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Geotextiles and geotextile-related products — Screening test method for determining the resistance to oxidation

Géotextiles et produits apparentés — Méthode de détermination de la résistance à l'oxydation

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Foreword

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ISO 13438 was prepared by the European Committee for Standardization (CEN) Technical Committee CEN/TC 189, *Geosynthetics* in collaboration with Technical Committee ISO/TC 221, *Geosynthetics*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

Introduction

In many civil engineering applications geotextiles and geotextile-related products may come into contact with water or aqueous solutions present in the soil environment. At the same time, in specific parts of the construction, they may be exposed to oxygen, giving rise to oxidative degradation processes. These processes are usually very slow.

Polyolefin materials such as polypropylene (PP) and polyethylene (PE) are inherently more sensitive to oxidation than those based on polyethylene terephthalate (PET). This behaviour can be improved very effectively by the use of appropriate stabilizing additives.

It is the purpose of this international standard to provide a method for screening the resistance to oxidation of geotextiles and geotextile-related products in service up to 25 years. In order to achieve the sufficiently short exposure times needed for screening tests, it is necessary to accelerate the oxidative degradation process. This acceleration can be achieved either by raising the temperature or by increasing the concentration of the active reaction partner. Raising the temperature may lead to the oxidation rate being limited by oxygen diffusion, thus invalidating the acceleration. This applies particularly to materials with a low surface-to-volume ratio and less to nonwovens made from fine fibres. Two methods are therefore proposed.

Methods A1, A2, B1 and B2 use temperature alone as the accelerating factor.

Methods C1 and C2 operate at moderately high temperatures and at the same time the oxygen concentration is increased by using pure oxygen at high pressure.

Each test may be performed at a shorter duration for non-reinforcing materials (A1, B1, C1) or for a longer duration for reinforcing materials (A2, B2, C2).

NOTE This International Standard should be used with reference to ISO/TR 13434. For further information see Annex A.

INTERNATIONAL STANDARD **ISO 13438:2004(E)**

Geotextiles and geotextile-related products — Screening test method for determining the resistance to oxidation

1 Scope

This International Standard specifies a screening test method for determining the resistance of geotextiles and geotextile-related products to oxidation. The test is applicable to polypropylene- and polyethylene-based products.

The data are suitable for screening purposes but not for deriving performance data such as lifetime unless supported by further evidence.

2 Normative references

The following referenced documents are indispensable for the application 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 188:1998, *Rubber, vulcanized or thermoplastic — Accelerated ageing and heat resistance tests*

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

EN 12226, *Geotextiles and geotextile-related products — General tests for evaluation following durability testing*

3 Methods A1, A2, B1 and B2

3.1 Principle

Test specimens are exposed to an elevated temperature in air over a fixed time period, using a regulated laboratory oven without forced air circulation.

Oven ageing on polypropylene shall be carried out at a temperature of (110 ± 1) °C (Methods A1 and A2).

Oven ageing of polyethylene shall be carried out at a temperature of (100 ± 1) °C (Methods B1 and B2).

The test specimens shall hang freely in the oven space.

After the fixed time period of oven ageing, the exposed test specimens are submitted to a tensile test. The tensile strength and the strain at maximum load are measured for both the control specimens and the exposed specimens. The tensile test shall be carried out in accordance with EN 12226. For woven fabrics both the machine and cross direction shall be tested, unless otherwise agreed.

3.2 Specimens

Products shall have been manufactured at least 24 h prior to testing. The number of specimens shall be five test specimens and five control specimens, unless further specimens are required to assure statistical significance.

The specimens to be tested shall be in accordance with EN 12226.

NOTE It is recommended to expose additional specimens in case an extra mechanical test is required.

3.3 Apparatus

For the tests, a thermostatically regulated oven without forced air circulation, in accordance with 3.2.2 of ISO 188:1998, is required, with an internal volume of sufficient size, capable of exposing test specimens to a temperature of (110 ± 1) °C or (100 ± 1) °C.

The oven without forced air circulation shall be provided with a ventilation opening which shall be adjusted such that the set temperature can be maintained in that part of the oven in which the specimens are to be suspended.

The specimens shall be suspended from glass or other chemically inert fixtures in the centre of the oven, spaced and not touching; the distance from each wall being at least 100 mm.

The temperature around the specimens shall be recorded, for instance, with the aid of suitable calibrated thermocouples and a data logger.

3.4 Conditioning

Conditioning of the specimens before exposure in the laboratory oven is not required. Because of the possible occurrence of shrinkage during the oven test, the control specimens shall be exposed for 6 h, to the same conditions as in the oven test.

3.5 Test procedure

3.5.1 Oven temperature

Set the oven temperature at (110 ± 1) °C or (100 ± 1) °C in accordance with the chosen method (A1, A2, B1 or B2).

3.5.2 Specimens

Attach the specimens to the fixtures. Once the temperature has reached a steady value, place the specimens in the oven. Suspend the specimens in the centre of the oven, spaced, not touching each other, and so that the distance from each wall is at least 100 mm.

3.5.3 Duration of the oven test

Geotextile specimens for reinforcing applications, or for other applications where long-term strength is a significant parameter, shall be exposed to the durations shown in Table 1.

Table 1 — Temperatures and durations

Method	Material	Application of material	Temperature	Duration
A1	polypropylene	non-reinforcing	110 °C	14 days
A2	polypropylene	reinforcing	110 °C	28 days
B1	polyethylene	non-reinforcing	100 °C	28 days
B2	polyethylene	reinforcing	100 °C	56 days

The control specimens shall be exposed to the same oven temperature for (6 ± 1) h and then removed and stored.

NOTE Practical experience has shown that, to achieve good reproducibility, the following should be taken into account:

a) the specimens should be placed in the middle of the oven;

b) draughts near the oven should be avoided if a reproducible natural air circulation is to be maintained;

c) the oven and the fixtures should be cleaned of any remaining residues before each new test;

d) thermo-oxidative degradation of polymer material (e.g. polypropylene) may release substances which have a catalytic effect; therefore, polymers containing different stabilizers should not be tested at the same time in the same oven, with the exception of geotextile composites.

4 Methods C1 and C2

4.1 Principle

Test specimens are exposed for a specified time to an aqueous test liquid enriched with oxygen at a pressure of 5 000 kPa and at a temperature of 80 °C. Method C1 specifies a duration of 14 days and Method C2 (intended for reinforcing applications or those where tensile strength is a relevant parameter) specifies 28 days.

The properties of the specimens are tested after this exposure in accordance with EN 12226. The tensile strength and the strain at maximum load are measured for both the control and the exposed specimens.

4.2 Apparatus and reagents

4.2.1 Apparatus

4.2.1.1 A pressured vessel (autoclave), large enough for the test liquid (see 4.3.1) that shall cover the specimens completely during the test. The free space above the liquid should be at least 20 % of the liquid volume. The material of the vessel and equipment shall be resistant to the test liquid under the conditions used, e.g., high-grade stainless steels.

4.2.1.2 A pressure transducer, to measure the oxygen pressure above the test liquid, with a precision of ± 50 kPa.

4.2.1.3 A temperature sensor, to measure the temperature, with a precision of ± 0,5 °C.

4.2.1.4 Specimen holders, to ensure correct placing of specimens (see 4.3.2).

4.2.1.5 Stirring device, to maintain the homogeneity of solvent, solutes and temperature, and to allow exchange of matter between specimens and solvent.

4.2.1.6 **Valves,** for filling the vessel with oxygen and for emptying the vessel.

4.2.1.7 **Drain-off valve,** to drain the solvent from the vessel after testing.

4.2.1.8 **Heating device,** to maintain the test liquid at a constant temperature of (80 ± 1) °C.

4.2.1.9 **Monitoring device,** for regular monitoring (at least every 15 min) temperature and pressure inside the vessel.

4.2.1.10 **Pressure release safety equipment,** if required by safety regulations.

4.2.2 Reagents

4.2.2.1 **Aqueous solution of $NaHCO_3$,** 0,01 mol/l, adjusted to pH 10,0 at 20 °C with NaOH 1 mol/l.

4.2.2.2 **Oxygen,** at a purity of at least 99,999 % by volume.

4.2.2.3 **Water,** in accordance with ISO 3696.

4.2.3 Specimens

Products shall have been manufactured at least 72 h prior to testing. The number of specimens shall be five in each test direction in accordance with EN 12226

The specimens to be tested shall be in accordance with EN 12226. For woven fabrics both the machine and cross direction will be tested, unless otherwise agreed.

NOTE Recommended to expose additional specimens in case an extra mechanical test is required.

4.3 Test procedure

4.3.1 Quantity of the test liquid and of the gas phase

The mass of the test liquid shall be greater than 20 times the mass of the specimens. The free space above the liquid shall be at least 20 % of the volume of the liquid. The liquid shall cover all specimens completely during the whole test.

4.3.2 Positioning and installing the specimens

The specimens shall be held in place by a specimen holder made from an inert material. Position the specimen holder in the vessel such that the mean distance between the specimens and the walls of the vessel, between the specimens and the surface of the liquid, and between one specimen and another, shall be at least 1 cm.

Fill the autoclave with the correct volume of liquid and apply a pressure of 5 000 kPa for a minimum of 16 h while stirring the liquid and maintaining a temperature of (80 ± 1) °C. Adjustment of the pressure is not necessary.

Slowly release the pressure and open the autoclave. Adjust the intensity of stirring the liquid such that no oxygen bubbles are introduced into the liquid and that the distance between the specimens and the liquid surface is maintained. Close the autoclave and increase the oxygen pressure steadily over approximately 5 min. The total time for depressurizing, loading and pressurizing should not exceed 30 min.

NOTE This process should restore the test temperature, ensure enrichment of the liquid with oxygen and remove other gases dissolved in the liquid or present in the free space in the vessel.

4.3.3 Test duration

Method C1: 14 d.

Method C2: 28 d.

4.3.4 Procedure during exposure

During the first 24 h maintain the pressure inside the vessel at (5 000 ± 200) kPa. This can be achieved by several pressure adjustments especially in the first hours following loading. After this period maintain it at (5 000 ± 100) kPa. Maintain the temperature at (80 ± 1) °C.

NOTE The changes of pressure are caused by the solution process of oxygen in the liquid and by possible leaks in the system, which should be detected and eliminated.

After the initial 24 h, keep the oxygen pressure, the temperature of the liquid and the intensity of stirring constant. Record the pressure and temperature regularly.

4.3.5 Removing the specimens

On completion of exposure, reduce the oxygen pressure gradually over a period of about 5 min, open the autoclave and remove the specimens from their holder.

Rinse the specimens in deionized water and condition them in accordance with EN 12226.

4.3.6 Control specimens

Expose the control specimens to the same liquid and temperature under atmospheric conditions for (24 ± 2) h and then rinse and condition them in the same way as the tested specimens.

NOTE 1 After each test, the vessel and its equipment should be carefully cleaned of any remaining residues.

NOTE 2 Because of the possible effect of such residues on the oxidative testing process, no products manufactured using different polymerization processes or containing different additives should be tested in the same oven at the same time.

The national safety regulations for handling and for disposal of test liquids and for working with pressured oxygen should be followed. For safety reasons it is essential that the specimens tested be completely covered by the test liquid during exposure to pressured oxygen.

5 Determination of mechanical properties

The following applies to all methods.

When the fixed time period of oven ageing has elapsed, remove the specimens and test them in accordance with EN 12226. Determine both the tensile strength and the strain at maximum load for both the control specimens and the exposed specimens. Determine the ratios (as percentages) of the properties of the exposed specimens to those of the control specimens.

6 Test report

The test report shall include the following information and applies to all methods.

a) reference to this International Standard, i.e., ISO 13438;

b) method used (A1, B1, A2, B2, C1 or C2);

c) test laboratory;

d) identification of the product tested;

e) for methods A1, A2, B1 and B2: type and internal volume of oven, observation of the effect of temperature on the control specimen;

f) for methods C1 and C2: quality of oxygen used, liquid temperature and range, pressure of vessel and range;

g) test duration;

h) results, expressed in accordance with EN 12226;

i) any deviation from this International Standard or any factor likely to have influenced the results;

j) dates of testing.

Annex A
(informative)

Background information on oxidation processes and oxidation measurements

The principal cause of degradation of polyethylene and polypropylene is oxidation, i.e. the reaction of the polymer with oxygen. It leads to rupture of the main polymer chain, reduction in molecular weight and a corresponding reduction in mechanical strength. Other effects are embrittlement, surface cracking and a change in colour.

Oxidation of polyolefins is a chain reaction whose chemistry is complex but quite well understood. The chain reaction is started by radicals, which may be produced by energizing radiation such as ultraviolet light (photo-oxidation) or by heat, and may be accelerated by catalysts such as ions of heavy metals, including iron. The high processing temperature of polyethylene and polypropylene will lead to oxidation taking place while the polymer is being moulded.

All commercial polyolefin materials contain stabilizers, especially antioxidants, to reduce oxidation. Antioxidant batches usually include hindered phenols and/or hindered amine (light) stabilisers (HALS or HAS), phosphites and sulfur compounds. These intercept the chain reaction in various ways, leading to a dramatic reduction in the rate of oxidation and extending lifetime hundreds or thousands of times over, if the batch is properly designed. Commonly there is a combination of stabilizers: phosphites and hindered phenols are active at the high processing temperatures, while sulfur compounds usually act as co-stabilizers, whilst HALS and HAS, and certain types of phenol stabilizers inhibit the long-term oxidation at lower temperatures. Polymers stabilized with some antioxidants are observed to degrade rapidly once the antioxidants have been consumed, while polymers stabilized with others, such as HALS or HAS, are observed to degrade more slowly. Antioxidants can be lost prematurely by migration, evaporation, leaching and may be deactivated by other additives, by reaction or simple adsorption, or by incompatibilities arising in the polymer compound. Appropriate stabilizer formulations are known and applied to ensure long-term durability.

The degradation of a stabilized polymer can be divided into three stages: i) reaction with surplus antioxidant; ii) consumption of the antioxidant; iii) degradation of the unprotected polymer. To establish which stage has been reached for some master batches, use is made of oxidation induction time (OIT) measurement. In this method a sample of material is heated to a high temperature (typically 200 ºC) in an inert atmosphere, pure oxygen is admitted and the time to oxidation measured thermally by differential scanning calorimetry (DSC). For materials in the first stage of oxidation the OIT is unchanged. In the second stage the OIT reduces progressively as the antioxidant is consumed, but the mechanical properties remain unchanged. In the third stage the OIT is low and the mechanical strength and elongation at break diminish. This method cannot be applied universally because it only relates to antioxidants active at the testing temperature; the reaction is performed in the molten state of the materials tested. This limitation has to be taken into account.

Typical durations for the three stages at room temperature have been estimated as tens or hundreds of years for various geotextiles (Elias *et al.* [3]). All three stages are accelerated by heating, but the rate at which each stage is accelerated depends on temperature in a different manner. To predict lifetime it is necessary firstly to define the “end of life”, the point at which the material is regarded as no longer fulfilling its engineering function, e.g., a 50 % drop in strength. The duration of each stage is then measured as a function of temperature and extrapolated to lower temperatures and longer times using Arrhenius’ formula. Finally the individual extrapolated durations are added to give the total predicted lifetime.

This procedure is costly and time-consuming. Historically, as in Methods A and B, overall lifetime has been predicted by treating the entire oxidation process as a single stage. Time to end-of-life is measured at different temperatures and extrapolated to the service temperature to define the service life, or to the service life to define the operating temperature. In the past, this has suffered from inaccuracies due to the following:

- the oven temperature cannot be taken above the melting temperature of the polymer, or, for semi-crystalline materials such as PE and PP, the crystalline melting temperature, thus restricting the exposure temperatures to a narrow range;

- the mechanism of oxidation can change at higher temperatures, invalidating the extrapolation;

- the different rates of degradation described above can lead to large errors in the measurement of time to end-of-life and of its extrapolation;

- surface cracking has been observed in certain grades of polypropylene; this increases the access of oxygen to the polymer and invalidates any prediction based on uncracked material;

- diffusion of antioxidants plays a major part at all stages of oxidation: the rate of diffusion of oxygen from the outside, the rate of diffusion of antioxidants, and the rate of migration of radicals produced by the chain reaction all increase at higher temperatures and decrease with crystallinity and orientation of the polymer. These effects are even accelerated by a high surface-to-volume ratio. Hence a high surface-to-volume ratio and a low degree of orientation will clearly shorten all stages of oxidation. This is a well-known phenomenon;

- leaching may also occur in materials having a high surface-to-volume ratio or containing leach-sensitive antioxidants. For these materials, correct selection of stabilizers is essential.

Polyethylene and polypropylene geotextiles cover a wide range of structures from fine, highly oriented fibres to thick and less strongly oriented geosynthetic barriers. They contain different combinations of antioxidants. Some exhibit surface cracking. In synthetic barriers and the less oriented areas of extruded geogrids the rate of oxidation should be higher due to the lack of orientation, but simultaneously lower due to the small surface-to-volume ratio.

This explains why it has been proven impossible to define a single oven ageing test as a screening test for all geotextiles. Various attempts to do so have either failed to eliminate poorly stabilized material or conversely have eliminated material which would be expected to be durable. Better results may be obtained by restricting the temperature to 80 °C or below, but then the test duration may be extended to months or years, which is unacceptable for a screening test. Even then the accuracy achieved may still be unsatisfactory. Further, oxygen diffusion and limited oxidation may be apparent even under these conditions (forced air circulation and 80 °C).

The rate of oxidation depends on the availability of oxygen. Disturbed coarse aggregates generally assure sufficient access to oxygen and the rate of oxidation in the soil is not expected to be substantially less than that in air; in fine-grained soil backfills with no soil cracking the oxygen ratio is less (Hsuan *et al.*[4]). It is assumed that under these natural in-soil conditions oxidation is so slow that oxygen diffusion is not rate-limiting. Under these conditions the thickness of materials is of no direct advantage with regard to resistance to oxidation. This situation is comparable to resistance of PET materials to inner hydrolysis.

Therefore raising the availability of oxygen by using pure oxygen gas under pressure presents an alternative method of acceleration. It compensates the distortion of rate of oxidation found by limiting oxygen diffusion in products with a high surface-to-volume ratio in oven testing at elevated temperatures and accelerates oxidation to a certain degree due to a higher oxygen concentration in all the materials. Further, the test is performed with the geotextiles suspended in an aqueous phase in order to simulate leaching effects which could be especially serious for materials with a high surface-to-volume ratio.

Methods C and D have been shown to separate poorly-stabilized from well-stabilized geotextiles. Other methods may be acceptable provided that they demonstrate plausibly that a geotextile has sufficient durability for the intended design lifetime.

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