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**Textiles and textile products — Microplastics from textile sources — Part 3:
Measurement of collected material mass released from textile end products by
domestic washing method**

*Textiles et produits textiles — Microplastiques d'origines textiles — Partie 3: Mesurage de
la masse de matériaux collectés libérés par les produits finis textiles par la méthode de
lavage domestique*

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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.

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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/TC 248, *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

Daily washing of textile end products generates fibre fragments which are discharged from washing machine. The purpose of this test method is to collect materials including fibre fragments which are discharged from the domestic washing machine through the washing process.

Although there are many types of washing machines used at home, ISO 6330 has been developed for the domestic washing test method, which specifies the domestic washing machines and test conditions. This document utilizes one of the standard washing machines in ISO 6330.

In addition, a care label is attached to textile end products in many countries of the world and this care label specifies washing conditions to use in this document.

Due to the complicated internal structure of washing machines, ~~theits~~ cleaning process ~~of the washing machines~~ is very important to avoid ~~the effect of~~ contamination ~~of washing machine~~. This document proposes the checking method and the requirements of the washing machine to be used in this document.

The identification and quantification of components contained in the collected materials through the washing process ~~will beis~~ determined by applying ISO 4484-2.

~~This~~The information gathered from this document can be utilized by the textile industry for the development of textile end products to reduce or minimize shedding materials from textile end products through the washing process.

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Textiles and textile products — Microplastics from textile sources — Part 3: Measurement of collected material mass released from textile end products by the domestic washing method

1 Scope

This document specifies a method for ~~measurement of~~ measuring the collected material mass released from the outlet hose of a standard washing machine, described in ISO 6330, through the washing process.

NOTE The washing condition of textile end products is indicated by the care labelling according to ISO 3758.

This document is applicable to textile end products ~~which is~~ (including consumer textile products, such as clothing, ~~such as made of~~ fleece, shirts, trousers, blouse, etc.,) and home textile end products, ~~(such as, blankets, rugs, curtains, etc.,)~~ which are composed of all fibres such as natural fibres, and man-made fibres, including mixture of the fibres that can be washed in a domestic washing machine.

This document is not applicable to fabrics and cut textile products, ~~and~~ It does not cover the test for washing machines and detergents as well.

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 139, *Textiles — Standard atmospheres for conditioning and testing*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 3758, *Textiles — Care labelling code using symbols*

ISO 6330:2021, *Textiles — Domestic washing and drying procedures for textile testing*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological 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/>

3.1

collected material

material collected by the filter attached at the outlet hose of a washing machine through the washing process, which include fibre fragments and ~~not non-~~ water-soluble materials

3.2

empty washing cycle

washing cycle without test specimens by using the test washing program

4 Principle

A given mass of textile end products is washed in a domestic washing machine under certain ~~conditions of~~ temperature and mechanical conditions for a given number of washing cycles. The material shed through the washing cycles is collected ~~by using~~ a filter bag which is attached at the outlet hose of the washing machine. The collected materials ~~by the~~ filter bag are transferred to ~~the~~ membrane filter and the membrane filter with the collected materials are weighed in the weighing bottle. The membrane filter and weighing bottle are prepared in combination. The mass of the collected materials is calculated by the ~~subtraction of~~ ~~subtracting~~ the original mass of the membrane filter and weighing bottle from the mass with the collected materials. The mass of collected materials, the ratio of the mass of the collected materials and the test specimen mass and the ratio of the mass of the collected material and the number of the test specimen are reported.

5 Apparatus

5.1 Domestic washing machine, specified in ISO 6330, with built-in filters removed. The domestic washing machine used ~~to this~~ in document shall ~~be met~~ ~~meet~~ the ~~requirement~~ ~~requirements~~ in Annex B.

5.2 Filter bag, woven mesh, with aperture size of $(10 \pm 4) \mu\text{m}$ ~~and~~; the specification for the materials and the construction is described in Annex A. The aperture size of the filter is determined according to Annex I.

5.3 Analytical balance, with a resolution of 0,1 mg or better.

5.4 Balance, with a resolution of 1 g or better.

5.5 Oven, capable of maintaining a temperature of $(105 \pm 3) ^\circ\text{C}$, without fan.

5.6 Cable ties, made of polyamide.

5.7 Polycarbonate membrane filter, approximately 47 mm in diameter with $10 \mu\text{m}$ aperture size. Stainless steel filter is also applicable.

5.8 Weighing bottle or dish, made of metal or glass, with a minimum diameter, approximately 47 mm, with a lid.

5.9 Vacuum filtration device, consisting of sintered filter platform and funnel, with vacuum pump.

5.10 Wash bottle, made of polyethylene or silicon resin, etc., squeezable and with nozzle.

5.11 Tweezers, made of stainless steel or plastics.

5.12 Glass beaker, with a minimum capacity of 1 000 ml.

5.13 Desiccator, with drying agent.

6 Material and reagent

6.1 Water, distilled or grade 3 water according to ISO 3696.

7 Test condition

7.1 Standard atmosphere for preparation of test specimens and mass measurement

The standard atmosphere for test specimen preparation and mass measurement are at the temperature of (20 ± 2) °C and the relative humidity of (65 ± 4) % ~~according to~~ in accordance with ISO 139.

7.2 Test atmosphere for washing test

The condition for the washing test is ambient condition.

8 Test specimen preparation

Only one kind of textile end products is applied in this washing test. Different kinds of textile end products shall not be mixed in the washing test. The number of the test specimens per washing test (S_n) is at least two textile end products. The maximum dry volume of all test specimens per washing test shall be not more than half of the volume of the washing tub.

The washing test is repeated for three (3) sets.

The number of the test specimens can affect ~~to~~ the test result, which is shown in Annex G as an example.

The test specimens are conditioned ~~according to~~ in accordance with ISO-139. The total mass of the conditioned test specimens (M_p), which is measured ~~by the using a~~ balance (5.4), shall be up to 1 g and reported in the test report.

10 Test procedure

10.19.1 General

The washing machine used in this test shall meet the requirement described in Annex-B. Once met the requirement, the washing machine can be used continuously for the washing test.

For the consecutive washing test, take a procedure from ~~Clause~~ 9.2.

The washing conditions of this test procedure is indicated by the care label attached to the textile end products ~~according to~~ in accordance with ISO 3758. The details of the washing condition are referred to ISO 6330.

If the countries use their own care label system or care instruction system, set the washing condition indicated by their system or if there is no label, run the washing program agreed among the interested parties.

10.29.2 Empty washing cycle before the washing test

Mount the filter bag (5.2) to the outlet hose of the domestic washing machine (5.1) and tie up by cable tie (5.6). Run one washing cycle of the washing program without test specimens. The washing program is the same as the washing test as ~~Clause~~ 9.3. After completion of the washing cycle, remove the filter bag from outlet hose. Measure the mass of the collected materials by following the steps from ~~Clause~~ 9.4 to 9.5 and ~~Clause~~ 10 and report the mass of the collected materials from the empty washing cycle in the test report. This is an information of the test washing machine condition.

10.39.3 Washing test of the test specimen

Mount the filter bag (5.2) to the outlet hose of the domestic washing machine (5.1) and tie up by cable tie (5.6). Put the test specimens into the washing tub and run one test washing cycle of the washing program.

The detergent may be used for washing test and the preferable detergent is introduced in Annex D.

When the multiple numbers of the test washing cycle are required, run the number of the washing cycles as determined by the interested parties according to Annex-E. An example of the results of the multiple numbers of test washing cycles are shown in Annex-E.

After completion of the washing cycle, remove the test specimens from the washing tub.

Then, run 2 empty washing cycles by the same washing program.

The number of empty washing cycles (2 empty washing cycles) after test washing was determined by the results as shown in Annex C.

After completion of the empty washing cycle, remove the filter bag from outlet hose.

For the 2nd and 3rd sets of the test specimens, run the washing test in ~~Clause~~9.3.

10.49.4 -Washing of the collected material from the filter bag

Prepare four (4) glass beakers (5.12) containing 1_l of water (6.1) separately. Wash the filter bags of ~~Clause~~ 9.2 and 9.3 in the glass beakers by turning inside out the filter bags. The solution in the beaker is the suspension solution of the collected materials.

10.59.5 -Measurement of the mass of the collected materials

Prepare four (4) polycarbonate membrane filters (5.7) and four (4) weighing bottles (5.8) in combination and separately, it is essential to keep each membrane filter/weighing bottle combination consistent throughout the duration of the test. Measure the mass of the original membrane filter (5.7) and a weighing bottle (5.8), respectively, and record them up to 0,1_mg and denoted as (M_{f1}).

Membrane filters (5.7) are handled by tweezers (5.11). The membrane filter (5.7) shall be kept in the weighing bottle (5.8) to prevent contamination from the environment.

Set the membrane filter (5.7) on a vacuum filtration device (5.9).

Filter the suspension solution of ~~Clause~~9.4 by the membrane filters on a vacuum filtration device (5.9). Add 1_l of water (6.1) to the beaker and wash the filter bag again and filter the suspension solution by the same membrane filter. Repeat this procedure at least twice. Finally, wash the surface of the filter bag and the inside of the beaker by water (6.1) using a wash bottle (5.10), and this suspension solution is also filtered by the same membrane filter.

Place the membrane filter with collected materials into the original weighing bottle of ~~Clause~~9.5 in combination and dry for 2 h at (105_± 3) °C in the oven (5.5). Then place the weighing bottle with the membrane filter and collected materials in desiccator (5.13) to cool down to the room temperature.

Condition the membrane filter with the collected materials and the weighing bottle under standard atmospheres in ~~Clause~~7.1.

Measure the mass of the conditioned membrane filter with the collected material and the weighing bottle up to 0,1 mg by the analytical balance (5.3) and record the result as (M_{f2}).

At this time, measure of the mass several times at intervals of 1 h or more. The mass is determined as the weighed value that shows a difference compared to the former and subsequent measurements within 0,1 % of the subsequent value.

11.10 Calculation

11.10.1 Calculation for each test run

The mass of the collected materials, C_m in mg is calculated by the Formula (1).

$$C_m = M_{f2} - M_{f1} \quad (1)$$

where

C_m is the mass of the collected materials through the test washing process in mg;

M_{f1} is the mass of the original membrane filter and weighing bottle in mg;

M_{f2} is the mass of the membrane filter with the collected materials and weighing bottle in mg;

The mass of the collected material per textile end product (C_{mp}) in mg per product is calculated by Formula (2).

$$C_{mp} = \frac{C_m}{S_n} \quad (2)$$

where

C_{mp} is the mass of the collected materials in mg/product;

C_m is the mass of the collected materials through the test washing process in mg;

S_n is the number of test specimens for one washing test;

The mass of the collected materials per the mass of the textile end products, C_{mw} in mg/kg is calculated by Formula (3).

$$C_{mw} = \frac{C_m \times 1\,000}{M_p} \quad (3)$$

where

C_{mw} is the mass of the collected materials per mass of textile end products in mg/kg;

C_m is the mass of the collected materials through the test washing process in mg;

M_p is the mass of test specimens in g;

11.2.10.2 Calculation of average for 3 test runs

Take averages of 3 test runs for C_m , C_{mp} and C_{mw} as the test results.

12.11 Repeatability and reproducibility

The interlaboratory test according to this document is ~~shown~~ given in Annex F.

13.12 Test report

The test report shall include the following information:

- a) a reference to this document, i.e. ISO 4484-3:2023;
- b) date of the test;
- c) type of a washing machine and test conditions;
- d) test method (number of washing cycles, etc.);
- e) test environment conditions;
- f) ___ number of test specimen put into the washing tub;
- g) ___ total mass of the test specimens put into the washing tub;
- h) the collected mass in the empty washing cycle before test washing;
- i) the average mass of the collected materials through the test washing process;
- j) the average mass of the collected materials per a textile end product;
- k) the average mass of the collected materials per the mass of textile end products;
- l) material, fibre, product construction, etc. -of specimen tested;

- m) any deviation from the procedure;
- n) any unusual features observed;
- o) ~~if~~ if detergent is used, report the kind and amount of detergent.

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