Performance of Corrugated Pipe Manufactured with Recycled Polyethylene Content

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NATIONAL COOPERATIVE HIGHWAY RESEARCH PROGRAM

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Performance of Corrugated Pipe Manufactured with Recycled Polyethylene Content

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FOREWORD

By Edward T. Harrigan
Staff Officer
Transportation Research Board

This report provides potential specifications for corrugated drainage pipe manufactured with recycled high-density polyethylene (HDPE). The report details the research performed and includes proposed draft specifications for recycled HDPE, formulations of virgin and recycled HDPE, and drainage pipe containing recycled HDPE. Thus, the report will be of immediate interest to materials engineers and bridge and structural design engineers in state highway agencies, as well as to thermoplastic pipe suppliers.

The corrugated drainage pipe market in the United States consumes in excess of a billion pounds of virgin HDPE annually. Previous research has demonstrated the feasibility of blending virgin HDPE with recycled HDPE (millions of pounds of which are generated each year) to manufacture drainage pipe. To produce pipe of satisfactory quality, HDPE blends must consistently maintain critical material properties that affect pipe durability, such as strength and structural properties, density, melt index, environmental stress-crack resistance, and thermal stability, at the levels required by relevant AASHTO specifications.

The objective of this research was to develop specifications for (1) recycled HDPE intended for use in the manufacture of corrugated drainage pipe and (2) corrugated pipe manufactured from blends of virgin and recycled HDPE. To accomplish this objective, the research examined the structural and service capabilities of corrugated drainage pipe manufactured with recycled HDPE content in typical transportation applications and evaluated the applicability of existing design and performance standards for corrugated drainage pipe manufactured with virgin HDPE to that containing HDPE.

The research was conducted by TRI/Environmental, Inc. of Austin, Texas in three phases. In the first phase, key material properties of a wide range of post-consumer recycled (PCR) and post-industrial recycled (PIR) HDPEs were compared to those of virgin HDPEs typically used to produce corrugated drainage pipe. The results showed that PCR, mixed-color, reprocessed HDPE was the preferable recycled material because of its wide availability and good consistency. In the second phase, 67 different blends of PCR mixed-color, reprocessed HDPEs and virgin HDPEs were prepared and tested to determine their suitability for production of corrugated drainage pipe of satisfactory quality and performance. In the final phase, several promising blends of recycled and virgin HDPEs were used to manufacture 15 12-in.-diameter pipe samples at three different manufacturing plants. The short-term material properties and long-term durability of these pipe samples were tested and compared to control pipe manufactured under the same conditions from virgin HDPE.

Overall, the research demonstrated that (1) pipe made with a 50% or greater content of recycled HDPE can provide adequate short- and long-term properties and a service life comparable to pipe manufactured with 100% virgin HDPE and (2) the best blends are those that combine recycled HDPE with virgin HDPE lower in density and higher in stress-crack resistance than those typically used in the production of corrugated drainage pipe.

The report fully documents the research leading to the development of draft specifications for recycled HDPE, formulations of virgin and recycled HDPE, and two sizes of corrugated drainage pipe proposed for review and possible adoption by the AASHTO Highway Subcommittee on Materials. Besides the full text of the contractor's final report, this report contains six printed appendixes:

APPENDIX A: Procedures and Test Methods
APPENDIX E: Proposed Specification for Reprocessed, Mixed-Color, PCR-HDPE
APPENDIX F: Proposed Specification for Recycled Containing HDPE Resin Formulations for Corrugated Pipe Made to AASHTO Specification M252-Recycled
APPENDIX G: Proposed Specification for Recycled Containing HDPE Resin Formulations for Corrugated Pipe Made to AASHTO Specification M294-Recycled
APPENDIX H: Proposed Specification for Corrugated Polyethylene Drainage Pipe Containing Recycled Polyethylene, 75- to 250-mm Diameter
APPENDIX I: Proposed Specification for Corrugated Polyethylene Drainage Pipe Containing Recycled Polyethylene, 300- to 1500-mm Diameter

In addition, three appendixes are available to download from the NCHRP Project 04-32 web page at http://apps.trb.org/cmsfeed/TRBNetProjectDisplay.asp?ProjectID=865:

APPENDIX B: Recycled Polyethylene Resins
APPENDIX C: Recycled-Resin Blends
APPENDIX D: Pipe Containing Recycled HDPE

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H-1 **Appendix H** Proposed Draft Standard Specification for Corrugated Polyethylene Drainage Pipe Containing Recycled Polyethylene, 75- to 250-mm Diameter
I-1 **Appendix I** Proposed Draft Standard Specification for Corrugated Polyethylene Drainage Pipe Containing Recycled Polyethylene, 300- to 1,500-mm Diameter

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Note: Many of the photographs, figures, and tables in this report have been converted from color to grayscale for printing. The electronic version of the report (posted on the Web at www.trb.org) retains the color versions.

AUTHOR ACKNOWLEDGMENTS

There were many organizations and people who contributed to this project. Input was requested and received from virgin resin suppliers, recyclers, trade associations, and corrugated pipe manufacturers. The majority of the laboratory testing was performed by David Cuttino of TRI. The evaluation of designing with recycled-containing HDPE was performed by Sarah L. Gassman of the University of South Carolina. She wrote the section called "Designing Pipe with Recycled Content."

The companies that contributed samples to the project were:

Berou International Inc.
Blue Ridge Plastics LLC
ChevronPhillips Chemical Co.
Clean Tech, Inc.
Custom Polymers Inc.
Entropex, Inc.
Envision Plastics
Ineos Olefins and Polymers USA
KW Plastics, Recycling Division
LyondellBasell Advanced Polyolefins USA, Inc.
Polychem Products Ltd
Recyc RPM
Solplast
Trademark Plastics Corporation

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Blue Diamond Industries
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SUMMARY

Performance of Corrugated Pipe Manufactured with Recycled Polyethylene Content

The corrugated drainage pipe market in the United States consumes in excess of a billion pounds of virgin high-density polyethylene (HDPE) annually. At the same time, millions of pounds of recyclable HDPE are generated each year. Industry-funded research has demonstrated the feasibility of blending recycled and virgin HDPE to manufacture corrugated drainage pipe. The working hypothesis is that good quality pipe can be manufactured from such blends if critical material properties that affect pipe performance and durability, such as strength and structural properties, density, melt index, environmental stress-crack resistance, and thermal stability, are consistently maintained at specified levels in the blends.

At present, AASHTO specifications do not permit the use of recycled HDPE in the production of corrugated drainage pipe. Research was needed to determine if there were blends of virgin and recycled HDPE that met the current requirements of AASHTO Specifications M252 and M294. Additionally, the long-term performance of pipe made with these blends had to be assessed and specifications were needed to ensure that quality products were made.

The objectives of this study were to

1. Determine the availability, properties, and consistency of recycled (HDPE) intended for use in the manufacture of corrugated drainage pipe.
2. Prepare blends of recycled HDPE with virgin corrugated pipe resins and determine the properties of the blends.
3. Select candidate resin formulations containing recycled HDPE and manufacture 12 in. diameter, dual wall, corrugated pipe from the blends.
4. Determine the short- and long-term properties of the blends and evaluate if the currently used design standards can be used for recycled content–containing pipe.
5. Prepare specifications and standard test methods for resins and pipe that contain recycled HDPE.

The study was divided into three main parts:

1. Recycled polyethylene resins,
2. Blends with recycled and virgin pipe resins,
3. Pipe made from blends containing recycled PE.

There were 25 samples of recycled PE obtained for the first part of the study. These included 22 post-consumer recycled (PCR) and three post-industrial recycled (PIR), and both mixed-color and natural PCR. The results demonstrated that post-consumer, mixed-color, reprocessed HDPE was the best available recycle material available because of its availability and consistency. PIR PE could be found with superior properties to PCR, but it is not consistently available. Natural PCR (milk bottles) is consistent, but it is also more expensive than mixed color and is more brittle. Mixed-color PCR (PCR-MCR) resins were favored because there are about a dozen suppliers, the products are more consistent, and there is a trade association dedicated to the production of high quality materials. The resins have good strength properties, but poor stress-crack resistance and contain contaminants that can limit their performance over the long term.

The second part of the study included the preparation and testing of 67 different blends. These were all characterized by a variety of tests. Over 700 tests were performed. The results demonstrated that some properties [density, percentage polypropylene, percentage black, percentage ash, oxidative induction time (OIT), and tensile] varied in a linear manner with recycled content. Other properties (melt index, stress-crack resistance) varied exponentially with recycled content. This is important because it allows one to estimate the properties of a blend from the properties of the components.

The final part of the study involved the manufacture of 15, 12-in.-diameter pipe samples at three different manufacturing plants. Each plant made five pipe samples, and all three plants made a sample with 100% virgin HDPE and a sample with 30% recycled HDPE for controls. The short-term properties were measured on all 15 samples and long-term durability studies were performed on six candidate blends. The results showed that it is difficult to make blends better than virgin if one simply adds recycled resin to virgin resin. In fact, the results showed that the study was limited by its focus on blending with virgin corrugated pipe resins. Much better properties can be obtained by blending recycled resins with virgin resins lower in density and higher in stress-crack resistance than the virgin pipe resins. It seems clear from the results that pipe made with significant amounts (>50%) of recycled PE can be developed with adequate short-term properties and long-term properties that suggest that the pipe will last 50 to 100 years.

These results led to the development of five proposed individual product specifications. These included a specification for mixed-color PCR resins intended to be used in formulations to make AASHTO M252 or M294 pipe. Two separate specifications for fully formulated resins intended for pipe for either M252 or M294 applications. And two specifications for pipe that are to be used in M252 or M294 applications.

CHAPTER 1

Introduction

The corrugated drainage pipe market in the United States consumes in excess of a billion pounds of virgin high-density polyethylene (HDPE) annually. At the same time, millions of pounds of recyclable HDPE are generated each year. Industry-funded research has demonstrated the feasibility of blending recycled and virgin HDPE to manufacture corrugated drainage pipe. The working hypothesis is that good quality pipe can be manufactured from such blends if critical material properties that affect pipe performance and durability, such as strength and structural properties, density, melt index, environmental stress-crack resistance, and thermal stability are consistently maintained at specified levels in the blends.

At present, AASHTO specifications do not permit the use of recycled HDPE in the production of corrugated drainage pipe. Research was needed to

1. Determine if there are blends of virgin and recycled HDPE that meet the current material property requirements specified by AASHTO and defined in ASTM D3350;
2. Perform post-production tests on pipe manufactured with blends of virgin and recycled HDPE to assure its long-term stress-crack resistance and antioxidant effectiveness are equivalent to that of pipe made from virgin HDPE, as currently specified in AASHTO M294 or M252;
3. Identify whether contaminants present in recycled HDPE will adversely affect the long-term service life of finished pipe;
4. To the extent possible, evaluate the performance of existing installations of corrugated drainage pipe produced from blends of virgin and recycled HDPE; and
5. If warranted, develop specifications and test methods to support the use of recycled HDPE in the manufacture of corrugated drainage pipe.

The objectives of this study were to

1. Determine the availability, properties, and consistency of recycled HDPE intended for use in the manufacture of corrugated drainage pipe;
2. Prepare blends of recycled HDPE with virgin corrugated pipe resins and determine the properties of the blends;
3. Select candidate resin formulations containing recycled HDPE and manufacture 12-in.-diameter, dual wall, corrugated pipe from the blends;
4. Determine the short- and long-term properties of the blends and evaluate if the currently used design standards can be used for recycled-containing pipe; and
5. Prepare specifications and standard test methods for resins and pipe that contain recycled HDPE.

CHAPTER 2

Research Approach

The approach taken to meet the objectives mentioned in chapter 1 was to divide the project into three distinct phases. These phases were Recycled Resins, Recycled-resin Blends, and Pipe Made from Recycled-resin Blends.

Phase 1—Recycled PE Resins

The purpose of Phase 1 was to determine which types of recycled PE are available, where these materials can be obtained, and what their properties are. The approach was to collect samples of recycled HDPE, determine what their typical properties were, study the effects of some common contaminants, and develop test methods to characterize the materials. The results of this phase of the study allowed the assessment of the best kind of recycled PE for use in corrugated drainage pipe.

Phase 2—Recycled Resin Blends

The purpose of Phase 2 was to determine how the addition of recycled HDPE would affect the properties of Canadian Petroleum Products Institute (CPPI)-certified resins and to select specific blends for trial corrugated pipe manufacturing. The approach was to prepare blends of virgin pipe resins that contained different types and amounts of recycled HDPE. Once the blends were made, key relationships were developed that allowed one to predict the properties of blends so that optimized formulations could be used in the next phase to make actual pipe.

Phase 3—Pipe made from Recycled-Resin Blends

The purpose of this phase of the project was to manufacture dual wall corrugated drainage pipe out of resin formulations containing recycled HDPE. Once the pipe was made, the short-term properties were measured and compared with the requirements of AASHTO M294. Additionally, some longer-term stress-crack tests were performed. And, finally, some candidate pipe samples were evaluated for their long-term creep strength, creep modulus, and stress-crack resistance.

Short-Term Properties

The purpose of short-term tests is to characterize a resin or pipe enough to feel confident that the properties are consistent from lot-to-lot. This is especially important for recycled resins, which are known to be variable. For example, in post-consumer recycled (PCR) HDPE the percentage of milk bottles mixed in with colored bottles will change. Since these two types of HDPE have different properties, the properties of the recycled product will change. Short-term tests can reveal these differences in properties.

Another use for short-term properties is to control contamination. For example, the amounts of particulate matter can be measured by burning off the polymers and carbon black and measuring the ash content. The control of contaminants can also be achieved through mechanical properties. When a tensile test is performed, the test specimen will always break at a flaw. Samples with more potential flaws (particles, unblended polymers, gels) will break at lower strains. One can therefore get an idea about the level and size of contaminants by measuring the strain at break.

And finally, the results of some short-term tests can offer a bit of information about the long-term serviceability of the material. When the flexural modulus or tensile yield stress is measured, one gets a feeling for how the material might respond to stress. Stress-crack tests, like the notched, constant tensile load (NCTL) (ASTM D5397) or notched, constant ligament-stress (NCLS) (ASTM F2136) can give an idea of the relative stress-crack resistance between materials. These are important for use with recycled materials because the particulates and other polymers may promote crack initiation or growth. And, when one measures the oxidative-induction time (OIT) (ASTM D-3895) or the oxidative-induction temperature (OITemp) (ASTM D3350), the presence of stabilizers can be detected, offering some assurance that stabilizers are present (note that the OIT or OITemp tests do not predict long-term performance).

The short-term properties used for this project included:

1. Density—basic property of PE;
2. Melt Index (MI)—basic PE property relating to molecular weight;
3. High Load Melt Index (HLMI)—ratio of 2 MI tests relates to molecular weight distribution;
4. Percentage Color—organic fillers like colorants and carbon black;
5. Percentage Ash—inorganic fillers and contaminants;
6. Differential Scanning Calorimetry (DSC)—detects presence of other semicrystalline polymers like PE;
7. Flexural Modulus—indicator of stiffness;
8. Tensile Yield Strength;
9. Strain at Break—sensitive to contaminants;
10. Notched, Stress-Crack Test—determines relative crack initiation and growth;
11. Un-Notched, Stress-Crack Test—sensitive to contaminants that may initiate cracks; and
12. OIT and OITemp—indicators of stabilization.

Long-Term Properties

There is absolutely no doubt that the most important part of this study is to generate reliable data concerning the long-term performance of pipe made with recycled PE content. The approach for this project was to use the solid wall pipe industries' practices as a model for the development of tests for use with corrugated pipe resins.

Service Lifetime of PE

The long-term service lifetime of HDPE is often presented in a graph like the one in Figure 1. An understanding of this graph is necessary to develop test methods to characterize the long-term behavior of PE.

It shows three distinct stages of aging. The first stage is the likely service lifetime for materials placed under significant loads (>30% of yield). Over time, because of the time-dependent process of creep, the material will fail by yielding or stretching in a ductile manner. The second stage 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 somewhat dependent on the material's yield strength, the service temperature, and the stress. Long-term tests for yield strength 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 sometimes used to accelerate the process and the results are 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 PE reinforcement products for civil engineering applications. The end of

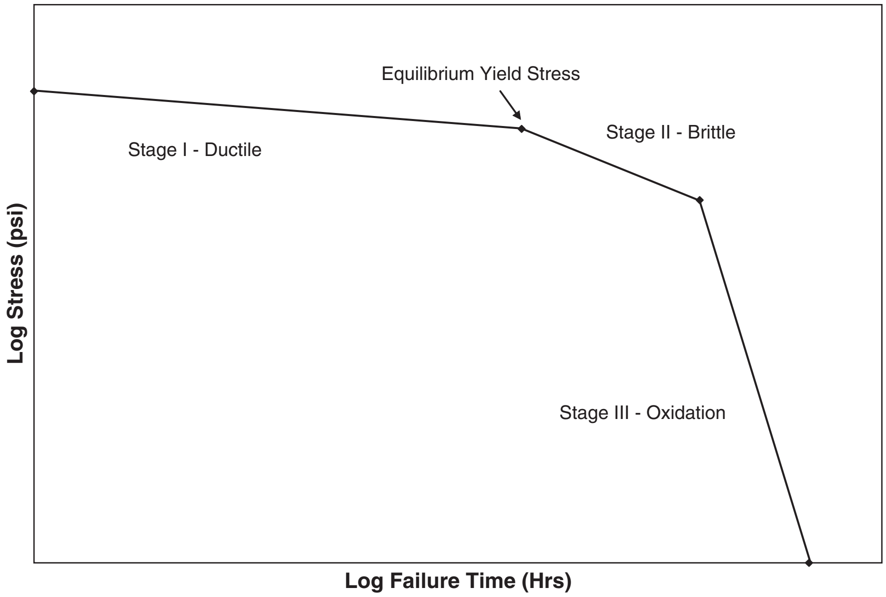

Figure 1. Hypothetical service lifetime for HDPE.

this stage is characterized by the equilibrium tensile strength. Any service stresses lower than the equilibrium yield stress will not cause a ductile failure during the service lifetime.

The second stage 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 predict 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 within 100,000 h. Similar tests for corrugated pipe have been presented, including the Federal Institute for Materials Research and Testing (BAM) Test (*1, 2*), the Florida Department of Transportation (FL-DOT) junction test (*3*), the ring stress-crack test (*4*), and the BAM–FL-DOT–Fathead (BFF) Test (*5*).

It should be clearly stated that both Stage I and Stage II service-lifetime plots are necessary to determine which process will limit the service lifetime of the pipe. If the point where the two lines cross on the time scale is greater than the expected service lifetime, the most important failure mode is ductile. If the crossover point on the time scale is less than the expected service lifetime, then stress cracking is the life-limiting failure mode. In the early days of pressure pipe, most of the failures occurred by slow crack growth. But, as the stress-crack resistance of the resins got better and better, the primary failure mode during service became ductile.

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 that contains the long-term antioxidants and/or light stabilizers. Oxidation is largely a nonissue for properly stabilized resins. However, there should be some requirement for an OIT value or a specified minimum additive package placed in the final specification for resins containing recycled PE. An OIT of 50 minutes should suffice and there are commercial additive packages that 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 pipelines. 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 h (1.14 years). These points are then plotted as Log Stress vs. Log Time and the resulting line extrapolated to 100,000 h (11.4 years). The 100,000 h stress is called the 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 2.

This shows a good example of a situation where only Stage I failures are observed during the test. The ASTM standard also accounts for Stage II or Brittle Failure through slow crack

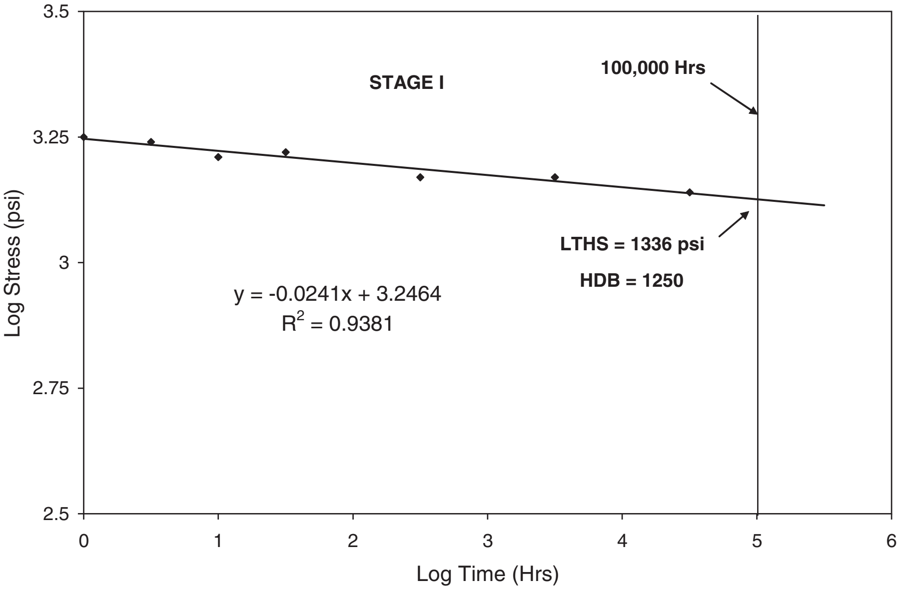

Figure 2. Determination of the hydrostatic design basis (HDB).

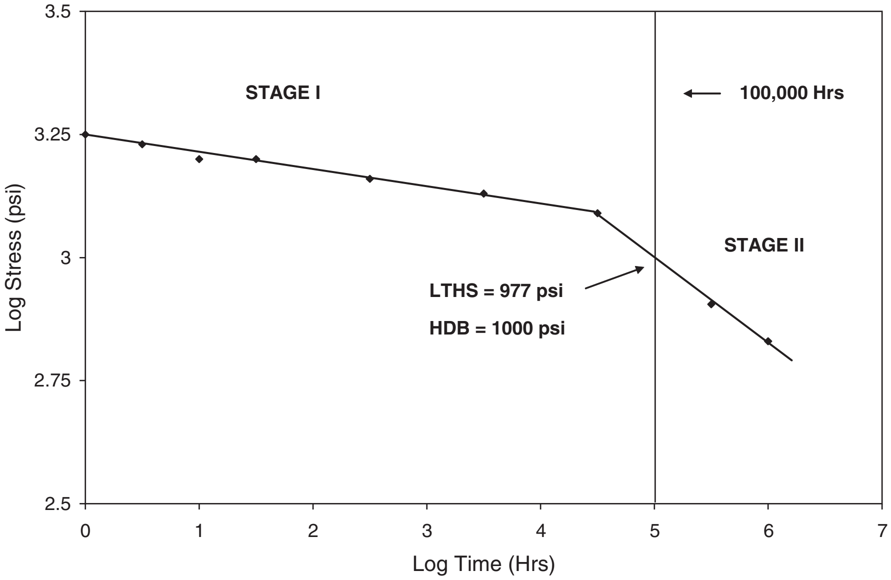

Figure 3. Determination of the HDB when Stage II is involved.

growth. This is shown in Figure 3, 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 h.

Figure 4 shows Stage I and Stage II for examples of both a HDB 1250 and HDB 1000 resin.

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 to Stage II) occurs later in time. This is why just specifying the yield stress or flexural modulus is not enough. One needs to determine the slope of the line in Figure 2 to get a good understanding of long-term behavior under stress.

Once the HDB category is found, the hydrostatic design stress (HDS) is found by applying a design factor (DF) to the HDB. The DF is similar to a factor of safety, where reductions are estimated for installation damage, lot-to-lot variability, and so forth. 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.

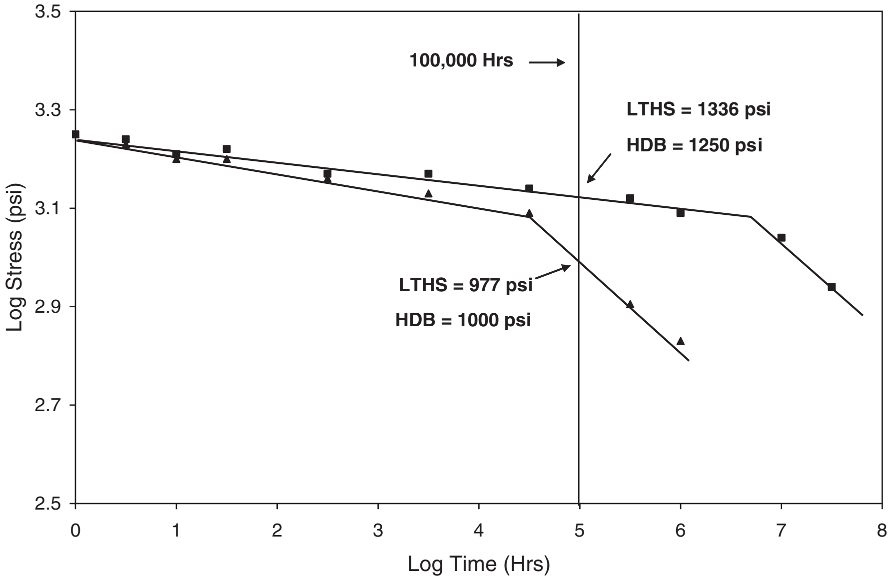

Figure 4. A comparison between the examples shown in Figures 2 and 3.

One of the advantages of hydrostatic testing on plastic pipe is that 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 and 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).

Along with the long-term strength (Stage I), AASHTO requires long-term creep modulus and long-term creep strain as part of the design for corrugated pipe. The approach to determine both the creep and creep rupture properties of pipe containing recycled PE is through the SIM accelerated test.

The SIM for Predicting Creep and Creep Rupture (Stage I) Properties

The SIM is a special form of TTS that has been used to extrapolate short-term creep results (~24 h) 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 (*6, 7*). 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 (*8*). It has also been used by others on other PET fibers, Kevlar, and polyethylene naphthanate (PEN)(9).

It has also been used in these laboratories to examine polypropylene (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 h conventional creep tests is shown in Figure 5.

The main difference between PET and HDPE is their respective temperature dependencies at temperatures from ambient to 80°C. 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 as the difference in creep rates at two different temperatures. A comparison for the two materials is shown in Figure 6.

TTS has been used for decades and it is the basis for the validation procedures for PE pipe materials in ASTM D2837 and Plastics Pipe Institute (PPI) Technical Report TR-3 (*10*). TTS can be 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 specimens. Two examples of TTS are the Rate Process Method and Popelar Bi-Directional Shifting Method.

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.

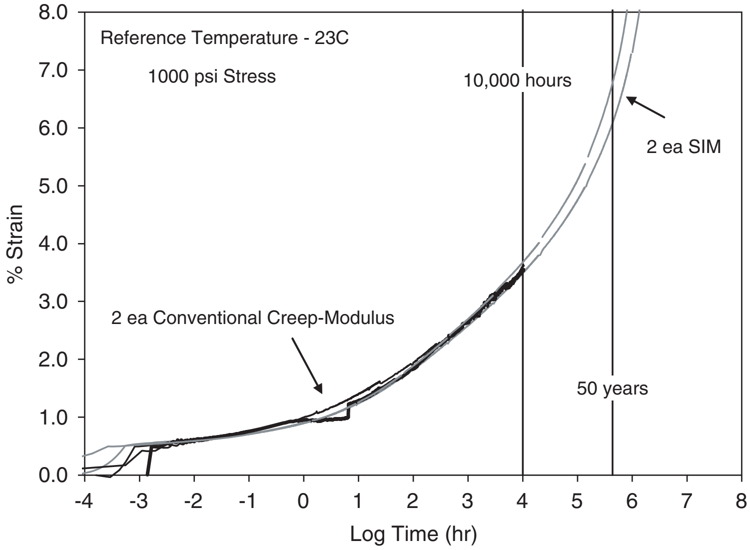

Figure 5. Comparison between conventional creep and SIM for a PP storm chamber under 1,000 psi of stress.

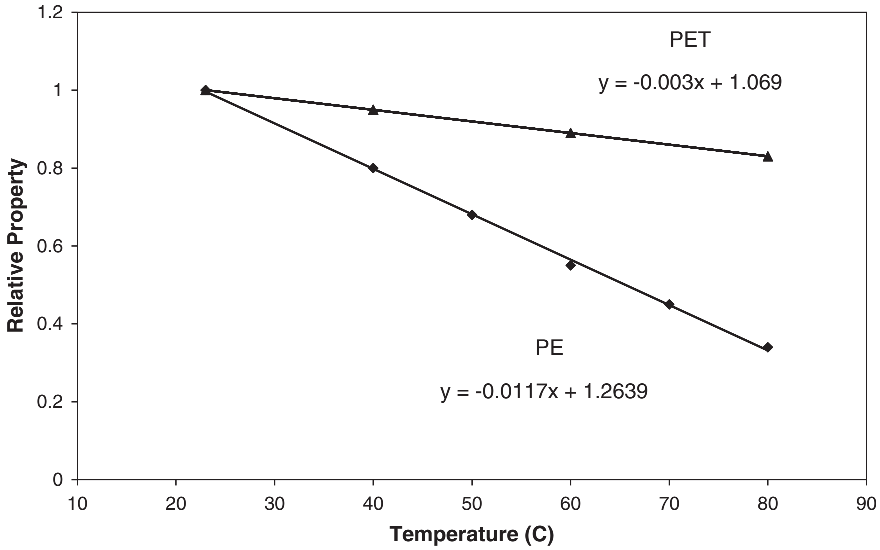

Figure 6. Temperature dependence of PET and PE.

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 h).

The raw, unshifted data are shown in Figure 7.

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's also easy to see that at each successive temperature step, the creep rate increases. This is due to the increased temperature, but also because PE gets softer as the temperature rises. So, in reality, there is a double acceleration.

The next step in the analysis is to determine what is referred to as the virtual starting time (t′) for each step above the first one. 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 rescale the individual creep curves and get them all on a common time scale.

The t′ is found by plotting creep modulus vs. log time for the end of one step and the beginning of the next step. Then, one can adjust the t′ iteratively until the slopes of the two curves are parallel. A vertical shift is also added at this time

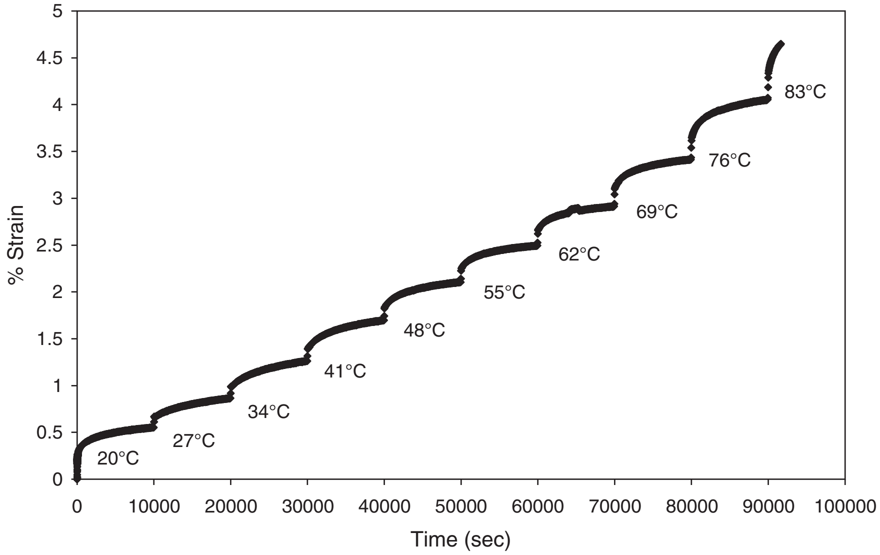

Figure 7. Raw SIM data.

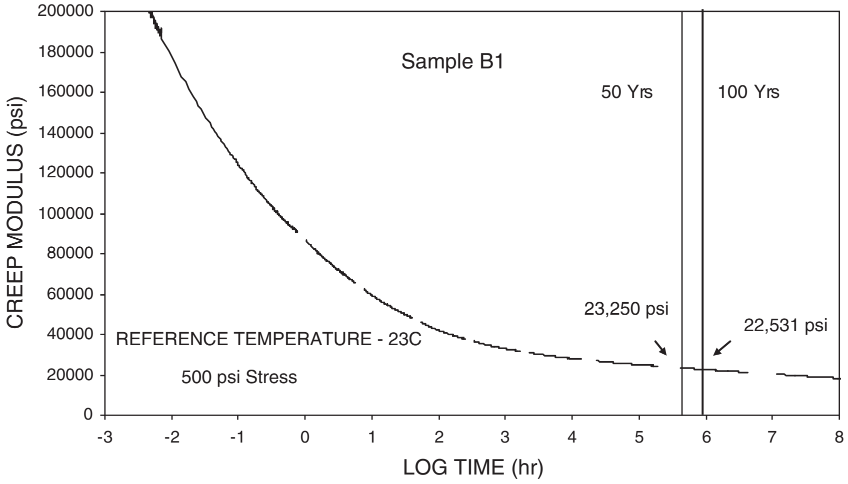

Figure 8. Creep modulus master curve under 500 psi of stress.

until the two parallel lines line up. The matching of the slopes of the end of one step with the beginning of the next step is the critical step for accurate extrapolations.

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 8 and 9.

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.

Notice that there are gaps between the extrapolated steps. 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. The researcher 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.

SIM can also be performed under higher loads to create a creep-rupture environment. Recall that the SIM test above was performed under an applied stress of 500 psi (about 12.4% of ultimate). If one does the same test at 1,000 psi, the master curve can produce the time it would take for the sample to yield under the applied load. This is shown in Figure 10. The extrapolate time to Stage I failure under these conditions is about 1,900 years. Shorter times are found for applied stresses of 1,500 and 2,000 psi (Figures 11 and 12).

These three results can then be put together on a plot of Log Stress vs. Log Time, to generate a Creep Rupture Master Curve. The one for the results above is shown in Figure 13.

The results from these tests show that the 50-year yield strength will be about 1,161 psi.

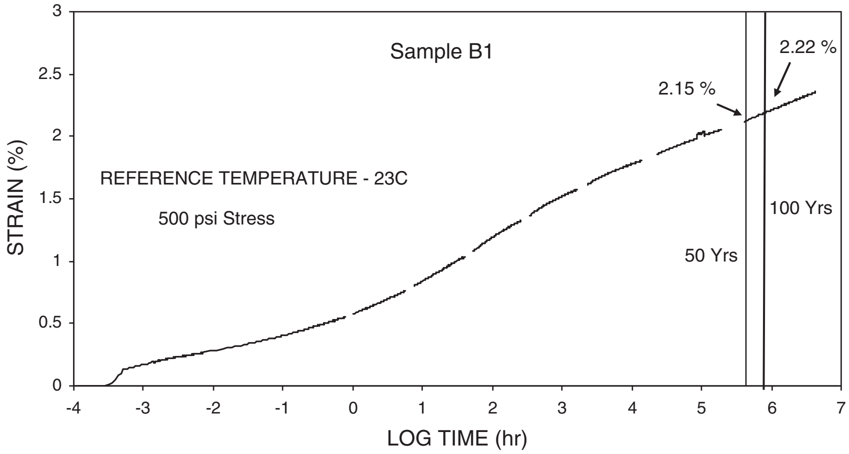

Figure 9. Creep strain master curve under 500 psi of stress.

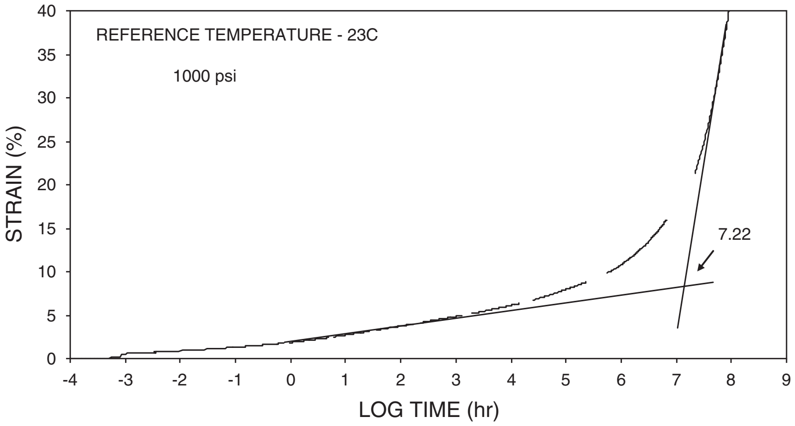

Figure 10. Long-term yield stress by SIM at 1,000 psi.

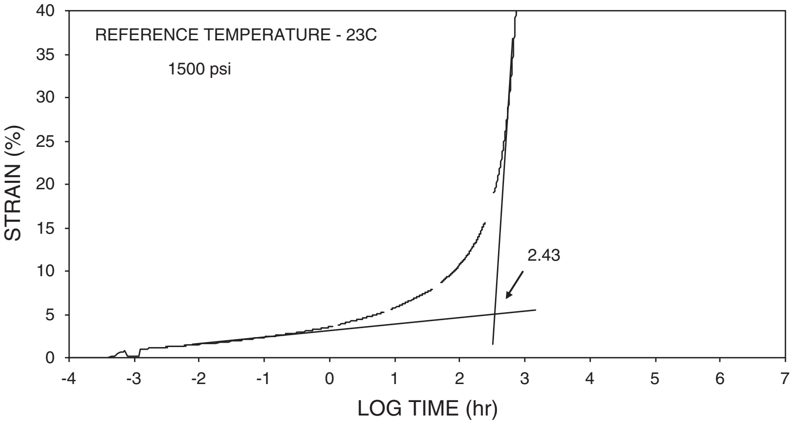

Figure 11. Long-term yield stress by SIM at 1,500 psi.

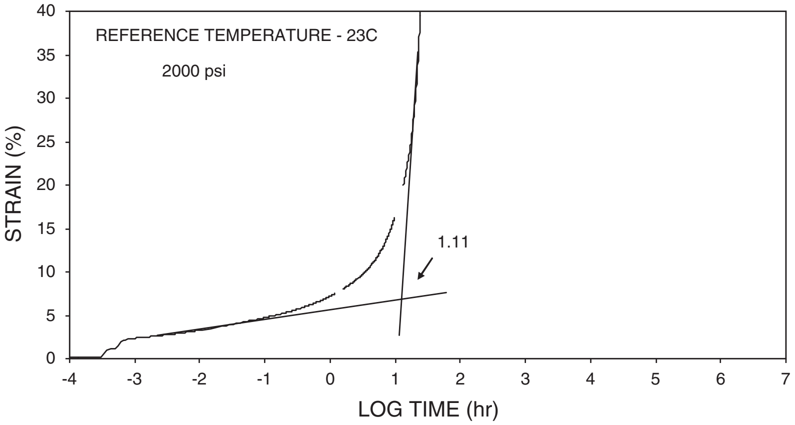

Figure 12. Long-term yield stress by SIM at 2,000 psi.

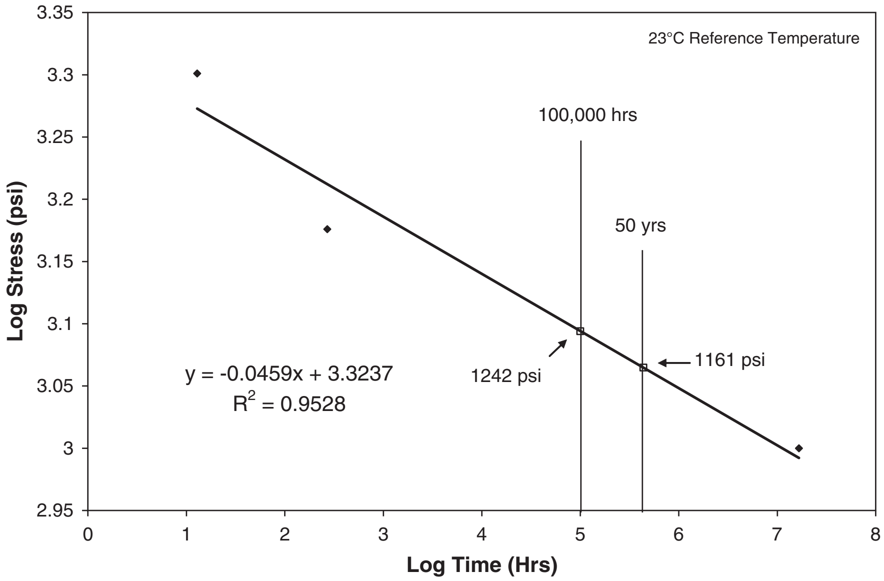

Figure 13. Creep rupture master curve.

The Long-Term Stress-Crack (Stage II) Resistance

The long-term stress-crack resistance of pipe formulations containing recycled content will certainly be the life-limiting property for properly installed corrugated pipes. The contaminants present can 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 content–containing pipe blends. For pressure pipe, the long-term strength is determined first (Stage I) at room temperature, then a validation test is performed at 80°C or 90°C to ensure that brittle crack growth (Stage II) will not occur during the service lifetime of the pipe. Validation can be performed two ways; one based on the Rate Process Method (RPM) (*11*) and one based on Popelar bi-directional shifting (POP) (*12*). These two methods of TTS are described in detail in Appendix D, Section D.8.2.

A test similar to the long-term hydrostatic strength test for pressure pipe is needed and will be sought as part of this study.

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

- 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 (*1*, *3*, *4*). These are commonly known as the BAM Test, the FL-DOT junction test, and the pipe ring test.

The FL-DOT Junction Test

The FL-DOT has sponsored a research project to develop test methods to determine if corrugated pipe can have a service lifetime of 100 years (*3*). 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. FL-DOT 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 in. × 1.3 in. The advantages for this test include the following:

- 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 RPM 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.

Table 1. FL-DOT FM 5-573 test conditions.

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

- Only the thinnest part of the specimen experiences the full stress.
- There is 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 three different conditions of stress and temperature in deionized (D.I.) water. These are shown in Table 1. Five replicates under each set of conditions are tested.

The three sets of results are then analyzed by the 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 POP.

The BFF Test

The BFF test uses a specimen, cut from a plaque, about the size of the BAM specimen (4 in. l × 0.5 in. w). The test is performed under the conditions of the FL-DOT durability protocol (80°C/650 psi, 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 5 times larger than the FL-DOT specimen.
- The thickness is controlled so the stress is even throughout the specimen.
- The specimen thickness (0.0040 in. to 0.0045 in.) 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 sensitive to contaminants found in PCR HDPE, like particulates.

However, this test will not be able to evaluate manufacturing stresses like the FL-DOT test. A test that still holds promise for that purpose is the ring stress-crack test (*7*). It could not be used during this project because of the large variability in pipe-wall thicknesses measured. However, there may be a way to modify it, or test multiple sections from a full diameter that would result in a valuable test for evaluating pipes containing recycled HDPE. More time and funding would be required to develop the concept into a valuable quality control (QC) or qualification test.

Similarly, if the FL-DOT junction test could be performed on wider 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 FL-DOT junction test with a Type IV (as defined by ASTM D638) specimen can be performed on most conventional stress-crack frames.

These tests will be evaluated for use as a tool for predicting the long-term stress-crack (Stage II) resistance of pipe-resin formulations containing recycled PE.

The Long-Term Oxidation (Stage III) Resistance

At very low service stresses and after very long times, a PE part can become oxidized and fail by many cracks forming in a short period of time. This failure mechanism is known, especially in exposed applications where UV initiates oxidation or in potable water applications where higher concentrations of chlorine can initiate oxidation. However, for typical buried pipe applications the rate of oxidation and the extraction of long-term antioxidants are very, very slow. Over the past 20 years, accelerated aging results on PE stabilized with common additive packages such as Irganox 1010* and Irgaphos 168 have suggested that the service lifetime is easily in the hundreds of years. Therefore, it is believed that as long as there is some basic stabilization, Stage III oxidation is not likely to occur in typical buried pipe applications. Specifications for pipe resins containing recycled content can specifically state that a particular stabilizer package be used. This can be verified with an OIT test. The OIT test cannot identify specific additives, but it can measure concentration if 1,010 and 168 are used. These ingredients are two of the least expensive stabilizers for PE. When used at levels of 1,000 ppm Irganox 1010 and

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*The Transportation Research Board, the National Research Council, the Federal Highway Administration, the American Association of State Highway and Transportation Officials, and the individual states participating in the National Cooperative Highway Research Program do not endorse products or manufacturers. Trade or manufacturers' names appear herein solely because they are considered essential to the clarity and completeness of the project reporting.

1,000 ppm Irgaphos 168, the OIT value will be around 70 minutes and the pipe will be well stabilized.

Specifications

Ensuring consistent properties for blends containing recycled resins will be challenging. There are a number of different kinds of companies that will be involved once AASHTO allows recycled use.

At the beginning of the process are the recycled-resin suppliers. These companies specialize in locating, cleaning, reprocessing, and selling recycled materials. Some are small family-owned businesses that simply grind and clean recycled materials while others are fairly sophisticated with automated cleaning and optical color sorting. And there are many in-between. Some will be happy just selling reprocessed recycled HDPE and others will want to prepare and sell fully formulated blends directly to the corrugated pipe manufacturer. Next will be the compounders. This group may include the recycled-resin supplier, but other companies with established sources of sub-prime virgin resins will use their blending and formulating technology to make fully formulated recycled blends for direct sales to the corrugators. And, finally, some pipe manufacturers themselves may want to prepare their own blends and only buy high quality reprocessed recycled material from the recycled suppliers. Because of this complicated way in which recycled resin may make its way into AASHTO approved pipe, there could be up to five different specifications.

First, one may need a specification for post-consumer recycled, mixed-color reprocessed (PCR-MCR) HDPE in pellet form. This will be used by compounders and corrugators to ensure that they are receiving a consistent stream of material from which to make recycled blends. Its focus will be mostly on how well the material is melt filtered and how much polypropylene is present, among other things. These are believed to be important properties for good recycled material.

Then there may be two specifications for fully formulated recycled content–containing blends. One will be for use in pipe for AASHTO M252 and drainage applications and the other for M294 applications. Apparently, these two applications are different enough that a different quality and type of resin is used for each one. These are necessary for the corrugators to know what is being purchased to be converted to pipe.

Finally, there will also be two specifications—M252-Recycled and M294-Recycled—for pipe made from recycled content–containing resins. These specifications will include both resin properties and the properties of the final pipe; all testing will be conducted on the pipe or on samples taken from the pipe.

CHAPTER 3

Findings

Phase 1—Recycled Polyethylene Resins

There are many different types of recycled PE available. One way to classify PE resins based on density is found in Table 2. Notice that at the extremes of density one finds LDPE and HDPE homopolymers. These were the first two types of PE commercially produced. LDPE has the most branching and HDPE has the least. The others are all made by copolymerizing ethylene gas with other α-olefins, which produces small branches in a controlled manner. The α-olefins are butene, hexene, and octene, which produce 2, 4, and 6 carbon branches, respectively. The density is varied by the amount of α-olefin added.

All of these types of PE are available on the recycled-resin market. However, some are certainly more available than others. The main subclasses for recycled PE are post-industrial recycled (PIR) and PCR. There are also several different physical forms one can buy. PIR resins are often sold in their bulk forms or the plastic parts may be ground into chips and sold as regrind. PCR resins are sold as regrind chips, or as a reprocessed resin (repro). Reprocessed resins are chips that have been melted, filtered and pelletized. They are more blended than chips, cleaner from the melt filtration step, and in a better form to feed into an extruder.

There were about 50 different recycled polyethylene suppliers identified and many were contacted for samples. A list can be found in Appendix B, Section B.8.

Post-Industrial Recycled Polyethylene

This is a large category that includes scrap from processes such as pipe, sheet, thermoforming, injection molding, blown film, tubing, and more. This includes low density, linear low density, linear medium density, and high density (homo- and co-polymer). The molecular weight also varies from injection molding grade (low molecular weight [LMW], high MI) to thermoforming or blown film (high MW [HMW], low MI).

There are certainly resins that would be appropriate for incorporation into pipe available from the PIR market. However, there is a downside. PIR material is sold mainly through brokerage firms and is commonly sold on a lot-to-lot basis. This means that reliable and continuous waste streams are not commonly found. Additionally, it is often in a bulk form and/or co-mingled with different grades of PE. The result is that the PIR material is used mostly in noncritical applications by plastic processors who have the capabilities to accommodate different grades of PE.

Despite the limitations mentioned above, three PIR PE samples were received for evaluation in this study. Two [HDPE and low-density PE (LDPE)] were provided by a re-processor that also produces post-consumer polyethylene. The third sample, medium-density PE (MDPE), was regrind scrap from a manufacturer of geomembranes for landfills. Properties of each of these recycled resins are found in Table 3.

Notice that each of these has stress-crack resistance (15% NCTL) higher than most, if not all of, the current AASHTO resins for pipe. They are also much higher in cracking resistance than post-consumer HDPE. Therefore, these resins could be useful for enhancing the properties of post-consumer HDPE. They are also much lower in density, which would limit how much could be used in a blend. Notice that the density of the low density material is actually higher than the density of the MDPE. This is due to the high level of inorganic fillers displayed by the percentage ash. In fact, each of the three density values are misrepresented because of fillers in the cases of the HDPE and LDPE and carbon black in the case of the MDPE sample. The density can be calculated from the yield stress through the following relationship:

$$\text{Yield Stress} = 81{,}250\ (\text{Density}) - 73{,}500$$

The calculated values for the HDPE, MDPE, and LDPE are 0.943, 0.937, and 0.925 g/cm^3, respectively. One can also correct for the carbon black from the MDPE sample because each 1

Table 2. Types of PE.

Common Name	Type (ASTM D1248)	Density (g/cm^3)
LDPE (homopolymer)	0	<0.910
LLDPE (copolymer)	1	0.910–0.925
LMDPE (copolymer)	2	>0.925–0.940
LHDPE (copolymer)	3	>0.940–0.960
HDPE (homopolymer)	4	>0.960

Note: LDDPE = linear low-density polyethylene, LMDPE = linear medium-density polyethylene, LHDPE = linear high-density polyethylene.

percentage of carbon black increases the density 0.0044 g/cm^3. This also produced a calculated value of 0.937 g/cm^3 for MDPE.

Post-Consumer Recycled High-Density Polyethylene

This is a smaller category, in terms of different resins, that includes primarily recycled bottles and high-strength shopping or trash bags. The volume of good recycled bags is low because the bulk density (volume/weight ratio) is low and the high quality bags are usually contaminated with lower quality LDPE bags. Additionally, the volume of plastic bags is shrinking because their use is being legislated against in a growing number of communities throughout the United States.

The bottles can either be natural or colored. The natural bottles are most often the ½ to 1 gallon water, juice, or milk jugs. They are made from HDPE homopolymer, which is a resin with high strength and poor stress-crack resistance.

The colored bottles include liquid detergent, cleaners, shampoos, fabric softeners, and others marked with a number "2" recycling code. The resin used in colored bottles is an HDPE co-polymer which is not as strong as the homopolymer but has better cracking resistance.

Both the natural and colored HDPE resins are available as regrind (chips) or reprocessed (pellets). The reprocessed pellets have the advantage of being melt-filtered during the pelletizing process. This is essentially one additional purification step. The colored-bottle recycled resin contains a significant amount of natural bottle in it. There is an increasing number of recyclers who are separating the natural bottles from the mixed-color bottles because the natural resin has significantly higher value on the recycled-resin market. The difference can be $0.10 per pound, or more.

Most of the samples found during this study came through contact with the Association of Post-Consumer Plastic Recyclers (APR).

Twenty-two samples of PCR HDPE were received from six member companies of the APR and two nonmembers. The samples included natural regrind (*2*), natural reprocessed (*4*), mixed-color regrind (MCRG) (*5*), MCR (*8*), and three special

Table 3. PIR resin properties.

	Resin Type		
Property	HDPE Reprocessed	MDPE Regrind	LDPE Reprocessed
Density (g/cm^3)	0.970	0.942	0.952
Calculated Density[1] (g/cm^3)	0.943	0.937	0.925
Melt Index (g/10 min)	0.32	0.66	0.80
MFR (21.6/2.16kg)	101	46	34
% Volatiles	0.24	—	0.18
% Color	0.28	1.05	0.15
% Ash	3.69	0.05	3.75
Yield Stress (psi)	3143	2631	1686
Break Strain (%)	628	662	727
15% NCTL (h)	104	>800	>300
OIT (min)	18.2	61.4	6.3

[1] Calculated from the yield stress.

Note: MFR = melt flow rate; "—" = no data available.

blends labeled "mixed color plus" by project personnel. Two of these are MCR with added high molecular-weight (HMW)-HDPE to assist with processing, and to improve properties such as impact strength and stress-crack resistance. The third is a blend of mixed-color PCR HDPE with PIR HDPE.

These resins were made into plaques and characterized by a variety of tests. The full test reports for these materials are found in Appendix B, Section B.5. The results were originally generated on plaques made from the "as-received" pellets. Test results indicated that further blending was necessary to obtain homogeneous material for characterization. This is reasonable because single screw production lines are not as well suited for blending as a twin screw. If the recyclers were to improve the mixing on their extruders, the changes could cause more thermal-oxidative degradation to the material. Therefore, the MCRGs and reprocessed materials were further blended and recharacterized. In general, the regrind samples received were melt-blended at least twice and the reprocessed samples were further blended at least one additional time.

PCR Natural Resin

The average resin properties for post-consumer natural recycled HDPE have been summarized in Table 4. TRI-repro was a blend of the others made in-house as an "average" sample. A milk bottle was also tested for comparison.

Observations from the summary table include the following:

- The density and melt flow values are higher than typically found for AASHTO pipe resin. AASHTO pipe resins are typically around 0.950 g/cm^3 and 0.15 g/10 min. The ash content is low. This demonstrates that recycled natural resin is very clean.
- The high yield stress is due to the higher density. Yield stress and density are linearly related.
- The percentage strain-at-break was low compared to virgin materials (>500%). This property can be considered a flaw detector, since every break in a tensile test occurs at a flaw. These results indicate that the small amount of particles present in the recycled result in early breaks. In turn, the flaws can also be initiators of stress cracks in the resultant pipe.
- The 15% NCTL times are low, which is a result of the high density and the fact that milk bottle resin is a homopolymer.
- The OIT values are indicative of stabilizer concentration, but how much is there cannot be determined without knowing the specific additive package and what the relationship between OIT and concentration is for that specific package. However, the data do indicate that there is some antioxidant protection remaining in the recycled HDPE.

Table 4. Post-consumer natural recycled PE properties.

Property	Resin Type TRI Repro	 Milk Bottle	Average	Range
Density (g/cm^3)	0.960	0.958	0.957 ± 0.002	0.955–0.960
Melt Index (g/10 min)	0.79	0.74	0.67 ± 0.1	0.57–0.81
MFR (21.6/2.16kg)	71	75	87 ± 8	76–96
% Ash	0.06	0.04	0.11 ± 0.05	0.05–0.14
Yield Strength (psi)	4523	4316	4402 ± 79	4304–4489
Break Strain (%)	365	114	157 ± 73	75–229
15% NCTL (h)	2.0	5.4	3.7 ± 1.6	1.8–5.7
OIT (min)	—	23	16.5 ± 7.5	9–27

Note: "—" = no data available.

The advantage of this recycled resin is that it is clean and strong; the disadvantages include the cost and the poor cracking resistance.

PCR Mixed-Color Resin

Most of the samples obtained fell into the category of PCR mixed-color resin. There were 15 different samples from eight different suppliers. There were five regrind resins and 10 reprocessed resins. And, of the 10 reprocessed resins, three had something else added. Suppliers 2 and 3 sent samples with added HMW PE to improve processing and stress-crack resistance. Supplier 5 sent a product with additional PIRHDPE added to improve its properties. Suppliers 1 and 3 sent samples of regrind resins at different times, so the consistency of their products could be examined.

The average properties of all the mixed-color resins received are found in Table 5. This table provides a snapshot of the PCR resins available in the first half of 2007. The sample called TRI Repro 1 was a batch prepared in house on the twin screw extruder. All the available mixed-color reprocessed samples were combined to prepare 20 lbs of resin that was basically an average of the samples received.

There are a number of important observations from these results:

- The density, melt index, and yield stress vary with the amount of milk bottles present in the mixed-color waste stream. The values are probably more consistent in 2010 than in 2007 because more processors are separating the milk bottles due to their increased value over colored bottles.
- These resins typically have little color. In this case, all the values were less than 0.4%, except one sample where the manufacturer added 1.3%.
- The percentage ash in the products can be reduced by melt filtration through a larger mesh size (smaller openings). This, in turn, will improve the percentage break strain.

Table 5. Post-consumer mixed-color recycled PE properties.

Property	TRI Repro 1	Average	Range
Density (g/cm^3)	0.960	0.955 ± 0.005	0.946–0.960
Melt Index (g/10 min)	0.54	0.48 ± 0.10	0.37–0.64
MFR (21.6/2.16kg)	83	97 ± 14	75–122
% Color	0.4	0.31 ± 0.32	0–1.3
% Ash	1.2	1.1 ± 0.3	0.4–1.5
% PP	5.7	3.6 ± 1.5	0.8–6.3
Yield Strength (psi)	3,685	3,728 ± 218	3,327–4,037
Break Strain (%)	46	90 ± 81	9–302
15% NCTL (h)	8.8	7.8 ± 2.9	4.5–14.8
OIT (min)	12	13.6 ± 7.2	9–39

- The percentage PP reported was typical in 2007. The average is closer to 10% in 2010, due to the popularity of 2X and 3X detergent bottles. The percentage PP by weight can be as high as 30% in the 3X bottles. The caps and spouts are made from PP.
- The stress-crack values are typical for these products. Note that the NCLS stress-crack times (F2136) are even lower because the yield stresses are less than 4,000 psi. The NCLS test assumes a 4,000 psi yield stress and loads the samples at 600 psi, which is 15% of 4000. The load as a percentage of the yield stress will increase for resins with yield stresses less than 4,000 psi. For example, a sample with a yield stress of 3,327 will be loaded at 18% of its yield stress during the NCLS test.
- The OIT values show that some stabilizers are present, although it is not possible to know what they are from the results. That some stabilization is present, even after reprocessing, shows that the material is probably not affected by the additional stress of reprocessing. The rather large variability suggests that either some reprocessors are adding stabilizers, or additives are being consumed at different rates by different reprocessors.

OIT vs. OITemp

This has been a controversial issue for about 5 years now. ASTM D3350 requires the OITemp test to be performed on resins and that a minimum temperature of 220°C be obtained. Other industries, most notably geosynthetics, have used the OIT test for over 15 years to specify HDPE geomembranes. There have been discussions about which one was most appropriate for different applications and there have also been misinterpretations about the meaning of the results.

In the OIT test (ASTM D3895), a small sample (~5.0 mg) is maintained at 200°C in a pure oxygen environment until the protective stabilizers are consumed by the oxygen and the sample decomposes exothermally (it basically catches on fire). The time it takes from the addition of oxygen to the onset of oxidation is the OIT. It is known that in well-behaved additive packages, there is a relationship between the OIT value and the amount of additives present in the sample (*1, 2*). It is also known that the OIT value does not relate to the long-term performance of the material. The test merely shows the oxidation behavior at 200°C, which has little to do with real-time aging at ambient temperatures. However, if one exposes a sample to a condition that consumes additives (oven, chlorinated water, UV light) then the residual lifetime can be determined by the amount of OIT left after the exposure. This seems to be a reasonable approach. It is known from assessments of old geomembrane liners (*3*—TRI, Confidential files) that as the OIT value approaches zero, other changes can be seen such as a reduction in stress-crack resistance or change in melt index, both signs of polymer oxidation.

The OITemp test has historically been used on scrapings from the inside walls of thick-walled pipe after manufacturing. The inside wall is exposed to more heat longer and is the first place that will become oxidized during the process if adequate stabilizers are not present. Therefore, when a sample shows an OITemp value less than 220°C, that means it has been degraded during processing. So, it really does not say how good something is, but instead that it has not been made worse. That said, there is a relationship between OIT and OITemp.

Figure 14 shows the relationship between OITemp and OIT for 76 data points. The data includes eight PPI-certified virgin corrugated pipe resins, two geomembranes, 21 PCR samples, and 45 points from the Pennsylvania Deep Burial Project (*13*).

These results should settle the controversy. Since the relationship appears to be logarithmic, there will always be a larger change in the OIT value for a given change in the OITemp value. That means the OIT test is more sensitive and can distinguish materials better. Additionally, the OIT test is likely to be more sensitive to long-term antioxidants, while the OITemp is better suited to evaluate process stabilizers, since these are made to be active at higher temperatures. For these reasons, the OIT test will be used in the recommended specifications for the use of recycled resins.

The Effects of Contamination

The types of foreign matter that may be found in PCR HDPE include labels, paper, cardboard, dirt, aluminum foil, adhesives, and other polymers like PP (from caps), ethylene vinyl alcohol (EVOH), Nylon, PET or polyvinyl chloride

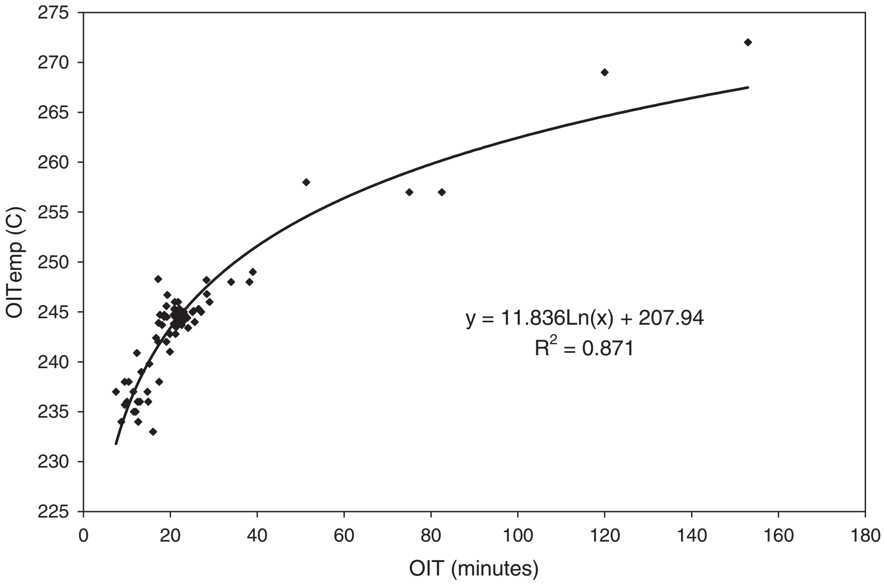

Figure 14. Comparison between the OITemp and the OIT.

(PVC). There is also a new plastic called poly(lactic acid) (PLA), which is derived from corn and is biodegradable. The recycled HDPE may be contaminated with other grades of PE, like LDPE, which could affect the cracking resistance. Finally, there may be milk and detergent residue, which produce smoke and odors during processing.

The foreign particulate matter can be removed during the washing step at the recycler and filtered out during extrusion by either the recycler or pipe manufacturer. However, the recyclers who produce reprocessed HDPE are more capable of this because some of them already melt filter to 100–120 mesh while the pipe manufacturers generally filter at a mesh size of 80 or below.

PP comes from bottle closures (caps) and is found in mixed-color reprocessed HDPE at levels as high as 20%. When processed, the PP will blend in with sufficient mixing, or be melted and spread out in the final part. This could affect the behavior of the pipe, especially the stress-crack resistance. The HDPE milk bottle resin contains no PP because the caps are made from LDPE. In fact, the recycled natural homopolymer (milk bottle) is much cleaner than the mixed color.

The effects of contamination have been investigated three ways. First, samples containing a known size and percentage of angular sand were prepared to see how the sand affected properties such as tensile elongation and stress-crack resistance. These results will offer some guidance concerning how much filtration to specify.

Secondly, two samples containing MCRG were prepared at three levels of filtration.

And, finally, samples containing different amounts of PP were prepared and evaluated.

The Effects of Particulates

Angular “playground” sand was obtained and sifted with both a 100 mesh and a 120 mesh screen. The sand that passed the 100 mesh, and retained by the 120 mesh was collected. The sand particles ranged in size from 0.0052 to 0.0061 in. From this material, a master batch containing 2.5% sand in Virgin Resin 1 (VR1) was prepared. The 2.5% master batch was diluted with more VR1 to produce samples of about 1.6%, 0.8%, 0.4%, and 0.2% sand. The actual values measured by ash analysis on duplicate samples were 1.61%, 0.79%, 0.41%, and 0.25% respectively.

Compression-molded plaques were prepared and the breaking strain and the BAM stress-crack test were performed on the samples. The BAM test is a stress-crack test without a notch and is described in Appendix A, Section A.3.2. The results are shown in Figures 15 and 16.

Notice that this size of sand particle has an extremely strong effect on the break strain, even at a level of 0.25%. The percentage break strain went from 468% to 148%, or to 32% of the original value. This demonstrates that it’s important to keep the particle size small and the amounts as low as possible.

The effect is similar for the BAM stress-crack test results. Sand in the amount of 0.25% reduced the stress-crack resistance to 62% of the original value.

The Effects of Melt Filtration

Two types of samples were prepared for this study. The first was 100% MCRG and the second was 50% MCRG + 50% Virgin MDPE. Each of these was melt filtered at three different

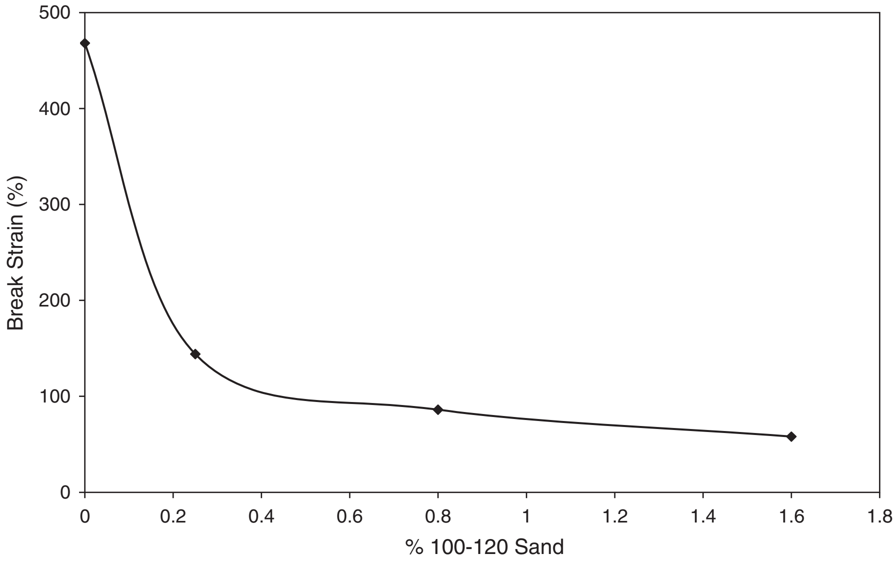

Figure 15. The effect of sand on the break strain.

levels. It should also be mentioned that the two sample sets were made with two different batches of MCRG HDPE.

The 100% MCRG was blended without filtration and also melt filtered at 100 and 150 mesh.

The 50/50 MCRG/MDPE samples were melt filtered at 40, 100, and 150 mesh. The filtration at 40 mesh was to help the material process better by removing larger particles that would cause the melt strand to break.

The tensile properties were measured on the two sample types. The percentage strain-at-break was chosen as the best result to follow the effects of blending. Plots of the effects of melt filtration on both types of samples are shown in Figures 17 and 18. Five replicates were tested for the 100% MCRG and 10 were tested for the 50/50 MCRG/MDPE.

Both sets of results indicate that melt filtration dramatically improves the properties of recycled HDPE and blends containing HDPE. Notice in Figure 18 that the scatter in results gets smaller with better melt filtration. This is important data because tensile specimens always fail at a defect. The plotted results clearly show that melt filtration removes defects. The coefficients of variation (COV) (standard deviation/mean × 100) for the three results were 69%, 30%, and 10% with increasing mesh size. A 10% COV is acceptable, even for virgin resins. This is a direct result of fewer larger particles in the samples.

The BAM stress-crack test is usually performed at 80°C in a surfactant (Igepal CA720) on a specimen without a notch. It is basically a flaw detector. The results were similar to the tensile results. Longer failure times were associated with higher

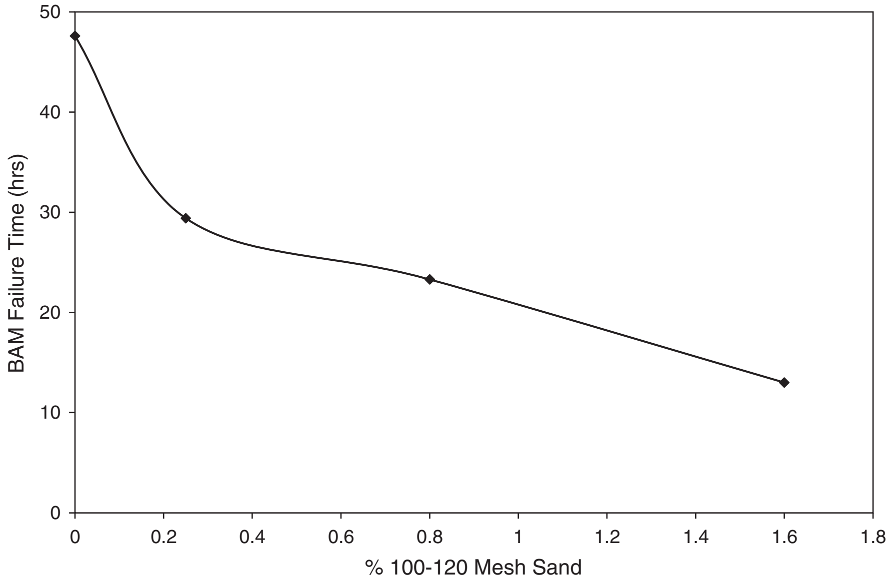

Figure 16. The effect of sand on the BAM stress-crack resistance.

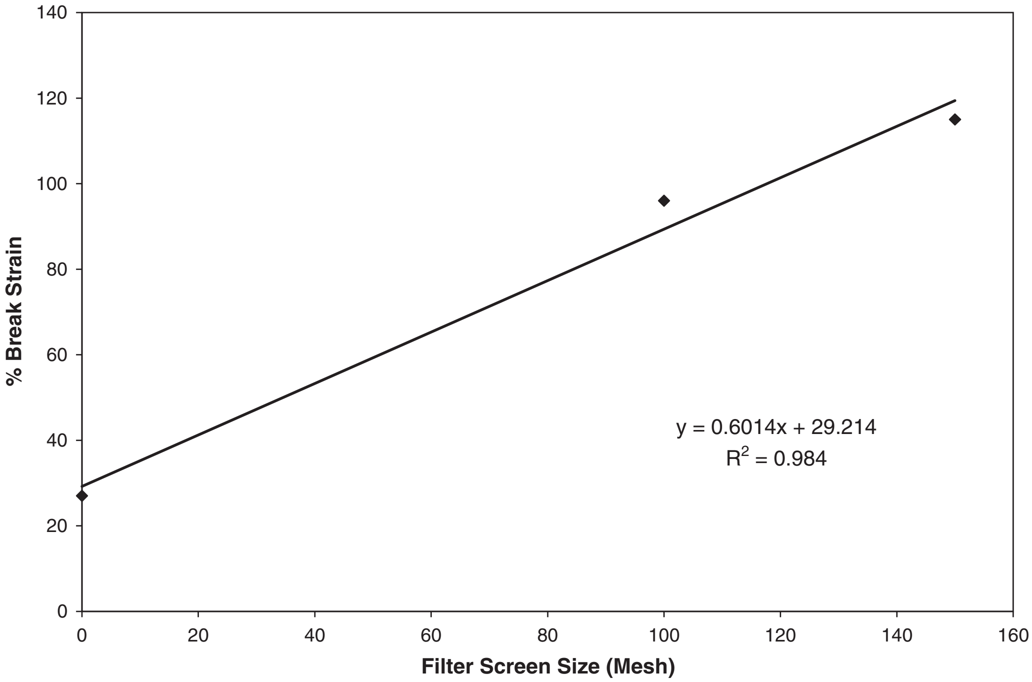

Figure 17. The effect of melt filtration on the percentage break strain of 100% MCRG.

mesh size. The results for both sets of results are shown in Figures 19 and 20. The tests run on the 100% MCRG were at 70°C.

The results of these experiments revealed that the degree of melt filtration is an important parameter for using recycled materials. It appears as if filtration at a mesh size in excess of 100 mesh should be a minimum requirement. However, the results of the BAM test did not show as much improvement in the results with filtration as one might think. The reason for this is found in the next section.

The Effect of Silicone Rubber

One of the advantages of the BAM test is that the fractured surface can be examined to see where the crack started. Examples are shown in Figures 21 and 22.

The clear, rubbery material was identified as silicone rubber by Fourier transform infrared (FTIR) analysis. The IR results are shown in Figure 23.

Four different rubbery particles were tested, and they all looked the same. It is not clear where the silicone rubber came

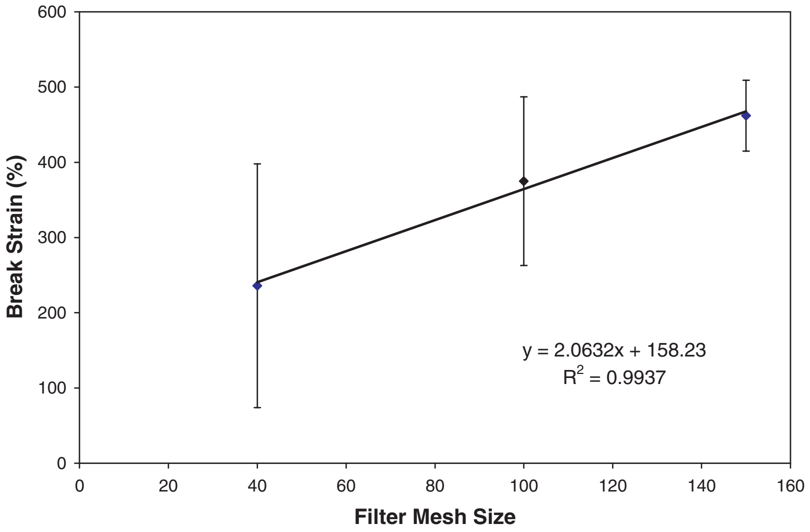

Figure 18. The effect of melt filtration on the percentage break strain of 50/50 MCRG/MDPE.

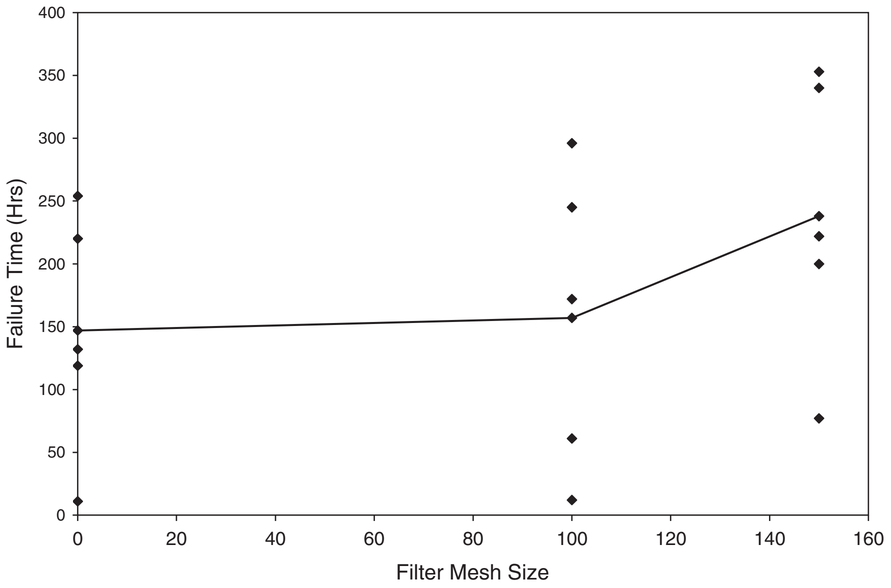

Figure 19. The effect of melt filtration on the BAM failure time of 100% MCRG at 70°C.

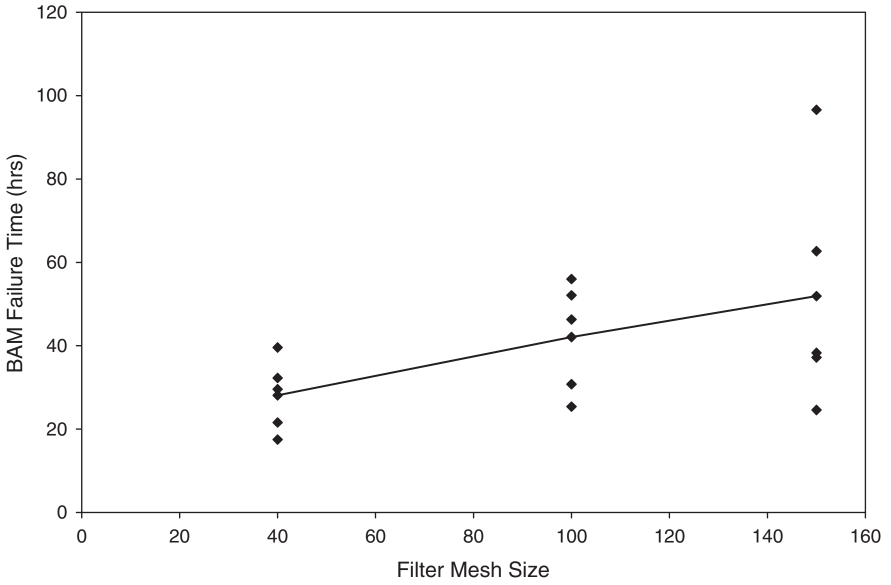

Figure 20. The effect of melt filtration on the BAM failure time of 50/50 MCRG/MDPE at 80°C.

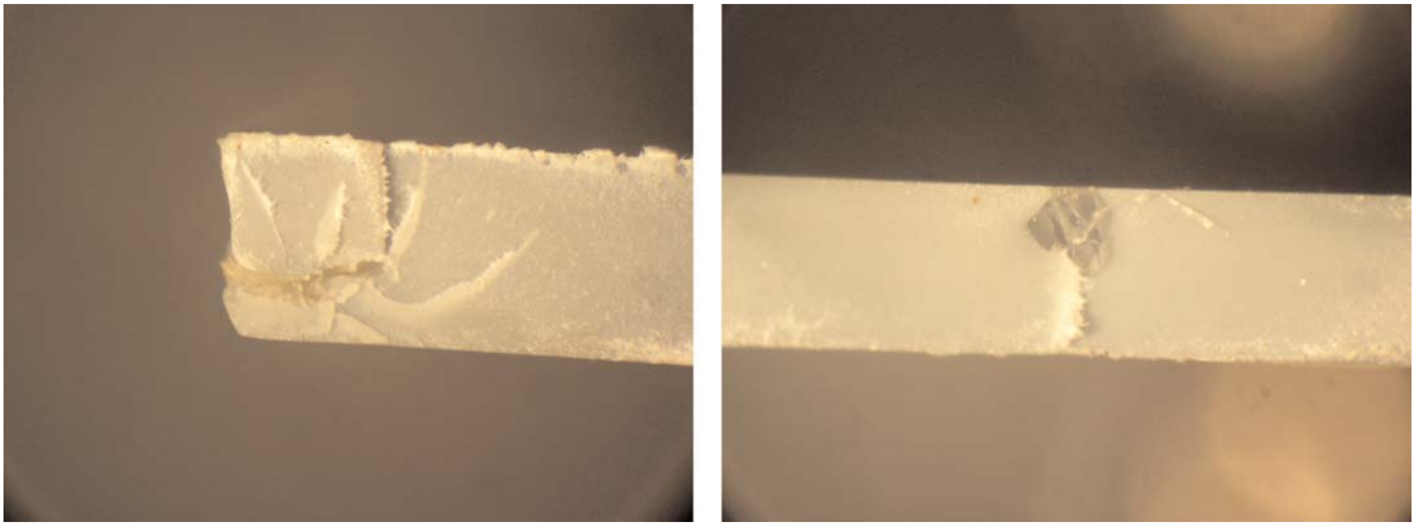

Figure 21. BAM test fracture surfaces for failure times of 11 h (left) and 133 h (right) (unfiltered).

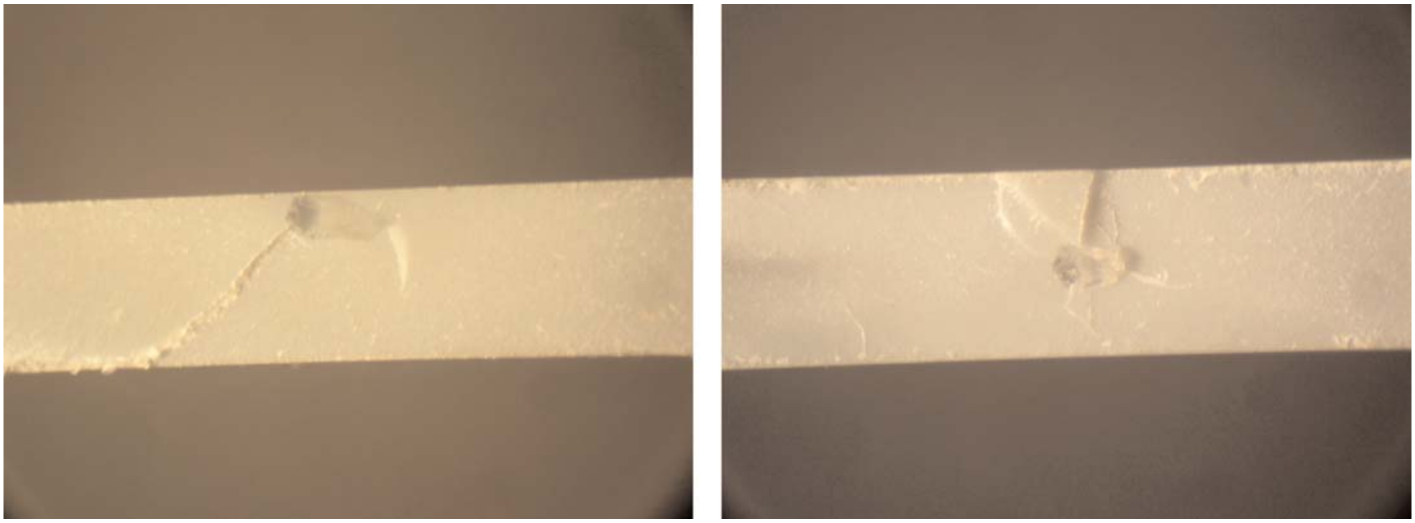

Figure 22. BAM test fracture surfaces for failure times of 12 h (left) and 172 h (right) (100 mesh).

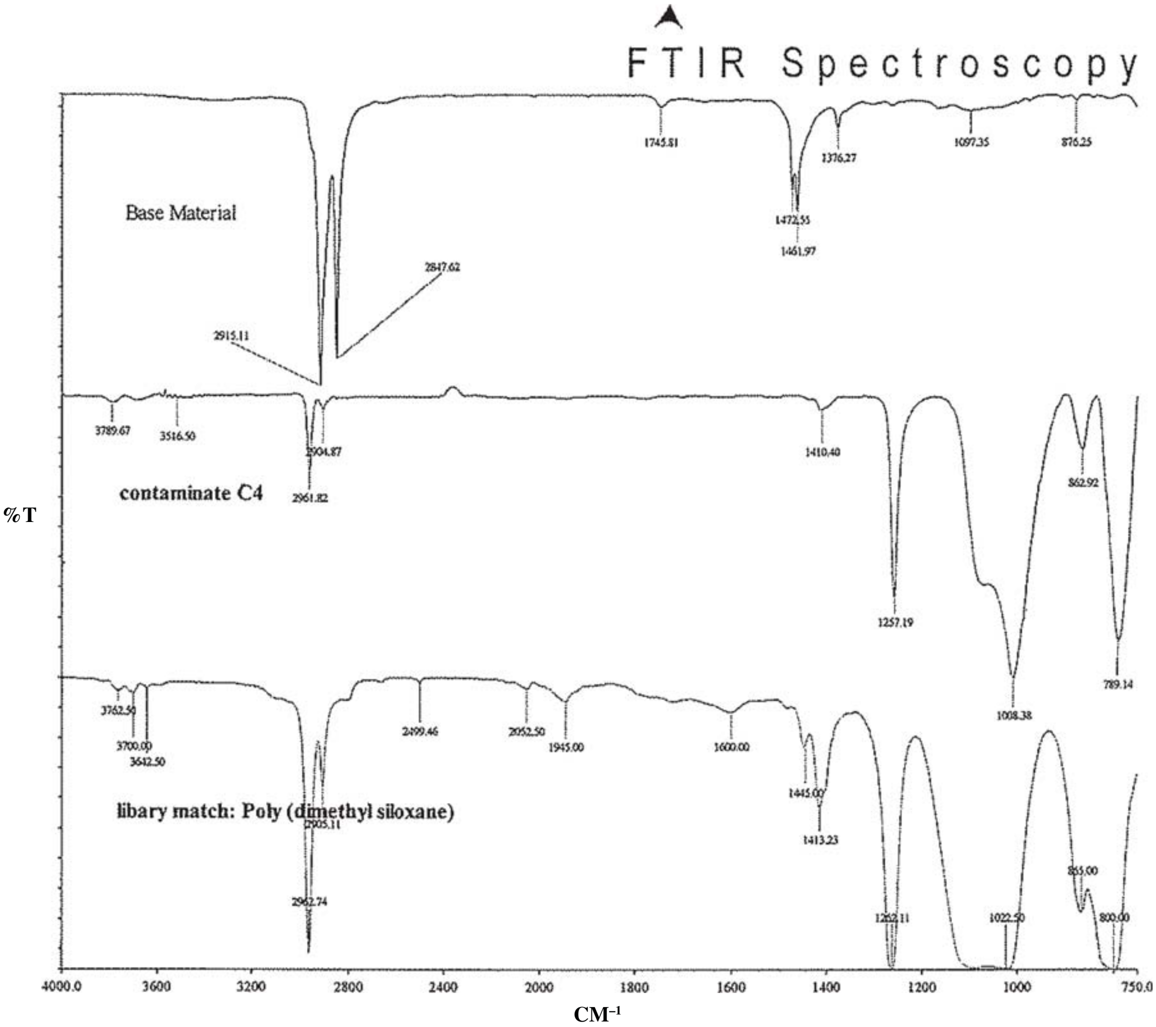

Figure 23. Fourier transform infrared (FTIR) spectra for HDPE (top), the rubbery contaminant (middle), and the best library match, silicone rubber.

from, but it is known that the rubber bladder found in the caps of some wide-mouth bottles is made from silicone rubber. Rubber has been seen in ketchup, mustard, jelly, and dessert topping bottles. Unfortunately, the rubber particles are so flexible that they pass through the filter screens. However, it is believed that the particles can be broken up by the screens, so better filtration should produce smaller particles. If this is combined with improvements in stress-crack resistance, then it is believed that the effect of this contaminant can be minimized.

The Effect of Polypropylene

PP is a contaminant in post-consumer MCRG and reprocessed resins that comes from the colored-bottle closures. The recyclers report PP at levels up to 20% by weight. Therefore, it is important to know its effect on the properties of HDPE.

There are two obvious ways to measure percentage PP. The first is with the use of FTIR spectroscopy. FTIR is an analytical technique that takes advantage of the fact that different combinations of atoms absorb IR radiation at different frequencies. The technique produces a chemical fingerprint of absorption bands of different intensities and at different frequencies. This can be used as a quantitative tool because the height of a particular band is directly related to its concentration, assuming the specimen thickness (path length) is a constant. With a blend of PE and PP, one can ratio two peaks, each specific to one of the polymers. The ratio of these peaks will be linearly related to the relative concentrations, up to a certain limit that can be determined experimentally. This technique works well for natural resins but becomes more difficult in the presence of colorants, particularly carbon black. These absorb IR radiation and can affect the peak heights and shift the linear portion of a calibration curve.

The other measure is with the use of DSC. A DSC measures thermodynamic transitions, like melting or decomposition. This is the method of choice during this study and details of the method can be found in Appendix, Section A.2.6.

Samples of VR1 with 2%, 5%, and 10% PP were prepared and tested. The properties evaluated were density, melt index (MI), break strain, and two different stress-crack tests (15% NCTL and NCLS). The results are shown in Figures 24 through 27.

The density and MI change in a predictable way because this PP has a lower density and a higher MI than VR1.

The break strain values reflect the lack of miscibility between HDPE and PP during extrusion.

The most interesting results were from the stress-crack tests (Figure 27). Notice that the stress crack resistance actually increased between 2% and 5%, all three times the test was run. The effect is not so great in the NCLS tests as in the 15% NCTL test. The NCLS results are effectively normalized by all the samples being placed under the same applied load. It is clear though, that the stress-crack resistance begins to be compromised around 5% PP.

Phase 2—Recycled-Resin Blends

The results from Phase I of the project showed that recycled HDPE had properties that were below the established limits of AASHTO-approved pipe. Therefore, the percentage of recycled material that can be blended with pipe resin will be limited by these properties. Efforts were undertaken to determine

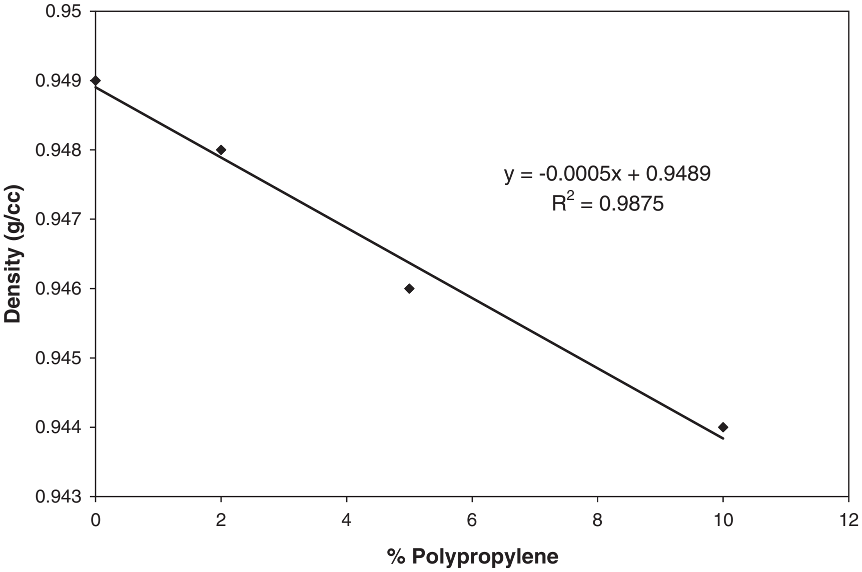

Figure 24. The effect of percentage PP on density.

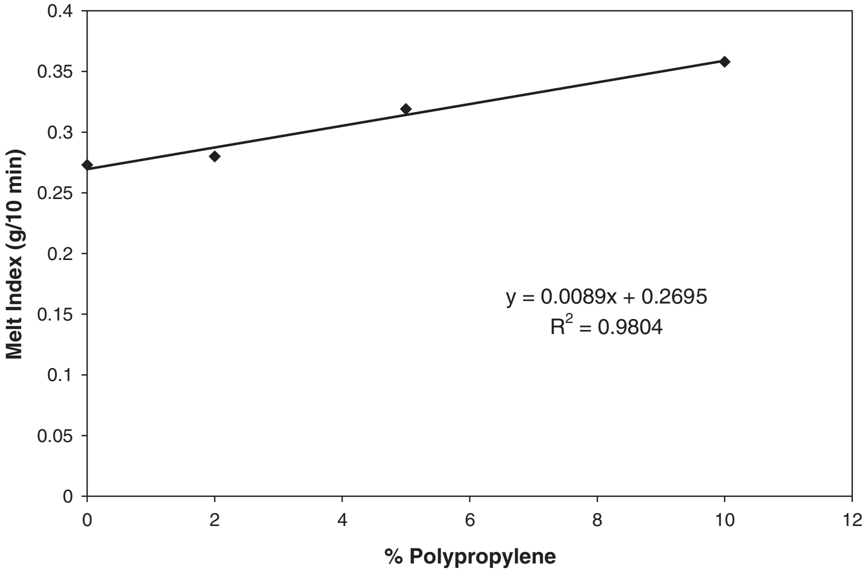

Figure 25. The effect of percentage PP on MI.

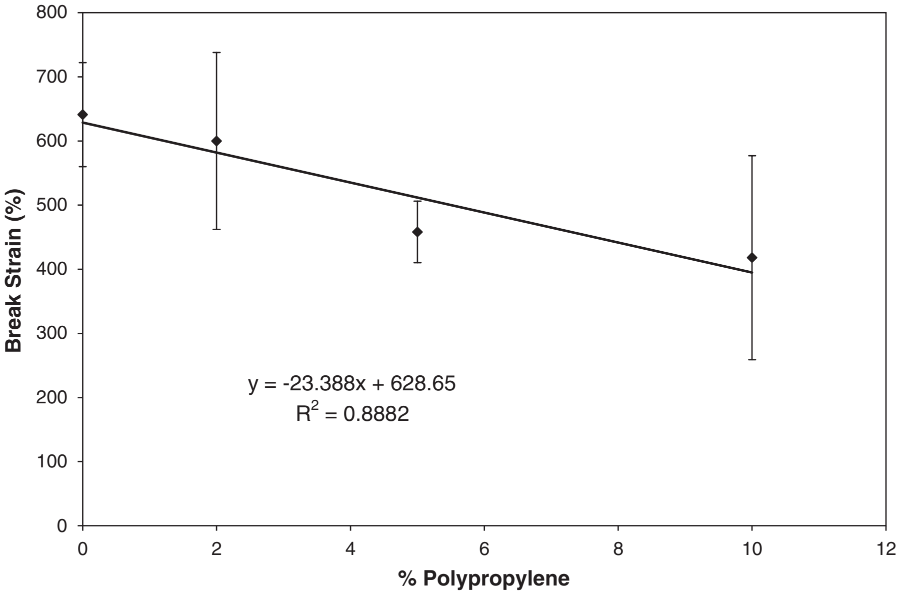

Figure 26. The effect of percentage PP on break strain.

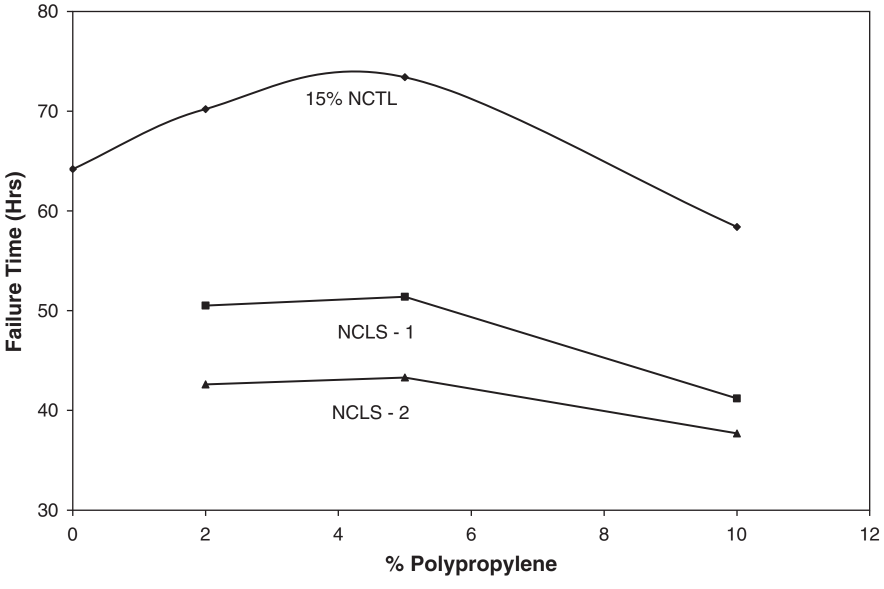

Figure 27. The effect of percentage PP on the stress-crack resistance.

Table 6. Component resins for blend preparation.

Resin	Abbreviation	Description
Virgin Resin 1	VR1	PPI-certified AASHTO HDPE pipe resin.
Virgin Resin 2	VR2	PPI-certified AASHTO HDPE pipe resin.
Virgin Resin 3	VR3	PPI-certified AASHTO HDPE pipe resin.
Virgin LLDPE	LLDPE	Commercial linear low-density polyethylene resin from a supplier that makes AASHTO pipe resin.
Virgin LMDPE	LMDPE	Commercial linear medium-density polyethylene resin from a supplier that makes AASHTO pipe resin.
Mixed-Color PCR 1	MCR1	Mixed-color post-consumer reprocessed HDPE pellets composed of colored and natural bottles.
Mixed-Color PCR 2	MCRG	Mixed-color post-consumer regrind HDPE chips composed of colored and natural bottle.
Natural PCR	NAT	Post-consumer reprocessed HDPE pellets made from milk, juice, and water bottles.
Natural PCR + 10% LLDPE	N10LL	Blend of NAT with 10% LLDPE to enhance the properties of the NAT.
Natural PCR + 35% LLDPE	N35LL	Blend of NAT with 35% LLDPE to enhance the properties of the NAT.
PIR Low Density	PIR-LD	Post-industrial low-density polyethylene reprocessed pellets believed to contain mostly film and bags.
PIR Medium Density	PIR-MD	Post-industrial linear medium-density polyethylene regrind chips from the sheet market.
PIR High Density	PIR-HD	Blend of PCR high-density bottles with PIR polyethylene.

what those limits were and to also enhance the properties of recycled HDPE by blending it with nonpipe virgin resins such as LLDPE and LMDPE. A secondary but important objective was to determine the relationships between the percentage component in a blend and the resulting blend's properties. Resins used during the blending study are given in Table 6.

A few selected properties of these resins are shown in Table 7. Notice the wide variability in the yield stress and NCTL values.

Blends Made with Mixed-Color PCR

A total of 29 blends were prepared with the use of Mixed-Color PCR bottle resin. They included

VR1 + MCR1 @ 20, 40, 60, and 80%,
VR1 + MCRG @ 20, 40, 60, and 80%,
VR2 + MCR1 @ 20, 40, 60, and 80%,
VR3 + MCR1 @ 20, 40, 60, and 80%,

Table 7. Properties of component resins for blending.

Resin	**Density (g/cm^3)**	**Yield Stress (psi)**	**Break Strain (%)**	**15% NCTL (h)**
VR1	0.950	3,688 ± 58	478 ± 45	45.8 ± 2.5
VR2	0.953	3,924 ± 58	639 ± 98	38.5 ± 3
VR3	0.949	3,764 ± 52	647 ± 83	36.2 ± 2.5
LLDPE	0.919	1,616 ± 19	771 ± 94	>1,000
LMDPE	0.934	2,732 ± 24	645 ± 49	>1,000
PCR-MCR1	0.960	3,620 ± 86	62.5 ± 27	7.6 ± 1
PCR-MCRG	0.960	3,527 ± 58	164 ± 30	7.6 ± 1
PCR-NAT	0.960	4,525 ± 42	302 ± 115	2.3 ± 0
PCR-N10LL	0.957	4,037 ± 60	411 ± 191	3.0 ± 0
PCR-N35LL	—	3,203 ± 54	655 ± 32	19.5 ± 3
PIR-LD	0.952	1,686 ± 42	727 ± 14	>300
PIR-MD	0.942	2,662 ± 27	692 ± 43	>300
PIR-HD	0.968	3,157 ± 32	684 ± 40	97.5 ± 9

Note: "—" = data not available.

MCRG + MDPE @ 25, 50, and 75%,
MCR1 + MDPE @ 25, 50, and 75%,
MCRG + PIR-MD @ 25, 50, and 75%,
75% MCR1 + 25% PIR-HD, and
50% VR3 + 25% MDPE + 25% MCR1.

The effects of recycled content on the yield strength for four blends of virgin pipe resins with mixed-colored PCR are shown in Figure 28.

It is fairly clear from these graphs that the yield strength is a linear function with respect to recycled content. That means that a simple mixing equation can be used to approximate the yield strength of a blend. This information will allow one to blend different resins to make sure that the resulting blend always stays within the specified yield strength requirements. The correlation is not particularly good, but this is likely caused by the combination of the two blend components not being too far apart in strength and the higher scatter found with recycled materials.

Similar plots are shown for the breaking strain in Figure 29. Notice that these are even farther away from the theoretical line, and the scatter in the results is quite high. This is a reflection of the contaminants found in PCR resins and demonstrates the need to control contamination.

Plots of the NCTL stress-crack resistance determined at 15% of the yield strength are shown in Figure 30. In this case, the curves are obviously exponential in nature and the match between theoretical and actual is much better.

Appendix C, Section C.9 contains summary tables for all the blends made with PCR-MCR, plots of properties versus percentage recycled content, and individual property reports for the 29 blends. Examination of the results reveals that all the properties change in either a linear or an exponential manner. More specifically, all the property changes are linear except for the melt flow (both loads) and the stress-crack resistance. This is powerful information because the properties of blends can be predicted based on these relationships. However, some of the inherent scatter found in certain properties makes such predictions unreliable. It is believed, though, that the relationships can be used as a guide for preparing blends with the understanding that actual blend testing will still be required.

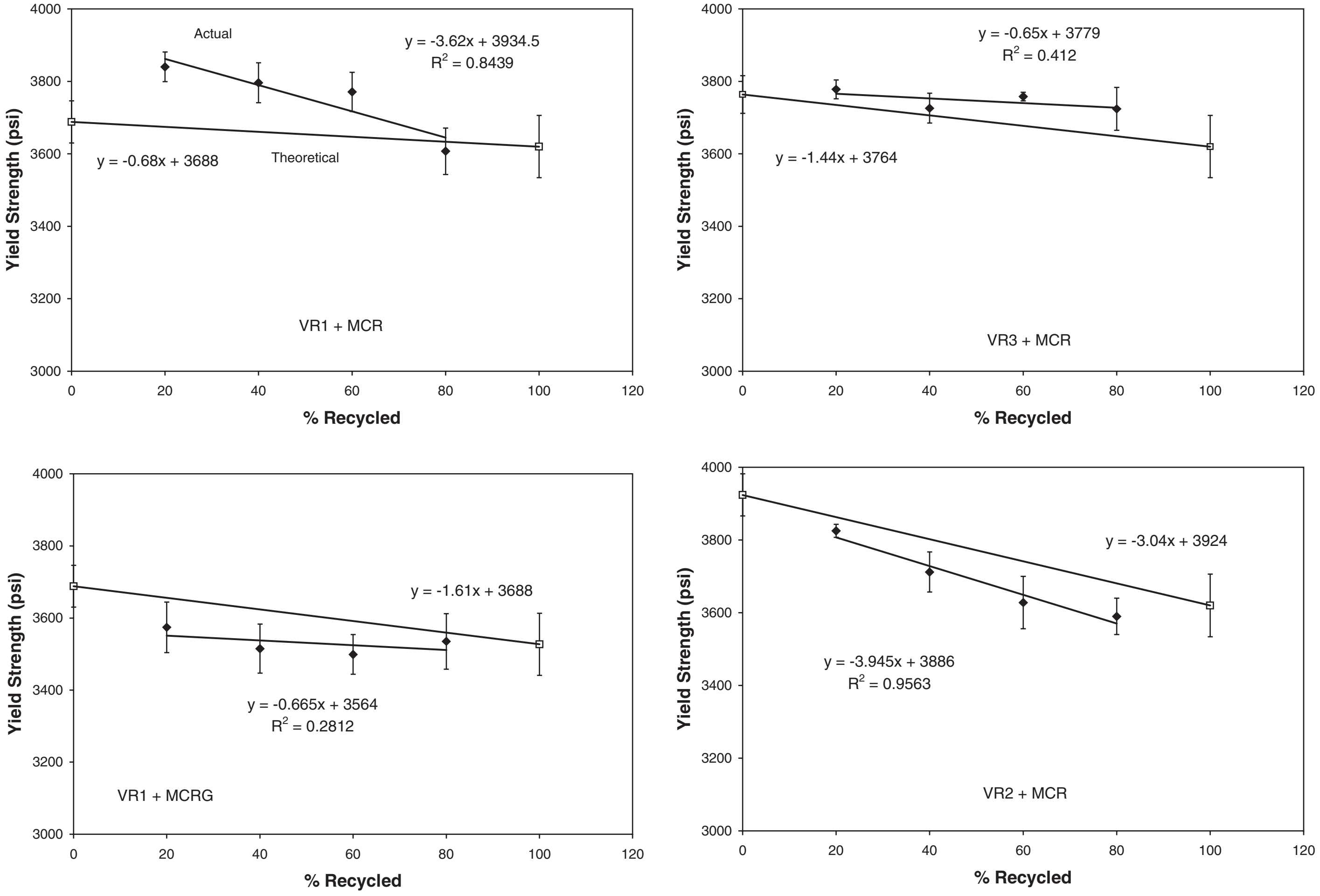

Figure 28. The effect of recycled content on the yield strength of PCR-MCR blends.

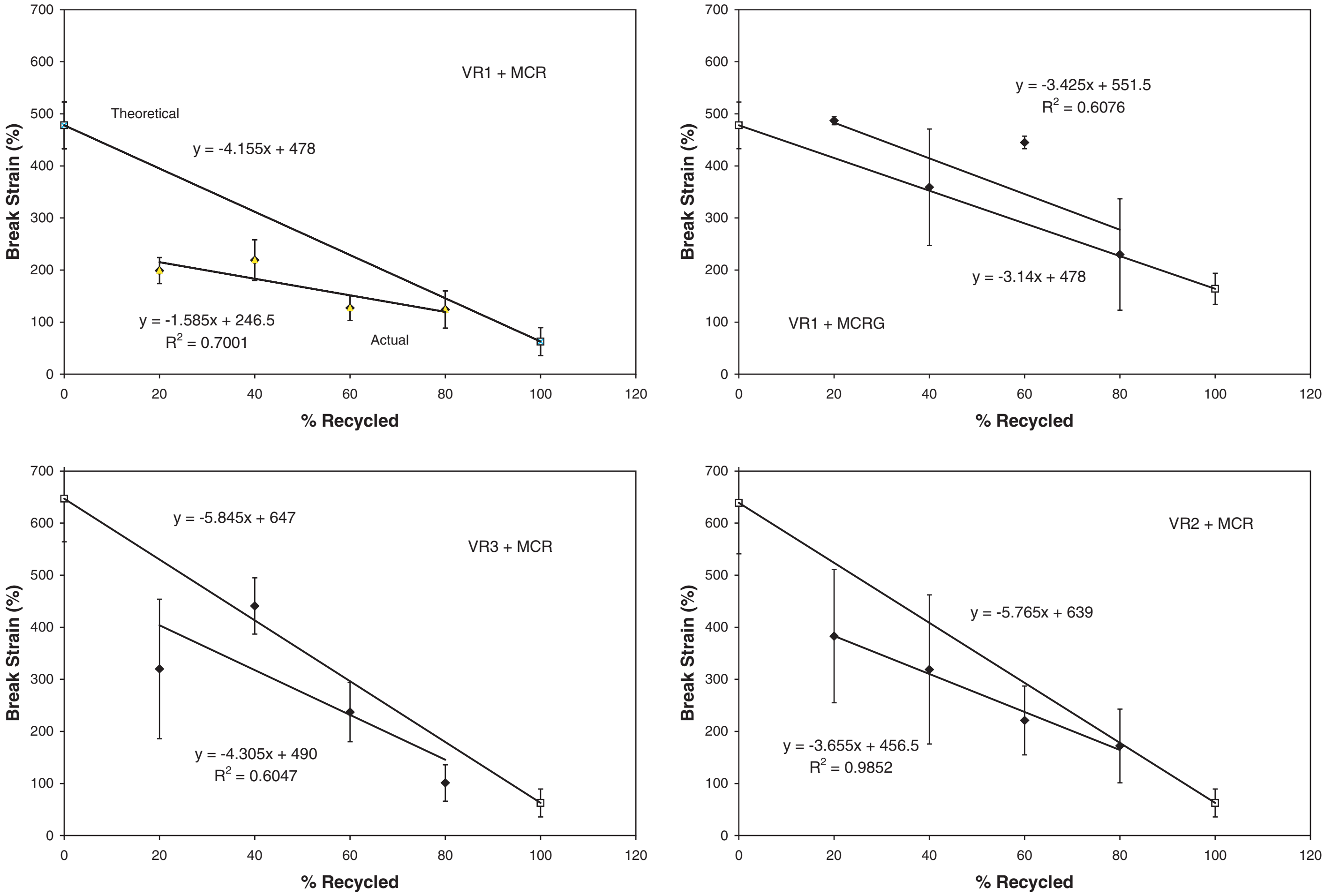

Figure 29. The effect of recycled content on the break strain of PCR-MCR blends.

The results of the blending and testing with mixed-color PCR HDPE have produced the following findings:

1. The maximum amount of mixed-color PCR that can be blended with one of the pipe resins and meet 24 hours of stress-crack resistance is about 20%. And, since the 15% NCTL is less aggressive that the NCLS test, a conservative number is closer to 15%.
2. At 15% added MCR, all the AASHTO requirements of pipe would be met.
3. The two different batches of mixed-color PCR (MCR1, MCRG) behaved dramatically different. The latter produced much better correlation to theory and had a much higher break strain, showing that there were fewer contaminants in the sample.
4. The difference between the predicted and actual values of percentage strain-at-break might be used to evaluate the level of contamination in the recycled material.
5. Much greater stress-crack resistance may be required to offset the deleterious effects of contamination.
6. The stress-crack resistance can be dramatically improved by the addition of MDPE to the mixed-color PCR. A 50:50 blend would produce a resin with about 200 hours in the 15% NCTL test. The yield stress would be reduced to about 3,250 psi, so this must be kept in balance.
7. The PIR-MD evaluated also improved the resistance to cracking, but not as much as the virgin MD.

Blends Made with Natural PCR

There were 27 blends made with natural PCR HDPE. They included

VR2 + NAT @ 20, 40, 60, and 80%,
NAT + LLDPE @ 20, 40, 60, and 80%,
NAT + MDPE @ 20, 40, 60, and 80%,
VR1 + N10LL @ 20, 40, 60, and 80%,
VR2 + N10LL @ 20, 40, 60, and 80%,
VR1 + N35LL @ 20, 40, 60, and 80%,
50% NAT + 50% MDPE,

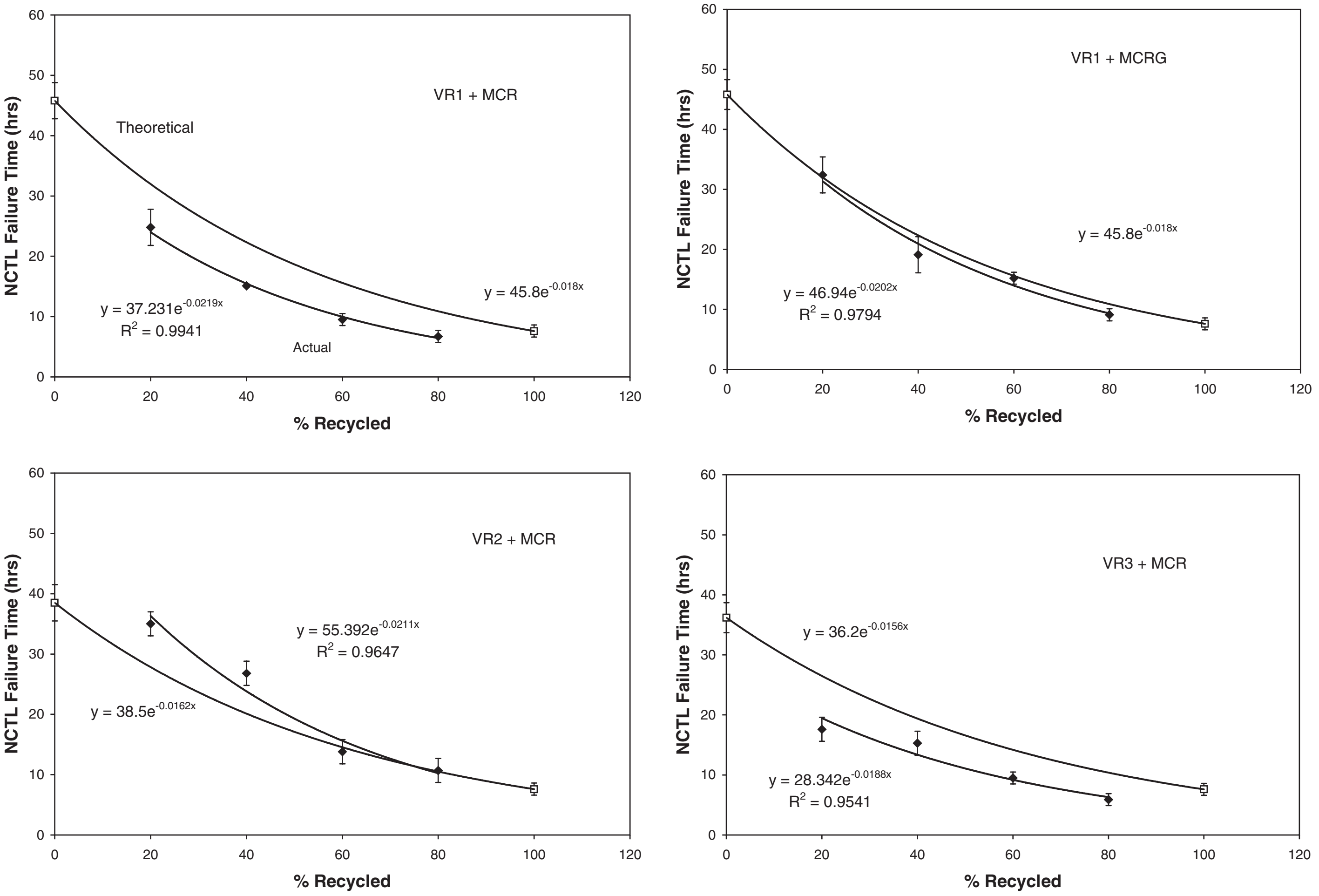

Figure 30. The effect of recycled content on the 15% NCTL on PCR-MCR blends.

65% NAT + 35% PIR-LD, and
50% VR3 + 25% MDPE + 25% NAT.

The effect of recycled content on the yield strength for four blends of virgin pipe resins with natural PCR is shown in Figure 31.

The N10LL and N35LL are natural PCR with added LLDPE at 10% and 35% by weight. The correlation coefficients for the lines and the agreement between the actual and theoretical are poor.

These results are similar to those seen for the mixed-color resin. Once again the correlation between the actual results and the theoretical ones are relatively poor.

Similar plots for the breaking strain and the 15% NCTL stress-crack resistance are shown in Figures 32 and 33.

The poor correlation in the break strain for the NAT + LLDPE series is easily explained by contaminants. The NAT itself has a COV of 38%, which is carried over into the blends with low amounts of LLDPE. The COVs for 5%, 10%, and 20% LLDPE were 33%, 38%, and 25% respectively. Interestingly, the series of NAT with MDPE also has high scatter, but the averages happen to fall in line so it doesn't present as dramatically as the NAT + LL results. The COVs for 20% and 40% MDPE were 36% and 28%.

These results show a good correlation between the actual and theoretical. This may be due to the fact that the applied loads are based on the yield strengths of the samples. Differences from plaque to plaque, which may contribute to the scatter in the yield stress, are minimized by the applied load being 15% of the yield stress. Regardless, these results show a definite exponential relationship between the stress-crack resistance and the recycled content.

Appendix C, Section C.10 contains summary tables for the blend series, plots of properties versus percentage recycled, and individual property reports for the 27 blends.

The results of the blending and testing with natural PCR-HDPE have produced the following findings:

1. Only about 10% of natural PCR-HDPE can be added to virgin pipe resins and meet a 15% NCTL time of 24 h.

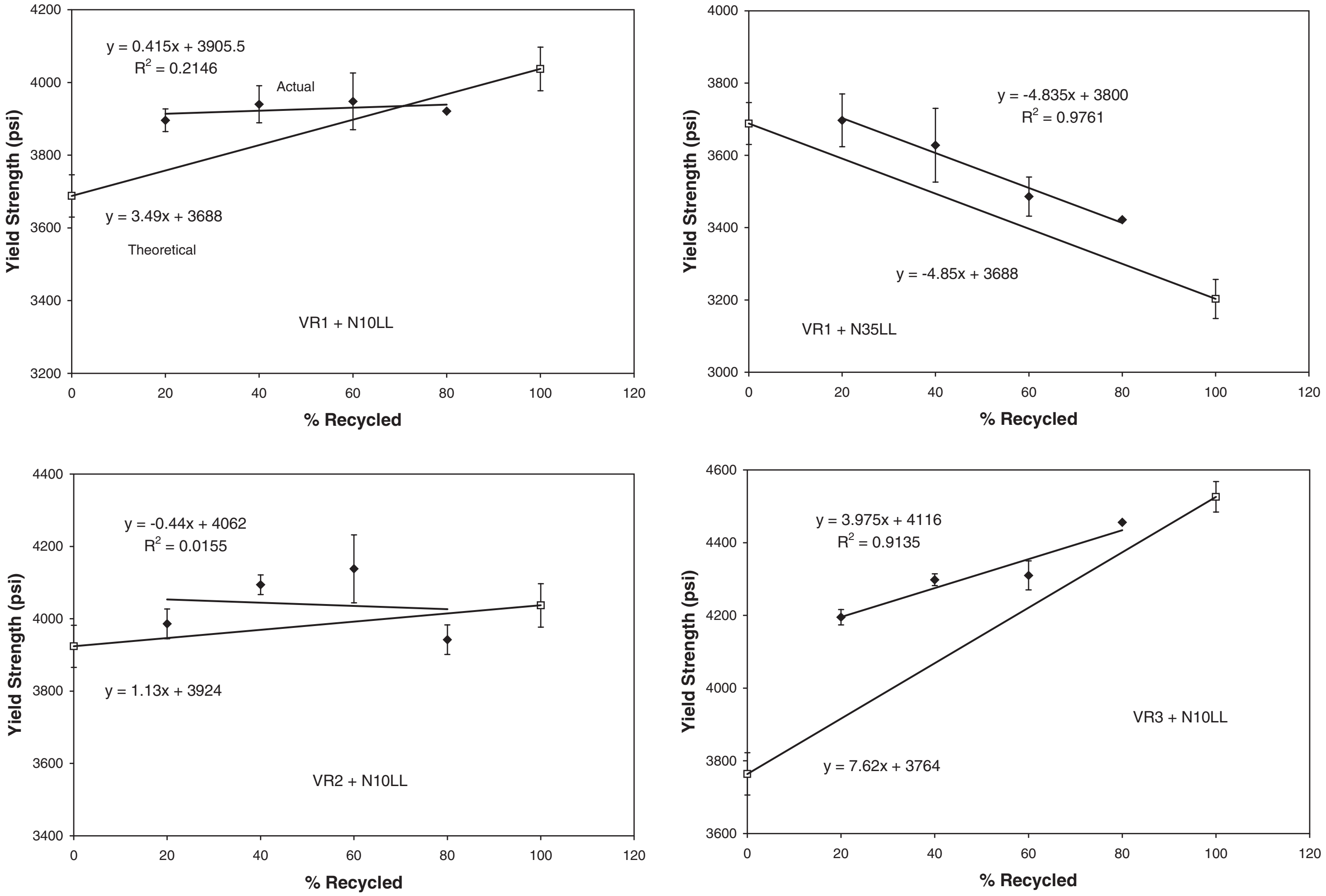

Figure 31. The effect of recycled content on the yield strength of PCR-NAT blends.

However, the yield will be over 4,000 psi, so the NCLS test will be less severe for this blend. That means that the limit might be closer to 15%.

2. Dramatic improvements in stress-crack resistance can be obtained by blending the NAT with either LLDPE or MDPE. A failure time of 50 h in the 15% NCTL test can be obtained with around 45% of added LL and 55% of added MD.
3. Blends between NAT and MDPE are preferred because the yield stress remains higher for the MD blends. For example, the yield stress for 45% LL is around 2,900 psi, while the yield stress for 55% MD is around 3,400 psi. The AASHTO minimum-density requirement for pipe resins is 0.948 g/cm^3, which correlates to a yield stress of around 3,500 psi.
4. The addition of only 10% LLDPE does very little to improve the properties of resulting blends.
5. A blend of 50% VR3, 25% NAT and 25% MDPE has properties very close to a PPI-certified pipe resin.

Blends Made with PIR-HD

A total of 12 blends were prepared with PIR-HDPE. Appendix C, Section C.11 contains summary tables for the blend series, plots of properties versus percentage recycled content, and individual property reports for the 12 blends. Tables containing correlation coefficients and predicted versus measured properties are found in Tables C-7 and C-8.

The blends were

VR1 + PIR-HD @ 20, 40, 60, AND 80%,
VR2 + PIR-HD @ 20, 40, 60, AND 80%,
VR3 + PIR-HD @ 20, 40, 60, AND 80%.

This series behaved more predictably. The PIR-HD had 3.9% color + ash but also a high break strain of 720%. The average yield stress of 3,157 psi suggests its true density is around 0.943 g/cm^3 and its 15% NCTL time is around 98 h. This is a very good resin for blending because it seems to lack the type of contamination that produced the high scatter in the other blends.

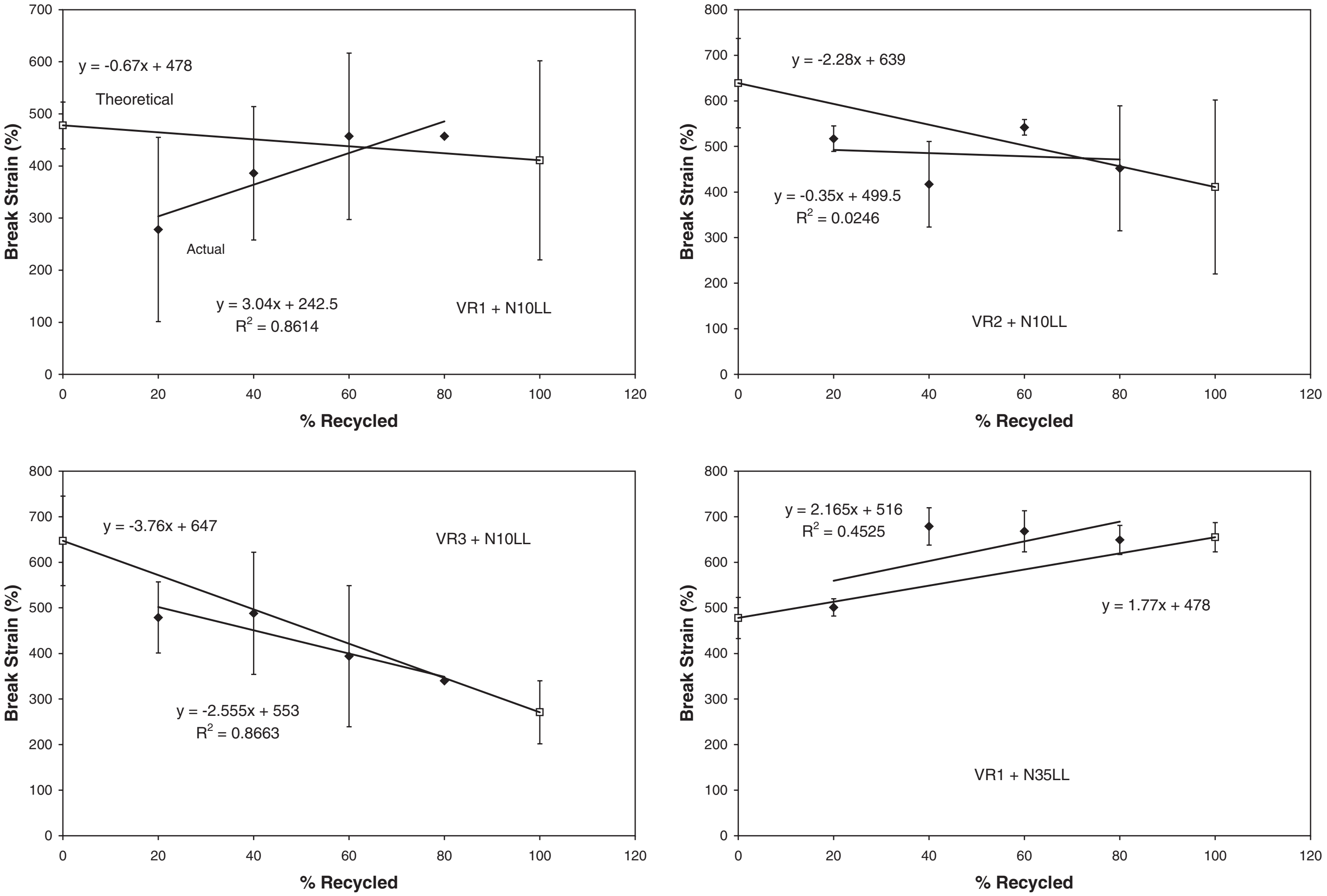

Figure 32. The effect of recycled content on the break strain of PCR-NAT blends.

The results of blending and testing with PIR-HD have led to the following findings:

1. This PIR resin is apparently void of the contaminants found in PCR bottles that create high scatter in some properties, particularly break strain.
2. A resin with a base density of around 0.943 g/cm^3 is an excellent resin for blending because it has a yield stress of around 3,150 psi and stress-crack resistance around 100 h.
3. These test results served to validate the relationships found in the other blends.
4. Blends of virgin resins containing up to 40% PIR-HD had yield stresses around 3,500 psi, break strains above 550%, and 15% NCTL times greater than 40 h. This blend would meet the resin properties found in AASHTO M294 for pipe.

A total of 66 blends were prepared and tested to find out how much recycled resin could be used in three PPI-certified resins where the final product would still meet the AASHTO M294 resin requirements for corrugated pipe. It was determined that for simple, two component blends, the maximum amount of PCR-HDPE is around 15%, while a specific PIR-HD obtained could be used in amounts up to 40%.

More importantly, it was found that through the relationships discovered during this task, other two and three component blends could be designed and optimized for the specific purpose of maximizing the amount of recycled HDPE used. This information will be invaluable to those developing new blends for improved short- and long-term properties of corrugated pipe resins. Contaminants like particles and silicone rubber seemed to affect the relationships in a negative way so the relationships are probably most useful as guidelines; some actual testing will still be required.

It also should be stated that much better recycled blends can be made than the ones described in this report. The results herein were limited by the fact that the recycled resins were blended with PPI-certified pipe resins. The virgin resins only had approximately 50 h of NCLS time to begin with. Starting

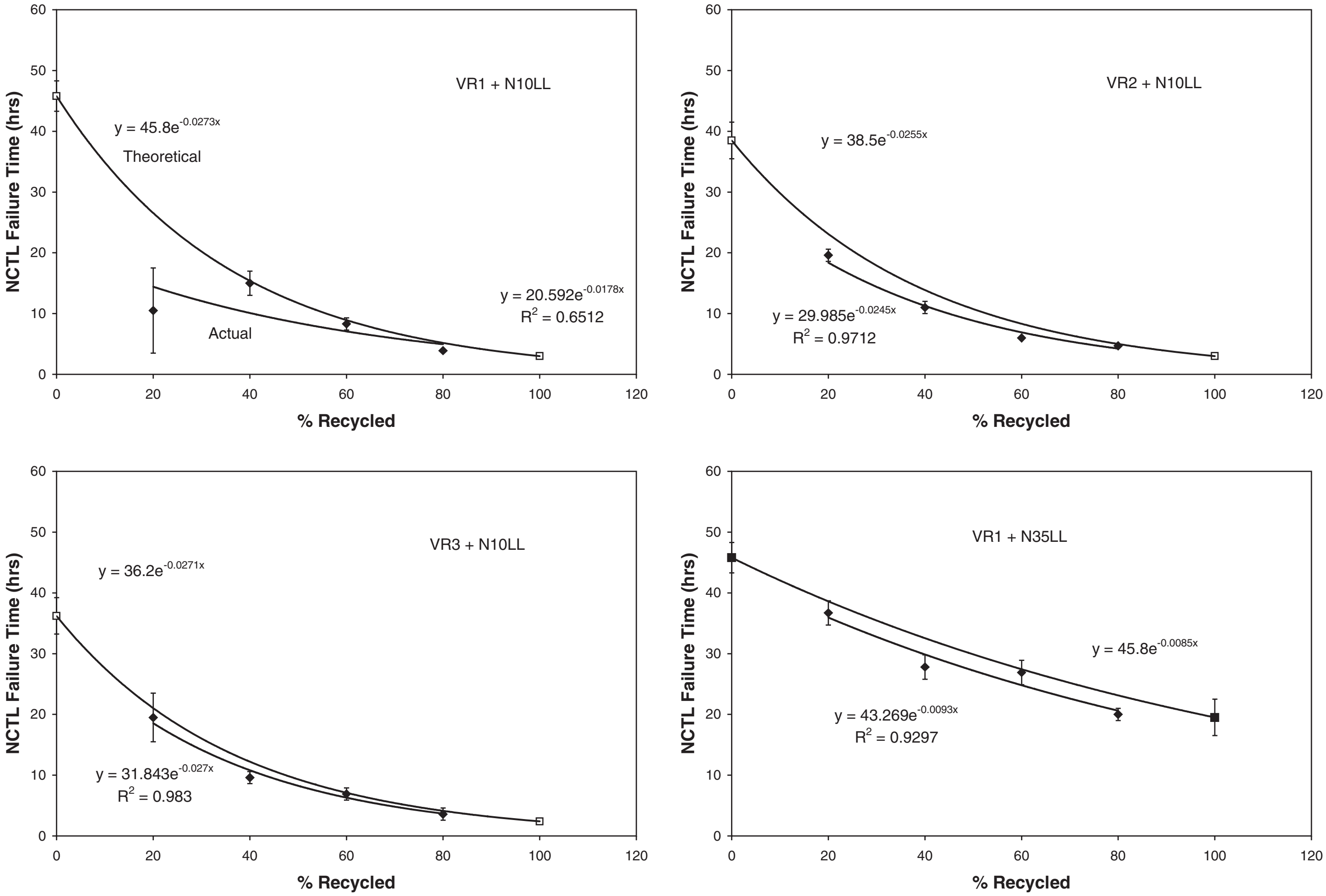

Figure 33. The effect of recycled content on the 15% NCTL of PCR-NAT blends.

with similar resins with 100 or 150 h of NCLS time would allow for much more recycled content to be used.

Additionally, there are new bi-modal resins with densities that meet M294 requirements and that have thousands of hours of stress-crack resistance. It would be valuable to see the effects of adding these resins to recycled content.

Phase 3—Pipe Made from Recycled-Resin Blends

Trial Pipe Manufacturing

A total of 15 trial pipe samples were prepared at three different manufacturing plants, designated Plants A, B. and L. Five, 20-feet-long samples were made from each formulation for a total of 1,500 feet of pipe. The formulations made at the three plants are shown in Table 8.

Each of the three plants used the same lots of PCR-MCR and PCR-NAT from a single supplier. Each plant used a different lot of the VR1 and each plant used their own carbon black master batch. The MDPE used was also from the same lot.

Each plant made a sample from 100% virgin resin 1 and a 50/20/30 blend of VR1, MDPE, and MCR.

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.

Short-Term Properties

Index Test Results

After the 15 sample 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 15°C/min. Complete reports are given for each pipe formulation in Appendix D, Section D.11. This section will focus on specific properties that may be important for future specifications. A summary of the short-term properties is shown in Table 9.

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 accord-

Table 8. Trial pipe formulations.

Sample	% VR1	% Virgin LMDPE	% PCR-MCR	% PCR-NAT
A1	100	0		
A2	85	0	15	0
A3	85	0	0	15
A4	50	20	30	0
A5	40	30	0	30
B1	100	0	0	0
B2	50	20	30	0
B3	20	40	24	16
B4	0	50		50
B5	0	40	36	24
L1[1]	0	0	0	0
L2	50 (VR2)	20	30	0
L3	100 (VR2)	0	0	0
L4	100	0	0	0
L5	50	20	30	0

[1] Proprietary formulated pipe resin containing about 50% recycled content.

ing to ASTM D3350. When directly measured, all but four of the samples had densities higher than the upper limit of 0.955 g/cm^3. This was caused by the presence of carbon black and particles that are certainly denser than HDPE. However, according to ASTM D3350, these values can be corrected for the percentage carbon black, according to the relationship $D_{corr} = D - 0.0044C$, where C is the percentage carbon 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

$$\text{Yield Stress} = 81{,}250 \times \text{Density} - 73{,}500$$

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

Both values are shown in Table 9. It is believed that the density measured in a gradient density column and corrected for percentage carbon black is inaccurate because of the presence of particles and PP. Their presence will influence the measured value. Also, the direct density measurement might be influenced by the very small size of the test specimen. A piece around 1⁄16 in. × 1⁄16 in. is often the size tested. If this small

Table 9. Short-term properties for 15 trial pipe samples.[1]

Property	Sample														
	A1	A2	A3	A4	A5	B1	B2	B3	B4	B5	L1	L2	L3	L4	L5
Density[2] (g/cm^3)	0.952	0.943	0.963	0.952	0.948	0.951	0.948	0.948	0.948	0.952	0.950	0.950	0.952	0.946	0.946
Density[3] (g/cm^3)	0.952	0.951	0.952	0.950	0.948	0.952	0.949	0.945	0.948	0.945	0.949	0.951	0.955	0.955	0.953
Melt Index (g/10 min)	0.12	0.15	0.16	0.19	0.21	0.13	0.22	0.27	0.36	0.34	0.20	0.33	0.36	0.12	0.17
% Carbon Black	1.6	1.4	2.2	0.5	1.5	2.0	2.0	1.9	1.4	1.9	2.1	1.5	1.5	2.5	2.6
% Ash	0.1	0.3	0.2	0.4	0.2	0.0	0.4	0.3	0.1	0.4	0.7	0.4	0.0	0.0	0.3
% PP	0.0	0.7	0.2	1.4	0.3	0.0	1.9	1.7	1.0	2.2	2.5	2.4	0.0	0.0	1.8
Yield Stress (psi)	3851	3775	3863	3710	3623	3865	3638	3251	3523	3311	3631	3751	4079	4145	3922
% Break Strain	183	168	139	241	159	163	343	300	190	351	277	195	252	93	108
OIT (min)	49	39	38	32	96	49	54	93	110	75	13	78	78	70	78

[1] All properties measured on compression molded plaques made from the pipe.

[2] Measured density corrected for percentage carbon black.

[3] Density calculated from yield stress.

piece contains a particle or an air bubble, the number could be skewed in either direction.

For this reason, it is believed that for a specification of resin blends containing PCR-HDPE, the density is of very limited value. It would be better to simply specify mechanical properties that demonstrate what the density is. Two examples are shown in Table 10.

These are the required mechanical property values to ensure that the base resin density was in the range specified by AASHTO M294. Unfortunately, the yield stress and flexural modulus values do not fit neatly into cell classes themselves. Cell class 5 for yield stress is 3,500–4,000 psi, so one could specify a cell class of 5 or higher. If the density were too high, the stress-crack resistance would suffer from too much homopolymer. 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 3,500 psi.

The MI values were consistent and very close to the theoretical values, except in a few cases. This suggested that, for the most part, the pipe formulations were made correctly.

The percentage carbon black results were a little surprising. Each manufacturer was asked to control the carbon content to 2% to 3%. The results showed that only six samples had the correct amount of carbon black, the others were all less than 2.0%. It should be noted that AASHTO M294 allows from 2% to 5%, so the manufacturers were asked to do something they normally do not do.

The percentage ash and percentage PP were both close to the theoretical values with the percentage ash having a linear correlation coefficient (R^2) of 0.75 and the percentage PP's was 0.91.

The yield stress values showed that all the samples were above 3,500 psi except samples B3 and B5. These two were slightly lower and actually were very close to their calculated values. Either of these could easily be adjusted higher with added virgin pipe resin or recycled PE homopolymer.

The percentage strain-at-break values were all much lower than expected. Apparently, the carbon black has a large effect on the break strain. This is one area that should be looked at because, historically, there has been little attention paid to carbon black for corrugated pipe. It is a very important aspect for solid-wall pipe and PE resins used for geomembranes. Specifications for recycled materials could improve the quality of the carbon blacks used simply by setting a high standard for break strain.

The OIT values were consistently above 25 min, except for pipe sample L1, which was 13 min. This was a proprietary blend from a single supplier, an example of a fully formulated resin one might obtain from recycled-resin suppliers. In this case, the OIT was below a suggested value of 25 min. The values for the blends chosen for this study varied based on the virgin resin content, because both the HDPE and LLDPE resins were well stabilized.

In conclusion, the main findings concerning the short-term properties of the trial pipes were the following:

1. A direct measurement of the density of pipe containing recycled content is of limited value, even when the result is corrected for percentage carbon black.
2. The percentages of carbon black were below 2.0% in nine of 15 samples, which might suggest a lack of control by the corrugated pipe manufacturers.
3. The break strains were significantly lower than predicted on blends without carbon black. This could be caused by poor carbon blending, the quality of the carbon black, or even the quality of the carrier resin in the carbon black concentrate.

Stress-Crack Test Results

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), in which each sample is loaded at a constant ligament stress of 600 psi. The 600 psi is a result of taking 15% of 4,000 psi, which is about the yield stress of the PPI-certified resins for corrugated pipe. So, the test basically assumes that most of the samples tested will have a yield stress near 4,000 psi. The NCTL test (ASTM D5397, Appendix A, Section A.3) applies a load based on the actual yield stress of the material tested. Since the yield stresses measured on the 15 pipes ranged from 3,251 to 4,145 psi, many of the samples were not very close to 4,000 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 34. 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 4,000 psi, so the 600 psi is a higher load

Table 10. 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)	3,500–4,100
Flexural Modulus (psi)	130,000–160,000

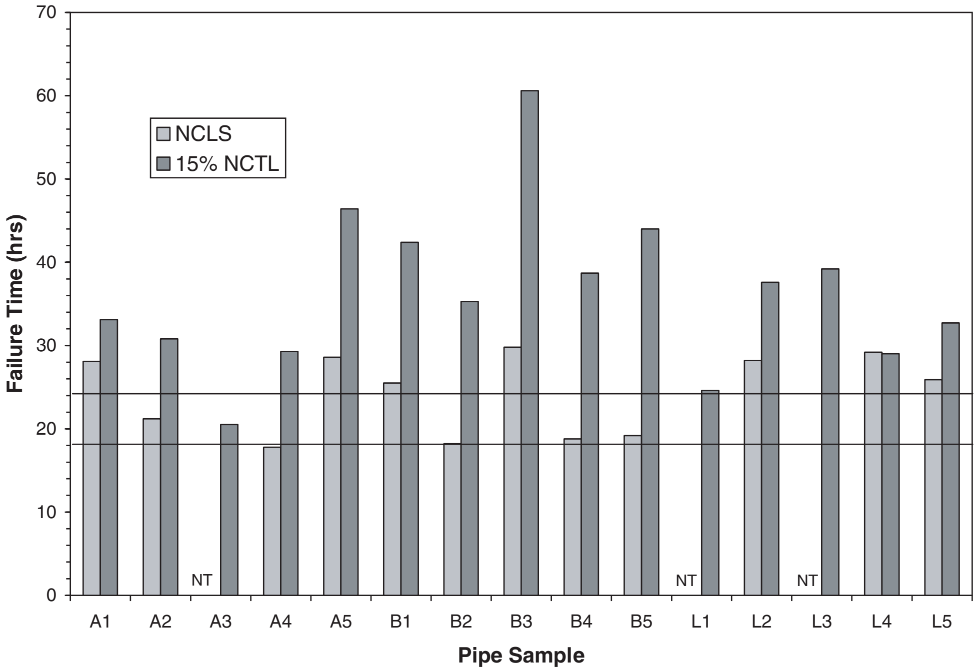

Figure 34. Stress-crack resistance of 15 pipe samples (NT = no test).

than 15% of the actual yield stress. Two lines are shown on the graph. The higher one is at 24 h, which is the required NCLS time for virgin blends used to make AASHTO M294 pipe. The lower line is 18 h, which has been suggested for pipe samples made into plaques (*14*).

Three of the samples tested failed to meet the 18 h requirement. Also, the value for plaques will soon be a minimum of 24 h. Eight of the 15 met 24 h and seven were lower than 24 h.

BFF Test. A test has been developed that combines some features from the BAM test with the criteria set in the FL-DOT 100-year durability assessment (*3*). The test is a stress-crack test without a notch. Whenever there is a critically sized defect present, a crack will initiate at the defect and ultimately create a running crack through the thickness and width of the test specimen. This test is important for blends containing recycled materials because it is very sensitive to contaminants.

It is complimentary to the FL-DOT junction test because it can be performed on the same equipment and is run under the same conditions. The FL-DOT 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 D.I. water at 80°C. Samples cut directly from pipe are evaluated under the conditions in Table 11.

The results are then used with the 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 11 after 110, 430, and 500 h, respectively.

The BFF test uses a larger test specimen, namely an ASTM D638 Type I dumbbell. The mold in which the plaques are made for this test has been modified to make the ends of the specimen thicker than the reduced section of the specimen. A drawing of the specimen is shown in Figure 35 and a picture of the head in Figure 36.

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

Table 11. FL-DOT junction test conditions.

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

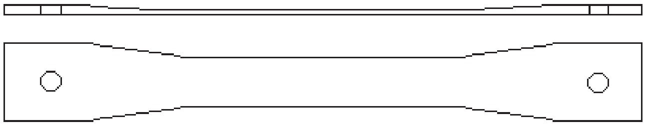

Figure 35. The fathead test specimen.

times the thickness of the reduced width section. The use of this test specimen has essentially eliminated grip failures.

Figures 37 and 38 show pictures of both the FL-DOT Junction Specimen and the BFF specimen.

Tests have been performed on compression molded plaques from each of the 15 trial pipes. Preliminary results are presented in Table 12.

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

The failure surfaces were examined to determine exactly where the specimens broke. There were five categories used to characterize the fracture face. They were the following:

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.

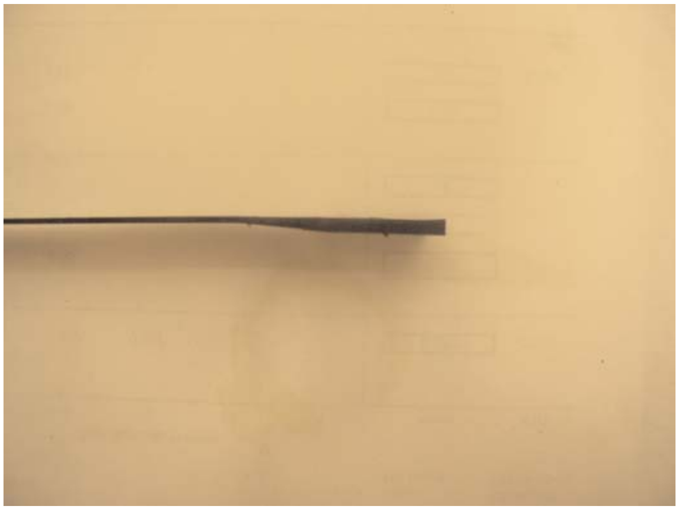

Figure 36. Side view of fathead specimen.

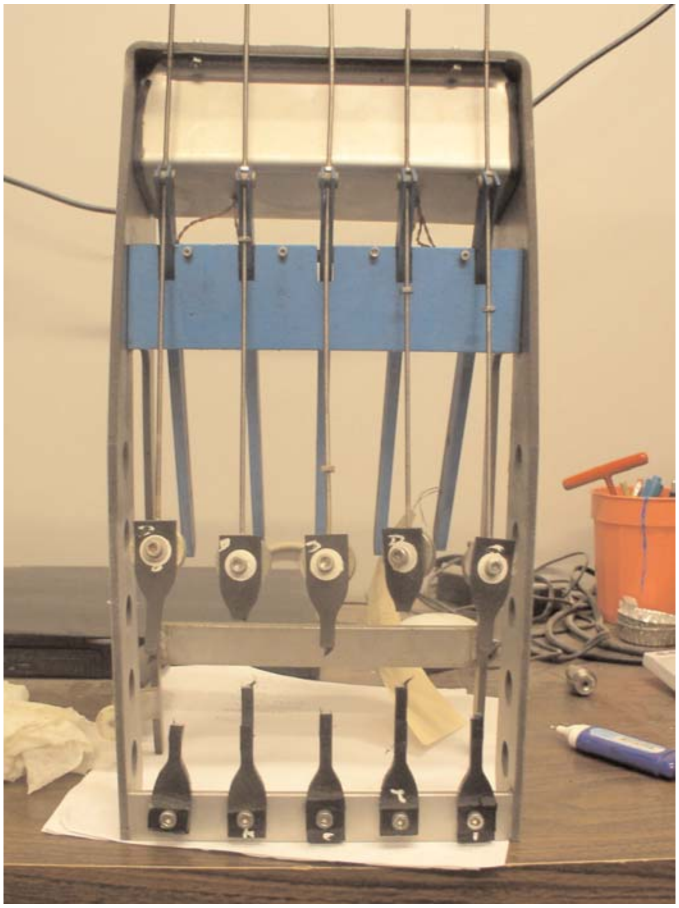

Figure 37. Junction specimen.

Examination of fracture faces from the tests reported in Table 12 produced the following breakdown:

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

Rubber particles are involved in at least 48% of the cracks observed.

BFF Test Reproducibility. It became clear early on that the BFF test results were influenced by residual surfactants in the exposure bath, reflected in the increasing failure times over time. 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 13. 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.

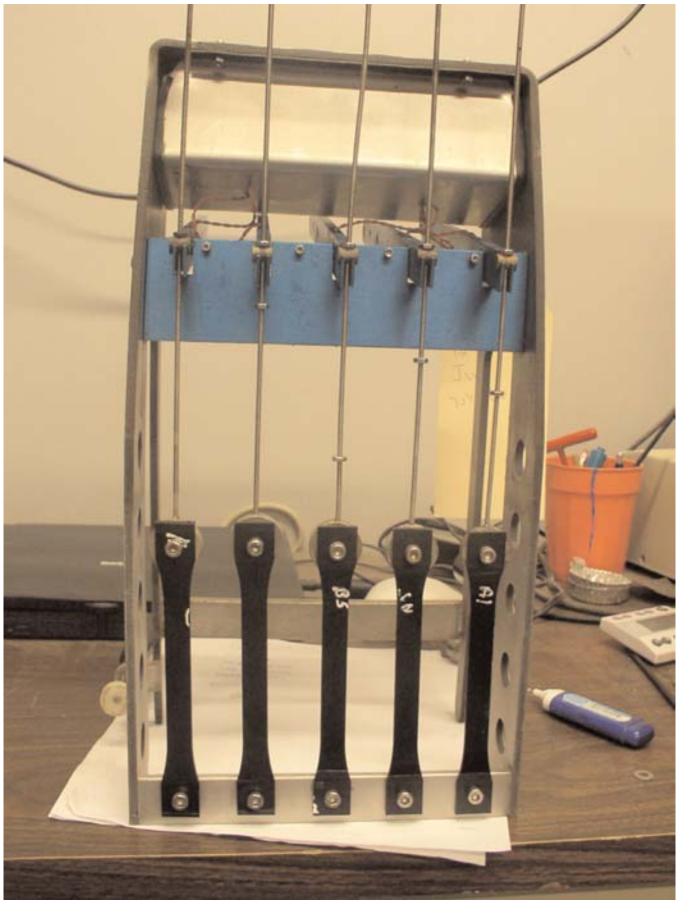

Figure 38. Fathead specimen.

AASHTO M294 Properties

It is useful to see how the trial pipe formulations performed as measured by the AASHTO M294 requirements of a cell class of 435400, and the additional requirements of a percentage carbon black between 2% and 5%, a NCLS value on a plaque from the pipe of 18 h, and an OITemp of 220°C. The results for the 15 pipe samples are shown in Table 14.

There were six samples that did not meet the density requirement when the measured density was corrected for carbon black. This is an issue with the contaminants interfering with the accuracy of the test. If one uses the yield stress to calculate the density, only two samples, B3 and B5, do not meet the requirement, and these were designed to be lower in density.

There were eight samples that were low in carbon black, but this was a manufacturing mistake that can be easily adjusted.

And, finally, there were two samples that did not meet the suggested 10 h of NCLS time on molded plaques from pipe. If one applies the new criteria of 24 h on plaques from pipe, then six of the 15 would not comply. It is clear from these results that more attention needs to be placed on the NCLS test when developing recycled resins for corrugated pipe. There is little doubt that stress-crack resistance is the key property for the successful use of recycled materials in corrugated pipe.

Long-Term Properties

The results of the short-term tests have shown that it will not be too difficult to make blends with recycled contents over 50% that will meet the property requirements of AASHTO M294. By far, the biggest challenge with recycled materials is controlling and minimizing the effects of contamination. Once more, the effects of contaminants will not be seen in short-term tests, but instead will present themselves through a limited service lifetime of the pipe. This makes an understanding of the long-term properties of the pipe critical to the successful use of recycled resins in pipe.

Since these are all longer-term tests, not all of the 15 pipe formulations could be evaluated. Therefore, six samples were selected based mainly on the percentage of recycled content and the type of recycled content (colored or natural). The formulations selected were as follows:

- 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 content, and there is one formulation with only natural recycled content. Some of their properties are given in Table 15. The properties were measured on compression-molded plaques from the pipes.

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 (shown in bold in Table 15).

Long-Term Tensile Strength by SIM

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

The test specimens were placed under the appropriate load then a series of 10,000 second (166 min) creep rupture tests were performed on the same specimen and separated by 7°C

Table 12. BFF test results on 15 pipe samples at 80°C/650 psi in D.I. water.

Sample	Formulation	Failure Time (h) (COV)
A1	100% VR1	175 ± 25 (14%)
A2	85% VR1 + 15% MCR	157 ± 45 (29%)
A3	85% VR1 + 15% NAT	123 ± 17 (14%)
A4	50% VR1 + 20% MD + 30% MCR	130 ± 35 (27%)
A5	40% VR1 + 30% MD + 30% NAT	245 ± 11 (4%)
B1	100% VR1	188 ± 36 (19%)
B2	50% VR1 + 20% MD + 30% MCR	155 ± 62 (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 ± 35 (19%)
L5	50% VR1 + 20% MD + 30% MCR	190 ± 53 (28%)

Table 13. Effect of residual surfactant on BFF failure times.

Sample/ Formulation	Time to Failure (h) at 80ºC/650 psi (COV)			
	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%)

Note: "—" = data not available.

Table 14. AASHTO M294 requirements for 15 trial pipe samples.

Sample	Density[1]	MFI	Flexural Modulus	Yield Stress	% Carbon Black	NCLS	OITemp
M294	**4**	**≥3**	**≥5**	**≥4**	**2–5%**	**>18 hrs**	**>220°C**
A1	4	4	5	5	1.6	28	257
A2	3	3	5	5	1.4	21	256
A3	5	3	5	5	2.2	<18	257
A4	4	3	5	5	0.5	18	256
A5	4	3	5	5	1.5	29	260
B1	4	4	5	5	2.0	26	253
B2	4	3	5	5	2.0	18	253
B3	3	3	5	4	2.0	30	256
B4	4	3	5	5	1.4	19	257
B5	3	3	5	4	1.9	19	256
L1	4	3	5	5	2.1	<18	240
L2	4	3	5	5	1.5	28	256
L3	4	4	5	6	1.5	39	256
L4	3	3	5	6	2.5	29	254
L5	3		5	5	2.6	26	256

[1] Measured density corrected for percentage carbon black.
Note: MFI = melt flow index.

Table 15. Select index properties of final six candidate formulations.

Property	Sample					
	B1	A2	L5	A5	B3	B5
Density[1] (g/cm^3)	0.951	**0.943**	**0.946**	0.948	0.948	0.952
MI (g/10 min)	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 (psi)	148,210	152,607	142,618	140,065	128,361	128,758
Yield (psi)	3,865	3,775	3,922	3,623	3,251	3,311
Break Strain (%)	165	168	108	159	300	351
NCLS (h)	25.5	**21.2**	25.9	28.6	29.8	**19.2**
OIT (mib)	49	39	78	96	93	75
BFF Test[2] (h)	411	257	382	323	330	254

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

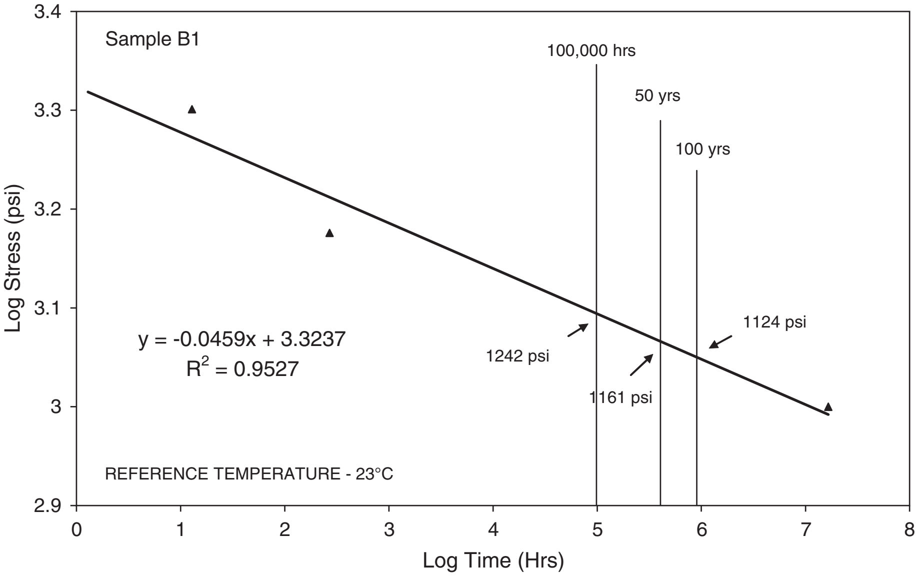

Figure 39. Long-term yield strength of Sample B1.

temperature steps. The test was continued until the specimen yielded. The results from the three 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 39. Tabulated results for the six candidate formulations are shown in Table 16.

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 time-dependent tensile strength. 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 1,000 psi, the material will not fail in a ductile manner. The long-term tensile strength is largely governed by the short-term strength. Therefore, to ensure a 1,000 psi yield strength after 50 years, the short-term strength should be over 3,500 psi.

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

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 nonreversible creep actually begins.

Table 16. Long-term tensile stress of six formulations.

Sample/ Formulation	Long Term Tensile Stress (psi)		
	100,000 h	50 years	100 years
B1 100% VR1	1,242	1,161	1,124
A2 85% VR1 + 15% MCR	1,227	1,145	1,108
L5 50% VR1 + 20% MD + 30% MCR	1,184	1,103	1,067
A5 40% VR1 + 30% MD + 30% NAT	1,192	1,105	1,066
B3 20% VR1 + 40% MD + 24% MCR + 16 NAT	1,064	973	934
B5 40% MD + 36% MCR + 24% NAT	1,164	1,082	1,045

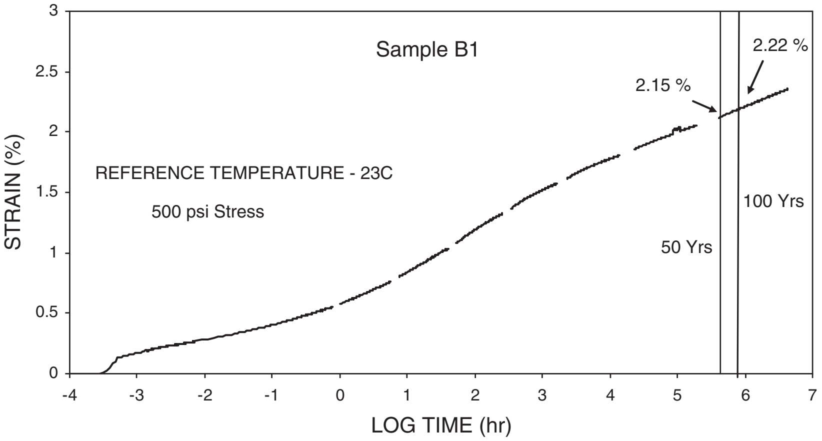

Figure 40. Creep strain master curve for Sample B1.

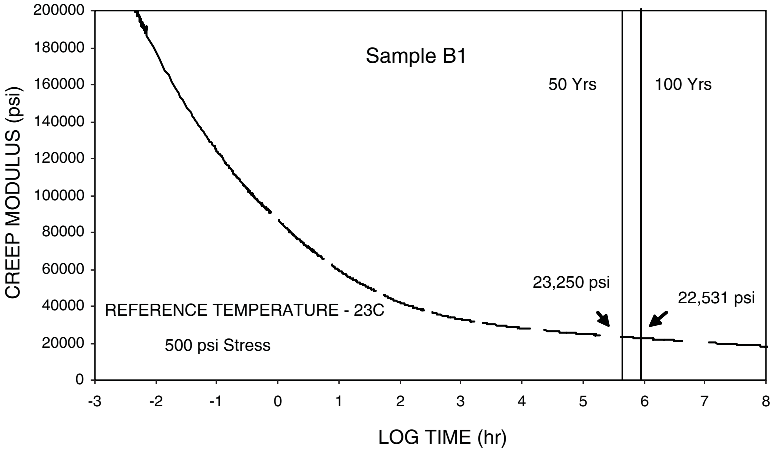

Figure 41. Creep modulus master curve for Sample B1.

Next, the 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°C to 83°C. The raw data are then shifted through 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 40 and 41, and all of the results are shown in Table 17.

The raw data and the master creep curves for these samples are found in Appendix D, 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 LRFD Bridge Design Specifications* (*15*).

Long-Term Stress-Crack Resistance

The six final candidate formulations and the PCR MCR resin were evaluated by the BFF test under the three sets of conditions

Table 17. Long-term creep strain and creep modulus under 500 psi of stress.

	Creep Strain		Creep Modulus	
Sample	50 year	100 year	50 year	100 year
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

Table 18. Recent BFF test results.

Sample/Formulation	BFF Failure Time (h) (COV)		
	80ºC/650 psi	80ºC/465 psi	70ºC/650 psi
B1 100VR1	411 ± 135 (33%)	1,601 ± 537 (34%)	1,627 ± 680 (42%)
A2 85VR1 + 15MCR	257 ± 85 (33%)	—	—
L5 50VR1 + 20MD + 30MCR	431 ± 86 (20%)	—	—
A5 40VR1+ 30MD + 30NAT	323 ± 60 (19%)	1,364 ± 571 (42%)	1,615 ± 644 (40%)
B3 20VR1+40MD+24MCR+16NAT	330 ± 66 (20%)	—	—
B5 40MD + 36MCR + 24NAT	280 ± 95 (34%)	—	—
L1 Proprietary Blend – 50% Recycled content	201 ± 39 (19%)	—	—
RPM Proprietary Blend 100% Recycled content	620 ± 376 (61%)	1,480, 4>3,000[1]	—

[1] Four specimens terminated after 3,000 h.
Note: "—" = data not available.

cited in the FDOT 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 eight samples, under two sets of conditions for three samples, and under three sets of conditions for two samples. The results are shown in Table 18.

Notice that a new sample was evaluated. This material is a fully formulated pipe blend containing 100% PIR recycled and PCR PE, submitted by a recycled-resin supplier. 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 19.

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 percentage 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 bidirectional shifting. The master curves for stress-crack resistance for Samples B1 (100% Virgin) and A5 (30% Recycled) are shown in Figures 42 and 43.

These results suggest that the service lifetime under 500 psi of stress will be well above 1,000 years and that even at stresses of 900 psi, the estimated service lifetime is over 100 years.

Combined SIM (Stage I) and BFF (Stage II) Service-Lifetime Estimates

The SIM test produced information about the long-term tensile strength and the BFF test provided information about the long-term stress-crack resistance. Now, the two results can be combined into one global service-lifetime estimate. This is shown in Figures 44 for Sample B1.

This curve shows both the long-term tensile strength that might relate to a buckling failure and the long-term stress-cracking strength. The first thing to keep in mind is these data

Table 19. 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	3,260 psi
Break Strain	21%
NCLS Stress-crack Resistance	220 ± 54 hrs

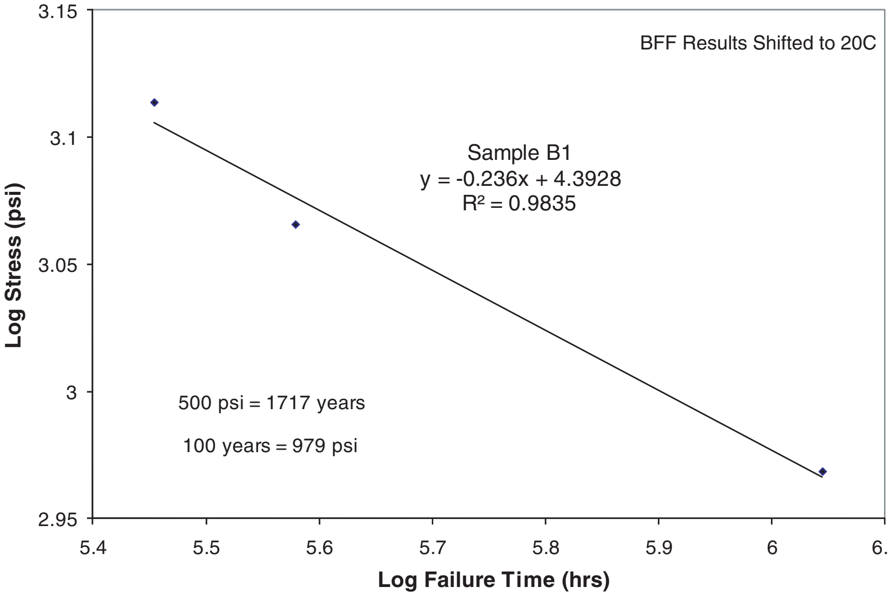

Figure 42. BFF stress-cracking master curve at 20°C for sample B1.

were generated on tensile dumbbell specimens with very simple geometry. A pipe is likely to behave differently. Secondly, if these data relate to pipe, they relate to an *unconfined* pipe. A properly installed pipe would also be a different situation. That said, there is still valuable information contained in the master curves.

The curve shows a ductile-to-brittle transition stress of 1,153 psi. This is just above 30% of the material's initial yield stress. This also means that at applied stresses less than 1,153 psi, the pipe is more likely to crack than to buckle. In fact, as most involved in this project thought, slow crack growth is the key to service lifetime in corrugated pipe, according to these results.

The curve also shows that even at the very high service stress of 900 psi, the estimated lifetime is over 100 years. Once more, at a typical operating stress of 500 psi, the service lifetime estimate is over 1,500 years.

A similar master curve is shown for Sample A5 in Figure 45.

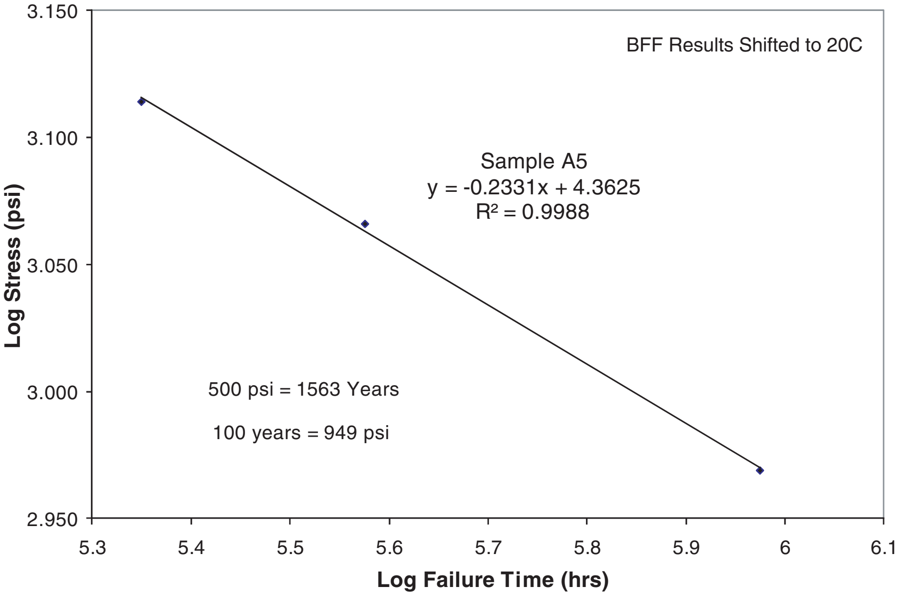

Figure 43. BFF stress-cracking master curve at 20°C for sample A5.

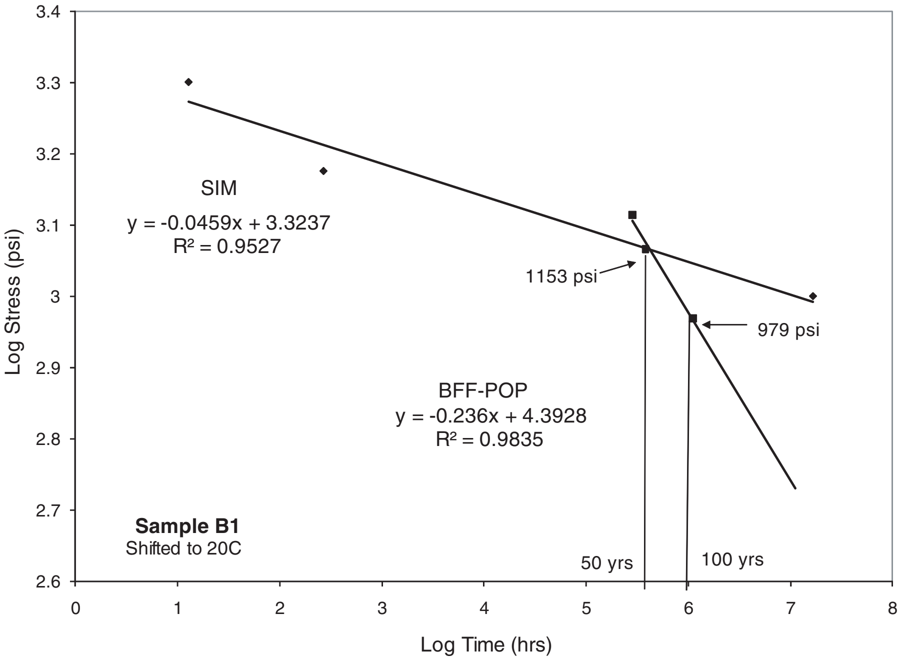

Figure 44. Combined master curve for Sample B1 (100% virgin).

Sample A5 contained 30% PCR-NAT, which is basically ground up milk and water jugs. The service-lifetime estimates are similar to 100% virgin. Because of limitations of time and funding, more of the recycled-content-containing pipes were not evaluated.

One important aspect here is to define the "long-term strength." The long-term strength referred to in Section 12 of the LRFD design manual is a long-term yield strength. The results generated during this project suggest that the long-term stress-crack resistance is more important and will limit the lifetime of the pipe. So, it might be more appropriate to determine a long-term stress-crack strength, which is the stress a material can be subjected to without cracking for its service lifetime. In the two examples above, the 100-year tensile

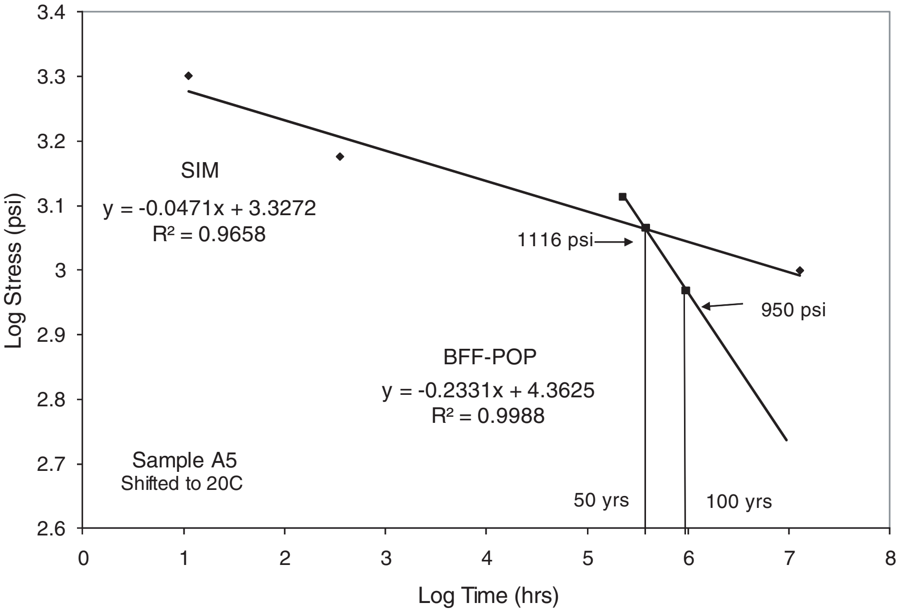

Figure 45. Combined master curve for Sample A5 (30% recycled).

Table 20. Comparison between BFF and junction tests at 80°C/650 psi.

Sample	BFF Time (h) (COV)	Junction Time (h) (COV) [liner breaks]	Junction/BFF
B1	414 ± 97 (23%)	1,076 ± 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%)	1,342 ± 300 (22%) [4]	4.0

strengths are 1,124 and 1,067 psi, while the 100 year stress-crack strengths are 979 psi and 950 psi for samples B1 and A5, respectively.

The BFF Test for QC

There is no question that the successful use of recycled PE 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 QC 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 FL-DOT junction test does. 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 80°C/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 20.

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 FL-DOT 100-year service-lifetime protocol calls for the junction test to be run under three sets of conditions and sets minimum time requirements for each condition. These are shown in Table 21.

These values were determined with the use of POP factors and the 95% lower confidence interval based on a Student-t distribution. When one applies the Popelar shifts, the 20°C master curve can be generated. This is shown in Figure 46.

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

Now, since there is uncertainty regarding the use of recycled PE, 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 1,000 years. The master curve for 1,000 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 (*16*), Hsuan (*17*), and this study on 17 data sets was −0.26, with only four values over −0.27.

The minimum FL-DOT times to generate the 1,000-year lifetime curve are shown in Table 22.

This shows that if a junction specimen lasts 360 h without failure in a 80°C bath under 4.48 MPa (650 psi) of load, it will be estimated to last 1,000 years.

Since it has been demonstrated that the BFF test is at least 1.7 times faster than the junction test at 80°C/4.48 MPa (650 psi), it would be conservative to set the requirement for the BFF test at 200 h. Therefore, the proposed minimum average failure time in the single point BFF test for QC is 200 h, or 8.3 days. This is not an ideal time for a QC/quality assurance (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 h. 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 42 and 43 independently support the value of 200 h for a QC test at

Table 21. FL-DOT junction test conditions and minimum failure times.

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

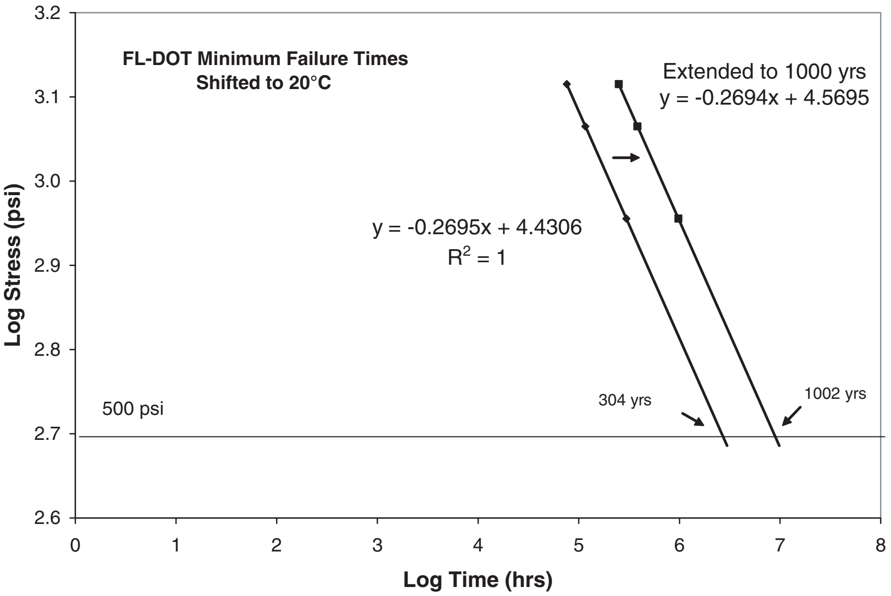

Figure 46. Master curve from FL-DOT minimum times.

80°C/650 psi. From the slopes of those lines and shifting back to 80°C, the time that represents 1,000 years of service for both samples is 200 h.

Designing Pipe with Recycled Content

An evaluation of the existing design methodology outlined in Section 12 of the *AASHTO LRFD Bridge Design Specifications* was performed to determine its applicability to pipe manufactured with blends of virgin and recycled HDPE. In particular, the material properties needed to meet M294 required for the AASHTO LRFD design methodology were assessed.

The design methodology in Section 12 of the *AASHTO LRFD Bridge Design Specifications* (and McGrath et al.'s recent recommendations in *NCHRP Report 631: Updated Test and Design Methods for Thermoplastic Drainage Pipe* (*13*) evaluates local buckling resistance to check the structural capacity of profile wall plastic pipe. The total factored compressive and tensile strains in a pipe wall due to thrust and bending are evaluated to ensure that specified strain limits are not exceeded. Bending strain is also evaluated. The methodology also incorporates a vertical arching factor to account for load relief when the pipe is less stiff than the surrounding backfill.

Table 22. Minimum times for a 1000-year estimated lifetime.

Test Conditions	Minimum Failure Time
80ºC/4.48 MPa (650 psi)	360 h
80ºC/3.10 MPa (450 psi)	1,401 h
70ºC/4.48 MPa (650 psi)	1,647 h

Table 12.12.3.3-1 Mechanical Properties of Thermoplastics Pipe in *AASHTO LRFD Bridge Design Specifications* Section 12 sets forth the requirements for the minimum cell class, the initial and 50-year design tensile strength and the initial 50-year modulus of elasticity. Recently, McGrath et al.'s *NCHRP Report 631* from project NCHRP Project 04-26, "Thermoplastic Drainage Pipe, Design and Testing" recommends 75-year design tensile strength and modulus of elasticity requirements as well as service long-term tension strain and factored compression strain limits. Note that the estimated modulus of elasticity values proposed in *NCHRP Report 631* for *AASHTO LRFD Bridge Design Specifications* Section 12 are based on observed field performance of pipe designed with 50-year modulus and relaxation tests on PE pipe in parallel plate tests to estimate 75-year modulus. Values are reported to be reasonably conservative.

The material properties studied in this work included the minimum cell class (density, MI, flexural modulus, tensile yield stress), NCLS, NCTL, thermal stability, and oxidation induction time. Long-term properties (50 and 100 years) included creep modulus, creep strain, yield stress, stress-crack stress, service long-term tension strain and factored compressive strain limit. Properties based on parallel plate tests on finished pipe products included pipe stiffness, flattening, environmental stress-cracking resistance (ESCR), and brittleness.

The laboratory results demonstrated that pipe can be made with blends of virgin and recycled HDPE to meet AASHTO M294. Three (A5, B1, and L5) of the six final candidate formulations met the required cell class for AASHTO M294. Pipe A2 met all requirements except for NCLS. Pipe B3 met all requirements except for the density requirement. Pipe B5 met all except for the density and NCLS requirements.

Five of the six pipes made from the final candidate formulations met the *NCHRP Report 631* recommendation for creep modulus and yield stress (long term property requirements). Note that the long-term tests were performed on plaques made from manufactured pipe. This was done to erase all previous stress due to manufacturing to facilitate the comparison of the blended resins without the influence of differences in manufacturing (such as manufacturing speed). Additional tests should be performed on specimens cut directly from the pipe.

Parallel plate tests were performed on the finished pipe product made from the six final candidate formulations. The results indicate that all six final candidate formulations meet the pipe stiffness requirement of AASHTO M294.

For the pipes herein made with blends of virgin and recycled HDPE that have material properties meeting the requirements in Table 12.12.3.3-1, current AASHTO LRFD Section 12 design specifications should apply; however, long-term in-situ load tests and accelerated laboratory tests on finished pipe product embedded in soil are needed to finalize the proposed changes to the AASHTO LRFD design methodology (i.e., structural design equations for thrust, buckling, and bending and resistance factors based on field performance data).

Proposed Draft Specifications

Currently, both AASHTO M252 and M294 have property requirements for both the virgin resins used to make the pipe and the pipe itself. And the National Transportation Product Evaluation Program (NTPEP) requires the use of a PPI-certified resin, or the testing of each different blend used to make pipe. It is well known that some pipe manufacturers make their own virgin blends, while others prefer to use certified and listed resins.

A similar situation would exist if AASHTO allows the use of recycled material containing resins to make pipe. Some manufacturers will prepare their own blends and others will choose to purchase fully compounded pipe resins already containing recycled HDPE. For this reason, it is suggested that there should be both required resin properties and required pipe properties in the specifications for M252 and M294 applications allowing recycled HDPE. It is also suggested that the specifications move away from listing cell classification by ASTM D3350. Cell classification is a living standard subject to continuous revisions, which have affected the AASHTO standards in the past. Also, there are tests in the cell classification that may not be necessary in D3350 and tests that are necessary but are not in D3350 (NCLS, OIT). So, instead of a required cell class, the standards could simply have a table of required properties, like is shown in Table 23.

Table 23. Properties for AASHTO M294 pipe resin (cell class 335500).

Property	Requirement
Density	0.948-0.955 g/cm^3
MI	<0.4 g/10 min
% Carbon Black	2%–5%
Yield Stress	≥3,500 psi
Flexural Modulus	≥110,000 psi
NCLS Stress Crack	>24 h
OIT	>50 min

Ultimately, all the resin tests should be performed on compression molded plaques from the pipe itself, but resin specifications will allow independent compounders and pipe manufacturers to make appropriate blends and control their properties. Once more, a specification on the recycled material itself will be beneficial to resin compounders and pipe manufacturers. Therefore, five different specifications are proposed and presented in Appendices E through I. The titles and scopes of each one are shown below.

Appendix E—Proposed Draft Standard Specification for PCR MCR High-Density PE Bottles for Use in AASHTO-Approved Corrugated Drainage Pipe

1. **Scope**
   1.1 This specification covers the quality of PCR, MCR, high-density PE bottles for use in AASHTO approved corrugated drainage pipe.
   1.2 This specification presents a set of properties to be met for the resin.
   1.3 This specification can be used by recycled-resin suppliers as part of manufacturing quality control (MQC), or by pipe manufacturers or independent bodies as manufacturing quality assurance (MQA).

Appendix F—Proposed Draft Standard for Recycled Content Containing HDPE Resin Formulations for Corrugated Pipe Made to AASHTO Standard M252-Recycled

1. **Scope**
   1.1 This specification covers the quality of HDPE-resin formulations containing recycled HDPE that are intended for use in AASHTO M252—Recycled approved corrugated drainage pipe for subsurface drainage applications, in sizes 75- to 250-mm diameter (3 to 10 in.).
   1.2 The recycled PE may be either post-consumer or post-industrial.

1.3 This specification presents a set of properties and test frequencies to be met for the resin.

1.4 This specification can be used by suppliers of resin containing recycled PE as part of MQC, or by pipe manufacturers or independent bodies as MQA.

Appendix G—Proposed Draft Standard Specification for Recycled Content Containing HDPE Resin Formulations for Corrugated Pipe Made to AASHTO Standard M294-Recycled

1. Scope

1.1 This specification covers the quality HDPE-resin formulations containing recycled HDPE that are intended for use in AASHTO M294–Recycled approved corrugated drainage pipe, in sizes 300- to 1500-mm (12- to 60-in.) diameter.

1.2 The recycled PE may be either post-consumer or post-industrial.

1.3 This specification presents a set of properties and test frequencies to be met for the resin.

1.4 This specification can be used by suppliers of resin containing recycled PE as part of MQC, or by pipe manufacturers or independent bodies as MQA.

Appendix H—Proposed Draft Standard Specification for Corrugated Polyethylene Drainage Pipe Containing Recycled Polyethylene, 75- to 250-mm Diameter

1. Scope

1.1 This specification covers the requirements and methods of test for corrugated PE pipe, couplings and fittings for use in subsurface drainage systems, storm sewers, and in surface drainage (culverts), where soil support is given to the pipe's flexible walls in all applications.

1.2 This standard allows for the use of recycled polyethylene in the pipe, but not in the couplings nor the fittings.

1.3 Nominal sizes of 75 mm to 250 mm are included.

1.4 Material properties, dimensions, pipe stiffness, perforations, joining systems, and forms of marking are specified.

Note 1—When PE pipe is to be used in locations where the ends may be exposed, consideration should be given to combustibility of the PE and the deteriorating effects of prolonged exposure to ultraviolet radiation.

1.5 The following precautionary caveat pertains only to the test method portion, Section 9.3, of this specification. *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Appendix I—Proposed Draft Standard Specification for Corrugated Polyethylene Drainage Pipe Containing Recycled Polyethylene, 300- to 1,500-mm Diameter

1. Scope

1.1 This specification covers the requirements and methods of tests for corrugated PE pipe, couplings, and fittings for use in surface and subsurface drainage applications.

1.1.1 This standard allows for the use of recycled PE in the pipe, but not in the couplings nor the fittings.

1.1.2 Nominal sizes of 300 to 1,500 are included.

1.1.3 Materials, workmanship, dimensions, pipe stiffness, slow crack growth resistance, joining systems, brittleness, and forms of markings are specified.

1.2 Corrugated PE pipe is intended for surface and subsurface drainage applications where soil provides support to its flexible walls. Its major use is to collect or convey drainage water by open gravity flow, as culverts, storm drains, and so forth.

Note 2—When PE pipe is to be used in locations where the ends may be exposed, consideration should be given to protection of the exposed portions due to combustibility of the PE and the deteriorating effects of prolonged exposure to ultraviolet radiation.

1.3 This specification does not include requirements for bedding, backfill, or earth cover load. Successful performance of this product depends upon proper type of bedding and backfill, and care in installation. The structural design of corrugated PE pipe and the proper installation procedures are given in *AASHTO LRFD Bridge Design Specifications,* Section 12, and *LRFD Bridge Construction Specifications,* Section 30, respectively. Upon request of the user or engineer, the manufacturer shall provide profile wall section detail required for a full engineering evaluation.

1.4 The following precautionary caveat pertains only to the test method portion, Section 9.4 of this specification. *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

CHAPTER 4

Conclusions and Suggested Research

Phase 1—Recycled PE Resins

The purpose of this phase was to determine which types of recycled PE were available, where these materials could be obtained, and what their properties were. Both PIR and PCR PE were initially examined, but the effort focused on PCR because it is readily available, more consistent than PIR, and there is a trade association called the Association of Post-consumer Plastic Recyclers (APR). PIR should not be ruled out as a source of material, but PCR will be more consistent and more widely available. A total of 25 samples of recycled PE were obtained from nine different suppliers. There were three PIR samples and 22 PCR samples. Of the PCR samples, six were natural and 16 were colored. The natural resins came from milk bottles and the colored mostly from detergent bottles.

The results showed that mixed-color PCR was an excellent candidate for use in corrugated HDPE pipe. The density averages around 0.949 g/cm^3, which is close to virgin pipe resins, so the strength and flexural modulus properties are in the AASHTO specified cell class for M294 pipe. The MI averaged around 0.5 g/10 min while the M294 maximum is 0.4 g/10 min. It also had poor stress-crack resistance and contaminants present that could be stress-crack initiation sites.

A test method was developed to determine the percentage PP in the recycled HDPE. PP is a contaminant that comes from the tops and pour spouts of detergent bottles. It can be found in amounts of 10% or higher. Results showed that the PP content could be as high as 5% without hurting the stress-crack properties of the resin. Results also showed that melt filtration at a mesh size of 120 or greater can reduce the amount of contamination to about 0.5% and raise the elongation-at-break to over 100%. These values will be specified to ensure proper filtration occurs.

The research needs from this phase involve the test method for percentage PE. The method needs to be written into a standard test method and evaluated through a consensus group such as ASTM, or developed through AASHTO. Also, FTIR spectroscopy is an alternative method to measure percentage PP. It should be added to the method as an alternative to the DSC method presented in this report.

Phase 2—Recycled PE Blends

The purpose of Phase 2 was to determine how the addition of recycled HDPE would affect the properties of CPPI-certified resins and to select specific blends for trial corrugated pipe manufacturing. A total of 66 blends were prepared and their properties evaluated. One significant finding was that stress-crack resistance changed in an exponential manner when two different PE materials were blended together. Additionally, the yield strength changed in a linear manner. This means that when one blends two materials with different strengths and stress-crack resistances, the strength will change more quickly than the stress-crack resistance. This also means that one needs to find resins to blend with the recycled resins that have similar or greater strengths and significantly better stress-crack resistances. This project was limited by the original concept of blending recycled resins with PPI-certified corrugated pipe resins. Just since this project began, data has become available about the stress-crack resistance of bimodal HDPE resins. There are at least six companies in the United States that produce resins with densities in cell class 4 (required by AASHTO M294) and that have NCLS values in the thousands of hours. In fact, they are tested with a more aggressive stress-crack test called the PENT Test (ASTM F1473) and some resin suppliers report values greater than 10,000 h.

Besides these “super” resins, a recycled supplier submitted a sample for this study recently that was made from 100% recycled material. Its NCLS stress-crack time was 220 h, and it lasted for over 600 h in the BFF test at 80°C/650 psi when 200 h suggests 1000 years of estimated lifetime.

Research is needed to determine how the addition of these new, bimodal resins will affect the stress-crack resistance of mixed-color PCR. It's possible that the addition of just around 10% of these resins would raise the NCLS times of the recycled above the required value of 24 h. Of course, an unnotched stress-crack test like the BFF test would also be required to assess the long-term performance of the blends.

Phase 3—Pipe Made from Recycled-Resin Blends

The purpose of this phase of the project was to manufacture dual wall corrugated drainage pipe out of resin formulations containing recycled HDPE. Fifteen pipe samples were prepared at three different manufacturing plants. Each plant made two common formulations, so there was a total of 11 different blends evaluated. Once the pipe was made, the short-term properties were measured and compared with the requirements of AASHTO M294. It was determined that it is not too difficult to create blends within the required cell classification required by AASHTO M294. It was more difficult to produce blends with adequate stress-crack resistance. If the 2010 requirement of 24 h on a plaque made from a finished pipe were in place, only eight of the 15 samples would meet the requirement. Again, this is a result of making blends with pipe resin with about 50 h of NCLS time. Resins tested in 2010 have had times greater than 150 h, which would make the resulting blends higher in stress-crack resistance. And, of course, the new bimodal HDPE resins could be used to raise the value even more.

The long-term performance of the pipe formulations was also evaluated. Accelerated creep and creep rupture tests were performed by the SIM on tensile dumbbells taken from compression molded plaques made from pipe. These results showed that the materials all met the requirements of Section 12 of the LRFD Bridge Design Specification for 50- and 75-year service lifetimes.

The long-term stress-crack resistance was determined with a new test called the BFF test. This test was found to be sensitive to contaminant particles, while other tests are not. Results were generated that suggested that recycled content-containing pipe formulations can be developed that have estimated service lifetimes greater than 1000 years. This allows one to apply a very conservative design factor of 0.10 for a 100-year service lifetime.

The long-term oxidation resistance of pipe containing recycled HDPE is often thought to be a significant issue. However, studies over the past 20 years have consistently showed that in the absence of UV radiation, the oxidation rate for modestly stabilized HDPE materials is low. One example is the Pennsylvania Department of Transportation Deep Burial study in which the OIT times were measured after 20 years of buried service. The results showed that very little, if any, change had occurred to the OIT, except on the pipe ends exposed to the sun (*13*). Another is a recent study that showed that an additive package containing 0.05% Irganox 1010 and 0.10% Irgaphos 168 had an estimated antioxidant depletion time of 130 years (*18*). Oxidation failures are believed to be an unlikely event if the formulations are made with a minimum of 0.1% Irganox 1010 and 0.1% Irgaphos 168.

The ultimate goal of this project concerning the long-term properties is to create a master curve, similar to those used for solid wall pressure pipe. An example for Sample B1 is shown in Figure 47.

This curve is an estimate of the relationship between applied stress and service lifetime for a sample made from a

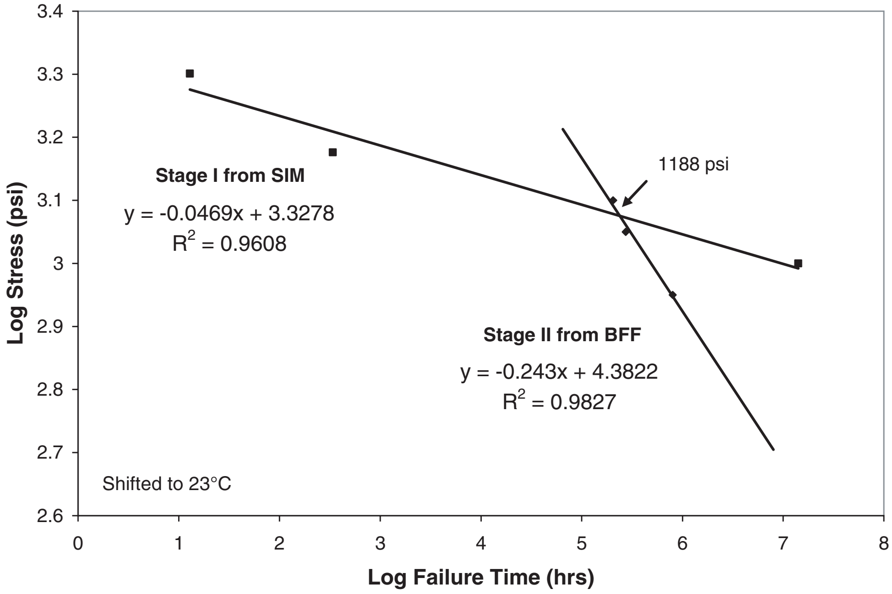

Figure 47. Master curve at 23°C for Sample B1.

PPI-certified, 100% Virgin HDPE. The information in this plot includes the following:

1. At applied loads less than 1,188 psi, the life-limiting failure mechanism is slow crack growth (Stage II).
2. At 900 psi, the lifetime estimate is 86 years.
3. At 500 psi, the lifetime estimate is 965 years.
4. The life-limiting load at 50 years is 1,026 psi
5. The life-limiting load at 75 years is 930 psi.
6. The life-limiting load at 100 years is 867 psi.

This plot was generated from the SIM accelerated creep rupture test and from the BFF accelerated stress-cracking test. Both of these test methods require further development, but the example above should demonstrate the usefulness of the two.

The test methods need standard methods written and round-robin tests conducted. There is interest in a SIM method in ASTM because it is already being specified for some drainage products and the only SIM standard available is for reinforcing geotextiles and geogrids.

The estimates of long-term stress and strain and of long-term stress-crack resistance should be verified through accelerated laboratory testing on the pipes or field studies on buried pipe. There are some specific ways to do this.

SIM tests should be performed on pipe samples to determine the relationship between a dumbbell test on a compression molded plaque and an actual section of pipe. This is easily done on 12-in. diameter pipe. All that is required is an appropriate environmental chamber that can hold a 12-in. × 12-in. piece of pipe and a linear variable differential transformer (LVDT) to measure inside diameter displacement. The pipe sample would be placed between parallel plates and loaded with the appropriate mass. The results of this study will be an estimated service-lifetime curve. This curve will help determine appropriate loads for actual field tests.

For example, if the SIM results on pipe are similar to the results on a dumbbell from a plaque, one could use the estimated lifetime curve to select loads for a field study. The SIM creep rupture curve for sample B1 on a plaque is shown in Figure 48.

Notice that one could apply loads of 40%, 38%, and 36% of the ultimate stress and create failure times in less than one year. The measured buckling load of Pipe B1 was 1,053 lb. So, the corresponding loads would be 442 lb, 421 lb, and 400 lb. These loads should create buckling failures in less than a year. This is the type of experiment that could validate the results of the SIM test on formulations containing recycled HDPE.

Similar experiments on solid wall pressure pipe are ongoing in these laboratories. The SIM results on a plaque are being compared to sustained burst test results at room temperature. The slopes of the log stress vs. log time curve are nearly identical, but the curves are shifted vertically. The pipe failures all occur at higher stresses than the dumbbells. It's a linear offset and believed to be caused by sample geometry. There may also be an offset between a pipe plaque and a pipe in compression, but the slopes of the lines may be the same. If the results between a SIM test on a plaque and long-term compression tests on pipe are comparable, then a powerful new tool would exist for predicting long-term behavior.

In summary, the suggested follow-on research pertaining to the SIM test and long-term service estimates are to do the following:

1. Develop a standardized method specific to performing SIM accelerated creep and creep rupture tests on HDPE.

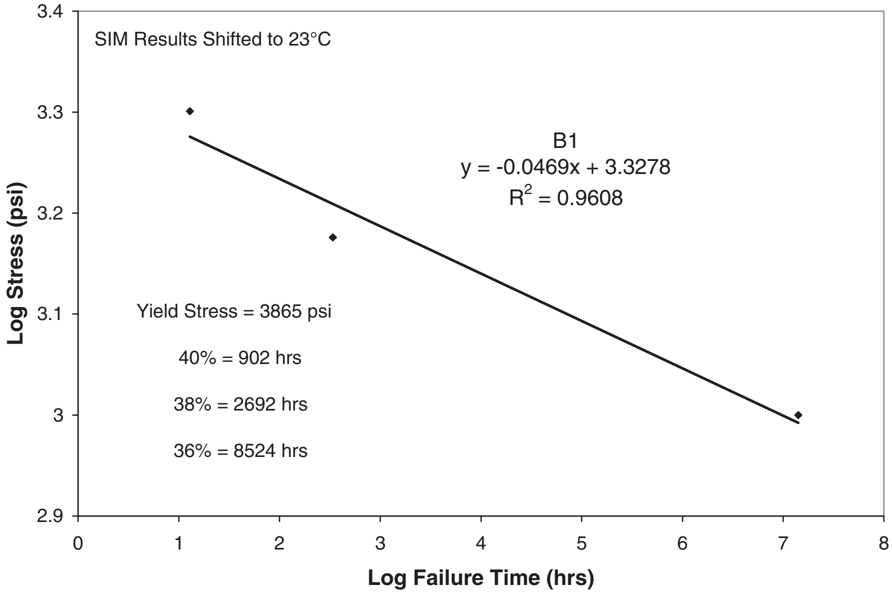

Figure 48. SIM creep rupture master curve for Sample B1.

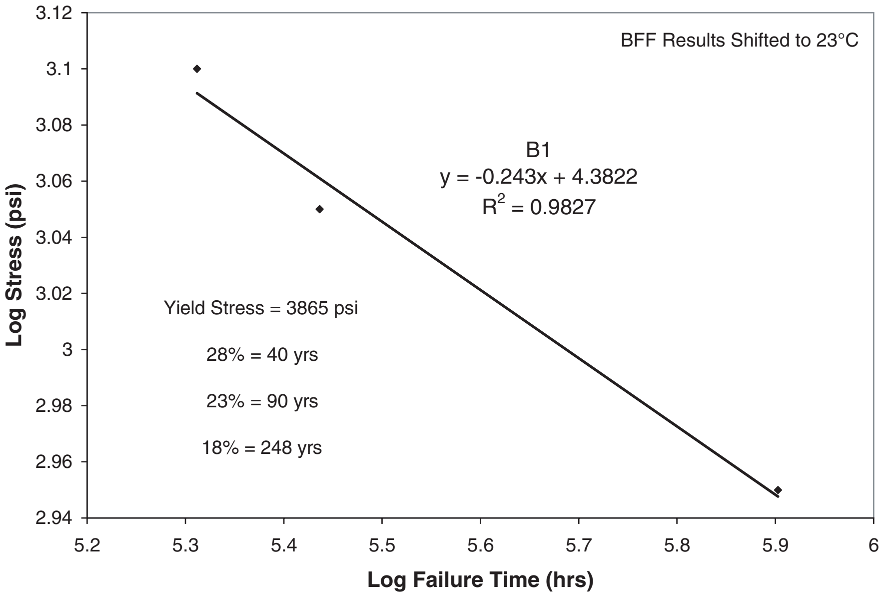

Figure 49. BFF stress-cracking master curve at 23°C for Sample B1.

2. Perform SIM on 12-in. diameter pipe samples to determine the relationship between a dumbbell specimen and an actual pipe.
3. Validate the SIM results by laboratory tests on pipe under compressive loading at room temperature.

The BFF test also needs to be validated in a similar manner to the SIM test. Multiple tests should be performed on the same samples across time to determine how reproducible the test is in a single laboratory. Then, tests should be performed by different laboratories to determine the lab-to-lab variability. Only then would the true value of this test be known.

The stress-cracking master curve for sample B1, generated by the BFF test, is shown in Figure 49.

Notice that the failure times for the brittle (Stage II) portion of the predicted curve are very long. Therefore, it is not practical to try to validate these results at room temperature. The same BFF test results shifted to 50°C (122°F) are shown in Figure 50.

In this case, applying loads of 295 lb, 242 lb, and 190 lb to a pipe sample at 50°C (122°F) should create stress-crack failures in about a year. This experiment could be done in a room controlled to 50°C. The results from this study will show if the master curves generated by the BFF test actually relate to

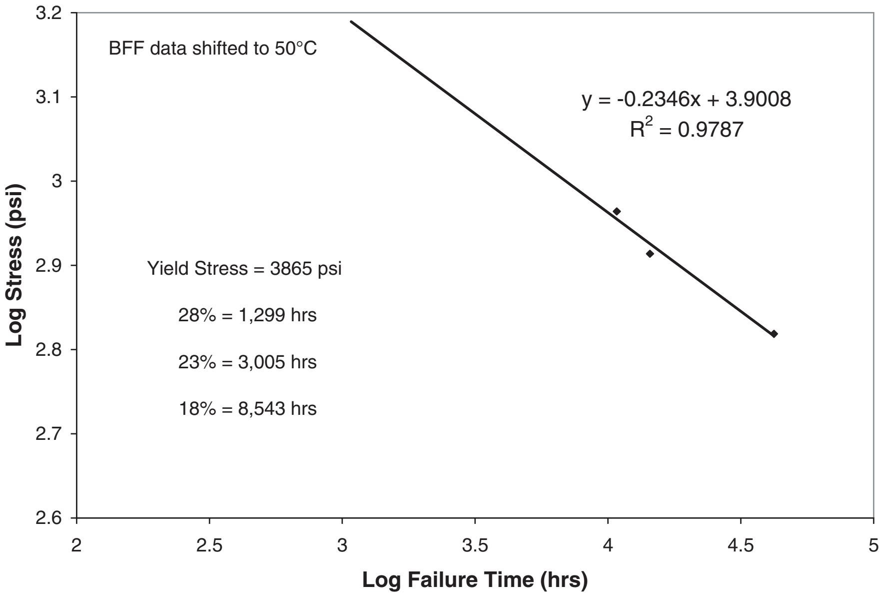

Figure 50. BFF stress-cracking master curve at 50°C for Sample B1.

a corrugated pipe under a direct compressive load. This test is especially important for pipe formulations containing post-consumer mixed-color recycled HDPE because they are the ones that are more likely to stress crack over their service lifetimes.

Validation of the BFF test results for predicting long-term stress-crack failures is very important because it is crystal clear that this is the main concern for corrugated pipe containing recycled HDPE. Of equal importance is validating the proposed BFF test under a single set of temperatures and stresses as a quality control test. Such a test is necessary to ensure that pipe formulations containing recycled HDPE have adequate stress-crack resistance over their intended service lifetime.

Finally, 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 unnotched 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.

Unfortunately, the times to grow stress cracks are quite long in a field study. The research suggests that the maximum stress that will cause a stress crack instead of local buckling is 1,000 to 1,200 psi, depending upon the yield stress. So, in a field environment at 23°C, it would take over 25 years to grow a crack in a sample with 30% recycled content. Acceleration through temperature seems to be the only way to verify the results of the lifetime estimates based on the BFF test.

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APPENDIX A

Procedures and Test Methods

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A.1 Procedures

A.1.1 Melt Blending by Extrusion

All blending and extruding was performed on a laboratory line consisting of a Welding Engineers Model HT8-222-2251 Twin Screw Extruder, a custom-built cooling bath equipped with a chiller and circulator and a Berlyn Corporation Model HV1 Pelletizer. The extruder has counter-rotating screws at an L:D ratio of 24:1. The extruder temperatures were set at 115°C (240°F) for the feed zone, and 177°C (350°F) for the mixing and metering zones. The die temperature was also 177°C (350°F). The die was a single 6.35-mm (0.25-in.) diameter rod and the die was fitted with two, 1-in. diameter melt filter screen holders.

All samples for extrusion were dry blended then gravity fed. The batch size was typically 2.2 lb (1 kg). The screw speed was set at 220 rpm, which produced an output rate of about 6.8 kg/hr (15 lb/hr). The residence time in the extruder was less than 45 seconds.

Samples of recycled regrind chips were melt blended twice and recycled reprocessed pellets were melt blended once.

All the experimental blends were melt blended three times to ensure optimum blending. Virgin resins were also blended in the same manner to duplicate the heat history of the blends.

It is worth mentioning that the processing conditions used during this study are different than what is actually going on in the recycled suppliers' plants or in the pipe manufacturers' plants. First of all, the twin screw mixes much better than a single screw, or even a high-length/diameter single screw. So, the blends studied herein were optimally blended. Secondly, the study on the effects of polypropylene (PP) might not translate well to the plants because the PP may not be as well blended as in the study. And thirdly, the twin screw processing is very gentle compared to a large single screw. So, even though there were no signs of oxidation in the study on the twin screw, oxidation may still be an important consideration in the plants.

A.1.2 Plaque Preparation (ASTM D4703)

Sample plaques were prepared according to ASTM D4703, *Practice for Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets* on a Pasadena Hydraulic Industries Model P215H Platen Press. Two sizes of plaques were prepared. The plaque used for the BAM test specimens was 15.24 cm × 15.24 cm × 1.27 mm (6 in. × 6 in. × 0.05 in.). The plaque used for all other tests was 17.78 cm × 17.78 cm × 1.9 mm (7 in. × 7 in. × 0.075 in.). The smaller mold used 50g of pellets while the larger one used 75g.

The open cavity mold was charged with pipe pieces about 5 cm × 5 cm in between two Mylar release sheets and placed in the press, which was preheated to 190°C. Once the mold reached 160°C, the set-point temperature was reduced to 177°C and the mold quickly pressed to 20.68 MPa (3000 psi). After the plastic was allowed to relax for at least 5 minutes, the mold temperature was recorded and the cooling process begun. The mold temperature was lowered by slowly opening a needle valve with cooling water. The temperature was recorded every 30 seconds and the cooling was done at a rate of 15°C ± 2°C. Once the temperature was below 50°C, the mold was removed.

A.2 Index Test Methods

A.2.1 Density (ASTM D1505)

The density of the samples was determined by ASTM D1505, *Standard Test Method for Density of Plastics by the Density-Gradient Technique.* The test is performed with the use of a glass column filled in a specified procedure with isopropanol and water that creates a gradient of densities from around 0.910 to 0.970 g/cm^3. Standard glass density beads are placed in the column for calibration. Samples are cut into small pieces (1⁄16-in. diameter), degassed under vacuum in isopropanol, and placed in the column. Once settled, the position is recorded from a graduated scale on the glass column and the density calculated from the position of the standard beads.

A.2.2 Melt Flow Index and Flow Rate (ASTM D1238)

The melt index was determined in accordance to ASTM D1238, *Standard Test Method for Flow Rates of Thermoplastics by Extrusion Plastometer.* A sample of plastic was heated inside a standard-sized cylinder for a controlled amount of time. Then, a weight was applied, forcing the molten plastic through a standard die. The mass of plastic that is extruded through the die in a specific period of time was reported. Two conditions were used for the polyethylene samples in this study. The first were a temperature of 190°C and a load of 2.16 kg (Melt Index) and the second were a temperature of 190°C and a load of 21.6 kg (Flow Rate). The results are reported as grams per 10 minutes.

The melt flow tests were performed on pellets after melt blending.

A.2.3 Percentage Volatiles (TRI Method)

Duplicate 2.000 g ± 0.100 g samples were placed in an aluminum weighing boat and heated at 175°C for 1 hour in a forced air oven. Upon cooling, the final weight was obtained and the percentage volatiles were calculated. This test was performed on the as-received pellets, before melt blending.

A.2.4 Percentage Black/Color + Ash (ASTM D4218)

This was done by the muffle furnace method described in ASTM D4218, *Test Method for Determination of Carbon Black*

Content in Polyethylene Compounds by the Muffle-Furnace Technique. About 1 g of sample was placed in an aluminum dish and heated in a small muffle furnace for 3 minutes. Upon cooling, the final weight was obtained and the percentage black/color + ash was calculated.

A.2.5 Percentage Ash (ASTM D5630)

This was generally done in accordance with Procedure B of ASTM D5630, *Ash Content of Plastics.* The sample mass was 1 g and the ashing temperature was 800°C. After heating for at least 10 minutes, the sample was cooled, reweighed, and the % Ash determined.

A.2.6 Percentage Polypropylene (TRI Method)

The percentage polypropylene was determined with a differential scanning calorimeter (DSC) and by a procedure developed for this project.

A combination DSC curve with both high-density polyethylene (HDPE) and PP melting profiles is shown in Figure A-1.

Notice that there is an overlap between the two peaks near 136°C. The area under each of these two curves is the heat of fusion, which relates to the percentage crystallinity of the two polymers. The tail of the HDPE curve will vary, depending upon the size and quality of the crystallites formed during cooling of the plastic from the melt. One would normally integrate the PP curve from 100°C to 175°C to determine the heat of fusion. However, in a sample containing both HDPE and PP, the end of the HDPE curve will cut off a portion of the PP curve. So, a method was devised to account for the missing portion of the PP curve.

The first step was to obtain a bag full of PP chips from a recycler who separates out PP caps. Then, 15 different colored chips were analyzed by DSC to determine the Heat of Fusion, the Melting Onset Temperature and the Melting Peak Temperature. The results are shown in Table A-1.

Next, a master curve was prepared by determining the heat of fusion of the PP starting at different initial temperatures. This is demonstrated in Figure A-2.

The four sets of onset temperatures and heats of fusion represent the area under the curve starting at temperatures of 130, 135, 140, and 145°C. This represents what would happen at different HDPE overlap temperatures in a blend of HDPE and PP.

This was done for all 15 cap samples and a master curve was generated by plotting the average heat of fusion as a function of overlap temperature. This is shown in Figure A-3. When a blend of PE and PP is analyzed, the temperature at the end of the HDPE peak is noted along with the area under the PP curve. This is shown in Figure A-4.

The inset graph is the portion of the curve from 135°C to 180°C. Notice the overlap temperature is 139.1°C and the heat of fusion for the PP curve is 3.18 J/g.

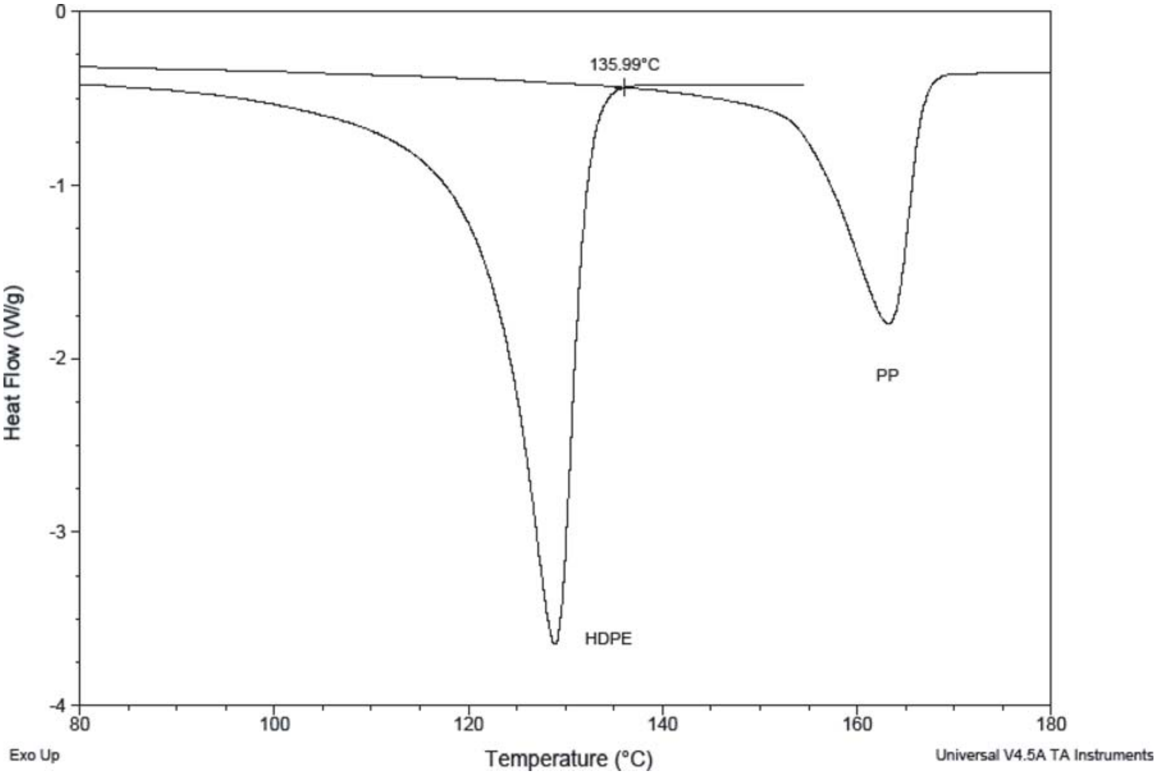


Figure A-1. DSC melting profiles for HDPE and PP.

Table A-1. Average melting properties for 15 different colored PP bottle closures.

Heat of Fusion (J/g)	Onset Temperature (°C)	Peak Temperature (°C)
100.0 ± 4.9	156.9 ± 3.4	167.2 ± 2.2

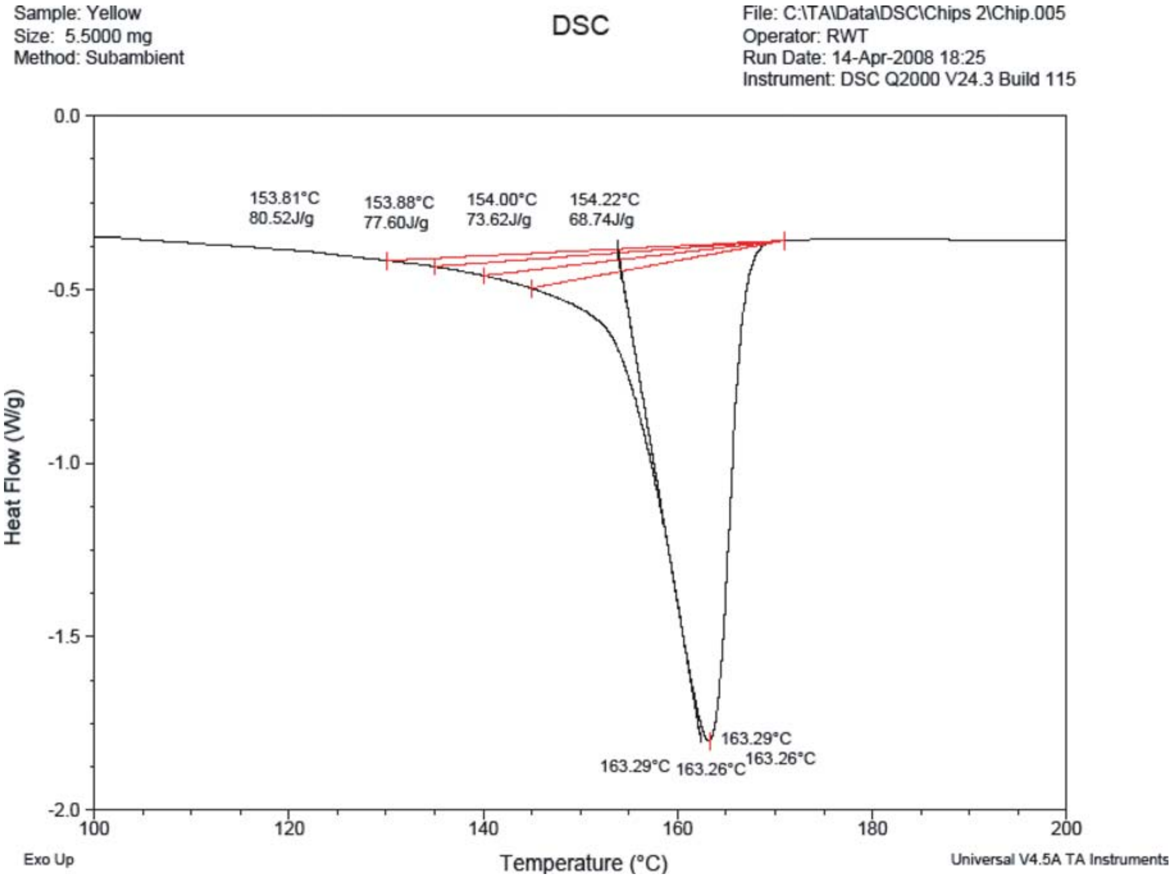


Figure A-2. DSC curve for PP showing integration from four starting temperatures.

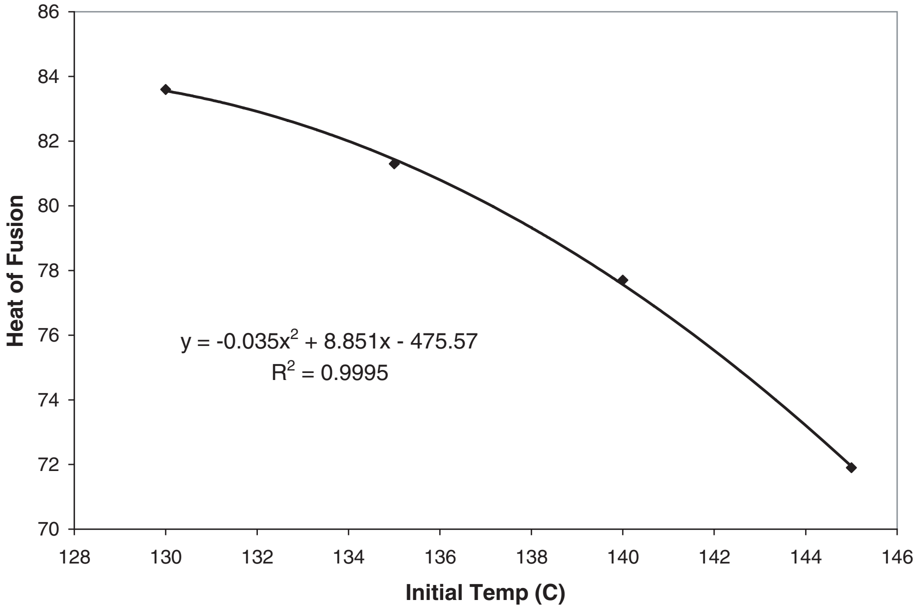


Figure A-3. PP heat of fusion as a function of overlap temperature (average of 15 PP caps).

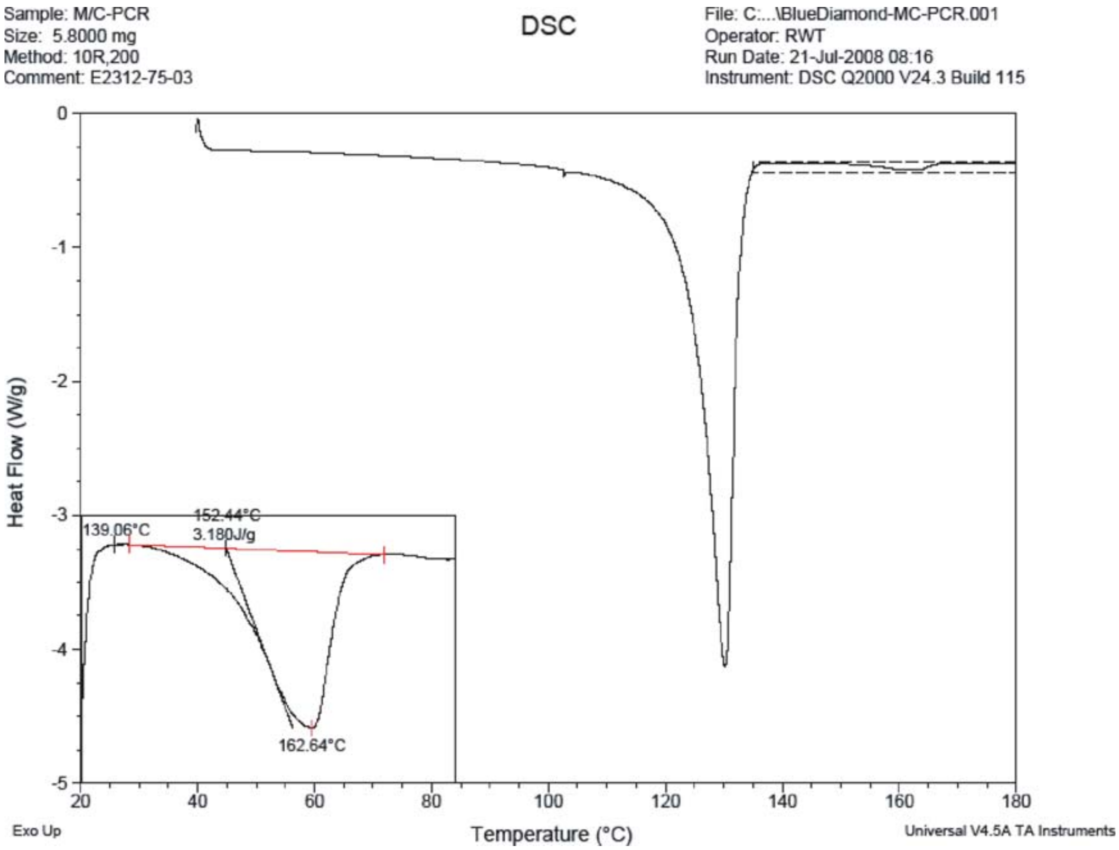


Figure A-4. DSC curve for a mixed-color recycled HDPE.

From the equation in Figure A-3, the heat of fusion for PP from 139.1°C to 175°C is 78.4 J/g. The percentage PP is then 3.18/78.4 × 100 or 4.0%.

A.2.7 Tensile Properties (ASTM D638)

The tensile properties were evaluated according to ASTM D638, *Standard Test Method for Tensile Properties of Plastics.* Type IV dumbbell specimens were used with a cross-head speed of 50.8 mm/min (2 in./min). The gage length for break strain was 50.8 mm (2.0 in.). The displacement was measured by cross-head travel. The tensile yield stress and the strain-at-break were determined. Five replicates were tested.

A.2.8 Flexural Modulus (ASTM D790)

The flexural modulus was determined in accordance to ASTM D790, *Test Method for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials.* Procedure B was used according to ASTM D3350. The specimen was a bar, 12.7-mm wide by 7.63-mm long (0.5 in. × 3 in.). The thickness varied within the sample, which was a compression molded plaque of a nominal thickness of 0.125 mil (3.175 mm).

The test span was 2.0 in. and the cross-head rate of deflection was 0.5 in. per minute. A Load vs. Deflection plot was obtained for each of the five specimens. Then, the displacement corresponding to a 2% strain in the outside surface was calculated by:

$$D = rL^2/6d$$

where:

D = displacement at 2% strain (in),
r = strain (in/in),
L = support span (in.), and
d = specimen thickness.

The required displacement varies with the thickness of the specimen. Therefore, each of the five test replicates will likely have a different displacement representing 2% strain. Once the displacement is determined, the corresponding load is found and the flexural stress at 2% strain is calculated by:

$$\sigma = 3PL/2bd^2$$

where:

σ = stress at 2% strain (psi),
P = load at 2% strain (lbf),
L = support span (in.),
b = specimen width (in.), and
d = specimen thickness.

Once the stress is found, the modulus is determined by dividing by the strain (0.02).

A.2.9 Oxidative Induction Temperature (OITemp) (ASTM D3350)

The procedure for this test is found in ASTM D3350, *Specification for Polyethylene Plastics Pipe and Fittings Materials.* An unweighed chip of material was placed in an open DSC sample pan and heated at 20°C/min in air until an exothermic oxidation occurred. The onset temperature of the oxidation peak is reported.

A.2.10 Oxidative Induction Time (OIT) (ASTM D3895)

This test was done in accordance to ASTM D3895, *Test Method for Oxidative-Induction Time of Polyolefins by Differential Scanning Calorimetry*. A sample weighing from 5–10 mg was placed in a DSC under nitrogen and heated to 200.00°C. Once the temperature was steady, the atmosphere was changed from nitrogen to oxygen. The test continued until an exothermic peak was obtained. The time from the introduction of oxygen to the onset of the exothermic peak is the oxidative induction time (OIT).

A.2.11 Pipe Deflection Tests (ASTM D2412)

The stiffness and flattening characteristics were determined by ASTM D2412 as modified by AASHTO M294. The testing machine used was a Lo-Tes Model LT-10, manufactured by Plowman Brothers (now Varicore). Duplicate pipe samples, about 12-in. long were deflected at a rate of 0.5 in./min, with a 5 lb preload and the load/deflection curve obtained. One pipe specimen was tested along the mold lines and the other was tested 90° offset from the mold lines. The test was continued up to a 40% linear deflection of the inside diameter occurred. The pipe stiffness was calculated by:

$$\text{PS}(\text{psi}) = \text{Load}(\text{lb})/\text{Deflection}(\text{in.})/\text{Length}(\text{in.})$$

The peak load and peak deflection were also recorded, which can be considered the buckling point of the pipe.

A.3 Stress-Crack Tests

A.3.1 Notched Stress-Crack Tests (NCLS and NCTL)(ASTM F2136, D5397)

There are two notched stress-crack tests that have been used during this project. The NCLS test is ASTM F2136, *Standard test Method for Notched, Constant Ligament-Stress (NCLS) Test to Determine Slow-Crack-Growth Resistance of HDPE Resins or HDPE Corrugated Pipe.* The NCTL test is ASTM D5397, *Standard Test Method for Evaluation of Stress Crack Resistance of Polyolefin Geomembranes Using Notched Constant Tensile Load Test.* The appendix of D5397 describes a single load test that is very similar to F2136.

The main difference between the two test methods is the applied load. For D5397, the applied loads are based on percentages of the measured yield stress of the material to be tested. For F2136, the applied load is a constant 600 psi (4.14 MPa). This is the applied load that would be used in D5397 if the measured yield stress was 4,000 psi (27.59 MPa) and the load was 15% of the yield stress. The constant applied stress of 600 psi has been accepted for pipe resins in the density range of 0.948–0.955 g/cm^3.

For most of this study, the NCTL test was performed at an applied stress equal to 15% of the materials yield stress. This was done for all materials submitted for characterization. The reasoning was that samples of recycled resins were obtained with yield stresses from 3,300 to 4,500 psi (22.76–31.03 MPa). Applying a constant stress of 600 psi would load these materials from 13 to 18% of their yield stress. Since stress crack times are highly dependent on applied stress, it was believed that the NCTL test at an applied stress of 15% of yield would produce more accurate results. Eventually, however, resin blends for consideration for AASHTO will require NCLS test results.

Besides how the load is determined, the tests are run basically the same way. Small dumbbell shaped specimens are cut and a face notch equal to about 20% of the specimens thickness is cut in a controlled manner with a razor blade and a notching device. Five specimens are then hung on a lever-load frame and placed in the 10% Igepal CO-630 surfactant solution. The loads are applied and the timers reset to zero hours.

The variables that need to be controlled in this test include the plaque preparation, the notches, the loads and the exposure environment. Of these, the environment seems to be the most dynamic. It was determined that for these laboratories, the Igepal baths required changing about every 4–5 weeks.

A.3.2 BAM Stress-Crack Test (TRI Method)

This test was introduced by researchers in Germany at the Bundesanstalt für Materialforschung und-prüfung (BAM). It was developed for evaluating textured coatings for Geomembranes (landfill liners). It was used successfully by the researcher on both textured Geomembranes and heat-bonded seams (*1, 2*).

The test involves the use of the same load frame as the NCTL or NCLS tests, except that special clamps are needed to test the 15.24-cm × 1.27-cm (6-in. × 0.5-in.) test specimens. The test is normally run under a constant load of 580 psi (4MPa), at 80°C in 5% Igepal CA-720. However, different temperatures were explored to determine appropriate conditions for evaluating recycled materials. A different surfactant is necessary because the cloud point of CO-630 is 55°C, so it is immisible at 80°C. The cloud point of CA-720 is 82°C. Because there is no notch in the test specimens, cracks will only grow if there are critical defects in the specimen. This makes the test useful to evaluate the effects of contamination, especially for recycled plastic.

Test results on geomembrane seams showed clearly that the test was sensitive to both the inherent stress-crack resistance of the sheet and the defects where cracks could initiate (*2*). The results also showed that very good stress-crack resistance resins were much more forgiving, in terms of critical defect size. Therefore, by choosing a better resin, one can lessen the effects of defects that are present.

BAM Test Procedure

1. Specimen preparation:

   Five samples, 0.6 in. × 6 in. (1.52 cm × 15.24 cm) were cut from the plaque with a die. The edges of these samples were shaved with a sledge microtome to remove any deformations caused by the cutting die and to reduce the sample width to about 0.5 in. (1.27 cm). The shaving operation is shown in Figure A-5. The result is five test specimens, 0.5-in. wide by 6-in. long with perfectly smooth edges.
2. Test Apparatus:

   The test was run with the use of a BT Technology stress crack fixture that had been modified with special grips and weights. The top grips were simply 1-in.^{2} (2.54-cm^2) smooth faced, stainless steel grips with a screw in each corner for tightening. These can be easily made from 1-in. bar stock, ¼-in. thick. The top of the back plate was tapped to accept 1⁄16-in. all thread. The back plate of the bottom grip was 1.5-in. (3.81-cm) long and had a 3⁄16-in. (4.76-mm) hole at the bottom so it could be mounted to the frame. The grips are shown in Figure A-6. The ones pictured show serrations on the outside. The insides of the grips are smooth.

   The test requires more weight than can be added to a typical NCTL weight tube. The tubes used had ⅛-in. all-thread running through the length of the tube. The tube was reduced in length by 4 in., and in its place were added two or three, 1-in. × 1-in. × 2-in. brass blocks. A combination of brass blocks and zinc coated steel shot was used to obtain the required loads. This is shown in Figure A-7.

   Exposure bath: A Blue M Model 1140A was used to perform the tests. A solution of 5% Igepal CA-720 in deionized (D.I.) Water was the test solution. The solution was found to have a limited life of around 300 h, so it was made fresh at the beginning of each test. The bath had an external stirrer along with a magnetic "flapper" to help control bath temperature.
3. Procedure: The test specimens with the shaved edges were carefully mounted in the grips. The pair of screws closest to the ends of the specimens were tightened first, followed by the pair of screws closest to the center. The amount of pressure on the screws is critical. If the grips are too loose, the specimen slips out. If the grip is too tight, a stress crack is initiated at the edge of the grip.

   Once the specimens are mounted, the five station load frame is placed in the bath, the pre-weighed weight tubes hung immediately, and the timers reset to zero. This way, as the test specimens heated up, the load was applied gradually.

   After the specimens failed or 300 h was reached, the test was terminated. The failed specimens were examined to be sure they did not break at a grip, and the crack faces

Figure A-5. Edge shaving of BAM test specimens.

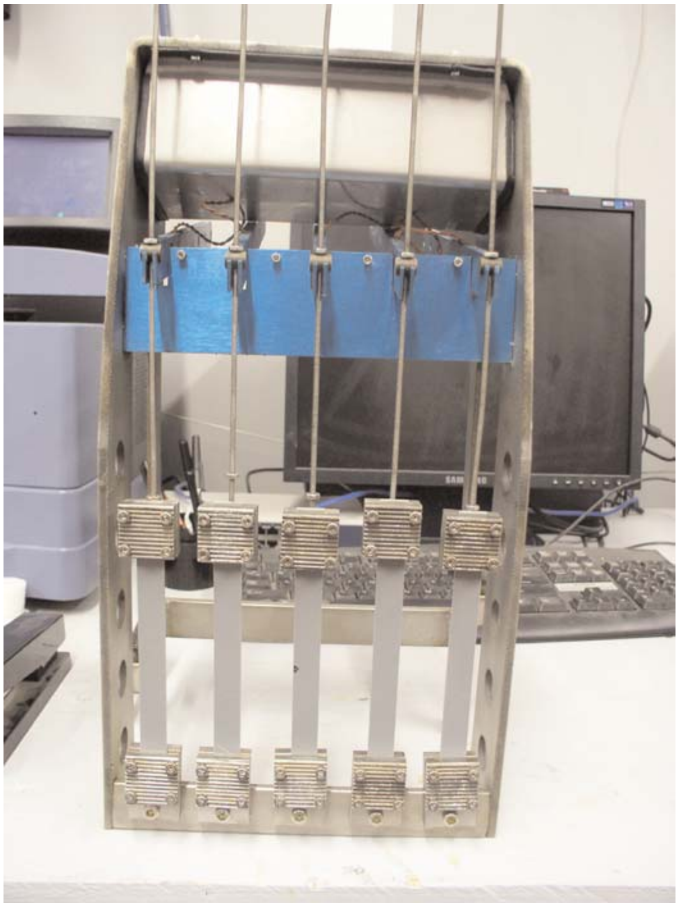


Figure A-6. Grips for the BAM test.

Figure A-7. Weights for the BAM test.

were examined microscopically to determine if the source of the crack could be identified. In almost every case involving post-consumer recycled (PCR) bottles (natural or mixed colored), the crack started at a piece of silicone rubber.

A.3.3 BFF Test (TRI Method)

This test method was first used in these laboratories as a means for classifying the cracking resistance of post-consumer recycled HDPE for a commercial client. The BAM test (*1*) was much too severe, so a test was needed that could distinguish poor stress cracking materials. The first tests were performed with the testing apparatus used for the BAM test, under the same 580 psi of load, but the exposure environment was changed from a surfactant to D.I. water. This environment was inspired by the Florida Department of Transportation (FDOT) durability protocol, also performed in D. I. Water (*2*). The test worked very well at 580 psi, but it was changed to 650 psi to match the FDOT testing conditions. And eventually, the test specimen was changed from the bar used in the BAM test to an ASTM D638 Type I Dumbbell. This was done to open up the possibilities for the test to be performed on unmodified stress-crack testing devices.

The current version of this test takes some aspects of the **B**am Test, with the exposure conditions of the **F**L-DOT test and uses a test specimen with a **F**athead, so the test is referred to as the BFF Test.

Test Specimen

The specimen is basically an ASTM D638 Type I Dumbbell with a couple of modifications. First, a 7⁄32-in. hole is drilled into each head portion so that the specimens can be mounted with a screw in a conventional stress-crack testing device. However, because of the holes, the tabs on each side of the hole are narrower than the reduced section of the dumbbell. Therefore, the highest stressed areas are the tabs, so all the specimens would naturally fail at the tabs. To get around this, a 6–in. square, open-faced mold was modified to produce plaques that were about 45 mil in the center and over 90 mil on the edges. This way, the tabs are under less stress than the

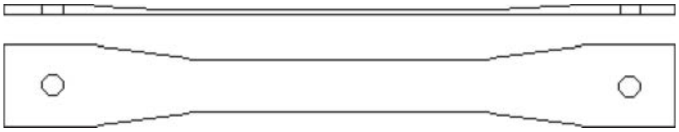


Figure A-8. Fathead specimen.

reduced section so nearly all of the failures occur in the reduced section. A drawing of the fathead test specimen is shown in Figure A-8.

Test Apparatus

The tests were performed with the use of a BT Technology NCTL stress crack frame. A picture of the test set-up is shown in Figures A-9 and A-10.

The test specimens are mounted in the frame, which is then placed in a constant temperature bath. Then, the weight tubes are attached to the back of the lever arm to apply the stress.

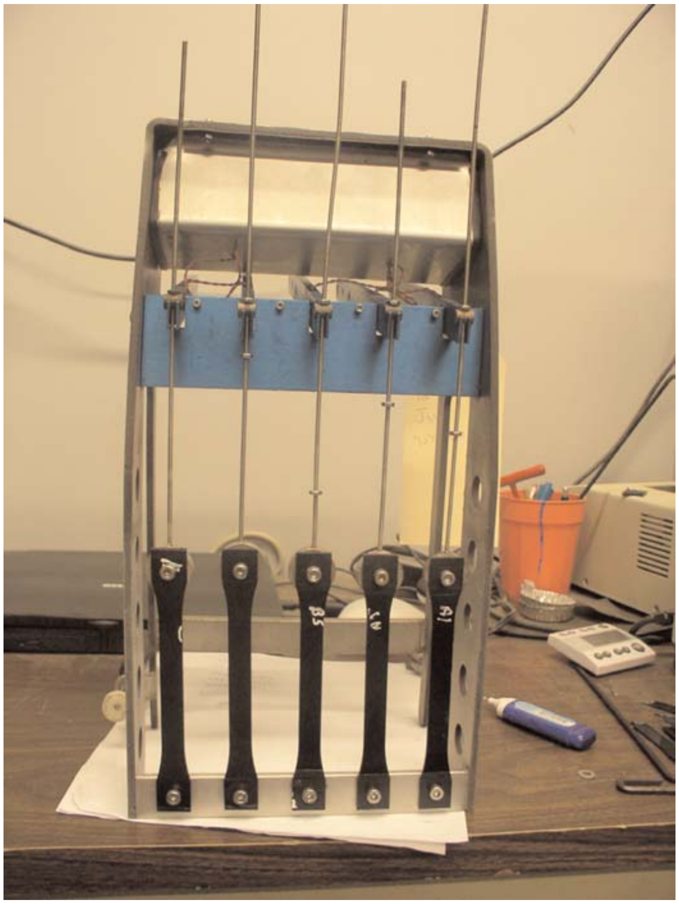


Figure A-9. BFF test set-up—front.

Figure A-10. BFF test set-up—back.

Exposure Conditions

Screening Test: Five specimens are placed in deionized water at 80°C and under an applied stress of 650 psi., and the times-to-failure recorded.

A.3.4 Junction Test (FDOT FM 5-572)

The junction test is a Florida DOT test method (FM 5-572) and involves a test specimen cut from the pipe itself. An ASTM Type IV dumbbell specimen is used and it is cut from the pipe in a way that leaves the junction between the corrugation and the liner intact in the center of the test specimen. A cross section of the specimen is shown in Figure A-11.

The specimen is loaded in tension and placed in D.I. Water under 650 psi at 80°C. The test was performed on six pipe

Figure A-11. Cross section of junction specimen.

samples and the results compared to the BFF test results under the same conditions.

A.4 Service-Lifetime Estimation Tests

A.4.1 Stepped Isothermal Method (SIM) For Long-Term Creep Modulus and Strain (ASTM D6992)

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 h) 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. 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. It has also been used by others on other PET fibers, Kevlar, and Polyethylene Naphthanate (PEN).

It has also been used by TRI to examine PP buried structures and most recently on HDPE resins used for corrugated drainage pipe. Only preliminary validation tests have been performed on PP, but the results are favorable.

The main difference between PET and HDPE is their respective temperature dependencies at temperatures below 80°C. 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. The sample-to-sample variability could be as large as the difference in creep rates at two different temperatures. A comparison for the two materials is shown in Figure A-12 below.

Time-temperature-superpositioning (TTS) has been used for decades and it is the basis for the validation procedures for polyethylene pipe materials in ASTM D2837 and Plastics Pipe Institute (PPI) Technical Report TR-3. TTS is 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 and 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.

SIM is a form of TTS in which 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)
Stress: 1000 psi.
Dwell Time: 10,000 seconds (2.78 h).

The raw, unshifted data are shown in Figure A-13.

There are nine temperature steps shown on the plot, so the highest temperature was 76°C. Notice that the sample yielded catastrophically during the early part of the 76°C step.

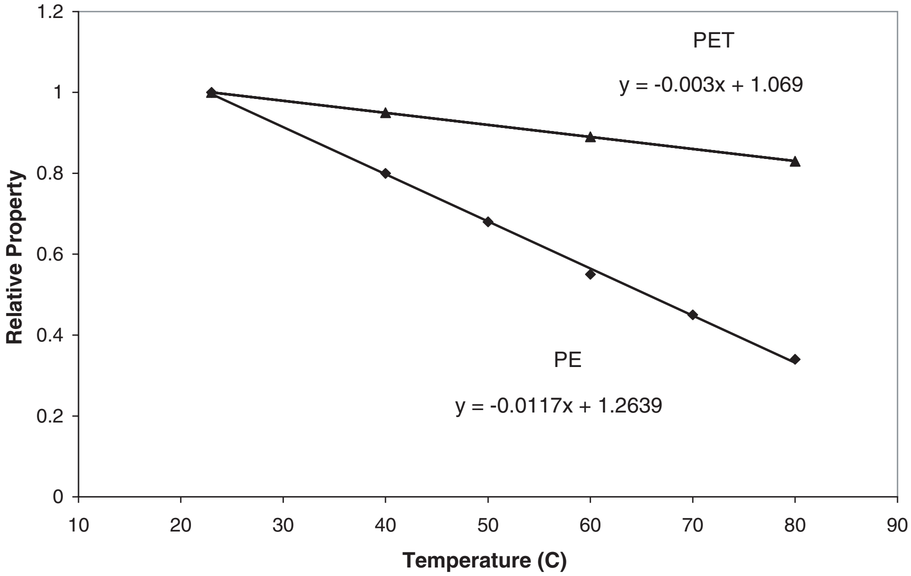


Figure A-12. Temperature dependence of PET and PE.

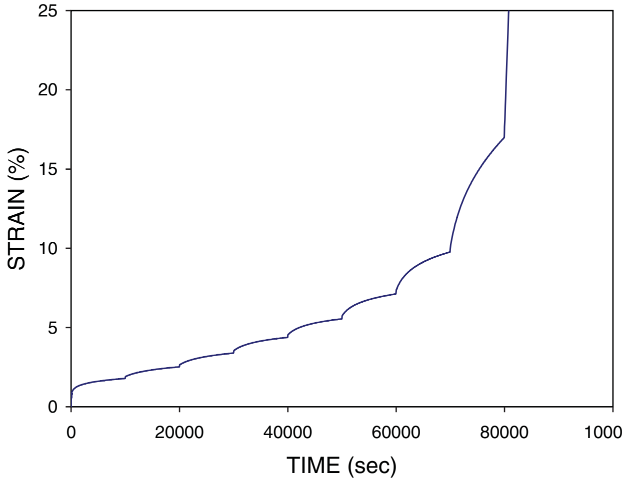


Figure A-13. Raw SIM data.

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 for each step above the first one, or t'. 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 rescale the individual creep curves and get them all on a common time scale.

The virtual starting time is found by plotting creep modulus vs. log time for the end of one step and the beginning of the next step. Then, one can adjust the virtual starting time until the slopes of the two steps align. A vertical shift is also added at this time to aid in the alignment process. This is illustrated in Figures A-14a to A-14d for the end of the 41°C step and the beginning of the 48°C step.

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 A-15 and A-16.

From these two curves, one can obtain both the 50-year creep modulus and 50-year creep strain. In this case, they are 10,000 psi and 8.9% respectively. These represent the behavior of the material when placed under a 1,000 psi load for 50 years.

A.4.2 Stepped Isothermal Method (SIM) for Creep Rupture (ASTM D6992)

Additionally, if one runs separate experiments at several loads, a master curve of stress vs. time can be obtained. Figure A-17 shows the results of SIM tests at 2,000, 1,500, and 1,000 psi.

Subsequent tests revealed that better results were obtained at applied stresses of 1,500, 1,250, and 1,000 psi. The failure times at 2,000 psi were thought to be too quick for good extrapolations.

The results from these test show that the 50-year strength of this material will be about 1,074 psi.

A.4.3 BFF Test for Long-Term Stress Crack Resistance (TRI Method)

Three sets of test conditions are required to perform a service lifetime prediction with the use of the BFF test. The conditions selected are the same ones used in the FDOT 100-year service-lifetime protocol. They are the following:

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

Service Lifetime Prediction

The service lifetime is found by application of the rate process method (RPM), described in ASTM D2837. The basic equation is:

$$\log t = A + B/T + C \log S/T \qquad (1)$$

where

t = failure time (hrs)
T = temperature (°K)
S = stress (psi), and
A, B and C are constants.

Under the three sets of test conditions, the values of the constants are found by solving three equations with three unknowns. The three equations are the following:

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:

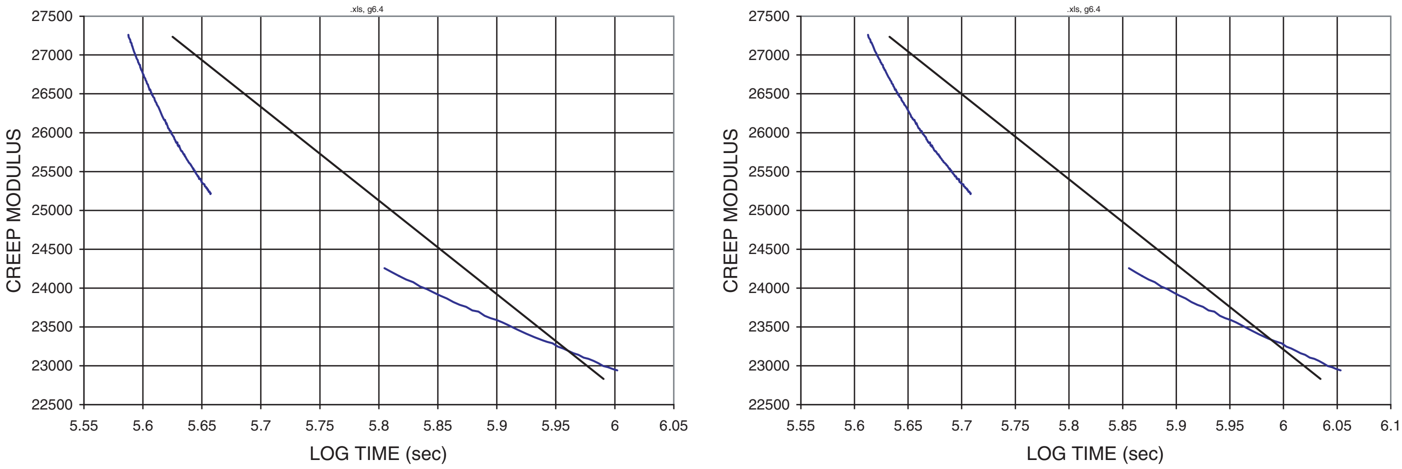
$$353 \log t_1 - 343 \log t_3 = 353A - 343A = 10A$$

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

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

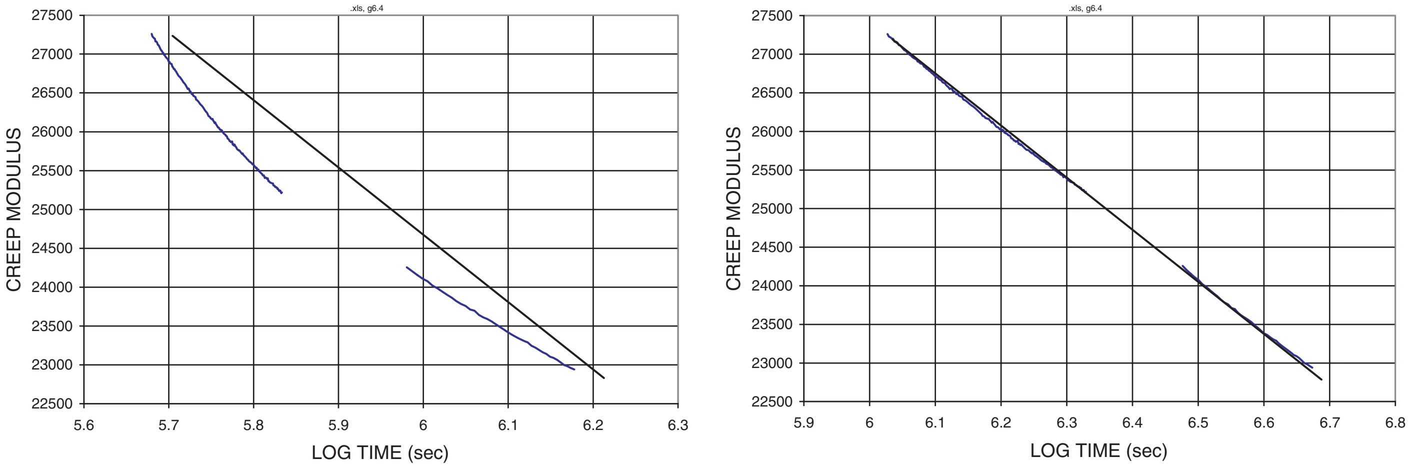
$$353 \log t_1 - 343 \log t_2 = C \log 650 - C \log 450$$

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



(a) t’ = 0 sec

(b) t’ = 10,000 sec



(c) t’ = 20,000 sec

(d) t’ = 28,100 sec

Figure A-14. Curve alignments at different virtual starting times.

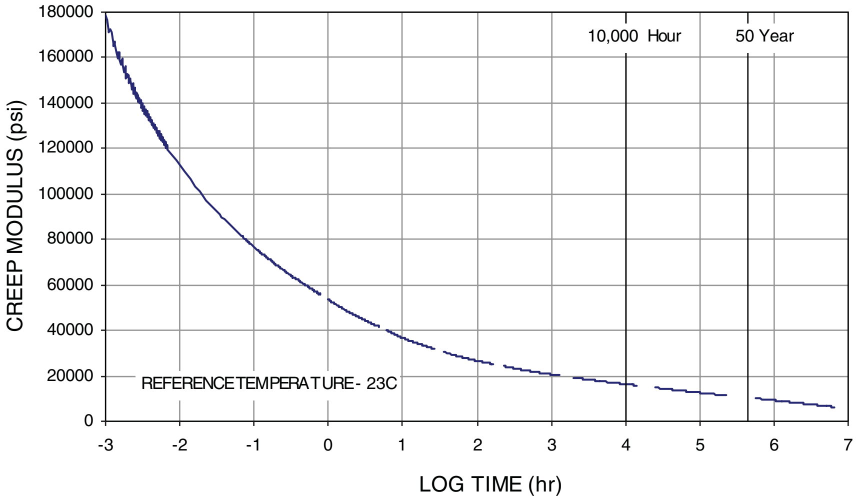


Figure A-15. Creep modulus master curve under 1,000 psi of stress.

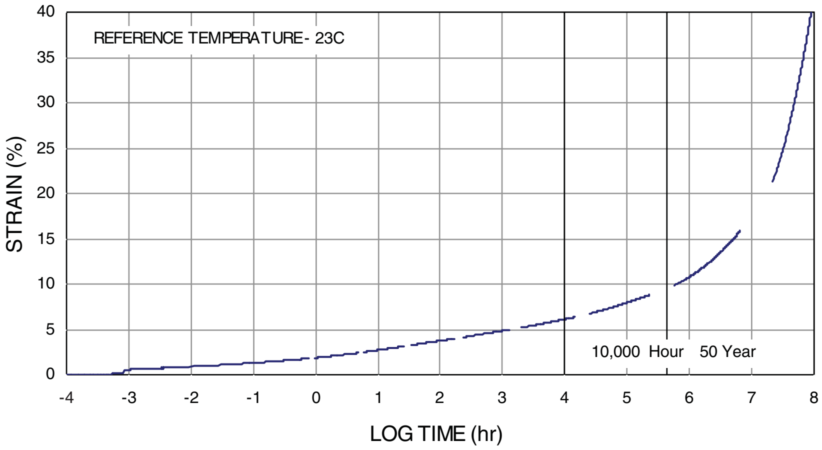


Figure A-16. Creep strain master curve under 1,000 psi of stress.

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

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

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

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

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

$80°C/650\ psi = 110 \pm 33\ hrs = t_1 \qquad \log\ t_1 = 2.041$
COV = 30%

$80°C/450\ psi = 430 \pm 172\ hrs = t_2 \qquad \log\ t_2 = 2.633$
COV = 35%

$70°C/650\ psi = 500 \pm 175\ hrs = t_3 \qquad \log\ t_3 = 2.699$
COV = 40%

Determine A

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

$$A = -20.50$$

Determine C

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

$$C = -1306.10$$

Determine B

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

$$B = 720.47 + 7247.09 + 3674.06$$

$$B = 11{,}628.95$$

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:

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

$$\log\ t = -20.50 + 11{,}628.95/296 - (1306.10\ \log 500)/296$$

$$\log\ t = -20.50 + 39.29 - 11.98$$

$$\log\ t = 688$$

$$t = 7{,}568{,}640\ h = 886\ \text{Years}$$

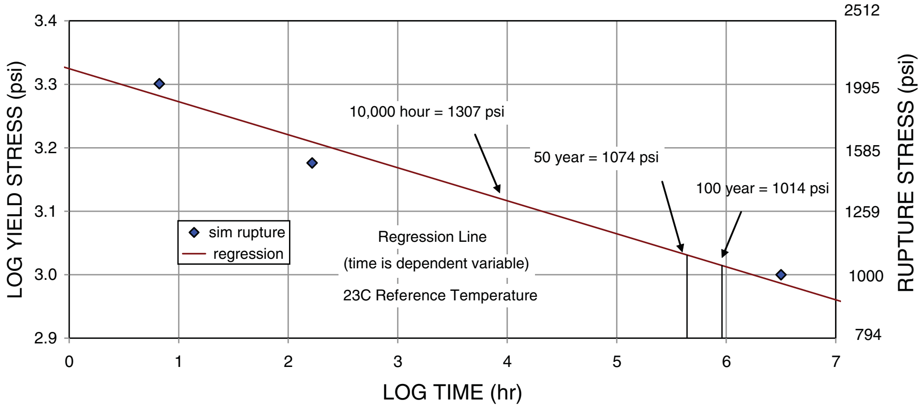


Figure A-17. Long-term yield stress by SIM.

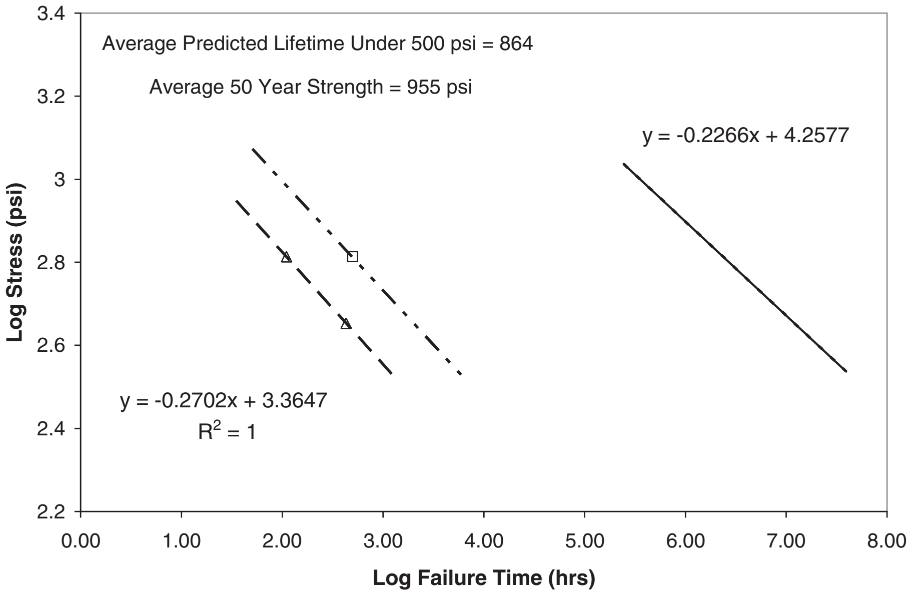


Figure A-18. BFF lifetime prediction.

This value represents the average lifetime based on the test results.

Long-Term Strength

Once the constants are known, one can also calculate the 50 year strength at 23°C from equation 1. 50 years = 438,000 h

$$\log\ 438{,}000 = A + B/296 + (C \log S)/296$$

$$5.641 = -20.50 + 11{,}628.95/296 + (-1306.10 \log S)/296$$

$$1669.74 = -6076.88 + 11{,}628.95 - 1306.10 \log S$$

$$2.98 = \log\ S, \quad S = 950\ \text{psi}$$

The rate process method predicted a service lifetime at 23°C and 500 psi of stress to be 176 years and also determined the 50-year strength would be 661 psi. This strength represents the stress under which stress cracks will not form for 50 years. These results can be presented graphically as in Figure A-18.

APPENDICES B, C, AND D

Appendices B, C, and D are not published herein but are available on the NCHRP Project 04-32 web page at http://apps.trb.org/cmsfeed/TRBNetProjectDisplay.asp?ProjectID=865.

APPENDIX B: Recycled Polyethylene Resins
APPENDIX C: Recycled-Resin Blends
APPENDIX D: Pipe Containing Recycled HDPE

APPENDIX E

Proposed Draft Standard Specification for PCR MCR High-Density PE Bottles for Use in AASHTO-Approved Corrugated Drainage Pipe

DRAFT

Draft Standard Specification for

Post-Consumer Recycled Mixed-Color Reprocessed High Density Polyethylene Bottles for Use in AASHTO Approved Corrugated Drainage Pipe

AASHTO Draft Standard XXX-XX

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1. SCOPE

1.1 This specification covers the quality of post-consumer recycled, mixed-color, reprocessed, high density polyethylene bottles for use in AASHTO approved corrugated drainage pipe.

1.2 This specification presents a set of properties to be met for the resin.

1.3 This specification can be used by recycled resin suppliers as part of manufacturing quality control (MQC), or by pipe manufacturers or independent bodies as manufacturing quality assurance (MQA).

2. REFERENCED DOCUMENTS

2.1 ASTM Standards:

- D638, Test Method for Tensile Properties of Plastics
- D792, Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement
- D1238, Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer
- D1505, Test Method for Density of Plastics by the Density-Gradient Technique
- D4218, Test Method for Determination of Carbon Black Content in Polyethylene Compounds By the Muffle-Furnace Technique
- D4703, Practice for Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets
- D5630, Test Method for Ash Content in Plastics

3. TERMINOLOGY

3.1 *Post-Consumer Recycled HDPE (PCR-HDPE)* – Polyethylene that has been discarded by consumers after use, then collected, cleaned, and washed. The density is greater than 0.940 g/cm^3 and typical sources include primarily bottles and some bags.

3.2 *PCR-HDPE Regrind* – Post-Consumer Recycled HDPE that has been cleaned, washed and ground into plastic chips.

3.3 *Reprocessed PCR-HDPE* – Post-Consumer Recycled HDPE that has been cleaned, washed, melt-filtered, and pelletized. Commonly known as "Repro."

3.4 *Mixed-Color PCR-HDPE* – Post-Consumer Recycled HDPE that is composed of mostly colored detergent bottles.

3.5 *Natural PCR-HDPE* – Post-Consumer Recycled HDPE that is composed of milk bottles.

4. PROPERTIES

4.1 The required properties for PCR mixed-color reprocessed resin are given in Table 1.

4.2 Specific details of the tests are found in Section 7.

Table 1. Required properties for PCR mixed-color reprocessed resin.

Property	Test Method	Sample	Specified Value	Frequency
Density	ASTM D1505 ASTM D792	Pellets/Plaque	0.945-0.960 g/cm	1 per shift
Melt Index	ASTM D1238 190°C/2.16 Kg	Pellets/Plaque	<0.60 g/10 min	1 per shift
% Volatiles	175°C/1 Hour	Pellets[a]	<0.2 %	Certify[b]
% Ash	ASTM D5603	Pellets/Plaque	<1.5 %	1 per shift
% PP	DSC	Plaque	<10.0 %	1 per lot[c]
Yield Stress (psi)	ASTM D638[d]	Plaque	>3500 psi	1 per lot
Break Strain (%)	ASTM D638[e]	Plaque	>100 %	1 per lot

[a] *Tested on pellet samples before melt blending.*

[b] *Manufacturer will provide a certificate stating their material will lose less than 0.2% of its weight when a 2.000 ± 0.100 g sample is heated at 350°F for 1 hour.*

[c] *A production lot is defined as either a truckload, or one compartment of a railcar. Its weight is generally between 40,000 and 50,000 lb.*

[d] *Strain rate shall be 2.0 in. per minute.*

[e] *Assume 2.0-in. gage length.*

5. SAMPLING

5.1 A production lot shall be considered as a truckload or shipping container full of 1,500 lb boxes of resin, or one compartment of a railcar. The weights normally fall between 40,000 and 50,000 lb.

5.2 A truckload shall be sampled by obtaining 0.5 lb of material from each of eight boxes, for a total of 4 lb of resin. Combine the eight samples to form a lot sample.

5.3 A compartment of a railcar shall be sampled by obtaining 2 lb from the top and 2 lb from the bottom of the compartment. Combine the two samples to form a lot sample.

5.4 The entire lot sample shall be dry blended before sample preparation. A convenient way to do this is by hand in a 5-gallon plastic pail.

5.5 Divide the lot sample into two; one for immediate testing and the other for validation testing, should the need arise. The back-up sample shall be kept for 90 days after the resin is sold.

5.6 Clearly label the sample and include a traceable production number and a date.

6. SAMPLE PREPARATION

6.1 Homogenize about 2 lb (900 g) of material by melt blending without filtration, preferably with a twin screw laboratory extruder. A two roll mill is also acceptable.

6.2 The blended material shall be compression molded according to ASTM D4703, with the use of a 15°C/min cooling rate.

6.3 The plaque shall be 1.9- ± 0.08-mm (0.075- ± 0.003-in.) thick.

6.4 The plaque shall be allowed to equilibrate at standard laboratory conditions (72°F, 50% RH) for at least 12 hours before tensile testing.

7. TEST METHODS

7.1 *Test Conditions* – Unless otherwise specified in the test methods or in this specification, conduct tests at the standard laboratory temperature of 23 ± 2°C (73.4 ± 3.6°F).

7.2 *Density* – Test methods ASTM D1505 or D792 are acceptable. Make three separate determinations using separate homogenized pellets or separate portions of a plaque. The plaque thickness shall be 1.9 ± 0.08 mm (0.075 ± .003 in.). Calculate and report the average and standard deviation from the mean.

7.3 *Melt Index* – Test method ASTM D1238, using Condition 190/2.16. Make duplicate determinations on either pellets or a plaque and calculate the average.

7.4 *% Volatiles* – This test will be performed in aluminum pans that have been heated at 350°F (177 °C) until they obtain a constant weight.

7.4.1 Test duplicate samples.

7.4.2 Weigh 2.000 ± 0.100 g of pellets or a plaque to an accuracy of three places past the decimal point into an aluminum pan.

7.4.3 Place the pan in a forced-air recirculating oven preheated and stabilized to 350 ± 5°F (177 ± 2°C) for a period of 1 hour.

7.4.4 Remove the pan and let it completely cool to room temperature in a desiccator.

7.4.5 Reweigh the pan and calculate the percentage weight loss of the sample. Report the average to the nearest 0.01%.

7.5 *% Ash* - Test method ASTM D5630, Procedure B.

7.5.1 Sample size shall be 1g.

7.5.2 Heat at least 10 minutes at 800°C.

7.5.3 Test in triplicate samples.

7.6 *% Polypropylene*

7.6.1 Generate a melting curve by differential scanning calorimetry (DSC) at a heating rate of 10°C/min. See Figure 1.

7.6.2 The curve shall be expanded to a range that includes the end of the HDPE peak and the complete PP peak. See Figure 2.

7.6.3 Integrate the curve from a flat point before the PP melting to a point where the PP curve returns to baseline.

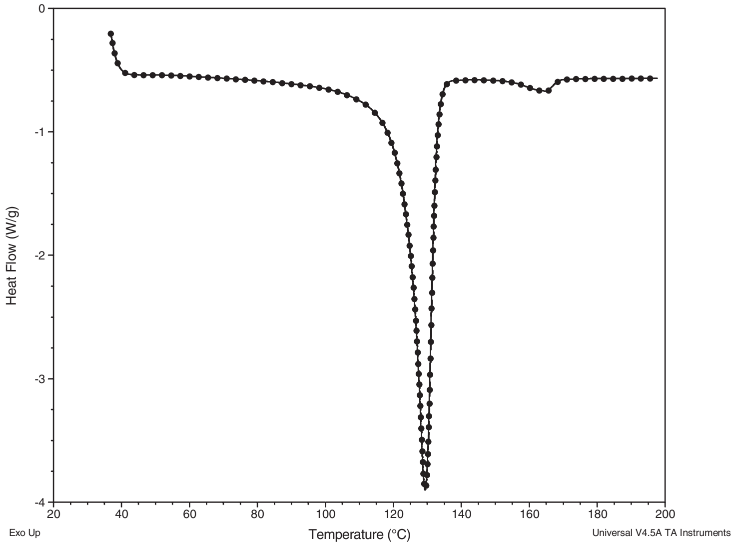

Figure 1. DSC curve of HDPE containing PP generated.

7.6.4 Mark the point where the HDPE melt returns to baseline. The end of the HDPE melt and the beginning of the PP melt will not necessarily be the same point.

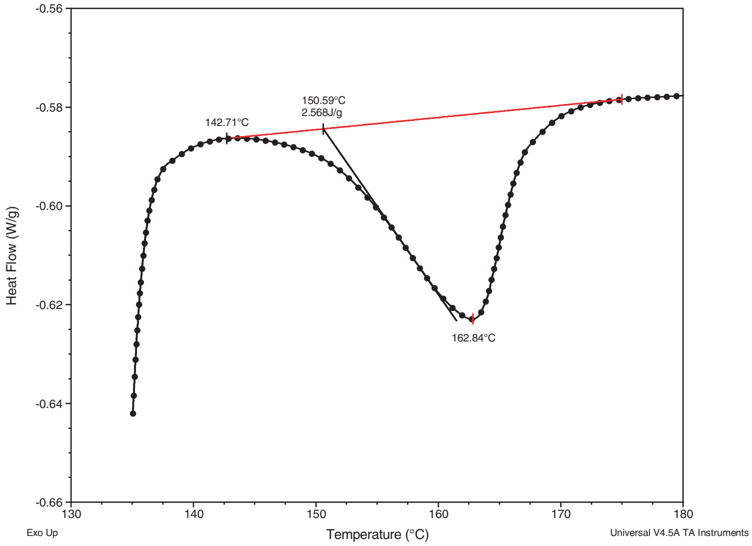

Figure 2. DSC curve of HDPE containing PP expanded.

7.6.5 Record the HDPE end-of-melt temperature and the PP heat of fusion in J/g.

7.6.6 Calculate the theoretical heat of fusion for 100% polypropylene for the portion of the PP melting curve that does not overlap with HDPE by:

$$Y = -0.035X^2 + 8.851X - 475.6 \quad (1)$$

Where: X = HDPE end-of-melt temperature (°C)
And $Y = \Delta H_f$ of 100% PP not overlapped with HDPE.

For $X = 142.7$, $Y = 74.7$ J/g.

7.6.7 Calculate the percentage PP by:

$$\%PP = \Delta H_f \text{ sample} / \Delta H_f \text{ 100\% PP} \times 100 \quad (2)$$

For ΔH_f sample = 2.57 J/g, %PP = 3.4 %

7.6.8 Test two specimens from different parts of the molded plaque and report the average.

7.7 *Yield Stress/Break Strain* – Test ASTM Method D638, with Type IV dumbbell, 2-in./min crosshead speed.

7.7.1 Test five specimens, individually measured for thickness.

7.7.2 Assume 1.3-in. gage for yield stress and 2.0-in. gage length for break strain. Use crosshead to follow displacement.

7.7.3 Report 5 results, average, and standard deviation.

8. RETEST AND REJECTION

8.1 If any failure occurs, the material shall be retested to establish conformity in accordance to the specified property. A second failed result will result in rejection of the lot for the purposes of this specification.

9. CERTIFICATION

9.1 Upon request of the purchaser in the contract or order, a manufacturer's certification that the material was manufactured and tested in accordance with this specification, together with a report of the test results, shall be furnished at the time of shipment.

APPENDIX F

Proposed Draft Standard for Recycled Content Containing HDPE Resin Formulations for Corrugated Pipe Made to AASHTO Standard M252-Recycled

DRAFT

Draft Standard Specification for

RECYCLED CONTAINING HDPE FOR CORRUGATED PIPE MADE TO AASHTO STANDARD M252-RECYCLED

AASHTO Draft Standard XXX-XX

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1. SCOPE

1.1 This specification covers the quality of HDPE resin formulations containing recycled HDPE that are intended for use in AASHTO M252–Recycled approved corrugated drainage pipe for subsurface drainage applications, sizes 75- to 250-mm diameter (3-10 in.).

1.2 The recycled polyethylene may be either post-commercial or post-industrial.

1.3 This specification presents a set of properties and test frequencies to be met for the resin.

1.4 This specification can be used by suppliers of resin containing recycled PE as part of manufacturing quality control (MQC), or by pipe manufacturers or independent bodies as manufacturing quality assurance (MQA).

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2. REFERENCED DOCUMENTS

2.1 ASTM Standards:

- D638, Test Method for Tensile Properties of Plastics
- D792, Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement
- D1238, Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer
- D1505, Test Method for Density of Plastics by the Density-Gradient Technique
- D3895, Standard Test Method for Oxidative-Induction Time of Polyolefins by Differential Scanning Calorimetry
- D4218, Test Method for Determination of Carbon Black Content in Polyethylene Compounds By the Muffle-Furnace Technique
- D4703, Practice for Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets
- D5630, Test Method for Ash Content in Plastics
- F2136, Standard Test Method for Notched, Constant Ligament-Stress (NCLS) Test to Determine Slow-Crack-Growth Resistance of HDPE Resins or HDPE Corrugated Pipe

3. TERMINOLOGY

3.1 *Mixed-Color PCR-HDPE* – Post-Consumer Recycled HDPE that is composed of mostly colored detergent bottles.

3.2 *Natural PCR-HDPE* – Post-Consumer Recycled HDPE that is composed of milk bottles.

3.3 *Post-Consumer Recycled HDPE (PCR-HDPE)* – Polyethylene that has been discarded by consumers after use, then collected, cleaned, and washed. The density is greater than 0.940 g/cm^3 and typical sources include primarily bottles and some bags.

3.4 *PCR-HDPE Regrind* – Post-Consumer Recycled HDPE that has been cleaned, washed and ground into plastic chips.

3.5 *Post-Industrial Recycled HDPE (PIR-HDPE)* - Polyethylene that has been obtained for recycling from industrial sources. This may include industrial scrap, rejected parts, or surplus goods.

3.6 *Reprocessed PCR-HDPE* - Post-Consumer Recycled HDPE that has been cleaned, washed, melt-filtered, and pelletized. Commonly known as “Repro.”

4. REQUIRED PROPERTIES

4.1 The required properties for corrugated pipe resins containing recycled HDPE are given in Table 1. Specific details of the tests are found in Section 7.

Table 1. Required properties for M252 resins containing recycled HDPE.

Property	Test Method	Sample	Specified Value	Frequency
Density	ASTM D1505 ASTM D792	Pellets/Plaque	≥0.948[a] g/cm	1 per shift
Melt Index	ASTM D1238 190°C/2.16 Kg	Pellets/Plaque	<1.0 g/10 min	1 per shift
% Volatiles	175°C/1 Hour	Pellets[b]	<0.2 %	Certify[c]
% Carbon Black	ASTM D4218	Pellets/Plaque	2-4 %	1 per shift
% Ash	ASTM D5603	Pellets/Plaque	<0.75 %	1 per shift

% PP	DSC Section 7.6	Plaque	<7.5 %	1 per lot[d]
Yield Stress	ASTM D638[e]	Plaque	>3500 psi	1 per lot
Break Strain	ASTM D638[f]	Plaque	>150 %	1 per lot
NCLS Stress Crack Test	ASTM F2136	Plaque	>20 hrs	1 per lot
OIT	ASTM D3895	Pellets/Plaque	>25 min[g]	1 per lot

[a] Correct for carbon black by $D_{cor} = D - 0.0044C$, where C is % carbon black.

[b] Tested on pellet samples before melt blending.

[c] Manufacturer will provide a certificate stating their material will lose less than 0.2% of its weight when a 2.000 ± 0.100 g sample is heated at 350ºF for 1 hour.

[d] A production lot is defined as either a truckload, or one compartment of a railcar. Its weight is generally between 40,000 and 50,000 lbs.

[e] Strain rate shall be 2.0 in. per minute, assume 1.3 in. gage.

[f] Assume 2.0" gage length.

[g] A common additive package that meets this requirement is 1000 ppm Irganox 1010 plus 500 ppm Irgaphos 168, or an equivalent.

5. SAMPLING

5.1 A production lot shall be considered as a truckload or shipping container full of 1,500 lb boxes of resin, or one compartment of a railcar. The weight is normally between 40,000 and 50,000 lbs.

5.2 Sample a truckload by obtaining 0.5 lb of material from each of eight boxes, for a total of 4 lbs of resin. This is about every fourth box. Combine the eight samples to form a lot sample.

5.3 Sample a compartment of a railcar by obtaining 2 lb from the top and 2 lb from the bottom of the compartment. Combine the two samples to form a lot sample. Therefore, each railcar will have four lot samples for evaluation.

5.4 The entire lot sample shall be dry blended before sample preparation. A convenient way to do this is by hand in a 5-gallon plastic pail.

5.5 The lot sample shall be divided into two; one for immediate testing and the other for validation testing, should the need arise. The back-up sample shall be kept for 90 days after the resin is sold.

5.6 Clearly label the sample and include a traceable production number and a date.

6. SAMPLE PREPARATION

6.1 Homogenize about 2 lb (900 g) of material by melt blending, preferably with a twin screw laboratory extruder. A two roll mill is also acceptable.

6.2 The blended material shall be compression molded into plaques according to ASTM D4703, with the use of a 15°C/min. cooling rate.

6.3 The plaques shall be 1.9- ± 0.08-mm (0.075- ± 0.003-in.) thick for tensile and NCLS testing.

6.4 Allow the plaques to equilibrate at standard laboratory conditions (72°F, 50% RH) for at least 12 hours before they are tested.

7. TEST METHODS

7.1 *Test Conditions* – Unless otherwise specified in the test methods or in this specification, conduct tests at the standard laboratory temperature of 23 ± 2°C (73.4 ± 3.6°F).

7.2 *Density* – Test methods ASTM D1505 or D792 are acceptable. Make three separate determinations using separate pellets or separate portions of a plaque. The plaque thickness shall be 1.9 ± 0.08 mm (0.075 ± .003 in.). Calculate and report the average and standard deviation from the mean. Correct the density value for % carbon black by subtracting 0.0044 g/cm^3 for each percent of carbon black.

7.3 *Melt Index* – Test method ASTM D1238, using Condition 190/2.16. Make duplicate determinations on either pellets or a plaque and calculate the average.

7.4 *% Volatiles* – This test shall be performed in aluminum pans that have been heated at 350°F (177°C) until they obtain a constant weight.

7.4.1 Test duplicate samples.

7.4.2 Weigh 2.000 ± 0.100 g of pellets or a plaque to an accuracy of three places past the decimal point into an aluminum pan.

7.4.3 Place the pan in a forced-air recirculating oven preheated and stabilized to 350 ± 5°F (177 ± 2°C) for a period of 1 hour.

7.4.4 Remove the pan and let it completely cool to room temperature in a desiccator.

7.4.5 Reweigh the pan and calculate the percentage weight loss of the sample. Report the average to the nearest 0.01%.

7.5 *% Ash* - Test method ASTM D5630, Procedure B.

7.5.1 Sample size shall be 2 g.

7.5.2 Heat for 5 minutes at 800°C.

7.5.3 Test triplicate samples.

7.6 *% Polypropylene*

7.6.1 A melting curve shall be generated by differential scanning calorimetry (DSC) at a heating rate of 10°C/min from room temperature to 200°C. See Figure 1.

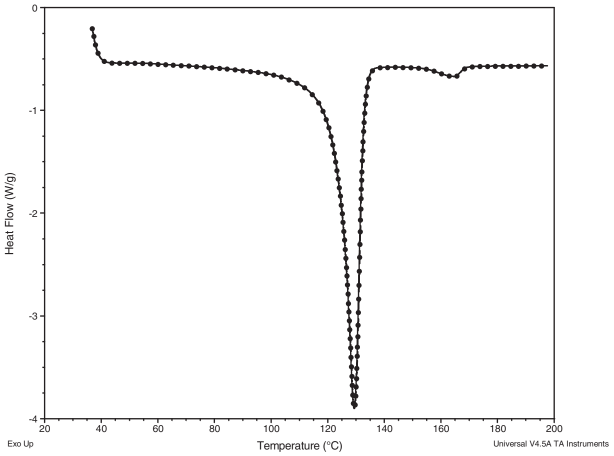

Figure 1. DSC curve of HDPE containing PP generated.

7.6.2 Expand the curve to a range that includes the end of the HDPE peak and the complete PP peak. See Figure 2.

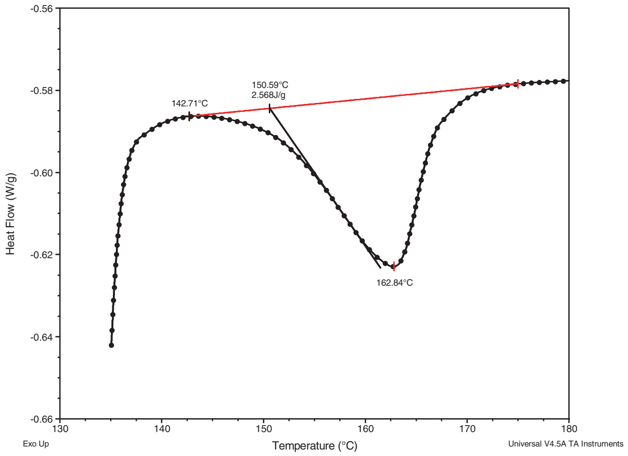

Figure 2. DSC curve of HDPE containing PP expanded.

7.6.3 Integrate the curve from a flat point before the PP melting to a point where the PP curve returns to baseline.

7.6.4 The point where the HDPE melt returns to baseline will be marked. The end of the HDPE melt and the beginning of the PP melt will not necessarily be the same point.

7.6.5 Record the HDPE end-of-melt temperature and the PP heat of fusion in J/g.

7.6.6 Calculate the theoretical heat of fusion for 100% polypropylene for the portion of the PP melting curve that does not overlap with HDPE by:

$$Y = -0.035X^2 + 8.851X - 475.6 \quad (1)$$

Where: X = HDPE end-of-melt temperature (°C)
And $Y = \Delta H_f$ of 100% PP not overlapped with HDPE.

For $X = 142.7$, $Y = 74.7$ J/g.

7.6.7 Calculate the percentage PP by:

$$\%PP = \Delta H_f \text{ sample} / \Delta H_f \text{ 100\% PP} \times 100 \quad (2)$$

For ΔH_f sample = 2.57 J/g, %PP = 3.4 %

7.6.8 Test two specimens from different parts of the molded plaque and report the average.

7.7 *Yield Stress/Break Strain* – Test by ASTM Method D638, with a Type IV dumbbell at 2”/min crosshead speed.

7.7.1 Test five specimens, individually measured for thickness.

7.7.2 2.0-in. gage length, use crosshead to follow displacement.

7.7.3 Report five results, average, and standard deviation.

7.8 *NCLS Stress-Crack Test* – Test 1.9-mm plaque for slow crack growth with ASTM F2136 with a notch depth of 20% of the test specimen thickness and under an applied load of 4100 kPa (600 psi).

7.8.1 Test five replicates and report the average and standard deviation.

7.9 *Oxidative Induction Time (OIT)* – Test by ASTM D3895 at 200°C in oxygen in duplicate and report the average time.

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8. RETEST AND REJECTION

8.1 If any failure occurs, the material shall be retested to establish conformity in accordance to the specified property. A second failed result will result in rejection of the lot for the purposes of this specification.

9. CERTIFICATION

9.1 Upon request of the purchaser in the contract or order, a manufacturer's certification that the material was manufactured and tested in accordance with this specification, together with a report of the test results, shall be furnished at the time of shipment.

APPENDIX G

Proposed Draft Standard Specification for Recycled Content Containing HDPE Resin Formulations for Corrugated Pipe Made to AASHTO Standard M294-Recycled

DRAFT

Standard Specification for

SPECIFICATION FOR RECYCLED CONTAINING HDPE RESIN FORMULATIONS FOR CORRUGATED PIPE MADE TO AASHTO SPECIFICATION M-294-RECYCLED

AASHTO Draft Standard XXX-XX

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1. SCOPE

1.1 This specification covers the quality HDPE resin formulations containing recycled HDPE that are intended for use in AASHTO M294–Recycled approved corrugated drainage pipe, in sizes 300- to 1500-mm (12- to 60-in) diameter.

1.2 The recycled polyethylene may be either post-commercial or post-industrial.

1.3 This specification presents a set of properties to be met for the resin and test frequencies.

1.4 This specification can be used by resin containing recycled PE suppliers as part of manufacturing quality control (MQC), or by pipe manufacturers or independent bodies as manufacturing quality assurance (MQA).

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2. REFERENCED DOCUMENTS

2.1 ASTM Standards:

- D638, Test Method for Tensile Properties of Plastics
- D790, Test Method for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials
- D792, Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement
- D1238, Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer
- D1505, Test Method for Density of Plastics by the Density-Gradient Technique
- D3895, Standard Test Method for Oxidative-Induction Time of Polyolefins by Differential Scanning Calorimetry
- D4218, Test Method for Determination of Carbon Black Content in Polyethylene Compounds By the Muffle-Furnace Technique
- D4703, Practice for Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets
- D5630, Test Method for Ash Content in Plastics

- F2136, Standard Test Method for Notched, Constant Ligament-Stress (NCLS) Test to Determine Slow-Crack-Growth Resistance of HDPE Resins or HDPE Corrugated Pipe

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3. TERMINOLOGY

3.1 *Mixed-Color PCR-HDPE* – Post-Consumer Recycled HDPE that is composed of mostly colored detergent bottles.

3.2 *Natural PCR-HDPE* – Post-Consumer Recycled HDPE that is composed of milk bottles.

3.3 *Post-Consumer Recycled HDPE (PCR-HDPE)* – Polyethylene that has been discarded by consumers after use, then collected, cleaned and washed. The density is greater than 0.940 g/cm^3 and typical sources include primarily bottles and some bags.

3.4 *PCR-HDPE Regrind* – Post-Consumer Recycled HDPE that has been cleaned, washed, and ground into plastic chips.

3.5 *Post-Industrial Recycled HDPE (PIR-HDPE)* – Polyethylene that has been obtained for recycling from industrial sources. This may include industrial scrap, rejected parts, or surplus goods.

3.6 *Reprocessed PCR-HDPE* – Post-Consumer Recycled HDPE that has been cleaned, washed, melt-filtered, and pelletized. Commonly known as “Repro.”

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4. REQUIRED PROPERTIES

4.1 The required properties for corrugated pipe resins containing recycled HDPE are given in Table 1. Specific details of the tests are found in Section 7.

4.2 In addition to the properties in Table 1, the percentage recycled shall be disclosed by the resin supplier and included on the resin certification.

Table 1. Required properties for corrugated pipe resins containing recycled HDPE.

Property	Test Method	Sample	Specified Value	Frequency
Density	ASTM D1505 ASTM D792	Pellets/Plaque	>0.947- 0.955 [a] g/cm	1 per shift
Melt Index	ASTM D1238 190°C/2.16 Kg	Pellets/Plaque	<0.4 g/10 min	1 per shift
% Volatiles	175°C/1 Hour	Pellets[b]	<0.2 %	Certify[c]
% Carbon Black	ASTM D4218	Pellets/Plaque	2-4 %	1 per shift
% Ash	ASTM D5603	Pellets/Plaque	<0.5 %	1 per shift
% PP	DSC Section 7.6	Plaque	<5.0 %	1 per lot[d]
Flexural Modulus	ASTM D790	Plaque	>130,000 psi	1 per lot
Yield Stress	ASTM D638[e]	Plaque	>3500 psi	1 per lot
Break Strain	ASTM D638[f]	Plaque	>200 %	1 per lot
NCLS Stress-Crack Test	ASTM F2136	Plaque	36 hrs	1 per lot
OIT	ASTM D3895	Pellets/Plaque	50 min[g]	1 per lot
BFF Stress-Crack Test	Section 7.9 80°C/650 psi	Plaque	200 hrs	1 per lot

[a] Correct for carbon black by $D_{cor} = D - 0.0044C$, where C is % carbon black.

[b] Tested on pellet samples before melt blending.

[c] Manufacturer will provide a certificate stating their material will lose less than 0.2% of its weight when a 2.000- ± 0.100-g sample is heated at 350°F for 1 h.

[d] A production lot is defined as either a truckload, or one compartment of a railcar. Its weight is generally between 40,000 and 50,000 lbs.

[e] Strain rate shall be 2.0 in. per minute.

[f] Assume 2.0–in. gage length.

[g] A common additive package that meets this requirement is 1,000 ppm Irganox 1010 plus 1,000 ppm Irgaphos 168, or equivalent.

5. SAMPLING

5.1 A production lot shall be considered as a truckload or shipping container full of 1,500 lb boxes of resin, or one compartment of a railcar. The weight is normally between 40,000 and 50,000 lb.

5.2 A truckload shall be sampled by obtaining 0.5 lb of material from each of eight boxes, for a total of 4 lb of resin. This is about every fourth box. Combine the eight samples to form a lot sample.

5.3 A compartment of a railcar shall be sampled by obtaining 2 lb from the top and 2 lb from the bottom of the compartment. Combine the two samples to form a lot sample. Therefore, each railcar will have four lot samples for evaluation.

5.4 The entire lot sample shall be dry blended before sample preparation. A convenient way to do this is by hand in a 5-gallon plastic pail.

5.5 Divide the lot sample into two; one for immediate testing and the other for validation testing, should the need arise. The back-up sample will be kept for 90 days after the resin is sold.

5.6 Clearly label the sample and include a traceable production number and a date.

6. SAMPLE PREPARATION

6.1 Homogenize about 2 lb (900 g) of material by melt blending, preferably with a twin screw laboratory extruder. A two roll mill is also acceptable.

6.2 The blended material shall be compression molded into plaques according to ASTM D4703, with the use of a 15°C/min. cooling rate.

6.3 The plaques shall be 1.9- ± 0.08-mm (0.075- ± 0.003-in.) thick for tensile and NCLS testing, 3.2-mm (0.125-in.) thick for flexural modulus, and 1.1-mm (0.043-in.) thick for the BFF test.

6.4 The plaques shall be allowed to equilibrate at standard laboratory conditions (72°F, 50% RH) for at least 12 hours.

7. TEST METHODS

7.1 *Test Conditions* – Unless otherwise specified in the test methods or in this specification, conduct tests at the standard laboratory temperature of 23 ± 2°C (73.4 ± 3.6 °F).

7.2 *Density* – Test methods ASTM D1505 or D792 are acceptable. Make three separate determinations using separate pellets or separate portions of a plaque. The plaque thickness shall be 1.9 ± 0.08 mm (0.075 ± .003 in.). Calculate and report the average and standard deviation from the mean. Correct the density value for % carbon black by subtracting 0.0044 g/cm^3 for each percent of carbon black.

7.3 *Melt Index* – Test method ASTM D1238, using Condition 190/2.16. Make duplicate determinations on either pellets or a plaque and calculate the average.

7.4 *% Volatiles* – This test shall be performed in aluminum pans that have been heated at 350°F (177°C) until they obtain a constant weight.

7.4.1 Test duplicate samples.

7.4.2 Weigh 2.000 ± 0.100 grams of pellets or a plaque to an accuracy of three places past the decimal point into an aluminum pan.

7.4.3 Place the pan in a forced-air recirculating oven preheated and stabilized to 350 ± 5°F (177 ± 2°C) for a period of 1 h.

7.4.4 Remove the pan and let it completely cool to room temperature in a desiccator.

7.4.5 Reweigh the pan and calculate the percentage weight loss of the sample. Report the average to the nearest 0.01%.

7.5 *% Ash* - Test method ASTM D5630, Procedure B.

7.5.1 Sample size shall be 1 g.

7.5.2 Heat at least 10 minutes at 800°C.

7.5.3 Test triplicate samples.

7.6 *% Polypropylene*

7.6.1 Generate a melting curve by differential scanning calorimetry (DSC) at a heating rate of 10°C/min from room temperature to 200°C. See Figure 1.

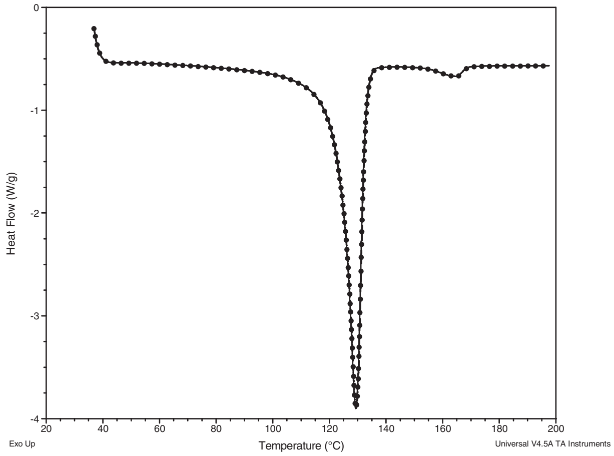

Figure 1. DSC curve of HDPE containing PP generated.

7.6.2 The curve shall be expanded to a range that includes the end of the HDPE peak and the complete PP peak. See Figure 2.

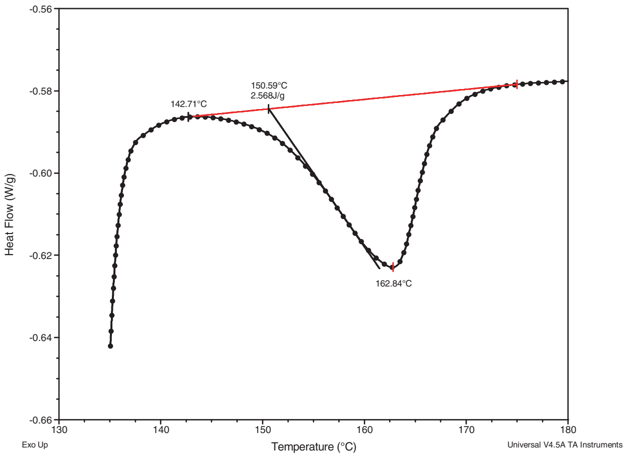

Figure 2. DSC curve of HDPE containing PP expanded.

7.6.3 Integrate the curve from a flat point before the PP melting to a point where the PP curve returns to baseline.

7.6.4 Mark the point where the HDPE melt returns to baseline. The end of the HDPE melt and the beginning of the PP melt will not necessarily be the same point.

7.6.5 Record the HDPE end-of-melt temperature and the PP heat of fusion in J/g.

7.6.6 Calculate the theoretical heat of fusion for 100% polypropylene for the portion of the PP melting curve that does not overlap with HDPE by:

$$Y = -0.035X^2 + 8.851X - 475.6 \qquad (1)$$

Where: X = HDPE end-of-melt temperature (°C)
And $Y = \Delta H_f$ of 100% PP not overlapped with HDPE.

For $X = 142.7$, $Y = 74.7$ J/g.

7.6.7 Calculate the percentage PP by:

$$\%PP = \Delta H_f \text{ sample} / \Delta H_f \text{ 100\% PP} \times 100 \qquad (2)$$

For ΔH_f sample = 2.57 J/g, %PP = 3.4 %

7.6.8 Test two specimens from different parts of the molded plaque and report the average.

7.7 *Flexural Modulus* – Test by ASTM Method D790. The test specimens shall be 2.5 in. x 0.5 in. (w) x 0.125 in. (t). A span of 2.0 in. will be used at a crosshead rate of 0.5 in. per minute. Report the 2% secant modulus. Test five replicates and report each result, along with the average and standard deviation.

7.8 *Yield Stress/Break Strain* – Test by ASTM Method D638, with a Type IV dumbbell at 2-in./min crosshead speed.

7.8.1 Test five specimens, individually measured for thickness.

7.8.2 2.0-in. gage length, use crosshead to follow displacement.

7.8.3 Report five results, average, and standard deviation.

7.9 *NCLS Stress Crack Test* – Test 1.9-mm plaque for slow-crack growth with ASTM F2136 with a notch depth of 20% of the test specimen thickness and under an applied load of 4,100 kPa (600 psi).

7.9.1 Test five replicates and report the average and standard deviation.

7.10 *Oxidative Induction Time (OIT)* – Test by ASTM D3895 at 200°C in oxygen in duplicate and report the average time.

7.11 *BFF Stress Crack Test* – This test is performed with the use of common stress crack frames in deionized water at 80°C (176°F), under an applied stress of 650 psi.

7.11.1 A bath that has never contained Igepal or other surfactants is preferred. Residual surfactant can dramatically accelerate the test.

7.11.2 The test specimen is basically an ASTM D638 Type I Dumbbell with a couple of modifications. First, a 7/32–in. hole is drilled into each head portion so that the specimens can be mounted with a screw in a conventional stress crack testing device. However, because of the holes, the tabs on each side of the hole are narrower than the reduced section of the dumbbell. Therefore, the highest stressed areas are the tabs, so all the specimens would naturally fail at the tabs. To get around this, a 6–in. square open faced mold was modified to produce plaques that were about 45 mil in the center and over 90 mil on the edges. This way, the tabs are under less stress than the reduced section so nearly all of the failures occur in the reduced section. A drawing of the fathead test specimen is shown in Figure 3.

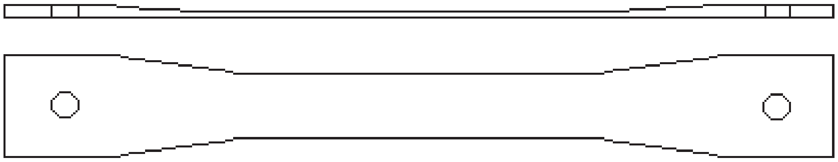

Figure 3. Fathead specimen used in the BFF test.

7.9.2 The test device is manufactured by BT Technology and is shown in Figure 4. The weight tubes were modified to allow for the higher loads used in this test.

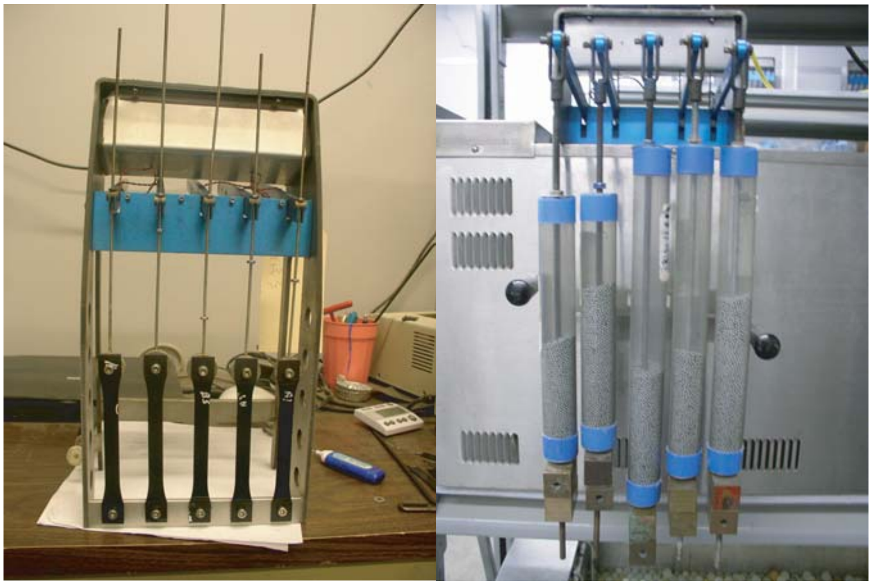

Figure 4. Load frame for the BFF test.

7.9.2 Five replicates are tested and the average and standard deviation reported.

8. RETEST AND REJECTION

8.1 If any failure occurs, the material shall be retested to establish conformity in accordance to the specified property. A second failed result will result in rejection of the lot for the purposes of this specification.

9. CERTIFICATION

9.1 Upon request of the purchaser in the contract or order, a manufacturer's certification that the material was manufactured and tested in accordance with this specification, together with a report of the test results, and the percentage recycled shall be furnished at the time of shipment.

APPENDIX H

Proposed Draft Standard Specification for Corrugated Polyethylene Drainage Pipe Containing Recycled Polyethylene, 75- to 250-mm Diameter

DRAFT

Draft Standard Specification for

Corrugated Polyethylene Drainage Pipe Containing Recycled Polyethylene, 75- to 250-mm Diameter

AASHTO Designation: M252-Recycled-XX

1. SCOPE

1.1. This specification covers the requirements and methods of test for corrugated polyethylene (PE) pipe, couplings, and fittings for use in subsurface drainage systems, storm sewers, and in surface drainage (culverts), where soil support is given to the pipe's flexible walls in all applications.

1.1.1. This standard allows for the use of recycled polyethylene in the pipe, but not in the couplings or the fittings.

1.1.2. Nominal sizes of 75 to 250 mm are included.

1.1.3. Material properties, dimensions, pipe stiffness, perforations, joining systems and forms of marking are specified.

Note 1—When polyethylene pipe is to be used in locations where the ends may be exposed, consideration should be given to combustibility of the polyethylene and the deteriorating effects of prolonged exposure to ultraviolet radiation.

1.2. The following precautionary caveat pertains only to the test method portion, Section 9 of this specification. *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. REFERENCED DOCUMENTS

2.1. *AASHTO Standard*:

- R 16, Regulatory Information for Chemicals Used in AASHTO Tests

2.2. *ASTM Standards*:

- D618, Method for Conditioning Plastics and Electrical Insulating Materials for Testing
- D638, Test Method for Tensile Properties of Plastics
- D792, Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement

- D883, Definition of Terms Relating to Plastics
- D1238, Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer
- D1505, Test Method for Density of Plastics by the Density-Gradient Technique
- D2122, Method of Determining Dimensions of Thermoplastic Pipe and Fittings
- D2412, Test Method for Determination of External Loading Characteristics of Plastic Pipe by Parallel-Plate Loading
- D3350, Standard Specification for Polyethylene Plastics Pipe and Fittings Materials
- D3895, Standard Test Method for Oxidative-Induction Time of Polyolefins by Differential Scanning Calorimetry
- D4218, Standard Test Method for Determination of Carbon Black Content in Polyethylene Compounds by the Muffle-Furnace Technique
- D4703, Practice for Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets
- D5630, Test Method for Ash Content in Plastics
- F412, Definitions of Terms Relating to Plastic Piping Systems
- F2136, Standard Test Method for Notched, Constant Ligament-Stress (NCLS) Test to Determine Slow-Crack-Growth Resistance of HDPE Resins or HDPE Corrugated Pipe

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3. TERMINOLOGY

3.1. The terminology used in this standard is original to this standard, unless otherwise noted.

3.2. *Crack* – any narrow opening or fissure in the surface that is visible to the naked eye. (ASTM F412)

3.3. *Crease* – An irrecoverable indentation; generally associated with wall buckling. (AASHTO M252)

3.4. *Mixed-Color PCR-HDPE* – Post-Consumer Recycled HDPE that is composed of mostly colored detergent bottles.

3.5. *Post-Consumer Recycled HDPE (PCR-HDPE)* – Polyethylene that has been discarded by consumers after use, then collected, cleaned, and washed. The density is greater than 0.940 g/cm^3 and typical sources include primarily bottles and some bags.

3.6. *Post-Industrial Recycled HDPE (PIR-HDPE)* – Polyethylene that has been obtained for recycling from industrial sources. This may include industrial scrap, rejected parts, or surplus goods.

3.7. *Reprocessed PCR-HDPE* – Post-Consumer Recycled HDPE that has been cleaned, washed, melt-filtered, and pelletized. Commonly known as “Repro.”

3.8. *Reworked Material* – A plastic from a processor’s own production that has been reground, pelletized, or solvated after having been previously processed by molding, extrusion, etc. (ASTM D883).

4. CLASSIFICATION

4.1. The corrugated polyethylene pipe covered by this specification is classified as follows:

4.1.1. *Type C* – This pipe shall have a full circular cross section, with a corrugated surface both inside and outside. Corrugations may be either annular or helical.

4.1.1.1. *Type CP* – This pipe shall be Type C with Class 2 perforations.

4.1.2. *Type S* – This pipe shall have a full circular cross section, with an outer corrugated pipe wall and a smooth inner liner. Corrugations may be either annular or helical. Type S pipe is not available in nominal sizes of less than 100 mm.

4.1.2.1. *Type SP* – This pipe shall be Type S with either Class 1 or Class 2 perforations.

4.2. Class 1 and Class 2 perforations are as described in Sections 7.4.1 and 7.4.2.

5. ORDERING INFORMATION

5.1. Orders using this specification shall include the following information as necessary to adequately describe the desired product:

5.1.1. AASHTO designation and year of issue;

5.1.2. Type of pipe (Section 4.1);

5.1.3. Diameter and length required, either total length or length of each piece and number of pieces;

Note 2—Type C and CP pipe less than 200 mm in diameter may be supplied coiled; coiling of Type C and CP pipe 200 mm in diameter or greater is not recommended; Type S and SP pipe is not supplied in coils.

5.1.4. Number of couplings;

5.1.5. For Type SP pipe, class of perforations (Class 2 is furnished if not specified) (Section 7.4); and

5.1.6. Certification, if desired (Section 12.1).

6. MATERIALS

6.1. *Basic Materials*:

6.1.1. *Extruded Pipe* – Pipe shall be made from resin compounds that meet the material properties found in Table 1. Compliance with the standard can be obtained by testing either the resin or the pipe. In case of disputes, all tests will be performed on compression molded plaques from the pipe. The pipe requirements are the same as the resin except for the break strain and the NCLS stress-crack resistance, which can be reduced when a plaque is made from the pipe instead of the resin. Pipe samples are commonly exposed to dust and dirt that can compromise these properties.

Table 1. Required properties for corrugated polyethylene pipe containing recycled polyethylene.

Property	Test Method	Required Value
Density	ASTM D1505 ASTM D792	≥0.948[a] g/cm
Melt Index	ASTM D1238 190°C/2.16 Kg	<0.6 g/10 min
% Carbon Black	ASTM D4218	2-4 %
% Ash	ASTM D5603	<0.75 %
% PP	DSC Section 7.6	<7.5 %
Yield Stress	ASTM D638[b]	>3500 psi
Break Strain	ASTM D638[c]	>100 % pipe
NCLS Stress-Crack Test	ASTM F2136	>15 h pipe
OIT	ASTM D3895	25 min[d]

[a] Correct for carbon black by $D_{cor} = D - 0.0044C$, where C is % carbon black.

[b] Strain rate shall be 2.0” per minute.

[c] Assume 2.0” gage length.

[d] A common additive package that meets this requirement is 1000 ppm Irganox 1010 plus 500 ppm Irgaphos 168, available from Ciba-Geigy.

6.1.2. Blow Molded Fittings – Fittings shall be made of virgin PE resin compounds meeting the requirements of ASTM D3350 and cell classification 424420C, except that the carbon-black content shall not exceed five percent. Resins that have higher cell classifications in one or more properties are acceptable provided product requirements are met.

6.1.3. Rotational Molded Fittings and Couplings – Fittings and couplings shall be made of virgin PE resins meeting the requirements of ASTM D3350 and cell classification 213320C, except that the carbon black content shall not exceed five percent. Resins that have higher cell classifications in one or more properties are acceptable provided product requirements are met.

6.1.4. Injection Molded Fittings and Couplings – Fittings and couplings shall be made of virgin PE resins meeting the requirements of ASTM D3350 and cell classification 314420C, except that the carbon black content shall not exceed five percent. Resins that have higher cell classifications in one or more properties are acceptable provided product requirements are met.

6.2. *Reworked Material* – In lieu of virgin PE, clean reworked material may be used, provided that it meets the cell class requirements as described in Section 6.1.

7. REQUIREMENTS

7.1. *Workmanship* – The pipe and fittings shall be free of foreign inclusions and visible defects as defined herein. The ends of the pipe shall be cut squarely and cleanly so as not to adversely affect joining or connecting.

7.1.1. Visible Defects – Cracks, creases, unpigmented, or nonuniformly pigmented pipe are not permissible.

7.1.2. Inner Liner – For Type S and SP pipe, the inner liner shall be fused to the outer corrugated wall at all internal corrugation crests.

7.2. *Pipe Dimensions*:

7.2.1. Nominal Size – The nominal size for the pipe and fittings is based on the nominal inside diameter of the pipe. Nominal diameters shall be sized for Type C and CP pipe in not less than 25-mm increments from 75 to 250 mm. Nominal sizes shall be sized for Type S and SP pipe in not less than 50-mm increments from 100 to 250 mm.

7.2.2. Inner Liner – For Type S and SP pipe, the inner liner shall have a minimum thickness of 0.5 mm for pipe of 100 mm and 150 mm nominal size and a minimum thickness of 0.6 mm for pipe of 200 mm and 250 mm nominal size, when measured in accordance with Section 9.13.4.

7.2.3. Inside Diameter Tolerances – The tolerance on the specified inside diameter shall be +4.5, –1.5 percent when measured in accordance with Section 9.13.1.

7.2.4. Length – Corrugated PE pipe is an extruded product and may be sold in any length agreeable to the user. Lengths shall not be less than 99 percent of the stated quantity when measured in accordance with Section 9.13.2.

7.3. *Fitting and Coupling Dimensions*:

7.3.1. The maximum allowable gap between fitting or coupling and pipe shall not exceed 3 mm unless otherwise specified.

7.3.2. All fittings and couplings shall be within an overall length dimensional tolerance of ±12 mm of the manufacturer's specified dimensions.

7.4. *Perforations* – When perforated pipe is specified, the perforations shall conform to the requirements of Class 2, unless otherwise specified in the order. Class 1 perforations are for pipe intended to be used for subsurface drainage or combination storm and underdrain. Class 2 perforations are for pipe intended to be used for subsurface drainage only. The perforations shall be cleanly cut so as not to restrict the inflow of water. Where circular perforations are preferred, the drill shall not penetrate the side walls of the corrugations. Pipe connected by couplings or bands may be unperforated within 100 mm of each end of each length of pipe.

Note 3—Pipe ordered under Class 1 perforations has no requirement as to inlet area because it specifies size, number, and location of holes. Alternate perforation patterns should be agreed to between the purchaser and manufacturer.

7.4.1. *Class 1 Perforations* – The perforations shall be approximately circular and shall have nominal diameters of not more than 5 mm for 100- and 150-mm diameter pipe and not greater than 10 mm for 200- and 250-mm diameter pipe. The holes shall be arranged in rows parallel to the axis of the pipe. The location of the perforations shall be in the valley of the outside corrugation and in each corrugation. The rows of perforations shall be arranged in two equal groups placed symmetrically on either side of the lower unperforated segment corresponding to the flow line of the pipe. The spacing of the rows shall be uniform. The distance of the centerlines of the uppermost rows above the bottom of the invert, and the inside chord lengths of the unperforated segments illustrated in Figure 1 shall be as specified in Table 2. All measurements shall be made in accordance with Section 9.13.3.

Table 2. Rows of perforations, height *H* of the centerline of the uppermost rows above the invert, and chord length *L* of unperforated segment, for Class 1 perforations.

Nominal Diameter, mm	Rows of Perforations[a]	*H*, Max,[b] mm	*L*, Min,[b] mm
75	2	35	50
100	2	45	65
150	4	70	95
200	4	94	130
250	4	120	160

[a] Minimum number of rows. A greater number of rows for increased inlet area shall be subject to agreement between purchaser and manufacture. Note that the number of perforations per meter in each row (and inlet area) is dependent on the corrugation pitch.

[b] See Figure 1 for location of dimensions *H* and *L*.

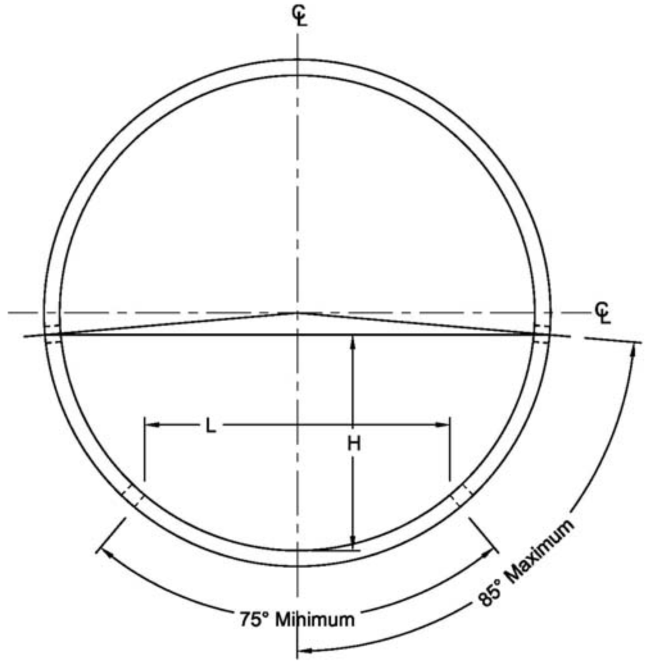

Figure 1. Requirements for perforations.

7.4.2. *Class 2 Perforations*—Circular and slotted perforations shall conform to the maximum dimensions as shown in Table 3. Perforations shall be placed in the outside valleys of the corrugations. The water inlet area shall be a minimum of 20 cm^2/m of pipe. All measurements shall be made in accordance with Section 9.13.3.

Table 3. Maximum perforation dimensions.

Nominal Pipe Diameter, mm	Drilled Hole Diameter, mm	Slotted Holes	
		Width, mm	Length, mm
75	4.75	3	25
100	4.75	3	25
150	4.75	3	25
200	6.25	3	30
250	8.00	3	30

7.5. *Density* – The density, measured on a piece of the pipe or a plaque compression molded from the pipe shall be between 0.947 and 0.955 g/cm^3 when tested according to ASTM D792 or D1505, and Section 9.2 of this standard.

7.6. *Melt Index* – The melt index (MI), measured on a piece of the pipe or a plaque made from the pipe shall be <0.4 g/10 min., when tested according to ASTM D1238 and Section 9.3 of this standard.

7.7. *% Carbon Black* – The % carbon black, measured on a piece of the pipe or a plaque made from the pipe will be between 2% and 4%, when tested according to ASTM D4218 and Section 9.4 of this standard.

7.8. *% Ash* – The % ash, measured on a piece of the pipe or a plaque made from the pipe shall be <0.75%, when tested according to ASTM D5603 and Section 9.5 of this standard.

7.9. *% Polypropylene* – The % PP, measured on a piece of the pipe or a plaque made from the pipe shall be <7.5% when tested according to Section 9.6 of this standard.

7.10. *Yield Stress* – The yield stress shall be >3500 psi when tested according to ASTM D638 and section 9.7 of this standard.

7.11. *Break Strain* – The break strain shall be >100% when tested according to ASTM D638 and section 9.7 of this standard.

7.12. *NCLS Stress-Crack Resistance* – The resistance to stress cracking shall be >15 h, when tested according to ASTM F2136 and Section 9.8 of this standard.

7.13. *Oxidative Induction Time (OIT)* – The OIT shall be >25 minutes when tested according to ASTM D3895 and Section 9.9 of this standard.

7.14. *Pipe Stiffness* – Type C pipe, as described in Section 4.1.1, shall have a minimum pipe stiffness (PS) of 240 kPa at five percent deflection; and Type S pipe, as described in Section 4.1.2, shall have a minimum pipe stiffness (PS) of 340 kPa at 5 percent deflection when tested in accordance with Section 9.10. The pipe tested shall contain perforations, if specified.

7.15. *Pipe Flattening* – There shall be no evidence of wall buckling, cracking, splitting, delamination or decrease or downward deviation in the load-deflection curve, when the pipe is tested in accordance with Section 9.11.

7.16. *Brittleness* –There shall be no cracking of the pipe wall when tested in accordance with Section 9.12 except as specified in Sections 7.16.1 and 7.16.2.

Note 4—The brittleness test is similar to that described in ASTM F405.

7.16.1. Cracks with a maximum chord length of 10 mm that originate at a perforation or at either end of the sample shall not be cause for rejection.

7.16.2. Splitting along a seam or mold parting line is not caused by brittleness and should be evaluated as a workmanship defect as described in Section 7.1.1, if no split exceeds 50 mm in chord length.

7.17. *Fitting and Coupling Requirements*:

7.17.1. The fittings and couplings shall not reduce or impair the overall integrity or function of the pipe line.

7.17.2. Common corrugated fittings include reducers, tees, wyes, and end caps.

Note 5—Only fittings and couplings supplied or recommended by the pipe manufacturer should be used.

7.17.3. Fittings and couplings shall not reduce the inside diameter of the pipe being joined by more than five percent of the nominal inside diameter. Reducer fittings shall not reduce the cross-sectional area of the smaller size.

7.17.4. Pipe connected by in-line couplers shall not separate when tested in accordance with Section 9.14.1.

7.17.5. The coupling shall not crack or crease when tested in accordance with Section 9.14.2.

7.17.6. The design of the couplers shall be such that when connected with the pipe, the axis of the assembly will be level and true when tested in accordance with Section 9.14.3.

8. CONDITIONING

8.1. *Conditioning* – Condition the specimen prior to test at 23 ± 2°C for not less than 24 hours in accordance with Procedure A in ASTM D 618 for those tests where conditioning is required, and unless otherwise specified.

8.2. *Condition* – Conduct the test in a laboratory temperature of 23 ± 2°C unless otherwise specified herein.

9. TEST METHODS

9.1. *Compression Molded Plaque* – A plaque will be prepared in accordance with ASTM D4703 and cooled by cooling method B (15 ± 5°C per minute). The plaque thickness shall be 1.9 ± 0.2 mm (0.075 ± .008 in.)

9.2. *Density* – Test methods ASTM D1505 or D792 are acceptable. Make three separate determinations using separate portions of a plaque. The plaque thickness shall be 1.9 ± 0.08 mm (0.075 ± .003 in.). Calculate and report the average and standard deviation from the mean. Correct the density value for % carbon black by subtracting 0.0044 g/cm^3 for each percent of carbon black.

9.3. *Melt Index* – Test method ASTM D1238, using Condition 190/2.16. Make duplicate determinations and calculate the average

9.4. *Carbon Black Content* – Test method ASTM D4218, with a 1g sample heated at 600°C for 3 minutes. Report the average of triplicate determinations, after subtracting the average % ash from each result.

9.5. *% Ash* - Test method ASTM D5630, Procedure B. A 1g sample heated at 800°C for 10 minutes in a porcelain crucible. Report the average of triplicate determinations.

9.6. % Polypropylene-

9.6.1. Generate a melting curve by differential scanning calorimetry (DSC) at a heating rate of 10°C/min from room temperature to 200°C. See Figure 2.

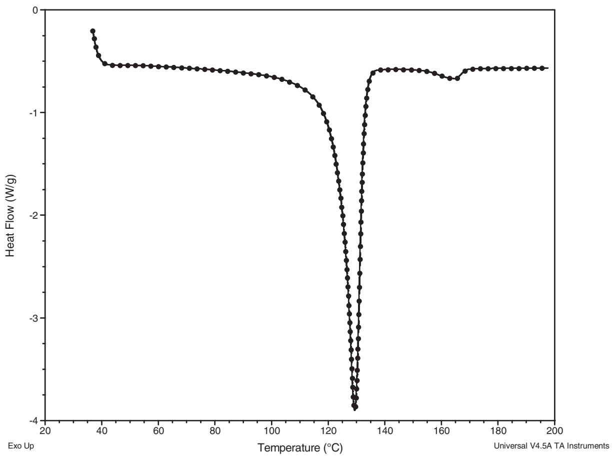

Figure 2 DSC curve of HDPE containing PP generated.

9.6.2. The curve shall be expanded to a range that includes the end of the HDPE peak and the complete PP peak. See Figure 3.

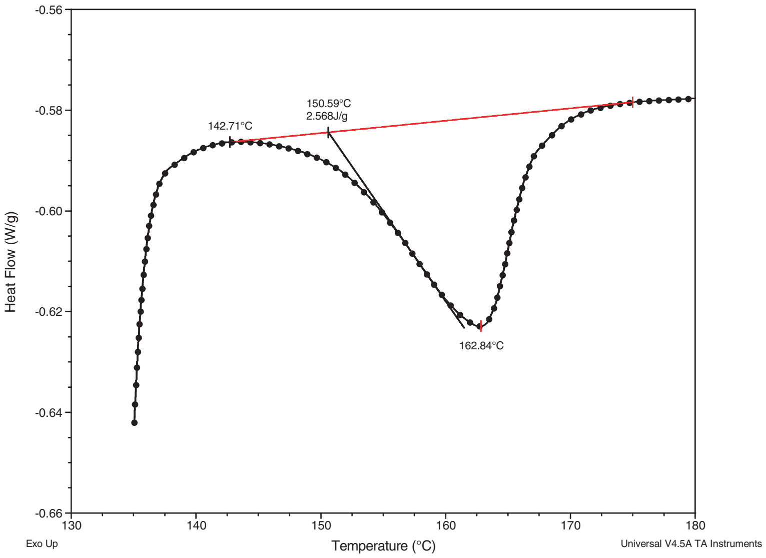

Figure 3. DSC curve of HDPE containing PP expanded.

9.6.3. Integrate the curve from a flat point before the PP melting to a point where the PP curve returns to baseline.

9.6.4. Mark the point where the HDPE melt returns to baseline. The end of the HDPE melt and the beginning of the PP melt will not necessarily be the same point.

9.6.5. Record the HDPE end-of-melt temperature and the PP heat of fusion in J/g.

9.6.6. Calculate the theoretical heat of fusion for 100% polypropylene for the portion of the PP melting curve that does not overlap with HDPE by:

$$Y = -0.035X^2 + 8.851X - 475.6 \qquad (1)$$

Where: X = HDPE end-of-melt temperature (°C)
And $Y = \Delta H_f$ of 100% PP not overlapped with HDPE.

For $X = 142.7$, $Y = 74.7$ J/g.

9.6.7. Calculate the percentage PP by:

$$\%PP = \Delta H_f \text{ sample} / \Delta H_f \text{ 100\% PP} \times 100 \qquad (2)$$

For ΔH_f sample = 2.57 J/g, %PP = 3.4 %

9.6.8. Test two specimens from different parts of the molded plaque and report the average.

9.7. *Yield Stress/Break Strain* – Test by ASTM Method D638, with a Type IV dumbbell at 2 in./min crosshead speed. Measure the thickness of the five specimens separately to generate the yield stress. Assume a gage length of 2.0 in. for % Strain. Report the average and standard deviation.

9.8. *NCLS Stress-Crack Test* – Test specimens from a 1.9 ± 0.2 mm (0.075 ± 0.008 in.) thick plaque for slow-crack growth by ASTM F2136 with a notch depth of 20% of the test specimen thickness and under an applied stress of 4100 kPa (600 psi). Test five replicates and report the average and standard deviation.

9.9. *Oxidative Induction Time (OIT)* – Test by ASTM D3895 at 200°C in oxygen in duplicate and report the average time.

9.10. *Pipe Stiffness* – Select a pipe specimen and test for pipe stiffness (PS) as described in ASTM D2412, with the following exceptions.

9.10.1. The test specimen shall be 300- ± 10-mm long, cut to include full corrugations.

9.10.2. Locate the specimen in the loading machine with an imaginary line connecting the two seams formed by the corrugation mold (end view) parallel to the loading plates. The specimen must lie flat on the plate within 1 mm and may be straightened by hand bending at room temperature to accomplish this.

9.10.3. The deflection indicator shall be readable and accurate to ±0.03 mm.

9.10.4. The residual curvature found in corrugated pipe, especially that furnished in coils, frequently results in an erratic load/deflection curve. When this occurs, the beginning point for deflection measurements shall be at a load of 20 ± 5 Newtons (4.5 ± 1 lbf.). This point shall be considered as the origin of the load/deflection curve.

Note 6—The parallel plates must exceed the length of the test specimen as specified above.

Note 7—Additional pipe specimens may be tested at other orientations for pipe stiffness and flattening if desired.

9.11. *Pipe Flattening* – Flatten the pipe specimen from Section 9.1 until the vertical inside diameter is reduced by 20%. The rate of loading shall be the same as in Section 9.1. The specimen shall fail if wall buckling, cracking, splitting, or delamination is observed with the unaided eye or if there is a decrease or downward deviation in load-deflection curve at 20% or less deflection. The load-deflection curve shall be carried beyond 20% deflection so that the shape of the curve at 20% deflection can be determined.

9.12. *Brittleness* – Test two samples of pipe at an impact of 45 joules between two flat parallel plates using the apparatus depicted in Figure 4.

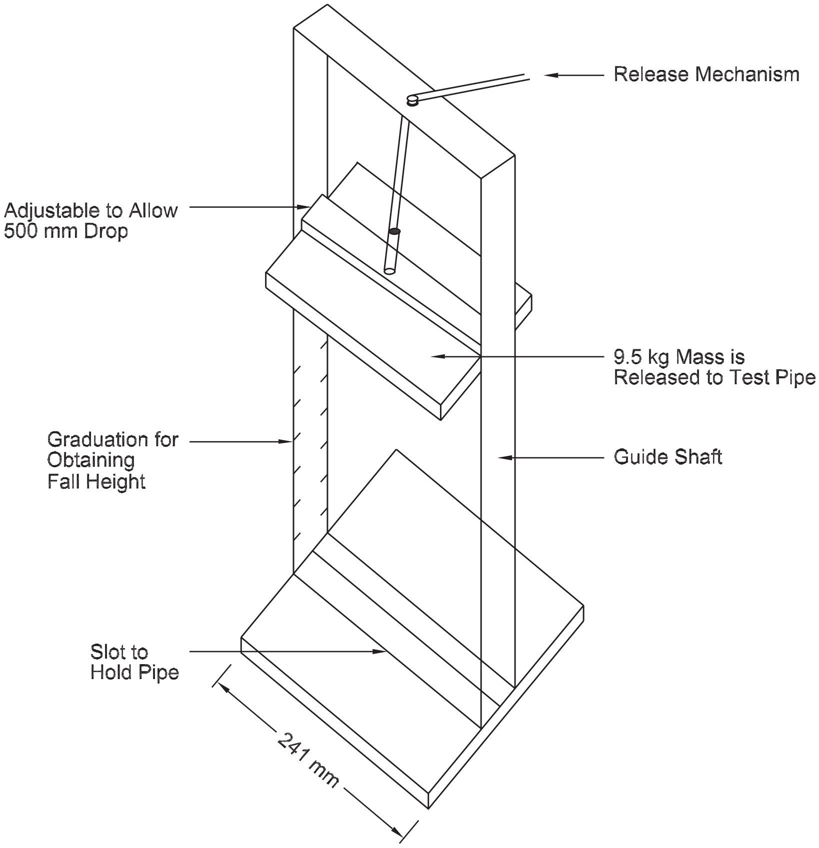

Figure 4. Brittleness test device.

9.12.1. Cut the sample specimens 150-mm long from one continuous length.

9.12.2. Condition specimens at –4°C ± 2°C for a minimum of one hour.

9.12.3. Set the 9.5-kg top plate for a free fall of 500 mm to the uppermost surface of the specimen.

9.12.4. Locate the specimen on the bottom plate with the plane of the corrugated seams parallel to the plate.

9.12.5. Drop the upper plate and impact test the specimen within 30 seconds of removal from the conditioning environment.

9.12.6. Remove and inspect in accordance with the requirements of Section 7.16.

9.13. *Pipe Dimensions*:

9.13.1. *Inside Diameter* – Measure the inside diameter of two sections of pipe with a tapered plug in accordance with ASTM D 2122. Alternatively, measure the inside diameter of two sections, with a suitable device accurate to ±0.2 mm the mold partline and 90 degrees to it, and average the measurements.

9.13.2. *Length* – Measure pipe with any suitable device accurate to 0.2%. Make all measurements on the pipe while it is stress free and at rest on a flat surface in a straight line. The length measurements may be taken at ambient temperature.

9.13.3. *Perforations* – Measure dimensions of perforations on a straight specimen with no external forces applied. Make linear measurements with instruments accurate to 0.2 mm.

9.13.4. *Inner Liner* – Measure the thickness of the inner liner with a digital micrometer or ultrasonic thickness gauge in accordance with ASTM D2122.

9.14. *Couplings*:

9.14.1. *Joint Integrity* – This test is limited to only Type C and Type CP pipe supplied in coils. Assemble couplings to appropriate pipe in accordance with the manufacturer’s recommendations. Use pipe samples at least 150 mm in length. Vertically suspend two pipe lengths connected by the joint couplings along their longitudinal axis. Then hang a tare mass from the lower end of the assembled pipe specimen for three minutes. Apply the test mass gently. Verify that the joint will support a mass along the pipe axis equal to 0.090 kg/mm of the nominal inside diameter, without separating. Test two coupling of each type.

9.14.2. *Strength* – Assemble each coupling to the appropriate pipe in accordance with the manufacturer’s recommendations. Use pipe samples at least 150 mm in length. Load the connected pipe and coupling between parallel plates at the rate of 12.5 mm/minute until the vertical inside diameter is reduced by at least 20% of the nominal diameter of the coupling. Inspect for damage while at the specified deflection and after load removal, and report the results of this inspection.

9.14.3. *Alignment* – Assure that the assembly or joint is correct and complete; if the pipe is bent, it should be hand-straightened prior to performing this test. Lay the assembly or joint on a flat surface and verify that it will accommodate straight-line flow.

10. INSPECTION AND RETEST

10.1. *Inspection* – Inspection of the material shall be made as agreed upon by the purchaser and the seller as part of the purchase contract.

10.2. *Retest and Rejection* – If any failure to conform to these specifications occurs, the pipe or fittings or couplings may be retested to establish conformity in accordance with agreement between the purchaser and seller. Individual results, not averages, constitute failure.

11. MARKING

11.1. All pipe shall be clearly marked at intervals of not more than 3.5 m, and fittings and couplings shall be clearly marked as follows:

11.1.1. Manufacturer's name or trademark,

11.1.2. Nominal size,

11.1.3. The specification designation AASHTO M252-Recycled,

11.1.4. The plant designation code,

11.1.5. The percentage recycled, and

11.1.6. The date of manufacture or an appropriate code. If a date code is used, a durable manufacturer sticker that identifies the actual date of manufacture shall be adhered to the inside of each length of pipe.

Note 8—A durable sticker is one that is substantial enough to remain in place and be legible through installation of the pipe.

12. QUALITY ASSURANCE

12.1. A manufacturer's certification that the product was manufactured, tested, and supplied in accordance with this specification and containing the percentage recycled shall be signed by a person authorized by the manufacturer.

APPENDIX I

Proposed Draft Standard Specification for Corrugated Polyethylene Drainage Pipe Containing Recycled Polyethylene, 300- to 1,500-mm Diameter

Draft Standard Specification for

Corrugated Polyethylene Drainage Pipe Containing Recycled Polyethylene, 300- to 1500-mm Diameter

AASHTO Designation: AASHTO M294-Recycled - XX

1. SCOPE

1.1. This specification covers the requirements and methods of tests for corrugated polyethylene (PE) pipe, couplings, and fittings for use in surface and subsurface drainage applications.

1.1.1. This standard allows for the use of recycled polyethylene in the pipe, but not in the couplings nor the fittings.

1.1.2. Nominal sizes of 300 to 1500 mm are included.

1.1.3. Materials, workmanship, dimensions, pipe stiffness, slow-crack-growth resistance, joining systems, brittleness, and form of markings are specified.

1.2. Corrugated polyethylene pipe is intended for surface and subsurface drainage applications where soil provides support to its flexible walls. Its major use is to collect or convey drainage water by open gravity flow, as culverts, storm drains, etc.

Note 1—When polyethylene pipe is to be used in locations where the ends may be exposed, consideration should be given to protection of the exposed portions due to combustibility of the polyethylene and the deteriorating effects of prolonged exposure to ultraviolet radiation.

1.3. This specification does not include requirements for bedding, backfill, or earth-cover load. Successful performance of this product depends upon proper type of bedding and backfill, and care in installation. The structural design of corrugated polyethylene pipe and the proper installation procedures are given in AASHTO's *LRFD Bridge Design Specifications,* Section 12, and *LFRD Bridge Construction Specifications,* Section 30, respectively. Upon request of the user or engineer, the manufacturer shall provide profile wall section detail required for a full engineering evaluation.

1.4. The following precautionary caveat pertains only to the test method portion, Section 9 of this specification. *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. REFERENCED DOCUMENTS

2.1. *AASHTO Standards*:

- R 16, Regulatory Information for Chemicals Used in AASHTO Tests
- *AASHTO LRFD Bridge Construction Specifications*

- *AASHTO LRFD Bridge Design Specifications*
- *Standard Specifications for Highway Bridges*

2.2. *ASTM Standards*:

- D618, Standard Practice for Conditioning Plastics for Testing
- D638, Standard Test Method for Tensile Properties of Plastics
- D792, Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement
- D883, Standard Terminology Relating to Plastics
- D1238, Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer
- D1505, Test Method for Density of Plastics by the Density-Gradient Technique
- D2122, Standard Test Method for Determining Dimensions of Thermoplastic Pipe and Fittings
- D2412, Standard test Method for Determination of External Loading Characteristics of Plastic Pipe by Parallel-Plate Loading
- D2444, Standard Test Method for Determination of the Impact Resistance of Thermoplastic Pipe and Fittings by Means of a Tup (Falling Weight)
- D3212, Standard Specification for Joints for Drain and Sewer Plastic Pipes Using Flexible Elastomeric Seals
- D3350, Standard Specification for Polyethylene Plastics Pipe and Fittings Materials
- D3895, Standard Test Method for Oxidative-Induction Time of Polyolefins by Differential Scanning Calorimetry
- D4218, Standard Test Method for Determination of Carbon-Black Content in Polyethylene Compounds By the Muffle-Furnace Technique.
- D4703, Practice for Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets
- D5630, Test Method for Ash Content in Plastics
- F412, Standard Terminology Relating to Plastic Piping Systems
- F477, Standard Specification for Elastomeric Seals (Gaskets) for Joining Plastic Pipe
- F2136, Standard Test Method for Notched Constant Ligament-Stress (NCLS) Test to Determine Slow-Crack-Growth Resistance of HDPE Resins or HDPE Corrugated Pipe

3. TERMINOLOGY

3.1. The terminology used in this standard is original to this standard, unless otherwise noted.

3.2. *Buckling* – any reverse curvature or deformation in the pipe wall that reduces the load-carrying capability of the pipe. Any decrease or downward deviation in the pipe stiffness test curve shall be considered a wall-buckling point.

3.3. *Crack* – any narrow opening or fissure in the surface that is visible to the naked eye. (ASTM F412)

3.4. *Crease* – an irrecoverable indentation, generally associated with wall buckling.

3.5. *Delamination*—A separation between the inner liner and outer corrugated wall of Type S pipe as evidenced by a visible gap extending completely through at least one corrugation valley at any point around the circumference of the pipe. For Type D pipe, delamination is a separation of the inner and outer wall as evidenced by a visible gap extending completely between the internal supports and inner or outer wall at any point around the circumference of the pipe.

3.6. *Mixed-color PCR-HDPE* – Post-Consumer Recycled HDPE that is composed of mostly colored detergent bottles.

3.7. *Polyethylene (PE) plastics* – plastics based on polymers made with ethylene as essentially the sole monomer (ASTM D883).

3.8. *Post-consumer recycled HDPE (PCR-HDPE)* – Polyethylene that has been discarded by consumers after use, then collected, cleaned and washed. The density is greater than 0.940 g/cm^3 and typical sources include primarily bottles and some bags.

3.9. *Post-industrial recycled HDPE (PIR-HDPE)* – Polyethylene that has been obtained for recycling from industrial sources. This may include industrial scrap, rejected parts, or surplus goods.

3.10. *Reprocessed PCR-HDPE* – Post-Consumer Recycled HDPE that has been cleaned, washed, melt-filtered, and pelletized. Commonly known as “Repro.”

3.11. *Reworked plastic* – A plastic from a processor’s own production that has been reground, pelletized, or solvated after having been previously processed by molding, extrusion, etc. (ASTM D883).

3.12. *Virgin polyethylene material* – PE plastic material in the form of pellets, granules, powder, floc, or liquid that has not been subject to use or processing other than required for initial manufacture.

3.13. *Slow crack growth* – A phenomenon by which a stress crack may form. A stress crack is an external or internal crack in plastic caused by tensile stresses less than its short-time mechanical strength.

4. CLASSIFICATION

4.1. The corrugated polyethylene pipe covered by this specification is classified as follows:

4.1.1. *Type C* – This pipe shall have a full circular cross section, with a corrugated surface both inside and outside. Corrugations shall be annular.

4.1.1.1. *Type CP* – This pipe shall be Type C with perforations.

4.1.2. *Type S* – This pipe shall have a full circular cross section, with an outer corrugated pipe wall and a smooth inner liner. Corrugations shall be annular.

4.1.2.1. *Type SP* – This pipe shall be Type S with perforations.

4.1.3. *Type D* – This pipe shall consist of an essentially smooth inner wall/liner braced circumferentially or spirally with projections or ribs joined to an essentially smooth outer wall.

4.1.3.1. *Type DP* – This pipe shall be Type D with perforations.

4.2. Two classes of perforations are as described in Sections 7.3.1 and 7.3.2.

5. ORDERING INFORMATION

5.1. Orders using this specification shall include the following information as necessary to adequately describe the desired product:

5.1.1. AASHTO designation and year of issue;

5.1.2. Type of pipe (Section 4.1);

5.1.3. Diameter and length required, either total length or length of each piece and number of pieces;

5.1.4. Number of couplings;

5.1.5. Class of perforations (Class 2 is furnished if not specified) (Section 7.3); and

5.1.6. Certification, if desired (Section 12.1).

6. MATERIALS

6.1. *Basic Materials*:

6.1.1. *Extruded Pipe* – Pipe shall be made from resin compounds that meet the requirements shown in Table 1. Compliance with the standard can be obtained by testing either the resin or the pipe. In case of disputes, all tests will be performed on compression molded plaques from the pipe. The pipe requirements are the same as the resin except for the break strain and the NCLS stress crack resistance, which can be reduced when a plaque is made from the pipe instead of the resin. Pipe samples are commonly exposed to dust and dirt that can compromise these properties.

6.1.2. **Table 1**. Required properties for corrugated polyethylene pipe containing recycled polyethylene.

Property	Test Method	Required Value
Density	ASTM D1505 ASTM D792	>0.947-0.955[a] g/cm
Melt Index	ASTM D1238 190°C/2.16 Kg	<0.4 g/10 min
% Carbon Black	ASTM D4218	2-4 %
% Ash	ASTM D5603	<0.5 %
% PP	DSC Section 9.6	<5.0 %
Yield Stress	ASTM D638[b]	>3500 psi
Break Strain	ASTM D638[c]	>200% resin >150% pipe
NCLS Stress Crack Test	ASTM F2136	36 hrs resin 30 hrs pipe
BFF Stress Crack Test	Section 9.9 80°C/650 psi	200 hrs
OIT	ASTM D3895	50 min[d]

[a] Correct for carbon black by $D_{cor} = D - 0.0044C$, where C is % carbon black.

[b] Strain rate shall be 2.0" per minute.

[c] Assume 2.0" gage length.

[d] A common additive package that meets this requirement is 1000 ppm Irganox 1010 plus 1000 ppm Irgaphos 168, available from Ciba-Geigy.

6.1.3. *Blow Molded Fittings* – Fittings shall be made of virgin PE resin compounds meeting the requirements of ASTM D3350 and cell classification 435400C, except that the carbon black content shall not be less than two percent but does not exceed five percent, and the density shall not be less than 0.948 gm/cc nor greater than 0.955 gm/cc. Resins that have higher cell classifications in one or more properties, with the exception of density, are acceptable provided product requirements are met. For slow-crack-growth resistance, acceptance of resins shall be determined by using the Notched Constant Ligament-Stress (NCLS) test according to the procedure described in Section 9.8. For slow-crack-growth resistance, acceptance of pipe shall be determined by tests on finished pipe using the Notched Constant Ligament-Stress (NCLS) test according to the procedure described in Section 9.8 The average failure time of the pipe liner shall not be less than 18 hours.

Note: If profile geometries do not have a flat portion of sufficient length to produce an NCLS tensile specimen, the pipe sample should be ground and a test plaque made in accordance with ASTM D1928 and tested per ASTM F2136. The average failure time of the test specimens from plaques shall not be less than 24 hours.

6.1.4. *Rotational Molded Fittings and Couplings* – Fittings and couplings shall be made of virgin PE resins meeting the requirements of ASTM D3350 and cell classification 213320C, except that the carbon-black content shall not be less than two percent but does not exceed five percent. Resins that have higher cell classifications in one or more properties are acceptable provided product requirements are met.

6.1.5. *Injection Molded Fittings and Couplings* – Fittings and couplings shall be made of virgin PE resins meeting the requirements of ASTM D3350 and cell classification 314420C, except that the carbon-black content shall not be less than two percent but does not exceed five percent. Resins that have higher cell classifications in one or more properties are acceptable provided product requirements are met.

6.2. *Reworked Plastic* – In lieu of virgin PE, clean reworked plastic may be used by the manufacturer, provided that it meets the cell class requirements as described in Section 6.1.

6.3. *Resin Blending* – When blended resins are used, the components of the blend must be virgin PE and the final blend must meet all of the requirements of Section 6.1.1 for extruded pipe and blow molded fittings, Section 6.1.3 for rotational molded fittings and couplings, and Section 6.1.5 for injection molded fittings and couplings.

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7. REQUIREMENTS

7.1. *Workmanship* – The pipe and fittings shall be free of foreign inclusions and visible defects as defined herein. The ends of the pipe shall be cut squarely and cleanly so as not to adversely affect joining or connecting.

7.1.1. *Visible Defects*—Cracks, creases, delaminations, and unpigmented or nonuniformly pigmented pipe are not permissible in the pipe or fittings as furnished. There shall be no evidence of delamination when tested in accordance with Section 9.16.

7.2. *Pipe Dimensions*:

7.2.1. *Nominal Size*—The nominal size for the pipe and fittings is based on the nominal inside diameter of the pipe. Nominal diameters shall be 300, 375, 450, 525, 600, 675, 750, 900, 1050, 1200, 1350, and 1500 mm.

7.2.2. *Wall Thickness*—The inner wall of Type S pipe, and both inner and outer walls of Type D pipe, shall have the following minimum thicknesses, when measured in accordance with Section 9.15.4:

Diameter, mm	Wall Thickness, mm
300	0.9
375	1.0
450	1.3
525	1.5
600	1.5
675	1.5
750	1.5
900	1.7
1050	1.8
1200	1.8
1350	2.0
1500	2.0

7.2.3. *Inside Diameter Tolerances*—The tolerance on the specified inside diameter shall be 4.5 percent oversize and 1.5 percent undersize, but not more than 37 mm oversize when measured in accordance with Section 9.15.1.

7.2.4. *Length*—Corrugated PE pipe may be sold in any length agreeable to the user. Lengths shall not be less than 99 percent of the stated quantity when measured in accordance with Section 9.15.2.

7.3. *Perforations*—When perforated pipe is specified, the perforations shall conform to the requirements of Class 2, unless otherwise specified in the order. Class 1 perforations are for pipe intended to be used for subsurface drainage or combination storm and underdrain. Class 2 perforations are for pipe intended to be used for subsurface drainage only. The perforations shall be cleanly cut so as not to restrict the inflow of water. Pipe connected by couplings or bands may be unperforated within 100 mm of each end of each length of pipe. Pipe connected by bell and spigot joints may not be perforated in the area of the bells and spigots.

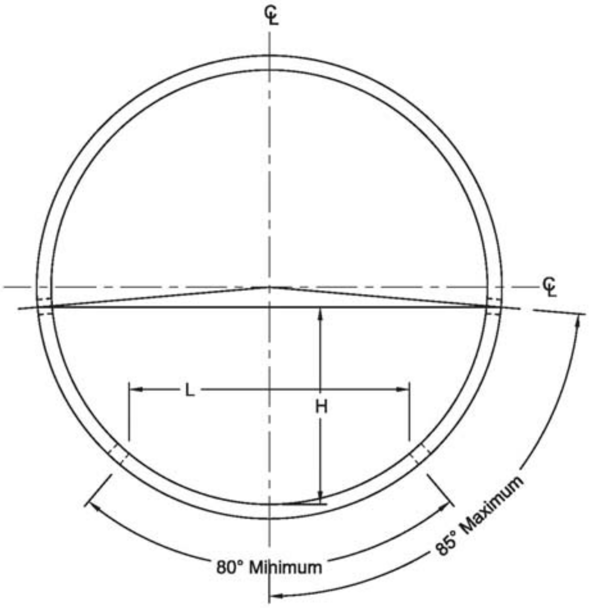

Figure 1. Requirements for perforations.

Table 2. Rows of perforations, height *H* of the centerline of the uppermost rows above the invert, and chord length *L* of unperforated segment, for Class 1 perforations.

Nominal Diameter, mm	Rows of Perforations[a]	*H*, Max,[b] mm	*L*, Min,[b] mm
300	6	138	192
375	6	172	240
450	6	207	288
525	6	241	336
600 and larger	8	[c]	[c]

[a] Minimum number of rows. A greater number of rows for increased inlet area shall be subject to agreement between purchaser and manufacturer. Note that the number of perforations per meter in each row (and inlet area) is dependent on the corrugation pitch.

[b] See Figure 1 for location of dimensions *H* and *L*.

[c] *H*(max) = 0.46*D*; *L*(min) = 0.64D, where *D* = nominal diameter of pipe, mm.

7.3.1. *Class 1 Perforations* – The perforations shall be circular and shall have nominal diameters of at least 5 mm but not greater than 10 mm and shall be arranged in rows parallel to the axis of the pipe. For Type CP and SP pipe, the perforations shall be located in the external valleys with perforations in each row for each corrugation. (The perforations shall not cut into the corrugation sidewalls.) For Type DP pipe, perforations shall be located in the center of the cells. The perforations shall not cut into the vertical sections of the cells. The rows of perforations shall be arranged in two equal groups placed symmetrically on either side of the lower unperforated segment corresponding to the flow line of the pipe. The spacing of the rows shall be uniform. The distance between the centerlines of the rows shall not be less than 25 mm. The minimum number of longitudinal rows of perforations, the maximum height of the centerlines of the uppermost rows of perforations above the bottom of the invert, and the inside chord lengths of the unperforated segments illustrated in Figure 1 shall be as specified in Table 2.

7.3.2. *Class 2 Perforations* – Circular perforations shall be a minimum of 5 mm and shall not exceed 10 mm in diameter. The width of slots shall not exceed 3 mm. The length of slots shall not surpass 70 mm for 300-mm and 375-mm pipe and 75-mm for 450-mm and larger pipe. Perforations shall be placed in the external valleys for Type CP and SP pipe and in the center of the cells for Type DP pipe. Perforations shall be uniformly spaced along the length and circumference of the pipe. The water inlet area shall be a minimum of 30 cm^2/m for pipe sizes 300 to 450 mm and 40 cm^2/m for pipe sizes larger than 450 mm. All measurements shall be made in accordance with Section 9.15.3.

7.4. *Density* – The density, measured on a piece of the pipe or a plaque compression molded from the pipe shall be between 0.947 and 0.955 g/cm^3 when tested according to ASTM D792 or D1505, and Section 9.2 of this standard.

7.5. *Melt Index* – The melt index (MI), measured on a piece of the pipe or a plaque made from the pipe shall be < 0.4 g/10 min., when tested according to ASTM D1238 and Section 9.3 of this standard.

7.6. *% Carbon Black* – The % carbon black, measured on a piece of the pipe or a plaque made from the pipe will be between 2 and 4%, when tested according to ASTM D4218 and Section 9.4 of this standard.

7.7. *% Ash* - The percentage ash, measured on a piece of the pipe or a plaque made from the pipe shall be <0.5%, when tested according to ASTM D5603 and Section 9.5 of this standard.

7.8. *% Polypropylene* – The % PP, measured on a piece of the pipe or a plaque made from the pipe shall be <5.0% when tested according to Section 9.6 of this standard.

7.9. *Yield Stress* – The yield stress shall be >3500 psi when tested according to ASTM D638 and section 9.7 of this standard.

7.10. *Break Strain* – The break strain shall be >200% on a plaque made from the resin and >150% on a plaque made from the pipe when tested according to ASTM D638 and section 9.7 of this standard.

7.11. *NCLS Stress-Crack Resistance* – The resistance to slow-crack growth in the presence of a face notch shall be >36 hrs on a plaque made from the resin and >30 hrs on a plaque from the pipe, when tested according to ASTM F2136 and Section 9.8 of this standard.

7.12. *BFF Stress-Crack Resistance* – The resistance to slow-crack growth in the absence of a face notch shall be >200 hrs when tested according to Section 9.9 of this standard.

7.13. *Oxidative Induction Time (OIT)* – The OIT shall be >50 minutes when tested according to ASTM D3895 and Section 9.10 of this standard.

7.14. *Pipe Stiffness*—The pipe shall have a minimum pipe stiffness at five percent deflection as follows when tested in accordance with Section 9.11:

Diameter, mm	Pipe Stiffness, kPa
300	345
375	290
450	275
525	260
600	235
750	200

900	160
1050	150
1200	140
1350	125
1500	110

7.15. *Pipe Flattening*—There shall be no evidence of wall buckling, cracking, splitting, delamination or decrease or downward deviation in the load-deflection curve when the pipe is tested in accordance with Section 9.12.

7.16. *Brittleness*—Pipe specimens shall not crack or split when tested in accordance with Section 9.13. Five non-failures out of six impacts will be acceptable.

7.17. *Fitting Requirements*:

7.17.1. The fittings shall not reduce or impair the overall integrity or function of the pipe line.

7.17.2. Common corrugated fittings include in-line joint fittings; such as couplings and reducers, and branch or complimentary assembly fittings; such as tees, wyes, and end caps. These fittings are installed by various methods.

7.17.3. All fittings shall be within an overall length dimensional tolerance ±12 mm of the manufacturer's specified dimensions when measured in accordance with Section 9.15.2.

7.17.4. Fittings shall not reduce the inside diameter of the pipe being joined by more than 12 mm. Reducer fittings shall not reduce the cross-sectional area of the small size.

7.17.5. Couplings shall be corrugated to match the pipe corrugations and shall provide sufficient longitudinal strength to preserve pipe alignment and prevent separation at the joints. Couplings shall be bell and spigot or split collar. Split couplings shall engage at least two full corrugations on each pipe section.

7.17.6. The design of the fittings shall be such that when connected with the pipe, the axis of the assembly will be level and true when tested in accordance with Section 9.14.1.

7.17.7. Other types of coupling bands or fastening devices which are equally effective as those described, and which comply with the joint performance criteria of AASHTO's *LRFD Bridge Construction Specifications*, Section 26, may be used when approved by the purchaser.

7.18. Only fittings supplied or recommended by the pipe manufacturer should be used. Fabricated fittings should be supplied with joints compatible with the overall system. All joints shall meet the requirements of a Soiltight Joint unless otherwise specified by the owner/designer.

7.18.1. *Soiltight joints* are specified as a function of opening size, channel length, and backfill particle size. If the size of the opening exceeds 3 mm, the length of the channel must be at least four times the size of the opening. A backfill material containing a high percentage of fine-graded soils requires investigation for the specific type of joint to be used to guard against soil infiltration. Information regarding joint soiltightness criteria can be found in AASHTO's *LRFD Bridge Construction Specifications*, Section 26, "Metal Culverts."

7.18.2. *Silt-tight joints* should be used where the backfill material has a high percentage of fines. Silt-tight bell and spigot joints will utilize an elastomeric rubber seal meeting ASTM F477. Silt-tight joints must be designated to pass a laboratory pressure test of at least 14 kPa (2.0 psi).

7.18.3. *Watertight joints* must meet a 74 kPa (10.7 psi) laboratory test per ASTM D 3212 and utilize a bell and spigot design with a gasket meeting ASTM F477.

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8. CONDITIONING

8.1. *Conditioning* – Condition the specimen prior to test at 21 to 25°C for not less than 24 hours in accordance with Procedure A in ASTM D618 for those tests where conditioning is required, and unless otherwise specified.

8.2. *Conditions* – Conduct all tests at a laboratory temperature of 21 to 25°C unless otherwise specified herein.

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9. TEST METHODS

9.1. *Test Conditions* – Unless otherwise specified in the test methods or in this specification, conduct tests at the standard laboratory temperature of 23 ± 2°C (73.4 ± 3.6°F).

9.2. *Density* – Test methods ASTM D1505 or D792 are acceptable. Make three separate determinations using separate pellets or separate portions of a plaque. The plaque thickness shall be 1.9 ± 0.08 mm (0.075 ± .003 in.). Calculate and report the average and standard deviation from the mean. Correct the density value for % carbon black by subtracting 0.0044 g/cm^3 for each percent of carbon black.

9.3. *Melt Index* – Test method ASTM D1238, using Condition 190/2.16. Make duplicate determinations on either pellets or a plaque and calculate the average.

9.4. *% Carbon Black* – Test method ASTM D4218 with 1g samples. Make duplicate determinations on either pellets or a plaque and calculate the average.

9.5. *% Ash* - Test method ASTM D5630, Procedure B.

9.5.1. Porcelain crucibles shall be used.

9.5.2. Sample size shall be 1g.

9.5.3. Heat at least 10 minutes at 800°C.

9.6. % Polypropylene

9.6.1. Generate a melting curve by differential scanning calorimetry (DSC) at a heating rate of 10°C/min from room temperature to 200°C. See Figure 2.

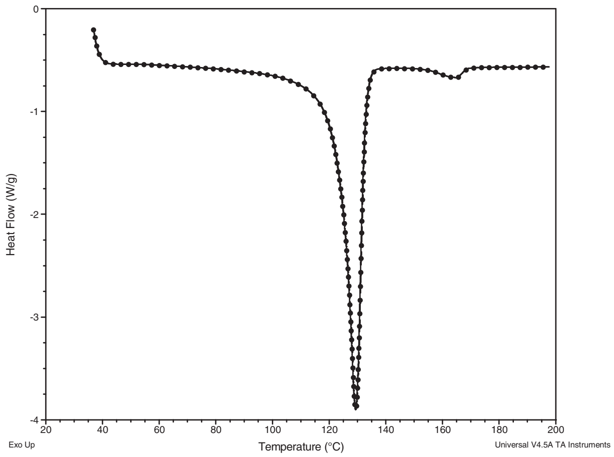

Figure 2. DSC curve of HDPE containing PP generated.

9.6.2. Expand the curve to a range that includes the end of the HDPE peak and the complete PP peak. See Figure 3.

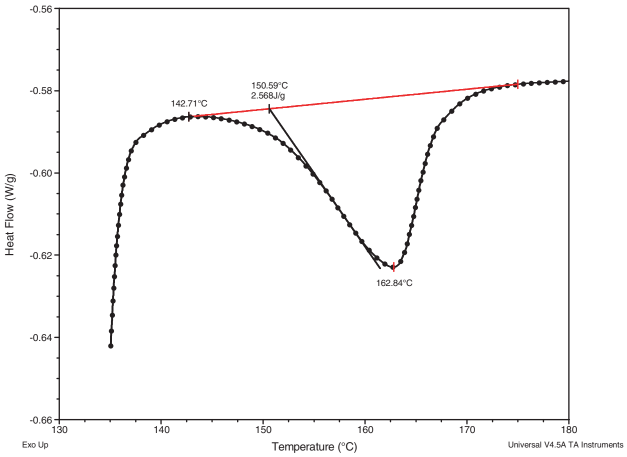

Figure 3. DSC curve of HDPE containing PP expanded.

9.6.3. Integrate the curve from a flat point before the PP melting to a point where the PP curve returns to baseline.

9.6.4. The point where the HDPE melt returns to baseline shall be marked. The end of the HDPE melt and the beginning of the PP melt will not necessarily be the same point.

9.6.5. Record the HDPE end-of-melt temperature and the PP heat of fusion in J/g.

9.6.6. Calculate the theoretical heat of fusion for 100% polypropylene for the portion of the PP melting curve that does not overlap with HDPE by:

$$Y = -0.035X^2 + 8.851X - 475.6 \qquad (1)$$

Where: X = HDPE end-of-melt temperature (°C)

And $Y = \Delta H_f$ of 100% PP not overlapped with HDPE.
For X = 142.7, Y = 74.7 J/g.

9.6.7. Calculate the percentage PP by:
%PP = ΔH_f of sample/ ΔH_f of 100% PP x 100
For ΔH_f of sample = 2.57 J/g, %PP = 2.57/74.7 x 100 = 3.4%

9.6.8. Test two specimens from different parts of the molded plaque and report the average.

9.7. *Yield Stress/Break Strain* – Test by ASTM Method D638, with a Type IV dumbbell at 2"/min crosshead speed.

9.7.1. Test five specimens, individually measured for thickness.

9.7.2. 2.0-in. gage length, use crosshead to follow displacement.

9.7.3. Report five results, average, and standard deviation.

9.8. *NCLS Stress-Crack Test*– Test 1.9-mm plaque for slow-crack growth with ASTM F2136 with a notch depth of 20% of the test specimen thickness and under an applied load of 4100 kPa (600 psi).

9.8.1. Test five replicates and report the average and standard deviation.

9.9. *BFF Stress-Crack Test* – This test is performed with the use of common stress-crack frames in deionized water at 80°C (176°F), under an applied stress of 650 psi.

9.9.1. A bath that has never contained Igepal or other surfactants is preferred. Residual surfactant can dramatically accelerate the test and is very difficult to remove.

9.9.2. Prepare a compression molded plaque that is a minimum of 15.24 cm x 15.24 cm (6 in. x 6 in.) and has a center section, that is 84-mm wide and 1.14-mm thick (3.3 in. x 0.045 in.), end sections that are 19-mm wide and 2.29-mm thick (0.75 in. x 0.09 in.) and two sections 16.5-mm wide that transitions from 1.14-mm to 2.29-mm thick.

9.9.3. The test specimen is a ASTM D638 Type I Dumbbell with two modifications. First, drill a 7/32" hole into each head portion so that the specimens can be mounted with a screw in a conventional stress-crack testing device. However, because of the holes, the tabs on each side of the hole are narrower than the reduced section of the dumbbell. Therefore, the highest stressed areas are the tabs, so all the specimens would naturally fail at the tabs. To circumvent this limitation, a 6" square open faced mold was modified to produce plaques that were about 45 mil in the center and over 90 mil on the edges. This way, the tabs are under less stress than the reduced section so nearly all of the failures occur in the reduced section. A drawing of the fathead test specimen is shown in Figure 4. Notice that only the mold cavity needs modification, the top plate of the mold remains flat.

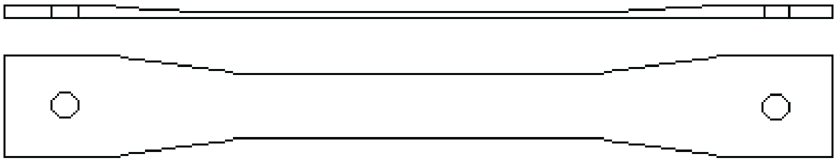

Figure 4. Fathead specimen.

9.9.4. The test device is manufactured by BT Technology and is shown in Figure 5. The weight tubes were modified to allow for the higher loads used in this test.

9.9.5. Five replicates are tested and the average and standard deviation reported.

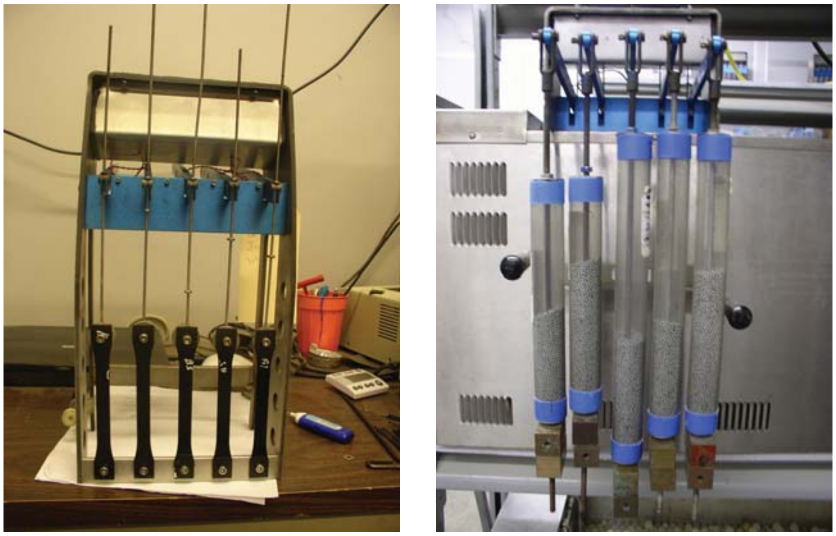

Figure 5. BFF test device.

9.10. *Oxidative Induction Time (OIT)* – Test by ASTM D3895 at 200°C in oxygen in duplicate and report the average time.

9.11. *Pipe Stiffness* – Select a minimum of two pipe specimens and test for pipe stiffness (PS), as described in ASTM D2412 except for the following: (1) the test specimens shall be a minimum of one diameter length; (2) locate the first specimen in the loading machine with an imaginary line connecting the two seams formed by the corrugation mold (end view) parallel to the loading plates, when applicable. The specimen must lie flat on the plate within 3 mm and may be straightened by hand bending at room temperature to accomplish this. Use the first location as a reference point for rotation and testing of the other specimen. Rotate the pipe 90 degrees from the first orientation and test. Test each specimen in one position only; (3) the deflection indicator shall be readable and accurate to ±0.02 mm; (4) the residual curvature found in tubing frequently results in an erratic initial load/deflection curve. When this occurs, the beginning point for deflection measurement shall be at a load of 20 ± 5 N. The point shall be considered as the origin of the load deflection curve. (5) The crosshead speed shall be the faster of 12.7 mm per minute (0.5 in. per minute) or 2% of the nominal inside diameter per minute.

Note 2—The parallel plates must exceed the length of the test specimen as specified above.

Note 3—Additional pipe specimens may be tested at other orientations for pipe stiffness and flattening if desired.

9.12. *Pipe Flattening* Flatten the two-pipe specimens from Section 9.11 until the vertical inside diameter is reduced by 20 percent. The rate of loading shall be the same as in Section 9.11. Examine the specimen with the unaided eye for cracking, splitting, or delamination. The specimen shall fail if wall buckling, cracking, splitting, or delamination is observed with the unaided eye or if there is a decrease or downward deviation in load-deflection curve at 20% or less deflection. The load-deflection curve shall be carried beyond 20% deflection so that the shape of the curve at 20% deflection can be determined.

9.13. *Brittleness* – Test pipe specimens in accordance with ASTM D2444 except six specimens shall be tested, or six impacts shall be made on one specimen. In the latter case, successive impacts shall be separated by 120 ± 10 degrees for impacts made on one circle, or at least 300 mm longitudinally for impacts made on one element. Impact points shall be at least 150 mm from the end of the specimen. Tup B shall be used, with a mass of 4.5 kg. The height of drop shall be 3.0 m. Use a flat plate specimen holder. Condition the specimens for 24 hours at a temperature of –4 ± 2°C, and conduct all tests within 60 seconds of removal from this atmosphere. The center of the falling tup shall strike on a corrugation crown for all impacts.

9.14. *Joints and Fittings*:

Joint Integrity – Pipes that have a welded bell shall be tested to verify the strength of the weld as follows: Assemble the joint in accordance with the manufacturer's recommendations. Use pipe samples at least 300 mm in length. Assemble a specimen at least 600 mm in length with the connection at the center. Load the connected pipe and joint between parallel plates at the rate of 12.5 mm per minute until the vertical inside diameter is reduced by at least 20 percent of the nominal diameter of the pipe. Inspect for damage while at the specified deflection and after load removal.

9.14.1. *Alignment* – Assure that the assembly or joint is correct and complete. If the pipe is bent, it should be straightened prior to performing this test. Lay the assembly or joint on a flat surface and verify that it will accommodate straight-line flow.

9.15. *Dimensions*:

9.15.1. *Inside Diameter* – Measure the inside diameter of the pipe with a tapered plug in accordance with ASTM D 2122. As an alternative, measure the inside diameter with a suitable device accurate to ±3.0 mm by taking two inside diameter measurements, the first at the seam and the second 90 degrees from the seam, and averaging the two measurements. The average inside diameter shall meet the requirements of Section 7.2.3.

9.15.2. *Length* – Measure pipe with any suitable device accurate to ±6.0 mm . Make all measurements on the pipe while it is stress free and at rest on a flat surface in a straight line. These measurements may be taken at ambient temperature.

9.15.3. *Perforations* – Measure dimensions of perforations on a straight specimen with no external forces applied. Make linear measurements with instruments accurate to 0.2 mm.

9.15.4. *Wall Thickness* Measure the wall thickness in accordance with ASTM D2122.

9.16. *Delamination* – Examine Type S pipe for evidence of delamination as defined and described in Section 3.5 by cutting the pipe at the corrugation crest as shown in Figure 6 and attempting to insert a feeler gauge between the inner and outer corrugated wall at the corrugation valley as shown in Figure 6. The feeler gauge should not pass through the corrugation valley into a void at any location along the circumference of the pipe.

Examine Type D pipe for evidence of delamination as defined and described in Section 3.5 by cutting a section through the pipe as shown in Figure 6 and attempting to insert a feeler gauge between the internal supports and the inner and outer walls as shown in Figure 7. The feeler gauge should not pass between the internal support and the inner or outer wall at any point along the circumference of the pipe.

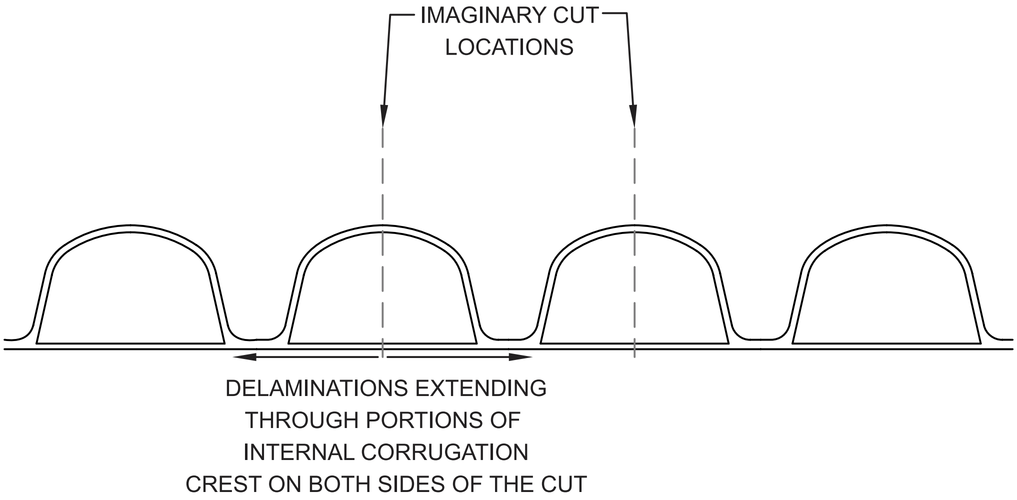

Figure 6. Location of pipe cut.

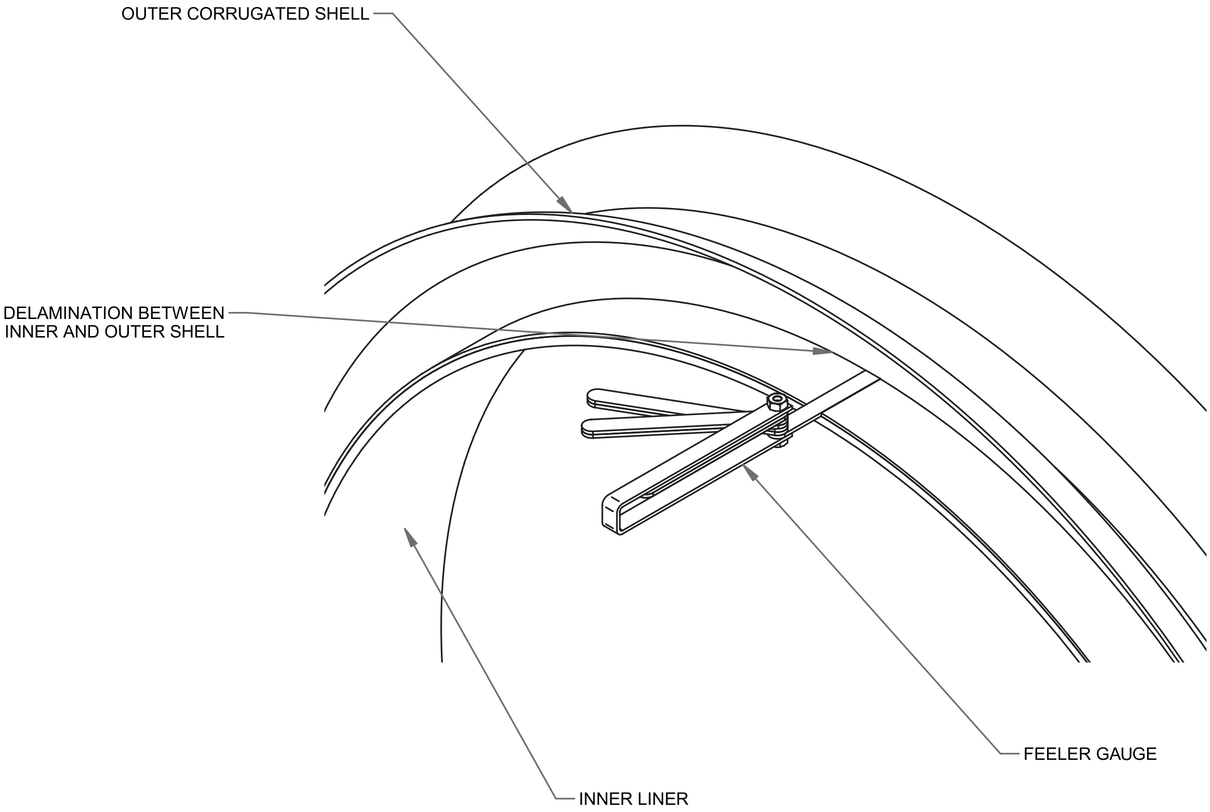

Figure 7. Feeler gauge insertion.

10. INSPECTION AND RETEST

10.1. *Inspection* – Inspection of the material shall be made as agreed upon by the purchaser and the seller as part of the purchase contract.

10.2. *Retest and Rejection* – If any failure to conform to these specifications occurs, the pipe or fittings may be retested to establish conformity in accordance with agreement between the purchaser and seller. Individual results, not averages, constitute failure.

11. MARKING

11.1. All pipe shall be clearly marked at intervals of no more than 3.5 m as follows:

11.1.1. Manufacturer’s name or trademark;

11.1.2. Nominal size;

11.1.3. This specification designation, M 294Recycled;

11.1.4. The percentage recycled content;

11.1.5. The plant designation code; and

11.1.6. The date of manufacture or an appropriate code. If a date code is used, a durable manufacturer sticker that identifies the actual date of manufacture shall be adhered to the inside of each length of pipe.

Note 5—A durable sticker is one that is substantial enough to remain in place and be legible through installation of the pipe.

11.2. Fittings shall be marked with the designation number of this specification, M 294, and with the manufacturer's identification symbol.

12. QUALITY ASSURANCE

12.1. A manufacturer's certificate that the product was manufactured, tested, and supplied in accordance with this specification, together with a report of the test results, and the date each test was completed, shall be furnished upon request. Each certification furnished shall be signed by a person authorized by the manufacturer.

Manufacturer Records-Manufacturers shall keep records of the following: (1) resin manufacturers data sheets and certification that the base resin meets minimum cell class requirements of the product specification; (2) manufacturer's data sheets and quantities for all additives blended with the resin by the pipe manufacturer; (3) test results to demonstrate that, if resins of two different cell classifications are blended, the resulting mixture meets the requirements of the specified cell classification; (4) correlation of resin shipment source with pipe markings.

ANNEX

(Mandatory Information)

A1. GUIDELINES FOR PIPE MADE FROM BLENDED RESINS

A1.1.1. If pipe is made from a blend of component resins or from off-prime resin or blends, the manufacturer of each component resin should supply a certificate of analysis for each lot that includes the lot specific melt index and density. The pipe manufacturer should have a resin testing program in place that includes the testing of each resin blend lot for density and melt index and have the means of conducting (in-house or contract lab) the remaining cell class and NCLS testing as specified in this standard. The manufacturer shall test each resin or resin blend lot for full cell class, NCLS and IT, unless a regular quality assurance program is in place to correlate cell class results to density and melt index. In any case, full cell class and NCLS testing should be performed on all approved/certified resin blends at least quarterly. Additionally, the pipe manufacturer shall have the resin blend independently tested and pre-qualified based on a "recipe" with component percentage tolerances of no more than plus or minus 1.5% substantiated by independent testing.

A1.1.2. When blends of resins are used for pipe manufacture, the final blended resin must meet the requirements of the standard. It is not necessary for the individual components of the blend to meet the cell class or NCLS requirements of this standard, provided that the final blend meets all the requirements.

A1.1.3. Sampling and testing of the final blended resin shall be performed as follows:

A1.1.3.1. The sample shall be prepared either by direct sampling from the feed hopper of the extruder (after the material has already been weigh-blended by the blenders or other means), or by manually weigh blending the individual components to the specified finished blend ratios. The manufacturer shall have current calibration records for the automated or manual weigh-blending equipment.

A1.1.3.2. The blended sample shall not contain carbon black concentrate. If carbon black concentrate is present in the sampled material, the carbon black concentrate shall be removed prior to further sample preparation.

A1.1.3.3. The dry-blended sample shall be fully homogenized by melt blending via a twin screw lab extruder prior to testing for physical properties.

A1.1.3.4. The melt-blended sample shall be tested for melt index, density, and NCLS in accordance with the amount of hours per the requirements of the standard.

APPENDIX

(Nonmandatory Information)

X1. QUALITY CONTROL/QUALITY ASSURANCE PROGRAM

X1.1. *Scope*:

X1.1.1. As required in Sections 10 and 12, the acceptance of these products relies on the adequate inspection and certification agreed to between the buyer and the seller/producer. This appendix should serve as a guide for both the manufacturer and the user. It places the responsibility on the producer to control the quality of the material they produce and to provide the quality control information needed for acceptance by the buyer/user. The producer is required to perform quality control sampling, testing and record-keeping on materials they ship. It also sets forth quality assurance sampling, testing and record keeping that should be performed by the buyer/user to confirm the performance of the producer’s control plan.

X1.2. *Program Requirements*:

X1.2.1. The producing company must have a quality control plan approved by the specifying agency.

X1.2.2. The producing plant must have an approved quality control plan.

X1.2.3. The plant must have an approved laboratory, either within the company or an independent laboratory.

X1.2.4. The producing plant(s) must have a designated quality control technician(s).

X1.3. *Quality Control Plan*:

X1.3.1. The producer must supply to the specifying agency a written quality control plan that shows how the producer will control the equipment, materials, and production methods to insure that the specified products are supplied. The following information must be included in the plan:

X1.3.1.1. Titles of the personnel responsible for production quality at the plant(s);

X1.3.1.2. The physical location of the plant(s);

X1.3.1.3. The methods of identification for each lot of material during manufacturer, testing, storage, and shipment. The method of identification shall allow the specifying agency to trace the finished product to the material provider;

X1.3.1.4. The method of sampling and testing of raw materials and of finished product, including lot sizes and types of tests performed; and

X1.3.1.5. A plan for dealing with nonconforming product, including how the producer plans to initiate immediate investigation and how corrective action will be implemented to remedy the cause of the problem.

X1.4. *Approved Laboratory*:

X1.4.1. All tests must be conducted at laboratories approved by the specifier. Each manufacturer may establish and maintain its own laboratory for performance of quality control testing or may utilize an approved independent laboratory. Records of instrument calibration and maintenance and sample collection and analysis must be maintained at the laboratory.

X1.5. *Quality Control Technician*:

X1.5.1. All samples must be taken and tested by the quality control technician(s) designated by the producer. The designated quality control technician(s) will be responsible for overall Quality Control at the producing plant.

X1.6. *Annual Update*:

X1.6.1. An annual update may be required. The annual update may be submitted by the manufacturer to the specifying agency by December 31st of each calendar year.

X1.7. *Plant Approval*:

X1.7.1. The plant approval process requires the manufacturer to submit an annual update to the specifying agency. The update must identify the specific product manufactured at the plant.

X1.7.2. The specifying agency will review the manufacturer's written quality control plan and a plant inspection may be scheduled. This inspection will verify that the quality control plan has been implemented and is being followed and that at least one designated quality control technician is on-site and will be present when material is being produced under this program. The laboratory will be inspected and approved if it meets the requirements.

X1.8. *Sampling and Testing*:

X1.8.1. The quality assurance plan approved for each manufacturer, and/or manufacturer's location, shall detail the methods and frequency of sampling and testing for all raw materials and products purchased or manufactured at that location. All testing shall be in accordance with current specifications and procedures referenced in M294.

X1.8.2. Samples of materials and pipe may be taken by the specifying agency.

X1.8.3. The specifying agency may require an annual third-party independent assurance test.

X1.9. *Sample Identification and Record Keeping*:

X1.9.1. Manufacturer's Quality Control samples are to be uniquely identified by the producing plant.

X1.9.2. Quality Control and Quality assurance data is to be retained by the manufacturer for two years and made available to the specifying agency upon request.

X1.9.3. Quality Control test reports shall include the lot identification.

X1.9.4. Unless requested at the time of ordering, test reports do not have to be filed for specific projects.

X1.9.5. Reports shall indicate the action taken to resolve nonconforming product.

Abbreviations and acronyms used without definitions in TRB publications:

AAAE	American Association of Airport Executives
AASHO	American Association of State Highway Officials
AASHTO	American Association of State Highway and Transportation Officials
ACI–NA	Airports Council International–North America
ACRP	Airport Cooperative Research Program
ADA	Americans with Disabilities Act
APTA	American Public Transportation Association
ASCE	American Society of Civil Engineers
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
ATA	Air Transport Association
ATA	American Trucking Associations
CTAA	Community Transportation Association of America
CTBSSP	Commercial Truck and Bus Safety Synthesis Program
DHS	Department of Homeland Security
DOE	Department of Energy
EPA	Environmental Protection Agency
FAA	Federal Aviation Administration
FHWA	Federal Highway Administration
FMCSA	Federal Motor Carrier Safety Administration
FRA	Federal Railroad Administration
FTA	Federal Transit Administration
HMCRP	Hazardous Materials Cooperative Research Program
IEEE	Institute of Electrical and Electronics Engineers
ISTEA	Intermodal Surface Transportation Efficiency Act of 1991
ITE	Institute of Transportation Engineers
NASA	National Aeronautics and Space Administration
NASAO	National Association of State Aviation Officials
NCFRP	National Cooperative Freight Research Program
NCHRP	National Cooperative Highway Research Program
NHTSA	National Highway Traffic Safety Administration
NTSB	National Transportation Safety Board
PHMSA	Pipeline and Hazardous Materials Safety Administration
RITA	Research and Innovative Technology Administration
SAE	Society of Automotive Engineers
SAFETEA-LU	Safe, Accountable, Flexible, Efficient Transportation Equity Act: A Legacy for Users (2005)
TCRP	Transit Cooperative Research Program
TEA-21	Transportation Equity Act for the 21st Century (1998)
TRB	Transportation Research Board
TSA	Transportation Security Administration
U.S.DOT	United States Department of Transportation