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Second edition
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Dyestuffs — Determination of solubility in organic solvents — Gravimetric and photometric methods

Colorants — Détermination de la solubilité dans les solvants organiques — Méthodes gravimétrique et photométrique

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 7579 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 2, *Pigments and extenders*.

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This second edition cancels and replaces the first edition (ISO 7579:1990), in which the mixing time has been reduced from 24 h to 3 h but the temperature has been increased from 105 °C to 150 °C, Method B and Annex B have been deleted and a photometric test method has been added.

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Introduction

Many dyestuffs are soluble in a solvent to an extent which is independent of the amount of dyestuff present in the solvent, as long as excess dyestuff is present. This concentration is defined as the saturation concentration and represents the solubility of the dyestuff in the solvent. In some cases, however, there is no fixed saturation concentration and the amount of dyestuff which dissolves increases with the amount of dyestuff added. A gravimetric and a photometric procedure to assess the solubility of these dyes are described in this International Standard.

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Dyestuffs — Determination of solubility in organic solvents — Gravimetric and photometric methods

1 Scope

This International Standard specifies two methods for determining the solubility of dyestuffs in organic solvents. They are applicable to dyestuffs that do not change chemically under the influence of the solvent and are stable and non-volatile under the specified drying conditions. For volatile solvents (boiling point < 120 °C), the gravimetric procedure is recommended and, for less volatile solvents (boiling point > 120 °C), the photometric procedure is recommended. The choice of procedure should be made on a case-by-case basis.

The methods are suitable for concentrations between 1 g and 1 000 g of dyestuff per litre of solvent. Higher concentrations can be used provided the viscosity of the solution is such that the procedure can be carried out readily.

The methods are not suitable for the determination of insoluble matter in a dyestuff.

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2 Normative references

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The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 787-2, *General methods of test for pigments and extenders — Part 2: Determination of matter volatile at 105 °C*

ISO 2811-1, *Paints and varnishes — Determination of density — Part 1: Pyknometer method*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

3 Terms and definitions

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

3.1
solubility
maximum mass of a dyestuff that is soluble in a given volume of a particular solvent under specified conditions

NOTE Solubility is expressed in grams per litre of solvent (see Annex A). No distinction is made between “true” solubility and “colloidal” solubility.

4 Principle

Different amounts of the dyestuff are each dispersed in a defined volume of a solvent at 23 °C. After mixing for 3 h, each dispersion is centrifuged and the solids content of the supernatant liquid is determined by either gravimetric or photometric measurements.

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When using the gravimetric procedure, the amount of dyestuff which dissolves is assessed by determination of the non-volatile-matter content of the solution.

When using the photometric procedure, the amount of dyestuff which dissolves is assessed by determination of the light absorbance compared to that of a standard solution.

5 Solvent

When using the gravimetric procedure, the organic solvent used shall be completely volatile at a temperature below the temperature of decomposition of the dyestuff. The density of the solvent at 23 °C shall be known. The density of the solvent can be determined by the method described in ISO 2811-1 at 23 °C using a glass pyknometer. The dyestuff shall not react chemically with the solvent.

As organic solvents are normally not chemically pure, the grade of purity (including the type and quantity of any major secondary constituents) shall be given in the test report.

With solvents having a boiling point above 120 °C, the photometric method is recommended.

6 Apparatus

Ordinary laboratory apparatus and glassware, together with the following:

6.1 Balance, accurate to 0,0001 g.

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6.2 Weighing bottles, squat form, with stoppers.

6.3 Containers, cylindrical, of capacity about 50 ml, of inert material, with tightly fitting lids.

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6.4 Pipette, capacity 20 ml (tolerance: ± 0,03 ml).

6.5 Volumetric flask, capacity 100 ml (tolerance: ± 0,5 ml).

6.6 Injection syringes, capacity 2 ml and 5 ml.

6.7 Mechanical shaker, with speed control and, preferably, a cooling unit.

6.8 Laboratory centrifuge, capable of imparting a relative centrifugal acceleration of about 10 km/s^2. A centrifuge with a rotational frequency of 2 000 min^{-1} to 3 000 min^{-1} is suitable.

6.9 Centrifuge tubes, of transparent and inert material, with tightly fitting lids.

6.10 Drying oven, with air circulation and temperature control up to 150 °C (tolerance ± 2 °C).

6.11 Spectrophotometer.

6.12 Ultrasonic bath.

7 Sampling

Take a representative sample of the product to be tested, as described in ISO 15528.

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8 Procedure

8.1 Preliminary determination

If the approximate solubility of the dyestuff in the solvent is not known, carry out a preliminary determination, using the procedure described in 8.2.2 and 8.2.3, with one of the following series of test portions of the dyestuff:

— dyestuff of low solubility: 1 g, 10 g and 50 g of dyestuff per litre of solvent;

— dyestuff of high solubility: 100 g, 500 g and 1 000 g of dyestuff per litre of solvent.

Take as the approximate solubility of the dyestuff the concentration of the dispersion in which the undissolved matter after centrifuging is greater than about one-quarter of the total quantity of dyestuff dispersed.

8.2 Preparation of test solutions

8.2.1 When the approximate solubility of the dyestuff is known or has been determined in the preliminary determination, carry out the following procedure.

Prepare six suspensions by taking test portions of the dyestuff to give concentrations of about 40 %, 60 %, 80 %, 100 %, 120 % and 140 % (by mass) of the approximate solubility, using the procedure described in 8.2.2 and 8.2.3. If, at the lowest concentration employed, 10 % or more of the dyestuff remains undissolved, reduce the concentration until more than 90 % of the dyestuff is dissolved. If, at the highest concentration, the residue is less than 25 % of the test portion, then increase the mass of the test portion.

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If the solubility limit cannot be obtained because of a large increase in viscosity with increasing amount of the test portion, the last value obtained shall be recorded (see Clause A.4.)

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8.2.2 Place the required amount of dyestuff in a container (6.3) and add exactly 20 ml of the solvent, using the pipette (6.4). Close the container immediately to prevent loss of solvent.

Shake the container using a mechanical shaker (6.7) at (23 ± 2) °C for 3 h. Check that no significant agglomerates are visible. If an orbital shaker is used, this shall be stated in the test report.

8.2.3 After shaking the dyestuff and solvent for 3 h, place portions of the suspension in centrifuge tubes (6.9) and cap each tube. Centrifuge the tubes at (23 ± 2) °C for 10 min. Check whether the supernatant liquid is clear, for instance by observing whether the supernatant liquid flows smoothly from a pipette tube dipped into it; if not, or if in doubt, centrifuge for another 10 min. Decant the supernatant liquid from each tube into a clean, dry container (6.3) and close securely.

8.3 Gravimetric determination of the concentration of the dissolved dyestuff

8.3.1 General

Take a known amount of the supernatant liquid prepared in 8.2.3 and determine the concentration of the dyestuff using the method described in 8.3.2. Carry out the weighings to the nearest 0,2 mg.

For each determination, the amount of supernatant liquid taken shall contain at least 30 mg of the dyestuff.

Two determinations on each supernatant liquid shall be carried out. The mean values shall be recorded and used for the calculation as given in 8.3.4.

8.3.2 Procedure

Place a portion (about 3 g) of the clear supernatant liquid into a previously tared weighing bottle (6.2), insert the stopper and weigh (m_0). Remove the stopper, place the weighing bottle in the oven (6.10) maintained at a

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