Designation: D8402 – 23

Standard Practice for Development of Microplastic Reference Samples for Calibration and Proficiency Evaluation in All Types of Water Matrices with High to Low Levels of Suspended Solids[1]

This standard is issued under the fixed designation D8402; 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

1.1 This practice describes manufacturing methods to create microplastic particles from pellets of common polymers and the preparation of microplastic reference samples for calibration and proficiency evaluation of microplastic collection practices, preparation practices, and identification methods.

1.2 This practice does not describe methods for controlling or characterizing the shapes of particles. The procedures have been observed to yield irregularly shaped particles, the use of which in many cases will serve to remove the analytical bias inherent with using distinctive manufactured spherical beads. Other procedures should be used if spheres or elongated fibers are desired.

1.3 This practice does not describe handling procedures for waste generated when executing the procedures described herein. It is the responsibility of the user of this practice to follow applicable laws and regulations when manufacturing and disposing of microplastic particles, and to establish appropriate procedures to minimize the amount of waste generated.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *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

D1193 Specification for Reagent Water

D1921 Test Methods for Particle Size (Sieve Analysis) of Plastic Materials

D5905 Practice for the Preparation of Substitute Wastewater

D8333 Practice for Preparation of Water Samples with High, Medium, or Low Suspended Solids for Identification and Quantification of Microplastic Particles and Fibers Using Raman Spectroscopy, IR Spectroscopy, or Pyrolysis-GC/MS

2.2 *ISO Standards:*[3]

ISO 8573-1:2010 Compressed air — Part 1: Contaminants and purity classes

3. Terminology

3.1 For definitions of terms used in this practice, refer to Terminology D883.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *glass transition temperature, n*—a temperature above which an amorphous material transitions from a relatively brittle material to a viscous, supercooled liquid.

3.2.2 *microplastic, n*—any solid, synthetic organic polymeric material to which chemical additives or other substances may have been added, which are particles <5 mm in their largest dimension, and fibers no longer than 15 mm in length, with an aspect ratio of at least 30:1 and <500 µm in its smallest dimension.

3.2.3 *suspended solids, n*—includes all matter that is removed by a 0.45 µm pore size filter.

4. Summary of Practice

4.1 Polymer pellets are processed in a laboratory grinder that utilizes a spinning blade to fragment the pellets into

[1] This practice is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.06 on Methods for Analysis for Organic Substances in Water.

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[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] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

powders containing particles of various sizes. This practice does not describe methods of separation of the mixed-size powder into fractions based on size ranges. Test Methods D1921 describes methods for dry sieve separation of polymers that could be used to separate the powders described in this practice. If particle fractions under approximately 20 µm in diameter are desired, suspension of the particles in liquid followed by filtration or sedimentation methods may be more effective than dry sieve separation.

4.2 Polymers with a glass transition temperature (T_g) below room temperature require a different grinding procedure than polymers with T_g above room temperature. Polymers with high T_g tend to undergo brittle fracture during grinding, while polymers with low T_g tend to be stretched into very elongated structures, and special steps must be taken to grind them effectively.

4.3 Two grinding procedures are described:

4.3.1 *Procedure A*—This method uses a blunt-edged blade to fragment brittle polymers with a T_g above room temperature, such as polystyrene (PS), polyvinyl chloride (PVC), and polyethylene terephthalate (PET).

4.3.2 *Procedure B*—This method uses liquid nitrogen to embrittle polymers with T_g below room temperature, such as polyethylene (LDPE, HDPE) and polypropylene (PP). Cryo-milling or cryo-crushing is the process of cooling or chilling a material and then reducing it into a smaller particle size. While the use of a specialized cryo-mill may be considered to ensure production of a wide size distribution of particles in this procedure, the cooled polymers are coarsely fragmented with a blunt-edged blade and then fragmented into smaller particles with a sharp cutting blade.

4.4 Microplastic reference sample preparation includes two approaches, *(1)* count-based reference samples for % recovery and *(2)* mass-based proficiency samples, which possess some inherent variability between samples.

4.4.1 *Count-based Microplastics Reference*—40 mL vials of DI H_2O containing 20 to 40 microplastic particles are shipped at room temperature to recipient laboratories to spike their own water matrix samples. Because a lab's water samples may already contain microplastics, the reference microplastics for each vial should be documented with saved microscopy images, so their morphologies can be checked against the particles identified by the recipient lab. A 100 % transfer efficiency of particles to vials should be demonstrated. This method is best suited to low numbers of reference samples due to the effort of documenting and transferring individual particles. As such, this approach is most applicable to generating reference samples for one's own laboratory or partner laboratories. In addition, due to the relatively low numbers of microplastics per sample, it will yield the most environmentally realistic concentrations when spiking already collected samples such as filters or subsamples for analysis, rather than the original water volumes, which can be orders of magnitude larger.

4.4.2 *Mass-based (Gravimetric) Microplastic Standards*—Gravimetric reference samples are prepared in large batches of simulated water matrices. Bulk quantities of reference microplastics are measured in terms of mass on a balance and added to a simulated water matrix substitute, for example, Practice D5905. Measurable amounts of microplastic (>1 mg) are diluted into several liters of simulated water matrices, then split into several 1 L reference samples. This approach is typically used to prepare reference or proficiency samples for other common environmental analytes such as organic solvents or asbestos. Even with the homogenization step described in 12.6, uniform microplastic counts may be more difficult to achieve in split samples compared to other analytes, so assessments of the standard deviations of both the microplastic mass/sample within the batch, as well as the equivalent number of particles per sample, are critical for quality control. Depending on the standard deviation, this approach may only be suitable for order of magnitude assessments of accuracy. In addition, the completed reference samples should be analyzed by one or more reference laboratories to determine the true mean value of the reference samples. Alternatively, the mean count may be determined from all participant labs. Despite the inherent variability, this approach is capable of generating many more reference microplastics per batch. As such, it may be a desirable when creating samples for large numbers of laboratories, or if environmentally realistic microplastic concentrations are required in very large water volumes. If shipping 1 L vessels of non-DI water, the procedure must include packing safely with ice and providing any needed shipping documentation. This approach will also yield a more direct comparison to the results of mass-based methods such as Pyr-GC/MS.

5. Significance and Use

5.1 These procedures can be used to generate microplastic particles as a simulation of microplastic particles found in the natural environment. Suitable uses may include evaluation of microplastic detection and imaging methods. Use of reference samples will support estimation of ambient and flux concentrations in drinking water, wastewater and natural environments, investigations of microplastic particle degradation, and ingestion of microplastics by animals in the contexts of food safety and human health risk assessment.

6. Apparatus

6.1 *Glass Sample Jars*—Clean glass jars to store polymer powder. If polymer lids are used, lid composition should be recorded in case potential contamination is discovered; lids with polymer foam liners should be avoided.

6.2 *Timer*—A timer with a resolution of 1 s or less to measure time intervals during grinding.

6.3 *Laboratory Grinder*—A spinning blade grinder with interchangeable blades and a maximum speed of 20 000 rpm to 30 000 rpm and output power of approximately 100 W.[4] The blade height within the grinding chamber should be adjustable during grinding. Coffee grinders or other consumer-grade

[4] Cooling times described in this standard were established by tests performed with a specific make and model of grinder – thus, cooling times as stated may vary with other devices.