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# **How to Test Fluids for Shale Compatibility**

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#### **Abstract**

Fluid-shale compatibility testing is as old as the drilling fluid industry itself. When drilling fluids started to be deliberately used for hole-making, the (in-)compatibility of these fluids with clay-rich shale formations became immediately apparent, and industry scientist have been trying to make sense of it all ever since. With a plethora of possible shale tests available, a key question remains: what are the best, most representative tests to characterize fluid-shale interactions and avoid making decisions based on less sensitive tests that may suffer from artifacts and yield misleading results?

This paper argues for the use of a representative set of shale tests that includes accretion tests, cuttings dispersion tests and mud pressure transmission tests, while pointing out issues and problems with other tests such as atmospheric swelling tests and capillary suction tests, which still find wide-scale application in the industry. Moreover, it introduces a novel, low cost borehole stability test in the form of a modified thick wall cylinder test. This new test exposes a cylindrical shale samples, confined under downhole temperature and pressure, to mud formulations at overbalance for a specified period of time and assesses the failure strength of the sample thereafter. The test is thereby capable of mimicking the results of much more sophisticated, and much more expensive, test protocols such as the downhole simulation cell test.

The details on the new test protocol are given here. Moreover, it is shown how the proposed test protocol can be used to the comprehensive qualification of the merits of new nano-particle and high-salinity fluid formulations.

### Introduction

A significant body of literature dedicated to shale-fluid compatibility and associated testing exists and forms a discipline in itself. Shale-fluid testing remains an active topic of interest, not only for drilling fluid evaluation but also for drill-in / completion fluid development and optimizing waterinjection treatments. Water injection for reservoir pressure maintenance and enhanced oil recovery in particular appears to be a growing area interest, judging from publication activity in recent years. This, of course, is related to the fact that hydrocarbon production in many of the major reservoirs around the world is in decline, placing emphasis on enhanced

oil recovery. Moreover, many operators have experienced issues with the integrity of shale zones and cap rocks overlying reservoirs during prolonged water injection due to shale-fluid incompatibility. It is important that such operators have access to the best possible advice on shale-fluid compatibility in order to avoid costly mistakes that may jeopardize the economics of their waterflooding projects.

Another recent trend (Teke et al., 2012; Maghrabi et al., 2013) is the use of more sophisticated data-analytics techniques for analyzing the results from simple shale-fluid tests such a Capillary Suction Tests (CST) and Atmospheric Swelling Tests. The concern here is not with the new analysis techniques themselves, which appear to be quite appropriate in the current era of dealing with big data. Rather, the issue is the validity of the results provided by the simple tests: sophisticated analysis of flawed test results still leads to flawed outcomes and advice.

In this paper, we analyze the strengths and weaknesses of shale-fluid compatibility test protocols, and highlight potential issues that may occur with certain tests. From this, guiding principles and criteria for test selection and execution are derived. Moreover, laboratory test sets that reflect most accurately on shale-fluid interactions in the field are recommended. The test set recommended for shale wellbore stability evaluation is augmented with a new test protocol based on modification of the thick walled cylinder test.

### **Guiding Principles for Testing**

Figure 1 shows a model (van Oort, 2003) for the forces acting on a shale system containing clays and other minerals. These forces are divided into mechanical and physio-chemical forces. The former include:

- the in-situ stresses;
- the pore pressure;
- the stress (acting either in tension or compression, depending on the overall stress state) acting at intergranular contact points in cementation bonds.

The latter, acting primarily in the clay fabric, include:

- the van der Waals attraction;
- the electrostatic Born repulsion;
- short-range repulsive and attractive forces that stem from hydration / solvation of clay surfaces and associated ions.

The physio-chemical forces acting within the shale fabric combine to generate what is generally known as the "swelling pressure", which is responsible for the shale swelling phenomena that are observed in shale fluid compatibility tests.

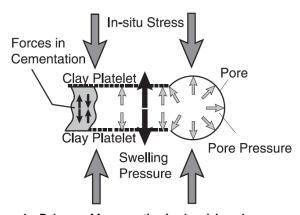


Figure 1 – Balance of forces acting in clay-rich rocks.

The shale forces model is useful to help guide us in the selection of suitable representative shale-fluid compatibility tests and test conditions.

First, let us explore the effect of limited external stress acting on the shale sample. This is the situation when shale-fluid interaction tests, particularly swelling tests, are conducted at atmospheric pressure conditions. Note that an actual shale under downhole in-situ stress conditions never experiences this condition. Even when the in-situ stress is removed when the shale is cut by the bit, the static or dynamic annular pressure still acts as an effective confining force. Of course, this force diminishes as shale cuttings are circulated up the annulus, but atmospheric pressure conditions are experienced only very briefly when the cuttings is circulated out of the well and screened out on the solids control equipment.

Looking at Figure 1, it is immediately clear that tests with limited or no confining pressure will release the hold on the swelling pressure, which in turn will exacerbate shale swelling tendencies observed during the test. These effects, however, are quite uncharacteristic of the shale's downhole behavior in response to applied fluids. This can significantly mislead the experimenter on the "reactivity" of the shale, with associated misguidance on the selection of compatible fluid type and formulation.

Secondly, we explore the effects of rock cementation that is either compromised or destroyed. This is, of course, the case when shale material is ground up / pulverized and possibly reconstituted (e.g. pressed into pellets that will fit certain types of test apparatus). Again, inspection of Figure 1 immediately makes clear that removing cementation will release the hold on the swelling pressure, such that swelling effects during shale-fluid compatibility testing may become exaggerated. This swelling artifact becomes magnified even further when ground-up shale is tested at atmospheric pressure conditions, when there is no restriction whatsoever on the swelling pressure and unbridled clay hydration may occur.

Finally, let us take a look at the effects of poor sample preservation and dehydration of shale samples. The importance of good sample preservation has been stressed before (see e.g. Chenevert, 2001), with emphasis on obtaining a representative shale water activity. When shales dehydrate, water evaporates from the pore spaces, which are then filled with air, as shown in Figure 2. Upon exposing the dried out shale to a test fluid during a shale-fluid compatibility test, the test fluid will invade the pore space, which in turn may trap the air inside of it. The migration of fluid into the pore space is governed by several factors, including the viscosity of the invading fluid, the wetting preference of the clay surfaces, the tortuosity of the pore space network, etc. If air pressure is not able to escape, it will build up within the shale and ultimately reach such a high value that it literally blows the shale apart from the inside out. If this happens during a swelling test, the result will be recorded as a strong "swelling" effect in response to the applied fluid; this may then be interpreted as evident shale-fluid incompatibility. This test artifact can lead to highly misleading results, including observations where fresh water appears to be more inhibitive (i.e. control shale swelling better) than salt solutions.

An instructive example of this artifact, which readers can easily verify for themselves, involves Pierre Type I shale. Well-preserved Pierre Type I is low-reactivity shale that is perfectly stable in tap water, and plugs can be stored almost indefinitely in it. However, if a plug has been left out to dry for a day or so and is re-inserted in water, it will fall apart violently, presumably under the influence of rapid "swelling". Of course, the dehydration of the plug does not magically sensitize the shale to water: the swelling and disintegration is entirely due to entrapment of air pressure upon re-wetting of the sample and its consequences.

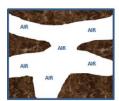






Figure 2 – Graphical representation of core dehydration artifact in shale testing. Left – pore space of poorly preserved, dehydrated shale sample contains air. Middle – air is entrapped and compressed by invading fluid during shale testing. Right – air pressure rises to the point where it blows apart the shale fabric from the inside out. None if this is representative of true shale-fluid interaction behavior, but is often mistaken for it. Test results are invalidated by the occurrence of this artifact.

When selecting a suitable test protocol as well as shale material for shale-fluid compatibility testing, it is essential to address the following three questions and use them as guiding principles in order to obtain meaningful, non-misleading results without artifacts:

1. Is the shale material in a properly preserved state that is representative of its downhole state? As indicated above, if the shale is dried out and / or ground up, this will lead to test artifacts.

- 2. Are the test conditions such that they represent the downhole environment properly? As indicated above, if all confining stress and/or cementation is removed, this will remove the "hold" on the swelling pressure and exacerbate swelling effects during the test.
- 3. What aspects of shale-fluid interaction does the test reflect on, and what conclusions can and cannot be drawn from the test results for field applications?

To clarify point (3), it is important to make a distinction between shale cuttings stability vs. shale borehole stability. These are NOT the same. A detailed discussion has been given elsewhere (Bol et al., 1994; van Oort, 2003), but the essence is that cuttings stability revolves around controlling (the effects of) the swelling pressure. This can be achieved with inhibitive salts (e.g. KCl) and organic compounds (e.g. quaternary amines), which may favorably exchange at clay surface sites to lower hydration/solvation forces, as well as certain polymers (e.g. polyamines) with chemically active groups that can bind onto shale surfaces and temporarily prevent them from disintegrating. Borehole stability revolves primarily around application of the right mud weight (more accurately: maintaining the right dynamic downhole pressure) to prevent mechanical failure: if the wrong mud weight / downhole pressure is applied, immediate borehole caving will occur, irrespective of mud type or composition. Once the correct mud weight is established, however, instability over time may still occur if mud pressure can diffuse into the near-wellbore zone and raise near-wellbore pore pressure. This is usually avoided in OBM / SBM due to capillary forces (but may occur in (micro-) fractured shales where such forces are absent) but does occur when WBMs are exposed to low permeability shales at overbalance. The increase in pore pressure over time reduces the near-wellbore effective stresses, driving the stress state toward failure, as shown in Figure 3.

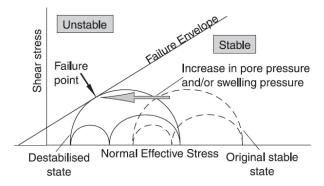


Figure 3 – Mohr-Coulomb representation of shale failure over time. An initially stable stress state with correct mud weight application moves towards the failure envelope when pore pressure or swelling pressure is increased. Mud pressure diffusion in the near-wellbore zone drives the pore pressure increase, which reduces the effective normal stresses (whereas the shear stresses remain unaffected). This shale destabilizing mechanism is not represented by simple swelling / dispersion tests, and requires more realistic downhole testing including pressure transmission and borehole collapse tests.

The key to point (3) is that not all shale-fluid interaction tests reflect on cuttings and borehole stability equally. Shale swelling and disintegration tests, for instance, are relevant primarily to cuttings stability, but have very limited value for borehole stability evaluation. The latter requires more representative downhole testing, such as pressure transmission tests and borehole collapse tests, as discussed below. It would be a mistake to deduce borehole stability information from simple shale swelling tests, but this is done often, unfortunately. Case in point: conventional inhibitive muds, such as KCl/polymer muds, usually perform well in swelling tests, but they provide no safeguard against mud pressure invasion and effective stress reduction, and may thereby cause borehole instability over time. The latest generation of high performance water-based mud was developed with the guidance derived not only from swelling tests, but also from more sophisticated testing such as pressure transmission and borehole collapse testing. Test best practices include:

- Always use representative shale material (such as cuttings, cavings, whole core) that has not been altered.
   Material that has been ground-up and/or reconstituted should not be used for shale-fluid compatibility testing (although it may be suitable for shale characterization testing, such as XRD analysis, see below).
- Always use shale material that is well-preserved and has not been allowed to dry out, or that has been properly reconditioned (e.g. using desiccators to re-establish its native water activity) after drying out.
- Always attempt to recreate the downhole environment experienced by the shale when it is interacting with a certain fluid as best as possible. Swelling tests should be conducted in the presence of confining pressure, and never be done at atmospheric conditions.
- Always be aware of what information and guidance, relevant to either cuttings stability, borehole stability or other shale-fluid interaction phenomenon, the test result can – and cannot – deliver. The limitations of a particular test may be more important than its merits.
- Ease of testing and favorable test costs should be of secondary concern only, and looked at holistically. The wrong advice based on the flawed results from cheap tests may be inexpensive in direct cost, but can have a very high negative holistic cost impact when implemented in the field (e.g. selecting a brine formulation for EOR water injection that may be incompatible with the shale zones in the flooded reservoir, see e.g. Russel et al, 2007).

#### **Conventional Test Review**

A recent AADE paper by Stephens et al. (2009) recommends atmospheric swelling tests, dispersion tests and capillary suction tests for examining high reactivity shales, dispersion tests, bulk hardness tests and immersion test for moderate reactive shale, and fracture development tests for low reactivity shale. We will review several of these tests in the following and assess their suitability for shale testing.

# CST testing

The capillary suction time tests measures the travel time of a shale/clay slurry across a thick porous filter paper (Wilcox et al., 1987). This requires grinding up the shale sample and shearing it into solution, creating a colloidal suspension. The dispersion of the shale will affect its cementation, and the test is carried out without confining pressure. Both will exacerbate clay swelling tendencies, and it should come as no surprise that inhibitive salts that can favorably exchange at clay sites and flocculate the clays do particularly well in this test, particularly for shales with higher CEC and MBT. The test furthermore introduces significant unknowns and unrealistic factors, such as the interaction between the fluid-colloid system and the filter paper. An extensive CST study (Hart, 1989) found the results of CST tests to be strongly dependent on shearing history with poor reproducibility. The appeal of the test, of course, is its ease of execution and low cost. However, since its results may at best be only tangentially relevant to shale inhibition and cuttings stability, and at worst provide grossly misleading results, this test is best avoided altogether for shale-fluid interaction characterization purposes.

## **Atmospheric Swelling Testing**

The atmospheric linear swelling test continues to be very popular, but clear drawbacks to it exist. Shale material, even when intact plugs are used, is usually dry / poorly preserved and may have significantly changed by grinding / pulverizing to create fine material for pellets that are suitable for linear swell meters. This removes almost all of the cementation between the clay fabric that holds the material together and gives it its strength. Tests are done under atmospheric conditions (i.e. atmospheric pore and confining pressure, temperature may be varied). Both tend to exacerbate swelling phenomena, as described above. Artifact susceptibility is particularly high for these tests, and relates primarily to trapping of air pockets inside the shale upon wetting the dry material with a test fluid, as described above. This can lead to highly puzzling results, such as freshwater showing better test responses than inhibitive KCl solutions (see e.g. Gomez and Patel, 2013 – Figure 16). A significant improvement would be to execute the test on well-preserved core material in the presence of an external load, as is done in an oedometer test where swelling is quantified during an unloading cycle (Bol, 1986), as described below. The appeal of the atmospheric swelling test is, like the CST, its relative ease and low cost, but its results and limitation need to be placed in the proper perspective in order to avoid erroneous field guidance on mud system formulation, selection and application.

Fracture development tests and immersion tests of shale exposed to fluids are essentially variations of the atmospheric swelling test, with emphasis on recording (e.g. using time-lapse photography) and documenting fracture development, changes to microstructure, and disintegration of the shale material. They also suffer from the same artifacts as the atmospheric swelling test, being highly sensitive to sample preservation and the lack of external confining pressure. Caution is advised when using the results they provide.

## **Bulk Hardness Testing**

In the Bulk hardness test, sized shale fragments (derived from cuttings, cavings or core) are hot-rolled in test fluid, typically for 16 hours at 150°F. After hot-rolling, the shale pieces are retained on a 50 mesh sieve and placed in a torque wrench where they are extruded on a perforated plate. The observed torque reading, its maximum value and behavior during the extrusion process, is a qualitative measure of the hardness of the intact shale.

Bulk hardness testing may have an application as an extension to cuttings dispersion testing (see below) to aid in assessing cuttings stability, but it is important to realize that its readings may be influenced by sample preservation and the lack of confining pressure during the hot-rolling cycle.

# **Recommended Test Methods**

# Shale / Clay Characterization Tests

In the following, we distinguish between fluid-shale compatibility testing and shale / clay characterization. Typical characterization tests that may be of use include:

- X-Ray Diffraction (XRD) mineralogy
- Cation Exchange Capacity (CEC)
- Methylene Blue Test (MBT)
- Thin Section Analysis
- Scanning Electron Microscopy (SEM)
- Shale Water Activity Determination

Care should be taken when attributing value and relevance to these characterization tests for shale-fluid compatibility evaluation. All of these measurements reflect only indirectly on the compatibility – or lack thereof – of fluids with shales. For instance, the presence in the shale of certain amounts of high surface area clays such as smectites, as shown by XRD data, should not lead to the automatic assessment that the shale will be highly reactive in a water-based environment without properly assessing e.g. the level of cementation within the shale matrix. Also, sample preparation may modify the material and associated test readings: grinding up shale material for CEC and MBT tests may remove cementation and expose surface area that would normally not be readily accessible within the shale under in-situ conditions. Paraphrasing point (3) above, the question to answer is: what aspects of shale-fluid interaction does a particular shale / clay characterization test reflect on, and what conclusions can - and cannot - be drawn from the test results for field applications?

# **Cuttings Stability & Bit Balling Tests**

To investigate fluids for cuttings stability and bit balling behavior, three types of tests are recommended:

- Accretion tests
- Cuttings dispersion tests
- Oedometer Swelling Test

Each of these tests is briefly described in the following.

### Accretion Test

The accretion test reflects on the tendency of shale/clay to adhere to steel surfaces. It thus provides a qualitative indicator of the likelihood of experiencing bit-balling and ROP reduction problems in the field. In the test, the degree of shale/clay adherence onto a steel bar is measured during a hot-rolling test. The test protocol is given elsewhere (see e.g. van Oort et al., 2015). On the positive side, the test uses actual shale cuttings and can be carried out at elevated temperatures, representative of downhole conditions. On the negative side, tests are typically carried out at atmospheric pressures (but could in theory be performed in pressurized cells). Even though the accretion test is a crude and unsophisticated tool, it does quantify the tendency of mud systems to cause accretion, and its results generally extrapolate well to field practice.

# Cuttings Dispersion Test

The cuttings dispersion test is a hot-rolling test carried out with shale/clay cuttings/fragments. The retention percentage is measured after hot-rolling the cuttings for a specific amount of time at a test temperature, which can be varied. Like the accretion test, the merits of the test consist of the fact that actual shale cuttings can be used and tests can be carried out at elevated temperatures. On the downside, tests are typically carried out at atmospheric pressures (but could in theory be performed in pressurized cells). Moreover, well-preserved cuttings should be used to prevent test artifacts. The cuttings dispersion test is also sensitive to the viscosity of the test fluid (Hale, 1991): the lower the viscosity, the higher the mechanical erosion rate during the test (i.e. less "cushioning" of the cuttings while hot-rolling). It is therefore important that test fluids are of similar viscosity when running comparative tests. Even though its results are only qualitative, the cuttings dispersion has proven itself useful particularly when screening different fluid formulation for their ability to stabilize cuttings in comparative fluid evaluations.

### Oedometer Swelling Test

A considerable improvement to the traditional atmospheric swelling test is to carry out swelling investigations in the presence of an applied load. This can be accomplished in the oedometer test (see e.g. Bol, 1986), where swelling of a shale/clay sample is measured in the presence of an applied uniaxial load during an unloading cycle. The swelling tendency in this test, derived from soil mechanics practice, is characterized by a swelling index  $C_S$  as:

$$C_S = \frac{-\Delta e}{\Delta log\sigma} \tag{1}$$

Where e is void ratio (pore volume divided by grain volume) and  $\sigma$  is the applied load. The test protocol is discussed in detail elsewhere (Bol, 1986). The test is typically carried out on pulverized soil material, but can also be carried out on intact shale samples Again, in order to get the most representative test result, it is important to use well-preserved shale/clay material.

## **Borehole Stability Tests**

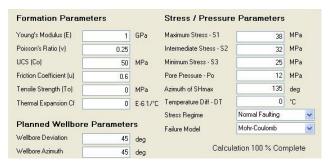
As discussed previously, it would be a mistake to draw significant conclusions for borehole stability from the above-mentioned tests. For wellbore stability assessment, three types of tests are recommended:

- Triaxial failure tests
- Pressure transmission tests (PTT)
- Modified Thick-Walled Cylinder tests (TWC) with drilling fluid exposure

Each of these tests is briefly described in the following.

#### Triaxial Failure Test

Triaxial failure tests are the most common way to characterize the failure envelope shown in Figure 3, which represents the rock strength and failure behavior at depth. Triaxial tests (which should really be called bi-axial tests, as only two of the stresses applied to the sample are truly independent) measure the failure of a radially confined cylindrical shale/clay sample while ramping up the applied axial stress. Repeated failure measurements on representative shale plugs at different confining pressures will map out the failure envelope. When the failure envelope is linearized (as in Figure 3), it can be characterized with two independent parameters, the cohesion S<sub>0</sub> and coefficient of internal friction  $\mu_i$ . It is preferred to carry out the test in a drained rather than undrained mode, but this may lead to excessive test times given the low permeability of most shales. Triaxial failure tests on well-preserved core material are the most direct way to determine the required mud weight for wellbore stability, and this is its main purposes. Figure 4 shows a typical assessment of optimum mud weight based on information provided by triaxial tests.



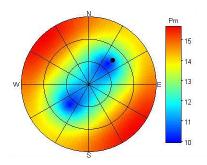


Figure 4 pressure P<sub>m</sub> (in MPa, right) as a function of deviation borehole (circles at 0°, 30°, 60°, 90° deviation from inside out) azimuth for a given set of well, pore pressure, stress and rock strength / failure parameters (above). the latter derived from triaxial failure testing.

#### Pressure Transmission Test

Even when the correct mud weight is applied, wellbore instability may still occur with time. The pressure transmission test measures the tendency of a mud's filtrate, applied at overbalance pressure, to invade the shale fabric and elevate the near wellbore pore pressure (van Oort, 1994; van Oort et al., 1996). This "mud pressure penetration" effect can be an important cause of time-delayed shale failure: as nearwellbore pore-pressure goes up, the effective stress state moves progressively towards the failure envelope until at some point in time shear failure will occur. Mud pressure penetration is normally not a concern in OBM/SBM systems, because capillary entry pressures are usually high enough to restrict entry by OBM/SBM filtrate; this, however, may not be the case when the shale is (micro-)fractured. Mud pressure penetration may be an issue in WBM / HP-WBM systems, and is in fact the main cause of wellbore instability in shales when using these fluid systems.

Figure 5 shows a typical PTT set-up. The test protocol and data analysis procedure has been described elsewhere (van Oort, 1994). Downstream pressure build-up behavior due to pressure diffusion through the shale sample is similar to the charging of a capacitor in a RC circuit, and is given by:

$$\frac{P(l,t) - P_o}{P_m - P_o} = 1 - \exp\left[-\frac{Akt}{\mu\beta Vl}\right] \tag{2}$$

where

 $P_o$ = initial pore pressure (Pa), = upstream fluid pressure (Pa),  $P_{m}$ = downstream pressure at sample end (Pa) P(l,t)= sample length (m)  $\boldsymbol{A}$ = sample cross-sectional area (m<sup>2</sup>) = volume of downstream reservoir (m³) β = fluid compressibility (Pa-1) = fluid viscosity (Pa.s) μ k = relative shale permeability (m<sup>2</sup>)

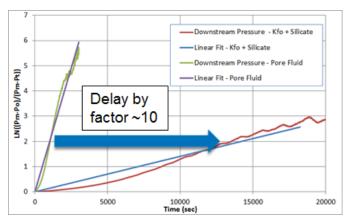
Tests are typically performed with two distinct cycles: a first cycle using pore fluid, to characterize rock permeability, and a second cycle (after re-equilibrating the rock sample to initial conditions) with test fluid. Since the viscosity  $\mu$  and compressibility  $\beta$  of the filtrate of the test mud are generally unknown, a hydraulic conductivity  $k/\mu\beta~(m^2/s)$  is characterized for each pore fluid cycle and subsequent test fluid cycle. These are compared to yield a "delay factor" given by:

$$Delay Factor = \frac{Hydraulic Conductivity (Test Mud)}{Hydraulic Conductivity (Pore Fluid)} (3)$$

The delay factor shows the delay in the rate of mud pressure invasion and pore pressure elevation that is expected for a particular fluid system. This delay factor is directly related to trouble-free open hole time, as it indicates by how much the dynamics of the shale destabilizing pressure invasion can be slowed down. An overview of delay factors for novel high-performance WBM systems is given in Table 1.



Figure 5 - (right) Photograph equipment in a of PTT controlled temperature environment, showing sample core holder with an upstream downstream and fluid that allows reservoir transmission pressure through a cylindrical shale sample to be measured; (below) example of test result, with downstream pressure data processed in accordance with Eq.(2) and fitted with a least-squares linear fit. From the slope of the fitted lines obtained for the pore fluid (first) cycle and the test fluid (second) cycle, a delay factor can be characterized according to Eq.(3). The delay factor is a quantitative measure for the available trouble-free open hole time expected with the test fluid in the shale borehole.



#### Modified Thick Walled Cylinder Test

The downhole simulation cell (DSC) test (Simpson et al., 1989; Salisbury et al., 1991) is by many, including the authors of this paper, regarded as a "gold standard" for shale borehole stability testing. The main concern with the DSC test is that it requires large, well-preserved core samples and large-scale equipment for drilling these samples and exposing them to various loads. These requirements make the test expensive, labor-intensive and less suitable for quick screening of fluid formulations. In this paper, we are introducing a less sophisticated version of the DSC test based on modification of the thick walled cylinder (TWC) test. For this, a modified triaxial load frame can be used. A photograph and schematic of the test set-up is given in Figure 6. The test protocol for this test is given in the Appendix.

Whereas the PTT test investigates the dynamics of mud filtrate invasion, the modified TWC test actually looks at the failure behavior upon exposure to drilling fluid applied at overbalance. Shale failure behavior is probed after exposing a cylindrical shale sample with a borehole under hydrostatic compression confinement to a test fluid at overbalance for a

12-24 hour period. Failure is triggered by ramping up the confining pressure after test fluid exposure and observing the pressure as which failure occurs using volumetric strain measurements. A sudden increase in volumetric strain during the ramp-up of the confining pressure indicates borehole collapse. A representative example is shown in Figure 7. The difference in shale (de-)stabilizing effects between various fluids systems manifests itself as a difference in the measured confining pressure at failure.



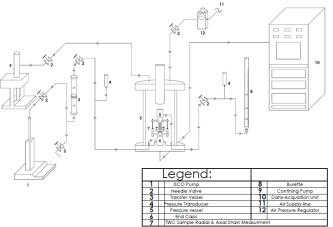
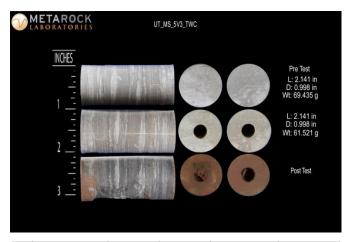


Figure 6 – (top) Photograph of TWC equipment, based on a modification of a triaxial load frame; (bottom) Schematic of TWC setup.

As part of a larger investigation into high-performance WBM formulations, a series of PTT and modified TWC was conducted. The results are given in Table 1. A series of fluids was tested that included nano-particle (NP) formulations, formulations based on 25% potassium formate (KFo) solutions with additional osmotic membrane enhancers (such as polyglycol, polyglycerol and salt-tolerant silicate, see van Oort



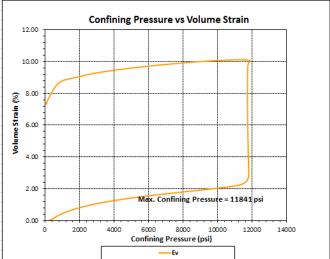


Figure 7 – (top) Photographs of cylindrical Mancos shale samples (1" diameter, 2" length) before coring the 0.3" borehole, before TWC testing, and after TWC testing when collapse has occurred; (bottom) behavior of volumetric strain during a TWC test as a function of ramping up the confining pressure; a sudden increase in volumetric strain signals borehole collapse (occurring at a maximum confining pressure of 11.831 psi for this example).

et al., 1995), as well as OBM and high-performance WBM (HP-WBM) formulations used in the field, all tested on Mancos shale. The PTT delay factors and TWC collapse pressures that were obtained for these fluids are cross-plotted in Figure 8. There was good correlation between higher delay factors observed in the PTT series and higher collapse pressures observed in the TWC test series. Note that this is fully consistent with the notion that pressure transmission is the main mechanism causing borehole destabilization and failure. Figure 8 shows that the observed collapse pressures asymptotically approach ~11,500 psi (~80 MPa), which is the native TWC strength of the Mancos shale when it is not exposed to any fluids. Particularly good results were obtained with a particular type of nano-particle solution (NP1 - but note that not all nano-particle fluids performed as well as NP1), as well as with potassium formate solutions with added membrane enhancers. The detailed discussion of these results is postponed to future reports.

Table 1 – PTT delay factors and TWC collapse pressures at failure
from a dedicated series of experiments using nano-particle (NPx,
x = 1,2,3) fluids, potassium formate (KFo) fluids, as well as OBM
and HP-WBM formulations used in the field.

	PTT Delay	TWC Collapse
Fluid Type	Factor	Pressure (psi)
NP1	3.2	11,841
KFo + Silicate	10.3	11,521
KFo + Polyglycol	4.2	11,149
HP-WBM1	4.6	10,055
OBM	3.4	9,953
KFo + Polyglycerol	4.5	9,503
HP-WBM2	2.0	9,321
NP2	2.0	9,240
NP3	1.3	9,108

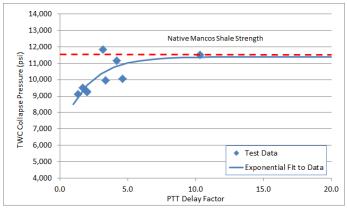


Figure 8 – Cross-plot of delay factors obtained in PTT tests and borehole collapse pressures obtained in TWC tests (Table 1). Data fit is an exponential rise to a limit: TWC = 6576 + 4814(1-e<sup>-0.51\*PTT</sup>).

### Miscellaneous Shale-Fluid Tests

The tests recommended and described above are specific to the evaluation of cuttings stability, bit-balling and borehole stability. There are, of course, other shale-fluid interactions to consider, particularly those relevant to lubricity and friction reduction, and the behavior of clays/shales in hydrocarbon reservoirs when contacted by fluids e.g. during waterflooding. The guidelines outlined in the above apply equally when selecting suitable test protocols here. For instance, for steelon-shale lubricity tests it is important that well-preserved shale material is used and that tests are performed at realistic contact loads that properly reflect the interaction of drillstring tooljoints and shale formations. Most small-scale lubricity test devices unfortunately do not provide this, which can result in unrealistic results and misleading guidance. Likewise, when probing for the effects of clay swelling and fines migration during waterflooding and enhanced oil recovery, it is best to obtain realistic guidance from representative coreflood and return permeability tests, even if this means having to invest in obtaining a reservoir core. This approach is superior to trying to infer shale behavior from indirect and possibly very misleading tests such as CST and atmospheric swelling tests.

#### **Conclusions**

This paper provides guidelines and criteria for shale –fluid compatibility testing, identifying issues associated with sample preservation, dehydration and lack of confining pressure that plague conventional shale-fluid test protocols, such as those used in CST and atmospheric swelling tests. Despite the fact that these tests are low cost and easy to carry out, their results can be erroneous and misleading. Guidance derived from them can have far-reaching negative consequences and cost-implications when implemented in field practice. A case is made in favor of abandoning them altogether (or at least using their outcomes with utmost caution).

As an alternative, specific shale-fluid test sets are recommended for cuttings stability and bit-balling (including accretion test, cuttings dispersion test, oedometer swelling test) and for borehole stability (triaxial failure test, pressure transmission test, modified thick-walled cylinder test). The modified thick-walled cylinder test, a scaled-down version of the downhole simulation test that probes the collapse behavior of shale when exposed to fluids, is described in detail here.

A series of pressure transmission and modified thick-walled tests was carried out on a variety of fluids including nano-particle formulations and potassium formate solutions with membrane builders, as well as WBM/HP-WBM and OBM formulations used in the field. The results show clear correlation between pressure transmission delay factors and borehole collapse pressures, indicating that pressure transmission (and not swelling) is the lead cause of time-delayed borehole instability, even when mud pressure requirements for mechanical wellbore stability are satisfied.

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### **Nomenclature**

AADE = American Association of Drilling Engineers

CEC = Cation Exchange Capacity
CST = Capillary Suction Test
DSC = Downhole Simulation Cell

HPWBM = High Performance Water-Based Mud

KFo = Potassium Formate
MBT = Methylene Blue Test
NP, NPx = Nano-Particle Fluid
OBM = Oil Based Mud

PTT = Pressure Transmission Test SBM = Synthetic Based Mud

SEM = Scanning Electron Microscopy TWC = Thick Walled Cylinder Test

 $WBM = Water\ Based\ Mud$  $XRD = X-Ray\ Diffraction$ 

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### Appendix

The modified TWC test procedure is as follows:

- A sample of 1" in diameter, 2" in length (i.e. length-to-diameter ratio of 2) is cut from representative, well-preserved shale material that does not show noticeable cracks. A 0.3" centralized borehole is drilled along the entire length of the sample. The sample is photographed in axial and radial directions before and after the borehole is drilled, prior to testing. Care should be taken not to allow the sample to dry out during this phase.
- A Viton sleeve is measured to the size of the particular sample, and fitted around it. An endcap is placed at the bottom of the sample. Utilizing a heat gun, the bottom of the sleeve is heated around the endcap.
- A cantilever bridge is placed around the sample.
   Additionally, a band and curved brass spacers are placed around it to help hold it in place. This will also assist in measuring the radial strain of the sample
- The top endcap is placed on the sample. This also aids in ensuring the sleeve will hold in place and everything will attach more effectively
- Internal LVDT's measuring axial strain are attached to the end caps.
- The top of the sleeve is heated up around the sample. The sample is then ready to be loaded into the triaxial test cell.
- Fluid lines (for pore/confining pressure) and measurement channels are connected and brought online as necessary.
- The sample is saturated at a pore pressure value (typically 50-200 psi) with synthetic pore fluid in the borehole for a period of 3-4 hours. The synthetic pore fluid is based on water analysis from pore water e.g. obtained during a prior shale squeeze test. This saturation step is essential to ensure that samples are fully saturated when tested with test fluid: (partial) dehydration of the sample would lead to significant test artifacts. Once stabilization occurs the pore pressure is dropped to 0 psi and the brine is flushed from the borehole.
- The borehole is now displaced to a drilling fluid test formulation and borehole pressure is held constant at overbalance (typically 200 psi) for a period in the range of 12-24 hours (note that longer time periods can be applied, as necessary/desirable).
- After 12-24 hours, confining stress around the sample is increased at 1 µstrain/sec until the point of failure. Expelled volume and strains are monitored throughout the test. A sudden increase in volumetric strain indicates borehole failure. The confining pressure at failure is reported as shale borehole strength upon fluid exposure.
- The sample is then unloaded and the sleeve is removed. The sample is subsequently photographed in axial and radial directions to document the sample failure mode.
- The failed sample is safely stored in a storage unit for tested samples where an accurate inventory is kept. This allows for any further post-mortem inspection, if necessary/desirable.