Regulation (EU) No 1007/2011 of the European Parliament and of the Council of 27 September 2011 on textile fibre names and related labelling and marking of the fibre composition of textile products and repealing Council Directive 73/44/EEC and Directives 96/73/EC and 2008/121/EC of the European Parliament and of the Council (Text with EEA relevance)

ANNEX VIII

Methods for the quantitative analysis of binary and ternary textile fibre mixtures
(referred to in Article 19(1))

CHAPTER 3
QUANTITATIVE ANALYSIS OF TERNARY TEXTILE FIBRE MIXTURES

I. General information on methods for the quantitative chemical analysis of ternary fibre mixtures

Information common to the methods given for the quantitative chemical analysis of ternary fibre mixtures.

I.1. FIELD OF APPLICATION

The field of application of each method for analysing binary fibre mixtures specifies to which fibres the method is applicable (see Chapter 2 relating to methods for quantitative analysis of certain binary textile fibre mixtures).

I.2. PRINCIPLE

After the identification of the components of a mixture, the non-fibrous material is removed by suitable pre-treatment and then one or more of the four variants of the process of selective solution described in the introduction is applied. Except where this presents technical difficulties, it is preferable to dissolve the major fibre component so as to obtain the minor fibre component as final residue.

I.3. MATERIALS AND EQUIPMENT

I.3.1. Apparatus

I.3.1.1. Filter crucibles and weighing bottles large enough to contain such crucibles, or any other apparatus giving identical results.

I.3.1.2. Vacuum flask.

I.3.1.3. Desiccator containing self-indicating silica gel.

I.3.1.4. Ventilated oven for drying specimens at 105 ± 3 °C.

I.3.1.5. Analytical balance, accurate to 0,0002 g.

I.3.1.6. Soxhlet extractor or other apparatus giving identical results.

I.3.2. Reagents

I.3.2.1. Light petroleum, redistilled, boiling range 40 to 60 °C.

I.3.2.2. Other reagents are specified in the appropriate sections of each method.

I.3.2.3. Distilled or deionised water.

I.3.2.4. Acetone.

I.3.2.5. Orthophosphoric acid.

I.3.2.6. Urea.

I.3.2.7. Sodium bicarbonate.

All reagents used shall be chemically pure.

I.4. CONDITIONING AND TESTING ATMOSPHERE

Because dry masses are determined, it is unnecessary to condition the specimen or to conduct analyses in a conditioned atmosphere.

I.5. LABORATORY TEST SAMPLE

Take a laboratory test sample that is representative of the laboratory bulk sample and sufficient to provide all the specimens, each of at least 1 g, that are required.

I.6. PRE-TREATMENT OF LABORATORY TEST SAMPLE[1]

Where a substance not to be taken into account in the percentage calculations (see Article 19) is present, it shall first be removed by a suitable method that does not affect any of the fibre constituents.

For this purpose, non-fibrous matter which can be extracted with light petroleum and water is removed by treating the laboratory test sample in a Soxhlet extractor with light petroleum for 1 hour at a minimum rate of six cycles per hour. Allow the light petroleum to evaporate from the laboratory test sample, which is then extracted by direct treatment consisting in soaking the laboratory test sample in water at room temperature for 1 hour and then soaking it in water at 65 ± 5 °C for a further hour, agitating the liquor from time to time. Use a liquor: laboratory test sample ratio of 100:1. Remove the excess water from the laboratory test sample by squeezing, suction or centrifuging and then allow the laboratory test sample to become air-dry.

In the case of elastolefin or fibre mixtures containing elastolefin and other fibres (wool, animal hair, silk, cotton, flax (or linen), true hemp, jute, abaca, alfa, coir, broom, ramie, sisal, cupro, modal, protein, viscose, acrylic, polyamide or nylon, polyester, elastomultiester) the procedure just described shall be slightly modified, in fact light petroleum ether shall be replaced by acetone.

Where non-fibrous matter cannot be extracted with light petroleum and water, it shall be removed by substituting for the water method described above a suitable method that does not substantially alter any of the fibre constituents. However, for some unbleached, natural vegetable fibres (e.g. jute, coir) it is to be noted that normal pre-treatment with light petroleum and water does not remove all the natural non-fibrous substances; nevertheless additional pre-treatment is not applied unless the sample contains finishes insoluble in both light petroleum and water.

Analysis reports shall include full details of the methods of pre-treatment used.

I.7. TEST PROCEDURE

I.7.1. General instructions

I.7.1.1. Drying

Conduct all drying operations for not less than 4 hours and not more than 16 hours at 105 ± 3 °C in a ventilated oven with the oven door closed throughout. If the drying period is less than 14 hours, the specimen must be checkweighed to determine whether its mass is constant. The mass may be considered as constant if, after a further drying period of 60 minutes, its variation is less than 0,05 %.

Avoid handling crucibles and weighing bottles, specimens or residues with bare hands during the drying, cooling and weighing operations.

Dry specimens in a weighing bottle with its cover beside it. After drying, stopper the weighing bottle before removing it from the oven, and transfer it quickly to the desiccator.

Dry the filter crucible in a weighing bottle with its cover beside it in the oven. After drying, close the weighing bottle and transfer it quickly to the desiccator.

Where apparatus other than a filter crucible is used, drying operations shall be conducted in the oven so as to determine the dry mass of the fibres without loss.

I.7.1.2. Cooling

Conduct all cooling operations in the desiccator, placed beside the balance, until the cooling of the weighing bottles is complete, and in any case for not less than 2 hours.

I.7.1.3. Weighing

After cooling, complete the weighing of the weighing bottle within 2 minutes of its removal from the desiccator; weigh to an accuracy of 0,0002 g.

I.7.2. Procedure

Take from the pre-treated laboratory test sample a test specimen of at least 1 g (in mass). Cut yarn or cloth into lengths of about 10 mm, dissected as much as possible. Dry the specimen in a weighing bottle, cool it in the desiccator and weigh it. Transfer the specimen to the glass vessel specified in the appropriate section of the Union method, reweigh the weighing bottle immediately and obtain the dry mass of the specimen by difference; complete the test as specified in the appropriate section of the applicable method. Examine the residue microscopically to check that the treatment has in fact completely removed the soluble fibre(s).

I.8. CALCULATION AND EXPRESSION OF RESULTS

Express the mass of each component as a percentage of the total mass of fibre in the mixture. Calculate the results on the basis of dean dry mass, adjusted by (a) the agreed allowances and (b) the correction factors necessary to take account of loss of non-fibrous matter during pre-treatment and analysis.

I.8.1. Calculation of percentages of mass of clean dry fibres disregarding loss of fibre mass during pre-treatment.

I.8.1.1. - VARIANT 1 -

Formulae to be applied where a component of the mixture is removed from one specimen and another component from a second specimen:

$$P_1\,\% = \left[\frac{d_2}{d_1} - d_2 \times \frac{r_1}{m_1} + \frac{r_2}{m_2} \times \left(1 - \frac{d_2}{d_1}\right)\right] \times 100$$

$$P_2\,\% = \left[\frac{d_4}{d_3} - d_4 \times \frac{r_3}{m_2} + \frac{r_1}{m_1} \times \left(1 - \frac{d_4}{d_3}\right)\right] \times 100$$

$P_3\% = 100 - (P_1\% + P_2\%)$

$P_1\%$	is the percentage of the first clean dry component (component in the first specimen dissolved in the first reagent),
$P_2\%$	is the percentage of the second clean dry component (component in the second specimen dissolved in the second reagent),
$P_3\%$	is the percentage of the third clean dry component (component undissolved in both specimens),
m_1	is the dry mass of the first specimen after pre-treatment,
m_2	is the dry mass of the second specimen after pre-treatment,

r_1 is the dry mass of the residue after removal of the first component from the first specimen in the first reagent,

r_2 is the dry mass of the residue after removal of the second component from the second specimen in the second reagent,

d_1 is the correction factor for loss in mass in the first reagent, of the second component undissolved in the first specimen[(2)];

d_2 is the correction factor for loss in mass in the first reagent, of the third component undissolved in the first specimen,

d_3 is the correction factor for loss in mass in the second reagent, of the first component undissolved in the second specimen,

d_4 is the correction factor for loss in mass in the second reagent, of the third component undissolved in the second specimen.

I.8.1.2. - VARIANT 2 -

Formulae to be applied where a component (a) is removed from the first test specimen, leaving as residue the other two components (b + c), and two components (a + b) are removed from the second test specimen, leaving as residue the third component (c):

$P_1\% = 100 - (P_2\% + P_3\%)$

$$P_2\% = 100 \times \frac{d_1 r_1}{m_1} - \frac{d_1}{d_2} \times P_3\%$$

$$P_3\% = \frac{d_4 r_2}{m_2} \times 100$$

$P_1\%$ is the percentage of the first clean dry component (component in the first specimen dissolved in the first reagent),

$P_2\%$ is the percentage of the second clean dry component (component soluble, at the same time as the first component of the second specimen, in the second reagent),

$P_3\%$ is the percentage of the third clean dry component (component undissolved in both specimens),

m_1 is the dry mass of the first specimen after pre-treatment,

m_2 is the dry mass of the second specimen after pre-treatment,

r_1 is the dry mass of the residue after removal of the first component from the first specimen in the first reagent,

r_2 is the dry mass of the residue after removal of the first and second components from the second specimen in the second reagent,

d_1 is the correction factor for loss in mass in the first reagent, of the second component undissolved in the first specimen,

d_2 is the correction factor for loss in mass in the first reagent, of the third component undissolved in the first specimen,

d_4 is the correction factor for loss in mass in the second reagent, of the third component undissolved in the second specimen.

I.8.1.3. - VARIANT 3 -

Formulae to be applied where two components (a + b) are removed from a specimen, leaving as residue the third component (c), then two components (b + c) are removed from another specimen, leaving as residue the first component (a):

$$P_1\% = \frac{d_3 r_2}{m_2} \times 100$$

$P_2\% = 100 - (P_1\% + P_3\%)$

$$P_3\% = \frac{d_2 r_1}{m_1} \times 100$$

P_1%	is the percentage of the first clean dry component (component dissolved by the reagent),
P_2%	is the percentage of the second clean dry component (component dissolved by the reagent),
P_3%	is the percentage of the third clean dry component (component dissolved in the second specimen by the reagent),
m_1	is the dry mass of the first specimen after pre-treatment,
m_2	is the dry mass of the second specimen after pre-treatment,
r_1	is the dry mass of the residue after the removal of the first and second components from the first specimen with the first reagent,
r_2	is the dry mass of the residue after the removal of the second and third components from the second specimen with the second reagent,
d_2	is the correction factor for loss in mass in the first reagent of the third component undissolved in the first specimen,
d_3	is the correction factor for loss in mass in the second reagent of the first component undissolved in the second specimen.

I.8.1.4. - VARIANT 4 -

Formulae to be applied where two components are successively removed from the mixture using the same specimen:

$$P_1\% = 100 - (P_2\% + P_3\%)$$

$$P_2\% = \frac{d_1 r_1}{m} \times 100 - \frac{d_1}{d_2} \times P_3\%$$

$$P_3\% = \frac{d_3 r_2}{m} \times 100$$

P_1%	is the percentage of the first clean dry component (first soluble component),
P_2%	is the percentage of the second clean dry component (second soluble component),
P_3%	is the percentage of the third clean dry component (insoluble component),
m	is the dry mass of the specimen after pre-treatment,
r_1	is the dry mass of the residue after elimination of the first component by the first reagent,
r_2	is the dry mass of the residue after elimination of the first and second component by the first and second reagents,
d_1	is the correction factor for loss in mass of the second component in the first reagent,
d_2	is the correction factor for loss in mass of the third component in the first reagent,
d_3	is the correction factor for loss in mass of the third component in the first and second reagents[(3)].

I.8.2. Calculation of the percentage of each component with adjustment by agreed allowances and, where appropriate, correction factors for losses in mass during pre-treatment operations:

Given:

$$A = 1 + \frac{a_1 + b_1}{100}$$

$$B = 1 + \frac{a_2 + b_2}{100}$$

$$C = 1 + \frac{a_3 + b_3}{100}$$

then:

$$P_1\text{A}\,\% = \frac{P_1 A}{P_1 A + P_2 B + P_3 C} \times 100$$

$$P_2\text{A}\,\% = \frac{P_2 B}{P_1 A + P_2 B + P_3 C} \times 100$$

$$P_3\text{A}\,\% = \frac{P_3 C}{P_1 A + P_2 B + P_3 C} \times 100$$

$P_1A\%$	is the percentage of the first clean dry component, including moisture content and loss in mass during pre-treatment,
$P_2A\%$	is the percentage of the second clean dry component, including moisture content and loss in mass during pre-treatment,
$P_3A\%$	is the percentage of the third clean dry component, including moisture content and loss in mass during pre-treatment,
P_1	is the percentage of the first clean dry component obtained by one of the formulae given in I.8.1,
P_2	is the percentage of the second clean dry component obtained by one of the formulae given in I.8.1,
P_3	is the percentage of the third clean dry component obtained by one of the formulae given in I.8.1,
a_1	is the agreed allowance of the first component,
a_2	is the agreed allowance of the second component,
a_3	is the agreed allowance of the third component,
b_1	is the percentage of loss in mass of the first component during pre-treatment,
b_2	is the percentage of loss in mass of the second component during pre-treatment,
b_3	is the percentage of loss in mass of the third component during pre-treatment.

Where a special pre-treatment is used the values b_1, b_2 and b_3 shall be determined, if possible, by submitting each of the pure fibre constituents to the pre-treatment applied in the analysis. Pure fibres are those free from all non-fibrous material except those which they normally contain (either naturally or because of the manufacturing process), in the state (unbleached, bleached) in which they are found in the material to be analysed.

Where no clean separate constituent fibres used in the manufacture of the material to be analysed are available, average values of b_1, b_2 and b_3 as obtained from tests performed on clean fibres similar to those in the mixture under examination, must be used.

If normal pre-treatment by extraction with light petroleum and water is applied, correction factors b_1, b_2 and b_3 may generally be ignored, except in the case of unbleached cotton, unbleached flax (or linen) and unbleached hemp where the loss due to pre-treatment is usually accepted as 4 % and in the case of polypropylene as 1 %.

In the case of other fibres, losses due to pre-treatment are usually disregarded in calculations.

I.8.3. Note:

Calculation examples are given in Section IV.

(**1**) See Chapter 1.1.

(**2**) The values of d are indicated in Chapter 2 of this Annex relating to the various methods of analysing binary mixtures.

(**3**) Wherever possible d_3 should be determined in advance by experimental methods.

Status:
Point in time view as at 31/01/2020.

Changes to legislation:
There are outstanding changes not yet made to Regulation (EU) No 1007/2011 of the European Parliament and of the Council. Any changes that have already been made to the legislation appear in the content and are referenced with annotations.