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NATIONAL METROLOGICAL VERIFICATION

SPECIFICATION OF THE PEOPLE'S REPUBLIC OF CHINA

JJG 178-2007

Ultraviolet, Visible, Near-Infrared Spectrophotometers

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Issued on: November 21, 2007 **Implemented on: May 21, 2008**

Issued by: General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China

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Ultraviolet, Visible, Near-Infrared Spectrophotometers

1 Scope

This regulation is applicable to the initial verification, subsequent verification and in-use inspection of visible, ultraviolet-visible, ultraviolet-visible-near-infrared spectrophotometers with wavelengths ranging from 190nm to 2600nm and with continuously adjustable wavelengths.

2 Normative References

This Regulation quoted the following references:

International Recommendation OIML R135 Edition 2004 Spectrophotometers for Medical Laboratories

JJF 1001-1998 General Terms in Metrology and their Definitions

JJF 1059-1999 Evaluation and Expression of Uncertainty in Measurement

When using this Regulation, pay attention to use the effective edition of the above quoted references.

3 Overview

Ultraviolet-visible-near-infrared spectrophotometer (hereinafter referred to as the instrument) is an instrument for the quantitative analysis and qualitative identification of the substance based on the selective absorption of radiation (light) in the ultraviolet, visible and near-infrared regions by the molecules against the substance, and the Lambert-Beer law.

The mathematical expression of the Lambert-Beer law is:

$$A = -\lg(I/I_o) = -\lg T = klc$$

Where:

A – absorbance of the substance;

transmittance valley value or absorbance peak wavelength λ_i; and measure 3 times continuously.

When selecting a mercury lamp, place the mercury lamp in the light source room so that the light of the mercury lamp is incident on the entrance slit of the monochromator. Select the energy measurement method of the instrument; set an appropriate gain; and adjust the position of the mercury lamp to maximize the energy value. Then measure the peak wavelength corresponding to the maximum energy value in one-way and point-by-point near the peak wavelength; record the λ_i; and measure 3 times continuously.

b) Automatic scanning instrument

Set the wavelength scanning range (if the wavelength scanning range is wide, segment scanning is allowed), common spectral bandwidth, slow scanning, sampling interval less than the wavelength repeatability index of the instrument (if the wavelength sampling interval cannot be set, a slower scan speed shall be selected) of the instrument according to the selected verification wavelength. When using a solution or filter standard substance, the transmittance or absorbance measurement method is used. According to the set scanning parameters, use air as a blank to perform baseline calibration of the instrument; and use a light baffle to perform dark current correction. Then place the standard substance vertically on the sample optical path; set an appropriate recording range; scan 3 times continuously; and detect (or measure) the transmittance valley value or absorbance peak wavelength λ_i, respectively.

When using a low-pressure quartz mercury lamp, scan 3 times continuously according to 6.3.2.2a) to detect (or measure) the peak wavelength λ_i of the energy, respectively.

6.3.2.3 Calculation of the result

Calculate the wavelength indication error for each measurement wavelength according to Formula (1):

$$\Delta\lambda = \bar{\lambda} - \lambda_s \qquad (1)$$

Where:

$\bar{\lambda}$ – average value of the 3 measurements;

λ_s – standard value of wavelength.

Calculate the wavelength repeatability according to Formula (2):

$$\delta_\lambda = \lambda_{max} - \lambda_{min} \qquad (2)$$

Where:

λ_{max}, λ_{min} – maximum and minimum values of the 3 measurement wavelengths, respectively.

6.3.3 Noise and Drift

According to the working waveband range of the instrument, select Section-A 250nm, Section-B 500nm, Section-C 1500nm as the measurement wavelength of noise; and 500nm as the measurement wavelength of drift.

Set the scanning parameters of the instrument as: time scanning mode (or constant wavelength scanning), spectral bandwidth 2nm (not setting for instruments with fixed spectral bandwidth), time sampling interval (or integration time) 1s, photometric measurement mode as transmittance, recording range 99%~101% (not setting for non-scanning instruments), at each measurement wavelength, both the reference beam and the sample beam are treated as air blanks, adjust the transmittance of the instrument to 100%, scan for 2 min, and measure the difference (for non-scanning instrument, record the maximum and minimum values within 2 minutes) between the maximum and minimum values on the spectrum, which is the noise with 100% instrument transmittance. Insert a light baffle in the sample optical path to adjust the transmittance of the instrument to 0%; scan for 2 min; and measure the difference (for non-scanning instrument, record the maximum and minimum values within 2 min) between the maximum and minimum values on the spectrum, which is the noise with 0% instrument transmittance.

When the wavelength is switched, allow the visible light to stabilize for 5min.

Automatic scanning instrument, after testing the noise with transmittances of 0% and 100% according to the above requirements, set the wavelength at 500nm, scan for 30min; read out the difference between the maximum value and the minimum value of the center line of the envelope of the scanning spectrum, which is the drift of the line with 100% instrument transmittance.

6.3.4 Minimum spectral bandwidth

For instruments with a deuterium lamp, select the 656.1nm characteristic spectral line of the deuterium lamp. For instruments without a deuterium lamp, choose the 546.1nm (or 253.7nm) characteristic spectral line of the mercury lamp; select the minimum spectral bandwidth; and record the characteristic spectral lines of the deuterium lamp or mercury lamp according to the method in 6.3.2.2; measure the half-peak width, which is the minimum spectral bandwidth.

6.3.5 Maximum allowable error of transmittance and repeatability

6.3.5.1 Verification steps

a) Using the standard substance and standard absorption tank in 6.1.2.1, measure the transmittance for three times at 235nm, 257nm, 313nm, and 350nm, respectively.

   It can also be measured by the ultraviolet transmittance filter in 6.1.2.2.

b) Using spectral neutral filters with nominal transmittance values of 10%, 20%, and 30%, measure the transmittance for three times at 440nm, 546nm, and 635nm, respectively, with air as a reference.

6.3.5.2 Calculation of the result

Calculate the transmittance indication error according to Formula (3):

$$\Delta T = \overline{T} - T_s \tag{3}$$

Where:

$\overline{T}$ – average value of the 3 measurements;

T_s – standard value of the transmittance.

Calculate the transmittance repeatability according to Formula (4):

$$\delta_T = T_{max} - T_{min} \tag{4}$$

Where:

T_{max}, T_{min} – maximum and minimum values of 3 measurement transmittances.

6.3.6 Baseline flatness

After baseline calibration according to the requirements of the instrument, set the spectral bandwidth of the instrument to 2 nm (not setting for instruments without spectral bandwidth adjustment block), the scanning speed to medium, the sampling interval to 1 nm; and the appropriate absorbance range is set according to the instrument manual; scan with 10 nm being added to the lower wavelength limit, and 50nm being minus to the upper limit of the wavelength; and measure the difference between the absorbance at the starting point in the spectrum and the absorbance deviating from the starting point (taking the maximum deviation point) as the baseline flatness (a momentary beat is allowed when the light source or receiver is replaced).

6.3.7 Adaptation of power supply voltage

Use the voltage regulator to input 220V voltage; at the selected wavelengths of 250nm, 500nm, and 1500nm, adjust the transmittance indication value to 100%. Change the input voltage; record the transmittance indication value of the instrument at 198V and 242V, respectively; and calculate the difference from 100%, which is the adaptability of power supply voltage.

6.3.8 Stray light

Select the stray light measurement standard substance specified in 6.1.3; measure the transmittance of the standard substance at the corresponding wavelength; and the transmittance value is the stray light of the instrument at the wavelength.

a) Use sodium iodide standard solution (or cut-off filter) at 220nm for Section-A; sodium nitrite standard solution (or cut-off filter) at 360nm (tungsten lamp), 10nm standard quartz absorption tank; distilled water as reference, and at the spectral bandwidth of 2nm (not setting for the instrument without the spectral bandwidth adjustment block) to measure its transmittance indication value.

b) Section-B prism-type instrument, use a cut-off filter, at a wavelength of 420nm, with air as a reference, to measure its transmittance value.

c) Use H_2O, at the wavelength of 1420nm, in the Section-C to measure the indication value of its transmittance, taking air as the reference.

d) When it is necessary to measure the low stray light value of the instrument, an attenuator shall be used. First measure the transmittance of the attenuator; and then use the attenuator as a reference to measure the transmittance of the above standard substance; and the product of the two transmittances is the stray light.

6.3.9 Compatibility of the absorption tank

In the same optical path absorption tank attached to the instrument, put distilled water at 220nm (quartz absorption tank) and 440nm (glass absorption tank); adjust the transmittance of one absorption tank to 100%; and measure the transmittance value of other tanks. The difference is the compatibility of the absorption tank.

For instruments whose transmittance range is only 0~100%, 95% can be used instead of 100%.

6.3.10 For other types of spectrophotometers whose measurement methods are not completely included in the scope of this regulation, the main technical indicators can be verified by referring to the above verification methods; and the technical requirements can be referred to the exit-factory technical indicators requirements of the instrument.

6.4 Processing of verification results

6.4.1 The newly-manufactured instruments shall be fully verified according to the first verification items in Table 10 of 6.2. Subsequent verification and in-use inspection shall, in principle, be carried out according to the content of the subsequent verification and in-use inspection specified in Table 10; and shall be carried out according to the requirements of the first verification, if necessary.

6.4.2 For the instruments that have passed the verification according to this regulation, a verification certificate shall be issued; and the qualification level of the instrument shall be indicated with the lowest level in the verification results. If there is one item that fails the verification items (excluding 4.10), which is judged to be unqualified; issuing a verification notice, and indicating the unqualified item.

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