# SCA2003-38: RESISTIVITY INDEX MEASUREMENT WITHOUT THE POROUS PLATE: A DESATURATION TECHNIQUE BASED ON EVAPORATION PRODUCES UNIFORM WATER SATURATION PROFILES AND MORE RELIABLE RESULTS FOR TIGHT NORTH SEA CHALK.

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## ABSTRACT

Measurement of Archie's Resistivity Index 'RI' and saturation exponent 'n' can be very time consuming for low permeable core samples using the established porous plate techniques. An evaporation technique circumventing the porous plate difficulties has been developed, and is demonstrated for the tight Lower Cretaceous carbonates in the Danish North Sea. Samples are saturated with a diluted formation brine and by evaporation water is removed from the diluted brine in the samples until the original brine concentration has been restored and the samples has attained a water saturation < 1. By this technique any water saturation down to approximately 0.1 can be imposed on the sample in less than a week. The movement of water in the sample during desaturation is documented by a tracer technique and core scanning, and data from the Lower Cretaceous carbonates are presented.

## INTRODUCTION

Archie's parameters (F, RI, m and n) are determined in the laboratory by analysing small core samples taken from reservoir core material. The parameters are used to predict porosity and water saturation from logged downhole formations, and it is therefore very important to determine these parameters correctly. However, current laboratory procedures often fail to produce accurate RI and n values for tight (low permeable) rocks. Archie's equations:

$$F = F^{-m} \qquad\qquad\qquad (1)$$
$$RI = S_w^{-n} \qquad\qquad\qquad (2)$$

presume that the material is isotropic and uniformly saturated with the conducting liquid. Lyle & Mills [1] have shown that measurement of RI and n will be in error if the core saturation is non-uniformly distributed. This holds true for F and m as well [2], and can be understood from the relations $S_w^{-n}$ vs. $S_w$ and $\Phi^{-m}$ vs. $\Phi$ being convex functions, Figure 1. This means that any non-uniform porosity or saturation distribution in the core sample will cause RI and F values that are higher than the figure measured for a uniform distribution, and the calculated m and n values will be overestimated as shown in the model calculations in Table 1.

**Chalk And RI Measurement**

In RI measurement a number of laboratory techniques are at hand to adjust a core sample to water saturations $S_w < 1$. The centrifuge is known to generate an uneven saturation profile in the sample [3] and is therefore not directly suited for resistivity measurements. Simple dynamic flooding down causes uneven saturation profiles as well due to BL shock front artefacts and end-effects in waterwet samples [4], and the resistivity plot shows a curved line. Examples can be found from the literature [5, fig. 5]. The preferred desaturation methods for RI measurement are drainage by constant injection rate or constant pressure applied to a sample confined in a resistivity core holder and equipped with a porous plate or membrane in the downstream end of the sample. With respect to uniform core saturation there is no significant difference between the two methods. For tight materials like chalk it is mandatory to use a 15 bar porous plate to obtain a fair range in water saturations. In terms of experimental time and drainage capacity it is the porous plate that is the limiting factor in most cases. Some examples will illustrate this. Figures 3 and 4 show constant pressure drainage through a 15 bar porous plate - the water is piled up in front of the porous plate and only very slowly drains away to reach equilibrium. The experimental time for Figure 3 was four weeks. The uneven saturation distribution produces a curve in the RI plot as predicted from theory [1, 4]. In Figure 4 after six months the system is slowly approaching equilibrium and thus a straight-line relationship emerges in the RI plot. An advanced steady state method utilizing saturation monitoring and multiple electrode recording of the sample interval resistivity to solve the inherent problems with the former methods was presented by Maas et al. [4]. This solution is convincing but requires expensive instrumentation, and establishment of a steady state condition for very low permeable materials may take a long time.

Lower Cretaceous Carbonates: The argillaceous to marly chalks of the Lower Cretaceous in the Danish Central Graben have a large areal extention and huge accumulations of oil. However, in the Valdemar field the original recovery is estimated at only 1%. This is due to the very poor reservoir quality. In the producing reservoir zones the insoluble residue amounts to 10-40 w% (kaolinite and $\alpha$-quartz), $\Phi$ is 25-45% and $K_g$ in the range 0.1-2 mD [6]. The matrix permeability is then 5-10 times lower than known for the Upper Cretaceous chalk reservoirs. Few SCAL studies have been performed to determine RI and n, and being short-term porous plate or simple gas blow down experiments they are subject to erroneously high RI and n values. Further, it is observed that draining on a porous plate is a slow process for the very low permeability carbonates and $S_w$ is only seldom taken below 60% [7]. In a RI plot the limited range in $S_w$ will greatly restrict the precision of the calculated saturation exponent n as seen from Figure 2.

It was clear that to obtain high quality RI and n data for the Lower Cretaceous chalks traditional techniques involving static or dynamic draining through a porous plate could not be used if data should be obtained in less than a year and with a fair range in $S_w$ values. Therefore an alternative desaturation technique had to be developed.

## ANALYTICAL

As far as it may be relevant laboratory procedures have followed the guidelines given by SCA [8]. However, the desaturation technique based on evaporation differs from earlier methods and is therefore described and verified below. Evaporation has been used earlier in core experiments. Land [9] used evaporation of oil to establish a range of oil saturations in gas-oil relative permeability experiments, and Viksund et al. [10] described an evaporation technique to establish ve ry low initial water saturations in low permeability materials which is essentially the same technique applied in this paper.

### Desaturation

The evaporation technique can be applied as a desaturation technique whereby nearly any water saturation can be imposed on a tight core sample without generating a directional flow inside the core sample, thereby overcoming problems with end-effects. . However, the resulting fluid distribution has not been documented and the technique not used in electrical measurements before as far as we know. Assume that we want to establish a certain water saturation $S_x < 1$:

$$S_x = 1/f , \qquad f > 1 \qquad\qquad (3)$$

The simulated formation brine of initial concentration $C_i$ is then diluted f times (volume to volume), and the cleaned and dried samples are completely saturated with this diluted brine of concentration $C_x$:

$$[C_x] = [C_i]/f \qquad\qquad (4)$$

By evacuation or desorption using silica gel or simple room condition evaporation water is removed from the diluted brine in the sample until the brine has been concentrated f times. By this exercise the brine has been restored to the original concentration $C_i$, and the water saturation in the sample has attained a new figure $S_x$ as given by eq. (3). By using a number of predetermined volume dilution factors a sensible range of water saturations may be imposed on the samples for RI testing. However, the sample cannot remain in the core holder during a complete cycle, it must be taken out and resaturated at each saturation step. Evaporation down to the precalculated water saturation and following equilibration to a homogeneous water distribution can normally be done in less than one week. The non-wetting phase is air.

This study was carried out with the samples in a cleaned water-wet state and a gas-brine – rock system to obtain the RI data. This is important because a homogeneous fluid distribution would take much longer time in an oil-water-rock system because of the low mobility of the liquid-liquid system. The difference between a gas-brine –rock vs. an oil-brine –rock system in terms of resistivity properties for water-wet rocks is known to be insignificant [11]. Also it should be observed that the majority of North Sea chalk fields including the Lower Cretaceous chalks are preferentially water-wet [12].

**Verification of the method**

The position of salt in two plugs was followed by spiking the brine with radioactive $^{22}$NaCl, which upon decay of $^{22}$Na emits gamma photons. With a suitable detector system (Figure 5) the movement of the salt could then be recorded. The plug was kept under vacuum in a container (Figure 6) and then saturated with a specially prepared de-aired brine with 0.60 w% salt - designed for reaching 3.5 w% brine concentration at $S_w$ = 17%. After vacuum saturation the plug and surplus spiked brine was transferred to a plastic bag that was sealed and placed in at high pressure container at 11 MPa for 48 hours. The now 100% saturated plug was measured in the scanner, Figure 5. The gamma-ray detector (NaI crystal) records the gamma activity of the plug through a 1-mm wide collimator whereby the activity within a 1-2 mm slice of the plug was measured. By moving the plug in front of the collimator the 1D distribution of salt along the plug axis was recorded. The first two measurements, 29 July and 15 August in Figure 9, showed a constant level along the plug i.e. it was verified that the plug was homogeneously saturated.

The saturation was next reduced to $S_w$ = 38% (and 1.6% salt) during a 19.5-hour period in the desiccator shown in Figure 8. The air in the evaporation chamber is kept dry by the silica gel. In this way the plug loses water by evaporation at the top end of the plug. The brine in the plug hereby is re-distributed i.e. some brine moves upwards replacing the lost water here. The concentration of salt in the upper part of the plug increases, and a diffusion of salt away from the top end begins. However, diffusion downwards is much slower than the upward movement of brine. Therefore, a skew distribution of salt is obtained in the plug. Figure 10 shows the distribution just after the evaporation and for the following five days. A strong signal peak is at first observed at the top end of the plug i.e. the amount of salt is increased here. Just after the evaporation the amount of water in the top end is slightly lower than at the bottom. The water soon reaches an equilibrium with a saturation close to $S_w$ = 38% all over the plug. A redistribution of the salt, however, takes several days. After 5-6 days the distribution of salt has reached a steady state with a distribution almost similar to that recorded for $S_w$ = 100%.

Additional reduction of $S_w$ from 38% to 17% (average and with 3.5% salt) was next done with the set-up shown in Figure 7. The plug was kept at vacuum for 90 minutes and water was lost through all parts of the surface. Hereby brine was transferred from the interior of the plug to the surface. The resulting distribution of the salt is shown in Figure 11. Minor signal peaks are seen at both ends of the plug, 1 September. When the detector "looks" at the ends of the plug it "sees" more plug surface than when "looking" only at the cylindrical part of the plug. The figure therefore verifies that some brine has been transferred from the interior of the plug to the surface. The non-uniform distribution disappears within 5 days and a distribution of salt equal to the previous distribution for $S_w$ = 100% is obtained. The experiments demonstrate that the dilution-evaporation method generates a uniform brine distribution 5-6 days after the evaporation down to $S_w$ = 17% or lower.

The experiments with the spiked brine included a number of other techniques to be tested. The saturation reduction to $S_w$ = 17% was followed by the addition of a portion of stronger

spiked brine (3.5 w% NaCl) to the plug top end hereby increasing the average $S_w$ to 25%. The results are shown in Figure 12. The lowermost curve describes the equilibrium state at $S_w = 17\%$. The added brine caused a strong peak at the top end. A transition towards a steady state was obtained within 9 days and a steady state finally reached within a month. The saturation distribution was almost identical to the original 100% saturated plug.

The experiment also gave additional information. Immediately after addition of extra brine to the top end the saturation increased all over the plug. Notice for example from Figure 12 that the first measurement (6 September) after addition shows a higher signal at the bottom end than before addition (5 September) although diffusion cannot have carried additional radioactivity down to the bottom that fast. Therefore the amount of "17% brine" immediately increases at the bottom when brine is added to the top end. However, the level here only increased to $S_w = 21\%$ to be compared to 25% for the plug in average. This observation was repeated in a number of similar experiments with the plug i.e. when brine was added to one end of the plug then $S_w$ at the opposite end immediately increased but only halfway towards the average $S_w$ of the plug. Obviously capillary forces and the high mobility of air help redistribute the brine in the plug; fast in the beginning and then slower depending on the capillary gradient. Diffusion of salt then continues until a steady state for both water and salt is obtained.

Other experiments, designed to test the mobility of the brine at low saturations, showed that after evaporation down to 13-14% the salt was still mobile and could reach the former distribution within 5-6 days. At $S_w \sim 9\%$ the salt was no longer movable indicating that there was no longer a continuous water phase. Resistivity measurements in the low saturation range (below 13-14% $S_w$) is then subject to uneven saturation distributions, and it is furthermore doubtful if the evaporation method can be used for obtaining very low water saturations as originally envisaged [10].

**Observations**

The evaporation method implies that water is removed from one end or the total surface of the plug, i.e. there is a transport of brine along the plug axis or from the plug interior in all directions towards the surface. By this process salt is concentrated close to the plug end or the surface, i.e. a chemical gradient is present that drives a diffusion process in the counter current direction to the brine movement. The capillary driving force will seek to establish brine equilibrium with a rate dependent on $vK/\Phi$ and a characteristic sample length [10]. Diffusion is described by Fick's law:

$$M = - D \times dC/dx \qquad (5)$$

It appears that the flux M is proportional to dC/dx. If a disequilibrium is imposed on the sample by evaporation of water, the time required to establish salt equilibrium by diffusion will be proportional to the square of the plug length L - or to another typical dimension of the plug. Assume two plugs with the same physical properties - $\Phi$, $S_w$ etc - but with different lengths. Further assume that similar inhomogeneities have been introduced in the plugs. Then the concentration gradient at similar positions in the plugs will be inverse

proportional to the length. Furthermore, the amount of salt having to pass any cross section before homogeneity is reached will be proportional to the length.

Next consider the influence of $\Phi$ and $S_w$. It is obvious to compare with the influence of these parameters on the resistivity of the plug. In both cases ions have to move in the brine. In one case an electric field is the driving force, in the other case a concentration gradient is the driving force. One therefore may assume that the time needed for obtaining homogeneity after an uneven distribution of salt along the plug axis will be proportional to $S_w^{(1-n)} \Phi^{(1-m)}$. In all therefore the diffusion time needed for obtaining homogeneity is proportional to $L^2 S_w^{(1-n)} \Phi^{(1-m)}$.

## RESULTS

The desaturation method will be demonstrated for the low permeability Lower Cretaceous chalk and some aspects of the precision of the method will be discussed.

### Samples

The Lower Cretaceous chalk is loosely consolidated, very fragile and fatal fracturing is a frequent problem. Due to the difficulty of saturating the tight samples and to help chemical equilibration by diffusion the 38 mm diameter samples were cut rather short, 20-25 mm being the typical sample length. This allowed a second sample be taken from the same original plug if the first one failed, but more important, twin plugs allow a direct comparison of the "electrical homogeneity" of the sample material and precision check on the saturation method, on the condition that there is a reasonable agreement between the porosity of the two halves. Conventional porosity is 31-43% for the analysed plug samples.

### Preparation

The cleaned and dried samples were vacuum and pressure saturated. Final saturation was then = 97%; complete saturation could not always be obtained within 1-2 weeks for the tight samples. Samples to be de-saturated to a certain $S_w$ were then left to evaporate until a precalculated weight had been obtained as explained earlier. Due to the extensive sample handling and the fragile samples only a sparse number of saturation steps were measured. After each saturation step the samples were not cleaned but dried at 60 °C and re-saturated with a diluted formation water as described above. The samples were left to equilibrate in the diluted water for 2-3 weeks, and the water was changed 2-3 times during this period until a stable water resistivity reading equal to the stock diluted water resistivity was obtained. Chemical equilibration by diffusion was shown in the tracer studies to be a viable procedure, Figure 12.

### Instrumental

The electrical resistivity cell is a hydrostatic core holder with gold plated electrodes, one electrode being the floating end piece that is electrically isolated from the core holder assembly. The measurement principle is known as the two-electrode method.

The resistivity cell is connected to a HP 4276A LCZ-meter capable of measuring the electrical impedance at a range of frequencies from 0.1 to 20 kHz. A sweep confirms that the system is sensitive to the frequency. Measurement frequency was then set at 10 kHz were phase shift is generally below one degree when measuring $R_0$. To protect the fragile samples from fracturing, the hydrostatic confining sleeve pressure was set at a low value of 2 MPa. Data presented in figures and tables are overburden corrected. All measurements were carried out in a temperature-controlled laboratory at $25 \pm 0.5$ °C.

### RI Data

Less than half of the original samples survived measurement of 3 saturation points. Data represent two different reservoir units with 5 and 6 samples respectively, and because a limited spread was observed within each geological unit a pooled n value has been calculated for each formation, Table 2. Considering the number of samples in each formation no statistical significant difference was observed between the n-values, and a grand average n has been calculated for all analysed Lower Cretaceous chalk samples. The total set of samples measured are shown in Figure 13 with an average n=1.73±0.09; a significantly lower value than the n=2.1-2.2 hitherto used in wireline log interpretation [6]

### Error Analysis

A study of the precision (= reproducibility) of the method applied to the single samples have not been performed due to the fragile nature of the material. However, a more demanding precision test, taking the variation in sample material within a cm scale into consideration, can be carried out on the duplicate sample sets (A and B plugs). Three such plug sets having approx. the same porosity have been measured in most desaturation steps, plug no.: 11A/11B, 13A/13B and 20A/20B, Table 2.

The measurements show a coefficient of variation (= relative standard deviation) of CV = 10% on the single saturation step determination. However, if N samples are selected from the same lithological unit the CV will be √2N times better than for the single sample at each water saturation step. If N=5 then CV~3% for each saturation step. If several saturation steps are measured, the regression line from which the saturation exponent 'n' is estimated will be even better determined.

Pooling the data for the two geological formations in table 2 returns a CV~5% for the slope of the regression line (= n) in Figure 13, which is higher than expected from theoretical grounds. This is attributed to geological (sample) scatter and probably higher experimental error for the fragile samples than believed.


### CONCLUSIONS

- An evaporation method, circumventing the porous plate in electrical measurements, has been devised specially for tight reservoir rocks. The dilution-evaporation method generates uniform water saturation profiles along the core, which is a primary requirement for reliable electrical measurements. Cleaning between the

saturation steps is not necessary, chemical equilibration with a new brine formulation is obtained in less than 2-3 weeks by diffusion.

- The method does not depend on a slow drainage process; therefore it is possible to cover a wide range in water saturation meaning a more precise determination of the saturation exponent n. The method does depend on capillary redistribution of water and diffusion of salt, and it is therefore recommended to check representative samples from the actual reservoir unit in a core scanner to make sure that the dilution-evaporation method works as conceived.

- Perfect homogeneity does not occur in natural rocks, therefore it is an advantage to measure a large number of samples to minimize random scatter in electrical measurements. The evaporation method can run a large number of samples at the same time. For a tight formation each saturation step can be measured in 3-4 weeks.

- A disadvantage is that the method requires extensive sample handling and that the sample cannot remain in the resistivity cell during a complete desaturation cycle.

**Acknowledgements**

**NOMENCLATURE**

| | | | |
|---|---|---|---|
| D | = diffusion coefficient [m2*s$^{-1}$] | Subscripts: | |
| C | = concentration [mg/L] | g | = gas |
| CV | = coefficient of variation [%] | i | = initial |
| f | = dilution factor | x | = precalc. saturation |
| F | = formation resistivity factor | w | = water |
| $\Phi$ | = porosity [fraction or %] | k | = sample interval |
| K | = specific permeability [mD] | 0 | = initial |
| L | = length [m] | | |
| M | = mass flux [mol*m$^{-2}$*s$^{-1}$] | | |
| m | = Archie's cementation exponent | | |
| n | = Archie's saturation exponent | | |
| N | = number of samples | | |
| R | = resistivity [$\Omega$] | | |
| RI | = resistivity index | | |
| S | = saturation [fraction or %] | | |
| SCAL | = special core analysis | | |
| w% | = weight percent | | |

Table 1. The saturation and resistivity measured in the laboratory is the sample bulk $S_w$ and RI; the various subinterval $S_{wk}$ and $RI_k$ values are generally unknown. The deviation in RI from the uniform minimum value can be considerable for uneven saturation distributions as shown in the table. The examples assumes the intrinsic sample n=2 for all intervals.

| Subinterval | $S_{wk}$ distribution | | |
|---|---|---|---|
| | ex. 1 | ex. 2 | ex. 3 |
| $k_1$ | 0.25 | 0.19 | 0.10 |
| $k_2$ | 0.25 | 0.21 | 0.15 |
| $k_3$ | 0.25 | 0.23 | 0.20 |
| $k_4$ | 0.25 | 0.25 | 0.30 |
| $k_5$ | 0.25 | 0.37 | 0.50 |
| RI | 16.0 | 18.5 | 36.9 |
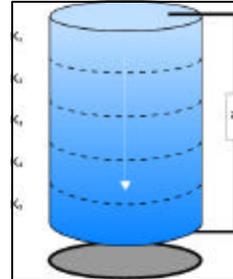
Table 2. Saturation exponent data calculated from regression analysis on 3 saturation steps. Samples were taken from two different reservoir units in the Valdemar field.

| Plug no | $\Phi_{300}$ | n | Unit | Unit n | Plug no | $\Phi_{300}$ | n | Unit | Unit n |
|---|---|---|---|---|---|---|---|---|---|
| 11A | 0.422 | 1.83 | | | 20A | 0.328 | 1.52 | | |
| 11B | 0.429 | 1.75 | | | 20B | 0.316 | 1.83 | | |
| 12 | 0.423 | 1.72 | Upper | 1.76 | 22 | 0.357 | 1.82 | Upper | 1.71 |
| 13A | 0.398 | 1.72 | Sola | ±0.05 | 23 | 0.333 | 1.72 | Tuxen | ±0.12 |
| 13B | 0.407 | 1.77 | Fm. | | 24 | 0.376 | 1.65 | Fm | |
| | | | | | 27 | 0.324 | 1.68 | | |
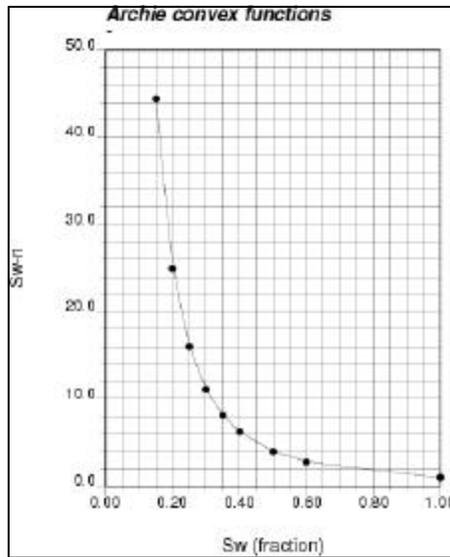


Figure 1. $S_w^{-n}$ vs. $S_w$ is a convex function. The curve is shown for n=2. Partly after Lyle & Mills [1].
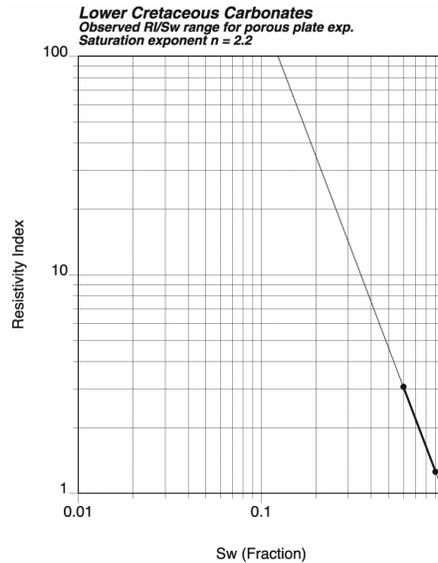


Figure 2. Previous RI measurements of Lower Cretaceous chalk falls within a narrow saturation range.
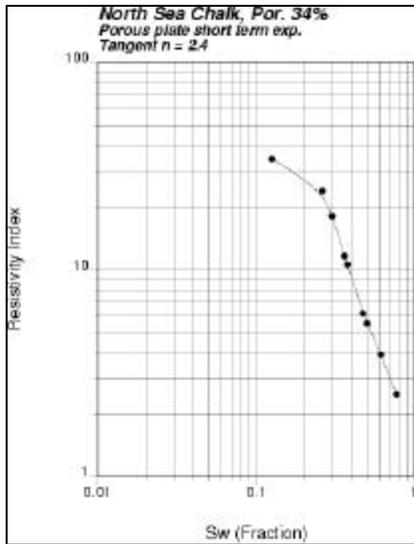
Figure 3. Tor Fm. chalk, constant
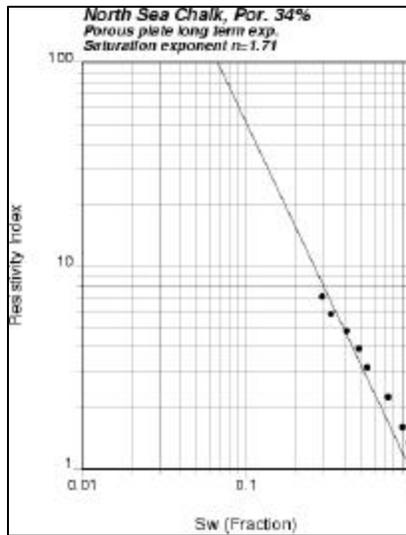pressure drainage; 15 bar plate.
Experimental time four weeks.



Figure 4. Ekofisk Fm. chalk, constant
pressure drainage; 15 bar plate.
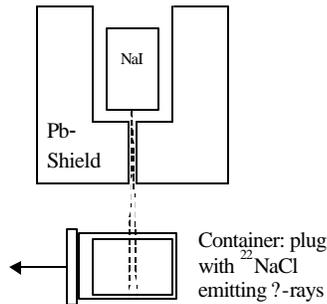Experimental time six months.



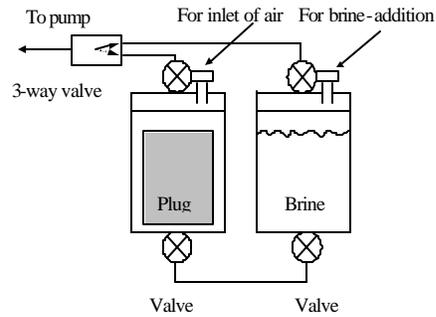Figure 5. Scanning of plug with
$^{22}$Na-spiked brine.
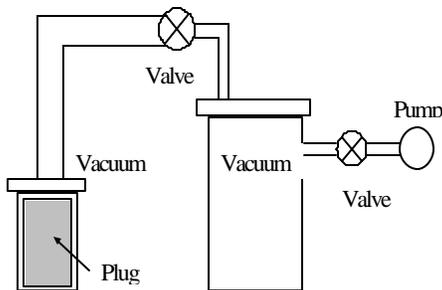


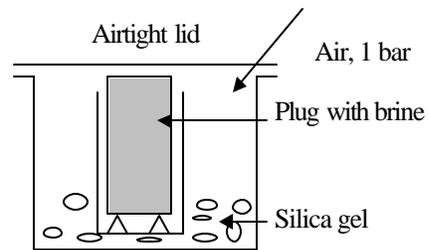Figure 6. Vacuum saturation set-up.



Figure 7. Vacuum evaporation.



Figure 8. Evaporation with silica gel.
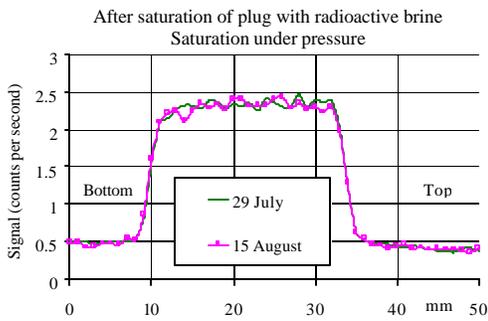
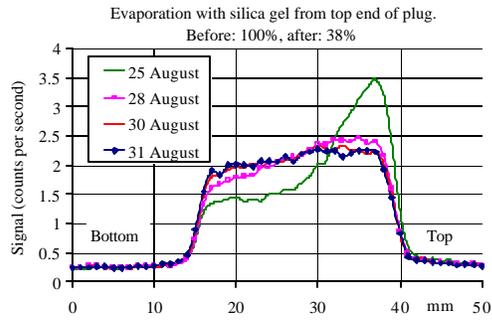Figure 9. Concentration profiles in plug after pressurised saturation.



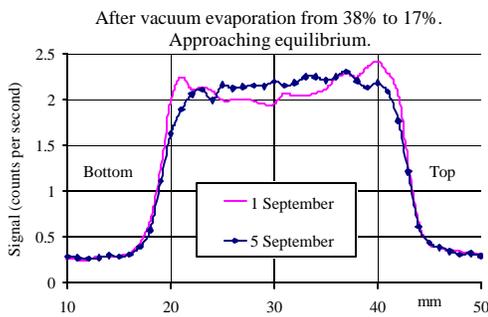Figure 10. Evaporation with silica gel, from $S_w = 100\%$ to $S_w = 38\%$.



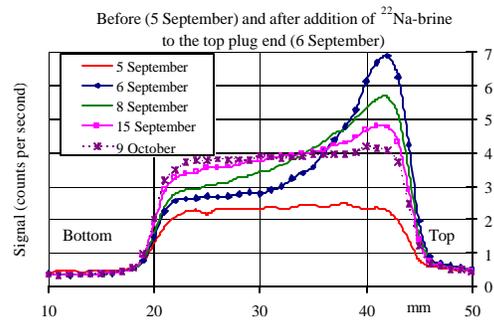Figure 11. Vacuum evaporation from $S_w = 38\%$ to $S_w = 17\%$.



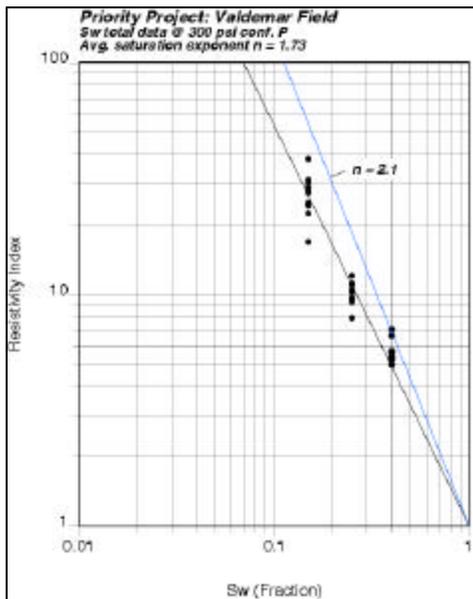Figure 12. Addition of brine increasing saturation from $S_w = 17\%$ to $S_w = 25\%$



Figure 13. RI plot of the total set of samples (N=11) where measurement of 3 saturation steps could be performed without fatal fracturing. The former used n=2.1 reference line has been added for comparison

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