A STUDY TO DETERMINE THE CEMENT SLURRY BEHAVIOUR TO PREVENT THE FLUID MIGRATION

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ABSTRACT

A STUDY TO DETERMINE THE CEMENT SLURRY BEHAVIOUR TO PREVENT THE FLUID MIGRATION

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Fluid migration behind the cased holes is an important problem for oil and gas industry both considering short terms and long terms after cementing operation. For many reasons like high formation pressures, high shrinkage rate of cement slurry while setting, lack of mechanical seal, channeling due to cement slurry setting profile, hydrocarbon migration may occur and lead expensive recompletion operations and sometimes abandonment. Solutions to this problem vary including high density-low fluid loss cement slurry or right angle cement setting profile.

During this study, the effect of “free water” which is the basic quality property of API G class cement, on fluid migration potential has been tested for different samples and in combination with different physical conditions. For this study API G class cements have been used. In order to justify the quality of each cement sample standard API G class quality tests were conducted. Moreover, as a main instrument “Static Gel Strength Analyzer” is used to measure the static gel strength of cement slurry and how long it takes to complete transition time.

Bolu cement, Nuh cement, and Mix G cement samples were tested according to their free fluid values which are %2.5, %5, %3.12 respectively, and it is found that the

Bolu cement with lowest free fluid content has the lowest potential for fluid migration.

As a conclusion, fluid migration through behind the cased hole is a major threat for the life of the well. Appropriate cement slurry system may easily defeat this threat and lead cost saving well plans.

Key words: Fluid migration, fluid loss, transition time, channeling, right angle, API G class cement, free water, high formation pressure

ÖZ

ÇİMENTO KARIŞIMININ AKIŞKAN GÖÇÜNÜ ENGELLEMEYE YÖNELİK DAVRANIŞLARINI BELİRLEMEYE YÖNELİK BİR ÇALIŞMA

Karakaya, Güray

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Çimentolama operasyonundan sonra ki hem kısa zaman periyodunda hem de uzun zaman periyodunda koruma borusu arkasına doğru gelişen akışkan göçü petrol endüstrisi için önemli bir problemdir. Yüksek formasyon basıncı, çimento karışımının donma esnasında yüksek oranda hacimsel küçülmesi, mekanik izolasyonun olmayışı ve de çimento karışımının donması esnasında istenilen donma eğrisinin elde edilememesi gibi sorunlar hidrokarbon göçüne neden olarak oldukça pahalı tamamlama operasyonlarına ve hatta kuyu terkine sebep olabilmektedir. Çözümler çeşitlilik göstermekle beraber yüksek yoğunluklu ve düşük su kaybı değerine sahip çimento karışımları ya da dik açı yapan donma profillerine sahip çimento karışımları bu çözümlere dahil edilmelidir.

Bu çalışma kapsamında yapılan işlerde API G sınıfı çimentolarının temel kalite özelliklerinden biri olan serbest su değerinin akışkan göçü potansiyeline etkisi, farklı çimento örneklerinde ve de farklı fiziksel şartları da kapsayacak şekilde test edilerek değerlendirilmiştir. Bu çalışmada API G sınıfı çimento örnekleri kullanılmıştır. Her çimento numunesinin API G sınıfı çimento olduğunu standart kalite testleri yapılarak doğrulanmıştır. Ek olarak, “Static Gel Strength Analyzer” temel test aleti olarak durgun jel mukavemeti ölçümünde kullanılmış ayrıca bu test sırasındaki faz arası geçiş zamanı ölçülmüştür.

Bolu çimento, Nuh çimento ve Mix G çimentosu numuneleri, sırasıyla %2.5, %5, ve %3.12 serbest su değerlerine sahip olup test edilmişlerdir. En düşük serbest su miktarına sahip olan Bolu çimentonun, en düşük akışkan göçü potansiyeline sahip olduğu görülmüştür.

Sonuç olarak, koruma borusu arkasına doğru gelişen akışkan göçü bir kuyunun ömrü için temel tehditlerden biridir. Uygun çimento karışımları bu tehdidi kolayca ortadan kaldırabilir ve de masrafları azaltan kuyu planlamalarını sağlayabilir.

Anahtar kelimeler: Akışkan göçü, su kaybı, geçiş zamanı, kanallaşma, dik açı, API G sınıfi çimento ,serbest su, yüksek formasyon basıncı

To Burcu

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NOMENCLATURE

Abbrevations

API	: American Petroleum Institute
NDT	: Non-Destructive Test
ASTM	: American society for Testing and Materials
BHST	: Bottom hole static temperature
BHP	: Bottom hole pressure
SGS	: Static gel strength

CHAPTER 1

INTRODUCTION

Cementing is the most practical and beneficial method to create a strength zone against the physical and chemical forces in a well exposing the underground environment. Properties of the cement placed between the protective tubular (casing) and open hole have important roles for the life of the well. Oil and Gas industry is still in progress to improve the properties of that strength zone. There are some parameters to control the how this isolating zone works. Those parameters can be classified into four main branches. (Nelson, 1990)

First one is related to the all parameters provided by cement sheath on reservoir, affecting the well performance. Second one is considering parameters of design works for cement slurry to maintain desired physical and chemicals properties for each individual well condition. Third one is capturing various aspects of cementing job for different purposes including pre job works, like squeeze cementing, foamed cement or horizontal well cementing. Final main branch is considering all cementing job evaluation for short term and long term after the placement of cement slurry.

As stated above, first and second main branch and parameters defining main quantities to control the cement-reservoir interactions and cement slurry design work. They are key issues for oil and gas industry. After a conventional drilling operation; according to your depth and casing design parameters, each section shall be definitely isolated from the other sections. Not only from the reservoir part but also cement slurry part should be hydraulically sealed. Zonal isolation preserves the reservoir conditions including permeability for vicinity of wellbore but more important than, zonal isolation preserves cement slurry against unwanted fluid migration from the reservoir.

Santra et al. (2007) stated formation fluid influx into cement slurries presents not only short term problems like losing appropriate composition by shallow water influxes but also present long term problems by gas migration. There are a very large number of wells that leak or have sustained casing pressure (SCP). In Central Europe and the Middle East there are hundreds of wells with reports of trapped pressure that cannot be bled off. In USA and Canada there are thousands of wells leaking to the surface which may or may not be discharge to atmosphere. Furthermore 25% of wells in Gulf of Mexico have measurable sustained casing pressures maintained at Cavanagh et al. (2007). More clear way for maintain this fact that 25 % of primary cementing job failures are because of gas migration. (Gonzalo, 2005)

Mainly fluid influx (migration) into the cement slurry or into a cement sheath may occur because of following circumstances.

Gas migration behind shallow casing strings or behind long liners are firstly distinctive reasons can be followed by large annular volume leading difficulties to displace the remaining drilling fluid stated by Brooks R. et al. (2008). High shrinkage rates for setting cement together with a thick mud filter cake plays also a bad role and resulting channeling in the cemented section. (Kellingray, 2005) “Gel” concept has been stated to understand the hydrostatic balance with respect to time and kept in mind always as a reason for unwanted fluid influx declared by Bahramian et al. (2007). Moreover, fluid loss value of cement slurry and related parameters of slurry like relative permeability of gas may describe the reason for gas migration. Rheological properties of cement slurry finally can be stated as an undeniable fact when investigating the possible reasons of gas migration. Lower ECD value is crucial in order to decrease the circulation pressure and to prevent annular fluid losses from the cement slurry.

There are plenty of works to detect and to solve that unwanted fluid influx problem. At the present day, drilling industry is emphasizing the solutions for gas migration with increasing frequency. The reason is the benefits of nearly 100 % gas producing reservoirs without any sustained casing pressures and reduced remedial cementing job costs.

Due to the complexity of detecting the migration also defining the reasons for problems together with long term results of gas migration, urges the designers for multi protective designs. Rogers M.J. et al. (2004) emphasize those are;

-Zero free fluid slurry

-Low fluid loss property

-Short transit time

-Minimized volume reduction

-Low slurry permeability

In this study, some of mentioned design features were tested for different physical environments simulating the different well conditions. It is obvious that those tests were conducted with API G Class cement samples.

CHAPTER 2

LITERATURE REVIEW

An oil well has to be cased to continue its production in an optimized and predetermined flow rate till the end of production cut off. Open hole completion is also an option to complete a well but in most cases pay zone characteristics forces industry to complete the wells in cased hole forms. After this stage cased hole must keep its physical properties with its all components such as casing, cement production string (if any) and well head. Cement, behind the casing is our major focused part. When we are descending the problems related to the cased hole; "fluid migration", especially gas migration, behind the cased hole to the surface or to the upper most casing annulus is very important problem.

The most widely accepted theory for the occurrence of gas migration is the change of physical behavior of cement column due to the various factors. In other way some drastic changes also may occur after the cement slurry placed to the well bore but those rarely seen problems is out of our scope.

Literature is mainly states this problem in two different ways. First one is the chemical processes during the hydration of cement slurry and results of those reactions. The Second one is the general mechanical or operational failures that can occur during the cement job.

When we look for curing, solving and describing the problems; previous studies maintains some prediction methods to see the potential of the gas migration problem in a mathematical way, cures with some special chemicals to heal the problematic cement zone, operational precautions during the cement job and to examine the mechanical forces exerted by both formation and cement slurry itself to cement slurry. (Figure 2-1)

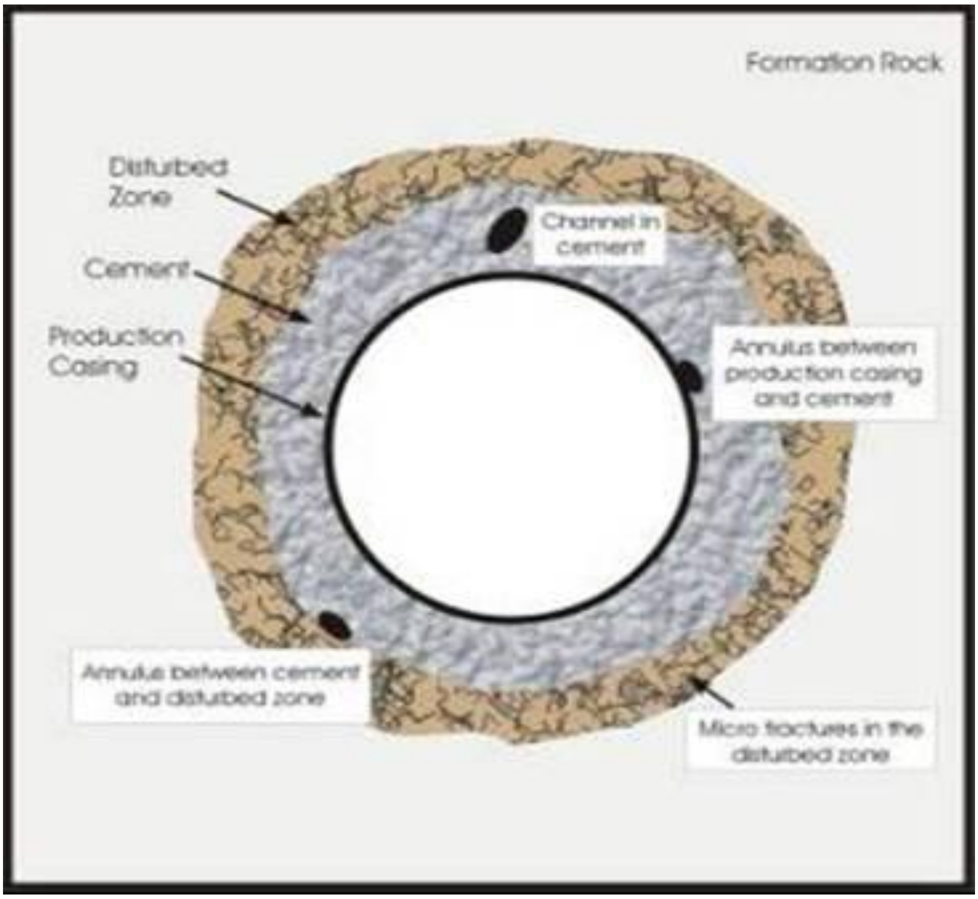

Figure 2-1: Cemented Section Upper View

(Taken from http://www.gchem.ca)

Chemical background of the cement setting process is based on hydration process. This subject can be well defined by chemical equations for each cement type but the results of the post hydration process are a bit confusing. Shrinkage phenomena and measurements for this are well defined by Reddy et al. (2007). "Gel" term and also volumetric shrinkage terms are together mentioned in this paper with three different measurement techniques. When we moved from "Gel" concept it is easy to understand resistance to pressure transmission to stop the invasion of formation fluids into cement slurry. Because once the slurry is static while keeping its hydrostatic pressure, it begins to develop static gel strength. This gelation of the cement provides an internal resistance to movement, and effectively prevents full transmission of hydrostatic pressure because the cement column becomes capable of supporting some of its weight. The following Bour et al. (1988), Dusterhoft et al. (2002), Jennings et al. (2003), Prohoska et al. (1994), Dean et al. (1992), Harris et al. (1990), Sutton et al. (1992), Stone et al. (1974) are mostly focused on gel strength, final static gel concept (symbolizes the last infinitesimal time before the slurry acts as a semi-solid), transition time, and main theories behind those concepts.

As mentioned before some other studies tried to make predetermination about the severity of gas migration by some numerical expressions. Likewise the subjects which are stated in following by Gonzalo et al. (2005).

Cures of gas migrated cement column is another part of previous studies. New cement systems or just additives developed to heal the cement system in the well bore. For different chemical purposes those products are tested in the field or in the laboratory and gives good results to prevent gas migration into the cement slurry. Especially for to slow down shrinkage effect, to make a liquid barrier inside the slurry to stop migration, to increase the elasticity of cement matrix after set in order to prevent micro cracks and micro annuli or to cure gas migrated cement column by reacting with migrated fluid. Mata et al. (2006), Moroni et al. (2007), Bouras et al. (2008), Jones et al. (1991), Coker et al. (1992), Seidel et al. (1985), Sepos et al. (1985)

Not only problems related to the gas migration are coming from physical or chemical issues due to the cement slurry itself, but also some problems may occur related to the cementing operation and well conditions.

The last statement about the general cures, solutions and descriptions mentioned in the fourth paragraph is to combine all the chemical and physical parameters for every single element of the system (cement slurry, well bore vicinity, exerted physical well forces, temperature and pressure). This one is simulating the all mechanism both in a laboratory environment and in a mathematical expression. Permeability of the cement matrix, pore size distribution, compressive strength, and channeling, rate of pressure decrease inside the cement pores, elasticity and bridging effect of cement particles could be the parts of the whole picture to examine and define the mechanism.

2.1 Static Gel Strength

As cement changes from slurry to a solid, the matrix develops a structure that behaves neither as a liquid or a solid. This process occurs before any measurable compressive strength has developed. This gelation characteristic must be understood and measured since it determines the gas or liquid in-flow potential and it may cause lower formations to be subjected to high pressures if the job is halted and restarted. As cement slurry develops static gel strength, it may become self supporting in the annulus. In some respects, during the gel phase, cement may be considered as a material with similarities to a polymer. This is true since the cement matrix exhibits non-newtonian rheological behavior and exhibits a yield point, also known as static gel strength (SGS).(Figure 2-2) Commonly expressed in units of shear stress (lbf/100ft2), the term may be considered as the shear stress that exists at the wall boundaries at the onset of movement of a column of cement in an annulus due to the presence of a head pressure.

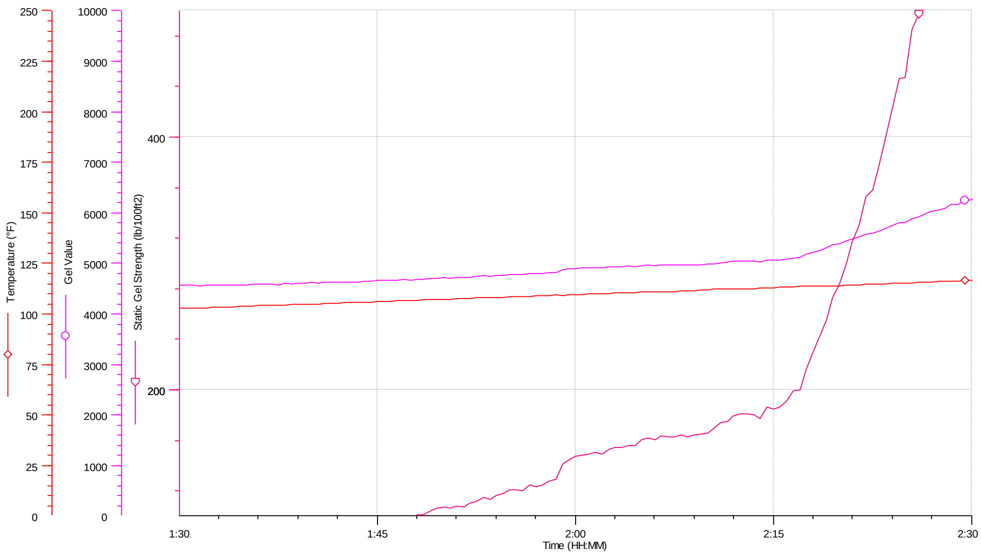

Figure 2-2: Static Gel Strength Measurement (TPAO Cement Laboratory)

Development of Static Gel Strength measurements basically relies on Ultrasonic analyzing theory. Velocity of the sound inside the cement slurry varies due to the on setting cement slurry. This differentiation of velocity is numerically turned to be a mathematical expression and calculated as strength of the material whatever the sound travels in. On the other hand we should maintain that SGS is not the direct indication of velocity of acoustic signals. The power of the signal is detected with very frequent measurements and the attenuation of signals are converted again a mathematical expression to monitor the SGS. The frequencies of the measurement not only detect the velocity of the signal but also content of it.

Someone could suggest that the Static Gel Strength Analyzer (SGSA) is a viscometer that uses acoustic signal analysis as a basis for the SGS measurement. This is not the case. A viscometer measures the shear stress corresponding to a known fluid shear rate. From this data, the viscosity of the sample is determined. One of the advantages of an acoustic measurement is that the sample is not sheared, thereby providing a fluid property measurement at zero shear rate. Research of existing literature discovered comparable techniques for fluid property measurement used in the polymer industry. Acoustic signal analysis provides measurements of the gelation characteristics of epoxy samples and curing agents. Interestingly, based on the data presented in the signal waveforms are similar to those found with a cement slurry analysis. Additional study of the process suggests that the change in the transmitted signal energy occurs due to the chemical reactions in the slurry. For example, as the amount of the unreacted water decreases during hydration, the transmitted signal energy increases. From this observation, it is expected that a continuous measurement of the unreacted water within slurry will be achieved. Once the gelation phase of the cement is complete and presumably, most of the water has been absorbed by the reaction, the signal attenuation characteristics are no longer of interest with respect to SGS measurements. In fact, the acoustic velocity begins to change rapidly as the sample develops compressive strength. Intuitively, one would expect this to be true since a substance that exhibits compressive strength is no longer a gel. Moon et al. (1999)

2.2 Compressive Strength

The compressive strength of the material describes the stress at which a material fails when a compressive load is applied. When a compressive load is applied to a sample of brittle elastic material such as cement, stress generally increases linearly with strain until small micro cracks in the sample begin to grow. This is progressive mechanism and manifests itself strain-stress pilot by the change from linear proportionally between stress and strain to a softening section of the curve near the failure point. Once the cracks coalesce and reach a critical size, the sample will fracture via a complicated mechanism, which is determined by boundary stress conditions and the geometry of the sample.

Compare this with the definition of cement toughness. Simplistically, toughness describes the property of material initiation and propagation of cracks in a particular orientation. Fracture toughness is quantitatively defined as the energy required propagating a fracture a unit width by unit length. Without considering mathematical details, a reasonable indication of toughness for similar materials is given by the area under stress-strain curve to the failure point. This area varies according to the toughness of the material being tested. For example consider two objects X and Y that have the same compressive strength. The material X has a much smaller strain to failure than material Y, which contains latex. Therefore material Y can deform further and absorb more energy before it fractures. Material Y is tougher than material X. (Figure 2-3)

Due to the API specifications there are three basic methods to measure the compressive strength of cement slurry. For high pressure conditions it is needed curing the cement sample for the defined pressure and temperature and after an axial load applied until the first indication of cracks occurrences. Second way is to scale the atmospheric conditions for both cold and hot atmosphere. A water bath or cooling bath to simulate moderate or permafrost conditions than same procedure can easily be followed in order to apply uniaxial load by hydraulic press.(Nelson, 1990) Third method is a NDT (nondestructive test) measure the sonic travel time of ultrasonic energy through a cement sample as it cures under simulated wellbore conditions of temperature and pressure. The ultrasonic velocity directly measures the bulk compressibility of the sample, but this is found to be well-correlated with compressive strength. Still weakest point of those nondestructive measurements is lack of lateral force application on the slurry sample inside the cell. Since in realistic casing cement not encounter only axial strain but also some lateral forces can be applied by the formations.

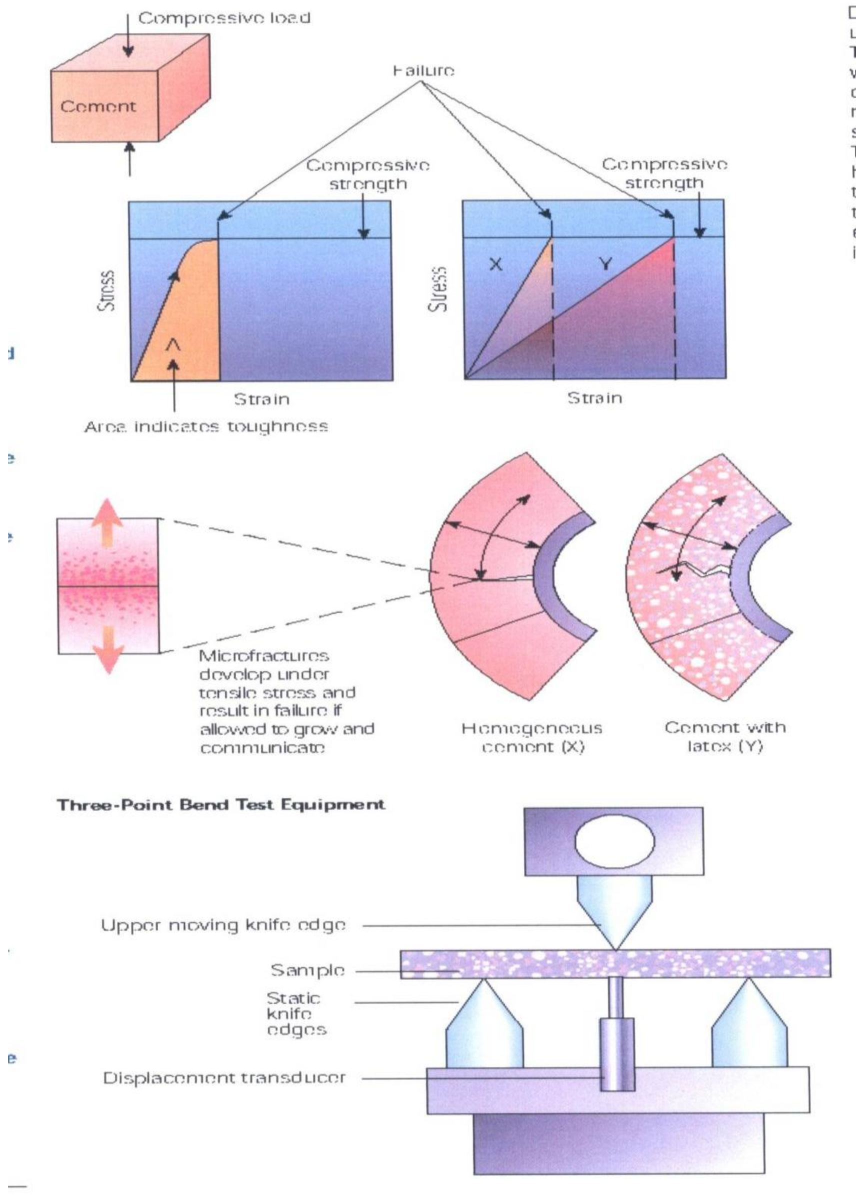

Figure 2-3 Cement Behavior under Compression and Three-point Bend Test (Bonnet and Pafitis, 2001)

2.3 Portland Cement and G Class

Bottomhole well conditions vary each day for oil industry when we go deeper and hotter including deep oceans and tectonically unstable sediments. It is obvious that still best material to create the bond between tubular and formation is a type of Portland cement. Result coming from hydration is most probably insoluble in water, have resistance to pressure and very tight material in order to make real zonal isolation. (Nelson 1990)

Chemistry of Portland cement is based on the anhydrous components. Hydration occurs with different rates and gives different products for each component of Portland cement. Here, our point is to know the one of the mechanisms during the hydration which is volume change of cement body by the change of free water amount in the environment. Inevitably, net volume is going to be decrease for the materials giving the reaction but also final solid matrix will have more porous media compared to initial situation. Net volume change may be positive if we apply the cement job to atmospheric pressure conditions but if you pump this slurry inside a 3000 m. oil well, hydrostatic pressure transmission of cement itself and the lateral compaction of formations will try to squeeze your net volume.

Without adding any special chemicals outside chemical notation can be changed in order to prepare more suitable cement for oil wells. This classification made by API and G class cement will show up as a result of that classification.

The product obtained by grinding Portland cement clinker, consisting essentially of hydraulic calcium silicate usually containing one or more forms of calcium sulfate as an interground additive. No additives other than calcium sulfate or water, or both, shall be interground or blended with the clinker during manufacture of Class G well cement. This product is intended for use as basic well cement. Available in moderate sulfate resistant (MSR) and high sulfate resistant (HSR) grades.

2.4 Free Fluid (Free water)

The optimum water amount in order to hydrate a given cement sample can be chemically proven. Free water amount as an indication of chemical stability of cement itself is not an only chemical behavior but also a result of gravitational segregation due to the hydrostatic pressure occurred by cement itself. (Figure 2-4)

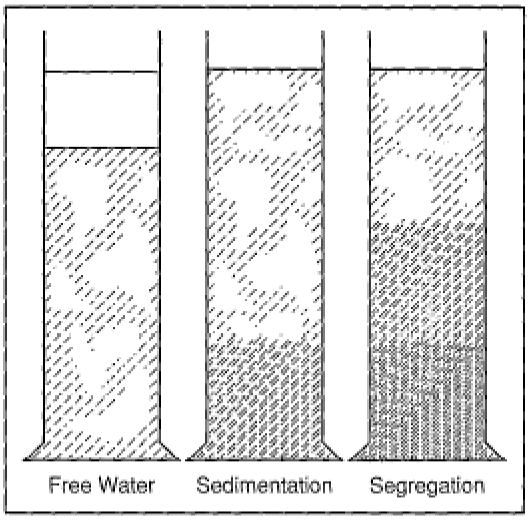

Figure 2-4 Three different cement slurry settling processes

(Nelson, 1990)

CHAPTER 3

STATEMENT OF PROBLEM

Fluid migration through the cement slurry is one of the mutual problems of oil industry. There are several ways for prevent unwanted fluid migration. Using low fluid loss, short transition time behavior and heavy cement slurry is the most conventional way to solve that problem because of all routine tests data's show those three parameters importance.

The aim of this work to control more parameters related to cement slurry behavior by the help of other laboratory tests like free fluid determination and differently sized dry cement samples. Because of the difficulties to validate all well site operational parameters in a laboratory, operational standards are assumed in an acceptable industry standard. The results are compared and try to find a multi control system to prevent gas migration.

This study is also limited with the effect of free fluid against fluid migration just on API G class cement slurries. Other parameters like different fluid loss values and retarding, accelerating effects of chemicals on cement setting time are not taken in consideration.

CHAPTER 4

EXPERIMENTAL SETUP AND PROCEDURE

4.1 Experimental Set-Up

During the experiments SGSA is used to determine the final static gel and transition time. Free fluid test is conducted by following the standard API test procedure.

4.1.1 SGSA (Static Gel Strength Analyzer)

The 5265 Static Gel Strength Cement Analyzer (SGSA) is an instrument that measures the static gel strength of API cement under high temperature and high-pressure conditions. (Figure 4-1) The instrument captures ultrasonic signals that are passed through the sample then performs post processing of the data to determine the static gel strength (SGS) versus time plot. The data is presented graphically as well as being stored in a Microsoft Access database file (.MDB). Each 5265 autoclave connected as part of the SGSA system is equipped with an internal processor board that sends and receives an ultrasonic pulse through the slurry, performs digital signal processing of captured data and, as an option, measures the transit time of the pulse through the slurry.

Operational limits for instrument are 20,000 psig pressure and 400 °F temperatures. (Figure 4-2) Preparations for slurry can be changed due to the design factors. It is obvious to make it with API recommendations but if the user knows the real well site situation for simulation he or she may pass over the atmospheric curing process for slurry.

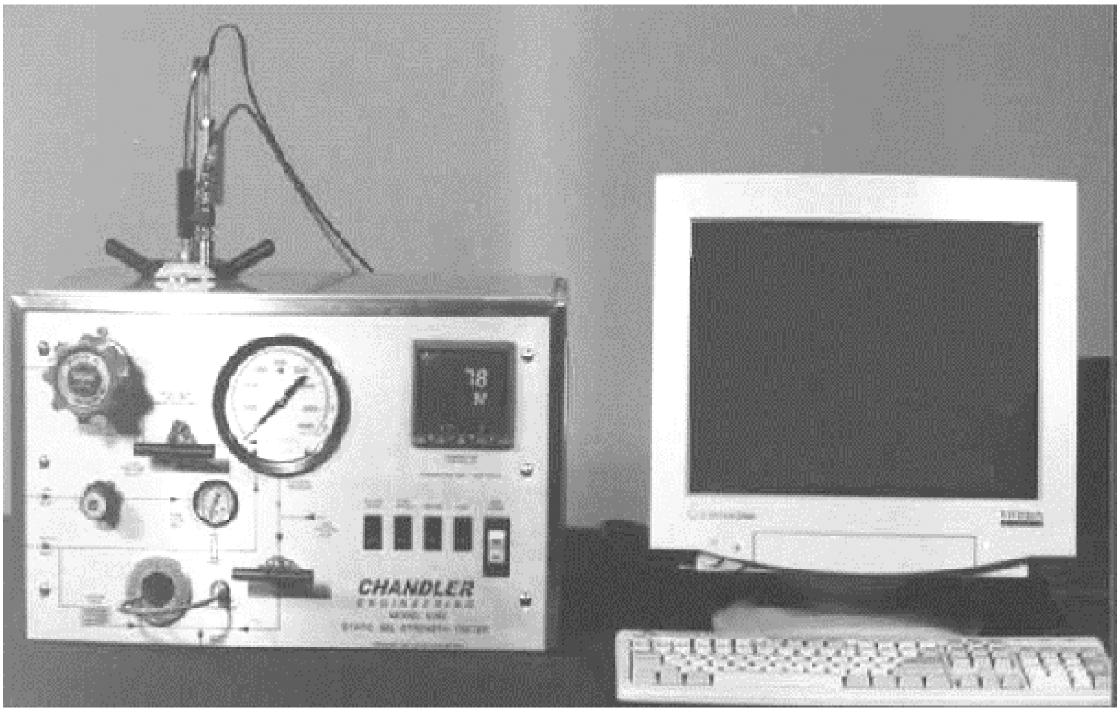

Figure 4-1: Static Gel Strength Analyzer (SGSA)

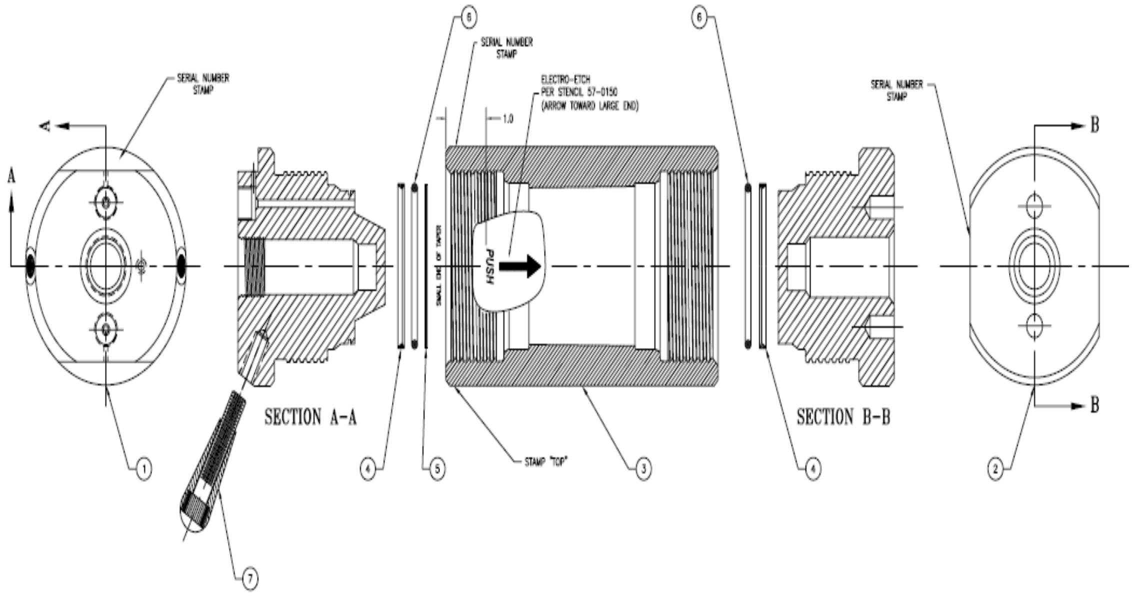

Figure 4-2: Static Gel Strength Analyzer Pressure Vessel

4.1.2 Free Fluid Test (Free Water)

The atmospheric pressure consistometer or the pressurized consistometer shall be used for stirring and conditioning the cement slurry for determination of free-fluid content. The atmospheric consistometer consists of a rotating cylindrical slurry container, equipped with an essentially stationary paddle assembly, in a temperature controlled liquid bath. (Figure 4-3) It shall be capable of maintaining the temperature of the bath at 27°C± 1.7°C (80°F±3°F) and of rotating the slurry container at a speed of 150 r/min. ± 15 r/min. (2.5 r/s±0.25 r/s) during the stirring and conditioning period for the slurry. The paddle and all parts of the slurry container exposed to the slurry shall be constructed of corrosion resistant materials.

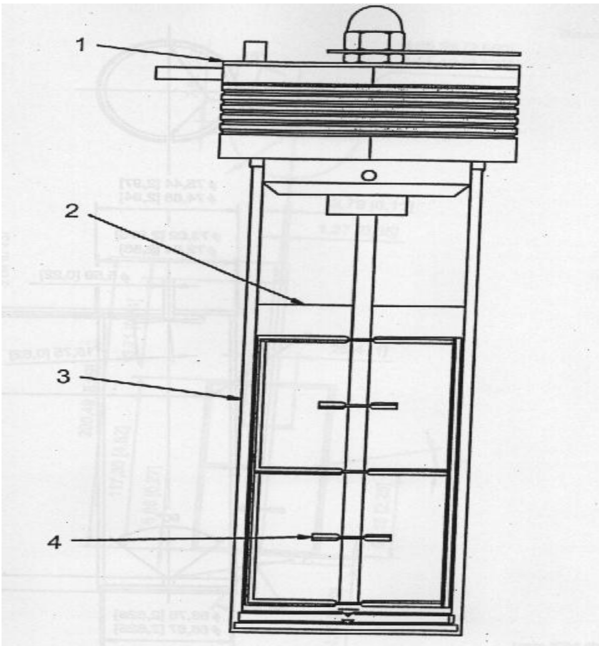

Figure 4-3: Container Assembly for atmospheric consistometer

A 500 ml. conical flask in accordance with ASTM E1404, Type I, and Class 2 shall be used. The temperature of the bath shall be measured by thermometer or thermocouple with digital indicator which are accurate to ±1.7 °C (±3 °F). Thermocouple shall be type J. Thermocouples with digital indicators and thermometers shall be checked for accuracy against a certified thermometer traceable to the reference of the national body responsible for standards of temperature measurement, no less frequently than monthly. The timer shall be accurate to within ±30 s. per hour. It shall be checked for accuracy no less frequently than semi annually.

4.1.2.1 Calculation of free fluid percent

$\% FF = ((V_{FF} * \rho) / m_S) * 100$

Where, %FF is the free fluid content of the slurry, in percent

V_{FF} is the volume of free fluid collected, expressed in millimeters

ρ is the specific gravity of slurry

m_S is the initially recorded mass of slurry, expressed in grams.

4.1.3 Rheological Measurements

The Chandler Engineering Model 3500 viscometer is used to measure the rheological properties of two different compositions of cement slurry samples in order to see any effect of rheology onto the gel readings. The measurement fluid is contained within the annular space or shear gap between the rotor and bob. The rotor is rotated at known velocities (shear rates) and the viscous drag exerted by the test fluid creates torque on the bob. This torque is transmitted to a precision torsion spring, and its deflection is measured and related to shear stress.

4.1.4 HP/HT Consistency

Another key instrument for to prove the cement samples classification is Pressurized Consistometer.

The Pressurized Consistometer incorporates a rotating, cylindrical Slurry Cup equipped with a stationary paddle assembly enclosed in a pressure chamber designed for a working pressure of 170 MPa (25,000 psi) at a maximum temperature of 200°C (400°F). (An air-operated hydraulic pump generates pressure to the cylinder assembly.) The hydraulic system incorporates a reservoir, piping, valves and filters. Heat is supplied to the chamber by a 4000-watt, internal, tubular heater controlled by the automatic temperature control system program. Thermocouples are provided for determining the temperatures of the oil bath and cement slurry. The programmable temperature controller will automatically control the rate of temperature rise of the slurry (like temperature gradient). When the slurry reaches the desired maximum temperature, the controller will hold the slurry temperature at that level. Pressure settings are maintained through the control of a pressure release valve and air pressure available to the pump.

The slurry container is rotated at a constant speed of 150 +/- 15 rpm by a Magnetic Drive. Drive torque is transmitted from a set of outside drive magnets, through a nonmagnetic housing, to permanent magnets attached to the rotating shaft within the cylinder. Permanent, rare earth magnets are used to ensure high torque and a long magnetic-field life. The viscosity (consistency) of the cement slurry is indicated by a meter and is recorded on a chart as a DC voltage obtained from a potentiometer installed within the pressure cylinder. (Figure 4-4) The potentiometer contains a standardized torsion spring, which resists the rotating force of the paddle. Rotational force is proportional to consistency of the cement slurry.

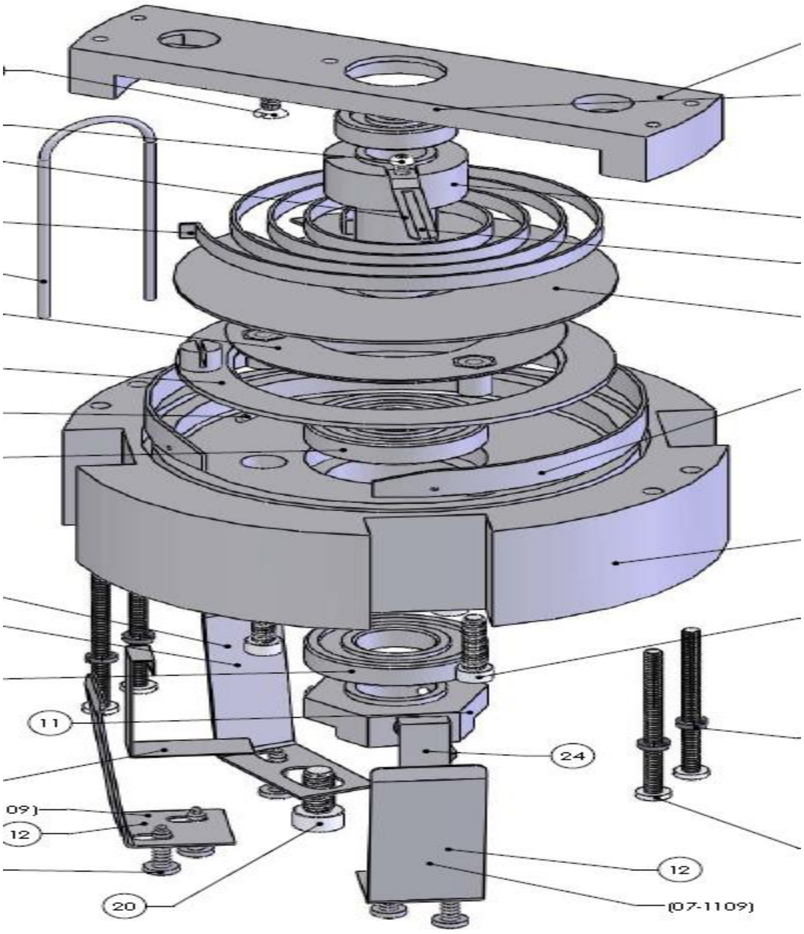

Figure 4-4: Potentiometer Mechanical Assembly

CHAPTER 5

RESULTS AND DISCUSSION

The main scope of this study is to prevent unwanted fluid migration. Cement slurry should consist of relevant properties to create a strength zone against any invasion from wellbore. Before trying to design necessary parameters, we should be sure about the environment that we are working in. Cement slurry physicochemical environment and physical test parameters are so much important. During the tests, chemical composition of the dry cement blend is not investigated since, in the oil industry just measurable properties symbolizes the type of cement as our working environment. G – Class quality tests are held in Cement Quality Laboratory in TPAO Research Center and shown in the table below. (Table 5-1)

Table 5-1: API G Class Cement Quality Test Results

PROPERTIES	**Unit**	**Expected Values**	**TEST RESULTS**		
			Mix G	Bolu	Nuh
Maximum consistency	Bc	30 max.	21	23	18
Thickening time (100 Bc)	Min.	90 min.-120 max.	96	99	107
Compressive Strength					
Atmospheric pressure, @140 °F after 8 hrs curing	psi	>1500	2608	1962	2322
Atmospheric pressure, @100 °F after 8 hrs curing	psi	>300	651	608	735
Free Water	%	5.9 max.	3.2	2.5	5

Moreover basic measurements for SGS value for different cement compositions held in also TPAO Cement Laboratory in order to see the difference against hydrocarbon migration potential by transition time values (100 lb/100 ft^2 to 500 lb/100 ft^2)

As a rule of thumb, gel strength value below 100 lb/100 ft^2 clearly indicates fully transmission of hydrostatic pressure of cement slurry thorough lower parts of the borehole to at least balance the possible formation pore pressure. This does not most probably stop the high pressure hydrocarbon migration, but other parameters in order to interrupt fluid migration are always thought to be ideal including good mud removal, optimized length for stage cement, low fluid loss value for slurry, and low shrinkage rates for onset cement. When the slurry reaches a SGS value of 500 lb/100 ft^2 the solidification process lowers the hydrostatic pressure acting to the bottom of the borehole. Transition time which symbolizes that 100-500 lb/100ft^2 SGS transition period is the key measurable factor for our study in order to be sure about the right angle setting profile.

Free fluid as an independent cement property plays a role here. Different percentages of free fluid for three different types of cement samples were tested and theoretically offering results are reached. Less unreacted reactants inside the chemical environment means more stable reaction. Free fluid in that situation symbolizes unreacted water and may create unstable reactant-product balance. Intuitively, somebody may think about if the amount of reactants for a G class hydration why that free water will cause an unstable reaction. The answer is hidden under the complexity of hydration process, wellbore physics and temperature effect. The homogeneous onset cement column in a 3000 m. well is just a dream.

5.1 SGS Analyzer Tests

Three different types of G Class cement is used during the tests. As mentioned before a real well data taken from TPAO Thrace region well is applied as temperature (BHST: 155 °F) and pressure (BHP: 3000 psi) data. Cement placement time is selected as 4 hours in order to achieve real operation time.

According to test results; Bolu cement slurry with fluid loss control and rheology control agents shows the minimum transition time for fluid migration potential. Nuh cement slurry has a broad time gap (transition time) when same fluid loss and rheology control chemicals used in Bolu cement sample. Mix G cement has a moderate free fluid percentage %3.12 when compared to Nuh and Bolu %5, %2.5 respectively, and has also median transition time results when fluid loss control-rheology control and moderate rheology control-without fluid loss control compositions used as expected. It was observed that, increasing amount of free fluid percentage increases the potential for fluid migration (Table 5-2). On the other hand when adverse effects of chemicals eliminated and only cement+ water slurries are used all three samples have close transition time results. (Figure 5-2A-J)

Table 5-2: SGS Test Results (* Repeated Experiment Values)

Composition	BHST (°F)	BHP (psi)	Transit time (min) (100-500 lb/100ft^2)		Final SGS time (1200 lb/100ft^2)	Final Compressive Strength (psi)
Mix G + %44 tap water + %0.7 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl	155 °F	3000 psi	77 min.	82 min*.	07:29	3144
Nuh + %44 tap water + %0.7 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl	155 °F	3000 psi	256 min.	175 min.*	16:10	2180
Bolu + %44 tap water + %0.7 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl	155 °F	3000 psi	55 min.	56 min.*	06:55	2350
Mix G + %44 tap water + %0.3 D-65 + %0.3 D-59 + % 3 KCl	155 °F	3000 psi	59 min.		02:53	3481
Nuh + %44 tap water + %0.3 D-65 + %0.3 D-59 + % 3 KCl	155 °F	3000 psi	63 min.		02:43	3924
Bolu + %44 tap water + %0.3 D-65 + %0.3 D-59 + % 3 KCl	155 °F	3000 psi	40 min.		02:43	2653
Mix G+ %44 tap water	155 °F	3000 psi	73 min.	67 min.*	02:24	3551
Nuh + %44 tap water	155 °F	3000 psi	39 min.	44 min.*	02:12	3489
Bolu+ %44 tap water	155 °F	3000 psi	39 min.	66 min.*	02:44	2556
Mix G + %44 tap water + %0.2 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl	155 °F	3000 psi	33 min.		03:42	3464
Nuh + %44 tap water + %0.2 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl	155 °F	3000 psi	27 min.		04:12	3291
Bolu + %44 tap water + %0.2 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl	155 °F	3000 psi	33 min.		03:06	3378
Mix G + %44 tap water + %1 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl	155 °F	3000 psi	49 min.		10:06	2739
Nuh + %44 tap water + %1 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl	155 °F	3000 psi	207 min.		17:34	1607
Bolu + %44 tap water + %1 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl	155 °F	3000 psi	63 min.		06:54	2456

During all experiments total test time limited with 24 hours in order to see the final compressive strength. Reaching to final SGS takes less time compared to total test schedule but it is beneficial to see the final strength regarding with free water. SGSA test results for different samples based on different rheological properties are shown in the Appendix B (Figure 5-2K-P). Also repeatability results for 6 different situations are shown in the Appendix C. (Figure 5-2R-Y)

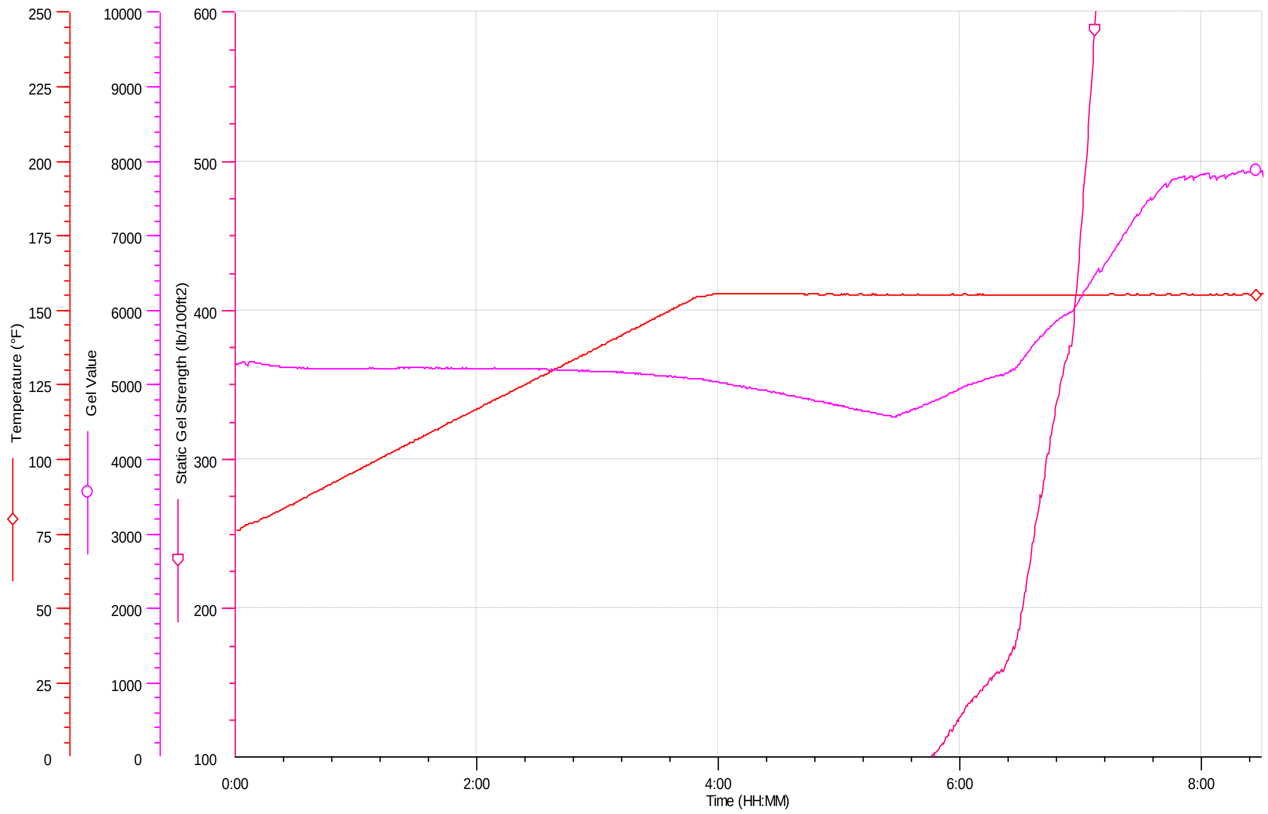

Figure 5-2.A: SGS test for Mix G + %44 tap water + %0.7 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl

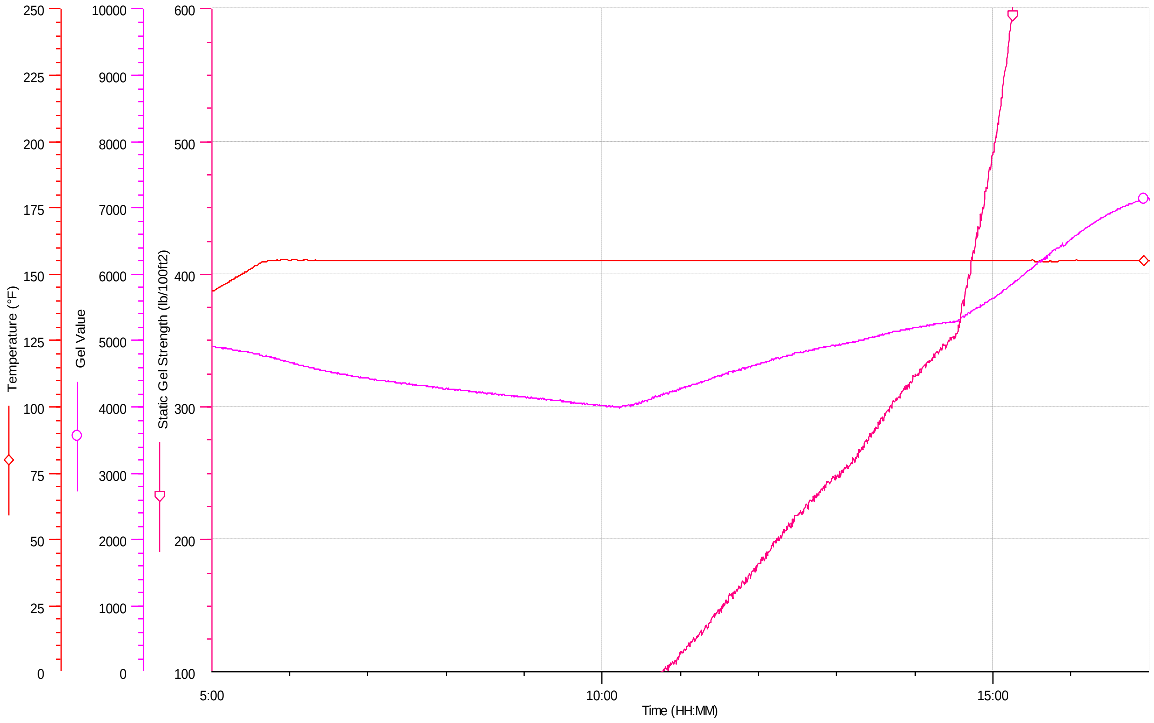

Figure 5-2.B: SGS test for Nuh + %44 tap water + %0.7 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCL

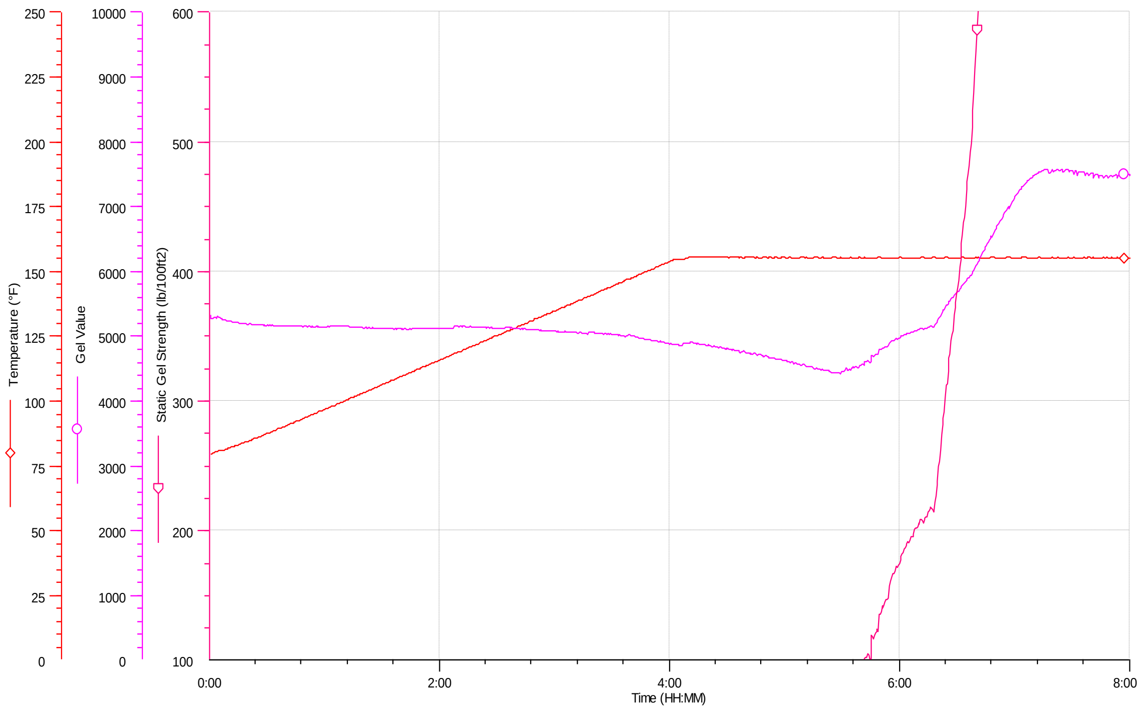

Figure 5-2.C: SGS test for Bolu + %44 tap water + %0.7 D-65 + %0.6 D-59 + 0.05 D-28 + % 3 KCl

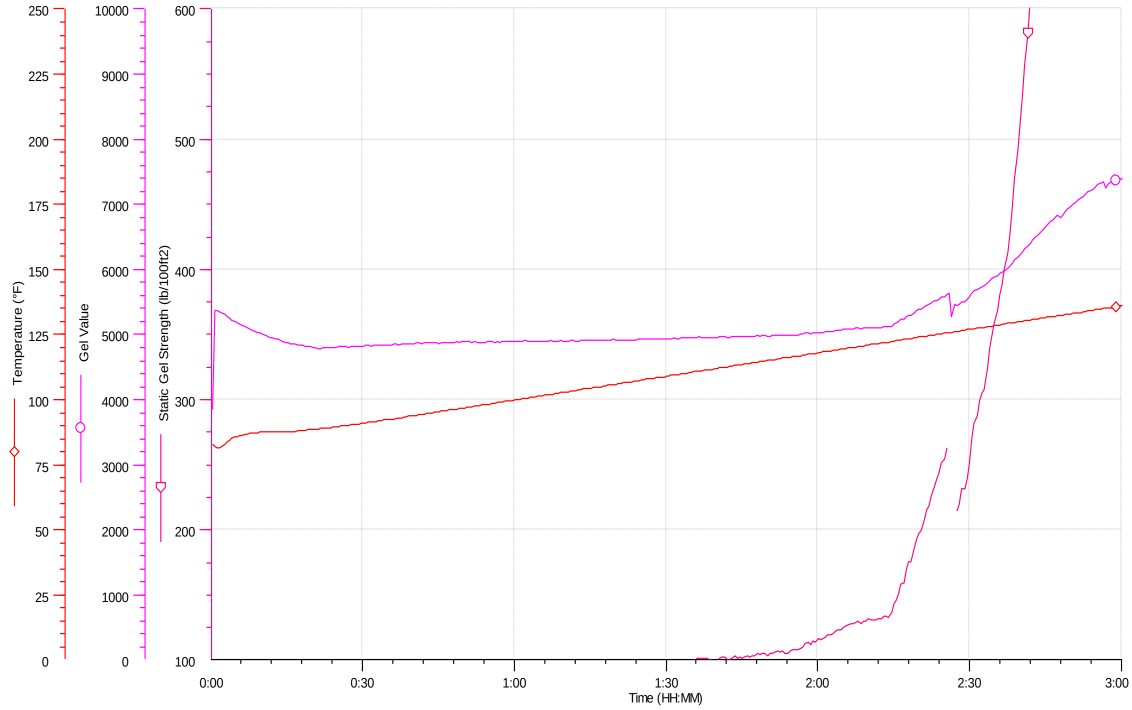

Figure 5-2.D: SGS test for Mix G + %44 tap water + %0.3 D-65 + %0.3 D-59 + % 3 KCl

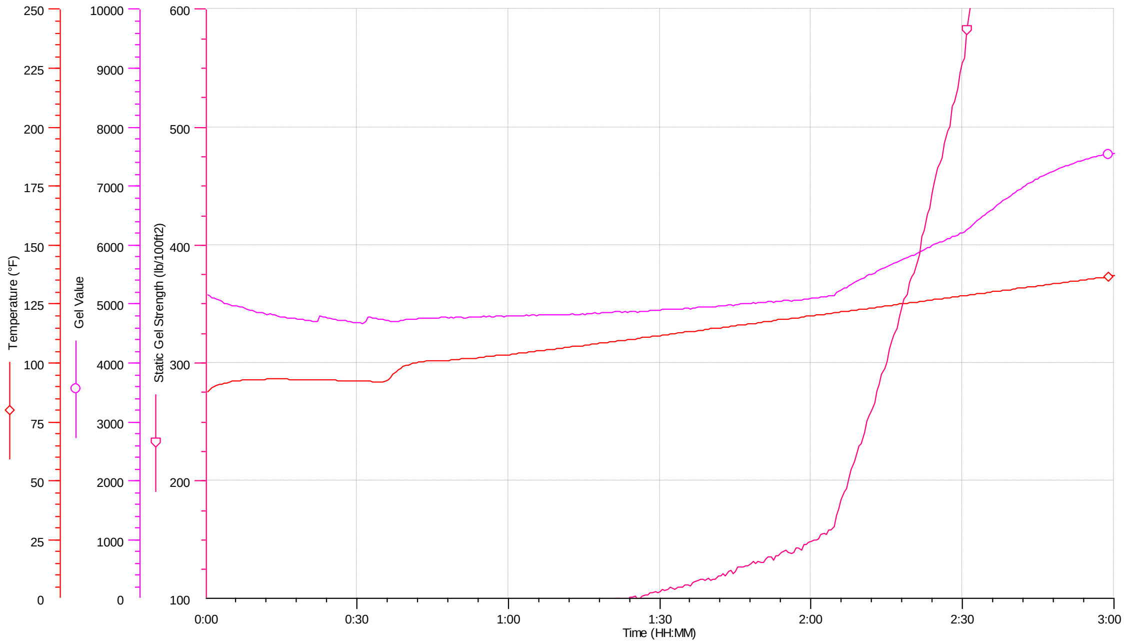

Figure 5-2.E: SGS test for Nuh + %44 tap water + %0.3 D-65 + %0.3 D-59 + % 3 KCl

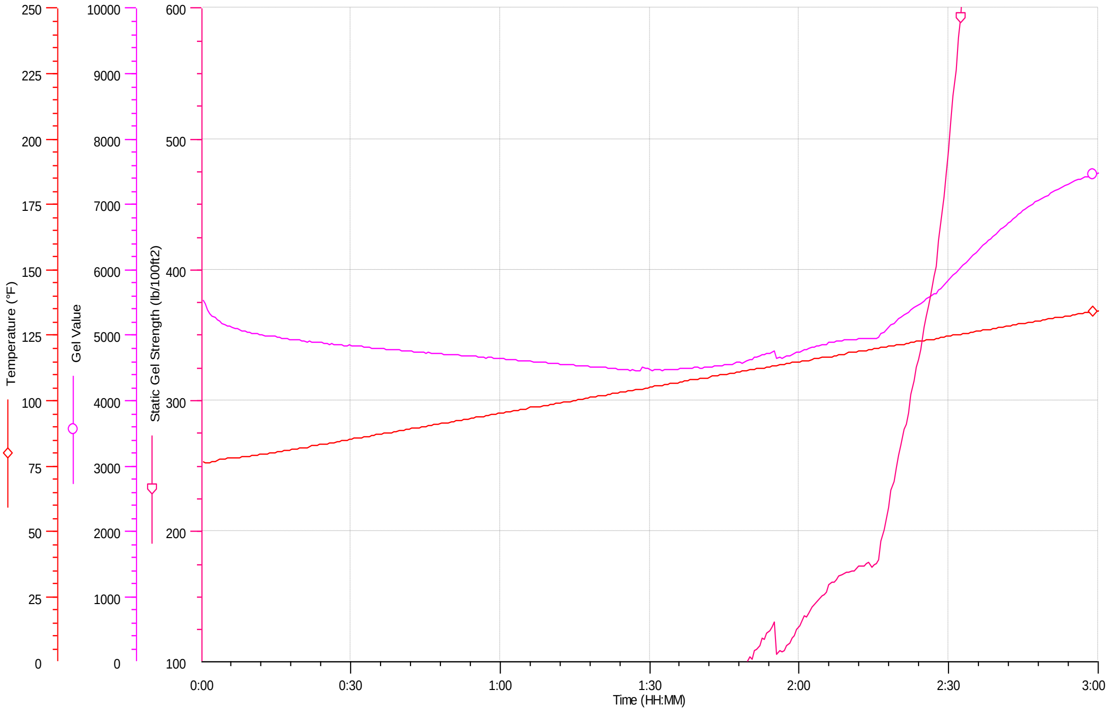

Figure 5-2.F: SGS test for Bolu + %44 tap water + %0.3 D-65 + %0.3 D-59 + % 3 KCl

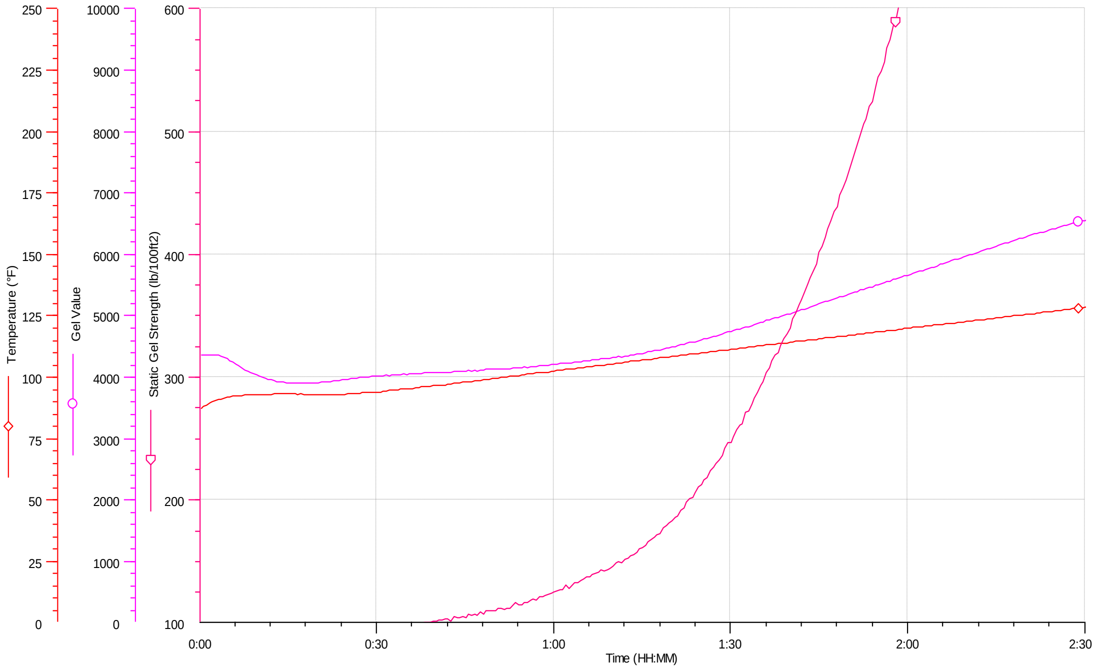

Figure 5-2.H: SGS test for Mix G+ %44 tap water

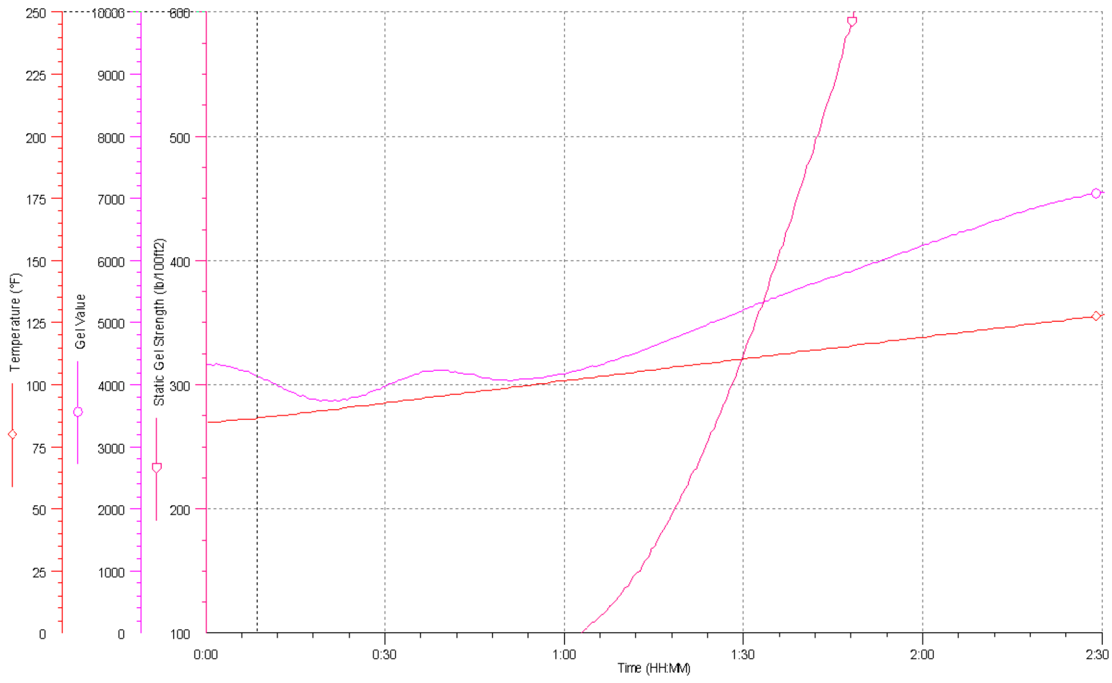

Figure 5-2.I: SGS test for Nuh + %44 tap water

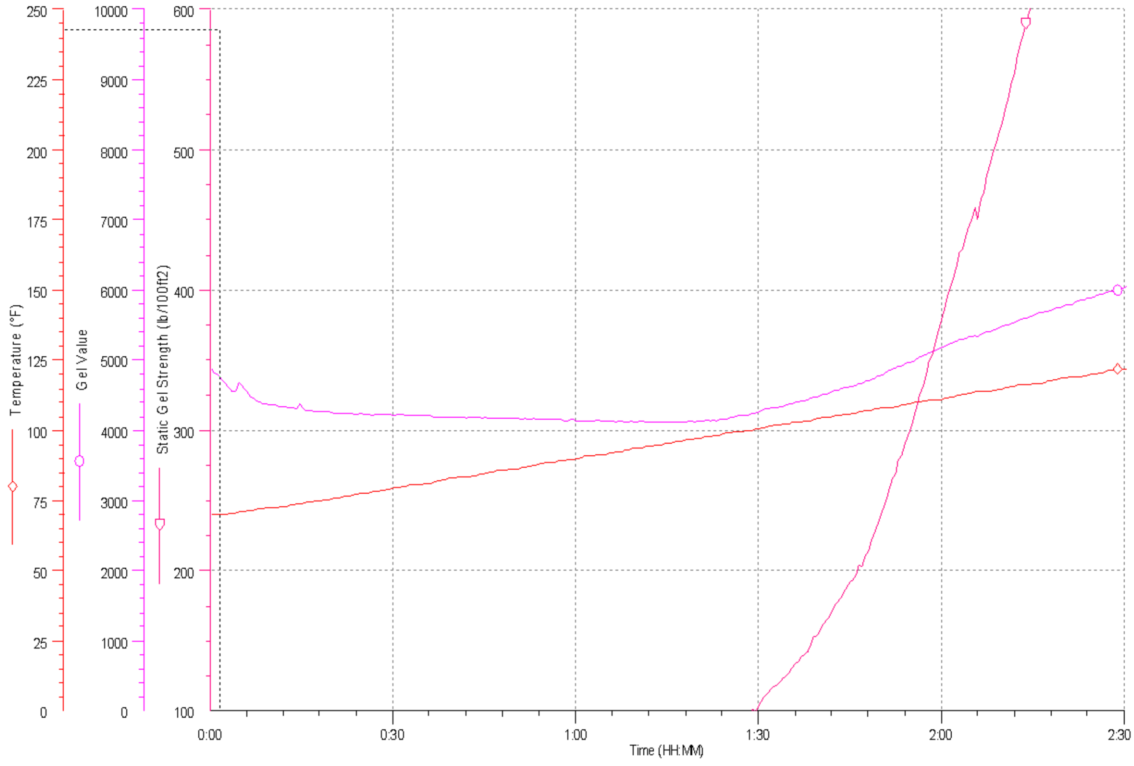

Figure 5-2.J: SGS test for Bolu + %44 tap water

5.2 Free Fluid Measurements

The following results were achieved from free fluid tests according to API Spec 10A mentioned procedures in Experimental Set-up and Procedures part

Bolu Cement: % 2.5

Mix G Cement: %3.12

Nuh Cement: %5

5.3 Rheological Measurements

Rheology of cement slurry is a property that can be measurable and meaningful for the first stage of the cement mixtures. Staging is inevitable for a cement+water+chemical mixture, since it has always three different phases until the total solidification. For the first stage like we measures rheological properties, slurry behaves as a liquid structure that can flow. Semi-solid stage which is always needed to understand, symbolizes second one. Last and the third is total solid stage which has totally different properties like strength, porosity, permeability, heat conductivity and perhaps plasticity.

In our tests in order to justify that some properties we see for the first stage do not mean anything for the second stage. Rheology is a property like this. Intuitively someone may think that if a slurry composition has high rheological properties including gel, it will stops hydrostatic transmission early and leads a possible migration from pore spaces. But tests show that migration can be related with some properties but only after the transition time starts. The tables below show the different rheological measurements for same cement slurry samples. (Table 5-3-8)

Table 5-3: Nuh + %44 tap water + %0.2 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl

Viscometer speed (rpm)	300	200	100	6	3	Gel
Readings (Chan3500)	**249**	**184**	**113**	**25**	**21**	**17/39**

Table 5-4: Bolu + %44 tap water + %0.2 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl

Viscometer speed (rpm)	300	200	100	6	3	Gel
Readings (Chan3500)	**300+**	**300+**	**220**	**104**	**100**	**95/111**

Table 5-5: Mix G + %44 tap water + %0.2 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl

Viscometer speed (rpm)	300	200	100	6	3	Gel
Readings (Chan3500)	**300+**	**270**	**205**	**115**	**93**	**93/113**

Table 5-6: Nuh + %44 tap water + %1 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl

Viscometer speed (rpm)	300	200	100	6	3	Gel
Readings (Chan3500)	**257**	**188**	**109**	**18**	**15**	**17/30**

Table 5-7: Bolu + %44 tap water + %1 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl

Viscometer speed (rpm)	300	200	100	6	3	Gel
Readings (Chan3500)	**270**	**207**	**128**	**34**	**32**	**34/60**

Table 5-8: Mix G + %44 tap water + %1 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl

Viscometer speed (rpm)	300	200	100	6	3	Gel
Readings (Chan3500)	**279**	**207**	**125**	**25**	**19**	**22/43**

CHAPTER 6

CONCLUSION

During this study, three different cement samples are tested in order to measure the fluid migration capacity of each one for defined wellbore temperature and pressure. As a main property “free fluid” for each sample is key factor to make a difference. The experiments were carried out in TPAO Research Center. The results are analyzed and following conclusions drawn from this study.

- It was observed that free fluid percentages of cement slurries varied from %2.5 to %5 including %3.12.

- For %44 water + %0.6 fluid loss agent + %0.7 dispersant + %0.05 retarder + %3 KCl composition ; Bolu cement sample with lowest free fluid percentage %2.5 lowers the capacity of cement sheath against unwanted fluid migration compared with Nuh cement and Mix G cement samples with free fluid percentages %5, %3.12 respectively.

- Without fluid loss control but with rheology control to create pumpable cement slurry again Bolu cement sample with lowest free fluid percentage value plays a positive role.

- There is no significant effect of rheological properties on fluid migration capacity of cement slurry.

- Final compressive strength values varied from 1607 psi to 3924 psi but no significant relation with transition time and compressive strength observed.

- It is obvious that in the case of high percentage free water cement sample used there is an adverse effect of more chemicals (in concentration) on transition time. (e.g. Nuh cement)

CHAPTER 7

RECOMMENDATIONS

This study is step to understand the effect of free fluid parameter on the setting profile of cement slurry and fluid migration capacity. On the other hand further studies are recommended in order to see the real case for gas migration cut capacity of different cement slurries in a pressurized vessel that can be feed by a outside measurable gas source, such as:

- Although different chemical compositions were tested, more tests can be conducted in order to fulfill parametric study standards.
- Different API classes cement should be tested.
- Further tests should be conducted for varying formation temperatures and pressures.
- Different operational schedule might be tested, including the temperature profile.

REFERENCES

American Petroleum Institute; API Specification for Materials and Testing for Well Cements, Latest Edition 2002; Dallas,TX.

Bahramian Y., Movahadenia A., and Rasaei M.R. "Prediction Slurry Permeability, *k*, Using Static Gel Strength, Fluid Loss Value, and Particle Size Distribution" paper SPE 106983 presented at SPE Production and Operations Symposium held in Oklahoma City, Oklahoma, U.S.A., 31 March-3 April 2007.

Bouras H., Doumaz N., Sonatrach, Bellabarba M., Caceres E., Mananga S., Leroy-Delage S., Aziz M.G.A., and Benberner M.R., "Responsive Cementing Material Prevents annular Leaks in Gas Wells" paper SPE 116757 presented at 2008 SPE Annual Technical Conference and Exhibition held in Denver, Colorado, U.S.A., 21-24 September 2008.

Bour D.L., and East L.E., "Expansion: Anti-Fluid Migration Technology Solves South Texas Fluid Migration" paper IADC/SPE 17259 presented at 1988 IADC SPE Drilling Conference held in Dallas, Texas, February 28- March 2, 1988.

Brooks R., Newberry J., Cook P., and Wendlinger F. "Preventing Annular Gas Flow in Conjunction with 2 Stage Cementing" paper SPE 116447 presented at 2008 SPE Asia Pacific Oil & Gas Conference and Exhibition held in Perth, Australia, 20-22 October 2008.

Cavanagh P., Johnson C.R., LeRoy-Delage S., DeBruijn G., Cooper I., Guillot D., Bulte H., and Dargaud B. "Self-Healing Cement- Novel Technology to Achieve Leak-Free Wells" paper SPE/IADC 105781 presented at 2007 SPE/IADC Drilling Conference held in Amsterdam, The Netherlands, and 20-22 February 2007

Coker O.D., Harris K.L., and Williams T.A., "Preventing Shallow Gas Migration in Offshore Wells: The Performance of Lead Cements" paper SPE 24978 presented at European Petroleum Conference held in Cannes, France, 16-18 November 1992.

Dean G.D., and Brennen M.A. "A Unique Laboratory Gas Flow Model Reveals Insight to Predict Gas Migration in Cement" paper SPE 24049 presented at Western Region Meeting held in Bakersfield, California, March 30-April 1, 1992.

Dusterhoft D., Wilson G., and Newman K., "Field Study on the Use of Cement Pulsation to Control Gas Migration" paper SPE 75689 presented at SPE Gas Technology Symposium held in Calgary, Alberta, Canada, 30 April-2 May 2002.

GCHEM Ltd. http://www.gchem.ca, 14th September, 2010

Gonzalo V., Aiskely B., Alicia C. "A Methodology to Evaluate Gas Migration in Cement Slurries" paper SPE 94901 presented at 2005 SPE Latin American and Caribbean Petroleum Engineering Conference held in Rio de Janerio, Brazil, 20-23 June 2005.

Harris K.L., Ravi K.M., King D.S., Wilkonson J.G., and Faul R.R. "Verification of Slurry Response Number Evaluation Method for Gas Migration Control" paper SPE 20450 presented at 65th Annual Technical Conference and Exhibition of the SPE held in New Orleans, LA, September 23-26, 1990.

Jennings S.S., Al-Ansari A.A., and Al-Yami A.S., "Gas Migration after Cementing Greatly Reduced" paper SPE 81414 presented at 13th Middle East Oil Show & Conference held in Bahrain, 5-8 April, 2003.

Jones R.R., and Carpenter R.B., "New Latex, Expanding Thixotropic Cement Systems Improve Job Performance and Reduce Costs" paper SPE 21010 presented at International Symposium on Oilfield Chemistry held in Anaheim California, February 20-22, 1991

Mata F., Diaz C., and Villa H., “Ultra lightweight and Gas Migration Slurries: An Excellent Solution for Gas Wells” paper SPE 102220 presented at 2006 SPE Annual Technical Conference and Exhibition held in San Antonio, Texas, U.S.A., 24-27 September, 2006.

Moon J., and Wang S., “Acoustic Method for Determining the Static Gel Strength of Slurries” paper SPE 55650 presented at 1999 SPE Rocky Mountain Regional Meeting held in Gilette, Wyoming, 15-18 May 1999.

Moroni N., Panciera N., Zanchi A., Stogit, Johnson C.R., LeRoy-Delage S., Bulte-Loyer H., Cantini S., Belleggia E., and Illuminati R. “Overcoming the Weak Leak in Cemented Hydraulic Isolation.” paper SPE 110523 presented at 2007 SPE Annual Technical Conference and Exhibition held in Anaheim, California, U.S.A., 11-14 November 2007.

Nelson, E.B.: “Well Cementing” Developments in Petroleum Science, 1990, 1-1, 1-6, B-7, B-8, 2-1, 2-2, 2-9

Reddy B.R., Xu Y., Ravi K., Grey D., and Pattillo P.D. “Cement Shrinkage Measurement in Oil Well Cementing- a Comparative Study of Laboratory Measurements and Procedures” paper SPE 103610 presented at 2007 SPE Rocky Mountain Oil & Gas Technology Symposium held in Colorado, Denver, U.S.A., and 16-18 April 2007.

Rogers M.J., Dillenbeck R.L., and Eid R.N., “Transition Time of Cement Slurries, Definitions and Misconceptions, Related to Annular Fluid Migration” paper SPE 90829 presented at SPE Annual Technical Conference and Exhibition held in Houston, Texas, U.S.A., 26-29 September 2004.

Santra, A., Reddy B.R. and Antia M. “Designing Cement Slurries for Preventing Formation Fluid Influx after Placement” paper SPE 106006 presented at the 2007 SPE International Symposium on Oilfield Chemistry held in Houston, Texas, U.S.A., 28 February-2 March 2007..

Seidel F.A., and Greene T.G., "Use of Expanding Cement Improves Bonding and Aids in Eliminating Annular Gas Migration in Hobbs Grayburg-San Andreas Wells" paper SPE 14434 presented at 60th Annual Technical Conference and Exhibition held in Las Vegas, Nevada, September 22-25, 1985.

Stone W.H., and Christian W.W., "The Inability of Unset Cement to Control Formation Pressure" paper SPE 4783 presented at AIME Symposium on Formation Damage Control held in New Orleans, LA, February 7-8, 1974.

Sutton D.L., and Ravi K.M., "New Method for Determining Downhole Properties that Affect Gas Migration and Annular Sealing" paper SPE 19520 presented at 64th annual Technical Conference and Exhibition of the SPE held in San Antonio, TX, October 8-11, 1992.

APPENDIX A

EXPERIMENTAL PROCEDURES FOR SGSA, API FREE FLUID TEST AND HP/HT CONSISTOMETER

Static Gel Strength Analyzer instrument has a 20,000 psig pressure and 400 °F temperatures limit, so that without precautions and relevant laboratory working conditions, it could be harmful for human health. All personal protection equipments for laboratory conditions must be used before the test run. Moreover, it is recommended that an auxiliary technician or helper needed for test run since, the cell for the test is heavier than a human handling capacity with one hand.

Preparing the Cell and the Sample for the SGSA Test

The recommended procedure for preparing the test cell and slurry for testing are outlined in the following steps:

1. Always check the sealing components to make sure they are clean and in good condition. If the O-rings are deformed or hardened replace them.

2. Apply a light coating of high temperature grease inside the cylinder up to the threads and on each plug up to the threads. This will prevent cement from sticking to the metal and will make cleanup easier.

3. Place the bottom plug in a vice. Install seal ring and O-ring. Apply a thin coat of high temperature grease on the O-ring and sealing ring.

4. Screw the cylinder onto the bottom plug located in the vise with the cylinder end marked **T** at the top. It is recommended that the plug be screwed in by hand and that the plug be tightened so that it just contacts the cylinder. Further tightening after the plug has contacted the cylinder will not cause more effective sealing, and will cause plug removal difficulty.

5. Install the transducers in the top and bottom plug using the spring and transducer support plug. Always place a thin coating of high temperature ultrasonic couplant on the sensor each time it is removed. Use the couplant sparingly. Excessive buildup of couplant can lead to instrument malfunction.

6. Mix the slurry for the test in accordance with API Spec 10 procedures. Approximately 200 mL of slurry is required to fill the cell.

7. Pour approximately 200cc of cement slurry into the greased test cell. Be careful not to get cement into the threads. If cement sets up in the threads it may make plug removal and installation difficult.

8. Continue to pour cement in test cell until level is 1/4 inch (6mm) below the circular lip in the cylinder. Use the Slurry Level Gauge to obtain the proper fill level. The slurry should touch the lower tab marked **WET** but not touch the upper tab marked **DRY**. Do not overfill the test cell, or cement will be forced into the pressure and/or thermocouple ports, and plugs them.

9. Use a small amount of water to continue filling the cell up to the water fill line

indicated on the slurry level gauge.

10. Screw the top plug into the top of the cylinder. It is recommended that the plug be screwed in by hand and that the plug be tightened so that it just contacts the cylinder. Further tightening after the plug has contacted the cylinder will not cause more effective sealing, and will cause plug removal difficulty.

11. The test cell is ready to be installed in the heating jacket.

12. Wipe the cylinder assembly clean and place in autoclave chamber. Carefully guide the bottom transducer cable through the bottom of the heater assembly and out the front panel of the instrument. Be certain that the cylinder is not sitting on the cable. Do not crimp the cable in any way since it can change the signal characteristics. Connect the cable to the connector on the front panel.

13. Align the pressure port in the top plug with the high-pressure filter on top of the autoclave assembly. Rotate the cell clockwise to align.

14. Position the filter with the arrows (located on the side) pointing in the downward position and attach the short end of the U-tube connection to the top of the filter

15. Attach the filter assembly into the bulkhead located on top of the instrument.

16. Connect the longer end of the tube into the top of the cylinder. Hand-tighten initially to start the threads then use a 5/8" wrench to tighten.

17. Connect the top transducer cable to the BNC connector labeled **Top Transducer** at the back of the autoclave.

18. Install the thermocouple in the other high-pressure port in the top plug. Hand tightens only.

19. Connect the thermocouple cable to the receptacle labeled **J Thermocouple** at the back of the autoclave.

20. Turn ON the water supply switch until water exits the thermocouple vent hole. Tighten the thermocouple connection using a 5/8-inch wrench.

21. The test cell and autoclave are now ready to begin a compressive strength test.

Running a test

1. Make certain the test cell is installed properly, the HIGH PRESSURE INLET port on the rear of the instrument is plugged, the PUMP switch is in the OFF position, the PUMP WATER valve is turned to the ON position, and the instrument is supplied with compressed air.
2. Turn the PUMP PRESSURE ADJUST regulator clockwise until air pressure is sufficient to raise pressure to the desired pressure set point. Each 5 psig (34.5 kPa) air pressure results in approximately 1000 psig (6895 kPa) hydraulic pressure. The air pressure should not exceed 100 psig (690 kPa). Note that the pump may not be capable of achieving pressures in excess of 16,000 psig without using heat to expand the fluid and increase pressure.
3. Turn the Relief Valve knob clockwise until the release pressure is sufficient to prevent the relief valve from opening at the desired pressure set point.
4. Turn the PUMP switch to the ON position until pressure exceeds the desired set point. Turn the PUMP switch to the OFF position. Make certain the system is holding pressure before proceeding.
5. Turn the Relief Valve knob counterclockwise slowly until the test cell pressure begins to drop. Continue turning the regulator knob slowly until the pressure in the test cell equals the upper limit of the desired test pressure.
6. Turn the PUMP PRESSURE ADJUST regulator counterclockwise until the air pressure is approximately zero.
7. Turn the PUMP switch to the ON position.
8. Slowly turn the PUMP PRESSURE ADJUST regulator knob clockwise until the pump begins to stroke. Continue to slowly turn the regulator knob clockwise until the lower limit for the control pressure is achieved. Note that failure to apply any pressure to the slurry may result in a loss of transit time signal through the slurry.

All the tests during the thesis work conducted at 155°F BHST and 3000 psi BHP

BHST value is taken from a real well data drilled in Thrace region by TPAO completed as a gas producing well at a depth of 1585 m.

Procedure or Free Fluid Test

1. Fill a clean and dry consistometer slurry container to the proper level.
2. Assemble the slurry container and associated parts. Place the container into atmospheric consistometer and start the mixing by starting motor. Time interval between loading the slurry to container and starting the mixing procedure shall not be more than 1 min.
3. Stir the slurry in the consistometer for a period of 20 min.±30 s. Maintain the bath temperature at 27 °C±1.7°C (80°F±3°F)
4. Transfer 760 g ± 5 g of G Class slurry directly into conical flask within 1 min. Record the actual mass transferred. Seal the flask with a self sealing film to prevent evaporation.
5. Set the slurry filled flask on a surface nominally level. The temperature of the laboratory should be 22.8 °C ± 2.8 °C. Then leave the slurry filled flask undisturbed for 2h ± 5 min.
6. At the end of 2 h., remove the supernatant fluid that has developed with a pipette or syringe. Measure the volume of supernatant fluid to an accuracy of ± 0.1 ml and record it as ml. free fluid.

HP/HT Consistency Test Run

Air pressure, temperature, and oil viscosity will all have a significant effect on the time required to fill and drain the cylinder. Optimum air pressure is 100 psi. For example, with a 60 psi air supply, your fill time will be doubled and the drain time tripled over those obtainable with a 120 psi air supply. Low ambient air temperature will have a similar effect. At 45°F, expect the fill time to double and the drain time to be triple of those at 70°F.

1. Turn the Power switch ON.
2. Attach the long bail through the holes on the top of the prepared slurry cup and insert it into the test cell, rotating it until the bottom pins engage the cup drive table. Remove the bail.

3. After the slurry cup is loaded into the cell, the potentiometer mechanism (pot. mech.) is pushed onto the slurry cup paddle shaft and the test cell contact pins. Attach the short bail to the top of the potentiometer and lower the pot mech into the test cell. When properly engaged, the top of the paddle shaft will be flush with the top of the torque measurement potentiometer bearing. Remove the bail.

4. Check to be certain that the slurry cup and pot mech are properly engaged. Turn the Motor switch to ON. No rubbing noise should be heard. The Model 7025C10 is supplied with two types of O-Ring seals for the cylinder plug. (See drawing 08-0280, item 18.)

• The **viton** O-Ring (C09762) is suitable only for low temperature/pressure tests **below** 20,000 psi (138 Mpa), or 275°F (135°C).

• The **metal** O-Ring (P-4080) is suitable for testing at any rated temperature or pressure.

5. Close the pressure cylinder by swinging the Swivel Arm Assembly and plug, vertically above the cylinder, lowering the plug until the tapered threads engage. Screw the plug down until it is firmly engaged. In order to assure that the cylinder will operate **at the maximum rated working pressure and temperature**, we recommend that you work the plug down until the line up mark on the plug matches the mark on the cylinder. Never run a test with the line up mark on the plug tightened down past the mark on the cylinder. Under these conditions, the plug may not unscrew from the cylinder without damaging the threads or plug handles.

6. Slide the thermocouple through the test cell plug into the slurry cup paddle shaft. Start the threads of the sealing gland into the test cell plug, but do not tighten the thermocouple at this time. Verify that the thermocouple is plugged in.

7. Next, fill the test cell with oil. To accomplish this, close the Pressure Release Valve, turn the Oil Supply switch to ON, and turn the AIR control switch to the FILL position. When oil escapes from the top thermocouple high-pressure fitting, tighten the sealing gland with a 5/8” wrench. Leave the Oil Supply switch in the ON position, and the AIR control switch in the FILL position during the test.

8. To apply the initial pressure to the test cell on a Model 7025C10 without pressure

control, turn the Pump Switch to the MANUAL position. When the pressure reaches the desired level turn the Pump Switch to the OFF position. Adjust the pressure as required throughout the test by turning the Pump Switch to MANUAL to increase pressure or by slowly cracking open the Pressure Release Valve to relieve pressure. Use care to open the Pressure Release Valve slowly when attempting to bleed pressure.

9. Turn the Heater Switch to the ON position, the Pump Switch to the AUTO position (Model 7025C10 with pressure control), and start the timer. (The heater and pump will not start until the program start up is initiated through the controller.)

10. To begin the test, the Temperature Controller and Pressure Controller (if equipped) programs must be started as follows.

11. Press the round Auto/Man button on the lower left side to place the controller in the Auto mode. Press the round Run/Hold button on the lower right side to start the program. The OP1 light should begin flashing indicating the control output to the heater and or pump. After the final temperature is reached for the schedule being run, the controller will continue on a programmed soak until the schedule is completed.

APPENDIX B

SGSA TEST RESULTS FOR DIFFERENT RHEOLOGICAL PROPERTIES

The following figures for SGSA test are scaled down intentionally in order to see the exact comparison between the transition time values. The actual test time is 24 hours.

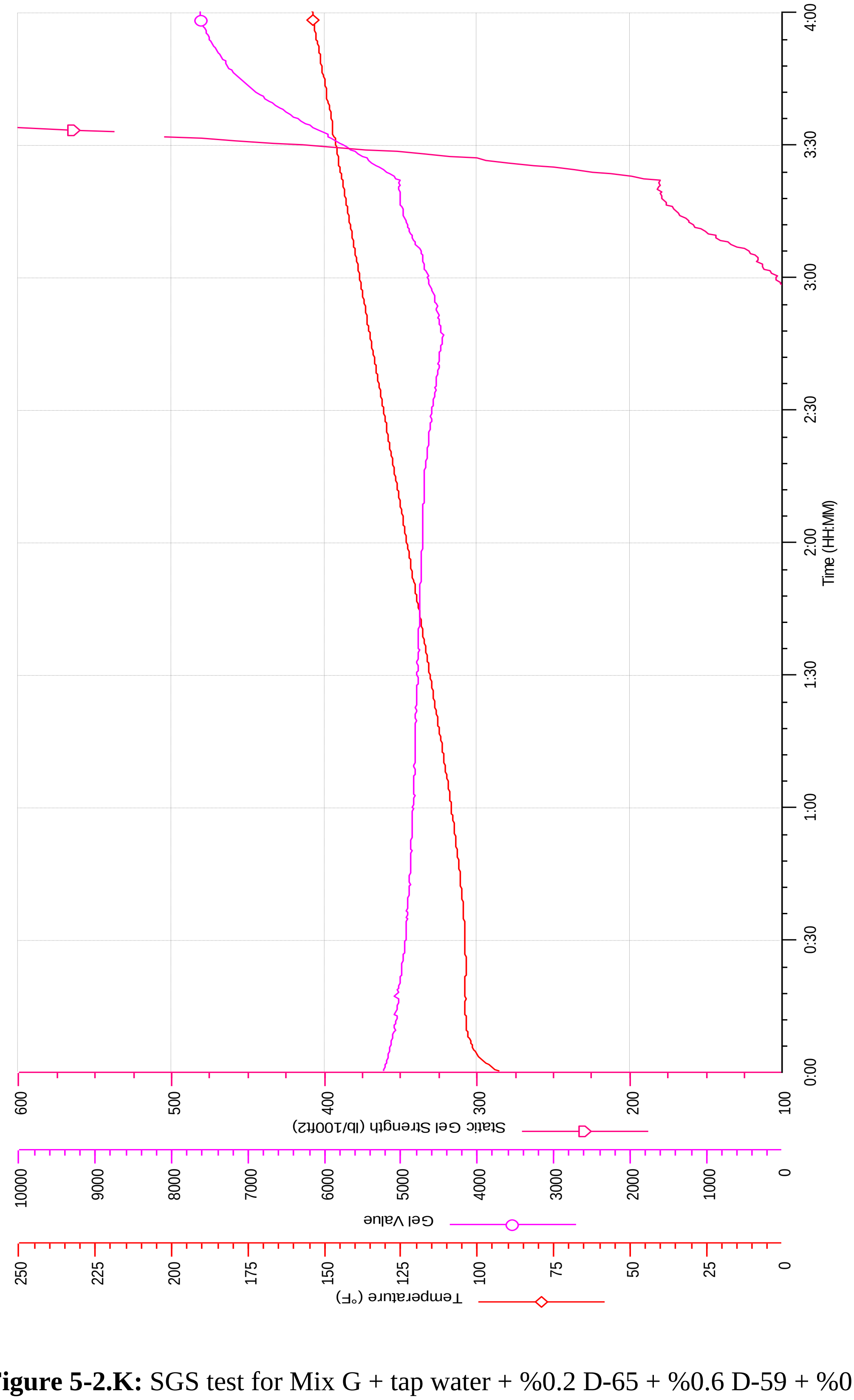

Figure 5-2.K: SGS test for Mix G + tap water + %0.2 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl

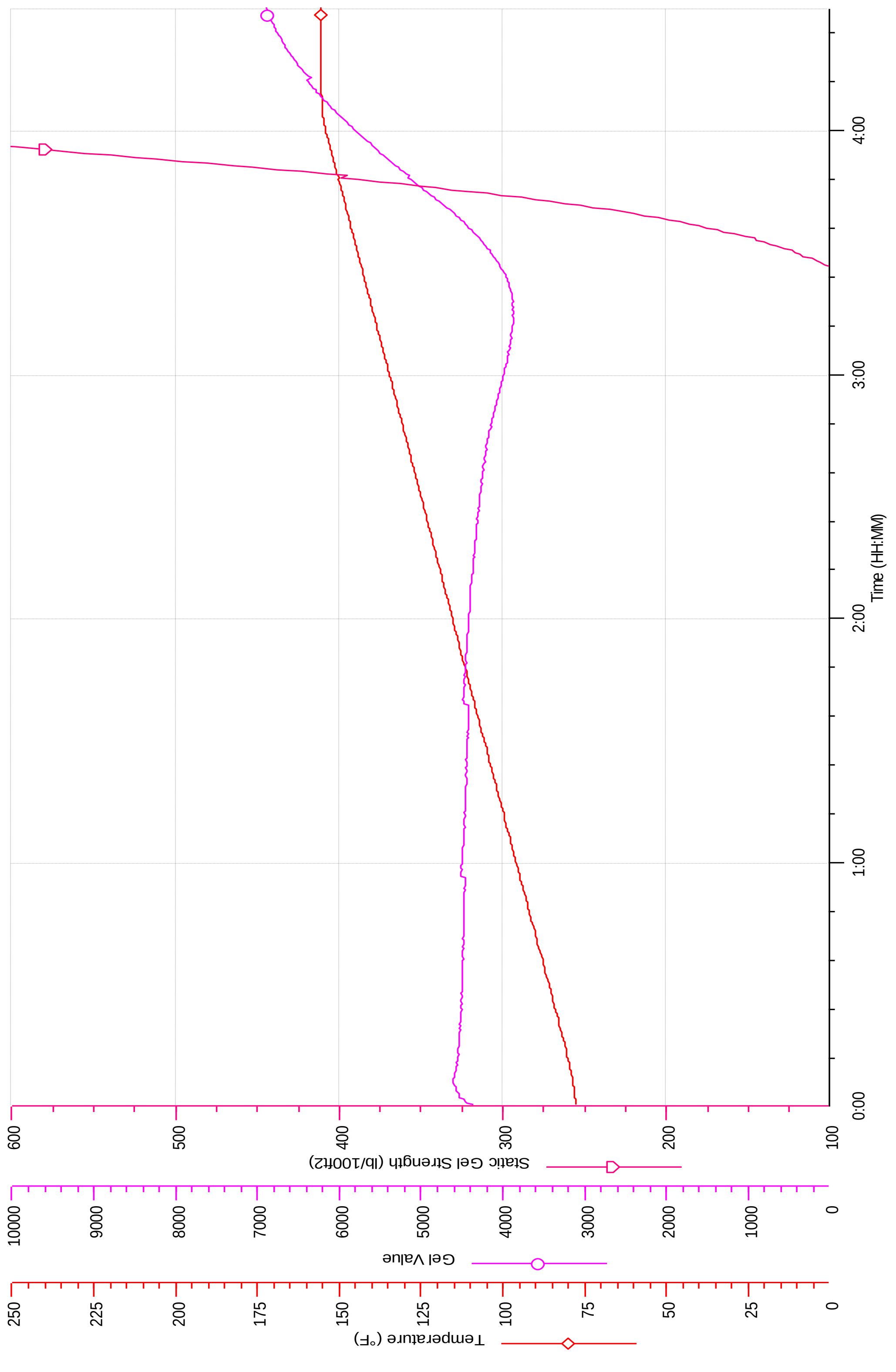

Figure 5-2.L: SGS test for Nuh + %44 tap water + %0.2 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl

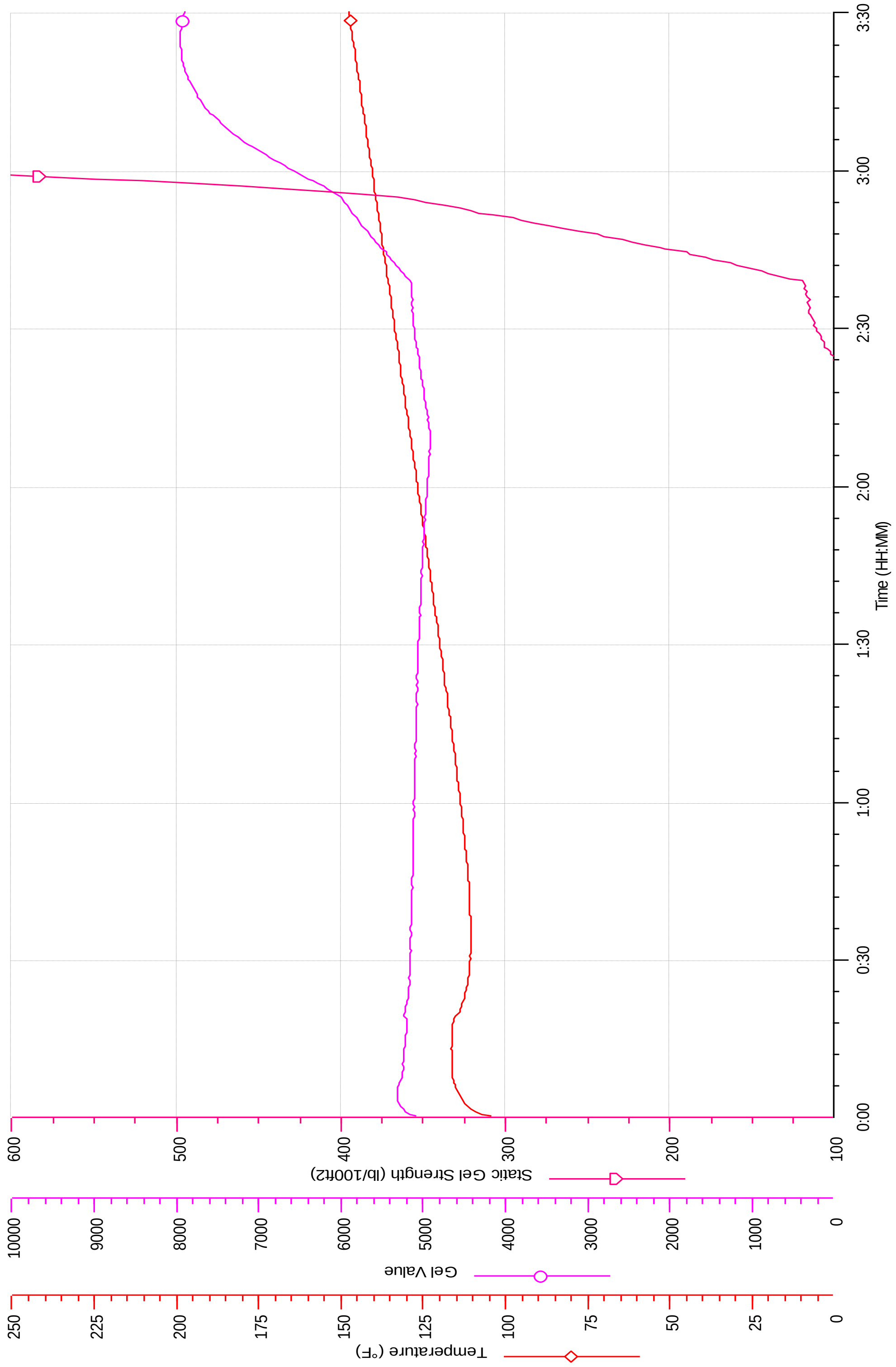

Figure 5-2.M: SGS test for Bolu + %44 tap water + %0.2 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl

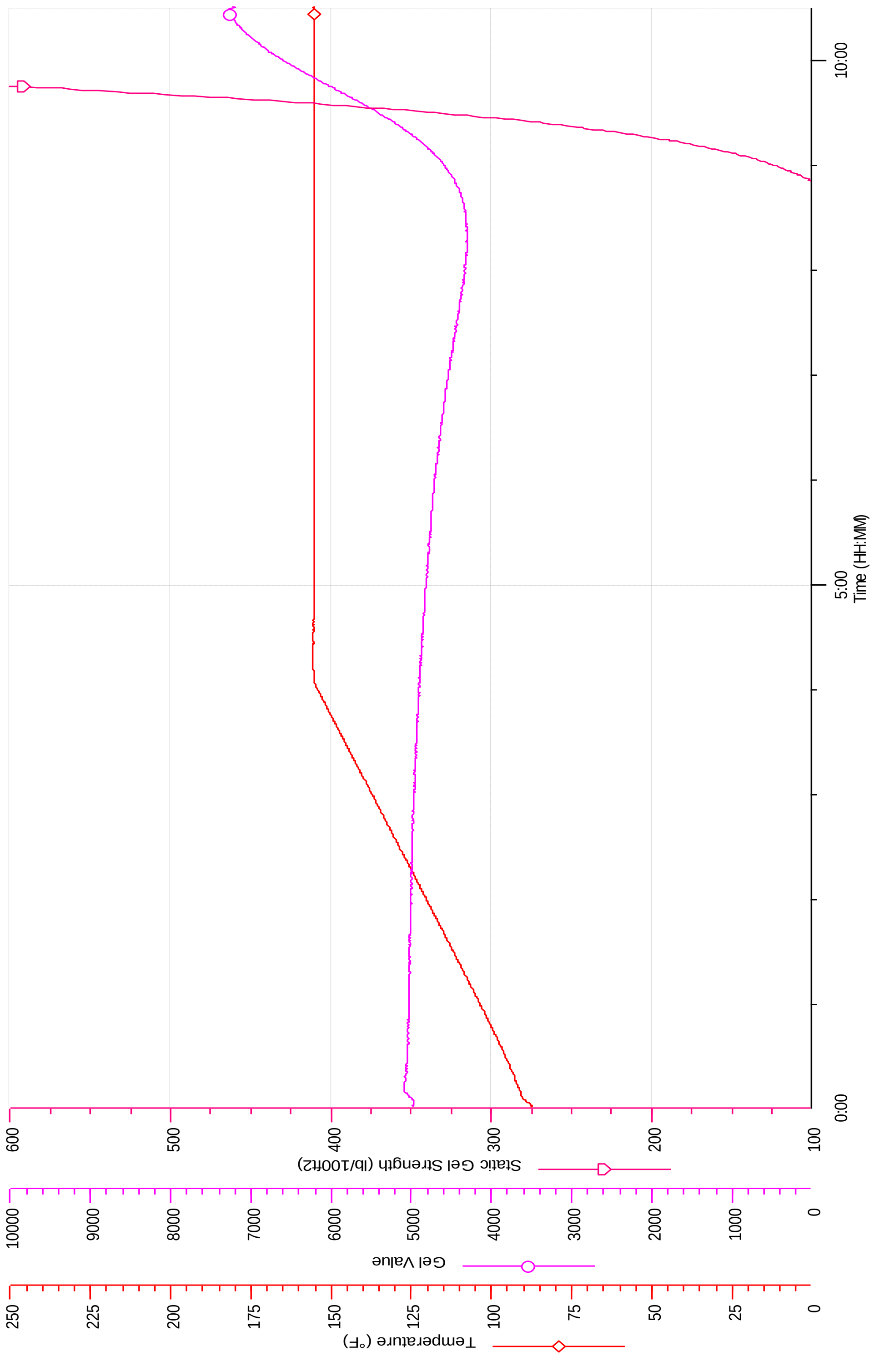

Figure 5-2.N: SGS test for Mix G + %44 tap water + %1 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl

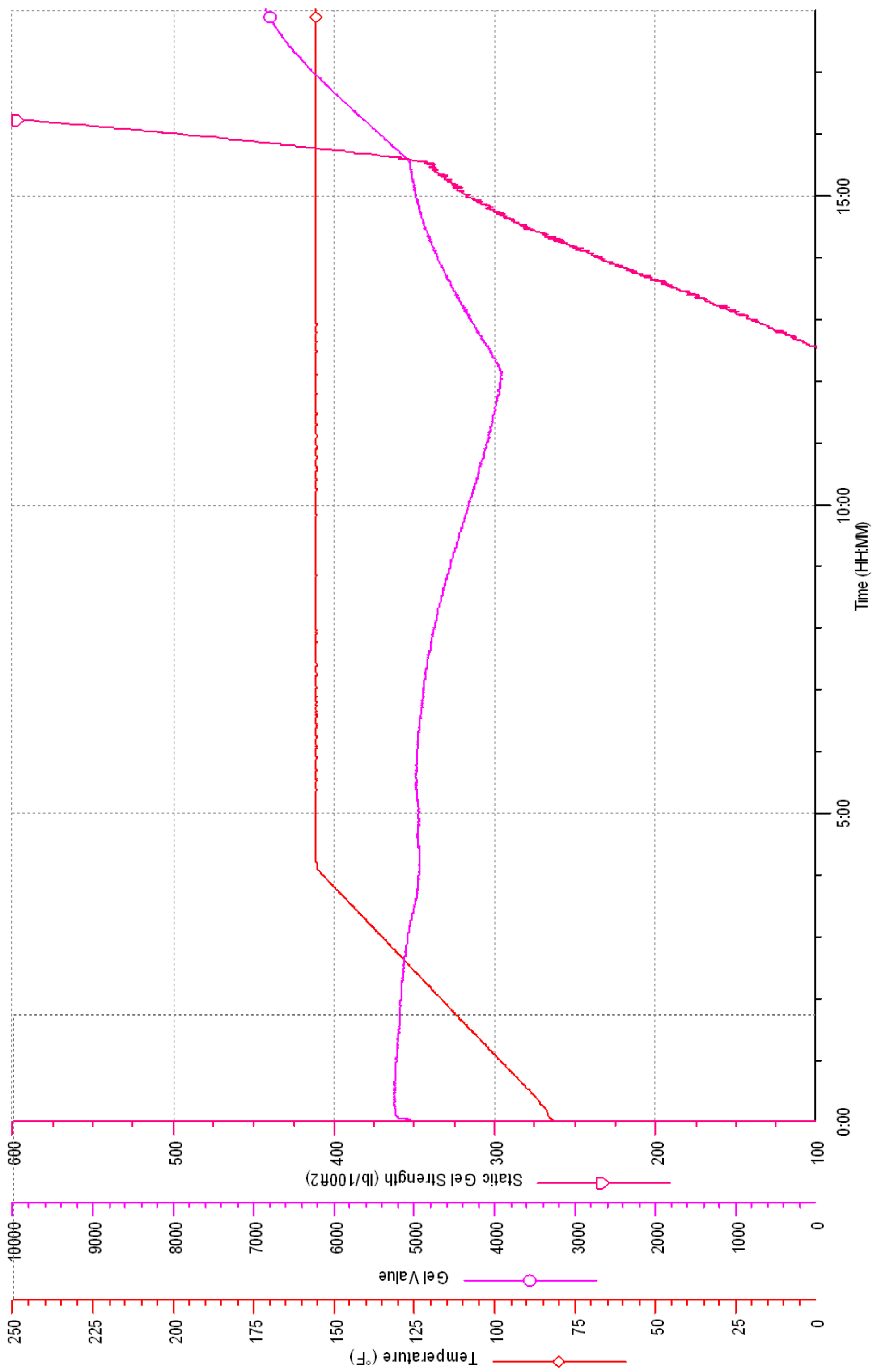

Figure 5-2.O: SGS test for Nuh + %44 tap water + %1 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl

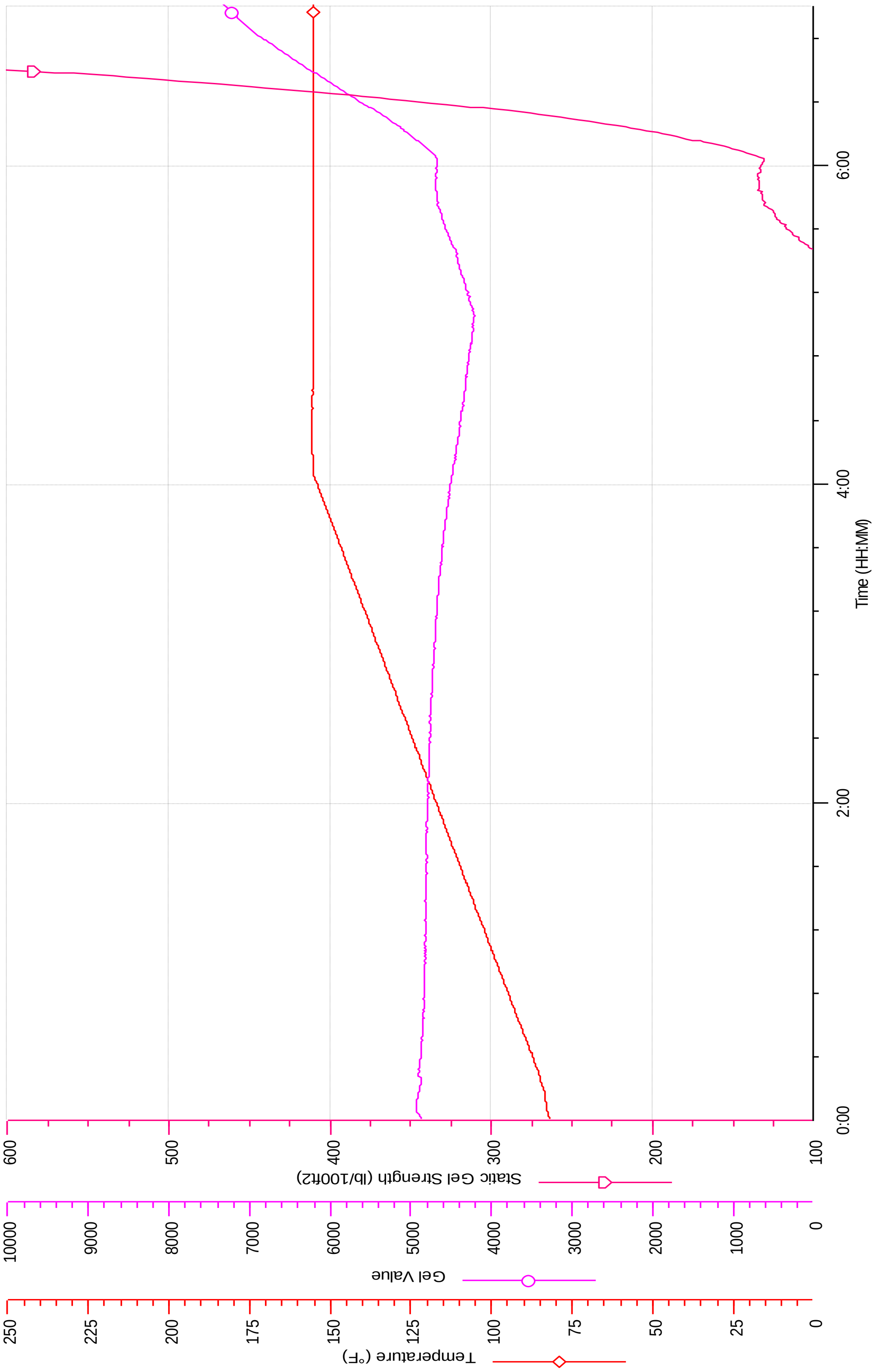

Figure 5-2.P: SGS test for Bolu + %44 tap water + %1 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl

APPENDIX C

SGSA RESULTS FOR REPEATED TESTS

Repeated tests were done exactly with the same samples and for same physical conditions. Although storage conditions for dry cement samples are suitable, some differences might be occurred because of homogeneity of cement samples that was produced in the production facility.

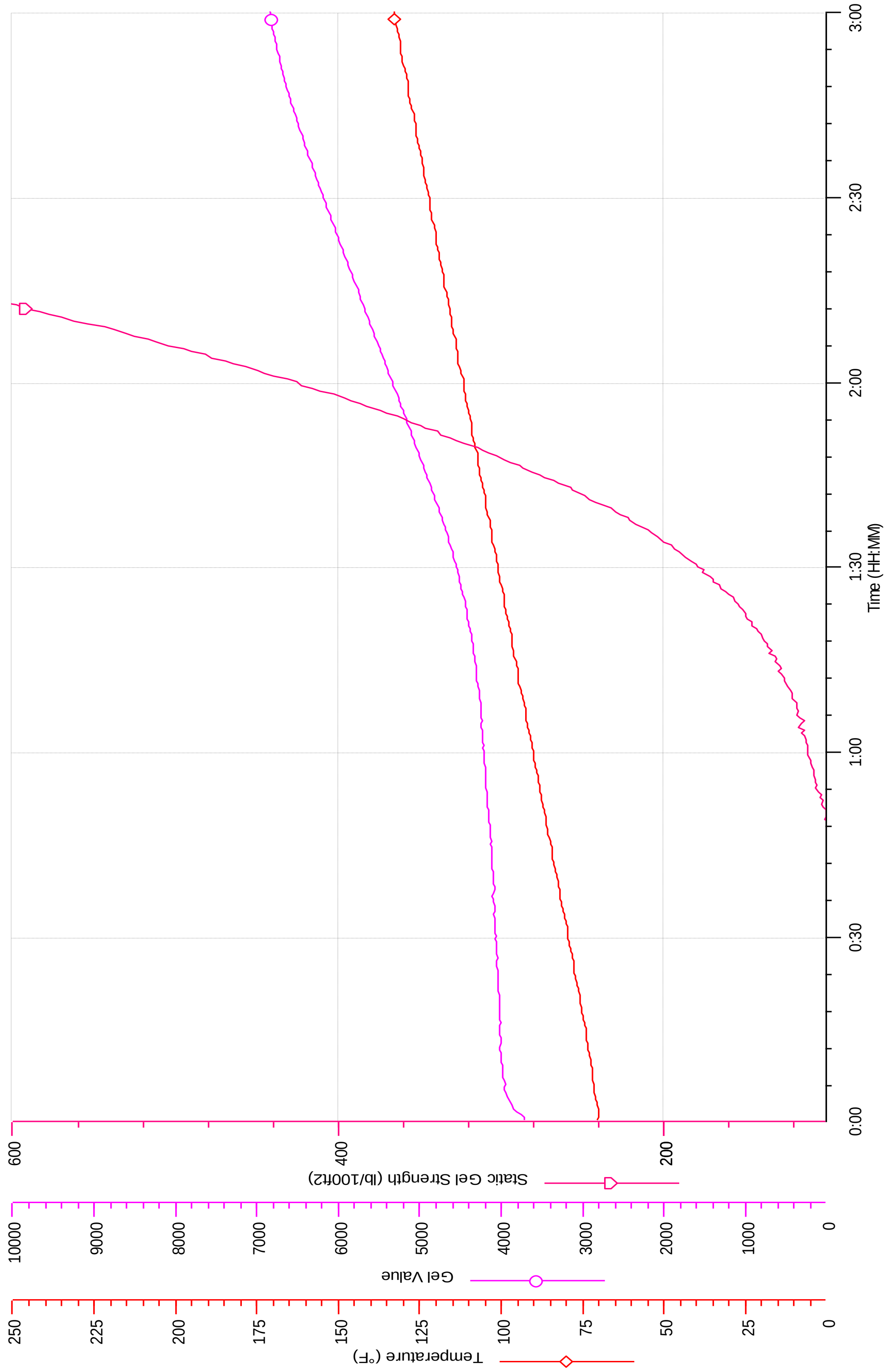

Figure 5-2.R: SGS test for Bolu + %44 tap water

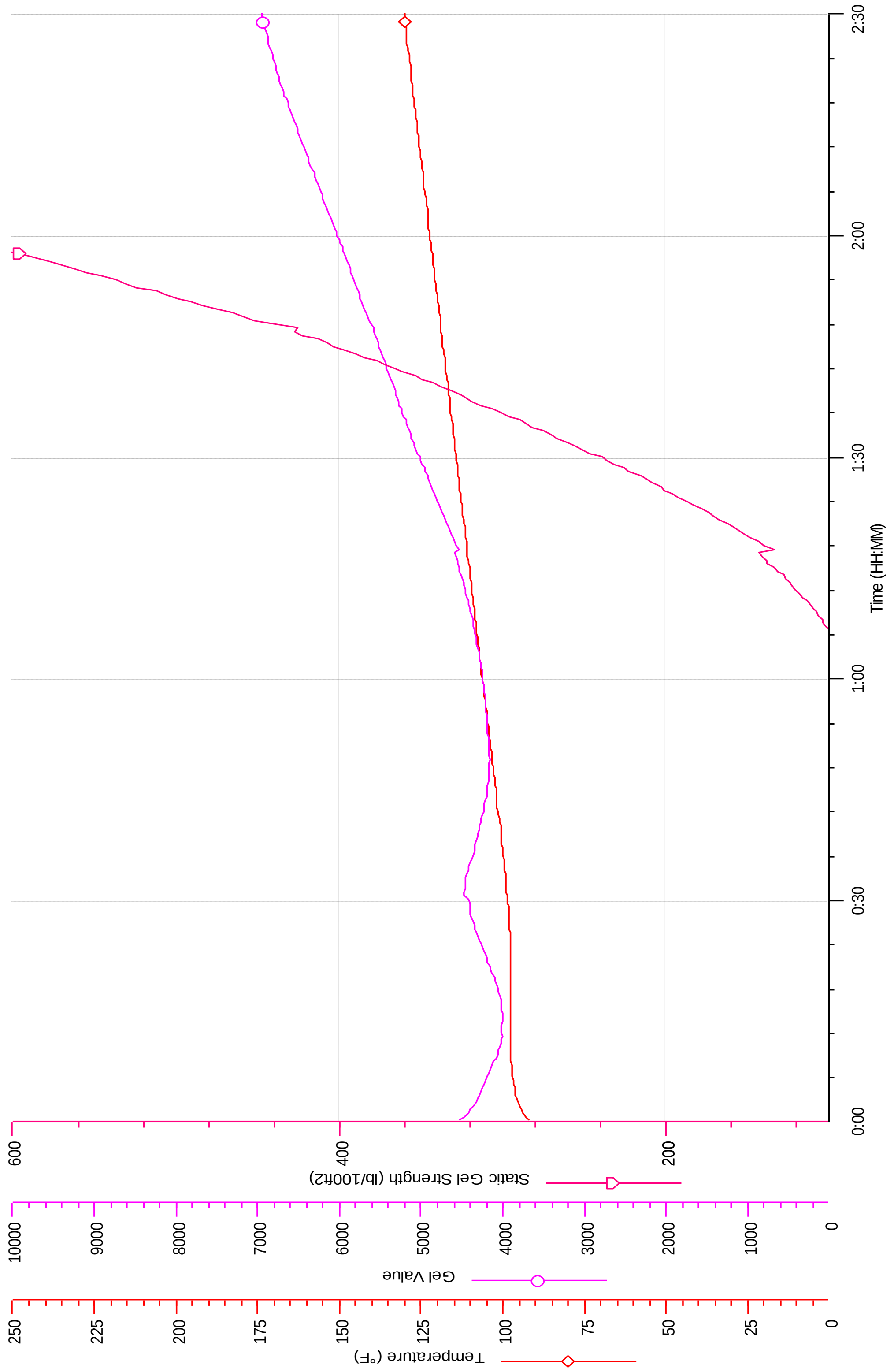

Figure 5-2.S: SGS test for Nuh + %44 tap water

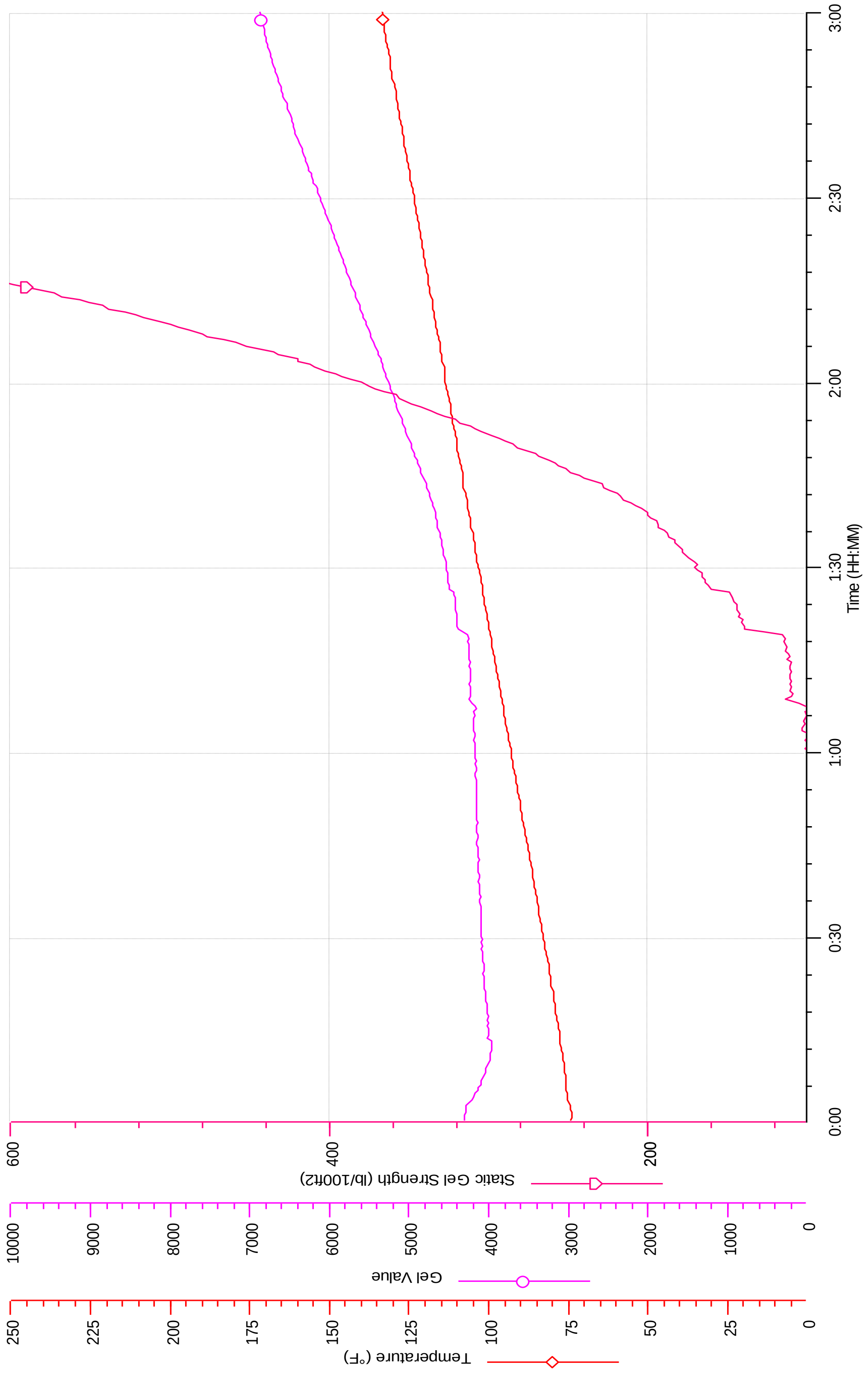

Figure 5-2.T: SGS test for Mix G + %44 tap water

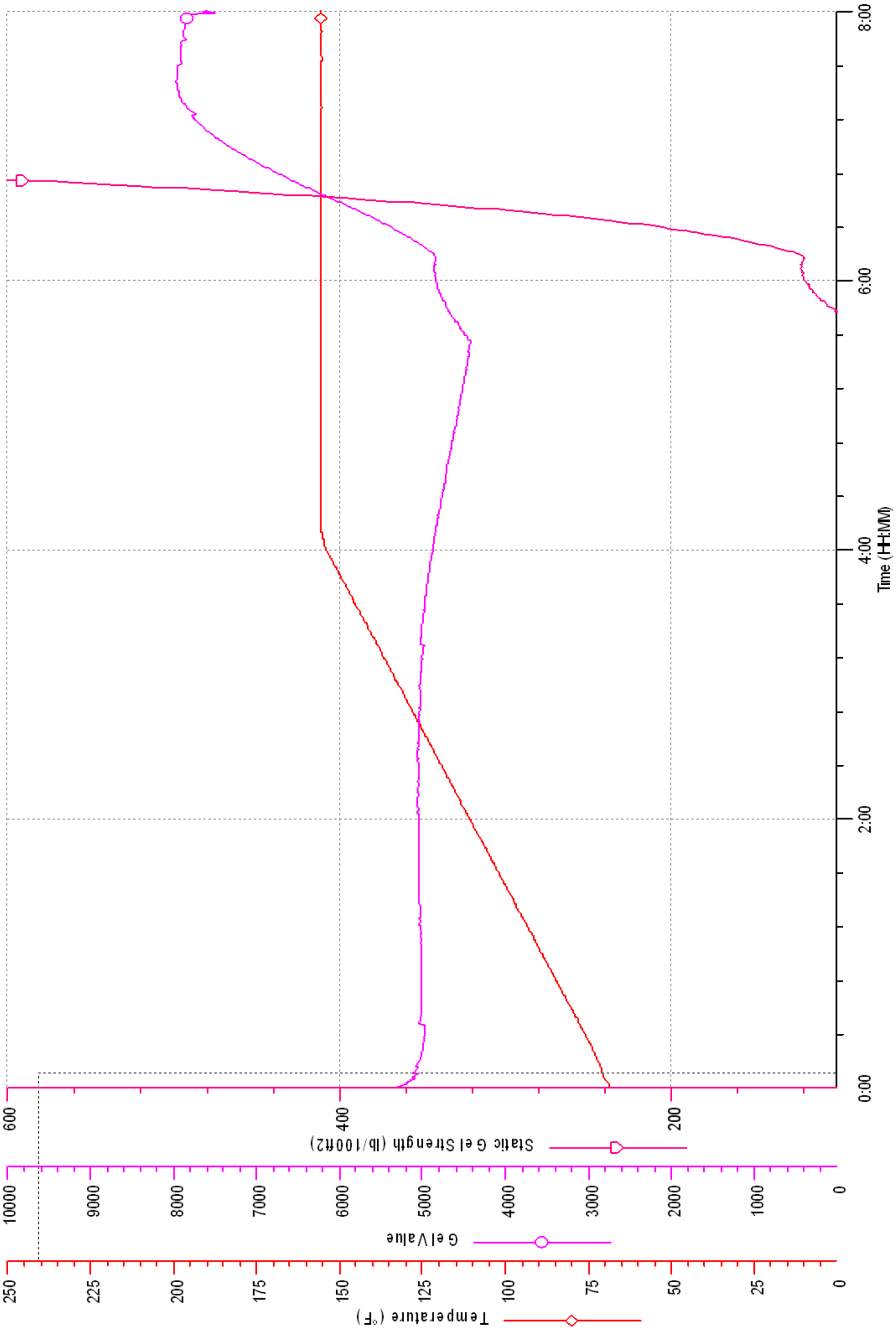

Figure 5-2.U: SGS test for Bolu + %44 tap water + %0.7 D-65 + %0.6 D-59 + 0.05 D-28 + % 3 KCl

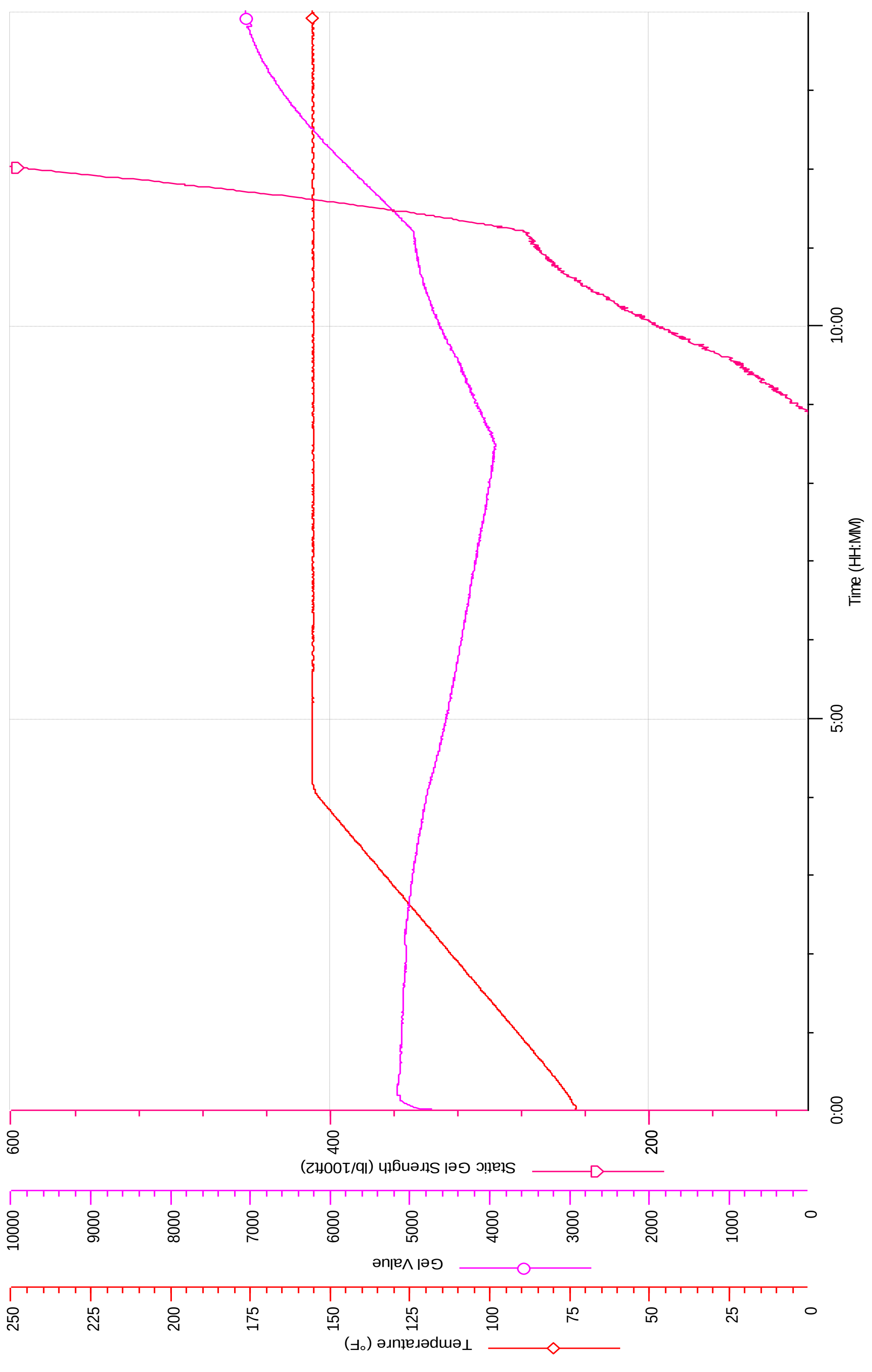

Figure 5-2.V: SGS test for Nuh + %44 tap water + %0.7 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCL

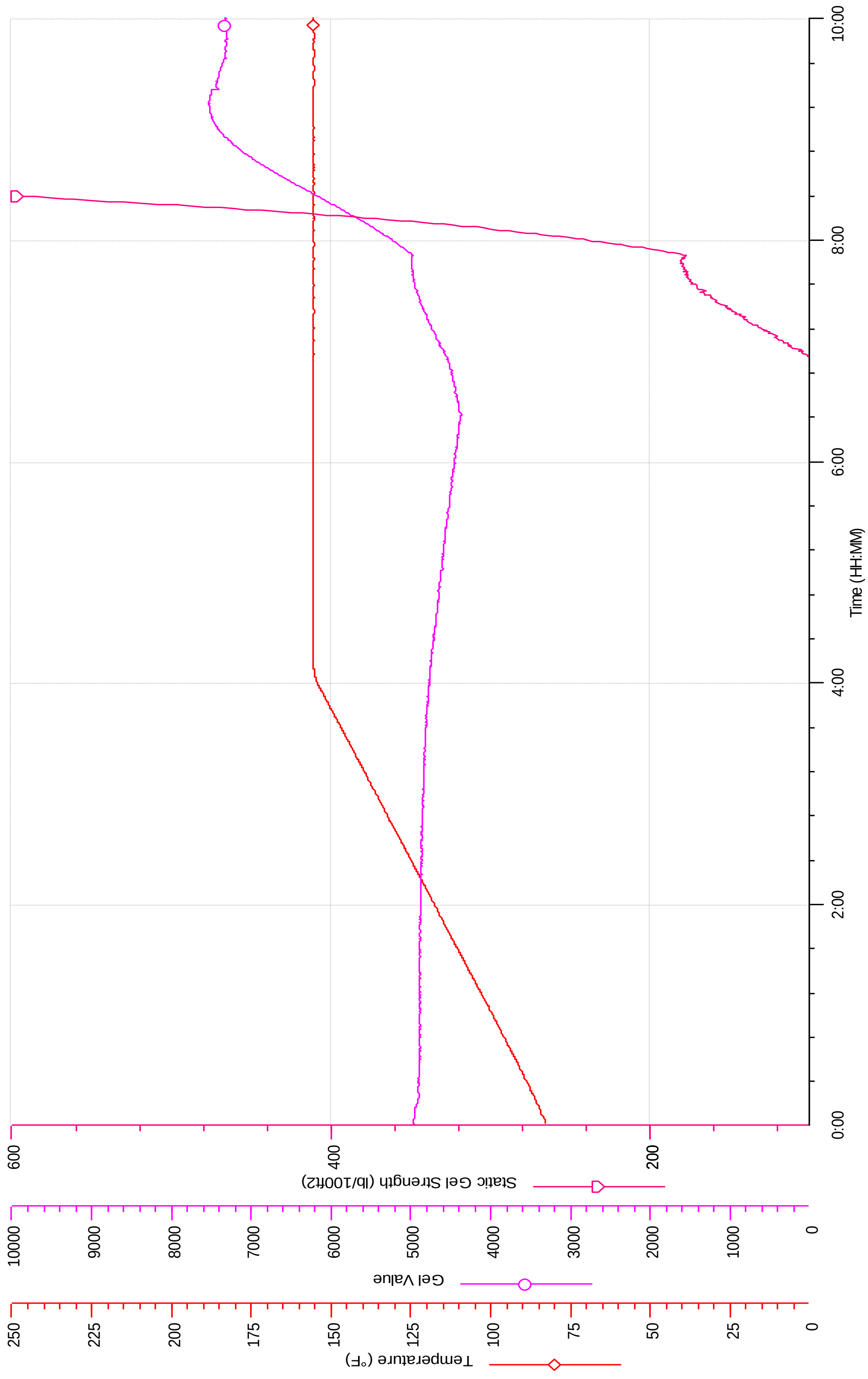

Figure 5-2.Y: SGS test for Mix G + %44 tap water + %0.7 D-65 + %0.6 D-59 + %0.05 D-28 + % 3 KCl