INTERNATIONAL STANDARD

ISO 105-Z11

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Textiles — Tests for colour fastness —

Part Z11:
Evaluation of speckiness of colorant dispersions

Textiles — Essais de solidité des teintures —

Partie Z11: Évaluation de l'uniformité des dispersions de colorants

Reference number
ISO 105-Z11:1998(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-Z11 was prepared by Technical Committee ISO/TC 38, *Textiles*, Subcommittee SC 1, *Tests for coloured textiles and colorants*.

ISO 105 was previously published in 13 "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.

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Textiles — Tests for colour fastness —

Part Z11:
Evaluation of speckiness of colorant dispersions

1 Scope

This International Standard describes a test method to determine speckiness primarily of disperse dye, vat dye and pigment dispersions.

Agglomerates in colorant dispersions may become apparent as specks on a continuously dyed (padded), or on a printed fabric, especially when pale and light shades are produced.

2 Normative reference

The following standard contains provisions which, through reference in this text constitutes provisions of this part of ISO 105. At the time of publication, the edition indicated was 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 edition of the standard listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 105-A01:1994, *Textiles — Tests for colour fastness — Part A01: General principles of testing.*

3 Definitions

For the purposes of this International Standard the following definitions apply.

3.1 disperse dye: non-ionic dye which is sparingly soluble in water and which has substantivity, when properly dispersed, for polyester, polyamide and some other manufactured polymeric fibres.

3.2 dispersion: In textile dyeing and printing, a suspension of very fine colorant particles in a liquid phase.

3.3 pigment: colorant in particulate form which is insoluble in the substrate but which can be dispersed in the substrate to modify its colour.

3.4 speck: small particle, such as an agglomerate in liquid dispersion, or a very small spot of dark colour on a dyed substrate.

3.5 speckiness: In textile dyeing and printing, the quality or state of containing specks.

3.6 vat dye: water-insoluble dye, usually containing keto groups, which is normally applied to the fibre from an alkaline aqueous solution of the reduced enol (leuco) form which is subsequently oxidized in the fibre to the insoluble form.

4 Principle

A dispersion of a dye is filtered through a polyester/cotton blend fabric, then dried and thermofixed.

The degree of speckiness is assessed visually.

5 Safety precautions

Good laboratory practices should be followed. Wear safety glasses in all laboratory areas.

All chemicals should be handled with care

Use heat resistant gloves when working at hot air ovens and thermofixation devices.

Users should also comply with any national and local safety regulations.

NOTE:These safety precautions are for information purposes only. The precautions are ancillary to the test procedure and are not intended to be all-inclusive. It is the user's responsibility to use safe and proper techniques in handling materials in this test method. Consult manufacturers for specific details such as material safety data sheets and other manufacturer's recommendations.

6 Reagents

6.1 Sodium hydroxide, 30 % (m/V) $\widehat{=}$ 300 g/l.

6.2 Sodium hydrosulfite, powder ($Na_2S_2O_4$).

6.3 Hydrogen peroxide, 30 % (m/V) $\widehat{=}$ 300 g/l.

6.4 Acetic acid, 80 % (m/V) $\widehat{=}$ 800 g/l.

6.5 Grade 3 water, (see 8.1 of ISO 105-A01:1994).

7 Apparatus

7.1 Filter cloth, 65/35 (m/m) polyester/cotton, bleached woven broad cloth (shirting weight, approximately 100 g/m^2), approximately 240 mm x 240 mm square. A fabric of similar construction and of varying blend ratios may be used, but it shall then be stated in the test report.

NOTE The following fabric has been found to be suitable.

— Warp and weft/filling yarns: approximately 12 tex.

— Construction: warp approximately 50 ends/cm

weft/filling approximately 28 picks/cm

— The fabric is desized, bleached, dried and framed to 150 cm to 155 cm and heat set at 200 °C. These processes are carried out in accordance with generally accepted industrial practice.

7.2 Buchner funnel, polypropylene, 110 mm diameter, with separate top and bottom. Cut out the perforated bottom smoothly and even with the rim, using a knife or other suitable implement. Both parts are used in the test.

7.3 Filtering flask, 2 l, heavy wall with side tabulation.

7.4 Rubber stopper, 1 hole, to fit the filtering flask.

7.5 Stirrer, small propeller type, diameter approximately 20 mm, speed up to 2 200 rpm (37 s^{-1}).

7.6 Evaporating dishes, 1 l, glass or porcelain, (approximately 150 mm diameter), three, for testing vat dyes.

7.7 Ovens for the following operations:

— Drying, without air circulation;

— Thermofixation.

8 Preparation of liquid dispersion for sampling

Before removing a sample for testing, mix dispersions thoroughly in drums using a mechanical mixing device, such as a propeller stirrer or homogenizer, so that the liquid is smooth and free of sediment and lumps. Shake laboratory samples thoroughly to ensure that all dried material adhering to the cover and sides of the container is reincorporated into the liquid. Then remove the cover and stir the sample either mechanically, or by hand, until all sediment and/or lumps are completely redispersed. Replace the cover and once again shake to ensure complete homogeneity. Once the test sample has been removed for use and the balance of the dispersion is to be stored for future use, clean the lid and the container's lip thoroughly prior to closure. Test the sample immediately after homogenization.

NOTE On prolonged storage, liquid dispersions tend to settle and may develop a more or less tacky sediment. It is imperative to ensure complete homogeneity of the dispersion prior to testing.

9 Procedure

Identify the filter cloth with the experiment or sample number in one of the corners and ensure that it is free of extraneous specks.

Clean and dry the funnel. Prepare the funnel assembly by placing the top part of the funnel upside down on a clean surface and drape the fabric over it as flat as possible with the identification mark facing down towards the surface. The side of the fabric with the identification will then be on top during filtration and is used for subsequent evaluation. Snap and press the bottom half of the funnel to the top half, causing the fabric to become a tight, smooth filter in the funnel.

Place the assembled funnel straight on the filtering flask using the rubber stopper to ensure that the funnel remains upright (and the filter fabric horizontal) during filtration and rinsing.

Weigh the powder or liquid dye into a weighing cup and transfer to a 400 ml graduated beaker containing grade 3 water at 20 °C to 30 °C. Rinse the weighing cup with water from a spray bottle. Record the quantity of dye used.

NOTE The amount of dye used is directly proportional to its dyeing strength and is typically (2,5 ± 0,025) g for a very high strength solid brand to (20 ± 0,2) g for a low strength liquid. As a reference (7,5 ± 0,075) g of a dye is used if it gives a 1/1 standard depth of shade from 2 % (on mass of fibre) of dye applied (in an exhaust dyeing process). The masses of other dyes used in either liquid or solid forms are proportional to this reference.

With powder and granular forms, stir for 3 min with a small propeller stirrer with the propeller in the centre and just above the container bottom. Adjust the speed to create a vortex terminating at the top of the propeller.

With liquid forms, stir as for powders, but for 30 s.

After the specified stirring time, transfer the dispersion to the 1 l beaker. Rinse the 400 ml beaker with 200 ml of water (6.5) at 20 °C to 30 °C and pour the rinse water into the 1 l beaker. Dilute the dispersion further to 800 ml.

Pre-wet the fabric in the filter with 200 ml water (6.5).

Stir the dispersion in the beaker for approximately 30 s and pour it into the funnel.

Rinse the beaker with 200 ml water and pour it into the funnel.

Rinse the funnel with an additional 200 ml water and let stand for about 1 min until dripping has completely stopped.

Carefully remove the filter fabric from the funnel and place it on a blotting paper to remove excess water.

Optionally, when the colorant under test is a vat dye, the filter fabric may be further treated, at this stage, in an evaporating dish and reducing solution followed by oxidation using the following procedure.

a) Prepare 400 ml of a fresh reducing solution in an evaporating dish at 60 °C to 70 °C containing:

   1) 30 ml/l sodium hydroxide (6.1);

   2) 20 g/l sodium hydrosulfite (6.2).

Fully immerse the filter fabric in the reducing solution in the evaporating dish for 5 min. Do not move the fabric during this time.

b) Immerse the filter fabric for 1 min, without agitation in water (6.5) at 15 °C to 25 °C in an evaporating dish.

c) Prepare 100 ml of an oxidizing solution in an evaporating dish at 40 °C to 50 °C containing 10 ml/l hydrogen peroxide (6.3). Fully immerse the filter fabric in the oxidizing solution in the evaporating dish for 2 min. Do not move the fabric during this time.

NOTE Evaporating dishes are used for reduction and oxidation of vat dyes to keep the fabric flat and without agitation, which prevents any specks from dissolving and levelling.

d) Neutralize the filter fabric for 2 min at 15 °C to 25 °C using 200 ml/l acetic acid (6.4) in a 400 ml beaker. Rinse for approximately 30 s in cold running water.

Dry the fabric in an oven without air circulation at (80 ± 5) °C.

For disperse dyes thermofix for 60 s at 210 °C to 220 °C.

NOTE The interested parties may agree a speckiness rating under which the sample tested may be rated as acceptable, marginal or unacceptable .

10 Evaluation

Visually examine the fabric for specks on the side with the identification mark. Count individual specks.

11 Test report

The test report shall include the following information.

a) reference to this part of ISO 105, i.e. ISO 105-Z11:1998;

b) the dyestuff tested and mass used;

c) the number of specks;

d) where a vat dye is evaluated state whether reduction or oxidation, as defined in paragraph 13 of clause 9, has been carried out.

NOTE A precision statement is not applicable, because data are not generated by this test method. The number of specks detected in 12 tests run over several days on a single sample varied from a high of 56 to a low of 23.