



PP Benchmark Test PP-B-01

Introduction

This document is intended to allow a stake holder to understand if a polypropylene (PP) article that is in the market today, or intended to be brought to the market, can be considered recyclable. Meeting the criteria outlined in this Test Document will allow materials and design innovators the opportunity to receive recognition from the APR that a PP article is compatible both with today's recycling processes and the types of PP materials currently being recycled. The APR is providing this guidance to allow stake-holders to assess whether a PP article is compatible with the recycling process. Further, the APR recognizes that new packaging innovations, such as new resins, labels, or additives, may create products that present technical challenges for recycling. The APR wishes to offer guidance to product innovators to confirm that their innovations lead to finished articles that are compatible with recycling operations.

The APR encourages Innovators to perform comprehensive recycle evaluation studies on new materials, additives and articles intended to be introduced into the PP mixed-rigid stream. A definition of the mixed-rigid stream is listed in the Attachment at the end of the document. This document describes the protocol to be followed to evaluate the mechanical recyclability of rigid packaging articles that may consist of any combination of the materials listed below. Additionally, guidance is provided to help determine if the recycle properties of these Innovation articles meet basic requirements.

1. PP resins
2. Additives
3. Coatings
4. Labels
5. Adhesives
6. Multilayer resins
7. Closures and lidding materials
8. Attachments
9. Direct printing on packaging

This document also allows for the study of molded/shaped/formed PP articles that might result from new:

1. Manufacturing methodologies
2. Package designs



3. Package structures

One focus of the testing is to confirm that an article will not interfere with the recycling process, so impact on process steps such as float/sink, wash, air elutriation, and melt filtration are evaluated. A second focus is to confirm that the finished article does not interfere with the physical properties that a recycled PP must offer to the industry.

Because the mixed rigids stream is comprised of parts made with a variety of colors and direct printing, the impact of color is not considered in this test document. Also, the APR does not support the use of additives that promote oxidative degradation or bio-degradation without extensive testing beyond the scope of this test protocol. Therefore, molded articles with oxo or bio degradation additives are not considered within the scope of this test document.

This test document will help determine if an Innovation will be problematic for mechanical recycling and determine, after molding into test specimens, that basic physical property parameters can be met. These guidelines may change as the evolution of the PP mixed rigid stream evolves over time, and thus periodic analyses of the current PP recycle stream will be made and adjustments to the test requirements made over time as necessary to update PP recyclability standards. This test document does not consider the effectiveness of NIR detectors at misidentifying PP test articles due to the presence of labels or large non-PP closures. Currently there is no reliable laboratory test that can predict how well these mixed material articles will be identified by material auto-sortation machines. Once developed, a test such as this can be incorporated into future revisions to this document. Additionally, PP structures made using EVOH or nylon may require further study. The use of these materials in a PP article should be identified by the submitting party and the APR can decide what further testing may be needed at that time.

The APR's Champions for Change program recognizes Innovators for employing and meeting the guidelines as established in a wide variety of testing protocols. This recognition occurs when an Innovator petitions the APR Technical Committee for review and confirmation that they have met the guidelines established by the APR for the article of study. Innovators may then publicize this recognition.

Note to the Reader

THIS TEST DOCUMENT HAS BEEN PREPARED AS A SERVICE TO THE PLASTICS PACKAGING INDUSTRY TO PROMOTE THE MOST EFFICIENT USE OF THE NATION'S PLASTICS RECYCLING INFRASTRUCTURE AND TO ENHANCE THE QUALITY AND QUANTITY OF RECYCLED POSTCONSUMER PLASTICS. THE INFORMATION CONTAINED HEREIN REFLECTS THE INPUT OF



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APR MEMBERS FROM A DIVERSE CROSS-SECTION OF THE PLASTICS RECYCLING INDUSTRY, INCLUDING PROFESSIONALS EXPERIENCED IN THE RECYCLING OF THE POSTCONSUMER PLASTIC ARTICLES DISCUSSED IN THIS DOCUMENT. IT OFFERS VALUABLE INSIGHT ON HOW MATERIALS, ADDITIVES, LABELS, ADHESIVES, MULTILAYER STRUCTURES ETC. IMPACTS CONVENTIONAL POLYPROPYLENE PLASTIC RECYCLING SYSTEMS AND PROVIDES USEFUL RECOMMENDATIONS FOR UNDERSTANDING HOW TO MAKE THEM COMPATIBLE WITH CURRENT RECYCLING SYSTEMS.

BECAUSE NEW TECHNOLOGY DEVELOPMENTS ARE ALWAYS BEING MADE, THIS DOCUMENT CANNOT ANTICIPATE HOW THESE NEW DEVELOPMENTS MIGHT IMPACT PLASTIC BOTTLE RECYCLING. WHILE THE INFORMATION IN THIS DOCUMENT IS OFFERED IN GOOD FAITH BY APR AS AN ACCURATE AND RELIABLE DISCUSSION OF THE CURRENT CHALLENGES FACED BY THE PLASTICS RECYCLING INDUSTRY, IT 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.

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Polypropylene Control

This test document is unique in that there are no virgin control materials that need to be evaluated for performance with the Innovation article being studied. 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, co-monomer type and content, flexural modulus, impact resistance, additives, and colors. Thus the selection of a virgin control material for use in comparing and blending with the Innovation test article is not practical. In place of a control, the test document will evaluate the Innovation's critical physical data when this test molded article is subjected to two additional melt histories that simulate the recycling of mixed rigid PP articles. In order to set-up the elutriator, a material or article should be selected that does not



contain the innovation or select an article that is in the market and being recycled that is similar in nature to the Innovation

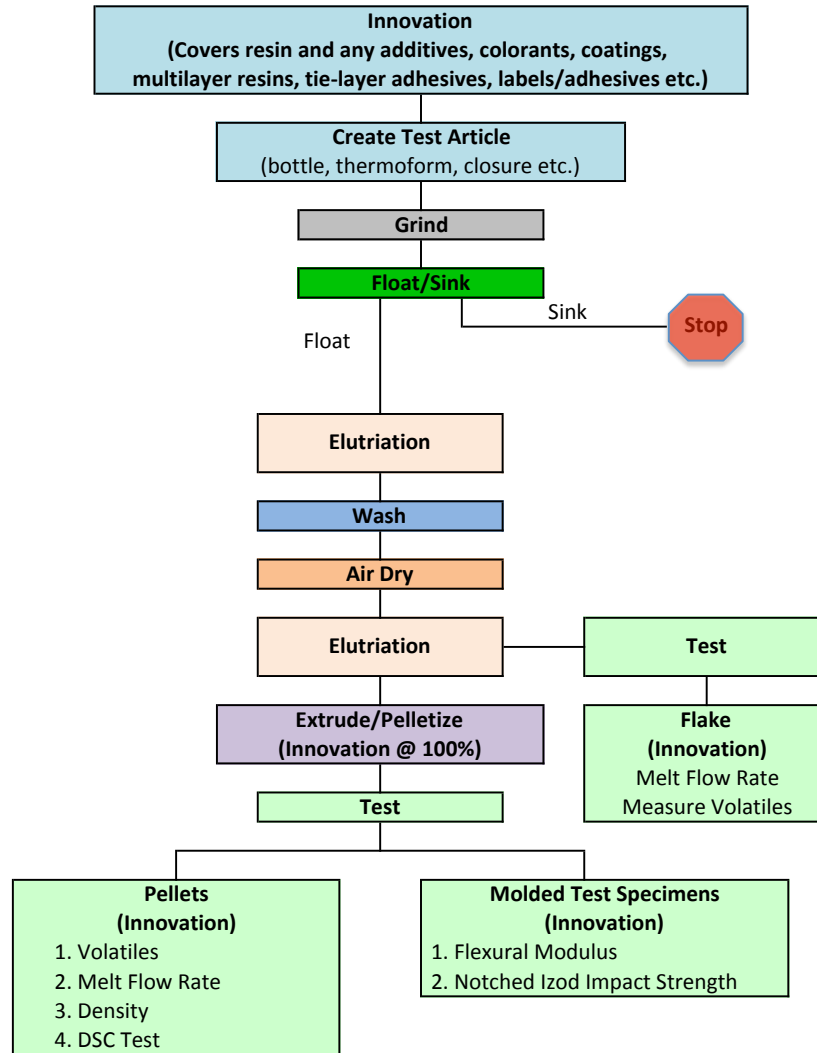
Innovation Polypropylene (Material or Article)

- If the Innovation is a resin, additive, adhesive, label etc., it must be made into or affixed to a molded test article for study. Ideally this article should represent the form that will be introduced into the market (bottle, thermoform, cup, closure etc.). Other test forms may be acceptable, but will require the approval of the APR Technical Director prior to initiation of the study.
- The Innovation PP article to be studied should be submitted in its final fully formed packaging shape (bottle, thermoform, closure etc.) along with all labeling, decoration and attachments that would be present when this package is recycled.
- No test blend variables are required. The Innovation article is subjected to recycle processing and converted into tensile bars for testing to determine final test properties.

There are three basic types of PP: Homopolymer, Random Copolymer, and Impact Copolymer. All three are present in the mixed rigid PP recycling stream, however, each is unique and has its own typical physical properties. For that reason, different minimum flexural modulus and notched izod impact resistance limits have been specified for each. Furthermore, impact copolymers have a wide range of physical properties based on how much rubber is incorporated into the specific grade by the resin manufacturer. In order to allow for this broad range of physical properties, but also maintain the integrity of the testing and the PP recycling stream, separate minimum flexural modulus values were set for grades that have very high levels of rubber (as indicated by notched izod values greater than 2.0 ft-lb/in), and separate minimum notched izod limits were set for grades that have very low levels of rubber (as indicated by flexural modulus values greater than 200,000 psi).



Polypropylene Rigid Packaging Recycle Process Flow Chart



Re-claim Processing Test Protocol

The previous flow diagram illustrates the recycling steps listed below:

1. The Innovation article should be dry-ground to nominal ¼" to ½" size flake.
2. Measure the MFR of the Innovation Material.
3. Perform a sink/float test on the Innovation material by adding 500g of the ground flake to 2000 mL of water.
 - a. Stir vigorously for 1 minute. Be sure all air bubbles are released from flakes.



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- b. Allow the suspension of flake in the water to settle and float.
 - c. Isolate, air dry, and weigh all sinking particles/flakes.
 - d. Isolate and air dry floating flakes
 - e. If the dry weight of sinking particles/flakes exceeds 0.05g, stop the test as the Innovation fails. There is no need to proceed further.
4. Set-up the air elutriation process using the same type article made without the Innovation or a similar PP article currently in the market and found in the recycle stream (See Attachment at end.). Dry grind this article to nominal ¼" to ½" size flake and elutriate to remove light fractions with one pass and with less than 2% loss. Elutriate the Innovation flake using the same setting, and record the weight loss.
 5. Prepare a wash solution of 0.3% by weight Triton X-100 (6.0 gms or 5.7 mL per 2,000 ml water), adjust the pH of the wash solution using caustic (NaOH) to achieve a pH of 9.5 to 10. Note: Triton X-100 must be dissolved in warm (nominal 100°F) water prior to the addition of any caustic! Report wash solution composition.
 6. Mix ground flake Sample Variables at a ratio of 500 grams solids per 2,000 mL wash solution.
 7. Wash in highly agitated (1,000 rpm with impeller 0.6 cm from bottom of wash container) water at 60±2°C for 15 minutes.
 8. After 15 minutes of washing, stop agitation and remove agitator. Cease heating. Let mixture of solids and solution stand for several minutes to allow floatable materials to float. Skim off floatables and save for rinsing. Separate sinking solids from wash solution and discard.
 9. Remove and save a sample of the wash water for color measurement.
 10. Neutralize the wash water and discard.
 11. Re-suspend all of the floating material in fresh tap water at a ratio of 500 grams to 2L of water and stir at a low agitation rate of 250 rpm for 5 minutes. Stop agitation and separate the floatables by either straining or draining the rinse water from the wash device. Repeat this rinse step one more time.
 12. Isolate all floatables and air dry.
 13. Using the same setting established in Step 4, perform a second elutriation with one pass on the dried washed flake; record the weight loss.
 14. Suitably dry the test variable to ensure no surface moisture remains. Samples can be air-dried.
 15. Measure volatiles on flake sample variables per Testing Section 1.0 below.
 16. Set-up the extruder using virgin PP (the testing lab can select a suitable PP) and then extrude the PP Innovation flake adjusting settings as necessary to produce a uniform steady extrudate. Melt filter pelletize using a 40/150/40 mesh screen, for a minimum of 30 minutes. The extrusion pressure and amperage draw should be monitored and the melt filter screens checked to ensure no buildup has occurred on the screen. Extrude at



a rate of at least 500 g/cm² per hour.

- a. Note if the Innovation variable exhibits unique odors, fumes, smoking, extrudate spitting, or shows evidence of blistering of the extrudate.

17. Injection mold test plaques/test specimens.

18. Test sample pellets, plaques, and tensile specimens per the tests outlined below.

Testing Requirements and Guidelines

1.0 Flake Testing

1.1 Elutriation Loss

- 1.1.1 Report loss for first and second elutriations.

1.2 Volatiles Test:

- 1.2.1 Air-dry flake samples
- 1.2.2 Heat 10 grams of each test variable at 180°C for 10 minutes
- 1.2.3 Record the weight before and after heating (moisture analyzer may be used for this measurement)
- 1.2.4 There should be no more than a 1% weight loss

1.3 Melt Flow Rate

- 1.3.1 ASTM D1238 (no guidance value)

2.0 Filterability Test

2.1 Extrusion Melt Pressure

- 2.1.1 Less than a 10% increase in pressure for the Innovation Variable after extruding through 40/150/40 mesh for 30 minutes.

3.0 Pellet Testing

3.1 Volatiles Test

- 3.1.1 Air-dry pellet samples
- 3.1.2 Heat 10 grams of each test variable at 180°C for 10 minutes
- 3.1.3 Record the weight before and after heating (moisture analyzer may be used for this measurement)
- 3.1.4 There should be no more than a 1% weight loss

3.2 Melt Flow Rate

- 3.2.1 ASTM D1238 (no guidance value)

3.3 Density

- 3.3.1 Density <1 g/cm³

3.4 DSC Test

- 3.4.1 No high melting materials
- 3.4.2 Polyethylene content <5%
- 3.4.3 Note if there is evidence of EVOH or Nylon content



4.0 Physical and Mechanical Properties on molded Test Specimens

- 4.1.1 Flexural Modulus @ 73°F (ASTM D790) minimum values are based on the type of PP used in the Innovation. In the event that a PP blend is used, the predominant polymer by wgt-% will determine that article's category.
 - 4.1.1.1 Homopolymer PP
 - 4.1.1.2 Random copolymer PP
 - 4.1.1.3 Impact copolymer PP
- 4.1.2 Notched Izod Impact Strength @73°F (ASTM D256)
 - 4.1.2.1 Homopolymer PP
 - 4.1.2.2 Random copolymer PP
 - 4.1.2.3 Impact copolymer PP

5.0 General Issues

5.1 Recommended Guidelines

- 5.1.1 No additional fuming or smoking compared to controls during extrusion
- 5.1.2 No sticking between flakes
- 5.1.3 No fouling of process equipment
- 5.1.4 No creation of unsafe conditions, such as increased fire potential
- 5.1.5 No generation of black specks, color streaking, gels, or other surface defects in excess of the Control when viewing the molded polypropylene specimens.

Innovation Test Requirements to meet Guidance Criteria

Test	Guidance
Innovation @100%	
Float/Sink in H₂O	Must float, Density <1
Extrusion	<10% D increase in pressure over 30 min.
Flake Volatiles	No fuming/smoking/odors etc. <1% Wgt. Loss
Pellet Volatiles	<1% Wgt. Loss
MFR	No guidance
DSC	No high melting peaks PE content ≤ 5%
Molded Test Specimens	
Flex. Modulus @73°F (ASTM D790A)	
Homopolymer	³ 160,000 psi
Random Copolymer	³ 100,000 psi
Impact Copolymer	³ 150,000 psi for most impact copolymers
	If <150,000 psi, then the Notched Izod must be ³ 2.0 ft-lb/in



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Notched Izod @ 73°F (ASTM D256A)	
Homopolymer	³ 0.4 ft-lb/in
Random Copolymer	³ 0.4 ft-lb/in
Impact Copolymer	³ 0.9 ft-lb/in for most impact copolymers
	If <0.9 ft-lb/in, then the Flex. Mod. must be ³ 200,000 psi
Appearance	No black specks, color streaking, gels, or unusual surface defects

Attachment: Product Category Descriptions Mixed Rigid Polypropylene Articles

Bottles and Closures: Narrow neck, blow molded containers.

Buckets: All buckets and lids. Primarily 5 gallon buckets, smaller buckets such as those holding products such as cat litter as well as wide mouth containers.

Cups and Lids: All drinking cups, injection molded or thermoformed.

Containers: All non-bottle packaging containers and their lids, sealed at point of purchase. Containers may have snap-on lids, such as for sour cream tubs or baby wipe containers. Containers may have screw on lids, such as for coffee. Containers may have foil lids, such as for peel-able lids on yogurt containers. This category does include thermoformed tubs with sealed closures at point of purchase. Durable storage containers are excluded.

Bulky Items: Any large, bulky items such as milk crates, totes, and pallets.

Nursery Pots: All nursery pots, all sizes.

Thermoforms: Shaped packaging or flats from films or sheets, not sealed into a container at point of purchase. This includes clamshells, plates, blister packs, cake domes, cookie trays, and deli containers. This does not include thermoformed tubs and small containers sealed at point of purchase.

Other: Any item not previously mentioned (such as clothes hangers, small toys, cassette boxes, and small pipe).