ASSOCIATION OF PLASTIC RECYCLERS

EARLY EVALUATION TEST METHOD:

Preliminary Screening Evaluation for Clear PET Packaging with Oxygen-Ingress and/or Carbonation-Retention Barrier

DOCUMENT NUMBER: PET-EE-02

Revision Date: November 12, 2024

Introduction

Scope, Significance, and Use

This laboratory scale procedure provides a standardized screening method to employ in early evaluations of the potential impact of PET containers that employ an oxygen-ingress or carbonation-retention barrier on any color and haze formation that can occur when these containers are recycled into the clear PET stream.

This method can be employed by materials and packaging developers, as well as those who specify packaging and materials, to evaluate variables that impact color and haze formation and to identify candidate materials and packaging designs expected to have the least impact on color formation in recycling. While any laboratory may employ this evaluation, it can be valuable to use an independent contract lab when comparing a variety of materials to reduce the impact of laboratory to laboratory differences in the outcome of the color of molded plaques.

As specific examples, this method can be employed to:

- Compare color and haze formation of different oxygen-ingress-barrier technologies or products.
- Compare color and haze formation resulting from carbonation retention barriers.
- Compare the color and haze impact of various addition levels of a barrier additive in PET.
- Evaluate the color and haze impact of employing a barrier additive in a monolayer versus multilayer package.
- Evaluate any impact of package age or storage conditions on color and haze formation.

The test method was primarily developed with the assessment of active oxygen barrier materials in mind. These materials are typically additives mixed with PET resin just ahead of an injection press or extruder; the additives are melt-blended with PET and intended to react with oxygen to reduce oxygen ingress into a filled package during shelf storage of foods and beverages. Oxygen scavenging materials can also be employed in multi-layer constructions.

This test was also developed specifically to employ a small number of barrier containers for testing so that availability of containers for testing, and the cost/time to create test containers, have least impact on those conducting this valuable testing. The APR encourages the use of packages and materials that perform well in this early evaluation method over materials that do not. However, this test is not to be misconstrued with APR's more rigorous Tests PET-B-01 or PET-CG-01. Specifically, this screening test does not include an assessment of flake extrusion and melt filtration, nor impact on IV and IV build rate that are necessary components of the Critical Guidance Protocol for clear PET

containers. This screening test does not employ a 50% blend of test and control material as called for in PET-CG-01. This screening test does not consider the effects of package design on sortation in a MRF or reclaiming process.

Some gas permeation barrier technologies employ non-PET barrier layers or coatings. In recycling evaluation, liberation and separation of these barriers from PET might benefit from mechanical granulation, or from flake wash, or an elutriation step that is not a part of this screening test method for oxygen scavenger additives. A route is given in this procedure that will allow comparing barrier technologies that benefit from granulation, wash and/or elutriation steps.

Test Method Summary

Evaluation of barrier PET containers involves the following steps:

- A first step is to secure PET containers for testing. Containers can be secured from any source of interest: a laboratory molding process, a production process, from a retail store shelf, or a bale of recycled bottles are example sources. The Appendix to this test discusses sample selection considerations in detail.
- Labels and any adhesive residue are removed from the container or sheet employed before testing. Any product residue or dirt from handling are also removed by washing.
- Containers can be cut up to approximately 1 cm pieces with hand shears, heavy duty paper cutter, or with a mechanical granulator if preferred.
- A blend of 10 wt% barrier package is hand blended into 90% virgin PET control resin – so for example 50 grams of test bottle is mixed with 450 grams of virgin control resin.
- The blend is dried and injection molded into 3 mm thick amorphous plaques.
- The color and haze values of the plaques are measured in transmission and reported as delta values to 100% control resin with no scavenger sample included.

Since most PET packages weigh between 10 and 50 grams and 500 grams of blend material can be more than sufficient for testing, only a few individual packages are required to conduct this test. The Appendix contains information that can allow investigators to estimate the impact of using a barrier material at higher concentrations such as 25 or 50% as employed in a Critical Guidance test.This assessment calls for evaluation of containers that have been aged because bottle age can impact color when active barrier technologies are employed. An assessment of the impact of package age on color and haze formation is

recommended as a component of this evaluation.

Reference Documents

- PET-P-00 PET Standard Laboratory Processing Practices
- PET-P-12 Preparation of PET Articles with Potential Time Dependent Behavior
- PET-B-01 Benchmark Test for Clear PET Resin and Molded Articles
- PET-CG-01 Critical Guidance Protocol for Clear PET Resin and Molded Articles
- PET-S-10 PET Flake Oven Bake Test

Equipment Required

- A plastic granulator or heavy hand or table shears for cutting up barrier packaging samples.
- A hot air oven or desiccant drier to dry samples and provide oven heat aging.
- Injection molding equipment for producing 3 mm thick amorphous plaques
- Color spectrophotometer that can measure color and haze of molded plaques in transmission.
- For test materials where separation and liberation of a barrier material might be impacted by flake wash and/or elutriation..... equipment for washing and elutriation as described in PET-P-00 Practices.

Materials Required

- PET containers that contain scavenger/barrier materials of interest
- A pelletized virgin PET control resin for comparison. The control resin might be a named PET control resin, or a PET resin that has been otherwise qualified as described in PET-P-00.

Test Method Steps

For samples that do not require or benefit from flake washing or elutriation:

- Obtain samples of barrier packaging to test.
    - At least 50 grams of test material are required for molding when molding a 500 gram total weight sample.
    - It is recommended to secure extra containers as retains to be used for oven bake testing or follow-up tests if desired.
- Clean off any labels and adhesives. Hot water with dish soap can remove some

adhesives. Mineral spirits or other common laboratory solvents will remove adhesives commonly used for labels.
- Wash the containers thoroughly with hot soapy water to clean the inside and outside surfaces of the container.
- Either cut up each test sample into approximately 1 cm square pieces, or granulate each sample separately, and place the resulting material into a labeled container.

For test samples that benefit from exposure to a flake wash and/or an elutriation step to help liberate and separate barrier material from PET:

- Secure the number of bottles that will result in at least 500 grams of granulated material for each sample of interest. Best practice is to use the product of complete bottles when making a blend with virgin resin. For example, let's say in individual bottle weighs 30 grams; clean and cut up two bottles and combine the 60 grams of bottle with 540 grams of virgin resin to make the 10% blend. This approach insures that the impact, or absence, of barrier material in the finish section of the bottle and base of the bottle is adequately represented in the test.
- Granulate each sample set separately with reference to PET-P-03, and place granulated material in a labeled container.
- Granulate a sample of PET bottles that do not contain any barrier material. About 1 kg of granulated flake is necessary. This bottle flake will be used to establish the air flow rate in the elutriator.
- Wash, dry and elutriate the granulated samples with reference to the procedure in PET-B-01 as well as the PET Practices PET-P-04 and -05.
- Place washed and elutriated material into labeled containers.

For all samples:

- Create a 10 wt% blend of test material with virgin control resin; for example, blend 50 grams of test material with 450 grams of control PET pellets.
- Dry a sample of control resin only, as well as 10% blends in a desiccant drier or a hot air oven for 6 hours at 160° C.
- Injection mold the dried samples with reference to PET-P-08.
- Evaluate the color of the molded plaques following PET-S-09

Some optional steps that investigators might take:

- Heat a test bottle, and/or granulated flake, in an air oven following PET-S-10.
- Save a sample of test bottle flake stored at room temperature and open to the atmosphere, and re-test after at least 90 days to evaluate for an aging

effect; this is referred to as a "coarse aging" evaluation in the Appendix. See PET-P-12 for APR's standard aging protocol.
- Measure the oxygen barrier performance of the packages prior to testing to confirm that packages provide the expected barrier performance.
- Create 25 or 50 wt% blends to investigate the impact of concentration of test material.

Measurements, Reports and Guidance Values

This information will be reported

- A description of each sample in the test that provides as much information as possible. Suggested description content: scavenger employed, additive concentration, target shelf life of the package, bottle resin employed, age of the bottle tested, aging conditions, container description.
- The general plaque injection molding conditions will be recorded to allow judgements to be made concerning any data developed with different molding conditions or developed at different molding locations. Information recorded will include: barrel temperature profile, diameter of injection screw, volume of injection mold, cycle time, barrel volume as number of shots in the barrel to give a measure of residence time in the barrel.
- The transmission color values and % haze of molded 3 mm thick plaques measured in transmission.
- The absolute color values of the control plaques will be reported.
- Color values will be reported as a delta to the control resin with no barrier content.

Guidance given in the method

All guidance below is qualitative and directional in nature:

- Provide at least one coarse aging result for a given scavenger candidate to gage any impact of container age on b* values.
- In ranking a set of samples, samples with the lowest delta b* value will be the better candidates for least impact on PET recycling.
- Samples that have a low b* value approaching that of the control PET resin are candidates for more rigorous evaluation using PET-B-01 or PET-CG-01.
- The L, a* and % haze values are not expected to reveal any remarkable differences in samples in this test. Should there be a sample with an L, a* or % haze value that stands out from others in the sample set that sample

can be subject for more detailed evaluation.

Safety Statement: *APR Test and Practice documents do NOT CLAIM TO ADDRESS ALL OF THE SAFETY ISSUES, IF ANY, ASSOCIATED WITH THEIR USE. These Tests and Practices may require the use of electrically powered equipment, heated equipment and molten polymers, rotating motors and drive assemblies, hydraulic powered equipment, high pressure air, and laboratory chemicals. IT IS THE RESPONSIBILITY OF THE USER TO ESTABLISH AND FOLLOW APPROPRIATE SAFETY AND HEALTH PROCEDURES WHEN UNDERTAKING THESE TESTS AND PRACTICES THAT COMPLY WITH APPLICABLE FEDERAL, STATE AND LOCAL REGULATORY REQUIREMENTS. APR and its members accept no responsibility for any harm or damages arising from the use of or reliance of these Tests and Practice documents by any party.*

Disclaimer: *This document has been prepared by the Association of Plastic Recyclers as a service to the plastic industry to promote the most efficient use of the North American plastic recycling infrastructure and to enhance the quality and quantity of recycled postconsumer plastic. The information in this document is offered without warranty of any kind, either expressed or implied, including WARRANTIES OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE, which are expressly disclaimed. APR and its members accept no responsibility for any harm or damages arising from the use of or reliance upon this information by any party. Participation in the Recognition Program is purely voluntary and does not guarantee compliance with any U.S. law or regulation or that a package or plastic article incorporating the innovation is recyclable or will be recycled.*

DOCUMENT VERSION HISTORY

Version	Publication Date	Revision notes
1	November 12, 2024	Original Publication

Appendix

Background information to employ before conducting any testing with this method

This screening test was developed to encourage testing and ranking of the color impact of barrier materials on PET recycling. It purposely allows the use of a low amount of test material so that the cost and complexity of molding, storing, and handling large numbers of containers is avoided. The test was developed to be conducted in any laboratory with common plastic processing equipment. Individual investigators can use the method for a wide variety of uses, or buyers and sellers of barrier containers can use the method to evaluate a range of end use situations.

Investigators are encouraged to consider the bullet points below before embarking on a testing program with this method. These bullet points reflect experience and insights from the several individuals who were instrumental in drafting this test method.

- Any color generated by barrier materials can be a function of the concentration of the barrier material in the sample or may result from a difference in the formulation of the barrier package. When comparing different barrier samples, it will be a benefit to know the specific barrier content, targeted package shelf life and materials used when possible.
- Package dimensions such as side-wall thickness or container surface area can impact color development. When making comparisons, it can be valuable to record the dimensions of the various packages evaluated. Best comparisons will result from testing different barrier materials in the same container format.
- This test allows for comparing any one package to another. But keep in mind that different packages can employ different barrier levels and be designed for different end use applications. Best comparisons will be made for package and barrier combinations intended for a focused end use and shelf-life requirement such as, for illustration:
    - 16 ounce PET beverages with 6 month shelf life requirement.
    - 10 ounce PET beverages with a one year shelf life requirement
    - A PET thermoformed package providing a 3 month shelf life.
- Package age can impact color development. Oxygen scavengers perform by reacting with oxygen; experience shows that the age of containers can impact color development with older

containers developing more color than fresh ones. This method recommends investigators confirm any impact of age of container on color formation.
    - The method suggests a "coarse aging" study where containers recently made are compared to others that have been in storage or use for at least 90 days – just a coarse comparison of "fresh" to "aged" without concern for the details of the aging period or conditions. Use of this coarse aging approach is encouraged because in comparing various barrier options, it will sometimes to be difficult to obtain samples that are all the same age and aged under similar conditions! And a coarse aging comparison is adequate to reveal the approximate impact that aging might have.
    - APR does offer an aging protocol that can be used for more controlled comparisons of any impact of aging – PET-P-12.
- It can be valuable to pair a color evaluation test to one that evaluates the amount of oxygen that has been consumed by a given active barrier material. This step will help confirm that samples tested for color formation were also effective at providing the required barrier protection.
- Heat exposure can impact comparisons of barrier materials. So, it is important to take steps to control heat exposure of samples in the laboratory. Close control of drying temperatures and times, as well as injection molding conditions will benefit results. Should data developed at different laboratories be compared, there may be some variation between laboratories because of the details of drying and molding conditions employed.
- The 10 wt% test material in virgin PET control resin was selected with these objectives in mind:
    - To allow for a low amount of test material. Availability of large numbers of containers can often be a cost and time barrier to conducting testing.
    - A 10% blend of bottle flake with virgin pellets is generally easy to mold in a laboratory injection molding machine – higher flake contents may make feeding the injection unit consistently more difficult.
    - Virgin PET control resins listed in the PET-P-00 Practices are commonly available.
    - Allow for comparison of the widest variety of testing variables. Injection molded plaques can be made with low cost in many labs and so allows for a wide variety of materials and package designs to be evaluated.
- When molding 10% samples, the color that is measured is roughly linear with concentration. This means that a b* color value of "X" in a 10% molded plaque can be estimated to be 5X in a plaque made with 50% content; but this is only an estimate, not a certain outcome!
- It is common practice to look for factors that impact color of recycled PET by heating either a complete package, or granulated packages, in an air oven for one hour at 220° C. (PET-S-10).

Investigators are encouraged to use this bake evaluation to compare color formation in samples before injection molding, and this oven test might narrow the number that are tested by injection molding.

- This test anticipates that finished packages will be employed in testing. But there may be cases where it is desirable to test a preform or a sheet before it is formed into a finished container. This may be done providing comparisons are only made with like samples! It is not appropriate to compare the results obtained with a preform to those obtained from a finished container because of the substantial differences in surface area and cross-sectional thickness exposed.