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Application News

Fourier Transform Infrared Spectrophotometer IRTracer™-100
Infrared Microscope AIMsight™
Infrared/Raman Microscope AIRsight™

Sensitivity Evaluation and Example Analysis of Microscopic Targets with Thermoelectrically Cooled MCT Detector

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User Benefits

- A thermoelectrically cooled MCT detector can measure microscopic targets down to 25 µm in size without needing liquid nitrogen.
- An MCT detector is compatible with transmission, reflection, and ATR method for analyzing a range of sample types.
- For targets smaller than 25 µm, the standard T2SL detector can measure targets down to 10 µm in size with good sensitivity.

■ Introduction

Infrared spectroscopy is extremely useful for qualitative analysis of organic compounds and some inorganic compounds. Infrared microscopy can be used for spectroscopic analysis of microscopic targets that are otherwise difficult to analyze. The AIMsight Infrared Microscope and the infrared mode of the AIRsight Infrared/Raman Microscope are equipped as standard with the T2SL detector, which can analyze microscopic targets down to 10 µm in size if it is cooled with liquid nitrogen. However, the difficulties associated with procuring and handling liquid nitrogen mean there is increasing demand for infrared spectroscopic analysis of microscopic targets without using liquid nitrogen.

The thermoelectrically cooled (TEC) MCT detector is a new optional detector in Shimadzu's product lineup that can analyze microscopic targets down to 25 µm in size without liquid nitrogen. This Application News compares this new detector with existing detectors and presents two example analyses of microscopic targets using the new detector.

Fig. 1 IRTracer™-100 and AIMsight™

■ Detectors Used in FTIR Spectroscopy

Infrared detectors can be broadly divided into thermal detectors and quantum detectors.[1)]

Thermal detectors convert the energy of infrared light into heat and use the pyroelectric effect or thermo-electromotive force to output an electrical signal. Thermal detectors are used as general-purpose detectors because of their compatibility with a wide range of wavelengths and room temperature. However, thermal detectors cannot detect the very weak infrared signals used in infrared microscopy.

Quantum detectors are semiconductor detectors that measure the change in sensor resistance caused by the photoelectric effect or photovoltaic effect in response to incident infrared light. Their detecting power is over 100 times greater than thermal detectors. They are often used for analysis in low light conditions such as infrared microscopy, reflection absorption spectroscopy (RAS) and gas analysis with long-path gas cells. However, because quantum detectors generate signals from all incident infrared light, the temperature of the sensor element must be lowered to prevent the output signal from being drowned in noise. This is why liquid nitrogen is normally used to cool quantum detectors. But there is a growing reluctance to use liquid nitrogen because of the risk of oxygen deprivation if it vaporizes in a closed room or frostbite if it comes into contact with skin.

■ Comparing Three Different Detectors

The Shimadzu's infrared microscope and the infrared mode of infrared/Raman microscope is equipped as standard with the T2SL quantum detector, which must be cooled with liquid nitrogen. The DLATGS thermal detector, which operates at room temperature, is also available as an optional detector, but it lacks the sensitivity to produce good data from microscopic targets several tens of micrometers in size. Now, Shimadzu is offering the TEC MEC detector, a quantum detector cooled with a Peltier element, as an optional detector. This Application News compares the TEC MCT quantum detector with the T2SL quantum detector and the DLATGS thermal detector.

TALC-containing polypropylene plastic, which is used to produce automobile bumpers, was analyzed, and spectroscopic measurements were obtained by transmission analysis. The conditions used during analysis are shown in Table 1. The sample was analyzed on a diamond cell with the aperture sizes shown in Table 1.

Table 1 Measurement Conditions

Instruments:	IRTracer-100, AIMsight
Aperture:	25 × 25 µm (TEC MCT, T2SL), 100 × 100 µm (DLATGS)
Resolution:	8 cm^{-1}
Number of scans:	20 scans (T2SL), 500 scans (TEC MCT, DLATGS)
Apodization function:	SqrTriangle
Detector:	TEC MCT, T2SL, DLATGS

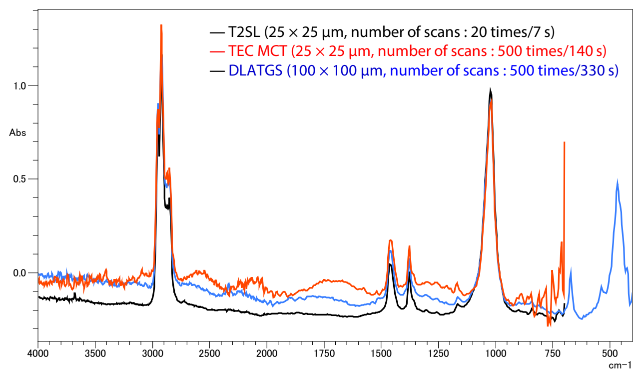

Fig. 2 Comparing Spectra Acquired with Three Detectors

Comparing the infrared spectra acquired by each of the three detectors, the TEC MCT detector could acquire an infrared spectrum with minimal noise from a microscopic target 25 × 25 µm in size (Fig. 2), However, it requires more scan time in comparison to the T2SL detector. The DLATGS thermal detector lacks the sensitivity to produce good data from a microscopic target as small as several tens of micrometers. However, unlike the TEC MCT and T2SL detectors, it produces data at low wavenumbers (700 to 400 cm^{-1}), so it can be used to analyze compounds with absorption in this wavenumber range. Only the TEC MCT detector or the DLATGS detector can be added as an option to the standard T2SL detectors. they cannot be installed simultaneously.

As mentioned, the TEC MCT detector requires more scans than the T2SL detector to produce a good spectrum with little noise, so this section evaluates the aperture size and number of scans of each detector. The spectrum acquired with the TEC MCT detector (the red line in Fig. 2) was obtained by 500 scans over an analysis time of 140 seconds.

In Fig. 3, infrared spectra acquired with the TEC MCT detector at different aperture sizes and the numbers of scans (time) are compared. The aperture sizes and numbers of scans used are shown on the graph and other measurement conditions are the same as those in Table 1.

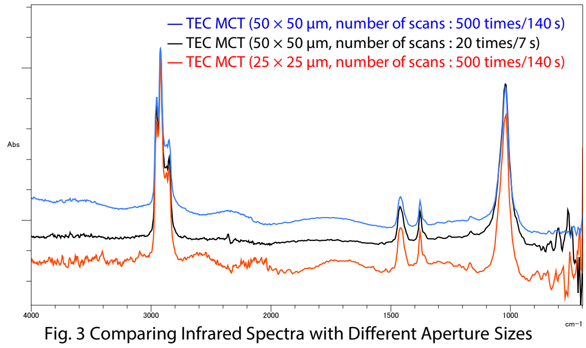

Fig. 3 Comparing Infrared Spectra with Different Aperture Sizes and Numbers of Scans

Increasing the aperture size to 50 × 50 µm reduced the noise dramatically for the same number of scans (the blue line in Fig. 3), but it also offered good data from just 20 scans with a measurement time of 7 seconds (the black line in Fig. 3).

■ Example Analysis 1 (Reflection Measurements of Substance Attached to Board Terminal)

Contaminants adhered to board terminals can cause poor conduction in electronic parts. We used the TEC-MCT and T2SL detectors to analyze a substance found attached to a board terminal.

Fig. 4 shows a photograph of the substance attached to the board terminal and the infrared spectra measured with the TEC MCT and T2SL detectors. Since the substance is adhered to the surface of the board terminal and can reflect infrared light, the spectra were measured by reflection method with no sample pretreatment. The aperture size was set to 30 × 40 µm and all other measurement conditions were the same as those in Table 1. Although spectroscopy by reflection measures a smaller amount of light and has a somewhat lower sensitivity than spectroscopy by transmission, the results show microscopic samples smaller than 50 µm can be measured by reflection without liquid nitrogen. Also, although the search results are not presented here, the spectral profile of the adhered substance matched quite closely with a material found in an area away from the circuit board pattern, suggesting it was solder resist, a material used to protect the circuit pattern.

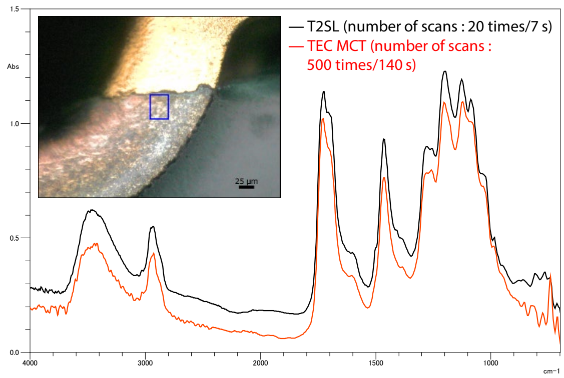

Fig. 4 Photograph and Infrared Spectra of Substance Attached to Board Terminal

■ Example Analysis 2 (ATR Measurements of Microscopic Contaminant)

Quality issues caused by contamination are prevented by investigating sources of contamination at the manufacturing site and taking appropriate measures against them. The key to this is to identify what kinds of contaminants are present at the site. This can be done by using an adhesive roller to periodically clean workstations and other areas at the manufacturing site and then use infrared microscopy to analyze potential contaminants collected on the roller. Fig. 5 shows the infrared spectra of a contaminant collected on an adhesive roller measured by direct ATR using the TEC MCT and T2SL detectors. The aperture size was set to 200 × 200 µm, and all other measurement conditions were the same as those in Table 1. Spectroscopy by ATR, which measures a sample pressed up against a prism, can analyze samples not suitable for transmission or reflection method. With the ATR method, rather than measuring the full area of the set aperture size, the measured area is determined by the size of the light-receiving surface in the detector and the magnification from the light-receiving surface to the aperture settings position. It is recommended that a large aperture size be used when measuring with the ATR method to achieve good sensitivity.

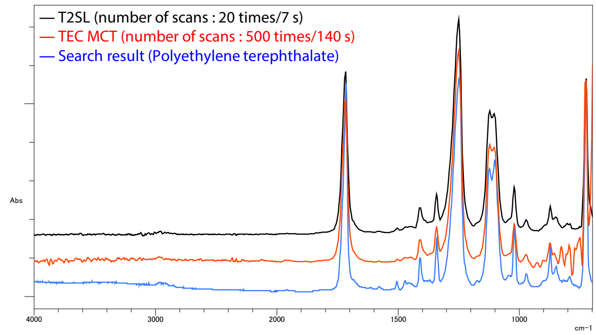

Fig. 5 Infrared Spectra and Library Search Result for Microscopic Contaminant Collected on Adhesive Roller

With the TEC MCT detector, even ATR measurements can be performed without liquid nitrogen. Based on a spectral library search, which showed the spectral profile of the contaminant was similar to that of polyethylene terephthalate, the contaminant was assumed to be a polyester-based material.

■ Conclusion

This Application News compares the sensitivity of the new TEC MCT detector with existing detectors and presents two example analyses performed using the TEC MCT detector. The comparisons show the TEC MCT detector, which operates without liquid nitrogen, can measure targets as small as 25 µm with good sensitivity, and it is compatible with reflection and ATR spectroscopy as well as transmission spectroscopy for analysis of a range of sample types.

The T2SL detector, which requires liquid nitrogen, is recommended when analyzing targets smaller than 25 µm or when using fewer scans to shorten the analysis time. However, the minimum target size for infrared spectroscopic analysis with the T2SL detector is 10 µm, so targets smaller than 10 µm, Raman spectroscopy with the AIRsight infrared/Raman microscope should be used.

<References>

1) FTIR TALK LETTER vol.12: Using Infrared Detectors —Pyroelectric Detectors—

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01-00826-EN First Edition: Jan. 2025