JCR 7

Advanced Core Measurements
“Best Practices”
for Low Reservoir Quality Chalk

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Introduction

Marginal secondary or tertiary chalk reservoirs with lower porosity and very low mobility are becoming increasingly important for a growing number of mature chalk fields. Significant volumes of hydrocarbons are often trapped in chalk with low reservoir quality representing an interesting economical potential, but uncertainty relates to estimation of in-place volumes as well as prediction of reserves and production profiles. Flow simulation rests on a number of advanced core measurements and many of those have shown to be intrinsically difficult in low mobility rock giving rise to spurious and less credible data. Laboratory techniques seem to struggle to acquire reliable data that are representative of in-situ reservoir conditions in very low mobility/quality chalk.

Under the present Joint Chalk Research Programme, Phase 7 (JCR 7) one of the themes is called "Low Reservoir Quality Chalk" (LRQC), and the objective is to identify challenges associated with the development of marginal quality chalk with low porosity/permeability and make steps that may push the boundary separating prolific chalk and sub-economic chalk. Under this theme, a project called "Advanced Core Measurements - Best Practices for LRQC" has been approved. This project should define best practices with respect to core measurements on low mobility rock and thus the project aims at increasing the quality and consistency of laboratory derived core data. The ultimate goal is to reduce uncertainty associated with assessment of in-place volumes, production profiles and reserves.

A document should be written to provide guidance to the E&P companies (i.e. to the staff ordering studies) on what to request with respect to laboratory techniques, procedures, and documentation in order to obtain data to the current highest standards. The document should further address available techniques with the view to rank them in terms of data quality, their shortcomings and suggestions for improvement.

The outcome of this project is the present "Best Practices Manual".

An Example ...

During reviewing and presentation of draft copies of the manual, the wishes for examples of good vs. bad core data were raised and reiterated. The editors were finding it hard to meet that request since no data could be identified with two sets of measurements that represent good and bad quality data respectively. However, one case history that comes close was identified serving as an example of the consequences of using data of lesser quality.

In 2006, a permeability transform was established for the Danish South Arne Field allowing oil permeability to be calculated from total porosity. The transform was based on routine core data (i.e. helium porosity and quick look oil permeability measured at ambient conditions) acquired during the 90’s from various discovery and appraisal wells across the field. In 2008, 24 new plugs were submitted for measurements of oil permeability at net overburden pressure this time with the objective to acquire the optimum quality data (without using a PVT cell) while testing the relationships from 2006. The outcome was new and what is interpreted to be improved poro-perm relationships (Figure i1).

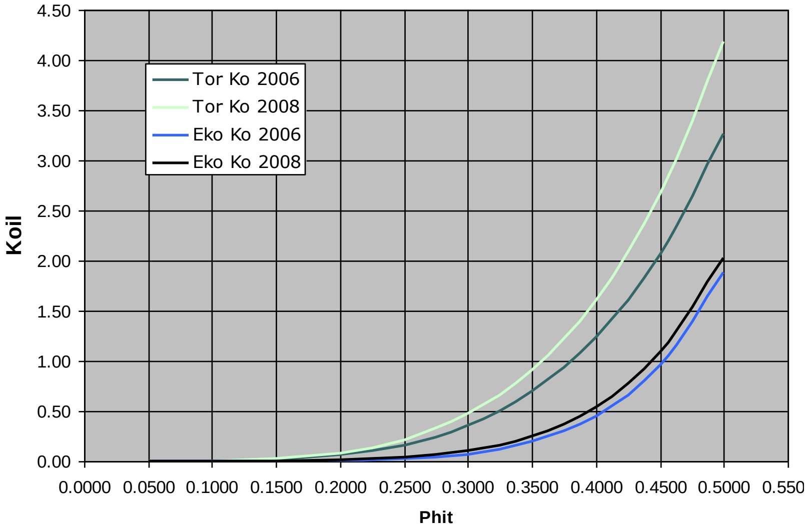

Figure i1 Previous and updated poro-perm relationships for the South Arne Field

For both the Ekofisk and Tor Fm. the new transforms predict improved permeability for porosities above 25% while none or little improvement is seen for porosities lower than ca. 25%. It can also be seen that the largest improvement is achieved for good quality rock of the Tor Fm. In other words, there seem to be a correlation between the improvement in measured permeability and rock quality (i.e. permeability). Possibly the outcome of this exercise demonstrate the limitations in modern laboratory techniques when dealing with low permeability rock? For the poorest of qualities of chalk, laboratory techniques may simply fall short of being able to produce data that represents the rock at reservoir conditions.

Relationships like those above are often used in flow simulation to establish matrix oil permeability prior to performing permeability enhancement. If the relationship is too pessimistic, the starting point becomes too conservative. For a potential development of low reservoir quality chalk, this may translate into difficulties establishing whether the relationship between return on investment, capital expenditures and associated economical risk is acceptable. Ultimately, wrong conclusions may be made.

Acknowledgements

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Very special thanks to Jos Maas and Niels Springer for your continuous and relentless contributions to the process of making this document. You know what your contributions are. Without your efforts, the project would have struggled and the document would never have materialized the way it has.

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About this manual

The North Sea is a mature petroleum province and core analysis data on the chalk fields are in general more than 10 years old, often much older. Present interest in development of marginal fields or low reservoir quality chalk units may provoke new studies on old core or coring of new wells, and there is business value in doing it right, and not copy an earlier program just because it is held up as “best practice”. Proper quantification of reservoir properties and their uncertainties is of critical importance for field development planning. Advanced or Special Core Analysis (SCAL) is an important investment in basic data in support of field development.

When designing a core analysis programme, the routine core analysis (RCA) measurements are straightforward in the sense that they are standardised and follow in general the recommendations given by the American Petroleum Institute (API). The latest Recommended Practice for Core Analysis RP40, 2nd edition was published by API in 1998 and is now withdrawn. It is normal practice for API to revise or withdraw their publications after 5-7 years, but this does not infer that the standard is obsolete, and reference to API RP40 is made throughout this manual where we feel it still applies or is at least informative to the reader.

No formalised standard or recommended practice exists for special core analysis (SCAL) measurements that (opposite to RCA measurements) are performed at overburden or reservoir conditions. This is due to the physical conditions, previous life and future development of a field that is unique. The type of SCAL tests remain much the same, but material handling and execution of tests should respect the specific interests of the client and utilise the present advancement in written scientific papers and laboratory test equipment. The present "Best Practices Manual" therefore reflects what the members of the Expert Group consider the best procedures for advanced core analysis of low reservoir quality chalk. The different categories of analyses are discussed in chapters that can be read separately and each with its own reference list.

Chapter 10 “Numerical Simulation” has real life examples to facilitate optimal design of SCAL experiments on LRQC core plugs. In addition, it shows the business values of applying state-of-the-art interpretation-by-simulation of SCAL experiments. A must read for all involved in SCAL for LRQC.

The JCR companies should observe that advanced core analysis on low quality chalk may take long or even very long analytical time in order to obtain best quality, and this may justify increased costs for such studies.

We like to stress that the design of a SCAL program is definitely a multi-disciplinary effort: close collaboration between the client and the service provider and geological, petrophysical, production technical and reservoir engineering input is required to obtain the optimum value of an advanced core analysis study.

Jos Maas
Niels Springer
Editors

References

For the sake of simplicity, a number of abbreviations have been used in referencing:

AAPG: *American Association of Petroleum Geologists*

AIME: *American Institute of Mining, Metallurgical and Petroleum Engineers*

API: *American Petroleum Institute*

JPT: *Journal of Petroleum Technology*

SCA: *Society of Core Analysts*

SEG: *Society of Exploration Geophysicists*

SPE: *Society of Petroleum Engineers*

SPEFE: *Society of Petroleum Engineers Formation Evaluation*

SPWLA: *Society of Petrophysicists and Well Log Analysts*

Many references relates to papers presented at the annual symposium held by SCA. They can be downloaded free of charge from the Society's web page http://www.scaweb.org/. Search the paper by the number given in the reference list. Papers published in SPE journals can be downloaded from the pay site https://www.onepetro.org/.

1. Routine Core Analysis (RCA)

1.1. Abstract

Routine core analysis (RCA) in the petroleum industry serves the purpose of describing and sampling a native core, accurately analysing the porosity, permeability, grain density and fluid saturations, and cutting and preserving plugs and full core pieces for future advanced core analysis, rock mechanics, rock physics etc. experiments.

1.2. Introduction

For many years the procedures for routine (or conventional) core analysis within the petroleum industry have been treated in publications from the American Petroleum Institute (API) of which the latest is *”Recommended Practices for Core Analysis, RP 40”, 2nd ed. (1998)*. Although withdrawn by API, the publication can still be downloaded from the API website. RP 40 gives a detailed and very structured description of methods, instrumentation, accuracy and precision in core analysis. The following subjects are treated in RP 40:

- Planning a coring programme
- Well-site core handling procedures and preservation
- Core screening and core preparation
- Fluid saturation
- Porosity determination
- Permeability determination
- Supplementary tests (among others petrographic characterization, grain size distribution and core water salinity)
- Reporting

The procedures and recommendations are valid for chalk as well, and all core laboratories and anyone commissioning a core analysis project should know about this publication and comply with the recommendations given therein. Further advice on planning a core analysis programme can be found in a paper by *Owens and Cockcroft (1990)*.

It should be observed that routine core analysis is carried out at room conditions i.e. at 20-25°C and at confining stresses below 1000 psi, very often 400 psi in routine gas permeability measurements. It is the client’s responsibility to overburden correct RCA data before input to a reservoir model. Corrections should be based on data obtained from overburden and/or reservoir condition special core analysis (SCAL) studies, cf. Chapter 4.

The section below summarizes the detection limits and errors in RCA from API RP 40 and from experience among the core laboratories involved in analysis of North Sea chalk.

1.3. Recommendations

1. Laboratories and anyone commissioning a core analysis project should be aware of and in general comply with the Recommendations given by the American Petroleum Institute (API) in their latest publication *”Recommended Practices for Core Analysis, RP 40”, 2nd ed. (1998)*.

2. If determination of unbiased initial water saturation and formation water composition is an objective of a coring operation, it is recommended to spike the drilling mud with a tracer e.g. D_2O.

3. Quality steady state and un-steady state gas permeameters are recommended for measuring the permeability of low quality chalk. For very tight chalk having slip-corrected (*Klinkenberg*) gas permeability at or below approx. 0.01 mD, an un-steady state (transient type) instrument is preferred for speed of operation.

4. Laboratories should measure internal gas permeability standards at regular intervals, e.g. in conjunction with major core analysis jobs; figures outside the accepted error bands should invoke a check of procedures and instruments.

5. When a slip-corrected (*Klinkenberg*) gas permeability is obtained from steady state measurements, at least four mean pressure measurements are recommended in the calculation of the slip-corrected gas permeability. A correlation coefficient, indicating the goodness of fit of the regression line, may be given as well. If the slip-corrected gas permeability is obtained from an un-corrected gas permeability measurement by a correlation, it should be clearly stated in the report.

6. RCA plugs for poro-perm analysis are normally taken at ~ 30 cm (1-foot) intervals along the core. Mini permeametry is recommended as a cost effective and valuable tool in resolving small-scale textural features (fractures, hardgrounds, chert bands etc.) that may escape the normal sampling interval and laboratory practice, and otherwise have a profound effect on fluid flow.

7. Boyle's Law Helium porosity measurement is recommended in routine core analysis with unconfined bulk volume determined from submersion in Mercury for the best possible accuracy.

8. Laboratories should perform frequent calibration runs of their Boyle's Law Helium porosity equipment for the best accuracy in porosity determination; cf. *API RP40, p. 5-10 to 5-14*.

9. The value of measuring routine poro-perm data at additional overburden stress conditions is questionable because compaction is different for dry and wet (reservoir) chalk. Overburden data should be obtained from SCAL experiments, cf. the Chapter on Electrical Measurements.

10. Routine chalk samples having grain densities outside the range 2.71±0.02 g/cc should be inspected for unusual mineralogy or possible laboratory errors (insufficient cleaning and/or drying).

11. RCA on chalk formations known to have insoluble residue (ISR) ≥ 5 wt-% should include determination of ISR and pct. silica at regular intervals along the core, e.g. one sample every meter (or 3 feet) of core; semi-quantitative XRD mineralogy may be an option as well.

12. RCA plugs that are selected for advanced SCAL tests should be effectively cleaned by flow-through cleaning as explained below.

1.4. Sampling

Drilling of small, horizontal (relative to the bedding) cylindrical plugs of 25 mm (1") or 38 mm (1½") diameter with a spacing of ~ 30 cm (1 foot) along the length of the core has for many years (by convention) become the normal practice in RCA. The advantage is a (nearly) statistical unbiased sampling, an easy correlation with other wells or fields having the same sampling equidistance. However, a rigorous sample spacing is not always possible in chalk; rubble intervals, fracture zones and chert bands will, based on experience, not be sampled during a routine operation. Also a disadvantage of the conventional practice is the risk of overlooking textural elements smaller than the spacing of ~ 30 cm that may have a significant effect on the reservoir performance, e.g. textures like thin chert bands, hardgrounds, stylolites and fractures/fractures zones. Such features may be analysed by mini permeametry, a fast and cost effective technique that has become an important supplement to conventional core plug analysis. This is covered in more detail below.

RCA data, mini permeametry and X-ray CT-screening of core and plugs is a powerful tool in selecting representative and homogeneous plugs for advanced core analysis the results of which is input to reservoir simulators, cf. the Chapter on Screening of Samples and *Maas and Hebing (2013)*.

1.5. Technology

Measurement of routine porosity and permeability is solely performed in gas injection and gas flow instruments on cleaned and dried cylindrical plug samples. The measurement gas is helium or nitrogen and the measured property known as He-porosity and gas permeability respectively. RCA is offering fast and cheap data, but it should be observed that a gas permeability differs from a liquid permeability because the fundamental physical properties of gases and liquids are different. The methods and principles behind the instrumentation used in routine core analysis are covered in detail in the former API RP40 publication (1998); a summary is given below with a view to analysis of chalk.

Permeability

Steady state gas permeameters have been widely used in routine measurement of un-corrected gas permeability and slip corrected (*Klinkenberg*) gas permeability. With decent instruments equipped with quality pressure regulators, pressure transducers and orifices or thermal mass flow meters the following performance should normally be expected for routine core analyses performed since the mid 1990's (partly after *API RP40, p. 6-42 and Thomas & Pugh, 1989*):

Table 1.1: Analytical precision that should generally be expected from steady state gas permeability instruments

Measurement	Range, mD	Precision
gas permeability, conventional (un-corrected)	< 0.01	≥ 25%
	0.01-0.1	8%
	0.1-1.0	8%
	> 1	5%
gas permeability, Klinkenberg (slip corrected)	0.01-0.1	15%
	0.1-1	10%
	> 1	5%

The precision (= reproducibility) in the table is given at the 68% level of confidence (±1 standard deviation; in the table expressed as a relative standard deviation in percent, also known as the coefficient of variation). Lower Limit of Detection: Due to the long analytical times required for measuring very low gas permeabilities, it is not practical to use a steady state instrument to measure Klinkenberg permeability below approx. 0.01 mD.

Accuracy (=true value) is very important in core analysis but difficult to control for permeability measurement. No certified reference materials are available for permeability measurement, and API therefore suggest core laboratories keep internal reference samples that have been control measured by one or more third party laboratories (*API RP40, p. 6-39*). However, robust porous ceramic materials with a controlled pore size distribution and given porosity and hydraulic conductivity can be obtained from several manufacturers in sizes that match the frequent plug sizes in RCA. Such materials may be used as internal standards in RCA.

<u>Pitfalls:</u> Steady state measurement of the slip corrected (*Klinkenberg*) gas permeability requires several independent measurements at different mean gas pressures (at least four is recommended) from which the slip corrected gas permeability is derived by linear regression. Not all laboratories do this, but gives a slip corrected figure based on internal laboratory derived correlations between un-corrected and slip corrected data for a range of samples of different lithology and permeability.

It should be observed that the inertial resistivity increases for low permeability rocks and at increasing mean gas pressure (that also affects the gas density). To reduce errors in measurement and calculation of routine gas permeabilities it is essential not to exceed the maximum allowable pressure gradient imposed on the plug sample during measurement, cf. *API RP40, p. 6-13 ff*.

<u>Un-steady state gas permeameters</u>, also known as transient or pulse type instruments, can measure very low permeability samples faster. No independent surveys of the precision of these instruments have been published. Lower limit of the pressure-falloff instruments are approximately 0.001 mD for routine tests. The advantage of the pressure-falloff method is that the un-corrected and the slip corrected (*Klinkenberg*) gas permeability can be determined from one single pressure-falloff test, while 4 independent measurements are recommended for the steady state method.

Very few comparative studies have been published on the two gas permeability methods for low permeability rocks. *Charles et al. (2007)* found that the unsteady state method gave systematically higher figures than the steady state method in a study on low permeability rocks. No explanation for the difference was offered.

<u>Mini or probe permeameters</u> have become a valuable supplement to plug permeability measurements. They offer a non-destructive, high-density permeability sampling of core material that is not available from RCA core plugs. Automatic steady state and unsteady state instruments are in use, and they have the same accuracy as given for the plug instruments above. A description of the two different instruments is given in *API RP40 (1998), p. 6-25 and 6-35*. A description and practical approach to mini permeametry is also given by *Halvorsen and Hurst (1990)*.

Mini permeameters can position the probe tip with a precision below 1 mm, and the step distance is for most practical purposes between 1 and 6 cm. The step distance may be decided from the size of the textural elements believed to affect fluid flow, e.g. based on observations from core description and core photos.

Mini permeametry on low quality reservoir chalk is recommended on dry, slabbed core or thin cuts placed in trays (core biscuits). Observe that mini permeametry on fresh core surfaces is possible, but may be heavily affected by uncontrollable relative permeability effects from mobile fluids, mud invasion in the surface rim of the core and potential probe sealing problems on a curved surface. Unsteady state probe permeameters may have an edge relative to the steady state instruments because of the expected very low permeability of low quality uncleaned chalk. Further, the unsteady state instruments, besides giving a conventional gas permeability, also returns a slip corrected (*Klinkenberg*) gas permeability that is not possible with the steady state instruments.

Pitfalls: Probe permeability measurements are performed on unconfined core material (no stress applied) and readings may appear optimistic compared to plug data measured at 400 or 800-psi confining stress. Otherwise, probe data may sometimes appear low due to the uncleaned state of the core. Comparison with core plug data can be done by an analytical solution (*Halvorsen and Hurst, 1990*) or by calibration with probe measurements on homogeneous RCA plugs.

Observe that probe readings may be biased near core edges and plug holes. Unexpected high (or low) readings may be screened from comparison with core photos and the core description.

Confining stress: In routine core analysis, a radial confining stress of 400 psi (2.8 MPa) to the Hassler type of core holder used in gas permeability measurement and the combination instruments have become the standard. However, other standard stress figures have been used, e.g. 800 psi. The advantage of a higher stress is to minimize variation in effective stresses during steady state slip corrected (*Klinkenberg*) gas permeability measurements (different mean pore pressures extending to 100 - 200 psi), or to allow higher transient pressures be used in un-steady state instruments.

Porosity

Boyle’s Law Helium porosity: A range of methods is available for determination of sample porosity in core analysis; for a detailed description, reference is made to *API RP40, chapter 5*. However, the great majority of porosity determinations made on North Sea chalk samples during the last approx. 30 years have utilized Boyle’s law single or double cell instruments with He as the injection gas for determination of pore volume or grain volume respectively. Bulk volume has been measured unconfined by Archimedes test from submersion in a mercury bath, or by using a mercury displacement pump, or by calliper techniques in combination instruments. The accuracy of porosity determination by these techniques is given in Table 1.2 that also lists the error on bulk volume determination alone, cf. *API RP40 (1998) chapter 5*.

Pitfalls: It should be observed that very low permeability chalks may require long equilibration times when measured for He porosity. Failure to reach equilibrium will result in erroneously low porosity and grain density figures, and grain density is therefore a quality check figure in chalk analysis.

Table 1.2: Measurement accuracy that should generally be expected from He-porosity and bulk volume determinations

Porosity	Range	Accuracy
Boyle’s Law, single cell (confined)	All plug and core sizes	≤ 0.3 porosity-%
Boyle’s Law, double cell (un-confined)	Plug size	≤ 0.3 porosity-%

Bulk volume	Range	Accuracy
Submersion in Hg	Plug size	± 0.01 cc
Submersion in other fluids (brine or oil)	All plug and core sizes	± 0.01 cc
Mercury displacement pump	Plug size	± 0.01 cc *
Calliper	All plug and core sizes	± 0.15 cc **

* often much higher due to trapped air in the sample cup.

** assuming a perfect cylinder shape.

Combination instruments: Some manufacturers produce instruments with sample changers or conveyor belts that feed plug samples of 25 or 38 mm diameter into a core holder for measurement of gas permeability and porosity. The measurement gas is Helium and the confining stress can be varied from the traditional 400 psi (2.8 MPa) to a client selected overburden stress. The porosity measured by such instruments is a Boyle's Law single cell He-porosity under confining stress. Bulk volume appears from automatic callipering or dimensions from laser type of instruments.

Pitfalls: Some chalk samples may be soft (slightly deviating from a perfect cylinder due to the grinding action of a rotating drill) or small chips may have broken off the edges of the plug, therefore the submersion method is preferred relative to the mercury pump and the calliper measurements/instruments when measuring the bulk volume.

Sample changers/conveyor belts may sometimes cause damage to fragile chalk samples.

Some service companies have used a very high overburden stress, up to or even exceeding 3000 psi (20.7 MPa) as a supplement to the conventional 400-psi (2.8 MPa) confining stress for routine core analysis. This caused permanent compaction of high porosity chalks, but may not have affected medium to low porosity chalk samples. However, some low quality chalks (Lower Cretaceous) have experienced very little chemical diagenesis due to a high content of clay minerals, and high confining stresses may easily cause damage to these samples. Besides the business value of overburden measurements on dry samples may be seriously questioned as wet (reservoir), chalk is known to have compaction properties quite different from dry chalk (*JCR-5, 2000* and *Fabricius et al., 2010*).

Grain Density

With Boyle's Law He-porosity and submersion in mercury as the preferred method of determining porosity (pore volume, grain volume and bulk volume), the grain density emerges from an additional determination of the dry weight of the plug after drying at 110 °C to remove adsorbed water. With the accuracy/precision figures given in the tables above and the weight from an electronic balance with a resolution of 0.01 g, grain density should be given to ≤ 0.01 g/cc on the 2nd or 3rd decimal for plug size samples of approx. 25% porosity.

Clean North Sea chalk consists of low-Mg calcite with a grain density of 2.71±0.01 g/cc. However, accessory minerals are present in all chalk formations, mainly silica with minor clay minerals, dolomite and pyrite. Assuming 15% silica (α-quartz) of density 2.65 g/cc in a chalk sample would lower the overall grain density to 2.70 g/cc. Small amounts of pyrite with grain density 5 g/cc would raise the measured sample grain density above 2.73 g/cc. When grain densities below 2.69 or above 2.73 g/cc is observed for chalk samples, it is recommended to check the mineralogy (from plug description and possible XRD mineralogy) or lab procedures (e.g. cleaning or drying state, instrument or operator errors) as part of the internal laboratory QC procedure.

Pitfalls: Some chalk formations with high content of expandable clays should be treated differently, cf. the section on cleaning and drying.

Insoluble Residue

Insoluble residue (ISR) in chalk formations is the solid compounds left over from a mild dissolution that removed all calcite from the sample. A chemical dissolution procedure that is gentle to fine grained delicate clay minerals have been described by *Jakobsen et al. (2000)*.

Approximate ISR figures for North Sea chalk reservoirs are: Tor Fm contains < 3 wt-% ISR, Ekofisk and Hod Fm < 15 wt-% ISR and the Lower Cretaceous Sola and Tuxen Fm in general < 50 wt-% ISR. RCA programmes on Ekofisk, Hod and Lower Cretaceous chalk should include determination of ISR at regular intervals along the core to help interpret reservoir quality and underpin RCA data.

The amount of ISR in chalk is important because it may have a serious effect on reservoir quality; the majority of the ISR fraction belongs to the fine clay size fraction (< 0.2 μm) and is therefore much smaller than the coccolith fragments making up the major part of the chalk formations. The amount of ISR should be given as a weight percent relative to the original sample before dissolution.

A major compound of the ISR fraction in the North Sea chalk fields can be silica that is present as disseminated nano size particles of α-quartz, in hardgrounds and in chert bands and layers (*Jakobsen et al. 2000, Lindgreen et al. 2011*). Silica should be determined by (semi-) quantitative XRD analysis and the amount given as a weight percent relative to the ISR fraction. Other ISR compounds are clay minerals (kaolinite, mixed-layers of illite-smectite, dolomite and pyrite). They may be determined from semi-quantitative XRD as well, and may form an independent check on the grain density determination from routine core analysis.

Fluid Saturation

Analysis of fluid saturations should be done as fast as possible after the core has been retrieved at the well site or received in the laboratory, and before other routine plugs are cut with coolants that may change the saturation or the wettability of the core as received.

When cutting plugs for fluid saturation or fresh state wettability analysis it is recommended to use compressed gas (nitrogen or air) or Blandol as a coolant, not kerosene or water. Due to the low permeability and soft nature of chalks, plugs can be drilled fast and with insignificant drying if a gas is used as a coolant.

Core contained in ordinary liner tubes will yield fluid saturations different from the reservoir cf. the pitfall section below. Pressure retained core analysis may in theory give downhole fluid saturations except for drilling fluid invasion that may be accounted for by using a tracer; T_2O and D_2O have been used, but D_2O is preferred for environmental reasons. Sponge core analysis (available in water wet or oil-wet designs) may yield more reliable results for water and oil saturations relative to the ordinary core preservation technique, but cannot account for the solution gas. Further details of these methods and filtrate invasion analysis can be found in *API RP40 (1998), chapter 4*.

A range of methods are available to determine fluid saturation; for a detailed description reference is made to *API RP40 (1998), chapter 5*. However, only two methods have been widely used among laboratories analysing chalk, and will be shortly covered here.

<u>Dean Stark distillation extraction:</u> A widely used method among the North Sea chalk core laboratories. Extraction may take a week for tight chalk, and daily recording of the produced water in the graduated trap is necessary to know when condensed water remains constant and the extraction can stop. The calculation of fluid saturation presumes that the water and oil density is known. If it is unknown, a value is assumed in the final calculation, usually 1.0 g/ml for the brine and 0.85 g/ml for the oil. The percentage of the plug pore volume that is not occupied by either water or oil is the gas saturation.

Table 1.3 gives the accuracy (internal laboratory experience) that may be obtained for different plug sizes and range of porosities under ideal conditions; i.e. with laboratory prepared fluids of well-known salinity and density and careful handling to prevent evaporation before the plug is placed in the extractor:

Table 1.3: Measurement accuracy that should generally be expected from Dean Stark extraction of RCA plug sizes

Fluid saturation by Dean Stark	Accuracy [saturation-%]

Plug porosity [%]	1" x 1.5" plugs	1.5" x 3" plugs
> 20	5	1
10 - 20	10	2
5 - 10	20	5
< 5	> 20	> 5

Certain factors might alter the stated precision of the fluid saturation determination. Loss of material during handling of the plug will result in an increase in the calculated oil saturation, and a similar decrease in the calculated gas saturation. This may occur for fragile for loosely consolidated rocks. As the lost material usually has a greater density than oil, it may happen that the estimated volume of oil and the measured volume of water all together take up more space than the actual pore volume after cleaning.

Solvent flushing/Karl Fischer titration

As an alternative to the Dean Stark extraction method a miscible solvent flushing may be carried out while the sample remains confined in a core holder, and the water content in the effluents determined by Karl Fisher titration. After extraction the core is dried (in place or detached from the core holder) and the pore volume determined by the relevant He-injection technique. The oil saturation is determined indirectly from weight difference relative to the final dry weight corrected for the water content, and is therefore sensitive to grain loss like the Dean Stark method.

The Karl Fisher titration should be performed as soon as possible after solvent flushing is completed to avoid excessive absorption of moisture from the environment, but handled carefully is very precise and measured water content should be within 2% relative from the true value (*API RP 40, section 5.4*).

Pitfalls: During pulling the core to the surface, it experiences pressure depletion, expansion of the oil phase and escape of solution gas. Thus, the fluid saturations at surface conditions will deviate from the downhole reservoir conditions but the effect on the separate saturations (water/oil/gas) is unknown to the analytical laboratory and is therefore not accounted for in the reporting of RCA data.

Cleaning and Drying

The preferred RCA method for cleaning of chalk plugs have been the hot Soxhlet reflux cleaning with methanol and toluene as the most used cleaning liquids. Extended cleaning for 2-4 weeks are often necessary due to the low permeability of chalk. Drying is normally done at 105-110 °C to remove adsorbed humidity and obtain the most accurate porosity and grain density figures. Grain density thus serves as a second order check on the cleaning and drying process because even small amounts of residual salt and oil (and cleaning liquids) will push the measured grain density below the clean calcite grain density of 2.71 g/cc. However, soxhlet cleaning does not clean the chalk plugs completely, but the effect on the grain density will normally be on the third decimal if proper cleaning for an extended period of time has been installed.

Ekofisk Fm and Lower Cretaceous chalk with significant amounts of expandable clay minerals may be cleaned differently using liquids with boiling point < 100 °C, e.g. acetone, chloroform or dichloromethane instead of toluene, and humidity dried at 60 °C and 40% relative humidity. Alternatively, a cleaning and drying study with SEM inspection can be initiated to investigate how delicate clay minerals are best preserved, e.g. *Pallatt et al (1984)*.

Fresh state plugs or preserved uncleaned plugs to be used for SCAL experiments should be flush cleaned in core holders using the cold flush, miscible liquids cleaning technique. If water based mud has been used when cutting the core , initial water flooding may be undertaken to remove polymers from the mud filtrate prior to the solvent cleaning (to avoid possible precipitates). In case wax is present, the temperature can be increased to ~ 50 °C during the solvent cleaning. Cleaning liquids may be methanol and toluene or a range of other solvents able to make chalk water wet before further SCAL experiments, cf. *Hirasaki (1990)*. A number of solvent cycles should normally be run with different flow rates and change of flow direction to clean the sample effectively. When the effluent is clear, free of salt and fines the sample is ready for SCAL testing.

Some old core material drilled with oil base mud may be difficult to clean. It is observed that additional cleaning with a sodium chloride brine of composition as seawater could be very effective in cleaning up plugs from such core material. The NaCl brine should be combined with the methanol cleaning cycle (miscible liquids). Recent research (*Austad et al., 2008*) have indicated that synthetic prepared seawater flushed through chalk plugs at temperatures > 90 °C is very effectively making chalk water wet.

1.6. Practical Application

RCA data (overburden corrected) are used when setting up a reservoir model, in reservoir simulators and in reserves estimation. They are used in log interpretation and planning of new wells and RCA data are the foundation for all SCAL tests.

1.7. Reporting Requirements

RCA data are normally collected in a hardcopy or electronic report with data presented in tabular and graphical format. The report should contain a description of the analytical methods as well as a statement of the accuracy and precision where relevant. Basic (raw) and calculated data should further be attached in digital format. A reporting format is given in *API RP40, chapter 8 (1998)*.

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2. Nuclear Magnetic Resonance (NMR)

2.1. Abstract

Laboratory Nuclear Magnetic Resonance (NMR) measurements on chalk core samples are used to determine several key petrophysical parameters, including porosity, pore size and fluid saturations. While the actual measurements are relatively simple to perform, considerable care in setting up a laboratory NMR system is required in order to obtain high-quality results. Results are used primarily in calibration and interpretation of NMR logs, and as independent estimates of pore volume and fluid saturations. NMR as a supplement to other SCAL measurements is still a subject of research.

2.2. Introduction

Nuclear Magnetic Resonance (NMR) measurements of relaxation processes are useful in core analysis in that they respond to the abundance of Hydrogen nuclei (H^1) in the sample and to the rate of the relaxation of the nuclei to an equilibrium state. While H^1 abundance can be linked easily to porosity and pore volumes, it is the rate information inherent in a NMR experiment that provides its unique contribution to reservoir rock characterisation. Relaxation rates are associated with the properties of the fluids, such as viscosity, and the interactions of the pore-filling fluids with the pore walls. It is this interaction term that provides insights into pore size and pore wall composition. Principles of NMR logging can be found in textbooks, e.g. *Coates et al. (1999)*.

NMR measurements of chalk are relatively easy to execute. Chalk generally have medium to high porosity, hence good signal strength when the pores are filled with water and/or liquid hydrocarbon. While the absolute size of the pores in chalk is small (in general less than 1 micron in pore throat radius), the relaxation rates measured on water-saturated samples are in the range of 20 to 100 msec. The addition of a light hydrocarbon liquid into the pores introduces a second distinct relaxation component in chalk reservoirs (*Howard and Spinler, 1995; Kenyon, 1997; Straley et al., 1995*).

Definitions

The NMR relaxation response of a single water filled pore can be expressed by the exponential decay of the transverse magnetization signal (*Grunewald and Knight 2009, Coates et al. 1999*):

$$M(t) = A_o \exp(-t/T_2) \qquad \text{(Eq. 2.1)}$$

where A_o is the initial signal amplitude at t=0, t is the time and T_2 is transverse relaxation time. A_0 is proportional to the number of hydrogen nuclei and therefore related to the fluid saturated pore volume of a rock sample. A single-exponential solution for the rate constant T_2^{-1} is generally satisfactory for chalk saturated with one fluid since most chalks are characterized by a fairly narrow range of pore sizes. Samples saturated with oil and water are characterized by two distinct relaxation components and can be evaluated with a bi-exponential model.

The T_2 value of a single water filled pore is proportional to S/V, the surface-to-volume ratio of the pore, and therefore to the pore body size. The measured T_2 relaxation time distribution of all pores in a rock sample may then be considered equivalent to the pore body-size distribution of the rock, which is different from the mercury injection method that measures the pore throat-size distribution of the rock.

2.3. Recommendations

1. Cylindrical plugs prepared for routine porosity – permeability measurements are preferred for laboratory NMR studies. Measurements can be made on “as-received” or “cleaned and resaturated” samples.

2. Samples from wells drilled with Oil-Based Drilling muds should be treated with caution. Plugs should preferably be cut from regions unaffected by filtrate invasion, and effectively cleaned.

3. Low-field NMR instrument should be tuned to resonance frequency for every sample, or a dedicated bulk fluid sample (about 20ml) of $MnCl_2$-doped water (T_1 and T_2 are about 50 - 100ms) may be used for this adjustment before a new sample set is measured. Pulse widths should be adjusted daily or when the type of sample is changed.

4. A set of intensity calibration standards should be prepared. Pure brine samples with precisely known volume can be accurately prepared each time a new plug set is measured. The instrument response to the measured fluid volume should be checked to ascertain linearity. Porosity can be determined from these calibrated measurements of pore volume.

5. An audit trail of weights and dimensions should be kept for each sample, especially when subjected to multiple measurements under different saturation conditions.

6. The sample should be centered in the instrument’s sensitive region. This can be determined by applying a 1D profile measurement or by using a standardized sample holder. Sample length should not exceed the length of the instrument’s sensitive region.

7. Acquisition parameters should be set for a suite of samples and left unchanged. Parameter settings should maximize the information collected without overwhelming the instrument’s capabilities. The recovery delay time should be sufficiently long enough to allow for full repolarization of the slowest relaxation component (i.e. 5 x T_1slow).

8. The number of scans collected per test should be determined either as a fixed number or as the number needed to achieve a pre-determined signal-to-noise level. If the number of scans is set to a fixed number, then the signal-to-noise level should be recorded for each sample.

9. Data processing, whether by simple single or bi-exponential functions or with multi-exponential fitting algorithms, should be standardized for a set of samples. The time-basis for multi-exponential inversions should cover the range of the experimental data.

10. Water and oil saturation estimates based on relaxation time measurements should account for small variations in hydrogen density (Hydrogen Index) between water and the oil used in the experiment.

11. Efforts to estimate surface relaxivity should use a single source of external pore size measurements (e.g. Mercury porosimetry, thin section image analysis, SEM-based image analysis) in order to ensure consistency.

2.4. Technology

Laboratory NMR measurements on rocks and core samples are best done at relatively low magnetic field strengths (e.g. 0.047 – 0.235 Tesla, equivalent to resonance frequencies of 2-10 MHz for H^1) that reduce the impact of paramagnetic impurities (e.g. Fe, Mn) on the relaxation processes. High-resolution NMR spectroscopy is best done at high fields, (e.g. ~4.7 Tesla or a resonance frequency of 200 MHz or greater). The lower resolution instruments are ideal for rock studies in that they can be adapted to detect only the hydrogen present in the pore-filling fluids and not any of the hydrogen found in mineral structures (e.g. hydroxyls in various clay minerals) that are characterized by very fast relaxation times. In addition, the low field permanent magnets have almost zero running/maintenance cost, allowing sufficiently large sample sizes at low magnet cost. Recent development of intermediate magnetic field strength instruments operating at resonance frequencies of 13-20 MHz, optimized for studies of carbonate rocks and shales *(Mitchell, 2014; Fleury, 2014)*, may be potentially interesting in characterization of low quality reservoir chalk with high content of clay and/or nano silica (and therefore having a significant nano porosity).

Most NMR instruments used in SCAL have a resonance frequency for H^1 of approximately 2.0 MHz (~0.047 T magnetic field), comparable to the values used in downhole logging tools. These instruments include a RF probe that acts as transmitter and receiver and have bore diameters that range from 40 to 100 mm. Probe designs for these instruments are highly variable. The two most critical parameters in the probe design are sensitivity to H^1 content and the speed at which RF pulses can be turned on and off. These two parameters generally work against each other, so probe design often has to balance the need of one parameter against the other. More sensitive probes allow for signal detection in samples with smaller pore volumes, either smaller samples or those with reduced porosity. The use of faster RF probes allows for the detection of faster relaxing H^1 nuclei, (e.g. those in smaller pores or more viscous fluids). These low-field instruments have suitable amplifiers, electronics and software so that a number of different data acquisition sequences can be run. Some instruments also have a single gradient amplifier that allows for measurements of diffusion and collection of a 1D profile along the length of the core.

Low-field NMR measurements can be made on samples with a wide range of preparation. While drilling cuttings can be used, the goal is to maximize the pore volume to be measured. The use of the standard core-analysis core plug is preferred since it makes comparison with conventional porosity and permeability measurements easier. NMR is a non-invasive and non-destructive for the sample technique, which ensures that this analysis can be made at practically any stage of a SCAL program. The only constraint is that the plug length does not exceed the length of the instrument’s sensitive region.

2.5. Practical Application

Laboratory NMR measurements are used to calibrate downhole NMR logs. The NMR T_2 distribution is related to the porosity and pore size distribution of the reservoir rock. Because T_2 values are related to the pore size distribution, a T_2 value can be selected below which the fluids are bound in small pores, including clay bound water, and above which they are expected to reside in larger pores and thus being mobile fluids. This T_2 value is called the T_2 cut off and it does require a full relaxation time distribution. The T_2 cut off can be determined from laboratory NMR measurements on fully water saturated core plugs from the logged formations, followed by drained measurements on the same plugs. The de-saturation is normally obtained by centrifuging the plugs at a specified air-brine capillary pressure.

The fluid saturations can be estimated from laboratory NMR relaxation measurements with either the results of a relaxation time distribution calculation or a simple bi-exponential fit (*Howard and Spinler, 1995*). The water and oil relaxation components are very distinct for water-wet chalk, with water-filled pores characterized by relaxation times between 10 and 50 milliseconds and the light oil relaxing around 1 second. The intensity of each component is directly related to the abundance of water and oil in the sample (*Howard et al., 2002*). The only correction to the intensity results is to account for slight differences in Hydrogen Index of the oil relative to the water. Shifts in the relaxation time distributions for chalk saturated with oil and water can be used to estimate wettability (*Howard, 1998; Howard and Spinler, 1995*).

The best practice in any laboratory NMR testing is to use the same core plug prepared for conventional porosity and permeability measurements (*Basan et al., 1994*). This allows for direct comparison of NMR signal intensity with independent porosity measurements and the use of the relaxation time distribution as the basis for any permeability estimates (*Kenyon, 1997; Howard et al, 1995*). Samples from wells drilled with oil-based drilling muds should be cleaned prior measurement, with the recognition that some OBM additives cannot be removed by normal cleaning techniques (*Shafer et al., 2004*). The sample should be saturated with a liquid or combination of liquids. Formation water is preferred for basic porosity and relaxation time distributions. Mineral oil is often a suitable analogue for the reservoir hydrocarbon as long as the viscosities are matched. Mineral oil (e.g. decane, kerosene) has the advantage of compositional simplicity that is reflected in single-exponential relaxation behaviour when measured in bulk (*Howard and Spinler, 1995; Straley et al., 1995*).

The relaxation time T_2 and thus the measurement of NMR porosity are very dependent on the temperature of the sample. Therefore, it is important to indicate at which sample temperature the measurement has been performed.

A set of standards are recommended to calibrate the instrument’s determination of fluid volume. Since NMR signal intensity is directly proportional to the number of H^1 nuclei in the pore-filling fluids, a simple linear relationship can be determined for a set of fluid standards of varying volume. Two aspects characterize the best fluid standard suite; it covers the range of anticipated fluid volumes and the relaxation time of the sample. Since most water-saturated chalk samples have relaxation times of less than 100 milliseconds, distilled water is not a good choice; a $CuSO_4$ solution can be tailored to have a comparable relaxation time (*Straley et al., 1997*), or preferably, a reference can be prepared from the correct formation fluid (brine). A set of water standards that range in volume from 1 to 25 cm^3 can then be used to generate a calibration curve for interpretation of the measured intensities. The relative error in estimating fluid volume from NMR is roughly 2-3% (internal laboratory experience).

Using NMR to estimate absolute permeability and wettability is still subject of research.

2.6. Reporting Requirements

A summary report for each sample should include a listing of key parameters for every experiment run and the initial results from those tests. The parameters include the settings for the amplifier gain, the widths of the P90 and P180 pulses, the recovery delay time, the echo spacings (or tau) and number of echoes, the number of co-added scans, and the resonance frequency. The list of initial results should include the signal intensity, the signal-to-noise ratio, and a single-exponential estimate of the relaxation rate. The multi-exponential relaxation time distribution should include information on the nature of the time basis used in the calculation, the type of smoothing or regularization in the inversion and an estimate of the goodness-of-fit.

The fluid volume – NMR intensity calibration curve used in data reduction should be included with the report.

2.7. Pitfalls

Observe that pore volume and fluid volume are approximately the same only for 100%-saturated samples. NMR signal amplitude is proportional to the fluid volume, also for desaturated cores.

Core samples that exceed the length of the instrument’s sensitive region can be measured, but the calculated NMR porosities will be strongly underestimated, while the T_2 distribution will represent only the part of the sample within the sensitive region.

Error in NMR porosity may be due to lack of temperature equilibration between the reference fluid and the core samples before measurement.

Extensive calibration is required, while error bars are rarely quoted.

2.8. References

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3. Core Water Salinity and Simulated Formation Water

3.1. Abstract

Information about the chemical composition of the downhole formation water can be obtained from produced water that has not yet been mixed with injected water, and from fresh core samples if a fair amount of water can be obtained for analysis and the water sample is uncontaminated or if a previous spiking of the drilling mud allows a reasonable precise calculation of the original formation water composition. Dry (un-cleaned) core may sometimes be the only option to obtain an estimate of the initial formation water composition for mature fields. Formation water salinity and composition data are used in core analysis and log interpretation, and failure to use the right formation-water composition may affect electrical measurements, core wettability, sensitive clay minerals (swelling) and initiate fines migration.

3.2. Introduction

Low quality chalk often contains minor or even significant (Lower Cretaceous) amounts of clay minerals that may be sensitive to the formation water salinity and chemical composition, and ions like K^+, Mg^{2+} and Ca^{2+} is important in stabilizing clay minerals in sedimentary rocks. The salinity and composition of the formation water (and injection water as well) may further affect the inter-facial tension in the fluid-rock system (*Anderson 1986, Puntervold and Austad 2007*).

Analysis of core water is treated in American Petroleum Institute (API) publication *”Recommended Practices for Core Analysis, RP 40”, 2nd ed., chapter 7 (1998)*. Guidelines for preparation of brines and measurement of resistivity in SCAL experiments can be found in a *SCA Survey, Part 1 (1990)*.

Definitions

Salinity, also known as Total Dissolved Solids (TDS), is an expression of the amount of dissolved salt in the core water (cationic species + anionic species + elements); the unit of measurement is [mg/kg], i.e. mg of salt dissolved in 1 kg of solution, which may also be expressed as [ppm] by weight. TDS may also be given by volume, i.e. in mg of salt in 1 L of solution. Salinity is sometimes calculated from the amount of Cl^- present in the core water and is then given as an equivalent amount of NaCl.

Conductivity of an electrolyte solution is a measure of its ability to carry an electrical current; the SI unit of measurement is [S/m], i.e. Siemens per meter, and unless otherwise stated refer to a standard temperature of 25 °C. Vintage literature sometimes gives the conductivity in [mhos/m]; the conversion is simple

$$1\ [\text{mho/m}] = 1\ [\text{S/m}]. \qquad \text{(Eq. 3.1)}$$

Resistivity of an electrolyte solution is a measure of its resistance towards a flowing electrical current; it is the reciprocal of conductivity; the SI unit of measurement is [Ωm], i.e. ohm meter.

Density of the pore water is the mass per unit volume of the fluid contained in the void space (pore volume) of a core sample; the SI unit of measurement is [kg/m^3], the cgs unit of measurement is [g/cm^3].

3.3. Recommendations

1. Core water-salinity measurements should be planned in combination with a coring operation.
2. Plug samples for analysis should be taken as fast as possible after the core has been retrieved at the drilling rig.
3. Plugs should be taken from the centre of the core, sealed airtight and kept in a cold store (not freezer) until analysis; it is recommended that the drilling mud be spiked with a Deuterium (or Tritium) tracer that allows correction for mud filtrate contamination.
4. The preferred method of extraction of core water from fresh state plugs is by centrifuge or immiscible fluid flush; if water cannot be collected by these methods, the second choice is by water extraction that is less accurate than the preferred methods cf. the pitfall section.
5. Old, dry and uncleaned core may be analysed for salinity by the water extraction method, but interpretation of results may be questionable, and the method is not recommended.
6. Sulphate in the original formation water and in injected seawater may affect the wettability of reservoir chalk particularly at higher reservoir temperatures; simulated formation water and seawater used for advanced core analysis should include sulphate in the same concentration as observed for the original formation water or, for seawater, as given in Table 3.1 below.
7. Ageing for 2 days at reservoir temperature in simulated formation water is recommended for cleaned SCAL plugs that should be saturated before further experiments are carried out, even if the temperature should then be reduced to room conditions.

3.4. Technology

Methods to extract and analyse water samples from core material are covered in *API RP40 (1998)*:

- Centrifugation
- Immiscible fluid flush
- Water extraction of core salt
- Determination of core water salinity

Advantages and limitations of the different methods are also covered and should be observed when designing a core water study.

Preparation of synthetic formation brines and pure sodium chloride solutions for SCAL work, and measurement and calculation of their resistivities are further treated by SCA in *"Guidelines for measurement of electrical resistivity", Part I (The Log Analyst, 1990*).

The preferred methods for extraction of core water are centrifugation and immiscible fluid flush of fresh core samples. Samples for salinity measurement including LRQC should be taken as fast as possible after the core has been received at the rotary table, drilled from the centre of the core and sealed airtight until they can be measured. During transportation or storage, the samples should be kept in a cold store or cooling bag. Spiking of the drilling mud may improve the quality of salinity and chemical composition analysis to obtain the best estimate of the downhole formation-water composition.

A disadvantage with the centrifuge is the long spinning time needed to extract water from low permeability rocks with risk of evaporation due to heating. Unconfined samples may break at high rotational speeds.

A disadvantage with the immiscible fluid flush method is the long flushing time and high differential pressure needed to displace core water from low permeability plugs. This may also imply a high confining stress that may further reduce the permeability in the downstream end of the sample where the effective stress is at a maximum.

If water cannot be obtained from the above methods, drying and crushing of the plug sample, followed by extraction of salt with pure water, may be the only option cf. *API RP40*. This method is also the only available for analysis of old dry core, but observe the pitfall section.

3.5. Practical Application

A close estimate of the formation water composition is an important piece of information when planning a SCAL study, in log interpretation for calibration of electrical logs and in pore pressure vs. depth calculations where the density of the formation water can be estimated from the chemical composition or from a salinity measurement.

A simulated formation water for SCAL experiments on chalk should match as close as possible the in situ formation water electrolyte concentration and contain at least the major elements Na, K, Ca, Mg and preferably also Sr, and be balanced with respect to bi-carbonate (HCO_3^-). If bi-carbonate has not been determined from chemical analysis, cleaned and mildly crushed chalk (mm size) from the core to be analysed should be added in small amount to the simulated formation water to secure that the water is in equilibrium with respect to bi-carbonate. After 24 hours, the simulated formation water should be filtered and de-aerated before use.

North sea chalk fields: With very few exceptions have original formation water TDS concentrations in the interval 30,000 - 110,000 mg/L. Potassium is generally low < 500 mg/L; Calcium can be very high in a few fields (e.g. Ekofisk and South Arne) having relatively high initial reservoir temperatures >100 °C.

Sulphate can be present in the rock matrix and formation water in some chalk fields in quantities shown to affect the wettability (*Puntervold and Austad 2007*). When SCAL experiments are conducted on chalk from these fields, sulphate should be added to the simulated formation water in concentrations equal to sulphate determined from the produced water or core water (before mixing with injection water and corrected for mud filtrate contamination).

Seawater is used in water flooding of the North Sea chalk fields. The concentration of sulphate in seawater is much higher than found from formation water in the chalk fields, and it is therefore important that sulphate be added to the simulated seawater used in SCAL experiments. A general seawater formulation with sulphate used by the oil companies operating in the North Sea is attached below.

It is suggested that SCAL experiments on cleaned and dried chalk plugs that involves saturation with simulated formation water during the analytical procedure, also includes an ageing step, e.g. 2 days ageing at reservoir temperature saturated with simulated formation water. If the temperature is then reduced to room conditions, flood the sample with at least one PV of fresh brine to displace the ageing liquid.

3.6. Reporting Requirements

Data sheets for core water analysis should give dates of coring, sampling and analysis. Further core diameter, geological formation and mud type should be listed. Analytical method and results should be given with an indication of the expected analytical precision. Data on conductivity, resistivity and density must contain the temperature of measurement. In SCAL studies the composition, density, viscosity and resistivity/conductivity of the simulated formation water must be given for the specific tests.

3.7. Pitfalls

Speed is essential in sampling from fresh LRQC core because strong capillary forces, water imbibition and chemical diffusion will seek to transport mud filtrate into the centre of the core and thus change the original formation water composition in less than 1-2 weeks after the core has been cut *cf. Springer (2003)*.

KCl mud (water base) have been used extensively in coring in the North Sea chalk fields, and the concentration of Potassium measured in the extracted water may give an idea of the degree of contamination from the drilling fluid that can indeed be very high. The concentration of Potassium in uncontaminated formation water from the chalk fields is always < 500 ppm K but varies from field to field and Potassium can only be used as a secondary tracer.

It is notoriously difficult to obtain reliable formation water salinity and composition data from dry uncleaned core material that has been stored for months or years (*API RP40, p. 7.16*). During drying, formation water moves towards the core surface forming a rim of highly concentrated core water that further mixes with mud filtrate water and finally precipitates the salt due to evaporation. In the opposite direction away from the surface is chemical diffusion seeking to establish equilibrium as long as a continuous water phase exist (*Springer, 2003*). Plug samples taken from the centre of an old dry core will therefore not be representative for the formation water composition; rather a long plug or slab taken perpendicular to the core axis should be used for extraction. However, the analyst is left with the complication of correcting the data for mud filtrate contamination and estimating the original S_w of the analysed core section.

Contamination from degraded (oxidized) minerals like pyrite that dissolves during the clean water leach of a crushed sample will give high sulphate readings and the now acid leach water will dissolve calcite and thereby enhance divalent ion readings of Ca, Mg and Sr (*Pan, 2005*).

Table 3.1: Chemical composition of seawater from the North Sea

Element	**Conc.**	**Compound**	**Compound per litre of solution**	**Conc.**
	[mg/L]		**[g]**	**[mol/l]**
Na total	11090			0.4824
Na^+	9713	$NaCl$	24.692	0.4225
Na^+	46	$NaHCO_3$	0.169	0.0020
Na^+	1331	Na_2SO_4	4.111	0.0579
K^+	408	KCl	0.778	0.0104
Mg^{++}	1370	$MgCl_2, 6H_2O$	11.460	0.0564
Ca^{++}	434	$CaCl_2, 2H_2O$	1.592	0.0108
Sr^{++}	6.8	$SrCl_2, 6H_2O$	0.021	0.0001
Ba^{++}	0	$BaCl_2, 2H_2O$	0.000	0.0000
Cl^-	20119			0.5675
HCO_3^-	123			0.0020
SO_4^{--}	2780			0.0289

TDS $_{(\text{by volume})}$: 36331 mg/L

TDS $_{(\text{by weight})}$: 35514 mg/kg

Density $_{(@\ 25\ °C)}$: 1.023 g/cm^3

3.8. References

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4. Screening of Samples

4.1. Abstract

The objective of the screening exercise is to pick the samples that qualify for a specific advanced core analysis test. Careful geological description will help selecting samples from a certain chalk lithology, and analysis of X-ray CT-images will characterize and quantify the internal matrix texture that is not visible from outside inspection by the naked eye. In addition, a correlation has become available that allows quantification of heterogeneity in a core plug through analysis of X-ray CT images. SCAL plugs can then be selected based on a heterogeneity cut-off value.

4.2. Introduction

Cores cut from the North Sea chalk fields may represent more than one lithological formation/unit and any pre-selection of samples for SCAL shall therefore involve a collaboration between a geologist, a petrophysicist and a reservoir engineer, i.e. between the client and the service company before samples are sent for X-ray CT-screening. Populating flow units with core plugs to collect basic flow data should account for the field development under consideration: gravity segregation may easily dominate flow patterns. Reservoir depths not likely to be affected need less data, while the depths that will be flooded will benefit from a more detailed characterisation.

The full potential in X-ray CT-imaging is not always realized by service companies in terms of positioning of samples, selecting homogeneous samples and samples free of impeding structures in electrical and flow experiments, a topic especially important in analysis of LRQC.

X-ray CT scanning is used on core and plugs because there is business value in

- Representative core and samples
- Core damage assessment (see e.g. *Gilliland and Coles 1990; Bartko et al. 1995; and van der Zwaag et al. 1997*)
- Plug homogeneity

4.3. Recommendations

Methodology

1. A pre-screening of plugs is recommended, based on geological core description, plug description and core photos, to select the most homogeneous plugs for a closer X-ray CT-screening. Select more samples (at least 50%) than needed for the final SCAL study.
2. For SCAL measurements, plugs need to be homogeneous irrespective of the heterogeneity of the reservoir. This is due to the fact that the interpretation of the measurement data, be it with analytical calculations or through interpretation-by-simulation, implicitly assumes that the plugs are homogeneous
3. Homogeneity should be assessed through analysis of the digital information in X-ray CT-images (so-called DICOM formatted images). Recently, a correlation has been derived (*Maas and Hebing, 2013*) to quantify heterogeneity using the standard deviation in the DICOM images in combination with poro-perm data of the plugs.
4. Heterogeneous reservoirs need special sampling protocols to ensure that the heterogeneities are captured by a sufficiently large selection of individually homogeneous samples. No industry accepted protocols exist today to quantify the required number of samples based on reservoir heterogeneity.

X-ray CT Scanning

1. Ensure that the X-ray CT images are made available in DICOM format; images in jpeg, tiff or similar cannot be analysed for digital information.
2. Grey-scale settings depend on individual choices made by the CT operator and therefore may give a false impression of homogeneity. Consequently, visual inspection of the DICOM images is not recommended to assess homogeneity.
3. A good first impression (only) of homogeneity can be obtained when analysing the DICOM images with e.g. Sante DICOM viewer free, with a “level” setting close to the average HU value in the image and a “width” setting of 200. See for detailed procedures the *SCA paper by Maas and Hebing (2013)*.

4.4. Pre-selection of Chalk Samples

Any SCAL study starts identifying the relevant chalk formation (Ekofisk, Tor etc.) and the appearance of cores and plugs. The selection of homogeneous plugs may benefit from reviewing core descriptions, core photos and plug descriptions accompanying the routine core analysis report. Such information will often be present before a SCAL study starts.

Description and classification of chalk cores and plugs have been treated previously in a JCR-4 project (*Fritsen, 1996*). The project strived to establish a common understanding among the North Sea oil companies of chalk description, and introduced a description system covering among others rock texture, composition, layering, bioturbation, secondary minerals, stylolites and fractures. A spreadsheet was also introduced to simplify the plug description and make digitization possible. A copy of a partly filled in spreadsheet (Figure 4.1) is attached with the Appendix to this chapter.

By use of the fairly detailed spread sheet data, a pre-screening of plugs with undesirable structural or compositional elements, as e.g. shown in Figure 4.2 in the Appendix below, can easily be carried out before the remaining plugs are sent for X-ray CT-screening.

4.5. Technology

An X-ray CT scanner consists of a rotating source and detector arrangement, with a sliding sample table at the centre of rotation. One operating option is to keep the source and detector stationary and move the table. X-ray absorption images can be taken at a sequence of table positions, e.g. at every mm. The resulting multi-line image is called a “topogram” and would look like a standard X-ray photograph of a core or core plug. Another option is to rotate the source and detector while the table is kept stationary. Absorption images are now taken at a sequence of angular positions. The scanner software will employ a back-projection algorithm to reconstruct an absorption image of a slice through the sample. This image is called a “tomogram”.

Topograms are displayed with a grey or colour scale, but the scale itself is not calibrated. Tomograms are calibrated into “Hounsfield values” (see e.g. *Maas and Hebing, 2013 and references therein*).

Slice thickness in tomograms can be chosen by the operator and may run from 1 to 10 mm or so. Tomograms show usually sub-millimetre resolution. Standard image size is often 512 x 512 pixels, with a data collection and imaging time of less than 10s per image or so. Images are stored in “DICOM” formatted files that can be viewed by dedicated software.

The above information pertains to medical scanners often used for core and core plug scanning. Various research facilities operate “micro X-CT” scanners that have resolutions down to tens of micro-metre or better. Slice thicknesses are also much smaller than in medical CT equipment. In view of limitations on the maximum number of pixels that can be managed practically in back-projection algorithms, micro X-CT scanners have a field of view that is much smaller than in medical scanners. For real micro-metre resolution, the volume of investigation is often only a few mm^3. Data collection time may prohibit large number of samples to be scanned in a reasonable time span.

All X-ray CT scanners suffer from beam hardening artefacts, due to halo generation (“cupping effect”) in the reconstruction algorithms at any transition of high-low X-ray “density”. Medical scanners are optimised to correct for that, given standard bone-tissue contrasts. This optimisation is insufficient for core and core-plug scanning. As a result, X-ray images of core plugs show artificial high-density lining around the plug, obscuring possibly heterogeneities occurring close to an edge (*Maas and Hebing, 2013*).

4.6. Practical Application

Ideally, whole core is scanned in topogram mode to select the best locations for drilling homogeneous core plugs. If whole core scanning is not possible, a surplus of core plugs should be drilled to account for dropout of core plugs not meeting the cut-off for homogeneity. In our experience, more than 50% of plugs not pre-screened by topograms will need to be discarded.

Again ideally, plugs need to be cleaned and dried before CT-scanning for tomograms, because fluid content has a strong impact on the Hounsfield (HU) values. In the case that cleaning-and-drying cannot be done before scanning, again a surplus of plugs should be drilled to account for increased drop-out.

CT scanning should preferably be conducted at high voltage and current settings of the X-ray source (typically 125kV, 500mA, 1000ms), limited slice thickness (2.5 mm) with multi frame mode if available (4 slices of 2.5mm each, so effectively scanning 10mm total thickness per "single" slice).

As discussed above, beam hardening may interfere with the analysis of medical images. Beam hardening may be alleviated by encapsulating the samples during scanning with X-ray absorbing "shells", to reduce high-low X-ray density contrasts around a plug. Such shells effective "pre-harden" the X-ray beam (*Maas and Hebing, 2013*).

Plugs should be scanned in tomogram mode to collect two longitudinal cross-sections at right angles to each other (see *Maas and Hebing 2013 for details*). In co-operation with the reservoir engineer, a maximum acceptable permeability variation will be established that is assessed through statistical analysis of the HU values in each tomogram. For that, the images need to be available in DICOM format. Tiff, jpeg and other formats essentially "freeze" the grey-scale and blocks statistical analysis of the HU data.

Several free-ware viewers are available for DICOM images: Sante DICOM viewer free, eFilm, ImageJ. Preferred grey-scale setting to pre-screen the images is to have the centre level of the scale set close to the average HU value in the image and the scale width set to 200 HU.

Image analysis should be conducted over a rectangular region of interest (ROI), somewhat away from the edges. The image analysis needs to be combined with poro-perm data from RCA (see Chapter on Routine Core Analysis), to convert standard deviation of HU in an ROI into a permeability variation (see for details of the whole procedure *Maas and Hebing, 2013*).

4.7. Reporting Requirements

DICOM formatted digital data, including settings for kV, mA, slice thickness, sample ID in image, mean HU and standard deviation per image, DFOV ("Display Field Of View").

4.8. Pitfalls

Core plugs for SCAL need to be homogeneous, irrespective of the heterogeneity of the reservoir. Data interpretation, analytical or through simulations, implicitly assumes homogeneity. Heterogeneity on reservoir scale needs to be addressed through judicious choices of homogeneous core plugs (see also *Zweers et al. 1999*) and should not be seen as a reason to do SCAL on heterogeneous plugs.

Statistically, the larger the sample the more it is unlikely that the sample can meet a given homogeneity criterion. Therefore, in practice, whole-core may well be unfit to meet “best practice” quality standards.

Partial volumes can cause underestimates of heterogeneities, nodules. High and low density features may appear exaggerated.

Disseminated (evenly distributed) pyrite may increase the standard deviation in HU in the images that would translate into a large apparent permeability variation. On the other hand, pyrite in chalk may affect electrical measurements.

Beam hardening artefacts may obscure local heterogeneities close to the edge of a sample, while a local distortion very close to in- or out-flow ends can severely impact flow (*Maas and Hebing, 2013*). Beam hardening needs to be optimised for core plug scanning, e.g. by encapsulating the samples in the scanner with high-density “shells”.

Grey-scale window settings can be very deceiving and may hide or exaggerate heterogeneities. The digital images need to be objectively analysed (*Maas and Hebing, 2013*)

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4.10. Appendix

JCR plug description scheme

Well: **Field:**

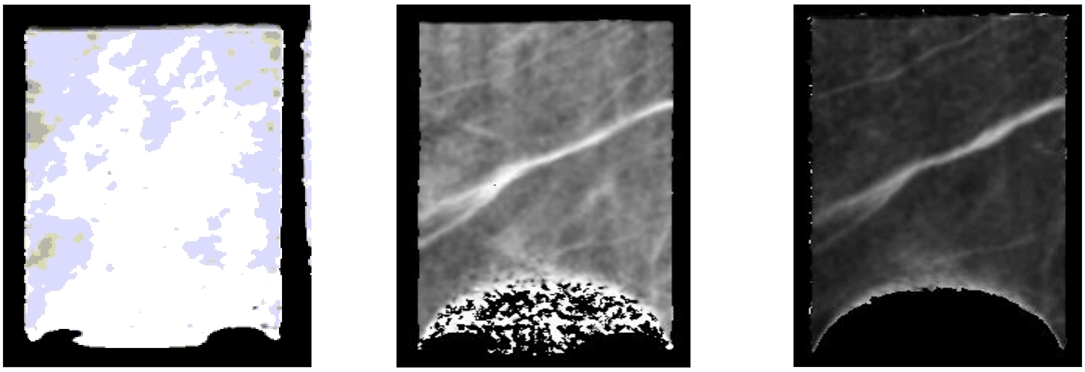
Sheet no.: **10**

Geologist: **Nis**

Plug size Ø = 1½''			**Depositional fabric**									**Composition**									**Stratification**								
			Rock type									Matrix			Grains						Bedding				Lamination				
Plug	Core depth	Lithotype	M	Mf	Mc	Wf	Wc	Pf	Pc	Gf	Gc	Ch	Arl	Arh	Gn	Gsh	Gch	Gsk	Gct	Go	Bn	Btn	Bm	Btk	Ln	Lpl	Lun	Lx	Lg
no.	m/ft.		1	2	3	4	5	6	7	8	9	1	2	3	1	2	3	4	5	6	1	2	3	4	1	2	3	4	5
249	7236.58	MaMu	1									1			1						1				1				
59V	7237.08	MaMu	1									1			1						1				1				
250	7237.83	MaMu	1									1			1						1				1				
251	7238.83	MaMu	1									1			1						1				1				
252	7239.83	MaMu	1									1						4			1				1				
253	7240.83	MaMu		2	3							1					3	4			1				1				
254	7242.17	MaMu	1									1						4			1				1				
60V	7242.42	MaMu	1									1			1						1				1				
255	7243.75	MaMu	1									1			1						1				1				
256	7244.75	MaMu	1									1			1						1				1				
257	7246.00	MaMu	1									1			1						1				1				
61V	7247.00	MaMu	1									1			1						1				1				

Plug	**Deformation structures**						**Bioturbation**				**Hardness**					**Stylolites**				**Secondary minerals**						**Fractures**							**Comments**
							Trace fossils																										
Plug	Dn	Dfl	Dfh	Dcl	Dch	Ds	Tn	Tl	Tm	Th	Hvs	Hs	Hm	Hh	Hvh	Sn	Sl	Sm	Sh	Mn	Mca	Mpy	Mdo	Msi	Mo	Fn	Fo	Fm	Fhl	Fhh	Fs	Plug	
no.	1	2	3	4	5	6	1	2	3	4	1	2	3	4	5	1	2	3	4	1	2	3	4	5	6	1	2	3	4	5	6	no.	
249	1							2							5		2					3							4			249	owh, 2S
59V	1						1								5			3				3							4			59V	owh, 3S
250	1							2							5				4	1									4			250	owh, 4S
251	1						1								5			3		1									4			251	owh, 2S, 1 mic S
252	1						1								5		2			1									4			252	owh, GIn, 2S
253	1							2							5		2			1						1						253	owh, 2S
254	1						1								5	1				1									4			254	owh
60V	1							2							5		2					3							4			60V	owh, 1S
255	1							2							5		2					3							4			255	owh, 1S, 1 mic S
256	1							2							5			3				3							4			256	owh, 2S, 1 mic S
257	1								3					4	5	1				1									4			257	owh, biotex
61V	1							2						4	5	1						3							4			61V	owh

Figure 4.1: Example of a RCA plug description scheme for chalk introduced during JCR-4 (Fritsen, 1996).



P_C (bar)	2.0	2.5	3.5
Mean $S_{W,\text{ IMAGE}}$	0.93	0.51	0.21

Figure 4.2: The business value of sample screening before SCAL testing. Oil-water capillary pressure drainage on porous plate (bottom) with quantitative 2D saturation imaging by CSI NMR. Distortion of the NMR-signal in the bottom of the images is due to the porous plate. Single sample experiment in Perspex core holder at 400 psi confining P. Duration of experiment 3.5 y. Tor Fm chalk, Φ ~ 0.3, k_{∞}~ 1 mD. (Credits: Olsen et al., 2001).

At P_c = 2 bar (left hand image), slightly above the entry pressure of 1.7 bar, an irregular fluid distribution is observed with grey areas having sporadic $S_o \approx 0.25$. At P_c = 2.5 bar (centre image) the oil phase is now more uniformly distributed, but a network of healed hairline fractures has emerged particularly in the centre of the plug with S_w close to 1 (white). At P_c = 3.5 bar (right hand image) the high S_w of the centre healed hairline fracture still persist. Healed hairline fractures are known from flow experiments to sometimes impede flow, forming shadow areas before and after the hairline. Hairlines are clearly visible from X-ray screening images as well.

5. Electrical Resistivity Measurements

5.1. Abstract

Electrical measurements on core samples are performed to underpin downhole log interpretation to determine hydrocarbons in place based on Archie's equations (*Archie, 1942*). These measurements may be severely affected by scale effects that result from the sample size used in core analysis, and failure to fulfil the mathematical/physical assumptions behind Archie's equations (*Lyle & Mills, 1989*).

Electrical resistivity measurements may further be used in other SCAL tests to check for stable fluid distributions in e.g. Steady State relative permeability and Porous Plate capillary pressure experiments.

5.2. Introduction

Archie's equations are convex functions of porosity and water saturation and, when used in core analysis, subject to scale effects (homogeneity of the porous network and the fluid distribution). It can be shown that for the common 2-electrode laboratory measurements, the Formation Factor and Resistivity Index recorded at non-uniform conditions will always be higher than the true values measured at uniform saturation and homogeneous porosity distribution along the core (*Lyle & Mills, 1989*). Uneven saturation distributions have been demonstrated for both porous plate and continuous injection (RICI) experiments if proper planning of the experiment is not done by numerical simulation, and this will affect RI measurements at both high and low water saturations (*Maas et al. 2000 and 2011*). Chalk is no exception and Archie's saturation exponent 'n' is the poorest determined petrophysical parameter contrary to the fact that chalk is among the most homogeneous reservoir rocks. Refer to the Pitfall section for further details.

In 1988 the Society of Core Analysts (SCA) conducted an inter laboratory electrical resistivity study on sets of standard core plugs distributed to 25 core laboratories worldwide. The results showed unsatisfactory large scatter in the measured Archie parameters and brine resistivities. This encouraged the SCA to establish a set of guidelines Part 1-4 and a suggested reporting format for measurement of electrical resistivity of core samples that was published in *The Log Analyst (1990)*. In many respects, this can still be considered the state of the art in preparation, handling, measurement and reporting of brine resistivity, Archie's Formation Factor and Resistivity Index.

Definitions

In a “clean” formation (non-shaly) Archie’s Formation Factor 'F' and Resistivity Index 'RI' is given by:

$$F = \frac{R_0}{R_w} = \frac{a}{\emptyset^m} \qquad \text{(Eq. 5.1)}$$

$$RI = \frac{R_t}{R_0} = \frac{1}{S_w{}^n} \qquad \text{(Eq. 5.2)}$$

The Cation Exchange Capacity (CEC) is the quantity of cations that a clay mineral can accommodate on its negatively charged surface, expressed as milli-equivalent (meq) per 100 g of sample material. Alternatively, the CEC may be expressed in terms of its contribution per unit pore volume, Q_v, as calculated from the eq. below (*Keelan and McGinley, 1979*):

$$Q_v = \frac{CEC\ x\ (1-\emptyset)x\ GD}{100\ x\ \emptyset} \qquad \text{(Eq. 5.3)}$$

R_0 = resistivity of sample @ $S_w = 1$ [ohm-m]

R_t = resistivity of sample @ $S_w < 1$ [ohm-m]

R_w = resistivity of formation water (brine) [ohm-m]

S_w = water saturation [fraction]

a = constant

m = cementation exponent

n = saturation exponent

Ø = porosity [fraction]

GD = dry grain density [g/cm^3]

P_c = capillary pressure [Pa] (but bar and psi used frequently)

C_o = Conductivity of an electrolyte saturated plug sample in [S/m]

C_w = Conductivity of the saturating electrolyte (formation water) in [S/m]

5.3. Recommendations

1. Laboratories and anyone commissioning an electrical resistivity study should be aware of and in general comply with the "Guidelines for measurement of electrical resistivity samples and reporting" set out by the SCA and published in *The Log Analyst (SCA, 1990)*.
2. It is recommended to use a conductivity/resistivity meter and measurement cell from a recognized commercial instrument company when determining the resistivity of simulated formation brines. Certified KCl standard solutions (not NaCl solutions) are offered by the instrument companies for proper calibration of the cell, and can be used with confidence for brine concentrations to approx. 200,000 ppm.
3. Measurement of electrical resistivity of brines and plugs should be conducted in temperature-controlled rigs/ovens, or alternatively in a temperature controlled environment at 25 °C that allows direct use of the equations for calculating brine resistivity/concentration published by the SCA in *The Log Analyst (SCA, 1990)*. A temperature log should be recorded and included with the lab report.
4. Measurement of electrical resistivity of plugs should preferably be conducted in single resistivity cells at a suitable confining stress to secure a good electrode contact and minimize handling/grain loss errors. Measurement of RI at room conditions (unconfined) after drainage in multi-sample pressure pots are not recommended.
5. Samples that should undergo electrical resistivity measurement as part of a SCAL programme should preferably by cleaned and dried before they are X-ray CT-screened, and the most homogeneous selected for measurement, cf. the Chapter on Screening of Samples.
6. Based on porosity data measured on routine plug sets (cleaned), the maximum possible range in porosity is looked for to obtain a precise determination of Archie's cementation exponent.
7. It is recommended to use numerical simulation in planning of porous plate RI experiments to obtain the most homogeneous saturation distribution, cf. the Chapter on Numerical Simulation.
8. When measuring RI from simple two or four-electrode configurations, drainage equilibrium should be documented from production data and stable impedance readings at minimum phase angles. A uniform saturation distribution may further be demonstrated from external saturation monitoring by e.g. X-ray or gamma ray techniques.
9. Unconventional methods (to the Industry) may be preferred for LRQC to determine the saturation exponent within a reasonable time, e.g. non-equilibrium RI measurements with saturation monitoring and mathematical modelling, or multi-electrode configurations with saturation monitoring to derive an un-biased RI curve even under non uniform saturation conditions.
10. LRQC with high clay content should be measured for CEC as a supplement to electrical resistivity measurement of C_o/C_w and Archie's F.
11. It is recommended to measure RI on chalk samples from oilfields using an oil-brine and not a gas-brine fluid system to minimize surface conduction.

5.4. Advanced core measurements – test conditions

Electrical measurements, like other advanced core measurements, are conducted at overburden or reservoir conditions, and some of the measured data are further used to correct routine core analysis (RCA) data to downhole conditions. There are two different test strategies:

1. Overburden condition studies, conducted at a controlled room temperature and stepwise increasing effective stress (cf. the definition below) at low pore pressure, i.e. the experiment may start at a typical RCA confining stress of 400/800 psi and then proceed at stepwise increasing confining stress steps of 400-1000 psi until the max. effective stress that may be foreseen during the life of the field.
2. Reservoir condition studies conducted at reservoir temperature and pressures equal to the initial reservoir gross overburden stress (lithostatic stress cf. the definition below) and reservoir pore pressure; the pore pressure is then decreased in steps of 400-1000 psi at a constantly held gross overburden stress until reaching the max. effective stress, equal to the max. pressure drawdown, the reservoir may experience during field life.

The majority of special core analysis studies on North Sea chalk have been conducted at hydrostatic stress conditions using the effective stress concept introduced by *Terzaghi (1923)* and corrected according to *Teeuw (1971)* to adapt to hydrostatic test conditions.

For indurated sediments like the North Sea chalk, a better approach is to use the effective stress concept by *Biot (1941)*:

$$\text{Effective stress} = \text{Lithostatic stress} - \alpha \times \text{reservoir pore pressure} \qquad \text{(Eq. 5.4)}$$

α is Biot’s effective stress coefficient

Effective stress is also known as net overburden pressure

Lithostatic stress is also known as gross overburden pressure

For $\alpha = 1$ Biot's equation translates into Terzaghi’s stress concept for unconsolidated sediments and soils. *Engstrøm (1992*) showed that Terzaghi’s principle is not valid for low porosity chalk (porosity < 25-30 %). In case of low porosity, the Biot effective stress coefficient can be estimated from $\alpha = 1-(K_{bulk}/K_{calcite})$, where K_{bulk} is the bulk modulus of the chalk and $K_{calcite}$ is the bulk modulus for the grains (calcite for chalk). A review of the effective stress coefficient can be found in *Alam et al. (2012)*.

Several other SCAL data may be measured in conjunction with electrical measurements, e.g. pore volume compressibility, porosity and permeability reduction as a function of effective stress and the increase in Archie's formation factor (and the cementation exponent 'm') as a function of stress.

5.5. Technology

The measurement of electrical resistivity of core plugs is done in specially designed core holders with one end piece electrically isolated from the main body of the core holder. To avoid electrical polarization effects at the electrodes, an alternating current with a frequency in the interval 1-10^7 Hz are normally used and the impedance measured using a conductivity/resistivity meter or a LCZ-meter capable of reading both impedance and phase angle. In general, impedance readings are preferred at the minimum phase shift. From experience it is known that resistivity measurement of homogeneous North Sea chalk always yields phase angles close to 0° when fully saturated with electrolyte (formation water), i.e. behaves nearly as a perfect Ohm'ian resistor.

The electrode configuration may vary, the most common being the 2-electrode setup with current and voltage electrodes being identical. This configuration measures the resistivity of the whole plug, which allows a fairly precise determination of the water saturation. The 4-electrode method offers the advantage of reducing the bias from a capillary end-effect, but introduces the challenge of determining the interval saturation delimited by the voltage electrodes with sufficient precision.

Electrode configurations and frequency range in resistivity measurements are treated by *Bona et al. (2008)*.

Multi-electrode configurations: With such systems, it is in theory possible to correct for inhomogeneous porosity and saturation distributions, but this requires that the fluid saturation in the sub-intervals be determined by other means, e.g. porosity mapping and saturation monitoring by X-ray or gamma ray techniques (*van der Post et al. 2000*).

Determination of the saturation exponent: The preferred method to obtain RI and 'n' data is porous plate drainage and imbibition experiments, cf. the chapter on Capillary Drainage and Imbibition Experiments. It may be difficult to obtain water saturations below approx. 50% for tight low-permeable chalk due to limitations in porous-plate specs and long drainage times. This will affect the accuracy of the regression line and thereby of the saturation exponent 'n'. Resistivity Index measurement by Continuous Injection (RICI) is not recommended for low quality chalk, cf. the Chapter on Numerical Simulation.

An evaporation method to obtain low water saturations in tight chalk during RI measurement was proposed by *Springer et al. (2003)*. Strong capillary forces in tight chalk will seek to equilibrate saturation gradients in an air-water system; this can be accomplished in a week for saturations below approx. 50%. Due to extensive sample handling, this method is limited to a small number of saturation steps. Porous plates with high break-through pressure specs may hold promise for an extension of the porous plate method to approximately 30 bar (450 psi) in an oil-water system, cf. Chapter 6.4 on Capillary Drainage and Imbibition Experiments

Time constrains may dictate alternative methods to determine the saturation exponent, e.g. non-equilibrium RI measurements with saturation monitoring and mathematical modelling (*Jing et al. 1993A and B*) to derive the intrinsic 'n', or the "FRIM" method (*Fleury 1998*) that utilizes a special multi-electrode set-up in a radial configuration to derive an un-biased continuous RI curve even under non uniform saturation conditions. These methods seem potentially promising for LRQC.

5.6. Practical Application

Porous plate experiments: Compared to RI and 'n' values measured during primary drainage from S_w=1 (oil displacing water), *Elashahab et al. (1995)* reported cycle dependant hysteresis for water-wet sandstones where a secondary drainage cycle is measured after a dynamic water flood, i.e. starting from $S_w = 1-S_{or}$. Results showed that saturation exponents obtained from the second drainage cycle were significantly lower than that determined from the first cycle starting from $S_w = 1$, an observation that may relate to chalk as well. Internal laboratory experience confirms that lower 'n' exponents were also obtained for an imbibition experiment to residual gas saturation. The previous history was a porous plate gas-water drainage experiment to S_{wi} followed by water imbibition to S_{gr} at P_c=0.

Lab experience from measurement of North Sea chalk generally show that the regression line in F vs. Ø diagrams always passes through the (1,1) intercept (fully saturated samples), and in C_o/C_w diagrams have zero or close to zero intercept. This indicates insignificant excess conductivity, i.e. a negligible shale effect at full brine saturation. Most chalk samples are therefore "clean" in the sense of Archie at full brine saturation, but this is not necessarily valid for LRQC with high clay content, or during desaturation (RI measurements) particularly for mid and low porosity chalk samples. Although the number of measurements are limited it is observed that the saturation exponent 'n' decreases with decreasing S_w, which could indicate surface conduction as predicted by the model for micro porous rocks by *Worthington et al. (1989)*.

5.7. Reporting Requirements

X-ray CT screening images and gray values for selected plugs cf. the chapter on Screening of Samples. Basic data includes impedance, resistivity and conductivity measurements corrected for wire resistance, temperature and phase angle. Besides, RI measurements must include pressure and production data corrected for dead volumes and starting offsets. A temperature log must be given. Other requirements as per *SCA Guidelines (Sprunt, 1990)*.

By request, the service laboratory must be able to document that their electrical resistivity equipment measures correctly, e.g. by measurement of precision resistors placed in the resistivity cells cf. SCA Guidelines Part 3 (*The Log Analyst, 1990*), and that conductivity meters/cells are calibrated against a certified conductivity normal, e.g. KCl of a suitable concentration (NaCl solutions would be more appropriate for core analysis applications but are not normally offered by instrument or chemical companies).

5.8. Pitfalls

An extreme scatter in measured 'n' values is observed for all chalk formations that cannot easily be explained from geology and texture. It is due to poor or missing sample screening and not recommended practices, e.g. room condition measurements, centrifuge and dynamic flooding down methods for 2- and 4-electrode configurations, poor electrode contact at low water saturations during porous plate RI measurements and failure to optimize the measurement frequency relative to the resistivity equipment configuration.

Excess and surface conductivity: North Sea reservoir chalk has average pore sizes around 1μm and should be considered a micro porous rock. As porosity decreases and the insoluble residue (ISR) increases for some chalk formations (Ekofisk and Lower Cretaceous) it is to be expected that LRQC will have a significant fraction of pores with diameters at and below 0.1 μm. LRQC may therefore be subject to excess conductivity due to the content of clay minerals and to surface conduction during desaturation experiments because of micro porosity (*Worthington et al. 1989 and 1990*).

Chalk is a rate type material and therefore sensitive to stress and subject to creep, i.e. momentary application of overburden stress may damage the sample, and pore volume adjustment relative to changes in stress depends on permeability, and settling times must be respected, cf. *Ruddy et al. (1989), Anderson (1992) and Omdahl (2010)*.

5.9. References

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6. Capillary Drainage and Imbibition Experiments

6.1. Abstract

Three methods are available to measure capillary pressures. Mercury injection, only suitable for primary drainage on disposable samples, is the preferred method if a large number of samples need to be measured and characterized on pore size distribution and capillary (entry) pressure in a relatively short time. The porous plate method is applicable for smaller sample sets, both for drainage and imbibition, if more time is available (up to 6 months or longer for a single experiment). Porous plate allows both spontaneous imbibition/drainage and forced imbibition/drainage data to be collected. The multi-speed centrifuge method is applicable only for forced drainage and forced imbibition data, within a shorter time span than porous plate. However, special care is to be taken to avoid samples to disintegrate at high rotational speeds. Another limitation is that the centrifuge cannot probe capillary pressures as high as the porous plate can do.

All three methods rely on achieving a stationary state, i.e. zero flow, to exclude the effect of viscous forces through the relative permeabilities. In practice, this cannot be guaranteed to occur at all saturation levels. Particularly near low water saturations and near low (residual) oil saturation, this premise breaks down. To unravel the interference between capillary pressure and relative permeabilities, interpretation-by-simulation of the experiments needs to be carried out. For that, capillary pressure measurements need to be accompanied by corresponding relative permeability measurements.

6.2. Introduction

Water saturation is commonly determined by use of empirical models relying on well logs. Results depend on interpreter assumptions, model uncertainty and log quality. Primary capillary drainage curves allow independent modelling of initial water saturation as a function of rock type. The logs and capillary pressure data sets are frequently used in pair to construct field models of initial water saturation with the purpose of calculating OOIP and optimizing prediction accuracy outside well-controlled area.

Chalk reservoirs are well known to be characterized by low matrix permeability *(Scholle 1977, Megson and Hardman 2001, Fabricius 2007)*. As a result, even high quality chalk reservoirs tend to experience significant hydrocarbon volumes located in transition zones. This becomes more pronounced for LRQC, making it increasingly important to have high quality capillary drainage data available.

Imbibition data are equally important for evaluation and modelling of water imbibed sections of the reservoir. It is also key input for interpretation-by-simulation of experiments such as Steady-State and UnSteady-State to measure relative permeability, to un-entangle the interference between capillary pressure and relative permeabilities in laboratory experiments.

Definitions

Capillary pressure P_c in an oil-water system is defined as

$$P_c = P_o - P_w \qquad \text{(Eq. 6.1)}$$

with P_o the pressure in the oil phase and P_w the pressure in the water phase. Defined as a pressure difference, capillary pressure can take on positive as well as negative values.

6.3. Recommendations

Methodology

1. Ensure that plugs have been selected to be sufficiently homogeneous (see Chapter on Screening of Samples) and have been cleaned and dried (See Chapter on RCA) to prepare these in a water-wet state (see Chapter on Representative Initial Water Saturation and Wettability).
2. Sensitivity to confining stress needs to be assessed.
3. When both drainage and imbibition data are required, the porous plate is the likely candidate because it will allow larger capillary pressures to be measured than the centrifuge. However, the experiments take much longer time (6 months and more) than with the centrifuge, which can be prohibitive. If just drainage experiments on a large sample set are required to establish a saturation height distribution, mercury injection is the likely candidate.
4. Use experimental design, preferably by numerical simulation (see Chapter on Numerical Simulation) to select a technique, choosing from mercury injection, porous plate and centrifuge, with the notion that mercury injection is only suitable for drainage and should not be used for imbibition measurements.
5. Experimental design should also be used to decide on pressure steps (mercury injection and porous plate) or on speed settings (centrifuge technique) and on the criterion to be used on deciding when production levels are considered stationary.
6. To evaluate production levels to be steady, it is recommended to plot produced water (and/or intrusion of mercury and resistivity in porous plate experiments) versus square root of time and/or versus log(time).
7. It is recommended to perform mercury injection on a plug trim-end (but away from the mud invaded core surface) or a twin plug (1”x1.5”) to better define entry pressure and general shape of drainage curve for optimal experimental design.
8. Generally, some 7-8 pressure steps during drainage or forced imbibition processes in the porous plate or centrifuge will be programmed. It is recommended to probe capillary pressure in small increments in parts of the curves where changes in saturation occur rapidly. This must however be balanced against the total time required for the experiment.
9. For LRQC, the porous plate method is generally preferred over centrifuge and mercury injection in terms of acquiring representative capillary pressure curves. Improved porous plates rated for significantly higher capillary pressure are commercially available. Such plates should generally be considered in order to improve the saturation range analysed in tight chalk samples.
10. There are observations where mercury capillary pressure data under-predicts water saturation with respect to modelling transition zones (see *Greder et al., 1997 and Nørgaard et al., 1999*). In such cases, it is recommended to critically review both laboratory and field data to resolve such inconsistency.

11. All data need to be extracted using interpretation-by-simulation of the experiments to unravel the interference between capillary pressure and relative permeabilities (see Chapter on Numerical Simulation)

General

1. In order to minimize effect of experimental dead volumes and maximize the pore network, it is recommended to use 1.5” diameter plugs. In most cases, plug length will be limited by the experimental equipment.
2. Clean mineral oil (decane) and nitrogen gas can be used as non-wetting phase for drainage-only experiments. Although crude oil experiments are available, we do recommend the use of simpler fluid when possible. It is not recommended to use crude oil due to the experimental complexity required for achieving valid data for drainage-only experiments.
3. To verify validity of data obtained with crude oil, it should be considered to run two drainage experiments in parallel and comparing results. The primary plug set is to be run with crude oil as non-wetting phase to include both drainage and imbibition measurements. The secondary plug set uses gas or mineral oil.
4. After completion of the capillary drainage experiment, water saturation should be confirmed by independent Dean Stark measurements (or after completion of imbibition if included). Initial water saturation representative of the log calculated water saturation is recommended as starting point for the imbibition process.
5. If imbibition is part of the study, it is recommended not to use mineral oil or gas. Because of very low permeability in LRQC material, it can be challenging to properly displace these non-wetting phases with crude oil for ageing and restoration of wettability. Hence, for studies that include imbibition, it is recommended to perform sophisticated drainage experiments using properly filtered crude oil, at elevated temperature if viscosity so dictates.
6. Imbibition experiments should only be conducted after ageing at the appropriate initial water saturation (see Chapter on Representative Initial Water Saturation and Wettability). After completion of ageing, reservoir oil could be displaced by decalin and subsequently decane. This allows wettability to be maintained at lower experimental temperatures.
7. We generally recommend continuing forced imbibition until a near asymptotic curve is obtained.
8. Weighing of the plugs before, during and after the experiments will facilitate quality assessment.

Mercury Injection Method

1. Laboratories conducting mercury injection measurements should be aware of and comply with the ASTM testing protocol standard D4404-84 (1998) and the experimental protocol described by *Shafer and Neasham (2000)*.
2. The conventional apparatus for mercury injection is the Autopore equipment. To explore sensitivity, tests should however be accompanied by selected experiments performed on companion plugs in core holders at net confining pressure. For a further discussion on the impact of stress, see *Andersen (1992)*.

3. Closure corrections are required to cancel the effect of surface unconformities and potential fractures. The laboratory should provide documentation on how this correction is applied, and provide uncorrected data on request.

Porous Plate Method

1. It is critical to perform the experiment with plugs mounted in individual core holders at net confining pressure, i.e. avoid unconfined multi sample pressure pots.
2. In addition, if forced imbibition is part of the study, we recommend mounting both water wet and oil-wet porous plates prior to initiating drainage experiments. Alternatively, the oil-wet plate can be installed after spontaneous imbibition at zero capillary pressure, but this increases measurement uncertainty.
3. The experimental set up should incorporate resistivity measurement as primary input for evaluation of equilibrium at each capillary pressure and/or to facilitate resistivity measurements in combination with capillary pressure (see Chapter on Electrical Measurements).

Centrifuge Method

1. Be aware that high rotational speeds easily cause plug failure on high porosity chalk. Unless LRQC plug competence is known to be of good quality, it is recommended to perform a pre-test on selected plugs to verify successful experimental procedure.
2. Based on assumed plug competence, consider capsuling samples in Teflon with Teflon tape and Teflon shrink tubing. This allows safer plug handling during the experiment.
3. For chalk samples, it is generally recommended to use a centrifuge that allows application of confining pressure. This is not only to better maintain plug integrity during the analysis, but also to address stress sensitivity of the data.
4. Contrary to what is commonly used by several service providers, and in line with the general recommendations above, we stress that it is recommended to use 1.5" diameter samples in the centrifuge for optimal saturation accuracy
5. Use only automated centrifuge equipment with production data collected through a stroboscopic read-out.
6. To allow re-distribution of fluids after primary drainage or imbibition, it is recommended that the plug should rest for a longer period (typically 1-2 days, but in an oil-water system this may take much longer time due to the unsuitable oil viscosity). Inspection of saturation distribution can be performed on separate core plugs using NMR imaging techniques (see Chapter 2 on Nuclear Magnetic Resonance). The centrifuge analysis does not allow proper description of spontaneous imbibition curves. It is recommended to perform spontaneous imbibition as described in the Chapter on Wettability measurements.
7. The centrifuge analysis does not allow proper description of spontaneous imbibition curves. It is recommended to perform spontaneous imbibition as described in the Chapter on Wettability measurements. Given the low permeability and assumed mixed to water-wet preference, it is critical to allow long stabilisation times.

6.4. Technology

A fluid will not enter a rock pore body via the pore aperture without pressure if it does not wet the pore surface. Thus the external pressure required to force a non-wetting liquid into a pore is inversely related to the pore aperture diameter or radius.

Three experimental techniques are available in the industry to measure capillary pressure curves: mercury injection, porous plate and centrifuge. All techniques have in common that capillary pressure experiments are only valid if equilibrium saturations can be achieved at each pressure step. The laboratory should not use time-fixed tables as basis, but maintain displacement until volumetric and electrical stability has occurred. To determine equilibrium, it is recommended to plot produced water, mercury intrusion or resistivity versus square root of time and/or versus log (time).

Mercury Injection

Mercury injection is performed by placing a clean and dried rock sample in a small glass cup called a penetrometer. The cup is subsequently filled with none-wetting mercury in incremental pressure steps, ranging from vacuum to experimental maximum. The analysis generally provides mercury-air capillary pressure curves, pore throat size distribution and estimates of porosity and permeability.

Although frequently used in carbonate reservoirs, the validity of capillary pressure curves from mercury injection has been questioned by several authors. Comparing capillary drainage curves from porous plate method to that obtained from mercury injection, they are frequently found not to match *(Greder et al. 1997)*. Data indicate that mercury capillary curves often are optimistic. The discrepancy is not fully understood, but could be linked to some of the following items discussed by *Shafer and Neasham (2000)*:

- Mercury-air capillary pressure curves must be transposed to equivalent reservoir fluids using conversion factors. Regardless of the uncertainty in their values, the resulting drainage curve will only represent a strongly water-wet system.
- Sample size has the potential to affect the result. The maximum size allowed in the penetrometer is 1”x1”, but often smaller samples (plug trims) are measured. This is considerable smaller than the typical SCAL plugs used for porous plate measurements. Additionally, percolation theory has established that sample size and shape can also affect capillary pressure data. Higher ratio of surface area to sample bulk volume tends to lower initial pore aperture entry pressure, creating more optimistic results
- *Hirsch et al (1994, 1995a, 1995b)* have shown by percolation theory that different capillary pressure curves will result from 3-D drainage versus 1-D drainage testing of a sample. Results are more optimistic in the case of performing 3-D drainage, which is the case for mercury injection analysis.
- Unlike porous plate, mercury injection is most commonly performed without confining pressure. Some laboratories do however provide manual 1-D mercury injection on larger samples. The confining pressure is increased in steps with injection pressure, keeping the net confining pressure constant. Costs are generally higher and experimental time longer compared to automated measurements. *Andersen (1992), Mitchell et al (2003)* and previous JCR Phase 3 and 4 experiments found that overburden pressure can impact results significantly.

- Sample unconformities, such as minor fractures, would be evident by mercury intrusion at low capillary pressures. The first pressure that is connected to valid pore volume is detected as the inflection point on a semi-log plot of “SHg(%BV)” vs.” PcHg”. Unless properly corrected for, this will also affect porosity and permeability estimates from the mercury injection experiment.
- *Greder et al (1997)* and *Shafer and Neasham (2000)* indicate that the amount of discrepancy between mercury injection and porous plate capillary drainage is proportional to the clay content and structure. In addition, the heterogeneity and tortuosity are likely candidates to cause saturation discrepancy. The understanding is that all these factors lead to the trapping of water that is not captured in the mercury injection experiments. Tests performed indicated that the best curve match was obtained in clean rocks.

Due to effects of high-pressure mercury on rock matrix and the absence of a representative fluid pair, it is recommended to disregard mercury withdrawal data (that correspond to spontaneous imbibition curves) from any mercury injection test. Moreover, early snap-off usually restricts the saturation range in imbibition.

Porous Plate Method

Fully water saturated core plugs are de-saturated by increasing the pressure of the non-wetting phase in increments. A semi-permeable water-wet porous plate allows only the wetting phase fluid to pass through it in drainage experiments. For imbibition measurements, the water-wet plate is exchanged for an oil-wet plate. When performing primary drainage and imbibition experiments at ambient conditions, conversion factors are required to convert the results to representative fluid pair at reservoir conditions. Although considered the conventional approach, surface tension and contact angles are often not well described for each reservoir system, and tabulated values are frequently used. This introduces a significant uncertainty to the core data set when compared to water saturation calculated from open-hole logs.

In an attempt to mitigate this uncertainty, some laboratories perform experiments at pseudo reservoir conditions, i.e. elevated temperature and net confining pressure. Applying an assumed representative fluid pair in the experiment, such as crude oil and synthetic formation water in an oil-bearing reservoir, capillary pressure curves will theoretically represent reservoir conditions. There remain however sizable uncertainties.

Focusing on LRQC, a new set of experimental challenges emerges. Conventional porous plates have capillary pressure limitations. When this pressure is exceeded, typically between 100-200 psi depending on fluid pair, the injected fluid will break through the plate and invalidate the experimental result. Given the low permeability and well sorted pore throats in chalks, first observation of hydrocarbons can be several 100 ft. above the corresponding FWL. This translates to high entry pressure on the capillary drainage curve, often at or above the experimental boundary, which limits the value of porous plate method for LRQC. Some laboratories do however provide alternative porous plates that allow significantly higher capillary pressures, up to 1000-psi gas/water fluid pair. Although commercially available, fewer experiments have been conducted using this technology. It would however be of significant importance for investigation of capillary pressure in LQRC.

Stability at each pressure step (constant within the noise level) should be evaluated using resistivity as primary qualifier. Assuming experiments are conducted in individual core holders, which is preferred relative to unconfined experiments in multi-sample pressure pots, time required in the laboratory to reach equilibrium at each pressure step is directly dependent on core permeability and sample length. Displacement in tight chalk is severely delayed by narrow pore throats and relative permeability restrictions. Chalk might have considerable porosity despite its low permeability, i.e. significant volumes of water need to be displaced at low rates. Consequently, porous plate experimental time requirements are often not competitive compared to other techniques.

The imbibition data measured by the porous plate technique provides good quality spontaneous and forced imbibition capillary curves. Because of hysteresis, the electrical properties under imbibition are in most cases not the same as experienced during drainage (see Chapter on Electrical Measurements). Porous plate imbibition can as such provide saturation exponents for calculation of water saturation from open-hole logs in imbibed sections of the reservoir.

Centrifuge Analysis

Obtaining capillary pressure curves from centrifuge technique requires two steps; measurements of fluid production data and transformation of that data into capillary pressure curves. Capillary pressure curves are in other words not measured directly, but modelled as function of other parameters. By increasing the rotational speed of the centrifuge, gravitational forces are increased in steps.

Experience from centrifuge experiments performed on chalk plugs shows that especially high porosity material may fail when exposed to high rotational speeds. It might be that LRQC material is more competent, but caution should be taken if centrifuge tests are considered. Some centrifuges allow application of modest confining pressure, and are generally preferred for chalk experiments.

The existence of several transform algorithms, together with variation in centrifuge equipment and experimental procedures, encouraged SCA (Society of Core Analysts) to conduct an extensive inter-laboratory survey. The aim of the study was to evaluate how the different methods of implementing the centrifuge technique impact results on drainage capillary pressure measurements by centrifuge. The results of that survey were analysed and discussed during a SCA workshop in Houston in 1993 *(Ruth and Chen 1995)*. Together with findings from an additional SCA survey *(Forbes, 1997),* the following was concluded:

- The main source of inaccuracy in the drainage capillary pressure curve determination by centrifuge is related to the interpretation process and not to experimental procedures (assuming accepted procedures are in use) nor to shape of the capillary pressure curve.
- The inaccuracy depends on the centrifuge geometry (and resulting contributions of centrifugal and radial effects) and on the method used for solving the centrifuge equation. The choice of the method, according to the centrifuge/core geometry, appears to be of prime importance, much more than improving experimental conditions. This is especially critical for short radius geometry as applicable to drainage experiments, and includes the most common geometry of 1" x 1" sample run at 8.6 cm from the centrifuge axis.
- Additional errors due to the inversion process may be very large (+/-10 saturation units). They cannot be reduced significantly by increasing the number of rotation steps or by reducing the experimental error below two saturation units or by averaging the results from different interpretation processes. Because most of these processes introduce a negative bias, the common practice of using different interpretations and averaging the results is totally irrelevant. Errors being dominantly negative, no compensation is achieved by averaging.

- It was found that from the available methods for analytical data inversion, the so-called Forbes-2nd method worked best. Note these inversion algorithms (including e.g. the well-known Hassler-Brunner approach) convert the measured average saturations into inflow-face saturations necessary to define the capillary pressure from centrifuge data[1].

The above-mentioned approach to improve on data interpretation, however, still assumes implicitly that relative permeabilities do not play a role. In fact, all analytical methods for data interpretation of capillary pressure measurements ignore relative permeabilities (see Chapter on Numerical Simulation). The method of Interpretation-by-simulation uses the analytical results obtained with Hassler-Brunner and/or Forbes as starting point for history matching capillary pressure experiments. In practice, this state-of-the-art method has an impact on the data inversion that tends to dominate over the correction from the Forbes method.

6.5. Practical Application

The vast majority of modern capillary pressure analyses are performed using automated high-pressure mercury injection equipment capable of reaching 60.000-psi capillary pressure, enabling description of the complete transition zone and reservoir thickness. Mercury injection provides a rapid quantification of a samples interconnected pore system and the size distribution of pore apertures that strongly influence non-wetting phase saturations and fluid flow.

As a result of the many factors affecting mercury injection capillary pressures curves, it is recommended to use data with careful consideration. Primary product from the analysis should be pore size distribution and also detection of entry pressure. However, in the worst cases of LRQC, mercury injection might be the only option to acquire capillary drainage data. In these situations, we encourage frequent acquisition in order to perform a capillary screening of the entire cored sections, preferably including other petrographic analyses as XRD, SEM and specific surface area by gas absorption (N_{2BET}). Together with open hole logs, geological description and routine core data, this should enable the interpreter to establish typical rock type trends. The corresponding capillary pressure curves might not perfectly represent reservoir fluid pairs at reservoir conditions, but they could indicate relative difference between the rock types. Any use of capillary data for modelling of initial water saturation should be done with great caution, generally using log calculated water saturation as a calibration point.

The porous plate method is generally the preferred industry technique to obtain capillary pressures if both drainage and imbibition capillary pressure curves need to be measured. The porous plate method is the only technique that allows measurement of the spontaneous parts of the capillary pressure curve under confining stress conditions.

Provided application of a suitable experimental setup, the porous plate method allows also measurements of resistivity across core plugs at each pressure step. This is a major advantage, since is allows calculation of saturation exponent in conjunction with porous plate capillary drainage and imbibition experiments (see Chapter on Electrical Measurements). Moreover, the porous plate experiment can be conducted at overburden pressure to deal with stress sensitivity.

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[1] Forbes' approach to correct for "radial effects" is designed as a separate data pre-processing step before analytical inversion algorithms are applied, including the Forbes-2nd method.

The porous plate method has one important drawback: due to the low permeability of the plates (usually less than 5 μD), experiments are very time consuming and may take 6 months or longer to complete.

In those cases where the timing of the data collection does not allow porous plate, the centrifuge can be used. The range in capillary pressure (and therefore also in saturation) is more limited than with the porous plate. Moreover, chalk samples may need special precautions in mounting these in the centrifuge to reduce the risk of sample disintegration due to high centrifugal forces.

All methods require interpretation-by-simulation to unravel the interference with relative permeability in the capillary pressure measurements.

6.6. Reporting Requirements

The following information should be reported from mercury injection experiments:

- Raw data: Sample weight, Hg bulk volume, Hg porosity, type of blank correction conducted, IFT used, what “surface correction” has been applied, percentage stem volume used at maximum intrusion, Hg injection pressure and saturation from start of experiment (i.e. before “surface correction”), pore throat radius, normalized pore size and permeability distribution, equivalent injection pressure for an air/brine (lab), oil/brine (lab) and oil/brine (res) system including conversion factors used, height above free water level, Leverett-J function data. Optionally: calculation of theoretical permeability.

- Diagrams: - air/mercury capillary pressure vs. mercury saturation on log and linear scale

  - mercury saturation vs. pore throat radius

  - normalized pore size and permeability distribution vs. pore throat radius

  - reservoir oil/brine capillary pressure and height above free water level vs. water saturation

  - Entry pressure (drainage cycle injection pressure vs. equivalent saturation)

  - Leverett J function vs. wetting phase saturation

- Conversion factor data: brine and oil density gradient figures, effective surface tension data at laboratory (lab) and reservoir (res) conditions.

  (effective surface tension is = $\gamma \times \cos\theta$)

For porous plate experiments, details about the plate need to be provided: breakthrough pressure, permeability, porosity, thickness and diameter.

Both for porous plate and centrifuge experiments, the criterion used to assess stable saturation levels must be reported. Also, the material balance verification (by weight and/or Dean&Stark) needs to be reported. Report any “grain loss”. In addition, the time series of the production data and fluid data (density, viscosity) has to be reported to facilitate quality assessment of the reported capillary pressure data, as well as to facilitate to conduct interpretation-by-simulation.

Centrifuge capillary pressure data need to be provided based on several analytical interpretations that should include at least Hassler-Brunner and Forbes’ second method.

6.7. Pitfalls

As evident from permeability measurements discussed separately, any normalization or rock typing that involves permeability (directly or indirectly) will be very sensitive in LRQC. It is important to be aware of the different types of permeability, and also to maintain consistency when comparing data sets. Any construction of Leverett J-functions should have a permeability not outside the established trend lines.

Pressure steps may require weeks to months to stabilize. Errors in the resulting curves can be very large and are difficult to assess without experimental design.

Note that with mercury-air capillary pressure measurements, an irreducible water saturation cannot be actually measured. Rather, S_{wirr} is determined applying a capillary pressure cut-off value.

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7. Representative Initial Water Saturation (Swi) and Wettability

7.1. Abstract

Relative permeability and capillary pressure depend strongly on wettability. Representative wettability depends on representative initial water saturation S_{wi}. In turn, S_{wi} depends on the local saturation height distribution in the reservoir. Therefore, it is imperative that the correct S_{wi} is used in advanced core analysis experiments. The service provider is not in a position to provide this value, because one needs to have access to the geological maps together with the relevant saturation height function. The client, in a multi-disciplinary effort shall provide S_{wi} to be used for individual core plugs. The service provider then selects a laboratory method to bring about the requested saturation level. Recommended methods for LRQC are porous plate, evaporation and, in some cases, the centrifuge.

In this chapter, a case is made to avoid fresh state core experiments and instead use restored-state samples. This implies that plugs need to be cleaned to a water-wet state, brought to S_{wi}, and that the plugs then need to be subjected to an ageing process.

7.2. Introduction

It is experimentally challenging to obtain low initial water saturations (S_{wi}) in low permeable rock, while wettability and therefore relative permeabilities depend strongly on correct initial water saturation, representative of the reservoir flow unit targeted by the SCAL study.

Definitions

A source of confusion exists around the definition of the connate water saturation S_{wc}: reservoir engineers will use S_{wc} interchangeably with the irreducible water saturation S_{wirr}. By definition, S_{wirr} represents the lowest water saturation that can be reached at infinite capillary pressure. However, geologists may take S_{wc} as the water saturation at which the virgin flow unit was found, so in fact geologists may take S_{wc} as S_{wi} rather than as S_{wirr}.

In addition, modern reservoir simulators (ECLIPSE, GEM, STARS, etc.) allow a distinction to be made between S_{wc} (in the reservoir engineering sense, so: S_{wirr}) and the critical water saturation S_{wcrit}. S_{wcrit} is defined as the water saturation above which water becomes mobile. In our experience, S_{wcrit} is identical to S_{wirr} for all practical purposes.

Wettability

Wettability will significantly affect fractional flow in porous media. E.g. for chalk reservoirs with a high fracture intensity, water flooding production is strongly dependent on spontaneous imbibition potential (*Graue et al, 2001*). Understanding and being able to incorporate wettability in reservoir simulations are as such critical.

Wettability is not a direct input parameter in the simulation, but masked in other wettability dependent parameters like relative permeability and imbibition capillary pressure curves. Regardless of its importance, the industry still does not have a rigid tool to determine in-situ wettability preference. Assumptions are often made from field observations like rock mineralogy, open-hole log response, fluid compositions, pressure, temperature, production profiles and core data.

Reservoir wettability is determined by the interactions between constituting components in the crude oil the brine and the rock (so-called COBR system, see *Morrow, 1990*). Moreover, the rock surface is covered by minerals that may partially or fully shield the rock matrix proper from the fluids that add complexity to wettability characterisation. Any deviation of any of the COBR components in the laboratory compared to the situation in the field prevents accurate reproducibility of representative wettability.

The pore surface in the core is sensitive to environmental conditions and hence easily altered from its native state during coring operation, core handling or core experiments. Brine composition is notoriously difficult to assess and even live crude samples may not be fully representative for reservoir oil if one cannot rule out chemical interactions with the sample container walls and or lines in the test equipment in the lab, oxidation during experimentation, etc. Consequently, fresh state wettability measurements require extreme caution on all COBR constituents in order to provide representative results. Alternatively, the core plugs can be cleaned and wettability attempted to be restored by introducing reservoir oil in a controlled laboratory environment, and allowed a long soaking period ("ageing") to establish equilibrium between crude oil, brine and rock.

Parts of the industry prefer using fresh core material, arguing that it is not possible to restore wettability representative to that developed over geological time. On the other hand, the division that prefers restored state wettability would argue that wettability on fresh core material is most likely not representing in-situ conditions due to inevitable artificial alterations.

However, in the continuing absence of a tool that can measure wettability down hole and in the laboratory so that one would assess reliably whether a cleaning-and-restoration procedure or rather fresh-state approach for the COBR system is a good representation of down-hole wettability, the industry cannot reliably determine which route should take preference for SCAL experiments.

From a technical point of view, SCAL can be performed on both sets of core material. Unless extreme caution is taken during coring and core handling, fluid sampling and fluid handling (both for oil and brine)[2], it is recommended to perform SCAL experiments on restored state core material. In order to compare results across wells or fields, it is equally recommended to be consistent in choice of method. Finally, a definite advantage of restored-state experiments over fresh state is that restored-state experiments can be repeated on the same plug to verify reproducibility of unexpected results. Fresh-state core experiments can only be conducted once in the life of a core plug.

Representative S_{wi}

Very close to the oil-water contact, rock in the abundant presence of water and low oil saturation, is likely to remain much more water wet than rock higher up from the oil-water contact. Therefore, for wettability in the experiments to be representative for the reservoir, we need to restore wettability at a representative S_{wi}.

It must be stressed that a representative initial water saturation is not necessarily equal to the irreducible water saturation S_{wirr}. Dependent on the height above the free water level, this theoretical minimal value may only be approximated in the field (see Figure 7.1). In so-called transition zone reservoirs, the

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[2] Note that this would include sampling of core as well as all fluids under oxygen-free conditions, both in the well and in the laboratory, to avoid chemical alteration of any of the COBR constituents, rendering fresh-state experiments impractical

saturation-height distribution may well demonstrate water saturations significantly (e.g. 30 or more saturation units) higher than the theoretically lowest limit at infinite capillary pressure.

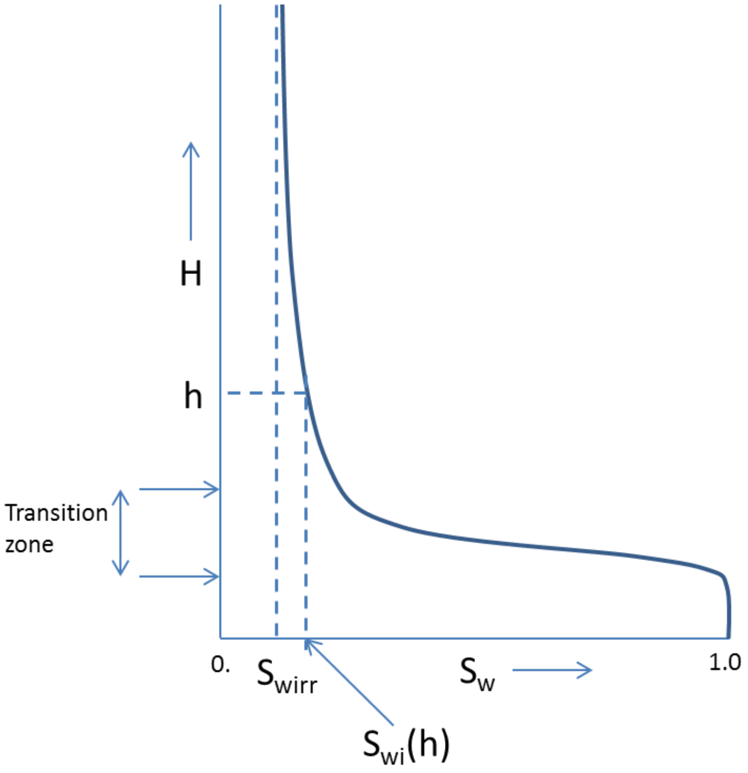

Figure 7.1: Typical saturation-height distribution, H(S_w) above the free water level. S_{wi} depends on the height h of an individual flow unit. S_{wi} varies strongly over the transition zone. S_{wirr} corresponds to the saturation reached asymptotically at large height.

The initial saturation-height distribution is brought about during charging of the reservoir and represents the equilibrium between gravity forces (buoyancy of the oil in a brine environment) and capillary forces restraining water from being forced down by the upward migrating oil. Once the equilibrium is established over geological times, water at saturations higher than S_{wirr} is immobile vertically, under the influence of gravity. However, such saturations may well be movable horizontally during depletion. This concept impacts on which methods to use establishing at a definitely mobile S_{wi} and measuring relative permeabilities from that point on.

Restored State Core Preparation

The first step into restored state preparation is cleaning the plugs to a water-wet state (see Chapter on RCA), possibly with a verification of the wettability after cleaning (see Chapter on Wettability Experiments). Such verification is particularly important in cases where oil-based mud has been used.

Alteration of wettability in a core plug is very complex. Several of the key aspects are discussed by *Fernø (2010)* and listed below.

The oil composition greatly impacts the wettability change of naturally water-wet rock surfaces during aging because of differences in the aging ability for the components present in crude oil. The exact nature of the adsorbed polar components in the crude oil is still unknown but is believed to be governed by more than one mechanism. The main mechanisms of wettability alteration in porous media by crude oil include the polar interaction between oil components and solid, surface precipitation, acid/base interactions controlling the surface charge at interfaces, and ion binding between charged sites and higher valency ions.

As discussed by *Morrow (1990)*, wettability is not only sensitive to crude oil composition, but also to formation brine composition. In practice, it is very difficult to obtain reliable brine compositions for a reservoir, particularly the divalent ion composition. However, these ions may impact significantly on clay swelling (*Scheuerman, 1990*) and jeopardise any SCAL experimental result.

In addition, drilling mud invasion may alter wettability in a possibly irreversible way. Oil-based mud contains surfactants to keep the oil in suspension and these surfactants may well change the rock surface chemistry and may be very difficult to remove by cleaning. Cores taken for SCAL measurements, with their strong sensitivity to wettability, should be taken with water-based mud. Tracers in the drilling mud help to delineate the invaded zone so that the chances are improved to obtain unaffected plugs, cf. section on Fluid Saturation in the RCA chapter.

The wettability actually achieved through restoration is in addition sensitive to several experimental factors (*Graue et al, 2002*). Results indicate that wettability alteration by submerging core plugs in crude oil and ageing for long times may cause heterogeneous wettability conditions. The process is more efficient if crude oil is flushed in reversing directions through the core plug during the ageing process.

As discussed above, once that the system is brought to S_{wi}, the samples need to be aged. Ageing is conducted preferably at reservoir temperature and at a pressure sufficient to keep dissolved gas in solution. The ageing period required to restore wettability has typically been set to 4 weeks or 1000 hrs (41 days), (*Anderson, 1986*).

Gas-brine Systems

Gas-brine systems are assumed to be water wet and therefore do not require ageing, after establishing S_{wi}.

Impact of Stress

Stress impacts porosity and pore throat radii, and therefore capillary pressure, and therefore S_{wirr}. For a given S_{wi}, the mobility of water is then impacted as well, because the larger the saturation difference is between S_{wi} and S_{wirr}, the larger k_{rw} is likely to be. Particularly for chalk samples, with the possible presence of micro fractures, stress needs to be representative for the respective flow unit.

Another aspect of stress is that with grains moving, the geometry on the pore scale is affected. There is literature that suggests that wettability is determined by pore geometry (*Skauge et al, 2004*). This presents another reason to ensure representative stress for representative SCAL.

7.3. Recommendations

Methodology

1. Ensure that plugs have been selected to be sufficiently homogeneous (See Chapter on Screening of Samples) and have been cleaned and dried (See Chapter on RCA) to prepare these in a water-wet state.
2. If core was taken with oil-based mud, water-wet state should be verified (see Chapter on Wettability Experiments)
3. Obtain value for required S_{wi} from saturation-height distribution of the relevant flow unit, to ensure that the initial water saturation is representative of the flow unit. The saturation-height distribution is usually built with data from resistivity logs in combination with data from primary drainage capillary pressure curves (see Chapter on Capillary Pressure).
4. Note that S_{wi} is first step in a SCAL measurement program and usually ageing is performed at S_{wi}. Consequently, it is recommended to avoid a non-representative S_{wi} that subsequently will result in non-representative wettability, invalidating any measurement data.
5. Use experimental design, preferably by numerical simulation (See Chapter on Numerical Simulation) to select a technique to obtain the chosen S_{wi} within the time schedule set of the measurement program. Likely candidates for S_{wi} laboratory techniques are: a) Porous Plate; b) Evaporation; and c) Centrifuge for K_{abs} higher than a few mD and therefore less relevant for LRQC.

General

1. To achieve S_{wi}, the samples need to be prepared first at S_w=1.0, using a representative brine (See Chapter on Salinity), paying attention to the di-valent ion composition.
2. The displacing phase to reach S_{wi} usually is the relevant crude oil. However, dependent on the experimental design, it may be necessary to use a synthetic oil instead, of (more) suitable viscosity.
3. In the event that S_{wi} has been achieved with synthetic oil, this oil needs to be replaced by the relevant crude before ageing.
4. Ageing to be conducted for 1000 hrs. (41 days) or at least for 4 weeks[3].

Gas-brine

1. Gas-brine systems are assumed to be water wet and do not require ageing.

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[3] In line with standard industry procedures, see e.g. *Villard et al. (1993).*

7.4. Technology

Four laboratory methods are in use to establish S_{wi}. The main characteristics of each are discussed below.

- Porous plate: cheap; long duration (several months) particularly for low perm rock such as chalk; individual cells rather than large, “Soil Moisture” cells, stress can thus be applied; high pressure plates are now available (up approx. 70 bar entry pressure for gas-water drainage and 24 bar oil-water drainage). Higher-pressure plates require even longer duration, because the permeability of the plates reduces with increasing entry pressure. With long duration laboratory procedures, careful planning of the SCAL program is paramount in order to deliver results in time to secure impact on field development plans.
- Centrifuge: expensive; particularly for low permeable rock (because of the long occupancy of expensive equipment); limited stress can be applied (variable from 200-350 bar, laboratory & equipment specific); limited capillary pressure range (about 10 bar for oil-brine and 30 bar for gas-liquid, these limits are also variable lab-to-lab and dependent on conditions, i.e. high temperatures may limit angular velocity), while a large water capillary end-effect is probable in the preparatory step on low-perm cleaned samples. Unstressed weak chalk samples may disintegrate in the centrifuge due to large centrifugal forces: need to be assessed on sacrificial samples before centrifuge runs on larger number of samples are conducted.
- Flooding: cheap; quick; for oil-brine, the capillary end-effect may be manageable with a significant viscous pressure drop. Viscous oil could be used to achieve S_{wi}, with the oil subsequently to be displaced by the crude proper before ageing commences.
- Evaporation method: cheap, fast (several days) and reliable (*see Springer 2003*). Salinity/salt deposition to be controlled through careful experimental design; lower limit for S_{wi} is around 0.1, more evaporation in practice brings about salt deposition near the outer surfaces, that may not re-dissolve easily later.

7.5. Practical Application

S_{wi} cannot be defined in the lab since it needs to be representative of the reservoir, i.e. corresponding to a particular height above the oil-water contact. This information has to come from the client, in an auditable fashion.

Caution should be taken when exposing tight chalk to crude oil. Filters used to prepare the oil should be considerably smaller than the dominating pore throat size found in the core sample. It is generally recommended to filter the crude oil through one or more “sacrificial plugs” of similar petrophysical properties. It is also crucial to keep the oil above cloud point throughout the experiment. Below this temperature, paraffinic components in the crude oil will precipitate as wax, which again has the potential to coat pore walls and artificially make chalk plugs less water wet and reduce permeability.

For LRQC, methods of choice to obtain S_{wi} are Porous Plate and Evaporation. Experimental design is essential for both techniques. Design S_{wi} preparation through Porous Plate with simulations (see Chapter on Numerical Simulation). A tool is available at jgmaas.com.

7.6. Reporting Requirements

- Documentation to justify chosen S_{wi} values.
- Design document to assess experimental parameters to be used for procedure.
- Production data vs. time, corrected for dead volumes, gravity head, etc.

7.7. Pitfalls

Note that because relative permeabilities and capillary pressures depend on wettability, so on representative S_{wi}, also the residual oil saturation depends on S_{wi}. Therefore, working with a non-representative S_{wi} is jeopardising all SCAL data.

Clients are not always aware that they are responsible for providing a best estimate for Swi, for individual plugs.

Obtaining S_{wi} through gas flow may suffer from viscous fingering with the gas channelling through the brine.

There appears to be undocumented observations of dry oil production in wells in chalk reservoirs with S_w above S_{wirr}. In such a case, S_{wcrit} would be used as a tuning parameter in reservoir simulation. Rather, it is recommended to critically review both laboratory and field data and find a way to use consistently S_{wirr} to be equal to S_{wcrit}. It may well be that the measurement protocol needs to be adapted to reflect transition zone behaviour as shown by *Masalmeh (2000 and 2007).* See also discussion on transition zone measurements in Section 9.5.

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8. Wettability Experiments

8.1. Abstract

The industry works mainly with two wettability indices only: the Amott-Harvey and the USBM index. It is known that these indices do not correlate well. Moreover, wettability is not an input parameter into reservoir simulators. Therefore, the indices should only be taken as a qualitative indicator.

8.2. Introduction

Wettability is assessed through the measurements of a wettability index. A variety of different indices is proposed in the literature, but industry practice focuses on only two indices: the Amott-Harvey index and the USBM index.

Wettability is difficult to quantify, which is already demonstrated by the fact that the Amott-Harvey and USBM indices do not correlate well *(Dixit et al. 1998).* Wettability indices are not used as an input parameter in reservoir simulations but serve to only broadly characterize reservoir rock.

Definitions

Amott-Harvey is determined (see Figure 8.1) by measuring basically four saturations: S_{wirr}, S_{spw}, S_{spo} and S_{or}. The trajectory between S_{wirr} and S_{spw} represents the spontaneous imbibition of water after primary drainage. At S_{spw}, the capillary pressure equals zero. Subsequently, a forced imbibition process moves the system from S_{spw} to S_{or}. At that point, oil can be allowed to spontaneously imbibe the plug to reach S_{spo}. At S_{spo}, capillary pressure equals zero again. A forced (or secondary) drainage process can then bring the system back to S_{wirr}.

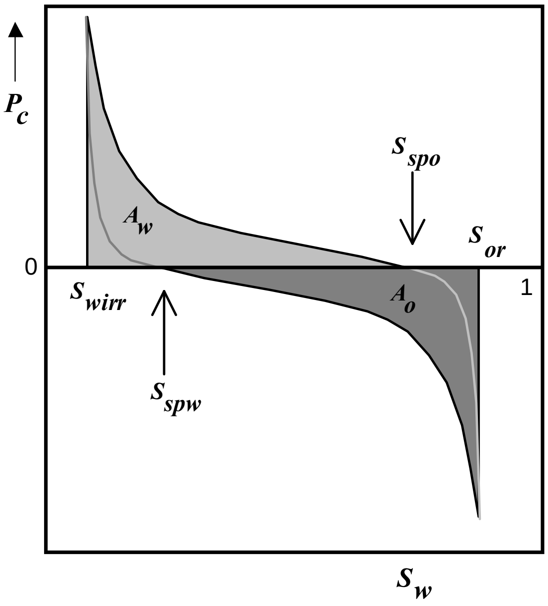

Figure 8.1: Wettability index parameters used for Amott-Harvey and USBM

The Amott-Harvey index is calculated through the water index I_w:

$$I_\mathrm{w} = \frac{\mathrm{S}_{spw} - \mathrm{S}_{wirr}}{(1 - \mathrm{S}_{wirr} - \mathrm{S}_{or}} \qquad \text{(Eq. 8.1)}$$

and the oil index I_o:

$$I_\mathrm{o} = \frac{\mathrm{S}_{spo} - \mathrm{S}_{or}}{(1 - \mathrm{S}_{wirr} - \mathrm{S}_{or}} \qquad \text{(Eq. 8.2)}$$

The combined index is given by:

$$I_{AH} = I_w - I_o \qquad \text{(Eq. 8.3)}$$

An Amott-Harvey index between +1 and +0.3 indicates a preferentially water-wet system, between +0.3 and -0.3 indicates intermediate or mixed wetness, and an index between -0.3 and -1 indicates a preferentially oil-wet system.

The USBM index is calculated from the areas A_w and A_o in Figure 8.1, using

$$U = \log\left(\frac{A_w}{A_o}\right) \qquad \text{(Eq. 8.4)}$$

An USBM index of around +1 indicates a preferentially water-wet system, an index of around zero indicates intermediate or mixed wetness, and an index of around -1 indicates a preferentially oil-wet case.

The parameters shown in Figure 8.1 are measured through a combination of laboratory techniques.

8.3. Recommendations

Methodology

1. It is generally recommended to perform wettability experiments on restored state core material (see Chapter on Initial Water Saturation). This will allow repeating the measurement to verify reproducibility. Fresh state wettability measurements cannot be repeated on the same sample.
2. Regular Amott-Harvey is the preferred wettability test on chalk samples, unless the absolute permeability is too low for reliable assessment of spontaneous imbibition and drainage within a reasonable amount of time. USBM tests are not as widely used as the Amott-Harvey tests. On the other hand, a combined Amott/USBM test could well be performed as an extension of a porous plate or centrifuge capillary drainage and imbibition experiment. Experimental design using numerical simulation is necessary to assess which method to employ (see Chapter on Numerical Simulation).
3. For a representative wettability, one needs to pay equal attention to the rock, brine and crude oil for these all to be representative of the reservoir.

Amott-Harvey and USBM Method

1. Plug selection and preparation at representative initial water saturation as per recommendations in Sections on RCA, Homogeneity Assessment and on Initial Water Saturation.
2. Given the expected low permeability for LRQC, it is critical to allow sufficient equilibrium time for spontaneous imbibition processes (spontaneous imbibition is used in the calculation of the Amott-Harvey index, or combined Amott/USBM index). Production plots vs. log time need to be used to assess stationary conditions. Frequent production recordings are recommended during the first 24 hours where imbibition may be strong.
3. Perform forced imbibition by a water displacing oil flooding experiment in steps of increasing differential pressure or by multi-speed centrifuge (see Chapter on Capillary Pressure for details).
4. Perform spontaneous imbibition of oil and secondary drainage using the same experimental logic as described for above.
5. Conduct interpretation-by-simulation of the experiments (see Chapter on Numerical Simulation).

8.4. Technology

Amott-Harvey Method

This method is commonly used in order to evaluate wettability. A wetting index is calculated from water saturations obtained after spontaneous and forced imbibition in water, spontaneous imbibition in oil followed by secondary drainage of oil *(Amott, 1959)*. As such, it is independent of the shape of drainage and imbibition curves.

At initial water saturation, a single plug is submerged in a glass beaker (imbibiometer) to spontaneously imbibe water. Volume of produced oil is recorded and the analysis continues until stability occurs. For tight chalk material, this process is expected to be delayed. In order not to under-estimate water-wet preference, it is critical to allow sufficient equilibrium time. A forced imbibition is subsequently performed, in most cases using a multi-speed centrifuge analysis. Given the low permeability for LRQC material, it is expected that residual oil saturation is not reached using a centrifuge. In such cases, it would be recommended to perform a water displacing oil flooding experiment in steps of increasing differential pressure. Conventional single-speed centrifuge by itself or flooding at one differential pressure do not allow proper validation of obtained residual oil saturation, and is not recommended.

A similar spontaneous imbibition in oil and secondary drainage is subsequently conducted. Chalk is known to be mixed wet with a tendency to water wet, and spontaneous imbibition in oil may be very small or even absent. If the oil part of Amott's test is performed, oil imbibition and secondary drainage by oil injection should continue until water saturation matches initial water saturation.

An alternative experimental route is to conduct the whole capillary loop in a porous plate set-up. To reduce experimental errors, the porous plate set-up may be modified to contain both a water-wet filter at one end and an oil-wet filter at the other end.

In any case, experimental design of each individual step through simulation is necessary. In this way, also the uncertainty in the end result can be estimated for both experimental routes (see Chapter on Numerical Simulation).

USBM Method

There is no well-established theoretical basis for selecting methodology for testing wettability, using either Amott-Harvey or USBM. The relationship between the two, if any, is unclear. USBM methodology considers not only the end point saturations, but it takes into account the shape of the forced imbibition and secondary drainage curves *(Donaldson et al, 1969)*. The corresponding index is calculated considering the energy required to perform brine and oil drive, i.e. the area defined by the capillary pressure curves. A combined Amott/USBM method was later published *(Sharma and Wunderlich, 1985)* that takes into account the spontaneous imbibition in water and oil.

To satisfactorily define the shape of the curves, a significant number of pressure points are required for both imbibition and secondary drainage, using the centrifuge or the porous plate method (see Chapter on Capillary Pressure). An option would be to perform a combined USBM and Amott test as an extension of a porous plate or centrifuge capillary drainage and imbibition experiment, possibly already programmed as part of a SCAL program.

Experimental design is necessary to decide on the pressure steps. In this way, also the uncertainty in the end result can be estimated.

Amott-Harvey results are more frequently reported in publications than USBM, and chalk is often only defined by its water wettability index.

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9. Relative Permeability Methods

9.1. Abstract

There is no single best method to measure relative permeabilities. Each method has its own strengths and weaknesses. Therefore, methods need to be combined. The ideal measurement program consists of flooding experiments (Steady-State and/or UnSteady-State) as well as single-speed centrifuge experiments. Even then, capillary forces interfere and make residual oil saturations to appear 10 to 15 saturation units too high. To unravel this interference, any relative permeability measurement program needs to include capillary pressure measurements. The combined data of all relative permeability measurements and capillary pressure measurements need then to be history matched with one common data set. Only by such an interpretation-by-simulation approach, representative data are generated, provided that sufficient care has been executed to obtain representative samples, at representative wettability and initial water saturation.

9.2. Introduction

LRQC relative permeability measurements are notoriously difficult and require special attention to laboratory procedures to obtain representative data.

Wettability brings about the fluid distribution within the pores: fluids are attracted by minerals lining the pore walls, or can be repelled from the surface. Therefore, wettability determines relative permeability, irreducible water saturation and residual oil. Figure 9.1 shows semi-log plots of typical oil-brine relative permeabilities for extreme water-wet, oil-wet and mixed-wet (intermediate wet) cases.

A convenient way to describe relative permeabilities is through the so-called Corey formulations:

$$k_{rw}(S_w)=k_{rwor}\left(\frac{S_w-S_{wirr}}{1-S_{wirr}-S_{orw}}\right)^{n_w} \qquad \text{(Eq. 9.1)}$$

and

$$k_{ro}(S_w)=k_{rowirr}\left(\frac{1-S_w-S_{orw}}{1-S_{wirr}-S_{orw}}\right)^{n_o} \qquad \text{(Eq. 9.2)}$$

with k_{rwor} and k_{rowirr} the end point relative permeability to water at residual oil saturation S_{or} and to oil at irreducible water saturation S_{wirr}, respectively; and n_w and n_o the water and oil Corey exponents.

It is important to realise that Eqs. 9.1 and 9.2 are correlations that often appear to describe reasonably well relative permeability data measured in the laboratory, but there is no physical law that would force measurements to follow Corey formulas. Occasionally deviations are noted and so-called dual-Corey-exponent formulations are used, with different Corey exponents n_w and n_o in different saturation ranges. Typical Corey parameter values are listed in Table 9.1.

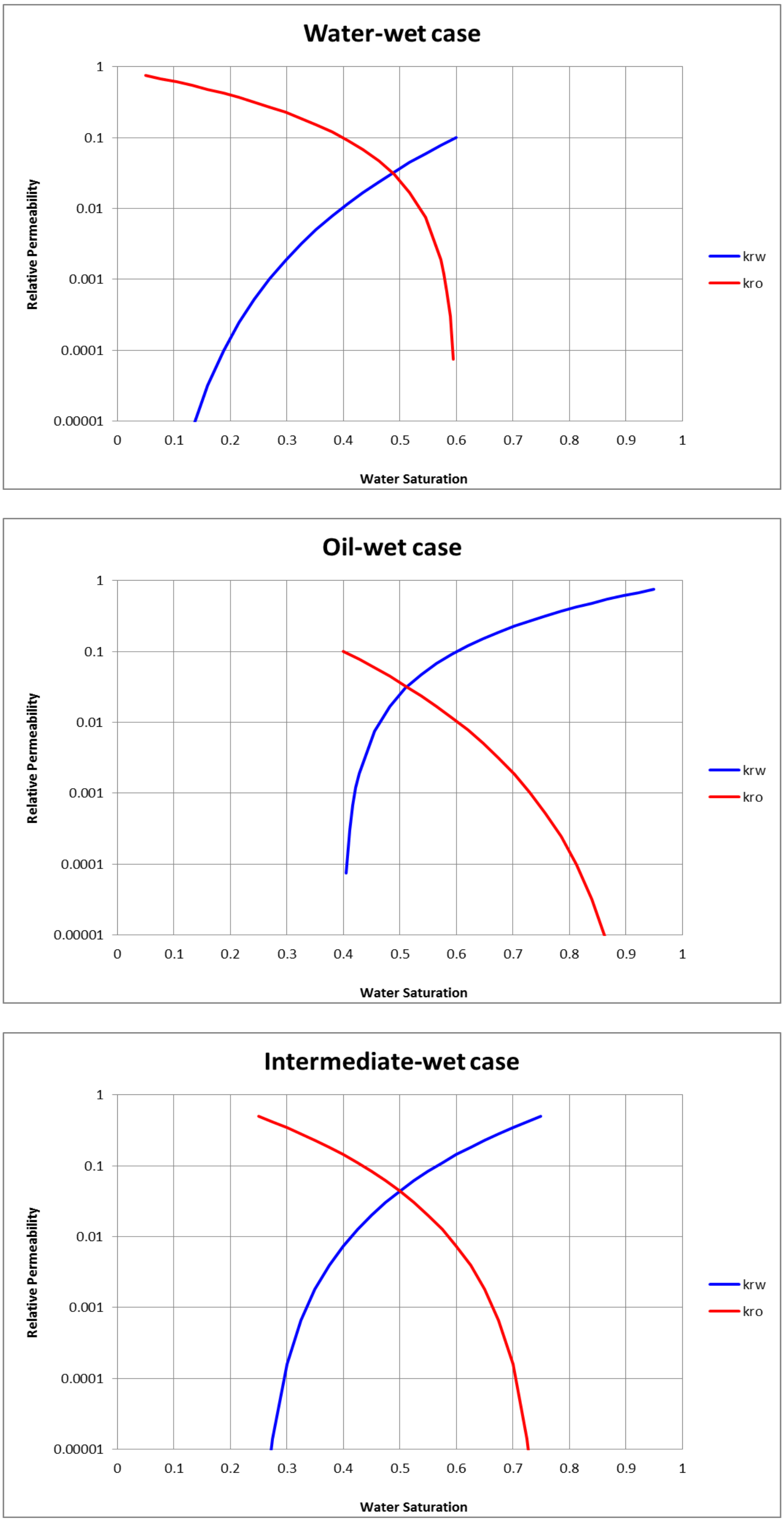

Figure 9.1: Typical shapes of the oil and water relative permeability for water-wet, oil-wet and mixed-wet (intermediate-wet) conditions.

Table 9.1: Range of Corey parameters for different wettabilities (Maas 2013). For oi- wet, the water-wet values may be used with oil and water changing places

Water wet	Mixed wet
$S_{wirr} \cong 0.1$or lower	$S_{wirr} \cong 0.2$
$S_{or} \cong 0.3$ or higher	$S_{or} \cong 0.2$
k_{rwor}: 0.1 – 0.4	$k_{rwor} \cong 0.5$
k_{rowirr}: 0.6 – 0.8	$k_{rowirr} \cong 0.5$
n_w: 4- 6	n_w: 3 – 5
n_o: 2 -3	n_o: 3 - 5

There have been a number of attempts to correlate Corey parameters to rock and fluid properties, but none of these correlations is reliable for oil-brine systems. Note that the Land correlation has been established for gas-brine, i.e. for perfectly water-wet systems.

Relative permeability measurements in the laboratory are analysed through analytical methods under the assumption that capillary forces are negligible. However, both for drainage and imbibition experiments, significant capillary forces exist that depend on wettability. On the scale of the core plugs in the laboratory, these forces give rise to saturation end-effects that may dominate flow, while on the scale of the reservoir such effects play usually a much smaller role, particularly in less heterogeneous reservoirs.

Summarising, analytical methods interpret zero flow as caused by zero relative permeability and, by definition, read the lowest oil saturation reached as the residual oil saturation. However, in reality zero oil flow is caused by capillary forces blocking the flow on the scale of the core plug, while the oil mobility is still non-zero. We are looking therefore at an interpretation issue. To unravel the interference between relative permeabilities and capillary forces, the experiments need to be history matched (see Chapter on Numerical Simulation) conducting an “interpretation-by-simulation”.

Local heterogeneities in a core plug may dominate flow, while the interpretation techniques (analytical and through simulation of the experiments) assume plugs to be homogeneous (see Chapter on Screening of Samples).

Relative permeabilities are also known to be saturation-history dependent and significant hysteresis has been observed between drainage and imbibition curves, particularly for the non-wetting phase.

For LRQC, the effect of overburden pressure on relative permeability may be significant, so measurements should be conducted at representative loading conditions. Several approaches are possible to load samples under stress (*Ewy et al., 2013*), and section 5.4 Advanced core measurements – test conditions.

Fractured reservoirs require a special approach for relative permeability data collection, with “conventional” data for the rock proper, and fractured reservoir simulation in so-called dual-permeability mode, employing straight-line relative permeability curves with artificial S_{wirr} and S_{or} for the fractures (*Por et al. 1989 and Horie et al. 1990*) to allow for so-called re-imbibition of rock matrix blocks.

9.3. Recommendations

Methodology

1. Ensure homogeneity of the plugs (see Chapter on Screening of Samples) and ensure representative S_{wi} and wettability (see Chapter on Representative Wettability and Initial Water Saturation) and representative brine composition (see Chapter on Core Water Salinity).
2. Dependent on the objective of the SCAL study, have focus on mid-saturation range or include measurements of the tail end of the oil relative permeability.
3. Mid-saturation range is to be addressed preferably by the Steady-State method, UnSteady-State (Welge) is second with possibly limited saturation range and larger sensitivity to heterogeneities.
4. The tail end of the oil relative permeability is to be addressed with the single-speed centrifuge technique.
5. Use experimental design, preferably by numerical simulation (see Chapter on Numerical Simulation) for each experiment. Notably, the time periods for each fractional flow setting in the Steady-State must follow from such design and should never be taken of constant length.
6. Relative permeability measurements need to be accompanied by capillary pressure measurements on twin plugs (see Chapter on Numerical Simulation), even if these capillary pressures would not be of interest on the scale of the field. Note that on the scale of the laboratory experiments, capillary pressure is known to seriously interfere with relative permeability measurements.
7. Data interpretation to be conducted through history matching of the experiments (see Chapter on Numerical Simulation).

General

1. Saturation measurements from material balance are acceptable for the UnSteady-State method, but in-situ saturation profiles through X-ray or gamma ray are preferred. Steady-State experiments require in-situ saturation readings because material balance is highly inaccurate. X-ray absorption enhancing dopants must be avoided due to the risk of interference with wettability.
2. Flooding experiments must be conducted with at least one “bump rate” at the end of the experiment.
3. Oil viscosity may be used as an optimising parameter in experimental design, as long as viscous fingering is avoided.
4. Intermediate reporting, particularly for Steady-State experiments will facilitate early detection of measurement problems.
5. Reporting needs to include production curves, both for quality management as well as for input for interpretation-by-simulation.

9.4. Technology

Four laboratory methods are available to measure relative permeabilities: the UnSteady-State (or Welge), the Steady-State, the Centrifuge Single-Speed technique and the FRIM technique. The main characteristics of each are presented below.

UnSteady-State or Welge Technique

This is a flow experiment where only one phase is injected, either at constant injection rate (usually for liquid-liquid relative permeability measurements) or at constant pressure drop (usually in gas-brine experiments). As a function of time, pressure drop and cumulative fluid injection are measured. Production flow rates of the individual phases, as a function of time, are measured using a phase separator. The method is cheap because only one pump is used and because it is relatively fast. However, only a limited water saturation range can be probed: from shock front saturation up to 70%. The relative permeability range is limited roughly down to 10^{-2} to 10^{-3}.

The saturation is derived from material balance through the so-called JBN analytical technique (*Johnson et al., 1959*) that also delivers the corresponding relative permeabilities. The saturations should preferably be verified and obtained in more detail through in-situ X-ray saturation monitoring. Gamma ray monitoring is operationally more complicated, because of higher noise levels that bring about long measurement times that would require the pump to be stopped.

Steady-State Technique

With the Steady-State (SS) technique, two fluids are injected at a given fractional flow. Differential pressure and saturation are measured once that a steady-state has been achieved. Subsequently, the fractional flow is adjusted for the next measurement. This technique is more time consuming than the UnSteady-State (USS) because of waiting on steady-state at each chosen fractional flow. The long duration and the fact that two pumps are needed make the technique more expensive than USS.

The water saturation range that is probed is wider than with USS, but still limited roughly from 30% to around 70%. Similar to USS, the relative permeability range is limited down to 10^{-2} to 10^{-3}.

Saturations measured from material balance are not reliable due to large volumes being put through and from differences in fractional volume in the lines compared to set pump fraction (for closed systems). In-situ X-ray saturation monitoring is required. As for USS, gamma ray monitoring is operationally more complicated, because of higher noise levels that force long measurement times.

Relative permeabilities are calculated analytically by a straightforward application of Darcy’s law.

Single-Speed Centrifuge Technique

This technique employs the Hagoort (1980) theory on gravity-drainage to measure the relative permeability of the expelled phase in a centrifuge experiment. This translates into the measurement of only k_{ro} in imbibition mode and of only k_{rw} in drainage mode. The method is expensive and is likely of long duration for LRQC low permeable rock.

The method is particularly suitable to measure tail-end relative permeabilities from roughly 10^{-3} to 10^{-6}. The measured parameters are cumulative production vs. rotational speed. Automated stroboscopic read-out of production and rotational speed is essential for reasonable data quality.

IFP’s FRIM Technique

The FRIM (Fast Resistivity Index Measurement) method is a modification of the Porous Plate method (see Chapter on Capillary Drainage and Imbibition Experiments) as discussed by *Fleury et al. (2005)*, limited to water saturations between roughly 0.6 and 0.85. Equilibrium needs to be achieved at each saturation point. Measured parameters are production, pressure drop and resistivity.

9.5. Practical Application

Relative permeability measurements must be conducted on samples that have been selected and prepared following recommendations discussed in the Chapters on “Sample Screening”, “Core Water Salinity and Simulated Formation Water” and “Representative Initial Water Saturation and Wettability”.

Flooding experiments cannot provide a good estimate of the residual oil saturation due to capillary end-effects. In practice, S_{orw} from USS or SS could well be 10 to 15 saturation units too high (*Kokkedee et al., 1996*). In addition, k_{ro} at high water saturations may well be one to two orders of magnitude too low. The single-speed centrifuge technique generates much better data for k_{ro} at high water saturation and for S_{orw}, but is less reliable in the mid-saturation range. The strengths and weaknesses of various methods need to be combined to cover the saturation range of interest. As a result, a relative permeability measurement program will consist both of (U)SS and single-speed centrifuge experiments.

Since data interpretation assumes 1-D flow, flooding experiments need to be gravity stable, so vertical. For imbibition experiments, injection is conducted from the bottom, for drainage experiments from the top.

In principle, in-situ saturation measurements can be conducted with gamma or X-ray sources. Photon energies of gamma sources are relatively low, which increases the absorption difference between oil and brine. This enhanced contrast would increase saturation accuracy. However, absorption at these energies is much stronger than at the higher photon energies that are achieved in X-ray sources. For reasonable data accuracy, measurement times to deal with photon statistics will be much longer with gamma rays than with X-rays.

Consequently, in-situ saturation measurements should be taken preferably with X-rays, and avoiding the use of dopants. Dopants are known to be surface active and may interfere with wettability. In addition, dopants may absorb onto the rock and interfere with the fluid absorption signal. Measurement accuracy is limited to some five saturation units in practice (even with dopants). Measurements without dopants require special attention to X-ray source and detector stability (both mechanical, electrical and thermal) and the use of a reference signal detector to normalise absorption data on the remaining X-ray source signal variations.

As mentioned above, all flow experiments suffer from capillary end-effects. To reduce the capillary end effect, both USS and SS should be run with at least one “bump” rate and, if possible and practical, more bump rates should be added at the end of the standard flow period(s). On the other hand, flow rates need to be low enough to avoid fines migration.

Experiments (both flow and centrifuge) need to be designed either 1) based on earlier lab experience with similar porosity/permeability plugs and similar viscosity oil and brine, or 2) by simulation using Corey parameters in the range typical of the expected wettability (see Chapter on Numerical Simulation). Oil may need to be replaced (after ageing with the relevant crude) by oil with a different viscosity if expected differential pressures are too high and limiting the injection rates possible to unpractical low values.

Interference with capillary end effect, even at high bump rates, remains likely and therefore there is a need for interpretation-by-simulation (after measurement of relevant imbibition or drainage capillary pressure). This history matching of experiments for improved data interpretation may have a significant impact, particularly for low permeability/high capillary pressure plugs (see Chapter on Numerical Simulation).

Relative Permeabilities for Transition Zones

The Steady-State, combined possibly with the Single-Speed Centrifuge, is the technique of choice for transition zone relative permeability measurements. USS experiments are less suited, because only one phase is injected and the saturations will start changing immediately. In addition, the saturation range in USS is more restricted than in SS. Measurement protocols to establish so-called scanning curves are further discussed by *Masalmeh and Abu Shieka in various SCA papers (2000-2009)*.

Relative Permeability Measurements with Gas

Flow experiments with gas are difficult to conduct. The low viscosity of gas brings about a low differential pressure, with comparatively large noise levels. For reasonable measurement accuracy, the flow rates will need to be increased to achieve a larger pressure drop. On the other hand, to avoid phase exchange, the gas needs to be equilibrated with the liquid(s) and this can only be done at one particular pressure level. Consequently, phase exchange cannot be ruled out anymore.

In SS experiments, large volumes are pumped through the core plug and phase exchange with a gas phase may well be very significant. This rules out SS experiments for gas systems. In USS experiments, apart from the phase exchange problem, the extreme viscosity ratios introduce other problems. In imbibition mode, due to the very low viscosity ratio, the shock front saturation will be very large and the saturation range that can be probed will be very small, typically less than five saturation units. In drainage mode, the shock front saturation will be small, but a serious risk will exist for viscous fingering due to the now very high viscosity ratio.

The single-speed centrifuge is the only technique left to measure relative permeabilities with gas. Only the forced drainage mode can be conducted, so oil or water draining with gas as invading phase. This limits the measurements to drainage k_{ro} or k_{rw}.

Gas-oil gravity drainage at S_{wirr} requires a special design: at reservoir conditions, oil is usually a spreading phase between gas and brine. In the centrifuge, oil is likely to be non-spreading. For centrifuge experiments to be representative of the field, the oil needs to be replaced by Soltrol that will spread between gas and brine at laboratory conditions (*Vizika and Duquerroix, 1997*).

Although, for technical reasons, it is not possible to measure reliably relative permeabilities in flooding experiments with gas, there is a work-around making use of the fact that wettability determines relative permeability and residual saturation. Gas-brine systems can be assumed fully water wet, with gas being the non-wetting phase. Such an extreme wettability situation can be simulated in the laboratory by cleaning the rock to a water-wet condition and then use in the flooding experiment brine and a non-wetting mineral oil like decane as the two fluids. A non-miscible liquid-liquid experiment will not suffer from phase exchange and is expected to deliver representative relative permeabilities because the wettability is representative for a gas-brine reservoir fluid system.

9.6. Reporting Requirements

For flow experiments: Injection and production data as a function of time, together with differential pressure as a function of time, all corrected for dead volumes. Differential pressure data corrected for gravity head.

For Steady-State: The criterion used to establish steady-state needs to be documented. That criterion should refer to stable differential pressure within signal noise limit, over a certain period of time, e.g. half a day, together with stable production rates of water and oil.

For the centrifuge: Production data vs. time, dead volume corrected and speed corrected level detected. Centrifuge start-up time characteristics, as well as the criterion used to end the centrifuge experiment need to be reported.

9.7. Pitfalls

Samples must be homogeneous, quantitatively assessed by CT *(Maas and Hebing, 2013*), and aged with representative crude and brine. UnSteady-State is known to be more sensitive to heterogeneity than Steady-State experiments (*Zweers et al. 1999*).

For increased volumetric precision, sometimes composite or “butted” core may be considered, but this increased precision brings about complexity in the experimental procedures. A first problem area is ensuring good capillary contact between the plugs: attention needs to be paid to well-trimmed end-faces of each plug and axial force applied to bring and keep the plugs together.

In addition, experiments on composite core require pressure taps at each plug boundary for a meaningful interpretation-by-simulation, and even then can only be interpreted properly in Steady-State experiments, with in-situ saturation monitoring on each individual plug.

In an UnSteady-State experiment on a composite core, the collective samples act as one large sample for which the outflow fractional flow is measured, at the end of the last plug only. For a meaningful interpretation-by-simulation, it is then necessary that the collective plugs would obey the chosen quantitative homogeneity criterion. This homogeneity condition may be difficult to achieve in practice.

Divalent ion composition in the brine needs to be checked (*Scheuerman, 1990*) for rock (mineral) compatibility.

Any S_{orw} determined without interpretation-by-simulation represents really only “remaining” oil saturation and is likely to be strongly dependent on experimental conditions.

Simulation of a flow experiment in isolation or of only a centrifuge experiment cannot be seen as interpretation-by-simulation, because the history match is non-unique in such cases.

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10. Numerical Simulation

10.1. Abstract

It is imperative that laboratory experiments for advanced core analysis be designed for optimal data generation, certainly in the case of LRQC where low permeabilities usually slow down the measurements significantly and costs of the individual measurements are increased because of the longer duration. Simulation of the experiments is a good tool for such design.

Equally important is interpretation-by-simulation of flow and centrifuge experiments. This amounts to history matching several experiments in parallel, with one common data set for relative permeabilities and capillary pressure, to unravel the experimentally unavoidable mutual interference between capillary pressure and relative permeabilities in each experiment. Without this state-of-the-art interpretation technique, wrong data are extracted: e.g. routinely residual oil saturations from analytical-only interpretations are 10 to 15 saturation units too high *(Maas et al. 2011)*.

Several simulation tools are available, and several service providers offer this interpretation service.

10.2. Introduction

Very low permeability chalk may require very long experimental times in a number of SCAL experiments; numerical simulation is a powerful tool in planning it right.

Secondly, interpretation-by-simulation has to be used to unravel the mutual interference of relative permeability and capillary pressure in a SCAL flow experiment: the measurement of the relative permeability is influenced by capillary pressure and vice versa *(Nordtvedt et al. 1999, Poulsen et al. 2000 and Sylte et al. 2004)*. History matching just a Welge or Steady-State experiment does not provide enough constraints for the possible combinations of relative permeabilities and capillary pressures. The same holds for history matching a multi- or single-speed centrifuge experiment. This is mainly due to the fact that each of these experiments is designed to probe only for relative permeabilities or for capillary pressure individually. This is resolved through history matching a flow experiment (designed to measure relative permeabilities) on one plug and e.g. a multi-speed centrifuge experiment (designed to measure capillary pressure) on a twin plug[4], and to ensure a simultaneous match be obtained with one common data set for relative permeabilities and capillary pressure.

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[4] Preferably taken from the same depth, having similar porosity and permeability

10.3. Recommendations

1. Use numerical simulations to screen and design measurement techniques. SCORES (license free), Cydar and Sendra can all be used, dependent on availability. All experiments (flooding, including porous plate, centrifuge) can be simulated with today’s simulators.
2. Interpretation-by-simulation history matching is recommended for reliable residual oil saturation measurement and reliable oil mobility at high water saturations. Residual oil saturations are known to be too high by 10 to 15 units if not interpreted by simulations.
3. Interpretation-by-simulation requires the production curves: time series of injected and produced fluids and of the pressure drop across the core plug in a flow experiment, or of the speed table in a centrifuge experiment.
4. History matching needs to be conducted on several experiments in parallel: at least one relative permeability measurement in combination with one capillary pressure measurement on a twin plug. The simulations are conducted with one common data set for relative permeabilities and capillary pressure.
5. For reliable tail-end oil relative permeabilities, the parallel history matching will include a single-speed centrifuge experiment (see Chapter on Relative Permeability measurements).
6. When automatic history matching software is available, it will improve quality and reduce cost of interpretation.

10.4. Technology

The interpretation-by-simulation approach is documented by a growing list of papers and offered by several service providers using dedicated simulators, which usually also can be used for experimental design. Tools available: Cydar *(Cydarex website)* or SCORES[5] *(Maas and Schulte 1997, Maas et al. 2011)* for design and interpretation by history matching. Automatic history matching tools are now also on the market (Sendra from Weatherford) or available soon (SCALAnalyser, under development jointly by TNO and Panterra Geoconsultants, *(Loeve et al. 2011)).*

10.5. Practical Application

Experimental Design

SCAL service provider and client need to work together on an agreed experimental design for SCAL experiments, particularly on low permeable chalk. A balance needs to be found between acceptable data quality and acceptable time period involved in flooding or centrifuging. In practice, the access to simulator technology may already be used as a selection criterion for service providers.

To demonstrate how simulations can be used to design SCAL experiments, a number of cases are presented below. Data of an Ekofisk Fm chalk plug and fluids are listed in Table 10.1. Table 10.2 shows the measured primary drainage capillary pressure curve (all data courtesy GEUS).

Table 10.1: Core plug data and fluid properties

Absolute permeability	0.472 mD
Porosity	.339
Length	4.43 cm
Diameter	3.80 cm
Brine density	1031 kg/m^3
Brine viscosity	1.035 cP
Oil density	763 kg/m^3
Oil viscosity	1.33 cP

The primary drainage capillary pressure data in Table 10.2 were generated in a special long-duration Porous-Plate experiment run at overburden stress conditions in a core holder. Saturation development over time was tracked continuously to ensure that only a next pressure step was implemented when the rate of change in saturation was below 0.3 %PV/d, for the highest-pressure steps until production ceased. The experiment was halted after 6.4 months due to operational constraints. Figure 10.1 shows the production data obtained in the experiment. The highest pressure used in the experiment is 85 psi (5.86 bar); the data in Table 10.2 at lower S_w are extrapolated.

Analysing the experiment, Corey parameters were estimated for the primary drainage brine and oil relative permeabilities. Note that these parameters are only approximate, because from a single experiment no unique parameters can be extracted (see also discussion below in the Section on Interpretation-by-Simulation). These parameters are typical for water-wet rock (see Table 9.1), as expected to occur at primary drainage and are listed in Table 10.3

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[5] SCORES simulations free of charge available at jgmaas.com

Table 10.2: Primary drainage capillary pressure, including extrapolated data (in italics)

S_w	P_c(psi)
0.1	*500 (34.48 bar)*
0.2	*200 (13.79 bar)*
0.29	85 (5.86 bar)
0.33	80 (5.52 bar)
0.41	75 (5.17 bar)
0.49	70 (4.83 bar)
0.55	65 (4.48 bar)
0.73	60 (4.14 bar)
0.9	55 (3.79 bar)
0.98	50 (3.45 bar)
0.995	45 (3.10 bar)
0.996	40 (2.76 bar)
0.998	35 (2.41 bar)
1	30 (2.07 bar)

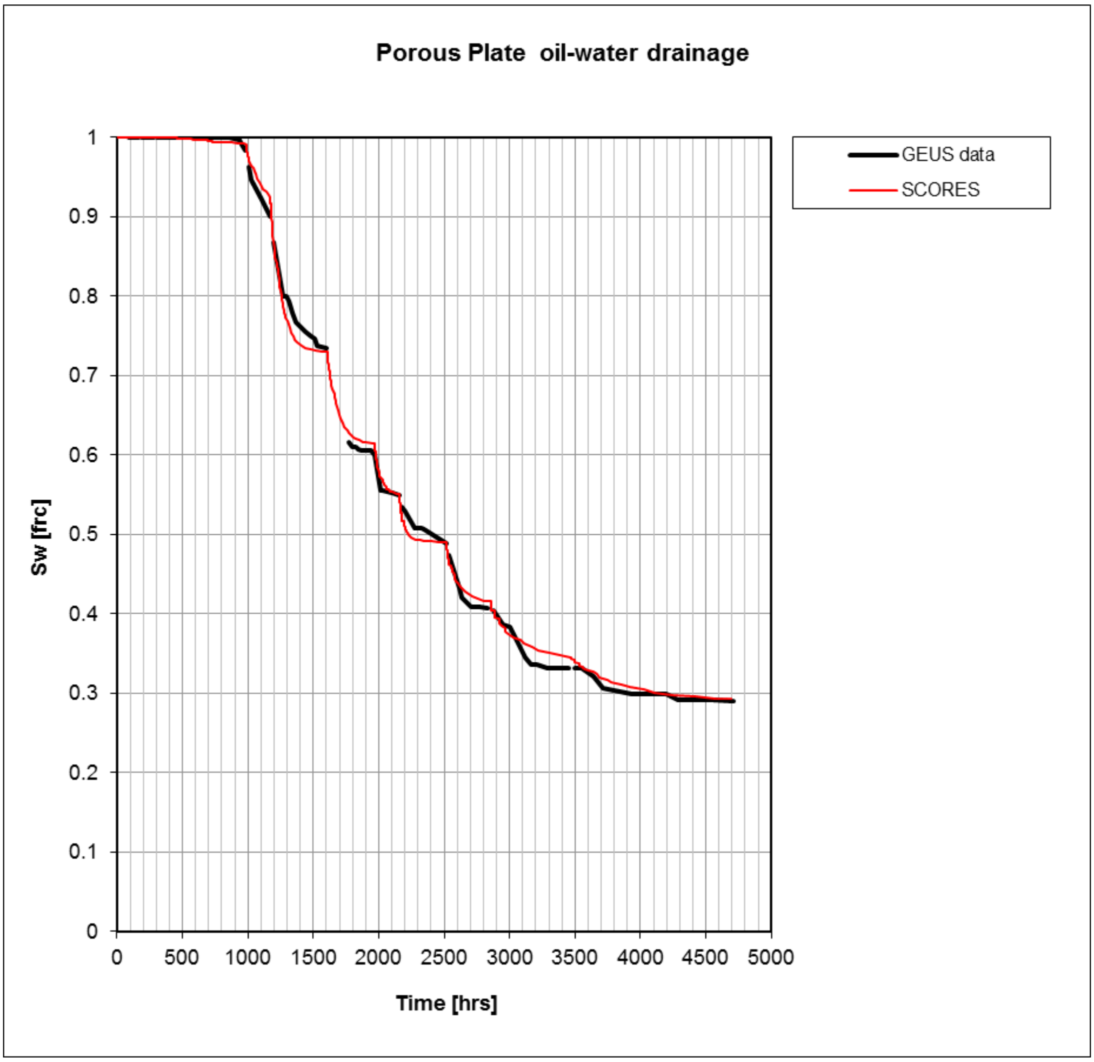

Figure 10.1: Porous-Plate experiment with long-duration equilibration times, experimental data compared with SCORES simulation results

Table 10.3: Corey parameters for the primary drainage relative permeabilities used in the experimental design, as derived analysing the actual Porous-Plate experiment to measure the primary drainage capillary pressure listed in Table 10.2

S_{wc}	0.1
S_{or}	0.0
k_{rwor}	1.0
k_{rowc}	0.7
n_w	5
n_o	2

Figure 10.1 shows how a SCORES simulation duplicated the actually measured production profile quite well, using the data in Table 10.1, Table 10.2 and Table 10.3. Subsequently, the simulation was extended with additional pressure steps, Figure 10.2, to show how the experiment could have been continued, based on the extrapolated capillary pressure data in Table 10.2.

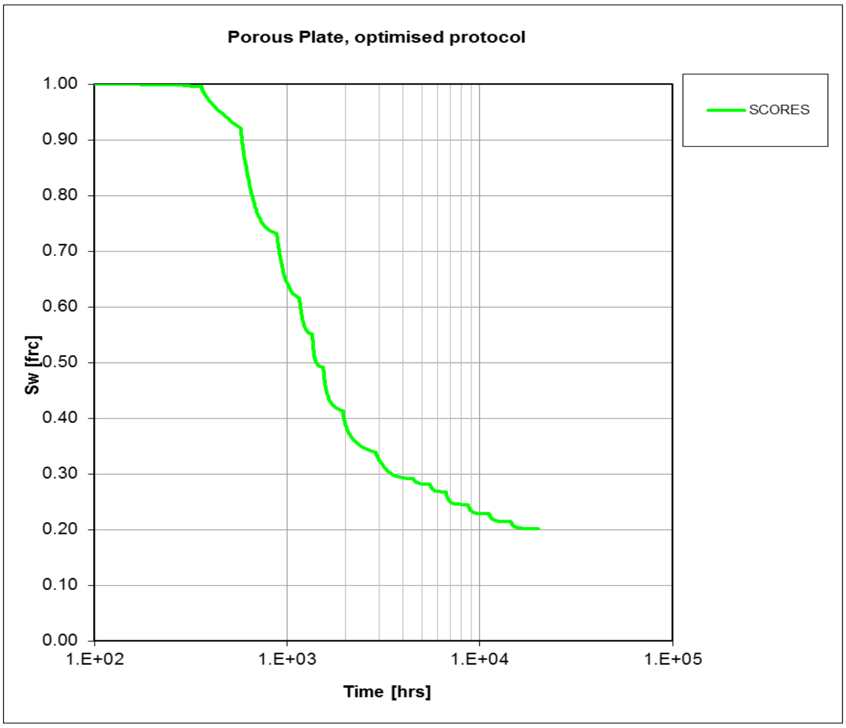

Figure 10.2: Porous Plate experiment with an optimised protocol for an extended experiment, with a maximum pressure level set at 200 psi (13.79 bar)

The times at which a change of pressure level was implemented in the simulations initially were chosen such that all levels were very likely to achieve equilibrium. With that simulated production profile, it then was possible to construct an optimal protocol to achieve the lowest saturation while remaining below an assumed entry pressure of the porous plate of 45 bar. The optimal protocol is shown in Figure 10.2 and extends to 20000 hrs (833 days, or just over 2 years). Clearly, this is completely impractical in the laboratory. On the other hand, such low water saturations and high capillary pressures may well come about in the reservoir where a hydrostatic equilibrium is established over geological times. Experimental design by simulation is therefore a tool to screen techniques that are available in the laboratory to obtain representative initial water saturations within realistic time frames.

As a sensitivity test, the simulations were repeated on a core plug that was given a permeability of ten times higher than listed in Table 10.1, with all other data (including capillary pressure) left unchanged. The simulated production curve is shown in Figure 10.3.

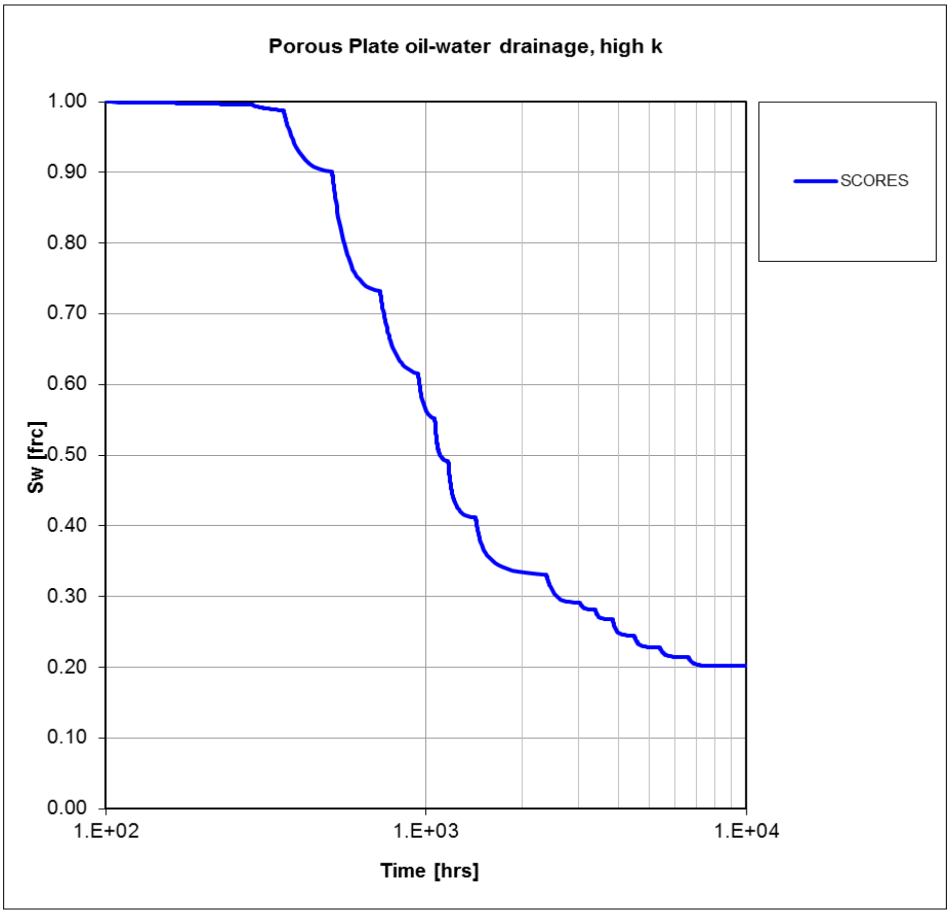

Figure 10.3: Simulation of a synthetic Porous-Plate experiment with a plug that has a permeability of ten times higher than the case shown in Figure 10.1, all other data kept unchanged

Clearly, the experiment is now significantly faster, but not faster by a factor ten. This is caused by the unchanged and therefore low permeability of the plate. The effect of an increase in absolute permeability on the measurement duration of a given protocol is difficult to predict without simulations of the experiment.

Establishing S_{wi}

All SCAL experiments will start to prepare a plug at initial water saturation S_{wi}. As discussed in Chapter 7, Representative Initial Water Saturation (Swi) and Wettability, S_{wi} depends on the height above the OWC and needs to be specified by the operator.

We will consider three different values: 1) S_{wi}=0.15; 2) S_{wi}=0.2; 3) S_{wi}=0.25[6].

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[6] From $P_c(S_w)=\Delta\rho gh$ ($\Delta\rho$ being the density difference between brine and oil) at hydrostatic equilibrium, and the data in Table 10.1 and Table 10.2, we can establish the height h above the OWC corresponding to these three cases as: 787 m, 525 m and 315 m respectively. For gas-brine, the height above the GWC would be around 270, 180 and 110 m respectively

Experimental design with the simulator SCORES has been conducted on the various techniques available to prepare a plug at S_{wi}. All simulations consider a core plug and fluids with properties as listed in Table 10.1, Table 10.2 and Table 10.3.

S_{wi} established by Porous-Plate

To achieve S_{wi}, with values as chosen above, the Porous-Plate experiments would need to be operated at a pressure of 300 psi (20.69 bar), 200 psi (13.79 bar), and 120 psi (8.28 bar) respectively (see Table 10.2). In practice, one would select a pressure level and observe the saturation development in the laboratory over time. If the target S_{wi} would be reached prematurely, one would then stop the experiment at that time. In the case that the target S_{wi} would not be reached at equilibrium conditions, one would increase the pressure level and continue to observe saturation changes as before, and halt the experiment or go to yet another level as appropriate. Figure 10.4 shows the saturation development of a Porous Plate protocol with chosen pressure levels of 100 psi (6.9 bar) up to six hrs, 200 psi (13.79 bar) up to 12 hrs, 400 psi (27.59 bar) up 18 hrs, and finally 600 psi (41.38 bar) until the end of the experiment, respectively. The time required to achieve the three chosen S_{wi} values is listed in Table 10.4

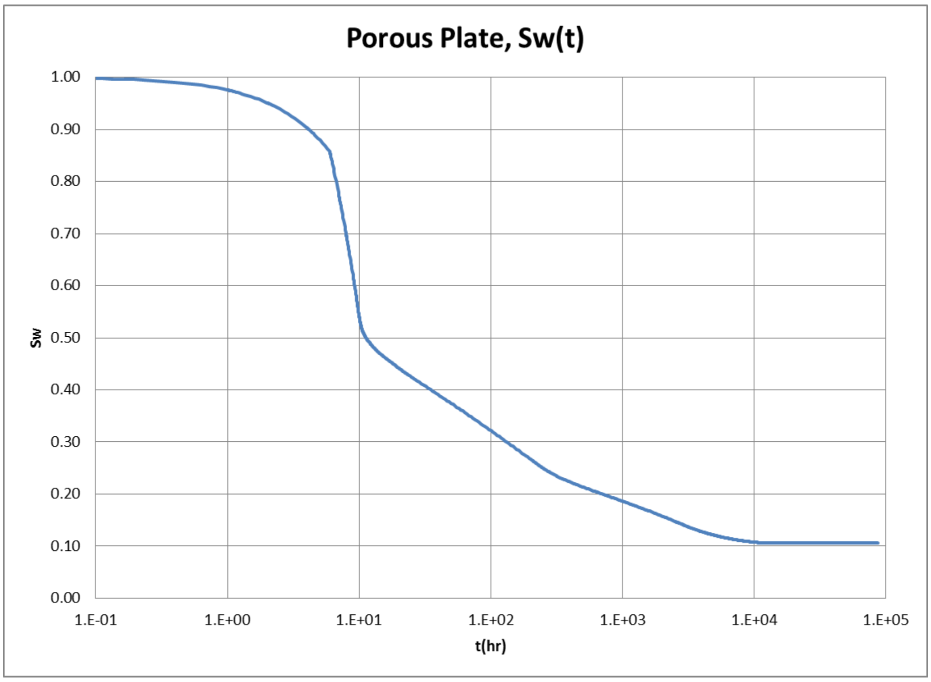

Figure 10.4: Water saturation development in a Porous-Plate experiment to achieve low S_{wi}

Data such as presented in Table 10.4 will facilitate judging whether a laboratory technique such as the Porous-Plate is a viable technique to achieve a required S_{wi}.

Table 10.4: Duration needed (see Figure 10.4) to achieve a chosen S_{wi}, assuming a core plug that has properties as listed in Table 10.1, Table 10.2 and Table 10.3

S_{wi}	Time (days)
0.15	99
0.2	29
0.25	10

S_{wi} established by UnSteady-State

While with a Porous-Plate experiment one needs to balance the capillary pressure at S_{wi} by the injection pressure, in UnSteady-State experiments, in a first order approximation, the viscous pressure drop during injection needs to balance capillary pressure at S_{wi}. Mechanical integrity of the core plug limits the maximum pressure drop, and therefore the maximum injection rate is limited. Figure 10.5 shows the production profile of an UnSteady-State experiment at three different injection rates: 10 cm^3/hr, 50 cm^3/hr and 100 cm^3/hr. Note the maximum pressure drop across the core plug at water breakthrough. The time required to achieve the three chosen values for S_{wi} are listed in Table 10.5.

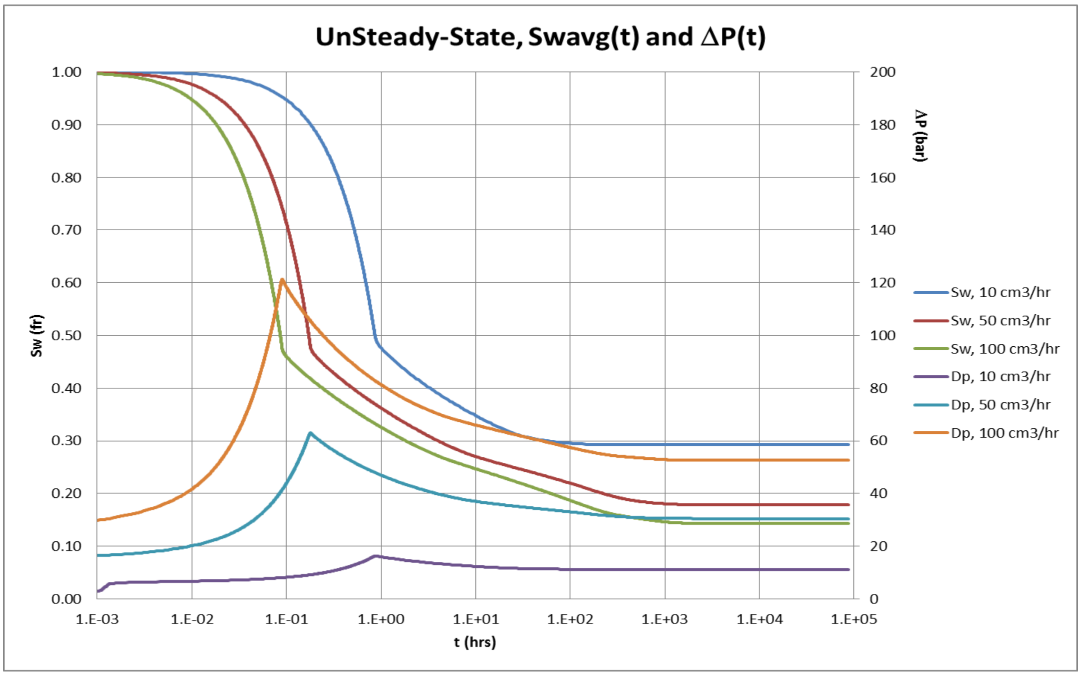

Figure 10.5: Simulated saturation development of an UnSteady-State experiment to achieve low S_{wi}, at different injection rates. Core plug and fluid data are listed in Table 10.1, Table 10.2 and Table 10.3

An important difference with the Porous-Plate experiments is hidden inside the core plug: the saturation profile along the length of the core plug. Figure 10.6 shows the saturation profile as calculated by SCORES for an UnSteady-State experiment at 50 and 100cm^3/hr, compared with the saturation profile as calculated for the Porous-Plate, with all experiments aiming to achieve S_{wi}=0.2. Note that aiming to achieve a saturation means that in the laboratory, the experiment is halted when the average saturation has reached the target value.

Table 10.5: Duration (days) needed to achieve a chosen S_{wi}, at different injection rates with the synthetic UnSteady-State experiment (rounded to nearest half day).

S_{wi}	T (days)		
	q_i= 10 cm^3/hr	q_i = 50 cm^3/hr	q_i = 100 cm^3/hr
0.15	Not possible	Not possible	27.5
0.2	Not possible	9.5	2.5
0.25	Not possible	1	0.5

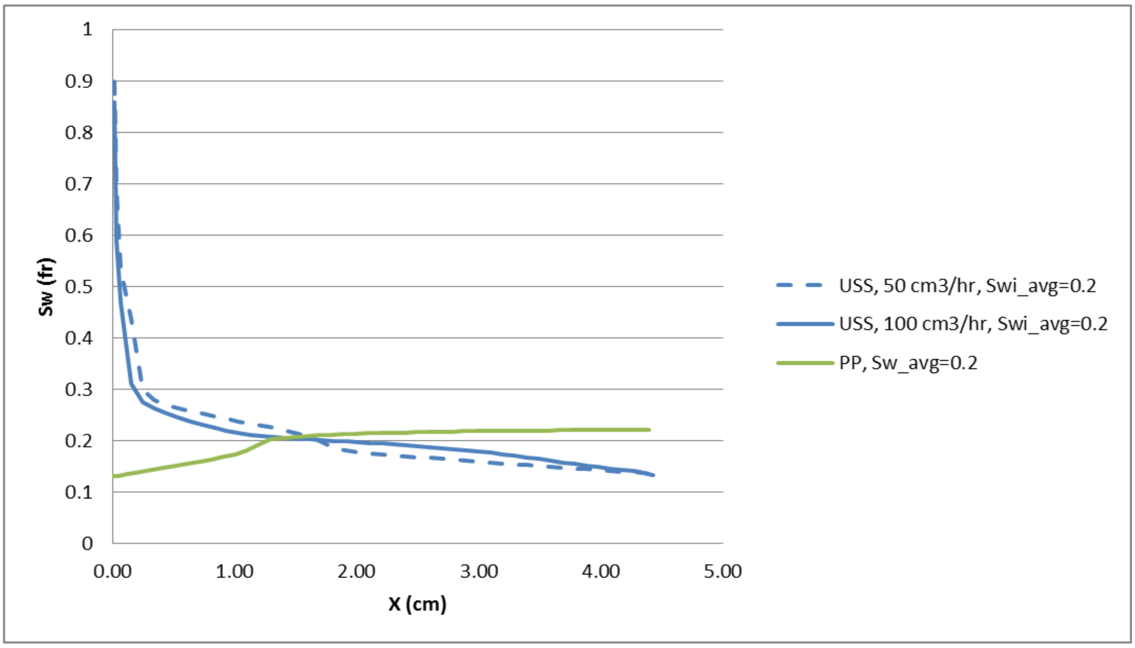

Figure 10.6: Comparison of saturation profiles in the core plug, when aiming for S_{wi}=0.2, using the UnSteady-State technique and the Porous-Plate technique. The outflow end of the core plug is at X=0.

Clearly, with the UnSteady-State experiment a significant capillary end effect is brought about, even at high flow rate (100cm^3/hr roughly corresponds to 10 PV/hr). Therefore, it is recommended to do reverse injection at the end of the injection period[7], to reduce the end effect at the original out-flow face.

In the Porous-Plate experiment, the end-effect is moved into the plate. However, the oil that cannot surpass the entry pressure of the plate will build-up against the plate and distort the saturation profile in the plug, particularly when large pressure steps are used in the Porous-Plate experiments.

S_{wi} established by Continuous Injection

Simulations of the Continuous Injection technique applied to the same core plug and fluids as above showed that injection at fixed rates may easily result in highly distorted saturation profiles along the core plug and even in surpassing the entry pressure of the plate at relatively low oil saturations. An elaborate scheme of flow rate adjustments (to change the injection rate to lower and lower values, each constant for a limited period of time) was required. This is not really bringing an improvement over what can be done with the Porous-Plate technique, both in view of the total time required to achieve Swi and in view of the resulting saturation profile. Figure 10.7 shows a typical saturation profile as generated with the Continuous Injection technique.

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[7] Note that for gravity-stable reverse oil injection, the core holder should be re-mounted upside-down.

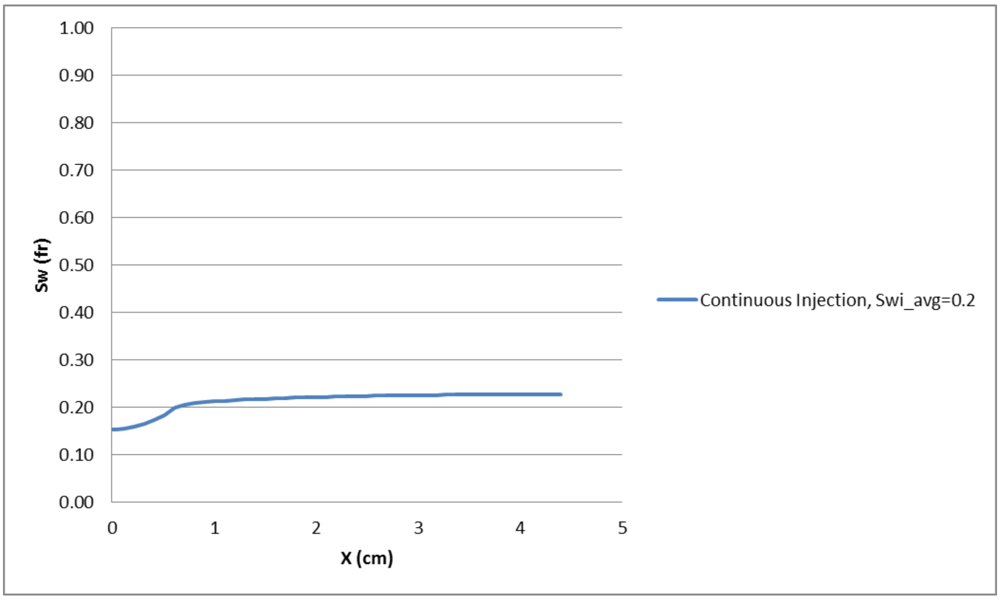

Figure 10.7: Saturation profile along the core plug, when aiming for S_{wi}=0.2 with a Continuous Injection experiment. The outflow end of the core plug is at X=0

S_{wi} established by Centrifuge

With the centrifuge technique, the maximum achievable capillary pressure depends on $\Delta\rho$ (density difference between brine and oil), maximum RPM and on R_m, the distance of the centre of the core plug to the centre of rotation. For drainage, R_m is typically around 9cm and the maximum RPM is in the order of 16000[8]. This results in drainage capillary pressure at the inflow face corresponding to saturations below 0.2, but the average water saturation in the core plug with data listed in Table 10.1, Table 10.2 and Table 10.3 is much higher. Therefore, a significant saturation-height distribution remains in the core plug at the maximum attainable speed. This makes the centrifuge for the plug under consideration not a realistic alternative to the Porous-Plate or the UnSteady-State to achieve a chosen S_{wi}. In addition, the equilibration time in the centrifuge turned out to be some 1000hrs (41 days), which would bring the cost of such centrifuge experiments to values many times more than charged for the Porous-Plate or for the UnSteady-State.

Design of Amott wettability index experiments

Once that a core plug is brought to S_{wi}, the plug will be aged to facilitate subsequent SCAL experiments to be conducted at representative wettability (see Chapter 7, Representative Initial Water Saturation (Swi) and Wettability). After ageing, wettability can be tested through the Amott or USBM method. We will now demonstrate how an Amott test can be designed through simulation.

As discussed in Chapter 8, Wettability Experiments, Amott tests are conducted to measure specific saturation points on the capillary pressure loop encompassed by the primary imbibition capillary pressure curve (starting at S_{wi}), and the secondary drainage capillary pressure curve. Each of these curves consists of a spontaneous and forced fluid invasion part (see Figure 10.8).

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[8] Note that in some centrifuges the core plugs can be spun at a net overburden of some 5000 psi, but at the cost of a lower maximum RPM. Secondly, in imbibition, both the R_m and the maximum RPM are different and need to be verified with the service provider.

As an example, we performed simulations on the same plug as discussed above. S_{wi} was set at 0.16. For the spontaneous imbibition after ageing, we used synthetic primary imbibition relative permeability (see Table 10.7) and a synthetic capillary pressure curve (see Table 10.6) for a mixed-wet case[9].

Table 10.6: Corey parameters for brine and oil in primary imbibition after ageing, for a typical mixed-wet case (see Table 9.1)

S_{wc}	0.15
S_{or}	0.15
k_{rwor}	0.4
k_{rowc}	0.6
n_w	3
n_o	4

Table 10.7: Mixed-wet primary imbibition capillary pressure curve, with S_{wc} and S_{or} consistent with data in Table 10.6

S_w	P_c (bar)
0.1509	320.856
0.155	31.4199
0.159	2.47629
0.197	0.546713
0.235	0.225117
0.274	0.144718
0.312	0.064319
0.351	0.048239
0.389	0.03216
0.427	0.01608
0.466	0
0.504	-0.01608
0.543	-0.03216
0.581	-0.04824
0.62	-0.06432
0.658	-0.0804
0.696	-0.41003
0.735	-0.73967
0.773	-32.8992
0.812	-322.335
0.85	-1000

Note that the imbibition capillary pressure curve crosses the $P_c=0$ line at $S_w=0.47$. Spontaneous imbibition usually is performed by submerging the core plug in brine in a glass vessel in the laboratory. We have simulated this procedure by using SCORES in centrifuge mode, with a choice of RPM and R_m to obtain $g=9.81 m/s^2$, i.e. imbibition at average earth gravity. The simulated water development is shown in Figure 10.9.

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[9] Ageing usually brings about some degree of mixed wetness, see Chapter 7, Representative Initial Water Saturation (Swi) and Wettability

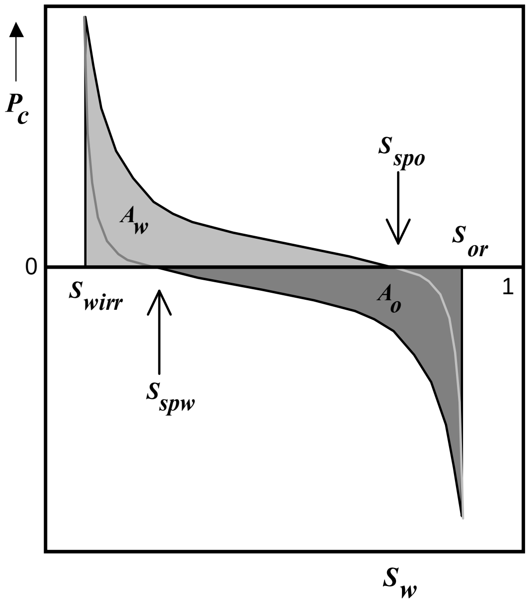

Figure 10.8: Capillary pressure loop and saturation points used in the Amott wettability assessment (see also Chapter 8, Wettability Experiments).

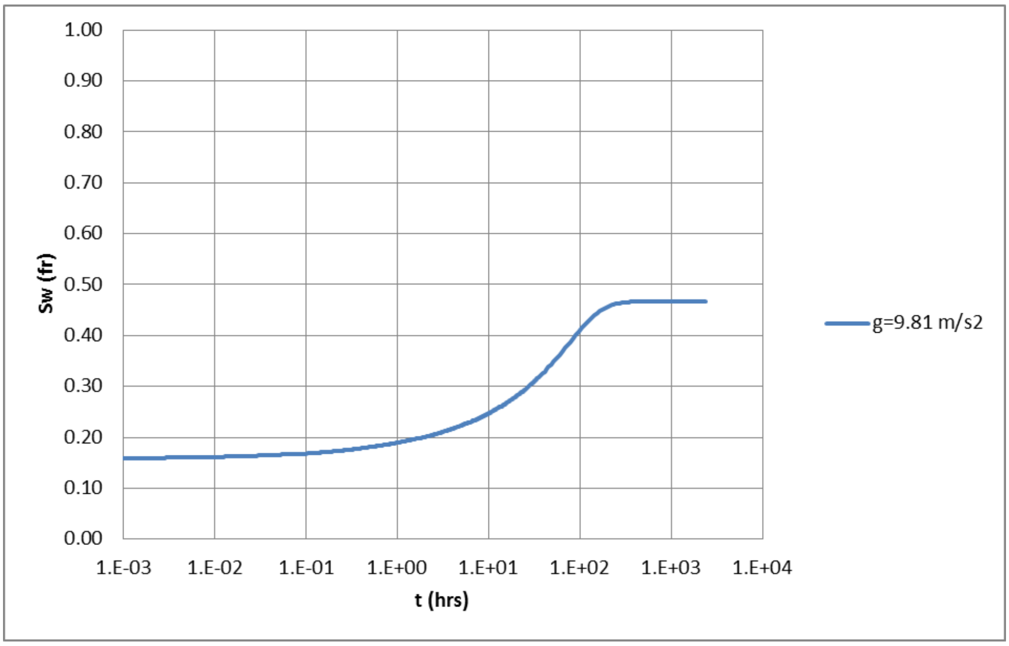

Figure 10.9: Average water saturation development during simulation of spontaneous imbibition, based on data in Table 10.1, Table 10.6 and Table 10.7.

We observe that S_{wi} increases from 0.16 to 0.2 in two hrs; for S_{wi} to increase to 0.25 it takes an additional seven hrs and S_w=0.30 is observed after 1.2 days. It takes in total 15 days for the water saturation to reach the capillary pressure zero-crossing point at S_w=0.47. Note that the capillary pressure has a non-zero slope at S_w=0.47 and that the speed of spontaneous imbibition at this point is proportional to this slope. There is little information in the literature on spontaneous imbibition capillary pressure curves, particularly close to the zero-crossing point. Therefore, for a more accurate estimate (i.e. design) of the spontaneous imbibition period it is recommended to collect imbibition capillary pressure data using a modified Porous-Plate experiment: one that has the plug sandwiched between a water-wet plate and an oil-wet plate. After in-situ ageing at S_{wi}, imbibition capillary pressure can be measured both for the spontaneous and for the forced imbibition part.

For the secondary drainage, capillary pressure similar simulations should be set up. Given the analysis of a primary imbibition experiment, it is clear that when in the laboratory no oil seems to be spontaneously moving into the core plug over a week or so, this may well be caused by a small slope of the secondary drainage capillary pressure curve close to the zero-crossing point of that curve. Before we can conclude from such a laboratory observation that the particular core plug is water wet, we need first to assess the secondary drainage capillary pressure curve in a modified Porous-Plate set-up with plates at either side of the core plug as mentioned above.

Interpretation-by-Simulation

The goal is to history match the experiments against production data (in many cases must be separately asked-for from the service provider, at possibly a surcharge) and saturation profiles (particularly for Steady-State, because material balance is not very reliable at high throughput), with one common data set for relative permeabilities and capillary pressure measured on a twin set of plugs. The conventional, analytically derived, relative permeabilities and capillary pressure are the starting point for the history match procedure. The known strengths and weaknesses of each method provide constraints to adjusting the analytically derived data. The procedure is not straightforward in practice, even with automated tools.

In our experience, the results provided by interpretation-by-simulation are often significantly different from the original analytical interpretation. For instance, the newly derived residual oil saturations are routinely 10 to 15 saturation units lower than the analytical data. This has serious effects on movable oil, and therefore also on scope for EOR (*Maas et al. 2011*).

As an example, we have simulated with SCORES a synthetic UnSteady-State experiment, based on an actual chalk core plug. The data on rock and fluids are listed in Table 10.8, with Corey parameters in Table 10.9 and the imbibition capillary pressure listed in Table 10.10. Note that the residual oil saturation is set at 0.15. The simulated production data are shown in Figure 10.10.

Table 10.8: Core plug data and fluid properties used in a synthetic UnSteady-State experiment

Absolute permeability	0.5 mD
Porosity	.28
Length	4.5 cm
Diameter	3.81 cm
Brine density	1050 kg/m^3
Brine viscosity	1 cP
Oil density	750 kg/m^3
Oil viscosity	1.5 cP

Table 10.9: Corey parameters for the primary imbibition relative permeabilities used for the synthetic UnSteady-State experiment. Note that these data are typical for a mixed-wet core plug (see Table 9.1)

S_{wc}	0.1
S_{or}	0.15
k_{rwor}	0.25
k_{rowc}	0.6
n_w	4
n_o	2

Table 10.10: Primary imbibition capillary pressure used in the synthetic UnSteady-State experiment, with S_{wc} and S_{or} consistent with data in Table 10.9

S_w	P_c
0.1	141.659
0.104	13.872
0.108	1.09329
0.149	0.241376
0.191	0.09939
0.232	0.063894
0.273	0.028397
0.314	0.021298
0.355	0.014199
0.397	0.007099
0.438	0
0.479	-0.0071
0.52	-0.0142
0.562	-0.0213
0.603	-0.0284
0.644	-0.0355
0.685	-0.18103
0.726	-0.32657
0.768	-14.5252
0.809	-142.312
0.85	-1000

Without interpretation-by-simulation, the standard approach would take the production data and analyse these with JBN (see Chapter 9, Relative Permeability Methods). We followed this workflow and then found an apparent S_{or} of 0.23, with oil relative permeabilities being lower than the original (Table 10.9) by some three orders of magnitude (see Figure 10.11).

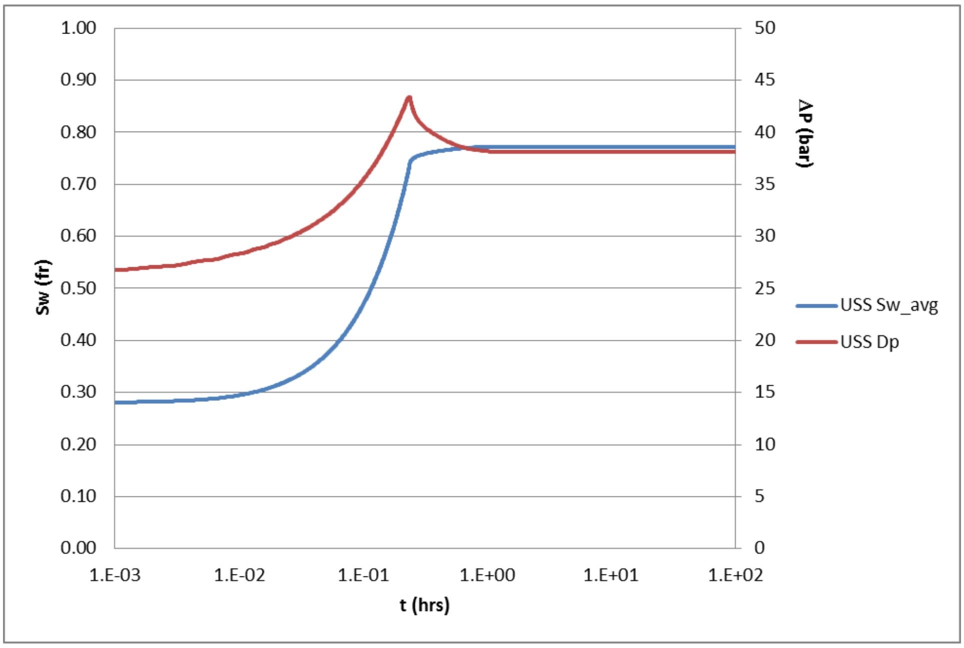

Figure 10.10: Simulated saturation development and pressure drop as a function of time, based on data in Table 10.8, Table 10.9 and Table 10.10

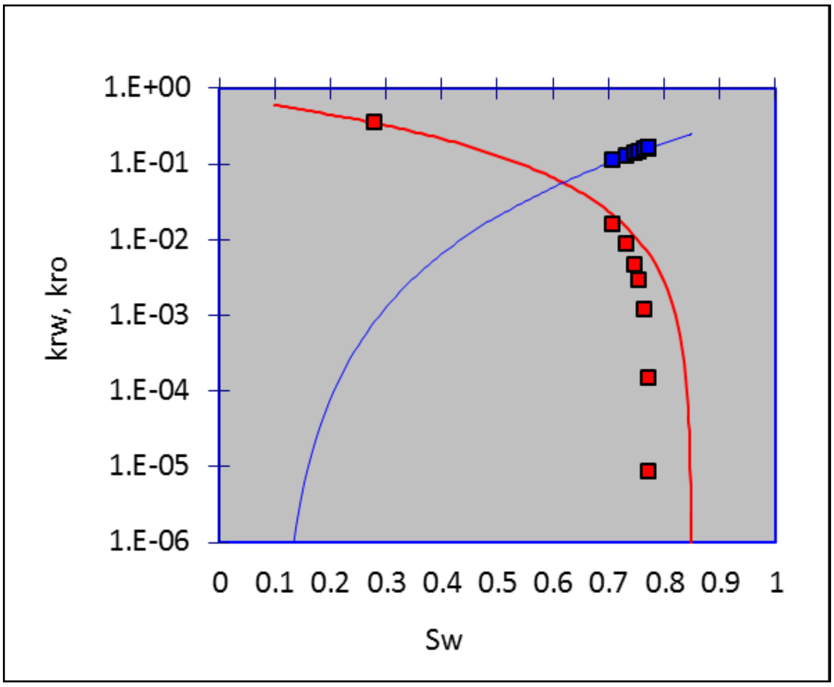

Figure 10.11: Comparison between original relative permeabilities (solid lines) and JBN derived data points for the case defined by Table 10.8, Table 10.9 and Table 10.10

Note that JBN assumes that capillary pressure is zero. When in actuality, as is the case here, the imbibition capillary pressure brings about an oil-end effect (typical for mixed-wet cases); the oil flow is inadvertently interpreted by JBN to be zero due to it being at residual oil. Information on imbibition capillary pressure must be obtained on a twin plug, as discussed above.

10.6. Reporting Requirements

Name and version of the simulation tools used must be documented.

All data input into a simulation study, be it for design or for interpretation must be reported.

Data interpretation should deliver not only the curves for relative permeability and capillary pressure, but must also include error margins. The range of possible curves can then be used by reservoir engineers to include a statistical distribution of these parameters in the field simulations.

10.7. Pitfalls

History matching of just a flow experiment or just a centrifuge experiment in practice is meaningless because relative permeabilities and capillary pressures are not well constrained in these instances: a combination approach of experiments (flow combined with centrifuge) has been proven necessary.

Saturation monitoring with X-ray dopants in one of the flowing phases is a questionable approach: dopants are known to be surface active and therefore may affect wettability.

10.8. References

CYDAR is developed by R. Lenormand, Cydarex. For information, see http://www.cydarex.fr/

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