LOW PERMEABILITY MEASUREMENTS USING STEADY-STATE AND TRANSIENT METHODS

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This paper was prepared for presentation at the International Symposium of the Society of Core Analysts held in Calgary, 10-12 September 2007

ABSTRACT

This paper presents results from a laboratory study comparing different techniques to measure permeabilities below 10 microDarcy such as tight reservoir and cap rock. Permeabilities were measured with gas using three different methods. An unsteady-state method based on the pressure fall off technique and a conventional steady-state method were used to measure permeabilities in conventional plugs of 70 mm length, 40 mm diameter. An unconventional method, was also implemented through a new permeameter device (Darcygas). The technique consists in setting rock fragments or a small plug in a small cell and studying the response to a pressure pulse. This method has the advantage to require very small pieces of rock (like drill cuttings) without any conditioning. Moreover, the measurement is extremely fast since the relaxation time is proportional to the square of the sample size.
Once all the measurements were corrected from Klinkenberg effects, the permeabilities measured with the three techniques were compared. Permeabilities determined from unsteady-state method are systematically higher values than permeabilities obtained with the steady-state method, even though the difference between the values remains low. Darcygas measurements performed on crushed samples lead to lower permeabilities than the other methods applied on plugs. However Darcygas values fit well with permeability values derived from mercury injection performed on neighbour small size samples. Experimental and theoretical factors are discussed to explain the results and to evaluate the efficiency and accuracy of the various techniques applied to tight formation characterization.

INTRODUCTION

Two fields are interested in accurately measuring tight rocks:
- In the petroleum industry, tight gas reservoirs constitute a significant percentage of the world gas accumulations and offer an important potential in terms of future reserve. In order to correctly evaluate the reserves of such reservoir, accurate petrophysical measurements of the formation are mandatory, among them monophasic permeability measurements.

- In addition to this valuable issue, accurate permeability measurement of the cap rock (most commonly clayey rocks of very low permeability) is a key issue for storage purposes such as CO_2 sequestration, and underground radioactive waste repository.

Difficulties in measuring permeability in rocks below 10 microDarcy are mainly due to extremely low flow rate. Experiments require therefore an adaptation of the experimental devices initially used for more permeable rocks. In recent years, both pulse technique and steady-state technique have been used to make gas permeability measurements in tight rocks.

The purpose of this study is to compare all the existing methods, from the traditional steady-state method to the less conventional pressure pulse technique applied on crushed cores, as well as permeability estimation from mercury-injection capillary pressure data. Another objective of the study was to build up a database through a wide range of permeability in order to determine correlations for Klinkenberg correction factor.

EXPERIMENTAL PROTOCOLE

Test materials

Permeability measurements were performed on core samples from caprock of three different oilfields. The core samples are mainly carbonates with a few percent of clay minerals, and sandstones with 20 to 30 % of clay minerals. The porosity varies between 2 and 22%. Three different sample sizes were used for this study.

- Samples referred as "Plug" are cylinders of 80 mm in length and 50 mm in diameter
- Samples referred as "Miniplugs" are cylinders of 15 mm in length and 10 mm in diameter
- Crushed samples of the mm size

For the plugs, NMR analysis, is performed 3 times: on fresh state, after drying and on brine saturated samples. High pressure mercury injection (HPMI) is also performed on companion plugs. NMR and HPMI measurements give the porosity and the pore size distribution. The porosity is an important input parameter for the interpretation of the transient response to get the absolute permeability.

Laboratory apparatus and experimental procedure

Steady-state gas permeability on plugs

Nitrogen is used for both steady-state and unsteady-state measurements. Steady-state measurements are performed on plugs placed in Hassler cells under hydrostatic confining pressure. Downstream pressure is kept at 10 bars while upstream pressure is fixed at several pressures in order to get several equilibrium points . The confining pressure is

maintained at 60 bars. For some samples the confining pressure is increased at 110 bars to evaluate the stress impact on the permeability. Flow rates are measured using a bubble tube flow meter, while upstream and downstream pressures are measured separately.

Unsteady-state gas permeability on plugs

The same apparatus as previously is used for the unsteady-state (or pulse decay) method. Initially, the sample is at equilibrium at atmospheric pressure and the outlet of the sample remains closed during the whole experiment duration. A pressure pulse of 30 bars is applied at the inlet and the decrease of the inlet pressure is analyzed and simulated to get the permeability. This method is particularly adapted to tight rocks (<10 microD) according to the Recommended Practices for Core Analysis (API, 1998). The standard interpretation method described in the API is based on an analytical solution that does not allow precise Klinkenberg correction since the pore pressure profile inside the sample is unknown. For this reason a numerical tool had been developed to model the pressure transient response. This numerical simulation allows to apply Klinkenberg correction in each grid block and for each time step. Klinkenberg factor b is calculated from an appropriate correlation (see chapter below). The absolute permeability is obtained by history matching.

Gas permeability on miniplugs or crushed cores (Darcygas)

This method uses pulse pressure testing with air (Luffel, 1993). The miniplug or crushed cores are placed into a cell. The air inside the cell is then quickly compressed using a piston. This overpressure decays with time to a lower pressure as the air moves into the pores within the sample. The pressure transient response is modeled to get the permeability. The main difference with the standard pulse-decay method is that there is no sleeve around the sample. The gas (air) enters on all the surface of the sample.

Liquid permeability on crushed cores (Darcylog)

The principle is quite similar to the previous method, except that gas is replaced by a viscous liquid in order to slow down the pressure decrease. The method was developed for drill cuttings but can be applied for miniplugs or crushed cores (Egermann et al., 2005). The experimental apparatus consists in a "cuttings cell" containing the sample and a pressure cell containing a bellow coupled to a spring and a pressure sensor. All the apparatus is filled with oil. Initially the samples are saturated with oil by spontaneous imbibition in a beaker. At the end of the spontaneous imbibition, some air remains trapped inside the cuttings. The cuttings are then poured in the cutting cell connected to the "pressure cell" (around 10 bars). Oil enters into the sample and the trapped gas is compressed. The pressure is recorded and the oil volume entering into the cuttings is derived from a calibration of the bellow. The rate of invasion depends on the fluid viscosity and the rock permeability. The permeability is calculated using a numerical model based on the equations describing the flow of a viscous fluid into a compressible medium. The pressure is chosen around 10 bars in order to have a negligible compressed air volume (around 1%), allowing interpretation as a monophasic flow of oil. Capillary pressure is also used to calculate the initial pressure in the trapped air bubbles (using

Laplace's law, the pore diameter being derived from permeability using a standard relationship).

Indirect Permeability Evaluation Methods

1) Many papers have been published on the permeability evaluation from NMR measurements, but very few refer to application on tight rocks. Several empirical laws (Kenyon, 1989; Timur, 1968) can be used with default parameters depending on the nature of the rock (sandstone or carbonate) but none of them has been calibrated with tight rocks and their application is questionable without further investigations.

2) A thin section can also be obtained from cuttings to evaluate the porosity and the permeability from image analysis. Unfortunately, this approach fails with tight rocks due to the insufficient resolution in the actual image analysis tools.

3) Several type of approaches have been proposed to derive permeability from mercury porosimetry curves (Hg Pc). The first one has been proposed by Swanson (1981) and consists in determining one particular point of the Pc curve that represents the characteristic throat size when the pore network connectivity is reached. This point (called APEX: $\left(\dfrac{V_b}{P_c}\right)_{max}$) corresponds to the tangent between the line of slope -1 with the Pc curve in appropriate scales (Figure1). The coordinates of the APEX point have been related to the permeability using a power law function:

$$k = 355\left(\frac{V_b}{P_c}\right)_A^{2.005}$$

The main advantage of Swanson's approach is that the APEX can be derived in a robust manner even when the first part of the P_c curve is not of sufficient quality, which is often the case with cuttings as rock samples. Therefore its application can be considered in a wide context.

The second type of approaches consists in history matching the Hg Pc curves with a parametric function and to link the permeability to the optimized parameters . Thomeer (1960, 1983) proposed an exponential law:

$$P_c = P_d \exp\left(-\frac{G}{Ln\left(\dfrac{V_b(P_c)}{V_b(P_\infty)}\right)}\right)$$

with G a parameter to shape the curve, P_d the displacement pressure and $V_{b\infty}$ the percentage volume occupied by mercury at the end of the invasion. Thomeer (1960, 1983) used 297 reservoir samples to establish the correlation between the permeability and the 3 parameters given hereafter:

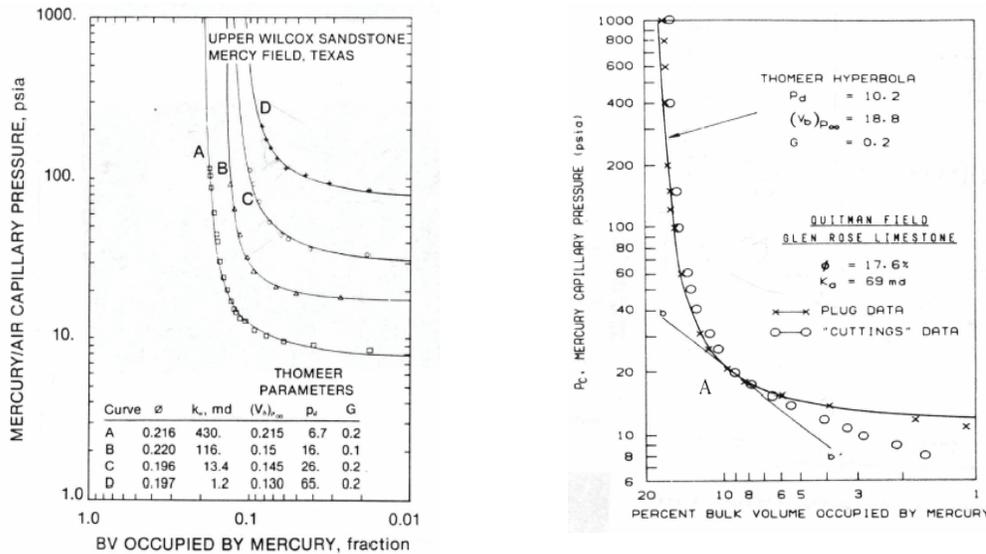$$k = 3.8068\,G^{-1.334}\left(\frac{V_{b\infty}}{P_d}\right)$$

Figure 1 : (a) Thomeer history matching (1960) – (b) Swanson APEX point determination (1981)

More recently Kamath (1992) performed a comparison between these methods and concluded that the best result is obtained with new correlations based on the Swanson characteristic length (1981). Two power law correlations were proposed according to the permeability range:

$$k = 413\,L_{\text{max}}^{1.85} \qquad \text{if } k < 1\,\text{md}$$

$$k = 347\,L_{\text{max}}^{1.60} \qquad \text{if } k > 1\,\text{md}$$

with L_{max} the characteristic length defined as followed (w for wetting, nw for non-wetting and r for residual):

$$L_{\text{max}} = \left(\frac{\phi\,S_{nw}}{P_c}\right)_{\text{max}} = \frac{\phi\,\lambda\,(100 - S_r)}{P_d\,(1+\lambda)^{\frac{1}{\lambda}+1}} \quad and \quad \left(\frac{P_d}{P_c}\right)^{\lambda} = \frac{S_w - S_r}{100 - S_r}$$

With this formulation, the Swanson characteristic length is calculated using a Brooks – Corey formulation of the Pc curve (λ is the exponent to shape the Pc curve). An interesting point of Kamath's study is the establishment of a dedicated formulation for low-permeability rocks (<1md). By referring to the calibration curve, it can be assessed that this empirical relation can be used for samples with permeability as low as 0.001md, which is adapted to tight reservoir rocks but not for caprocks.

Swanson, Kamath and Thomeer approches were tested on the mercury Pc-curves obtained on our selected samples.

KLINKENBERG CORRECTION

Darcy's law is based on the assumption that the flow is governed by the viscosity (Poiseuille flow). In this flow condition only collisions between molecules occurs which are represented by the notion of viscosity. However, when the pore size is small compared to the mean free path of the fluid molecules (low pressure or very small pores), there are no collisions between molecules but only between solid walls and molecules (Knudsen, 1950). For this type of flow, called Knudsen flow, there is still proportionality between flow rate and pressure drop, but the coefficient is not related to viscosity. Between these two extreme flow regimes, there is an intermediate case where the flow is controlled by the two types of collisions.

According to Scott and Dullien (1962), this transitional flow regime appears to extend from values of r/λ between 0.1 and 10, r being the channel pore radius and λ the mean free path of the gas at the experimental pressure and temperature conditions. In our case the ratio r/λ varies between 0.1 and 5 for all the samples. A correction (Klinkenberg 1941) can be applied to Darcy's law to account for the deviation from the Poiseuille's law.

$$Kg = K\infty \, (1 + b/P)$$

where $K\infty$ is the absolute permeability, Kg is the apparent gas permeability, P is the average pore pressure and b the Klinkenberg coefficient that depends on the gas and rock properties.

The steady-state method allows to perform gas permeability measurements at different pore pressures on the same sample. By extrapolating to the high pressure case $K\infty$ and b are directly given. We applied this method to all the available tight samples in order to create a database. This database was completed by results published in the literature (Jones, 1980) in order to extend the range of permeability up to 1 Darcy, until the Klinkenberg effect becomes negligible. The results of this database (representing a total of 46 samples) is given in Figure 2 as a plot of the logarithm of the Klinkenberg factor versus the logarithm of the permeability $K\infty$. The data are scattered closely about a straight line. Three correlations found in the literature, based also on nitrogen permeability measurements, are added to the graph. For the range of permeability of our samples (0.0001 to 1mD), the correlation that best fits the data is the one from Jones (1979) based on tight sand measurements.

This correlation is used to correct from the Klinkenberg effect the permeability measured with the transient methods (unsteady-state method on plugs and Darcygas on miniplugs) that were not performed at different pressures.
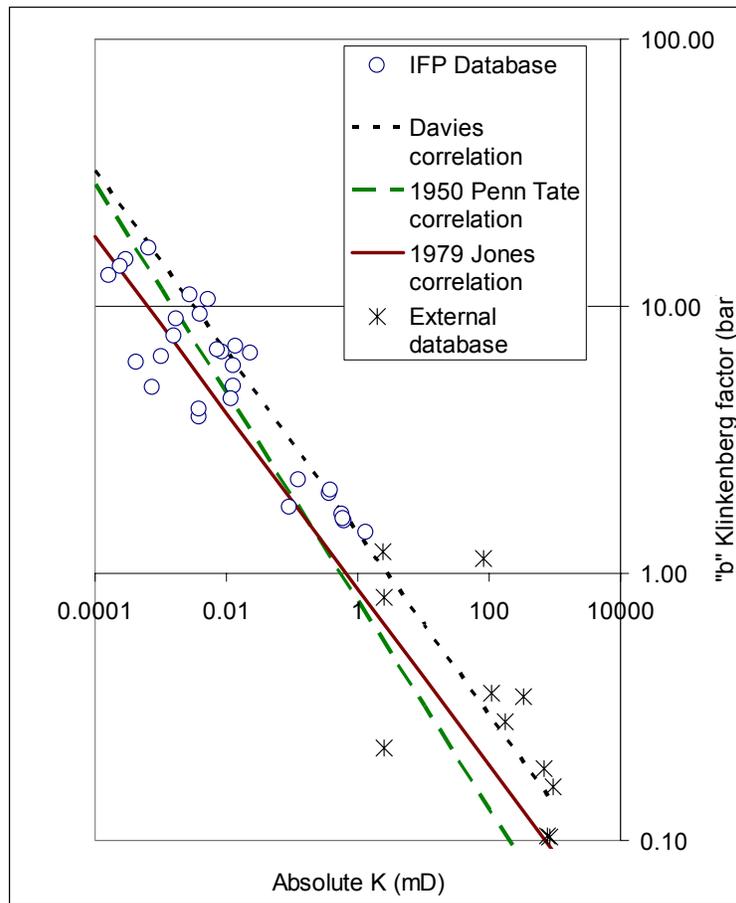
Figure 2: Klinkenberg factor versus absolute permeability for samples of various permeability and tested with nitrogen

RESULTS

Steady-state versus unsteady-state permeability measurements on plugs

All the permeability values plotted in Figure 3 are corrected from the Klinkenberg effect. Steady-state and unsteady-state measurements were performed on 8 samples with a wide range of permeability. In order to make the comparison valuable, each sample was tested with both techniques, under the same effective stress, using the same apparatus with the same gas (nitrogen).

As seen in this plot, the unsteady-state technique gives always higher values than the steady-state technique. The differences between the permeabilities are directly proportional to the absolute permeability and the permeabilities from unsteady-state are roughly twice the permeability from steady-state. Other authors (Rushing et al., 2004) have reported a ratio of about 8 for the two methods for permeabilities less than 0.01mD, but higher values for steady-state permeabilities. Up to now, we have no explanation for this difference between the techniques.
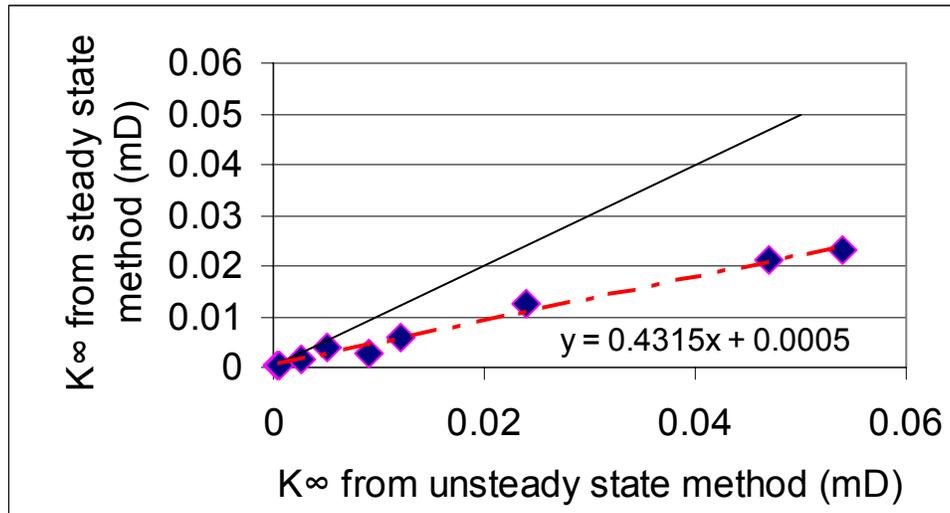
Figure 3: Comparison of Klinkenberg-corrected permeabilities measured on the same plugs using the unsteady-state and the steady-state methods.

Comparison between permeability results from Darcygas, Darcylog and mercury porosimetry curves

Comparison between permeability from Darcygas and correlations from mercury injection are shown in Figure 4 and Figure 5. On the first graph (Figure 4), the samples have a permeability in the order of 10^{-1} microDarcy. Except for sample 7, Darcygas method gives higher absolute permeability than permeability given from Kamath, Swanson or Thomeer correlations. Among correlations from mercury injection, Kamath correlation, which was developed for samples below 1mD, gives a better estimate of the absolute permeability measured with Darcygas method. On the other hand Swanson correlation underestimates significantly the permeability. Between Kamath correlation and Klinkenberg-corrected Darcygas permeability, the maximum margin of error is 0.4 microDarcy.

Permeability results from samples with a different lithology (more clay-content) are presented on Figure 5. These samples (samples 8 to 11) being ten times more permeable (in the order of the microDarcy) than the previous ones, Darcylog technique could have been performed for comparison. As seen on the graph, Klinkenberg-corrected Darcygas and Darcylog permeabilities are close. Again Kamath correlation gives permeabilities closer to Darcylog and Darcygas values while Swanson correlation underestimates much the permeability. Between these three methods (Kamath, Darcygas and Darcylog), the maximum margin of error is 2 microDarcy.
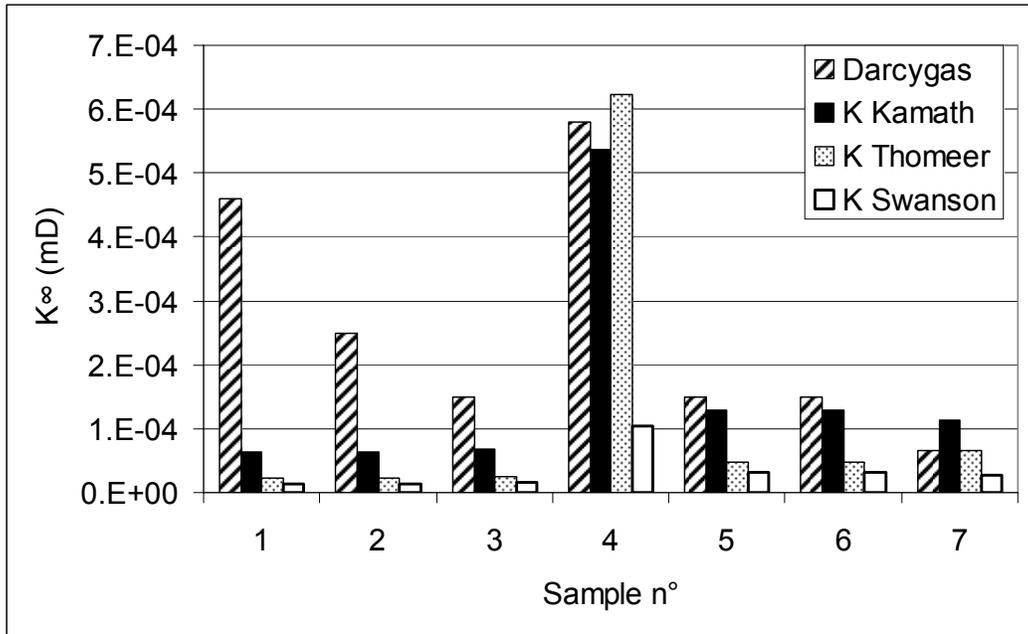
Figure 4: Comparison between Klinkenberg-corrected permeability from Darcygas and permeability from mercury correlations
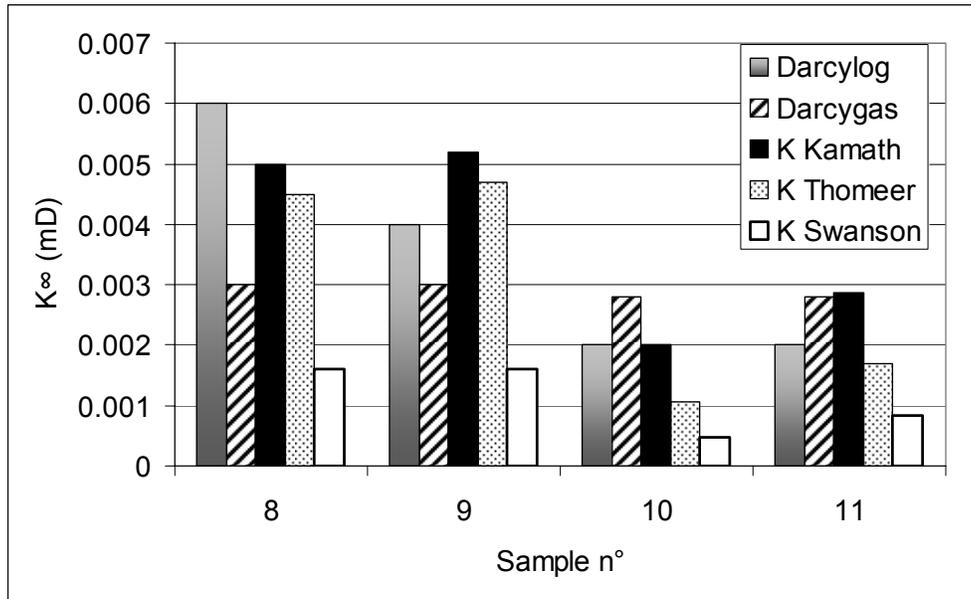

Figure 5: Comparison between permeabilities from Darcylog and Klinkenberg-corrected permeabilities from Darcygas

Comparison between permeability results from Darcygas on miniplugs and from steady-state technique on neighboring plugs

Measurements using the steady-state technique were performed with a confining pressure of 60 bars and a pore pressure varying during the experiment from 10 to 20 bars. This leads to a mean effective stress of about 45 bars applied to the samples during the steady-state measurement while Darcygas measurement was performed without confining pressure (atmospheric pressure). Because of the effective stress difference, permeabilities from Darcygas were expected to be higher than with the steady state technique, which is not the case from our results (Figure 6). In order to evaluate roughly the stress effect on the permeability, some plugs were tested in steady-state at a higher confining pressure (110 bars and one sample up to 240 bars). The effect of confining pressure on our samples is in agreement with the permeability reduction found in the literature on low permeability sandstones (Jones, 1980). In average the permeability is 1.1 to 1.7 times higher at 60 bars than at 110 bars of confining pressure (Figure 7).
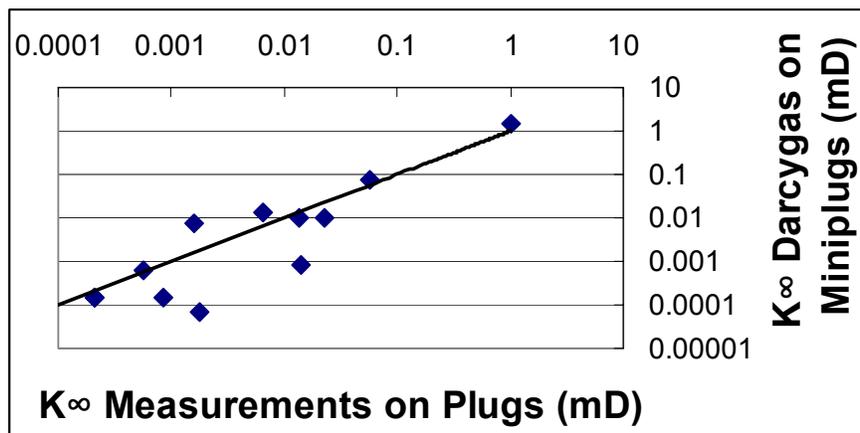

Figure 6: Comparison between permeability on miniplugs with Darcygas and permeability on neighbor plugs with the steady-state method.

DISCUSSION/CONCLUSIONS

- Using gas instead of brine in permeability measurements has the advantages to be non reactive (a crucial parameter for clay-rich samples), and less prone than liquid to mobilize the fines. However, while making gas permeability measurements on tight rock, different flow regimes may occur: Poiseuille, Knudsen, or a transition stage between this two regimes. The gas molecule mean free path and the pore throat size sample are parameters easy to estimate and can help in determining which law better describes the gas flow inside the sample. For our samples and gas type, the flowing regime is transitional and the Klinkenberg correction can be applied to Darcy's law.

- One of the advantage of using the steady-state technique in this flowing regime is the possibility to perform gas permeability measurements on the same sample, at

different pore pressures, in order to get directly the Klinkenberg-corrected permeability. However measuring steady-state permeability of tight plugs is time consuming: for each pressure point, the equilibrium is very long to reach since the relaxation time is proportional to the square of the sample size. Moreover artifacts can occur in long time experiments (sample drying or condensating, leaks,..).
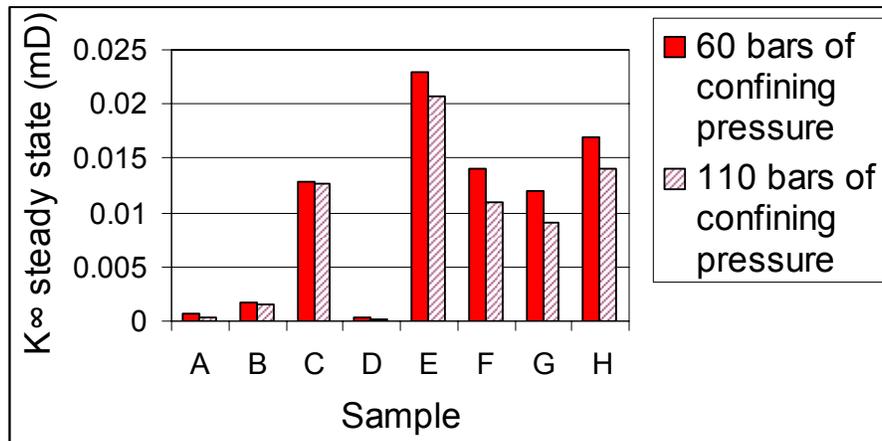

Figure 7 : Stress effect on permeability

- The unsteady-state technique is faster than the conventional one. However the absolute permeability is not given directly from the test but is calculated with Klinkenberg factors from existing correlations. Moreover, during the test only the inlet and the outlet pressure are monitored. It is impossible to know precisely the sample pressure profile and therefore the real average pore pressure needed in the Klinkenberg equation for Klinkenberg correction. This leads to more uncertainties in absolute permeability calculations.

- From our results, unsteady-state method gives systematically higher values than the steady-state method. The difference between the permeabilities is directly proportional to the absolute permeability values. Whether it is a technical, numerical or physical discrepancy is not clear yet. Further investigations are on going.

- Darcygas technique applied on miniplugs gives promising results in quite good agreement with steady-state technique and Darcylog measurements. More samples need to be tested with the methods presented in this paper in order to better quantify the uncertainties. The Darcygas has the same advantages as the Darcylog (fast, easy to handle, no specific conditioning, small sample size) and complete the range of measurable permeability (Darcylog can measure down to 10 microDarcy while Darcygas can be used below 10 microDarcy). Currently additional tests are performed with smaller samples like drill cuttings. One of the disadvantages of the Darcygas is related to the fact that Klinkenberg effect is

enhanced by low pore pressure measurements. Some studies are on going to set up similar experimental apparatus at higher pore pressure.

ACKNOWLEGMENTS

REFERENCES

API, "Recommended Practices for Core Analysis", *Recommended Practice 40*, second Edition, February 1998.

Egermann P., Lenormand R., Longeron D., and Zarcone C.: "A Fast and Direct Method of Permeability Measurements on Drill Cuttings", August 2005 *SPE Reservoir Evaluation & Engineering,* p. 269-275.

Jones, FO and Owens WW: "A Laboratory Study of Low-Permeability Gas Sands", *Journal of Petroleum Technology*, september 1980, p. 1631-1640.

Kamath J., Boyer R. E. et Nakagawa F. M. : "Characterisation of Core-scale Heterogeneities using Laboratory Pressure Transients", *SPE Formation Evaluation,* September, pp 219-227, 1992.

Kenyon W. E. : "A Three-part Study of NMR Longitudinal Relaxation Properties of Water Saturated Sandstones", SPE Formation Evaluation, March, pp 622-636, 1989.

Klinkenberg L.J., "The permeability of Porous Media to Liquids and Gases: Drilling and production Practice", 1941, p.200-213.

Luffel D.L., Hopkins C.W., and Shettler, P.D., "Matrix Permeability Measurements of Gas Productive Shales" SPE 26633, October 1993, *68th Annual Technical Conference and Exhibition of the SPE held in Houston.*

Rushing J.A., Newsham K.E., Lasswell P.M., and Blasingame T.A., "Klinkenberg-Corrected Permeability Measurements in Tight Gas Sands: Steady-State Versus Unsteady-State Techniques", SPE 89867, September 2004, *Annual Technical Conference and Exhibition of the SPE held in Houston.*

Scott, DS and Dullien FAL, : "Diffusion of Ideal Gases in Capillaries and Porous Solids" *A.I.Ch.E.Journal*, 1962, V.8, p.113-117.

Swanson B. F. : "A Simple Correlation between Permeability and Mercury Capillary Pressures", JPT, December, pp 2498-2504, 1981.

Thomeer J. H. M. : "Introduction of a Pore Geometrical Factor defined by the Capillary Pressure Curve", *Trans AIME*, March, pp 73-77, 1960.

Thomeer J. H. M. : "Air Permeability as a Function of Three Pore Network Parameters", *Trans AIME,* April, pp 809-814, 1983.

Timur A. : "An investigation of Permeability, Porosity and Residual Water Saturation Relationships" , 1968, *SPWLA*.