Designation: ~~D7473 – 12~~ D7473/D7473M – 21

Standard Test Method for Weight Attrition of Non-floating Plastic Materials ~~in the Marine Environment~~ by Open System Aquarium Incubations[1]

This standard is issued under the fixed designation ~~D7473~~;D7473/D7473M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. ~~Scope~~ Scope*

1.1 This test method is used to determine the weight loss as a function of time of non-floating plastic materials (including formulation additives), when incubated under changing, open, marine aquarium conditions, which is representative of aquatic aerobic environments near the coasts and near the bottom of a body of water in the absence of sunlight, particularly UV and visible portions of the spectrum. The goal of this test is to obtain data that ~~will predict real world experiences based on the extent and rate of biodegradation data of the same materials obtained from the laboratory Test Method~~ can be used ~~D6691. The aquarium incubated films are examined for visual degradation and dry weight loss over time. This test is not a replacement to Test Method~~to assess the ~~D6691, but rather an additional ASTM method for weight attrition. The standard addresses weight loss of the plastics in a marine environment and cannot be used for demonstrating biodegradation for which Specification~~potential for physical degradation of the test material. Such potential for physical degradation will be affected ~~D7081 needs to be used.~~by real life environmental conditions.

1.2 In particular this test method does not take into consideration the possible effects of solar irradiation.

1.3 The aquarium-incubated plastic materials are examined for visual degradation and dry weight loss over time. This test does not provide information on ultimate biodegradation (that is, it is not a replacement for Test Method D6691), but it is an ASTM method for weight attrition. The standard addresses only weight loss as a function of time of the plastics materials in a marine environment and shall not be used for demonstrating ultimate biodegradation.

1.4 This test method does not provide information regarding the potential formation of microplastics due to the physical degradation of the samples.

1.5 Plastic ~~film~~ pieces of known size and thickness are used at levels so as not to exceed the availability of micronutrients essential for and therefore limit the microbial ~~biodegradation process.~~processes.

1.6 The aquarium incubation test method allows for representative indigenous microorganisms present in seawater and marine sediment to be enriched for and carry out the biodegradation. It is recommended that the test be carried out in the geographical vicinity (latitudinal area) where the test ~~film is~~materials are likely to be ~~used and potentially disposed of in the marine environment if biodegradable criteria are met.~~ used. These Aquarium studies are conducted in indoor environments, hence any sunlight-induced effects on degradation, or biodegradation, or both, are not taken into account.

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.96 on Environmentally Degradable Plastics and Biobased Products.

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***A Summary of Changes section appears at the end of this standard**

~~1.4 Prior to conducting this aquarium test method (weight loss data) for the verification of biodegradability, Test Method D6691 shall be run on the same materials to establish quantitative levels of the plastic organic carbon oxidation and levels of carbon dioxide recovered there from. If Test Method D6691 achieves 30 % mineralization, then apply this Aquarium test and perform it. If the results from Test Method D6691 do not achieve 30 % mineralization, then aquarium incubation testing need not be done and the material shall be considered non-biodegradable in the marine environment.~~

1.7 This test by itself shall not be used as the basis for claims, such as "Biodegradable in Marine Environments" since it is only a weight loss test method. This test method is solely a means for measuring a characteristic (physical degradation) under standard conditions. It does not assess the general environmental impact of plastic products.

1.8 *Units*—The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system are not necessarily exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems has the potential to result in non-conformance with the standard.

1.9 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate ~~safety~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—There is no known ISO equivalent to this standard.

1.10 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D883 Terminology Relating to Plastics

D6691 Test Method for Determining Aerobic Biodegradation of Plastic Materials in the Marine Environment by a Defined Microbial Consortium or Natural Sea Water Inoculum

~~D7081 Specification for Non-Floating Biodegradable Plastics in the Marine Environment (Withdrawn 2014)[3]~~

3. Terminology

3.1 *Definitions*—Definitions of terms applying to this test method appear in Terminology D883.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *absence of light*—absence of electromagnetic radiation with the focus on visible and ultraviolet portions of the spectrum of sunlight or other light with similar wavelength frequencies from artificial sources.

3.2.2 *natural seawater (NSW)*—seawater unamended with any additives.

3.2.3 *indigenous microbes*—those microbes naturally occurring in a seawater or sediment sample.

3.2.4 *mesophilic*—temperature range from approximately 20 to 40°C over which microorganisms adapted to moderate conditions maintain active metabolic rates.

3.2.5 *psychrophilic*—temperature range from approximately 2 to 20°C over which microorganisms adapted to cold conditions maintain active metabolic rates.

3.2.6 *sulfate reduction*—the anaerobic microbial process whereby sulfate acting as an electron acceptor is converted to hydrogen sulfide as an end product.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.2.7 *surface marine ~~sediment~~ sediment (SED)*—the upper few ~~millimetres~~millimeters to several ~~centimetres~~centimeters of oceanic bottom sediments containing the natural indigenous microbial populations and ranging from oxic to potentially anoxic conditions with increasing sediment depth.

4. Summary of Test Method

4.1 This test method consists of the following:

4.1.1 Selecting, characterizing and preparing plastic ~~films~~materials for testing (formulation, carbon content, molecular weight, ~~film~~ thickness and uniformity).

4.1.2 Running ~~short term~~ short-term (4 days) sterile seawater controls of the ~~films~~ plastic materials to determine level of loss due to soluble components (plasticizers ~~etc).~~etc.). See Section 11.

4.1.3 ~~Collecting~~Collection and storage in the absence of light of marine sediment from the local coastal marine environment for use in aquarium incubations.

4.1.4 Having access to a continuous flow of natural seawater.

4.1.5 Exposing ~~film~~ plastic material pieces in the absence of light to natural flowing seawater or sediment surfaces under natural flowing seawater in open tray incubations in a marine aquarium at seasonally varying water temperatures. See Section 8.

4.1.6 Harvesting ~~film~~ plastic material pieces at varied time intervals to assess visual impacts of exposure and degradation and determining the percentage loss in dry weight and weight loss per unit area.

~~4.1.7 The film material is related for its attrition and weight loss in this realistic open system aquarium incubation, to the prior determination of its organic carbon biodegradability to CO_2 based on the outcome of Test Method D6691 testing of the same film.~~

4.2 ~~Conventional plastics are not allowed to be disposed of at sea, and yet the use of such materials aboard ships has increased in recent years. A technological goal is to develop a test method for plastics, designed to biodegrade safely in the marine environment (conversion to carbon dioxide by means of microbial metabolism). These can be used in place of conventional plastics which will fulfill the criteria for allowing them to be disposed of in the marine environment.~~ This aquarium incubation test method has been developed and is used to assess the rate and extent of attrition of ~~biodegradable plastics~~plastic materials as a loss in dry weight during incubation exposure to indigenous marine microorganisms. The test assesses weight loss under continuous flow (open system) aquarium conditions in which microbial growth processes rely on the naturally occurring supply of nutrients (for example, nitrogen and phosphate) in the incoming seawater and use the plastic as the carbon source. Aquarium testing is more realistic of the actual marine environment than a closed flask laboratory test (that is, Test Method D6691) as it allows flushing, exposure to a diverse population of microbes, removal of metabolic end products, re-supply of oxygen, exposure to anoxic conditions in sediment, and exposure to seasonal temperature variation of the incoming seawater and natural concentration of macro- and micronutrients. ~~The~~ This dynamic test is carried out as close to the geographical vicinity (latitudinal area) where the ~~test film~~tested material is likely to be used in product form.

4.3 The test does not quantify the conversion of plastic organic carbon to carbon dioxide, but rather the loss in dry weight of the material over time. Therefore, other test methods, such as Test Method D6691, must be run ~~prior to this test~~ in order to determine the maximum CO_2 production from the test ~~film~~sample and therefore indicate the degree of biodegradation under the more optimum conditions of the ~~laboratory but which are less realistic of the actual marine environment.~~laboratory.

~~4.4 Conducting Test Method D6691 initially as a closed system test in the laboratory will determine if the plastic items meet criteria of acceptable biodegradability to the pass level for mineralization specified in 1.4 and if so, the open system aquarium test is warranted. The rate of biodegradation can be expected to be faster under laboratory conditions compared to the Aquarium test since the latter is conducted under changing and often colder temperatures and a more limited supply of nutrients relative to the available carbon.~~

5. Apparatus

5.1 *Borosilicate ~~Glass Beakers,~~glass beakers,* varied sizes, (250 mL to 4 L as needed for sediment).

5.2 *Autoclave* capable of steam sterilizing. The autoclave is run at 121°C for 20 min.

5.3 *Drying oven* for obtaining constant dry weight of samples

5.4 *Analytical balance, ~~(±0.1 mg)~~* for weighing test samples

5.5 *Access to flowing natural seawater aquarium.*

5.6 *Plastic boxes* (lids removed) with open compartments for holding samples incubated in open aquarium trays of flowing seawater.

5.7 *Nylon mesh screening,* (⅛ to ¼ in. ~~openings)~~openings).

5.8 *Opaque ~~plastic~~ or transparent plastic materials in the form of film or fabric.*

6. Hazards

6.1 While there are no known specific hazards associated with this test procedure, care must be taken in handling of all samples. Latex gloves are used when handling the marine sediment.

6.2 Before preparing chemical stock solutions read the manufacturer's ~~Material~~ Safety Data Sheets.

7. Inoculum

7.1 *Natural Seawater (NSW),* as a continuous fresh supply avoiding collections sites influenced by storm water runoff or ~~have~~having major oil slicks on the surface. For the purposes of this standard, a major oil slick is the one that can be clearly noticed by visual inspection.

7.2 *Surface Marine Sediment (SED),* collected on or before (1 day) the day the Aquarium incubations are to be initiated. Surface sediment, preferably of a muddy nature as opposed to sand, can be collected from any coastal location at or close to the NSW source site.

8. Procedure (Open System Aquarium Incubation)

8.1 *~~Film~~ Plastic Materials Preparation:*

8.1.1 Determine the average mil thickness of the ~~film.~~ plastic material. Cut pre-dried ~~film~~material in 0.5 by 0.5 in. pieces. Weigh individual pieces of ~~film~~ the material and record weight.

8.1.2 Samples shall not be subjected to any conditions or treatments designed to accelerate weight loss prior to 8.3.2.

8.1.3 The use of a benchmark material (for example, a material whose potential for physical degradation is known) to be tested in parallel with the tested material is highly recommended.

8.2 *Aquarium Inoculation Preparation:*

8.2.1 Collect surface SED from area of the NSW site and bring to aquarium incubation site. Keep sediment in place with absence of light prior to incubation and testing. Collect enough to half fill the needed number of sections in chosen incubation containers (for example, plastic boxes with 12 separate sections and lids removed—these boxes will be placed in the flowing seawater aquarium trays). Sections of about 4 in. by 2 in. are sufficient for each sample piece. The same types of incubation containers and