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Textiles and textile products — Microplastics from textile sources —

Part 1: Determination of material loss from fabrics during washing

Textiles et produits textiles — Microplastiques d'origines textiles —

Partie 1: Détermination des pertes de matière des étoffes pendant le lavage

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Foreword

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This document was prepared by Technical Committee ISO/TC 38, *Textiles*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC248, *Textiles and textile products*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

A list of all parts in the ISO 4484 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

There is significant evidence that during laundering of textiles, release of fragments can occur. The purpose of this document is to provide a method of assessment, to be used in laboratories, of the degree to which different fabrics shed fibres and fibre fragments of all types. The results obtained by using this document should enable manufacturers of textile articles to make an informed choice about the type of fabric to use to reduce/minimize shedding as well as to test different methods of manufacture that minimize material loss during laundering.

Textiles and textile products — Microplastics from textile sources —

Part 1: Determination of material loss from fabrics during washing

1 Scope

This document describes a method for systematically collecting material loss from fabrics under laundering test conditions to achieve comparable and accurate results. There is no direct correlation to material loss during domestic and commercial laundering. The method is designed to assess material loss of all types.

NOTE In this document, any collected debris is assumed to be fibre fragments. For the identification of the nature/composition of this debris, the method described in ISO 4484-2 can be used.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 4915, *Textiles — Stitch types — Classification and terminology*

ISO 4916, *Textiles — Seam types — Classification and terminology*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

The test specimen is subjected to an accelerated laundering process under conditions of temperature, time, and mechanical action. The resultant wash liquor is vacuum filtered. Material loss is assessed gravimetrically to approximate material loss during simulated domestic laundering. The ratio of the mass of the material loss by the test specimen mass is then reported.

Note Consideration on detergent can be found in A.1.

5 Reagents

5.1 Water, distilled or grade 3 according to ISO 3696.

6 Apparatus

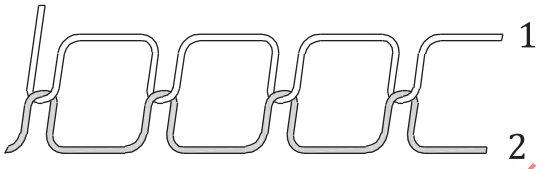
6.1 Lab gloves, which shall be worn during all stages of method execution to prevent contamination by foreign matter(s).

6.2 Lab coat, which shall be worn during all stages of method execution to prevent contamination by foreign matter(s).

NOTE Consideration on contamination reduction can be found in A.2.

6.3 Cutting device, e.g. scissors or cutting press.

6.4 Sewing machine, single needle, capable of lock stitch, type 301 in accordance with ISO 4915 (see Figure 1).



Key

1 needle thread

2 bobbin thread

Figure 1 — Stitch type 301

6.5 Sewing needle, size appropriate for sewing thread, point appropriate for specimen fabric.

6.6 Sewing thread, 100 % polyester or polyamide continuous filament thread, size appropriate for the specimen fabric.

6.7 Oven, capable of maintaining a temperature of (50 ± 3) °C, without air circulation.

6.8 Analytical balance, with a resolution of at least 0,1 mg.

6.9 Glass fibre filter, 1,6 µm pore size, 47 mm diameter, no binder.

6.10 Aluminium pans/specimen tray (non-plastic), with a minimum diameter of 47 mm to support filter when not in use in the filter assembly.

6.11 Suitable mechanical device, consisting of a water bath containing a rotatable shaft, which supports radially, stainless steel containers lying horizontally on the shaft, see Figure 2.

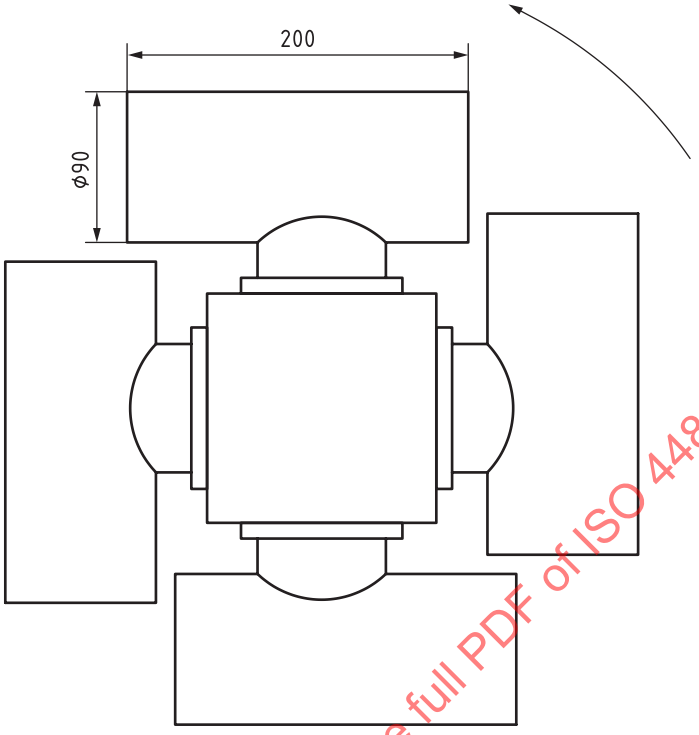


Figure 2 — Orientation of containers around the rotatable shaft

The shaft/container assembly is rotated at a frequency of (40 ± 2) min^{-1}. The temperature of the water bath is thermostatically controlled to maintain the test solution at the prescribed temperature ±2 °C.

Other mechanical devices may be used for this test, provided that the results are identical to those obtained using the apparatus described.

6.12 Stainless steel containers, (1 200 ± 50) ml capacity with diameter of (90 ± 5) mm and depth of (200 ± 10) mm, with lids and seals.

Note Consideration on further investigations can be found in Annex C.

6.13 Non-corrodible (stainless) steel balls, approximately 6 mm in diameter.

6.14 Vacuum filtration device, consisting of a separating sintered non-plastic filter platform and cylindrical glass or stainless-steel funnel with a suitable vacuum source. Shall be suitable for use with 47 mm diameter filters.

6.15 Squeezable wash bottle.

6.16 Stainless steel sieve, with capture holes between 2 mm and 6 mm diameter.

6.17 Glass beaker, with a minimum capacity of 1 500 ml.

6.18 Tweezers, round nosed, non-serrated.

6.19 Desiccator with suitable dessicant.

Before testing thoroughly, clean all work surfaces and working areas to reduce risk of contamination.

7 Preparation of test specimen

7.1 General

For each laboratory sample, a set of test specimens shall be cut as described in 7.2. A set shall consist of a minimum of 4 specimens.

7.2 Sampling

A test specimen is prepared by cutting a rectangle of dimensions (150 ± 10) mm × (290 ± 10) mm with cutting device (6.3).

Test specimens should be taken from the fabric roll ensuring they are at least 100 mm from the cut edge of the laboratory sample and at least 150 mm from the selvedge.

Take an equal number of specimens with their long dimensions parallel to both fabric directions. Specimens for a given fabric direction should be spaced along a diagonal of the fabric to allow for representation of different warp and filling yarns, or machine and cross direction areas, in each specimen.

7.3 Preparation of test specimens

Fold each edge towards the reverse of the specimen twice to form a double rolled hem ≈ 10 mm wide (type 6.03.01 in accordance with ISO 4916) (see Figure 3) working on a total seam allowance of 25 mm.

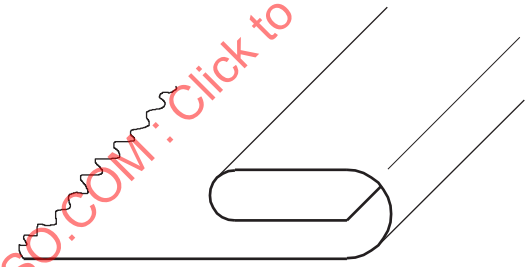


Figure 3 — Double rolled hem type 6.03.01

Finish all hems by sewing (6.4) along the centre line using a lock stitch type 301 as described in ISO 4915 (see Figure 1), ensuring that the needle (6.5) is passing through all layers of the hem to fully enclose the cut edge.

Where possible, sew a continuous row of stitching along all edges and carefully around all corners. It may be necessary to hand turn the sewing machine (6.4) handwheel through the corner sections. In exceptional cases where it is not possible to sew continuously around the corners due to fabric bulk, stop the row of stitching just before the corner section, raise the needle and presser foot and reposition the specimen to recommence the row of stitching just after the corner section. Note in the test report which hemming procedure has been followed.

Repeat the hemming procedure on all edges of the specimen such that the resultant specimen size is (100 ± 10) mm × (240 ± 10) mm.

Some fabrics thickness can make it difficult to produce this hem. In this instance, the seam allowance can be increased to 50 mm. It is important that the finished specimen size is (100 ± 10) mm × (240 ± 10) mm.

8 Procedure

8.1 Determination of dry mass of test specimens

Dry the test specimens in an oven (6.7) at (50 ± 3) °C to reach a constant mass, which shall be considered as attained when no progressive change in mass greater than 0,1 mg occurs in successive weighings separated by a drying period of at least 40 min.

NOTE Experience has shown that a 4 h drying period could be sufficient.

Allow to cool in a desiccator (6.19) before weighing.

Determine and record the dry mass of each specimen (Sm_1), to an accuracy of 0,1 mg, using the analytical balance (6.8).

8.2 Filter preparation

Pre-rinse the filters (6.9) by placing them, using tweezers (6.18), into the filtration device (6.14) and turning on the vacuum, seat the filter by wetting thoroughly with the wash bottle (6.15), rinse the filter three times with 20 ml water (5.1) to remove soluble compounds.

Place each filter (6.9) into a separate, labelled tray (6.10), it is essential to keep each filter/tray combination consistent throughout the duration of the test. Place the prepared filter/tray combination in the oven (6.7) at a temperature of (50 ± 3) °C for a minimum of four hours.

8.3 Determination of dry mass of the filter

Determine and record the mass of the filter/tray combination (Fm_1) to be used, using the analytical balance (6.8), to an accuracy of 0,1 mg.

8.4 Washing process

Pre-heat accelerated laundering device (6.11) to specified temperature (40 ± 2) °C.

Prepare the container (6.12) by placing 360 ml water (5.1) and 50 steel balls (6.13) inside and close the container.

Pre-heat containers (6.12) in the accelerated laundering device (6.11) for approximately 10 minutes. Remove the containers from the laundering device and add one prepared specimen to each container.

Put the containers (6.12) back into the accelerated laundering device (6.11) and agitate at (40 ± 2) °C for (45 ± 1) min, with a rotation speed of (40 ± 2) min^{-1}.

NOTE The volume of water specified in the test was considered in relation to the size of the test specimen and ability to achieve suitable and effective agitation.

8.5 Filtration process

Use the following procedure for each container (6.12) and specimen.

Filter the liquor from each container separately, DO NOT mix liquors and use a different filter (6.9) for each container. Remove the lid from the container and pour all wash liquor into the sieve (6.16) over a beaker (6.17), capturing the specimen and steel balls (6.13). Be careful to avoid splashing.

Use a wash bottle (6.15) to rinse the inside of the container (6.12), the lid and seal, collecting all rinse water in the same beaker (6.17). Repeat for a total of 3 rinses.

Rinse gloved hands discarding the liquor resulted from the rinsing before squeezing the specimen.

Gently lift the specimen out of the sieve (6.16) and rinse it 3 times with the wash bottle (6.15), spraying the specimen on the front and back sides to free loose fibre fragments from the surface. Collect all rinse water in the beaker (6.17), through the sieve (6.16).

Gently hand squeeze the specimen to remove excess water. Rinse the gloved hand with wash bottle (6.15) in case of any fibre transfer. All rinse liquor shall be poured into the beaker (6.17).

Use the wash bottle (6.15) to rinse the steel balls (6.13) and the sieve (6.16) three times each with water (5.1).

Use tweezers (6.18) to place a filter (6.9) into the filtration device (6.14), ensure it is flat and placed correctly on the filter holder. Fix the filtration device funnel and turn on the vacuum. Seat the filter by wetting with wash bottle (6.15).

Carefully pour the contents of the collection beaker (6.17) on the filter (6.9). Avoid splashing. Using a wash bottle (6.15), rinse the beaker three times, pouring all rinsed liquor into the filtration device (6.14). If the filter has become too clogged to allow the filtration to be completed, a second dried filter (8.3) can be substituted and the results combined.

Using wash bottle, rinse the funnel of the filtration device three times, turn off the vacuum source, remove the funnel, and using tweezers (6.18), return the filter to its tray (6.10).

Carefully use the wash bottle (6.15) to rinse any remaining fibres from the bottom edge of the funnel (6.14) into the specimen tray (6.10).

Return filters (6.9) and trays (6.10) to the oven (6.7) until dry (8.3), for a minimum of 4 h.

Determine and record the dry mass of each filter/tray combination (Fm_2), to an accuracy of 0,1 mg, using the analytical balance.

Assign a zero value to the test specimen(s) if the calculated *Mf* is negative.

9 Expression of results

Calculate the mass of fibre fragment release for specimen(s) using Formula (1):

$$Mf = Fm_2 - Fm_1 \quad (1)$$

where

Mf is the mass of fibre released expressed in mg;

Fm_2 is the mass of the filter/tray combination after testing expressed in mg;

Fm_1 is the mass of the filter/tray combination before testing expressed in mg.

Calculate the mass of fibre fragment release as a ratio of the specimen(s) mass using Formula (2):

$$Rf = \frac{Mf}{Sm_1} \quad (2)$$

where

Rf is the ratio of the mass of the fibre released, related to the test specimen mass, expressed in mg/kg;

Mf is the mass of fibre released expressed in mg;

Sm_1 is the mass in kg of test specimen before testing.

If a blank is carried out according to A.3, correct the test specimen mass by the blank mass using Formula (3):

$$Rf = \frac{(Mf - Mb)}{Sm_1} \quad (3)$$

where

Rf is the ratio of the mass of the fibre released, related to the test specimen mass, expressed in mg/kg;

Mf is the mass of fibre released expressed in mg;

Mb is the mass of fibre collected from the blank expressed in mg;

Sm_1 is the mass in kg of test specimen before testing.

NOTE Statistical data can be found in Annex B.

10 Test report

The test report shall specify the following:

a) the method used, by reference to this document, i.e. ISO 4484-1:2023;

b) identification of the sample;

c) sizes of thread (6.6) and needle (6.5) used;

d) hemming procedure used (7.3);

e) mass of individual specimen(s) before testing to the nearest 0,1 mg

f) individual specimen results of mass loss expressed to the nearest 0,1 mg;

g) arithmetic mean of mass loss to the nearest 0,1 mg;

h) individual specimen results of mass loss as a ratio of the original specimen, as expressed in mg/kg;

i) arithmetic mean of mass loss as a ratio of the original specimen, as expressed in mg/kg;

j) any deviation from the given procedure;

k) any unusual features observed;

l) date of test.

Annex A
(informative)

Rationale

A.1 Use of detergent

Detergent has intentionally been left out of this test method. Many detergents have been found to clog filters during the filtration process, as well as stick to fibres and filters during the whole testing procedure, adding mass which can distort data and lead to misinterpretation of results.

A.2 Quality control to reduce contamination

When using this test method, due care and attention shall be observed to reduce the risk of contamination. Risk of contamination is high from both a probability and severity standpoint, and contamination from all sources can falsify results during gravimetric measurements.

The test method calls for the use of lab gloves and lab coats during the testing procedure, however, additional care and attention shall be paid to all working areas (including benches and ovens), as well as during the storage and use of samples, filters and other apparatus.

The following are additional measures to minimize the effect of environmental contamination.

a) All glassware shall be previously washed with water (5.1) then rinsed before each use.

b) All washbottles shall be previously washed with water (5.1) then rinsed before each use.

c) After washings, the glassware and washbottles shall be stored and protected with the aid of suitable barriers (e.g. aluminium foil closing the inlets) to reduce the possible deposition of fibre fragments present in the air.

d) The same applies to specimen containers for which glass and metals should be preferred.

e) Wipe down all surfaces before starting each test

f) Rinse all tweezers, probes and hands before of each procedure such as filtration, opening a petri dish for inspection.

g) Minimize traffic in your lab or working space.

A.3 Internal quality assessment

An optional method to consider the “environmental contamination” can be carried out in parallel. Whereby a “blank test” comprising a sample of water (5.1) and any other washing solution used in the test is subjected to the analysis procedure. The fibre fragment value detected (see Clause 9) can then be defined as “environmental contamination” and shall be deducted from the values obtained for the corresponding specimens. The determination of the blank test can be carried out for each batch of specimens. Blank tests shall be carried out in multiples of two to mitigate machine imbalance issues.