



The Association of Postconsumer Plastic Recyclers

HDPE Flake Wash Test HDPE-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 natural HDPE 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, natural HDPE flakes about 3/8" nominal (500 grams/test) (depending upon particular evaluation, component may be applied to HDPE 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)



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Be sure to read all material safety data sheets.

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Natural HDPE Sample Preparation 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 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 natural HDPE flakes with or without 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 immediately strain the solution, test component, and flake.



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9. Rinse the HDPE flakes while still in the strainer with cold running tap water and stir vigorously for 5 minutes using the manual stirring bar. Drain the material.
10. 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 HDPE flake for comparison, follow above procedure while omitting the container component material.

HDPE Plaque Production 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 HDPE 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 HDPE 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.

Equipment/Supplies List

- Plaque mold, capable of producing 1/8 inch (0.125 inches) thick plaque. The minimum recommended plaque size is 2 inches x 2 inches.
- Natural or clear HDPE flakes prepared as required for evaluation. The amount needed is determined by the plaque mold size and design.
- Balance or scale capable of weighing 100 +/- 0.1 grams.



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- Hot press, capable of heating to 350°F and applying 10,000 pounds of force in approximately 15 seconds.
Warning – Equipment is a potential source of burns, pinching and crushing

Procedure

1. Preheat hot press to 350°F.
2. Weigh and add sample to mold cavity. Add 2 to 10% more than required for the specific mold volume to allow for flask. Close mold.
3. Place mold in hot press. Bring platens together without applying force (just until they touch). Allow to preheat for 10 minutes
4. Apply force to mold smoothly from 0 to 10,000 pounds in 15 seconds.
5. Maintain 10,000 pounds force for 1 minute
6. Release force and remove mold from hot press
7. Allow to air cool until warm to the touch. Remove plaque.

HDPE Plaque Evaluation Procedure

Inclusions and 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. Visually inspect plaque samples for discoloration, inclusions of extraneous material, and gross differences. Pay particular attention to gels, specks, and voids, describing each in size and frequency
2. 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:



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- 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. The **L** measures brightness vs dinginess of the flake. **a** measures red/green color 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 four **a** or **b** units or five **L** units is cause for concern. Absolute **b** values over 13 and **L** values under 68 may be unacceptable for many uses. 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 HDPE material to determine usefulness of subject test materials.