APPENDIX D

PIPE CONTAINING RECYCLED HDPE

APPENDIX D – PIPE CONTAINING RECYCLED HDPE

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D.1 TRIAL PIPE MANUFACTURING

A total of 15 trial pipe samples were prepared at three different manufacturing plants. Five, twenty feet long samples were made from each formulation for a total of 1500 feet of pipe. The manufacturing plants were the Advanced Drainage Systems plant in Ennis, Texas (Plant A), the Blue Diamond Industries plant in Roseboro, North Carolina (Plant B), and the Lane Enterprises plant in Wytheville, Virginia (Plant L). The formulations made at the three plants are shown in Table D-1.

Table D-1 - Trial Pipe Formulations

Sample	% VR1[1]	% Virgin MDPE	% PCR-MCR[2]	% PCR-NAT[3]
A1	100			
A2	85		15	
A3	85			15
A4	50	20	30	
A5	40	30		30
B1	100			
B2	50	20	30	
B3	20	40	24	16
B4		50		50
B5		40	36	24
L1[4]				
L2	50 (VR2)	20	30	
L3	100 (VR2)			
L4	100			
L5	50	20	30	

1. Virgin Resin 1
2. Post-Consumer Recycle Mixed Colored Reprocessed
3. Post-Consumer Recycled Natural Reprocessed
4. Proprietary formulated pipe resin containing about 50% recycled.

Each plant made a sample from 100% Virgin Resin 1 and a 50/20/30 blend of VR1, MDPE, and Mixed Color Reprocessed. All of the recycled resins were supplied by a single recycled processor and each plant received material from the same batch.

Sample L1 was a proprietary pipe resin formulated by the recycled resin supplier. This was included as a representation of the type of resin that could be supplied by the recycled resin companies.

There were no difficulties encountered during the pipe trials. Each manufacturer tried to keep the pipe weight constant during the trial. The only processing adjustment needed was a change in extruder output. It should be noted, however, that these trial runs were

very short, the conditions were not optimized for pipe stiffness, and the effects of using recycled resins over longer production runs was not evaluated.

D.2 PIPE INDEX TEST RESULTS

After the 15 samples pipes were manufactured, their properties were measured on compression molded plaques made from the pipe. All the plaques were made in accordance with ASTM D4703 at a cooling rate of 15ºC/min. Complete reports are given for each pipe formulation in section D.11. This section will focus on specific properties that may be important for future specifications.

D.2.1 Density

The density of base resins for AASHTO M294 pipe must be between 0.948 and 0.955 g/cm^3. This is Cell Class 4, according to ASTM D3350. When directly measured, all but 4 of the samples had densities higher than the upper limit of 0.955 g/cm^3. However, these values can be corrected for the % carbon black, according to the relationship $D_{corr} = D - 0.0044C$, where C is the % black. Density values can also be determined from other material properties, such as the yield stress or flexural modulus. The equation relating density to yield stress is:

Yield Stress = 81,250 x Density – 73,500

when yield stress is expressed as psi and density as g/cm^3.

The equation relating flexural modulus to density is:

Flex. Mod. = 4.46 x 10^6 x Density – 4.10 x 10^6

when flexural modulus is in psi and density in g/cm^3.

The original values, the corrected values, and the values calculated from the yield stresses and flexural moduli are shown in Table D-2. They are also shown graphically in Figure D-1.

The samples marked with an asterisk are samples containing only virgin resin and carbon black. If the other contaminants like particulates and polypropylene affect the density measurement, one would expect these samples to have the best correlation between the different methods to determine density.

It appears that the density measured in a gradient density column and corrected for % carbon black is the least accurate method because this value shows the largest disagreement from the other two values. The direct density measurement might also be influenced by the very small size of the test specimen. A piece around 1/16” x 1/16” is often the size tested. If this small piece contains a particle or an air bubble, the number could be skewed in either direction.

Table D-2 - Pipe Formulation Density Values

Sample	Density (g/cm^3)	Corrected for % Black (g/cm^3)	Calculated from Yield Stress (g/cm^3)	Calculate from Flex. Mod. (g/cm^3)
A1*	0.959	0.952	0.952	0.955
A2	0.949	**0.943**	0.951	0.953
A3	0.973	**0.963**	0.952	0.955
A4	0.954	0.952	0.950	0.952
A5	0.955	0.948	0.949	0.951
B1*	0.960	0.951	0.952	0.953
B2	0.957	0.948	0.949	0.950
B3	0.957	0.948	**0.945**	0.948
B4	0.954	0.948	0.948	0.949
B5	0.960	0.952	**0.945**	0.948
L1	0.959	0.950	0.949	0.950
L2	0.957	0.950	0.951	0.950
L3*	0.958	0.952	0.955	0.953
L4*	0.957	**0.946**	0.955	0.953
L5	0.957	**0.946**	0.953	0.951

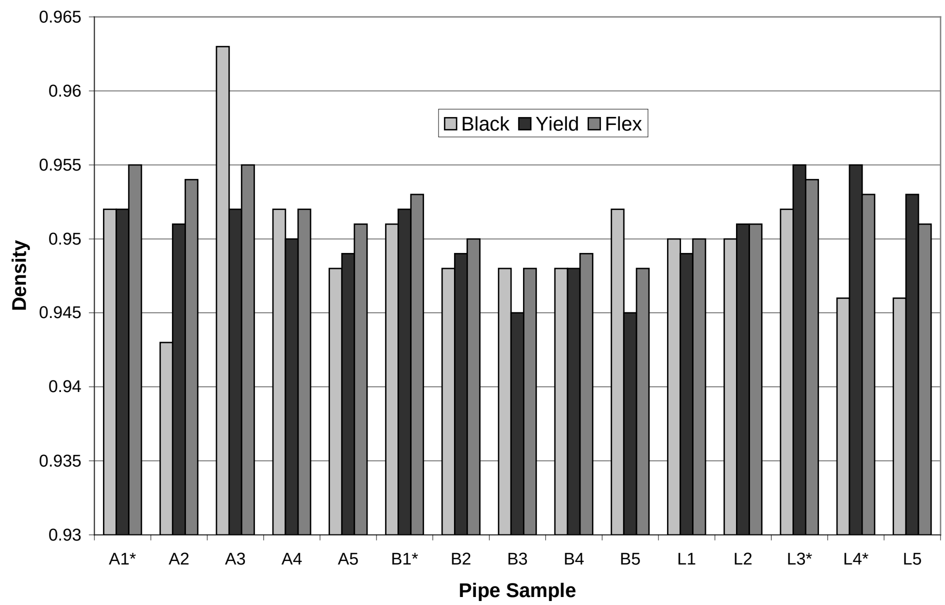

Figure D-1 - Density of 15 Pipe Samples Generated Three Ways

All of these results suggest that for the specification of resin blends containing PCR-HDPE, the density is of limited value. It would be better to simply specify mechanical properties that demonstrate what the density is. Two examples are shown in Table D-3.

Table D-3 - Mechanical Properties Related to Base Resin Density

Property	Range Equivalent to Density Cell Class 4 (>0.947-0.955 g/cm^3)
Tensile Yield Stress (psi)	3500 – 4100
Flexural Modulus (psi)	130,000 – 160,000

These are the required mechanical property values to ensure that the base resin density was in the range specified by AASHTO M-294. Unfortunately, the yield stress and flexural modulus values do not fit neatly into cell classes themselves. Cell class 5 for Yield Stress is 3500 – 4000 psi, so one could specify a cell class of 5 or higher. If the density were too high, the stress crack resistance would suffer. The cell class 5 for flexural modulus is broad (110,000 – 160,000), but could be used. A resin too low in flexural modulus would also be below the minimum yield stress of 3500 psi.

D.2.2 Melt Index

The melt flow index values for the 15 pipe formulations are shown in Figure D-2. From knowing the melt index values for the individual components, and the fact that the MI is an exponential relationship with percentage composition, one can calculate the theoretical values for most of the samples.

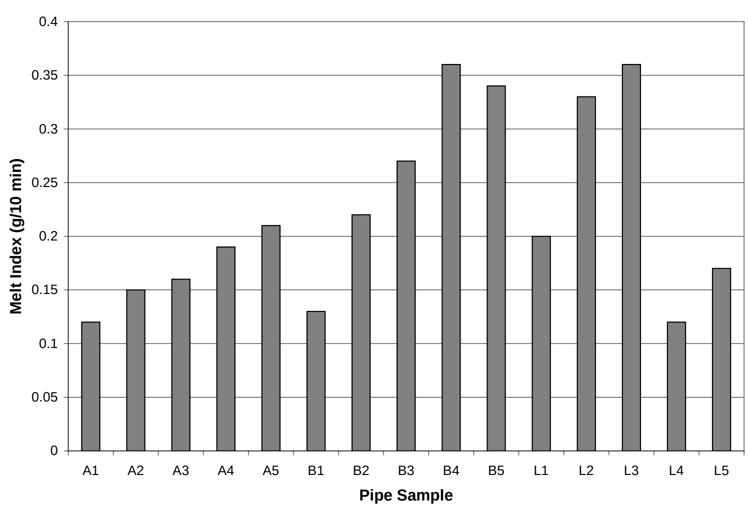

Figure D-2 - Melt Index of 15 Pipe Samples

This is shown in Table D-4. The agreement between the predicted and actual values is excellent, except in a few cases where they were different by more than 0.02 g/10 min. The correlation coefficient (R^2) for a line of actual vs. predicted was 0.85.

Table D-4 - Comparison Between Predicted and Measured Melt Index Values

Sample	Predicted MI (g/10 min)	Measured MI (g/10 min)
A2	0.15	0.15
A3	0.16	0.16
A4	0.20	0.19
A5	0.24	0.21
B2	0.20	0.22
B3	0.27	0.27
B4	0.29	0.36
B5	0.37	0.34
L2	0.35	0.33
L5	0.20	0.17

D.2.3 Percentage Color

The MCR-PCR HDPE only contained about 0.05% color, so it can be assumed that the % color measured was nearly all carbon black. The result from the 15 pipe formulations are shown in Figure D-3.

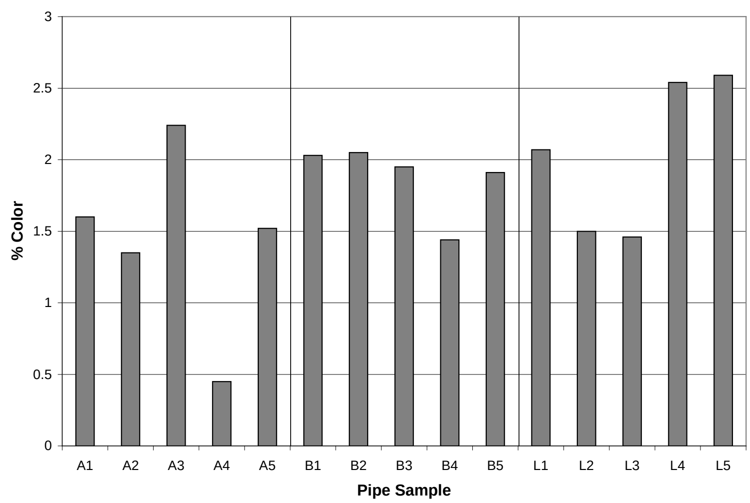

Figure D-3 - Percentage Carbon Black of 15 Pipe Samples

Each manufacturer was asked to control the % carbon black to a range from 2-3%. Obviously, % carbon black is something that is not well controlled in the corrugated pipe market.

D.2.4 Percentage Ash

The percentage ash should correlate well with the % PCR-MCR because the virgin resins typically have less than 0.1% ash. Also, the PCR-NAT has low ash because this recycled source is much cleaner because the milk bottles are lifted out of the waste stream, so the large majority of the contamination stays with the mixed colored bottles.

The percentage ash values measured are plotted in Figure D-4 along with the predicted values. The prediction was based on the PCR-MCR ash content of 1.2% and the assumption that virgin resins and PCR-NAT have very little ash. There is general agreement between actual and predicted values in some cases, but there are also samples like A1, A3, A5 and B4 that have ash without containing MCR. This suggests that there is a small amount of ash in the NAT recycled material or in the carbon black masterbatch. The correlation coefficient (R^2) for a line of actual vs. predicted was 0.75.

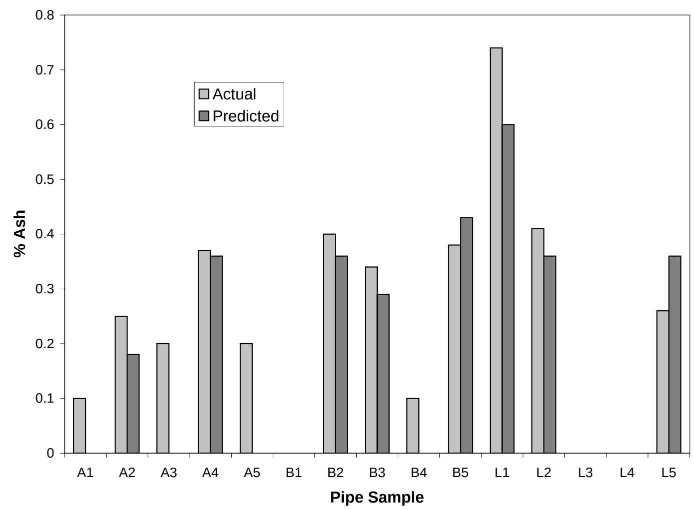

Figure D-4 - Percentage Ash Found in 15 Pipe Samples

D.2.5 Percentage Polypropylene

The percentage polypropylene from the bottle closures is shown for each pipe sample in Figure D-5. The actual and predicted values are presented. Similarly to the ash samples, the three samples containing PCR-NAT and not PCR-MCR each showed a small amount of polypropylene. This suggests that the PCR-NAT or the color concentrate had a small amount of PP. Unfortunately, the PCR-NAT was not tested for %PP. The correlation coefficient (R^2) was 0.91 for the line of actual vs. predicted, excluding any 0,0 points.

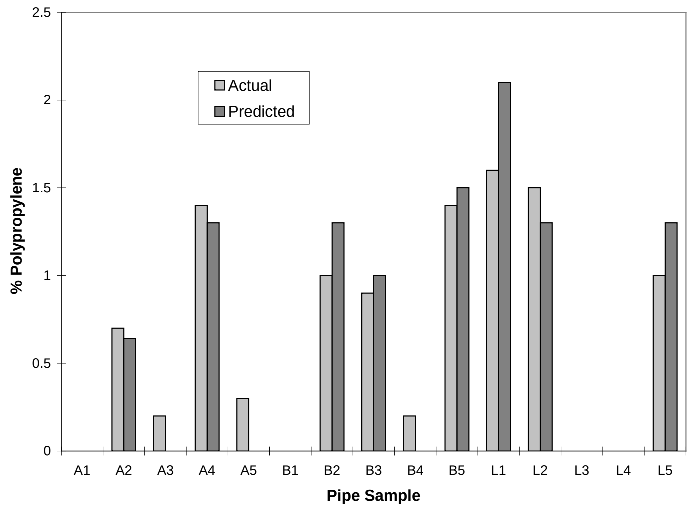

Figure D-5 - Percentage Polypropylene Found in 15 Pipe Samples

D.2.6 Tensile Yield Stress

The tensile yield strength values for the 15 pipe samples are shown in Figure D-6. Notice that all of the pipe samples meet the AASHTO M294 minimum of 3000 psi, which is cell class 4. If one were to specify a minimum tensile strength for cell class 5 (3500 psi), samples B3 and B5 would not meet the cell class. The strength values could be raised in these two formulations by adjusting the virgin to MDPE ratio. An increase of 10% VR1 and a reduction of 10% MDPE would make both these formulations meet 3500 psi for yield stress. Of course, the stress crack resistance and perhaps the long-term performance will change also.

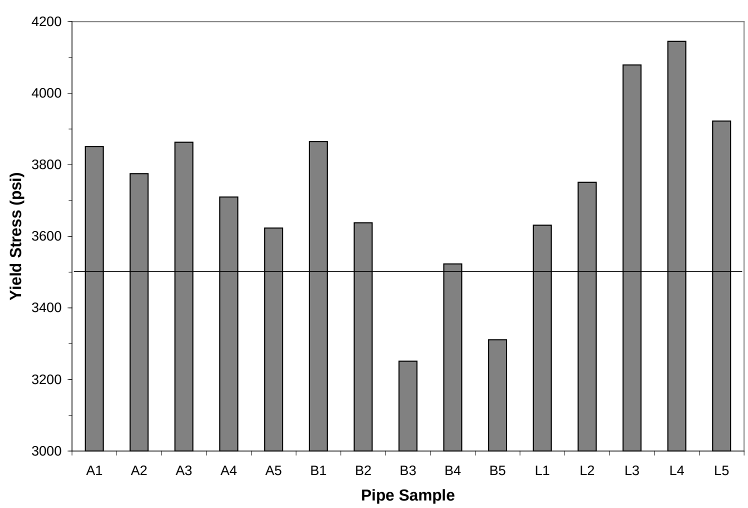

Figure D-6 - Tensile Yield Stress of 15 Pipe Samples

D.2.7 Tensile Break Strain

The break strain of the 15 pipe samples are shown in Figure D-7.

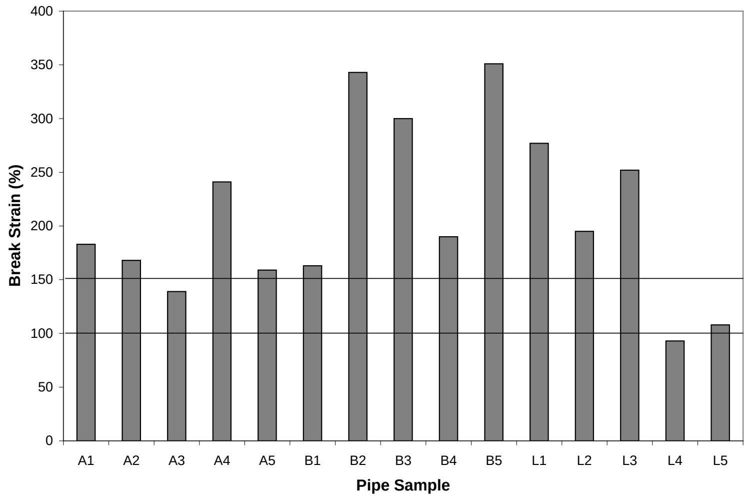

Figure D-7. Tensile Break Strain of 15 Pipe Samples

D.2.8 Flexural Modulus

The flexural modulus results for the 15 pipe samples are shown in Figure D-8. All the values easily exceed the AASHTO M294 cell class minimum requirement of 110,000 psi (Cell Class 5) and generally track with yield strength.

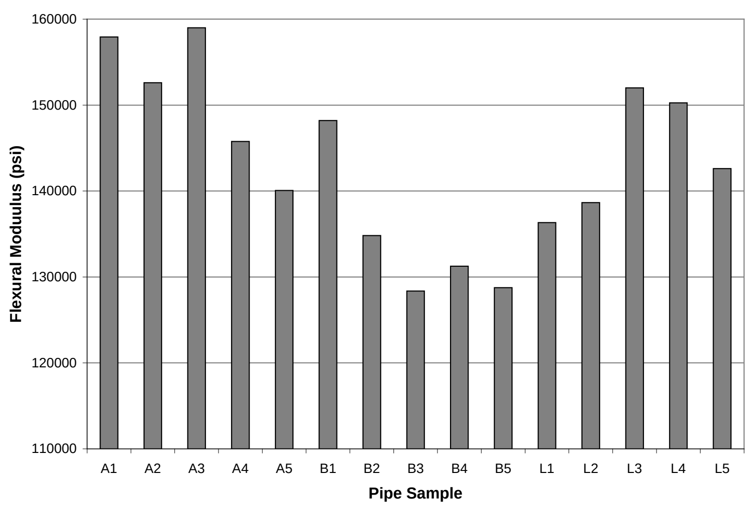

Figure D-8 - Flexural Modulus of 15 Pipe Samples

D.2.9 Thermal Stability (OITemp)

The OITemp values for all 15 pipe samples are shown in Figure D-9. Notice that they all easily meet the required value of 220ºC found in ASTM D3350. Recall that this property was adopted by the Plastic Pipe Institute as part of the resin requirements for AASHTO M294. However, this test is really not appropriate for corrugated drainage pipe. The OITemp is mainly used in solid wall pipe to determine if the inside surface of the pipe has been oxidized during processing. A scraping is taken from the inside surface and tested. The minimum value of 220ºC is close to the value one would obtain from flake resin without any antioxidants or processing stabilizers. Therefore, it shows that oxidation of the inside surface has not occurred rather than show that there are protective stabilizers present in the compounded resin.

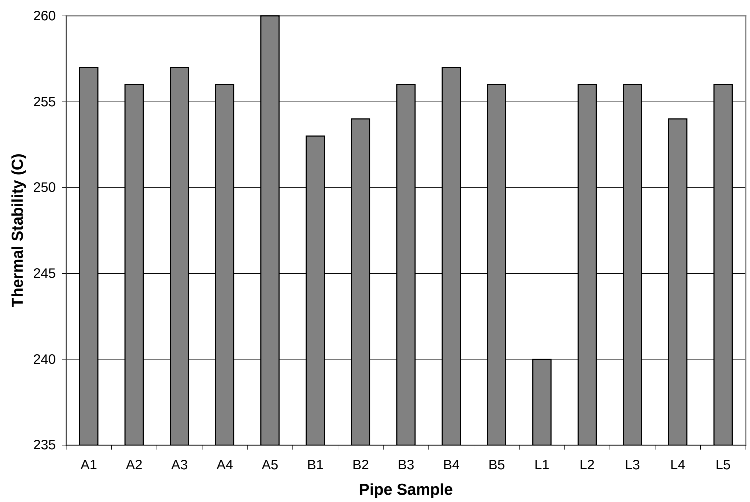

Figure D-9 - Thermal Stability (OITemp) of Pipe Samples

D.2.10 Oxidative Induction Time (OIT)

A test that gives some basic information about the additive package is the OIT test (ASTM D3895). The results for the 15 pipe samples are shown in Figure D-10. Notice that the OIT test can distinguish different materials better than the OITemp. However, it cannot be overstated that the OIT test just confirms that something has been added to the resin, it cannot say what was added or how well it will perform in service. Table D-5 shows the OIT values for different components and additive packages. The purpose of the table is to show that different additives have different OIT times at the same concentration. A package like 1000 ppm of Irganox 1010 and 750 ppm of Irgaphos 168 would be a good additive package for this application. Notice the OIT would be well over 50 minutes.

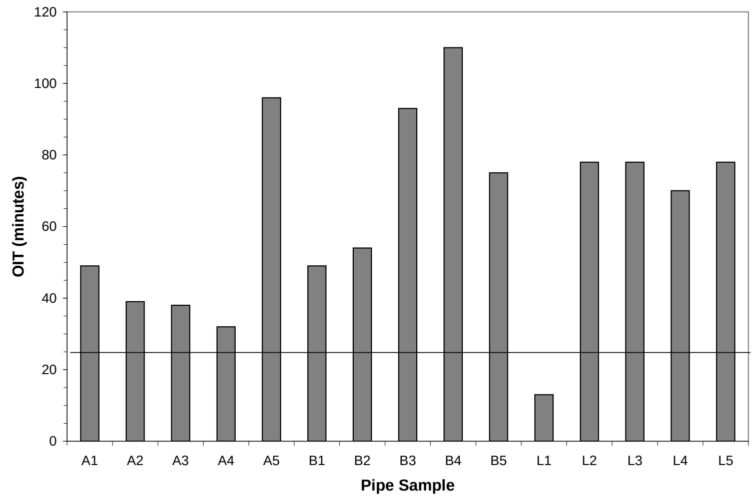

Figure D-10 - OIT of 15 Pipe Samples

Table D-5 - OIT Values for Additives in Geomembranes (1)

Ingredient	Concentration (ppm)	OIT at 200ºC (min)
Irganox 1010	1000	56
Irganox 1076	1000	23
Santowhite Crystals	1000	23
Irgaphos 168	1000	6
Irganox 1010 + Irgaphos 168	1500 + 750	87
Irganox 1010 + Irgaphos 168	3000 + 750	125

D.3 STRESS CRACK TEST RESULTS

D.3.1 Notched Stress Crack Tests (NCLS and 15%NCTL)

There were two different stress crack tests performed on the plaques made from the 15 pipe samples. The first was the NCLS test (ASTM F2136), where each samples is loaded at a constant ligament stress of 600 psi. The 600 psi is a result of taking 15% of 4000 psi, which is about the yield stress of the PPI certified resins for corrugated pipe. So the test basically assumes most of the samples tested will have a yield stress near 4000 psi. The NCTL test (ASTM D5397, Appendix) applies a load based on the actual yield stress of the material tested. Since the yield stresses measured on the 15 pipes ranged from 3251 to 4145 psi, many of the samples were not very close to 4000 psi. Therefore, the NCTL test was also used at an applied stress equal to 15% of the measured yield stress. This means that the applied stresses varied from 488 to 622 psi.

Both the NCLS and 15% NCTL results are seen in Figure D-11. Notice that the 15% NCTL results are almost always higher than the NCLS. This is due to the fact that most of the yield stresses are less than 4000 psi, so the 600 psi is a higher load than 15% of the

actual yield stress. Two lines are shown on the graph. The higher one is at 24 hours, which is the required NCLS time for virgin blends used to make AASHTO M294 pipe. The lower line is 18 hours, which has been recommended for a test performed on the actual pipe liner (2).

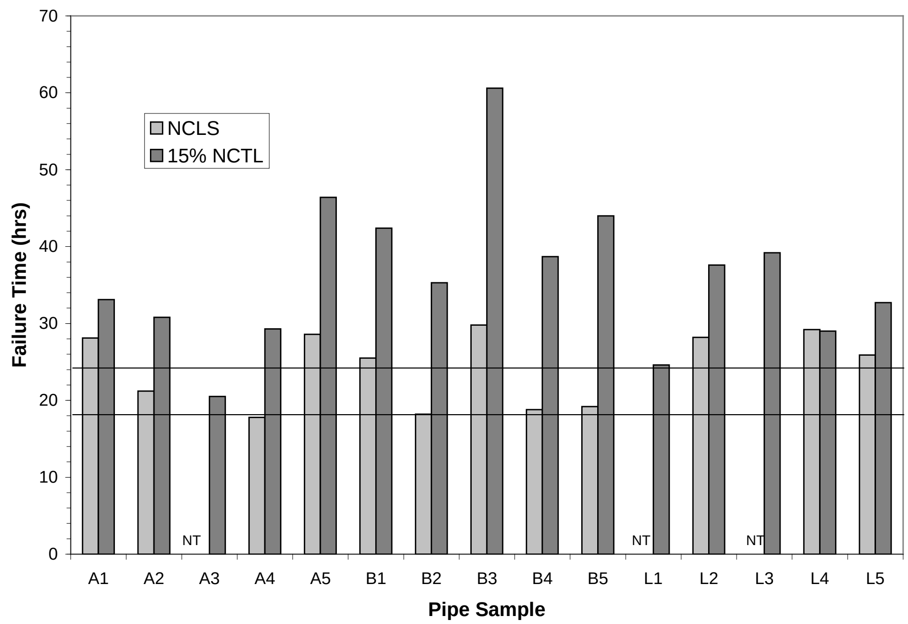

Figure D-11 - Stress Crack Resistance of 15 Pipe Samples

D.3.2 BFF Test

A test has been developed that combines some features from the BAM test with the criteria set in the Florida DOT 100 year durability assessment (3).

The FLDOT protocol calls for the evaluation of the junction between the corrugation and the liner with a stress crack test on a 0.25 in wide test specimen in deionized water at 80°C. Samples cut directly from pipe are evaluated under the conditions in Table D-6.

Table D-6 - FLDOT Junction Test Conditions

Temperature (°C)	Applied Stress (psi)
80	650
80	450
70	650

The results are then used in a rate process method (RPM) model to estimate the service lifetime at 23°C and an applied tensile stress of 500 psi. The specification allows one to terminate the tests if no failures occur under the conditions shown in Table D-6 after 110, 430, and 500 hrs, respectively.

The BAM stress crack test (4,5) is a useful tool for the evaluation of the effects of residual stress or defects on the stress crack resistance of HDPE. However, there are some disadvantages to the test including:

- The activity of the Igepal solution changes over time.
- Special grips are required and cracks initiate at the grips about 25% of the time.
- One cannot predict service lifetime from the results because the accelerating factor of the Igepal is not known.

A test has been developed that addresses these limitations. First, the solution has been changed from Igepal to deionized water. Second, a new test specimen has been developed that reduce grip failures.

Photographs of the junction specimen from the FLDOT standard and the new specimen (Fathead) for this study are shown in Figures D-12 and D-13.

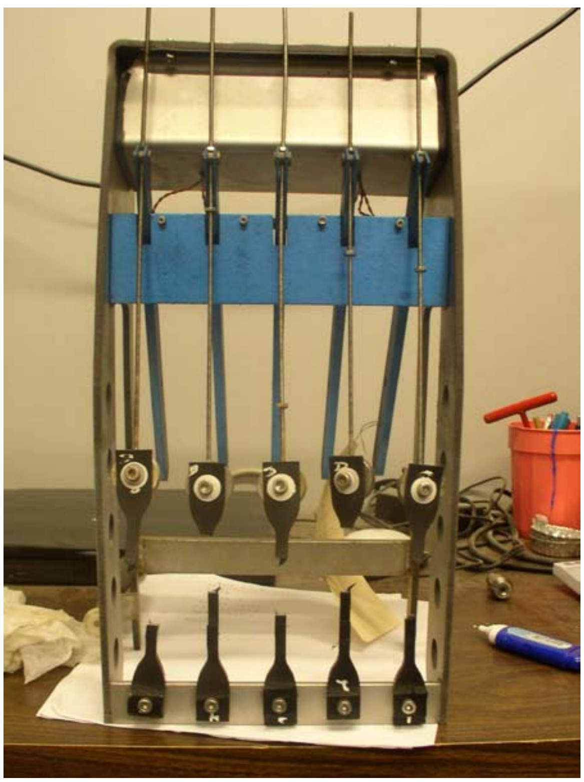

Figure D-12 - Junction Specimen

Figure D-13 - Fathead Specimen

The main difference in the specimens is the size. The junction specimen is a Type IV and the Fathead is a Type I, as defined by ASTM D638. The Type I has a surface area of 1.12 in^2 while the Type IV has a surface area of 0.32 in^2. Depending on the sample thicknesses, the Type I has 2.5 to 3.5 times the volume of the Type IV. This is important when one is looking for flaws or defects, like small silicone rubber particles. The new

specimen is called ‘Fathead” because the plaque mold was modified to make the heads more than two times the thickness of the reduced width section. This is shown in Figure D-14. The use of this test specimen has essentially eliminated grip failures.

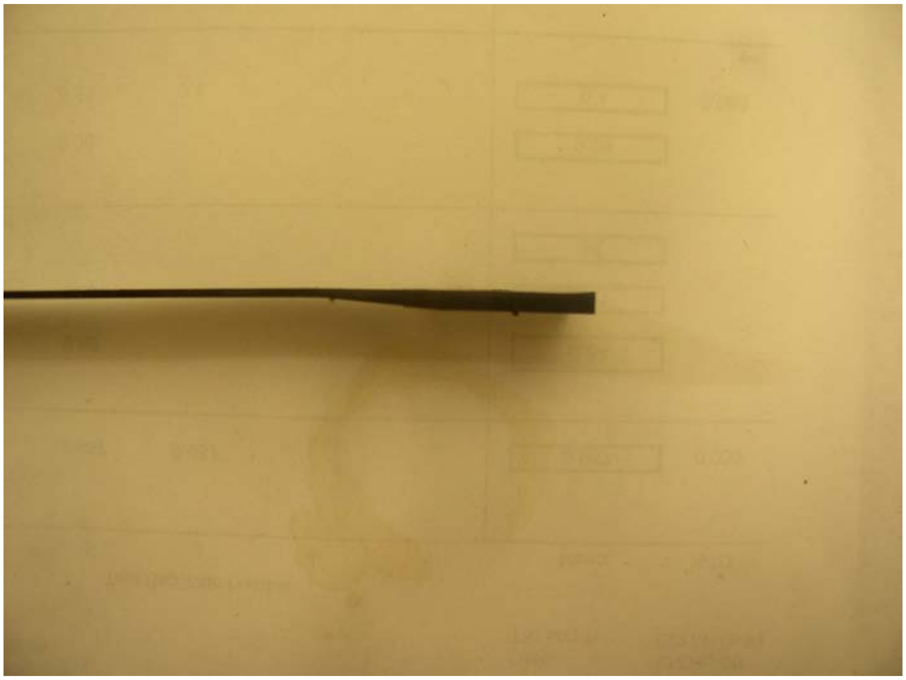

Figure D-14 - Side View of Fathead Specimen

Tests have been performed on compression molded plaques from each of the 15 trial pipes. Preliminary results are presented in Table D-7.

Almost all of the samples had average values greater than 100 hrs. The 3 VR1 samples from each of the 3 plants have coefficients of variation of 14, 19 and 19%. The 3 samples of the 50% VR1 + 20% MD + 30% MCR had COV of 27, 40, and 28%. The three samples that only contained natural recycled HDPE had COV of 14, 4, and 21%. These results suggest that the highest variability is in the MCR recycled.

The failure surfaces were examined to determine how often test specimens broke at a silicone rubber particle. There were actually 5 categories used to characterize the fracture face. They were:

1. Rubber Particle – a clearly present soft particle.
2. Not Obvious – no visible sign of a crack initiator.
3. Gel – a classic unmelt; harder and darker than a rubber particle.
4. Imperfection – an ambiguous flaw or void.
5. Tiny particle – too small to tell if it’s hard or soft.

Table D-7 - BFF Test Results on 15 Pipe Samples at 80°C/650 psi in D.I. Water

Sample	Formulation	Failure Time (hrs) (COV)
A1	100% VR1	175 ± 31 (18%)
A2	85% VR1 + 15% MCR	157 ± 45 (29%)
A3	85% VR1 + 15% NAT	123 ± 17 (14%)
A4	50% VR1 + 20% MD + 30% MCR	130 ± 42 (33%)
A5	40% VR1 + 30% MD + 30% NAT	245 ± 11 (4%)
B1	100% VR1	188 ± 44 (23%)
B2	50% VR1 + 20% MD + 30% MCR	169 ± 68 (40%)
B3	20% VR1 + 40% MD + 24% MCR + 16% NAT	149 ± 43 (29%)
B4	50% MD + 50% NAT	108 ± 23 (21%)
B5	40% MD + 36% MCR + 24% NAT	145 ± 29 (20%)
L1	Solplast Pipe Resin	56 ± 9 (16%)
L2	50% VR2 + 20% MD + 30% MCR	150 ± 35 (23%)
L3	100% VR2	147 ± 81 (55%)
L4	100% VR1	181 ± 42 (33%)
L5	50% VR1 + 20% MD + 30% MCR	190 ± 65 (34%)

Examination of fracture faces from the tests reported in Table D-7 produced the following breakdown:

Rubber Particle	48%
Not Obvious	28%
Gel	5%
Imperfection	13%
Tiny Particle	6%

The fracture surfaces for the virgin resins were not included in this analysis. This shows that nearly half the time a rubber particle is the stress crack initiation site.

However, examination of the failure times did not suggest that the failures from the rubber particles had shorter failure times than the 'not obvious' failures. This was looked at 3 different ways. First, within a set of 5 specimens, the cause of the lowest failure time and the highest failure time were recorded. Next, the cause of failure in the 10 shortest individual failures times was noted. And finally, the cause of failure in the 10 longest individual failures times was looked at. The results of these analyses are shown in Table D-8.

Table D-8 - Frequency of Failure Types

Category	1st Break in a Set of 5	5th Break in a Set of 5	10 Shortest Failure Times	10 Longest Failure Times
Rubber part.	4	7	2	4
Not obvious	3	6	3	4
Gel	3	0	2	0
Imperfection	3	1	2	1
Tiny particle	2	1	1	1

These results seem to suggest that all the different failure categories are involved in the shortest failure times and that every rubber particle does not necessarily cause an early failure. The fracture surfaces will continue to be monitored to see if any definite patterns emerge.

D.3.3 BFF Test Reproducibility

It became clear early on that the BFF test results were influenced by residual surfactants in the exposure bath. However, it was also learned that once a bath has been used with surfactants, they are difficult to remove, even after multiple rinsings. The results of repeated BFF tests over time are shown in Table D-9. The time period represented was about 18 months. It is clear from the table that baths that have never contained surfactants are preferred for the BFF test.

Table D-9 – Effect of Residual Surfactant on BFF Failure Times

Sample	Time-to-Failure (Hrs) (80ºC/650 psi)			
	September 2008	October 2008	October 2009	March 2010
A1 VR1	175 ± 31 (18%)	226 ± 46 (20%)		
B1 VR1	188 ± 44 (23%)	215 ± 97 (45%)	411 ± 135 (33%)	414 ± 97 (23%)
L4 VR1	181 ± 42 (23%)	211 ± 46 (22%)		
A4 VR1+MD+MCR 50/20/30	130 ± 42 (33%)	148 ± 73 (50%)		
B2 VR1+MD+MCR 50/20/30	169 ± 68 (40%)	234 ± 52 (22%)		
L5 VR1+MD+MCR 50/20/30	190 ± 65 (34%)	255 ± 54 (21%)	431 ± 86 (20%)	334 ± 117 (35%)
A2 VR1+MCR 85/15	157 ± 56 (36%)		257 ± 85 (33%)	
A5 VR1+MD+NAT 40/30/30	253 ± 14 (6%)		323 ± 70 (22%)	
B3 VR1+MD+MCR+NAT 20/40/24/16	166 ± 36 (22%)		330 ± 66 (20%)	
B5 MD+MCR+NAT 40/36/24	145 ± 35 (24%)		280 ± 95 (34%)	228 ± 141 (62%)
L1 Proprietary	56 ± 9 (16%)		201 ± 39 (19%)	208 ± 73 (36%)

D.4 COMPARE RESIN PROPERTIES TO PIPE PROPERTIES

Samples of the dry blended resins were obtained at two of the plants. In one case, the blends were prepared separately and pumped into boxes, which were in turn pumped to the extruder feed hopper. The samples were taken from the boxes. In the other case, the blends were made by pumping out of the component boxes, into a mixing hopper, then into the feed hopper. The samples were taken from the feed hopper. Most of the resin samples obtained were not tested because their evaluation was not part of the original statement of work. Additionally, it is strongly believed that sampling from the feed hopper takes such a small sample that it may not be representative of the pipe, after its

extruded and mixed some more. The only way to get a reasonable representative batch of a blended resin would be to obtain one directly from a blending silo. Or, one might consider testing the pipe. The main problem with testing the pipe is making sure the sample is cleaned so that particulates are not introduced into the plaque. Comparisons for four key properties on six different resin blends are shown in Figure D-15 through D-18.

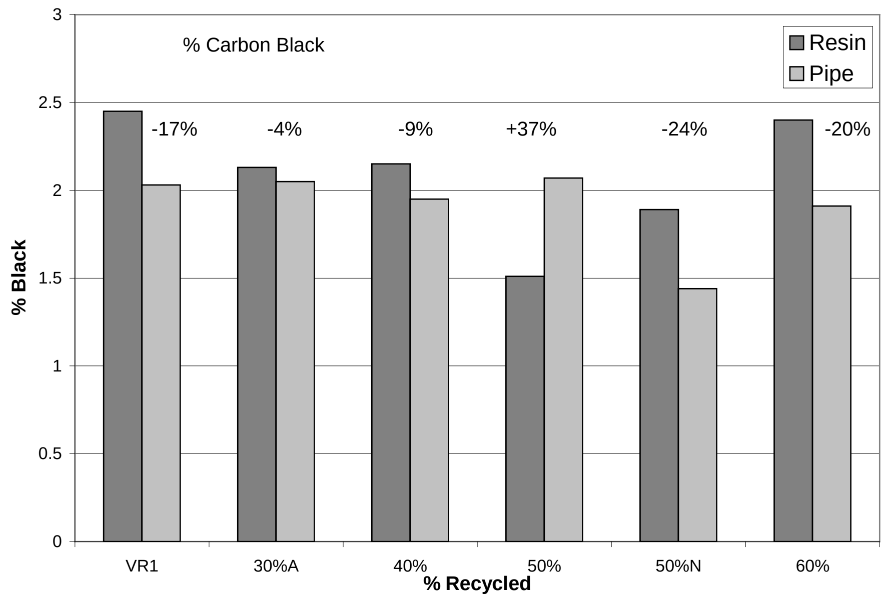

Figure D-15 - Density Comparison Between the Resin and the Pipe for Six Formulations

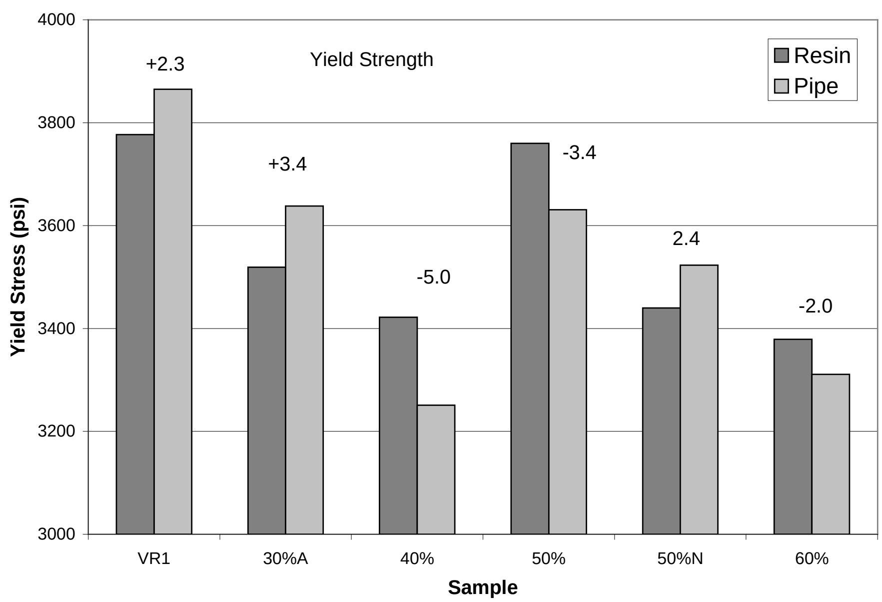

Figure D-16 - Yield Strength Comparison Between the Resin and the Pipe for Six Formulations

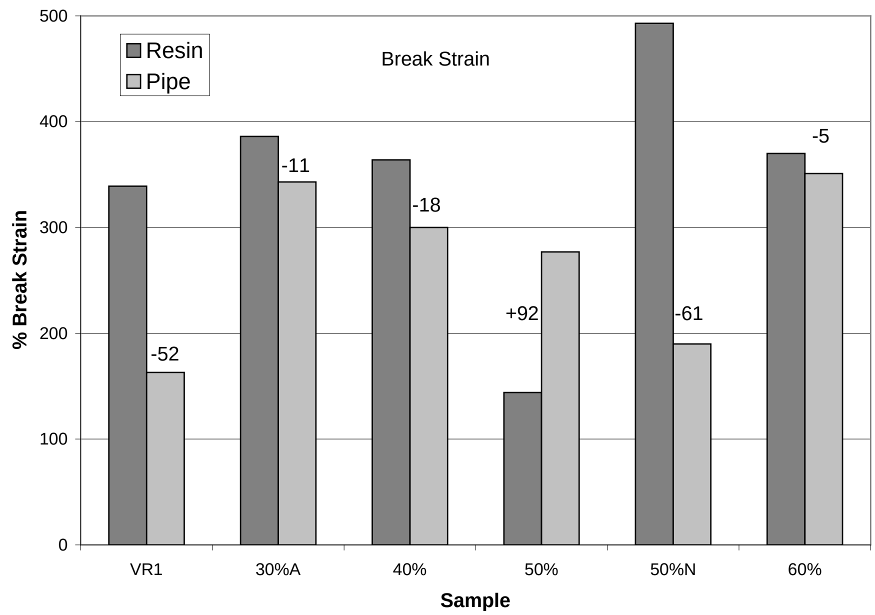

Figure D-17 - Break Strain Comparison Between the Resin and the Pipe for Six Formulations

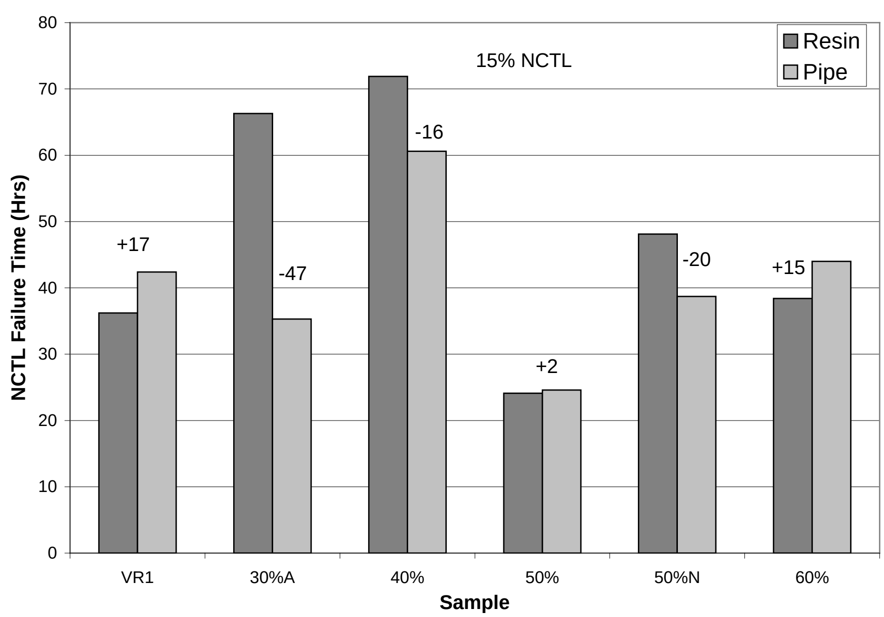

Figure D-18 - 15% NCTL Comparison Between the Resin and the Pipe for Six Formulations

There were large differences between the resin and pipe for each property measured, except for the yield strength. The most likely cause for these differences is that the resin

samples were so small (2-3 lbs), that they did not reflect the entire blend very well. This is not too surprising because the dry blends were not blended particularly well. The pipe extrusion process helps to homogenize the blends, so the pipe should be more representative of the blend than a grab sample from the feed hopper.

There was an opportunity to look at the properties of the mixed color PCR that was supplied for the project. Each of the three plants obtained recycled resin from the same lot that had been dry blended in a mixing silo. Each plant received at least one box (about 1400 lbs). Samples (2-3 lbs) were obtained from each plant and their properties measured. Table D-10 shows the respective results for six key properties.

Table D-10 - Properties from Three Samples of a Lot of Mixed Color PCR Resin

Sample	Density (g/cm^3)	MI (g/10 min)	% Color + % Ash	Yield Stress (psi)	% Break Strain	15% NCTL (Hrs)
Plant 1	0.959	0.47	1.28	3757	90 ± 41	6.1
Plant 2	0.958	0.50	1.21	3711	45 ± 16	5.9
Plant 3	0.961	0.50	1.23	3978	65 ± 13	5.1
Avg.	0.959	0.49	1.24	3816	66.7	5.7
SD	0.0015	0.017	0.035	143	22.5	0.53
COV	0.16 %	3.5 %	2.9 %	3.7 %	33.8 %	9.3 %

It is clear from these data that a lot blended in a mixing silo has much low variability, except for the breaking strain. However, one must look at the large variability in the breaking strain results. The coefficients of variability for the breaking strains for the individual sets of results were 45%, 36%, and 20% respectively. Additionally, the high standard deviation for the combined sets of data is essentially the average of the standard deviations of the three individual results. These results really show how sensitive the breaking strain is to contaminants.

D.5 COMPARE FORMULATIONS MADE AT ALL THREE PLANTS

Each manufacturing plant made pipe from 100% Virgin Resin 1 (VR1) and a blend of 50% Virgin Resin 1, 20% of Virgin MDPE (MD), and 30% of Post-Consumer Mixed Color HDPE Resin (PCR-MCR). Each plant had a different lot of the VR1, and the same lots of MD and PCR-MCR. All the properties that were measured are shown in Figures D-19 through D-28.

It was anticipated at the time that measuring the properties of common formulations would yield information about plant-to-plant variability. However, these results indicate that it is not so simple to make comparisons. First of all, it is not known how much variability there is in the virgin resins, and especially, the recycled resins. Secondly, there is variability in the plaque manufacturing process and the individual tests' variability. If the only variable was the plant, one would expect to see consistent differences between properties.

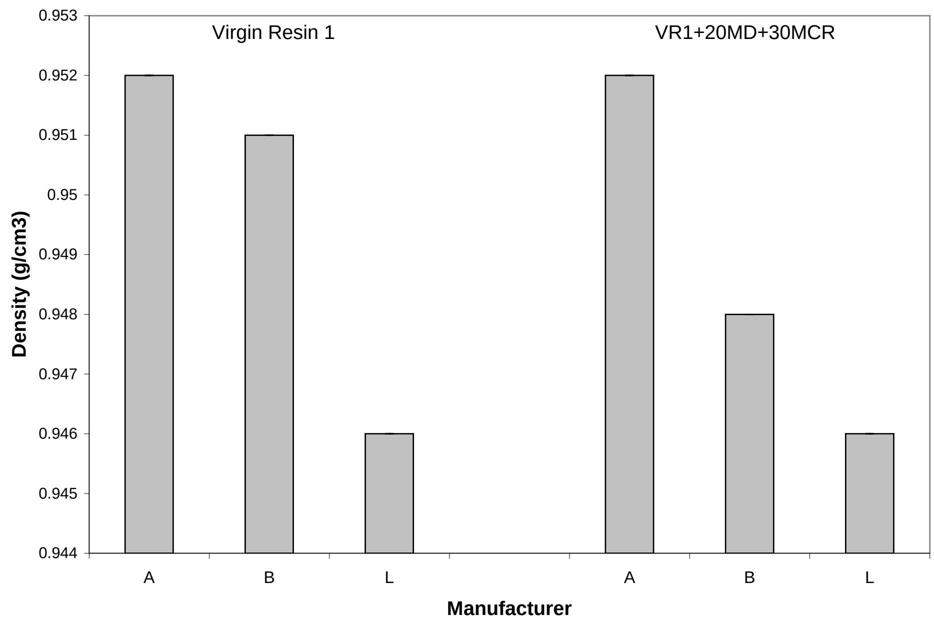

Figure D-19 - Corrected Density of Samples Made at All Three Plants

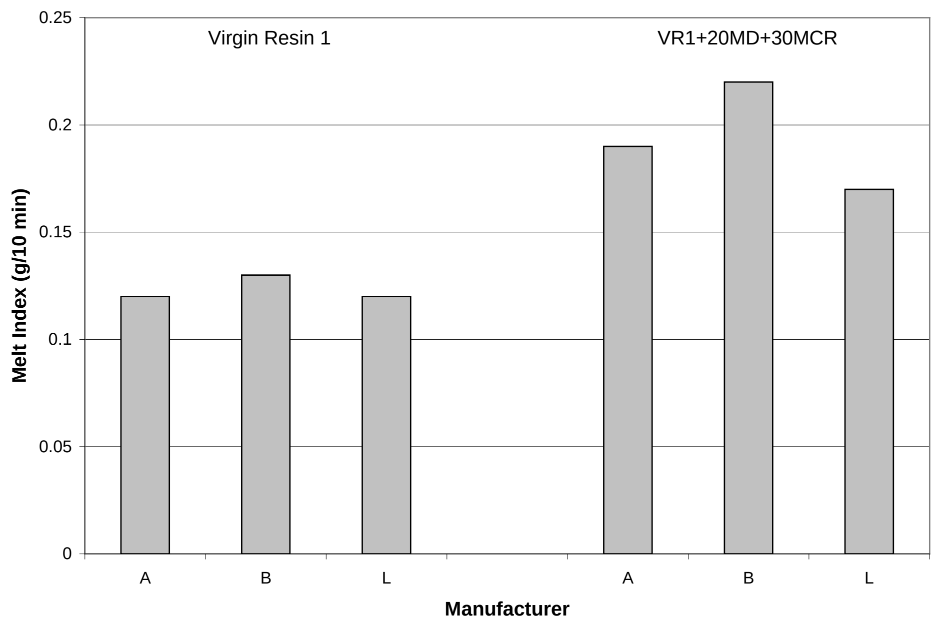

Figure D-20 - Melt Index of Samples Made at All Three Plants

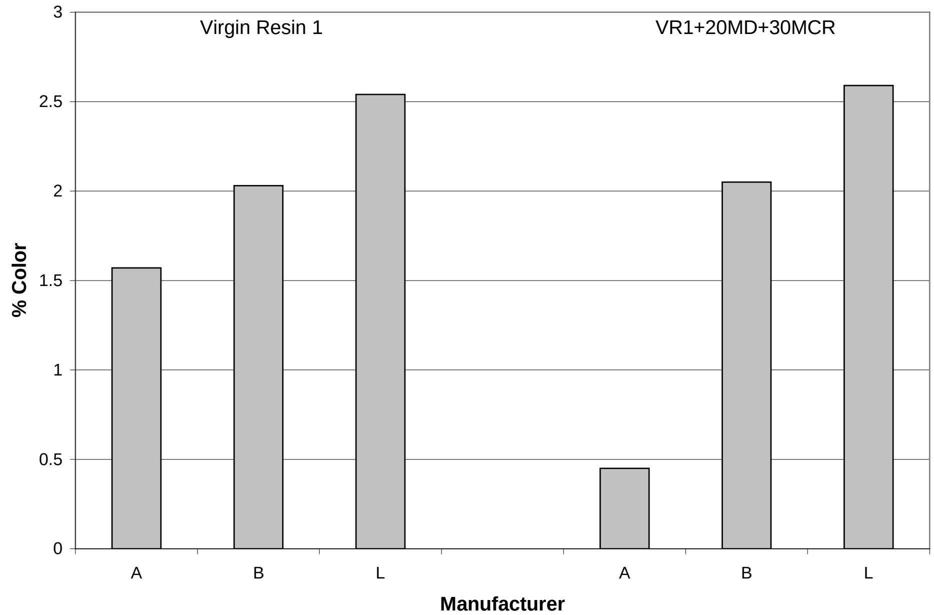

Figure D-21 - Percentage Color of Samples Made at All Three Plants

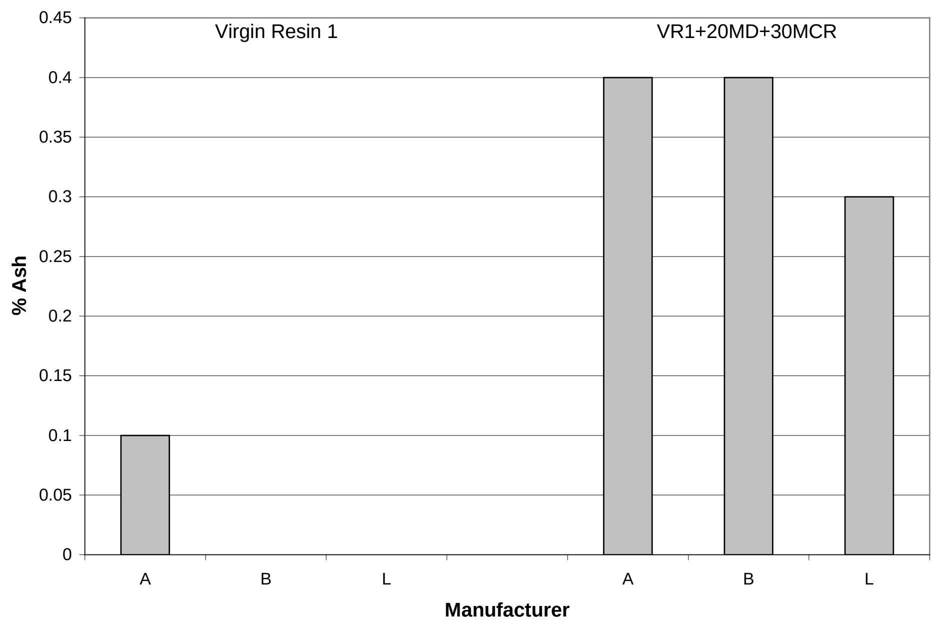

Figure D-22 - Percentage Ash of Samples Made at All Three Plants

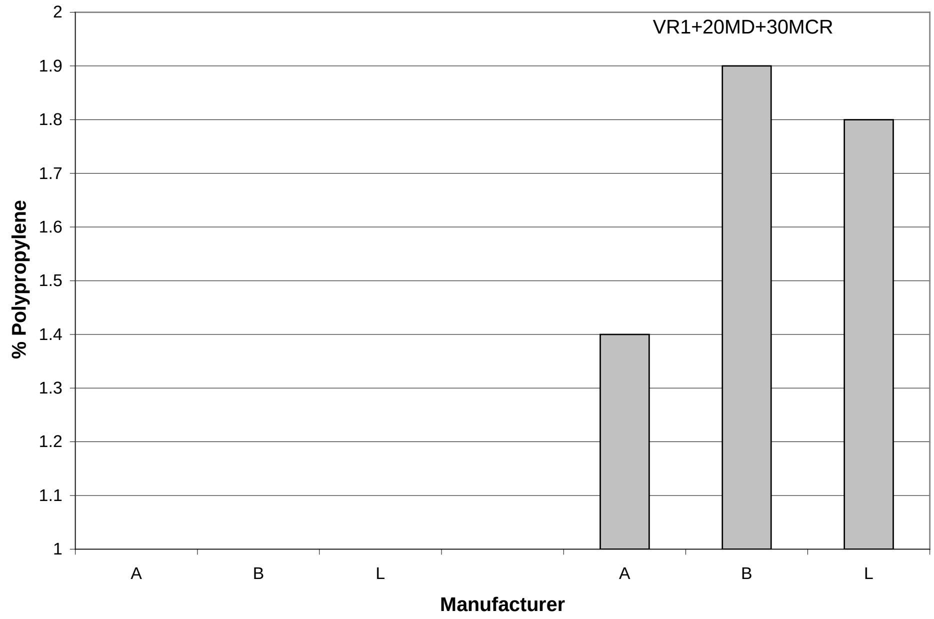

Figure D-23 - Percentage Polypropylene of Samples Made at All Three Plants

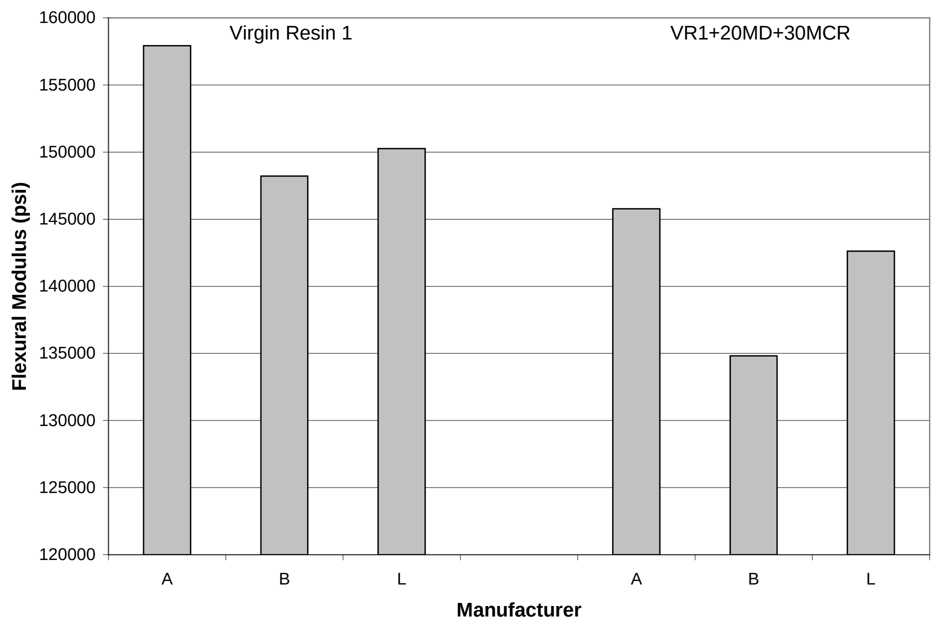

Figure D-24 - Flexural Modulus of Samples Made at All Three Plants

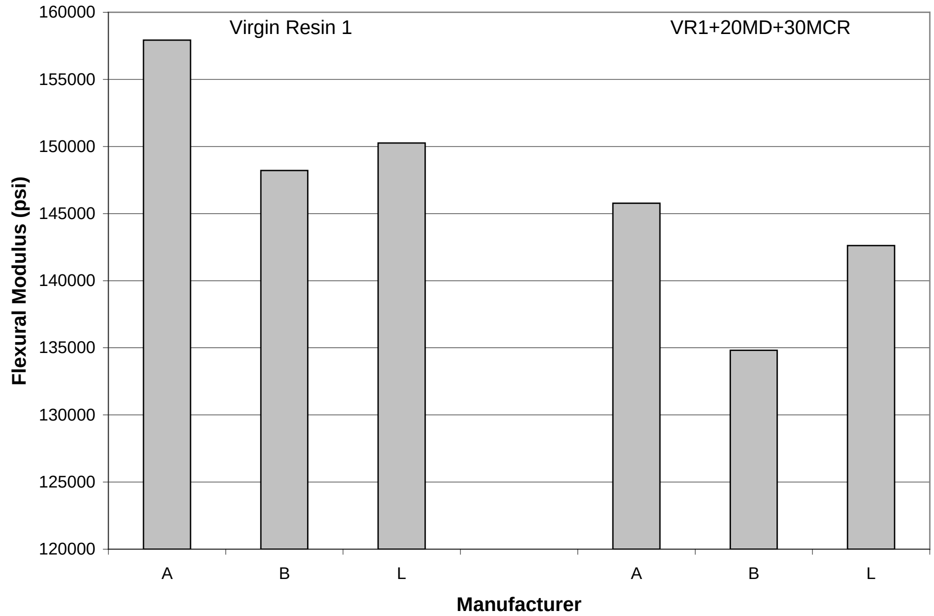

Figure D-25 - Yield Stress of Samples Made at All Three Plants

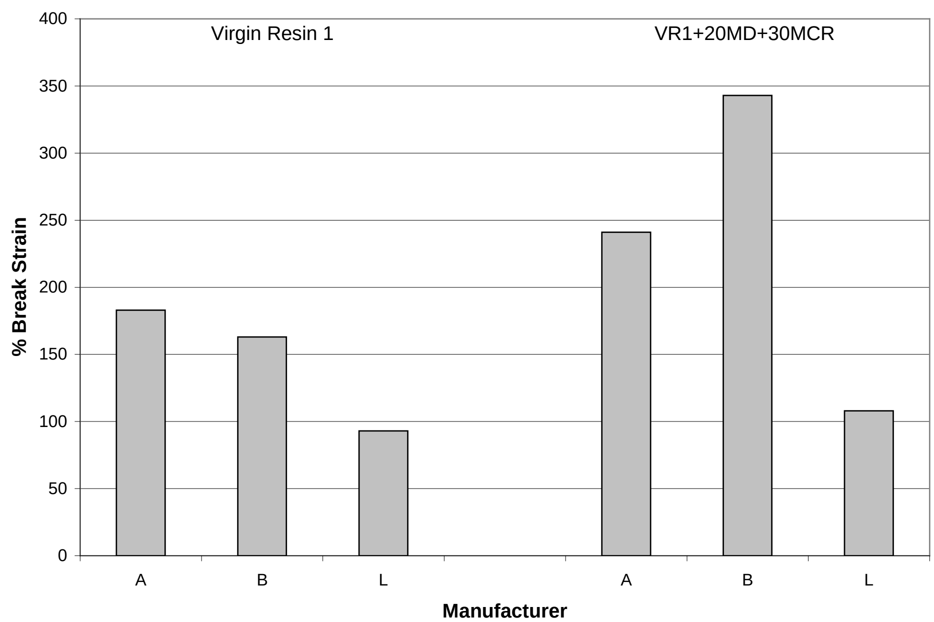

Figure D-26 - Break Strain of Samples Made at All Three Plants

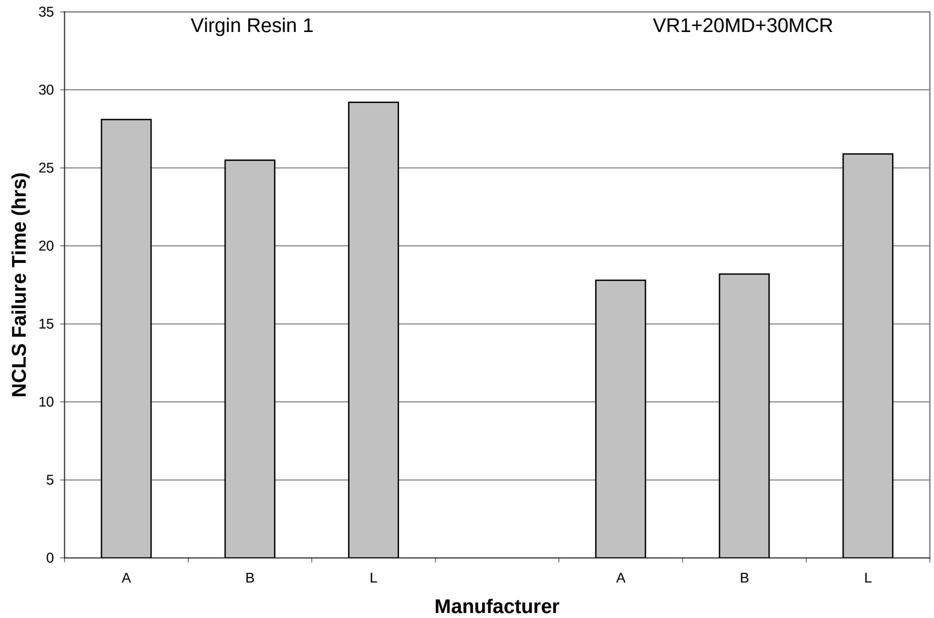

Figure D-27 - NCLS Failure Time of Samples Made at All Three Plants

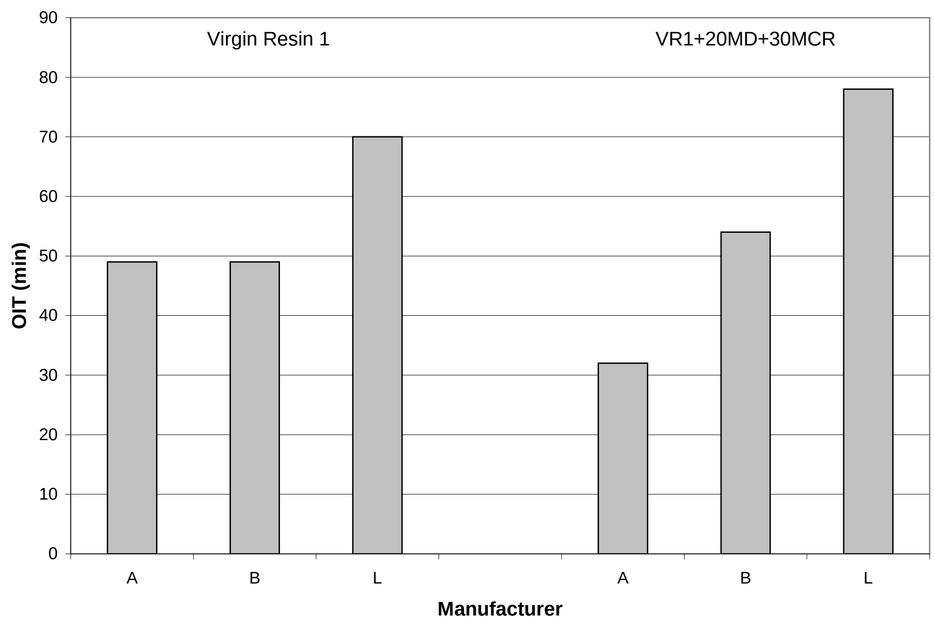

Figure D-28 - OIT of Samples Made at All Three Plants

For example, the order seen in density should also be observed in the tensile yield stress and the flexural modulus since they are both dependent on the density. This was not observed although there seemed to be mostly consistency between samples. The plant with the highest density in the virgin sample had the highest density in the blend. However, this could be biased by the fact the each pair of samples were tested on different days and perhaps by different operators.

Although it is not possible to know all the causes, there were some general observations:

1. The density results showed that the samples from Plant L were lower than expected. However, it is not know if the difference is in the lab manufactured plaque, the actual density measurement, or the % carbon, since they were corrected for carbon content.
2. The melt index values showed very good agreement on the virgin resin. This should be expected since the melt index is a control property for the resin supplier. This should also indicate that the plaques were made in a consistent manner, even on different days. The differences seen in the blend could be caused by the proportion of the ingredients, or the carrier resin for the carbon concentrate. Typically, the carrier resins are of higher melt index than the matrix resin to improve the mixing of the carbon particles. Each manufacturer used their own carbon concentrate.
3. The percentage color does show plant-to-plant variability. It is clear that for this trial, Plant A had less control over the carbon content than the other two plants. It should be mentioned that Plant A ran the same formulation of virgin resin, color concentrate, and in-house reclaim day after day and adding 1 or 2 additional components to a blend was more of a stretch for them than the other two plants, which had more experience with multiple component blends.
4. The percentage ash was fairly consistent except that Plant A had a small amount (0.1%) in the virgin pipe. This could have come from the carbon concentrate. The % ash for the blends were consistent around the theoretical value of 0.36%.
5. The percentage polypropylene showed some variability in the blends, but it cannot be known if this was caused by the relative percentages in the blends, or the variability of polypropylene content in the recycled resin.
6. The flexural modulus values showed consistency between manufacturing plants and about the same coefficient of variability (3.4% and 4.0%) for the virgin resins and blends.
7. The tensile yield stress results showed similar behavior to the flexural modulus. There was a decrease in the blends and the COV were similar for the virgin and blend samples (4.2% and 3.9%). The COV values for flexural modulus and yield stress indicate very good agreement between the manufacturing plants.
8. The break strain values showed an increase in the blends due to the MD resin, but also showed that the samples from Plant L were lower than the other two. Surprisingly, the pipe samples had lower break strain values than was expected by tests on the resin blends tested in house. One possible cause is surface dirt on the pipe that made its way into the compression molded plaque.
9. The NCLS stress crack test results were surprising in two ways. First, the addition of carbon black has a larger effect than was anticipated. The virgin resin without carbon black averages around 45-50 hours. Secondly, the blend did not perform as well as it was expected too, based on blend results generated during the blending study. The blending study suggested that this blend would show an increased stress crack resistance, compare to the virgin resin. This clearly was not the case for this particular PCR-MCR resin.

10. The OIT values were not consistent. A twenty minute difference in the virgin resins seems high. And, in two cases, the blends were higher than the virgin and it the third it was much lower. Its difficult to draw any conclusions here since only a single replicate was tested.

D.6 PIPE DEFLECTION TESTS

This was the only test performed on the pipes themselves. This was a decision made by the technical oversight committee to keep the focus on the pipe formulations and not the design of specific pipes. However, these tests were performed to aid in the last task of the program, which involves an evaluation of current design methodologies for pipe containing recycled. The pipe stiffness at 5 % deflection, the peak load and the peak percentage deflection are shown in Table D-11.

Table D-11 - Recycled Pipe Stiffness Results

Pipe Sample	Formulation	Pipe Stiffness At 5% (psi)	Peak Load (lbs)	Deflection at Peak Load (%)
A1	100% VR1	54.8	>1281	>40
A2	85% VR1 + 15% MCR	54.5	>1293	>40
A3	85% VR1 + 15% NAT	56.1	>1304	>40
A4	50% VR1 + 20% MD + 30% MCR	52.1	>1271	>40
A5	40% VR1 + 30% MD + 30% NAT	54.3	>1277	>40
B1	100% VR1	62.0	1053	15.7
B2	50% VR1 + 20% MD + 30% MCR	58.3	1043	18.3
B3	20VR1+40MD+24MCR+16NAT	54.0	836	16.7
B4	50% VR1 + 50% NAT	49.2	860	15.6
B5	40% MD + 36% MCR + 24% NAT	53.9	1024	21.3
L1	Proprietary Resin 50% MCR	47.0	1088	28.1
L2	50% VR2 + 20% MD + 30% MCR	45.1	1015	25.7
L3	100% VR2	50.9	1238	34.0
L4	100% VR1	56.2	1301	31.0
L5	50% VR1 + 20% MD + 30% MCR	53.5	1260	34.5

Most would agree that this property is controlled by wall thickness, so any manufacturer can meet the requirement by adding more plastic to the wall. This is typically done by slowing down the process. In this case, the manufacturers were not asked to meet the specification, but to instead keep the pipe weight as constant as they could.

The results reflect consistency at each plant. The fact that there are three sets of pipe with different deflection characteristics is very beneficial if one were to consider a follow-on field study. AASHTO M294 requires a pipe stiffness of 50 psi and buckling at strains greater than 20%. This set of pipes has 3 samples under 50 psi stiffness and 4 samples under 20% for buckling. These requirements could be evaluated in a field setting with the use of these pipes.

D.7 SELECTION OF SIX CANDIDATE FORMULATIONS

The purpose of this task was to reduce the number of formulations to six, so that more extensive testing could be performed on them. The final six candidate formulations were selected based mainly on the % recycled and the recycled type (colored or natural). The formulations selected were:

B1 – 100% VR1
A2 – 85% VR1 + 15% MCR
L5 – 50% VR1 + 20% MD + 30% MCR
A5 – 40% VR1 + 30% MD + 30% NAT
B3 – 20% VR1 + 40% MD + 24% MCR + 16% NAT
B5 – 40% MD + 36% MCR + 24% NAT

These formulations vary from 0% to 60% recycled, and there is one formulation with only natural recycled. Some of their properties are given in Table D-12. The properties were measured on compression molded plaques from the pipes.

Table D-12 - Select Index Properties of Final Six Candidate Formulations

Property	B1	A2	L5	A5	B3	B5
Density[1]	0.951	**0.943**	**0.946**	0.948	0.948	0.952
Melt Index	0.13	0.15	0.17	0.21	0.27	0.34
% Color	2.0	1.4	2.6	1.5	2.0	1.9
% Ash	0.0	0.3	0.3	0.2	0.3	0.4
% PP	0.0	0.7	1.8	0.3	1.7	2.2
Flexural	148,210	152,607	142,618	140,065	128,361	128,758
Yield	3865	3775	3922	3623	3251	3311
Break	165	168	108	159	300	351
NCLS	25.5	**21.2**	25.9	28.6	29.8	**19.2**
OIT	49	39	78	96	93	75
BFF Test[2]	188	157	190	253	166	145

1. Corrected for % color
2. At 80ºC and 650 psi of stress.

This group of formulations is believed to be a good representation of the recycled pipe formulations that might be used. Notice that A2, L5, and B5 have properties outside of the current M294 requirements for virgin, uncompounded resins.

D.8 LONG-TERM PROPERTIES OF SIX FORMULATIONS

D.8.1 Introduction and Background

The long-term properties of HDPE are often presented in a graph like the one in Figure D-29.

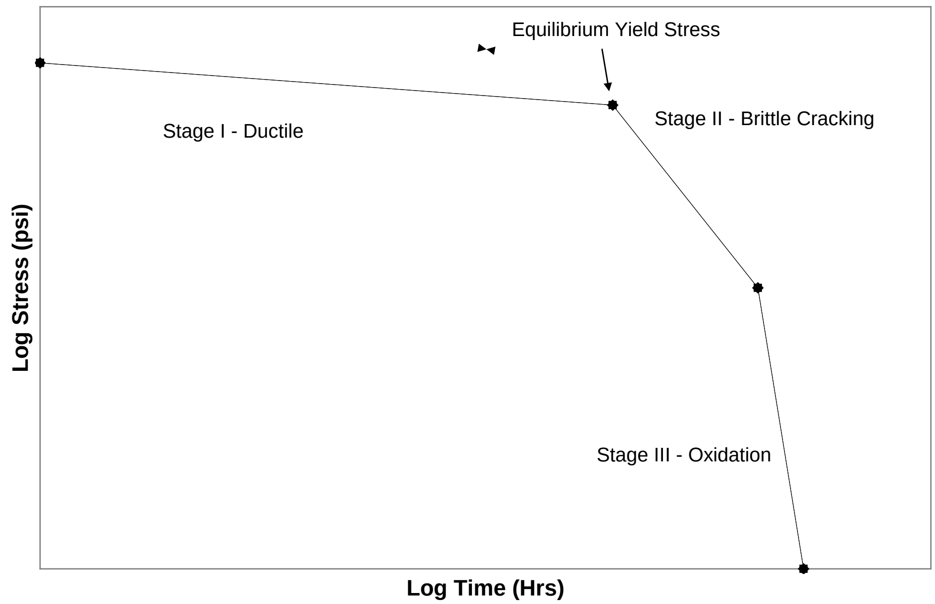

Figure D-29 - Hypothetical Service Lifetime for HDPE

It shows three distinct stages of aging. The first phase is the likely service lifetime for materials placed under significant loads. Over time, because of the time dependent process of creep, the material will fail by yielding or stretching in a ductile manner. The second phase is at intermediate loads and involves failures by slow crack growth (stress cracking). And finally, under low stresses the material will fail only after the additive package has been consumed and the HDPE undergoes oxidation.

The first stage is largely dependant on the materials yield strength, the service temperature, and the stress. Long-term tests for this property usually involve placing the material under a load less than its strength and waiting for failure to occur. This is called stress rupture, if the sample is placed under a constant strain or creep rupture, if the sample is placed under a constant stress. Temperature is sometime used to accelerate the process and the results analyzed through a method called time-temperature superposition (TTS), which assumes that higher temperatures and shorter times can be related to lower temperatures and longer times. A specialized form of TTS is called the stepped isothermal method (SIM) and has been used successfully on polyester, polypropylene, and polyethylene reinforcement products for civil engineering applications. The end of this stage is characterized by the equilibrium tensile strength. Any service stresses lower than the equilibrium yield stress will not cause a ductile failure.

The second phase involves brittle cracking through slow crack growth. During this stage, a defect in the material can initiate a craze, which can, in turn, become a running crack eventually causing a break in the material. For a brittle crack to grow there has to be a significantly sized and shaped defect, and sufficient load. HDPE materials have an inherent stress crack resistance that can be measured, but manufacturing defects and flaws can accelerate cracking. For PE pressure pipe, the best way to determine service

lifetime is the long-term hydrostatic strength test. This entire technology has been developed for pressure pipe and the results are relied upon for the design of pressure piping systems. ASTM D2837 describes a method called hydrostatic design basis for evaluating the service lifetime of pressure pipe. This involves high loads (ductile failure) at room temperature to determine the long-term hydrostatic strength (LTHS) and intermediate loads (slow crack growth) at elevated temperatures to validate that slow crack growth will not occur in 100,000 hrs. Similar tests for corrugated pipe have been presented, including the BAM Test (4,5), the FLDOT Junction Test (6), the Ring Stress Crack Test (7), and the BFF Test.

The final stage occurs at service stresses below the stress that would cause a stress crack during the service lifetime. In this case, the entire part becomes brittle through chemical oxidation and fails by many cracks starting at the same time. This region is controlled by the additive package which contains the long-term antioxidants and/or light stabilizers. Oxidation has not been observed during this study and it is largely a non-issue if stabilized virgin pipe resins are used. However, there should be some requirement for an OIT value or a specified minimum additive package placed in the final specification. An OIT of 25 minutes should suffice and a recommended additive package containing 1000 ppm Irganox 1010 and 500 ppm Irgaphos 168 would meet the criteria.

The PE pressure pipe industry has used this failure envelope for many years to ensure the quality of resins used for gas and water distribution lines. The main protocol for this is ASTM D2837, “Standard Test Method for Obtaining Hydrostatic Design Basis for Thermoplastic Pipe materials or Pressure Design Basis for Thermoplastic Pipe Products”.

For medium and high density PE pipe, the method requires that at least 18 data points are generated at room temperature with one point over 10,000 hrs (1.14 yrs). These points are then plotted as Log Stress vs. Log Time and the resulting line extrapolated to 100,000 hrs (11.4 yrs). The100,000 hrs stress is called the Long Term Hydrostatic Stress (LTHS). The LTHS value is then fitted within a range of values given in the standard to define the Hydrostatic Design Basis (HDB). An example is shown in Figure F-30.

This shows a good example of a situation where only Stage I failures are observed during the duration of the test. The ASTM standard also accounts for Stage II or Brittle Failure through slow crack growth. This is shown in Figure F-31 and one can see that the failure mechanism has transitioned from Stage I to Stage II at the stresses evaluated.

It’s important to understand that the first example will undergo Stage II, it’s just that it occurs at a time over 100,000 hrs. Figure D-32 shows Stage I and Stage II for both the HDB 800 and HDB 1600 resins.

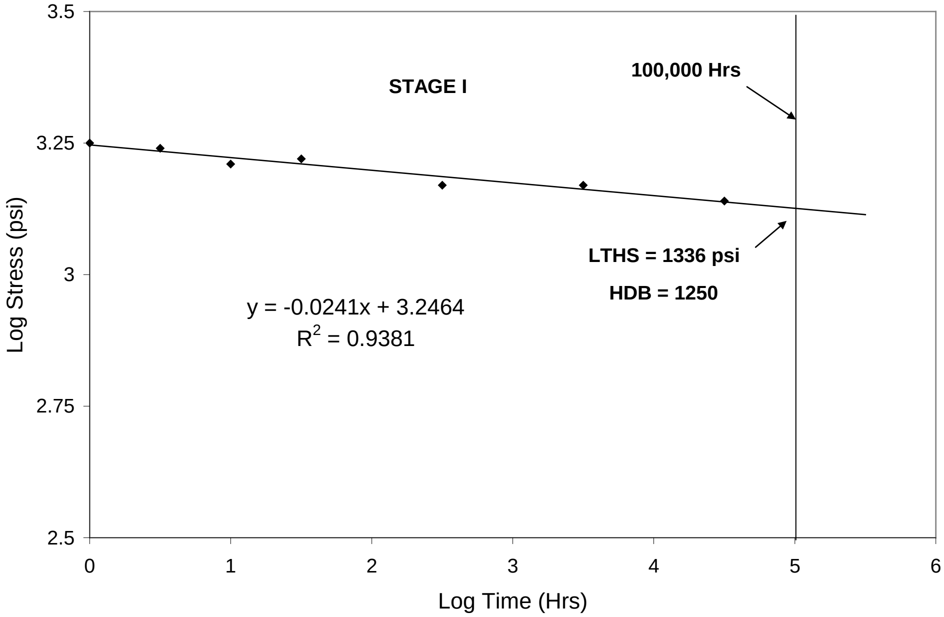

Figure D-30 - Determination of the Hydrostatic Design Basis (HDB)

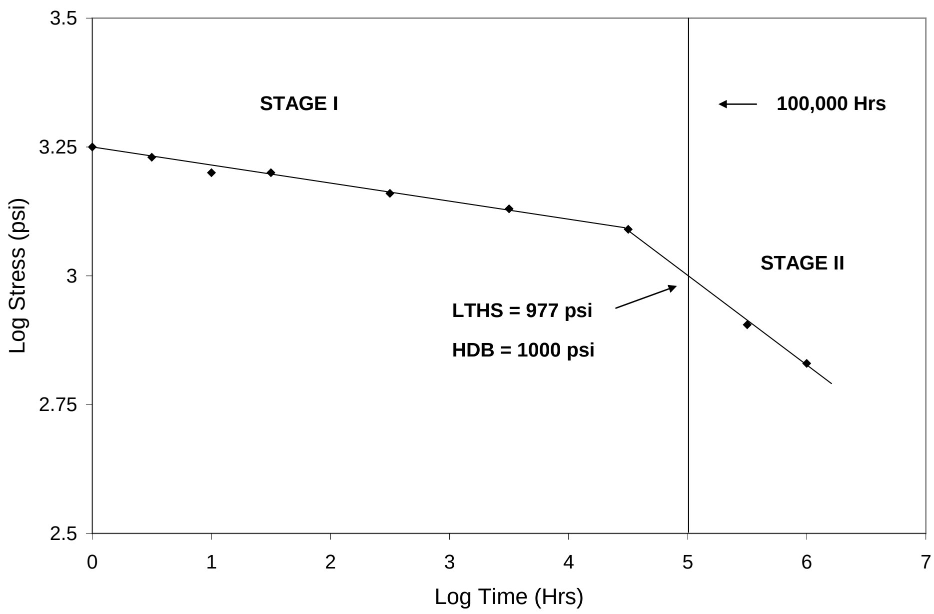

Figure D-31 - Determination of the HDB when Stage II is Involved

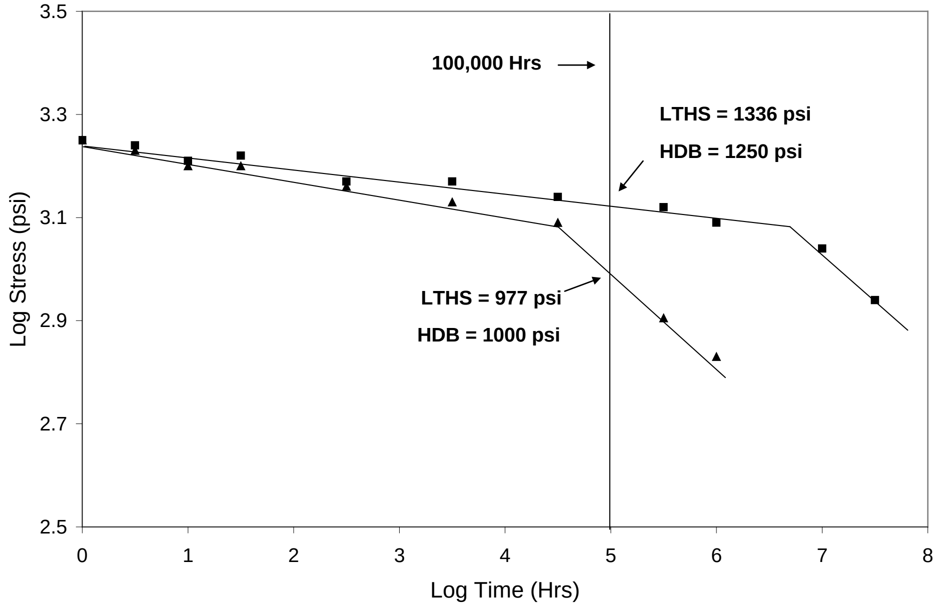

Figure D-32 - A Comparison Between the Examples
Shown in Figures D-30 and D-31

These examples clearly show that the differences seen between resins is that the slope of the ductile line is shallower and therefore, the transition from ductile to brittle (Stage I – Stage II) occurs farther out in time.

Once the HDB category is found, the Hydrostatic Design Stress (HDS) is found by applying a design factor (DF) to the HDB. The design factor is similar to a factor of safety, where reductions are estimated for installation damage, lot-to-lot variability, etc. In the pressure pipe industry a DF of 0.50 (or 0.62) is applied to water pipe and a DF of 0.32 is applied to gas pipe. So, a 1250 HDB resin would have a HDS of 625 psi and 400 psi in water and gas applications.

One of the advantages of hydrostatic testing on plastic pipe is one can generate both Stage I and Stage II failures with the same basic test. Additionally, a resin and carbon black formulation is certified through the room temperature testing. Then, as long as the formulation stays the same, there is no additional testing. In the present case, where we want to evaluate pipe resins containing recycled materials, every lot may be different, so accelerated tests must be used to estimate the long-term performance of the materials. Moreover, there will have to be two accelerated tests performed, one for Stage I (ductile) and another for Stage II (brittle).

D.8.2 Accelerated Test Methods

F.8.2.1 The Stepped Isothermal Method (SIM)

The Stepped Isothermal Method (SIM) is a special form of Time-Temperature-Superpositioning (TTS) that has been used to extrapolate short-term creep results (~24 hrs) into long-term estimates of creep behavior (50, 100 years). It was originally developed in these laboratories on polyester (PET) geogrids used for reinforcement applications (8-9). The application of SIM to PET has been verified and validated by several other laboratories comparing the SIM results to conventional creep tests performed at room temperature (10). It has also been used by others on other PET fibers, Kevlar, and Polyethylene Naphthanate (PEN)(11).

It has been used in these laboratories to examine PP buried structures and most recently on HDPE resins used for corrugated drainage pipe. It has been validated for PP by comparing SIM results to conventional creep results. A plot of duplicate SIM results compared with two, 10,000 hrs conventional creep tests is shown in Figure D-33.

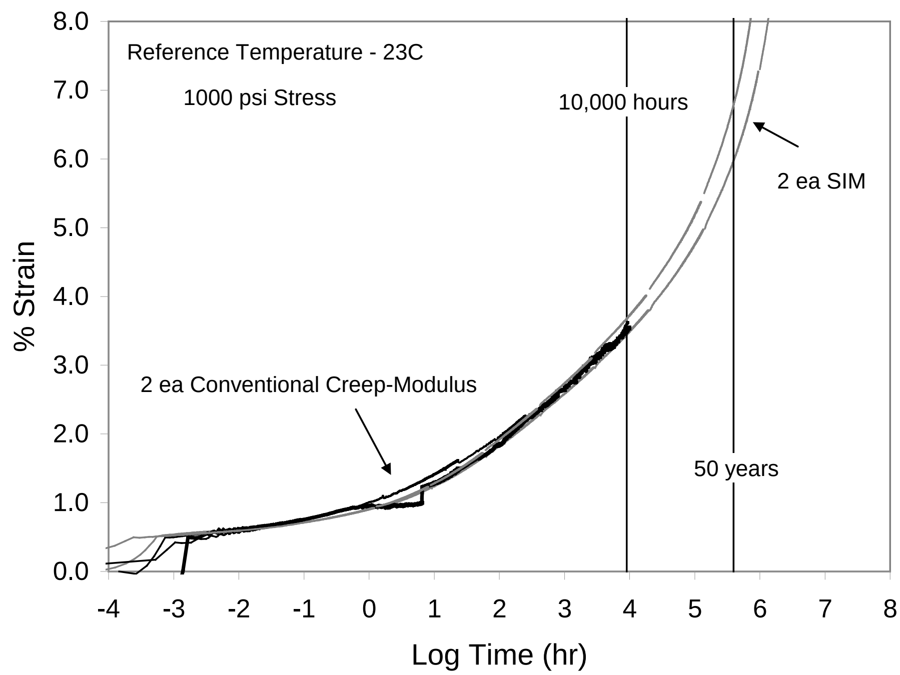

Figure D-33 - Comparison Between Conventional Creep and SIM
For a Polypropylene Storm Chamber under 1000 psi of Stress

The main difference between PET and HDPE is their respective temperature dependencies at temperatures below 80ºC. HDPE's properties change at a higher rate with temperature than PET's properties. In fact, the low temperature dependency of PET strength was the main reason SIM was developed in the first place. The sample-to-sample

variability could be as large and the difference in creep rates at two different temperatures. A comparison for the two materials is shown in Figure D-34 below.

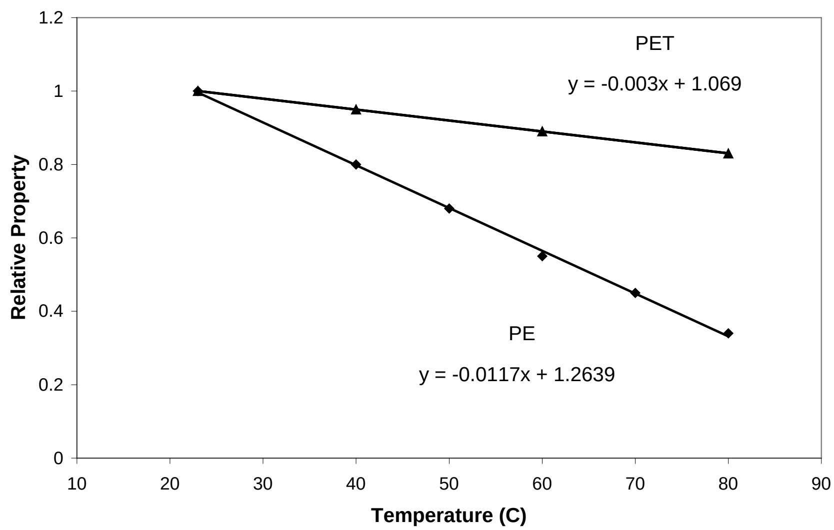

Figure D-34 - Temperature Dependence of PET and PE

Time-temperature-superposition (TTS) has been used for decades and it is the basis for the validation procedures for polyethylene pipe materials in ASTM D 2837 and PPI Technical Report TR-3. TTS can used to project the long term hydrostatic strength of pressure pipe.

Basically, increasing the temperature of a process like creep, stress relaxation, or slow crack growth is equivalent to performing the test at longer times. The higher the temperature, the longer the accelerated time.

In the case of traditional TTS, tests are performed at various elevated temperatures on different samples and the results shifted to a lower target temperature. Because of the sample-to-sample variability, the result of TTS can be uncertain and requires tests on many test specimens. Two examples of TTS are the Rate Process Method and Popelar Bi-Directional Shifting.

SIM is a form of TTS where behavior at multiple temperatures is observed on a single test specimen, which reduces the uncertainty of the behavior due to sample-to-sample variability.

An example SIM test for HDPE was performed under the following conditions:

Sample: Type I Dumbbell.
Strain Measurement: Extensometer.
Initial Temperature: 20°C.

Temperature Steps: 7°C (20, 27, 34, 41, 48, 55, 62, 69, 76, 83)
Stress: 500 psi.
Dwell Time: 10,000 seconds (2.77 hrs).

The raw, unshifted data are shown in Figure D-35.

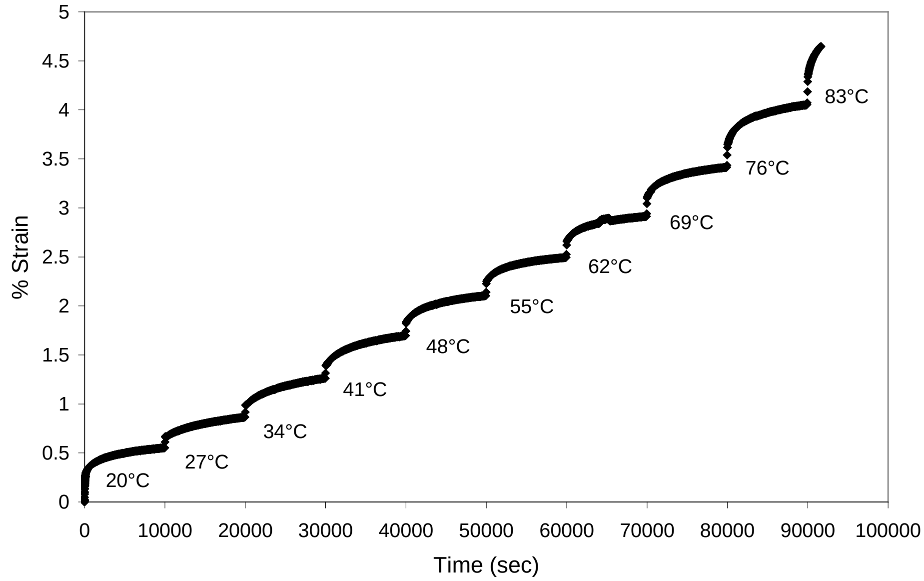

Figure D-35 - Raw SIM Data

There are 10 temperature steps shown on the plot. Notice that the sample yielded catastrophically during the early part of the 83°C step.

It should be mentioned here that the transition from one temperature to the next is an important variable in SIM testing. The time it takes for the specimen to equilibrate at the new temperature should be just a few minutes. Other things that occur during the transition time are thermal expansion or contraction of the specimen as well as re-equilibration of the grips and extensometer. TRI excludes the data from the transition region, but keeps the time scale in place. The transitions then show up as blank spots in any plot with time as the abscissa.

The next step in the analysis is to determine what we refer to as the virtual starting time (t') for each step above the first one (Figure D-36). This accounts for the effects of the creep that occurred at the lower temperature. This step is necessary because the specimen "remembers" what had occurred at the previous creep step. This also allows one to re-scale the individual creep curves and get them all on a common time scale.

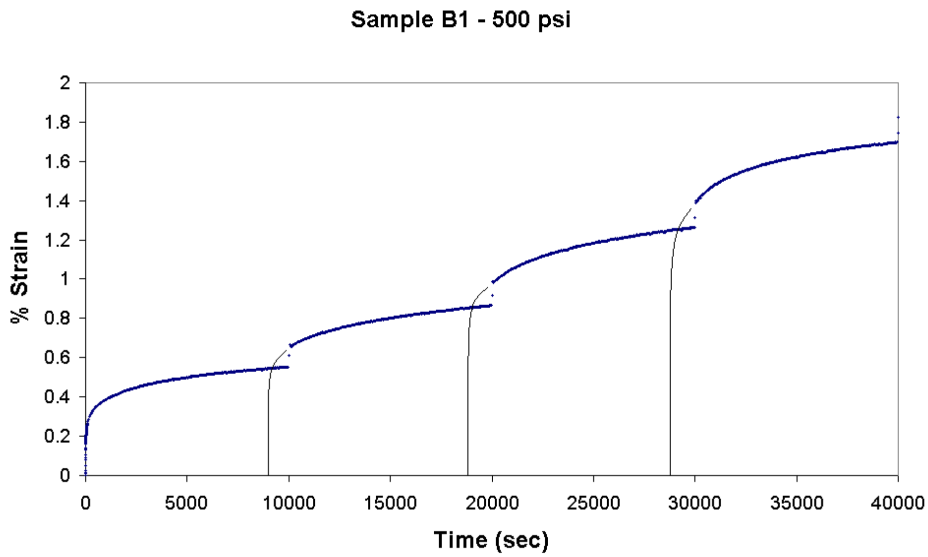

Figure D-36 - Virtual Starting Time (t')

The virtual starting time is found by plotting creep modulus vs. log time for the end of 1 step and the beginning of the next step. Then, one can adjust the virtual starting time iteratively until the slopes of the 2 steps are parallel. A vertical shift is also added at this time until the two parallel lines line up. This is illustrated in Figures D-37a through D-37d for the end of the 41°C step and the beginning or the 48°C step.

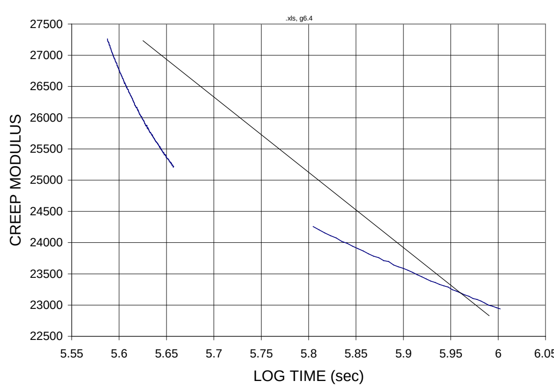

Figure D-37a - t' = 0 sec

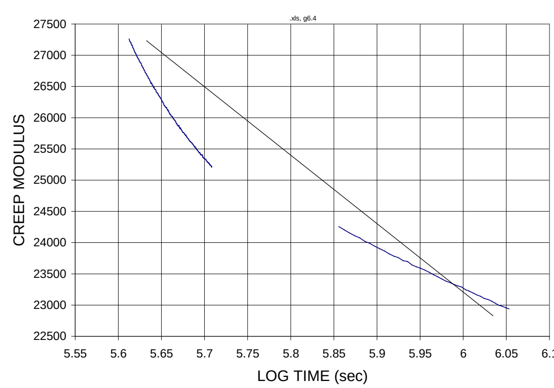

Figure D-37b - t' = 10,000 sec

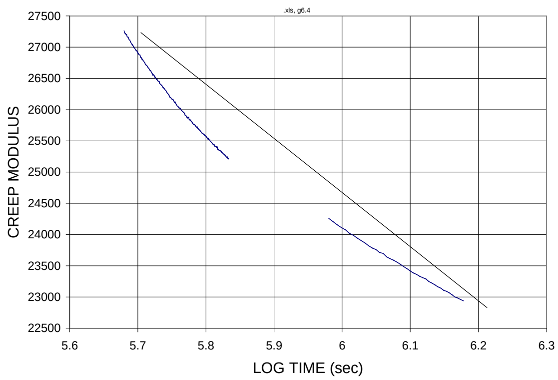

Figure D-37c - t’ = 20,000 sec

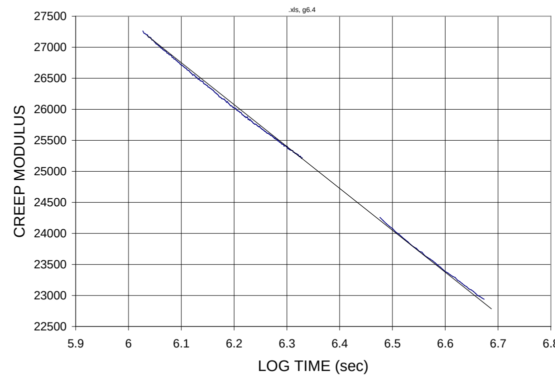

Figure D-37d - t’ = 28,100 sec

Figure D-37 - Curve Alignments at Different Virtual Starting Times

Once this is done for each step, master curves can be presented as either creep modulus or strain. Master curves for this data set are shown in Figures D-35 and D-36.

From these two curves, one can obtain both the 50 year creep modulus and 50 year creep strain. In this case, they are 23,250 psi and 2.15 % respectively. These represent the behavior of the material when placed under a 500 psi load for 50 years.

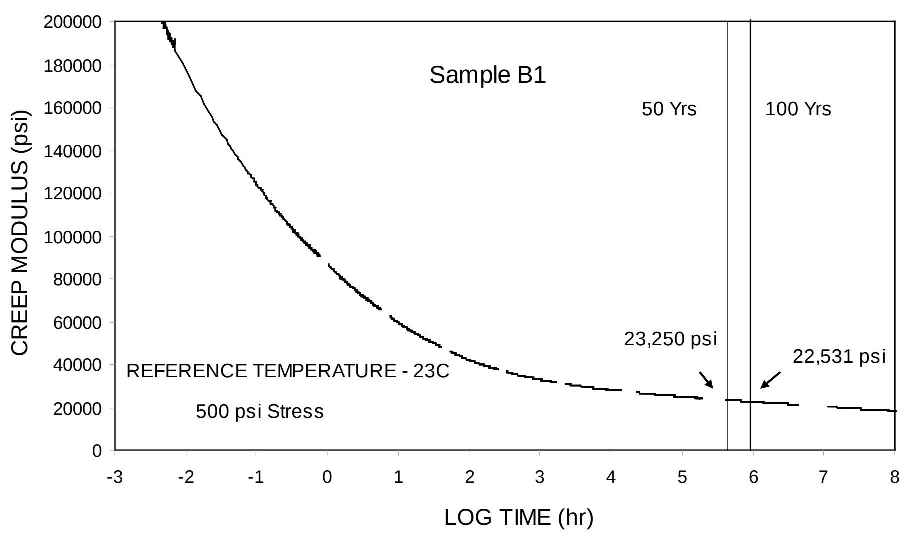

Figure D-38 - Creep Modulus Master Curve Under 500 psi of Stress

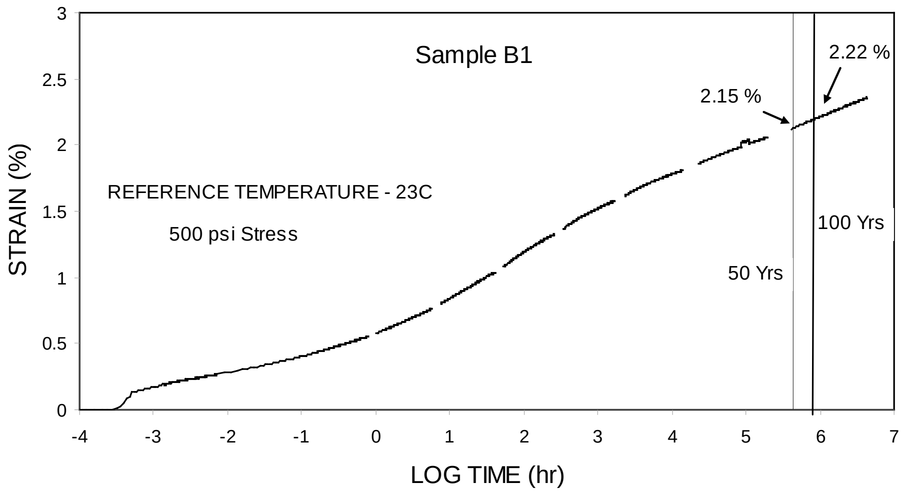

Figure D-39 - Creep Strain Master Curve Under 500 psi of Stress

Additionally, if one runs separate experiments at several loads, a master curve of stress vs. time can be obtained. Figures D-37 through D-39 show the results of SIM tests at 2000, 1500, and 1000 psi.

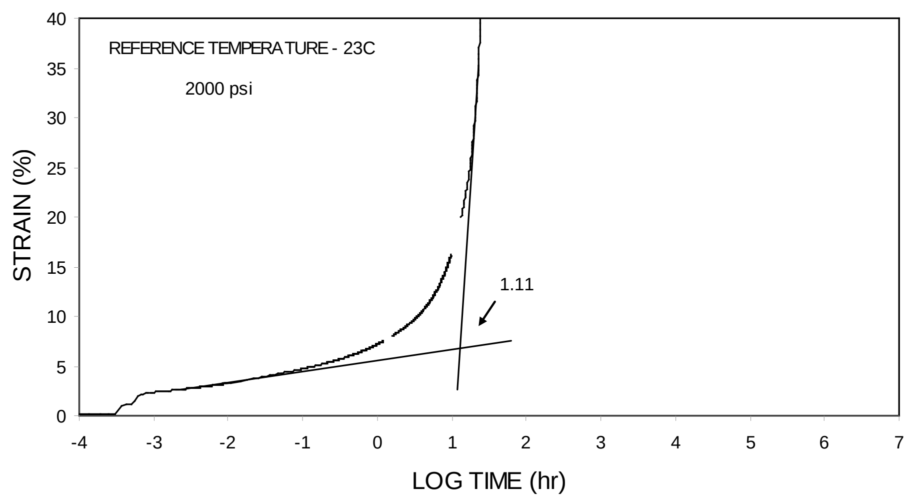

Figure D-40 - Long-Term Yield Stress by SIM at 2000 psi

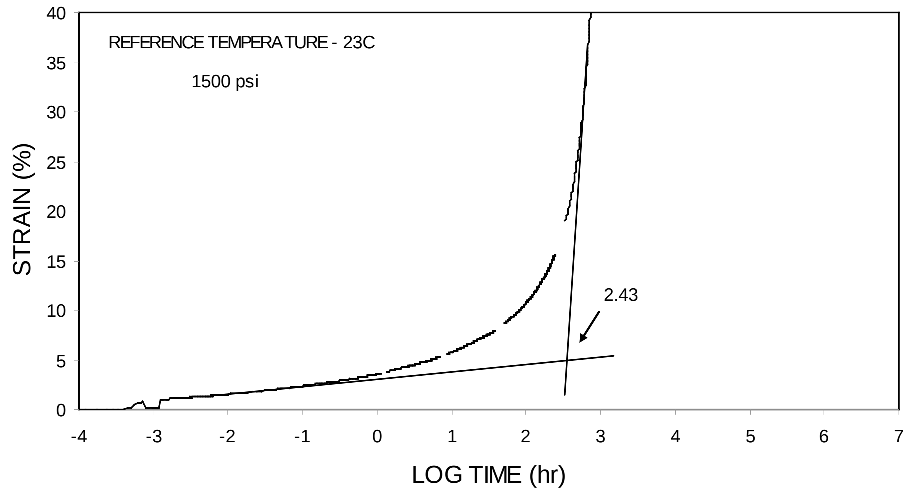

Figure D-41 - Long-Term Yield Stress by SIM at 1500 psi

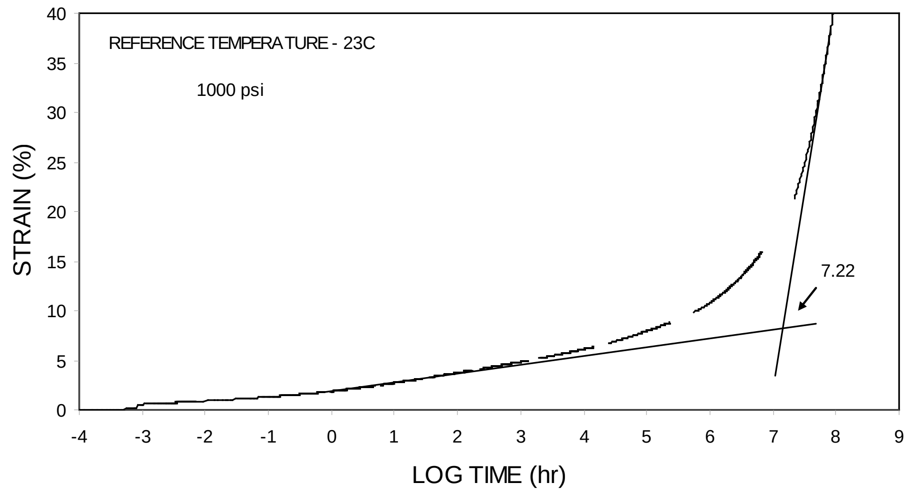

Figure D-42 - Long-Term Yield Stress by SIM at 1000 psi

One can then take the extrapolated creep rupture times, and plot Log Stress vs. Log Time, to generate a Creep Rupture master Curve. The one for the results above is shown in Figure D-43.

The results from these tests show that the 50 year strength of this material will be about 1161 psi. Recently, it has been discovered that applied stresses of 1500, 1250, and 1000 psi produce more consistent and linear long-term rupture plots.

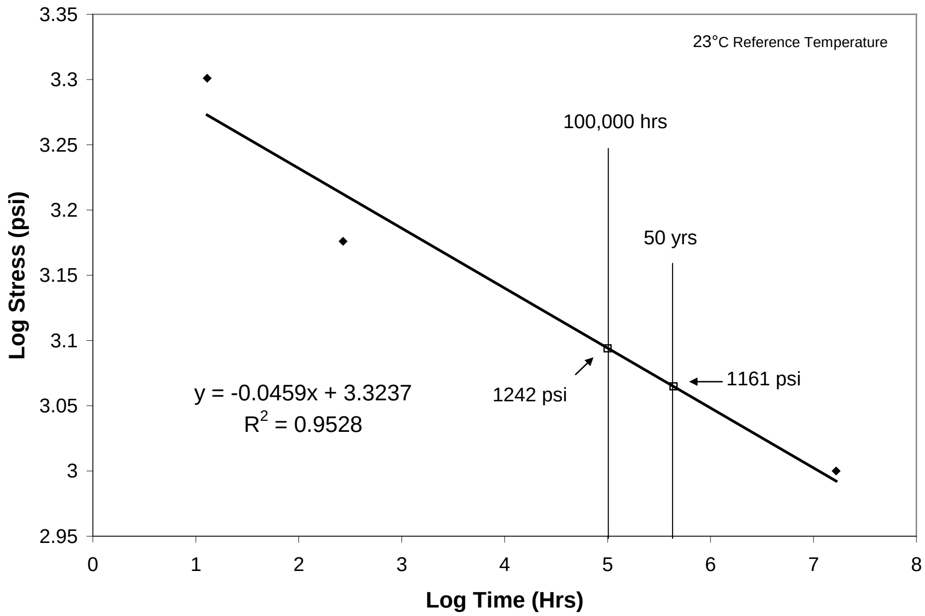

Figure D-43 - Creep Rupture Master Curve

D.8.2.2 The Rate Process Method (RPM)

The rate process method (RPM) is a well known method for translating data from one set of temperature and stress to another set of temperature and stress. It was introduced in 1980 (12) and has wide-spread acceptance as a valid time-temperature-superpositioning (TTS) method. It is used in ASTM D2837 to validate PE pipe performance and has recently been used for transforming stress crack growth data in corrugated plastic pipe applications. The FLDOT 100 year Service Lifetime Protocol relies heavily on the RPM for data analysis. Five replicates, under three sets of test conditions are required to perform a service lifetime prediction by the FLDOT standard. They are:

80ºC at 650 psi applied load in water,
80ºC at 450 psi applied load in water, and
70ºC at 650 psi applied load in water.

The RPM results can be used to determine the service lifetime under a given stress, or calculate the long-term stress for a given service lifetime.

The service lifetime is found by application of the rate process method (RPM). The basic equation is:

$$\log t = A + B/T + C \log S/T$$

where t = failure time (hrs)

T = temperature (°K)

S = stress (psi), and

A, B and C are constants.

Under the 3 sets of test conditions (or more), the values of the constants are found by solving three equations with three unknowns. The 3 equations are:

1. 80°C/650 psi $\log t_1 = A + B/353 + C \log 650/353$
2. 80°C/450 psi $\log t_2 = A + B/353 + C \log 450/353$
3. 70°C/650 psi $\log t_3 = A + B/343 + C \log 650/343$

Constant A can be found by subtracting equation 3 from equation 1:

$$353 \log t_1 – 343 \log t_3 = 353A – 343A = 10A$$

$$A = (353 \log t_1 – 343 \log t_3)/10$$

Constant C can be found by subtracting equation 2 from equation 1:

$$353 \log t_1 – 353 \log t_2 = C \log 650 – C \log 450$$

$$C = 353 (\log t_1 - \log t_2)/ 2.813 – 2.653$$

And, Constant B can be found by substituting the equations derived above into Equation 1.

$$\log t_1 = A + B/353 + C \log 650/353$$

$$353 \log t_1 = 353 A + B + C \log 650$$

$$B = 353 \log t_1 – 353 A – C \log 650$$

Example Problem: Given the following failure times, calculate the 3 constants.

80°C/650 psi = 110 ± 33 hrs = t_1	$\log t_1 = 2.041$	COV = 30%
80°C/450 psi = 430 ± 172 hrs = t_2	$\log t_2 = 2.633$	COV = 35%
70°C/650 psi = 500 ± 175 hrs = t_3	$\log t_3 = 2.699$	COV = 40%

Determine A

$$A = (353 \log t_1 – 343 \log t_3)/10$$

A = - 20.53

Determine C

C = 353 (log t_1 - log t_2)/ 2.813 – 2.653

C = - 1306.10

Determine B

B = 353 log t_1 – 353 A – C log 650

B = 720.47 + 7247.09 + 3674.06

B = 11,641.62

Now, with the use of these constants one can calculate the failure time, t, for any other set of temperature and stress. For example the service lifetime at 23°C and 500 psi would be found from:

log t = A + B/296 + (C log 650)/296

log t = -20.53 + 11,641.62/296 – (1306.10 log 500)/296

log t = -20.53 + 39.41 – 11.98

log t = 6.89

t = 7,762,471 hrs = 864 Years

This value represents the average lifetime based on the test results. However, since there can be significant variability in these test results, a lower confidence limit should also be considered.

The other way the RPM results can be used is to determine the stress that the material will have over a given period of time. Once the constants are known, one can also calculate the 50 year strength at 23°C from equation (1)

50 years = 438,000 hrs

log 438,000 = A + B/296 + (C log S)/296

5.641= -20.53 + 11,641.62/296 + (-1306.10 log S)/296

1669.74 = -6076.88 + 11,641.62 – 1306.10 log S

$2.98 = \log S, \quad S = 950$ psi

The rate process method predicted a service lifetime at 23°C and 500 psi of stress to be 864 years and also determined the 50 year strength would be 950 psi. This strength represents the stress under which stress cracks should not form for 50 years. These results can be presented graphically as seen in Figure D-44 below.

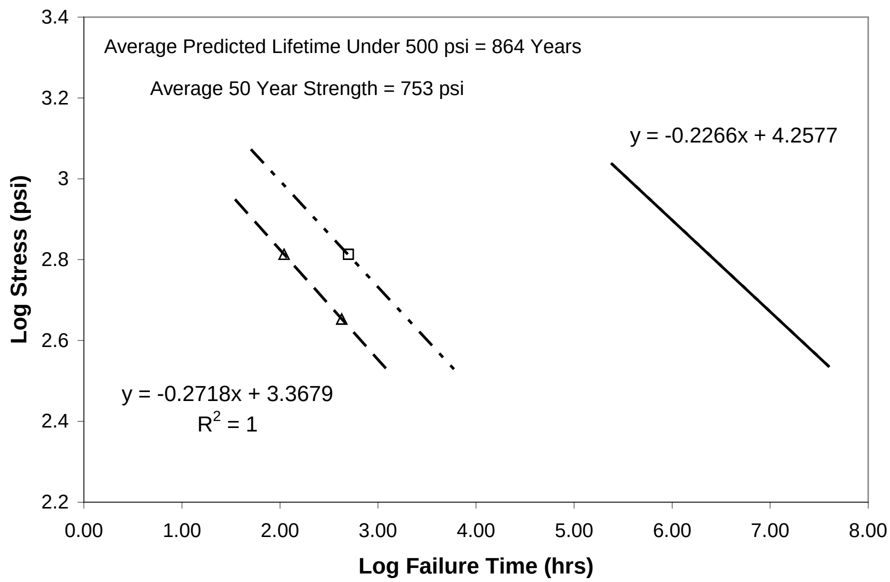

Figure D-44 - Lifetime Prediction

There are also equations that have been worked out to determine a lower confidence limit (LCL). These are found in ASTM D2837 and ISO 9080.

F.8.2.3 Popelar Bi-Directional Shifting (POP)

Another way to estimate the effects of stress and temperature at one set of conditions from results at a different set of conditions is with a technique called bi-directional shifting (13). This was first developed in 1990, has proven reliable through use on dozens of data sets, and produces results comparable to the Rate Process Method (RPM) (14). The advantage of bi-directional shifting is that one can shift data from a single set of conditions. It is not necessary to have data from multiple temperatures and stresses.

Bi-directional shifting involves the use of two constants, one for a horizontal shift (time) and another for a vertical shift (stress). Shifts are performed with the following two functions:

$a_T = exp[0.109\ (T - T_R)]$ for a horizontal shift and

$b_T = exp[-0.0116\ (T - T_R)]$ for a vertical shift.

An example of how these are used is shown in Figure D-45. The data were previously published by Hsuan (15), and generated by a notched stress crack test on HDPE geomembranes.

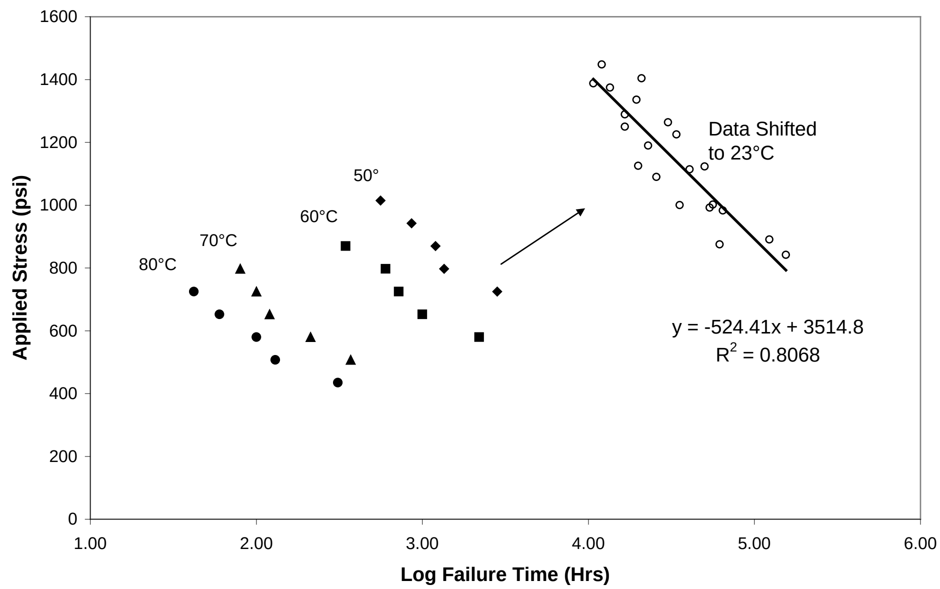

Figure D-45 - Bi-Directional Shifting

D.8.2.4 Compare RPM to POP

Two recent publications on long-term testing of corrugated drainage pipe can be used to evaluate both the rate process method (RPM) and the bi-directional shifting method (Popelar)(7,16). The first paper described a new pipe ring test and the results of this test were modeled by the RPM. Since the data were published, the results could be compared with the bi-directional shifting. The comparisons are shown in Figures D-46 and D-47 for the control pipe and pipe containing recycled, respectively.

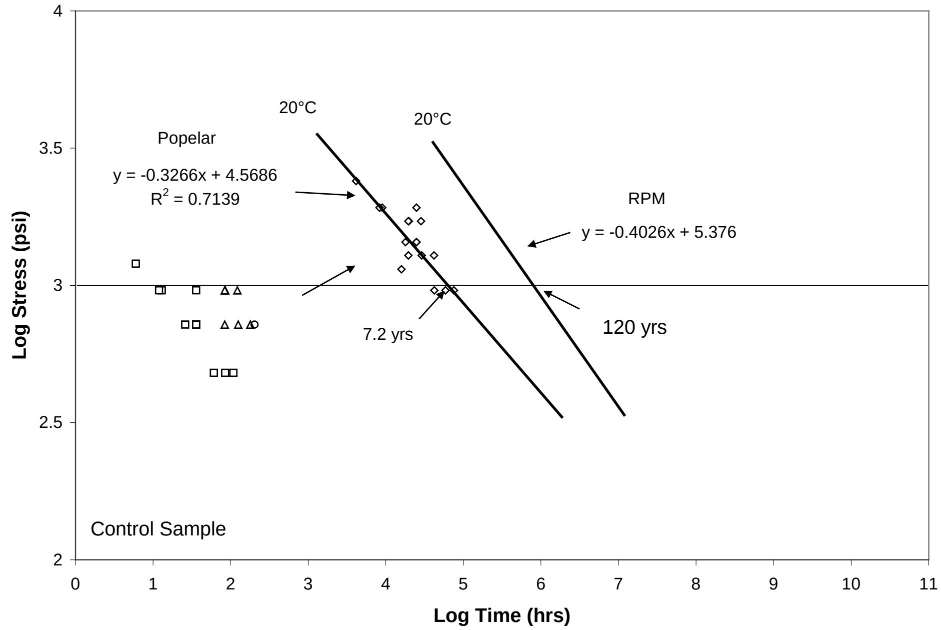

Figure D-46 - Comparison Between RPM and Popelar Projections on Control Sample from Reference 9

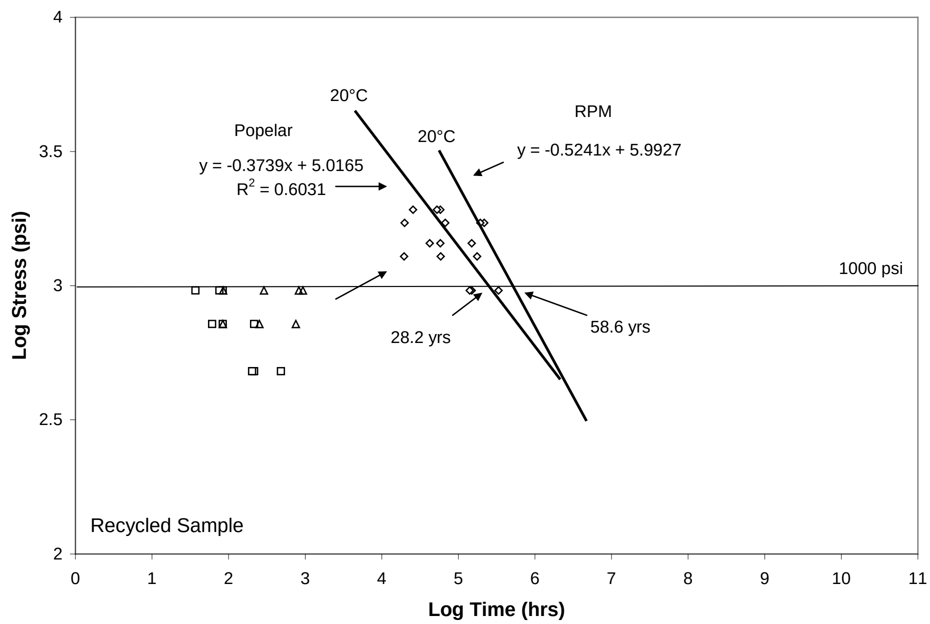

Figure D-47 - Comparison Between RPM and Popelar Projections on Recycled Sample from Reference 9

In this particular case, the bi-directional shifting is conservative compared to the rate process method for both sets of data.

The second paper reported a comparison between these two methods of data projection on both a notched liner test and the FLDOT junction test (16). For the liner test, samples were evaluated at 80, 70, 60, and 40ºC and the first three temperatures projected to 40ºC. The result was that the RPM method produced a projected line that fit the 40ºC data very well, while the bi-directional shifting was non-conservative. The projections showed that at about 460 psi, the RPM projected the sample to break at 80 days, while the bi-directional shifting projected 152 days.

The results were different for the junction tests. These were performed at 80, 70, and 60ºC and the two highest temperatures were projected to 60ºC. In this case, both the RPM and bi-directional shifting gave very similar results. And, both were close to the actual results, but slightly conservative.

A third comparison was done on the hydrostatic test results presented in PPI TN-16. This is shown in Figure D-48.

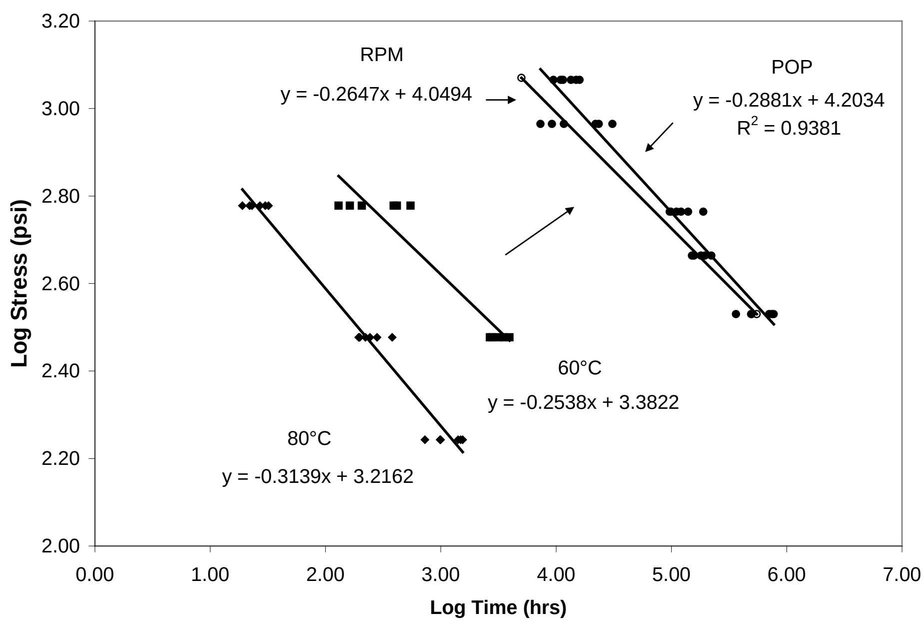

Figure D-48 - Comparison Between RPM and POP for Data Found in PPI TN-16

In this case, there was excellent agreement between the two data analysis methods. A comparison of 10 other data sets was made in 1990 and the agreement was generally good, except in those cases with smaller data sets (14). It could be argued that the quality of the data set determines how well the two methods correlate. Smaller data sets with more scatter do not seem to fit as well as larger, more uniform results. It's possible that

the agreement between the two methods can be used to evaluate the quality of the original data.

D.8.2.5 Long-Term Stress Crack Resistance

The long-term stress crack resistance of pipe formulations containing recycled will certainly be the life-limiting property for properly installed corrugated pipes. The contaminants present will be locations for stress cracks to grow. How quickly a crack grows will depend on the size, shape, and hardness of a particle along with the inherent stress crack resistance of the resin or resin blend. In fact, it would not be an understatement to say that the resistance to slow crack growth is the critical property for the success of this project.

The success that the gas pressure pipe industry has had reducing failures can be used as a model for creating a testing protocol to evaluate recycled containing pipe blends. For pressure pipe, the long-term strength is determined first (ductile failures) at room temperature, then a validation test is performed at 80ºC or 90ºC to ensure that brittle crack growth will not occur during the service lifetime of the pipe. Validation can be performed two ways; one based on the Rate Process Method (RPM) and one based on Popelar bi-directional shifting (POP).

Therefore, a test similar to the long-term hydrostatic strength test for pressure pipe is needed.

The best features of the long-term hydrostatic strength test include:

- The finished product can be tested,
- The thickness control of the test specimen is excellent,
- The applied stress is uniform throughout the specimen,
- There is no notch, so failures occur at naturally occurring flaws,
- The results relate to the real world.

There have been three different long-term stress cracking tests proposed for corrugated drainage pipe in the past few years (3,4,7). These are commonly known as the BAM Test, the FLDOT Junction Test, and the Pipe Ring Test.

The test used in this project for the purposes of evaluating recycled resin formulations is a modified version of the BAM test called the BFF test. This combines the specimen size of the BAM test, with the conditions of the FL-DOT durability test, and with a new test specimen called the Fathead. This test was discussed in detail in Section D.3.2

The FLDOT Junction test was also performed under one set of conditions to determine how the trial pipes matched up to the end-of-test time in the junction test.

D.8.2.5.1 The FLDOT Junction Test. The FLDOT has sponsored a research project to develop test methods to determine if corrugated pipe can have a service lifetime of 100

years (6). One of the tests involves stress crack tests without a notch on specimens taken from the pipe. The particular location tested is the junction between the liner and the corrugation. FLDOT Test Method FM 5-572 covers the performance of the test and FM 5-573 covers the procedures for extrapolation of the test results. The junction test specimen is an ASTM Type IV dumbbell, which has a reduced area that is ¼” x 1.3”. The advantages for this test include:

- It is performed on specimens from the finished pipe.
- It is related to field failures.
- It is more sensitive to the manufacturing process than to the basic resin.
- The well known rate process method for data analysis can be applied.

On the other hand, some disadvantages are:

- The thinnest part of the test specimen is often not the junction but the liner on either side.
- Only the thinnest part of the specimen experiences the full stress.
- There are significant sample preparation stresses imparted, especially in larger diameters.
- There are significant edge effects from the cutting die, especially on larger diameters.

Test method FM 5-573 calls for the test to be run under 3 different conditions of stress and temperature in D.I. water. These are shown in Table D-13. Five replicates under each set of conditions are tested.

Table D-13 - FLDOT FM 5-573 Test Conditions

Test Temp. (°C)	Applied Stress (psi)
70	650
80	650,450

The 3 sets of results are then analyzed by the rate process method (RPM), which has been used for many years on pressure pipe.

The test protocol also allows the tests to be terminated when they reach the following conditions:

- Terminate at 110 hours for 80°C/650 psi.
- Terminate at 430 hours for 80°C/450 psi.
- Terminate at 500 hours for 70°C/650 psi.

These times were determined with the use of an average slope of a number of stress crack tests and Popelar Bi-Directional Shifting.

D.8.2.5.2 The BFF Test. The BAM-FLDOT-Fathead (BFF) Test was described in detail previously. Recall that it uses a specimen, cut from a plaque, about the size of the BAM specimen (4 in x 0.5in). The test is performed under the conditions of the FLDOT durability protocol (80°C/650psi, 80°C/450 psi, 70°C/650 psi) in D.I. water with a dumbbell shaped test specimen in which the ends are twice as thick as the center to reduce failures at the grips. The advantages of this test for testing pipe containing recycled HDPE are:

- The exposed surface area is about 7 times larger than the FLDOT specimen.
- The thickness is controlled so the stress is even throughout the specimen.
- The specimen thickness (0.0040”-0.0045”) means flaws will have a greater effect than on thicker specimens.
- A wider and thinner test specimen means less edge and cutting die effects.
- It is very sensitive to the kinds of contaminants found in PCR-HDPE, especially silicone rubber.

On the other hand, this test will not be able to evaluate manufacturing stresses like the FLDOT test.

Similarly, if the Junction test could be performed on wider and longer test specimens, the results would be more meaningful. However, this would require stress crack testing devices that were larger to accommodate a wider specimen and greater loads. The Junction Test with a Type IV specimen can be performed on most conventional stress crack frames.

D.8.3 Long-Term Test Results on 6 Pipe Formulations

D.8.3.1 Long-Term Tensile Strength by SIM

The long-term tensile yield strength (Stage I) was determined by SIM. It was used to determine the 50 and 100 year tensile strengths of the six candidate pipe formulations. SIM tests were performed at 3 levels of stress, namely 1000, 1500, and 2000 psi.

The test specimens were placed under the appropriate load then a series of 10,000 second (166 minute) creep rupture tests were performed on the same specimen and separated by 7°C temperature steps. The test was continued until the specimen yielded. The results from the 3 loads were analyzed according to the standard and master creep rupture curves were prepared. The rupture point for each load was defined by the intersection of two tangent lines drawn before and after yielding.

The results for Sample B1 (100% VR1) are shown in Figure D-49. Tabulated results for the six candidate formulations are shown in Table D-14.

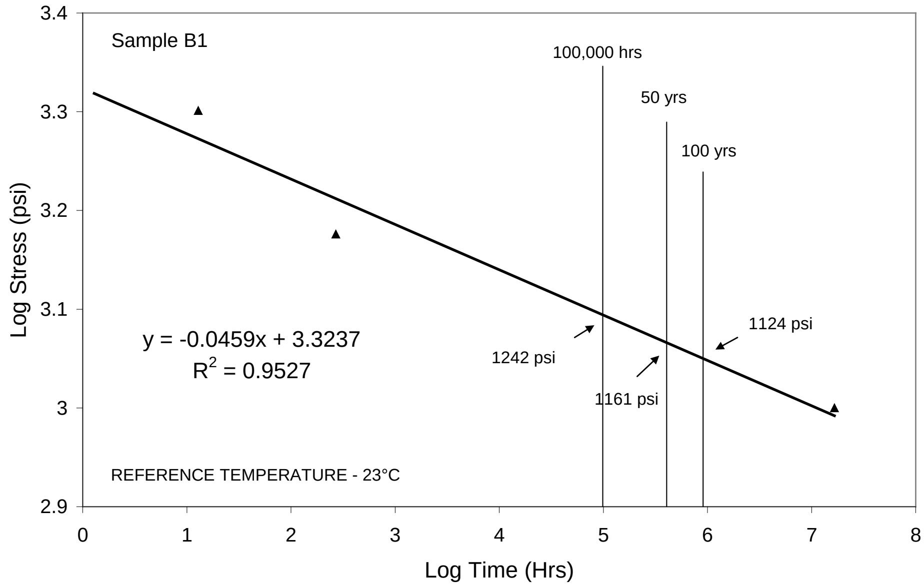

Figure D-49 - Long-Term Yield Strength of Sample B1

Table D-14 - Long-Term Tensile Stress of Six Formulations

Sample/Formulation	Long Term Tensile Stress (psi)		
	100,000 hrs	50 Yrs	100 Yrs
B1 100% VR1	1242	1161	1124
A2 85% VR1 + 15% MCR	1227	1145	1108
L5 50% VR1 + 20% MD + 30% MCR	1184	1103	1067
A5 40% VR1 + 30% MD + 30% NAT	1192	1105	1066
B3 20% VR1 + 40% MD + 24% MCR + 16 NAT	1064	973	934
B5 40% MD + 36% MCR + 24% NAT	1164	1082	1045

These predicted values assume that the material will stay basically unchanged over the 50 or 100 years of service lifetime. This, of course, is not true; all materials undergo aging effects during service. However, these are the best models currently available, and give a good approximation of the loss in tensile strength over time. These results basically suggest that the yield strength of the material will be about 30% of the initial strength after 50 years. So, as long as the applied stress is less than about 1000 psi, the material

will not fail in a ductile manner. The long-term strength is largely governed by the short-term strength. Therefore, to ensure a 1000 psi yield strength after 50 years, the short-term strength should be over 3500 psi.

It would be interesting to compare these results with others obtained through long-term hydrostatic testing, either at room temperature of elevated temperatures to verify that the two methods produce similar results.

D.8.3.2 Long-Term Creep Strain and Modulus by SIM

The long-term modulus values on the six final candidate resins were also determined in accordance to ASTM D6992.

Initially, the elastic limit of the samples was determined by two, short-term (15 min) creep experiments. This is important for defining when non-reversible creep actually begins.

Next, the stepped isothermal method (SIM) was used to determine the creep properties of the samples when placed under an applied stress of 500 psi. This involves a series of 10,000 second (166 min) creep tests, each done on the same test specimen and separated by 7°C in temperature. In this case, creep curves were generated from 20 to 83°C. The raw data are then shifted through time-temperature superposition (TTS) to generate a master creep curve at a reference temperature (23°C). Creep strain and creep modulus master curves for sample B1 (100% VR1) are shown in Figures D-50 and D-51, and all of the results are shown in Table D-15.

The raw data and the master creep curves for these samples are found in Section D.12. These results will be used to evaluate how one would design with recycled formulations in the context of Section 12 of the AASHTO LFRD Bridge Design Specifications.

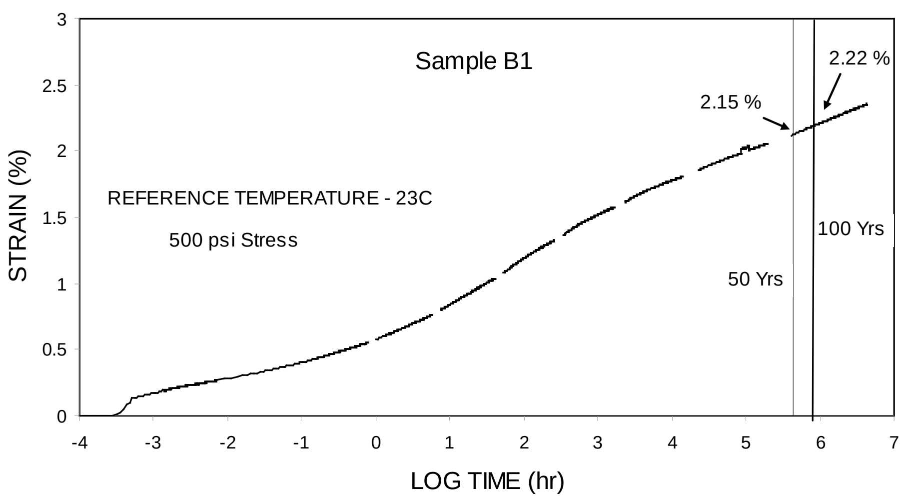

Figure D-50 - Creep Strain Master Curve for Sample B1

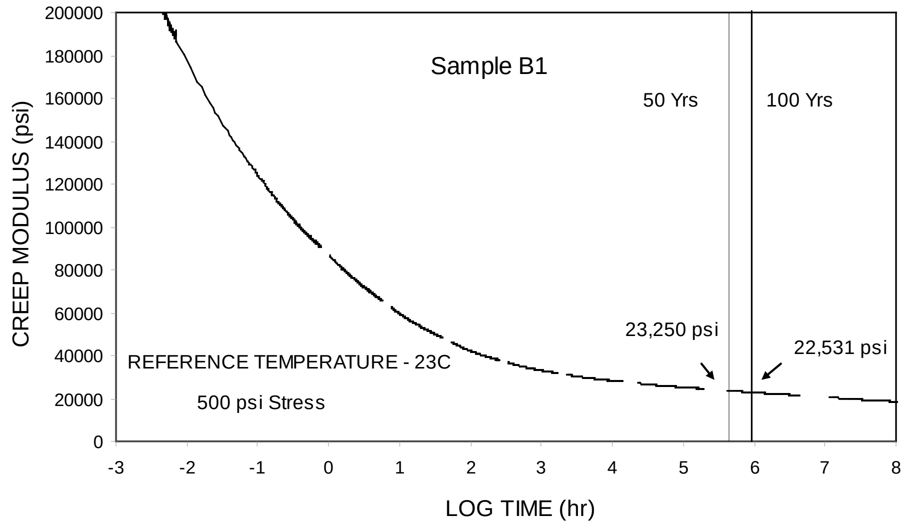

Figure D-51 - Creep Modulus Master Curve for Sample B1

Table D-15 - Long-Term Creep Strain and Creep Modulus Under 500 psi of Stress

Sample	Creep Strain		Creep Modulus	
	50 yr.	100 yr.	50 yr.	100 yr.
B1	2.15	2.22	23,250	22,531
A2	2.07	2.14	24,160	23,358
L5	2.22	2.30	22,549	21,719
A5	2.19	2.26	22,821	22,091
B3	2.24	2.32	22,300	21,532
B5	2.64	2.72	18,965	18,406

D.8.4 Long-Term Stress Crack Resistance Test Results

The six final candidate formulations and the PCR mixed color reprocessed resin were evaluated by the BFF test under the 3 sets of conditions cited in the FLDOT protocol. However, the results generated are believed to be unreliable due to the presence of residual surfactant in the baths. Therefore, there are no reliable service lifetime estimates for all of the six final candidates.

Once the issue with the baths was resolved, the BFF tests were repeated under one set of conditions for 8 samples, under two sets of conditions for 3 samples, and under three sets of conditions for 2 samples. The results are shown in Table D-16.

Table D-16 – Recent BFF Test Results

Sample	BFF Failure Time (hrs)		
	80ºC/650 psi	80ºC/465 psi	70ºC/650 psi
B1 100VR1	411 ± 135 (33%)	1601 ± 537 (34%)	1627 ± 680 (42%)
A2 85VR1 + 15MCR	257 ± 85 (33%)		
L5 50VR1 + 20MD + 30MCR	431 ± 86 (20%)		
A5 40VR1+ 30MD + 30NAT	323 ± 60 (19%)	1364 ± 571 (42%)	1615 ± 644 (40%)
B3 20VR1+40MD+24MCR+16NAT	330 ± 66 (20%)		
B5 40MD + 36MCR + 24NAT	280 ± 95 (34%)		
L1 Propriety Blend – 50% Recy.	201 ± 39 (19%)		
RPM Propriety Blend 100 % Recy.	620 ± 376 (61%)	1480, 4>3000	

Notice that a new sample was evaluated. This material is a fully formulated pipe blend containing 100% PIR and PCR recycled polyethylene, submitted by a recycled resin supplier (Recyc RPM, Inc.). It represents the possibilities for recycled resins when they are not based on typical virgin corrugated pipe resins. Some of the important properties of this resin are shown in Table D-17.

Table D-17 – Properties of Pipe Resin Containing 100% Recycled PE

Property	Result
Corrected Density	0.948 g/cm^3
Melt Index	0.11 g/10 min
% Color	3.9
% Ash	0.7
% PP	4.8
Flexural Modulus	128,606 psi
Yield Stress	3260 psi
Break Strain	21 %
NCLS Stress Crack Resistance	220 ± 54 hrs

The stress crack resistance for this material was outstanding, but the yield stress was a little low, the break strain was poor, and it contained about 0.7 % of ash. Melt filtration

at 150 mesh reduced the %Ash to 0.4% and raised the Break Strain to 225%. This is the type of material that can be developed for use in corrugated pipe.

Since there are failure times under three sets of conditions for samples B1 and A5, one can make a service lifetime estimate through bi-directional shifting. The master curves for stress crack resistance for Samples B1 (100% Virgin) and A5 (30% Recycled) are shown in Figures D-52 and D-53.

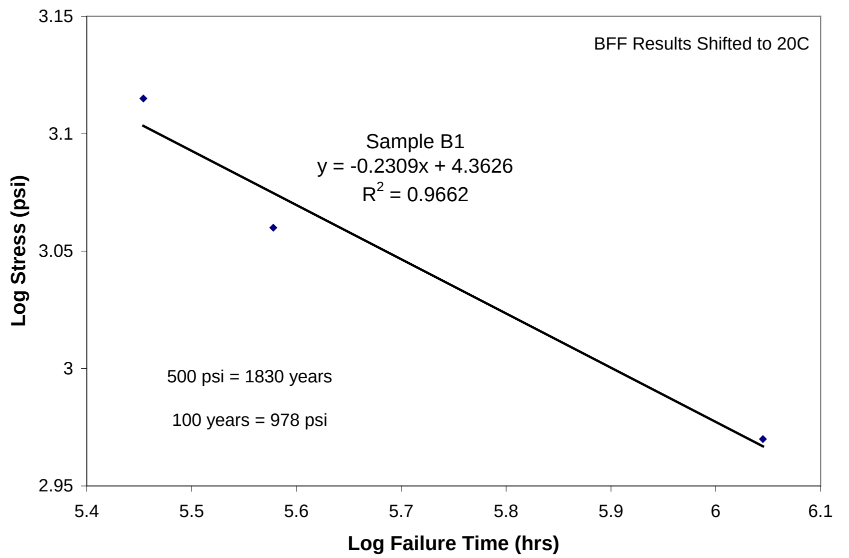

Figure D-52 – BFF Stress Cracking Master Curve at 20ºC for Sample B1

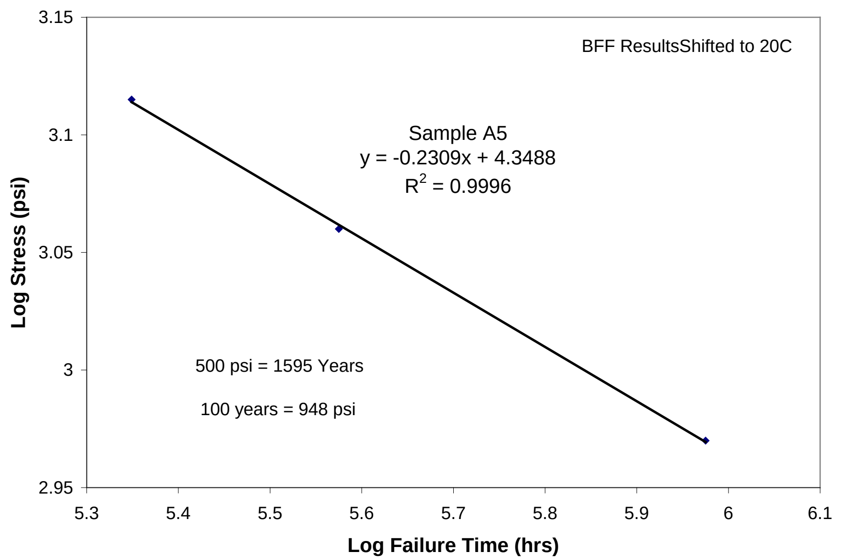

Figure D-53 – BFF Stress Cracking Master Curve at 20ºC for Sample A5

These results suggest that the service lifetime under 500 psi of stress will be well above 1000 years and that even at stresses of 900 psi, the estimated lifetime is over 100 years.

D.8.5 The BFF Test for Quality Control

There is no question that the successful use of recycled polyethylene depends on the stress crack resistance of the manufactured pipe. And, because of the inherent variability in recycled resins, each lot should be tested for stress crack resistance. Therefore, a quality control test is needed that will relate to the long-term stress crack resistance of the pipe, be of a reasonable duration of time, be sensitive to the base resin stress crack resistance, and be able to determine the effects of contaminants.

The BFF test is a good candidate. This test is done on a relatively thin plaque made from the pipe. The test specimen is a modified ASTM D638, Type I dumbbell, which has a surface area of 7.2 cm^2 (1.1 in^2). The thickness is about 1.1 mm (0.045 in), which means that even a 0.12 mm (0.005 in) particle will be 11% of the thickness. This ensures that the effects of particles will be determined. The test does not evaluate the actual end product, like the FLDOT Junction Test. However, because of its thickness, larger surface area, and consistent stress, the BFF test is the more aggressive test. In fact, results showed that failure times at 80ºC/650 psi averaged 2.5 times faster in the BFF test compared to the Junction Test when 12 inch pipe was tested. The results from four recent, side-by-side tests are shown in Table D-18.

Table D-18 – Comparison between BFF and Junction Tests at 80ºC/650 psi

Sample	BFF Time (Hrs)	Junction Time (Hrs) [liner breaks]	Junction/BFF
B1	414 ± 97 (23%)	1076 ± 561 (52%) [4]	2.6
B5	228 ± 141 (62%)	578 ± 224 (39%) [4]	2.6
L1	208 ± 73 (35%)	362 ± 201 (55%) [4]	1.7
L5	334 ± 116 (35%)	1342 ± 300 (22%) [4]	4.0

This comparison is complicated by the fact that nearly all the junction test specimens failed in the liner and not at the junction. The average of these data sets show that the BFF test is at least 1.7 times faster than the junction test.

The FLDOT 100 year service lifetime protocol calls for the junction test to be run under 3 sets of conditions and sets minimum time requirements for each condition. These are shown in Table 19.

Table 19 - FLDOT Junction Test Conditions and Minimum Failure Times

Test Conditions	Minimum Failure Time
80ºC/4.48 MPa (650 psi)	110 hrs
80ºC/3.10 MPa (450 psi)	430 hrs
70ºC/4.48 MPa (650 psi)	500 hrs

These values were determine with the use of Popelar Bi-directional shift factors and the 95% lower confidence interval based on a Student-t distribution. When one applies the Popelar shifts, the 20ºC master curve can be generated. This is shown in Figure D-54.

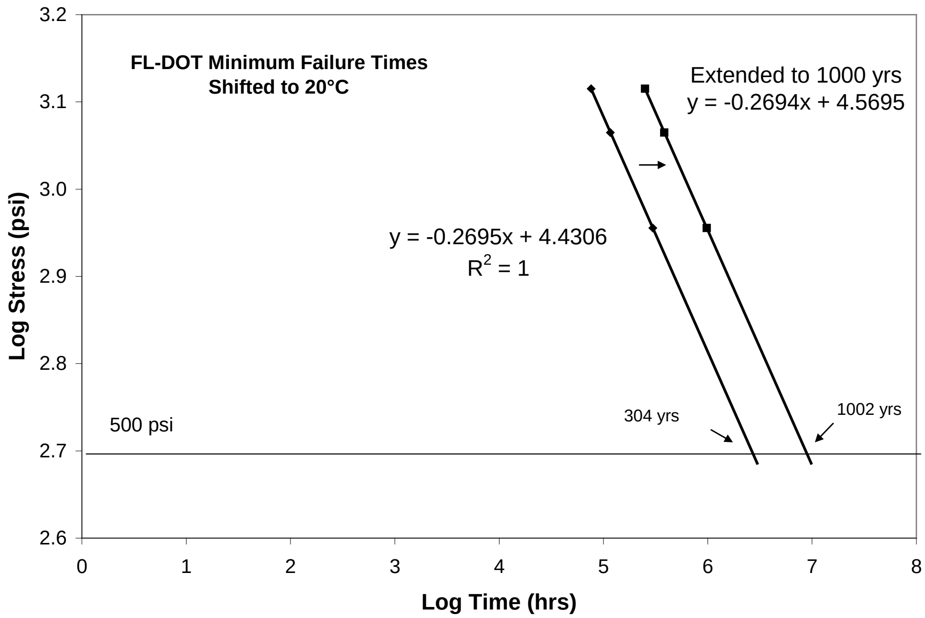

Figure D-54 - Master Curve from FLDOT Minimum Times

The master curve shows that a pipe sample under 3.45 MPa (500 psi) of load will not stress crack until 304 years. This shows that the design factor for a 100 year life is 0.33 (307 x 0.33 = 100). This is more conservative than the design factor of 0.50 used for solid wall water pipe.

Now, since there is uncertainty with the use of recycled polyethylene, a very conservative approach would be to use a design factor of 0.10. That would mean that the test results will have to show that the pipe will last for 1000 years. The master curve for 1000 years is also shown in Figure 30.

The slope of the line is reasonable for the resins used in corrugated pipe. The average slope from PPI (14), Hsuan (15), and this study on seventeen data sets was -0.26, with only four values over -0.27.

The minimum FLDOT times to generate the 1000 yr lifetime curve are shown in Table D-19.

Table D-20 - Minimum Times for a 1000 Year Estimated Lifetime

Test Conditions	Minimum Failure Time
80ºC/4.48 MPa (650 psi)	360 hrs
80ºC/3.10 MPa (450 psi)	1401 hrs
70ºC/4.48 MPa (650 psi)	1647 hrs

This shows that if a Junction Specimen lasts 360 hrs without failure in a 80ºC bath under 4.48 MPa (650 psi) of load, it will be estimated to last 1000 years.

Since it has been demonstrated that the BFF test is at least 1.7 times faster than the junction test at 80ºC/4.48 MPa (650 psi), it would be conservative to set the requirement for the BFF test at 200 hrs. Therefore, the proposed minimum average failure time in the single point BFF test for quality control is 200 hrs, or 8.3 days. This is not an ideal time for a QC/QA test, but even longer times are used for HDPE geomembranes. The geomembrane manufacturers perform the test on every railcar of resin and some QA specifications require the test for each batch of resin, or sometimes every 100,000 sq. ft. of material. Additionally, these tests are typically run for 300 or 400 hrs. A durability test like this one is critical for the successful use of recycled HDPE in corrugated drainage pipe for highway applications. And, every lot of recycled-containing resin needs to be tested because of the variable nature of recycled resins.

The BFF stress cracking master curves in Figures D-52 and D-53 independently support the value of 200 hrs for a QC test at 80ºC/650 psi. From the slopes of those lines and shifting back to 80ºC, the time that represents 1000 years of service for both samples is 200 hrs.

D.9 CONCLUSIONS

This phase of the project covered the manufacturing and testing of 15 pipe samples made in three different manufacturing plants. Both short-term and long-term properties were evaluated and the results analyzed and presented. Some of the more significant conclusions include:

1. Pipe resins containing recycled HDPE can be easily processed on current equipment. The only adjustments made at any of the three manufacturing plants were small changes in line speed. Of course, only limited runs were performed. There could be problems with die-lip build-up or frequent screen changes during longer production times.
2. It was found that the density of pipe resins containing recycled HDPE is difficult to measure and that it may be more valuable to just specify a mechanical property like yield strength or flexural modulus.
3. The melt index, % ash, and % polypropylene results indicated that most of the blends were made correctly. The % carbon black results showed that this property is more difficult for pipe manufacturers to control.

4. The BFF test shows promise as both a quality control test and a test used to make lifetime projections. Validation tests showed that the exposure bath was changing so more work is required to ensure a consistent exposure bath and a more useful test.
5. The BFF test was shown to be very sensitive to the types of contaminants found in recycled resins and is more aggressive than the FL-DOT Junction test because the specimen is uniform in thickness and relatively thin.
6. It was demonstrated that pipe containing up to 60% recycled content could be manufactured that meets the required cell classification and 18 hrs of stress crack resistance on a plaque made from the pipe. The yield stress was a bit low, so the sample did not meet the 50 year creep modulus of 22,000 psi.
7. The results indicate that pipe containing recycled HDPE would be appropriate for AASHTO M252 applications. M252 requires that the base resin be in the D3350 Cell Classification of 324420C. Even 100% recycled would probably meet this specification. M252 requires the resin to be exposed to 100% Igepal at 50ºC for 24 hours and have 50% of the samples not crack. This is a very weak requirement that any of the blends made for this study would meet. This requirement should be strengthened to the NCLS test with a reasonable requirement, like 15 hrs on a plaque made from the pipe. Resin blends containing 50% and more recycled could easily be made that would meet the requirements of M252.
8. The main question of this research has not been adequately answered. How much recycled can one use and still have an equivalent product to virgin? If equivalency means meeting M294, then up to 60% recycled is possible. As far as cracking resistance, 100% recycled better than virgin in the NCLS, while 30% was as good as virgin in the BFF test. The true answer will only come from refinement of the BFF test, and including newer bi-modal resins as blending resins.

The most significant conclusion of this effort may be the realization of the value of the pipe samples. There are 80 feet of pipe left for each of the 15 samples. The pipes have a variety of properties. Some do not meet the cell classification for AASHTO M294 pipe. Some display low density or low elongation at break or have poor stress crack resistance. They vary in their stress crack resistance in an un-notched test. The poorest had a failure time about 25% of the best's time. There are some that have peak loads less than 20% deflection and others that are over 40% in pipe deflection tests. And, there are some that won't meet the required 50 year creep modulus of 22,000 psi. These pipe samples are well suited to evaluate some of the assumptions and long-term projections made for corrugated drainage pipes. The service lifetime projections made suggest that these pipes should fail by brittle cracking in less than 5 years under 1500 psi of stress and in less than 1 year under 2000 psi of stress. And these are average failure times. The known variability in the samples suggests that initial breaks will come much earlier. This can easily be verified, or not verified, by room temperature laboratory tests under dead loads, or field tests on installed pipes. One could even perform tests at slightly elevated temperatures of 35 or 40º C to generate results even more quickly or to reduce the high applied loads.

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D.11 TEST REPORTS ON PLAQUES MADE FROM RESINS AND PIPES

TEST RESULTS
Corrugated Pipe with Recycled HDPE
ADS Pipe Trial
100% PCR MCR

Material: Plaque from pellets
Sample: 100% Solplast MCR

Date: 28-May-08
TRI Log #: F7601

PARAMETER	1	2	3	4	5	Mean	STD
	Test Replicate Number						
Density (ASTM D 1505)							
Density (g/cm3)	0.958	0.959	0.959			0.959	0.000
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.49	0.44				0.47	
Composition							
% Color	0.06	0.09	0.09			0.08	0.00
% Ash	1.17	1.17	1.16			1.17	0.01
% PP	4.6	4.3	4.4			4.43	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	146,991	141,026	137,813	141,302	149,266	143,280	1584
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3695	3795	3725	3783	3786	3757	31
Break Strain (%)	141	100	90	102	15	90	5
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	6.5	5.8	5.8	6.1	6.1	6.1	0.1
Thermal Stability							
Induction Temp.(C) (ASTM D3350	236	237	237			237	

TEST RESULTS
Corrugated Pipe with Recycled HDPE
ADS Pipe Trial
100% PCR Natural

Material: Plaque from pellets
Sample: 100% Solplast Natural Repro

Date: 28-May-08
TRI Log #: F7601

PARAMETER	Test Replicate Number 1	2	3	4	5	Mean	STD
Density (ASTM D 1505)							
Density (g/cm3)	0.961	0.961	0.961			0.961	0.000
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.71	0.69				0.70	
Composition							
% Color	0	0	0			0.00	0.00
% Ash	0	0	0			0.00	0.00
% PP	0					0.00	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	184,286	186,461	179,398	180,555	174,010	180,942	3093
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	4563	4559	4446	4521	4500	4518	47
Break Strain (%)	26	28	97	23	15	38	34
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	2.4	2.6	2.9	2.8	2.6	2.7	0.1
Thermal Stability							
Induction Temp.(C) (ASTM D3350	234	235	238			236	

TEST RESULTS
Recycled HDPE Blend
Blue Diamond Pipe Trial
PCR-MCR

Material: Plaque from blended resin (MB 3X)
Sample: 100% MCR

Date: 19-Sep-08
TRI Log #: F7601

PARAMETER	1	2	3	4	5	Mean	STD
	Test Replicate Number						
Density (ASTM D 1505)							
Density (g/cm3)	0.958	0.958	0.958			0.958	0.000
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.49	0.50				0.50	
Composition							
% Color	1.22	1.17	1.24			1.21	0.03
% Ash							
% PP	0					0.00	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	141,204	141,519	141,865	144,680	144,970	142,848	1416
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3647	3635	3785	3799	3687	3711	74
Break Strain (%)	52	65	35	20	52	45	19
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	5.8	6.3	6.5	4.9	6.0	5.9	0.7
Thermal Stability							
Induction Temp.(C) (ASTM D3350	239	243	243			242	
Induction Time (min) (ASTM D3895)							

TEST RESULTS
Recycled HDPE Blend
Blue Diamond Pipe Trial
VR1 - Resin

Material: Plaque from blended resin (MB 3X)
Sample: 100% VR1

Date: 26-Jun-08
TRI Log #: F7601

	Test Replicate Number						
PARAMETER	1	2	3	4	5	Mean	STD
Density (ASTM D 1505)							
Density (g/cm3)	0.962	0.964	0.961			0.96	0.002
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.14	0.14				0.14	
Composition							
% Color	2.46	2.48	2.41			2.45	0.03
% Ash	0.15	0.18	0.15			0.16	0.02
% PP	0					0.00	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	159,880	152,301	153,846	162,229	160,750	157,801	4362
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3716	3877	3732	3842	3718	3777	62
Break Strain (%)	439	329	469	231	227	339	98
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	36.7	36.6	30.5	43.6	33.6	36.2	5.4
Thermal Stability							
Induction Temp.(C) (ASTM D3350	257	256	256			256	
Induction Time (min) (ASTM D3895)							

TEST RESULTS
Recycled HDPE Blend
Blue Diamond Pipe Trial
50% VR1 + 20% MDPE + 30% MCR

Material: Plaque from blended resin (MB 3X) | Date: 26-Jun-08
Sample: B-2 | TRI Log #: F7601

PARAMETER	1	2	3	4	5	Mean	STD
	Test Replicate Number						
Density (ASTM D 1505)							
Density (g/cm3)	0.960	0.960	0.962			0.961	0.001
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.19	0.21				0.20	
Composition							
% Color	2.13	2.12	2.15			2.13	0.01
% Ash	0.39	0.36	0.39			0.38	0.02
% PP	1.3					1.30	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	153,314	142,988	141,865	138,641	141,804	143,722	1842
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3548	3526	3513	3513	3493	3519	6
Break Strain (%)	264	461	356	420	427	386	43
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	63.1	44.7	94.1	55.4	74.4	66.3	21.2
Thermal Stability							
Induction Temp.(C) (ASTM D3350	255	255	256			255	
Induction Time (min) (ASTM D3895)							

TEST RESULTS
Recycled HDPE Blend
Blue Diamond Pipe Trial
20% VR1 + 40% MDPE + 24% MCR + 16% NAT

Material: Plaque from blended resin (MB 3X)
Sample: B-3

Date: 26-Jun-08
TRI Log #: F7601

	Test Replicate Number						
PARAMETER	1	2	3	4	5	Mean	STD
Density (ASTM D 1505)							
Density (g/cm3)	0.958	0.958	0.958			0.958	0.000
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.26	0.26				0.26	
Composition							
% Color	2.13	2.21	2.12			2.15	0.04
% Ash	0.31	0.36	0.31			0.33	0.03
% PP	1.2					1.20	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	144,208	134,770	133,125	132,671	129,480	134,851	902
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3408	3423	3443	3472	3365	3422	20
Break Strain (%)	458	222	234	462	443	364	110
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	105.0	62.2	58.2	71.0	63.3	71.9	5.3
Thermal Stability							
Induction Temp.(C) (ASTM D3350	258	258	258			258	
Induction Time (min) (ASTM D3895)							

TEST RESULTS
Recycled HDPE Blend
Blue Diamond Pipe Trial
50% VR1 + 50% NAT

Material: Plaque from blended resin (MB 3X)
Sample: B-4

Date: 26-Jun-08
TRI Log #: F7601

PARAMETER	1	2	3	4	5	Mean	STD
	Test Replicate Number						
Density (ASTM D 1505)							
Density (g/cm3)	0.955	0.955	0.955			0.955	0.000
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.37	0.36				0.37	
Composition							
% Color	1.88	1.83	1.96			1.89	0.06
% Ash	0.09	0.07	0.12			0.09	0.03
% PP	0					0.00	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	135,108	137,478	137,473	141,204	142,688	138,790	1758
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3486	3453	3434	3392	3435	3440	25
Break Strain (%)	438	520	476	508	522	493	19
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	62.3	42.4	40.5	58.5	36.6	48.1	8.1
Thermal Stability							
Induction Temp.(C) (ASTM D3350	261	258	260			260	
Induction Time (min) (ASTM D3895)							

TEST RESULTS
Recycled HDPE Blend
Blue Diamond Pipe Trial
40% MDPE + 36% MCR + 24% NAT

Material: Plaque from blended resin (MB 3X)
Sample: B-5

Date: 26-Jun-08
TRI Log #: F7601

PARAMETER	Test Replicate Number 1	2	3	4	5	Mean	STD
Density (ASTM D 1505)							
Density (g/cm3)	0.957	0.957	0.957			0.957	0.000
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.33	0.32				0.33	
Composition							
% Color	2.42	2.42	2.36			2.40	0.03
% Ash	0.48	0.42	0.42			0.44	0.00
% PP	1.8					1.80	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	134,087	133,307	136,413	140,739	137,081	136,325	3048
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3420	3389	3392	3377	3319	3379	6
Break Strain (%)	259	461	391	299	440	370	66
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	43.3	45.7	40.8	37.2	24.9	38.4	3.5
Thermal Stability							
Induction Temp.(C) (ASTM D3350	256	255	254			255	
Induction Time (min) (ASTM D3895)							

TEST RESULTS
Recycled HDPE Blend
Lane Pipe Trial
100% MCR

Material: Plaque resin (no blending)
Sample: 100% MCR

Date: 25-Jul-08
TRI Log #: F7601

PARAMETER	Test Replicate Number 1	2	3	4	5	Mean	STD
Density (ASTM D 1505)							
Density (g/cm3)	0.963	0.961	0.961			0.96	0.000
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.50	0.50				0.50	
Composition							
% Color	0.04	0	0			0.01	0.00
% Ash	1.18	1.17	1.23			1.19	0.03
% PP	0					0.00	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	145,169	156,267	152,778	147,733	150,872	150,564	3503
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	4098	3913	3948	4176	3755	3978	117
Break Strain (%)	47	57	85	64	74	65	12
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	5.4	4.4	5.1	5.3	5.1	5.1	0.4
Thermal Stability							
Induction Temp.(C) (ASTM D3350	240	242	243 `			242	
Induction Time (min) (ASTM D3895)							

TEST RESULTS
Recycled HDPE Blend
Lane Pipe Trial
100% Pipe Solplast Resin

Material: Plaque resin (no blending)
Sample: L-1

Date: 25-Jul-08
TRI Log #: F7601

PARAMETER	1	2	3	4	5	Mean	STD
	Test Replicate Number						
Density (ASTM D 1505)							
Density (g/cm3)	0.959	0.959	0.959			0.96	0.000
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.18	0.19				0.19	
Composition							
% Color	1.44	1.66	1.44			1.51	0.11
% Ash	0.91	0.91	0.9			0.91	0.01
% PP	0					0.00	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	132,800	130,667	134,366	134,330	134,531	133,339	1735
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3574	3846	3700	3891	3791	3760	82
Break Strain (%)	86	314	38	210	74	144	114
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	25.3	23.6	23.1	23.2	25.5	24.1	0.2
BAM Stress Crack Test							
Failure Time	8.30	6.50	6.50 `	7.40 grip	9.60	7.7	0.4
Thermal Stability							
Induction Temp.(C) (ASTM D3350	242	242	242 `			242	0.0

TEST RESULTS
Recycled HDPE Blend
Lane Pipe Trial
50% VR2 + 20% MDPE + 30% MCR

Material: Plaque from blended resin (MB 3X)
Sample: L-2

Date: 25-Jul-08
TRI Log #: F7601

PARAMETER	Test Replicate Number 1	2	3	4	5	Mean	STD
Density (ASTM D 1505)							
Density (g/cm3)	0.968	0.966	0.969			0.97	0.002
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.33	0.33				0.33	
Composition							
% Color	3.28	3.32	3.29			3.30	0.01
% Ash	0.52	0.5	0.58			0.53	0.04
% PP	0					0.00	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	134,259	141,274	132,966	138,067	133,421	135,997	3421
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3205	3266	3171	3205	3248	3219	39
Break Strain (%)	444	458	197	435	448	396	118
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	48.6	63.2	57.1	47.7	60.1	55.3	6.4
Thermal Stability							
Induction Temp.(C) (ASTM D3350	258	256	256			257	
Induction Time (min) (ASTM D3895)							

TEST RESULTS
Recycled HDPE Blend
Lane Pipe Trial
50% VR1 + 20% MDPE + 30% MCR

Material: Plaque from blended resin (MB 3X)
Sample: L-5

Date: 25-Jul-08
TRI Log #: F7601

PARAMETER	1	2	3	4	5	Mean	STD
			Test Replicate Number				
Density (ASTM D 1505)							
Density (g/cm3)	0.963	0.964	0.961			0.96	0.002
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.19	0.21				0.20	
Composition							
% Color	2.43	2.49	2.45			2.46	0.02
% Ash	0.32	0.31	0.34			0.32	0.02
% PP	0					0.00	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	145,727	159,270	151,899	149,014	155,844	152,351	4318
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3614	3687	3633	4044	3729	3741	182
Break Strain (%)	428	386	441	303	441	400	57
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	28.9	29.4	17.3	24.5	28.5	25.7	5.0
Thermal Stability							
Induction Temp.(C) (ASTM D3350	256	256	257			256	
Induction Time (min) (ASTM D3895)							

TEST RESULTS
Corrugated Pipe with Recycled HDPE
ADS Pipe Trial
100 % VR1

Material: Plaque from pipe A1
Sample: 100% VR1

Date: 19-Sep-08
TRI Log #: F7601

PARAMETER	1	2	3	4	5	Mean	STD
	Test Replicate Number						
Density (ASTM D 1505)							
Density (g/cm3)	0.959	0.959	0.959			0.959	0.000
Corrected Density (for carbon black)	0.952	0.952	0.952			0.952	0.000
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.12	0.12				0.12	
Composition							
% Color	1.58	1.58	1.59			1.58	0.01
% Ash	0.09	0.11	0.11			0.10	0.00
% PP	0					0.00	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	163,434	162,162	153,846	152,849	157,362	157,931	4175
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3880	3799	3887	3886	3803	3851	41
Break Strain (%)	244	127	197	164		183	29
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	34.0	34.0	27.4	35.9	34.4	33.1	3.6
(ASTM F2136, 600 psi)							
Failure Time (hours)	22.9	32.6	29.3	28.2	27.4	28.1	1.9
Thermal Stability							
Induction Temp.(C) (ASTM D3350	257					257	
Induction Time (min) (ASTM D3895)	49					49	

TEST RESULTS
Corrugated Pipe with Recycled HDPE
ADS Pipe Trial
VR1 + 15% MCR

Material: Plaque from pipe A2
Sample: 85% VR1 + 15% MCR

Date: 19-Sep-08
TRI Log #: F7601

PARAMETER	Test Replicate Number 1	2	3	4	5	Mean	STD
Density (ASTM D 1505)							
Density (g/cm3)	0.949	0.949	0.949			0.949	0.000
Corrected Density (for carbon black)	0.943	0.943	0.943			0.943	0.000
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.15	0.15				0.15	
Composition							
% Color	1.31	1.38	1.36			1.35	0.01
% Ash	0.25	0.25	0.25			0.25	0.00
% PP	0.7					0.70	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	151,381	151,753	154,778	150,310	154,813	152,607	1862
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3876	3815	3708	3812	3665	3775	50
Break Strain (%)	293	88	85	263	109	168	83
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	28.1	29.3	28.6	32.7	35.5	30.8	1.8
(ASTM F2136, 600 psi)							
Failure Time (hours)	19.3	23.2	23.2	16.5	23.7	21.2	2.8
Thermal Stability							
Induction Temp.(C) (ASTM D3350)	256					256	
Induction Time (min) (ASTM D3895)	38.6					39	

TEST RESULTS
Corrugated Pipe with Recycled HDPE
ADS Pipe Trial
VR1 + 15% NAT

Material: Plaque from pipe A3
Sample: 85% VR1 + 15% NAT

Date: 19-Sep-08
TRI Log #: F7601

PARAMETER	Test Replicate Number 1	2	3	4	5	Mean	STD
Density (ASTM D 1505)							
Density (g/cm3)	0.972	0.976	0.972			0.973	0.002
Corrected Density (for carbon black)	0.962	0.966	0.962			0.963	0.002
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.16	0.16				0.16	
Composition							
% Color	2.01	2.32	2.39			2.24	0.04
% Ash	0.26	0.2	0.15			0.20	0.03
% PP	0.20					0.20	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	160,398	161,410	155,939	161,011	156,250	159,002	2490
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3925	3659	3916	3992	3821	3863	142
Break Strain (%)	133	115	57	123	267	139	29
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	16.9	21.8	22.9	17.9	22.9	20.5	2.1
Thermal Stability							
Induction Temp.(C) (ASTM D3350)	257					257	
Induction Time (min) (ASTM D3895)	38					38	

TEST RESULTS
Corrugated Pipe with Recycled HDPE
ADS Pipe Trial
VR1 + 20% MD + 30% MCR

Material: Plaque from pipe A4
Sample: 50% VR1 + 20% MD + 30 MCR

Date: 19-Sep-08
TRI Log #: F7601

PARAMETER	Test Replicate Number 1	2	3	4	5	Mean	STD
Density (ASTM D 1505)							
Density (g/cm3)	0.953	0.954	0.954			0.954	0.000
Corrected Density (for carbon black)	0.951	0.952	0.952			0.952	0.000
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.18	0.19				0.19	
Composition							
% Color	0.49	0.4	0.47			0.45	0.04
% Ash	0.34	0.4	0.38			0.37	0.01
% PP	1.4					1.40	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	144,676	145,243	142,998	150,104	145,833	145,771	2966
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3677	3743	3739	3769	3620	3710	13
Break Strain (%)	153	307	254	275	217	241	22
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	28.5	26.5	31.4	26.7	33.6	29.3	2.3
(ASTM F2136, 600 psi)							
Failure Time (hours)	21.0	15.6	16.7	16.5	19.0	17.8	2.0
Thermal Stability							
Induction Temp.(C) (ASTM D3350)	256					256	
Induction Time (min) (ASTM D3895)	32					32	

TEST RESULTS
Corrugated Pipe with Recycled HDPE
ADS Pipe Trial
VR1 + 30% MD + 30% NAT

Material: Plaque from pipe A5
Sample: 40% VR1 + 30% MD + 30 NAT

Date: 19-Sep-08
TRI Log #: F7601

PARAMETER	1	2	3	4	5	Mean	STD
	Test Replicate Number						
Density (ASTM D 1505)							
Density (g/cm3)	0.955	0.955	0.956			0.955	0.001
Corrected Density (for carbon black)	0.948	0.948	0.949			0.948	0.001
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.21	0.21				0.21	
Composition							
% Color	1.52	1.55	1.49			1.52	0.03
% Ash	0.17	0.17	0.25			0.20	0.04
% PP	0.3					0.30	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	139,653	136,413	140,532	142,988	140,739	140,065	2713
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3672	3583	3613	3622		3623	17
Break Strain (%)	116	117	246	155		159	54
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	53.0	41.9	39.2	53.0	44.8	46.4	6.0
(ASTM F2136, 600 psi)							
Failure Time (hours)	27.7	27.8	29.2	28.3	29.8	28.6	0.6
Thermal Stability							
Induction Temp.(C) (ASTM D3350)	260					260	
Induction Time (min) (ASTM D3895)	95.5					96	

TEST RESULTS
Corrugated Pipe with Recycled HDPE
Blue Diamond Pipe Trial
100% VR1

Material: Plaque from pipe B1
Sample: 100% VR1

Date: 1-Sep-08
TRI Log #: F7601

PARAMETER	Test Replicate Number 1	2	3	4	5	Mean	STD
Density (ASTM D 1505)							
Density (g/cm3)	0.959	0.960	0.960			0.960	0.000
Corrected Density (for carbon black)	0.950	0.951	0.951			0.951	0.000
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.13	0.13				0.13	
Composition							
% Color	1.99	2.01	2.09			2.03	0.04
% Ash	0	0	0			0.00	0.00
% PP	0					0.00	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	150,109	149,458	142,625	150,394	148,466	148,210	3463
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3859	3852	3791	3959		3865	69
Break Strain (%)	125	297	85	143		163	89
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	44.4	35.5	50.5	42.3	39.2	42.4	6.1
(ASTM F2136, 600 psi)							
Failure Time (hours)	25.1	25.1	21.1	26.4	29.7	25.5	2.3
Thermal Stability							
Induction Temp.(C) (ASTM D3350)	253					253	
Induction Time (min) (ASTM D3895)	48.5					49.0	

TEST RESULTS
Pipe Containing Recycled HDPE
Blue Diamond Pipe Trial
50% VR1 + 20% MDPE + 30% MCR

Material: Plaque from Pipe B2
Sample: 50% VR1 + 20% MD + 30% MCR

Date: 1-Sep-08
TRI Log #: F7601

PARAMETER	Test Replicate Number 1	2	3	4	5	Mean	STD
Density (ASTM D 1505)							
Density (g/cm3)	0.956	0.956	0.958			0.957	0.001
Corrected Density (for carbon black)	0.947	0.947	0.949			0.948	0.001
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.22	0.21				0.22	
Composition							
% Color	2.05	2.10	2.00			2.05	0.05
% Ash	0.46	0.37	0.37			0.40	0.00
% PP	1.9					1.90	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	136,961	134,383	132,117	137,924	132,722	134,821	2390
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3643	3685	3691	3672	3500	3638	8
Break Strain (%)	446	157	226	451	433	343	126
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	38.2	36.1	28.2	35.7	38.2	35.3	3.6
(ASTM F2136, 600 psi)							
Failure Time (hours)	15.8	18.5	16.6	20.2	20.0	18.2	1.8
Thermal Stability							
Induction Temp.(C) (ASTM D3350)	254					254	
Induction Time (min) (ASTM D3895)	54					54	

TEST RESULTS
Pipe Containing Recycled HDPE
Blue Diamond Pipe Trial
20% VR1 + 40% MDPE + 24% MCR + 16% NAT

Material: Plaque from Pipe B3
Sample: 20% VR1 + 40% MD + 24% MCR + 16% NAT

Date: 1-Sep-08
TRI Log #: F7601

PARAMETER	Test Replicate Number 1	2	3	4	5	Mean	STD
Density (ASTM D 1505)							
Density (g/cm3)	0.957	0.957	0.957			0.957	0.000
Corrected Density (for carbon black)	0.948	0.948	0.948			0.948	0.000
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.27	0.27				0.27	
Composition							
% Color	1.96	1.93	1.96			1.95	0.02
% Ash	0.31	0.32	0.38			0.34	0.03
% PP	1.7					1.70	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	130,133	126,354	128,515	130,667	126,136	128,361	1761
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3071	3262	3187	3338	3398	3251	62
Break Strain (%)	94	196	433	437	341	300	113
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	63.2	65.6	35.1	68.5	70.5	60.6	15.1
(ASTM F2136, 600 psi)							
Failure Time (hours)	37.0	34.5	25.4	27.6	24.3	29.8	3.9
Thermal Stability							
Induction Temp.(C) (ASTM D3350)	256					256	
Induction Time (min) (ASTM D3895)	92.9					93	

TEST RESULTS
Pipe Containing Recycled HDPE
Blue Diamond Pipe Trial
50% MDPE + 50% NAT

Material: Plaque from Pipe B4
Sample: 50% MD + 50% NAT

Date: 1-Sep-08
TRI Log #: F7601

PARAMETER	1	2	3	4	5	Mean	STD
	Test Replicate Number						
Density (ASTM D 1505)							
Density (g/cm3)	0.954	0.954	0.955			0.954	0.001
Corrected Density (for carbon black)	0.948	0.948	0.949			0.948	0.001
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.36	0.36				0.36	
Composition							
% Color	1.48	1.4	1.44			1.44	0.02
% Ash	0.09	0.11	0.11			0.10	0.00
% PP	1.0					1.0	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	133,860	128,744	136,616	128,917	128,102	131,248	3671
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3521	3576	3499	3487	3533	3523	39
Break Strain (%)	197	149	197	283	125	190	55
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	34.1	33.9	40.2	47.0	38.1	38.7	5.3
(ASTM F2136, 600 psi)							
Failure Time (hours)	19.9	20.9	17.8	18.4	17.1	18.8	1.4
Thermal Stability							
Induction Temp.(C) (ASTM D3350)	257					257	
Induction Time (min) (ASTM D3895)	110					110	

TEST RESULTS
Pipe Containing Recycled HDPE
Blue Diamond Pipe Trial
40% MDPE + 36% MCR + 24% NAT

Material: Plaque from Pipe B5
Sample: 40% MD + 36% MCR + 24% NAT

Date: 1-Sep-08
TRI Log #: F7601

PARAMETER	Test Replicate Number 1	2	3	4	5	Mean	STD
Density (ASTM D 1505)							
Density (g/cm3)	0.960	0.960	0.960			0.960	0.000
Corrected Density (for carbon black)	0.952	0.952	0.952			0.952	0.000
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.34	0.34				0.34	
Composition							
% Color	1.9	1.95	1.89			1.91	0.03
% Ash	0.38	0.39	0.36			0.38	0.02
% PP	2.2					2.20	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	131,925	128,917	125,400	128,015	129,533	128,758	1491
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3350	3541	3211	3190	3263	3311	161
Break Strain (%)	281	454	413	352	256	351	42
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	47.5	46.7	39.7	44.5	41.4	44.0	2.9
(ASTM F2136, 600 psi)							
Failure Time (hours)	20.2	20.9	16.3	18.4	20.2	19.2	1.9
Thermal Stability							
Induction Temp.(C) (ASTM D3350)	256					256	
Induction Time (min) (ASTM D3895)	75.1					75	

TEST RESULTS
Pipe Containing Recycled HDPE
Lane Pipe Trial
Solplast Pipe Resin

Material: Plaque from Pipe L1
Sample: Solplast Pipe Resin

Date: 2-Sep-08
TRI Log #: F7601

PARAMETER	1	2	3	4	5	Mean	STD
	Test Replicate Number						
Density (ASTM D 1505)							
Density (g/cm3)	0.959	0.959	0.959			0.959	0.000
Corrected Density (for carbon black)	0.950	0.95	0.950			0.950	0.000
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.20	0.20				0.20	
Composition							
% Color	2.07	2.14	2			2.07	0.07
% Ash	0.8	0.69	0.73			0.74	0.02
% PP	2.5					2.50	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	138,748	136,751	137,774	132,031		136,326	2501
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3485	3705	3647	3688		3631	24
Break Strain (%)	215	163	463	268		277	124
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	24.4	22.3	26.2	24.3	25.7	24.6	1.6
Thermal Stability							
Induction Temp.(C) (ASTM D3350)	240					240	
Induction Time (min) (ASTM D3895)	13					13	

TEST RESULTS
Pipe Containing Recycled HDPE
Lane Pipe Trial
50% VR2 + 20% MDPE + 30% MCR

Material: Plaque from Pipe L2
Sample: 50% VR2 + 20% MD + 30% MCR

Date: 2-Sep-08
TRI Log #: F7601

PARAMETER	1	2	3	4	5	Mean	STD
	Test Replicate Number						
Density (ASTM D 1505)							
Density (g/cm3)	0.956	0.957	0.957			0.957	0.000
Corrected Density (for carbon black)	0.949	0.950	0.950			0.950	0.000
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.34	0.32				0.33	
Composition							
% Color	1.65	1.41	1.44			1.50	0.02
% Ash	0.43	0.41	0.4			0.41	0.00
% PP	2.4					2.40	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	139,434	140,046	139,468	137,925	136,358	138,646	895
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3733	3735	3794	3733	3758	3751	28
Break Strain (%)	121	239	111	304	200	195	80
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	40.4	33.4	41.1	33.7	39.2	37.6	3.6
(ASTM F2136, 600 psi)							
Failure Time (hours)	30.1	27.9	27.3	30.1	25.8	28.2	1.7
Thermal Stability							
Induction Temp.(C) (ASTM D3350)	256					256	
Induction Time (min) (ASTM D3895)	78					78	

TEST RESULTS
Pipe Containing Recycled HDPE
Lane Pipe Trial
100% VR2

Material: Plaque from Pipe L3
Sample: 100% VR2

Date: 2-Sep-08
TRI Log #: F7601

PARAMETER	1	2	3	4	5	Mean	STD
	Test Replicate Number						
Density (ASTM D 1505)							
Density (g/cm3)	0.958	0.958	0.958			0.958	0.000
Corrected Density (for carbon black)	0.952	0.952	0.952			0.952	0.000
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.36	0.36				0.36	
Composition							
% Color	1.36	1.56	1.46			1.46	0.05
% Ash	0	0	0			0.00	0.00
% PP	0					0.00	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	157,895	154,335	148,760	145,727	153,274	151,998	3565
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	4079	4008	4181	4076	4051	4079	71
Break Strain (%)	491	189	14	514	52	252	207
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	36.0	43.8	34.0	42.6	39.6	39.2	4.4
Thermal Stability							
Induction Temp.(C) (ASTM D3350)	256					256	
Induction Time (min) (ASTM D3895)	78					78	

TEST RESULTS
Pipe Containing Recycled HDPE
Lane Pipe Trial
VR1

Material: Plaque from Pipe L4
Sample: 100% VR1

Date: 2-Sep-08
TRI Log #: F7601

	Test Replicate Number						
PARAMETER	1	2	3	4	5	Mean	STD
Density (ASTM D 1505)							
Density (g/cm3)	0.958	0.956	0.957			0.957	0.001
Corrected density (for carbon black)	0.947	0.945	0.946			0.946	0.001
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.12	0.11				0.12	
Composition							
% Color	2.57	2.71	2.34			2.54	0.19
% Ash	0	0	0			0.00	0.00
% PP	0					0.00	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	146,275	152,860	157,589	149,993	144,590	150,261	3132
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	4201	4162	4079	4169	4114	4145	41
Break Strain (%)	65	141	67	129	63	93	32
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	25.5	28.5	26.0	33.7	31.2	29.0	3.2
(ASTM F2136, 600 psi)							
Failure Time (hours)	30.5	27.5	28.2	33.4	26.2	29.2	2.5
Thermal Stability							
Induction Temp.(C) (ASTM D3350)	254					254	
Induction Time (min) (ASTM D3895)	70					70	

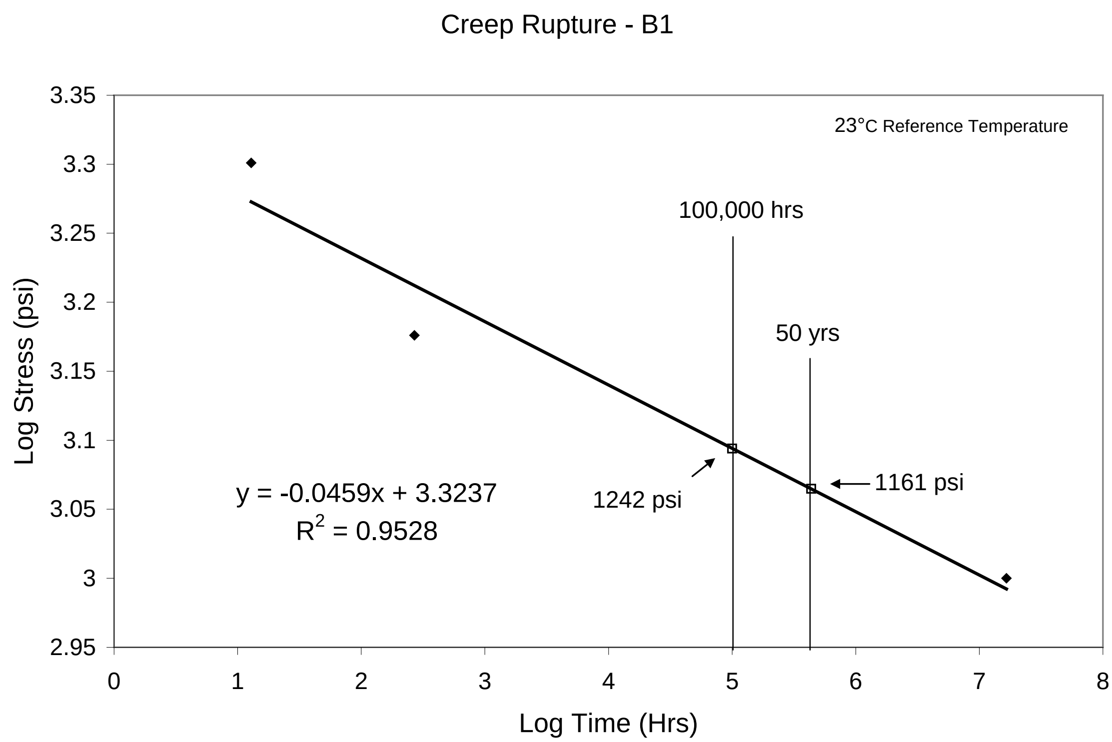
TEST RESULTS
Pipe Containing Recycled HDPE
Lane Pipe Trial
50% VR1 + 20% MDPE + 30% MCR

Material: Plaque from Pipe L5
Sample: 50% VR1 + 20% MD + 30% MCR

Date: 2-Sep-08
TRI Log #: F7601

PARAMETER	Test Replicate Number 1	2	3	4	5	Mean	STD
Density (ASTM D 1505)							
Density (g/cm3)	0.957	0.958	0.957			0.957	0.001
Corrected Density (for carbon black)	0.946	0.947	0.946			0.946	0.001
Melt Flow Index (ASTM D 1238)							
2.16 kg (g/10min)	0.17	0.17				0.17	
Composition							
% Color	2.4	2.6	2.78			2.59	0.09
% Ash	0.26	0.29	0.23			0.26	0.03
% PP	1.8					1.80	
Flexural Modulus (ASTM D3350)							
Flex. Modulus (psi)	146,275	140,800	143,984	138,414	143,616	142,618	2282
Tensile Properties (ASTM D 638)							
Yield Strength (psi)	3995	3901	3919	3902	3892	3922	8
Break Strain (%)	79	74	144	77	167	108	32
Environmental Stress Crack Resistance (ASTM D5397 @ 15% of Yield)							
Failure Time (hours)	37.4	31.8	28.9	39.7	25.9	32.7	4.6
(ASTM F2136, 600 psi)							
Failure Time (hours)	24.6	24.6	27.5	25.1	27.5	25.9	1.3
Thermal Stability							
Induction Temp.(C) (ASTM D3350)	256					256	
Induction Time (min) (ASTM D3895)	77.8					78	

D.12 LONG-TERM CREEP AND CREEP RUPTURE RESULTS


Creep Rupture - B1
23°C Reference Temperature
100,000 hrs
50 yrs
1242 psi
1161 psi
y = -0.0459x + 3.3237
R2 = 0.9528
Log Stress (psi)
Log Time (Hrs)
3.35
3.3
3.25
3.2
3.15
3.1
3.05
3
2.95
0
1
2
3
4
5
6
7
8

Recycled-B1-500psi-05n8.xls

SUMMARY CREEP PARAMETERS:

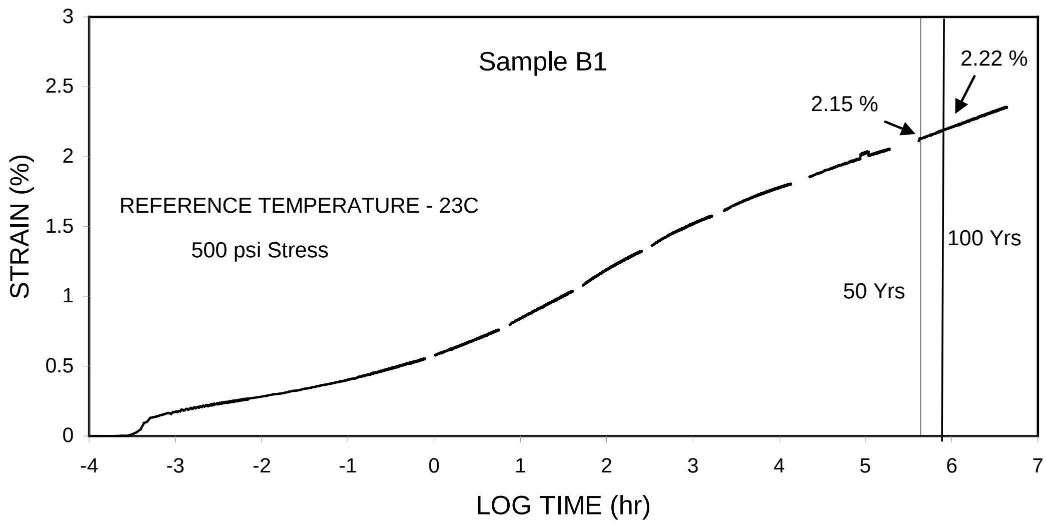
Recycled Resin B1

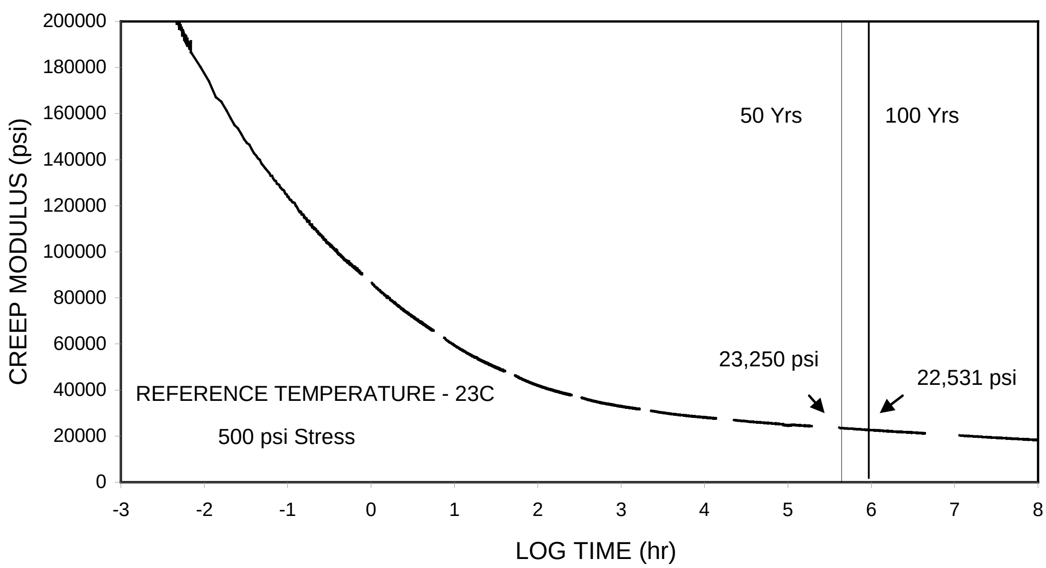
Specimen: rwt-b1-sim500 Test Date: 05-Nov-08 Method: SIM (10^4s, 7C),D638 Type 1 dogbone

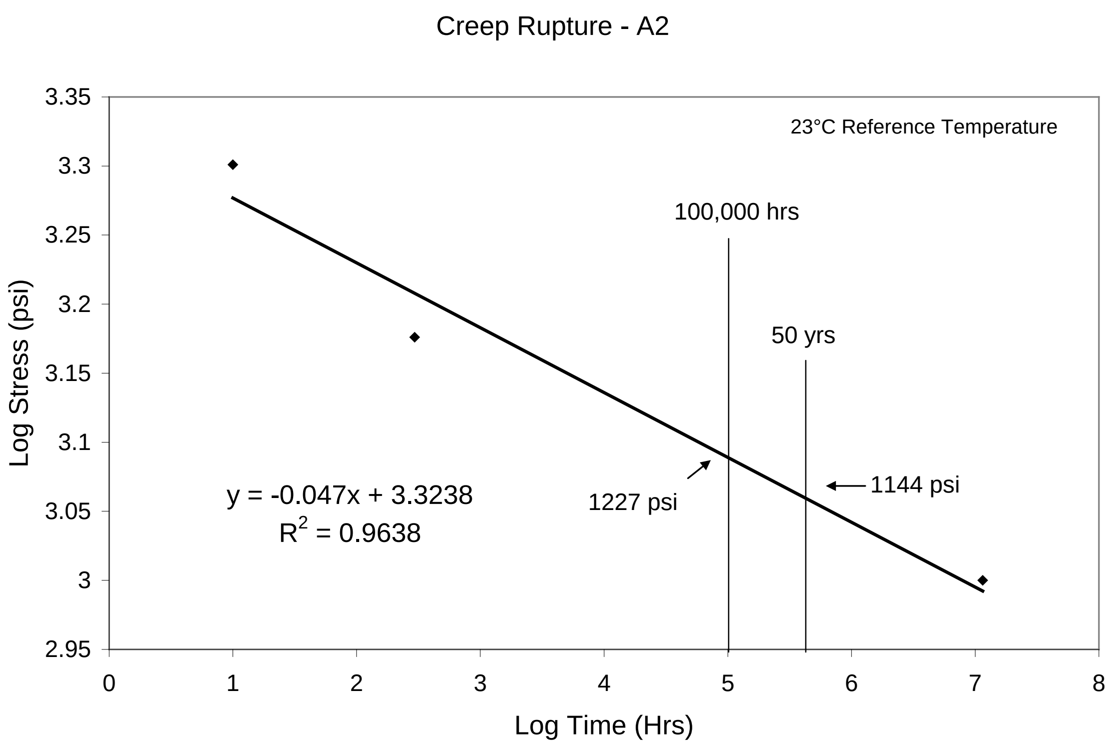
Average Creep Stress: 500.0 psi %UTS: #DIV/0!

Tensile Yield Strength: 0.0 psi Rupture: NO

Dwell Seq	t'	t	$(t-t')_i$	Vshift(%)	$logA_T$	Temp	$logA_T/T$
1	0	0.5	0.5	-	-	19.39	-
2	8400	10017	1617	-0.105	0.7908	26.61	0.1094
3	18400	20007	1607	-0.12	0.8577	33.78	0.1197
4	28100	29997	1897	-0.15	0.7852	40.98	0.1091
5	38200	39987	1787	-0.155	0.8219	48.30	0.1123
6	48600	49977	1377	-0.16	0.9311	55.30	0.1329
7	59200	59967	767	-0.17	1.1699	62.24	0.1687
8	69500	69957	457	-0.2	1.3710	69.27	0.1950
9	79600	79947	347	-0.23	1.4779	76.42	0.2068
10	89700	89937	237	-0.27	1.6392	83.72	0.2245
						AVG	0.1530







Creep Rupture - A2
23°C Reference Temperature
100,000 hrs
50 yrs
1227 psi
1144 psi
y = -0.047x + 3.3238
R^2 = 0.9638
Log Stress (psi)
Log Time (Hrs)
3.35
3.3
3.25
3.2
3.15
3.1
3.05
3
2.95
0
1
2
3
4
5
6
7
8

Recycled-A2-500psi-05n8.xls

SUMMARY CREEP PARAMETERS:

Recycled Resin A2

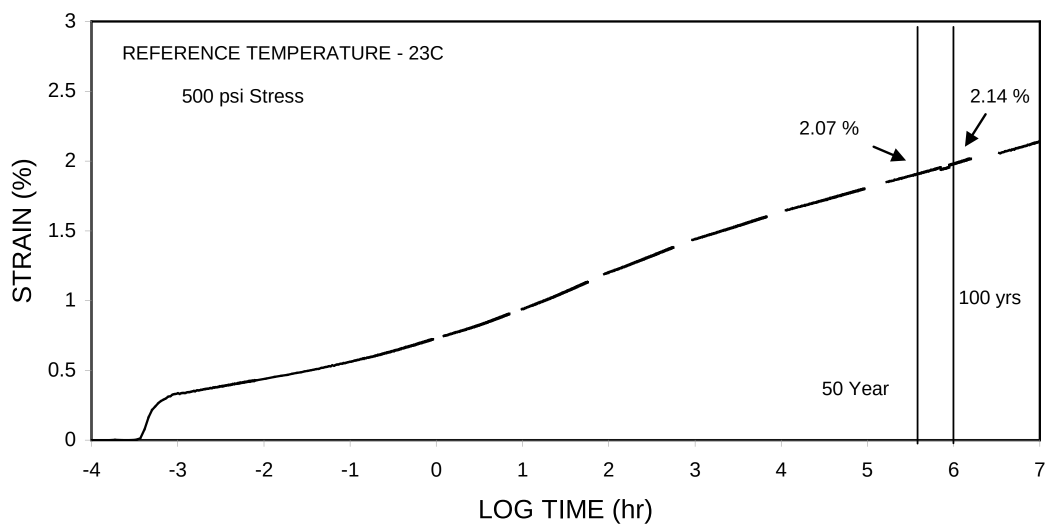
Specimen: rwt-a2-sim500 Test Date: 05-Nov-08 Method: SIM (10^4s, 7C),D638 Type 1 dogbone

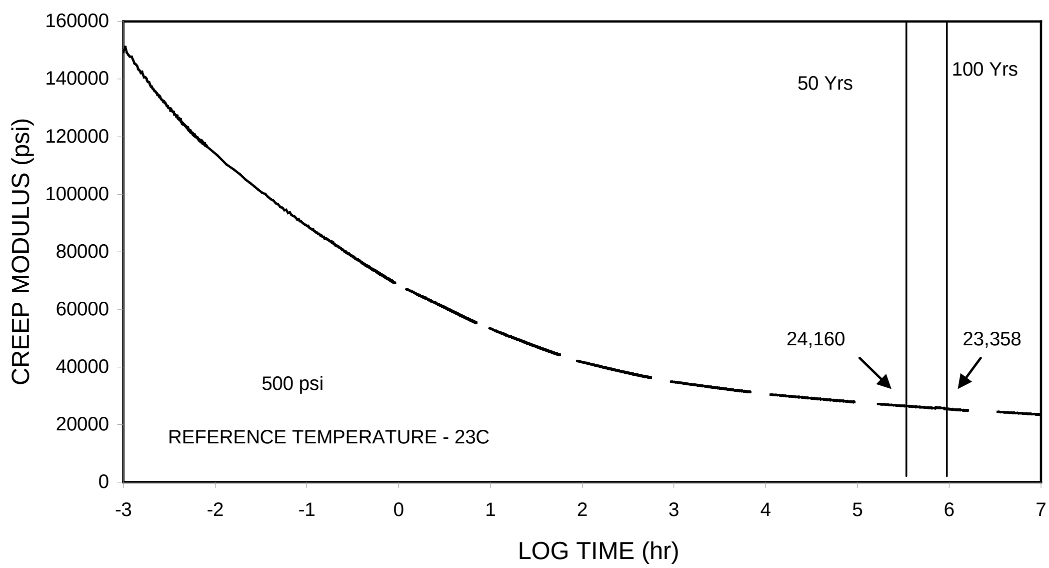
Average Creep Stress: 500.0 psi %UTS: #DIV/0!

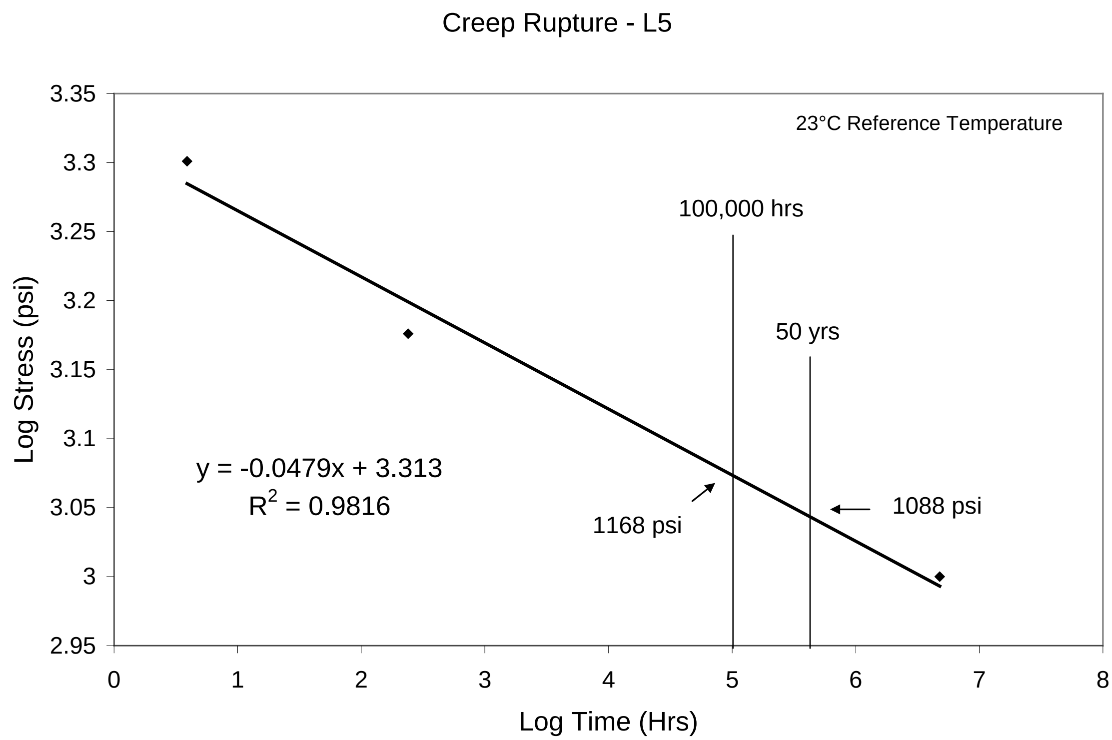
Tensile Yield Strength: 0.0 psi Rupture: NO

Dwell Seq	t'	t	$(t-t')_i$	Vshift(%)	$logA_T$	Temp	$logA_T/T$
1	0	0.5	0.5	-	-	20.09	-
2	8525	10017	1492	-0.108	0.8258	27.07	0.1182
3	18610	20007	1397	-0.15	0.9138	34.22	0.1278
4	28870	29997	1127	-0.15	1.0035	41.42	0.1394
5	39085	39987	902	-0.15	1.0898	48.64	0.1509
6	49190	49977	787	-0.17	1.1402	55.62	0.1633
7	59350	59967	617	-0.2	1.2413	62.64	0.1770
8	69485	69957	472	-0.27	1.3508	69.66	0.1922
9	79595	79947	352	-0.335	1.4723	76.73	0.2084
10	89675	89937	262	-0.44	1.5958	83.83	0.2247
						AVG	0.1668

1.973






Creep Rupture - L5
23°C Reference Temperature
100,000 hrs
50 yrs
y = -0.0479x + 3.313
R2 = 0.9816
1168 psi
1088 psi
Log Stress (psi)
Log Time (Hrs)
3.35
3.3
3.25
3.2
3.15
3.1
3.05
3
2.95
0
1
2
3
4
5
6
7
8

Recycled-L5-500psi-05n8.xls SUMMARY CREEP PARAMETERS:

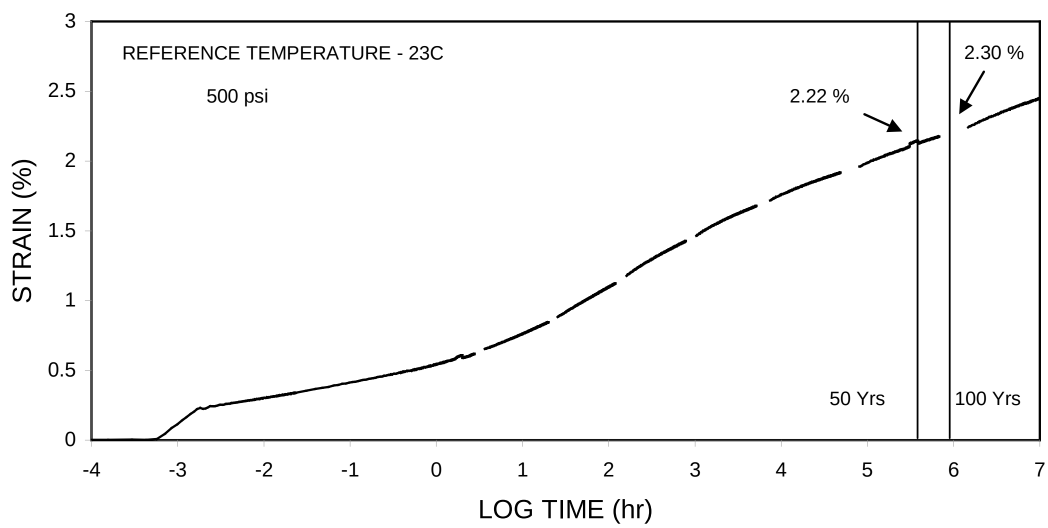
Recycled Resin L5

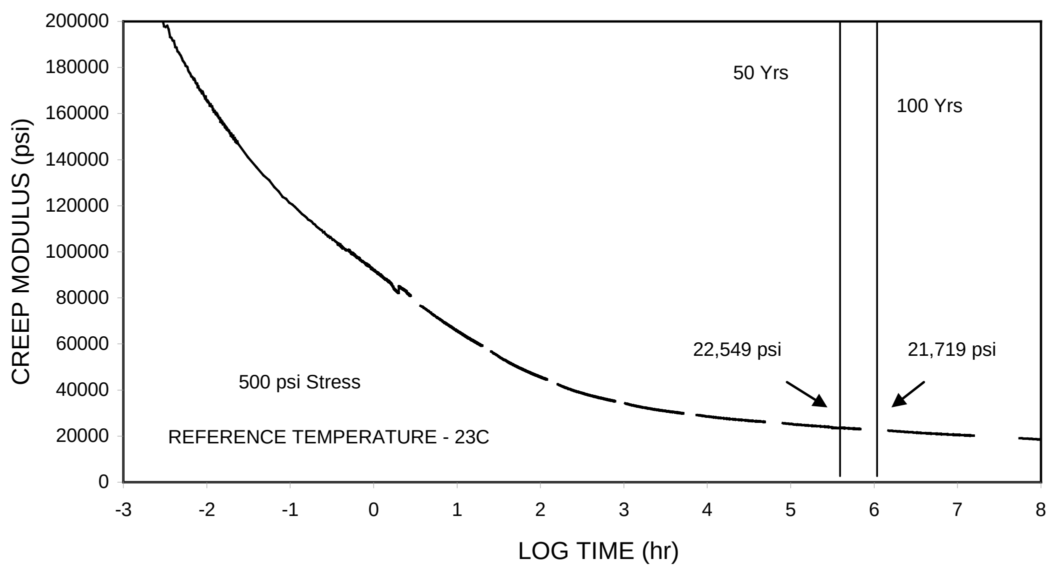
Specimen: rwt-l5-sim500 Test Date: 05-Nov-08 Method: SIM (10^4s, 7C),D638 Type 1 dogbone

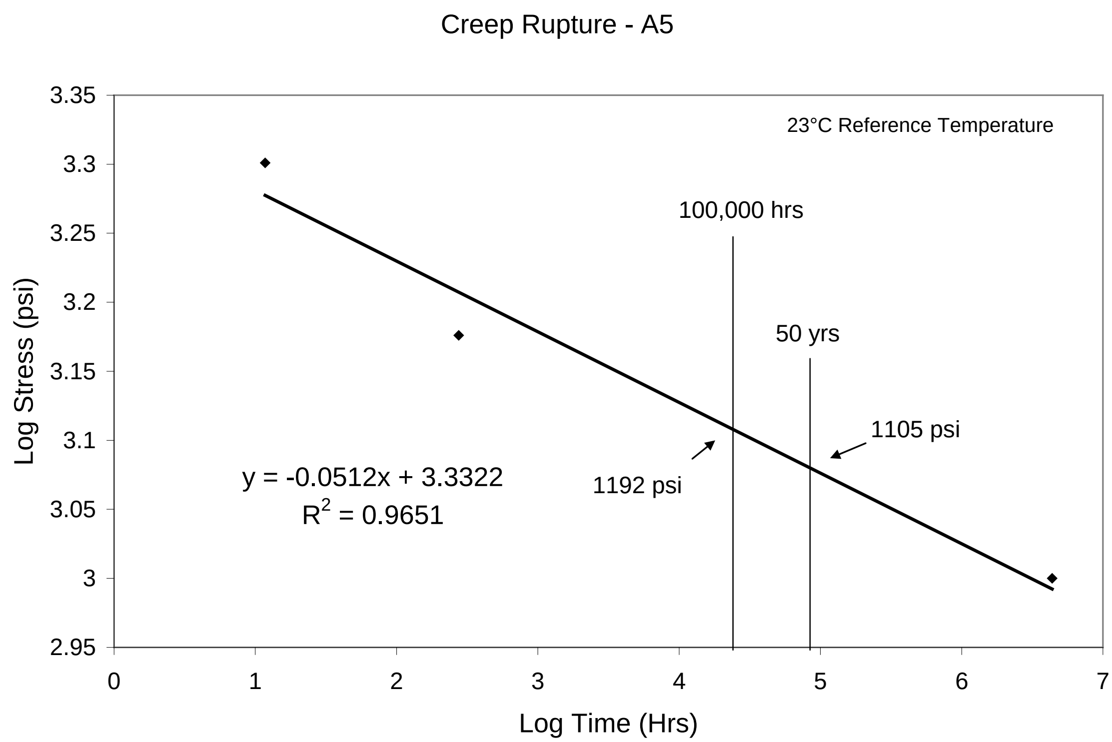
Average Creep Stress: 499.9 psi %UTS: #DIV/0!

Tensile Yield Strength: 0.0 psi Rupture: NO

Dwell Seq	t'	t	$(t-t')_i$	Vshift(%)	$logA_T$	Temp	$logA_T/T$
1	0	0.5	0.5	-	-	20.30	-
2	8400	10017	1617	-0.05	0.7908	26.72	0.1233
3	18000	20007	2007	-0.07	0.7611	33.82	0.1072
4	28200	29997	1797	-0.08	0.8235	41.03	0.1142
5	38200	39987	1787	-0.1	0.8183	48.16	0.1148
6	48800	49977	1177	-0.1	0.9993	54.95	0.1470
7	59200	59967	767	-0.11	1.1622	61.77	0.1705
8	69500	69957	457	-0.11	1.3710	68.54	0.2026
9	79725	79947	222	-0.05	1.6722	75.44	0.2422
10	89700	89937	237	-0.11	1.6339	80.79	0.3054
						AVG	






Creep Rupture - A5
23°C Reference Temperature
100,000 hrs
50 yrs
1105 psi
1192 psi
y = -0.0512x + 3.3322
R2 = 0.9651
Log Stress (psi)
Log Time (Hrs)
3.35
3.3
3.25
3.2
3.15
3.1
3.05
3
2.95
0
1
2
3
4
5
6
7

Recycled-A5-1000psi-31j9.xls

SUMMARY CREEP PARAMETERS:

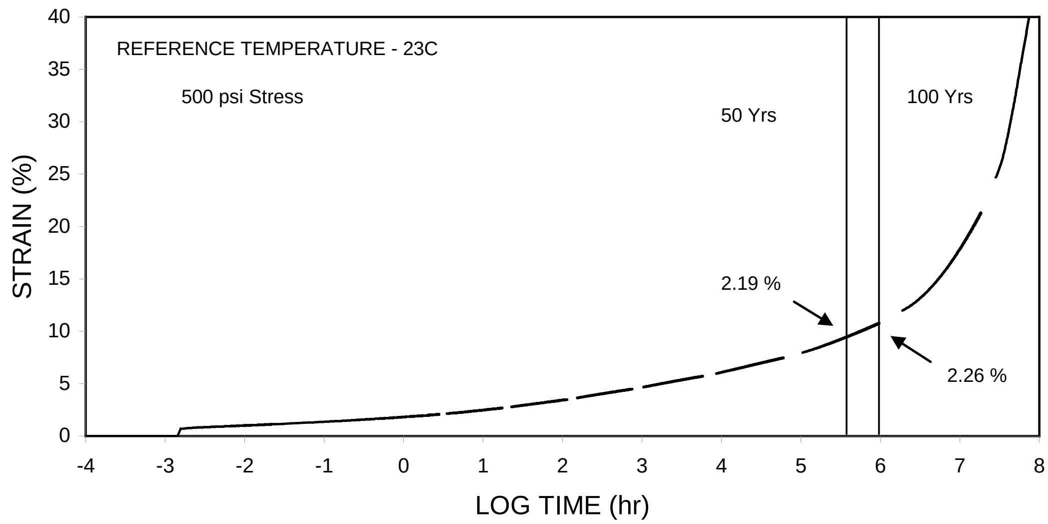
Recycled Resin A5

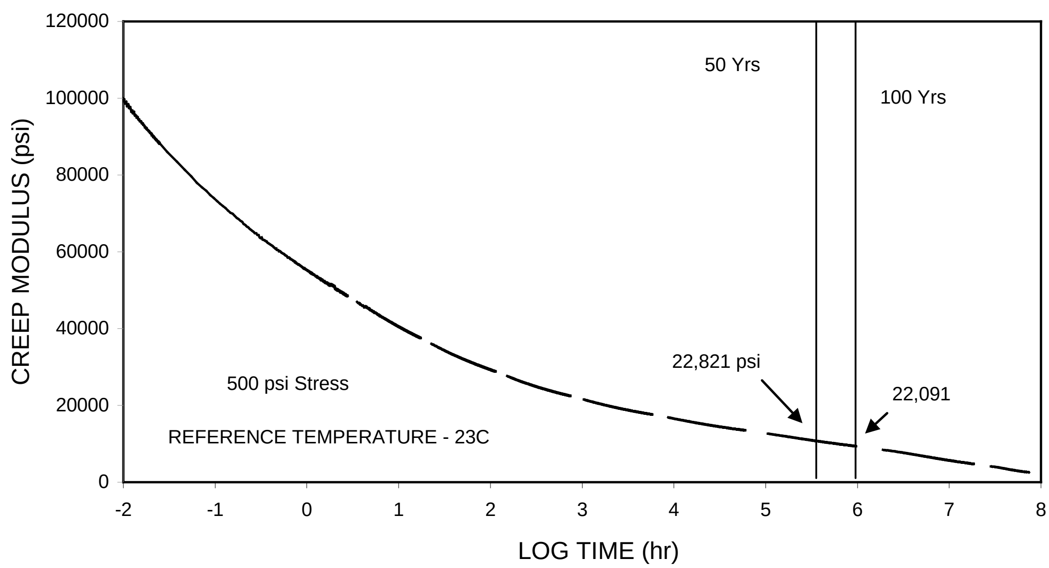
Specimen: rwt-a5-sim1000 Test Date: 31-Jan-09 Method: SIM (10^4s, 7C),D638 Type 1 dogbone

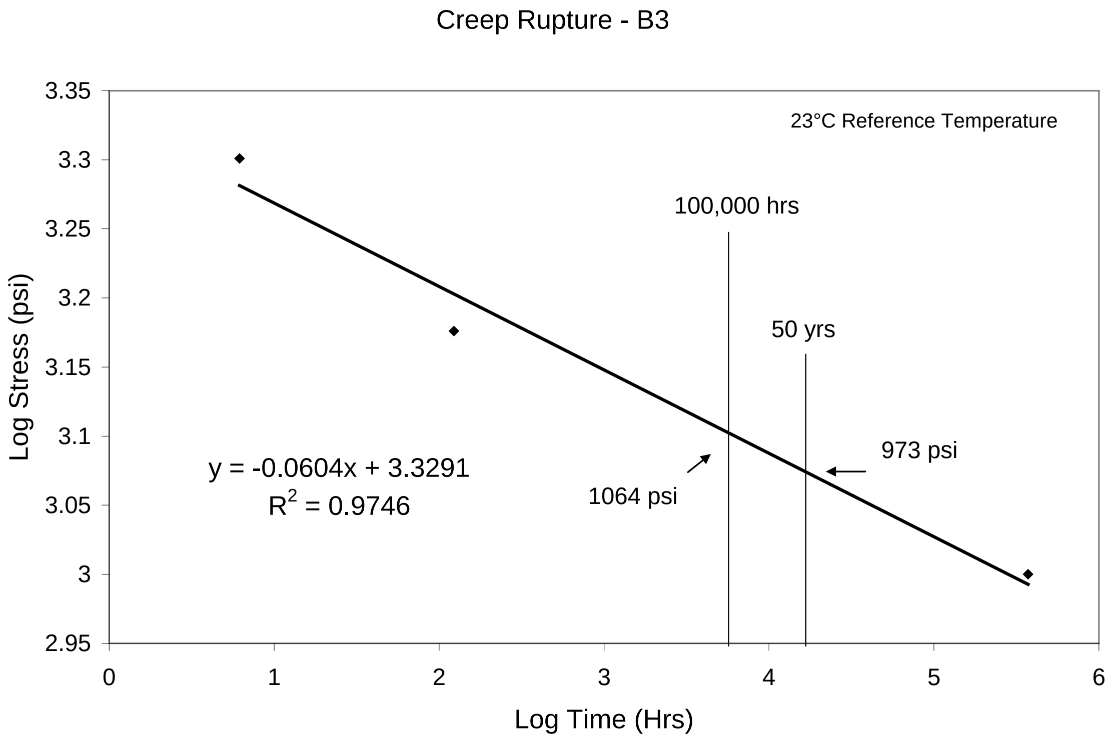
Average Creep Stress: 999.9 psi %UTS: #DIV/0!

Tensile Yield Strength: 0.0 psi Rupture: YES

Dwell Seq	t'	t	$(t-t')_i$	Vshift(%)	$logA_T$	Temp	$logA_T/T$
1	0	0.5	0.5	-	-	20.25	-
2	8100	10008	1908	-0.04	0.7184	26.62	0.1129
3	18200	19998	1798	-0.05	0.8196	33.71	0.1156
4	28200	29988	1788	-0.08	0.8179	40.89	0.1139
5	38500	39978	1478	-0.08	0.9002	48.01	0.1264
6	48900	49968	1068	-0.08	1.0297	54.81	0.1514
7	59300	59958	658	0.06	1.2242	61.64	0.1792
8	69400	69948	548	0.4	1.2871	68.49	0.1879
9	78900	79938	1038	1	1.0053	74.19	0.1765
10							
						AVG	






Creep Rupture - B3
23°C Reference Temperature
100,000 hrs
50 yrs
973 psi
1064 psi
y = -0.0604x + 3.3291
R2 = 0.9746
Log Stress (psi)
Log Time (Hrs)
3.35
3.3
3.25
3.2
3.15
3.1
3.05
3
2.95
0
1
2
3
4
5
6

Recycled-B3-500psi-07n8.xls

SUMMARY CREEP PARAMETERS:

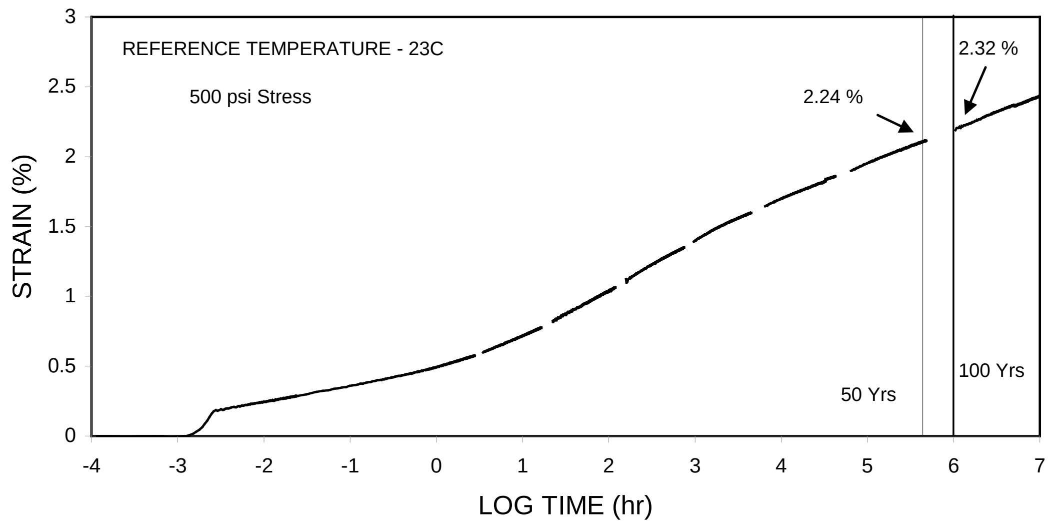
Recycled Resin B1

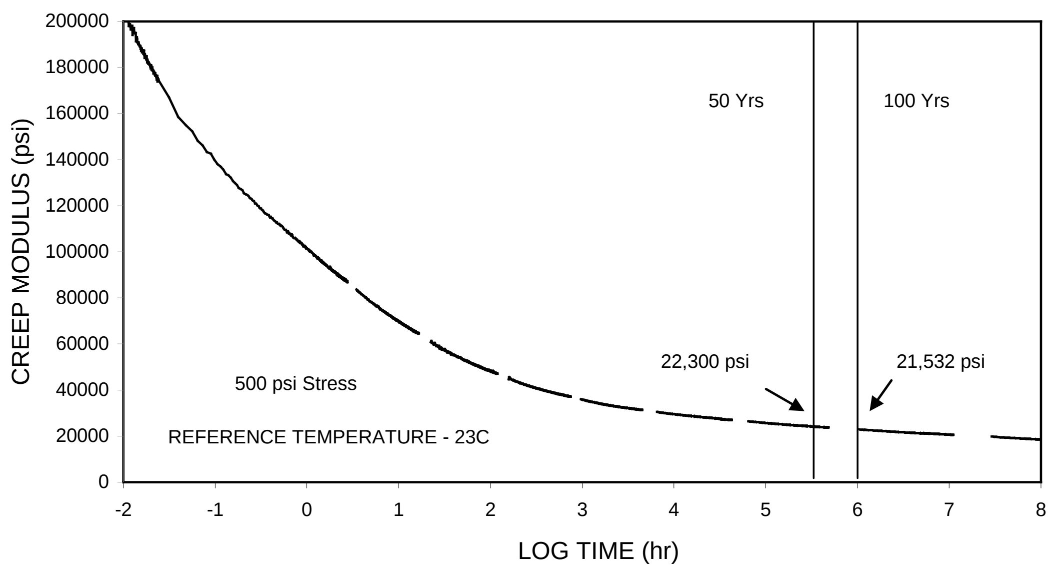
Specimen: rwt-b3-sim500 | Test Date: 07-Nov-08 | Method: SIM (10^4s, 7C),D638 Type 1 dogbone

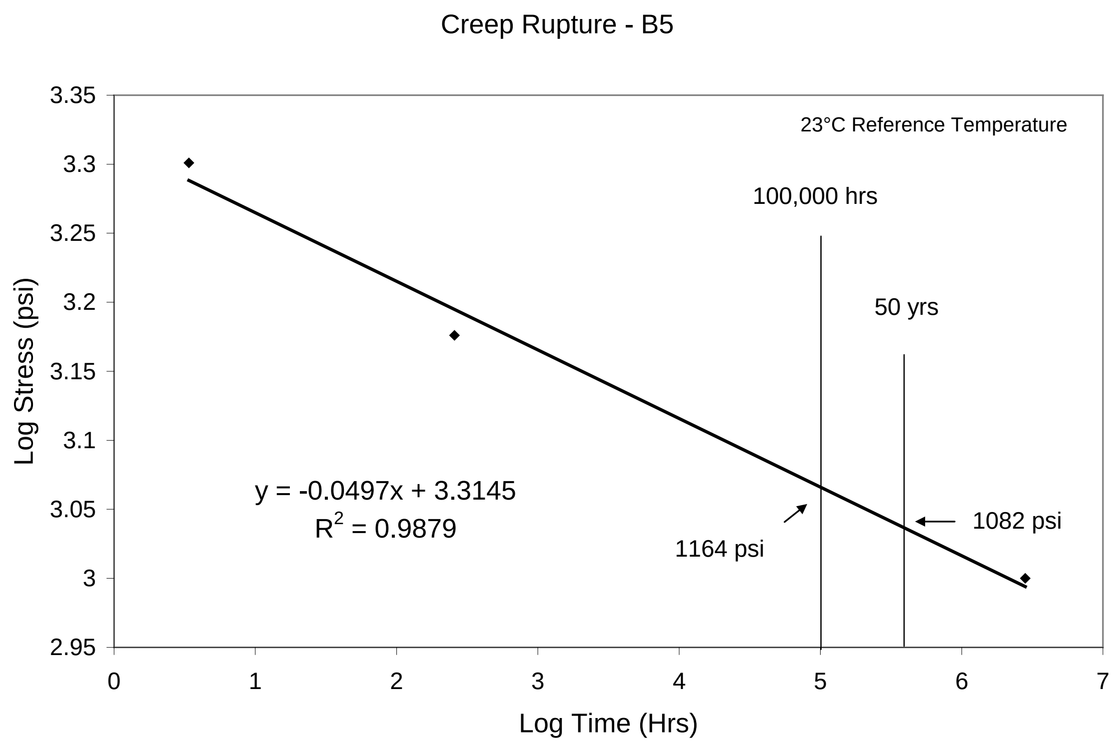
Average Creep Stress: 500.0 psi | %UTS: #DIV/0!

Tensile Yield Strength: 0.0 psi | Rupture: NO

Dwell Seq	t'	t	$(t-t')_i$	Vshift(%)	$logA_T$	Temp	$logA_T/T$
1	0	0.5	0.5	-	-	19.28	-
2	8000	10015	2015	-0.115	0.6950	26.65	0.0944
3	18400	20005	1605	-0.125	0.8727	33.80	0.1220
4	28100	29995	1895	-0.155	0.7855	40.99	0.1093
5	38000	39985	1985	-0.16	0.7761	48.28	0.1064
6	48800	49975	1175	-0.165	1.0070	55.28	0.1439
7	59000	59965	965	-0.2	1.0620	62.31	0.1511
8	69500	69955	455	-0.22	1.3800	69.38	0.1951
9	79600	79945	345	-0.29	1.4793	76.45	0.2092
10	89700	89935	235	-0.35	1.6412	83.72	0.2257
						AVG	






Creep Rupture - B5
23°C Reference Temperature
100,000 hrs
50 yrs
y = -0.0497x + 3.3145
R2 = 0.9879
1164 psi
1082 psi
Log Stress (psi)
Log Time (Hrs)
3.35
3.3
3.25
3.2
3.15
3.1
3.05
3
2.95
0
1
2
3
4
5
6
7

Recycled-B5-500psi-07n8.xls

SUMMARY CREEP PARAMETERS:

Recycled Resin B5

Specimen: rwt-b5-sim500 Test Date: 07-Nov-08 Method: SIM (10^4s, 7C),D638 Type 1 dogbone

Average Creep Stress: 500.1 psi %UTS: #DIV/0!

Tensile Yield Strength: 0.0 psi Rupture: NO

Dwell Seq	t'	t	$(t-t')_i$	Vshift(%)	$logA_T$	Temp	$logA_T/T$
1	0	0.5	0.5	-	-	21.85	-
2	6200	10015	3815	-0.02	0.4178	26.55	0.0889
3	18800	20005	1205	-0.04	1.0580	33.60	0.1500
4	28600	29995	1395	-0.06	0.9032	40.82	0.1250
5	38700	39985	1285	-0.07	0.9462	47.95	0.1327
6	49000	49975	975	-0.08	1.0618	54.83	0.1545
7	59500	59965	465	-0.06	1.3710	61.66	0.2007
8	69700	69955	255	-0.04	1.6108	68.51	0.2350
9	79700	79945	245	-0.06	1.6193	75.58	0.2291
10	89700	89935	235	-0.04	1.6370	80.72	0.3185
						AVG	0.1805