INTERNATIONAL STANDARD

ISO 11721-1

First edition
2001-01-15

Textiles — Determination of resistance of cellulose-containing textiles to micro-organisms — Soil burial test —

Part 1: Assessment of rot-retardant finishing

Textiles — Détermination de la résistance aux micro-organismes des textiles contenant de la cellulose — Essai d'enfouissement —

Partie 1: Évaluation d'un traitement d'imputrescibilité

Reference number
ISO 11721-1:2001(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

Printed in Switzerland

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 3.

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 member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this part of ISO 11721 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 11721-1 was prepared by the European Committee for Standardization (CEN) in collaboration with ISO Technical Committee TC 38, *Textiles*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

Throughout the text of this standard, read "...this European Standard..." to mean "...this International Standard...".

ISO 11721 consists of the following parts, under the general title *Textiles — Determination of resistance of cellulose-containing textiles to micro-organisms — Soil burial test*:

— *Part 1: Assessment of rot-retardant finishing*

— *Part 2: Attack by micro-organisms/mixed culture*

— *Part 3: Toxicity of textile materials and finishing agents*

— *Part 4: Saturated atmosphere test (mildew)*

Annex A forms a normative part of this part of ISO 11721.

Contents

Page

Foreword

The text of EN ISO 11721-1:2001 has been prepared by Technical Committee CEN/TC 248 "Textiles and textile products", the secretariat of which is held by BSI, in collaboration with Technical Committee ISO/TC 38 "Textiles".

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by July 2001, and conflicting national standards shall be withdrawn at the latest by July 2001.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Introduction

Cellulose-containing textiles are considered resistant to attack by micro-organisms present in soil if their structure, appearance and tensile strength are not essentially altered after a soil burial test. The resistance to deterioration is assessed by measuring the relative reduction in tensile strength between buried and unburied specimens.

If an assessment of long term resistance is required, the procedure described in ISO 11721-2 (in preparation) is applicable.

Due to the biological nature of the soil burial test, and the fact that the test soil cannot be precisely standardized, this standard is only intended to assess the resistance of a fabric to micro-organisms after comparing the performance of specimens with and without a finish.

1 Scope

This standard specifies a method for determination of the resistance of chemically-pretreated textiles to the action of micro-organisms present in soil in comparison with untreated textiles.

This method is applicable to flat textiles made of cellulosic-containing yarns (tentage, tarpaulins, webbing and tapes) that will typically come into contact with soil during use.

Due to the inherent resistance of most synthetic fibres to attack by micro-organisms, fabrics containing a high proportion of synthetic fibres can only be judged by these methods for changes in structure and appearance.

Although this method allows good reproducibility of results, it is intended to show comparative performance rather than provide absolute values.

NOTE Heavy tarpaulin fabrics and webbing may be of such a structure that samples without finish are resistant within a 14 days soil burial period. Also in such cases the decay rate of the untreated sample determines the length of the burial period.

2 Normative references

This International Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this International Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

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

ISO 7218, *Microbiology of food and animal feeding stuffs — General rules for microbiological examinations*

ISO 8022, *Surface active agents — Determination of wetting power by immersion*

ISO 13934-1, *Textiles — Tensile properties of fabrics — Part 1: Determination of maximum force and elongation at maximum force using the strip method*

3 Safety precautions

This method requires the use of viable mould propagules and ambient conditions which promote mould and bacterial growth. Any safety precautions and personal hygiene for microbiological examinations shall be followed (e.g. ISO 7218).

4 Principle

Cellulose-containing textiles are considered resistant to attack by micro-organisms present in soil if their structure, appearance and tensile strength remain essentially unaltered during a soil burial test. This method compares the relative reduction in tensile strength of specimens before and after soil burial.

The method is used to compare finished and unfinished specimens of the same quality. The specimens are buried in a test soil of controlled water-holding capacity and optimum water content for microbial activity. The finished and unfinished specimens are buried for a period of up to nine days,

when the specimens without a finish show a reduction in tensile strength of approximately 80 %. At this stage the tensile strength loss of the finished and unfinished specimens are determined.

The limitation of the soil burial test to a maximum nine days for the unfinished specimens to lose 80 % of their initial tensile strength serves as validation of the microbial activity of the test soil and the test system.

NOTE 1 The duration of the test will vary depending on the activity of the soil. It is important to note that the reduction in strength between specimens with and without an applied finish is the critical factor, and not the duration of the test. A soil of lower activity will produce the same result as one of higher activity, but the test will take longer.

NOTE 2 A fixed-time soil burial test may allow an undefined attack on the cellulosic material. The reproducibility of results for reduction of tensile strength of finished and unfinished test specimens is the critical factor.

5 Apparatus and reagents

5.1 *Containers*, of unglazed pottery or reinforced cement (asbestos substitute materials), at least 150 mm deep.

Containers with more than 150 mm filling height shall have holes at the base for exchange of air.

5.2 *Test soil*, commercial grade compost or compost-containing garden soil as purchased in the manufacturer's original packing may be used. Fresh compost shall be mixed with compost previously used for soil burial testing or with soil previously acclimatized at equilibrium moisture to (95 to 99) % relative humidity and thoroughly mixed before use.

The test soil shall be sufficient to fill all the containers used in a series of tests, and shall be fine-grained, free-flowing and not sticky or lumpy. The water-holding capacity (WHC) (or maximum moisture retention capacity) of the test soil shall be determined and the water content (WC) shall be (60 ± 5) % of the WHC.

The pH of the test soil shall be between 4,0 and 7,5.

Prepare the test soil of known WHC for moisture content determination before commencing the test. If the soil is too moist, let it air dry. If the soil is too dry, overspray it to a WC of (60 ± 5) % of the WHC.

NOTE 1 See annex A for a suitable method.

NOTE 2 60 % of WHC guarantees best microbial activity and optimum water activity for microbial growth. The water content necessary for one soil is therefore always prescribed as approximately 60 % of WHC of the sample. Lower humidity slows down or prevents rotting; higher humidity may lead to unevenness of results and anaerobic processes.

NOTE 3 Soil blends with high clay or loam contents are unsuitable because of low air permeability and insufficient moisture distribution.

5.3 *Incubator*, for incubation of the soil burial containers during the entire test at (95 to 100) % relative humidity of the air at (29 ± 1) °C.

5.4 *Balance*, for determining WC and WHC of the test soil.

5.5 *Oven*, for drying soil samples at (104 ± 2) °C and for drying specimens at (45 ± 5) °C.

5.6 *Ethanol/water (70 %/30 % volume fraction) solution*, for cleaning the buried specimens before tensile strength determination.

5.7 *Microscope (x30 to x40 magnification)*, for visual examination of the buried specimens.

6 Test specimens

6.1 Preparation

Prepare test specimens from the middle of the laboratory samples by cutting the specimens lengthwise from the warp direction of the fabric.

With blended fabrics and/or if agreed between the interested parties, test specimens may be taken from the weft direction.

Blended fabrics containing 100 % cellulose fibres in the warp or weft direction shall be sampled by taking the test specimens from the 100 % cellulose direction only.

Cut test specimens 300 mm long and 30 mm wide and then fray down both sides to give a central width of 20 mm (see ISO 13934-1).

For fabrics that are too narrow to allow the specified specimen width, carry out the test at full fabric width.

NOTE An effective width of 20 mm and distance between the jaws of 100 mm is used due to the large quantities of untreated fabrics required and the capacity of the soil containers.

6.2 Number of specimens

For each test prepare 20 test specimens, 10 for testing without burial and 10 for testing after soil burial.

For determination of the soil burial duration, i.e. time at which there is an 80 % reduction in strength in the control specimens without applied finish, prepare at least 20 control specimens of the unfinished test sample. These control specimens shall be of the same fabric quality, i.e. same yarn(s), mass per unit area. construction/weave, preparation and dyeing with no additional applied finish such as a water repellent, softener or biocide.

NOTE For blends with synthetic fibres, see 8.3.

7 Leaching procedure

Unless otherwise agreed between the interested parties, subject all the finished and unfinished test specimens (i.e. those for testing without soil burial and those for testing after soil burial) to the following leaching procedure. Exclude the control specimens from the leaching procedure.

Hold the test specimens under fresh running tap water at (20 ± 5) °C for 24 h in a container large enough that the specimens do not touch each other. The flow rate shall be (10 ± 2)l/h.

After leaching, drain the test specimens, and oven dry (5.5) at (45 ± 5) °C. Condition the test specimens as specified in ISO 139.

Treat test specimens, both leached and unleached in parallel.

NOTE 1 Leaching at higher temperature is possible if agreed between the interested parties.

NOTE 2 Other treatments such as weathering and exposure to light may be carried out, if agreed between the interested parties.

8 Assessment of the level of microbial activity of the soil

8.1 Assessment of the level of microbial activity of the test soil is not required provided the test specimens remain buried until the tensile strength of the control specimens has been reduced by 80 % of their original value.

The level of microbial activity of the soil shall be monitored by the use of the control specimens. This time-trial is dependent on the microbial activity of the soil and shall not exceed nine days. If the time for 80 % reduction of tensile strength of the control specimens exceeds nine days, another soil sample and new test and control specimens shall be used.

8.2 If required, the microbial activity of the soil may be assessed using a standard cotton fabric, e.g. as specified in ISO 8022.

8.3 For tests with spun blends of cellulose fibres with synthetic fibres determine the test duration using a 100 % cotton fabric of construction and mass per unit area similar to the test specimen, so that a reduction in tensile strength of 80 % within 14 days is obtained.

9 Procedure

NOTE 1 The specimens are not autoclaved or chemically sterilized before the commencement of the test, because sterilization can affect many finishes.

NOTE 2 The specimens may be wetted with tap water just prior to the burial, if agreed between the interested parties and described in the test report.

9.1 Bury the 10 test specimens and at least 20 control specimens in two different containers in the soil in a U-form, with the centre portion of 150 mm in intimate contact with the soil. Press the soil lightly over the specimens.

Bury the specimens at least 50 mm apart.

Incubate the soil containers at (29 ± 1) °C at a relative humidity of 95 % to 100 %.

Maintain the water content of the soil throughout the test and replace any losses.

NOTE A foil covering helps to maintain the water content of the soil.

9.2 The water content may be assessed by weighing the soil containers at intervals of two weeks to four weeks. If necessary, replace water. The burial should last until unfinished specimens have lost 80 % of their initial tensile strength.

NOTE This incubation period can be determined by the following procedure: From the third day of burial to the seventh day, at intervals of two or three days, remove two control specimens each time. From day eight, or when 50 % reduction in tensile strength has been found, remove three control specimens each time at one- or two- day intervals.

Do not recondition these specimens. Lightly rinse the control specimens with running tap water and immerse in 70 % ethanol (5.6) for 30 min before drying at (45 ± 5) °C. Determine the tensile strength in accordance with ISO 13934-1.

9.3 Calculate the percent reduction in tensile strength of buried control specimens against unburied control specimens (see clause 10). Stop the burial when a comparative reduction of (80 ± 5) % is found for the control specimens.

9.4 Remove the remainder of the control specimens and the test specimens from the soil, rinse in running tap water and immerse in 70 % ethanol (5.6) for 30 min. Dry at (45 ± 5) °C and then condition in accordance with ISO 139 for a minimum of 48 h and a maximum of 120 h.

Immerse the unburied test specimens in tap water for 30 min, subject them to the same rinsing procedure and subsequently treat identically to the buried samples.

9.5 In addition to the tensile strength reduction (see 10.1), report any change in the structure or appearance of the buried specimens in comparison to the unburied specimens (see 10.2).

9.6 Determine the tensile strength of all the test specimens in accordance with ISO 13934-1.

Determine the water content of the soil at the end of the soil burial test using the method in annex A.

10 Calculation and expression of results

10.1 Calculate the relative reduction in tensile strength $q_{H,M}$ of the buried samples compared with the unburied samples from the mean values of at least 10 measurements, using the formula:

$$q_{H,M} = \frac{F_{H,E}}{F_{H,O}}$$

where

$F_{H,E}$ is the tensile strength of the buried test specimens;

$F_{H,O}$ is the tensile strength of the unburied test specimens.

If the reduction in tensile strength of the buried finished specimen is less than 25 % ($q_{H,M}$ is more than 0,75) a rot proof finish effect is considered to exist.

10.2 Assess the buried samples visually using a microscope (5.7) and report any changes, for example :

— colour change;

— partial or complete removal of cellulose fibres;

— appearance in transmitted light;

— disruption of surface structure.

Comparison shall be made between corresponding test specimens:

a) Unburied unleached with buried unleached;

b) Unburied leached with buried leached;

c) Unburied finished unleached with buried finished unleached;

d) Unburied finished leached with buried finished leached.

The finish of most fabrics consisting of synthetic fibers cannot be declared rot proof only on the basis of the reduction in tensile strength. For these fabrics, visual evaluation of the appearance is essential.

11 Test report

The following information shall be included in the test report:

a) number and year of publication of this international standard, i.e. ISO 11721-1:2001;

b) description and technical data of the sample, e.g. whether finished or unfinished;

c) test specimen pretreatment, e.g. leaching, weathering ;

d) water retention capacity of the soil;

e) water content of the soil at the beginning and at the end of testing;

f) individual tensile strengths and average values for the unburied test specimens;

g) individual tensile strengths and average values for the buried test specimens;

h) relative reduction in tensile strength;

i) appearance and structure changes of the buried specimens;

j) details of any deviations from the specified procedure;

k) dates of test procedure, e.g. leaching, beginning of burial, end of burial;

l) dates of removal of control specimens at early stages of the test with individual tensile strengths and average values;

m) name and address of test laboratory;

n) date of publication of the test report with the name of the responsible person ;

Annex A
(normative)

Determination of water holding capacity and water content of the test soil

A.1 Determination of the water content (WC)

In each of three glass Petri dishes spread one soil sample of approximately 50 ml. Then dry the dishes containing the soil sample at a temperature of (104 ± 1) °C until constant mass is obtained, i.e. when the soil, previously dried in an oven and cooled in a desiccator, does not lose more than 0,1 % of its initial mass during further 4 h of drying. This is usually achieved in a drying time of 4 h. For materials of unknown water holding capacity and/or for larger quantities, follow the procedure to constant mass; for soils of known composition empirical values may be used.

Carry out several weighings and calculations to determine the water content. Weigh to an accuracy of 1 mg. Round the results for WC to the nearest percent. Calculate the average of three determinations.

Example

a)	glass dish, empty		11,325 g
b)	dish containing fresh soil		20,475 g
c)	quantity of fresh soil	(b) – (a)	9,150 g
d)	dish containing dried soil		16,600 g
e)	dry soil	(d) – (a)	5,275 g
f)	WC(drying loss)	(b) – (d)	3,875 g
g)	WC,%	[(f)/(e)] x 100	73 %

Result: the water content amounts to 73 % of the dry mass.

A.2 Correction of water content from a lower to a higher value

The amount of water (W) necessary to change the water content of 400 g (q) of soil with a water content of 73 % (WC_0) to a higher water content of 85 % (WC_E) is calculated as follows:

Example for correcting the water content

$$W = \frac{q\left(WC_{\mathrm{E}} - WC_0\right)}{\left(WC_0 + 100\right)}$$

$$W = \frac{400\ (85 - 73)}{(73 + 100)} = 27{,}7\ \text{g}\ (\text{i.e. add } 27{,}7\ \text{g})$$

A.3 Determination of water holding capacity (WHC)

Fill up each of three glass filter crucibles (filter size 3; e.g. sD3) of capacity 50 ml with one soil sample 0,5 cm below the brim. Drop the crucibles three times from a height of 1 cm onto a wooden surface in order to compact the soil and then reweigh each crucible.

Place each crucible in a glass beaker. Pour water into the glass beaker until the water level reaches 1 cm above the filter plate of the crucible. As soon as the soil surface looks moist as a result of capillary sorption, add water up to the brim of the crucible.

After 12 h to16 h (overnight) remove the crucible from the beaker. Suck off the water not retained in the soil with a vacuum or water-jet pump for (10 ± 1 min). During this process cover the crucible with a wet cloth on which a glass plate is placed. After suctioning reweigh each crucible.

Oven dry the crucible containing the water-saturated soil at (104 ± 1) °C to constant mass, i.e. when the soil, previously dried in an oven and cooled in a desiccator, does not lose more than 0,1 % of its initial mass during a further 4 h of drying. This is usually achieved in a drying time of 4 h. For materials of unknown water holding capacity and/or for larger quantities, follow this procedure to constant mass; for familiar materials, empirical values may be used.

Carry out several weighings and calculations to determine the WHC. Weigh to an accuracy of 1 mg. Round the results for WHC to the nearest percent.

Example

a)	Glass filter crucible, empty		11,325 g
b)	Crucible containing fresh soil		20,475 g
c)	Fresh soil	(b) – (a)	9,150 g
d)	Crucible containing water saturated soil		24,105 g
e)	Soil, water saturated	(d) – (a)	12,780 g
f)	Crucible, containig dry soil		16,600 g
g)	Soil, dry	(f) – (a)	5,275 g
h)	Water holding capacity (in grams)	(e) – (g)	7,505 g
i)	Water holding capacity (in %)	(h)/(g)x100	142 %

Result: The soil is water saturated at a water content of 142 %. The maximum quantity of water the soil can retain amounts to 142 % of its dry mass.

A water content of 60 % of WHC of this soil would be 0,6 x 142 % = 85 % of its dry mass.

ICS 59.081.01

Price based on 8 pages