

INTERNATIONAL STANDARD

**ISO
4312**

Second edition
1989-09-01

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*



Reference number
ISO 4312 : 1989 (F)
AVA Documents
Document Sharing Hub

Contents

	page
Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Selection of specimens and samples from test pieces	1
4 Determination of intrinsic greying (greying measured in the absence of ultraviolet radiation)	1
5 Determination of intrinsic yellowing (yellowing measured in the absence of ultraviolet radiation)	4
6 Determination of increase in organic deposit content	5
7 Determination of increase in incineration residue (ash)	7
8 Determination of overall decrease in breaking strength (total wear)	8
9 Determination of decrease in breaking strength resulting from chemical degradation of cellulose due to laundering (chemical wear)	9
10 Determination of decrease in breaking strength resulting from mechanical factors in laundering (mechanical wear)	14
11 Test report	14
 Annexes	
A Determination of increase (or decrease) in degree of whiteness	16
B Determination of chemical wear based on variation in average degree of viscometric polymerization in an ammonium copper(II) hydroxide solution	18

C	Determination of chemical wear based on variation in average degree of viscometric polymerization in a copper(II) di(ethylenediamine) (Cuen) solution	21
D	Grey scale and measurement of reflection	26

Figures

1	Viscometer	12
2	Supporting tube for viscometer	12
B.1	Mean degree of viscometric polymerization as a function of fluidity value ...	20
C.1	Examples of vessels for dissolution of cellulose	24
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	25

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 laundings.

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 affects 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 selvages. 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 whiteness (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 45-05-150)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 element 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.

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

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

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 $103\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

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 $103\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, allowed to cool in a desiccator and weighed. Place the weighing bottle and its stopper separately in the oven (6.4.3), set at $103\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, 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 re-established.

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 $103\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, allowed to cool in a desiccator and weighed, and the condenser (6.4.1.3) to the extractor, then pour in a suf-

ficient 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 $103\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, 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 $103\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, 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, $20\text{ }^{\circ}\text{C}$).

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 $\pm$ 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 $\pm$ 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 $\pm$ 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;