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Printed papers with patterns of the beauties of nature and geometry handed down from ancestors since the Edo era in Japan.

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Spectral Emissivity Measurement in the Infrared Region

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While fossil fuels have been instrumental as energy sources, many of them have been released into the atmosphere as heat without being used effectively, causing global warming. In order to efficiently use thermal energy that is not effectively utilized, research and development has been carried out on thermal insulation and heat radiation materials, devices for converting thermal energy, etc. In addition, there have been numerous attempts to improve performance by controlling spectral characteristics.

At the same time, multiple instruments have been developed to measure the heat emitted from various substances. These include a contact thermometer using a thermocouple or a temperature-measuring resistor, a radiation thermometer capable of measuring the temperature of distant substances in a non-contact manner, and temperature thermographic cameras that can visualize the degree distribution, many of these instruments are now being used in the medical and security fields, as well as in product development and defect inspection. Emissivity is an important parameter to accurately indicate the temperature of the object to be measured in instruments that handle such heat.

1. Terms relating to infrared radiation

The following terms are related to radiation measurement in the infrared region.

Black body: A complete radiator that has the maximum radiation energy is called a black body. Since the black body does not actually exist, the black body furnace and black body paint, which emit almost the same amount of radiation as the black body, are used as reference materials. Carbon nanotubes have the emissivity closest to a black body.

Radiant exitance: The amount of infrared radiation energy emitted from a sample per hour. The signal strength of the measuring instrument is calibrated by using a black-body furnace as a reference for radiation energy.

Total emissivity: The ratio of all energy emitted from a sample to all energy emitted from a black body at the same temperature as the sample is called total emissivity.

Spectral emissivity: The ratio of the amount of radiation from the sample to the black body measured for each wavelength is called the spectral emissivity.

Integral emissivity: The average spectral emissivity in a particular wavelength range is called the integral emissivity.

The required emissivity for the measurement of radiation thermometers and thermographic cameras is set according to the emissivity table provided by the manufacturer, but the indicated emissivity is the integrated emissivity corresponding to the measurement wavelength range of the equipment.

The integral emissivity in all measurable wavelength ranges is called the total emissivity.

Hemispherical emissivity: The ratio of the radiant emittance emitted in all directions (180 degrees) from the sample surface to the radiant emittance emitted in all directions from a black body of the same temperature.

Hemispheric emissivity represents all the thermal energy emitted from the sample surface, and can be used to evaluate the transfer of thermal energy emitted from the sample.

Vertical emissivity: The ratio of the emissivity emitted perpendicular (normal) from the sample surface to the emissivity emitted perpendicular from a black body at the same temperature.

Since the degree of emission does not change greatly even at a certain angle from the vertical direction, it is possible to treat it as the emissivity of the substance by measuring the vertical emissivity.

In this way, the emissivity seen from the integral range of the wavelength axis has total emissivity and integrated emissivity, and is divided into hemispherical emissivity and vertical emissivity from the direction in which they are emitted.

The total emissivity of the hemisphere is required to evaluate the total energy emitted from the sample, and the vertically integrated emissivity is required to evaluate the radiation thermometer, etc.

Black body furnace

The black body furnaces on the market have spherical and wedge-shaped structures to produce emissivity close to that of the black body (Fig. 1). This black body furnace is constructed by forming wedge-shaped and other irregularities into a flat shape. In recent years, flat black-body furnaces without cavities (void) have been sold, but the surface of these furnaces is covered with a fine wedge-shaped structure, and many conventional conical black-body furnaces have been installed.

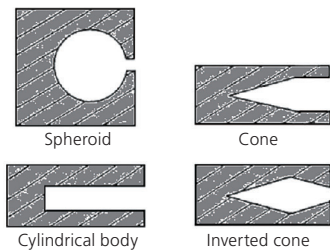


Fig. 1 Structures of various black body furnaces

Emissivity table

Emissivity tables generally published are obtained by taking the average value of the emissivity in a specific wavelength range from the measurement of a planar emission spectrum.

Since the radiation spectrum varies with the temperature and surface conditions of the material, some detailed emissivity tables describe the surface conditions and temperature of the material.

Table 1 shows the integral emissivity of common materials.

Table 1. Emissivity of common materials

Materials	(Integral) emissivity
Common metals (mirror surface)	1 ~ 10%
Common metal oxides	50 ~ 90%
Ceramics	80 ~ 90%
Plastics	80 ~ 90%
Carbon	95 ~ 97%
Paper	85 ~ 90%

Ceramics, plastics, and carbon generally have a high emissivity of 80% or more. Specifically with carbon, carbon nanotubes are known to have a three-dimensional structure and a high spectral emissivity of 98 to 99% over a wide wavelength range. They are used in a variety of applications, including anti-reflection coatings for thermal detectors, black body paints, light shielding sheets, and radiation sheets for radiators.

On the other hand, gold and platinum, which have high

reflectivity, are made into fine particles. The scattered light that remains in the particles lowers the reflectivity, and is used as an anti-reflection film on the surface of the element of a thermal detector known as gold-black or platinum-black.

When measuring the temperature with a radiation thermometer, etc., the material is checked from the emissivity table provided by each manufacturer and input. However, caution is required because the measuring wavelength of the radiation thermometer detector may differ. In the field of infrared thermography cameras, models for specific applications, such as flame measurement models and glass surface measurement models, are also available. The measurement of wavelength is also tailored to the application.

2. Factors that change emissivity

Although the emissivity table used as a radiation thermometer or a thermographic camera parameter often indicates a specific value depending on the material, the emissivity (to be precise, integrated emissivity) is actually different. It depends not only on the material but also on the conditions, such as surface temperature and surface shape (ruggedness, oxidation, etc.).

Factors that change the integrated emissivity include the following.

A) Surface temperature

Fig. 2 shows the spectral radiance of the black body at each wavelength.

The higher the temperature of the material's surface, the greater the amount of emitted light and, in particular, the stronger the radiation intensity on the shorter wavelength side (Planck's law).

On the other hand, at low temperatures, the emitted light is dominated by the longer wavelength side. The emissivity shown in the emissivity table, for example, is expressed as the average emissivity (integral emissivity) for a specific wavelength range, so the surface temperature is different.

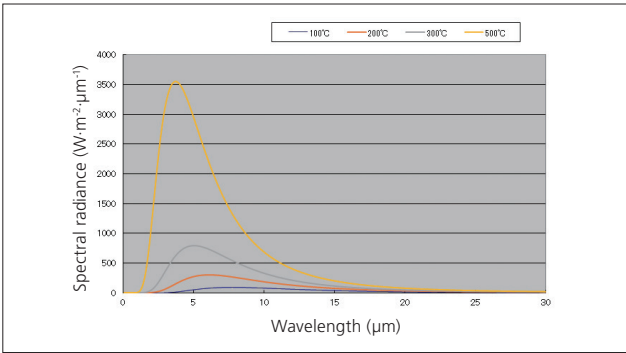


Fig. 2 Spectral radiance of black body

B) Material

If the material is flat and at a certain temperature, the emissivity is specific to that material. However, if the surface is uneven, oxidized, or reflected on a mirror surface, the emissivity is different from the actual temperature.

C) Surface condition (surface roughness)

The typical emissivity is the vertical emissivity measured by infrared light emitted perpendicularly from a planar material. However, if the surface is uneven instead of flat, the amount of emitted light increases, or the emissivity increases, at a wavelength corresponding to the degree of roughness (the depth or size of unevenness).

The spectral emissivity shows a change in the spectral characteristics in the wavelength range according to the roughness scale.

3. Emissivity measuring methods

Let's consider the equipment and methods used to measure emissivity.

A) Radiation measurement with an emissivity meter (Fig. 3)

The sample is placed in the meter, and the ratio of the amount of energy emitted from the sample warmed by the irradiated infrared ray (radiant exitance) to the radiant exitance obtained by a similar measurement of the reference black body is displayed as emissivity (hemispherical integral emissivity).

The emissivity displayed is determined by the temperature of the black body light source and the measurement wavelength range of the detector.

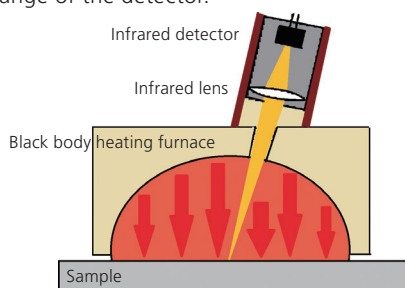


Fig. 3 Principle of the emissivity meter

[Advantages]

- Many of them are small and portable.
- Since the infrared rays are only applied to the sample and the reference black body, the measurement can be easily performed in a short time.

[Problems]

- Samples with wavelength characteristics must have emissivity measured at each temperature.
- Only the integrated emissivity in the wavelength range with the sensitivity of the instrument can be measured.

B) Emissivity measurement by heating (Figure 4)

By measuring the spectral characteristics of the infrared light emitted from the sample surface by heating the sample, the difference in the amount of radiation as determined by the wavelength of the infrared light actually emitted can be seen. For this reason, measurement of emissivity by heating can provide useful information for the development of materials that control wavelengths and for understanding the amount of radiation due to temperature changes.

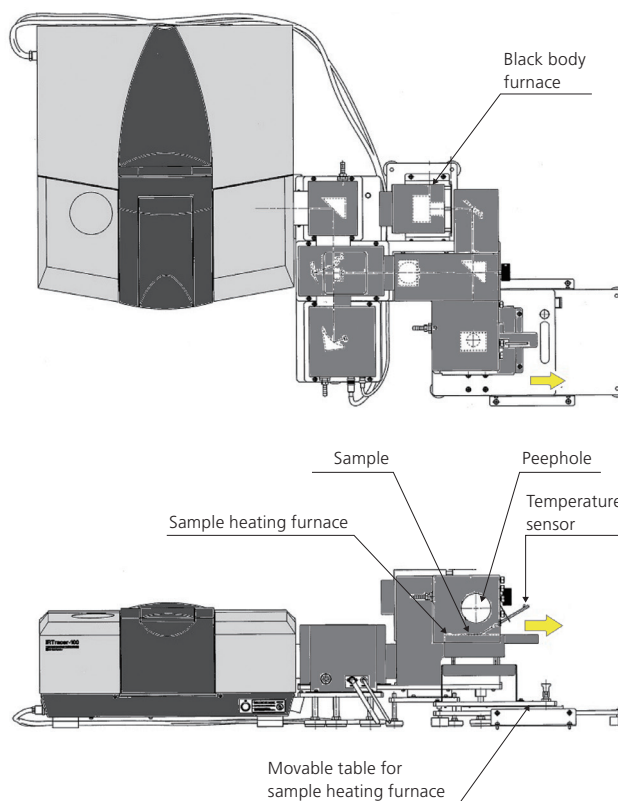


Fig. 4 Emissivity measuring device by FTIR

[Advantages]

- The wavelength dependence of the emissivity can be obtained.
- There is little error due to measurement of actual radiation from the sample.

[Problems]

- The equipment required, such as a black body furnace and a sample heating furnace, is expensive and stationary.
- To make the sample surface temperature coincide with the black body furnace temperature requires time for pretreatment, such as coating the sample with a pseudo black body paint and drying it.
- Because of the structure of the optical system, only the radiant exitance in the vertical direction can be measured.

C) A measurement that estimates emissivity by calculation from reflectance measurements
Spectral emission spectra can be estimated from reflection spectra of materials that do not transmit in the spectral region.

The following equation can be derived from the law of conservation of energy (Fig. 5).

$$(\text{Transmittance}) + (\text{Reflectance}) + (\text{Absorptance}) = 1 \quad \dots (A)$$

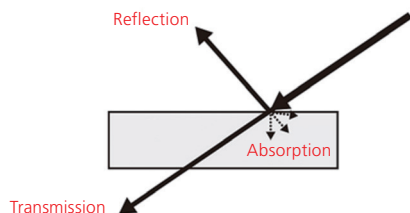


Fig. 5 Relationship between reflection, absorption, and transmission

And according to Kirchhoff's law, the absorption rate is equal to the emissivity, so the following equation holds;

$$(\text{Emissivity}) = 1 - (\text{Reflectance}) \quad [\text{where Transmittance} = 0] \quad \dots (B)$$

Therefore, you can obtain the emissivity by measuring the reflectance. Since the emitted light from a material is emitted over a wide range of angles, an integrating sphere that can measure reflections at a wide angle is generally used even if the sample is uneven (Fig. 6).



Fig. 6 Middle infrared integrating sphere accessory

(Left: Mid-IR IntegratIR™ by PIKE TECHNOLOGIES,
Right: Golden Eye III by Systems Engineering)

Structure of the integrating sphere

A middle infrared integrating sphere has an inner surface coated with gold, an infrared incidence port, a sample measurement port, and a detector exit port. Infrared rays reflected at various angles on the sample surface are multiplexed on the integrating sphere's inner surface and received by the infrared detector (Fig. 7).

The infrared spectral reflectance is measured using a gold diffuser as a reference, and the intensity ratio with the sample is plotted on the wavelength axis.

For samples with zero transmittance, the emissivity is calculated using equation (B).

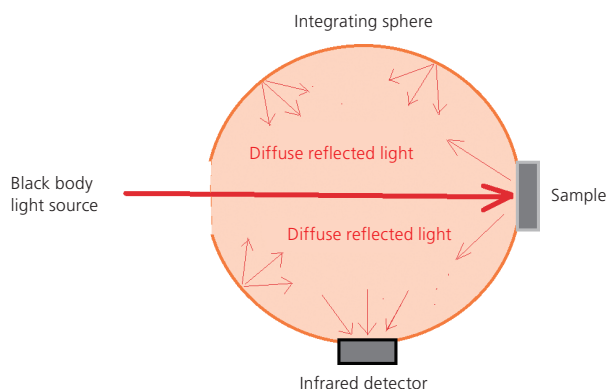


Fig. 7 Principle of the integrating sphere

When the material surface is close to the mirror surface, the reflectance varies little with the angle, so it is possible to obtain a rough spectral emissivity (vertical spectral emissivity) from the spectral reflectance measured using a specular reflection measuring device, shown in Fig. 8 and Fig. 9.



Fig. 8 Specular reflection measuring device SRM-8000

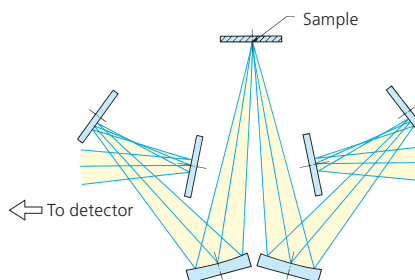


Fig. 9 Optical system of SRM-8000

[Advantages]

- The wavelength dependence of the vertical emissivity can be estimated.
- The emissivity at low temperatures can be calculated.
- Since no pretreatment is required, measurement can be performed in a short time.

[Problems]

- The integrating sphere is expensive.
- Since the reflected light emitted from the front of the sample is measured, the diffuse reflection component is removed in the case of a sample having a complicated surface structure. Therefore, the calculated emissivity may not match the actual emissivity.
- The actual emissivity may not match if infrared light is transmitted, the transmitted component is on the surface, or the sample surface has a three-dimensional structure.

4. Areas where emissivity measurements are required

There is a growing need for emissivity measurements in various research and industrial fields where materials, products, and devices are being developed.

Material development

1) Photovoltaic material

Since it is not dependent on fossil fuels, solar energy is one option for mitigating global warming. In addition, it has the added benefit of reducing costs associated with transporting fossil fuels and installing/maintaining production facilities.

A high-efficiency heat collection system is required to generate electricity by condensing light using sunlight, and a surface material with low reflectivity (high emissivity) is required for the light receiving part.

2) New material

The development of materials, structures, and paints with high thermal insulation properties is progressing in order to reduce furnace power consumption by cutting off heat from heating furnaces, and to protect living and equipment environments from high and low temperatures through thermal insulation.

In order to evaluate these materials, measurement of spectral emissivity is an important factor because the thermal insulation properties of the materials vary with temperature.

Product development

3) Miniaturization of electronic equipment

In recent years, electronic devices have become smaller and faster, thereby requiring methods to dissipate a large amount of heat generated from these devices.

In particular, due to the increased processing capacity of PCs, the processing of heat generated from ICs such as CPUs and the processing of heat generated from the light source of video projectors have become essential.

Device development

4) New device

By placing a large number of micron-order structures on the surface of a substance and increasing the emissivity of the substance with respect to a specific wavelength, it is possible to increase the sensitivity of a detector to a specific wavelength, and to emit infrared radiation specific to a wavelength as a thermal radiation device. Emissivity is a very important measurement parameter for evaluating new device development in the infrared region.

Calibration index for physical property measurement

5) Non-contact temperature monitoring parameter setting by radiation thermometer or thermographic camera

Radiation thermometers and thermographic cameras are very useful tools for measuring the temperature of a material without contact. Therefore, in recent years, as the price has fallen, thermography cameras with various purposes have become available. They are used for security, product development and evaluation, deterioration evaluation of structures, and facility evaluation in factories. However, in order to accurately determine the temperature of the object to be measured, it is necessary to set its emissivity. A simple method is to set the emissivity by referring to the emissivity table supplied by the manufacturer. However, if the substance is not in the emissivity table or its surface condition is complex, the emissivity must be measured.

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Shimadzu FTIR Memoirs

Global Application Development Center, Analytical & Measuring Instruments Division

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1. Introduction

In 1984, our company launched its first Fourier Transform Infrared (FTIR) Spectrophotometer. Over the last 36 years, more than 20,000 Shimadzu FTIR units have been sold around the world. These systems are used in a variety of fields, including academia, research institutions, chemistry, pharmaceuticals, food, and electricity. I have been involved in FTIR since its inception. In this article, I look back at the history of FTIR in our company from its birth to the present.

2. Birth of our FTIR

In early summer 1981, an engineer in his 20s who was in charge of light absorption analysis in the Application Development Division was transferred to the Spectrophotometer Development Division.

This was the starting point of Shimadzu's FTIR business. There was a university laboratory in Osaka that produced many engineers in the field of measuring instruments, such as spectrophotometers, and many of the graduates from that laboratory, including the afore-mentioned engineer, were also working in our company. Based on this relationship, we enlisted support from the laboratory for FTIR development. The following year, a graduate student who was involved in FTIR development in that laboratory joined our company, and FTIR development began in earnest with the addition of people who had been involved in other projects.

FTIR incorporates an interferometer consisting of a moving mirror, a fixed mirror, and a beam splitter. The moving mirror must be scanned smoothly at a constant speed. Air bearings were most suitable for this purpose at the time. They consist of a metal guide (support shaft) and a movable part that can be moved without contact by floating on it with compressed air. In addition, by attaching a mirror to the movable part, it can be used as an ideal moving mirror. However, since the optical system was assembled by placing an air bearing on an optical bench made of a different metal at the beginning of development, when the temperature of FTIR rose over time, the optical system was distorted. Consequently, the interference condition

worsened and the spectrum could not be measured. Come to think of it, this was simple because the two metals had different coefficients of linear expansion, but we did not notice this immediately, and it took time to solve the problem.

After overcoming many difficulties, the FTIR-4000, shown in Fig. 1, was completed in April 1984. Equipped with air bearings, the maximum resolution was 2 cm^{-1} . At that time, double-beam dispersive infrared spectrophotometers (dispersive IR) were the mainstream, so the FTIR-4000 offered a choice between double-beam and single-beam systems. In the double-beam system, the beam switching mirror installed in front of the sample chamber was switched every specified number of scan, and the light was measured alternately on the reference side and the sample side of the sample chamber. On the other hand, in the single-beam system, the light beam passed only through the sample side. Since measurements using KBr tablets and liquid cells were still the mainstream, we designed the system so that measurements could be performed with the same sense as with dispersive IR. Because of this system, the size of the optical bench containing the optical system was 1,000 mm wide x 690 mm deep x 350 mm high, and the weight was 115 kg due to the use of an aluminum die-cast optical bench, requiring 3 to 4 adults to move it. The control panel was a proprietary device with a monochrome CRT display. At that time, it was equipped with a high-performance American-made CPU (central processing unit). However, the spectrum for each scan was not displayed as it is now, and was displayed a few seconds after the specified number of scans was completed. The storage medium for the spectrum was a large 8-inch floppy disk (FD) with a capacity of 512 Kbytes. The list price was in the 9 million Japanese yen level, which was a bargain considering the 20 to 30 million yen range was common, but because the list price of dispersive IR was in the 3 million yen level, it was still expensive, and the number of units sold was only about 50.



Fig. 1 FTIR-4000

Ongoing development led to the FTIR-4100 (maximum resolution: 2 cm^{-1}) and FTIR-4300 (maximum resolution: 0.5 cm^{-1}) in accordance with improvements in data processing functionality and maximum resolution, the adoption of color for CRT displays, and the adoption of 5 inch and 3.5 inch FDs.

In addition, in 1987, we launched the FTIR-4200 (maximum resolution: 2 cm^{-1}). This was a more cost-effective model, priced in the 600 thousand yen range. The optical bench was downsized by adopting the single beam optical system. As a result, more than 200 FTIR-4200 units were sold, providing a foothold for the spread of FTIR.

3. Growth period in the 1990s

In the late 1980s, an American manufacturer launched an FTIR system with a list price of 3 million yen level. Since it has the same price as a dispersive IR, it quickly swept the domestic market. Our company lost its market share, and it was urgent to develop a counter model. An air bearing was excellent as a moving mirror, but it was expensive and required additional equipment to supply compressed air.

Therefore, it was necessary to develop a moving mirror that could reduce cost but be equivalent in performance to air bearings. In order to achieve this goal, we connected with another Shimadzu department specializing in the development of elemental technologies and completed the moving mirror using the FJS (Flexible Joint System) mechanism shown in Fig. 2. In the FJS mechanism, the mirror supporting part with the mirror was supported from the top plate by the parallel plate like a swing, and the parallel plate, top plate, and mirror supporting part were fixed by a durable special film. Since this film had no contacts other than the fixed part, the mirror supporting part was able to move straight with the same smoothness as air bearings.

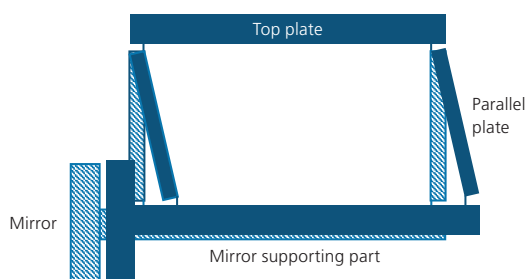


Fig. 2 Moving mirror using the FJS mechanism

Furthermore, the dynamic alignment mechanism shown in Fig. 3 was developed simultaneously with the FJS mechanism in order to enable measurement after a short warm-up time and to prevent the interference condition of the interferometer from being affected by room temperature changes.

The dynamic alignment mechanism automatically optimized the

interference condition of the interferometer at all times. A part of the He-Ne laser beam used for digitizing the infrared interfering light from the interferometer was received by detector A (as shown in Fig. 3), and the deviation from the optimum condition was calculated by the digital signal processor (DSP) based on the signal. Next, based on the calculated value, the deviation was corrected by changing the tilt of the fixed mirror with the piezo actuator in order to optimize the interference condition. The dynamic alignment mechanism was an epoch-making function that enabled measurement shortly after the power supply was turned on. Installed in every FTIR system since its development, this mechanism remains an essential component of our engineering and design.

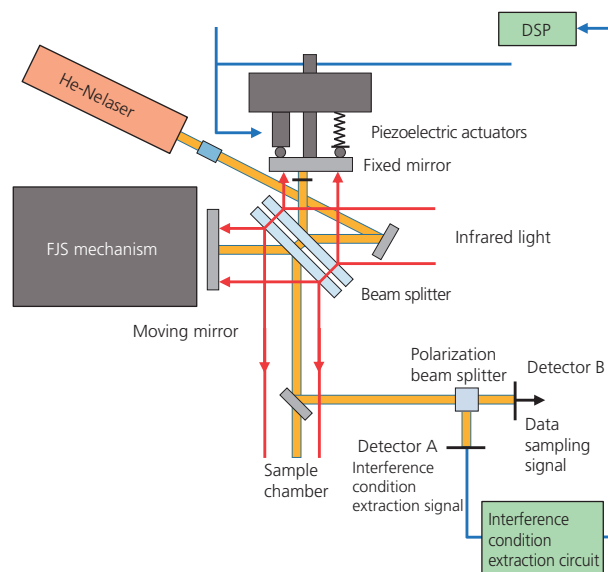


Fig. 3 Dynamic alignment mechanism

The FTIR-8100 (maximum resolution: 2 cm^{-1}), shown in Fig. 4, was launched in 1990 with a new appearance and equipped with FJS and dynamic alignment mechanisms.

By incorporating these functions, we were able to compete with the U.S. manufacturer in terms of price, allowing us to quickly recover our market share. The FTIR-8100 also had a dedicated control unit, but the function keys on the keyboard were used for spectrum measurement, spectrum screen enlargement/reduction, data processing, etc., which greatly improved operability compared to the FTIR-4000 series.



Fig. 4 FTIR-8100

Another major turning point for FTIR in the 1990s came with the spread of personal computers (PC). PCs running Microsoft's Windows® operating system began to become popular in the early 1990s. This trend impacted the world of FTIR because its control software was required to operate on Windows®. Our company needed to act quickly and we decided to work with a German software company to commercialize software. The first models of PC control were the FTIR-8200 PC (maximum resolution: 1 cm⁻¹) and FTIR-8600 PC (maximum resolution: 0.5 cm⁻¹), which were launched in 1994. The existing FTIR-8200/8600 optical bench, which used a dedicated control unit, was converted to a PC control system with a three-part interface between optical bench and the PC.

In 1997, we launched the FTIR-8300 (maximum resolution: 1 cm⁻¹) and FTIR-8700 (maximum resolution: 0.5 cm⁻¹), which were developed on the premise of PC control. Exports exceeded domestic sales for the first time that year. There were several factors behind the increase in overseas sales, one of which was thought to be the availability of PC control.

Since then, the number of exports has skyrocketed, and in fiscal 2018, overseas sales was about five times higher than domestic sales.

4. From the technological development in the 2000s to the present

In the past, our company's FTIR spectrophotometers were unable to extend the measurement wavenumber range because the beam splitter was fixed. However, in order to expand our systems' applicability to meet both internal and external demands, extension to the near infrared and far infrared regions would be required. To make this happen, the beam splitter needed to be replaced.

In replacing the beam splitter, however, the interference condition of the interferometer might deteriorate and the spectrum might become unmeasurable. To cope with this, the dynamic alignment mechanism was greatly improved, and optimization could be achieved by simply operating the software even if the beam splitter was replaced by the user. In FTIR, sensitivity is an important factor in measurement. The standard DLATGS detector was developed and manufactured by our company's Device Division, but further improvements were requested. In addition to these improvements in measurement, a dehumidifier was built into the interferometer to improve maintainability. In 2002, we launched the IRPrestige™-21, shown in Fig. 5, incorporating these technical elements. It was the first model from the 21st century, so "21" was added to the model number. The S/N ratio was 40,000:1, the highest at the time, and the control software entered its second generation.



Fig. 5 IRPrestige™-21

The IRTracer™-100 was launched in 2013 as a successor to the IRPrestige-21. The IRTracer-100 has a maximum resolution of 0.25 cm⁻¹, a S/N ratio of 60,000:1, and a rapid scanning function to measure infrared spectra scanning a movable mirror at a high speed. The S/N ratio of the FTIR-4000 was about 1,000:1 (At that time, the S/N ratio was not specified as a specification.), which means that this ratio has improved by a factor of 60 in about 30 years. Coinciding with the IRTracer-100, the control software was the third generation LabSolutions™ IR, offering improved operability and functionality.

Space saving is a trend with any type of equipment. The same is true of FTIR, and there had been a growing demand for compact FTIR systems that can more effectively utilize laboratory space and is available in a small space such as a glove box. In response, we launched the IRSpirit™ series, shown in Fig. 6, in 2017. It has a size of 390 mm wide x 250 mm deep x 210 mm high and weighs 8.5 kg. Compared to the FTIR-4000, it has a footprint of about 1/7 and a weight of about 1/14, making it portable. Despite its miniature size, it features a large sample chamber that can accommodate various accessories, and maintains the applicability of conventional FTIR. We also took input from our customers and our sales people, and made it accessible from two angles, a deviation from fixed concepts.



Fig. 6 IRSpirit™

5. Summary

Table 1 shows the model names and release years of FTIR systems released by Shimadzu from 1984 to the present. All FTIR-4000 development members have reached retirement age, and the IRSpirit was developed by engineers of the same generation as their children, clearly illustrating this 36-year timeline.

Table 1 History of Shimadzu's FTIR

Release year	Model name
1984	FTIR-4000
1986	FTIR-4100
1987	FTIR-4200, FTIR-4300
1990	FTIR-8100, FTIR-8100M
1991	FTIR-8500
1992	IRG-8000
1993	FTIR-8200, FTIR-8200D, FTIR-8600
1994	FTIR-8100A, FTIR-8200A, FTIR-8200PC, FTIR-8600PC, μ IR-8000
1997	FTIR-8300, FTIR-8700
1999	FAI-6000D
2000	FTIR-8400, FTIR-8900
2002	FTIR-8400S, IRPrestige-21
2008	IRAffinity-1
2013	IRTracer-100, IRAffinity-1S
2017	IRSpirit

Although there have been no major changes in the basic structure of FTIR since its inception, advances in elemental technology have led to improvements in FTIR system performance and functions.

It is expected that FTIR instruments incorporating new technologies will be launched in the future in order to expand the applicability of the systems and reduce the labor required for measurement.

FTIR TALK LETTER vol. 25 (Published September 2015) contains a "Reminiscence of an infrared spectrophotometer manufactured by Shimadzu Corporation from its birth to the present" recollecting the period from the birth of our company's infrared spectrophotometer in 1956 to 2015. Please check it along with this booklet.

UV-VIS Spectrophotometer

UV-i Selection

● *intelligence*

Improved Quality Control Productivity and Operators Freed from Repetitive Tasks

● *informatics*

Improved Productivity of Data Analysis Operations and Stronger Data Management

● *innovation*

Improved Administrative Productivity for High-Throughput Measurements

Three Kinds of Value Provided by Analytical Intelligence

A Reliable Partner

Can we achieve working practice reforms for spectrophotometer measurement operations? That was the question that inspired the UV-i Selection and LabSolutions UV-Vis products.



UV-1900i



UV-2600i/2700i



UV-3600i Plus



SolidSpec™-3700i

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