C101-E125

SHIMADZU
Excellence in Science

UV TALK LETTER

vol. 11

UV Talk Letter

Application – Solid Sample Transmission Measurements –

A UV-VIS spectrophotometer is often used for the measurement of liquid samples but can also be applied to measure solid samples.
Several accessories are required to measure solid samples and you need to be aware of certain aspects about them.
This UV Talk Letter explains the accessories required for the measurement of solid samples and the points you need to keep in mind.

1. Introduction

Transmission measurements involve shining light on a sample and measuring the light transmitted through it. The transmitted light is generally classified as shown in Fig. 1.

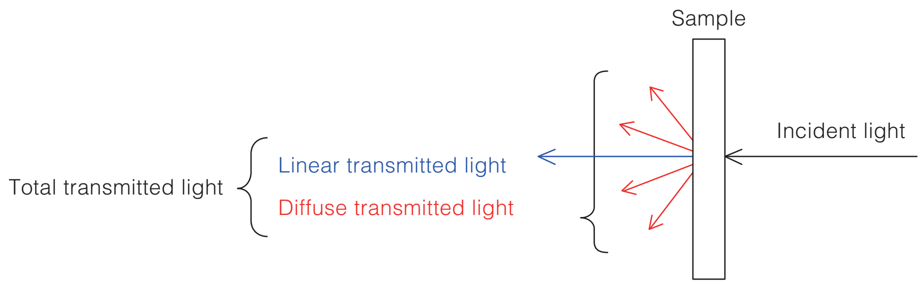


Fig. 1 Types of Transmitted Light

A UV-VIS spectrophotometer offers three types of measurements: linear transmission measurements of light that passes straight through the sample, diffuse transmission measurements of light that scattered inside the sample when passing through it, and total transmission measurements of all the light transmitted through the sample.

The characteristics of the measurements and the points to be aware of differ according to the type of transmitted light measured. These are described on the following pages, along with measured results on actual samples. We prepared a clear glass sample (glass filter) and opaque and translucent sample (opal glass). Fig. 2 shows a photograph of the measured samples.

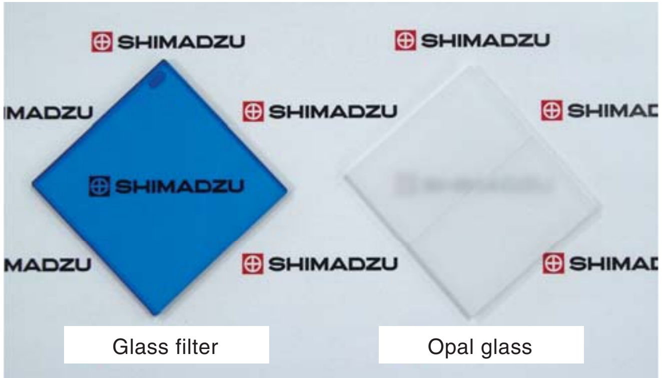


Fig. 2 Photograph of Samples

2. Linear Transmission Measurements

Linear transmission measurements measure the part of the transmitted light that passes straight through the sample. Fig. 3 shows a schematic view of the measurement. This method does not detect the diffuse transmitted light.

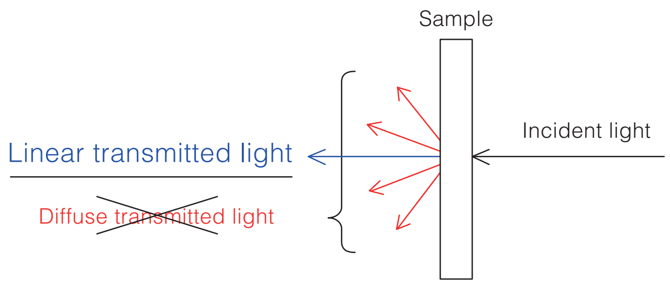


Fig. 3 Linear Transmission Measurements

This measurement method is generally used on thin transparent film or glass not exceeding approximately 3 mm thickness. It is also used to confirm almost 100 % transmittance in a sample with double-sided anti-reflection coatings applied to suppress reflection.

The film holder allows easy sample positioning. Fig. 4 shows the appearance of the film holder.

Fig. 4 Appearance of the Film Holder

During actual measurements, the film holder is installed in the standard sample compartment. Baseline correction is then performed. Baseline correction generally involves measurements on air with no sample mounted. After baseline correction is complete, mount the sample at the sample side and perform sample measurements. Fig. 5 shows the sample mounting position, viewed from directly above the film holder.

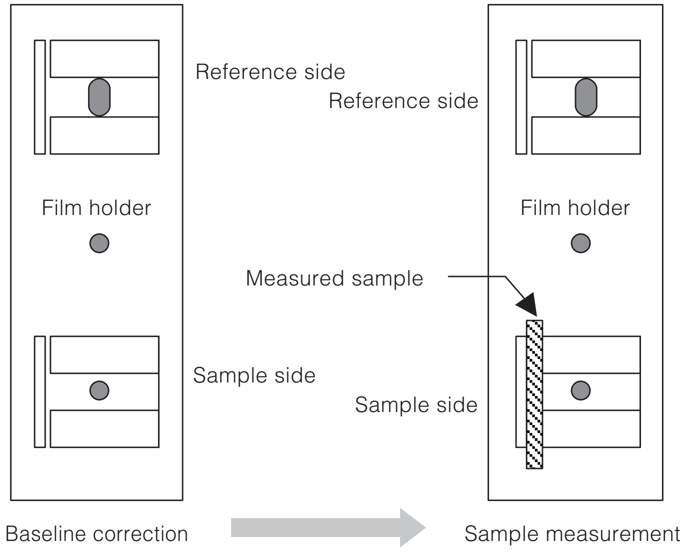


Fig. 5 Sample Mounting Position for Linear Transmission Measurements

Fig. 6 shows the measured results on the glass filter and opal glass. The transmission characteristics are clearly represented in the spectrum of a transparent sample, such as the glass filter. However, the transmittance is approximately 0 % for the opal glass, an opaque and translucent sample, due to the extremely low level of linear transmitted light.

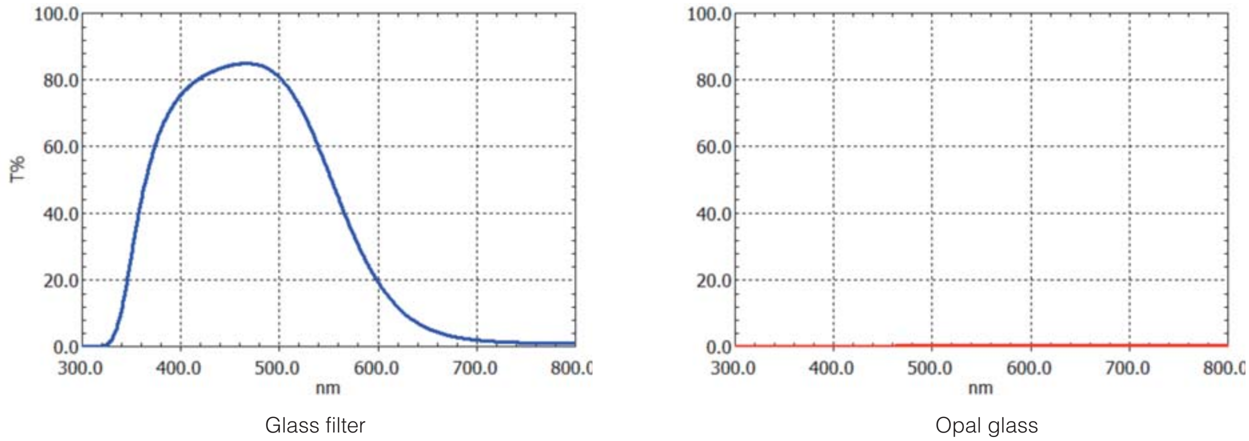


Fig. 6 Results of Linear Transmission Measurements

Linear transmission measurements are relatively simple to perform but there are several points you need to be aware of. One is the sample thickness. If the sample thickness exceeds about 3 mm, the focal point will change considerably between baseline correction and sample measurement. This results in changes in the beam size at the detector light-receiving surface, making it impossible to obtain accurate transmittance values. This change in beam size results from the difference in refractive index between air and the sample. If the transparent sample is sufficiently thin, the change in beam size will be small and not cause any problems during measurements. As samples become thicker, this effect becomes more difficult to ignore. Fig. 7 shows a schematic view of the change in beam size. Total transmission measurements using an integrating sphere, as described below, are suitable for thicker samples. It is impossible to obtain accurate transmittance values for samples, such as lenses, which are thin but the focal point changes significantly between baseline correction and measurement. Total transmission measurements are also suitable for these samples.

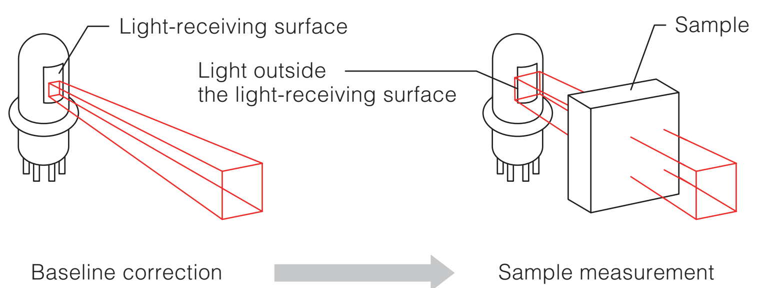


Fig. 7 Change in Beam Size When Measuring Thick Samples and Lenses

If linear transmission measurements are performed on an opaque and translucent sample, some light is scattered in the sample as diffuse light and does not reach the detector. As the distance from the sample mounting position to the detector differs according to the type of spectrophotometer, the amount of diffuse light reaching the detector differs from instrument to instrument, even when the same sample is measured. Consequently, each instrument produces different measurement results. Total transmission measurements are also suitable for such samples.

3. Total Transmission Measurements

Total transmission measurements measure all of the light passing through a sample, combining the linear transmitted light and diffuse transmitted light. Fig. 8 shows a schematic view of the measurement. A spectrophotometer fitted with an integrating sphere is used for the measurements. Therefore, such measurements are sometimes called "integrating sphere measurements." Fig. 9 shows an example of an integrating sphere attachment used for such measurements.

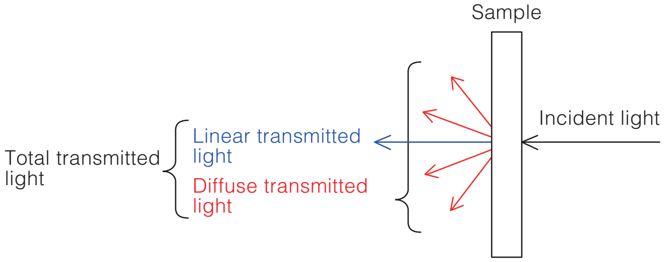


Fig. 8 Total Transmission Measurement

Total transmission measurements are often used to measure the transmittance of opaque and translucent samples. They are also used to evaluate the performance of shading film and UV protection film.

When a transparent sample is measured, total transmission measurements acquire the same data as linear transmission measurements because there is no scattering of diffuse light in the sample. If the sample is adequately thin, linear transmission measurements provide data with less noise. This is because much of the light entering the integrating sphere used for total transmission measurements does not reach the detector.

Several types of integrating spheres are available for measurements. The sizes of the integrating spheres can differ dramatically. A 60-mm-diameter integrating sphere is normally used but a large 150-mm-diameter integrating sphere attachment is also available. Integrating spheres dedicated to transmission measurements are also available with different numbers of openings. Such transmission integrating spheres are suited to the measurement of samples when the focal point differs considerably between baseline correction and sample measurement.

For total transmission measurements, care must be taken when comparing measured data. It is sometimes impossible to accurately compare data measured using different integrating spheres. This situation often occurs with samples that scatter light and create a lot of diffuse transmitted light. For baseline correction, the light incident on the integrating sphere first hits a standard white plate and makes multiple reflections inside the integrating sphere before reaching the detector. During sample measurements, however, the light first hits the interior of the integrating sphere before it reaches the detector. Consequently, differences occur in the data due to differences in reflectance at the position of the first reflection. When comparing total transmittances, you are recommended to use data measured with the same integrating sphere.


Fig. 9 ISR-2600 Plus Integrating Sphere Attachment

To measure an actual sample, install the integrating sphere in the instrument and perform baseline correction. Baseline correction generally involves measurements on air with no sample mounted. After baseline correction is complete, mount the sample and perform sample measurements. Fig. 10 shows the sample mounting position, viewed from directly above the integrating sphere.

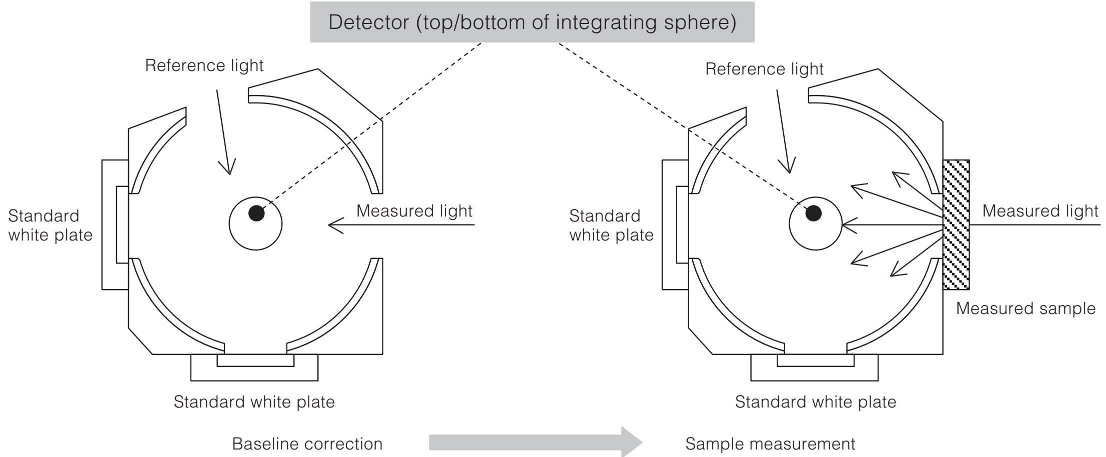
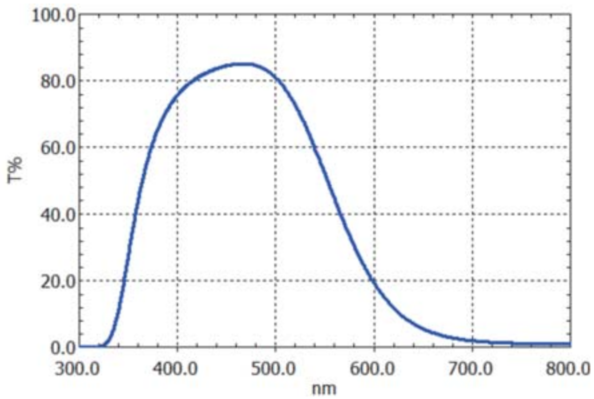
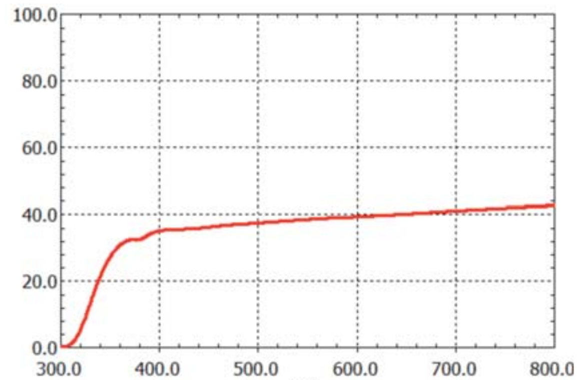


Fig. 10 Sample Mounting Position for Total Transmission Measurements

Fig. 11 shows the measured results on the glass filter and opal glass. For a transparent sample such as a glass filter, there is virtually no difference between this spectrum and the linear transmission measurement spectrum. With a translucent sample such as opal glass, on the other hand, as total transmission measurements also detect the diffuse transmitted light, a transmittance apporoximately 40% higher than the linear transmittance of apporoximately 0% is produced.



Glass filter



Opal glass

Fig. 11 Results of Total Transmission Measurements

4. Diffuse Transmission Measurements

Diffuse transmission measurements measure the part of the transmitted light that is scattered and does not pass straight through the sample. Fig. 12 shows a schematic view of the measurement. The linear transmitted light is not allowed to enter the detector during these measurements.

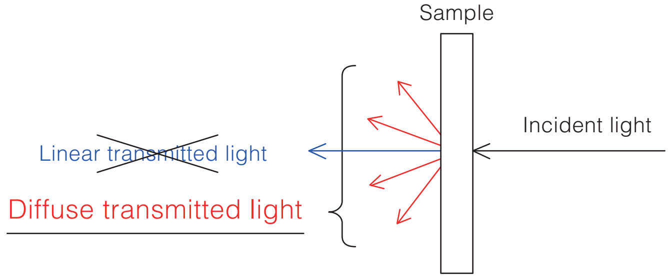


Fig. 12 Diffuse Transmission Measurements

Diffuse transmission measurements are commonly used to evaluate the scattering performance of translucent film. They are also used for haze (cloudiness) measurements. However, a special integrating sphere attachment is required to perform haze measurements according to the JIS standards, which strictly define the beam size, aperture ratio, and aperture size of the integrating sphere.

As in the case of total transmission measurements, an integrating sphere attachment is used for diffuse transmission measurements. But it is used slightly differently.

To measure an actual sample, install the integrating sphere in the instrument and perform baseline correction. At this time, mount standard white plates at the prescribed positions. Baseline correction generally involves measurements on air with no sample mounted. Next, mount the sample and perform measurements with the standard white plate on the opposite side of the integrating sphere. (In some cases a light trap is mounted at the position where the standard white plate was removed.) This allows the linear transmitted light to exit the integrating sphere, such that the integrating sphere captures only the diffuse light. Fig. 13 shows the sample mounting position, viewed from directly above the integrating sphere.

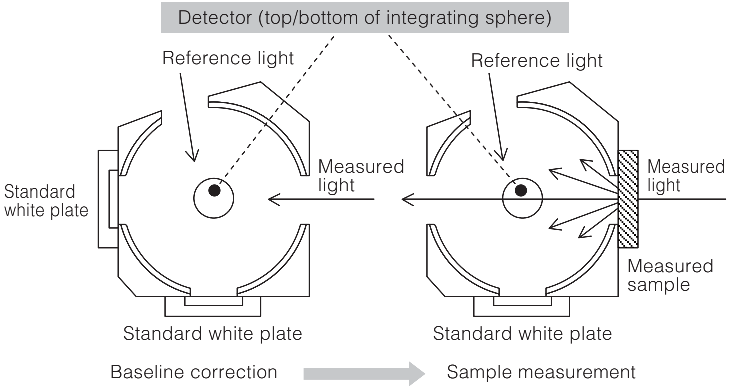
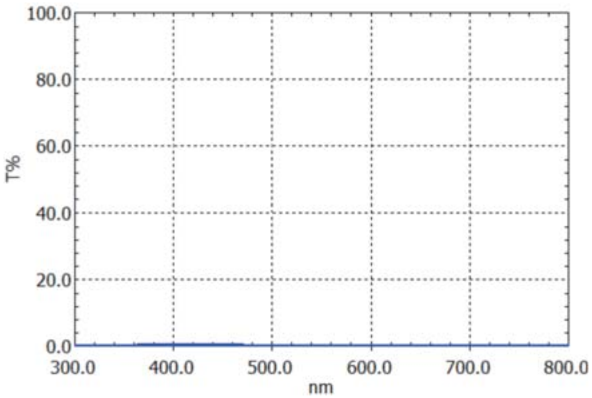
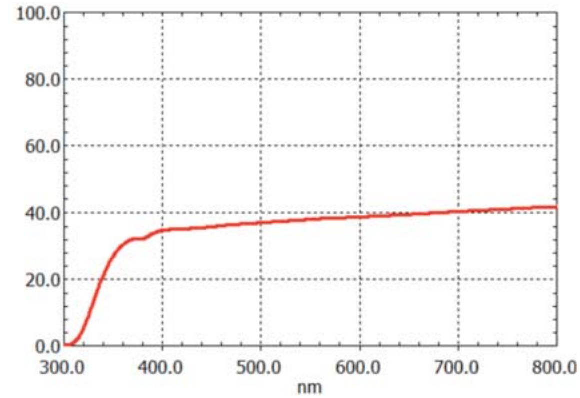


Fig. 13 Sample Mounting Position for Diffuse Transmission Measurements

Fig. 14 shows the measured results on the glass filter and opal glass. As a transparent sample such as the glass filter produces no diffuse transmitted light, the diffuse transmittance is approximately 0 %. A translucent sample such as opal glass, on the other hand, produces almost only diffuse transmitted light such that the transmittance is approximately 40 %. This does not differ significantly from the total light transmittance value.



Glass filter



Opal glass

Fig. 14 Results of Diffuse Transmission Measurements

5. Baseline Correction

For solid sample transmission measurements, baseline correction generally involves measurements on air with no sample mounted. However, to measure the transmission characteristics of only the film or coating on a substrate, before measuring the sample, perform baseline correction on a blank substrate with no film or coating applied. As it may be impossible to obtain accurate measured results at wavelengths where the blank substrate exhibits high absorbance, it is extremely important to confirm the transmission characteristics of the blank substrate.

6. Relationships Between the Types of Transmission Measurements

Subtracting the diffuse transmitted light from the total transmitted light produces the linear transmitted light. However, in actual measurements, the linear transmitted light spreads out before it is detected. Consequently, the linear transmitted light results measured in the standard sample compartment may differ from the linear transmittance calculated as the difference between the total transmitted light and diffuse transmitted light measured using an integrating sphere.

7. Summary

We described the characteristics of the three transmission measurement methods for solid samples and the important points to consider when using them. Duly considering these points achieves accurate measurement results.

Reflectance measurements, which are rarely performed on liquid samples, are commonly used on solid samples. As in the case of transmission measurements, several reflectance measurement methods are available, each with unique accessories, characteristics, and points that need to be kept in mind. These will be discussed on a future occasion.

Global Application Development Center, Analytical & Measuring Instruments Division
Hirokazu Abo

Q

Various cells are available for UV-VIS spectrophotometers, including quartz, glass, and disposable cells. How do I decide which type to use?

A

Quartz and glass cells are the most commonly used types. They differ in the wavelength ranges they can measure.

Quartz cells can be used across a wide wavelength range from the ultraviolet to the near infrared (approximately 190 nm to 2500 nm). These cells are often marked "S" and are expensive, at several tens of thousand yen each.

As glass is opaque in the ultraviolet region, glass cells offer a measurement range of approximately 320 nm to 2500 nm, which is narrower than quartz cells. At just several hundred dollars each, however, they are a convenient and cheaper option than quartz cells if measurements are not required in the short wavelength region below about 320 nm. These cells are often marked "G".

To eliminate variations in the optical path length from cell to cell, a series of measurements is performed using the same cell and rinsing the cell between measurements when glass or quartz cells are used. For details about absorption spectrum measurement procedures, see Q&A in UV TALK LETTER Vol. 3.

Disposable cells are made of plastic and can be discarded after use. Unlike the glass and quartz cells described above, a different disposable cell is used for each sample. This makes it difficult to acquire accurate data because variations in the optical path length readily occur from cell to cell. Disposable cells are used when high measurement accuracy is not required. They are useful for the measurement of biological samples where it is difficult to wash the sample out of the cell. As disposable cells are made of plastic, they are unable to measure samples that attack plastics, such as organic solvents. They are also opaque in the ultraviolet and near-infrared regions; as a result, measurements are restricted to the visible light region. These cells are often marked with an inverted triangle mark and cost just several tens of yen each.

Fig. 1 shows the transmission spectra from 190 nm to 1100 nm measured using each type of cell. It is apparent that the quartz cell allows ultraviolet light to pass through. Table 1 lists the characteristics of each type of cell. Fully understand the characteristics before using a cell.

Fig. 1

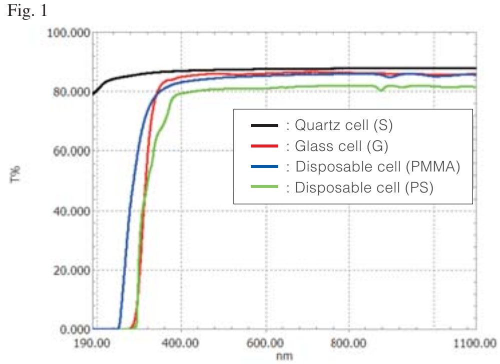


Table 1

	Quartz Cell	Glass Cell	Disposable Cell
Marking	S	G	▽
Measurable wavelength range	Approx. 190 to 2,500 nm	Approx. 320 to 2,500 nm	Approx. 340 to 900 nm (made of PS[1]) Approx. 340 to 900 nm (made of PMMA[2])
Price	Several 10 thousand yen each	Several thousand yen each	Several tens of yen each
Appearance			

1) Polystyrene resin (PS)
2) Polymethyl methacrylate resin (PMMA)

SHIMADZU

Shimadzu Corporation

www.shimadzu.com/an/