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Verification Regulation for Wavelength Dispersive X-Ray Fluorescence Spectrometers

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Verification Regulation for Wavelength Dispersive X-Ray Fluorescence Spectrometers

This Verification Regulation is applicable to the calibration of all types of wavelength dispersive X-ray fluorescence spectrometers that are newly manufactured, in use, or after repair.

1 Overview

X-ray fluorescence spectrometers are used for elemental analysis of solid, powder or liquid substances. The basic principle of operation is that the primary X-rays emitted by the X-ray tube excite the atoms in the sample. The generated fluorescent X-rays are split by a crystal and measured by a detector. The qualitative and quantitative analysis of the elements is carried out based on the wavelength and intensity of the characteristic X-ray fluorescence spectrum of various elements.

The basic structure of a wavelength dispersive X-ray fluorescence spectrometer is shown in Figure 1.

specified in the instrument technical standards" as the standard for calibrating the X-ray count rate. If the initial X-ray count rate after the replacement of the parts is equal to or lower than the factory index value, the factory index value will replace the original "initial count rate under the measurement conditions specified in the instrument technical standards" as the standard for verifying the X-ray count rate.

For new instruments within the quality assurance period, this technical requirement shall be implemented in accordance with the product technical standards.

(4) λ - The wavelength of the X-rays of the analyzed element, in nm.

(5) If the maximum count rate measured when the X-ray tube is at the maximum rated power is 61~89% of the maximum linear count rate specified by the instrument, then the count rate value deviation of Class A is calculated based on the maximum count rate measured. If the maximum count rate measured is equal to or lower than 60% of the instrument's current maximum linear count rate, no distinction is made between level A and level B. The count rate deviation is calculated based on the maximum count rate actually measured. When CD≤1%, it is level A.

2 Technical requirements

1 Appearance

1.1 The instrument shall be marked with the instrument name, manufacturer, production date and serial number.

1.2 All parts are well connected and functioning normally。

1.3 The instruments, indicator lights and safety protection devices on the panel are working properly.

2 Technical performances

Technical performance is divided into two levels: A and B, which include precision, stability, X-ray count rate, detector resolution and instrument counting linearity (see Table 1).

For new instruments within the warranty period, this technical requirement shall be implemented in accordance with the product technical standards.

3 Verification conditions

3 Verification conditions

3.1 Laboratory conditions

Power supply: There are two types of power supply: three-phase and single-phase, 220V, voltage fluctuation does not exceed ±10%.

Grounding: Single grounding resistance <30 Ω.

Cooling water: Water temperature <30°C, water pressure >9.8×10^4 Pa/cm^2, flow rate >41/min.

Room temperature: (15~28)°C±3°C.

Humidity: <75%RH.

NOTE: Different types of instruments have different requirements for the working conditions of the laboratory. Specific requirements can be determined according to the regulations of the instrument manufacturer.

3.2 Before calibration, preheat the instrument for at least 2 h at the measuring power.

3.3 Verification sample[1]

a. Pure copper or brass round block

b. Pure aluminum round block

c. Chrome nickel stainless steel round block

NOTE: Other samples for verification can be made according to the special requirements of the instrument being tested.

4 Verification items and verification methods

4 Appearance inspection

Check the appearance of the instrument visually according to 1.1~1.3.

5 Precision verification

The precision is expressed as the relative standard deviation (RSD) of 20 consecutive repeated measurements. Each measurement requires changing the mechanical setup, including crystal, counter, collimator, 2θ angle, filter, attenuator, and sample turntable position.

[1] Once the national metrology administration department has approved the application of the standard material in this regulation, it shall be used.

(1) This test is used to check the scanning channels in sequential and compound X-ray fluorescence spectrometers, as well as simultaneous X-ray fluorescence spectrometers. When testing the scan path in a simultaneous instrument, the factors in the measurement conditions vary depending on the specific instrument. For simultaneous X-ray fluorescence spectrometers without scanning tracks, this item is not tested.

(2) Measurement conditions 2 is used only as a variable test condition. The results do not need to be calculated.

6 Stability verification

The stability of the instrument is expressed by the relative range RR:

$$RR = \frac{N_{max} - N_{min}}{\bar{N}} \times 100\% \qquad (5)$$

Where,

N_{max} - the maximum count value during measurement;

N_{min} - the minimum count value during measurement;

$\bar{N}$ - the average count value for the entire measurement.

Measurement conditions: Measure the count value or count rate of CrK_α or NiK_α using a stainless-steel block sample; LiF crystal; adjust the voltage and current so that the count rate of CrK_α or NiK_α is higher than 100 kCPS; counting time is 40 s; continue measuring for 400 times.

NOTE: For simultaneous X-ray fluorescence spectrometers, measurements can be performed in fixed or scanning channels.

7 Verification of X-ray count rate

According to the test conditions specified in the technical standards of the instrument being tested, measure the count rate of the characteristic X-ray of a certain analytical element for each crystal and each fixed channel.

8 Verification of detector energy resolution

The energy resolution of the detector is expressed as a percentage of the half-peak width of the pulse height distribution and the average pulse height.

voltage of the X-ray source is set at 40 kV or 50 kV. The current is 2, 5, 10, 15, 20, 25, 30, 40, 50, 60 mA. Measure the count rate of CuK_α in sequence. The counting time is 10 s. Measure the count rate 3 times for each current value. Take the average value. Calculate the deviation of the count rate value in the same way as in 9.1.

9.3 Closed gas proportional counter. Select one of the fixed channels such as Ti, Fe, Ni, Cu or Zn for testing. The method is the same as 9.1.

5 Verification result processing and verification cycle

10 Fill in the test and calculation results according to the format in Appendix 1.

The appearance inspection of newly manufactured instruments shall be qualified. According to the technical requirements in Table 1, the tested instruments are classified as level A, level B or unqualified. Certain measures shall be taken during the use of level B instruments to ensure the accuracy of the test data. Unqualified instruments can be re-calibrated after repair.

The standards for determining instruments are as follows:

a. Level A instruments. The instrument can measure ^{11}Na to ^{92}U when all the test items listed in Table 1 are level A. The following situations are still handled as level A instruments: For sequential and compound X-ray fluorescence spectrometers, crystals with non-level-A X-ray count rates may be replaced by other level A crystals (see Appendix 2). For compound and simultaneous X-ray fluorescence spectrometers with scanning channels, non-level-A fixed channels can be replaced by test conditions that all belong to level A in the goniometer or scanning channel in the compound type, or elements that do not all meet the level A measurement conditions in the goniometer and scanning channel can be measured using level A fixed channels. For X-ray fluorescence spectrometers with only fixed channels, all channels shall be level A.

NOTE:

(1) For new instruments within the quality assurance period, the X-ray count rate and the counting linearity of the instrument shall not be lower than the exit-factory index value, otherwise the instrument cannot be judged as level A.

(2) The standard for measuring the crystal level in this article refers only to the X-ray count rate, while the standard for measuring the fixed level refers only to the X-ray count rate and the resolution of the detector. The same applies below.

b. Level B instruments. The instrument can measure ^{11}Na to ^{92}U when the verification items listed in Table 1 reach level A or level B. The following situations are still handled as level B instruments: For sequential and compound instruments, crystals with unqualified X-ray count rates may be replaced by other level A or B crystals. For composite and simultaneous instruments with scanning channels, unqualified fixed

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