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Nanotechnology - Characterization of CdSe quantum dot nanocrystals - Fluorescence spectroscopy

纳米技术 硒化镉量子点纳米晶体表征 荧光发射光谱法

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1 Scope

China's national standard for characterising cadmium selenide quantum dot nanocrystals by fluorescence spectroscopy. It specifies the method principle, the instruments, the samples, the testing and the test report, with informative annexes. A quantum dot is a semiconductor crystal small enough that its electronic states are set by its size rather than only by its composition, which means the colour it emits is chosen by making the particle bigger or smaller. Cadmium selenide is the material that demonstrated the effect and is still the reference system. Fluorescence spectroscopy is the natural way to characterise them because the emission spectrum reports directly on what matters: the peak wavelength gives the mean particle size, the width of the peak gives the size distribution - a narrow emission means a uniform batch - and the quantum yield gives how much of the absorbed light comes back out, which is what determines whether the material is usable in a display or a biological label.

This standard specifies the characterization method for the fluorescence emission spectrum detection of cadmium selenide quantum dot nanocrystals. This standard is applicable to the fluorescence emission spectrum characterization of cadmium selenide quantum dot nanocrystals; the fluorescence emission spectrum characterization of other nanomaterials can also refer to this standard.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) is applicable to this standard.

GB/T 19267.2 Physical and chemical examination of trace evidence in forensic sciences - Part

3 Terms and definitions

The terms and definitions as defined in GB/T 30544.1, as well as the following terms and definitions, apply to this document.

5 Instruments

5.1 UV-visible spectrophotometer Select a UV-visible spectrophotometer that complies with JJG 178, which has a wavelength range of 300 nm ~ 800 nm.

5.2 Fluorescence spectrophotometer Fluorescence spectrophotometer is also called fluorescence spectrometer. Select a fluorescence spectrophotometer that complies with JJG 537, which has an excitation wavelength range of 250 nm ~ 700 nm and an emission wavelength range of 300 nm ~ 950 nm.

5.3 Sample cell A quartz sample cell with two or four sides transparent for spectral detection in the ultraviolet and visible light regions, which has a 1 cm optical path.

Note. The sample cell can be selected according to GB/T 9721.

5.4 Instrument calibration Depending on the instrument type, the detection instrument shall be verified/calibrated according to JJG 178, JJG 537 or tested according to GB/T 19267.2.

6 Samples

6.1 Sample preparation For the preparation method of cadmium selenide quantum dot nanocrystals, see Appendix B of GB/T 24370-2009.Cadmium selenide quantum dot nanocrystals need to be modified by specific surfactant molecules, before they can be uniformly dispersed in the solvent as experimental samples for UV-visible absorption spectrum and fluorescence emission spectrum detection. The surface ligands of cadmium selenide quantum dot nanocrystals prepared by high-temperature oil phase pyrolysis are aliphatic hydrocarbon molecules, which can be uniformly dispersed in non-polar organic solvents such as n-hexane, toluene, dichloroethane.

6.2 Test sample requirements The quantum dot samples to be measured shall be uniformly dispersed in a suitable solvent, to form a clear and transparent sol.

7 Sample testing

7.1 UV-visible absorption spectrum Use a UV-visible spectrophotometer to detect the UV-visible absorption spectrum of the cadmium selenide quantum dot nanocrystal test sample 1). Mark 450 nm and the band edge absorption peak wavelength (lambdaabs) and the corresponding absorbance on the measured UV-visible absorption spectrum line. The absorbance of the test sample used to test the fluorescence emission spectrum at the selected excitation wavelength shall be less than 0.05; the recommended absorbance is between

0.03 and 0.05.

7.2 Fluorescence emission spectrum

7.2.1 Selection of fluorescence excitation wavelength Use a fluorescence spectrophotometer to detect the fluorescence emission spectrum of the test sample in 7.1. When detecting the fluorescence emission spectrum, the excitation wavelength (lambdaex) is recommended to be selected as follows:

a) For quantum dot test samples intended for photoelectric conversion (especially in the display field), select 450 nm as the excitation wavelength;

b) For test samples intended for biomedicine or other research or application fields, any wavelength within the wavelength range of (lambdaabs - 150, lambdaabs - 30) nm can be selected as the excitation wavelength; (lambdaabs - 60) nm is recommended.

7.2.2 Test conditions The following test conditions are recommended.

a) Fluorescence excitation wavelength (lambdaex). lambda nm;

b) Fluorescence emission wavelength (lambdaem) range. (lambda + 15, lambda + 200) nm)2);

c) Slit width. 5 nm or 10 nm;

d) Temperature. 23 °C ± 2 °C.

7.2.3 Test steps

1 For test samples, test conditions, test steps, please refer to GB/T 24370-2009.

2 To avoid the influence of the double frequency peak, the maximum value of the fluorescence emission wavelength range is usually less than 2 times the excitation wavelength.

d) Temperature. 23 °C ± 2 °C.

7.3.3 Test steps The test steps for fluorescence excitation spectrum are as follows:

2.5 mL of test sample to a quartz sample cell with four transparent sides; cover it;

b) Place the sample cell in the sample slot; apply the instrument sample compartment cover to ensure good sealing and opaqueness;

c) Set the emission wavelength, excitation wavelength range, slit width, scanning speed and other parameters;

d) Start scanning to obtain the fluorescence excitation spectrum of the test sample;

e) Mark the fluorescence excitation wavelength on the fluorescence excitation spectrum line. For the detection example of cadmium selenide quantum dot fluorescence emission spectrum, please refer to Appendix A. For the measurement of relative fluorescence quantum yield of quantum dot nanocrystals, please refer to Appendix B.

8 Testing report

The testing report of quantum dot nanocrystal fluorescence emission spectrum shall include the following.

a) Test sample. 1) Test sample name; 2) Solvent.

b) Test conditions. 1) Instrument model (UV-visible spectrophotometer, fluorescence spectrophotometer); 2) Test parameters (sample cell, excitation wavelength, emission wavelength range, slit width, testing temperature).

c) Test results. 1) UV-visible absorption spectrum of the test sample; 2) Fluorescence emission spectrum of the test sample;

Appendix B

(Informative) Measurement of relative fluorescence quantum yield of quantum dot nanocrystals B.1 Overview Fluorescence quantum yield indicates the ability of a substance to emit fluorescence. According to different measurement methods, it can be divided into relative fluorescence quantum yield and absolute fluorescence quantum yield. The absolute fluorescence quantum yield can be directly measured by a calibrated steady- state/transient fluorescence spectrometer, whilst the relative fluorescence quantum yield needs to be detected and calculated using a fluorescent standard substance as a reference sample. B.2 Relative fluorescence quantum yield The measurement of relative fluorescence quantum yield requires a dilute solution of fluorescent nanomaterials as the sample to be measured. Use UV-visible spectrophotometer to test the absorbance of fluorescent standard substance and test sample at the excitation wavelength, so that the absorbance (OD value) is not greater than 0.05 4). Use fluorescence spectrophotometer to measure the fluorescence emission spectrum of fluorescent standard substance and test sample; calculate the relative fluorescence quantum yield of test sample. When measuring relative fluorescence quantum yield, the selection of the fluorescence standard substance as a