

Benchmark Evaluation for Clear PET Articles with Labels and Closures

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Introduction – Scope, significance, and use

This is a laboratory scale evaluation that can be used to assess the compatibility of PET packaging design features such as labels, closures, dispensers, and attachments with some of the common commercial scale recycling process steps. This test is only applicable to clear PET articles. Product developers, as well as those who specify products, can employ this test to maintain and improve the quality and productivity of PET recycling.

The evaluation is conducted with molded articles made from clear PET and employs the packaging design feature of interest, (the “innovation material”). Clear PET refers to natural color PET with no colorants added at the molding process. Molded articles are most often expected to be PET packages: injection stretch blow molded containers, extrusion blow molded containers, or thermoformed sheet products; but may include any article that would be sorted as clear PET in the collection and sorting system.

This test is used to understand how molded articles behave in the wash, float/sink, and elutriation steps. The test is also valuable for understanding any impact of resin drying and melt heat history during injection molding on the formation of color, haze, or inclusions in molded PET.

This test does not include an assessment of flake extrusion and melt filtration that is a necessary component of the Critical Guidance Protocol for PET Articles with Labels and Closures. This test does not consider the effects of package design on sortation in a MRF or reclaiming process.

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Method summary and flow diagram

Molded articles used for control can either be made from a named control resin or by qualifying a resin for control using the PET Heat History and Color Evaluation Test, PET-S-03.

Innovation articles are created according to the “Preparation of PET Articles for Evaluation” Practice which explains how to create articles for evaluation that include labels, closures, or attachments. Innovation articles are usually mixed with control articles that do not have the design feature of interest; this mix is granulated to make a flake blend sample used in the evaluation.

The evaluation involves:

- Granulation, wash and elutriation of each set of control bottles and articles which employ the innovation.
- Injection molding plaques directly from washed flake made from each material.

Inks and adhesives can impact the costs of managing a wash system and waste disposal. Therefore, this procedure requires specific observations be made to characterize the impact of inks on the wash water for inks printed onto labels, or directly on to the PET package surface. These observations are presented in PET-S-01.

This procedure offers some optional steps that can be valuable in certain investigations. Examples are:

- The oven bake test for flake, and flake color evaluation to evaluate for contamination before injection molding of flake, PET-S-10.
- A materials balance to confirm that materials are recovered as expected as sinking or floating solids, PET-S-04.

A flow diagram in Appendix I illustrates the testing steps.

Reference Documents

The following documents are referenced in this Benchmark Protocol:

APR PET Standard Laboratory Practices, PET-P-00

APR PET Screening Test Methods:

PET Package Materials Balance, PET-S-04

Labels for PET - Wash Water Evaluation, PET-S-01

PET Flake Clumping Evaluation, PET-S-08

PET Flake Oven Bake Evaluation, PET-S-10

Measurement of PET Flake or Pellet Discoloration, PET-S-02

Evaluation of PET Plaques for Color, Haze, and Inclusions, PET-S-09

ASTM Methods

ASTM D4603-18 Standard Test Method for Determining Inherent Viscosity of Poly(Ethylene Terephthalate) (PET) by Glass Capillary Viscometer

ASTM D1238 – 13 Standard Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer

Method steps

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The following steps are taken with reference to the PET Practices, PET-P-01 through PET-P-08 (included in document PET-P-00) and the flow diagram shown in Appendix I:

1. Secure molded control articles.
2. Prepare these innovation articles according to the Practice for Preparation of PET Articles for Evaluation.
3. Create a blend of control and innovation articles as described in the Preparation of PET Articles for Evaluation
4. Separately grind control articles to create flake sample A and then grind the blend of control and innovation articles to create flake sample B.
5. Separately wash flake samples A and B.
6. When flake sample B contains labels, adhesives, or printed surfaces, retain a sample of wash water created from washing flake sample B for evaluation.
7. Elutriate flake samples A and B. Note: this is an optional step in this test method. It is not necessary if the investigator has prior experience and confidence that elutriation is not necessary to remove any innovation material from flake sample B and impact final injection molding and color results.
8. If necessary, crystallize flake samples A and B prior to desiccant drying to prevent any sticking in the drier.
9. If necessary to allow feeding to the injection molding unit, grind flake samples A and B to a finer flake size.
10. Desiccant dry and injection mold plaques from each of flake samples A and B to create amorphous plaques A and B.

Measurements, report, and guidance values

Wash and elutriation evaluations

| Property | Test method | APR Guidance Preferred values | Additional Guidance |
|-----------------------------------|--|--|---|
| <u>Required values</u> | | | |
| Clumping test with flake sample B | PET Flake Clumping Evaluation, PET-S-08 | <1.0 wt% retention on screen and foil for each weighted flake evaluation | The clumping test is employed only for those design features that employ components that will sink in water with PET flake. |
| Hot caustic resistant ink | Assessment given in PET-S-01 | An assessment of “good ink retention” | Present photo(s) for documentation |
| Wash-off ink | Assessment given in PET-S-01 | An assessment of “ink removal by Whatman #1 filter paper” | Present photo(s) for documentation |
| <u>Optional values</u> | | | |
| Flake bake test | PET Flake Oven Bake Evaluation, PET-S-10 | | Can reveal contamination before later evaluations |
| Flake color | Measurement of PET Flake or Pellet Discoloration, PET-S-02 | | Can reveal contamination before later evaluations |
| Materials balance | PET Package Materials Balance, PET-S-04 | | Can reveal contamination before later evaluations |

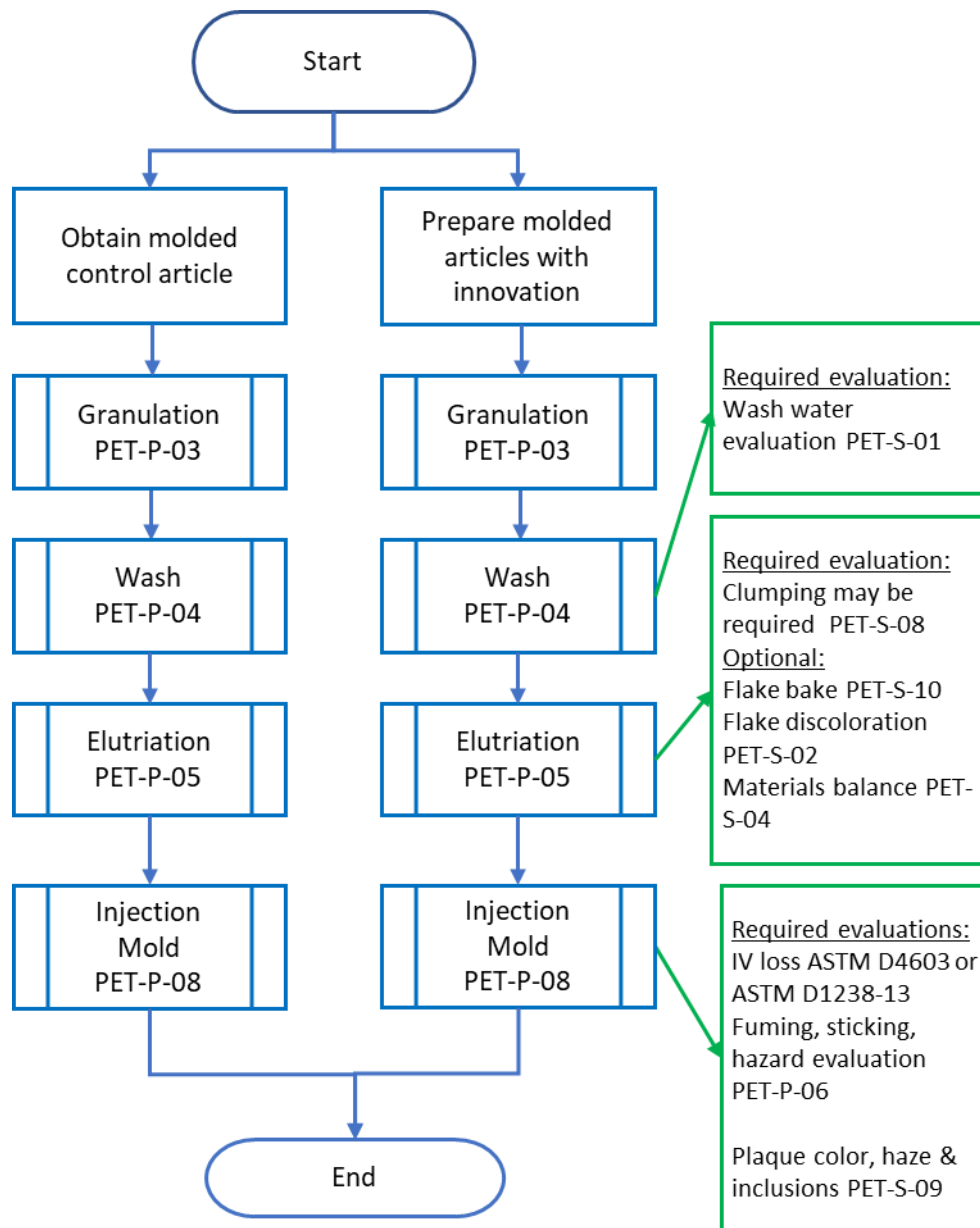
Evaluation of molding and molded plaques

IV loss - The table below calls for reporting the IV loss when samples are injection molded into plaques. The following steps are used to report this value using samples A and B for illustration:

- Measure the IV of flake created from control articles as well as from innovation articles.
- Measure the IV of the resulting injection molded plaques for each of the control and innovation samples.
- Measure the IV loss for sample A with molding and call that value A'. This is the IV loss for the control.
- Measure the IV loss for Sample B with molding and call that value B'. This is the IV loss for the innovation sample.

| Property | Test method | APR Guidance Preferred values |
|---|--|---|
| <u>Required values</u> | | |
| Assess any adhesion of PET flakes on the inside surface of the drier | Visual inspection of the insider of the drier after dried flake is removed. | The drier should be clean with no flakes that adhere to the drier walls after tapping outside of unit with a rubber mallet. |
| Assess adhesion of PET flakes to the inside surface of the hopper or feed on injection molding unit | Visual inspection of the injection feed hopper and feed throat after molding a sample. | No adhesion should be observed after tapping outside of unit with a rubber mallet. |
| IV loss when molding plaques | ASTM D 4603 solution IV with phenol/tetrachlorethane at 30°, or ASTM D1238 – 13 method B | Difference in A' and B' is 0.025 dg/l or less |
| L value of plaques B | Plaque Color Measurement, PET-S-09 | >82 |
| a* value of plaques A and B | Plaque Color Measurement | Less than 1.5 units difference between plaques A and B |
| b* value of plaques A and B | Plaque Color Measurement | Less than 1.5 units difference between A and B |
| % haze of plaques A and B | Plaque Color Measurement | Control not to exceed 9% value, and test not to exceed more than 10% units greater than control. |
| Inclusions and specks in plaques T5 | Plaque Color Measurement | If A = 0; B is 2 or less If A = 1; B is 4 or less If A = 2; B is 6 or less |

Appendix I Benchmark Flow Diagram Natural PET Articles with Labels and Closures



DOCUMENT VERSION HISTORY

| Version | Publication Date | Revision notes |
|---------|-------------------|---|
| 1 | November 16, 2018 | |
| 2 | April 11, 2019 | Revised Haze Guidance Preferred Values as approved by PTC in March 2019 |
| 3 | November 01, 2022 | Revised guidance for ink performance and clump test |
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