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# Master Thesis
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# Effects of Lost Circulation Material on Cement Properties

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# Abstract

This study aims to find the effect lost circulation materials has on cement slurries through testing in viscometer, pressurised consistometer and static gel strength analyser at selected concentrations. A worksheet containing a basic cement slurry recipe was used as basis for comparison against the slurries containing lost circulation material. SafeCarb 250, OptiSeal II, OptiSeal IV and G-Seal were the materials used to obtain the results for this thesis.

The chosen concentrations for lost circulation material were, by recommendation, 100 kg/m$^3$, 150 kg/m$^3$ and 200 kg/m$^3$. For the thickening time and compressive strength tests, only the highest and lowest concentrations were chosen. All three concentrations were used when conducting tests on the viscometer.

The rheology of the slurries were tested in the viscometers subjected to surface and downhole conditions. It was found that OptiSeal II, containing both graphite and calcium carbonate, had 73,5% higher viscosity and 15,24% higher yield stress at surface conditions compared to the base slurry. G-Seal, containing coarse-sized graphite, had 100% higher viscosity and 56,4% higher yield stress at downhole conditions. OptiSeal IV, a calcium carbonate, showed the overall lowest values compared to the base slurry with 28,1% lower yield stress at surface conditions, 32% lower yield stress and 8,36% lower viscosity at downhole conditions.

Thickening times were tested in a pressurised consistometer. Tests showed that the addition of SafeCarb 250, a calcium carbonate, decreased thickening time with 18% as concentrations of SafeCarb increased.

Compressive strengths were tested in a static gel strength analyser. All materials containing calcium carbonate achieved a higher compressive strength than the base slurry. OptiSeal II was 47,5% higher than base slurry on the highest concentration. G-Seal had a 34% lower compressive strength on the lowest concentration but exceeded the base slurry again at the highest concentration.

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# Nomenclature

## Abbreviations

| | |
|---|---|
| BET | Brunauer-Emmett-Teller |
| LCM | Lost Circulation Material |
| OPC | Ordinary Portland Cement |
| PSD | Particle Size Distribution |
| RAM | Right after mixing |
| SGSA | Static Gel Strength Analyser |
| TT | Thickening time |
| UCA | Ultrasonic Cement Analyser |

# 1 Introduction

Cement is one of the most important materials used in the oil industry today. Its widespread use in well cementing operations possibly also makes it the most abundant manufactured material. The cement type most used for such purposes is the Ordinary Portland Cement (OPC).

The type of cement used in well cementing operations differ much from those used under normal construction operations. Portland cements are specifically manufactured to tackle the encountered downhole conditions of a well. Other special cements are also manufactured, these are meant to solve specific problems related to individual situations of a well.

Portland cement is what we call a hydraulic cement. Characteristics of such a cement include development of compressive strength as a result of hydration. This means that a chemical reaction occurs between the compounds in the cement and the water added to it. For hydraulic cements, setting and hardening is possible both in air, and underwater. This is essential for well cementing operations as it allows for the cement to be pumped downhole as a liquid and left to develop its properties into a solid. Another characteristic with the Portland cement is that its strength development is uniform, predictable and very rapid. Once the cement is set, its got a low permeability and is nearly insoluble in water. This prevents the cement from deterioration at exposure to water. To achieve and maintain zonal isolation, this is an essential property of the cement [1].

## 1.1 Thesis Objectives

The main objective of this thesis is to investigate whether the addition of lost circulation material to cement slurries has any effect on its properties. Specifically, laboratory tests on viscometers, pressurised consistometers and static gel strength analyser will be done to evaluate the effect materials have on the rheology, thickening time and compressive strength development of a cement slurry.

# 2 Portland Cement

In this chapter we will be discussing the raw materials needed to make the Portland cement, the processes which makes the finished cement product, phenomenons which might occur during hydration as well as parameters affecting the end result. The chapter is based on the well cementing book by Nelson and Guillot [1].

## 2.1 Raw materials of Portland cement

Portland cement is the result we get from pulverising clinker with the right consistency of calcium, silica, alumina and iron compounds. The calcined (burned) material that exits a rotary kiln in a cement plant is what we call clinker. The finished product of Portland cement usually has one or more forms of calcium sulfate inter ground with the clinker. To ensure that the finished product meets the quality requirements, frequent chemical analyses of all materials are made.

The clinker used to produce Portland cement contains two raw materials. These two are argillaceous materials, which contains silica, alumina, and iron oxide, and calcareous materials, which contain lime. A great variety of raw materials, both artificial and natural, is used in the production of the cement depending on the location of the cement plant.

As it is beneficial that the chosen material has compositions similar to that of Portland cement, some materials are more important for production than others. For calcareous materials, sedimentary and metamorphic limestones, shell deposits, coral and "cement rock" are most similar to Portland cement and is therefore the most important materials. When looking at artificial calcareous materials, wastes from some industrial processes like precipitated calcium carbonate is most common.

Important natural argillaceous raw materials can be found in clays, marls, shales, slate, mudstones volcanic ashes, schist and alluvial silt. The most important artificial sources include fly ash from coal-fired power plants and blast-furnace slag from steelworks.

As Portland cement is produced from clinker, its properties are decided by the clinkers mineralogical composition. A conventional mineralogical composition of Portland cement may be seen in Table 1.

**Table 1:** A conventional mineralogical composition of Portland cement[1]

| Oxide composition | Cement Notation | Common Name | Concentration (wt%) |
|---|---|---|---|
| $3CaO \cdot SiO_2$ | $C_3S$ | Alite | 55-65 |
| $2CaO \cdot SiO_2$ | $C_2S$ | Belite | 15-25 |
| $3CaO \cdot Al_2O_3$ | $C_3A$ | Aluminate | 8-14 |
| $4CaO \cdot Al_2O_3Fe_2O_3$ | $C_4AF$ | Ferrite phase | 8-12 |

The finished cement may be sensitive to impurities in the raw material and kiln fuels. These impurities may significantly affect the properties of the product and special consideration to the amount needs to be addressed. As an example, in quantities from 0,5wt% of manganese, large alite crystals may develop which has an impact on the strength development of the cement. In different ways, magnesia, chlorides, fluorine, phosphates, lead, zinc and alkali content can, in excessive amounts, disrupt the normal development of cement properties.

An important aspect with Portland cement is its sensitivity towards moisture. There is no expiry date for cement, meaning that it will retain its quality as long as it's kept dry. However, if the cement ever comes in contact with moisture or damp air, the cement will set more slowly and have less strength development. To keep the cement in as good quality as possible, storing cement in low relative humidity is therefore important.

## 2.2 Hydration

As mentioned in Section 1, a typical characteristic of Portland cement is that strength development happens through hydration. In this process, hydrated compounds of the cement are formed when the cement is brought into contact with water, unstable and supersaturated solutions form, gradually depositing excess solids. The hydration of the anhydrous cement compounds will continue until the complete hydration has occurred.

Portland cement is a multicomponent system, meaning that the principal components ($C_3S$, $C_2S$, $C_3A$, and $C_4AF$) each have different hydration kinetics and form different hydration products, with their combined performance being the Portland cement. This thesis will not go in depth on the performance of individual components but rather discuss the combined results.

As hardened Portland cement comprises mostly of silicate phases with concentrations as high as 80% of the totalt material, hydration of $C_3S$ (as much as 68% concentration) is often used as a model for the hydration of Portland cement. Hydration of $C_2S$ does normally not exceed 30% of the totalt quantity. The hydration product of both phases is what we call Portlandite and calcium hydroxide, Portlandite is calcium silicate hydrate or $C_3S_2H_3$. Fully hydrated Portland cement contains usually about 15% to 20% calcium hydroxide and roughly 65% Portlandite. Portlandite is therefore considered to be the main binder in the finished product.

The hydration of Portland cement is a complex dissolution and precipitation process from a chemical point of view. The hydration reactions of the various components of the cement proceeds simultaneously at different rates and even influence each other. Using only the hydration of $C_3S$ as a model is therefore a bit idealised and remembering that many additional parameters are involved in the process is therefore important.

### 2.2.1 Volume changes during setting

Net volume diminution or absolute volume decrease is a term used for Portland cement as it reacts with water. This happens because the hydrated material has a higher absolute density than that of the initial reactants. However, the bulk volume remains the same or is slightly increased due to the increase of the internal porosity of the system. The fact that the absolute volume decreases can affect the transmission of hydrostatic pressure to the formation. This may in turn lead to annular fluid migration.

### 2.2.2 Effect of temperature

One major factor affecting the hydration of Portland cement is temperature. This parameter affects the cements hydration rate and nature, stability and morphology of the hydration products.

At elevated temperatures, like downhole temperatures, the hydration of cement is also elevated. Fig. 1 shows how increased temperature can affect Class G Portland cement.

Nelson and Michaux [1] explains that changes in the microstructure and morphology of the Portlandite phase takes place at temperatures above 40°C. These changes include a more fibrous material and the degree of silicate polymerization is higher.

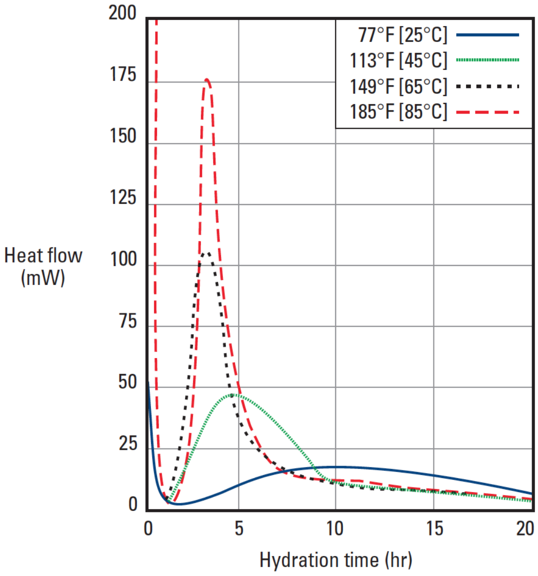


**Figure 1:** Effect of temperature on hydration kinetics of Portland cement Class G.[1]

### 2.2.3 Flash set and false set

"Flash set" or "quick set" is a phenomenon which occurs when Portland cement clinker is ground alone and mixed with water. As components like calcium sulfates, in the form of gypsum, are not present, $C_3A$ and $C_4AF$ rapidly reacts. A marked increase in slurry temperature can be noted, and an irreversible stiffening occurs, followed quickly by a pseudoset. This phenomenon can be a problem in well cementing operations as a flash set could prevent proper placement of the cement slurry in the annulus.

To counter the effects of the rapid reaction of $C_3A$ and $C_4AF$, calcium sulfates are added. The quantity of added calcium sulfates are balanced with the reactivity of the clinker, this is done in order to achieve optimal cement performance. This is important as a flash set can still occur due to insufficient calcium sulfates present in the blend. No rule of thumb exists for this balance as this depends on a variety of parameters, including calcium sulfates reactivity, alkali content, cement particle size distribution and aluminate phase content [2].

Another important phenomenon which can occur in a cement slurry is "false set". This is a stiffening or gelation of the cement slurry which can be reversed by vigorously agitating the slurry. False set happens as the aqueous phase of the cement slurry becomes supersaturated with respect to gypsum and "secondary gypsum" is precipitated. This supersaturation of gypsum is a result of the gypsum being dehydrated due to heat generated during the grinding process at the cement mill. Other sources like calcium sulfate hemihydrate, which is another form of calcium sulfate, might also be present in the blend. Compared to gypsum, it is around three times as soluble as gypsum at ambient temperatures, which in turn causes the supersaturation of gypsum. During a well cementing operation, agitation to reverse false set is not possible, and dispersants are therefore added to reduce the rheological impact of false sets.

### 2.2.4 Effects of aging

Silk [3] explains if Portland is exposed to the atmosphere and/or high temperatures during storage, cement properties may be significantly degraded. Possible effects include:

- Increased slurry viscosity
- Increased thickening time
- Decreased heat of hydration
- Decreased compressive strength

The relative humidity of the storage environment is the key factor that decides the rate of degradation of the cements properties. In this process, Portlandite is carbonated and free $CaO$ and $C_3A/C_4AF$ is partially hydrated. During this reaction, water is liberated, causing the aluminate, silicate and alkali phases to prehydrate. This imbalance between the aluminates and the sulfates can cause a false set when the cement eventually is hydrated in water. Furthermore, transport between containers might be difficult as the cement tends to form lumps as a result of this reaction.

### 2.2.5 Influence of alkalis

Alkaline elements in Portland cement affects strength development and the principal elements are sodium and potassium. Because of how alkaline elements affects Portland cement, they are usually kept below 1%, 0,75% for well cement.

### 2.2.6 Influence of surface area

Cement reactivity and slurry rheology is mostly affected by surface area. To measure surface area, or cement fineness, we can use Blaine method. This is done by measuring the air permeability of a small layer of lightly compacted cement. This information is used to calculate the a theoretical surface area for the cement, under the assumption that the cement particles are spherical. It is however important to note that Blaine method underestimates the true surface area, which can be measured using the Brunauer-Emmett-Teller (BET) gas-adsorption method[4].

To maintain consistency and performance, surface area is controlled by the cement manufacturer. This is done because fineness is directly related to the water-to-cement ratio required to prepare a pumpable slurry and cement particle wetting[5].

Cement surface area can also be correlated with the development of compressive strength. The greater the compressive strength of the cement, the higher surface area of the cement.

### 2.2.7 Sulfate resistance

Certain cement hydration products like precipitated calcium hydroxide has a tendency to react with downhole brines. This reaction with magnesium sulfate and sodium sulfate causes the formation of magnesium hydroxide, sodium hydroxide and calcium sulfate. These products lead to swelling of the cement as well as an increase in cement porosity. This can be bad as the reaction happens after the cement has developed strength, making an uncontrolled expansion of cement to loose compressive strength, crack, and damage tubulars. By adding pozzolanic materials such as fly ash to the cement blend it is possible to significantly reduce the sulfate attacks.

## 2.3 Summary

The widespread use of Portland cement in well cementing operations makes it an abundantly manufactured material. It is also one of the most important materials used in the oil industry today. Depending on the blend and additives used, cement can tackle most downhole conditions including special conditions a well might have. Portland cement hardens through a process called hydration which allows it to be pumped down a well as a liquid and left to develop into a solid. Portland cement is made from clinker which contains the two raw materials, argillaceous and calcareous materials.

# 3 Lost Circulation

Lost circulation (or lost returns) is a term encountered during drilling or cementing operations where drilling fluids or cement slurries are partially or completely lost to the surrounding formation. This can be due to the formation being fractured during these operations, the surrounding formation zone is highly permeable or due to cavernous formations and fractures [6]. In this chapter, consequences of lost circulation and remedial actions will be discussed.

## 3.1 Consequences of lost circulation

To be able to solve the problems of lost circulation with the correct measures, it is important that we acquire knowledge about the type of circulation zone, the severity of the losses and the drilling history of the well before the losses occurred [7]. Daccord et al.[7] lists the major consequences related to lost circulation in drilling, cementing and completion and workover operations found in Table 2.

**Table 2:** Major consequences of lost circulation[7]

| Drilling | Cementing | Completion and Workover |
|---|---|---|
| Loss of mud | Reduced annular coverage | Loss of completion fluid |
| Lost time | Casing corrosion | Lost time |
| Poor cement job | Poor zonal isolation | Formation damage |
| Reduced safety | Reduced safety | Reduced safety |
| Stuck in hole | | Lost reserves |
| Wasted casing string | | Loss of well |
| Failure to reach target depth | | |
| Blowout and kill operations | | |
| Downhole blowouts | | |
| Environmental incidents | | |

To able to choose a fitting measure to deal with a lost circulation situation we classify the amount of fluid loss according to their severity. This categorisation helps to quickly make a decision to wether the losses are tolerated, and drilling is continued, or corrective measures will need to be implemented. Table 3 shows the relation between the severity of losses and the type of loss that equals to.

**Table 3:** Severity Classification for Lost Circulation[7]

| Type of Losses | Severity |
|---|---|
| Seepage (minor) | < 10 bbl [1.5 $m^3$]/hr |
| Partial (medium) | 10 to 100 bbl [1.5 $m^3$ to 15 $m^3$]/hr |
| Severe (massive) | 100 to 500 bbl [15 $m^3$ to 75 $m^3$]/hr |
| Total (complete) | Unable to keep the hole full |

For a situation where seepage is present, one might decide that the losses are tolerable and that drilling is continued. This is often due to the simple fact that the cost of drilling fluids and rig rates will accumulate too a far greater expense than the consequences of the seepage.

A partial loss is like the seepage but at a greater loss of fluids, and so the decision to either correct the problem or continue with operations proves a harder choice. Generally, drilling may continue if the pressures are within acceptable limits and the cost of drilling fluids are not expensive.

In a severe loss situation, only a little fluids returns are recorded and it is of outmost importance that full circulation is regained. To do this, a lower-density fluid may be pumped into the well to find the required volume to fill the well. If the well stabilises, the well is filled by calculating the hydrostatic pressures. In the case of continued loss of fluids, the use of lost circulation material (LCM) may be necessary to bridge the source of loss, or if that does not work, progress to plugs. Since drilling fluids balances out the pressures in a well, the loss of too much drilling fluids may cause an influx. It is therefore important that the well is monitored in an event of lost circulation. Continued drilling in some areas is possible if the pressures are manageable and the cost of drilling fluid is low.

Total losses are the worst case scenario in a lost circulation situation, and all the drilling fluid is lost to the formation. One begins treatment, like the severe loss situation, with LCM pills and progresses to plugs if normal measures fail to correct the problem.

One of the most serious lost circulation conditions occurs during an underground blowout. This happens when we have cross-flow of fluids from one formation zone to an upper weaker or hydraulically fractured zone. Other than losing fluid, we must also deal with a dangerous well-control situation. Such an event is usually indicated by unstable surface-pressure readings. To avoid total loss of fluid, correct casing placement is critical.

## 3.2 Summary

During drilling or cementing operations we sometimes encounter a problem where some or all of the drilling fluid or cement slurry is lost to the formation. This phenomenon is called lost circulation. The consequences of such problems can range from poor zonal isolation to reduced safety and environmental incidents. It is therefore important to classify the losses and choose a fitting remedial action of the problem accordingly.

# 4 Lost Circulation Material (LCM)

Lost Circulation Material (LCM) is a bridging agent used to create a barrier against a pore throat opening to prevent loss of fluids. This can either be done through mixing the LCM into the drilling fluid, either through a pill or in the fluid itself. Depending on their physical properties and in the manner of how they work, they can be classified into five groups[7]:

- Granular
- Lamellar (or flake-like)
- Fibrous
- Mixed
- Encapsulated fluid-absorbing particles

This chapter will elaborate on the different groups of particles, application areas and properties.

## 4.1 LCMs for drilling fluids

### 4.1.1 Granular LCM

Granular LCMs, with their spherical nature, can form two types of sealing. The first is formed at the formation face while the second bridges within the formation matrix. The latter is preferred as, even though they are rigid materials, the first is easily dislodged by movement of pipe in the wellbore. This is an unwanted situation as this weakens the sealing capabilities of the bridging agent. Gatlin and Nemir [8] found that proper filtration with bridging agent depended on the particle-size distribution across the pore throat. In general, larger sized particles should first form a bridge within the void followed by smaller sized particles to fill in the interstitial void. This process then continues until a filtration problem is achieved as the void space now is smaller than the drilling-mud solids.

Abrams showed in 1977[9] that the median particle size of the bridging additive should be slightly greater than or equal to one-third the median pore size of the formation.

Dick et al. [10] wrote a software application based on the ideal packing theory [11] rather than Abrams' rule. The software determines the optimal blend of bridging agents according to the maximum pore size and the formation permeability.

### 4.1.2 Lamellar (or flake-like) LCM

When treating losses to permeable and porous formations, lamellar LCMs provide the best results. With their flat, layer-like properties with limited or no rigidity they are designed to form a mat on the formation face rather than within the matrix.

### 4.1.3 Fibrous LCM

Like the lamellar LCMs, the fibrous LCMs form a mat-like bridge over the formation face. Therefore it is best suited for controlling losses to porous and highly permeable formations. The reduction of pore openings causes the colloidal particles to more easily form a filtercake against the formation.

### 4.1.4 Mixed LCM

Mixed LCMs are, like the name suggests, a mix between granular, fibrous and lamellar LCMs. These LCMs also provides a gradation of particle sizes as well as a variation of material types for sealing different classes of lost circulation zones.

### 4.1.5 Encapsulated fluid-absorbing particles

The encapsulated particle technique consists of encapsulating bentonitic particles within a water-insoluble polymeric coating, through which a small hole is drilled. The idea is that when the encapsulated bentonite is pumped into the well, water from the mud seeps through the drilled hole. As the bentonite swells it ultimately ruptures the coating. The result is that it seals the void in the lost circulation zone.

## 4.2 LCMs for cement slurries

LCMs may also be used in a cement slurry, however, it must be ensured that the materials used does not directly affect the cement composition. To avoid plugging and bridging of downhole equipment, it is also necessary to select correct size and concentration range of the LCMs. LCMs effectiveness in cement slurries are more limited than in a regular drilling operation. Nayberg and Petty [12] and Turki and Mackay [13] agreed that their effectiveness is limited to minor or partial losses in highly permeable formations. They are not suitable for correcting problems where total lost circulation occurs in naturally fractured or cavernous formations. An overview of the most common LCMs used in cement slurries are showed in Table 4.

**Table 4:** Common LCM in Cement Slurry[7]

| Type | Material | Nature of particles | Amount Used | Water Required |
|---|---|---|---|---|
| Granular | Gilsonite | Graded | 5 to 50 lbm/sk | 2 gal/50 lbm |
| | Perlite | Expanded | 1/2 to 1 $ft^3$/sk | 4 gal/$ft^3$ |
| | Walnut shells | Graded | 1 to 5 lbm/sk | 0.85 gal/50 lbm |
| | Coal | Graded | 1 to 10 lbm/sk | 2 gal/50 lbm |
| Lamellar | Cellophane flake | Flaked | 1/8 to 2 lbm/sk | none |
| Fibrous | Nylon | Short fibers | 1 1/8 to 1/4 lbm/sk | none |
| | Glass | Long fibers | 2 to 3 lbm/sk | none |

Of the LCMs types available, the granular type is the most common in use. It stops lost circulation by bridging at the formation face or within the formation matrix. Gilsonite is an example of such a LCM. It is a naturally occurring asphaltine hydrocarbon which usually range between 2,38mm - 0,25mm (8-60 mesh) in particle size. The use of Gilsonite has however some limitations as it has a fairly low melting point of 104°C and is therefore not suitable for use in high temperature environments. In such environments, coal, with a melting point of 538°C, is much better suited and may be used in the same manner as gilsonite. The use of shells from pecans, walnuts, and other nuts is also a commonly used granular LCM. They are available in fine, medium, and coarse grades. It is, however, important that particular care is taken with concentration levels above 4 lbm/sk as downhole equipment is prone to plugging in such concentrations.

The most commonly used LCM among the flake materials is the Cellophane flakes. With diameters of 9,5 to 19 mm in concentrations below 2 lbm/sk the bulk loading and mixing of cement slurry is kept within manageable conditions. On concentrations above 2 lbm/sk this becomes very difficult, and should therefore be avoided to ensure proper mixing of the cement slurry.

For fiber LCMs, a common choice would fall upon nylon and polypropylene. These LCMs form a mat-like structure on the formation face which allows the cement to quickly develop a filtercake. A disadvantage of using such fibrous LCMs is their tendency for improper mixing with the cement slurry. Fibers are often seen nested up in a "fur ball" floating on the cement slurry surface. This problem often leads to plugging of cement equipment like pump plungers and float equipment. An additional problem with the fibrous LCMs is the cost of most fibers available on the market is very high. In many cases, this does not make them economically viable for use as a lost circulation material. On top of this, the correct blend of fiber sizes required to form an effective mat on the formation surface to stop circulation losses is difficult to achieve. This motivates for the development of a fibrous LCM with more desirable properties with regards to dispersing of fibers in cement, more specific size of fibers to control losses and cost [14].

The resulting development has lead to silica-based fibers. These fibers can vary in length of up to a maximum of 16 mm and are chemically inert to the hydration of Portland cement at temperatures below 110°C. They are also widely compatible with all cement additives and systems. At temperatures above 110°C, the fibers would contribute with a small amount of silica to the cement composition. This small incremental amount would only add to the silica cement or flour already present to combat the strength retrogression under those temperature conditions. The fibers are easy to handle due to the bundles or "tows" they are supplied in. In Fig. 2 we can see an example of these "tows" and how they are dispersed in liquid.

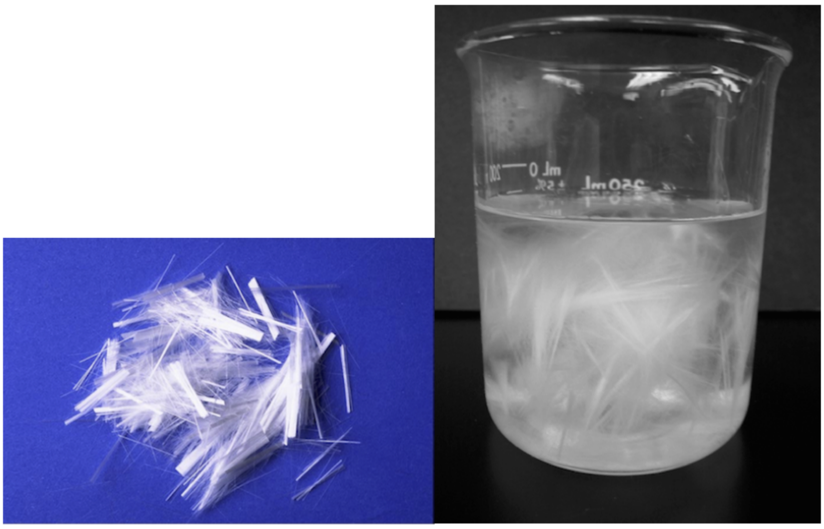


**Figure 2:** Silica fiber "tows" (left) and the fibers dispersed in water (right) [14]

## 4.3 Thixotropic cement systems

Another way of solving lost circulation problems is to use a thixotropic cement system. If an ordinary cement slurry should fracture the formation and loose some or all of its volume to the formation, there is not enough cement to fill up the intended space. With a thixotropic cement system, the slurries are still able to fill the desired fill-up as they quickly gel, leading to some of the hydrostatic pressure to be transmitted to the formation face and casing walls, acting as a self-support system.

## 4.4 Foamed cement systems

As an alternative to conventional cement systems when solving lost circulation problems, foamed cement systems are used due to their low density [12]. They have excellent fluid-loss control and are lightweight and compressible, have good thermal insulation properties and short transition times. Foamed cement systems are considered a special cement system.

## 4.5 Summary

LCMs are bridging agents commonly used to combat the problem of lost circulation. With its wide variety of types and materials, there are always an alternative to the severity of the problem. We have five classes of LCMs: granular, lamellar, fibrous, mixed and encapsulated fluid-absorbing particles. Of these, granular is the most commonly used LCM for cement systems. Alternatives to LCM may be thixotropic cement systems, used as they gel quickly which makes them able to fill the intended volume, or foamed cement systems, which are very lightweight and compressible.

# 5 Rheology

Rheology is important to understand the properties of a cement slurry. Viscosity is perhaps the most important in this regard and the aim is to determine the relationship between the shear stress and shear rate; in other words the pressure gradient and the flow rate element that causes fluid movement. Guillot [15] defines rheology as:

> "Rheology is the study of the deformation and flow of materials"

This chapter will describe some basic rheological principles, flow models and calculations needed to understand the tests conducted in the lab.

## 5.1 Types of flow

Under the flow conditions of isothermal and steady-state, fluids will have a distinct flow pattern. These can be either laminar, transitional or turbulent flows. The result of displacing a cement slurry in these flow patterns give widely different end results and it is therefore important to understand the difference between them. For the purpose of simplicity, flow in a pipe will be used as an example to describe these flow patterns.

### 5.1.1 Laminar flow

Laminar flow is when the particles in the fluid flows parallel to the pipe. The difference in particle velocity vary with the distance to the pipe walls. Particles close to walls tend to be stationary while the particles in the center of the pipe usually travels with the highest velocity. Fig. 3 is a normal representation of what a velocity profile for laminar flow will look like.

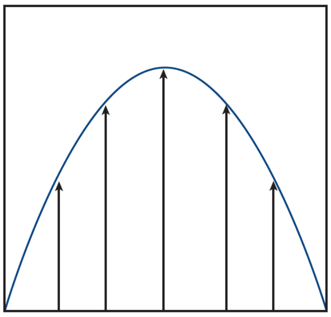


**Figure 3:** Laminar flow velocity profile for a Newtonian fluid[16]

All fluids will have their own specific velocity profile as it depends on their respective rheological behaviour. Fig. 3 shows a typical velocity profile of water with its parabolic shape.

### 5.1.2 Transitional Flow

Transitional flow is the pattern a fluid takes in a small window when transitioning from laminar to turbulent flow. The resulting velocity profile is alternating between that of the laminar and turbulent shapes.

### 5.1.3 Turbulent flow

Turbulent flow is the flow pattern achieved after transitional flow and it is associated with high flow rates. Particles are now moving in a more chaotic way where they swirl in a rolling motion along the pipe, the movement is no longer parallel to the pipe wall. A turbulent flow velocity profile is illustrated in Fig. 4.

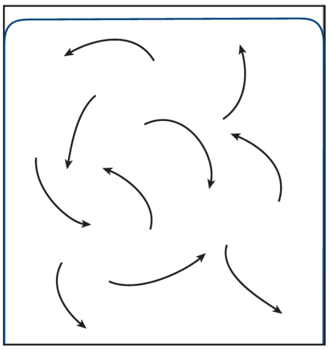


**Figure 4:** Turbulent flow velocity profile[15]

## 5.2 Viscosity

Viscosity is an important fluid property to explain the relationship between the shear stress and shear rate which causes fluid movement. This relationship, among other rheological properties of a cement slurry, must be understood in order to properly design, execute and evaluate a cement job. According to Guillot [15], proper rheological characterisation is important to:

- evaluate the slurry's mixability and pumpability (thickening time)
- optimise placement of slurry and mud removal

- friction pressure determination for when slurry flows in annuli and pipes
- evaluate a slurry's ability to transport larger particles like lost circulation materials
- predict how a wellbore-temperature profile affects placement of cement slurry
- predict the annular pressure after placement of cement slurry

If we imagine a laminar flow regime, the fluid movement may be represented by a large number of plates that move at different velocities and parallel to each other. This representation is illustrated in Fig. 5.

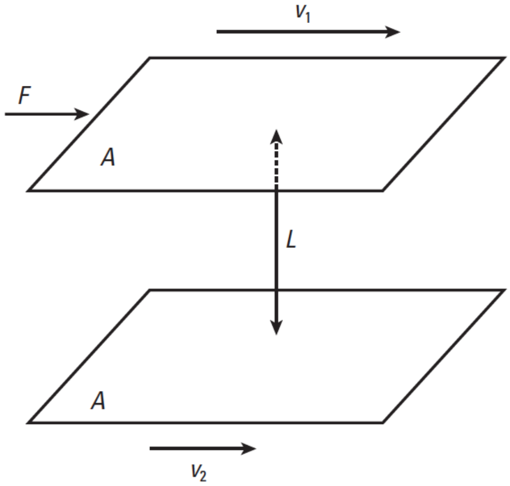


**Figure 5:** Flow between parallel plates[15]

Guillot [15] explains that in this laminar flow regime, the fluid particle velocity varies linearly from one plate to the other. The shear rate (or velocity gradient) is constant and expressed in Eq. 1 and mathematically in Eq. 2 using Fig. 5 as reference.

$$\text{Shear rate} = \frac{\text{the velocity difference between 2 plates}}{\text{the distance between 2 plates}}, \tag{1}$$

or

$$\frac{dv}{dx} = \frac{v_1 - v_2}{L}, \tag{2}$$

where $x$ is an axis perpendicular to the plates. The dimensions of Eq. 2 are

$$\frac{\text{length} \times \text{time}^{-1}}{\text{length}} = \text{time}^{-1}. \tag{3}$$

From Eq. 3 we can see that the unit for shear rate is $\text{s}^{-1}$. Further, the symbol used for shear rate is $\dot{\gamma}$.

Shear stress is the force per unit surface area that causes the shearing of the fluid, denoted by $\tau$ in Eq. 4. From Fig. 5:

$$\tau = \frac{F}{A}. \tag{4}$$

The dimensions of Eq. 4 are

$$\frac{\text{force}}{\text{length}^2} = \text{force} \times \text{length}^2. \tag{5}$$

The unit of shear stress is often expressed in common oilfield units and is lbf/100 $\text{ft}^2$. Pascal (Pa) is the units in the SI system.

From the shear stress, $\tau$, and shear rate, $\dot{\gamma}$, the viscosity, $\mu$, can now be found as a ratio between the two.

$$\mu = \frac{\tau}{\dot{\gamma}} \tag{6}$$

The dimensions of Eq. 6 are

$$\frac{\text{force}}{\text{length}^2} = \text{force} \times \text{length}^2 \times \text{time}. \tag{7}$$

The unit of viscosity is expressed as centipoise (cp) in common oilfield units. In the SI system, the unit is pascalsecond (Pa·s).

Guillot [15] further explains that in the simple case of a laminar flow regime in a pipe, the friction pressure gradient (or friction losses) can be considered to be proportional to the shear stress. The flow rate can considered as being proportional to the shear rate; however, this is not entirely correct. The relationship between the friction pressure gradient and flow rate relationship is controlled by viscosity. Viscosity is dependent on the temperature and pressure in the given situation. Also, for most fluids used in well construction, viscosity is dependent on shear rate. This opens for more complex situations than those encountered in the laminar flow regime in a pipe example.

In common well cementing operations, the situation described in Fig. 5 is not likely the normal case. Most likely, individual particles in the fluid would move at different velocities, in more than one direction and possibly with more than one component. In such a situation, the shear-rate and shear-stress fields would be described by a tensor, with the viscosity remaining a scalar dependent on some invariants of these tensors. To understand this would require knowledge of the relationship between the shear-rate and shear-stress tensors. This involves understanding multiple fluid properties that affect the fluid; however, this is beyond the scope of this thesis and we will limit the discussion to fluid viscosity.

### 5.2.1 Rheological models

In laminar flow, Newtonian and non-Newtonian fluids are defined by the relationship between shear rate and shear stress. Guillot [15] describes, in the following sections, the differences between Newtonian and non-Newtonian fluids and the different rheological models used to describe viscosity.

**Newtonian fluids** Newtonian fluids can simply be explained as fluids that follows Newton's laws. Such fluids follows the following equation

$$\tau = \mu \dot{\gamma} \tag{8}$$

A visual representation of this can be seen in Fig. 6. In the figure we see the linear line which represents the viscosity, $\mu$, of the fluid. This property only depends on temperature and pressure and is constant. Examples of a Newtonian fluid may include gasoline or water.

Flow is initiated as soon as a pressure gradient is applied to the fluid. This behavior is depicted in Fig. 7. We can also note that the shear stress/shear rate relationship is linear as long as the fluid is in laminar flow. This is also valid for the friction-pressure gradient/flow rate relationship. As the flow starts the transition from laminar into turbulent flow, we can se that this relationship is no longer linear. We can note that the friction pressure increases faster than in laminar flow.

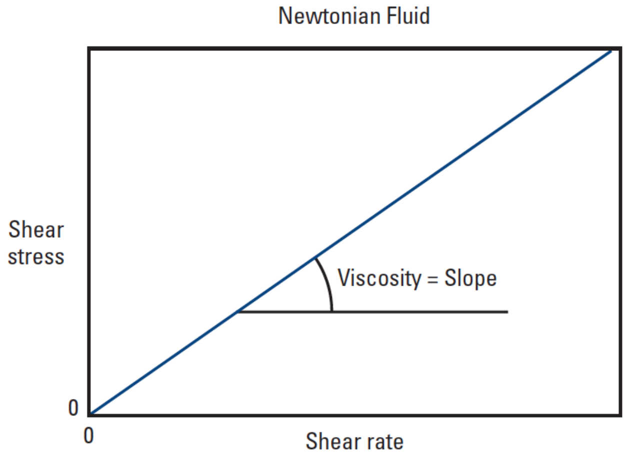


**Figure 6:** Shear-stress/shear-rate relationship for a Newtonian fluid[15].

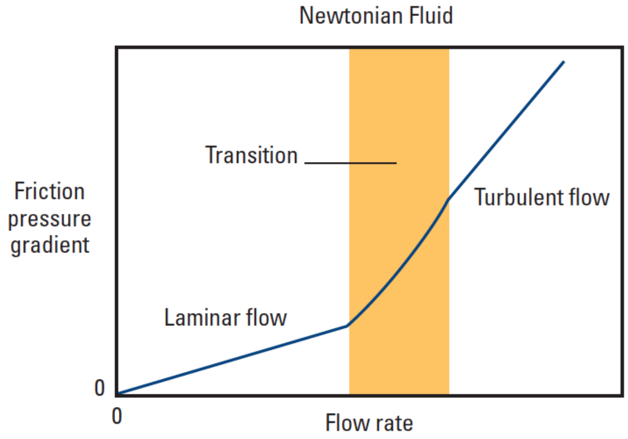


**Figure 7:** Friction pressure gradient/flow rate relationship for a Newtonian fluid flowing in a pipe[15].

**Non-Newtonian fluids** Unlike the Newtonian fluids, non-Newtonian fluids do not follow Newton's laws. These fluids also introduce the terms shear thinning and shear thickening. This means that viscosity is either decreased with shear rate, in which case the fluid is shear thinning, or increases with shear rate, shear thickening. Common examples of shear thinning fluids include most cement slurries, heavy oils and drilling muds. To describe the behaviour of these fluids, it's common in the oil industry to use these mathematical models:

- Power-law model
- Bingham model
- Herschel-Bulkley model

These will be described in the following sections.

**Power-law fluids** Fluids in the Power-law model are part of a class known as pseudo-plastic fluids. Power-law fluids have similarities with Newtonian fluids in that they start to flow as soon as a pressure gradient is applied. However, they differ in that the shear stress/shear rate relationship is not linear in Power-low fluids, even in laminar flow. This is illustrated in Fig. 8 below.

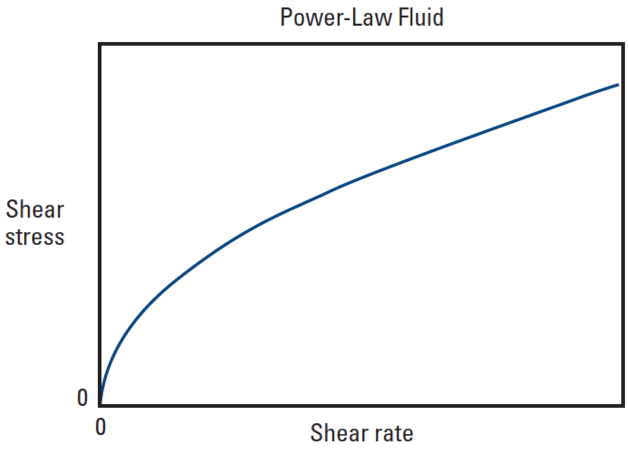


**Figure 8:** Shear-stress/shear-rate relationship for a power-law fluid with a power-law index, n, of < 1[15].

Fluids in the Power-law model is described by the following equations where $k$ stands for the consistency index.

$$\tau = k\dot{\gamma}^n \tag{9}$$

$$\mu = k\dot{\gamma}^{n-1}. \tag{10}$$

The power-law index, n, indicates wether or not a fluid is shear thinning (n < 1) or shear thickening (n > 1). If the index is equal to 1 then the power-law model is reduced to the Newtonian model. Particular caution needs to be exercised in situations where a shear thinning power-law fluid is characterised outside of its shear rate range. This is because the viscosity of these fluids varies from infinity at 0 shear rate to 0 at infinite shear rate, and the lower limit is therefore not physically sound.

From Fig. 9 we can note that as long as a power-law fluid is in laminar flow, the friction pressure gradient/flow rate relationship follows the power law. However, when transitioning to turbulent flow, the laminar model is no longer valid as friction pressure increases more quickly than suspected.

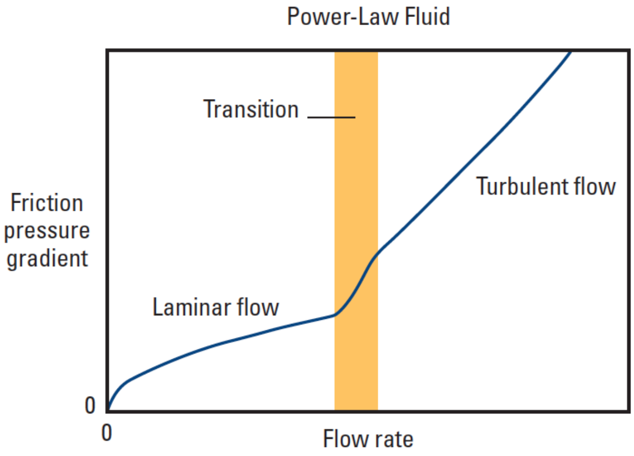


**Figure 9:** Friction pressure gradient/flow rate relationship for a power-law fluid with a power-law index, n, of < 1[15].

**Bingham plastic fluids** Bingham plastic fluids differ from the other models in that a minimum applied stress is required before the fluid is sheared. This is illustrated in Fig. 10.

The Bingham plastic model is defined by two parameters:

- the value of $\tau$ for $\dot{\gamma} = 0$, $\tau_y$
- the slope of the straight line, $\mu_p$.

Here, $\tau_y$ is notation for Bingham yield stress, while $\mu_p$ is a constant and is called the plastic viscosity.

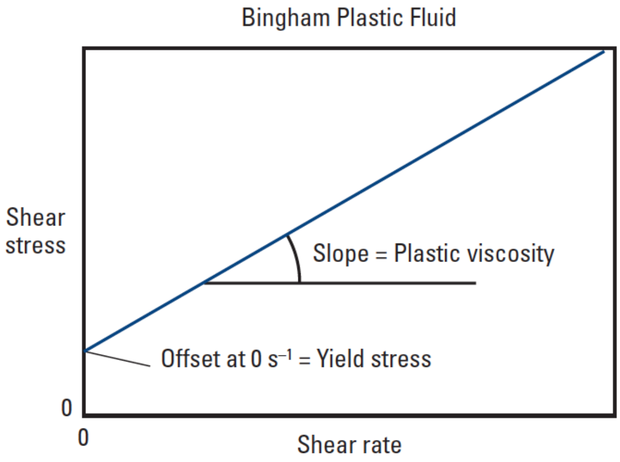


**Figure 10:** Shear-stress/shear-rate relationship for a Bingham plastic fluid[15].

We can express Bingham fluids by the following equations:

$$\tau = \tau_y + \mu_p \dot{\gamma} \quad \text{when} \quad \tau > \tau_y \tag{11}$$

$$\dot{\gamma} = 0 \quad \text{when} \quad \tau \leq \tau_y \tag{12}$$

or

$$\mu = \mu_p + \frac{\tau_y}{\dot{\gamma}}. \tag{13}$$

From this we can deduct that Bingham plastic fluids are shear thinning. The viscosity of such fluids varies from infinity at 0 shear rate to their plastic viscosities at infinite shear rates.

A minimum pressure gradient is required for a Bingham plastic fluid to initiate flow, as illustrated in Fig. 11. In laminar flow, the friction pressure gradient/flow rate relationship increases nonlinearly before it usually changes to a linear increase. As discussed in Section 5.2, flow rate cannot be considered to be considered to be proportional to the shear rate. As a result, the friction pressure gradient/flow rate relationship differs from the shear-stress/shear-rate behaviour. Further, as the flow rate increases towards transitional and turbulent flow, friction pressure increases at an unpredictable rate, as with the other fluid models.

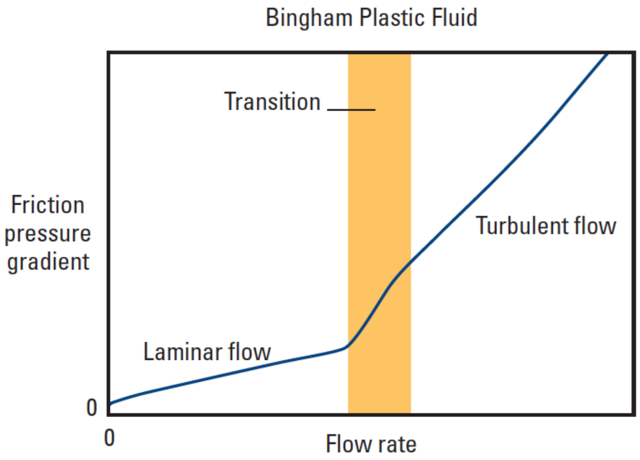


**Figure 11:** Friction pressure gradient/flow rate relationship for a Bingham plastic fluid[15].

**Herschel-Bulkley fluids** The behaviour of Herschel-Bulkley fluids is a combination of the behaviours from Bingham plastic fluids and power-law fluids. Like the Bingham plastic fluids, a minimum yield stress is necessary to initiate flow of the fluid. Above the initial yield stress, the shear-rate/shear-stress relationship follows the power-law model.

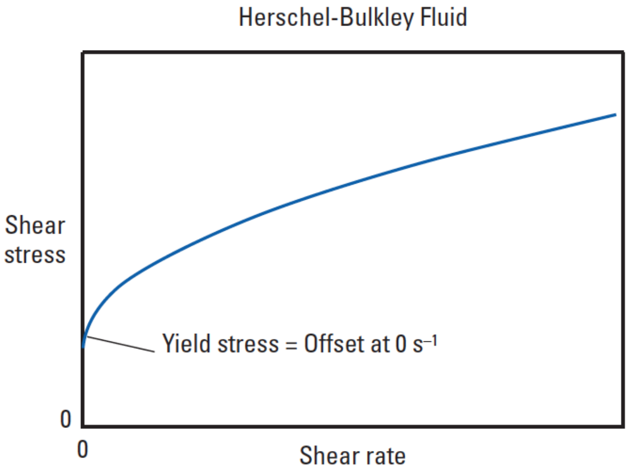


**Figure 12:** Shear-stress/shear-rate relationship for a Herschel-Bulkley fluid with n < 1[15].

To describe Herschel-Bulkley fluids we use the following equations:

$$\tau = \tau_y + k\dot{\gamma}^n \quad \text{when} \quad \tau > \tau_y \tag{14}$$

or

$$\mu = \frac{\tau_y + k\dot{\gamma}^n}{\dot{\gamma}} \tag{15}$$

Most Herschel-Bulkley fluid are shear thinning, in which case $n$ is smaller than 1. Viscosities for such fluids are ranging from infinity at 0 shear rate to 0 at infinite shear rate. We note that this is the same case as for the power-law fluids, and the lower limit is not physically sound. As with the power-law, caution should be exercised when using this model to characterise a fluid outside its shear-rate range.

The friction pressure gradient/flow rate relationship begins with a 0 flow rate offset before it increases nonlinearly, for as long as the fluid is in laminar flow. This is illustrated in Fig. 13. In the transition to turbulent flow, the relationship changes and friction pressure increases at an unpredictable rate, faster than predicted by the laminar relationship.

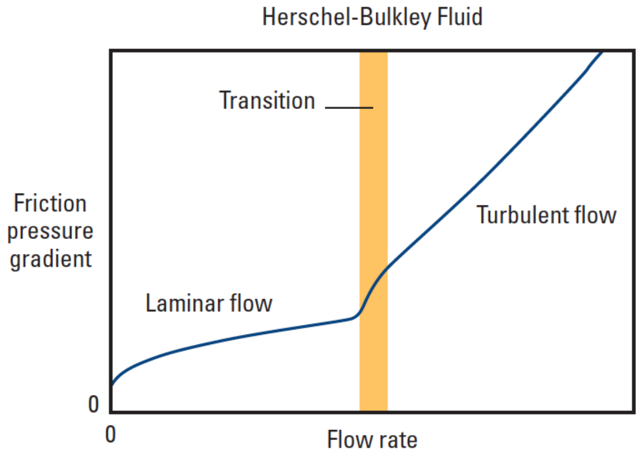


**Figure 13:** Friction pressure gradient/flow rate relationship for a Herschel-Bulkley fluid flowing in pipe with n < 1[15].

## 5.3 Summary

Rheology gives un an understanding of the flow properties of a fluid. Fluids have different flow patterns when under isothermal and steady-state conditions. These are: laminar, transitional and turbulent flow. Proper understand of these patterns is necessary for correct displacement of cement slurries to yield a wanted end result. Understanding viscosity, the relationship between shear stress and shear rate, encourages proper design, execution and evaluation of a cement job. Viscosity can be classified into rheological models depending on how the shear stress/shear rate relationship behaves. These are Power-Law, Bingham plastic and Herschel-Bulkley.

# 6 Laboratory testing

The practical part of the thesis, the laboratory testing, is covered in this section. Here, all the necessary tests needed for comparisons were performed. In addition to the operational procedures for how the tests were conducted, this section also includes descriptions what equipment was used and which chemical additives and LCM was added to the slurries.

## 6.1 Equipment

Testing equipment plays a big part in the success of this thesis. Without it, producing results for comparison simply would not be possible. Schlumberger was kind enough to allow access to their laboratory during for the purpose of this thesis. This enabled the use of mixing devices, viscometers, pressurised and atmospheric consistometers, SGSAs as well as all associated equipment needed to perform the tests. These will be described in the following sections.

### 6.1.1 Mixing device

Operational procedures and equipment specification for preparation of cement slurries in the laboratory is subjected to API RP 10B [17]. the mixer is a propeller-type mixer with two speed settings (4000 and 12000 rpm) shown in Fig. 14.

The normal amount of finished slurry prepared in the mixer is 600mL. Any liquid additives are added and thoroughly dispersed in the mix water before the cement is added at a mixing speed of 4000 rpm, preferably within 15 seconds. Immediately after cement is added, speed is increased to 12000 rpm and left to mix for 35 seconds. This is to ensure that the slurry is uniformly blended without lumps. If liquid additives are added to the mix water, the order of which they are added may be critical for the final properties of the cement and should be documented.

Not all variations of cement slurries are suitable for this mixing procedure. Cement systems containing microspheres or nitrogen as extenders, like ultralow-density systems, are examples of such systems. Special considerations needs to be shown as the microspheres may be easily broken in the mixer. Normally, the mixing procedure is changed for such systems as mixing speeds are reduced to 4000 rpm and duration of mixing is increased to 300 seconds.

Mixing procedure is also changed for foamed cement systems. The same mixing device may be used, but the blade is often changed to a special multiblade assembly. The foamed cement slurry is placed in the blending container, the contained is capped and sealed, adter which the mixing device is operated at high speed until the container is completely filled with foam. By adjusting the volume of base slurry added to the container, the density and quality of foamed cement may be varied.

Fig. 14 shows the actual two-speed mixing device used for mixing cement slurries.

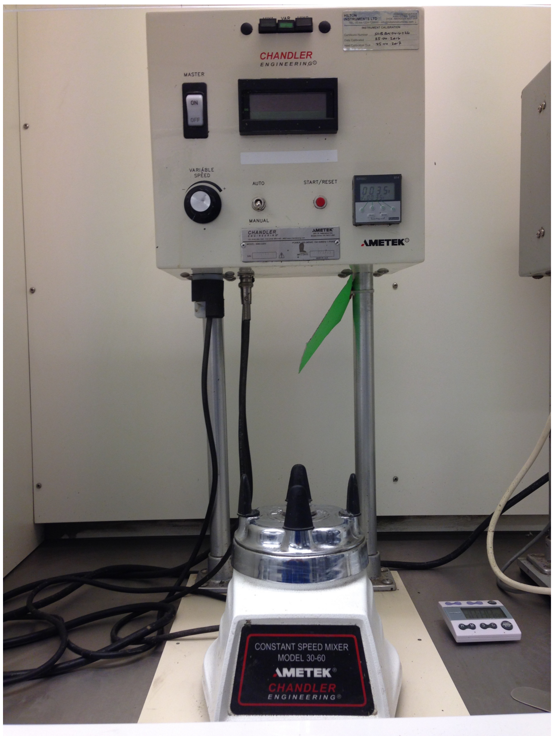


**Figure 14:** Cement slurry mixing device.

### 6.1.2 Slurry density

A commonly used equipment used to determine slurry density is the pressurised mud balance. The operational procedure for this equipment can be found in API RP 10B. It is operated by pouring slurry before a cap is screwed on. Any excess air in the cup is collapsed as a plunger filled with slurry is attached to the cap and used to pressurise the cup. Slurry density is then determined as the device is placed on a fulcrum, and balanced by adjusting a sliding weight [17].

### 6.1.3 Thickening time

Knowing the length of time a cement slurry remains in a pumpable is important information in a well cementing operation. Thickening-time tests are designed to to determine the duration the cement slurry is in a fluid state under simulated wellbore conditions of pressure and temperature. API RP 10B contains the operational procedures for the determination of thickening time.

Using a pressurised consistometer, as depicted in Fig. 15, we are able to evaluate the test slurry consistency under simulated wellbore temperature and pressure. The test slurry is contained in a cup which rotates in the consistometer capable of exposing the slurry to a maximum of 204°C and 175 MPA (400°F and 25000 psi). The slurry consistency is measured in Bearden units ($B_c$). Bearden units has no direct conversion factor to more common units of viscosity and is a dimensionless quantity. As the cement slurry begins to set, point of departure, 30 $B_c$, 70 $B_c$ and 100 $B_c$ is noted. Point of departure is the point in time at which the consistency of the slurry begins to increase. Although 100 $B_c$ is the consistency at which the test ends, 70 $B_c$ is considered to be the maximum consistency for the slurry to still be pumpable.

Thickening time for a slurry during laboratory testing may differ from the same slurry in the wellbore. This is because the consistometer cup has no design for slurry fluid loss provision.

Measured thickening time is dependent on temperature and pressure which may significantly alter the outcome. Additionally, thickening time is also dependent upon the rate at which the final pressure and temperature is reached. Continuously changing temperature and pressure in the wellbore makes it difficult to get an accurate measurement of the circulating-temperature and pressure during a cement job. The cement job design and wellbore conditions affects the rates at which the final temperature and pressure is reached. Before a cement job is performed, variables like geothermal gradient, well depth, the effects of well operations and anticipated job duration must be taken into account. Testing guidelines for various cement jobs, like primary cementing and squeeze cementing, is contained in API RP 10B.

**Figure 15:** Pressurised consistometer used to determine thickening time of a cement slurry.

### 6.1.4 Static Gel Strength Analyser (SGSA)

A static gel strength analyser (SGSA) is a new measurement technique for evaluation of static gel strength under downhole temperature and pressure. It relies on the analysis of the acoustic waveform transmitted through the cement. The acoustic waveform transmitted through the cement can be transformed into an evaluation of static gel strength using proprietary algorithms. It is also possible to determine the compressive strength of the cement using the SGSA. An SGSA is shown in Fig. 16.

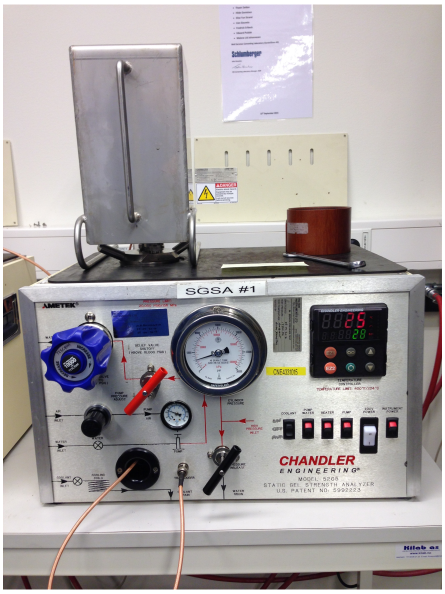


**Figure 16:** Static Gel Strength Analyser used to determine compressive strength development of cement slurry.

### 6.1.5 Atmospheric consistometer

An atmospheric consistometer, shown in Fig. 17, is a non-pressurised type of consistometer used for conditioning of cement slurries before fluid-loss, free-fluid or rheology tests.

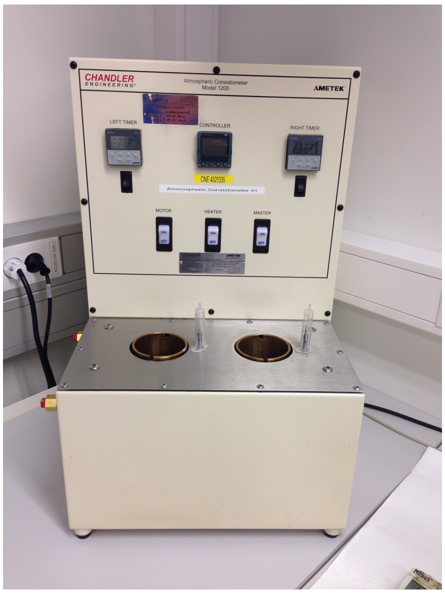


**Figure 17:** Atmospheric consistometer used to condition a cement slurry.

### 6.1.6 Free fluid

Free fluid tests are intended to measure any water which may have separated from the slurry during periods of standstill. Such separation may impair zonal isolation, especially for high deviation wellbores. Using a graduated cylinder, simulating a wellbore, this separation may be measured usually either in a vertical or 45°position. Measured from the moment the slurry is poured into the cylinder, the test duration is 2 hours. The operational procedure for this test is contained in API RP 10B [17].

After the initial 2 hours test is done, it is normal to check for settling of suspended solids which may have separated from the cement slurry. Such a separation is particularly evident for cement slurries containing weighting agents. It is desirable to avoid this settling as this can produce variations in slurry density, leading to possible loss of well control due to annular invasion.

### 6.1.7 Viscometer

To determine the rheological properties of the cement slurries, proper equipment is needed. Viscosity of a fluid can be determined by using a coaxial cylinder viscometer, shown in Fig. 18, as described by Savins and Roper [18]. In this instrument the cement slurry is contained in a cup and placed under the viscometers bob and rotor. The slurry is then sheared between the rotor (the outer sleeve) and the bob (inner cylinder). As the bob is connected to a torsion spring, the required torque needed to shear the slurry is reflected on a dial reading showing values from 0 to 300. The rotor can spin on preselected rotational speeds ranging from 1 to 600 rpm, and the standard speeds used in Schlumberger is 3, 6, 30, 60, 100, 200 and 300 rpm. During a test, torque readings corresponding to the rotational speed is recorded, first in ascending order, then decending order, as the fluid is sheared for 10 sec at each speed. This is known as a hysteresis loop with the goal of detecting true time-dependent effects. The dial readings and rotational speeds are then converted to shear stress in lbf/100 $\text{ft}^2$ (or Pa) and shear rate in $\text{s}^{-1}$, respectively.

This 12 speed instrument can then cover shear-rate range from at least 5 $\text{s}^{-1}$ to 1,022 $\text{s}^{-1}$, and shear-stress range from 0 to 153 Pa (0 to 320 lbf/100 $\text{ft}^2$). It is then assumed that a length correction of 1.064% is applied to account for end effects and that these values are calculated at the inner cylinder surface. It is also possible to use torsion springs with one-fifth, one-half, two, or five times the stiffness of the standard spring. Of these values we can generate a shear-stress/shear-rate plot. The nature of the curve then decides which rheological model the fluid falls in under. For the the cement samples used in this thesis, the appropriate rheological model was Bingham plastic. Once the rheological model is established, the necessary fluid parameters, like plastic viscosity and yield value, can be determined.

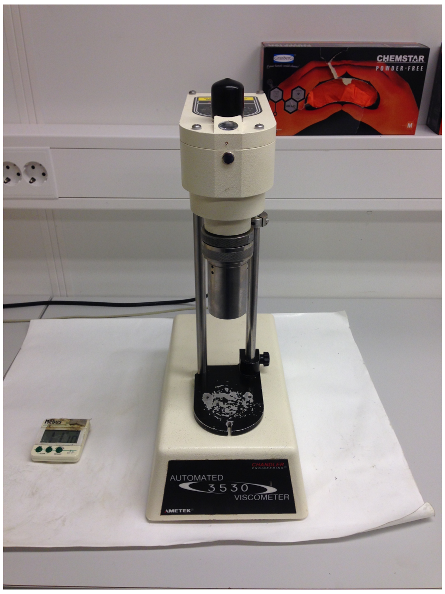


**Figure 18:** Viscometer used to determine the viscosity of a cement slurry.

## 6.2 Gel strength

Under static conditions, the measure of attractive forces that exists between particles is called gel strength. For flowing conditions, this measure is called yield value and is expressed in Pa or lbf/100 ft$^2$.

Gel strength is normally measured after the hysteresis loop, as is done in this thesis, but may also done as an independent measure. In the lab, 10-sec gel strength and 10-min gel strength is measured respectively. This means that the viscometer is turned off for 10 seconds, after which the rotational speed is set to 3 rpm and the following maximum reading is the 10-sec gel strength. The same procedure is done for the 10-min gel strength after the viscometer is shut off for a period of 10 minutes.

## 6.3 Data analysis

Before we are able to convert the rotational speeds, $\Omega$, and dial readings, $\theta$, to shear rates and shear stresses, we need to average the ramp-up/ramp-down readings for any given rotational speed. The shear rate and shear stress at the bob (inner cylinder) are then calculated using the following equations[17]:

$$\dot{\gamma} = 16.28 \times \Omega, \tag{16}$$

where $\Omega$ is in rad/s, or

$$\dot{\gamma} = 1.705 \times \Omega, \tag{17}$$

where $\Omega$ is in rpm.

$$\tau = 0.5109 \times \theta, \tag{18}$$

where $\tau$ is in Pa, or

$$\tau = 1.067 \times \theta, \tag{19}$$

where $\tau$ is in lbf/100 ft$^2$. Using equation (18) we can now convert the 10-sec and 10-min gel strength values to Pa.

Dargaud and Boukhelifa [17] points out that the shear-rate formula is only truly valid for Newtonian fluids, but that the values for other fluids are reasonably accurate. The shear-rate/shear-stress values are then used in the model that best fits the data.

## 6.4 Additives and LCMs

With the help of Schlumberger and MI SWACO, the procurement of four LCMs was made possible. These materials were SafeCarb 250, OptiSeal 2, OptiSeal 4 and G-Seal and are the basis for all the tests. By recommendation from MI SWACO the chosen LCM concentrations would be 100 kg/m$^3$, 150 kg/m$^3$ and 200 kg/m$^3$.

### 6.4.1 B411 - Liquid Antifoam

B411 is used as an antifoam liquid in treatment of oil and gas wells. The liquid is not classified as environmentally harmful, it does not pose a considerable threat to health and is considered inert. It is stable under normal conditions and for its recommended use.

### 6.4.2 B165 - Environmentally Friendly Dispersant

B165 is used as a cement additive in treatment of oil and gas wells and it is classified as environmentally friendly. It is used to reduce both the yield value and plastic viscosity of the cement slurry.

### 6.4.3 SafeCarb 250

SafeCarb 250 is made from ground marble, which gives it a physical appearance of a white powder, and is a acid-soluble, high-purity, calcium carbonate. It is used as a bridging and weighting agent in drill-in, wokover/completion fluids and drilling operations. Due to its high purity, which makes it more soluble in acid, and high hardness it is often preferred over limestone.

SafeCarb has a particle size distribution where D10 is 65 $\mu$m, D50 is 160 $\mu$m and D90 is 381 $\mu$m. Its high hardness makes it able to resist degradation of particle-size over time, it is also essentially inert, meaning that it has minimum effect on fluid properties.

Additional information regarding SafeCarb 250 may be found in Fig.D.1

### 6.4.4 G-Seal

G-seal is a coarse-sized graphite used to bridge and seal permeable formations. G-Seal may also reduce the differential-pressure sticking tendencies when drilling in depleted zones where high differential pressures exist by controlling seepage loss. In partial to sever lost-circulation zones it may even be used to gain control of such a situation. Additionally it is completely inert and will therefore not affect rheological properties. G-Seal also increases lubricity, reducing torque and drag as a result. The particle size distribution for G-Seal is 188 $\mu$m for D10, 406 $\mu$m for D50 and 734 $\mu$m for D90.

Additional information about G-Seal can be found in Fig.D.2

### 6.4.5 OptiSeal II

OptiSeal II is a blend between graphite and calcium carbonate designed to be used as a seepage and lost circulation material in oil and water based drilling fluids. It is effective at plugging and sealing naturally occurring and drilling-induced fractures, with apertures up to 1200 $\mu$m. It prevents seepage losses, reduces wall cake permeability, helps prevent differential sticking and has minimum effects on drilling fluids rheology at normal dosages. Due to graphite being blended with the calcium carbonate, the physical appearance of OptiSeal II is a black and white speckled powder.

OptiSeal II has a particle size distribution of D10 is 53 $\mu$m, D50 is 479 $\mu$m and D90 is 927 $\mu$m.

Fig.D.3 contains more information on OptiSeal II.

### 6.4.6 OptiSeal IV

OptiSeal IV is a calcium carbonate designed to be used as a seepage and lost circulation material. As OptiSeal II, it is very effective for plugging and sealing naturally occurring and drilling-induced fractures, with apertures up to 1200 1200 $\mu$m. It prevents seepage losses, reduces wall cake permeability, helps prevent differential sticking, is acid-soluble which minimises formation damage, and has minimum effects on drilling fluids rheology at normal dosages.

OptiSeal IV has a particle size distribution of D10 is 91 $\mu$m, D50 is 456 $\mu$m and D90 is 851 $\mu$m.

Fig.D.4 contains more information on OptiSeal II.

## 6.5 Test procedure

This subsection will present the lab work of the thesis. It will cover practical considerations that had to be made and operational procedures of how the various tests were conducted.

The idea behind this thesis is to use a base slurry as a comparative foundation against the LCM-added slurries. Therefore, a test of a base slurry was conducted for every test done. For this thesis the following tests were conducted:

- Rheology
- Static Gel Strength Analyser (SGSA)
- Thickening time (TT)

To keep consistent results, the same cement blend and chemicals were used to perform all the test. Furthermore, the same machines were used to perform the tests where practically possible. For example, the same atmospheric consistometer and viscometer was used to perform all the rheology tests. Seeing that a SGSA test may take a minimum of 24 hours to complete, using only one machine for all tests was viewed as counterproductive. Testing on several machines was therefore necessary to produce the amount of results wanted.

The recipe used for the testing was initially used as an abandonment plug for a well on the Heidrun rig. No changes were done to recipe, although new cement and chemicals were used. All pressures and temperatures was chosen from what the actual conditions were in that well.

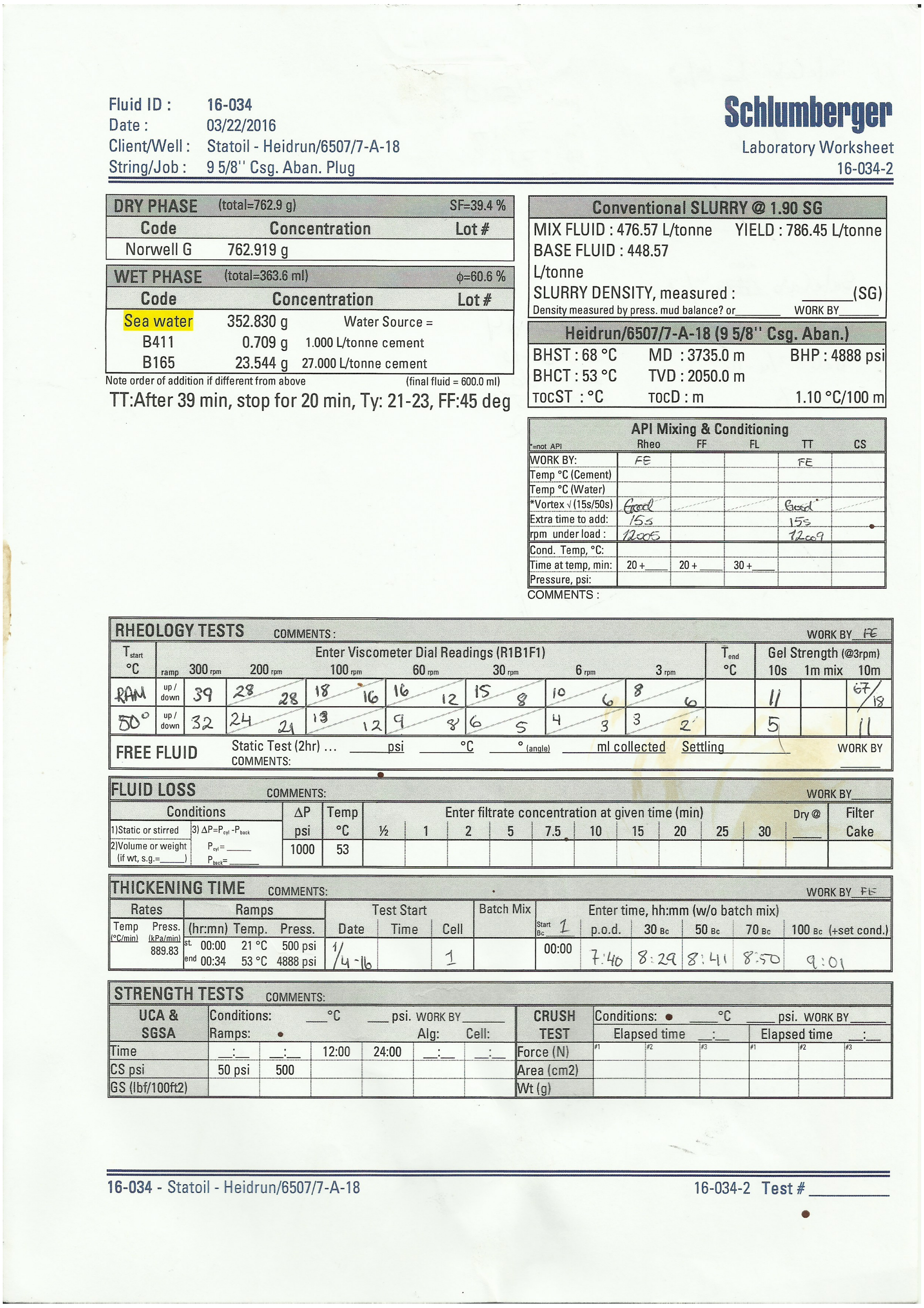
### 6.5.1 Mixing of the cement slurry

Fig. 19 depicts the worksheet used for base slurry tests. It shows the composition of the cement slurry as well as the results of the test conducted on it.

Each test started with the weighing of the slurry components. The slurry was then mixed according to API RP 10B in the mixing device.

An important aspect of the mixing is that the worksheet is considering a final volume of 600 mL. This is only valid for the base slurry as a new worksheet was not adjusted for the addition of each LCM. This means that the actual final volume of the LCM-added slurries might be higher and therefore might affect the results to be shown in Section 6.5.2.

After the cement slurry was prepared, the slurry was transferred to an overhead stirrer to mix in the LCM. LCM was added at 3000 rpm and mixed at 5000 rpm for 30 seconds.



Fluid ID : 16-034
Date : 03/22/2016
Client/Well : Statoil - Heidrun/6507/7-A-18
String/Job : 9 5/8'' Csg. Aban. Plug

**Schlumberger**
Laboratory Worksheet
16-034-2

| DRY PHASE (total=762.9 g) | | SF=39.4 % |
|---|---|---|
| Code | Concentration | Lot # |
| Norwell G | 762.919 g | |

| WET PHASE (total=363.6 ml) | | | ϕ=60.6 % |
|---|---|---|---|
| Code | Concentration | | Lot # |
| Sea water | 352.830 g | Water Source = | |
| B411 | 0.709 g | 1.000 L/tonne cement | |
| B165 | 23.544 g | 27.000 L/tonne cement | |

Note order of addition if different from above (final fluid = 600.0 ml)

TT:After 39 min, stop for 20 min, Ty: 21-23, FF:45 deg

**Conventional SLURRY @ 1.90 SG**
MIX FLUID : 476.57 L/tonne YIELD : 786.45 L/tonne
BASE FLUID : 448.57 L/tonne
SLURRY DENSITY, measured : ________(SG)
Density measured by press. mud balance? or________ WORK BY________

**Heidrun/6507/7-A-18 (9 5/8'' Csg. Aban.)**
BHST : 68 °C MD : 3735.0 m BHP : 4888 psi
BHCT : 53 °C TVD : 2050.0 m
TOCST : °C TOCD : m 1.10 °C/100 m

**API Mixing & Conditioning**

| *=not API | Rheo | FF | FL | TT | CS |
|---|---|---|---|---|---|
| WORK BY: | FE | | | FE | |
| Temp °C (Cement) | | | | | |
| Temp °C (Water) | | | | | |
| *Vortex √ (15s/50s) | Good | | | Good | |
| Extra time to add: | 15s | | | 15s | |
| rpm under load : | 12005 | | | 12009 | |
| Cond. Temp, °C: | | | | | |
| Time at temp, min: | 20 +____ | 20 +____ | 30 +____ | | |
| Pressure, psi: | | | | | |

COMMENTS :

**RHEOLOGY TESTS** COMMENTS : WORK BY FE

| T_start °C | ramp | 300 rpm | 200 rpm | 100 rpm | 60 rpm | 30 rpm | 6 rpm | 3 rpm | T_end °C | Gel Strength (@3rpm) 10s | 1m mix | 10m |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| RAM | up / down | 39 | 28<br>28 | 18<br>16 | 16<br>12 | 15<br>8 | 10<br>6 | 8<br>6 | | 11 | | 67/18 |
| 50° | up / down | 32 | 24<br>21 | 13<br>12 | 9<br>8 | 6<br>5 | 4<br>3 | 3<br>2 | | 5 | | 11 |

**FREE FLUID** Static Test (2hr) … ______psi ______°C ______° (angle) ______ml collected Settling ______ WORK BY ______
COMMENTS:

**FLUID LOSS** COMMENTS: WORK BY______

| Conditions | | ΔP psi | Temp °C | ½ | 1 | 2 | 5 | 7.5 | 10 | 15 | 20 | 25 | 30 | Dry @ ____ | Filter Cake |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1)Static or stirred<br>2)Volume or weight (if wt, s.g.=_____) | 3) ΔP=P_cyl -P_back<br>P_cyl= ______<br>P_back= ______ | 1000 | 53 | | | | | | | | | | | | |

Enter filtrate concentration at given time (min)

**THICKENING TIME** COMMENTS: WORK BY FE

| Rates Temp (°C/min) | Rates Press. (kPa/min) | Ramps (hr:mn) | Temp. | Press. | Test Start Date | Time | Cell | Batch Mix | Start Bc 1 | p.o.d. | 30 Bc | 50 Bc | 70 Bc | 100 Bc (+set cond.) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 889.83 | st. 00:00<br>end 00:34 | 21 °C<br>53 °C | 500 psi<br>4888 psi | 1/4-16 | | 1 | | 00:00 | 7:40 | 8:29 | 8:41 | 8:50 | 9:01 |

Enter time, hh:mm (w/o batch mix)

**STRENGTH TESTS** COMMENTS:

| UCA & SGSA | Conditions: ___°C ___ psi. WORK BY________ Ramps: | | Alg: | | Cell: | |
|---|---|---|---|---|---|---|
| Time | __:__ | __:__ | 12:00 | 24:00 | __:__ | __:__ |
| CS psi | 50 psi | 500 | | | | |
| GS (lbf/100ft2) | | | | | | |

| CRUSH TEST | Conditions: _____°C _____ psi. WORK BY______ Elapsed time __:__ | | | Elapsed time __:__ | | |
|---|---|---|---|---|---|---|
| | #1 | #2 | #3 | #1 | #2 | #3 |
| Force (N) | | | | | | |
| Area (cm2) | | | | | | |
| Wt (g) | | | | | | |

16-034 - Statoil - Heidrun/6507/7-A-18 16-034-2 Test #__________

**Figure 19:** Worksheet used for tests

### 6.5.2 Rheology tests

After mixing of the cement slurry a portion of it was poured into a cup for conditioning in an atmospheric consistometer, while another was prepared for rheology test. This initial rheology test is done right after mix (RAM) and represents the surface reading of the cement slurry at around 21°C. The slurry in the atmospheric consistometer is conditioning for 30 minutes after which a new rheology test is performed to determine the downhole viscosity readings of the cement slurry. These readings were done at 50°C to simulate downhole conditions the slurry might be subjected to.

It was assumed that the added LCM would prevent any fluids from leaking out of the slurry, any further test of free fluid or fluid loss was therefore omitted.

### 6.5.3 Thickening time tests

SafeCarb 250 was the only LCM which TT tests were performed on. This was mainly due to SafeCarb being the only LCM with enough material to conduct tests with. The chosen concentrations for SafeCarb were 100 kg/m$^3$ and 200 kg/m$^3$. The tests were conducted on the dual pressurised consistometers illustrated in Fig. 15.

As with the other tests, a base slurry was first tested after which the SafeCarb concentrations were done. The simulated test conditions of the well were set to 4888 psi and 53 °C.

### 6.5.4 SGSA tests

SGSA tests were performed, like the TT tests, on both the highest and lowest LCM concentration on the machines pictured in Fig. 16. The ramp times for the test was 1 hour to 53°C and then 23 hours to 68°C without preconditioning of the cell. The pressure in the tests were set to 4500 psi for all the tests. Cement slurries were prepared as explained in Section 6.5.1 according to API RP 10B.

## 6.6 Test Results

This subsection presents the individual results of the performed tests in column charts. This way, any deviations from the base slurry can easily be seen. Rheology results can be found in Appendix A, while all the individual charts for TT and SGSA results can be found in Appendix B and Appendix C, respectively.

### 6.6.1 Rheology results

After the initial rheology test of the base slurry, it soon became clear that using the standard BOB1 with the standard cup would not suffice. Even at low concentrations of added LCM, a "grating" sound could be heard from between the bob and the rotor as particles would get stuck between them. Additionally, a clear reading of the dial was not possible as a constant jerking motion of the dial would occur as a result of the grating. At higher LCM concentrations, the grating would also be present as particles would get stuck between the rotor and the cup. As this proved to give inaccurate results, the next tests were therefore done on a BOB5 and using a larger cup to leave enough space for the LCM. A retest of the base slurry and SafeCarb 250 with BOB5 and the larger cup was also done to get a more correct comparative foundation.

A total of 34 rheology tests were performed for surface and downhole readings. In Appendix A, the full extent of the test may be shown with readings from the hysteresis loop as well as 10-second and 10-minute gel strengths. In Fig.20 to Fig.23 we have two comparisons: the clustered columns marked "Base Slurry" and "SafeCarb 250 BOB1" illustrates test done with the normal cup and BOB1 used for cement readings. The other columns shows the results with a larger cup and BOB5.

Fig. 20 and Fig. 21 illustrates the surface and downhole results, respectively, for viscosity in cP.

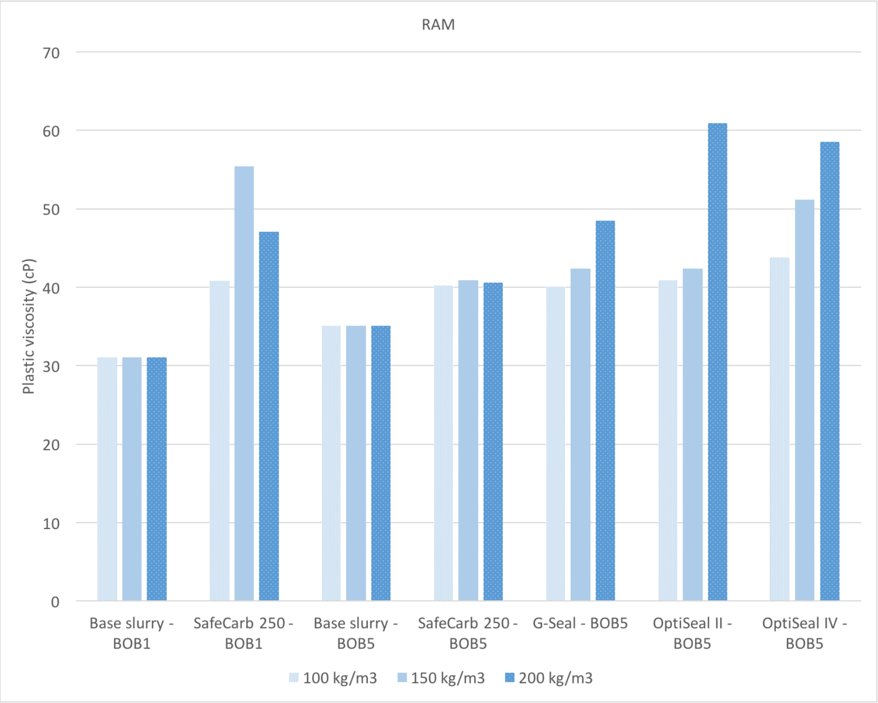


**Figure 20:** Viscosity results RAM.

From the first two clustered columns marked "Base Slurry" and "SafeCarb 250 BOB1" in Fig. 20 it is evident that the results are affected by a too small cup and too small gap between the outer rotor and bob. The 150 kg/m$^3$ shows a large spike before the viscosity drops for the 200 kg/m$^3$ concentration.

The next five clustered columns do, however, show some interesting developments. SafeCarb 250 seems to be stable at 40 cP for all three concentrations, and therefore it is reasonable to assume that it has not affected the cement slurry in terms of viscosity. G-Seal, OptiSeal II and OptiSeal IV all show an increase in viscosity, at surface conditions, as the concentrations increase. The 200 kg/m$^3$ concentration of OptiSeal II yields the highest viscosity increase of 73,5%.

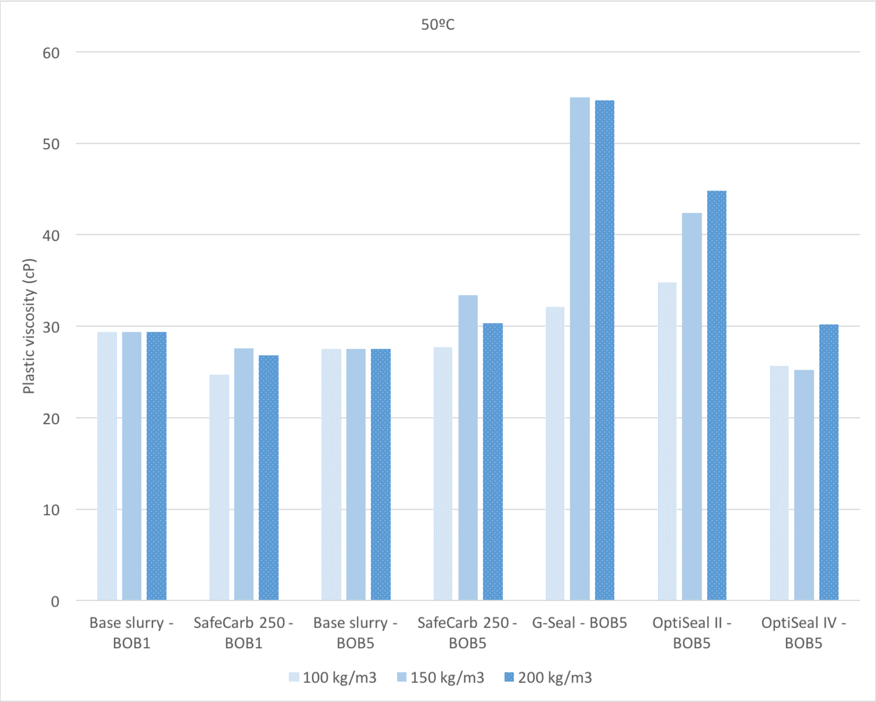


**Figure 21:** Viscosity results at 50°C.

Fig. 21 again shows discrepancies between "Base Slurry" and "SafeCarb 250 BOB1". The viscosity of SafeCarb 250 was reduced below the base slurry and, as with the surface readings, showed a drop in viscosity on the 200 kg/m$^3$ concentration.

As we look at the BOB5 columns the readings does not show the same pattern for all the materials as on the surface readings. SafeCarb 250 with BOB5 is not as stable around a value of viscosity; showing an increase on 150 kg/m$^3$ before it drops again at 200 kg/m$^3$. G-Seal has a 100% higher viscosity than base slurry after 100 kg/m$^3$ concentration, even exceeding the viscosity of its surface reading for both the 150 kg/m$^3$ and 200 kg/m$^3$. OptiSeal II keeps a steady increase in viscosity with increasing concentration. Optiseal IV does show a discrepancy between the concentrations as it drops in viscosity after 100 kg/m$^3$ to 8,36% lower than base slurry and then increases again on the 200 kg/m$^3$ concentration. The 150 kg/m$^3$ concentration of G-Seal has the highest increase of 100% from the base slurry.

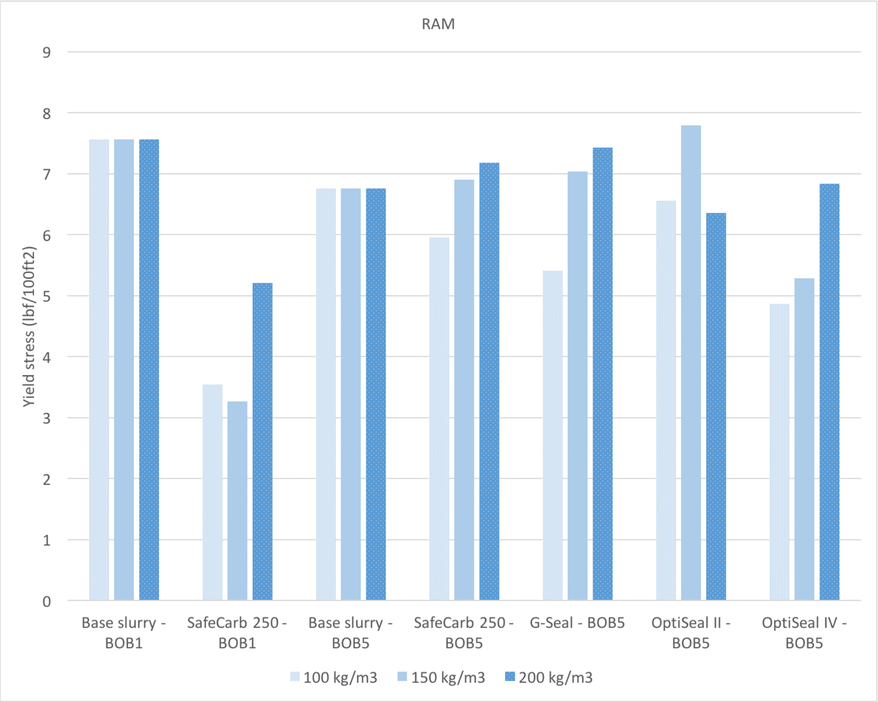


**Figure 22:** Yield stress results at RAM.

Fig. 22 shows a great difference between "Base Slurry" and "SafeCarb 250 BOB1". The base slurry shows a yield stress of 7,56 lbf/100ft$^2$ while SafeCarb 250 starts off at 3,54 lbf/100ft$^2$ for the 100 kg/m$^3$ concentration, ending at 5,21 lbf/100ft$^2$ for the 200 kg/m$^3$ concentration. Again, these values for SafeCarb should not be trusted as they are inaccurate.

Looking at the BOB5 results we see that SafeCarb 250, G-Seal and OptiSeal IV all have increasing yield stress with increasing concentrations. These LCM also start out with a lower yield stress than the base slurry. SafeCarb 250 and G-Seal, however, exceeds the base slurry yield stress on the 150 kg/m$^3$ concentration. OptiSeal IV only exceeds the base slurry yield on the 200 kg/m$^3$ concentration.

OptiSeal II indicates that the yield stress increases with increasing concentration, but dips below the 100 kg/m$^3$ concentration at 200 kg/m$^3$ concentration. The OptiSeal II 150 kg/m$^3$ concentration sees the highest yield stress overall with a 15,24% increase, although the lowest yield stress can be seen on the 100 kg/m$^3$ concentration of OptiSeal IV with 28,1% decrease.

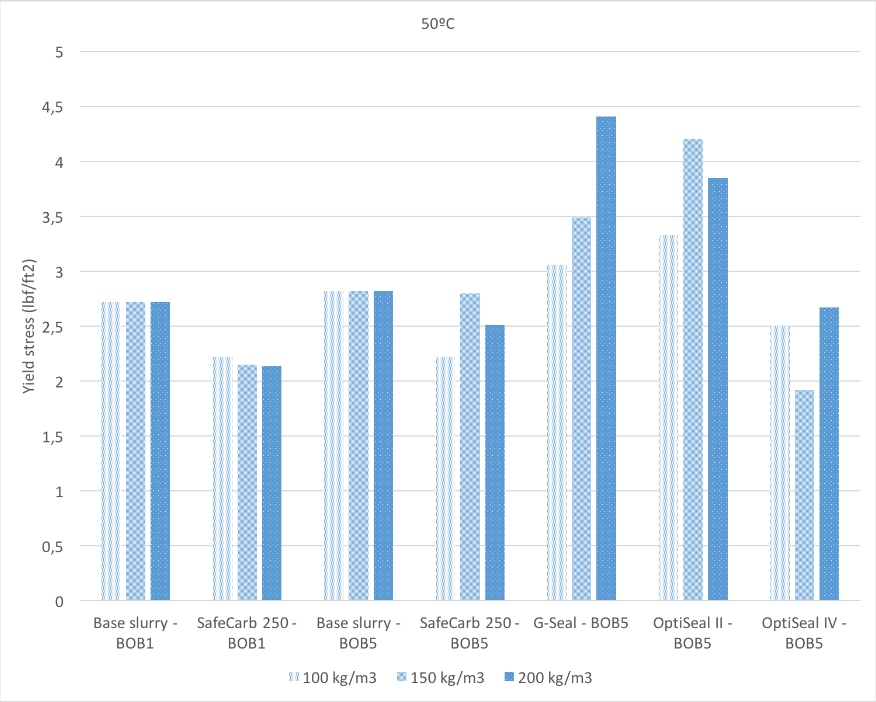


**Figure 23:** Yield stress results at 50 °C.

In Fig. 23 we see the yield stresses at 50°C. Generally, we see that all the slurries has decreased in yield stress at downhole compared to surface conditions. With "Base Slurry" and "SafeCarb 250 BOB1" columns we see that SafeCarb has stabilised at around 2,15 lbf/100ft$^2$.

The BOB5 readings shows some irregularities with SafeCarb, OptiSeal II and OptiSeal IV. SafeCarb and OptiSeal II both increase on 150 kg/m$^3$ concentration before they decrease on the 200 kg/m$^3$ concentration. OptiSeal IV decreases after 100 kg/m$^3$ concentration before it again increases on the 200 kg/m$^3$ concentration. G-Seal is the only one which increased in yield stress steadily with concentration, the highest yield stress showing 56,4% increase from that of the base slurry at the 200 kg/m$^3$ concentration.

OptiSeal IV decreases the most with 31,9% from the base slurry on the 150 kg/m$^3$ concentration.

It is likely that some of this effect is caused by the addition of material; however, the differences between the individual materials indicate that they can affect the slurry differently. SafeCarb 250 and OptiSeal IV, which is both made from calcium carbonate, saw different effects on the base slurry. SafeCarb had little result through the concentrations while OptiSeal IV saw an increase at surface, but had a 8,36%

lower viscosity at downhole conditions compared to base slurry.

When looking at the decrease in viscosity and yield stress, OptiSeal IV and SafeCarb, being both pure calcium carbonates, has around the same specific gravity of 2,7 sg; however, SafeCarb has a much lower particle size distribution with a D50 of 160 $\mu$m. The higher size distribution of OptiSeal IV might explain why it decreases in yield stress at surface, and viscosity and yield stress at downhole conditions while SafeCarb do not change as much.

### 6.6.2 TT results

The amount of thickening time tests performed was lower than what was initially hoped for. The reason for this was the availability of LCM. Most of the LCM was used for the rheology tests and the SGSA tests, hence the remaining material for thickening time tests was only enough for a SafeCarb 250 comparison. These results are presented in Fig. 24

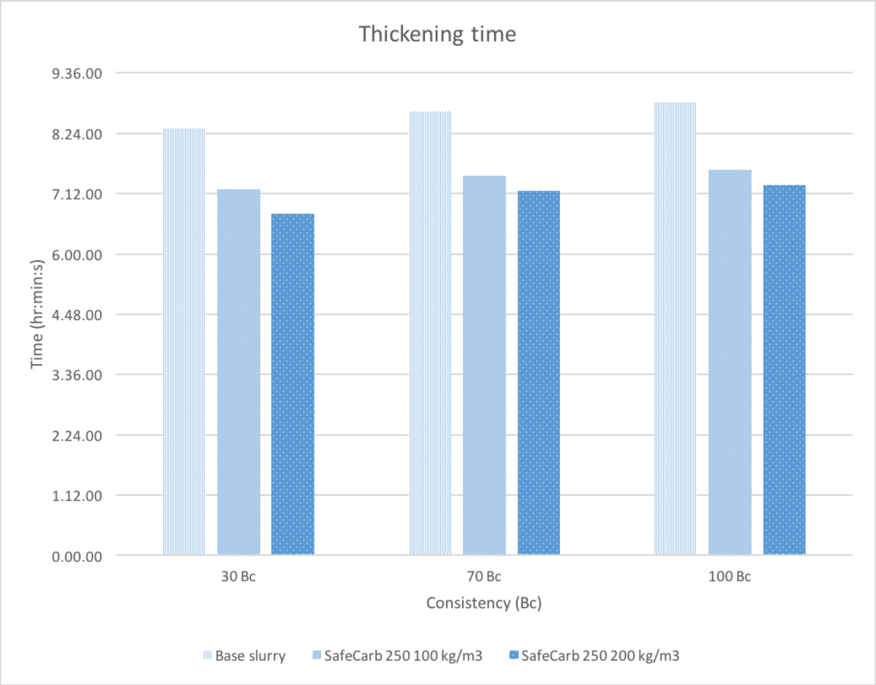


**Figure 24:** Thickening time results.

From Fig. 24 we see a trend as the concentration of SafeCarb 250 increases. Generally we see that thickening time was reduced with incremental increase of SafeCarb 250 concentrations to as low as 18% of the base slurry. The largest difference can be seen between the base slurry and the 100 kg/m$^3$ concentration. As explained in Section 6.1.3, 70 Bc is considered the maximum consistency the cement can have and still be pumpable. At this consistency there is a 1 hour and 17 minutes difference between the base slurry and the 100 kg/m$^3$ concentration, an 18% decrease. Between the 100 kg/m$^3$ and the 200 kg/m$^3$ concentrations the difference was not as great, but an 18 minutes gap was still noted.

SafeCarb is said to be essentially inert, any accelerative effect on thickening time may therefore be ruled out. The reduced thickening time is likely to be due to the addition of material without altering the original recipe.

### 6.6.3 SGSA results

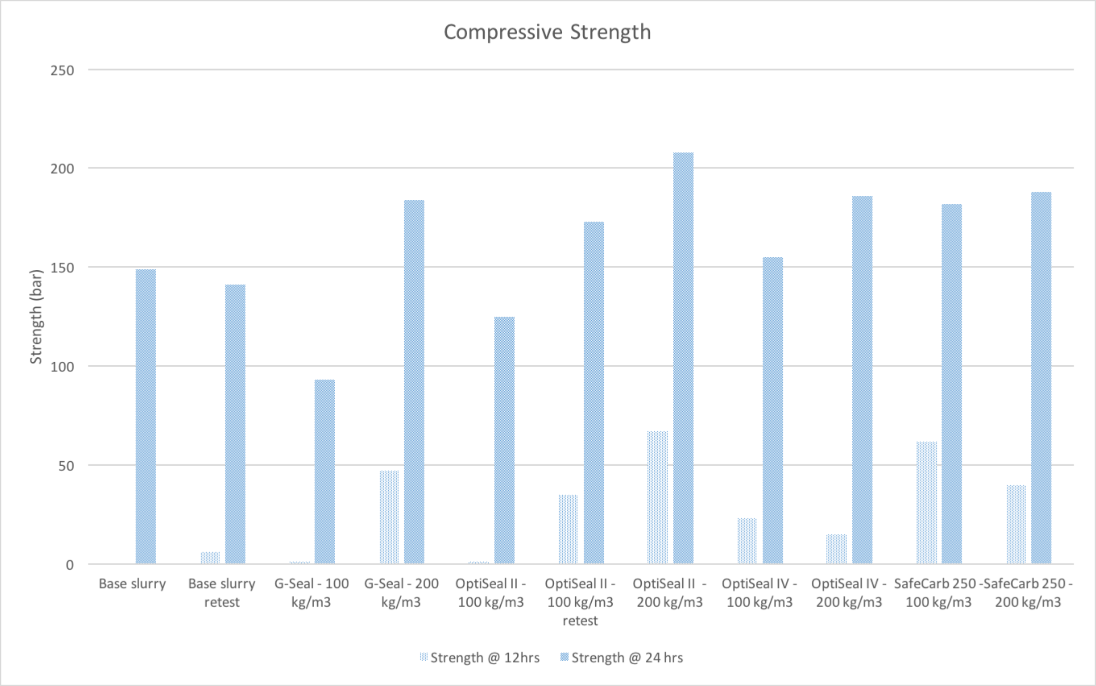


**Figure 25:** Compressive strength results.

In Fig. 25 we see the results of the SGSA tests performed. The blue columns, as the figure explains, are the developed strength after 12 hours while the orange columns are after 24 hours. The overall results of the test showed a higher compressive strength on the slurries with added LCM compared to the base slurry. There were, however, some issues regarding slurries not reaching it's correct ramp temperature and/or pressure.

The first base slurry test was not programmed correctly to the right temperature. As seen on the chart in Fig.C.1, the slurry only reached a temperature of 63°C, resulting in an inaccurate compressive strength development. A retest of the base slurry was therefore necessary, yielding a compressive strength of 141 bar. A retest of OptiSeal II at the 100 kg/m$^3$ concentration was also performed as the first test yielded 125 bar. This was suspected a discrepancy as the 200 kg/m$^3$ concentration resulted in a compressive strength as high as 208 bar. The following retest resulted in a very different compressive strength, 173 bar.

Fig. 25 also shows that the graphite and calcium carbonate based OptiSeal II yields the overall highest compressive strength at the 200 kg/m$^3$ concentration of 208 bar, a 47,5% increase. G-Seal had a decrease of 34% from the base slurry on the 100 kg/m$^3$ concentration. As both materials contains graphite, either the 100 kg/m$^3$ concentration of G-Seal was a discrepancy or the increase on OptiSeal II could be due to the calcium carbonate component of the material. The other LCMs reached an average compressive strength of 186 bar on the 200 kg/m$^3$ concentration.

### 6.6.4 Sources of Error

It is hard to avoid all sources of errors that might affect the outcome of the performed tests. Minimising these sources is therefore important to be able to provide reproductive and consistent results throughout all of the test. This section will try to cover all the possible errors that might have affected this thesis.

Having performed all the tests in Schlumberger's lab, which have their machines calibrated by a specialist company at regular intervals, should make calibration a small source of error. Even so, it can not be completely ruled out. Examples of such errors could be seconds off on a timer, scale readings are off when weighing in, incorrect viscometer dial readings, incorrect pressure/temperature in SGSAs and TT machines, or incorrect density readings from pycnometer.

As mentioned in Section 6.5.1 the worksheet does not account for the LCM in the final volume of the base slurry. A new worksheet accounting for the LCM would change the results as the comparative foundation would change.

The software used for converting the dial readings and rotational speeds into viscosity and yield stress has an option for tests which uses BOB5; however, it might not have been designed for slurries with LCM added to it. This might be a source of error and potentially yield inaccurate results.

Human error is considered the largest source of error in this thesis. All the tests were performed by a person without extensive experience with laboratory work. Some experience was acquired during one year working as a lab technician performing similar tests, but errors could still happen. In this thesis, such errors may be related to: reading errors of the worksheet, resulting in wrong composition of cement slurry; incorrect dial reading on viscometer, leading to wrong yield stress and viscosity values; and wrong programming of TT and SGSA machines, leading to inaccurate results.

The availability of LCM was a limiting factor in this thesis. Ideally all results should be confirmed by new tests, but the remaining amount of material would only allow for a selected few retests. This leaves a number of tests which should be confirmed but is not. As a result, some of these tests could contain hidden errors which will not be uncovered.

## 6.7 Summary

Laboratory testing equipment enables us to produce information about the properties of a cement slurry. Viscometers produces values which we can convert into the slurry's viscosity and yield stress. Pressurised consistometers records the consistency development and determines how long the slurry is pumpable. The compressive strength of the slurry can be determined using a static gel strength analyser.

SafeCarb 250, OptiSeal II, OptiSeal IV and G-Seal were added to the cement slurries. They vary in material, particle size distribution, properties and applied areas.

The test procedures for each set of tests were discussed before the results were presented. Lastly, all the potential sources of errors were attempted to be uncovered.

# 7 Conclusion

Through the course of this paper the goal have been to understand how LCM affects cement properties. Other studies show little in regards to this and a clear understanding can therefore be hard to obtain. The fact that most cement slurries are tested in a laboratory before they are used in a well, might be explain the lack of studies. It should, however, not exclude the knowledge of how a LCM affects the slurry when it might be used to improve the design of it. As all the LCM used in this thesis claims to be inert, any results from the tests is unlikely to be due to a chemical reaction between the material and the slurry but rather because of the physical properties of the individual LCMs. The findings of this thesis are summarised below;

1. OptiSeal II and G-Seal both contains graphite, this indicates that graphite materials are able to produce higher viscosity and yield stress values in cement slurries at both surface and downhole conditions. High particle size distributed calcium carbonates, like OptiSeal IV, decreases viscosity and yield stress at downhole conditions, and yield stress at surface conditions compared to base slurry.
2. High particle size distributed calcium carbonates lowers thickening time by 18% compared to base slurry.
3. Compressive strength was 47,5% higher on the highest concentration of LCM containing both graphite and calcium carbonate, like OptiSeal II, compared to base slurry. For G-Seal, a pure graphite LCM, a 34 % decrease was noted compared to base slurry.

# 8 Future Work

The few studies done on how LCM affects cement properties could be an indication that we don't know enough about it. The results presented in Section 6.6 clearly shows that the impact LCM has on cement slurries are noticeable, and in some cases very significant. In the bullet points below are some suggestions for future work;

- More tests with a broader range of LCM, with worksheets accounting for the addition of material, could help relate specific components and particle size distribution of materials to increases/decreases in slurry properties. Testing graphite based LCM against other LCM for effect on yield stress and viscosity could help verify or disprove the findings in this thesis that graphite based slurries see an increase on these properties.
- Testing more cement slurries with added LCM in pressurised consistometers will provide more thickening time results. These results can then be used to establish a relationship between different LCM and possible effects they have on the cement slurry.
- Additional SGSA tests may be done to further investigate any effects LCM may have on cement slurry properties. As per this thesis findings, the graphite and calcium carbonate components of OptiSeal II yielded the highest compressive strength overall. New studies could investigate whether other combinations can yield better compressive strength characteristics in cement slurries.
- The lack of materials impaired the ability for quality control of all the performed tests; hence, confirmation tests could help solidify the findings in this thesis.

# References

[1] E.B. Nelson and M. Michaux. Chemistry and characterization of portland cement. In E.B. Nelson and D. Guillot, editors, *Well cementing, Second Edition*, chapter 2, pages 23–48. Schlumberger, Sugar Land, Texas, 2006. ISBN -13: 978-097885300-6./ ISBN-10: 0-9788530-0-8.

[2] BW Ost. Optimum sulfate content of portland cements. *American Ceramic Society Journal*, 53(8), 1974.

[3] IM Silk. Exposure to moisture alters well cement. *Pet. Eng. Int.;(United States)*, 58(3), 1986.

[4] B Vidick, R Oberste-Padtberg, JP Laurent, and F Rondelez. Selective surface determination of the silicate phases in portland cement powders using alkyltrichlorosilane. *Cement and Concrete Research*, 17(4):624–632, 1987.

[5] Siegbert Sprung, K Kuhlmann, and H-G Ellerbrock. Particle size distribution and properties of cement: Ii, water demand of portland cement. *ZKG, Zement-Kalk-Gips, Edition A*, 38(9):528–34, 1985.

[6] WC Goins Jr. How to combat circulation loss. *Oil and Gas Journal*, 51:71, 1952.

[7] G. Daccord et al. Cement-formation interactions. In E.B. Nelson and D. Guillot, editors, *Well cementing, Second Edition*, chapter 6, pages 191–232. Schlumberger, Sugar Land, Texas, 2006. ISBN -13: 978-097885300-6./ ISBN-10: 0-9788530-0-8.

[8] Carl Gatlin, Charles E Nemir, et al. Some effects of size distribution on particle bridging in lost circulation and filtration tests. *Journal of Petroleum Technology*, 13(06):575–578, 1961.

[9] Albert Abrams et al. Mud design to minimize rock impairment due to particle invasion. *Journal of petroleum technology*, 29(05):586–592, 1977.

[10] MA Dick, TJ Heinz, CF Svoboda, M Aston, et al. Optimizing the selection of bridging particles for reservoir drilling fluids. In *SPE International Symposium on Formation Damage Control*. Society of Petroleum Engineers, 2000.

[11] M Kaeuffer. Determination de loptimum de remplissage granulometrique et quelques proprietes sy rattachant. In *Congres International de IAFTPV, Rouen*, pages 1–12, 1973.

[12] TM Nayberg, BR Petty, et al. Laboratory study of lost circulation materials for use in oil-base drilling muds. In *SPE Deep Drilling and Production Symposium*. Society of Petroleum Engineers, 1986.

[13] WH Turki, AS Mackay, et al. Primary cementing across massive lost circulation zones. In *Middle East Oil Technical Conference and Exhibition*. Society of Petroleum Engineers, 1983.

[14] E Messier, D Stiles, R Morgan, et al. Controlling cement tops through use of fiber-based slurries reduces drilling costs. In *Canadian International Petroleum Conference*. Petroleum Society of Canada, 2002.

[15] D. Guillot. Rheology and flow of well cement slurries. In E.B. Nelson and D. Guillot, editors, *Well cementing, Second Edition*, chapter 4, pages 93–142. Schlumberger, Sugar Land, Texas, 2006. ISBN -13: 978-097885300-6./ ISBN-10: 0-9788530-0-8.

[16] Erik B Nelson and Dominique Guillot. *Well cementing*. Schlumberger, Sugar Land, Texas, 2006.

[17] B. Dargaud and L. Boukhelifa. Laboratory testing, evaluation, and analysis of well cements. In E.B. Nelson and D. Guillot, editors, *Well cementing, Second Edition*, chapter B, pages 627–657. Schlumberger, Sugar Land, Texas, 2006. ISBN -13: 978-097885300-6./ ISBN-10: 0-9788530-0-8.

[18] JG Savins, WF Roper, et al. A direct-indicating viscometer for drilling fluids. In *Drilling and Production Practice*. American Petroleum Institute, 1954.

# A Appendix A - Rheology Results

This appendix contains all the rheology results.

Base Slurry - BOB1

| RAM | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 39 | 39 |
| 200 | 28 | 28 |
| 100 | 18 | 16 |
| 60 | 16 | 12 |
| 30 | 15 | 8 |
| 6 | 10 | 6 |
| 3 | 8 | 6 |
| | | |
| 10 sec gel | 11 | |
| 10 min gel | 67/18 | |
| Pv | 31,1 | cP |
| Ty | 7,56 | lbf/100ft2 |

| 50 degC | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 32 | 32 |
| 200 | 24 | 21 |
| 100 | 13 | 12 |
| 60 | 9 | 8 |
| 30 | 6 | 5 |
| 6 | 4 | 3 |
| 3 | 3 | 2 |
| | | |
| 10 sec gel | 5 | |
| 10 min gel | 11 | |
| Pv | 29,4 | cP |
| Ty | 2,72 | lbf/100ft2 |

Base Slurry - BOB5

| RAM | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 22 | 22 |
| 200 | 18 | 17 |
| 100 | 13 | 11 |
| 60 | 10 | 8 |
| 30 | 10 | 7 |
| 6 | 7 | 6 |
| 3 | 6 | 6 |
| | | |
| 10 sec gel | 11 | |
| 10 min gel | 33 | |
| Pv | 35,1 | cP |
| Ty | 6,76 | lbf/100ft2 |

| 50 degC | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 15 | 15 |
| 200 | 11 | 11 |
| 100 | 7 | 7 |
| 60 | 5 | 5 |
| 30 | 4 | 4 |
| 6 | 3 | 3 |
| 3 | 3 | 2 |
| | | |
| 10 sec gel | 4 | |
| 10 min gel | 11 | |
| Pv | 27,5 | cP |
| Ty | 2,82 | lbf/100ft2 |

SafeCarb 250 - 100 kg/m3 - BOB1

| RAM | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 46 | 46 |
| 200 | 27 | 30 |
| 100 | 16 | 17 |
| 60 | 11 | 11 |
| 30 | 8 | 7 |
| 6 | 6 | 5 |
| 3 | 5 | 5 |
| | | |
| 10 sec gel | 10 | |
| 10 min gel | 39 | |
| Pv | 40,8 | cP |
| Ty | 3,54 | lbf/100ft2 |

| 50 degC | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 27 | 27 |
| 200 | 19 | 18 |
| 100 | 11 | 10 |
| 60 | 7 | 7 |
| 30 | 5 | 4 |
| 6 | 3 | 3 |
| 3 | 2 | 2 |
| | | |
| 10 sec gel | 5 | |
| 10 min gel | 10 | |
| Pv | 24,7 | cP |
| Ty | 2,22 | lbf/100ft2 |

SafeCarb 250 - 150 kg/m3 - BOB1

| RAM | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 62 | 62 |
| 200 | 32 | 39 |
| 100 | 18 | 21 |
| 60 | 14 | 14 |
| 30 | 10 | 9 |
| 6 | 7 | 6 |
| 3 | 5 | 6 |
| | | |
| 10 sec gel | 11 | |
| 10 min gel | 47 | |
| Pv | 55,4 | cP |
| Ty | 3,26 | lbf/100ft2 |

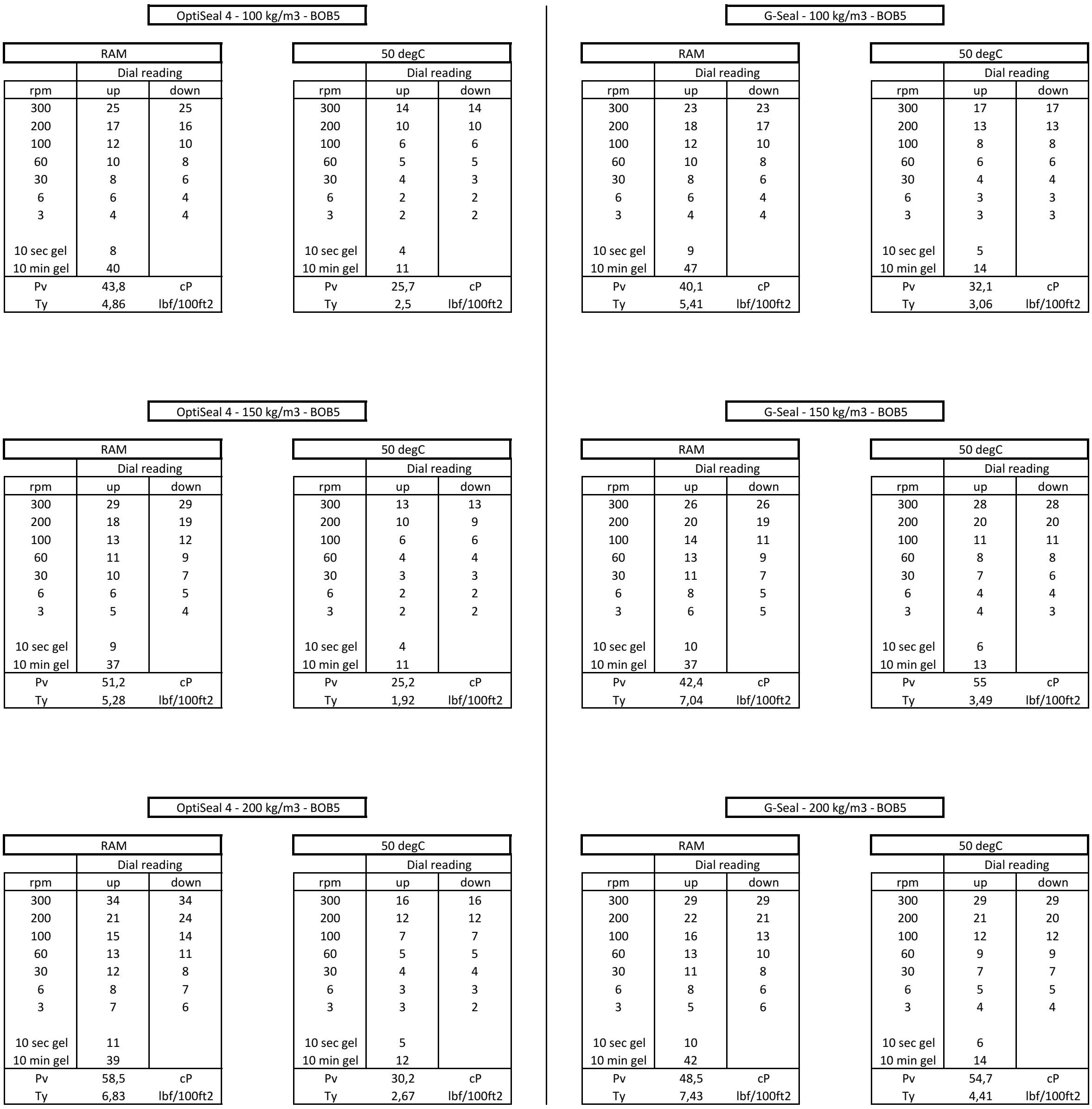
| 50 degC | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 30 | 30 |
| 200 | 21 | 19 |
| 100 | 12 | 11 |
| 60 | 8 | 7 |
| 30 | 6 | 5 |
| 6 | 3 | 2 |
| 3 | 2 | 2 |
| | | |
| 10 sec gel | 5 | |
| 10 min gel | 13 | |
| Pv | 27,6 | cP |
| Ty | 2,15 | lbf/100ft2 |

SafeCarb 250 - 200 kg/m3 - BOB1

| RAM | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 54 | 54 |
| 200 | 32 | 36 |
| 100 | 19 | 20 |
| 60 | 14 | 14 |
| 30 | 10 | 10 |
| 6 | 8 | 7 |
| 3 | 7 | 7 |
| | | |
| 10 sec gel | 10 | |
| 10 min gel | 34 | |
| Pv | 47,1 | cP |
| Ty | 5,21 | lbf/100ft2 |

| 50 degC | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 29 | 29 |
| 200 | 21 | 19 |
| 100 | 11 | 11 |
| 60 | 8 | 7 |
| 30 | 5 | 4 |
| 6 | 3 | 3 |
| 3 | 2 | 2 |
| | | |
| 10 sec gel | 4 | |
| 10 min gel | 11 | |
| Pv | 26,8 | cP |
| Ty | 2,14 | lbf/100ft2 |

**Figure A.1:** Base slurry and SafeCarb 250 - BOB1 rheology



OptiSeal 4 - 100 kg/m3 - BOB5

| RAM | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 25 | 25 |
| 200 | 17 | 16 |
| 100 | 12 | 10 |
| 60 | 10 | 8 |
| 30 | 8 | 6 |
| 6 | 6 | 4 |
| 3 | 4 | 4 |
| | | |
| 10 sec gel | 8 | |
| 10 min gel | 40 | |
| Pv | 43,8 | cP |
| Ty | 4,86 | lbf/100ft2 |

| 50 degC | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 14 | 14 |
| 200 | 10 | 10 |
| 100 | 6 | 6 |
| 60 | 5 | 5 |
| 30 | 4 | 3 |
| 6 | 2 | 2 |
| 3 | 2 | 2 |
| | | |
| 10 sec gel | 4 | |
| 10 min gel | 11 | |
| Pv | 25,7 | cP |
| Ty | 2,5 | lbf/100ft2 |

OptiSeal 4 - 150 kg/m3 - BOB5

| RAM | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 29 | 29 |
| 200 | 18 | 19 |
| 100 | 13 | 12 |
| 60 | 11 | 9 |
| 30 | 10 | 7 |
| 6 | 6 | 5 |
| 3 | 5 | 4 |
| | | |
| 10 sec gel | 9 | |
| 10 min gel | 37 | |
| Pv | 51,2 | cP |
| Ty | 5,28 | lbf/100ft2 |

| 50 degC | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 13 | 13 |
| 200 | 10 | 9 |
| 100 | 6 | 6 |
| 60 | 4 | 4 |
| 30 | 3 | 3 |
| 6 | 2 | 2 |
| 3 | 2 | 2 |
| | | |
| 10 sec gel | 4 | |
| 10 min gel | 11 | |
| Pv | 25,2 | cP |
| Ty | 1,92 | lbf/100ft2 |

OptiSeal 4 - 200 kg/m3 - BOB5

| RAM | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 34 | 34 |
| 200 | 21 | 24 |
| 100 | 15 | 14 |
| 60 | 13 | 11 |
| 30 | 12 | 8 |
| 6 | 8 | 7 |
| 3 | 7 | 6 |
| | | |
| 10 sec gel | 11 | |
| 10 min gel | 39 | |
| Pv | 58,5 | cP |
| Ty | 6,83 | lbf/100ft2 |

| 50 degC | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 16 | 16 |
| 200 | 12 | 12 |
| 100 | 7 | 7 |
| 60 | 5 | 5 |
| 30 | 4 | 4 |
| 6 | 3 | 3 |
| 3 | 3 | 2 |
| | | |
| 10 sec gel | 5 | |
| 10 min gel | 12 | |
| Pv | 30,2 | cP |
| Ty | 2,67 | lbf/100ft2 |

G-Seal - 100 kg/m3 - BOB5

| RAM | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 23 | 23 |
| 200 | 18 | 17 |
| 100 | 12 | 10 |
| 60 | 10 | 8 |
| 30 | 8 | 6 |
| 6 | 6 | 4 |
| 3 | 4 | 4 |
| | | |
| 10 sec gel | 9 | |
| 10 min gel | 47 | |
| Pv | 40,1 | cP |
| Ty | 5,41 | lbf/100ft2 |

| 50 degC | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 17 | 17 |
| 200 | 13 | 13 |
| 100 | 8 | 8 |
| 60 | 6 | 6 |
| 30 | 4 | 4 |
| 6 | 3 | 3 |
| 3 | 3 | 3 |
| | | |
| 10 sec gel | 5 | |
| 10 min gel | 14 | |
| Pv | 32,1 | cP |
| Ty | 3,06 | lbf/100ft2 |

G-Seal - 150 kg/m3 - BOB5

| RAM | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 26 | 26 |
| 200 | 20 | 19 |
| 100 | 14 | 11 |
| 60 | 13 | 9 |
| 30 | 11 | 7 |
| 6 | 8 | 5 |
| 3 | 6 | 5 |
| | | |
| 10 sec gel | 10 | |
| 10 min gel | 37 | |
| Pv | 42,4 | cP |
| Ty | 7,04 | lbf/100ft2 |

| 50 degC | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 28 | 28 |
| 200 | 20 | 20 |
| 100 | 11 | 11 |
| 60 | 8 | 8 |
| 30 | 7 | 6 |
| 6 | 4 | 4 |
| 3 | 4 | 3 |
| | | |
| 10 sec gel | 6 | |
| 10 min gel | 13 | |
| Pv | 55 | cP |
| Ty | 3,49 | lbf/100ft2 |

G-Seal - 200 kg/m3 - BOB5

| RAM | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 29 | 29 |
| 200 | 22 | 21 |
| 100 | 16 | 13 |
| 60 | 13 | 10 |
| 30 | 11 | 8 |
| 6 | 8 | 6 |
| 3 | 5 | 6 |
| | | |
| 10 sec gel | 10 | |
| 10 min gel | 42 | |
| Pv | 48,5 | cP |
| Ty | 7,43 | lbf/100ft2 |

| 50 degC | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 29 | 29 |
| 200 | 21 | 20 |
| 100 | 12 | 12 |
| 60 | 9 | 9 |
| 30 | 7 | 7 |
| 6 | 5 | 5 |
| 3 | 4 | 4 |
| | | |
| 10 sec gel | 6 | |
| 10 min gel | 14 | |
| Pv | 54,7 | cP |
| Ty | 4,41 | lbf/100ft2 |

**Figure A.2:** OptiSeal IV and G-Seal rheology

SafeCarb 250 - 100 kg/m3 - BOB5

| RAM | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 24 | 24 |
| 200 | 17 | 17 |
| 100 | 14 | 11 |
| 60 | 11 | 8 |
| 30 | 9 | 6 |
| 6 | 6 | 5 |
| 3 | 5 | 5 |
| 10 sec gel | 10 | |
| 10 min gel | 34 | |
| Pv | 40,2 | cP |
| Ty | 5,95 | lbf/100ft2 |

| 50 degC | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 15 | 15 |
| 200 | 11 | 11 |
| 100 | 7 | 7 |
| 60 | 5 | 5 |
| 30 | 4 | 4 |
| 6 | 3 | 2 |
| 3 | 3 | 2 |
| 10 sec gel | 4 | |
| 10 min gel | 13 | |
| Pv | 27,7 | cP |
| Ty | 2,76 | lbf/100ft2 |

SafeCarb 250 - 150 kg/m3 - BOB5

| RAM | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 25 | 25 |
| 200 | 19 | 19 |
| 100 | 14 | 11 |
| 60 | 12 | 8 |
| 30 | 11 | 7 |
| 6 | 7 | 5 |
| 3 | 5 | 5 |
| 10 sec gel | 10 | |
| 10 min gel | 41 | |
| Pv | 40,9 | cP |
| Ty | 6,9 | lbf/100ft2 |

| 50 degC | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 17 | 17 |
| 200 | 13 | 14 |
| 100 | 8 | 8 |
| 60 | 5 | 6 |
| 30 | 4 | 4 |
| 6 | 3 | 3 |
| 3 | 3 | 2 |
| 10 sec gel | 4 | |
| 10 min gel | 14 | |
| Pv | 33,4 | cP |
| Ty | 2,8 | lbf/100ft2 |

SafeCarb 250 - 200 kg/m3 - BOB5

| RAM | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 25 | 25 |
| 200 | 18 | 20 |
| 100 | 15 | 12 |
| 60 | 12 | 9 |
| 30 | 10 | 7 |
| 6 | 8 | 6 |
| 3 | 7 | 5 |
| 10 sec gel | 10 | |
| 10 min gel | 40 | |
| Pv | 40,6 | cP |
| Ty | 7,18 | lbf/100ft2 |

| 50 degC | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 16 | 16 |
| 200 | 12 | 11 |
| 100 | 7 | 7 |
| 60 | 5 | 5 |
| 30 | 4 | 4 |
| 6 | 3 | 2 |
| 3 | 3 | 2 |
| 10 sec gel | 4 | |
| 10 min gel | 11 | |
| Pv | 30,3 | cP |
| Ty | 2,51 | lbf/100ft2 |

OptiSeal 2 - 100 kg/m3 - BOB5

| RAM | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 25 | 25 |
| 200 | 18 | 18 |
| 100 | 13 | 12 |
| 60 | 11 | 9 |
| 30 | 10 | 7 |
| 6 | 7 | 6 |
| 3 | 6 | 6 |
| 10 sec gel | 13 | |
| 10 min gel | 53 | |
| Pv | 40,9 | cP |
| Ty | 6,56 | lbf/100ft2 |

| 50 degC | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 19 | 19 |
| 200 | 14 | 13 |
| 100 | 8 | 8 |
| 60 | 6 | 6 |
| 30 | 6 | 5 |
| 6 | 4 | 3 |
| 3 | 3 | 3 |
| 10 sec gel | 4 | |
| 10 min gel | 14 | |
| Pv | 34,8 | cP |
| Ty | 3,33 | lbf/100ft2 |

OptiSeal 2 - 150 kg/m3 - BOB5

| RAM | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 27 | 27 |
| 200 | 19 | 20 |
| 100 | 14 | 13 |
| 60 | 12 | 10 |
| 30 | 11 | 9 |
| 6 | 8 | 8 |
| 3 | 7 | 8 |
| 10 sec gel | 12 | |
| 10 min gel | 57 | |
| Pv | 42,4 | cP |
| Ty | 7,79 | lbf/100ft2 |

| 50 degC | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 23 | 23 |
| 200 | 17 | 17 |
| 100 | 10 | 10 |
| 60 | 8 | 8 |
| 30 | 6 | 6 |
| 6 | 5 | 4 |
| 3 | 4 | 4 |
| 10 sec gel | 5 | |
| 10 min gel | 18 | |
| Pv | 42,4 | cP |
| Ty | 4,2 | lbf/100ft2 |

OptiSeal 2 - 200 kg/m3 - BOB5

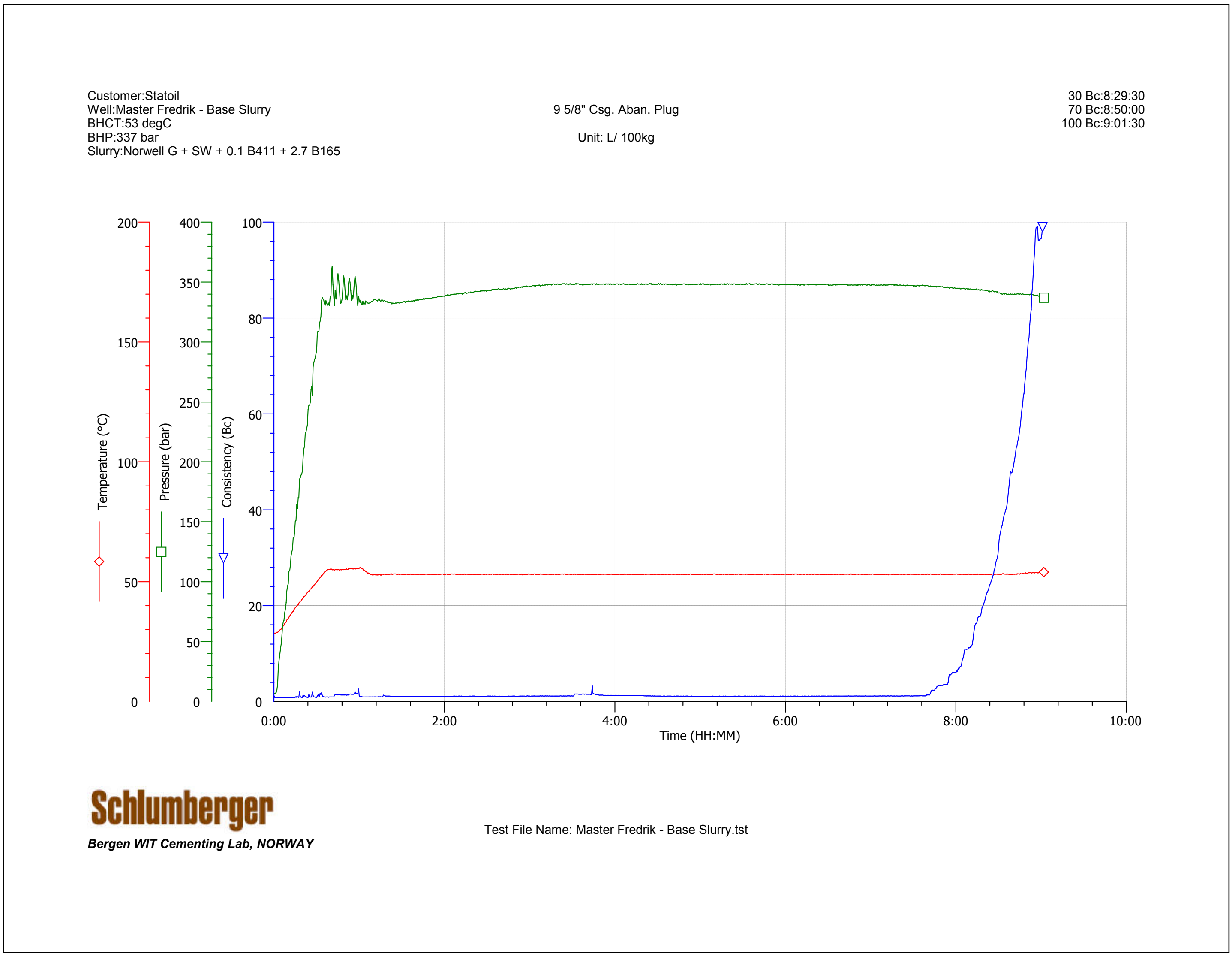
| RAM | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 35 | 35 |
| 200 | 22 | 23 |
| 100 | 14 | 14 |
| 60 | 12 | 10 |
| 30 | 11 | 8 |
| 6 | 8 | 7 |
| 3 | 7 | 7 |
| 10 sec gel | 11 | |
| 10 min gel | 43 | |
| Pv | 60,9 | cP |
| Ty | 6,36 | lbf/100ft2 |

| 50 degC | | |
|---|---|---|
| | Dial reading | |
| rpm | up | down |
| 300 | 24 | 24 |
| 200 | 17 | 17 |
| 100 | 10 | 10 |
| 60 | 8 | 8 |
| 30 | 6 | 5 |
| 6 | 5 | 4 |
| 3 | 4 | 4 |
| 10 sec gel | 5 | |
| 10 min gel | 10 | |
| Pv | 44,8 | cP |
| Ty | 3,85 | lbf/100ft2 |

**Figure A.3:** SafeCarb 250 - BOB5 and OptiSeal II rheology

# B Appendix B - TT Results

This appendix contains all the results from the thickening time tests.



Customer:Statoil
Well:Master Fredrik - Base Slurry
BHCT:53 degC
BHP:337 bar
Slurry:Norwell G + SW + 0.1 B411 + 2.7 B165

9 5/8" Csg. Aban. Plug

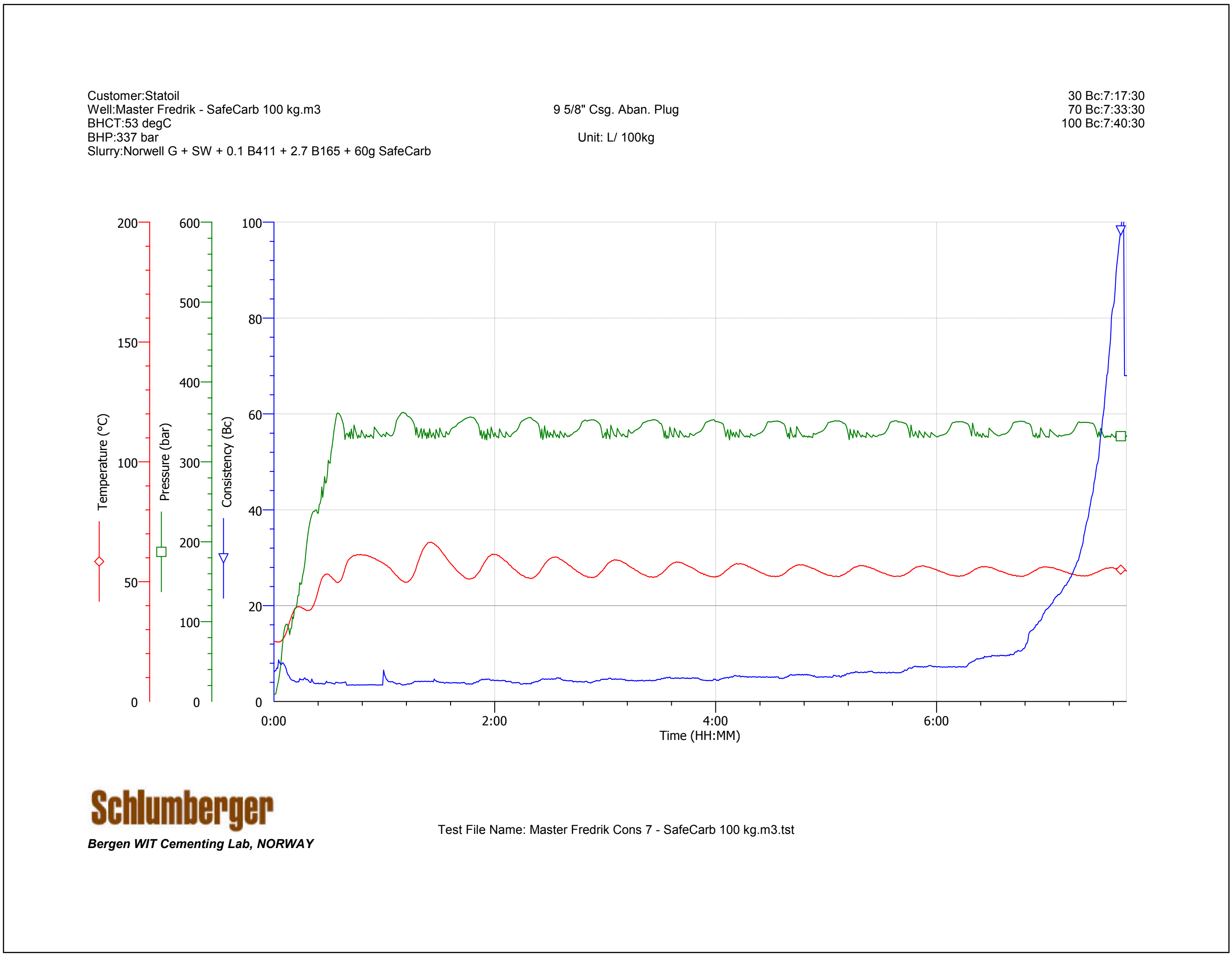
Unit: L/ 100kg

30 Bc:8:29:30
70 Bc:8:50:00
100 Bc:9:01:30

Schlumberger
Bergen WIT Cementing Lab, NORWAY

Test File Name: Master Fredrik - Base Slurry.tst

**Figure B.1:** TT base slurry



Customer:Statoil
Well:Master Fredrik - SafeCarb 100 kg.m3
BHCT:53 degC
BHP:337 bar
Slurry:Norwell G + SW + 0.1 B411 + 2.7 B165 + 60g SafeCarb

9 5/8" Csg. Aban. Plug

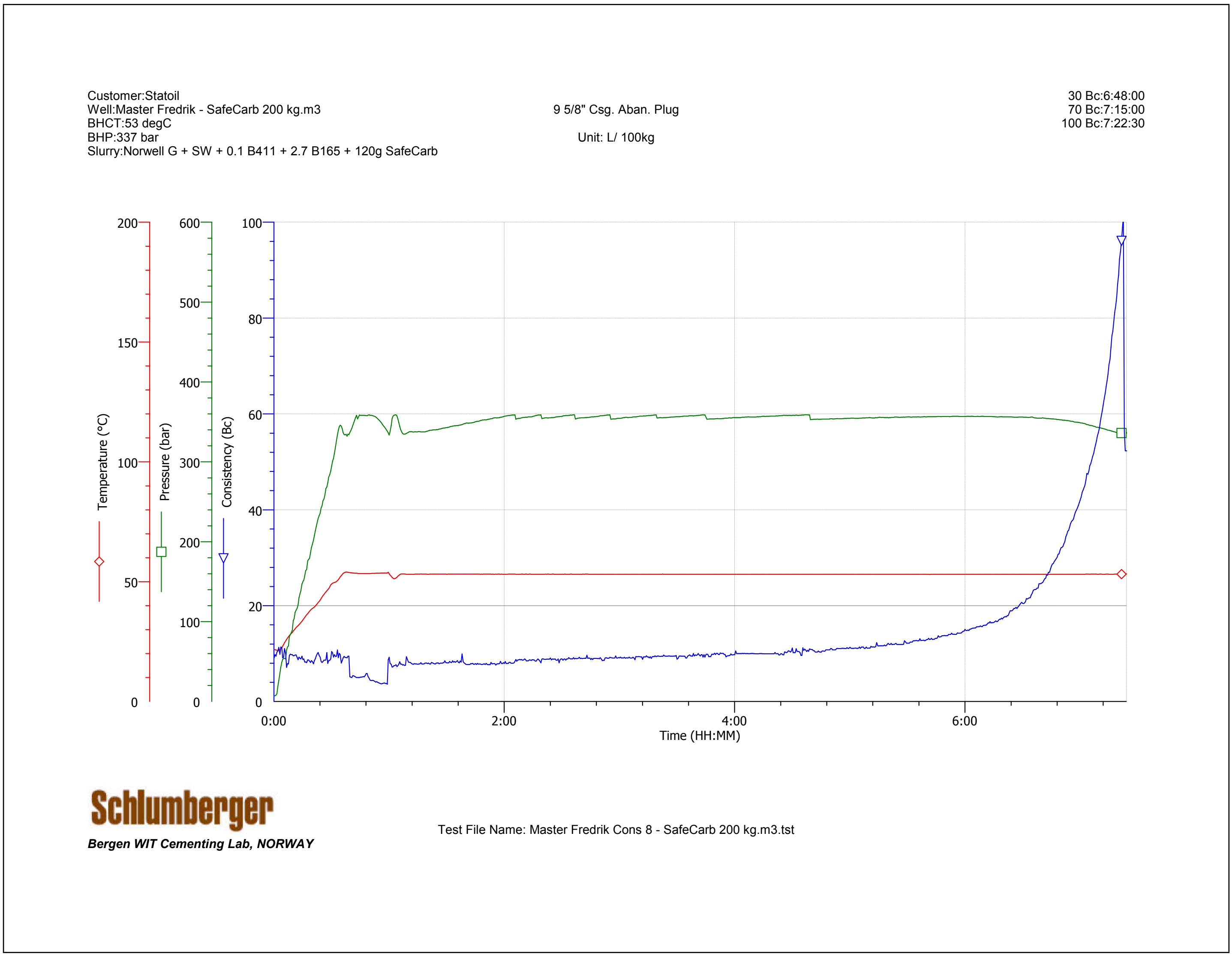
Unit: L/ 100kg

30 Bc:7:17:30
70 Bc:7:33:30
100 Bc:7:40:30

Schlumberger
*Bergen WIT Cementing Lab, NORWAY*

Test File Name: Master Fredrik Cons 7 - SafeCarb 100 kg.m3.tst

**Figure B.2:** TT SafeCarb 100 kg/$m^3$



Customer:Statoil
Well:Master Fredrik - SafeCarb 200 kg.m3
BHCT:53 degC
BHP:337 bar
Slurry:Norwell G + SW + 0.1 B411 + 2.7 B165 + 120g SafeCarb

9 5/8" Csg. Aban. Plug

Unit: L/ 100kg

30 Bc:6:48:00
70 Bc:7:15:00
100 Bc:7:22:30

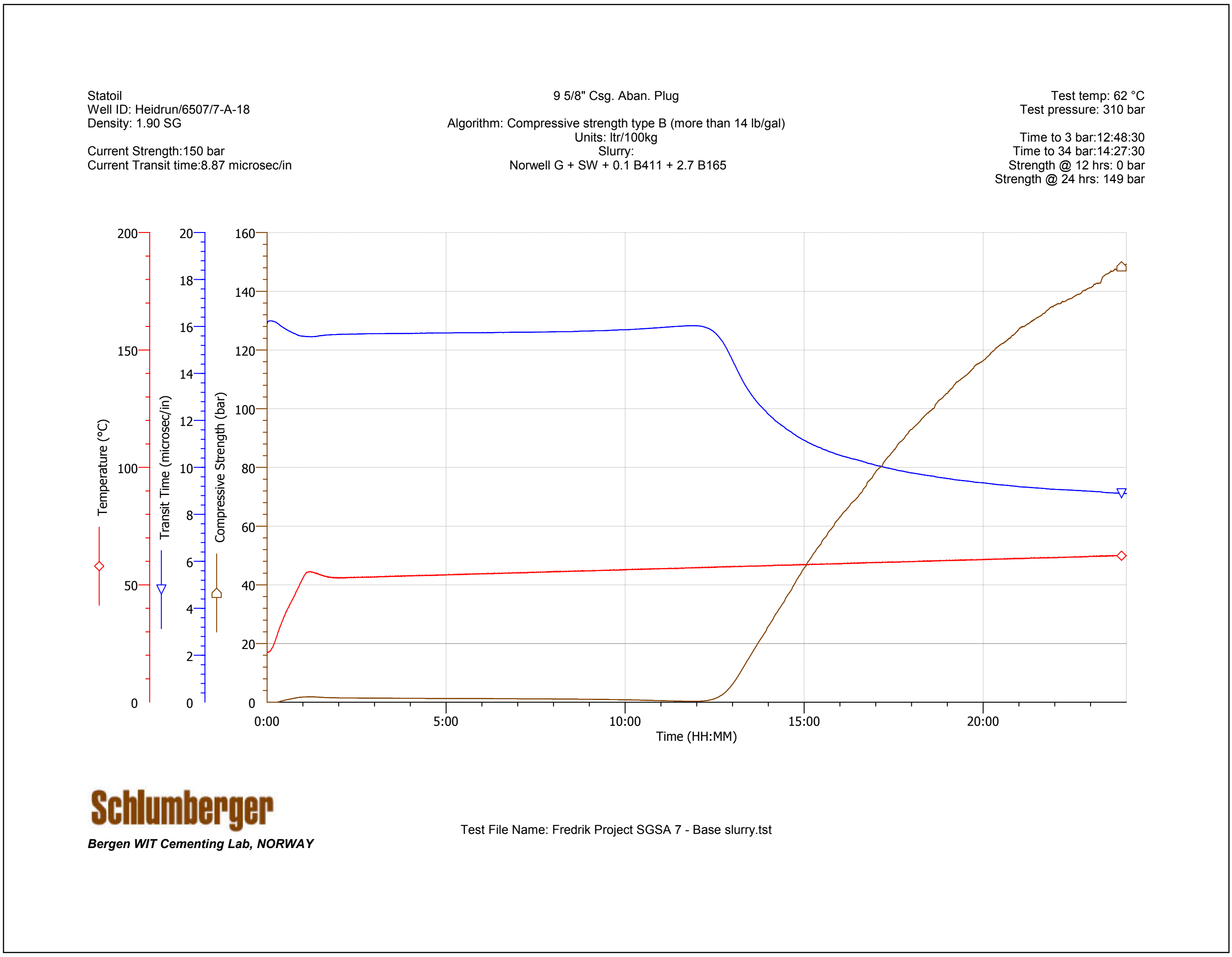
Bergen WIT Cementing Lab, NORWAY

Test File Name: Master Fredrik Cons 8 - SafeCarb 200 kg.m3.tst

Figure B.3: TT SafeCarb 200 kg/m$^3$

# C Appendix C - SGSA Results

This appendix contains all the results from the compressive strength tests.



Statoil
Well ID: Heidrun/6507/7-A-18
Density: 1.90 SG

Current Strength:150 bar
Current Transit time:8.87 microsec/in

9 5/8" Csg. Aban. Plug

Algorithm: Compressive strength type B (more than 14 lb/gal)
Units: ltr/100kg
Slurry:
Norwell G + SW + 0.1 B411 + 2.7 B165

Test temp: 62 °C
Test pressure: 310 bar

Time to 3 bar:12:48:30
Time to 34 bar:14:27:30
Strength @ 12 hrs: 0 bar
Strength @ 24 hrs: 149 bar

Schlumberger
Bergen WIT Cementing Lab, NORWAY

Test File Name: Fredrik Project SGSA 7 - Base slurry.tst

**Figure C.1:** SGSA base slurry

Statoil
Well ID: Heidrun/6507/7-A-18
Density: 1.90 SG

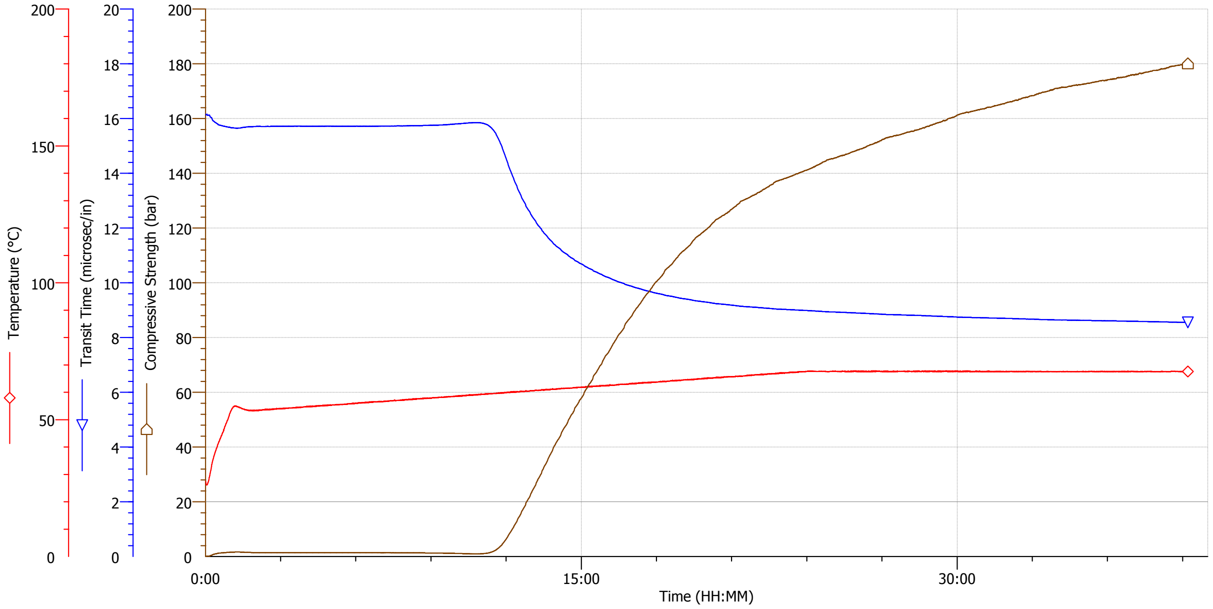
Current Strength:180 bar
Current Transit time:8.55 microsec/in

9 5/8" Csg. Aban. Plug

Algorithm: Compressive strength type B (more than 14 lb/gal)
Units: ltr/100kg
Slurry:
Norwell G + SW + 0.1 B411 + 2.7 B165

Test temp: 68 °C
Test pressure: 310 bar

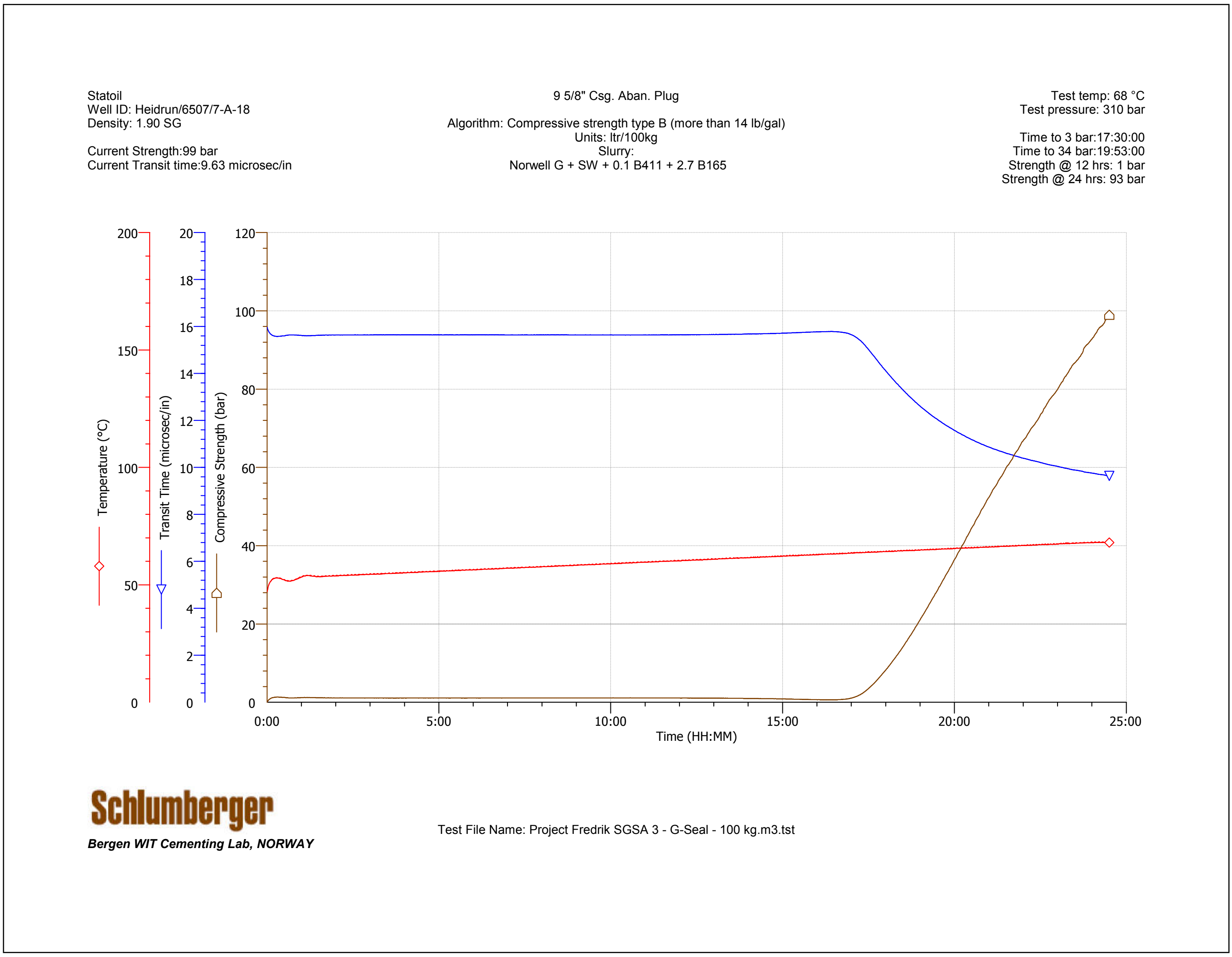
Time to 3 bar:11:44:30
Time to 34 bar:13:37:30
Strength @ 12 hrs: 6 bar
Strength @ 24 hrs: 141 bar



*Bergen WIT Cementing Lab, NORWAY*

Test File Name: Fredrik Project SGSA 2- Base slurry.tst

**Figure C.2:** SGSA base slurry retest



Statoil
Well ID: Heidrun/6507/7-A-18
Density: 1.90 SG

Current Strength:99 bar
Current Transit time:9.63 microsec/in

9 5/8" Csg. Aban. Plug

Algorithm: Compressive strength type B (more than 14 lb/gal)
Units: ltr/100kg
Slurry:
Norwell G + SW + 0.1 B411 + 2.7 B165

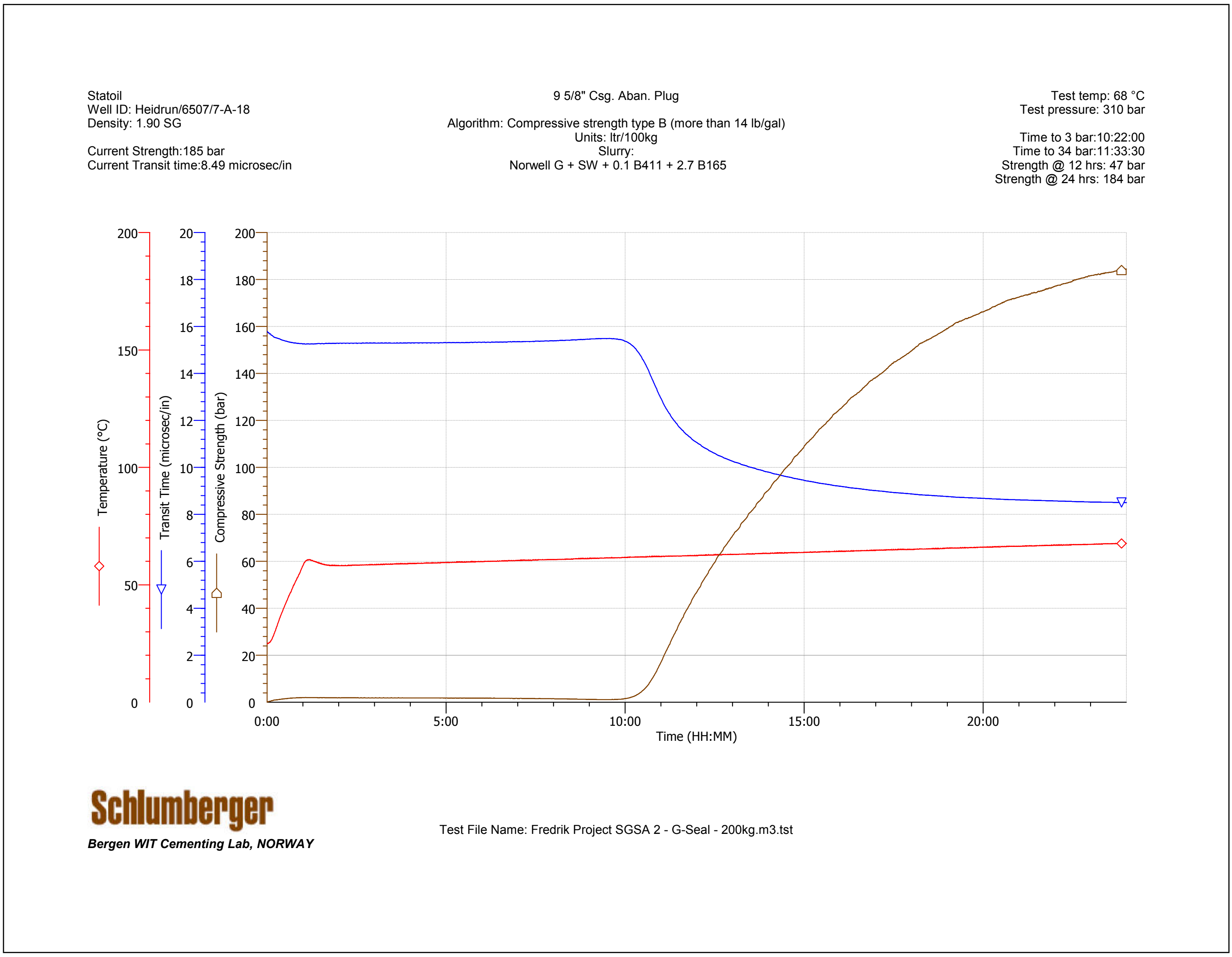
Test temp: 68 °C
Test pressure: 310 bar

Time to 3 bar:17:30:00
Time to 34 bar:19:53:00
Strength @ 12 hrs: 1 bar
Strength @ 24 hrs: 93 bar

Schlumberger
*Bergen WIT Cementing Lab, NORWAY*

Test File Name: Project Fredrik SGSA 3 - G-Seal - 100 kg.m3.tst

**Figure C.3:** SGSA G-Seal 100 kg/m$^3$



Statoil
Well ID: Heidrun/6507/7-A-18
Density: 1.90 SG

Current Strength:185 bar
Current Transit time:8.49 microsec/in

9 5/8" Csg. Aban. Plug

Algorithm: Compressive strength type B (more than 14 lb/gal)
Units: ltr/100kg
Slurry:
Norwell G + SW + 0.1 B411 + 2.7 B165

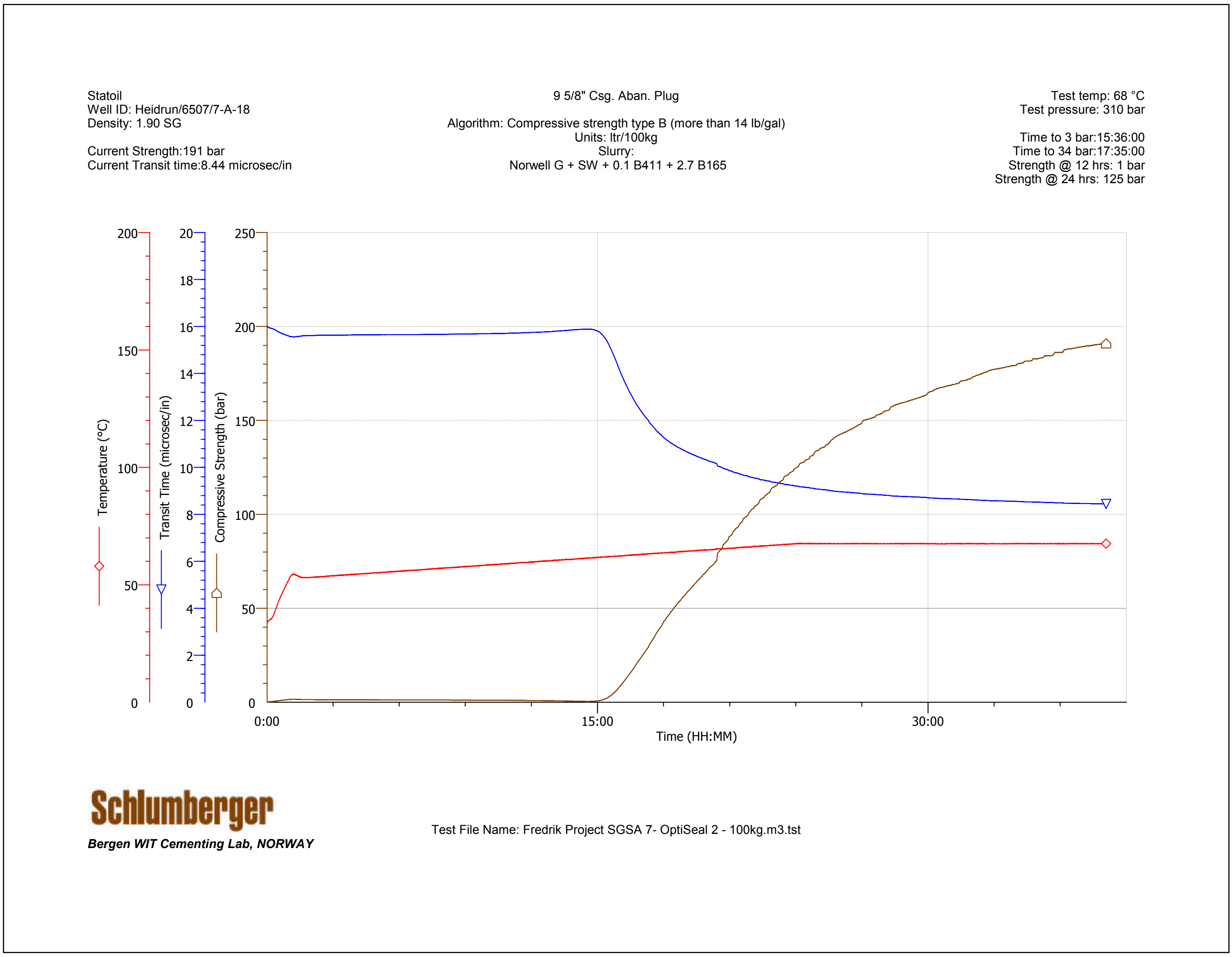
Test temp: 68 °C
Test pressure: 310 bar

Time to 3 bar:10:22:00
Time to 34 bar:11:33:30
Strength @ 12 hrs: 47 bar
Strength @ 24 hrs: 184 bar

Bergen WIT Cementing Lab, NORWAY

Test File Name: Fredrik Project SGSA 2 - G-Seal - 200kg.m3.tst

**Figure C.4:** SGSA G-Seal 200 kg/m$^3$



Statoil
Well ID: Heidrun/6507/7-A-18
Density: 1.90 SG

Current Strength:191 bar
Current Transit time:8.44 microsec/in

9 5/8" Csg. Aban. Plug

Algorithm: Compressive strength type B (more than 14 lb/gal)
Units: ltr/100kg
Slurry:
Norwell G + SW + 0.1 B411 + 2.7 B165

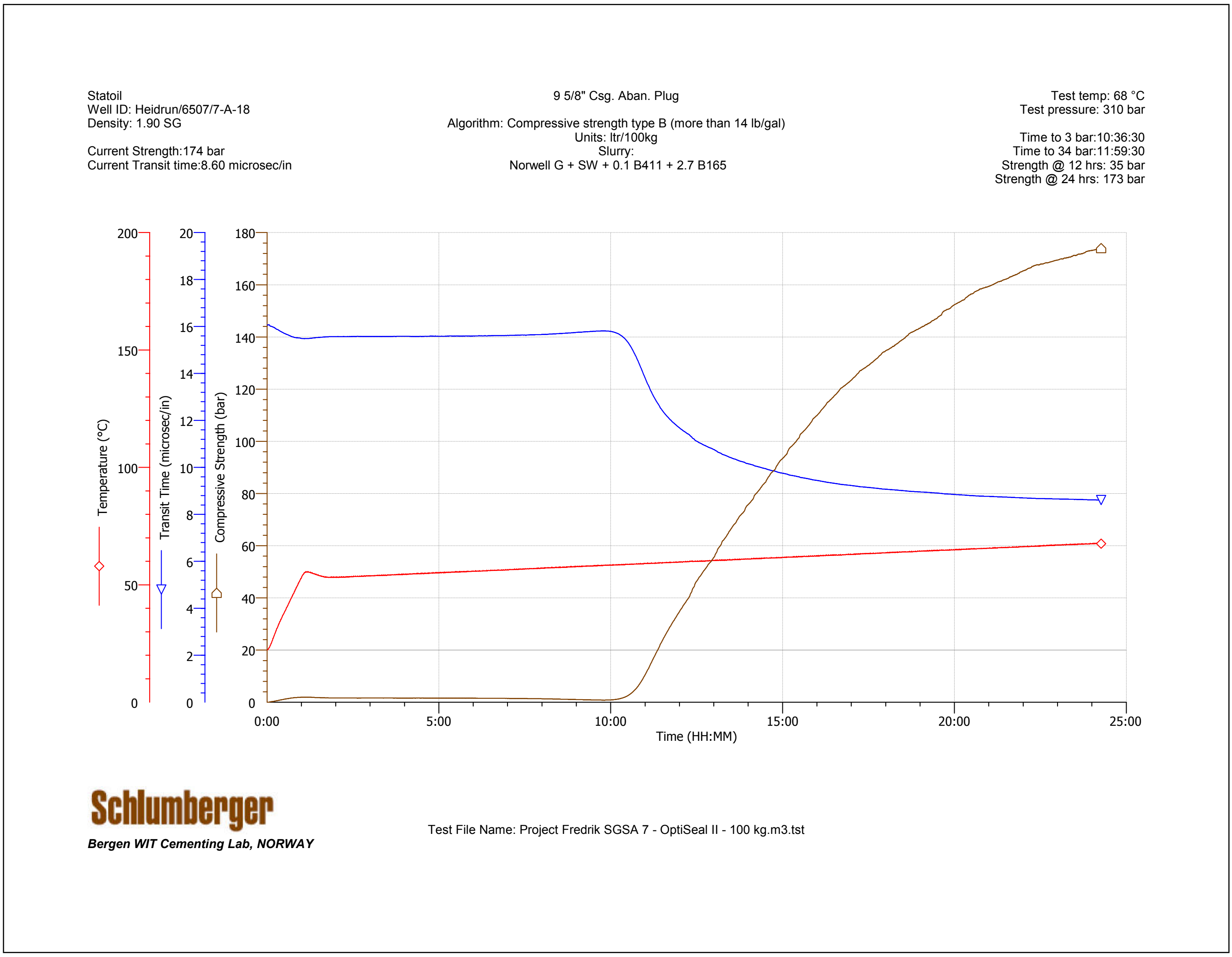
Test temp: 68 °C
Test pressure: 310 bar

Time to 3 bar:15:36:00
Time to 34 bar:17:35:00
Strength @ 12 hrs: 1 bar
Strength @ 24 hrs: 125 bar

Schlumberger
*Bergen WIT Cementing Lab, NORWAY*

Test File Name: Fredrik Project SGSA 7- OptiSeal 2 - 100kg.m3.tst

**Figure C.5:** SGSA OptiSeal II 100 kg/m$^3$



Statoil
Well ID: Heidrun/6507/7-A-18
Density: 1.90 SG

Current Strength:174 bar
Current Transit time:8.60 microsec/in

9 5/8" Csg. Aban. Plug

Algorithm: Compressive strength type B (more than 14 lb/gal)
Units: ltr/100kg
Slurry:
Norwell G + SW + 0.1 B411 + 2.7 B165

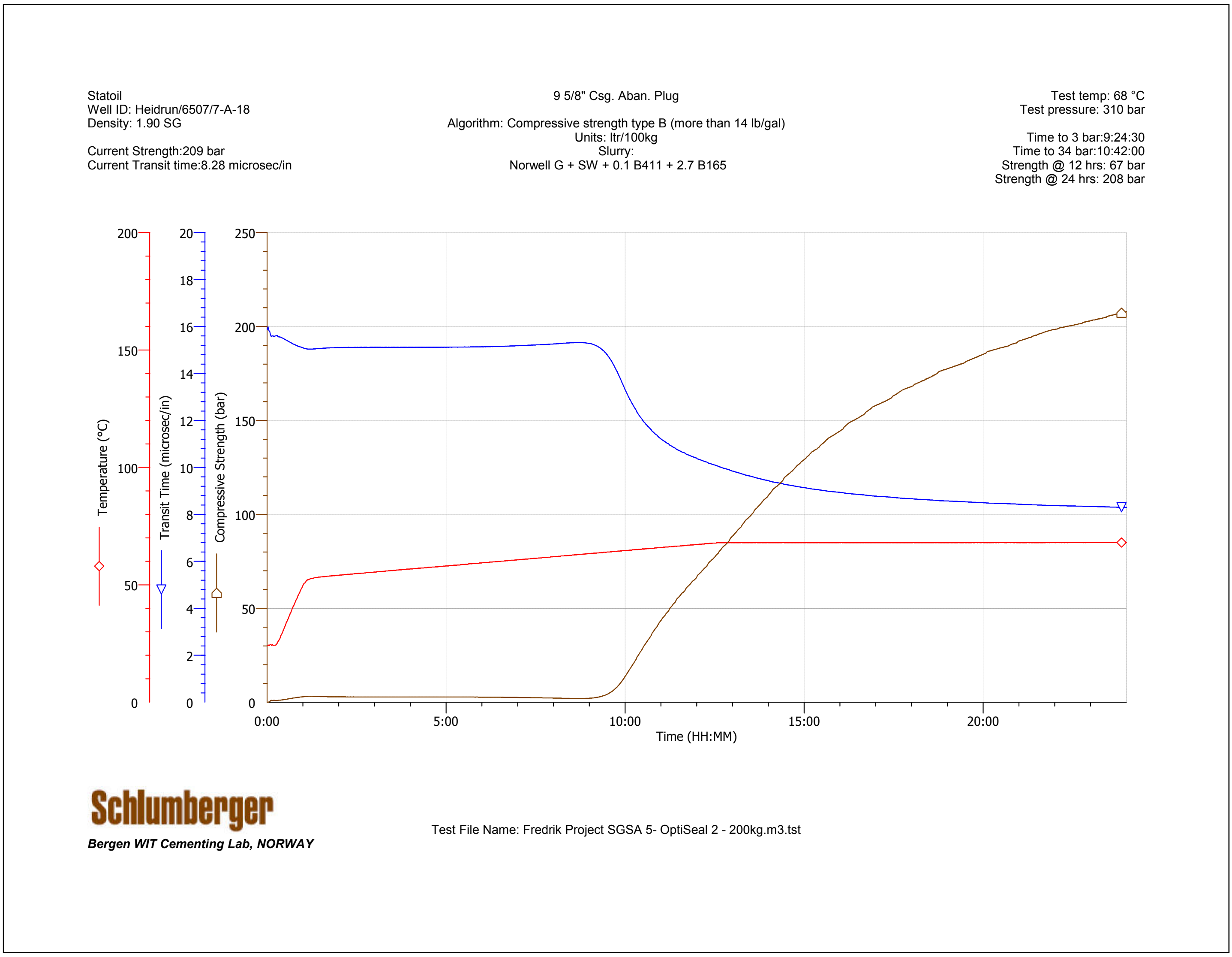
Test temp: 68 °C
Test pressure: 310 bar

Time to 3 bar:10:36:30
Time to 34 bar:11:59:30
Strength @ 12 hrs: 35 bar
Strength @ 24 hrs: 173 bar

*Bergen WIT Cementing Lab, NORWAY*

Test File Name: Project Fredrik SGSA 7 - OptiSeal II - 100 kg.m3.tst

**Figure C.6:** SGSA OptiSeal II 100 kg/m$^3$ retest



Statoil
Well ID: Heidrun/6507/7-A-18
Density: 1.90 SG

Current Strength:209 bar
Current Transit time:8.28 microsec/in

9 5/8" Csg. Aban. Plug

Algorithm: Compressive strength type B (more than 14 lb/gal)
Units: ltr/100kg
Slurry:
Norwell G + SW + 0.1 B411 + 2.7 B165

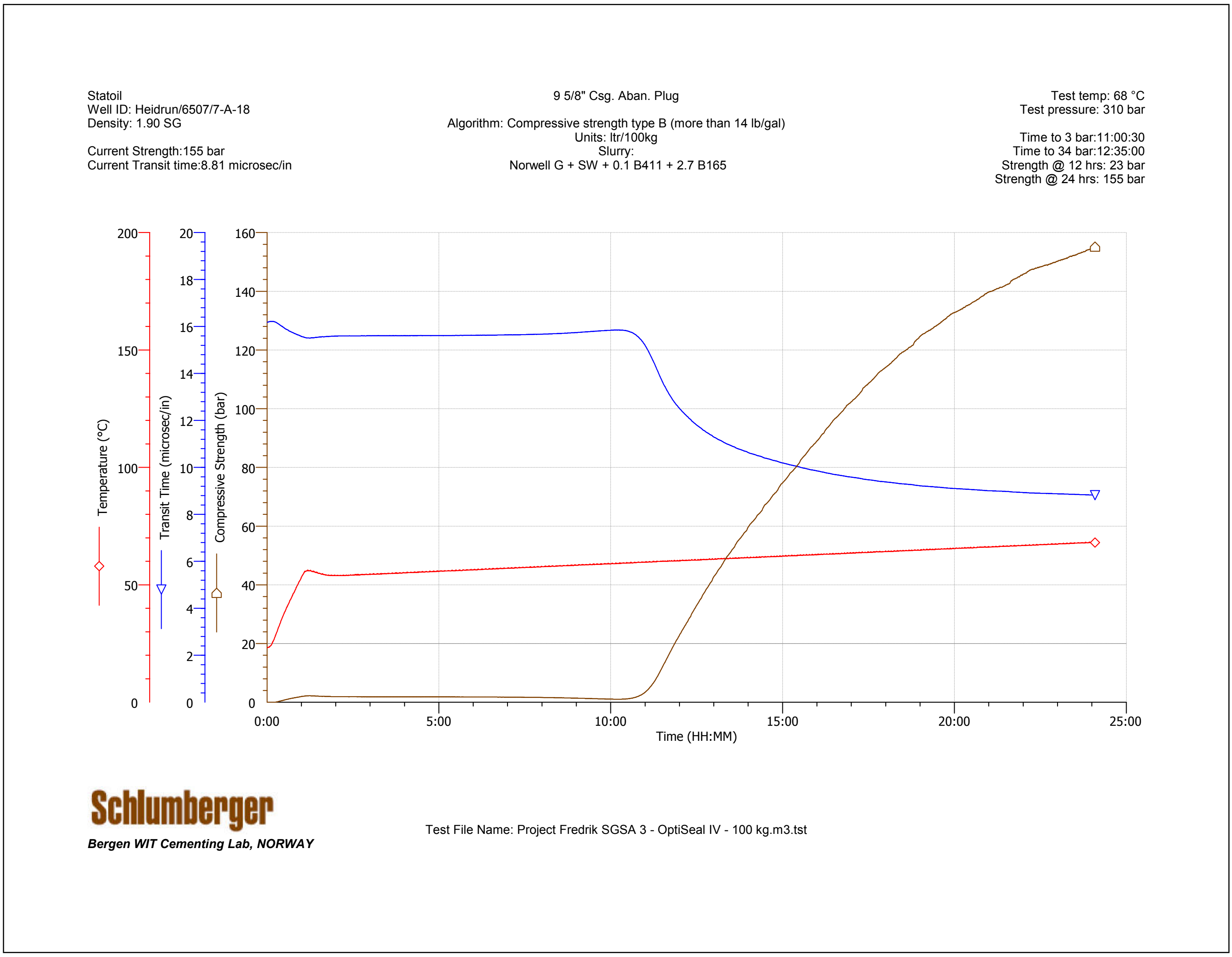
Test temp: 68 °C
Test pressure: 310 bar

Time to 3 bar:9:24:30
Time to 34 bar:10:42:00
Strength @ 12 hrs: 67 bar
Strength @ 24 hrs: 208 bar

Schlumberger
Bergen WIT Cementing Lab, NORWAY

Test File Name: Fredrik Project SGSA 5- OptiSeal 2 - 200kg.m3.tst

**Figure C.7:** SGSA OptiSeal II 200 kg/m$^3$



Statoil
Well ID: Heidrun/6507/7-A-18
Density: 1.90 SG

Current Strength:155 bar
Current Transit time:8.81 microsec/in

9 5/8" Csg. Aban. Plug

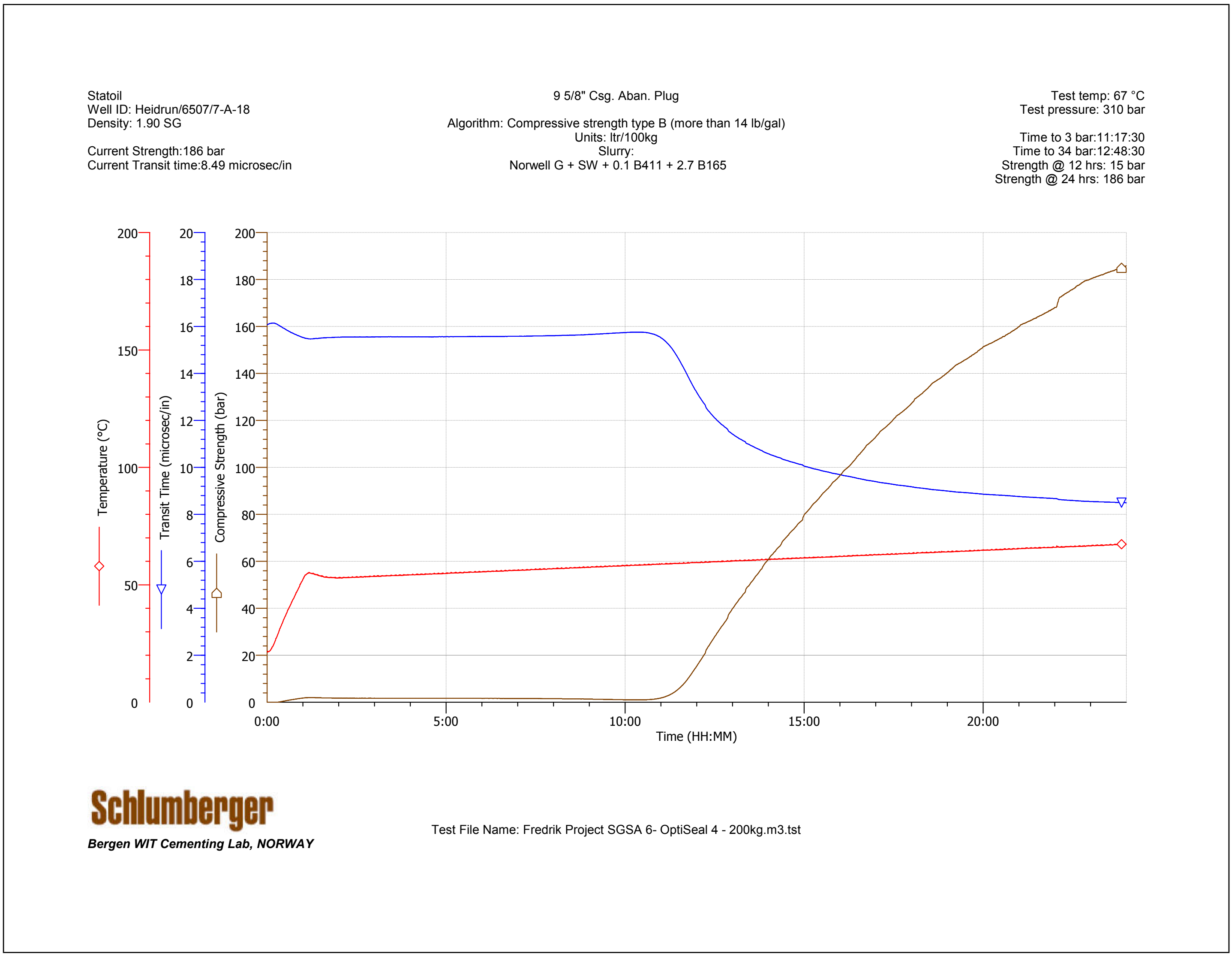
Algorithm: Compressive strength type B (more than 14 lb/gal)
Units: ltr/100kg
Slurry:
Norwell G + SW + 0.1 B411 + 2.7 B165

Test temp: 68 °C
Test pressure: 310 bar

Time to 3 bar:11:00:30
Time to 34 bar:12:35:00
Strength @ 12 hrs: 23 bar
Strength @ 24 hrs: 155 bar

Test File Name: Project Fredrik SGSA 3 - OptiSeal IV - 100 kg.m3.tst

**Figure C.8:** SGSA OptiSeal IV 100 kg/m$^3$



Statoil
Well ID: Heidrun/6507/7-A-18
Density: 1.90 SG

Current Strength:186 bar
Current Transit time:8.49 microsec/in

9 5/8" Csg. Aban. Plug

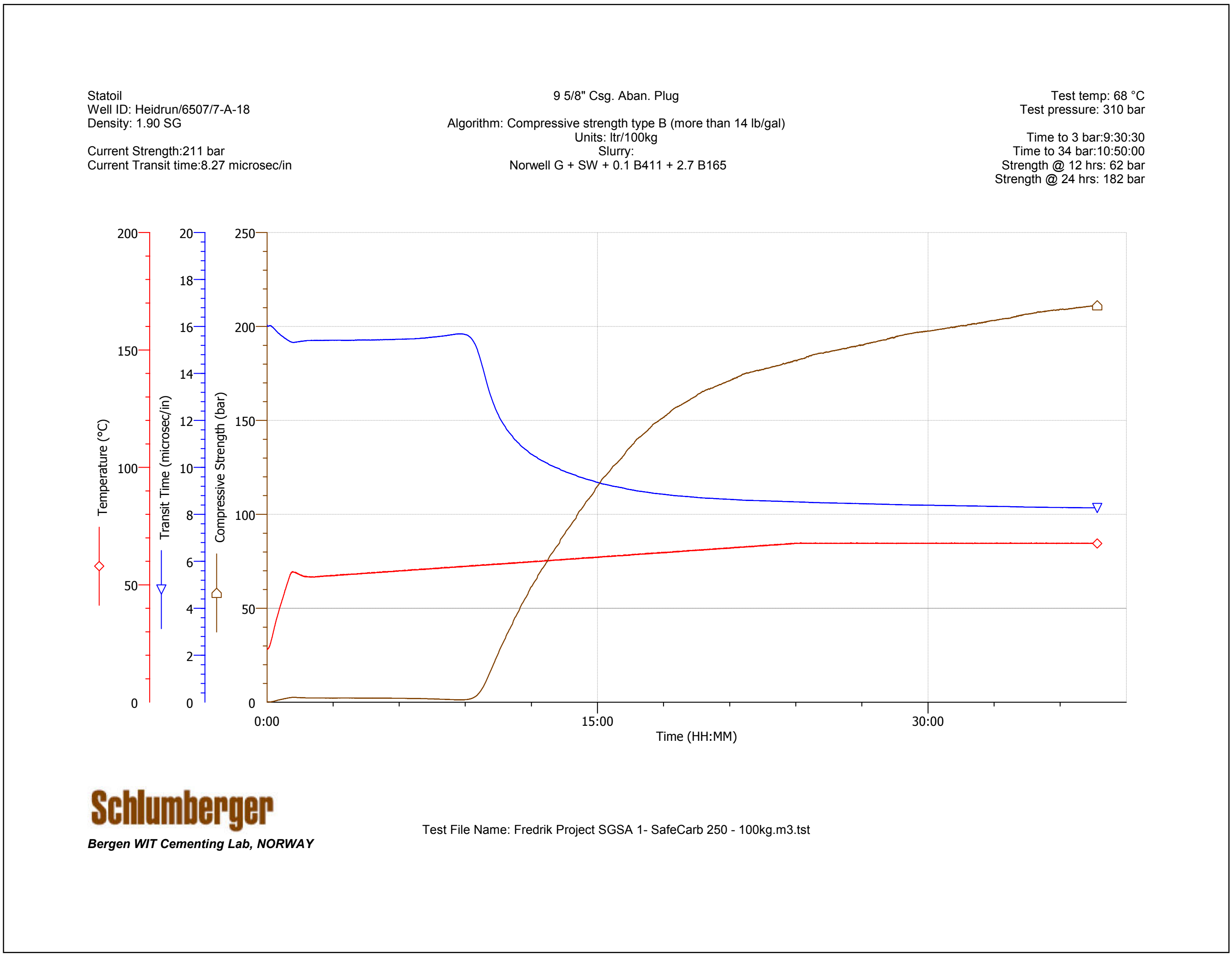
Algorithm: Compressive strength type B (more than 14 lb/gal)
Units: ltr/100kg
Slurry:
Norwell G + SW + 0.1 B411 + 2.7 B165

Test temp: 67 °C
Test pressure: 310 bar

Time to 3 bar:11:17:30
Time to 34 bar:12:48:30
Strength @ 12 hrs: 15 bar
Strength @ 24 hrs: 186 bar

Test File Name: Fredrik Project SGSA 6- OptiSeal 4 - 200kg.m3.tst

**Figure C.9:** SGSA OptiSeal IV 200 kg/m$^3$



Statoil
Well ID: Heidrun/6507/7-A-18
Density: 1.90 SG

Current Strength:211 bar
Current Transit time:8.27 microsec/in

9 5/8" Csg. Aban. Plug

Algorithm: Compressive strength type B (more than 14 lb/gal)
Units: ltr/100kg
Slurry:
Norwell G + SW + 0.1 B411 + 2.7 B165

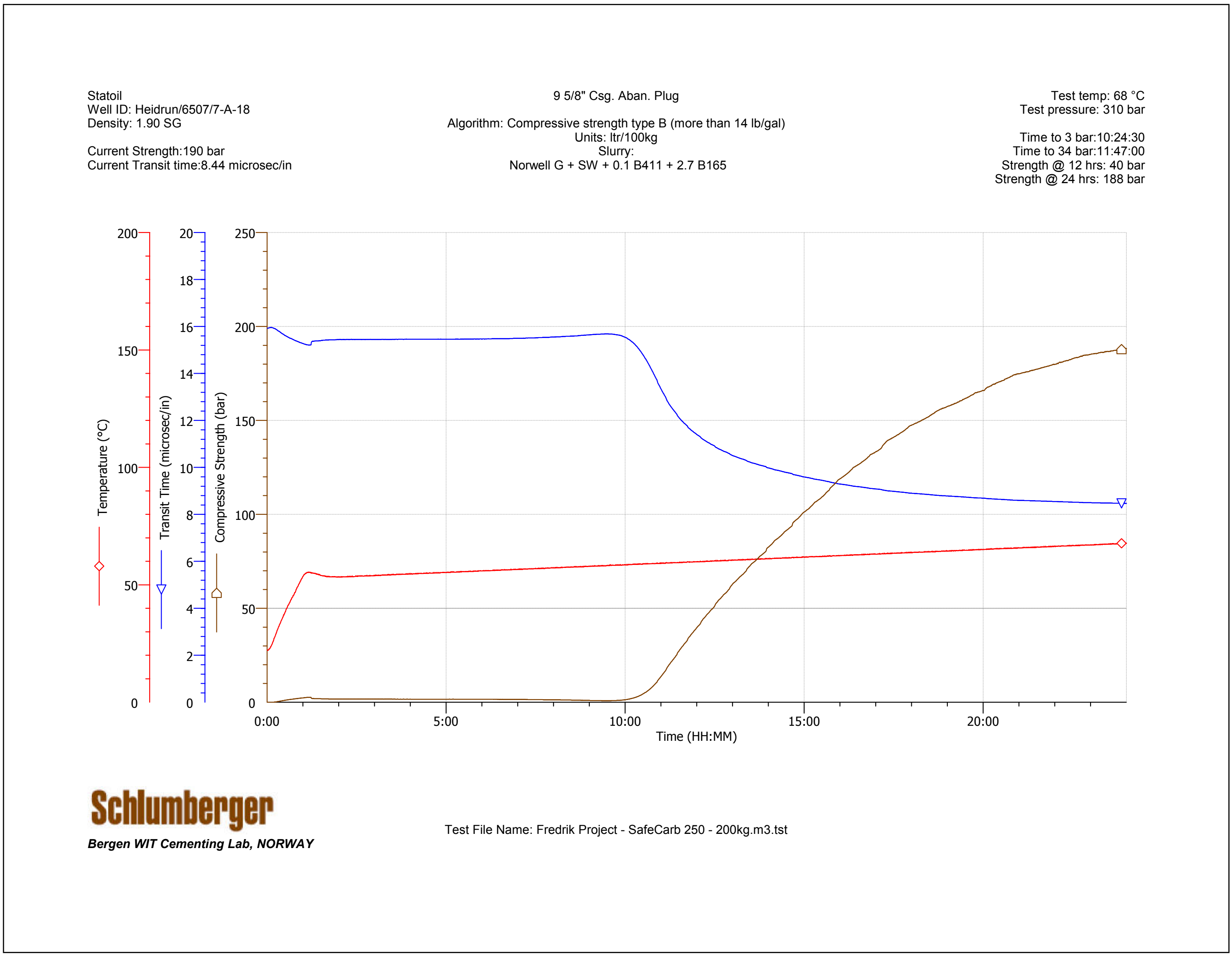
Test temp: 68 °C
Test pressure: 310 bar

Time to 3 bar:9:30:30
Time to 34 bar:10:50:00
Strength @ 12 hrs: 62 bar
Strength @ 24 hrs: 182 bar

Bergen WIT Cementing Lab, NORWAY

Test File Name: Fredrik Project SGSA 1- SafeCarb 250 - 100kg.m3.tst

**Figure C.10:** SGSA SafeCarb 250 100 kg/$m^3$



Statoil
Well ID: Heidrun/6507/7-A-18
Density: 1.90 SG

Current Strength:190 bar
Current Transit time:8.44 microsec/in

9 5/8" Csg. Aban. Plug

Algorithm: Compressive strength type B (more than 14 lb/gal)
Units: ltr/100kg
Slurry:
Norwell G + SW + 0.1 B411 + 2.7 B165

Test temp: 68 °C
Test pressure: 310 bar

Time to 3 bar:10:24:30
Time to 34 bar:11:47:00
Strength @ 12 hrs: 40 bar
Strength @ 24 hrs: 188 bar

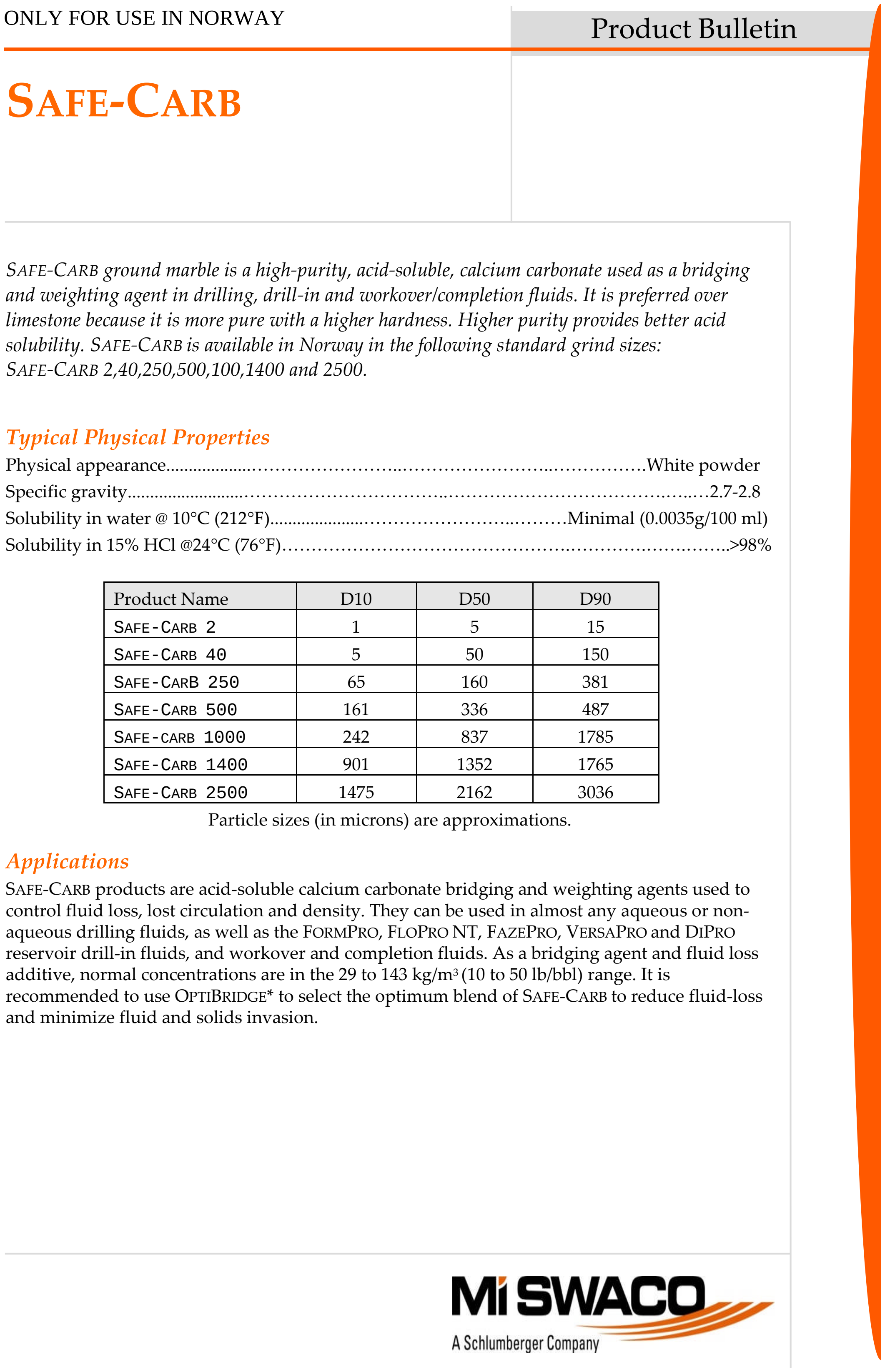
Bergen WIT Cementing Lab, NORWAY

Test File Name: Fredrik Project - SafeCarb 250 - 200kg.m3.tst

**Figure C.11:** SGSA SafeCarb 250 200 kg/m$^3$

# D Appendix D - LCM Data Sheets

In this appendix the product bulletin for the LCMs used in the thesis may be found.



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Product Bulletin

# SAFE-CARB

*SAFE-CARB ground marble is a high-purity, acid-soluble, calcium carbonate used as a bridging and weighting agent in drilling, drill-in and workover/completion fluids. It is preferred over limestone because it is more pure with a higher hardness. Higher purity provides better acid solubility. SAFE-CARB is available in Norway in the following standard grind sizes: SAFE-CARB 2,40,250,500,100,1400 and 2500.*

## *Typical Physical Properties*

Physical appearance.....................................................................................White powder
Specific gravity......................................................................................................2.7-2.8
Solubility in water @ 10°C (212°F)..............................................Minimal (0.0035g/100 ml)
Solubility in 15% HCl @24°C (76°F)..........................................................................>98%

| Product Name | D10 | D50 | D90 |
|---|---|---|---|
| SAFE-CARB 2 | 1 | 5 | 15 |
| SAFE-CARB 40 | 5 | 50 | 150 |
| SAFE-CARB 250 | 65 | 160 | 381 |
| SAFE-CARB 500 | 161 | 336 | 487 |
| SAFE-CARB 1000 | 242 | 837 | 1785 |
| SAFE-CARB 1400 | 901 | 1352 | 1765 |
| SAFE-CARB 2500 | 1475 | 2162 | 3036 |

Particle sizes (in microns) are approximations.

## *Applications*

SAFE-CARB products are acid-soluble calcium carbonate bridging and weighting agents used to control fluid loss, lost circulation and density. They can be used in almost any aqueous or non-aqueous drilling fluids, as well as the FORMPRO, FLOPRO NT, FAZEPRO, VERSAPRO and DIPRO reservoir drill-in fluids, and workover and completion fluids. As a bridging agent and fluid loss additive, normal concentrations are in the 29 to 143 kg/$m^3$ (10 to 50 lb/bbl) range. It is recommended to use OPTIBRIDGE* to select the optimum blend of SAFE-CARB to reduce fluid-loss and minimize fluid and solids invasion.

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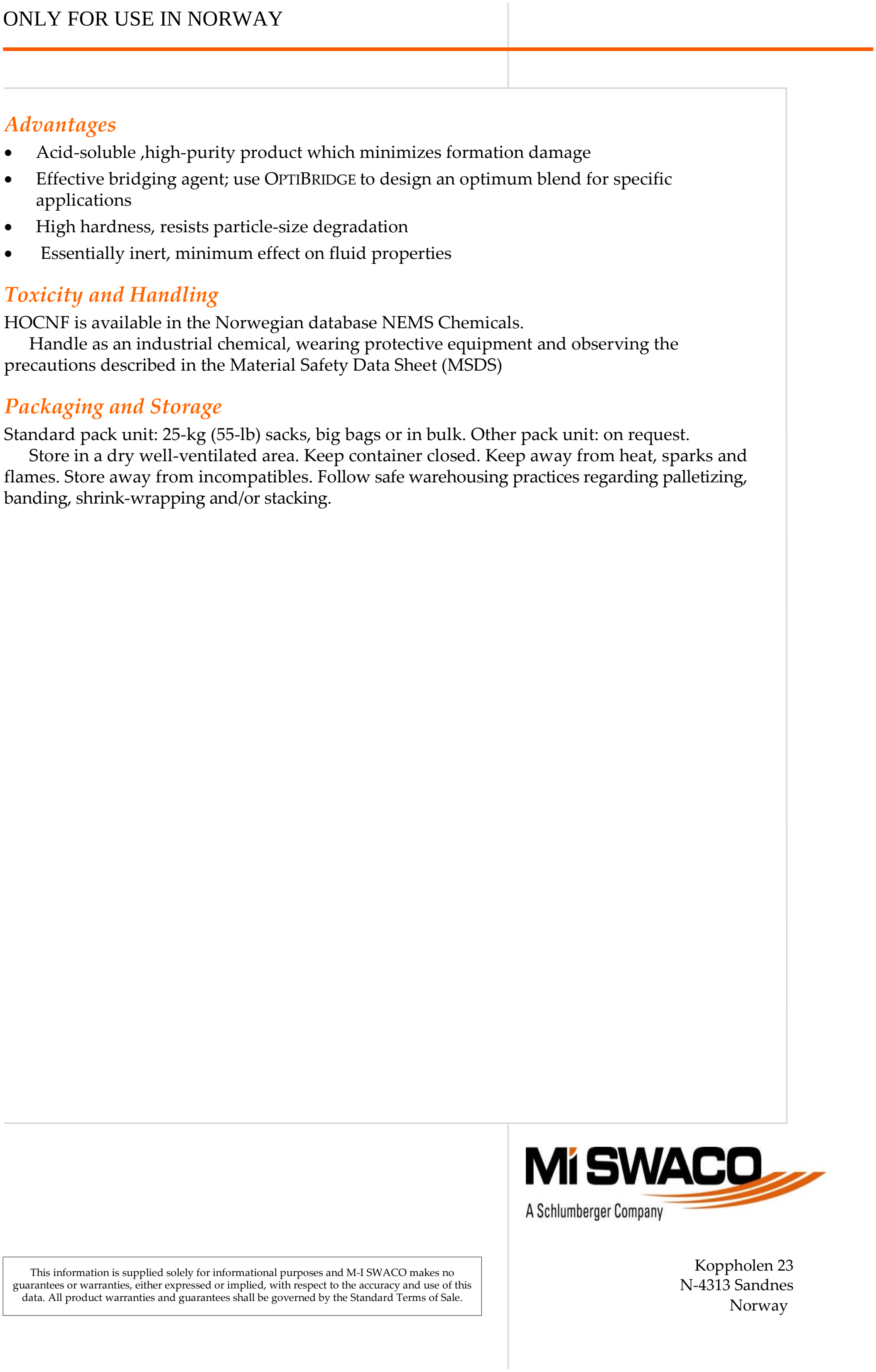
**Figure D.1:** SafeCarb 250



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## Advantages

- Acid-soluble ,high-purity product which minimizes formation damage
- Effective bridging agent; use OPTIBRIDGE to design an optimum blend for specific applications
- High hardness, resists particle-size degradation
- Essentially inert, minimum effect on fluid properties

## Toxicity and Handling

HOCNF is available in the Norwegian database NEMS Chemicals.

Handle as an industrial chemical, wearing protective equipment and observing the precautions described in the Material Safety Data Sheet (MSDS)

## Packaging and Storage

Standard pack unit: 25-kg (55-lb) sacks, big bags or in bulk. Other pack unit: on request.

Store in a dry well-ventilated area. Keep container closed. Keep away from heat, sparks and flames. Store away from incompatibles. Follow safe warehousing practices regarding palletizing, banding, shrink-wrapping and/or stacking.

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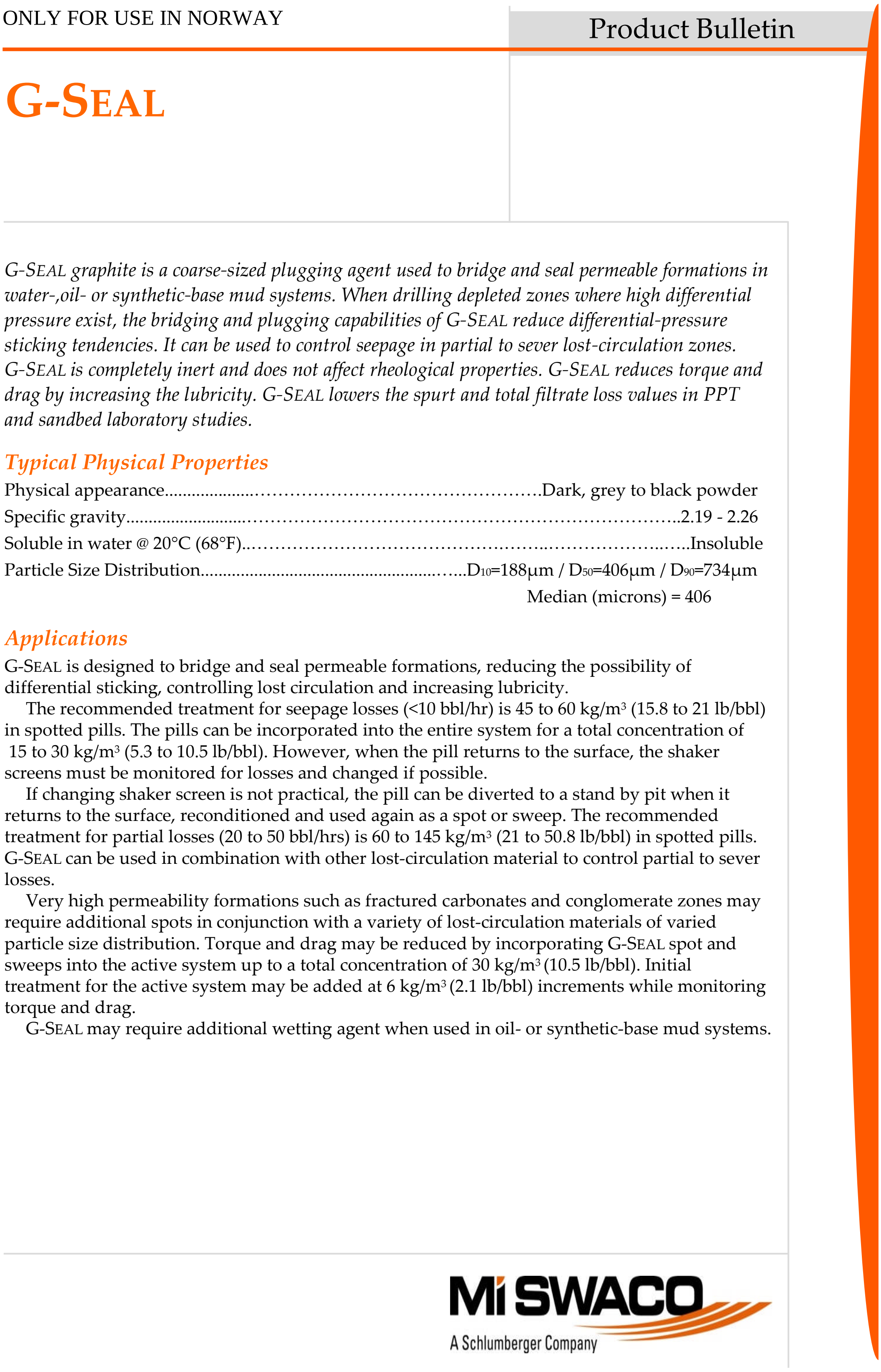
Norway

Figure D.1: SafeCarb 250 (Continued)



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Product Bulletin

# G-SEAL

*G-SEAL graphite is a coarse-sized plugging agent used to bridge and seal permeable formations in water-,oil- or synthetic-base mud systems. When drilling depleted zones where high differential pressure exist, the bridging and plugging capabilities of G-SEAL reduce differential-pressure sticking tendencies. It can be used to control seepage in partial to sever lost-circulation zones. G-SEAL is completely inert and does not affect rheological properties. G-SEAL reduces torque and drag by increasing the lubricity. G-SEAL lowers the spurt and total filtrate loss values in PPT and sandbed laboratory studies.*

## *Typical Physical Properties*

| | |
|---|---|
| Physical appearance | Dark, grey to black powder |
| Specific gravity | 2.19 - 2.26 |
| Soluble in water @ 20°C (68°F) | Insoluble |
| Particle Size Distribution | $D_{10}$=188µm / $D_{50}$=406µm / $D_{90}$=734µm<br>Median (microns) = 406 |

## *Applications*

G-SEAL is designed to bridge and seal permeable formations, reducing the possibility of differential sticking, controlling lost circulation and increasing lubricity.

The recommended treatment for seepage losses (<10 bbl/hr) is 45 to 60 kg/$m^3$ (15.8 to 21 lb/bbl) in spotted pills. The pills can be incorporated into the entire system for a total concentration of 15 to 30 kg/$m^3$ (5.3 to 10.5 lb/bbl). However, when the pill returns to the surface, the shaker screens must be monitored for losses and changed if possible.

If changing shaker screen is not practical, the pill can be diverted to a stand by pit when it returns to the surface, reconditioned and used again as a spot or sweep. The recommended treatment for partial losses (20 to 50 bbl/hrs) is 60 to 145 kg/$m^3$ (21 to 50.8 lb/bbl) in spotted pills. G-SEAL can be used in combination with other lost-circulation material to control partial to sever losses.

Very high permeability formations such as fractured carbonates and conglomerate zones may require additional spots in conjunction with a variety of lost-circulation materials of varied particle size distribution. Torque and drag may be reduced by incorporating G-SEAL spot and sweeps into the active system up to a total concentration of 30 kg/$m^3$ (10.5 lb/bbl). Initial treatment for the active system may be added at 6 kg/$m^3$ (2.1 lb/bbl) increments while monitoring torque and drag.

G-SEAL may require additional wetting agent when used in oil- or synthetic-base mud systems.

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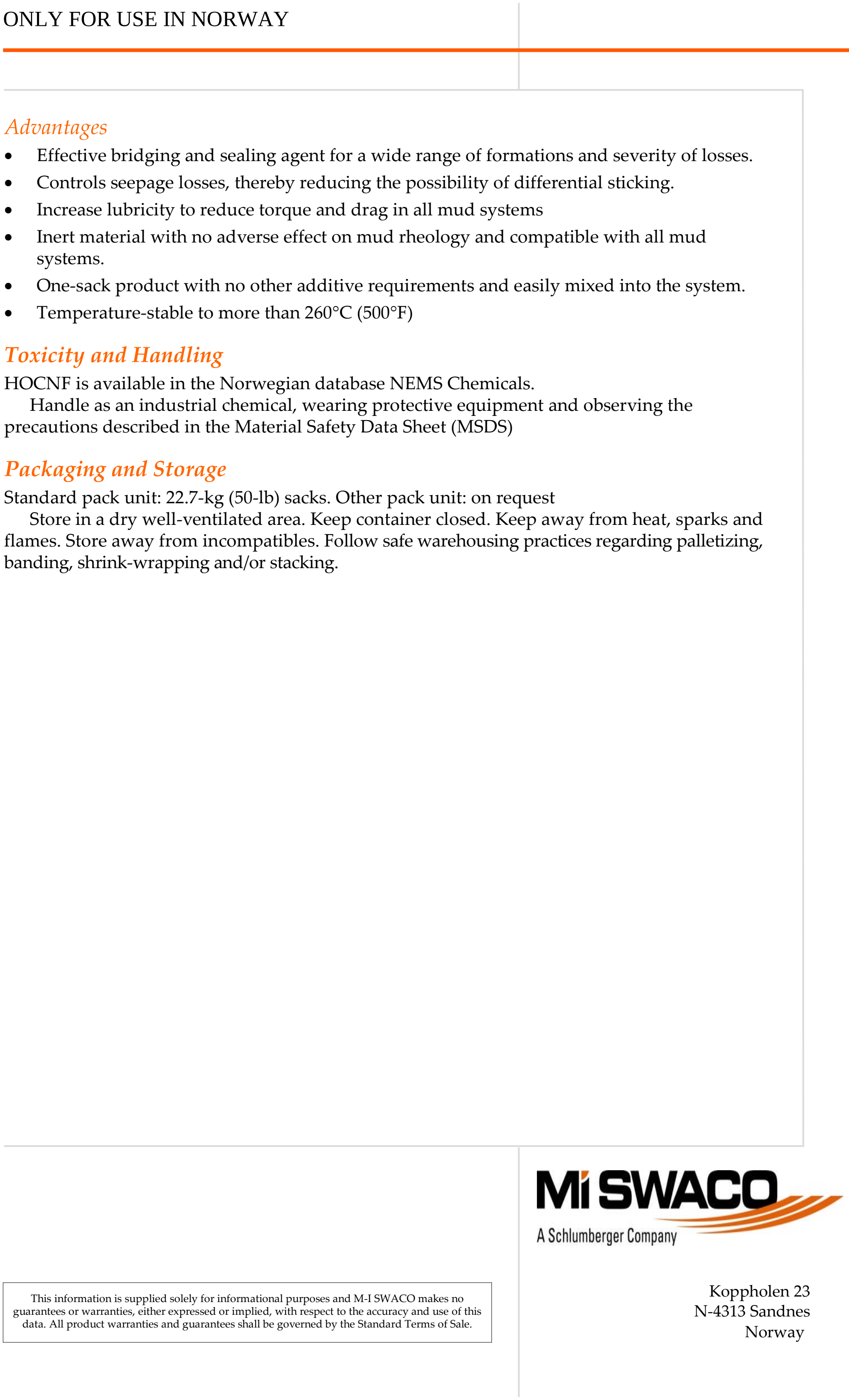
**Figure D.2:** G-Seal



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### *Advantages*

- Effective bridging and sealing agent for a wide range of formations and severity of losses.
- Controls seepage losses, thereby reducing the possibility of differential sticking.
- Increase lubricity to reduce torque and drag in all mud systems
- Inert material with no adverse effect on mud rheology and compatible with all mud systems.
- One-sack product with no other additive requirements and easily mixed into the system.
- Temperature-stable to more than 260°C (500°F)

### *Toxicity and Handling*

HOCNF is available in the Norwegian database NEMS Chemicals.

Handle as an industrial chemical, wearing protective equipment and observing the precautions described in the Material Safety Data Sheet (MSDS)

### *Packaging and Storage*

Standard pack unit: 22.7-kg (50-lb) sacks. Other pack unit: on request

Store in a dry well-ventilated area. Keep container closed. Keep away from heat, sparks and flames. Store away from incompatibles. Follow safe warehousing practices regarding palletizing, banding, shrink-wrapping and/or stacking.

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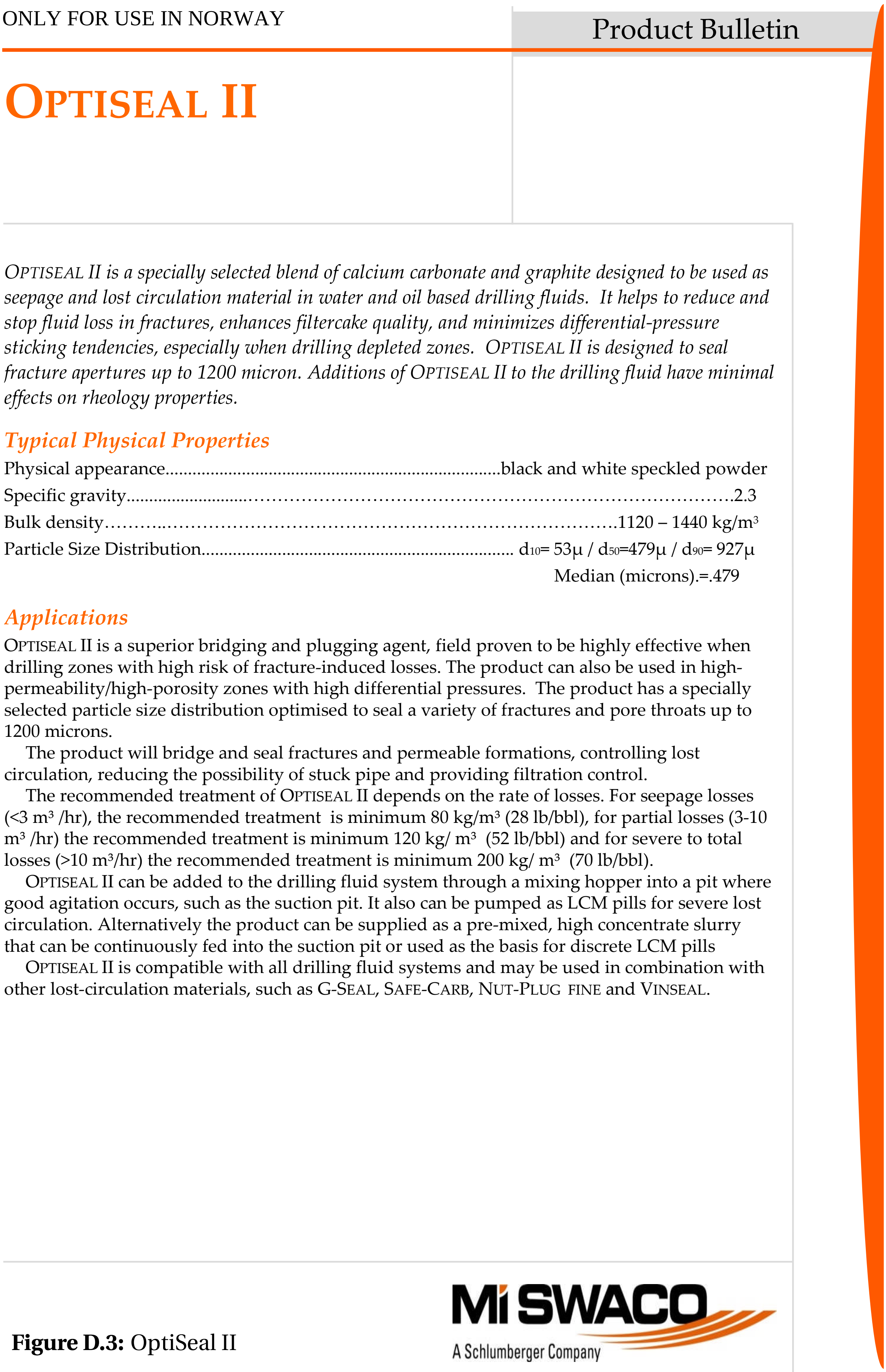
Figure D.2: G-Seal (Continued)



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Product Bulletin

# OPTISEAL II

*OPTISEAL II is a specially selected blend of calcium carbonate and graphite designed to be used as seepage and lost circulation material in water and oil based drilling fluids. It helps to reduce and stop fluid loss in fractures, enhances filtercake quality, and minimizes differential-pressure sticking tendencies, especially when drilling depleted zones. OPTISEAL II is designed to seal fracture apertures up to 1200 micron. Additions of OPTISEAL II to the drilling fluid have minimal effects on rheology properties.*

## *Typical Physical Properties*

| | |
|---|---|
| Physical appearance | black and white speckled powder |
| Specific gravity | 2.3 |
| Bulk density | 1120 – 1440 kg/m$^3$ |
| Particle Size Distribution | $d_{10}$= 53µ / $d_{50}$=479µ / $d_{90}$= 927µ<br>Median (microns).=.479 |

## *Applications*

OPTISEAL II is a superior bridging and plugging agent, field proven to be highly effective when drilling zones with high risk of fracture-induced losses. The product can also be used in high-permeability/high-porosity zones with high differential pressures. The product has a specially selected particle size distribution optimised to seal a variety of fractures and pore throats up to 1200 microns.

The product will bridge and seal fractures and permeable formations, controlling lost circulation, reducing the possibility of stuck pipe and providing filtration control.

The recommended treatment of OPTISEAL II depends on the rate of losses. For seepage losses (<3 m$^3$ /hr), the recommended treatment is minimum 80 kg/m$^3$ (28 lb/bbl), for partial losses (3-10 m$^3$ /hr) the recommended treatment is minimum 120 kg/ m$^3$ (52 lb/bbl) and for severe to total losses (>10 m$^3$/hr) the recommended treatment is minimum 200 kg/ m$^3$ (70 lb/bbl).

OPTISEAL II can be added to the drilling fluid system through a mixing hopper into a pit where good agitation occurs, such as the suction pit. It also can be pumped as LCM pills for severe lost circulation. Alternatively the product can be supplied as a pre-mixed, high concentrate slurry that can be continuously fed into the suction pit or used as the basis for discrete LCM pills

OPTISEAL II is compatible with all drilling fluid systems and may be used in combination with other lost-circulation materials, such as G-SEAL, SAFE-CARB, NUT-PLUG FINE and VINSEAL.

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**Figure D.3:** OptiSeal II

## Advantages

- Effective plugging and sealing of naturally occurring and drilling-induced fractures, with apertures up to 1200 microns.
- Effective bridging and sealing agent for a wide range of formations
- Prevent seepage losses
- Reduces wall cake permeability
- Helps prevent differential sticking
- Easily mixed and dispersed into drilling fluid system
- Can be used in water-, oil-, and synthetic-base drilling fluids systems
- One-sack product with no other additive requirements
- Compatible with other lost-circulation materials
- Has minimum effects on drilling fluids rheology at normal dosages

## Toxicity and Handling

HOCNF is available in the Norwegian database NEMS Chemicals.

Handle as an industrial chemical, wearing protective equipment and observing the precautions described in the Material Safety Data Sheet (MSDS)

## Packaging and Storage

Standard pack unit: 25-kg (55-lb) sacks. Other pack unit: on request.

Store in a dry well-ventilated area. Keep container closed. Keep away from heat, sparks and flames. Store away from incompatibles. Follow safe warehousing practices regarding palletizing, banding, shrink-wrapping and/or stacking.

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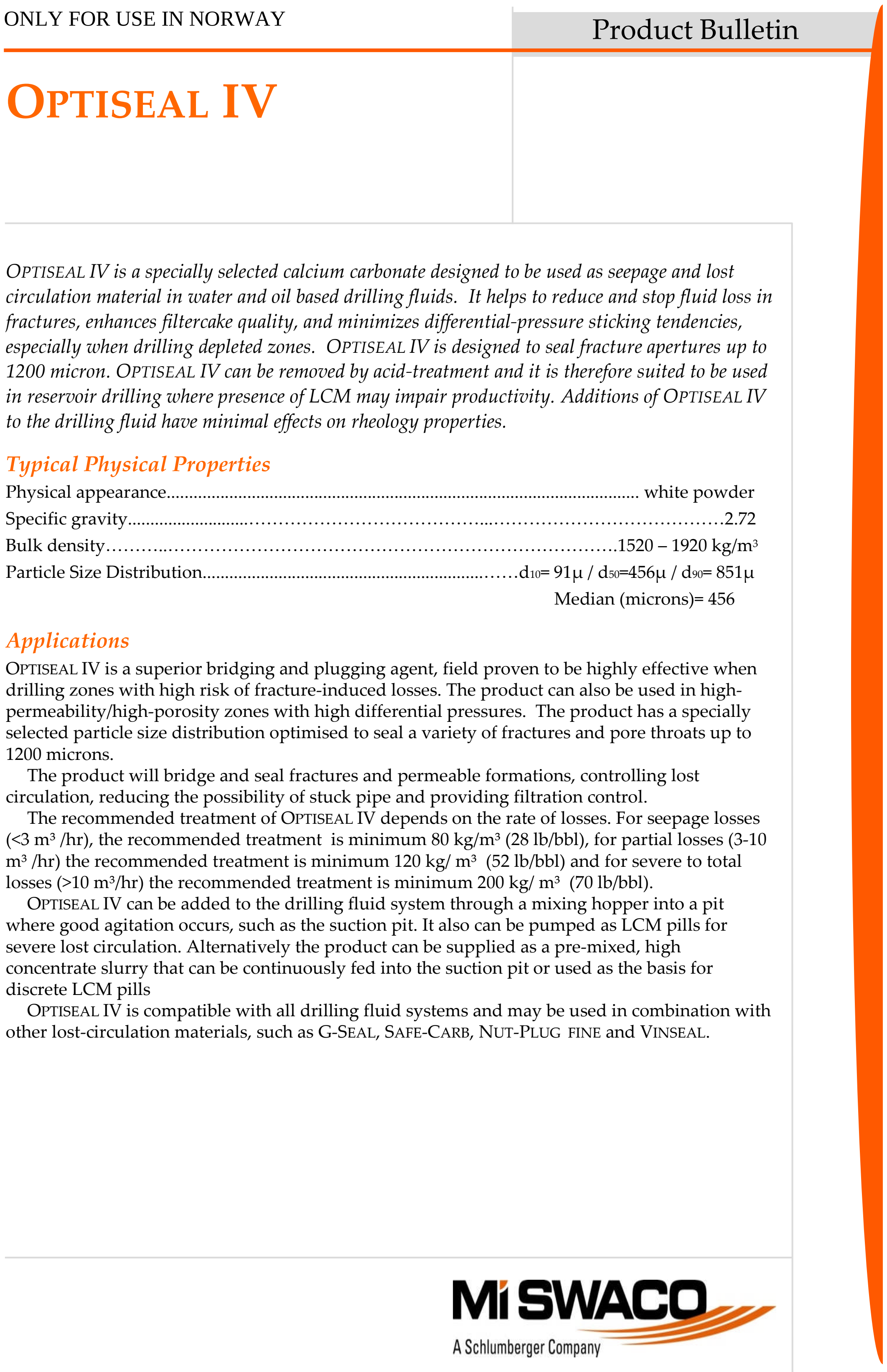
Figure D.3: OptiSeal II (Continued)



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Product Bulletin

# OPTISEAL IV

*OPTISEAL IV is a specially selected calcium carbonate designed to be used as seepage and lost circulation material in water and oil based drilling fluids. It helps to reduce and stop fluid loss in fractures, enhances filtercake quality, and minimizes differential-pressure sticking tendencies, especially when drilling depleted zones. OPTISEAL IV is designed to seal fracture apertures up to 1200 micron. OPTISEAL IV can be removed by acid-treatment and it is therefore suited to be used in reservoir drilling where presence of LCM may impair productivity. Additions of OPTISEAL IV to the drilling fluid have minimal effects on rheology properties.*

## *Typical Physical Properties*

| | |
|---|---|
| Physical appearance | white powder |
| Specific gravity | 2.72 |
| Bulk density | 1520 – 1920 kg/$m^3$ |
| Particle Size Distribution | $d_{10}$= 91µ / $d_{50}$=456µ / $d_{90}$= 851µ<br>Median (microns)= 456 |

## *Applications*

OPTISEAL IV is a superior bridging and plugging agent, field proven to be highly effective when drilling zones with high risk of fracture-induced losses. The product can also be used in high-permeability/high-porosity zones with high differential pressures. The product has a specially selected particle size distribution optimised to seal a variety of fractures and pore throats up to 1200 microns.

The product will bridge and seal fractures and permeable formations, controlling lost circulation, reducing the possibility of stuck pipe and providing filtration control.

The recommended treatment of OPTISEAL IV depends on the rate of losses. For seepage losses (<3 $m^3$ /hr), the recommended treatment is minimum 80 kg/$m^3$ (28 lb/bbl), for partial losses (3-10 $m^3$ /hr) the recommended treatment is minimum 120 kg/ $m^3$ (52 lb/bbl) and for severe to total losses (>10 $m^3$/hr) the recommended treatment is minimum 200 kg/ $m^3$ (70 lb/bbl).

OPTISEAL IV can be added to the drilling fluid system through a mixing hopper into a pit where good agitation occurs, such as the suction pit. It also can be pumped as LCM pills for severe lost circulation. Alternatively the product can be supplied as a pre-mixed, high concentrate slurry that can be continuously fed into the suction pit or used as the basis for discrete LCM pills

OPTISEAL IV is compatible with all drilling fluid systems and may be used in combination with other lost-circulation materials, such as G-SEAL, SAFE-CARB, NUT-PLUG FINE and VINSEAL.

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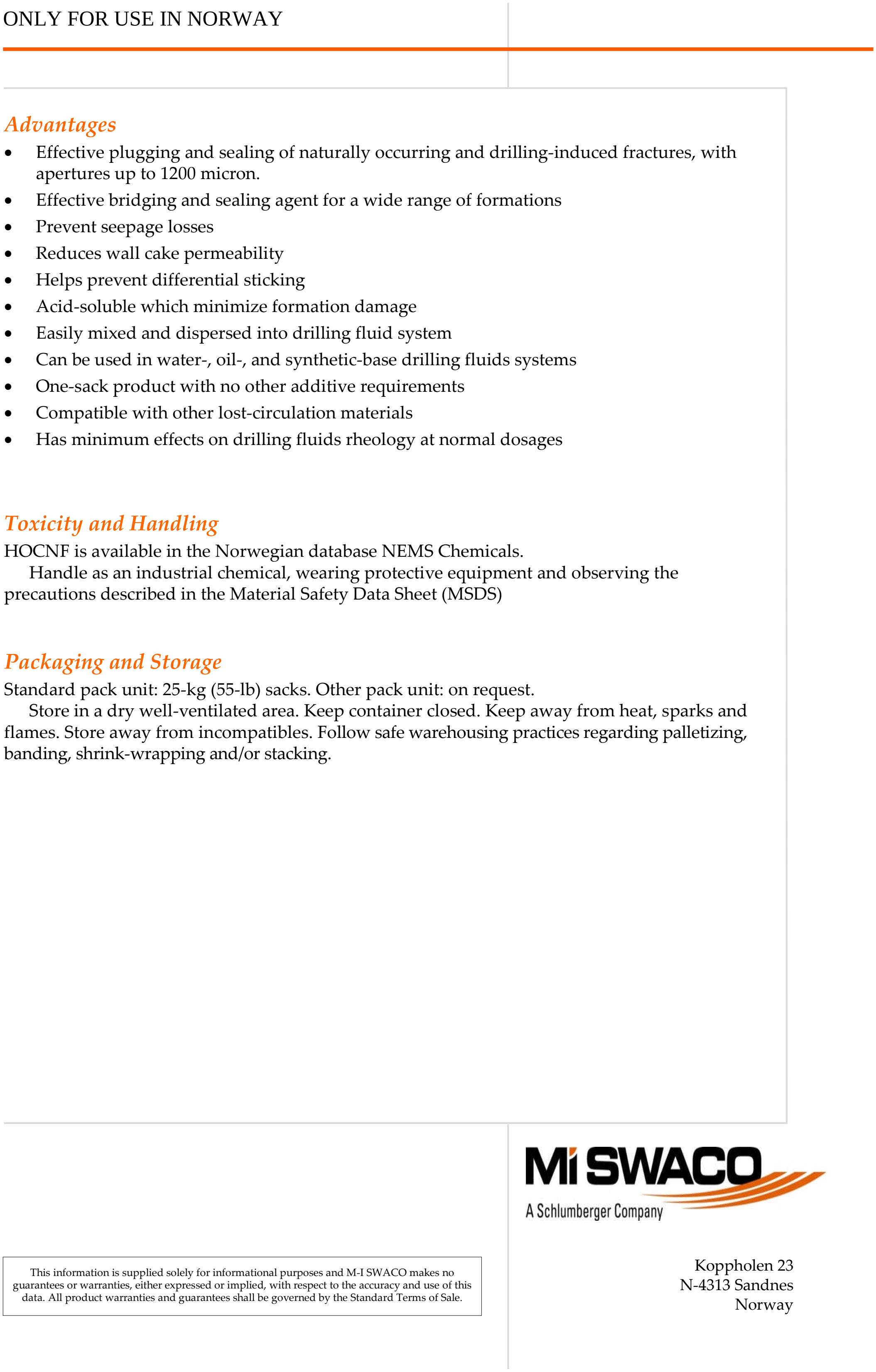
**Figure D.4:** OptiSeal IV



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## *Advantages*

- Effective plugging and sealing of naturally occurring and drilling-induced fractures, with apertures up to 1200 micron.
- Effective bridging and sealing agent for a wide range of formations
- Prevent seepage losses
- Reduces wall cake permeability
- Helps prevent differential sticking
- Acid-soluble which minimize formation damage
- Easily mixed and dispersed into drilling fluid system
- Can be used in water-, oil-, and synthetic-base drilling fluids systems
- One-sack product with no other additive requirements
- Compatible with other lost-circulation materials
- Has minimum effects on drilling fluids rheology at normal dosages

## *Toxicity and Handling*

HOCNF is available in the Norwegian database NEMS Chemicals.

Handle as an industrial chemical, wearing protective equipment and observing the precautions described in the Material Safety Data Sheet (MSDS)

## *Packaging and Storage*

Standard pack unit: 25-kg (55-lb) sacks. Other pack unit: on request.

Store in a dry well-ventilated area. Keep container closed. Keep away from heat, sparks and flames. Store away from incompatibles. Follow safe warehousing practices regarding palletizing, banding, shrink-wrapping and/or stacking.

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Figure D.4: OptiSeal IV (Continued)