

Plastics Forming Enterprises LLC



Laboratory Report #2084
APR PET CG-01 Critical Guidance (Path 1)

Applicant:

Avantium

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Plastics Forming Enterprises, LLC

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Objectives:

- To determine the impact of an innovation material on the color properties and IV change over 3 heat histories including solid state polymerization and IV build.

Materials:

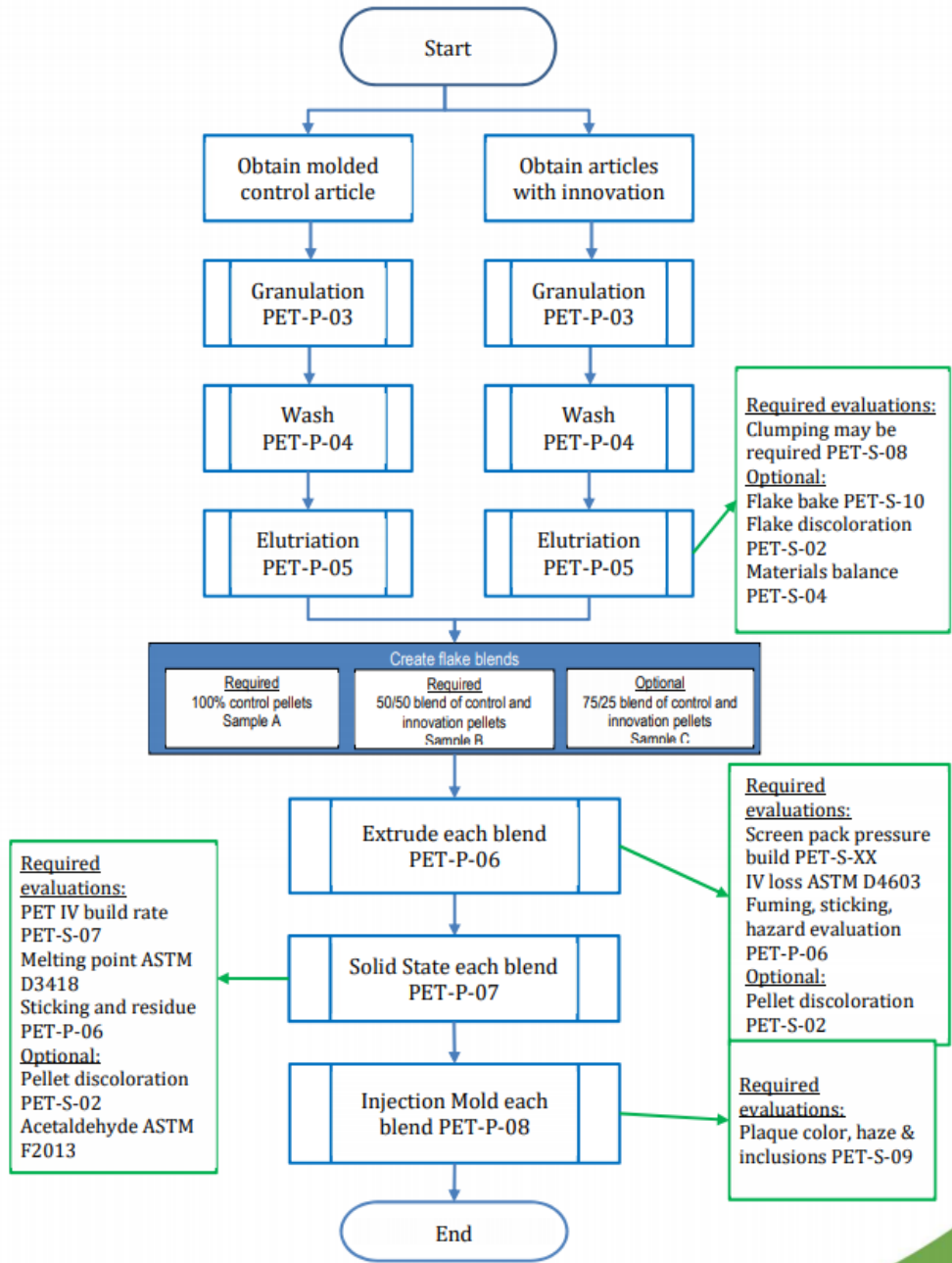
- Control – Reference bottle 100% PET DAK E60A
- Test 1 – Test sample 1 (95% PET + 5% PEF)
- Test 2 – Test sample 2 (90% PET + 10% PEF toned)
- Test 3 – Test sample 3 (93% PET + 7% PEF toned)
- Test 4 – Test sample 4 (95% PET + 5% PEF toned)

Equipment:

- Recycle System
 - Grinder with 3/8” Screen
 - Pilot Plant Wash System
 - Air Dry
 - Kice Elutriator
 - TEC Extruder
 - Desiccant Dryer
 - Solid State Polymerization Vessel
 - Arburg Injection Molder
- Test Equipment
 - Moisture Analyzer
 - Melt Indexer
 - Color Spectrophotometer
 - Differential Scanning Calorimeter
 - Vacuum Oven
 - Convection Oven
 - Viscometer



Path 1: Critical Guidance Protocol for Clear PET Resin and Molded Articles (PET-CG-01)



*This test protocol flow diagrams include a notice of copyright by APR, and appear on PFE's report with permission from APR. The flow diagram provided is a summary of the protocols for illustration. To view the complete test protocol, visit the APR's website.

APR PET Critical Guidance Summary for Test Material

Property	Critical Value	Control	Test 1	Test 2	Test 3	Test 4
Wash Floatables (%)	Record Value	0.00%	0.00%	0.00%	0.00%	0.00%
Elutriation Lights (%)	Record Value	0.71%	6.71%	11.01%	11.18%	7.44%
Clump Testing (%)	< 1% weight retention on screen	0.00%	0.00%	0.00%	0.00%	0.00%
Screen Pack Pressure Build (%)	End pressure no greater than 25% over starting pressure value	1.95%	7.80%	-6.62%	-1.11%	-9.09%
1 st Heat Processed Flake IV (dL/g)	Record Value	0.762	0.768	0.767	0.757	0.763
2 nd Heat Pellet IV (dL/g)	The value of IV loss for Test is no greater than 0.025 units when compared to Control	0.722	0.731	0.716	0.700	0.703
Melting Point SSP (°C)	Between 225 and 255°C	247.12	244.92	244.41	246.44	244.75
2 nd Heat SSP Pellet IV (dL/g)	Approximately 0.8 IV	0.798	0.825	0.820	0.805	0.798
Normalized (dL/g)	IV Delta of control and normalized value of test material: At 8 hours < 0.04 units and sample B SSP demonstrates 0.90 IV within 15 hours' time	0.835	0.850	0.834	0.842	0.850
Time to reach 0.9 IV (hours)		13	11	13	13	12
After SSP 3 rd Heat Plaque IV (dL/g)	The value of IV loss for Test is no greater than 0.025 units when compared to Control	0.684	0.701	0.723	0.713	0.724

APR PET Critical Guidance Summary for Test Material Continued

Property	Critical Value	Control	Test 1	Test 2	Test 3	Test 4
Before SSP Plaque Colors L*	Value > 82	92.26	92.77	N/A	N/A	90.84
Before SSP Plaque Colors a*	Delta to Control < 1.5	-0.26	-0.43	N/A	N/A	-0.46
Before SSP Plaque Colors b*	Delta to Control < 1.5	2.55	3.91	N/A	N/A	3.56
Before SSP Plaque Colors Haze %	Delta to Control < 10	11.62	3.95	N/A	N/A	4.72
Before SSP Black Specks	Control = 0, Test = 2 or less Control = 1, Test = 4 or less Control = 2, Test = 6 or less	0	0	N/A	N/A	0
After SSP Plaque Colors L*	Value > 82	93.65	92.95	90.19	92.36	89.20
After SSP Plaque Colors a*	Delta to Control < 1.5	-0.42	-0.67	-0.76	-0.65	-0.42
After SSP Plaque Colors b*	Delta to Control < 1.5	2.56	5.14	2.33	3.55	8.16
After SSP Plaque Colors Haze %	Delta to Control < 10	4.09	2.69	2.47	2.49	3.42
After SSP Black Specks	Control = 0, Test = 2 or less Control = 1, Test = 4 or less Control = 2, Test = 6 or less	0	0	0	0	0

Conclusions

1. Some haziness in the wash water was observed.
2. No floatables were observed.
3. No irregularities were observed during elutriation.
4. Clump testing did show favorable results, as the tests did not exceed clumping of more than 1% which is the max allowable limit in accordance with APR guidelines.
5. Pellet extrusion meets APR guidelines as a pressure build greater than 25% was not observed over a 30-minute run time.
6. IV build rate evaluation meets all APR guidelines.
7. After SSP, tests 1 and 4 did not meet all APR guidelines for b*.
 - a. Tests 2 and 3 showed a very low delta to control for b* value of -0.23 and 0.99 respectively, meeting the APR Guidelines.
8. IV data meets all APR guidelines.
9. Plaques meet the APR guidelines for black specks and inclusions analysis.



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Flake Processing and Testing

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APR Granulation

Test Summary:

Granulate incoming articles to the required grind size







Procedure:

Granulator is cleaned of any contaminating flake from previous runs. Articles are then manually fed into a grinder with screen holes ranging from 9.5-12 mm in diameter. Ground flake is then retained and packaged for washing. The granulator is cleaned between each variable.

Grind Process

Variable	Incoming Article	Ground Article
Control		
Test 1		
Test 2		

Grind Process Continued

Variable	Incoming Article	Ground Article
Control		
Test 3		
Test 4		

APR PET Wash and Sink/Float Evaluation

References:

PET-P-04

Test Summary:

Remove any contamination from PET and help in the liberation of labels, adhesives, coatings, and layers.

Procedure:

WASH:

Hot wash solution is prepared in a stainless-steel square vessel. Tap water, 1% Caustic, and 0.3% Triton X-100 or McDermid RP24 are all added to the solution. The wash is heated to 85°C and mixed with an impeller for 15 minutes at least 600 rpm. After 15 minutes, the impeller is stopped, and the mixture is removed from the heat source. The mixture sits for five minutes and floatables are skimmed off and saved. 1000mL of wash water is then retained.

RINSE:

The remaining washed PET flake sample is added to water at 45°C. The rinse is mixed with an impeller for five minutes at least 600 rpm. After the five minutes, the container is removed from heat and allowed to sit for five minutes. Floatables are then skimmed off and saved and 1000mL of the rinse water is retained.







SINK/FLOAT:

A tank with clean tap water is loaded with the rinsed PET flake. The tank is agitated for five minutes and then allowed to sit for five minutes. Floatable are skimmed off and saved


Data Table:

Run Number	Wash Temperature (°C)	Rinse Temperature (°C)	Starting Flake Weight (kg)	Total Floatables (grams)	% Total Floatables
Control	83	47	23	0.00	0.00%
Test 1	86	45	27	0.00	0.00%
Test 2	85	45	27	0.00	0.00%
Test 3	86	47	27	0.00	0.00%
Test 4	86	44	27	0.00	0.00%




Wash/Rinse Pictures

Variable	Settled Waters	Shaken Waters
Control	 <p>Two glass bottles with white caps. The left bottle is labeled '2084 - Control Wash Water' and the right is '2084 - Control Rinse Water'. Both contain clear, colorless liquids.</p>	 <p>Two glass bottles with white caps. The left bottle is labeled '2084 - Control Wash Water' and the right is '2084 - Control Rinse Water'. Both contain clear, colorless liquids with a thin layer of white foam on top.</p>
Test 1	 <p>Two glass bottles with white caps. The left bottle is labeled '2084 - Test 1 Wash Water' and the right is '2084 - Test 1 Rinse Water'. Both contain clear, colorless liquids.</p>	 <p>Two glass bottles with white caps. The left bottle is labeled '2084 - Test 1 Wash Water' and the right is '2084 - Test 1 Rinse Water'. Both contain clear, colorless liquids with a thin layer of white foam on top.</p>
Test 2	 <p>Two glass bottles with white caps. The left bottle is labeled '2084 - Test 2 Wash Water' and the right is '2084 - Test 2 Rinse Water'. Both contain clear, colorless liquids.</p>	 <p>Two glass bottles with white caps. The left bottle is labeled '2084 - Test 2 Wash Water' and the right is '2084 - Test 2 Rinse Water'. Both contain clear, colorless liquids with a thin layer of white foam on top.</p>




Wash/Rinse Pictures Continued

Variable	Settled Waters	Shaken Waters
Control		
Test 3		
Test 4		

Washed Flake/Floatable Pictures

Variable	Washed Flake	Floatables
Control		NOT APPLICABLE
Test 1		NOT APPLICABLE
Test 2		NOT APPLICABLE

Washed Flake/Floatable Pictures Continued

Variable	Washed Flake	Floatables
Control		NOT APPLICABLE
Test 3		NOT APPLICABLE
Test 4		NOT APPLICABLE

APR PET Elutriation

References:

PET-P-05

Test Summary:

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







Procedure:

The elutriator is calibrated using the control flake post wash. Air-dried washed control PET flake is sent through the elutriator; the air speed is adjusted until the value for 0.5% for lights is achieved. Once the elutriator is calibrated, the rest of the control and tests are run. Air speed, heavies weight, lights weight, and lights % are recorded for each test.







Data Table:

Run Number	Air Speed (mps)	Heavies Weight (kg)	Lights Weight (grams)	% Lights Removed
Control	5.75	21	150.50	0.71%
Test 1	5.75	20	1430.50	6.71%
Test 2	5.75	18	2232.00	11.01%
Test 3	5.75	18	2227.50	11.18%
Test 4	5.75	19	1500.50	7.44%

Heavies/Lights Pictures

Variable	Heavies	Lights
Control		
Test 1		
Test 2		

Heavies/Lights Pictures Continued

Variable	Heavies	Lights
Control		
Test 3		
Test 4		

Clumping Evaluation

References:

PET-S-08 (No Load)

Test Summary:

Evaluate the amount of clumping caused by low melting polymers or other contamination in washed and elutriated PET flake

Procedure:

Crystallize 1.5 kg of material at 165°C for 30 minutes breaking up every 15 minutes. Oven is then pre-heated to a temperature of 210°C. One kilogram of washed, elutriated, and crystallized PET flake is placed in an aluminum baking pan. The pan is then put in the oven for 90 minutes. After the 90 minutes, the flake is allowed to cool to room temperature. The pan is then emptied through a 12.5 mm screen. All agglomerated flake that does not sieve through or leave pan is weighed and recorded. For clump under load the same procedure is followed, but in addition a nine-kilogram weight is uniformly pressed on the flake.

Data Table:

Run Number	Stuck on Pan (g)	Stuck On Sieve (g)	% Clumped
Control	0.00	0.00	0.00%
Test 1	0.00	0.00	0.00%
Test 2	0.00	0.00	0.00%
Test 3	0.00	0.00	0.00%
Test 4	0.00	0.00	0.00%

APR Guidelines: Percent clumped cannot exceed 1% by weight retention on screen or pan for each variable.

1st Heat Processed Flake Colors

References:

ASTM D6290
 PET-CG-01

Test Summary:

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

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 pellets or flakes being tested. The cell is the inserted into the testing location 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%.

Equipment/Uncertainty: Konica Minolta 36dG / L*: ± 0.37, a*: ±0.07, b*: ±0.13

Data Table:

Run #	L * Values	a* Values	b* Values	L* Average	a* Average	b* Average
Control	73.70	-0.40	-0.79	73.12	-0.38	-1.04
	73.98	-0.28	-1.27			
	69.98	-0.42	0.07			
	76.16	-0.50	-2.10			
	71.80	-0.31	-1.12			
Test 1	77.18	-0.46	-1.56	72.63 -(0.49)	-0.40 -(0.02)	-1.17 -(0.13)
	71.07	-0.34	-1.95			
	68.20	-0.38	-0.85			
	74.93	-0.34	-1.24			
	71.77	-0.50	-0.24			
Test 2	75.00	-0.40	-2.94	69.33 -(3.80)	-0.30 (0.08)	-3.88 -(2.84)
	67.29	-0.36	-5.26			
	68.44	-0.33	-3.69			
	70.28	-0.19	-4.02			
	65.62	-0.23	-3.48			
Test 3	67.64	-0.36	-2.69	71.59 -(1.54)	-0.40 -(0.02)	-2.20 -(1.15)
	74.77	-0.43	-2.18			
	70.04	-0.33	-2.15			
	73.29	-0.46	-2.21			
	72.19	-0.42	-1.75			
Test 4	72.72	-0.36	-2.77	67.95 -(5.17)	-0.34 (0.04)	-2.56 -(1.52)
	68.12	-0.39	-2.73			
	63.94	-0.27	-1.17			
	68.88	-0.26	-3.21			
	66.11	-0.42	-2.93			

1st Heat Processed Flake Solution Intrinsic Viscosity

References:

ASTM D4603

Test Summary:

Determine the inherent viscosity of PET using a glass capillary viscometer.

Procedure:

Sample is weighed into a clean, dry volumetric flask. A stirring bar and solvent are added to the flask. The flask is then preheated to 110°C. After 15 minutes, the sample is inspected for undissolved PET (additional time may be required if the sample is not completely dissolved). The flasks are then removed and allowed to cool to room temperature. flasks are then put into a constant water bath at 30±0.01°C for ten minutes. The solution is then poured into the viscometer through a funnel and filter. The time it takes for the solution to reach the other set line is used to calculate the inherent viscosity of the plastic.

Data Table:

Run Number	Solution IV (dL/g)
Control	0.762
Test 1	0.768
Test 2	0.767
Test 3	0.757
Test 4	0.763

APR Guidelines: Difference in IV loss for control to test needs to be 0.025 units or less.



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Pellet Processing and Testing

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2nd Heat APR PET Extrusion

References:

PET-P-06

Test Summary:

Add a heat history to the material, homogenize blends, and filter out contamination

Procedure:

Extrusion material is prepped in a dryer for 4-6 hours until the moisture content is below 50 ppm. A screen pack of 20/40/250/40/20 is used for PET. PET is extruded at a target melt temperature of 280°C. The extruder is purged, and the screen pack is changed between each test innovation. Pressure and melt temperature are recorded throughout the run. Amorphous pellet is then crystallized before proceeding to injection.

Data Table:

Run Number	Moisture (ppm)	Average Melt Temperature (°C)	First 5 Minutes (psi)	Last 5 Minutes (psi)	Pressure Delta over Run	Number of screen Changes
Control	24	264	514	524	1.95%	0
Test 1	22	264	205	221	7.80%	0
Test 2	26	267	317	296	-6.62%	0
Test 3	36	266	270	267	-1.11%	0
Test 4	44	263	198	180	-9.09%	0

No fuming or stickiness was seen during extrusion.

APR Guidelines: Pressure delta over the run should no increase by more than 25%.

Standard Operating Conditions:

Zone 1 Temperature	Zone 2 Temperature	Zone 3 Temperature	Zone 4 Temperature	Zone 5 Temperature	Clamp Temperature	Die Temperature
271 °C	271 °C	271 °C	276 °C	276 °C	282 °C	282 °C

2nd Heat Control Extrusion Overview

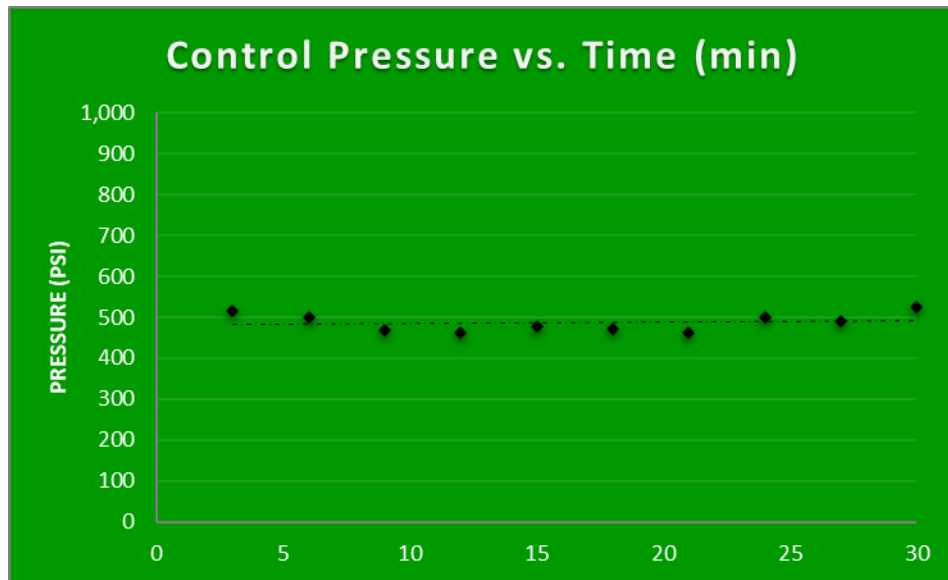
Amorphous Pellet



Screen



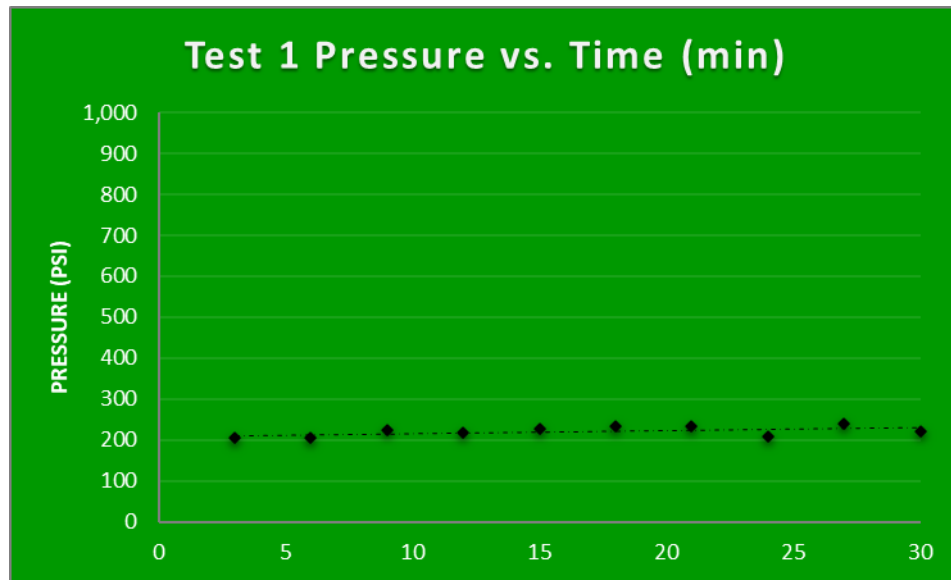
Extrusion Trace



2nd Heat Test 1 Extrusion Overview



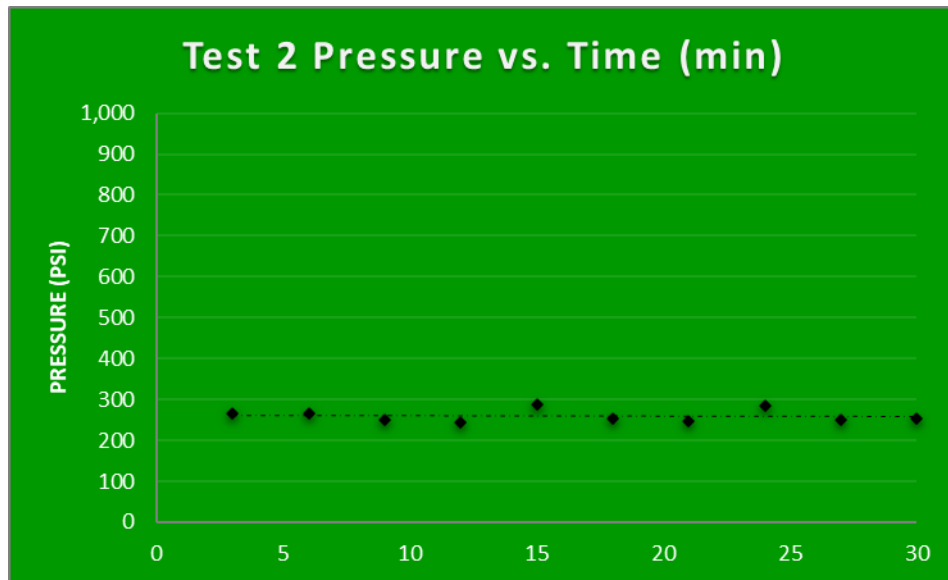
Extrusion Trace



2nd Heat Test 2 Extrusion Overview



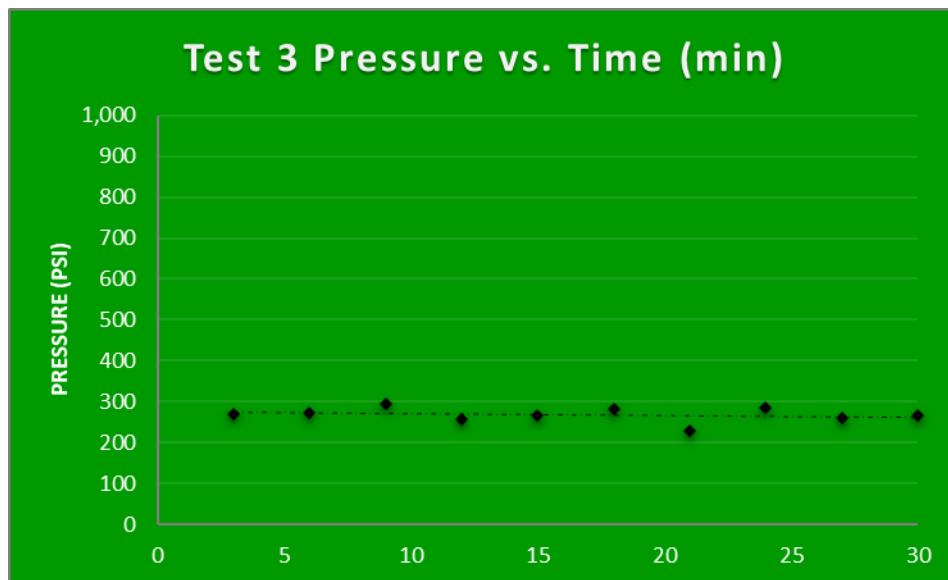
Extrusion Trace



2nd Heat Test 3 Extrusion Overview



Extrusion Trace



2nd Heat Test 4 Extrusion Overview

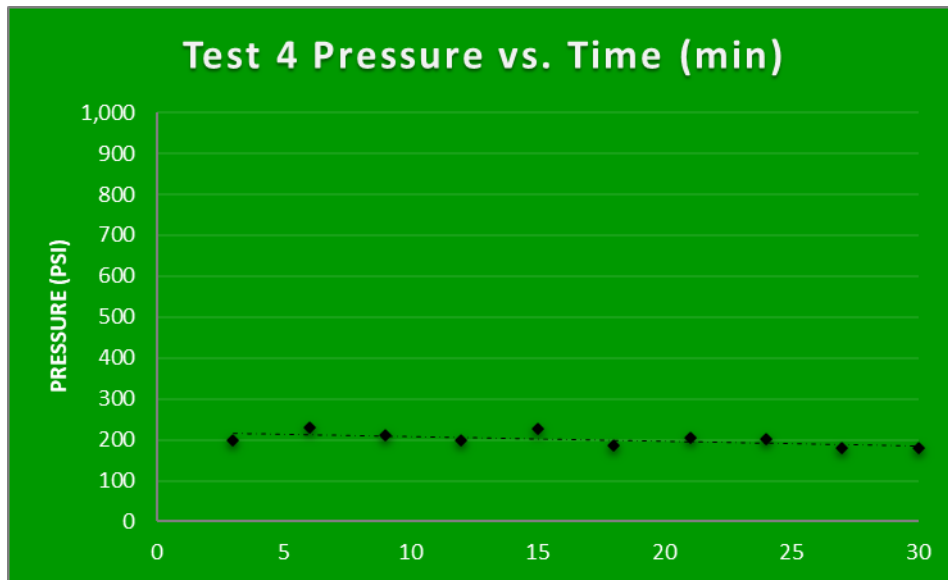
Amorphous Pellet



Screen



Extrusion Trace



2nd Heat Amorphous Pellet Colors

References:

ASTM D6290
PET-CG-01

Test Summary:

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

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 pellets or flakes being tested. The cell is the inserted into the testing location 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%.

Equipment/Uncertainty: Konica Minolta 36dG / L*: ± 0.37, a*: ±0.07, b*: ±0.13

Data Table:

Run #	L* Values	a* Values	b* Values	L* Average	a* Average	b* Average
Control	65.04	-0.70	0.07	64.80	-0.68	0.09
	64.49	-0.71	0.21			
	65.36	-0.67	0.04			
	65.04	-0.64	0.00			
	64.05	-0.70	0.15			
Test 1	65.02	-0.74	3.18	64.43 (-0.36)	-0.74 (-0.06)	3.14 (3.04)
	64.44	-0.76	3.09			
	64.14	-0.73	2.96			
	64.60	-0.77	3.31			
	63.96	-0.72	3.14			
Test 2	59.01	-0.76	-4.02	59.47 (-5.32)	-0.73 (-0.05)	-3.99 (-4.08)
	59.64	-0.74	-4.03			
	59.92	-0.70	-4.13			
	59.37	-0.76	-3.77			
	59.43	-0.70	-4.00			
Test 3	64.25	-0.86	-0.30	63.69 (-1.11)	-0.83 (-0.14)	-0.54 (-0.64)
	63.66	-0.85	-0.35			
	63.53	-0.83	-0.60			
	63.47	-0.83	-0.59			
	63.53	-0.77	-0.87			
Test 4	62.17	-0.71	0.72	62.51 (-2.28)	-0.70 (-0.01)	0.62 (0.53)
	62.61	-0.70	0.68			
	63.00	-0.69	0.55			
	61.92	-0.69	0.53			
	62.87	-0.70	0.62			

2nd Heat Pellet Solution Intrinsic Viscosity

References:

ASTM D4603

Test Summary:

Determine the inherent viscosity of PET using a glass capillary viscometer.

Procedure:

Sample is weighed into a clean, dry volumetric flask. A stirring bar and solvent are added to the flask. The flask is then preheated to 110°C. After 15 minutes, the sample is inspected for undissolved PET (additional time may be required if the sample is not completely dissolved). The flasks are then removed and allowed to cool to room temperature. flasks are then put into a constant water bath at 30±0.01°C for ten minutes. The solution is then poured into the viscometer through a funnel and filter. The time it takes for the solution to reach the other set line is used to calculate the inherent viscosity of the plastic.

Data Table:

Run Number	Solution IV (dL/g)	IV Drop from Incoming (dL/g)	IV Drop A'-B'
Control	0.722	0.040	N/A
Test 1	0.731	0.037	0.003
Test 2	0.716	0.051	-0.011
Test 3	0.700	0.057	-0.017
Test 4	0.703	0.060	-0.020

APR Guidelines: Difference in IV loss for control to test needs to be 0.025 units or less.



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Solid Stating Pellet Processing and Testing

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APR PET Solid Stating

References:

PET-P-07
ASTM D1238

Test Summary:

Build intrinsic viscosity of PET pellets

Procedure:

Amorphous pellet is loaded into solid state reactor. Using an oil heater, the material is crystallized at 160-170°C for approximately one hour under vacuum. The temperature is then increased to 210°C to induce solid state polymerization under vacuum. The time of solid stating is dependent on the desired intrinsic viscosity. Randomly selected material is tested every hour to measure intrinsic viscosity as to not overshoot the desired value. Material temperature, vacuum level, oil temperature, and intrinsic viscosity are recorded every hour. This test is completed in a conditioned laboratory at a temperature of 23±2°C and a relative humidity of 50±10%.

Equipment / Uncertainty: Dynisco LMI5500 / ±0.02 dL/g

Process Data Table:

Run Number	Oil Temperature (°C)	Material Temperature (°C)	Vacuum Pressure (inHg Gauge)
Control	223	207	15
Test 1	222	206	15
Test 2	223	206	15
Test 3	223	206	15
Test 4	223	206	15

IV Data Table:

Run Number	Starting Material IV (dL/g)	Ending Material IV (dL/g)	IV Build Rate (dL/g/hour)
Control	0.722	0.798	0.014
Test 1	0.731	0.825	0.016
Test 2	0.716	0.820	0.014
Test 3	0.700	0.805	0.015
Test 4	0.703	0.798	0.016

APR Guidelines: No sticking or residue in SSP unit and material reaches desired IV of 0.80 dL/g

2nd Heat SSP Pellet Colors

References:

ASTM D6290
 PET-CG-01

Test Summary:

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

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 pellets or flakes being tested. The cell is the inserted into the testing location 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%.

Equipment/Uncertainty: Konica Minolta 36dG / L*: ± 0.37, a*: ±0.07, b*: ±0.13

Data Table:

Run #	L* Values	a* Values	b* Values	L* Average	a* Average	b* Average
Control	84.08	-1.12	0.76	83.88	-1.13	0.81
	84.10	-1.14	0.84			
	84.09	-1.11	0.82			
	82.27	-1.19	0.96			
	84.84	-1.10	0.67			
Test 1	83.92	-0.90	3.31	83.28 (-0.60)	-0.87 (0.26)	3.59 (2.78)
	83.13	-0.90	3.67			
	83.75	-0.86	3.41			
	82.38	-0.85	3.96			
	83.22	-0.83	3.58			
Test 2	79.26	-1.71	-2.82	78.28 (-5.59)	-1.72 (-0.58)	-2.69 (-3.50)
	77.63	-1.76	-2.70			
	78.87	-1.72	-2.53			
	77.57	-1.72	-2.87			
	78.09	-1.67	-2.51			
Test 3	81.33	-1.39	0.45	80.71 (-3.16)	-1.39 (-0.25)	0.45 (-0.36)
	80.59	-1.41	0.32			
	81.14	-1.40	0.45			
	78.81	-1.41	0.56			
	81.69	-1.32	0.46			
Test 4	79.84	-0.36	3.20	78.90 (-4.98)	-0.38 (0.75)	3.40 (2.59)
	77.34	-0.44	3.13			
	79.50	-0.39	3.72			
	78.37	-0.35	3.53			
	79.44	-0.38	3.43			

APR 8-Hour IV Build Evaluation

References:

PET-S-07
PET-CG-0
ASTM D1238

Test Summary:

Measures the rate of extrusion of molten resins through a specified die at specified conditions

Procedure:

Sample Preparation

Randomly selected PET sample weighed out to ten grams and placed in a clean aluminum dish. Vacuum oven is set to $210 \pm 5^\circ\text{C}$, once the oven reached this temperature a vacuum is pulled. Vacuum is broken with a nitrogen purge and the samples are placed in the oven. A vacuum is then immediately pulled, and the samples are allowed to heat up to temperature. The samples are removed and tested after eight hours of IV build time.

Sample Testing

Melt Indexer is set to $285 \pm 0.2^\circ\text{C}$. The PET sample is then transferred from the oven to the equipment where it is loaded. The cylinder (100 grams) and weight (2060 grams) are placed in the machine with a plug blocking the PET from escaping. The PET is softened for 7 ± 0.5 minutes, at the two-minute mark the plug is removed allowing the PET to flow. Extrudate is the automatically timed as to obtain a melt flow rate as well as a melt intrinsic viscosity. This test is completed in a conditioned laboratory at a temperature of $23 \pm 2^\circ\text{C}$ and a relative humidity of $50 \pm 10\%$.

Equipment / Uncertainty: Dynisco LMI5500 / ± 0.02 dL/g

Data Table:

Run Number	Start IV (dL/g)	End IV (dL/g)	Normalized IV (dL/g)	IV Build Rate (dL/g/hr)	Time to 0.90 IV (hours)
Control	0.722	0.834	0.834	0.014	13
Test 1	0.731	0.859	0.850	0.016	11
Test 2	0.716	0.828	0.834	0.014	13
Test 3	0.700	0.82	0.842	0.015	13
Test 4	0.703	0.807	0.850	0.016	12

APR Guidelines: IV delta of control and normalized IV value of the test material at eight hours is not to exceed 0.04dL/g. Normalized MIV indicates the calculated 8-Hour MIV based off the control starting MIV + the IV build rate observed during the IV Build Evaluation. IV build evaluation must display material ability to achieve a 0.90 IV within a 15-hour process.

PET Differential Scanning Calorimetry (DSC)

References:

ASTM D3418
PET-CG-01

Test Summary:

Evaluates the temperatures and enthalpies of fusion and crystallization of polymers

Procedure:

Randomly selected test material is cut down to a size between 1-5 mg. The polyolefin piece is then prepped in an aluminum pan/lid container and crimped to allow complete contact between the polyolefin piece and the aluminum. The container now holding the prepped polyolefin is inserted into the DSC. A test method is chosen dependent on the customers proposal. This test is completed in a conditioned laboratory at a temperature of $23\pm 2^{\circ}\text{C}$ and a relative humidity of $50\pm 10\%$.

For ASTM HDPE-CG-01/ASTM D3418,

The DSC heated to $\sim 30^{\circ}\text{C}$ above the suspected temperature of fusion (melt temperature) at $10^{\circ}\text{C}/\text{min}$ and then cooled to room temperature.

This method leaves a trace that can be assessed allowing the determination of temperatures and enthalpies of fusion and crystallization of the polymer. From these values percent crystallinity can be calculated. The traces can also allow for identification of any contaminants/barrier polymers.

Equipment/Uncertainty: TA instruments DCS Q20 / $\pm 0.8^{\circ}\text{C}$ and $\pm 6.0 \text{ J/g}$

Data Table:

Variable	Primary Peak Temperature ($^{\circ}\text{C}$)	Secondary Peak Temperature ($^{\circ}\text{C}$)	Primary Peak (J/g)	Secondary Peak (J/g)
Control	247.12	N/A	36.14	N/A
Test 1	244.92	N/A	34.72	N/A
Test 2	244.41	N/A	30.10	N/A
Test 3	246.44	N/A	32.92	N/A
Test 4	244.75	N/A	34.53	N/A

APR Guidelines: The primary peak temperature should fall between 225 and 255°C



Plastics Forming Enterprises, LLC

Plastics Consulting, Research and Engineering

Plaque Processing and Testing

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3rd Heat APR 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 50PPM. PET is injection molded at a target melt temperature of 275°C. The injection unit is purged between each test innovation. Melt temperature, room temperature, and mold temperature are recorded for each innovation variable.

Data Table:

Run Number	Moisture Content (ppm)	Melt Temperature (°C)	Mold Temperature (°C)
After SSP Control	9	276	21
After SSP Test 1	22	276	23
After SSP Test 2	13	276	20
After SSP Test 3	15	276	19
After SSP Test 4	23	276	19

APR Guidelines: Moisture content of material needs to be below 50ppm, nozzle melt temperature of 275°C, mold temperature of 20°C.

Standard Operating Conditions:

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

BSSP 3rd Heat Injection Molded Plaques Pictures



BSSP 3rd Heat Transmission Plaque Colors and Black Specks/Inclusions

References:

PET-P-08, PET-S-09

Test Summary:

Measure the color values L*, a*, b*, and haze of a 3mm injection molded plaque. Visually identify the presence of black specks and inclusions. Data is designed to be correlated to production of reproduced bottles.

Procedure:

Flakes/Pellets are dried to below 50PPM and injection molded into 3mm plaques. 50 molded plaques are visually examined for black speck or inclusions of 0.1mm² in size

The color analyzing equipment is set up on transmittance and calibrated using pure white and black standards.

The molded plaque is the inserted into the testing location and the equipment runs the color test. The test indicates L*, a*, b* values and haze percentage.

Run #	Black Specks	L * Values	a* Values	b* Values	L* Average	a* Average	b* Average	Haze	Haze Average
Control	0	92.91	-0.26	2.58	92.96	-0.26	2.55	11.85	11.62
		92.97	-0.24	2.48				11.53	
		92.98	-0.30	2.69				10.69	
		92.90	-0.23	2.51				12.26	
		93.04	-0.26	2.48				11.77	
Test 1	0	92.81	-0.47	4.11	92.77 (-0.19)	-0.43 (-0.17)	3.91 (1.36)	3.51	3.95 (-7.67)
		92.87	-0.50	4.16				3.66	
		92.77	-0.40	3.71				3.93	
		92.73	-0.37	3.58				4.22	
		92.69	-0.42	3.97				4.41	
Test 4	0	90.87	-0.46	3.47	90.84 (-2.12)	-0.46 (-0.21)	3.56 (1.01)	4.79	4.72 (-6.90)
		90.85	-0.44	3.33				4.97	
		90.81	-0.47	3.64				4.38	
		90.88	-0.50	3.76				4.24	
		90.77	-0.45	3.61				5.21	

APR Guidelines: L* value cannot be less than 82, a* value and b* value cannot be more than a 1.5 difference when compared to control, and haze cannot differ from the control by more the 10%. Control haze value cannot exceed 9.5%, and b* must be below 4.

ASSP 3rd Heat Injection Molded Plaques Pictures

After SSP Control

After SSP Test 1

After SSP Test 2



After SSP Control

After SSP Test 3

After SSP Test 4



ASSP 3rd Heat Transmission Plaque Colors and Black Specks/Inclusions

References:

PET-P-08, PET-S-09

Test Summary:

Measure the color values L*, a*, b*, and haze of a 3mm injection molded plaque. Visually identify the presence of black specks and inclusions. Data is designed to be correlated to production of reproduced bottles.

Procedure:

Flakes/Pellets are dried to below 50PPM and injection molded into 3mm plaques. 50 molded plaques are visually examined for black speck or inclusions of 0.1mm² in size

The color analyzing equipment is set up on transmittance and calibrated using pure white and black standards.

The molded plaque is the inserted into the testing location and the equipment runs the color test. The test indicates L*, a*, b* values and haze percentage.

Run #	Black Specks	L * Values	a* Values	b* Values	L* Average	a* Average	b* Average	Haze	Haze Average
Control	0	93.67	-0.41	2.52	93.65	-0.42	2.56	4.09	4.09
		93.62	-0.42	2.58				4.17	
		93.65	-0.43	2.55				4.12	
		93.62	-0.45	2.72				3.91	
		93.67	-0.40	2.43				4.16	
Test 1	0	93.01	-0.68	5.16	92.95 (-0.70)	-0.67 (-0.25)	5.14 (2.58)	2.39	2.69 (-1.40)
		92.88	-0.66	5.21				2.27	
		93.04	-0.69	5.08				2.73	
		92.95	-0.67	5.07				2.87	
		92.87	-0.65	5.17				3.17	
Test 2	0	90.18	-0.76	2.33	90.19 (-3.45)	-0.76 (0.34)	2.33 (-0.23)	2.51	2.47 (-1.62)
		90.21	-0.76	2.30				2.44	
		90.21	-0.77	2.33				2.38	
		90.22	-0.77	2.29				2.72	
		90.15	-0.76	2.39				2.30	
Test 3	0	92.38	-0.66	3.50	92.36 (-1.29)	-0.65 (0.23)	3.55 (0.99)	2.58	2.49 (-1.60)
		92.36	-0.64	3.55				2.55	
		92.37	-0.65	3.52				2.46	
		92.33	-0.66	3.60				2.38	
		92.34	-0.64	3.56				2.48	
Test 4	0	89.26	-0.44	8.13	89.20 (-4.44)	-0.42 (0.00)	8.16 (5.60)	3.21	3.42 (-0.67)
		89.07	-0.38	8.10				3.83	
		89.21	-0.41	8.20				3.60	
		89.22	-0.44	8.27				3.24	
		89.26	-0.45	8.12				3.23	

APR Guidelines: L* value cannot be less than 82, a* value and b* value cannot be more than a 1.5 difference when compared to control, and haze cannot differ from the control by more the 10%. Control haze value cannot exceed 9.5%, and b* must be below 4.

After SSP 3rd Heat Plaque Melt Indexer

References:

PET-CG-01
ASTM D1238

Test Summary:

Measures the rate of extrusion of molten resins through a specified die at specified conditions

Procedure:

Sample Preparation

Randomly selected PET sample weighed out to ten grams and placed in a clean aluminum dish. Vacuum oven is set to $160 \pm 5^\circ\text{C}$, once the oven reached this temperature a vacuum is pulled. PET is dried for at least 4 hours before testing.

Sample Testing

Testing equipment is set to $285 \pm 0.2^\circ\text{C}$. The randomly selected PET sample is then transferred from the oven to the testing equipment where it is loaded. The cylinder (100 grams) and weight (2060 grams) are placed in the machine with a plug blocking the PET from escaping. The PET is softened for 7 ± 0.5 minutes, at the two-minute mark the plug is removed allowing the PET to flow. Extrudate is the automatically timed as to obtain a melt flow rate as well as a melt intrinsic viscosity. This test is completed in a conditioned laboratory at a temperature of $23 \pm 2^\circ\text{C}$ and a relative humidity of $50 \pm 10\%$.

Equipment / Uncertainty: Dynisco LMI5500 / ± 0.02 dL/g

Data Table:

Run Number	Plaque Melt IV (dL/g)	IV Drop from Pellet (dL/g)	IV Drop A'-B'
Control	0.684	0.114	N/A
Test 1	0.701	0.124	-0.010
Test 2	0.723	0.097	0.017
Test 3	0.713	0.092	0.022
Test 4	0.724	0.098	0.016

APR Guidelines: Delta in loss of MIV between incoming flake/pellet and extruded flake/pellet compared to control ± 0.025 dL/g

Report Statements

Next Steps Statement:

In the event that all results are favorable for APR CG, please understand that these results should not be represented as certification or compliance with the APR protocol. We strongly encourage you to submit these results to APR for formal recognition. We would be happy to work with you to guide you through this process.

Conformance Statement:

Plastics Forming Enterprise evaluated the material through testing that conforms to the Critical Guidance Document testing protocols as described, omitting no steps. The Evaluation was processed according to the APR's Critical Guidance Protocol for Clear PET Resin and Molded Articles Path 1.

Conclusions Statement:

Any interpretations of data are done by experienced staff members with a good knowledge of the materials used during testing. PFE prides itself on being able to help the clientele understand possible failures with over 40 years of experience in plastics recycling. All equipment uncertainty values were obtained through internal experimentation using 95% confidence intervals.

Amendments:

Date of Amendment	What was Amended?	Reason for Amendment

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