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Standard Guide for Techniques to Separate and Identify Contaminants in Recycled Plastics[1]

This standard is issued under the fixed designation D5577; 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 guide is intended to provide information on available methods for the separation and classification of contaminants such as moisture, incompatible polymers, metals, adhesives, glass, paper, wood, chemicals, and original-product residues in recycled plastic flakes or pellets. Although no specific methods for identification or characterization of foam products are included, foam products are not excluded from this guide. The methods presented apply to post-consumer plastics.

1.2 For specific procedures existing as ASTM test methods, this guide only lists the appropriate reference. Where no current ASTM standard exists, however, this guide gives procedures for the separation or identification, or both, of specific contaminants. Appendix X1 lists the tests and the specific contaminant addressed by each procedure.

1.3 This guide does not include procedures to quantify the contaminants unless this information is available in referenced ASTM standards.

1.4 *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.*

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

1.5 *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]

D789 Test Method for Determination of Relative Viscosity of Concentrated Polyamide (PA) Solutions

D792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement

D883 Terminology Relating to Plastics

D1003 Test Method for Haze and Luminous Transmittance of Transparent Plastics

D1193 Specification for Reagent Water

D1238 Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer

D1457 Specification for Polytetrafluoroethylene (PTFE) Molding and Extrusion Materials (Withdrawn 1996)[3]

D1505 Test Method for Density of Plastics by the Density-Gradient Technique

D1898 Practice for Sampling of Plastics (Withdrawn 1998)[3]

D1925 Test Method for Yellowness Index of Plastics (Withdrawn 1995)[3]

D3418 Test Method for Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry

D4019 Test Method for Moisture in Plastics by Coulometric Regeneration of Phosphorus Pentoxide (Withdrawn 2002)[3]

D5033 Guide for Development of ASTM Standards Relating to Recycling and Use of Recycled Plastics (Withdrawn 2007)[3]

D5227 Test Method for Measurement of Hexane Extractable Content of Polyolefins

E169 Practices for General Techniques of Ultraviolet-Visible Quantitative Analysis

E355 Practice for Gas Chromatography Terms and Relationships

[1] This guide is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.95 on Recycled Plastics.

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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] The last approved version of this historical standard is referenced on www.astm.org.

E682 Practice for Liquid Chromatography Terms and Relationships
E794 Test Method for Melting And Crystallization Temperatures By Thermal Analysis
E1252 Practice for General Techniques for Obtaining Infrared Spectra for Qualitative Analysis
2.2 *ISO Standards:*[4]
ISO 3451/1-1981 Plastics—Determination of Ash; Part 1: General Methods
ISO 1183-1987 Methods for Determining the Density and Relative Density of Noncellular Plastics

3. Terminology

3.1 This terminology used in this guide is in accordance with Terminology D883 and Guide D5033.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *chemicals*—nonhazardous or hazardous materials (for example, insecticides or herbicides) potentially used in contact with plastic materials.

3.2.2 *glue*—adhesives used for labels or joining bottle parts (for example, ethylene-vinyl acetate).

3.2.3 *heavy metals*—metals heavier than sodium on the periodic table (for example, lead, arsenic, cadmium, chromium, or copper).

3.2.4 *heavy plastic*—unfilled polymers such as polystyrene, poly(ethylene terephthalate), and poly(vinyl chloride) and filled materials with densities greater than 1.00 g/cm^3.

3.2.5 *light plastic*—polymers such as polyethylene and polypropylene with densities less than 1.00 g/cm^3.

3.2.6 *original-product residues*—residues from any original-product contents of a plastic package (for example, milk, juice, or detergent).

3.2.7 *particles*—piece of metal, glass, wood, paper, or other discreetly shaped material equal to or larger than 0.1 mm^2.

3.2.8 *specks*—any material equal to or less than 0.1 mm^2.

4. Summary of Guide

4.1 This guide provides details of several procedures used to separate and classify contaminants including, but not limited to, moisture, original product residues, incompatible plastic, metal, paper, glass, adhesives, and wood in recycled plastic flakes or pellets. This guide lists existing ASTM and ISO methods that can be used to characterize solid and some liquid contaminants. In addition, this guide presents details of some industry procedures for identification of contaminants. Appendix X1 provides information on quantitative aspects of some of these industry standards that can also be used to estimate the concentration of various contaminants.

5. Significance and Use

5.1 Recycled plastic materials may contain incompatible plastic or other undesirable contaminants that could affect the processing or quality, or both, of the plastic prepared for reuse. Techniques to separate and identify incompatible plastics, moisture, chemicals, or original product residues, and solid contaminants such as metals, paper, glass, and wood are essential to the processing of recycled plastic materials.

5.2 This guide lists existing ASTM and ISO methods plus currently practiced industrial techniques for identification and classification of contaminants in recycled plastics flake or pellets.

6. Sampling

6.1 Unless otherwise stated, materials should be sampled in accordance with the procedures described in Practice D1898. Adequate statistical sampling should be considered as an acceptable alternative.

7. Existing ASTM or ISO Procedures

7.1 *Moisture:*

7.1.1 A coulometric method (Test Method D4019), the standard test method for haze (Test Method D1003), Karl Fisher titration (Test Method D789), or a gravimetric procedure (13.6.1 of Specification D1457) can be used to estimate the moisture content of recycled plastic materials.

7.2 *Visual Inspection and Product Uniformity:*

7.2.1 *Color:*

7.2.1.1 Test Method D1925 measures the yellowness index of clear acrylic plastics and the haze and the luminous transmittance procedure (Test Method D1003) characterizes the color of transparent unpigmented recycled plastic materials. These tests are not readily applied to pigmented plastic samples.

NOTE 2—Test Method D1925 is currently being revised by ASTM Subcommittee D20.40 to address reproducibility and bias problems.

7.2.2 *Melt Flow for Product Uniformity*—Uniformity of some recycled plastic flakes or pellets can be estimated by measuring the flow rate of the material using an extrusion plastometer (Test Method D1238).

7.3 *Density or Specific Gravity*—The displacement method for specific gravity or relative density (Test Method D792) or the density-gradient procedure for density (Test Method D1505) are useful techniques to determine contamination of recycled plastic flakes or pellet samples with one or more other polymers.

NOTE 3—Test Method D1505 uses relatively small test specimens, so it may not be applicable for analysis of nonhomogeneous recycled plastic materials.

7.4 *Inorganic Contaminants:*

7.4.1 An ash test, such as ISO 3451/1, or the muffle-furnace techniques currently being evaluated within ASTM Subcommittee D20.70 (project designation X70-8702) can be used to estimate the inorganic filler content of recycled plastic flake or pellets.

NOTE 4—Some volatile metals may be lost using the test indicated in 7.4.1. ASTM Subcommittee D20.70 is currently developing a test method (project X70-9201) for metals, including heavy metals, that will include sample-preparation techniques to minimize the loss of volatile metals prior to analysis by X-ray fluorescence or spectroscopic techniques.

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

7.4.2 Ferrous (iron) contaminants can be removed with a magnet and aluminum contaminants are separated from plastic materials using density procedures in accordance with 8.3.

7.5 *Thermal Analysis:*

7.5.1 Since most polymers exhibit unique temperatures for melting or other phase transitions, measurement of these transition temperatures (Test Method D3418) or the melting and crystallization temperatures (Test Method E794) of a sample may provide useful information regarding the identity of polymeric components present in a recycled plastic material.

7.5.2 Both Test Methods D3418 and E794 involve thermal gravimetric analysis (TGA) or differential scanning calorimetry (DSC). These techniques utilize small samples (5 to 15 mg), so they may not be practical for use in characterization of potentially nonhomogeneous recycled plastic materials.

7.6 *Infrared Analysis*—Qualitative infrared analysis using the techniques of Practice E1252 can be used to identify polymeric, chemical, and, in some cases, inorganic components of recycled plastic materials. Sample size considerations indicated in 7.5.2 may also apply to preparation of samples for infrared analysis.

7.7 *Chromatographic Analysis*—The principles of gas chromatography, described in Practice E355, and liquid chromatography, described in Practice E682, are useful for separation and classification of chemical contaminants or residues from original-use contents of plastic packages.

8. Additional Industrial Procedures

8.1 *Specimen Preparation:*

8.1.1 Using standard injection molding equipment, prepare homogenized sample plaques.

8.1.2 Plaques, or slices from plaques prepared in 8.1.1, can be used for differential scanning calorimetry (DSC), infrared analysis, and other test procedures requiring small, homogeneous specimens.

8.2 *Visible Inspection Procedures:*

8.2.1 *Inspection Table for Large, Visible Contaminants:*

8.2.1.1 Using a laboratory spatula, spread 450 ± 20 g of recycled plastic flakes or pellets on a clean, white inspection table.

8.2.1.2 Without the benefit of magnification, describe the types of individual contaminant “particles” as defined in 3.2.7, then using 10× magnification, describe the “specks” as defined in 3.2.8.

8.2.1.3 Thermal techniques (see 7.5) and infrared analysis (see 7.6) can be used to identify some of the isolated contaminants.

NOTE 5—To obtain a quantitative estimate of the contaminants, these contaminants can be removed and weighed, but there is not existing precision and bias data related to this estimated contaminant concentrations in recycled plastics.

8.2.2 *Inspection of Molded Specimens or Plaques:*

8.2.2.1 Weigh 4 to 5 g of dry plastic flake on to a polyester sheet or aluminum foil in a 15.2 by 15.2 by 0.013-cm mold. Cover with another sheet of polyester film or aluminum foil, then adjust the press temperature to at least 10°C above the melting temperature of the bulk of the test material.

8.2.2.2 Press a plaque from the recycled plastic sample. Remove the plaque from the press and cool.

8.2.2.3 Visually examine the test plaque within a 10-cm^2 area using a fluorescent-light table. For comparison, repeat 8.3.1 and 8.3.2 with a portion of virgin resin representing the bulk of the test material (for example, poly(ethylene terephthalate) (PET) if you are interested in contaminants in recycled PET).

NOTE 6—The presence of glue contamination is indicated by bonding of the plastic to the polyester sheet used as a release material during molding of the plaque.

NOTE 7—An alternative procedure for poly(ethylene terephthalate) involves melting pellets for 10 min at 275°C in an aluminum pan. This melt is rapidly quenched in ice water to prevent crystallization and the resulting disk or plaque is visually inspected for contaminants and black specks by comparison with a control disk or plaque prepared from virgin PET. In this case, black specks are attributed to degraded paper, adhesives, poly(vinyl chloride), or other contaminants in the poly(ethylene terephthalate).

8.3 *Separations Based on Density:*

8.3.1 *Water-Density Separation:*

8.3.1.1 Fill a clean plastic container with 2 L of clean water. Add sufficient nonionic surfactant to make a 2 % (weight/volume) solution and mix thoroughly.

NOTE 8—Acknowledging that water quality varies from one part of the country to another, minimum water quality for this test includes properties of Type III grade reagent water as defined in Specification D1193.

NOTE 9—Air pockets within flake material may cause the material to fold back on itself. The surfactant (for example, Triton X-100[5]) helps eliminate this problem with plastic flakes.

8.3.1.2 Obtain a representative sample of recycled plastic flakes (see 6.1) and weigh 100 ± 10 g into a clean, dry plastic container.

NOTE 10—The sample should be free of particles identified by a procedure such as that described in 8.1.

8.3.1.3 Add the surfactant solution from 8.3.1.1 to the sample container and mix well with a spatula. Allow solids to settle for at least 5 min.

8.3.1.4 Skim light plastic and any contaminants (for example, paper) from the top of the water using a small kitchen strainer. Transfer these materials to a larger strainer and rinse with water to remove residual surfactant.

8.3.1.5 Pour the remaining contents from the sample container (see 8.3.1.4) through another large strainer and wash these heavier materials with water to remove residual surfactant.

8.3.1.6 If desired, these collected heavy materials are dried and characterized by thermal (see 7.5) or infrared (see 7.6) techniques.

8.3.2 *Propanol/Water Density Separation:*

8.3.2.1 Add 1840 mL of 2-propanol and 1660 mL of water (drinking, distilled, or deionized) to a 4-L plastic bottle. Mix well to provide a solution containing 52 % (volume:volume) 2-propanol in water.

8.3.2.2 Pour 200 mL of the solution from 8.3.2.1 into a 500-mL graduated cylinder and measure the specific gravity of

[5] Available from Rohm & Haas Co., Independence Mall West, Philadelphia, PA 19105.