INTERNATIONAL STANDARD

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Textiles — Quantitative chemical analysis —

Part 22: Mixtures of viscose or certain types of cupro or modal or lyocell and flax fibres (method using formic acid and zinc chloride)

Textiles — Analyse chimique quantitative —

Partie 22: Mélanges de viscose ou de certains types de cupro, modal ou lyocell et de fibres de lin (méthode à l'acide formique et au chlorure de zinc)

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 1833-22 was prepared by Technical Committee ISO/TC 38, *Textiles*.

ISO 1833 consists of the following parts, under the general title *Textiles — Quantitative chemical analysis*:

— *Part 1: General principles of testing*

— *Part 2: Ternary fibre mixtures*

— *Part 3: Mixtures of acetate and certain other fibres (method using acetone)*

— *Part 4: Mixtures of certain protein and certain other fibres (method using hypochlorite)*

— *Part 5: Mixtures of viscose, cupro or modal and cotton fibres (method using sodium zincate)*

— *Part 6: Mixtures of viscose or certain types of cupro or modal or lyocell and cotton fibres (method using formic acid and zinc chloride)*

— *Part 7: Mixtures of polyamide and certain other fibres (method using formic acid)*

— *Part 8: Mixtures of acetate and triacetate fibres (method using acetone)*

— *Part 9: Mixtures of acetate and triacetate fibres (method using benzyl alcohol)*

— *Part 10: Mixtures of triacetate or polylactide and certain other fibres (method using dichloromethane)*

— *Part 11: Mixtures of cellulose and polyester fibres (method using sulfuric acid)*

— *Part 12: Mixtures of acrylic, certain modacrylics certain chlorofibres, certain elastanes and certain other fibres (method using dimethylformamide)*

— *Part 13: Mixtures of certain chlorofibres and certain other fibres (method using carbon disulfide/ acetone)*

— *Part 14: Mixtures of acetate and certain chlorofibres (method using acetic acid)*

— *Part 15: Mixtures of jute and certain animal fibres (method by determining nitrogen content)*

— *Part 16: Mixtures of polypropylene fibres and certain other fibres (method using xylene)*

— *Part 17: Mixtures of chlorofibres (homopolymers of vinyl chloride) and certain other fibres (method using sulfuric acid)*

— *Part 18: Mixtures of silk and wool or hair (method using sulfuric acid)*

— *Part 19: Mixtures of cellulose fibres and asbestos (method by heating)*

— *Part 20: Mixtures of elastane and certain other fibres (method using dimethylacetamide)*

— *Part 21: Mixtures of chlorofibres, certain modacrylics, certain elastanes, acetates, triacetates and certain other fibres (method using cyclohexanone)*

— *Part 22: Mixtures of viscose or certain types of cupro or modal or lyocell and flax fibres (method using formic acid and zinc chloride)*

— *Part 24: Mixtures of polyester and some other fibres (method using phenol and tetrachloroethane)*

— *Part 25: Mixtures of polyester and cotton or aramid fibres (method using trichloracetic acid and chloroform)*

— *Part 26: Mixtures of melamine and cotton or aramide fibres (method using hot formic acid)*

The following part is cancelled:

— *Part 23: Mixtures of polyethylene and polypropylene (method using cyclohexanone)*

Textiles — Quantitative chemical analysis —

Part 22: Mixtures of viscose or certain types of cupro or modal or lyocell and flax fibres (method using formic acid and zinc chloride)

WARNING — This part of ISO 1833 calls for the use of substances/procedures that may be injurious to the health/environment if appropriate conditions are not observed. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety/environment at any stage.

1 Scope

This part of ISO 1833 is applicable, after removal of non-fibrous matter, to binary mixtures of

— viscose or certain types of the current cupro or modal or lyocell fibres

with

— flax fibres.

If a cupro or modal fibre is found to be present, a preliminary test should be carried out to see whether it is soluble in the reagent.

The part of ISO 1833 is not applicable to mixtures in which the flax fibre has suffered extensive chemical degradation, nor when the viscose, cupro, modal or lyocell fibre is rendered incompletely soluble by the presence of certain permanent finishes or reactive dyes that cannot be removed completely.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1833-1:2006, *Textiles — Quantitative chemical analysis — Part 1: General principles of testing*

3 Principle

After the removal of the non-cellulosic components (pectin, etc.) related to the flax fibre internal structure by means of pre-treatment with sodium hydroxide, the viscose, cupro or modal or lyocell fibre is dissolved out from a known dry mass of the mixture, with a reagent composed of formic acid and zinc chloride. The residue is collected, washed, dried and weighed; its corrected mass is expressed as a percentage of the dry mass of the mixture. The percentage of viscose, cupro, modal or lyocell fibre is found by difference.

4 Reagents and apparatus

4.1 General

Use the reagents and the apparatus described in ISO 1833-1, together with those specified in 4.2 and 4.3.

4.2 Reagent

4.2.1 Sodium hydroxide solution of 1,5 mol/l.

4.2.2 Acetic acid solution of 0,1 mol/l.

4.2.3 Formic acid/zinc chloride reagent.

Prepare a solution containing 20 g of anhydrous zinc chloride and 68 g of anhydrous formic acid, made up to 100 g with water.

SAFETY PRECAUTIONS — The harmful effects of this reagent shall be borne in mind, and full precautions shall be taken during use.

4.2.4 Ammonia, dilute solution.

Dilute 20 ml of concentrated ammonia solution (ρ = 0,880 g/ml) to 1 I with water.

4.3 Apparatus

4.3.1 Flask with ground glass stopper, of minimum capacity 250 ml.

4.3.2 Reflux condenser.

4.3.3 Heating apparatus capable of maintaining the boiling of the content of the flask during the pre-treatment.

4.3.4 Conical flask, of minimum capacity 200 ml, glass-stoppered.

4.3.5 Heating apparatus, capable of maintaining the temperature of the flask at (40 ± 2) °C.

5 Test procedure

5.1 General

Follow the general procedure described in ISO 1833-1, and then proceed as follows.

5.2 Removal of the non-cellulosic components of the flax fibres

Based on the intensity of the flax retting and of the applied treatments, from 10 % to 40 % of non-cellulosic components (pectin, etc.) of the flax fibres shall be removed prior to the dissolution of viscose or cupro or modal or lyocell.

Place at least 1 g of the specimen in a weighed filter crucible.

Place and dry the weighed filter crucible and the specimen in an oven, then cool down and weigh.

Allow the sodium hydroxide solution to boil for at least 15 min in the flask equipped with a reflux condenser. The bath ratio of the solution to the specimen shall be at 1/100.

After removal of air from the solution (due to boiling), place the specimen in the flask and continue boiling for 1 h. Wet out constantly the specimen in the solution.

Rinse the specimen by continuous siphoning with distilled or deionized water for 5 min.

Immerse the specimen in the acetic acid solution for 10 min.

Filter the contents of the flask through the weighed filter crucible and wash any fibres from the flask into the crucible with 0,1 mol/l acetic acid solution.

Drain the crucible with suction and wash with water until neutralization is achieved. Do not apply suction until the washing liquor has drained under gravity.

Finally, drain the crucible with suction, dry the crucible and residue, cool and weigh them.

5.3 Dissolution of viscose, cupro, modal or lyocell fibre

Place the pre-treated specimen without delay in the conical flask pre-heated to 40 °C.

Weigh again the filter crucible because of remaining loose fibres after the transfer.

Determine the dry mass of the mixture before the dissolution test.

Add 100 ml of formic acid/zinc chloride reagent per gram of specimen, pre-heated to 40 °C.

Stopper the flask and shake it.

Allow the flask and contents to remain at 40 °C for 2 h 30 min, shaking it twice during this time at intervals of about 60 min.

Filter the contents of the flask through the weighed filter crucible and wash any fibres from the flask into the crucible with the reagent.

Rinse with a further 20 ml of reagent.

Wash the crucible and residue thoroughly with water at 40 °C.

Rinse the residue with 100 ml of cold ammonia solution, ensuring that the residue remains totally immersed in the solution for 10 min, and then rinse with cold water. (Do not apply suction until each washing liquor has drained under gravity.)

Finally, drain the crucible with suction, dry the crucible and residue, and cool and weigh them.

Examine the residue microscopically, or otherwise, as appropriate, to check that the treatment has in fact completely removed the soluble fibre [see 9.2 Testing execution in ISO 1833-1:2006].

6 Calculation and expression of results

6.1 Calculation of loss in mass during pre-treatment

$$P_s = \frac{m_1 - m_2}{m_1} \times 100$$

where

P_s is the percentage of loss in mass during pre-treatment with sodium hydroxide;

m_1 is the dry mass of the test specimen before the pre-treatment with sodium hydroxide;

m_2 is the dry mass of the test specimen after the pre-treatment with sodium hydroxide.

6.2 Calculation of dry mass of after-transfer mixture corrected to its initial dry mass before pre-treatment

The dry mass of the after-transfer mixture corrected to its initial dry mass before the pre-treatment with sodium hydroxide shall be calculated using the following formula:

$$M = \frac{100\, m_3}{100 - P_s}$$

where

M is the dry mass of the mixture corrected to its initial dry mass before the pre-treatment with sodium hydroxide and corrected for the loss in mass during the transfer;

m_3 is the dry mass of the mixture for the dissolution test;

P_s is the percentage of loss in mass during pre-treatment with sodium hydroxide.

6.3 Calculation of dry masses of viscose or cupro or modal or lyocell and pretreated flax fibres

The dry masses of the viscose or cupro or modal or lyocell and pretreated flax fibres shall be calculated using the following formulae:

$$v = d_1 \left(m_3 - d_2\, m_4 \right)$$

$$f = M - v$$

where

v is the dry mass of viscose or cupro or modal or lyocell in the mixture for the dissolution test;

f is the dry mass of pretreated flax fibre in the mixture for the dissolution test;

M is the dry mass of the mixture corrected to its initial dry mass before the pre-treatment and corrected to the loss in mass during transfer;

m_3 is the dry mass of the mixture for dissolution testing;

m_4 is the dry mass of the residue after testing;

d_1 is a correction factor for loss in mass of the viscose or cupro or modal or lyocell during pre-treatment;

d_2 is the correction factor for loss in mass of flax fibres with formic acid/zinc chloride reagent.

The values of d_1 and d_2 are respectively 1,05 and 1,00 for viscose or cupro or modal or lyocell and flax fibre, both in raw condition.

The values of d_1 and d_2 are respectively 1,16 and 1,02 for viscose or cupro or modal or lyocell and flax fibre, both in bleached condition.

6.4 Calculation of the percentages of each component with agreed percentage additions for moisture

The percentage of each component in the mixture with agreed percentage addition for moisture shall be calculated using the following formulae:

$$V = \frac{v\left(1+\frac{tc_1}{100}\right)}{v\left(1+\frac{tc_1}{100}\right)+f\left(1+\frac{tc_2}{100}\right)}\times 100$$

$$F = \frac{f\left(1+\frac{tc_2}{100}\right)}{v\left(1+\frac{tc_1}{100}\right)+f\left(1+\frac{tc_2}{100}\right)}\times 100$$

where

V is the percentage of viscose or cupro or modal or lyocell fibre in the mixture for the dissolution test;

F is the percentage of flax fibre with agreed percentage addition for moisture;

v is the dry mass of viscose or cupro or modal or lyocell for the dissolution test;

f is the dry mass of flax for the dissolution test;

tc_1 is the agreed percentage addition for moisture added to the dry mass of viscose or cupro or modal or lyocell fibre;

tc_2 is the agreed percentage addition for moisture added to the dry mass of pre-treated flax fibre.

7 Precision

On a homogeneous mixture of textile materials, the confidence limits of the results obtained by this method are not greater than ± 3 for the confidence level of 95 %.

Annex A
(informative)

Proficiency results

A.1 General

A proficiency testing, based on the current method, was organized in 2008, by ASQUAL, a proficiency testing organization located in Paris, France.

Twenty-five international laboratories participated to this trial. The laboratories had to carry out both manual separation and chemical methods on the same linen/flax woven fabric.

The construction of the woven fabric allowed the determination of the composition by manual/mechanical separation (according to ISO 1833-1, but the objective of the proficiency testing was really to perform the method by chemical analysis.

Table A.1 shows the percentages of viscose and linen obtained from the manual separation.

Table A.1 — Percentages of viscose and linen obtained from the manual separation

Test specimens		Sample A			Sample B		
		#1	#2	#3	#1	#2	#3
percentage of viscose with agreed percentage additions for moisture	V %	71,88 %	71,76 %	71,66 %	71,23 %	71,62 %	71,70 %
percentage of flax with agreed percentage additions for moisture	L %	28,12 %	28,24 %	28,34 %	28,77 %	28,38 %	28,30 %
				Viscose (%)		**71,64 %**	
				Linen (%)		**28,36 %**	

The laboratory pilot imposed the values d_1 = 1,05 and d_2 = 1,00 (as if linen was raw flax) for the trial. Indeed, the flax was dyed and the values d_1 = 1,16 and d_2 = 1,02 were suitable for this situation. The application of d_1 = 1,16 and d_2 = 1,02 allows to find the values of the percentages calculated from the manual separation, as described in Table A.2.

NOTE In order to select a suitable pair of correction factors d_1 and d_2, correct distinction between the raw linen and bleached linen has to be known, made and indentified, as it leads to a significant influence on the final result.

A.2 Statistical analysis on dry mass loss

The statistical analysis[1)] is based on the percentage of loss in dry mass of the test specimen during the pre-treatment.

An application of Cochran's statistical test (on deviations) led to the subsequent exclusion, from the final calculation, of the values from the following laboratories: O and AJ.

An application of Grubbs's statistical test (on mean values) led to the subsequent exclusion, from the final calculation, of values from the following laboratories: C, T, AF, N and AG.

1) Reported laboratory test values were analysed with all the results obtained from the participants, taking into account and using the following statistical tests: Cochran's test (ISO 5725-2) for repeatability and Grubbs's test (ISO 5725-2) for reproducibility. A determination of z'-score values was made according to ISO 13528, taking into account the homogeneity of distributed samples.

A z'-score test result indicate that the data from 4 laboratories C, N, AF and AJ are outliers (see Figure A.1).

NOTE The neutralization step was identified as a factor which has a significant influence on the final result.

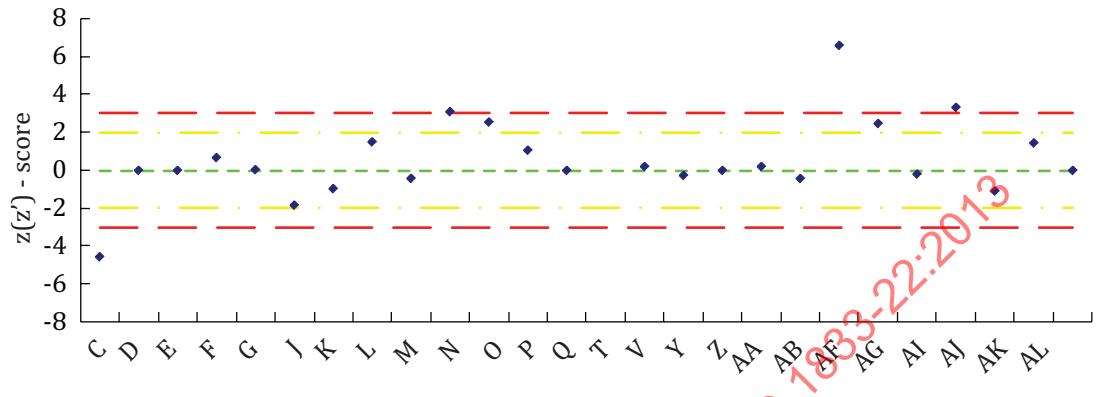

Figure A.1 — Graph of z'-score of the measurement of dry mass loss percentage

The mean value of test results is 11,61 % for percentage of loss in dry mass during the pre-treatment (see Figure A.2).

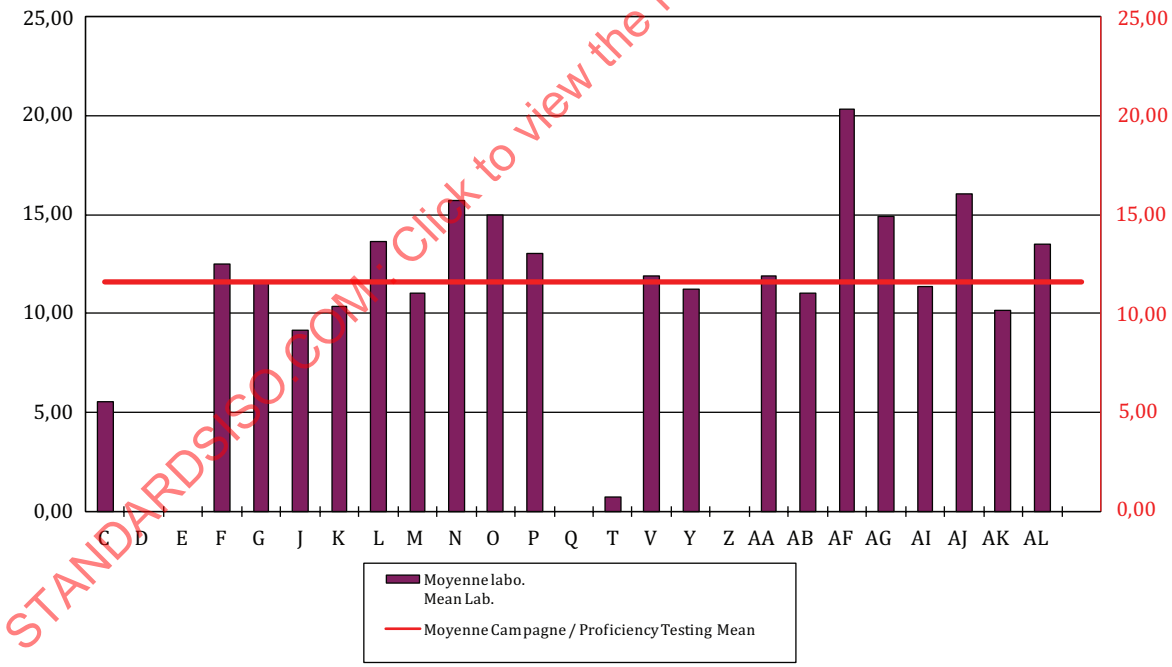

Figure A.2 — Histogram of the distribution of dry mass loss percentages

A.3 Statistical analysis based on dry mass of viscose

The statistical analysis is based on the dry mass percentage of viscose.

An application of Cochran's statistical test (on deviations) led to the subsequent exclusion, from the final calculation, of values from the following laboratories: N, O and P.

An application of the Grubbs's statistical test (on mean values) led to the subsequent exclusion, from the final calculation, of values from the following laboratories: C, T, G, P, AK et AL.

A z'-score test result indicates that the data from 5 laboratories C, N, P, T and AJ are outliers (see Figure A.3).

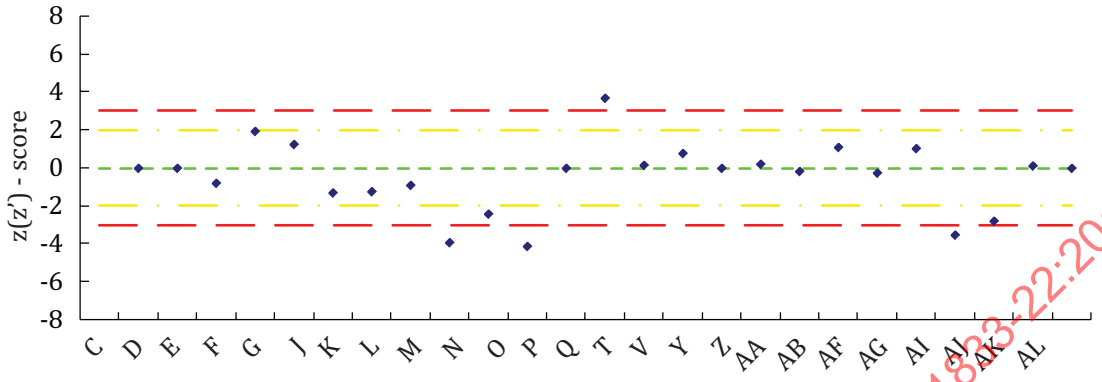

Figure A.3 — Graph of z'-score of the measurement of dry mass percentage of viscose

The mean value of test results is 67,03 % for the dry mass percentage of viscose (see Figure A.4 and Table A.2).

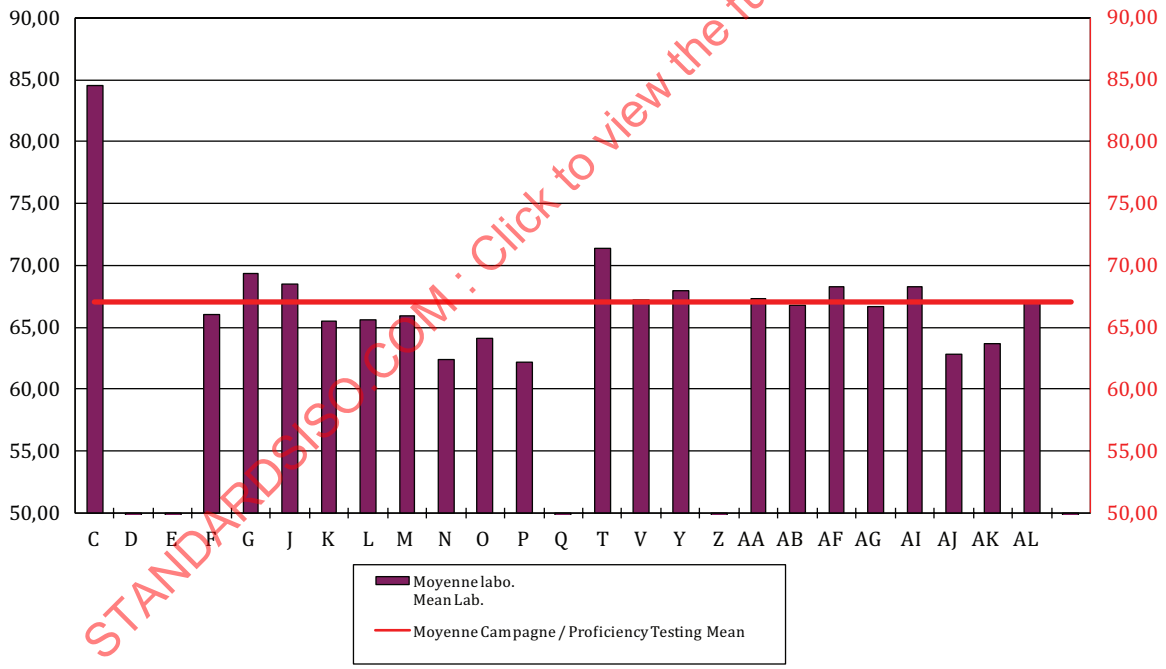

Figure A.4 — Histogram of the distribution of dry mass percentages of viscose