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Standard Practice for Separation and Identification of Poly(Vinyl Chloride) (PVC) Contamination in Poly(Ethylene Terephthalate) (PET) Flake [1]

This standard is issued under the fixed designation D 5991; 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] This practice is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee ~~D20.70~~ D20.95 on ~~Analytical Methods.~~ Recycled Plastics .

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1. Scope

1.1 This practice covers four procedures for separation and qualitative identification of poly(vinyl chloride) (PVC) contamination in poly(ethylene terephthalate) (PET) flakes.

NOTE 1—Although not presented as a quantitative method, procedures presented in this practice may be used to provide quantitative results at the discretion of the user. The user assumes the responsibility to verify the reproducibility of quantitative results. Data from an independent source suggest a PVC detection level of 200 ppm (w/w) based on an original sample weight of 454 g.

1.2 *Procedure A* is based on different fluorescence of PVC and PET when these polymers are exposed to ultraviolet (UV) light.

1.3 *Procedure B* is an oven test based upon the charring of PVC when it is heated in air at 235°C.

1.4 *Procedures C and D* are dye tests based on differential staining of PVC and PET.

NOTE 2—Other polymers (for example, PETG) also absorb the stain or brightener. Such interferences will result in false positive identification of PVC as the contaminant.

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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazards see Section 8.

NOTE 3—There is no equivalent ISO standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1600 Terminology for Abbreviated Terms Relating to Plastics[2]

D 5033 Guide for the Development of Standards Relating to the Proper Use of Recycled Plastics[2]

E 380 Practice for Use of the International System of Units (SI) (the Modernized Metric System)[3]

3. Terminology

3.1 The terminology used in this practice is in accordance with Terminology D 1600 and Guide D 5033. Units and symbols are in accordance with Practice E 380.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *light material, n*—paper, polymers such as polyethylene and polypropylene, and other materials with densities less than 1.00 g/cm^3.

4. Summary of Practice

4.1 For the Beilstein Test, chlorine-containing materials heated in a flame in contact with a copper wire produce a characteristic green flame.

4.2 With Procedure A, a known amount of PET flakes is exposed to ultraviolet radiation. PET normally fluoresces with a blue or violet color. Flakes fluorescing with different colors are removed, weighed, and identified as PVC if they burn with a bright green flame when heated on a copper wire.

[2] *Annual Book of ASTM Standards,* Vol 08.03.

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

4.3 With Procedure B, PET flakes are heated in an oven maintained at 235 ± 5°C. After 45 min, the flakes are visually examined with removal and subsequent weighing of black, charred pieces. Pieces are confirmed as PVC using the same flame test mentioned in 4.1.

4.4 With Procedure C, PET flakes are soaked in a blue acetone dye solution that preferentially stains any PVC flakes. These flakes are visually identified and subsequently removed, and weighed. If desired, the identification of PVC flakes is confirmed with the flame test.

4.5 With Procedure D, PET flakes are heated with an aqueous solution containing an optical brightener that also preferentially stains the PVC flakes. The flakes are sorted under UV light in a dark room with removal and subsequent weighing of any blue fluorescing PVC flakes.

5. Significance and Use

5.1 Presence of even low concentrations of PVC in recycled PET flakes may result in equipment corrosion problems during processing. The level of PVC contamination may also dictate the market for use of the recycled polymer in secondary products. Procedures presented in this practice are used to identify and, if desired, estimate the concentration of PVC contamination in recycled PET flakes.

6. Apparatus

6.1 *Procedure A*:
6.1.1 *Bright Aluminum Tray*, shallow depth (0.75 cm).
6.1.2 *Light Fixture*, equipped with GE F40/BLB, or equivalent, black light bulbs.
6.1.3 *Ultraviolet Shield Glasses* .
6.1.4 *Tweezers*.
6.2 *Procedure B*:
6.2.1 *Circulating or Forced-Air Oven* , equipped with a temperature readout device for the range of 230 to 240°C.
6.2.2 *Baking Trays*, 14 by 20 by 3 cm.
6.2.3 *Tweezers*.
6.2.4 *Thermally Insulated Gloves* , or equivalent.
6.3 *Procedure C*:
6.3.1 *Measuring Cup*, 1.5 L.
6.3.2 *Plastic Stretch or Cling Wrap* .
6.3.3 *Paper Towels*, newspaper, or equivalent.
6.3.4 *Tweezers*.
6.4 *Procedure D*:
6.4.1 *Measuring Cup*, 1.5 L.
6.4.2 *Tweezers*.
6.4.3 *Hot Plate*, or equivalent heating device.
6.4.4 *Light Fixture*, equipped with GE F40/BLB, or equivalent, black light bulbs.
6.4.5 *Ultraviolet Shield Glasses* .

7. Materials

7.1 *Detergent*.
7.2 *Rit™ Tint and Dye* (Navy Blue #30),[4] or equivalent, required for Procedure C.
7.3 *Rit™ Brightener and Whitener*,[4] or equivalent, required for Procedure D.

8. Hazards

8.1 The ultraviolet light used in Procedures A and D may be harmful to exposed skin and unprotected eyes, so a UV filtering face shield or UV absorbing glasses should be worn when performing this test. Exposed hands and arms should be covered with gloves and long sleeves.

8.2 Hot trays and flake samples should be handled with thermally insulated gloves to avoid burns while placing trays containing samples into or removing them from the oven (Procedure B).

8.3 Acetone used to prepare the dye solution for Procedure C is flammable. Use this reagent only in a well ventilated area free of open flames or spark sources.

9. Procedure

9.1 *Flame Test to Verify PVC Identification (Beilstein Test)*:
9.1.1 Heat a copper wire in a propane torch flame until it is very hot.

[4] Available from CPC Specialty Products, Indianapolis, IN.

9.1.2 Touch the hot wire to a suspected PVC flake to melt a small amount of the flake on to the wire.

9.1.3 Reheat the wire tip in the flame. If the tested flake is PVC, a bright green flame will be visible as the wire heats and the plastic burns. Absence of a green flame indicates that the test flake is not PVC.

9.2 *Procedure A*:

9.2.1 Weigh 454 ± 10 g of clean, dry PET flake sample and transfer the sample into a shallow aluminum tray.

9.2.2 Place the tray under the UV light in a dark environment.

9.2.3 Using tweezers, sort through the flakes in the tray and remove all particles that do not exhibit the standard PET blue or violet-blue color. Set the isolated flakes aside in a separate tray for subsequent weighing or testing in accordance with 9.1.

NOTE 4—Finding uniquely colored flakes under UV light should not be interpreted as fact that these particles are PVC. PVC can exhibit many colors (yellow/green, bright blue, dark violet, light orange) under UV light, and because of the blue colors exhibited by some PVC material, these flakes may not be discernible from PET flakes under UV light.

NOTE 5—Clear PET fluoresces strongly emitting a blue to violet-blue color. Other PET products, however, have been observed to fluoresce with pale blue or clear, dark purple, or turquoise colors. Green PET used for soft drink containers normally fluoresces from a bright to a dull green under UV light and amber PET flakes appear black under UV light.

NOTE 6—Paper, labels, and assorted pigmented plastics may also exhibit a spectrum of fluorescent colors or these materials may appear very dark under UV light.

9.3 *Procedure B*:

9.3.1 Weigh 454 ± 10 g of clean, dry PET flake sample and transfer the sample into a baking tray.

9.3.2 Adjust and equilibrate the oven temperature to 235 ± 5°C.

9.3.3 Quickly place the tray containing the flake into the heated oven.

9.3.4 After 45 min, remove the hot tray from the oven and place it on a suitable surface to cool to room temperature.

NOTE 7—If heating times are short, any PVC may not char completely and the discoloration may stop at a yellow-orange state that is typical of the color produced by similar heating of glue residues. The heating time must be sufficient to totally blacken any PVC flakes. Flakes in the center of a mound of PET flakes may degrade at a slower rate than material on the surface.

9.3.5 Spread the cool, baked flake on a clean, white surface. Using tweezers, sort through the flakes in the tray and remove any black, burnt pieces of plastic.

NOTE 8—Burnt, melted polyethylene or PET with blackened glue residues usually have a different physical appearance than PVC. The suspected presence of these contaminants will usually require testing by 9.1 to verify the presence of PVC.

NOTE 9—While placing wet flake into the high temperature oven is possible, the moisture will tend to keep the flake cooler for a longer period of time. Consequently, heating times longer than 45 min may be needed to compensate for this cooling effect.

9.4 *Procedure C*:

9.4.1 Transfer clean PET flake from 9.12 into a 3 to 4 L metal container by tapping the flake-filled strainer against the rim of the container.

NOTE 10—Since it is a good practice to run a standard with any test, add a 1.54 cm square piece of known PVC to the unknown flakes.

9.4.2 Add 1.2 L of the blue acetone dye solution (10 % w/v) to the container to ensure coverage of all of the flakes with the solution. Cover the container tightly with plastic stretch wrap.

9.4.3 Soak the flake for 15 to 20 min, then pour the blue flake and dye solution through a strainer. Transfer the acetone/water dye solution into an appropriate glass storage vessel and seal tightly.

NOTE 11—If the test square of PVC (Note 8) does not stain a very dark blue, the blue dye solution may no longer be usable and it should be replaced. As acetone evaporates from an uncovered container or loss during transfer of the flake to the strainer, longer time will be required to carry out this test.

NOTE 12—The blue acetone/water dye solution should be reused as many times as possible. Add small amounts of 50:50 acetone:water to replenish lost liquid.

9.4.4 Immediately rinse the dyed flake under running water until there does not appear to be any blue liquid being rinsed from the flakes.

9.4.5 Spread the washed flake on white paper towels or a clean, white surface. Using tweezers, sort through the flakes in the tray and remove any uniformly dark blue stained pieces of plastic.

9.4.6 Allow the isolated blue PVC flakes to air dry.

9.5 *Procedure D*:

9.5.1 Transfer clean PET flake into a 3 to 4 L metal container by tapping the flake-filled strainer against the rim of the container.

NOTE 13—Since it is a good practice to run a standard with any test, add a 1.54 cm square piece of known PVC to the unknown flakes.

9.5.2 Add 1500 ± 10 mL of water and 3.5 ± 0.2 g of the Rit Brightener and Whitener to the container. Bring the mixture to a boil on a hotplate.

9.5.3 After boiling for 30 min, remove the container from the hotplate and pour the contents through a strainer. Rinse all remaining flakes into the strainer using additional water.

NOTE 14—Additional paper floating on the surface may also be skimmed off at this time.

9.5.4 Immediately rinse the dyed flake under running water to cool it down and rinse off any remaining optical brightener solution.

9.5.5 Transfer the flake from the strainer to a shallow aluminum pan making sure not to lose any of the flakes. Place the tray under the UV light in a dark environment.

9.5.6 Using tweezers, sort through the flakes in the tray and remove all particles that glow bright blue. Set the isolated flakes aside in a separate tray for subsequent weighing or testing in accordance with 9.1.

NOTE 15—Pieces of label and PET with adhering glue residues will also glow blue, although PET with glue residue will not show the intense blue fluorescence observed for PVC. Flimsy pieces of label are quickly identified from the rigid PVC flake.

10. Report

10.1 Report the test procedure used to isolate and identify PVC flakes.

11. Keywords

11.1 contamination; poly(ethylene terephthalate) (PET); poly(vinyl chloride) (PVC); recycled plastics