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Textiles — Tests for colour fastness —

Part Z08:
Determination of solubility and solution stability of reactive dyes in the presence of electrolytes

Textiles — Essais de solidité des teintures —

Partie Z08: Détermination de la solubilité et de la stabilité en solution des colorants réactifs en présence d'électrolytes

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ISO 105-Z08:1995(E)

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-Z08 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 Z08:
Determination of solubility and solution stability of reactive dyes in the presence of electrolytes

1 Scope

This part of ISO 105 describes a method for the determination of the solubility and the solution stability of reactive dyes for use in batchwise and continuous dyeing processes in the presence of electrolytes.

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 solution stability limit, of the dye to be tested are prepared at a specified temperature in accordance with the dye manufacturer's recommendation. The dye solution is adjusted to the desired test temperature. A specified amount of the desired electrolyte solution is added and the resultant solution either filtered immediately or stored for a specified time at a specified temperature and subsequently filtered under suction.

The solubility and/or solution stability limits of the dye solution to the addition of electrolyte are determined by visual assessment of the filter residues and the measured flow-through time of the filtrate.

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.

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 mm^2.

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 (recommended pH 7), used as solvent.

4.10 Electrolyte solution (stock solution), prepared using grade 3 water (4.9) according to the concentrations given in table 1 or such that the required quantity of the desired electrolyte is contained in 50 ml of solution.

NOTES

3 The reproducibility of the test method depends on the quality of the electrolyte. For this reason, chemically pure substances should be used.

4 Although the electrolytes used are mostly simple salts, tests of the stability of their solutions, which need to be carried out rapidly, often prove not very meaningful. Electrolyte solutions should therefore be discarded at the latest one week after their preparation.

5 Test parameters

The parameters chosen for the test, including dissolving, storage and filtration temperatures, electrolyte chosen and its concentration, will depend on the type of dyestuff being tested. Examples of combinations of test parameters are given in table 1.

6 Preparation of solutions

6.1 The concentrations at which the test dye solutions are prepared shall be chosen considering the expected solution stability limit of the dye in the presence of electrolyte:

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 30 g/l	5 g/l
30 g/l to 100 g/l	10 g/l

The maximum dye concentrations used for the test shall be:

30 g/l for batchwise dyeing processes;

100 g/l for continuous dyeing processes.

6.2 Place a known amount of the dye to be tested in the wide-mouthed Erlenmeyer flask (4.1) and add 150 ml water (4.9) at about 60 °C (but not above the dissolving temperature). Swirl the flask by hand until the dye is fully wetted out (predissolving).

If the addition of urea is required as a dissolving agent in accordance with the recommendations of the dye manufacturer, reduce the amount of water by 1 ml per gram of urea added, so as not to change the total volume of the test solution.

Place the solution into the heating bath (4.2), set at the desired dissolving temperature and stir for 10 min at 500 r/min to 600 r/min.

6.3 In order to simulate dyebath compositions used in commercial practice, additions of other components to the test solution at this point are permissible (see dye manufacturer's recommendations). Any such additions, including their influence on the volume of test solution, shall be noted in the test report.

NOTE 5 Changes in volume attributable to changes in temperature need not be taken into account.

6.4 Adjust the dye solution to the desired temperature for filtration or storage using the water bath (4.3), add 50 ml of the electrolyte solution (4.10) previously adjusted to the test temperature, and homogenize the resulting solution.

Table 1 — Examples of combinations of test parameters

Dyeing process	Dissolving temperature of dye °C	Filtration/ storage temperature °C	Storage time min	Electrolyte added and concentration of stock solution
Batchwise	90	40	120	Sodium chloride, 50 g/l
	60	25	30	Sodium chloride, 60 g/l Sodium carbonate ($Na_2CO_3 \cdot 10H_2O$), 5 g/l
	50	50	0	Sodium chloride, 60 g/l
	80	25	60	Sodium chloride, 50 g/l Sodium carbonate, 5 g/l Sodium hydroxide, 2 ml/l prepared from solution containing approximately 435 g/l
	90	40	90	Sodium sulfate (anhydrous), 50 g/l Sodium carbonate, 5 g/l Sodium hydroxide, 2 ml/l prepared from solution containing approximately 435 g/l
Continuous	90	25	30	Sodium hydroxide, 50 g/l Sodium hydroxide, 30 g/l prepared from solution containing approximately 435 g/l
	90	25	120	Sodium carbonate ($Na_2CO_3 \cdot 10H_2O$), 40 g/l
	90	25	60	Sodium silicate, 95 g/l prepared from solution containing between 34 % and 40 % sodium silicate Sodium hydroxide, 38 g/l prepared from solution containing approximately 435 g/l
	90	25	30	Urea, 100 g/l Sodium carbonate ($Na_2CO_3 \cdot 10H_2O$), 30 g/l

7 Storage

Allow the final test solution to stand at constant temperature for the specified storage time (see table 1).

8 Filtering the solutions

NOTE 6 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.

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

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

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

8.4 Before filtering, homogenize the test solution by swirling it in the flask (visual check). Filter the entire test solution (do not decant) at the desired temperature. Measure the flow-through time with a stopwatch.

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

8.6 After the solution has passed through, continue to extract the filters uniformly under full vacuum for 1 min.

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

9 Evaluation

9.1 Compare visually the dried filters after filtration of the various test solutions of known dyestuff concentrations. The solubility limit, or the solution stability limit, in the presence of electrolyte 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.

9.2 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 solubility limit has been exceeded or that the solution is no longer stable.

10 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-Z08:1995;

b) full identification of the dyestuff under test;

c) full identification and amount of added electrolyte(s);

d) type of filter paper used and its manufacturer;

e) solubility limit of the dyestuff, in the presence of electrolyte expressed in grams per litre, including the dissolving temperature;

f) solution stability in the presence of electrolyte, expressed in grams per litre, including the dissolving and storage temperatures;

g) flow-through time, where applicable (see 9.2);

h) any special observations during the test or evaluation procedure;

i) any deviation, by agreement or otherwise, from the procedure specified (e.g. total volume of solution other than 200 ml, addition of urea or other components, etc.).

Annex A
(informative)

Factors affecting results

This test method has been successfully used to monitor the quality of dyes for some time. However, since there are many test variants that depend on the production and application methods used, the electrolyte stability data given in the literature are not as easy as, for example, solubility data to evaluate. Also, with electrolyte stability different test conditions may lead to different results.

In addition to the factors already known to influence solubility testing without electrolyte, such as filter quality, dissolving temperature, storage temperature and storage time, here the influence of the electrolyte should also be taken into account. This includes such factors as electrolyte purity and ageing of the electrolyte stock solution. In order to increase comparability and reproducibility, the test should be carried out with chemically pure substances, which are normally not used in works practice. Depending on the type and amount of impurity, the values found in the evaluation with salts of technical quality may be widely scattered.

Ageing of the stock solution will of course only affect laboratory tests, since in actual practice such solutions are hardly ever used, certainly not solutions that have been stored for any length of time. However, in the laboratory the salts in highly concentrated solutions that are mostly used may, with prolonged storage, crystallize out or become chemically changed.

For the sake of simplifying the work, fresh solutions as a rule are not used for each test; however, the age of the solutions is limited, for the reasons given above. In cases of dispute, fresh solutions should always be prepared.

It should also be borne in mind that the occurrence of an unexpected interaction between the dye and the electrolyte is possible, and this may lead to equivocal results. Thus, for example, with a particular high concentration of electrolyte and a low mass of dye, precipitation may occur that does not take place with high masses of dye.

Although many process variants are possible in dyeing with reactive dyes, and production dyeing conditions are often not comparable with laboratory conditions, this test provides useful indications about the sensitivity of individual reactive dyes to the presence of electrolytes.

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