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Universitetet
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Tlf. 51 83 10 00 - Fax 51 83 10 50

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Besøksadresse: Servicebygget, Ullandhaug. E-post postmottak@uis.no

EVALUATION OF KINETIC GAS HYDRATE INHIBITORS IN 1.1 S.G. DW WBM

MARIANNE NILSEN

UNIVERSITY OF STAVANGER

MASTER THESIS
2011

1 ABSTRACT

Intention: This thesis is written as a finishing part of my master degree at the University of Stavanger (UiS). The project has been in collaboration with MI-SWACO, A Schlumberger Company, with Irene M. Færgestad as my teaching supervisor in this Company. Malcolm Kelland has been my teaching supervisor at the UiS.

Objectives: This thesis is a project on drilling fluids and gas hydrates linked together with laboratory testing. The project is a large project done for a customer of MI-SWACO, going over several years where this thesis just is a small part of it. The main part for me has been standard laboratory testing of water based drilling fluids, and a smaller part on the gas hydrate testing. The purpose has been to be able to make a good 1.1 s.g. water based drilling fluid with all its qualities intact, which also contains an effective chemical inhibitor to prevent gas hydrate formation, without the inhibitor affecting the drilling fluid negatively.

Methods: A base fluid formulation was made and tested. Further drilling fluids consisting of the base formulation added chemical inhibitors were also made and tested. The drilling fluid tests consisted of: Rheology, pH, API fluid loss, ageing, dispersion and accretion –tests. Based on the results from these tests, some of the drilling fluids were taken to the next level and the gas hydrate test. The gas hydrate testing was performed using a Physica rheometer. The formation of gas hydrates could be seen as an increase in viscosity and decrease in pressure. Also, the melting process was able to be detected by using this equipment.

Results: The drilling fluid test showed that the mud-formulations with best result were formulations 3 and 9. These two had acceptable results throughout the entire testing. Thereby these two, together with formulations 1 and 7 were tested on the Physica gas hydrate test equipment.

Throughout the gas hydrate test, only formulation 7 gave no gas hydrate formation and seemed to be successful. But his was no surprise, since it contained thermodynamic inhibitors known to have a good preventive effect on gas hydrate formation. Unfortunately, none of the other formulations tested had any good results. A lot of gas hydrate was formed and the inhibitors did not seem to work very well.

Conclusion: Based on these tests results, more testing must be performed to reach the goal of creating good water based drilling fluid at 1.1 s.g. for deepwater drilling, with a combination of LDHI and THI inhibitor having a good inhibitive effect on gas hydrate formation.

2 ACKNOWLEDGEMENT

This project is the final part of my master degree in Offshore Environmental Engineering at UiS. It started autumn of 2010 when Mr. Malcolm Kelland at UiS put me in contact with Mr. Egil Ronaes, the R&D Manager ETC at MI-SWACO to explore the possibility of cooperation. The first meeting concluded that my master thesis should deal with drilling fluids and gas hydrates in collaboration with MI-SWACO. Several meetings were held throughout the autumn, also including Mrs. Irene Mangersnes Færgestad, Research Chemist ETC MI at MI-SWACO, who was appointed as my teaching supervisor at the company. Laboratory and theory preparations were also performed during this time, before my master thesis could formally start in January 2011.

Working with this thesis has been an extremely interesting process of learning. It has been a great opportunity to learn about a topic that I find really interesting and I have gained knowledge that will be of great value for me later. An added bonus has been to experience how the industry and an industrial company works, an experience I, as a student, am very grateful to get. Throughout the months I have learned a lot about drilling fluids, laboratory work, gas hydrates and the process of writing a thesis in such a large scale as a master thesis is. It has been a steep learning curve, but I have enjoyed every moment of it. This is thanks to all the great employees at MI-SWACO. They have always been very helpful and supporting all the time I have spent there, and always with a smile for me.

I want to use this opportunity to give a special thanks to Mrs. Irene M. Færgestad, who has been an excellent teaching supervisor at MI-SWACO. She has trained me well in the laboratory and I have learned a lot from her that I will benefit from in the future. She has been a great support during the writing of this thesis, and she has truly given of herself for me to make this master thesis as good as possible and to make me feel comfortable at MI-SWACO. It has been a pleasure to work with her all these months!

Special thanks also go to Mr. Egil Ronaes, who let me take part in their project and write my thesis at MI-SAWACO. He made all conditions favorable for me and introduced me to Mrs. Irene M. Færgestad. At the same time I want to thank all the great people at the RD and DS -department for all the help and support. You all made me feel welcome and also made me look forward to come to MI-SWACO every day!

I also want to thank Mr. Malcolm Kelland, professor at the University of Stavanger, for once again being my supervisor at the UiS and supporting me in getting a project at MI-SWACO and throughout this master thesis project.

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6 INTRODUCTION

Oil and gas industry is a large industry in Norway and other parts of the world. A lot of oil and gas reservoirs have been discovered and developed over the last few years. Platforms have been built, wells have been drilled, and there are still reservoirs that are discovered and new wells being drilled. In order to drill a well, a lot of advanced equipment and people with expertise are required. One important component used during drilling is the drilling fluid. A drilling fluid is a mixture of several different components, where each component has a purpose. The composition of each drilling fluid can vary, depending of the field it will be used in. A drilling fluid should possess many qualities to ensure a successful and safe drilling. Drilling fluids are also called drilling mud, or just mud. Drilling fluids have several important functions as will be described later in this thesis. Several types of drilling fluids are used offshore in the oil and gas industry. There are 3 main types of drilling fluids: [1]

- Water based drilling fluid (WBDF)
- Oil based drilling fluid (OBDF)
- Synthetic based drilling fluid (SBDF)

SBDF is the most effective of them, but since it is expensive, it is not preferred. OBDF have negative impact on the environment and has no relation to this thesis. Neither of these two fluids will be further mentioned in this thesis. [1]

Another big issue in the oil and gas production is safe flow assurance. It is extremely important to have a secure and safe transport of the hydrocarbons throughout the different systems. A problem connected to this is the formation of gas hydrate. Gas hydrate is like an "ice structure" containing gas and water. It is formed under high pressure and low temperature. Gas hydrates may cause huge problems if they are allowed to grow in the all the pipelines and equipment belonging to the oil and gas plant/ installation. Gas hydrates may also cause problems in completion fluid and drilling fluids. There are three gas hydrate structures, and these are: [2]

- Structure I
- Structure II
- Structure H

Structure II is most common for the oil and gas industry. There are many ways of preventing gas hydrate, where the use of a chemical inhibitor is most common. Chemical inhibitors can be divided into groups, as explained later in this thesis.

7 THEORY

7.1 DRILLING FLUID

7.1.1 WATER BASED DRILLING FLUID

Water based drilling fluid (WBDF) is one of the most common types of drilling fluids used offshore. This is mainly due to the environmental considerations and new technology. WBDF is friendlier to the environment because it consists of a large part of water. WBDF cannot be used in all drilling scenarios offshore (In such situations oil based drilling fluid (OBDF) is used instead). A WBDF usually consists of: [1]

- A water phase; this can be salt water, saturated salt water, or fresh water
- A reactive phase; clay
- A non reactive phase; weight materials
- Other chemicals; polymers, thinners

There are generally 3 different types of WBDF: [1]

- Seawater mud
- Freshwater mud
- Inhibitive water based mud

7.1.1.1 SEAWATER MUD

Seawater mud has a salt content which is similar to the salt content of the sea water. It is normal to use pre-hydrated bentonite or seawater-lignosulphonate to achieve the necessary viscosity and to help fluid loss additives provide fluid loss control. If bentonite is used, it has to be added continuously because the pre-hydrated effect will decrease over time. [1]

7.1.1.2 FRESHWATER MUD

Within the freshwater drilling fluids there is one type of mud that is essential, and that is bentonite-mud. One of the benefits of this mud is that it is not necessary to use high volumes of bentonite to reach the wanted viscosity. It is also possible to use thinners or polymers to control the viscosity. Another benefit with this mud is that it has little or no need for other chemicals. This mud is also easy to recycle, which means that the loss of mud is little, which is an economic benefit. The disadvantage of a freshwater mud is that it is not inhibitive; you need to make it inhibitive by adding suitable chemicals, but that can also affect the viscosity negatively. Another downside is that this mud does not cope well with salt pollutions. [1]

7.1.1.3 INHIBITIVE WATER BASED MUD

An inhibitive water based mud will prevent swelling of clay from happening while drilling the formation. This mud is used where there is risk that the clay will swell, or in salt formations where there is risk of scouring. When clay swells it expands due to free space between the layers of montmorillonite. It has the ability to receive compounds that will attach in between the layers and increase the size of the clay. This swelling problem can be seen in figure 1. There are different mechanisms of inhibition: [1, 3]

- Polymer inhibition
- Salt inhibition
- Alcohol inhibition

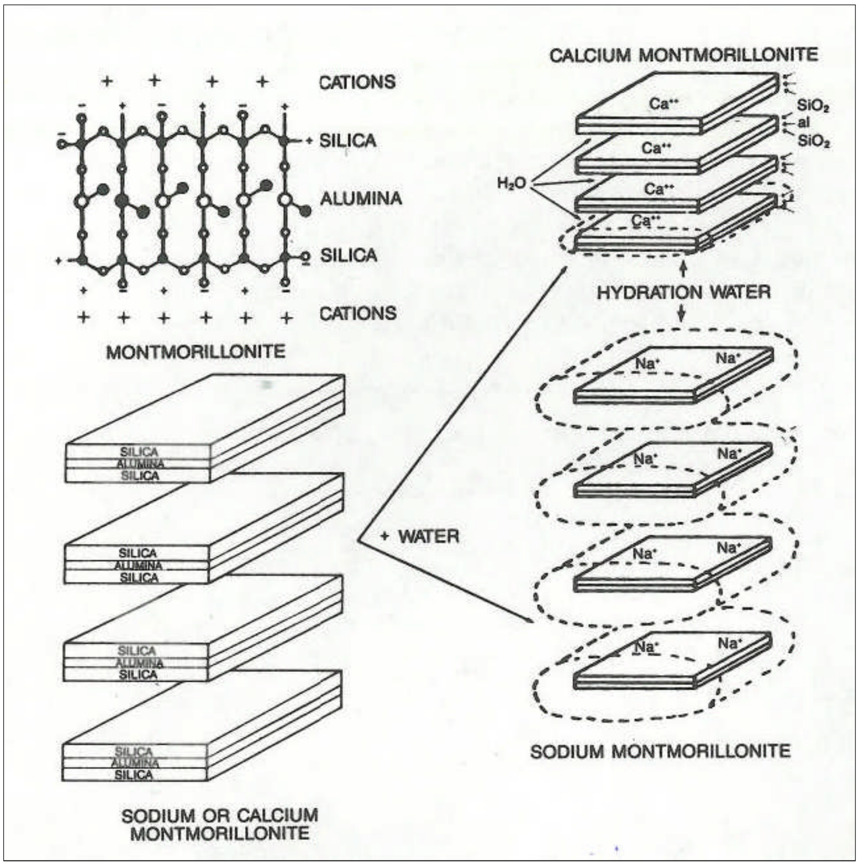

Figure 1: Swelling of clay. [4]

7.1.1.3.1 POLYMER INHIBITION

These polymers are large molecules, with high molecular weight. They will easily adhere to the clay particle surface and plug or seal the pores/ cracks in the formation, so that the access of water will be limited and the swelling reduced. The polymers must be added continuously because they will be 'consumed' during drilling. [1]

7.1.1.3.2 SALT INHIBITION

Salt inhibition means that metal-ions which have higher binding energy will replace the interchangeable ions in the clay. This is normally done by Na^+, but also K^+ and Ca^{2+}. When it happens with Ca^{2+}, the inhibition mechanism is related to the divalent ion and will bond to clay structures. This will create an osmosis-swelling and prevent the water from penetrating into clay structures. The inhibition effect of K^+ is that it is so small that it can enter the hole in the hexagonal structure in tetrahedron-layer and make the clay particles come closer together and thereby prevent hydration. [1]

Normally combinations of both of these two inhibition mechanisms are used. Different types of combinations in water based muds used to inhibit clay formations are: [1]

- Lime ($Ca(OH)_2$
- Gypsum ($CaSO_4$)
- Calcium Chloride (KCl)
- Sodium Chloride (NaCl)
- Lignosulphonate

7.1.1.3.3 ALCOHOL INHIBITION

This is a new technology, based on inhibitive water based mud mixed with multivalent alcohols (3-6 volume %). Normally KCl and Glycol are added simultaneously, which gives a good inhibition effect on the clay structure. [1]

7.1.2 FUNCTIONS OF A DRILLING FLUID

When drilling a well, a suitable drilling fluid is essential to achieve a successful drilling. Which drilling fluid to use, depends of the area drilled in, well conditions and the current operations offshore. The drilling fluid has several different important qualities as listed below: [5-7]

- Remove cuttings from the well
- Control formation pressure
- Suspend and release cuttings
- Seal permeable formations
- Maintain wellbore stability
- Minimize reservoir damage
- Cool, lubricate and support the bit and drilling assembly
- Transmit hydraulic energy to tools and bit
- Ensure adequate formation evaluation
- Control corrosion
- Facilitate cementing and completion
- Minimize impact on the environment

7.1.2.1 REMOVE CUTTINGS FROM THE WELL

During drilling cuttings are generated by the bit and must immediately be removed. If not, these cuttings will be crushed into even smaller parts by the bit and be more difficult to remove and also slow down the drilling rate. Thus the drilling fluid is constantly circulated down the drill string and pushed out through the bit in order to remove cuttings. Removal of cuttings is also called “hole cleaning”. The drilling fluid will transport the cuttings to annulus and all the way to the surface, where it will be separated from the drilling fluid by use of different equipment. [5, 6]

Cuttings are heavier than the drilling fluid, so the particles will sink in the fluid as they are transported up to the surface. This rate of particle settling is called “slip velocity”. The slip velocity depends on the cuttings shape, size, density and the velocity of the drilling fluid. To get the cuttings transported to the surface, the velocity of the drilling fluid (called *annular velocity*) must be greater than the slip velocity of the cuttings. The total speed of the cuttings from annulus is then the difference between annular velocity and the slip velocity and is called *"transport velocity".* In a vertical well, the case will be as follows: [5]

Transport velocity = Annular velocity – slip velocity. [5]

Drilling fluids are often thixotropic. This means that under a down time where there is no circulation, the fluid will form a gel and all the cuttings will stay suspended in the fluid, and thus not sink to the bottom. But as soon as drilling starts up again, the gel is broken. [5]

As mentioned above, rate of descent depends on the cuttings shape, size and density, but also "rate of penetration" (ROP) and viscosity. The viscosity should be as low as possible to get through the nozzles, but at the same time it should also be high enough to get the necessary lifting capacity. A drilling fluid with low viscosity will have high settling rate of the cuttings, while a fluid with high viscosity will improve the transport of cuttings. The balance can be difficult to find and tests in the laboratory are carried out to help find the best choice here. [5, 6].

7.1.2.2 CONTROL THE FORMATION PRESSURE

It is of high importance that the formation pressure is controlled at all times in order to prevent severe drilling difficulties. [5]

Under normal circumstances, when formation pressure increases, barite is added to the fluid to increase the density of the fluid to maintain control over the formation pressure and wellbore stability. If this is not done, fluid will flow into the wellbore and may cause a blowout situation. [5]

The pressure from the static drilling fluid column is called the hydrostatic pressure and is a function between the true vertical depth (TVD) and the density of the drilling fluid. The density of the drilling fluid is limited by the maximum weight that will not crack the formation and by the minimum weight needed to control the formation pressure. This means that the density of the drilling fluid should be as low as possible to achieve wellbore stability and control over the well drilled in. It is essential to keep the hydrostatic pressure equal to or greater than the formation pressure, because then the fluid will not flow into the wellbore and the chance of a blowout is avoided. However, sometimes the formation fluid is anyhow allowed to flow into the wellbore, but this is closely monitored during the operation. This is done when the levels of background gases are high and it is producing oil and gas during drilling. [5]

7.1.2.3 SUSPEND AND RELEASE CUTTINGS

Drilling fluids should be able to suspend weight materials and cuttings, but at the same time it must let the cuttings be removed on the surface in the solids control equipment. Particles that settle during down time or any other static conditions may cause problems such as stuck pipe or lost circulation. These settling particles create a variation in the density of the fluid often referred to as *"sag".* Sag normally occurs during drilling in high angles with a dynamic situation where the circulation has a low annular velocity or in static conditions. [5]

High concentrations of solids in the drilling fluid will negatively affect many sides of the drilling operation. The drilling efficiency and the ROP will decrease. More power to achieve the right circulation will also be necessary. The weight of the drilling fluid and the viscosity will increase, which will increase maintenance and costs. It will also give the need for dilution of the drilling fluid. The filter cake may as a result turn out thicker and differential sticking of the drill string can also become a problem with a lot of solids in the drilling fluid. Therefore, the properties of the drilling fluid must allow cuttings removal by solids control equipment. This is a fine balance, since cuttings suspension requires shear thinning thixotropic qualities and high viscosity, also the solids removal will function better for a drilling fluid with low viscosity. The solids removal equipment does not work at its optimum on fluids that are shear thinning, with high content of solids and high plastic viscosity. It is therefore very important to remove as much as possible during the first circulation and prevent cuttings from following back down and into the well. Then they will be crushed into even smaller pieces and be more difficult to remove (as mentioned above). A method used to see if the cuttings are removed or not is to compare the sand content in the drilling fluid at the flow line and suction pit. [5, 8]

7.1.2.4 SEAL PERMEABLE FORMATIONS

Permeability says something about the ability of fluid to flow through porous formation. In order for hydrocarbons to be produced, the rock/formation must be permeable. If the formation pressure is too low, fluid will escape into the formation, and a thick filter cake of drilling fluid particles will deposit on the wall of the well. A good drilling fluid will create a thin and firm low permeability filter cake, so the fluid will not be lost to the formation. A good filter cake prevents a lot of drilling and production problems and helps ensure wellbore stability. If not, problems like poor logging qualities, too tight hole conditions, increased drag and torque, lost circulation, stuck pipe and formation damage can occur. If the formation is highly permeable, almost all of the drilling fluid can be lost into the formation only depending on the size and shape of the solids. To prevent this, agents that will create a bridge and block large openings so that the particles can seal the opening, are used. These bridging agents must at least be half the size of the biggest opening to function. It can consist of ground cellulose, calcium carbonate and a wide variety of seepage loss or other fine lost circulation materials. [5, 9]

There are several chemicals that can be used to help provide a good filter cake, such as asphalt, bentonite, gilsonite, polymers (natural or synthetic) or organic deflocculating chemicals. [5, 7]

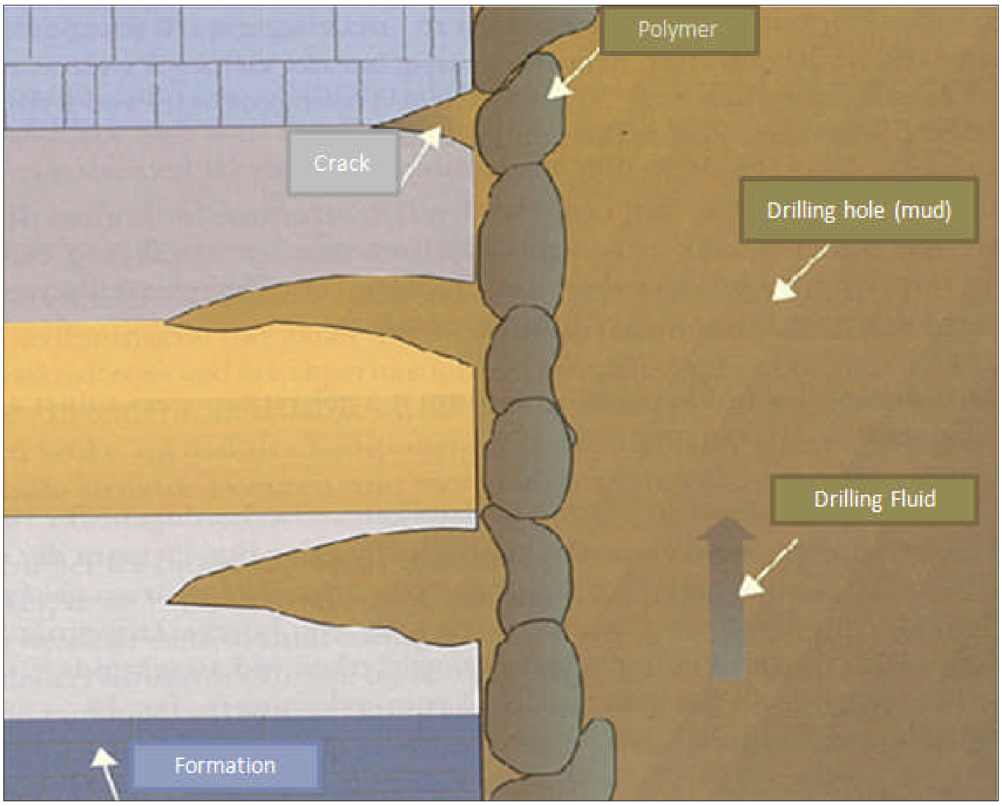

Figure 2: Filter cake (grey) on the hole wall. [9]

7.1.2.5 MAINTAIN WELLBORE STABILITY

To obtain wellbore stability during drilling, the balance between mechanical and chemical aspects must be in order. Before the casing is set and cemented, the chemical properties of the drilling fluid must meet set specifications to make sure that the wellbore is stable. Also, mechanical aspects like stress and pressure must be in place. To balance the mechanical forces on the wellbore it is also important that the drilling fluid has the correct weight. Indication of uncontrolled wellbore can often be seen as *"tight hole, sloughing formation, bridges and fill on tips".[5]* This can develop so it can be necessary to go back to the original depth. Too big a hole is also not a wanted situation because it can lead to several problems; poor hole cleaning, low annular velocity, more solids, higher cost due to cementing and inadequate cementing and poor formation evaluation. The hole can be too big trough sand and sandstone formations, because these need large mechanical forces. To limit the hole size a good hydraulic program and a good filter cake with bentonite can be used. Drilling in shale normally provides stable conditions in the beginning, but with water based mud it can over time lead to swelling or softening. This will further lead to new problems that give unstable drilling conditions. Swelling/softening can be prevented by using systems containing potassium or calcium, or chemicals like salts, glycols, polymers, oils, asphalt materials, surfactants or other shale inhibitors. If it is too difficult to drill in a shale formation using WBDF, oil based or synthetic drilling fluids are preferred since these are better suited to inhibit swelling or softening. [5]

7.1.2.6 MINIMIZE FORMATION DAMAGE

Formation damage may severely reduce the production, and should be avoided. If the permeability or the porosity is reduced in any way, this is considered as formation damage or skin damage. The reason for this can be mud or solids plugging the pores. [5]

When completing the well, the damage on the formation is evaluated and a protection procedure is carried out. Normally it is possible to produce oil and gas is even if there is some formation damage. To avoid or create as little damage as possible, special drilling fluids, work over and completion fluids are used.[5]

The most common mechanisms causing formation damage during drilling are: [5]

- *Mud or solids invading the formation matrix, plugging pores.*
- *Swelling of formation clays within the reservoir, reducing permeability.*
- *Precipitation of solids as a result of mud filtrate and formation fluids being incompatible.*
- *Precipitation of solids from the mud filtrate with other fluids, such as brines or acids, during completion or stimulation procedures.*
- *Mud filtrate and formation fluids forming an emulsion, restricting permeability.*

7.1.2.7 COOL, LUBRICATE AND SUPPORT THE BIT AND DRILLING ASSEMBLY

When a well is drilled a large amount of heat is produced by the mechanical and hydraulic forces. Thus another quality the drilling fluid should possess, is the ability to cool the drill bit, because during a drilling operation there will be a lot of heat generation due to the pressure, the circulating of the bit and stone being crushed . To maintain a cooled drill bit, cold drilling fluid is constantly being pumped down and warm drilling fluid is transported back to the top, where the heat is transferred to the atmosphere. [5, 6]

The drilling fluid should also lubricate and reduce friction. All the equipment used in the drilling, like mud motors, the bit and components from the drill string, will fail more rapidly if it is not cooled down or lubricated throughout the drilling. It is possible to measure how well a drilling fluid lubricates. Factors that affect the lubrication is the amount of cuttings, type of cuttings, hardness, the pH, salinity, chemicals and the weight material used. If a drilling fluid does not lubricate as well as desired, this can be seen in high torque, much wear, drag and abnormal heat creation. But all of these problems can also have another reason. [5, 6]

7.1.2.8 TRANSMIT HYDRAULIC ENERGY TO TOOLS AND BIT

When drilling a well, the rate of penetration (ROP) should be as high as practically possible, which can be achieved by using the hydraulic energy and remove all of the cuttings at the bit. Hydraulic energy is also (a part of) giving energy to mud motors. The size of the nozzle is important to help get rid of all the cuttings in the well. Therefore, the size of the nozzles is carefully considered before drilling. The best suited drilling fluid with properties as being shear thinning, having low solids content and being able to reduce drag will help the hydraulic system to be at its best function. [5]

7.1.2.9 ENSURE ADEQUATE FORMATION EVALUATION

A drilling fluid can provide information about geological and formation aspects by means of special analyses of the fluid and cuttings. All the chemical and physical qualities of a drilling fluid will affect the system further and be an important part of the formation evaluation. Thus a good drilling fluid is not only essential in the well, but also afterwards when it comes to the surface. [5, 6]

7.1.2.10 CONTROL COROSION

A drilling fluid must not be a source of corrosion on the drill string, the casing or any other part of the drilling equipment. Therefore, drilling fluids must have good chemicals that are alkaline, and normally with pH > 9,5. High pH has two positive sides; it keeps corrosion away and maintains chemicals in solution. For example; for lignosulphonate, which is a chemical that can be degradable whilst drilling, degradation will not be a problem when pH is kept high. [6, 8]

Different chemicals can be added to a drilling fluid to prevent corrosion. It can be components that will reduce oxygen content or make a protective film on the steel. The companies usually have a long list of chemicals that can be added for this purpose. [6, 9]

7.1.2.11 FACILITATE CEMENTING AND COMPLETION

Drilling a well usually starts with a 36“ drilling hole, followed by a casing and cementing around to secure the well, and this goes on further and further down as the well is being drilled, with smaller and smaller holes. Normally the narrowest hole is around 8 ½“ followed by a smaller casing and cementing. During this operation it is extremely important that the drilling fluid is at its optimum for that exact well and area drilled in, having a thin and firm filter cake, low viscosity, and low, non-progressive gel strengths. [5, 9]

7.1.2.12 MINIMIZE IMPACT ON THE INVIRONMENT

The use of drilling fluids is also a question of environmental impact. If it is possible to dispose the used drilling fluid on site this is obviously the best solution, but then it must be environmentally friendly. It will therefore depend on which drilling fluid it is and what chemicals it contains. Water based drilling fluid is more environmentally friendly than oil based drilling fluid, but sometimes it is necessary to drill with oil based drilling fluid. Then the fluid must be transported away in tankers after use for safe disposal or it can be recycled. Normally, when making a drilling fluid the chemicals used are green or yellow, which means that they would not harm the environment. If it is necessary to use red or black chemicals (which represent a hazard to the environment) this has to be applied for to the government, according to the rules for the Norwegian continental shelf (offshore). [5, 9]

7.1.3 CONFIGURATION AND STRUCTURE OF MONTORILLONITE

During a drilling operation the drill bit has to pass several different geological structures. It is normal to come in contact with clay, since clay is a normal component in the geological structure. Also, during mixing of drilling fluid, clay is added to give some of the qualities necessary in a drilling fluid. So it is of importance to understand the structure of the most common clay; montmorillonite. [1]

During a drilling operation contact with clay will occur, mainly in two settings. First when the drilling takes place, because it will then make contact with clay through the formation. Formation structures often consist of shale and clay. Secondly in connection with drilling fluids: Clay is normally added to the drilling fluid to give the appropriate/ necessary viscosity. Normally this clay will be in the form of bentonite. The name "bentonite" originally comes from USA, in Wyoming, where clay was found and named bentonite. Mineralogists call bentonite for smectite, but in the oil and gas industry nowadays the name montmorillonite is more common and is used further in this paper. [6]

The structure of clay minerals depends on two main layers. It is the composition of these two layers that gives different clay minerals different properties. These two layers are called: "*a tetrahedral layer*" and "*an octahedron layer*". Montmorillonite is built up of two tetrahedral layers with an octahedron layer in the middle, as shown in figure 3 below. [1, 6]

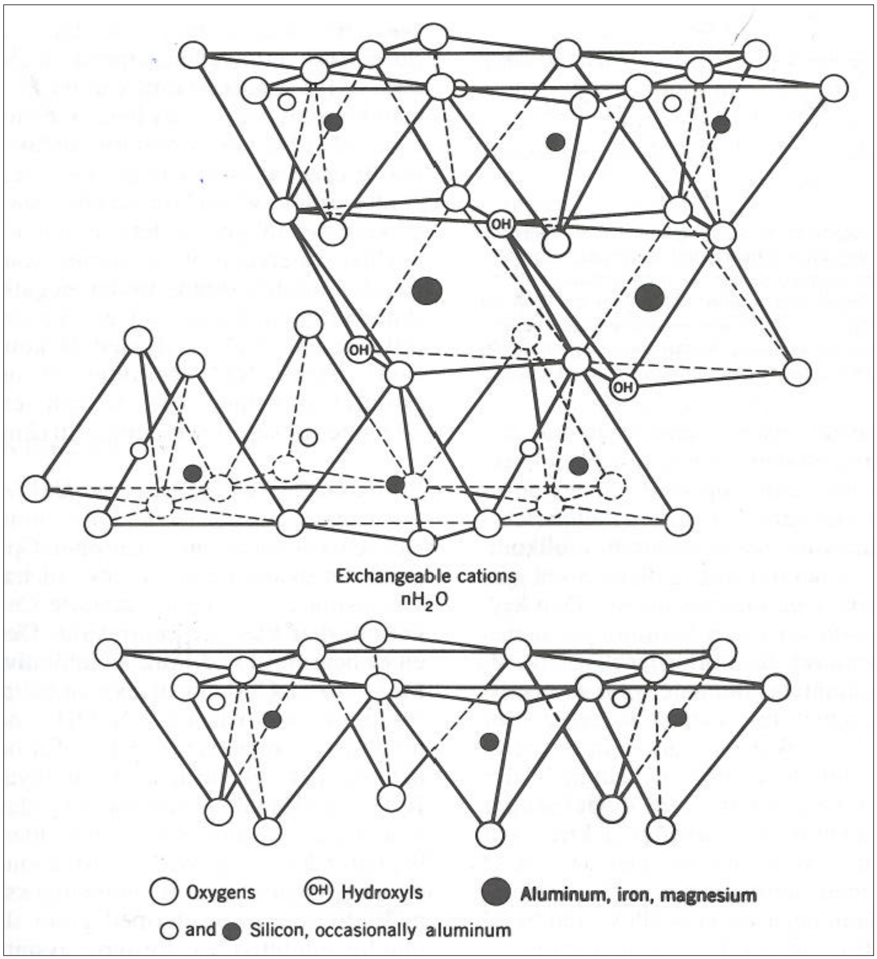

Figure 3: The structure of montmorillonite. [6]

7.1.3.1 THE OCTAHERDON LAYER

The octahedron layer consists of a dense layer of oxygen (O) molecules or hydroxide (OH) in combination with aluminum (Al). It is possible to replace aluminum with magnesium (Mg) or iron (Fe). In montmorillonite it can vary how many aluminum atoms that are replaced by magnesium. Figure 4 shows the structure of an octahedron layer as seen below. [6]

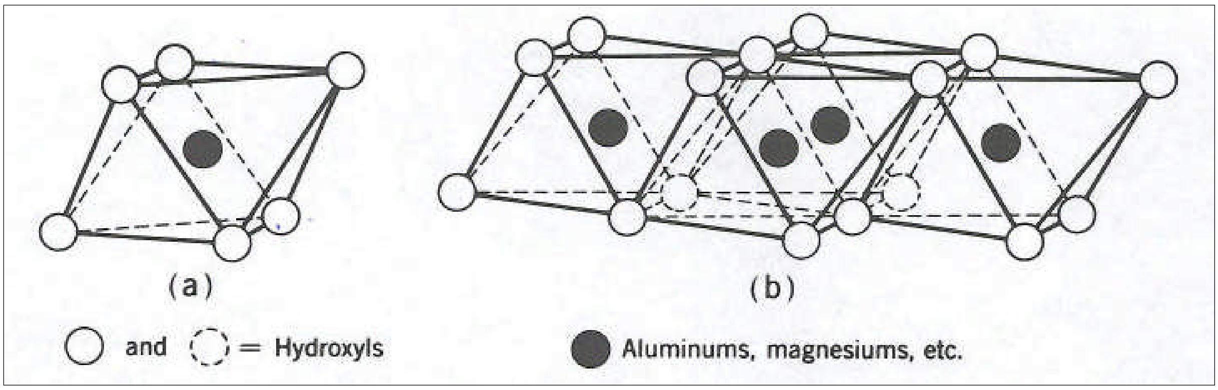

Figure 4: The structure of the octahedron layer. [6]

7.1.3.2 THE TETRAHEDRAL LAYER

The tetrahedral layer has one silicon atom (Si) in the middle and is surrounded by 4 oxygen (O) or hydroxide molecules (OH) as shown in figure 5. [6]

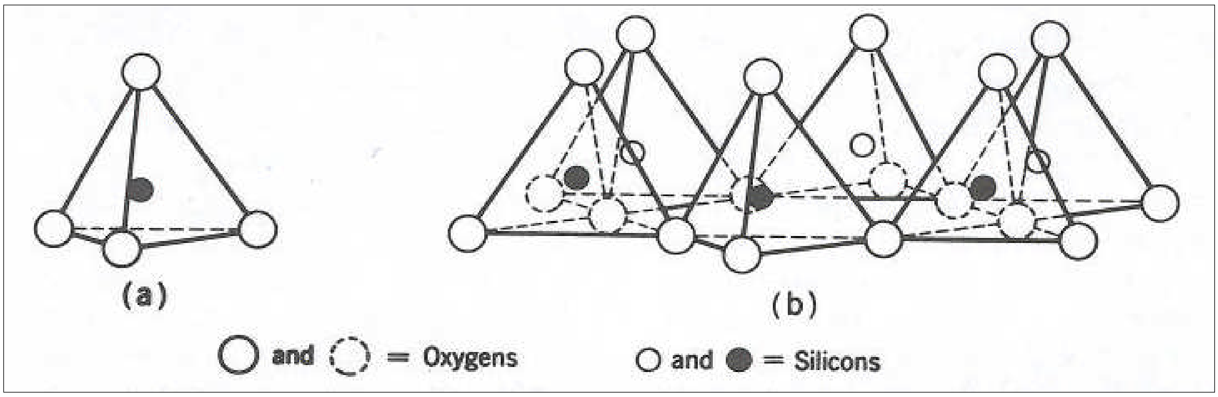

Figure 5: The structure of the tetrahedral layer. [6]

7.1.4 FOAM

Foam in a drilling fluid may occur when mixing in polymers. It can usually happen if there is enough gas that will break through from the formation. Foam in drilling fluids is not wanted because there are many negative impacts of foam. In contact with foam, pumps will go a bit “wild” and reduce the circulation stability. The weight of the fluid will decrease. Particles will not separate as well as required and instead attach to the gas and be transported back to the well hole. A solution to reduce foaming can be to let the air out “naturally” by having no circulation activity in the well over a period of time, or to add chemicals that will either decrease the foam (defoamers) or chemicals that will stop the creation of foam (anti-foamers). [9]

7.2 GAS HYDRATES

7.2.1 WHAT ARE GAS HYDRATES

Gas hydrates look like “Ice- crystals” and have gas inside the structure. There are 3 main structures of gas hydrates. These are named structure I, structure II, and structure H. These structures will be explained in more detail later. Gas hydrates are formed under situations where the pressure is high (≥ 38 bar at -46,15 °C) and the temperature is low (≤ 26,85 °C), when water and hydrocarbons are present. The temperature where formation of gas hydrates occur increases with increasing pressure. This means that the temperature can be as high as 25-30°C if the pressure is high enough. This is especially a problem for the oil and gas industry, where the operating pressure normally is high, the temperature is low, and hydrocarbons and water are present. Gas hydrates in the oil and gas industry normally occur e.g. in the drilling fluid, completion fluid/operation, production and processing in pipes, in valves and in well heads. In worst case, gas hydrates can plug different systems belonging to the oil and gas installation, which can lead to shut down of the rig or down time. Plugging of systems and pipelines is an expensive and comprehensive situation that is not wanted. There are several methods that can be used to prevent gas hydrate formation. These methods are listed here: [10, 11]

- Prevent the operating pressure and temperature from being within the gas hydrate formation zone. This zone is shown in figure 6 below.
- Remove all the water from the gas. Dry the gas with dryers or another form of dehydration (dehydration towers).
- Try to mix in another gas which is not “a gas hydrate formation gas” (this is explained under the gas hydrate structures section).
- Injection of chemicals that will prevent gas hydrate formation. These chemicals are known as “Inhibitors” (which means “preventing”).

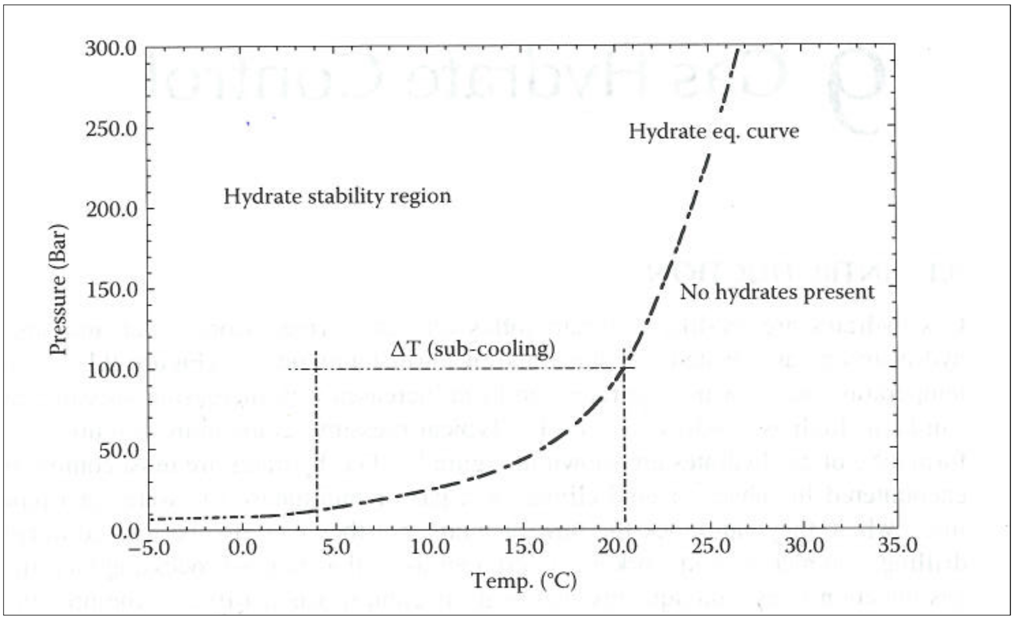

Figure 6: Gas hydrate curve. [10]

7.2.2 THE GAS HYDRATE STUCTURES

There are three different structures of gas hydrates. These are called structure I, structure II and Structure H and an overview of them is given in figure 7 and table 1 in page 27. They are separated from each other by the type of gas they contain inside the lattice, how big the structure is, the shape of it and also where they are normally located. What makes the formation of hydrates possible is the hydrogen bond, which is called the “host molecule”. Water molecules can align in regular orientations due to this hydrogen bond. The formation of solids occurs when some other compounds molecules attaches and makes it more stable. These are called the “guest” molecules. The gas hydrate structure consists of a three-dimensional structure of the host molecule with guest molecules inside of it. [2]

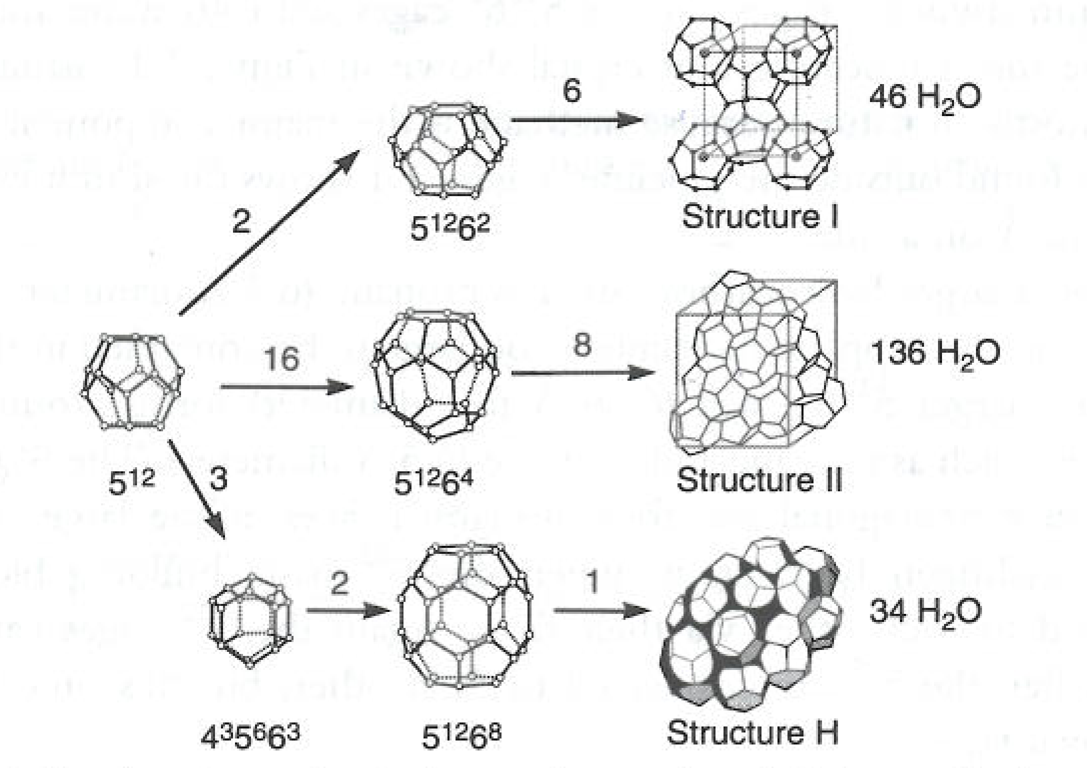

Figure 7: Gas hydrate structures. [12]

7.2.2.1 STRUCTURE I

Structure I is the smallest and simplest of these three structures. It is formed by two different cages. One is the "*Dodecahedron*", which is a polyhedron with twelve sides. Each side forms a pentagon. This is often written as this: 5^{12}. The number five refers to pentagon and the number twelve to the twelve sides. The next cage is called *"Tetrakaidecahedron".* This name comes from a polyhedron with fourteen sides connected to twelve pentagonal and two hexagonal faces, written like this: 6^2. Combining the two then gives: $5^{12}\ 6^2$. [2, 13]

There are 46 water molecules in structure I and the guest molecule can vary between methane (CH_4), ethane (C_2H_6), hydrogen sulfide (H_2S) or carbon dioxide (CO_2). If the hydrate molecule is written as "*X*", the theoretical formula will be: *X*5 ¾* H_2O. Structure I is shown in figure 8 below. [2]

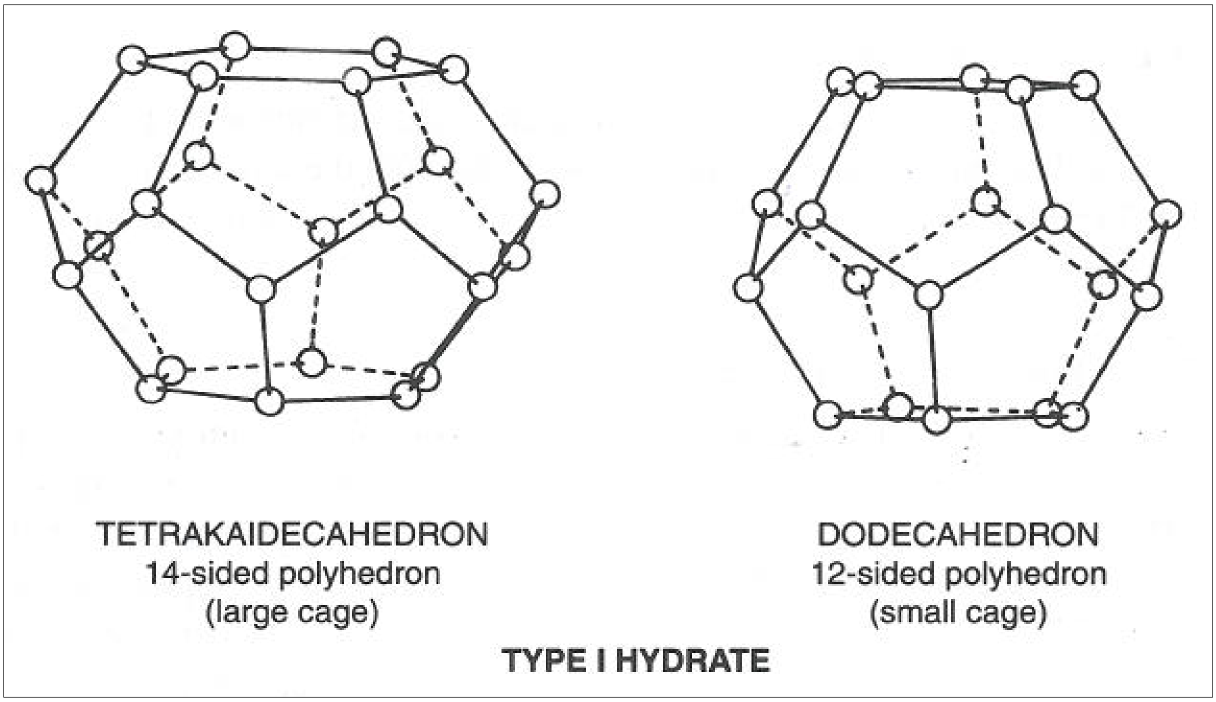

Figure 8: Structure I. [2]

7.2.2.2 STRUCTURE II

This structure is more advanced than structure I but is, like structure I, also formed by to different cages, as seen in figure 9. One is Dodecahedron, which was described above. The second cage is called *"Hexakaidecahedron".* This is a polyhedron with sixteen sides and four hexagonal and twelve pentagonal faces. [2]

There are 136 water molecules in structure II and if all cages are filled with guest molecules, the theoretical formula is then: $X * 5\ 2/3\ H_2O$. Normally though, only the biggest cages are occupied by guest molecules. The correct theoretical composition is then: $X*17H_2O$ .However, the composition may vary, thus each case may be unique. [2]

Structure II is the structure which is most common in the oil and gas industry. This has its explanation in the gas composition which will support the formation of structure II gas hydrates. Structure II will utilize gases like propane (C_3H_8), nitrogen (N_2) and isobutene to form, and these are gases that there are a lot of in the oil and gas industry. [2]

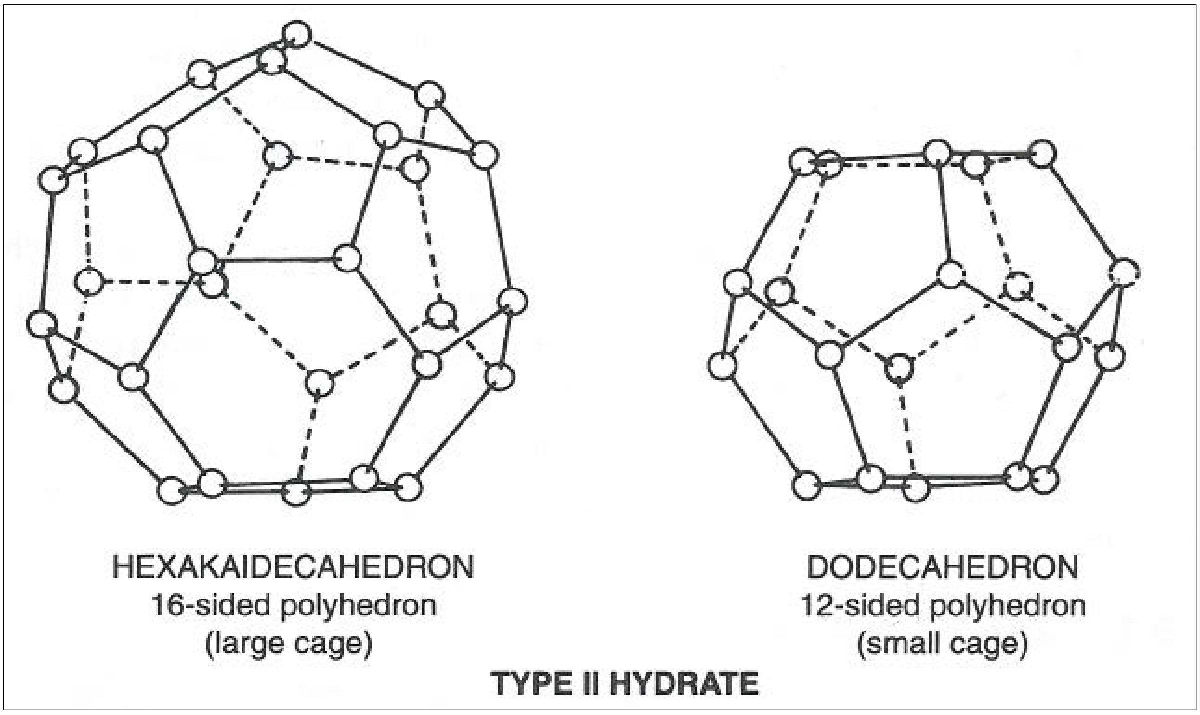

Figure 9: Structure II. [2]

7.2.2.3 STRUCTURE H

This structure is the least common of the three gas hydrate structures. Structure H is built up by three cages: First the dodecahedron, which is a small cage, the second cage is an *"irregular dodecahedron"*, medium size, which has three square faces with three hexagonal and six pentagonal faces. The third cage is larger, an *"irregular icosahedron"*. This cage has a polyhedron with twenty sides, eight hexagonal and twelve pentagonal faces. Combined it consists of 34 water molecules as shown in figure 10 below. (This was the best possible scan obtained, a better picture can be found in the original reference 7.) [2]

In structure H, normally gas as methane (CH_4) enters the small and also the medium size cages. In the largest cage there will be gases, like *2-methylbutane, 2,2-dimethylbutane, 2,3 dimethylpentane, methylcyclopentane, ethylcyclopentane, methylcyclohexane, cycloheptane and cyclooctane. [2]* Structure H is rarely seen in the oil and gas industry because these gases are not present there. The theoretical formula for structure H is rather difficult to give, but it is always made of double hydrates and is therefore given like this: $Y * 5X * 34\ H_2O$. [2]

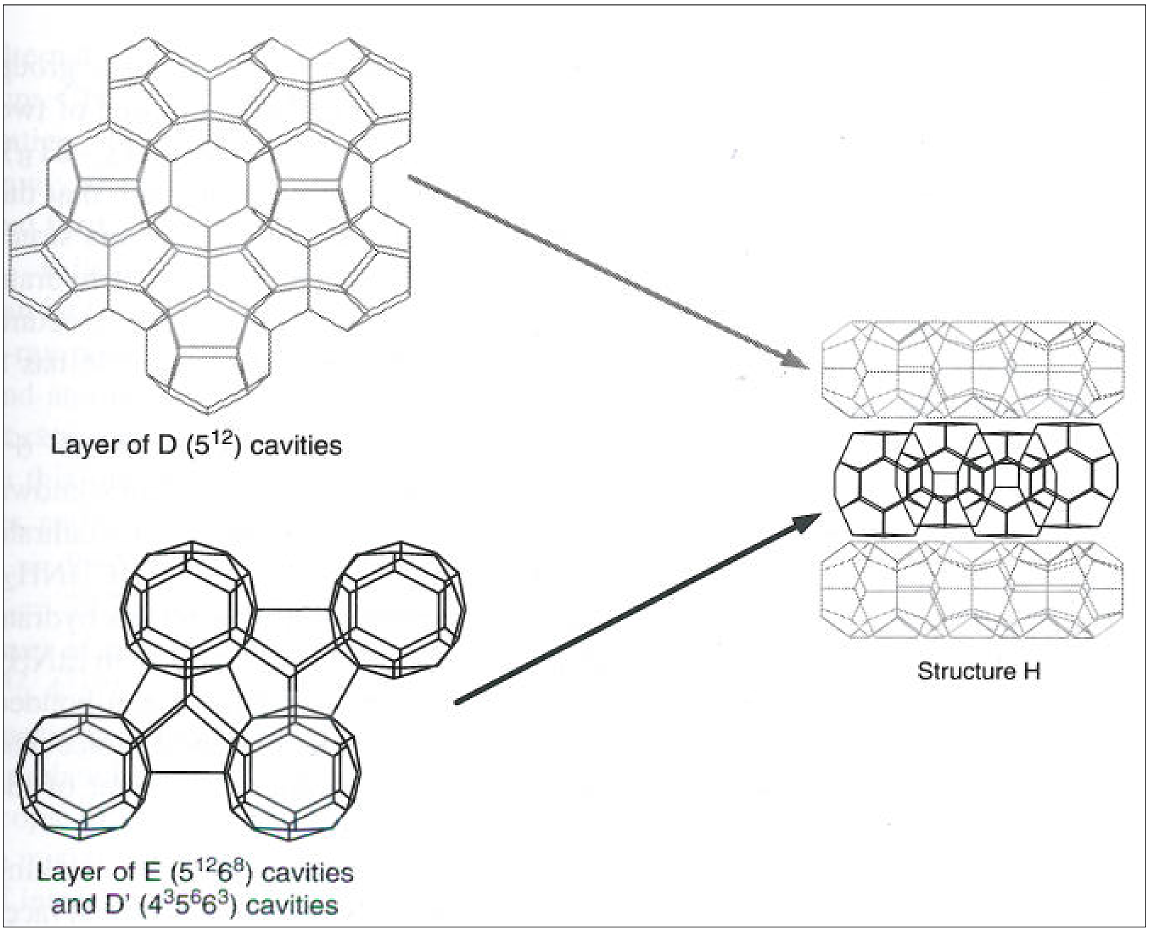

Figure 10: Structure H. [13]

Table 1: An overview of all the structures and their properties. [2]

	Type I	**Type II**	**Type H**
Water Molecules per Unit Cell	46	136	34
Cages per Unit Cell			
Small	6	16	3
Medium	-	-	2
Large	2	8	1
Theoretical Formula *			
All cages filled	X*5 ¾ H_2O	X*5 2/3H_2O	5X*Y*34 H_2O
Mole fraction hydrate former	0.1481	0.1500	0.1500
Only large cages filled	X* 7 2/3 H_2O	X*17 H_2O	-
Mole fraction hydrate former	0.1154	0.0556	
Cavity Diameter (Å)			
Small	7.9	7.8	7.8
Medium	-	-	8.1
Large	8.6	9.5	11.2
Volume of Unit Cell (m^3)	$1.728 * 10^{-27}$	$5.178 * 10^{-27}$	
Typical Formers	CH_4, C_2H_6, H_2S, CO_2	N_2, C_3H_8, i-C_4H_{10}	See text
** where X is the hydrate former and Y is a type H former*			

7.2.3 THE DIFFERENT TYPES OF INHIBITORS

One of the methods used in the oil and gas industry to avoid formation of gas hydrates is adding suitable chemicals. This is a part of safe flow assurance, preventing plugging of pipes and securing other technical equipment on or attached to an oil/gas installation. The chemicals used for this purpose can be divided in two main categories: "Thermodynamic Hydrate Inhibitors" (THI) and "Low Dosage Hydrate Inhibitors" (LDHI). The last category is further divided into "Kinetic Hydrate Inhibitors" (KHI) and "Anti-Agglomerants" (AA). These categories of inhibitors will be described in more detail in sections below. [10]

7.2.3.1 THERMODYNAMIC HYDRATE INHIBITORS (THI)

THI is a chemical that is often referred to as an "antifreeze" product. This is due to its effect on the equilibrium properties for gas hydrate. THI lowers the temperature where hydrates are formed by pressure. This means that it is possible to work hydrate free under a lower temperature or higher pressure than without the THI. The equilibrium conditions are moved and the hydrate forming curve (seen earlier in figure 6), will thereby move to the left. [10]

THIs are the chemicals which are normally used to inhibit hydrate formation, even if they need to be added in high concentrations. Even though the concentration needed is high, it is vital that the dosage is correct at all times for the inhibition to work. The correct dosage can be calculated by use of the "Hammerschmidt equation" as shown below: [10]

$$\Delta T = [Ks / M (100-s)]$$

ΔT = hydrate suppression in ^{o}F
K = Hammerschmidt constant: 2,335
s = THI concentration
M = molecular weight of the THI

THIs are normally divided into these groups: alcohols, glycols and salt. In the oil and gas industry Monoethylene (MEG) and Methanol (CH_3OH) are most efficient in preventing the formation of gas hydrates. Triethylene Glycol (TEG) and Diethylene Glycol (DEG) are used for the same purpose, but these are not as effective as MEG and Methanol. Some different THIs are presented in table 2, ranked according to performance. In drilling fluids, salts like calcium chloride ($CaCl_2$), potassium chloride (KCl) and sodium chloride (NaCl) are most often used. [10]

Use of THI in an operation can cause several problems or concerns. Some of them are: THI can be lost into the liquid hydrocarbon phase and thereby create wax, or into the gas phase. There is also chance of scale formation. High amounts generate large transport costs and demand more storage space. Methanol is toxic and flammable, and can cause pollution of the environment. Corrosion can be an issue and fouling may occur in the regeneration. The THI can freeze in the pipelines or in processing systems. As a result, careful considerations must be made when use of THI is an option. [10]

Table 2: Overview of the most common THIs and their subcooling. [10]

Concentration (wt. %)	MeOH	EtOH	MEG	DEG	TEG	NaCl	HCOOK
5	2.0	1.4	1.05	0.63	0.46	1.96	
10	4.2	3.0	2.25	1.4	1.05	4.3	2.5
20	9.3	6.6	5.2	3.3	2.7	10.7	7.1
30	15.3	10.7	9.0	5.9	5.0	15.0	19.9
35	18.6	13.0	11.35	7.5	6.5	-	
40	22.2	15.4	14.0	9.3	8.2	-	

7.2.3.2 LOW DOSAGE HYDRATE INHIBITORS (LDHI)

LDHIs are hydrate inhibitors that are used in very low dosages to prevent hydrate formation. There are two kinds of LDHI; KHI (Kinetic Hydrate Inhibitor) and AA (Anti-Agglomerants). LDHI inhibition is a technology that has been developed during the past few years, and the main purpose is to find some good chemicals to perform the same level of gas hydrate inhibition as THIs, only in a much lower dosage. With LDHIs, only 0,1-0,2% by weight is needed, which means 40-80 times less than for THIs. Even though the price of LDHI is 20-30 times the price of THI, it will save money in the long run. With use of an LDHI, the amount is far less and this will decrease injection, transport and storage cost and there will be no need for regeneration. [14, 15]

7.2.3.2.1 KINETIC HYDRATE INHIBITORS (KHI)

KHIs are polymers that are low in molecular weight and also water soluble. To increase their effect, some small organic molecules are added. A KHI will delay nucleation and often crystal growth of the gas hydrate. This means that KHI perturb water structure and stops nucleation, and can also attach to hydrate surface and stop crystal growth. This depends on the pressure and the subcooling (normally with an optimum of 9-10^{o}C). This gives the advantage that gas can be transported from one location to another, e.g. from an offshore installation to an onshore gas processing plant, without any formation of gas hydrates in the pipelines on the way. On the other hand, if a static situation should occur, e.g. a shut down for a long period of time, THI will only be able to prevent/ delay gas hydrate formation for a limited period of time (this is often around 24 hours) and after that gas hydrates will grow again. Therefore, THI is not a good option if the process shuts down for more than 24 hours. THI is also not a good option in deep water drilling/ wells, since these have high pressure and subcooling. [10, 13]

KHI came on the marked in 1995 and has the benefit of being a low dosage hydrate inhibitor and is needed in concentrations of less than 1 wt%,-only 0,1-0,2 wt% is enough. This is a considerable reduction compared to THI, which is normally needed in concentrations of 20-60 wt%. As a result, cost of storage space, transportation and regeneration is reduced significantly if one can add KHI instead of THI. A down side of using KHIs is that they respond negatively in the presence of high concentrations of the gases CO_2 and H_2S. The cause of this has still not been established. [10, 13]

7.2.3.2.2 ANTI- AGGLOMERANTS (AA)

AA is a surface active chemical that is soluble in oil or water, and works by preventing particles from agglomerating into bigger particles and becoming gas hydrates. The hydrates are present in the solution, but only as small lumps, like a "slurry–like" solution. How the AA works is shown in figure 11. AA is a better chemical for preventing hydrates than KHI. This is because AA has a better subcooling than THI and is therefore also suitable for deep water operations. [10, 13, 14]

AAs can be divided in to two groups: [10]

- *Gas well AA*
- *Production or pipeline AA*

These are both common to use, and the main difference is that in a gas well AA, water must be added. This will not be described more in this thesis. [10]

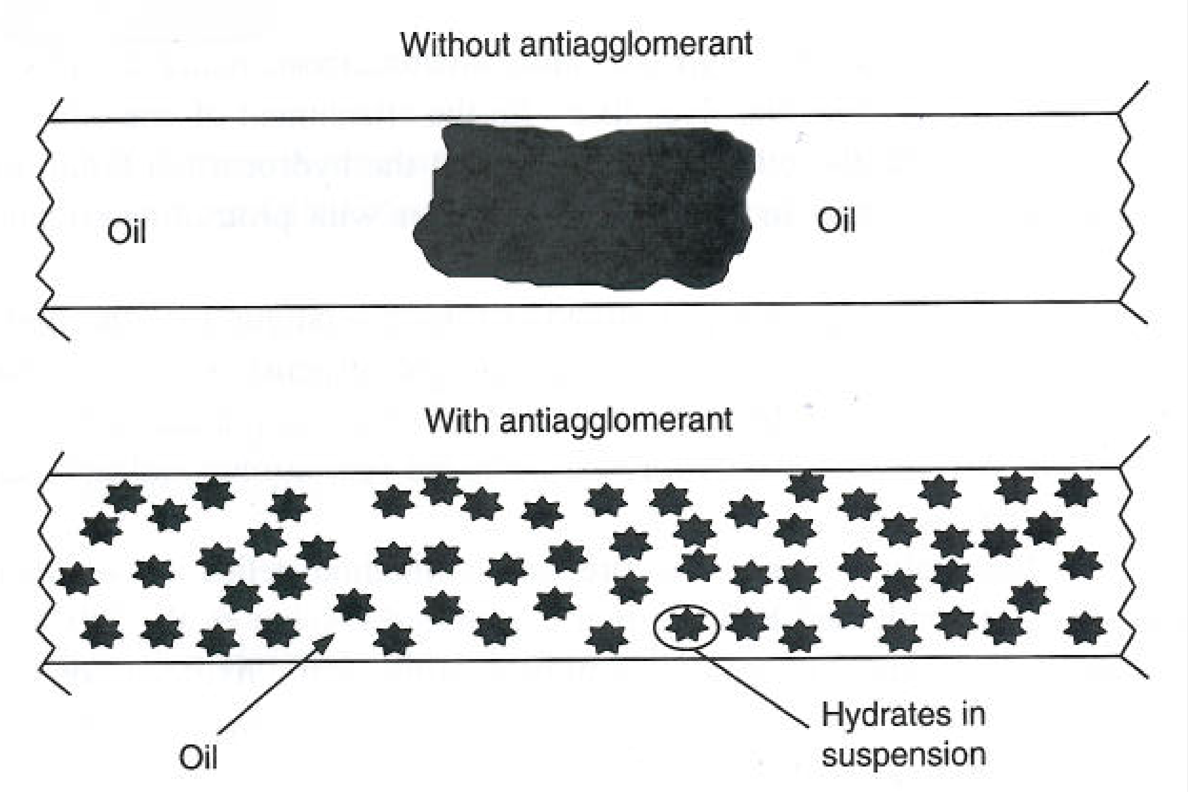

Figure 11: Description of the effect of adding AA. [13]

8 EXPERIMENTAL

8.1 DRILLING FLUID

All tests were performed according to standard procedures as given in the attachments in appendix 1.1.

Following formulations/samples were tested:

- Formulation no. 1: The base fluid
- Formulation no. 2: The base fluid + Luvicap 55W
- Formulation no. 3: The base fluid + RS-10475
- Formulation no. 4: The base fluid + RS-10419
- Formulation no. 5: The base fluid + MEG
- Formulation no. 6: The base fluid + MEG + RS-10475
- Formulation no. 7: The base fluid + MEG + NaCl
- Formulation no. 8: The base fluid + MEG + NaCl + RS-10475
- Formulation no. 9: The base fluid + CdId 161
- Formulation no. 10 The base fluid + CdId 161 in double concentration

A complete calculation sheet with the chemicals, dosage and test result of all these formulations (1-10) can be found in the attachments in appendix 1.2.

A short summary of the procedures is presented below.

8.1.1 MUD MIXING

The mud used during this experiment was mixed according to the formulation given in table 3 and the order of addition as listed in table 4. The mixing equipment was a Silverson mixer for water based mud (big holes in the mixing head). This base formulation was used throughout the entire project. Only minor alterations were made, depending on the addition of other products/chemicals. In addition, different inhibitors like Luvicap 55W (from BASF) which is a well known inhibitor from the industry, RS-10475, RS-10419, Cdld 161, MEG and NaCl were added in varying concentrations and combinations to the base mud. The inhibitors: RS-10475, RS-10419 and Cdld 161 are experimental products from internal and external suppliers and no structural data can be obtained at this point. These are listed in table 5 below. The mixing procedure and order of adding chemicals is important to follow because otherwise some additives may not be properly incorporated in the mud. If powders (viscosifiers/fluid loss agents etc) are added carelessly, lumps may form. If the mud becomes very “airy” during mixing, a few drops of defoamer should be added.

Table 3: Base formulation [5]

Product	Internal reg.	Concentration g/l	Mixing volume 3000ml
Sea water	MIN-08-443	954,1	2862,3 g
Duotec NS	MIN-08-269	4,0	12 g
Trol FL	MIN-09-049	15,0	45 g
Kla Stop (Ultrahib NS)	MIN-10-131	20,0	60 g
Barite	MIN-10-131	97,4	292,2 g
EMI-2223	EXP-10-154	10,0	30 g

Table 4: The order of adding the products in the formulation [16]

Order of addition	Mixing time in minutes
Sea water	1
Viscosifier (Duotec NS)	10
Fluid loss control additive (Trol FL)	10
Other liquid additives (Kla Stop/ Ultrahib NS)	5
Weighting agent (Barite)	20
EMI-2223	10

Table 5: The chemicals/inhibitors for inhibition of hydrates

Chemicals/inhibitors	ppm	g/l
Luvicap 55W	6000	12
RS-10475	6000	20,7
RS-10419	6000	14,3
MEG	6000	200
NaCl	6000	109
CdId 161	6000	6 and 12

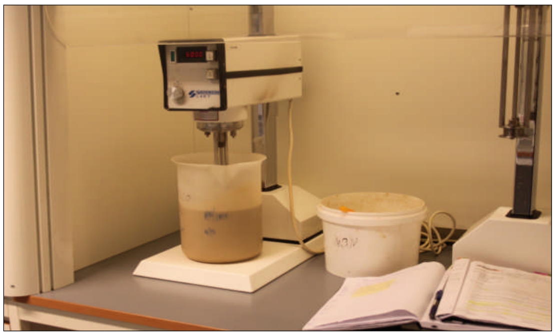

Figure 12: Mud mixing.

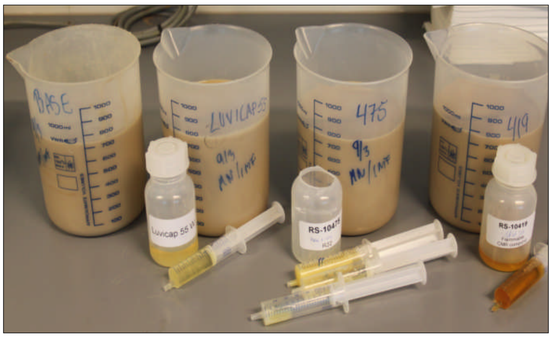

Figure 13: Mud after mixing and ready for addition of inhibitors.

8.1.2 pH

A pH-meter was used to measure the pH of all samples, and the same pH-meter was used for all samples tested. It was frequently calibrated to give the correct values. The pH was measured according to procedures as given in the attachments.

8.1.3 RHEOLOGY

To measure rheology a Fann 35 rheometer, figure 14, was used. Then 600, 300, 200, 100, 6 and 3 rpm readings could be recorded. The 10 second and 10 minutes gel were also recorded according to the provided procedures as given in the attachments. From calculations the plastic viscosity and yield point was found as shown in the formula below.

Figure 14: VG-meter (Fann 35).

PLASTIC VISCOSITY (PV) FORMULA [16]:

PV = 600 rpm – 300 rpm

YIELD POINT (YP) FORMULA [16]:

YP = 300 rpm - PV

8.1.4 DYNAMIC & STATIC AGEING

The rheology of the fresh mixed mud is measured, without the 10 second and 10 minute gels. The mud is then transferred to steel ageing cells and put in a dynamic oven for 16 hours (ageing). This is done to simulate what happens with the mud when circulated during a drilling operation (figures 15 and 16). After 16 hours of dynamic ageing the cells are taken out of the oven and cooled down. Rheology of the aged mud is then measured, including 10 second and 10 minute gels.

Ageing temperature was set to 100°C throughout the entire project. The samples for static ageing are first aged 16 hours dynamically, and then transferred directly to a static oven for another 24 hours of ageing. This is done to simulate what happens with the mud under down time, when drilling stops. Rheology is measured both before and after ageing, to see if the properties of the mud have changed during ageing.

Figure 15: Dynamic ageing.

Figure 16: Static ageing.

8.1.5 API FLUID LOSS

API (American Petroleum Institute) fluid loss is a test which gives an indication on how much of the mud will be lost to the formation. It also gives an indication on whether the filter cake is good or not (thin/thick or firm/loose). In short, mud is filled in 1/3 of the API cell, and with help of pressure (100 psi) the fluid is pushed through a filter in the bottom, see figure 17. The test duration is 30 minutes and the fluid is collected in a measuring cylinder. The total API fluid loss is recorded and the filter cake thickness is measured as shown in figure 18.

Figure 17: The API fluid loss equipment set up.

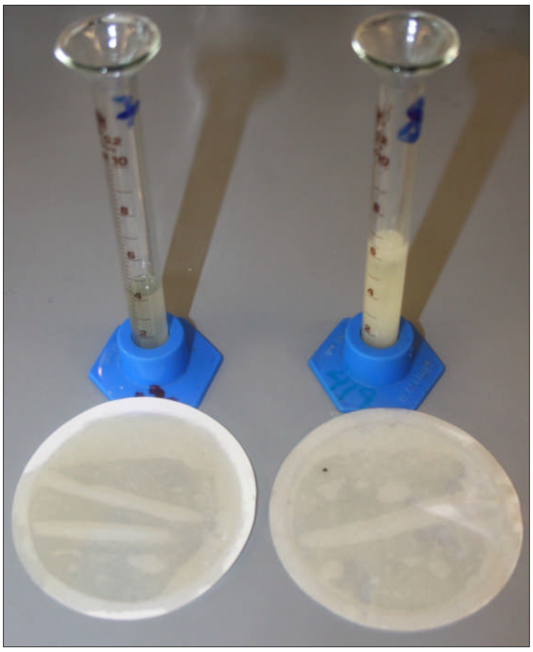

Figure 18: The collected filtrate and the filter cake.

8.1.6 STATIC SAG TEST

Mud is mixed and aged as explained above; 16 hours dynamic aging followed by 24 hours (or any required length of time) static aging. The cell is then taken out of the oven and cooled. The free fluid on top goes in one cup; the rest of the fluid is divided into 5 different cups. From the cups containing the top, middle and bottom layers, 20 ml of fluid is taken out and the specific weight of the three layers is measured by use of a pycnometer, as shown in figure 19. This is to find the density of the mud in these sections and thus find the Sag Index. Sag Index will tell whether or not the mud will separate and settle during down time in an offshore operation. The Sag Index can be found as shown in the calculations below.

CALCULATIONS [16]:

A = Volume of aged mud (ml)

B = Free fluid on top (ml)

MW= mud weight of drilling fluid

Free fluid on top (V / V %) = (B* 100) / A

Sag Index = SG bottom / (MW * 2)

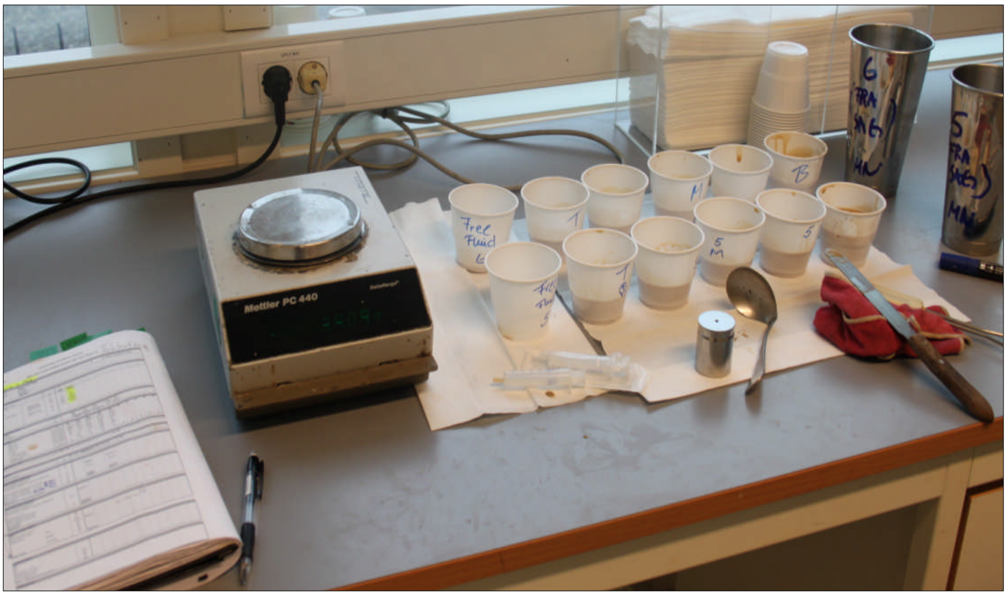

Figure 19: Static sag test.

8.1.7 SIFTING AND CHRUSHING CUTTINGS

To have the correct size of clay needed for the dispersion and accretion tests, sifting and crushing of clay was necessary. Two sizes of sieves were used: 4 mm and 6,3 mm. A large amount of clay was crushed to have enough from the same batch to use in all the testing. The clay was sifted and crushed according to the procedure in the attachments.

8.1.8 HOT ROLL DISPERSION TEST

The test starts with adding mud (350 ml) and 30 g of Arne Clay (cuttings) to a cell, as shown in figure 20 below. Then the cell is dynamically aged for 16 hours. Afterwards the cuttings are separated from the mud by using two different size sieves, and cleaned using 10 % KCl solution, as shown in figure 21. The two different sieves give two different sizes of wet cuttings. These are measured and placed in an oven for drying at a temperature of 105°C and reweighed when dry. Recovery and moisture of the sample can be calculated from the formulas given below. Then, after being exposed to clay/cuttings, pH and rheology including gels is measured to check whether the properties of the fluid have been affected.

CALCULATIONS [16]:

To find the moisture content and % recovery from the samples, these calculations are used:

Correction factor = B/A
% Moisture of recovered sample = $[(W_3 – W_4) / ((W_3 – W_2) * 100]$
% Recovery = $[(W_4 – W_2) / ((W_1 – C) * 100]$

Where: [16]
A = Initial weight of cuttings (determination of natural moisture content of shale).
B = Final dried cuttings
C = Correction factor
W_1 = Initial weight of cuttings used
W_2 = Empty dish weight
W_3 = Total dish and “wet” sample weight after hot rolling
W_4 = Total Dish and “dry” sample weight (after drying to constant weight)

Figure 20: Ageing cells.

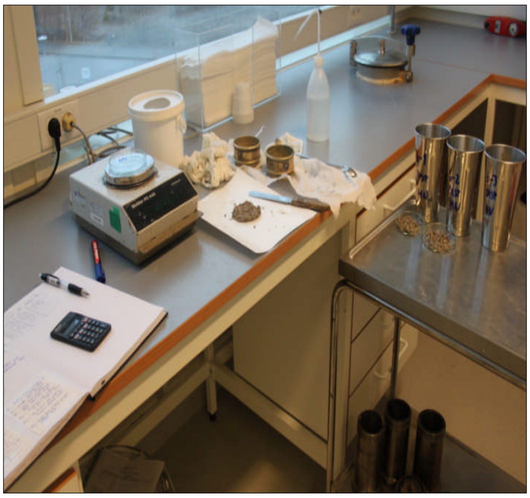

Figure 21: Dispersion test- weighing wet clay.

8.1.9 ACCRETION TEST

Accretion is the mechanism by which partially hydrated cuttings stick to part of the bottom hole assembly and accumulate as a compacted layered deposit. The test starts with mixing mud, which is then added to plastic bottles (350 ml) with clean hollow steel cylinders inside (figure 22). 30 g of Arne clay is added to each bottle and then placed in a cold dynamic oven and aged for 10 minutes (figure 24). After this, the steel cylinder is taken out, carefully rinsed with water and the cuttings/clay attached (figure 23) is transferred to a glass dish and weighed (figure 25). Then the cuttings are placed in an oven at 105^{o}C for drying and the dry weight is recorded. This test simulates how much of cuttings/clay that will attach to the drill string, and from calculations as shown below it is then possible to find the percent accretion and the percent moisture.

CALCULATIONS [16]:

To find the correction factor, clay is weighed and then dried and weighed again. The correction factor may then be found as shown below:

Correction factor = Final weight / Initial weight

% Accretion= [Final Wt of cuttings / (Initial Wt of cuttings * Correction factor)]* 100

% Moisture = [(Wet Wt – Final Wt of cuttings) / Final Wt of cuttings] * 100

Figure 22: Mud/steel cylinder in the plastic bottles.

Figure 24: 10 minute cold ageing.

Figure 23: Clay attached to steel cylinders.

Figure 25: Wet clay ready to be dried.

8.2 GAS HYDRATE TESTING (GHT)

8.2.1 THE PROCEDURE FOR GAS HYDRATE TEST (GHT)

This is a procedure under development by MI-SWACO, A Schlumberger Company. The equipment for this method is a Physica rheometer which measures viscosity and pressure changes as its main parameters. It is connected to a computer, and high temperatures (approximately 191 bar) and low temperatures (-1°C) are used.

Gas hydrate formation will show as increase in viscosity and decrease in pressure, and gas hydrate melting will have opposite effect; decreasing viscosity and increasing pressure. The temperature is controlled during the test.

The Gas Hydrate Test (GHT) was performed as described below:

1. Ensure XL-cell and CC35.29 measuring system is installed and calibrated.
2. Boost pressure up to 200 bar in the gas cylinder.
3. Add 10 ml of sample to be tested to the measuring cell.
4. Mount and tighten the top (cylinder) of the measuring system.
5. Attach the gas outlet to the cell, fasten well.
6. Flush with Green Canyon Gas (GCG, the composition is shown in figure 6 below) 3 times, to ensure no air is left in the cell.
7. Boost as necessary to 190 bar.
8. Mount magnet head and move into measuring position (N = ± 0,05)
9. Start test, wait until a start pressure (P) of 190- 192 bar is recorded (1-2 points). If P ≠ 190-192 bar, adjust pressure and restart to achieve P = 190 – 192 bar.

Table 6: Green Canyon Gas Composition, according to NORSOK standard.

Components	Quantity	Unit	Relative uncertainty %
Ethane	7.6	%	± 0.5 %
Propane	3.1	%	± 0.5 %
Isobutante	0.2	%	± 5 %
n-butane	0.8	%	± 1 %
Isopentane	0.2	%	± 5 %
n-pentane	0.4	%	± 5%
(balance) Methane	87.5	%	± 0.2 %

Test conditions:

- $\gamma = 130\ s^{-1}$ (rotation/speed)
- T = - 1°C, 4 hours hold time
- P= 190-192 bar

Intervals:

1. 10 min at 20°C
2. Heat to 80°C
3. 80°C for 30 min
4. Cool to 20°C quickly
5. Cool to -1,5°C slowly (0,04°C/min)
6. -1°C, 4 hours
7. Heat to 20°C slowly (0,04/°C min)
8. 10 min at 20°C

When finished, release pressure carefully. Dismount cell and clean properly. Save result at desired location.

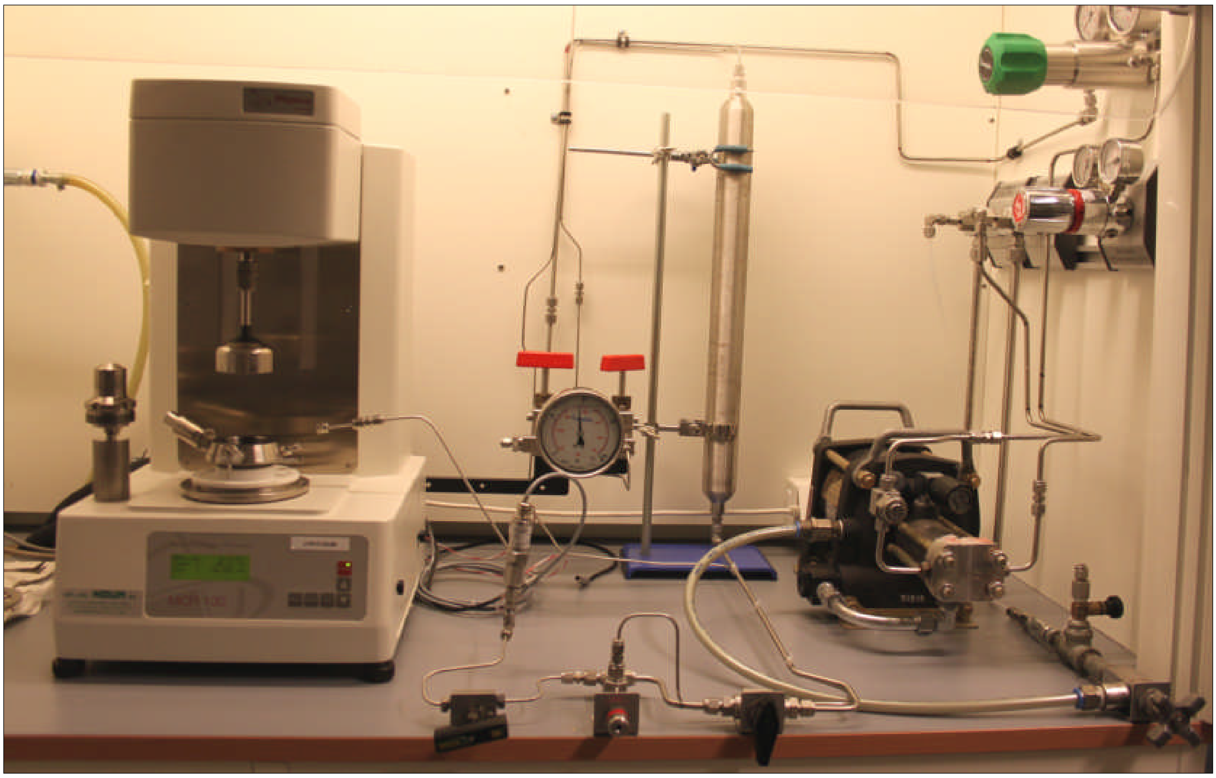

Figure 26: The setup of the gas hydrate test equipment rig.

9 RESULTS

9.1 DRILLING FLUID PROPERTIES

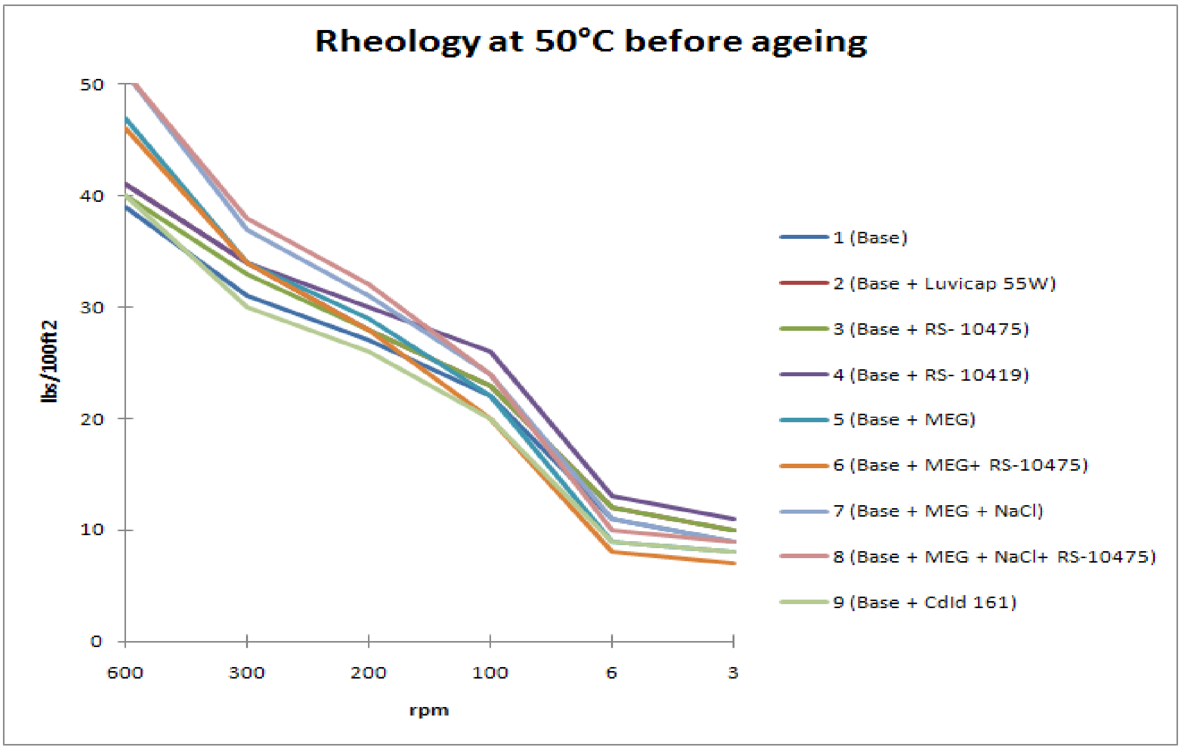

Figure 27: Rheology at 50°C before ageing.

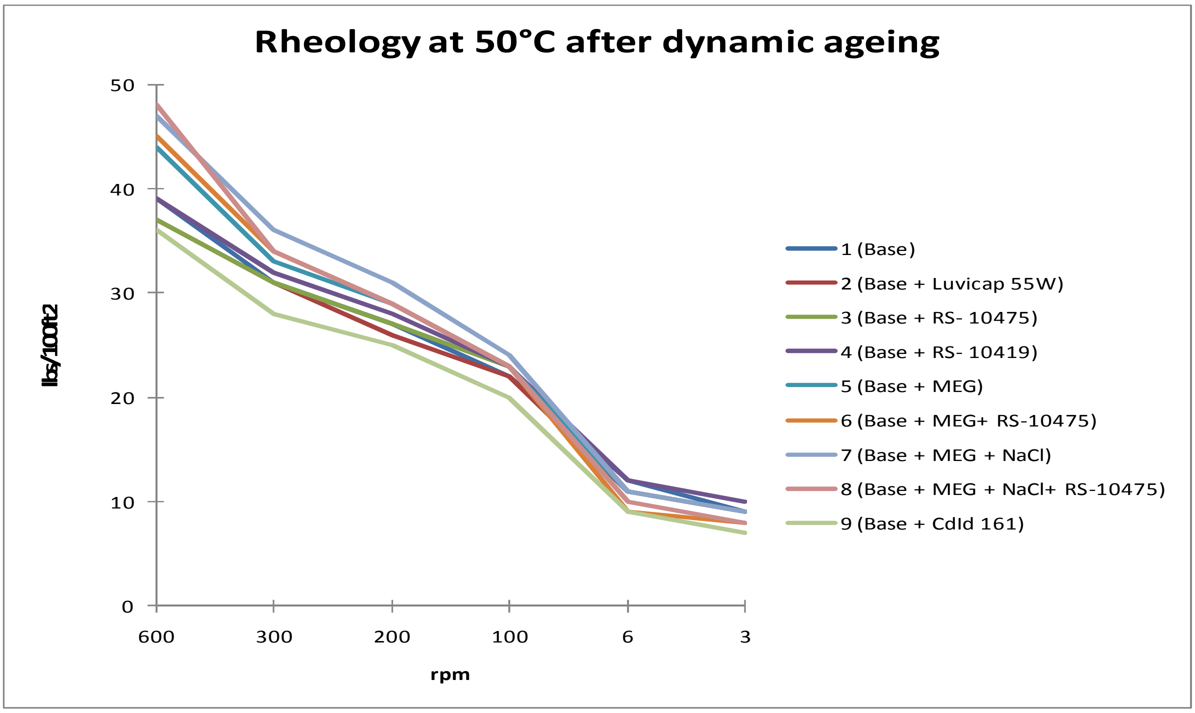

Figure 28: Rheology at 50ºC after dynamic ageing.

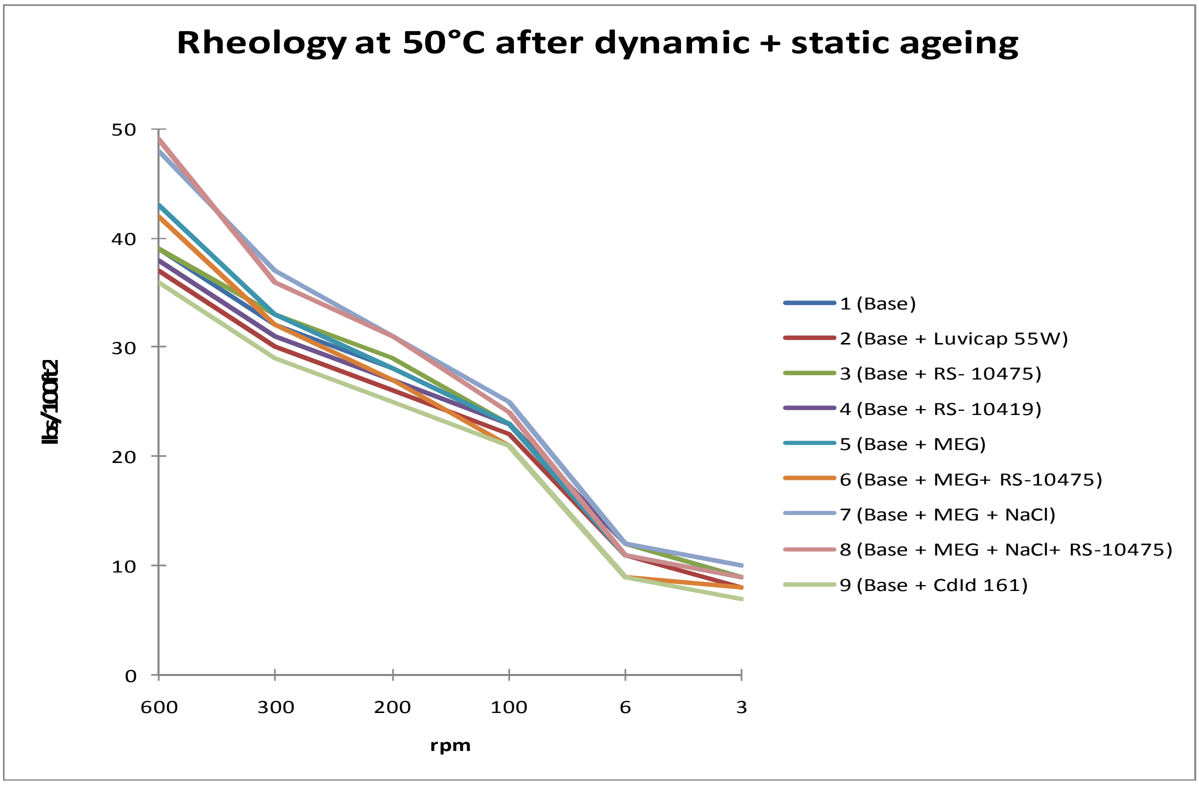

Figure 29: Rheology at 50ºC after dynamic + static ageing.

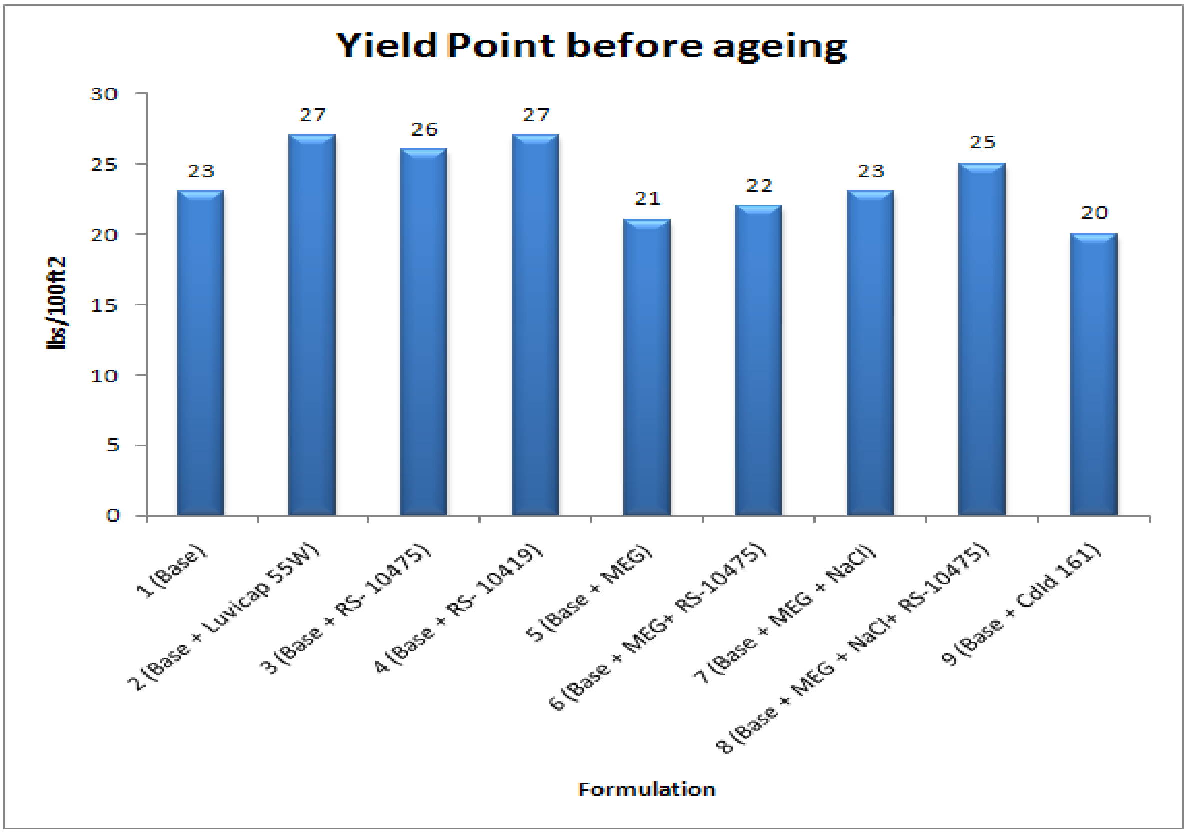

Figure 30: Yield Point before ageing.

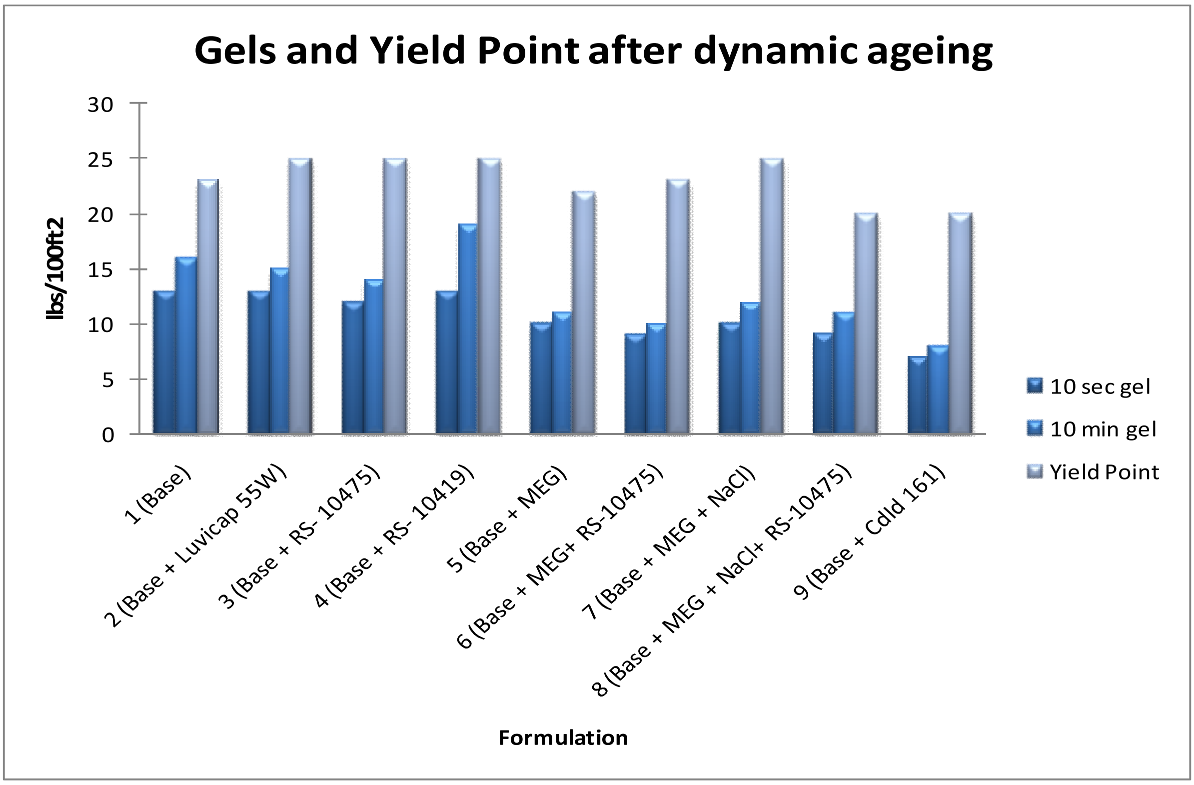

Figure 31: Gels and Yield Point after dynamic ageing.

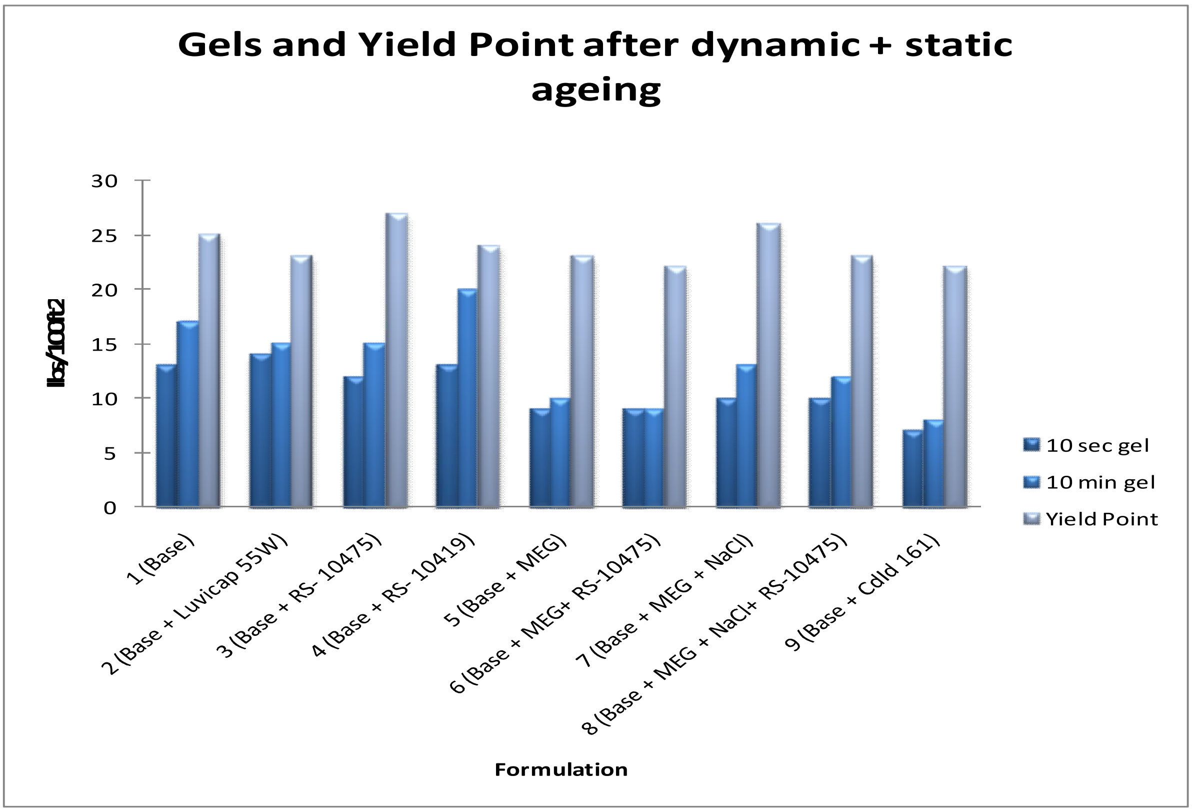

Figure 32: Gels and Yield Point after dynamic + static ageing.

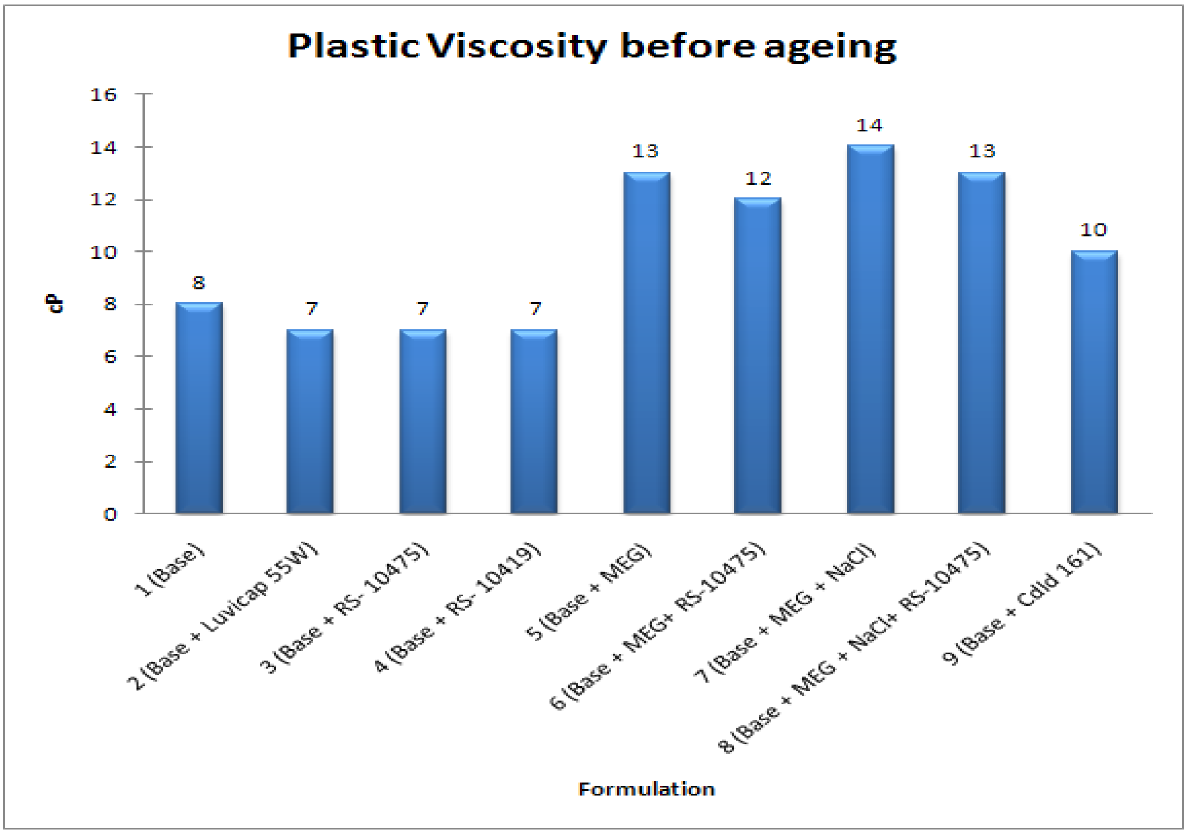

Figure 33: Plastic Viscosity before ageing.

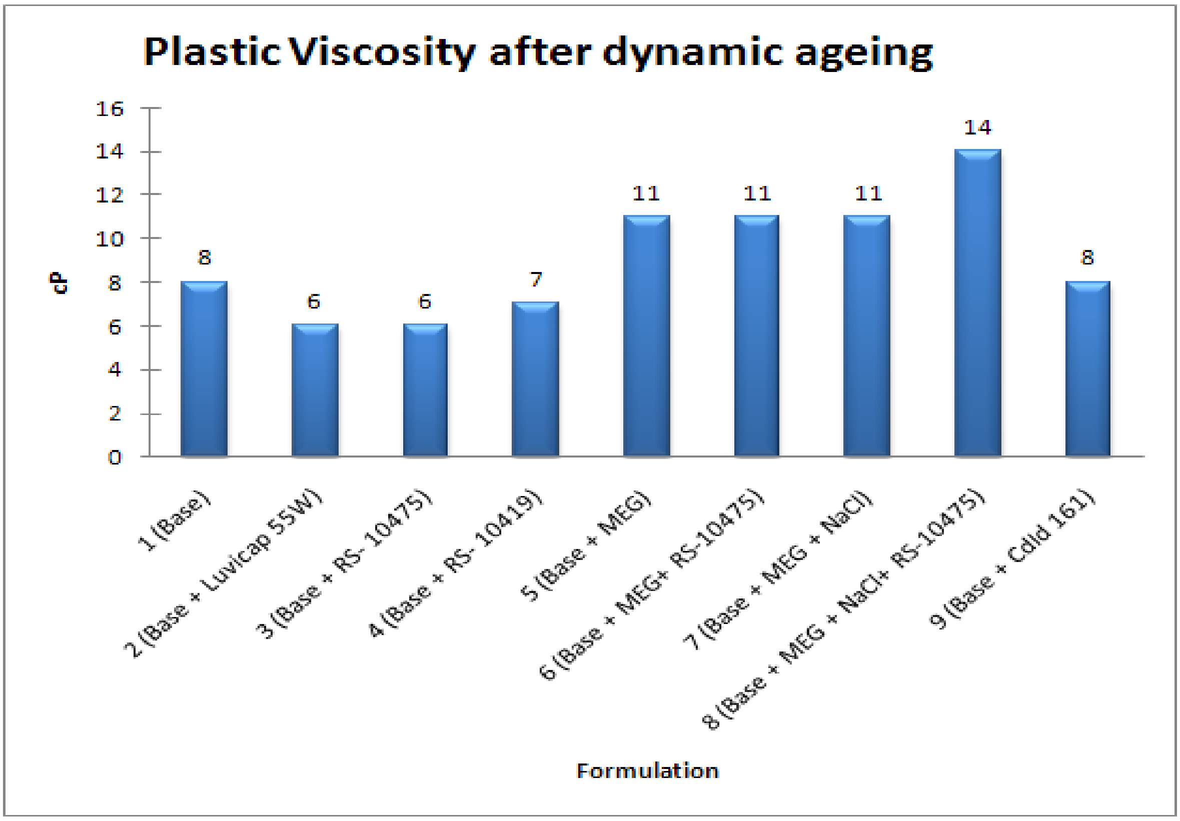

Figure 34: Plastic Viscosity after dynamic ageing.

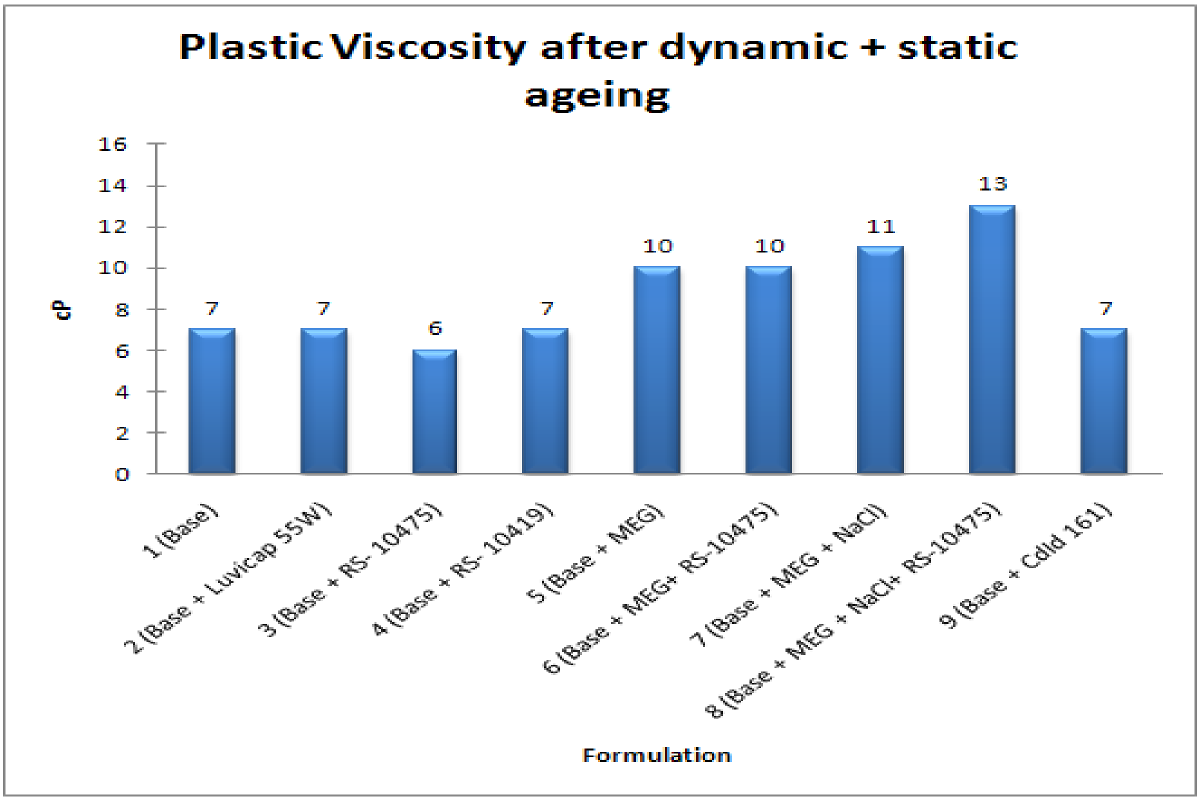

Figure 35: Plastic Viscosity after dynamic + static ageing.

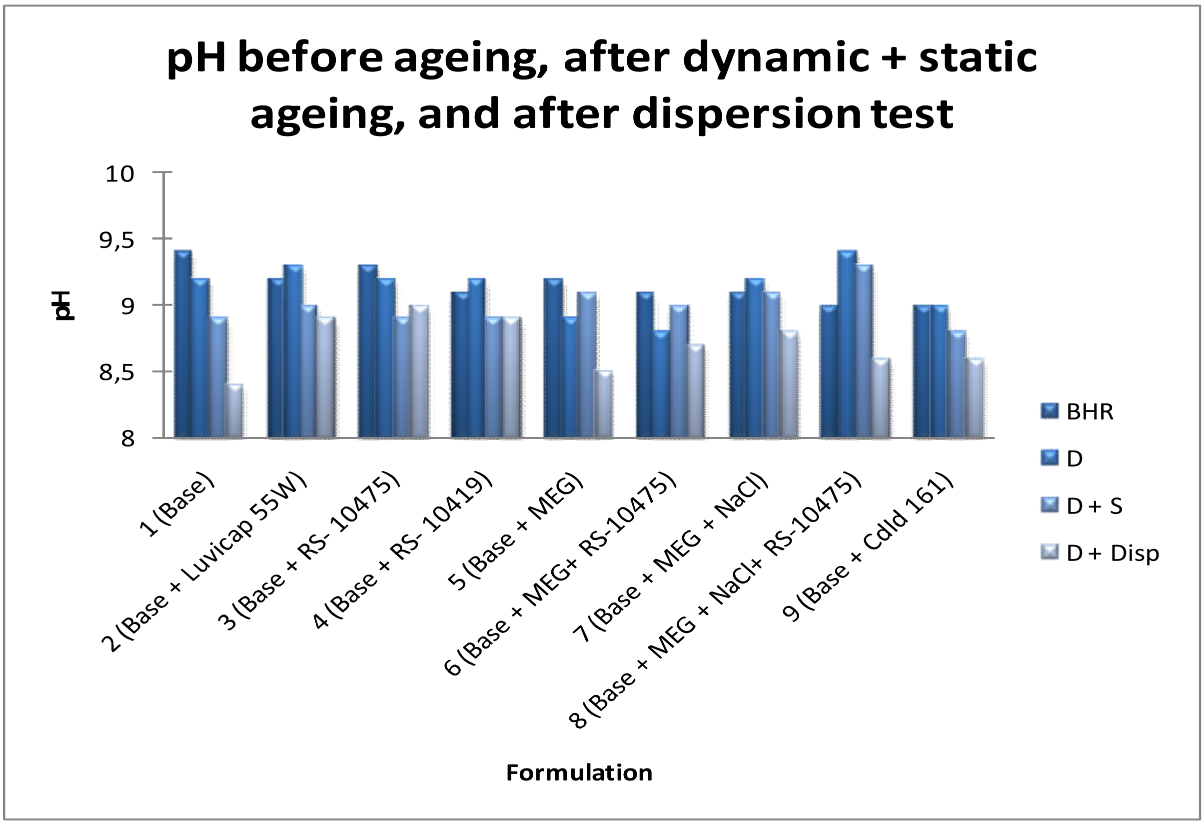

Figure 36: pH before ageing, after dynamic + static ageing, and after dispersion test.

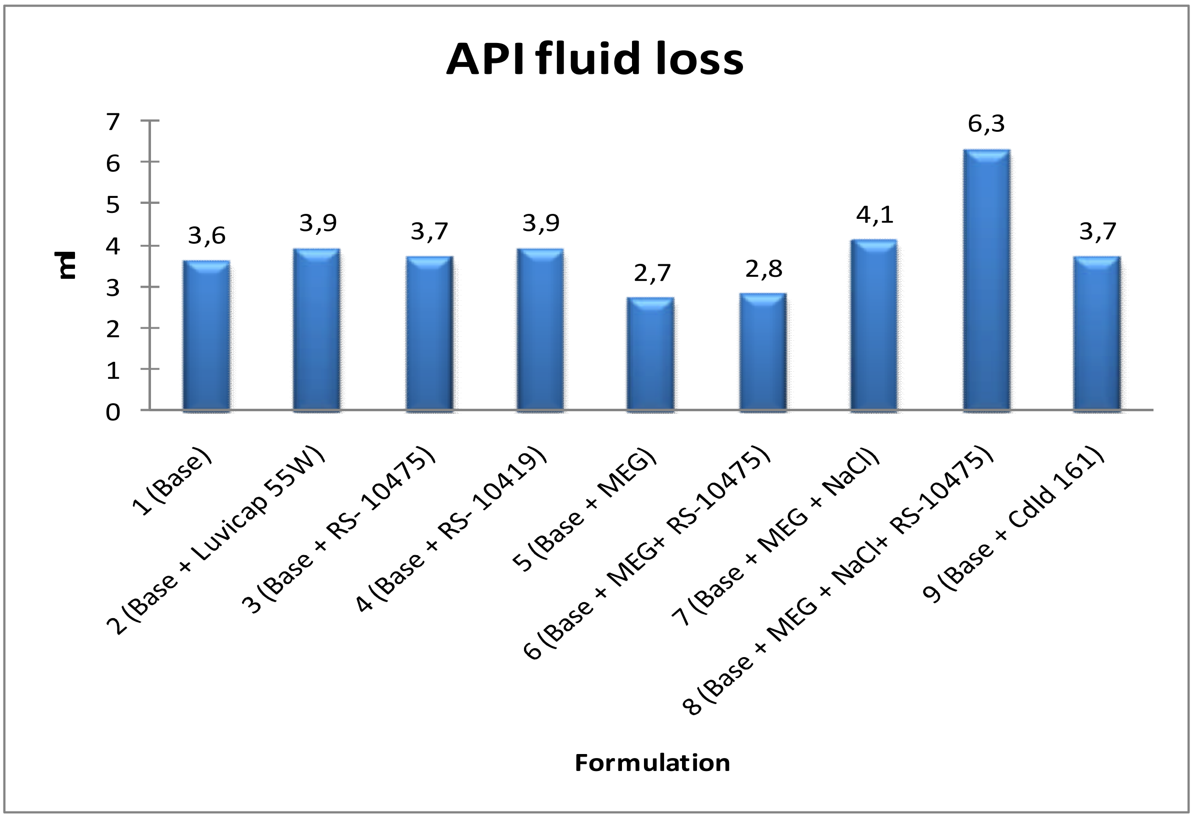

Figure 37: API fluid loss results.

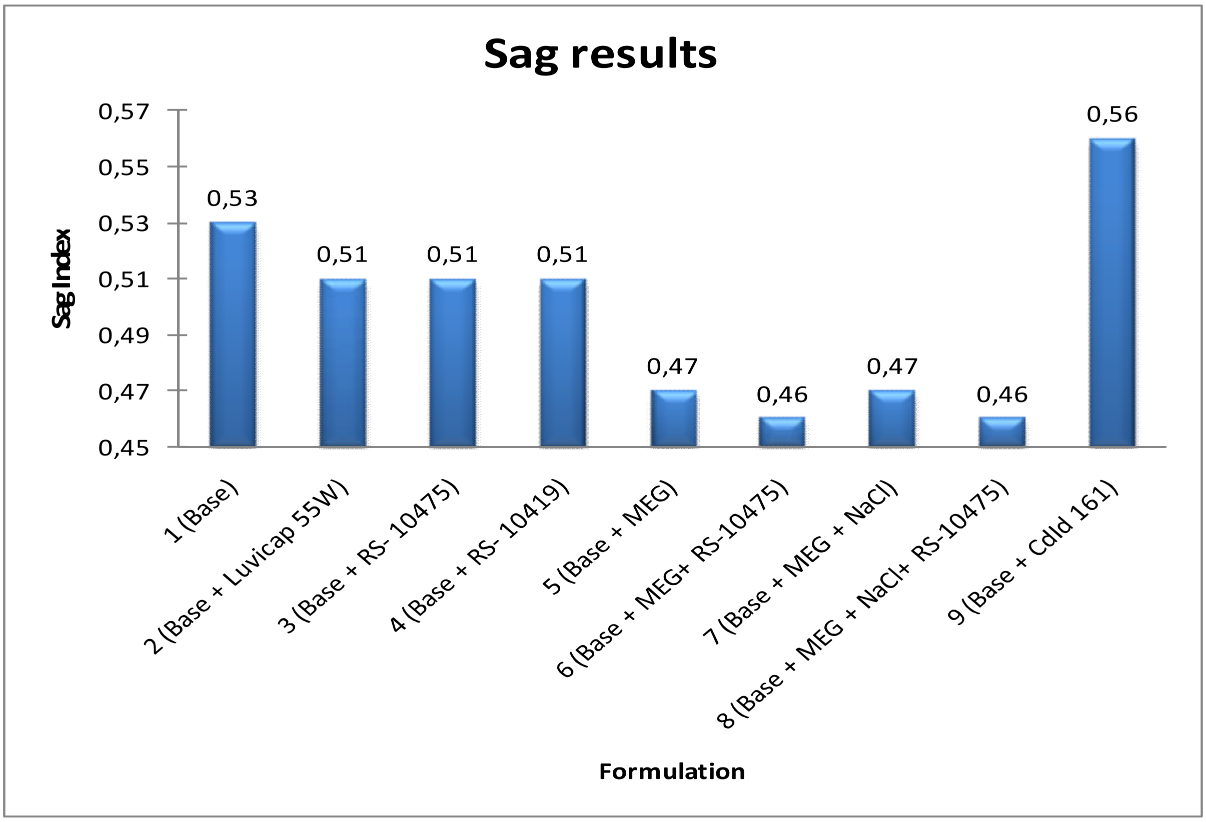

Figure 38: Sag test results.

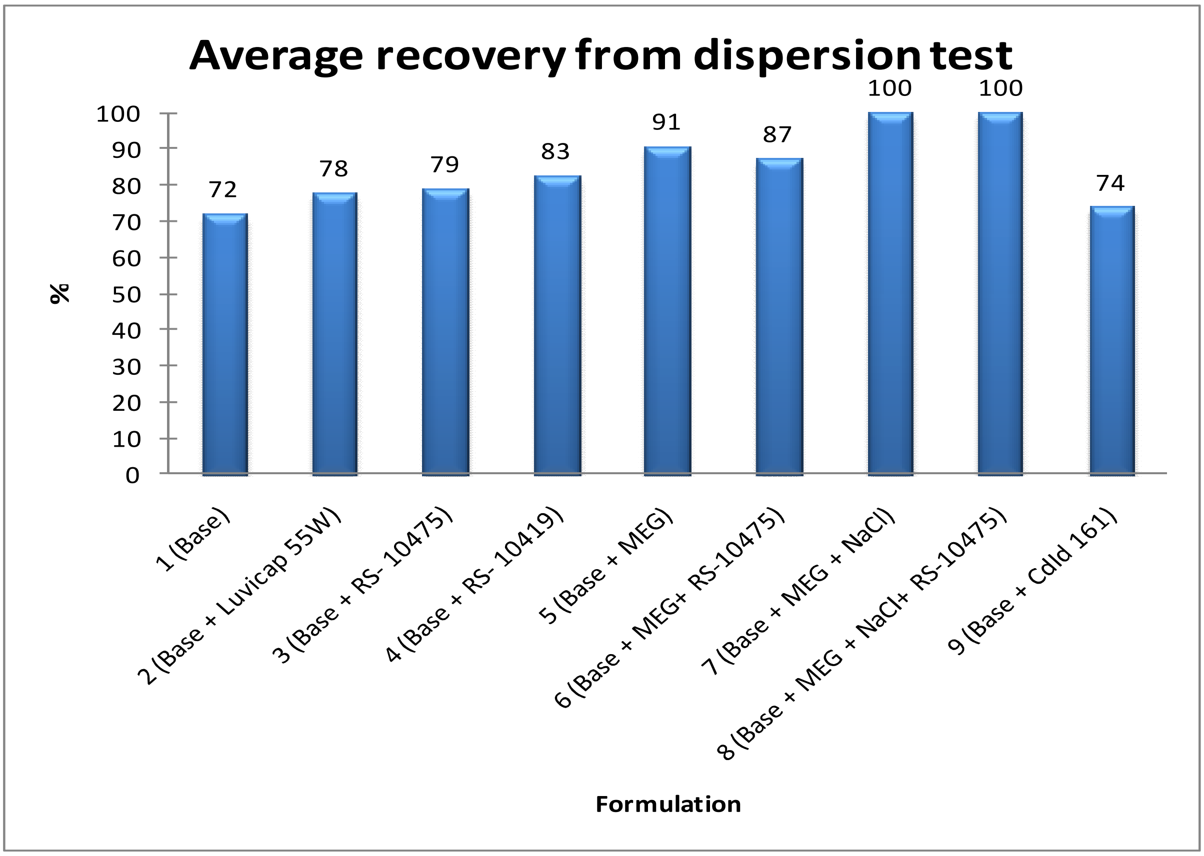

Figure 39: Average recovery from dispersion test.

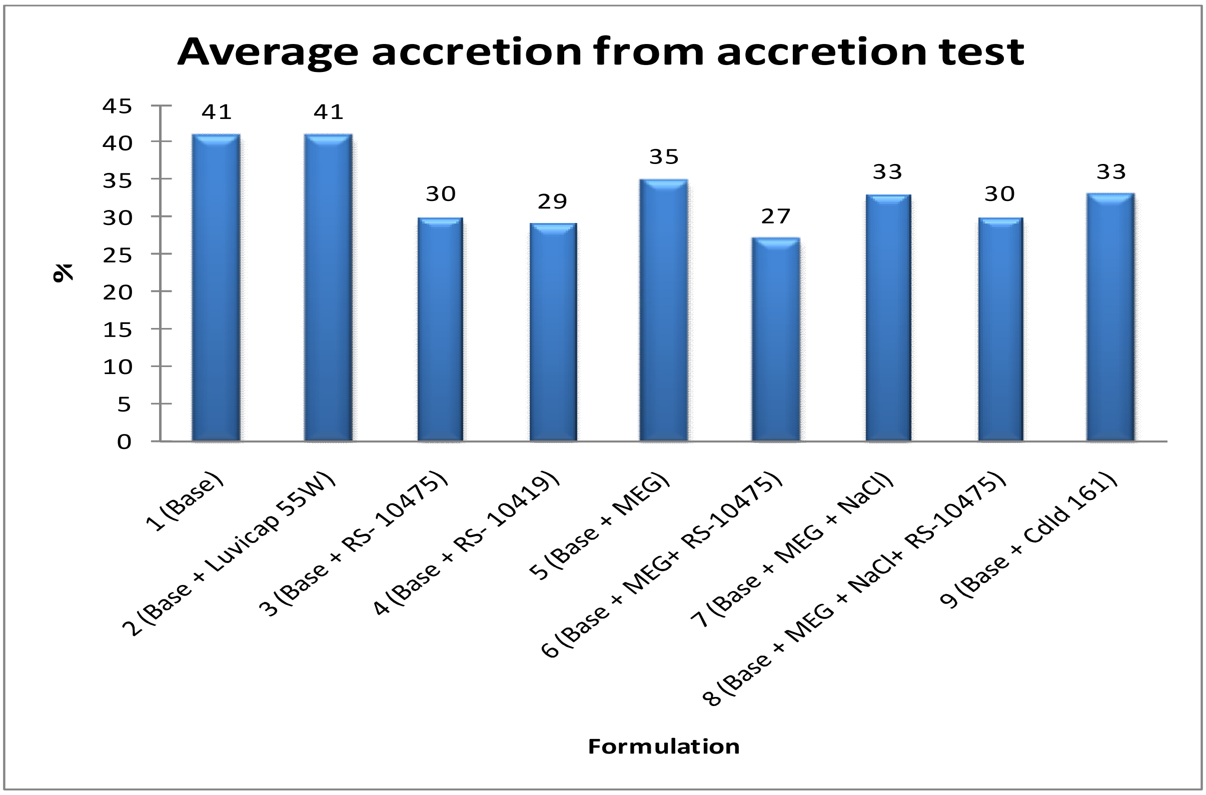

Figure 40: Average accretion from accretion test.

9.2 GAS HYDRATES

The following presentation of the results from gas hydrate testing (GHT) will contain two graphs for each sample tested. The first is presented with size 4*10^{13} cP in the y- axis and the next one will be presented with size 1000 cP in the y-axis (zoomed view). This is to be able to see the relation and what really happens. The formulations that were evaluated as acceptable based on the drilling fluids tests and taken further to the GHT were as follows:

- Formulation no. 1: The base fluid (reference sample)
- Formulation no. 3: The base fluid + RS 10475 (KHI)
- Formulation no. 7: The base fluid + MEG + NaCl (THI)
- Formulation no. 9: The base fluid + CdId 161 (AA)
- Formulation no. 10: The base fluid + CdId 161 in double concentration (AA)

The reason for selecting these formulations was their properties shown in all the standard WBDF tests. The base fluid obviously had to be tested, since all the other formulations are compared to this one. Formulation number 3 and 9 had acceptable results from the standard tests. Formulation number 9 also had a positive result overall and was selected for GHT. It was also decided to increase the concentration of CdId 161 (AA) in formulation 10 to the double to see if this gave a better inhibition effect.

All the formulations tested were tested in 3 parallels. All parallels are showing in the same graph, only with different colors.

This testing of gas hydrate is as mentioned in the experimental part, a method under development. The equipment used is a Physica rheometer, which measures viscosity during a certain pressure at a controlled temperature. When gas hydrate formation occurs the viscosity will increase and the pressure decrease. The temperature is controlled and thus the same for all parallels, which is the reason for the temperature curves for the parallels showing only as one line. (All the temperature parallels will be logged at the same position and will therefore show as only one.) When the melting of the gas hydrate happens, the viscosity will decrease and the pressure increase again. The formation and the melting process can not only be seen by increasing and decreasing viscosity, but also as at little "drop" in the pressure curve.

In addition to the gas hydrate test a WHYP Predicted hydrate temperature/pressure curve was made. This was done to have some idea about the subcooling temperature and how a gas hydrate temperature/pressure curve would be before the testing. The curve was made for these selected fluids:

a) Distilled water (Predicted hydrate formation temperature) : 22.2ºC
b) Base fluid (Predicted hydrate formation temperature): 22.2ºC
c) Base + MEG + NaCl (Predicted hydrate formation temperature) : 6,7ºC

Where the predicted hydrate formation temperatures are based on a 190 bar pressure. The pressure /temp. curves are given in figure 41 below.

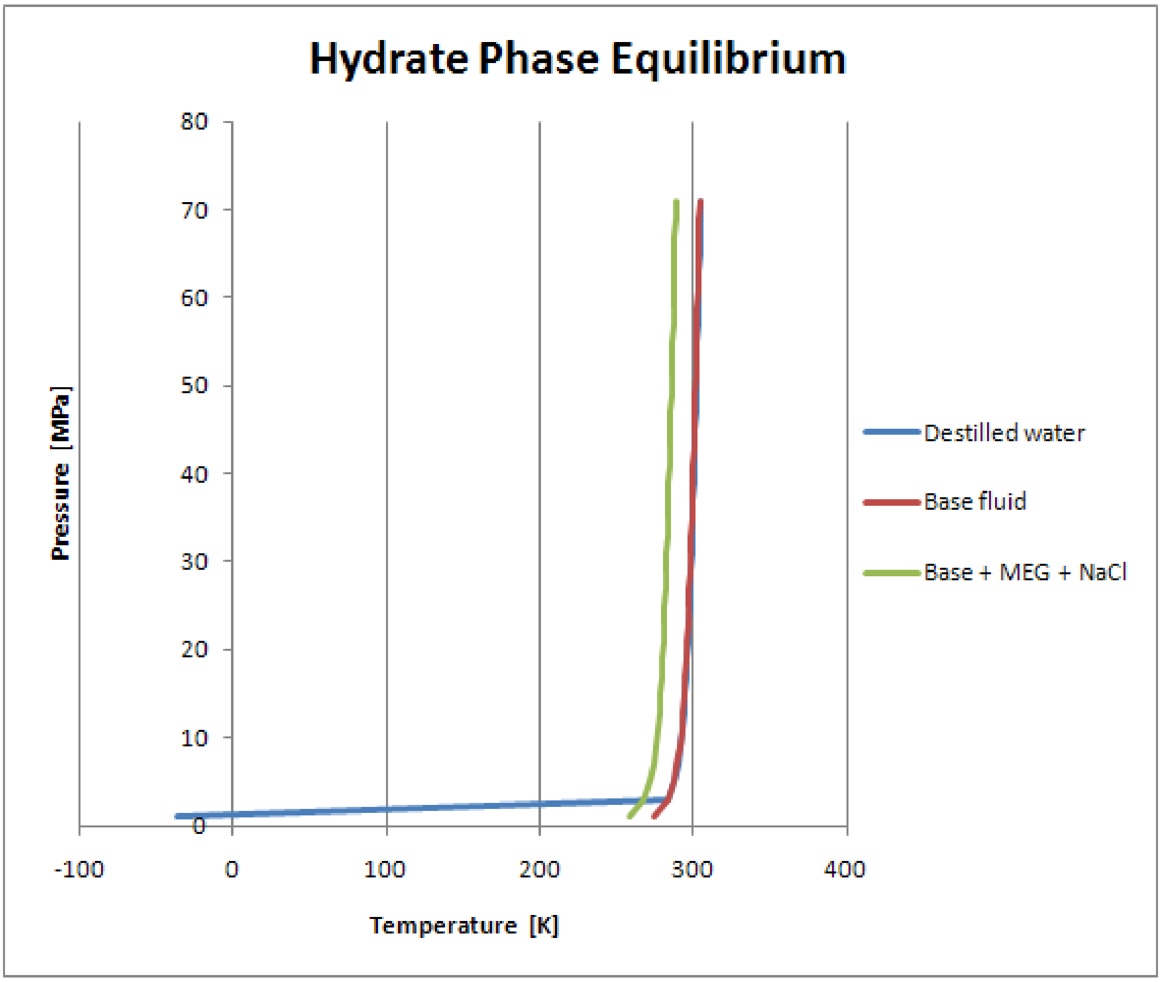

Figure 41: Hydrate phase equilibrium curve.

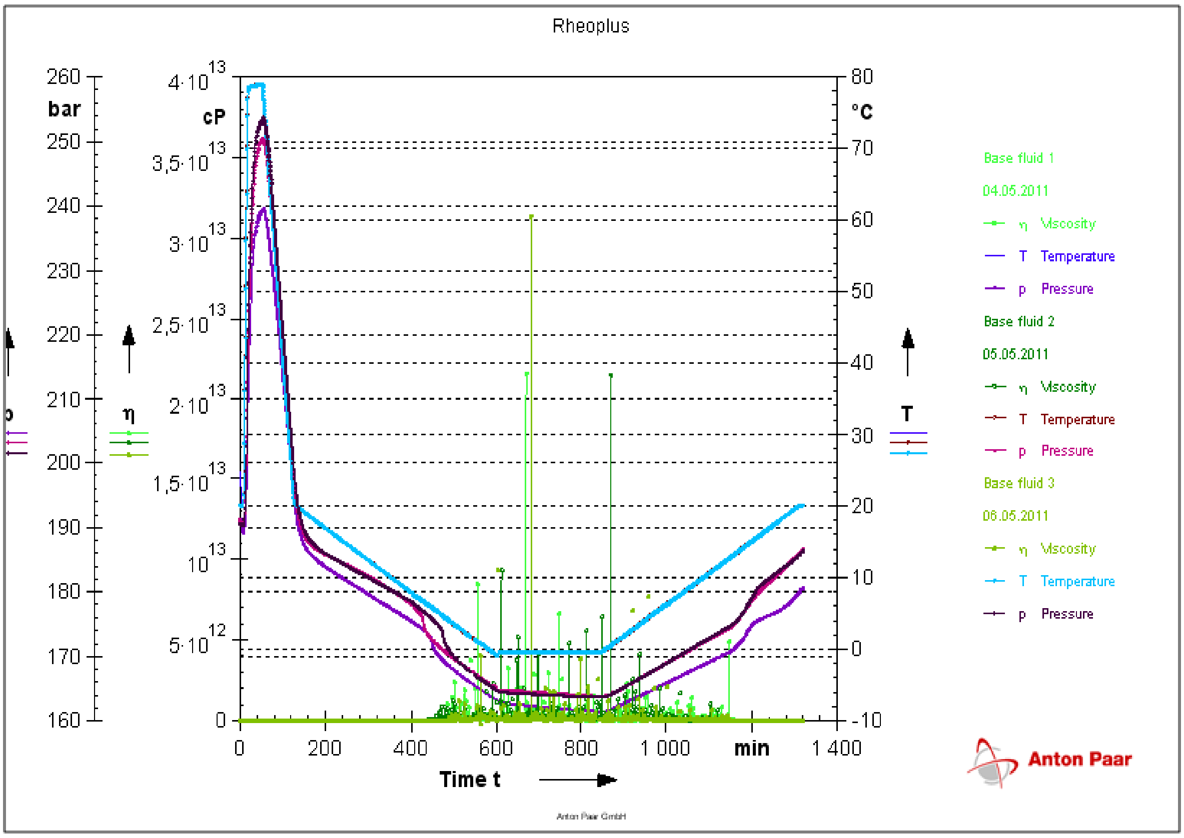

Figure 42: Formulation 1, Base fluid, with viscosity range cP to $4*10^{13}$.

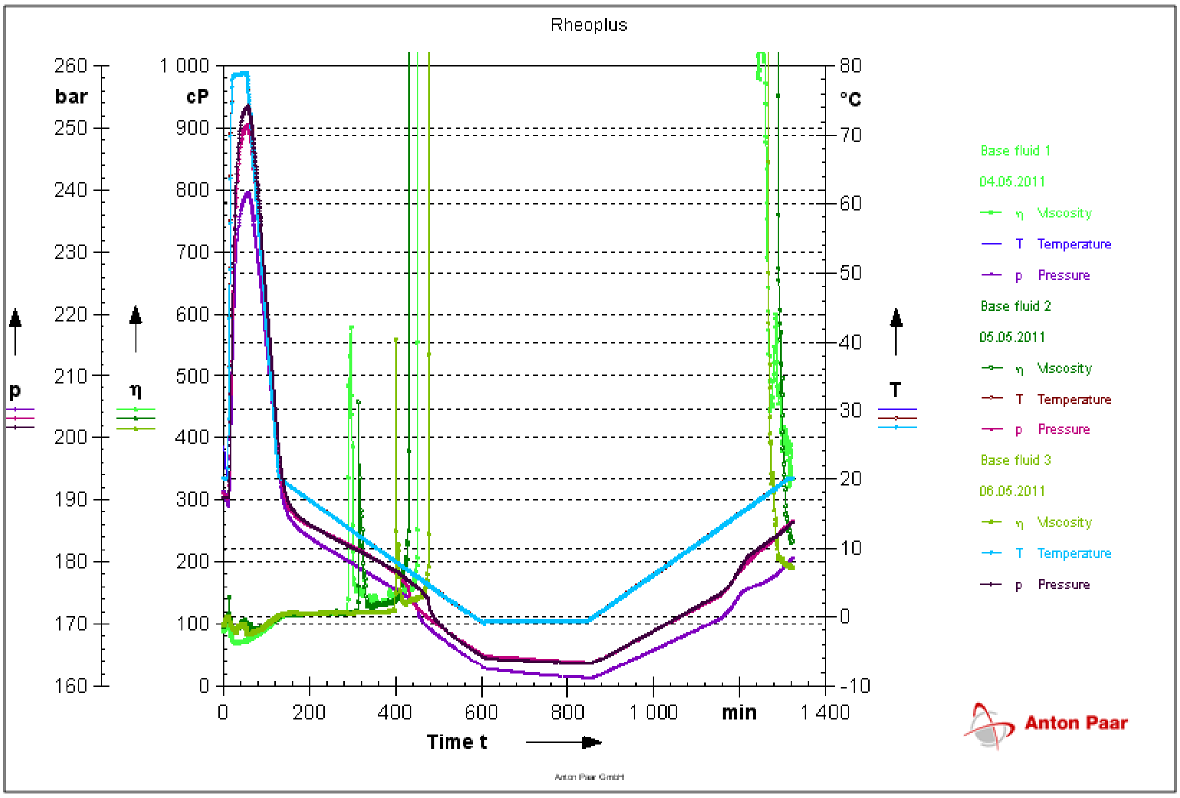

Figure 43: Formulation 1, Base fluid with viscosity range at cP to 1000.

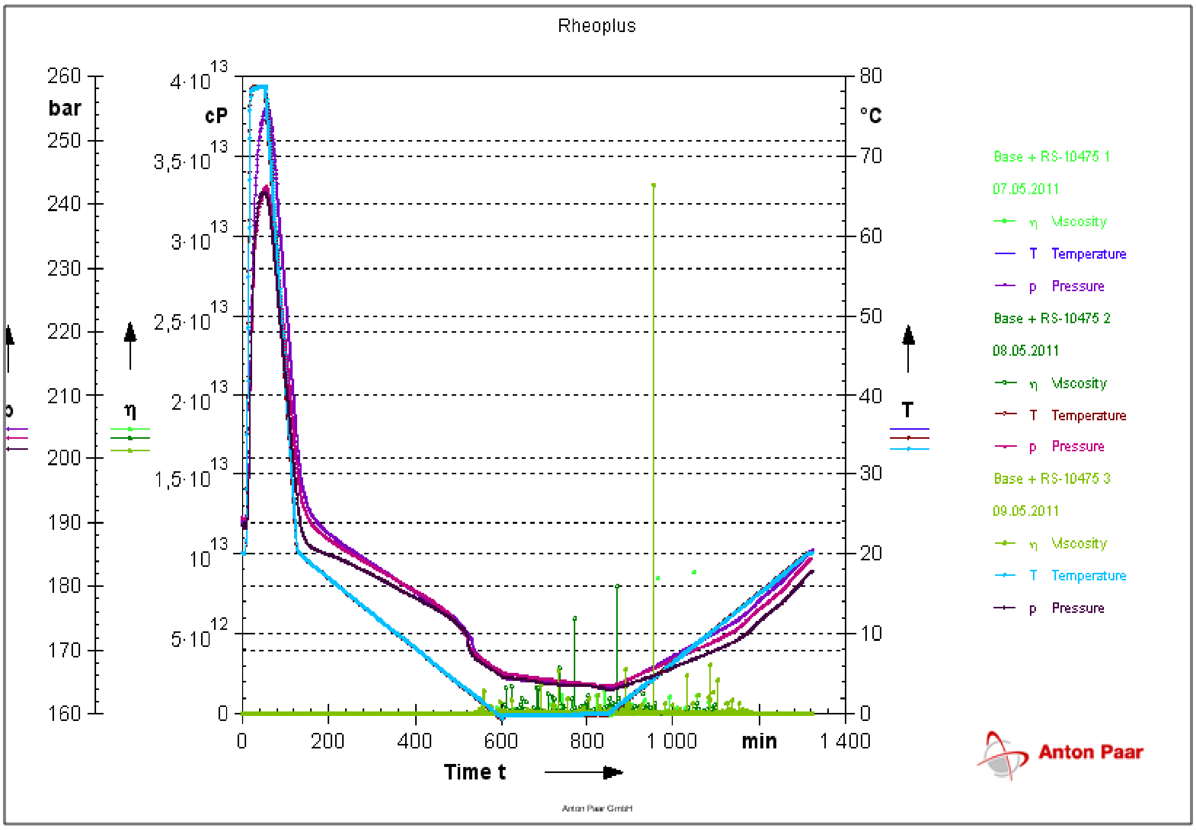

Figure 44: Formulation 3, base fluid + RS 10475 with viscosity range cP to 4*10^{13}.

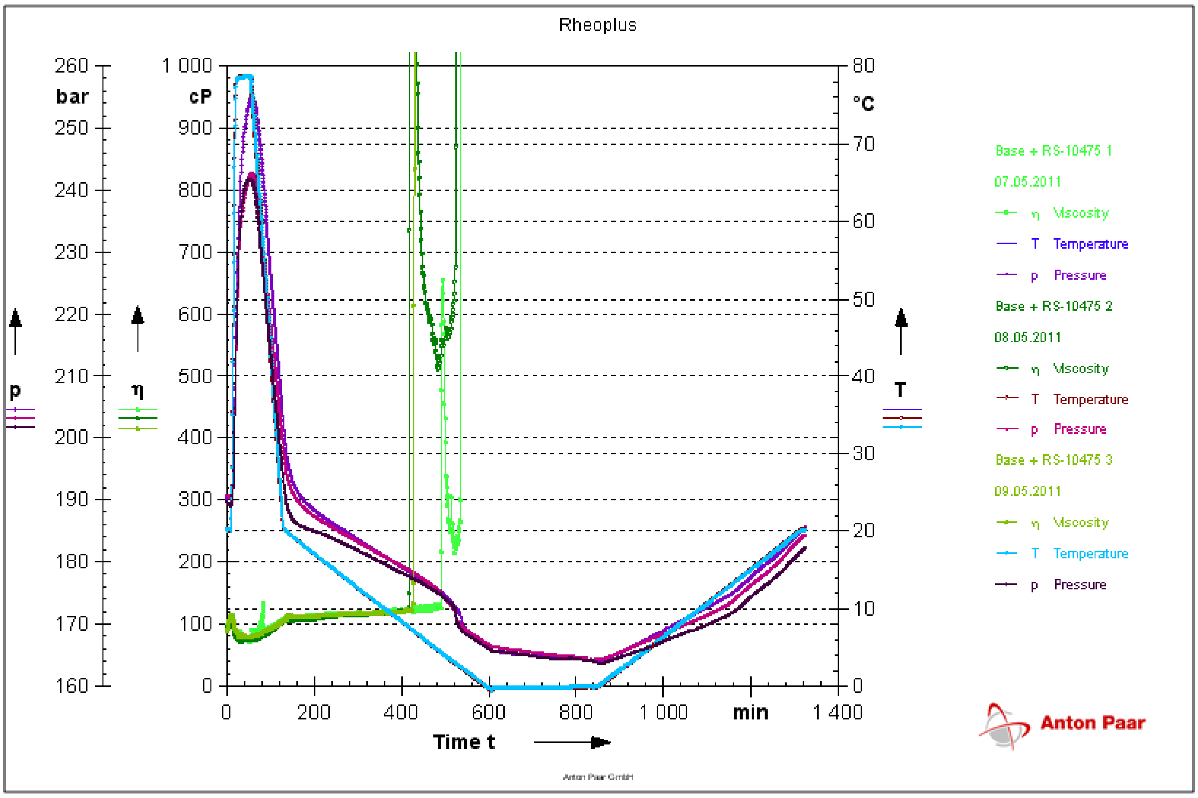

Figure 45: Formulation 3, base fluid + RS 10475 with viscosity range cP to 1000.

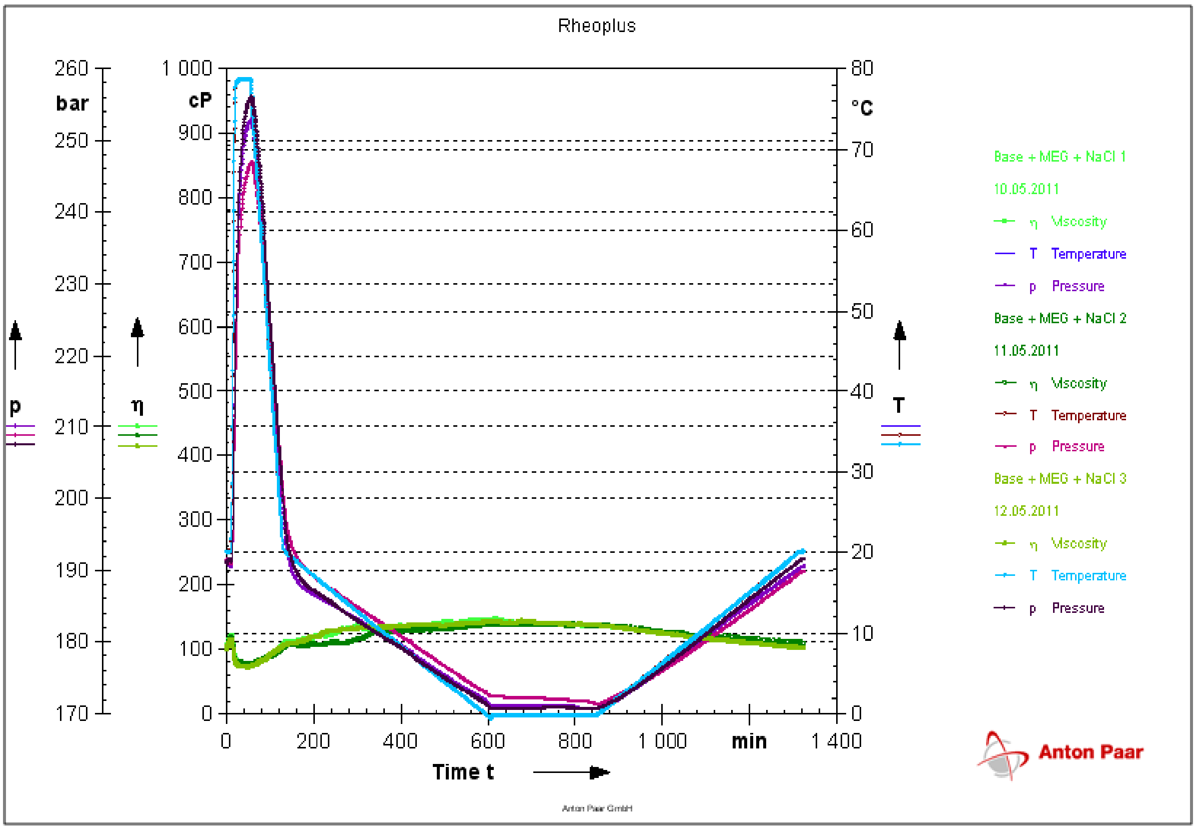

Figure 46: Formulation 7, base fluid + MEG + NaCl with viscosity range cP to 1000.

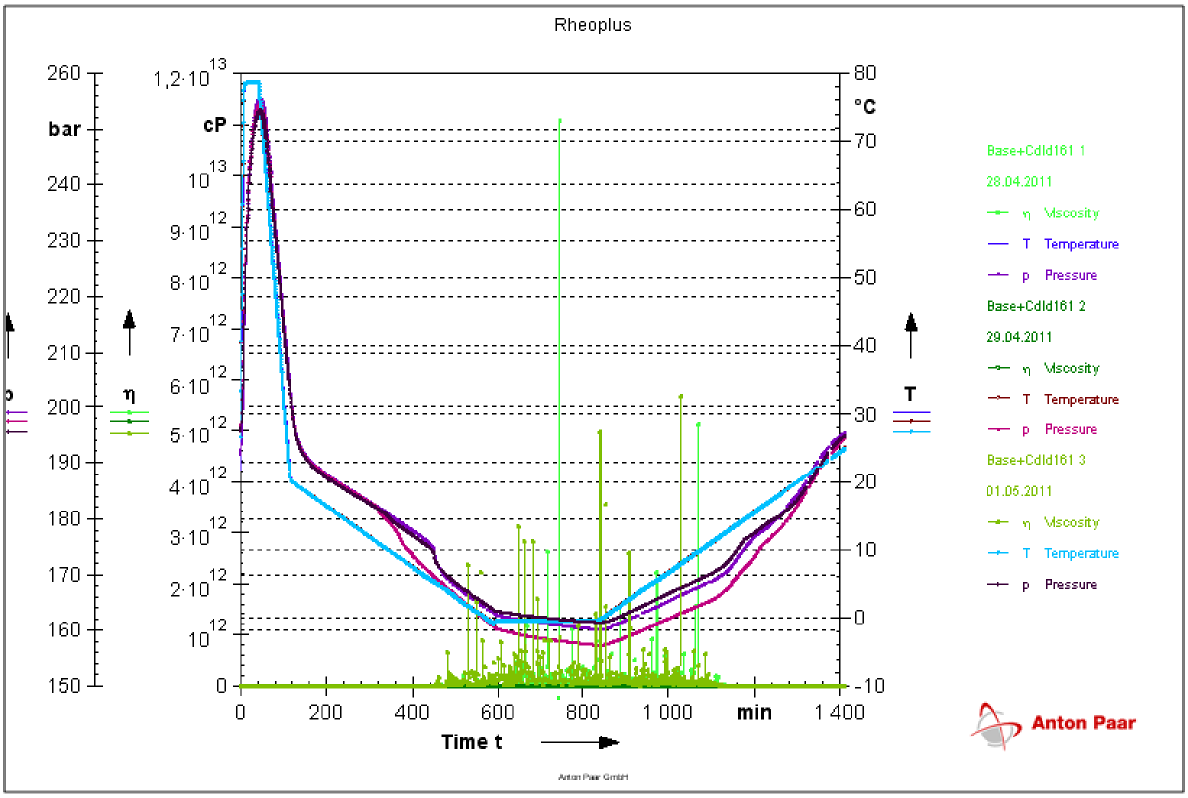

Figure 47: Formulation 9, base fluid + CdId 161 with viscosity range cP to $4*10^{13}$

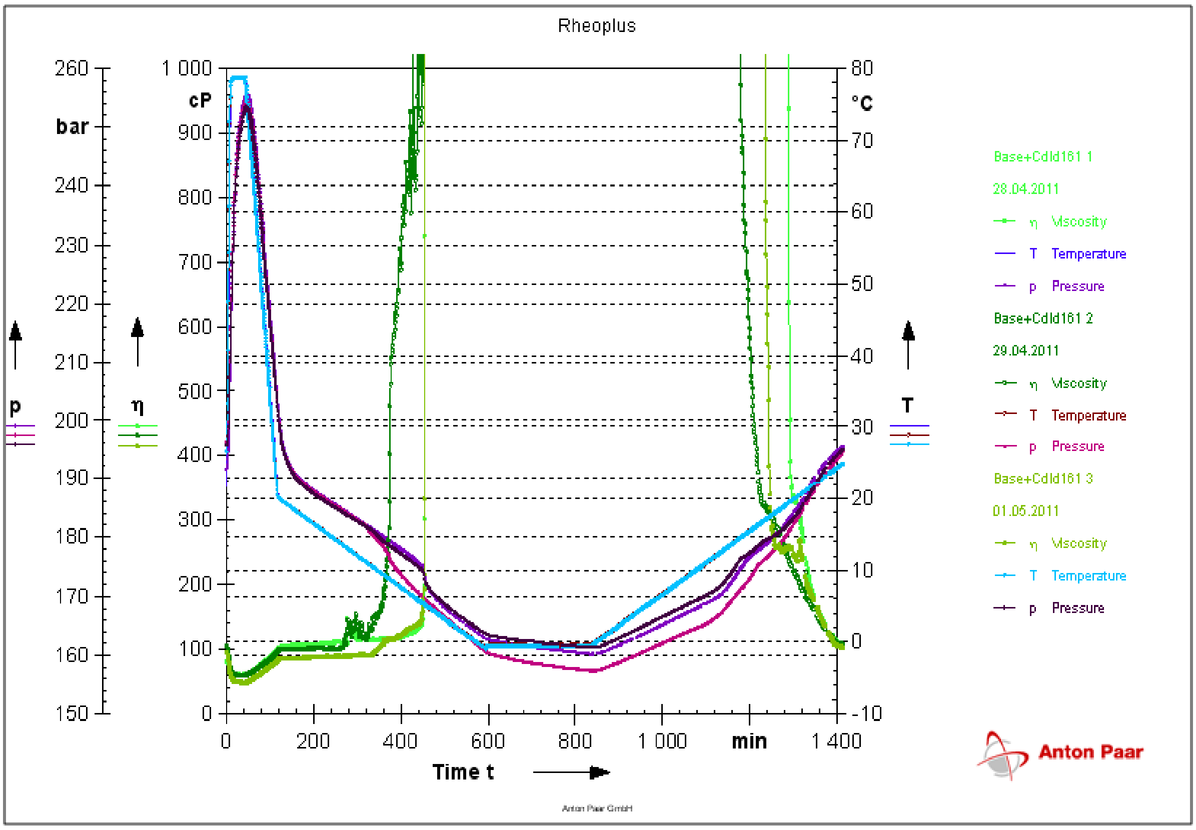

Figure 48: Formulation 9, base fluid + CdId 161 with viscosity range cP to 1000.

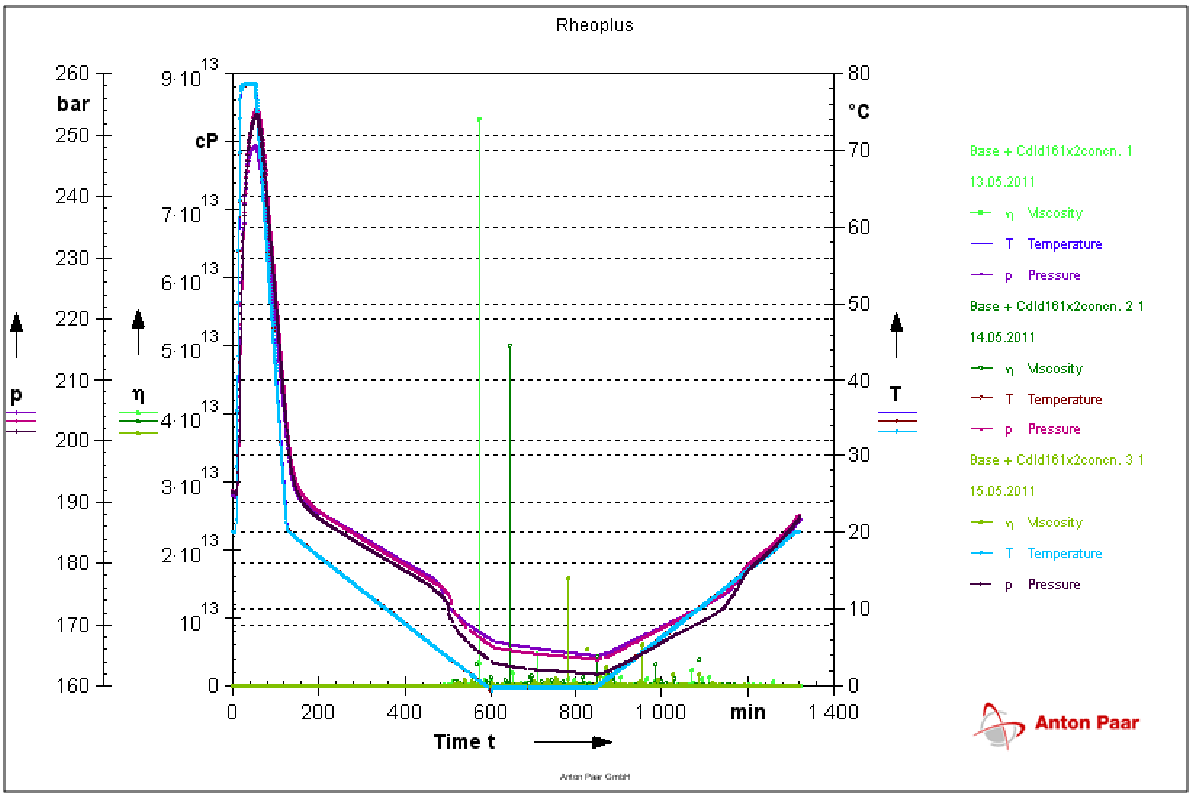

Figure 49: Formulation 10, base fluid + CdId 161 in double concentration with viscosity range cP to 4*10^{13}.

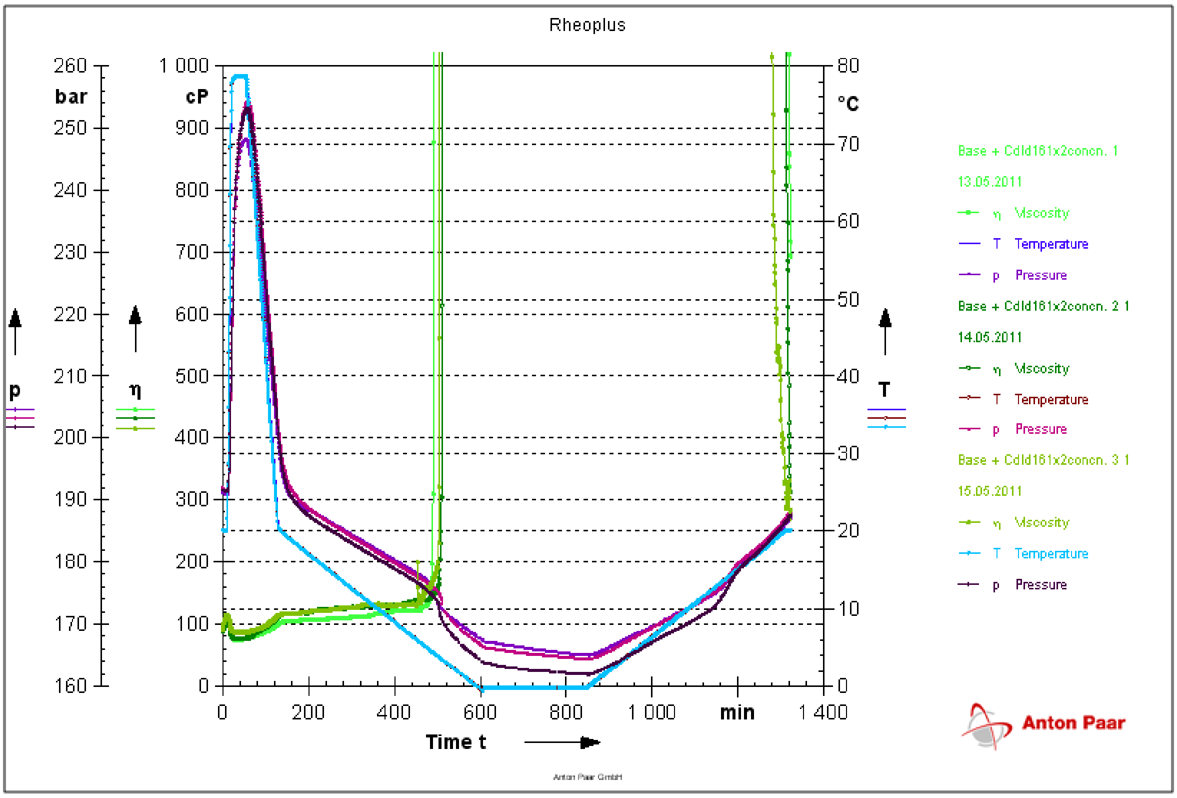

Figure 50: Formulation 10, base fluid + CdId 161 in double concentration with viscosity range cP to 1000.

10 DISCUSSION

10.1 DRILLING FLUID PROPERTIES

10.1.1 RHEOLOGY, GELS, YIELD POINT AND PLASTIC VISCOSITY

When a drilling fluid is evaluated, rheology is an important and common factor to measure, and the word itself means the "study of deformation and flow". According to the procedure there is no need for several tests/parallels for this test. Therefore it was decided that one was enough. Rheology was determined by measuring the viscosity of the fluid at different rpm values (600, 300, 200, 100, 6, and 3 rpm). This was done by using a Fann35 rheometer and the standard testing temperature is $50^{o}C$, as mentioned in the experimental section. These values give a graph which follows a theoretical model and tells us something about the properties of the drilling fluid and also which type of drilling fluid it is. The main theoretical models are: [1, 17]

- *Bingham Plastic model for fluids containing suspensions of solids and have a yield point*
- *Power law model for pseudo plastic fluids and shear thickening fluids*
- *Herschel Bulkley model (modified power law)*
- *Newtonian model for Newtonian fluids*

Only the actual model and fluids relevant for the results will be discussed further here.

From the results from figure 27, 28 and 29, it appears that the graphs are a mixture of the Bingham Plastic model and the Newtonian model. Typical for Bingham Plastic model is that it contains some particles and it is in a need for a force to be in movement. This force is named "shear strength" (SS) and when the SS becomes high enough, it goes from acting as a Bingham Plastic fluid to acting as a Newtonian fluid. Just in the border of switching from Bingham to Newtonian, the fluid has a curtain value that is named Yield Point (YP). YP is a measure of the power of the charged particles and how they reflect to each other (attracting and repulsing forces of the particles). High YP means strong forces between the particles and is most important when the fluid is not circulating. The formulations used in this project do not have a lot of particles, but there are some particles present and they have a YP the same way the Bingham plastic model has. The Bingham model is very common and is the ideal in theory. The most important rpm values here are 300 rpm and 600 rpm. In this model, the plastic viscosity (PV) and YP comes from the formulas as also mentioned in the experimental part: [1, 9]

$PV = \theta_{600} - \theta_{300}$, and is given in cP. $YP = \theta_{300} - PV$, and is given in $lbs/100ft^2$

For Bingham Plastic fluids, viscosity will decrease with increasing shear rate. These types of fluids are often fluids with charged particles. This fits with the fluids we tested, which contain water (seawater), since seawater has a lot of charged particles. These are mostly NaCl. Therefore this also fits with the theory of being a Bingham plastic fluid. At the same time NaCl is also added in some of the formulations, which also gives more charged particles to the fluid. [1]

As mentioned above, the fluids appear to go from Bingham plastic fluids to Newtonian fluids. Newtonian fluids are characterized as simple fluids with small or little particles, e.g. glycol, water and oil, and the fluid is not dependent on the shear rate. The Newtonian model can also correspond with the drilling fluids used here, as they contain water and some also contain MEG. [1, 9]

The figures 27, 28 and 29 show that the rheology is very similar for all samples tested and that there is no significant variation in the graphs. The values are at normal range and within acceptable levels for water based mud. The rheology for the fluids has not changed significantly and is more or less the same before ageing and after dynamic/static ageing. This means that dynamic and static ageing will not affect the drilling fluid negatively. Thus, during drilling and during down time the rheology of the fluid will more or less be unaffected. This is a good result and will be considered as a positive outcome. The rheology of the drilling fluid will be intact and not affected by the process.

As seen in the graphs in figure 30, 31 and 32, the values are more or less equal for yield point and gels, both before ageing and after dynamic/static ageing. If anything should be said about this, it is that the highest values after dynamic ageing are from samples 2, 3, 4 and 7, but also number 1 (the base fluid) is a bit higher. However, compared to the other samples, this slight difference does not give any big difference in the drilling properties. Looking at figure 32 compared with figure 31, the values have increased slightly, but not so much that it causes concern. The values are still within acceptable level for water based drilling fluids for both gels and YP.

Figure 33, 34 and 35 show some minor changes in the values. These differences are so small that they are considered as insignificant. This means that the PV has not been significantly/ negatively affected during the tests.

None of these fluids stand out negatively neither in rheology, gels, YP or PV, and there are no big differences from before ageing, after dynamic ageing, to after dynamic + static ageing. All the values are within acceptable and expected range to perform as good drilling fluids, so the results from these tests are considered as acceptable.

10.1.2 pH

A drilling fluid (WBDF) should have a pH between 8- 10 as mention in section 7.1.2.10 in page 18. All the fluids tested were within that range. From the pH–graph in figure 36, it can be seen that pH is decreasing slightly from before ageing to after the dispersion test. This is normal and excepted.

10.1.3 API FLUID LOSS

When testing API fluid loss, the outcome from the test is the fluid loss in mls and a filter cake which form on the filter. A good result from this test is a low fluid loss and a thin and firm filter cake. According to the procedure, one parallel for this test is enough, so only one parallel was performed for each fluid/ sample. During the testing of the 9 samples there was only one that had a high fluid loss; number 8 with 6,3 ml, as seen in figure 37. The best outcome from this test was for samples number 5 and 6, with low fluid loss values of 2.7 and 2.8 ml. This is a really good result in this test. The other samples had acceptable fluid loss results. All the filter cakes were thin and firm. This is shown below in figure 50, where both the filter cake and fluid loss had a good result. Therefore, the result from the API fluid loss test was considered to be acceptable.

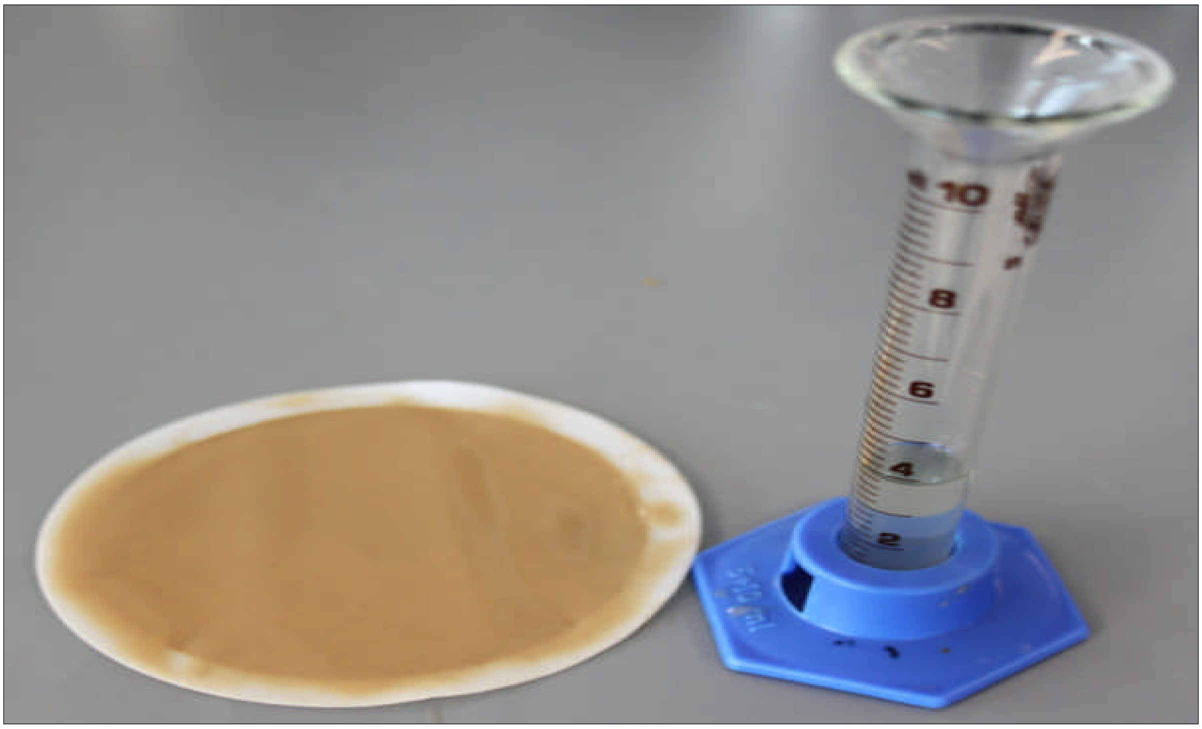

Figure 51: API fluid loss results, filter cake to the left and fluid loss to the right.

10.1.4 SAG TEST

Sag test is a well known test and according to the procedure only one parallel on each sample is necessary. Testing of sag is done to see how much of the particles that will separate under a circulation stop, and if the mud weight is different from the top or in the bottom. The Sag Index is a number of this and a good sag result is within a Sag Index of; 0,5- 0,52. An ideal sag test should also have as little free fluid on top as possible.

Looking at figure 38, Sag results, it is easy to see that the fluids differ from each other. Fluids 1, 2, and 4 all have a good sag index, which means that there is very little difference in the mud weight from top to bottom, and that during down time the particles will not sink to the bottom, but stay dispersed in the drilling fluid. Also, there was little free fluid in all of these, except number 1 with 53 ml, and number 5 where some free fluid appeared in the middle. Looking at number 2, 3, and 4, these fluids have given exactly the kind of outcome wanted from a good drilling fluid when analyzing the sag performance.

Samples 5, 6, 7, and 8 have a sag index value below 0.5, which in theory is not possible. The reason for this is that these samples do not contain any weighting agents; there are no barite in the mud and therefore no particles to sink to the bottom either. At the same time there was considerable amounts of air in these fluids, many tiny air bubbles which were impossible to get rid of. Thereby, when measuring the weight of the mud layers using the pycnometer, it gave the wrong weight and therefore incorrect sag values. Since this is possible to explain by to all the air trapped in the fluids, and also that these fluids do not contain any solids, the sag test result for these fluids are not important, and will not have any effect when considering the drilling fluid properties overall.

10.1.5 DISPERSION AND ACCRETION

During drilling the drill string will come across different geological structures in the formation. Drilling through clay is common and the clay will affect the drilling process and also the drilling fluid. Drilling through clay can be a troublesome process, because it will react with the drilling fluid and swell. As mentioned earlier in the theory section, swelling of clay is an unwanted process. One of the most swelling clays possible to meet during drilling is named “Arne Clay” and is from the U.K. This clay has a high swelling potential and will most likely cause problems in the bottom hole assembly and all the way to the surface. Since this clay is the one that is causing most trouble, it is the clay used in both the dispersion test and accretion test. Both these tests are used to evaluate whether the drilling fluid will react with the clay and, if so, how much. [18]

10.1.5.1 DISPERSION

The dispersion test will give a measure of how the particles will transform in size and shape from the bottom hole assembly and further up to the rig. Ideally the particles should be unchanged during this process, but normally with Arne clay they will swell and become bigger and softer on the way up. The clay will disperse into the drilling fluid and the recovery will be low. If the percent recovery is high and the moisture content low, this is a good indication that the fluids are inhibitive towards clay. In dispersion testing, 100 % recovery will be optimum. If the recovery content is equal, it is possible to compare the weights from the two sieves used. Then the one with highest percent recovery from the larger sieve is the most inhibitive fluid and thus the best.

The dispersion test is normally performed with only one parallel, but here 3 parallels were performed on samples number 1 and 5- 9. This was only done to learn the procedure properly and was not really necessary for the test results. In samples 2- 4, the parallels were reduced to two, for the same reason.

The graph of dispersion, figure 39 was made by using the average result of the parallels and then finding the standard deviation from this. This made it possible to determine which, if any, of the parallels that were outliers. These outliers are marked with bold/grey in the calculation sheets in the appendixes. After excluding the outliers, a new average was calculated, and this is the one showing in the graph in the result chapter. In the dispersion test, the best possible result will be 100% recovery. Any number in the calculation sheets in appendix 1.2a higher than 100% are considered as 100%

anyway. The reason for numbers being higher than 100% may be because of mud, polymers or salt particles sticking to the clay.

Based on the results from the dispersion tests, samples 7 and 8 are the best, since these two are at maximum value, with 100 % recovery. The lowest recoveries are for samples 1 and 9. It should be noted that samples 2, 3 and 4 were tested using a different batch of Arne Clay than the others. This can account for some of the reason for a lower recovery number, but not entirely since also 1 and 9 are low, and these were performed with the same batch of Arne Clay used on the samples yielding 100 % recovery. When observing the clay before and after the test, they had not changed much in shape and appearance. They looked almost the same, which is good. It means that in a drilling situation they will stay the same all the way up to the surface, not react and affect the drilling or the properties of the mud used during drilling. This can be seen in figure 51 below:

Figure 52: Dispersion result; appearance of Arne clay after the test was performed.

10.1.5.2 ACCRETION

The accretion test will give an indication of how much clay is excepted to attach to the drill string; if it will be a small amount that attaches and in form of small lumps, or if it will be big areas of the drill string that is covered with a layer of clay. The accretion value should be as low as possible. This is shown in the figure 52 below. Cylinder number 2 from the left has little that attaches and in form of small lumps. This is the wanted outcome of an accretion test, but as you see in this figure, there is only one cylinder looking like this and is thereby an outlier. Therefore is it the 3 other cylinders that are representing the actual test results. These 3 cylinders are on the other hand representing a negative outcome of the test. As seen, the clay is sticking to the cylinders and there is also a lot of it. This shows how it not supposed to be.

Figure 53: Accretion results.

In the accretion, test the same batch of Arne clay was used throughout the entire testing. Four parallels were performed, since this a rater inaccurate test. The reason for the test being so inaccurate is due to measuring the wet chippings, which can vary because the person performing the test may use a bit more time before doing the wet chippings measurement. Then more water may allowed to run down and away from the clay, making the wet chippings not as wet as they are supposed to be during the weighing. This is also the reason why the moisture of recorded sample has not been a part of the results. It is too incorrect and impossible to perform identically.

The graphs in figure 40 of accretion, were also made by calculating the average, the standard deviation and discarding the outliers (marked as bold/grey in the appendix 1.2), and calculating the average again after removing the outliers. In the graph, the samples have somewhat varying accretion values. However, this is acceptable because accretion values must vary at least 10-15% to be able to say that they are better or worse than each other. Accretion will almost always give a number around 30 %, more or less, based on results from accretion tests performed over the years. Therefore, samples 3-9 are considered to yield a good result, while samples 1 and 2 are slightly higher. Thus if any samples were to be excluded based on this test, it should be samples 1 and 2. [19]

10.1.6 UNCERTAINTY OF THE TESTS

During the testing some uncertainties appeared that must be mentioned. This was mainly in the sag, dispersion and accretion tests. In sag testing, it is important that there is no air, or as little air as possible in the mud, for the test to give as correct values as possible. Some formulations had a lot of air (as mentioned earlier), and this is an inaccuracy. In the sag test the cup content (volume of mud in each cup) may vary a bit, especially if the free fluid on top fluctuates.

With dispersion and accretion testing the uncertainty appears in the weighing of the wet chippings. This is because of some samples may contain a bit more water than the otter if they has been allowed to dry off meanwhile other samples are weighed. The time when the water dries of makes the uncertainty in the wet weighing. Therefore the test should be performed with around the same time limit for each sample. There will anyhow be some inaccuracy here, even if the person performing the test is really precise in his work, but it is unable to improve anymore.

All the uncertainty has been taken into consideration during the evaluation of the test results.

10.1.7 SUMMARY OF ALL THE STANDARD DRILLING FLUID TEST RESULTS:

Looking at all the tests performed on the drilling fluids (Samples 1-10), the rheology is acceptable, the gels, YP and PV are also acceptable. If a lower viscosity is wanted, this can easily be obtained by adding less viscosifier (Duotec NS). Therefore viscosity is not a big issue. Seeing as all pH values are within acceptable range, nothing needs to be done to modify here. Therefore, when determining which of the drilling fluids (samples 1-10) to test further on the gas hydrate part, the decision should be based on API fluid loss, sag, dispersion and the accretion results. The formulations with best result from these tests are formulations 3, 9 and 10, and will therefore be further tested in the gas hydrate part. Formulation 1, the base fluid, must be tested further at GHT, since this is the reference mud for all the formulations. Formulation 7 did not give particularly good results in the drilling fluids tests, but it is expected to give a good result on GHT, because of the content of MEG and salt (NaCl). MEG and salt are known from the industry to be good THI, and will therefore also be set up for gas hydrate testing. This is done to see the effect of adding THI compared to KHI or AA in the gas hydrate test. All formulations chosen for the GHT are listed below.

<u>The formulations that qualified for GHT were as follows:</u>

- Formulation no. 1: The base fluid
- Formulation no. 3: The base fluid + RS 10475 (KHI)
- Formulation no. 7: The base fluid + MEG + NaCl (THI)
- Formulation no. 9: The base fluid + CdId 161 (AA)
- Formulation no. 10: The base fluid + CdId 161 in double concentration (AA)

10.2 GAS HYDRATE TESTING

10.2.1 FORMULATION 1 – THE BASE FLUID

As shown in figures 42 and 43, which show formulation 1 the base mud, there is a lot of hydrate in the sample. This is expected, since there are no inhibitors in the formulation. The gas hydrate formation occurs after approximately 300- 450 minutes, at a pressure of 172-174 bar and around 8-12 ^{0}C. This is seen by a rapid and really large increase in the viscosity. The viscosity goes from about 100 cP to $10^{12}/10^{13}$ which is a high viscosity. This viscosity increase is approximately mutual for the formulation 1, 3, 9 and 10. It will therefore only be mention here. The gas hydrate starts to melt after 1300 minutes at a temperature around 18-20 ^{0}C, showing as decrease in the viscosity curve.

10.2.2 FORMULATION 3 – BASE FLUID ADDED KHI RS-10475

The GHT results for sample 3, base fluid added the KHI RS-10475, are given in figures 44 and 45. As shown in the figures, sample 3 also yields a lot of gas hydrate. The formation of gas hydrate occurs at a pressure of 170-174 bar (which is almost the same pressure as for the base fluid), after ca 400-450 minutes, and a temperature of: 4-9 ^{0}C.

The chemical RS-10475 does not seem to have any good effect on inhibiting gas hydrate formation or gas hydrate melting. It acts poorly as an inhibitor since the gas hydrate growth already shows at 4-9 ^{0}C. It is also negative that the gas hydrate does not melt when increasing the temperature again.

Due to a lot of gas hydrate and also that gas hydrate melting does not occur, this inhibitor (RS-10475) was considered as not acceptable and was not tested any further with other combinations.

10.2.3 FORMULATION 7 – BASE FLUID ADDED THIs MEG AND NaCl

Figure 46 shows the GHT results for sample 7, base fluid added MEG and NaCl. There is no sign of gas hydrates at all. This is an expected result since both MEG and salt (NaCl) are known from the industry as good THI inhibitors for preventing gas hydrate formation. This figure show how a good inhibition chemical work and how the curves corresponding to this will look like.

It is important to remember that salt mixed with glycols has a limitation; too much salt added to glycol can make the salt insoluble if the solution is already saturated. Then the solution will have high solid content from salts and cause problems during drilling. Another issue with this formulation is that MEG is needed in a high concentration to be a good inhibitor. It is both expensive and has an environmental concern due to the large amounts used. Therefore, even if the result form formulation 7 is acceptable in terms of preventing gas hydrate formation as shown in this test; it may not be a very good option anyway. [20-23]

10.2.4 FORMULATION 9 – BASE FLUID ADDED AA CdId 161

Formulation 9 containing the inhibitor CdId 161 was first tested using the standard concentration. Then it was tested with double concentration. The reason for this was that at first the inhibitor was considered to be 100 active % material/ component. As this is, however, an experimental AA and one is not sure that it is 100% active material, it was decided to double the concentration to check if that yielded better results.

10.2.4.1 FORMULATION 9 –STANDARD CONCENTRATION

Figures 47 and 48, representing the CdId 161 in standard concentration, show significant gas hydrate formation. The gas hydrate formation starts at a pressure of 165-175 bar, a temperature of 4-9 ^{o}C and after 250- 450 minutes.

The gas hydrate melting occurs after ca. 1300 minutes, at a temperature of around 16-20 ^{o}C and a pressure of 184-186 bar.

The chemical inhibitor (AA) Cdld 161 did not appear to have a good inhibition effect on gas hydrate formation and the result is unacceptable.

10.2.4.2 FORMULATION 10 - DOUBLE CONCENTRATION

GHT results for the double concentration of CdId 161 are given in figures 49 and 50. This sample also shows a high degree of gas hydrate formation. The formation starts after 400-500 minutes, which is a bit later than the formulation with single concentration. It happens at a temperature of 3-4^{o}C, and with a pressure of: around 172-174 bar.

The gas hydrate melting is after ca. 1300-1400 minutes, also a bit later than the formulation with single concentration. Both temperature and pressure of the melting process is unclear (melting is not complete at 20ºC) in the figures and cannot be discussed.

Doubling the CdId 161 concentration did not improve the hydrate inhibition. The inhibitor Cdld 161 is, as mentioned earlier, an AA and is therefore expected to be effective at < 1 weight % concentration. A factor that may be relevant is that the activity of the chemical was not 100% certain which could contribute to a poorer result than anticipated. If Cdld 161 was an option for use to inhibit gas hydrate formation, one would have to limit the use to areas with low water cut. Further, the subcooling is very high; around 20ºC, thus it might be way too much for one single inhibitor to achieve by itself.

10.2.5 SUMMARY OF ALL GHT RESULTS

If shut down or down time of any other reasons occurs, it may affect the temperature of the drilling fluid. The temperature will then decrease, making the conditions for gas hydrate formation more favorable. In other words, this means that the equilibrium curve for gas hydrate will move. Hydrate formation can affect the drilling fluid negatively, by affecting the rheological properties and can in worst case scenario result in no fluid circulation. It is therefore important for the kind of research done in this project, and during the last years, to go on and continue. [24, 25]

Underneath in figure 54 it is shown cleaning after a test, when the sample is removed. Here it is possible to see the gas in the sample as lots of bubbles.

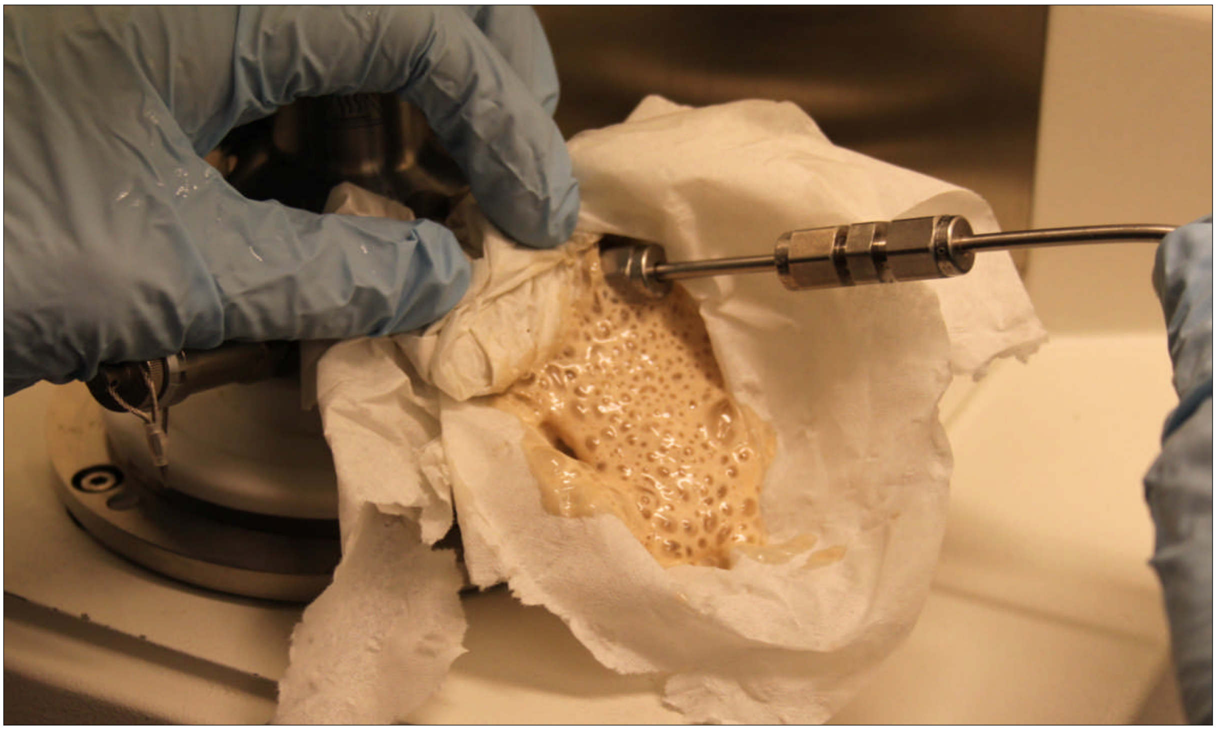

Figure 54: Removing of gas hydrate test sample.

During drilling in deep water, there is always a risk of hydrate formation. The temperature is low and pressure is high, and as the drilling continues further down in the formation, the pressure increases further and temperature keeps sinking. This makes the gas hydrate formation risk very large. Former research shows that water based drilling fluid has been used in deep water drilling and THI has also been used in the drilling fluid, to prevent gas hydrates (and also increasing the temperature). NaCl has been the most effective and economical chemical added to a THI WBDF. This also shows here in this project in sample 7 (base added MEG and NaCl), where no gas hydrate occurs. According to the calculation program WHYP, formulation 7 was not supposed to give this result. Predicted temperature for hydrate formation was 6,7^{o}C, but for test results shows no indication of hydrate formation after 4 hours at -1 ^{o}C. The reason for this is unclear and may be because of reactions between the components added to the drilling fluids, as a synergy effect. All the other formulations tested yielded unacceptable results, showing gas hydrate formation at relatively high temperatures.

A reason for the inhibitors in formulation 3, 9 and 10 not being able to hinder the gas hydrate formation may be linked to the low density of the fluid (1.1 s.g.). Low density fluids have shown poor effect on preventing gas hydrate in previous studies. Thereby this may be a reason for the negative outcome of the GHTs in this project. As mentioned before, too high concentrations of salt can also cause problems with the solubility of the salt and the density of the fluid and it must be taken into consideration in the final evaluation. [23]

Another reason for the KHIs/AAs in formulations 3, 9 and 10 not having any effect in preventing gas hydrate formation is most likely that the subcooling in the gas hydrate test has been too high. It is highly unlikely that any KHI/AA will be able to handle a 20^{o}C subcooling by itself. The tests were done to see if it was possible to observe any difference in the performance of the inhibitors in the gas hydrate test. One goal of this project was to find a good inhibitor for gas hydrate formation, acting together with the drilling fluid, without causing any negative effect on the drilling fluid properties. If it was possible to use a KHI/AA alone or even together with a THI it would be a positive outcome, since a THI alone is not economical due to the large amounts needed. The test procedure is still under development by MI-SWACO, as mention in the experimental part, and these tests result have showed that the gas hydrate equipment test setup works. It gives high viscosity levels and pressure drop for gas hydrate formation and it has also showed how no hydrate formation in formulation 7 looks as expected, with no/little viscosity increase and no pressure drop. Even if no good single LDHI was identified, the procedure of the test has been proven reliable and repeatable, and as said before this master thesis is only a small part of the project and MI-SWACO is far from finished with this project.

11 FUTURE RECOMMENDATIONS

Previous research has shown the need for effective inhibitors in WBDF for deepwater drilling. Looking at the test results for the KHI and AA here, none of them are acceptable inhibitors for deepwater drilling. Low dosage hydrate inhibitors have been under development some years and still are. This emphasizes that is has a high potential and until then, THI is the best option. The result of the GHT in this thesis is far from satisfying and much more testing must be done. Sadly there are now more time for this in this master thesis, but more tests on this will be carried out by MI-SWACO. There has been some previous research on hydrates using rheometer, also which showed high increase in viscosity when gas hydrate occurs. It also relates too many of the same important parameters for gas hydrate testing. This accentuates that the method used to test gas hydrate formation and dissociation here in this project, has a large potential. This research emphasizes that it is possible to measure gas hydrate formation and that it is important to continue the testing and improve the method along the way. [26]

12 CONCLUSION

The standard laboratory drilling fluid tests showed that formulations 3, 7 and 9 had acceptable performances. This means that the inhibitors added to the base fluid did not affect the drilling fluid properties negatively. These formulations, together with the base fluid, (formulation 1) and formulation 10, were therefore brought further to GHT. All the GHT results were negative, apart from sample 7 containing the THI, as they all formed a lot of gas hydrates. Formulation 7 gave a positive and acceptable performance, since no gas hydrate formation occurred. However, formulation 7 contained MEG and NaCl, which are THI inhibitors. Thereby this result was as predicted, and was mainly included in the testing matrix to compare against the other inhibitors as well as prove that the GHT method did deliver results as expected. The gas hydrate test procedure has, on the other hand, shown that it works and can be used for measuring gas hydrate formation/melting, even if no good inhibitor was found in this master thesis. The desired outcome of this project was that some of the LDHI`s would have worked, alone or together with an THI, which unfortunately did not happen. MI-SWACO must therefore continue to run more tests and try to find an acceptable combination of THI and LDHI for use in the 1.1 s.g. drilling fluid for deep water drilling.

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1 APPENDIX

Contents

1.1 PROCEDURE

1.1.1 MUD MIXING

M-I SWACO

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CHAPTER 3.2 INHIBITIVE / POLYMER WATER BASED FLUID

This procedure is generally applicable for the following drilling fluid systems:

GLYDRIL
GLYDRIL DW
KCL / POLYMER
SILDRIL

EQUIPMENT

Hamilton Beach mixer / Silverson mixer
Electronic balance
pH meter
Mixing container, weighting boats, 10 ml syringe, spatula

ORDER OF ADDITION

The order of mixing is given in table below. If some of the products are not included in the formulation, please skip these steps.

Order of addition	Mix time - min.
Prehydrated Bentonite (PHB)	0
Fresh Water	1
Salts	2
pH additive to treat out any hardness	2
Viscosifier	10
Fluid loss control additives	10
Other liquid additives	5
Weighting agent	20
pH additive to specified pH - range	2

Prepared by:	Verified by:	Approved by:
Renate Mikalsen	Jorunn Øvsthus	Beathe M. Pettersen

PROCEDURE

1. For large volumes mix 1000 ml or 1400 ml on a Silverson mixer. For small volumes mix 350 ml or 500 ml on a Hamilton Beach mixer.

2. Add prehydrated bentonite (if required) followed by water to a mixing container. Mix for 1 minute at a speed adequate to maintain a vortex. If PHB is not included, just add water to the mixing container.

3. Add salt and mix for 2 minutes.

4. Add pH additive to treat out any hardness, and mix for 2 minutes.

5. Add viscosifier slowly into the vortex, ensuring that all polymers have completely dispersed and that nothing adheres to the side of the mixing container or the mixing head. Total mixing time = 10 minutes.

6. Add the fluid loss additives slowly into the vortex, ensuring that all polymers have completely dispersed and that nothing adheres to the side of the mixing container or the mixing head. Total mixing time = 10 minutes.

7. Add other liquid additives and mix for 5 minutes.

8. Add weighting agent during a 5 minutes period and mix for further 20 minutes.

9. Add pH additive to adjust to specified pH-range and mix for 2 minutes.

Procedural note: Ensure temperature of the fluid does not exceed 60ºC during the mixing process. If the fluid reaches 60ºC, place the mixing cup in a water bath during further mixing to cool it down.

STANDARD PRODUCTS - INHIBITIVE / POLYMER WATER BASED FLUIDS

Examples of typical products for the different inhibitive / polymer water based fluids are given in the table below

Products	Glydril	Glydril DW	KCl/Polymer	Sildril
Prehydrated Bentonite			(PHB)	(PHB)
Fresh Water	FW	FW	FW / SW	FW
Salts	KCl	NaCl / KCl	KCl	NaCl / KCl
PH additive to treat out any hardness	Soda Ash Pot. Carb. Lime NaOH KOH	Soda Ash Pot. Carb. Lime NaOH KOH	Soda Ash Lime NaOH KOH	Soda Ash Lime NaOH KOH
Viscosifier	Duovis, Duotec NS, Flowzan, Duovis Plus NS, Celpol R, CMC HV			
Fluid loss control additives	Flotrol, Trol FL, Polypac ELV, Polypac UL, CMC LV/HV			
Hydrate Inhibition		MEG		
Scale Inhibitition	Glydril MC	Glydril MC		Sildril L
Weighting agent	Barite $CaCO_3$	Barite $CaCO_3$	Barite $CaCO_3$	Barite $CaCO_3$
pH additive to adjust to specified range	**pH = 8-8.5**	**pH = 8-8.5**	**pH = 9-9.5**	**pH = 11-12***

***) The pH is depending on the silicate concentration in the formulation.**

1.1.2 pH

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CHAPTER 5.1 pH			

Standard pH range in water based drilling fluids is pH 9-12. Many of the additives have an optimum pH range for maximum performance (yield, solubility), but higher pH also reduces corrosion of steel and reduces bacteria growth.

EQUIPMENT

- Electronic glass-electrode pH meter
- Buffer solutions (pH 4.0, 7.0 and / or 10.0 - as applicable)
- Deionised water

CALIBRATION

1. Calibrate pH meter using appropriate buffer solutions.
2. Rinse probe using deionised water.
3. Store probe in 3M KCl solution.

PROCEDURE

1. Rinse electrode with deionised water.
2. Place electrode in sample to be tested and stir gently. Allow time for the reading to stabilize.
3. Report sample pH to nearest 0.1 pH unit.
4. Clean electrode and store in a vial or beaker with 3M KCl solution.

For additional information, see API Recommended Practice 13B-1, section 9.

Prepared by: Irene M. Færgestad	Verified by: Jorunn Øvsthus	Approved by: Beathe Pettersen

1.1.3 RHEOLOGY

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CHAPTER 4.2.2 FANN 35			

EQUIPMENT

VG-meter (model FANN 35) (Figure 1)
Thermo-cup
Thermometer/ Temperature regulator

Figure 1. VG-meter

PROCEDURE

1. Place a sample of mud in the thermo-cup. Immerse the rotor into the mud exactly to the scribed line.

2. Set the rotation speed to 600 rpm and heat the mud to the desired temperature and a steady reading are obtained, record this reading.

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Renate Mikalsen	Jorunn Øvsthus	Beathe M. Pettersen

1. While rotation, shift to 300 rpm and wait until the reading is steady prior to recording it.

2. Record the readings at 200, 100, 6 and 3 rpm

3. Shift to 600 rpm once more and stir the mud for 10 seconds.

4. Allow the mud to stand undisturbed for 10 seconds then turn the instrument on 3 rpm and record the maximum reading. This reading represents the 10 seconds GEL strength in lbs/100 sq ft.
   (The reading divided by 2 represents the 10 seconds GEL strength in Pa).

5. Restir the mud at 600 rpm for 10 seconds then allow the mud to stand undisturbed for 10 minutes prior to repeating the test at 3 rpm. This reading represents the 10 minutes GEL strength in lbs/100 sq ft.
   (The reading divided by 2 represents the 10 minutes GEL strength in Pa).

The PLASTIC VISCOSITY, PV, in centipoise equals the 600 rpm reading (step 2 above) minus the 300 rpm dial reading (step 3 above).

The YIELD POINT, YP, in lbs/100ft^2 equals the 300 rpm dial reading (step 3 above) minus the Plastic Viscosity.
(The YIELD POINT, YP, in Pa is YP in lbs/100ft^2 divided by 2).

Caution: Stop the VG-meter before cleaning the rotor. Wear heat-resistant gloves when cleaning hot thermo-cup.

For additional information, see API Recommended Practice 13B-1, section 4.3 for water based mud and API Recommended Practice 13B-2, section 4.3 for oil based mud.

1.1.4 DYNAMIC AND STATIC AGEING

CHAPTER 4.3.1 HOT-ROLLING AND STATIC AGEING

EQUIPMENT:

Heating oven, roller oven, ageing cell (figure 1)

Figure 1: Aging Cell

SAMPLE:

Recommended volume is 350 ml fluid per cell. If more fluid is needed, remember that the fluid will need some space for expanding when heated. At temperatures above 150°C a volume of 350 ml is the maximum.

NOTE: At temperatures above 120°C, WBM mud requires a few drops of oxygen scavenger to protect the polymers from "burning". The following oxygen scavengers are recommended dependent on base fluid:

Brines	Oxygen scavenger
Monovalent brines; i.e. Sodium, Potassium	**Safe-Scav NA** (Ammonium bisulfite)
Divalent brines; i.e. Calcium, Zink also Chloride/Bromine	**Safe-Scav CA**
Formate brines	**Safe-Scav CA**

PROCEDURE FOR HOT-ROLLING:

1. Ensure that all assembling parts of the ageing cell are marked with the same reference number and that the stem valve and lid is not blocked.

2. Transfer approximately 350 ml of fluid to an ageing cell (a small metal device made especially for the cell, showing the correct level, may be used).

3. Close the cell by the two lid-parts, with a metal-ring placed between them and tighten the screws. Insert the stem valve, if not already in place, and leave it slightly open.

4. Pressurise the cell through the stem valve with nitrogen (inert gas). Consult the table of pressure required vs. vapour pressure of the fluid phase and ageing temperature for the drilling fluid, Table 1. This is done to prevent the fluid from boiling (both OBM and WBM) and to remove oxygen to prevent bacterial growth and/or "burning" of the polymers (WBM). Check for leakage, applying water on top of the lid and the valve and look for air bubbles.

5. Preheat the roller oven to the desired temperature, and place the cell horizontally onto the rollers. Standard hot-rolling time is 16 hours, or hot-roll for the required time.

6. **Use dry heat resistant gloves when taking the cell out of the oven.** Cool down the cell, and **carefully bleed off the pressure. This should be done in a ventilated area or in a fume cupboard.**

PROCEDURE FOR STATIC AGEING:

Follow the same procedure as for hot-rolling, but place the cell in a vertical position without rolling.

Table 1: Suggested Applied pressure at various ageing temperatures.

Ageing Temperature	Water Vapour Pressure	Suggested Applied Pressure
ºC	**psi**	**psi**
100	14,7	25
121	30	50
149	67	100
177	135	150
205	247	250

1.1.5 API FLUID LOSS

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CHAPTER 4.4.1 API FLUID LOSS TEST			

EQUIPMENT:

API Filterpress, 90-millimeter Whatman Filterpaper No.50 or equivalent, timer, graduated cylinder

Figure 1: API Static Fluid Loss Test Equipment

PROCEDURE:

1. Make sure that all parts of the standard API filter press are clean, dry and that nothing is plugging the outlet in the bottom part.

Place the bottom part in a stand with the outlet facing downwards. Insert a <u>sealing ring</u> into the groove, and then the <u>screen/sieve</u> is followed by a <u>filter</u> and a <u>second sealing</u>

Prepared by: Siv Elisabeth Vik	Verified by: Jorunn Øvsthus	Approved by: Beathe M. Pettersen

2. ring on the top. Screw the body cell into the bottom part ensuring that they are properly mounted and sealed; this to avoid leakages.

3. Fill 1/3 volume of the cell with fluid and mount it into the support frame with a clean lid and sealing. Turn the locking screw downwards into the centred groove in the lid and seal the assembly properly.

4. Place a dry graduated cylinder under the drain tube to collect the filtrate. Close the pressure relief valve and apply pressure by adjusting the regulator to give a pressure of 100 (+/-5)psi.

5. Start the timer when the pressure is first applied. The total filtrate volume is measured after 30 minutes. NB! Maintain the pressure at 100 psi during the test.

6. Shut off the pressure regulator after 30 minutes and **carefully open the pressure relief valve. Disassemble the cell after all pressure is released. Pour out the fluid and examine the filter cake.**

7. Report the filter cake thickness in mm and the API fluid loss in ml.

For additional information, see API Recommended Practice 13B-1, section 5.2.

1.1.6 STATIC SAG TEST

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CHAPTER 4.6.1 STATIC SAG TEST USING AGEING CELL

EQUIPMENT

Ageing cell / Heating oven
Syringes
Measuring cylinder
Tablespoon
Plastic cups
Pycnometer
Mixing cup
Analytical balance

PROCEDURE

1. Mix the actual drilling fluid according to formulation and procedures.

2. Measure required initial properties (rheology, pH/ES, etc.).

3. Fill drilling fluid in ageing cell and record accurate volume of mud added (standard volume is 350 ml) *(A)*.

4. Close the cell properly and pressurise the cell with N_2 gas.

5. Dynamic age cell at specified temperature for 16 hours. Do not cool down or open cell.

6. Transfer dynamic aged cell directly to static oven and static age cell at specified temperature and angle for the specified period of time.

7. When ageing time is completed, take ageing cell out of oven and cool down to room temperature. Remember to keep the cell in the same angle as at which it has been aged.

8. Carefully bleed off the pressure prior to disassembling the cell.

9. Measure amount (ml) of liquid separated (using syringe and measuring cylinder) on top of drilling fluid *(B)*. Transfer the liquid to a mixing cup. Report free fluid on top as volume-% of aged m

Prepared by:	Verified by:	Approved by:
Siv Elisabeth Vik	Jorunn Øvsthus	Beathe M. Pettersen

1. Divide the drilling fluid volume in 5 parts and determine density of the 5 different layers according to the following procedure:

2. By using a syringe or a tablespoon, transfer 1/5 part of the mud from the top of the cell to a plastic cup. Repeat this 4 times i.e. the mud is divided in 5 parts.

3. Stir the mud in the cups with a spoon to remove any air.

4. Determine the density of the mud by using a 20 ml pycnometer. Remember to remove any trapped air.

A = Volume of aged mud (ml)

B = Free fluid on top (ml)

MW = Mud weight of drilling fluid

Free fluid on top (V/V %) = (B * 100) / A

Sag Index = SG bottom / (MW * 2)

After sag test is done, collect the free fluid and the five samples and mix together on a Hamilton Beach Mixer at low speed for 10 minutes. Measure required properties after ageing (rheology, pH/ES, fluid loss etc.).

1.1.7 SIFTING AND CHRUSHING CUTTINGS

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CHAPTER 7.1 SIFTING AND CHRUSHING CUTTINGS			

EQUIPMENT

Sieve	2 mm
Sieve	4 mm
Sieve	6,3 mm
Collecting pan	
Mortar and pistil / knife and wooden board	

MATERIALS

Oxford Clay, Arne Clay or other types of clays/shale

PROCEDURE

1. Arrange the sieves on top of the collecting pan, the one with the largest holes size on top.
2. Crush the lumps of material with a pistil in a mortar or use a knife and a wooden board.
3. Pour the material onto the sieves and shake it vigorous back and forwards.
4. Repeat until you have sufficient quantity of the required cuttings size.
5. Sort the chipping of various types into buckets. Keep the buckets closed.

Cuttings used in:

Dispersion test: 2-4 mm
Slake durability test: 2-4 mm
Accretion test: 4-6,3 mm

Prepared by: Jorunn Øvsthus	Verified by: Anne M.Fosse	Approved by: Beathe M.Pettersen

1.1.8 HOT ROLL DISPERSION TEST

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CHAPTER 7.4 HOT-ROLL DISPERSION TEST / RECOVERY			

This is a commonly used test method in optimising drilling fluids to reactive shale formations. The API test procedure, currently in the process of review by API Committee 13, is based on this method.

EQUIPMENT Laboratory balance ± 0,01g
Drying oven
Hot rolling oven running at approximately 40 RPM
500 ml Ageing cells, stainless steel 316 or plastic bottles
Weighing boats
Spatula
10 cm sieves of 10 mesh and 20 mesh aperture size (or 1 and 2 mm)
Inhibitive wash water (10% KCl)

PROCEDURE

1. Size the shale to between 2 and 4 mm (cuttings samples should be kept at all times in a sealed plastic container when not in use).
2. Determine the initial moisture content of the shale.
3. Prepare drilling fluids to be tested in accordance with standard procedures for the respective fluid, and test the properties required.
4. Place 350 ml drilling fluid in hot rolling cells (or plastic bottles if ageing temperature is < 65ºC). Preheat the oven to the desired temperature.
5. Accurately weigh approximately 30 grams of sized cuttings (W_1) and add to the 350 ml of the drilling fluid to be tested.
6. Use a spatula and stir gently to wet and separate the shale cuttings prior to sealing the cells.
7. Pressurise steel ageing cell to 100 psi with nitrogen.
8. Place the cells in the roller oven and age the sample at the required time and temperature.
9. After ageing, take the cell out of the oven and cool in a water bath. When the cells have reached room temperature the pressure is released and the cells are opened.
10. Prepare an inhibitive wash solution (10% w/v KCl for water based drilling fluids and baseoil and then water/detergent solution for oil-based drilling fluids).
11. Pour the content of the cell onto two sieves (stacked with 10 mesh above 20 mesh or 2 mm above the 1 mm sieve) taking care of the drilling fluid in a mixing cup.

Prepared by:	Verified by:	Approved by:
Jorunn Øvsthus	Anne M. Fosse	Beathe M. Pettersen

12. Carefully pour the wash water over the cuttings on the sieve to remove any adhering mud and solids using the wash solution already prepared (in sec. 10).
13. Weigh cuttings after removal from sieves (Best facilitated by knocking out cuttings onto a polyethylene sheet, pouring off excess surface fluid and patting dry cuttings, then transferring to a clean aluminium or glass dish. Record empty dish weight (W_2). Record wet cuttings weight from each sieve size (W_{10} and W_{20}) plus combined recovered cuttings weight (W_3).
14. Dry the cuttings to constant weight in an oven set to 105°C/ 225°F.
15. Re-weigh dish with "dry" cuttings (W_4).

CALCULATIONS

From the mass measurements taken, the percent recovery and moisture contents are determined:

Correction factor = B / A

% Moisture of recovered sample = $[(W_3 - W_4) / (W_3 - W_2)] \times 100$

% Recovery = $[(W_4 - W_2) / W_1 \times C] \times 100$

Where:
W_1 = Initial weight of cuttings used
W_2 = empty dish weight
W_3 = total dish and "wet" sample weight after hot rolling
W_4 = total dish and "dry" sample weight (after drying to constant weight).
A = Initial weight of cuttings (determination of natural moisture content of the shale).
B = Final dried cuttings
C = Correction factor

TREATMENT OF RESULTS

High percent recoveries and low moisture contents are indicative of inhibitive fluids. Clearly with a poorly inhibitive fluid, cuttings will disperse into the fluid and zero recovery (and therefore no moisture content determination) will result. It is occasionally observed that some shale undergo initial dispersion, which is then arrested by the drilling fluid. The result is 'medium' recovery but low moisture content. If two fluids gave the same percent recovery, the fluid which gave the lower moisture content would be regarded as being slightly more inhibitive because the water uptake by the cutting is less with a consequent reduced risk of dispersion or swelling in the wellbore. It is always advised, therefore, that moisture contents are determined on the wet residues. In addition a comparative measurement of inhibition can be obtained by reviewing the relative cuttings weights retained on each of the screen sizes. Higher percentages of the cuttings recovered from the larger (10 mesh / 2 mm) screen relate to a more inhibitive fluid, even with similar total recovery percentages.

1.1.9 ACCRETION TEST

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CHAPTER 7.5 ACCRETION TEST			

Test method for evaluation of a drilling fluid tendency to promote the occurrence of accretion

EQUIPMENT

- Hollow steel bar (length 120 mm, OD 35 mm, ID 32 mm)
- Plastic bottles (uni-diameter, 500 ml)
- Roller oven
- Laboratory balance ± 0,01g
- Drying oven
- Spatula

PROCEDURE

1. Any rust should be removed from the surface of the bar with detergent and 1000 grit wet & dry paper, following by a hot water rinse. Dry the hollow cylinders with clean paper towel and isopropanol. This cleaning procedure will also remove any chemical contamination remaining from previous tests.
2. Add 350 ml of test fluid to the plastic bottle
3. Place the clean hollow steel bar centrally in the 500 ml plastic bottle.
4. Add 30 g of sized Arne clay (4-6.3 mm) or test material (see NOTE) to the plastic bottle, evenly distributed around the central bar. Gentle agitation is applied with a spatula to ensure even distribution of the solids in the fluid.
5. Seal the bottle and invert 3-4 times to minimise the chances of cuttings or test material sticking to the bottom of the cell.
6. Immediately transfer the bottle to the roller oven and roll at room temperature for 10 minutes.
7. After rolling, remove the bar and wash gently with tap water.
8. Scrape off any accreted solids from the bar using a spatula and weigh wet solids into a Petri dish.
9. Dry to constant weight at 105°C and then reweigh to find final weight.
10. Calculate the % accretion and % moisture.

Prepared by:	Verified by:	Approved by:
Anne M Fosse	Jorunn Øvsthus	Beathe M. Pettersen

1. Clean the hollow cylinders and plastic bottles and run a minimum of 3 parallels of each test to improve accuracy of test procedure. Use average accretion value of the number of tests performed.

CALCULATIONS

% accretion = [Final Wt of cuttings / (Initial Wt of cuttings x corr. factor)] x 100

Correction factor = Final weight/initial weight

% moisture = [(Wet Wt – Final Wt of cuttings) / Final weight of cuttings] x 100

NOTE

Arne clay and 10 minutes rolling time should always be performed due to comparison reasons between internal lab requests and work carried out at other M-I locations. Other clay types and/or rolling times could be included if necessary.

By omitting step 4, this procedure can be used for determine accretion of solids in the mud.

1.2 WORK SHEETS

1.2.1 FORMULATION 1: BASE

SAMPLE INFO		
REQUEST NO	**Deepwater WBM**	CONFIDENTIAL
FLUID SYSTEM	**WBM**	
DESCRIPTION	**1.1sg**	
TEST DATE	**02.03- 05.05**	
TESTED BY	**MN**	

SAMPLE COMPOSITION																
Sample			**1**													
Mixing volume		ml	**1000**													
Sea water		g/l	954,1													
Duotec NS	MIN 08-443	g/l	4													
Trol FL	MIN 08-269	g/l	15													
Kla Stop (UltrahibNS)	MIN 09-049	g/l	20													
Barite	MIN 10-131	g/l	97,4													
EMI-2223	EXP 10-154	g/l	10													
Defoamer EMI-1705	MIN 08-319															
AGING CONDITIONS																
Period aged		HOURS		**16**	**16**	**16**	**16**	**16**	**16 + 24**							
Temperature		ºC		**100**	**100**	**100**	**100**	**100**	**100**							
Dynamic / Static		D/S	**BHR**	**D + Disp**	**D+ Disp**	**D+ Disp**	**D+ Disp**	**D**	**D + S**	**ACCR**	**ACCR**	**ACCR**	**ACCR**	**ACCR**	**ACCR**	**ACCR**
Sample				**1**	**2**	**3**	**4**			**1**	**2**	**3**	**4**	**5**	**6**	**7**
RHEOLOGY																
Temperature		ºC	**50**	**50**	**50**	**50**	**50**	**50**	**50**							
600 rpm		lbs/100ft²	39	42	42	42	42	39	39							
300 rpm		lbs/100ft²	31	34	34	34	33	31	32							
200 rpm		lbs/100ft²	27	30	30	29	29	27	28							
100 rpm		lbs/100ft²	22	25	25	24	24	22	23							
6 rpm		lbs/100ft²	11	11	12	10	10	12	12							
3 rpm		lbs/100ft²	9	9	9	8	8	9	9							
10 second gel		lbs/100ft²		8	8	7	7	13	13							
10 minute gel		lbs/100ft²		9	9	8	8	16	17							
Plastic Viscosity		cP	8	8	8	8	9	8	7							
Yield Point		lbs/100ft²	23	26	26	26	24	23	25							
FILTRATION - API																
API		ml						3,6								
OTHER																
pH			9,4	8,4	8,5	8,4	8,3	9,2	8,9							
Specific gravity		sg														
DISPERSION TESTING																
Initial weight chippings		g		30,0	30,0	30,0	30,0									
Dry weight chippings		g		29,4	29,4	29,4	29,4									
Correction factor - natural moisture		%		1,0	1,0	1,0	1,0									
Initial weight chippings (2-4 mm)		g		30,0	30,0	30,0	30,0									
Wet weight chippings : 1-2 mm		g		1,2	2,8	2,1	3,2									
Wet weight chippings : > 2 mm		g		31,5	31,0	30,7	32,5									
Final weight chippings		g		21,2	21,2	20,9	21,5									
Recovery		%		**71,9**	**72,0**	**70,9**	**73,1**									
Average recovery		%		71,9												
Moisture of recovered sample		%		**35,2**	**37,2**	**36,2**	**39,6**									
ACCRETION TESTING																
Initial weight chippings		g								30	30	30	30	30	30	30
Dry weight chippings		g								28	28	28	28	28	28	28
Correction factor - natural moisture		%								1	1	1	1	1	1	1
Exposure time		min								10	10	10	10	10	10	10
Initial weight chippings		g								30	30	30	30	30	30	30
Wet weight chippings		g								6	17	1	16	19	18	18
Final weight chippings		g								4	12	1	11	12	12	12
Accretion		%								**14**	**41**	**2**	**39**	**44**	**43**	**41**
Average accretion		%								**41**						
Moisture		%								**40**	**33**	**34**	**33**	**34**	**34**	**33**
STATIC SAG TEST																
MW		sg							1,1							
Volume aged mud		ml							350							
Free fluid on top		ml							53							
Separation		V/V%							15,1							
	Top	sg							1,042							
		sg														
	Middle	sg							1,056							
		sg														
	Bottom	sg							1,17							
Sag Index									**0,53**							

COMMENTS

Accretion: 3 more parallels here because the results varied a lot. Possible source of error can be because the samples did not roll during the 10 min of rolling.
Since this is the base mud/ the refrerence mud, we tok some more parallels on the accretion test to be sure of the results.
Dispersion: Normal with only one test, but since this is the base mud and all the other tests reffered to this, it has 4 parallels.

Accretion and dispesion values in bold/grey are considered outliers and not included in the average calculation

1.2.2 FORMULATION 2: BASE + LUVICAP 55W

SAMPLE INFO		
REQUEST NO	**Deepwater WBM**	CONFIDENTIAL
FLUID SYSTEM	**WBM**	
DESCRIPTION	**1.1sg**	
TEST DATE	**02.03- 18.03**	
TESTED BY	**MN**	

SAMPLE COMPOSITION											
Sample			**2**								
Mixing volume		ml	**1000**								
Sea water		g/l	954,1								
Duotec NS	MIN 08-443	g/l	4,0								
Trol FL	MIN 08-269	g/l	15,0								
Kla Stop (UltrahibNS)	MIN 09-049	g/l	20,0								
Barite	MIN 10-131	g/l	97,4								
EMI-2223	EXP 10-154	g/l	10,0								
Defoamer EMI-1705	MIN 08-319										
Luvicap 55W			12,0								
AGING CONDITIONS											
Period aged		HOURS		**16**	**16**	**16**	**16 + 24**				
Temperature		°C		**100**	**100**	**100**	**100**				
Dynamic / Static		D/S	**BHR**	**D + Disp**	**D+ Disp**	**D**	**D + S**	**ACCR**	**ACCR**	**ACCR**	**ACCR**
Sample				**1**	**2**			**1**	**2**	**3**	**4**
RHEOLOGY											
Temperature		°C	**50**	**50**	**50**	**50**	**50**				
600 rpm		lbs/100ft²	41	45	45	37	37				
300 rpm		lbs/100ft²	34	36	35	31	30				
200 rpm		lbs/100ft²	28	32	32	26	26				
100 rpm		lbs/100ft²	23	26	26	22	22				
6 rpm		lbs/100ft²	12	13	13	11	11				
3 rpm		lbs/100ft²	10	11	10	9	8				
10 second gel		lbs/100ft²		13	12	13	14				
10 minute gel		lbs/100ft²		15	15	15	15				
Plastic Viscosity		cP	7	9	10	6	7				
Yield Point		lbs/100ft²	27	27	25	25	23				
FILTRATION - API											
API		ml				3,9					
OTHER											
pH			9,2	8,9		9,3	9,0				
Specific gravity		sg									
DISPERSION TESTING											
Initial weight chippings		g		30,0	30,0						
Dry weight chippings		g		29,4	29,4						
Correction factor - natural moisture				1,0	1,0						
Initial weight chippings (2-4 mm)		g		30,0	30,0						
Wet weight chippings : 1-2 mm		g		2,3	1,4						
Wet weight chippings : > 2 mm		g		33,9	34,7						
Final weight chippings		g		23,1	22,6						
Recovery		%		**78,5**	**76,8**						
Average recovery		%		**77,7**							
Moisture of recovered sample		%		**36,0**	**37,2**						
ACCRETION TESTING											
Initial weight chippings		g						30	30	30	30
Dry weight chippings		g						28	28	28	28
Correction factor - natural moisture		%						1	1	1	1
Exposure time		min						10	10	10	10
Initial weight chippings		g						30	30	30	30
Wet weight chippings		g						23	15	26	15
Final weight chippings		g						16	10	18	10
Accretion		%						54	**35**	**62**	**34**
Average accretion		%						41			
Moisture		%						**33**	**33**	**33**	**36**
STATIC SAG TEST											
MW		sg					1,1				
Volume aged mud		ml					350				
Free fluid on top		ml					16,5				
Separation		V/V%					4,7				
	Top	sg					1,033				
		sg									
	Middle	sg					1,034				
		sg									
	Bottom	sg					1,111				
Sag Index							**0,51**				

COMMENTS

Dispersion: Only one dispersion parallel is standard procedure, but I did 2 to learn the testing procedure properly.

Accretion and dispesion values in bold/grey are considered outliers and not included in the average calculation

1.2.3 FORMULATION 3: BASE + RS- 10475

SAMPLE INFO		
REQUEST NO	**Deepwater WBM**	CONFIDENTIAL
FLUID SYSTEM	**WBM**	
DESCRIPTION	**1.1sg**	
TEST DATE	**02.03- 18.03**	
TESTED BY	**MN**	

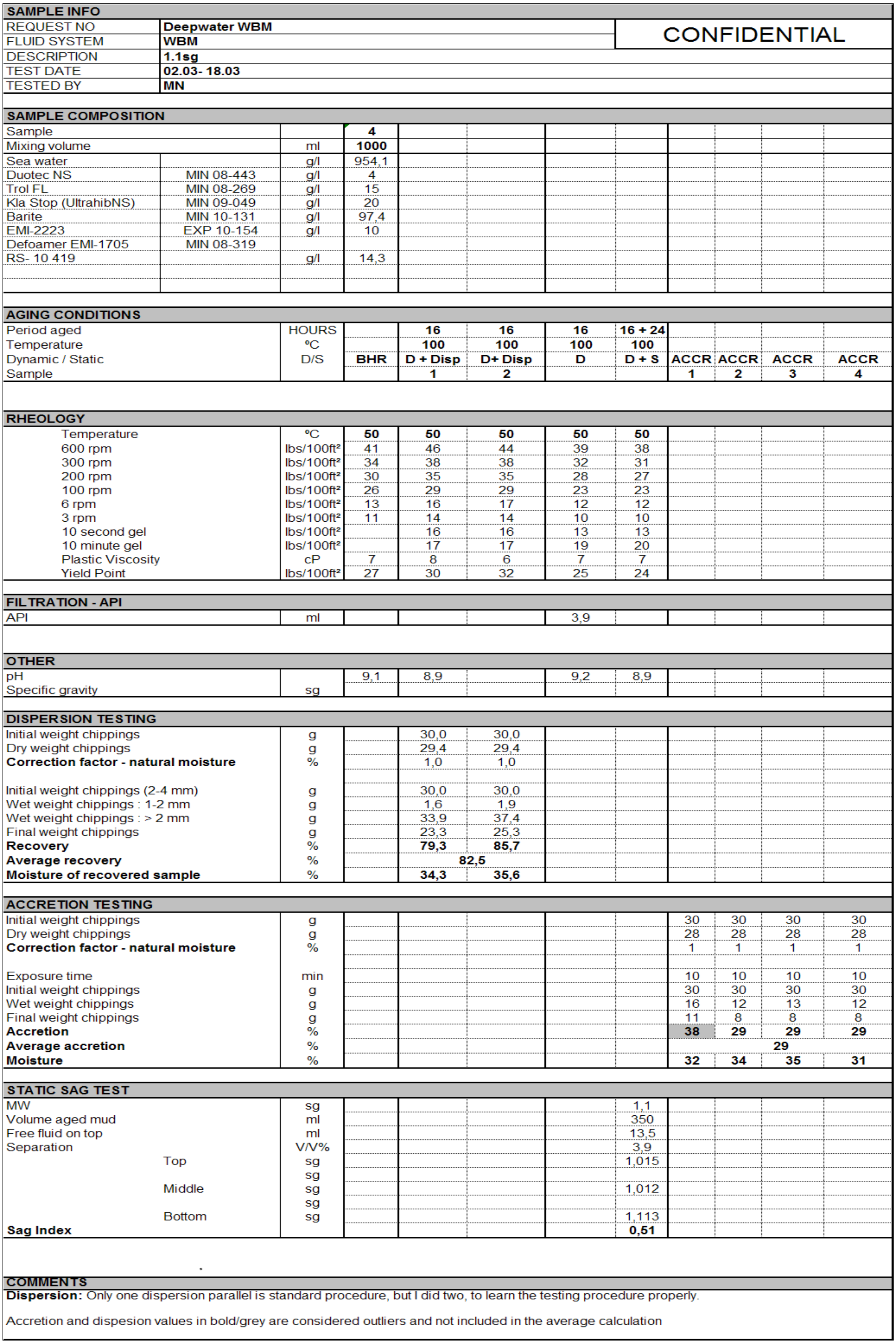
SAMPLE COMPOSITION											
Sample			**3**								
Mixing volume		ml	**1000**								
Sea water		g/l	954,1								
Duotec NS	MIN 08-443	g/l	4								
Trol FL	MIN 08-269	g/l	15								
Kla Stop (UltrahibNS)	MIN 09-049	g/l	20								
Barite	MIN 10-131	g/l	97,4								
EMI-2223	EXP 10-154	g/l	10								
Defoamer EMI-1705	MIN 08-319										
RS- 10475		g/l	20,7								
AGING CONDITIONS											
Period aged		HOURS		**16**	**16**	**16**	**16 + 24**				
Temperature		ºC		**100**	**100**	**100**	**100**				
Dynamic / Static		D/S	**BHR**	**D + Disp**	**D+ Disp**	**D**	**D + S**	**ACCR**	**ACCR**	**ACCR**	**ACCR**
Sample				**1**	**2**			**1**	**2**	**3**	**4**
RHEOLOGY											
Temperature		ºC	**50**	**50**	**50**	**50**	**50**				
600 rpm		lbs/100ft²	40	44	43	37	39				
300 rpm		lbs/100ft²	33	37	36	31	33				
200 rpm		lbs/100ft²	28	33	32	27	29				
100 rpm		lbs/100ft²	23	26	25	23	23				
6 rpm		lbs/100ft²	12	13	12	11	12				
3 rpm		lbs/100ft²	10	11	10	9	9				
10 second gel		lbs/100ft²		12	11	12	12				
10 minute gel		lbs/100ft²		15	14	14	15				
Plastic Viscosity		cP	7	7	7	6	6				
Yield Point		lbs/100ft²	26	30	29	25	27				
FILTRATION - API											
API		ml				3,7					
OTHER											
pH			9,3	9,0		9,2	8,9				
Specific gravity		sg									
DISPERSION TESTING											
Initial weight chippings		g		30,0	30,0						
Dry weight chippings		g		29,4	29,4						
Correction factor - natural moisture		%		1,0	1,0						
Initial weight chippings (2-4 mm)		g		30,0	30,0						
Wet weight chippings : 1-2 mm		g		1,9	1,5						
Wet weight chippings : > 2 mm		g		35,5	34,0						
Final weight chippings		g		23,4	23,0						
Recovery		%		**79,5**	**78,1**						
Average recovery		%		**78,8**							
Moisture of recovered sample		%		**37,5**	**35,2**						
ACCRETION TESTING											
Initial weight chippings		g						30	30	30	30
Dry weight chippings		g						28	28	28	28
Correction factor - natural moisture		%						1	1	1	1
Exposure time		min						10	10	10	10
Initial weight chippings		g						30	30	30	30
Wet weight chippings		g						13	12	13	18
Final weight chippings		g						9	8	9	12
Accretion		%						**30**	**29**	**31**	**41**
Average accretion		%						**30**			
Moisture		%						**34**	**31**	**35**	**34**
STATIC SAG TEST											
MW		sg					1,1				
Volume aged mud		ml					350				
Free fluid on top		ml					11,5				
Separation		V/V%					3,3				
	Top	sg					1,025				
		sg									
	Middle	sg					0,996				
		sg									
	Bottom	sg					1,125				
Sag Index							**0,51**				

COMMENTS

Dispersion: Only one dispersion parallel is standard procedure, but I did 2 to learn the testing procedure properly.

Accretion and dispesion values in bold/grey are considered outliers and not included in the average calculation

1.2.4 FORMULATION 4: BASE + RS- 10419



SAMPLE INFO											
REQUEST NO	**Deepwater WBM**							CONFIDENTIAL			
FLUID SYSTEM	**WBM**										
DESCRIPTION	**1.1sg**										
TEST DATE	**02.03- 18.03**										
TESTED BY	**MN**										
SAMPLE COMPOSITION											
Sample			**4**								
Mixing volume		ml	**1000**								
Sea water		g/l	954,1								
Duotec NS	MIN 08-443	g/l	4								
Trol FL	MIN 08-269	g/l	15								
Kla Stop (UltrahibNS)	MIN 09-049	g/l	20								
Barite	MIN 10-131	g/l	97,4								
EMI-2223	EXP 10-154	g/l	10								
Defoamer EMI-1705	MIN 08-319										
RS- 10 419		g/l	14,3								
AGING CONDITIONS											
Period aged		HOURS		**16**	**16**	**16**	**16 + 24**				
Temperature		°C		**100**	**100**	**100**	**100**				
Dynamic / Static		D/S	**BHR**	**D + Disp**	**D+ Disp**	**D**	**D + S**	**ACCR**	**ACCR**	**ACCR**	**ACCR**
Sample				**1**	**2**			**1**	**2**	**3**	**4**
RHEOLOGY											
Temperature		°C	**50**	**50**	**50**	**50**	**50**				
600 rpm		lbs/100ft²	41	46	44	39	38				
300 rpm		lbs/100ft²	34	38	38	32	31				
200 rpm		lbs/100ft²	30	35	35	28	27				
100 rpm		lbs/100ft²	26	29	29	23	23				
6 rpm		lbs/100ft²	13	16	17	12	12				
3 rpm		lbs/100ft²	11	14	14	10	10				
10 second gel		lbs/100ft²		16	16	13	13				
10 minute gel		lbs/100ft²		17	17	19	20				
Plastic Viscosity		cP	7	8	6	7	7				
Yield Point		lbs/100ft²	27	30	32	25	24				
FILTRATION - API											
API		ml				3,9					
OTHER											
pH			9,1	8,9		9,2	8,9				
Specific gravity		sg									
DISPERSION TESTING											
Initial weight chippings		g		30,0	30,0						
Dry weight chippings		g		29,4	29,4						
Correction factor - natural moisture		%		1,0	1,0						
Initial weight chippings (2-4 mm)		g		30,0	30,0						
Wet weight chippings : 1-2 mm		g		1,6	1,9						
Wet weight chippings : > 2 mm		g		33,9	37,4						
Final weight chippings		g		23,3	25,3						
Recovery		%		**79,3**	**85,7**						
Average recovery		%		**82,5**							
Moisture of recovered sample		%		**34,3**	**35,6**						
ACCRETION TESTING											
Initial weight chippings		g						30	30	30	30
Dry weight chippings		g						28	28	28	28
Correction factor - natural moisture		%						1	1	1	1
Exposure time		min						10	10	10	10
Initial weight chippings		g						30	30	30	30
Wet weight chippings		g						16	12	13	12
Final weight chippings		g						11	8	8	8
Accretion		%						**38**	**29**	**29**	**29**
Average accretion		%						**29**			
Moisture		%						**32**	**34**	**35**	**31**
STATIC SAG TEST											
MW		sg					1,1				
Volume aged mud		ml					350				
Free fluid on top		ml					13,5				
Separation		V/V%					3,9				
	Top	sg					1,015				
		sg									
	Middle	sg					1,012				
		sg									
	Bottom	sg					1,113				
Sag Index							**0,51**				
COMMENTS											

Dispersion: Only one dispersion parallel is standard procedure, but I did two, to learn the testing procedure properly.

Accretion and dispesion values in bold/grey are considered outliers and not included in the average calculation

1.2.5 FORMULATION 5: BASE + MEG

CONFIDENTIAL

SAMPLE INFO	
REQUEST NO	**Deepwater WBM**
FLUID SYSTEM	**WBM**
DESCRIPTION	**1.1sg**
TEST DATE	**22.03- 29.03**
TESTED BY	**MN**

SAMPLE COMPOSITION												
Sample			**5**									
Mixing volume		ml	**1000**									
Sea water		g/l	775,2									
Duotec NS	MIN 08-443	g/l	4									
Trol FL	MIN 08-269	g/l	15									
Kla Stop (UltrahibNS)	MIN 09-049	g/l	20									
MEG 09-038	MIN 10-038	g/l	200									
Barite	MIN 10-131	g/l	75,8									
EMI-2223	EXP 10-154	g/l	10									
Defoamer EMI-1705	MIN 08-319											
AGING CONDITIONS												
Period aged		HOURS		**16**	**16,00**	**16**	**16**	**16**				
Temperature		ºC		**100**	**100,00**	**100**	**100**	**100**				
Dynamic / Static		D/S	**BHR**	**D + Disp**	**D + Disp**	**D + Disp**	**D**	**D + S**	**ACCR**	**ACCR**	**ACCR**	**ACCR**
Sample		no		**1**	**2**	**3**			**1**	**2**	**3**	**4**
RHEOLOGY												
Temperature		ºC	**50**	**50**	**50**	**50**	**50**	**50**				
600 rpm		lbs/100ft²	47	49	48	48	44	43				
300 rpm		lbs/100ft²	34	37	37	37	33	33				
200 rpm		lbs/100ft²	29	33	32	32	29	28				
100 rpm		lbs/100ft²	22	26	25	26	23	23				
6 rpm		lbs/100ft²	9	13	12	12	11	11				
3 rpm		lbs/100ft²	8	11	10	10	9	9				
10 second gel		lbs/100ft²		11	11	10	10	9				
10 minute gel		lbs/100ft²		12	12	12	11	10				
Plastic Viscosity		cP	13	12	11	11	11	10				
Yield Point		lbs/100ft²	21	25	26	26	22	23				
FILTRATION - API												
API		ml					**2,7**					
OTHER												
pH			9,2	8,5	8,5	8,5	8,9	9,1				
Specific gravity		sg										
DISPERSION TESTING												
Initial weight chippings		g		30,0	30,0	30,0						
Dry weight chippings		g		29,4	29,4	29,4						
Correction factor - natural moisture		%		1,0	1,0	1,0						
Initial weight chippings (2-4 mm)		g		30,0	30,0	30,0						
Wet weight chippings : 1-2 mm		g		1,7	2,0	4,5						
Wet weight chippings : > 2 mm		g		40,2	41,4	33,4						
Final weight chippings		g		26,6	26,8	24,2						
Recovery		%		**90,3**	**91,0**	**82,3**						
Average recovery				**90,6**								
Moisture of recovered sample		%		**36,5**	**38,3**	**36,0**						
ACCRETION TESTING												
Initial weight chippings		g							30	30	30	30
Dry weight chippings		g							28	28	28	28
Correction factor - natural moisture		%							1	1	1	1
Exposure time		min							10	10	10	10
Initial weight chippings		g							30	30	30	30
Wet weight chippings		g							17	10	16	14
Final weight chippings		g							11	6	10	9
Accretion		%							**39**	**22**	**35**	**32**
Average accretion		%							**35**			
Moisture		%							**34**	**37**	**36**	**34**
STATIC SAG TEST												
MW		sg						1,1				
Volume aged mud		ml						350				
Free fluid on top		ml						1				
Separation		V/V%						0,3				
	Top	sg						1,009				
		sg										
	Middle	sg						1,011				
		sg										
	Bottom	sg						1,037				
Sag Index								**0,47**				

COMMENTS

Sag: Very little free fluid on top from the sag test. But suddenly some free fluid appeared in the middle of the cell.

Accretion and dispesion values in bold/grey are considered outliers and not included in the average calculation

1.2.6 FORMULATION 6: BASE + MEG + NaCl

SAMPLE INFO		
REQUEST NO	**Deepwater WBM**	CONFIDENTIAL
FLUID SYSTEM	**WBM**	
DESCRIPTION	**1.1sg**	
TEST DATE	**22.03- 29.03**	
TESTED BY	**MN**	

SAMPLE COMPOSITION												
Sample			**6**									
Mixing volume		ml	**1000**									
Sea water		g/l	775,2									
Duotec NS	MIN 08-443	g/l	4									
Trol FL	MIN 08-269	g/l	15									
Kla Stop (UltrahibNS)	MIN 09-049	g/l	20									
MEG 09-038	MIN 10-038	g/l	200									
Barite	MIN 10-131	g/l	75,8									
EMI-2223	EXP 10-154	g/l	10									
Defoamer EMI-1705	MIN 08-319											
RS-10475		g/l	20,7									
AGING CONDITIONS												
Period aged		HOURS		**16**	**16**	**16**	**16**	**16**				
Temperature		°C		**100**	**100**	**100**	**100**	**100**				
Dynamic / Static		D/S	**BHR**	**D + Disp**	**D + Disp**	**D + Disp**	**D**	**D+ S**	**ACCR**	**ACCR**	**ACCR**	**ACCR**
Sample		no		**1**	**2**	**3**			**1**	**2**	**3**	**4**
RHEOLOGY												
Temperature		°C	**50**	**50**	**50**	**50**	**50**	**50**				
600 rpm		lbs/100ft²	46	45	45	45	45	42				
300 rpm		lbs/100ft²	34	34	34	34	34	32				
200 rpm		lbs/100ft²	28	29	29	29	29	27				
100 rpm		lbs/100ft²	20	23	23	23	23	21				
6 rpm		lbs/100ft²	8	10	10	10	9	9				
3 rpm		lbs/100ft²	7	8	8	8	8	8				
10 second gel		lbs/100ft²		9	9	9	9	9				
10 minute gel		lbs/100ft²		10	10	10	10	9				
Plastic Viscosity		cP	12	11	11	11	11	10				
Yield Point		lbs/100ft²	22	23	23	23	23	22				
						6						
FILTRATION - API												
API		ml					**2,8**					
OTHER												
pH			9,1	8,7	8,6	8,7	8,8	9,0				
Specific gravity		sg										
DISPERSION TESTING												
Initial weight chippings		g		30,0	30,0	30,0						
Dry weight chippings		g		29,4	29,4	29,4						
Correction factor - natural moisture		%		1,0	1,0	1,0						
Initial weight chippings (2-4 mm)		g		30,0	30,0	30,0						
Wet weight chippings : 1-2 mm		g		1,6	1,3	1,7						
Wet weight chippings : > 2 mm		g		38,7	40,3	39,2						
Final weight chippings		g		25,2	26,2	25,7						
Recovery		%		**85,6**	**88,8**	**87,2**						
Average recovery		%			**87,2**							
Moisture of recovered sample		%		**37,4**	**37,1**	**37,2**						
ACCRETION TESTING												
Initial weight chippings		g							30	30	30	30
Dry weight chippings		g							28	28	28	28
Correction factor - natural moisture		%							1	1	1	1
Exposure time		min							10	10	10	10
Initial weight chippings		g							30	30	30	30
Wet weight chippings		g							9	12	11	14
Final weight chippings		g							6	8	7	9
Accretion		%							**20**	**28**	**25**	**33**
Average accretion		%								**27**		
Moisture		%							**35**	**33**	**36**	**34**
STATIC SAG TEST												
MW		sg						1,1				
Volume aged mud		ml						350				
Free fluid on top		ml						9,5				
Separation		V/V%						2,7				
	Top	sg						0,993				
		sg										
	Middle	sg						1,004				
		sg										
	Bottom	sg						1,018				
Sag Index								**0,46**				

COMMENTS

Sag: The free fluid on top from sag testing was partly a mix- not only free fluid..

Accretion and dispesion values in bold/grey are considered outliers and not included in the average calculation

1.2.7 FORMULATION 7: BASE + MEG + NaCl

SAMPLE INFO		
REQUEST NO	**Deepwater WBM**	CONFIDENTIAL
FLUID SYSTEM	**WBM**	
DESCRIPTION	**1.1sg**	
TEST DATE	**30.03- 06.04.**	
TESTED BY	**MN**	

SAMPLE COMPOSITION												
Sample			**7**									
Mixing volume		ml	**1000**									
Sea water		g/l	742,2									
NaCl	MIN 10-038	g/l	109,0									
Duotec NS	MIN 08-443	g/l	4,0									
Trol FL	MIN 08-269	g/l	15,0									
Kla Stop (UltrahibNS)	MIN 09-049	g/l	20,0									
MEG 09-038	MIN 10-038	g/l	200,0									
EMI-2223	EXP 10-154	g/l	10,0									
Defoamer EMI-1705	MIN 08-319											
AGING CONDITIONS												
Period aged		HOURS		**16**	**16**	**16**	**16**	**16 + 24**				
Temperature		°C		**100**	**100**	**100**	**100**	**100**				
Dynamic / Static		D/S	**BHR**	**D + Disp**	**D+ Disp**	**D+ Disp**	**D**	**D + S**	**ACCR**	**ACCR**	**ACCR**	**ACCR**
Sample				**1**	**2**	**3**			**1**	**2**	**3**	**4**
RHEOLOGY												
Temperature		°C	**50**	**50**	**50**	**50**	**50**	**50**				
600 rpm		lbs/100ft²	51	49	49	49	47	48				
300 rpm		lbs/100ft²	37	38	38	37	36	37				
200 rpm		lbs/100ft²	31	32	32	32	31	31				
100 rpm		lbs/100ft²	24	25	25	25	24	25				
6 rpm		lbs/100ft²	11	11	12	11	11	12				
3 rpm		lbs/100ft²	9	9	10	9	9	10				
10 second gel		lbs/100ft²		10	10	10	10	10				
10 minute gel		lbs/100ft²		12	11	13	12	13				
Plastic Viscosity		cP	14	11	11	12	11	11				
Yield Point		lbs/100ft²	23	27	27	25	25	26				
FILTRATION - API												
API		ml					4,1					
OTHER												
pH			9,1	8,8	8,7	8,8	9,2	9,1				
Specific gravity		sg										
DISPERSION TESTING												
Initial weight chippings		g		30,0	30,0	30,0						
Dry weight chippings		g		29,4	29,4	29,4						
Correction factor - natural moisture		%		1,0	1,0	1,0						
Initial weight chippings (2-4 mm)		g		30,0	30,0	30,0						
Wet weight chippings : 1-2 mm		g		2,9	3,0	3,4						
Wet weight chippings : > 2 mm		g		36,6	47,4	46,3						
Final weight chippings		g		29,4	30,3	29,5						
Recovery		%		**99,8**	**102,9**	**100,1**						
Average recovery		%			**100,0**							
Moisture of recovered sample		%		**25,5**	**39,9**	**40,7**						
ACCRETION TESTING												
Initial weight chippings		g							30	30	30	30
Dry weight chippings		g							28	28	28	28
Correction factor - natural moisture		%							1	1	1	1
Exposure time		min							10	10	10	10
Initial weight chippings		g							30	30	30	30
Wet weight chippings		g							13	21	13	17
Final weight chippings		g							9	14	9	11
Accretion		%							**30**	**48**	**30**	**38**
Average accretion		%							**33**			
Moisture		%							**34**	**33**	**34**	**35**
STATIC SAG TEST												
MW		sg						1,1				
Volume aged mud		ml						350				
Free fluid on top		ml						0,5				
Separation		V/V%						0,1				
	Top	sg						1,03				
		sg										
	Middle	sg						0,1315				
		sg										
	Bottom	sg						1,0345				
Sag Index								**0,47**				

COMMENTS

Sag: A lot of foam in the mud, impossible to get rid of it. Almost no free fluid on top.

Accretion and dispesion values in bold/grey are considered outliers and not included in the average calculation

1.2.8 FORMULATION 8: BASE + MEG + NaCl + RS- 10475

SAMPLE INFO		
REQUEST NO	**Deepwater WBM**	CONFIDENTIAL
FLUID SYSTEM	**WBM**	
DESCRIPTION	**1.1sg**	
TEST DATE	**30.03- 06.04.**	
TESTED BY	**MN**	

SAMPLE COMPOSITION												
Sample			**8**									
Mixing volume		ml	**1000**									
Sea water		g/l	742,2									
NaCl	MIN 09-038	g/l	109,0									
Duotec NS	MIN 08-443	g/l	4,0									
Trol FL	MIN 08-269	g/l	15,0									
Kla Stop (UltrahibNS)	MIN 09-049	g/l	20,0									
MEG 09-038	MIN 10-038	g/l	200,0									
EMI-2223	EXP10- 154	g/l	10,0									
Defoamer EMI-1705	MIN 08-319											
RS-10475		g/l	20,7									
AGING CONDITIONS												
Period aged		HOURS		**16**	**16**	**16**	**16**	**16 + 24**				
Temperature		ºC		**100**	**100**	**100**	**100**	**100**				
Dynamic / Static		D/S	**BHR**	**D + Disp**	**D+ Disp**	**D+ Disp**	**D**	**D + S**	**ACCR**	**ACCR**	**ACCR**	**ACCR**
Sample				**1**	**2**	**3**			**1**	**2**	**3**	**4**
RHEOLOGY												
Temperature		ºC	**50**	**50**	**50**	**50**	**50**	**50**				
600 rpm		lbs/100ft²	51	49	49	52	48	49				
300 rpm		lbs/100ft²	38	37	37	39	34	36				
200 rpm		lbs/100ft²	32	31	31	33	29	31				
100 rpm		lbs/100ft²	24	24	35	26	23	24				
6 rpm		lbs/100ft²	10	10	11	11	10	11				
3 rpm		lbs/100ft²	9	8	9	9	8	9				
10 second gel		lbs/100ft²		10	10	11	9	10				
10 minute gel		lbs/100ft²		12	11	12	11	12				
Plastic Viscosity		cP	13	12	12	13	14	13				
Yield Point		lbs/100ft²	25	25	25	26	20	23				
FILTRATION - API												
API		ml					6,3					
OTHER												
pH			9,0	8,6	8,7	8,6	9,4	9,3				
Specific gravity		sg										
DISPERSION TESTING												
Initial weight chippings		g		30,0	30,0	30,0						
Dry weight chippings		g		29,4	29,4	29,4						
Correction factor - natural moisture		%		1,0	1,0	1,0						
Initial weight chippings (2-4 mm)		g		30,0	30,0	30,0						
Wet weight chippings : 1-2 mm		g		2,8	3,4	2,5						
Wet weight chippings : > 2 mm		g		44,4	46,4	45,6						
Final weight chippings		g		29,6	29,8	29,2						
Recovery		%		**100,3**	**101,0**	**99,2**						
Average recovery		%			**100,0**							
Moisture of recovered sample		%		**37,4**	**40,2**	**39,4**						
ACCRETION TESTING												
Initial weight chippings		g							30	30	30	30
Dry weight chippings		g							28	28	28	28
Correction factor - natural moisture		%							1	1	1	1
Exposure time		min							10	10	10	10
Initial weight chippings		g							30	30	30	30
Wet weight chippings		g							13	19	12	13
Final weight chippings		g							9	12	8	9
Accretion		%							**31**	**43**	**28**	**30**
Average accretion		%								**30**		
Moisture		%							**34**	**35**	**34**	**36**
STATIC SAG TEST												
MW		sg						1,1				
Volume aged mud		ml						350				
Free fluid on top		ml						0,4				
Separation		V/V%						0,1				
	Top	sg						1,0535				
		sg										
	Middle	sg						1,043				
		sg										
	Bottom	sg						1,021				
Sag Index								**0,46**				

COMMENTS

Sag: A lot of foam in the mud, impossible to get rid of it. Almost no free fluid on top.

Accretion and dispesion values in bold/grey are considered outliers and not included in the average calculation

1.2.9 FORMULATION 9: CdId 161

SAMPLE INFO												
REQUEST NO	**Deepwater WBM**						CONFIDENTIAL					
FLUID SYSTEM	**WBM**											
DESCRIPTION	**1.1sg**											
TEST DATE	**18.04- 26.04**											
TESTED BY	**MN**											
SAMPLE COMPOSITION												
Sample			**9**									
Mixing volume		ml	**1000**									
Sea water		g/l	954,1									
Duotec NS	MIN 08-443	g/l	4,0									
Trol FL	MIN 08-269	g/l	15,0									
Kla Stop (UltrahibNS)	MIN 09-049	g/l	20,0									
Barite	MIN 10-131	g/l	97,4									
EMI-2223	EXP 10-154	g/l	10,0									
Defoamer EMI-1705	MIN 08-319											
Cdld 161		g/l	6,0									
AGING CONDITIONS												
Period aged		HOURS		**16**	**16**	**16**	**16**	**16 + 24**				
Temperature		ºC		**100**	**100**	**100**	**100**	**100**				
Dynamic / Static		D/S	**BHR**	**D + Disp**	**D+ Disp**	**D+ Disp**	**D**	**D + S**	**ACCR**	**ACCR**	**ACCR**	**ACCR**
Sample				**1**	**2**	**3**			**1**	**2**	**3**	**4**
RHEOLOGY												
Temperature		ºC	**50**	**50**	**50**	**50**	**50**	**50**	**50**	**50**	**50**	**50**
600 rpm		lbs/100ft²	40	40	40	40	36	36				
300 rpm		lbs/100ft²	30	32	31	32	28	29				
200 rpm		lbs/100ft²	26	28	28	28	25	25				
100 rpm		lbs/100ft²	20	23	22	23	20	21				
6 rpm		lbs/100ft²	9	11	10	10	9	9				
3 rpm		lbs/100ft²	8	9	8	9	7	7				
10 second gel		lbs/100ft²		9	9	9	7	7				
10 minute gel		lbs/100ft²		10	10	10	8	8				
Plastic Viscosity		cP	10	8	9	8	8	7				
Yield Point		lbs/100ft²	20	24	22	24	20	22				
FILTRATION - API												
API		ml					3,7					
OTHER												
pH			9,0	8,6	8,5	8,6	9,0	8,8				
Specific gravity		sg										
DISPERSION TESTING												
Initial weight chippings		g		30,0	30,0	30,0						
Dry weight chippings		g		29,4	29,4	29,4						
Correction factor - natural moisture		%		1,0	1,0	1,0						
Initial weight chippings (2-4 mm)		g		30,0	30,0	30,0						
Wet weight chippings : 1-2 mm		g		4,2	2,8	3,6						
Wet weight chippings : > 2 mm		g		32,3	28,3	31,1						
Final weight chippings		g		22,0	20,4	21,4						
Recovery		%		**74,6**	**69,1**	**72,8**						
Average recovery		%			**73,7**							
Moisture of recovered sample		%		**39,7**	**34,4**	**38,3**						
ACCRETION TESTING												
Initial weight chippings		g							30	30	30	30
Dry weight chippings		g							28	28	28	28
Correction factor - natural moisture		%							1	1	1	1
Exposure time		min							10	10	10	10
Initial weight chippings		g							30	30	30	30
Wet weight chippings		g							9	15	20	15
Final weight chippings		g							5	9	13	10
Accretion		%							**19**	**32**	**46**	**34**
Average accretion		%							**33**			
Moisture		%							**39**	**38**	**35**	**36**
STATIC SAG TEST												
MW		sg						1,1				
Volume aged mud		ml						350				
Free fluid on top		ml						130				
Separation		V/V%						37,1				
	Top	sg						1,02				
		sg										
	Middle	sg						1,046				
		sg										
	Bottom	sg						1,2425				
Sag Index								**0,56**				
COMMENTS												
Sag: A lot of free fluid on top (130 ml), with density of 0,969												
Accretion and dispesion values in bold/grey are considered outliers and not included in the average calculation												