

HDPE/PP Degradable Additives Test

HDPE-S-03

Introduction

APR's position is that suppliers of degradable additives have the burden of showing that their materials have 'drop in replacement' compatibility for the recycling process and for the next application with postconsumer polyethylene (HDPE and LDPE) and polypropylene (PP) not containing the degradable additives. This includes safety considerations, performance, materials handling equipment needs, and maintaining yields and processing conditions.

Degradable additives as this term is used in this guidance refers to those additives, catalytic and non-catalytic, that are intended to reduce the molecular weight of polyolefins, fragment the polymer and/or convert the carbon of the polymer to carbon dioxide or methane. These additives may or may not be compatible with polyolefin (HDPE/LDPE/PP) recycling and they may or may not affect the service life of postconsumer polyolefins.

Other APR guidance documents for polyolefins recycling do not include time as a test variable and are not designed to measure performance of applications containing recycled polyolefins over their service life. For this reason, APR's Critical Guidance and Applications Guidance documents have been modified to demonstrate compatibility.

Any testing can only examine one or a limited set of circumstances. Success at any given circumstance does not mean a system is always successful, only success at that circumstance. Therefore, a series of tests are provided in this guidance in order to specifically gain an understanding of the impact of degradable additives on current estimates for the service life of postconsumer polyolefins in specified applications. Sample preparation aging conditions suggested by this guidance are intended to simulate reasonably severe but realistic conditions for the test materials. Testing is generally in comparison to controls and also some measurements for absolute properties.

Due to the emerging nature of the degradable additive technologies, these materials are not currently part of the APR recognition program.

APR offers this guidance to provide a framework for decision making. The evaluation guidances are based on the experience of sellers of recycled plastic. In most respects the testing called for is conventional and often includes ASTM Standard Test Methods. During the initial implementation of this guidance, however, some method development and validation should be anticipated by users of the guidance concerning the accelerated aging procedures described

in the guidance, to represent the specific real time exposures. Efforts in this area will need to be determined on a case-by-case basis for each commercial degradable additive chemistry.

Disclaimer: *This document has been prepared by the Association of Plastic Recyclers as a service to the plastic industry to promote the most efficient use of the nation's plastic recycling infrastructure and to enhance the quality and quantity of recycled postconsumer plastic. The information in this document 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. APR and its members accept no responsibility for any harm or damages arising from the use of or reliance upon this information by any party. Participation in the Recognition Program is purely voluntary and does not guarantee compliance with any U.S. law or regulation or that a package or plastic article incorporating the innovation is recyclable or will be recycled.*

Specified Product Applications Covered by this Guidance

The applications addressed in this guidance document are: (1) PCR bales, (2) bottles, (3) injection molded articles, (4) extruded lumber, and (5) pipe. Each is described below.

1. PCR Bales

Bales postconsumer polyolefin packaging may be stored inside, outside on concrete slabs protected from sun exposure, or outside with full exposure to sun and rain for many months. This guidance is based on an APR survey of actual practices which showed outside storage for 8 months or more is possible and not atypical.

2. Bottles

Bottles may include 25%, but also up to 100%, recycled HDPE or PP a service life of up to 5 years may be required to assure consumer protection. Bottle performance requirements include the ability to withstand a combination of temperature, UV light, humidity, liquid water, and microbial conditions. Bottle failure may lead to incremental costs, possible injury, and loss of brand reputation.

3. Injection Molded Articles

HDPE and PP may be used for long-life injection moldings, such as for automobile ventilation/heating ductwork and fender inner liners and fairings in the engine compartment. Service lives of 20 years are expected with exposure to heat, moisture, UV, and decaying organic material (leaves) for fender liners. Failure decreases reputation and increases costs.

4. Extruded Lumber

Products may consist of up to 100% recycled polyolefin. Service life capability of 20 or more years may be expected with soil contact and weather exposure. Product quality specifications include the ability to withstand a combination of temperature, sunlight exposure, moisture, UV, and microbial contact conditions. Plastic lumber failure may cause extensive replacement costs, possible injury, and product reputation loss.

5. Extruded Pipe

Products may consist of 5 to 50% recycled polyolefin. Service life capability of 100 years may be expected with soil contact for corrugated pipe applications¹. Product quality specifications include the ability to withstand a combination of temperature, sunlight exposure, moisture, UV, and microbial contact conditions. Plastic pipe failure may cause extensive replacement costs, possible injury, and product reputation loss.

Modified Critical Guidance Testing

The following modified Critical Guidance is used to determine the compatibility of degradable additives with polyolefin recycling. Performance of one additive formulation may not be applicable to another. Each additive formulation should be examined separately at upper recommended use or specification limit concentrations.

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.*

Step 1.

Prepare polyolefin bottles or sheet with manufacturer’s recommended maximum loading of degradable additive. Bottles or 25 mil sheets are tested to allow for surface/volume exposure and material transport.

- a. Any standard (typical) bottle design may be used.
- b. Bottles or 25 mil sheet must be tested, not pellets, to allow for proper surface/volume exposure and material transport.
- c. An APR copolymer control HDPE resin should be used for HDPE bottles or sheet proxy. For sheet samples, use commercially available HDPE, LDPE, or PP resins, typical of the end use application.

APR Designated copolymer HDPE Control Resins
Chevron Phillips Marlex® HHM 5502BN
Chevron Phillips Marlex® 9505H
Dow UNIVAL™ DMDA-6230 NT 7

¹ “The service life of corrugated HDPE pipe manufactured from today’s materials is expected to exceed 100 years.” Plastic Pipe Institute. Chapter 7, 2011

Dow UNIVAL™ DMDA-6200 NT 7
ExxonMobil Paxon™ HDPE AB50-003

- d. For degradable additives that include an antioxidant, the loading of the antioxidant shall be based on commercial formulations *and must be used within the specification range specified in commercial formulations.*
- e. Prepare control bottles or sheet (without the degradable additive) in the same manner as the test bottles or sheet, using the same control resin.

Step 2.

Bales of test bottles/sheet from Step 1 are aged under identical conditions of temperature, light (UV), and moisture to simulate storage of postconsumer bales.

- a. Aging time must equal 8 months of such exposure.
- b. Conditions that simulate outdoor exposure conditions in south Florida are needed. These include the following parameters: Stored on concrete pad with roof, but no sides. Partial exposure to sun and rain. No soil contact, per APR survey.
- c. Bales should be nominal 125 pound bales.

- 1. Any accelerated aging of bottles needs to be validated to equal 8 months storage yard exposure for south Florida conditions.
- 2. Any accelerated aging must be shown valid for the system of additive and resin in the physical thicknesses of the end use as the additives involve surface transport mechanism and activity. Aging thick moldings is not the same as aging thin films. Accelerated aging must consider the multiple chemical, biological, and physical mechanisms present and operating and represent the limiting mechanism at ambient conditions. Simple time/temperature superposition must be validated before included in the test design. Any accelerated aging for catalytic or non-catalytic strictly biological systems must be validated against real time testing.
- e. Aged bottles/sheet containing the degradable additive will be tested for whether they meet or exceed the strictest Critical Guidance. Investigators should plan for more test material than needed for Critical Guidance testing when conducting the original aging because additional testing will be in order if the Critical Guidance results are first met.

Step 3.

Aged control and test bottles/sheet are processed separately to washed flake per the APR Critical Guidance washing procedure, detailed in Appendix 1.

- a. Prepare a blend of 50% aged test bottle/sheet flake containing the degradable additive and 50% control flake i.e. from washed flake made from aged bottles/sheet without degradable additive. (Blend C)

- b. The Blend C should be tested vs. 100% control bottle/sheet flake (Blend A) per the APR Critical Guidance tests for recycled HDPE, included as Appendix 1. Testing includes changes in density, volatiles, melt filtration, and melt index. Failure to meet the strictest guidance recommended by the Critical Guidance indicates that the test material is not technically compatible with current HDPE recycling conditions. Passage of the recommended Critical Guidance levels is a pre-condition to performing additional testing.

Modified Applications Guidance Testing

Bottles

Testing HDPE or PP with 25% degradable additive level vs. control. The PP control resin shall be an appropriate resin for bottle or rigid container manufacture.

1. Bottles shall be produced per the APR HDPE Bottle-to-Bottle Applications protocol. Those bottles then aged and tested per the protocol, attached as Appendix 2. Aging of bottles shall be completed in accordance with this Modified Applications Guidance Protocol, section 6 below.
2. Raw materials for control and test bottles shall be as described in Steps 1-3 above.
3. Per the APR HDPE Applications Guidance, the Bottle-to-bottle test bottles are to be produced from (1) 25% by weight aged bottles containing a 100% recommended level of degradable additive, (2) 25% by weight aged control bottles/sheet (aged without the degradable additive) and (3) 50% virgin control HDPE pellets.
4. The Bottle-to-Bottle control bottles are to be made of 50% aged raw material control bottle/sheet and 50% virgin HDPE control pellets.
5. Test bottles and control bottles shall be made per the APR HDPE Applications Guidance Document. Polypropylene bottles should be made at nominal one quart size.
6. Test and control bottles shall be aged for 5 years under nominal exposure conditions which include 50% RH, 25°C and indirect sunlight and fluorescent lighting. This five year aging cycle is consistent with required bottle service life.
7. If accelerated aging is conducted in lieu of the preceding aging protocol (subsection 6 above), equivalency of the accelerated aging conditions must be provided. Any accelerated aging must be validated to reflect nominal exposure for 5 years.
8. Aged test and aged control bottles shall be examined by the procedures described in the APR Bottle-to-Bottle protocol within the HDPE Applications Guidance Document. Polypropylene bottles shall be tested to the requirements of the APR HDPE Applications Guidance Document.

Injection Molding and Extruded Lumber and Pipe Applications

Testing shall be conducted on polyolefins with maximum recommended degradable additive level and a non-modified control (does not contain degradable additive). Long-life injection molded articles and extruded plastic lumber are tested using the same protocol which reduces the number of testing steps. The following tests apply to high density polyethylene, low density polyethylene, and polypropylene.

1. Raw material test bottles/sheet shall be made as in Steps 1-3 including aging of bales. LDPE films and PP bottles should be aged and processed to melt filtered pellets per the HDPE Critical Guidance. The LDPE and PP control resins shall be appropriate for film and bottle/rigid packaging resins.
2. Make blends.
 - i. The Test Blend shall consist of (1) 50% aged bottles/sheet containing degradable additive from Step 3 and (2) 50% unaged HDPE control copolymer resin.
 - ii. The Control Blend shall consist of (1) 50% aged bottles/sheet without degradable additive from Step 3 and (2) 50% unaged HDPE control copolymer resin.
 - iii. Blends of LDPE and HDPE should maintain the same ratios for Test and Control Blends.
3. Make tensile properties bars per ASTM D638 and flexural modulus bars per ASTM D 790 and unnotched Izod bars per ASTM D4812 for Test Blend and Control Blend. No additional antioxidants are to be added.
4. Divide bars into two sets
 - a. Age one set of bars the equal of 100 years at south Florida temperature and humidity (nominal daily cycling from 50°F to 85°F, 50% RH) with full sunlight, exposure and with damp top soil contact. Specimens placed on damp top soil would be appropriate.
 - b. Age a second set of bars the equal of 100 years at south Florida conditions of temperature and moisture, buried in moist topsoil with an inoculum present equal to that specified for ASTM D 5526 at a ratio of 1part inoculum to 10 parts dry topsoil.
 - c. Due to length of aging requirement, accelerated aging will be required. However, accelerated aging conditions must be shown to equal actual aging conditions.
 - d. Testing at 10, 25, and 50 years, or equivalent, is recommended.
5. Conduct tensile strength and modulus testing per ASTM D638 and flexural strength and modulus testing per ASTM D790 and unnotched Izod impact testing per ASTM D 790 for aged Test Blend Bars and aged Control Blend bars.
6. Calculate the mean properties for each test using the Student t test of means at 0.05 significance. The means of the two populations must not be statistically significantly different.
7. A minimum of 20 test bars for each condition are to be tested.

SUMMARY OF TEST PREPARATION AND TESTS

	Modified Critical Guidance Document Testing	Modified Applications Guidance Document - Bottle Testing	Modified Applications Guidance Document: Injection Molded Articles & Extruded Lumber
Starting material	Bottles or sheet aged for 8 months under South, Florida conditions and converted to washed flake Any accelerated aging must be validated	Pellets made from aged bottles/sheet containing normal additive level, aged control bottles/sheet, and virgin pellets. Bottles or sheet aged 8 months, Florida conditions.	Pellets made from aged bottles/sheet containing normal additive level, aged control bottles/sheet, and virgin pellets. Bottles or sheet aged 8 months, Florida conditions.
Test Blends and % degradable additive in test specimens	Test Blend is 50% aged flake with additive at 100% normal level, 50% aged control flake. Control blend is 100% aged control flake (no additive in controls). All flake from 8 month aged bottles/sheet. 50% material with normal additive level	Test Bottles made from pellets made from 25% flake with normal additive level from aged bottles, 25% control flake from aged control bottles, 50% virgin resin pellets. Control Bottles of 50% control flake from aged bottles and 50% unaged virgin resin 25% material with normal additive level	Test Bars made from pellets made from 50% flake with normal additive level from aged bottles/sheet and 50% unaged virgin resin. Control Bars made from pellets made from 50% flake with no additive from aged bottles/sheet and 50% unaged virgin resin. 50% material with normal additive level
Test specimens and aging	Flake, pellets and plaques. No additional aging of Critical Guidance test specimens.	Bottles, as made and aged 5 years in specified indoor conditions, or validated acceleration	Tensile bars, flexure bars, and unnotched Izod impact bars, aged 100 years in specified outside conditions, or validated acceleration. One set with sun and soil exposure. A second set buried in inoculated topsoil.

Tests	APR Critical Guidance. Meet or exceed all guidance for 50% additive Test Blend.	APR Bottle-to-Bottle tests on bottles aft. Meet or exceed all guidance at 25% additive level.	ASTM D 4812, ASTM D638, and ASTM D 790 with no differences of means at 0.05 significance, 50% additive level.
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Appendix 1.

Critical Guidance Testing, per APR’s HDPE Critical Guidance Document

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 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

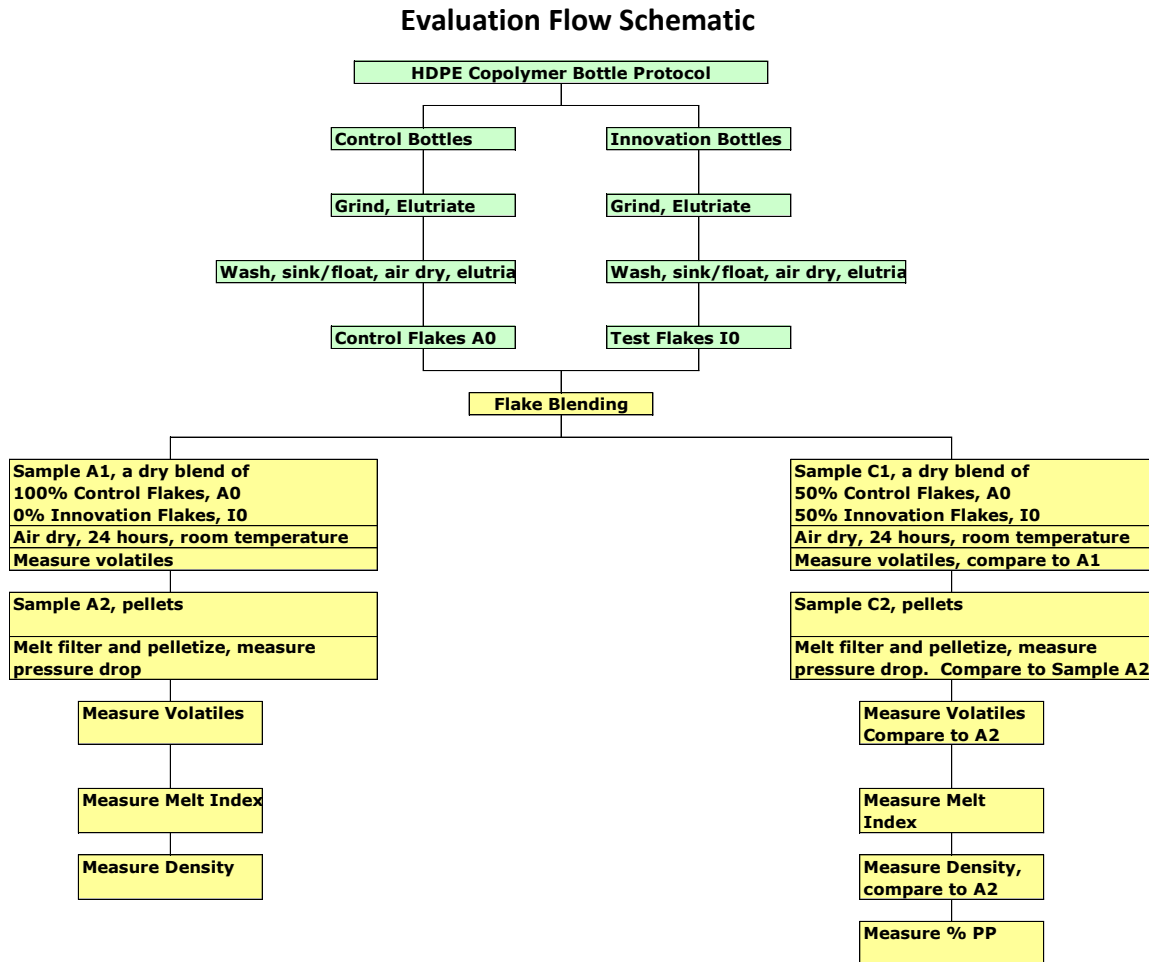
For copolymer, named HDPE Control Resins:
Chevron Phillips Marlex® HHM 5502BN
Chevron Phillips Marlex® 9505H
Dow UNIVAL™ DMDA-6230 NT 7
Dow UNIVAL™ DMDA-6200 NT 7
ExxonMobil Paxon™ HDPE AB50-003

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.



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 and C1.

1.0 Flake testing

1.1 Flake Volatiles

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

1.2 Filterability Test

Property	Critical Guidance
Screen pack life. Feeding flake blends, Sample A1 and C1 to make Samples A2 and C2. Dry Samples A1 and C1 at no more than 150 F for 10 minutes prior to extrusion.	For Sample 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. Pellet Sample A2 made from Flake Sample A1 and Pellet Sample C2 made from Flake Sample A1

2.1 Pellet Volatiles

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

2.3 Density

Property	Test Method	Critical Value
Density	ASTM D1505, ASTMD792, or equivalent	Samples 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 C2. (4% maximum polypropylene in innovation bottle pellets)

3.0 General Issues

Recommended Guidelines:

- 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

Appendix 2

APR HDPE Applications Guidance

2.0 APPLICATIONS GUIDANCE DOCUMENT

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.

Create the following test blends with Samples A2 and C2 from the Critical Guidance testing above by blending each at 50% with the Control virgin resin pellets.

Sample D: 50% Virgin HDPE/PP control resin pellets + 50% Sample A2. (0% innovation)

Sample F: 50% Virgin HDPE/PP control resin pellets + 50% Sample C2. (25% innovation)

Examination One: Blow Molding of Bottles

The blends of Samples D 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 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 F to Control Sample D

- a. There should be no significant differences required when processing Samples F compared to Control Sample D. Small differences in blow mold settings are acceptable.***
- b. A reduction in performance of bottles made from Samples 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 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) 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 Sample 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 Sample J at the time for 50% failure rate of the control Sample G found in d. above..***
- f. The rate of failures difference for Sample 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.***