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Development of Laboratory Test Methods to Replace the Simulated High-Temperature Grout Fluidity Test

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SI* (Modern Metric) Conversion Factors-Approximate Conversions to SI Units

SYMBOL	WHEN YOU KNOW	MULTIPLY BY	TO FIND	SYMBOL
LENGTH				
in	inches	25.4	millimeters	mm
ft	feet	0.305	meters	m
yd	yards	0.914	meters	m
mi	miles	1.61	kilometers	km
AREA				
in^2	square inches	645.2	square millimeters	mm^2
ft^2	square feet	0.093	square meters	m^2
yd^2	square yard	0.836	square meters	m^2
ac	acres	0.405	hectares	ha
mi^2	square miles	2.59	square kilometers	km^2
VOLUME				
fl oz	fluid ounces	29.57	milliliters	mL
gal	gallons	3.785	liters	L
ft^3	cubic feet	0.028	cubic meters	m^3
yd^3	cubic yards	0.765	cubic meters	m^3
NOTE: volumes greater than 1000 L shall be shown in m^3				
MASS				
oz	ounces	28.35	grams	g
lb	pounds	0.454	kilograms	kg
T	short tons (2000 lb)	0.907	Megagrams	Mg (or "t")
TEMPERATURE (exact degrees)				
^oF	Fahrenheit	5(F-32)/9 or (F-32)/1.8	Celsius	^oC
ILLUMINATION				
fc	foot-candles	10.76	lux	lx
fl	foot-Lamberts	3.426	candela/m^2	cd/m^2
FORCE and PRESSURE or STRESS				
kip	1000 pound force	4.45	kilonewtons	kN
lbf	pound force	4.45	newtons	N
lbf/in^2	pound force per square inch	6.89	kilopascals	kPa

*SI is the symbol for the International System of Units. Appropriate rounding should be made to comply with Section 4 of ASTM E380.

SI* (Modern Metric) Conversion Factors-Approximate Conversions from SI Units

SYMBOL	WHEN YOU KNOW	MULTIPLY BY	TO FIND	SYMBOL
LENGTH				
mm	millimeters	0.039	inches	in
m	meters	3.28	feet	ft
m	meters	1.09	yards	yd
km	kilometers	0.621	miles	mi
AREA				
mm^2	square millimeters	0.0016	square inches	in^2
m^2	square meters	10.764	square feet	ft^2
m^2	square meters	1.195	square yards	yd^2
ha	hectares	2.47	acres	ac
km^2	square kilometers	0.386	square miles	mi^2
VOLUME				
mL	milliliters	0.034	fluid ounces	fl oz
L	liters	0.264	gallons	gal
m^3	cubic meters	35.314	cubic feet	ft^3
m^3	cubic meters	1.307	cubic yards	yd^3
MASS				
g	grams	0.035	ounces	oz
kg	kilograms	2.202	pounds	lb
Mg (or "t")	megagrams (or "metric ton")	1.103	short tons (2000 lb)	T
TEMPERATURE (exact degrees)				
^oC	Celsius	1.8C+32	Fahrenheit	^oF
ILLUMINATION				
lx	lux	0.0929	foot-candles	fc
cd/m^2	$candela/m^2$	0.2919	foot-Lamberts	fl
FORCE and PRESSURE or STRESS				
kN	kilonewtons	0.225	1000 pound force	kip
N	newtons	0.225	pound force	lbf
kPa	kilopascals	0.145	pound force per square inch	lbf/in^2

*SI is the symbol for the International System of Units. Appropriate rounding should be made to comply with Section 4 of ASTM E380.

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16. Abstract

This report contains a summary of the research performed to develop a replacement for the high-temperature grout fluidity (HTGF) test. The HTGF test was employed in the past by FDOT to qualify post-tensioning (PT) grouts for use in post-tensioned bridge construction. The HTGF test, however, is expensive and cumbersome to conduct in a typical construction materials testing laboratory.

The objective of this research project was to develop a replacement for the HTGF test using a dynamic shear rheometer (DSR). Initially, exploratory studies were conducted using a number of DSR test methods and geometries to develop or adopt a method that provides more consistent and rational rheological results than the currently used flow cone test method. These studies led to the adoption of the viscosity test with a cup and ribbon geometry. The shear rate used in the viscosity test (50 s^{-1}) was based on data from the literature and from the estimated shear rate at the nozzle of a flow cone.

In addition to the DSR test development, HTGF tests were conducted twice on four different commercially available PT grouts. PT grout temperatures increased an average of 7°F (4°C) over the course of the circulation during HTGF testing. Line pressures measured at the pump during circulation ranged from 100 to 350 psi (4 kPa to 17 kPa).

Viscosity results measured during the HTGF test were correlated to performance of the PT grouts. The viscosity results compared well with flow cone results with an R^2 value of 0.85. From these data, performance classifications were developed based on the results of the testing.

Finally, DSR testing was conducted at University of Minnesota-Duluth (UMD) and National Institute of Standards and Technology (NIST) to provide a comparison of the viscosity results from these laboratories to those produced by the University of Florida. Trends in viscosity test results compared well with results from both UMD and NIST. Absolute magnitudes, however, varied somewhat. This is thought to be due to the sensitive nature of the mixing and conditioning process. Additional performance classifications were developed based on the findings of these studies.

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Executive Summary

This report contains a summary of the research performed to develop a replacement for the high-temperature grout fluidity (HTGF) test. The HTGF test was employed in the past by FDOT to qualify post-tensioning (PT) grouts for use in post-tensioned bridge construction. The test is conducted by pumping a batch of grout through 400 feet (122 m) of 1-in. (25 mm) diameter hose in a 90°F (32.2°C) environment continuously for 60 min and periodically taking modified flow cone measurements (ASTM C939). The efflux time on the modified flow cone test must be less than 30 s after 1 h to pass the test. In the past, the HTGF test was a requirement for FDOT's standard specifications. The HTGF test, however, is expensive and cumbersome to conduct in a typical construction materials testing laboratory.

The objective of this research project was to develop a replacement for the HTGF test using a dynamic shear rheometer (DSR). The DSR is a relatively common countertop laboratory apparatus that is used to assess the rheological properties of fluid materials. Research using a DSR has been performed on plain grouts in the past but to a limited extent.

Initially, exploratory studies were conducted using a number of DSR test methods and geometries. The primary aim of these studies was to develop or adapt a method that provided a more reliable and consistent rheological results than the modified flow cone test method (ASTM C939). These studies led to the adoption of the viscosity test with a cup and ribbon geometry. The shear rate used in the viscosity test (50 s^{-1}) was based on data from the literature and from the estimated shear rate at the nozzle of a flow cone.

In addition to the DSR test development, HTGF tests were conducted twice on four different commercially available PT grouts. While conducting the HTGF test, the following data were recorded: flow cone efflux time, viscosity, ambient temperature, grout temperature, pumping pressure, grout flow rate, wet density, and unit weight. PT grout temperatures increased an average of 7°F (4°C) over the course of the circulation during HTGF testing. Line pressures measured at the pump during circulation ranged from 100 psi to 350 psi (4 kPa to 17 kPa).

The acceptable performance objective of PT grout was based on a grout that would maintain fluidity at 90°F (32.2°C) for at least 1 h. Viscosity measurements using the DSR correlated well with the flow cone efflux time with an R^2 value of 0.85. In addition, an

alternative grout conditioning technique was developed that utilized the DSR rather than the full-scale grout plant. From these data, performance classifications were developed based on the results of the DSR testing.

Finally, DSR testing was conducted at University of Minnesota-Duluth (UMD) and the National Institute of Standards and Technology (NIST) to compare the DSR viscosity results from these laboratories to those produced by the University of Florida. Trends in viscosity test results compared well with results from both UMD and NIST. Absolute magnitudes, however, varied somewhat. This is thought to be due to the sensitive nature of the mixing and conditioning process. Additional performance classifications were developed based on the findings of these studies.

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1 Introduction

Maintaining the fluidity of post-tensioning grout during high-temperature grouting conditions is an important aspect of post-tensioning bridge construction in Florida. The high-temperature grout fluidity (HTGF) test was developed in Florida to ensure that prepackaged grouts used to fill post-tensioned tendons maintain their fluidity during grouting operations in the hot summer months. The method, however, is not a standard test and it requires the facility, grout, water, duct, pump, mixer and all other equipment to be conditioned to a temperature of 90°F (32.2°C). Grout is then mixed and pumped through 400 ft (122 m) of 1-in. (25.4 mm) diameter hose and recirculated for an hour. Grout fluidity is checked periodically and must never have a modified flow cone efflux time (ASTM C939) greater than 30 s. This test procedure requires the use of unusual equipment and procedures that are not typically available in construction materials testing laboratories. Nevertheless, the HTGF test was previously a requirement for Florida Department of Transportation (FDOT) for a material to be on the pre-qualified products list (QPL) (FDOT 2007). Furthermore, the FDOT does not have the capabilities on a production level to conduct the HTGF test and no accredited laboratory is available to conduct the test. For these reasons, the test was removed from FDOT's bridge construction specifications (FDOT 2013) and limits were placed on the temperature of the grout during injection.

The research objectives were to create a test method for post-tensioning grout that (a) is calibrated to the simulated high-temperature fluidity test and (b) can be conducted by a construction materials testing laboratory. These objectives were achieved by the development of a test method using a dynamic shear rheometer (DSR). The DSR is a counter top laboratory apparatus that is used to assess the rheological properties of fluids.

This report is organized as follows. Chapter 2 defines rheology terminology with which the reader may be unfamiliar. Chapter 3 provides an overview of the approach used in this research. Chapter 4 provides a brief overview of post-tensioning grout and its use in post-tensioned bridges in Florida. Chapter 5 covers fundamentals of rheology including several DSR geometries that were investigated for their suitability to test PT grout. Chapter 6 presents past research related to rheology of plain and prepackaged grouts. Chapter 7 presents the materials used for testing, and Chapter 8 covers the equipment used to mix and agitate the grout. Chapter

9 covers the DSR equipment and geometry used in the testing. Chapter 10 reviews existing DSR test methods and Chapter 11 covers the test method developed to replace the HTGF test. Chapter 12 relates the procedures used to conduct the HTGF test with the results of such located in Chapter 13. Chapter 14 presents the development of performance classifications based on the rheological results and pumpability. Chapter 15 covers conditioning techniques alternative to those of the HTGF test. Chapters 16 and 17 cover DSR testing conducted at other laboratories. Chapter 18 is summary and conclusions and Chapter 19 summarizes the test methods and performance classifications.

2 Definitions

This research involves discussion of rheological properties that may be unfamiliar to the reader. The definitions in this section are meant to aid in understanding the rheological discussions in this report and are taken from Hackley and Ferraris (2001), unless otherwise noted.

Anti-thixotropy: A reversible time-dependent increase in viscosity at a particular shear rate. Shearing causes a gradual growth in structure over time (Figure 1)

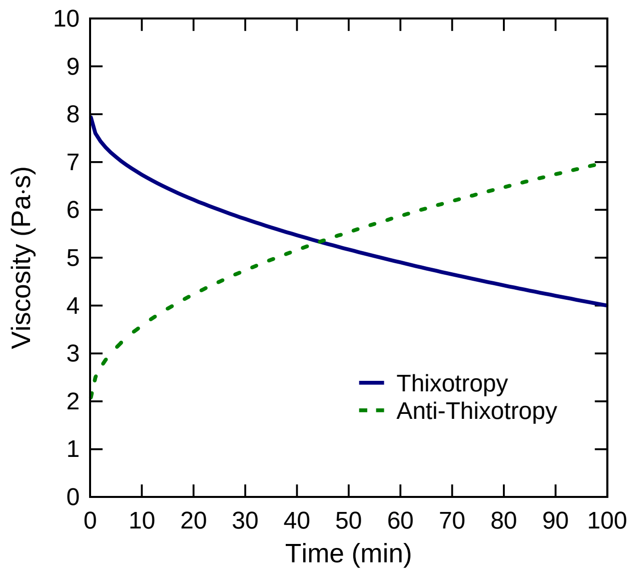


Figure 1—Thixotropic effects observed in material subjected to constant shear rate over a period of time

Apparent viscosity: The value of viscosity evaluated at some nominal average value of shear rate, such as the shear rate in the nozzle of the flow cone or in a DSR using a tool that does not shear with a uniform shear rate.

Bingham equation or model: The Bingham relation is a two parameter model for describing viscoplastic fluids exhibiting a yield response. The ideal Bingham material is an elastic solid at low shear stress values and a Newtonian fluid above a critical value called the Bingham yield stress, σ_0. The plastic viscosity, η_{pl}, is the slope of the linear portion of the curve shear stress vs. shear rate. It is usually described using the following equation:

$$\sigma = \sigma_0 + \eta_{pl} \dot{\gamma}$$

Colloid: State of subdivision implying that the particles have at least in one direction a dimension roughly between 1 nm and 1μm.

Colloidal grout: Grout in which a substantial proportion of the solid particles have the size range of a colloid. (ACI 2010)

Colloidal mixer: A mixer designed to produce colloidal grout (ACI 2010). Grout mixer used to disperse cementitious material down to its finest particle size to achieve complete particle wetness (ChemGrout).

Flow curve: A graphical representation of the behavior of flowing materials in which shear stress is related to shear rate (Figure 2).

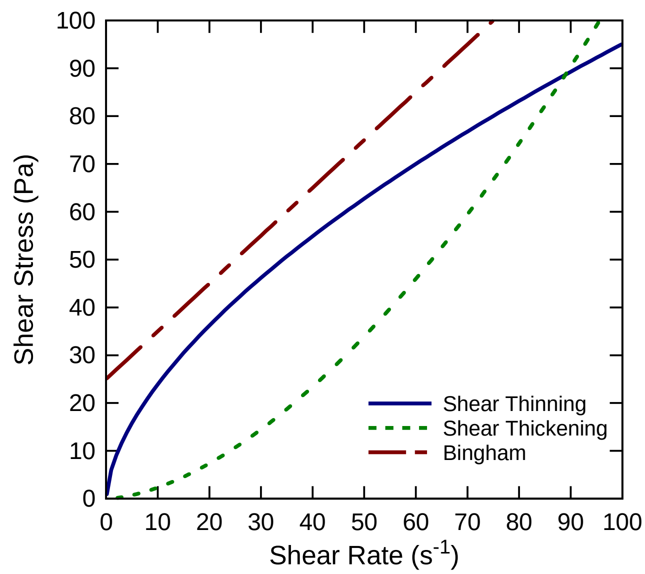


Figure 2—Flow curves for several non-Newtonian fluid models

Flow curve test: A DSR test in which a linearly-increasing shear rate is imposed on a material. Shear stress is measured and plotted against shear rate to generate a flow curve.

Grout: A mixture of cementitious material and water, with or without aggregate, proportioned to produce a pourable consistency without segregation of the constituents; also a mixture of other composition but of similar consistency. (ACI 2010)

Herschel-Bulkley equation: A three parameter model used to describe viscoplastic materials exhibiting a yield response, with a power-law relationship between shear stress and shear rate above the yield stress, σ_0. The Herschel-Buckley relation reduces to the equation to the Bingham plastic equation when n=1.

$$\sigma = \sigma_0 + k\dot{\gamma}^n$$

Loss modulus: The out-of-phase (viscous) component of oscillatory flow (Hackley and Ferraris 2001). The liquid-like component of a material measured using an oscillatory test (Barnes 2000).

Newtonian fluid: Flow model of fluids in which a linear relationship exists between shear stress and shear rate, where the coefficient of viscosity is the constant of proportionality.

Non-Newtonian fluid: : Any laminar flow that it is not characterized by a linear relationship between shear stress and shear rate.

Plain grout: A grout composed of portland cement and water only.

Plastic viscosity: For the Bingham model, the excess of the shear stress over yield stress divided by shear rate and equal of the differential viscosity. Note of pure Bingham material, it is the slope of the shear stress vs. shear rate.

Post-tensioning: Method of prestressing in which prestressing steel is tensioned after concrete has hardened. (ACI 2010)

Post-tensioning, bonded: Post-tensioned construction in which the annular spaces around the tendons are grouted after stressing, thereby bonding the tendon to the concrete section. (ACI 2010)

Post-tensioning grout (PT grout): A colloidal grout composed of portland cement, water, and other additives intended to improve the fluid and the hardened properties.

Shear rate: The rate of change of shear strain with time. For liquids, the shear rate, rather than strain, is generally used in describing flow.

Shear stress: The component of stress that causes successive parallel layers of a material body to move, in their own planes (i.e., the plane of shear), relative to each other.

Shear thickening: An increase in viscosity with increasing shear rate during steady shear flow. See Figure 2.

Shear thinning: A decrease in viscosity with increasing shear rate during steady shear flow. See Figure 2.

Storage modulus: The in-phase (elastic) component of oscillatory flow (Hackley and Ferraris 2001). The solid-like component of a material measured using an oscillatory test. (Barnes 2000).

Stress growth: When an instantaneous and constant strain (or shear rate) is applied to a material while stress is measured over time, an increasing stress vs. time or modulus vs. time function is termed stress growth.

Structure: In rheology, structure is a term that refers to the formation of stable physical bonds between particles (or chemical bonds between macromolecules) in a fluid. These bonds result in aggregate, floc, or network structure, which impacts the rheological behavior of the fluid and provides elastic and plastic properties. The term may be extended to include structural effects caused by electroviscous interactions, physical bonds between polymers (e.g., associative thickeners), shear-induced alignment of anisotropic particles, and close-packing (radial distribution) correlations in concentrated suspensions. The term "structure" is commonly invoked even when little is known about the cause of observed changes in rheological properties.

Thixotropy: A reversible time-dependent decrease in viscosity at a particular shear rate. Shearing causes a gradual breakdown in structure over time. See Figure 1.

Viscoplastic: A hybrid property in which a material behaves like a solid below some critical stress value, the yield stress, but flows like a viscous liquid when this stress is exceeded, often associated with highly aggregated suspensions and polymeric gels. See Figure 2.

Viscosity: Viscosity is the ratio of shear stress to shear rate under simple steady shear.

Yield Stress: A critical shear stress value below which an ideal plastic or viscoplastic material behaves like a solid (i.e., will not flow). Once the yield stress is exceeded, a viscoplastic material flows like a liquid. See Bingham curve in Figure 2.

3 Research Approach

The primary purpose of the HTGF test was to ensure that qualified grouts would not thicken excessively during pumping in hot-weather conditions. An excessive early thickening may result in a viscosity that renders the material unpumpable. This research project was aimed at developing a test method that provided a more direct measure of the rheological properties of grout at high temperatures and did so with a test method more amenable to a typical construction materials testing laboratory's equipment. To develop an adequate replacement for the HTGF test, two aspects of the test were considered. The first was the measure of the fluidity of the grout, which the HTGF test addressed using the flow cone test. The second aspect was the conditioning of the grout between sampling. The HTGF test imposed a dynamic mechanical shearing action on the grout between sampling; this conditioning needed to be replicated to produce the same rheological response in the grout.

After settling on a DSR geometry for testing, existing DSR test methods were reviewed to determine if a suitable test was available. Simultaneous DSR and flow cone tests were conducted as a part of the test method development. Due to the complex rheological properties of the commercially available PT grouts, a viscosity test was developed so that all of the grouts could be evaluated. The viscosity test was conducted in conjunction with the HTGF test on four commercial PT grouts. Once the effects of the HTGF test's conditioning process on the rheological properties of the grout were known, two techniques were investigated to simulate the conditioning. Ultimately, several test methods were developed that could provide an alternative to the HTGF test. The grout conditioning can be performed using either a laboratory-sized high-shear mixer or the DSR. The fluidity assessment can be performed using either a flow cone or the DSR. The flow chart shown in Figure 3 provides a summary of the research approach.

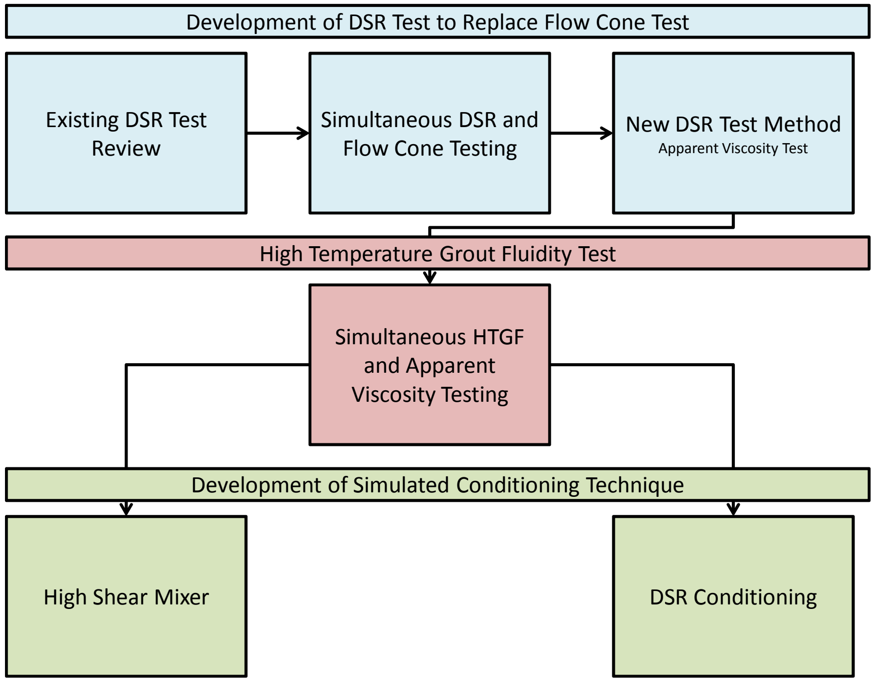


Figure 3—Flow chart depicting research approach

4 Post-Tensioning Grouts

Post-Tensioning (PT) grouts are composed primarily of Portland cement and water. Prepackaged proprietary grouts may contain such supplementary cementitious materials (SCM) as silica fume, fly ash, or others along with chemical admixtures to improve the fluid and hardened properties of the finished grout. PT grouts are used to fill ducts in precast concrete bridge sections.

Figure 4 shows a precast concrete bridge section with PT ducts and tendons visible. The tendons are used to impose compression on the concrete so that it can carry the tensile stresses without cracking. The PT grout fills the cavities between the tendons and the wall of the duct. It also fills the cavities between the individual strands. This creates a bond between the PT tendon and the precast concrete section in addition to providing chemical and physical corrosion protection for the prestressing steel.

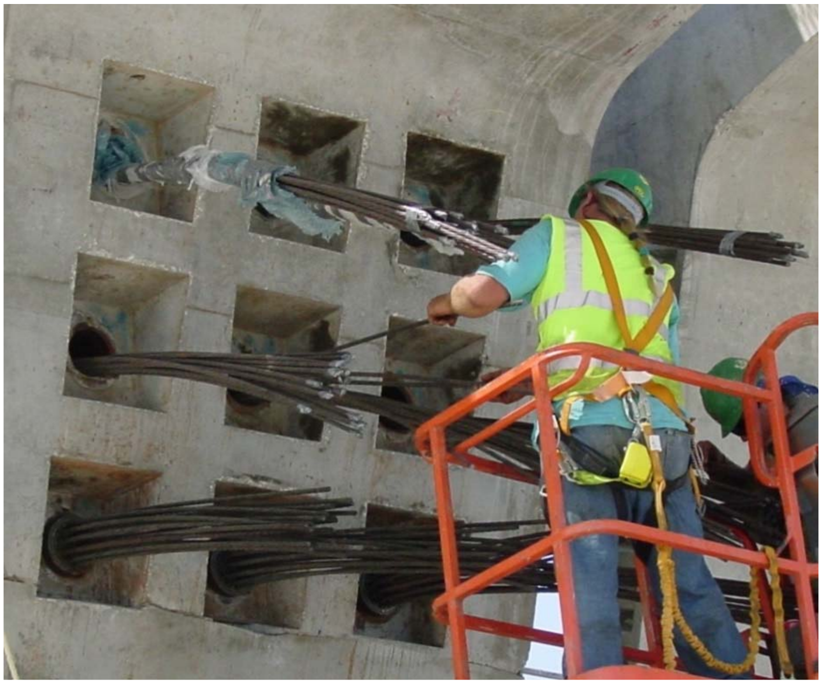


Figure 4—Precast concrete bridge segment with PT ducts and tendons shown

When the PT grouts are pumped into the ducts they must remain fluid enough to flow past the tendons, but they also must not segregate or bleed before they set. Highly viscous grout can cause excessive pumping pressures, which may in turn damage the pumping equipment and prevent the successful completion of the grouting. Consequently, the rheological behavior of the grout is very important. The rheological properties are measured in the field using a modified flow cone test (ASTM C939 as modified by PTI (2012)) during mixing to ensure that the grout has adequate fluidity (Figure 5). The test method is conducted by filling the funnel-like container with the grout to the top of the cone. The time required for 1 liter of grout to be discharged is the efflux time. The single laboratory standard deviation for the unmodified test procedure is reported in ASTM C939 as 0.88 s.

Figure 5—Flow cone used to determine rheological properties of grout in the field. See ASTM C939 for dimensions

5 Rheology

Rheology is the study of the flow and deformation of materials and is used to study materials in a wide range of industries from pharmaceuticals to construction materials. (Barnes 2000).

Newtonian fluids, such as oil, exhibit the same viscosity at any shear rate. In more complex fluids, the viscosity will vary with the applied shear rate and are known as non-Newtonian fluids. (Barnes 2000). Examples of non-Newtonian fluid include cement paste and concrete.

5.1 Rheology Equipment

A rheometer is a device used to measure the rheological properties over a varied and extended range of conditions (Hackley and Ferraris 2001). Most rheometers are rotational rheometers. In these instruments, the test fluid is continuously sheared between two surfaces one or both of which are rotating. Commercially available rotational rheometers are designed to shear the material using various geometries, such as parallel plates, coaxial, or vane.

Three geometries were considered for use in this study: parallel plate, vane and helical ribbon. The parallel plate geometry is shown in Figure 6. This configuration is typically used with a DSR for asphalt.

Before testing begins, the top plate is raised above the bottom plate, which is stationary. A sample of material is loaded onto the bottom plate, and the top plate is lowered to within less than 1.5 mm (gap on Figure 6) of the bottom plate. The material being tested fully contacts both plates. The top plate then rotates at a given angular velocity, and a sensor in the rheometer measures the resulting torque. The shear rate is not constant in the material as it varies from zero at the center of the plates, to its maximum value at the outer diameter. As a matter of convention for parallel plate rheometers, the reported values are the torque and the shear rate calculated at the outer diameter. Equations are available to integrate the response of the material over the various shear rates [Barnes et al. 1989]. The viscosity is calculated from the shear rate and shear stress. The whole flow curve can be obtained if the shear rate is ramped between two values.

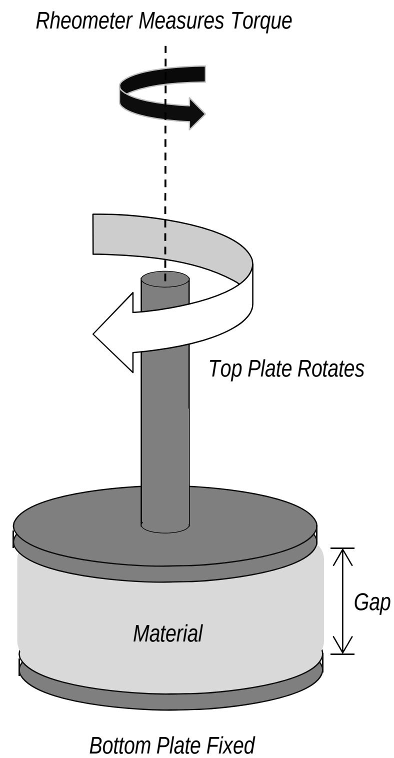


Figure 6—Parallel plate rheometer geometry. The white arrow signifies the rotation of the top plate, while the black arrow is the measurement of the torque or resistance to rotation.

The vane and the helical ribbon tools operate differently from the parallel plate tool in that they are immersed in the material being measured. The material is in a container that should have a diameter that is at least 2 mm to 3 mm larger than the tool and it should be deep enough for the tool to be fully immersed. The gap between the tool and the container is sufficiently wide to allow the sand/aggregates to flow between the tool and container without jamming. Conversely, the gap needs to be small enough to ensure sufficient shearing of the material. The specific geometry used in this research will be described in Chapter 9. These tools provide a convenient measurement method for PT grout. Analytical methods, however, are not available to calculate imposed shear rate and shear stress from measured angular velocity and torque.

The cup and vane are primarily used for yield stress testing with many different types of materials (Cullen 2003). The vane is assumed to act as a solid cylinder when it rotates inside the cup. Zhu et al. (2010) completed a numerical simulation to study the behavior of particles within a suspension as they were agitated by the vane. Their model showed that this assumption is incorrect.

Although the helical ribbon tool geometry (see 9.1.2 and Figure 19) is more complex than traditional geometries, it offers several benefits. Suspensions with large particle sizes that cannot be tested using traditional geometries due to the narrow gaps can be tested in the cup and ribbon. It also continuously mixes the material as it tests, which prevents larger particles from settling out. Ait-Kadi et al. (2002) demonstrated both of these properties by testing ketchup, which is a suspension with large particles that tend to flocculate. They were able to obtain repeatable flow curves when using the helical ribbon geometry; they were not able to do so with simple geometries, such as the vane. Cullen et al. (2003) explained that the helical ribbon can be used to determine how a suspension builds structure where other geometries cannot. They also state that the helical ribbon has been used to study flow curves and viscosity of suspensions.

One solution, which was adopted in this research, is to calibrate the geometry using a reference material SRM 2492 from NIST. Reference material is provided with a certificate showing the value of the viscosity and a calibration factor that can be used to calculate the viscosity in fundamental units from the slope of the flow curve (Section 9.2)

5.2 Flow curves and type of materials

The main goal of a rheological measurement using a rotational rheometer is to generate a flow curve with shear stress versus shear rate. This curve can then be used to calculate the viscosity or the Bingham viscosity and the yield stress. The rotational rheometer apparatus imposes a rotational velocity to the tool and measures the material response by measuring torque. To generate a flow curve, the angular velocity is increased in steps (up-curve) and the reduced in steps (down-curve). The conversion to shear stress and shear rate is not always possible if the shear is not constant between the two rotating surfaces, as is the case for the vane or helical ribbon geometry.

Some high-performance post-tensioning (PT) grouts behave as shear-thinning fluids (the viscosity decreases at increasing shear rates). The viscosity of a PT grout is designed to be very low during high shear applications, such as mixing and pumping, to reduce pumping pressures. When the applied shear is reduced, as when the PT grout is resting in the duct, the viscosity is designed to increase so that the grout stops flowing.

A common material model for non-Newtonian fluids is the Bingham model. A Bingham fluid is essentially a Newtonian fluid that exhibits a yield stress. Below its yield stress, the material behaves in an elastic manner, like a solid. Above its yield stress, the material deforms and flows, becoming plastic. Materials that exhibit this property are also called viscoplastic. The plastic viscosity of a Bingham material is constant with respect to shear rate. (Hackley and Ferraris 2001). For Bingham materials the plastic viscosity and yield stress are calculated from a linear fit of the flow curve, with the plastic viscosity being the slope and the yield stress the intercept (Figure 7).

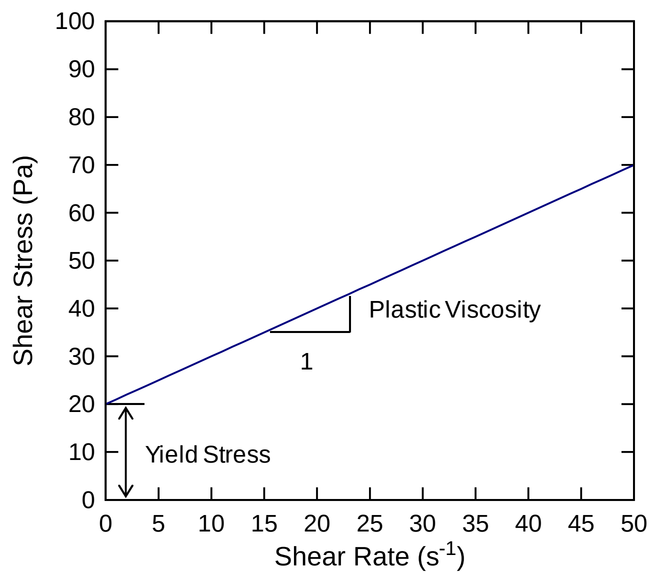


Figure 7—Flow curve for Bingham plastic material

Time is another important variable to consider in the study of non-Newtonian fluids. Thixotropy is the model used to describe a material that exhibits time dependent rheological properties. Thixotropy is illustrated in Figure 1.

A thixotropic material's viscosity will decrease over time, while a constant shear rate is applied. An anti-thixotropic material's viscosity will increase over time, while a constant shear rate is applied (Hackley and Ferraris 2001). Both of these properties are reversible. It usually occurs, however, in conjunction with shear-thinning or shear-thickening effects.

The Post-Tensioning Institute (PTI) specification (2012) defines thixotropy as "The property of a material that enables it to stiffen in a short time while at rest, but to acquire a lower viscosity when mechanically agitated, the process being reversible." This is not in agreement with the rheological definition presented in Chapter 3 that is considered the more generic definition of thixotropy. The prepackaged grouts tested in this research stiffen quickly at rest, which is due to anti-thixotropy. Furthermore, the decrease in viscosity when mechanically agitated is due to shear-thinning rather than thixotropy.

From a rheological standpoint, the mechanical behavior of different fluids is dependent on the shape of their flow curves. In addition to the flow curve, most materials display time-dependent thixotropic properties. PT grouts, which are proprietary mixtures of cement and other admixtures, can exhibit very different flow curves and thixotropic properties when compared to one another. Care must be taken to develop a test that allows the rheological properties of materials with unknown constituents to be compared.

6 Literature Review

6.1 Definition and Properties of a Suspension

PT grout is a suspension of cement particles in water. The use of SCMs or chemical additives may affect the rheological behavior of the suspension as they will prevent flocculation or regulate the viscosity of the fluid. The interstitial fluid acts as a lubricant that allows particles to move past each other. Consequently, if the volume fraction of the particles increases, then the viscosity of the suspension increases.

Another phenomenon to consider is that a PT grout suspension ages, the cement particles will agglomerate and hydration will reduce the amount of free water, thus increasing the viscosity. Coussot (2005) states that increasing the volume fraction of particles in a suspension determines whether or not the suspension behaves in a non-Newtonian manner. For example when the viscosity of the suspension is not proportional to the viscosity of the interstitial fluid, then the suspension does not have Newtonian properties. As more particles of varying sizes are added to the suspension, which is the case with PT grouts, it becomes less likely that they will disperse in an isotropic manner. This causes the suspension to become non-Newtonian.

Coussot (2005) also states that a structure parameter is often used to describe a suspension. Essentially this parameter is a number between zero and one that can be used to represent the number of internal bonds between particles. A simple model describes de-structuring as the breakage in these bonds due to flow whereas re-structuring is the tendency of the system to return to its initial configuration.

6.2 DSR Testing of Grout

Four prevalent DSR test methods for grout and cement paste were mentioned in the literature. The specific test conditions for the rotational rheometer tests were as follows:

- stress growth with constant shear rate
- low-amplitude shear strain oscillations
- Increasing-decreasing shear rate
- predetermined shear rate

The most common test in the literature, the increasing-decreasing shear rate, is conducted by testing the material over a range of increasing and decreasing shear rates. The shear stress is recorded and plotted against shear rate. The flow curve obtained is used to describe rheological properties of the material; various models can be fitted to the curve (Figure 2) (Hackley and Ferraris 2001). The most commonly used model for grouts is the Bingham model, which provides values for yield stress and plastic viscosity. Many researchers studied the connection between the workability of concrete and the rheology of cement paste. Ferraris et al. (2007) developed a methodology using serrated parallel plates that was also used for cement paste. Khayat et al. (1999) used the Bingham model to determine the yield stress of grouts with high-range water-reducing admixtures. Yahia and Khayat (2003) performed a flow curve test on grouts containing various supplementary cementitious materials (SCM). They showed that the Bingham model was not a good fit for grouts containing SCM. They fitted various empirical models to the grouts containing various SCM to determine which model was most valid. Rosquoet et al. (2003) studied the rheology of fresh cement paste at high shear rates. They ran a flow curve test with higher shear rates than Ferraris and Gaidis (1992). They found that, when considering the higher shear rates, the cement paste behaved as a shear-thinning material. Amziane and Ferraris (2007) completed a study of the rheological properties of cement paste as it hydrated using the Bingham model. Their research used serrated parallel plates, and showed that the Bingham model was not entirely appropriate for cement paste when close to initial setting time. The yield stress evolution with time since mixing of the grouts could be determined by stress growth. Plastic viscosity growth, however, could not be determined using the Bingham model because the viscosity did not change with time when the shear rate was held at a constant 50 s^{-1}. Petit et al. (2010) used the flow curve test to characterize the rheological properties of the grouts at high temperatures. They found that, as temperature increased, the yield stress of the grouts also increased.

The stress growth test is conducted by shearing the material at a very low shear rate over a period of several minutes. The shear stress is recorded and plotted vs. time. The yield stress value can be extracted from the shear stress versus time curve as the highest shear stress measured. This value depends on the shear rate used. Amziane and Ferraris (2007) ran several stress growth tests on cement pastes using parallel plate geometry. Their research showed that the stress growth test could be used to determine the yield stress of the cement pastes. Sant et al. (2008) used the stress growth test to evaluate the rheological properties of fresh grout. In addition, Dehadrai et al. (2009) made use of the stress growth test to attempt to locate the fluid-solid transition point of a cement paste. Their research showed that they could not determine this value using the stress growth test.

Low-frequency oscillatory test was also covered in the literature. In this test, the rheometer imposes small oscillatory strains on the material (low amplitude). The material does not yield at these low strains, so this test provides insight into the elastic behavior of the material. The response of the material is measured under oscillatory strain, and a modulus can be plotted to determine the increase in stiffness of the material. Schultz and Struble (1993) studied the behavior of fresh cement paste using low amplitude oscillatory tests with a cup and bob geometry. They found that this was a valid way of studying the change in the elastic properties of a cement paste over time. Struble and Lei (1995) used creep/recovery tests to study the setting of cement paste with a cup and bob geometry. They determined that this was also a valid method of assessing the rheological properties of cement paste. Chen et al. (2006) completed additional research using the oscillatory tests to measure cement-admixture interaction. Al Martini and Nehdi (2009) studied the effects of high-range water-reducing admixtures and temperature on the rheology of grout. They made use of oscillatory sweep tests in their study. They observed higher stiffness values when the temperature of the grouts was increased, and lower stiffness values when the amount of high-range water-reducing admixtures was increased.

The predetermined shear rate test measures the viscosity at a selected nominal shear rate. This is typically done with viscometer or instrument that can only rotate the tool (usually a cylinder) at a single angular velocity. This method is widely used in the polymer industry but not often applied to grouts. Khayat et al. (1999) studied the rheology of grouts with high-range water reducing admixtures using a rheometer. These grouts were meant to mimic PT grouts. Their experimental setup made use of a cup and bob geometry. They ran a series of tests in which they determined the viscosity at different shear rates. Amziane and Ferraris (2007) determined that the viscosity measured over a range of shear rates while the grout is setting is not a reliable method to characterize the grout rheological properties.

6.3 Relationship between Flow Cone and Rheology

Extensive research has been performed to determine whether or not different pieces of field equipment are suitable tools for performing quality-control checks. This research is useful to this project because it studies the relationship between field equipment, such as the flow cone, and laboratory equipment, such as the DSR.

Khayat and Yahia (1998) showed that the flow cone test is an accurate predictor of the viscosity measured using a DSR where a shear rate in the range of 340 s^{-1} to 510 s^{-1} is used for flow times in the range of 40 s to 200 s. This shear rate range corresponds to the shear rates imposed on the grout during pumping and mixing operations in the field (Khayat and Yahia (1998)).

Le Roy and Roussel (2005) showed that the flow cone efflux time is proportional to the Bingham plastic viscosity when the flow cone efflux time is higher than 15 s and the material does not exhibit a Bingham yield stress greater than 20 Pa. These conclusions were made using a concentric-cylinder DSR geometry. At a low viscosity, the relationship between flow cone efflux time and Bingham plastic viscosity becomes non-linear.

Nguyen et al. (2006, 2011) have performed extensive research relating the Marsh flow cone time to rheological properties in considering the tested fluid using the Herschel-Bulkley model. This research was conducted using a concentric cylinder DSR geometry. They developed a semi-analytical solution for predicting Herschel-Bulkley parameters given a flow cone efflux time for cement grouts (Nguyen et al. 2006). This model was shown to be accurate for grouts that diverged from the Herschel-Bulkley model at very low shear rates due to high range water reducers (HRWRA) and viscosity modifying admixtures (VMA); PT grouts fall into this category. (Nguyen et al. 2011)

6.4 Shear Rates Imposed by Field Equipment

During the HTGF test the grout is subjected to a wide range of shear rates from the mixer, pump, and flow cone. Khayat and Yahia (1998) state that shear rates of 340 s^{-1} to 510 s^{-1} occur in the field during mixing and pumping, based on results measured using a flow cone and DSR. They also state that grout experiences a shear rate of less than 5 s^{-1} during placement. Ferraris and Gaidis (1992) state that cement paste will experience a shear rate of 1 s^{-1} to 20 s^{-1} during placement.

7 Materials

Testing was conducted on both plain portland cement grouts and prepackaged PT grouts. Plain grouts containing portland cement, pulverized limestone, high-range water reducing admixture, and water were tested. This chapter describes the materials used in testing.

7.1 Prepackaged PT grout

Table 1 summarizes the prepackaged PT grout tested. The testing identification PT*x*-*y* is used to identify the specific manufacturer (*x*) and the lot number (*y*).

Table 1—Summary of PT grout tested

Testing ID	Date manufactured	Expiration Date
PT1-2	11/17/11	05/17/12
PT1-3	06/25/12	01/01/13
PT1-4*	04/29/13	11/01/13
PT2-2	01/05/12	07/05/12
PT2-3	06/26/12	12/01/12
PT2-4*	03/28/13	09/28/13
PT3-1	10/19/11	04/19/12
PT3-2	06/01/12	12/01/12
PT3-3*	04/10/13	10/07/13
PT4-1	08/25/11	02/25/12
PT4-2	02/02/12	08/02/12
PT4-3	03/19/12	09/19/12
PT4-4	03/03/12	09/03/12
PT4-5	05/24/12	11/24/12
PT4-6*	05/04/13	11/04/13
PT5-1*	06/10/13	06/10/14
PT6-1*	06/03/13	06/03/14

*Indicates materials that were tested by University of Minnesota

Each of the proprietary PT grouts were pre-packaged in a 50-lb or 55-lb (22 kg to 25 kg) bag, which contained cementitious materials along with the various admixtures that give the PT grouts their specific properties. The inscription on the bag states the recommended quantity of water needed to be mixed with one bag of grout. Usually the manufacturer provides a range of water amount rather than one value.

7.2 Plain grout

Plain grouts were designated with codes such as C40 or C45. The "C" in the code indicates that the grout was a plain grout. The "40" or "45" indicates the water-to-cement ratio (i.e., 0.40 or 0.45).

7.3 Partial Bag Sampling

Portland cement, supplementary cementitious materials, and admixtures may not be distributed homogenously throughout a pre-packaged bag of PT grout. Consequently, a small sample of PT grout taken directly from the bag (partial-bag sample) may not contain the same proportion of constituents as that of the entire bag. For this reason, full bags of PT grouts should be blended thoroughly before taking small samples. This blending can be accomplished using an industrial blender designed to provide even dispersion of constituents when in powder form; these blenders are referred to as V-blenders.

A 3 ft^3 (0.085 m^3) capacity Patterson-Kelly twin shell V-blender with intensifier bar was used with the following procedures:

- Load pre-packaged dry grout into clean blender using scoop.
- Close blender.
- Start blender rotation and intensifier bar
- Mix for 30 min.
- Divide the material into approximately 400 g specimens and place in zippered plastic storage bags

8 Mixing and Agitation Equipment

High performance post-tensioning grouts are usually mixed with high-shear mixing equipment. As such, three mixers were studied during the course of research: a colloidal grout plant, a high shear sanitary mixer, and a blender mixer. Each of these mixers imposed a high shear rate to fully mix the grout samples. As indicated in Table 2, the primary difference among the three mixers is their capacity. One 50-lb (23 kg) bag of prepackaged PT grout yields approximately 0.5 ft^3 (0.014 m^3) of grout when mixed.

Table 2—Comparison of mixing volume for each mixer

Mixer	Mixing Volume
Colloidal grout plant	2 ft^3 to 10 ft^3 (56 L to 283 L)
High shear sanitary mixer	0.25 ft^3 to 0.75 ft^3 (7 L to 21 L)
Blender mixer	0.015 ft^3 to 0.035 ft^3 (0.42 L to 1 L)

8.1 Colloidal Grout Plant

Typically, a colloidal grout plant is used to mix and pump large quantities of grout in the field. It is unusual to find a colloidal grout plant in a construction materials testing laboratory because it is a large, production-oriented piece of equipment. The mix tank and agitation tank on a typical colloidal mixer are shown in Figure 8. The pump, which is not visible in the photograph, is located below the platform on the mixer. The colloidal mixer was equipped with a centrifugal diffuser-type pump rotating at speeds up to 2,000 RPM (209 rad/s) that disperses the cementitious material down to its finest particle size to achieve complete particle wetness. The agitator tank is equipped with a variable speed high-efficiency paddle mixer that prevents the grout from building structure before it is pumped. The grout pump, which is connected directly to the agitator tank, is a three stage progressing cavity, positive displacement, rotor-stator pump. The grout plant was powered by a 375 cfm (0.18 m^3/s) diesel air compressor. Two grout plant models were used in this research. One plant was powered by compressed air and the other was electric/hydraulic.

Figure 8—Colloidal grout plant with various parts labeled

The following procedure was used to mix and pump grout in the colloidal grout plant:

- Add water to mix tank and turn on colloidal mixer and agitator paddle.
- Add one bag of grout at a time. Add grout over a period of 20 s to 30 s per bag.
- Keep on the colloidal mixer and agitator paddle for 2 min after adding the last bag of grout.
- Transfer the grout to the agitation tank and start the paddle in the agitation tank.
- Transfer the grout to flow cone or container. See Figure 9.

Figure 9—Preparation for modified flow cone test

8.2 High-Shear Mixer

In addition to the colloidal mixer, a high shear sanitary mixer was also investigated (Figure 10). The high-shear mixer was well-suited to mix one 50-lb bag of PT grout at a time. The high-shear mixer contained both a low speed impeller and high speed impeller, which could be operated independently. The low speed anchor style impeller could operate at speeds up to 35 RPM (3.7 rad/s). The high-speed, saw-blade style impeller could operate at speeds up to 3,500 RPM (366 rad/s). The mix tank was jacketed by a hollow-wall, which when combined with a water bath allowed for precise temperature control during mixing. The mix tank also contained an air-actuated valve for sampling.

(a) (b)

Figure 10—High shear single bag mixer (a) over view and (b) close up of mixing impellers.

The following procedure was used to mix grout in the high-shear mixer:

- Ensure that the water bath is set at the desired testing temperature.
- Pour the water into the mixer tank and turn on the low speed impeller at 35 RPM (3.7 rad/s) until the water temperature is at the testing temperature (Figure 11).
- Continue mixing at the low speed impeller at 35 RPM (3.7 rad/s). Turn the high speed impeller on to 3,500 RPM (366 rad/s).
- Add the dry material over a period of 3 min to 5 min. Add slowly enough to avoid clumping of dry material (Figure 12).
- Once all dry material has been added, continue at the same speeds with both impellers for 30 s.

- Turn the impellers off, raise the mixer head, and scrape the mixer blades and walls of the mixer bowl over a period of 2 min (Figure 13).
- Lower the mixer head back into the mix tank. Turn again on the low speed and high speed impellers at 35 RPM (3.7 rad/s) and 3,500 RPM (366 rad/s) respectively for 90 s.

Figure 11—Dose tank with water

Figure 12—Add dry material to mixer over a period of 3 min to 5 min

Figure 13—Raise mixer head and scrape low speed agitator paddle

8.3 Blender Mixer

The blender mixer was used to mix small quantities of material (less than 0.25 L) that would be tested in the DSR. The blender was placed in series with a timer and speed controller that allowed for a repeatable mixing procedure. This research indicated that precise control of the mixing speed is necessary to ensure consistent results from DSR testing. The mixer could be operated at an angular velocity range from 500 RPM (52 rad/s) to 20,000 RPM (2094 rad/s). Figure 14 shows the blender on the right and the timer and speed controller on the left. Figure 15 shows the blade configuration in the blender. The wide blades allowed the mixer to impose a high shear rate on the grout, which caused the cement particles to disperse completely into the water. In addition, the blender was connected to a temperature-controlled water bath. This allowed control over the mixing temperature.

Figure 14—Waring HGBTAC30 commercial blender used for mixing

Figure 15—Blade configuration used in mix procedure with quarter shown for reference

The following procedure, which was based on ASTM C1738 (2011), was used to mix grout in the blender mixer:

- Set the temperature controller to desired testing temperature.
- Pour the water into the mixer and agitate at 4,000 RPM (418.8 rad/s) for 15 s.
- While the mixer continues to run at 4,000 RPM (418.8 rad/s), add the dry material over a period of 60 s.
- Once all dry material has been added, run the mixer at 10,000 RPM (1047 rad/s) for 30 s.
- Turn the mixer off and allow the sample to rest for 2 min.
- Run the mixer at 10,000 RPM (1047 rad/s) for 90 s.

After mixing, the grout was stored in a pre-heated stainless steel Thermos container which maintained the temperature to within 3°F (2°C) of the initial temperature over the duration of one hour. The grout was agitated using a plunger mixer (Figure 16) for 30 s before taking a sample to test in the DSR. This ensured that the specimen was homogenous.

Figure 16—Plunger mixer used to agitate grout before testing

9 Dynamic Shear Rheometer Equipment

A TA AR2000ex Dynamic Shear Rheometer (Figure 17) used for test method development had a torque range of 0.0001 mN·m to 200 mN·m. The torque range needed for test method development was approximately 0.5 mN·m to 20 mN·m. The maximum angular velocity was 300 rad/s. For test method development, the maximum angular velocity was approximately 70 rad/s.

DSR settings were adjusted by software that controlled angular velocity based on the desired shear rate desired. The software calculated and recorded shear rate, shear stress, and viscosity from the angular velocity and torque data. The software also allowed for control of sample temperature during testing.

Figure 17—Dynamic Shear Rheometer with helical ribbon and cup geometry

9.1 DSR Geometries

Serrated parallel plates and cup and helical ribbon were used in the initial DSR tests conducted on plain and PT grout. Parallel plates were the first geometry considered because the shear stress and strain imposed on the fluid can be computed directly. The complexity of the fluid motion caused by ribbon or vane geometries, however, does not allow the direct computation of shear stress and strain, which make its selection less desirable. This section describes the details of these geometries. (See Section 5.1 for more details)

9.1.1 Serrated parallel plates

A set of stainless steel serrated parallel plates was considered as a part of the DSR test method development. The serrated parallel plates were 40 mm in diameter. Serrations were 0.5 mm deep and formed 90° grid pattern over the surface of the plate (Figure 18).

Figure 18—Serrated parallel plate DSR geometry – bottom plate (left) and top plate (right)

The serrations on the parallel plate allowed for testing of suspensions with large particle sizes, as shown by Ferraris et al. (2007). If smooth plates are used for testing suspensions with large particles, slippage can occur that would invalidate the test. The DSR had a Peltier system that controlled sample temperature during testing.

Equation 1 and Equation 2 show how shear rate and shear stress were calculated using the parallel plates. If the serrated parallel plate geometry is used, a correction factor must be applied (Ferraris et al. 2007). The formula for correcting the viscosity of the plates is given in Equation 3.

$$\dot{\gamma} = \frac{\omega \cdot r}{h}$$ Equation 1

where $\dot{\gamma}$ is the shear rate, ω is the angular velocity, *r* is the radius of the plate, and *h* is the distance between the plates.

$$\tau = T \frac{1}{\frac{2}{3} \pi \cdot r^3}$$ Equation 2

where τ is the shear stress, *T* is the torque measured by the rheometer, and *r* is the radius of the plate.

$$\eta_c = \eta(1 + \frac{c}{h})$$ Equation 3

where η_c is the corrected viscosity (mPa·s), η is the viscosity measured using the serrated plate (mPa·s), c is the gap correction value (0.32 mm for the DSR and plates used in this research), and h is the size of the gap (mm).

9.1.2 Cup and ribbon

A stainless steel cup and helical ribbon geometry was investigated (Figure 19). Schematics of the helical ribbon and cup can be seen in Figure 20.

Figure 19—Helical ribbon (left) and cup (right) DSR geometry

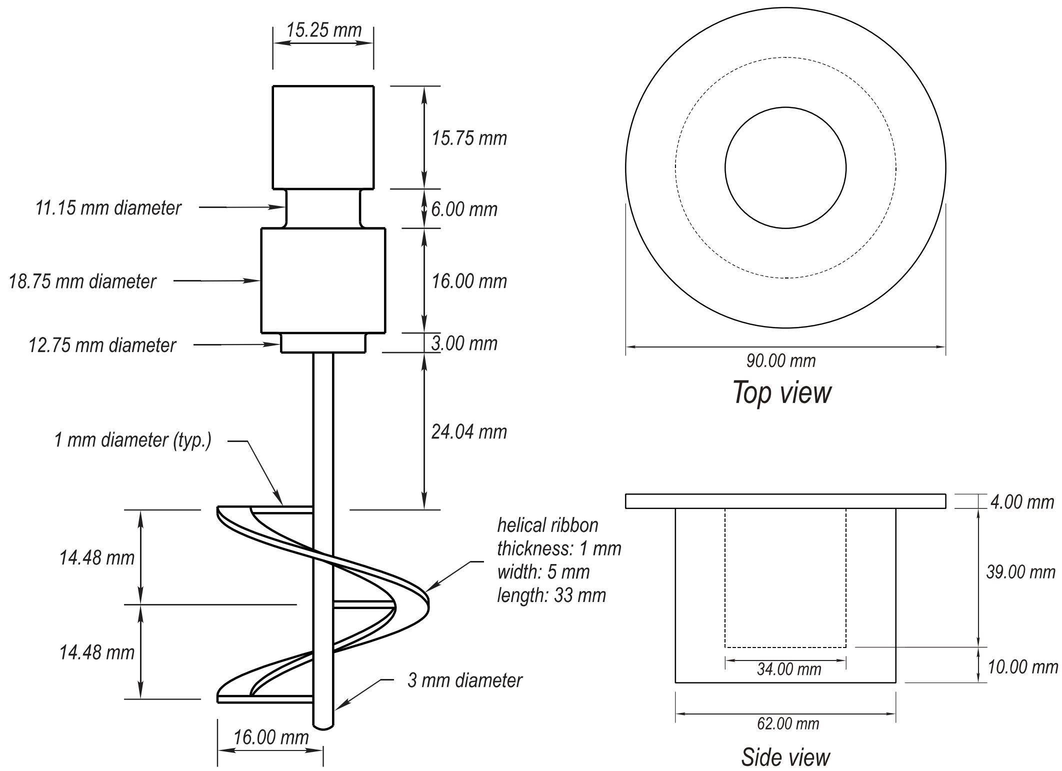


Figure 20—Helical Ribbon and Cup Schematic

To prevent sedimentation the cup and ribbon geometry was used over long test periods. The cup and ribbon geometry allowed for the testing of a suspension with larger particle sizes than the serrated parallel plate would allow. The cup and ribbon also allowed for the testing of materials with a very low viscosity, which may flow off of the parallel plates during testing. The cup is enclosed by a Peltier jacket (not shown), which controls sample temperature during testing. The procedure consisted of: 1) filling the cup with grout, and 2) then lowering the ribbon into the cup. The ribbon rotates at a given angular velocity, and the DSR measures the corresponding torque.

The helical ribbon was empirically calibrated by the manufacturer, probably using standard oils assuming that the flow is similar to a couette geometry. This was accomplished by determining average stress coefficients for the helical ribbon and using these to calculate stress from the strain measured by the rheometer. The manufacturer of the rheometer provided a coefficient for the shear rate based on the shape of the geometry. Equation 4 and Equation 5 show how shear stress and shear rate are calculated from the torque and angular velocity recorded.

$$\tau = F_{\sigma} \cdot T \qquad \text{Equation 4}$$

where τ is the shear stress, F_{σ} is the shear stress factor, and T is the torque measured by the rheometer. The values of F_{σ}, as given by the manufacturer was 2,730 m^{-3} for the helical ribbon geometry. The value of F_{σ} was calibrated using a NIST standard reference material (see Section 9.2 for more details).

$$\gamma = F_{\cdot} \cdot \omega \qquad \text{Equation 5}$$

where $\dot{\gamma}$ is the shear rate, $F_{\cdot}$ is the shear rate factor, and ω is the angular velocity. $F_{\cdot}$ from the manufacturer is equal to 2.460 for the helical ribbon geometry. This factor was not adjusted using SRM.

For the remainder of the report the calibration factors (as determined in Section 9.2) were used to transform the measured values of torque and rotational speed to fundamental units of shear stress and shear rate.

9.2 Cup and Ribbon Geometry Calibration

To use the cup and ribbon geometry for testing of PT grouts, it was necessary to ensure that the results were repeatable across multiple laboratories. During preliminary investigations on the grouts, the stresses measured using the cup and ribbon geometry did not match those measured using the parallel plate geometry when using the shear stress factor provided by the manufacturer.

Several standard materials were considered to calibrate the cup and ribbon geometry. A standard oil was inadequate for calibration because it was not a suspension and did not exhibit a yield stress as would a PT grout. A NIST Standard Reference Material (SRM 2492) was used to determine a more appropriate shear stress factor as the NIST SRM is a Bingham material similar to the grouts.

9.2.1 Reference Material

The NIST Standard Reference Material (SRM) 2492, “Bingham Paste Mixture for Rheological Measurements”, was used to calibrate the shear stress measured using the cup and ribbon geometry. The material was a mixture of corn syrup aqueous solution and limestone. The SRM included instructions for sample preparation and testing.

The reference material was a suspension that behaved as a Bingham plastic. The SRM had a certified yield stress (τ_{cert}) and plastic viscosity (η_{cert}) over a period of seven days after mixing (Figure 21). In addition to an expected mean value, standard uncertainty and expanded uncertainty values were provided. The standard uncertainty limits represent the standard deviation of the values measured during NIST testing. The expanded uncertainty limits represent a 95% confidence level.

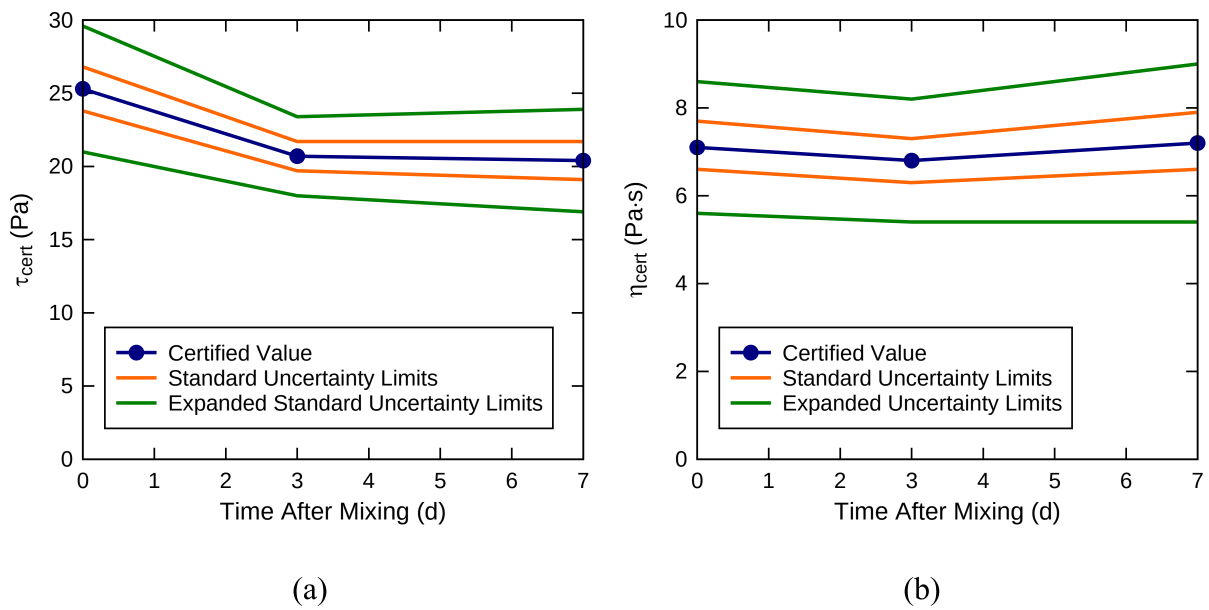


Figure 21—NIST SRM certified (a) yield stress and (b) viscosity with respect to time after mixing

9.2.2 DSR Calibration Results

The SRM was tested using the DSR with the cup and ribbon geometry to determine appropriate shear stress calibration factors. The following test procedure was used to generate a flow curve:

- Prepare sample according to ASTM C1738 (2011).
- Load the material into the cup and ribbon geometry.
- Shear the material at a rate of 0.01 s^{-1} for a period of 150 s.
- Shear the material at a rate of 0.1 s^{-1} to 50 s^{-1} with increasing steps. At each step measure every 1 s. If 3 consecutive points are within 5% of each other, the material has reached equilibrium. Record the equilibrium stress value. Ten points are recorded in this phase (up-curve)
- Shear the material at a rate of 50 s^{-1} to 0.1 s^{-1} with decreasing steps. At each step measure every 1 s. If 3 consecutive points are within 5% of each other, the material has reached

equilibrium. Record the equilibrium stress value. Twenty points are recorded in this phase (down-curve)

- Plot shear stress vs. shear rate and determine appropriate Bingham parameters.

Two batches of SRM were prepared and tested. The first batch was prepared with tap water although the SRM certificate specifies distilled water. The second batch was prepared with distilled water. Results of the flow curve test for the SRM prepared with tap water on the same day as mixing are indicated in Figure 22.

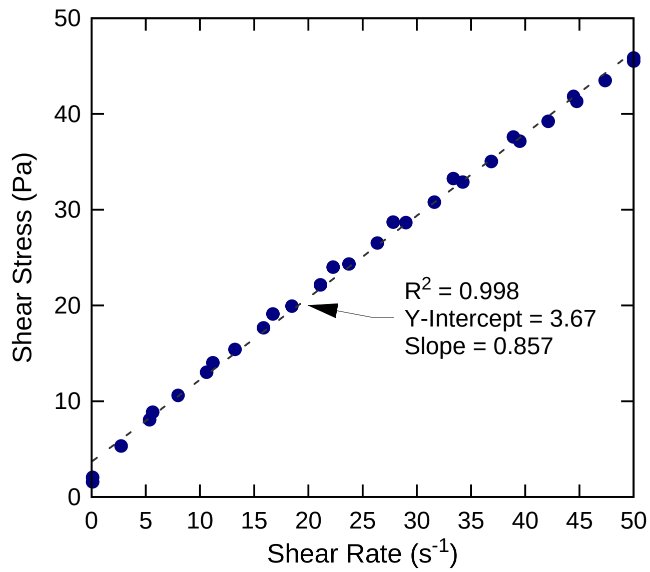


Figure 22—Flow curve results for SRM (prepared with tap water) on the same day as mixing. The uncertainty on the date shown is considered as calculated for the SRM 2492 (see Figure 22)

If a linear trend line is fitted to the flow curve in Figure 22, the yield stress was 3.67 Pa while the plastic viscosity was 0.857 Pa·s. Uncertainty cannot be calculated from this experiment as the measurement was done only once. The R^2 value was 0.998. The flow curve test was conducted on the material at the following times after mixing: 0 d (same day as mixing), 3 d, and 7 d. Figure 23 shows plots of τ_{cert}/τ_{meas} and η_{cert}/η_{meas} for batches prepared with tap water and distilled water. Table 3 provides the data plotted in Figure 23. As per the uncertainty from the SRM 2492 (see figure 22), the results obtained with tap water or distilled water are identical.

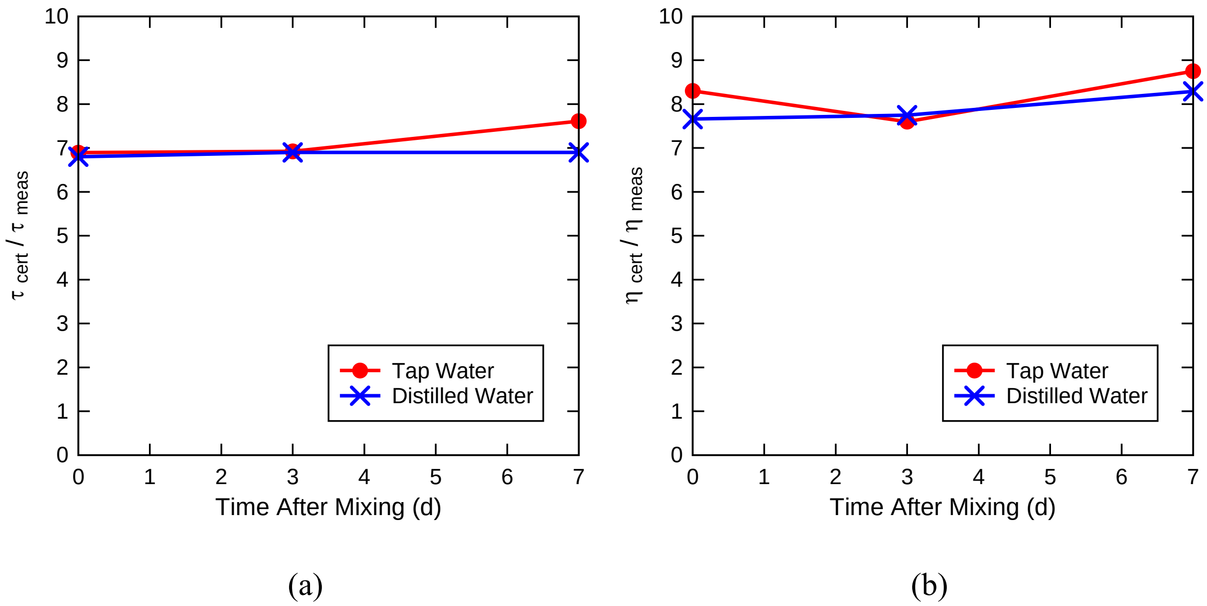


Figure 23—Ratios of (a) yield stresses and (b) viscosities for batches prepared with tap water and distilled water. These are single measurements. The uncertainty is considered as calculated for the SRM 2492 (see Figure 22)

Table 3—Summary of yield stress and viscosity measured on SRM

Water	Age (d)	τ_{meas} (Pa)	τ_{cert}/τ_{meas}	Age (d)	η_{meas} (Pa·s)	η_{cert}/η_{meas}
Tap	0	3.67	6.9	0	0.857	8.3
	3	2.99	6.9	3	0.891	7.6
	7	2.68	7.6	7	0.821	8.8
Distilled	0	3.74	6.8	0	0.927	7.7
	3	3.02	6.9	3	0.878	7.7
	7	2.98	6.9	7	0.869	8.3

Equation 6 and Equation 7 were used to adjust the measured yield stress and plastic viscosity to the corresponding certified values.

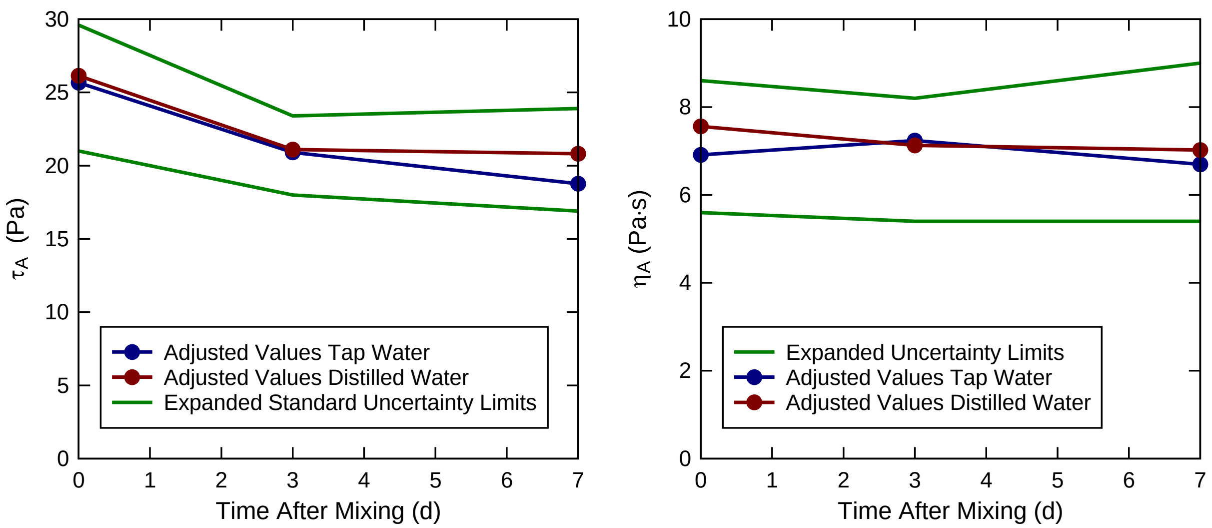
$$\tau_A = A \cdot \tau_{meas} \quad \text{Equation 6}$$

where τ_A is the adjusted yield stress, A is the adjustment factor, and τ_{meas} is the yield stress measured by the rheometer.

$$\eta_A = B \cdot \eta_{meas} \quad \text{Equation 7}$$

where η_A is the adjusted plastic viscosity, B is the adjustment factor, and η_{meas} is the plastic viscosity measured by the rheometer.

The adjustment factors *A* and *B* were determined by averaging the six yield stress and six viscosity ratios in Table 3, which resulted in average adjustment factors of *A* = 7.0 and *B* = 8.1. Figure 24 contains plots showing the adjusted yield stress (τ_A) and plastic viscosity (η_A) with respect to time after mixing respectively.



(a) (b)

Figure 24—Adjusted values of (a) yield stress and (b) plastic viscosity

The adjusted yield stress and the adjusted plastic viscosity are both within the expanded uncertainty limits of the SRM for both batches indicating that the selected adjustment factors *A* and *B* are appropriate. The adjustment factor was applied to the DSR by multiplying the manufacturer's shear stress factor by 7.0 (7.0 × 2,730 m^{-3} = 19,110 m^{-3}) for yield stress measurements and by 8.1 (8.1 × 2,730 m^{-3} = 22,110 m^{-3}) for viscosity measurements.

9.2.3 Uncertainty Estimation

All the data in this report are the results of singular tests with no repeated or replicate tests conducted. Consequently, no statistical evaluation was attempted. Nevertheless, the standard uncertainty of the measurement of viscosity and yield stress could be estimated from the data collected from the SRM as shown in Figure 23 (and the SRM certificate) and Figure 24 to be less than 10 %. Thus for the rest of the paper an estimate uncertainty of the upper limit will be 10 %.

9.3 DSR Geometry Selection

Both the serrated parallel plates and the helical ribbon allow for the testing of suspensions with large particle sizes. The size of the gaps between the serrated plates was dependent on the largest particle size in the suspension; the cup and ribbon had no such requirement. Because the PT grouts are all proprietary mixtures with different maximum particle sizes, a distinct gap parameter would be required to test each grout. This would complicate tests on future PT grouts that have not yet been developed, and increase the likelihood of error during testing.

The helical ribbon allows for testing over a longer time domain than the parallel plates, because it allows the remixing of the specimen while testing, thus preventing particle settlement during testing. The same sample can be kept in the rheometer during 1 h of testing for helical ribbon, while a new specimen must be loaded at regular intervals for parallel plate, preventing measurements while changing samples. Temperature control was another important factor for each geometry. The bottom plate in the parallel plate geometry was temperature controlled, but the top plate was not. Even when the top plate was left in contact with the bottom plate for approximately 5 min prior to testing, the temperature of the top plate was about 8°F (4°C) lower than the bottom plate when the bottom plate was 90°F (32.2°C). Maintaining the temperature is paramount to simulate the HTGF environment. The cup, however, is enclosed by a Peltier jacket, which more precisely maintains the sample temperature in the cup over long testing durations.

During preliminary testing, specimens were difficult to load onto the parallel plates. The cement pastes, even at a relatively high w/c ratio such as 0.5, were much more viscous than the PT grouts. If using the parallel plates, the material must not flow over the edge of the bottom plate to ensure that the gap between plates is uniformly filled. Because the PT grouts had a very low viscosity, some grout would flow out of the gap between the plates rendering the measurement invalid. Previous research performed on grouts with viscosity-reducing admixtures, such as that by Khayat et al. (1999), has also found that the cup-type rheometer geometry is more effective than parallel plate geometries for low viscosity grouts.

The primary disadvantage of the cup and ribbon geometry is that it does not allow for the exact calculation of shear stress and shear rate. Using the NIST SRM 2492 to calibrate the geometry, however, this inconvenience can be mitigated. For these reasons, the cup and ribbon was selected as the most suitable test geometry for PT grout testing.

10 Nominal Shear Rate (NSR) Viscosity Test Development

Non-Newtonian fluids exhibit different viscosities at different shear rates, so it follows that the viscosity of a non-Newtonian fluid is a function of the applied shear rate. The viscosity of grout was mentioned in the literature by Khayat and Yahia (1998) and Amziane and Ferraris (2007). Both reports indicated that the viscosity over a range of shear rates seemed to provide an indication of the fluidity of the grout. Consequently, a general procedure was:

- Load a sample of grout into the DSR.
- Subject the grout to the high pre-shear rate for 30 s to remove the effects of inconsistent shear histories during loading.
- Subject the grout to the testing shear rate for the testing time. The viscosity is the viscosity measured at the end of the testing time.
- Clean the grout out of the DSR.

This test was named the nominal shear rate (NSR) viscosity test. For the purposes of this research, the viscosity test should simulate the fluidity measurement from the flow cone test as closely as possible. Appropriate parameters were selected for the testing shear rates and testing time that simulated the flow cone test and allowed for an assessment of the fluid grout behavior at high temperatures.

10.1 Shear Rates to Simulate HTGF Test Conditions

To determine an appropriate shear rate domain for PT grout testing, the shear rate imposed by a flow cone nozzle was calculated. The actual shear rate at the outlet of the flow cone nozzle is dependent upon the viscosity of the grout which is being discharged as well as the geometry of the nozzle. The calculation of shear rate in the flow cone nozzle for a non-Newtonian fluid is very complex. For this reason, it was assumed that the material in the nozzle was Newtonian. This should provide a reasonable order-of-magnitude estimate for the shear rates to be used in testing. The equation for calculation of the shear rate of a Newtonian fluid at the wall of a pipe is given in Equation 8 [Collier, et al.].

$$\gamma = \frac{4Q}{\pi r^3} \qquad \text{Equation 8}$$

where $\dot{\gamma}$ is the shear rate, Q is the flow rate of the material in the pipe, and r is the radius of the pipe.

The flow cone efflux time of the grout has an impact on the shear rate calculation. Assuming a 30 s flow cone efflux time, which corresponds to the failure limit, the flow rate of the grout in the nozzle is approximately 2 in^3/s (33 mL/s). The shear rates imposed by different sized flow cone nozzles at this flow rate are given in Table 4.

Table 4—Summary of shear rates imposed by flow cone nozzle

Shear Rate	Flow cone nozzle diameter
165 s^{-1}	½ inch (12.7 mm)
50 s^{-1}	¾ inch (19.0 mm)

10.2 Testing Shear Rate

The ideal testing shear rate for the NSR viscosity test should allow for the comparison of grouts in the same manner as the flow cone test. A continuously varying range of shear rates are imposed on the grout in the nozzle of the flow cone during the flow cone test. The shear rate is dependent upon the viscosity of the grout and the geometry of the nozzle. The shear rate reduces as the pressure head of the grout above the nozzle drops. It was therefore necessary to investigate the grout behavior over a range of shear rates to determine empirically if a single shear rate could simulate the flow cone measurement.

As shown in Table 4, the shear rate in the ½-in.(12.7 mm) flow cone nozzle is 165 s^{-1}. Consequently, shear rates in the range from 0 to 165 s^{-1} were investigated.

To determine a relationship between NSR viscosity test and flow cone efflux time, simultaneous flow cone and DSR tests were conducted multiple times on all of the grouts. Figure 25 through Figure 30, show the viscosity plotted against the flow cone efflux time measured for the same grout sample at a shear rate of 5 s^{-1}, 25 s^{-1}, 50 s^{-1}, 75 s^{-1}, 100 s^{-1}, and 165 s^{-1} respectively. Results for all of the grouts are included in each of the plots. Table 5 contains a summary of trend-lines fitted to the data in each of these plots.

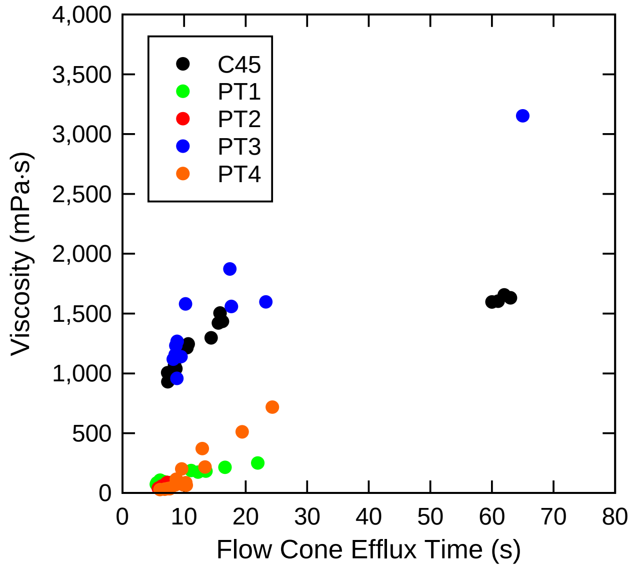


Figure 25—Comparison of DSR viscosity and flow cone efflux time for shear rate of 5 s^{-1}. The uncertainty is discussed in section 9.2.3 for viscosity and 0.88 s for the flow cone (ASTM C939)

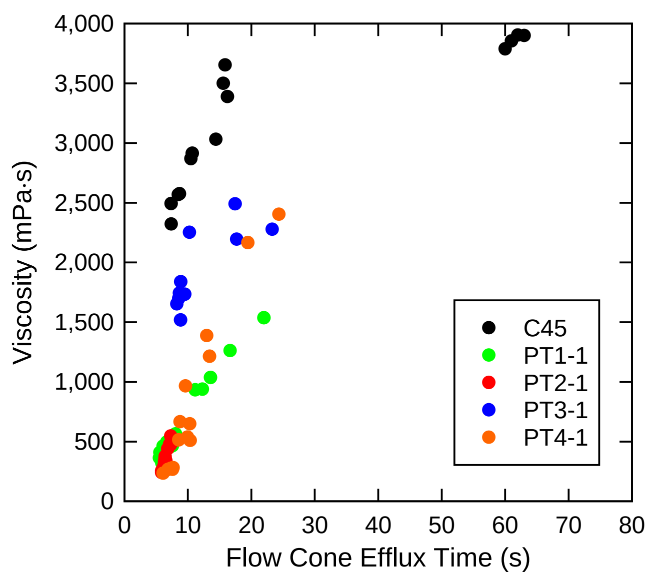


Figure 26—Comparison of DSR viscosity and flow cone efflux time for shear rate of 25 s^{-1}. The uncertainty is discussed in section 9.2.3 for viscosity and 0.88 s for the flow cone (ASTM C939)

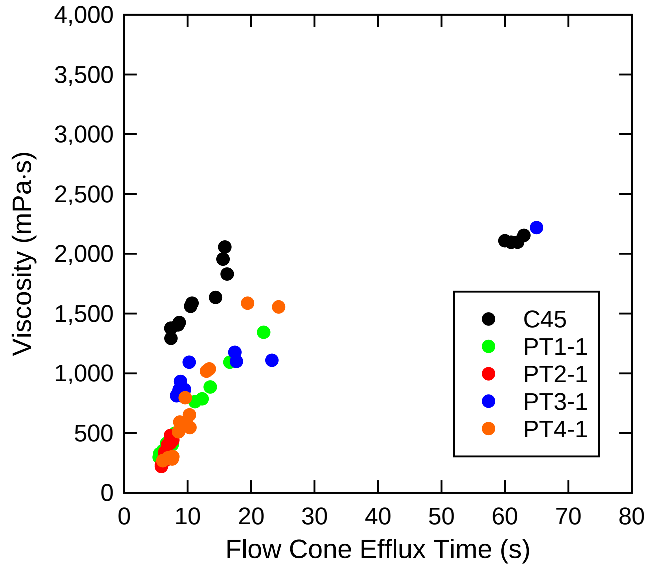


Figure 27—Comparison of DSR viscosity and flow cone efflux time for shear rate of 50 s^{-1}. The uncertainty is discussed in section 9.2.3 for viscosity and 0.88 s for the flow cone (ASTM C939)

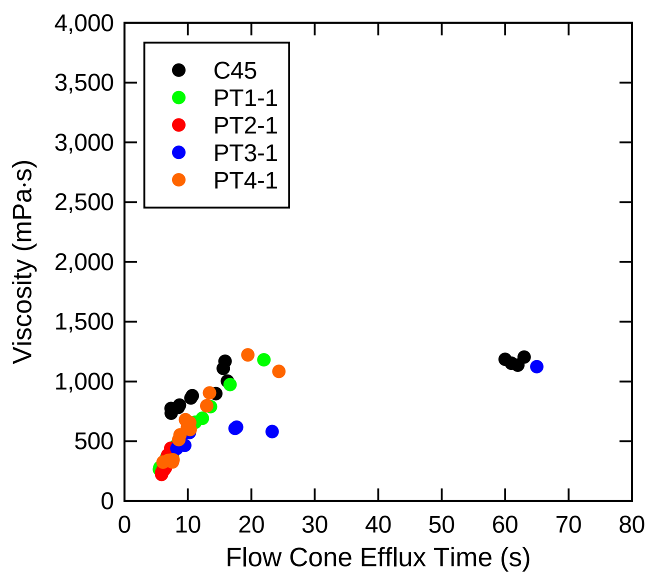


Figure 28—Comparison of DSR viscosity and flow cone efflux time for shear rate of 75 s^{-1}. The uncertainty is discussed in section 9.2.3 for viscosity and 0.88 s for the flow cone (ASTM C939)

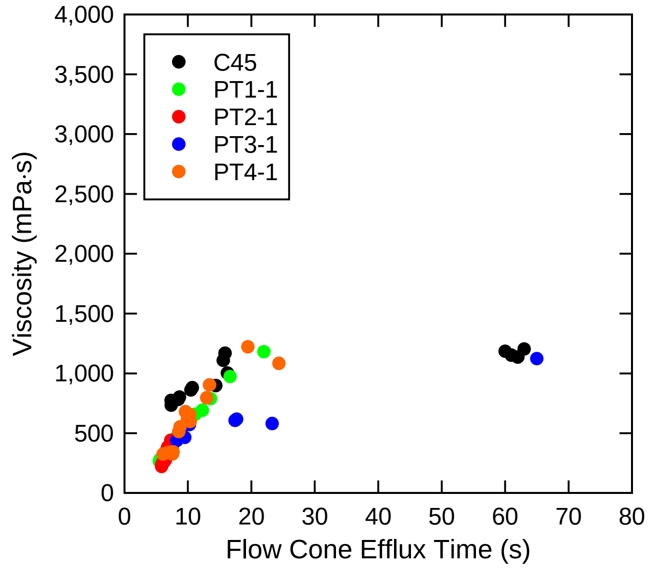


Figure 29—Comparison of DSR viscosity and flow cone efflux time for shear rate of 100 s^{-1}. The uncertainty is discussed in section 9.2.3 for viscosity and 0.88 s for the flow cone (ASTM C939)

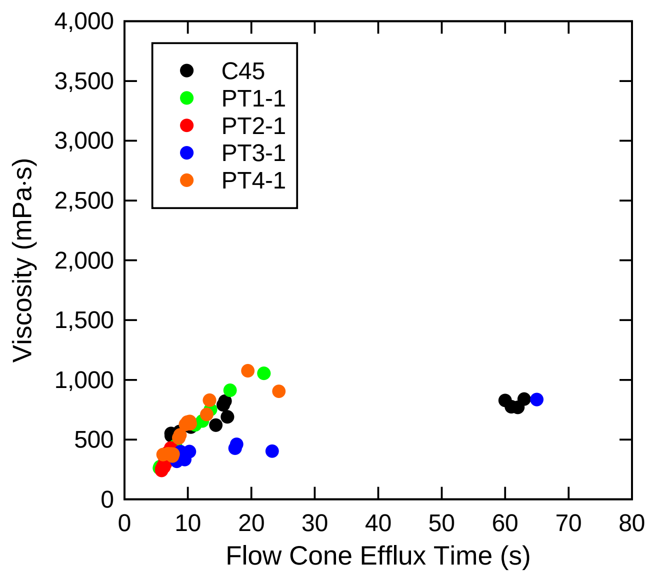


Figure 30—Comparison of DSR viscosity and flow cone efflux time for shear rate of 165 s^{-1}. The uncertainty is discussed in section 9.2.3 for viscosity and 0.88 s for the flow cone (ASTM C939)

Table 5—Summary of R^2 values for linear trend-line fit to viscosity versus flow cone efflux time results at different shear rates

Shear Rate	R^2 PT Grouts	R^2 All Grouts
5 s^{-1}	0.52	0.44
25 s^{-1}	0.70	0.54
50 s^{-1}	0.71	0.55
75 s^{-1}	0.61	0.53
100 s^{-1}	0.51	0.49
165 s^{-1}	0.35	0.35

Table 5 shows that the best linear trend exists between flow cone efflux time and viscosity when the shear rate is either 25 s^{-1} or 50 s^{-1}.

At a low shear rate, such as 5 s^{-1} or 25 s^{-1}, grout PT3 exhibits a much higher viscosity than the other PT grouts at the same flow cone efflux times. A shear rate of 25 s^{-1} or less does not allow for the comparison of PT grouts in the same manner as the flow cone test. At a shear rate of 75 s^{-1} or higher, the viscosity of grout that has a flow cone efflux time of approximately 20 s is nearly identical to the viscosity of a grout that fails with a flow cone efflux time over 60 s. DSR shear rates less than 75 s^{-1} provide viscosity test results that give a clear indication of grout performance.

At a shear rate of 50 s^{-1} all of the PT grouts exhibit the best linear trend when comparing the viscosity to the flow cone efflux time. Grout PT3 does not quite follow this trend, but the thixotropic and shear-thinning nature of the grout causes a variation in flow cone efflux time measurement. At this shear rate, there is a distinction in viscosity between the PT grouts with a flow cone efflux time less than 30 s and those with a flow cone efflux time greater than 30 s. The plain grouts do not follow the trend of the PT grouts at this shear rate. When the flow cone efflux times were the same, however, plain grouts appeared more viscous than the PT grouts. Previous research by Roussel and Le Roy (2005) showed that materials with very different viscosities could exhibit similar flow cone efflux times. For this reason, the viscosity test provides a more accurate measure of the fluidity of a grout than the flow cone test does.

Based on the above analysis, a testing shear rate of 50 s^{-1} was deemed the most appropriate for the NSR viscosity test to simulate the flow cone test. This shear rate resulted in a very similar viscosity for all of the PT grouts at a nominal flow cone efflux time. It also allows for a quantitative distinction between grouts that pass the flow cone test and grouts that fail the flow cone test. A pre-shear rate of 165 s^{-1} was selected to eliminate inconsistencies in the shear history of the grout during loading.

10.3 Testing Time

The time domain for the NSR viscosity test is critical. All of the grouts exhibit varying degrees of thixotropy and anti-thixotropy, meaning that their viscosity will change over time if they are subjected to a constant shear rate, as they are in the NSR viscosity test. Some grouts exhibit both thixotropy and anti-thixotropy, depending on the time domain over which they are tested. Figure 31 contains the results of a 60 min test conducted on grouts C45, PT1-1, PT2-1, PT3-1, and PT4-1. Grouts PT1-1, PT2-1, and PT3-1 were mixed at the manufacturer's maximum specified water content. Grout PT4-1 was mixed at the mid-range of the manufacturer's specified water content.

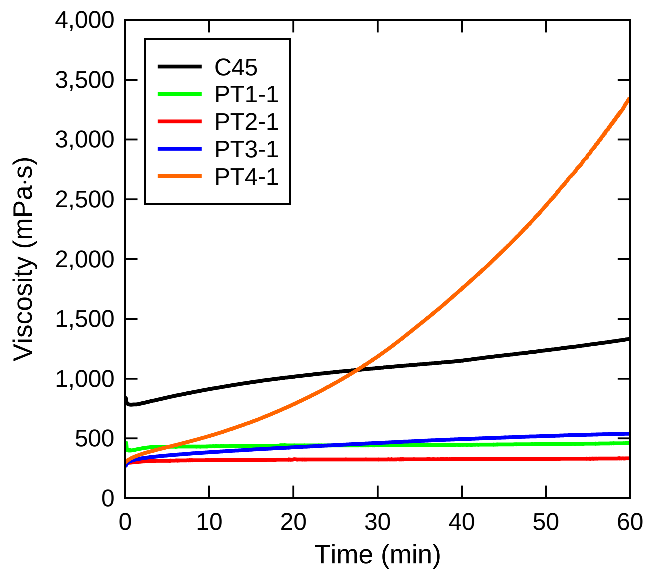


Figure 31—Results of 60 min NSR viscosity test conducted on grouts C45, PT1-1, PT2-1, PT3-1, and PT4-1. The uncertainty is discussed in section 9.2.3

The initial viscosity of grout C45 is approximately 750 mPa·s. After 30 min of testing, however, the viscosity is approximately 1,000 mPa·s. This is an indication that, at a shear rate of 50 s^{-1}, grout C45 is anti-thixotropic, because its viscosity increases over time when subjected to a constant shear rate with no rest intervals. Grout PT4-1 exhibits anti-thixotropy to a greater degree than grout C45. Grout PT3-1 exhibits a small degree of anti-thixotropy at a shear rate of

50 s^{-1} as well. Grouts PT1-1 and PT2-1 exhibit a nearly constant viscosity throughout the 60 min testing period. This is a good indication that, at a shear rate of 50 s^{-1}, grouts PT1-1 and PT2-1 are not thixotropic or anti-thixotropic.

The results of the first 5 min of the viscosity tests that are shown in Figure 31 are given in Figure 32.

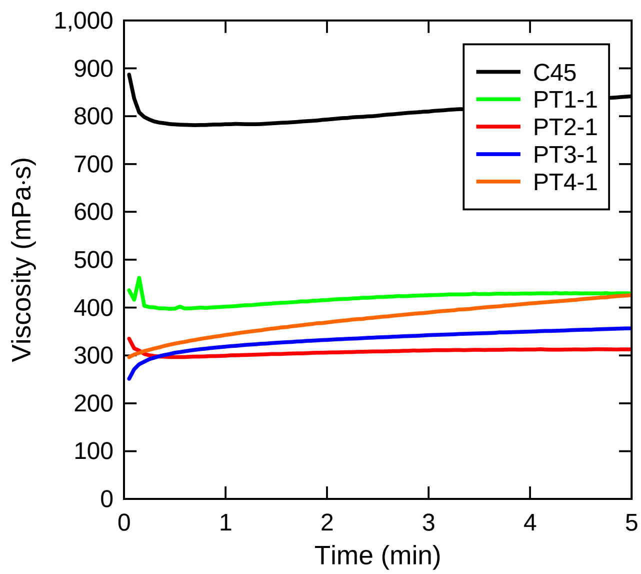


Figure 32—Results of first 5 min of NSR viscosity test conducted on grouts C45, PT1-1, PT2-1, PT3-1, and PT4-1. The uncertainty is discussed in section 9.2.3

Each of the grouts exhibits some thixotropy or anti-thixotropy over the first 15 s of testing. Grout C45 is initially thixotropic, before later exhibiting anti-thixotropy. Grouts PT3-1 and PT4-1 both exhibit anti-thixotropy in this time domain. Grouts PT1-1 and PT2-1 do not exhibit a large degree of thixotropy or anti-thixotropy. Hydration cannot be the source of these differences due to the short time.

A test length of 1 min is comparable to the total time it takes to conduct one flow cone test. At 1 min, the viscosities of grouts C45, PT3-1, and PT4-1 are in the middle of an increasing trend. Figure 31 showed that the viscosity of these materials will continue to increase as cement hydration progresses and structure builds. Taking a reading of the viscosity after 1 min negates the large effects of thixotropy in the first 15 s. A 1 min test also allows for testing to be completed in a reasonable amount of time without significant effects on the results. For these reasons, the NSR viscosity test should be run for 1 min to simulate the flow cone measurement.

10.4 Summary

The measurement of the viscosity at a specific shear rate of a non-Newtonian fluid was mentioned by previous grout researchers Khayat and Yahia (1998) and Amziane and Ferraris (2007). Parameters for the testing shear rate and testing time for the viscosity test were selected that allowed for a simulation of the flow cone test.

The following variables allow for the most direct relationship between flow cone efflux time and viscosity for the PT grouts:

- Pre-shear rate: 165 s^{-1}
- Testing Shear Rate: 50 s^{-1}
- Testing Time: 1 min

The NSR viscosity test can be used to simulate the flow cone test. The test results in the measurement of a similar viscosity for the PT grouts at a nominal flow cone efflux time. The finalized procedure for the viscosity test is as follows:

For comparison to flow cone test:

- Load a sample of grout into the DSR.
- Subject the grout to a shear rate of 165 s^{-1} for 30 s.
- Subject the grout to a shear rate of 50 s^{-1} for a period of 1 min, sampling every 1 s. The NSR viscosity is the viscosity measured at 1 min.
- Clean the grout out of the cup and ribbon.

To simulate HTGF conditioning and measure change in viscosity conduct test as follows:

- Set temperature control to 100° F (37.8° C)
- Load sample into the cup so that the free surface of the grout is in line with the cup rim.
- Pre-shear sample at 165 s^{-1} for 30 s.
- At the conclusion of pre-shearing, immediately initiate a shear rate of 50 s^{-1} continuously for 60 min.
- Record viscosity at one min and at 15-min intervals during the test.

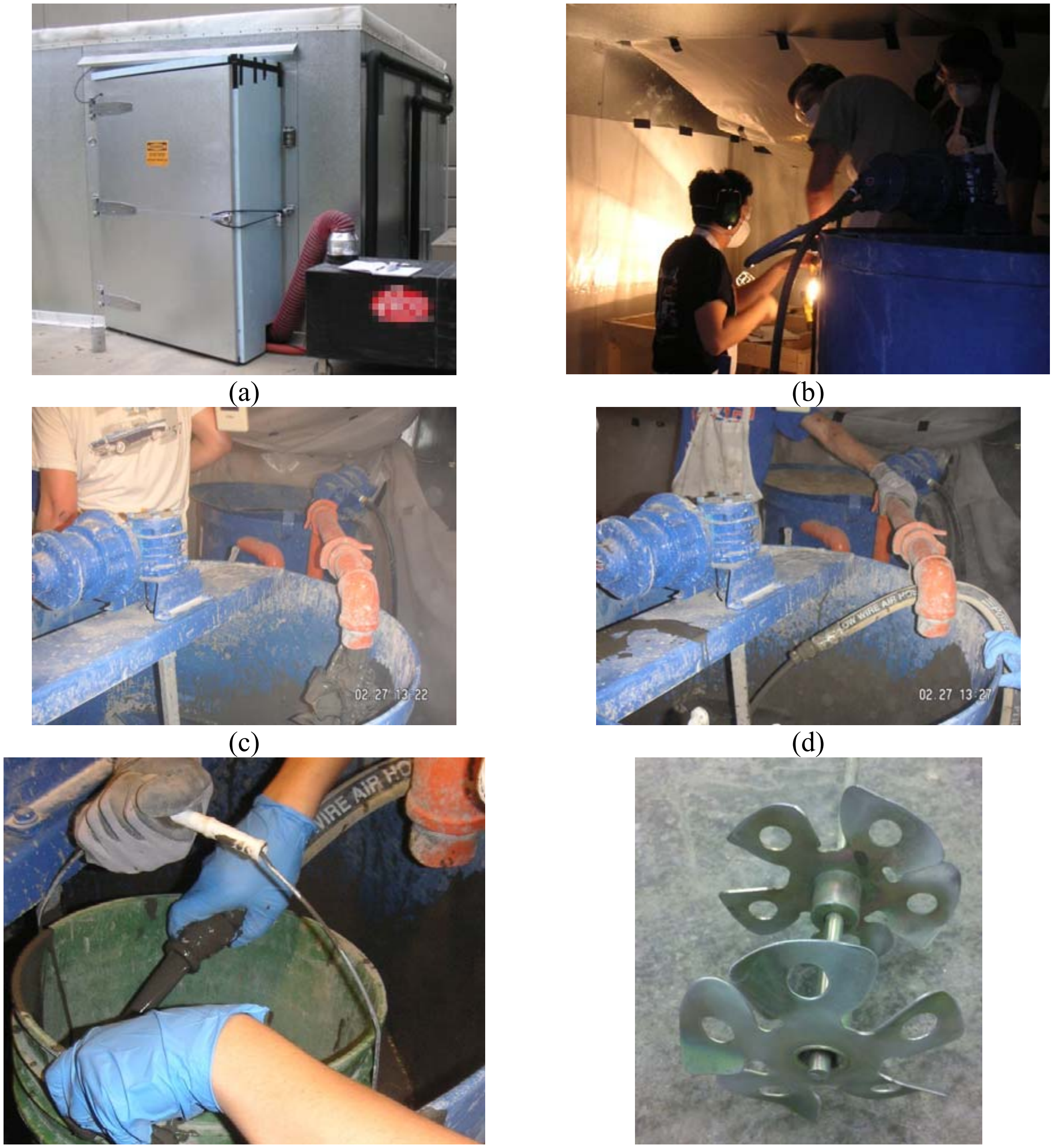
11 High-Temperature Grout Fluidity (HTGF) and Related Tests

The HTGF test is used to determine the pumpability of grout under simulated hot weather conditions. Tests were conducted by mixing a batch of grout using the colloidal mixer. The grout materials and equipment were conditioned in a 90°F (32.2°C) environment for at least 18 h prior to initiating the test. After mixing, the grout was continuously pumped through 400 ft. (122 m) of hose, also in the conditioned environment, for a period of 60 min. The 60 min conditioning period began when the grout had fully re-circulated through the 400 ft. (122 m) of 1-in. (25-mm) hose. Flow cone efflux times were measured every 15 min to check the fluidity of the grout. The grout was considered to pass the test if the flow cone efflux times were less than 30 s at every point during testing. The general procedure for the HTGF test as published by FDOT (2007) is as follows:

1. Mix grout in colloidal grout plant according to procedures in Section 8.1. Grout plant and grout materials are stored in conditioned environment (Figure 33).
2. Transfer grout to agitation tank and start agitator paddle.
3. Pump grout from agitation tank through 400 ft. (122 m) of 1-in (25.4 mm) grout hose which returns to agitation tank.
4. Continue re-circulating grout for 60 min. Maintain flow rate between 0.1 gal/s (1.36 m^3/h) and 0.25 gal/sec (3.4 m^3/h), per PTI specifications (Post-Tensioning Institute 2012).
5. Take a sample at the following times after re-circulation is complete: 0 min, 15 min, 30 min, 45 min, and 60 min. Record the following for each sample: ambient temperature, grout temperature, pumping pressure, hose flow rate, flow cone efflux time, wet density specific gravity, and unit weight.
6. Every time a sample is collected, use the DSR to conduct the NSR viscosity test.

The sampling procedure to take a sample for testing from the HTGF test is as follows:

1. Collect 2 gal to 3 gal (7.5 L to 11.3 L) sample from the return end of the hose into a 5-gal (19L) bucket
2. 10 s before performing a flow cone test, shear the grout in the bucket using a paint-paddle style agitator.
3. Collect specimens for the other tests from the flow cone discharge.



(a) (b) (c) (d) (e) (f)

Figure 33—HTGF test (a) environmental chamber, (b) inside chamber during test (c) grout transfer (d) grout recirculation, (e) grout sampling, and (f) paddle used to agitate grout before flow cone test.

11.1 Flow Cone Test

The flow cone is typically used in the field to measure the fluidity of freshly mixed grout. Figure 34 shows the flow cone test being setup. The flow cone test was conducted on each grout sample collected during the HTGF test.

Figure 34—Flow cone used to determine fluidity of grout samples during HTGF testing

The flow cone test procedure is a modified version of ASTM C939 (2010) using 0.5-in. (12.7 mm) diameter nozzle. The procedure for conducting the flow cone test is as follows:

1. Fill flow cone with water.
2. One minute or less before introducing the grout, drain the water out of the flow cone and close the outlet with a rubber stopper.
3. Fill flow cone to top with grout.
4. Place 1 L cylinder below flow cone.
5. Remove the rubber stopper from the outlet and start stopwatch.
6. After 1,000 mL has drained from flow cone, stop the stop watch.
7. Record the time as the modified flow cone efflux time.

11.2 Wet Density Test

The mud balance is used in the field to determine the wet density of a small sample of fresh grout (Figure 35). The test was conducted on each grout sample collected during the HTGF test in accordance with ASTM D4380 (2006). The uncertainty for this test method in single laboratory tests has not yet been established. The procedure was as follows:

1. Pour a sample of grout into the cup on the mud balance.
2. Tap the side of the cup to ensure that air bubbles rise to surface.
3. Place the cap onto the cup. Allow grout to flow out through the hole in the cap, ensuring that the cup is totally filled.
4. Place the mud balance assembly on the fulcrum in the case.
5. Adjust the weight on the balance arm until the mud balance is level.
6. Record the specific gravity of the grout sample.

Figure 35—Mud balance used to determine wet density of grout samples during HTGF testing

11.3 Unit Weight Test

The unit weight test was also conducted to determine the unit weight of fluid samples of grout using a vessel with a constant volume of 0.0141 ft^3 (400 ml) (Figure 36) as described in ASTM C185. The unit weight test was conducted on each grout sample collected during the HTGF test. The uncertainty in the mass measured should be less than 0.5 g. The procedure for the unit weight test was as follows (Figure 36):

1. Tare the unit weight cup and glass top.
2. Fill the cup with grout. Tap the side of the cup to ensure that air bubbles rise to surface.
3. Use the glass top to screed the surface of the grout in the cup.
4. Weigh the filled cup.
5. Calculate unit weight by dividing the weight of the grout by the volume of the cup.

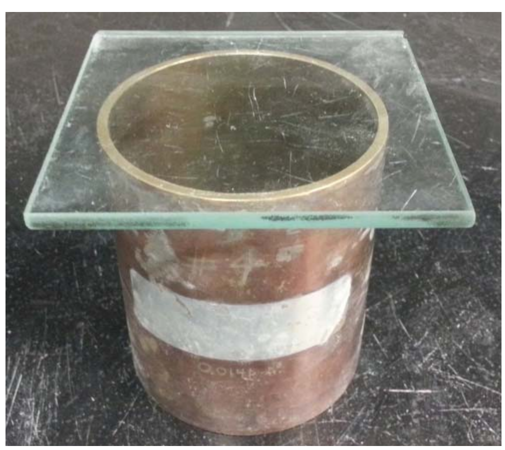


Figure 36—Unit weight measure

12 Results and Discussion - High-Temperature Grout Fluidity Test

The HTGF test was conducted on grouts PT1-2, PT2-2, PT3-1, and PT4-1. The PT grouts were tested at both the upper limit and median point of the manufacturer's water content specifications (see Section 7.1). The results obtained are presented in the following sections.

12.1 Flow Cone Test Results

The variation in flow cone efflux time with respect to recirculation time for all tests is shown in Figure 37.

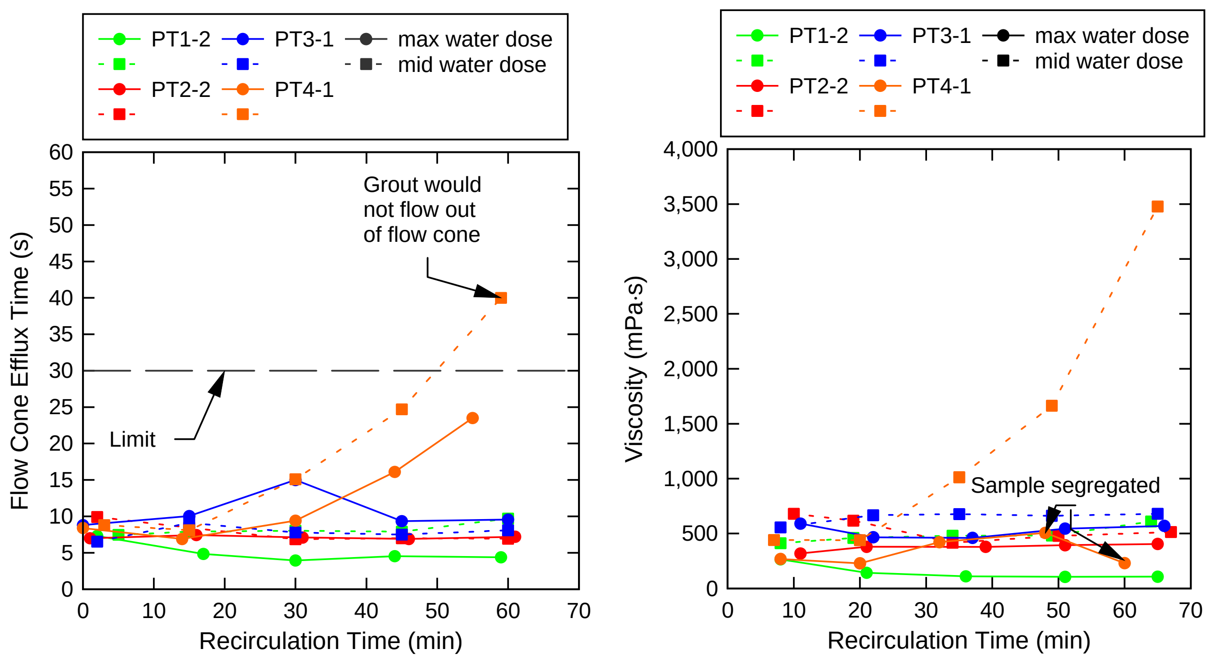


Figure 37—Results of flow cone tests conducted during HTGF. The uncertainty is estimated to 0.88 s (ASTM C939)

With the exception of grout PT4-1, the PT grouts maintained flow cone efflux times of less than 15 s with the majority of the measurements below 10 s. Grout PT3-1 at maximum water dosage exhibited a single high flow cone efflux time at the 30-min sampling time but then returned to less than 15 s for the subsequent two tests. Due to the shear-thinning and thixotropic properties of grout PT3-1, the time between collecting a sample and conducting the flow cone test had a major impact on the measured flow cone efflux time. This is likely the source of the single high flow cone efflux time in grout PT3-1 at maximum water dosage. Grout PT4-1 showed an increasing trend for both maximum and mid-range water addition rates, with the mid-range water dosage resulting in flow time higher than 30 s, which exceeds the limits of the HTGF test. The last sample taken from PT4-1 during the mid-range water dosage test would not flow out of the flow cone, resulting in a failed test.

12.2 NSR Viscosity Test Results

NSR Viscosity Tests were conducted every 15 min during the recirculation period. Variation in viscosity with respect to recirculation time for all of the samples tested is shown in Figure 38.

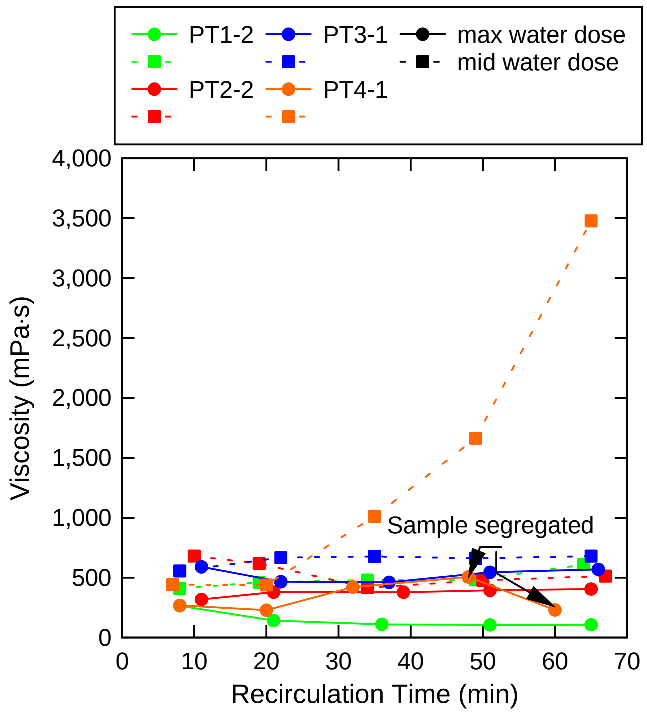
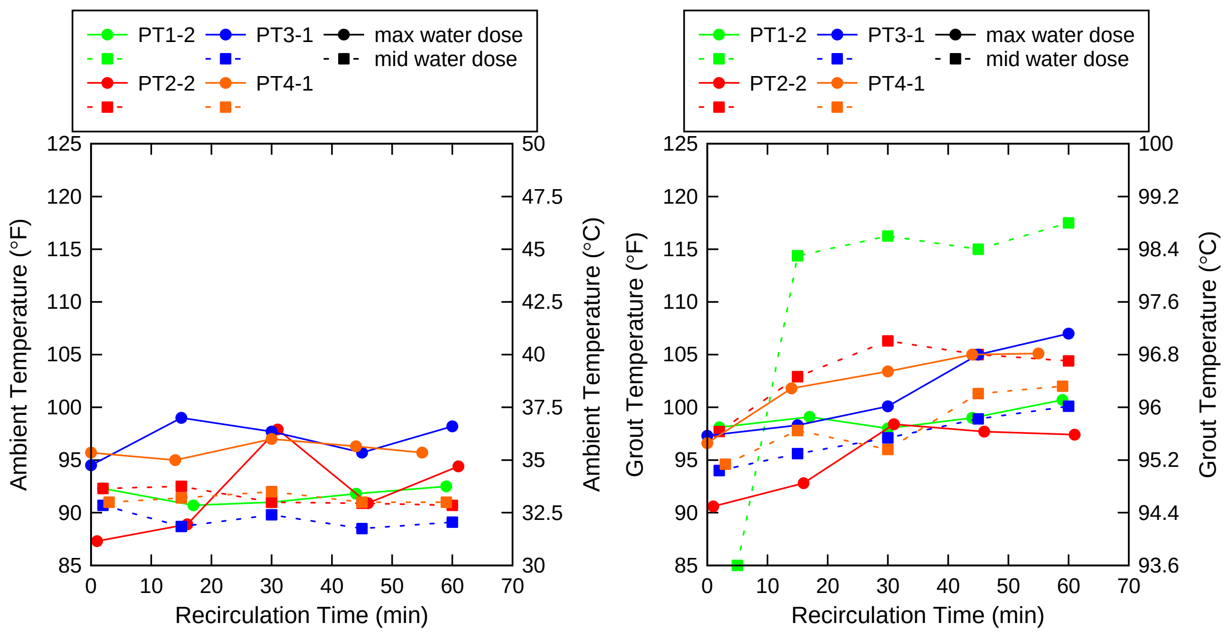


Figure 38—Results of NSR viscosity tests conducted during HTGF. The uncertainty is discussed in section 9.2.3

The viscosity curves display a similar trend to the flow cone curves for each grout in nearly every case. Grouts PT1-2, PT2-2, and PT3-1 each exhibited relatively low and constant apparent viscosities during the entire recirculation period at both their maximum and mid-range water dosage. These results are similar to the flow cone test results. At mid-range water dosage, grout PT4-1 exhibited a significant increase in viscosity over the one hour testing period, as it did during the flow cone test. When grout PT4-1 was tested at its maximum water dosage, issues were noted when conducting the DSR test at the 45-min and 60-min sampling times. The samples loaded into the DSR cup segregated and showed bleeding, resulting in the measurement of a very low viscosity. Therefore, the data measured at the 45-min and 60-min sampling points on grout PT4-1 at maximum water content are not considered valid.

12.3 Temperature Results

The ambient temperature and grout temperature were measured every time a sample was collected during the HTGF test. Figure 39 contains a plot showing the variation in ambient temperature and grout temperature with respect to recirculation time for each HTGF test. Table 6 contains a summary of the ambient temperatures, grout temperatures, and difference between grout temperature and ambient temperature that were measured during the HTGF test.



(a) (b)

Figure 39—Ambient temperature and (a) grout temperature (b) recorded during HTGF test. These are single measurements, but the uncertainty could be estimated ± 1 °F (0.5 °C) from the instrument used to measure the temperature.

The target ambient temperature was 90°F (32.2°C). Several of the batches exhibited a higher ambient temperature because it was difficult to precisely control the temperature of the environmental chambers used for testing. In all cases, the ambient temperature was fairly constant throughout the testing period.

Although the test is intended to be conducted at an ambient temperature of 90°F (32.2°C), the temperatures of the grouts were on average 7°F (4° C) (Table 6) higher than ambient during the test. When the HTGF test was conducted on grout PT2-2 at mid-range water content, the grout temperature was 13°F (7° C) higher than ambient in one case.

Table 6—Summary of ambient and grout temperatures measured during HTGF test (°F/°C)

Grout	Water Dosage	Ambient Temperature			Grout Temperature			Difference		
		High	Low	Avg	High	Low	Avg	High	Low	Avg
PT1-2	max	93/34	91/33	92/33	101/38	98/37	99/38	8/4	7/4	7/5
	mid	91/33	89/32	89/32	99/37	94/34	98/36	8/4	5/2	9/4
PT2-2	max	98/37	87/31	92/34	98/37	91/33	95/35	0/0	4/2	3/1
	mid	93/34	91/33	91/33	106/41	98/37	103/39	13/7	7/4	12/6
PT3-1	max	99/37	95/35	97/36	107/42	97/36	102/39	8/5	2/1	5/3
	mid	91/33	89/32	89/32	100/38	94/34	97/36	9/5	5/2	8/4
PT4-1	max	97/36	95/35	96/36	105/41	97/36	102/38	8/5	2/1	6/2
	mid	92/33	91/33	91/33	102/39	95/35	98/37	10/6	4/2	7/4

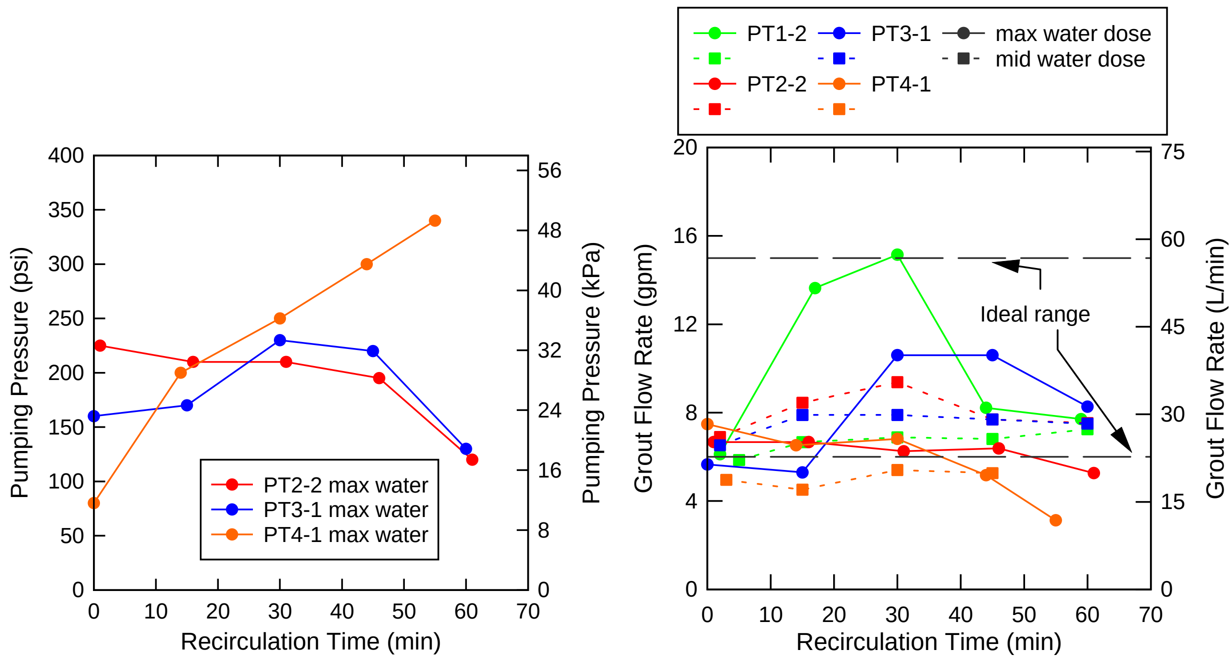
12.4 Pressure and Flow Rate Results

At the sampling intervals during grout recirculation, grout pressure at the pump outlet and grout flow rate at the discharge end of the 400 ft (122 m) recirculation hose were recorded. Figure 40 shows the variation in pumping pressure and grout flow rate during the course of recirculation. Note that the pumping pressure was not recorded during all of the HTGF tests.

During testing, the flow rate was adjusted to stay within the limits set forth by PTI (Post-Tensioning Institute 2012). These limits are indicated in Figure 40 (b). The pumping pressure was adjusted to increase or decrease the flow rate of the grout through the hose.

In nearly all cases, the flow rate of the grout through the hose was within the limits set forth by PTI. There is one exception with maximum water dosage of grout PT4-1, very high pumping pressures were used to attempt to maintain the flow rate. However, due to the high viscosity of the grout, it was not possible to maintain the flow rate within PTI's limits.

FDOT specifications indicate that normal pumping pressure should be between 10 psi (479 Pa) and 50 psi (2384 Pa) (FDOT 2002) while PTI states that normal pumping pressures should be less than 145 psi (Post-Tensioning Institute 2012). The grouts tested during the HTGF test required much higher pressures in each case where the pressure was recorded. Although no pressure data were recorded during PT4-1 testing at its mid-range water content, the grout had stiffened such that the grout could not be pumped out of the hose during cleanup.



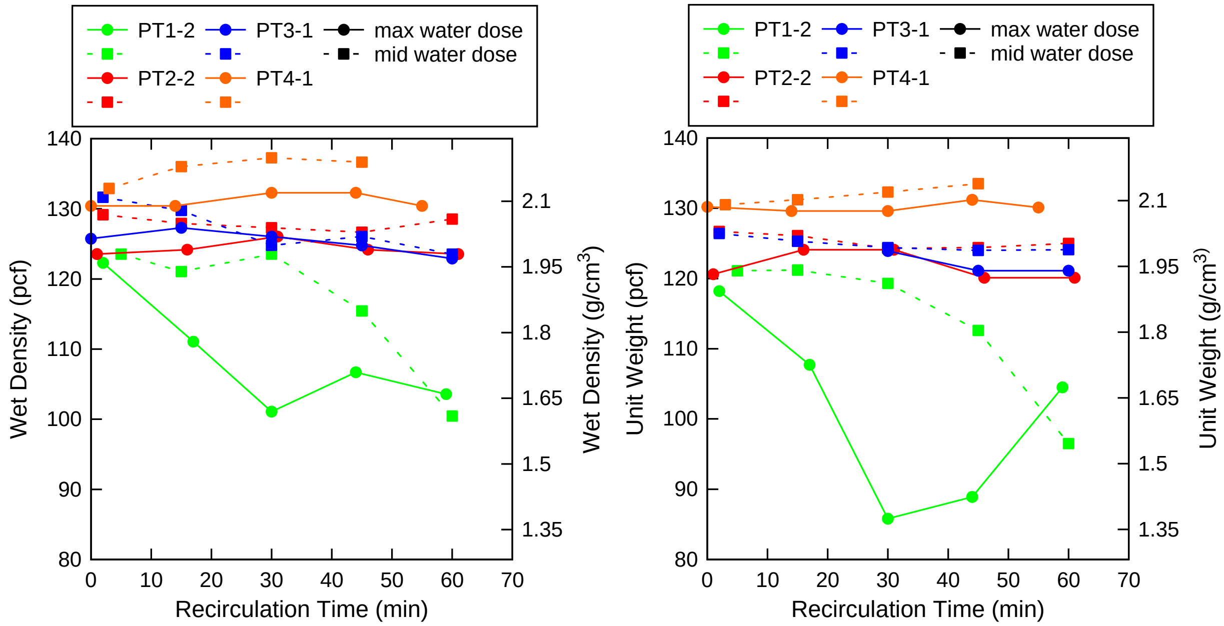
(a) (b)

Figure 40—Variation in pumping pressure (a) and grout flow rate (b) during HTGF test. These are single measurements, but the uncertainty could be estimated at 5 psi (MPa)

12.5 Wet density and Unit Weight Results

The wet density and unit weight tests were used to determine the density of the grouts during the HTGF test. Figure 41 shows the wet density and unit weight values of the grout taken during the regular sampling intervals. Table 7 provides a summary of the initial unit weight as well as the changes in unit weight during the recirculation period.

With the exception of grout PT1-2, all of the grouts exhibit a fairly constant density as measured by both the wet density test and the unit weight test. The maximum increase or decrease in unit weight during the tests on these grouts was 6.1%, which was measured using the wet density on grout PT3-1 at mid-range water content. Grout PT1-2, however, exhibited foaming during the recirculation period, which was accompanied by a decrease in unit weight between 17% and 27%, depending on the measurement instrument.



(a) (b)

Figure 41—Variation (a) wet density and (b) unit weight during HTGF test. These are single measurements, but the uncertainty could be estimated at 1 pcf (0. 016 g/cm^3) from previous measurements not reported here.

Table 7—Summary of initial unit weights and associated variation during HTGF test (pcf). The conversion factor to 1pcf is equal to 0.016 g/cm^3.

Grout	Water	Wet Density			Unit Weight		
		Initial	Increase	Decrease	Initial	Increase	Decrease
PT1-2	Max	122.3	-	21.2	118.2	-	32.4
	Mid	123.6	-	23.1	121.1	0.1	24.6
PT2-2	Max	123.6	2.5	0.0	120.6	3.5	0.5
	Mid	129.2	-	2.5	126.7	-	2.4
PT3-1	Max	125.7	1.6	2.8	123.9	-	2.8
	Mid	131.7	-	8.1	126.4	-	2.4
PT4-1	Max	130.4	1.9	-	130.2	1.0	0.6
	Mid	132.9	4.4	-	130.5	3.0	-

13 Performance Classification using NSR Viscosity and Flow Cone

Concurrent flow cone and viscosity tests were conducted at 15 min intervals during HTGF testing. Figure 42 compares the results of the concurrent viscosity and flow cone efflux times. Linear regression of the data, producing an R^2 value of 0.84 was calculated after excluding data from three tests. Two tests of PT4-1 were excluded; one was because the DSR samples contained excessive water and the other was because the grout did not flow out of the flow cone. A sample of grout PT3-1 at maximum water content was also excluded because of the excessively thixotropic nature of the grout, which resulted in an unusually high flow cone efflux time.

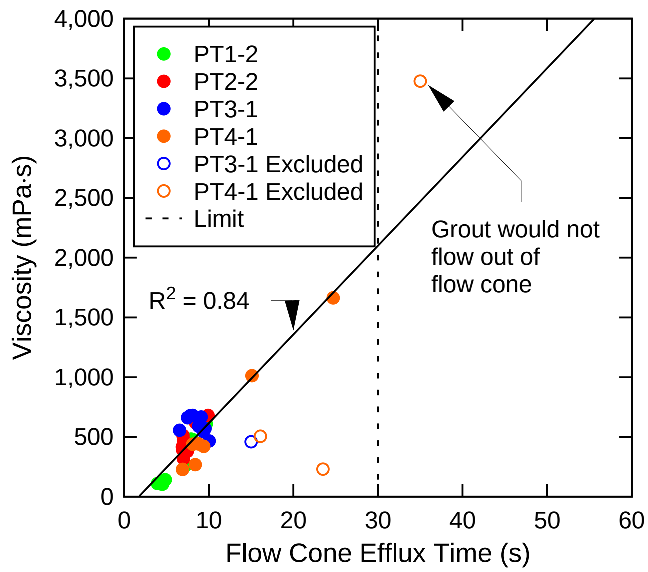


Figure 42—Comparison of NSR viscosity and flow cone efflux time for samples collected during HTGF test. The uncertainty is discussed in section 9.2.3 for the viscosity and 0.88 s for the flow cone (ASTM C939)

Based on these data, a flow cone efflux time of 30 s corresponds to a viscosity of approximately 2,000 mPa·s. Little data, however, were recorded for flow cone efflux times above 12 s. All of the PT grouts tested, with the exception of grout PT4-1, exhibited flow cone efflux times throughout the HTGF test lower than 12 s. Grout PT4-1 exhibited higher flow cone efflux times, but only two HTGF tests were conducted on each grout, thus yielding only a few points.

Another option would be to use the kinematic viscosity, which is the dynamic viscosity divided by the density. This would account for the fact that the flow rate from the flow cone is controlled in part by the density of the mixture. While the density of PT1-2 varies significantly during the recirculation (figure 42), this is not typical behavior and the remainder of the PT grouts appear to have similar unit weights. Furthermore, it is not necessarily practical for replacement of the HTGF test since the unit weight would not be known.

During the HTGF test, one sample of grout PT4-1 was collected that had a flow cone efflux time of about 25 s and a viscosity of about 1,500 mPa·s. The grout sampled at this point could still be pumped by the grout plant, and passed the HTGF test. In general, the PT grouts exhibited a viscosity lower than 750 mPa·s corresponding to flow cone efflux times of less than 12 s in the HTGF test. These demarcations are summarized and divided into performance categories in Table 8. Viscosity test results recorded during the HTGF test are given in Figure 43.

Table 8—Calibration of viscosity test to flow cone results with performance classification

Viscosity (mPa·s)	Efflux time (s)	Performance Classification
< 750	< 12	Moderate pumping pressure
750 to 1,500	12 to 25	Uncertain
> 1,500	> 25	Excessive pumping pressure possible

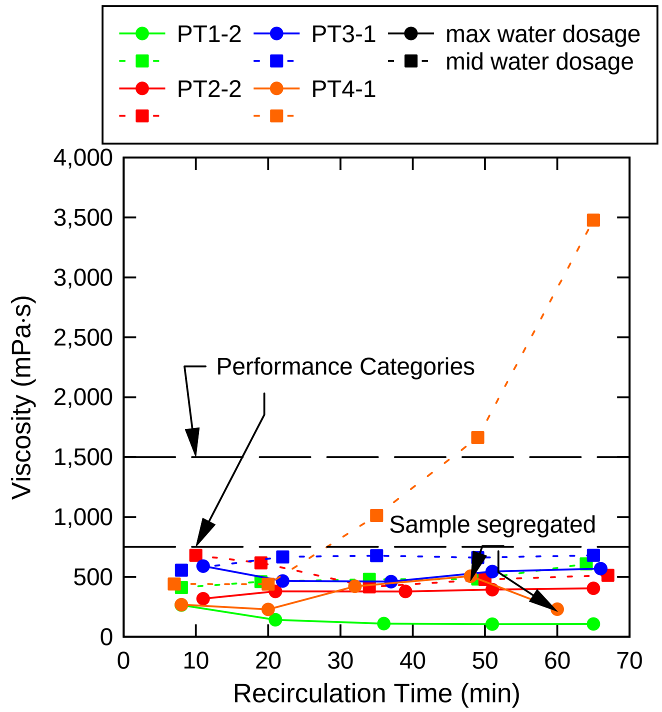


Figure 43—NSR viscosity results from HTGF test. The uncertainty is discussed in section 9.2.3

With the exception of the tests on grout PT4-1 at mid-range water content, all of the grouts exhibited acceptable pumping performance as indicated by the categories shown in Table 8 and had viscosity less than 750 mPa·s throughout the HTGF 60 min recirculation period. Measured apparent viscosities on PT4-1 (mid water dosage), however, exceeded 1,500 mPa·s during the 60 min recirculation period. Pressure was not measured during this test, but the grout stiffened to such an extent by the end of the test that it was impossible to clear the hoses. Further, PT4-1 (max water dosage) exhibited pumping pressures near 350 psi (17 kPa) during the final 15 min of the HTGF test, well above those of the other measured pressures.

14 Grout Conditioning Technique

The HTGF test imposed a dynamic mechanical shearing action on the grout nearly constantly during the 60 min test period, during which time the ambient temperature was maintained at 90°F (32.2°C). To further simplify the test procedures, a simulated conditioning technique was needed to obviate the need for a full-scale grout plant and associated equipment. The method must maintain a specified elevated temperature and impart shear either periodically or continuously throughout the 60 min conditioning period.

14.1 Temperature

The mixing and conditioning process imposed on the grout by the HTGF test caused the grout temperature to increase approximately 7°F (4°C) to 13°F (7°C) higher than the ambient temperature of 90°F (32.2°C). To better quantify the effect of temperature on the viscosity of both plain and PT grouts, NSR viscosity tests were conducted on both plain and PT grouts over a range of temperatures. No conditioning was performed between tests; the grout was allowed to remain at rest in a vacuum-sealed container.

Figure 44 contains the results of the viscosity test at temperatures between 75 °F (23.8 °C) and 120 °F (49 °C) for grout C45, Figure 45 contains similar results for grout PT1-1, and Figure 46 contains similar results for grout PT2-1. Note that the scale on Figure 44 is greater than that on Figure 45 and Figure 46 by an order of magnitude.

Generally, the viscosity of the plain grouts and the PT grouts increase over the course of the one hour testing period at any temperature. As the temperature is increased, the plain grouts see an increase in viscosity as well. The viscosity of the PT grouts, however, decreases as the temperature increases. These results indicate that an increase in temperature has a much larger effect on the viscosity of the plain cement grouts than it does on the viscosity of the PT grouts.

Based on the results of these tests and the measured temperatures during the HTGF, conditioning temperatures for the remainder of the testing was set to 100 °F (38 °C).

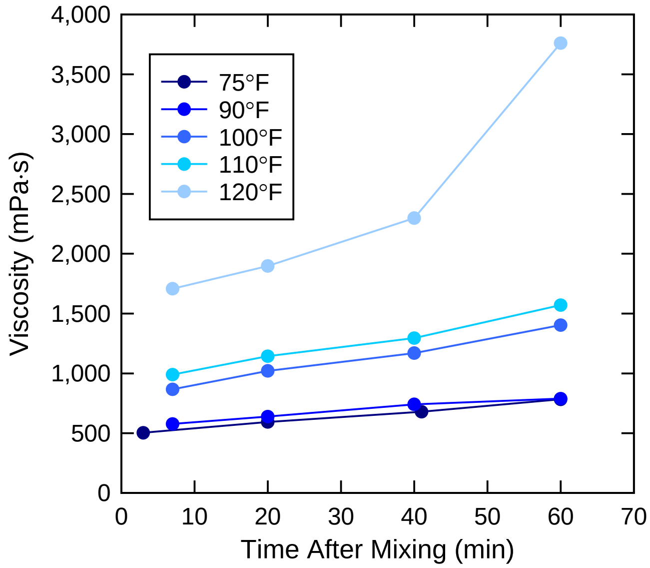


Figure 44—Variation of NSR viscosity over time grout C45 at varying temperatures with no conditioning between tests. The uncertainty is discussed in section 9.2.3

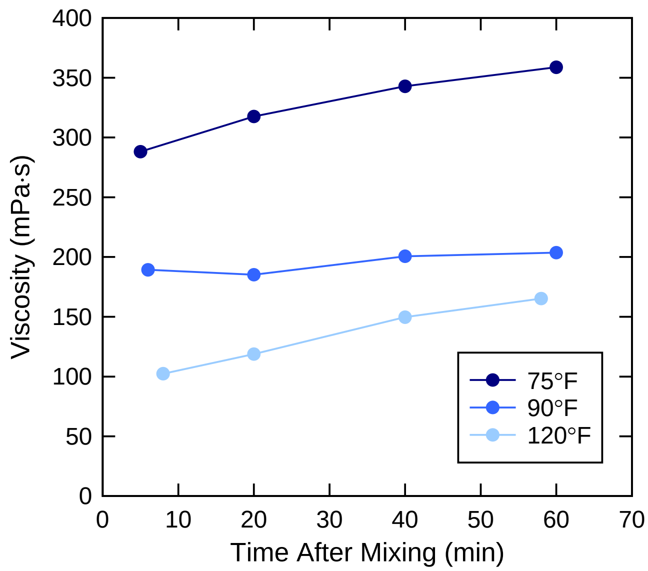


Figure 45— Variation of NSR viscosity over time grout PT1-1 at varying temperatures with no conditioning between tests. The uncertainty is discussed in section 9.2.3

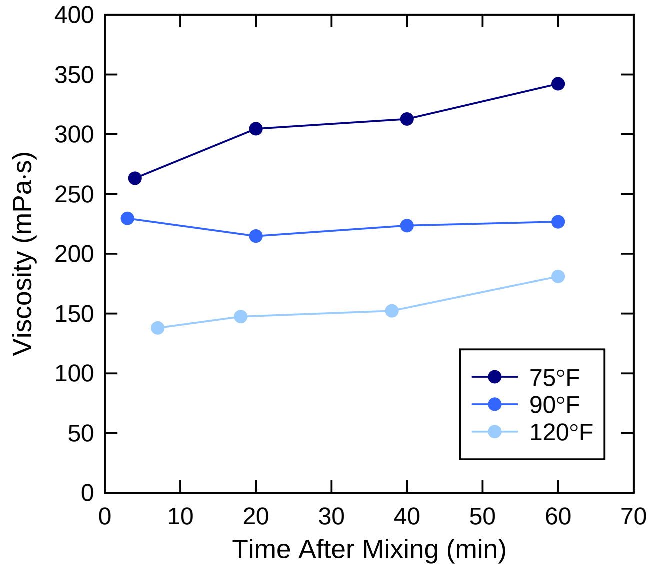


Figure 46—Plot of viscosity vs. time after mixing for grout PT2-1 at different temperatures. The uncertainty is discussed in section 9.2.3

14.2 Mixing

A series of tests were conducted on all of the PT grouts in which the DSR was used to condition and test the grout. Essentially, the viscosity test was conducted on a grout sample continuously for 1 h. The DSR had a temperature control system to ensure a stable temperature during testing. The procedure used for the test in which the DSR conditioned the grout is as follows:

1. Mix grout according to the mix procedure in high-shear mixer with water bath set to 100 °F (38 °C). The grout materials are stored at room temperature.
2. Set DSR temperature control to 100 °F (38 °C)
3. Load a sample of grout into the DSR.
4. Subject the grout to pre-shear rate of 165 s^{-1} for 30 s.
5. Subject the grout to a shear rate of 50 s^{-1} for a period of 60 min, sampling every 1 s.
6. Plot the viscosity at the following times: 1 s, 15 min, 30 min, 45 min, and 60 min.
7. Clean the grout out of the cup and ribbon.

A comparison of the viscosity measured using the DSR for grout conditioning versus that measured when the HTGF test was used for conditioning is shown in Figure 47. Samples collected at the same mixing water content and times after mixing are compared.

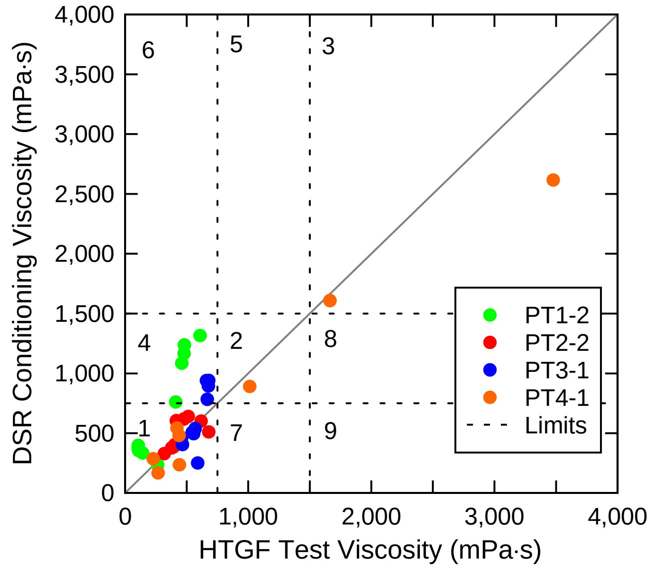


Figure 47—Comparison of NSR viscosity measured when DSR was used for grout conditioning versus NSR viscosity measured on specimens from HTGF test. The uncertainty is discussed in section 9.2.3

The plot is divided into nine zones, which correspond to the performance categories developed in Chapter 13. Table 9 provides a summary of the number of samples contained in each zone along with the implications of each zone when using the DSR is used to condition the grout instead of the grout plant in the HTGF test. In a neutral zone, both conditioning techniques result in the same performance outcome. In a conservative zone, the grout conditioned with HTGF is more likely to have a favorable performance category than the grout conditioned by the DSR. In a non-conservative zone, the grout conditioned with DSR is more likely to have a favorable performance category, which represents 3% of the tests conducted for this research.

Zones 1-3 represent a neutral result in which the same qualifying condition is met when either conditioning technique is used. 80% of the samples shown in Figure 47 fall within a neutral zone. This is a good indication that the DSR conditioning provides very similar results to the HTGF test conditioning.

Twenty percent of the samples are within a conservative zone, zone 4. This zone represents areas of uncertainty due to the lack of data. While the results from DSR conditioning indicated excessive pumping pressures, the test results for grout conditioned with the grout plant indicated moderate pumping pressures.

Table 9—Comparison of performance under different conditioning techniques

Zone	Conditioning		Samples	Percentage	Status
	HTGF	DSR			
1	M	M	27	71%	Neutral
2	U	U	1	3%	
3	P	P	2	6%	
4	M	U	8	20%	Conservative
5	U	P	0	-	
6	M	P	0	-	
7	U	M	0	-	Non-conservative
8	P	U	0	-	
9	P	M	0	-	

M–moderate pumping pressure
U–uncertain
P–excessive pumping pressure possible

None of the samples are in a non-conservative zone, zone 8. In this case, the DSR conditioning indicates uncertain performance while the HTGF conditioning gave excessive pumping pressures.

Zones 6 and 9 are the least desirable zones. None of the samples fell within either of these zones.

While conducting the NSR viscosity test continuously for 60 min, some segregation was observed in the grout sample in the cup. A photograph of this segregation is shown in Figure 48.

Figure 48—Segregation observed in PT grout during 60-min NSR viscosity test

Although grout segregation occurs during this test, the results presented in Table 9 indicate that the 60-min NSR viscosity test provides a good simulation of the HTGF test. The grout segregation can therefore be disregarded. Overall, the DSR conditioning provided a suitable alternative to the HTGF test for evaluating the performance of PT grout.

15 University of Minnesota Results

Testing was conducted at the University of Minnesota at Duluth (UMD) to compare the results of viscosity tests conducted on identical DSR equipment but in different laboratories.

15.1 Materials and Equipment

Six commercially available prepackaged grouts were V-blended and shipped to the UMD for NSR viscosity testing. V-blended samples were also shipped to University of Florida (UF) as well to compare both viscosity test results from the same grout lots at different laboratories using similar testing equipment. A summary of the grouts tested are shown in Table 10.

Table 10—Grouts tested at UMD and UF

Grout
PT1-4
PT2-4
PT3-3
PT4-6
PT5-1
PT6-1

TA AR2000ex Dynamic Shear Rheometer with the stainless steel cup and helical ribbon geometry illustrated in Figure 20 were used in both UMD and UF testing. In addition, mixing sequence RH-185 and similar mixing equipment were used.

15.2 Standard Reference Material Testing

The DSR at UMD was used to test the NIST SRM 2492 in accordance with the procedures given in Section 9.2. The calibration was conducted with the shear stress factor set to F_σ = 20,480 m^{-3}. Results of the calibration testing are shown in Table 11. The adjustment factors A and B were determined by averaging the three yield stress and three viscosity ratios in Table 11, which resulted in average adjustment factors of A = 1.62 and B = 1.75 (from Equation 4 and Equation 5). These factors were used to calibrate UMD data presented in this chapter.

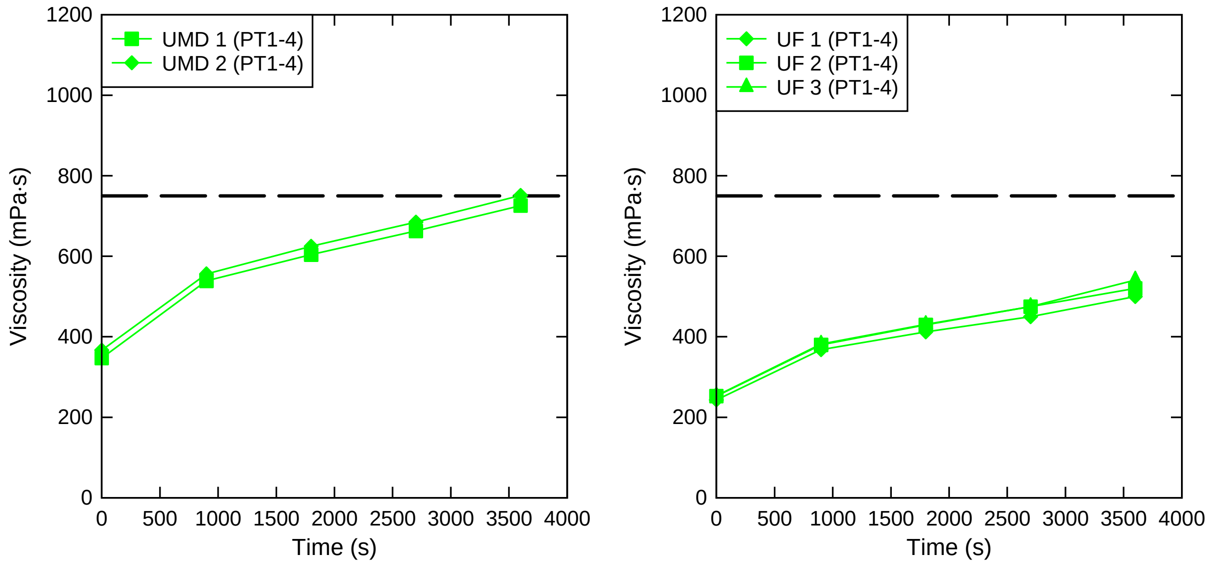
Table 11—Summary of measured Bingham standard material values at UMD. These are single measurements, but the uncertainty could be estimated at 10%

Measured Values					
Yield Stress (Pa)			Plastic Viscosity (Pa·s)		
Age (d)	Value	Ratio	Age (d)	Value	Ratio
1	16.50	1.53	0	4.07	1.75
3	13.77	1.50	3	4.41	1.54
7	11.10	1.84	7	3.65	1.98

15.3 Results and Discussion

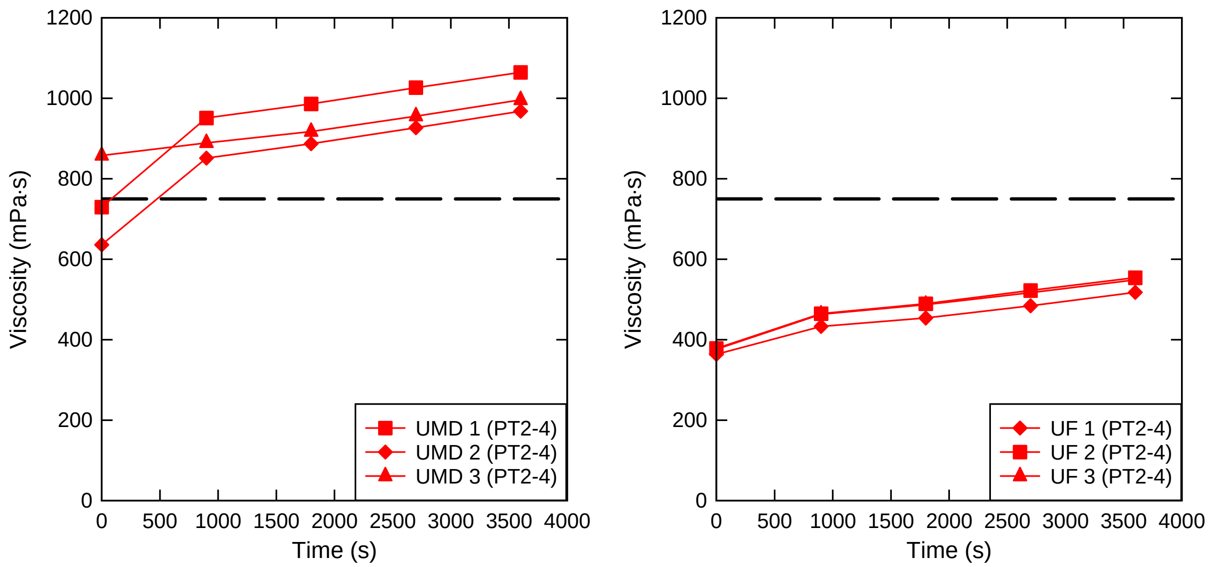
Companion NSR viscosity tests were conducted by mixing the grout in accordance with the test procedure, then sampling viscosity at 15 min intervals. Figure 49 through Figure 54 compare the results of laboratory testing at UMD and UF for each of the six grouts tested. Detailed results are located in the appendix. In general, the trends in viscosity growth are comparable between the tests, with the exception of the final reading in PT6-1, where the final values at UMD are an order of magnitude higher than that measured at UF. The absolute value of the viscosity, however, varied between laboratories. The differences in results indicated that the absolute magnitude of the results were sensitive to the speed and handling of the mixer, water content, and conditioning temperature. Additionally, if the calibration is done improperly, or the calibration material has degraded in some way, the magnitudes will not correspond between different laboratories.

One other possible source for the differences is a leaky mixer, which is difficult to detect during mixing. Leakage (or even spilling) of small quantities of water will cause undesired variations in grout viscosity.



(a) (b)

Figure 49—Results of NSR viscosity tests on PT1-4 at (a) UMD and (b) UF. The uncertainty is discussed in section 9.2.3



(a) (b)

Figure 50—Results of NSR viscosity tests on PT2-4 at (a) UMD and (b) UF. The uncertainty is discussed in section 9.2.3

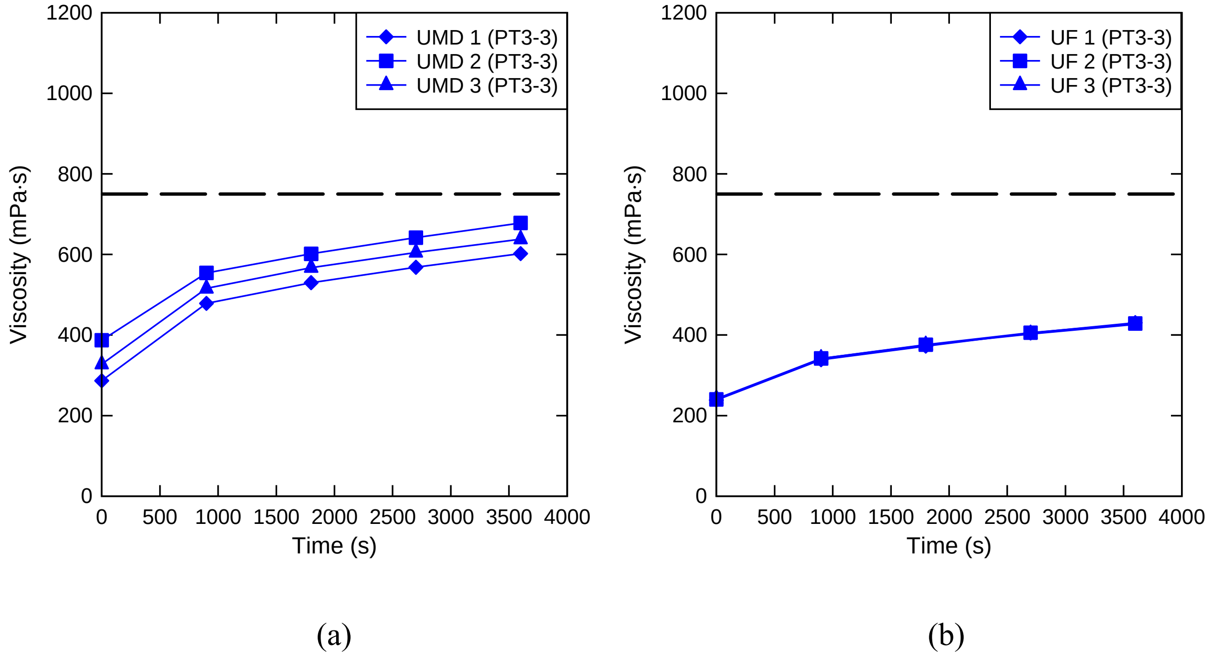


Figure 51—Results of NSR viscosity tests on PT3-3 at (a) UMD and (b) UF. The uncertainty is discussed in section 9.2.3

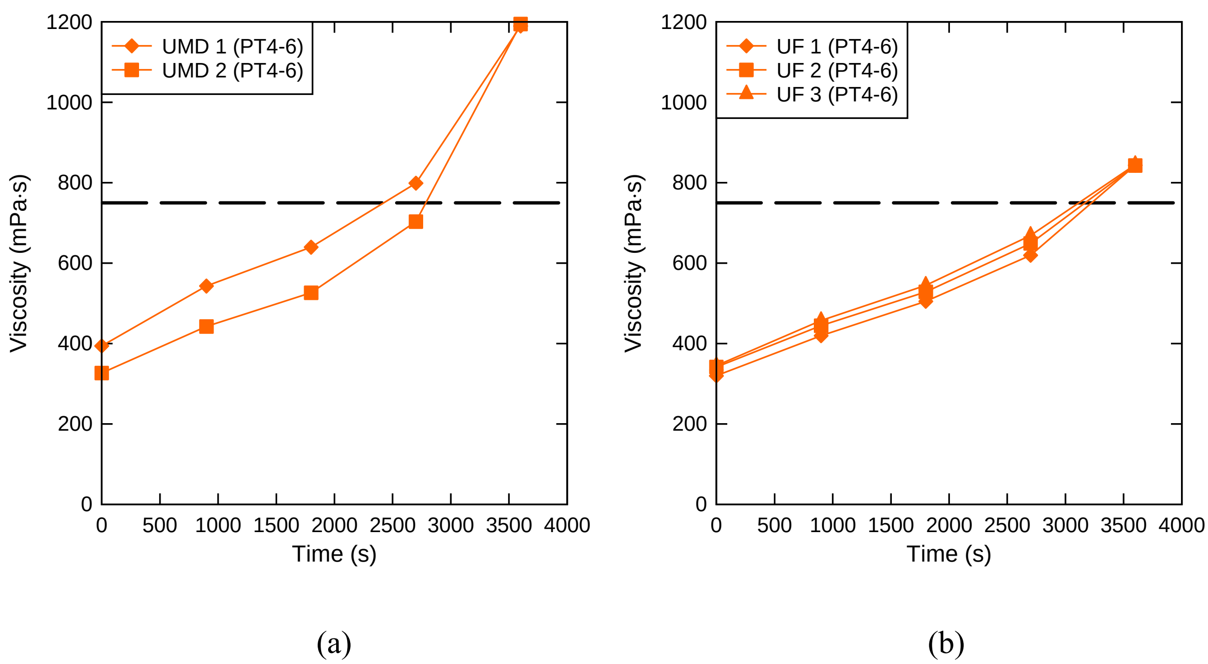
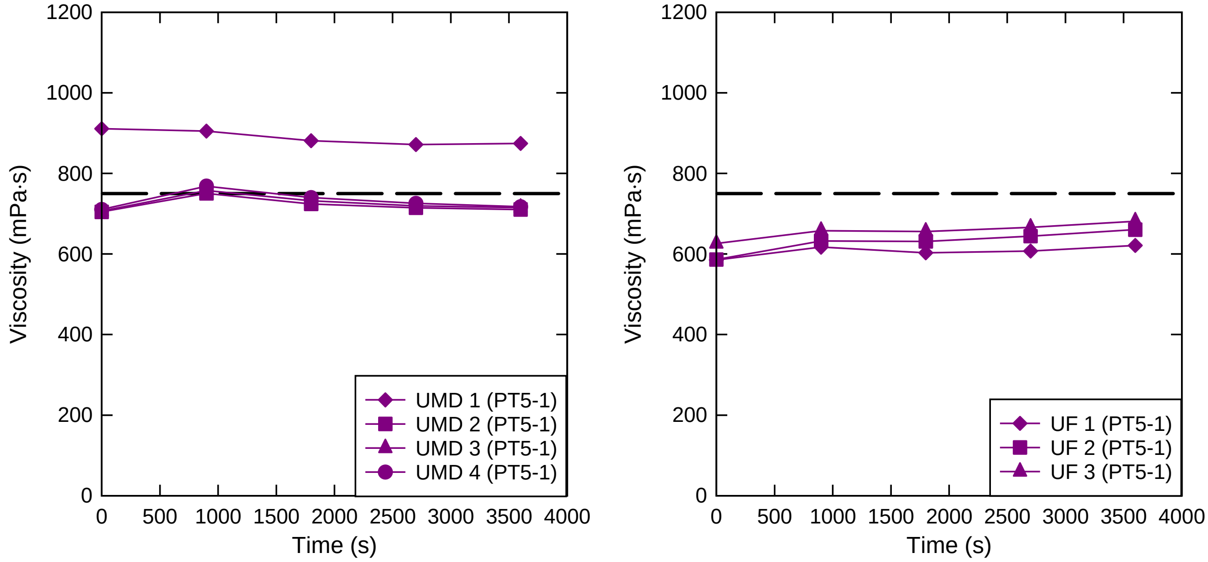
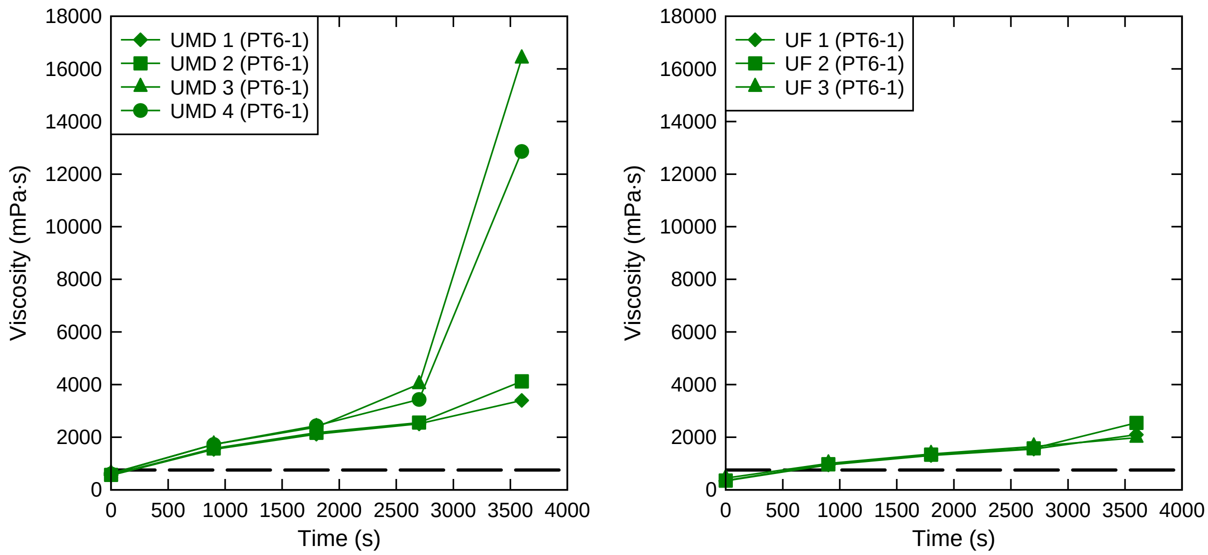


Figure 52—Results of NSR viscosity tests on PT4-6 at (a) UMD and (b) UF. The uncertainty is discussed in section 9.2.3



(a) (b)

Figure 53—Results of NSR viscosity tests on PT5-1 at (a) UMD and (b) UF. The uncertainty is discussed in section 9.2.3



(a) (b)

Figure 54—Results of NSR viscosity tests on PT6-1 at (a) UMD and (b) UF. The uncertainty is discussed in section 9.2.3

Variations between laboratories make uniform evaluation of grout using the DSR a challenge. All of the grouts tested at UMD, with the exception of PT4, however, would have been placed in the same performance category (Chapter 13) using the results from either laboratory. PT grout behavior during the course of the 60 min test results in an increase in viscosity with time. Larger increases in viscosity over this period indicate a reduction in grout performance. This increase can be evaluated by calculating the initial relative viscosity factor (RV_i) as follows:

$$RV_i = \frac{viscosity_{60}}{viscosity_0} \qquad \text{Equation 9}$$

where RV_i is the 60-min viscosity divided by the initial viscosity. This approach removes the calibration factor from the computation and should provide a more uniform comparison between laboratories. Figure 55 shows RV_i for the PT grouts tested at both UF and UMD. The factors vary considerably between six PT grouts from near 1.0 to well beyond 4.0, with no distinction among the grouts classified as moderate.

Alternatively, the final relative viscosity factor (RV_f) can be calculated as follows:

$$RV_f = \frac{viscosity_{60}}{viscosity_{45}} \qquad \text{Equation 10}$$

where RV_f is the 60-min viscosity divided by the 45-min viscosity.

Figure 56 shows RV_f for the PT grouts tested at both UF and UMD. With the exception of PT4-6 and PT6-1, the tested grouts have an overall average final relative viscosity factor that is approximately 1.06 with a Coefficient of variation (COV) of 3.6%. PT4-6 and PT6-1, however, have factors that are significantly greater than those of the PT grouts with moderate classification, each having an average of approximately 1.4.

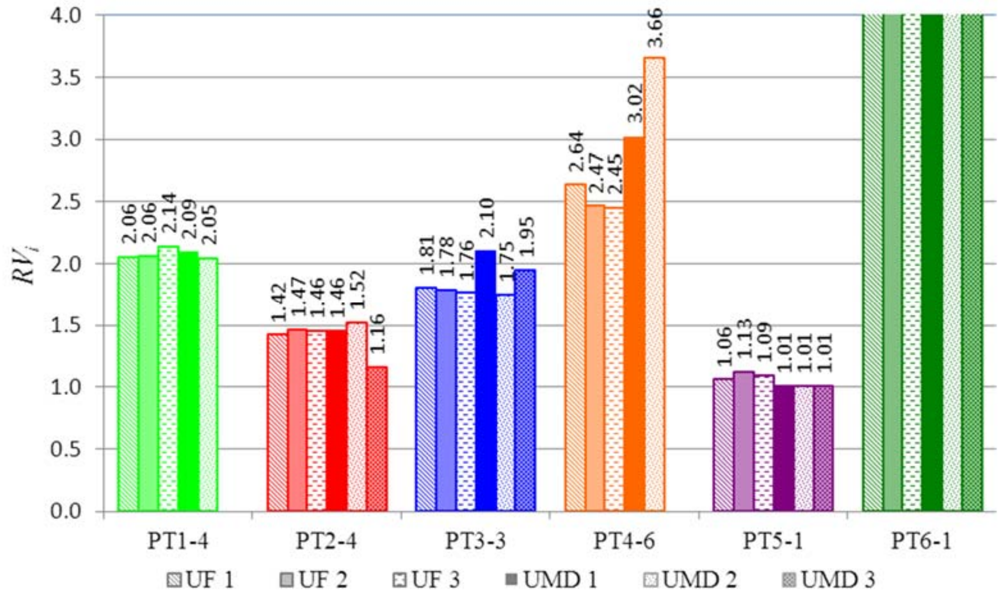


Figure 55—Comparison of RV_i from UF and UMD.

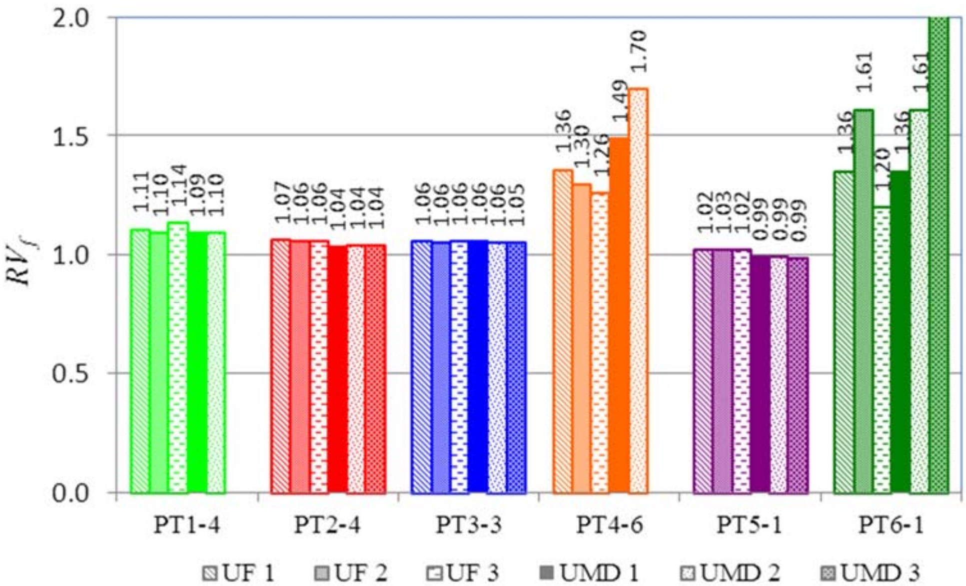


Figure 56—Comparison of RV_f from UF and UMD

If the average of 1.06 plus approximately two standard deviations is used to provide a demarcation between moderate performance and unclear performance this would result in a limit of 1.15. Clearly a ratio greater than 1.5 would indicate a possible problem with pumping pressures near the end of the test period as indicated by the efflux time exceeding the recommended value by the PTI procedure. Table 12 shows the performance categories based on the testing conducted at UF and UMD.

Table 12—Calibration of relative viscosity factor with performance classification

RV_f	Performance Classification
<1.15	Moderate pumping pressure
1.15 to 1.5	Uncertain
>1.5	Excessive pumping pressure possible

16 NIST Results

Testing was conducted at the National Institute of Standards and Technology (NIST) to compare the results of viscosity tests conducted on different DSR equipment but using the same ribbon.

16.1 Materials and Equipment

Four commercially available prepackaged grouts were V-blended and shipped to NIST for viscosity testing (Table 13).

Table 13—Summary of grouts tested at NIST

Grout
PT1-3
PT2-3
PT3-2
PT4-4

Apparent viscosities were measured using the DSR conditioning approach described in Chapter 14. The helical ribbon geometry described in Chapter 9 was fitted with an adapter that allowed its use in the NIST DSR (Figure 58). Tests were conducted at each grout manufacturer's maximum and mid-range specified water content.

A Haake Mars DSR was used at NIST to measure the viscosity of the V-blended grout samples (Figure 57). The helical ribbon geometry used to conduct the UF testing (Figure 19) was adapted to fit the NIST equipment (Figure 58). Use of an adapter necessitated adjustments in the NIST DSR to account for the inertia provided by the additional material. The resulting inertia was measured to be 1.7×10^{-6} kg·m^2.

Figure 57—DSR used for testing at NIST

Figure 58—Helical ribbon from UF testing with adapter mount

The cup used at NIST was slightly larger than the cup used at UF, with a measured inside diameter of 43 mm and a height of 96 mm. Additionally, the gap between the bottom of the cup and the helical ribbon tip was set to 33 mm (instead of the 3.92 mm used at UF). Figure 59 below shows the cup that was used for testing at NIST. The high-shear mixer used at NIST (Figure 60) was slightly different from the one used at UF or UMD. It was fitted with an external temperature control to regulate the temperature of the water that is constantly pumped into the stainless steel jacketed mixing cup. Additionally, the mixing speed was controlled by digital tachometer, which measured the load on the blender blade and adjusted the speed to meet the desired value at set increments in time.

Figure 59—Cup used in NIST DSR testing

Figure 60—High-shear mixer used at NIST

16.2 Standard Reference Material (SRM) Testing

Calibration was conducted on the NIST equipment with the UF ribbon using the SRM2492 to allow comparison with the results of the tests conducted at UF. Due to the limited time available, the reference material was tested on day 1, but not days 3 and 7.

The SRM was tested at a shear rate of 50 sec^{-1}. The resulting Bingham Plastic Viscosity was taken as the average of the viscosities once the rheology of the SRM became constant, and was 0.46 mPa·s. This corresponded to an adjustment factor of 15.41, which was obtained by dividing the certified Bingham Plastic Viscosity at 1 d stated on the NIST certificate by the measured value (7.1 mPa·s/0.46 mPa·s = 15.41).

16.3 Results and Discussion

Companion viscosity tests were conducted by mixing the grout in accordance with the test procedure, then measuring viscosity at 15 min intervals. Figure 61 through Figure 63 compare the results of laboratory testing at NIST and UF for each of the four grouts tested. Detailed results are located in the appendix. Despite the difference in DSR equipment used for this testing, the results compare well. In particular, the viscosity trend over the course of the test match well with all PT grouts. Significant differences, however, were noted in the magnitudes of PT3-2 and PT 4-4. It is unclear why. One possibility is that although the same ribbon geometry was used in the NIST DSR, the cup size was slightly larger, which may have affected the results, as the shear rate would be different.

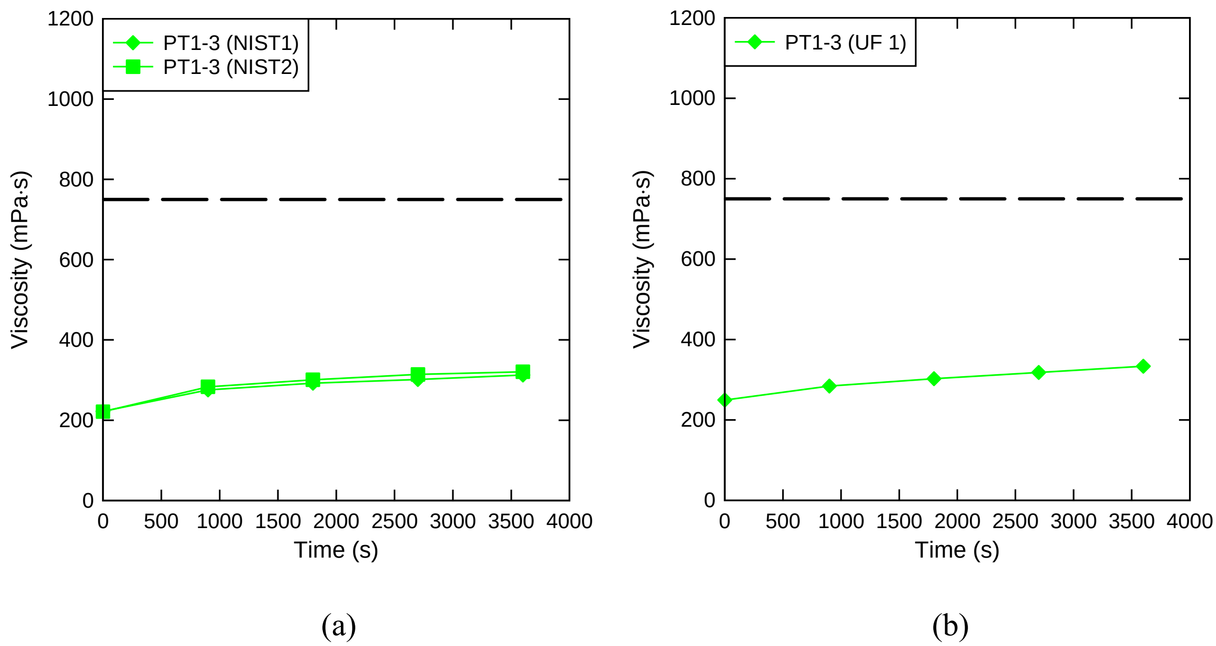
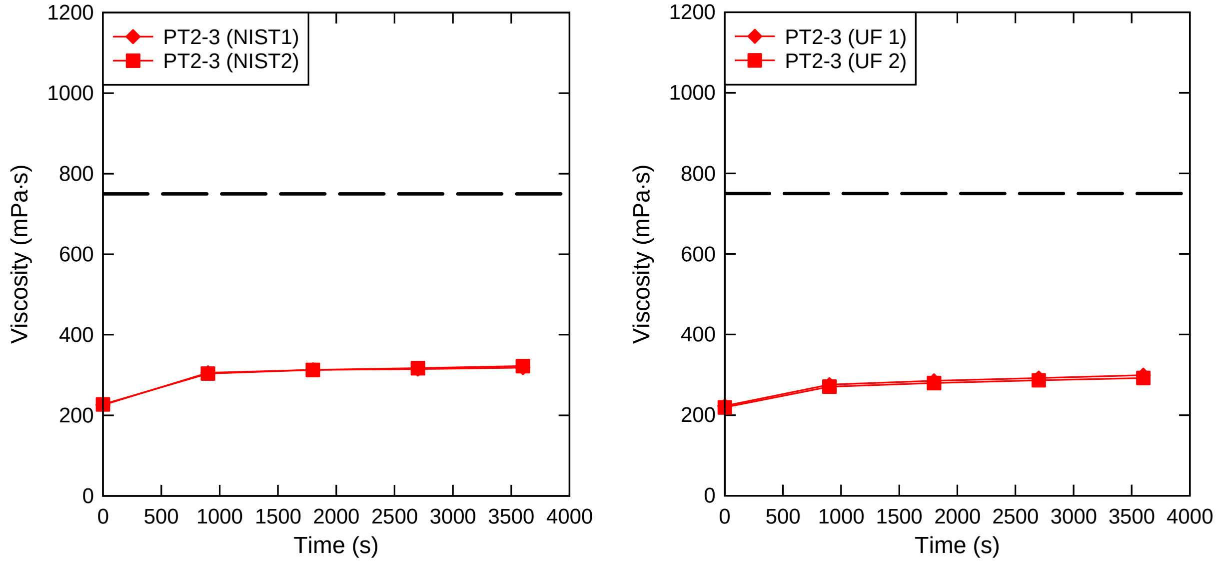
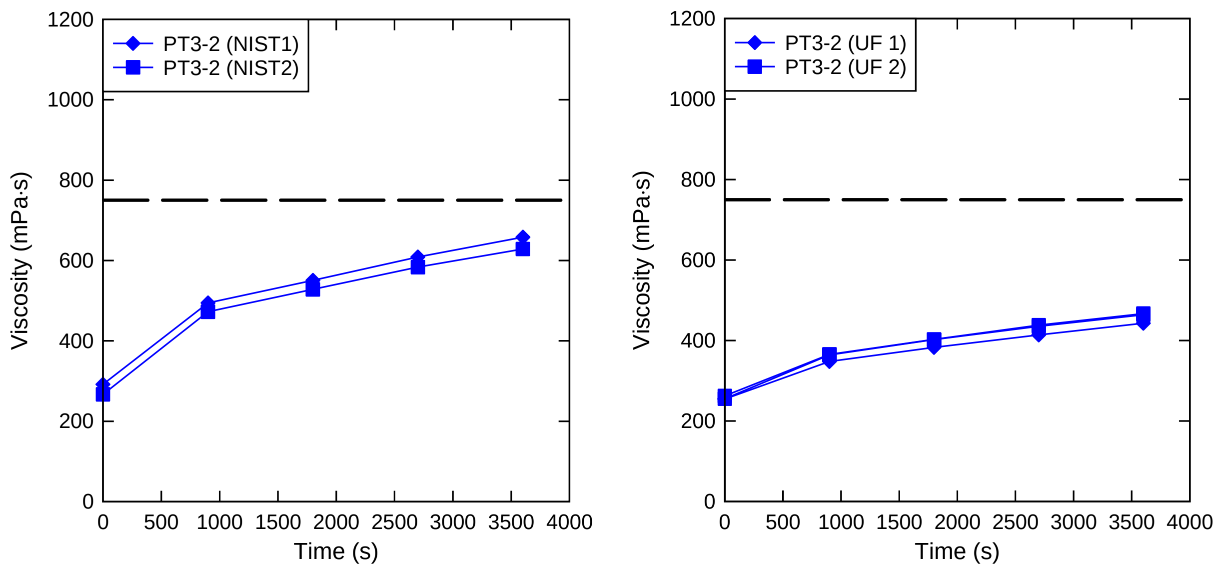


Figure 61—Comparison of results of testing on PT1-3 at (a) NIST and (b) UF. The uncertainty is discussed in Section 9.2.3



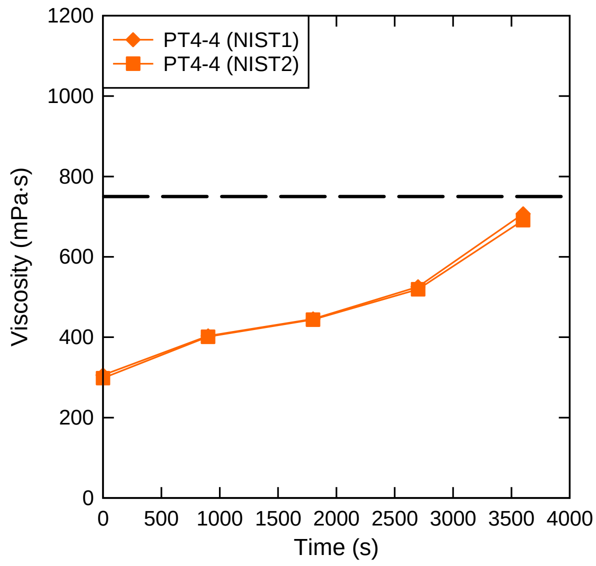
(a) (b)

Figure 62—Comparison of results of testing on PT2-3 at (a) NIST and (b) UF. The uncertainty is discussed in Section 9.2.3



(a) (b)

Figure 63—Comparison of results of testing on PT3-2 at (a) NIST and (b) UF. The uncertainty is discussed in Section 9.2.3



(a) (b)

Figure 64—Comparison of results of testing on PT4-4 at (a) NIST and (b) UF. The uncertainty is discussed in Section 9.2.3

Figure 65 and Figure 66 compares the relative viscosity factors of NIST and UF data for the tested PT grouts. While RV_i varies over the PT grouts tested, RV_f agrees well with the performance categories presented in Chapter 15 (Table 12).

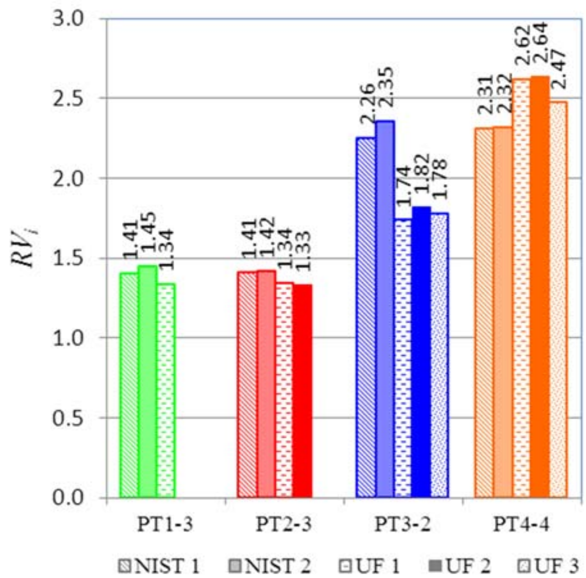


Figure 65—Comparison of RV_i from UF and NIST

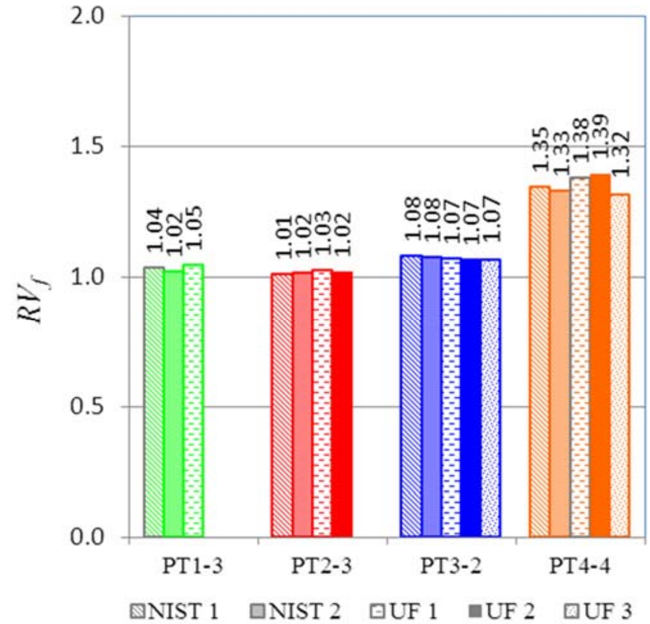


Figure 66—Comparison of RV_f from UF and NIST

17 Summary and Conclusions

Maintaining the fluidity of post-tensioning (PT) grout during high-temperature grouting conditions is an important aspect of post-tensioning bridge construction in Florida. The high-temperature grout fluidity test (HTGF) has traditionally been used to evaluate this fluidity. The HTGF test is conducted by pumping a batch of grout through 400 ft (122 m) of hose in a 90°F (32.2°C) environment continuously for 60 min and periodically taking flow cone efflux time measurements. The goal of this project was to develop a replacement for the HTGF test that utilizes a dynamic shear rheometer (DSR) to measure the rheological properties of PT grout. This new methodology would allow a more rapid and reliable qualification of the PT grouts.

Currently available DSR test methods that were thought to be appropriate for PT grout testing were reviewed. These included the flow curve test, the stress growth test, and the oscillatory time sweep test. None of these tests were found to be adequate to assess the rheological properties of the PT grouts. Instead, the nominal shear rate (NSR) viscosity test was developed to simulate the flow cone measurement in the HTGF test. The shear rate used in the viscosity test ($50\ s^{-1}$) was based on data from the literature and approximately on the calculated shear rate at the nozzle of a flow cone.

In addition to the DSR test development, HTGF tests were conducted twice on four different commercially available PT grouts. While conducting the HTGF test, the following data were recorded: flow cone efflux time, viscosity, ambient temperature, grout temperature, pumping pressure, grout flow rate, wet density, and unit weight. NSR viscosity results measured during the HTGF test were correlated to performance of the PT grouts. In addition, an alternative grout conditioning technique was developed that utilizes the DSR rather than the full scale grout plant. From these data, performance classifications were developed based on the results of the testing.

Finally, DSR testing was conducted at University of Minnesota-Duluth and National Institute of Standards and Technology to provide a comparison of the viscosity results from these laboratories to those produced by the University of Florida. Additional performance classifications were developed based on the findings of these studies.

The following conclusions were made after conducting research:

- PT grout temperatures increased an average of 7° F (4°C) over the course of the circulation during HTGF testing. Line pressures measured at the pump during circulation ranged from 100 psi (4.8 kPa) to 350 psi (16.7 kPa).

- Grout PT1-2 passed the HTGF test, but became aerated during the course of pumping, which resulted in a 27% decrease in unit weight due to the entrained air. DSR conditioning did not reveal this issue.
- Grout PT4-1, failed the HTGF test when mixed at the mid-range of the manufacturer's specified water content. All other PT grouts tested passed the HTGF test.
- The cup and helical ribbon geometry provided the most consistent results when testing PT grouts. Further, the use of cup and ribbon geometry reduced sedimentation and temperature loss sometimes experienced on the parallel plate geometry.
- Plain grout viscosity increased with increasing temperature, while PT1 and PT2 decreased with increasing temperature.
- The viscosity results compared well with flow cone results with an R^2 value of 0.85.
- Trends in viscosity test results compared well with results from both University of Minnesota-Duluth and NIST. Absolute magnitudes, however, varied somewhat. This is thought to be due to the sensitive nature of the mixing and conditioning process.
- A methodology was developed to rank the grouts by their performance and determine the grouts that could be used satisfactorily from the grouts that would fail. The method is summarized in Chapter 18.

18 Test Methods and Performance Classification

This chapter summarizes the test methods and performance classifications developed as part of this research. To conduct test on small samples that could be done in a lab, it is paramount to have a representative sample from a grout bag. This can be achieved by V-blending a bag and obtaining small samples of about 400 g each. The DSR procedure developed to condition the PT grout and evaluate the rheological properties in this research is as follows:

Grout sample preparation

1. V-blend a full bag of grout before sampling. Use high shear blender mixer with water bath set to 100°F (38°C) to mix PT grout. Method as ASTM C1738.
2. Mix full bag of PT grout using a high-shear mixer with water bath set water bath to 100°F (38°C). Mixer must impose a shear rate comparable to a colloidal mixer.

Rheological testing

1. Load a sample of grout into the DSR using the calibrated cup and ribbon geometry.
2. Shear the grout for 30 s at 165 s^{-1} .
3. Shear the grout for 60 min at 50 s^{-1}. Collect measurements of torque at 1 s intervals.
4. Calculate and record the viscosity initially and then at 15 min, 30 min, 45 min, and 60 min.

Compute the final relative viscosity factor RV_f by dividing the viscosity at 60 min by the viscosity at 45 min. If the testing is conducted using the same equipment as was used for UF testing, then the viscosity can be used directly to evaluate the potential pumping pressures (Table 14). For other DSR equipment, RV_f can be used as shown in Table 15.

Table 14—Performance classification for viscosity based on equipment used at UF

Viscosity (mPa·s)	Flow cone (s)	Performance Classification
< 750	< 12	Moderate pumping pressure
750 to 1,500	12 to 25	Uncertain
> 1,500	> 25	Excessive pumping pressure possible

Table 15—Final relative viscosity factor RV_f and performance classification

RV_f	Performance Classification
<1.15	Moderate pumping pressure
1.15 to 1.5	Uncertain
>1.5	Excessive pumping pressure possible

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Appendix A—Nominal Shear Rate (NSR) Viscosity Test Method

Equipment

Dynamic Shear Rheometer (DSR) or a fluid rheometer with temperature control system and cup and helical ribbon geometry (Figure 20):

1) Blender or equivalent (ASTM C1738) that is speed controlled with a feedback loop. A timer should be available to stop the mixer without operator intervention.
2) Stainless steel jacketed blender cup
3) Water bath for temperature control of the blender container.
4) Standard Reference Material ® 2492 Bingham Paste Mixture for Rheological Measurements (National Institute of Standards and Technology)
5) Distilled water
6) Insulated thermos sufficiently sized to hold single batch of blended grout
7) Utensils for measuring small amounts of liquid and powder – syringe without the needle.
8) 250 mL Pyrex beaker for weighing water
9) Small cup for weighing grout powder
10) Small rubber spatula
11) Digital scale
12) Teflon cup for calibration (if available)

Sample preparation

Before mixing, check blender for leaks. Water circulating through the temperature control system may leak into the cup. Water may also leak out of the mixer through the blade apparatus. For these reasons, it is imperative to check the mixer for all of these possible sources of error before mixing. Mix grout sample using ASTM C1738:

1) Weigh grout powder.
2) Weigh water. The Pyrex beaker should be pre-wetted before measuring the water by filling it up with water then draining it so that it is still damp on the interior walls. This ensures that when the mix water is poured out, any excess that remains behind on the walls of the beaker was already there, minimizing the amount lost during pouring.
3) Wet inside of the stainless steel cup using the same technique described in the previous step.
4) Set water bath to 100 ºF (38°C) and plug the water circulation tubes into the stainless steel cup.
5) Set blender timer to 60 s.
6) Place the stainless steel cup onto the mixer.

7) Place the mixing water into the stainless steel cup.
8) Place the lid with the funnel attached with the grout powder inside of the funnel on top of the stainless steel cup. Make sure to remove the cap from the funnel. Seat the lid as tight as possible on top of the stainless steel cup.
9) Press the start button.
10) Adjust blender speed to 4000 RPM (419 rad/s).
11) Add grout powder to cup through funnel within 60 s. As material is added, adjust blender to maintain 4000 RPM (419 rad/s).
12) When the mixer stops, start a stopwatch.
13) Scrape the inside of the stainless steel cup with a rubber spatula to push all of the grout paste down.
14) Set the blender timer to 30 s.
15) Place lid on the stainless steel cup and press down to ensure it is seated.
16) When the stopwatch reads 2.5 min, start blender.
17) Adjust speed to 10,000 RPM (1047 rad/s). As mixing continues further speed adjustment may be required to maintain 10,000 RPM (1047 rad/s).
18) When the mixer stops, measure grout temperature and record. Grout is ready to be loaded into the rheometer.

DSR conditioning and testing

Before mixing the grout, the dynamic shear rheometer (DSR) will have to be prepared and ready for the sample. After the DSR is prepared, and the grout has been mixed, the test can begin.

The DSR should be hooked up to a computer to control and acquire the data. The following steps must be followed consecutively to avoid any error messages.

1) Set temperature control to 100° F (37.8° C)
2) Load sample into the cup so that the free surface of the grout is in line with the cup rim.
3) Pre-shear sample at 165 s^{-1} for 30 s.
4) At the conclusion of pre-shearing, immediately initiate a shear rate of 50 s^{-1} continuously for 60 min.
5) Record viscosity at one min and at 15 min intervals during the test.

Calibration of the DSR using SRM 2492

The standard reference material developed by NIST is intended for use in calibration of rheometers for measuring the rheological properties of cement. The SRM has a certified Bingham plastic viscosity and yield stress that are within expanded uncertainty limits. All materials should be stored at room temperature before mixing. DSR calibration using SRM is as follows:

1) Calculate the plastic viscosity by plotting the shear rate (x-axis) versus shear stress (y-axis). The slope of this plot is the plastic viscosity.
2) The table of certified values on the NIST Certificate of Analysis for SRM 2492 states that the Bingham plastic viscosity at one day should have a mean value of 7.1 Pa·s (check the most recent certificate for the exact value).
3) After ratios of the certified mean value divided by the measured values are tabulated, the adjustment factor is then calculated by averaging of all of the tabulated ratios.
4) Now the shear stress factor for the helical ribbon geometry can be scaled by this adjustment factor, or each viscosity measurement can be manually scaled by the adjustment factor during post-processing.
5) The Bingham paste will then have to be tested again and plotted to see if it is within the expanded uncertainty limits found on the SRM 2492 Certificate of Analysis. Use the plunger blender for 30 s to reactivate the SRM 2492 before each use

Appendix B—Other DSR Tests

During the development of the NSR viscosity test, several other DSR methods were tested. The following sections document the results of these tests.

Flow Curve

Flow curve tests were conducted on all of the grouts several times over the period of one hour after mixing using the following procedures:

- Mix grout according to the mix procedure in high-shear mixer (ASTM C1738) with temperature control at 90°F (32.2°C). The grout materials are stored at room temperature.
- Load a sample of the grout into the DSR.
- Pre-shear grout at 165 s^{-1} for 30 s. Sample every 1 s.
- Allow grout to rest for 30 s.
- Ramp shear rate from 0.1 s^{-1} to 165 s^{-1} continuously over a period of 3 min. Sample every 1 s.
- Plot shear stress vs. shear rate. Fit model to data.

Results of the test for grout C40, 21 min after mixing are given in Figure 67. To apply the Bingham model, the material must exhibit a linear flow curve. This curve exhibits a good linear trend, and the Bingham model can be applied with an R^2 value of 0.983. The rheological Bingham parameters are a yield stress of 82.5 Pa and a plastic viscosity of 286 mPa·s. Additionally, the shear stress values measured here are similar to those measured by Amziane and Ferraris (2007).

Results of the flow curve test for grouts PT1-3, PT2-3, PT3-2, and PT4-3 mixed at the mid-range of the manufacturer's specified water content are given in Figure 68.

The Bingham model can be applied to the flow curves for grouts PT1-3, PT2-3 and PT4-3 because both exhibit a linear trend. The y-intercept of the trend line for PT4-3 is negative, however, indicating that the yield stress is negative, which is impossible. Thus, the Bingham model is not appropriate for grout PT4-3.

Grout PT3-2 exhibits both shear-thinning and shear-thickening properties, which results in nonlinear flow curve behavior. Thus, the Bingham model cannot be applied to grout PT3-2.

Previous research performed on plain grouts successfully used the Bingham model to measure the yield stress (Amziane and Ferraris 2007). The results presented here agree that the Bingham model is applicable to plain grouts. The Bingham model, however, cannot be used to assess the rheological properties of the PT grouts. For grout PT4-3, it results in the measurement of a negative yield stress. For grout PT3-2, the flow curve was nonlinear.

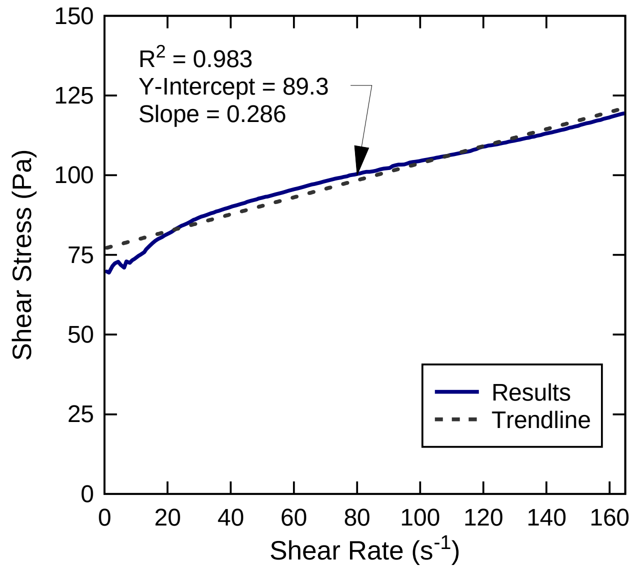

Figure 67—Flow curve for grout C40, 21 min after mixing

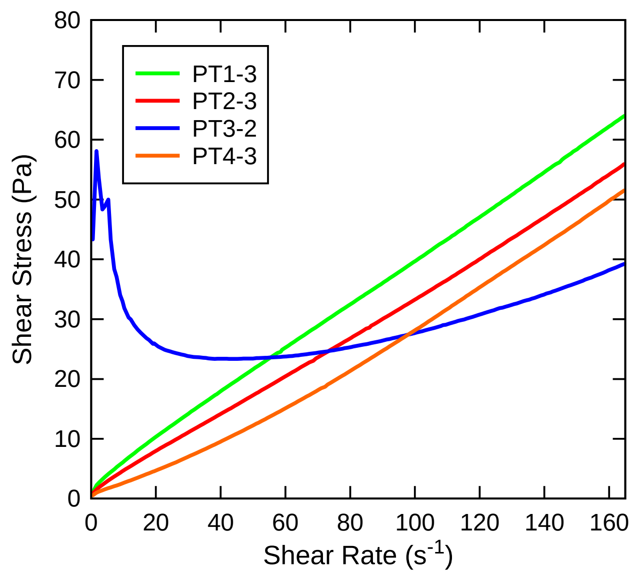

Figure 68—Flow curve for materials PT1-3, PT2-3, PT3-2, and PT4-3. The uncertainty is discussed in section 9.2.3

There may be separate models that can be fit to the individual flow curves for each of the PT grouts. However, this would not allow for a comparison between the PT grouts. Further, even if all of the tested PT grouts could be described by a single model, future grouts may be developed that cannot be described by the same model. This would make it impossible to qualify certain new PT grouts in the future.

Stress Growth

Stress growth testing on Portland cement pastes is reported in Amziane and Ferraris (2007). Both plain and PT grouts exhibited viscoplastic behavior, meaning that they should reach a certain yield stress for a given shear rate, marking the transition from solid-like behavior to fluid-like behavior. A low shear rate was imposed on the grout so that a well-defined yield point could be captured as the peak of a shear stress vs.time curve. A shear stress vs.time curve was determined every 15 min to 30 min. The calculated yield stress from each curve is plotted vs.time after mixing. In this test method, the grout must yield (i.e. begin to flow) to measure the yield stress. Several preliminary tests were conducted using the following test procedures to measure yield stress:

- Set the Peltier Jacket temperature to 90 °F (32.2 °C).
- Mix grout according to the mix procedure in high-shear mixer or blender mixer (ASTM C1738). The grout materials are stored at room temperature.
- Load a sample of the grout into the DSR.
- Subject the grout to a shear rate of 0.1 s^{-1} for a period of 5 min.
- Clean the grout out of the cup and, after 30 min, load a new sample of grout into the cup.
- Repeat steps 3 and 4 until the grout has become too thick to test.
- Plot shear stress vs. time for each sample. The maximum point on this plot is the yield stress.
- Plot the yield stress vs.time since mixing.

This test was conducted on grouts C45 and PT1-1. Ideally, if shear stress is plotted against time, the curve should exhibit a well-defined yield point. This point is taken as the yield stress of the grout (Amziane and Ferraris 2007). An idealized plot of the stress growth test is shown in Figure 69.

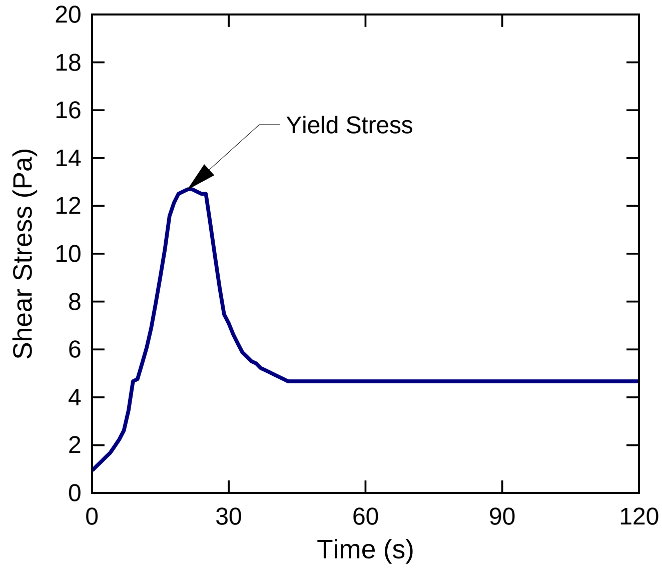

Figure 69—Idealized plot of stress growth test showing yield point. [Univ. of Florida]

Figure 69 shows that the measured shear stress in the grout increases initially, and then exhibits a well-defined yield point after approximately 20 s. Prior to yielding, the grout behaved in an elastic manner. After yielding, the grout became plastic and began to flow.

Results of the stress growth test for grout C45 and grout PT1-1 are shown in Figure 70 and Figure 71 respectively.

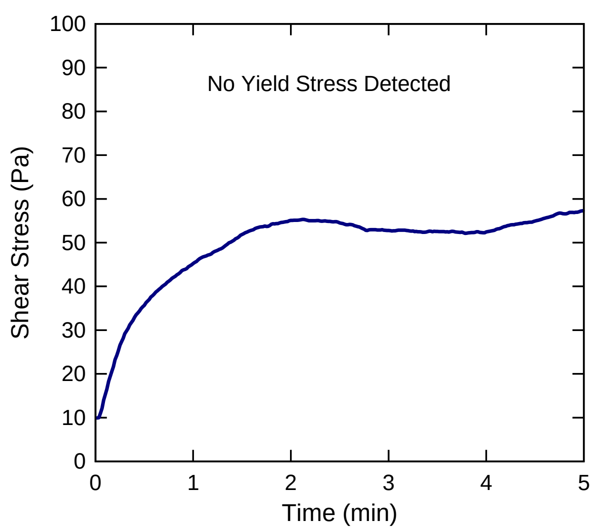

Figure 70—Plot of shear stress vs. time for grout C45. Tested using cup and ribbon with shear rate of 0.1 s^{-1}.

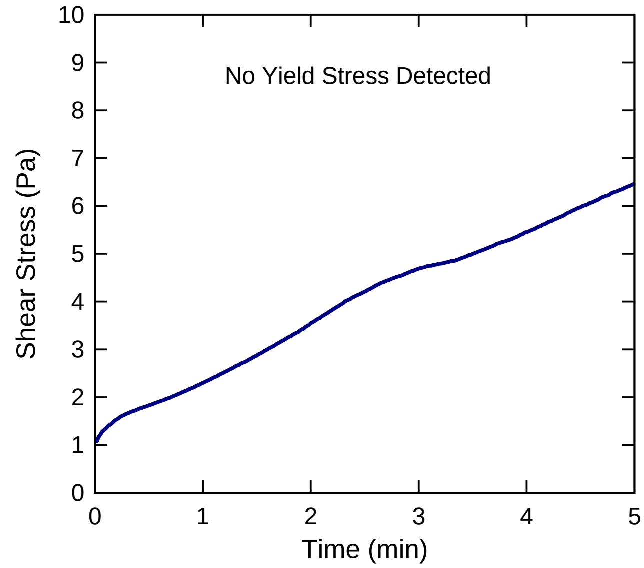

Figure 71—Plot of shear stress vs. time for grout PT1-1. Tested using cup and ribbon with shear rate of 0.1 s^{-1}.

The yield stress could not be measured in grout C45 or grout PT1-1 when using the cup and ribbon geometry. This is likely because, even at a very low shear rate, the cup and ribbon agitates the grout excessively and causes it to yield before the first data point is sampled.

The stress growth test was found to be unsuitable replacement test for the flow cone test because it cannot be used to assess the rheological properties of the grouts using the cup and ribbon geometry. The test uses the yield stress as a metric for comparison, but the grouts tested did not exhibit a well-defined yield point.

Low-Amplitude Shear Strain Oscillations

In this test method, the rheometer imposed an oscillating strain on the grout and measured the response strain of the grouts. The difference between the input and output strain signals was used to quantify the storage modulus, G', and the loss modulus, G". As the stiffness of the grout increased due to cement hydration, the storage modulus increased because the grouts became more solid-like. The oscillatory time sweep test method provided a way of measuring how the grout thickens over time without imposing enough shear to cause the grout to yield. This allowed for the testing of a single sample of the grout in the rheometer continuously.

The amplitude of the strain that the rheometer imposes on the grout must be within the grout's Linear-Viscoelastic Region (LVR), prior to the yield point. Similar materials (i.e. all grouts) should have similar LVRs. Preliminary tests were conducted on C45, PT1-1, and PT2-1. To determine the LVR, an oscillatory strain sweep was used. The procedure for determining the LVR was as follows:

- Set the Peltier Jacket temperature to 90 °F.
- Mix grout according to the mix procedure in high-shear mixer or blender mixer (ASTM C1738). The grout materials are stored at room temperature.
- Allow grout to rest for three min.
- The rheometer imposed an oscillating strain on the grout between 0.001 and 10 in a logarithmic manner at a frequency of 1 Hz. 10 points were sampled per decade.
- Plot G' and G" vs. Strain using logarithmic scales. G' and G" are calculated by the rheometer software.
- The flat region on the G' and G" curves provides an approximate range of the LVR.

Once the LVR of the grout was determined, a strain amplitude within the LVR was selected for the oscillatory time sweep. The procedure for the oscillatory time sweep was as follows:

- Set the Peltier Jacket temperature to 90 °F.
- Allow grout to rest for three min.
- Impose a shear rate of 100 s^{-1} for a period of 1 min to break down any structure the grout has built.
- Impose an oscillating strain within the LVR at a frequency of 1 Hz until the grout begins to set several hours later. Sampling time is 5 s.
- Plot G' vs. time.

Plots of the procedure used to determine the LVR for each type of grout are shown in Figure 72, Figure 73, and Figure 74. A summary of the LVR for each grout is provided in Table 16.

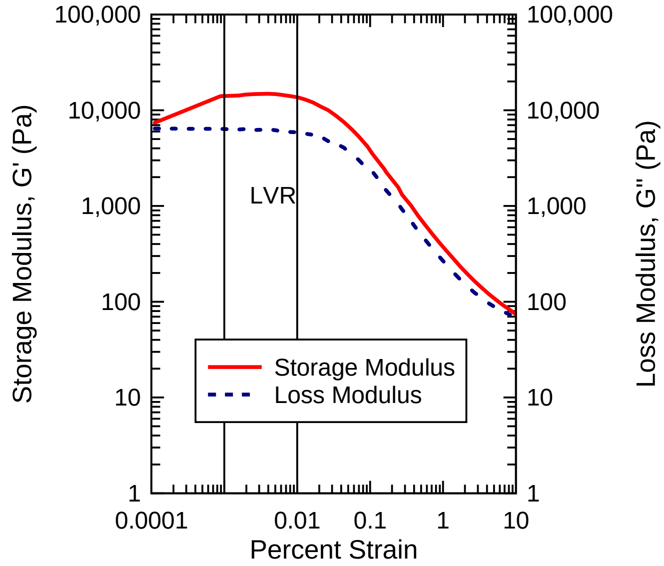

Figure 72—Plot of G’ and G” vs. strain for grout C45

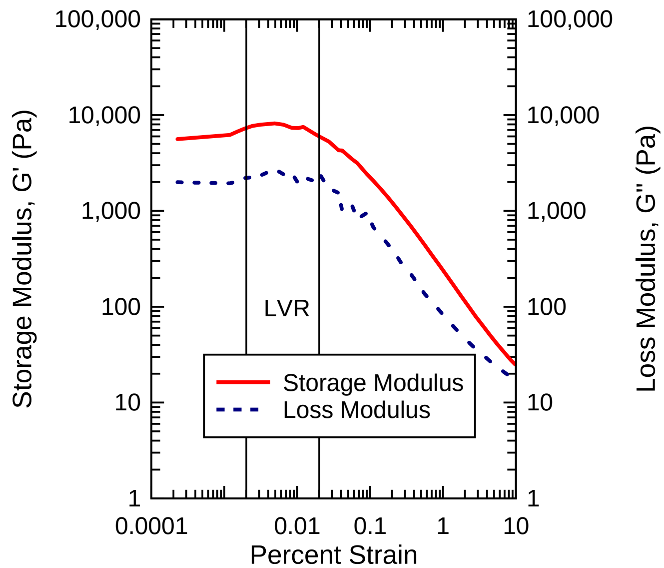

Figure 73—Plot of G’ and G” vs. strain for grout PT1-1

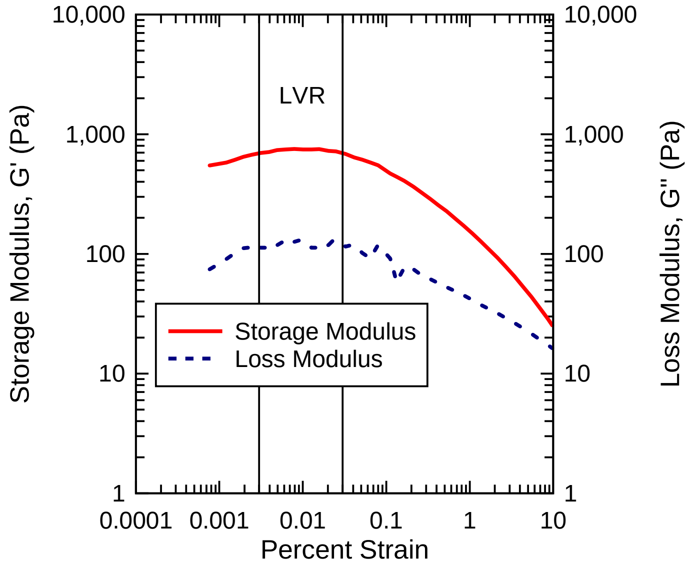

Figure 74—Plot of G' and G" vs. strain for grout PT2-1

Table 16—Summary of LVR results for each type of grout

Grout	LVR Min	LVR Max
C45	0.00001	0.0001
PT1-1	0.00002	0.0002
PT2-1	0.00003	0.0003

After the LVR was determined for each grout, an oscillatory time sweep test was conducted on each type of grout for varying lengths of time. A strain amplitude of 0.00004 was selected. Research by Schultz and Struble (1993) indicated that the critical strain for cement paste was approximately 0.0001. This agrees well with the results presented here.

A plot of the storage modulus vs. time for each of the grouts tested is shown in Figure 75. This test provides a method to accurately analyze how the grouts thicken over time where they are strained in the elastic region.

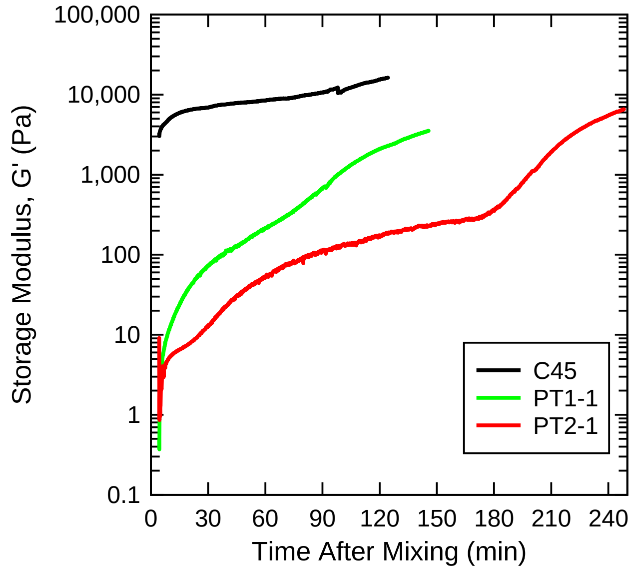

Figure 75—Plot of storage modulus vs. time for all tested grouts

It can be seen that the PT grouts exhibit a larger increase in storage modulus during the first hour than the plain grout does. However, the storage modulus of the PT grouts is still much lower than that of the plain cement grout. Schultz and Struble (1993) indicate that the storage modulus of the cement paste at the critical strain was approximately 14 to 24 kPa. This agrees with the results presented here.

The oscillatory test is useful for assessing the change in stiffness of the grouts where they are in the elastic range below their yield point. This research is aimed at studying the fluidity of the grouts, which occurs in the plastic region after the grouts have yielded. The oscillatory test does not assess the plastic properties of the grouts after they have yielded, so it was deemed unsuitable for fluidity assessment.

Increasing-Decreasing - Hysteresis

In the hysteresis loop test, the shear rate was increased from zero to a maximum value over a pre-determined amount of time, and then decreased from the maximum value back to zero over the same time period. This process was repeated several times until repeatable loops were obtained. Loops are considered repeatable when the area between the curves on consecutive runs is similar in size. The area inside the loops was then quantified as the degree of thixotropy. The procedure used was as follows:

1. Set the Peltier Jacket temperature to 90 °F.
2. Mix grout according to the mix procedure in high-shear mixer or blender mixer. The grout materials are stored at room temperature.
3. Apply a pre-shear of 50 s^{-1} to the sample for a period of 30 s.
4. Increase the shear rate from 0 s^{-1} to 200 s^{-1} at a constant rate over a period of 3 min.
5. Decrease the shear rate from 200 s^{-1} to 0 s^{-1} at a constant rate over a period of 3 min.
6. Repeat steps 3 and 4 until 10 complete loops have been obtained

This test was conducted on all of the grouts, except for PT3 and PT4. The tests were conducted using both a stainless steel cup and an aluminum cup. In addition, tests were conducted using the mix procedure described in Section 8.3 as well as another mix procedure. Results did not vary significantly with any of these permutations, so only the results for the stainless steel cup and the mix procedure described in Section 8.3 are included in this report. Figure 76 and Figure 77 show the hysteresis loop size plotted against the run number for the cement grouts. The average, standard deviation, and coefficient of variation for the last 5 loops on each of the runs are included with the plots.

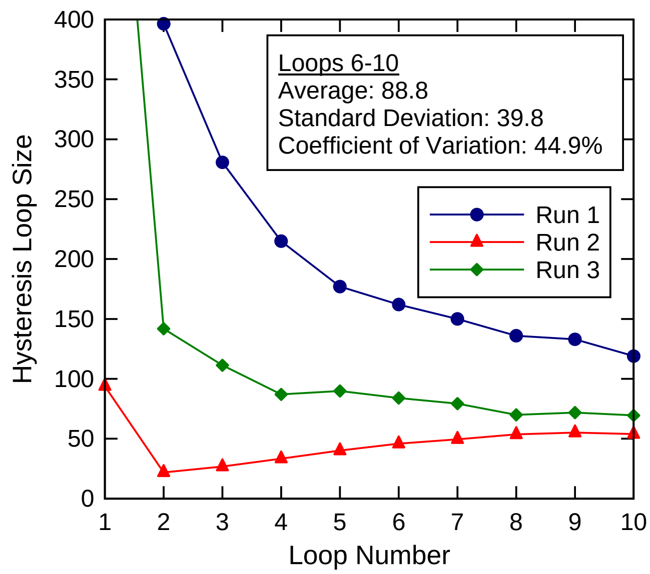

Figure 76—Plot of hysteresis loop size vs. loop number for grout C40

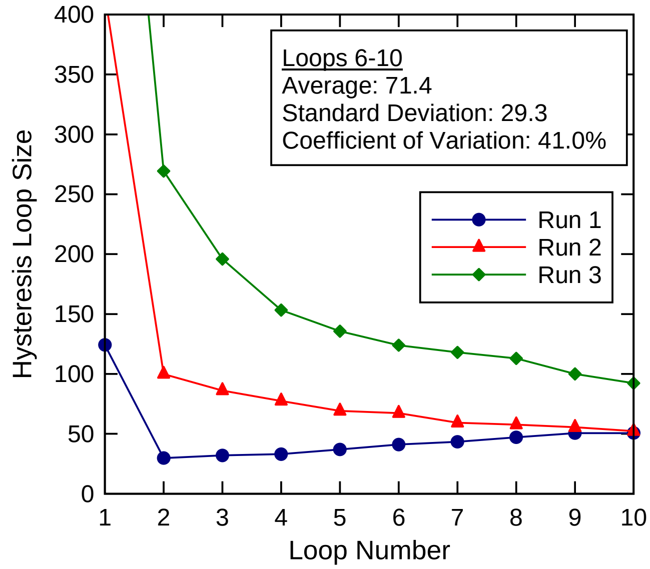

Figure 77—Plot of hysteresis loop size vs. loop number for grout C45

In the case of the plain cement grouts, it can be seen that there is very little repeatability within the individual runs and no repeatability between the runs. A very high coefficient of variation is seen between the data points obtained for the last 5 loops in all of the tests, which suggests that this test method is not appropriate for a plain cement grout.

Figure 78 and Figure 79 display the hysteresis loop size plotted against the run number for the PT grouts.

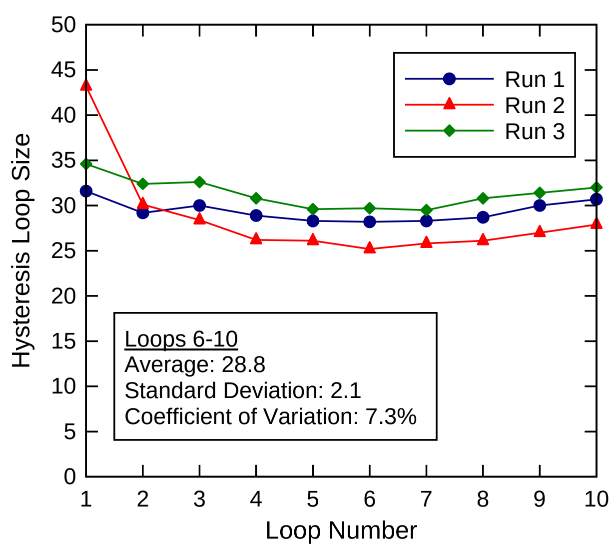

Figure 78—Plot of hysteresis loop size vs. loop number for grout PT1-1

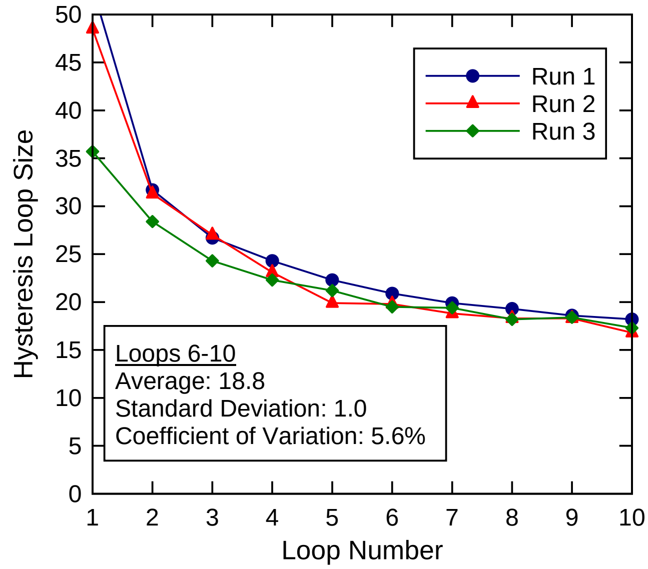

Figure 79—Plot of hysteresis loop size vs. loop number for grout PT2-1

The PT grouts exhibit much more repeatability in this test than the plain cement grouts did. Each test resulted in similar loop sizes over the course of the test, and the coefficient of variation between all of the data points for the last 6 loops was under 10 % in each case. This suggests that the hysteresis test may be appropriate for the PT grouts, even though it is clearly not appropriate for the plain cement grouts.

Increasing-Decreasing-Stepped

In this test procedure the shear rate was held constant until the viscosity reached equilibrium; shear rate was then instantaneously increased by an order of magnitude. This process was repeated over several decades. The procedure used for conducting the stepped test was as follows:

1. Set the Peltier Jacket temperature to 90 °F.
2. Mix grout according to the mix procedure in high-shear mixer or blender mixer. The grout materials are stored at room temperature.
3. Apply a pre-shear of 0.01 s^{-1} to the sample for a period of 10 s.
4. Hold the shear rate constant at 0.1 s^{-1} for a period of 10 min.
5. Instantaneously increase the shear rate to 1 s^{-1} and hold constant for a period of 10 min.
6. Instantaneously increase the shear rate to 10 s^{-1} and hold constant for a period of 10 min.
7. Instantaneously increase the shear rate to 100 s^{-1} and hold constant for a period of 10 min.

This test was conducted on all of the grouts, except for PT3 and PT4. The stepped test provides information on both the shear-thinning and thixotropic properties of the grout. A plot of the viscosity at different shear rates vs. time for grout C45 is shown in Figure 80, while the same plot for grout PT1 is shown in Figure 81.

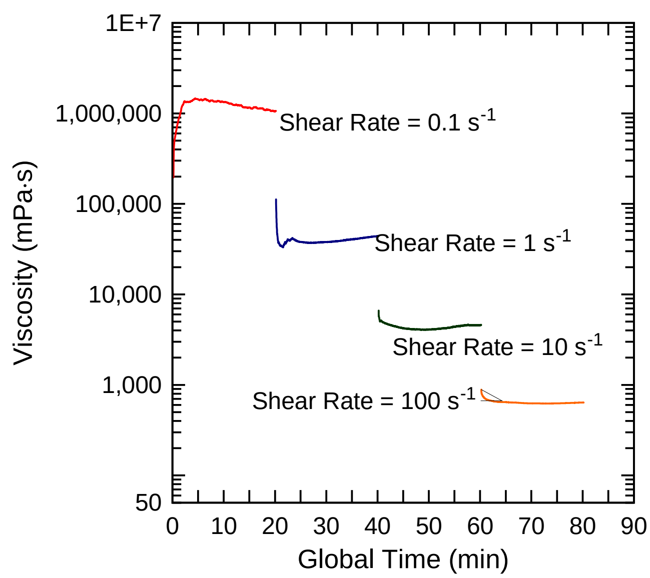

Figure 80—Plot of viscosity at different shear rates vs. time for grout C45

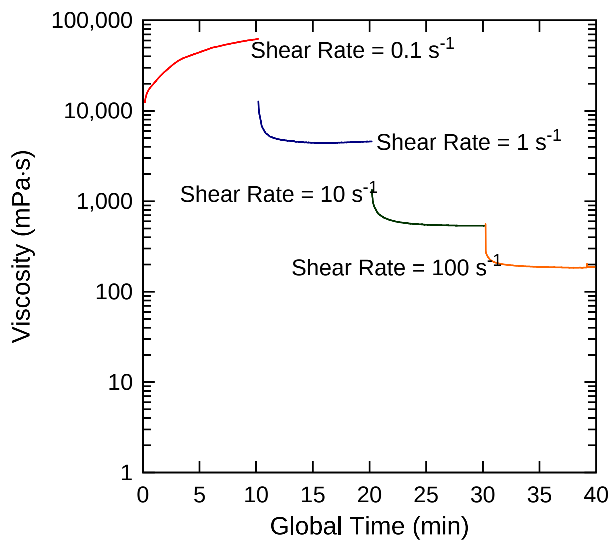

Figure 81—Plot of viscosity at different shear rates vs. time for grout PT1-1

The difference between the behavior of the plain cement grout and the behavior of the PT grout can be seen in the shapes of the viscosity versus time curves. The primary difference is seen at a shear rate of 0.1 s^{-1}. At this shear rate, the viscosity in the plain cement grout increases for several minutes and then remains approximately constant for the remainder of the hold period. The PT grout exhibits only an increase in viscosity at this shear rate. This behavior indicates that the plain cement grout exhibits thixotropic behavior at a very low shear rate while the PT grout exhibits anti-thixotropic behavior at a very low shear rate. When the PT grout is subjected to a low shear rate, as it is when it is in the post-tension duct, it is meant to thicken and set; for this reason, anti-thixotropic behavior is desired at a low shear rate. When the PT grout is subjected to a higher shear rate, as it is during pumping, it is meant to become very fluid so that it can fill the ducts; for this reason, thixotropic behavior is desired at a high shear rate. The stepped test confirms that this behavior occurs in the PT grout, while it does not in the plain cement grout.

Appendix C—Alternative High-Shear Mixer Test Procedure

The high-shear mixer was investigated for its potential to replace the conditioning aspect of the HTGF test. The water bath on the high-shear mixer was used to hold the sample temperature at approximately 100 °F, based on the results measured during the HTGF test. The procedure used to condition the grout over the period of one hour in the high-shear mixer was as follows:

1. Mix grout according to the mix procedure in high-shear mixer with water bath set to 100 °F (38 °C). The grout materials are stored at room temperature.
2. Run low speed agitator paddle at 35 RPM (3.7 rad/s) continuously for one hour.
3. One minute before sampling, run high speed agitator at 3,500 RPM (366 rad/s).
4. Collect sample every 15 min for flow cone and mud balance testing.

A comparison of the flow cone efflux time measured using the high-shear mixer for grout conditioning versus that measured during the HTGF test is shown in Figure 82.

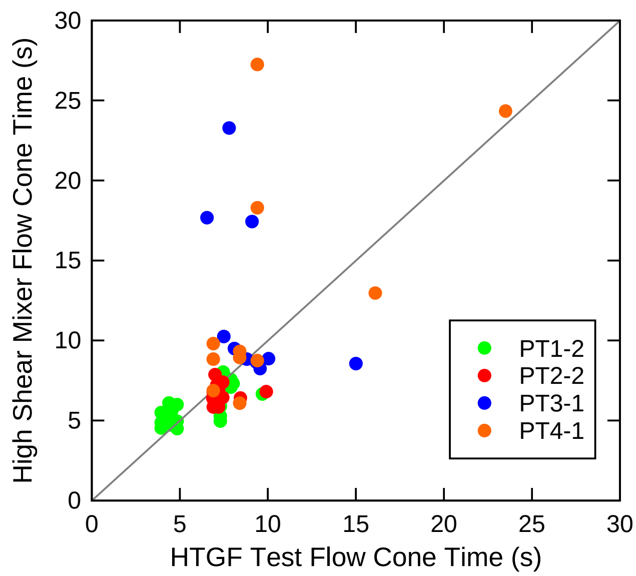

Figure 82—Plot of flow cone efflux time measured where high-shear mixer was used for grout conditioning versus flow cone efflux time measured during HTGF test. The flow cone time uncertainty is 0.88 s (ASTM C939)

Grout PT4-1 was tested after the manufacturer's expiration date when the high-shear mixer simulated conditioning tests were conducted. This may explain the higher flow cone efflux times measured during the simulated conditioning tests as opposed to the actual HTGF test.

Grout PT3-1 exhibited a large degree of shear-thinning and thixotropy. The amount of time that passed between collecting a sample and conducting a flow cone test had a significant effect on the measured flow cone efflux time. This provides an adequate basis to disregard the data collected for grouts PT3-1 and PT4-1. Figure 83 contains the same results shown in Figure 82 for grouts PT1-2 and PT2-2 only.

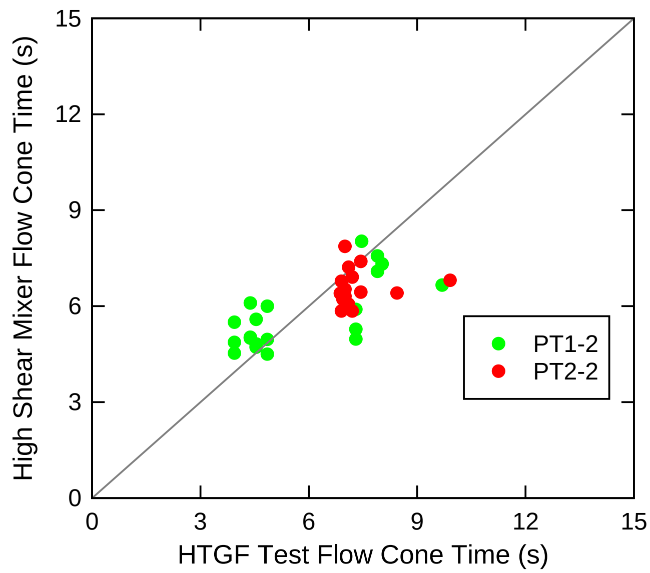

Figure 83—Selected data from plot of flow cone efflux time measured where high-shear mixer was used for grout conditioning versus flow cone efflux time measured during HTGF test. The flow cone time uncertainty is 0.88 s (ASTM C939)

Based on the results presented in Figure 83, the high-shear mixer can be used to replicate the conditioning process imposed on the grout by the HTGF test for grouts with low flow cone efflux times. Similar flow cone efflux times are measured where the high-shear mixer is used to condition the grout and during the HTGF test. However, very little data were recorded for flow cone efflux times above about 10 s. Grouts PT1-2 and PT2-2 exhibited very low flow cones during the entire 60-min simulated conditioning period and during the HTGF test's 60-min recirculation period. Performance classifications for flow cone results similar to those developed for the viscosity test are given in Table 17. These are based on the limited data presented here.

Table 17—Calibration of flow cone test with performance classification

Viscosity result	Performance Classification
Flow cone efflux time ≤ 10 s	Satisfactory
Flow cone efflux time > 10 s	Uncertain

In addition to flow cone tests, mud balance tests were conducted on each sample during the high-shear mixer simulated conditioning tests. A comparison of the wet density measured using the high-shear mixer for grout conditioning versus those measured during the HTGF test is shown in Figure 84.

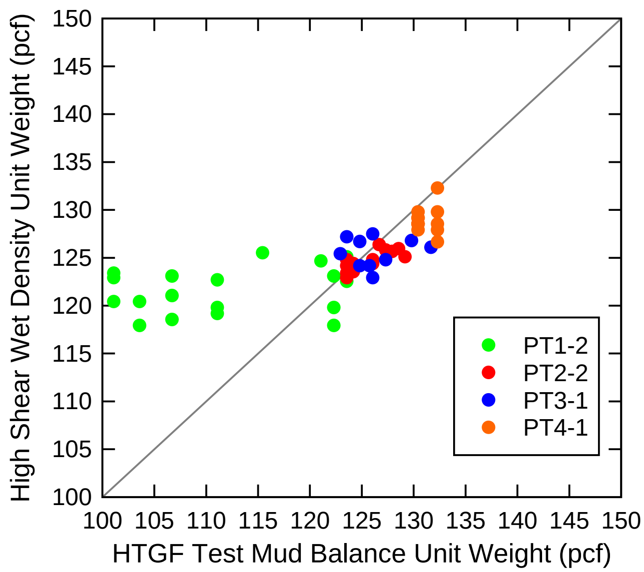

Figure 84—Plot of wet density value measured where high-shear mixer was used for grout conditioning versus mud balance value measured during HTGF test. The uncertainty for this test method in single laboratory tests has not yet been established.

With the exception of grout PT1-2, all of the grouts exhibit a fairly similar wet density value where the high-shear mixer was used to condition the grout as they do during the actual HTGF test. However, the significant drop in density in grout PT1-2 over the course of the one hour conditioning period during the HTGF test was not measured where the high-shear mixer was used. This indicates that, even though this conditioning technique can be used to assess the grout fluidity, it does not replicate the foaming issues in grout PT1-2 discovered during the HTGF test.

Appendix D—UMD Data

The results from the testing conducted at University of Minnesota-Duluth (UMD) can be seen in Table 18 below.

Table 18—Summary of UMD viscosity testing results

ID	TEST DATA					
	0 min (mPa·s)	45 min (mPa·s)	60 min (mPa·s)	MAX/MIN (60 min/0 min)	MAX/MIN (60 min/0 min)	FINAL SLOPE (mPa)
PT4-6	426	862	1286	3.02	1.49	0.42
PT4-6	353	759	1286	3.66	1.70	0.53
PT1-4	375	716	784	2.09	1.09	0.07
PT1-4	396	739	811	2.05	1.10	0.07
PT2-4	787	1108	1152	1.46	1.04	0.04
PT2-4	687	1001	1046	1.52	1.04	0.04
PT2-4	926	1032	1076	1.16	1.04	0.04
PT3-3	310	614	651	2.10	1.06	0.04
PT3-3	418	693	733	1.75	1.06	0.04
PT3-3	355	653	689	1.95	1.05	0.04
PT5-1	984	942	944	0.96	1.00	0.00
PT5-1	762	772	767	1.01	0.99	0.00
PT5-1	764	777	773	1.01	0.99	0.00
PT5-1	767	784	775	1.01	0.99	-0.01
PT6-1	592	2706	3668	6.20	1.36	0.96
PT6-1	609	2762	4451	7.30	1.61	1.68
PT6-1	672	4339	17670	26.3	4.08	13.3
PT6-1	654	3713	13867	21.2	3.74	10.1

Appendix E—NIST Data

The results from the testing conducted at NIST can be seen in Table 19 below.

Table 19—Summary of NIST viscosity testing results

ID	TEST DATA					
	0 min (mPa·s)	45 min (mPa·s)	60 min (mPa·s)	MAX/MIN (60 min/0 min)	MAX/MIN (60 min/45 min)	FINAL SLOPE (mPa)
PT4-4	306	526	707	2.31	1.35	0.202
PT4-4	298	519	691	2.32	1.33	0.191
PT1-3	222	301	313	1.41	1.04	0.013
PT1-3	221	314	321	1.45	1.02	0.007
PT2-3	226	315	318	1.41	1.01	0.004
PT2-3	227	317	322	1.42	1.02	0.006
PT3-2	292	609	658	2.26	1.08	0.055
PT3-2	267	583	629	2.35	1.08	0.050

Appendix F—UF Data

The results from the testing conducted at UF can be seen in Table 20 below.

Table 20—Summary of UF viscosity testing results

ID	TEST DATA					
	0 min (mPa·s)	45 min (mPa·s)	60 min (mPa·s)	MAX/MIN (60 min/0 min)	MAX/MIN (60 min/45 min)	FINAL SLOPE (mPa)
PT4-4	351	666	920	2.62	1.38	0.262
	349	660	919	2.64	1.39	0.268
	374	702	924	2.47	1.32	0.229
PT4-6	320	620	843	2.64	1.36	0.230
	341	649	842	2.47	1.30	0.199
	346	669	846	2.45	1.26	0.181
PT1-3	249	319	334	1.34	1.05	0.016
PT1-4	243	450	500	2.06	1.11	0.052
	253	475	521	2.06	1.10	0.047
	254	475	541	2.14	1.14	0.068
PT2-3	222	292	299	1.34	1.03	0.008
	219	286	292	1.33	1.02	0.006
PT2-4	364	484	517	1.42	1.07	0.034
	378	523	554	1.47	1.06	0.033
	376	517	549	1.46	1.06	0.032
PT3-2	255	414	443	1.74	1.07	0.030
	256	435	464	1.82	1.07	0.029
	262	438	467	1.78	1.07	0.030
PT3-3	239	405	431	1.81	1.06	0.026
	240	406	429	1.79	1.06	0.023
	242	403	427	1.76	1.06	0.024
PT5-1	585	607	621	1.06	1.02	0.014
	586	645	661	1.13	1.03	0.017
	624	666	681	1.09	1.02	0.016
PT6-1	338	1540	2100	6.20	1.36	0.566
	349	1580	2550	7.30	1.61	0.994
	443	1640	1980	4.46	1.20	0.342