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PET Standard Laboratory Processing Practices

Introduction

This document presents Standard Laboratory Practices recommended by the APR to prepare PET articles for use in tests that evaluate the compatibility of the article with commercial PET recycling processes. These practices are referenced and employed in various APR Test Methods and Guidance Protocols.

The general term PET article is used so that these practices can apply to any molded part made from PET. In practice, molded articles are expected to be packaging materials such as injection stretch blow molded bottles, extrusion blow molded bottles and thermoformed PET sheet stock. Testing can be employed with a PET molded article alone, or to evaluate the impact of design features such as closures, labels, or additives, for example, on recycling. These practices can also be used to evaluate PET virgin resins in the form of pellets.

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Selection of Control Resin, PET-P-01

Background

Recycle testing often involves comparing the performance of a plastic article containing a design feature or innovation to that of a control article that does not employ the design feature. This practice provides the steps used to select a control virgin resin and produce articles suitable for control.

A control resin can be:

1. Selected from a list of named commercial virgin PET resins.
2. One that is qualified by testing.

APR Named list

Low IV, Water Bottle Control Resins	CSD and Non-Water Bottle Control Resins
Auriga Polyclear® Splash 3301	Auriga Polyclear® Refresh 1101
DAK Laser+® W L40A	DAK Laser+® B90A
APG Polytech Turbo II	APG Polytech ClearTuf Max

These named resins are recommended for any laboratory evaluations that require a control resin.

Control resin selection criteria:

- They are widely available and are well known commercial resins, especially in North America.
- They represent resin compositions that are widely used commercially.
- These resins are expected to be among the resins with higher L values, and lower b* and haze values in their given applications.
- These resins demonstrate solid stating rates within industry norms.
- These resins will have a low increase in b* value resulting from multiple drying and melt heat histories.

European PET Bottle Platform's Named list

The EPBP maintains a listing like APR 's named control commercial PET resins featuring resins that are known in the European region. These resins are known to have low color, high clarity and typical solid state IV build characteristics. APR will accept those resins listed by EPBP for use with Critical Guidance Petitions.

Qualification of a control resin not from the named list

In the case of evaluations involving closures, attachments, lidding or labels, PET-S-03 (PET Heat History and Discoloration Test), can be used to qualify the control resin. The Color Formation Test will confirm that the PET resin used for control meets these CIELAB color requirements after three drying/melt/mold heat histories:

- L value greater than 82
- b* less than 4
- Haze less than 9.5%

In the case of Critical Guidance evaluations involving resins or additives, the control resin must also be shown to achieve a solid state IV build rate over an eight hour solid stating period of at least 0.012 dl/g·hr.

Control articles

The control resin is typically used to make molded control articles that are used in testing. The general term, article, is used to reflect that testing can be used for any PET molded product. These control articles are usually molded containers that reflect the expected use of the design feature being evaluated such as:

- Bottles or containers made by injection stretch blow molding
- Containers made by injection molding, or injection blow molding
- Bottles or containers made by extrusion blow molding
- Containers made by thermoforming sheet

Preparation of PET Articles for Evaluation, PET-P-02

Background

Recycle testing often involves comparing the recycle performance of an article that employs a design feature of interest to that of a control article that does not contain the design feature.

This practice lists the steps taken to prepare a test article for evaluation of these design features:

- Containers with attached labels or direct printed labels.
- Closures – including seals and dispensers.
- Attachments such as handles, or other feature incorporated with an article that is not a closure or label.

Evaluations can involve the “intended” design feature or a “generic” feature.

- An "intended" article is prepared as it will be sold in the market using the specific and intended materials and design that will be marketed.
- A "generic" article is one used in the laboratory to represent a variety of market products and represents an average of what will be commercialized. In the case of labels, to determine these averages, a range of articles in the market were evaluated for weight and label coverage to determine and average % by weight or % area coverage.

As an example, one might:

- Test a specific label with specific inks, graphics and a specific surface area of coverage that will be used on a commercial product; an intended label
- Test a generic label that is printed with several representative ink colors that represents a range of label graphics that might be used. The generic label is evaluated at a specified area of coverage or weight percent in PET.

Control articles

The control resin is used to make control articles that are used in testing. These control articles are usually molded containers that reflect the expected use of the design feature being evaluated such as containers made by:

- Injection stretch blow molding
- injection molding, or injection blow molding
- Extrusion blow molding
- Thermoforming sheet

Control Article Preparation

- For evaluations that involve design features such as resins, additives, layers, or coatings, the use of a listed control resin is recommended for efficiency and to ensure consistency when molding a control article; however, the control article can be made with an unlisted control resin that has been qualified by testing. When these design features are being tested, it is not necessary to include labels or closures on the containers when closures and labels are not associated with the design feature of interest.

- When labels, closures or attachments are being evaluated, a listed control resin can be used, or an unlisted resin can be qualified by testing. The unlisted but alternate qualified resin may be used for both control article and test article if desired.

Preparation of articles when closures or attachments are used

The intended design feature is applied to a container and test articles are blended with control articles at a 50:50 mix.

Preparation of containers for label testing

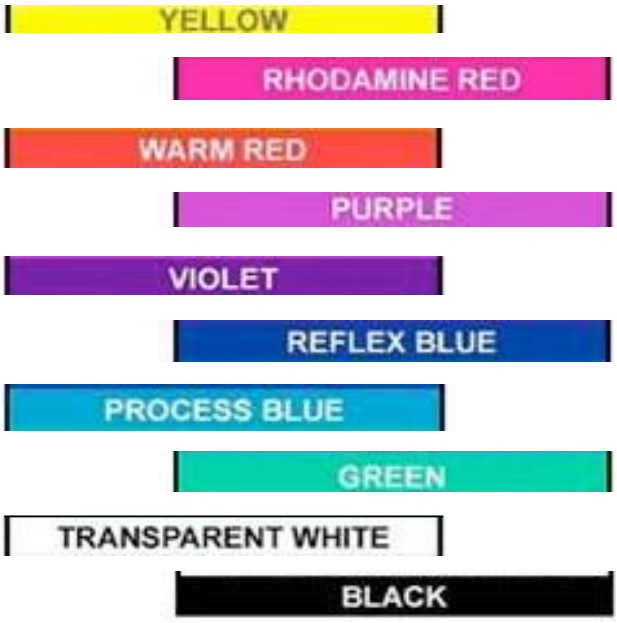
Labels can be tested printed or un-printed; with intended inks and graphics or with generic inks. Test containers may made with either a listed control resin, for efficiency and consistency, or an alternate qualified resin. The same resin is to be used for both Control and Test containers. Test containers with labels at the indicated levels will be compared to Control containers without labels. The table below presents options to select the mixture of label material and PET resin to be employed in the evaluation:

Label style	Intended labels	Generic labels
Pressure sensitive labels	Test intended labels on PET containers blended with un-labeled control containers 50:50.	Test labeled bottles with 40% surface area coverage blended with un-labeled control containers 50:50. Or, employ 20% surface coverage on all test containers.
Shrink and stretch sleeve labels	Test intended labels on PET containers and blend with un-labeled control containers 50:50.	Test labels at 3 wt% of all test containers, no 50/50 dilution with control bottles required.
Direct print labels	Test intended labels on PET containers and blend with un-labeled control bottles 50:50.	Test labeled bottles with 40% surface area coverage blended with un-labeled control containers 50:50. Or, employ 20% surface coverage on all test containers.
Wrap labels	Test intended wrap labels on PET containers blended with un-labeled control containers 50:50.	Test wrap labels at 0.6 wt% of all test containers, no 50/50 dilution with control bottles required.
ROSO labels	Test intended labels on PET containers and blend with un-labeled control containers 50:50.	No generic APR Guidance for ROSO labels today.

When using generic labels, the following images illustrate ink pattern options that may be used:

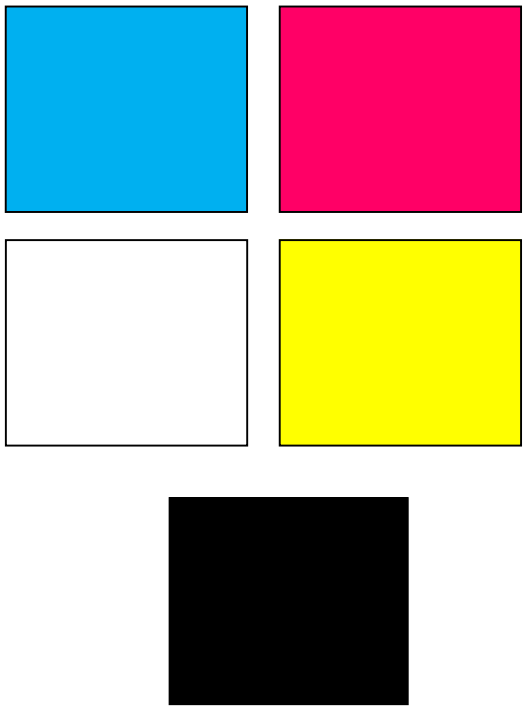
Option 1 Pantone– Clear Articles
(Multiple Labels with flood coat on each)

Option 2 Pantone– Clear Articles (One Label, Multiple Colors)



Option 3 w/4 Color – Clear Articles
(One Label Multiple Colors)

Option 4 w/4 Color – Clear Article
(Multiple Labels with flood coat on each)



Granulating PET Articles to Flake, PET-P-03

Background

PET articles are reduced in size by granulating (grinding) articles in a rotary granulator. This step creates PET flake that is used in subsequent process steps. Granulating is also intended to liberate package components such as labels, closures, dispensers and attachments from the PET container.

Equipment Required

- Weigh scale (0.01 ± grams)
- Rotary plastic granulator fit with a screen containing holes within the range of 9.5 to 12 mm. The machine is to be evacuated via gravity (without pneumatic transport)
- Compressed air line and/or shop vacuum cleaner to use in cleaning the granulator

Materials Required

- Control Article
- Test Article
- Soft cloths for cleaning the granulator
- Containers such as plastic pails or bags to hold granulated flake samples

Practice Steps

Test and control articles are each ground separately. The granulator is cleaned before and after granulating each sample:

1. Clean the plastic granulator prior to use with compressed air and/or a shop vacuum. Wipe up any fines or other contamination with a clean cloth, if necessary. Be aware of material hang-up inside the granulator behind the cutting head which may be difficult to reach and inspect. Do not leave fragments of the cleaning cloth in the machine.
2. Observe all safety practices relevant to the machine, including lock-out procedures.
3. Before grinding, retain five (5) samples each of the Control and Test articles.
4. Check to ensure the granulator screen is properly installed with the proper diameter holes.
5. Weigh the required number of Test and Control articles to provide the desired weight of granulate for each as specified in the test to be conducted
6. Granulate a given sample by manually feeding the article into the granulator.
    a. Granulated material may include PET flakes, label pieces, and closure pieces.
7. Store each sample in a sealed and labeled container.
8. Retain 50-100-gram samples of each ground sample.
9. Clean the granulator between each sample.

Washing and Sink/Float Separation of PET Flake, PET-P-04

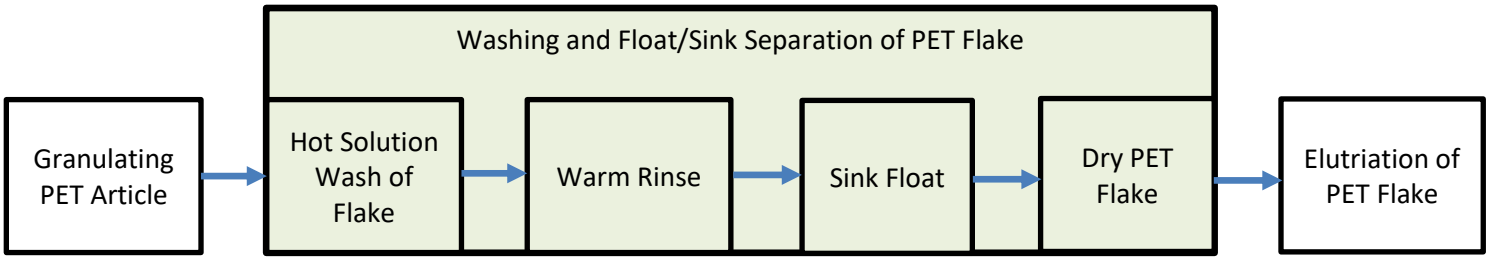
Background

After granulation, PET flake is washed in a hot aqueous caustic, detergent solution. The wash step is intended to remove contamination from PET and can contribute to liberating labels, adhesives, coatings and layers from PET flake.

Practice summary and illustration

Granulated PET is washed in a hot aqueous caustic solution with a high level of agitation. Washed flake is then rinsed in warm water. Rinsed flake is exposed to a final float/sink step in tap water. Separation of any floating materials that are free to float in water are removed at each of the 3 steps. Washed flake is then dried.

An investigator may want to conduct a materials balance of the flake washing process. A Materials Balance Evaluation, PET-S-04, can be used for that purpose.



Equipment required

- Stainless steel mixing tank with the following specifications:
  - Sized to allow water and PET flake to be mixed at a 4:1 ratio by weight.
  - Will allow a recommended ratio of the water height-to-the tank width of 0.8 to 1.0 and not less than 0.6 or greater than 1.5.
  - Fitted with a mixer with an impellor blade length at least 0.33 of the mixing tank diameter or width, and with variable speed capability for impeller.
  - Incorporating at least three baffles when a round tank is used; baffles not necessary in a square tank
- Hot plate, or a similar method of heating the mixing tank
- Immersible electronic thermometer
- A de-watering screen – this can be a wire mesh screen or a perforated metal surface with openings greater than 1 mm in size.
- A container to serve as a float/sink tank sized to hold room temperature water and flake at a 8:1 to no more than 10:1 by weight mix ratio.
- Lab oven and trays to dry samples

Materials and Reagents Required

- Materials for APR detergent wash solution
  - Triton X-100 nonionic surfactant
  - Sodium hydroxide, NaOH, granules or solution
- Optional alternative wash detergent
  - MacDermid RP24
  - Sodium hydroxide, NaOH, granules or solution
- Granulated flake from control article
- Granulated flake from test article

Practice Steps

Weigh the amount of PET flake required for the evaluation as specified in the test or Critical Guidance Protocol.

Hot Wash

1. Prepare the wash solution
   a. Add tap water to the stainless wash vessel. The weight of water to be four times the weight of PET flake planned for use in wash, for example, 2000g (2000 ml) water, 500 g of granulated PET flake.
   b. Place the container on the hot plate or heat source.
   c. Position the impellor between 0.5 to 1 impellor diameter from the bottom of the mix container.
   d. While the water is heating, add NaOH granules or concentrate solution to the water with agitation to create a 1% by weight content of NaOH.
   e. Add surfactant to the tap water to create a 0.3% by weight content of surfactant. (If the alternative MacDermid RP24 detergent is used, employ the RP24 at 0.3 wt% in a 1 wt% solution of NaOH.)
2. When the wash solution reaches 85° C, add the PET flake maintain wash solution temperature at 85° C.
3. Increase the agitator speed to achieve an impellor tip speed of at least 240 meters per minute and agitate for 15 minutes.
4. After 15 minutes, shut off the agitator impellor; remove the wash solution container from the heat source, and/or turn off the heat source.
5. Allow the container to sit for five minutes.
6. Skim off and save any materials floating on top of the surface.
7. Pour the liquid-flake mix over the de-watering screen and save the wash water.
8. Vigorously stir the retained wash water while taking a sample – about 1 liter in volume – for later evaluation.

Warm Rinse

1. Prepare the rinse container
   a. Fill a clean stainless container with tap water with 4x the weight of PET flake, place the container on the hot plate or heat source.
   b. Position the impeller 0.5 to 1 impeller diameter from the bottom of the container.

2. When the rinse water reaches 45° C, add the PET flake and maintain the rinse water temperature at 45° C
3. Increase the agitator speed to achieve a tip speed of at least 240 meters per minute and agitate for 5 minutes.
4. After 5 minutes, shut off the agitator impellor; remove the rinse container from the heat source, and/or turn off the heat source.
5. Allow the container to sit for five minutes.
6. Skim off and save any materials floating on top of the surface.
7. Pour the liquid-flake mix over the de-water screen to recover the PET flake as well as any sinking solids contained in the rinse water.

Sink Float

1. Fill a clean stainless container with tap water; employ at least 8x the weight of PET flake, and no more than 10x the weight of PET flake.
2. Add the PET flake to the container and agitate for 5 minutes. Agitation may be done by hand or with mild agitation from a mixer.
3. Allow the container to sit for five minutes.
4. Skim off and save any materials floating on top of the surface.
5. Pour the liquid-flake mix over the de-water screen to recover the PET flake as well as any sinking solids contained in the rinse water.

Dry recovered materials

1. Dry PET flake so that it is dry to the touch and free flowing. The flake can be dried in air, or in a lab oven where the flake temperature while drying does not exceed 60° C to prevent oxidation of any material in/on the flake.
2. Retain a 50-100-gram sample of the flake resulting from this practice step.

Elutriation of PET Flake, PET-P-05

Background

In the industrial PET recycling process, after flake is washed and dried it is common to conduct an air separation step known as elutriation to remove fines, small particles and light material from the relatively heavy PET flake. This step may remove label residue, dried inks, or layers, for example. This document presents standard practice to elutriate PET flake post wash.

Equipment Required

- Weigh scale (0.01 ± grams)
- Lab scale elutriator with the ability to adjust air velocity (typical < 8 m/s)
- Compressed air line and/or shop vacuum cleaner to use in cleaning the elutriator

Materials and Reagent Required

- Shop rags for cleaning the elutriator
- Containers such as plastic pails or bags to hold flake samples pre-and post-processing
- Washed and dried flake produced from the control article
- Washed and dried flake produced from the test article

Practice Steps

1. Weigh the amount of flake that will be elutriated.
2. Clean the elutriator unit. Use compressed air and/or shop vacuum. Wipe up any fines or other contamination with a clean shop rag if necessary.
3. Using the washed control flake, establish the required air velocity to remove no more than 0.5% by weight of the control article material when passed through the elutriator
4. Process all test flake samples in batches at the established air velocity.
5. Clean the elutriator between each control or test sample batch.
6. Weigh the amount of material removed and the remaining flake to calculate the total yield and yield loss % for each sample.
7. Retain each sample in a sealed and labeled container.
8. Retain 50-100-grams of each flake sample after elutriation
9. Retain additional final flake samples for IV measurement. Additional samples can be saved if desired for other measurements such as color, clumping or bulk density (approximately 100 grams each)

Elutriation Yield Data

Control Article Flake

Processing Practice Step	Article Weight (g)	Yield (%)
Starting Weight		
Recovered Flake		
Total Lights Removed		
Total Weight		

Test Article Flake

Processing Practice Step	Article Weight (g)	Yield (%)
Starting Weight		
Recovered Flake		
Total Lights Removed		
Total Weight		

Melt filtration, Pelletization and Crystallization, PET-P-06

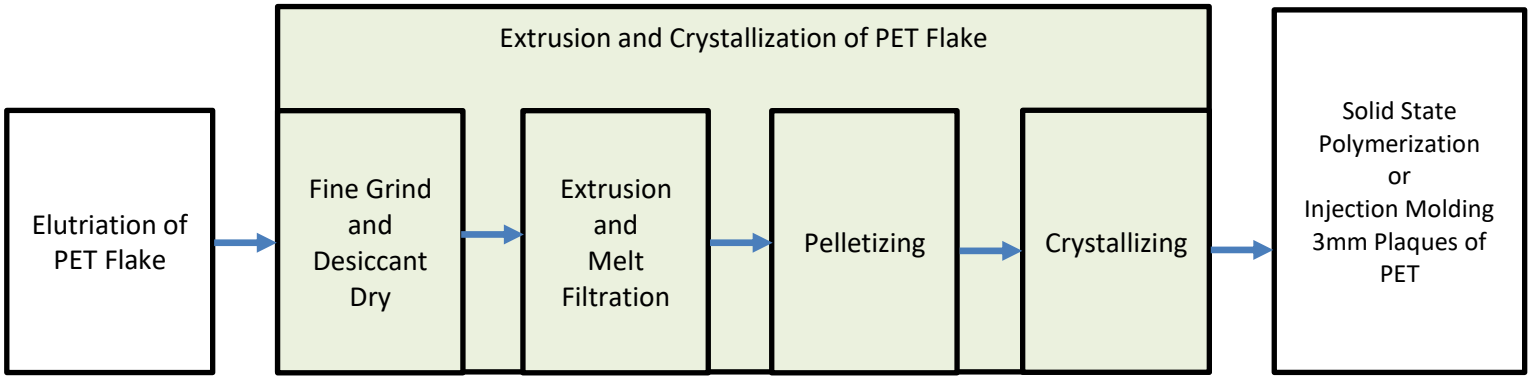
Background

In the industrial PET recycling process, after elutriation, it is common to melt the PET flake in an extruder so that the melted polymer can be melt filtered and so that the flake can be converted to pellets. This document presents a standard practice to extrude and create strand cut PET pellets from blended PET flakes.

The practice includes the steps of finer grinding of flake, drying, extrusion, pellet forming and crystallization. At the same time, it defines required observations and samples for further testing.

Practice summary and illustration

Elutriated flake is blended as required for a specific test method or Critical Guidance Protocol, and then dried in a desiccant drier. Dried flake is melted and filtered in an extruder to create strand cut pellets. Melt pressure is recorded ahead of the melt filter. Amorphous pellets can be crystalized for use in a next step when necessary



Note: There are situations in laboratory recycling assessments where it is necessary to add a heat history to PET resin to simulate a molding operation. This Extrusion Practice can be followed to add a heat history to PET samples – flake or pellets. Melt filtration and pressure measurement is not necessary when the practice is used only to add a heat history.

Equipment required

- Desiccant or vacuum dryer capable of achieving a moisture content of less than 50 ppm in PET dried in the unit at 160° C. A desiccant dryer air stream should have a dew point below -20°C.
- An extruder suitable for PET flake processing in the laboratory.
    - Usually a 25 to 35 mm extruder with a 24:1 to 36:1 L/D is suitable for laboratory use. A twin-screw extruder may also be used
    - Melt residence time in the extruder should be no more than 6 minutes.
    - The extruder requires a means for maintaining dried PET flake in a dry state while in the hopper and during extrusion.

  - The extruder is equipped with a breaker plate and screen pack. Extruder must be capable of supplying 0.38 kg/cm^{2}·hr through the screen pack.
  - The extruder employs a calibrated pressure transducer ahead of the screen and employs a data logger to record pressure values.
  - The extruder requires a strand die, water bath and pelletizer.
- A scale for measuring extruder output.

Materials required

- Containers such as plastic pails or bags to hold final pellet samples
- Screen pack for the extruder - 40/250/40 mesh
- Elutriated control flake, elutriated test flake, and/or blends of flake.

Practice steps

1. Crystallization of flake – if flake is largely amorphous material, it will necessary to crystallize the flake prior to the drying step below. The crystallization steps used for pellets below may also be used when it is necessary to crystallize a flake sample.
2. Finer Grinding – The flake size used for washing and elutriation will generally will not feed well on a small laboratory extruder. Finer grinding of flake is acceptable and may be required to allow good feeding into the extruder feed throat and to achieve steady pressure readings ahead of the screen pack. Each sample will be dry ground separately using a standard mechanical grinder, using a screen between 4 and 6.5 mm. It is important that the machine be cleaned well and all the material be captured.
3. Drying Step
    a. Each sample will be dried directly before extrusion to a moisture of less than 50 ppm in a hopper using dry hot air from a desiccant unit. The air temperature must remain between 150 and 160°C. Typical drying time at 160°C is 4-6 hours. The same conditions are used for all samples.
    b. Alternatively, a vacuum dryer, operating between 150 and 160°C for the hours necessary to achieve less than 50 ppm moisture in the PET pellet may be used.
    c. Make provision to ensure the resin is kept dry during extrusion.
    d. Inspect the drying hopper after emptying for sticking flake or contamination on the sides of the hopper. Inspect the hot PET flake for the presence of any unusual fumes or odors associated with the hot flake.
    e. Record any occurrence of sticking flake, residues, fumes or odor that occurred drying the resin drying step.
4. Extrusion Steps
    a. Extrude the control article flakes first at a target melt temperature of 280° C.
        i. The melt will be filtered with a clean 40/250/40 mesh melt filter (about 60 microns filtration)
        ii. The flow rate through the filter should be at least 0.38 kg/cm^{2}·hr of mesh area.

iii. The melt is extruded through a die into strands of approx. 2.5mm diameter. The strands are rapidly cooled in water and fed into a pelletizer to form amorphous pellets. It is important to achieve similar pellet sizes for each of the test and control materials for use in solid state IV build evaluations. A pellet weight of 1.6 to 2.0 g/100 pellets is suggested. Screening may be used to remove over and under sized pellets.

b. Start run timer
c. Record pressure values ahead of the screen pack for a 30 minute's run time.
d. Record any occurrence of unusual fumes, odor or build-up occurring at the feed throat or die exit of the extruder. Observe for any hazards associated with the innovation material.
e. Save a sample of extruded pellets for IV measurement. Additional samples can be retained for color and other measurements as desired.
f. Retain a 50-100-gram sample from this practice step.
g. Purge the extruder and change the screen pack between each run.
h. Store the pelletized samples in pails or bags
i. Extrude all subsequent samples at the same conditions; achieve pellets of similar size for each sample set when the pellets will be used for a solid stating evaluation.

5. Pellet Crystallization

If an injection molding test is to be conducted directly from amorphous pellets it will be necessary to crystallize them before they can be dried and used for injection molding. If pellets are intended solely for a solid stating test, some investigators may choose to crystallize the pellets prior to solid stating. Some solid stating processes can accommodate amorphous pellets; in this case there is no need for crystallization of the amorphous pellets. Crystallization involves these steps:

a. Each sample is homogeneously crystallized to obtain single, non-sticking PET pellets.
   Typical crystallization conditions are 1 hour in a pre-heated oven at 160°C.
   Alternatively crystallization can be performed in a fluid bed crystallizer for 20 minutes with heated air at 175°C. (pellet temperature must be >140°C for >10 minutes but remain <170°C).
   The same conditions are used for all samples.
b. After cooling break up any agglomerates to obtain single pellets.
c. Store crystallized pellets in a sealed container or under dry conditions to maintain their moisture content < 2500ppm.
d. Record any occurrence of unusual sticking during crystallization.
e. Retain a 50-100-gram sample from this practice step.

Steps Required to Assess Pressure Values

For the test material only:

Calculate the initial average pressure, P_i, over the first five minutes of extrusion as well as the average pressure value for the final five minutes of extrusion, P_f. When:

$(P_f – P,) / P_i$ is equal to or greater than 25%, this is taken as evidence for screen pack build-up.

Solid State Polymerization of PET Pellets, PET-P-07

Background

After pelletization, it is common in commercial practices to build the molecular weight of PET by using a process known as solid state polymerization (SSP). Molecular weight of PET is often reported using a measure called intrinsic viscosity, IV. This document presents a standard practice to raise IV of PET through exposure of pellets to heat and vacuum employing a rotary vacuum unit, a unit widely used in commercial practice. A laboratory scale rotary vacuum unit is commonly used to create quantity of 0.80 dl/g IV control and test blend flake required for the PET Resin and Additives Critical Guidance Protocol.

The Critical Guidance Protocol also calls for an evaluation of any impact of an innovation resin or additive on the solid stating build rate. The PET IV Build Rate Evaluation, PET-S-07, is used for this determination. The rotary vacuum unit described below can be used to create the solid stating build rate samples as well, but other commonly used methods for solid stating PET may be used as well.

Practice Summary

To build the IV of PET pellets, pellets are batch processed through a rotary vacuum solid state reactor controlling time, temperature and vacuum level. Solid stating rate is often diffusion rate limited – that is limited by the rate that water can diffuse out of the PET pellets. Controlling the PET pellet size and starting point moisture will influence the solid stating rate. During solid stating, control of temperature and vacuum (to the extent that it impacts removal of volatiles in the reactor) are important in achieving best results.

Solid stating must be conducted with crystallized PET to prevent fusion of amorphous pellets. Some investigators choose to crystallize in a separate step prior to solid stating. Others may choose to solid state within the rotary vacuum unit. Either approach is acceptable. This Practice anticipates crystallization in the rotary reactor.

Equipment Required

- Oil Heater and thermostat able to control PET stock temperature to 210^{o} C in vacuum sealed stainless steel vessel
- Vacuum pump
- Sample port capable of removing approximately 10 grams of pellets from the vessel without breaking vacuum

Materials and Reagent Required

- Dried glass jars and metal lids for use in sampling the solid stated resin throughout the test run
- Amorphous pellets from the control article
- Amorphous pellets made with blends of control resin with the test article.

Practice Steps

Solid stating steps

1. Prepare the amorphous pellet blends required/desired for testing. Steps to address:
    a. Screen the pellets to remove under and over-sized pellets that can impact results.
    b. Condition pellet samples in a standard environment so that the starting moisture content of pellets is similar from one evaluation to the next.
    c. Amorphous pellets may be crystallized prior to use or may be crystallized in the solid state reactor as desired by the evaluator.
2. Add a given individual pellet sample to the rotary vessel at room temperature. Insure the total weight is sufficient for the injection molded plaque or the performs to be produced in the appropriate tests. If pellets have been previously crystalized, step 2 can be skipped. Crystallization process:
    a. Start rotation of a rotary vessel to avoid PET pellets from sticking and clumping
    b. Start vacuum
    c. Raise temperature of heating oil as required to have pellet temperature reach 160-170°C
    d. Pellets must be at 160-170°C for 1 hour prior to raising temperature for SSP processing
3. Raise temperature of oil as required to have the PET temperature reach 210 ± 5 °C and keep the temperature at a constant.
4. Keep the vacuum and vessel rotation processing.
5. SSP time starts when article temperature reaches 190°C
6. Run the solid state reactor for the desired/required time increment(s).
7. When shutting the reactor down, do not break vacuum or stop rotation of vessel until after article reaches less than PET solid stating temperatures. (recommendation is less than 160°C)
8. Retain 50-100gram sample for further evaluations.
9. Note any unusual sticking behavior or unusual build-up within SSP vessel.
10. The reactor is typically fitted with a sample port so that the PET pellets can be sampled from time to time to monitor IV values.

Injection Molding 3mm Plaques, PET-P-08

Background

This document presents standard practice to Injection mold either dry flake or crystallized pellets into 3mm thick amorphous transparent plaques for evaluation of color, haze and presence of black specks and inclusions.

Practice Summary

Flake and/or pellet are dried and injection molded into 3mm thick plaques for evaluation for color, haze and content of specks and inclusions.

Equipment Required

- Desiccant or vacuum dryer
- Lab Scale injection molding machine allowing no more than 6 minutes of injection barrel residence time.
- 3mm thick plaque mold with nominal dimensions of 50x50 mm, or greater.
- Chiller for mold
- Moisture tester capable of evaluating < 50ppm water content in PET pellets

Materials and Reagent Required

- Flake, crystallized pellet and/or solid stated pellet made from control material
- Flake, crystallized pellet and/or solid stated pellet made from test material.

Practice Steps

Note that flake made with largely amorphous PET will benefit from being crystallized prior to use. Larger size flake may not feed well in a laboratory scale injection molding machine. A second granulation step for crystallized flake using a 4 to 6.5 mm screen can facilitate feeding the injection unit.

1. Weigh the required amount of flake or pellet for processing the number of plaques required for each control and test material.
2. Dry the flake or pellet in a desiccant or vacuum dryer capable of achieving a moisture content of less than 50 ppm for PET flake and/or pellet dried in the unit at 160° C.
3. Starting with the control material, prepare the injection machine to provide:
    a. Target melt temperature at nozzle to be 275°C
    b. Target mold temperature 20°C
    c. No more than 6 minutes barrel residence time.
4. Injection mold the required number of plaques; plaques should be transparent and contain no haze from crystallinity. Processing conditions should be the same for control and test. In the event of process change, it is required to record this change.
5. Purge injection unit with natural PET between samples

Injection Molding Preforms, PET-P-09

Background

This document presents standard practice to Injection mold crystallized pellets into preforms designed to be blown into carbonated soft drink (CSD) bottles ranging in size from 0.5L to 2L. This processing practice is used only as a requirement of the PET-A-01 Application Guidance for Recycled PET

Practice Summary

Crystallized pellets from each sample to be molded are dried and injection molded into preforms designed for the size bottle that they will be blown into. Samples of the molded preforms will be evaluated for color, haze, acetaldehyde concentration, and content of black specks and inclusions.

Equipment Required

- Desiccant or vacuum dryer
- Lab Scale injection molding machine allowing no more than 6 minutes of injection barrel residence time.
- Preform injection mold
- Chiller for mold
- Moisture tester capable of evaluating < 50ppm water content in PET pellets

Materials and Reagent Required

- Crystallized pellets made from control material with an IV of 0.78-0.82 dL/g.
- Crystallized pellet made from test material with an IV of 0.78-0.82 dL/g.

Practice Steps

Note that these preforms will typically be injection molded on a unit cavity injection press. Thus, the level of acetaldehyde (AA) developed during the injection molding process may be expected to be slightly higher than what would normally be achieved in high cavity injection molding operations.

1. Weigh the required amount pellets for processing the number of preforms required for each control and test material.
2. Dry the pellet samples separately in a desiccant dryer capable of achieving a moisture content of less than 50 ppm for the PET pellets dried in the unit at 160° C.
3. Starting with the control material, prepare the injection machine to provide:
    a. Target melt temperature at nozzle to be 275°C
    b. Target mold temperature 20°C
    c. No more than 6 minutes barrel residence time.

4. Injection mold the required number of preforms; preforms should be transparent and contain no haze from crystallinity. Processing conditions should be the same for control and test samples. In the event of a process change, it is required to record this change.
5. Purge injection unit with natural virgin PET between samples

Blow Molding Bottles, PET-P-10

Background

This document presents standard practice to blow mold preforms into carbonated soft drink (CSD) bottles ranging in size from 0.5L to 2L with a petaloid base. Since the bottle test performance of the Innovation recycle-content bottles will be compared to the control bottles, it will still be possible to judge the Innovation's acceptability for the recycle stream if the bottle test criteria are met. Because the non-CSD control materials are currently found in the recycle stream, then any new materials similar to these that do not result in significant differences in recycle-content bottle performance are, therefore, also expected to be acceptable. This processing practice is used only as a requirement of the PET-A-01 Application Guidance for Recycled PET.

Practice Summary

Preforms from each sample to be blow molded are blown into the appropriately sized, CSD bottle that they were designed for. Samples of the blown bottles will be visually evaluated for color, haze, black specks, particulates or gels, and tested to meet performance criteria.

Equipment Required

- Lab or small-scale blow molding machines utilizing infrared reheat technology
- Blow mold
- Chiller for mold

Materials and Reagent Required

- Preforms made from control material.
- Preforms made from test material.

Practice Steps

1. Preforms molded previously by PET-P-09 should be allowed to age a minimum of 1 day at ambient conditions. Preferably these preforms should be stored after molding in sealed polyethylene bags. Control and test preforms should be stored under identical conditions and blown at a similar age.
2. Starting with the control preforms, develop a set of blow molding process conditions to provide bottles that are expected to meet design sidewall thicknesses, section weights and top load performance criteria.
3. Blow mold the required number of preforms; bottles should be transparent and contain no haze from crystallinity. Processing conditions should be the same for control and test samples. In the event of a process change, it is required to record this change.

Sheet Extrusion, PET-P-11

Background

This document presents standard practice to extrude sheet from either flake or pellets. The test material will be extruded into sheet at a level of 50% blended with control material. The sheet thickness to be produced should be 0.015±0.002 in. The control material will be used to set-up and adjust the equipment and produce samples required for testing. The test sample should be processed under the identical conditions of the control. If this is not possible, then any changes made should be documented. The rPET-to-Sheet evaluation program is designed to show processing and unoriented sheet performance differences between a control material and that control material containing recycle-content Innovation material. It is a ***comparative*** study that does not rely on the final sheet meeting absolute performance criteria. This processing practice is used only as a requirement of the PET-A-01 Application Guidance for Recycled PET.

Practice Summary

Sample flake or pellets from each sample to be extruded into sheet with a target thickness of 0.015 ± 0.002 in. The width of the sheet produced is not critical and can vary based upon the equipment being used. Samples of the extruded sheet will be visually evaluated for black specks, particulates or gels, and tested to meet impact guidance criteria.

Equipment Required

- Lab or small-scale sheet extrusion machine
- Suitable take out chilled roll stacks

Materials and Reagent Required

- Flake or pellets made from control material.
- Flake or pellets made from test material.

Practice Steps

1. Dry the control and test flake or pellet samples (produced previously per PET-CG-01) separately in a desiccant dryer capable of achieving a moisture content of less than 50 ppm for the PET pellets dried in the unit at 160° C.
2. Starting with the control flake or pellet samples, develop the extrusion process parameters using standard process temperatures of 260° C to 302° C.
3. Extrude through a melt filter 40/150/40 mesh screen pack. (Note, this is a more, coarse filtration than other testing.) This represents minimum filtration level currently used for sheet extrusion. Processing conditions should be the same for control and test samples. In the event of a process change, it is required to record this change.
    a. Note: If the small lab scale equipment is unable to process the standard grind flake samples, it is permissible to perform a second grind through a 4mm screen insuring that all ground material is captured for the study and there is no loss of the Innovation being studied through static cling to the grinder or screen.

   b. Note: If pellets are used to make sheet that have been obtained following PET-P-06, the melt filtration step at 40/150/40 mesh can be eliminated as they have already been melt filtered to a higher extent.

4. The following processing characteristics should be monitored and reported
   a. Extruder amps (maximum difference ±10% difference)
   b. Melt drop between die and roll stack nip (no die drool or blowouts)
   c. Bank stability (no substantial change)
   d. Fuming (no increase)
   e. Roll plate out (no increase)
   f. General sheet optical quality (no substantial visual difference)

Preparation of PET Articles with Potential Time Dependent Behavior, PET-P-12

Background

Containers with certain additives such as oxygen scavengers can generate significant changes shown as lower L* and/or higher b* values immediately upon production or due to aging during the intended shelf life that could have a significant negative impact on the recycling stream. This phenomena of aging over the intended shelf life has been described as “Time Dependent Behavior”.

The following procedure(s) can be used when testing PET packaging that employs an additive or component with chemistries that may exhibit time dependent behavior during the lifecycle of the package as it is produced, used, consumed, and recycled. This sample preparation practice can be used in any laboratory evaluation directed to identify if any given container shows a “Time Dependent Behavior” that has significant negative impact on the clear PET recycling stream. This Practice is a required element when PET-CG-01 is employed to evaluate packages known to have time dependent behavior. However, this practice is not applicable to oxo or biodegradable additives that might require many months to many years of aging to show any impact.

Typical Chemistries that have shown Time Dependent Behavior in the past include:

- Additive(s) blended into PET at the injection press that contain an oxidizable component(s) and a transition metal catalyst(s) to allow for reacting with and binding oxygen. Or,
- A polymer layer in the wall section of a PET container that employs an oxidizable component(s) and transition metal catalyst(s).

Equipment Required

Finished Articles Storage room, area, or environmental chamber capable of maintaining a minimum of 21° C and at least 50% RH. The room/chamber must be large enough to hold the combined volume of finished articles required for the given test program.

Materials Required

- Finished Articles identified as the Control Articles.
- Finished articles identified as the Innovation Articles.
- Controlled environment.
- Instrumentation to measure temperature and humidity.
- 2 mil poly bags for alternative storage practice “B”.
- Higher temperature environment for alternative storage practice “C”.

Practice Steps

General method information – Method A

1. Determine the weight of the innovation packages and control articles required for use with PET-CG-01.
2. Produce the required number of control and innovation articles.
3. Store the finished articles in the following environmental conditions.
   a. Empty, uncapped, and unsealed finished articles can be stored at a minimum temperature of 21° C and at least 50% RH for a minimum of 90 days.
   b. Both the control and innovation must be held under the same conditions and in the same area.
   c. If the containers are palletized the stretch wrap must be cut, and air must freely circulate among the containers in storage. No forced air flow is required.

NOTE: Some laboratories may have environmental rooms or chambers that are large enough to provide the storage conditions listed above. However, storing the finished articles at a third-party lab is not required. If a third-party lab is not used, the innovator is required to describe the storage area and environmental controls to the third-party lab who will be conducting PET-CG-01, and it will be included in the report. For instance, the finished articles were stored in a non-climate-controlled warehouse in Orlando Florida during June, July, and September where the average temperature was over 21° C, and the humidity was over 80%. This is an extreme example but there are rooms with climate controls in office complexes capable of controlling the temperature and humidity within these recommended ranges. A plastic tent or enclosure containing means for humidity and temperature control might also be used.

Alternative storage Method B

If a shorter time duration is desired a higher temperature is required to increase any potential chemical reactions.

- Determine the weight of the innovation packages and control articles required for use with PET-CG-01.
- Produce the required number of control and innovation articles.
- Store the finished articles in the following environmental conditions.
- Empty, uncapped, and unsealed finished articles can be stored at or above 50^0C, but no more than 55° C, and at least 50% RH for a minimum of 30 days. It is also allowed to store granulated flake under these same elevated temperature conditions when open to atmosphere.

This high temperature accelerated aging can be employed by those who have prior data supporting that aging at 50 to 55° C for 30 days is predictive of the oxygen consumption and color formation results obtained at 21° C for 90 days.

Test Assessment

Innovation and control articles prepared according to this aging procedure are suitable for testing as outlined in the PET-CG-01 Protocol.

Those conducting the aging procedure will report:

1. The general approach to how packages or granulate were stored with specific reference to:

   - Procedure used: General, Alternative "B" or Alternative "C".
   - Date the control and test articles were produced.
   - Where the storage environment is located and who is controlling the aging environment.
   - Date the control and test articles were placed in the controlled environment (start of 90- or 30-day aging period).
   - Date the control and test articles were removed from the controlled environment (end of 90- or 30-day aging period).
   - Minimum temperature used for this procedure.
   - Minimum relative humidity used for this procedure.

2. In the case of oxygen scavenger/barrier technologies, provide data confirming that the containers provided the targeted level of oxygen barrier during the time period of aging of the containers.

3. Incorporate the above data in the standard PET-CG-01 summary and final report.

DOCUMENT VERSION HISTORY

Version	Publication Date	Revision notes
1	November 16, 2018	
2	April 11, 2019	Added APG resins to list of control resins per PTC approval Dec. 2018
3	Sept 17, 2019	Added Practices PET-P-09, PET-P-10, and PET-P-11.
4	November 01, 2022	Added new Practice PET-P-12.
5	September 3, 2024	Added updated hyperlinks to other testing protocols