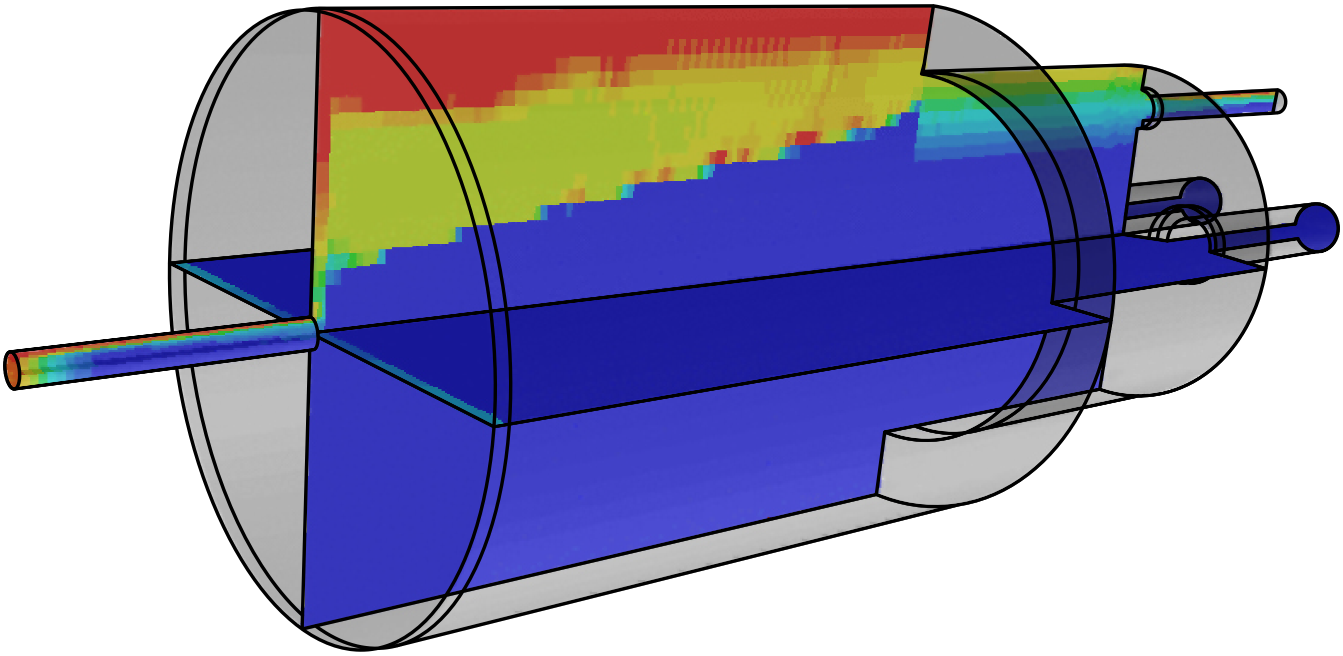
Coreflood testing

Recommended reservoir-condition coreflood test method for formate brines and fluids



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Summary

Obtaining meaningful results from reservoir-condition coreflood testing with formate fluids and brines has proven hard due to severe artifacts introduced by water saturation effects that are poorly understood and not given enough attention by test laboratories. Misunderstood water saturation effects can affect the effective permeability to oil and gas to such an extent that significant reductions in return permeability are measured. This alteration of effective permeability is typically labeled 'filtrate retention' and reported as severe formation damage, whilst a better description is 'an illusion created by poor attention to experimental procedure'.

Frequent users of formate drilling and completion fluids are aware of how difficult it is to perform meaningful coreflood testing of formate brines and no longer demand that the fluids pass such tests. Operators that are not as familiar with the fluids, would however, want to conduct a standard coreflood test to qualify the fluid for first-time use in their reservoirs. It is therefore important for Sinomine Specialty Fluids to be able to recommend a coreflood test method that can give meaningful results.

Unfortunately, Sinomine cannot recommend a test method that fully identifies and quantifies permeability alterations caused by temporary and tractable changes in water saturation. However, by adding additional steps to 'standard' coreflood procedure, useful knowledge can be gained about the sources of the permeability alterations. The coreflood test method recommended in this document includes many steps that are helpful when analyzing the mechanism of any return-permeability alteration and helps determine whether any permeability alteration is a result of temporary and tractable water saturation changes or real formation damage with permanent impairment.

1 Introduction

Since the introduction of low-solids/solids-free formate drilling, perforating, and screen-running fluids in the 1990s, formate fluids have earned a well-deserved reputation for enhancing well productivity. Unfortunately, this excellent field performance is most often not reflected in their performance in laboratory coreflood tests. Laboratory test results from flooding small core plugs have sometimes been so bad that operators have been seriously concerned and planned for acid treatment as a contingency. However, acid treatment has never been needed in the field.

Laboratory coreflood tests on small plugs of rock are extensively used to evaluate, compare, and rank the possible formation damage potential of prospective drilling and completion fluids prior to a new field development. The core plugs are first exposed to the test fluids at overbalance, under reservoir conditions, to simulate a period of drilling and completion. The recovery of the effective permeability of the core plug to gas or oil, termed return permeability, is then measured after a period of simulated drawdown with gas or oil. Any reduction in effective permeability of the cores to oil or gas after test-fluid exposure and simulated drawdown may indicate one or more of the following:

- Incomplete removal of the test fluids from the core by the selected drawdown procedure.
- Temporary or permanent obstruction of pore throats by insoluble or polymeric test-fluid components.
- Temporary or permanent obstruction of pore throats by products of interactions between the test fluid and the rock matrix or the original contents of the pores.

Despite their widespread use for screening and selecting reservoir drill-in and completion fluids, there is no agreed industry-wide method or recommended practice for coreflood testing. Each test laboratory has its own preferred method, and results can vary widely and randomly. The last attempt at test standardization between laboratories, made in 1999, ended in failure (Marshall et al. 1999). To make matters worse, formation damage predictions obtained from coreflooding tests with drilling and completion fluids are rarely, if ever, compared against actual well performance over the lifetime of the wells. Without this essential validation and calibration of laboratory test results against well performance, the tests have limited practical value.

Notwithstanding their limited predictive value, uncalibrated and unvalidated coreflood testing of formate fluids has been conducted frequently since the early 1990s.

Analyses of hundreds of old coreflood test results with formate brines have taught us some important lessons about how the monovalent 'reservoir-water-like' formate brine behaves when it is injected into a core plug. A typical outcome after formate brine has entered a core is that the chosen drawdown procedure does not reduce the residual water levels in the core back to the initial water saturation level. The presence of formate brine inside the core at levels exceeding the initial water saturation decreases the relative or effective permeability to oil and gas and thereby produces a bad coreflood test result with damage mechanism reported as '**filtrate retention**'.

Since water saturation is a very important parameter, which is hardly given any thought during coreflood testing, the 'filtrate retention' damage mechanism is turned into a laboratory artifact or an illusion created by poor attention to experimental procedure.

The good news is that the excess filtrate or filtrate/formation water blend residue left in the core by inadequate drawdowns after flooding with formate fluids is **inherently mobile and tractable**, i.e., it is not trapped in place by capillary pressures to cause permanent damage. Simply restoring the core to the same water saturation that it had before exposure to the formate brine/filtrate has recovered full permeability in many coreflood tests. In other cases, where the filtrate is difficult to displace out of the core after being trapped behind a thin filter cake, a mild cleanup with formation water was sufficient to return the core to its initial oil or gas permeability. This mild treatment may have accelerated the breakdown of the biopolymeric filter-cake constituents, which are stabilized by the presence of formate brines, to allow the water/brine to be ejected. In the field, the biopolymeric components of the formate filter cake break down over time – notably faster if exposed to other fluids and gasses. Furthermore, gravity drainage also removes heavy formate filtrate from the near wellbore. By not allowing enough time, for presence of other fluids, or for gravity drainage in coreflood tests, a **significant artifact** is introduced.

Although 'filtrate retention' is not a formate-specific phenomenon, but a mechanism of reduced effective permeability in all water-based fluids, it is more prominent in formate fluids. This is due to their low-solids design, which provides excellent completion performance, such as much-reduced risk of plugging perforation tunnels, sand screens, and other completion hardware. A photo of the very thin filter cake that forms with typical formate drilling fluid is shown in Figure 1.

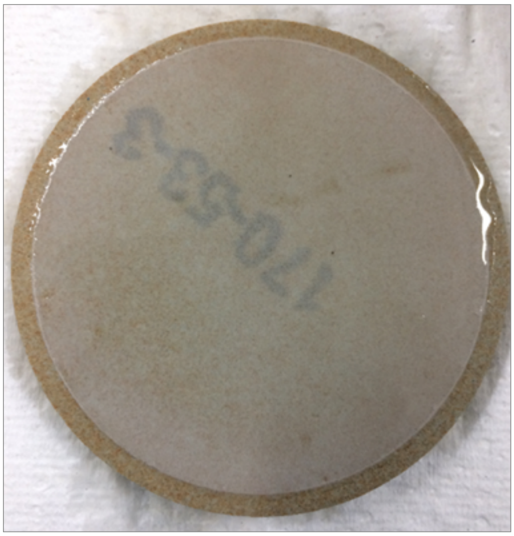


Figure 1 Photo of filter-cake from a typical hot-rolled formate drilling fluid on a 20-μm aloxite disk.

The consequence of using low-solids or solids-free formate fluids in laboratory tests can be a relatively high spurt loss of formate-brine filtrate through filtration media and core plugs until the filter-cake components make an effective seal. The relatively high initial loss of formate brine into the core plug does, of course, impact the water saturation in the core plug. Dependent on the core's initial water saturation level (Sw_i) and the drawdown/cleanup regime, this invasion of an aqueous filtrate can positively or negatively alter the effective permeability to oil or gas, as illustrated in Figure 2.

With a core's effective permeability to oil or gas so dependent on the amount of residual formation water and/or brine left in the core at the time of permeability measurement, the result of coreflood testing of formate brines comes down to the efficiency of the selected drawdown regime in reducing water levels to the initial water saturation level (Sw_i) (see Figure 2). If the final water saturation is higher than Sw_i, a reduced return permeability is reported, and if the final water saturation is lower than Sw_i, then a return permeability >100% is reported.

Indeed, the problem of filtrate retention in coreflood testing with formate brines has sometimes been so severe that formates have mistakenly been disqualified as reservoir drill-in fluids in some prominent HPHT field development projects. An example of this is the first phase of the Martin Linge field development project (Jøntvedt et al. 2018), where cesium formate drill-in fluid was disqualified because it failed to pass the initial coreflood testing, but outperformed other fluids used in the field.

Equinor, the operator with the most experience of using formate drilling and completion fluids, has performed coreflood testing on formate brines and formulated drilling fluids since before its first field use of cesium formate fluids in 2001 (Saasen et al. 2002). It is worth noting a couple of quotes from recent papers by Equinor regarding coreflood testing with formate fluids:

- **SPE-174217-MS – Formate Drilling & Completion Fluids: Evaluation of Potential Well Productivity Impact, Valemon** (Fleming et al. 2015, Fleming et al. 2016): "It should be noted that formate filtrate retention was an issue in lower permeability plugs, although it is believed that much of this liquid would be removed from the near wellbore with continued production". In the test this statement referred to, the coreflood test resulted in a return permeability of only 30%, mainly due to filtrate retention.
- **SPE-199266-MS – Well Productivity Evaluation and Implications for the Significance of Formation Damage: Oseberg Main Case History** (Fleming et al. 2020): For gas reservoirs with temperature >130°C, the following recommendation is given: "If non-formate-based fluids are to be used these will generally have a high solids-loading to achieve the desired density. Aside from having a high focus on stability and compatibility with the completion for such solids-laden fluids, coreflooding should be performed". Although not directly stated, the inference here is that there is no need for coreflood testing if formate fluids are used.

Most operators don't have the same degree of comfort provided by extensive laboratory and field experience as Equinor and therefore demand coreflood testing to qualify formate drilling and/or completion fluids. For these companies, it is important that their coreflood technique differentiates permeability alterations originating from temporary and tractable changes in water saturation, and any permeability reduction resulting from physical plugging of the core plug by particles and polymers. It is also important that the laboratory test method differentiates between mobile filtrate removed gradually through continuous production from the well, or by gravity drainage, and immobile liquid that might be stranded inside the reservoir by capillary forces (irreducible fluid or water-block).

The coreflood test method recommended in this document includes many steps that are helpful when analyzing the mechanism of any return-permeability alteration and helps determine whether any permeability alteration is a result of temporary or tractable water saturation changes or permanent impairment.

The reason these additional steps are required is that realistic drawdown is not feasible in the laboratory, which can have a huge impact on the final water saturation.

For a full review of potential damage mechanisms, reservoir water compatibility, and field experience with formate fluids, please see the Sinomine Formate Technical Manual, Section B10 Compatibility with the Reservoir.

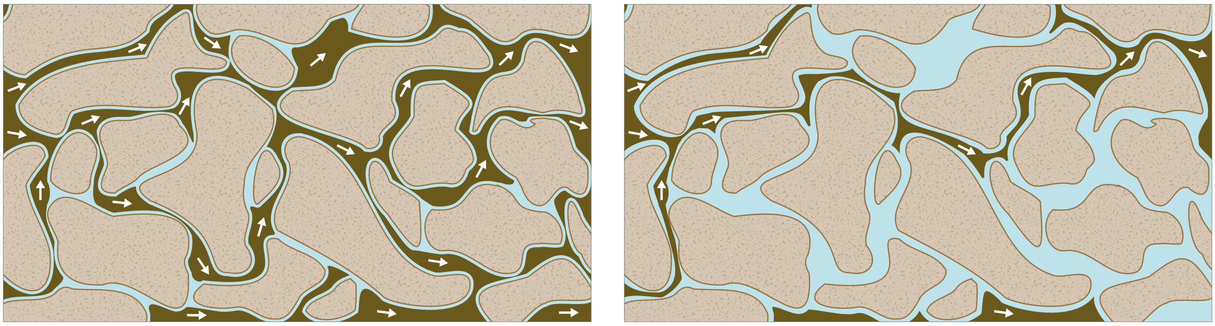


Figure 2 Left: Illustration of a core plug with low initial water saturation, Sw_i, resulting in high effective permeability to hydrocarbons. Right: Illustration of a core plug where the water saturation is significantly higher after cleanup/drawdown than it was before, resulting in a much lower effective permeability to hydrocarbons.

2 Preparation of core and reservoir fluids

2.1 Prepare core material

Cores obtained from the field are saturated with the drilling mud used in the coring operation. If an oil-based mud was used for coring then the external and internal surfaces of the cores will be coated in oil-wetting surfactants, emulsifiers, oil-wet/soluble polymers (viscosifiers), oil-wet fine barite and drilled solids, and emulsified calcium chloride brine. Plugs drilled out from these cores need lengthy Soxhlet-submerged extraction with solvents under mild conditions to remove these alien contaminants and restore the core surfaces to their native condition (Francis et al. 1995; Byrne et al. 2000; Downs 2009).

After drying the cleaned plugs at 60°C (140°F) in an oven, any fine rock flour or micronized rock particles on the plug surfaces need to be removed. The acetate peel technique (Byrne et al. 2000) used to remove rock flour (fines) from the plug's end faces must be done very carefully so that residues from the peel are not introduced into the pore throats. Normally, the plugs are then CT-scanned to visualize internal laminations, bedding planes, and inclusions in the plugs. Finally, the porosity, air permeability, and grain density of the plugs are measured.

When testing formates and other water-based fluids it is extremely important that any water-wet rock that may have been made oil-wet from the coring fluid is restored to its water-wet condition.

2.2 Prepare synthetic formation water

Synthetic formation water is normally formulated to match the composition and pH of mobile water sampled from a water-bearing formation in or below the net reservoir. It is assumed that compatibility between the formate brine and the formation water has been tested and any incompatibility with added carbonate buffer has been considered and potentially eliminated. In practice, the test laboratories often eliminate 'problem' elements like *Fe* and *S* compounds from their synthetic formation water recipes. The pH of formation waters in gas-bearing reservoirs is dictated by their natural carbonate/bicarbonate buffering and the concentration of acid gases in the gas phase.

2.3 Prepare realistic hydrocarbon phases

If the test is intended to determine the potential damage of a formate fluid to a gas-bearing reservoir, a gas composition matching the reservoir should be used. **It should be saturated with water at test temperature and test pressure, and it should enter the core plug at test temperature.** Failure to do so can cause significant laboratory artifacts related to dehydration of the formate brine (see section 6.2) and cooling of the core during drawdown. Use nitrogen gas if the test laboratory is unable to handle hydrocarbon gases. If realistic amounts of CO_2 and H_2S are not added to the test gas, ensure that this does not impact the test results (see section 6.7).

If the test is for an oil reservoir, the test laboratories normally substitute the live hydrocarbon with a 'dead' high-flash-point mineral oil with viscosity similar to the live hydrocarbon under reservoir conditions. Any emulsification tendencies identified between the laboratory oil and the formate filtrate is a laboratory artifact that needs to be addressed before testing proceeds. If any emulsification problems are identified between formate filtrate and real crude oil, which is unlikely, these need to be addressed separately, possibly with demulsifying additives.

2.4 Prepare core sample to initial formation water saturation (Sw_i)

The initial water saturation in the core plug, Sw_i, is probably the single most important factor for a successful coreflood test result with formate fluids. Despite this, it is often not measured, reported, or considered, and rarely set to match that of the reservoir. This is a major oversight, considering the impact this single factor has on the outcome of coreflood tests of water-based fluids, and particularly comparisons of formate fluids with oil-based muds. Consequently, by leaving it to the test laboratories to decide how Sw_i is established, they are given incredible power to determine the outcome of the tests.

NOTE. When comparing formate fluids with oil-based muds, test laboratories through their method of establishing Sw_i, are given incredible power to determine the outcome of important fluid-qualification tests.

To further exacerbate the situation, the coreflood test laboratories are not even in agreement with each other on the best method to prepare the core plug to initial water saturation. Two common methods are:

- Centrifuging with an ultra-centrifuge at ambient pressure and low temperature.
- Flooding with hydrocarbon – presumably at a drawdown pressure that matches the final drawdown after fluid exposure, which at this time is often an unknown.

In Sinomine's experience, any details of this step are rarely reported. Both methods create problematic laboratory artifacts, including uneven saturation distribution and end effects, which strongly impact the results of coreflood testing with water-based fluids (van der Zwaag et al. 2010), and specifically formate fluids due to their much higher filtrate loss.

Note also that the parameters used for centrifuging (capillary pressure, temperature) and flushing (flow rate/pressure-differential/temperature), which impact the initial water saturation, are often set without considering the actual water saturation of the reservoir. This is in complete contradiction to guidelines given by the group that first tried to standardize the coreflood test method (Marshall et al. 1997): "The plugs can be prepared to an initial water saturation based on reservoir water saturation values by one of the following methods…". In fact, Sinomine's experience is that most test laboratories strive to set the initial water saturation as low as possible and refer to it as 'irreducible water saturation' (Sw_{ir}). It is, however, doubtful if their 'irreducible water saturation' achieved by their selected centrifuge capillary pressure or flooding pressure, often in an outcrop core, at all resembles the actual reservoir's irreducible water saturation.

If centrifuging is used to establish Sw_i, this should be done at a temperature as high as practically possible. This is because when centrifuging again at the end of the test to reestablish the same capillary pressures, biopolymers that have not been given enough time to break down make it difficult for the filtrate to escape the core, which introduces a significant artifact.

It is beyond the scope of this document to evaluate the methods of establishing Sw_i. Whatever method the test laboratory uses or whether the selected initial water saturation represents that of the real reservoir or not, it is important that the same method is used again during drawdown/cleanup to reestablish equivalent capillary pressure conditions before measuring final return permeability (see section 5.5). Unfortunately, this important step is missed by most coreflood testing laboratories.

3 Evaluation of required cleanup time

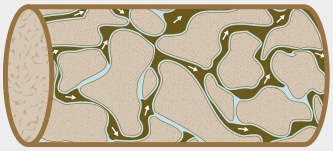
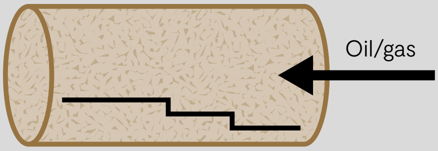
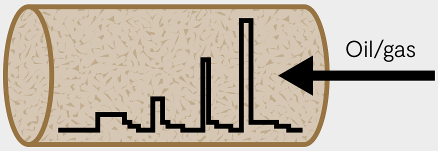
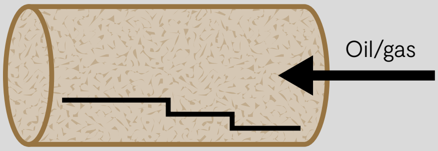
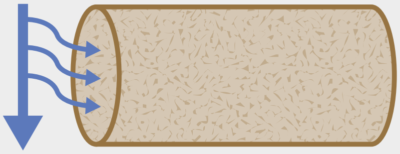
In the field, biopolymers in the drilling fluid enter the reservoir during drilling and are not only present in the internal and external filter cakes, but also to some extent in the filtrate. Biopolymers, although stabilized at high temperatures in formate brines, are self-breaking, at least down to 124°C (255°F) when hydrated in formate brines (Anderson et al. 2023). In the field, these biopolymers are given plenty of time to thermally degrade during drilling, logging, completion, and suspension periods, and even during well cleanup and early production. Unfortunately, in standard coreflood testing, all drilling fluid constituents are expected to break down and clean up during a few hours of exposure to reservoir temperature. This is a serious artifact in many coreflood tests and is discussed in detail in section 6.5. To perform a realistic coreflood test on any fluid containing self-breaking biopolymers, the test must apply a realistic exposure time to reservoir temperature.

4 Coreflood test procedure

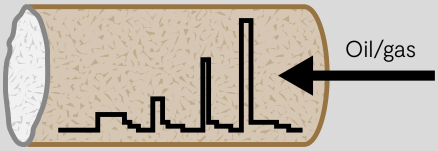
Sinomine's recommended test procedure for formate fluids is shown in Table 1. It contains some added drawdown and permeability measurement steps, which are very important for determining whether permeability changes are caused by tractable water saturation alterations or 'real' damage mechanisms.

These steps, which may appear unnecessary, can actually provide a lot more useful information than the various imaging methods used. Consequently, it may be a matter of reallocating resources rather than adding more cost to the test. Also, if at any point in the procedure, the permeability has been restored to an acceptable level, there is no need to continue. All steps apart from the centrifuging should be performed under reservoir conditions.

Table 1 Sinomine recommended coreflood test procedure for formate fluids. All steps apart from the centrifuging should be performed under reservoir conditions. If gas is used, it should be humidified at test temperature and test pressure. All permeability measurements are to be conducted at test temperature.

Step	Procedure
1. Establish initial water saturation 	• Establish initial water saturation (Sw_i) by either flooding or ultra-centrifuge. • Measure and record Sw_i.
2. Determine permeability  	• Measure permeability to hydrocarbon at three low flow rates.
3. Fingerprint the core  	• Carry out the full drawdown sequence, as in step 6, to hydrocarbon. • Ideally measure permeability to hydrocarbon at three low rates between each drawdown. Significant permeability reductions observed at any point in this first drawdown sequence signal a major experimental problem, e.g., fines migration or cooling of the core at high flow rates, and the test should be terminated or redesigned.
4. Determine permeability  	• Measure permeability to hydrocarbon at three low flow rates. This is the initial reference permeability used to calculate percent change in return permeability after exposure to drilling fluid. An initial permeability deviating from the permeability measured in step 2 and any permeabilities measured in step 3 are cause for concern and must be flagged, and the test potentially terminated.
5. Test-fluid exposure 	• Expose to drilling or completion test fluid by circulating the fluid across the wellbore face of the core under pressure conditions and for a time period that reflect real wellbore conditions.

6. Drawdown to hydrocarbon

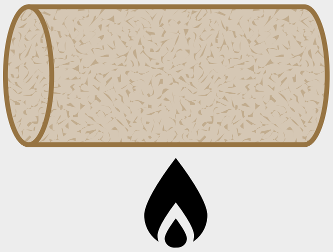


- Increase drawdown in stages.
- Keep flooding at each stage until permeability stabilizes.
- Stop flooding if permeability decreases. If fines migration was observed during fingerprinting in step 3, then it is also expected to occur after test-fluid exposure. Decreasing permeability during drawdown can also be an indication of unbroken biopolymers.
- Measure permeability at three low flow rates between each drawdown stage.
- If possible, measure and record volume filtrate/water ejected during each stage.

The permeability measured after the final drawdown can be used to calculate return permeability that includes effective permeability reduction from filtrate retention.

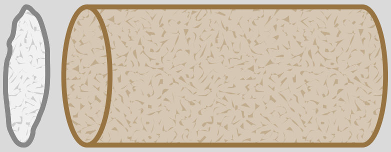
If the measured permeability is unacceptable and it is known that any biopolymers present have not been given enough time to break down by themselves, go to step 7. Otherwise go to step 8.

7. Shut in



- Shut in under realistic pressure and temperature conditions.
- Return to step 6.
- Repeat this as long as the measured permeability increases.
- If excessive time is needed, the temperature can be ramped up during the shut-in period to accelerate the biopolymer self-breaking rate.

8. Remove filter cake

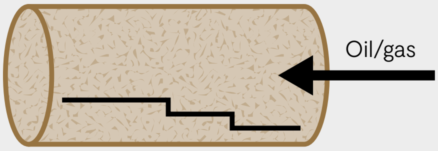


- If final permeability measured in step 6 is lower than reference permeability, then remove external filter cake, if there is any left.

Note: The filter cake should not be removed mechanically, but by flushing with warm base brine as described in section 4.1.

It is rare to see any significant external filter-cake residues left on the plug face after drawdown following exposure to formate fluids. Signs of filter cake or existence of slimy material in the core holder is a clear sign that the cleanup was terminated before all self-breaking biopolymers were given the time to degrade.

9. Determine permeability

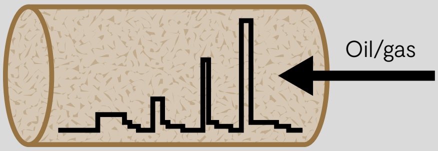


- Measure permeability to hydrocarbon at three low flow rates.

Any permeability increase gained by removing the external filter cake represents the permeability loss in the actual filter cake.

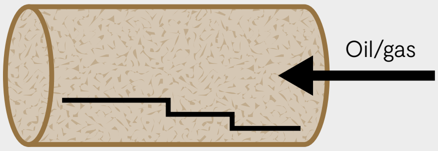
Normally an insignificant permeability increase is measured here.

10. Drawdown to hydrocarbon



- Carry out another drawdown sequence as shown in step 6.
- Ideally measure permeability to hydrocarbon at three low rates between each drawdown.

11. Determine permeability

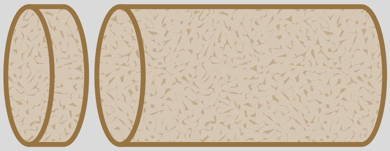


- Measure permeability to hydrocarbon at three low flow rates.

Any permeability increase observed in this step relates to removal of some further volume of retained filtrate mobilized by removing the small amount of filter cake left on the core face.

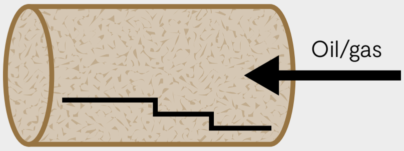
Any permeability increase created during this other drawdown is often significantly higher than the increase created by removing the filter cake alone.

12. Trim core



- If final permeability measured in step 11 is lower than reference permeability, then trim core, typically ~¼".

13. Determine permeability

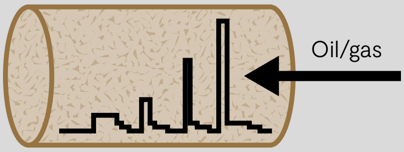


- Measure permeability to hydrocarbon at three low flow rates.

Any permeability increase gained by trimming the end of the core represents the permeability lost through the internal filter cake itself.

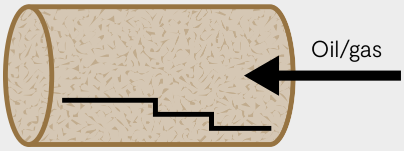
Normally a small permeability increase is measured here.

14. Drawdown to hydrocarbon



- Carry out another drawdown sequence as shown in step 6.
- Ideally measure permeability to hydrocarbon at three low rates between each drawdown.

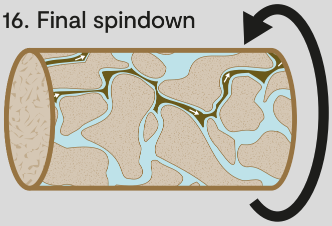
15. Determine permeability



- Measure permeability to hydrocarbon at three low flow rates.

Any permeability increase observed in this step is related to the removal of some further volume of retained filtrate mobilized by removing the internal filter cake. This contribution to permeability increase can be significantly higher than the increase created by removing the internal filter cake alone.

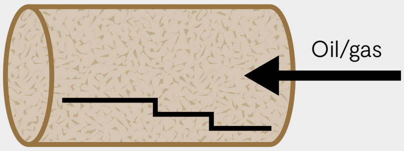
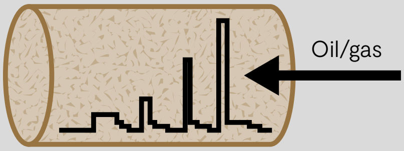
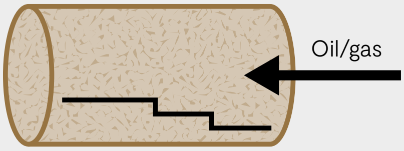
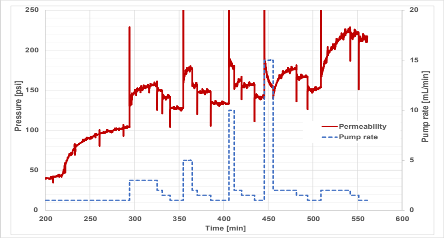
16. Final spindown



- If there was a spindown at start, then spin down at end. This is only needed if permeability measured in step 15 is smaller than the reference permeability.
- Measure and record volume filtrate/water ejected during centrifugation.

By spinning down at the same capillary pressure and for the same length of time used when establishing Sw_i, the permeability can be compared after exposure to the same capillary pressure, and any damage in the core, not related to water saturation or removed filter cake, can be identified.

Please note that if biopolymers that have not been given enough time to break down are present, spinning the core – especially at a lower temperature – may not remove a realistic amount of filtrate, and the test can still suffer from a 'filtrate retention artifact'.

17. Determine permeability  	• Measure permeability to hydrocarbon at three low flow rates. Any permeability increase created by the final spindown represents the amount of permeability impairment caused by the last remnants of mobile filtrate that would not come out at the highest drawdown pressure/flow rate, which in a real reservoir may come out with gravity over time.
18. Drawdown to hydrocarbon  	• Carry out a new drawdown sequence as shown in step 6. • Ideally measure permeability to hydrocarbon at three flow rates between each drawdown.
19. Determine permeability  	• Measure permeability to hydrocarbon at low flow rates. This permeability may deviate from the one measured in step 17, due to redistribution of water and filtrate inside of the core.
20. Perform thorough permeability analyses  	• Study pressure vs. flow rate and permeability vs. time plots for all steps. • Make sure they are 'seamless', with no sudden or unexpected changes, and make sense. • The permeability during the final stages of a drawdown should, within reason, be the same as the permeability measured at low flow rates a few minutes later. • Make sure all pressures and flow rates are measured and reported. • Look specifically for signs of unbroken biopolymers. This artifact may be mistaken for fines migration.

4.1 Method for removing external filter cake

The standard method for removing external filter cake is to carefully remove it mechanically with a spatula or similar tool. This may be suitable for thick filter cakes loaded with barite, but for a typical formate filter cake, such treatment can result in severe permeability reduction as unbroken biopolymers are pushed into the core. Sinomine has therefore developed a more suitable method for removing external filter cake from formate fluids, which is illustrated in Figure 3 and described below.

- Install the core or ceramic disk in a clamp.
- Squirt a jet of heated base brine at 80°C (176°F), or as hot as is considered safe, onto the filter-cake surface from a plastic bottle.

Figure 3 Method for removing external filter cake from formate fluids.

5 Examples of test result interpretation

The detailed procedure shown above may, at first glance, seem to have many unnecessary steps. Therefore, the importance of these steps is explained through examples.

5.1 Importance of step 1 – establish initial water saturation (Sw_i)

It is not the intention of this document to recommend a method to establish the initial water saturation, Sw_i, as there is no correct method. However, considering the importance of this step in determining the outcome of the coreflood test, where water saturation largely determines permeability, two examples are given that demonstrate its effect on test results.

Example 1: Coreflood testing was conducted to qualify cesium/potassium formate drill-in fluid for an upcoming field development. Two sets of tests were performed by the same laboratory. In the first set of tests, the Sw_i was established by flooding with carnation oil. In the second set of tests, the Sw_i was established with an ultracentrifuge, set to give a capillary pressure of 125 psi. The test temperature was 138°C (280°F) and carnation oil was, in both cases, used as crude oil replacement. There were some other differences between the two tests, but Sinomine considers the establishment of Sw_i the most significant. The results of the tests, reported after step 6 in the Sinomine-recommended procedure detailed in section 4, are shown in Table 2 and Table 3.

Table 2 Results of coreflood testing with three different core plugs. The initial water saturation, Sw_i, was set by flooding the core with carnation oil. In the core plug used in test 3, extra pressure was applied during flooding to reduce Sw_i, and a pressure peak as high as 2,745 psi was recorded.

Test #	Sw_i	Perm. at Sw_i [mD]	Max. pressure to establish Sw_i [psi]	Recovered permeability after constant pressure drawdown			
				10 psi	25 psi	50 psi	100 psi
1	45%	12	–	16%	44%	–	95%
2	30%	181	114	108%	154%	–	–
3	24%	16	2,745	5%	43%	77%	96%

Table 3 Results of coreflood testing with three different core plugs. The initial water saturation, Sw_i, was set with an ultra-centrifuge, giving an equivalent capillary pressure of 125 psi.

Test #	Sw_i	Perm. at Sw_i [mD]	Recovered permeability after constant flow-rate drawdown					Max. pressure during drawdown [psi]
			5 mL/min	10 mL/min	20 mL/min	30 mL/min	50 mL/min	
1	13%	166	69%	70%	73%	82%	84%	65
2	20%	46	30%	41%	54%	62%	61%	120
3	33%	7.9	32%	52%	67%	–	–	550

As can be seen in Table 2, all three tests in cores flushed with oil to establish Sw_i resulted in very high return permeabilities, indicated in green. Test 2 gave a return permeability of 108% just by applying a 10-psi pressure drawdown and 154% after a 25-psi drawdown. Somehow the 10-psi pressure applied during cleanup must have removed more water/brine from the core plug than was removed by the 114-psi differential pressure applied to establish the Sw_i. In this test, it appears that formate brine, or formate brine/formation water blend, is easier to remove than the formation water itself.

In the second set of experiments (Table 3), the initial permeability could not be recovered in two of the tests. Return permeabilities marked with red are typically not accepted by many operators. It was later shown that the permeability reduction in these 'red tests' was caused by mobile filtrate retained in the core. Obviously in this test, the water saturation after the test was higher than the initial water saturation, Sw_i. It appears that the centrifuge removed more water/brine than the highest drawdown used.

This is just one example where a core preparation procedure, i.e., establishing Sw_i, which is rarely given much thought by the operator or the test laboratory, can be the determining factor for the outcome of an important fluid-qualification exercise with a formate drilling fluid.

It should be noted that biopolymer self-breaking times were not considered when these tests were performed, so step 7 was omitted. From measured biopolymer self-breaking times, shown in section 6.5, it is clear that the biopolymers were not given enough time to break down during these tests. Unbroken biopolymers in the filter cake and filtrate hinder the removal of filtrate, both during flooding and centrifuging.

Example 2: A coreflood test was conducted to qualify a cesium/potassium formate drill-in fluid for an application in an oil reservoir. The testing was performed at a laboratory that uses centrifuging to establish Sw_i. No information was shared about the centrifuge capillary pressure used or the actual initial water saturation of the core. It was referred to as 'irreducible'. For this application, the operating company requested that drawdown should be performed at constant rates, with a maximum rate of 4 mL/min. As the formation water was removed with an ultra-centrifuge at an unknown rate, aiming at an 'irreducible' level, and the formate brine removed by a pump rate of only 4 mL/min through an internal filter cake with biopolymers that had not been given time to degrade, it was a test set up to fail.

To better understand the impact of Sw_i on the test result, the laboratory was asked to apply both methods of establishing Sw_i, so the resulting effective permeabilities could be compared. The following two methods were applied:

- Establish Sw_i by **flooding** the core, which had been fully saturated with formation water, using mineral oil at the highest drawdown rate applied during cleanup, i.e., 4 mL/min. The effective permeability to mineral oil at Sw_i was measured to **29.7 mD**.
- Establish Sw_i by **centrifuging** the core, which was fully saturated with formation water, at the standard (unknown) capillary pressure used by this specific laboratory in its attempt to achieve 'irreducible water saturation'. The effective permeability to mineral oil at Sw_i was measured to **37.9 mD**.

After step 6 of the Sinomine-recommended coreflood procedure (see section 4), conducted at 110°C (230°F), the permeability was measured to 29.9 mD, which corresponds to 79% return permeability when compared with the Sw_i established by the ultra-centrifuge. Compared with the Sw_i established by flooding, the permeability had fully recovered. It is unsurprising to learn, after performing the remaining steps, that most of this permeability loss was caused by the formate brine/formation water blend that had not come out of the core through the filter cake during the short and unrealistic drawdown.

This example demonstrates how two different methods commonly accepted in the industry produce Sw_i values so different that the initial reference permeability varies by almost 30%.

5.2 Importance of step 3 – fingerprint the core

The importance of this step was already discussed by the group that first tried to standardize coreflood testing in 1997 (Marshall et al. 1997): “To prevent potential fines mobilization in the sample, a critical velocity test may be required to define the maximum flow rates permissible without sample damage”. It is rare to see this done today, and if it is, it is typically not reported to clients. The importance of this step will also be demonstrated by examples.

Coreflood testing was performed to qualify cesium/potassium formate drill-in fluid for an upcoming field development. The test temperature was 138°C (280°F) and carnation oil was used as crude oil replacement. Drawdown/cleanup after exposure to drilling fluid was planned at five flow rates of 5, 10, 20, 30, and 50 mL/min. The same drawdown was conducted on the core already prepared to Sw_i with carnation oil by centrifuging at 125 psi capillary pressure before exposure to drilling fluid. The result of this ‘fingerprint’ testing is shown in Figure 4. A permeability to oil of 50 mD was measured in the core at low flow rate after Sw_i was established by centrifuging. During fingerprinting of this core containing formation water at Sw_i, a significant pressure buildup could be seen at the two highest flow rates, indicating a decrease in the core plug’s permeability to oil. When the permeability was measured again at low flow rate, it had dropped to 46 mD. This means that the planned cleanup regime itself was responsible for an 8% reduction in permeability, even before the core plug had been exposed to the drilling fluid. A similar test was completed under the same conditions, but with a different core type. See Figure 5. In this test, pressure buildup could also be seen during high-rate flow, but the initial permeability recovered after drawdown/cleanup.

5.3 Importance of step 7 – shut in under realistic pressure and temperature conditions

The importance of this step was demonstrated in a long-term coreflood test, during which a solids-free potassium formate drilling fluid, containing only xanthan gum and modified starch additives, was used. This represents a standard formate drilling fluid, with the calcium carbonate bridging package removed. The coreflood test simulated filtrate invasion into a water-saturated formation, while drilling an injection well. In this scenario, the core plug was initially 100% saturated with formation water, and return permeability was measured by injecting formation water through the core in the same direction as the drilling fluid was injected. The testing was initially conducted at 121°C (250°F).

Before exposure to the potassium formate drilling fluid and a clear-brine potassium formate completion fluid, a permeability of 212 mD was measured. After an initial 49-hour cleanup with formation water, permeability to formation water of 8.0 mD was measured, corresponding to a return permeability of only 3.8%. Since there was nothing in this fluid that could cause any formation damage, other than hydrated biopolymers, and it is known that biopolymers need a long time to thermally degrade at this relatively low temperature, it was decided to perform multiple shut-ins at the test temperature. The core was left in the test rig at test temperature and the confinement pressure was maintained during the test. A total of six shut-ins were performed during a period of 17 days. The following observations were made:

- Permeability increased after each shut-in period.
- Permeability decreased significantly during each injection period.

After the sixth shut-in period and 17 days of exposure to 124°C (250°F), the permeability had increased steadily to 24 mD, corresponding to a return permeability of 11.3%. It was clear that although formation water was injected into the core, the biopolymers were still protected by the potassium formate brine. By studying the self-breaking rate of xanthan and modified starch at various temperatures, see section 6.5, it was concluded that it would be too costly to continue coreflood testing at this temperature. Consequently, temperature was ramped up during the shut-in periods to accelerate thermal degradation of the biopolymers. Four week-long shut-ins were applied at 149°C (300°F). However, the core was cooled down to 121°C (250°F) and allowed to stabilize for a while at this temperature before formation water was injected and permeability measured. After a total of 41 days, the permeability had increased to 208 mD, corresponding to a return permeability of 98.2%.

Coreflood fingerprint test with permanent permeability loss

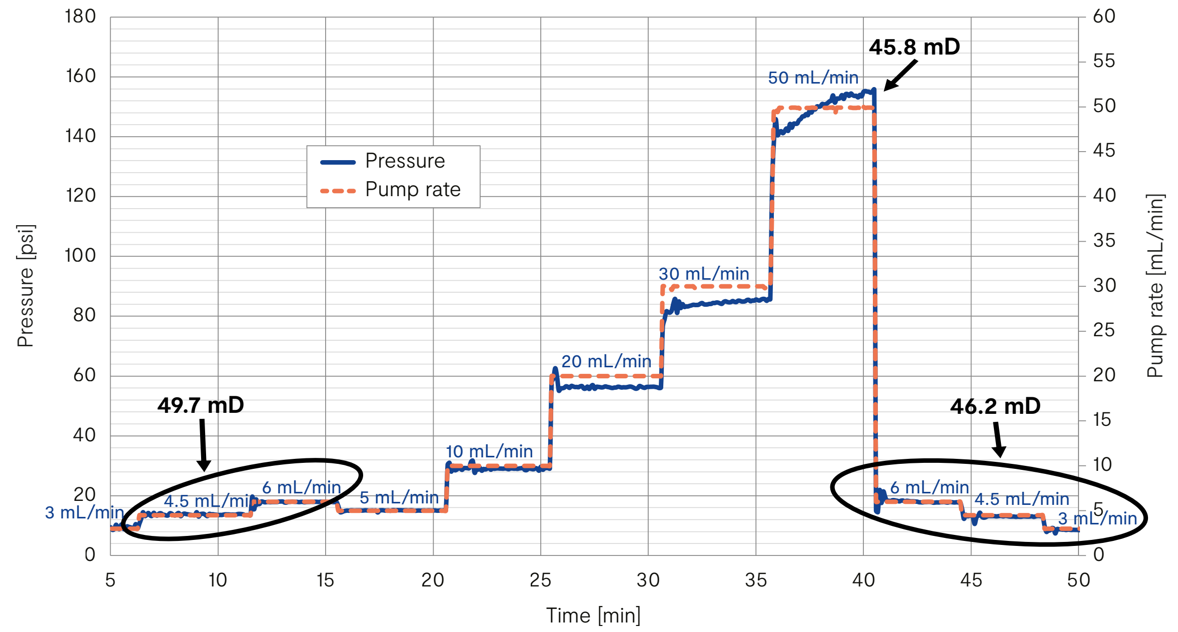


Figure 4 Pressure and flow rate as functions of time in a drawdown sequence performed before exposure to drilling fluid. Significant pressure buildup was recorded at the two highest flow rates, indicating that core-plug permeability had decreased. When measuring permeability again at low flow rates, permeability did not recover.

Coreflood fingerprint test with temporary permeability loss

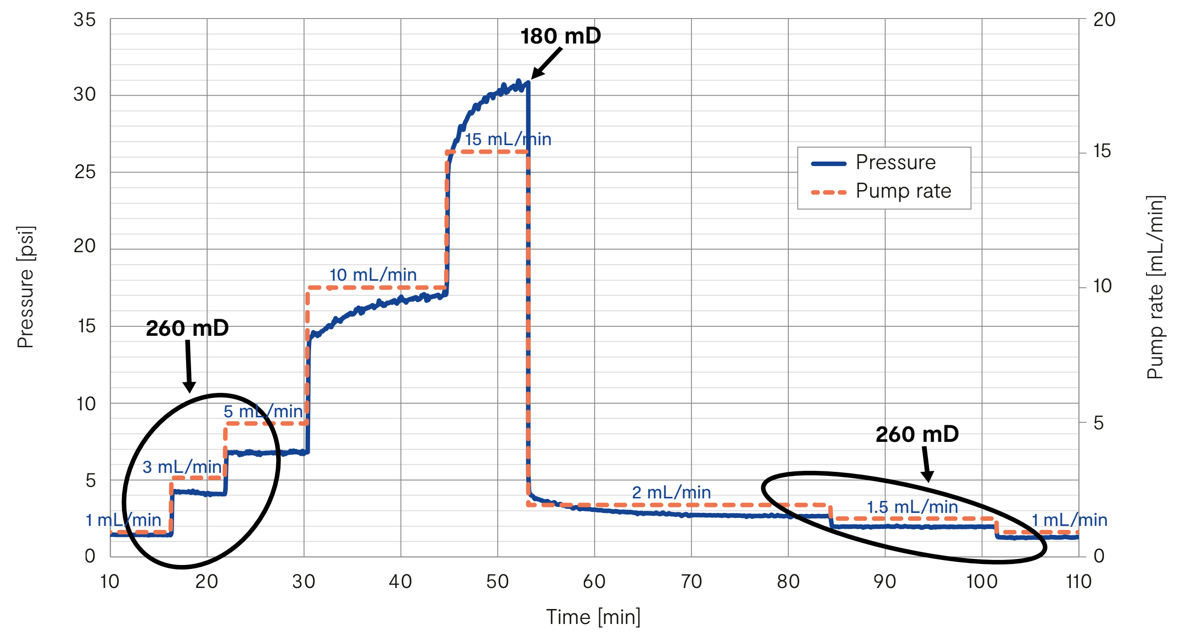


Figure 5 Pressure and flow rate as functions of time in a drawdown sequence performed before exposure to drilling fluid. Significant pressure buildup was recorded at the two highest flow rates, indicating a decrease in permeability. However, full recovery of permeability was obtained when measuring at low rate after the high-rate drawdowns.

Apart from the last injectivity periods when the core was fully cleaned up, decreasing permeability was measured during each injectivity period throughout the whole study – see example from the fifth injectivity test in Figure 6. It is believed that this happens because of redistribution of biopolymers that have still not broken down. Figure 7 shows the permeability recovery after each injection step as a function of temperature-exposure time. A full description of this test has been published in SPE-217909-MS (Howard 2024).

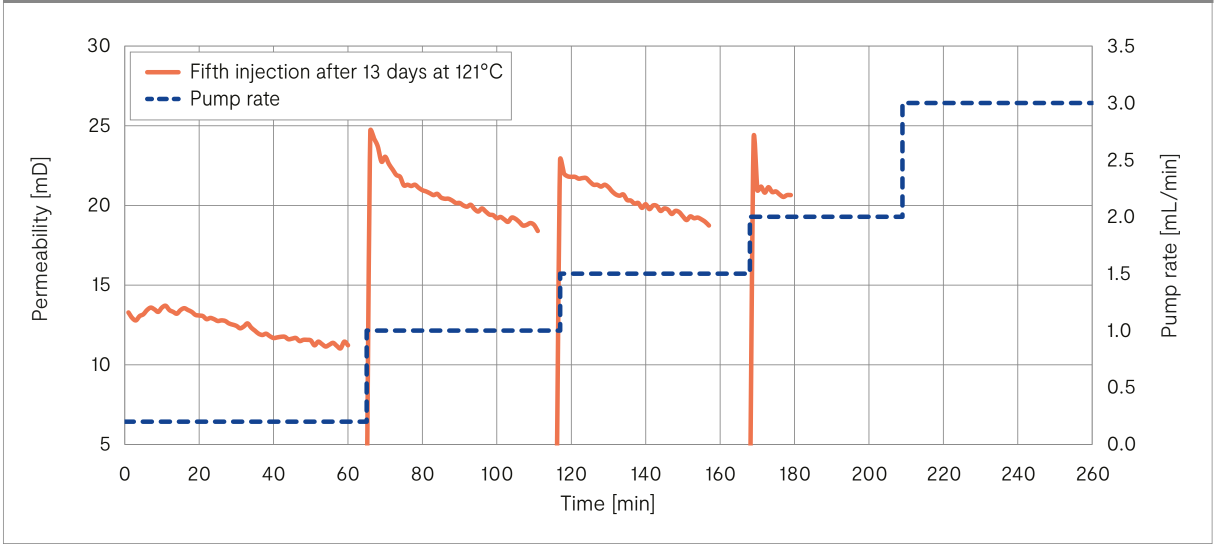

Permeability development over time

Figure 6 Permeability development during the fifth injectivity test, performed at 0.20, 1.0, 1.5, and 2.0 mL/min pump rates after 2-, 1-, and 2-day shut-ins at 121°C (250°F).

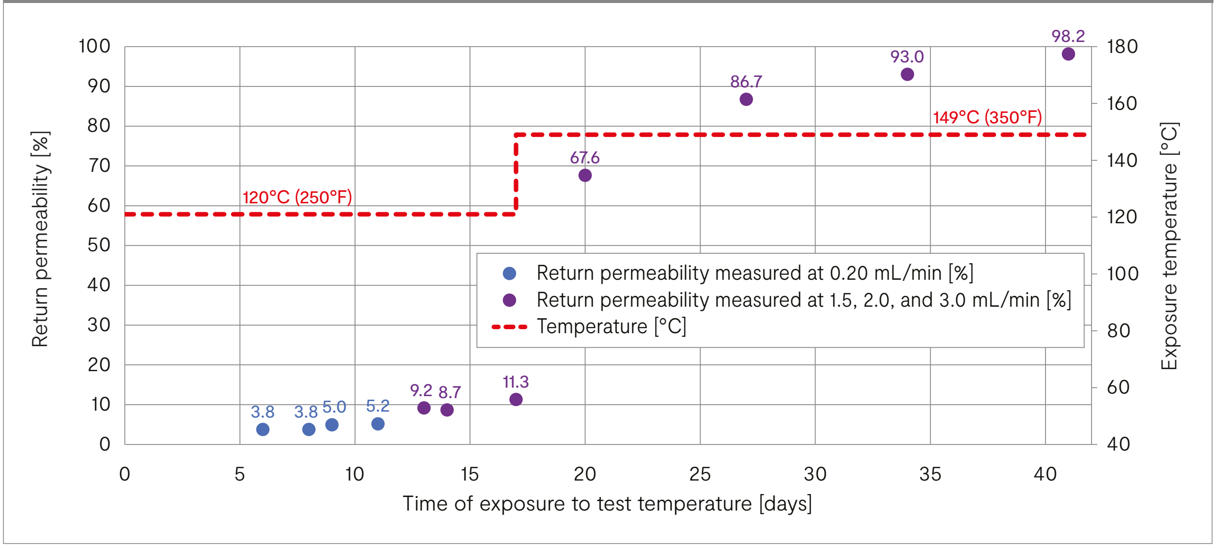

Effect of exposure time on return permeability

Figure 7 Permeability measured during injection testing after four shut-in periods at 149°C (300°F). The permeabilities are measured at 121°C (250°F) with four pump rates: 0.20 mL/min, 1.5 mL/min, 2.0 mL/min, and 3.0 mL/min.

5.4 Importance of steps 12, 13, 14, and 15 – trim core, determine permeability, drawdown, and permeability

The importance of these steps is not about determining how a wellbore without a filter cake performs in the coreflood test, but rather about learning what causes the permeability reduction in step 6, through additional drawdowns and permeability measurements. Two examples are provided that demonstrate the importance of not skipping any steps when the core plug is trimmed. The same applies to steps 8, 9, 10, and 11, when the external filter cake is removed.

Example 1: This example is from test 1 in Table 3. After step 6 in the Sinomine-recommended coreflood test procedure, **140 mD (84%)** of the permeability had been regained. Although this is a relatively good result, it is interesting to understand what caused the 16% permeability reduction. There was no external filter cake to be removed, so steps 8, 9, 10, and 11 were omitted. The core was therefore trimmed straight away and the following steps were performed:

13. Measure permeability at low flow rate. A permeability of **145 mD (87%)** was measured. As this represented an increase of only 3%, it was concluded that the ¼" trim (the internal filter cake) was only responsible for a very small part of the lost permeability.
14. Perform drawdown at the same pressures/rates as shown in step 6.
15. Measure permeability at low flow rate. The permeability had now increased to **168 mD (101%)**.

In this test, it could be concluded that the 16% permeability loss reported in step 6 was caused by filtrate retention. The filtrate was shown to be inherently mobile, i.e., it was not irreducible, capillary trapped or stranded, but did not flow out initially due to an internal filter cake blocking its only flow path out of the core holder.

Example 2: This example is from test 2 in Table 3. After step 6 in the Sinomine-recommended coreflood test procedure, **28.3 mD (68%)** of the permeability had been regained. An investigation was initiated to find out what caused this permeability decrease. There was no external filter cake, so steps 8, 9, 10, and 11 were omitted. The core was therefore trimmed straight away and the following steps were performed:

13. Measure permeability at low flow rate. A permeability of **47.2 mD (102%)** was measured. Since the main core had fully recovered its permeability, it was concluded that the entire permeability loss was in the trim.
14. Perform drawdown at the same pressures/rates as shown in step 6. This step was performed to ensure that the conclusion from step 13 was correct.
15. Measure permeability at low flow rate. The permeability had only increased to **47.7 mD (103%)**, which again confirmed that all permeability decrease was indeed in the trim, and the conclusion from step 12 was correct.

The result from example 2 was interesting. Normally, in such cases, it is concluded that the permeability decrease is caused by tractable filtrate retention in the main core, but in test 2, all of the permeability impairment was concentrated in a ¼" trim. Therefore, as it was a concern that there would be some 'real' damage, e.g., unrecoverable permeability loss in the trim, it was decided to investigate further by measuring the permeability and performing drawdowns on the trim to see if full permeability could be recovered. During the preliminary permeability measurement performed at low flow rates, 2.30 mL filtrate was ejected from the trim and permeability of **47.5 mD (103%)** was measured without performing any high-rate drawdown. In this test, it could be concluded that the average 32% permeability loss across the entire core seen after step 6 was caused by tractable/mobile filtrate concentrated in the small volume of core adjacent to the wellbore face. The filtrate was mobile and had moved all the way out to the wellbore face of the core, where flow was restricted by the internal filter cake, which still contained some unbroken biopolymers.

From example 1, it is evident that the internal filter cake itself, although not very permeable to water/filtrate, is not impermeable to oil as very little oil permeability was gained by removing it. The fact is that the internal filter cake prevents mobile filtrate to flow out of the wellbore end of the core, which again because of its presence, reduces the effective permeability to oil. Obviously by not allowing enough time for the biopolymeric components of the internal filter cake to break down naturally (see section 6.5) or allowing the filtrate to drain with gravity in another direction (see section 6.4), an unavoidable laboratory artifact has been created at the wellbore end of the core. The same laboratory artifact is seen in example 2, but here all of the mobile filtrate has accumulated in the very outer part of the core, indicating that the internal filter cake was very shallow.

5.5 Importance of step 16 – final spindown

The huge impact initial water saturation, Sw_i, has on the outcome of the coreflood test with formate fluids and other brine-based fluids cannot be stressed enough. A study performed by van der Zwaag et al. (2010), where coreflood procedures and test results from many laboratories were compared, concluded that the strongest impact on return permeability measurements was seen when comparing tests with samples prepared by centrifuging versus those prepared by flooding with hydrocarbons under reservoir conditions. The method delivering the highest return permeability varied according to the core permeability and the type of core material. Numerical simulations confirmed the experimental findings; high-permeability samples (Bentheimer sandstone) starting out with low initial water saturations created by centrifuging, showed lower permeability recovery after mud exposure and drawdown compared to samples with higher initial water saturations, typically created by flooding with hydrocarbons. The main reason for this is that the low water saturation obtained by the high capillary pressure produced in the centrifuge was not reestablished during drawdown after mud exposure. Based on his findings, van der Zwaag provides the following recommendation:

> **To distinguish 'true' water-block-type formation damage from laboratory artifacts caused by differing capillary pressure conditions before and after mud application, reestablish equivalent capillary pressure conditions wherever possible before measuring return permeability. Practically this implies removing the core plug from the core holder, spinning it again in a centrifuge under the same conditions as before the mud exposure experiment, remounting, reestablishing pressure and temperature conditions, and measuring another 'spindown' permeability.**

Sinomine highly recommends following this expert guidance from van der Zwaag. If all mobile filtrate hasn't been removed from the core plug after step 16 in the Sinomine-recommended test procedure and if Sw_i was established by centrifuging, the core plug should be centrifuged again under the same conditions before final permeability is measured. Failure to do so can result in formate fluids being mistakenly disqualified from important fluid-qualification exercises.

Since unbroken biopolymers may still be present in the formate filtrate, all centrifuging should be conducted at the highest possible temperature.

6 Coreflood testing of formate fluids – examples of laboratory artifacts and how to avoid them

Methodically following the steps detailed in the test method described above allows identification of the location and dimensions of tractable permeability reduction resulting from pockets of aqueous fluids left in cores by inadequate drawdowns and/or temporary flow barriers created in segments of the cores.

If, after performing all steps described in Table 1, there is still doubt that any permeability reduction experienced after step 6 in the Sinomine-recommended coreflood procedure is caused by retained mobile filtrate, then there are many potential laboratory artifacts that should be addressed and/or eliminated before drawing final conclusions. Apart from the impossible task of establishing the correct initial water saturation, Sw_i, these artifacts largely originate from unrealistic drawdown. As mentioned, Sinomine defines a 'realistic drawdown' as:

- Pressure differential to match the real well.
- Oil or gas velocity or flux (cm^3/cm^2) to match the real well.
- Time – long enough for thermally unstable drilling fluid constituents to break down and heavy brine to drain with gravity.
- Realistic configuration – eliminate effect of core holder walls and presence of the core plug reservoir end, which cause laboratory artifacts.
- Flowing phases contain realistic amounts of other mobile reservoir components, such as acid gasses present during production startup.

These laboratory artifacts are described in section 6.1 to section 6.7.

6.1 Artifact – use of incorrect initial water saturation (Sw_i)

To claim that reduction in effective permeability to hydrocarbon caused by increased water saturation of the core is a real formation damage effect, the core plug must be a real reservoir core, and the initial water saturation at which the reference permeability is measured must be set to exactly that of the reservoir. Sinomine is not aware of any coreflood test laboratories that have this level of control over Sw_i. Instead, experience shows that test laboratories strive to set the Sw_i as low as possible and refer to this as 'irreducible water saturation', Sw_{ir}.

6.2 Artifact – use of dry gas to perform drawdown

Natural gas in its native environment is in thermodynamic equilibrium with the connate liquid-water phase and is saturated with water vapor at reservoir conditions. Full water saturation of gases used in laboratory coreflood tests is often not achieved, which means that fluids inside of the core plug dehydrate when contacted by undersaturated gas.

This problem of permeability impairment resulting from dehydration of the filtrate by unsaturated gas is more evident in coreflood tests performed at high temperatures with high-salinity drilling/completion fluid filtrates, such as formate brines. These brines already contain salt concentrations close to saturation levels and are therefore susceptible to significant viscosity gain or crystallization by dehydration. Such dehydration can also lead to an increase in surface tension, which increases the likelihood of fluid retention.

Unfortunately, a lot of the earlier coreflood tests performed with formate brines used either dry nitrogen gas or nitrogen gas saturated with water at ambient temperature and high pressure to simulate the reservoir gas. All the gas tests performed by Corex in the period up to 2007 were carried out with dry gas. This may have significantly affected the results of the tests reviewed by Byrne et al. (2002). Sinomine is aware that many test laboratories still use dry gas or gas humidified at ambient temperature.

Sinomine has carried out some simple tests to look at the effect of flowing dry nitrogen gas through formate brine. A 2.20 g/cm³ (18.3 lb/gal) cesium formate brine sample was purged with dry nitrogen gas at 65°C (149°F). Brine density was measured as a function of purging time. The results in Figure 8 show that, even at this rather low test temperature of 65°C (149°F), a significant increase in brine density occurred as a result of dehydration by dry gas.

Effect of brine dehydration on density

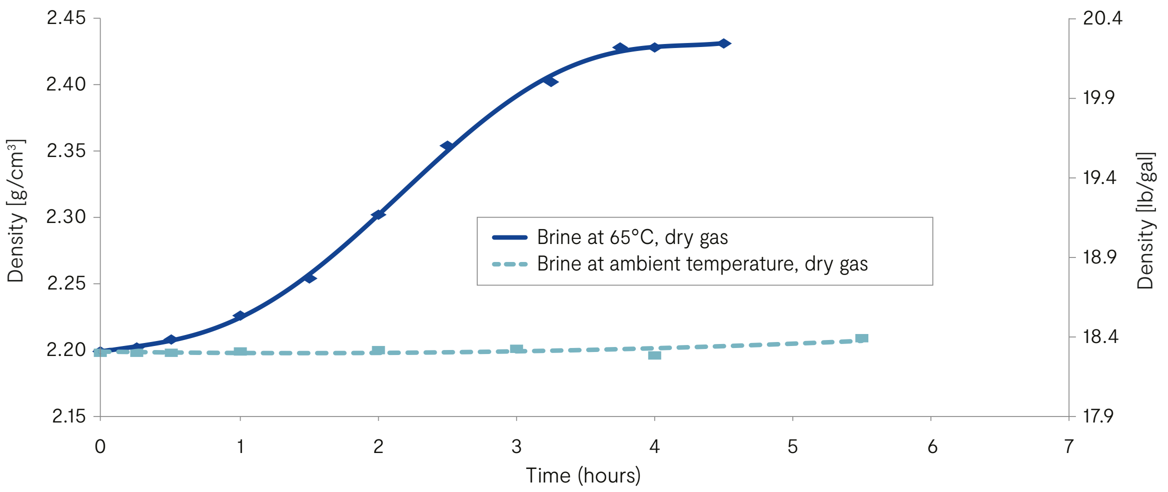


Figure 8 Density of 2.20 g/cm³ (18.36 lb/gal) cesium formate brine as a function of time while purging with dry nitrogen gas. The significant brine-density increase seen in the blue curve is a result of purging dry gas through formate brine held at 65°C (149°F).

A more sophisticated study was carried out by Downs (2009) to investigate the effect of gas humidification levels on gas permeability of North Sea HPHT reservoir core material exposed to high-density cesium formate brine under HPHT conditions in laboratory coreflooding experiments. Results from the coreflooding experiments at 200°C (392°F) indicate that full HPHT-humidification of the gas phase resulted in higher gas return permeability when compared with a test using gas humidified at room temperature and high pressure. The difference can be explained by studying the equilibrium water content of nitrogen gas as a function of pressure and temperature, shown in Table 4. High-pressure (34 MPa (5,000 psi)) gas saturated with water at 200°C (392°F) **contains almost 400 times more water** than the same gas saturated at room temperature. This means that gas saturated at room temperature can pick up a lot of water while passing through the brine-saturated core under HPHT conditions during the drawdown phase. This finding highlights the importance of ensuring that any gases used in HPHT coreflood tests are fully saturated with water vapor at test temperature and pressure. It seems likely that the impact of gas humidification levels is amplified in very low-permeability cores subject to high drawdown pressures.

Table 4 The equilibrium water content of nitrogen gas as a function of pressure and temperature.

Gas pressure [psi]	Equilibrium water content [ppm]				
	20°C (68°F)	75°C (167°F)	125°C (257°F)	175°C (347°F)	200°C (392°F)
14.7	23,165	381,987	–	–	–
100	3,479	56,997	342,427	–	–
500	768	12,145	71,895	275,272	476,445
1,000	432	6,543	37,914	143,983	250,102
2,000	267	3,743	20,865	77,429	133,911
3,000	212	2,803	15,125	55,984	94,429
5,000	185	2,324	10,415	36,631	62,114
8,000	–	–	–	25,799	43,125

(Source: AQUAlibrium 3.1 – copyright 2006, FlowPhase Inc.)

It should be noted that in addition to being humidified at test temperature, the gas also needs to enter the core plug at test temperature. If the gas is cooled down after it is humidified, it will not be fully saturated at the temperature inside of the core plug. This artifact is likely to have a higher impact when testing on high-permeability cores as gas rates are higher.

6.3 Artifact – failure to maintain reservoir temperature during testing

Some test laboratories perform coreflood testing at ambient temperature, or they cool the test cell down to measure permeability at ambient conditions. Cooling of the test cell leads to viscosity increase of formate filtrate and potential crystallization of water-soluble salts from contact between formate filtrate and formation water. This situation is exaggerated in situations where the filtrate is dehydrated with dry gas (see section 6.2) or when unbroken biopolymers are still present in the filter cake.

A remarkable artifact created by the combination of dry gas and cooling of the test cell was seen in a coreflood test with formate brine at 200°C (392°F). A return permeability >100% of the original permeability was measured in the core sample under reservoir conditions, but when the core was cooled to 60°C (140°F) some salt crystallization occurred within the core and caused severe permeability reduction. This artifact was created by cooling brine filtrate residues, which had been dehydrated by using gas that had not been water saturated at test temperature. It is therefore important, not only that the gas is fully water-saturated at test temperature, but that all permeability measurements are conducted at test temperature as well.

6.4 Artifact – lack of gravity drainage in coreflood test cell

Operators and coreflood laboratories often have preferences to whether a coreflood test should be conducted in a vertical or horizontal cell orientation. Unfortunately, neither orientation represents field conditions. This may not be a problem when testing traditional solids-laden drilling fluids where the main reduction in return permeability occurs due to plugging with weighting solids and a thick filter cake. Here, the solids and filter cake can only be removed by drawdown/hydrocarbon production, which can be simulated in the coreflood test cell. Mobile formate filtrate, however, also cleans up by gravity drainage, which is not properly addressed in coreflood tests. As illustrated in Figure 9, neither horizontal nor vertical cell configuration allow for gravity drainage, as it occurs in the reservoir after filtrate invasion:

- Vertical configuration: Allows for gravity drainage, but the heavy formate brine filtrate must drain through a low-permeability filter cake.
- Horizontal configuration: Does not allow for gravity drainage of the filtrate due to the cell configuration, although the brine may channel through the basement of a core during flow and slump further to the basement during static periods.

Effect of fluid gravity drainage in the field and in the coreflood test cell

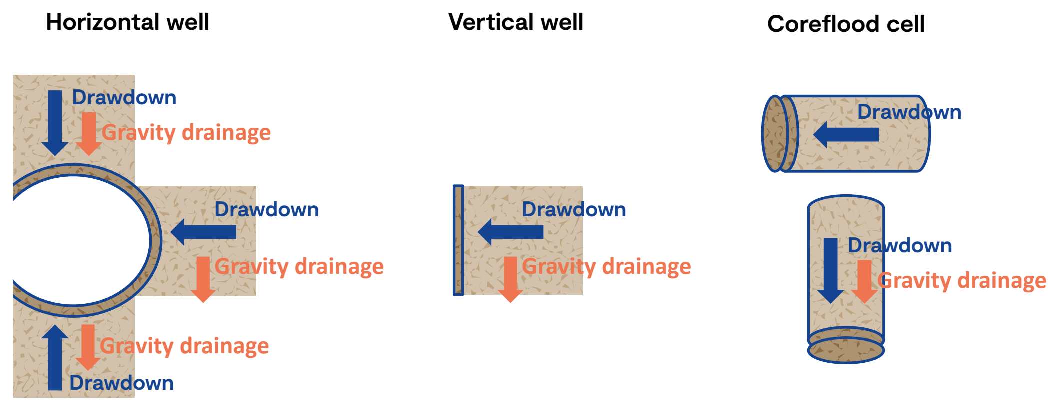


Figure 9 Illustration of how gravity and drawdown act in horizontal and vertical wells compared to horizontal and vertical coreflood test cells. Neither of the test-cell orientations allow for proper drainage of heavy formate filtrate through a permeable reservoir.

In gas-bearing sandstone reservoir sections surrounding wellbores drilled with heavy formate brines, it is common to observe the formate filtrate draining downwards through the rock, pulled by gravity. The filtrate is replaced by gas as it drains away. Equinor (Pedersen et al. 2006) reports: “LWD logs were acquired 3 days after drilling and the wireline logs were acquired 6–7 days after drilling. It is evident from the Figure 10 that wireline density tends to be less affected than the LWD density. This is commonly observed in these fields, which is probably due to the fact that the formations are rapidly invaded very fast during the drilling and, subsequently, due to partial displacement and gravity segregation, we see a reduced influence of cesium/potassium formate on logs at the time of wireline logging.” Movement of cesium formate filtrate is visible on the photoelectric factor (PEF) and resistivity logs, and Equinor uses this movement to define net reservoir (Downs and Fleming 2020).

To further study the differences between the gravity effects in a coreflood test cell and a real reservoir a study was carried out for Sinomine by Lloyds Register, using CFD modelling to study the behavior of high-density formate brine when left inside of a core plug in a core holder and when left inside of a reservoir. The results were presented at the SPE European Formation Damage Workshop in Budapest, Hungary in 2017. The presentation is available from Sinomine on request. Lloyds Register concludes that gravity affects the results of the coreflood study, and that there are significant differences between the coreflood test design and a real field situation. An example of how gravity affects a coreflood test in a horizontal test cell is shown in Figure 11. In this simulation, the horizontal core plug was exposed to formate drilling fluid and left static for 14 days. The core plug was then exposed to drawdown with gas. The conditions used were: temperature = 200°C (392°F), fluid density = 2.00 g/cm^3 (16.7 lb/gal), core plug permeability = 1,000 mD, filter-cake permeability = 1 mD, porosity = 25%. A simulation of a real reservoir was run under the exact same conditions. The results are shown in Figure 12.

Based on the logging observations by Equinor and the study by Lloyds Register, it can be concluded that gravity drainage is a real phenomenon that removes heavy formate filtrate from the near wellbore in the field. Unfortunately, in the laboratory, the cell configuration does not allow removal of vertically draining filtrate from the core.

In addition to the coreflood test configuration, the unrealistically short time used during drawdown in coreflood tests further prevents drainage of heavy formate filtrate.

Sinomine cannot offer any recommendation for how this artifact can be eliminated.

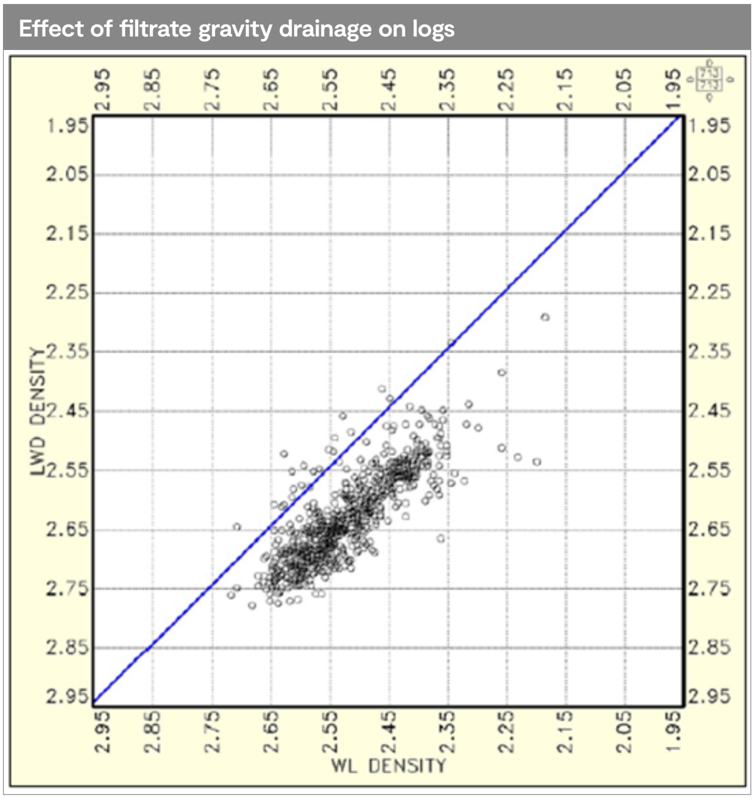


Figure 10 Cross-plot of wireline density (x-axis) vs. LWD density (y-axis) showing that wireline density is less affected than LWD density by the presence of cesium/potassium formate brine. The figure is taken from Pedersen et al. (2006).

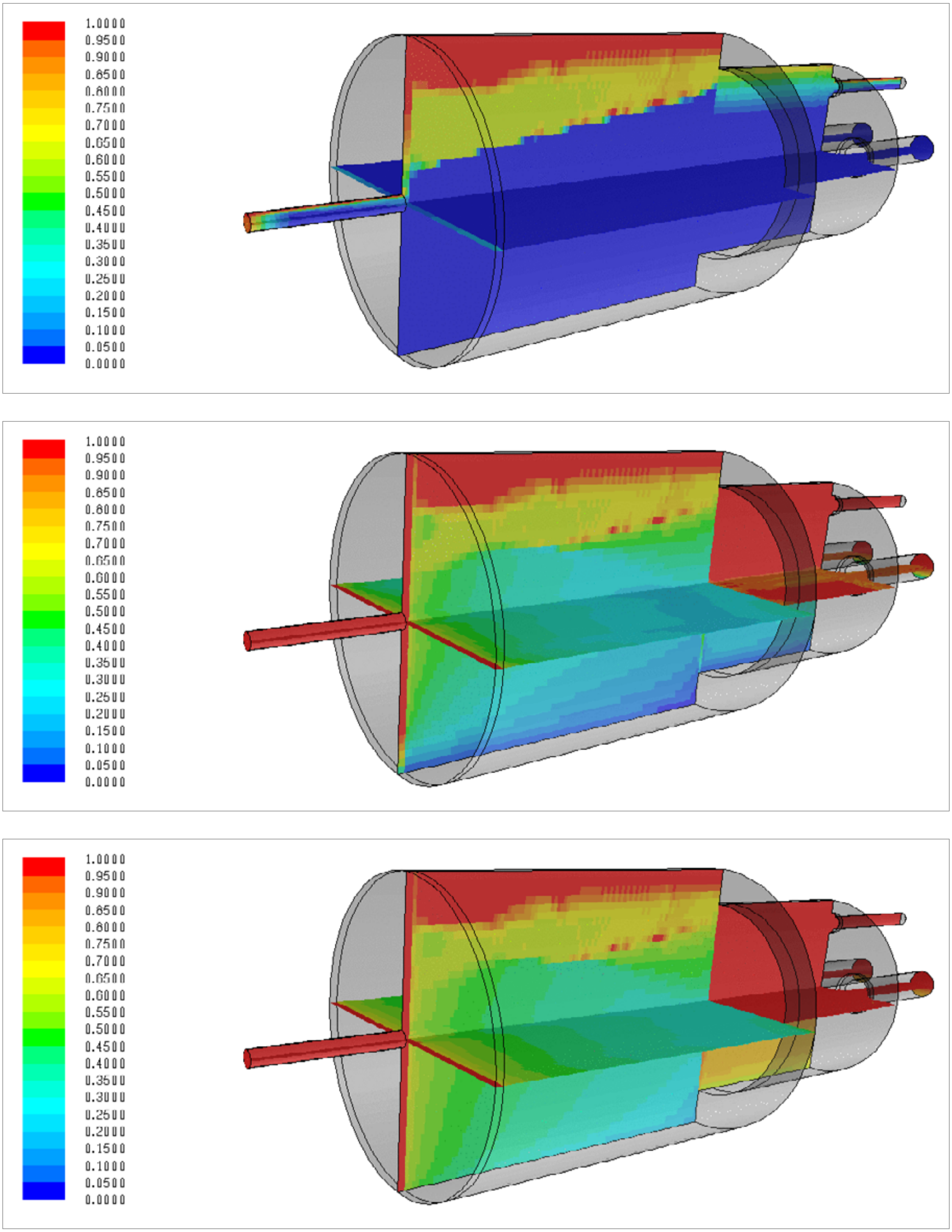


Figure 11 Impact of gravity on water saturation in a horizontal core plug in a constrained core holder. Temperature = 200°C (392°F), fluid density = 2.00 g/cm^3 (16.7 lb/gal), core plug permeability = 1,000 mD, filter-cake permeability = 1 mD, porosity = 25%. Top: Water saturation in core plug after 14-day suspension with formate brine. Middle: Suspended core after flushing with nitrogen gas for one hour. Bottom: After ten hours flushing with nitrogen gas.

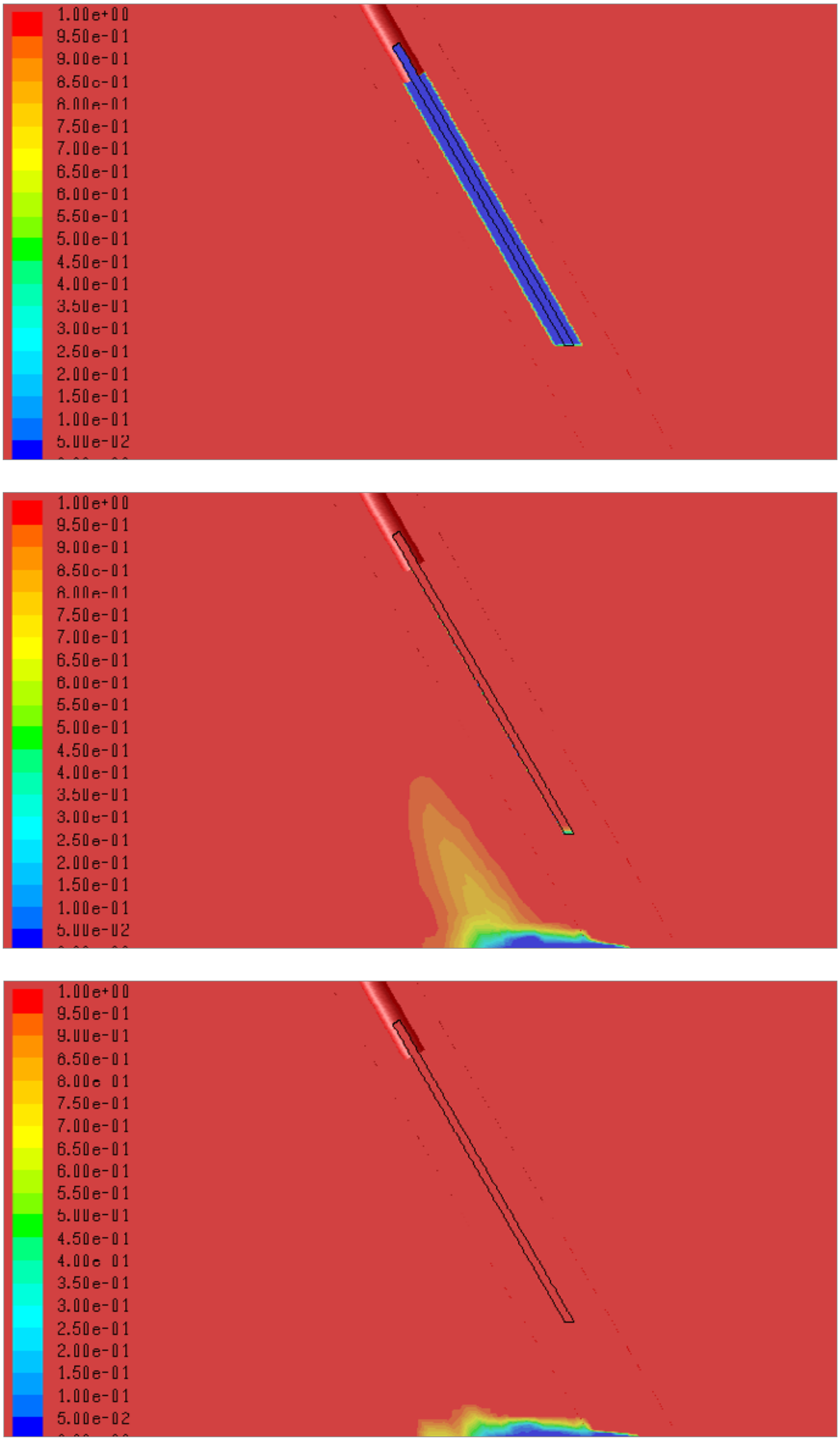


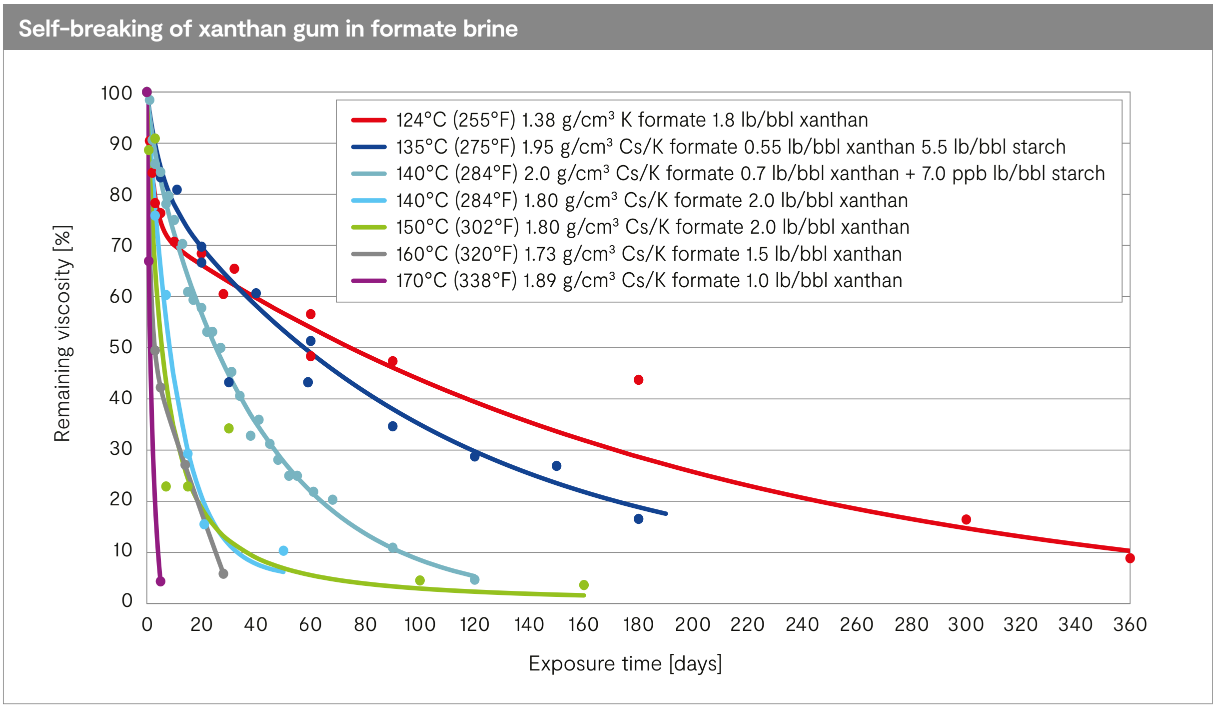
Figure 12 Impact of gravity on water saturation in the reservoir around a deviated well. Temperature = 200°C (392°F), fluid density = 2.00 g/cm^3 (16.7 lb/gal), core plug permeability = 1,000 mD, filter-cake permeability = 1 mD, porosity = 25%. Top: Water saturation in the reservoir around a wellbore with a filter cake after exposure to the formate fluid. Middle: After a two-day shut-in. Bottom: After four days' gas production.

6.5 Artifact – not allowing time for biopolymers to thermally degrade

Time is one of the factors that significantly differentiates a coreflood test from a real field application. In a coreflood test, the core is expected to clean up in hours, and sometimes even minutes. The coreflood test laboratory typically quits cleaning up the core as soon as the hydrocarbon flow has stabilized, or earlier if large amounts of gas are consumed, which is commonly accepted 'proof' that the core plug is no longer cleaning up. In the field, however, the cleanup process continues for days, weeks, and even months. A documented example of this is the bp Rhum application (see Roy et al. 2008).

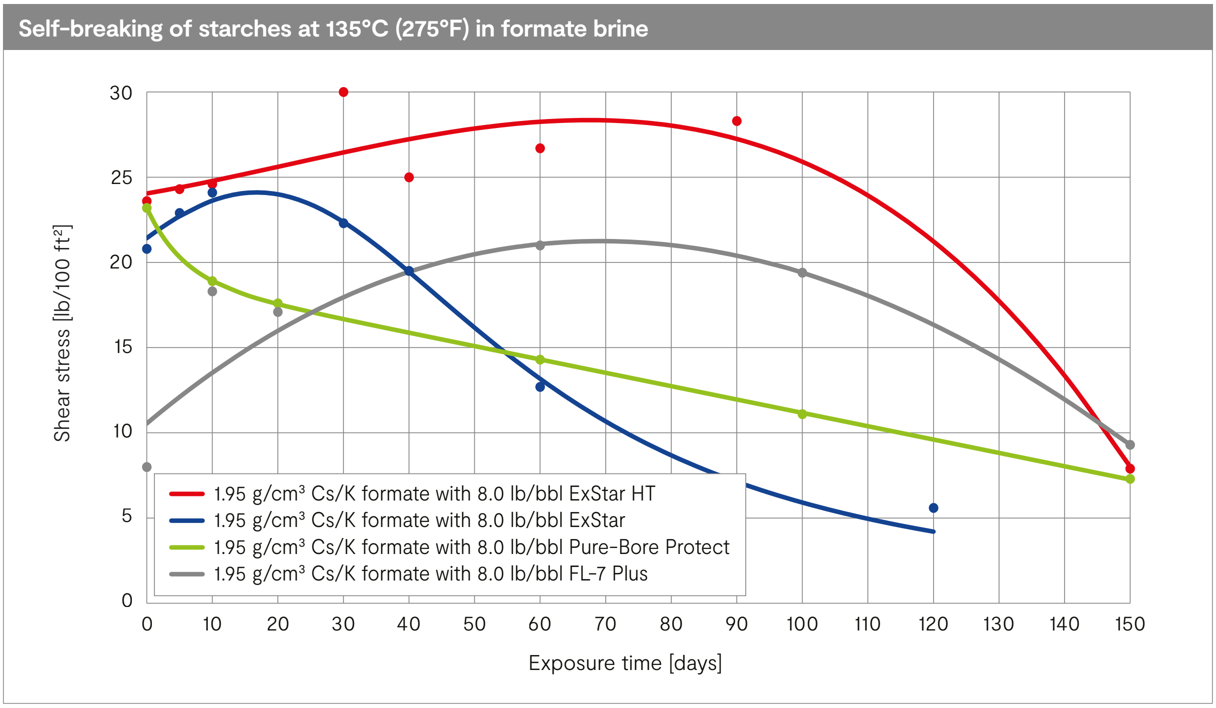
The most obvious artifact introduced by the time restriction is the prevention of natural self-breaking biopolymers within both the external and internal filter cakes, and to some extent, within the filtrate, from breaking down under hydrothermal conditions. Formate brines stabilize biopolymers to high temperatures (Howard 1995, Howard et al. 2015, Sinomine Formate Technical Manual, Section B5 Compatibility with Additives). This is highly beneficial as temperature-stable drilling, screen-running, and perforating fluids can be formulated by only using non-damaging, self-breaking additives. These additives, depending on the temperature they are exposed to and the type and concentration of the formate brine, eventually break down naturally over weeks or months (Anderson et al. 2023), but not during the typical timeline of a coreflood test.

Figure 13 shows the self-breaking rate of xanthan gum as a function of temperature in concentrated blends of cesium and potassium formate brines, and in the case of the 124°C (255°F) study, in a water-diluted potassium formate brine. Viscosity measurements taken at 49°C (120°F) were used as a measure of polymer degradation. The detailed measuring method is described by Anderson et al. (2023). The sample that was tested at 135°C (275°F) and one of the samples tested at 140°C (284°F) contained a significant amount of proprietary starch-based fluid-loss-control polymers. From the figure, it is evident that these two samples have lower initial degradation rates than similar formate brines containing only xanthan gum, which implies that the modified-starch-based fluid-loss-control polymers break down slower than xanthan gum. To verify this, similar testing was conducted on formate brine samples containing only modified starch. Four different kinds of modified starch were tested independently in the same 1.95 g/cm^3 (16.3 lb/gal) cesium/potassium formate brine at 135°C (275°F). The results, which are plotted in Figure 14, show that both the regular starches and the HT starch break down over time at 135°C (275°F) in the concentrated cesium/potassium formate brine, but at a much lower rate than xanthan gum. It is interesting to note that three of the four starches, at this temperature, continued hydrating for a significant number of days before they started degrading, something that can severely affect the results of standard short-term coreflood testing.



Self-breaking of xanthan gum in formate brine

Figure 13 Self-breaking rate of xanthan gum as a function of temperature in cesium/potassium formate blends and a diluted potassium formate brine. Two of the samples contained significant amounts of proprietary starch-based fluid-loss-control polymers. The figure shows the remaining viscosity percentage as a function of exposure time.



Self-breaking of starches at 135°C (275°F) in formate brine

Figure 14 Self-breaking rate of various proprietary starch-based fluid-loss-control polymers at 135°C (275°F). All starches were added to 1.95 g/cm³ (16.3 lb/gal) cesium/potassium formate brine at a concentration of 23 kg/m³ (8.0 lb/bbl). The viscosity, measured at 49°C (120°F), is plotted as a function of time after the brine viscosity has been subtracted.

Apart from temperature, there are other factors that determine the self-breaking rate of biopolymers. Formate brine concentration is one of them. Figure 15 shows a comparison of the xanthan self-breaking rate in samples with three different concentrations of formate. It also shows how xanthan degrades faster in an unbuffered formate sample than in a sample buffered with carbonate/bicarbonate pH buffer.

Self-breaking of xanthan at 124°C (255°F) in formate brine

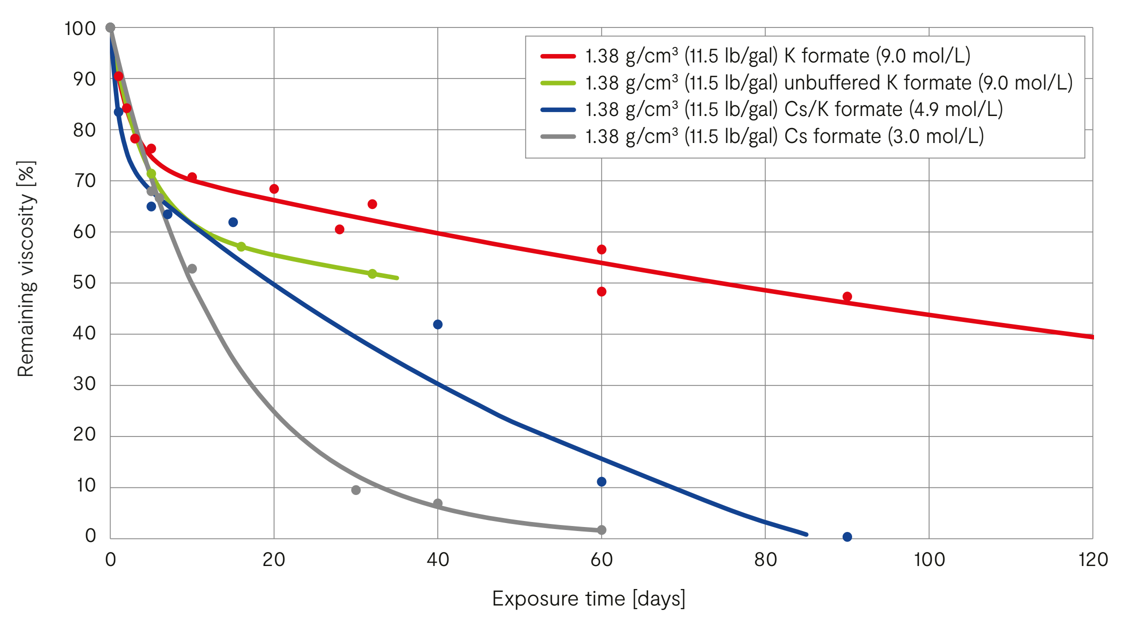


Figure 15 Effect of formate type on the self-degradation rate of 5.10 kg/m³ (1.8 lb/bbl) xanthan gum in four different formate brine compositions of the same density, but with different concentrations of cations, formate ions, and water. The figure shows the remaining viscosity percentage as a function of exposure time at 124°C (255°F).

The self-breaking rate of biopolymers is a factor that needs to be considered when coreflood testing all water-based fluid systems. However, given that formate brines are exceptionally good at slowing down the thermal degradation of biopolymers, and spurt losses can be high, delayed polymer degradation can easily become the prominent 'damage mechanism' in a short-term test. An illustration of how fast xanthan breaks down in different brine systems at 124°C (255°F) is shown in Figure 16.

Measuring and understanding the self-breaking rate of biopolymers in different base-brine systems is not only important for interpreting coreflood test results, but also for designing fluids for real reservoir applications, and for proper design of coreflood tests.

If increasing the time of a coreflood test to allow for polymer self-breaking is not feasible in the test laboratory, an alternative method involves accelerating polymer degradation by temporarily increasing the test temperature and lowering it again before permeability is measured. An example of such a coreflood test is given in section 5.3. Here, it is shown how the injectivity of formation water increased from 3.8% to 98% when biopolymers protected by potassium formate were allowed to break down for 41 days.

Self-breaking of xanthan at 124°C (255°F) in various brines

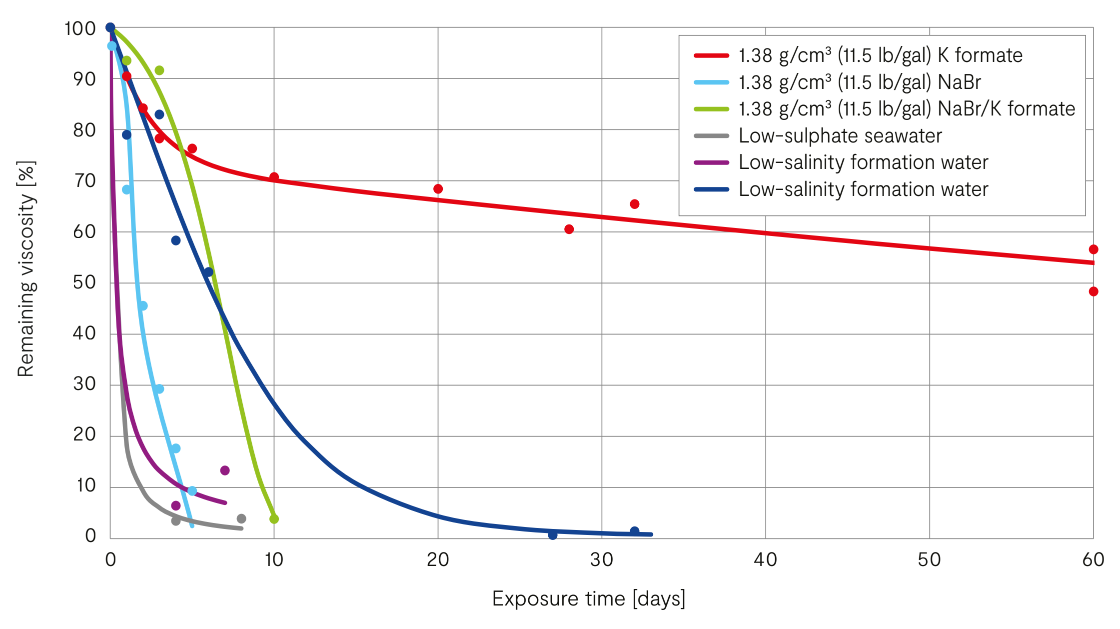


Figure 16 Effect of bromide brine and blends of formate and bromide brines on the self-degradation rate of 5.10 kg/m^3 (1.8 lb/bbl) xanthan gum at 124°C (255°F). All brines had a density of 1.38 g/cm^3 (11.5 lb/bbl). The figure shows the remaining viscosity percentage as a function of exposure time.

6.6 Artifact – presence of end effects

A test was conducted to study the behavior of cesium formate brine as it was injected from the bottom of a vertical core plug mounted in a core plug holder. One-third pore volume of brine was injected. Micro-CT images were taken before and after injection, and the change map was studied.

It was expected that most of the brine would be in the bottom of the core plug with a very small portion traveling to the top through microfractures and high-permeability channels. However, this was not what was seen from the change map shown in Figure 17. This shows that in addition to cesium formate in the bottom part of the core plug, there is quite a lot of cesium formate at the very top of the core plug compared to the center section.

It appears that the heavy cesium formate brine has channeled through the core, accumulated as a pool on top of the core, and partially drained back into the core plug. Considering that fluid loss from a formate coreflood test can easily exceed a third of pore volume, and most often even more than one pore volume, this can have quite a significant effect on water saturation levels at the reservoir face of the core, especially in a vertical coreflood test. See Figure 18. In the core plug, the whole reservoir face surface and the pores at some distance downwards are filled with formate brine.

During drawdown, the hydrocarbon phase has to push this front of filtrate through the whole core plug to restore the initial water saturation. In a real well situation, hydrocarbons flow freely through all areas not invaded by filtrate. Filtrate that then needs to be pushed out has entered the core from the wellbore side and can flow back out the same way it entered. There may be an option to trim the core at the reservoir side before cleanup to avoid this artifact. This option, which has not yet been explored by Sinomine, can introduce another artifact as the core needs to be cooled before drawdown.

Sinomine understands that in some coreflood test laboratories, all filtrate cannot be removed from the reservoir side before drawdown, so this artifact is also present in horizontal test configurations.

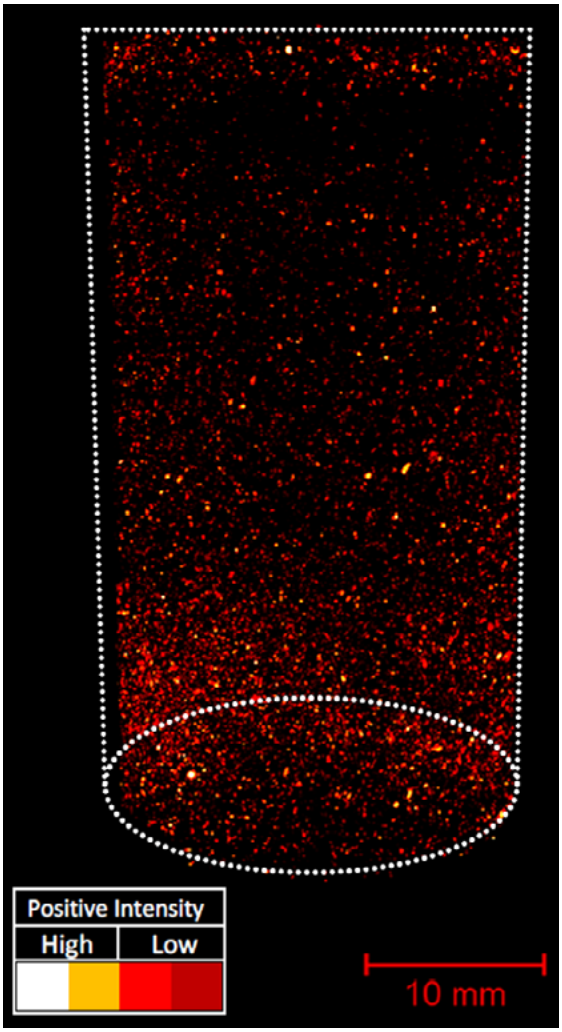


Figure 17 Micro-CT change map after injection of ⅓ pore volume of cesium formate brine from the bottom of a ~20 mD core plug mD at 149°C (300°F). The red and white dots show the pores invaded by cesium formate brine. Dot intensity has been increased evenly to improve clarity.

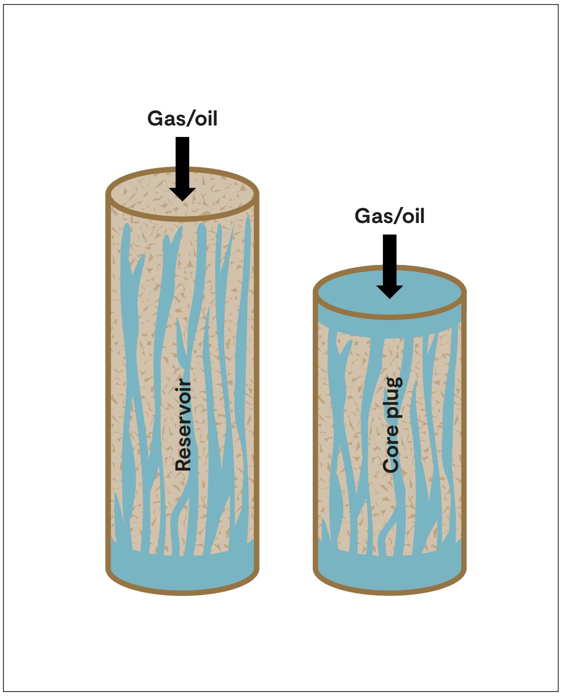


Figure 18 The difference between how hydrocarbons flow through the real reservoir compared to a short core plug, where formate brine has covered the whole reservoir core face. In the core plug, the whole surface and the top part inside the core plug are filled with formate brine.

6.7 Artifact – lack of CO_2 during drawdown

Substituting reservoir gas with nitrogen gas may not cause any problems in most coreflood tests. However, if the formation water contains enough divalent ions to react with the carbonate ions in the pH buffer commonly added to formate brine, absence of CO_2 in the test gas may be a problem. In the reservoir, equilibria establish between CO_2, bicarbonate, and carbonate. By eliminating one of these components, these equilibria are affected, and carbonate precipitates may form that do not form in a real reservoir situation.

7 Coreflood testing – other useful tests

To better understand how formate brine or filtrate interact with the reservoir, it is often useful to perform a simple brine test before the drilling fluid is tested. A simple coreflood test with formation water, in other words another brine system, may also give useful information about whether the test provides a reasonable chance of obtaining a successful result.

7.1 Perform a brine test in addition to testing the drilling fluid

If duplicate core material is available, it is highly recommended to perform a brine test in addition to the drilling fluid test. Replacing a predominantly monovalent sodium chloride-rich formation water with a predominantly monovalent sodium/potassium/cesium formate-rich brine, as the fluid filling 10–20% of the porosity of a rock, fingerprints how the core reacts to having one clear alkali-metal-salt brine replaced by, or mixed with, another clear alkali-metal-salt brine.

The composition of the brine should be the same as the drilling fluid's base brine, which should represent the drilling fluid filtrate. The brine test should be performed according to the same procedure as the drilling fluid test described in section 4, with the following exceptions:

- Instead of circulating the drilling fluid across the wellbore end of the core, pump approximately 10 pore volumes of filtrate through the core in the direction from the wellbore to the reservoir at a low pump rate of approximately 1 mL/min or preferably less.
- Removal of external and internal filter cakes is not required.
- Trimming of core is not required.

The results of the brine test will:

- Show if there are any incompatibility issues between the brine base itself and the core mineralogy or formation brine, free from the masking effects of polymers and fine solids present in drilling fluids.
- Indicate whether fluid retention can be expected from exposure to brine alone, free from the masking effects of polymers and fine solids present in drilling fluids.

7.2 Perform a complete coreflood test with formation water

Performing a coreflood test with formation water can be a useful exercise if the operator has designed a test where significant filtrate retention is a likely outcome. This is typically in a high-permeability core plug with the initial water saturation established by a centrifuge and with only a low drawdown applied during cleanup. If the formation water test shows low return permeability, then the test is set up to fail.

A core flooded with formation water in the same way as a test fluid and then exposed to the same drawdown procedure provides an excellent fingerprint or template to judge a test fluid put through the same procedure. If the selected drawdown cannot remove all of the formation-compatible formation water from the core with the same efficiency as the method used to establish Sw_i at the preparation stage, then the same drawdown regime is unlikely to succeed with formate brine in the core.

7.3 Perform a mild cleanup after return-permeability testing

It is useful to remember that there is nothing in a formate brine or a formate drilling fluid that cannot be readily eliminated from a core or formation by the application of a dilute organic acid. In other words, there is nothing in a properly designed formate fluid that causes permanent formation damage requiring by-pass or dissolution/removal by powerful acids.

Application of mild organic acid, or even water, in coreflood testing has proven to improve test results in cases where fluid retention has made it difficult to pass coreflood test requirements:

- During coreflood testing to qualify cesium/potassium formate drilling fluid for the Huldra field, Equinor struggled with poor return-permeability results. In all cases, core permeability was restored to original levels by soaking the wellbore face of the cores at balance for 24 hours with 15% acetic acid under reservoir conditions. The full restoration of permeability by non-invasive soaking of the core faces with dilute organic acid at balance suggests that the source of the tractable impairment was residual $CaCO_3$/polymer filter cake still present on the core faces after a very short cleanup period (Downs and Fleming 2018). The low return permeability is most likely not caused by the filter cake itself, but rather by the filtrate trapped behind it.
- During a follow-up coreflood study by Equinor for qualification of cesium/potassium formate drilling fluid for the Huldra and Kvitebjørn fields, Equinor experienced similar problems, and discovered that by applying a mild cleanup procedure, consisting of flushing the core with two pore volumes of formation water prior to repeating the return permeability test, all permeability was regained (Berg et al. 2007, 2009).

Equinor went ahead and used cesium formate as drilling fluid in both its Huldra and Kvitebjørn fields without applying any acid or water treatment. The good production from both Huldra and Kvitebjørn indicates that they have cleaned up naturally without any intervention. Most likely the acid and water treatments in the laboratory accelerated the degradation of natural biodegradable polymeric constituents in the formate drilling fluid filter cake in the same way that the extra time did in the field. Not only time, but also contact with acid gasses and condensed water during production startup, will accelerate degradation of biopolymers. In the field, of course, drainage of retained filtrate is also likely to have accelerated the cleanup.

It should be mentioned that although treatment with organic acid in some laboratory studies has proven to be a powerful method of removing the filter cake, thereby allowing formate filtrate to escape from the core plug, there has never been any need for acid treatment in the field. Sinomine is only aware of one occasion where acid has been applied in real wells, which is in Petronas' Kanowit field. Petronas treated one of two wells with high-rate acidization with 15% HCl and gel diverters. Multi-rate well testing showed that both wells could produce at an absolute open-flow rate greater than 150 MMscf/day, which is 50% higher than the original technical potential estimated in the field development plan (Carnegie et al. 2013).

7.4 Use of imaging techniques to identify damage mechanisms

Dry SEM and cryogenic SEM imaging methods are commonly used to identify damage mechanisms after coreflood testing. These methods can be powerful tools for identifying damage mechanisms, such as fines migration and plugging of core plugs with solids. However, **these imaging methods are not suited for identifying or quantifying retention of formate brine filtrates**. To understand why, it is important to have a good understanding of what happens when monovalent formate brine enters the core plug or reservoir, which have certain amounts of formation water present (Sw_i).

1. Monovalent Cs^+, K^+, or Na^+ and formate ($CHOO^-$) ions enter the pore volumes in the core plug and 'mingle' with formation water.
2. The monovalent ions from formate brine do not react with ions from the formation water to form any salts or scales, but they blend fully with the formation water they come in contact with.
3. During cleanup/drawdown, water is ejected from the core plug. This is a mix of formate brine filtrate and formation water.
4. In the same way as all water is not removed before testing, when Sw_i is established, some water is also present in the core plug after testing. The water left in the core plug after testing is naturally a blend of formation water and formate brine.
5. The amount of water left in the core plug impacts effective permeability, which consequently influences coreflood test results. This cannot be measured by detecting Cs^+ or K^+ ions in the core plug. Cs^+ and K^+ ions are present even in cases where water saturation has decreased and the coreflood experiment results in a return permeability >100%.
6. Even with the most efficient drawdown method or spindown with ultra-centrifuge, an irreducible amount of water is present. This irreducible amount of water must contain Cs^+ and/or K^+ ions.

In other words, the observation of Cs^+ and/or K^+ ions in a core plug after testing with cesium/potassium formate brine is **not** an indication of filtrate retention. If a test laboratory suggests such a scenario, it is incorrect.

Observation of Cs^+ and/or K^+ ions in a core plug after testing with potassium/cesium formate brine is not an indication of filtrate retention.

An example of a dry SEM image incorrectly interpreted as formate drilling fluid filtrate retention is shown in Figure 19. Here, Cs^+ and K^+ ions are seen throughout the image, which is a clear sign that this very mobile filtrate has entered this portion of the core. It is also a good indication that an end effect in the part of the core near the reservoir end-face could have been in play.

Please also note that the dry SEM images are compared with initial images from a different core plug not exposed to formation water and are therefore not representative of the actual core plug's initial condition. This means that if a core plug is not compatible with the formation water used, e.g., clay swelling is caused, this incompatibility would be assigned to the test fluid rather than the formation water. For a dry SEM comparison to be useful, the core plug used for the initial image needs to undergo the same procedure to establish Sw_i as the test plug.

Of the many cryogenic SEM images presented to Sinomine after coreflood testing with formate brines, no useful information has ever been extracted.

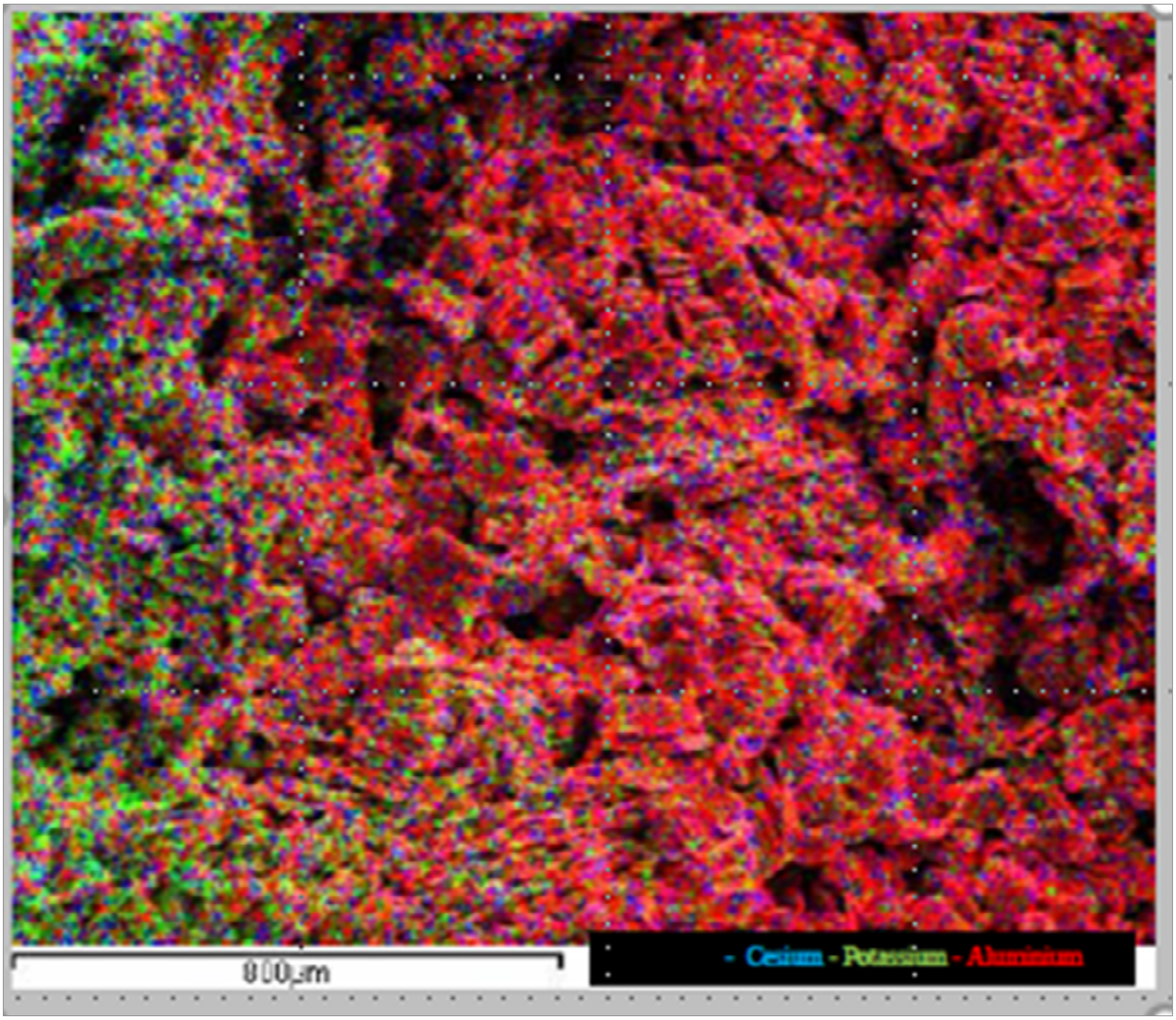


Figure 19 Typical dry SEM image showing presence of cesium and potassium ions after testing of cesium/potassium formate fluids. This is incorrectly referred to as filtrate retention.

Micro-CT imaging, displayed in Figure 17, is useful as it shows how quickly the formate brine injected from the bottom of the core plug channeled through the 20 mD sandstone and ended on top. However, the same applies to this type of imaging as to the dry SEM; it is not suited for identifying whether formate filtrate has cleaned up from the core plug. Due to the mixing of formation water and formate brine, Cs^+ and K^+ ions are still in the core after its water saturation has reduced to a level lower than the initial water saturation and even when it reaches its absolute minimum irreducible water saturation. Sinomine also warns against any method that involves taking the core plug out of the core holder and cooling it down. If dehydration takes place in the core, increased viscosity and possible crystallization of the retained filtrate may cause additional laboratory artifacts. If CT imaging could be conducted in real-time in the oven, it would be a more useful tool.

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