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**Dyestuffs – Determination of Solubility in Organic Solvents
– Gravimetric and Photometric Methods**
(ISO 7579:2009, MOD)

染料 在有机溶剂中溶解度的测定 重量法和光度法

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Dyestuffs – Determination of Solubility in Organic Solvents

– Gravimetric and Photometric Methods

1 Scope

This Document describes 2 methods for determining the solubility of dyes in organic solvents.

This Document applies to dyes that do not undergo chemical changes under the influence of solvents and are stable and non-volatile under specified drying conditions. The applicable dye mass concentration is 1g/L~1000g/L. If the viscosity of the solution satisfies the conditions for smooth operation, higher concentrations can also be used. For volatile solvents (boiling point less than 120 °C), the gravimetric method is used; for less volatile solvents (boiling point greater than 120°C), the photometric method is used.

This Document does not apply to the determination of insoluble matter in dyes.

2 Normative References

The provisions in following documents become the essential provisions of this Document through reference in 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 Document.

GB/T 3186 Paints varnishes and raw materials for paints and varnishes – Sampling (GB/T 3186-2006, ISO 15228:2000, IDT)

GB/T 5211.3 General methods of test for pigments and extenders - Part 3: Determination of matter volatile at 105°C (GB/T 5211.3-2020, ISO 797-2:1981, MOD)

GB/T 6750 Paints and varnishes - Determination of density - Pycnometer method (GB/T 6750-2007, ISO 2811-1:1997, IDT)

3 Terms and Definitions

For the purposes of this Document, the following terms and definitions apply.

3.1 Solubility

The maximum mass of dye that can be dissolved in a certain volume of a certain solvent under certain conditions.

NOTE: Solubility is expressed in grams per liter (see Appendix A). No distinction is made between "true" solubility and "colloidal" solubility.

4 Principle

At 23°C, dyes of different masses were dispersed in a certain volume of solvent. After mixing for 3 h, each dispersion was centrifuged, and the solid content in the supernatant was measured by gravimetric or photometric method.

When using the gravimetric method for measurement, the amount of dissolved dye is assessed by measuring the amount of non-volatile matter in the solution.

When using photometric method for measurement, the amount of dissolved dye is evaluated by comparing and determining the absorbance of the test solution and the standard solution.

5 Solvents

When using the gravimetric method, the used organic solvent shall evaporate completely below the decomposition temperature of the dye. The density of organic solvents at 23°C shall be known, and the density of the solvent shall be measured by using a glass pycnometer in accordance with the provisions of GB/T 6750. The dye shall not react chemically with the solvent.

Since organic solvents are usually not chemically pure, the purity grade (including the type and content of major contained impurities) shall be given in the test report.

For solvents with boiling points above 120°C, it is recommended to use the photometric method.

6 Apparatus

6.1 Balance: with accuracy of 0.0001g.

6.2 Weighing bottle: flat with lid.

6.3 Container, cylinder, with capacity of approximately 50mL, made of inert material, equipped with a sealing lid.

6.4 Pipette: with capacity of 20mL±0.03mL.

6.5 Volumetric flask: with capacity of 100mL±0.5mL.

If the limit value of the dye cannot be obtained because the viscosity continues to increase as the amount of dye increases, the last maximum obtained value shall be recorded in accordance with A.4.

8.2.2 Put the required amount of dye into the container (6.3); then accurately add 20mL of solvent with a pipette (6.4); and cover it immediately to prevent the solvent from evaporating. Shake with a mechanical vibrator for 3 h at $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$; and check to confirm that there is no significant agglomeration. If an orbital vibrator is used, it shall be stated in the test report.

8.2.3 After shaking the dye and solvent for 3 h, put the suspension into a centrifuge tube, cover it; and centrifuge the tube at $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$ for 10 min. Check whether the supernatant in the centrifuge tube is clear, for example, use a pipette to absorb the supernatant and observe whether the solution flows smoothly. If it is not clear or in doubt, centrifuge for another 10 min; pour the supernatant in each tube into a clean and dry container (6.3), and cover the lid.

8.3 Gravimetric determination of dissolved dye concentration

8.3.1 General

Take an appropriate amount of the supernatant prepared in 8.2.3; measure the dye concentration according to procedure 8.3.2, and weigh it accurately to 0.2 mg.

The amount of supernatant taken for each determination shall contain at least 30 mg of dye.

Each supernatant shall be measured at least 2 times, the average value shall be recorded, and calculated according to Formula (1).

8.3.2 Test procedures

Place about 3g of the clear supernatant into a weighing bottle with constant weight, cover it; and weigh the mass (m_0) of the supernatant. Then, remove the lid and place it in the oven (6.10); and maintain the temperature at 10°C below the boiling point of the solvent. Take it out after most of the solvent has evaporated (about 1 h).

Raise the temperature to about 30°C lower than the decomposition temperature of the dye, and continue drying for 3 h.

NOTE: Most metal complex dyes can be dried at 150°C .

If the drying temperature is no higher than 50°C above the boiling point of the solvent, extend the drying time or remove the residual solvent in a vacuum furnace so that there will be no loss caused by dye sublimation.

After drying, place the weighing bottle and lid in a desiccator to cool, and then weigh.

If this operation is performed the first time for a specific dye/solvent combination, check that the dye has dried to a constant mass by repeating the drying/weighing operation on a second

saturation concentration (see A.1), the repeatability is within $\pm 5\%$. If the situation is as described in A.2 and A.3, the repeatability will be very poor.

8.4 Photometric method for determination of dissolved dye concentration

8.4.1 General

This method is mainly aimed at non-volatile solvents. If used in volatile solvents, there will be a risk of data errors due to the loss of solvent during the test process.

If a large number of samples are to be analyzed, even with less volatile solvents, consider gravimetric analysis method as it allows for faster processing of samples in parallel. Alternatively, use the same solvent for both the calibration solution and all other sample solutions, so that the photometry shall be faster and there shall be no need to prepare many calibration solutions. At high dilutions, the presence of small solvent losses in the test solution is inconsequential.

Before using a photometric method, it is necessary to verify the applicability of the method to the used sample/solvent combination. If the verification is successful, there is no need to repeat the verification on subsequent samples of the same type.

The process can be significantly automated by using special measuring, dosing and dilution equipment.

The test solution shall comply with Beer-Lambert's law and be stable enough to ensure repeatability of measurement. If the maximum value of the absorption peak is unstable during multiple repeated measurements, another more stable peak shall be selected for calculation, or the entire spectrum shall be considered for evaluation.

8.4.2 Preparation of calibration solutions

Calibration solutions shall comply with Beer-Lambert's law. If you choose a 2cm colorimetric tank, it is more appropriate to choose a mass concentration of about 0.15g/L for yellow dye (low absorption) and about 0.02g/L for blue dye (high absorption).

Accurately weigh 100.0mg of dye into the weighing bottle (6.2) and transfer it to the 100mL volumetric flask (6.5). Be careful not to lose it. Add 60mL of solvent to dissolve the dye in the ultrasonic bath (6.12). If necessary, cool to room temperature. Dilute to volume with solvent and shake well.

The mass concentration of the dye solution prepared in this way is 1.0g/L. In order to comply with Beer-Lambert's law, the solution shall be diluted to 0.2g/L or 0.02g/L.

NOTE: If using volatile solvents and accurate dilution is difficult, consider using the gravimetric method.

Since the density of a low-concentration dye solution is almost the same as that of a pure solvent, the mass of the solution required for dilution is calculated using Formula (2):

$$m = V\rho \quad \dots\dots\dots (2)$$

Where:

m – mass of solution, in g;

V – volume of solution, in mL;

ρ – density of solution at 23°C, in g/mL.

Weigh the required mass of solution into a weighing bottle; or use a syringe (for volatile solvents) to absorb it; and transfer it to a 100mL volumetric flask (6.5). Rinse the weighing bottle or syringe; and pour the rinse solution together into a volumetric flask; dilute to volume with solvent and shake well.

8.4.3 Preparation and dilution of solution to be tested

Prepare test solutions according to 8.2 and dilute each solution to a concentration equivalent to the calibration solution.

For instance, in order to obtain the 0.02g/L solution, the following 2 dilution procedures are required:

- a) 1 g/L;
- b) 0.02 g/L.

It is the same procedures as for preparing calibration solution. It is very important that the weighing amount of the sample shall not be too small to minimize the error caused by the loss of solvent. The weighing amount of the sample shall be greater than 1g. Using a 10mm colorimetric tank can reduce the number of dilutions.

EXAMPLE:

The solubility of a dye in cyclohexanone is about 100g/L.

The cyclohexanone solution of the dye contains 2g of dye and 20mL of cyclohexanone. The density of cyclohexanone is 0.95g/mL, the mass of the solvent is 20mL×0.95g/mL=19g, and the total mass is 2g+19g=21g.

For dilution, 0.1g of dye is required, which is equivalent to 1/20 of the total mass.

Assuming that all dye is dissolved, 21/20g=1.05g of supernatant is required.

Weigh the above-mentioned mass of the supernatant into a weighing bottle and transfer it to a 100mL volumetric flask. Rinse the weighing bottle with solvent; merge the rinse liquid into the volumetric flask; dilute to the mark with solvent; and shake well.

C_c - The mass concentration of the dye in the calibration solution, in g/L;

A_c - the absorbance of the calibration solution at the selected maximum absorption wavelength;

C_s - The mass concentration of the diluted test solution based on the supernatant (see 8.4.3), in g/L.

Solubility (S) under the used conditions (see 3.1), the ratio of the mass of the dye in the supernatant to the volume of the solvent, that is, the mass concentration of the dye in the solvent is expressed in g/L, which is calculated according to Formula (4):

$$S = \frac{C_s \cdot D \cdot \rho}{C_s \cdot (1 - D)} = \frac{D \cdot \rho}{1 - D} \quad \dots\dots\dots (4)$$

Where:

S – solubility of the dye, in g/L;

ρ – density of the solution at 23°C, in g/L.

9 Report

The test report shall contain at least the following information:

- a) All details required to identify the product under test and its non-volatile substance content;
- b) This Document number;
- c) The used solvents and their purity;
- d) Mixing methods of dyes and solvents;
- e) Method used to determine the amount of dissolved dye (gravimetric or photometric method);
- f) Test results are expressed in accordance with the provisions of 8.3.4 or 8.4.5;
- g) Any deviation from prescribed procedures;
- h) Test date.

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