

SINGLE PURPOSE TEST PROTOCOL:

Labels for PET –

Wash Water Evaluation

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Introduction

Scope, Significance, and Use

When PET packaging is recycled, it is usually undesirable for inks and adhesives from film labels to become dispersed in the hot caustic wash water. Ink or adhesives must be removed from wash and rinse water with filtration and can lead to higher costs associated with operating a wash and waste water disposal system. On the other hand, certain innovative developments may require inks to be separated into wash water.

Reducing water utilization, energy for heating water, and waste-water disposal costs is a vital area of interest to all stake holders involved in plastics recycling and sustainability. This waste water evaluation provides a means for laboratory investigators to observe any inks or adhesives that are found in wash water, and then evaluate whether inks or adhesives are removed from wash water by settling or filtration in those cases where these components are removed in the hot caustic wash step.



This evaluation is used for measuring and reporting only. There are no APR Guidance values associated with this test at this time. Its purpose is to highlight the importance of this industry impact area and gain experience that can move the industry forward. This wash water report is a component of APR Benchmark and Critical Guidance tests involving film labels and direct printing applications. The tests outlined are not intended to replicate a complete wastewater treatment procedure. Additional steps, such as flocculation, are typically used in commercial practice, but are not included in this laboratory evaluation.

Other APR evaluations can be used to measure the impact of inks and labels on any discoloration of recycled PET. Preventing discoloration of recycled PET is the priority in developing and specifying label materials, and those other tests address that priority.

Disclaimer

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Reference Documents

• **Plastic Recycling Glossary** (APR Definitions for bleeding ink, thermoset ink, incidental ink and free adhesive): <u>https://plasticsrecycling.org/plastics-recycling-glossary/#Labels,Inks,andAdhesives</u>

Method Summary and Flow Diagram

A one-liter sample of wash water is saved after washing PET flake created from granulation of packages with film labels or direct printing. The wash water sample is evaluated for presence of ink and adhesives, filtering performance, and settling performance. Samples of printed surfaces are evaluated for the presence and appearance of graphics after the hot caustic wash step.

The following illustration references the steps involved:

PET Wash Water Evaluation Steps



Equipment required

- 1-liter beaker to hold wash water sample
- Buchner funnel that can accommodate 110 mm diameter filter media
- Side arm vacuum flask 500 ml
- Water aspirator for vacuum source
- 500 ml graduate cylinder
- Weigh scale (0.01 ± grams)

Materials required

- Whatman Grade 93 wet strength filter paper 10 micron, 110 mm diameter
- 250 mesh metal screen filter, about 60 micron openings, 110 mm diameter
- 1 liter sample of wash water after the PET flake and printed sample has been washed, referred to as Sample A.



- Retain of fresh printed label or direct print area from PET container, referred to as Sample B.
- Retain of any label film recovered at end of the hot caustic wash step, or PET flake if from a direct print label, referred to as Sample C.

Method steps

- 1. Have available:
 - a. Sample B: A sample of label material that has not been exposed to granulation and caustic wash.
 - b. Sample C: A sample of the film label material recovered from the wash water and exposed to hot caustic throughout the wash step or a sample of PET flake that was direct printed and washed.
 - c. Sample A: The 1 liter sample of wash water created from washing PET flake with labels.
- 2. Mix Sample A of wash water with a stir rod to suspend any ink or adhesive residue and decant the wash water from any PET fines that may be present so that PET fines content is negligible in the decanted liquid sample. (The wash water sample was taken after floating solids were removed from the flake wash step as well as after the wash water was passed through a dewatering screen. The expectation is that Sample A after the decant step contains only any ink or adhesive removed in the wash step.)
- 3. Visual evaluations:
 - a. Visually evaluate the printed film or PET exposed to the hot caustic; Sample B vs Sample C.
 - b. If there is adhesive used with the label, examine for visual evidence of the removal of adhesive because of the wash step.
 - c. Visually evaluate the wash water Sample A and observe for the presence of ink and free adhesive. If:
 - i. Ink on the label or printed area of PET is adhered, and there is no visible loss of ink from the printed area; and
 - ii. The text and graphics on the exposed printed area are fully legible; and
 - iii. There is no more than incidental ink in the wash water; and

iv. Adhesive is adhered to label, or there is no visible adhesive in the wash water, Then, the evaluation is complete.

When there is evidence of bleeding or thermoset ink, or free adhesive in the wash water, the following evaluations are conducted:



- 4. Settling evaluation
 - a. Place a well-mixed sample of Sample A wash water into the 500 ml graduate cylinder.
 - b. Let the cylinder stand for 30 minutes.
 - c. Evaluate the sample contained in the graduate cylinder qualitatively and assign one of these three rankings:
 - i. No clear evidence that any settling has taken place the sample is uniformly colored from top to bottom, there are no solids evident at the bottom of the graduate cylinder.
 - ii. Some settling has taken place
 - iii. Solids in the sample have substantially settled the amount of color at the top of the cylinder is substantially less than at the bottom, there is visible evidence of ink and/or adhesive at the bottom of the cylinder.
- 5. Screen filter evaluation
 - a. Set up the Buchner funnel with vacuum connection. Tare weigh the test 250 mesh screen. Place tared screen into funnel.
 - b. Pass 500 ml of well mixed wash water Sample A through the 250 mesh screen filter, making sure all liquid passes through the screen.
 - c. Make the following observations:
 - i. Did material collect on the screen? Yes or No. Further determination, only if Yes,
 - ii. Compare the appearance of the filtered water with a sample of the original wash water. Make the following determination:
 - 1. Appearance after filtering is very similar to the starting material.
 - 2. Some level of colored material or adhesive was filtered out.
 - 3. Most colored material was filtered out.
 - d. Air dry any material collected on the filter to constant weight; record the weight.
- 6. Whatman Grade 93 wet strength filter paper, 10 μ m
 - a. Set up the Buchner funnel with a vacuum connection of 10 mm mercury. Tare weigh the test filter paper. Place tared filter paper into funnel.
 - b. Pour a small amount of the recovered wash water sample onto the filter paper, allowing it to wet the entire surface
 - c. Pass the wash water sample recovered from the screen filter evaluation in step 5 through the Whatman 93 filter. Measure the time for all the wash water to pass through the filter.
 - d. Measure the time it takes for 500 ml of fresh tap water to pass through a second clean filter.



- e. Make the following observations:
 - i. Did material collect on the filter? Yes or No determination. Only if Yes,
 - ii. Compare the appearance of the filtered water after passing through the Whatman filter paper with a sample of the original wash water before filtration through the Whatman filter. Make the following determination:
 - 1. Appearance after filtering is very similar to the starting material.
 - 2. Some level of colored material was filtered out.
 - 3. Most colored material was filtered out, but water is still hazy.
 - 4. Color and haze is substantially absent in filtered water.
- f. Record the time to filter the wash water sample as well as the time to filter fresh tap water.
- g. Air dry any material collected on the filter to constant weight; record the weight.

Evaluation of results

A report form shown in Annex I can be used to record and report results.

The APR does not offer quantitative guidance for results obtained with this evaluation. The following qualitative statements can be used to evaluate and rank outcomes:

- 1. Results from this test compliment other Benchmark tests or Critical Guidance Protocols done to evaluate the impact of labels, inks and adhesives on the color and haze value of injection molded PET plaques. Labels, inks and adhesives that do not impact PET color and haze values are the primary design objectives when considering labels and printing.
- 2. As a general rule, the recycling industry prefers film labels that float in water and where the ink is retained on the label and does not enter the wash water.
- 3. In the case of adhesives, free adhesives that mix with water may be a source of contamination when they mix and entangle with PET flake.
- 4. In the case of inks that are designed to be removed in the flake wash process, inks that are easier to remove by filtration or settling are more desirable than inks that are finely dispersed, difficult to filter and best removed by dilution with more make-up water.



Annex 1: Report Form

Measurement or observation	Report values	Result
Visual Evaluation		
Describe label format or style	 Film label Direct print on PET 	
Wash impact on printed area appearance and legibility comparison with unwashed label	 No impact All ink/adhesive removed Some ink/adhesive adhering 	
Presence of ink in wash water	 No ink in wash water, incidental ink is acceptable Bleeding ink, discoloring wash water as a solution or fine dispersion Thermoset ink in wash water as discrete particles 	
Presence of free adhesive in wash water	 No adhesive in wash water Adhesive visible in wash water. 	
Settling evaluation		
Rating of a, b or c from step 4	Report rating	
250 mesh evaluation		
Any material filtered?	Report Y/N	
If Yes, Rating of a, b or c from step 5	Report	
Dry weight on filter	Report, grams	
Whatman filter evaluation		
Any material filtered?	Report Y/N	
If Yes, Rating of a, b, c, or d from step 6	Report	
Filtration time of wash water divided by that for water only	Report	
Dry weight on filter	Report, grams	



Document Version History

Version	Publication Date	Revision notes
1	11-16-2018	Original
2	09-03-2024	Updated hyperlink to match new website
3	02-13-2025	Changes per PET Tech Committee Vote on 12/20/2024 (see
		change log)