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Part Z07:

Determination of application solubility and
solution stability of water-soluble dyes

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*Partie Z07: Détermination de la solubilité à l'application et de la stabilité
en solution des colorants solubles dans l'eau*

INTERNATIONAL

ISO



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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.

International Standard ISO 105-Z07 was prepared by Technical Committee ISO/TC 38, *Textiles*, Subcommittee SC 1, *Tests for coloured textiles and colorants*.

ISO 105 was previously published in thirteen "parts", each designated by a letter (e.g. "Part A"), with publication dates between 1978 and 1985. Each part contained a series of "sections", each designated by the respective part letter and by a two-digit serial number (e.g. "Section A01"). These sections are now being republished as separate documents, themselves designated "parts" but retaining their earlier alphanumeric designations. A complete list of these parts is given in ISO 105-A01.

Annex A of this part of ISO 105 is for information only.

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Textiles — Tests for colour fastness —

Part Z07:

Determination of application solubility and solution stability of water-soluble dyes

1 Scope

This part of ISO 105 describes a method for the determination of the application solubility of water-soluble dyes in the range 40 °C to 90 °C and of their solution stability. The method is not intended to measure absolute solubility.

NOTE 1 Several factors which may influence test results are listed in annex A.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 105. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 105 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1773:1976, *Laboratory glassware — Boiling flasks (narrow-necked)*.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

3 Principle

Several solutions of known concentration, including the solubility limit, of the dye to be tested are pre-

pared at a specified temperature. The solutions are then filtered under suction at this temperature in a heatable Nutsch filter and the application solubility limit determined by visual assessment of the filter residues and the measured flow-through time of the filtrate.

The application solubility of dyes is normally determined at 90 °C. For certain classes of dyes the solubility is determined at a lower temperature. In selecting the test temperature, the manufacturer's recommendations are followed. The temperature is indicated in the test report (e.g. application solubility limit determined at 90 °C, 60 °C, etc.).

The solution stability of dyes is determined by storing for 2 h and, as the case requires, cooling the above-mentioned solution before filtration and assessment. The dissolving and storage temperatures are indicated in the test report (e.g. solution stability at 90 °C/60 °C, 60 °C/60 °C, etc.).

4 Apparatus and reagents

4.1 Erlenmeyer flask, wide-mouthed, capacity 500 ml, complying with ISO 1773.

4.2 Heating bath, thermostatically controlled, with magnetic stirring bar 40 mm long by 6 mm diameter, speed of stirrer 500 r/min to 600 r/min.

4.3 Water bath, with temperature regulator (heating/cooling) for adjusting the storage temperature (e.g. 60 °C, 30 °C or 25 °C).

4.4 Nutsch filter (Büchner funnel), heatable, of glass, steel or porcelain, of inner diameter 70 mm, capacity at least 200 ml, having more than 100 holes with a total surface area of holes (evenly distributed) of not less than 200 mm².

4.5 Thermostatic device (optional), with circulation pump to adjust temperature of Nutsch filter.

4.6 Vacuum apparatus.

4.6.1 Suction bottle, capacity 1 litre to 2 litres.

4.6.2 Piston or membrane pump, of sufficiently high suction capacity to create a full vacuum of at least 50 kPa under pressure.

4.6.3 Apparatus to adjust and maintain a given vacuum, preferably coupled with a manometer.

4.7 Stopwatch, to measure flow-through time.

4.8 Filter paper, circular, 70 mm ± 2 mm diameter.

NOTE 2 Filter papers of the following characteristics have been found suitable:

Property	Two typical sets of values	
Grammage, g/m ²	92	121
Thickness, µm	210	330
Air resistance, Gurley, s/100 ml	3,6	1
Wet burst strength, kPa	> 1	> 4
Surface appearance	smooth	smooth

See ISO 105-A01:1994, clause 8, note 1 for information on sources of supply of suitable filter paper.

The type of filter paper used and the manufacturer shall be listed in the test report.

4.9 Water, complying with grade 3 of ISO 3696, used as dye solvent.

An amount of 200 ml is designated as normal. More water may be added to the solution, but such additions shall be reported together with the dye solubility values.

NOTE 3 No account is taken of changes in volume as a function of temperature, or those caused by the addition of dye.

5 Preparation of solutions

5.1 The concentrations at which the dye solutions are prepared shall be chosen considering the expected application solubility limit of the dye:

Expected limit to fall between	Stepwise increase in dye concentration approaching limit
1 g/l to 10 g/l	1 g/l
10 g/l to 50 g/l	5 g/l
50 g/l to 100 g/l	10 g/l
above 100 g/l	20 g/l

5.2 To determine solubility at 90 °C, paste a known amount of the test dye and introduce it into the wide-mouthed Erlenmeyer flask (4.1) with a portion of the 200 ml water (4.9) at about 60 °C, but not in excess of the dissolving temperature of the dye. When the dye is completely wetted out, add the rest of the water to the flask.

Place this solution into the heating bath (4.2) maintained at 95 °C. Switch on the magnetic stirrer. When the solution has attained a temperature of 95 °C ± 2 °C, continue stirring at this temperature for a further 5 min (total stirring time approximately 10 min).

Then filter the solution (see clause 6) immediately to determine the solubility of the dye at 90 °C.

Repeat the process for each concentration of dye under test.

5.3 To determine solubility at temperatures below 90 °C, paste a known graduated amount of the test dye and introduce it into a wide-mouthed Erlenmeyer flask with a portion of the 200 ml water (4.9) at the desired dissolving temperature until the dye is completely wetted out. Then add the rest of the water to the flask.

Place the solution in the heating bath maintained at the desired dissolving temperature; stir the solution for 10 min and then filter (see clause 6).

Repeat the process for each concentration of dye under test.

5.4 To determine the solution stability at the desired temperature (e.g. 60 °C, 30 °C or 25 °C), place the Erlenmeyer flask with the solution prepared according to 5.1 or 5.2 into a bath maintained at the desired

temperature (see 4.3) and allow to stand for 2 h before filtration. Before filtering, mix the solution thoroughly by tilting the flask to and fro.

6 Filtering the solutions

NOTE 4 In order to avoid any temperature shock effects, it is essential that heated solutions are filtered through equipment already brought to the same temperature as the solution under test. Ideally this is best done using a jacketed filter funnel, but acceptable results can also be obtained using preheated funnels, either by immersion in a water bath or oven, or by passing water preheated to the test temperature through the equipment immediately prior to carrying out the test. When using this latter technique, the amount of water should be determined locally in order that the filter funnel can be heated to the same temperature irrespective of its geometry and the ambient conditions. In all cases when using preheating techniques rather than a jacketed funnel, the test solution should be passed through the test equipment immediately after removing it from its heating medium.

6.1 Preheat the Nutsch filter (4.4) to the test temperature and maintain at this temperature throughout the entire filtration operation.

6.2 Immediately before filtering, wet out two filter papers (4.8) in the Nutsch filter in a double layer using at least 50 ml water at the test temperature.

6.3 Adjust the vacuum (4.6) to 3 kPa to 4 kPa, which is equivalent to 300 mm to 400 mm water column pressure.

6.4 Filter the dye solution obtained as in 5.1, 5.2 or 5.3 at the recommended temperature and measure the flow-through time with a stopwatch. Visually examine the flask which contained the solution to determine whether any residue remains.

6.5 If the solution does not filter within 2 min at a stabilized vacuum, filter for an additional maximum 2 min under full vacuum (see 4.6.2).

6.6 After the solution has flowed through, continue to extract the filter uniformly under full vacuum for 1 min.

6.7 Allow the filters to dry completely at room temperature before evaluation.

7 Evaluation

7.1 Compare visually the dried filters after filtration of the various dye solutions of known concentrations. The application solubility limit, or the solution stability limit, is taken as that concentration at which filter residues are seen. Residues that are difficult to see may possibly be detected by gently rubbing the filter surface with a fingertip.

7.2 Flow-through time may be used as a further evaluation criterion. A sudden sharp increase in flow-through time when moving up the range of solution concentrations indicates that the application solubility limit has been exceeded or that the solution is no longer stable.

8 Test report

The test report shall include the following information:

- a) number and year of publication of this part of ISO 105, i.e. ISO 105-Z07:1995;
- b) full identification of the dyestuff under test;
- c) type of filter paper used and its manufacturer;
- d) application solubility limit of the dyestuff, expressed in grams per litre, including the dissolving temperature;
- e) solution stability, expressed in grams per litre, including the dissolving and storage temperatures;
- f) flow-through time, where applicable (see 7.2);
- g) any special observations during the test or evaluation procedure;
- h) any deviation, by agreement or otherwise, from the procedure specified (e.g. amounts of solvent other than 200 ml, etc.).

Annex A

(informative)

Factors affecting results

This test method has given good results over several years. However, it should be pointed out that test conditions which deviate from those specified may lead to quite different results.

For example, the results may be influenced when:

- a) A different filter is used. The filter selected for the test should represent a compromise with respect to permeability and should do full justice to the practical conditions.
- b) Other dissolving temperatures are used. Many dyes dissolve well at temperatures appreciably below 90 °C (or the given test temperature). However there are dyes which readily dissolve at 90 °C but are difficult to dissolve at 85 °C.
- c) Other storage temperatures and storage times are used.
- d) Water having different hardness or added electrolyte is used.

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