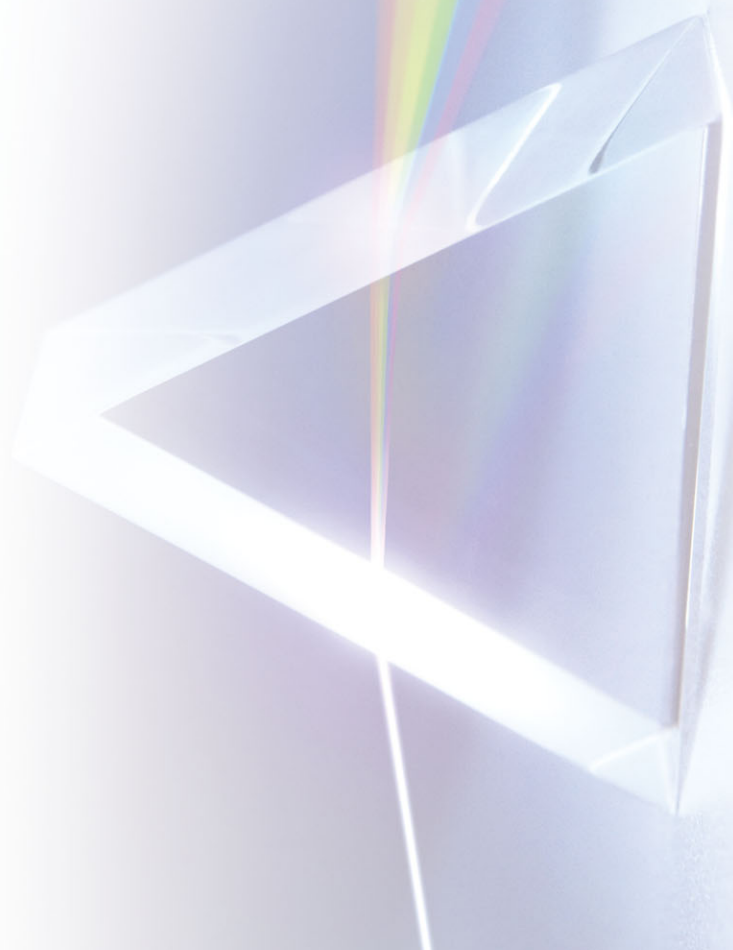Access to **your** success

⊕ SHIMADZU

UV
Talk Letter

Vol.5 *Summer 2010*

UV Talk Letter
Integrating Spheres

Integrating spheres are frequently used for measurement of solid samples using a UV-VIS spectrophotometer. These integrating spheres are introduced here.

1.Introduction

Integrating spheres have a spherical-shaped inner surface and inner wall made of light scattering material, such as barium sulfate, having high reflectance. Integrating spheres are effective in causing a light beam (measurement light) entering the sphere to scatter uniformly.

Integrating spheres have a hole (aperture) at the position irradiated by the measurement light. When measuring the measurement light which passes through the sample and enters the sphere, the sample is placed at this aperture. The detector is installed at an aperture (mainly, at the top or bottom of the integrating sphere) where it is not directly irradiated by the measurement light.

Integrating spheres are mainly used to measure samples having scattering properties or samples such as optical lenses that refract light. When measuring the transmittance of lenses that condense the measurement light after it has passed through the lens, as in the method where light is directly detected by a regular detector, the measurement light strikes the light-sensitive surface of the detector when performing baseline correction (100 % alignment) as shown in Fig. 2 (a). However, light after the sample is irradiated protrudes from the light-sensitive surface of the detector, preventing correct measurement from being

performed. With a sample having scattering properties, light that is scattered after the sample is irradiated cannot reach the light-sensitive surface of the detector. When an integrating sphere is used to measure a sample, correct measurement can be performed since all measurement lights are irradiated on the light-sensitive surface of the detector after being diffused inside the integrating sphere at both baseline correction and sample measurement, as shown in Fig. 2 (b).
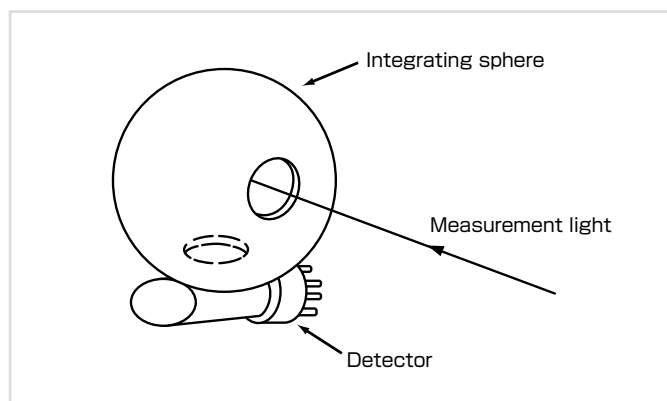

Fig.1 Schematic of Integrating Sphere

(a) When light is detected directly by the detector

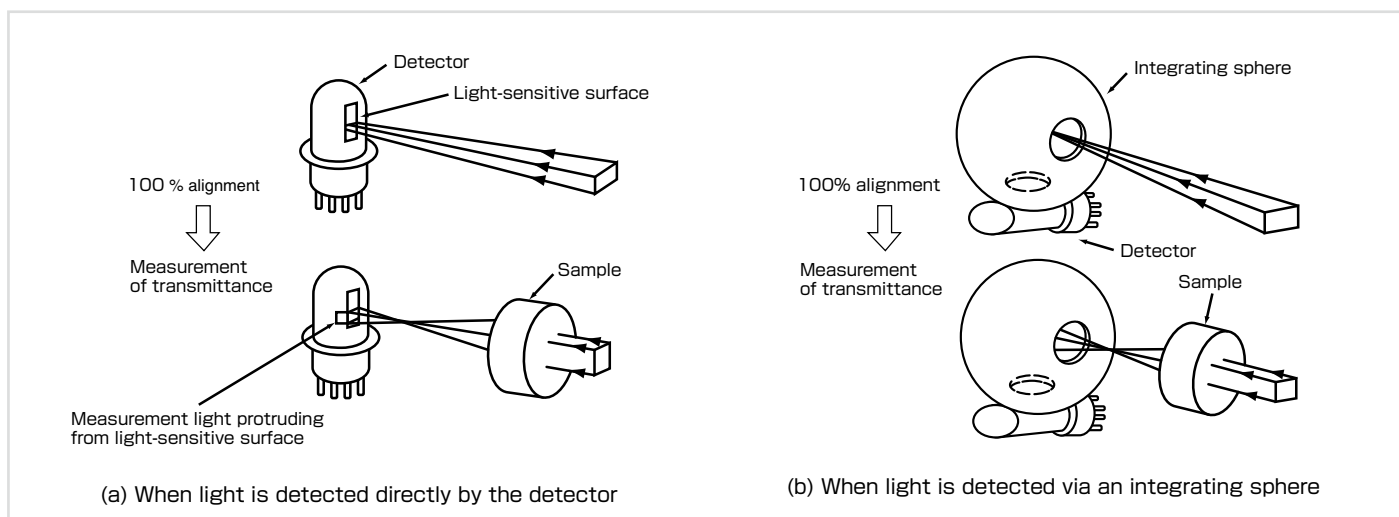(b) When light is detected via an integrating sphere

Fig. 2 Comparison of Direct Light Detection and Measurement Using Integrating Sphere

Transmittance measurement and reflectance measurement using an integrating sphere are introduced on the following pages.

1. Transmittance Measurement

As shown in Fig. 3, baseline correction is performed without a sample present at the aperture of the integrating sphere. When there is an aperture on the reflection side of the integrating sphere, a standard sample (e.g. white board filled with barium sulfate) is placed at this aperture. In regular transmittance measurement, both the scattered component and linear component that have passed through the sample are measured, as shown in Fig. 4.

To perform transmittance measurement of only the scattered component, place the sample with the aperture on the reflection side open as shown in Fig. 5.

Haze (turbidity) measurement of a suspension sample called can also be performed by transmittance measurement. Baseline correction is performed with the standard sample placed on the reflection side as shown in Fig. 3. Next, measurement is performed either with a light trap (tube for absorbing linear component light to prevent it from being introduced to the detector) installed at the reflection side, as shown in Fig. 6, or with the sample placed at the aperture and with the reflection side open. Turbidity can be obtained from these measurement values. To perform measurement more exactly, measure with the light trap installed or with the reflection side open, and then calculate correction with the resulting measurement value set as turbidity "0."

A slight difference sometimes occurs between the measurement result obtained by the integrating sphere of the spectrophotometer and the measurement value obtained by an exclusive haze meter since the irradiation state of the measurement light on the sample and the opening state, etc. of the integrating sphere are different.
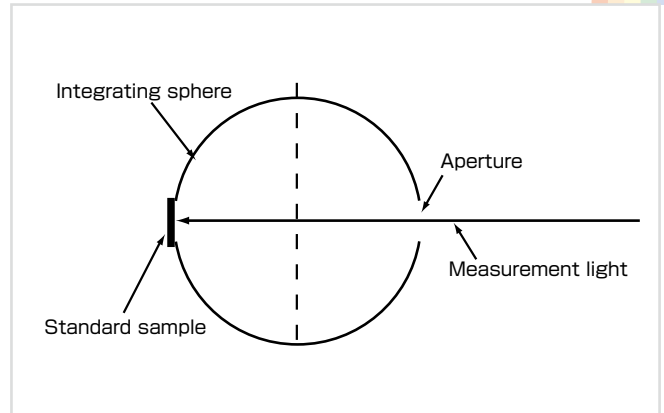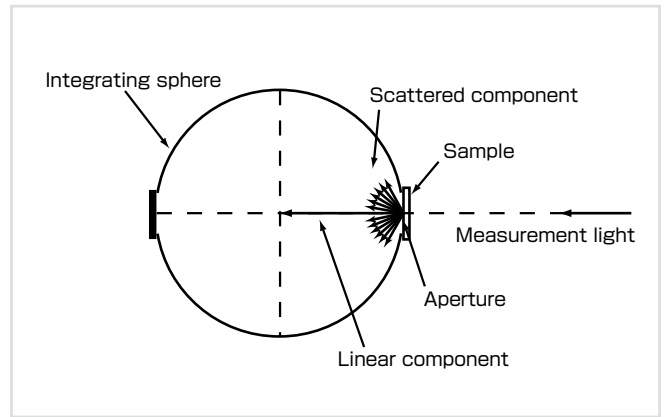

Fig. 3 Baseline Correction

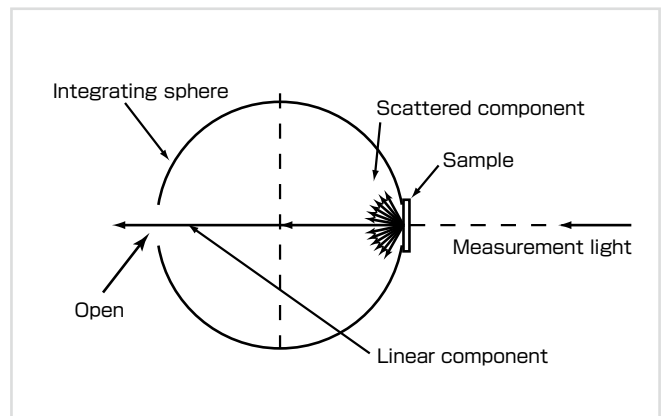Fig. 4 Schematic of Transmittance Measurement

Fig. 5 Transmittance Measurement of Only Scattered Component
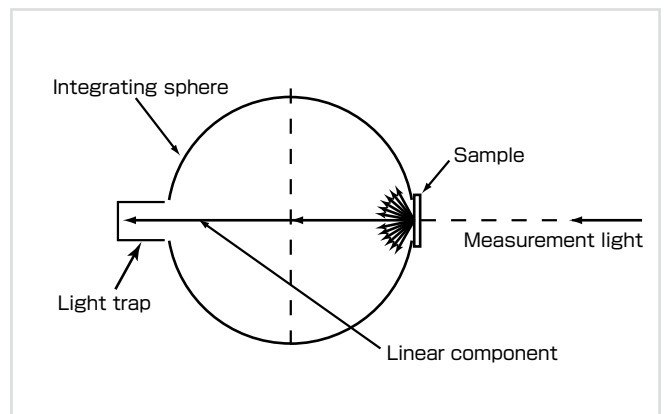

Fig. 6 Schematic of Haze Measurement

2. Reflectance Measurement

As shown in Fig. 7, after baseline correction is performed with the standard sample (e.g. white board filled with barium sulfate) placed at the position (reflection position) irradiated by the measurement light after it has entered the aperture and passed through the inside of the integrating sphere, the standard sample is replaced with the sample to be measured at the same position, and measurement is performed.

Reflectance measurement using an integrating sphere is called relative reflectance measurement since reflectance with respect to the standard sample is measured. Accordingly, changes (e.g. changes over time) in the reflectance of the standard sample affect the measurement value of the sample. Also, note that reflectance changes when the standard sample is changed.

There are two types of reflectance measurement, diffuse reflectance and total reflectance. With diffuse reflectance measurement, a normal (0°) measurement light is irradiated on the sample, as shown in Fig. 8. The diffuse reflectance component is diffused inside the integrating sphere, and the specular reflectance component exits to the outside of the integrating sphere from the hole at which the measurement light entered. For this reason, diffuse reflectance measurement involves only the diffuse reflectance component. With total reflectance measurement, however, the measurement light is irradiated tilted 8° from the normal of the sample, as shown in Fig. 9. With this measurement method, the diffuse reflectance component is diffused inside the integrating sphere, in the same way as in diffuse reflectance measurement, and the specular reflectance component is diffused inside the integrating sphere since it strikes the integrating sphere's inner wall. For this reason, total reflectance measurement involves both the specular reflectance component and the diffuse reflectance component.

With measurement using an integrating sphere, the light intensity is 1/100 to 1/1000 as compared with measuring light received directly by a detector. So, noise increases when measurement is performed at the same conditions as when light is received directly by a detector. To reduce noise, the light intensity must be increased. This can be remedied by widening the slit on a spectrophotometer.

Generally, the diameter of the inner wall of integrating spheres used in spectrophotometers is 60 mm. Some integrating spheres have an inner wall diameter of 150 mm. These have a smaller aperture ratio and higher diffusion properties, so they are less likely to be affected by the scattering state of the sample. However, due to the greater amount of space in the integrating sphere, the intensity of light incident on the detector decreases, resulting in increased noise. The aperture ratio refers to the area of the hole with respect to the entire area including the hole on the inner wall of the integrating sphere.

Fig. 7 Schematic of Reflectance Measurement
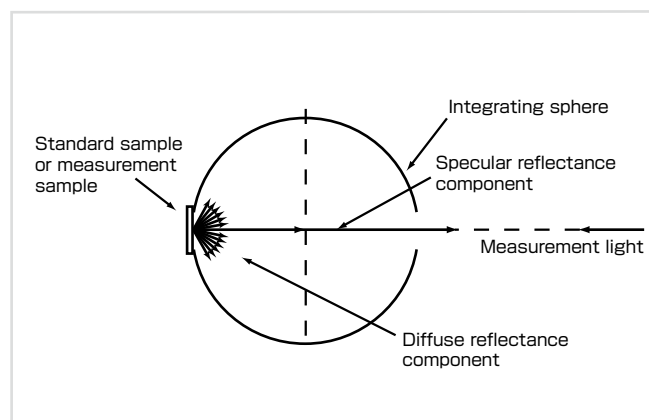
Fig. 8 Diffuse Reflectance Measurement

Fig. 9 Total Reflectance Measurement

3. Conclusion

An integrating sphere allows measuring samples that cannot be measured properly by the method where light is directly received by a regular detector. It is recommended to use an integrating sphere when measuring samples, such as semi-transparent or opaque solutions and lenses, that change the direction of light.
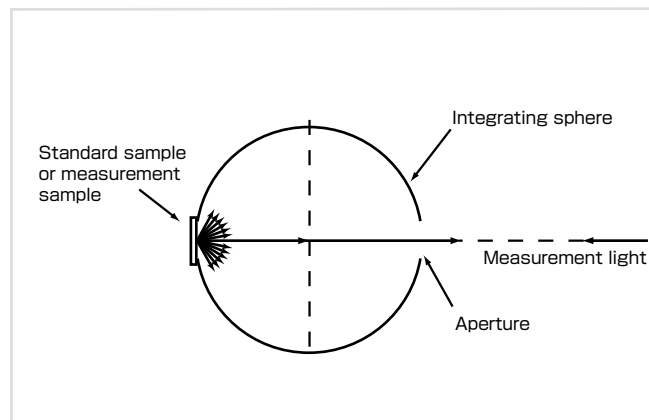
Eiji Ikeda,
Spectroscopy Business Unit, Analytical & Measuring Instruments Division
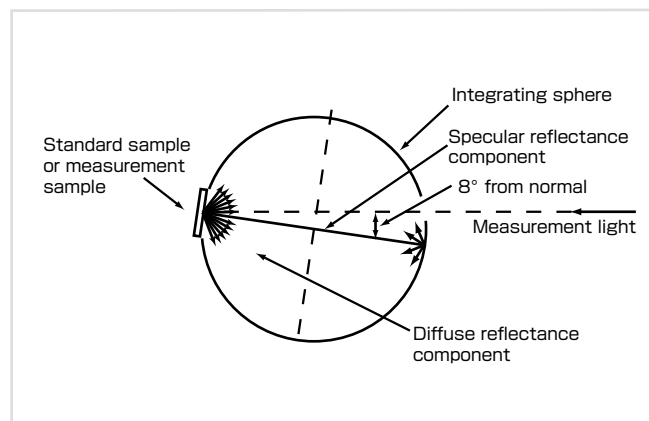
Solar Transmittance/Solar Reflectance Measurement

With global warming a major issue these days, lifestyles that place less impact on the environment are gaining in importance. For example, research to improve energy efficiency is being advanced in a variety of fields as typified by solar cells and hybrid cars. Measures for improving the efficiency of heating and cooling inside buildings are being devised. These include glazing and opaque film that allow visible light through but cut off the penetration of infrared light. Development is also is being conducted into roofing or paint for roofs that reflects infrared light. Measurement methods for evaluating the performance of these measures and calculation methods are stipulated in JIS standards, and are disclosed in JIS R3106 regarding flat glass, JIS A5759 regarding films for glazing used in construction, and JIS K5602 regarding paint films. The various measurement methods for flat glass conforming to JIS R3106 are introduced below.

1. JIS R3106 "Testing Method on Transmittance, Reflectance and Emittance of Flat Glasses and Evaluation of Solar Heat Gain Coefficient"

JIS R3106 stipulates methods for measuring and calculating visible transmittance, visible reflectance, solar transmittance, solar reflectance, and normal emittance as indices for expressing the properties of flat glass.

"Solar" in this context refers to the near ultraviolet, visible and near infrared wavelength region of 300 to 2500 nm that directly reaches Earth after penetrating the atmosphere. "Visible" refers to radiation of the wavelength range of 380 to 780 nm that is capable of passing through the visual sensory organs and causing visual sensation.

2. Visible Transmittance and Visible Reflectance

Visible transmittance (τ_v) and visible reflectance (ρ_v) refer to the ratio of the beam of visible light vertically incident on a glass surface to the incident beam of transmitted light or reflected light.

These values are calculated by equations (1) and (2) below, using spectral transmittance ($\tau(\lambda)$) and spectral reflectance ($\rho(\lambda)$), respectively, obtained by conducting transmittance measurement and reflectance measurement in the wavelength range of 380 to 780 nm using a UV-VIS spectrophotometer equipped with an integrating sphere. In the equations, $D\lambda \cdot V\lambda$ is the weight coefficient specified in JIS R3106 for calculating visible transmittance and visible reflectance.

$$\tau_v = \frac{\sum_{\lambda} D\lambda \cdot V\lambda \cdot \tau(\lambda)}{\sum_{\lambda} D\lambda \cdot V\lambda} \quad (1) \qquad \rho_v = \frac{\sum_{\lambda} D\lambda \cdot V\lambda \cdot \rho(\lambda)}{\sum_{\lambda} D\lambda \cdot V\lambda} \quad (2)$$

3. Solar Transmittance and Solar Reflectance

Solar transmittance (τ_e) and solar reflectance (ρ_e) refer to the ratio of the radiant flux of solar energy vertically incident on a glass surface to the transmitted radiant flux or reflected radiant flux.

These values are calculated by equations (3) and (4) below, using spectral transmittance ($\tau(\lambda)$) and spectral reflectance ($\rho(\lambda)$), respectively, obtained by conducting transmittance measurement and reflectance measurement in the wavelength range of 300 to 2500 nm using a UV-VIS-NIR spectrophotometer equipped with an integrating sphere. In the equations, $E\lambda \cdot \Delta\lambda$ is the weight coefficient indicating the standard spectrum distribution of solar energy as specified in JIS R3106.

$$\tau_e = \frac{\sum_{\lambda} E\lambda \cdot \Delta\lambda \cdot \tau(\lambda)}{\sum_{\lambda} E\lambda \cdot \Delta\lambda} \quad (3) \qquad \rho_e = \frac{\sum_{\lambda} E\lambda \cdot \Delta\lambda \cdot \rho(\lambda)}{\sum_{\lambda} E\lambda \cdot \Delta\lambda} \quad (4)$$

4. Normal Emittance (IR Measurement)

To determine normal emittance, specular reflectance ($\rho n(\lambda)$) measurement is conducted in the infrared region 2000 to 400 cm^{-1} using an IR spectrophotometer equipped with a specular reflectance measurement attachment. Reflectance ρn is calculated from equation (5) using the reflectance of 30 specified wavelengths among the measured values. Then, normal emittance (εn) is obtained from equation (6).

$$\rho_n = \frac{1}{30} \sum_{i=1}^{30} \rho_n(\lambda_i) \quad (5) \qquad \varepsilon_n = 1 - \rho_n \quad (6)$$

Measurement of specular reflectance ($\rho n(\lambda)$) is conducted in the range of at least 5 to 25 µm (wavenumber 2000 to 400 cm^{-1}) at a resolution of 4 cm^{-1} or less within the ambient temperature's heat emittance wavelength region of 5 to 50 µm (wavenumber 2000 to 200 cm^{-1}). If a certified surface-coated mirror is not available, the standard reflectance value specified in JIS R3106 is used. Also, if the measurement wavelength of 50 µm (200 cm^{-1}) cannot be attained, the specular reflectance value at the measured upper limit wavelength is used as the value for longer wavelengths.

5. Measurement Example

Measurements were conducted on four types of commercial plate glass to determine their respective visible transmittance, visible reflectance, solar transmittance, solar reflectance, and normal emittance values. Table 1 summarizes the measurement conditions that were used. Fig. 1 shows a glass sample placed on the integrating sphere. Figs. 2, 3 and 4 show the transmission spectra and reflection spectra in the UV-VIS-NIR region (correction by absolute reflectance of standard sample) and reflection spectra in the infrared region, respectively. The measured samples consisted of 1 type of transparent glass and three types of opaque glass (green: transparent glass, black: opaque glass 1, red: opaque glass 2, blue: opaque glass 3). It is clear from Figs. 2, 3 and 4 that large differences in both transmittance and reflectance exist in the UV-VIS-NIR region, but the reflectance values in the infrared region were about the same.

Table 2 shows the visible transmittance, visible reflectance, solar transmittance, solar reflectance, and normal emittance of each calculated sample. Calculation of the visible transmittance, visible reflectance, solar transmittance, and solar reflectance was conducted using the solar transmittance measurement software shown in Fig. 5 to easily obtain measurement results. Commercial spreadsheet software was used to calculate normal emittance.

	Visible		Solar		Normal Emittance
	Transmittance	Reflectance	Transmittance	Reflectance	
Analytical Instrument	UV-3600 UV-VIS-NIR Spectrophotometer ISR-3100 Integrating Sphere				IRAffinity-1 FTIR Spectrophotometer SRM-8000 (Specular Reflectance Measurement Attachment)
Measurement Wavelength Range	380 to 780 nm		300~2500nm		5~25 μm
Resolution	10 nm max.		Less than 300 nm: 5 nm max. 380 to 780 nm: 10 nm max. 780 nm min.: 50 nm max.		4 cm^{-1}max.
Incident Light Conditions	Close to parallel beam of light incident from the normal direction	Close to parallel beam of light incident from the radiation slit at an angle not exceeding 15°	Close to parallel beam of light incident from the normal direction	Close to parallel beam of light incident from the radiation slit at an angle not exceeding 15° Close to parallel bundle of rays incident from the normal direction	Irradiation at an angle not exceeding 15°
Standard Sample for Comparison	The air layer is used as the standard sample, and its spectral transmittance is taken to be 1	Specular reflector of reflectance specified by the absolute reflectance measurement method (sample Ⅰ), or specular reflector of reflectance specified by comparison with the sample Ⅰ	The air layer is used as the standard sample, and its spectral transmittance is taken to be 1	Specular reflector of reflectance specified by the absolute reflectance measurement method (sample Ⅰ), or specular reflector of reflectance specified by comparison with the sample Ⅰ	Aluminum-coated mirror with certified absolute reflectance (float flat glass with vacuum-deposited aluminum film)

Table 1 Measurement Conditions

	Visible		Solar		Normal Emittance
	Transmittance	Reflectance	Transmittance	Reflectance	
	τv [%]	ρv [%]	τe [%]	ρe [%]	εn [%]
Sample					
Transparent glass	91.25	4.41	89.52	5.08	87.39
Opaque glass 1	63.13	17.09	63.01	11.90	87.35
Opaque glass 2	53.79	13.88	37.86	8.57	87.44
Opaque glass 3	34.82	8.07	40.28	6.96	87.45

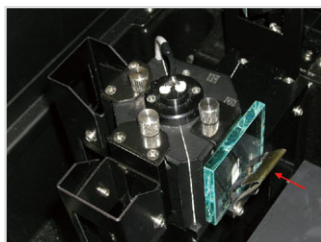Table 2 Calculation Results for Actual Samples

Fig. 1 Sample Placed on Integrating Sphere (Transmittance Measurement)
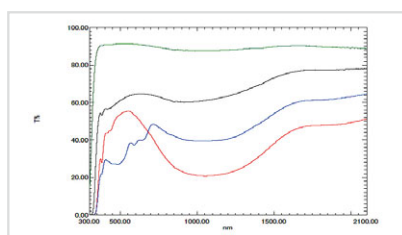

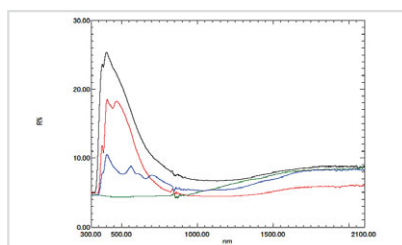Fig. 2 Transmission Spectra in UV-VIS-NIR Region

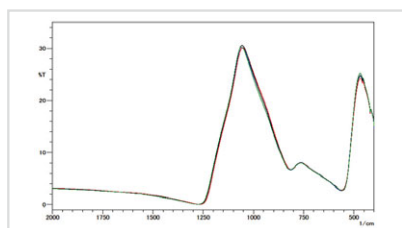Fig. 3 Reflectance Spectra in UV-VIS-NIR Region

Fig. 4 Reflectance Spectra in IR Region

Fig. 5 Solar Transmittance Measurement Software

6. Conclusion

Besides JIS R3106, which is related to flat glass, there are other solar measurement-related JIS standards: JIS A5759 regarding films for glazing used in construction and JIS K5602 regarding paint films. The explanation in this paper has dealt with JIS R3106. The next issue will deal with the other two standards. Though calculations based on JIS standards can also be performed by commercial spreadsheet software, calculation results can be obtained more easily by using dedicated software such as the Solar Transmittance Measurement Software shown in Fig. 5. Also refer to the following Shimadzu Application News for details about solar transmittance and solar reflectance.

Shimadzu Application News No. A396
"Daylight Transmittance Application Data of Glass Plate"
Shimadzu Application News No. A404
"Glass Plate Analysis in Accordance with JIS R3106"
Shimadzu Application News No. A412
"Analysis of Adhesive Films for Glazings Conducted in Accordance with JIS A5759"

Akara Hashimoto,
Kyoto Applications Development Center, Analytical Applications Department,
Analytical & Measuring Instruments Division

Q

What Is the Difference Between Relative Reflection and Absolute Reflection?

A

Relative reflectance (measurement) is used to obtain the relative reflectance referenced to a particular substance. With relative reflectance measurement, the reflectance of the sample is calculated on the assumption that the reflectance of the reflection standard is 100 %. For this reason, the obtained reflectance differs when the reflection standard is changed even if the same sample is measured. Generally, a mirror with vacuum-deposited aluminum film or barium sulfate is used as the reflection standard. Even if the same sample is measured in relative reflectance measurement, values measured on instrument A and on another instrument B will not necessarily be the same. This is because is used to obtain these instruments use different reflection standards.

On the other hand, absolute reflectance (measurement) refers to reflectance measurement for obtaining the absolute reflectance of a sample.
The reflectance referenced to an ideal reflection substance that reflects 100 % light, though this, in reality, does not exist, becomes the absolute reflectance. By adjusting the optical path at baseline correction and at sample measurement by an absolute reflectance measurement attachment, the absolute reflectance of a sample can be measured as if an ideal reflection standard has been used.
If the same sample is measured in absolute reflectance measurement, the reflectances measured on instrument A and on another instrument B match within a certain margin of measurement error.

Fig. 1 shows a comparison between the reflectances of anti-reflection film on a glass substrate measured by a specular reflectance measurement attachment (angle of incidence 5°) and by an absolute reflectance measurement attachment (angle of incidence 5°). As can be seen from the figure, the reflectance obtained by relative reflectance measurement using the reflection standard is higher than that obtained by absolute reflectance measurement.
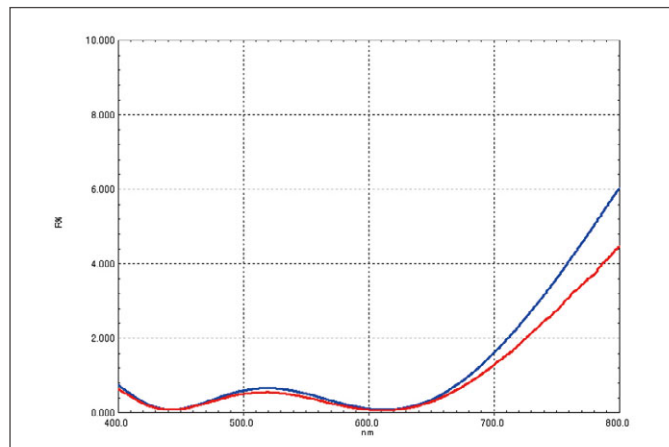

Fig. 1 Comparison Between the Relative Reflectance (——Blue Line)
and Absolute Reflectance (——Red Line) of Anti-Reflection Film

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*Survey by Shimadzu in January 2009.

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