Recycle studies with Eastman Aspira™ copolyester EN177

conducted December 2010

This white paper presents the study conducted to assess the recyclability of Eastman Aspira™ copolyester EN177 and its compatibility with the current PET post-consumer recycle (PCR) stream.

Executive summary and conclusions

- Eastman has developed a new polyester resin for extrusion blow molding, Eastman Aspira™ copolyester EN177, which combines superior processing and bottle aesthetics with recyclability far surpassing that of PETG copolyesters.
- Eastman Aspira™ copolyester EN177 has ease of processing and melt strength that makes it suitable for the full range of extrusion blow molding equipment used today, including shuttle, reciprocating, accumulator head, and wheel machines.
- Since the vast majority of Eastman Aspira™ copolyester EN177 is intended for clear handleware applications, it is anticipated that it will comprise less than 1% of the overall PET container volume.
- Eastman Aspira™ copolyester EN177 is not PETG. Both static and dynamic dryer testing, conducted internally and externally, demonstrates that Aspira EN177 performs approximately 25X better than PETG (and 5X better than Eastman Aspira™ copolyester EB062) regarding the level that can be tolerated in dryers before sticking or clumping becomes problematic.
- Eastman Aspira™ copolyester EN177 has been extensively tested according to The Association of Postconsumer Plastics Recyclers (APR) Critical Issues Guidance for PET Innovations. Aspira EN177 passed essentially all tests at levels up to 25% (i.e., 25X higher than the amount anticipated in the marketplace).

What is Eastman Aspira™ copolyester EN177?

Eastman Aspira™ copolyester EN177 is a polyester manufactured by Eastman Chemical Company for use in the extrusion blow molding process. It enables brand owners to develop packaging that combines innovative designs, shelf appeal, and an ergonomic use experience that will support more premium positioning, maintenance of brand identity, greater awareness and consideration on the shelf, and consumer satisfaction. The vast majority of Aspira EN177 is for clear handleware applications in the food, beverage, and consumer packaging market.

To keep the numbers in this report in perspective, it should be realized that the total amount of Eastman Aspira™ copolyester EN177 EBM bottles present in the market will be quite small compared with the amount of ISBM PET bottles. The National Association for PET Container Resources (NAPCOR) Report on Postconsumer PET Container Recycling Activity[a] notes that there were 5.3B lbs of PET bottles on U.S. shelves in 2008. Anticipated peak usage of Aspira EN177 suggests that it will be less than 1% of the national PET container volume.

Eastman Aspira™ copolyester EN177 is not PETG and is much more tolerable than PETG in the PET recycle stream. As such, it can be used as a replacement for PETG or a replacement for extrusion blow molded PVC.

While this study attempts to assess the impact of Eastman Aspira™ copolyester EN177 in the PET PCR stream derived from traditional consumer sources, Eastman recognizes that converters and brokers may occasionally have need for disposition of high concentrations of EBM bottles (>25% of recycle composition). In these instances, Eastman will provide guidance to direct those bottles to appropriate high-value end uses.

[a] *www.napcor.com/plastic/bottles/reports.html*

APR PET Critical Guidance Document protocol

Typical PET post-consumer recycle procedures

Recycling is not a charitable event. Recyclers are ultimately attempting to convert waste into profitable products. Although a few end-use products (such as plastic lumber) can utilize commingled plastic materials, the vast majority of end-use products require well separated incoming materials to make their operations profitable. Generally, recyclers will not hesitate to send poorly sorted or contaminated incoming materials into the landfill – while acknowledging the potential for lost profit. To maximize the profitability of their operations, recyclers demand high-quality incoming materials.

Most of the plastic bottles recycled today originate from curbside collection programs. Although some programs require that homeowners or collection crews separate materials based on resin identification code (RIC), many curbside collections are "single source" (i.e., commingled glass, aluminum, steel, paper, cardboard, and plastic). Single source collection relies on the material recovery facility (MRF) to perform basic separation and has the greatest potential for cross-contamination. Higher purity source materials are found where municipalities or states have mandated bottle deposit laws or use drop-off centers, but some cross-contamination can occur even in these situations.

Most plastics are sorted at the bottle (not flake) level, and this operation generally takes place at the materials recovery facility (MRF) or at an intermediate processing center (IPC). Sorting of commingled recyclable materials is an especially labor-intensive and expensive effort since the plastic must first be separated from the glass, aluminum, steel, paper, and cardboard. Sorting of whole bottles can occur manually (by visual inspection) or automatically (via optical detection systems).

In manual sorting, flattened and crushed bottles are passed on a conveyor in front of a team of human observers. The operators attempt to separate acceptable bottles via shape, color, and/or product recognition. However, human error naturally limits the accuracy of this method. The containers, crushed to reduce the cost of transportation, now are virtually unrecognizable to the human eye. In addition, some bottles of the same design may be fabricated from different plastic polymers.

In automatic sorting, machines utilizing an X-ray, visual light, and/or near-infrared analysis, can identify and separate plastic bottles based on color, resin type, or both. Automatic sorting can greatly improve the quality and efficiency of the separation process, but the technology may be more expensive than an MRF can afford and is still not 100% efficient.

Most plastic bottles contain a resin identification code (RIC) which supposedly facilitates recycling efforts, but in reality these stamped codes are of limited value to manual sorting personnel, as the rate at which the bottles pass on the conveyor precludes looking at the bottom of every bottle passing by. Slowing the conveyor rate would render the system unprofitable. These codes are also meaningless to automatic sorting equipment. Thus, these codes merely reflect the general recycle stream in which a material is best suited.

After sorting, the bottles are ground into flake. The flake is then air elutriated to separate light material, such as labels, from the heavier flake. The flake is then washed to remove any remaining dirt, ink, and glue. The cleaned flake is then run through a water bath to further separate any residual material from caps, basecups, attachments, etc., that might have been on the bottle. Materials with a density greater than one are separated from those below one in this process. This process separates the PP caps from PET, for example. Note that it is also possible to "auto-sort" flake at this step in the process.

Sorted, ground, and washed PET PCR flake is then subjected to some or all of the following procedures, depending on the final end use. Typical end uses for PET PCR flake include fiber (43%), bottles (22%), strapping (16%), and film (14%). (Percentages are based on the 2007 NAPCOR Report on Postconsumer PET Container Recycling Activity.) It is in these steps where novel materials (Innovative Resins) could cause issues.

- **Drying**
  - PET PCR flake is typically dried at 160°C for four hours, and the new material must not cause flakes to stick to the walls of the dryer or cause clumping.
- **Pelletizing**
  - The new material must not change the IV after pellitization or compromise the ability to filter contaminants during pelletization.
- **Solid stating**
  - The new material must not change the solid state rates needed to obtain the specified final IV of the PCR-based product.
- **End use**
  - The color, haze, and amount of black specs of the final PCR product must not significantly increase due to introduction of the new material. The melt point and crystallizability of the final PCR-based product must also not be affected.
  - The manufacturability and properties of the primary end uses (fiber, strapping, film, bottle) must not be adversely affected.

Protocol guidance

The Association of Postconsumer Plastic Recyclers (APR) has developed a PET Critical Guidance Document protocol that helps an innovator evaluate many of the potential concerns that could occur (i.e., solid-state rate, melt point, color, haze, black specks, etc.) when new resins are introduced into the PET recycle stream. The current protocol is similar to that proposed by PETCORE in Europe and calls for the following testing

- Selection of an appropriate control material, as recommended by the APR
- Caustic washing of the control and Innovation Resin
- Separate extrusions of the control resin and Innovation Resin at conditions that would simulate their respective primary use processing conditions
- A second extrusion of the control resin and mixtures of the Innovation Resin blended at 25% and 50% in the control resin
- Solid-stating of the extruded control resin and extruded mixtures
- Molding of the solid-stated control and innovation mixtures into plaques
- Assessment of any differences between the control and innovation blend properties (IV, melt point, color, haze, processability, safety, etc.)

A second portion of testing established by the APR is the Applications Guidance protocol, often referred to as the bottle-to-bottle (BtB) evaluation program. The BtB testing is designed to show processing and bottle (2L) performance differences between the control material and a mixture of the Innovation Resin blended at 25% and 50% in the control resin. It should be noted that in BtB testing the innovation mixtures are further diluted (simulating field use), by blending with 50% virgin control, to produce 25% and 12.5%, respectively. This evaluation is intended to examine processing differences (IV loss, black speck formation, and acetaldehyde generation) as well as bottle performance variation (section weights, burst strength, shelf-life analysis, stress cracking, and thermal stability). It is a comparative study that does not rely on the final blown bottles meeting absolute performance criteria.

Both the Critical Guidance Document and BtB test protocols suggest if the new material is intended for use in ISBM applications, it should be tested after blending at 25% and 50% levels. However, initial experiments with Eastman Aspira™ copolyester EN177 and PET PCR suggested that it cannot pass at the 50% level (due to the melt point reduction not meeting Critical Guidance requirements). Consequently, this study by Plastics Forming Enterprises, LLC (PFE) was conducted instead, at 10% and 25 % Aspira EN177 in the blends (5% and 12.5% in the BtB, per testing standards). In both cases, the standard Critical Guidance and BtB test methods were applied, and the results were measured for variation against the control resin's performance.

The Critical Guidance protocols were tested twice, with two different APR recommended controls (DAK L44A water bottle resin [.75 ItV] and Invista 1101 [0.71 ItV]. For simplicity, the application guidance testing was only conducted once with the Invista 1101 control resin.

Table 1 provides a general summary of all critical guidance and application guidance testing conducted with the Innovation Resin, Eastman Aspira™ copolyester EN177.

Table 1 Summary of results – critical guidance tests

APR Test	Parameter examined	Results			
Causti wash, pellets	Shift in b* color	Pass			
First extrusion	IV retention	Pass			
		Results for blends			
		Control resin: DAK L44A		Control resin: Invista 1101	
		10% blend	25% blend	10% blend	25% blend
Second extrusion	IV retention	Pass	Pass	Pass	Pass
	Filtration pressure	No increase	No increase	Pass	Pass
	Extrusion rate	Pass	Pass	Pass	Pass
Solid stating	IV build, 8 hr	Pass	Pass	Pass	Pass
	IV build, 15 hr	Pass	Pass	Pass	Pass
	SS to 0.80 ItV	Pass	Pass	Pass	Pass
Plaque molding	IV retention	Pass	Pass	Pass	Pass
	Shift in b* color	Needs study	Needs study	Needs study	Needs study
	Haze	Pass	Pass	Pass	Pass
	Black specks	Pass	Pass	Pass	Pass
DSC analysis	Melting point	Pass	Pass	Pass	Pass
Other observations	Fuming, smoking, or odor	Pass	Pass	Pass	Pass
	Equipment fouling/safety	Pass	Pass	Pass	Pass
Drying	Pellet sticking	Differences noted			
Bottle-to-bottle testing (virgin control is Invista 1101)					
				Control resin: Invista 1101	
				10% blend	25% blend
Preforms molding	Processing variation			Pass	Pass
	IV retention			Pass	Pass
	Change in AA level			Pass	Pass
	Black specks/gels			Pass	Pass
Blow molding 2L bottles	Processing variation			Pass	Pass
Appearance	Black specks/particulates/gels			Pass	Pass
Bottle dimensions	Height/diameter (upper, lower)			Pass	Pass
Section weights	Base/panel/shoulder			Pass	Pass
Total bottle weight	Weight variability			Pass	Pass
Capacity	Fill point/brimful			Pass	Pass
Burst strength	Burst pressure			Pass	Pass
Top load	Max load empty			Pass	Pass
Drop impact	Number of failures			Pass	Pass
Stress crack testing	Time to failure			Pass	Pass
Shelf-life	CO2 loss and septum test			Pass	Needs study
Thermal stability	Height			Pass	Pass
	Change in fill-line			Pass	Pass
	Upper panel increase			Pass	Pass
	Lower panel increase			Needs study	Needs study
	Number of rockers			Pass	Pass

Comments on critical guidance results

Following the test methods and procedures described in the APR Critical Guidance Document protocol and BtB testing, the blends of each control with 10% and 25% Eastman Aspira™ copolyester EN177 produce results that fall almost entirely within guidance values (indicated by "Pass"). There were only a few notable exceptions, which are not believed to be of severe concern.

For example, when testing with the innovation blends, a decrease in extrusion pressure was noted. However, a mere drop in extrusion pressure is not an inherently negative result. In reality, it is usually only a minor process variation with little consequence. Furthermore, it is important to emphasize that the Innovation Resin "passed" the test when used with a different control (Invista 1101). This illustrates that even the APR protocols have some statistical margin of error, that is not well defined. Other notable test exceptions were a slight shift in the b* color, a small decrease in the lower panel weight of the 2L bottle, and an elevated CO_2 loss during shelf-life testing (only at 25%). In every case, further study would be required to better understand the detailed reasons for encountering differences in these few tests. Nevertheless, given that there is some unknown level of intrinsic testing variability, stemming from both the test method and choice of control resin, it is doubtful that any of these minor exceptions directly imply adverse effects on the PET recycle stream. Finally, note in the chart that the dryer test revealed some pellet sticking differences. Drying (without sticking) is a crucial part of an industrial recycling operation. Consequently, this topic is explored more thoroughly in the next section.

In addition, it is imperative to emphasize that Eastman Aspira™ copolyester EN177 is intended for EBM handleware applications and is expected to be present in the marketplace at less than 1%, relative to PET. As noted in the APR Critical Guidance Document

> The testing called for in this document is intentionally rigorous with regard to test concentrations of the innovation 25% and 50%. As stated in the preamble, "Inability of an innovative bottle to meet specific critical values does not imply recycling failure, but should be a clear message that a significant issue might exist under certain circumstances and mitigation of the issue may be needed to avoid degrading the value of the stream of recyclable bottles." APR's criteria to consider when evaluating the recyclability of a PET variant in the PET bottle stream suggests the variant be evaluated at a multiple of the expected market penetration. The multiples suggested are between 2 and 10. A test at 5 times the expected developed market penetration is frequently used to reflect actual recycling impact.

Using this guidance, a 25% loading of Eastman Aspira™ copolyester EN177 in PET would represent a 25X multiple of the anticipated market penetration. As such, the largely acceptable results (i.e., "Pass") presented for the Critical Guidance and BtB test protocols are particularly compelling.

Dryer evaluations

Throughout the development of Innovative Resins, Eastman has long respected the recycling industry's concerns about low melting materials "clogging" a dryer. Therefore, in addition to the APR Critical Guidance and Application Guidance protocol testing, Eastman has conducted numerous internal and external dryer studies designed to mimic the setups at many recyclers. Drying is a critical part of the recycling process. PET PCR flake is typically dried at the same drying conditions as virgin PET (160°C) prior to pelletizing. It is well known that PETG becomes tacky when dried higher than 70°C because it will not crystallize. At 160°C, PETG sticks to the sidewalls of the dryer and also creates clumps of PCR that will not convey from the dryer. By contrast, Eastman Aspira™ copolyester EN177 will crystallize during the drying process and minimize tacking. Aspira EN177 is not PETG.

Ultimately, whether or not PET PCR flake "sticks and clumps" is a complex function of a number of variables. These include incoming flake geometry, percent amorphous content, bulk density, well-controlled dryer temperatures, height of the dryer, and continuity of operation (dynamic versus static). In a commercial recycling operation, it can be difficult – if not impossible – to control these variables in a manner which guarantees that "sticking and clumping" will not occur. Indeed, the recycling industry is aware that even the amorphous content of 100% ISBM PET bottle flake can occasionally cause "sticking" problems, depending upon the specific flake and process setup. To avoid this risk of a "clog," many recyclers even choose to pre-crystallize the flake in a stirring dryer (160°C –180°C). Results of two representative drying studies are presented on page 6, one for a static (or batch) drying scenario, and one for a dynamic (or moving flake) case.

Static (batch) dryer testing protocol – Plastics Forming Enterprises (PFE) study

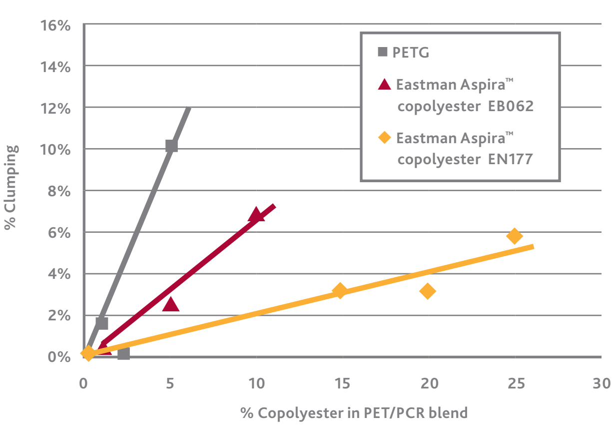
The worst case situation for "clogging and sticking" occurs during static (or batch) drying. Practically speaking, this may be indicative of "dryer startups," while the dryer slowly heats from room temperature and before the material begins to flow. To simulate this condition, "can" tests were conducted by PFE to benchmark Innovation Resins relative to historic experience with PETG. The test is conducted as follows

> Incoming material (bottles) were ground to 3/8" particle size. Ground flake was then air separated under typical recycle PET conditions for removal of any residual contaminants such as label, metals, etc., in the PCR material. Material was then blended with a combination of the recycled PET flake (RPET) and innovation flake at the predetermined levels. Material blends were placed in a specifically designed can with 175 lbs (1.2 lb/in^2) of weights placed on top to simulate the loading conditions present in a commercial dryer (approximately 8 ft. tall). A large dryer is preheated to 160°C (320°F) and the basket is inserted for 5 hours. After 5 hours, the weights are removed and the basket with material is allowed to cool. The basket is opened and the material is placed into a Sweco Vibrator for up to 2 minutes, allowing each material size to move through the screens and separate into >1/2" sizes. Material captured on the screen is then weighed, to estimate a percent (%) clumping. From Figure 1, it is apparent that Eastman Aspira™ copolyester EN177 can be blended into PCR flake at much higher quantities than Aspira™ copolyester EB062 or PETG, before clumping becomes a problem. For example, 5% clumping (arbitrarily chosen) is encountered at 2.5%, 7.5%, and 25% for PETG, EB062, and EN177, respectively.

Figure 1

Static (batch) dryer test

PFE static drying test for clumping effects, Easman Aspira™ copolyester EN177 vs. PETG, showing the superior performance of EN177.



Dynamic dryer testing protocol – Eastman study

A more typical dryer situation, like may be encountered at many recycling facilities, has flake constantly moving through the dryer in a plug flow manner. In order to better simulate this plug flow drying situation, a test protocol was developed by the Eastman lab and was conducted as follows

> A 21ft^3 mass flow drying hopper was loaded with PCR flake from United Resource Recovery Corporation (URRC) and set at 160°C. The dried flake was discharged at 180 lbs/hr via a vacuum loader while the dryer was refilled with the test blend, exposing the blend to the 160°C drying temperature for 4 hours. The dryer was kept full during the entire evaluation. The discharge orifice was 3" in diameter. The material discharged from the dryer was passed over a ½" screen to capture any clumped material. When the test was concluded, the dryer was also opened to observe any sticking on the walls. Figure 2 and Photo 1 illustrate the testing setup.

Figure 2

PCR dryer test arrangement

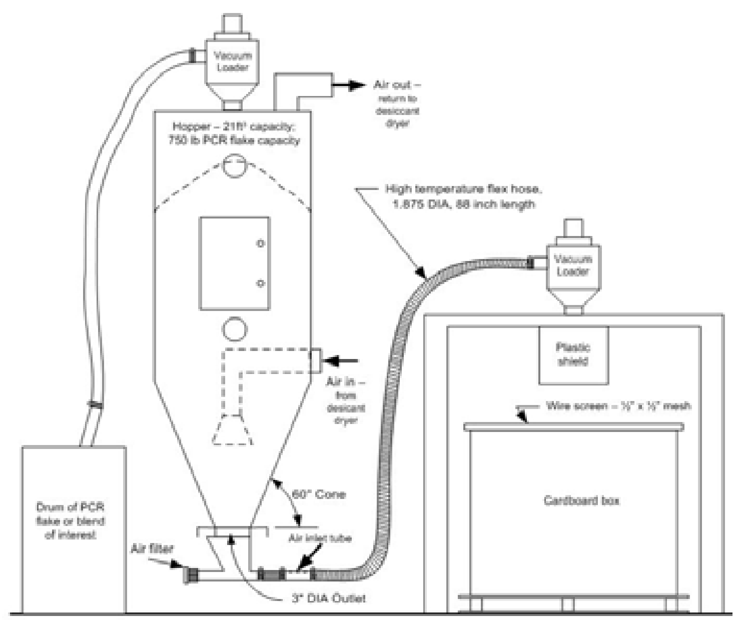


Photo 1

Dryer test arrangement

Photo 2

Flake clumps caught on screen

As shown in Photos 2-4 and Figure 3, the results of this test again show that Eastman Aspira™ copolyester EN177 can be blended into PCR flake at much higher quantities than Eastman Aspira™ copolyester EB062 or PETG before clumping becomes a problem. Under these drying conditions, 1% of PETG in the PET PCR blend created so much clumping that the flake material could barely be conveyed from the dryer. By comparison, Aspira EB062 shows significant clumping just above 5% loading, while Aspira EN177 exhibits minimal clumping at 25% levels in the PCR flake blend. Also notable is that significant amounts of PETG were found to stick to the dryer sidewalls (Photo 3), whereas that was not the case for Aspira EN177 (Photo 4). Summarizing the results of the dynamic test, Aspira EN177 at 25% in the blend

- Did not impede flow from the dryer
- Exhibited no residual sticking to the dryer walls (Photo 4)
- Generated virtually no significant clumping (Figure 3)

This dynamic test supports the results presented previously for the static PFE "can" test presented on page 6. Both experiments indicate that Eastman Aspira™ copolyester EN177 performs much better than Eastman Aspira™ copolyester EB062 in a commercial dryer under PCR flake drying conditions (160°C). Furthermore, the dynamic drying study demonstrates that that the rapid crystallization of Aspira EN177 makes it even less prone to tacking in the dryer than PETG under plug flow dryer conditions compared with static conditions. The dynamic dryer test suggests that PCR flake may flow without problem (clumping, sticking, blockage) - even when containing levels of Aspira EN177 as high as 25%. However, it is important to emphasize that this performance cannot be guaranteed in every practical drying situation. This is primarily due to setup variation within the industry, which cannot be as precisely controlled as the testing presented here.

Photo 3

PCR flake with 1% PETG PCR flake with 1% PETG – inside of dryer cone after testing. Significant sticking is noted.

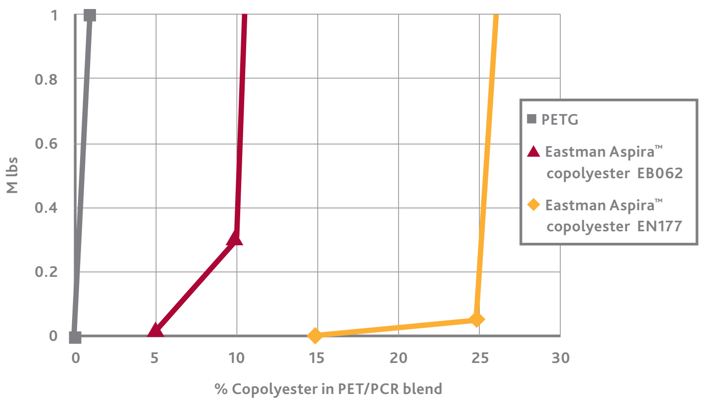
Photo 4

PCR flake with 25% Eastman Aspira™ copolyester EN177 – inside of dryer cone after testing. No sticking to dryer sidewalls is observed.

Figure 3

Dynamic (flowing) dryer test

Eastman dryer study comparison of clumping effects, Eastman Aspira™ copolyester EN177 vs. Eastman Aspira™ copolyester EB062 vs. PETG



Summary of Eastman Aspira™ copolyester EN177 in a dryer

The information provided within is intended to show the relative behavior of Eastman Aspira™ copolyester EN177 to other well established resins, such as PETG. Unfortunately, no two recycling facilities dry incoming flake identically. For example, although most recyclers dry at temperatures of 140°C-160°C, a few will dry much lower (104°C). Still other recyclers choose to solid-state flake directly at temperatures around 200°C-220°C. Because the above guidelines assume drying in the 120°C-180°C range (broadly acceptable for PET), significant deviation from these temperatures could considerably change the results. Conversely, to avoid "sticking" problems with the amorphous portion of ISBM PET bottles, many recyclers choose to pre-crystallize PET PCR flake in a stirring dryer (160°C-180°C). In this situation, with even higher levels of agitation, Aspira EN177 may behave even more favorably to what is shown here for the dynamic experiment. This was confirmed by testing Aspira EN177 in a 400-lb stirring dryer at 25% loading at Eastman's labs; no sticking or flow impediment of any kind was found.

While these smaller-scale experiments (static, dynamic, stirring) all show excellent performance for Eastman Aspira™ copolyester EN177 at 25% loading, they should not be interpreted to imply problem-free and full compatibility in every recycler's unique drying setup. Mimicking every possible dryer situation is impossible, and unfortunately there is no such thing as "typical." As such, Eastman has combined the results of simplified internal and external testing to generally suggest that Aspira EN177 is approximately 5X more compatible with PET PCR than Aspira EB062 and 25X more compatible with PET PCR than PETG. Given that the anticipated usage of Aspira EN177 is less than 1% of the overall PET volumes, Eastman Chemical Company is hopeful that our customers value the strong commitment to environmental awareness and sustainability, by the development of packaging resins with an increasingly strong recyclability message.

EASTMAN

Eastman Chemical Company

Corporate Headquarters
P.O. Box 431
Kingsport, TN 37662-5280 U.S.A.

Telephone:
U.S.A. and Canada, 800-EASTMAN (800-327-8626)
Other Locations, (1) 423-229-2000
Fax: (1) 423-229-1193

Eastman Chemical Latin America
9155 South Dadeland Blvd.
Suite 1116
Miami, FL 33156 U.S.A.

Telephone: (1) 305-671-2800
Fax: (1) 305-671-2805

Eastman Chemical B.V.
Fascinatio Boulevard 602–614
2909 VA Capelle aan den IJssel
The Netherlands

Telephone: (31) 10 2402 111
Fax: (31) 10 2402 100

Eastman (Shanghai) Chemical Commercial Company, Ltd. Jingan Branch
1206, CITIC Square
No. 1168 Nanjing Road (W)
Shanghai 200041, P.R. China

Telephone: (86) 21 6120-8700
Fax: (86) 21 5213-5255

Eastman Chemical Japan, Ltd.
AIG Aoyama Building 5F
2-11-16 Minami Aoyama
Minato-ku, Tokyo 107-0062 Japan

Telephone: (81) 3-3475-9510
Fax: (81) 3-3475-9515

Eastman Chemical Asia Pacific Pte. Ltd.
#05-04 Winsland House
3 Killiney Road
Singapore 239519

Telephone: (65) 6831-3100
Fax: (65) 6732-4930

www.eastman.com

The results of **insight**™

Material Safety Data Sheets providing safety precautions, that should be observed when handling and storing Eastman products, are available online or by request. You should obtain and review the available material safety information before handling any of these products. If any materials mentioned are not Eastman products, appropriate industrial hygiene and other safety precautions recommended by their manufacturers should be observed.

Neither Eastman Chemical Company nor its marketing affiliates shall be responsible for the use of this information, or of any product, method or apparatus mentioned, and you must make your own determination of its suitability and completeness for your own use, for the protection of the environment and for the health and safety of your employees and purchasers of your products. NO WARRANTY IS MADE OF THE MERCHANTABILITY OR FITNESS OF ANY PRODUCT, AND NOTHING HEREIN WAIVES ANY OF THE SELLER'S CONDITIONS OF SALE.

TRS-280 1/11
Printed in U.S.A.