PETRA
PET Resin Association

Recyclability & Innovation Model for PET Resin

October 2012

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Foreword:

Over the past decade, dramatic advances in PET resin science have led to major technical innovations and the widespread adoption of PET as a packaging material for protecting the integrity of foods and beverages. Given its unique hygienic properties and versatility, as well as its energy- and cost-efficiency, PET has become a globally valued packaging material and the world's most commonly recycled plastic.

At the same time, improved PET collection rates, advanced recycling technologies, and the growing demand for the sustainable re-use and recycling of raw materials have underscored the importance of fostering a PET recycling system that can incorporate innovation while ensuring the safety and quality of recycled PET.

The plastic recycling industry has traditionally assessed the recyclability of packaging innovations through the development of guidelines and testing protocols that evaluate the performance of innovations within typical plastic recycling systems. These assessment systems consider all elements of packaging, including closures, labels, adhesives, layers and functional additives, as well as the base PET resin.

In North America, the Association of Postconsumer Plastic Recyclers (APR) has developed its long-standing *PET Bottle Critical Guidance Document*, which focuses on evaluating innovations that will not impede or present technical challenges to the broadest range of North American recycling practices. In doing so, however, APR requires resin variants and innovations typically having a low market presence to meet the same requirements of a resin accounting for 25% or 50% of total North American PET production. In many cases, this can discourage the introduction of beneficial innovations.

In Europe, the European PET Bottle Platform (EPBP) – which represents a consortium of PET recycling, recovery and brand owner interests – recently published a *PET Recycling Test Protocol* to encourage the evaluation of innovations to meet European recycling practices and goals. The EPBP protocol utilizes a broader range of testing levels to better reflect the likely presence and performance of resins, and to assess their impact on the PET stream. However, the EPBP protocol is predicated on recycling and business systems that differ significantly from North America, making it difficult to adopt the full protocol for U.S. and Canadian use.

With neither protocol meeting the specific needs of PET stakeholders seeking to introduce new innovations in today's North American market, PETRA has developed the following *Recyclability & Innovation Model for PET Resin* as an alternative for industry use. As readers will note, the PETRA Model incorporates aspects of both the APR and EPBP protocols, but focuses solely on the base PET resin.

We believe the PETRA Model offers a more realistic and effective method for assessing recyclability that more accurately reflects the likely profile and performance of potential innovations and their impact on recycling and the quality of recycled PET. In addition, the PETRA Model calls for a monitoring system to annually test and quantify the integrity of virgin PET.

PETRA encourages the use of this model and is committed to enhancing it, based on feedback from all stakeholders, in order to support advancements in PET innovation and recycling.

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Recyclability & Innovation Model for PET Resin

The PETRA Recyclability & Innovation Model consists of four parts: Test Protocol, Dispersion Assessment, Provision of Control Resins, and Resin Supply Testing & Monitoring

I. Test Protocol

(See Diagram 1, page 7)

The Innovator will have produced bottles with the innovation resin (or additive at the intended level mixed with control resin), and will have produced bottles from 100% control resin. The control resin will be either a CSD-grade or Water-grade virgin resin generated by combining PET resins representative of the North American virgin PET supply. The control resin will be made available from PETRA at the time of testing.

The Innovator, based on his knowledge of the innovation, should assess the likely effects of its innovation at various levels in the recycle stream. Based on that assessment, the innovator should select sample Blends B and C for testing in the protocol to best represent possible effects on recycling.

Definitions of Test Blends

Blend A: 100% Control Bottle flake made from virgin PET Control Resin with bottles processed to flake as indicated in the Reclaim Processing Test (see Pg.2).

Blend B: 98% Control Bottle flake made from virgin PET Control Resins with bottles processed to flake as indicated in the Reclaim Processing Test, and 2% bottle flake made from Innovation Bottle processed to flake as indicated in the Reclaim Processing Test.

OR

90% Control Bottle flake made from virgin PET Control Resins with bottles processed to flake as indicated in the Reclaim Processing Test, and 10% bottle flake made from Innovation Bottle processed to flake as indicated in the Reclaim Processing Test.

OR

75% Control Bottle flake made from virgin PET Control Resins with bottles processed to flake as indicated in the Reclaim Processing Test, and 25% bottle flake

made from Innovation Bottle processed to flake as indicated in the Reclaim Processing Test.

Based on the Innovator's selection of the Blend B option above, the respective Blend C selected below will be used for higher concentration evaluation.

Blend C: 90% Control Bottle flake made from virgin PET Control Resins with bottles processed to flake as indicated in the Reclaim Processing Test, and 10% bottle flake made from Innovation Bottle processed to flake in the Reclaim Processing Test.

OR

75% Control Bottle flake made from virgin PET Control Resins with bottles processed to flake as indicated in the Reclaim Processing Test, and 25% bottle flake made from Innovation Bottle processed to flake as indicated in the Reclaim Processing Test.

OR

50% Control Bottle flake made from virgin PET Control Resins with bottles processed to flake as indicated in the Reclaim Processing Test, and 50% bottle flake made from Innovation Bottle processed to flake as indicated in the Reclaim Processing Test.

Blend B and Blend C should be selected respectively from the options listed so that an innovation is tested at 2% and 10%, or 10% and 25%**,** or 25% and 50%.

Reclaim Processing Test

The following protocol is from the "*APR Champions for Change, Critical Issues Guidance for Innovations*, *PET Bottle Critical Guidance Document*, Edition 5; May 2, 2011**.**

If the innovator knows of improved recycling technology for processing its innovation, the innovator may, at its discretion, complete the test protocol below using that alternate recycling technology. This can demonstrate the possibility of a higher acceptable market presence for the innovation over the longer term.

1. Grind the whole Innovation Bottles to nominal ¼ to ½ inch size flake. Separately, grind the whole Control Bottles to nominal ¼ to ½ inch size flake.

Steps 2-7 are to be performed separately for the Control resin and for the Innovation Resin

2. Utilize air elutriation to remove light fractions with one pass and with set up to accomplish less than 2% PET flake loss from the feed for Control Flake. (This step is only used for a multi-layer innovation.)

3. Prepare a wash solution of 0.3% by weight Triton X-l00 (6.0g or 5.7ml per 2,000 ml water) and 1.0% by weight caustic (20g NaOH per 2,000 ml water). Note that Triton X-100 must be dissolved in warm (nominal 100°F) water prior to the addition of caustic.

4. Mix bottle flake or pellets at a ratio of 500g solids per 2,000 ml wash solution. Wash in highly agitated (1,000 rpm with impeller 0.6 cm from bottom of wash container) water at 88+/-2°C for 15 minutes. Record the composition of the wash solution.

5. After 15 minutes of washing, stop agitation and remove agitator. Remove heating. Let mixture of solids and solution stand for several minutes to allow floatable materials to float. Skim off floatables. 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. Repeat sink/float step once again.

6. 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. Air dry flake.

7. Use air elutriation on air-dried, washed flake for one pass with set up to accomplish less than 2% PET flake loss from the feed for washed Control Flake.

Sample Preparation and Testing

1. After processing bottles through the Reclaim Processing Test defined above, blend the resulting Control bottle flake (A0) and Innovation bottle flake (I0) to create Test Blends A1, B1, and C1 at the levels chosen for the evaluation.

2 Melt extrude each test blend through 40/250/40 filtration to create A2, B2 and C2. Be sure each blend is dried for 4 to 6 hours with 340 ± 10°C air at a dew point of -20°F or lower. A moisture content of 50 ppm or less must be obtained before extrusion.

   a. IV Test Criteria (ASTM D4603 or equivalent): Compare IV drop through extrusion of samples B2 and C2 to sample A2. The delta of IV loss from the control should be ≤ 0.25 dl/g. [1]

   b. Filterability Test Criteria: Compare Sample B1 and C1 to the Control A1. Final pressure ≤10% above A1 after 30 minutes at an extrusion rate ≥ 375 g/cm^2 per hour. There should be no visible build-up on the screen.

[1.] *Intrinsic viscosity, IV, is a measured surrogate for molecular weight. If the Innovation Resin is known to have a molecular structure such that the measured IV does not represent equivalent melt rheology characteristics of PET at that IV, then the melt rheology should be measured for the Control resin, the Innovation resin, and the Blends. The Innovator then should estimate the impacts for measured melt viscosity differences at near-production conditions of temperature and shear rate to use as IV targets and differences in IV drop on melting where defined in this Model.*

3. Solid State Polymerize a portion of samples A2, B2 and C2 under identical conditions of temperature and vacuum or inert gas with temperature ≥ 205°C to create sample A3, B3 and C3.

    a. Solid State Polymerization Test Criteria: Withdraw a partial sample after 8 hours and measure IV (ASTM D4603 or equivalent). The delta between sample B3 and sample A3, and the delta between sample C3 and sample A3, should be ≤ 0.040 dl/g. Continue Solid State polymerization for 15 hours, withdraw a second sample and measure IV. The delta between sample B3 and sample A3, and the delta between sample C3 and sample A3, should be ≤ 0.075 dl/g.

    b. Melting Point Test Criteria: Using DSC set at 10°C/min. melt, crash cool and then reheat to melting. Melting point should be 235°C to 255°C for all samples.

4. Based on the solid state polymerization performance obtained from step 3, solid state polymerize additional quantities of samples A2, B2 and C2 to create samples A4, B4 and C4 that each have a measured IV of 0.80 ± 0.02 dl/g.

5. Blend pellets A4, B4 and C4 each with 50% virgin control resin pellets and injection mold into 3mm thick amorphous plaques nominally 2"x 2" square. Be sure each blend is dried for 4 to 6 hours with 340 ± 10 °C air at a dew point of -20°F or lower. A moisture content of 50 ppm or less must be obtained before injection molding. The plaques are samples A5, B5 and C5.

    a. Plaque Testing Criteria: Using CIELAB testing in transmission (ASTM D 1003-B), L* > 82.0 for A5, B5 and C5. The Δb* of both samples B5 or C5 from A5 should be ≤ 2.0. The % haze measured at 550nm should be ≤ 9.5%. Black specks counted visually without magnification on 50 plaques of both B5 and C5 should be no more that 10% above those of 50 plaques of sample A5. (Color measurements should be made with a Hunter Miniscan XE or equivalent using d65 light and 10° observation angle, a white background with specular component included and diffuse/8 sphere geometry. The values used for the test criteria should be the average of at least 5 measurements on at least 5 different plaques.)

6. No innovation sample should create fuming, smoking or odors above the control during extrusion or injection molding.

7. There should be no sticking of flakes during drying for any of the innovation samples.

8. There should be no fouling of equipment during processing of any samples, nor should there be any increased fire risk.

II. Dispersion Assessment

Based on the test results, determine at what level the innovation passes **all** listed criteria.
If the innovation does not pass at 2%, it is not recommended.

- For an innovation that passes all criteria at 2%, but not at 10%, the maximum dispersion would be ≤ 0.4%.
- For an innovation that passes all criteria at 10%, but not at 25%, the maximum dispersion would be ≤ 2.0%.
- For an innovation that passes all criteria at 25%, but not at 50%, the maximum dispersion would be ≤ 5.0%.
- For an innovation that passes all criteria at 50%, there would be no dispersion limitations.
- The corresponding dispersion volume (by weight) for the innovation equals the appropriate percentage above, multiplied by the total annual North American virgin PET resin tonnage of the previous calendar year.
- PETRA recommends completing the test protocol and dispersion assessment for all significant new resin innovations.

III. Provision of Control Resins

To ensure that recyclability assessment is based upon the actual composition of the virgin PET supply, PETRA will provide industry-representative Resin Control samples to testers.

Each PETRA member will generate two conglomerate resin samples that represent the weighted average of its previous year's (Jan-Dec) sales volume of:

- *PET resins intended for Water only, with no "functional" additives, such as barrier or UV blockers.*
- *PET resins intended for CSD only, with no "functional" additives, such as barrier or UV blockers.*

Each of these two sample types will be provided confidentially to PETRA. PETRA will then combine the submitted water-grade and CSD-grade samples to create representative industry Control Resin samples of each.

The industry Water-grade Control Resin and the CSD-grade Control Resin will be maintained by PETRA and provided to any innovator or testing facility seeking to complete the Model's test protocol.

IV. Resin Supply Monitoring & Testing

Upon prevalent industry use of this Model, PETRA will fund annual independent testing of the combined PET resin stream in order to monitor the on-going integrity of the virgin PET resin stream.

Three distinct industry-representative samples will be generated and tested as part of this combined PET resin stream monitoring:

- *Test Monitoring Sample 1 will consist of the industry representative Water-only Control Resin defined in Section III of the Model.*

- *Test Monitoring Sample 2 will consist of the industry representative CSD-only Control Resin defined in Section III of the Model.*

- *Test Monitoring Sample 3 will consist of all representative PET resin variants with and without any functional additives or barriers.*

Each PETRA member will generate and provide to PETRA these three distinct resin samples. The samples generated by each PETRA member will represent the previous year's (Jan-Dec) weighted average sales volume of each sample category.

PETRA will then combine the member-supplied samples in each category to create industry-representative Test Monitoring Samples 1, 2, and 3. If a sufficient amount of the applicable Water-only and CSD-only Control Resins are available for testing, these Control Resins will be used as Test Monitoring Sample 1 and Test Monitoring Sample 2, respectively.

The Test Monitoring Samples 1, 2 and 3 will each be tested through three heat histories, measuring IV drop, color/haze (3mm plaque) and SSP rate by an independent, accredited test laboratory.

The resulting data will be made available to the recycling community and other interested parties by PETRA.

References:

APR Champions for Change, Critical Issues Guidance for Innovations, PET Bottle Critical Guidance Document, (Edition 5, May 2, 2011)

EPBP Assessment Process Document, *PET Recycling Test Protocol* (October, 2011)

PETRA Recyclability & Innovation Model
Test Protocol

Diagram 1