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INTERNATIONAL STANDARD ISO 4312

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Surface active agents — Evaluation of certain effects of laundering — Methods of analysis and test for unsoiled cotton control cloth

Agents de surface — Contrôle de certains effets de blanchissage — Méthodes d'analyse et d'essai d'un tissu de coton témoin non souillé

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Surface active agents – Evaluation of certain effects of laundering – Methods of analysis and test for unsoiled cotton control cloth

0 INTRODUCTION

It will be recalled that some effects produced by laundering on textiles can be evaluated by means of test pieces of unsoiled cotton control cloth which are washed together with normally soiled textile articles. It is impossible to effect this evaluation with normally soiled textile articles themselves since these are subject to damage and to modification due to actual use between successive launderings and which cannot be controlled.

This damage and these modifications are not part of the effects of laundering and, furthermore, their extreme variability would make the measurements very difficult to reproduce and even harder to compare between one laboratory and another. In addition, it is hardly ever possible to operate with normally soiled textiles having exactly standardized properties.

The use of the test pieces of strictly defined unsoiled cotton control cloth enables the causes of variations which are extraneous to the laundering process itself to be largely eliminated. For this reason, the use of test pieces of cotton control cloth has been recognized as essential for verifying the laundering processes employed by industrial laundries.

Conclusions based on the behaviour of the unsoiled control cloth cannot be used to predict that of other textiles laundered in the same way, if these differ too much from the control cloth with regard to the nature of the fibres, the yarn linear density, the mass per unit surface, the presence of finishes or the initial degree of damage. In such cases, the results obtained on the control cloth can at most show qualitative differences between different laundering processes or variations of a process.

When the unsoiled cotton control cloth is used to determine the influence of a single factor (for example the type of washing machine, the nature or concentration of the detergent, the nature or concentration of the bleaching agent) from the point of view of the effects produced on the textiles, the comparisons are only valid, of course, so long as all the other factors are kept constant. In particular, care should be taken to see that the test pieces of cotton control cloth to be used come from the same consignment, that the normally soiled textiles are always of the same kind and have a uniform degree of soiling, and that the hardness of the water is always the same (so long as it is not precisely the influence of the hardness that is to be studied).

It follows from this that it is advisable in practice to compare the results obtained in different laboratories with one another only after a preliminary study has provided an assurance that all the conditions for comparison have been met.

In particular cases, it may be decided to determine only certain of the characteristics from among those the determination of which is described in this International Standard.

This International Standard should be read in conjunction with ISO 2267.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the methods to be used to determine, under strictly controlled conditions, some characteristics of unsoiled cotton control cloth, namely intrinsic greying and yellowing, increase in organic depositions content and incineration residue, determination of overall loss of breaking strength, of loss of breaking strength resulting from chemical degradation of cellulose and of loss of breaking strength resulting from mechanical factors in laundering both before and after processing, so that certain effects of laundering can be evaluated.

For the purpose of the routine assessment of the effects of processing on cotton textile articles in commercial laundries, the application of some of the methods only may be appropriate. Furthermore, while the methods given in this International Standard evaluate damage caused by excessive mechanical action, they do not distinguish between the effects of smaller differences in mechanical action on cleansing.

2 REFERENCES

ISO 139, *Textiles – Standard atmospheres for conditioning and testing.*

ISO/R 1628, *Plastics – Directives for the standardization of methods for the determination of the dilute solution viscosity of polymers.*

ISO 2267, *Surface active agents – Evaluation of certain effects of laundering – Preparation and use of unsoiled cotton control cloth.*

4 The degree of visible whiteness of white textiles is, of course, one of the factors that determine the quality of the laundering.

However, standardization of the measurement of the degree of visible whiteness is not sufficiently advanced to permit the inclusion of a method in this International Standard.

Annex A describes an empirical method which satisfies the majority of cases met with in practice.

4.3 Principle

Measurement of the reflectometer value (R_g) of the cotton control cloth before and after the 25 (or 50) laundering cycles, by means of a reflectometer (photoelectric colorimeter) fitted with a Y (green) tristimulus filter and under operating conditions selected so as to eliminate any fluorescent effect.

The decrease in the reflectometer value (perfect diffuser = 100), which is identical in this case to the decrease in the percentage luminance, is a measure of the intrinsic greying.

4.4 Apparatus

4.4.1 Reflectometer (photoelectric colorimeter), capable of being fitted with a Y (green) tristimulus filter which, in conjunction with the photoelectric cell and light source, affords a spectral sensitivity corresponding to the CIE colour-matching function $\bar{y}$ for the C source and enabling any fluorescent effects to be excluded (see note 2 following 4.2.5).

NOTES

1 There are two possible methods that may be used to eliminate fluorescence. The first consists in placing the Y (green) tristimulus filter between the light source and the cloth to be examined. This prevents the ultra-violet and violet light required to excite fluorescence from reaching the cloth. The second consists in illuminating the cloth with white light, but using a filter which blocks the ultra-violet rays between the light source and the cloth; the Y (green) tristimulus filter is placed between the cloth and the photoelectric cell. The U.V. filter should block all radiations below 450 nm.

2 In theory, it is possible to obtain an identical result by calculations based on the spectral reflectance curve determined by means of a spectrophotometer. In this case, the data obtained from the spectrophotometric curve must be multiplied by the tristimulus values $(\bar{y}H)\lambda$ for standard source C and the products integrated over the whole of the spectrum. In this case also, any fluorescence must, of course, be eliminated.

4.4.2 Two calibration plates, for calibrating the reflectometer (4.4.1), one of neutral white with a percentage luminance between 85 and 90 and the other of neutral grey with a percentage luminance between 60 and 75. The calibration of these plates should be based on the perfect diffuser (= 100) for the Y (green) and Z (blue) tristimulus filters, under the measuring conditions of the reflectometer used.

Calibration plates in various materials are obtainable from various national calibration bodies. Bright materials are easier to maintain but dull materials give reflectometer values which are less dependent on the geometrical conditions of lighting and observation.

NOTE — The use of a single calibration plate around 100 % leads to errors if the reflectometer response is not strictly linear over the whole of the measuring range. It is thus essential to use at least two calibrated calibration plates.

4.5 Procedure

The procedure is based on the apparatus used. It should enable a correct measurement of the percentage luminance of the cloth to be obtained with the Y (green) tristimulus filter specified, excluding any fluorescence phenomena.

Carry out the measurements on the various test pieces of cotton control cloth at least 12 h and at most 7 days after final ironing (or pressing). During this period, keep the test piece in conditions of the strictest cleanliness and protected from the light.

For each individual measurement, fold the test piece under test in such a way as to give a thickness of eight layers with the uppermost layer, on which the measurement is made, corresponding to the surface of the cloth which has been in contact with the polished part of the press or ironing machines. If it is not possible to distinguish the two surfaces, for example on a tumbler-dried test piece, carry out half the measurements on one surface and half on the other. Carry out ten individual measurements in this manner at different points on each test piece.

Repeat the measurements on each test piece.

4.6 Expression of results

The intrinsic greying ΔG is given, as a percentage, by the formula

$$\Delta G = R_{g_0} - R_g$$

where

R_{g_0} is the arithmetic mean, expressed to one decimal place, of the reflectometer values for the Y (green) tristimulus filter (equal to the percentage luminance) recorded on the cotton control cloth;

R_g is the arithmetic mean, expressed to one decimal place, of 3 × 10 reflectometer values for the Y (green) tristimulus filter (equal to the percentage luminance) recorded on the three test pieces that have undergone the 25 (or 50) laundering cycles.

Express the result to one decimal place.

NOTE — The R_{g_0} value for the cotton control cloth is not necessarily the highest value that might be obtained for it as the laundering operations that will subsequently be applied to it could, in some cases, perfect the results of the preparatory treatment operations alone.

In certain cases, therefore, it is possible to record negative ΔG values.

There is no disadvantage in this so far as comparative tests are concerned.

If, on the other hand, it is desired to use ΔG as an absolute index for the quality of laundering, for example in checking the quality

5.6 Expression of results

The degree of intrinsic yellow J of the laundered test pieces is conventionally obtained from the formula

$$J = R_g - R_b$$

and the degree of intrinsic yellow J_0 of the cotton control cloth from the formula

$$J_0 = R_{g_0} - R_{b_0}$$

where

R_g and R_{g_0} are as defined in 4.6;

R_b is the arithmetic mean, expressed to one decimal place, of 3 × 10 reflectometer values for the Z (blue) tristimulus filter as recorded on the three test pieces that have undergone 25 (or 50) laundering cycles;

R_{b_0} is the arithmetic mean, expressed to one decimal place, of the reflectometer values for the Z (blue) tristimulus filter recorded on the cotton control cloth.

The intrinsic yellowing ΔJ, caused by the 25 (or 50) laundering cycles on the control cloth, is given by the formula

$$\Delta J = J_{25(50)} - J_0$$

Express the result to one decimal place.

When $\Delta J > 0$, the laundering has caused intrinsic yellowing. If $\Delta J = 0$, the laundering has not caused intrinsic yellowing. Finally, if $\Delta J < 0$, the laundering has either reduced the degree of intrinsic yellow in the cotton control cloth or caused a turn towards blue.

NOTE – The R_{b_0} value for the cotton control cloth is not necessarily the highest value that might be obtained for it as the laundering operations to which it will later be subjected could, in some cases, perfect the results given by the preparatory treatment operations alone.

In certain cases, it is therefore possible to record negative values for ΔJ.

There is no disadvantage in this so far as comparative tests are concerned.

If, on the other hand, it is desired to use ΔJ as an absolute index for the quality of laundering, for example in checking the quality achieved by commercial laundries, it would be preferable to establish once and for all a constant value for R_{b_0} equivalent to the highest values obtained for the cotton control cloth in the course of the various tests.

As this maximum value depends on the working conditions and the apparatus used in each laboratory, it is impossible to propose a universally acceptable value. For guidance, it may be stated that it usually lies between 86 and 90 %.

6 DETERMINATION OF INCREASE IN ORGANIC DEPOSITIONS CONTENT

6.1 Scope and field of application

This clause specifies a method for the determination of the increase in the content of organic matter insoluble in water in the unsoiled cotton control cloth, caused by the 25 (or 50) laundering cycles specified in ISO 2267.

These deposits, arising mainly from calcium and magnesium soaps, can cause various undesirable conditions : greying, yellowing, a greasy feel, more difficult wetting, an unpleasant smell, calendering difficulties.

6.2 Principle

Treatment of a known mass of cotton control cloth with a benzene/ethanol mixture before and after the 25 (or 50) laundering cycles. After extraction, evaporation of the solution of organic matter extracted and weighing of the dry residue.

6.3 Reagent

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

Extraction solvent, consisting of a mixture of 9 volumes of benzene and 1 volume of 96 % (*V/V*) ethanol.

6.4 Apparatus

Ordinary laboratory apparatus and

6.4.1 Soxhlet type extraction apparatus, having standardized ground joints, capable of being protected against the ingress of humidity from the atmosphere, and comprising the following items :

6.4.1.1 Receiving flask.

6.4.1.2 Extractor, of capacity between 150 and 250 ml.

6.4.1.3 Condenser, fitting both the receiving flask (6.4.1.1) and the extractor (6.4.1.2).

6.4.2 Weighing bottle.

6.4.3 Oven, capable of being controlled at 103 ± 2 °C.

6.4.4 Drying tube.

6.4.5 Water bath or flameless heater.

6.4.6 Vacuum pump.

6.5 Procedure

6.5.1 Test portion

Take a test portion of mass approximately 3 g, weighed to the nearest 0,000 1 g, from that remaining part of the test piece which is intended for measurement of loss of tensile strength and has been prepared in accordance with clause 6 of ISO 2267 (see also clause 3).

At the same time, take a second test portion for determining the water content of the cloth.

In the first case, the ash consists of depositions of mineral salts as such or having undergone certain chemical modifications as a result of calcination, and of the residue of incineration of organic salts. As this residue comprises only a quite small fraction of the organic depositions, in the majority of cases, the amount of total ash depends above all on the magnitude of the mineral depositions.

However, the amount of mineral ash is a more accurate index of the presence of mineral depositions. The latter derive from salts contained in the washing water or from detergents or from the reaction of both.

Depositions of mineral products on cloth indicate a defect in the detergent action; they may contribute to the premature rejection of the cloth by modifying its organoleptic qualities (harsh feel, dull appearance, and greying or yellowing) or by promoting chemical or mechanical damage.

7.2 Principle

After free combustion of the cotton control cloth in a tared crucible, incineration of the resulting ash in a muffle furnace at 800 °C, followed by weighing. Determination of the ash before and after the 25 (or 50) laundering cycles.

The determination may be carried out on the cloth as it is, the result being the total amount of ash. It may also be carried out on the cloth after the extraction of organic depositions (clause 6) and, in this case, it will give the amount of mineral ash.

7.3 Apparatus

Ordinary laboratory apparatus and

7.3.1 Silica, porcelain or platinum crucible, in accordance with ISO 1772.

7.3.2 Mecker or Bunsen burner.

7.3.3 Muffle furnace, capable of being controlled at 800 ± 25 °C.

7.4 Procedure

7.4.1 *Test portion*

Weigh the crucible (7.3.1) after heating it in the muffle furnace (7.3.3), controlled at 800 ± 25 °C, and allowing it to cool to ambient temperature in a dessiccator.

To determine the amount of total ash, take a test portion of mass approximately 3 g, weighed to the nearest 0,000 1 g, and place it in the tared crucible (7.3.1). Determine the water content of a second test portion as indicated in 6.5.2.

To determine the amount of mineral ash, it is only necessary to take the whole of the test portion in the Soxhlet extractor (6.4.1), the dry mass of which is known. Place the test portion in the tared crucible and expel the solvent by evaporation under a ventilated hood.

7.4.2 *Determination*

Place the crucible with its contents on a tripod and heat it over a small flame from the burner (7.3.2) until the cloth catches fire. Withdraw the flame and leave the cloth to burn freely; when the flame goes out, allow the ignition of the combustion points to continue. Repeat the operation until complete combustion is achieved.

Transfer the crucible to the muffle furnace (7.3.3), controlled at 800 ± 25 °C, and calcinate for 1 h.

Transfer the crucible to a dessiccator, allow to cool to ambient temperature and reweigh.

NOTES

1 See note 3 to 6.5.3.

2 To obtain the ash of cotton control cloth before laundering, the procedure specified in note 4 to 6.5.3 may be followed.

7.5 Expression of results

The ash of the original dry cloth is given, as a percentage by mass, by the formula

$$A = \frac{m_4 - m_3}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion, referred to the dry state of the cloth;

m_3 is the mass, in grams, of the empty crucible;

m_4 is the mass, in grams, of the crucible and ash after calcination.

Express the result to one decimal place.

Calculate the result of the test by taking the arithmetic mean of the values for three test pieces that have undergone 25 (or 50) laundering cycles ($A_{25(50)}$) and three test pieces intended for the determination of the initial characteristics of the cotton control cloth (A_0).

The increase ΔA in the ash due to the 25 (or 50) laundering cycles is given by the formula

$$\Delta A = A_{25(50)} - A_0$$

8 DETERMINATION OF OVERALL LOSS OF BREAKING STRENGTH (TOTAL DAMAGE)

8.0 Introduction

After repeated washings, the cotton control cloth generally exhibits less breaking strength than it did originally, due to the combined action of the mechanical and chemical factors operating during the 25 (or 50) laundering cycles.

Any loss of breaking strength, expressed as a percentage of the initial breaking strength, is determined from the variation in the breaking strength measured in the direction of the warp of the control cloth before and after laundering.

The measurement is effected on cloth stabilized in the standard conditioning atmosphere specified in ISO 139. For particular purposes, however, measurements may be made on water-wet cloth (wet strength).

contains coloured or thicker threads demarcating the width of the specimens (see ISO 2267, sub-clause 5.1.1, note 2).

Place the specimens, after removal of deposits, where necessary, in the conditioning enclosure (8.4.3) and leave them there for 24 h before measuring.

8.5.2 *Measurement of the breaking strength*

Carry out the measurement in the direction of the warp on the conditioned cloth in the atmosphere specified in 8.4.3 and complying with the procedure of ISO 5081 for a specified period for the average time-to-break of 30 ± 5 s.

Examine the laundered control cloth for evidence of localized damage.

8.6 Expression of results

The total wear U_t of the cotton control cloth, expressed as a percentage, is given by the formula

$$U_t = \frac{F_0 - F_{25(50)}}{F_0} \times 100$$

where

$F_{25(50)}$ is the breaking strength, expressed in newtons, of cotton control cloth subjected to the 25 (or 50) laundering cycles;

F_0 is the breaking strength, expressed in newtons, of cotton control cloth measured under the same conditions.

Express the result to one decimal place.

The breaking strength is calculated by taking the arithmetic mean of the individual values determined for each specimen that has been subjected to measurement, i.e., in principle, 30 measurements for all three test pieces representing a test.

NOTES

1 F_0 can be determined by proceeding as specified in 6.1 of ISO 2267. In view of the inherent variability of textiles, it is always preferable to make the 30 measurements specified for each test and to calculate the F_0 value by taking the arithmetic mean of these measurements.

2 The conditioning of the specimen may be omitted when measuring the breaking strength of the piece in the wet state and refer to prescriptions of ISO 5081.
If any localized damage is evident, describe it fully.

9 DETERMINATION OF LOSS OF BREAKING STRENGTH RESULTING FROM CHEMICAL DEGRADATION OF CELLULOSE DUE TO LAUNDERING (CHEMICAL DAMAGE)

9.0 Introduction

The chemical degradation of the cellulose in cloth, caused by the action of chemical agents during the laundering operation, usually accompanies a variation in the intrinsic mechanical properties of the fibres and, correlatively, a loss of breaking strength in the cloth. This chemical damage, which is a function of the chemical agressiveness of the laundering process, is characterized by a reduction in the degree of polymerization of the cellulose constituent.

Knowing the degree of polymerization — or a related parameter — of the cotton control cloth before and after the 25 (or 50) laundering cycles, and the link between the variation in this parameter and the corresponding loss of tensile strength, it is possible to calculate the part of the loss of breaking strength in the cotton control cloth which is due solely to chemical degradation.

Chemical damage is caused in the first place by the chemical agressiveness of washing products, mainly oxidizing agents such as hypochlorite and peroxides.

9.1 Scope and field of application

This clause specifies a method for the determination of the loss of breaking strength corresponding to the chemical degradation of the unsoiled cotton control cloth, due to the 25 (or 50) laundering cycles specified in ISO 2267.

9.2 Principle

Measurement of the fluidity value of a solution [in ammonium copper(II) hydroxide] of cotton provided by warp threads from the cotton control cloth before and after the 25 (or 50) laundering cycles.

The loss of breaking strength due to chemical degradation, expressed as a percentage of the initial breaking strength, is an approximate linear function of the increase in the fluidity value.

NOTE — There are many methods of measuring viscosity or its reciprocal, the fluidity, of cellulose (see 9.6).

The present method has been chosen because it is the only one for which the relation with the loss of breaking strength in cotton control cloth has been established experimentally.

9.3 Preparation of test samples

Ravel out warp threads from each of the specimens intended for the determination of total damage and take enough to give a sample of mass about 4 g for each test piece to be examined.

If it has been necessary to remove deposits from the cloth (see 8.5.1.1), take the threads from the specimens after the treatment to remove the deposits.

Cut the threads into strands from 1 to 2 mm long. Homogenize them. One part of this test sample is intended for the measurement of the fluidity value (or of the viscosity or degree of polymerization), the other for the determination of the water content.

9.4 Determination of fluidity value in ammonium copper(II) hydroxide solution

WARNING — Appropriate safety precautions should be observed when carrying out the operations described in this sub-clause.

9.4.1 *Definition*

fluidity of a solution : The reciprocal of the viscosity of the solution expressed in pascal seconds (Pa·s); fluidity is thus expressed in reciprocal pascal seconds [(Pa·s)$^{-1}$]. For

V_4 is the volume, in millilitres, of the standard volumetric *di*ammonium iron bis-sulphate solution used for the titration of the ammonium copper(II) hydroxide solution.

9.4.4 *Apparatus*

9.4.4.1 Viscometers, of the shape shown in figure 1 and the following dimensions :

- inside diameter of the capillary E : 0,88 ± 0,01 mm
- outside diameter of the capillary E : 6 ± 2 mm
- length of the capillary E : 25 ± 0,5 mm
- inside diameter of the tube A : 10 ± 0,05 mm
- distance from the reference mark B to the end of the capillary E : 242 ± 0,5 mm
- distance from the reference mark C to the end of the capillary E : 122 ± 0,5 mm
- distance from the reference mark D to the end of the capillary E : 62 ± 0,5 mm

The end of the capillary should be flat. The viscometer may be closed at the top by means of a plastic stopper pierced by a capillary tube; the latter may be closed by a small cap. The lower capillary may be closed by means of a rubber tube and a clamp. For each viscometer, the internal volume must be determined for the closed viscometer, subtracting a volume of 0,7 ml corresponding to the stirring device (see 9.4.4.4).

9.4.4.2 Supporting tubes for viscometers (see figure 2 for an example of a suitable shape).

The tubes should support the viscometers (9.4.4.1) in the thermostatic bath (9.4.4.3) for the period required to reach a temperature of 20 °C and while the flow time is being measured. In the latter case, the viscometers must be kept absolutely vertical.

9.4.4.3 Glass-walled thermostatic bath, capable of being controlled at 20 ± 0,2 °C and large enough to enable the tubes containing the viscometers to be kept immersed up to the upper rim by means of suitable fixtures.

9.4.4.4 Rotary stirrer, enabling the viscometers (9.4.4.1) to be rotated in such a way that the mercury (or the stirrer) slides from one end to the other of the tube A during each demi-rotation. The rotational frequency may vary between 4 and 10 min^{-1}.

9.4.4.5 Filling device, allowing the ammonium copper(II) hydroxide solution (9.4.3) to be maintained under nitrogen pressure and the viscometers (9.4.4.1) to be refilled by causing the liquid to rise through the capillary E (see figure 1).

9.4.5 *Procedure*

Weigh a portion of the test sample (9.3), to the nearest 0,000 1 g, such that a solution containing 0,5 g of the pure, dry cotton in 100 ml can be placed in the viscometer (9.4.4.1). To achieve this, the water content of the test sample, determined separately in accordance with 6.5.2 (see also note 3 to 6.5.2), and the organic and mineral deposits present in the test sample, determined in accordance with clauses 6 and 7, must be taken into account.

Introduce the test portion into the viscometer, which must be held upright. Using the short tube fitted with a clamp, connect the lower capillary tube of the viscometer to the vessel containing the ammonium copper(II) hydroxide solution (9.4.3) and make the solution rise into the viscometer under nitrogen pressure. When the viscometer is almost full, pinch the clamp of the lower tube and detach the viscometer. Add 0,7 ml of mercury by means of a short burette (alternatively, a stainless steel spiral with a volume of 0,7 ml may be used). Close the viscometer by means of a plastic stopper so that the liquid fills the viscometer completely and overflows through the capillary tube in the stopper, close this tube with a cap.

It is important to ensure that there is not a single air bubble left in the viscometer. In order to achieve this, the stopper must be inserted carefully but quickly. For the same reason, it may be necessary to tap the viscometer lightly before it is closed in order to detach air bubbles that might cling to the cotton fibres.

Place the viscometer, which shall not be exposed to light, on the stirring device (9.4.4.4). Allow it to rotate overnight at a frequency of 4 min^{-1} or for 4 h at 10 min^{-1} (see note 1).

Withdraw the viscometer from the stirring apparatus, remove the rubber tube from the lower capillary tube and allow the mercury to flow by opening the capillary in the upper stopper for a moment (this is not necessary when a spiral is used).

Place the viscometer in a supporting tube (9.4.4.2) immersed in the thermostatic bath (9.4.4.3), maintained at 20 ± 0,2 °C. Wait until the temperature has stabilized (at least 1 h). Check that the viscometer is in a vertical position. Remove the stopper and leave the solution to flow freely into the bulb of the support tube. Measure the time t (in seconds to the nearest 0,2 s) required for the meniscus to fall from the reference mark B to the reference mark D (see note 2).

NOTES

1 Since the control cloth is bleached so that the fluidity value of the cotton always exceeds, by definition, 40 $(\text{Pa·s})^{-1}$, there is no difficulty in dissolving it in the ammonium copper(II) hydroxide solution and a stirring period of 4 h at 10 min^{-1} (or 1 night at 4 min^{-1}) is always adequate.

2 To check that the solution is flowing freely through the capillary, it may be useful to measure both the time t_1 taken by the meniscus to fall from B to C and the time t_2 taken by it to fall from C to D. The ratio t_1/t_2 should be greater than 0,85 for a fluidity value of 40 $(\text{Pa·s})^{-1}$ and should tend towards unity for fluidity values higher than 40 $(\text{Pa·s})^{-1}$.

9.4.6 *Expression of results*

The fluidity value F of a solution containing 0,5 g of pure

10 DETERMINATION OF LOSS OF BREAKING STRENGTH RESULTING FROM MECHANICAL FACTORS IN LAUNDERING (MECHANICAL DAMAGE)

10.0 Introduction

It may be useful to have some information concerning the loss of breaking strength in the cotton control cloth due solely to the mechanical factors that come into play during the laundering process (mechanical damage).

Within the limits of practical error, total damage is always greater than chemical damage. By convention, this difference between total damage and chemical damage is defined as representing mechanical damage.

In reality, mechanical damage as defined here, is not usually independent of the amount of chemical damage. It also depends on the texture and surface mass of the cloth and the results recorded for the cotton control cloth cannot simply be transported to other cotton cloth washed at the same time.

Finally, cumulative errors in the values for total damage and for chemical damage during the determination of mechanical damage make it extremely difficult to reproduce the value found for the latter. A large number of tests must be carried out if an accurate value is required.

For all these reasons, the rate of mechanical damage can only be interpreted with care. In particular, every laboratory should establish quality standards for laundering, as necessary, from the point of view of mechanical damage based on the experience gained during the application of these standards in practice.

10.1 Expression of results

The mechanical damage U_m of the cotton control cloth after the 25 (or 50) laundering cycles is conventionally calculated as a percentage, within the limits of this International Standard, by taking the difference between the total damage (clause 8) and the chemical damage (clause 9) :

$$U_m = U_t - U_c$$

Express the result to the nearest whole number.

NOTE — An indication of the extent of mechanical damage may also be deduced from the difference between the breaking strength measured with the cloth in the wet condition and in the dry condition (see 8.0, note 2).

11 TEST REPORT

To enable the test results to be used in a reasonable manner, it is advisable to give all the experimental data and all the information likely to clarify the significance of the results that have been recorded.

The test report shall include the following details, in so far as they are known :

a) characteristics of the washing machine

 1) make of the machine;

 2) manufacturer's trade name;

 3) type of construction and materials used;

 4) normal capacity (mass of dry textile articles, in kilograms, normally handled); normal load factor (mass of dry textile articles, in kilograms, in relation to the internal volume, in cubic decimetres); normal liquor ratio (mass of dry textile articles, in kilograms, in relation to the volume of the wash liquor, in cubic decimeters);

 5) speed of rotation and frequency of reversals in the direction of rotation (where applicable);

 6) extraction procedure, when this is integral with the washing machine.

b) characteristics of the test piece (after five preparatory treatments).

c) Composition of the articles used (optional)

 1) washing agent;

 2) fluorescent whitening agent (where applicable);

 3) nature of the water and the treatments it has undergone.

d) Treatments composing a complete laundering cycle

 1) quantity of dry textile articles used;

 2) number of preliminary laundering cycles for the textile articles used (optional);

 3) for each of the laundering cycles and for each stage of a cycle (soaking, washing, bleaching, rinsing, etc.)

  – duration in minutes,

  – temperature in degrees Celsius,

  – quantity of water used (or liquor ratio),

  – nature and quantity (in grams) of the products used;

 4) characteristics of the extraction, drying, pressing and calendering or ironing treatments;

 5) number of laundering cycles to which the test piece has been subjected (25, 50 or other number as applicable);

 6) number of test pieces introduced at each cycle;

 7) special details.

e) Results of measuring the characteristics of the test pieces

In principle, the test report should state the following for each characteristic : the arithmetic mean $\bar{x}$ of the absolute values recorded for the specimens of three (or more) test pieces which have been tested simultaneously and of original test pieces [measured on three (or more) test pieces] in the case of a single test, or on specimens judiciously selected from the whole piece if this is used for several simultaneous tests.

ANNEX A

DETERMINATION OF INCREASE (OR DECREASE) IN DEGREE OF WHITENESS

(see 4.2.5, note 4)

A.0 INTRODUCTION

Visual whiteness is an important characteristic for determining the quality of laundering of white textile articles. It depends on the intrinsic greying (clause 4) and the intrinsic yellowing (clause 5) but, also on the amount and nature of the fluorescent whitening agents present and on the nature of the illuminant.

A.1 SCOPE

This annex specifies a method which enables a conventional figure to be obtained which is directly related to the visual whiteness observed in daylight, so long as the dominant wavelength characterizing the shade of the control cloth, observed in daylight, is not too far from 471 nm (blue situated close to the limit with violaceous blue), the shade produced by most fluorescent commercial bleaching agents under normal circumstances and so long as the whiteness perception is dominant over the perception of blue.

A.2 PRINCIPLE

By means of a reflectometer (photoelectric colorimeter) which enables the control cloth to be lit with a light corresponding to the CIE standard illuminant D65 (CIE 45-15-145), determination of the reflectometer values R_{g_j}; for the reflected light that has passed through Y (green) tristimulus filter and R_{b_j} for the reflected light that has passed through a Z (blue) tristimulus filter.

Conventional evaluation of the degree of whiteness Bl by the formula

$$Bl = 3\,R_{b_j} - 2\,R_{g_j}$$

and, in consequence, of the increase (or decrease) of the degree of whiteness, by calculation of the difference between the degree of whiteness after the 25 (or 50) laundering cycles and the degree of original whiteness of the control cloth.

A.3 APPARATUS

A.3.1 Reflectometer (photoelectric colorimeter), in which the sample to be measured can be lit by a light which corresponds as closely as possible to the CIE standard illuminant D65 (CIE 45-15-145), especially in the ultraviolet region of the spectrum.

In practice, it is only the xenon arc lamp, fitted with a suitable filter, that fulfils this condition.

It should be possible to interpose a Y (green) tristimulus filter or a Z (blue) tristimulus filter in the path of the light reflected by the sample. These filters differ from those specified in 4.4 and 5.4 inasmuch as their spectral sensitivity corresponds to that of the CIE $\bar{y}$ and $\bar{z}$ colour-matching functions respectively, taking into account the spectral sensitivity of the photoelectric cell, but ignoring the spectral distribution of the illuminant.

In no case is it permissible to interpose these filters between the source and the specimen.

A.3.2 Calibration plates, as specified in 4.4.2.

These plates shall be calibrated under the measuring conditions used in the present method.

A.4 PROCEDURE

Measure the reflectometer values R_{b_j} and R_{g_j} of the control cloth, using the Z (blue) and Y (green) tristimulus filters respectively under the measuring conditions for the apparatus specified in A.3.1 and by reference to the perfect diffuser illuminated under the same conditions (= 100).

Carry out the measurements on the various test pieces of cotton control cloth at least 12 h and at most 7 days after final ironing (or pressing). During this period, the test pieces shall be kept in conditions of the strictest cleanliness and protected from the light.

For each individual measurement, fold the test piece under test in such a way as to give a thickness of eight layers with the uppermost layer, on which the measurement is made, corresponding to the surface of the cloth which has been in contact with the polished part of the press or ironing machines. If it is not possible to distinguish the two surfaces, for example in a tumbler-dried test piece, carry out half the measurements on one surface and half on the other. Carry out ten individual measurements in this manner at different points on each test piece.

Repeat the measurements on each test piece.

A.5 EXPRESSION OF RESULTS

The degree of whiteness Bl is conventionally defined by the formula

$$Bl = 3\,R_{b_j} - 2\,R_{g_j}$$

where

R_{b_j} is the arithmetic mean, expressed to one decimal place, of 3 x 10 reflectometer values obtained with the Z (blue) tristimulus filter;

R_{g_j} is the arithmetic means, expressed to one decimal place, of 3 x 10 reflectometer values obtained with the Y (green) tristimulus filter.

ANNEX B

DETERMINATION OF CHEMICAL DAMAGE BASED ON VARIATION IN AVERAGE DEGREE OF VISCOMETRIC POLYMERIZATION IN AN AMMONIUM COPPER(II) HYDROXIDE SOLUTION

(see 9.6)

B.1 SCOPE AND FIELD OF APPLICATION

This annex specifies a method for the determination of the chemical damage of the unsoiled cotton control cloth, due to the 25 (or 50) laundering cycles specified in ISO 2267, based on the variation in the average degree of viscometric polymerization of the cotton in an ammonium copper(II) hydroxide solution.

B.2 DEFINITIONS (in accordance with ISO/R 1628)

B.2.1 viscosity relative increment : The viscosity ratio minus one.

$$\frac{\eta - \eta_0}{\eta_0} = \frac{t - t_0}{t_0}$$

where

η is the viscosity of the solution;

η_0 is the viscosity of the pure solvent;

t is the flow time for the solution;

t_0 is the flow time for the pure solvent.

B.2.2 viscosity number (I.V.) : The ratio of the viscosity relative increment to the concentration.

$$\text{I.V.} = \frac{t - t_0}{t_0\, c}$$

where

t and t_0 are as defined in B.2.1;

c is the concentration of the solution.

B.2.3 limiting viscosity number (I.L.V. or $[\eta]$) : The limiting value of the viscosity number.

$$\text{I.L.V.} = \lim_{c \to 0} \left(\frac{t - t_0}{t_0\, c} \right)$$

For the purpose of the present method, it is accepted that

$$\text{I.L.V.} = \frac{\text{I.V.}}{1 + 0{,}28\ (\text{I.V.})\ c} \quad \text{(Schulz-Blaschke formula)}$$

B.2.4 mean degree of viscometric polymerization (DP) : The ratio of the mean viscometric molar mass to the molar mass of the polymerization unit or monomer (in this case anhydroglucose).

$(DP)_B$ is DP determined by the present method, assuming that $(DP)_B = 2\,000$ (I.L.V.).

B.3 PRINCIPLE

Measurement of the viscosity relative increment of a solution or air-dried cotton (from which deposits have been removed, where necessary) in a standard solution of ammonium copper(II) hydroxide. Using this value, calculation of the limiting viscosity number by means of the Schulz-Blaschke formula (see B.2.3).

Multiplication of the limiting viscosity number by 2 000 to obtain the mean degree of viscometric polymerization $(DP)_B$.

Reading of the corresponding fluidity value from a graph. Calculation of the chemical damage as in 9.5.

B.4 REAGENTS

Use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

B.4.1 Copper(I) chloride (CuCl).

B.4.2 Copper(II) hydroxide $[Cu(OH)_2]$.

Dissolve 250 g of copper(II) sulphate pentahydrate $[CuSO_4.5H_2O]$ in 1 250 ml of warm water, bring to the boil, slowly add concentrated ammonia solution [25 % (*m/m*) NH_3] until the solution above the green precipitate assumes a violet tinge (about 150 ml). Filter the precipitate and wash it with water. Transfer the precipitate to a flask, mix with a little cold water, and add a cold solution of 60 g of sodium hydroxide in 800 ml of water. Stir for 10 min, filter and wash the precipitate with water until the washing water shows a neutral reaction. Then wash with acetone. Dry at a temperature below 30 °C.

B.4.3 Ammonia, 20 to 24 % (*m/m*) solution (185 to 220 g of NH_3 per litre).

B.5 APPARATUS

B.5.1 Viscometer.

Any type of viscometer with a capillary flow may be used (Ubbelohde, Cannon-Fenske, etc.). The capillary should be about 5 cm long and have an internal diameter of 0,3 to 0,4 mm.

B.5.2 Glass-walled thermostatic bath, capable of being maintained at 20 ± 0,1 °C.

B.5.3 Rotary shaker, which enables the flasks used for the dissolving process to be rotated.