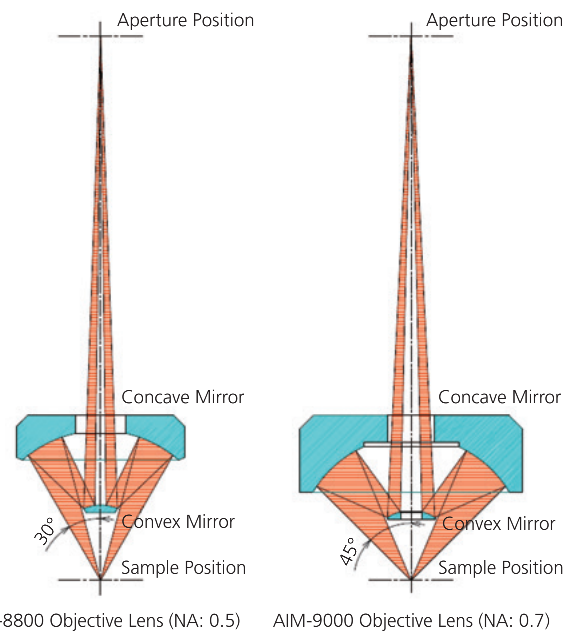



Fig. 3 Objective Lens Comparison (AIM-8800 and AIM-9000)

## 3. Performance Improvement from Changing the NA

This section explains how the performance of an infrared microscope is improved by increasing the NA.

### 3.1 Improved Resolution

The resolution of an objective lens is expressed by the Rayleigh criterion of resolution in equation (3). The larger the denominator (NA) of this equation becomes, the smaller (better) the resolution. Increasing the NA from 0.5 as in the AIM-8800 to 0.7 in the AIM-9000 reduced (improved) the resolution of the objective lens by 1.4 times and allows observation of finer details.

$$\delta = 0.61\frac{\lambda}{NA} \qquad (3)$$

$\delta$ : Resolution

$\lambda$ : Light Wavelength

### 3.2 Brighter Images

Increasing the numerical aperture allows more light to be gathered. Image brightness is expressed by equation (4). Increasing the NA to 0.7 in the AIM-9000 increased image brightness about two-fold. Although other optical system improvements and detector amplifier improvements were also made, increasing the amount of light directed onto the detector overall has resulted in a best-in-class S/N ratio of 30,000:1.

$$\text{Brightness} = K\frac{NA^2}{M^2} \qquad (4)$$

K : Constant

M : Magnifying Power of Objective Lens

### 3.3 Longer Maximum Optical Path Length

The maximum optical path length of infrared light that passes through a sample is expressed by equation (5). The larger the numerical aperture (larger the incident angle) the greater the maximum optical light path through the sample and the stronger the absorbance of infrared light by the sample. Increasing the maximum incident angle from 30° in the AIM-8800 model to 45° in the AIM-9000 (Fig. 4) increased the maximum optical path length 1.2 times. This increased the amount of infrared light absorbed by the sample and increased the peak intensity.

$$L = \frac{T}{cos\theta} \qquad (5)$$

T : Sample Thickness

L : Maximum Optical Path Length

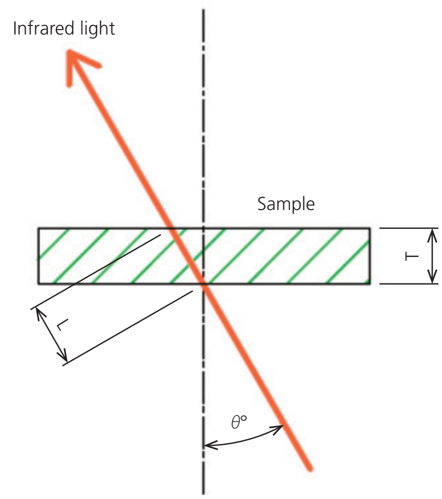



Fig. 4 Optical Path Length through Sample

### 3.4 Smaller Penetration Depth for Attenuated Total Reflection (ATR)

The penetration depth of attenuation total reflection (ATR) is expressed by equation (6). This equation shows the larger the incident angle the smaller the penetration depth of infrared light.

$$d_p = \frac{\lambda_1}{2\pi n_1\sqrt{sin^2\theta - (\frac{n_2}{n_1})^2}} \qquad (6)$$

dp : Penetration Depth

$\lambda_1$ : Infrared Light Wavelength

$n_1$ : Refractive Index of Prism

$n_2$ : Refractive Index of Sample

Measurements of black rubber that contains carbon black (hereinafter "carbon") are used as an example of improving performance for ATR.

Carbon is mixed into industrial rubbers used in the manufacture of automotive tires and water supply packing to improve abrasion resistance, but analysis of these rubbers by ATR must be performed carefully due to carbon absorbing all infrared light.

Penetration depth is proportional to wavelength according to equation (6), but the absorptive properties of carbon result in the rise of the baseline absorbance toward the right (See FTIR Talk Letter Vol. 11), which causes distortion of the spectra.

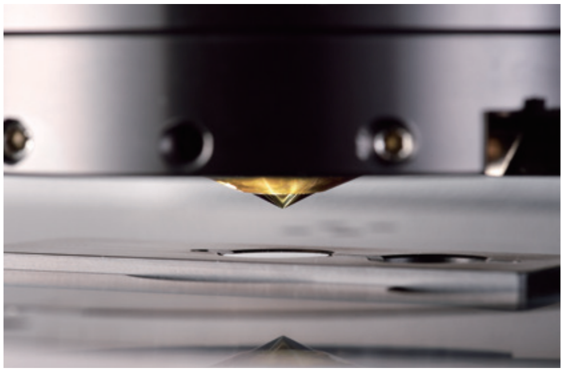
Increasing the maximum incident angle from 30° in the AIM-8800 model to 45° in the AIM-9000 allows for the acquisition of undistorted ATR spectra even when measuring samples of high refractive index that contain carbon.

Fig. 5 shows the ATR measurement results of NBR (acrylonitrile-butadiene rubber).

### ATR Measurement of Black Rubber

Acrylonitrile-butadiene rubber (NBR) containing 50 wt% carbon was measured with an ATR objective mirror (Ge prism).

Using the AIM-9000, a clear peak of C=C-H out-of-plane bending mode was obtained at 970 $cm^{-1}$, which was distorted when measured with the AIM-8800 model.



Sample: NBR with 50 wt% carbon content

Measurement Conditions: Aperture size 50 × 50 µm

Number of Scans: 20 (about 10 seconds)

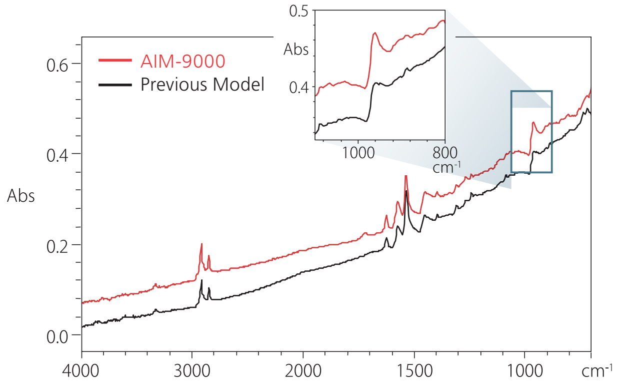



Fig. 5 Example of ATR Measurement of Black Rubber

## 4. Summary

As stated above, increasing the NA in the AIM-9000 improved various performance attributes compared to the AIM-8800 model. For instance, increasing the optical path length through the sample using the transmission/reflection method is expected to enhance peak intensity, and enable the acquisition of accurate spectra for carbon-containing black rubber with ATR measurement as described above.

Similar to the FNO (F-number) of a camera lens, the larger the NA of an objective lens the better its performance. However, the larger the NA becomes (the larger the incident angle becomes) the larger and heavier the objective lens becomes. Increasing the NA also increases aberration effects due to deviation of light beam from the paraxial trajectory, thereby increasing greatly the level of difficulty of lens design. Behind improvements in performance lies superior lens design technology.

# Introduction of Shimadzu Compact FTIR Spectrophotometer

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Shimadzu has launched IRSpirit, a new compact FTIR spectrophotometer. IRSpirit can be carried and used in small spaces while coming equipped with a variety of new functions. This article introduces the features and usability of IRSpirit.

## 1. Introduction

IRSpirit is a compact FTIR spectrophotometer with best-in-class S/N ratio and exceptional resolution (Fig. 1). The unit has a body size smaller than a piece of A3 paper (W390 × D250 × H210 mm), and employs a unique design that allows access from two directions, which enables the unit to be installed in locations with limited space. IRSpirit is compatible with accessories for transmission measurement with liquid cells and KBr pellets, as well as other existing and 3rd party accessories such as a single-reflection ATR measurement attachment and a diffuse reflectance attachment.

Fig. 1 IRSpirit, Portable FTIR Spectrophotometer

Fig. 2 IRSpirit Installed in a Draft Chamber (equipped with the ATR attachment, which is integrated with the sample compartment)

## 2. Even Fits in Small Spaces

Figs. 2 and 3 show IRSpirit being used while installed in a draft chamber. IRSpirit meets the needs of users wishing to install an FTIR spectrophotometer within a laboratory with limited space. IRSpirit can also be installed and used for measurement with the left side facing forward. This allows the user to perform measurements in locations with limited frontal space. IRSpirit is designed to allow operation of the instrument start switch and confirmation of the humidity indicator from both the front and side of the unit.


Fig. 3 IRSpirit Installed with Left Side Facing Forward

## 3. Newly Developed Dedicated IR Pilot Program

The LabSolutions IR software for IRSpirit comes with a newly developed dedicated program called IR Pilot. IR Pilot is a package of 23 applications that are macros created for performing four different types of measurements: identification test, contaminant analysis, quantitative analysis, and film thickness measurement. The user simply has to follow the instructions on screen to perform the correct procedures from measurement to data analysis and printout of results. There is no need to set complex parameters. Even users with minimal FTIR experience can analyze samples by simply selecting the analysis purpose and accessory. Multiple samples can also be analyzed consecutively with a single click. Additionally, up to four shortcuts to the most commonly used programs can be added to the main menu.

## 4. Contaminant Analysis with IR Pilot

Using FTIR spectrophotometry to analyze for contaminants is normally performed by the following procedures.

1. Select appropriate measurement method for the sample.
2. Set FTIR measurement parameters.
3. Perform background measurements.
4. Place contaminant in the instrument.
5. Perform sample measurements.
6. Perform data processing as required.
7. Perform a spectrum search.
8. Print out results.

A number of these steps involve configuration and option selection requiring due care and attention. IR Pilot navigates the user through method selection and parameter configuration via successive software panels displayed on screen.

The next section introduces an example of contaminant analysis performed using IR Pilot. The contaminant to be measured is a white contaminant discovered in the manufacturing process of a plastic product. Measurement parameters are shown in Table 1.

Table 1 Equipment and Analytical Conditions

| | |
|---|---|
| Equipment | : IRSpirit-L (KBr window plate), QATR-S (wide-band diamond disk) |
| Resolution | : 4 $cm^{-1}$ |
| Number of Scans | : 45 |
| Apodization Function | : Sqr Triangle |
| Detector | : $LiTaO_3$ |

When starting the IR Pilot, the spectral measurement window is displayed and initialization of IRSpirit is started automatically. After the main menu is displayed, click [Contaminant Analysis].

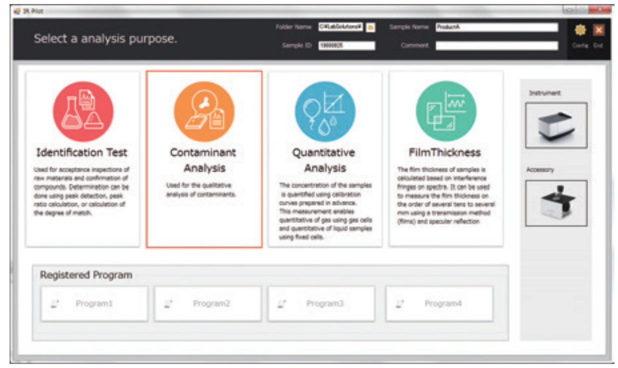



Fig. 4 IR Pilot Main Menu

When [Contaminant Analysis] is selected, a measurement method selection window as shown in Fig. 5 is displayed. Measurement is performed by ATR this time, so click [Yes]. Click [No] to use a different measurement method.

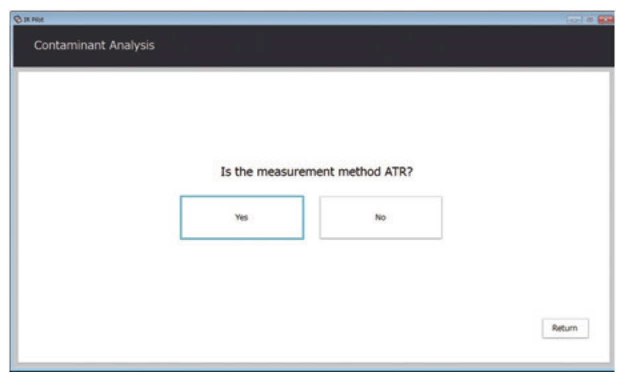



Fig. 5 Measurement Method Selection Window

Next, select the ATR prism to be used. A diamond prism is often used for ATR. However, using a diamond prism for measurement of high-refractive index samples such as black rubber can result in distorted spectra. Because of this, a germanium (Ge) prism is used for measurement of high-refractive index samples. Although the measurable wavenumber range changes depending on the type of ATR prism used, IR Pilot automatically configures the measured wavenumber range after the prism is selected, as shown in Fig. 6. On this occasion, a diamond prism is used for measurement.

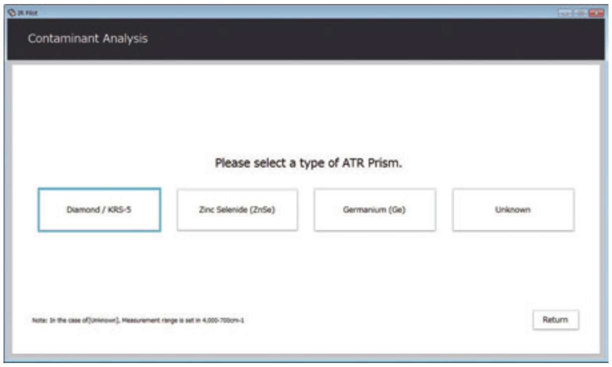



Fig. 6 ATR Prism Selection Window

Once the prism type is selected, a window is displayed that offers the choice of whether background measurements are necessary. Check that no sample is on the prism before performing background measurements. When background measurements are complete, a window is displayed asking the user to place the sample, so attach the sample to the prism and perform measurements.

Once sample measurements end, an ATR correction selection window is displayed. Perform ATR correction as required. Parameters must be configured to perform ART correction, as shown in Fig. 7. Refer to the instruction manual of the ATR measurement attachment for the incident angle and number of reflections of infrared light. The refractive index of the sample is also needed. If unknown, enter 1.5 since the refractive index of organic substances is generally around 1.4 to 1.7. The refractive index of the prism is also needed, but this is determined automatically by IR Pilot. Click [Execute] and ATR correction starts.

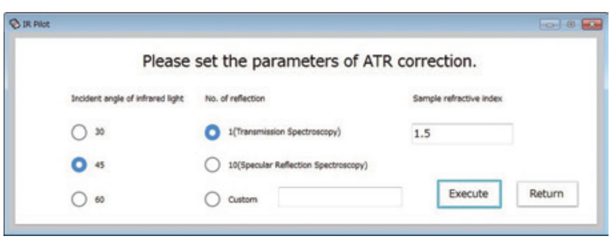



Fig. 7 ATR Correction Conditions Selection Window

Next, a window is displayed that asks whether a spectrum search is required. The spectrum search function is used to identify unknown materials. Search parameters are determined automatically, and a hit list is displayed. A window is also displayed asking whether a printout is required. Clicking [Yes] produces a print out of the spectrum search results. An example printout of spectrum search results is shown in Fig. 8. The printout allows comparison of the infrared spectrum of the sample against the infrared spectra of the top 3 hits from the hit list. Component names and information for the top 1 through 20 hits are also printed out. The white contaminant measured on this occasion is believed to be protein, and speculated to probably be a human-derived contaminant such as skin or a nail.

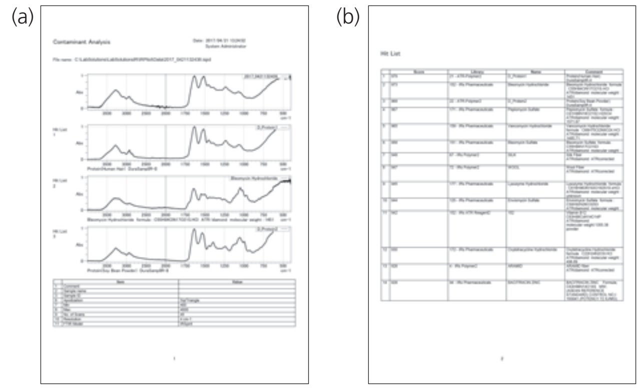



Fig. 8 Printout of Spectrum Search Result
(a) Comparison of Spectra from Sample and Top 3 Hits
(b) Top 20 Hits in the Hit List

Finally, the program registration window is displayed as shown in Fig. 9, which allows the user to add a shortcut for the entire workflow onto the main menu.

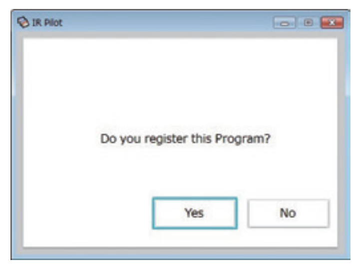



Fig. 9 Program Registration Window

Once registered, the registered program appears on the main menu as shown in Fig. 10. Selecting this program reduces the number of options to be selected and simplifies execution of the same procedure when used again.

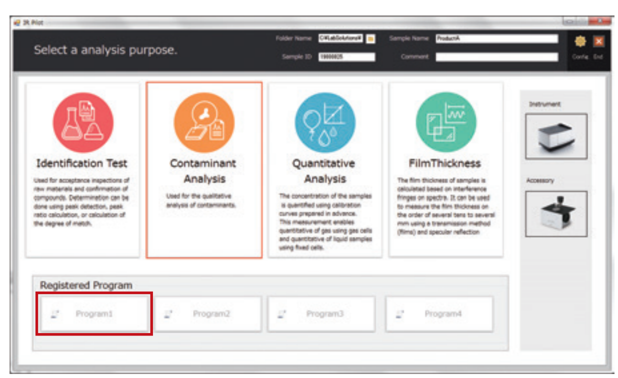



Fig. 10 IR Pilot Main Menu

## 5. Summary

IRSpirit offers a compact unit size that can be carried and used in small spaces and comes with a variety of new functions. We hope you will be able to experience this compact FTIR spectrophotometer with ready-to-use functionality.


# IRSpirit, Ready to Run

## Space-Efficient with High Expandability

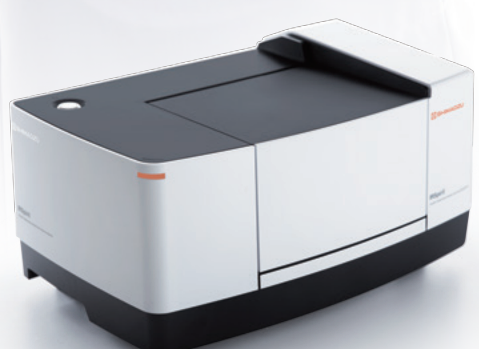
- Compact FTIR that travels where it's needed.
- For sites with only a narrow space available, samples can be measured with the unit positioned horizontally or vertically.
- With the widest sample compartment in its class, it easily accommodates Shimadzu and third-party accessories.

## Dedicated IR Pilot Program Ensures Immediate and Easy System Operation

- IR Pilot includes 23 application programs as standard.
- Includes an identification test program convenient for routine inspections as standard.
- Includes a pass/fail judgment program specialized for contaminant analysis as standard.

## High Reliability Ensures the System Can Be Introduced with Confidence

- Stable interferometer performance based on technology inherited from high-end models.
- Designed to endure even high-humidity environments (KRS-5 window is selectable).
- Instrument status monitoring function enables users to understand the instrument status easily.
- Anti-theft and anti-drop keylock can be installed.



Fourier Transform Infrared Spectrophotometer

# IRSpirit

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