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NATIONAL METROLOGY VERIFICATION REGULATION OF THE PEOPLE'S REPUBLIC OF CHINA

JJG 975-2002

Chemical Oxygen Demand (COD) Meters

化学需氧量(COD)测定仪

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Verification Regulation of Chemical Oxygen Demand (COD) Meters

1 Scope

This Regulation is applicable to the first-time verification, follow-up verification, and inservice inspection of the chemical oxygen demand meters; while the calibration of online chemical oxygen demand meters can refer to this Regulation for implementation.

2 Overview

The chemical oxygen demand (also called COD) indicates the amount of oxidant consumed by the substance that is easy to be oxidized by the oxidant in the water; it is the important indicator to evaluate the relative content of the organic substance in the water; it is mainly used for environmental monitoring in the petroleum, chemical industry, pharmaceutical-heath areas. The chemical oxygen demand meters can be divided, according to the technical principles, into 2 types; one type is spectrophotometric principle: use certain prescribed amount of potassium dichromate, under certain conditions, to oxidize water; so that the hexavalent chromium quantification changed into trivalent chromium; use the trivalent chromium absorption peak at 610nm or hexavalent chromium absorption peak at 420nm to measure the content of COD. Such meter consists of the digestion furnace part and the measurement part; the principle drawing of its measurement part can refer to Figure 1 (hereinafter refer to as Type A).

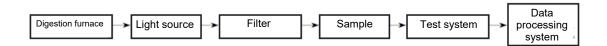


Figure 1 -- Schematic Diagram of Spectrophotometric Principle Instrument Structure

The other type is electrochemical principle: use quantitative potassium dichromate, under certain conditions, to heat and reflux the digestion sample; use the ferrous ions generated by electrolysis to react with the remained hexavalent chromium; when the hexavalent chromium is completely consumed, the electrolysis comes to end. Calculate the COD content according to the consumption of the electricity. Its principle drawing can refer to Figure 2 (hereinafter referred to as Type B).

3.2 Type B instrument

3.2.1 Indication error

Under the prescribed conditions, the instrument's indication error shall not exceed ±2.0mg/L.

3.2.2 Repeatability

Under the prescribed conditions, the measurement repeatability shall be no greater than 2%.

4 General Technical Conditions

4.1 Appearance

The instrument shall have the following signs: instrument name, model, exit-factory number, manufacturer name, exit-factory date, sign, working voltage and frequency, etc.; the instrument appearance is exempted from the damage that shall affect the normal work.

4.2 Safety performance

The insulation resistance of instrument shall be no less than $20M\Omega$.

5 Metrological Apparatus Control

The metrological apparatus control includes first-time verification, follow-up verification and in-service inspection.

5.1 Verification conditions

- **5.1.1** Equipment for verification
- **5.1.1.1** Thermometer: measuring range is 100~200°C (Grade-0.5).
- **5.1.1.2** 50, 100, 300, 1000 mg/L COD solution standard substance; its uncertainty shall be no greater than 3% (k=2).
- **5.1.1.3** 0.05mol/L $1/6K_2Cr_2O_7$ solution standard substance; its uncertainty shall be no greater than 1.0% (k=2).

5.3 Verification method

5.3.1 Appearance and routine requirements

It shall be performed through visual examination.

- **5.3.2** Access the insulation resistance meter to the power incoming line end and the housing; turn on the instrument switch, measure the insulation resistance.
- **5.3.3** Type-A instrument verification method

5.3.3.1 Error of temperature indication and temperature filed uniformity

After pre-heating the digestion furnace for 1h, evenly select 6 digestion holes; then separately insert the thermometer into them; after the temperature is stable; read 1 digit every 1min, totally read 3 times; then calculate their arithmetic average T_i . Calculate the error of temperature indication (Δ_T) and temperature filed uniformity (W) as per Formula (1) and Formula (3).

$$\Delta_T = T_0 - \overline{T} \tag{1}$$

$$\overline{T} = \frac{\sum_{i=1}^{n} T_i}{n} \tag{2}$$

Where:

 T_0 – setting temperature of digestion furnace, °C;

 T° - average temperature of the digestion furnace holes, °C;

 T_i – average temperature value for 3 times readings on the ith furnace hole, °C;

n – the number of measured furnace holes.

$$W = T_{\text{max}} - T_{\text{min}} \tag{3}$$

Where:

 T_{max} – temperature indication with maximum temperature of furnace hole, °C;

 T_{min} – temperature indication with minimum temperature of furnace hole, °C.

5.3.3.2 Error of digestion time indication

After the instrument is stable, press the instrument digestion key; stop timing when the digestion comes to end; record the digestion time; repeat for 3 times. Calculate the

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error of digestion time relative indication (Δ_t) as per Formula (4).

$$\Delta_t = \frac{x_0 - \bar{x}}{\bar{x}} \times 100\% \tag{4}$$

Where:

 Δ_t – error of digestion time relative indication;

 x_0 – setting value of digestion time;

 $\overline{x}\,$ - average value for 3 times measurements of digestion time.

5.3.3.3 Indication error

After the instrument is stable, calibrate the instrument as per the instructions; on the $0\sim150$ mg/L shift of the instrument, separately measure the 50, 100mg/L COD solution standard substance for 3 times; on the $0\sim1500$ mg/L shift of the instrument, separately measure the 300, 1000mg/L COD solution standard substance for 3 times. Calculate the indication error ($^{\Delta_{C_{A}}}$) as per the Formula (5).

$$\Delta_{C_{\rm A}} = \frac{\overline{C}_{\rm A} - C_{\rm S}}{C_{\rm S}} \times 100\% \tag{5}$$

Where:

 $\Delta_{C_{h}}$ - indication error of instrument;

 \overline{C}_{λ} - average value of 3 times measurements;

C_s – standard value of COD solution.

Take the maximum value of indication error Δ_{C_k} as the verification result of the indication error of instrument.

5.3.3.4 Repeatability

After the instrument is stable, calibrate the instrument as per the instructions; on the $0\sim150$ mg/L shift of the instrument, measure the 100mg/L COD solution standard substance for 6 times; on the $0\sim1500$ mg/L shift of the instrument, measure the 300mg/L COD solution standard substance for 6 times. Calculate the repeatability (S_A)

$$\Delta_{C_{\rm b}} = \overline{C}_{\rm B} - 40.0V \tag{8}$$

Where:

 Δ_{C_B} - indication error of instrument;

 \overline{C}_{B} - average value of 3 times measurements;

V – the number of liter of adding 0.05mol/L 1/6K₂Cr₂O₇ solution;

40.0 – the corresponding COD value for consumed 1.00mL of 0.050mol/L $1/6K_2Cr_2O_7$ solution.

Take the maximum indication error Δ_{C_B} as the verification result of indication error of the instrument.

5.3.4.2 Repeatability

Add 45mL of distilled water and 17mL of concentrated sulfuric acid into the measuring cup; slightly cool off; add 7mL of 0.5mol/L ferric sulfate solution; after cooling off to the room temperature, connect the electrode as per the instructions; perform the pre-electrolysis at the constant stirring speed. Then add 1.00mL of 0.05mol/L $1/6K_2Cr_2O_7$ solution to measure; read the indication; repeat for 6 times; then calculate the repeatability (S_B) as per Formula (6).

5.4 Treatment of verification result

The instrument that has been verified qualified as per the requirements of this Regulation shall be issued verification certificate. The disqualified instrument shall be issued notice of verification result, which indicates the disqualified items.

5.5 Verification cycle

The verification cycle is generally no more than 1 year.

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