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Textiles — Tests for colour fastness —

Part Z10:
Determination of relative colour strength of dyes in solution

Textiles — Essais de solidité des teintures —

Partie Z10: Détermination de l'intensité relative de la couleur des colorants en solution

Reference number
ISO 105-Z10:1997(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-Z10 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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INTRODUCTION

This part of ISO 105 describes a practicable method for determination of colour strength based on absorbance measurement of dye solutions. The relative colour strength is defined primarily by means of comparison of dyeings, which are prepared from the dye under test and the reference dye at the same depth. The relative colour strength is therefore the reciprocal ratio of the dye concentrations, expressed as a percentage, hence it is a value defined by means of an application technique and is associated with the conditions of preparation of the dyeings and their evaluation. Despite this general limitation, determination of colour strength in solution is useful because of the decisive advantage of higher accuracy (reproducibility) with little expenditure of effort. The validity of the result should be controlled by comparison with evaluation by means of an application method.

Textiles — Tests for colour fastness —

Part Z10:

Determination of relative colour strength of dyes in solution

1 Scope

This method is intended for the determination of the colour strength of a dye in relation to that of a reference dye by means of spectrophotometric absorption measurements on solutions of dyes.

NOTES

1 Basic requirements for this method are that the dye solutions do not scatter light and obey the Bougher-Lambert-Beer law as well as identical or similar absorption curves of the samples and the reference in the visible region of the spectrum.

2 Identical or similar absorption curves are usually obtained if the tests are carried out for the purpose of dye production control of batches/deliveries of the same dye. This method is not applicable for the evaluation of dyes with distinctly different adsorption curves.

3 The colour strength of a dye is not a physical constant, since it is dependent, for example, on the test medium and test method. The colour strength determined by this method therefore may differ from those found by other determinations, e.g. instrumental or visual assessments of dyeings.

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 648:1977, *Laboratory glassware — One-mark pipettes.*

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks.*

3 Definitions

For the purposes of this part of ISO 105 the following definitions apply :

3.1 colour strength: A measure of the ability of a dye to impart colour to other materials characterized by its absorption in the visible region of the spectrum and expressed as a colour strength value.

3.2 relative colour strength: The percentage ratio of the colour strength value of the sample to that of the reference dye, the colour strength of which is 100 %.

NOTE — As the relative colour strength is determined by a comparison with a reference dye, this is assumed to remain constant. It is essential therefore to ensure careful storage and control of the reference dye. Many dyes are hygroscopic and sensitive to oxidation. It is essential to protect the reference dye from exposure to light in firmly sealed containers that are impervious to moisture.

3.3 colour strength value: The extinction coefficient at the maximum absorption of the reference dye.

NOTE — Colour strength is traditionally a colouristic concept, being based essentially on a visual assessment. Therefore colour strength determined instrumentally should not contradict visual assessment. Such a contradiction would not be expected if the samples to be compared exhibit concentration-dependent differences in absorption only i.e. their absorption curves in the visible region are identical when the strength of the absorption curves is made equal, or show only insignificant differences.

If the absorption curves differ more markedly (shade differences), a better correlation with visual assessment may be obtained if the weighted total extinction in the visible region is used as the colour strength value.

4 Principle

Solutions of the dye under test and the reference dye are prepared and the absorptivity or colour strength values derived from them are determined.

The percentage relative colour strength F_s, is given by the following equation :

$$F_s = \frac{f_{k2}}{f_{k1}} \times 100$$

where

f_{k1} is the colour strength value of reference dye;
f_{k2} is the colour strength value of dye under test.

5 Apparatus

5.1 Flasks, in accordance with ISO 1042.

5.2 Pipettes, in accordance with ISO 648.

5.3 Cells.

5.4 Cuvettes or flow cells.

5.5 Analytical balance.

5.6 Spectrophotometer or filter colorimeter.

6 Solvents

WARNING — This part of ISO 105 calls for the use of substances and/or procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage. It has been assumed in the drafting of this part of ISO 105 that the execution of its provisions is entrusted to appropriately qualified and experienced people.

NOTE — When selecting solvents the following aspects are important :
solubility of dye;
stability of solutions;
reproducibility of test;
applicability of result to other media or practical applications.

The solvent used shall be selected from the following :

(a) for water soluble dyes : water (demineralized)

(b) for water insoluble dyes :
methanol, ethanol;
acetone;
N-methylpyrrolidone;
cellosolve, ethylene glycol monoethylether;
mixtures of the above solvents.

Dilute the concentrated stock solution (see 7.1.3 and 7.1.4) with the same solvents. Dissolve the stock dispersion in organic solvents and then dilute. The following additives are recommended to improve the stability and reproducibility.

(a) To adjust the pH :
acids (e.g. sulphuric acid, acetic acid);
bases (e.g. sodium hydroxide, sodium carbonate);
buffer systems.

(b) To eliminate the influence of unwanted metal ions :
sequestering agents.

(c) Agents with a deagglomerating or disaggregating action in water:
surfactants, generally ethylene oxide adducts to alkanols.

(d) Other additives:
dispersing agents;
antioxidants.

7 Procedure

7.1 Preparation of solutions for measurement

7.1.1 Samples

Store the samples in firmly sealed containers in order to avoid erroneous results arising from the release or uptake of moisture.

7.1.2 Weighing

Weigh an amount of not less than 0,5 g, to avoid errors attributable to micro-inhomogeneities to the nearest 0,000 5 g.

NOTE — It is essential to ensure that uptake of moisture by hygroscopic dye samples does not lead to errors during weighing.

7.1.3 Dissolving/dispersing

Transfer the weighed dye quantitatively to a graduated flask and then dissolve/disperse it. In general, pre-dissolving/dispersing in about 20 ml of the dissolving/dispersing medium or an additional solvent or auxiliary is advantageous. Water soluble dyes are preferably dissolved hot.

NOTE — It is essential to ensure that the solubility limit is not exceeded during subsequent cooling.

Equilibrate the solution to ambient temperature (20 °C to 25 °C). Make up the graduated flasks to the mark and homogenize the solution/dispersion by stirring or by mixing by turning the flask end over end.

7.1.4 Dilution

Dilute or dissolve the solution/dispersion prepared according to 7.1.3 so that the maximum absorption (the minimum transmittant at the lowest point) lies within the range of 10 % to 60 % transmittance.

NOTES

1 To avoid errors of dilution no pipette of total volume less than 5 ml nor graduated flask less than 100 ml should be used.

2 For commercial dyes the corresponding concentrations for measurement are within the range 10 mg/l to 100 mg/l for a 10 mm cell path length or proportionately more for shorter path length cells.

7.2 Measurement and evaluation

Carry out the measurement as soon as possible after preparation of the solution, in order to exclude the influence of changes in solution.

NOTE — When a dye is measured for the first time it is advisable to confirm the validity of the relationship (Beer's law) under the conditions selected (concentration, solvent) within a range of concentrations between one-half and twice that actually used.

Adopt appropriate techniques with light sensitive solutions e.g. using darkened flasks, or working in dimly lit surroundings.

NOTE — In certain instances the solution to be measured may be influenced adversely by the light energy of the source in the measuring instrument.

7.2.1 Measurement

Measure the absorbance of the solution in cells of identical path length (usually 5 mm or 10 mm). The difference in absorbance between the sample and the reference shall not exceed 30 % in order to minimize the influence of variations in the spectrophotometric response over a relatively wide range of concentrations. Depending on the measuring instrument used, the absorbance, A, of the dye solution is obtained directly as a measured result (double-beam spectrophotometer with simultaneous measurement of an identical cell filled with the solvent used for dilution, or a single beam instrument previously calibrated with a cell containing solvent), or is calculated as the difference between the absorbance of the dye solution A_R, and a blank value A_B (measurement of the solvent).

$$A = A_R - A_B$$

7.2.2 Evaluation

Base the evaluation on Beer's law, using the following equation :

$$A = \log_{10}(1/t) = \varepsilon c d$$

where

A is the absorbance;
t is the transmittance (decimal fraction, 0 to 1,0);
ε is the specific extinction coefficient, in square centimetres per gram;
c is the dye concentration, in grams per cubic centimetre;
d is the cell path length, in centimetres.

7.2.3 Relative colour strength

The relative colour strength is given by the following equation :

$$F_S = \varepsilon_2/\varepsilon_1 \times 100$$

or by substituting from the above equation :

$$F_s = \frac{A_2 C_1}{A_1 C_2} \times 100$$

where subscript 1 refers to the reference dye and subscript 2 refers to the dye under test.

If the measured concentrations are equal the following simplified equation is obtained :

$$F_s = \frac{A_2}{A_1} \times 100$$

If other colour strength values are used, they are dealt with accordingly.

8 Precision

The coefficient of variation for determinations under the same conditions (repeated conditions) is approximately 0,2 % to 0,5 %. Hence, this gives about 0,4 % to 1,0 % for the uncertainty of an individual determination with a confidence level of 95 %. For the uncertainty of determination under different conditions (various testing locations), at least double these values should be assumed.

9 Test report

The test report shall contain the following information:

(a) reference to this Part of ISO 105, i.e. ISO 105-Z10:1997;

(b) the type and designation of the sample tested and the reference dye;

(c) the relative colour strength.

NOTE — A full test report, as required for example in cases of dispute, may contain the following additional details :

(a) date of test;
(b) dye mass;
(c) solvent used;
(d) dissolving conditions, (e.g. concentration, temperature);
(e) dilution factor;
(f) concentration and temperature of measured solution;
(g) measuring instrument, i.e. type and band width;
(h) cell path length;
(i) wavelength;
(j) colour strength value.

ANNEX A

(informative)

Background information

The purpose of this part of ISO 105 is the description of a simple practicable determination of colour strength based on extinction measurement of dye solutions. Instrumental methods of this kind have been established for a long time and there is much practical experience of this. Amongst relevant publications those of the Inter-Society Color Council (ISCC) should be noted.

(a) *General procedure for the determination of relative dye strength by spectrophotometric transmittance measurement* (report of the ISCC):
KUEHNI R.G., Text. Chem. Colorist 4 (1972) 133.

(b) *Difficulties in preparing dye solutions for accurate strength measurement* :
COMMERFORD T.R. Text. Chem. Colorist, 6 (1974) 14.

(c) *Reproducibility of dye strength evaluation by spectrophotometric transmission measurement* (report of the ISCC: SWEENY C.D., Text. Chem. Colorist, 8 (1976) 31.

The relative colour strength is defined primarily by means of a comparison of dyeings, which are prepared from the dye under test and the reference dye at the same depth. The relative colour strength is thus the reciprocal ratio of dye concentrations, expressed as a percentage, with the reference dye set at 100 %. Hence it is a value defined by means of an application technique and is not a physical constant of the dye.

According to this general definition, a colour strength value is valid only within very narrow limits. It is associated with the conditions of preparation of the dyeings (substrate, dyeing conditions, concentration) and their evaluation. In the past the assessment of depth of colour has been mainly carried out by visual methods and has thus been strongly subjective. This was especially so with differences of tone, which often were also present. Furthermore, the increasingly applied instrumental methods (reflectance measurements) have not hitherto been able to eliminate the uncertainty of evaluation (resulting from shade differences), although the criteria of assessment have been modified, in order to attain as close a correlation as possible with visual evaluation. Thus the uncertainties of a 'correct' colour strength determination are still present.

In spite of these general limitations, it is in many cases convenient to refer to a single colour strength, particularly if the sensitivity to application conditions is relatively low and if solution measurement leads to comparable results. Hence colour strength determination in solution is useful because of the decisive advantage of higher accuracy (reproducibility) with little expenditure of effort. The validity of the result should be controlled by comparison with evaluation by means of an application method. If this is done for one individual dye and comparability is confirmed, the method can usually be applied successfully. Nevertheless in all cases a critical consideration of the result in relation to this comparability is necessary.

It is generally true that for dyes that are fully adsorbed by the substrate during the dyeing process, a good comparability of results is obtained. This is mainly the case with acid, basic and disperse dyes. On the other hand, care is necessary with dyes showing incomplete exhaustion or fixation, e.g. direct or reactive dyes, especially with changes in formulation. In general the method is not limited to certain dyes/dye classes or substrates.

The comparability of the results is an important consideration for the selection of the solvent or additives. Experience has shown that there is often no specific dependence on solvent, so that other considerations, e.g. toxicological properties, may also be taken into account.

The following comments may be made regarding reproducibility. The coefficient of variation for determinations under the same conditions (repeated conditions) is about 0,2 % to 0,5 %. Hence this gives about 0,4 % to 1,0 % for the uncertainty of an individual determination with a confidence level of 95 %, which can be reduced by repeat measurements (n repeats) according to the factor $1/n^{\frac{1}{2}}$. For the uncertainty of determination under different conditions (various testing locations), at least double these values must be assumed.

The colour strength value acquires a special significance for the comparison of dyes with different spectral absorption curves. Since the colour strength is associated with visual assessment, which cannot be described precisely and unambiguously by colorimetry, an evaluation at the absorption maximum is often not applicable for different shades, because the visual perception is also influenced by the absorption in the remainder of the visible spectrum. This is true primarily for the evaluation of a dyed substrate but by analogy also for dye solutions (with corresponding differences in the absorption curves) so that by taking into account these differences in absorption a better correlation with visual assessment can be achieved.

Thus if the total absorption in the visible region of the spectrum is used, a weighting corresponding to the colour matching functions x, y and z is normally applicable. To date, however, limited experience exists, regarding the advantages and disadvantages of this weighting. Effects of this kind are most evident in yellow hues, which can yield markedly different results with relatively minor tonal differences, depending on the criteria of assessment.

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