RELATIVE PERMEABILITY EFFECTS OVERLOOKED IN MICP MEASUREMENTS TRANSITION ZONES LIKELY TO BE SMALLER

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ABSTRACT
Mercury injection capillary pressure (MICP) measurements are frequently considered as part of routine core analysis (RCA) programmes, and a fair number of MICP samples are then taken from each core brought to surface. MICP data in combination with log data are used in building saturation-height functions that in turn are necessary to calculate STOIIP and initialise reservoir simulation models.

From a physics point of view, MICP has a resemblance to porous plate (PP) measurements: at a preselected number of pressure steps the amount of injected fluid is measured after equilibration, that is, when fluid movement has come to a stand-still. It is well known that PP experiments, just like other special core analysis (SCAL) measurement methods are subject to interference between capillary forces and viscous forces due to relative permeability. For that reason, state-of-the-art SCAL data are extracted from experiments through interpretation-by-simulation. In SCAL relative permeability experiments, the impact of such interpretation is often significant (*e.g.* residual oil saturations may reduce by 10 to 15 units).

Some 40 samples have been investigated by MICP, under various measurement protocols, to study a possible saturation shift due to an interference with relative permeability similar to what is observed in other SCAL experiments. The measurements were designed using an adapted version of the license-free SCAL simulator SCORES. In line with the simulations, the experiments show that mainly recordings of the saturations of the plateau of the capillary pressure curve do not reach equilibrium conditions in conventional MICP measurements. The plateau values should be shifted to lower wetting phase saturations by 10 to 15 units. In hydrocarbon reservoirs with an extensive transition zone, the transition zone may be significantly reduced and a sizeable effect on STOIIP and therefore on reserves can be expected.

The paper presents detailed information on an improved MICP measurement protocol.

INTRODUCTION
For many years, the industry has been aware of the mutual interference of relative permeability and capillary pressure in flow experiments. Several authors [1, 2] showed

the shortcomings of analytical data interpretation and Kokkedee [3] even suggested ways to exploit this interference. He suggested that relative permeability and capillary pressure could be extracted simultaneously by history matching his so-called CAPRICI experiment. The effect of this interpretation-by-simulation approach is often large. Notably, the residual oil saturation probed in imbibition flow experiments shifts to lower values by 10 to 15 saturation units [4]. The impact of interpretation-by-simulation on capillary pressure measurements was demonstrated more recently by Maas *et al.* [5].

Mercury injection capillary pressure (MICP) measurements as part of routine core analysis (RCA) measurements are the workhorse for assessing the saturation-height distribution in a hydrocarbon bearing reservoir. The experimental setup goes back to Washburn in 1921 [6]. The operator would increase the injection pressure in steps and monitor mercury intrusion. Routinely, 10 to 15 minutes equilibration time would be used at each pressure step. Around 1984 [7], automated equipment became available to measure capillary pressure with mercury injection on small core plugs and off-trims. Since then, measurement protocols *de-facto* became standardised by the default settings in the software provided with the equipment. In particular, the equilibration time is left at its default of usually 10s. The equilibration time may be increased only for low permeable (cap) rock studies. Not much information is available on such an adjusted measurement protocol, because standard laboratory MICP reports generally do not discuss the experimental settings. This leaves users of MICP data completely unaware of even the options that exist. MICP software may offer among other things rate-controlled injection and fixed equilibration times. The impact of the various options on pore size distribution and pore volume is discussed in a Micromeritics Application Note [8], but the effect on the resulting capillary pressure curve so far is unknown.

In fact, equipment such as the Autopore was developed in support of the pharmaceutical and chemical industry to measure porosity and pore size distribution on catalysts and powders. The petroleum industry, on the other hand, uses the automated equipment to measure (drainage) capillary pressure and saw automation as a way to obtain the data fast, that is, cheaply. Service providers are able to conduct two runs of two samples each per day at the default setting of 10s equilibration time.

The experience gained over the last years with the important effect of the interference between relative permeability and capillary pressure in laboratory samples has triggered our research into the impact of relative permeability in MICP measurements. Purcell [9] linked the capillary pressure curve to absolute permeability. Li and Horner [10] review various methods to extend Purcells approach towards the determination of relative permeability. However, we have not found a discussion on the possible impact of relative permeability on capillary pressure measurements in MICP.

The project was designed as follows:

- Conduct scouting simulations of synthetic experiments to test whether an effect of relative permeability could exist in standard MICP experiments

- Conduct scouting experiments measuring intrusion and pressure development during MICP experiments as a function of time to study whether/when intrusion would stabilise under a standard measurement protocol
- Assess the impact of varying equilibration times on the resulting capillary pressure on selected samples
- Develop an improved measurement protocol, balancing required accuracy in the determination of capillary pressure and the measurement time involved.

The paper is structured similarly: first the simulation work is presented, followed by a discussion of the scouting experiments. Subsequently, experiments with different rock types are discussed. The paper finishes with Conclusions and Recommendations.

SCOUTING SIMULATIONS

As a first step, the SCAL simulator SCORES [5] was extended to simulate injection of a liquid from two sides simultaneously into a one-dimensional core plug that contains an (ideal) gas. Injection pressure is input as a table that lists the pressures required at certain times, similar to the pressure table available for the simulation of a Porous Plate experiment with SCORES. The initial (absolute) pressure P_i and initial gas density is set under user control and is used by SCORES to calculate the gas density when pressure is increasing during the synthetic experiment. Gas viscosity, liquid density and viscosity, and temperature are kept constant in the simulations. Usually, samples are evacuated at the start of an MICP measurement to some finite residual pressure; therefore P_i can be set to any realistic sub-atmospheric pressure. Mercury viscosity was set to 1.5 cP [11] and gas viscosity to 0.01cP.

Because mercury is strongly non-wetting on most rock surfaces, simulations were run with synthetic relative permeabilities for such an extreme non-wetting case, with Corey parameters as listed in Table 1. Porosity was set to 0.25 and permeability was varied between 0.03 mD and 300 mD. SCORES reports the average wetting phase (*i.e.* gas or vacuum) saturation as a function of time, together with the applied injection pressure. From that, one can construct the capillary pressure, similarly to what the software does for an automated MICP apparatus.

Figure 1 shows the comparison between the inputted capillary pressure curve and the apparent capillary pressure as derived from the SCORES output. Several scenarios are shown for a fixed absolute permeability of 3 mD and fixed sample diameter of 1 inch: 1) L set to 0.5 cm, using an equilibration time of 10s at each pressure step; 2) L set to 0.5 cm, now with an equilibration time of 1hr; 3) a sample length L of 5 cm, also with an equilibration time of 1hr. Clearly, for all three cases a large deviation exists at high wetting saturation (*i.e.* low mercury saturation). This effect is due to the low relative permeability of the mercury at the start of the experiment. The run with an equilibration time of 10s deviates the most from the true Pc curve, up to at least S_{gas}=0.3. An equilibration time of 1hr brings the apparent Pc curve close to the true curve for gas saturations of 0.94 and lower. Still at higher gas saturations, that is, lower mercury saturation, the deviation is significant. A larger plug, even with an equilibration time of 1hr, would strongly deviate from the true curve for gas saturations higher than 0.5. So, smaller samples will generate more realistic Pc curves.

Another observation is that the results are not very sensitive to the relative permeability of the gas or vacuum. The compressibility of the gas “makes up” for a decreasing relative permeability at lower wetting phase saturation. We did note that if the initial pressure is not well below 0.1 bar, the compressed gas at maximum mercury injection pressure (60000 psia, being the industry standard) may reduce the effective pore volume as measured by MICP[1].

We found that even at equilibration times of many months, the very early part of the capillary pressure curve always deviates significantly from the true curve. This effect is stronger for lower permeability rock. Consequently, it may well be impossible to measure with reasonable accuracy the entry pressure of cap rocks and shales.

At high mercury saturation, SCORES shows that intrusion stabilises faster and ultimately may well stabilise within 10s. The saturation at which that occurs depends on the exact shape of the mercury relative permeability and on the absolute permeability.

It must be stressed that the reported simulations refer to a synthetic one-dimensional case, so only general conclusions can be drawn.

SCOUTING EXPERIMENTS

In order to test the conclusions drawn from the SCORES simulations of MICP experiments, it was decided to extend the electronics of the Autopore apparatus at PanTerra and measure both the time series of intrusion and of pressure directly; these measurements were otherwise not user-accessible through the Micromeritics software [12]. Figure 2 shows the typical behaviour of pressure and intrusion during a measurement on a test sample, as recorded through our electronics. In this case, the Autopore was run with a 10s equilibration time. The way the apparatus is programmed is that for each pressure specified by the operator in a table, the software assesses whether the actual pressure has actually reached the set value and then ensures that this pressure remains constant for the chosen equilibration time. The intrusion, as measured at the end of the equilibration time, is then recorded in the output file, together with the value of the pressure, and the software moves the apparatus to the next pressure level. In this “equilibration time” mode, the software does not verify whether the intrusion has reached a stable level, as is clearly demonstrated in Fig. 2.

The software does provide an option to equilibrate on intrusion, but the lowest limit that can be programmed is 0.001 μl/s per gram of sample, that is, the intrusion is declared

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[1] The pore volume as determined by MICP, by definition corresponds to the maximum amount of intrusion seen at maximum applied pressure, usually 60 000 psia.

stable if the actual intrusion does not change by more than 0.001 μl/s per gram of sample over the chosen equilibration time. Assuming a plug of 1 inch diameter by 1 inch length, with a porosity of 0.25 and a density of 2.65 g/cm^3, this translates into more than 2cm^3/day, while the pore volume would be around 3 cm^3. So, this lowest limit would not be very practical to use in capillary pressure measurements, while settings of 10^{-4} to 10^{-5} μl/s per gram may well be technically impossible in view of noise levels in the data.

In line with the SCORES simulations, the time series proved that with increasing mercury saturation, the intrusion stabilised faster. We conclude that there is scope for improved measurement protocols, possibly necessitating an upgrade in the software.

SAMPLE SELECTION AND REPRODUCIBILITY EXPERIMENTS

From the scouting experiments discussed above, it is clear that the mercury relative permeability may lengthen significantly the equilibration time required for a stable intrusion reading at a given pressure. The next step in the project was to quantify the effect on a number of samples. Ideally, one would want to conduct an MICP measurement using the default equilibration time of 10s, and then to repeat the experiment on the same plug using a longer equilibration time and compare results. However, once that a sample has been used, it is spent - it is practically impossible to remove the mercury from the pores. So, special attention needed to be paid in obtaining a set of samples that were as similar to one another as possible.

Statistics

From the scouting simulations and time series presented above it is fair to expect a difference in Pc curves dependent on the equilibration time for the MICP experiments. The objective of a statistical analysis is then to test for a possible significant difference and to what extent (range in P_c and S_{Hg}) such difference persists.

As the number of repeat measurements is limited, the t-distribution shall be used to test the difference between 2 independent means calculated at a number of capillary pressures above the entry pressure. A mean Hg-saturation and variance is calculated at each pressure step for the two sample series of MICP measurements conducted at 10s and 1000s equilibration time respectively. The assumption of equal variances for the two sample series is tested by an F-test at the 5% level. A null-hypothesis of no difference between the sample means is tested at the accepted 5% and 1% significance probabilities (2-sided test) for the t-distribution at a given degree of freedom (*i.e.* we can be more than 95 or 99% sure the two series are different if the calculated t-value exceeds the 5% or 1% test points of the t-distribution). Results of the statistical analysis are discussed below and given in table 2.

Sandstone samples for reproducibility experiments

Oberkirchener (OBKN) sandstone outcrop material was selected, because of it being readily available and because it was found to be relatively homogeneous in a recent study

[13]. Thirty-two samples were drilled and the poro-perm data were statistically analysed: samples were rejected when either porosity or (Klinkenberg corrected) permeability were found outside 1 standard deviation of the sample set mean value of around 8 mD. The remaining 16 samples were then CT-scanned with 12 CT-frames per plug, each frame being individually statistically analysed.

The final selection was based on the homogeneity number V as defined in [13], with a first cut-off at 0.25. Later the sample set was extended by allowing V to increase to 0.27. In this way, we collected 8 samples of 1 inch diameter and 1 inch length that each were cut into two samples of 0.5 inch length. Poro-perm was again conducted on the smaller samples. Several samples were used for preliminary tests, scouting for the best practical protocol. The repeatability of the MICP data across the samples was excellent, once that we applied as a final correction a scaling of the measured capillary pressure curves with $\sqrt{(\varphi/K)}$, with φ being the porosity and K the permeability. This is demonstrated in Fig. 3 that shows the capillary pressure curves of four samples, all run at 10s equilibration time (solid lines). The dashes lines in Fig. 3 are the capillary pressure curves measured on four other samples with a special protocol: around an intrusion of 0.05 PV, the software running in automatic mode with a 10s equilibration time was put in “suspend mode” (option available in the control screen). In suspend mode, the software continues to control the pressure and maintains it at the user-requested value. Meanwhile, the intrusion was monitored and recorded by hand at 10s, 100s and 1000s after the set pressure had been reached. Once that the 1000s limit was reached, the software was switched back (“resume mode”) into operation, to move the apparatus to the next pressure level specified by the user. About 20 pressure levels were measured in this way, until the shift reduced to less than 0.02 PV or so. In order to have a good coverage of the zone sensitive to the length of the equilibration time, the pressure table had been densely populated over the whole drainage trajectory (a factor of two more points than the default drainage table). The suspend mode applied to a limited number of points allowed us to keep the run time of the experiments below 8hrs; the software when set to 1000s equilibration time flat, would cause the run time to exceed possibly even two days. The software does not allow different equilibration times at different pressure levels.

The reproducibility of the suspend mode data is less than observed with the default protocol and default equilibration time of 10s, but it still allows access to the saturation shift with a high degree of certainty. This was substantiated by an in-depth statistical analysis of the data as discussed below.

Statistical analysis of the reproducibility of OBKN Pc data

The statistical analysis, as outlined above, included 11 Pc test levels between 28 to 188 psia and was applied to the two series of four samples each. The two sample series were found to differ significantly at the 5% level (see Table 2), and even at the 1% level in the Pc range of 34 to119 psia. The long equilibration time for the OBKN sandstone displaced the MICP curve towards lower capillary pressures relative to the short equilibration time runs, with shifts up to 20 saturation units, and affected most of the transition zone (see Table 2 and Fig. 3).

Micromeritics reference material for reproducibility experiments

In order to verify the impact of equilibration time on low permeable samples, we have run samples of so-called Micromeritics Reference Material, consisting of Silica-Alumina pellets, and characterised by a narrow pore size distribution with median pore diameter of 0.0074 µm, $\varphi \sim 0.62$ and K in the order of 1 nD. Fig. 4 shows the measured capillary pressure curves on 6 Reference Material samples, using 10s equilibration time throughout (solid lines). The reproducibility is excellent, as a reflection of the material marketed as reference material. Four samples were run with a protocol similar to mentioned above for the OBKN samples, using suspend mode and recording data at 10s, 100s and 1000s. As with the OBKN results, the reproducibility of the suspend mode data is somewhat less than of the base case runs at 10s, but a saturation shift is clearly visible of some 20 units at 23 000 psia. A similar statistical analysis was carried out as for the OBKN samples, to quantitatively assess the reproducibility.

It is interesting to note that the saturation shift at higher equilibrations times also brought about a shift towards larger values of the median pore diameter by 4 to 5%.

Statistical analysis of the reproducibility of Reference material Pc data

Reference samples 1 & 2 in Fig. 4, are shown for comparison but not included with the analysis because they were measured using another pressure table. The statistical analysis included 8 Pc test levels from 19000 to 27000 psia. The two sample series were found to differ significantly at the 5% level (see Table 2) and even at the 1% level in the Pc range of 19000 to 25400 psia.

EXPERIMENTS ON VARIOUS ROCK TYPES

Additional measurements were conducted to investigate whether other rock types would be sensitive to the setting of the equilibration time. Samples of limestone outcrop, North Sea chalk reservoir rock, and Rotliegendes sandstone reservoir rock were studied. To mimic actual practice, none of the samples were pre-screened by CT. For some samples, permeability data were not available. In each case, except where indicated differently, 4 samples were run under the default protocol with 10s equilibration time, and 4 samples under the suspend mode protocol, with data collected at 10s, 100s and 1000s.

Limestone outcrop

Euville outcrop samples were used with porosity ranging from 0.21 to 0.23. Permeability (Klinkenberg corrected) was between 210 and 750 mD. Samples of 200 to 300 mD showed a nominal effect, samples of higher permeability did not show a measurable effect at all.

North Sea chalk reservoir rock

Porosity ranged between 0.30 and 0.38. The samples were too friable for permeability measurements. Neighbouring samples of larger length had permeabilities in the order of 1 to 4 mD. Fig. 5 shows the measured capillary pressure curves. The spread in the curves prohibits an assessment of the effect of longer equilibration times. This was to be expected, because no pre-selection of any kind had been done on these samples. Still, on individual samples the effect of a longer equilibration time is very clear, as shown in Fig. 6. Here, the saturation shift is plotted at each pressure step as monitored between 10s and 1000s equilibration. The noise level was in the order of only 0.002 to 0.003. So, the observed shifts of 5 up to 12 saturation units are a factor of 25 or more above the noise level and therefore significant.

Note that the shift plotted in Fig. 6 should not be confused with the shift between the curves as shown in Figs. 3 and 4. The shifts observed in Fig. 6 can be nicknamed “differential shifts”, because the shift observed after 10s at a certain pressure level “builds” on the 1000s shift observed on a previous pressure step. The net total shift of the final curve, that cannot be determined directly in the absence of a reliable base case at 10s, is some kind of convolution of individually observed differential shifts. We have compared the plot in Fig.6 with similar plots as obtained for the OBKN and for the reference samples. Both showed a similar shape as seen in Fig. 6 and a maximum at a similar height of around 10 saturation units. From that we conclude that for the chalk samples, a net shift must have occurred of similar size as seen in Fig. 3 and 4, that is, a shift of some 20 saturation units.

Rotliegendes sandstone reservoir rock

Samples from two different reservoirs have been tested. For reservoir I, porosity ranged between 0.02 and 0.09, and (Klinkenberg corrected) permeability ranged between 0.03 and 0.23 mD. The spread of the capillary pressure curves both of the base case runs and of the runs with the suspend protocol was equally large and no distinction could be seen between base case and suspend protocol runs. Note the samples had not been screened with CT. In addition, a differential shift, as defined above, was hardly observable above the signal noise. This is probably due to the low pore volume in combination with the low permeability and the absence of a clear plateau in capillary pressure.

For reservoir II, the porosity was around 0.15 and permeability was 100 mD to 200 mD. In this case, only one sample was available for this study. The differential shift in this case was observed above signal noise, but was still low at about 0.15 saturation unit.

CONCLUSIONS AND RECOMMENDATIONS

- SCORES proved a good tool to scout for the interference by relative permeability in MICP measurements.
- The sample selection procedure based on the heterogeneity number V as outlined in [12] proved to work very well and delivered a sample set that could truly be

used as a standard, similar in quality as the reference samples provided by Micromeritics.

- A detailed statistical analysis proved essential in analysing the data of OBKN samples and of Micromeritics reference material in this study.
- MICP measurements have been proven to be dependent on relative permeability, similar to SCAL techniques such as the Centrifuge and Porous Plate.
- The interference of relative permeability in MICP may move the capillary pressure curve by 20 saturation units, particularly for saturations in the transition zone. Therefore, for low permeable transition zone reservoirs the reserves may be significantly higher than currently estimated.
- Interpretation-by-simulation of MICP experiments requires control software to record and output the time series of pressure and intrusion, just like is available in SCAL experiments.
- Due to the interference with relative permeability, the entry pressure in low permeability rock may well be lower than currently estimated. Consequently, the hydrocarbon-water contact may well be lower than currently estimated.
- Capillary pressure measurements in low permeable samples such as for gas and oil shales need to be carefully designed and executed. It is recommended to use a simulator for experimental design.
- The interference of relative permeability in MICP was observed particularly in rock of permeability below 10 mD, but cannot be excluded in rock of higher permeability.
- The pore size distribution is dependent on the equilibration time.
- Presently, software in the Autopore is not suited for automatically optimising equilibration times. Ideally, equilibration times need to be adapted to intrusion rate, at a value much lower than 0.001µl/s per gram of sample as is presently available.
- The suspend mode protocol as outlined in this paper needed to be applied for mercury saturations between 0.1 and 0.8 PV.
- The 1000s maximum equilibration time used in this study cannot be seen as the optimal maximum equilibration time. This value was chosen for practical reasons. Consequently, the presented capillary pressure curves for OBKN are probably still not the “true” capillary pressure curves.
- For improved transition zone definition, and in the absence of improved software, it is recommended to use the following protocol: 1) on a few plugs, run standard protocol to obtain a rough indication of where the transition zone is 2) on remainder of plugs, between 0.1 and 0.8 of the estimated pore volume, switch to suspend mode and monitor intrusion over time until it is stable within the noise level over a period of at least 3hrs. Ensure populating the transition zone with at least three pressure points.
- It is recommended that service laboratories report the option settings used in MICP for each study.
- The equilibration time is dependent on the size of the sample, as proven in 1-D SCORES simulations

- The MICP option in SCORES is now available at jgmaas.com/scores

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	S_{wc}	S_{nw_res}	n	k_{r_end}
Gas (wetting phase)	.0	.02	5	.98
Mercury (non-wetting)	.0	.02	3	.75

Table 1. Corey parameters for MICP simulations.

Statistic	**Reference material**	**OBKN sandstone**
N_1 (10s)	4	4
N_2 (1000s)	4	4
t_{95}	2.45	2.45
t_{99}	3.71	3.71
t_{calc} (range)	3.5 - 17.6	2.6 - 10.4
significant interval, P_c (psia)	19000 - 26300	30 - 169
significant interval, S_{Hg} (frc)	~ 0.08 – 0.8	~ 0.1 - 0.8

Table 2. Statistical data and MICP results for Micromeritics reference material and Oberkirchener sandstone. Number of samples N tested at each equilibrium time and the respective critical points for the t-probability distribution at degrees of freedom df = 6 [14]. t_{calc} is the calculated t-value for a range of capillary pressures P_c along the transition zone. The last 2 rows in the table list the interval with a significant difference at the 5% test level for P_c and S_{Hg} measured at 10s and 1000s respectively.

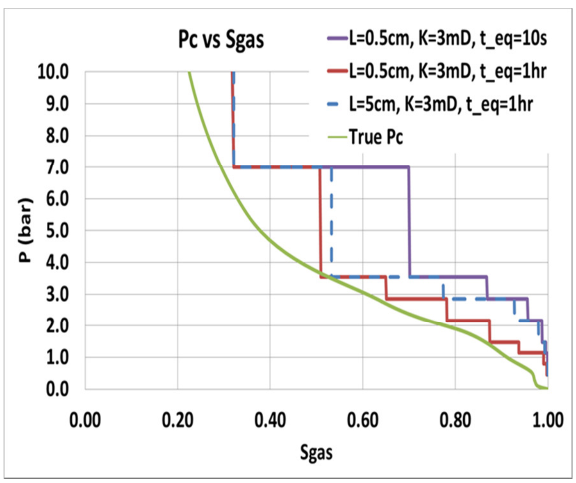

Figure 1. Simulations for synthetic 1-D MICP; comparison between inputted Pc and simulated apparent capillary pressure curve, effect of equilibration time and sample size.

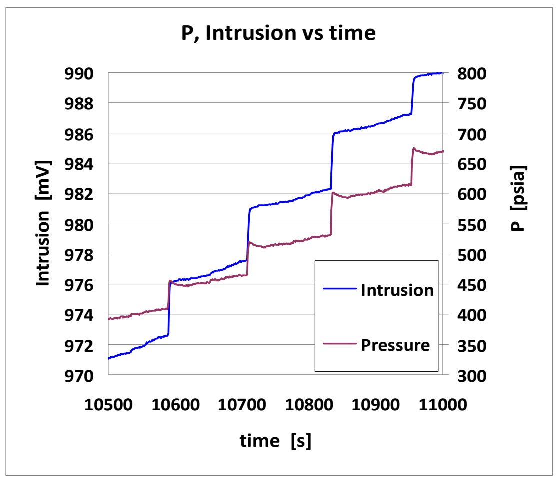

Figure 2. Time series (partial) of intrusion and pressure, as observed on a test sample. Note that it may take considerable time for the pressure to reach the set level. The 10s equilibration time is at the end of each pressure step.

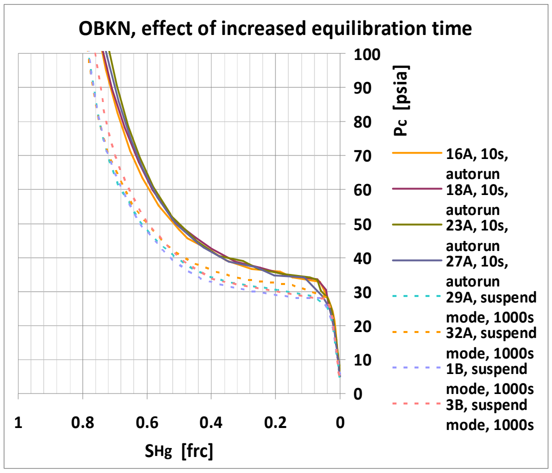

Figure 3. Measured capillary pressure curves on Oberkirchener sandstone outcrop. Four measurements were conducted with default equilibration time at 10s for all points, in automatic mode. A second set of four measurements was taken in "suspend" mode (see text), with 1000s equilibration time. The second set shows a saturation shift of some 20 saturation units around 35 psia.

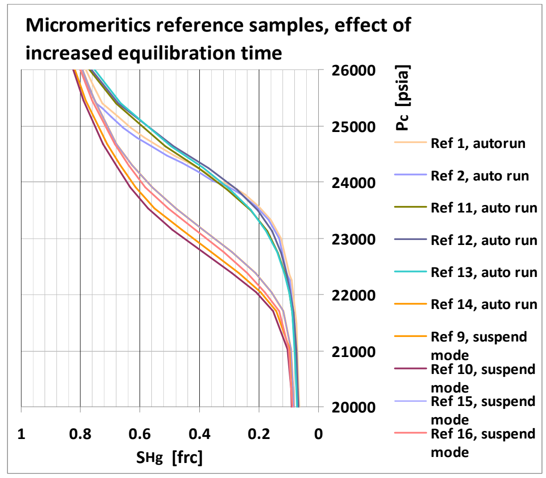

Figure 4. Measured capillary pressure curves on Micromeritics reference samples. Six measurements were conducted with the default equilibration time at 10s for all points, in automatic mode. A second set of four measurements was taken in "suspend" mode (see text), with 1000s equilibration time. The second set shows a saturation shift of some 20 saturation units around 23000 psia.

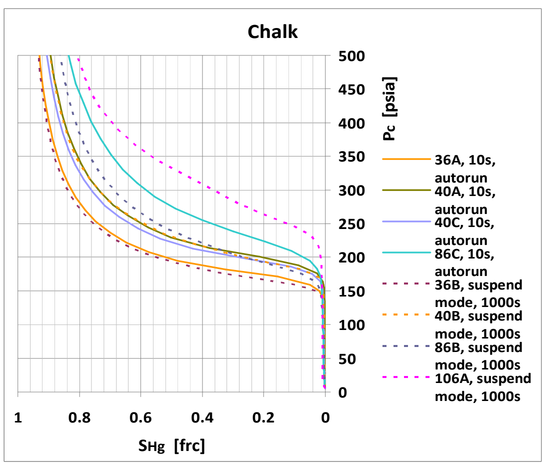

Figure 5. Measured capillary pressure curves on chalk samples. 4 measurements were conducted with the default equilibration time at 10s for all points, in automatic mode. A second set of four measurements was taken in "suspend" mode (see text), with 1000s equilibration time. The capillary pressures span a wide range and the effect of a longer equilibration time cannot be assessed by a comparison with base case runs.

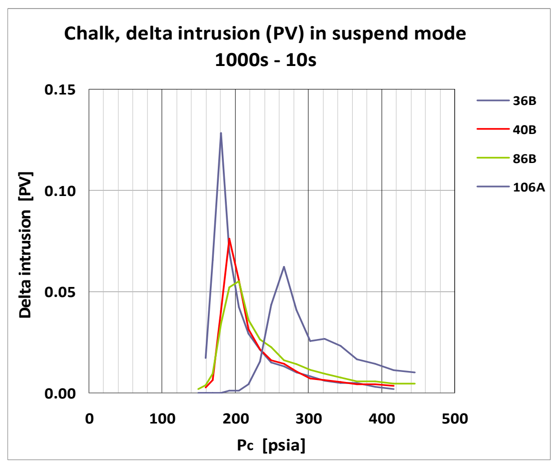

Figure 6. Measured shift in saturation at pressures around the plateau in the capillary pressure, comparing intrusion after 1000s equilibration time with intrusion after 10s. Samples differ in porosity and permeability. Maximum "differential" (see text) shift ranges from 5 to 12 saturation units.