Optimization of Laboratory apparatus for research of anorganic and organic deposits and selection of scale and paraffin inhibitors

Master Thesis

BSc Christian Horner

Submitted to the Department of Petroleum Engineering at the Mining University of Leoben, Austria

Leoben, 16.03.2009

Eidesstattliche Erklärung

Ich erkläre an Eides statt, dass ich diese Arbeit selbstständig verfasst, andere als die am Ende der Arbeit angegebenen Quellen und Hilfsmittel nicht benutzt und mich auch sonst keiner unerlaubten Hilfsmittel bedient habe.

Affidavit

Herewith I declare in the lieu of oath that this master thesis is entirely of my own work using only literature cited at the end of this volume.

_____

(Christian Horner)

Acknowledgements

Table of Contents

List of Figures

List of Tables

Kurzfassung

Da es bei paraffinhaltigen Rohölen sowohl bei der Förderung als auch beim Transport zu Ablagerungen und damit verbundenen Druckabfällen und Verstopfungen kommen kann, welche längere Stillstände und Kosten verursachen, ist es notwendig sich mit diesem Thema zu beschäftigen und Lösungen zu erarbeiten.

Zu diesem Zweck wurde bei der Firma OMV Exploration und Production ein Wax-Loop Apparat angeschafft. Dieser soll vorher angewandte Messmethoden, wie Cold-Finger Tests und Rheometermessungen, zur Bestimmung der Wirksamkeit von Wax-Inhibitoren ergänzen oder ersetzen, da die Testbedingungen besser an den jeweiligen Anwendungsfall anpassbar sind. Ziel der Masterarbeit war den Apparat in Betrieb zu nehmen, Testeinstellungen zu optimieren und darauf folgend chemische Wax-Inhibitoren auf ihre Wirksamkeit in bestimmten Anwendungsfällen zu testen. Des Weiteren wurde eine Arbeitsanweisung erstellt um künftige Tests einfacher durchführen zu können.

Der zweite Teil der Masterarbeit beschäftigt sich mit einem Scale-Loop Apparat, der zum Testen von chemischen Scale-Inhibitoren bestimmt ist. Analog zu den Problemen durch paraffinhaltige Öle, kann es hier zu anorganischen Ausfällungen von Lagerstätten- und eingepressten Wässern kommen, welche Leitungen verstopfen können und zu zusätzlichen Druckverlusten führen. Auch hier musste der Apparat in Betrieb genommen werden, und Testparameter für bestimmte Anwendungsfälle optimiert werden. Danach wurden verschiedene Scale-Inhibitoren auf ihre Wirksamkeit geprüft. Für die Verwendung des Scale Loop Apparates wurde auch eine Arbeitsanweisung erstellt.

Ein weiterer Punkt ist es Schwachstellen im Aufbau der Apparate zu identifizieren, die Einsatzgrenzen aufzuzeigen, sowie Verbesserungen vorzuschlagen.

Abstract

Because of the possible precipitation and deposition of organic waxes while the production and transport of paraffinic crude oils, plugging and pressure losses can occur, which cause longer workovers and additional costs. Therefore, it is necessary to investigate this topic and to develop solutions.

For this purpose, OMV Exploration and Productions purchased a wax-loop apparatus. Due to the better adjustability to certain applications, this apparatus should partly or completely replace former measurement methods of the effectiveness of wax inhibitors, like cold finger tests or rheometer measurements. The aim of this master thesis was to place the new device into operation, to optimize the test parameters and to test chemical wax inhibitors with certain parameters and crude oils. Further, a working instruction was gathered, to alleviate future measurements.

The second part of this master thesis deals with a scale loop apparatus, that was also purchased shortly before and which is used to test chemical scale inhibitors. Similar to the problems with paraffinic crude oils, anorganic deposits can be built from reservoir or injection water due to changing environment conditions. The consequences are also possible plugging of tubes or an increase in the pressure drop. This apparatus had to be placed into operation too and test parameters had to be optimized. After that, different scale inhibitors were tested. On the basis of this, a working instruction was written.

Another part of the work is to identify weak points of the build up of the two apparatus, to determine operational boundaries and to suggest improvements.

Abbreviations

a.k.a.	also known as
CCN	Critical Carbon Number
OSDC	Organic Solids Deposition and Control
HPLC	High performance liquid chromatography
PPD	Pour Point Depressant
PPE	Paraffin Phase Envelope
WAT	Wax Appearance Temperature
WDT	Wax Disappearance Temperature
w/	with
w/o	without

Introduction

Because of the composition of some crude oils, precipitation and deposition of paraffin waxes can occur if the temperature of the fluid decreases under a certain level. The precipitation changes the rheological properties of the fluid, such as viscosity. If the precipitated paraffin wax deposits in the reservoir, in the well, in flow lines or in a tank, actions are necessary, which may be time consuming and costly, because a production loss is the outcome. A possibility to overcome these downtimes is to use a continuous chemical inhibition. The used chemical should be easy to apply for the specific circumstances and cost efficient. To find an adequate wax inhibitor laboratory measurements have to be done, because it does not make sense to try every available chemical in the field. The closer the boundary conditions of the test are to the conditions in the field, the better the results will be. Therefore, so-called bench scale flow loops are available on the market now, which allow testing of wax inhibitors in field like conditions. Unlike to static tests like the Cold Finger Test or measurements on a rheometer, the fluid is pumped in a loop that can be cooled to simulate different environments. Because this technique is relatively new, almost no practical experience is available in the literature. The aim of this work is to put the apparatus into operation and to find proper parameters for the test runs, so that incoming oil samples can be tested with various inhibitors under applicable conditions. Another important factor is the addition of the wax inhibitor, which will also be researched.

The second part deals with anorganic precipitations and deposits. Although the scaling has a different background, the problems, if it occurs, are quiet similar. An anorganic precipitation can be found if two incompatible waters are mixed, which can occur when water is injected to boost productivity of a reservoir, or when the reservoir water itself is exposed to temperature or pressure changes. To research this issue it makes sense to use dynamic tests instead of former used static ones, because the process itself also takes place in a dynamic environment. The apparatus used is similar, as the one for wax precipitation. To find feasible parameters for future tests, solutions, which produce a carbonate scale, will be used, because carbonate scale is the most common one in the fields of OMV. The aim for the second part is, as for the first one, to define parameters for testing, a method to prepare the samples and to write a working instruction for everyday use.

1   Organic Deposits

Because the crystallization, deposition and the methods to remove paraffin wax in pipes are described in several works done before, only a short overview of these topics will be given here.

1.1   Crude oil Composition and Characterization of Paraffin Wax

The composition of crude oil varies with the type of oil, with the location of the reservoir and from well to well from one single reservoir. There are hydrocarbon components and non-hydrocarbon components.

The hydrocarbon components can be divided into the following three classes:

- Paraffin a.k.a. Alkanes

  Are saturated hydrocarbons with straight chains, called normal paraffin, or with branched chains, called isoparaffin.

- Cycloalkanes a.k.a. Naphtenes

  Are saturated hydrocarbons too, but they contain one or more rings, with possible side chains. Naphthenes are stiff and bulky and tend to hinder the wax nucleation and growth process.

- Aromatics and NSO Compounds

  Aromatics contain one or more aromatic nuclei.

  NSO compounds are as the name says:

    - Sulphur Compounds

    - Oxygen Compounds

    - Nitrogen Compounds (1), (2)

Based on the proportion of these classes a hydrocarbon fluid can be classified as shown in Figure 1.
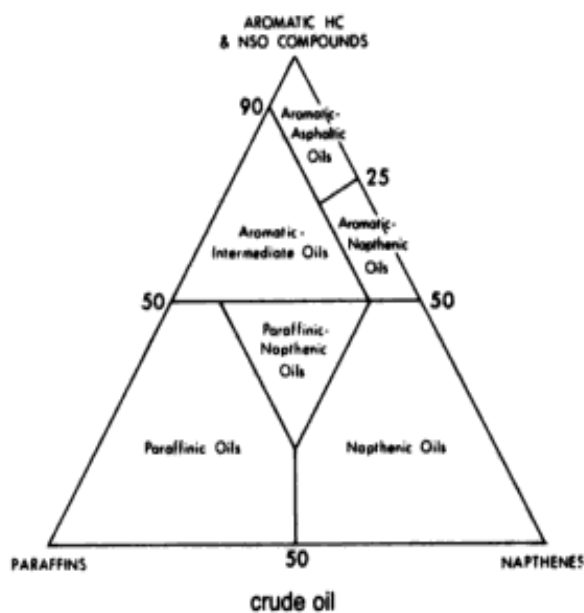
Figure 1: Classification of hydrocarbon fluids(2)

The average weight percent of the compounds of a crude oil can be seen in Table 1.

Table 1: Crude oil Composition (2)

Compound	Weight %
Carbon	84 – 87
Hydrogen	11 – 14
Sulphur	0,06 – 8
Nitrogen	0,02 – 1,7
Oxygen	0,08 – 0,14
Metals	0 – 0,14

Structural formulas of different paraffins are shown in Figure 2. The empirical formula of paraffin is C_nH_{n+2}. Paraffin waxes consist predominantly of normal (straight chain) paraffin with more than 15 carbon atoms and almost no isoparaffin. (3)
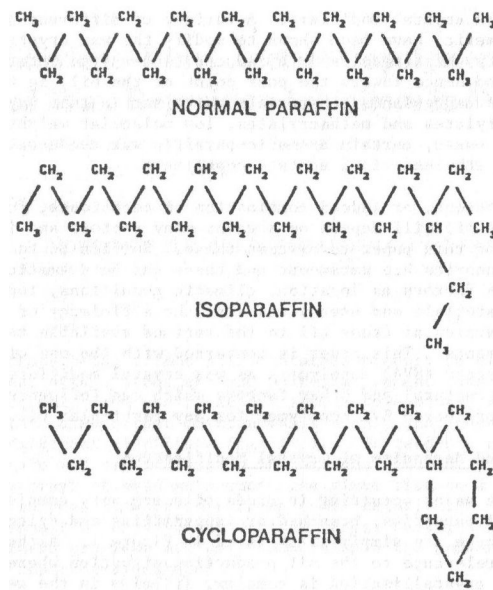
Figure 2: Structural formulae of paraffin

Asphaltenes

Another component in crude oil may be asphaltenes, which can precipitate too.

The literature reports that asphaltenes can prevent, but also facilitate paraffin depositions, dependent on their concentration, size and character.

As for paraffin an asphaltene flocculation envelope can be drawn. As seen in Figure 3 the flocculation depends on the temperature and on the pressure.(4)
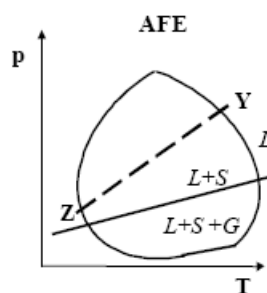
Figure 3: Asphaltene Flocculation Envelope(4)

While asphaltene molecules are polar and therefore have polar or hydrogen bond type molecular interactions, paraffin wax molecules are a-polar and have London dispersion type or van der Waals interactions.(5)

The nonhydrocarbon components in petroleum are:

- Porphyrins

- Metallic Constituents

- Nondistillable Constituents (1)

1.2   Precipitation, Crystal Growth and Deposition

The wax precipitation and deposition process can be divided into three stages: the separation of wax from the liquid phase, the growth of the wax crystals and the deposition. To overcome the problem one of the three stages has to be controlled. The first step also called nucleation starts as soon as the liquid is cooled below the wax appearance temperature. The molecular motion energy gets impaired and the randomly distributed molecules in the liquid move together and build clusters of directed chains. Paraffin molecules adhere and unpin to these clusters until a critical size is reached and the clusters become stable.

The so built nuclei are only stable below the wax disappearance temperature (WDT), above this temperature they are separated by the thermal motion.

The second step, the so-called growth process, begins when the stable nuclei are formed and the temperature is kept below the WAT. More wax molecules connect to the nucleation sites and the wax crystal grows.

The third step is the deposition to surfaces and the growth of the deposit, due to counter diffusion, which means that wax molecules diffuse into the deposit and oil molecules out. This changes the properties of the deposit over the time.(6)

There are several negative effects of precipitation and deposition of paraffin wax:

- Changed flow behaviour from Newtonian fluid to non-Newtonian (severely increase of viscosity)

- Increase of the surface roughness in the pipes

- Reduction of the internal cross section of pipes
  (in the worst case total plugging)

- Limited functionality of equipment due to deposits

- Pigging more often required (5)

- And therefore production losses due to interventions

Figure 4: Wax deposit in a pipe

1.3 Handle of Wax Deposits and Chemical Inhibition

Sometimes deposition can be avoided by heating, thermal insulation or other sanctions, but usually chemical inhibition is the approach of choice.

To prevent or remove wax deposition three classes of chemicals are used.

Wax crystal modifiers

This type of paraffin inhibitors is used to lower the pour point and the apparent viscosity. Therefore crystal modifiers are also called Pour Point Depressants. Crystal modifiers are built into the wax crystals and modify the growth of the crystals and their surface characteristics as seen in Figure 5. The acetate group of the inhibitor adds to the wax and disturbs further growth of the paraffin molecule. In modelling wax precipitation and deposition, the effect of pour point depressants can be seen as lowering the melting point of wax components for a certain range of molecular weights.
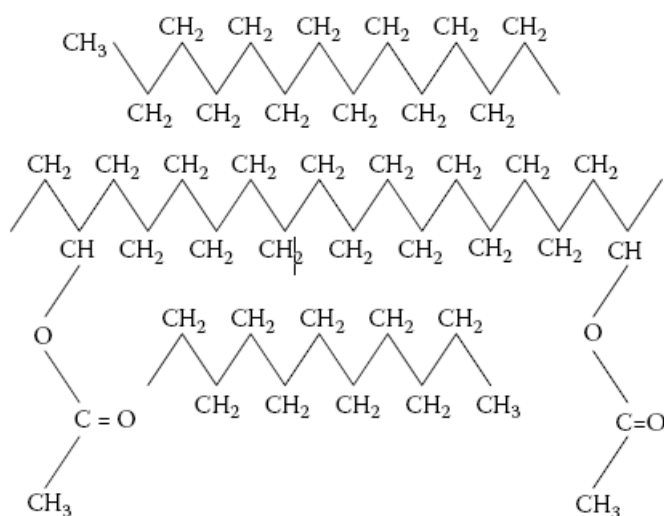
Figure 5: Crystal modifier mechanism (7)

They mainly consist of polyethylene waxes, aromatic paraffin condensation products and ethylene vinyl acetate copolymers. The way in which pour point depressants work is believed to be a combination of different mechanisms. These mechanisms are nucleation, co-crystallisation with the paraffin wax and absorption. (3)

Due to the working mechanisms of a PPD it only makes sense to add it to the oil when its temperature is above the WAT.

Dispersants

Dispersants are surface-active agents, they absorb to the growing wax crystals and prevent the already formed paraffin crystals from agglomerating together and therefore get a high enough mass to fall out of the liquid and deposit. Another mode of operation is to water wet the flow lines and pipelines to lower the tendency of wax crystals to ad here. The mode of operation can be seen in Figure 6. If they are formulated in organic solvents, they can be used in crudes with low water cut. Otherwise, if they are aqueous based, a pre-condition is the presence of water in the liquid stream. Chemicals of this class are for example poly-olesters and amine ethoxylates. (8)

An advantage of this type of inhibitor is that it can be used, if the temperature of the oil is below the WAT.
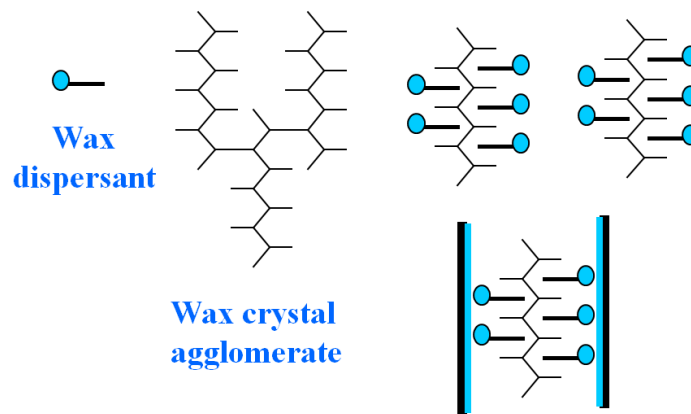
Figure 6: Wax dispersant mechanism (9)

Paraffin Dissolvers

Paraffin dissolvers, as Xylene, Toluene or Diesel, are used to dissolve already built paraffin wax.

The chemical of choice is a Pour Point Depressant, if continuous treatment is possible. It is cost efficient, because there are no downtimes for interventions. It lowers the viscosity, the pour-, and the yield point and it reduces the paraffin deposition. A disadvantage might be the high pour point of the PPD itself.

1.4 Factors influencing wax deposit formation

The solubility and therefore precipitation and deposition of specific n-paraffins depends on many factors, mainly these are:

- Actual temperature and temperature history
- Flow rate
- Pressure
- Composition of the oil (portion of the lighter components, molecular interaction)
- Its carbon number
- Surface characteristics

Temperature

When the crude oil is cooled, paraffin waxes start to crystallize and to deposit on cool surfaces, because the solubility of wax decreases with lower temperatures. The temperature difference between the fully liquid oil and the tube is believed to be main driving force for wax deposits, in detail the radial temperature gradient along the deposit itself. This temperature gradient causes a radial concentration gradient, because of molecular diffusion. (10)
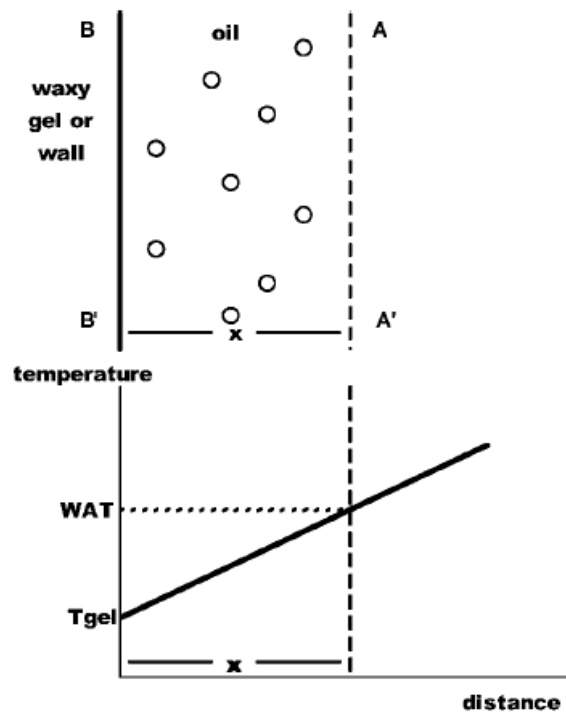
Figure 7: Temperature and Wax Concentration gradient(11)

In this figure, BB´ represents the boundary between the liquid and the already deposited wax or the wall of the tube. AA´ is the boundary where the liquid temperature falls under the wax appearance temperature, therefore on the right side of AA´ no precipitation occurs, but to the left side wax crystals are built. (11)

As the deposit is built over time, the temperature gradient is shifted towards the center of the pipe, because the deposit has an insulating behaviour. The change of temperature at a specific distance from the center of the pipe can be seen in Figure 8, which is a simulation for a time period of 30 minutes and 10 days.
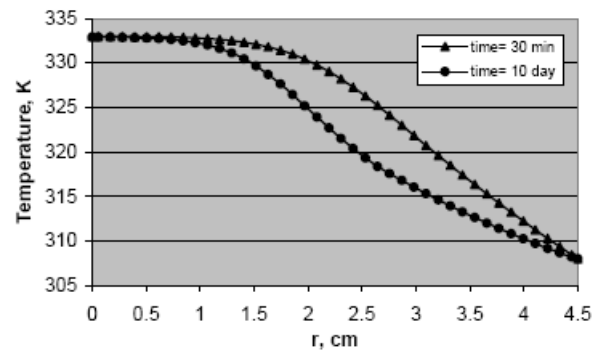
Figure 8: Temperature vs. Radius (12)

The insulating effect of a wax deposit can also be seen in Figure 9. After 5 days of flowing, the deposit is small and therefore the temperature decreases significant over distance. After 50 days in production, a bigger wax layer has been built at the pipe surface, so the insulation is better and the temperature does not decrease as much as after 5 days.
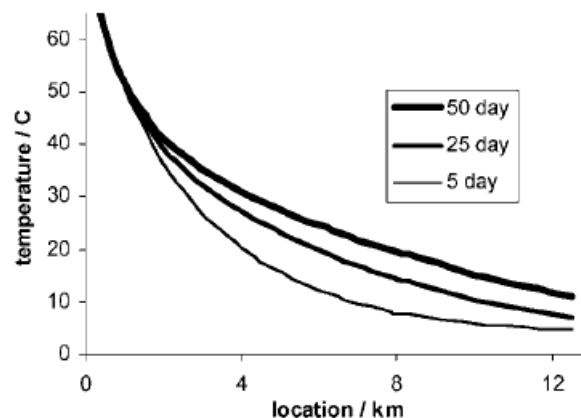
Figure 9: Temperature vs. Axial Location for different Times(11)

A possibility to counteract a possible deposition is to insulate the pipes. Figure 10 shows a simulation where an oil is flowing through two pipes, from which one has a better insulation than the other. This can have a great impact on the pumpability of the fluid. While the temperature of the liquid in the pipe with the better insulation clearly stays above the stock tank oil pour point, the temperature of the fluid in the pipe with the poorer insulation falls under the live oil pour point, which means that pumping problems will occur.
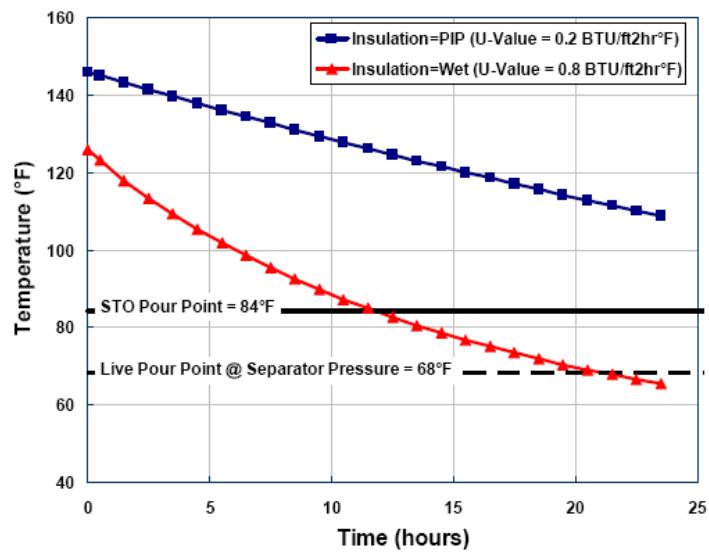
Figure 10: Temperature Profile w/ and w/o insulation(12)

Flow rate

Another parameter with a big influence on the wax deposition process is the flow rate and as a result the rate of shear. Some literature showed that independent of laminar or turbulent flow, the amount of deposited wax increases with lower flow rates and decreases with higher ones. The lower deposition in higher flow rates can be explained due to the fact, that flowing liquid shears off the soft deposit layers, which are in contact with the stream. Due to the disturbed nucleation process at high shear conditions, the feasibility of wax elements to solidify is reduced.(10)

Low flow rates and laminar flow on the other hand even can support the deposition building, as the paraffin molecules tend to be in line with the flow and aligned to each other, which can speed up the nucleation and growth process.. Another point is that due to the radial velocity gradient, precipitated particles are moved towards the pipe wall.(6)

This behaviour can be seen in Figure 11.
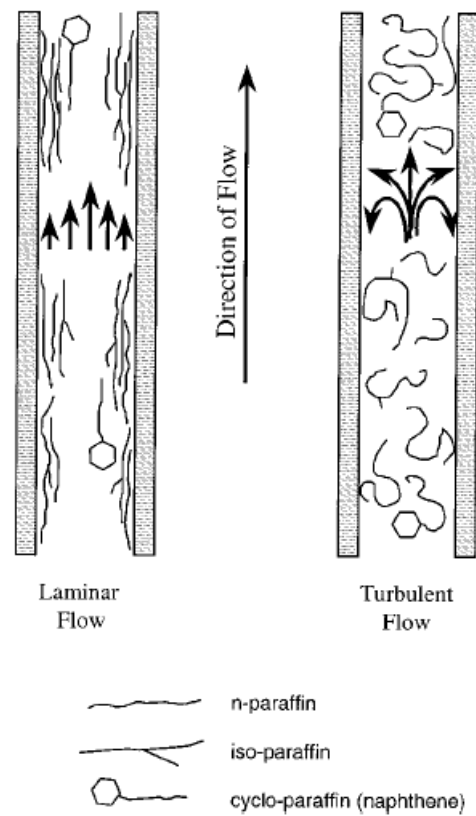
Figure 11: Effect of Flow regime for paraffin deposition(6)

Another issue is that a higher fluid velocity lowers the detention time in the line. This means a higher temperature of the liquid after a certain distance and a higher energy conservation.
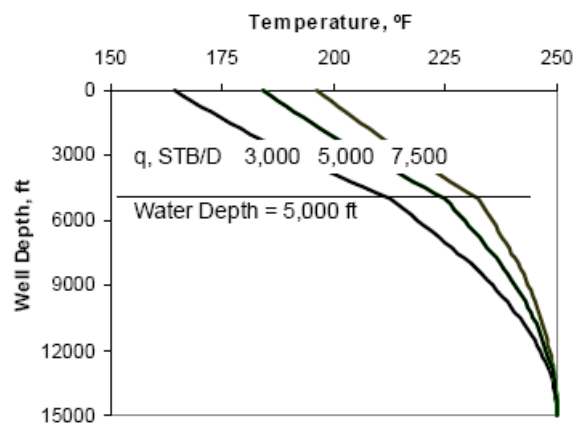
Figure 12: Temperature dependence on flow rate

Figure 12 shows the temperature over the depth of a deepwater well depending on the flow rate.(4)

Pressure

Beside the temperature, the pressure environment is also an important factor for the formation of paraffin waxes.
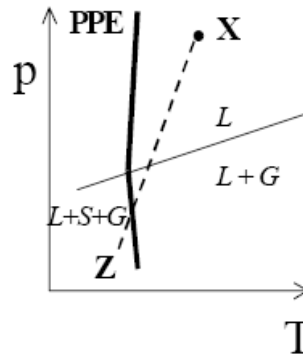
Figure 13: Paraffin Phase Envelope (4)

Figure 13 shows a paraffin phase envelope (PPE). Point X in the graph represents the condition of the fluid downhole in a well. Point Z the surface conditions. As the oil flows up the wellbore the pressure and the temperature decline.

Crossing the L, L+G border the single-phase oil gets to liquid and gas, before it intersects the solid envelope, where paraffin precipitation starts.

Not only is the wax appearance temperature affected by the pressure, but also the pour point and the gel strength.(Table 2)

Table 2: Dependence of WAT, Pour pint and gel strength of the pressure

Press (psia)	WAT (°F)	Pour Point (°F)	Gel Strength (Pa)
700	116	65	38
300	-	65	-
100	120	68	42
STO	139	84	142

The dependence of the gel strength from the pressure is also an issue in restarting a pipeline in the case of a shut in, where the flow rate is zero and the temperature is below the pour point. Under these conditions the content of the line gels.

If the line is held under pressure, the pressure necessary for restart is lower as it would be without.
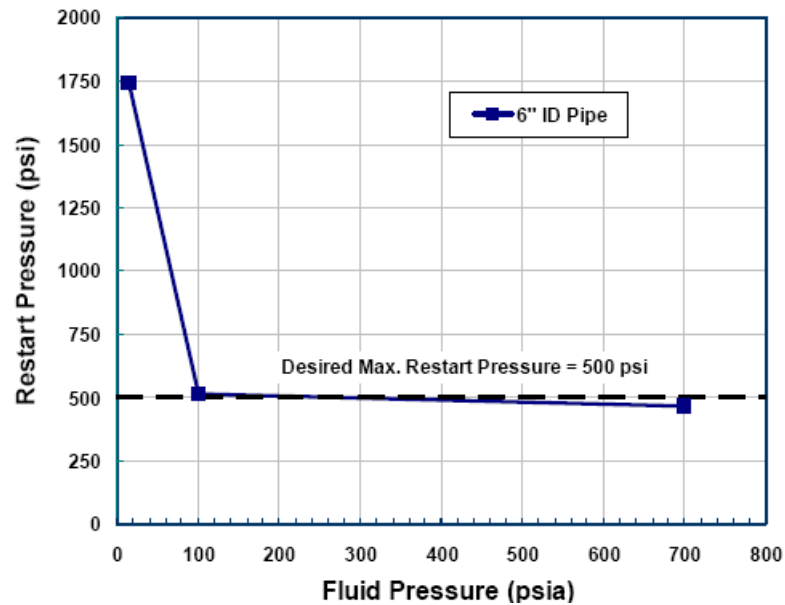
Figure 14: Fluid Pressure vs. Restart Pressure (6)

Surface characteristics

Surface properties, which affect the deposition of wax, are the surface wettability and the roughness of the pipe.(10)

Aging

Aging of a wax deposits means that the composition changes with time and therefore the depositions properties change, especially the hardness. To describe this process a critical carbon number was defined. Paraffins with a carbon number lower than the CCN diffuse out of the wax deposits and heavier ones diffuse in the deposit.(10)

Another process that causes the aging process might be the Ostwald ripening, which describes that while a precipitation larger particles grow and distend material from smaller ones, because of energetic issues.(11)

Beside this, also the solid/liquid ratio in the deposit changes over time. The fraction of the solids over the radius of the pipe can be seen for different time steps in Figure 15.
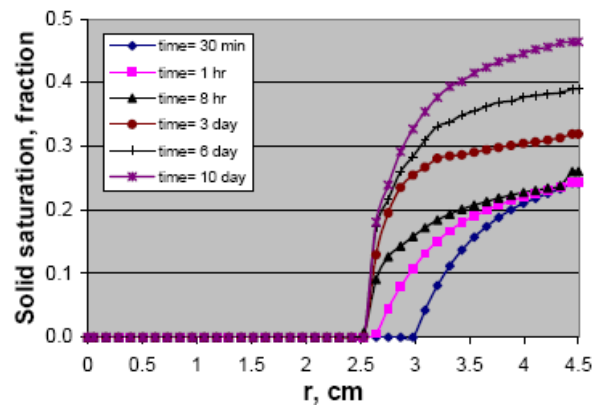
Figure 15: Solid fraction vs. radius, time dependence(12)

1.5 Wax and Oil Properties and their Measurement

Relevant properties of crude oil concerning paraffin wax are:

- Cloud Point

  "The temperature at which wax crystals first start to form in a crude oil. Wax appearance temperature (WAT) and wax precipitation temperature (WPT) are other synonyms." (13)

- Pour Point

  "The temperature at which a fluid ceases to pour. The pour point is established as that temperature at which oil ceases to flow when the sample is held at 90 degrees to the upright for five seconds." (14)

- Viscosity

  The viscosity is a measure for the inner resistance of a fluid against flowing, and is defined trough the resistance against a compression or shear stress.

  It is used to predict pressure drops in lines.

- Gel Strength

  The gel strength is the pressure required to break down a plug of wax gel under no flow conditions.(15)

  It is used to estimate the restart pressure of a shut in pipeline, where a wax gel has been formed.

Not only is the wax appearance temperature an important property, but also the amounts of precipitating wax at a given temperature.

In principle, there are two measurement techniques available, which can measure the wax precipitation and/or deposition. It is possible to use static techniques as the cold finger test or microscopy or dynamic techniques as the cylindrical Couette Taylor device or a dynamic tube-blocking test. Another possibility is to use rheometers to conclude from the viscosity behaviour on wax building tendencies.

Wax Appearance Temperature

In this work, the Wax Appearance Temperature (WAT) has been measured with a rotational viscosimeter. The measuring system has been described in detail in the literature, so only a short overview about this apparatus is given here.

It mainly consists of an inverted cone that rotates with a specific gap over a plate.
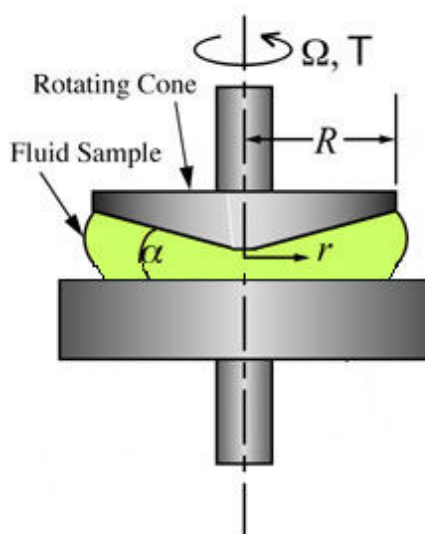
Figure 16: Principle Rotational Viscosimeter (16)

The lower plate can be heated or cooled, but at setting the parameters for a test run, it is necessary to not exceed the cooling or heating rates given by the manufacturer to get proper results.

Two test principals are possible:

- Constant shear stress test

  The shear stress is constant by applying a constant torque to the rotating cone and the shear rate is measured at each temperature point.

- Constant shear rate

  The shear rate is constant by rotating the cone at a constant speed and the shear stress is measured at each temperature point.

As outlined in a previous work it is favourable to work with constant shear stress, because a constant, too high shear rate can destroy an already built wax network while testing.

A constant torque of 115 µNm was used at the tests and the apparatus delivered the shear rate and the calculated dynamic viscosity.

Above the WAT the rheological behaviour of crude oil is like a Newtonian fluid, which means the viscosity is a function of temperature in laminar flow. Below the WAT it differs from this behaviour, the viscosity is also shear rate dependent. Therefore it is possible to determine the wax appearance temperature, it is at the point where the plot starts to differ from the Guzman-Andrade equation:

$$\mu = A * e^{\frac{Ea}{R*T}}$$

Where µ is the Newtonian dynamic viscosity, A is a constant dependent on the entropy of activation of flow, R is the universal gas constant ant T the temperature. A and Ea can be considered constant over a limited temperature range. Below the WAT the fluid does not behave like a Newtonian any more. Ea and A cannot be taken as constants any more, now they are functions of the shear rate or shear stress. The procedure to determine WAT can be seen in Figure 18. (17)

Figure 17: Rheometer by Anton Paar
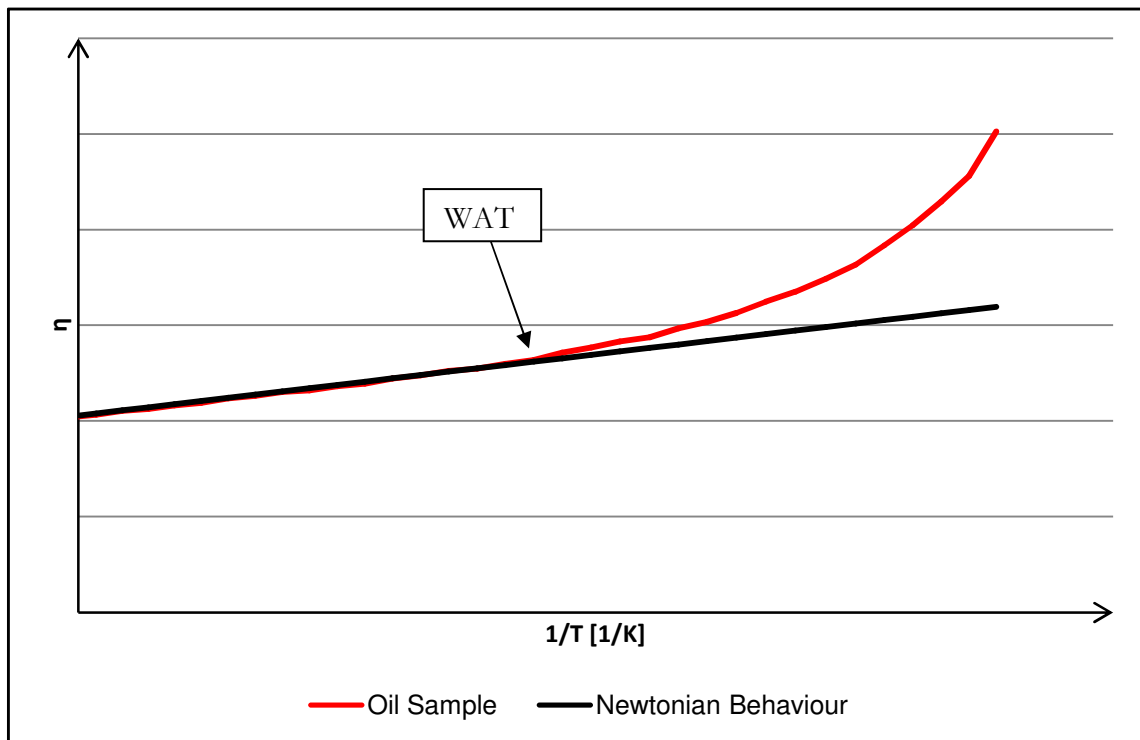
Figure 18: Determination WAT

On a plot with the axis $1/T$ and η the slope of η on a logarithm axis is Ea/R in the Newtonian region. As soon as the first crystals fall out of the liquid, Ea will increase and therefore the slope will be higher.

If a trend line is drawn for the Newtonian region and extrapolated to lower temperatures the WAT can be clearly seen.

Using a rheometer or the differential scanning calorimetry method to determine the WAT delivers different values, because only the effects of crystallisation are measured. The most accurate way to determine the WAT might be with microscopy.

Pour Point

The pour point or no flow point was measured with the Pour Point Tester PPT 45150 by PSL Systemtechnik. The measurement is according to ASTM D5985 (rotational method), with accuracy of $\pm$ 0.1 °C.

The working principle can be seen in Figure 19. The first step is to fill a sample cup with the assay that then rotates wit 0.1 rounds per minute. A temperature sensor which is dipped in the sample has a certain resistance to move. If the viscosity of the sample in-

creases, because the temperature is lowered, and the pour point is reached the sensor is moved from its original position and triggers a switch. (18)
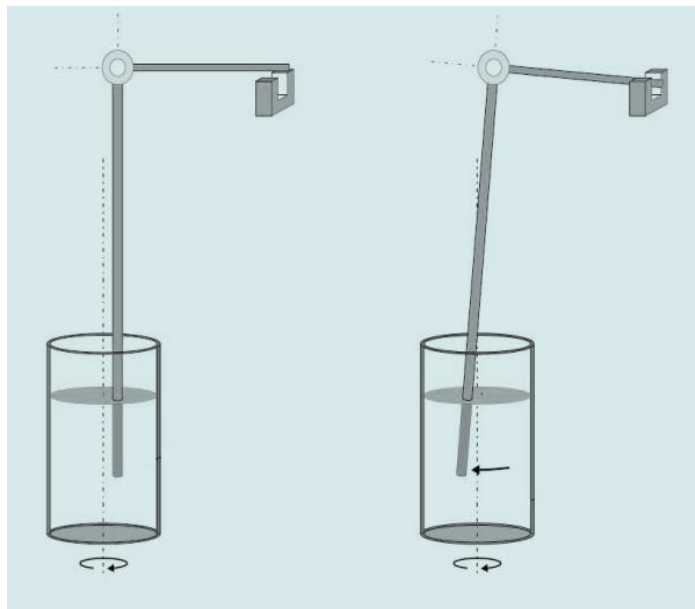
Figure 19: Scheme pour pint test (18)
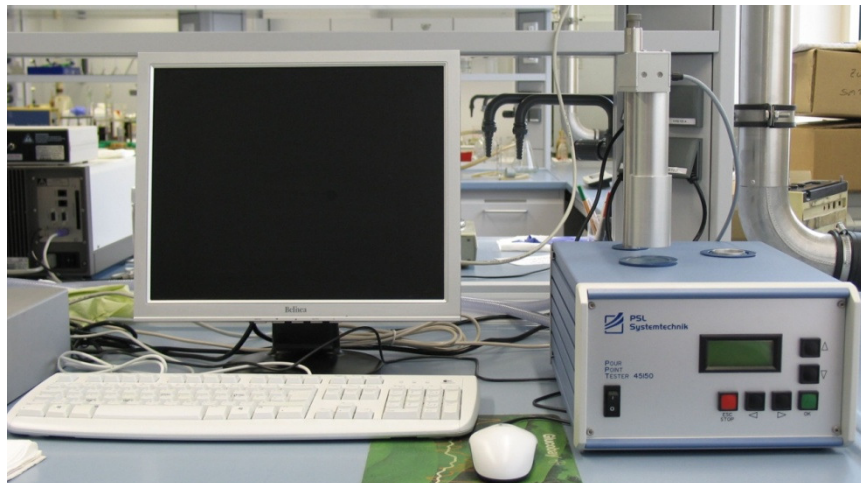
Figure 20: Pour point tester by PSL Systemtechnik

Cold Finger Test

The Cold Finger test can determine the amount of wax deposited on a surface colder than the oil temperature and below the WAT. A "Cold Finger", mostly a stainless steel cylinder or plate, which can be cooled, is dipped into the oil, that has to be at a temperature where the wax is dissolved. To get meaningful results the temperatures of the Cold Finger and the oil should be similar to the field conditions. After a specific time the steel cylinder is removed from the oil and weighted. The weight difference gives the amount of wax that has deposited on the cylinder. In Figure 21 Cold Fingers can be seen after removing them from the oil. Because the flow rate and the rate of shear in a tube also has a great influence on the wax depositions on the walls this method can deliver only results, which are hardly comparable to field conditions.(5)

Figure 21: Static Wax Deposition Apparatus (5)

A better approach is to measure wax deposition and its inhibitions in dynamic apparatus. One possibility is to use a Couette Taylor device. A schematic of this device is shown in Figure 22.
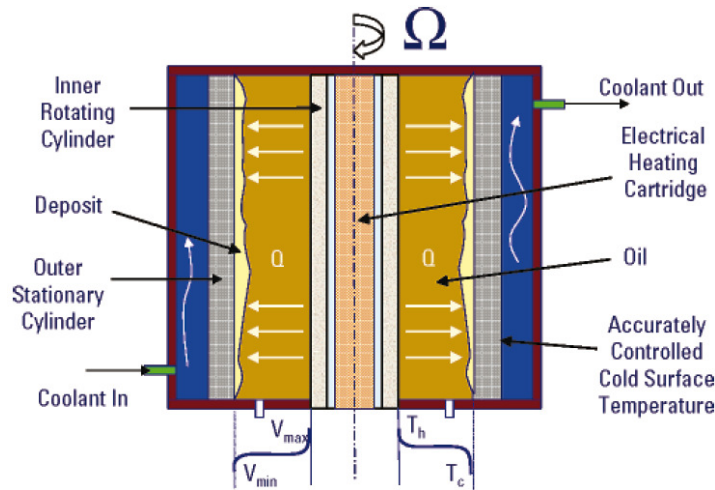
Figure 22: Scheme OSDC deposition cell (19)

In contrast to a flow loop the OSDC cell is based on the flow between rotating cylinders. It is built of an rotating inner cylinder and a static outer one. The inner cylinder acts like a pump and the outer cooled cylinder simulates the pipe wall. The whole cell can be pressurized to reproduce field conditions. Although the geometry of this cell is not similar to a pipe flow, a similitude is given, as Figure 23 shows.

Table 3. Similitude between CT and Pipe Flows

parameter	pipe	Couette−Taylor
Reynolds number[34]	$Re = \dfrac{uD_e}{\nu}$	$Re = \dfrac{u(r_o - r_i)}{\nu}$
wall shear stress[35,36]	$\tau_\omega = \dfrac{1}{8}\rho f_r u^2$	$\tau_\omega = \dfrac{1}{8}\rho \Psi u^{1.8}$
Nusselt number[37,38]	$Nu = A Re^a Pr^b$	$Nu = A' Re^{a'} Pr^{b'}$
dimensionless[6] $(\Delta P, G)$	$\Delta P = C_1 Re^{(3/2)+\alpha_1} + C_2 Re^{2+\alpha_2}$	$G = C_1' Re^{(3/2)+\alpha_1'} + C_1' Re^{2+\alpha_2'}$
friction factor[4,39]	$\dfrac{1}{\sqrt{f_r}} = C_1 \log(Re\sqrt{f_r}) + C_2$	$\dfrac{1}{\sqrt{f_r}} = C_1' \log(Re\sqrt{f_r}) + C_2'$
hydrodynamic boundary layer[40,41]	$\delta_p = 91 D_e \dfrac{\ln(Re)}{Re}$	$\delta_{CT} = 3.8(r_o - r_i)\dfrac{\ln(Re)}{Re^{0.8}}$

Figure 23: Formulas Couette Taylor and pipe flow (19)

The wax flow loop is described in detail in Chapter 1.6.

Water Content

To specify the amount of water in the crude oil samples a Dean-Stark apparatus was used. First 100ml of crude is mixed with 100ml of solvent naphtha in an extraction pot. The pot is heated to 350 °C. During the heating, the vapour from the mixture travels through the distilling trap in the water-cooled condenser, where it gets liquid again. The liquid flows down in the trap and by the overrun the lighter phase flows back to the extraction pot. In the trap, the condensate is separated due to the density difference between the water and the solvent. If the amount of water is greater than the volume of the trap, it has to be drained regularly, because otherwise the water will flow back to the pot with the solvent.
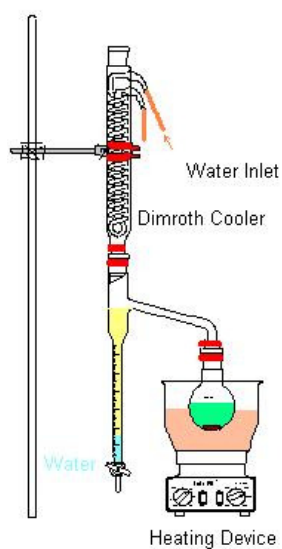
Figure 24: Scheme Dean-Stark apparatus

Figure 25: Dean-Stark apparatus

1.6   Setup Wax Loop
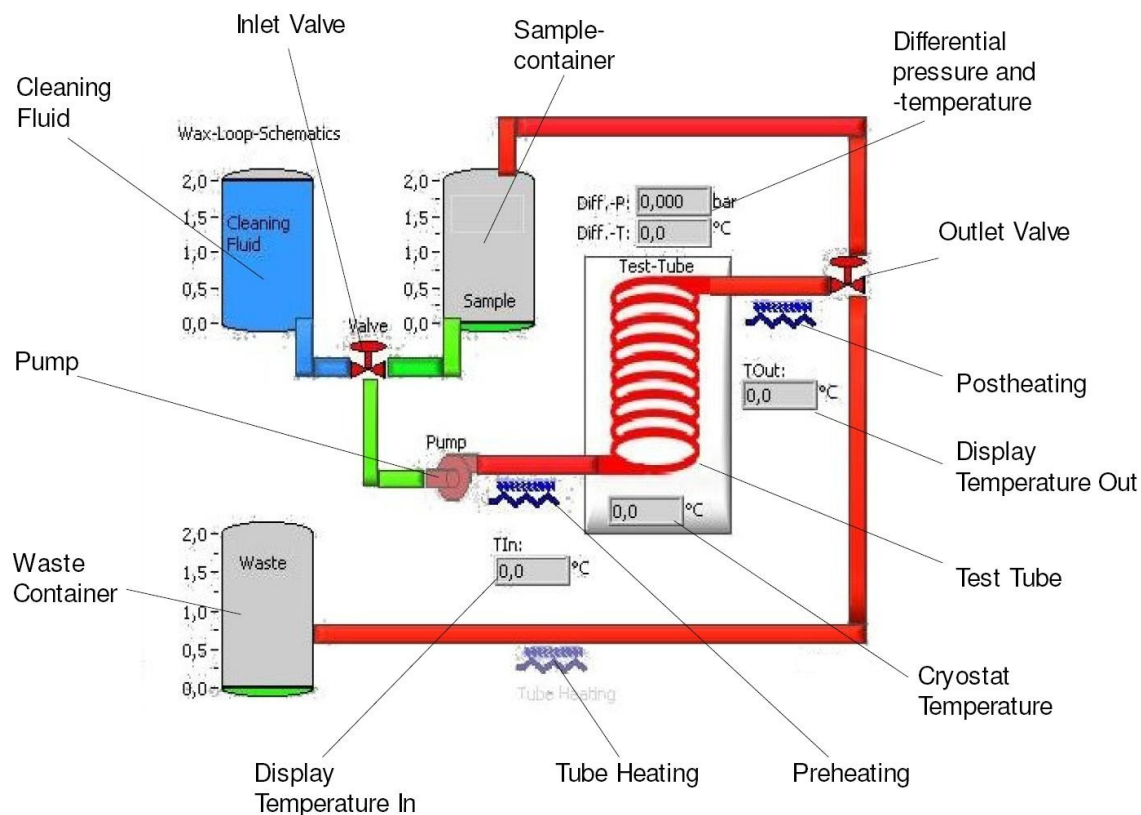
Figure 26: Schema Wax Loop (20)

Flow loop experiments were carried out with the wax flow loop by PSL Systemtechnik. The apparatus is build up of the following elements:

- Magnetic stirrer with heating device

  Rotating speed: 0 – 1400 RPM

  Temperature range: 20 – 300 °C

- Proportioning pump

  Flow rate: 0.3 – 30 ml/min

- Preheating Device

  Temperature range: 0 – 80 °C

- Cooled/Heated Test tube

  Temperature range: -25 – 150 °C

- Post heating Device

  Temperature range: 0 – 80 °C

The oil sample is put in the sample container, which stands on the heated magnetic stirrer. From there it is pumped through the test tube that can be tempered to start a precipitation and deposition there. Before and after the test tube heaters are installed, to keep the sample at a temperature above the WAT. That is necessary to avoid depositions outside the test tube in the supply and discharge pipes. It is possible to run experiments in an open-loop mode or in a closed-loop mode. In the later, the fluid is pumped back to the sample container and used again. This has the advantage that only small sample volumes are needed, starting from 50 ml.(20)

There are two valves installed, one to change from open loop to closed loop mode. The fluid flows into the waste container or back into the sample container. The second one is on the suction side of the pump, it`s used to switch to the cleaning fluid after testing.

As cleaning fluid solvent naphtha was used.

In a test run following parameters are measured by the apparatus and logged:

- Time

- Differential Pressure

  The differential pressure is measured across the test tube and is available as raw data, where the pressure peaks from the pump can be seen, and as already preprocessed value without the peaks.

- Differential Temperature

  The differential temperature is calculated from the preheating temperature, which is measured before the test tube and the outlet temperature from the test tube.

  Both raw data and preprocessed values are available.

- Cryostat temperature

  The cryostat temperature is the temperature to which the test tube is cooled down.

- Preheating temperature

- Flow rate

  The flow rate of the pump is measured and the values are given in ml/min.

- Outlet temperature

  The outlet temperature is measured just at the end of the test tube. Due to the better insulation of the flowing fluid in the case a wax deposit is built, this parameter can be used to draw conclusions of the deposit thickness in the tube.

Figure 27: Wax Flow Loop by PSL Systemtechnik

1.7   Wax Loop Measurements

1.7.1   Preparations

Before starting a test run, some preparations have to be done.

First of all the tubes and hoses have to be cleaned from wax deposits from former runs.

This is done by pumping solvent naphtha, until the differential pressure has been stabilized and no more wax particles can be seen in the waste line.

The next step is to preheat the oil sample above the WAT with the heating device. The WAT of the sample was determined with the rheometer before.

1.7.2   Matzen12 Oil Sample Date 08/2008

The first oil, which was tested, was a sample from the Matzen 12 well and it was taken in August 2008. In Table 3 the measured properties of this oil can be seen. The dynamic, the kinematic viscosity and the density were measured with the SVM 3000 Stabinger viscosimeter from Anton Paar. This device can be seen in Figure 28.

Figure 28: Stabinger Viscosimeter Anton Paar

The pour point was measured with the Pour Point Tester PPT 45150.

Table 3: Sample properties Matzen12 Sample Date 08/2008

Temperature T [°C]	20.00	29.99	40.00	50.01
Dynamic viscosity µ [mPa.s]	10.676	7.6631	5.7316	4.4360
Kinematic viscosity ν [mm².s⁻¹]	12.489	9.0320	6.8095	5.3113
Density ϱ [g.cm³]	0.8548	0.8484	0.8417	0.8352
Pour point [°C]	9.3			

After that, the WAT was determined with rotational viscosimeter. Both the blank sample and the samples with the added inhibitors were tested. As it can be seen in Figure 29 the deviation from the Newtonian behaviour below the WAT is larger for the blank sample than for the samples with the inhibitor. For this oil, the WAT is at about 22 °C.
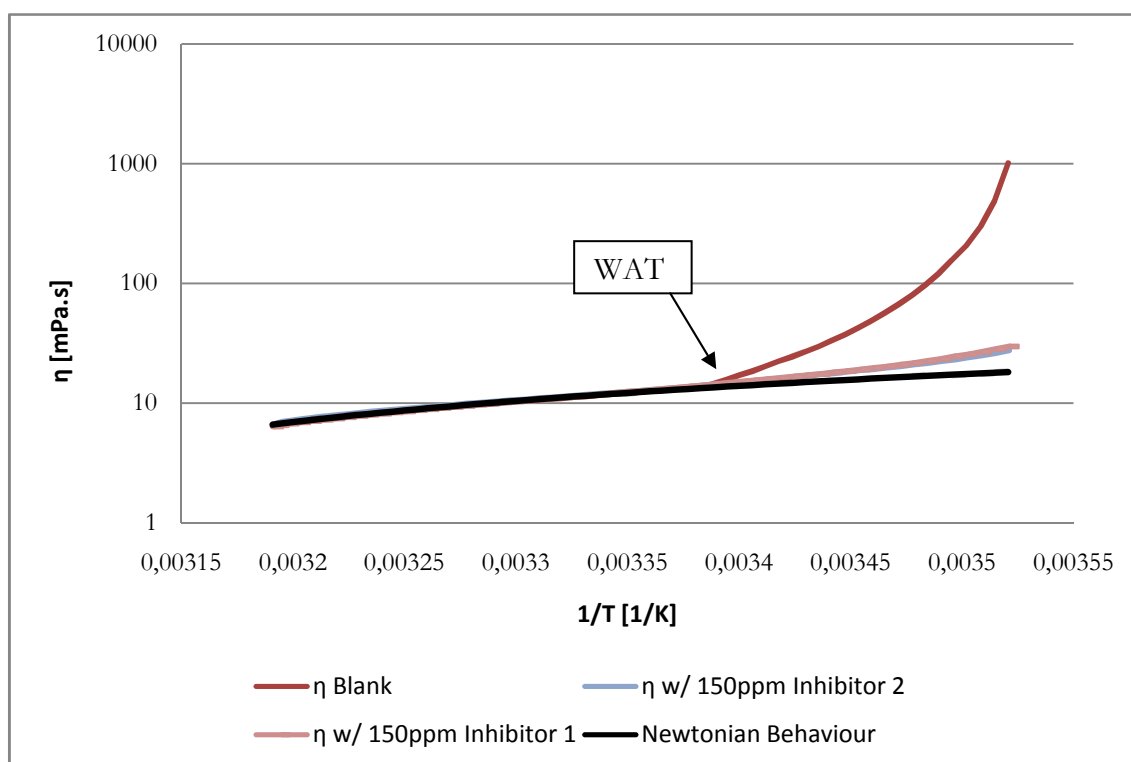
Figure 29: Viscosity, WAT Matzen12

Wax Loop Measurements

After knowing the WAT the first tests with the wax loop were started. For the first run the workflow was as followed:

- Preheat the Heating device, the Preheating device and the Post heating device to 45 °C

- Preheat 200 ml oil Matzen12 to 50 °C and stir it

- Clean the apparatus with 100 ml solvent naphtha

- Preflush the apparatus with 100 ml oil (50 °C)

- In case of inhibited oil: mix 100 ml oil (50 °C) with 15 µl (150 ppm) Inhibitor, stir for 10 min at 50 °C

- Flush the apparatus with 40 ml oil-inhibitor mixture

- Start the measurement with 60 ml oil-inhibitor mixture

To make sure all former deposits can be removed with the solvent naphtha the device is preheated. The preheating of the oil has to be done to solve all precipitations in it.

In case no inhibitor was used the device was flushed only with preheated oil after cleaning, in case inhibited oil was used for the test inhibited oil was also used for the flush, to achieve the desired concentration.

While the test following parameters were used:

- Preheating device   45 °C

- Postheating device   45 °C

- Oil Heater   50 °C

- Stirring device   250 rpm

- Flow rate   5 ml.min^{-1}

- Cryostat:

  Start   45 °C

  End   10 °C

  Step size   0,5 °C.min^{-1}

  Start of cooling after 5 min
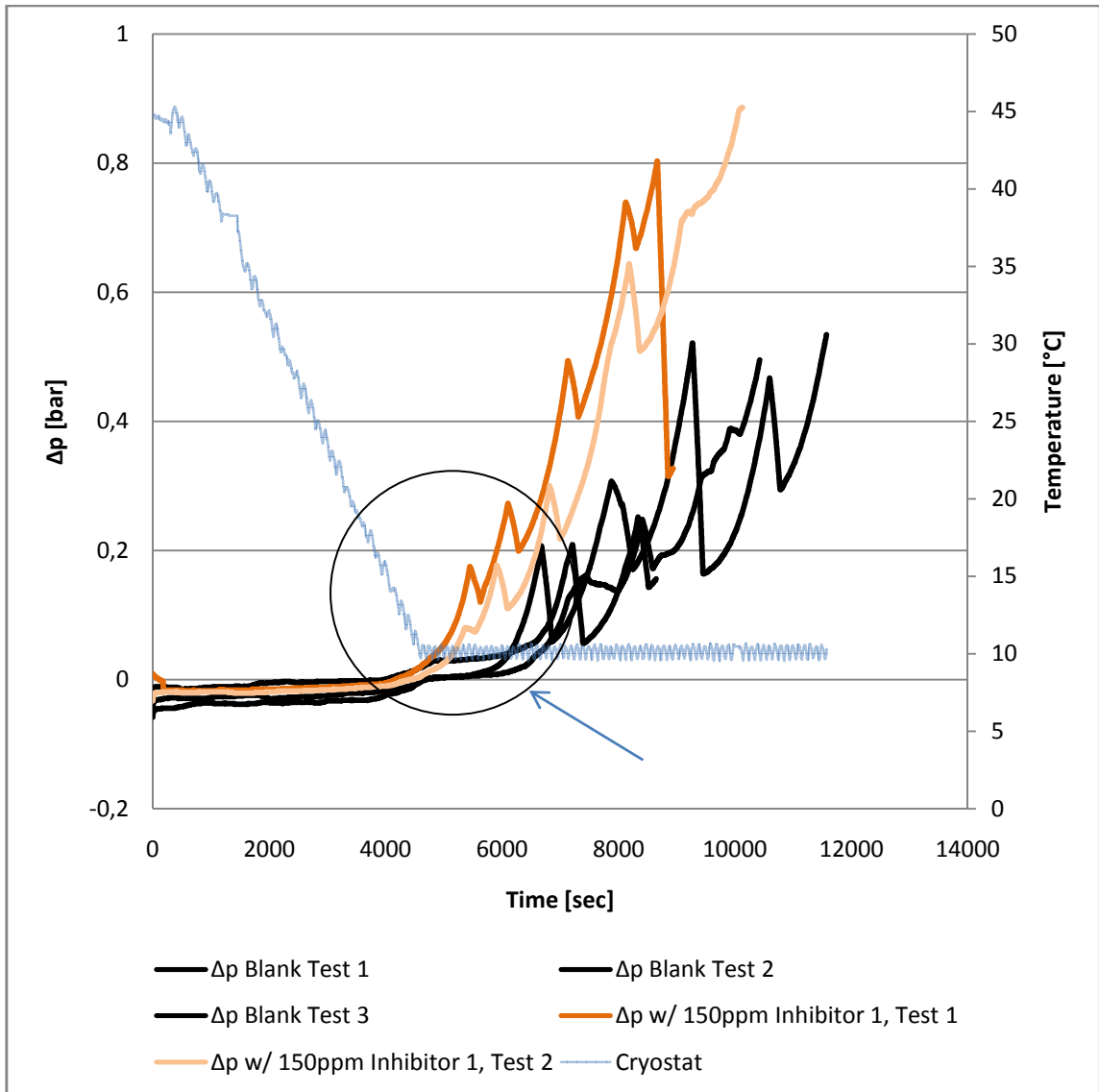
Figure 30: Wax Loop Measurements Matzen12 Workflow 1

As it can be seen in Figure 30, the differential pressure raised quicker in the case an inhibitor was used than in the case a blank sample was tested.

For the second test series the workflow was changed:

- The heating device, the Preheating device and the Post heating device were preheated to 50 °C instead of 45 °C

- In case of inhibited oil: mix 100 ml oil (50 °C) with 15 μl (150 ppm) Inhibitor, stir for 10 min at 50 °C

In addition, the parameters for the test differ:

- Preheating device   50 °C

- Postheating device   50 °C

- Oil Heater   50 °C

- Stirring device   250 rpm

- Flow rate   5 ml.min^{-1}

- Cryostat:

  Start   50 °C

  End   10 °C

  Step size   0,5 °C.min^{-1}

  Start of cooling after 5 min

The difference to the first test series is, that the device and the oil sample are preheated to the same temperature, to 50 °C.

The result can be seen in Figure 31.
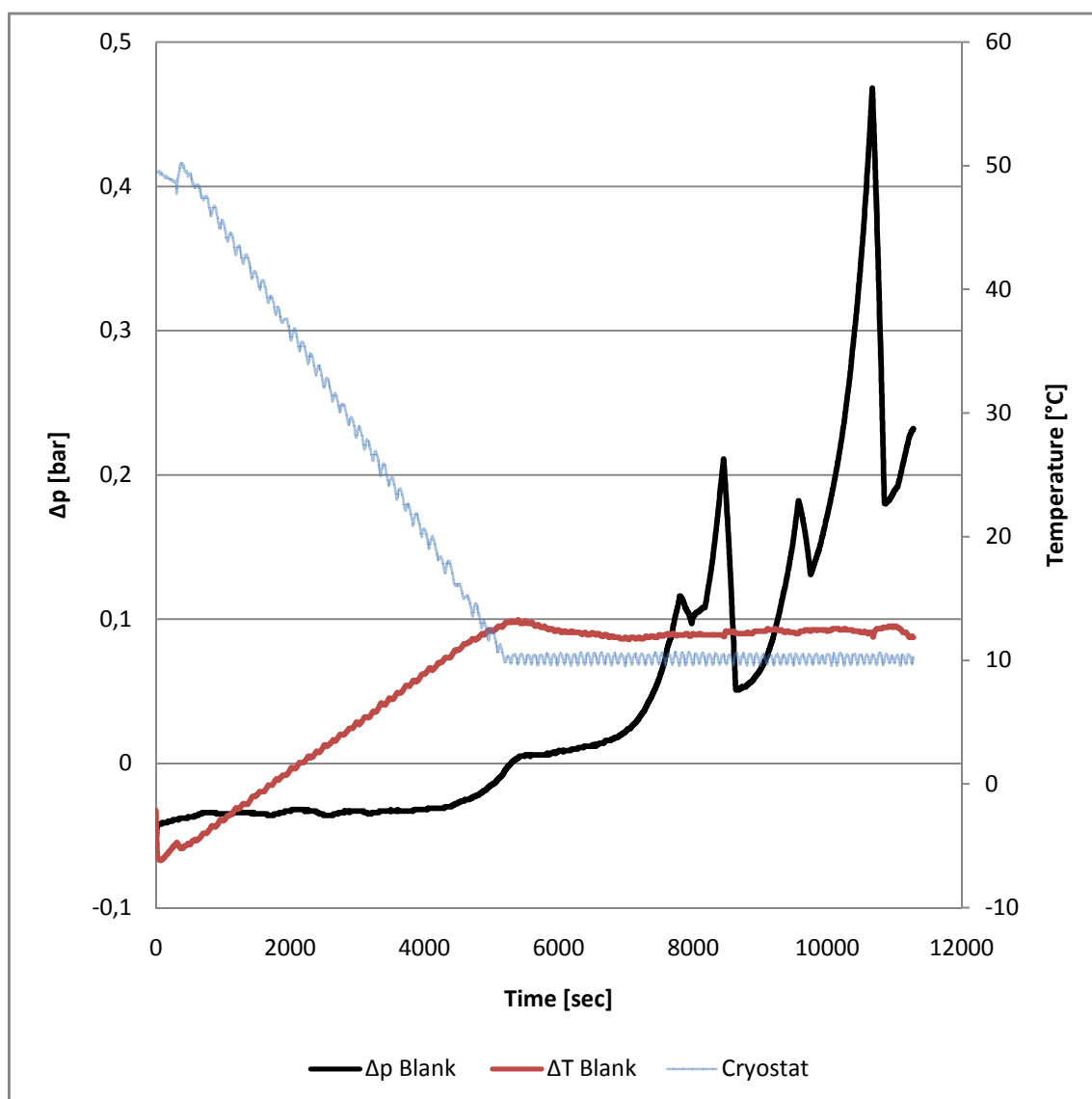
Figure 31: Wax Loop Measurement Matzen12 Workflow 2

For the third test series only the end temperature of the cryostat and therefore the outer temperature of the test tube were changed to 0 °C instead of 10 °C.
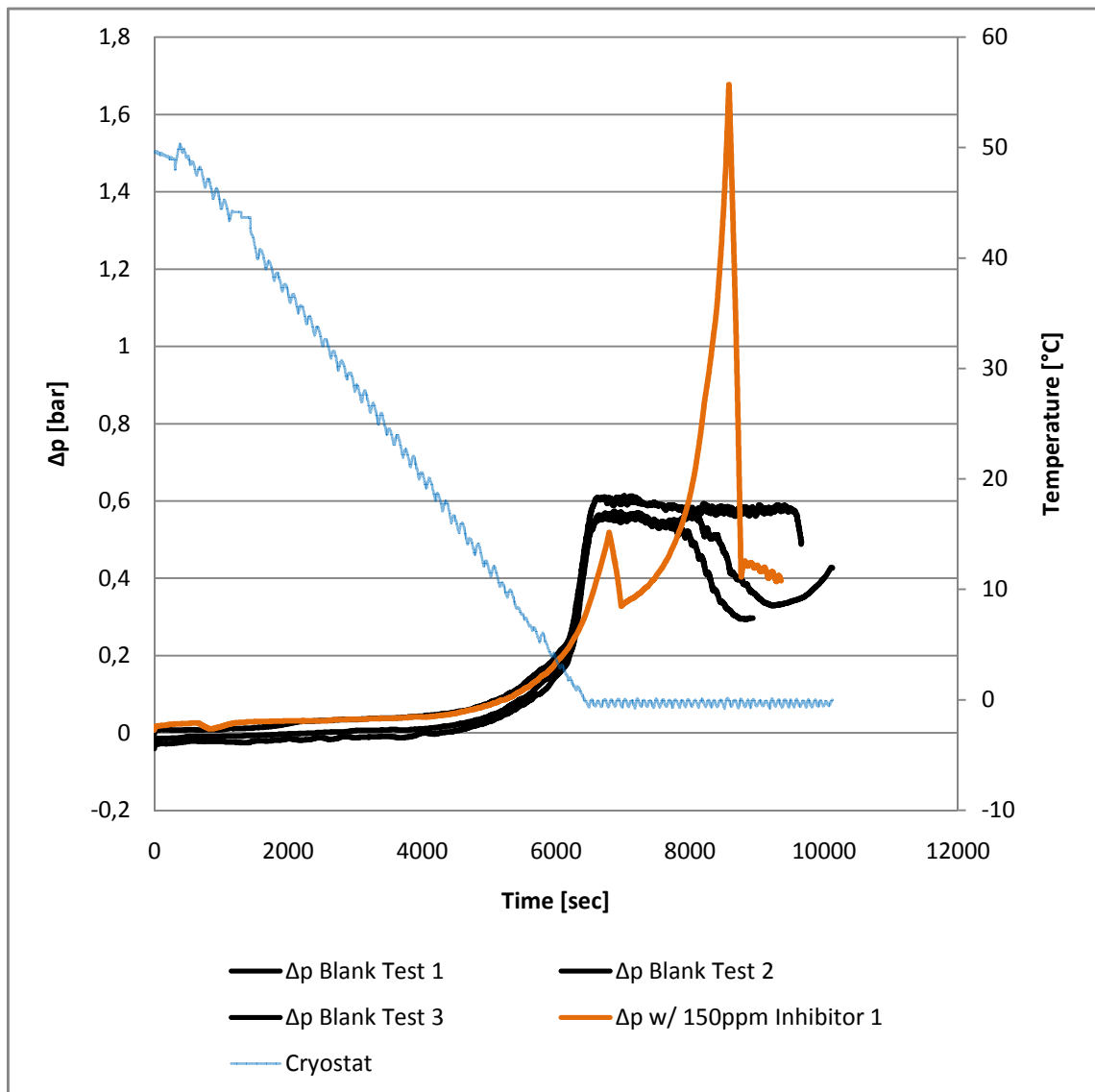
Figure 32: Wax Loop Measurement Matzen12 Workflow 3

1.7.3 Matzen12 Oil Sample Date 10/2008

Thermal History

Because it is known that the thermal history has an influence on the viscosity and the pour point of oil, this sample was treated in three different ways before making viscosity measurements.

It is believed that the dependence on the thermal history is associated with the content of indigenous pour point depressants, which are found in the asphaltenes and resins of the oil. If the oil sample is preheated to a high enough temperature that all wax content is dissolved completely (80 °C or higher), the indigenous pour point depressants can interact

with the wax crystals while they are built and change their surface characteristics and morphology. This changes the tendency of the wax crystals to build networks.

To observe this it is important that the preheating temperature is high enough, otherwise the indigenous pour point depressants are still immobilized, while the low molecular weight wax is already dissolved. (17)

For the first test in this series the oil sample was not preheated, it was used as it was received. The Pour Point was 1,1 °C.

As shown in Figure 33 the viscosity measurement showed a WAT of about 23,5 °C.
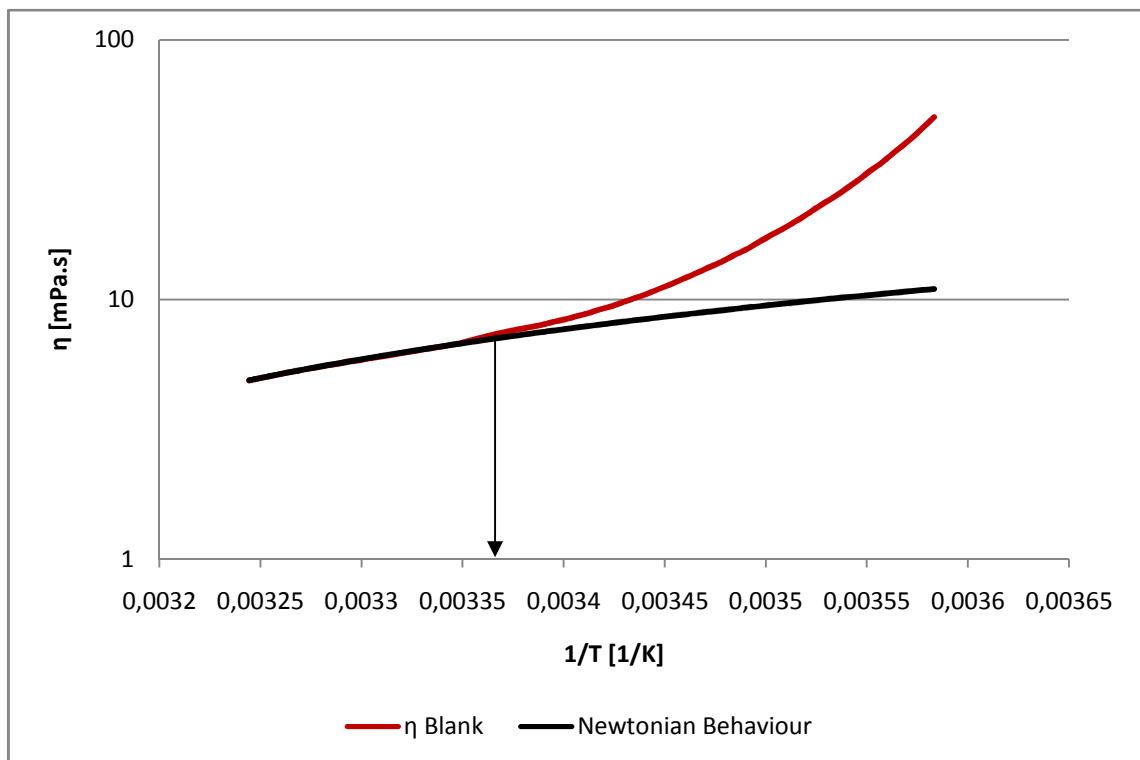
Figure 33: Viscosity Matzen12 Oil Sample Date 10/2008, Workflow 1

For the second test the oil sample was preheated in a gas tight autoclave to 80 °C for one hour and cooled down to about 40 °C before starting the measurement.

In this case the pour point was -9 °C.

The viscosimeter showed a WAT of 19 °C.
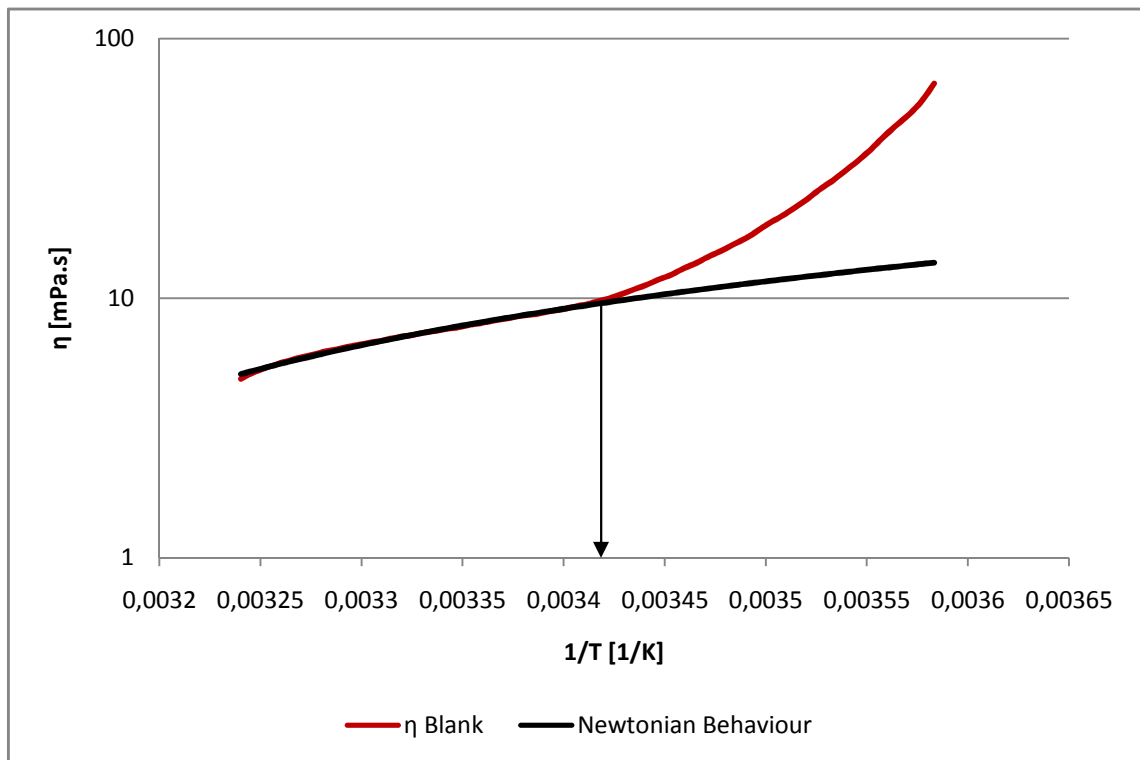
Figure 34: Viscosity Matzen12 Oil Sample Date 10/2008, Workflow 2

For the third test the sample was heated to 80 °C on a hot plate for one hour and stirred.
The container was not gas tight.

The pour point using this procedure was at 2,2 °C.

The viscosimeter showed a WAT of 19 °C.
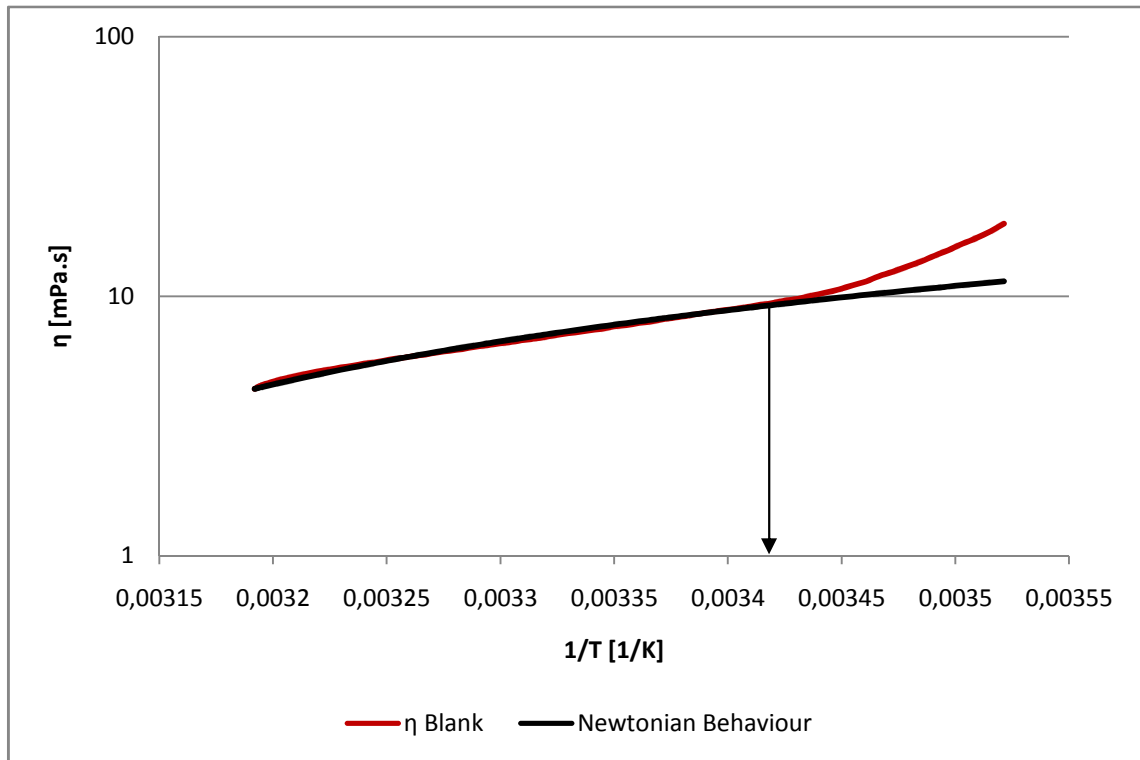
Figure 35: Viscosity Matzen 12 Oil Sample Date 10/2008, Workflow 3

Table 4 shows a comparison of the different heating methods and the observed pour points and wax appearance temperatures.

Table 4: Sample properties Matzen12 Sample Date 10/2008

Heating method	Temperature [°C]	Pour Point [°C]	WAT [°C]
None (as received)	-	1,1	23,5
Autoclave (gastight)	80 °C	-9	19
Hotplate	80 °C	2,2	19

For the following wax loop measurements the samples were not preheated, because there was no explosion protected oven available, which is needed to heat the sample in an autoclave and cool it down slowly.

1.7.4 Pirawarth1 Oil Sample

The next available oil sample was from the Pirawarth1 well. The starting point was like in the tests before to determine the WAT, to see if the sample is useable.

The first viscosity measurement was made from 40 to 7 °C. As seen in Figure 36 no wax precipitation was observed above 7 °C. For that reason another test was done.
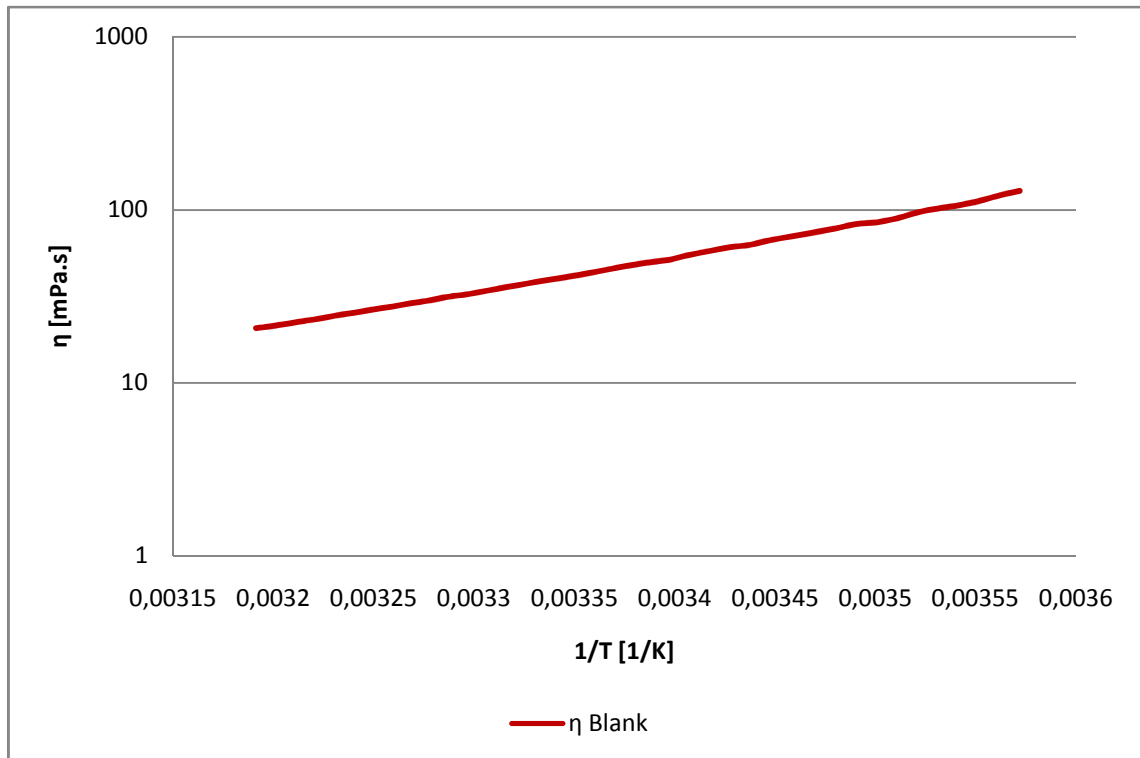
Figure 36: Viscosity Pirawarth 1 Oil Sample, Test 1

The second viscosity measurement was made from 10 to -6 °C.
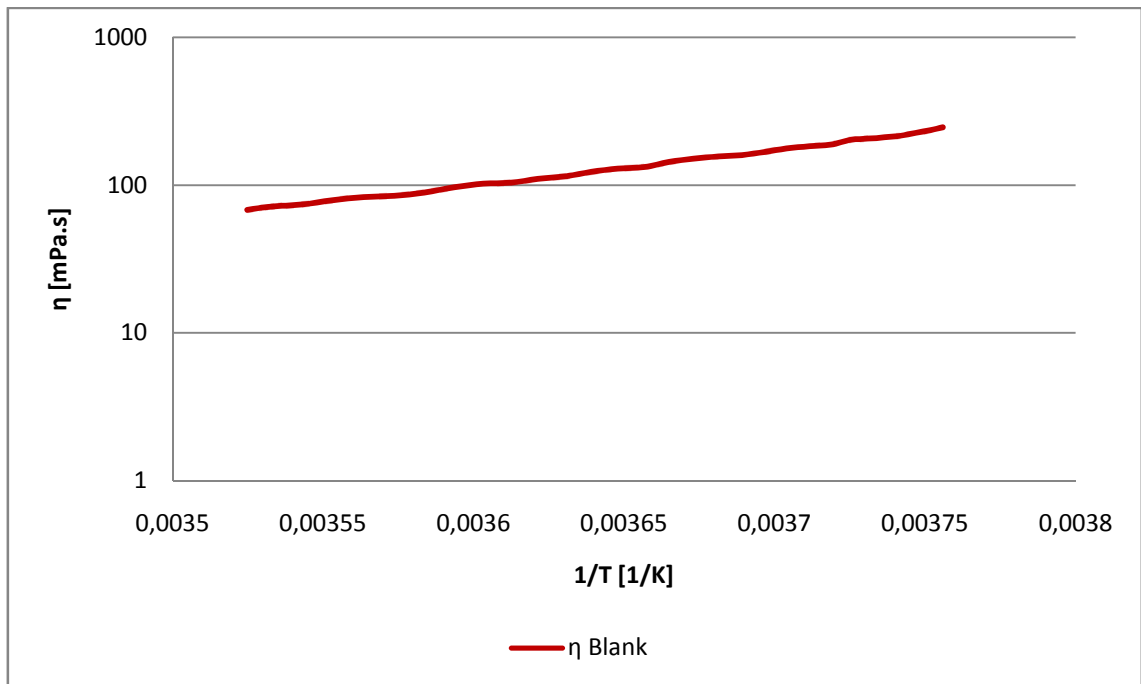
Figure 37: Viscosity Pirawarth 1 Oil Sample, Test 2

Because no deviation was recognisable, another test was made to verivy the results.

This test was in the temperature range from +10 to -10 °C.
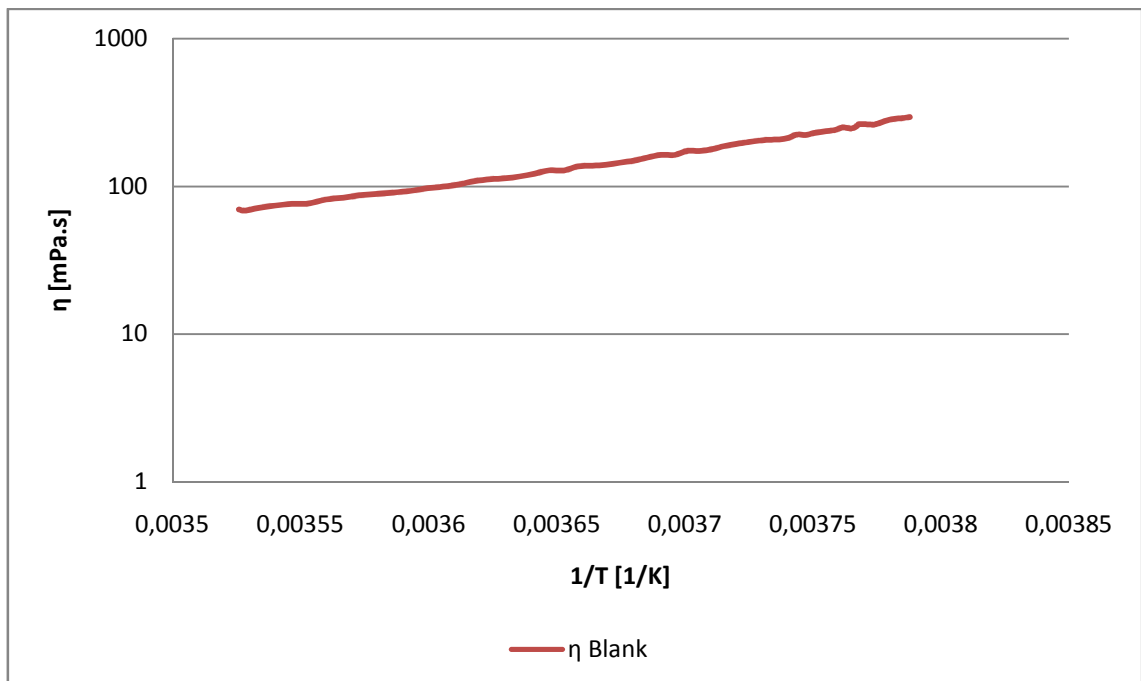
Figure 38: Viscosity Pirawarth 1 Oil Sample, Test 3

Because of the low WAT (below -10 °C), the oil was not used for further tests.

1.7.5   Ebenthal17a Oil Sample

The next and last tested oil sample was from the Ebenthal17a well. This oil was chosen because paraffin deposit problems occurred here.

Foremost a chromatography was made to identify the carbon number distribution.

The result can be seen in Figure 39 and Table 5.

Studies showed that deposited wax from heavy fuel oils, if it was cooled had a composition, which was centred on C20. The spread was from about C16 to C27. The lower the cooling temperature the lower are the average carbon numbers found in the wax deposit.(21)
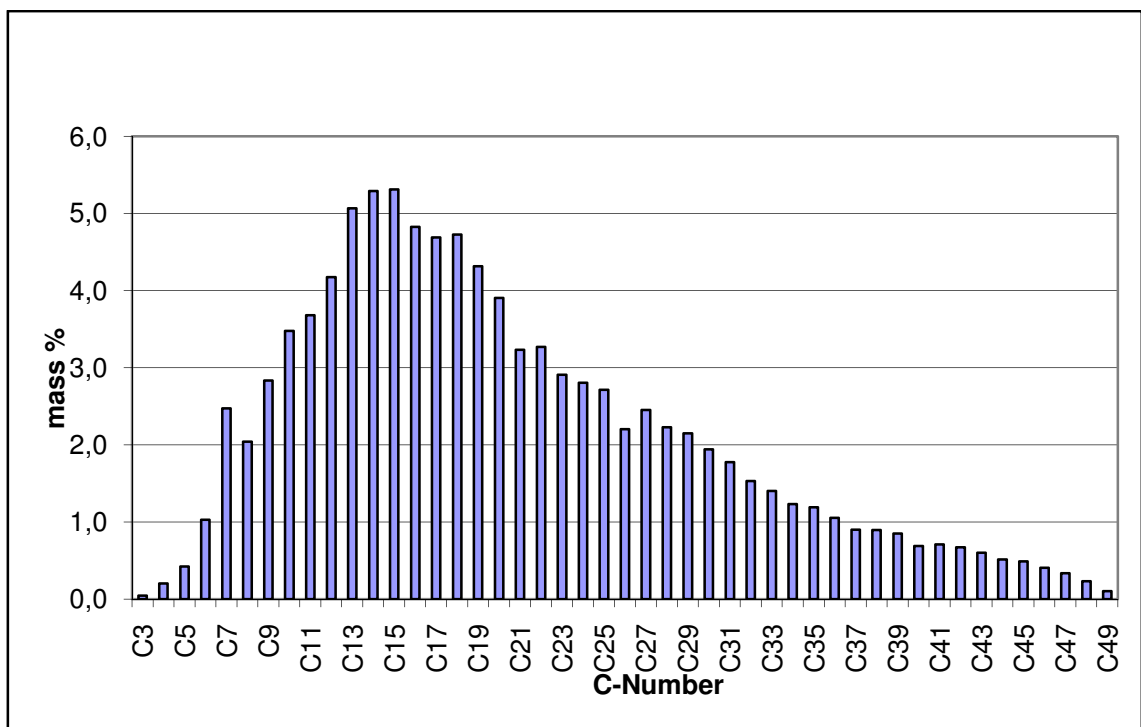
Figure 39: Carbon - Number distribution Ebenthal17a Sample

Table 5: Carbon - Number distribution Ebenthal17a Sample

Carbon - Number	mass %	Carbon - Number	mass %
C3	0,044	C27	2,453
C4	0,202	C28	2,228
C5	0,421	C29	2,149
C6	1,031	C30	1,942
C7	2,471	C31	1,777
C8	2,042	C32	1,53
C9	2,832	C33	1,401
C10	3,479	C34	1,234
C11	3,683	C35	1,191
C12	4,174	C36	1,054
C13	5,066	C37	0,899
C14	5,291	C38	0,897
C15	5,309	C39	0,849
C16	4,827	C40	0,687
C17	4,691	C41	0,711
C18	4,727	C42	0,673
C19	4,315	C43	0,601
C20	3,903	C44	0,515
C21	3,233	C45	0,492
C22	3,269	C46	0,406
C23	2,907	C47	0,335
C24	2,805	C48	0,232
C25	2,713	C49	0,105
C26	2,203		

After that two viscosity measurements were made.
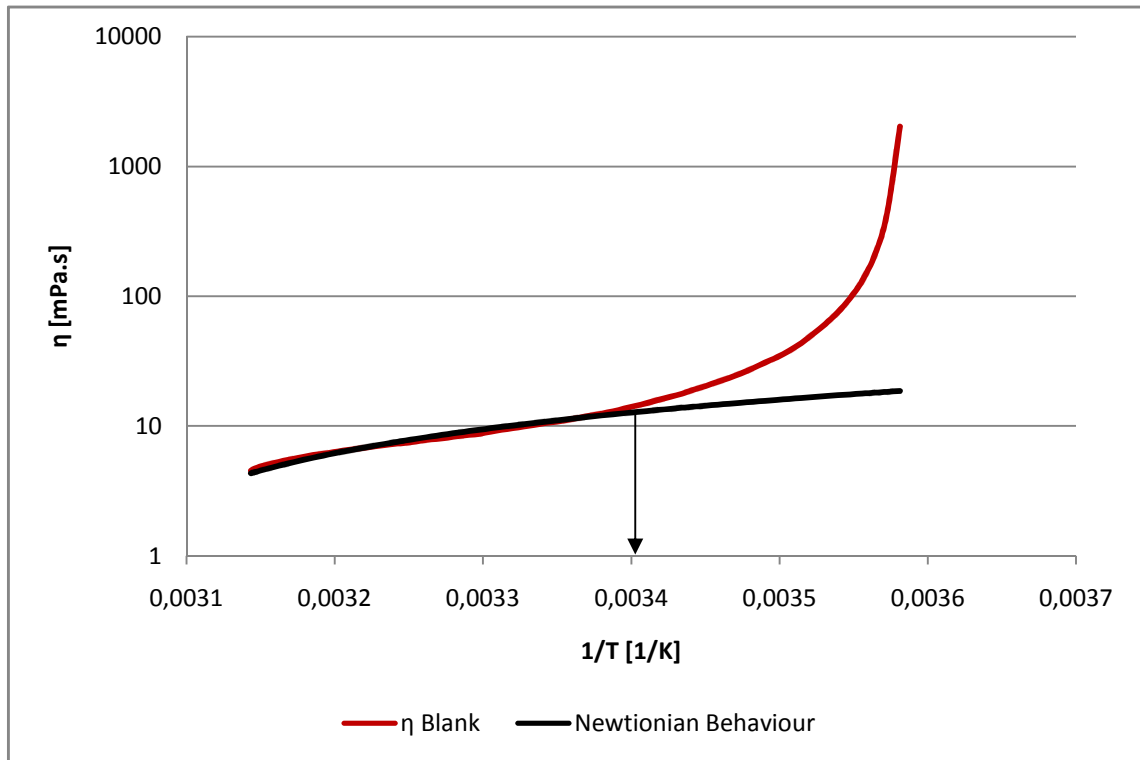
Figure 40: Viscosity Ebenthal17a Oil Sample, Test 1

The first measurement showed a WAT of about 20 °C.
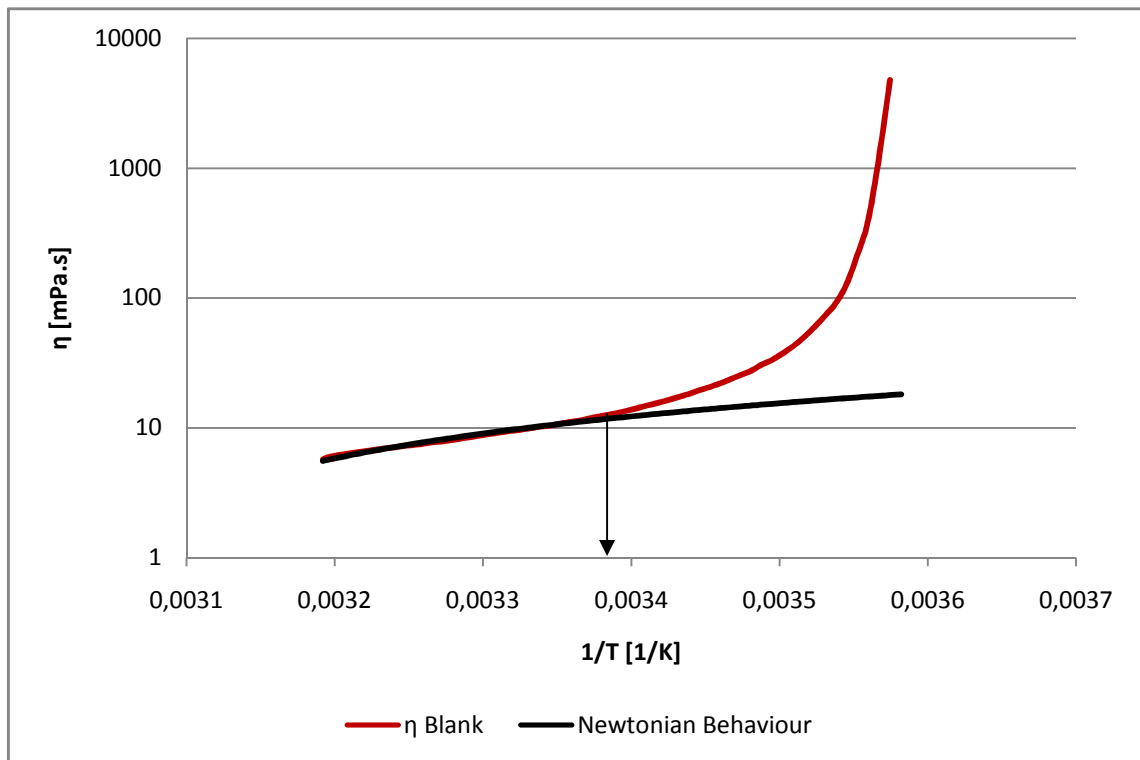
Figure 41: Viscosity Ebenthal17a Oil Sample, Test 2

The second measurement showed a WAT of about 22 °C. Therefore the end temperature of the cryostat was set to 10 °C, which is significant below the WAT.

All of the following test of the Ebenthal17a oil samples were done out with the following parameters:

- Preheating the hot plate to 35 °C

- Preheating device 35 °C

- Postheating device 35 °C

- Flow rate 5 ml/min

The first test had the purpose to determine the pressure difference without any wax deposition. To achieve this, the cryostat temperature was set to 35 °C as the pre- and post heating device.

As seen in Figure 42 the differential pressure climbed to nearly 0,01 bar after about 6000 seconds and stabilized there.
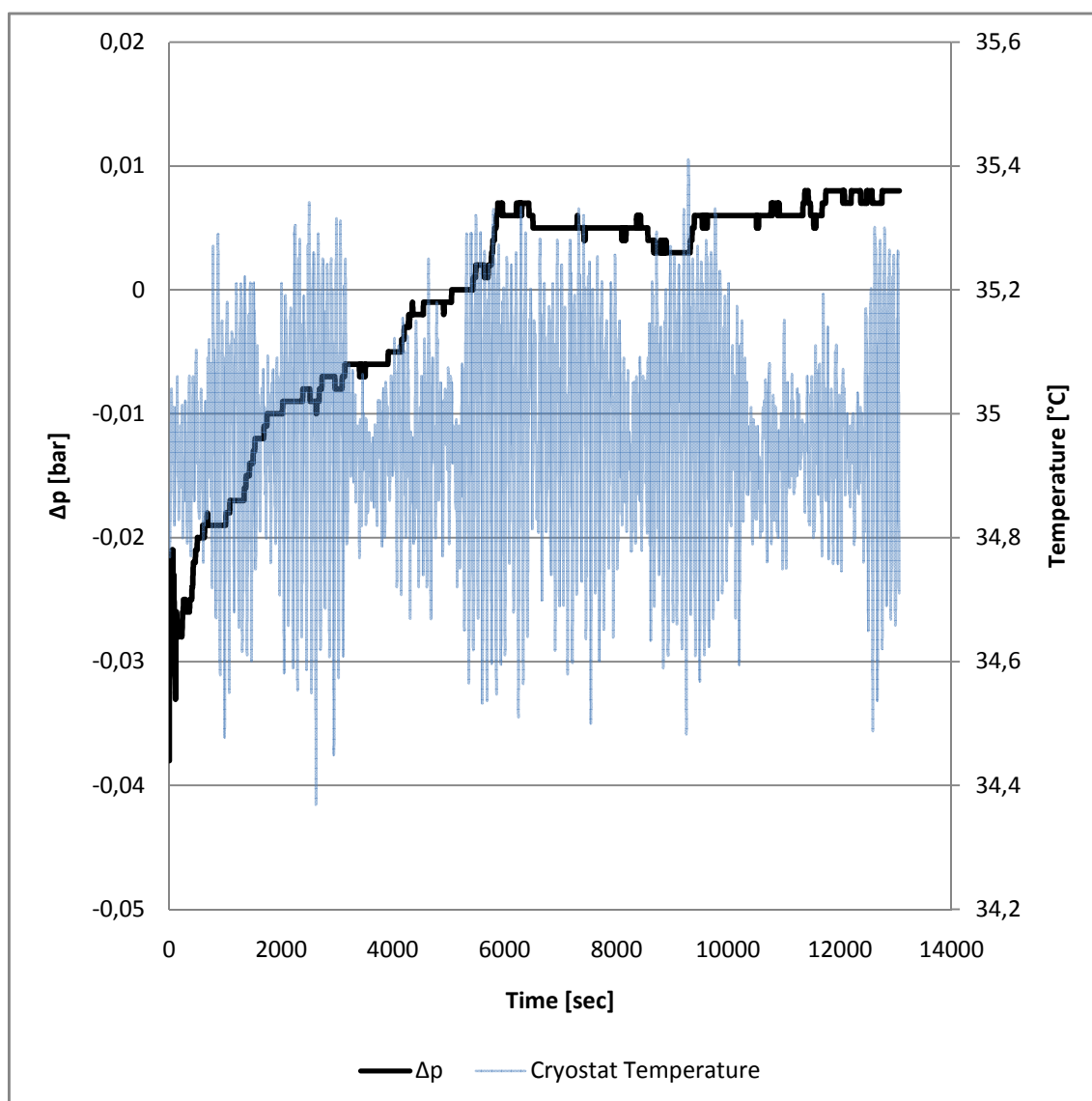
Figure 42: Wax Loop Measurement Ebenthal17a, w/o Cooling

The next step was to make three runs with a cryostat temperature of 10 °C. For this test no inhibitor was used.

Figure 43 shows the results. The result is slightly different, but in the same range of differential pressure. In addition, the time when the highest differential pressure is reached is almost at the same time.
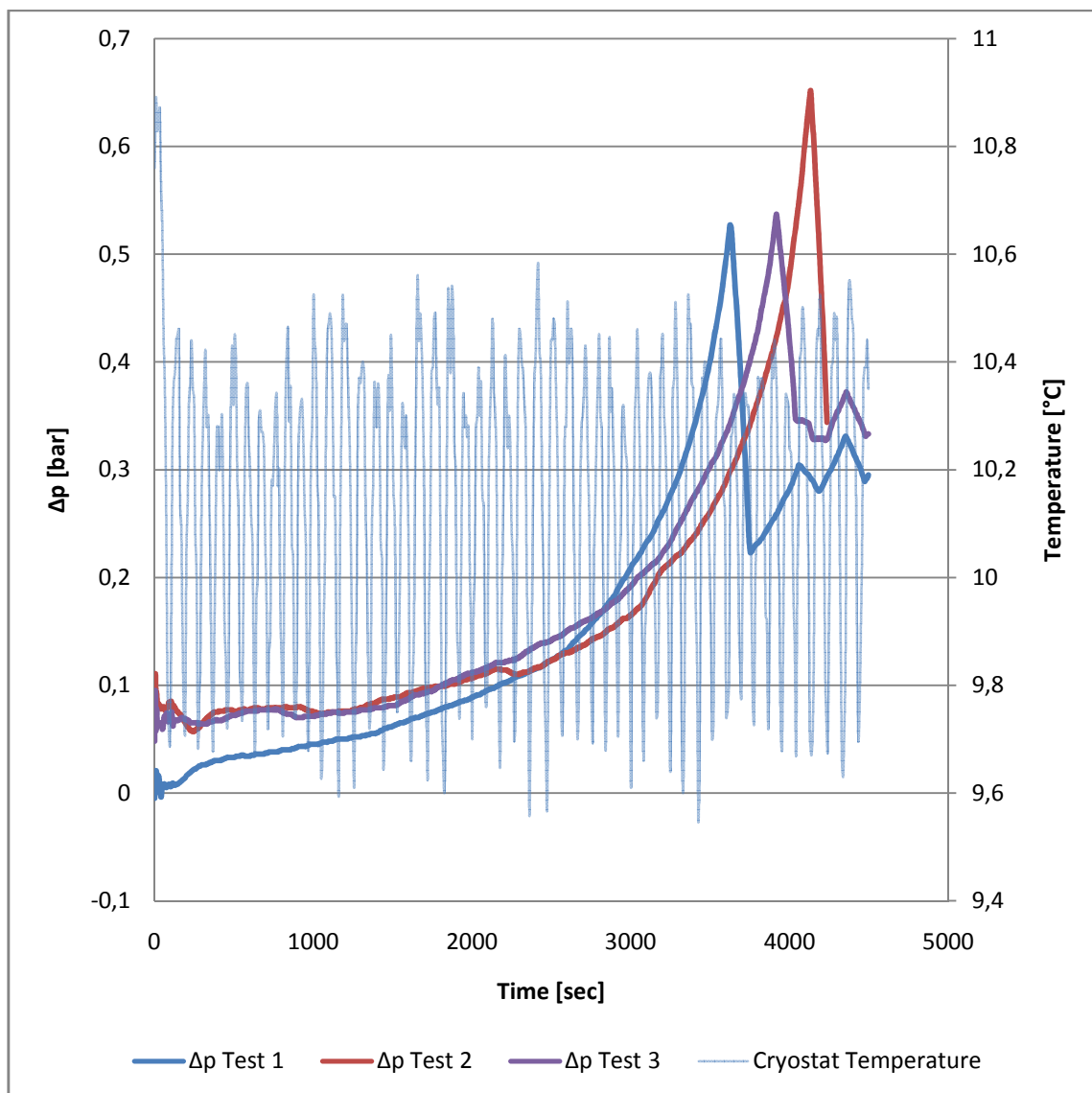
Figure 43: Wax Loop Measurement Ebenthal17a, Blank

Now five inhibitors were tested, all with the same parameters, and compared to the runs without inhibitor.

Inhibitor 1
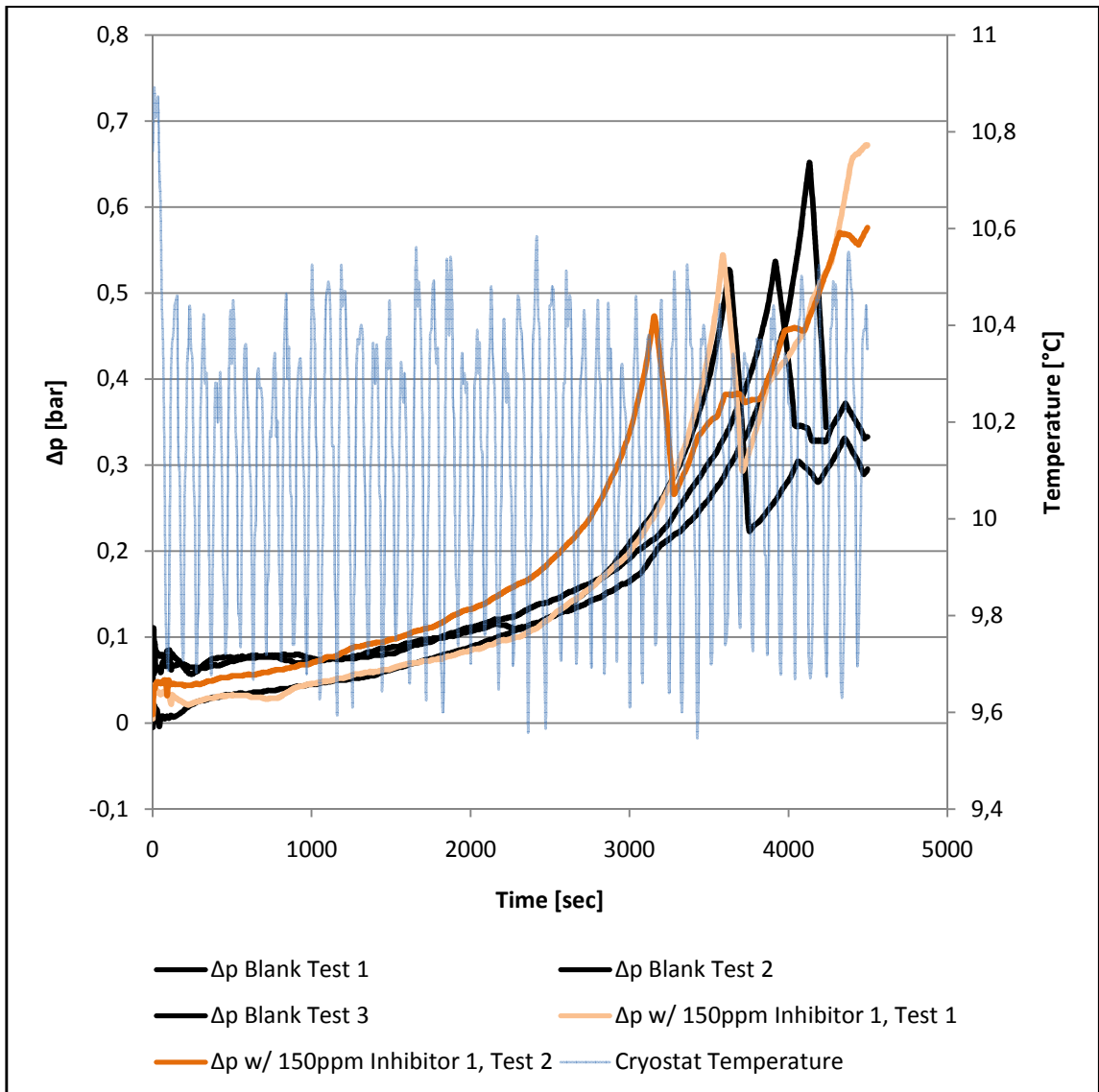
Figure 44: Wax Loop Measurement Ebenthal17a w/ 150ppm Inhibitor 1

Inhibitor 2
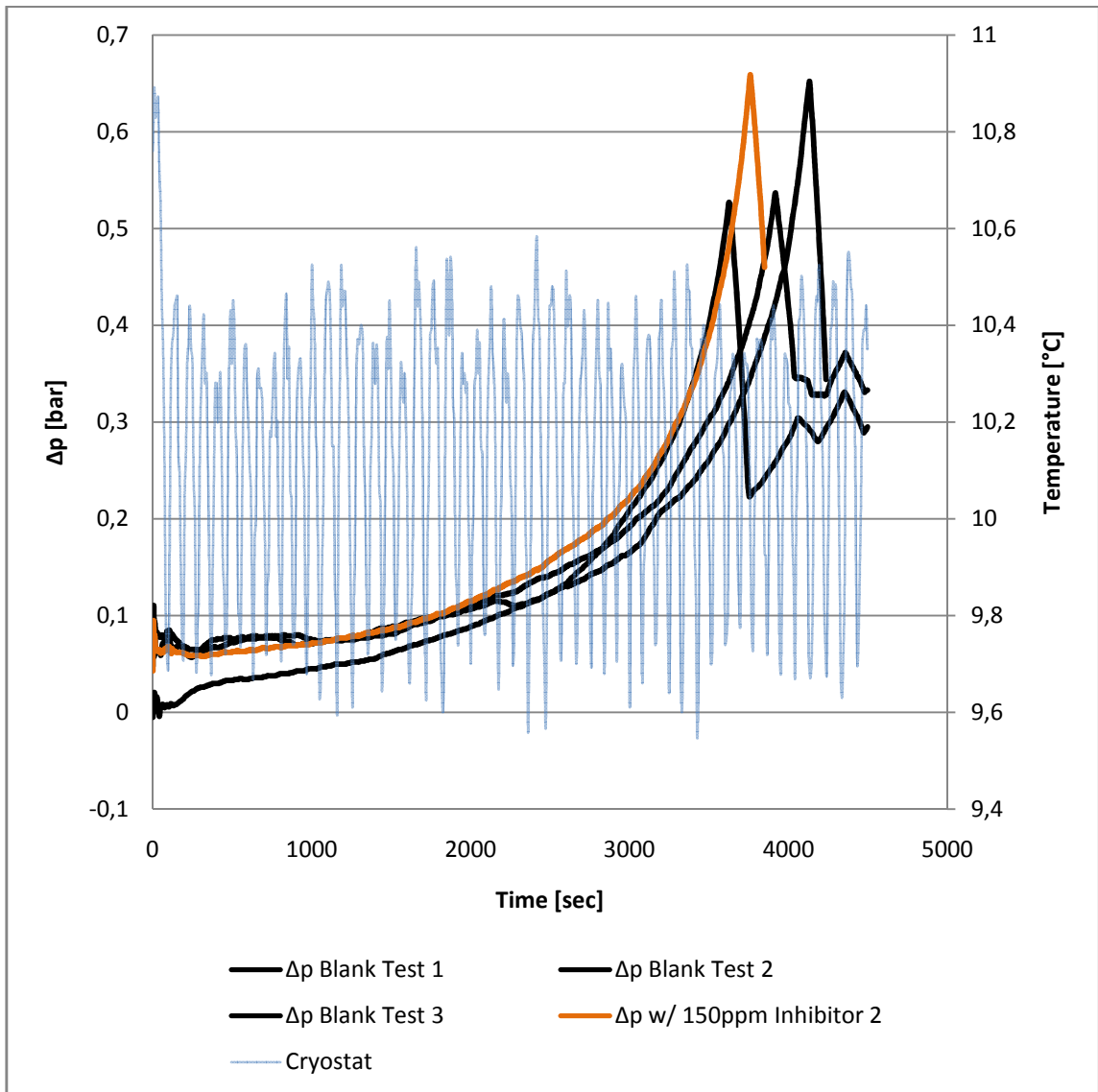
Figure 45: Wax Loop Measurement Ebenthal 7a w/ 150ppm Inhibitor 2

Inhibitor 3
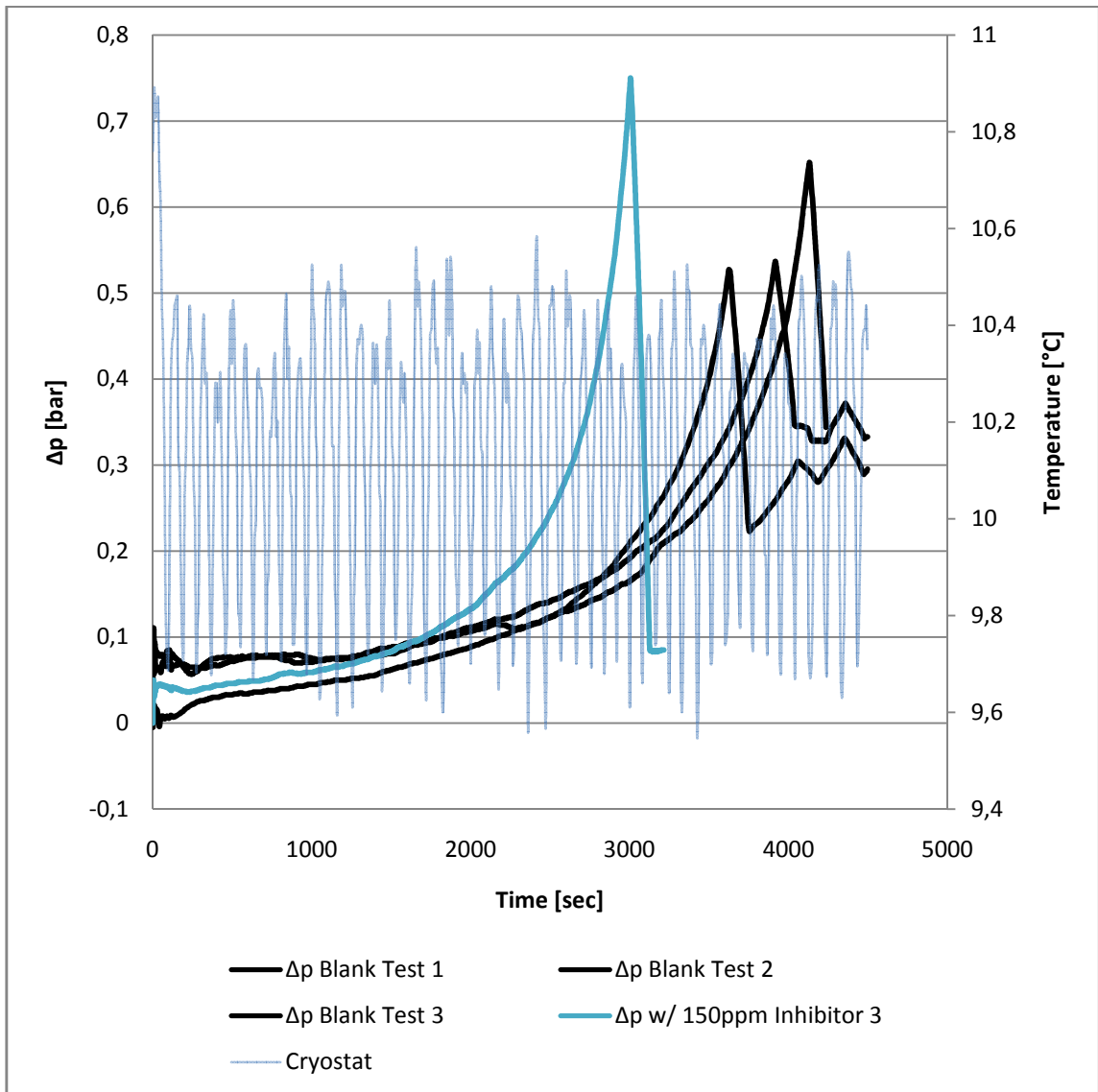
Figure 46: Wax Loop Measurement Ebenthal17a w/ 150ppm Inhibitor 3

Inhibitor 4
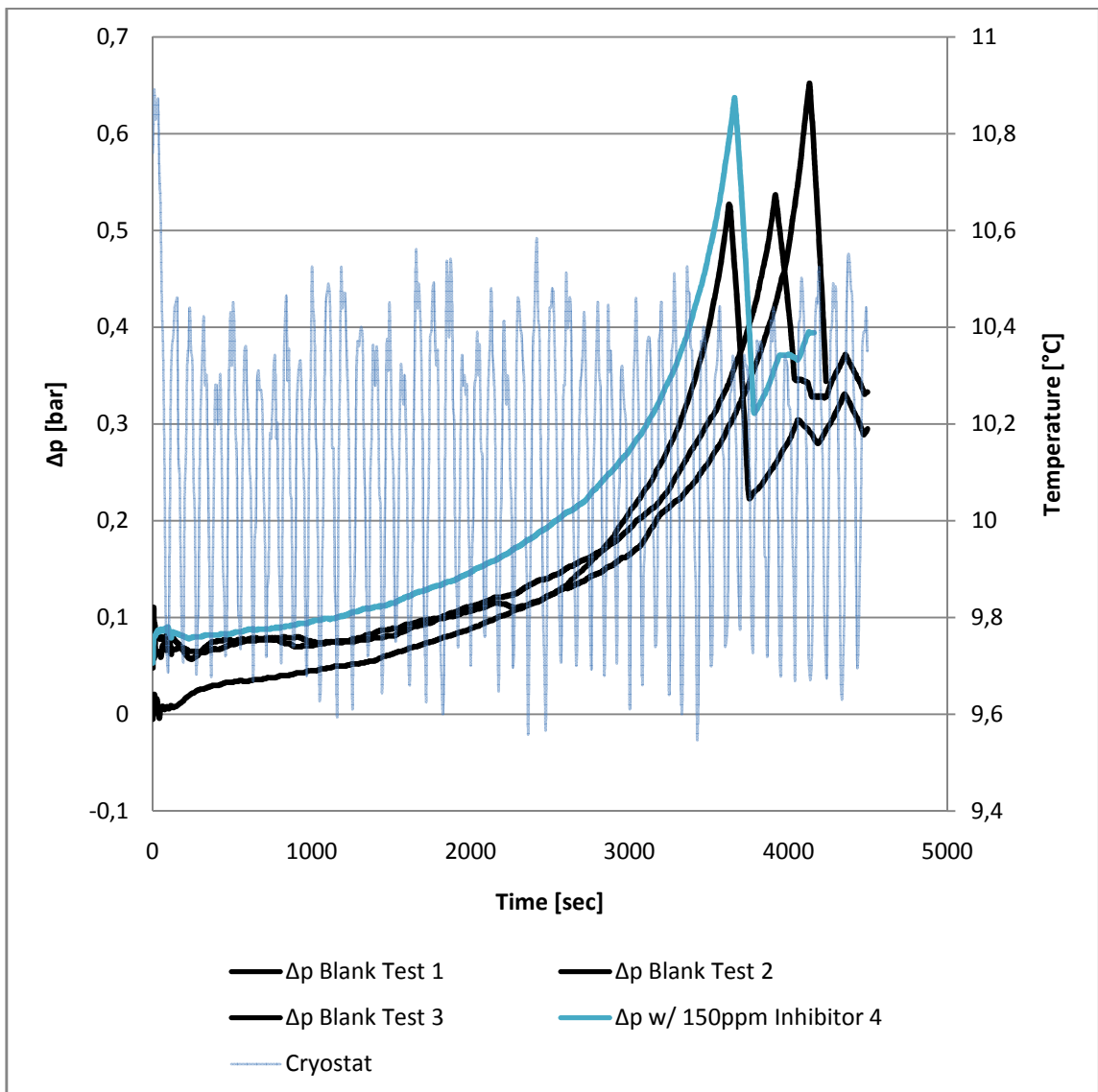
Figure 47: Wax Loop Measurement Ebenthal17a w/ 150ppm Inhibitor 4

Inhibitor 5
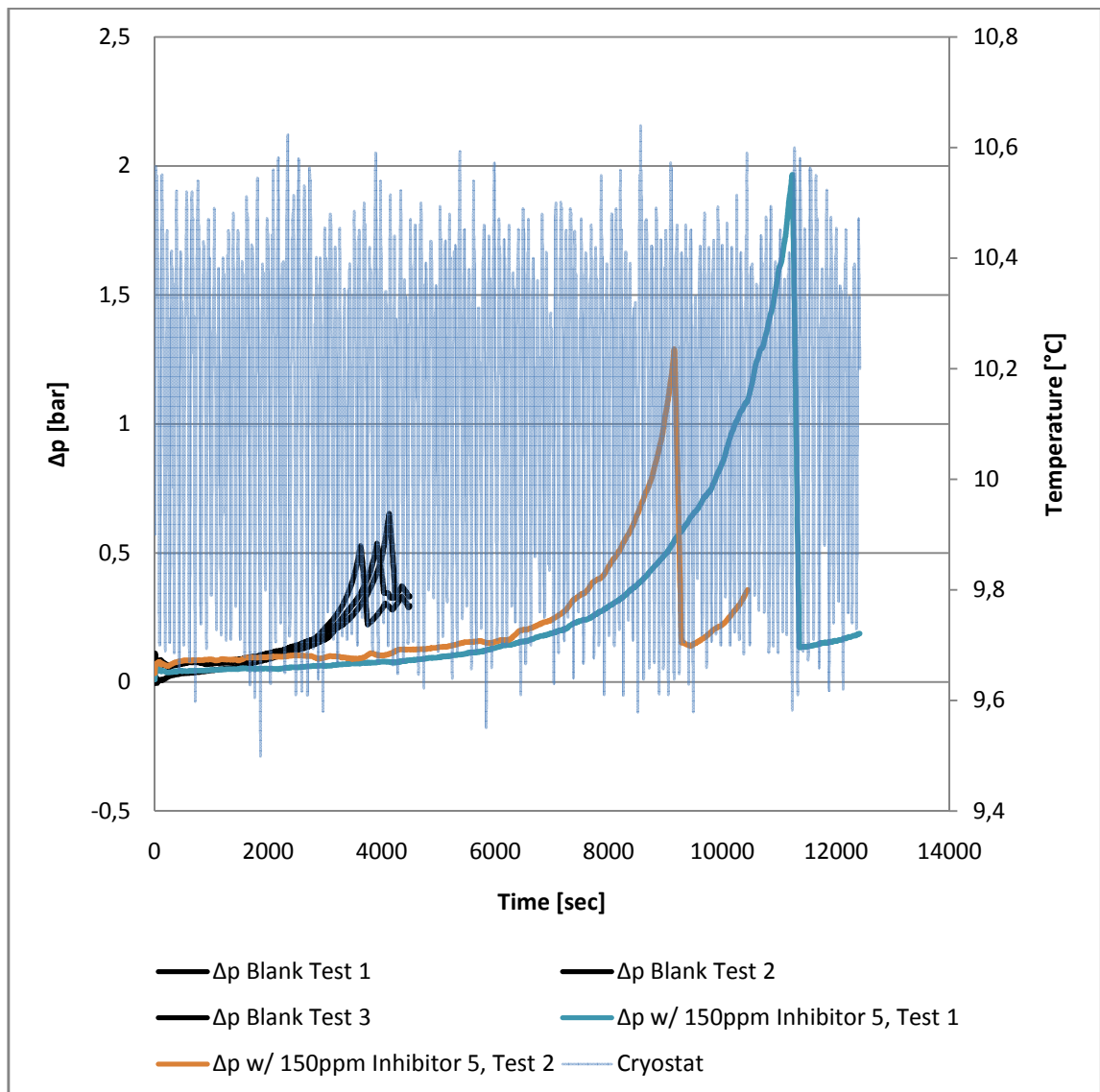
Figure 48: Wax Loop Measurement Ebenthal17a w/ 150ppm Inhibitor 5

As it can be seen in Figure 44 to Figure 48 only inhibitor 5 showed a great impact on the differential pressure and on the time when the maximum of the differential pressure is reached.

1.8   Comparison Viscosimeter – Wax Loop

Because only one from the tested inhibitors delayed the wax deposition in the wax loop, further tests were made. The best and the worst working inhibitor were measured on the rheometer again and the results compared. The tests were made with the Ebenthal17a oil sample and with the inhibitors number one and five. The samples for the rheometer test were prepared the same way as for the wax loop measurement.
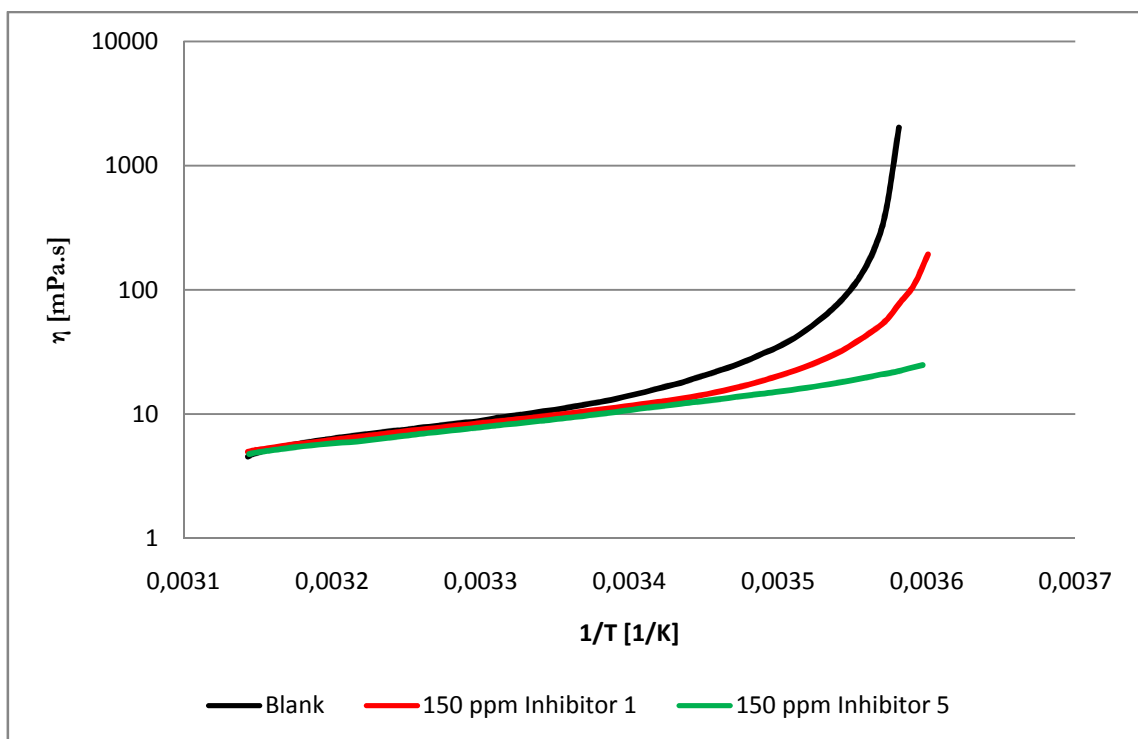
Figure 49: Inhibitor Screening on the Rheometer

Figure 49 show that both inhibitors reduce the viscosity when the sample is cooled down. Contrary to the expectations, the test on the wax loop did not produce the same results. While both inhibitors reduced the viscosity on the rheometer below the WAT, only one, namely inhibitor 5, delayed the augmentation of the differential pressure (Figure 50). The same is valid for the other tested inhibitors, as a former diploma thesis shows, where the inhibitors were only screened with the rheometer. Every product lowered the viscosity at temperatures under the WAT.
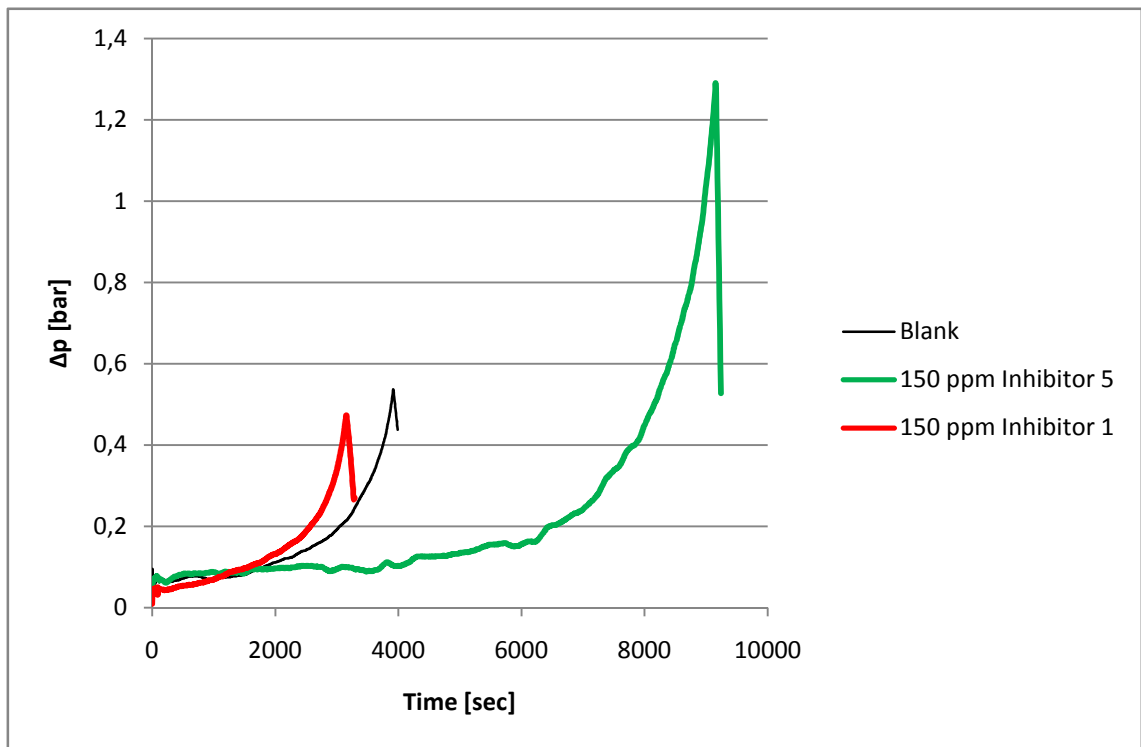
Figure 50: Inhibitor Screening Wax Loop

1.9   Limits of use and Suggestions

One weak point in the build up of the wax loop is the used pump. Tests with high viscosity oils showed, that it was not possible to maintain a certain rate. Although the oil sample was heated to 50° C and stirred, the rate decreased to about 1/3 of the setting under this conditions.

Also the differential pressure measuring device did not work proper all the time, especially at the beginnings of the tests. A magnification of a wax loop test is shown in Figure 51. Up to a time of 4000 seconds the differential pressure had a negative value.
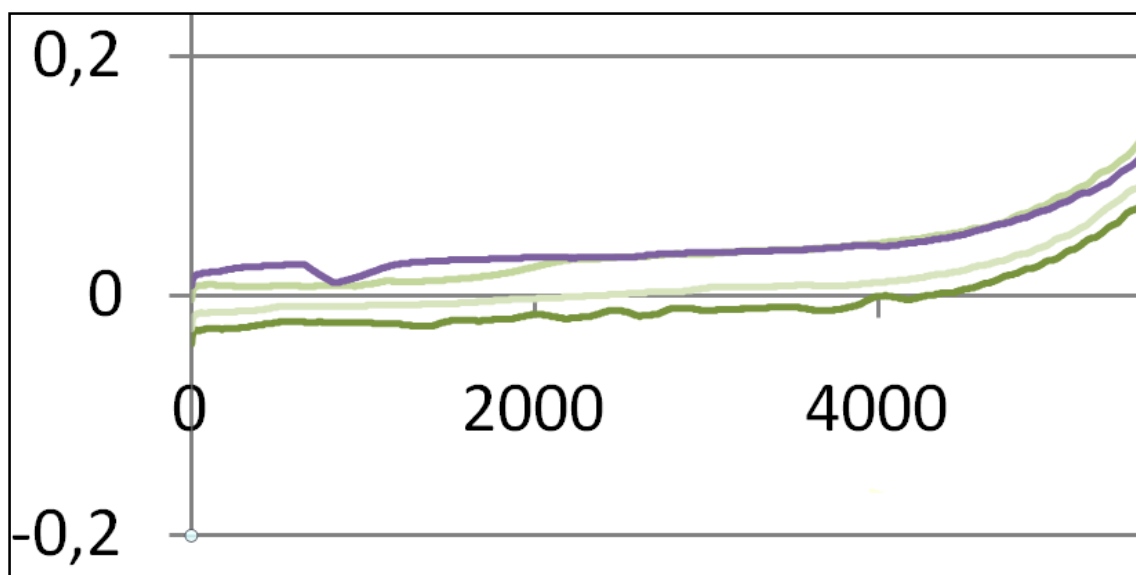
Figure 51: Differential Pressure Errors

Another point is the geometry of the test tube and the fact that it cannot be changed easily without a disassembly of the apparatus. It is also not possible to take samples from the deposit built in the tube.
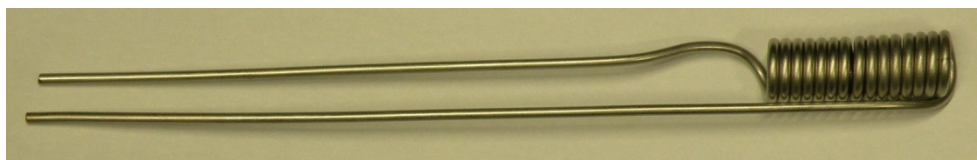
Figure 52: Test tube

Figure 52 shows a picture of the test tube. A suggestion is to use a straight tube instead of the coiled one, and a bigger diameter. There should be the possibility to disassembly it after every test run, so it could be weighted and the amount of wax could be estimated.

As the wax loop should simulate the real conditions, it should be possible to set parameters that are near on the real ones. It is not possible to simulate the turbulent flow in a pipe. Because of the dimensions of the test tube and the abilities of the pump Reynolds numbers as in turbulent flow cannot be achieved.

Further the conclusion from the measurement of the differential pressure on the wax deposit characteristics has to be investigated. It is not possible to embrace that the deposit has the same properties along the test tube; this concerns the composition and the deposit thickness, along the pipe. As the literature states the deposit thickness and the point of the magnitude will change over the distance and over time. Figure 53 shows this behaviour for field conditions but the same is true for the bench scale.
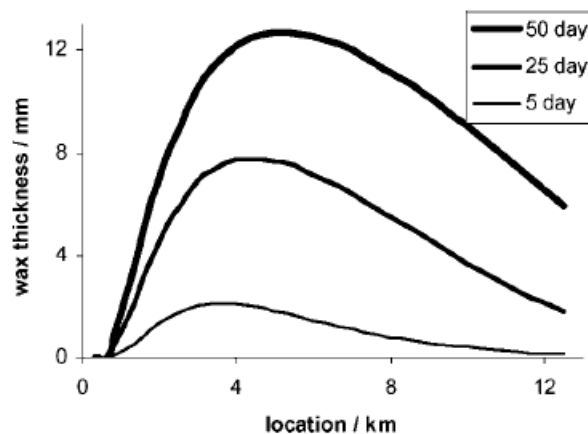
Figure 53: Deposit thickness distribution

Because this type of bench scale flow loop is a relatively new technology, now used only by some companies, it is not fully technically mature.

It´s not suggested to perform inhibitor screenings and rankings only based on the results obtained with it, but it might be a complement to viscosimeter measurements.

2   Anorganic Deposits

2.1   Precipitation and Types of Scale

The precipitation and accumulation of water formed deposits on flow surfaces is called scaling. Scale can be formed by the reservoir water itself, when the thermodynamic conditions change or it can be a result of the mixing of reservoir water with injection water (fresh water or seawater). (22)
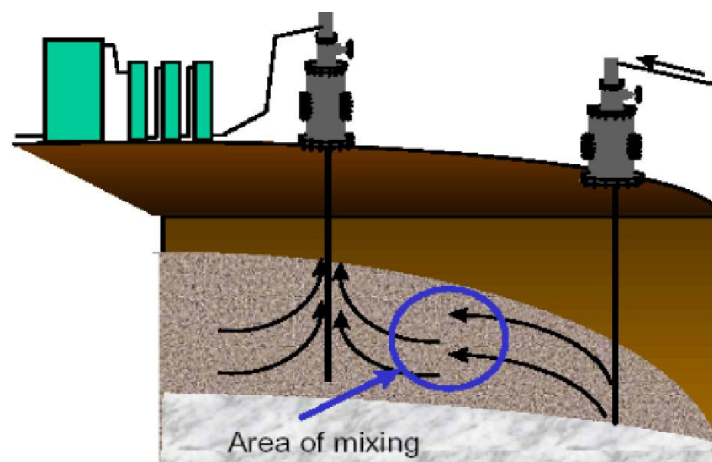
Figure 54: Mixing of reservoir water and injected water (23)

Common types of scale and the primary variables influencing the building can be seen in Table 6.

 "Scale begins to form when the state of any natural fluid is perturbed such that the solubility limit for one or more components is exceeded." (24)

Table 6: Common Oilfield Scales (9)

Name	Chemical Formula	Primary Variables
Calcium carbonate	$CaCO_3$	Partial pressure of CO2, Temperature, Total dissolved salts, pH
Calcium Sulphate: Gypsum Hemihydrate Anhydrite	 $CaSO_4.2H_2O$ $CaSO_4.1/2H_2O$ $CaSO_4$	Temperature, Total dissolved salts, Pressure
Barium Sulphate	$BaSO_4$	Temperature, Pressure
Strontium Sulphate	$SrSO_4$	Temperature, Pressure, Total dissolved salts
Iron Compounds: Ferrous Carbonate Ferrous Sulphide Ferrous Hydroxide Ferrous Hydroxide	 $FeCO_3$ FeS $Fe(OH)_2$ $Fe(OH)_3$	Corrosion, dissolved gases, pH

Figure 55: Scale deposit in a pipe

Normally if the temperature increases, the solubility increases too, because more ions are dissolved. Decreasing pressure lowers the solubility. But there are also exceptions like calcium carbonate, which shows an inverse trend for temperature changes. (25)

The dependence of solubility on temperature and the dependence on pressure (for $BaSO_4$) can be seen in Figure 56 and Figure 57.
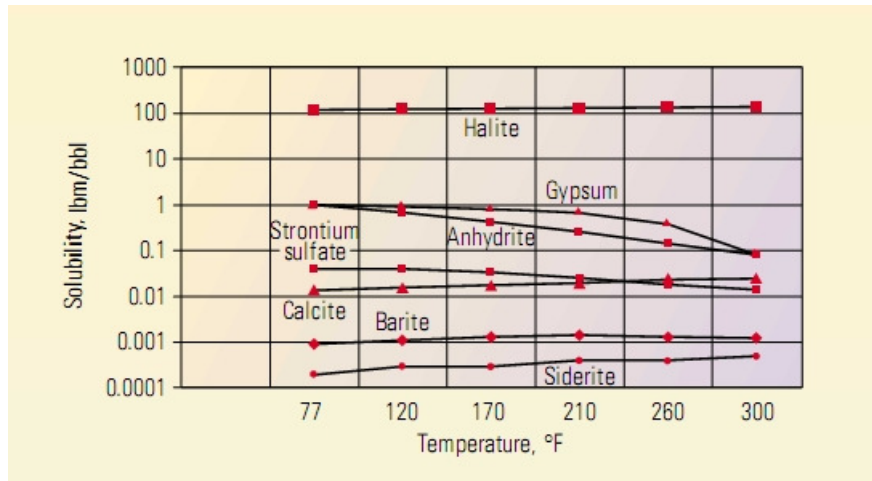
Figure 56: Scale Solubility vs. Temperature (22)
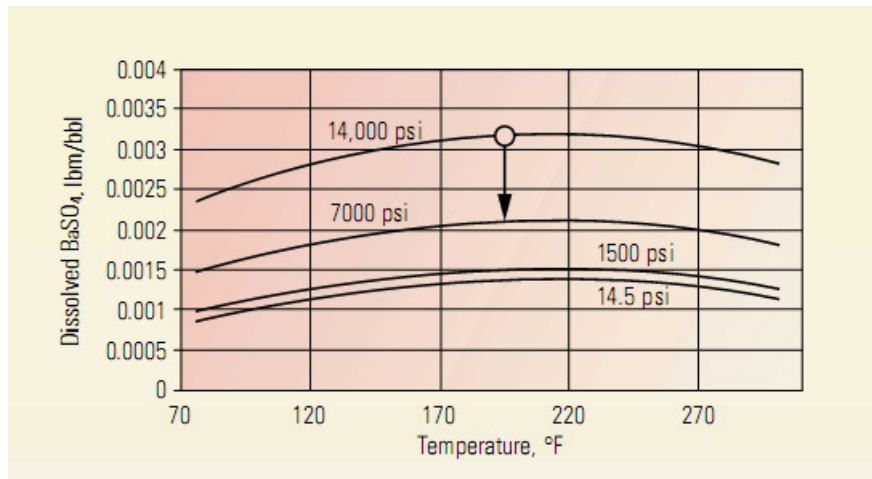
Figure 57: Scale Solubility vs. Pressure (BaSO₄) (22)

2.2   Handle Scale – Scale Inhibitors

In principle, there are two possibilities to handle scale. The first one is to prevent the precipitation and deposition of scale, the second one to remove the built scale. Removal of scale deposits can be done mechanical or chemical, dependent on the properties of the deposit.

Because removing the scale is always associated with a production stop, that costs a lot of money, the inhibition of scaling is the general practice.

There are two possibilities to achieve scale inhibition. One is to add substances that react with the scale forming ones, to reach a thermodynamically stable region, which does not allow precipitation. The second one is to add inhibitors, which suppress crystal growth.

- Dilution

  This method is mainly used to control the halite precipitation in high-salinity fluids.

  It reduces the saturation in the wellbore and the flow lines by delivering fresh water continuously.

- Chelating Inhibitors

  They consume scaling ions from the produced water. Because this happens in a stoichiometric ratio, they block the precipitation only for a limited level of oversaturation and the efficiency is poor.

- Threshold inhibitors

Threshold inhibitors maintain a supersaturated solution in an otherwise precipitating system by interacting chemically with the crystal nucleation sites. They minimize the scale deposition by a combination of scale stabilisation, by modifying the deposited scale structure and therefore preventing additional attachment and crystal dispersion (

Figure 58). This happens at concentrations of 1000 times less than the stoichiometric ratio, which makes them very cost efficient.(24)

Depending on the place of the scale problem, is it in the reservoir itself or in the pipes, two mechanisms of scale inhibition are wanted. If the scaling problem is mainly a problem in the vertical or horizontal pipes, there is no need for an inhibitor that suppresses the crystal growth of scales. The main goal is to avoid the adherence of the scale crystals to themselves or to the pipe walls. On the other hand, if an inhibitor only prevents the adherence this would not be enough in the porous medium. The crystals would be filtered out and

reduce the permeability of the pay zone. Because the brine is the same in the reservoir and the pipes, all two mechanism are needed. (26)
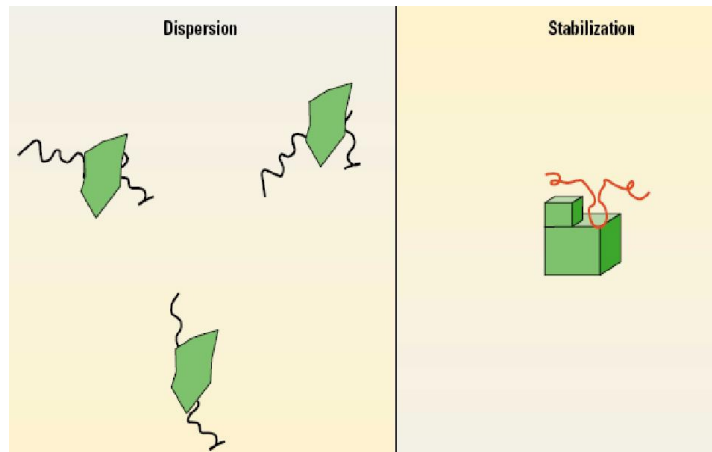
Figure 58: Working principle of threshold inhibitors (24)

2.3   Differential Scale Loop

2.3.1   Scale Inhibitor Testing Methods

Scale inhibitor can be tested in the field or in the laboratory. Testing in the field can be done with so-called scale coupons. Therefore steel coupons are placed in the flow lines. After a specific time the coupons are removed and the accumulated amount of scale is used to evaluate different inhibitors, which have been injected upstream of the coupon. The problem with this method is that the scaling tendency is only tested for the specific conditions at the coupon. Another possibility, but not a good one, is to try inhibitors in the field, and if the work use them.

Testing inhibitors in the laboratory can be done either with static or dynamic tests.

In a static or precipitation test two chemically incompatible brines, are mixed in a jar. Thermodynamic boundary conditions like temperature or pressure can be applied. Then the precipitation of solids which form the scale, for example $CaCo_3$, is measured analytically. This is done with a blank sample, without inhibitor, and with a sample containing an inhibitor. Later the amount of precipitated material is compared. The inhibitors can be ranked the following ways: The fewer solids have precipitated with the same amount of inhibitor the better the inhibitor is, or by the amount of inhibitor that is needed for the same amount of precipitated solids. This test can be done very quickly and without extensive equipment, but the results are often very poor and not reproducible.(26)
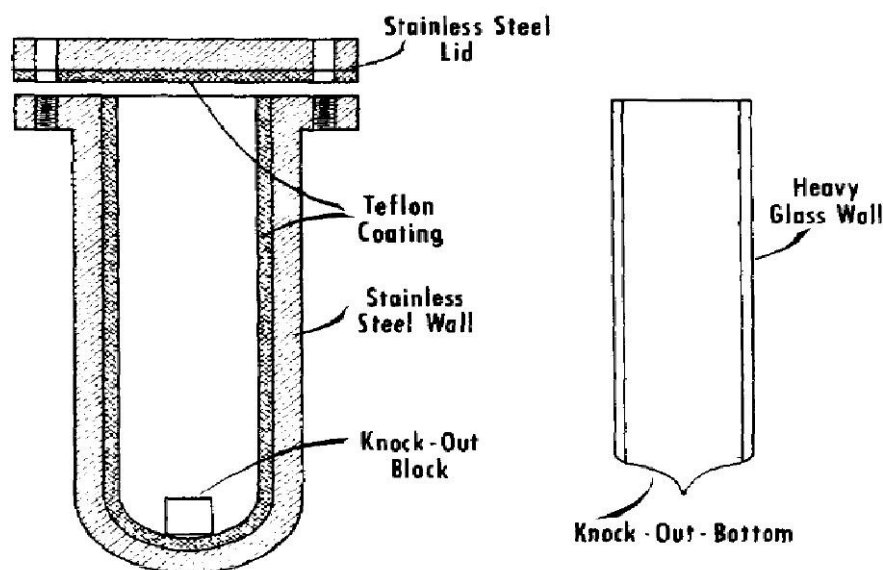
Figure 59: Static Jar test

Figure 59 shows a container for static jar testing. One solution is placed in the stainless steel jar, the other solution in the glass jar, which is then placed in the steel jar. Then the steel jar is closed, pressurized and heated to a specific temperature. Then it is shaken to break the glass, so that the brines can mix and scaling can be observed.

The differential scale loop apparatus is used to perform a dynamic tube-blocking test. It is used to evaluate the efficiency of chemical scale inhibitors. The aims are to find the minimum inhibitor concentration and to compare different inhibitors under the same test conditions. The efficiency of an inhibitor is given as the ratio of the time the test tube is blocked while pumping a fluid with inhibitor, to the time pumping the fluid alone.

The principle of this test is to measure the pressure drop or a differential pressure in a small tube versus the time a fluid is pumped through it.

Figure 60: Differential Scale Loop

The differential scale loop consists of three HPLC pumps, which pump the solutions into the device. HPLC stands for High Performance Liquid Chromatography. The big advantage of this pumps is, that they discharge exact the same volume of liquid independent of the system pressure.

One pump is for an anionic solution, one for a cationic solution and the third one for the inhibited cationic solution. It is possible to take fluid samples while operation with manually operated valves.

First the two cationic solutions are mixed, then the solutions are pumped into a heating cabinet, where they are heated to a previously defined temperature. This happens in two separated coiled capillary tubes with a diameter of 1/8". Afterwards the anionic and the cationic solutions are mixed together and pumped through a coiled capillary tub with an outer diameter of 1/16", which is the test tube. In this coiled tube, the pressure drop is measured with a differential pressure sensor. If precipitation occurs and deposits are built, the inner diameter of the tube is reduced and a higher pressure loss can be observed. Next the solution is drained trough a 1/8" capillary tube and a backpressure valve.

To remove the deposits from the previous runs it is possible to switch to a cleaning fluid with a valve setting combination.
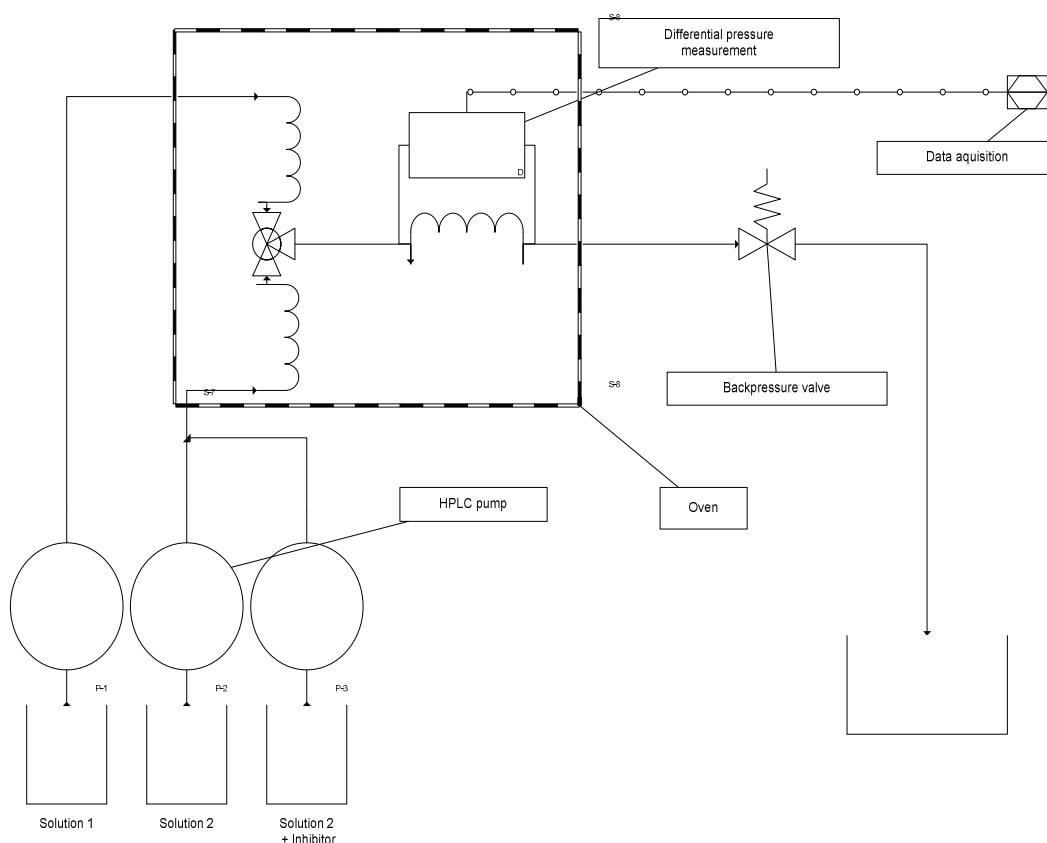
Figure 61: Differential Scale Loop Scheme

The whole apparatus is controlled with software called WinDSL. It is possible to control the components, the procedure of the run and to log the results. (27)

Backpressure Valve

With the backpressure valve, it is possible to change the test conditions. To change the backpressure, it is necessary to replace a spring in the valve. The possible value ranges from 3.4 to 51.7 bars. (27)

Because the used pumps were not able to deliver a constant rate with changing backpressure, in our test series a backpressure was not applied. The literature states that the effect of applied backpressure up to 100 bars does not have a significant influence on the $CaCo_3$ scaling rates.(28)

Rate Calibration

Before the test runs with the differential scale loop were started, the rates at different backpressures were measured. This was done with a weighing machine, which was connected to a computer. The weighing machine measured the weight at a specific interval and the computer logged this value with a timestamp. So the rate could be calculated. The total pressure was adjusted with the backpressure valve. The tested liquid was distilled water and the oven was heated to 60 °C. The observed rates before the adjustment can be seen in Table 7.

Table 7: Rates Scale Loop before Adjustment

Set value	5 ml/min		
Pump No.	Total pressure [bar]	Measured Rate [ml/min]	Deviation [%]
1	0,27	4,76	-4,76
2	0,23	4,88	-2,44
3	0,23	4,73	-5,44
1	4,86	4,67	-6,54
2	5,01	4,67	-6,54
3	5,01	4,73	-5,44
1	9,99	4,46	-10,71
2	9,67	4,55	-9,09
3	9,89	4,24	-15,19
Set value	9,99 ml/min		
Pump No.	Total pressure [bar]	Measured Rate [ml/min]	Deviation [%]
1	0,53	8,57	-14,20
2	0,51	9,23	-7,6
3	0,5	8,96	-10,36
1	4,98	8,33	-16,58
2	5,1	9,42	-5,66
3	5,1	9,18	-8,07
1	10,11	8,33	-16,58
2	9,65	8,49	-15,01
3	10,19	8,91	-10,8

Because the deviation between the set value and the measured rate were extensive, the producer of the apparatus was consulted.

The solution was to clean the ball valves of the pumps in an ultrasonic bath, to remove scale deposits in here. After that, the pumps were calibrated via the pump software, to achieve a deviation maximum of 10% for a given rate and a given backpressure.

2.4   Test Procedure

The test procedure of the differential scale loop consists of two parts. In the first part, a so-called prescale test has to be done. Here the time, in which a predefined differential pressure (in our case 0,4 bar) is reached, is measured. The cationic and the anionic solution are pumped in a 50/50 ratio, without inhibitor at a certain temperature.

The second part is the testing of the required inhibitor concentration, to prevent precipitation and deposition of scale. The test is carried out starting with higher inhibitor concentrations coming to lower. Every specific concentration is pumped through the apparatus, and if the predefined differential pressure is not reached, the concentration is lowered. Therefore, the mixture of the solutions should not take too long to reach the predefined differential pressure in the prescale test, because this time has to be waited, testing every inhibitor concentration. If the time, reaching the desired differential pressure in the prescale test is too long, the composition of the solutions, the temperature or the pressure can be changed.

2.5   Tested Solution

The scale loop apparatus was tested with synthetic brines. To avoid precipitation in the pumps, two solutions were prepared, pumped with different pumps and mixed in the apparatus before the test tube.

Prescale Test No.1

The solutions for the first test were as followed:

Solution 1:

Sodium Bicarbonate (NaHCO$_3$)	6 g	/ 1,5 l
Sodium Chloride (NaCl)	37,5 g	/ 1,5 l

Solution 2:

Calcium Chloride Dihydrate ($CaCl_2.2H_2O$)   3 g   / 1,5 l

Sodium Chloride (NaCl)   37,5 g / 1,5 l

The temperature was set to 50 °C, there was no backpressure applied and the overall pumping rate was 10 ml/min, which means 5 ml/min of solution one and 5 ml/min of solution two.
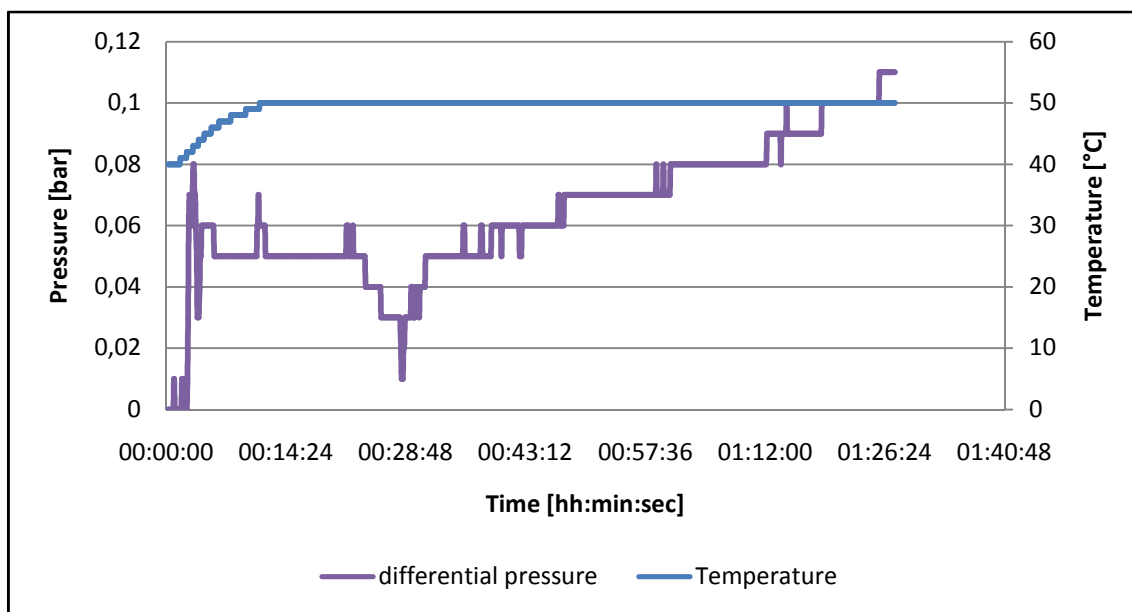
Figure 62: Prescale Test No.1

As seen in Figure 62 the desired differential pressure could not be reached in an adequate time, therefore the composition of the solutions were changed and the temperature was increased.

Prescale Test No.2

The altered solutions had the following composition:

Solution 1:

Sodium Bicarbonate ($NaHCO_3$)   6 g   / 1,5 l

Sodium Chloride (NaCl)   37,5 g / 1,5 l

Solution 2:

Calcium Chloride Dihydrate ($CaCl_2.2H_2O$)   3 g   / 1,5 l

Sodium Chloride (NaCl)   37,5 g / 1,5 l

The temperature was set to 50 °C, there was no backpressure applied and the overall pumping rate was 10 ml/min, which means 5 ml/min of solution one and 5 ml/min of solution two.
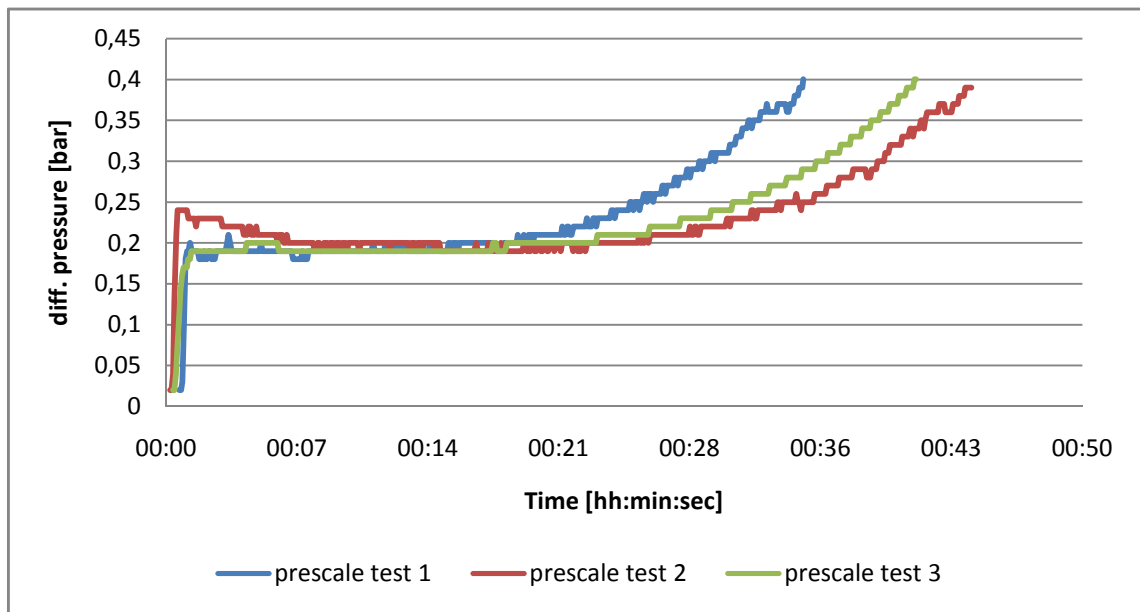
Figure 63: Prescale Test No.2

With this composition and temperature the differential pressure limit of 0,4 bar was reached in a suitable time, so the composition of the brines and the oven-temperature were used in all further tests. The differential pressure build-up of the three prescale tests is similar; the only difference is the onset. This might be due to initial nucleus. A possible reason for that is, that the cleaning process left some scale in the surface roughness of the test tube from former measurements, but it was not possible to avoid this behaviour with longer cleaning intervals.

2.6   Scaling Mechanism for the Tested Solutions

The sodium bicarbonate in the solution one dissociates in water to sodium ions and hydrogen carbonate ions.

$$NaHCO_3 \rightarrow Na^+ + HCO_3^-$$
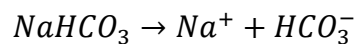
The calcium chloride in solution two dissociates in water to calcium ions and chlorine ions.

$$CaCl_2 \rightarrow Ca^{2+} + 2Cl^-$$
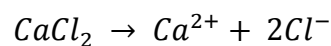
The carbon dioxide dissolves in water.

$$CO_2(g) \leftrightarrow CO_2(aq)$$
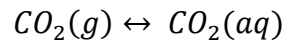
The carbon dioxide is at equilibrium with the carbonic acid.

$$CO_2(aq) + H_2O \leftrightarrow H_2CO_3(aq)$$
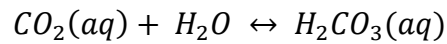
The carbonic acid is at equilibrium with the bicarbonate ion. This is dependent of the equilibrium constant Ka_1, which is $10^{-6,4}$ at 25 °C.
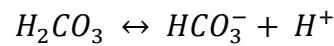
$$H_2CO_3 \leftrightarrow HCO_3^- + H^+$$

$$Ka_1 = \frac{[HCO_3^-] \cdot [H^+]}{[H_2CO_3]}$$

The hydrogen carbonate is at equilibrium with the carbonate ion. The associated equilibrium constant is Ka_2. Ka_2 has a value of $10^{-10,3}$ at 25 °C.

$$HCO_3^- \leftrightarrow CO_3^{2-} + H^+$$
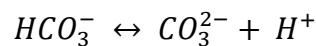
$$Ka_2 = \frac{[CO_2^{3-}] \cdot [H^+]}{[HCO_3^-]}$$

The calcium carbonate precipitates from the solution, its dependent on the solubility product Ksp. Ksp is $10^{-8,3}$ at 25 °C.
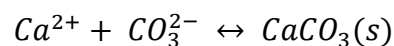
$$Ca^{2+} + CO_3^{2-} \leftrightarrow CaCO_3(s)$$

$$Ksp = \frac{[Ca^{2+}] \cdot [CO_2^{3-}]}{[CaCO_3]}$$

In the carbon dioxide saturated water the calcium carbonate is at equilibrium with the calcium bicarbonate.

$$Ca(HCO_3)_2 \leftrightarrow CaCO_3 + CO_2(g) + H_2O$$

This equilibrium is dependent on the activity a_i and further on the ionic strength I.

$$a_i = \Upsilon_i \cdot C_i$$
$$\Upsilon_i = \log I$$

The ionic strength is influenced by all the ions, including the ions built from the added NaCl. (29)

2.7   Calculation Method for the Probability of $CaCo_3$ Scale Formation

The following calculation can be used to obtain the Stability Index, which indicates the tendency of scale forming in oilfield brines (high dissolved salt waters).
$$SI = pH - pHs$$
Where pH is the actual pH value of the water and pHs is the predicted Ph value if the water is saturated with $CaCo_3$ at a given temperature T.

pHs can be calculated with the following formula.
$$pHs = K + pCa + pAlk$$
K is a constant, that depends on the temperature and on the total salt concentration.

pCa and pAlk can be calculated by the following formulas or determined by the graph in Figure 64.
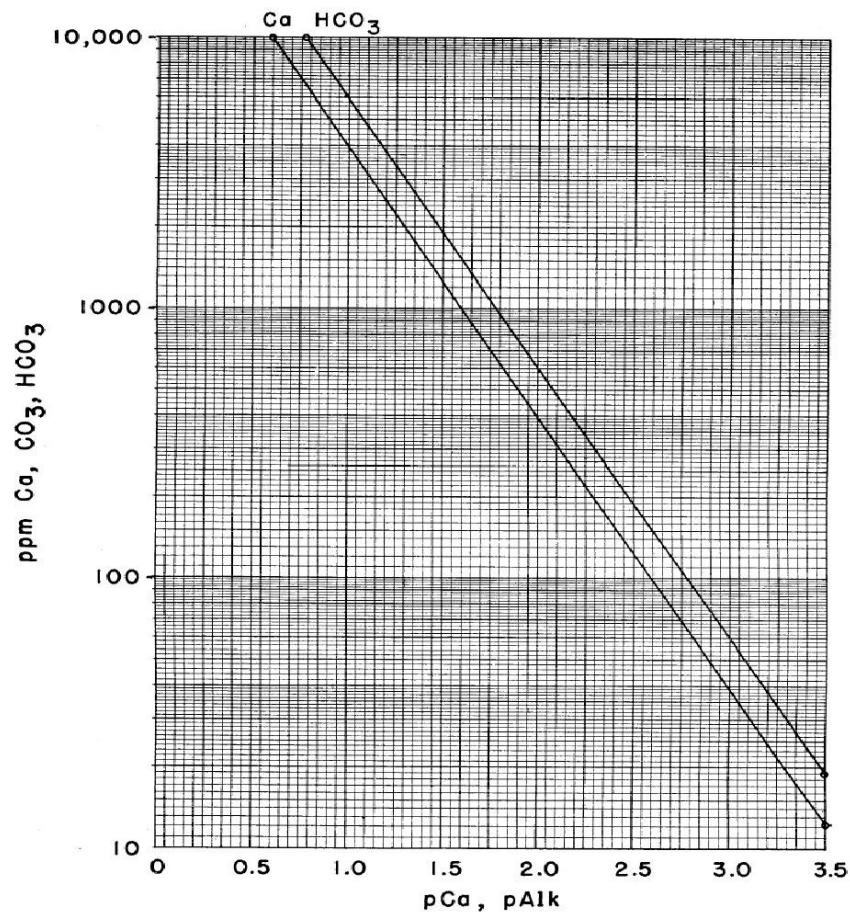
Figure 64: Determination pCa, pAlk

$$pCa = log \frac{1}{mols\ Ca^{++}/liter}$$

$$pAlk = log \frac{1}{total\ alkalinity/liter}$$

If a water is in equilibrium the Stability index is zero and K can be calculated.

$$0 = pH - K - pCa - pAlk$$

$$\rightarrow K = pH - pCa - pAlk$$

By making some measurements at equilibrium at different salt content a graph can be obtained.

If different salts are in the solution, they affect the value of K in different extent. In this case K can be found in Figure 65.
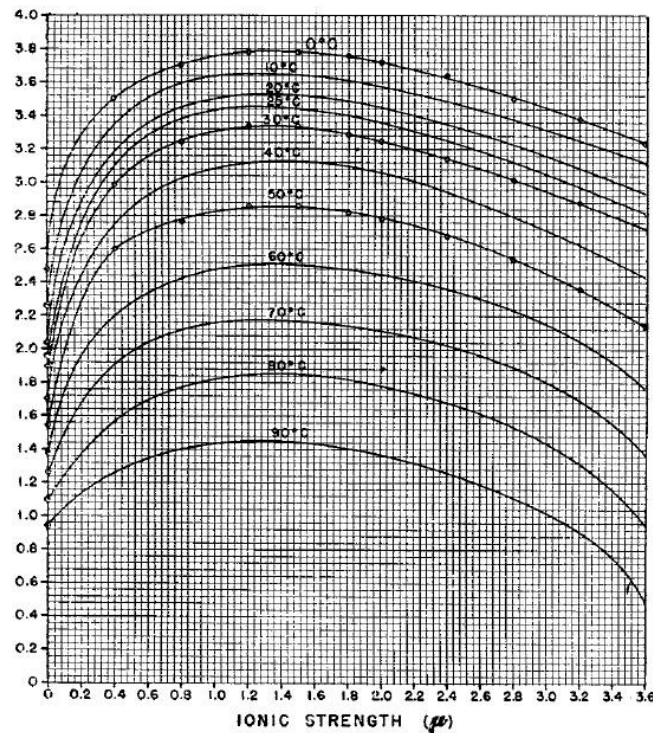
Figure 65: K values at different ionic strength

The following correction has to be made:

$$\mu = \frac{1}{2} * \sum (C_n * V_n^2)$$

Where μ is the ionic strength, C the concentration of the specific ion in gram ions per 1000 grams of solvent and V is the valence of the specific ion.

A positive value of the stability index SI (pH > pHs) means that the solution is supersaturated and precipitation is likely.

If SI = zero the solution is at equilibrium.

A negative SI value indicates that scale formation is not likely, because the solution is not saturated with calcium carbonate.(3),(30)

2.8   Inhibitor Screening

With the pump number 1 the pure anionic solution was pumped. Pump number 2 pumped the cationic solution. The third pump pumped the cationic solution too, but with the added inhibitor. The overall flow rate was always 10 ml/min. Solution 1 and 2 always had a ratio of 50:50. Because the inhibitor was needed only in small concentrations, 50 ppm or lower, it was not possible to mix the pure inhibitor with the solution. The first step was to mix 1 g of the desired inhibitor with 100 g of water. After that this 1:100 mixture was blended with the solution.

2.8.1   Scale Inhibitor No. 1
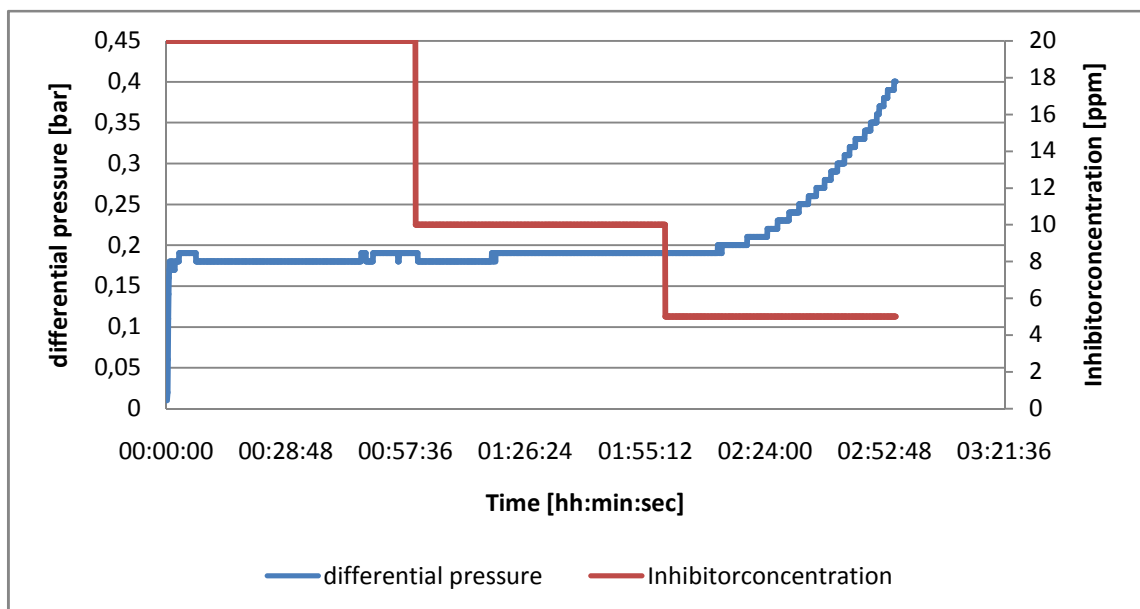
Figure 66: Performance Scale Inhibitor 1, Test 1

To achieve an overall inhibitor concentration of 20 ppm, pump number 3 pumped solution number 2 with an inhibitor concentration of 40 ppm at 5 ml/min.

In Table 8 the flow rates of the pumps and the obtained overall concentrations can be seen.

Table 8: Inhibitor concentrations – Pump rates

Pump No.	1	2	3	Total Inhibitor concentration [ppm]
Flow rate [ml/min]	5	0	5	20
Flow rate [ml/min]	5	2,5	2,5	10
Flow rate [ml/min]	5	3,75	1,25	5

As seen in Figure 66 at inhibitor concentrations of 20 and 10 ppm the differential pressure did not rise. With an inhibitor concentration of 5 ppm a deposition and the linked rise in pressure was observed. So the effective inhibitor concentration was between 10 and 5 ppm. To verify the results from the first test and to appraise the deviation from repeated tests, it was repeated one more time.
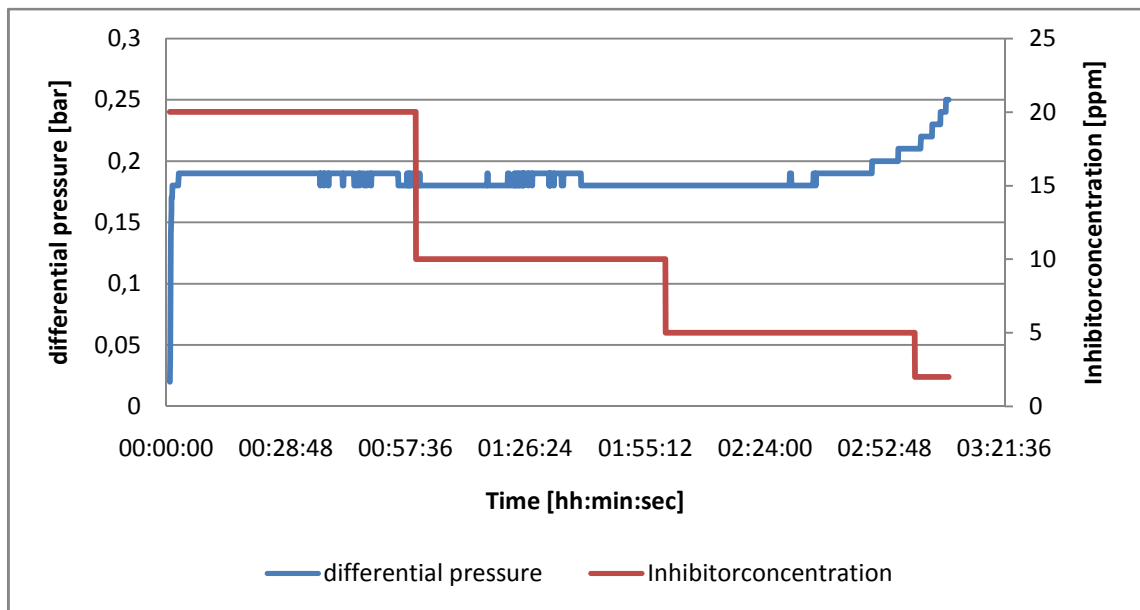
Figure 67: Performance Scale Inhibitor 1, Test 2

Knowing the effective concentration between 10 and 5 ppm the next test was made starting with 8 ppm inhibitor concentration. Figure 68 shows that 8 ppm are necessary to avoid scale building and deposition.
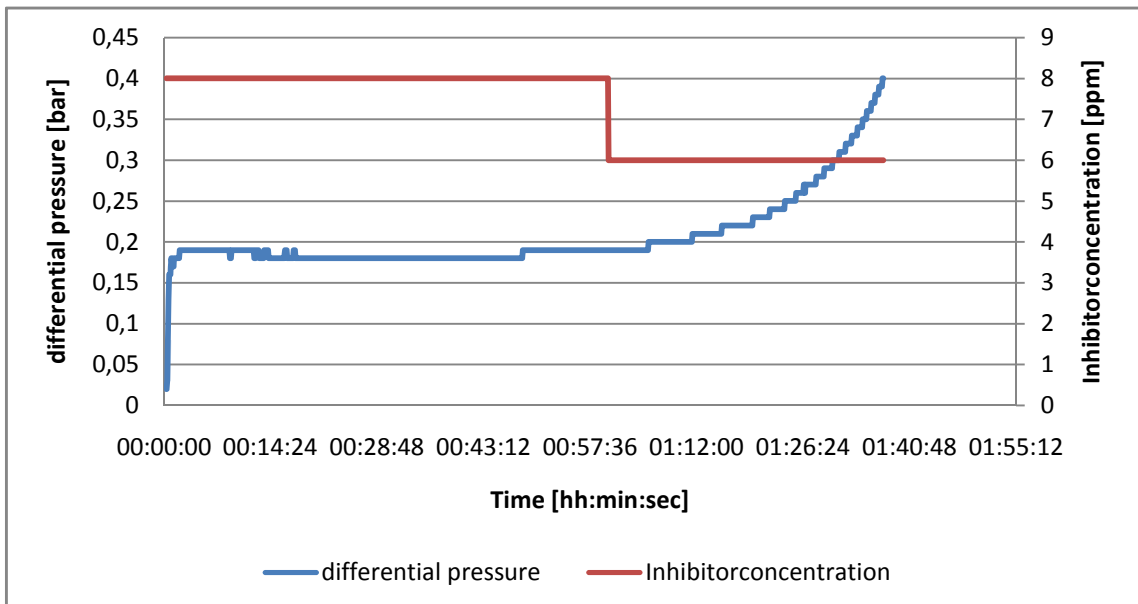
Figure 68: Performance Scale Inhibitor 1, Test 3

2.8.2 Scale Inhibitor No. 2
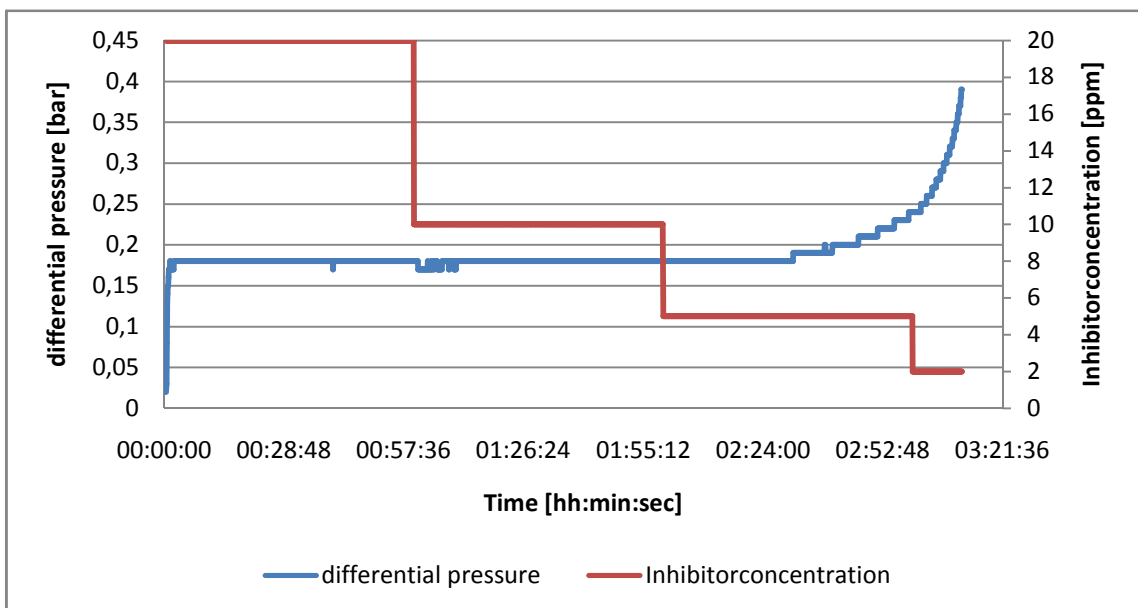
Figure 69: Performance Scale Inhibitor 2, Test 1

For Inhibitor number 2 the procedure was repeated. The first run (Figure 69) shows that the effective concentration is again between 10 and 5 ppm.
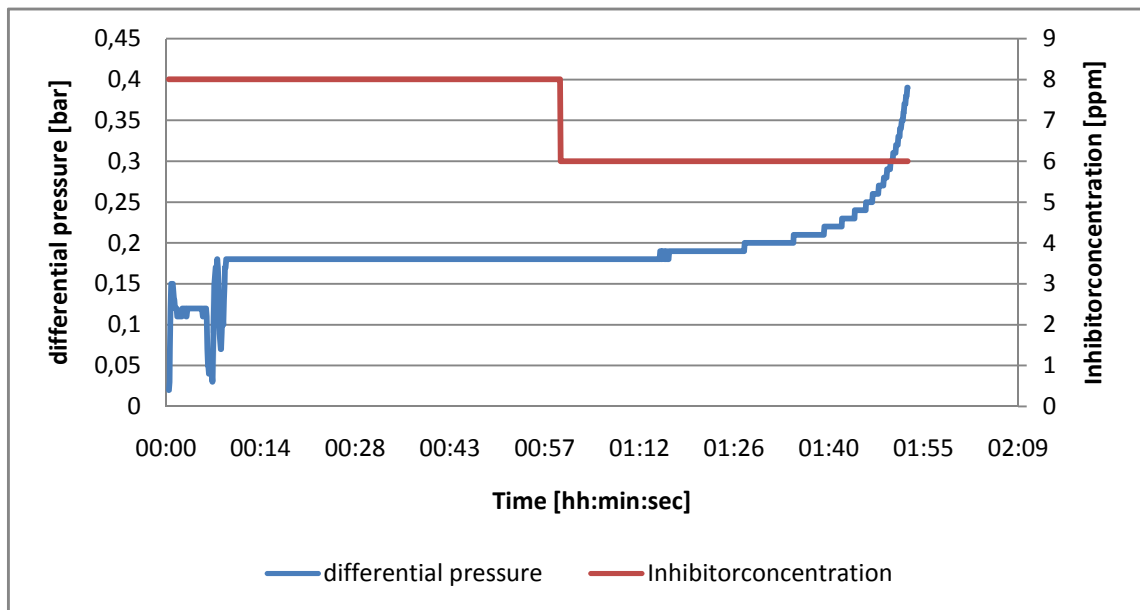
Figure 70: Performance Scale Inhibitor 2, Test 2

In Figure 70 it can be seen that a concentration of 8 ppm is necessary to avoid deposition in the test tube.

2.8.3 Scale Inhibitor No. 3

Scale inhibitor No. 3 was tested with 40 ppm. Because the pressure started to rise at this concentration already no further tests were made.
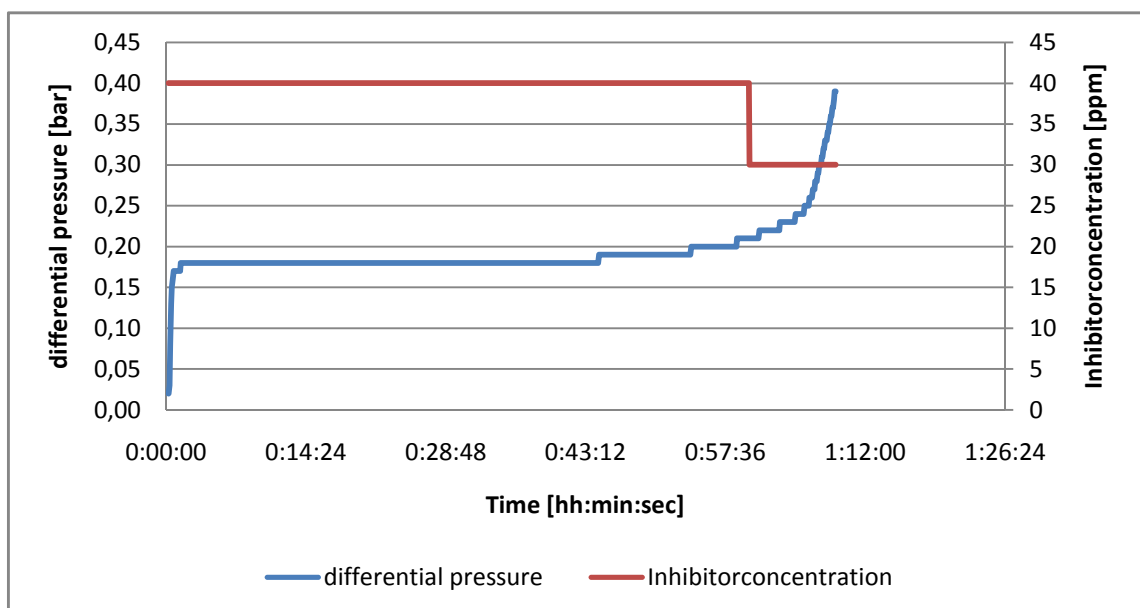
Figure 71: Performance Scale Inhibitor 3

2.8.4 Scale Inhibitor No. 4

Inhibitor No. 4 showed in the first run that the effective concentration is between 20 and 10 ppm.
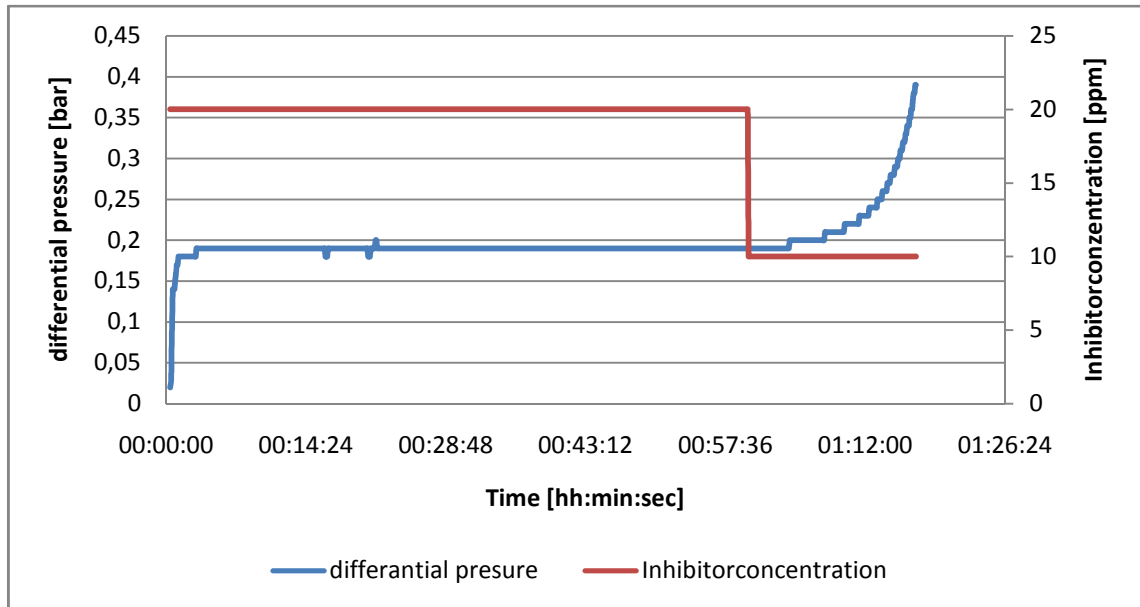
Figure 72: Performance Scale Inhibitor 4, Test 1
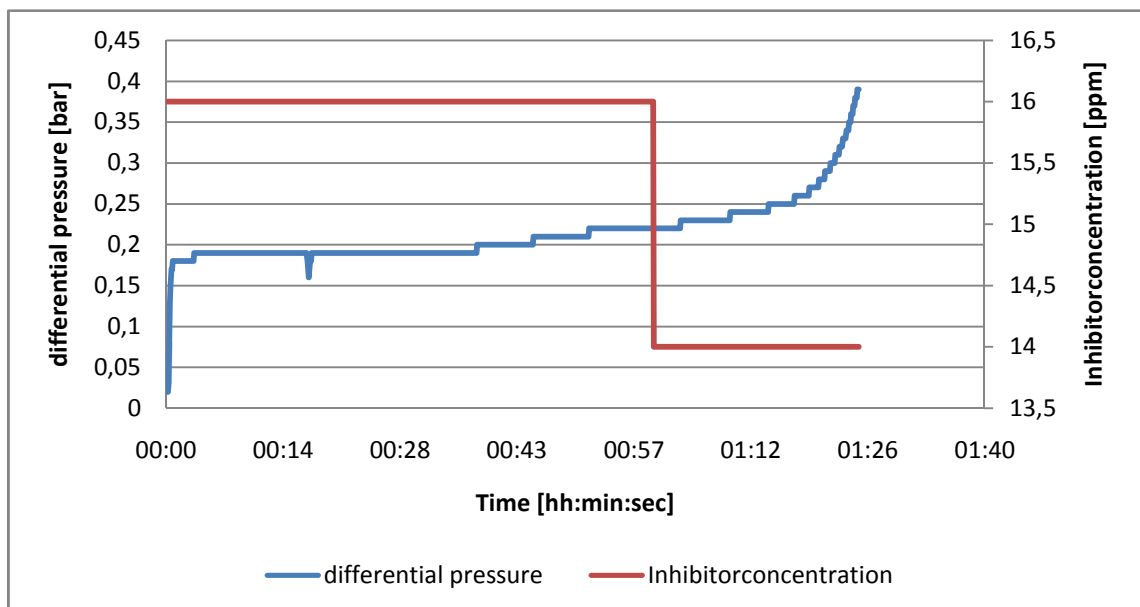
Figure 73: Performance Scale Inhibitor 4, Test 2

To appraise the exact concentration a run with the concentrations 16 and 14 ppm was done. Because the differential pressure raised at 16ppm already it can be concluded that 18ppm are necessary.

2.8.5 Scale Inhibitor No. 5

The first run of inhibitor No. 5 showed an effective concentration somewhere between 5 and 2,5 ppm.
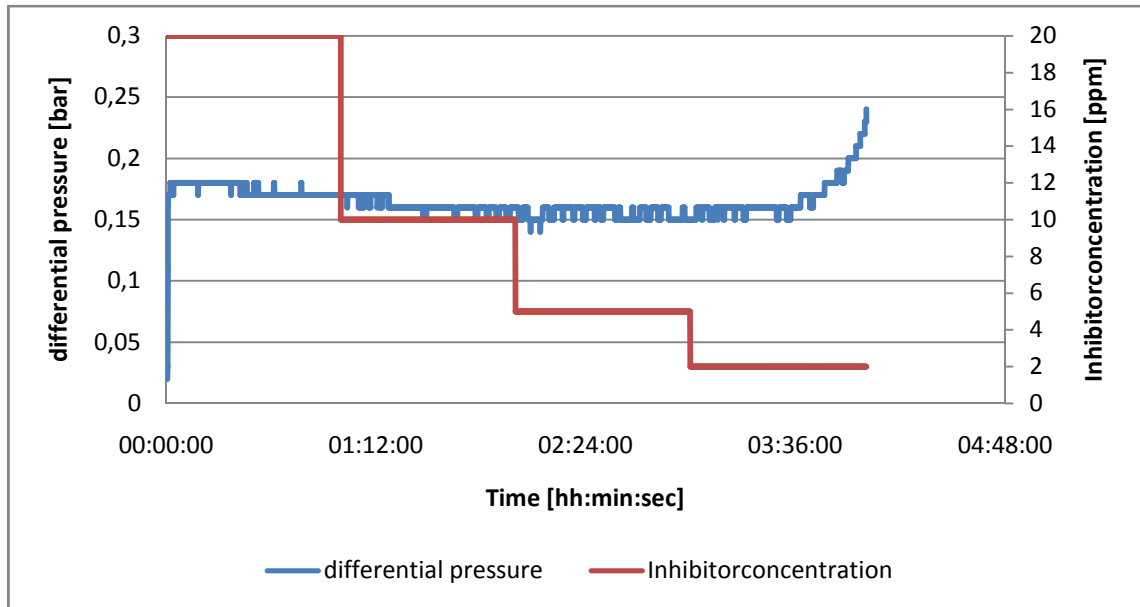
Figure 74: Performance Scale Inhibitor 5, Test 1

The second run showed that a concentration of 6 ppm can avoid a deposition in the test tube.
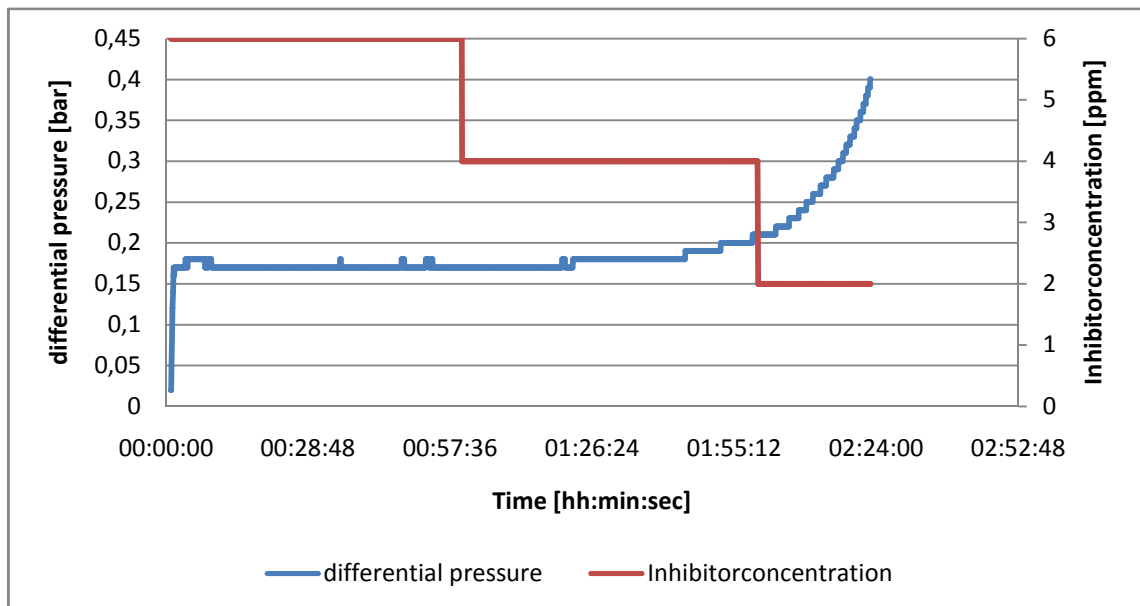
Figure 75: Performance Scale Inhibitor 5, Test 2

2.8.6 Scale Inhibitor Ranking and Comparison of Measurements with other Apparatus

The following table shows a comparison of the needed concentration of the inhibitors 1 to 5 for the synthetic brine tested above.
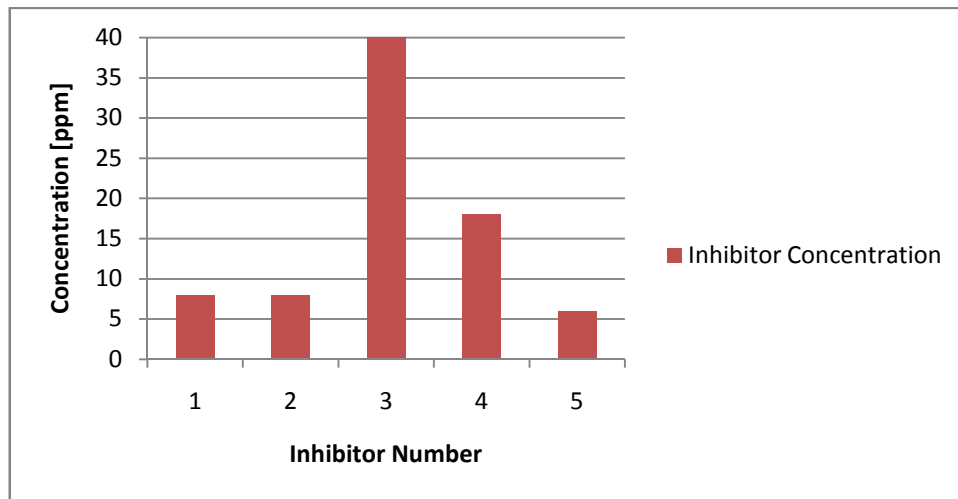
Figure 76: Comparison Scale Inhibitors

Inhibitor No. 5 is the preferred product for the tested solutions. Inhibitor No. 1 and 2 showed a fair efficiency, No. 3 and 4 need a too high concentration, which is economically not justifiable.

Table 9: Ranking of Scale Inhibitors

Tested Sample	Synthetic Water	Synthetic Water	Matzen 172	Turnu Est	Sampetru	Sampetru
Method	New Scale Loop	Old Scale Loop	Old Scale Loop	Old Scale Loop	Old Scale Loop	Old Scale Loop
Rank	Inhibitor Number	Inhibitor Number	Inhibitor Number	Inhibitor Number	Inhibitor Number	Inhibitor Number
1	5	1	3	4	5	1
2	1	3	4	3	4	3
3	2		1	1	3	2
4	4		2	5	1	5
5	3		5	2	2	4

Table 9 shows a ranking for the tested scale inhibitors. Results derived from this test series with the new differential scale loop are compared with the results of former testings with a different apparatus and a different test method. The method used earlier was as followed:

- The test was started with the blank sample and it was waited until a scale deposition was built and the differential pressure raised.

- At a certain differential pressure the scale inhibitor was added to the solution beginning with small concentrations leading to higher ones

- The flattening of the differential pressure curve was used to rank the inhibitors

Due to the different test method the results are comparable one to one.

2.9   Limits of use and Suggestions

A main weak point of the differential scale loop used is, that only brines can be tested, which will not precipitate in the pumps or in the preheating coils. This means if inhibitors should be screened for brines from the field, the brines have to be analyzed in the laboratory and then synthetically reproduced. The brine has to be separated into an anionic and a cationic solution to avoid precipitation before the test tube. Another point is that all the hoses of the apparatus, which forward the fluids to the pumps have to be refilled after a measurement brake, to avoid air bubbles.

Further measurements for the pressure dependence of scale building have to be done, because the pressure has beside the temperature a great impact. This might be a very time consuming task, as the pumps do not deliver the same rate if the backpressure is changed. To calibrate the pump the flow rate has to be measured from every single pump and changes in the pump software have to be made. Another topic to investigate may be the cross sensitivities between different inhibitors. This might be scale and corrosion inhibitors or other chemicals used in the oilfield. In addition, the impact of iron or other minerals in the brine has to be tested.

2.10   Detailed Work Instruction

Differential Scale Loop

Brief Instruction

The values in the brackets were used for calcium carbonate scaling.

1. Switch the computer and the scale loop on
2. Start Program WinDSL
3. Window WinDSL V1.21:
   →Extras → Ventile und Pumpen:

   - Switch off pump 1-3 , red status light

   - Switch off „Kolbenhinterspülung"

4. Ensure that the hoses for the „Kolbenhinterspülung" are completly filled with water (no air bubbels visible)
   - open the clamps on the hose pump and fill the hoses S1-S3 with destilled water (press out the air bubbles completely) with a syringe
   - close the clamps
   - Extras → Ventile und Pumpen: Switch on „Kolbenhinterspülung" and verify distilled water is dropping from the hoses
5. Ensure that the hoses for pump 1 to 3 are completly filled with water (no air bubbels visible)
   - fill the hoses S1-S3 with distilled water (press out the air bubbles completely) with a syringe
   - Extras → Ventile und Pumpen: Switch on pumps 1 to 3 separately with a flow rate of 5 ml/min select "Reinigungsmittel 1" and verify distilled water is drooping from the outlet
6. Switch off the pumps and close the window "Ventile und Pumpen"
7. Window WinDSL Programmfenster:
   →Ablauf:

   - Enter the pressure threshold for the abortion of the prescale test (0,4bar)

   - Enter the overall flow rate (10ml/min)

   - Enter the ratio L1 : L2,L3 (1:1)

   - Press "Reinigung Definieren"

        Select for Step 1 to Step3 the needed fluid and enter the flow rates and

        durations

		Pumpe 1	Pumpe 2	Pumpe 3	Duration
Schritt 1	Reinigungsmittel 1 (destilliertes Wasser)	9 ml/min	9 ml/min	9 ml/min	2 min
Schritt 2	Reinigungsmittel 2 (Lösungsmittel)	3 ml/min	3 ml/min	3 ml/min	10 min
Schritt 3	Reinigungsmittel 1 (destilliertes Wasser)	5 ml/min	5 ml/min	5 ml/min	30 min

- Enter the duration the bypass vent stays closed (2min)

- Press OK to close the window

- Enter the oven temperature (60 °C)

- Enter the maximum duration of the test (3600 sec)

Prescale Test

8. Window WinDSL Programmfenster:
   → Ablauf:

   - Select for Step 1 Prescale, the threshold pressure (0,4 bar) and the time limit (3600 sec), enter the oven temperature (60 °C)

9. Window WinDSL V1.21:
   Select " Setze Temp." and enter the needed temperature of the oven (60 °C)

10. Wait until the temperature is reached

11. Window WinDSL Programmfenster:
    → Ablauf:

    - Press "Starte Vortest"

12. Enter the flow rate (10ml/min)

13. Enter the ratio "Pumpe 1: Pumpe 2" (50:50)

14. Press "Start"

15. Enter the desired filename and directorywhere you want to save your data and press "Speichern"

16. Window WinDSL V1.21:
    Press "Anzeige groß" to display the absolute and the differential pressure

Inhibitor Testing

8. Window WinDSL Programmfenster:

   → Ablauf:

   - Enter the pressure threshold for the abortion of the inhibitor test (0,4 bar)

   - Enter the maximum duration of the test (3600 sec), dependent on the prescale test

- Define your inhibitor testing steps: Enter as many scale lines as you need and define the inhibitor conzentration, threshold pressure and maximum time

→Lösungen

Enter the concentration of the inhibitor in solution 3

→Fluide:

Enter the available fluid quantities and confirm

9. Window WinDSL V1.21:

Select " Setze Temp." and enter the needed temperature of the oven (60 °C)

10. Window WinDSL Programmfenster:

→Ablauf:

- Press "Start"

- Enter the desired filename and directory, where you want to save your data and press "Speichern"

11. Window WinDSL V1.21:

Press "Anzeige groß" to display the absolute and the differential pressure

3 Summary

OMV Exploration and Production purchased two new apparatus, a differential wax loop and a differential scale loop. The scope of the work was to place the machines into operation, to find suitable parameters for measurements and to test wax and scale inhibitors. Therefore, the first part of this work is about organic or wax depositions and the second one about anorganic or scale depositions.

In the part about the wax loop the first paragraphs concern the paraffin wax itself, its characterization and the principles of precipitation and deposition in pipes. An overview about the handling of wax problems is given and different classes of chemicals that impede precipitation and deposition are discussed. Necessary parameters, which have to be known to set parameters for loop testing, are described along the measurement techniques to determine them. The build-up of the used apparatus is described in detail, the limits of usability are shown and improvements are suggested. Later the preparation of the samples is discussed and five inhibitors from different companies are tested and ranked on the results. A comparison of the results obtained with the wax loop and former viscosimeter test completes the first part.

The second part is about anorganic deposits and the differential scale loop in special .First types of scale and their occurrence are characterized. Then various types of chemical scale inhibitors and the general handling of scaling problems are described. The main focus of attention was, as in the wax loop part, the build up of the apparatus itself and the necessary parameters for testing. The scaling mechanism of calcium carbonate is explained and an overview of a calculation method, which can be used to predict precipitations, is given. In the last part of this chapter five scale inhibitors have been tested with a synthetic brine and ranked. Further the results are compared to a different test method.

4   References

1. **Speight, James G.** *Handbook of Petroleum Analyses.* Canada : Wiley-Interscience, 2001.

2. **Hyne, Norman J.** *Dictionary of Exploration, Drilling & Production.*

3. **Odgen, P.H.** *Chemicals in the Oil Industry.* London : The Royal Society of Chemistry, 1983. Vol. Special Publication No 45.

4. **Alboudwarej, H., Huo, Z. and Kempton, E.** Flow-Assurance Aspects of Subsea Systems Design for Production of Waxy Crude Oils. *Paper SPE 103242.* 2006.

5. **Leonartis, Kosta J. and Leonartis, John D.** Cloud Point and Wax Deposition Measurement Techniques. *Paper SPE 80267.* 2003. prepared for presentation at the SPE International Symposium on Oilfield Chemistry held in Houston, Texas, U.S.A., February 5-7.

6. **Hammami, Ahmed and Raines, M. A.** Paraffin Deposition from C:rude Oils: Comparison of Laboratory Results with Field Data. *SPE Journal.* March 1999, Vol. 4.

7. **Pedersen, Karen Schou and Christensen, Peter L.** *Phase Behavior of Petroleum Reservoir Fluids.* s.l. : Taylor and Fancis, 2007. ISBN 0-8247-0694-3.

8. **Nguyen Phuong Tung, SPE, and Nguyen Thi Phuong Phong, and Bui Quang Khanh Long, IMS-Vietnam NCST, and.** Studying the Mechanisms of Crude Oil Pour Point and Viscosity Reductions When. *Paper SPE 65024.* 2001.

9. **Huijgen, Martijn.** *Paraffin Treatment - Flow Management.* s.l. : Champion Technologies, 2006. Presentation at OMV.

10. **Parthasarathi, Prashanth and Mehrotra, Anil K.** Solids Deposition from Multicomponent Wax-Solvent Mixtures in a Benchscale Flow-Loop Apparatus with Heat Transfer. *Energy & Fuels.* 19, 2005, pp. 1387-1398.

11. **Edmonds, Beryl, et al.** Simulating Wax Deposition in Pipelines for Flow Assurance. [ed.] American Chemical Society. *Energy & Fuels.* 2008, Vol. 22, pp. 729-741.

12. **Banki, Reza and Firoozabadi, Abbas.** Modeling of Wax Deposition in Pipelines from Irreversible Thermodynamics. *Paper SPE 77571.* 2002.

13. Schlumberger Oilfield Glossary. [Online] Schlumberger Limited, 2008. [Cited: 11 09 2008.] http://www.glossary.oilfield.slb.com/Display.cfm?Term=cloud%20point.

14. Schlumberger Oilfield Glossary. [Online] Schlumberger Limited, 2008. [Cited: 11 07 2008.] http://www.glossary.oilfield.slb.com/Display.cfm?Term=pour%20point.

15. **Venkatesan, Ramachandran, Singh, Probjot and Fogler, Scott H.** Delineating the Pour Point and Gelation Temperature of Waxy Crude Oils. *Paper SPE 72237*. 2002.

16. **Center, Material Research.** Rheological. [Online] [Cited: 27 12 2008.] http://www.usm.edu/mrsec/facilities/rheological.htm.

17. **Ronningsen, Hans Petter and Bjorndal, Brit.** Wax Precipitation from North Sea Crude Oils. [ed.] American Chemical Society. *Energy & Fuels.* 11 june 1991.

18. **Systemtechnik, PSL.** PSL Systemtechnik GmbH: Hintergründe. [Online] 18 09 2008. [Cited: 23 09 2008.] http://www.psl-systemtechnik.de/133.html?&L=0.

19. **Zougari, Mohammed and Jacobs, Scott.** Novel Organic Solids Deposition and Control Device for Live-Oils: Design and Applicatios. [ed.] American Chemical Society. *Energy and Fuels.* 2006, 20.

20. *Handbuch Differential Wax loop.* s.l. : PSL Systemtechnik GmbH, 2007.

21. **Misra, Sanjay, Baruah, Simanta and Singh, Kuiwant.** Paraffin Problems in Crude Oil Production And Transportation: A Review. *Paper SPE 28181.* 21 09 1994.

22. **Hasson, David, et al.** Influence of the flow system on the inhibitory action of CaCo3 scale prevention additives. *Desalination.* 1996, 108.

23. **Carageorgos, T., Marotti, M. and Bredrikovetsky, P.** A New Method To Characterize Scaling Damage From Pressure Measurements. *Paper SPE 112500.* 2008.

24. **Crabtree, Mike, et al.** Fighting Scale - Removal and Prevention. *Oilfield Review.* Autumn 1999.

25. **Chung, Ting-Horng.** Thermodynamic Modeling for Organic Solid Precipitation. *Paper SPE 24851.* 1992. prepared for presentation at the Annual Technical Conference and Exhibitionheld in Washington, DC, October 4-7.

26. **Vetter, O. J.** An Evaluation of Scale Inhibitors. *Journal of Petroleum Technology.* 1972.

27. *Handbuch Differential Scale Loop.* s.l. : PSL Systemtechnik GmbH, 2007.

28. **Zhang, Yuping and Farquhar, Rod.** Laboratory Determination of Calcium Carbonate Scaling Rates for Oilfield Wellbore Environments. *Paper SPE 68329.* 2001.

29. **Garrels, Robert M. and Christ, Charles L.** *Solutions, Minerals, and Equilibria.* Boston : Jones and Bartlett Publishers, 1990. ISBN 0-86720-148-7.

30. **Stiff, Henry A. and Davis, Lawrence E.** A method for predicting the tendency of oil field waters to deposit calcium carbonate. *Petrolium Transactions.* 1952, Vol. 195.

31. **Mackay, E.J.** Modeling In-Situ Scale Deposition: The Impact of Reservoir and Well Geometries and Kinetic Reaction Rates. *Paper SPE 81830.* 2003. presented at the SPE Oilfield Scale Symposium, Aberdeen, January 30-31.

32. **Moghadasi, J., et al.** Scale Formation in Iranian Oil Reservoir and Production Equipment During Water Injection. *Paper SPE 80406.* 2003. prepared for presentation at the 5th International Oilfield Scale Symposium and Exibithion held in Aberdeen, January 29-30.

33. **Bazin, B., Kohler, N. and Zaitoun, A.** Some Insights Into the Tube Blocking Test Method to Evaluate the Efficiency of Mineral Scale Inhibitors. *Paper SPE 96560.* 2005. prepared for presentation at the SPE Annual Technical Conference and Exhibition held in Dallas, October 9-12.

34. *Handbuch Pour Point Tester 45150.* s.l. : PSL Systemtechnik, 2007.

35. **Ratulowski, J., et al.** Flow Assurance and Subsea Productivity: Closing the loop with connectivity and measurements. *Paper SPE 90244.* 2004.

36. **Kabir, C. S., Hasan, A. R. and Wang, X.** An Approach to Mitigating Wellbore Solids Deposition. *Paper SPE 71558.* 2001.

5 Appendix

5.1 Scale Inhibitors

Table 10: Tested Scale Inhibitors

Inhibotor No.	Product Name	
1	POCAS2020	Na-Polycarboxylate
2	SCW80650	Polycarboxylate
3	SCW82556	Carboxylate
4	SI4572	Na- Aspartat
5	SI4041	Na- Phosphonate

5.2 Paraffin Inhibitors

Table 11: Tested Paraffin Inhibitors

Inhibotor No.	Product Name
1	WM1840
2	CW288
3	PAO042
4	PI780
5	PAO87119

5.2.1 Champion Technologies – Flexoil WM1840

The Flexoil WM1840 is a polymeric pour point depressant. The action is based on modification of wax crystals in the crude oil.

Table 12: Physical properties of FLEXOIL WM1840

Appearance	Clear colored liquid
Density @ 20°C [kg/m3]	890
Viscosity @ 20°C [mPa.s]	15
Flash Point [°C]	>62
Pour Point [°C]	-9
Field of application	Crude with low to moderate wax content
Dosage rate [ppm]	50 - 1000
Solubility	Soluble in most aromatic solvents

5.2.2 Champion Technologies – Flexoil CW288

Table 13: Physical properties of FLEXOIL CW288

Appearance	White liquid, dispersion
Density @ 20°C [kg/m3]	925
Viscosity @ 20°C [mPa.s]	500 - 900
Flash Point [°C]	85
Pour Point [°C]	>-40
Field of application	Highly paraffinic crude oils
Dosage rate [ppm]	25 - 1000
Solubility	Soluble in hydrocarbon solvents at low concentrations

5.2.3 Baker Hughes – PAO42

Table 14: Physical properties of PAO42

Appearance	Slightly hazy, gold liquid
Density @ 16°C [kg/m3]	850
Viscosity @ 16°C [SUS]	50
Flash Point [°C]	17
Pour Point [°F]	15
Field of application	
Dosage rate [ppm]	100 - 2000
Solubility	Oil soluble

Injection at least 20°F above cloud point is preferred.

5.2.4   Baker Hughes – PAO83119

Table 15: Physical properties of PAO83119

Appearance	Waxy, yellow to brown liquid
Density @ 50°C [kg/m3]	870
Viscosity @ 50°C [cST]	50
Flash Point [°C]	6
Pour Point [°F]	
Field of application	
Dosage rate [ppm]	100 - 1000
Solubility	Soluble in aromatic solvents,slightly in water

5.2.5   MI Production Chemicals – PI780

The PI-780 contains an ethylene/vinyl acetate polymer working as a wax inhibitor and a surfactant with prevents the wax particles from adhering to solid surfaces and to each other. Further it will dissolve and disperse already formed wax crystals.

Table 16: Physical properties of PI780

Appearance	Clear, yellow liquid
Density @ 20°C [kg/m3]	900
Viscosity @ 50°C [cST]	50
Flash Point [°C]	26
Pour Point [°C]	-1
Field of application	
Dosage rate [ppm]	10 - 100
Solubility	Hydrocarbon soluble