



The Association of Postconsumer Plastic Recyclers

PET Flake Wash Test PET-S-02

The following protocol is designed to provide a generic wash process for evaluation of the effects of container components (adhesives, labels, etc.) on recycled PET material. The method of incorporating the component with the flake for washing and the specific analysis technique will be provided in the component specific protocol.

This protocol does not purport to address all of the safety issues, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

Equipment/Supplies List

- Container component for evaluation (e.g. label or adhesive)
- Clean, PET flakes about 3/8" nominal (300 grams/test) (depending upon particular evaluation, component may be applied to PET container prior to granulation)
- Preferred - Baffled beaker - stainless steel, six inch diameter by nine inches in height with four 3/4-inch baffles.
Alternate - similar size beaker without baffles
- Hot plate capable of heating to 190⁰C
- Scale or balance capable of measuring 500 (+\ - 0.5) grams
- Oven capable of drying flake at 60⁰C
- Cookie sheet (or other suitable tray with a minimum area of 120 sq.in.)
- Cover for beaker which can accommodate thermometer and stirring impeller rod
- Overhead stirrer capable of 1,000 rpm
- Stirring impeller - pitched, 3 blades, and 3.0 inch diameter.
- Manual stirring rod
- Thermometer
- Strainer - non-aluminum
- Distilled or deionized Water
- Graduated cylinder
- Triton X- 100 nonionic surfactant (available from Union Carbide at 1-800-969-2707)
- Caustic (granular NaOH)



The Association of Postconsumer Plastic Recyclers

Be sure to read all material safety data sheets.

Prepared by Rutgers, The State University of New Jersey Center for Packaging Science
& Engineering

Bldg 3529 - Busch Campus, Piscataway, NJ 08855
Tel: (732) 445-3224 - Fax: (732) 445-0777

PET Sample Wash Procedure

Be sure to use appropriate laboratory safety procedures / Gloves, safety glasses, etc.

1. Preparation of container component for evaluation - this will be specific for each container component and may require affixing component to container prior to granulation.
2. Prepare "wash" solution in the beaker consisting of 2000 ml water, ~0.3% by weight (6.0 gms or 5.7 ml) of Triton X-100 and 1.0% by weight (20 gms) caustic (NaOH).
Note: Triton X-100 must be dissolved in cold water prior to the addition of caustic!
3. Heat solution on hot plate to 190°F while covered with beaker cover to minimize evaporation.
4. Insert overhead stirrer so that the impeller is 1/4 inch above the bottom of the beaker.
5. Turn on overhead stirrer and adjust to 1000 rpm.
6. With the overhead stirrer on, carefully add the 500 grams of PET flakes with the component for evaluation to the solution.
7. Readjust the overhead stirrer to 1000 rpm and continue agitation for 15 minutes while maintaining solution at 190°F. Monitor stirrer rpm and adjust as required to maintain 1000 rpm.
8. Turn off and remove overhead stirrer. Remove beaker from heat and let stand for five minutes to allow lights to float to the surface.



The Association of Postconsumer Plastic Recyclers

9. Pour solution and “floats” through strainer while maintaining PET flakes in beaker.
10. Add two liters of room temperature clean water to beaker and stir for one minute. Let stand for five minutes to allow remaining lights to float to the surface. Repeat step 9. Discard (or keep for analysis) the material remaining in strainer and rinse strainer.
11. Transfer PET flakes to strainer, rinse flakes in cold running tap water while vigorously stirring the flakes for 10 minutes using the manual stirring bar. Drain the material.
12. Spread flakes on cookie sheet (or other suitable tray), place in 60°C oven and allow to dry. Separate flake and any remaining component if required. Save for visual and instrumental evaluations, which will be detailed in the appropriate protocol.

Note

To prepare the control PET flake for comparison, follow above procedure while omitting the container component material.

PET Thermal Cycle Test Procedure

This procedure does not purport to address all of the safety issues, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

Important Note:

It is assumed that any label or similar contamination remaining after the wash protocol has been performed and which is not adhered to PET flake material would be removed by a subsequent separation process and would not present a problem. Therefore, prior to performing this procedure, any label contamination which is not adhered to the PET flake must be removed from the material. This may be accomplished by lab scale air classification equipment, by hand separation, or by any means sufficient to accomplish the task.



The Association of Postconsumer Plastic Recyclers

Equipment/Supplies List

Scale capable of weighing 100 +/- 1 grams.

Oven capable of heating to 200 +/- 5⁰C. Ideally, the oven should have a forced air circulation to maintain temperature.

Aluminum (or other suitable container for oven use) pan capable of holding 25 grams of PET flake.

Washed quantity of recycled PET, both control and test samples

Procedure

1. Preheat oven to 232 +/- 5⁰C.
2. Weigh 25 grams of dry washed PET flakes and place in the aluminum pan.
3. Place aluminum pan with PET flakes in oven for 30 minutes.
4. Remove aluminum pan from oven and allow to cool.



The Association of Postconsumer Plastic Recyclers

Evaluation of PET Container Component Color

The protocol does not purport to address all of the safety issues, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1. Produce 500gram clear PET flake samples according to the procedure specified in the *Protocol For Producing PET Flake For Evaluation "PET Sample Wash Procedure"*. Be sure to produce a sample of control flake from recycled PET bottles without the container component to be evaluated.
2. Visually inspect each flake sample for contamination. Remove extraneous material unless the component to be evaluated does not adhere to the PET.
3. Produce a heat-stressed sample per the "Thermal Cycle Test Procedure" for each sample being tested and controls, keeping some of each washed sample unheated.
4. Visually inspect samples after thermal cycling for discoloration and note gross differences.
5. Perform color analysis, according to the machine manufacturers instructions, on each sample produced (washed control sample, washed and thermally cycled control sample, washed test sample, and washed and thermally cycled test sample) using the following format:
 - a. Take five measurements, in reflectance or transmission, on each sample. Record data as X, Y, Z tristimulus values, CIE XYZ, CIE L*a*b* or Hunter **L a b** color coordinates, or equivalent. Adjust the position of the sample holder prior to each measurement to expose different sample areas to measurement.
 - b. Report all of the axis readings (such as **L, a, b**) for all five samples and the average for each sample.
6. Evaluation. All three color measurements, **L, a,** and **b,** are important to the possible use of the recyclate. The human eye can discern about one **b** unit change. The **L** measures brightness vs dinginess of the flake. **a** measures red/green color



The Association of Postconsumer Plastic Recyclers

components. **b** measures yellow/blue color components.

Depending on the end use, different amounts of variation from the control can be accepted. Generally, changes of more than two to three **a** or **b** units or five **L** units upon heating is cause for concern. Absolute **b** values over 3 for unheated flakes in reflection or transmission color measurement and absolute **b** values over 5 for heated flakes measured in reflection may be unacceptable for reuse in bottles.

Comparisons should be made between control and test samples for the same treatment and between thermally cycled and not thermally cycled for the same base sample.

The guidance contained in this document does not guarantee acceptance or rejection of tested materials. APR encourages users of this protocol to discuss results with various knowledgeable parties using and specifying post consumer PET material to determine usefulness of subject test materials.