The Association of Postconsumer Plastic Recyclers

HDPE Bottle Application Test
HDPE-A-01

The Association of Post Consumer Plastic Recyclers (APR) recognizes that packaging innovation drives the growth of bottles available for recycling and that growth in the supply of these bottles is essential to the well being of the plastic bottle recycling industry. APR also recognizes that some innovations may create bottles that present technical challenges for recycling. This document represents a tool to help the innovator understand the approximate effect of the innovation on high density polyethylene (HDPE) plastic bottle recycling processes.

The APR encourages Innovators to perform comprehensive recycle evaluation studies on new innovation materials intended to be introduced into the HDPE bottle stream. This Applications Guidance Document describes the protocols to be followed to evaluate the mechanical recyclability of the following Innovation materials that are intended to be made into or incorporated onto HDPE bottles:

1. HDPE Resins
2. Additives
3. Coatings
4. Labels
5. Adhesives
6. Multilayer resins

In particular, a comprehensive recycling evaluation is accomplished by following a step-wise process involving the evaluations of the innovation using Testing Protocols that have been developed by APR. APR recognizes accomplishment and allows upon petition for the Innovator to publicize that they have completed each step. The steps include:

STEP 1 1.00 CRITICAL GUIDANCE DOCUMENT
1.10 Resins, Additives, Coatings, Labels, Adhesives and Multilayer Resins Evaluation Protocol, Critical Guidance

STEP 2 2.00 APPLICATIONS GUIDANCE DOCUMENT
2.10 Bottle-To-Bottle Evaluation, (BtB)

Upon the completion of all parts of STEP 2, meeting or exceeding all of the strictest guidance, APR would consider a petition for full Recycling Guidance Recognition.

The screening tool referred to above in STEP 1 as the HDPE Critical Guidance document (CGD) is intended to help identify possible technical challenges that a new Innovation might create for the HDPE recycle stream. An Innovator is requested to first test an innovation material following the CDG before proceeding to STEP 2 and the more extensive Bottle-to-Bottle evaluation.

When coupling the CGD screening protocol with a Bottle-to-End-Use evaluation, it is necessary to increase the quantity of the Control and Innovation materials used in the CGD protocol to produce sufficient material for making the final test bottles

The guidance contained in this document does not include time as a variable. Innovations which include time as a factor will require additional considerations.

THIS DOCUMENT IS NOT A SPECIFICATION AND DOES NOT IMPLY IN ITS DEFINITIONS, PROCEDURES, OR VALUES FITNESS FOR USE, MARKET ACCEPTABILITY, SAFETY, OR ANY GUARANTEE OR WARRANTY. MEETING THESE GUIDELINES DOES NOT OBLIGATE APR MEMBERS TO BUY BOTTLES CONTAINING THE INNOVATION.

Moreover, the inability of an innovation to meet specified values does not imply recycling failure, but should be a clear message that significant technical challenges might exist under certain circumstances and mitigation of the issue may be needed to avoid degrading the value of the stream of recyclable bottles.

THE FOLLOWING PROTOCOLS DO NOT PURPORT TO ADDRESS ALL OF THE SAFETY ISSUES, IF ANY, ASSOCIATED WITH THEIR USE. IT IS THE RESPONSIBILITY OF THE USER TO ESTABLISH APPROPRIATE SAFETY AND HEALTH PRACTICES AND DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE.

1.0 CRITICAL GUIDANCE DOCUMENT (CGD)

Introduction

The CGD is intended to be a screening tool that can be used by Innovators to gain a quick understanding on the impact of their innovation on the HDPE recycle stream before proceeding to the more extensive Bottle-to-Bottle Protocol and other end use protocols. While the CGD protocol is designed as a recycle screening protocol, the material that has been processed in the CGD study can then be used as the starting material to continue the recycle evaluation through a Bottle-to-Bottle study.

Testing Procedures

The procedures needed for performing the tests called for in the recycling protocols are listed below:

- Color (ASTM D6290)
- Melt Index (ASTM D1238)
- Density (ASTM D1505, ASTMD792, or equivalent)
- Melt filtration
- % polypropylene in HDPE (ASTM D7399)

Testing Protocols

The detailed protocols to be followed are listed below and are described in detail in this document.

- 1.10 Resin, Additives, Coatings, Labels, Adhesives, and Multilayer Resins, Critical Guidance

Control Resins

The virgin control resins that can be selected for use following the CGD are listed below. The Innovator is requested to select a Control resin based upon its intended end-use application. These resins are to be used to make both the Control flake and the Innovation bottle flake that will contain the additive, coating, label, adhesive or multilayer resin for the recycle study.

Homo Polymer HDPE
Chevron Phillips Marlex® EHM 6007
Dow UNIVAL™ DMDH-6400 NT 7
Exxon-Mobil Paxon™ HDPE AD60-007

Copolymer HDPE
Chevron Phillips Marlex® HHM 5502BN
Chevron Phillips Marlex® 9505H
Dow UNIVAL™ DMDA-6230 NT 7
Dow UNIVAL™ DMDA-6200 NT 7
ExxonMobil Paxon™ HDPE AB50-003

Labels and Adhesives

If the innovation does not involve labels or adhesives, and if it can be correctly argued that labels and adhesives have no impact on the innovation, the innovation samples can be made and processed without the presence of labels or adhesives.

1.10 Resins, Additives, Coatings, Labels, Adhesives and Multilayer Resins Evaluation Protocol, CRITICAL GUIDANCE

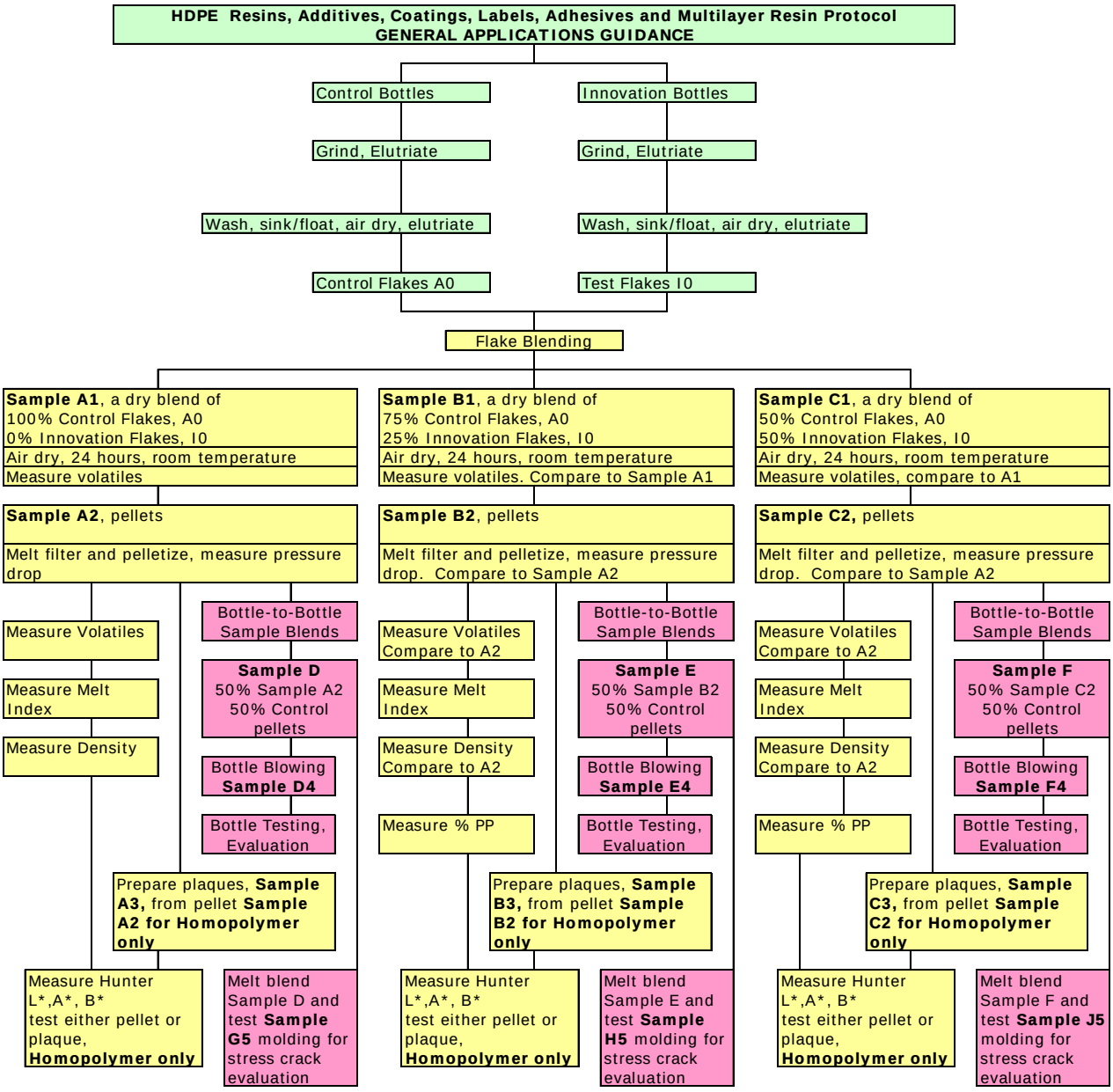
Background

The control bottles required for this test must be made from one of the APR-named control resins. The bottles can be supplied from commercial sources or blown as a separate test set and do not have to be of any special design or size. The additive, coating, label, adhesive or multilayer resin to be evaluated should be incorporated into or onto bottles made using the same control resin.

Resins, Additives, Coatings, Labels Adhesives, and Multilayer Resins Test Protocol

For all extrusion and molding steps, the process should first be established on the Control resin and then used without changes for the innovation materials. Any required processing changes for the innovation material content samples should be documented and reported. It is recognized that minor process changes may be needed and these will not be considered significant when judging the innovation materials suitability for recycling.

Resins, Additives, Coatings, Labels, Adhesives, and Multilayer Resins Evaluation Flow Schematic



Control and Innovation/Variant Bottle Manufacture

1. Control bottles should be made from one of the named resins listed in the APR Bottle Recycle Evaluation Protocol. The Innovation bottles should be made with this same resin incorporating the resin, additive, coating or multilayer resin at the intended use level.

Flake Preparation

The **Reclaim Processing Test Protocol** is to include but is not limited to the following:

1. The Control bottles and Innovation bottles should be dry-ground to nominal ¼" to ½" size flake.
2. Air elutriate to remove light fractions with one pass and with less than 2% loss set for the Control Flake. (Note: This step may be eliminated if these samples are wet ground. If omitted, more innovation failures may occur.)
3. Wash in highly agitated water at least 60° C for 10 to 15 minutes. Water may contain surfactants and have a pH of 12 to 13. Report wash solution composition.
4. Specific gravity separation in water of materials with density greater than 1.0.
5. Skim off and collect any material that is floating after the wash.
6. Air-dry flakes with no heat or vacuum
7. A second air elutriation to remove light fractions with one pass and with less than 2% loss set for the Control Flake may be carried out on the dried washed flake

Extrude, including melt filtration, to produce product pellets as below.

Sample Blending

Washed flake from number 6 or 7 above will be used to make the required Samples.

1. Prepare samples A0 and I0 above. Air dry A0 and I0 at room temperature for 24 hours, no vacuum or heat applied either as number 6 above or after number 7 above.
2. Create the following three test blends from the washed flake for study:

Sample A1:	100% Control Material, A0	0% Innovation Material, I0
Sample B1:	75% Control Material, A0	25% Innovation Material, I0
Sample C1:	50% Control Material, A0	50% Innovation Material, I0

3. Measure volatiles on flakes. For 10 grams of air-dried flake Samples A1, B1, and C1 exposed to 160°C for 10 minutes,

Guidelines comparing Samples B1 and C1 to Control A1

a. Less than 0.2% absolute difference after exposure for Samples B1 and C1 compared to Sample A1. Use moisture analyzer for measurement.

Extrusion/Pelletization

1. Dry samples A1, B1 and C1 at no more than 150 F for 10 minutes before extrusion.
2. Extrude under conditions determined by the control sample, Sample A1, using a 40/150/40 mesh screen pack.
3. Pelletize each of the sample blends, giving each its second melt history. Make Sample A2 from flake blend A1, Sample B2 from flake blend B1 and Sample C2 from flake blend C1.

Guidelines comparing Samples B2 and C2 to Control A2

a. ***No more than 10% higher pressure required on extrusion of the Innovation Samples B2 and C2 compared to the Control Sample A2 after 30 minutes of extrusion.***
b. ***The extrusion rate should be at least 500 gm/cm^2 per hour.***
c. ***No additional fuming, smoking or odors should be noticed when extruding the Innovation Samples compared to the Control.***
d. ***No build up on screen***
e. ***Resin pellets or flakes should not stick together during drying.***

Pellet evaluation

1. Measure volatiles on pellets after extrusion.
   For 10 grams of air-dried pellets Samples A2, B2, and C2 exposed to 160°C for 10 minutes.
   Guidelines comparing Samples B2 and C2 to Control A2
   a. ***Less than 0.1% absolute difference after exposure for Samples B2 and C2 compared to Sample A2. Use moisture analyzer for measurement.***

2. Measure melt index for samples A2, B2, and C2
   Guidelines
   a. ***For copolymer HDPE, 0.2 to 0.7 gm/10 minutes, Samples A2, B2, and C2***
   b. ***For homopolymer HDPE, 0.2 to 0.9 gm/10 minutes, Samples A2, B2, and C2***

3. Measure density for samples A2, B2, and C2
   Guidelines
   a. ***For copolymer and homopolymer HDPE, Samples B2 and C2 are +/- 0.010 gm/cm^3 compared to Sample A2. C2 always less than 0.995 gm/cm3.***

4. Measure percentage polypropylene for samples B2, and C2
   Guidelines
   a. ***For copolymer and homopolymer HDPE, Samples B2 and C2 are less than 2% polypropylene (total percent polypropylene in innovation bottles less than 4%).***

<u>Plaque Molding (natural homopolymer)</u>

1. Dry samples A2, B2, and C2 at no more than 150 F for 10 minutes before extrusion.
2. Injection mold 3mm plaques from the control Sample A2 first. Then mold 3mm plaques from Samples B2 and C2 under identical conditions if possible to form plaque samples

A3, B3, and C3. If the processing conditions need to be changed, then these changes must be documented and reported.

Guidelines comparing Samples B2 and C2 to Control A2 or comparing Samples B3 and C3 to Control A3

a. ***3mm Plaque Color for Sample Plaques A3, B3 and C3 or Pellets A2, B2, and C2.***
   i. ***For natural HDPE, L* should be >63 for Samples A3 and C3 or Samples A2 and C2***
   ii. ***a* greater than -4.5 for Samples A3 and C3 or Samples A2 and C2***
   iii. ***b* less than 13 for Samples A3 and C3 or Samples A2 and C2***

NOTE: color can be measured on plaques or pellets

NOTE: This completes the Critical Guidance Document requirements. If a Bottle-to-Bottle study is being performed, then continue with the following steps.

2.0 APPLICATIONS GUIDANCE

2.10 Bottle-to-Bottle Protocol (BtB)

The Bottle-to-Bottle evaluation program is designed to show processing and bottle performance differences between a control material and that control material containing recycle-content Innovation material. It is a generally *comparative* study that does not rely on the final blown bottles meeting absolute performance criteria other than minimums listed.

Since the Bottle-to-Bottle program is designed to use a one quart generic monolayer motor oil bottle the optimal study will be one where the initial control resin selected for use in the Critical Guidance Document screening will be selected from the list of APR-named control HDPE resins. The Control homopolymer virgin resin called for in Step 1 below ideally would be identical to the Critical Guidance Document control resin. It is recognized that if the Innovation being studied in the Critical Guidance Document is a copolymer resin or an additive incorporated into a copolymer resin, it may not be ideal for motor oil bottle performance. However this non-base material can still be blended with a control (selected from the table above) to begin the Bottle-to-Bottle evaluation. When this is the case, it is important to recognize that the resulting control bottles and Innovation recycle-content bottles may not perform ideally in all of the tests. 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 compatibility for the recycle stream if the bottle test criteria are met. Because the non control materials are currently found in the recycle stream, it can be presumed then any new material similar to these that does not result in significant differences in recycle-content bottle performance are, therefore, also expected to be compatible.

1. Create the following test blends of Samples A2, B2 and C2 by blending each at 50% with the Control virgin resin pellets.
   Sample D: 50% Virgin HDPE control resin pellets + 50% Sample A2. (0% innovation)
   Sample E: 50% Virgin HDPE control resin pellets + 50% Sample B2. (12.5% innovation)
   Sample F: 50% Virgin HDPE control resin pellets + 50% Sample C2. (25% innovation)

Examination One: Blow Molding of Bottles

The blends of Samples D, E and F should be blow molded into one quart straight-wall generic base monolayer "motor oil bottles". The cross-section can be rectangular or square. The bottom corners should have radii as small as commercial motor oil bottles. Bottle height should be typically for one quart motor oil bottles. Neck may be offset. The bottle must weigh 50 ± 5 grams.

Control Sample D Resin should be blown first and followed by Samples E and F each blown under the identical conditions used for Sample D if possible. If the processing conditions need to be changed, then these changes must be documented and reported.

Guidelines comparing Samples E and F to Control Sample D

- ***a. There should be no significant differences required when processing Samples E and F compared to Control Sample D. Small differences in blow mold settings are acceptable.***
- ***b. A reduction in performance of bottles made from Samples E and F should not exceed the specified test limits when compared to Sample D bottles for the following tests:***

  - ***i. Visual inspection for surface defects including 'orange peel' and other surface roughness***
  - ***ii. Bottle integrity***
  - ***iii. Bottle height***
  - ***iv. Bottle weight***
  - ***v. Capacity***
  - ***vi. Top load***
  - ***vii. Drop impact***
  - ***viii. Stress crack resistance***
  - ***ix. Fouling on tooling***

Bottle Test Guideline Criteria

Test	Measurement	Guidance
Appearance	Visual defects including surface roughness	No more than what seen for. Control D4 bottles, minimum 10 bottles.
Bottle Integrity	Examination for incomplete bottles, blowouts or pinholes or parison curling or excessive die lines or excessive flash or weak weld line or bottle warping, or incomplete pinch off	No more than what seen for Control D4 bottles, minimum 10 bottles.
Bottle Dimensions	Height	± 5% of Control D4
Thickness	Shoulder	0.012 inch minimum thickness
	Top, mid, bottom side wall	0.012 inch minimum thickness
	Base corner	0.012 inch minimum thickness
Total Bottle Weight	Weight	± 5% of Control D4
Capacity	Brimful	± 5% of Control D4
Top Load	ASTM D2659	No more than 5% decrease from Control D4
Drop Impact	Mean failure height per ASTM D2463, procedure B, Bruceton Staircase	No less than “95”% mean failure height vs. Control D4
Additional observation	Deposits on tooling	None observed vs. control for two hours of bottle making

Examination Two: Stress Crack Testing

Stress cracking is an important issue for bottles which might hold various liquids. Testing bottles themselves for stress crack performance can easily become a test of the method rather than a test of material because of how sensitive the outcome can be to how a bottle is made. For that reason testing is done per ASTM D 1693 in the latest version. A test sample must be

prepared for the Condition B (nominal 1.50 inches x 0.50 inches x 0.075 inches, or 38mm x 13 mm x 1.90 mm) and notched (nominal 0.013 deep, or 0.35 mm deep) and tested at 122°F (50°C) in the same solution of 10 to 100% by volume of Igepal CO-630 in water for all specimens.

Samples D, E, and F should be prepared with melt blended pellets to assure the same heat history as a bottle and to assure homogeneity of the test specimen. Specimens prepared may, per ASTM 1693, be compression molded or injection molded, but injection molding is preferred. Note the manner of test specimen preparation. Specimens should be notched per ASTM D 1693 Condition B. Such samples are now Control Sample G (made from Control Sample D), Sample H (made from blend Sample E), and Sample J (made from blend Sample F).

Innovation test results are after a specific number of hours of exposure, the hours to achieve 50% failure for the controls.

Guidelines comparing Samples H and J to Control Sample G

- ***a. A minimum of 20 test specimens must be examined for each sample composition. 30 or more specimens for each sample, the same number of specimens for each sample, is suggested.***
- ***b. Per ASTM D 1693, condition specimens for at least 40, no more than 96 hours before testing.***
- ***c. Per the ASTM D 1693, 3.1.2, failure is defined as "any crack visible to an observer with normal eyesight".***
- ***d. Calculate the 50% failure rate time for the Control Sample G either as the time for half of the initial control specimens to fail or graphically per ASTM D 1693 Appendix.***
- ***e. Either by count of specimens or graphically find the failure rate (number of samples failed/number of initial samples) for Samples H and J at the time for 50% failure rate of the control Sample G found in d. above..***
- ***f. The rate of failures difference for Samples H or J compared to Sample G (control) shall be no greater than 10 percentage points.***
  - ***i. The failure rate for Control Sample G should be 50% from d. above.***
  - ***ii. Failure rate difference is calculated as (number of Sample J failures/number of Sample J specimens) - (number of Sample G failures/number of Sample G specimens) to be equal to or less than 0.10 to be acceptable. This would allow two additional failures for the innovation sample more than control sample when 20 test specimens are used for each sample and three additional failures for the innovation sample more than the control when 30 test specimens are used for each sample.***

Conclusion

A Resin, Additive, Coating, Label, Adhesive or Multilayer resin that meets all of the above test criteria for the Sample C (50% innovation) and Sample F (25% innovation) and Sample J (25% innovation) would be considered technically compatible in the average reclaimer process for bottle making when introduced into the HDPE bottle recycle stream. The Innovation material may still be considered technically suitable for bottle making if the Sample F test criteria are not all met but all of the Sample C and Sample E and Sample H test criteria are met and the Innovation material under evaluation is not expected to be present in the recycle stream at the 25% innovation concentration of Sample F, even in localized recycling environments. If most, but not all, of the above test criteria are met for Samples C and F and J, further explanation or testing may be required to demonstrate technically compatibility for recycling.

Appendix A
Control resins

All Data shown in this table has been taken from what are believed to be current resin data sheets.

	density	flow rate	ESC	
APR Homopolymer Resin Listing	gm/cm^3	gm/10 min	hours	
Chevron Phillips Marlex® EHM 6007	0.964	0.65	15	ASTM D1693, Condition B
Dow UNIVAL™ DMDH-6400 NT 7	0.961	0.80	20	ASTM D 1693
Exxon-Mobil Paxon™ HDPE AD60-007	0.963	0.73	10	ASTM D1693, Condition B
APR Copolymer Resin Listing				
Chevron Phillips Marlex® HHM 5502BN	0.955	0.35	45	ASTM D1693, Condition B
Chevron Phillips Marlex® 9505H	0.949	0.34	250	ASTM D1693, Condition B
Dow UNIVAL™ DMDA-6230 NT 7	0.949	0.25	180	ASTM D1693, Condition B
Dow UNIVAL™ DMDA-6200 NT 7	0.953	0.38	80	ASTM D1693, Condition B
ExxonMobil Paxon™ HDPE AB50-003	0.950	0.30	65	ASTM D1693, Condition B