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Plastics — Methods for marine exposure

Plastiques — Méthodes d'exposition aux intempéries marines

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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 the member bodies casting a vote.

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

ISO 15314 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 6, *Ageing, chemical and environmental resistance*.

Introduction

Plastics are often used in outdoor applications where they are immersed or partially immersed in water. In some cases, materials made from plastic are designed to float on water. In others, plastic articles that are discarded end up as floating debris. In addition to the effects of sunlight and heat, plastic polymers or products exposed in marine environments may be subjected to hydrolysis, water absorption, extraction of stabilizers, erosion by wave action, corrosion by salts and/or attack by seaborne microorganisms. These stresses are not simulated in typical weathering exposures conducted in accordance with ISO 877. Therefore a separate standard is necessary to define procedures that realistically and consistently stress plastic materials in the same way that they would be in products used or discarded in marine environments. This International Standard describes three procedures for the exposure of plastic materials in the same way as they could be when used in marine environments.

There are four primary reasons why the rate of degradation of plastics exposed at sea can be different from that for the same plastic exposed on land[1]:

a) exposure in moist conditions is known to accelerate degradation of some polymers — small amounts of absorbed water may act as a plasticizer, increasing accessibility of the matrix to oxygen, or may leach out stabilizing additives;

b) differences in heat build-up between plastics exposed in water or on the surface compared to plastics exposed on land;

c) the action of microorganisms that may shield the plastic from UV radiation or may enhance biodegradation processes;

d) the action of macroorganism settlements that can produce disfigurement of surfaces.

It is essential to establish appropriate exposure procedures in order to properly assess the performance of plastics used in marine environments, and to evaluate how long plastics discarded as litter will persist in marine environments.

Plastics — Methods for marine exposure

1 Scope

This International Standard describes three methods for the exposure of plastics in a marine environment. Method A covers exposures where specimens float on the surface, method B covers exposures where specimens are partially immersed and method C covers exposures where specimens are completely immersed. Although intended for marine (salt water) exposure, the methodology might be used with outdoor brackish water and fresh-water exposures as well. Direct weathering of plastics on land is described in ISO 877.

Method A is particularly applicable to enhanced-degradability plastics where the environmental degradation under marine floating exposure is expected to be accelerated relative to that of regular plastic materials.

This International Standard specifies the general requirements for the apparatus, and procedures for using the test methods described.

It lists properties that may be used to evaluate changes in plastics subjected to marine exposure. More specific information about methods for determining the changes in properties of plastics on exposure and reporting these results is given in ISO 4582.

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 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 293, *Plastics — Compression moulding of test specimens of thermoplastic materials*

ISO 294-1, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 1: General principles, and moulding of multipurpose and bar test specimens*

ISO 294-2, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 2: Small tensile bars*

ISO 294-3, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 3: Small plates*

ISO 295, *Plastics — Compression moulding of test specimens of thermosetting materials*

ISO 877, *Plastics — Methods of exposure to direct weathering, to weathering using glass-filtered daylight, and to intensified weathering by daylight using Fresnel mirrors*

ISO 2818, *Plastics — Preparation of test specimens by machining*

ISO 3167, *Plastics — Multipurpose test specimens*

ISO 4582, *Plastics — Determination of changes in colour and variations in properties after exposure to daylight under glass, natural weathering or laboratory light sources*

3 Terms and definitions

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

3.1
control
⟨weathering⟩ a material which is of similar composition and construction to the test material, and which is exposed at the same time for comparison with the test material

NOTE An example of the use of a control material would be when a formulation different from one currently being used is being evaluated. In that case, the control would be the plastic made with the original formulation.

3.2
file specimen
portion of the material to be tested which is stored under conditions in which it is stable, and is used for comparison between the exposed state and the original state

4 Principle

4.1 General

Replicate specimens of the appropriate size and shape are exposed floating on the surface of water, partially immersed in water or completely immersed in water. After the prescribed exposure interval, the specimens are removed from the water and tested/examined for changes in chemical, physical and/or appearance properties. In addition, the specimens may be tested for the type and severity of microbial growth or biofouling. Unless otherwise specified, test specimens are exposed in an unstrained state.

The exposure intervals at which the specimens are tested/examined are typically defined in terms of a given length of time. In some cases, however, the exposure interval may be expressed in terms of the total solar or solar ultraviolet radiation dosage. The climatic conditions are monitored during the exposure and reported with the other conditions of exposure.

4.2 Significance

The relative durability of materials in marine exposures can vary depending on the location of the exposure because of differences in ultraviolet radiation, ambient air temperature, water temperature, microorganisms, tidal action and contaminants in the water. Therefore, it cannot be assumed that results from one exposure in a particular location will be useful in determining the relative durability in another location. Exposures in several locations which represent a broad range of anticipated service conditions are recommended.

Exposure of the same material for the same length of time at different marine sites is not expected to result in identical degrees of degradation. This is also true for exposures at the same site, but during different seasons or in different years. Thus, the length of exposure is only a general indication of the extent of exposure and should always be considered in relation to the characteristics of the exposure site. Because of year-to-year climatic variations, results from a single exposure test cannot be used to predict the absolute rate at which a material degrades in marine exposures. Several years of repeat exposures are needed to get an “average” test result for a given location.

It is strongly recommended that at least one control material be part of any marine exposure evaluation. It is preferable to use two control materials, one with relatively good durability and one with relatively poor durability.

This International Standard covers plastic materials in film, sheet, laminate, monofilament, fibre, rope or netting form. This includes, but is not limited to, packaging films, fishing gear, monofilament fibres and ropes.

When filaments, fibres, ropes or netting are exposed, it may be appropriate to apply a stress or use weights during exposure to give a more realistic estimate of performance in actual service.

When marine exposures are used to evaluate enhanced-degradability plastic material, a comparable material not formulated for enhanced degradability might be used for comparison. The test results can then be used to obtain the rate of breakdown of the enhanced-degradability material relative to the other material. For most enhanced-degradability materials, particularly the enhanced-photodegradability materials, the time to embrittlement (reduction of ultimate extensibility to < 2 %, and therefore failure of the material) is only a few months of exposure. Therefore, the test results will depend heavily on the time of year the exposure is conducted.

For materials that are intended to have enhanced degradability, it is important to evaluate the degraded material after exposure to determine whether it is biodegradable.

The test results pertain solely to the geographical location where the test was carried out. Marine weathering exposure test sites should be chosen on the basis of the geographical region in which the plastic products are intended to be used. Selecting a location with high levels of solar radiation and a high ambient temperature is recommended when high rates of breakdown are desirable in comparative studies of several different materials.

With plastic materials expected to undergo enhanced biodegradation (of any of their components), it might be important to select a test site where the incidence of microorganisms and biofouling species is relatively high all year round. This allows the exposure to be completed in a relatively short time.

5 Requirements for apparatus

5.1 General requirements

Unless otherwise specified, the test site selected shall be free from oil contamination, with no visible sheen of petroleum oil on the water surface, and free from any chemical influx from land-based sources of pollution. The exposure raft shall be placed at a location that has a depth of at least 1 m at low tide. Care shall be taken to ensure that no shadows from nearby structures or other obstructions fall on the exposure raft or specimen-mounting racks.

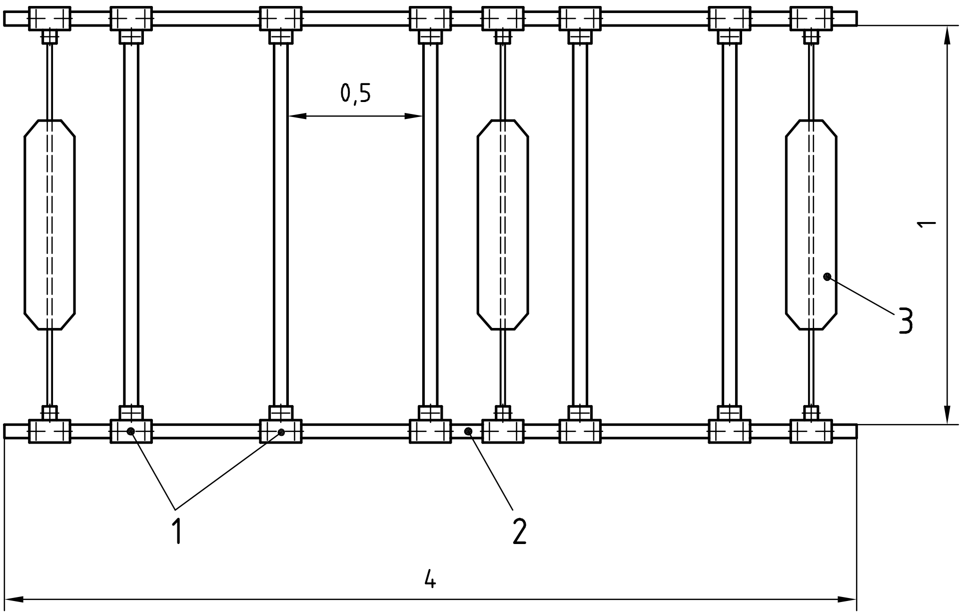
NOTE Contaminated sites may be used to assess the effect of specific contaminants or environments.

Materials used to construct the exposure raft and racks shall be resistant to corrosion and not interact with or contaminate the specimens being exposed. Unless otherwise specified, use nonmetallic fasteners to attach specimens to the exposure raft and racks. Use of plastic pipe components for exposure raft and rack construction is recommended.

5.2 Requirements for method A, floating exposure

The floating rack shall be constructed of heavy-duty plastic pipe material that is not susceptible to microbial attack, and with a sufficient number of floats to ensure that the rack will not sink. Securely anchor the floating rack and ensure that specimens are always in contact with the surface of the water, regardless of tidal movements. Position the floating rack in a location where the water depth is at least 1 m at low tide. Structural members of the rack shall not provide backing or support for materials exposed on it. Figure 1 is a diagram showing a typical floating rack, used for method A, that is made from 15 mm to 25 mm diameter plastic pipe.

Dimensions in metres



Key

1 T-fittings for plastic tubing [the use of non-corroding bolts (brass or stainless steel) in addition to glue is recommended for all joints]

2 15 mm to 25 mm diameter heavy-duty plastic tubing

3 foam float connected to frame using a plastic pipe that passes through the float (the use of three or more floats is recommended for a 4 m rack)

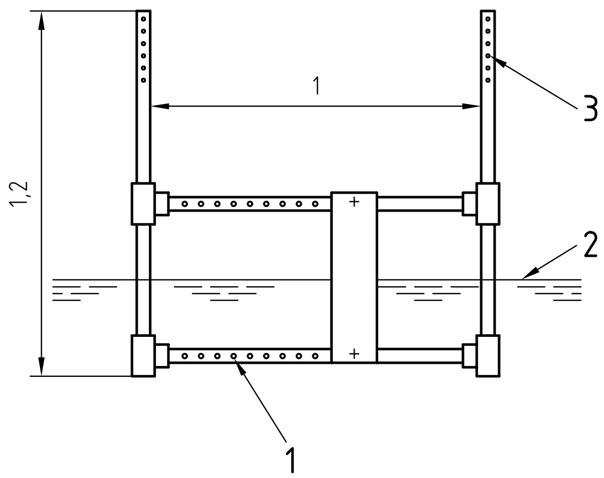
Figure 1 — Diagram of typical test rack used for floating exposures conducted in accordance with method A

5.3 Requirements for method B, partial-immersion exposure

Attach the test rack to a securely anchored floating raft to maintain the correct position in the water. Use a minimum amount of decking on the raft to ensure maximum exposure to sunlight of the surface of the test specimens. The exposure rack shall allow vertical installation of specimens and shall be positioned so that the prevailing tidal currents move parallel to the test specimen surface. Install racks to allow height adjustment to ensure that approximately one-half of each specimen is immersed and one-half is above the surface of the water. The maximum deviation from vertical of the lowest end of a test specimen shall be 20°. The minimum distance between adjacent rows of specimens shall be 30 cm, measured from the surface of the test specimens. The minimum distance between the edges of adjacent specimens shall be 15 mm. Figure 2 is a diagram of a typical rack used for method B. This rack would be attached to a floating raft at a distance of at least 0,5 m from the structural elements of the raft.

NOTE Placement of the exposure rack in accordance with methods B and C for flow of prevailing tidal current, and with the exposed face of the specimens oriented toward the equator, will ensure maximum exposure to sunlight.

Dimensions in metres



Key

1 15 mm to 30 mm diameter heavy-duty plastic tubing with series of regularly spaced holes for mounting specimens

2 surface of water

3 15 mm to 30 mm diameter heavy-duty plastic tubing with series of regularly spaced holes near top for attaching to floating raft and for adjusting specimens to appropriate immersion depth

Figure 2 — Diagram of typical rack used for partial-immersion exposures conducted in accordance with method B

5.4 Requirements for method C, shallow-immersion exposure

Attach the test rack to a securely anchored floating raft to maintain the correct position in the water. The exposure rack shall allow vertical installation of specimens and shall be positioned so that the prevailing tidal currents move parallel to the test specimen surface. If the exposure rack is constructed of metal, test specimens shall be electrically isolated from the rack. The exposure rack shall allow immersion of specimens to a depth of a least 0,3 m, but no more than 3 m. The minimum distance between the front face of a specimen to the back of the nearest specimen shall be 60 mm. The minimum distance between the edges of adjacent specimens shall be 15 mm. Figure 3 is a diagram of a typical rack used for method C. This example provides for two rows of test specimens.

6 Test specimens

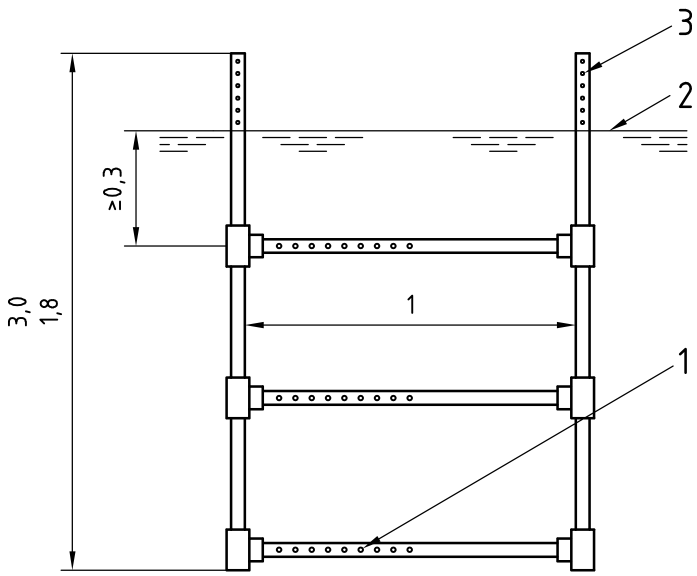
6.1 Form and preparation

The methods used for preparation of test specimens can have a significant impact on their apparent durability. Therefore, the method used for specimen preparation shall be agreed upon by the interested parties and should preferably be closely related to the method normally used to process the material in typical applications. A complete description of the method used for preparation of test specimens shall be included with the test report.

The dimensions of the test specimens are normally those specified in the appropriate test method for the property or properties to be measured after exposure. When the behaviour of a specific type of article is to be determined, the article itself shall be exposed whenever possible.

Label all specimens with an identifying code. It is recommended that this label be engraved or indented into the specimen. Writing on the specimen can be obliterated with exposure and therefore is not useful for specimen identification after exposure. If specimens are too small or too fragile for engraving or indenting, mark a separate tag and attach to the specimen in such a manner that it does not interact with the specimen or shield it from exposure.

Dimensions in metres



Key

1 15 mm to 30 mm diameter heavy-duty plastic tubing with series of regularly spaced holes for mounting specimens

2 surface of water

3 15 mm to 30 mm diameter heavy-duty plastic tubing with series of regularly spaced holes near top for attaching to floating raft and for adjusting specimens to appropriate immersion depth (there can be more rows for attaching specimens as long as immersion depth complies with requirements of method C)

Figure 3 — Diagram of typical rack used for shallow-immersion exposures conducted in accordance with method C

If the material to be tested is an extrusion- or moulding-grade polymer in the form of granules, chips, pellets or another raw state, specimens to be exposed shall be cut from a sheet produced by the appropriate method. The exact shape and dimensions of the specimens will be determined by the specific test procedure used for measurement of the property of interest. The procedures used to machine or cut individual test specimens from a larger sheet or part may affect the results of property measurements and hence the apparent durability of the specimens. For preparation of test specimens, procedures described in ISO 293, ISO 294-1, ISO 294-2, ISO 294-3, ISO 295 and ISO 3167 have been found to be satisfactory.

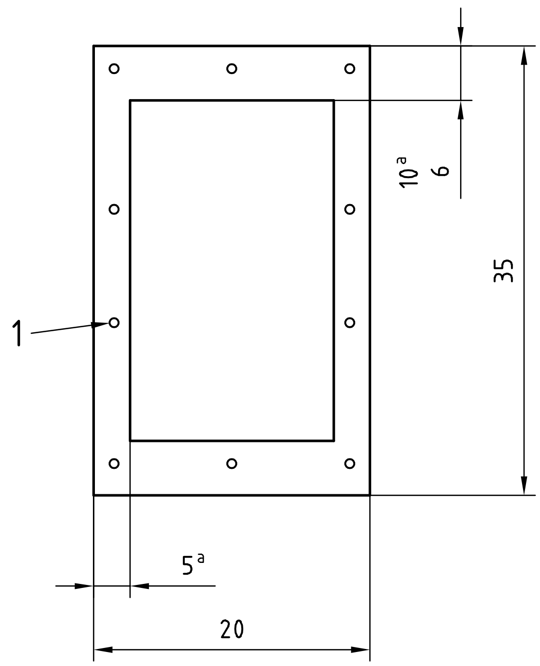
Unless otherwise specified, individual test specimens used for property measurements shall not be cut from larger pieces that have been exposed. However, in the case of plastic film specimens intended for the measurement of tensile properties, the test specimens (strips or dumbbells) shall be cut after exposure of the sample. Fouling on the edges of pre-cut specimens during marine exposure may introduce an error in the ultimate extension values measured using such specimens. However, the effects of any cutting or machining operation on the properties of individual test specimens may be much larger when the specimens are cut from a large piece after exposure. This is especially true for materials that embrittle on exposure. Follow the procedures described in ISO 2818 for preparation of test specimens by machining.

When test specimens are cut from an exposed sheet or larger part, they shall be taken from an area that is at least 20 mm from the fixture holding the material or from the exposed specimen edges. In no circumstances shall any material be removed from the exposed face during test specimen preparation.

When comparing materials in an exposure test, use test specimens that are similar in dimensions and exposed area.

When exposing thin or fragile specimens using method B or method C, clamp the specimens between the two halves of a frame and attach the frame to the test rack. Figure 4 is a diagram for a typical specimen-mounting frame. Use non-corroding materials to construct the frame and non-corroding fasteners to clamp the two frame halves together. Select materials for frame and fasteners that will not interact with the test specimens. Typically the thickness of materials used for the frame halves is 3 mm to 5 mm.

Dimensions in centimetres



Key

1 regularly spaced holes for bolting two frame halves together to lock test specimens in place (use non-corroding fasteners made of e.g. brass to bolt the frame halves together)

[a] Typical values.

Figure 4 — Typical frame section used to clamp fragile test specimens for attachment to racks used for method B and method C exposures

6.2 Number of test specimens

The number of test specimens for each set of test conditions or exposure period shall be that specified in the appropriate test method for the property or properties to be measured after exposure.

NOTE For the determination of mechanical properties, it is recommended that the number of exposed test specimens be twice that required by the relevant International Standard (due to the large standard deviation known to occur in measuring the mechanical properties of “weathered” materials).

If the test method used for property measurement does not specify the number of test specimens to be exposed, it is recommended that a minimum of three replicate specimens of each material be prepared for each exposure stage.

When destructive tests are used to determine the properties being measured, the total number of test specimens required will be determined by the number of exposure periods used and whether unexposed file specimens are tested at the same time as exposed specimens.

Control materials that are similar in composition and construction and which have known durability should preferably be included with each exposure test. It is recommended that control materials known to have relatively poor and relatively good durability be used. Control materials are used for the purpose of comparing the performance of the test materials with that of the controls. Before laboratory-to-laboratory comparisons are made, it is necessary for the control materials to be agreed upon by all interested parties. The number of specimens of the control material should be the same as that used for test materials.

Retain a supply of unexposed file specimens of all materials evaluated.

When destructive tests are run, ensure that sufficient file specimens are retained so that the property of interest can be determined on unexposed file specimens each time exposed materials are evaluated.

6.3 Storage and conditioning

If test and/or control specimens are cut or machined from larger pieces, they shall be conditioned, after preparation, in accordance with ISO 291. In some circumstances, it may be necessary to precondition the sheets prior to cutting or machining to facilitate specimen preparation.

When using tests to characterize the mechanical properties of the materials being exposed, specimens shall be appropriately conditioned before all property measurements. Use the conditions described in ISO 291. The properties of some plastics are very sensitive to moisture content, and the duration of conditioning may need to be longer than that specified in ISO 291, particularly where specimens have been exposed to climatic extremes.

File specimens shall be stored in the dark under normal laboratory conditions, preferably in one of the standard atmospheres given in ISO 291.

Some materials will change colour during storage in the dark, particularly after weathering. It is essential that colour measurement or visual comparisons be carried out as soon as possible after exposure once the exposed surface has dried.

7 Procedure

7.1 General

The conditions and procedure for the exposure depend upon the particular method selected. Refer directly to the specific instructions for method A, B or C, as appropriate.

Attach the specimens to the exposure rack in accordance with the specific procedure for the method used. Record on a diagram the location of each test specimen. Record the date the exposure started.

Depending on the material being evaluated, the length of the intervals at which specimens are removed from exposure and tested for degradation may be specified in terms of days, weeks, months or years. In the case of exposures that last for less than a full year, it is particularly important to indicate the date on which the exposure was initiated. In exposures lasting several years, the seasonal effects are averaged out; however, the test results in the case of exposures of shorter durations can depend on the season in which the exposure was carried out.

For method A and method B, solar radiation is a primary factor responsible for degradation of the material. If required, measure total solar, or solar ultraviolet, radiation in accordance with the procedures described in ISO 877, and report the solar radiant exposure, in joules per square metre, and the passband over which the radiation was recorded. When solar radiant exposure is reported, the orientation of the radiometer used for measurement shall also be reported. Unless specifically required, do not estimate solar radiation using changes in materials such as fabrics or polymers.

7.2 Specific procedure for method A, marine floating exposure

For lightweight specimens such as plastic netting, films or sheets, wrap one end of the specimen once over a structural member of the raft and secure it in place with one or more UV-resistant plastic cable ties. Attach the specimens to the raft member on one edge so that they are able to float freely in the water. With heavier specimens, use UV-resistant plastic cable ties to attach one or a set of free-floating specimens to the exposure raft. Except as noted above, structural members of the raft itself shall in no case provide a backing for the samples exposed on the raft.

For specimens likely to fragment during the exposure (for instance hydrophilic foam materials), enclose each specimen in a bag made of thermoplastic netting. The bag shall be large enough to contain the fully swollen specimen without restricting it in any manner. The specimen shall be able to move freely within the bag. The length and width of the bag shall be 30 % larger than that of the specimen swollen with sea water. The mesh size of the netting used shall be at least 20 mm. When specimens are placed in net bags, attach the bag to the raft and place the identifying code on the bag rather than on the specimen itself. Periodically examine the bag for fouling of the netting, which may restrict the free flow of sea water through the bag. If the bags themselves are fouled prior to completion of the exposure, replace them or clean them carefully, taking care not to damage the specimens.

Inspect the floating raft at least weekly and clear any debris trapped within the raft, taking care to ensure that the specimens are not cleaned or disturbed. Do not allow the specimens to dry out for any reason.

When specimens are removed from the floating raft at the designated test times, carefully wrap them in paper or plastic and store away from light until they are tested. Where analysis or enumeration of fouling organisms is anticipated after the specimens have completed the exposure, place the specimens in a large container with fresh water of the same type used for exposure. If it is not practical to place specimens in a container of water after removal from exposure, wrap them in several layers of paper towels wetted with the same water used for exposure. If the colony enumeration cannot be carried out within 24 h after removal of the specimens from exposure, place the wrapped specimens in a plastic bag and store in a refrigerator.

7.3 Specific procedure for method B, partial-immersion exposure

Attach specimens to the exposure rack with at least 15 mm between each specimen. All fasteners used to attach specimens to the rack shall be nonconductive. Cable bundle straps have proven to be very useful.

For fragile materials such as thin films, clamp specimens between two frames such as described in 6.1 and attach the frame to the test rack.

Install the exposure rack with specimens attached to the floating rack so that a minimum of 40 % of each specimen is above the surface of the water.

7.4 Specific procedure for method C, shallow-immersion exposure

Attach the specimens to the exposure rack with at least 15 mm between each specimen. Attach the specimens to one side of each exposure rack only. All fasteners used to attach the specimens to the rack shall be nonconductive. Cable bundle straps have proven to be very useful.

For fragile materials such as thin films, clamp the specimens between two frames such as described in 6.1 and attach the frame to the exposure rack.

Install the exposure rack with specimens attached so that all specimens are immersed at least 0,3 m, but no more than 3 m, below the surface of the water.

7.5 Evaluation of specimens after exposure

Note the appearance and/or photograph the exposed specimens to indicate the extent of fouling on the sample surface upon removal from exposure or from storage.

If any specimens have fragmented during the exposure, particularly those exposed in bags in accordance with method A, small pieces of the specimen can be used for analyses.

Measure the relevant physical, mechanical and chemical properties of the exposed materials to assess the extent of degradation. Typical properties used for the purpose include measurement of average molecular mass, measurement of tensile properties, infrared spectroscopy and measurement of specific gravity of fouled specimens to assess the degree of fouling.

If specified, determine quantitatively the fouling colony composition on the specimen surface in accordance with relevant standards.

Follow the procedures described in ISO 4582 for measuring properties on specimens before and after exposure and for expressing the change in properties on exposure. Refer to the relevant International Standard for the specific procedure to follow for measuring particular specimen properties.

If non-destructive testing is used to measure a property of the material being assessed, the property shall be measured before beginning the exposure. The same property is then measured after each exposure period. Care shall be taken to make the property measurement after each exposure period at the same position on the test specimen.

NOTE To monitor the response of the instrument used to measure the desired property, one can test a reference or calibration specimen each time the instrument is used.

If destructive testing is used to measure a property of the material being assessed, separate sets of specimens will be needed for each exposure period. The property is measured on each set of exposed specimens. The value of the property after exposure may be compared to the value obtained prior to exposure. Alternatively, the property can be measured on a separate set of file specimens at the same time as the property is measured on the exposed specimens. The results for the file specimens and from the exposed specimens can then be compared.

8 Test report

The test report shall contain the following information:

a) a complete description of the test and control specimens, including composition and method of preparation with reference to relevant standards;

b) the location of the exposure site;

c) the type of water at the exposure site (salt water, brackish water or fresh water);

d) if required, the salinity of the water;

e) if required, the pH of the water;

f) the exposure method used;

g) the date the exposure was started and the date the exposure was completed;

h) if required, the solar radiant exposure, including the wavelengths over which the radiant energy was measured;

i) the average temperature of the water at the exposure site;

j) for exposures of 60 days or less, the average daily water temperature;

k) for exposures of more than 60 days, the average daily water temperature for each week during which the exposure took place;

l) if required, the daily maximum and minimum ambient-air temperatures;

m) if required, the total rainfall during the exposure period;

n) the results of property measurements made on all specimens, including a reference to relevant standards or a complete description of the method used for property determination;

o) a description of the general appearance, especially of fouling, of all the specimens (the use of sketches, photographs or videotapes is recommended to document fouling, damage or fragmentation);

p) a rating of any biofouling, determined in accordance with the relevant International Standards;

q) a description of any holes that may have been caused by attack by marine microorganisms and of the location of such holes (it is important that these areas be avoided when specimens are prepared for tensile-property measurements);

r) if required, the specific gravity of fouled samples to ascertain if they are denser than sea water, i.e. negatively buoyant under free-floating conditions.

Bibliography

[1] PEGRAM, J.E., ANDRADY, A.A., Outdoor Weathering of Selected Polymeric Materials under Marine Exposure Conditions, *Polymer Degradation and Stability*, Vol. 26 (1989), pp. 333-345

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