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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 D 5577; 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 and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—Although this guide references ISO standards, there is no similar or equivalent ISO standard covering this topic.

2. Referenced Documents

2.1 *ASTM Standards:*

D 789 Test Methods for Determination of Relative Viscosity, Melting Point, and Moisture Content of Polyamide (PA)[2]
D 792 Test Methods for Specific Gravity (Relative Density) and Density of Plastics by Displacement[2]
D 883 Terminology Relating to Plastics[2]
D 1003 Test Method for Haze and Luminous Transmittance of Transparent Plastics[2]
D 1193 Specification for Reagent Water[3]
D 1238 Test Method for Flow Rates of Thermoplastics by Extrusion Plastometer[2]
D 1457 Specification for Polytetrafluoroethylene (PTFE) Molding and Extrusion Materials[2]
D 1505 Test Method for Density of Plastics by the Density-Gradient Technique[2]
D 1898 Practice for Sampling of Plastics[2]
D 1925 Test Method for Yellowness Index of Plastics[2]
D 3418 Test Method for Transition Temperatures of Polymers by Thermal Analysis[4]
D 4019 Test Method for Moisture in Plastics by Coulometric Regeneration of Phosphorus Pentoxide[4]
D 5033 Guide for the Development of Standards Relating to the Proper Use of Recycled Plastics[5]
D 5227 Test Method for the Measurement of Hexane Extractable Content of Polyolefins[5]
E 169 Practices for General Techniques of Ultraviolet-Visible Quantitative Analysis[6]
E 355 Practice for Gas Chromatography Terms and Relationships[6]
E 682 Practice for Liquid Chromatography Terms and Relationships[6]

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[1] This guide is under the jurisdiction of ASTM Committee D~~-~~20 on Plastics and is the direct responsibility of Subcommittee ~~D20.70~~ D20.95 on ~~Analytical Methods.~~ Recycled Plastics .

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[2] *Annual Book of ASTM Standards*, Vol 08.01.
[3] *Annual Book of ASTM Standards*, Vol 11.01.
[4] *Annual Book of ASTM Standards*, Vol 08.02.
[5] *Annual Book of ASTM Standards*, Vol 08.03.
[6] *Annual Book of ASTM Standards*, Vol 14.01.

E 794 Test Method for Melting and Crystallization Temperatures by Thermal Analysis[7]
E 1252 Practice for General Techniques for Qualitative Infrared Analysis[6]
2.2 *ISO Standards:*[8]
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 D 883 and Guide D 5033.

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 D 1898. 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 D 4019), the standard test method for haze (Test Method D 1003), Karl Fisher titration (Test Method D 789), or a gravimetric procedure (13.6.1 of Specification D 1457) 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 D 1925 measures the yellowness index of clear acrylic plastics and the haze and the luminous transmittance procedure (Test Method D 1003) characterizes the color of transparent unpigmented recycled plastic materials. These tests are not readily applied to pigmented plastic samples.

NOTE 2—Test Method D 1925 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 D 1238).

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[7] *Annual Book of ASTM Standards*, Vol 14.02.

[8] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

7.3 *Density or Specific Gravity*—The displacement method for specific gravity or relative density (Test Method D 792) or the density-gradient procedure for density (Test Method D 1505) are useful techniques to determine contamination of recycled plastic flakes or pellet samples with one or more other polymers.

NOTE 3—Test Method D 1505 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.

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 D 3418) or the melting and crystallization temperatures (Test Method E 794) 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 D 3418 and E 794 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 E 1252 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 E 355, and liquid chromatography, described in Practice E 682, 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 D 1193.

NOTE 9—Air pockets within flake material may cause the material to fold back on itself. The surfactant (for example, Triton X-100[9]) 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 this solution with a hydrometer. The specific gravity should be between 0.914 and 0.917. If not, add small amounts of 2-propanol or water to the solution from 8.3.2.1 to bring the specific gravity into the desired range.

8.3.2.3 Weigh 100 ± 10 g dry light plastic (see 8.3.1.4) into a 4-L plastic pail, then add the 52 % 2-propanol/water solution (see 8.3.2.1) to this container. Stir contents with a rubber spatula to wet all flakes, then allow solids to settle.

8.3.2.4 Skim any plastic from the top of this solution with a small kitchen strainer. Transfer the separated material to a larger strainer and rinse with water.

8.3.2.5 Pour the remaining contents from 8.3.2.3 through a large strainer and collect the 2-propanol/water solution in a clean, dry bottle. Save this solution for reuse in other separations. Rinse the plastic in the strainer with water to remove residual 2-propanol.

8.3.2.6 If desired, dry the plastic in the strainer, then characterize the material using thermal (see 7.6) or infrared (see 7.7) techniques.

8.4 *Extrusion/Melt Flow Test*:

8.4.1 To estimate the relative level of contamination, process the recycled plastic through a laboratory extruder equipped with a filter screen.

8.4.2 The level of contamination is measured as a function of the rate of pressure increase resulting from increased deposits on the filter screen.

8.4.3 Contaminants isolated on the screen can be characterized by thermal (see 7.5) or infrared (see 7.6) techniques.

8.5 *Chlorinated Polymer Contamination* :

8.5.1 *Flame Test (Beilstein Test)* :

8.5.1.1 Start the flame of a Bunsen burner, then pick up a sample of the test plastic with tweezers.

8.5.1.2 Place the bare end of a copper wire in the flame until it heats to a dull orange color, then quickly remove the wire from the flame and touch it to the sample.

8.5.1.3 Press the wire into the plastic sample and allow the sample to melt on to the wire.

8.5.1.4 Remove the wire from the sample, place the wire with the sample back into the flame, and hold the wire in the flame until the plastic sample burns off the wire.

8.5.1.5 The presence of a chlorinated polymer is indicated by a strong green flame during 8.5.1.4.

8.5.1.6 When all samples have been tested, turn off the gas to the burner and store the burner.

8.5.2 *Stain K Test*:

8.5.2.1 Prepare a Stain K master batch by adding 1.00 g Sevron Brilliant Red B, 1.00 g Irgalan Yellow DRL, and 2.00 g Terasil Brilliant Blue BGN dyes[10] to 1 L water heated to 82 ± 5°C. Stir until dyes are completely dissolved, then remove the mixture from the heat, cool, and transfer to a suitable container.

8.5.2.2 Prepare a test solution by mixing 15 mL of Stain K (see 8.5.2.1), 200 mL of water, 30 mL of 5 % Tanavol solution,[11] and 2 mL 5 % (volume:volume) acetic acid in a suitable container.

8.5.2.3 Place the test solution in a 400-mL beaker on a hot plate and add approximately 100 g of the recycled plastic.

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

[10] Sevron Brilliant Red dye is available from Crompton & Knowles, Charlotte, NC, and the other dyes are available from CIBA-Geigy Corp., Greensboro, NC.

[11] 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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8.5.2.4 Boil the mixture for 5 min, then remove the beaker from the hot plate.

8.5.2.5 Discard the liquid, rinse the plastic thoroughly with water, and transfer the washed plastic to a clear plastic bag.

8.5.2.6 Visually determine the presence of colored plastic under fluorescent light. Poly(vinyl chloride) and poly(vinylidene chloride) dye purple, while polyesters appear as blue regions, and polyamides appear yellow or orange.

8.6 *Stain 5 Test for Nylon and Polyesters*:

8.6.1 Add 1 mL NaCl, 2 drops of a nonionic surfactant, 0.3 g of Stain #5, and 300 mL of water to a 500-mL beaker.[12]

8.6.2 Add approximately 100 g of recycled plastic to the beaker and place the beaker on a hot plate.

8.6.3 Boil the mixture for at least 2 min, then remove the beaker from the hot plate.

8.6.4 Pour the beaker contents into a strainer and rinse with cold water.

8.6.5 Visually examine the washed plastic under fluorescent light. Nylon (polyamide) samples will turn green or blue-green, polyesters will turn yellow, and paper contamination will appear as red material.

8.7 *Solvent Extraction Procedures* :

8.7.1 *Xylene Extraction*:

8.7.1.1 Weigh 100 g of recycled plastic into an 800-mL Erlenmeyer flask and add 200 mL of *p*-xylene.

8.7.1.2 Seal the flask with a cork stopper fitted with a thermometer. Adjust the thermometer so that the bulb touches the bottom of the flask.

8.7.1.3 Maintain a temperature of 65 ± 2°C and heat the solvent and sample on a hot plate for 1 h. Shake the flask every 2 to 3 min to mix the contents.

8.7.1.4 After 1 h, cool the sample to room temperature.

8.7.1.5 Transfer a portion of the *p*-xylene extract to a suitable cell and obtain an infrared spectrum of the extract versus a *p*-xylene reference liquid in accordance with Practice E 1252. Appearance of an absorption band at 5.7 µm indicates the presence of an ethylene-vinyl acetate (EVA) copolymer. Other bands in the infrared spectrum may be characterized in accordance with Practice E 1252.

8.7.2 *n-Hexane Extraction*:

8.7.2.1 To adapt Test Method D 5227 to the characterization of contaminants in recycled plastic materials, add 1 L of *n*-hexane to the resin kettle in accordance with Test Method D 5227 and clamp the kettle assembly into a water bath maintained at 49.5 ± 0.5°C.

8.7.2.2 Add 2.5 to 5 g of the plastic sample, replace the kettle head with a condenser, and extract the sample for 2 h.

NOTE 11—This procedure developed for polyolefins, may be modified for use with other polymers by using higher boiling solvents and extraction at higher temperatures.

8.7.2.3 After extraction, filter the resin kettle contents through a fritted porcelain funnel.

8.7.2.4 Analyze an aliquot of the filtered extract using thermal techniques (see 7.5), infrared spectroscopy (see 7.6), or a suitable alternative technique such as gas chromatography to identify the extracted components.

8.8 *Polystyrene as a Contaminant* :

8.8.1 Weigh 10 g of the recycled plastic sample into an Erlenmeyer flask and shake 2 h with 100 mL of a solvent that is transparent at 254 nm (for example, dichloromethane or tetrahydrofuran) based on spectroscopic techniques of Practice E 169.

8.8.2 Dilute 2.5 mL of the extract to 50 mL with the same solvent.

8.8.3 Filter 5 mL of this solution through a 0.45-m filter into a 1-cm quartz cell.

8.8.4 Obtain the ultraviolet spectrum of this filtered extract in accordance with Practice E 169. Compare the absorbance at 254 nm with the absorbance of a solvent blank. Significant absorbance at 254 nm indicates the presence of a styrenic polymer or another aromatic species (for example, poly(ethylene terephthalate) or poly(butylene terephthalate)).

9. Report

9.1 Report the following information:

9.1.1 The types of contaminants, and

9.1.2 The techniques used to arrive at the identifications reported in 9.1.1.

10. Keywords

10.1 compression moldings; contaminants; density separations; extrusion; post-consumer plastics; recycled plastics; visual inspection

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[12] Triton N-101, available from Ashland Chemical, 729 Mauney Dr., Columbia, SC 29201, is a typical nonionic surfactant used for this test. The Stain #5 reagent is available from J. Belmar Inc., N. Andover, MA.

APPENDIX

(Nonmandatory Information)

X1. IDENTIFICATION OF CONTAMINANTS ADDRESSED BY SPECIFIC TEST PROCEDURES

TABLE X1.1 Identification of Contaminants Addressed by Specific Test Procedures

Test Procedure (Section Number)	ASTM or ISO Method	Components Detected
Ash test (7.4.1)	ISO 3451/1	Inorganic fillers, some metals
Chlorinated polymers (8.5)		Chlorinated materials
Chromatographic analysis (7.7)	Practice E 355; Practice E 682	Chemicals, original-use contents
Color or yellowness index (7.2.1)	Test Method D 1925	Colored or degraded materials
Density or specific gravity (7.3)	Test Methods D 792; Test Method D 1505	Contamination by other polymers
Density separations, water or propanol/water (8.3)		Paper, other polymers, metals
Extrusion/melt flow test (8.4; 7.2.2)	Test Method D 1238	Incompatible polymers, metals, dirt, insoluble material
Flame or beilstein test (8.4.1)		Chlorinated materials
Haze or transmittance (7.2.1)	Test Method D 1003	Moisture; incompatible polymers
Inspection table (8.2.1		Visible contaminants (specks, particles)
Infrared spectroscopy (7.6)	Practice E 1252	Contaminant functional groups
Magnets (7.4.2)		Ferrous metals
Moisture (7.1)	Test Methods D 789; Test Method D 1003; Test Method D 4019	Water content
Molded specimens or plaques (8.2.2)		Paper, adhesives, poly(vinyl chloride), incompatible polymers
Polystyrene contaminant (8.8)		Polystyrene; other aromatic polymers
Product uniformity (7.4)		
Solvent extraction procedures, hexane or xylene (8.7)		Glues, soluble contaminants
Specimen preparation (8.1)		
Stain 5 test (8.6)		Nylon, polyesters, paper
Stain K test (8.5.2)		Chlorinated polymers
Thermal analysis (7.5)	Test Method D 3418; Test Method E 794	Polymer identity
Ultraviolet spectroscopy (8.8)		Aromatic polymers
Visible inspection procedures (7.2; 8.2)		Dirt, specks, particles, materials with color different from bulk polymer