Causal protocols to assess the viability of native state or restored state preparation

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Abstract. Wettability is the fundamental attribute controlling reservoir rock-fluid properties such as capillary pressure and relative permeability. It is essential that a core study wettability represents the reservoir wettability and thus, one must decide whether native state or restored state analysis should be employed. There remains debate in our industry regarding which of these conditions should be more representative: some preferring native state (as recommended by Anderson's pivotal literature survey), whilst others favour the control and repeatability of restored state procedures. Recent increased use of wettability-altering mud additives, such as; asphaltic bridging agents, emulsifying agents, surfactants, etc., does not automatically preclude native state analysis, but inevitably complicates the process. Some consider native state analysis to be a simpler experimental method, assuming representative conditions and directly employing "as-received" core samples. However, "as-received" wettability and saturation may be altered during coring, wellsite core handling and laboratory processes, thus preventing native state analyses. If determining to employ native state core analysis, it is paramount, at an early stage in the program, to assess the impact of possible changes during coring through to laboratory processes; considering aspects such as: core damage, potential invasion of mud additives, saturation hysteresis, compositional change of the reservoir fluids, experimental conditions, laboratory methodologies, etc. In this paper, we show that rigorous native and restored state processes can give equally viable data and provide a suggested decision tree to guide considerations regarding the use of native or restored state analyses.

1 Introduction

Wettability is a fundamental attribute controlling reservoir rock-fluid properties such as capillary pressure and relative permeability [1-8]. Representative reservoir wettability is thus essential when performing most multi-fluid special core analysis experiments, such as: wettability, capillary pressure, core resistivity, relative permeability, etc. There are three main test states commonly used to represent wetting conditions for advanced core analyses: native (or alternatives), clean, and restored state. It is essential to be able to perform analyses under either one or all these conditions and understand when each may best represent the reservoir being studied.

It is important, early in this paper, to define "native state", because there are a few different terms with similar but slightly variant meaning: fresh, native and "as-received". This paper will employ the term "native" to describe the general state where it is deemed that the innate reservoir wettability has been sufficiently maintained in the core plugs until the point of testing, as defined by Anderson [3].

Anderson [3] provides more information about native state coring, core packing and preservation. He recognised, from the coring procedures and fluids of the time, that the biggest challenge to native state, without altering wettability or saturation, was during the trip to surface. The changes in pressure and temperature during the process of bringing core to surface can alter the composition and saturation of pore filling fluids: light-end oil components (gas) can liberate from the oil phase causing alteration of the reservoir oil composition; gas components may be lost from the system; gas movement will cause saturation redistribution, potentially of both water and oil, leading to uncertainties in the composition, saturation and rock-fluid contacts of pore filling fluids; heavy oil components (asphaltenes) may deposit; waxes may form; salt may precipitate from the water; solubility of wettability altering components may be changed, leading to both fluid-fluid and fluid-rock reassociations of those chemicals. In summary, any of these possible changes may alter authentic wettability.

There thus remains some debate on whether native state or restored state best represents original reservoir wettability, especially for liquid hydrocarbon reservoirs.

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Native state core material, fundamentally, must maintain the original (native), yet most often unknown, reservoir wettability. It also should maintain fluid saturations from the reservoir depth to surface to laboratory, and ensure that those fluids are free from precipitates and/or contaminants, since these would be non-native and may influence results. In reality, change is difficult to avoid and many, if not all, fluids within received cores may have been altered.

Results of several cleaning pre-studies performed in recent years, indicated that oil-based mud invasion had irreversibly altered wettability, creating strongly oil-wet core material. Cleaning did not render these plugs water-wet (see Figure 1). Many samples continued to exhibit strong oil wettability, even after employing alternative cleaning methods and solvent mixes. For some cases where water-wet state was achieved, the core mineralogy and pore structure were altered or damaged by the cleaning/drying process and became unrepresentative of reservoir petrophysical properties. Consequently, there may be high uncertainty of achieving representative reservoir wettability by later restoration ageing protocols and neither native nor restored state analyses would be viable. So, to minimise contamination and alteration, it is strongly advocated to use low invasion protocols for all coring operations. **Note**: Some rocks contain structural organic matter such as coal or pyrobitumen, and would not be expected to achieve water-wetness. Thus, rock compositional analysis is a vital step to aid understanding of the wetting complex to support design of appropriate preparation procedures.

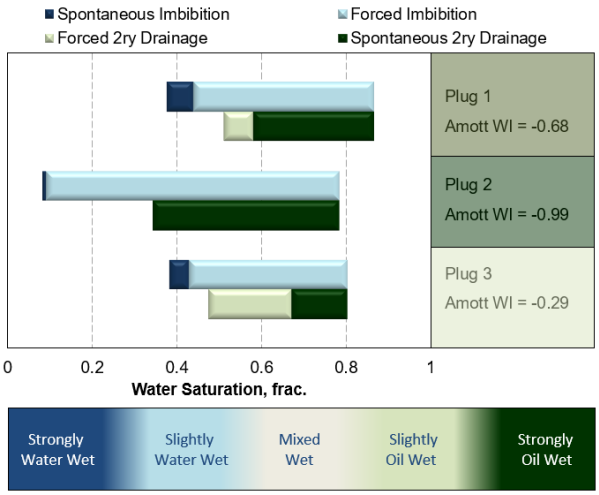

Figure 1. Clean state plugs showing oil-wetness (Mud-invaded)

A summary of wettability data from a North African Field is shown in Figure 2 - Amott wetting index versus permeability. Restored state (RS) plugs exhibited mixed wet results across the entire permeability range (ca. 100 to 2000 mD). The native state (NS) plugs with a similar permeability range, showed similar wetting indices, except a low permeability outlier (20 mD). The invaded plugs (INV) exhibit evidently more oil-wet results.

A low invasion coring program can be designed to reduce some of the changes by controlling the trip to surface rate thus minimising gas liberation rates and excessive expulsion of fluids, but generally, some alteration is unavoidable. Therefore, it is not a given that "as-received" samples may be considered to represent the native reservoir wettability, saturation or properties. If either wettability has altered or fluid saturations have changed, the core cannot be considered native state, nor fresh state, and using such samples as-received, could produce incorrect and misleading results.

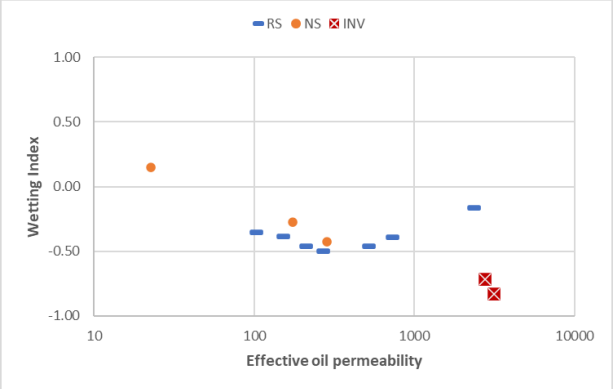

Figure 2: Amott wetting index as a function of permeability

As a case in point, Table 1 shows data from a core plug used for an as-received wettability study, with no pre-test preparation. The core had been acquired using water-based mud (WBM). Initially, Amott wettability results suggested a slightly oil-wet nature (Amott index = -0.121) but upon back-calculating initial water saturation (Swi = 0.557) from final saturation checks and production volumes, it was realised that it did not compare to other saturation datasets (capillary pressure and well log data). After cleaning and establishing representative Swi using relevant capillary pressure (Pc), from height above free water level (HAFWL) data, a corrected Amott index was calculated to be +0.253 (slightly water-wet). It was concluded that 32.3 saturation units (s.u.) of water mud filtrate had spontaneously imbibed before laboratory tests were initiated; during coring, wellsite core handling and shipment to the laboratory. This implies that core may not be merely used as-received but, to regard received core as native state, requires consideration of potential fluid changes and may need procedures to check and verify that connate saturations are present or can be re-established without significant hysteresis.

Table 1. Potential error in assuming as-received = native

Stage	As-Received	Corrected
Swi	0.557	0.234
Amott Imbibition Sw	0.634	0.634
Centrifuge Imbibition Swf	0.746	0.746
Spontaneous imbibition	0.077	0.4
Forced imbibition	0.112	0.112
Iw	0.407	0.781
Amott 2nd Drainage Sw	0.523	0.523
Centrifuge 2nd Drainage Swr	0.324	0.324
Spontaneous 2nd drainage	0.223	0.223
Forced 2nd drainage	0.199	0.199
Io	0.528	0.528
Amott Index	-0.121	0.253

Subsequently, the main requirements for ensuring representative native state core plugs would be:

unaltered wettability, unchanged saturation, no contamination and no change to fluid composition.

In contrast, Anderson [3] describes restored state core as a three-step process: cleaning, saturation with brine then oil, and ageing. Restored state procedures must: remove the connate fluids, precipitated solids and any invaded contaminants, whilst maintaining sample integrity (particularly clay/mineral structures) and without altering the capacity of pore-lining surfaces to associate with the correct fluid components that were involved in the in-situ wettability. The restoration process must then replace fluids: first, with a representative formation water, then re-introducing hydrocarbon to re-establish correct connate saturations at the equivalent pressure for HAFWL, and finally, restore the original reservoir wettability by ageing with representative reservoir oil under correct reservoir pressure and temperature. This assumes that the reservoir history is mimicked in this laboratory processes. So, designing a suitable procedure will benefit significantly from cross-disciplinary discussions to understand the history of the reservoir in question. General reservoir theory considers most reservoirs to have formed by deposition in a water-filled environment, thus being water-wet prior to the migration of oil into the system (primary drainage). Hence, it is largely accepted that cleaning should create water-wet conditions, except where naturally oil-wet matrix components may be present, e.g. organic material, chamosite, halite, etc. (note: this list is not exhaustive).

Preparation criteria are also important for digital rock properties (DRP), particularly when performing multi-phase corefloods on micro-plugs. Lin et al [9] stated the need for best practice preparation to initiate wettability, recommending a centrifuge method for establishing initial water saturation in a restored state protocol. Also, it must be noted that information gained from DRP show wettability to be more complex than is commonly understood, variable within a pore space and thus, a core plug merely captures an average of all the varying wetting states with the plug porosity.

2 Coring and Wellsite Core Handling

2.1 Native Wettability

It is well documented and accepted that oil-based mud (OBM) filtrates can alter wettability upon invasion into the reservoir pore system [2,10,11,12]. However, employing a low invasion OBM coring procedure could enable core acquisition with minimal or limited invasion [7,13,14,15]. Invasion is controlled by aspects like coring overbalance pressure, bit geometry, coring speed, mud circulation velocity, trip to surface rate, mud composition and rheological properties, petrophysical properties, reservoir fluids properties and compositions, reservoir wettability, initial fluids saturations, chemical concentration gradient and fluid connectivity to promote chemical diffusion. The key elements of low invasion coring deliberate all these parameters and set protocols to optimise as many as possible.

Success can only be known by determining the level of filtrate invasion in the core, which is most commonly accomplished by adding tracer chemicals into the mud filtrate and measuring for their presence within the core plugs to be used in native state testing, or within offcut trim sub-samples. Some authors suggest that mud chemicals may be used as a tracer when no specific tracer additive was used during coring. If low invasion is successful, the core centre will contain filtrate saturation below the detection limit of the tracer in most samples. This uninvaded centre is expected to be large enough to obtain a 1.5” diameter core plug.

SCA 2014-094 [16] highlighted the need for coring mud properties and components to be available to all involved in the core analysis program. It suggests that the base oil of an OBM or certain salts in the water phase of a WBM, may be used as tracing agents in lieu of traced additives. Glycols, which are often added to WBM, can dissolve both in oil and water phases, and can be easily detected in the oil phase. Also this mud type often has large quantities of salts (usually KCl) which could be analysed.

SCA 2016-32 [17] used centrifugation to extract oil from the core plugs. It demonstrated that properties of effluent oil can be different from those of oil remaining in the core: polar components and asphaltenes seem to be retained in the core, perhaps because these components, deemed to be significant in wetting, could adhere to core surfaces. This is aligned with published data within the geochemical community, Bennett et al [18], which also observed that mud filtrate appeared to be more easily removed than the in-situ crude oil. The work also demonstrated that mud components could be used as “natural” tracers, potentially eliminating the need to add specific tracers. Besides reducing cost, this approach has the following possible advantages:

- works for small fluid samples,
- can be acquired from cores of any shape,
- both oil and water samples may be analysed,
- preliminary identification of oil type,
- detect major differences in produced and in-place oil composition.

If analysis clearly indicates no significant mud invasion has occurred, native state wettability tests may represent the in-situ reservoir conditions, assuming that changes have not occurred from other sources. If not confirmed, any test and subsequent analysis data that are wettability sensitive, may be compromised and could be a waste of resources (finance, personnel and time).

2.2 Native Saturation

The second important aspect to assess native state is fluid saturation; especially initial water saturation. As discussed by Bennion, et al. [19], coring operations can result in significant flushing of the core, particularly higher permeability, by the mud fluids from injection ports at the bit. If the coring fluid is water based, this will obviously result in an undesirable increase in the measured water saturation (S_w). If the coring fluid is purely oil based, irreducible water saturation (i.e., immobile) will be unaltered but mobile water (such as in a transition or aquifer zone) may be displaced from the core by oil filtrate, reducing the apparent (measured) Sw. Oil-based coring fluids, therefore, usually provide a

good estimate of "irreducible" S_w, but not necessarily the innate water saturation in a reservoir. Pure oil-based muds (often diesel based) are less common in recent years, due to HSE concerns of its benzene content. Currently, most oil-based muds, particularly for offshore wells, are synthetic, low toxicity oil-based muds (LTOBM), containing at least 5% water content and once called invert emulsion muds. Surfactants are necessary for LTOBMs to hold the oil, water and other additives together in suspension but will alter pore space wettability if invaded.

Gas, occasionally, has been used as a coring fluid but heat generated during the coring process, combined with the dehydrating nature and high rate of gas circulation, often results in desiccation of the core and artificially low saturations. This is not recommended for native state core.

Selection of an appropriate coring fluid is thus essential for obtaining representative core that can be used for saturation and special core analysis measurements. The specific data requirements, hence, measurement program, should determine the optimal coring fluid for any given situation. Table 2 summarizes some advantages and disadvantages of various types of coring fluids with respect to in-situ saturation determination and wettability effects [19,20].

Wellsite handling of native state cores must focus on rapid processing because low invasion coring may not fully inhibit invasion. Figure 3 shows CT evidence of an invaded zone, seen as the denser "halo" at the outer edges. At the very least, the outer surface of the core will be exposed to the mud during coring, core handling and shipment to the lab. Whilst exposed, there is the potential for mud chemicals to be transported through diffusion gradients within the interconnected pore fluids. This process was shown to happen at very early stages, by Zhang et al. [21].

Table 2. Mud types: pros and cons

Fluid Type	Effect of InSitu Saturation	Effects of Wettability
Water Based Mud	Good for Immobile So	Possible Wettability Alterations
	May increase Sw	(pH changes and/or ionic interactions)
	May reduce mobile So and Sg	Polymers effects
Pure Oil Mud	Good for Immobile Swi	Possible Wettability Alterations
(Diesel based	May increase So	Higher potential for asphaltenes
no surfactants)	May reduce mobile Sw and Sg	precipitation
	May reduce mobile Sg (solubility effects)	
Pure Gas	Good for Immobile So	Unknown
	May reduce mobile So and Sw	
	May reduce immobile Sw (desiccation)	
Invert Emulsion Mud	May reduce mobile/immobile Sw	Wettability Alterations
	May reduce mobile/immobile Sg	due to surfactants (emulsifier or
	May increase So	wetting agent) and other chemicals
Low Toxicity	Good for Immobile Swi	Wettability Alterations
Oil based Mud	May increase So	due to surfactants (emulsifier or
	May reduce mobile Sw and Sg	wetting agent) and other chemicals
	May reduce mobile Sg (solubility effects)	

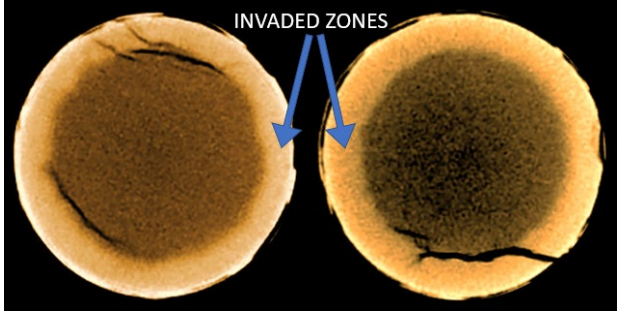

Figure 3: Outer layer mud invasion CT scan image

It is not normally recommended to cut plugs at wellsite, particularly SCAL plugs, owing to difficult conditions and impaired ability to see important sampling features, such as layer changes, bedding orientation and heterogeneities. But the rapid diffusion potential [20] and oxidation (atmospheric exposure) potential to alter wettability, Unsal et al [22] may necessitate rapid handling (wellsite plugging, trimming & preservation) for native state core, to separate the inner core material from an invaded outer layer or from surface fluids. Therefore, a decision must be made whether to process at wellsite (which pragmatically may not be possible) or to transport the core quickly to the lab for processing (recommended within 24 h, 48 h maximum).

If electing to process the core at wellsite, plugs should be cut with an appropriate fluid to minimise wettability and saturation alteration. Trim off the ends to create a 2" (5cm) long, right-cylinder plug. Weigh both the plug and both trim sections. Immediately preserve the plugs in Saran wrap, aluminium foil and wax. Weigh the wax-sealed plugs. The wax seal should be sufficient preservation, but further preservation could be to place the wax-sealed plug into a laminated aluminium (Mylar) sealable bag. If selecting to use sealable bags, the bag plus plug should be weighed once sealed. Trim sections should be wrapped in Saran wrap, aluminium foil, weighed and sealed within Mylar bags. Plug number and depth should be marked on wax coating and on the Mylar bags, McPhee et al [23].

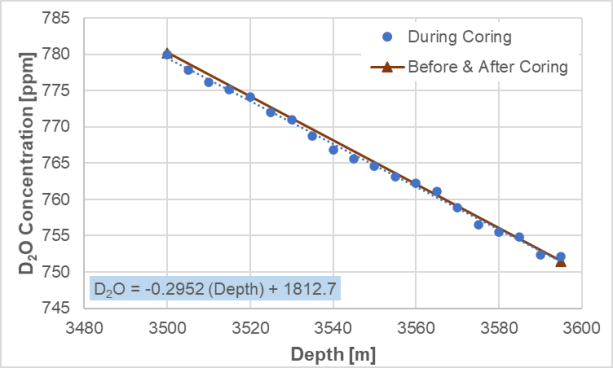

Figure 4: Tracer concentration in mud filtrate versus depth

To investigate the level of invasion, chemical tracers (e.g. tritiated or deuterated fluids, 1-bromonapthalene or other halogenated fluids, hexadecane, specific salts, etc.) should be added to the mud filtrate (either oil, water or both, dependent upon which phase is of interest) at known concentrations. Alternatively, as mentioned above, mud chemicals which can be detected and are not native to the reservoir may be used. The traced mud should be circulated around the full system three times, to ensure reasonable mixing before coring. Samples of the mud, 0.3 – 1 litre volume, should be taken at regular depth intervals throughout coring, approx. 10 – 20 samples across the cored reservoir section, plus a sample immediately prior to and after coring. The mud samples should be analysed to measure the tracer concentration as a function of depth, since this may change due to mixing with reservoir fluids. Data should be interpreted as a function of depth and regressed to obtain a depth-

based equation of average tracer concentration in the mud (see example shown in Figure 4).

3 Laboratory

As mentioned previously, the main requirements for ensuring native state would be unaltered wettability (which is unknown in most cases), unchanged saturation, no contamination, and no change to fluid composition. Whilst some aspects of change can be minimised and partially controlled, some cannot and laboratory protocols must be designed merely to verify whether or not as-received properties exhibit values that can evidence little or no alteration. Since reservoir wettability is unknown it is recommended to study all three states; native, clean and restored, to check water wet conditions can be achieved (clean state) and to compare native and restored state data. Laboratory wettability measurements are time consuming which often prohibits their use as a preassessment tool. The lab protocols discussed below will therefore largely focus on assessing and/or controlling saturation, contamination, and fluid composition.

3.1 Modified Routine Core Analysis

Since most core programs begin with routine core analysis (RCA), early assessment can begin with evaluating possible contamination from mud chemicals, by tracer analysis as part of a Dean-Stark (DS) analysis program. As a standard procedure, to avoid diffusive invasion, it is recommended to cut DS plugs at wellsite from the centre of the core (least invaded region), parallel to the length axis, McPhee et al [23]. However, in the laboratory, the experimental procedures of the DS program may require a re-design dependent upon the specific tracer used. For example, DS extraction is standardly employed as a method to measure water content; extracting the water from each plug in an individual apparatus in a few days, followed by batch hydrocarbon and salt extraction. If hydrogen isotopes (deuterium or tritium) were added to the water phase, then standard DS extraction may be viable. However, if hydrocarbon tracers or water-based salt tracers were used, batch processing cannot be used. Procedures must be adjusted to collect the used solvents for tracer concentration measurements, and solvent volumes should be minimised to increase the concentration of tracer extracted into its volume, reducing uncertainty in the tracer measurements.

The results of a DS tracer study will give an overview of invasion; the first evidence whether native state core material (uninvaded or minimally invaded) may be possible. It may show if little or no invasion occurred, or if specific reservoir layers have been invaded, or if generally high invasion has happened. The information may be used to optimise selection of native state SCAL samples. Only plugs in uninvaded depths can be selected as native state, otherwise restored state testing must be considered.

3.2 SCAL plug selection

Initial selection should be performed to obtain plugs having representative petrophysical properties aligned to rock types determined from RCA and well log data interpretation (and other reservoir information), ensuring the important zones of interest are captured. Comparison of RCA and SCAL data can be improved by cutting twin (or sister) plugs, i.e. the RCA plugs are cut as close as possible to the SCAL plugs, within the same rock layer. Selection should avoid any invaded depths, determined during RCA tracer analysis. CT images are recommended to support assessment of the selected plugs, Maas et al [24], avoiding undesirable attributes, such as: heterogeneity, fractures, etc. It is advisable to select more plugs than required, anticipating that some will be abandoned due to unrepresentative features and/or properties. This part of the selection process should be performed without opening the plug preservation materials, wax and/or Mylar bags.

End Trim Analysis

Analyses can be performed on the end trims to provide additional information to improve assessment of the SCAL plug selection and provide more relevant estimated properties (since basic properties cannot be performed on a native state plug, these properties must be estimated). It is important that the end trim material is comparable to the main plug. End trim measurements can be compared against RCA sister plug data for additional QC selection checks, since the end trim material is from the same depth and possibly a closer analogue of the core plug. Cutting plugs of approximately 5 cm long should provide sufficient end trim material to perform these suggested analyses for either 4” or 5¼” diameter core (see Figure 5). However, smaller diameter core (such as wireline core) is unlikely to have sufficient material to do this. As depicted in Figure 5, it is recommended to cut a 1” diameter trim plug from the offcut material. This supplies a SCAL core plug (1.5” diameter), a trim plug (1” diameter), plus additional trim material. The trim plug can undergo Dean-Stark extraction and routine analyses, to obtain basic rock properties, water and oil saturation, followed by high-pressure mercury injection to obtain pore throat size distribution and capillary pressure data. The remaining trim material might be used for petrographic analyses such as, x-ray diffraction (XRD), thin section analysis (TSA) and scanning electron microscopy (SEM), towards understanding the mineralogy and improved reservoir characterisation.

The following program is suggested for the end trim (plug offcut) material:

- Receipt and checking of trim preservation integrity and “as-received” weight
- Modified Dean-Stark extraction (Trim Plug)
  - measure water volume to determine saturation (equivalent to plug)
  - collect extracted oil in the toluene effluent
  - collect salt in methanol effluent (or consider the leach technique)
  - determine water saturation
  - determine oil saturation
- Measure tracer levels (compare to tracer levels in the plug)
  - from toluene effluent for hydrocarbon tracers

- and/or in the water volume (for water isotope tracers)
- and/or determine ionic composition of salt content and subsequent tracer concentration (if salt tracers were used)

- Measure grain volume, helium pore volume, porosity, grain density and gas permeability (if sufficient material is available)
- Measure mercury injection capillary pressure (trim plug, as depicted in Figure 5)

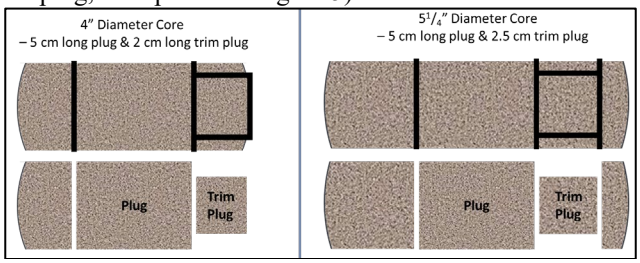

Figure 5: Schematic of possible cuts to obtain plug and trims (*note: the black lines in the upper images denote cutting transections. The lower images show resulting material*)

Contiguous end trim data can provide significant information about the core plug, if the material is relatively homogeneous. The properties of the end trim can be assumed as an estimate of the plug properties to ascribe estimated permeability, porosity, grain density and original water saturation and can be compared to the RCA (sister plug) data as additional quality control and verification of the comparability of the RCA to the SCAL plug data.

3.3 SCAL plug preparation

Native state preparation may require a customised program based on a variety of parameters, such as available laboratory techniques, core characteristics, degree of consolidation, core petrophysical properties, fluid types, pressure changes, temperature conditions and complex reservoir saturation history, such as paleocontacts. For instance, in their recent paper (Unsal et al [22]) highlighted the importance of the redox state, especially iron-rich minerals, on special core analysis, where oxidation from ferrous to ferric content could alter some basic rock properties, as well as wettability and dynamic properties. The paper recommends pre-studies to include the impact of different reducing agents on coreflood properties and suggests that reducing agents should always be considered particularly where core and/or fluids may have been oxidised. This is not widely considered in general SCAL procedures, where core and fluids are often exposed to atmosphere (though may be degassed and sometimes nitrogen purged). We have not addressed this in the following protocol but recommend that labs consider these chemical aspects.

The remainder of this section proposes one possible approach designed for plugs that might be considered "average" for a liquid hydrocarbon reservoir, for instance oil saturated above the transition zone at immobile water saturation, reasonably consolidated (not friable or unconsolidated), having permeability ranging 10 – 2000 mD and porosity ranging 0.15 – 0.30 v/v. Assuming this and assuming the core was cut using low invasion protocols based on Rathmell et al [13], oil is expected to be the mobile phase so that filtrate invasion might be considered to mix with the reservoir oil. This would replace an unknown amount of oil and alter composition. In addition, degassing will occur as the core is brought to surface, displacing the mobile oil phase. At a minimum, protocols must replace the altered oil and expanded gas volumes with a controlled oil. Dead crude oil (stock tank oil) is often used as a laboratory surrogate for live reservoir oil and would constitute a controlled oil. It is vital that the controlled oil has been appropriately prepared and checked that it is not contaminated by mud chemicals. PVT fluid analysis should include spectrographic analysis of the stock tank oil (STO) to be used, mud filtrates, and any other hydrocarbon additives to the wellsite fluid vessels (such as the stock tank or separator units, where test fluids were obtained). Spectrographic comparison should assess any reservoir oil samples for potential contamination and only uncontaminated fluids should be used in testing.

In some circumstances, refined mineral oil might be used as a controlled oil, so long as it can be shown that it is compatible with the reservoir oil without precipitating asphaltenes (Buckley et al. [25]). Where necessary, fluid compatibility testing should be performed and buffering fluids considered to understand what would constitute "controlled oil".

Water saturation may change due to invasion, depending upon the water content of the mud filtrate, but if invasion is detected within plug fluids, native state is not possible. However, salts may have precipitated dependent upon changes to temperature and pressure from reservoir to ambient conditions and upon water chemistry. The ionic composition of formation water should be reviewed for its potential to precipitate salts at ambient conditions. If salts precipitate, the composition and salinity of the autochthonous water will be altered.

Therefore, to ensure native state, any pore space saturated with evolved gas must be replaced with controlled, representative fluids. The autochthonous fluids, which may be contaminated with mud additives or particulates, or have altered during the trip to surface, must be replaced with controlled, uncontaminated fluids. It is then imperative to re-achieve original reservoir saturation after this exchange process, else significant saturation hysteresis will negate native state.

Native state preparation may thus include the following fluid protocols: exchange of altered connate water with synthetic formation water (SFW), collection and measurement of produced water for tracer analysis, replacement of altered oil with controlled oil, collection and measurement of produced oil for tracer analysis. Fluid exchange is achieved by centrifugation and/or flooding (see below protocols and bulleted summary).

After considering the fluids and possible changes to composition or saturation, if it is decided that both fluids must be replaced, flooding is not recommended because the exchanges will involve immiscible displacements to attempt to achieve residual fluid saturations, which are almost never achieved by flooding due to comparatively large capillary end effects, McPhee et al [23]. A mix of

centrifugation and flooding is recommended but must evidence that connate water saturation is re-established. To improve the probability of achieving residual saturations, it is recommended to use inert gas (nitrogen) as the displacing fluid owing to more favourable density difference, interfacial tension and contact angle than liquid-liquid centrifuge displacements. Simultaneously, it achieves residual saturation in both liquid phases, thereby minimising the volume of any particulates that may have precipitated and could be held as a colloid in these liquids. Another advantage of using an inert gas, such as nitrogen, is to reduce atmospheric exposure and potential oxidation, which can influence wettability. The centrifuge and plug storage equipment should be purged with nitrogen throughout the plug handling and loading process, to minimise exposure to air.

The rotational speeds for the centrifuge stages should be designed to apply a capillary pressure equivalent to a representative height above free water level (HAFWL), or apply a force expected to achieve a water saturation consistent with other water saturation data in similar rock types and similar HAFWL of the same reservoir. This requires discussions between the laboratory and oil company representatives, to review any relevant available reservoir data, such as well-logs, fluid properties, previous core data, etc. If end trim mercury injection was performed, these data can be used to support the comparison of reservoir to laboratory data. Care should be taken to consider the differences in fluid properties between reservoir and laboratory (surface tensions and contact angles), particularly the conversion from an oil-water to gas-water system, since nitrogen will be used as the displacing fluid.

The core plugs should be placed into a centrifuge and spun at the predetermined speed(s). Produced fluid volumes should be collected and recorded. Water production may imply that the applied centrifuge force was too high or that water has invaded from mud filtrate or that wettability has been altered. Tracer concentration of the liquid volumes should be measured. Volumes produced should be checked against the weight change to ensure agreement and verify fluid density values.

After each centrifuge step, nitrogen should be replaced by flooding with an appropriate controlled fluid, either degassed SFW, STO or refined oil, against a back-pressure of at least 10 bar (150 psi). Gas will be dissolved into the pressurised, undersaturated fluid. There is an option to obtain additional data during these floods by performing a miscible displacement, which will give information about the homogeneity of the pore space saturated by the mobile fluid and an estimate of mobile fluid volume. After replacing nitrogen with a controlled fluid, weight checks can be performed to determine the volume of fluids exchanged.

Added wettability information also may be gained by performing nuclear magnetic resonance (NMR) tests at liquid-filled residual saturation stages (see description later).

A cartoon representing the native state preparation process is given in Figure 6, and is described in bullet point form below:

- Remove from preservation
- Weigh “as-received” plug
  - weight check against wellsite recorded weight
- Measure NMR T1 and T2 (if possible T2-D analysis)
- Centrifuge under nitrogen to residual water (Swr) and residual oil (Sor) saturation
  - Collect & measure fluid volume(s)
  - Analyse fluid(s) for tracer
- Remove from centrifuge and weigh
  - Compare weight change with produced fluid volume
- Load to a coreholder under nominal confining stress and saturate with synthetic formation water (SFW) by waterflooding against 20 bar (300 psi) pore pressure for at least 5 pore volumes injection (PVI), until stable differential pressure (dP)
  - One method to verify removal of the gas volume, would be to carefully de-apply and re-apply back pressure whilst continuing injection. If no gas is present, differential pressure should be equivalent in both regimes.
- Measure effective water permeability at S_{or}
  - There is an option to perform miscible dispersion analysis using a doped SFW. Dispersion can provide an estimate of the mobile water volume, hence water saturation (S_w), in turn an indication of S_{or}.
- Remove from the coreholder and weigh
  - Determine fluid volume exchange from weight change
- Measure NMR T1 and T2 (if possible T2-D analysis)
- Centrifuge under nitrogen to S_{wr} and S_{or}
  - Collect & measure fluid volume(s)
- Remove from centrifuge and weigh
  - Compare weight change with produced fluid volume
- Load to a coreholder under nominal confining stress and saturate with controlled STO by flooding at 20 bar (300 psi) pore pressure for at least 5 PVI, until stable dP. Verify gas removal.
- Measure effective oil permeability at S_{wr}
  - There is an option to perform miscible dispersion analysis using a doped oil. Dispersion can provide an estimate of the mobile oil volume, hence oil saturation (S_o), in turn an indication of S_{wr}.
- Remove from the coreholder and weigh
  - Determine fluid volume exchange from weight change
- Measure NMR T1 and T2 (if possible T2-D analysis)

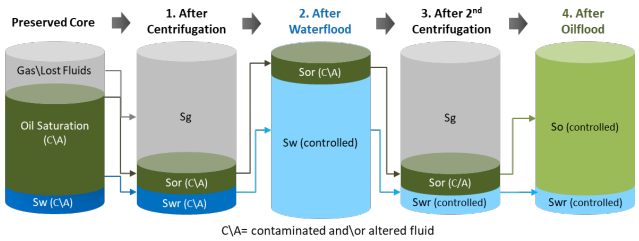

Figure 6. Cartoon of the process stages

Tracer analysis on fluid extracted by centrifugation

The fluid volumes extracted under the first centrifugation should be analysed for tracer content. In general, tracer concentrations measured on volumes produced under centrifugation will be more accurate than DS data because they are not diluted by solvent extraction volumes. Ideally, these should demonstrate that no tracer was detected or, at least, that concentrations are below the measurement limit. The measurement limit will be dependent upon the type of tracer and the test procedure used to measure its concentration. Therefore, it is wise to consider these measurement limits before selecting the tracer to be used. Any plug showing tracer concentration above the minimum measurable limit, should be disqualified due to invasion (hence, possible change to wettability). Invaded plugs cannot be used as native state and restored state must be considered for any wettability sensitive analyses. If this is the case, a cleaned state wettability analysis also should be performed to verify that water-wet conditions are achieved by cleaning before employing restored state protocols.

If no invasion is observed, the in-situ wettability is believed to have not been altered by mud filtrate invasion and protocols can continue to saturation checks to verify that connate water saturation can be re-established.

Table 3 shows the data from five (5) samples from a North African field study, where core was acquired using low invasion methods. Estimated tracer concentration within the hydrocarbon (HC) phase of the plugs is shown between 0.1 to 0.4 % for 4 plugs, across a permeability range of 100 to 2300 mD. However, the fifth (1500 mD permeability) shows high tracer content, 8.1 % and was subsequently disqualified from native state analysis. In this study, approximately 10% of all plugs were disqualified due to high invasion. Most of these plugs were from the top or bottom of individual core runs, where the rock is exposed for longer, or from zones where fracturing had occurred.

3.2 Saturation estimation

Dean-Stark saturation data, either from routine core analysis, or the trim plug, is useful to support estimations of irreducible water saturation, by estimating the "as-received" oil saturation ($S_{o,AR}$). S_w and S_o data obtained on the trim plugs can be used as a direct estimate of the "as-received" saturations of the SCAL plug. Otherwise, S_o from Dean-Stark of RCA plugs can be used to derive a correlation to gas permeability and thereby estimate $S_{o,AR}$ in each SCAL plug based on effective permeability measurements (either effective oil permeability at Swi or water permeability at Sor). **Note**: the native state permeability is always effective permeability since absolute permeability can only be measured once cleaning has been performed and the sample saturated with a single fluid phase. $S_{o,AR}$ values are used to estimate residual oil saturation (S_{or}) after the first centrifugation under gas, which then can be used to estimate S_{wi}.

$$S_{or} = S_{o,AR} - \frac{V_o}{V_p}$$

where V_o is the oil volume produced during centrifugation and V_p is pore volume.

During the second centrifugation the sample is mostly water saturated (after displacing nitrogen with SFW) but contains residual oil content. The second centrifugation should re-establish initial water saturation under nitrogen, whilst the S_{or} remains constant. Thus Swi can be calculated by:

$$S_{wi} = 1 - S_{or} - \frac{V_w}{V_p}$$

where V_w is the water volume produced during the second centrifugation under nitrogen gas.

Table 3 provides data from five (5) plugs undergoing a native state preparation for a large North African field. It does not show all measured or calculated values, but it indicates some of the important information pertinent to estimating saturations and for quality control.

Table 3: Table of measured properties for some samples

Estimated Permeability	**(mD)**	**100**	**350**	**850**	**2300**	**1550**
Estimated Porosity		0.135	0.175	0.191	0.221	0.210
Estimated pore volume		6.9	8.5	9.4	11.3	9.6
Centrifugation to Sor+Swi						
Weight before centrifuge	(g)	109.84	107.02	108.10	108.06	97.66
Weight after centrifuge	(g)	107.50	104.40	106.00	105.38	95.70
Volume water produced (burette)	(ml)	0	0	0.1	0	0
Volume oil produced (burette)	(ml)	2.5	2.7	2.1	1.8	2
Tracer concentration	(ppm)	0.1	0.1	0.4	0.6	11.3
Tracer in mud at sample depth	(ppm)	140.1	139.3	139.8	139.4	139.9
Filtrate Percentage in HC phase	%	0.1	0.1	0.3	0.4	8.1
Water saturation by flooding SFW						
kw(Sorg) - SFW	(mD)	59	367	1543	2458	1607
Weight after kw(Sorg)	(g)	113.46	113.40	114.95	115.46	104.73
Volume water replaced	(ml)	5.18	7.83	7.78	8.77	7.85
Establish Swr (gas-water centrifuge)						
Weight after centrifuge	(g)	107.58	104.53	106.26	105.64	96.09
Wt difference at centrifuge endpoints	(g)	0.08	0.13	0.26	0.26	0.39
Change in water saturation	(frac)	0.010	0.012	0.023	0.019	0.033
Oil saturation by flooding STO - 60°C						
ko(Swi)	(mD)	89	341	802	2269	1531
Weight after ko(Swi)	(g)	111.73	110.65	112.08	113.21	102.48
Volume oil exchanged	(ml)	5.17	7.63	7.26	9.44	7.97
Estimated Swi	(ml)	0.246	0.103	0.226	0.165	0.173

Estimated porosity and pore volume are shown. In this study, porosity was estimated from grain density and porosity properties of sister routine core plugs (similar depth and sand layer) and using measured calliper plug dimensions to calculate bulk volume. Bulk volume from calliper measurements assume a core plug to be a perfect cylinder and thus incorporate a higher potential error margin than the recommended ±0.5 porosity units (p.u.) in API RP40[26]. These uncertainties will be reduced at the end of testing, when measurements of grain, bulk, and/or pore volume can be performed, but in the meantime a higher error margin potential must be allowed – possibly ±1 p.u. In turn, saturation uncertainty will increase from the recommended ± 3 saturation units (s.u.). However, we would recommend a limit of ± 6 s.u. at this initial stage of preparation for selecting viable plug samples. **Note**: this limit is partly arbitrary, based on the understanding that most of the saturation error is derive from the porosity error, which varies between approximately 2 - 10 s.u. for ± 0.5 – 1 p.u. error in the porosity range of 0.100 – 0.300. Volumetric production measurement error contributes far less to the overall saturation error, usually between 0.3 – 1 s.u.

Regarding quality control considerations, weight changes should compare to equivalent fluid production

volumes during centrifuge displacement steps, and to the subsequent replacement volumes when re-saturating with water or oil by flooding. It is recommended to calculate the difference between volumetric and weight measurements to saturation difference and apply the recommended limit of ± 6 s.u. Any plug outside this limit should be discontinued from native state testing.

From the first centrifugation under gas to S_{wi} and S_{or}, produced fluid volumes should be recorded. Water production should be minimal, preferably zero, since any water production indicates a change in initial saturation conditions (hence, no longer native state). Any observed water production should be calculated as saturation change and should not exceed ± 6 saturation units (s.u.).

The weight after the two centrifugation steps should be approximately equal, although some differences might be expected if the autochthonous fluids and controlled replacement fluids have different density. In Table 3, the weight after centrifugation to S_{or}+S_{wi} and the weight after centrifugation to establish Swr should be equal. Assuming no difference in Sor (since centrifugation was performed under equal displacement force), the weight difference between these two centrifugations (neglecting measurement error) can be assumed as possible change in water saturation. Any change outside ± 6 s.u. should disqualify the plug from native state testing. **Note**: this is a worst-case error scenario, since it assumes that the total weight change is attributed to water; however, changes in oil saturation and grain loss may also contribute to weight errors. It is therefore important to observe, record and account for any visible grain loss. For this North African field, the 5 plugs shown in Table 3 show a change in water saturation between 1 and 3.3 s.u., implying that change is minimal and might be considered as native state.

Estimated S_{wi} values can be compared to DS S_w data from sister RCA plugs and/or trim plugs and to any well log petrophysical data available for the field under analysis, as another QC step.

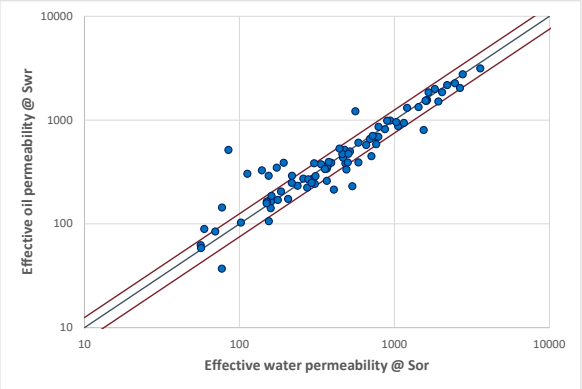

Figure 7: Effective oil versus effective water permeability

Although a weaker correlation, effective permeability data also may be used when considering selection and viability of core plugs. Figure 7 shows the correlation of a study from a large North African field; red lines indicating a 25% uncertainty. In this study, effective oil and effective water permeability was approximately equal but other reservoir systems and different wetting behaviour may result in different correlations, such as effective oil permeability being higher, or vice versa. Outliers may not require exclusion but must be deliberated whether or not to exclude.

3.2 Wettability measurements

Nuclear Magnetic Resonance (NMR) measurements (T1/T2 and Diffusion) can be performed at different stages during native state preparation process. 2D D-T2 intensity maps can provide additional semi-quantitative information about wettability for fresh state or restored state cores. Existing restricted diffusion models allow the estimation of surface relaxivity, tortuosity, and wettability from the diffusivity and T1 or T2 data in the D-T2 maps measured on partially saturated plugs, without the need for separate NMR measurements of the bulk fluids and fully water-saturated rocks. [27,28,29,30]

Comparison of T1-T2 maps at different initial or irreducible saturation stages (S_{wi}, S_{or}, S_{gr}) could provide added information of fluids type and distribution within the rock pore structure, at fresh state conditions and after the native state preparation procedure.

The first set should select non-invaded core plugs and perform native state followed by restored state wettability analysis. This is to investigate the native state wettability of the acquired, low-invasion core and also to compare against restored state wettability. Using the same plug, although increasing experimental time, allows direct comparison of results. Plugs should also be selected from a range of lithofacies, petrophysical properties and height above free water level, to aid understanding of wettability distribution within the reservoir.

A second set should select known invaded plugs to show any differences to non-invaded plugs, thus exhibiting the effect of mud filtrate invasion on in-situ wettability.

A third set should select both invaded and non-invaded plugs. These should undergo typical cleaning and drying procedures followed by clean state wettability. Data from this set should exhibit strongly water-wet behaviour in both invaded and non-invaded plugs (except where mineralogy may dictate other behaviour), thereby indicating that filtrate components can be removed, and restored state procedures may be considered viable. Like the first set, these plugs should be chosen from a range of rock properties. If results are not strongly water-wet, mineralogy should be analysed for known non-water-wet components, such as: organic material (coal, kerogen, pyrobitumen, etc.), halite, chamosite, etc.

Most commonly, wettability is studied using the combined Amott-USBM method. Wettability results from the North African field are shown in Figure 8, indicating wettability indices of non-invaded native state plugs (NS), invaded native state plugs (INV) and restored state plugs (RS). These indicate similar mixed-wet data in native and restored state plugs, but alteration to strongly oil-wet behaviour of the invaded plugs.

4 Discussion

Core Preparation Discussion

Native state core preparation should not use fixed, or standard, procedures, nor is it guaranteed to result in viable plugs for use in a native state SCAL study. Instead, process control must regard the various factors influencing wettability and wettability alteration during coring and core preparation, and experimental procedures should be designed accordingly with decision gates in place to determine whether or not native state analyses can be used. This will be reservoir and well specific, since lithology, fluid types and coring procedures have a direct impact on wettability. Figure 10 gives an example flow diagram with decision gates that might be used when assessing native state protocols. It cannot be exhaustive, since there may be other controlling variables that have been omitted.

The flow diagram indicates some of the important decision factors:

- Wettability altering mud components
  - If present (or unknown), then invasion must be investigated and will either exhibit; no invasion, whereby NS is viable, or invasion, in which case NS is unviable.
  - If not present, then other alteration factors need consideration (e.g. saturation change)
- Were tracers used in the mud system during coring?
  - If not, consider using isotope analysis to investigate mud components
  - Else, if mud invasion is unknown, native state may be unviable
- Is there potential for asphaltene precipitation?
  - If asphaltenes are unstable and likely to precipitate, then an asphaltene study must be performed to confirm or discount precipitate in the cores.
  - Presence of precipitate may exclude NS analyses, or further study must determine if asphaltenes can be removed without altering original wettability
- Is water saturation unchanged?
  - If changed, can S_{wi} be re-achieved?
  - If S_w has been altered and cannot be re-achieved, NS analysis is unviable
- Is water composition unchanged, or is salt precipitation likely?
  - Salt precipitate must be removed, usually by replacing the autochthonous water with controlled formation water
  - This requires change to saturation and hence, re-establishment of S_{wi} must be achieved

It is possible that old, preserved core (either wax-coated or Mylar-sealed) may be selected for use in a native state SCAL study. In such a case, the preservation must be scrutinised. Seal integrity must be investigated. Have fluids been maintained within the sealed material? Are the properties of the core congruous with previous data from the core? Assessing the viability of old, preserved core will require a rigorous process including all the aspects for investigating fresh core, described in this paper, but also ensuring comparison to previous study data. Any incongruity in reservoir properties should be deemed to negate native state analysis.

Wettability Discussion

Figure 8 shows data implying a distinct difference in the wetting index of invaded material (strongly oil-wet, Amott wettability indices between -0.72 and -0.83), compared to native and restored state core material (mixed-wet). Native and restored state plugs showed generally similar results with an index range between -0.16 to -0.50, in general. One native state sample, however, showed a slightly positive index (+0.15), but this may have been a function of poorer permeability. In general, poorer permeability will result in higher S_{wi} and hence, less oil present and less chance for wetting to be altered away from water wet. Figure 2 summarised the data as a function of permeability further supporting the evidence of invasion causing a change to wettability.

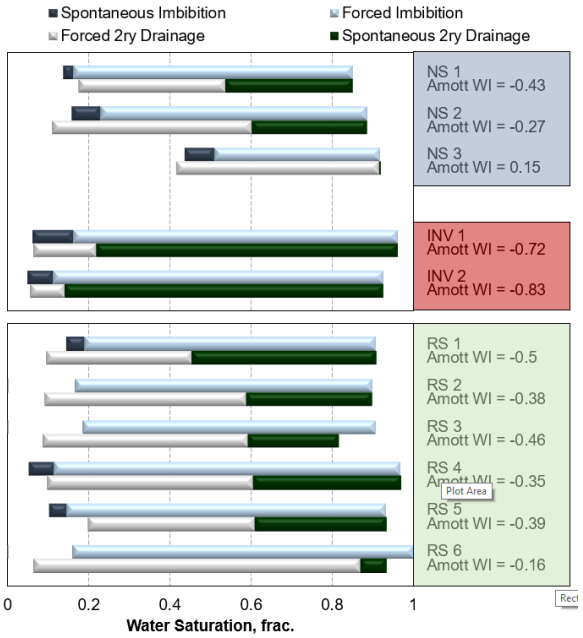

Figure 8: Wettability indices for native state (NS), invaded (INV) and restored state (RS) plugs

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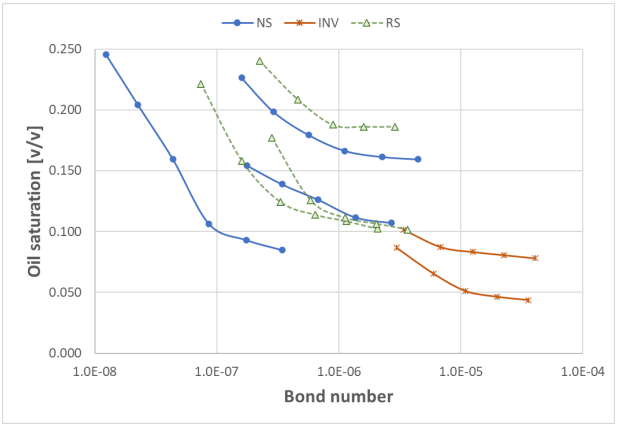

Figure 9: Centrifuge wettability data, So versus Nb

Remaining oil saturation was plotted against bond number (Figure 9) from centrifuge forced imbibition data, also indicating similar behaviour between the NS

and RS plugs (S_{or} stabilising between 10^{-7} to 10^{-6}), whilst invaded plugs did not stabilise before 10^{-5} and at generally lower residual oil saturation.

5 Conclusions

As-received core is probably not native state and SCAL should never be performed on as-received core without first considering the potential changes from coring, retrieval, wellsite handling and shipment.

Wettability analysis is an important stage in determining native and/or restored state feasibility.

We have defined a decision tree (illustrated in Figure 10) that could be used as an example process guide for considering whether native or restored state core should be used, though individual reservoirs should consider individual process design.

Data from the large North African field imply that native state preparations, for most of the plugs and the specific rock-water-oil combination in that particular reservoir system, were largely successful and that restored state procedures could be equally viable where invasion had occurred. Plugs were disqualified from native state testing where high invasion was seen whilst the majority of plugs showed low invasion.

Native state and restored state samples exhibited broadly similar behaviour, but invaded plugs exhibited strongly oil-wet characteristics.

The viability of a restored state process is largely determined by the ability of core cleaning to achieve strongly water-wet behaviour, except where certain organic and/or non-water wet mineral content exists naturally.

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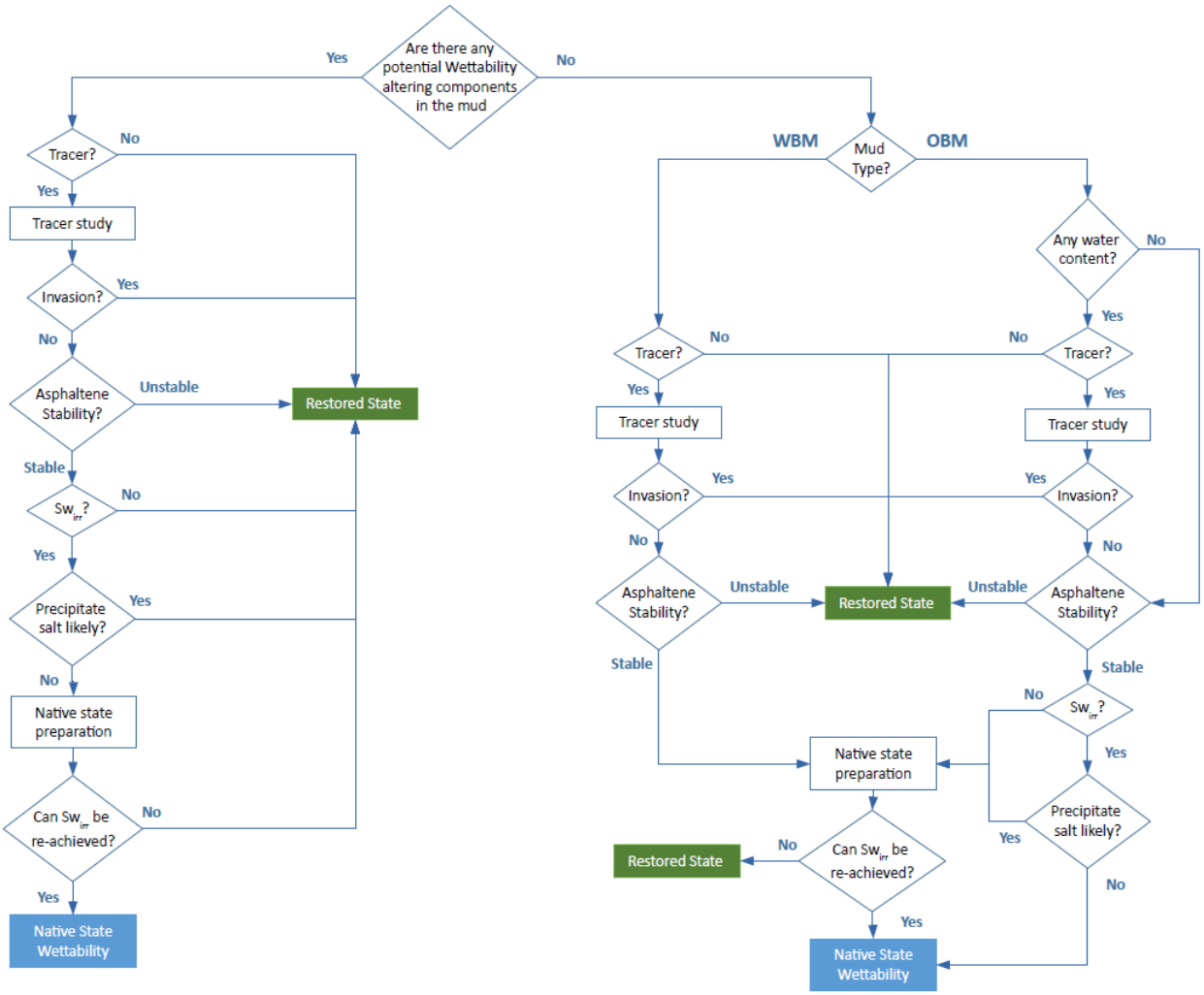

Figure 10. A suggested decision tree to determine viability of native state SCAL