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Textiles — Tests for colour fastness —
Part Z04:
Dispersibility of disperse dyes

Textiles — Essais de solidité des teintures —
Partie Z04: Dispersibilité des colorants de dispersion



Reference number
ISO 105-Z04:1995(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-Z04 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.

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Textiles — Tests for colour fastness —

Part Z04:

Dispersibility of disperse dyes

1 Scope

This part of ISO 105 describes a method for determining the dispersibility, as evaluated by filtering time and filter residue, of disperse dyes.

This test method is used for determining the degree of dispersion under specified conditions in aqueous media only.

NOTES

1 The results of this test method can vary widely unless all tests are run under exactly the conditions outlined. Any variation in the conditions can cause a test to be invalid. Results have been shown to be reproducible in several laboratories when the specified conditions are met.

2 Variations in results can be caused by differences in the diameter of the filter funnel, due to the differences in surface area which result, and to variations in the size and density of the holes in the filter funnel.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute 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 indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3696:1987, *Water for analytical laboratory use*.

3 Definition

For the purposes of this part of ISO 105, the following definition applies.

3.1 dispersibility: Degree to which particles can be broken down to some minimum size such that they will pass through the interstices of a reference filter paper.

4 Principle

A quantity of disperse dye is predispersed, heated and passed through filter papers of specified pore size. Using the time of passage and retention of dye on the filter paper, the dispersibility of the dye is then evaluated.

Three variants of the test, depending on the intended dye application, are outlined.

5 Safety precautions

5.1 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 recommendations.

5.2 Good laboratory practice should be followed. Wear safety glasses in all laboratory areas and a single-use dust respirator while handling powder dyes.

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

6 Apparatus and materials

During the test, use only reagents of recognized analytical grade and only grade 3 water as defined in ISO 3696.

6.1 Nutsch filter (Büchner funnel), of glass, stainless steel or porcelain, with inner diameter 110 mm, 192 holes and with a total surface area of holes (evenly distributed) of not less than 200 mm².

6.2 Filter papers¹⁾, of diameter 110 mm, as follows.

- Type A: Having a pore size to retain particles above 8 µm in diameter;
- Type B: Having a pore size to retain particles above 25 µm in diameter.

Other papers may be used if quality and microretention characteristics are exactly the same.

6.3 Stainless steel ring¹⁾, with approximate dimensions 103 mm i.d., 111 mm o.d. and 8 mm thickness, to hold filter paper of 110 mm diameter in place.

6.4 Filter flask, with side tube, of capacity 1 000 ml.

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

6.6 Apparatus to adjust and maintain the vacuum, including vacuum rubber tubing, preferably coupled with a manometer.

6.7 Stopwatch.

6.8 Beakers, of capacity 400 ml or larger.

6.9 Balance, analytical.

6.10 Tetrasodium pyrophosphate (TSPP), 10 % (m/V) (100 g/l) aqueous solution prepared with grade 3 water complying with ISO 3696.

6.11 Acetic acid, 10 % (V/V) (mass fraction 10 %) aqueous solution prepared with grade 3 water complying with ISO 3696.

6.12 Filter-residue scale²⁾, consisting of photographs representing five classes (levels) of retention of filter residue and used as part of the evaluation procedure.

6.13 Water, complying with grade 3 of ISO 3696, optionally containing a complex-forming agent [e.g. 0,25 g/l of 25 % (m/V) (250 g/l) solution of ethylenediaminetetraacetic acid (EDTA) or nitrilotetraacetic acid (NTA)]. Such addition shall be mentioned in the test report.

6.14 Stirrer, laboratory, magnetic.

6.15 Graduated cylinder, 250 ml capacity.

6.16 pH-meter.

7 Procedure

7.1 Selection of test

Select the test (I, II or III) to be used according to the intended application of the dye (see table 1).

Table 1 — Selection of applicable test

| Test | Application of dye | Filter paper combination | pH of dispersion |
|------|--|--------------------------|------------------|
| I | Where dispersion requirements are critical (such as package dyeing of polyester) | Type A over Type B | 4,5 to 5,0 |
| II | For dyeing at larger liquor ratios (such as beck dyeing) | Type B over Type B | 4,5 to 5,0 |
| III | For dyeing on polyamide (carpet and apparel dyeings) | Type B over Type B | 9,0 to 10,0 |

7.2 Preparation of dispersion

Using the analytical balance (6.9), weigh out 2,0 g ± 0,1 g of dye powder, to be tested.

Weigh out an equivalent quantity of the manufacturer's reference dye, and carry out the same test on this dye. The reference dye is always run as a control so that the influence of any test variables on the results is minimized.

1) For information on the source of supply of sets of filter papers (6.2), and stainless steel rings (6.3), apply to the organizations listed in clause 8 of ISO 105-A01:1994, *Textiles — Tests for colour fastness — Part A01: General principles of testing*.

2) This scale is available from the American Association of Textile Chemists and Colorists (AATCC), P.O. Box 12215, Research Triangle Park, NC 27709, USA.

If required, on dyes of high tinctorial strength, reduce the dye concentration to 1 g/200 ml and report this fact in the test report.

Add the weighed dye slowly to 200 ml of vigorously agitated water (6.13) at $45\text{ °C} \pm 2\text{ °C}$ in a 400 ml beaker (6.8). Use a magnetic stirrer (6.14). Adjust the pH with the pH-meter (6.16) as follows:

- for Tests I and II: pH 4,5 to pH 5,0 using acetic acid (6.11);
- for Test III: pH 9,0 to pH 10,0 using TSPP (6.10).

Heat to $70\text{ °C} \pm 2\text{ °C}$ and hold at this temperature for $5\text{ min} \pm 1\text{ min}$. Stir with the magnetic stirrer to prevent localized heating.

NOTE 3 Excessive time lags and holding temperatures may affect the outcome of the test.

Optionally, disperse and stir the dye at $25\text{ °C} \pm 2\text{ °C}$. In this case, no preheating of the Büchner funnel (see 7.3) is necessary. If this option is used, it shall be stated in the test report.

7.3 Filtration of dispersion

Heat 100 ml to 300 ml of water to 70 °C and pour into the Büchner funnel (6.1) without any filter paper in place, to preheat the funnel.

Wait $25\text{ s} \pm 10\text{ s}$. Turn the vacuum on (6.4 to 6.6) and keep on until the water has passed through the funnel. Turn the vacuum off, immediately dry the funnel and then place the correct filter papers (6.2) in the funnel (see table 1).

Two filter papers are used to give a more uniform vacuum across the filtering surface. The filter paper is used as received in the box, with the smooth side up to avoid variations due to texture differences on the rough side. If different types of filter paper are used, the coarser paper shall be underneath.

Insert the stainless steel ring (6.3) in the funnel on top of the filter papers, and turn on the vacuum. Adjust the vacuum to 3 kPa to 4 kPa, which is equivalent to a 300 mm to 400 mm water column under pressure.

NOTE 4 Since filter paper is cellulosic in nature, it swells readily when wet. Therefore, if the paper is prewetted to hold it in place in the funnel, the filtration time will be increased, this increase depending on the temperature of the wet filter paper and the time which has been allowed to elapse since wetting. Thus it is imperative that the filter paper is not wet prior to the test, since the microretention characteristics of the paper will be reduced. This is the reason for using the stainless steel ring, which holds the filter paper in place, making prewetting unnecessary. With dyes which have a very small particle size, the speed of filtration of the dispersion will not be changed to any great extent by changing the vacuum. However, with dyes of larger particle size, reducing the vacuum can result in shorter filtration times, even though the suction is not as great. This is because,

with an increased vacuum, dyes of larger particle size will block the filter paper, slowing the filtration rate and thus swelling the paper more. This results in longer filtration times and more dye being retained on the filter paper.

With the vacuum on, immediately pour the dye dispersion into the funnel and begin timing. Record the time of passage of the dispersion, up to 120 s to the nearest second. The end point is reached when the appearance of the filter paper changes from a wet look to a dry look.

Allow the filter papers to dry and evaluate as outlined in clause 8.

NOTE 5 In order to give more reproducible results, especially with dyes of residue Class 3 or lower, it may be advisable to rinse the filter paper while it is in the funnel with 10 ml to 15 ml of water (6.13) before drying. This will aid in the removal of any colloidal dye dispersion that actually has a particle size smaller than pore size of the paper being employed.

Complete the test from the start of dispersion to the end of filtration within 15 min.

8 Classification of dye dispersibility

Using the time of passage and the retention of dye on the filter paper, the dispersibility of the dye is evaluated.

8.1 From the recorded time of passage through the filter paper, classify the dispersibility of the dye as follows:

| | |
|---------|----------------|
| Class A | 0 s to 24 s |
| Class B | 25 s to 49 s |
| Class C | 50 s to 74 s |
| Class D | 75 s to 120 s |
| Class E | 120 s and over |

8.2 Compare the residue on the filter paper against the filter-residue scale (6.12). Also examine the paper for any visible coarse or granular particles. If particles are present, the dye is automatically rated as Class 1 (poor dispersibility). If no coarse residue is present, classify the dye as follows:

| | |
|---------|--------------------------|
| Class 5 | Excellent dispersibility |
| Class 4 | |
| Class 3 | |
| Class 2 | |
| Class 1 | Poor dispersibility |

Intermediate values in comparison with the filter residue scale are interpolated.

9 Test report

The test report shall include the following information:

- a) the number and year of publication of this part of ISO 105, i.e. ISO 105-Z04:1995;
- b) description of the sample tested;
- c) the test number (see table 1);
- d) whether or not the optional test temperature (25 °C) was used;

- e) the class according to filtration time (see 8.1);
- f) the class according to amount of residue (see 8.2).

EXAMPLE

A dyestuff tested using Test I, giving a filtration time of 17 s and a filter paper residue rated as Class 3 in comparison with the filter-residue scale is classified in accordance with this part of ISO 105 as having a dispersibility of I-A-3.

ICS 59.080.10

Descriptors: textiles, dyeing, colour fastness, dyes, tests, colour-fastness tests, determination, dispersibility.

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