

# Plastics Forming Enterprises LLC



## Case Study Report C101 Surfactant Wash Water Chemistry Case Study





*Plastics Forming Enterprises, LLC*

*Plastics Consulting, Research, and Engineering*

## **Surfactant Wash Water Chemistry Case Study**

### **Overview**

Plastics Forming Enterprises (PFE) conducted a case study to evaluate the efficacy of various industry-standard surfactants in wash chemistry, aiming to characterize performance variability. The study utilized both clean PET bottle flake, supplied by PFE, and dirty PET flake obtained from a local transfer station in Merrimack, NH.

The assessment of clean PET flake was designed to isolate the influence of residual wash chemistry, while the evaluation of dirty PET flake aimed to determine surfactant performance under real-world conditions. The primary performance metrics, identified based on in-field challenges encountered by plastics reclaimers, included water contamination, flake discoloration, and haze values. PFE hypothesized that these challenges could be attributed to the specific wash chemistries used or deviations from the recommended surfactant solution ratios.

Each surfactant was introduced at a predetermined concentration, as specified by the respective chemistry suppliers. The evaluation was conducted in accordance with a modified version of the PET-B-02 standard, as established by the Association of Plastics Recyclers (APR).

### **Conclusions**

Utilizing the data generated from this case study, PFE systematically assessed the efficacy of various industry-standard wash chemistries on both clean and contaminated PET flakes. The findings indicate that there are favorable outcomes depending upon the material supplied conditions and the attributes such as, color impact, haze reduction, water filterability and clarity. These attributes were ranked separately for clean flake and dirty flake.

The results offer critical insights for plastics recycling reclaimers, emphasizing the significance of selecting optimal wash chemistries to enhance material quality and mitigate processing challenges. By adhering to the prescribed solution ratios and employing the most effective surfactant, recyclers can improve the overall quality of recycled PET, thereby increasing its suitability for downstream applications in end-use markets.

Given the inherent variability in feedstock composition across different recycling facilities, the impact of wash chemistry is expected to vary based not only on solution ratio but also on the specific processing technologies employed. Factors such as flake-to-solution friction, temperature, and caustic concentration further influence the overall effectiveness of the wash process. Consequently, each reclamation facility should be evaluated on an individual basis, as the findings of this study may not be universally applicable across all recycling operations. In the event this evaluation was performed for a specific recycling facility, values and rankings would be weighted based on the reclaimers goals.

### Clean Flake Ranking

Property	Test 1	Test 2	Test 3	Test 4	Test 5
Washed Flake b*	3	2	1	5	4
Bake Flake b*	3	1	4	2	5
Plaque b*	3	1	5	4	2
Plaque Haze	3	5	4	2	1
Settled Water Clarity	3	4	1	5	2
Shaken Water Clarity	5	3	1	4	2
Filter Weights	3	4	2	1	5
Water Filter Time	3	4	2	1	5
<b>Total Score</b>	<b>26</b>	<b>24</b>	<b>20</b>	<b>24</b>	<b>26</b>

*Lowest Score = Best Result*

**KEY:**

- Green = Best Result**
- Orange = 2<sup>nd</sup> Best Result**
- Yellow = 3<sup>rd</sup> Best Result**
- Blue = 4<sup>th</sup> Best Result**
- Red = Worst Result**

The ranking scale of 1 to 5 is intended to offer a high-level interpretation of the data; however, it does not reflect the distribution within individual data categories. For a more precise understanding of the significance of these rankings, refer to the data tables within the report.

Additionally, the results are not weighted, as each recycling facility may prioritize different material properties based on its specific operational requirements.

## Dirty Flake Ranking

Property	Test 1	Test 2	Test 3	Test 4	Test 5
Washed Flake b*	4	1	3	5	2
Bake Flake b*	1	2	3	4	5
Plaque b*	4	1	5	3	2
Plaque Haze	2	1	5	4	3
Settled Water Clarity	1	3	2	4	5
Shaken Water Clarity	1	4	3	2	5
Filter Weights	5	1	2	4	3
Water Filter Time	5	1	2	4	3
<b>Total Score</b>	<b>23</b>	<b>14</b>	<b>25</b>	<b>30</b>	<b>28</b>

*Lowest Score = Best Result*

### KEY:

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**Yellow = 3<sup>rd</sup> Best Result**

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Additionally, the results are not weighted, as each recycling facility may prioritize different material properties based on its specific operational requirements.

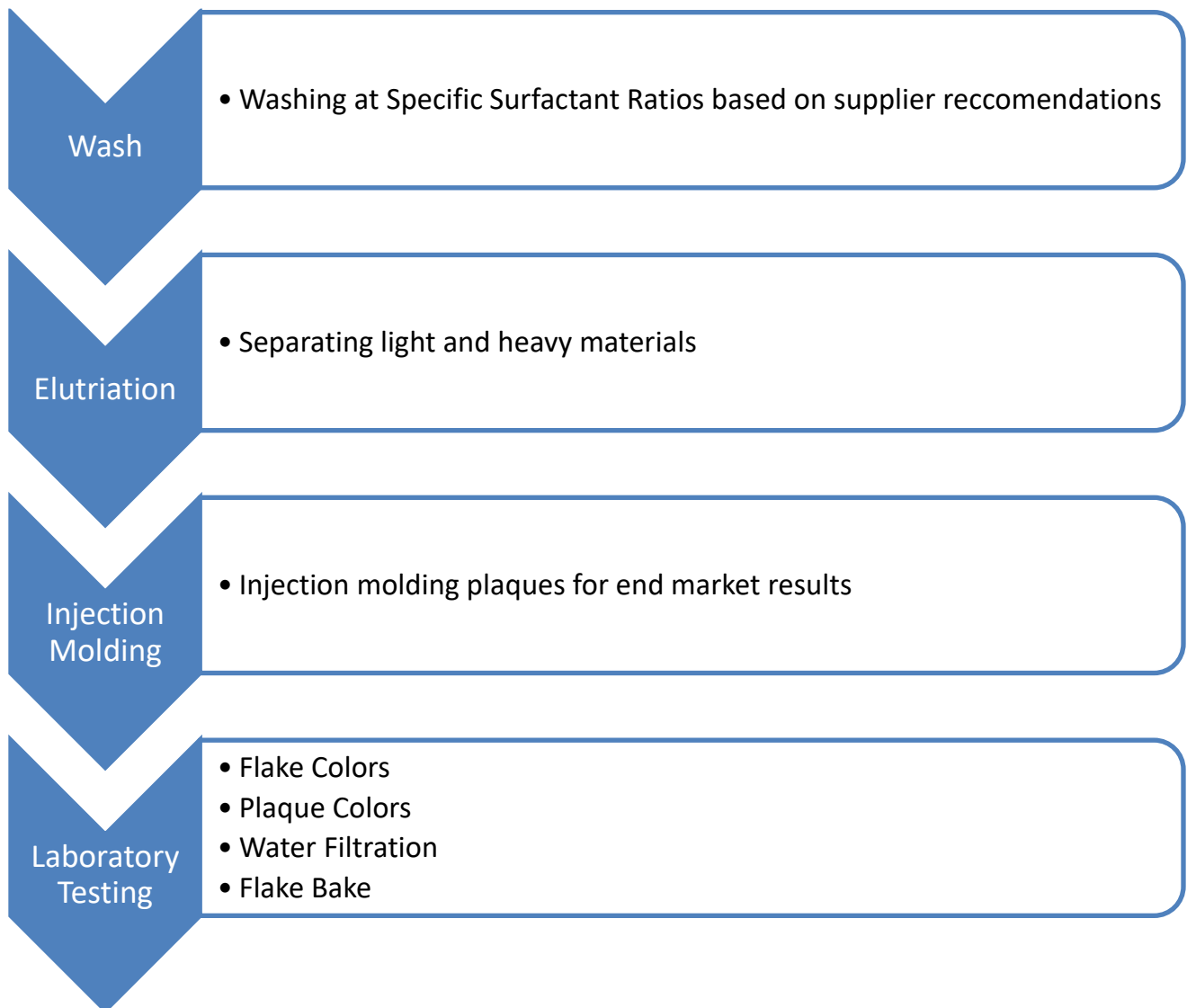
Project Partners



## Scope of Work

### Equipment

- Recycle System
  - Grinder with 3/8" Screen
  - Pilot Wash System
  - Kice Elutriator
  - Desiccant Dryer
  - Arburg Injection Machine
- Test Equipment
  - Water Filtration Analyzer
  - Color Spectrophotometer
  - Vacuum Oven
  - Convection Oven





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## Incoming Clean Flake Material Preparation

**References:**



PET-P-03

**Test Summary:**

Granulate incoming articles to the required size.

**Procedure:**

Articles are manually fed into a grinder with screen holes ranging from 9.5-12 mm in diameter.

Variable	Incoming Article	Ground Article
Clean Flake		



## Clean Flake PET Wash and Sink/Float Evaluation

### References:

PET-P-04 - Modified

### Test Summary:

#### Wash Process

PET Flake was washed at the intended surfactant to caustic ratios supplied by the manufacturer and heated to  $85 \pm 2^\circ\text{C}$ . The 4:1 ratio of water to flakes were mixed with an impellor for 15 minutes at least 546 rpm. After 15 minutes, the impeller was stopped, and the mixture was removed from the heat source and allowed to sit for five minutes. After five minutes, the wash water, floatables, and sinkables were retained.

#### Rinse

The sinking fraction of the washed PET flake is added to tap water at a 4:1 ratio of water to flake and heated to  $45 \pm 2^\circ\text{C}$ . The flake and water are mixed with an impeller for at least 546 rpm for five minutes. After five minutes, the mixture is removed from the heat and allowed to sit for five minutes. After five minutes, the rinse water, floatables, and sinkables are retained.







#### Sink/Float

The rinsed PET flake is added to tap water at 8:1 water to flake. The tank is agitated by hand or at 500 rpm for five minutes and then allowed to sit for five minutes. After five minutes, the floatables and sinkables are retained.





### Data Table:

Variable	Wash Temperature (°C)	Rinse Temperature (°C)	Starting Flake Weight (kg)	Total Floatables (grams)	Total Floatables (%)
Test 1	84	46	1.81	0.00	0.00%
Test 2	85	47	1.81	0.00	0.00%
Test 3	84	45	1.81	0.00	0.00%
Test 4	84	47	1.81	0.00	0.00%
Test 5	85	46	1.81	0.00	0.00%

## Clean Flake Wash/Rinse Pictures

Variable	Settled Waters		Shaken Waters	
	Wash	Rinse	Wash	Rinse
<b>Test 1</b>				
<b>Test 2</b>				
<b>Test 3</b>				

## Clean Flake Wash/Rinse Pictures Continued

Variable	Settled Waters		Shaken Waters	
	Wash	Rinse	Wash	Rinse
<b>Test 4</b>				
<b>Test 5</b>				

### Clean Flake Settled Wash Water Colors

Variable	L* Average	a* Average	b* Average	Haze Average	$\Delta E$ From Reference Control*
Test 1	95.69	-0.09	0.93	22.03	0.90
Test 2	95.63	-0.06	0.91	6.25	0.93
Test 3	96.64	-0.17	0.15	1.14	0.38
Test 4	95.06	-0.04	1.40	10.73	1.67
Test 5	96.51	-0.16	0.22	2.66	0.24

### Clean Flake Shaken Wash Water Colors

Variable	L* Average	a* Average	b* Average	Haze Average	$\Delta E$ From Reference Control*
Test 1	94.26	0.02	1.15	87.20	0.67
Test 2	92.62	0.09	2.21	24.75	2.06
Test 3	92.90	0.02	1.14	26.89	1.13
Test 4	92.86	0.07	2.15	24.54	1.86
Test 5	91.25	0.12	1.58	41.09	2.82

\*The reference control is a standard PFE PET control wash water sample, which is either allowed to settle or shaken for comparison.

## Clean Flake PET Wash Water Evaluation

### **References:**

PET-S-01

### **Test Summary:**

Visual evaluations of the wash water are conducted to determine the characteristics of the label substrate, inks, and adhesives utilized within the label. The water is examined for suspended ink or adhesive. If ink or adhesive is seen, the water is evaluated.

### **Procedure:**

500mL of water is transferred into a graduated cylinder, allowed to settle for 30 minutes, then ranked depending on the amount of settling of the inks and adhesive. After the setting evaluation, the wash water is passed through a 250-mesh screen using a Buchner funnel. The filtered water is ranked on its appearance. The wash water is then filtered again with a 10µm filter paper using a Buchner funnel. The time it takes to filter the wash water is measured and compared to the time it takes for clean tap water to filter. The filtered water is ranked on its appearance.

## Clean Flake PET Wash Water Filtration Evaluation

Variable	Test 1	Test 2	Test 3	Test 4	Test 5
<b>250-Mesh Evaluation</b>					
250-Mesh Starting Weight (g)	2.9754	2.9679	2.9781	2.9987	3.0238
250-Mesh Ending Weight (g)	2.9948	3.0141	3.0078	3.0448	3.1969
250-Mesh Collected Weight (g)	0.0194	0.0462	0.0297	0.0461	0.1731
250-Mesh Collection (Y/N)	Y	Y	Y	Y	Y
250-Mesh Appearance Ranking	A	A	A	A	A
<b>10µm Evaluation</b>					
10µm Filter Starting Weight (g)	0.8173	0.8020	0.8032	0.8099	0.8055
10µm Filter Ending Weight (g)	0.8493	0.8817	0.8531	0.8751	0.8645
10µm Filter Collected Weight (g)	0.0320	0.0797	0.0499	0.0652	0.0590
10µm Filter Collection (Y/N)	Y	Y	Y	Y	Y
10µm Filter Appearance Ranking	A	B	B	B	B
10µm Filter Time (s)	3:19	2:50	3:21	6:12	1:30
Tap Water Filter Time (s)	00:27				
10µm Filter Time / Tap Water Filter Time	7.37	6.30	7.44	13.78	3.33

### Ranking System for Filtering Evaluation:

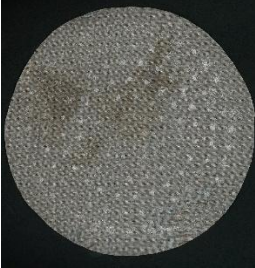
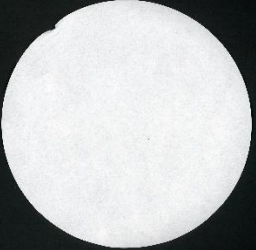




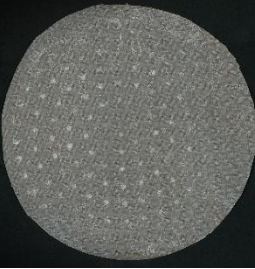
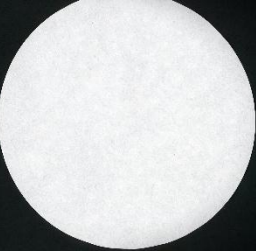




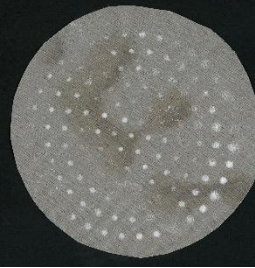
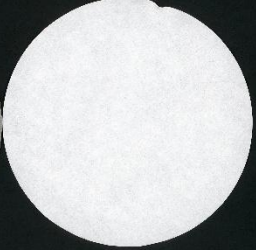




#### 250-Mesh Evaluation

- a. Appearance after filtering is similar to the starting material.
- b. Some level of colored material or adhesive was filtered out.
- c. Most colored material was filtered out.

#### 10µm Filter Evaluation



- a. Appearance after filtering is similar to the starting material.
- b. Some level of colored material was filtered out.
- c. Most colored material was filtered out, but water is still hazy.
- d. Color and haze are substantially absent in filtered water.

## Clean Flake Filter Pictures

Variable	250-Mesh Screen	10µm Filter Paper	Settled Water		Shaken Water	
			Wash	Filtered	Wash	Filtered
Test 1						
Test 2						
Test 3						



## Clean Flake Filter Pictures Continued

Variable	250-Mesh Screen	10µm Filter Paper	Settled Water		Shaken Water	
			Wash	Filtered	Wash	Filtered
Test 4						
Test 5						

### Clean Flake Filtered and Settled Wash Water Colors

Variable	L* Average	a* Average	b* Average	Haze Average	$\Delta E$ From Reference Control*
Test 1	94.37	-0.03	0.59	10.44	0.75
Test 2	95.69	-0.12	1.09	6.56	0.95
Test 3	96.60	-0.19	0.24	1.78	1.53
Test 4	95.49	-0.09	1.44	9.56	1.16
Test 5	96.60	-0.19	0.24	1.88	1.53

### Clean Flake Filtered and Shaken Wash Water Colors

Variable	L* Average	a* Average	b* Average	Haze Average	$\Delta E$ From Reference Control*
Test 1	94.32	0.00	0.87	85.01	0.28
Test 2	94.89	-0.05	1.74	11.61	1.28
Test 3	95.83	-0.14	0.54	7.17	1.51
Test 4	95.13	-0.07	1.54	11.83	1.24
Test 5	95.65	-0.12	0.64	10.59	1.32

\*The reference control is a standard PFE PET control wash water sample after filtration, which is either allowed to settle or shaken for comparison.

## Clean Flake PET Elutriation

### References:

PET-P-05 - Modified

### Test Summary:

Remove lights (fines, labels, layers) from washed flake before extrusion.







### Procedure:

The elutriator is calibrated based on historical PET control flake data. Once the elutriator is calibrated, heavies' weight, lights' weight, and the percentage of lights are recorded for each test. Heavies are then prepared for the next steps of processing.





### Data Table:

Variable	Heavies Weight (kg)	Lights Weight (grams)	Lights Removed (%)
Test 1	1.55	1.02	0.07%
Test 2	1.67	0.57	0.03%
Test 3	1.66	0.58	0.03%
Test 4	1.68	0.66	0.04%
Test 5	1.65	0.42	0.03%

## Clean Flake Heavies/Lights Pictures

Variable	Heavies	Lights
Test 1	 A petri dish containing a dense layer of large, irregular, translucent white flakes.	 A petri dish containing a sparse layer of small, irregular, translucent white flakes.
Test 2	 A petri dish containing a dense layer of large, irregular, translucent white flakes.	 A petri dish containing a sparse layer of small, irregular, translucent white flakes.
Test 3	 A petri dish containing a dense layer of large, irregular, translucent white flakes.	 A petri dish containing a sparse layer of small, irregular, translucent white flakes.

## Clean Flake Heavies/Lights Pictures Continued

Variable	Heavies	Lights
Test 4	 A petri dish filled with a dense layer of large, irregular, translucent white flakes.	 A petri dish containing a sparse amount of small, irregular, translucent white flakes.
Test 5	 A petri dish filled with a dense layer of large, irregular, translucent white flakes.	 A petri dish containing a sparse amount of small, irregular, translucent white flakes.



## Clean Flake 1<sup>st</sup> Heat Processed Flake Colors

**References:**

ASTM D6290

*Within PFE's scope of ISO 17025 accreditation, certificate number AT-3210.*

**Test Summary:**

Measure the degree of yellowness under daylight illumination of transparent, translucent, or opaque plastics.

**Equipment:**

Konica Minolta 36dG

**Procedure:**

The color analyzing equipment is set up on reflectance and calibrated using pure white and black standards. The sample cell is filled to the brim with randomly selected flakes. The cell is inserted into the machine, and the equipment runs the color test. The test indicates L\*, a\*, and b\* values. This test is completed in a conditioned laboratory at a temperature of 23±2°C and a relative humidity of 50±10%.

**Data Table:**

Variable	L* Values	a* Values	b* Values	L* Average	a* Average	b* Average
Test 1	69.04	-0.12	-0.18	66.97	-0.12	-0.10
	64.09	-0.16	-0.22			
	68.92	-0.12	-0.14			
	67.60	-0.11	0.10			
	65.21	-0.08	-0.04			
Test 2	64.23	-0.15	-0.19	66.79	-0.16	-0.20
	68.65	-0.20	0.16			
	65.97	-0.14	-0.46			
	69.13	-0.16	-0.06			
	65.99	-0.15	-0.43			
Test 3	68.60	-0.10	-0.50	67.2	-0.11	-0.31
	64.55	-0.15	-0.36			
	68.70	-0.11	-0.49			
	68.04	-0.07	0.14			
	66.11	-0.12	-0.32			
Test 4	65.54	-0.13	0.17	66.69	-0.13	0.29
	68.73	-0.14	0.38			
	65.10	-0.11	0.26			
	68.69	-0.14	0.44			
	65.41	-0.14	0.20			
Test 5	68.49	-0.10	0.27	67.24	-0.11	0.01
	66.76	-0.12	-0.22			
	68.40	-0.09	0.25			
	68.70	-0.13	0.19			
	63.86	-0.11	-0.42			

## Clean Flake Bake Evaluation

**References:**







PET-S-10

**Test Summary:**

Determine the color changes of the flake after baking.


**Procedure:**

100 grams of flake is placed into a convection oven heated at 220°C for 60 minutes.

Variable	Before Baking Flake	After Baking Flake
Test 1	 A petri dish containing numerous small, irregular, translucent white flakes of petroleum jelly against a black background.	 A petri dish containing numerous small, irregular, translucent white flakes of petroleum jelly against a white background.
Test 2	 A petri dish containing numerous small, irregular, translucent white flakes of petroleum jelly against a black background.	 A petri dish containing numerous small, irregular, translucent white flakes of petroleum jelly against a white background.
Test 3	 A petri dish containing numerous small, irregular, translucent white flakes of petroleum jelly against a black background.	 A petri dish containing numerous small, irregular, translucent white flakes of petroleum jelly against a white background.



**Clean Flake Bake Evaluation Continued**

<b>Variable</b>	<b>Before Baking Flake</b>	<b>After Baking Flake</b>
<b>Test 4</b>		
<b>Test 5</b>		

## Clean Flake Bake Evaluation Colors

**References:**

ASTM D6290

*Within PFE's scope of ISO 17025 accreditation, certificate number AT-3210.*

**Test Summary:**

Measure the degree of yellowness under daylight illumination of transparent, translucent, or opaque plastics.

**Equipment:**

Konica Minolta 36dG

**Procedure:**

The color analyzing equipment is set up on reflectance and calibrated using pure white and black standards. The sample cell is filled to the brim with randomly selected flakes. The cell is inserted into the machine and the equipment runs the color test. The test indicates L\*, a\*, b\* values. This test is completed in a conditioned laboratory at a temperature of 23±2°C and a relative humidity of 50±10%.

**Data Table:**

Variable	L* Values	a* Values	b* Values	L* Average	a* Average	b* Average
Test 1	74.28	-0.36	1.97	73.09	-0.38	1.36
	71.59	-0.43	1.00			
	72.83	-0.41	0.93			
	73.34	-0.30	1.65			
	73.41	-0.41	1.23			
Test 2	75.23	-0.62	0.66	75.53	-0.65	0.91
	72.33	-0.60	1.17			
	75.41	-0.61	0.56			
	73.73	-0.79	1.35			
	70.93	-0.65	0.79			
Test 3	74.32	-0.69	2.66	73.40	-0.70	1.53
	74.41	-0.83	0.90			
	73.64	-0.67	1.58			
	71.26	-0.60	1.07			
	73.38	-0.70	1.42			
Test 4	74.45	-0.49	1.11	72.70	-0.45	1.19
	72.10	-0.42	1.19			
	73.94	-0.47	1.02			
	71.73	-0.42	1.19			
	71.28	-0.44	1.45			
Test 5	73.66	-0.88	2.72	73.31	-0.87	2.11
	72.18	-0.90	1.13			
	73.73	-0.90	2.82			
	75.00	-0.85	2.36			
	71.99	-0.84	1.50			

## Clean Flake 2<sup>nd</sup> Heat PET Injection

### References:

PET-P-08

### Test Summary:

Injection mold 3mm plaques to test for color and haze values.

### Procedure:

Injection material is prepped in a dryer until the moisture content is below 50 ppm. PET is injection molded at a target melt temperature of 275°C. The injection unit is purged between each test innovation. Melt, room, and mold temperatures are recorded for each variable.

### Data Table:

Variable	Moisture Content (ppm)	Melt Temperature (°C)	Mold Temperature (°C)
Test 1	15	276	19
Test 2	12	276	19
Test 3	27	276	19
Test 4	5	277	20
Test 5	3	277	19

### Operating Conditions:

Zone 1 Temperature (°C)	Zone 2 Temperature (°C)	Zone 3 Temperature (°C)	Nozzle Temperature (°C)
265	266	266	275

## Clean Flake 2<sup>nd</sup> Heat Injection Molded Plaques Pictures

Test 1



Test 2



Test 3



Test 4



Test 5



## Clean Flake 2<sup>nd</sup> Heat Transmission Plaque Colors

**References:**

PET-P-08  
 PET-S-09  
 ASTM D1003

*Within PFE's scope of ISO 17025 accreditation; certificate number AT-3210.*

**Test Summary:**

Measure the color values L\*, a\*, b\*, and haze of a 3mm injection-molded plaque.

**Equipment:**

Konica Minolta 36dG

**Procedure:**

Pellets are dried to a moisture level below 50 ppm and injection molded into 3mm plaques. The color analyzing equipment is set up on transmittance and calibrated using pure white and black standards. The molded plaque is inserted into the testing location, and the equipment runs the color test.

**Data Table:**

Variable	L* Values	a* Values	b* Values	L* Average	a* Average	b* Average	Haze	Haze Average
Test 1	92.64	-0.50	4.40	92.77	-0.42	3.92	3.73	3.23
	92.85	-0.38	3.57				2.66	
	92.83	-0.37	3.58				3.14	
	92.84	-0.36	3.54				3.11	
	92.67	-0.51	4.52				3.51	
Test 2	92.57	-0.37	3.38	92.18	-0.51	3.54	3.58	3.70
	90.78	-0.86	3.23				4.59	
	92.80	-0.39	3.69				3.21	
	91.98	-0.52	3.58				3.66	
	92.77	-0.41	3.81				3.44	
Test 3	92.70	-0.48	4.25	92.69	-0.44	4.07	3.40	3.60
	92.56	-0.44	4.14				4.70	
	92.73	-0.46	4.12				3.49	
	92.74	-0.40	3.90				3.11	
	92.74	-0.42	3.93				3.29	
Test 4	92.92	-0.47	3.95	92.91	-0.46	3.95	2.85	3.07
	92.89	-0.45	3.92				3.36	
	92.89	-0.48	4.06				2.71	
	92.88	-0.44	3.92				3.22	
	92.95	-0.48	3.91				3.21	
Test 5	92.84	-0.45	3.94	92.85	-0.41	3.78	2.96	3.05
	92.84	-0.40	3.79				3.13	
	92.88	-0.39	3.70				2.95	
	92.83	-0.40	3.77				3.02	
	92.85	-0.39	3.71				3.18	



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## **Dirty Flake Contents**

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## Incoming Dirty Flake Material Preparation

**References:**

PET-P-03

**Test Summary:**

Granulate incoming articles to the required size.

**Procedure:**

Articles are manually fed into a grinder with screen holes ranging from 9.5-12 mm in diameter.

Variable	Incoming Article	Ground Article
Dirty Flake		



## Dirty Flake PET Wash and Sink/Float Evaluation

### References:

PET-P-04 - Modified

### Test Summary:

#### Wash Process

PET Flake was washed at the intended surfactant to caustic ratios as supplied by the manufacturer and heated to  $85 \pm 2^\circ\text{C}$ . The 4:1 ratio of water to flakes were mixed with an impeller for 15 minutes at least 546 rpm. After 15 minutes, the impeller was stopped, and the mixture was removed from the heat source and allowed to sit for five minutes. After five minutes, the wash water, floatables, and sinkables were retained.

#### Rinse

The sinking fraction of the washed PET flake is added to tap water at a 4:1 ratio of water to flake and heated to  $45 \pm 2^\circ\text{C}$ . The flake and water are mixed with an impeller for at least 546 rpm for five minutes. After five minutes, the mixture is removed from the heat and allowed to sit for five minutes. After five minutes, the rinse water, floatables, and sinkables are retained.

#### Sink/Float

The rinsed PET flake is added to tap water at 8:1 water to flake. The tank is agitated by hand or at 500 rpm for five minutes and then allowed to sit for five minutes. After five minutes, the floatables and sinkables are retained.

### Data Table:

Variable	Wash Temperature (°C)	Rinse Temperature (°C)	Starting Flake Weight (kg)	Total Floatables (grams)	Total Floatables (%)
Test 1	83	45	1.81	61	3.30%
Test 2	84	45	1.81	55	3.03%
Test 3	85	46	1.81	62	3.35%
Test 4	86	43	1.81	53	2.89%
Test 5	85	47	1.81	63	3.40%

## Dirty Flake Wash/Rinse Pictures

Variable	Settled Waters		Shaken Waters	
	Wash	Rinse	Wash	Rinse
Test 1				
Test 2				
Test 3				

## Dirty Flake Wash/Rinse Pictures Continued

Variable	Settled Waters		Shaken Waters	
	Wash	Rinse	Wash	Rinse
Test 4				
Test 5				

### Dirty Flake Settled Wash Water Colors

Variable	L* Average	a* Average	b* Average	Haze Average	$\Delta E$ From Reference Control*
Test 1	92.20	2.72	9.51	3.25	10.52
Test 2	78.52	2.65	11.57	48.27	21.24
Test 3	76.08	2.27	10.7	54.72	22.86
Test 4	83.20	3.68	11.74	28.52	17.82
Test 5	80.00	3.63	12.61	38.02	20.78

### Dirty Flake Shaken Wash Water Colors

Variable	L* Average	a* Average	b* Average	Haze Average	$\Delta E$ From Reference Control*
Test 1	75.87	0.64	11.13	61.81	20.88
Test 2	58.17	1.02	12.95	83.62	37.81
Test 3	60.75	1.46	12.19	79.37	35.14
Test 4	63.96	1.77	13.00	72.67	32.45
Test 5	47.29	1.73	14.06	87.20	48.54

\*The reference control is a standard PFE PET control wash water sample, which is either allowed to settle or shaken for comparison.

## **Dirty Flake PET Wash Water Evaluation**

### **References:**

PET-S-01

### **Test Summary:**

Visual evaluations of the wash water are conducted to determine the characteristics of the label substrate, inks, and adhesives utilized within the label. The water is examined for suspended ink or adhesive. If ink or adhesive is seen, the water is evaluated.

### **Procedure:**

500mL of water is transferred into a graduated cylinder, allowed to settle for 30 minutes, then ranked depending on the amount of settling of the inks and adhesive. After the setting evaluation, the wash water is passed through a 250-mesh screen using a Buchner funnel. The filtered water is ranked on its appearance. The wash water is then filtered again with a 10µm filter paper using a Buchner funnel. The time it takes to filter the wash water is measured and compared to the time it takes for clean tap water to filter. The filtered water is ranked on its appearance.

## Dirty Flake PET Wash Water Filtration Evaluation

Variable	Test 1	Test 2	Test 3	Test 4	Test 5
<b>250-Mesh Evaluation</b>					
250-Mesh Starting Weight (g)	2.9316	2.9660	2.9961	2.9945	3.0353
250-Mesh Ending Weight (g)	3.0643	3.0548	3.1108	3.1039	3.2847
250-Mesh Collected Weight (g)	0.1327	0.0888	0.1147	0.1094	0.2494
250-Mesh Collection (Y/N)	Y	Y	Y	Y	Y
250-Mesh Appearance Ranking	B	A	A	B	B
<b>10µm Evaluation</b>					
10µm Filter Starting Weight (g)	0.7828	1.5845	0.7847	0.7792	0.7880
10µm Filter Ending Weight (g)	0.9012	1.8936	0.9398	0.9173	0.9361
10µm Filter Collected Weight (g)	0.1184	0.3091	0.1551	0.1381	0.1481
10µm Filter Collection (Y/N)	Y	Y	Y	Y	Y
10µm Filter Appearance Ranking	C	C	C	C	C
10µm Filter Time (s)	04:01	21:11	06:45	04:40	05:19
Tap Water Filter Time (s)	00:27				
10µm Filter Time / Tap Water Filter Time	8.93	47.07	15.00	10.37	11.81

### Ranking System for Filtering Evaluation:

#### 250-Mesh Evaluation

- a. Appearance after filtering is similar to the starting material.
- b. Some level of colored material or adhesive was filtered out.
- c. Most colored material was filtered out.

#### 10µm Filter Evaluation

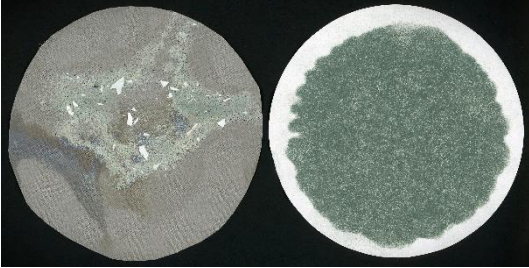


- a. Appearance after filtering is similar to the starting material.
- b. Some level of colored material was filtered out.
- c. Most colored material was filtered out, but water is still hazy.
- d. Color and haze are substantially absent in filtered water.

## Dirty Flake Filter Pictures

Variable	250-Mesh Screen	10µm Filter Paper	Settled Water		Shaken Water	
			Wash	Filtered	Wash	Filtered
Test 1						
Test 2						
Test 3						



## Dirty Flake Filter Pictures Continued

Variable	250-Mesh Screen	10µm Filter Paper	Settled Water		Shaken Water	
			Wash	Filtered	Wash	Filtered
Test 4						
Test 5						

### Dirty Flake Filtered and Settled Wash Water Colors

Variable	L* Average	a* Average	b* Average	Haze Average	$\Delta E$ From Reference Control*
Test 1	91.51	2.32	7.28	11.09	8.16
Test 2	89.30	3.09	7.96	16.23	10.08
Test 3	91.34	4.00	7.72	5.58	9.23
Test 4	89.42	4.39	10.08	12.56	12.12
Test 5	88.11	4.29	10.89	15.85	13.38

### Dirty Flake Filtered and Shaken Wash Water Colors

Variable	L* Average	a* Average	b* Average	Haze Average	$\Delta E$ From Reference Control*
Test 1	91.04	2.14	8.55	8.71	8.88
Test 2	86.26	2.79	7.81	28.19	11.19
Test 3	90.04	3.51	7.41	9.69	8.80
Test 4	87.79	3.87	9.62	16.18	11.81
Test 5	82.85	3.78	11.45	34.32	16.25

\*The reference control is a standard PFE PET control wash water sample after filtration, which is either allowed to settle or shaken for comparison.

## Dirty Flake PET Elutriation

### References:

PET-P-05

### Test Summary:

Remove lights (fines, labels, layers) from washed flake before extrusion.




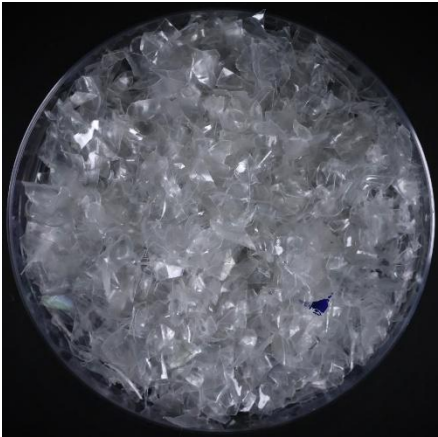


### Procedure:

The elutriator is calibrated based on historical PET control flake data. Once the elutriator is calibrated, heavies' weight, lights' weight, and the percentage of lights are recorded for each test. Heavies are then prepared for the next steps of processing.





### Data Table:

Variable	Heavies Weight (kg)	Lights Weight (grams)	Lights Removed (%)
Test 1	1.48	34.12	2.25%
Test 2	1.48	30.80	2.04%
Test 3	1.47	33.85	2.25%
Test 4	1.45	32.01	2.17%
Test 5	1.46	36.87	2.46%

## Dirty Flake Heavies/Lights Pictures

Variable	Heavies	Lights
<b>Test 1</b>		
<b>Test 2</b>		
<b>Test 3</b>		

## Dirty Flake Heavies/Lights Pictures Continued

Variable	Heavies	Lights
Test 4	 A petri dish containing a large quantity of heavy, dirty, translucent flakes. The flakes are irregular in shape and appear to be coated with a dark, thin layer, giving them a mottled appearance. They are densely packed in the dish.	 A petri dish containing a large quantity of light, dirty, translucent flakes. The flakes are irregular in shape and appear to be coated with a dark, thin layer, giving them a mottled appearance. They are densely packed in the dish.
Test 5	 A petri dish containing a large quantity of heavy, dirty, translucent flakes. The flakes are irregular in shape and appear to be coated with a dark, thin layer, giving them a mottled appearance. They are densely packed in the dish.	 A petri dish containing a large quantity of light, dirty, translucent flakes. The flakes are irregular in shape and appear to be coated with a dark, thin layer, giving them a mottled appearance. They are densely packed in the dish. A small red marker is visible on the surface of the flakes.



## Dirty Flake 1<sup>st</sup> Heat Processed Flake Colors

**References:**

ASTM D6290

*Within PFE's scope of ISO 17025 accreditation, certificate number AT-3210.*

**Test Summary:**

Measure the degree of yellowness under daylight illumination of transparent, translucent, or opaque plastics.

**Equipment:**

Konica Minolta 36dG

**Procedure:**

The color analyzing equipment is set up on reflectance and calibrated using pure white and black standards. The sample cell is filled to the brim with randomly selected flakes. The cell is inserted into the machine, and the equipment runs the color test. The test indicates L\*, a\*, and b\* values. This test is completed in a conditioned laboratory at a temperature of 23±2°C and a relative humidity of 50±10%.

**Data Table:**

Variable	L* Values	a* Values	b* Values	L* Average	a* Average	b* Average
Test 1	66.46	-0.70	0.57	65.01	-0.78	0.79
	64.91	-0.73	1.02			
	63.57	-0.79	1.06			
	66.40	-0.84	0.30			
	63.70	-0.85	1.02			
Test 2	68.15	-0.38	0.02	67.83	-0.34	-0.08
	67.01	-0.26	0.15			
	67.22	-0.41	-0.22			
	67.91	-0.28	-0.01			
	68.85	-0.37	-0.36			
Test 3	65.50	-0.65	0.51	66.16	-0.58	0.54
	64.44	-0.52	0.54			
	66.79	-0.37	0.55			
	66.18	-0.76	0.48			
	67.91	-0.58	0.62			
Test 4	67.25	-1.16	0.62	65.34	-1.19	0.98
	62.19	-1.16	1.22			
	66.92	-1.15	1.21			
	65.31	-1.34	1.13			
	65.04	-1.14	0.70			
Test 5	66.92	-0.41	0.54	67.45	-0.37	0.51
	67.53	-0.34	0.47			
	66.06	-0.33	0.67			
	69.66	-0.26	0.40			
	67.08	-0.49	0.45			

## Dirty Flake Bake Evaluation

**References:**







PET-S-10

**Test Summary:**

Determine the color changes of the flake after baking.





**Procedure:**

One hundred grams of flake is placed into a convection oven heated at 220°C for 60 minutes.

Variable	Before Baking Flake	After Baking Flake
Test 1	 A petri dish containing clear, translucent, irregularly shaped petroleum jelly flakes against a black background.	 A petri dish containing the same petroleum jelly flakes after baking, appearing significantly more yellowed and opaque.
Test 2	 A petri dish containing clear, translucent, irregularly shaped petroleum jelly flakes against a black background.	 A petri dish containing the same petroleum jelly flakes after baking, appearing significantly more yellowed and opaque.
Test 3	 A petri dish containing clear, translucent, irregularly shaped petroleum jelly flakes against a black background.	 A petri dish containing the same petroleum jelly flakes after baking, appearing significantly more yellowed and opaque.



## Dirty Flake Evaluation Continued

Variable	Before Baking Flake	After Baking Flake
Test 4	 A petri dish containing numerous small, clear, irregularly shaped flakes. The flakes are translucent and appear to be made of a solid material, possibly a polymer or wax, before being heated.	 A petri dish containing the same number of flakes as in the 'Before Baking' image. However, the flakes are now a pale, off-white color, indicating they have been heated and possibly oxidized or changed in composition.
Test 5	 A petri dish containing numerous small, clear, irregularly shaped flakes, identical in appearance to the 'Before Baking' image for Test 4.	 A petri dish containing the same number of flakes as in the 'Before Baking' image. The flakes are now a pale, off-white color, identical in appearance to the 'After Baking' image for Test 4.

## Dirty Flake Bake Evaluation Colors

**References:**

ASTM D6290

*Within PFE's scope of ISO 17025 accreditation, certificate number AT-3210.*

**Test Summary:**

Measure the degree of yellowness under daylight illumination of transparent, translucent, or opaque plastics.

**Equipment:**

Konica Minolta 36dG

**Procedure:**

The color analyzing equipment is set up on reflectance and calibrated using pure white and black standards. The sample cell is filled to the brim with randomly selected flakes. The cell is inserted into the machine, and the equipment runs the color test. The test indicates L\*, a\*, and b\* values. This test is completed in a conditioned laboratory at a temperature of 23±2°C and a relative humidity of 50±10%.

**Data Table:**

Variable	L* Values	a* Values	b* Values	L* Average	a* Average	b* Average
Test 1	68.56	-0.58	4.31	70.29	-0.62	3.05
	69.18	-0.57	2.88			
	69.62	-0.64	1.88			
	72.17	-0.71	2.81			
	71.93	-0.59	3.37			
Test 2	70.02	-0.28	3.24	71.89	-0.36	3.58
	72.57	-0.36	4.26			
	70.72	-0.43	2.70			
	73.4	-0.32	3.65			
	72.74	-0.39	4.03			
Test 3	68.54	-0.33	4.18	69.90	-0.28	4.81
	72.15	-0.18	5.55			
	68.01	-0.33	4.63			
	71.93	-0.30	4.63			
	68.89	-0.25	5.06			
Test 4	71.04	-0.36	5.49	69.80	-0.49	4.94
	69.97	-0.58	4.23			
	67.03	-0.54	4.59			
	70.49	-0.59	5.27			
	70.49	-0.39	5.14			
Test 5	71.71	-0.16	5.97	70.88	-0.21	4.98
	69.21	-0.19	5.41			
	69.4	-0.34	4.44			
	71.65	-0.11	4.93			
	72.43	-0.24	4.17			

## Dirty Flake 2<sup>nd</sup> Heat PET Injection

### References:

PET-P-08

### Test Summary:

Injection mold 3mm plaques to test for color and haze values.

### Procedure:

Injection material is prepped in a dryer until the moisture content is below 50 ppm. PET is injection molded at a target melt temperature of 275°C. The injection unit is purged between each test innovation. Melt, room, and mold temperatures are recorded for each variable.

### Data Table:

Variable	Moisture Content (ppm)	Melt Temperature (°C)	Mold Temperature (°C)
Test 1	8	274	18
Test 2	14	275	18
Test 3	9	275	18
Test 4	11	274	18
Test 5	8	274	18

### Operating Conditions:

Zone 1 Temperature (°C)	Zone 2 Temperature (°C)	Zone 3 Temperature (°C)	Nozzle Temperature (°C)
265	266	266	275

## Dirty Flake 2<sup>nd</sup> Heat Injection Molded Plaques Pictures

Test 1



Test 2



Test 3



Test 4



Test 5



## Dirty Flake 2<sup>nd</sup> Heat Transmission Plaque Colors

**References:**

PET-P-08

PET-S-09

ASTM D1003

*Within PFE's scope of ISO 17025 accreditation; certificate number AT-3210.*

**Test Summary:**

Measure the color values L\*, a\*, b\*, and haze of a 3mm injection-molded plaque.

**Equipment:**

Konica Minolta 36dG

**Procedure:**

Pellets are dried to a moisture level below 50 ppm and injection molded into 3mm plaques. The color analyzing equipment is set up on transmittance and calibrated using pure white and black standards. The molded plaque is inserted into the testing location, and the equipment runs the color test.

**Data Table:**

Variable	L* Values	a* Values	b* Values	L* Average	a* Average	b* Average	Haze	Haze Average
Test 1	85.66	-1.51	6.97	85.49	-1.51	7.09	11.72	12.37
	85.41	-1.54	7.32				13.33	
	85.44	-1.58	7.24				12.19	
	85.73	-1.49	6.88				11.28	
	85.20	-1.44	7.05				13.32	
Test 2	87.03	-0.81	4.07	86.96	-1.18	6.24	10.39	9.77
	87.04	-1.23	6.54				9.11	
	86.72	-1.29	7.05				9.98	
	87.15	-1.23	6.38				9.22	
	86.84	-1.32	7.14				10.15	
Test 3	84.55	-1.71	7.27	84.56	-1.64	7.22	14.30	14.90
	84.48	-1.62	6.85				15.05	
	84.81	-1.52	7.51				13.50	
	84.36	-1.63	7.20				17.28	
	84.59	-1.72	7.27				14.38	
Test 4	84.04	-1.99	7.00	84.03	-2.05	7.05	15.11	14.80
	84.13	-2.07	7.22				14.15	
	84.27	-1.98	6.66				14.00	
	83.94	-2.16	7.24				15.56	
	83.77	-2.07	7.13				15.17	
Test 5	85.43	-1.61	7.00	85.38	-1.43	6.64	12.90	13.10
	85.07	-1.35	6.86				14.08	
	86.05	-1.13	4.69				11.49	
	85.50	-1.57	6.97				11.86	
	84.83	-1.48	7.68				15.16	

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