ASSOCIATION OF PLASTIC RECYCLERS

Polyolefin Standard Laboratory Processing Practices

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Introduction

Scope, Significance, and Use

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

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

Contents

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Selection of Control Resin, O-P-01

Background

Recycle testing 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 or innovation. This practice provides the steps used to select a control virgin resin and produce articles suitable for control.

HDPE control resin

HDPE control resin can be

1. Selected from a list of named commercial virgin HDPE resins.
2. Qualified by testing per the APR HDPE Critical Guidance Protocol, HDPE-CG-01

APR Named List

Homopolymer Resins	Copolymer Resins
Chevron Phillips Marlex® EHM 6007	Chevron Phillips Marlex® HHM 5502BN
Dow UNIVAL™ DMDH-6400 NT 7	Chevron Phillips Marlex® 9505H
Exxon-Mobil Paxon™ HDPE AD60-007	Dow UNIVAL™ DMDA-6230 NT 7
	Dow UNIVAL™ DMDA-6200 NT 7
	ExxonMobil Paxon™ HDPE AB50-003

These named resins are recommended for any laboratory evaluations that require a control resin. Higher melt flow resins are not defined here as the rigid stream is predominantly fractional melt. When there are higher melt materials these are expected to blend in and not impact the material properties and performance. If a higher melt resin is required for the control, contact the APR technical director for guidance.

HDPE control resin selection criteria:

- They represent resin compositions that are widely used and available commercially.

PP control resin

There are hundreds of PP materials available in the North American market that can be used to make rigid PP packaging articles. These differ in melt flow rate, may contain comonomers which vary by type and content, flexural modulus, impact resistance, and additives. Thus, the selection of a universal virgin control resin for use in comparing and blending with the Innovation test article is not practical.

To assess the effectiveness of the innovation, the natural-colored base polypropylene without the innovation present should be used as the control. This polypropylene must be consistent with the ASTM D4101 definition of polypropylene.

Control articles

The control resin is typically used to make extruded or molded control articles that are used in testing.

These articles may include:

- Bottles or containers made by blow molding
- Containers made by thermoforming sheet
- Containers made by injection molding
- Pellets for testing resins and resin blends that incorporate the innovation being tested but have not been converted into package form (HDPE-CG-01 or PP-CG-01 Path 2)

Preparation of Polyolefin Articles for Evaluation, O-P-02

Background

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

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

- Labels attached with adhesive or direct printed.
- Closures – including seals and dispensers.
- Attachments such as handles, or other features incorporated with an article that are not closures or labels.
- Functional additives such as moisture and gas barriers.

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

- An "intended" product is prepared as it will be sold in the market using the specific materials and design that will be marketed. Typically, intended products are ready for production or commercialization.
- A "generic" product is one used in the laboratory to represent a variety of market products and represents a facsimile of what will be commercialized. For example, the ink colors and

composition are represented but the label is not the branded label. Typically, generic products are still in the development stage.

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 unprinted, with intended inks and graphics or with generic inks. Test containers may be 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 HDPE or PP resin to be employed in the evaluation:

Label style	Intended labels	Generic labels
Pressure sensitive labels	Test intended labels on HDPE or PP containers blended with un-labeled control containers 50:50. Same resin used for both test and control containers.	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. Same resin used for both test and control containers.
Shrink and stretch sleeve labels	Test intended labels on HDPE or PP containers and blend with un-labeled control containers 50:50. Same resin used for both test and control containers.	Test labels at 3 wt% of all test containers, no 50/50 dilution with control bottles required. Same resin used for both test and control containers.
Direct print labels	Test intended labels on HDPE or PP containers and blend with un-labeled control bottles 50:50. Same resin used for both test and control containers.	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. Same resin used for both test and control containers.
Wrap labels	Test intended labels on HDPE or PP containers blended with un-labeled control containers 50:50. Same resin	Test wrap labels at 0.6 wt% of all test containers, no 50/50 dilution with control bottles required. Same resin used for both test and control containers.

	used for both test and control containers.	
ROSO labels	Test intended labels on HDPE or PP containers and blend with un-labeled control containers 50:50. Same resin used for both test and control containers.	No generic APR Guidance for ROSO labels today. Same resin used for both test and control containers.
In-Mold Labels	Test intended labels on HDPE or PP containers and blend with un-labeled control containers 50:50. Same resin used for both test and control containers.	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. Same resin used for both test and control containers.

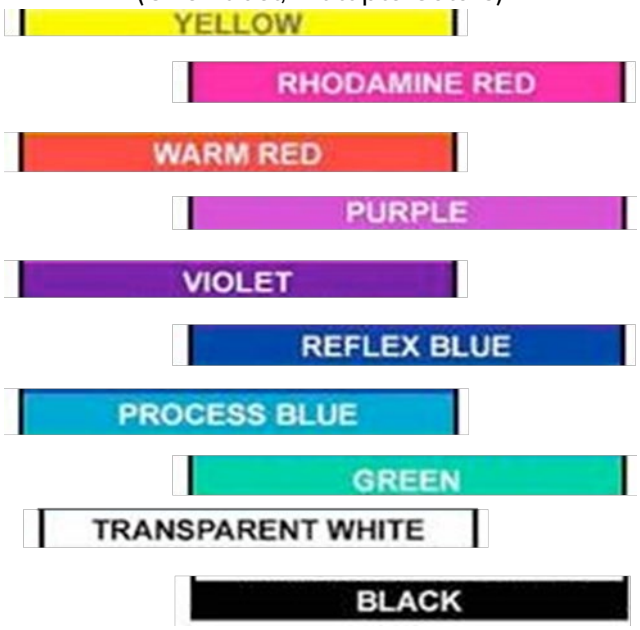
When using generic labels, the following images illustrate ink pattern options that may be used:

Option 1 Pantone– Clear Articles

(Multiple Labels with flood coat on each)

Option 2 Pantone– Clear Articles

(One Label, Multiple Colors)

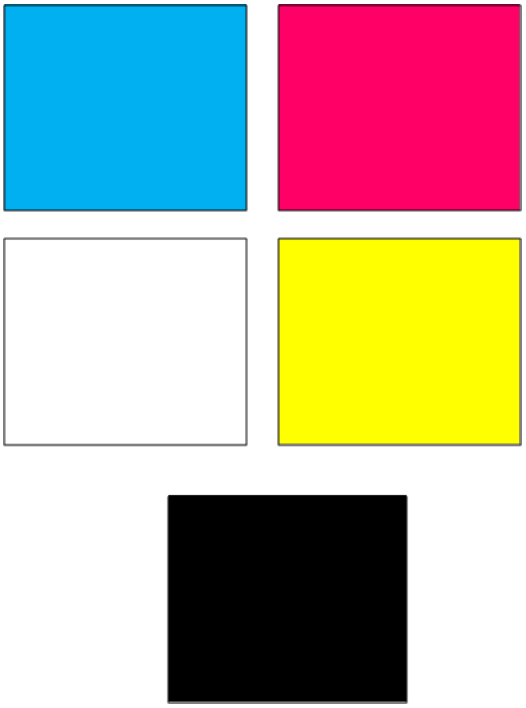


Option 3 w/4 Color – Clear Articles

(One Label Multiple Colors)

Option 4 w/4 Color – Clear Article

(Multiple Labels with flood coat on each)



Granulating Polyolefin Articles to Flake, O-P-03

Background

HDPE and PP polyolefin articles are reduced in size by granulating (grinding) articles in a rotary granulator. This step creates HDPE and/or PP 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 polyolefin 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. Observe all safety practices relevant to the machine, including lock-out procedures.
2. 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.
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 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 samples.

Commercial Basic and Caustic Wash of Polyolefin Flake, O-P-04

Background

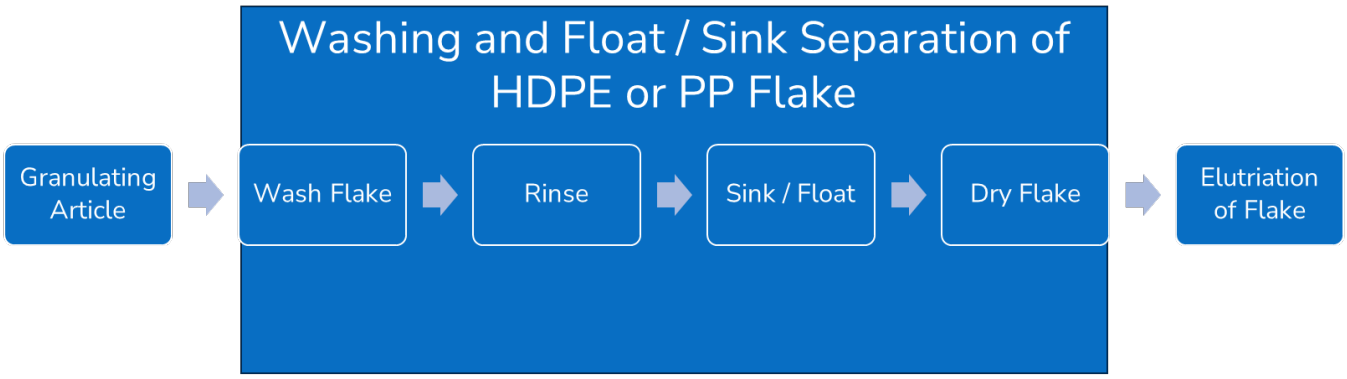
In commercial reclaiming facilities, after granulation the polyolefin flake is washed in water with or without caustic and detergent solution, depending on the feedstock and the individual reclaimer's quality requirements. The wash step is intended to remove contamination from flake and can contribute to liberating labels, adhesives, coatings and layers from the flake.

In laboratory testing, washing conditions should approximate the practice with the most risk of any contaminants, particularly labels and adhesives, remaining in the washed flake. This allows evaluation of innovations in label technology through melt filtration, pelletization, pellet testing and bottle testing. For this reason, APR recommended lab practice is for a room temperature wash without added surfactants or caustic, referred to as Basic Commercial Wash.

A small sample is retained for washing under stronger conditions, using detergent and caustic solution with hot water, to test label and contaminant removal on its own merits.

Practice summary and illustration

Granulated material is washed in a water solution with agitation. Washed flake is then rinsed in 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.



Equipment required

- Stainless steel mixing tank with the following specifications:
  - Sized to allow water and 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 impeller 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 an 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 polyolefin flake required for the evaluation as specified in the test or Critical Guidance Protocol.

Wash Procedure 1 - Commercial Basic 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 the polyolefin flake planned for use in wash, for example, 2000 g (2000 ml) water, 500 g of granulated flake.
   b. Add 0.3% surfactant
   c. Position the impeller between 0.5 to 1 impeller diameters from the bottom of the mix container.
2. When the water reaches 20° C +/- 2 °C, add the polyolefin flake
3. Increase the agitator speed to achieve an impeller tip speed of at least 240 meters per minute and agitate for 10 minutes.
4. After 10 minutes, shut off the agitator impeller; 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.

Proceed to rinse, sink-float and dry steps in this procedure document.

Wash Procedure 2 – Caustic 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 the polyolefin flake planned for use in wash, for example, 2000 g (2000 ml) water, 500 g of granulated flake.
    b. Place the container on the hot plate or heat source.
    c. Position the impeller between 0.5 to 1 impeller diameters 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 0.5% 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 75 °C, add the olefin flake while maintaining wash solution temperature at 75 °C.
3. Increase the agitator speed to achieve an impeller tip speed of at least 240 meters per minute and agitate for 10 minutes.
4. After 10 minutes, shut off the agitator impeller; 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.
9. Proceed to rinse, sink-float and dry steps in this procedure document.

Rinse, Sink-Float and Dry Recovered Materials from Wash Procedures 1 and 2

Rinse

1. Prepare the rinse container
    a. Fill a clean stainless container with tap water with 4x the weight of polyolefin flake.
    b. Position the impeller 0.5 to 1 impeller diameters from the bottom of the container.
2. When the rinse water reaches 20 °C +/- 2 °C, add the flake and maintain the rinse water temperature
3. Increase the agitator speed to achieve an impeller tip speed of at least 240 meters per minute and agitate for 5 minutes.
4. After 5 minutes, shut off the agitator impeller; 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 for drying and testing.
7. Pour the liquid-flake mix over the de-water screen to recover 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 flake, and no more than 10x the weight of flake.
2. Add the polyolefin flake to the container and agitate for 5 minutes. Agitation may be done by hand or with mild agitation from a mixer at a maximum of 500 RPM.
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 mixture over the de-water screen to recover any sinking solids contained in the rinse water.

Dry Recovered Materials

1. Dry 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 Polyolefin Flake, O-P-05

Background

In the industrial HDPE and PP 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 HDPE and PP flake. This step may remove label residue, dried inks, or layers, for example. This document presents standard practice to elutriate HDPE and PP 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 1.0% 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 MFR 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 and Pelletization, O-P-06

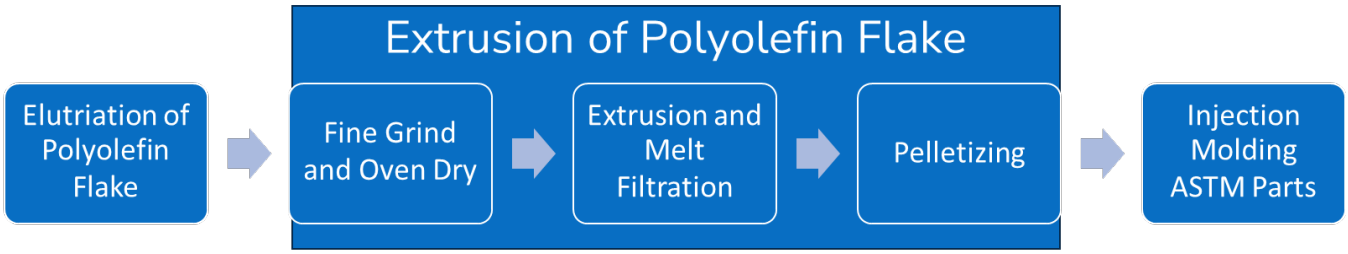
Background

In the industrial HDPE and PP recycling process, after elutriation the HDPE and PP flake is extruded and melt filtered to be converted into pellets. This document presents a standard practice to extrude and create strand cut HDPE or PP pellets from blended polyolefin flakes.

The practice includes the steps of finer grinding of flakes (if required), drying, extrusion, and pellet forming. 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 an oven to remove surface moisture. Dried flake is melted and filtered in an extruder to create strand cut pellets. Melt pressure is recorded ahead of the melt filter.



Note: There are situations in laboratory recycling assessments where it is necessary to add a heat history to polyolefin resin to simulate a molding operation. This Extrusion Practice can be followed to add a heat history to polyolefin 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

- Dryer capable of drying HDPE or PP flake and/or pellet at 60 °C.
- Extruder suitable for HDPE or PP flake or pellet processing in the laboratory.
    - A 25 to 35 mm extruder with a 24:1 to 36:1 L/D is suitable for laboratory use.
    - Melt residence time in the extruder should be no more than 6 minutes.
    - The extruder is equipped with a breaker plate and screen pack.
    - The extruder must display pressure values ahead of the screen so that pressure data may be recorded.
    - The extruder requires water bath and pelletizer.
- A scale for measuring extruder output.

Materials required

- Products such as plastic pails or bags to hold final pellet samples.
- Screen pack for the extruder - 50-120-50 mesh (300-120-300 µm).
- Processed control article, processed test article, consisting of blends of flake or densified material.

Practice steps

1. Densification may be required to allow good feeding into the extruder feed throat and to achieve steady pressure readings ahead of the screen pack. If densification is used both the control and test will need to be prepared using the same practice. Reference P-FPE-04.

2. Weigh the required amount of flake or pellet for processing the required minimum run time of 30 minutes.
3. Dry the flake or pellet at 80-90 °C for a minimum of 10 minutes and up to one hour to remove surface moisture to less than 1% by weight. Weigh the sample before and after drying and record the weight percent of moisture removed by scale or moisture analysis equipment.
4. Starting with the control material, prepare an extruder suitable for PE flake or pellet processing in the laboratory.
5. Extrusion Steps
    a. Extrude the control article flake or pellet first at a target melt temperature in range of 190-245 °C.
        i. The melt will be filtered with a clean 50-120-50 mesh (300-120-300 µm) melt filter
        ii. 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. It is important to achieve similar pellet sizes for each of the test and control materials.
    b. Start run timer after a steady state has been reached
    c. Record pressure values ahead of the screen pack at a minimum of every five minutes for a 30 minute run time after the steady state has been reached. This may be done with a continuous automatic pressure recorder or manually.
    d. Record any occurrence of unusual, sticking, fumes, odor or build-up occurring at the feed throat or die exit of the extruder.
    e. Record surface irregularities such as but not limited to porosity, roughness, grainy, gloss etc.
    f. Save a sample of extruded pellets for melt flow index measurement. Sample pellets to be obtained ½ way through run time or later at random. Additional samples can be retained for color and other measurements as desired.
    g. Retain a 50-100 gram sample from this practice step for later use as necessary.
    h. Purge the extruder and change the screen pack between each run with virgin resin
    i. Store the pelletized samples in pails or bags
    j. Extrude all subsequent samples at the same conditions; achieve pellets of similar size for each sample set when the pellets will be used for part production.
    k. Processing conditions should be the same for control and test. In the event of process change, it is required to record this change and report.

Steps Required to Assess Pressure Values

For the control and test material:

After the steady state has been reached, 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.

$(P_f - P,) / P_i$ this is recorded as evidence for later determination of screen pack build-up.

Record control and the test graphically to represent the potential for screen pack build up.

Injection Molding ASTM Parts, O-P-07

Background

This document presents standard practice to Injection mold either dry flake or pellets into ASTM parts for testing Tensile Strength, Flexural Modulus and/or Izod Impact.

Equipment Required

- Dryer capable of drying HDPE or PP flake and/or pellet at 90 °C.
- Lab Scale injection molding machine allowing no more than 6 minutes of injection barrel residence time.
- Heater and/or Chiller for mold

Materials and Reagent Required

- Flake, and/or pellet made from control material
- Flake, and/or pellet made from test material

Practice Steps

1. Weigh the required amount of flake or pellet for processing the number of parts required for each control and test material.
2. Dry the flake or pellet at 80-90 °C for a minimum of 10 minutes and up to one hour to remove surface moisture to less than 1% by weight. Weigh the sample before and after drying and record the weight percent of moisture removed by scale or moisture analysis equipment.
3. Starting with the control material, prepare the injection machine to provide:

a. Target melt temperature at nozzle of 190-245 °C depending on primary base resin
b. Target mold temperature 20-40 °C
c. No more than 6 minutes barrel residence time

4. Injection mold the required number of parts for testing. Processing conditions should be the same for control and test. In the event of process change, it is required to record this change and report.
5. Tested parts to be obtained ½ way through run time or later at random.
6. Purge injection unit with virgin resin between samples

DOCUMENT VERSION HISTORY

Version	Publication Date	Revision notes
1	10-23-2019	Updated entire document, approved by OTC on 10/2/19
2	07-24-2020	Wash process updated, approved by OTC on 6/4/2020
3	07-18-2024	Updated PP control, editorial and formatting changes
4	09-09-2024	Updated hyperlinks to new website
5	02-05-2025	Updated screen pack mesh size, editorial and formatting changes