

FTIR TALK LETTER

vol. **15**

December 2010

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Why are ripples in the baseline of the infrared spectrum?
What are interference fringes?

Fourier Transform and Apodization

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Interferograms

When analyzing a sample by a Fourier transform infrared (FTIR) spectrophotometer, the measurement mode is normally set to transmittance or absorbance. However, unlike a dispersive spectrophotometer, the FTIR instrument does not measure these spectra directly.

In FTIR analyses, Infrared light from the light source passes through

a Michelson interferometer along the optical path.

The Michelson interferometer comprises a beam splitter, moving mirror, and fixed mirror. The light beam split into two by the beam splitter is reflected from the moving mirror and fixed mirror, before being recombined by the beam splitter. As the moving mirror makes reciprocating movements, the optical path difference to the fixed mirror changes, such that the phase difference changes with time. The light beams are recombined in the Michelson interferometer to produce interference light. The intensity of the interference light is recorded in an interferogram, with the optical path difference recorded along the horizontal axis.

The data directly acquired by the FTIR instrument is in the form of an interferogram of the infrared light that passed through the sample. Looking at the interferogram does not give an understanding of the sample characteristics. To get a normal spectrum with the wavenumber (wavelength) along the horizontal axis requires Fourier transform by a computer. This is the major characteristic of the FTIR instrument and differentiates it from a dispersive spectrophotometer, which measures spectra directly.

Principle of Fourier Transform Spectrophotometry

The final data required from sample measurements has the wavenumber along the horizontal axis, as described above. A spectrum shows the light separated into its component wavelengths and the intensity plotted at each wavelength. This separation process is called "spectroscopy."

When the infrared light at wavelength λ enters the Michelson interferometer, if the optical path difference is an integer multiple, the peaks and valleys superimpose, increasing the light intensity. Conversely, when the optical path difference is a half-integer multiple (integer + 1/2), the light becomes weaker. If the original light intensity is denoted as $P(\lambda)$, the intensity $I(x)$ at the optical path difference x can be expressed as

$$I(x) = \frac{1}{2} P(\lambda) \left(1 + \cos \frac{2\pi x}{\lambda} \right)$$

In actual Fourier transform, the AC component (cos) in the above expression forms the interferogram with respect to infrared light at wavelength λ .

As the light actually emitted by the light source combines light at various wavelengths, the interferogram obtained is the sum of the expression above at various wavelengths λ . If the optical path difference is zero (0), the light is reinforced at all wavelengths, such that the interferogram exhibits high intensity. This is called "center burst."

Fourier transform is the process of calculating the wave intensity at each period from the sum at all wave periods. Applying Fourier transform to an interferogram obtains the intensity at each period, that is, at each wavelength.

If an interferogram $I(x)$ for infrared light at continuous wavenumbers can be created using the wavenumber ν instead of the wavelength λ , $I(x)$ can be expressed as

$$I(x) = \int_0^\infty S(\nu) \cos 2\pi \nu x d\nu$$

$$S(\nu) = \int_0^\infty I(x) \cos 2\pi \nu x dx$$

Where, $S(\nu)$ is the infrared light intensity at wavenumber ν . $S(\nu)$ can be calculated by Fourier transform.

The data obtained is a power spectrum. The ratio between the background and the sample power spectrum produces a spectrum expressed as transmittance.

Apodization

As described above, a transmittance spectrum (or a spectrum converted to an absorbance spectrum) is obtained when Fourier transform is applied to the measured interferogram.

However, the description above applies to a theoretical situation. Actual measurements differ from the ideal state. In particular, the integration range for the expression above is from 0 to infinity. This supports an infinite range of movement of the moving mirror. However, such a movement is impossible. The moving mirror reciprocates through a finite distance, such that in practice this integration has to be cut off in a finite range.

For example, if the integration range is restricted to $[-L, L]$, such that the contributions outside this range are not calculated, the Fourier transform expression can be written as

$$S(\nu) = \int_0^\infty W(x) I(x) \cos 2\pi \nu x dx$$

Where,

$$W(x) = 1 (|x| \leq L)$$

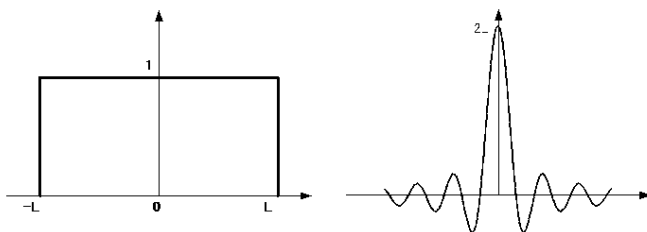
$$W(x) = 0 (|x| > L)$$

What effect does this calculation have on the spectrum $S(\nu)$ that is obtained? $S(\nu)$ incorporates images from the Fourier transform of function $W(x)$. The Fourier transform of function $W(x)$ produces

$$w(\nu) = 2L \frac{\sin 2\pi \nu L}{2\pi \nu L}$$

Ripples are formed around a large peak.

Box-car waveform $W(x)$ and $w(\nu)$



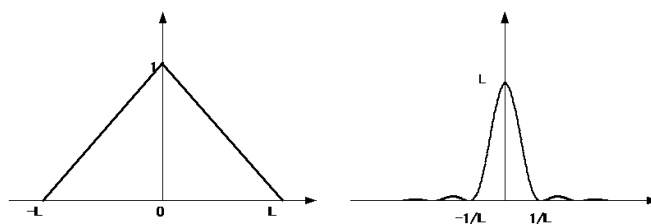
Incorporating $w(\nu)$ into spectrum $S(\nu)$ results in ripples like $W(x)$ around a peak in the spectrum. For a simple box-car waveform such as $W(x)$ above, the largest ripple after the peak is the adjacent valley, which has a depth of approximately 21 % peak height. If the ripples are large, the spectrum obtained contains irregularities analogous to peaks, and it will differ from the actual spectrum. However, if we use a triangular waveform instead of suddenly discontinuing the calculation in the integration range, we get

$$A(x) = 1 - \frac{|x|}{L} \quad (|x| \leq L)$$

If this is used instead of $W(x)$ above, the Fourier transform image $a(\nu)$ becomes

$$a(\nu) = L \left(\frac{\sin \pi \nu L}{\pi \nu L} \right)^2$$

Triangular waveform $A(x)$ and $a(\nu)$



At a glance, it is apparent that the ripples are smaller. This is because the integration is not suddenly discontinued at the limit. Rather, the contributions decrease at a constant rate as the limit is approached.

However, the central peak is also lower and somewhat wider. This appears as reduced resolution in the actual spectrum.

Applying some type of function to Fourier transform integration to reduce the ripples, as in this example, is called "apodization" and the function is known as an "apodization function." It can be seen from the examples of the box-car waveform and triangular waveform that reducing the ripples implies a compromise between the resolution and peak height. Improving one makes the other worse. Therefore, the apodization function must be selected to suit the intended purpose.

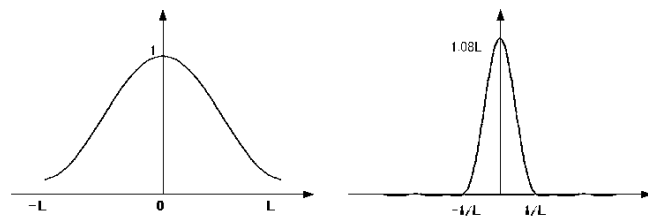
The Happ-Genzel function is often used to achieve a good balance between ripple size and resolution.

$$A(x) = 0.54 + 0.46 \cos \left(\frac{\pi x}{L} \right)$$

Applying it to the Fourier transform image gives

$$a(x) = \frac{0.54}{\pi \nu} \frac{0.46 \times 4\pi \nu L}{\pi^2 (2\pi \nu L)^2}$$

Happ-Genzel $A(x)$ and $a(\nu)$



The Fourier transform image shows a high central peak and well-suppressed ripples.

The Happ-Genzel function is normally used. However, in cases where good resolution is required, such as gas measurements, the box-car function is used.

The apodization functions must be separated according to the intended purpose, as shown above.

Appendix: Major Apodization Functions

Apodization Function ($ x \leq L$)	Fourier Transform Image	FWHM	Resolution	Peak Height	Ripple
Box-car 1	$2L \frac{\sin 2\pi\nu L}{2\pi\nu L}$	0.60/L	0.68/L	2.0L	-0.215
Triangular $1 - \frac{ x }{L}$	$L \left(\frac{\sin \pi\nu L}{\pi\nu L} \right)^2$	0.88/L	0.88/L	1.0L	+0.045
Happ-Genzel $0.54 + 0.46 \cos\left(\frac{\pi x}{L}\right)$	$\left\{ \frac{0.54}{\pi\nu} + \frac{0.46 \times 4\pi\nu L^2}{\pi^2 - (2\pi\nu L)^2} \right\} \sin(2\pi\nu L)$	0.91/L	0.89/L	1.08L	-0.006
Cosine (Cos) $\cos\left(\frac{\pi x}{2L}\right)$	$\frac{\pi L}{(\pi/2)^2 - (2\pi\nu L)^2} \cos(2\pi\nu L)$	0.82/L	0.85/L	1.27L	-0.067
Lorenz $\exp\left(-\frac{ x }{L}\right)$	$\frac{2L}{1 + (2\pi\nu L)^2} \times$ $[+e^{-1}\{2\pi\nu L \sin(2\pi\nu L) - \cos(2\pi\nu L)\}]$	0.71/L	0.73/L	1.26L	-0.055
Gaussian $\exp\left\{-\left(\frac{2.24x}{L}\right)^2\right\}$	$\frac{L\sqrt{\pi}}{2.24} \exp\left\{-\left(\frac{\pi\nu L}{2.24}\right)^2\right\}$	1.17/L	1.16/L	0.79L	-0.000

Note) The resolution applies to a 2 % dip with respect to the peak height. The ripple value expresses a proportion of the peak height.

Errata

Incorrect diagrams appeared in the article "Spectral Emissivity Measurement Using FTIR Spectrophotometry" in FTIR TALK LETTER Vol. 13.

We apologize for the inconvenience. Please replace the diagrams, as follows.

Replace Fig. 1 and Fig. 7 with the diagrams below.

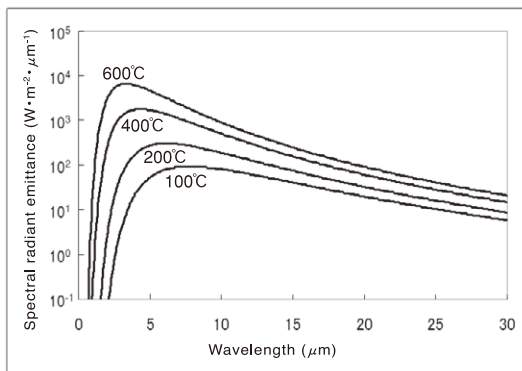


Fig. 1 Spectral Radiant Emittance of Black Body

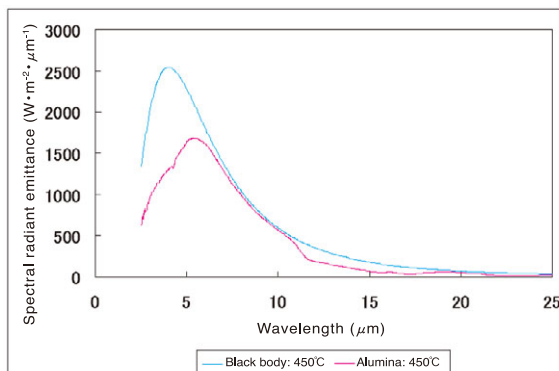


Fig. 7 Spectral Radiant Emittance at 450°C

ABC of the FTIR Measurement Methods: Liquid Membrane Method

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A Fourier transform infrared (FTIR) spectrophotometer offers a variety of measurement methods. FTIR TALK LETTER Vol. 14 introduced precautions regarding the concentration and moisture content during pellet formation for the potassium bromide (KBr) pellet method and gave actual examples of samples that cause ion exchange reactions. Here, we introduce how to select the type of cell and aperture plate for the liquid membrane method and describe precautions when determining the cell thickness for more accurate quantitative analysis.

1. Types of Cells

(1) Liquid cell

Three cell types are available for the transmission measurements of liquids: liquid cell, air-tight cell, and fixed cell. What type of cell to use is described below according to the sample characteristics and purpose of the analysis.



Fig. 1 Appearance of the Liquid Cell

The liquid cell comprises a metal cell holder, aperture plates, and spacers (metal foil such as tin or lead; or plastic such as polyethylene or fluoropolymer). It is held together by four knurled screws for easy assembly and disassembly. Put several drops of liquid sample onto one aperture plate. Sandwich it under another aperture plate, ensuring that no air bubbles are trapped. The sample forms a liquid membrane between the two aperture plates. Transmission measurements are performed to obtain an infrared spectrum of the absorption intensity corresponding to the membrane thickness. If the sample absorption intensity is low, insert spacers between the two aperture plates. As the cell can be disassembled, the aperture plates are easy to wash. This type of cell is suitable for measuring viscous samples or samples that are difficult to remove without disassembling the cell and washing. Conversely, this type of cell is unsuitable for quantitative analysis, as the cell thickness may differ from measurement to measurement if the screws are not tightened equally each time. Care is required when analyzing volatile liquid samples, as the trapped liquid membrane may gradually evaporate. However, this type of cell can be used to measure powder using the Nujol paste method.

(2) Air-tight cell

Fig. 2 shows the appearance of the air-tight cell.

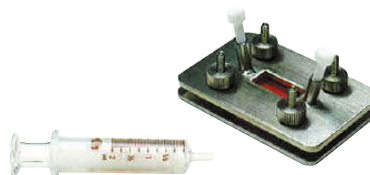


Fig. 2 Appearance of the Air-Tight Cell

After this cell is assembled, the sample can be introduced or extracted using a syringe or dropper. Because the sample is introduced after the cell is assembled, this cell is suitable for highly volatile liquid samples. In addition, since the cell remains assembled, its thickness is constant, making it suitable for quantitative analysis.

(3) Fixed cells

Fig. 3 shows the appearance of the fixed cell.



Fig. 3 Appearance of the Fixed Cell

This cell is preassembled to prevent liquid leaks. It cannot be disassembled by the analyst. Due to its constant thickness, it is suitable for quantitative analysis. However, as the cell thickness cannot be changed, cells of different thickness are required to meet different purposes. Like the air-tight cell, the sample is introduced using a syringe or dropper. Therefore, the fixed cell is suitable for highly volatile liquid samples.

2. Aperture Plates

After determining the appropriate cell for the analysis, suitable aperture plates must be selected. The types of aperture plates are described below.

Table 1 lists the characteristics of typical aperture plates used for infrared transmission measurements.

Table 1 Characteristics of Aperture Plates for Infrared Transmission Measurements

Sample Name	Transmission Wavenumber Range cm ⁻¹ (μm)	Refractive Index 1000cm ⁻¹	Aqueous Solution 20°C g/100gH ₂ O	Transmittance % (thickness)	Max. Operating Temperature (melting point)	Hardness 200 g Knoop Number
KBr Potassium bromide	40,000~340 (0.25~29.4)	1.52	65	90 (5mm)	300 (730)	7.0 (<'00)
NaCl Sodium chloride	50,000~600 (0.2~16.6)	1.49	36	90 (5mm)	400 (801)	18.2 (<100)
KCl Potassium chloride	40,000~600 (0.25~20.0)	1.46	34	90 (10mm)	(776)	9.3 (<'00)
CsI Cesium iodide	33,000~200 (0.3~50.0)	1.74	44 (0°C)	90 (5mm)	200 (621)	—
KRS-5 (TlBr + TlI) Thallium bromide + Thallium iodide	16,600~250 (0.6~40.0)	2.37	0.05	70 (2mm)	200 (414)	40.2
ZnS Zinc sulfide	10,000~725 (1.0~13.3)	2.2	Insoluble	70 (1mm)	300 (Sublimates at 1180)	35.4
ZnSe ^{Note 1)} Zinc selenide	10,000~550 (1.0~18.1)	2.4	Insoluble	65 (1mm)	350 (1700)	25.0
BaF ₂ ^{Note 2)} Barium fluoride	50,000~770 (0.2~12.9)	1.42	0.004	80 (1mm)	500 (1280)	82 (500g)
CaF ₂ Calcium fluoride	50,000~1,100 (0.2~9.0)	1.38 (2000cm ⁻¹)	Insoluble	95 (3.6mm)	900 (1402)	158 (500g)
Si Silicon	8,000~560 (1.25~15.1)	3.4	Insoluble	55 (2.5mm)	300 (1420)	1150
Ge Germanium	5,500~660 (1.8~16.6)	4.0	Insoluble	50 (2mm)	270 (938)	24
Diamond type II	40,000~125 (0.35~800)	2.38	Insoluble	70 (1mm)	(400)	10 Morse hardness
SiO ₂ (Fused Silica) Fused silica (crystal)	50,000~2,500 (0.20~40)	1.42 (3000cm ⁻¹)	Insoluble	85 (1mm)	1710 Softens	500

Note 1) ZnSe is a prescribed toxic substance (selenium compound) under the Poisonous and Deleterious Substances Control Law. Handle, trade, and dispose of this substance according to the law.
Toxic hydrogen selenide may be generated if strongly acidic samples are measured using the ZnSe prism.
Samples can be measured between pH 5 and 9.

Note 2) BaF₂ is a prescribed toxic substance (barium compound) under the Poisonous and Deleterious Substances Control Law. Handle, trade, and dispose of this substance according to the law.

While alkali halide aperture plates, such as KBr or NaCl, offer the advantage of a high infrared transmittance, they are soluble in water and therefore unsuitable for the analysis of liquid samples containing water. These aperture plates may become cloudy when liquid samples are measured. (The surface can be polished if the aperture plate becomes cloudy. For details, refer to Q&A in FTIR TALK LETTER Vol. 14.) To measure a liquid sample containing water, use KRS-5 (thallium bromide/iodide crystal), BaF₂, or CaF₂ aperture plates.

The measured wavelength range must also be considered when selecting the aperture plates. As verification testing in accordance with the Japanese Pharmacopoeia requires measurement between 4000 and 400 cm⁻¹, select KBr or KRS-5 aperture plates.

3. Important Points

When Using Cloudy Aperture Plates

Using cloudy aperture plates can cause baseline fluctuations or differential noise.

Fig. 4 shows the transmission spectrum of silicone oil measured using two cloudy KBr aperture plates after background measurement.

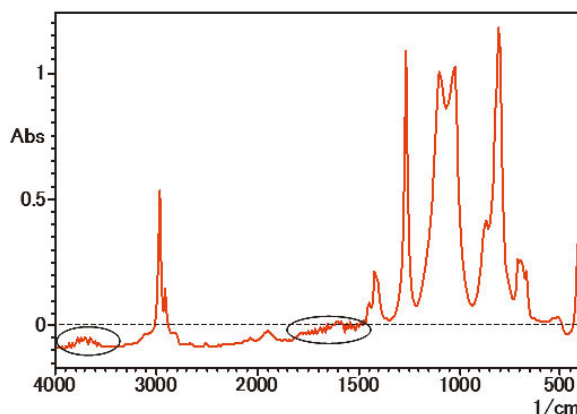


Fig. 4 Transmission Spectrum of Silicone Oil

In Fig. 4, the baseline absorbance value drops below zero and exhibits noise in the water vapor absorption regions (near 4000 to 3500 cm⁻¹ and 1800 to 1400 cm⁻¹).

If measurements are performed when the aperture plates are cloudy (fine irregularities on the aperture plate surfaces), diffuse reflection of the light occurs due to differences in the refractive index between the two aperture plates during background measurement. However, when the sample is sandwiched between the two aperture plates for sample measurement, diffuse reflection tends not to occur due to the smaller difference in the refractive index. Consequently, less light reaches the detector during the background measurement than during sample measurement, resulting in a baseline absorbance value below zero.

If water vapor exists in the measurement environment and if the difference in light intensity between the background measurement and sample measurement is large, the absorption of the water vapor is not cancelled out, resulting in differential noise in the baseline.

To avoid this phenomenon, the drop in light intensity due to the aperture plates must be minimized during the background measurement. Remedies include the following two methods:

- (1) Replace the aperture plates with non-cloudy aperture plates or polish the aperture plates;
- (2) Use only one aperture plate for the background measurement and then two aperture plates to hold the sample for analysis.

Fig. 5 shows the spectrum in Fig. 4 overlaid with the transmission spectrum of silicone oil measured between two aperture plates after measuring the background using one cloudy aperture plate. The blue line shows the results of the background measurement using only one aperture plate and the red line shows the results using two aperture plates.

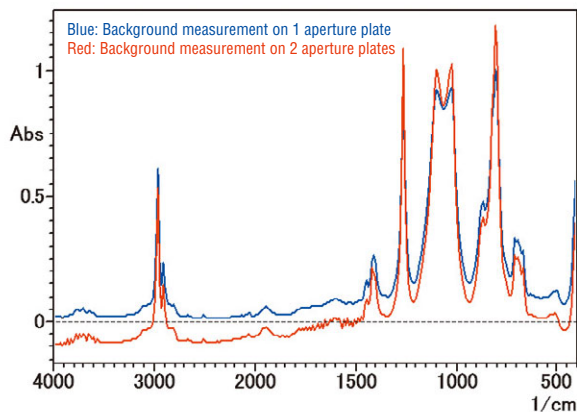


Fig. 5 Transmission Spectrum of Silicone Oil

By measuring the background with one aperture plate only, the baseline absorbance value is close to zero and the noise due to water vapor is reduced.

4. Precautions During Quantitative Analysis – Determining the Cell Thickness –

When performing quantitative analysis, the same cell is used to measure a sample of known concentration to create the calibration curve and to measure the unknown sample. As only a single cell is used, any effects due to differences in cell thickness are eliminated. However, it may be impossible to maintain a constant cell thickness if multiple fixed cells are used to efficiently analyze a number of unknown samples or if an air-tight cell must be reassembled during the measurements. Correcting for cell thickness by plotting [absorbance value/cell thickness] on the vertical axis of the calibration curve allows the same calibration curve to be used with different cells.

The cell thickness can be accurately determined by using the interference fringes that occur between the two aperture plates.

If the refractive index between the aperture plates is n (as the cell normally contains air, this value is $n = 1.0$) and the angle of incidence is θ , the cell thickness d is given by Expression (1) below.

$$d = \frac{\Delta m}{2\sqrt{n^2 - \sin^2 \theta}} \times \frac{1}{(\nu_1 - \nu_2)} \quad \text{Expression (1)}$$

Where, ν_1 and ν_2 are two wavenumbers in the interference fringe (normally selected at peaks or valleys) and Δm is the number of waves between ν_1 and ν_2 .

In practical situations, the background measurement is performed with the FTIR sample compartment empty. Next, the empty cell for which the thickness is to be determined is inserted in the sample compartment and sample measurement performed. Fig. 6 shows the interference fringes measured for a KBr fixed cell marked as 0.025 mm cell thickness.

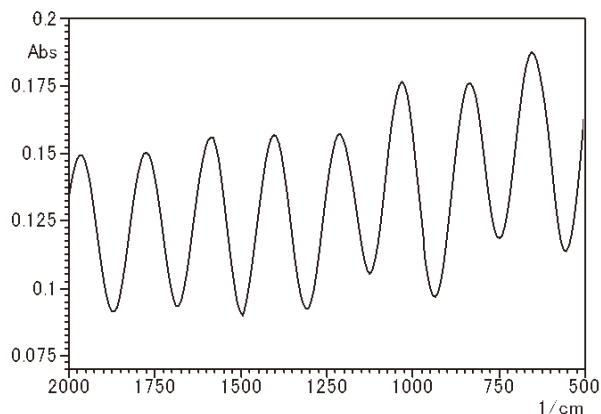


Fig. 6 Interference Fringes from a 0.025 mm KBr Fixed Cell

The thickness was calculated by selecting two appropriate wavelengths in the spectrum (2000 cm^{-1} and 1000 cm^{-1}) and substituting refractive index $n = 1.0$ and angle of incidence $\theta = 0$. The calculated cell thickness was approximately 0.027 mm, which is an error of approximately 8 % with respect to the indicated thickness.

Calculations using the interference fringes are the most commonly used for determining the cell thickness.

5. Analysis Examples

5-1 Deterioration of Mineral Oil

Mineral oils are widely used as machine oils in the automobile, steel, and other industries. There are concerns that the quality may deteriorate over long-term use due to contamination and oxidative degradation. Therefore, periodic inspection or replacement is normally required. We performed transmission measurements on used and unused machine oil in a 0.5 mm-thick fixed cell with KBr aperture plates to determine the differences between the oils.

Fig. 7 shows the two transmission spectra overlaid. The red line shows the results for the unused machine oil and the blue line shows the used oil.

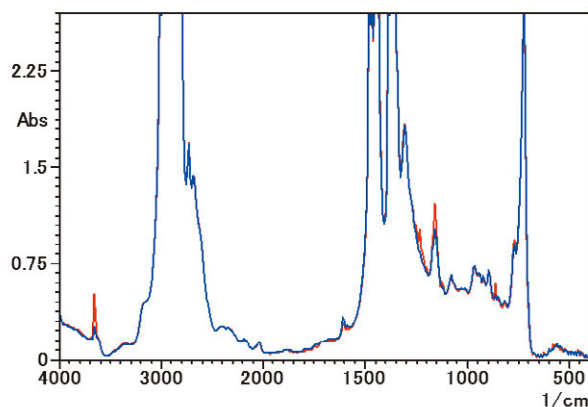


Fig. 7 Transmission Spectra of Used and Unused Machine Oil

Fig. 7 confirms differences near 3650 cm^{-1} , 1155 cm^{-1} , and 1260 to 1190 cm^{-1} . The differential spectrum was determined for the used and unused machine oil and a spectrum search performed using the Sadtler database (polymer additives) from Bio-Rad Laboratories, Inc.

Fig. 8 shows the results. The green line is the differential spectrum. The red line is the infrared spectrum for a phenolic antioxidant produced by the search.

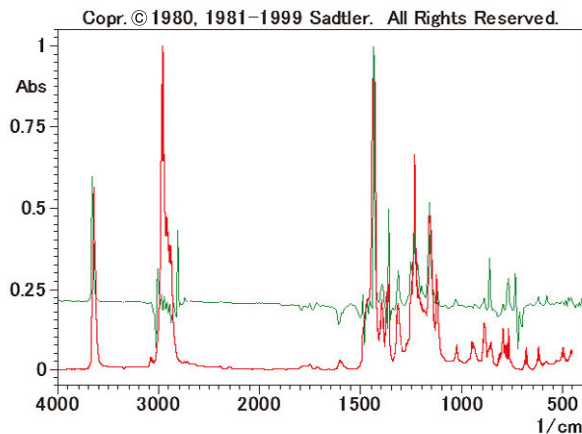


Fig. 8 Search Results for the Differential Spectrum

5-2 Quantitative Analysis of Biodiesel

Biodiesel is produced by methyl esterification of a vegetable oil to remove the glycerin, and then converting the oil to fatty acid methyl esters (FAME). European standards (BS EN 14214: 2003)¹⁾ prescribe a maximum limit of 5 % FAME mixed in automobile fuel. Japanese standards²⁾ also comply with this limit.

We performed transmission measurements on a FAME standard sample diluted to 0.1 to 0.7 % in accordance with BS EN 14078: 2003³⁾.

A 0.5 mm-thick fixed cell was used for the measurements.

A calibration curve was created using the carbonyl group peak heights in the measured infrared spectra. Fig. 9 shows the carbonyl group peaks in the FAME standard sample.

Fig. 10 shows the calibration curve. An excellent 0.9998 coefficient of correlation was obtained.

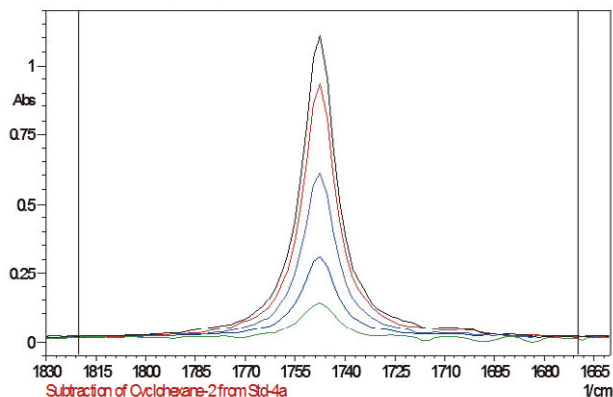


Fig. 9 Carbonyl Group Peaks in FAME Standard Sample

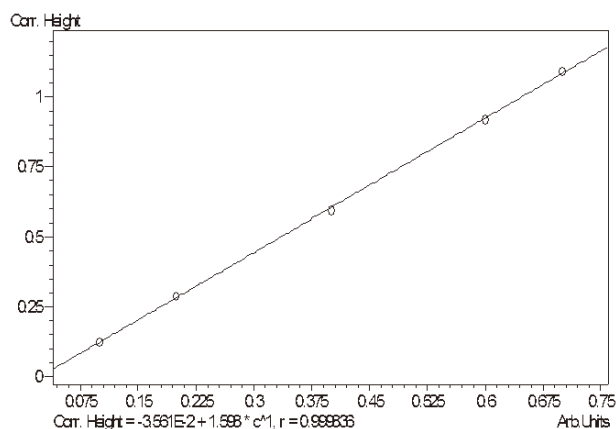


Fig. 10 Calibration Curve for FAME

6. Summary

The liquid membrane method described above is widely used for quantitative analysis, as well as qualitative analysis. However, the optimal cell type, cell thickness, and aperture plate material must be selected to suit the purpose of the measurement and the target sample.

References

- 1) Automotive fuels. Fatty acid methyl esters (FAME) for diesel engines. Requirements and test methods
- 2) Japanese Agency for Natural Resources and Energy website: Ministerial ordinance concerning partial revisions to the Act on the Quality Control of Gasoline and Other Fuels
<http://www.enecho.meti.go.jp/info/event/data/077115a.pdf>
- 3) Liquid petroleum products. Determination of fatty acid methyl esters (FAME) in middle distillates. Infrared spectroscopy method

Q&A

Question

**Why are ripples in the baseline of the infrared spectrum?
What are interference fringes?**

Answer

Sometimes the infrared spectrum baseline appears to have ripples or to be

superimposed on a regularly fluctuating wave, like a sine wave.

Fig. 1 shows the results of transmission measurements using an infrared microscope on a polypropylene (PP) fragment squashed onto a diamond cell. The baseline measured for the red frame in the photograph exhibits waves similar to sine waves.

These waves are called "interference fringes." They are caused by multiple internal reflections of the light inside the sample. As shown in Fig. 2, some of the infrared light shone onto the sample passes through the sample (A), some undergoes specular reflection from the sample front surface (B), some undergoes specular reflection from the sample rear surface (C), some undergoes double specular reflections between the front and rear surfaces (D), and some undergoes three or more specular reflections between the front and rear surfaces (E). If the sample is a plastic film, the reflected light (B) to (E) is weaker than the transmitted light (A) because the difference in refractive index at the boundary with the air is 10 % maximum. Light components (A) and (D) move toward the detector. However, due to the multiple reflections in the film, the phase of (D) differs from (A).

The mixture of these two different phases produces the interference fringes.

The interference fringes affect the peak positions and intensities. In addition, interference fringes can hinder both the quantitative analysis and the identification and qualification of unknown substances.

To prevent interference fringes, avoid positions on the sample surface that appear relatively smooth, as shown in Fig. 1. Perform the measurements at rough positions on the sample. Interference fringes can also be prevented by sample pretreatment. Actual examples of this are introduced in "Pretreatment Method for Eliminating Interference Fringes" in Application News A350.

Note, however, that interference fringes can be used to measure the sample's thickness. For details, see ABC of the FTIR Measurement Methods above.

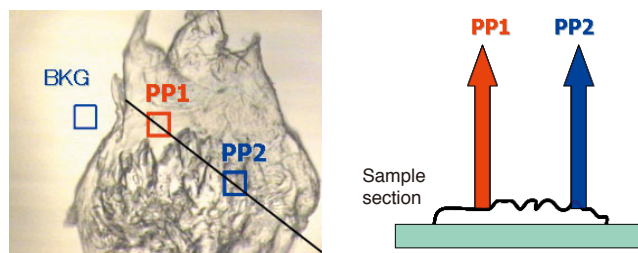
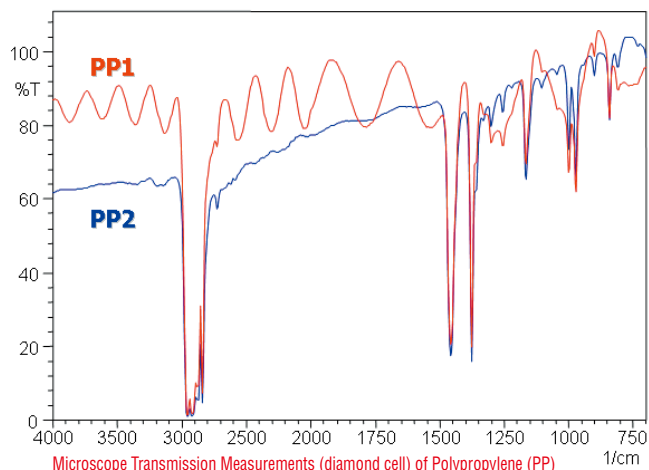


Fig. 1 Transmission Spectrum of Polypropylene (PP)
Appearance of Interference Fringes Due to Measured Position

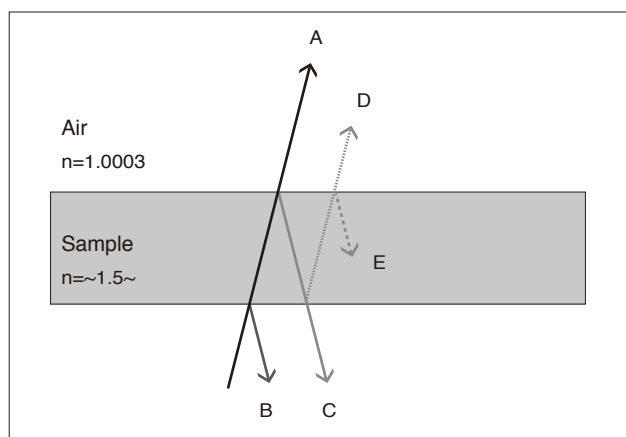


Fig. 2 Overview of Transmission Method

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