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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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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 approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4312 was prepared by Technical Committee ISO/TC 91, *Surface active agents.*

This second edition cancels and replaces the first edition (ISO 4312 : 1979), and its addendum (ISO 4312 : 1979/add. 1 : 1983), of which it constitutes a minor revision.

Annexes A to C form an integral part of this International Standard. Annex D, which was previously ISO 4312 : 1979/Add. 1 : 1983, is for information only.

Introduction

It will be recalled that some effects produced by laundering of 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 wear and to modification, which cannot be controlled, due to actual use between successive launderings.

This wear 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 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 wear. 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 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.

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

1 Scope

This International Standard specifies the methods to be used to determine, under strictly controlled conditions, certain characteristics of unsoiled cotton control cloth, namely intrinsic greying and yellowing, increase in organic deposit content and incineration residue, the overall decrease in breaking strength, the decrease in breaking strength resulting from chemical degradation of the cellulose and the decrease in 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 of 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 the wear caused by vigorous mechanical action, they do not distinguish between the effects of smaller differences in mechanical action during laundering.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 139 : 1973, *Textiles — Standard atmospheres for conditioning and testing.*

ISO 1628-1 : 1984, *Plastics — Guidelines for the standardization of methods for the determination of viscosity number and limiting viscosity number of polymers in dilute solution — Part 1: General conditions.*

ISO 1772 : 1975, *Laboratory crucibles in porcelain and silica.*

ISO 2267 : 1986, *Surface active agents — Evaluation of certain effects of laundering — Method of preparation and use of unsoiled cotton control cloth.*

ISO 3105 : 1976, *Glass capillary kinematic viscometers — Specification and operating instructions.*

ISO 5081 : 1977, *Textiles — Woven fabrics — Determination of breaking strength and elongation (Strip method).*

CIE Publication No. 17 (E-1.1.) : 1979, *International lighting vocabulary.*

CIE Publication No. 38 (TC-2.3.) : 1977, *Radiometric and photometric properties of materials and their measurement.*

3 Selection of specimens and samples from test pieces

The measurements of intrinsic greying and intrinsic yellowing shall be carried out over the whole of the test piece.

The measurement of breaking strength shall be effected on specimens cut in the direction of the warp, as shown in figure 2 of ISO 2267 : 1986.

The measurement of chemical degradation shall be carried out on an average sample of the warp of the cloth composed of the ravelled threads of the specimens intended for use in the measurement of breaking strength.

The determination of ash and organic deposit shall be carried out on the remaining cloth, i.e. the end strips next to the selvedges. This cloth is cut into thin strips of suitable size (for example 1 cm wide and a few centimetres long) which are made into a homogeneous mixture.

4 Determination of intrinsic greying (greying measured in the absence of ultraviolet radiation)

4.1 Scope

This clause specifies a method for the determination of the intrinsic greying of the unsoiled cotton control cloth, due to the 25 (or 50) laundering cycles specified in ISO 2267.

The object of this determination is to provide information on the redeposition of coloured pigment soils from soiled white loads, usually grey, on the cloth. Too high a redeposition indicates a defect in the laundering. Greying may also be caused by the staining from dyes; obviously, this can only occur when coloured materials are present in the wash.

Intrinsic greying, as defined in 4.2.4, is not necessarily related to visual whitness (see notes 2 and 3 following 4.2.4).

NOTE — Varying results for intrinsic greying may be obtained in different laboratories. This does not detract from the value of the determination, which enables comparative tests to be made within a laboratory.

4.2 Definitions

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

4.2.1 radiance or luminance at a point of a source (CIE 45-06-150)[1] and (CIE 45-10-080)[1], L_v: The radiant or luminous flux leaving an element of surface at a point on the surface of a source and propagated in directions defined by an elementary cone containing the given direction, divided by the product of the solid angle of the cone and the area of the orthogonal projection of the elemant of surface on a plane perpendicular to the given direction.

In other words, the luminance measures the radiant or luminous intensity emitted in a given direction by the unit of surface.

4.2.2 radiance (luminance) factor (CIE 45-20-200)[2], β: The ratio of the radiance (luminance) of the medium to that of a perfect reflecting diffuser identically irradiated (illuminated).

$$\text{Spectral radiance factor } \beta(\lambda) = \frac{L_{e\lambda}}{L_{e\lambda w}}$$

where

$L_{e\lambda}$ is the spectral radiance of the medium;

$L_{e\lambda w}$ is the spectral radiance of a perfect reflecting diffuser.

The radiance (luminance) factor is only useful for diffuse radiation.

NOTES

1 In German, this quantity "Leuchtdichtefaktor" was formerly called "Remissionsgrad".

2 In the case of fluorescent media, the radiance (luminance) factor is the sum of two portions: the reflected radiance (luminance) factor β_S and the fluorescent radiance (luminance) factor β_L.

$$\beta_T = \beta_S + \beta_L$$

3 The fluorescent spectral radiance factor is a theoretical quantity which depends not only on the properties of the medium but also on the relative spectral distribution of the energy of the incident radiation.

4 The luminance factor is a photometric quantity which expresses most satisfactorily the visual perception of lightness.

5 If the radiance factor is multiplied by 100, the degree of radiance is obtained, as a percentage.

6 In the case of a totally diffuse surface (orthotropic diffuser), the radiance factor is identical to the reflectance in all conditions of illumination and observation.

If the surface is not totally diffuse (mixed reflexion) the radiance factor may be greater than the reflectance (if the direction of observation is such that a large proportion of the light reflected regularly is obtained) or smaller than the reflectance (in the case of directions of observation which exclude light reflected regularly).

The cloths used for examining the effects of washing are quite matt and the quantity measured is not important (provided that it is always the same). The same does not apply to glossy surfaces where generally it is desired to measure the diffuse reflection which corresponds to the normal visual observation of these surfaces which disregards the gloss In this case, the use of the reflectance is not suitable, the radiance factor shall be measured in a direction of observation which excludes regular reflection.

4.2.3 percentage degree of luminance: The luminance factor multiplied by 100.

NOTE — The percentage degree of luminance can be satisfactorily determined by means of a reflectometer fitted with a Y (green) tristimulus filter. This gives a reflectometer value (see 4.2.5) practically equal to the percentage degree of luminance, at least for a surface as matt as that of the control cloth and provided that it is measured on a 0 (absolute black) to 100 (perfect diffuser) scale and perpendicular to the surface, with illumination at an angle of 45°. With other geometries, different values may be obtained, depending on the amount of the specular component included in the measured luminous flux.

On the other hand, results which are generally not identical are obtained if the reduction in the spectral reflectance at an arbitrarily chosen wavelength situated in the green region of the spectrum is measured by means of a spectrophotometer, or if the reflectometer value for green light obtained by means of an arbitrarily chosen green filter is measured by means of a reflectometer. In both cases, the results depend on the wavelength or on the filter used and may differ appreciably between one laboratory and another. Of course, it is possible to calculate the percentage degree of luminance from the spectral reflectance curve (see note 2 to 4.4.1).

4.2.4 intrinsic greying: The decrease in the percentage degree of luminance of the control cloth, under lighting conditions such that the fluorescent effects due to any fluorescent whitening agents that may be present are eliminated.

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[1] See CIE Publication No. 17 (E-1.1.).

[2] See CIE Publication No. 38 (TC-2.3.).

NOTES

1 The definition of intrinsic greying enables a definite and unique value to be obtained in all cases, even if the greying is accompanied by yellowing or some other change of shade.

2 From the definitions given, it follows that intrinsic greying cannot necessarily be ascertained by visual inspection in daylight or even in light provided by other sources of illumination. In fact, all the usual sources of light, even incandescent lamps, emit ultraviolet radiation, thus inducing, to some extent, the fluorescence of fluorescent whitening agents that may be present.

In cases where the control cloth contains fluorescent whitening agents, intrinsic greying is not directly related to visual whiteness.

3 The degree of visual 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 visual 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, and annex D specifies the conditions for measuring reflectance, as well as the grey scale standards.

4.2.5 reflectometer value (CIE 45-20-202)[1]: The value measured by means of a particular reflectometer.

NOTE — The reflectometer employed should be specified. The measured reflectometer value depends on the geometrical configuration of the reflectometer, on the illuminant, on the spectral sensitivity of the receiver (even when equipped with filters) and on the reference standard used. These conditions should be specified, as and when applicable.

4.3 Principle

Measurement of the reflectometer value R_g of the cotton control cloth before and after 25 (or 50) laundering cycles, by means of a reflectometer (tristimulus 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 degree of luminance, is a measure of the intrinsic greying.

4.4 Apparatus

4.4.1 Reflectometer (tristimulus 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 enables any fluorescent effects to be excluded (see 4.2.3, note).

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 ultraviolet and violet light required to cause fluorescence from reaching the cloth. The second consists in illuminating the cloth with white light, but using a filter which blocks the ultraviolet rays between the light source and the cloth; the Y (green) tristimulus filter is placed between the cloth and the photoelectric cell. The UV filter should block all radiation 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 degree of luminance between 85 % and 90 % and the other of neutral grey with a percentage degree of 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 made of 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 will depend on the apparatus used. It should enable a correct measurement of the percentage degree of 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 pieces in conditions of the strictest cleanliness and protected from the light.

For each individual measurement, fold the test piece 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 machine. Carry out ten individual measurements in this manner at different points on the 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

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

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[1] See CIE Publication No. 38 (TC-2.3.).

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 degree of 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 degree of 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 achieved by commercial laundries, it would be preferable to establish once and for all a constant value for R_{g_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 88 % and 92 %.

5 Determination of intrinsic yellowing (yellowing measured in the absence of ultraviolet radiation)

5.1 Scope

This clause specifies a method for the determination of the intrinsic yellowing of the unsoiled cotton control cloth, due to the 25 (or 50) laundering cycles specified in ISO 2267.

In practice, the change of hue resulting from faulty laundering is often yellowing but it may be camouflaged by the presence of fluorescent whitening agents or ordinary blueing agents. There may be various reasons for this yellowing: the deposition of iron salts from the washing bath, inadequate rinsing which leaves alkaline substances in the cloth, development of the colour due to fatty soap residues, etc.

The object of this determination is to measure this yellowing under conditions such that it cannot be concealed by the presence of fluorescent whitening agents.

NOTE — Varying results for intrinsic yellowing may be obtained in different laboratories. This does not detract from the value of the determination, which enables comparative tests to be made within a laboratory.

5.2 Definitions

For the purposes of this clause, the definitions given in 4.2 and the following definitions apply.

5.2.1 degree of intrinsic yellow: The difference between the reflectometer value for the control cloth, measured with a reflectometer fitted with a Y (green) tristimulus filter, and the value measured with the same apparatus fitted with a Z (blue) tristimulus filter under measuring conditions such that any fluorescent effect due to any fluorescent whitening agent that might be present is eliminated, the measurement being made on the 0 (absolute black) to 100 (perfect diffuser) scale.

The degree of intrinsic yellow is always positive for the control cloth before it has been brought into use.

5.2.2 intrinsic yellowing: The increase in the degree of intrinsic yellow of the control cloth.

NOTES

1 These definitions enable a definite and unique value to be obtained both for the degree of intrinsic yellow and for intrinsic yellowing.

Results which are generally not identical are obtained if the difference between the percentage spectral reflectances at an arbitrarily chosen green or blue wavelength is measured (by means of a spectrophotometer) or if the difference between the reflectometer values using an arbitrarily chosen green or blue filter is measured (by means of a reflectometer). In such cases, the results depend on the choice of wavelength or filter and may differ appreciably between one laboratory and another. It is, of course, possible to obtain the same results by calculations based on the spectral reflectance curve.

2 From the definitions given above, it follows that the degree of intrinsic yellow, and thus the intrinsic yellowing, is not necessarily ascertainable by visual inspection in daylight or, for that matter, light from other sources of illumination, and is not necessarily directly related to visual whiteness (see 4.2.4, note 2).

3 Reflectometers with different illumination and observation configurations can, in principle, give different reflectometer values (see 4.2.3, note).

4 The degree of intrinsic yellow of cloth containing fluorescent whitening agents, measured under conditions such that any effect of fluorescence is eliminated, still differs from the degree of intrinsic yellow that the same cloth would possess if no fluorescent whitening agents were present. In fact, the absorption of the latter in the visible violet portion of the spectrum modifies the reflectometer values, particularly in the case of the light obtained with the Z (blue) tristimulus filter. For this reason, intrinsic yellowing should not be used for comparing the performance of detergents without fluorescent whitening agents with that of detergents with fluorescent whitening agents.

5.3 Principle

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

Evaluation of the degree of intrinsic yellow J in terms of the difference between R_g (see 4.6) and R_b.

The intrinsic yellowing ΔJ corresponds to the increase in the degree of intrinsic yellow after 25 (or 50) laundering cycles.

5.4 Apparatus

Identical to that specified in 4.4 but with the addition of a Z (blue) tristimulus filter which, in conjunction with the light source and the photoelectric cell of the reflectometer, affords a spectral sensitivity corresponding to the CIE colour-matching function $\bar{z}$ for the C source.

NOTES

1 See 4.4.1, note 1.

2 In theory, it is possible to obtain R_b by calculation based on the spectral reflectance curve determined by means of a spectrophotometer. In this case, the ordinates of the spectrophotometric curve must be multiplied by the tristimulus values $(\bar{z}H)\lambda$ for standard source C and the products integrated over the whole range of the spectrum. In this case too, any fluorescence must, of course, be eliminated.

5.5 Procedure

After effecting the measurements described in 4.5, repeat them after replacing the Y (green) tristimulus filter by the Z (blue) tristimulus filter, the reflectometer value being measured in relation to the perfect diffuser (= 100) under the same lighting conditions.

5.6 Expression of results

The degree of intrinsic yellow J of the laundered test pieces is normally 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 from the Z (blue) tristimulus filter as recorded on the three test pieces after they 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, of 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 deposit content

6.1 Scope

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-glass 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 ml 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 maintaining a temperature of 103 °C ± 2 °C.

6.4.4 Drying tube.

6.4.5 Water bath or **flameless heater.**

6.4.6 Vacuum pump.

6.5 Procedure

WARNING — Benzene is toxic and easily flammable. Carry out the determination under a well ventilated hood.

6.5.1 Test portion

Take a test portion of mass approximately 3 g, weighed to the nearest 0,1 mg, from that remaining part of the test piece which is intended for measurement of decrease in breaking strength and has been prepared in accordance with clause 6 of ISO 2267: 1986 (see also clause 3).

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

6.5.2 Determination of water content

Place the second test portion in the weighing bottle (6.4.2), previously dried at 103 °C ± 2 °C, allowed to cool in a desiccator and weighed. Place the weighing bottle and its stopper separately in the oven (6.4.3), set at 103 °C ± 2 °C, and leave for at least 2 h. At the end of this period, stopper the weighing bottle and leave it to cool completely in a desiccator. When it is to be weighed again, open and close the weighing bottle very quickly so that the atmospheric pressure inside it is reestablished.

The difference in mass before and after drying, expressed as a percentage by mass of the dry cloth, will give the water content of the cloth at the time of the initial weighing (see 6.5.1).

Calculate, by proportion, the dry mass of the first test portion.

6.5.3 Determination

Place the first test portion in the extractor (6.4.1.2); fit the receiving flask (6.4.1.1), which has been previously dried at 103 °C ± 2 °C, allowed to cool in a desiccator and weighed, and the condenser (6.4.1.3) to the extractor, then pour in a sufficient amount of the extraction solvent (6.3) via the condenser to fill the extraction thimble once; add about 10 % to 20 % of the previous quantity of solvent.

Fit the drying tube (6.4.4) to the top of the condenser so that only dry air enters the apparatus.

Warm the receiving flask by means of the water bath or flameless heater (6.4.5) and control the heating so that the extraction thimble fills at least five times per hour.

Continue with the extraction for 6 h.

Connect the receiving flask to the condenser (rearranged for distillation), heat in the water bath or over the flameless heater and distil the solvent as completely as possible. Detach the flask and warm it in the water bath or over the flameless heater for 5 min, drawing the air from inside the flask by means of a glass tube connected to the vacuum pump (6.4.6).

Heat the flask and residue in the oven (6.4.3), set at 103 °C ± 2 °C, for 1 h. Insert a tube connected to the vacuum pump for a few seconds to remove completely all vapour.

Heat the receiving flask in the oven, set at 103 °C ± 2 °C, for a further 5 min, then allow it to cool in the desiccator to ambient temperature and weigh it.

This weighing operation shall be carried out under exactly the same conditions as those under which the receiving flask was calibrated.

NOTES

1 In cases where the test portion is introduced into the apparatus by means of a filter paper cartridge, care must be taken to ensure by means of a blank test that there is no extractable matter in the cartridge.

2 When the organic or mineral deposit content is presumed to be small [less than 5 % (m/m)], it is permissible to calculate the dry mass of the test portion, instead of measuring it by the loss of mass after drying, by multiplying by 0,93 the mass of the test portion conditioned in the standard atmosphere (65 % relative humidity, 20 °C).

In all cases, state the procedure selected in the test report.

3 In the case of large batch production, it is permissible to determine once and for all the organic deposit content of control cloth that has been obtained from the same piece of cloth and subjected to the same preparatory treatments (see 5.2 of ISO 2267 : 1986).

6.6 Expression of results

The organic deposit content, I, expressed as a percentage by mass of the dry cloth, is given by the formula

$$I = \frac{m_2 - m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the dry test portion, calculated as in 6.5.2;

m_1 is the mass, in grams, of the empty receiving flask;

m_2 is the mass, in grams, of the flask with the dry extract.

Express the result to one decimal place.

Calculate the arithmetic mean of the results for three test pieces that have undergone 25 (or 50) laundering cycles ($I_{25(50)}$) and three test pieces intended for the determination of the initial values (I_0).

The increase ΔJ in the organic deposit content caused by the 25 (or 50) laundering cycles is given by the formula

$$\Delta J = I_{25(50)} - I_0$$

Express the result to one decimal place.

7 Determination of increase in incineration residue (ash)

7.1 Scope

This clause specifies a method for the determination of the increase in the incineration residue (ash) of the unsoiled cotton control cloth, caused by the 25 (or 50) laundering cycles specified in ISO 2267.

The ash content of the cotton control cloth is very low [of the order of 0,10 % to 0,20 % (m/m)]. After the 25 (or 50) laundering cycles, the ash may be higher due to the deposition of mineral or organic salts.

NOTE — The amount of incineration residue obtained depends on the conditions of the operation itself, mainly on the incineration temperature.

The incineration residue may be determined directly on the control cloth (total ash) or after the organic deposits have been eliminated (mineral ash).

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

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

Deposits of mineral products on cloth indicate unsatisfactory detergent action; they may shorten the useful life of the cloth by modifying its organoleptic qualities (harsh feel, dull appearance, and greying or yellowing) or by promoting chemical or mechanical wear.

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 the organic deposits (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 Meker or Bunsen burner.

7.3.3 Muffle furnace, capable of maintaining a temperature of 800 °C ± 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) set at 800 °C ± 25 °C, and allowing it to cool to ambient temperature in a desiccator.

To determine the amount of total ash, take a test portion of mass approximately 3 g, weighed to the nearest 0,1 mg, 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, the whole of the test portion in the Soxhlet extractor (6.4.1), the dry mass of which is known, may be taken. 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 the burner (7.3.2) with a small flame until the cloth catches fire. Withdraw the flame and leave the cloth to burn freely; when the flame goes out, allow the cloth to continue smouldering. Repeat the operation until complete combustion is achieved.

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

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

NOTES

1 See note 2 to 6.5.3.

2 To obtain the ash of cotton control cloth before laundering, the procedure specified in note 3 to 6.5.3 may be followed.

7.5 Expression of results

The ash A 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 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 decrease in breaking strength (total wear)

8.1 General

After repeated washings, the cotton control cloth generally has a lower 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 decrease in 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).

Different types of dynamometer may give different values for the breaking strength of a given cloth, and also for the difference in the breaking strengths of two given cloths. Thus, the results of different laboratories will not always be comparable; therefore, every laboratory should, as necessary, establish standards for the quality of laundering, based on the measuring equipment used.

Nevertheless, knowledge of the total wear of the control cloth determined under given conditions enables different laundering procedures to be compared from the point of view of their effect on the probable wear life of cotton articles.

In fact, although the total wear as measured only applies, strictly speaking, to the cotton control cloth under the measuring conditions used, it is obvious that, if one of two laundering procedures causes more wear to the cotton control cloth, it will also cause more wear to any cotton textile.

NOTES

1 Experience has shown that, when the breaking strength of cloth falls below 147 N, the appearance of tears during washing or during normal use is virtually inevitable.

2 The breaking strength measured in the wet state is greater than that measured in the dry state for the cotton control cloth in its initial state.

As the chemical degradation increases, the difference diminishes and becomes negative at a certain level of chemical degradation. Comparison of the breaking strength measured in the wet state and that in the dry state may therefore provide useful information concerning the nature of the degradation.

8.2 Scope

This clause refers to ISO 5081 for the determination of the breaking strength of the unsoiled cotton control cloth before and after the 25 (or 50) laundering cycles specified in ISO 2267.

Conventionally, the difference in the breaking strength of the control cloth before and after the 25 (or 50) laundering cycles, expressed as a percentage of the breaking strength of the cotton control cloth, characterizes the total wear of the cloth resulting from these launderings.

8.3 Principle

Determination of the breaking strength of fabric strips (specimens) of cotton control cloth before and after the cloth has undergone 25 (or 50) laundering cycles, measured in the direction of the warp of the conditioned cloth (see ISO 5081).

8.4 Reagent for removal of deposits

Disodium salt of (ethylenedinitrilo)tetra-acetic acid (EDTA), recognized analytical grade, 5 g/l solution in distilled water or water of zero hardness (i.e. 0 mg of Ca^{2+} ions per litre).

8.5 Apparatus

8.5.1 Washing machine, of a type such that the test pieces (from which the specimens will subsequently be cut) can be put into it after the washing solution has been heated to at least 85 °C.

8.5.2 Dynamometer, for measuring the breaking strength of fabric strips and enabling a constant breaking time of 30 s ± 5 s to be obtained for the specimens.

8.5.3 Air-conditioning enclosure, capable of maintaining the standard atmosphere (temperature of 20 °C ± 2 °C and relative humidity of 65 % ± 2 %) in accordance with ISO 139.

8.6 Procedure

8.6.1 Preparation of specimens

8.6.1.1 Removal of deposits, where necessary

If the total ash (see clause 7) exceeds 5 % (m/m), or if the organic deposit content of the cloth (see clause 6) exceeds 10 % (m/m), remove the deposits from the test pieces by the following method.

Fill the washing machine (8.5.1) with the disodium EDTA solution (8.4) and heat it to at least 85 °C.

Add the test pieces from which the deposits are to be removed. The amount added shall be such that the ratio is not more than 1 : 20 (a maximum of 1 kg of strips to 20 litres of solution).

Leave the test pieces in the machine for 30 min, taking care the temperature does not fall below 85 °C.

Agitate at regular intervals so as to maintain a uniform temperature and to ensure uniformity in the removal of deposits.

Empty the machine of disodium EDTA solution. Rinse the test pieces in the machine twice with cold water having zero hardness, at a ratio of not more than 1 : 20.

Hydroextract the test pieces and dry them in air.

Where necessary, ensure that the total ash is lower than 5 % (*m/m*) and that the organic deposit content of test pieces from which deposits have been removed is lower than 10 % (*m/m*), following the procedures specified in clauses 6 and 7. If this is not the case, repeat the procedure for removing deposits.

In order to ensure that the operation of removing deposits does not cause additional wear, it is recommended that the test pieces that have not undergone the 25 (or 50) laundering cycles should be similarly, but separately, treated.

8.6.1.2 Cutting and conditioning

Cut the test pieces into specimens as shown in figure 2 of ISO 2267 : 1986 and complying with the prescriptions of ISO 5081.

It is very much easier to carry out the operation if the cloth contains coloured or thicker threads demarcating the width of the specimens (see ISO 2267 : 1986, sub-clause 5.1.1, note 2).

Place the specimens, after removal of deposits, where necessary, in the conditioning enclosure (8.5.3) and leave them there for 24 h before measuring.

8.6.2 Localized wear

Examine the laundered control cloth for evidence of localized wear.

8.6.3 Measurement of the breaking strength

Carry out the measurement in the direction of the warp on the conditioned specimens in the atmosphere specified in 8.5.3 and complying with the procedure of ISO 5081 with an average time-to-break period of 30 s ± 5 s.

8.7 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 25 (or 50) laundering cycles;

F_0 is the breaking strength, expressed in newtons, of unlaundered 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 : 1986. 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. In this case, refer to the prescriptions of ISO 5081.

3 If any localized wear is evident, describe it fully.

9 Determination of decrease in breaking strength resulting from chemical degradation of cellulose due to laundering (chemical wear)

9.1 General

The chemical degradation of the cellulose in cotton cloth, caused by the action of chemical agents during the laundering operation, usually leads to changes in the intrinsic mechanical properties of the fibres and a decrease in the breaking strength of the cloth. This chemical damage, which is a function of the chemical aggressiveness 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 decrease in breaking strength, it is possible to calculate the part of the decrease in breaking strength of the cotton control cloth which is due solely to chemical degradation.

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

9.2 Scope

This clause specifies a method for the determination of the decrease in 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.

9.3 Principle

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

The decrease in breaking strength due to chemical degradation, expressed as a percentage of the initial breaking strength, is approximately proportional to the increase in the fluidity value.

NOTE — There are many methods of measuring the viscosity or its reciprocal, the fluidity, of cellulose (see 9.7).

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

9.4 Preparation of test samples

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

If it is has been necessary to remove deposits from the cloth (see 8.6.1.1), take the threads from the specimens after the treatment to remove the deposits.

Cut the threads into strands from 1 mm 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.

9.5 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.

9.5.1 Definition

fluidity of a solution: The reciprocal of the viscosity of the solution expressed in pascal seconds (Pa·s); fluidity is thus expressed in pascal seconds to the power of minus one [(Pa·s) $^{-1}$]. For the purposes of this International Standard, the fluidity shall always be related to that of a standard solution measured under standard conditions.

9.5.2 Principle

Dispersion of a quantity of air-dried cotton, from which deposits have been removed where necessary, in a standard solution of ammonium copper(II) hydroxide to give a solution of 0,5 g of pure, dry cotton per 100 ml. Introduction of the solution into a standard viscometer. Free flow of the solution through the capillary tube of the viscometer, maintained at 20 °C. Measurement of the flow rate by the time taken for the meniscus to fall from one fixed reference mark to another. Calculation of the fluidity using a formula with two constants determined by calibrating the viscometer with liquids of known viscosity.

9.5.3 Reagent

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

Ammonium copper(II) hydroxide, standard solution containing per litre

15 g ± 0,2 g of copper (Cu)

200 g ± 5 g of ammonia (NH_3)

less than 0,5 g of nitrite, calculated as nitrous acid (HNO_2).

The density at 20 °C, ϱ_{20}, of this solution is 0,94 g/ml; it shall be kept in the dark or in a dark-brown glass flask, protected from the oxygen in the air.

9.5.3.1 Preparation

Dissolve about 31 g of dicopper carbonate dihydroxide [$CuCO_3.Cu(OH)_2$] in 1 litre of concentrated ammonia solution 25 % (*m/m*). Stir it for 1 h in a hermetically sealed dark-brown glass flask at a temperature below 20 °C. Leave it overnight and decant off the supernatant liquid from any deposit of copper(II) oxide that may appear.

Analyse the solution as described in 9.5.3.2 and correct the composition, if necessary, by small additions of dicopper carbonate dihydroxide, ammonia solution or water.

9.5.3.2 Standardization

9.5.3.2.1 Determination of copper content

Place 25 ml of the solution prepared as in 9.5.3.1 into a 250 ml conical flask. Boil it until the ammonia is eliminated. Acidify with dilute sulfuric acid and boil to eliminate any nitrous acid that may be present. Cool, add a few drops of bromine, and boil until the bromine is eliminated. Neutralize with ammonia solution, add 7 ml of concentrated acetic acid, cool and add 3 g of potassium iodide. Titrate the liberated iodine with a standard volumetric sodium thiosulfate solution, $c(Na_2S_2O_3) = 0{,}1$ mol/l, using soluble starch as indicator.

The copper content c, in grams per litre, is given by the formula

$$c = 0{,}254\,V_1$$

where V_1 is the volume, in millilitres, of the standard volumetric sodium thiosulfate solution used for the titration.

9.5.3.2.2 Determination of ammonia content

Carefully draw off exactly 2,0 ml of the solution prepared as in 9.5.3.1 by means of a pipette, and introduce it into 50 ml of a standard volumetric sulfuric acid solution, $c(H_2SO_4) = 0{,}5$ mol/l, keeping the end of the pipette below the surface of the acid solution. Titrate the excess acid by means of a standard volumetric sodium hydroxide solution, $c(NaOH) = 1$ mol/l, using methyl red as indicator.

The ammonia (NH_3) content, in grams per litre, is given by the formula

$$8{,}5\,(50 - V_2) - 0{,}536\,c$$

where

V_2 is the volume, in millilitres, of the standard volumetric sodium hydroxide solution used for the titration;

c is the copper content calculated as specified in 9.5.3.2.1.

9.5.3.2.3 Determination of nitrite content

First prepare as follows a solution of cerium(IV) sulfate, $c(Ce(SO_4)_2.4H_2O)$ = 0,01 mol/l: add, to 4 g of cerium(IV) sulfate tetrahydrate $[Ce(SO_4)_2.4H_2O]$, 12,5 ml of concentrated sulfuric acid and, with care, 12,5 ml of water. Boil for 5 min, cool and dilute with water to 250 ml. Determine the concentration exactly by titrating with a standard volumetric solution of diammonium iron(II) sulfate, $c[(NH_4)_2Fe(SO_4)_2]$ = 0,01 mol/l, using 0,5 ml of a 10 g/l ferroin[1] solution as indicator, until the appearance of a red colour.

Add 10 ml of the ammonium copper(II) hydroxide solution prepared in 9.5.3.1 to a mixture of 25 ml of the cerium(IV) sulfate solution, $c[Ce(SO_4)_2.4H_2O]$ = 0,01 mol/l, and 100 ml of sulfuric acid solution, $c(H_2SO_4)$ = 1 mol/l. After 10 min, determine the nitrite content, proceeding as for the determination of the concentration of the cerium(IV) sulfate solution.

The nitrite content, in grams of nitrous acid (HNO_2) per litre, is given by the formula

$$0{,}094\,(V_3 - V_4)$$

where

V_3 is the volume, in millilitres, of the standard volumetric diammonium iron(II) disulfate solution used to determine the strength of the cerium(IV) sulfate solution;

V_4 is the volume, in millilitres, of the standard volumetric diammonium iron(II) disulfate solution used for the titration of the ammonium copper(II) hydroxide solution.

9.5.4 Apparatus

9.5.4.1 Viscometer, of the type shown in figure 1 and with the following dimensions:

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

The end of the capillary shall 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 (corresponding to the closed viscometer) shall be determined, subtracting a volume of 0,7 mm corresponding to the stirring device (see 9.5.4.4).

9.5.4.2 Supporting tube for viscometer (see figure 2 for an example of a suitable type).

The tube shall support the viscometer (9.5.4.1) in the thermostatic bath (9.5.4.3) for the period required to reach a temperature of 20 °C and while the flow time is being measured. During the latter, the viscometer shall be kept absolutely vertical.

9.5.4.3 Glass-walled thermostatic bath, capable of maintaining a temperature of 20 °C ± 0,2 °C and large enough to enable the tube containing the viscometer to be kept immersed up to the upper rim by means of suitable fixtures.

9.5.4.4 Rotary stirrer, enabling the viscometer (9.5.4.1) to be rotated in such a way that the mercury (or the stirrer) slides from one end to the other of tube A during each half-rotation. The rotational frequency may vary between 4 min^{-1} and 10 min^{-1}.

9.5.4.5 Filling device, allowing the ammonium copper(II) hydroxide solution (9.5.3) to be kept under nitrogen pressure and the viscometer (9.5.4.1) to be refilled by causing the liquid to rise through the capillary E (see figure 1).

9.5.5 Procedure

Weigh a portion of the test sample (9.4), to the nearest 0,1 mg, such that a solution containing 0,5 g of the pure, dry cotton in 100 ml can be placed in the viscometer (9.5.4.1). To achieve this, the water content of the test sample, determined separately in accordance with 6.5.2 (see also 6.5.3, note 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 (9.5.3) and make the solution rise into the viscometer under nitrogen pressure. When the viscometer is almost full, close 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 the overflow tube with a cap.

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[1] Ferroin is the tris(1,10-phenanthroline) iron(II) ion.

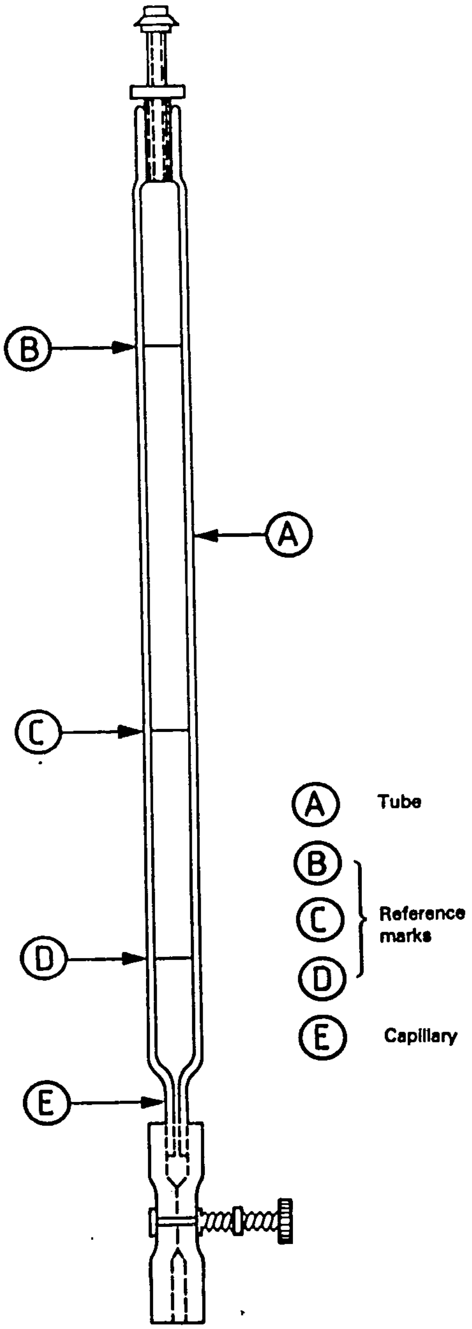

Figure 1 — Viscometer (see 9.5.4.1)

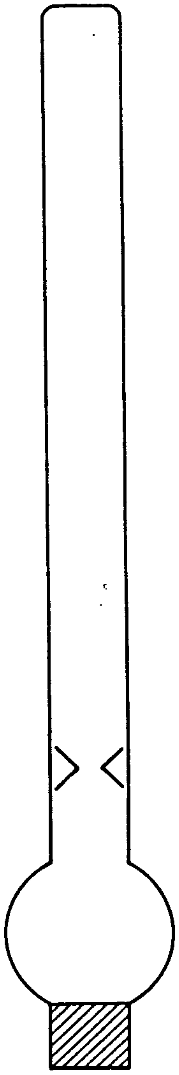

Figure 2 — Supporting tube for viscometer (see 9.5.4.2)

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 (9.5.4.4). 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 out 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 (9.5.4.2) immersed in the thermostatic bath (9.5.4.3), maintained at 20 °C ± 0,2 °C. Wait until the temperature has stabilized (at least 1 h). Check that the viscometer is in the vertical position. Remove the stopper and allow 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 $(\text{Pa}\cdot\text{s})^{-1}$, 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 $(\text{Pa}\cdot\text{s})^{-1}$ and should tend towards unity for fluidity values higher than 40 $(\text{Pa}\cdot\text{s})^{-1}$.

9.5.6 Expression of results

The fluidity value F of a solution containing 0,5 g of pure and dry cotton in 100 ml of the ammonium copper(II) hydroxide solution (9.5.3) standardized at 20 °C, expressed in pascal seconds to the power of minus one, is given by the formula

$$F = \frac{C}{0{,}94\,(t - K/t)}$$

where

t is the flow time (from B to D), in seconds;

C and K are the viscometer constants (determined as specified in 9.5.7.2);

0,94 is taken to be the density at 20 °C, in grams per millilitre, of the solution.

Calculate F to one decimal place for each of three test pieces that have undergone the 25 (or 50) laundering cycles and the arithmetic mean of the three results to one decimal place.

Determine F_0 for each of three unlaundered test pieces and calculate the arithmetic mean of the results to one decimal place. In some cases, it is possible to proceed as described in 6.5.3, note 3.

9.5.7 Calibration of viscometer

9.5.7.1 Determination of useful internal volume

Fill the previously weighed viscometer with distilled water at 20 °C under normal test conditions. Reweigh and calculate the internal volume, in millilitres, from the difference in mass. This volume, reduced by 0,7 ml, is the internal volume used in calculating the quantity of pure, dry cotton to be introduced into the viscometer.

9.5.7.2 Determination of constants C and K

Determine the flow time, at 20 °C, of the three following liquids:

distilled water;

solution 1 (a mixture a glycerine and water, the density and viscosity of which are known precisely, and having a viscosity of less than 10 mPa·s);

solution 2 (a mixture of glycerine and water, the density and viscosity of which are known precisely, and having a viscosity of about 40 mPa·s).

The density of these mixtures is determined by means of a pycnometer to four places of decimals. The corresponding viscosity is obtained from standard tables.

For viscosities higher than 10 mPa·s, K may be taken as 0. The flow time for solution 2 then enables C to be determined directly from the formula

$$C = \frac{1\,000\,\varrho\,t}{\eta} \qquad \dots (1)$$

where

ϱ is the density, in grams per millilitre, of solution 2;

t is the flow time, in seconds, for solution 2;

η is the viscosity, in millipascal seconds, of solution 2.

Calibration with water permits the calulation of K from the formula

$$K = t^2 - \frac{C\,t}{1\,000} \qquad \dots (2)$$

where

t is the flow time, in seconds, for the water;

C is given by formula (1)
(taking ϱ_{20} = 1 g/ml and η = 1 mPa·s for water).

By using this K value, C can be calculated for solution 1 by means of the formula in 9.5.6. The mean of the C values determined with solutions 1 and 2 is taken as the calibration value for the viscometer.

9.6 Calculation of chemical wear

The fluidity value F_0 for unlaundered cotton control cloth [lying between 40 $(\text{Pa}\cdot\text{s})^{-1}$ and 50 $(\text{Pa}\cdot\text{s})^{-1}$, in accordance with ISO 2267] is subtracted from the fluidity value $F_{25(50)}$ for cloth that has undergone the 25 (or 50) laundering cycles:

$$\Delta F = F_{25(50)} - F_0$$

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

$$U_c = 0{,}165\,\Delta F$$

It is only significant up to a value of 40 %, which is the upper limit of the validity of the above relationship.

NOTE — This linear relationship gives the best representation of all the experimental results up to 40 % chemical wear [see *J. Soc. Dyers and Colourists*, 83, 152 (1967)].

9.7 Other viscometer methods

As stated in 9.3, it is possible, in principle, to establish a relationship between chemical wear and the variation in either the viscosity, measured in accordance with any of the numerous existing methods, or the degree of polymerization of the cellulose in the cotton, calculated from the viscometric measurements.

Up till now, this relationship has been established experimentally only for measurements carried out using the procedure specified in 9.5.

However, the relation between the fluidity determined in accordance with 9.5 and the degree of polymerization calculated on the basis of the limiting viscosity numbers has been established in the course of numerous inter-laboratory tests for two methods of determining viscosity.

These methods are described in annexes B and C.

Whichever of these methods is used, it is therefore possible to convert the results obtained into fluidity values and from these into chemical wear values.

10 Determination of decrease in breaking strength resulting from mechanical factors in laundering (mechanical wear)

10.1 General

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

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

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

Finally, cumulative errors in the values for total wear and for chemical wear used in determining mechanical wear 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 value obtained for the rate of mechanical wear should be interpreted with care. In particular, every laboratory should establish quality standards for laundering, based on the experience gained during the application of this International Standard in practice.

10.2 Expression of results

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

$$U_m = U_t - U_c$$

Express the result to the nearest whole number.

NOTE — An indication of the extent of mechanical wear 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 8.1, note 2).

11 Test report

To enable the test results to be interpreted in a rational 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 decimetres);

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

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

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

c) Composition of the washing products 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 cyles 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 value recorded for the specimens of three (or more) test pieces which have been tested simultaneously and of unlaundered 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.

In the latter case, the number of specimens and the way in which they have been selected from the piece of cloth should be stated and the choice justified by statistical calculations which are sufficiently detailed to permit an objective evaluation of the accuracy obtained.

The repeatability standard deviation (s_r) should be given for each mean $\bar{x}$ of the quantities measured.

The presentation of results should show the comparison between the values recorded for the unlaundered test piece and that recorded for the strip that has undergone the laundering cycles, for each characteristic.

f) Conclusion of test and comments.

NOTE — It is impossible to give all the values for repeatability and reproducibility that might be expected from the results obtained by the methods specified in this International Standard. In practice, the total variance is not due simply to the variance in the method itself but also to the variance in the control cloth before it is brought into use and the variance resulting from laundering. In each case, therefore, only a complete statistical study will make it possible to separate the individual variances.

Annex A
(normative)

Determination of increase (or decrease) in degree of whiteness

(see 4.2.4, note 3)

A.1 General

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), and also on the amount and nature of the fluorescent whitening agents present and on the nature of the illuminant.

A.2 Scope

This annex specifies a method which enables a value 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 of 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.3 Principle

By means of a reflectometer (tristimulus colorimeter) which enables the control cloth to be illuminated with light corresponding to the CIE standard illuminant D_{65} (CIE 45-15-145), determination of the reflectometer values R_{g_j} for the reflected light that has passed through a Y (green) tristimulus filter and R_{b_j} for the reflected light that has passed through a Z (blue) tristimulus filter.

Calculation 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) in the degree of whiteness from the difference between the degree of whiteness after 25 (or 50) laundering cycles and the degree of whiteness of the unlaundered control cloth.

A.4 Apparatus

A.4.1 Reflectometer (tristimulus colorimeter), in which the sample to be measured can be illuminated by light which corresponds as closely as possible to the CIE standard illuminant D_{65} (CIE 45-15-145), especially in the ultraviolet region of the spectrum. In practice, only a xenon arc lamp, fitted with a suitable filter, fulfils this condition.

It should be possible to interpose a Y (green) tristimulus filter or 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 in as much as their spectral sensitivity corresponds to that of the CIE colour-matching functions $\bar{y}$ and $\bar{z}$ 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.4.2 Calibration plates, as specified in 4.4.2.

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

A.5 Procedure

Measure the reflectometer values R_{b_j} and R_{g_j} for the control cloth, using the Z (blue) and Y (green) tristimulus filters respectively under the measuring conditions described for the apparatus specied in A.4.1 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 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 machine.

Carry out ten individual measurements in this manner at different points on each test piece.

Repeat the measurements on each test piece.

A.6 Expression of results

The degree of whiteness Bl is normally 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 × 10 reflectometer values obtained with the Z (blue) tristimulus filter;

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

The increase in the degree of whiteness is given by the formula

$$\Delta Bl = Bl_{25(50)} - Bl_0$$

where

$Bl_{25(50)}$ is the degree of whiteness of the control cloth after the 25 (or 50) laundering cycles;

Bl_0 is the initial degree of whiteness of the control cloth (after the five preparatory treatments).

A negative value corresponds to a decrease in the degree of whiteness.

NOTES

1 If the shade of the control cloth, observed in daylight, is characterized by a dominant wavelength lying outside the interval 466 nm to 476 nm, i.e. if it exhibits a greenish or reddish tinge, this method gives results which are no longer in accordance with the whiteness as evaluated visually. Furthermore, the present method should not be used when the value of R_{g_j} is less than 75, unless the value of R_{b_j} is lower than that of R_{g_j}.

2 Owing to the difficulty of reproducing standard illuminant D_{65}, especially in the ultraviolet region, the values obtained by this method in different laboratories may show large variations, even when instruments of the same type are used. However, the order, by degree of whiteness, of different samples will in general be much more reproducible.

Annex B
(normative)

Determination of chemical wear based on variation in average degree of viscometric polymerization in an ammonium copper(II) hydroxide solution

(see 9.7)

B.1 Scope

This annex specifies a method for the determination of the chemical wear of 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

For the purposes of this annex, the following definitions, as given in ISO 1628-1, apply.

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

$$\left(\frac{\eta}{\eta_0}\right) - 1 = \frac{\eta - \eta_0}{\eta_0} = \frac{t - t_0}{t_0}$$

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, in seconds;

t_0 is the flow time for the pure solvent, in seconds.

B.2.2 viscosity number (V.N.): The ratio of the viscosity ratio increment to the concentration, c, of cotton in the solution:

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

where t and t_0 are as defined in B.2.1.

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

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

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

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

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

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

B.3 Principle

Measurement of the viscosity ratio increment of a solution of 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 wear as in 9.6.

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) sulfate 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 washings show a neutral reaction. Then wash with acetone. Dry at a temperature below 30 °C.

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

B.5 Apparatus

B.5.1 Capillary viscometer, of any type meeting the requirements of ISO 3105 (Ubbelohde, Cannon-Fenske, etc.).

The capillary shall be about 5 cm long and have an internal diameter of 0,3 mm to 0,4 mm.

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

B.5.3 Reciprocating shaker.

B.5.4 Device to enable the solution of cotton in ammonium copper(II) hydroxide to be transferred from the flask in which it was dissolved to the viscometer (B.5.1) under nitrogen pressure (the viscometer having been previously purged with nitrogen), so as to prevent any contact between the solution and the oxygen in the air.

B.6 Preparation of the test sample

See 9.4.

B.7 Procedure

B.7.1 Test portion

Weigh a mass of cotton corresponding to x mg of pure and dry cotton, where $x = \frac{30\,000}{\text{DP}}$, DP being the degree of polymerization assumed (if this is completely unknown, one or more preliminary tests must be carried out in order to determine its approximate value).

B.7.2 Determination

Place the test portion (B.7.1) in a 100 ml brown-glass volumetric flask. Add about 0,5 g of the copper(I) chloride (B.4.1) and 1,00 g ± 0,5 g of the copper(II) hydroxide (B.4.2). Make up to the mark with the ammonia solution (B.4.3) and close the flask by means of a rubber or plastic stopper.

Attach the flask to the shaker (B.5.3) and leave it to shake overnight. Fill the viscometer (B.5.1), preferably using the device (B.5.4). After stabilizing the temperature in the thermostatic bath (B.5.2) at 20 °C ± 0,1 °C, determine the flow time t of the cotton solution to the nearest 0,2 s. The viscometer shall be kept strictly vertical. In the same way, determine the flow time t_0 of a solution containing no cotton to the nearest 0,2 s.

B.8 Expression of results

The viscosity number (V.N.) is given by the formula

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

and the limiting viscosity number (L.V.N.) by the formula

$$\text{L.V.N} = \frac{\text{V.N.}}{1 + 0{,}28\,(\text{V.N.})\, c}$$

where c is the concentration, in grams per litre, of the cotton solution.

The mean degree of viscometric polymerization, $(\text{DP})_\text{B}$, of the cotton cellulose, measured under the conditions specified for the present method, is given by the formula

$$(\text{DP})_\text{B} = 2\,000\,(\text{L.V.N.})$$

Express the result to the nearest whole number.

Determine the mean degree of viscometric polymerization $(\text{DP})_\text{B}$ for each of three test pieces which have been subjected to 25 (or 50) laundering cycles and calculate the arithmetic mean of the three results to the nearest whole number. Do the same for three unlaundered test pieces. Convert $(\text{DP})_\text{B}$ into a fluidity value by means of the graph shown in figure B.1 in order to obtain $F_{25(50)}$ and F_0.

Calculate the chemical wear as indicated in 9.6.

State in the test report that this method has been used.

NOTES

1 In certain cases, the initial $(\text{DP})_\text{B}$ value may be determined in a similar way to that described in 6.5.3, note 3.

2 The curve in figure B.1 is based on numerous inter-laboratory tests carried out during the preparation of this International Standard.

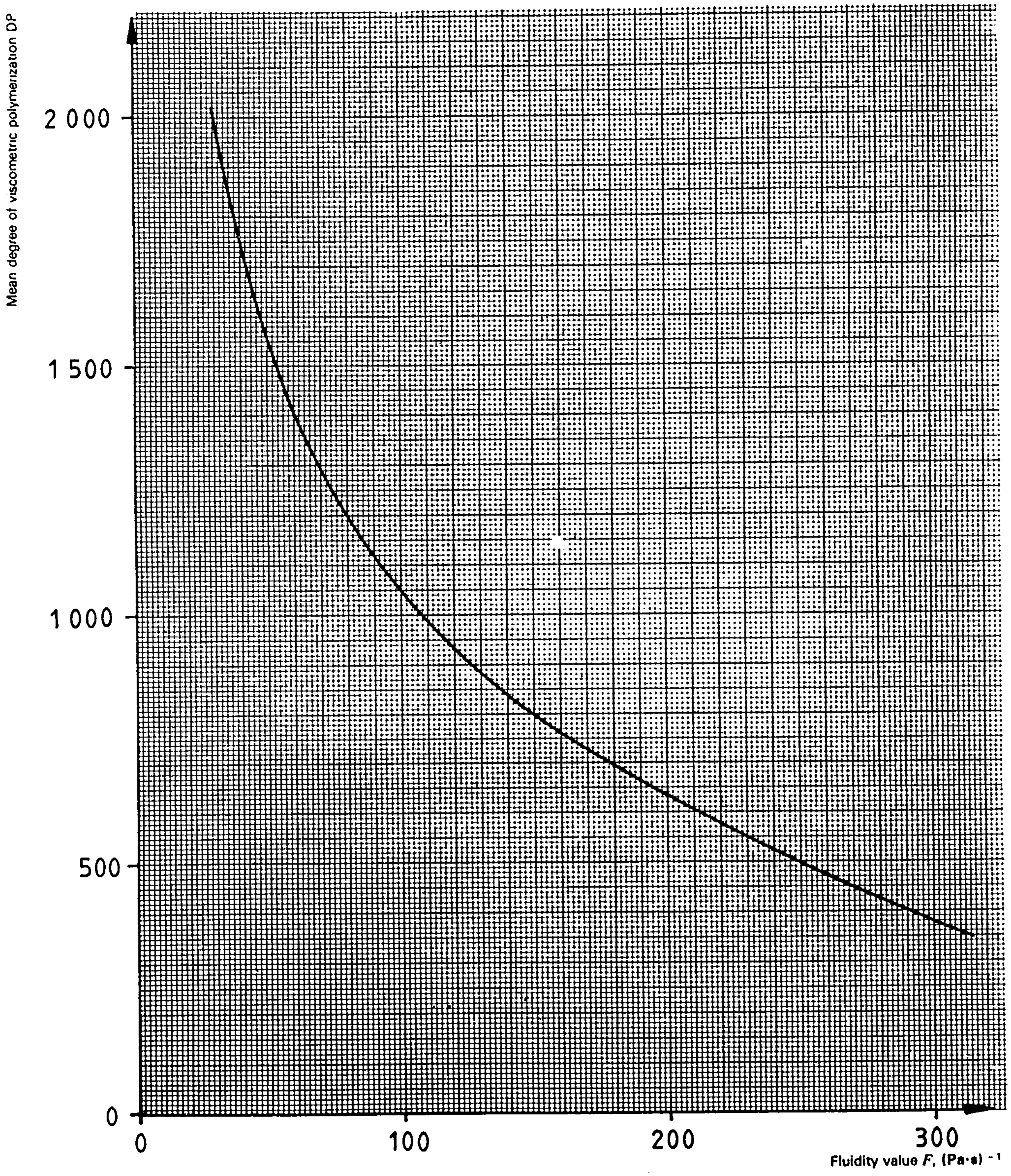

Figure B.1 — Mean degree of viscometric polymerization as a function of fluidity value (see clause B.8 and annex C, clause C.8)

Annex C
(normative)

Determination of chemical wear based on variation in average degree of viscometric polymerization in a copper(II) di(ethylenediamine) (Cuen) solution

(see 9.7)

C.1 Scope

This annex specifies a method for the determination of the chemical wear of 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 a copper(II) di(ethylenediamine) (Cuen) solution.

C.2 Definitions

For the purposes of this annex, the definitions given in annex B, clause B.2, apply.

In the present method, it is assumed that, in accordance with the Martin formula

$$\log_{10} (\text{V.N.}) = \log_{10} (\text{L.V.N.}) + 0{,}14\ (\text{L.V.N.})\ c$$

The average degree of viscometric polymerization DP is the ratio of the average viscometric relative molecular mass to the relative molecular mass of the polymerization unit (monomer) concerned (in this case anhydroglucose).

$(DP)_C$ is DP determined in accordance with this method, assuming that $(DP)_C = 133{,}3\ (\text{L.V.N.})$

C.3 Principle

Measurement of the viscosity ratio increment of a cellulose solution of known concentration (from which deposits have been removed, where necessary) in a standard copper(II) di(ethylenediamine) (Cuen) solution. Using this value, calculation of the limiting viscosity number by means of the Martin formula. Multiplication of the limiting viscosity number by 133,3 to obtain the mean degree of viscometric polymerization $(DP)_C$.

C.4 Reagents

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

C.4.1 Copper(II) hydroxide $[Cu(OH)_2]$ (see annex B, B.4.2).

Determine the copper content by iodometry.

C.4.2 Ethylenediamine $(NH_2CH_2CH_2NH_2)$, pure and colourless, or an aqueous solution thereof.

Where necessary, determine the ethylenediamine content by titration with a standard volumetric sulfuric acid solution, $c(H_2SO_4) = 0{,}5$ mol/l, using methyl orange as indicator.

C.4.3 Copper(II) di(ethylenediamine) $[Cu(NH_2CH_2CH_2NH_2)_2(OH)_2]$ (Cuen), standard solution, $c(C_4H_{18}O_2N_2Cu) = 0{,}5$ mol/l (about 109 g/l).

C.4.3.1 Preparation

Place a quantity of dry copper(II) hydroxide precipitate (C.4.1) corresponding to 32 g ± 0,5 g of copper, into a 1 000 ml one-mark volumetric flask. Add about 50 ml of water and a quantity of the ethylenediamine solution (C.4.2) corresponding to 60 g ± 0,5 g of the anhydrous product, and make up to the mark with water, keeping the temperature below 20 °C throughout. Pass a current of inert gas through the solution for 2 h in order to expel the air it contains.

C.4.3.2 Standardization

Add 4 ml of the solution prepared as in C.4.3.1 to 100 ml of water. Titrate with standard volumetric sulfuric acid solution, $c(H_2SO_4) = 0{,}5$ mol/l (using methyl orange as indicator or by means of a potentiometer to pH 3,05).

Add 25 ml of acetic acid solution, $c(CH_3COOH) = 2$ mol/l, and 3 g of potassium iodide. Titrate with standard volumetric sodium thiosulfate solution, $c(Na_2S_2O_3) = 0{,}1$ mol/l, until the colour of the iodine disappears (when the titration is nearly finished, starch may be added, followed by 1 g of potassium thiocyanate or ammonium thiocyanate).

C.4.3.3 Calculation

Calculate the ethylenediamine/copper ratio R as follows:

$$R = \frac{X}{Y}$$

where

X is the number of moles of ethylenediamine;

Y is the number of moles of copper;

$$X = \frac{V_1 c_1 - 2 V_2 c_2}{8}$$

$$Y = \frac{V_2 c_2}{4}$$

V_1 being the volume, in millilitres, of the sulfuric acid solution used;

V_2 being the volume, in millilitres, of the sodium thiosulfate solution used;

c_1 being the exact concentration, expressed in moles of H_2SO_4 per litre, of the sulfuric acid solution;

c_2 being the exact concentration, expressed in moles of $Na_2S_2O_3$ per litre, of the sodium thiosulfate solution.

The ratio R shall lie between 1,9 and 2,0.

C.5 Apparatus

C.5.1 Apparatus for dissolution of the cellulose, comprising the following items.

C.5.1.1 Glass or plastic vessel, the volume of which (25 to 50 ml) is selected according to the type of viscometer to be used. This vessel shall permit the exclusion of oxygen during the dissolution.

Various types of vessel are suitable.

a) A vessel in which the liquid fills the whole of the volume, contact between the air and the liquid being non-existent or negligible [see figure C.1a) and C.1b)]. The vessel shall contain glass balls (of diameter 4 mm to 7 mm) to enable the solution to be stirred.

b) A vessel in which air is replaced by an inert gas (nitrogen, hydrogen, etc.) [see figure C.1c)].

C.5.1.2 Stirring device, for example a magnetic stirrer [see figure C.1c)], which enables the liquid to be stirred mechanically during the dissolving process.

If the vessel is filled completely by the liquid [see C.5.1.1a)], it is recommended that stirring be effected by a gentle rotary movement ($0,5\ s^{-1}$ to $1\ s^{-1}$).

C.5.2 Apparatus for measurement of viscosity, comprising the following items:

C.5.2.1 Capillary viscometer, having the following essential characteristics:

- a precision capillary tube
- length of capillary tube: 150 mm ± 20 mm
- inside diameter of the capillary tube ($2r$): 0,45 mm ± 0,05 mm
- reference volume (V) between the two reference marks such that the flow time (t_0) of this volume of solvent lies between 70 s and 100 s

This reference volume is usually about 1 ml.

C.5.2.2 Thermostatic bath, capable of being controlled to within ± 0,1 °C of the required temperature.

Depending on the atmospheric conditions, the temperature shall be set to between 20 °C and 35 °C, preferably to 20 °C.

The shear rate G for the solvent under these conditions shall lie between $700\ s^{-1}$ and $850\ s^{-1}$, when calculated in accordance with the formula

$$G = \frac{4V}{\pi r^3 t_0}$$

where

G is expressed in seconds to the power of minus one;

V, r and t_0 are as defined in C.5.2.1 and are expressed in millilitres, centimetres and seconds, respectively.

If the operation has to be carried out under extreme conditions so far as temperature or the viscometer characteristics are concerned, it is necessary to check that the shear rate for the solvent does in fact lie between these limits.

C.6 Preparation of test sample

See 9.4.

C.7 Procedure

C.7.1 Test portion

Weigh a quantity of cotton corresponding to x mg of pure and dry cotton, where x is a function of the presumed limiting viscosity number of the cellulose and the volume of solvent. It is recommended that, for 100 ml

0,50 g of pure, dry cotton should be weighed for DPs between 100 and 300;

0,20 g of pure, dry cotton should be weighed for DPs between 300 and 700;

0,10 g of pure, dry cotton should be weighed for DPs between 700 and 1 500.

C.7.2 Determination

Place the test portion (C.7.1) into the vessel (C.5.1.1) to be used for the dissolution of the cellulose. Add the volume of Cuen solution (C.4.3), accurately measured, corresponding to the capacity of the vessel. Stir until the dissolution is complete.

Fill the viscometer (C.5.2.1) with the solution.

Place the viscometer in an upright position in the thermostatic bath (C.5.2.2). Allow the temperature of the solution to stabilize.

Raise the level of the liquid, by some suitable means, above the upper reference mark.

Allow the liquid to flow while measuring with a stopwatch the time taken by the meniscus to fall from the upper reference mark to the lower; this is noted as flow time t for the solution, to the nearest 0,1 s.

Ignore the first measurement.

Repeat the operation three times and note the three flow times, which should not differ from one another by more than 0,2 s; if there is a larger difference, repeat the test on a new test portion.

Under strictly similar conditions and using the same stopwatch, measure the flow time t_0 for the solvent that has been used to dissolve the cellulose.

C.8 Expression of results

The viscosity ratio increment is given by the formula

$$\frac{t - t_0}{t_0}$$

where

t is the flow time, in seconds, for the solution;

t_0 is the flow time, in seconds, for the solvent.

Calculate the concentration c, in grams, of pure, dry cotton per 100 ml of solution.

Determine the limiting viscosity number either by means of table C.1, or by using the curve in figure C.2.

Table C.1 — Limiting viscosity number as a function of the viscosity ratio increment

Calculated value for viscosity ratio increment $\frac{t - t_0}{t_0}$	Product of limiting viscosity number and concentration (L.V.N.)c
0,103 3	0,1
0,213 3	0,2
0,330	0,3
0,455	0,4
0,587	0,5
0,728	0,6
0,877	0,7
1,035	0,8
1,203	0,9
1,380	1,0
1,568	1,1
1,767	1,2
1,977	1,3
2,199	1,4
2,433	1,5

The mean degree of viscometric polymerization of the cotton cellulose $(DP)_C$, measured under the conditions specified for the present method, is given by the formula

$$(DP)_C = 133,3 \text{ (L.V.N.)}$$

Express the result to the nearest whole number.

Determine the mean degree of viscometric polymerization $(DP)_C$ for each of three test pieces that have undergone 25 (or 50) laundering cycles and calculate the arithmetic mean of the three results to the nearest whole number. Do the same for three unlaundered test pieces.

Within the scope of this International Standard, this method provides $(DP)_C$ values numerically equal to $(DP)_B$, taking into account the practical limits of error.

Convert the two mean values of $(DP)_C$ into fluidity values by means of the graph in figure B.1, thus obtaining $F_{25(50)}$ and F_0.

Calculate the chemical wear as indicated in 9.6. State in the test report that the present method has been used.

NOTE — See the notes to clause B.8 of annex B.

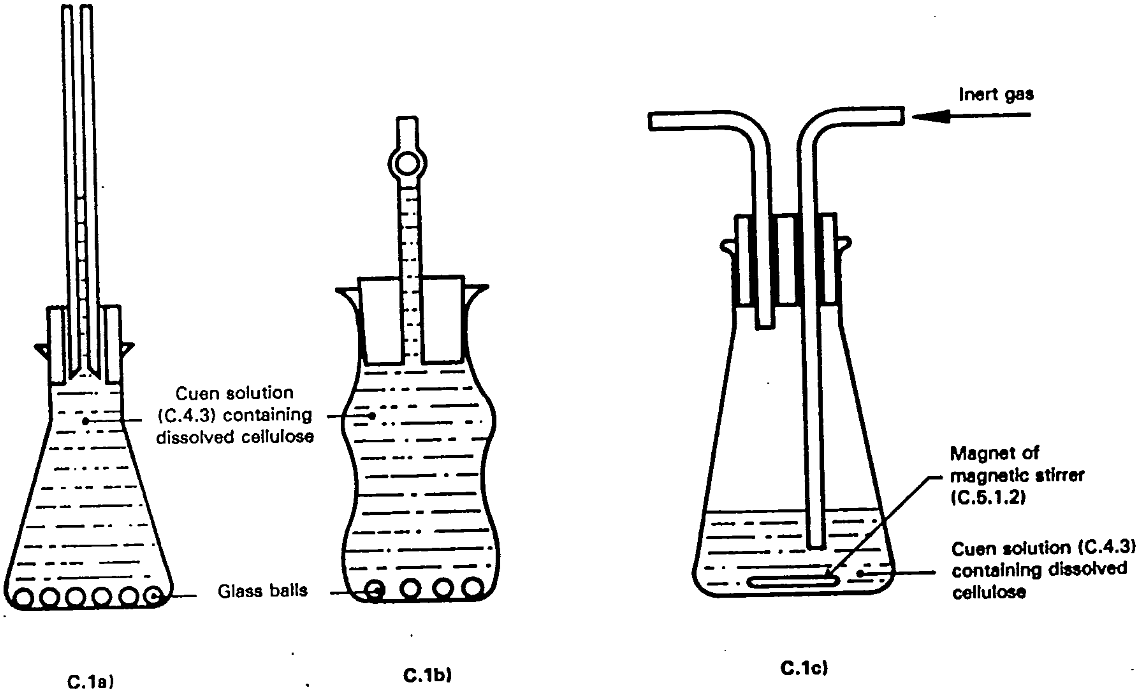

Figure C.1 — Examples of vessels for dissolution of cellulose (see C.5.1)

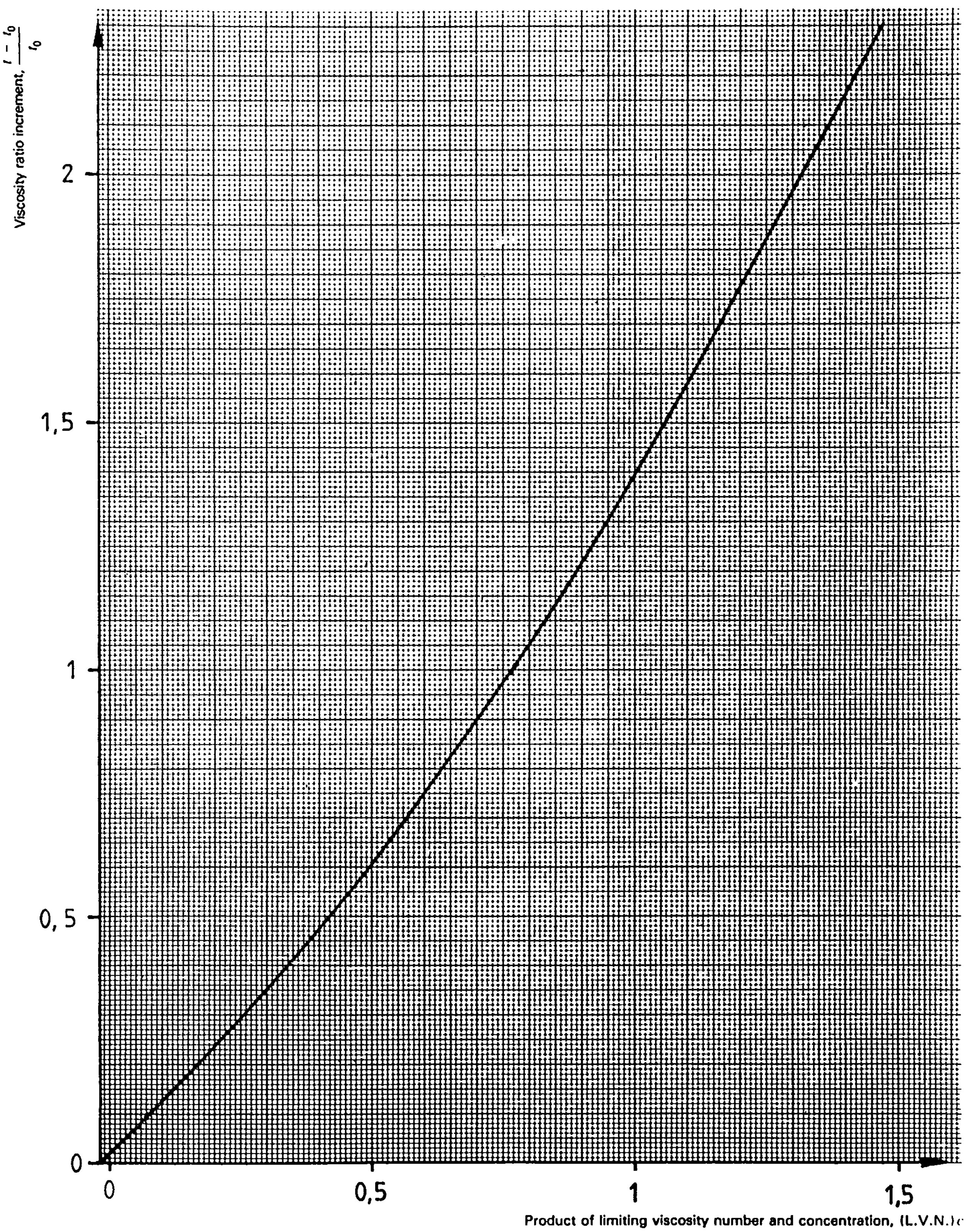

Figure C.2 — Curve showing the variation in the viscosity ratio increment of cellulose solutions in Cuen solution as a function of the product of the limiting viscosity number and the cellulose concentration in the solutions (see clause C.8)

Annex D
(informative)

Grey scale and measurement of reflection

(see 4.2.4, note 3)

D.1 General

When verifying certain effects of laundering on cloth, measurement of the luminance factor or the reflectance is essential in order to

a) assess the elimination of coloured pigment soils, which are generally grey, from soiled control cloths, i.e. to evaluate the effect of washing or bleaching, or a combination of the two;

b) assess the greying or colouring of white control cloths (independently of fluorescence), i.e. to evaluate the effect of redeposition of coloured pigment soils, which are generally grey, or chemical bleaching agents or a combination of the two;

c) assess the degree of whiteness, taking into account the contribution of the fluorescence of white control cloths, i.e. evaluate the quality of optical bleaching alone or in combination with the effect measured in b); in the absence of fluorescence, the total visual effect of the phenomena referred to in b) is evaluated.

Comparison of results from different laboratories is only valid when correct calibration is ensured.

D.2 Scope

This annex specifies, within the context of the verification of certain effects of laundering cloth, the conditions for measuring reflectance and the standards to be used in establishing a grey scale. It gives the definitions of units and phenomena encountered and also a list (incomplete) of the known measuring instruments in existence as well as a brief description of each.

D.3 Definitions

For the purposes of this annex, the following definitions apply.

D.3.1 Phenomena

D.3.1.1 reflection (CIE-45-05-060) 1): The return of radiation by a medium, without change of wavelength.

NOTES

1 The reflected radiation can be regular, diffuse or mixed.

2 One portion of the flux falling on a medium is reflected at the surface of the medium (surface reflection); another portion can be backscattered from the interior of the medium (volume reflection).

D.3.1.1.1 regular or specular reflection (CIE 45-20-010) 2): Reflection without diffusion in accordance with the laws of optical reflection as in a mirror.

D.3.1.1.2 diffuse reflection (CIE 45-20-015) 2): Diffusion by reflection in which, on the macroscopic scale, there is no regular reflection.

D.3.1.1.3 isotropic diffuse reflection (CIE 45-20-25) 1): Diffuse reflection in which the spatial distribution of the reflected radiation is such that the radiance (luminance) is the same in all directions of the hemisphere of incidence (in which the radiation is reflected).

D.3.1.1.4 mixed reflection (CIE 45-20-020) 1): Partly regular and partly diffuse reflection.

NOTE — The irradiance (illuminance) received from a point source, after regular reflection, varies inversely as the square of the distance from the image of the source and, after diffuse reflection, as the square of the distance from the diffuser.

D.3.1.2 radiance or luminance at a point of the source (CIE 45-05-150) 2) and (CIE 45-10-080) 2), L_v

See 4.2.1.

D.3.2 Physical quantities

D.3.2.1 reflectance (CIE 45-20-040) 1), ρ: The ratio of the reflected radiant (luminous) flux to the incident flux.

$$\text{Spectral reflectance } \rho(\lambda) = \frac{\Phi_{e\lambda\rho}}{\Phi_{e\lambda}}$$

where

$\Phi_{e\lambda\rho}$ is the reflected spectral radiant flux;

$\Phi_{e\lambda}$ is the incident spectral radiant flux.

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1) See CIE Publication No. 38 (TC-2.3.).

2) See CIE Publication No. 17 (E-1.1.).

NOTE — Where mixed reflection (D.3.1.1.4) occurs, the (total) reflectance may be divided into two parts: the regular reflectance, ϱ_r, and diffuse reflectance, ϱ_d, corresponding respectively to the two modes of reflection referred to in D.3.1.1.1 and D.3.1.1.2.

$$\varrho = \varrho_r + \varrho_d$$

D.3.2.2 reflectivity (CIE 45-20-050)[1], ϱ_∞: The reflectance of a layer of material of such a thickness that there is no change of reflectance with increase of thickness.

D.3.2.3 radiance (luminance) factor (CIE 45-20-200)[1], β

See 4.2.2.

D.3.2.4 reflectance factor (CIE 45-20-201)[1], R: The ratio of the radiant (luminous) flux reflected in the directions delimited by the cone to that reflected in the same directions by a perfect reflecting diffuser identically irradiated (illuminated).

NOTES

1 For specularly and very concentratedly reflecting surfaces that are irradiated (illuminated) by a source of small solid angle, the reflectance may be much greater than 1 if the cone includes the mirror image. In this case, the value of the reflectance obtained with a measuring instrument depends on the measuring geometry (specifically sample size, source-sample distance, and sample-receptor distance) unless the sample size is sufficiently small to permit all of the specularly reflected flux to be incident on the receiver.

2 If the solid angle of the cone approaches 2π sr, the reflectance factor approaches the reflectance. In this case, a regular component (if present) must be included. This can be excluded if the regular component is sufficiently defined and a suitable trap is used, then the reflectance factor approaches the diffuse reflectance.

3 If the solid angle of the cone approaches 0 sr, the reflectance factor approaches the radiance (luminance) factor provided there is no regular reflection in the direction of observation.

4 The term "directional reflectance" is used currently in the United States in this sense.

5 It is recommended that the reflectometer value (D.3.2.6) be used rather than reflectance factor.

D.3.2.5 spectral reflectance factor: The value measured by means of a commercial spectrophotometer, at a given wavelength, and which depends on the geometrical properties of the instrument.

D.3.2.6 reflectometer value (CIE 45-20-202)[1]

See 4.2.5.

D.3.2.7 The considerations discussed above are illustrated by table D.1.

D.3.3 Measurement geometries

D.3.3.1 45°/normal (abbreviation: 45/0) (radiance factor $\beta_{45/0}$): Geometry in which the specimen is illuminated by one or more beams whose axes are at an angle of 45° ± 5° from the normal to the specimen surface.

The angle between the direction of viewing and the normal to the specimen should not exceed 10°. The angle between the axis and any ray of an illuminating beam should not exceed 5°. The same restriction should be observed in the viewing beam.

D.3.3.2 normal/45° (abbreviation: 0/45) (radiance factor $\beta_{0/45}$): Geometry in which the specimen is illuminated by a beam whose axis is at an angle not exceeding 10° from the normal to the specimen.

The specimen is viewed at an angle of 45° ± 5° from the normal. The angle between the axis and any ray of the illuminating beam should not exceed 5°. The same restriction should be observed in the viewing beam.

D.3.3.3 diffuse/normal (abbreviation: d/0) (radiance factor $\beta_{d/0}$): Geometry in which the specimen is illuminated diffusely by an integrating sphere (Ulbricht sphere).

Table D.1

Quantity	Value measured	
	Perfect diffuser	**Perfect mirror**
Reflectance	1,00	1,00
Diffuse reflectance	1,00	0
Regular reflectance	0	1,00
Radiance factor	1,00	Not defined, as it depends on the measuring geometry and may vary from 0 to a value much greater than 1
Reflectometer value	1,00	Depending on the measuring geometry, < 1 or > 1
Spectral reflectance factor	1,00	Depending on the measuring geometry, < 1 or > 1

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[1] See CIE Publication No. 38 (TC-2.3.).

The angle between the normal of the specimen and the axis of the viewing beam should not exceed 10°. The integrating sphere may be of any diameter provided the total area of the ports does not exceed 10 % of the total internal reflecting sphere area.

The angle between the axis and any ray of the viewing beam should not exceed 5°. According to Helmholtz reciprocity law, the radiance factor $\beta_{d/0}$ is identical to the reflectance ϱ for directional normal incidence.

D.3.3.4 normal/diffuse (abbreviation: 0/d) (radiance factor $\beta_{0/d}$): Geometry in which the specimen is illuminated by a beam whose axis is at an angle not exceeding 10° from the normal to the specimen.

The reflected beam is collected by the integrating sphere. The angle between the axis and any ray of the illuminating beam should not exceed 5°.

The integrating sphere may be of any diameter provided the total area of the ports does not exceed 10 % of the internal reflecting sphere area.

NOTES

1 In view of the variety of geometric conditions permitted in colour measurement, radiance factor is not always measured; in some cases reflectance factor is measured instead.

2 In measurements using a biconical geometry, the reflectance factor can exceed unity, whereas the reflectance cannot. For colorimetric measurements in which the measurement of properties greater than 1 may prove impractical, it is recommended that the reflectance be measured.

3 When measuring glossy surfaces using geometry 0/d (D.3.3.4) it is recommended not to illuminate them exactly at right angles. In many instruments, an illumination angle of 8° has been chosen.

4 When using certain commercial instruments it is not possible to comply exactly with the conditions defined above.

D.4 Apparatus[1]

D.4.1 Spectrophotometers

These instruments are not necessary for ordinary measurements for verifying the effects of laundering. Theoretically, they allow measurements to be carried out at a given wavelength and therefore better reproducibility between different laboratories and different instruments should be expected. In fact, fluorescent samples may give very different results (see clause D.6).

D.4.1.1 Zeiss spectrophotometers

D.4.1.1.1 Zeiss PMQ II with reflecting device

Spectrophotometer with double prism radiation selector and wide spectral range (200 nm to 2 000 nm). Illumination by monochromatic or polychromatic light. Different geometries are possible depending on the reflecting devices used:

- RA 2 with geometry 45/0 (monochromatic light);
- RA 3 with geometry d/0 (polychromatic light) or 0/d (monochromatic light).

Manual operation. No recorder.

Possibility of measuring fluorescent samples subject to the use of a suitable attachment.

D.4.1.1.2 Zeiss RFC 3

This instrument is used in particular for the automatic measurement of colours. Spectral selection is by 24 or 21 interference filters, distributed uniformly between 400 nm and 700 nm. The pass band width is approximately 10 nm. Alternatively three tristimulus filters may be used. Polychromatic illumination by D_{65} CIE standard illuminant (CIE 45-15-145) (illumination is also possible with illuminant A). The spectral range of low wavelengths, below 400 nm and 460 nm, may be masked with a filter of geometry d/0 (D.3.3.3) (diffuse illumination and observation at an angle of 8°). Alternatively, a 45/0 device may be used for illuminant A. Manual operation (digital read-out) or automatic (if the instrument is connected to a digital computer). Circular measuring surface with diameter varying from 30 mm to 5 mm.

D.4.1.1.3 Zeiss RFC 16

Instrument identical with the Zeiss RFC 3 (D.4.1.1.2) but completely automatic. With 16 interference filters for 400 nm to 700 nm. Geometry d/8.

D.4.1.1.4 Zeiss DMC 25

Spectrophotometer with double prism radiation selector for the automatic measurement of colours with either an incandescent lamp or a xenon lamp, and measuring geometry of 0/d or d/0 and more recently 45/0; the diameter of the sample must be at least 0,5 cm.

Possibility of measuring fluorescent samples with geometry d/0. Possibility of recording.

D.4.1.1.5 Zeiss DMC 26

Instrument identical with the Zeiss DMC 25 (D.4.1.1.4) but with a double radiation selector with gratings.

D.4.1.2 Pretema FS 4

Instrument comparable to Zeiss RFC 3 (D.4.1.1.2) with 35 interference filters, uniformly distributed between 380 nm and 720 nm, pass band width approximately 10 nm. Illumination by polychromatic light corresponding to CIE standard illuminant

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[1] The instruments mentioned in this clause are examples of suitable apparatus available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these instruments.

D_{65}. Xenon lamp geometry d/0 with a small observation cone solid angle (3°) and a large sphere (diameter 33 cm). Circular measuring surface, diameter 10 mm to 40 mm. Automatic operation by digital computer with recorder

D.4.1.3 Datacolor spectrophotometers

D.4.1.3.1 Datacolor 7080

Instrument comparable to Zeiss RFC 3 (D.4.1.1.2) but with a continuous interference filter of 380 nm to 700 nm, pass band width approximately 10 nm; 16, 32 or 63 measuring points.

D.4.1.3.2 Datacolor 3080

Instrument comparable to Datacolor 7080 (D.4.1.3.1).

D.4.1.4 Other automatic and non-automatic spectrophotometers

D.4.1.4.1 Diano Match Scan, for fluorescent and non-fluorescent samples.

D.4.1.4.2 Beckmann DB-G, for non-fluorescent samples only.

D.4.1.4.3 Beckmann DU with reflector

One of the oldest instruments of this type. Monochromatic illumination. Geometry 0/45. One advantage is that the reflected light is collected by an annular mirror, such that a mean of all the horizontal observation directions is obtained. No recorder.

D.4.1.4.4 Spectronic 505, for non-fluorescent samples; measuring range may go up as far as UV.

D.4.1.4.5 Carry model 14, for non-fluorescent samples; measuring range may be from UV up to IR.

D.4.1.4.6 Macbeth MS 2000, with gratings and xenon pulse lamp with filter for CIE standard illuminant D_{65}, including UV.

D.4.1.4.7 Hunter D 54 P-5, with continuous interference filter, geometry d/8 halogen lamp with filter for illuminant D_{65}. Automatic operation. For fluorescent and non-fluorescent samples.

D.4.2 Reflectometers and tristimulus colorimeters

These instruments fulfil the requirements for the verification of effects on cloth of laundering with detergents. Thin band or line interference filters are used or wide band absorbing filters which overlap. These instruments are therefore also called photometers. If the instrument has only very few filters, it may be called a reflectometer and used to measure spectral reflectometer values at wavelengths specified for the filters

The use of filters is necessary in order to be able to measure coloured samples, e.g. to assess yellowing. The measurements carried out on coloured samples may differ greatly from one instrument to another, which also applies to the spectrophotometers described in D.4.1, although to a lesser extent.

Tristimulus filters are so constructed that, in conjunction with the photoelectric detector, they give values weighted according to the CIE colorimetric functions. Generally, the sample is illuminated with standard illuminant A (incandescent lamp) and the results of measurement should be valid for standard illuminant D_{65} or C (daylight).

The transformation of the spectral distribution of illuminant A into that of illuminant D_{65} or C is also carried out by the tristimulus filters. CIE standard illuminant D_{65} has recently been standardized for daylight and it should replace the older standard illuminant C (still in use).

In practice, therefore, the instruments give tristimulus values X, Y, Z for the CIE-1931 2° observer with illuminant D_{65} or C, or for the CIE-1964 10° observer with illuminant D_{65}. (In 1931 an angle of observation of 2° was fixed, which corresponds approximately to the visual field of a fairly large coin at a normal distance of observation. In 1964, however, the visual field was fixed at 10°, because colours are then assessed by the eye with a different distribution of sensitivity.)

Amongst the currently available instruments, attention is drawn to the following.

D.4.2.1 Zeiss Elrepho

Illumination by polychromatic light from an incandescent lamp or a xenon lamp reproducing CIE standard illuminant D_{65}. Possibility of inserting 2 filters, one cutting off radiation below 400 nm and the other that below 460 nm. Geometry d/0 (aperture of the reflected beam 7°). Illumination is not however strictly diffuse (see D.3.3.3) nor identical for the two methods of illumination. Filter holder for seven band pass filters of 380 nm to 780 nm. Possibility of including tristimulus filters either for illuminant C (in conjunction with the incandescent lamp) or for CIE standard illuminant D_{65} (in conjunction with the xenon lamp) or for illuminant A (in conjunction with the incandescent lamp).

Measuring ports 34 cm in diameter, or rectangular measuring 9,5 mm × 27,5 mm.

Very accurate, but fairly slow, instrument. Very suitable for measuring fluorescence and the whiteness of fluorescent samples.

D.4.2.2 Zeiss Elrephomat DFC 5

Instrument identical to the Zeiss Elrepho (D.4.2.1) but completely automatic with tristimulus filters. Geometry d/8.

D.4.2.3 Hunterlab D 25

Polychromatic illumination with incandescent lamp and the possibility of using UV filters. Geometry 45/0, with illumination from two opposite sides, or circular. Possibility of using a geometry of 0/d. Four interference filters for CIE standard illuminant C

Measuring surface with a diameter of 100 mm, 50 mm or 12 mm.

Accurate and rapid instrument; not recommended for measuring the degree of whiteness of fluorescent samples.

D.4.2.4 Datacolor 3800

With continuous interference filters of 350 nm to 700 nm.

D.4.2.5 Other instruments

D.4.2.5.1 EEL (Evans Electroselenium Ltd) Reflectometer.

D.4.2.5.2 Photovolt 610 Reflectometer.

D.4.2.5.3 Hilger Interference Colorimeter.

D.4.2.5.4 Macbeth MC 1010.

D.5 Calibration

The accuracy of reflectometer measurements depends on exact calibration using carefully chosen standards.

D.5.1 Theoretical standards

Such standards cannot be made.

D.5.1.1 Perfect reflecting diffuser

Ideal isotropic diffuser, with a reflectance (transmittance) equal to 1.

D.5.1.2 Black body

Body which completely absorbs the incident radiation, irrespective of its wavelength, direction and polarization.

D.5.2 Practical standards — grey scale

If the response of the instruments used were completely linear, two measurements, preferably as close as possible to the matt white surface or the black body, would be enough to calibrate the scale. Unfortunately this does not happen often, especially with a simple reflectometer. Calibration therefore has to be carried out by means of several operations at regular intervals in order to carry out any corrections which may be necessary.

From the above, it seems that the standards used should themselves be calibrated in the geometrical measuring conditions which are usual for the instrument to be calibrated. This is all the more important if the standards are very glossy, i.e. if the directional reflection is very strong. Glossy standards keep more easily and look better that matt standards, however they are less suitable.

Fortunately, the best white standards (made from compressed mineral powders) and the best black standards (hollow black bodies) are very good diffusers.

D.5.2.1 Compressed barium sulfate

This standard is obtained by compression (at an adequate pressure) or very pure barium sulfate into small discs approximately 5 mm thick. Its reflectance is approximately 0,985, and its radiance factor is approximately 1,01 for geometry 45/0. In comparison with compressed magnesium sulfate, it has the advantage of remaining stable for several days and weeks, when kept away from the light and dust.

D.5.2.2 Black body

It is impossible to make black surfaces with a reflectance of less than approximately 0,02. On the other hand, a hollow black body gives a reflectance of almost zero. Its internal volume should be as large as possible. Alternatively, a lamellar surface may be used. The internal coating must be black and matt.

D.5.2.3 Opal glass

Some firms supply opal glass calibrated for spectrophotometers and reflectometers.

D.5.2.4 Miscellaneous standards

Some manufacturers of instruments for colorimetry supply standards made of enamelled metal, ceramic, etc. These standards can only be used with the instruments for which they are intended.

Glossy standards (ceramic or enamel) are only suitable for instruments in which regular reflection does not take place at all, and if they themselves have been calibrated in the same conditions.

D.5.3 Recommendations for calibration

In order to calibrate a reflectometer or colorimeter with the required high accuracy, it is essential to have accurately calibrated standards and to know exactly the measuring conditions of the instrument to be calibrated, and those used during calibration of the standard. The difficulties can be reduced by choosing an instrument with a photometric measuring scale which is as linear as possible, as accurate calibration is then possible with only two measurements. Finally, the measurements must be interpreted with extreme caution for glossy and semi-glossy samples. It is always best to work with matt samples.

It is very important that the standards are as similar as possible to the sample, both in the nature of their surface and in their colour. Barium sulfate is to be recommended as a standard, particularly when working with several samples, because compressed discs made from it have an almost ideal matt surface.

D.6 Measurement of fluorescent samples

Cloths which contain an optical whitening agent are fluorescent. This fluorescence is caused by ultraviolet and short-wavelength visible radiation which is always present in daylight and in smaller proportions in the radiation from all artificial light sources.

If a cloth of this nature is illuminated by such radiation, it reflects part of this radiation and emits a supplementary radiation as a result of fluorescence. These two different types of radiation cannot be separated by the eye. They give a total light, whose spectral distribution is a function of the reflected and emitted radiation.

In such cases, the radiance and luminance factors measured depend on the spectral distribution of the illuminating light source. Reproducible values can therefore only be obtained with a standard illuminant.

Since 1964, the CIE has recommended standard illuminant D_{65} representing daylight at a colour temperature approximately equal to 6 504 K and whose relative radiance distribution has been fixed at between 320 nm and 780 nm. This distribution may be obtained in a satisfactory manner by means of a xenon lamp with filters.

If a fluorescent cloth is illuminated by monochromatic or almost monochromatic radiation, and the total reflected light is measured, there are two possibilities:

- The radiation causes fluorescence at various wavelengths, which may be picked up when measuring polychromatic radiation. In this case, the radiance factor measured is generally greater than the radiance factor for spectral reflection at this wavelength.
- The radiation does not cause fluorescence. In this case, the measured spectral radiance factor is generally smaller than the "total" factor measured at the corresponding wavelength with polychromatic illumination. Only reflection is measured at this wavelength and not any fluorescence which has been caused by radiation at other wavelengths.

When studying washing, one is in the first place interested in the effects of laundering, i.e. only the action of surface active agents contained in the detergents, or of the washing auxiliaries, on the removal of soils. If the detergents examined contain optical whitening agents, a greater brightening of the cloth is observed than that obtained by the active washing substances themselves. The fluorescent light emitted by the optical whitening agents should therefore be excluded. In this case two methods can be used:

a) Illumination with polychromatic light, but from which the exciting radiation has been removed (by means of suitable filters). Theoretically, this is not entirely possible. In practice, one can do it with a compromise which leaves a weak fluorescence at short wavelengths. If measurements are carried out at long wavelengths, they correspond, in practice, only to the usual reflection.

b) Long-wavelength monochromatic light is used so that fluorescence is not caused. Thus only the usual reflection is measured.

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