Closure Test
PET-CG-02
HDPE-CG-02

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 the benefits of closures for PET HDPE and PP bottles in that the design and materials have properties for product use and life cycle. This document outlines other considerations relative to evaluating closures for their impact on PET and HDPE bottle recycling; with a special focus on the separation of closure parts that are not recycle friendly due to incompatibility of materials and recycle processing.

This following protocol does not evaluate degradable additives in the closure structure. It should be noted that this document does not include time as a variable. Innovations which include time as a factor will require separate analysis.

In addition, this document does not address the detailed questions about bottle production or other applications production and performance. APR has a separate Applications Guidance Document that provides guidance on testing for applications which may use postconsumer PET and HDPE, including bottles. It is recommended that those evaluations be conducted only after the innovator is satisfied that the innovation has satisfied the intent of the guidance herein offered.

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

- A limited number of critical, testable properties for PET and HDPE bottles that represent key technical considerations for recycling. Other issues may also be important.
- Guidance on test sample preparation and test methods.
- Guidance values for interpreting test results.

Innovators may petition APR for recognition for meeting or exceeding the most stringent guidance for all parts of this document.

The inability of an innovation to meet specified guidance 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 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 test protocol is designed to test both an "Intended" and a "Generic" closure. An "Intended" closure is a bottle combination defined as a closure, attached to a specified bottle that will be sold in the market. A "Generic" closure is one that can be used on a wide variety of bottles.

For an "Intended" closure and bottle combination, this document recommends testing at 50% Intended closure attached to the Intended bottle blended with 50% control bottle with no closure. During the grinding process the package should be ground at the intended ratios of bottle to closure.

For a "Generic" closure and bottle combination, this document recommends testing a closure and generic bottle at 100% where the closure should represent 10% by weight of the Generic control bottle weight.

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.

PET Recommended Test Protocol – SECTION 1

Safety Statement: *APR Test and Practice documents do NOT CLAIM TO ADDRESS ALL OF THE SAFETY ISSUES, IF ANY, ASSOCIATED WITH THEIR USE. These Tests and Practices may require the use of electrically powered equipment, heated equipment and molten polymers, rotating motors and drive assemblies, hydraulic powered equipment, high pressure air, and laboratory chemicals. IT IS THE RESPONSIBILITY OF THE USER TO ESTABLISH AND FOLLOW APPROPRIATE SAFETY AND HEALTH PROCEDURES WHEN UNDERTAKING THESE TESTS AND PRACTICES THAT COMPLY WITH APPLICABLE FEDERAL, STATE AND LOCAL REGULATORY REQUIREMENTS. APR and its members accept no responsibility for any harm or damages arising from the use of or reliance of these Tests and Practice documents by any party.*

1. PET Section 1 - Materials:

a. PET Control Bottle (hereinafter referred to as the "Intended Test Bottle" for the specific closure that is the subject of the testing)

i. Bottles should be made by using an APR PET control resin on the list below if possible.

Low IV, Water Bottle Innovation Controls	*CSD and Non-Water Bottle Innovation Controls*
Auriga Polyclear® Splash 3301	**Auriga Polyclear® Refresh 1101**
M&G Cleartuf® Turbo II	**M&G Cleartuf® MAX**
DAK Laser+® W L40A	**DAK Laser+® B90A**

ii. If an APR control resin is not used, the PET resin used to make these bottles should meet the PET QUICK TEST FOR COLOR:

After two heat histories, namely preform into bottle and forming into 3 mm plaques, the resin used should have a transmission CIELAB L* greater than 82 and should exhibit a b* less than 3 units greater than a plaque made from a named Control Resin also with two melt histories.

If the resin used to make the bottle is not an APR Control Resin and does not meet the conditions listed above, please discuss the situation with APR. Because this testing is designed to evaluate the closure rather than the PET resin, accommodation is possible for non-conforming PET resins.

iii. The same resin must be used for all bottles used in this evaluation.

b. Closure (hereinafter the "Innovation closure" that is the subject of the testing). For the purposes of this testing document, the closure will be defined as an article that is designed to seal a container such as a bottle, jar, or tube. The closure construction may consist of numerous components. Therefore a "closure" must be tested with all of these components in mind post consumer. The closure may be a snap fit, threaded, unthreaded, valve, gasket, child resistant,

lined or liner-less design. It may be designed to function with a feature designed to stay with the bottle such as a tamper-evident tear-away or breakaway bead. Additionally the closure may be designed to insert a pour spout, fitment, or sealing valve into the finish of the container on application. The closure may consist entirely of plastic or non-plastic components such as liners. Lined closures may be designed with compression molded, adhesively adhered, or friction-fit liners. The closure may also be designed to seal the container with an induction seal. Closures may also be dyed, pigmented, printed, or otherwise decorated. They may also carry an attachment to impart decoration or supply additional information that the closure is intended to carry along with the package such as a sticker.

2. PET Section 1 - Applicable Terms:

PET Control Bottle is defined as 100% by weight of control bottles. Any PET bottle produced from an APR approved resin is acceptable, or after passing the quick color test if not made from an APR PET approved resin.

Closure and PET Bottle Sample is the Intended Bottle with Closure.

The closure test blend shall constitute either (1) 50% intended bottle along with the intended closure with 50% approved intended control bottle (No Closure), or (2) 100% generic control bottle with a minimum of 10% by weight closure to PET control bottle.

3. PET Section 1 - Preparation of Test Samples

Sample A: PET Control Bottles

Grind the Intended Control Bottles to nominal ¼ to ½ inch size flake.

Sample B: PET Closure Test Blend

50% intended bottle with attached closure to each bottle with 50% approved intended control bottle (No Closure)
OR
100% generic control bottle with a minimum of 10% by weight of closure to PET control bottle.

a. Grind the Intended Test Blend or the Generic Test Blend closure to nominal ¼" to ½" inch size flake ensuring that no closure material is lost to static cling in the grinder. Intended bottle flake is then blended 50:50 with control bottle flake. Note that Generic bottle flake is tested at 100% with no dilution with control flake.

Closures must be applied to their respective containers prior to flake grinding if the closure either affixes or leaves a component of the closure system on the container after removal of the closure such as the following examples:

- Leaves a tamper evident band on the container after removal
- Inserts a plug or valve during application
- Incorporates an induction seal during application.

If it can be shown that the closure can be properly tested without having to apply it to container finishes prior to grinding, then the closures and containers may be ground by physically mixing them together during the grinding process. However it is suggested that if this approach is to be taken, approval from the APR Technical Director is sought prior to initiating the study.

4. **PET Section 1 - Air Elutriation**

a. A pre-wash elutriation will not be performed after grinding, in order to produce a robust-case scenario approximating a pre-wash situation.

b. Elutriate a portion of Sample A to establish an elutriation setting that allows no more than 1.2% of the PET to be carried over with closure. Save sample for subsequent testing.

5. PET Section 1 - Flake Washing

a. Prepare a wash solution of 0.3% by weight Triton X-l00 (6.0 gms or 5.7 m1 per 2,000 ml water) and 1.0% by weight caustic (20 gms NaOH per 2,000 ml water).

Note: Triton X-100 must be dissolved in warm (nominal 100°F) water prior to the addition of caustic.

b. Wash each Bottle Flake Sample separately at a ratio of 500 grams solids per 2,000 ml wash solution. Wash in highly agitated water at 88 ± 2 °C (190°F) for 15 minutes. After 15 minutes of washing, stop agitation and remove agitator. Stop heating. Let mixture of solids and solution stand for several minutes to allow floatable materials to float. Skim off floatables.

i. Note the weight of closure system removed from Bottle Flake Sample B and list as a percent of initial weight and actual weight.

c. Separate sinking solids from wash solution by pouring mixture through a strainer. Add sinking solids to room temperature rinse water at an approximate ratio of 500 grams sinking solids to 2 liters of water. Let stand for five minutes to allow remaining lights to float to the surface.

i. Note the weight of closure system removed for Bottle Flake Sample B and list as a percent of initial weight and actual weight.

d. Repeat sink/float once again.

i. Note the weight of closure system removed and list as a percent of initial weight, ___ %, and actual weight, _____ grams.

e. Transfer PET flakes to strainer, rinse flakes in cold running tap water while vigorously stirring the flakes for 10 minutes using the manual stirring bar. Drain the material.

f. Air dry flake without losing any residual closure material. Visually examine flake for the presence of closure residue and note approximate weight percentage, ___%. Examine without magnification from a distance of 12 inches using illumination typical for reading.

g. If, in the opinion of the investigator, a dye staining can make more certain the presence of closure material among PET flakes, the investigator may choose to use and discuss the staining technique.

h. Air elutriate to remove light fractions with one pass. Set up air elutriation system so that it is 1.2% as outlined in step 4b above.

i. Note the weight of closure and PET removed and list as a percent of initial weight and actual weight removed for each Bottle Flake Sample. Note the weight of closure removed and list as a percent of initial weight and actual weight.

ii. Visually examine the PET flake for the presence of closure residue and note approximate weight percentage, ___%. Examine without magnification from a distance of 12 inches using illumination typical for reading. If, in the opinion of the investigator, a dye staining can make more certain the presence of closure material among PET flakes, the investigator may so do and discuss the staining technique.

iii. If necessary for those closure materials that are less dense than water, sink/float the air separated closure material to find the weight % of closure of the total mass removed.

i. On the basis of weighed closure system material removed, calculate the amount of residual closure material still with flake of Bottle Flake Sample B

j. Retain 2 lb. samples of each variable for clumping evaluation.

6. **PET Section 1 - Clumping/Agglomeration Evaluation**

   Use the 2 lb samples of washed flake for each Bottle Flake Sample from step 5j. above. Flakes should represent product ready for desiccant drying, and have been processed to remove closure residue by washing, sink/float processing, and elutriation.

   a. Adjust the circulating oven temperature to 407 ± 5 °F (208 ± 3°C).

   b. Weigh and record the washed flake samples.

   c. Using a glass or Teflon®-lined baking dish for each washed flake sample, layer the washed flake to a depth of 1.5 +/- 0.25 inches.

   d. After 1.5 hours, remove the samples from the oven and allow to cool to room temperature without disturbing

   e. Gently transfer the contents of the pan to a sieve with 0.625 inch opening and gently shake the screen to cause single flakes to fall through. Hand remove single flakes that are oversized and unable to pass through the sieve and place with flakes that passed through. Agglomerated flake that break up during this sieving would not be deemed to be a problem.

   f. Weigh all agglomerates that cannot pass through the sieve. Include material fused to the baking dish, if any. Any flake that melts and sticks to the baking pans should be weighed and added to the weight of agglomerated material.

   g. Calculate the % of clumping as (weight of material left on sieve and in baking pan)/ (initial weight).

7. **PET Section 1 - Extrusion/Pelletization (second melt history for polymer, first melt history with closure material potentially present)**

   a. Desiccant dry Bottle Flake Samples A and B for at least 4 hours at 320 ± 20°F (160 ± 12°C) to achieve moisture below 50 ppm. Do not remove clumps of closure residue and flakes or closure residue.

   b. Extrude and pelletize the Sample A and Test Sample B separately.

c. For Samples A and B measure back pressure after extruding through 40/250/40 mesh, equal to 63 micron, for 30 minutes. Extrusion rate should be at least 375 gm/cm^2 per hour.

i. Note any fuming, smoking, or odors during extrusion, sticking between flakes during drying, fouling of process equipment, or creation of unsafe conditions, such as increased fire potential.

ii. Note any buildup on the screen pack.

iii. Measure IV of extrudate from each sample.

d. Calculate the delta IV (IV drop), extruded pellet, for Samples A to B

8. PET Section 1 - Plaque Molding (third melt history for polymer, second melt history with closure material potentially present)

a. Dry each of the pelletized Samples with desiccated air at 320 ± 20°F (160 ± -12°C) for 4 to 6 hours to achieve less than 50 ppm moisture content.

b. Injection mold a minimum of 50, 3 mm plaques from Sample A first. Then mold a minimum of 50, 3 mm plaques from Samples B under identical conditions if possible. If the processing conditions need to be changed, document and report the changes.

c. Randomly select 5 plaques from each sample for color and haze measurement.

9. PET Section 1 - Data Reporting and Guidance

a. Record Sample B amount in weight and/or ppm of any contaminates remaining after washing using a minimum sample size of 500 grams:

1. A staining for the closure material can be done so long as stained closure material is not included in any further testing of color effects on PET.

b. Record Sample B amount in weight and/or ppm of any contaminates remaining after post-wash elutriation using a minimum sample size of 500 grams:

1. A staining for the closure material can be done so long as stained closure material is not included in any further testing of color effects on PET.

c. Clumping/Agglomeration of flake

i. Examine the agglomerated weight of Samples A & B

≤1% by weight clumping (guidance value to be met)
>1% by weight (technically problematic for recycling)

d. Extrusion/pelletization

i. Report extrusion pressure for Sample A (no guidance value)

Guidance: less than 10% higher extrusion pressure for 30 minutes for Sample B and vs. Sample A. No build up on screen.

ii. Extrude at a rate of at least 375 gm/cm^2 per hour.

iii. Measure IV on each pelletized material with ASTM D 4603 and solution IV with phenol/tetrachlorethane at 30°C.

The Δ IV for Sample B vs. Sample A pellets guidance: ≤0.025 (guidance value to be met) 0.025-0.04 (further study recommended) >0.04 (technically problematic for recycling)

e. Plaque Molding (3 mm) (nominal 2 inches x 2 inches plaques), at least 50

i. Measure IV on plaques of each Sample with ASTM D 4603 and solution IV with phenol/tetrachlorethane at 30°C.

The Δ IV for Sample B plaques vs. Sample A plaques guidance: ≤0.025 (guidance value to be met) 0.025-0.04 (further study recommended) >0.04 (technically problematic for recycling)

ii. Color and Haze Measurements. Measure CIELAB in transmission on 5 randomly selected plaques for each Sample. Average results.

L* >82 for all Samples Δ b* & Δ a* of Sample B vs. Sample A guidance: < 1.5 Haze of Sample A guidance: < 9.5% Haze Haze of Sample B guidance: < 20% Haze

Note: Color Measurement

1. Measure color in transmission for color and haze using 3 mm amorphous plaques.
2. Calibrate spectrophotometer to the manufacturer's recommendations.
3. Measurements should be made with Hunter Miniscan XE or equivalent using d65 light in transmission. The reported number should be the

average of at least five color measurements of CIELAB on at least five plaques.

iii. Black Specks – 50 plaques molded each for Sample A and B viewed without magnification from 12 inches away. Count any plaque with a speck greater than 0.015 inches as failed.

Failures seen for Sample A	0	1	2	3 or more, retest
Allowed failures for Sample B	2	4	6	

1. Pass/Fail based on 5% Significance using an unpaired t-Test comparing Sample B vs. Sample A. 50 plaques of each.
2. Note if streaks of haze or color are seen in any examined plaque.
3. Also, note the presence of unmelted materials other than black specks (such as fibers of paper or metallized material) and provide a quantification of such.

iv. Fluorescence (visual, no more for Sample B than for Sample A)

v. Other observations and guidance for Sample B

1. Fuming during extrusion (no more than for Sample A)
2. Smoking during extrusion (no more than for Sample A)
3. Unusual odor during extrusion (no more than for Sample A)
4. Equipment fouling (no more than for Sample A)
5. Unsafe condition (no more than for Sample A)

HDPE Recommended Test Protocol – SECTION 2

The following protocol is designed to provide a generic processing for evaluation recycled polyolefin material. The HDPE/PP stream that is either individual or the floatable yield loss of a PET line can be evaluated. There are parts of closures that have specific concerns regarding processing such as but not limited to additives, coatings, pigments, fillers, and other non melting materials.

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1. HDPE Section 2 – Materials

a. HDPE Control Bottle (hereinafter referred to as the "Intended Test Bottle" for the specific closure that is the subject of the testing)

i. Bottles should be made by using an APR HDPE control resin on the list below if possible.

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

ii. If an APR control resin is not used it should be discussed with the director of the APR for approval.

iii. The same resin must be used for all bottles used in this evaluation.

b. Closure (hereinafter the "Innovation closure" that is the subject of the testing). For the purposes of this testing document, the closure will be defined as an article that is designed to seal a

container such as a bottle, jar, or tube. The closure construction may consist of numerous components. Therefore a "closure" must be tested with all of these components in mind post consumer. The closure may be a snap fit, threaded, unthreaded, valve, gasket, child resistant, lined or liner-less design. It may be designed to function with a feature designed to stay with the bottle such as a tamper-evident tear-away or breakaway bead. Additionally the closure may be designed to insert a pour spout, fitment, or sealing valve into the finish of the container on application. The closure may consist entirely of plastic or non-plastic components such as liners. Lined closures may be designed with compression molded, adhesively adhered, or friction-fit liners. The closure may also be designed to seal the container with an induction seal. Closures may also be dyed, pigmented, printed, or otherwise decorated. They may also carry an attachment to impart decoration or supply additional information that the closure is intended to carry along with the package such as a sticker.

2. HDPE Section 2 - Applicable Terms

HDPE Control Bottle is defined as 100% by weight of control bottles. Any HDPE bottle produced from an APR approved resin is acceptable. If not an APR HDPE approved resin it is required to discuss with the technical director for use approval.

Closure and HDPE Bottle Sample is the Intended Bottle with Closure.

The closure test blend shall constitute either (1) 50% intended bottle along with the intended closure with 50% approved intended control bottle (No Closure), or (2) 100% generic control bottle with a minimum of 10% by weight closure to HDPE control bottle.

3. HDPE Section 2 - Preparation of Test Samples

Sample C: HDPE Control Bottles

Grind the Intended Control Bottles to nominal ¼ to ½ inch size flake.

Sample D: HDPE Closure Test Blend

50% intended bottle with attached closure to each bottle with 50% approved intended control bottle (No Closure)
OR
100% generic control bottle with a minimum of 10% by weight closure to PET control bottle.

b. Grind the Intended Test Blend or the Generic Test Blend closure to nominal ¼" to ½" inch size flake ensuring that no closure material is lost to static cling in the grinder. Intended bottle flake is then blended 50:50 with control bottle flake. Note that Generic bottle flake is tested at 100% with no dilution with control flake.

Closures must be applied to their respective containers prior to flake grinding if the closure either affixes or leaves a component of the closure system on the container after removal of the closure such as the following examples:

- Leaves a tamper evident band on the container after removal
- Inserts a plug or valve during application
- Incorporates an induction seal during application.

If it can be shown that the closure can be properly tested without having to apply it to container finishes prior to grinding, then the closures and containers may be ground by physically mixing them together during the grinding process. However it is suggested that if this approach is to be taken, approval from the APR Technical Director is sought prior to initiating the study.

4. HDPE Section 2 - 1st Air Elutriation

a. Air elutriate to remove light fractions with one pass and with less than 2% loss set for the unwashed Blend C. (Note: This step may be eliminated if these samples are wet ground. If omitted, more innovation failures may occur.)

5. HDPE Section 2 - Flake Washing

a. Prepare a wash solution of 0.3% by weight Triton X-l00 (6.0 gms or 5.7 m1 per 2,000 ml water) and 1.0% by weight caustic (20 gms NaOH per 2,000 ml water).

Note: Triton X-100 must be dissolved in warm (nominal 100°F) water prior to the addition of caustic.

b. Wash each Bottle Flake Sample separately at a ratio of 500 grams solids per 2,000 ml wash solution. Wash in highly agitated water at 60°C (140°F) for 15 minutes. After 15 minutes of washing, stop agitation and remove agitator. Stop heating. Let mixture of solids and solution stand for several minutes to allow floatable materials to float.

c. Separate and save floating solids from wash solution for rinsing. Add floating solids to room temperature rinse water at an approximate ratio of 500 grams floating solids to 2 liters of water. Let stand for five minutes to allow remaining lights to float to the surface.

d. Transfer floatable flakes to strainer, rinse flakes in cold running tap water while vigorously stirring the flakes for 10 minutes using the manual stirring bar. Drain the material.

e. Air dry flake without losing any residual closure material.

6. HDPE Section 2 – 2nd Air Elutriation

a. 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.

7. HDPE Section 2 - Extrusion/Pelletization (second melt history for polymer, first melt history with closure material potentially present)

a. (Optional) Desiccant dry Bottle Flake Samples C and D for at least 10 minutes at 150°F. Do not remove clumps of closure residue and flakes or closure residue.

b. Extrude and pelletize the Sample C and Test Sample D separately.

c. For Samples C and D measure back pressure after extruding through 40/150/40 for 30 minutes. Extrusion rate should be at least 500 gm/cm^2 per hour.

iv. Note any fuming, smoking, or odors during extrusion, sticking between flakes during drying, fouling of process equipment, or creation of unsafe conditions, such as increased fire potential.
v. Note any buildup on the screen pack.
vi. Measure flow rate of extrudate from each sample.

8. HDPE Section 2 – ASTM Test Bars and/or Plaque Molding

a. (Optional) Desiccant dry Bottle Flake Samples C and D for at least 10 minutes at 150°F. Do not remove clumps of closure residue and flakes or closure residue.

b. Injection mold test samples for C and D separately.

9. HDPE Section 2- Data Reporting and Guidance (Extrusion)

Properties	Test Methods	Critical Values
Melt Index	ASTM D1238	0.2 to 0.9 g/10 minutes C and D
% Polypropylene	ASTM D7399	Sample C cannot exceed > 4% PP Sample D cannot exceed 15% PP
Density	ASTM D1505 / D792 or equivalent	Sample D to be +/- 0.010 g/cm^3 compared to Sample C. Always less than 0.990 g/cm^3
Volatiles Test		For 10 grams of air-dried pellets Samples C and D exposed to 160°C for 10 minutes, less than 0.05% absolute difference after exposure for Sample D compared to Sample C. Use moisture analyzer for measurement.
Color L*/a*/b*		Record – No Guidance

10. HDPE Section 2- Data Reporting and Guidance (Injection Molding)

Properties	Test Methods	Critical Values
Melt Index	ASTM D1238	0.2 to 0.9 g/10 minutes C and D
% Polypropylene	ASTM D7399	Sample C cannot 4% PP Sample D cannot exceed 15% PP
Density	ASTM D1505 / D792 or equivalent	Sample D to be +/- 0.010 g/cm^3 compared to Sample C. Always less than 0.990 g/cm^3
Volatiles Test		For 10 grams of air-dried pellets Samples C and D exposed to 160°C for 10 minutes, less than 0.05% absolute difference after exposure for Sample D compared to Sample C. Use moisture analyzer for measurement.
Flexural Modulus	ASTM D790	Minimum of 85,000 psi
Tensile Yield Strength @ Yield	ASTM D638	Minimum of 2,600 psi
Color L*/a*/b*	Hunter / Minolta	Record – No Guidance