

EARLY LAB EVALUATION TEST METHOD:

Laboratory Procedure for an Initial Evaluation of the Potential Impact of Label Adhesives on PET Recycling

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fixed with either a hot melt or waterbased adhesive. • Pressure sensitive film labels, as well as pressure sensitive paper labels. For these label styles, it is highly desirable that the label and adhesive cleanly separate from the PET during the recycling process. In the specific case of film pressure sensitive labels, it is futher desirable that the adhesive remain adhered to the label material. Hot melt and acrylic emulsion adhesives are typically not compatible with PET. These adhesives can discolor when exposed to the temperatures used to dry and melt process PET.

Polypropylene films are a source of haze when mixed in recycled PET. The components of paper labels can discolor recycled PET and may cause black specks if not removed completely during the recycling process.

During the recycling process, labels are

exposed to different washing conditions. For

while on bottles in either whole bottle wash

example, labels can be exposed to a hot wash

equipment or a wet de-labeler. Bottles are wet

labels fixed with a strip of adhesive.

- Roll fed paper and polypropylene film •

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Label Adhesives on PET Recycling

Scope, Significance, and Use

onto PET packaging. Specific label and

Introduction

this procedure include:

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- Paper or film based cut and stacklabels
- surfactant. It is very desirable that adhesives Adhesives are routinely used to adhere labels that are not compatible with PET recycling cleanly separate from the PET and can be adhesive styles anticipated for evaluation using rinsed cleanly from the PET flake. Should the adhesive and label not be removed during this wash step, the adhesive, label, and inks can all become sources of contamination and

discoloration in recycled PET.

This laboratory procedure is offered by the APR to those who are developing label adhesives and desire a laboratory procedure to screen a wide variety of adhesive candidates and rank them for their potential impact to the recycling process. This lab test is intended for early innovation development testing only: this test is not used in APR Recognition Programs and is does not by itself confirm that an adhesive or label can be compatible with PET recycling. As specific examples, this test can be employed to:

granulated into flake comprising PET flakes as

well as flakes which contain PET adhered with label. The resulting flake is washed in hot

caustic wash water, typically containing a

- Evaluate the efficiency of removal of different adhesives from a PET surface.
- Visually observe the mechanism by which an adhesive is removed.
- Observe if adhesive remains attached to the label base film and detaches from the PET article
- Observe the impact of a label stock on adhesive removal.
- Observe the interaction of adhesive removed from the PET surface with the wash solution.
- Determine any impact of sample age on adhesive removal from a PET surface..



• Evaluate the sensitivity of an adhesive to wash process variables such as wash water temperature, caustic content, or degree of agitation.

A specific objective of this evaluation is to encourage development of adhesives that readily separate from the PET under mild wash conditions and do not require abrasion and high shear that can generate PET fines and yield loss when PET is recycled. This procedure offers a standard hot caustic wash condition, and encourages evaluation of innovations in milder wash conditions, specifically, lower:

- Wash temperatures
- Caustic levels
- Surfactant levels
- Wash times

This evaluation employs a magnetic stirrer providing very low agitation compared to wash conditions in other APR tests and commercial wash conditions where adhesives can be removed by friction between flakes of PET, or shear at the tip of a tank agitator blade. The ratio by weight of water to PET is also much higher than that used in a commercial process.

Materials that perform well in this evaluation can be evaluated in more detail with APR test methods PET-CG-02 or PET-B-02 for Preferred Design Recognition or Critical Guidance testing.

While this evaluation is directed to adhesives for labels, adhesives can be used on PET packaging for other purposes such as: adhering absorbent pads within thermoforms, tamper evidence seals, lidding adhesives, or adhering an attachment. This evaluation procedure can be adapted to evaluate adhesives for these applications as well.

Method Summary

Adhesive is applied to PET cut from a bottle wall or sheet of extruded PET (the choice should be dependent on what type of material the adhesive is expected to be used with). The bond area should be at least 100 cm² and only the laminated area is used in this testing. The sample is cut into squares, approximately 1 cm² in size and the sample is washed in caustic wash water contained in a beaker on a hot plate with magnetic stirrer. This evaluation can also be used to evaluate the impact of a label stock applied over the adhesive. In most cases it will be important to understand the impact of a label stock on adhesive removal.

After washing, the PET is observed for removal of adhesive and any label stock that is employed. Removal of adhesive can be evaluated gravimetrically. Those with access to FTIR equipment might employ spectroscopy to evaluate for any residual adhesive. The wash water is observed to determine the fate of any adhesive removed from the PET; is it dispersed in the wash water, floating, or sinking with the PET sample or attached to the label stock?

The method provides guidance on desirable evaluation outcomes. Appendix One provides an example test report.





These are test methods for early evaluation of adhesives offered by other plastic recycling organizations, and which have influenced the development of this APR procedure.

- EPBP QT-508 found here: <u>Microsoft</u> <u>Word - EPBP QT508 - Labels and</u> <u>adhesives (November 2022).docx</u>
- Korean Packaging Recycling Cooperative - Label Recyclability Evaluation for PET bottles found here on page 36: <u>https://kprc21482.cafe24.com/admin/bb</u> <u>s/down.php?code=eprdown&idx=7170</u> <u>0&no=1</u>
- RecyClass test method <u>RecyClass-</u> <u>Recyclability-Evaluation-Protocol-for-</u> <u>adhesives-for-labels-on-PET-bottles-</u> <u>v1.0-FINAL.pdf</u>

Equipment Required

- Camera and a well-lit neutral background for visual observation and photography of samples. Suggested background is a black cloth
- Lab scale with milligram accuracy
- 400 ml beakers to use for washing evaluations

- Hot plate with magnetic stirring capable of heating and maintaining temperatures up to 90 °C
- 4 cm stir bar
- Thermometer that can be clipped to the inside of a beaker

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- Heavy hand shears or paper cutter for cutting up a PET sheet or bottle side wall
- A hot air oven to dry and heat samples at up to 220 °C
- 50 ml beakers, aluminum weigh pans, or similar containers for samples when dried and heated in the oven
- Deionized or purified water if available, tap water is satisfactory
- Sodium hydroxide
- A surfactant can be used in the wash solution at up to 0.3% of the total caustic solution Examples are Dow Triton X-100, Dow Tergitol 15-S-9, MacDermid RP-24, or other commercial detergents or individual surfactants can be employed that are of interest in a specific evaluation. See Appendix Two for information about sourcing surfactants.
- Strainer used to separate PET pieces from wash water and for rinsing.
- Glass stir rod
- FT-IR spectrophotometer to measure non-visible left-over adhesive is desirable, but not necessary.
- An overhead mixer can be used in an optional evaluation for impact of agitation on adhesive and label removal.



Materials Required

- PET substrate Any convenient source of clear natural PET bottle wall or sheet that is similar to the expected material of use can be employed for this test. Appendix Two discusses specific sources of PET that are suitable for use in this test. 100 cm² of substrate is required for each test. Example sources of substrate can be:
 - The sidewall cut from a PET bottle
 - An amorphous extruded sheet

Note: while the method allows for the use of any convenient source of PET, investigators will want to evaluate at least a few different sources of PET bottle wall or sheet to confirm whether there is likely to be any impact of the source of the PET on adhesive wash-off results. (Lubricants used when blowing bottles, de-nesting agents on PET sheet, and degree of shrinkage of PET in a bottle wall are possible sources of variation.)

- The adhesive, or adhesives being evaluated
- Any unprinted or undecorated label stock of interest. Can be a film or paper label stock. A white pigmented film label is suggested so that the film and PET can be readily distinguished by visual examination.

- Isopropyl alcohol in a wash bottle used to clean the PET surface before applying adhesive
- Lint-free paper towels (e.g. Kimwipe)

Method Steps

A comprehensive analysis requires evaluation of the impact of an adhesive when covered by a label substrate. The label substrate can limit the access of wash water to the adhesive and the interface between the adhesive and PET. But there can be situations where it is first desirable to evaluate the wash-off characteristics of an adhesive alone. The method allows for a first evaluation of the adhesive alone, and then the adhesive that employs a label substrate.

Preparation of test samples with adhesive only

- Cut a piece of PET bottle wall or sheet with an area of 100 cm²; one can also use multiple pieces of wall/sheet totaling 100 cm² in area.
- 2. Clean the PET by rinsing with isopropyl alcohol from the wash bottle. Objective is to remove surface contamination from the PET created by handling. This alcohol wash might also rinse off any organic components that have bloomed to the surface during storage, or any lubricants that were applied to the PET surface. Allow the PET to air dry.

represents the thickness, or weight per

3. Weigh the PET substrate using a

balance with 0.001 gram sensitivity.

PET using any convenient means that

will result in a layer of adhesive that

4. Apply adhesive to only one side of the

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unit area, of adhesive that is recommended for commercial use. For illustration, spray, brush, draw-down bar, solvent casting.... Any are acceptable. If a solvent is employed, allow the solvent to fully evaporate.

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- 5. Weigh the sample with adhesive to obtain the weight of adhesive.
- 6. Place the pieces into a labeled sample bag or container for later use in wash testing.
- 7. Samples should be "aged" before use in wash testing. This method does not specify an aging time because any impact of age might be impacted by the adhesive formulation and not be the same for all materials. Laboratory investigators are encouraged to include an assessment of any impact of aging in their assessment.
- 8. Cut the sample up into square pieces that are no less than 9 mm and no greater than 12 mm in length. Cutting can be done with hand shears, or an office paper cutter, for example. These cut pieces will be referred to below as "flakes" of PET.

Note: the procedure calls for 100 cm² of PET substrate. APR recommends this surface area

for results reported publicly or for making final decisions about ranking candidate materials. But less surface area can be used if the investigator desires in initial investigations to simplify sample preparation.

<u>Preparation of samples that employ a label</u> <u>stock applied to an adhesive</u>

- Follow the steps immediately above to apply an adhesive to the PET substrate and determine the weight of adhesive. Note: if the adhesive must be wet with water or solvent to adhere the label, One can obtain the weight of adhesive by subtracting the weight of the label stock and PET sheet from the total weight of the sample laminate of PET/adhesive/label stock.
- Use any convenient means to apply a label stock over the adhesive that mimics what will be done in a production environment. For illustration:
 - a. Cut an area of label greater that 100 cm², place the label over the PET with adhesive, and use a hand roller to apply the label to the adhesive. Trim any excess label stock from the PET with hand scissors or a utility knife.
 - b. If using a hot melt adhesive, one method can be to place the PET with adhesive in an oven, or on a hot plate to heat the adhesive to the application temperature recommended for commercial use. Apply a label stock over the



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adhesive and adhere with a roller or by otherwise applying pressure to the sample laminate construction. Note that PET can deform if heated over its glass transition temperature of about 75 °C, so an alternate approach might be required for high melting point adhesives.

- Cut the sample into pieces no less than 9 mm and no greater than 12 mm is size. These cut pieces will be referred to below as "flakes" of PET.
- 4. Place the samples into a labeled bag for later use in wash testing.
- 5. Age the sample as prescribed above before wash testing.

<u>Preparation of samples when adhesive is first</u> <u>applied to the label stock.</u>

In the case of pressure sensitive labels, the adhesive is first applied to the label stock. Or there might be cases for roll-fed or cut and stack labels where the adhesive is first applied to the label, not the PET. In these cases:

- 1. Clean the PET substrate with isopropyl alcohol and lint-free paper towel.
- 2. Weigh the substrate.
- 3. Apply the label with adhesive to the substrate and adhere the label to the substrate with a properly weighted roller.
- 4. Trim any excess label material from the PET substrate.

- 5. As above, cut the laminated sample into pieces and save them in a labeled bag.
- 6. Age the sample as prescribed above before wash testing.

Note: in the case of adhesives applied to labels, it may not possible to obtain an adhesive weight. In this case, the washed PET can be weighed and evaluated for any weight gain that would indicate that adhesive was not removed.

Preparation of standard wash solution

In commercial practice, when PET packaging is recycled, bottles and/or thermoforms are granulated into flakes which are then washed in hot water that contains hot caustic made with NaOH, and a commercial proprietary detergent product. This test method recommends a starting point "standard wash" condition. Optional wash compositions are also suggested to those who desire to demonstrate adhesive performance over a wider range of wash conditions.

Standard wash solution

- Make a stock solution that can be used for all samples for a day based on 200 ml of wash solution per sample.
- 2. Tap, purified, or softened water are acceptable.
- Make this stock solution with 1.0% by wt. NaOH content. Slowly add the NaOH to the water with mixing.



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- 4. Add the desired surfactant or detergent to the solution to yield a 0.3% by wt. loading.
- 5. Heat the wash solution to 85 °C with stirring prior to starting the test.

APR Practice PET-P-04 describes making wash water solution in detail.

Alternate wash conditions

An evaluator may want to consider the impact of a range of wash conditions. After evaluating a sample with standard wash conditions, one might want to consider the impact of an adhesive with these variations:

- Lower temperature can the adhesive be removed at temperatures below 85 °C? If so, this would be a benefit to PET reclaimers since wide use of such an adhesive will allow for less energy to heat water. Also, lower removal temperatures may help label removal in process steps where whole bottles are washed in hot wash water. Adhesive and label removal at temperatures in the range of 50 to 75 °C can benefit PET reclaimers.
- Lower caustic levels addition of caustic is a materials cost, and this caustic must be neutralized with acid before wastewater disposal. Adhesives and labels that can be removed with low caustic content, perhaps in the

range of 0.25 to 0.5% by wt. can be a benefit to the recycling industry.

- Tergitol 15-S-9 is a well-known nonionic surfactant used in the cleaning industry. One might want to evaluate the impact of lower levels of Tergitol 15-S-9, such as in the range of 0 to 0.2% by wt.
- It is possible that removal of adhesive or label is impacted by the choice of surfactant. Other surfactants can be substituted for Tergitol 15-S-9 to evaluate their impact. Triton X-100 was listed in the materials section because this surfactant has widely been used in washing PET – but is used less today because it is an ethoxylated nonylphenol compound. MacDermid RP-24 is mentioned in the materials section of this procedure because it is a proprietary detergent formulation that finds wide use in the PET recycling industry. Any surfactant or commercial wash detergent is a candidate for use in this evaluation.
- Higher wash agitation this method purposefully employs a flake wash step with low agitation from only a magnetic stir bar to encourage development of adhesives that can be removed from PET under mild agitation conditions. But APR tests PET-B-02 and PET-CG-02 do allow for washing with an overhead mixer that of course provides substantially higher levels of agitation to assist label and adhesive removal.



This evaluation can be done with an overhead mixer to evaluate for impact of higher agitation if desired.

Wash procedure steps

- Add 200 ml of wash solution to a 400 ml glass beaker.
- Place the beaker on the hot plate. Add the magnetic stir bar to the beaker and turn on the stirrer. Note: this method employs a magnetic stirrer for simplicity and to encourage development of adhesives that can be removed under mild agitation. Should there be a reason that a magnetic stirrer is not effective, an overhead mixer run at low rpm can be substituted.
- Heat that wash solution to 85 °C and maintain the temperature to 80-85 °C. The wash temperature should be monitored throughout the test.
- Add the cut pieces of the PET sample to the wash solution and start a 15minute timer. Adjust the speed of the magnetic stirrer so that the flakes are in constant motion within the beaker, but not so high that there is risk of splashing or spilling of the wash solution.
- Observe the wash solution after each 5and 10-minute increment for visual evidence of removal of the adhesive, or label if employed with the sample.

- After 15 minutes, remove the beaker from the hot plate and let the water cool for five minutes. Should any label appear to be separated from the PET, but physically entangled with the PET, gently stir the wash water with a glass rod to give the label pieces an opportunity to float. Then, once again carefully observe the beaker and record any visual evidence that the adhesive was removed and its fate in the wash water.
 - Record the fate of any label used. For example: Did the label float, sink in water, or become dispersed in the water? Also observe for anything floating on the surface of the wash water or anything that has sunk in the wash water along with the pieces of PET. Did the adhesive remain attached to the label?
- Fate of the adhesive; for example: Does it float? Does it sink in water? Does the adhesive disperse in the wash water or remain as a distinct second phase? In cases when there are small amounts of adhesive applied to the PET surface, it may be difficult to observe the adhesive in the wash water, and the evaluator might simply report, no visual evidence of the adhesive in the wash water was observed.





• Use the camera to document the visual appearance of the PET flakes and adhesive in the wash water.

Recovery of the PET pieces

- Skim, or otherwise remove, any floating pieces from the wash water.
- Decant the entire remaining contents of the 400 ml beaker into a strainer to collect the sunken solids.
- Rinse the flakes by submersion into 500 ml of tepid tap water. Pour the water slowly over the flakes to remove any dispersed materials that can pass through the strainer, and to rinse the caustic from the flake. Gently tap the strainer to remove as much rinse water as possible from the PET sample.
- The appearance of any adhesive or label material mixed with the PET flake should be documented with a photograph.
- Place the rinsed PET flakes into a 50 ml glass beaker – The reason is so that the flakes are stacked together and in contact with one another, not spreadout during drying.
- One can evaluate the impact of heat on the washed flake using one or more of these options:

- Heat flake for one hour at 220
 °C to evaluate any color
 development on the flake using
 a method following PET-S-10.
 This exposure is the most highly
 recommended conditions, but
 there may bevalue in the next
 two options in some cases.
- Heat the flake at 160 °C for six hours to simulate heat exposure in a flake drier as described in PET-P-08.
- Heat flake at 195 °C for 90 minutes to evaluate for clumping as described in APR's PET-S-08.

Observing and evaluating the dried PET flakes

Make the following observations of the heated flake starting with the 50 ml beaker:

- Can the PET flakes be easily removed from the beaker, or do they stick inside the beaker? Sticking is evidence of residual adhesive still on the flakes.
- Do the PET flakes easily separate into individual flakes, or are any of the flakes clumped together as evidence of residual adhesive on the sample?
- Observe the flakes for discoloration did the heating exposure cause any residual adhesive to discolor?
- When a label stock is employed, record how many pieces of PET still have label stock adhered to the PET. For Example: 10 pieces of PET flake had adhering



label, therefore 10 out of the original 100 shows 10% of the label stock not removed.

- The camera can be used to record the appearance of samples that display either clumping or discoloration.
- Only for the case of samples tested with no label stock, or where the label stock was removed 100% by washing, weigh the dried flakes and calculate and record the weight difference from the original PET sample with no adhesive.

Measurements, Reports, and Guidance

An example evaluation report form is shown in Appendix One.

Qualitatively, the following results are desirable to benefit PET recycling:

- Complete removal of the adhesive and any label stock from the PET flakes using standard wash conditions.
- Adhesive is dispersed in the wash water and rinses cleanly from the PET flakes, or the adhesive remains adhered to the label stock, or the adhesive floats in the wash water.

- On this scale it can be difficult to ascertain the full consequences of adhesives that become dispersed into the aqueous phase, but if there is evidence of the adhesive redepositing on the PET or the equipment, it should be noted because this could cause issues in real world situations.
- Washed flakes do not clump or show evidence of discoloration after exposure to a minimum of 160 °C, but preferrable up to 220 °C for one hour.

Further, those that recycle PET packaging will appreciate adhesives that meet the above criteria along with:

- Wash temperatures lower than 85 °C.
- Caustic levels below 1% NaOH.
- Low surfactant requirements.
- Short wash times such as only 5 or 10 minutes required to remove adhesives and labels.





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DOCUMENT VERSION HISTORY

Version	Publication Date	Revision notes
1	October 16, 2024	Original



Appendix One

Example Report Form

Adhesive wash evaluation report for	Sample identification #1	Sample identification #2	Sample identification #3
Sample aging method	oample identification #1	Sample identification #2	Sample identification #3
Cample aging method			
Adesive weight and weight loss, in grams			
Weight of PET substrate with no adhesive			
Weight of PET substrate with applied adhesi	ve		
Weight of label used			
Weight of adhesive			
Weight of dried PET flakes after washing			
weight of adhesive remaining after wash			
Wt % of adhesive removed by wash			
Wash conditions			
Wash water temperature, °C			
Caustic content, wt%			
Surfactant product identification			
Surfactant content, wt%			
Observations after 5 minutes of washing			
Adhesive removal			
Adhesive appearance			
Label stock removal			
Observations after 10 minutes of washing			
Adhesive removal			
Adhesive appearance			
Label stock removal			
Observations after 15 minutes of washing			
Sector and the arter to minutes of it as ming			
Adhesive removal			
Adhesive appearance			
Label stock removal			
Observations after oven drying			
Drying temperature used (deg C)			
Any clumping?			



Appendix Two

Sourcing Materials for this Evaluation

Surfactants to use in testing

Surfactants such as Triton X-100 or Tergitol 15-S-9 are commercial products that can be sourced from chemical distributors or those that sell laboratory chemicals.

MacDermid R-24 is a commercial product that can be requested directly from MacDermid. A contact that is current as of the time this procedure was published is:

Robert Berry

Technical Manager Plastics Recycling

North America

MacDermid Enthone Industrial Solutions

245 Freight St.

Waterbury CT, 06702 USA

Rob.Berry@macdermidenthone.com

- 0 203.575.5764
- M 203.706.8966





Sourcing PET substrates to use in testing

As it relates to sourcing PET substrates for testing, here are some considerations:

- 1. This evaluation allows for testing on any PET substrate of interest; a bottle wall, a PET sheet, or a PET thermoform for common examples.
- 2. A word of caution is that not all PET surfaces may be the same with respect to adhesion properties. For example:
 - It is common to use lubricants on the surface of PET packaging. Thermoforms use lubricants so that sheets of PET can be more easily separated. Lubricants are also used on thermoforms to improve de-nesting properties.
 - PET bottles might also employ lubricants, but not all do. Lubricants are used when making PET bottles to help bottles release better from blow molding tooling as well as to prevent bottles sticking together creating jams when bottles pass through filling equipment.
 - Lubricants can be added to PET prior to forming the PET into a sheet or bottle preform. Lubricants can be applied to finished packaging.
 - When PET flake from a bottle side wall is heated in wash water at 85° C, the PET is above the glass transition temperature and so can shrink in dimension the PET elongated wall remembers that it started out as a short and thick preform! Further, the flake can curl. There are anecdotal reports that shrinking and curling can help separate adhesives and labels from the flake surface. A sample taken from PET sheet may not show such shrinkage and curling.
 - This lab procedure recommends sourcing PET from different end uses to help evaluate any impact of the PET surface on adhesion and adhesive removal. The procedure also calls for a wash of the surface with isopropyl alcohol as a means to remove any readily soluble material on the PET surface and reduce variation.
- 3. PET bottles can be sourced from packaging distributors such as Berlin Packaging or TriCor Braun. Berlin Packaging stocks a 16 ounce boston round bottle made with a known APR PET control resin. <u>16 oz Natural Plastic Boston Round Bottle made of B90A Resin - APR Approved</u> <u>| Berlin Packaging</u>



4. KP Films can provide an amorphous PET sheet made from an APR PET control resin, the product designation is KP Mono APET FD-E610F04 A contact at KP Films that is current as of the time of publishing this method is:

Dominique Schoech

Innovation Manager Americas

Food Packaging

Klöckner Pentaplast

Klöckner Pentaplast of America, Inc.

3585 Klöckner Road

Gordonsville, VA 22942 USA

Mobile: 540.406.9597

Dominique.Schoech@kpfilms.com

www.kpfilms.com

- 5. Other example sources of PET substrate include:
 - Cutting the side wall from 2 liter softer drink bottles.
 - Cut the flat side wall from a container such as a ketchup bottle or a trigger spray bottle.