

# FTIR

## TALK LETTER

### Vol. 24



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# Lasers Used in FTIR Systems (2)

## —Comparison of He–Ne and Semiconductor Lasers—

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A key feature of Fourier transform infrared spectrophotometers (FTIR) is high wavenumber accuracy. This feature is achieved by using a He–Ne laser<sup>\*1</sup>, which provides very high oscillation wavelength accuracy for the reference beam used to accurately measure an interferogram<sup>\*2</sup>. For that reason, FTIR systems have generally used He–Ne lasers, but in recent years, low-cost and compact FTIR systems that use semiconductor (diode) lasers have started appearing on the market. However, with semiconductor lasers, the center wavelength of oscillation differs for each individual lasers; as a result, this requires adjusting various instrument parameter settings for each laser. Furthermore, any fluctuations in the semiconductor laser element temperature or input current can cause the center wavelength of oscillation to vary, resulting in wavenumber deviation. This article discusses the operating principle and problems associated with semiconductor lasers used in such low-cost/compact FTIR systems, in comparison to He–Ne lasers.

### 1. Semiconductor Lasers

The operating principle of He–Ne lasers was described in *FTIR TALK LETTER* Vol. 18: Lasers Used in FTIR Systems, which explained how He–Ne lasers emit light with a uniform wavelength and phase by inducing a chain reaction of light emission as gas molecules excited to a higher energy state transition to a lower energy state (induced emission). Now consider the semiconductor laser. What principle does it use to emit light with a uniform wavelength and phase? Semiconductor lasers are a type of light emitting diode, like the blue light emitting diode that drew attention for winning the 2014 Nobel Prize in Physics. Consequently, semiconductor lasers are also commonly referred to as diode lasers or LD (laser diodes). Just as with other types of diodes, it works on the same principle as common LEDs (laser emitting diodes). When a voltage is applied to the band gap that forms in the depletion layer of a p–n junction, electrons from the n-type semiconductor are able to recombine with electron holes in the p-type semiconductor, releasing energy in the form of light. In the case of LEDs, the wavelengths that can be emitted correspond to the band gap energy. Therefore, the wavelength is approximately uniform. However, depending on the impurity doping status, a variety of transition processes can occur, resulting in light with a range of wavelengths and non-uniform phase. Unlike LEDs, semiconductor lasers must emit light with a specific wavelength and uniform phase. Therefore, semiconductor lasers include a double heterojunction<sup>\*3</sup> structure (see Fig. 1), where the middle layer is referred to as the active layer. In a normal p–n junction, the electrons injected from the n-side to the p-side diffuse a distance of about 2 to 3  $\mu\text{m}$ , but in double heterojunctions, electrons are trapped without diffusing by an additional energy barrier at the boundary between the active layer and p-side of the semiconductor. This allows a distribution of inverted carriers to be easily created, as was described when explaining the light emission

principle of He–Ne lasers. In addition, the active layer is made of a substance with a higher refractive index than the p-type or n-type semiconductors.

Consequently, total reflection occurs at the boundary, so that the light emitted from electrons recombining with holes is trapped within the active layer. When the boundary reflects the light, induced emission occurs due to the excitation of separate electrons, which allows for obtaining light with uniform wavelength and phase.

Lastly, regarding the structure of the resonator, a characteristic of crystals called a cleavage<sup>\*4</sup> is used to cut crystals for double heterojunctions. The surface cut based on a cleavage is called a cleavage plane. Cleavages not only facilitate cutting crystals, but also result in mirror-like surfaces that are a key characteristic of cleavage planes.

Consequently, crystals can be created with parallel mirror surfaces on opposite sides, which allows light to be amplified by reflecting it back and forth between the planes.

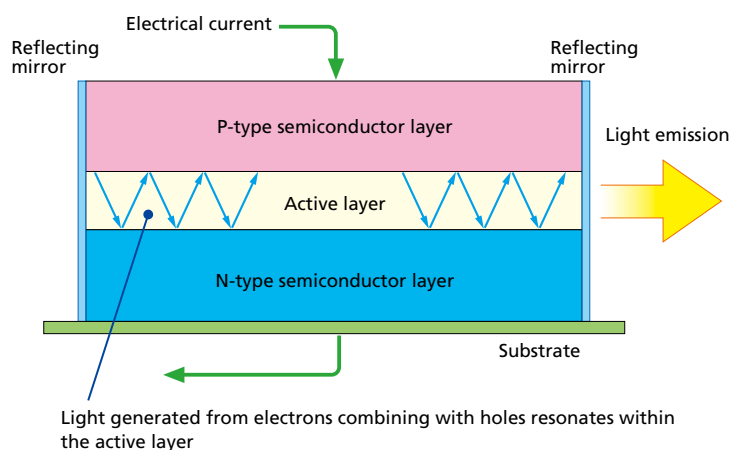


Fig. 1 Structure of Semiconductor Lasers

In the basic semiconductor laser described on the previous page, the fact that crystals must be cleaved precisely and, even if they are successfully created on a wafer, be cut out of the wafer before they can emit light, makes them costly and time-consuming to produce. To help solve such issues, semiconductor lasers called vertical cavity surface emitting lasers (VCSEL) are now available. These lasers have resonators arranged vertically on the substrate. The VCSEL structure is shown in Fig. 2. VCSEL advantages, such as resonators being formed from multilayer semiconductor film and metal film without cleaving, the ability to emit light for viewing laser characteristics while still on the wafer, and the round shape and narrow emission angle of the beam, which makes it easier to fit the optics inside, have resulted VCSELs becoming popular as a low-cost semiconductor laser. The following compares He-Ne lasers to VCSELs, which offer a significant cost benefit.

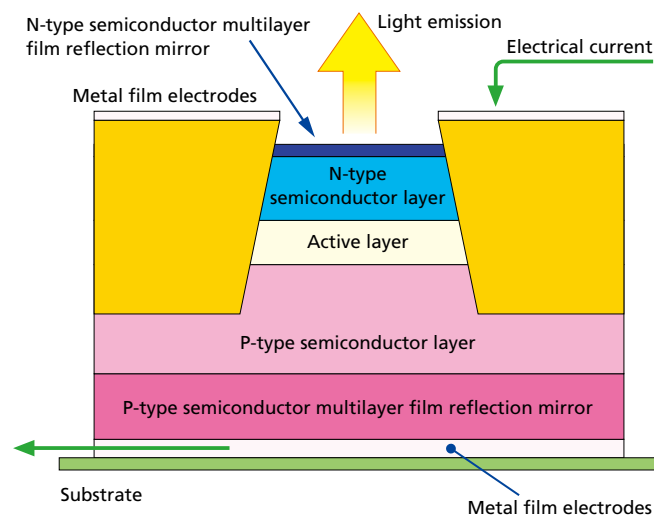


Fig. 2 Structure of Vertical Cavity Surface Emitting Lasers

## 2. Differences between He-Ne Lasers and VCSELs

So far the explanation has mainly focused on how semiconductor lasers emit light. Now, the major differences between He-Ne lasers and VCSELs when used in an FTIR system will be highlighted; this is summarized in Table 1.

Table 1 Comparison of He-Ne Lasers and VCSELs

Comparison Criteria	He-Ne Lasers		VCSELs	
Oscillation Wavelength	O	632.8 nm	X	Various of wavelengths are available, but low-cost lasers used in communications are typically around 850 nm. Due to individual differences, the center wavelength varies within about a $\pm 10$ nm range.
Laser Size	X	Large About 20 to 30 cm long tube shape	O	Small Package about 5 mm in each direction
Ambient Temperature Effects on Oscillation Wavelength	O	Small Ex: $0.0506 \text{ cm}^{-1}/\text{K}$ (for oscillator tube with low-expansion glass)	X	Large Ex: $0.83 \text{ cm}^{-1}/\text{K}$ (for 850 nm center wavelength)
Laser Class <sup>5</sup>	O	Class 2, 2M, or 3R Ex: Class 2 or 2M for 0.5 mW	X	Class 1, 1M, 3R, or 3B for oscillation wavelength of 850 nm Ex: Class 3R or 3B for 0.5 mW
Coherent Distance	O	Long (prone to interference)	X	Short (resistant to interference)
Service Life	X	Shimadzu He-Ne lasers warranted for 30 months	O	5 to 10 years

Note: The information indicated above is for lasers typically used in common FTIR systems.

The "O" symbol in the table indicates a performance advantage for use in FTIR systems and the "X" indicates a disadvantage.

### • Oscillation Wavelength

For He-Ne lasers, the oscillation wavelength is determined by the transition energy of the neon atom from excited to stable state. Consequently, there is no difference in oscillation wavelength between individual devices. Some have other oscillation wavelengths, due to other available differences between excited and relaxed energy levels, but the most common is 632.8 nm for red light. In contrast, electrons can exist at a wider range of energies in VCSELs. In addition, the center wavelength of oscillation can vary by  $\pm 10$  nm due to variability in the impurity concentration in each

semiconductor laser, which is made in the tens of thousands per wafer. FTIR systems use a reference laser as a "measuring stick" for measuring an interferogram and then set the wavenumber after applying a Fourier transform based on the intervals between data<sup>1)</sup>. However, in the case of VCSELs, the data intervals vary for each device, which requires wavenumber correction. Furthermore, due to the large number of lasers with an 850 nm center wavelength used for communication and computer mice, there seem to be many cheap ones available. Whereas the center wavelength of He-Ne lasers is

stable, the band gap energy of VCSELs varies depending on the temperature. That means that even the oscillation center wavelength can vary due to the element temperature or current during laser emission. Consequently, the status of the installed VCSEL must be checked at regular intervals and each instrument parameter corrected to make sure they are consistent with the VCSEL wavelength.

#### • Laser Size

Due to the long cylindrical resonator used for oscillation, He-Ne lasers are about 20 to 30 cm long. In contrast, the crystal in VCSELs is only a few hundred  $\mu\text{m}$  square and is packaged with lead wires in a device only about  $5 \times 5 \times 5$  cm in size, as mentioned above in 1. Semiconductor Lasers. Therefore, VCSELs are better suited for reducing the size of FTIR systems.

#### • Ambient Temperature Effects on Oscillation Wavelength

The oscillation wavelength can be easily changed on VCSELs depending on the input current or element temperature. Consequently, if the element temperature increases due to the input current or ambient temperature, the spacing of the crystal lattice will increase, which will change the status of trapped electrons and, in turn, change the band gap energy. However, because the oscillation wavelength is used as a reference for the reference laser in FTIR systems, it is desirable to have an oscillation wavelength that always remains constant. Any variation in the wavelength will affect wavenumber accuracy in measurement spectra by a corresponding amount. If the VCSEL fluctuates by  $0.06 \text{ nm/K}$ , for example, then a mere  $1^\circ\text{C}$  change in the element temperature will change the wavelength by  $0.06 \text{ nm}$ . Given a center wavelength of  $850 \text{ nm}$ , that corresponds to a deviation of  $0.83 \text{ cm}^{-1}$  in the laser wavelength. The wavenumber  $\nu_i$  for the  $i$ th spectral data point is calculated using the following formula.

$$\nu_i = (i - 1) \times 2\nu_r / N$$

In this formula,  $\nu_r$  is the wavenumber of the reference laser and  $N$  is the number of measurement data points.  $\Delta\nu_i$ , the deviation in wavenumber  $\nu_i$  given a shift  $\alpha$  in the reference laser wavenumber, is expressed by the following formula.

$$\Delta\nu_i = (i - 1) \times 2\alpha / N$$

Based on the above formula, the wavenumber deviation at  $3,060^{-1}$ , for example, would be  $0.21 \text{ cm}^{-1}$ , which means it would be impossible to achieve a maximum resolution over  $0.5 \text{ cm}^{-1}$ . Therefore, high-resolution measurements using a VCSEL require highly precise temperature control.

#### • Laser Class<sup>\*5</sup>

The classification and safety level of lasers are determined by international standards (IEC), as described in *FTIR TALK LETTER* Vol. 18. He-Ne laser

classifications depend on the oscillation wavelength and output level, but given a center wavelength of  $632.8 \text{ nm}$  and  $0.5 \text{ mW}$  output, they are classified as either class 2 or 2M. In contrast, semiconductor lasers with a center wavelength of  $850 \text{ nm}$  and  $0.5 \text{ mW}$  output are classified as class 3R or 3B. Even given the same output level, the class is higher because blinking or other avoidance measures cannot be expected for invisible wavelengths outside the  $400$  to  $700 \text{ nm}$  range. Directly viewing the laser beam is considered dangerous in Class 3B products, so they must be used within the context of a specified control system. Making the system equivalent to a class 1 device (considered inherently safe) requires installing an external filtering device to cut the laser output emitted from the system or installing an interlock device, such as on the access panel.

#### • Coherent Distance

The oscillation wavelength of He-Ne lasers is determined by the fixed transition energy between energy levels. That means spectra are narrower, though there can be some broadening due to a Doppler effect. On the other hand, there is a wider range of electron energy states possible for VCSELs, which means a wider range of possible recombination energies. Furthermore, due to the short resonator, which is only about  $10 \mu\text{m}$  long, the energies exist in multiple resonance states within a wide wavelength range in the resonator. The emission spectrum also broadens due to the effects of resonance states for wavelengths other than the center wavelength. Considering coherence, the narrower the spectrum, the longer the coherent distance, which is an advantage. This makes it easy to create an interference state used to match phases when recombining the beams split by the beam splitter in FTIR systems. However, if light that was made coherent is propagated for a long distance, its coherence could deteriorate. Since improving FTIR resolution requires lengthening the difference in optical path lengths (the distance the moving mirror travels), the above characteristic is an advantage for the He-Ne laser.

#### • Service Life

The He-Ne laser uses an internal plasma discharge to accelerate electrons. Therefore, the lasers can eventually fail when the electrodes deteriorate. However, as long as the crystal structure inside the VCSEL is not damaged, the VCSEL will not fail to illuminate, with some devices reported to have an MTBF<sup>\*6</sup> value exceeding several tens of thousands of hours. However, the crystal structure is extremely vulnerable to heat. Therefore, they can fail if used at high temperatures or if the interior is damaged due to a power surge, for example.

### 3. Conclusion

The FTIR systems currently sold by Shimadzu (the IRAffinity-1S and IRTracer-100) are able to obtain measurements with a resolution of  $0.5\text{ cm}^{-1}$  and  $0.25\text{ cm}^{-1}$ , respectively. A resolution of  $0.25\text{ cm}^{-1}$  means data points can be plotted at  $0.125\text{ cm}^{-1}$  intervals. However, as explained in 2. Differences between He-Ne Lasers and VCSELs, if a VCSEL is used for the reference laser, then a  $1\text{ }^{\circ}\text{C}$  change in the element temperature will cause a  $0.21\text{ cm}^{-1}$  (at  $3,060\text{ cm}^{-1}$ ) shift in the wavenumber, which would prevent obtaining the accurate wavenumber data required for high-resolution measurements. That is why Shimadzu FTIR systems use a He-Ne reference laser to ensure high wavenumber accuracy and high-resolution measurements. The semiconductor lasers used in low-cost compact FTIR systems, in contrast, introduce many problems, such as oscillation wavelength fluctuation, and require wavenumber correction measures, such as carefully controlling the laser current and element temperature, to compensate for differences between individual VCSEL devices if accuracy is required.

#### Terminology

##### \*1: He-Ne laser

Helium-neon lasers are a type of gas laser that uses a mixture of helium (He) and neon (Ne) gases. The helium atoms energized by electrons accelerated using a plasma discharge collide with the neon atoms to elevate their energy to an excited state and form an inversion distribution. He-Ne lasers are used for interferometer measurements and laser microscopes because they offer extremely high wavelength stability that lasts a long time.

##### \*2: Interferogram

Obtained with a Michelson interferometer inside the infrared spectrophotometer, it is a plot of infrared interference intensity across the moving mirror movement range.

##### \*3: Double heterojunction

A structure with two heterojunctions. A heterojunction is a junction of two differing (heterogeneous) materials. A double heterojunction comprises two heterojunctions. In this case, one junction is between the p-type semiconductor and the active layer. The other junction is between the active layer and the n-type semiconductor.

##### \*4: Cleavage

This refers to the characteristic of crystals to split (cleave) along a specific plane where there are weaker bond strengths between atoms. The resulting face where the crystal was split apart is referred to as a cleavage plane. Cleavage planes are extremely flat and smooth, though there can be differences in flatness and smoothness depending on the type of crystal.

##### \*5: Laser class

JIS C 6802 (which is compliant with IEC 60825-1) classifies lasers into seven classes, as summarized in Table 2. The Japanese Ministry of Health, Labour and Welfare has specified measures to prevent laser hazards for each class as occupational health measures for the workplace. In particular, class 3R or higher laser devices require the following actions.

- Designate a laser device manager.
- Clearly label control areas with signage or other means (class 3B or 4).
- Use an interlock system (class 3B or 4).

Table 2 Laser Classifications

Class	Description
class 1	Inherently safe.
class 1M	Safe to look at with bare eyes. In some cases, viewing 1M lasers using optical devices (such as a magnifying glass or binoculars) may be dangerous.
class 2	Applicable to visible light lasers from 400 to 700 nm. Eyes can be protected through measures to avoid the light, such as by blinking.
class 2M	Applicable to visible light lasers from 400 to 700 nm. Eyes can be protected through measures to avoid the light, such as by blinking. However, in some cases, viewing 2M lasers using optical devices could be dangerous.
class 3R	Looking directly at the laser beam could be dangerous in some cases.
class 3B	Looking directly at the laser beam or its specular reflected light is dangerous. Viewing diffused reflected light is normally safe.
class 4	Viewing diffused reflected light is also dangerous.

##### \*6: MTBF

Acronym for mean time between failures. It is a guideline that indicates how much time, on average, will elapse after usage starts, or after it is restored, before it fails again.

#### Reference

1) Mitsuo Tasumi

*Fundamental Principles and Practical Use of FT-IR Spectroscopy* (Tokyo Kagaku-Dojin, 1986) pp. 36 to 39



# Tips for Successful Spectrum Searches—Part 1 —Utilizing Combined Searches—

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Available search functions for spectral analysis include a combination search function, which searches for both spectra and text. This function can be especially useful for comparing measured spectra to the spectra for expected components or as a means of refining searches when investigating unknown substances, such as during contaminant analysis. An appropriate use of this function can improve the efficiency of identification processes. The following gives examples of using the combination search function and describes key points to consider when using the function.

## 1. Introduction

We routinely receive many questions about spectral analysis from customers using FTIR systems for contaminant analysis. Typically, they are able to obtain the infrared spectrum of the contaminant, but are frustrated with analyzing the data. We often hear that a library search for the infrared spectra obtained does not result in any complete matches or that they are able to determine the areas that differ between the spectra, but cannot interpret specifically what is different about the substances and how. To provide advice on how to analyze such spectra, volume 22 of this publication described using difference spectra.

Subtracting one spectrum from another can highlight the differences between the spectra and make it easier to identify components. Consequently, the difference spectrum process is one of the most important tools in spectral analysis.

However, the combination search function described in this article also can be very useful for investigating whether or not a sample contains a predicted component or for refining searches. An appropriate use of the difference spectrum and combination search functions can improve the efficiency of identification processes.

## 2. Combination Search

Four search functions are available in LabSolutions IR and IRsolution software: spectrum, peak, text, and combination. These functions are displayed on a tab page in the search window, as shown in Fig. 1. The spectrum search function uses the specified algorithm to search for the target spectrum by comparing it to the spectral waveforms registered in the library. Search results are listed in descending order of their search match score (hit quality index or HQI), which indicates how closely it matches the

target spectrum. The text search function searches for spectra based on names (such as registered chemical compound and trade names) and keywords. The combination search function combines both the spectrum and text search functions. It limits searches to library spectra that include the keywords specified for text searching and lists results in descending order of match score.

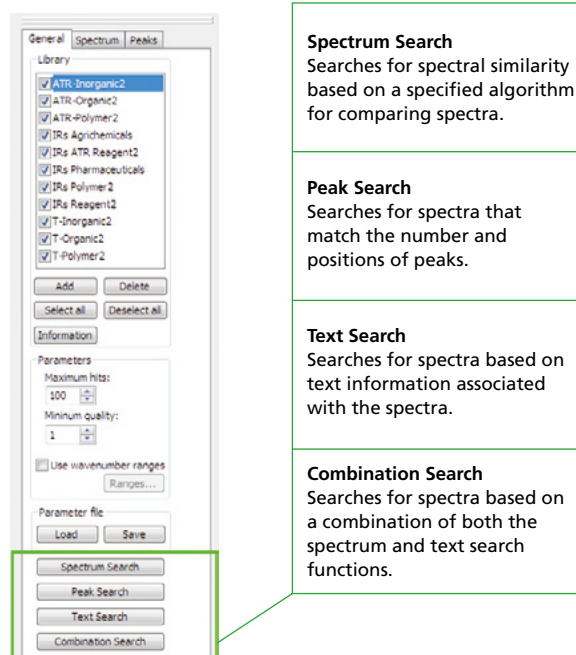


Fig. 1 Tab Page and Functions in the Search Window

## 3. Example of Identification Using the Combination Search Function

*Application News* No. A467 Investigation of Additives in Plastics by FTIR-ATR Spectroscopy described an example of measuring and evaluating the additives in

a commercial plastic bag using a single reflection ATR method. Fig. 2 shows spectrum search results for measurements from the surface of a plastic bag, including a photograph of the bags. It is relatively easy to see that the primary component of the plastic bag is polyethylene (PE).

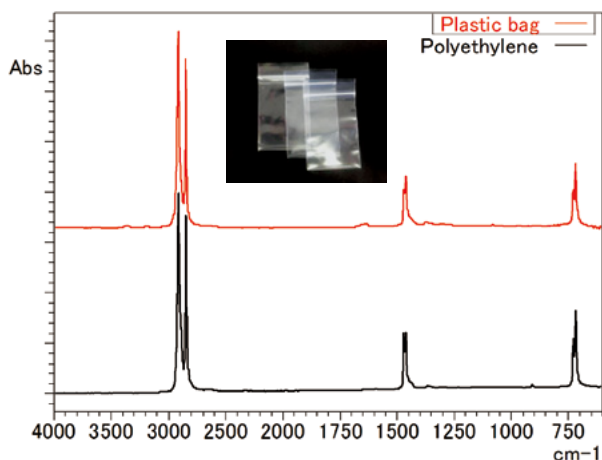


Fig. 2 Infrared Spectrum and Search Results for Plastic Bag

Fig. 3 is an enlarged view near the baseline in Fig. 2. The three peaks indicated with green arrows are presumably from additives in the plastic bag. Search results indicate that the peaks match the spectrum of oleamide. However, identification may not be simple for small peaks such as these, because simply performing a spectrum search may not be adequate for finding a good library match among results listed near the top. As an alternative, difference spectra could be calculated before searching; however, if the differences are particularly small, then it may not be possible to obtain a satisfactory difference spectrum.

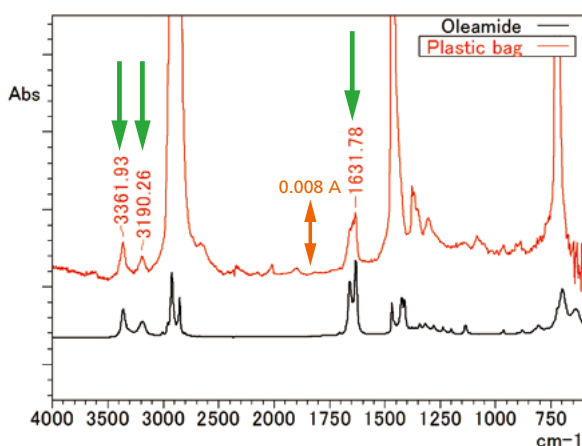


Fig. 3 Oleamide Spectrum Versus Enlarged View Near Baseline in Fig. 2

However, based on such keywords as "low-friction material" and "lubricant," determined from the characteristics and purpose of the measurement sample, candidate components such as stearate could

be identified. In addition, if it is known that a relatively sharp peak between 4000 and 3000  $\text{cm}^{-1}$  indicates the NH functional group, amines and amides can be considered as candidates as well. If the same keywords are used with the text search function, then all components with that keyword information will be listed, regardless of spectral shape. That would require comparing each spectrum individually, which would not be very practical. Therefore, by using the combination search function, the search can be restricted to spectra for components with "stearate" or "amide" in the name. Fig. 4 shows the boxes for entering keywords for combined searches. Fig. 5 shows a comparison of spectra in combination search results. Successively comparing the listed spectra based on the three peaks indicated with green arrows in Fig. 3 reveals that the spectra match components that have a  $\text{-NH}_2$  group, such as an oleic amide or stearic acid amide.

Fig. 4 Boxes for Entering Combination Search Keywords

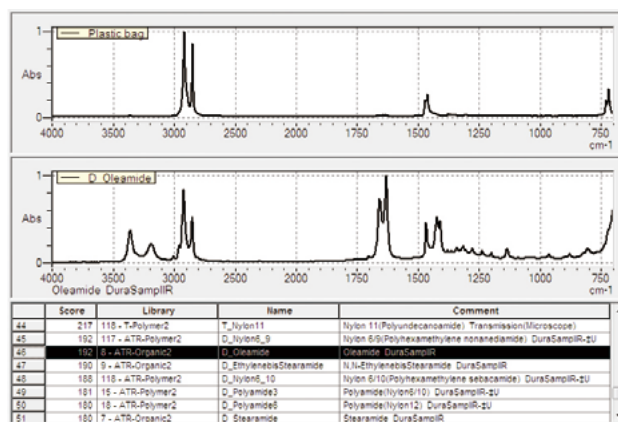


Fig. 5 Comparison of Spectra in Combination Search Results

## 4. Utilizing Sample Information

If the user creates their own library with detailed information not available from infrared spectra, such as the sample color, shape, and size, then the combination search function can be used to refine searches based on that information.

Fig. 6 shows the spectrum search results for a contaminant presumed to be polyethylene terephthalate (PET) using a private library with spectral information registered for seven types of PET products. The information registered in the private library also includes the sample name, compound name, and the sample shape, size, and color. Using the private library alone results in a high score for all seven PET products, which makes it difficult to identify the contaminant.

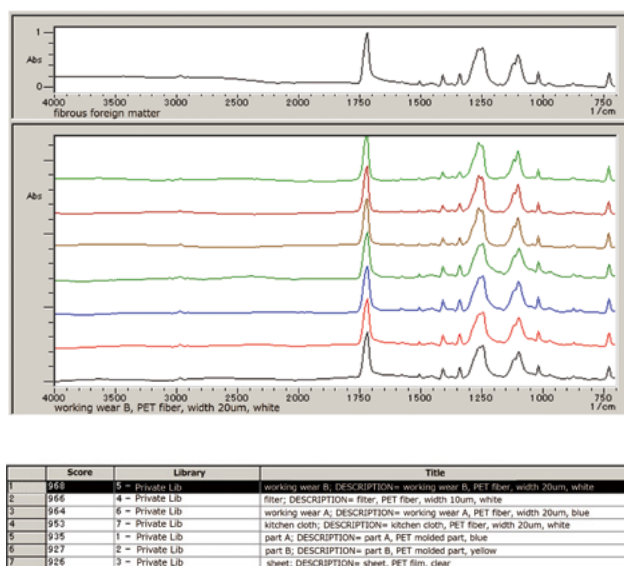


Fig. 6 Spectrum Search Results Using a Private Library

Fig. 7 shows the combination search results from entering information about the appearance as keywords "fiber," "20  $\mu$ m width," and "blue." The refined results show that the contaminant is from Work Clothes A. Therefore, by adding important information about the sample shape, information from other analytical results, and so on, the combination search function can be very useful for identifying contaminants.

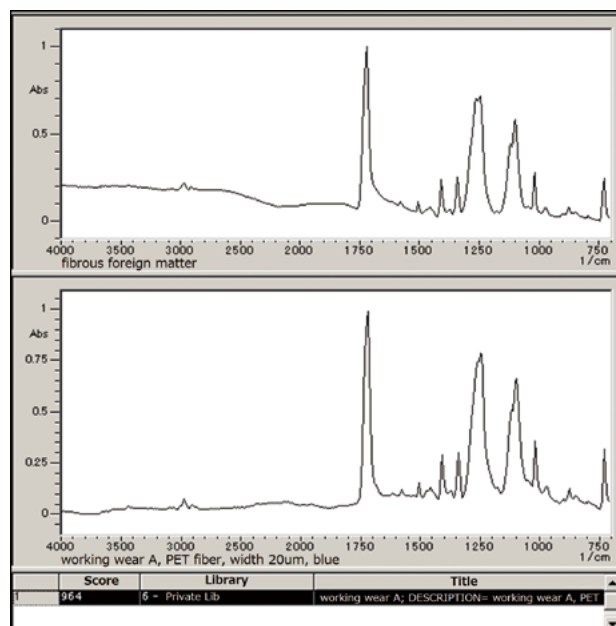


Fig. 7 Combination Search Results Using a Private Library

## 5. Conclusion

When searching spectra to identify an unknown substance, we typically have some suspected substances already in mind, such as objects or chemicals used in a manufacturing process.

In such cases, try using the combination search function. Another data analysis technique is to limit the spectrum search to a narrower range of wavenumbers.

Having knowledge about the peak positions and shapes corresponding to key functional groups can be very helpful for searching and analyzing data efficiently, as illustrated in the example of the amide peaks described above. Therefore, to provide an opportunity to learn about peak information for functional groups, Shimadzu periodically holds training sessions on how to interpret infrared spectra. We hope you will attend. You are welcome to attend even if you do not have a Shimadzu system.



# Q&A

## Question

### How is the search match score calculated?

## Answer

IRsolution and LabSolutions IR software determine the match score by numerically calculating how similar two

spectra are to each other. If the match score evaluation method is set to correlation coefficient or square of the correlation coefficient, then the match score is a value between zero and 1, where the closer the value is to 1, the higher the correlation between the two spectra and the closer the value is to 0, the lower the correlation. The match score value is determined based on the correlation coefficient calculated from plotting a graph of peak intensity values at the same wavenumber from the two spectra. The peak intensity for the target spectrum is plotted on the horizontal axis and the peak intensity for the reference spectrum is plotted on the vertical axis.

In this case, two infrared spectra for polycarbonate (PC) and a spectrum for a copolymer of PC and polybutylene terephthalate (PBT) were compared, as shown in Fig. 1.

The match score can be represented either by the correlation coefficient or square of the correlation coefficient; in this case the square was used. To correct for differences in sample concentration or thickness, a parameter is used to normalize the spectra. In this case maximum intensity was used for normalization.

Performing the calculation results in a regression curve and calculation results as shown in Fig. 2 and 3. The two PC spectra were obtained by successively analyzing the same PC sample, which resulted in a relatively high correlation (match score of 0.9955) in Fig. 2. However, though there are some similar areas between the PC and PC/PBT copolymer as shown in Fig. 3, the difference in overall spectral shape resulted in a low correlation (match score of 0.7233).

Calculating the match score allows you to numerically quantify the difference between two spectra, which is useful for tests performed to verify materials or for acceptance inspections. When analyzing unknown samples, it also allows for checking how similar an infrared spectrum is to the infrared spectrum for an item to which it is being compared.

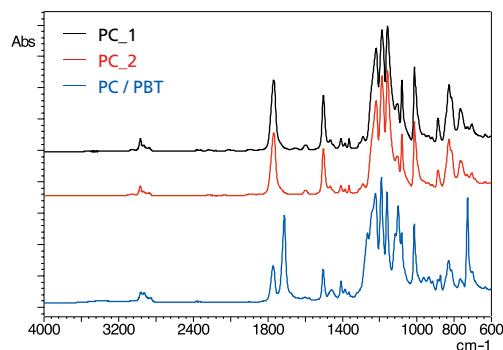
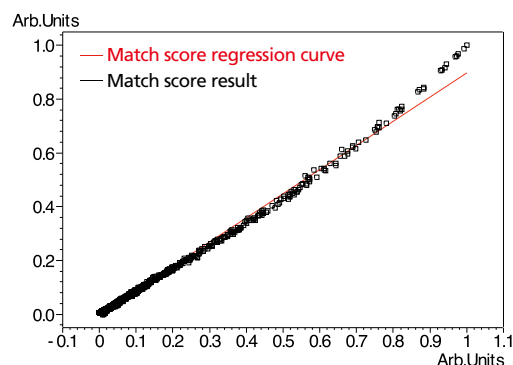
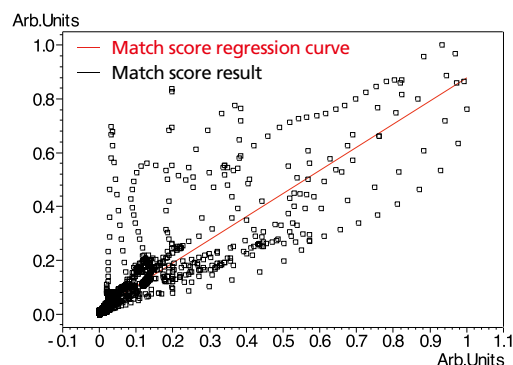


Fig. 1 Respective Infrared Spectra



Match score calculation result  
Match score (purity index): 0.9955  
Slope: 0.8979  
Intercept: -0.0006

Fig. 2 Match Score Calculation Result for PC vs. PC



Match score calculation result  
Match score (purity index): 0.7233  
Slope: 0.8606  
Intercept: 0.0182

Fig. 3 Match Score Calculation Result for PC vs. PC/PBT Copolymer

# Striving for the Ultimate in Accuracy and Easy Operability

By combining new technologies with those cultivated over Shimadzu's long history, the Shimadzu spectrofluorophotometer has been reborn as the RF-6000. Combined with new LabSolutions RF software, designed for unrivaled measurement accuracy and easy operation, the RF-6000 offers the ultimate performance for a diverse range of customers' measurement needs.

Spectrofluorophotometer

## RF-6000



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