Flame Retardants for Textiles

Description of test methods and their application

Mildew & Rot Proof Standards

- ▣ DIN 53931; 1993
- ▣ DIN – Draft 4 & DIN 53933 Part 1; 1992

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DIN 53931

Mildew Resistance of Textiles

1. Scope:

This growth test is intended for determinating the susceptability of textiles to mildew. Furthermore, the stability of mildewproof finishes to watering, weathering, exposure to light, etc. shall be evaluated. This test applies to both technical textiles and home textiles/apparel.

2. Sample preparation:

a) Sterilisation of the equipment

* Sterilisation of Erlenmeyer flasks, measuring cylinders and test tubes Cork them with cotton wool and sterilize them in the drying cabinet at 200 °C for 5 – 10 min. Remove sterilized cotton wool only shortly before use.

* Sterilisation of water Pour distilled water in bottles and place lids upon them (do not screw them shut because of the risk of overpressure). Sterilize at 110 °C for 20 min. in the pressure cooker containing 1/4 l water (1st ring visible). Pressure cooker: Fissler vitavit

* Sterilisation of pipettes Place one-way pipettes in a metal container with a piece of cotton wool at its bottom, close the container and sterilize it at 200 °C for 5 – 10 min.

Cut the test fabric into square cloths of 5 x 5 cm.

b) Preparation of culture medium

The following recipe, which gives 1 l culture medium, is sufficient for

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40 –50 petri dishes or

150 – 200 test tubes

10 grolled oats, ground

20 g malt extract
20 g agar-agar
1000 ml water

Flood 10 g rolled oats in a pot with 400 ml cold water. Dissolve 20 g malt extract in 200 ml cold water and pour it in the pot. Bring the whole mixture to the boil. Dissolve 20 g agar-agar in 200 ml cold water and add it to the heated mass. The rest of the water is intended for rinsing the beakers. Bring the whole mass shortly to the boil once again and fill it in preserving jars. The bottles with their lids laid upon shall then be sterilized with the culture medium in the pressure cooker at 110 °C for 20 min. The culture medium that shall still be hot shall be stirred with a glass stick previously sterilized with a flame. Fill it then immediately in a sterilized 100 ml measuring flask. From then on absolute sterility shall be maintained. 20 – 25 ml shall be poured from the measuring cylinder in one petri dish each. For sterility reasons, the lid shall only be touched on the outside and shall not be put down. The petri dish shall be closed again immediately. Allow it to cool it down to room temperature before placing it in the refrigerator. The petri dish may then be used.

c) Preparation of inclined agar

The culture medium shall be prepared as described above. The test tubes shall be placed in grille boxes provided for this purpose. After filling 5 ml culture medium in one test tube each, the tubes shall be closed with aluminium lids. Sterilize the grille box with the filled and closed test tubes in

a pressure cooker at 110 °C for 20 min. Remove the grille box and put it immediately at an angle by partly placing it on a piece of wood. Allow it to cool down to room temperature in this position before placing it in the refrigerator. The inclined agar may then be used.

d) Growth of fungus strains After transferring the fungus culture from the sealed ampoule to a sterile test tube containing 2 ml sterile water, this test tube shall be shaken slightly. 1 ml solution shall be added to each prepared inclined agar with a sterile pipette.

After one week the mildew shall be evaluated for the first time. If it is considered to be good, it shall be placed in the refrigerator. If it has not developed completely, it shall remain in the incubator. When kept cool, the mildew may be stored for up to 6 months.

e) Growth of the fungus culture Each mildew is cultivated on oats–malt–agar respectively in an inclined agar tubes (test tubes with agar hardened at an angle). The inclined agar tubes shall be inoculated with a loop. Use one loop for each mildew. Allow it to cool down completely before use. Some mildew shall be removed from the tube containing the fungus strain by means of the sterile loop. Open a prepared inclined agar tube and distribute the mildew gently without hurting the surface of the agar. Close the tube and put it in the incubator. As soon as the cultures show good spore formation after incubation at 29 +/– 1 °C (if necessary at room temperature), they may be stored at 5 – 10 °C (if necessary at room temperature). When kept cool, they may be stored for up to 2 weeks.

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3. Working method:

a) Suspension of spores

Flood twice with 5 ml sterile water each to gain spore suspension.

b) Inoculation of petri dishes

Put 0.5 ml spore suspension on the prepared culture medium with a sterile pipette. The liquid shall be distributed evenly over the whole surface with a glass stick bent in a right angle (so-called Drigalski spatula). The glass stick had been sterilized over a flame before use. The culture medium plates shall be incubated at 29 +/- 1 °C immediately after inoculation.

c) Preparation of specimens

After 24 hours each specimen and control specimen shall be placed on an inoculated, still humid culture medium and slightly pressed against it with a glass stick bent in a right angle or with tweezers.

d) Incubation period

Unless otherwise specified, the test dishes shall be incubated at 29 +/- 1°C. The relative humidity shall be kept between 60 – 80 % by additionally placing water dishes in the incubator in order to prevent the culture medium from drying out. Unless otherwise specified, the incubation period is 2 weeks; than the specimens shall be evaluated after that time.

4. Evaluation:

At the end of the incubation period the specimen will be examined for the following (by means of a magnifying glass if necessary):

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-Both the samples to be evaluated and the control samples have to be examined as to whether mildew growth on the samples and the culture medium has been caused by the test mildew itself or by foreign organisms (impurities). If foreign organisms grew to a large extent, the test would be considered inconclusive and would have to be repeated.

-The samples shall be evaluated for growth on the surface and the inten-sity of that growth.

-Growth of the culture medium surrounding the specimen shall be assessed. If there is a growth-free zone around the sample, its dimensions shall be measured (possibly by indicating the smallest and largest distance to the edge of the specimen; do not consider those parts of the sample that are not in direct contact with the culture medium).

-Look under the sample to determine possible growth underneath.

-Consider if the sample shows change in colour or decomposition.

If the sample is growth-free of any type of mildew, the mildew-resistant finish shall be classified as efficient. By the end of the incubation period limited growth may be noticed on the extreme edge of the sample (mostly without spore formation). This should not exclude the sample being qualified as mildew-resistant. This especially applies to copper-containing finishes exposed to Aspergillus Niger.

When a sample is surrounded by a more or less growth-free zone, the active substance of the finish may have diffused into the culture medium (because of its water solubility or evaporation). In extreme cases, growth on the culture medium is inhibited completely. This shall be considered in the

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following assessment scheme with regard surface growth and growth intensity.

Key

00 total culture area free of growth 0 halo formation (growth-free zone surrounds sample)
(0) fungus growth right up to sample 1 growth on edge of sample only 2 growth from edge of sample inwards (less than 25%) 3 growth of individual colonies on sample (25 to 75%) 4 extensive growth on sample surface (75% and above, but
not the whole surface) 5 sample completely covered (100%)

Intensity of growth

+ slight growth (mostly mycelium only) ++ considerable growth, some spore formation
+++ heavy growth and heavy spore formation

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DIN Draft 4 + DIN 53933 Part 1

Mildew Resistance of Cellulosic Textiles (Soil Burial Method)

1. Scope:

This method is intended for evaluating the efficiency of mildew-resistant finishes exposed to microorganisms in the soil. It applies to cellulosic textiles (e.g. tents, tarpaulins, belts, ribbons) and threads (e.g. cords, yarns, sewing cotton) where occasional contact with soil is to be expected. On blends with synthetic fibres, only a modified soil burial method is conclusive because their specific resistance to microorganisms mostly inhibits any impact on their tensile strength by the soil burial test.

Note: Although this procedure is easy to reproduce, the results obtained according to this standard may not be seen as absolute, but only relative within one soil burial test.

2. Preparation of specimens

Laboratory samples shall be taken from the middle of textile fabrics suitable in size. The samples shall then be cut out of the middle of the laboratory samples. Their long direction shall be parallel to the warp, which guarantees higher regularity. Exceptionally, for example to test blends, the specimens may also be cut out parallelly to the weft when agreed upon. The gauge length between the clamps shall be 65 mm. The samples shall be cut out in a width of 30 mm and ravelled 5 mm on both sides to obtain a definitive width of 20 mm. Specimens of the same fabric shall have the same number of threads in length. Considering the pretension weight, the total length of a specimen should be approximately 150 mm to correctly lock the samples.

Note: The effective width of each specimen with 65 mm gauge length between the clamps shall be 20 mm, because considerable amounts of non-finished material are required for the tensile strength test. Furthermore, the capacity of the soil boxes is limited, considering the number of specimens necessary.

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DIN Draft 4 + DIN 53933 Part 1

Mildew Resistance of Cellulosic Textiles (Soil Burial Method)

When test fabrics have been treated with non-readily water-soluble finishes, samples shall be taken and watered as described below, unless otherwise specified: Laboratory samples shall be taken from the middle of the fabric. These samples shall be leached for 24 hours in tap water of 15 to 20 °C before the test. The water container shall be big enough to ensure that the samples are surrounded by water on each side and that they do not touch each other. Water flow is adjusted to ensure a change of water 5 times per hour. After leaching the textiles shall be hydroextracted (squeezed or centrifuged) before predrying them in warm air of max. 60 °C. Leached and non-leached samples shall be tested in the same way. In special cases further pretreatment may be required, e.g. weathering or irradiation of the textile.

3. Procedure:

Before the test the specimens shall be watered for 20 min. The non-finished and finished samples shall be buried separately in the test soil vertically in U-shape. Only one end of the specimen's leg shall stand out of the test soil. During the burial, the test soil shall be pressed gently on the specimen.

Note 1: As many finishes risk to be changed by sterilization, the samples shall not be sterilized before the test.

The distance between the specimens and the individual sample series shall not be below 50 mm. After covering the soil boxes containing the specimens tightly with a film to maintain humidity, they shall be stored in a damp atmosphere (95 – 100% air humidity) in a drying cabinet or incubator with regular, allsided heat exposure.

Note 2: A water basin placed in the drying cabinet or incubator maintains the damp atmosphere. The moisture content of the test soil shall be kept at 50 60%, based on the dry weight of the soil, and monitored during the test

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period. Water lost during use must be replaced evenly if necessary.Experience shows, however, that the film cover is sufficient to maintain humidity.

The specimens shall be removed from the soil after 14 days (exceptionally 21to 28 days if specified).
The removed specimens shall be rinsed with cold water and dried on a mesh in the drying cabinet at 60 °C for min. 4 hours.
Their tensile strength shall then be determined in the test laboratory.

4. Apparatus:

4.1 Containers for the soil (soil boxes)

Containers of fibrous cement (replacement of asbestos) or unglazed clay shall be used for the storage of the soil and for the soil burial test. Containers of glass, plastic or other airtight material are inappropriate. The soil boxes shall be deep enough for soil beds of 75 mm, corresponding to U-shaped burials, and easy to be closed with films.

4.2 Test soil

New wrapped potting compost shall be used as the test soil. The well mixed test soil shall be fine and loose. It shall not tend to pack closely or become sticky. For the soil burial test, the soil shall have 60 +/- 5 % moisure (based on the dry weight of the soil).

Note 1: Before starting the test, the test soil shall be spread on a film to adjust its moisture to 50 – 60% by spraying it with water and air-drying it. It shall then be mixed thoroughly. This is especially important when several soil boxes are used for one test series. The moisture content FG shall be determined in percent by drying three soil samples at 105 °C for 4 hours and calculated as follows:

$$FG = \frac{\text{(weight of moist soil - weight of dry soil) x 100}}{\text{weight of dry soil}}$$

Note 2: Higher moisture does not speed up the soil burial test, but may lead to irregularities. Lower moisture, however, slows rotting down. Soil mixtures with high clay content are inappropriate because of their low air permeability and insufficient moisture distribution.

5. Evaluation:

The maximum tensile strength of all samples shall be determined according to DIN 53857 Part 1. The relative loss in maximum tensile strength M of the buried samples in comparison with the maximum tensile strength of the corresponding non-buried samples, based on the average rating of at least 10 samples, shall be calculated as follows:

$$M = \frac{FO - FE}{FO} \times 100$$

FO = Maximum tensile strength of the unburied sample
FE = Maximum tensile strength of the buried sample

Compare those samples that belong together, i.e.
*non-buried, non-leached with buried, non-leached
*non-buried, leached with buried, leached
*non-buried, finished, non-leached with buried, finished, non-leached
*non-buried, finished, leached with buried, finished, leached

Efficiency of the rot resistance shall be given when the loss of the buried samples in maximum tensile strength does not exceed 25%.

Apart from the loss in max. tensile strength it shall be determined in as much as the samples tested acc. to the soil burial method differ from the non-buried samples in structure and appearance. This is often the only possibility for evaluating textiles containing synthetic fibres, as their change in max. tensile strength tends to be low or inexistent.