

UV

TALK LETTER

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Characteristics of Single and Double Monochromator UV-VIS Spectrophotometers — 02

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UV Talk Letter

Characteristics of Single and Double Monochromator UV-VIS Spectrophotometers

Two types of UV-VIS Spectrophotometers are available: the single monochromator type and the double monochromator type. As the names suggest, the single monochromator type contains one monochromator, while the double monochromator type contains two. But why are two types available? This UV Talk Letter explains the reasons and introduces the characteristics of each type of instrument.

1. What Is a Monochromator?

A monochromator is a mechanism that emits monochromatic light from a light source. Refer to the detailed discussions on monochromators in UV Talk Letter Vol.3. A dispersive element, generally a prism or diffraction grating, is used to create the monochromatic light. A prism splits light into a spectrum by exploiting the fact that the refractive index differs according to

the wavelength when light passes through glass. A diffraction grating has parallel grating lines ruled on the surface. The serrated grating lines cause the reflected light to diffract and split into a spectrum. Fig. 1 shows schematic diagrams of monochromators based on a prism and diffraction grating.

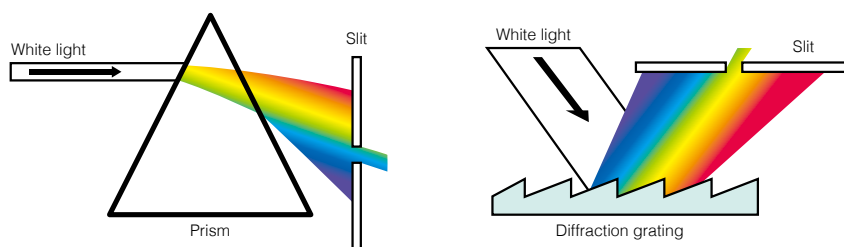


Fig. 1 Principles of Monochromators

2. Stray Light

Monochromatic light exiting the monochromator slit contains a small amount of light at wavelengths besides the target wavelength. This light is known as "stray light." If the monochromatic light shone onto the sample contains 0.01% stray light and if the measured sample has a 3 Abs peak (0.1% transmittance) at that wavelength, then the 0.01% unabsorbed stray light is added to the 0.1% transmitted light, resulting in 0.11% transmittance. This converts to a measured absorbance of 2.959, which is 0.041 lower than the actual absorbance. Fig. 2 shows an example of absorbance measurement of potassium dichromate. It is apparent that the measured absorbance is lower than the actual absorbance when the incident light includes stray light.

A double monochromator spectrophotometer with extremely low stray light is used for measurements of samples with high absorbance, as shown in Fig. 2.

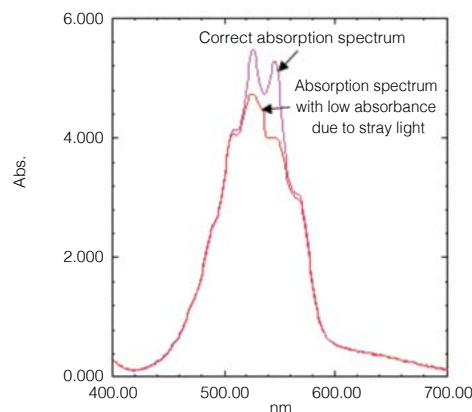


Fig. 2 Effects of Stray Light Measurement of Potassium Dichromate



3. Configuration of Single and Double Monochromator Instruments

Fig. 3 shows the difference in construction of single monochromator and double monochromator instruments.

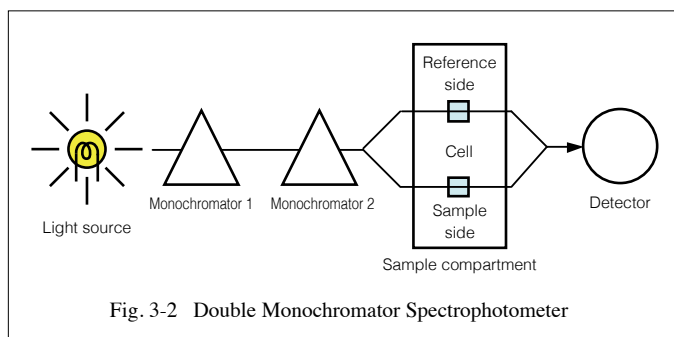
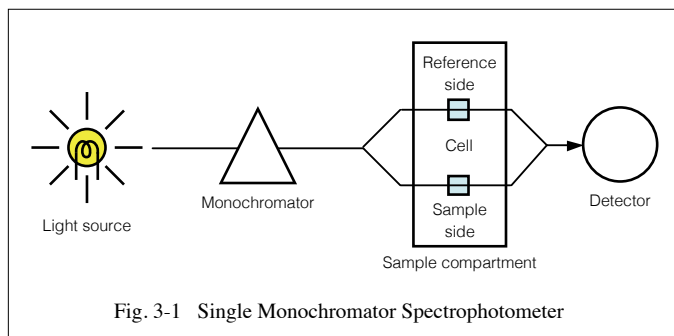


Fig. 3 Construction of Single and Double Monochromator Instruments

In contrast to a single monochromator instrument, a double monochromator type contains two monochromators in series. Light split into a spectrum by the first monochromator (Monochromator 1 in the diagram above) is split further in the second monochromator (Monochromator 2) to create purer monochromatic light with reduced stray light.

4. Optical Layout and Characteristics of Single and Double Monochromator Instruments

Fig. 4 shows optical path diagrams for Shimadzu UV-2600 and UV-2700 UV-VIS spectrophotometers.

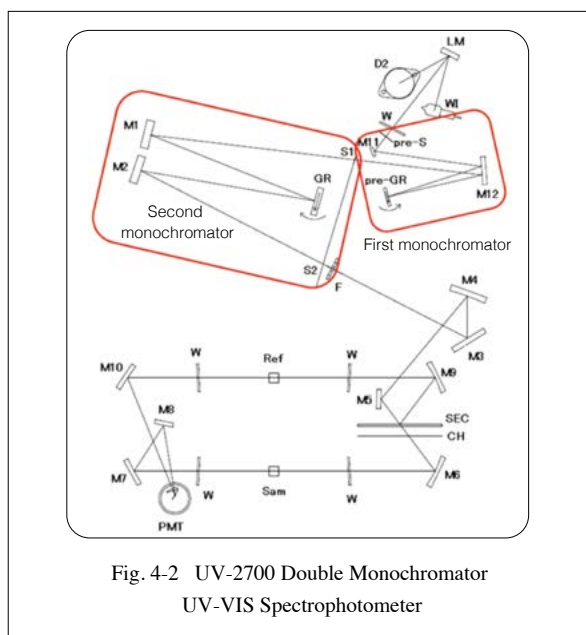
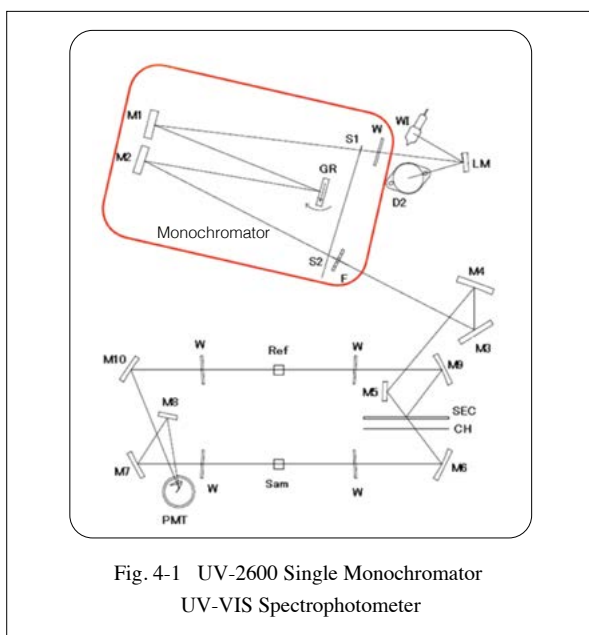


Fig. 4 Optical Path Diagrams for UV-VIS Spectrophotometers

4-1. Characteristics of Single Monochromator Spectrophotometers

The single monochromator type offers a brighter optical system than double monochromator instruments. It is suitable for measurements involving high light losses, such as spectral measurements using an integrating sphere attachment on transmissive or reflective samples that scatter light or measurements of small samples with a finely collimated light beam. Fig. 5 shows accessories for single monochromator measurements.



Fig. 5-1 ISR-2600 Integrating Sphere Attachment



Fig. 5-2 Small Sample Holder

Fig. 5 Accessories for Single Monochromator Spectrophotometers

4-2. Characteristics of Double Monochromator Spectrophotometers

The double monochromator spectrophotometer achieves high linearity by ensuring extremely low stray light in comparison to a single monochromator system. This type of instrument is suitable for measurements of high-concentration sample solutions and low-transmittance materials, such as optical filters. Fig. 6 shows the measurement of a sample with high light absorbance. It is apparent that the Shimadzu UV-2700 double monochromator UV-VIS spectrophotometer is able to measure 8 Abs absorption spectra.

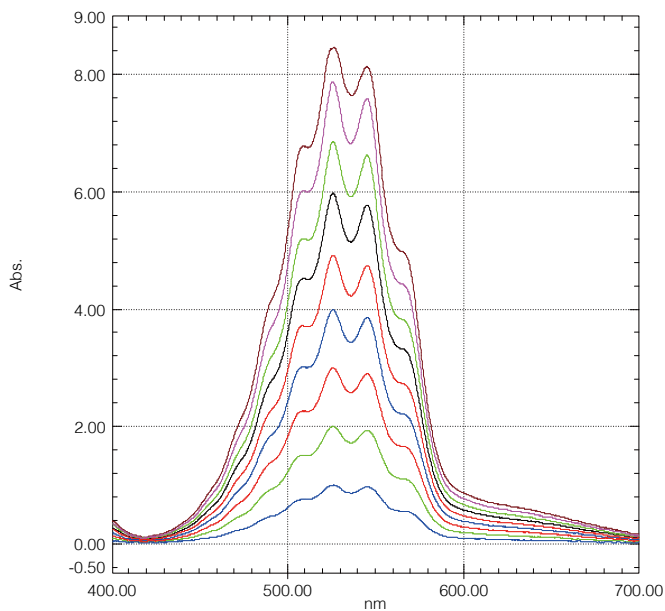


Fig. 6 Measurement of Concentrated Potassium Permanganate

5. Summary

This article discussed the characteristics of single monochromator and double monochromator spectrophotometers. To achieve highly accurate spectral measurements, it is best to fully understand each spectrophotometer's characteristics and to use the one most suitable to the sample properties and aim of the measurements.

Turbidity Measurements in Accordance with JIS K 0101 Testing Methods for Industrial Water

Turbidity is an indicator of the cloudiness of water. JIS K 0101, Testing methods for industrial water, prescribes four methods for the analysis of turbidity: (1) visual-sensation turbidity, (2) transmitted light turbidity, (3) scattered light turbidity, and (4) integrating sphere turbidity. A kaolin or formazin standard solution is used to measure turbidity. When kaolin is used, turbidity is expressed in degrees (kaolin) and when formazin is used, turbidity is expressed in degrees (formazin).

This UV Talk Letter first lists the turbidity measurements prescribed in JIS K 0101 and then introduces transmitted light turbidity measurements using a UV-VIS spectrophotometer.

1. Measuring Turbidity

Table 1 lists the four turbidity measurements prescribed in JIS K 0101. Tables 2 to 4 define how to prepare the standard solutions for transmitted light turbidity, scattered light turbidity, and integrating sphere turbidity measurements, respectively.

Table 1 Four Types of Turbidity Measurement Prescribed in JIS K 0101

	(1) Visual-Sensation Turbidity	(2) Transmitted Light Turbidity	(3) Scattered Light Turbidity	(4) Integrating Sphere Turbidity
Overview	Determined through comparison with a kaolin standard solution using the naked eye	Determined by measuring the intensity of transmitted light near the 660 nm wavelength that passed through the sample and using a calibration curve created with a kaolin or formazin standard solution.	Determined by measuring the intensity of light near the 660 nm wavelength that was scattered by particles in the sample and using a calibration curve created with a kaolin or formazin standard solution.	Determined by measuring the ratio of scattered light intensity and transmitted light intensity and using a calibration curve created with a kaolin or formazin standard solution.
Standard Solution	Kaolin	Kaolin or formazin	Kaolin or formazin	Kaolin or formazin
Measurement Range	1 to 10 degrees (kaolin)	50 mm absorption cell: 5 to 50 degrees (kaolin) or 4 to 80 degrees (formazin) 10 mm absorption cell: 25 to 250 degrees (kaolin) or 20 to 400 degrees (formazin)	1 to 5 degrees (kaolin) or 0.4 to 5 degrees (formazin)	50 mm absorption cell: 0.2 to 5 degrees (kaolin) or 0.2 to 5 degrees (formazin) 10 mm absorption cell: 5 to 100 degrees (kaolin) or 5 to 100 degrees (formazin)
Equipment and Instruments	Dark box, colorimetric tubes	Spectrophotometer or photoelectric photometer	Scattered light turbidity meter	Integrating sphere turbidity meter
Calibration Curves	Kaolin turbidity standard solutions 1 to 10 degrees (kaolin)	See Table 2.	See Table 3.	See Table 4.
Operations	Thoroughly shake colorimetric tubes containing the sample and kaolin turbidity standard solutions and put them in the dark box. View from above to select the matching standard solution.	Measure the absorbance of transmitted light near the 660 nm wavelength.	Measure the intensity of the scattered light near the 660 nm wavelength.	Measure the sample scattered light intensity T_d and the sample total transmitted light intensity T_t .
Turbidity Calculations	Calculate the sample visual turbidity using the following expression: $T = T_s \times \frac{100}{V}$ T_s = turbidity of kaolin standard solution V = sample volume (mL)	Determine the sample transmitted light turbidity from the calibration curve.	Determine the sample scattered light turbidity from the calibration curve.	Calculate the value $T_d/T_t \times 100$ and determine the integrating sphere turbidity from the calibration curve. Where, T_d = scattered light intensity T_t = sample total transmitted light intensity

Table 2 Standard Solutions for Transmitted Light Turbidity Measurements

	50 mm Absorption Cell	10 mm Absorption Cell
Kaolin Standard Solutions	100 degree (kaolin) stock solution Take 5 to 50 mL in incremental steps to prepare 5 to 50 degree (kaolin) standard solutions.	1000 degree (kaolin) stock solution Take 2.5 to 25 mL in incremental steps to prepare 25 to 250 degree (kaolin) standard solutions.
Formazin Standard Solutions	400 degree (formazin) stock solution Take 1 to 20 mL in incremental steps to prepare 4 to 80 degree (formazin) standard solutions.	400 degree (formazin) stock solution Take 5 to 100 mL in incremental steps to prepare 20 to 400 degree (formazin) standard solutions.

Table 3 Standard Solutions for Scattered Light Turbidity Measurements

Kaolin Standard Solutions	100 degree (kaolin) stock solution Take 1 to 5 mL in incremental steps to prepare 1 to 5 degree (kaolin) standard solutions.
Formazin Standard Solutions	40 degree (formazin) stock solution Take 1 to 12.5 mL in incremental steps to prepare 0.4 to 5 degree (formazin) standard solutions.

Table 4 Standard Solutions for Integrating Sphere Turbidity Measurements

	50 mm Absorption Cell	10 mm Absorption Cell
Kaolin Standard Solutions	100 degree (kaolin) stock solution Take 0.2 to 5 mL in incremental steps to prepare 0.2 to 5 degree (kaolin) standard solutions.	1000 degree (kaolin) stock solution Take 0.5 to 10 mL in incremental steps to prepare 5 to 100 degree (kaolin) standard solutions.
Formazin Standard Solutions	40 degree (formazin) stock solution Take 0.5 to 12.5 mL in incremental steps to prepare 0.2 to 5 degree (formazin) standard solutions.	400 degree (formazin) stock solution Take 1.25 to 25 mL in incremental steps to prepare 5 to 100 degree (formazin) standard solutions.

2. Analysis of Actual Samples

Transmitted light turbidity measurements (one of the four analysis techniques in Table 1) are introduced below for river water, aquarium water, whey (supernatant from yoghurt), and water left over from washing rice.

First, put 400 degree (formazin) stock solution into a 100 mL flask in 1 to 20 mL incremental steps and fill to the marked line with water to prepare 4 to 80 degree (formazin) standard solutions for the calibration curve, as shown in Table 5.

Table 5 Formazin Standard Solutions to Create Calibration Curve

Volume of 400 Degree (Formazin) Stock Solution	Turbidity of Prepared Standard Solution
1 mL	4 degrees (formazin)
2 mL	8 degrees (formazin)
5 mL	20 degrees (formazin)
10 mL	40 degrees (formazin)
20 mL	80 degrees (formazin)

Next, the formazin calibration curve standard solutions were measured with a Shimadzu UV-2600 UV-VIS spectrophotometer using the conditions shown in Table 6. A 50 mm absorption cell was used. Table 7 shows the results and Fig. 1 shows the resulting calibration curve for transmitted light turbidity measurements obtained with formazin standard solutions. The calibration curve equation is $y = 0.00776x$, with $r^2 = 0.99996$ correlation coefficient. If a 10 mm absorption cell is used, take 400 degree (formazin) stock solution in 5 to 100 mL incremental steps to prepare 20 to

400 degree (formazin) standard solutions for the calibration curve. If no commercial formazin stock solution is available, put 1.00 g hydrazinium sulfate into a 100 mL volumetric flask and fill to the marked line with water. Put 10.0 g hexamethylenetetramine into a separate 100 mL volumetric flask and fill to the marked line with water. Take 10 mL of each solution into a 200 mL volumetric flask and shake well. Leave at $25 \pm 3^\circ\text{C}$ for 24 hours and then fill the flask to the marked line with water.

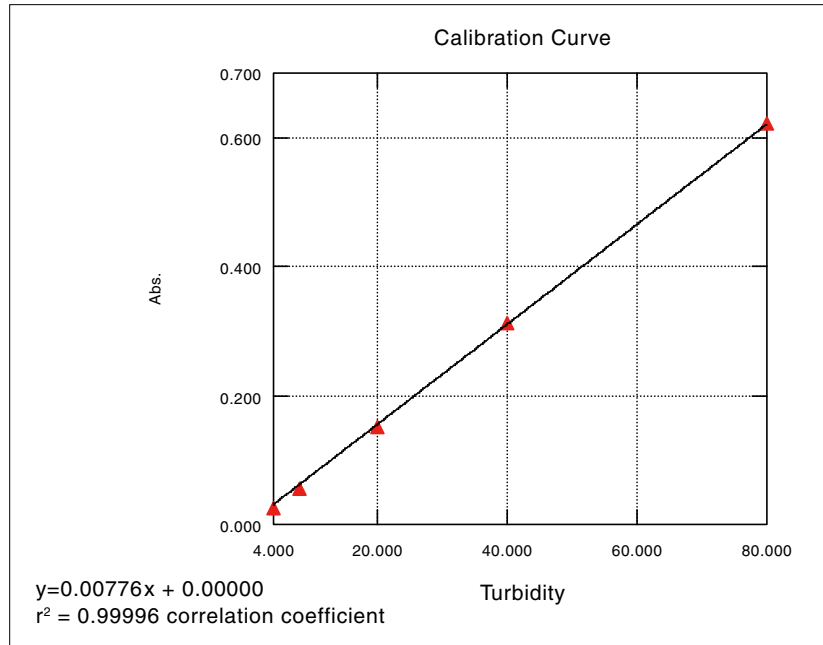


Fig. 1 Formazin Calibration Curve for Transmitted Light Turbidity Measurements

Table 6 Analysis Conditions

Instrument	: UV-2600
Measuring Mode	: Photometric-Quantitation
Measurement Wavelength	: 660 nm
Slit Width	: 2 nm
Optical Path Length	: 50 mm

Table 7 Calibration Curve Measured Results

Transmitted Light Turbidity (Formazin)	Absorbance (A)
4	0.025
8	0.056
20	0.152
40	0.311
80	0.622

This calibration curve was used to measure the turbidity of river water, aquarium water, whey (supernatant from yoghurt), and water left over from washing rice. Table 8 shows the measured results.

Table 8 Measurement Results on Actual Samples

Sample Name	Absorbance (A)	Transmitted Light Turbidity
River water	0.023	3.0 degrees (formazin)
Aquarium water	0.021	2.7 degrees (formazin)
Whey	0.640	82.4 degrees (formazin)
Water from washing rice	0.436	56.2 degrees (formazin)

3. Summary

It is apparent that the turbidity is quite high when a liquid appears cloudy to the naked eye, and that turbidity is not zero when the liquid appears quite clear. Samples must be shaken well before preparation and before measurement to ensure

that no sample remains precipitated out, as this can be difficult to see.

Refer to JIS K 0101 for more details.

Reference

JIS K 0101 Testing methods for industrial water



What is the relationship between transmittance and absorbance?



Fig. 1 shows a schematic diagram of monochromatic light passing through a sample. As the light passes through the sample, the ratio of the incident light beam intensity (I_0) and the transmitted light beam intensity (I_t) is known as the transmittance (t). This is usually expressed as a percentage (transmittance (%T)), as shown in Equation (1).

$$\text{Transmittance (t)} = \frac{I_t}{I_0} \quad \text{.....Equation (1)}$$

$$\text{Transmittance (%T)} = \frac{I_t}{I_0} \times 100$$

If the sample absorption coefficient is denoted as ϵ , the sample concentration as c , and the length (optical path length) as l , the relationship between I_0 and I_t can be expressed as shown in Equation (2). This relationship is called the Lambert-Beer Law, as described in JIS K 0212 Technical terms for analytical chemistry (optical part).

$$I_t = I_0 \times 10^{-\epsilon cl} \quad \text{.....Equation (2)}$$

On the other hand, the absorbance (A) indicates how much light has been absorbed. It is determined as the negative of the logarithm of the transmittance, as shown in Equation (3).

$$\text{Absorbance (A)} = -\log t = -\log \frac{I_t}{I_0} = \log \frac{I_0}{I_t} \quad \text{.....Equation (3)}$$

Substituting Equation (2) into Equation (3) gives:

$$\text{Absorbance (A)} = \log \frac{I_0}{I_0 \times 10^{-\epsilon cl}} = \log \frac{1}{10^{-\epsilon cl}} = \log 10^{\epsilon cl} = \epsilon cl \quad \text{.....Equation (4)}$$

Therefore, the absorbance is the product of the absorption coefficient, sample concentration, and the optical path length. Consequently, when the target substance is fixed (absorption coefficient ϵ fixed) and the same cell used for measurements (optical path length l fixed), the absorbance is proportional to the concentration (c). This relationship allows the absorbance to be used for quantitation.

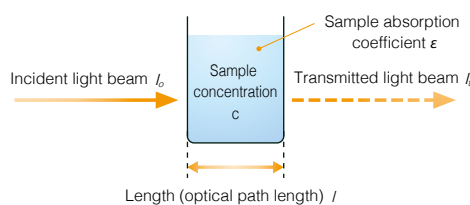


Fig. 1 Schematic Diagram of Monochromatic Light Passing Through a Sample