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**Determination of formaldehyde content of waterborne
coatings - Spectrophotometric method with
acetylacetone**

水性涂料中甲醛含量的测定 乙酰丙酮分光光度法

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Determination of formaldehyde content of waterborne coatings - Spectrophotometric method with acetylacetone

1 Scope

This Standard specifies the method for determination of formaldehyde content of waterborne coatings.

This Standard applies to the test of waterborne coatings and their raw materials with a formaldehyde content of not less than 5 mg/kg.

2 Normative references

The following documents contain provisions which, through reference in this Standard, constitute provisions of this Standard. For the dated references, their subsequent amendments (excluding corrections) or revisions do not apply to this Standard. However, the parties who enter into agreement based on this Standard are encouraged to investigate whether the latest editions of these documents are applicable. For undated reference documents, the latest editions apply to this Standard.

GB/T 601 Chemical reagent - Preparations of reference titration solutions

GB/T 3186 Paints, varnishes and raw materials for paints and varnishes - Sampling (GB/T 3186-2006, ISO 15528: 2000, IDT)

GB/T 6682 Water for analytical laboratory use - Specification and test methods (GB/T 6682-2008, ISO 3696:1987, MOD)

3 Principle

Distill the formaldehyde out of the sample. In the acetic acid-ammonium acetate buffer solution with pH=6, the formaldehyde in the fraction reacts with acetylacetone under heating to form a stable yellow complex. After cooling, at a wavelength of 412 nm, perform an absorbance test. According to the standard working curve, calculate the content of formaldehyde in the specimen.

continue the titration until the blue color just disappears as the end point; record the volume V_2 (mL) of the sodium thiosulfate standard solution consumed. At the same time, make a blank sample; record the volume of the sodium thiosulfate standard solution consumed, V_1 (mL). According to formula (1), calculate the concentration of formaldehyde standard solution:

$$\rho(\text{HCHO}) = \frac{(V_1 - V_2) \times c(\text{Na}_2\text{S}_2\text{O}_3) \times 15}{20} \dots\dots\dots(1)$$

Where:

$\rho(\text{HCHO})$ - The mass concentration of formaldehyde standard solution, in grams per liter (g/L);

V_1 - The volume of sodium thiosulfate standard solution consumed in the titration of the blank sample, in milliliters (mL);

V_2 - The volume of sodium thiosulfate standard solution consumed for calibration of formaldehyde solution, in milliliters (mL);

$c(\text{Na}_2\text{S}_2\text{O}_3)$ - The concentration of sodium thiosulfate standard solution, in moles per liter (mol/L);

15 - 1/2 of the molar mass of formaldehyde;

20 - The volume of the formaldehyde standard solution pipetted during calibration, in milliliters (mL).

4.12 Formaldehyde standard diluent: 10 $\mu\text{g/mL}$. Pipette 10 mL of calibrated formaldehyde standard solution (4.11); place it in a 1000 mL volumetric flask; use water to dilute to the mark.

5 Instruments and equipment

5.1 Distillation device: 100 mL distillation flask, serpentine condenser, distillate receiver;

5.2 Graduation tube with stopper: 50 mL (the same container as the distillate receiver in 5.1);

5.3 Pipette: 1 mL, 5 mL, 10 mL, 20 mL, 25 mL;

5.4 Heating equipment: Electric heating jacket, water bath;

5.5 Balance: Accuracy is 1 mg;

5.6 Ultraviolet-visible spectrophotometer.

6 Sampling

In accordance with the provisions of GB/T 3186, take a representative sample of the product under test.

7 Test procedure

7.1 Drawing of standard working curve

Take several graduation tubes with stopper (5.2); pipette 0.00 mL, 0.20 mL, 0.50 mL, 1.00 mL, 3.00 mL, 5.00 mL, and 8.00 mL of formaldehyde standard diluent (4.12) into them, respectively. Add water to dilute to the mark; add 2.5 mL of acetylacetone solution (4.4) and shake well. In a 60 °C constant-temperature water bath, heat for 30 min; take it out and cool to room temperature. Use a 10 mm cuvette (with water as a reference) to test the absorbance at a wavelength of 412 nm on an ultraviolet-visible spectrophotometer (5.6).

Use the mass (μg) of formaldehyde in the graduation tube with stopper as the abscissa; use the corresponding absorbance (A) as the ordinate, to draw a standard working curve. The calibration coefficient of the standard working curve shall be ≥ 0.995 . Otherwise a new standard working curve shall be made.

7.2 Test of formaldehyde content

Weigh about 2 g (accurate to 1 mg) of the well-stirred specimen; place it in a 50 mL volumetric flask; add water and shake well, and dilute to the mark. Then use the pipette to transfer the specimen aqueous solution in the 10 mL volumetric flask; place it in the distillation flask (5.1) which has been pre-filled with 10 mL of water. And add a small amount of zeolite to the distillation flask; add an appropriate amount of water to the distillate receiver (5.2) in advance; immerse the distillate outlet; use an ice water bath to cool the exterior of the distillate receiver (5.2) (see Figure 1 for the distillation device). Heat and distill to steam the specimen to nearly dryness; remove the distillate receiver (5.2); use water to dilute to the mark and wait for measurement.

If the specimen to be tested is not easy to disperse in water, directly weigh about 0.4 g (accurate to 1 mg) of the evenly-stirred specimen; place it in a distillation flask that has been pre-added with 20 mL of water; shake it gently, and then carry out the distillation process.

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