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Champions for Change Critical Issues Guidance for Innovations “HDPE Bottle Critical Guidance Document”

APR recognizes that packaging innovation drives the growth of bottles available for recycling and growth of supply of 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. Innovations can be changes in resin, additives to resin, adhesives used, layers and coatings, and labels.

This document is a subset of a larger Applications Guidance Document that provides additional guidance on testing final applications including bottles. As such, this Critical Guidance does not address the detailed questions about bottle making or other applications making and performance. Those evaluations should be conducted after the innovator is satisfied that the innovation has satisfied the intent of the guidance here offered.

This document represents a screening tool to help the innovator understand the approximate effect of the innovation on plastic bottle recycling in several concentration scenarios. It strives to accomplish the following:

- a. Limited number of critical, testable properties for HDPE bottles. Other issues may also be important. The properties listed are deliberately few and represent key concerns.
- b. Offer test samples and test methods.
- c. Recommend critical guidance values for interpreting test results
- d. Set the stage for further investigations into the effects on specific end uses after completion of this initial, critical issues examination.

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

Approved by APR Board, April 9, 2009

Edition 2

Page 1 of 12

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.

While sorting capability may address the effect of technically problematic bottles on the current stream of recyclable bottles, innovators are cautioned not to rely on either automatic sorting or dilution as justification for introducing innovations that have not been further evaluated. Through the former, new introductions may contribute to decreased yields and increased costs. The latter does not preclude the possibility of overall degradation of the recyclables stream.

This document lists testing at 0%, 25%, and 50% innovation material. The 0% innovation testing is baseline or control testing. Due to the commercial reality of variable and diverse bale content, it is advisable for innovators to consider the impacts of high levels of their innovations on the bottle reclaiming industry.

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

APR would consider a letter of recognition, upon petition, for those innovations that meet or exceed all of the strictest guidance listed. Full Recycling Guidance Recognition would follow meeting or exceeding the strictest guidance for all parts of the Applications Guidance Document.

Champions for Change HDPE Copolymer Critical Guidance

(Generally colored HDPE)

(Testing intended to represent severe occurrence in the post consumer material stream.)

THIS PROTOCOL DOES NOT PURPORT TO ADDRESS ALL OF THE SAFETY ISSUES, IF ANY, ASSOCIATED WITH ITS 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.

Test blends are defined as follows:

- Blend A: 100% Control Bottle flake made from virgin HDPE Control Resins with bottles processed to flake as indicated below. 0% bottle flake made from Innovation Bottle processed to flake as indicated below.
- Blend B: 75% Control Bottle flake made from virgin HDPE Control Resins with bottles processed to flake as indicated below. 25% bottle flake made from Innovation Bottle processed to flake as indicated below.
- Blend C: 50% Control Bottle flake made from virgin HDPE Control Resins with bottles processed to flake as indicated below. 50% bottle flake made from Innovation Bottle processed to flake as indicated below.

Control Resins

For copolymer, named HDPE Control Resins:
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

Reclaim Processing Test Protocol: Label Consideration

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, bottles can be made and processed without the presence of labels or adhesives.

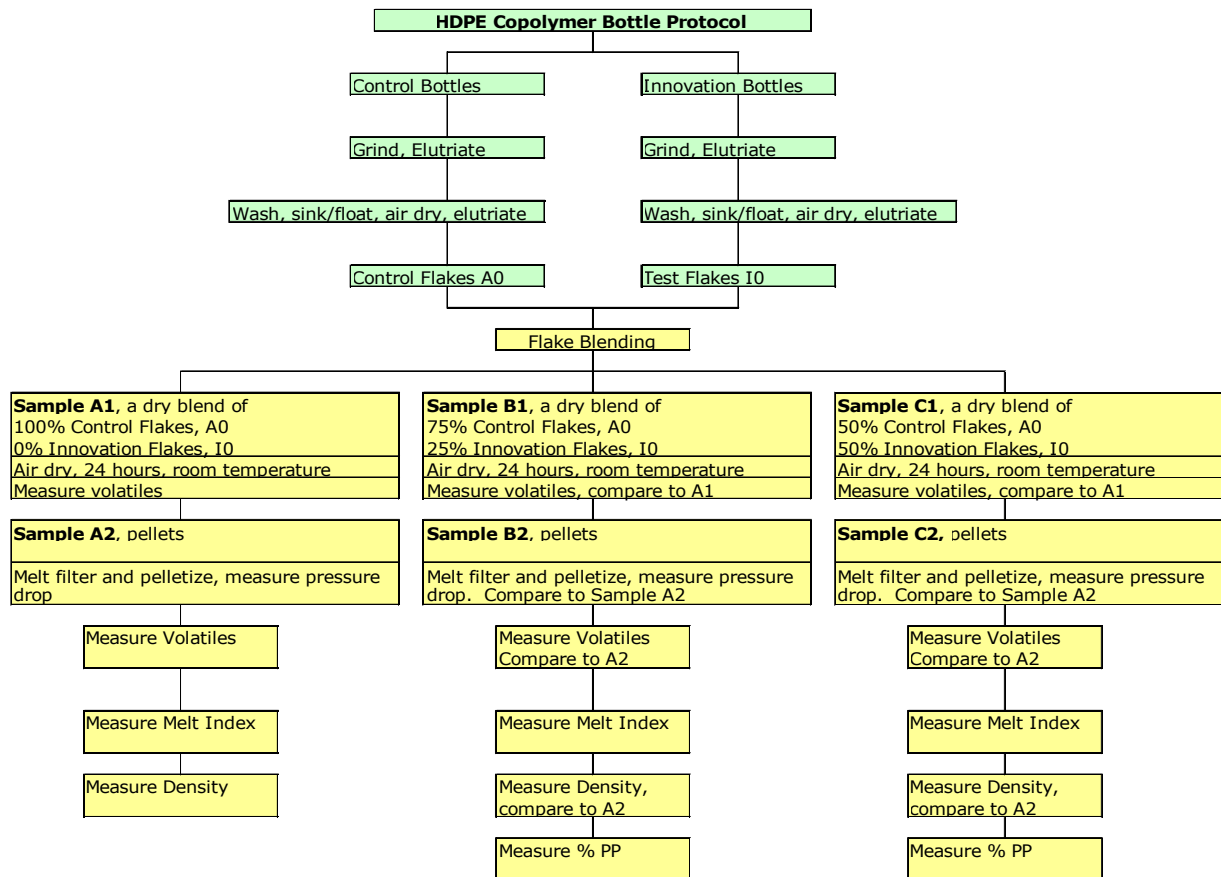
The **Reclaim Processing Test Protocol** is to include but is not limited to the following:

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.
8. Extrude, including melt filtration, to produce product pellets.

NOTE: INDUSTRY PROCESSING MAY DIFFER FROM THESE STEPS.

Evaluation Flow Schematic



Test Sample preparation

Prepare flake from Innovation Bottle (I0) and Control Bottle (A0) per the instructions above, steps 1 through 6.

Air dry A0 and I0 at room temperature for 24 hours, no vacuum or heat applied. Blend. These are Samples A1, B1, and C1.

1.0 Flake testing

1.1 Flake Volatiles

Property	Critical Guidance
Volatiles test.	For 10 grams of air-dried flake Samples A1, B1, and C1 exposed

Comparing flakes, Sample A1, B1, and C1	to 160°C for 10 minutes, less than 0.2% absolute difference after exposure for Samples B1 and C1 compared to Sample A1. Use moisture analyzer for measurement.
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1.2 Filterability Test

Property	Critical Guidance
Screen pack life. Feeding flake blends, Sample A1, B1, and C1 to make Samples A2, B2, and C2. Dry Samples A1, B1 and C1 at no more than 150 F for 10 minutes prior to extrusion.	For Sample B2 and C2, less than 10% higher pressure after extruding through 40/150/40 mesh for 30 minutes compared to Sample A2. No buildup on screen. Rate at least 500 gm/cm ² per hour. Make sufficient amounts of pellets for Critical Guidance testing and Applications testing

2.0 Pellet testing

2.1 Pellet Volatiles

Property	Critical Guidance
Volatiles test. Comparing pellets, Sample A2, B2, and C2	For 10 grams of air-dried pellets Samples A2, B2, and C2 exposed to 160°C for 10 minutes, less than 0.1% absolute difference after exposure for Samples B2 and C2 compared to Sample A2. Use moisture analyzer for measurement.

2.2 Melt Index

Property	Test Method	Critical Value
Melt Index	ASTM D1238	0.2 to 0.7 gm/10 minutes, Samples A2, B2, and C2

2.3 Density

Property	Test Method	Critical Value
Density	ASTM D1505, ASTM D792, or equivalent	Samples B2 and C2 are +/- 0.010 gm/cm ³ compared to Sample A2. C2 always less than 0.995 gm/cm ³ .

2.4 Polypropylene

Property	Test Method	Critical Value
% polypropylene	ASTM D7399	Less than 2%, measured by spectroscopic means, Samples B2 and C2. (4% maximum polypropylene in innovation bottle pellets)

3.0 General Issues

<p>Recommended Guidelines:</p> <p>No additional fuming or smoking compared to controls during extrusion</p> <p>No sticking between flakes</p> <p>No fouling of process equipment</p>
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No creation of unsafe conditions, such as increased fire potential. No generation of black specks in HDPE
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3.0 Further testing

As previously noted, this guidance does not address the detailed questions about bottle making and performance or for other applications. Those evaluations should be conducted after the innovator is satisfied that the innovation has met the intent of the guidance here offered.

The testing called for in this document is intentionally rigorous with regard to test concentrations of the innovation, 25 and 50%. In addition, APR's "Criteria to Consider When Evaluating the Recyclability of a HDPE Variant in the HDPE Bottle Stream" suggests the variant be evaluated at a multiple of the expected market penetration. The multiples suggested are between 2 to 10. A test at 5 times the expected developed market penetration is frequently used to reflect actual recycling impact.

Champions for Change HDPE Homopolymer Critical Guidance

(Generally translucent or natural HDPE)

(Testing intended to represent severe occurrence in the post consumer material stream.)

THIS PROTOCOL DOES NOT PURPORT TO ADDRESS ALL OF THE SAFETY ISSUES, IF ANY, ASSOCIATED WITH ITS 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.

Test blends are defined as follows:

- Blend A: 100% Control Bottle flake made from virgin HDPE Control Resins with bottles processed to flake as indicated below. 0% bottle flake made from Innovation Bottle processed to flake as indicated below.
- Blend B: 75% Control Bottle flake made from virgin HDPE Control Resins with bottles processed to flake as indicated below. 25% bottle flake made from Innovation Bottle processed to flake as indicated below.
- Blend C: 50% Control Bottle flake made from virgin HDPE Control Resins with bottles processed to flake as indicated below. 50% bottle flake made from Innovation Bottle processed to flake as indicated below.

Control Resins

For homopolymer, named HDPE Control Resins:
Chevron Phillips Marlex® EHM 6007
Dow UNIVAL™ DMDH-6400 NT 7
Exxon-Mobil Paxon™ HDPE AD60-007

Reclaim Processing Test Protocol: Label Consideration

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, bottles can be made and processed without the presence of labels or adhesives.

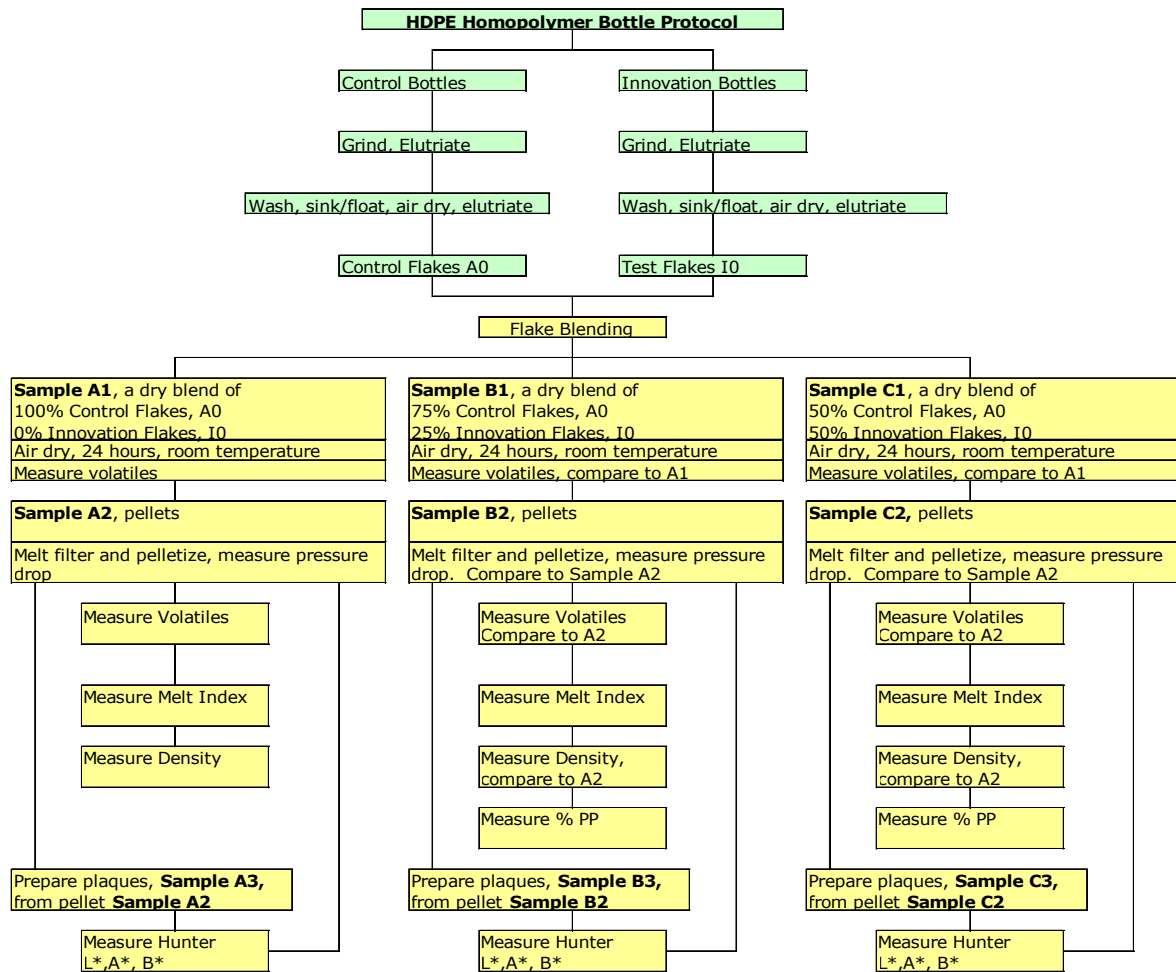
The **Reclaim Processing Test Protocol** is to include but is not limited to the following:

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.
8. Extrude, including melt filtration, to produce product pellets.

NOTE: INDUSTRY PROCESSING MAY DIFFER FROM THESE STEPS

Evaluation Flow Schematic



Test Sample preparation

Prepare flake from Innovation Bottle (I0) and Control Bottle (A0) per the instructions above, steps 1 through 6.

Air dry A0 and I0 at room temperature for 24 hours, no vacuum or heat applied. Blend. These are Samples A1, B1, and C1..

1.0 Flake testing

1.1 Flake Volatiles

Property	Critical Guidance
Volatiles test. Comparing flakes, Sample A1, B1, and C1	For 10 grams of air-dried flake Samples A1, B1, and C1 exposed to 160°C for 10 minutes, less than 0.2% absolute difference after exposure for Samples B1 and C1 compared to Sample A1. Use moisture analyzer for measurement.

1.2 Filterability Test

Property	Critical Guidance
Screen pack life. Feeding flake blends, Sample A1, B1, and C1 to make Samples A2, B2, and C2. Dry Samples A1, B1 and C1 at no more than 150 F for 10 minutes prior to extrusion.	For Sample B2 and C2, less than 10% higher pressure after extruding through 40/150/40 mesh for 30 minutes compared to Sample A2. No buildup on screen. Rate at least 500 gm/cm ² per hour. Make sufficient amounts of pellets for Critical Guidance testing and Applications testing

2.0 Pellet testing

2.1 Pellet Volatiles

Property	Critical Guidance
Volatiles test. Comparing pellets, Sample A2, B2, and C2	For 10 grams of air-dried pellets Samples A2, B2, and C2 exposed to 160°C for 10 minutes, less than 0.1% absolute difference after exposure for Samples B2 and C2 compared to Sample A2. Use moisture analyzer for measurement.

2.2 Melt Index

Property	Test Method	Critical Value
Melt Index	ASTM D1238	0.2 to 0.9 gm/10 minutes, Samples A2, B2, and C2

2.3 Density

Property	Test Method	Critical Value
Density	ASTM D1505, ASTMD792, or equivalent	Samples B2 and C2 are +/- 0.010 gm/cm ³ compared to Sample A2. C2 always less than 0.995 gm/cm ³ .

2.4 Polypropylene

Property	Test Method	Critical Value
% polypropylene	ASTM D7399	Less than 2%, measured by spectroscopic means, Samples B2 and C2. (4% maximum polypropylene in innovation bottle pellets)

3.0 Plaque/pellet testing

We believe that color testing in reflectance for pellets or plaques produces similar test values. For clean natural HDPE homopolymer we do not anticipate color being dependent on heat history. Therefore, the color guidance in Sections 3.1, 3.2, and 3.3 can be for either plaques (Samples A3, B3, and C3) or pellets (Samples A2, B2, and C2) made from Samples A1, B1, and C1.

Produce plaques A3, B3, and C3 from pellet blends A2, B2, and C2. Dry the samples A2, B2, and C2 at 150° F for 30 minutes before making plaques.

3.1

Property	Test Method	Critical Value
L*	Hunter LAB on pellets or plaques, ASTM D6290	For natural HDPE, greater than 63, Sample A3 and C3

3.2

Property	Test Method	Critical Value
A*	Hunter LAB on pellets or plaques, ASTM D6290	For natural HDPE, greater than -4.5, Sample A3 and C3

3.3

Property	Test Method	Critical Value
B*	Hunter LAB on pellets or plaques, ASTM D6290	For natural HDPE, less than 13, Sample A3 and C3

4.0 General Issues

Recommended Guidance:
 No additional fuming or smoking compared to controls during extrusion
 No sticking between flakes
 No fouling of process equipment
 No creation of unsafe conditions, such as increased fire potential.
 No generation of black specks in HDPE

5.0 Further testing

As indicated above, this guidance does not address the detailed questions about bottle making and performance or for other applications. Those evaluations should be conducted after the innovator is satisfied that the innovation has met the intent of the guidance here offered.

Notes.

Color measurement

1. Color is to be measured in reflectance from nominal 3 mm natural plaques or chips/pellets (50 +/- 10 per gram).
2. Measurements should be made with a Hunter Miniscan XE or equivalent using d65 light and 2° observation angle. The reported number should be the average of at least

four color measurements, rotating the sample container 90 degrees with each measurement.

The testing called for in this document is intentionally rigorous with regard to test concentrations of the innovation, 25 and 50%. In addition, APR's "Criteria to Consider When Evaluating the Recyclability of a HDPE Variant in the HDPE Bottle Stream" suggests the variant be evaluated at a multiple of the expected market penetration. The multiples suggested are between 2 to 10. A test at 5 times the expected developed market penetration is frequently used to reflect actual recycling impact.

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	<u>gm/cm³</u>	<u>gm/10 min</u>	<u>hours</u>	
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