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Advances in Measurement Standards and Flow Properties Measurements for Tight Rocks such as Shales

S.Sinha, SPE, E.M. Braun, SPE, Q.R. Passey, SPE, S.A. Leonardi, SPE, A.C. Wood III, T. Zirkle, SPE, J.A. Boros, SPE, and R.A. Kudva, SPE, ExxonMobil Upstream Research Co.

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Abstract

Determination of permeability of unconventional reservoirs is critical for reservoir characterization, forecasting production, determination of well spacing, designing hydraulic fracture treatments, and a number of other applications. In many unconventional reservoirs, gas is produced from tight rocks such as shale. Currently the most commonly used industry method for measuring permeability is the Gas Research Institute (GRI) technique, or its variants, which involve the use of crushed samples. The accuracy of such techniques, however, is questionable because of a number of inadequacies such as the absence of reservoir overburden stress while conducting these measurements. In addition to questionable accuracy of crushed rock techniques, prior studies have indicated that there is significant variability in results reported by different laboratories that utilize crushed-rock technique to measure permeability on shale samples. Alternate methods are required to obtain accurate and consistent data for tight rocks such as shales. In this paper we discuss a robust steady-state technique for measuring permeability on intact tight rock samples under reservoir overburden stress. Permeability measurement standards for low permeability samples are critical for obtaining consistent results from different laboratories making such measurements, regardless of the method used for measuring permeability. In this paper we present permeability measurement standards developed based on first principles that serve as the "ground-truth" for permeability in the 10 – 10,000 nanoDarcy range. These standards can be used to calibrate any permeability measurement apparatus used to measure permeability on intact tight rock samples such as shales, to enable delivery of consistent results across different laboratories conducting measurements on intact tight rock samples.

Introduction/ Background

The permeability of conventional reservoir rocks has been measured using intact plug samples for decades in the oil and gas industry using various methods, most commonly steady-state and pulse decay. These rocks often have permeability greater than 1 milliDarcy, and the methods indicated above have been found to provide reasonably accurate results in relatively short times. Furthermore, there is little discrepancy in permeability measurement results reported by different laboratories even when different methods are used to measure permeability on intact plug samples (Thomas & Pugh, 1989). For low permeability reservoirs such as shales, the most commonly used technique for measuring permeability is the GRI method (Luffel et al., 1993; Guidry et al., 1996) which is carried out on crushed rock samples. The method is popular because of the relatively quick times associated with the measurements due to the increased surface area available while using crushed rock samples, and because of the perception that measurements on intact low permeability plug samples using well established methods such as steady-state or pulse decay could take such long times that it would make such measurements impractical. However, using crushed rock for measuring permeability has a number of inadequacies as is discussed in the next section, resulting in skepticism associated with the accuracy of such measurements. Furthermore, there was no prescribed mathematical formulation presented in the GRI literature to analyze the measured data, resulting in an absence of standardized methodology to deduce a permeability value from the raw data. Each vendor offering permeability measurements using crushed rocks has developed its own proprietary mathematical formulation to analyze the data; neither the methods nor the data are typically shared with customers. Perhaps the most serious drawback of the crushed-rock method for measuring permeability, as presented in a number of prior studies, is the wide variability in results reported by different

vendors, with results that could differ by as much as 3 orders of magnitude (Passey et al., 2010). This variability makes it difficult to compare different sections of the same field or different fields, when rock samples are sent to different vendors for permeability measurements. Inaccessibility to the measured raw data and mathematical formulation used for analysis makes it extremely difficult for data users to determine the root cause of the discrepancies. The observed variation in results reported by the different vendors using similar techniques indicates a critical need for permeability measurement standards for low permeability rocks to enable calibration of equipment. In addition, there is a need for "ground truth" permeability standards, to determine if a laboratory apparatus and associated technique is suitable for permeability measurements of tight rock samples. Such permeability standards must be in the same permeability range as low permeability reservoir rocks, able to be easily handled, and insensitive to environmental factors such as humidity and mechanical stress. There are a few commercially available products that could be used to calibrate apparatus used for permeability measurements, but none of these are suitable or convenient for testing and calibration of equipment used for measuring low permeability samples. Some of these products are discussed below.

Comercially available calibration standards

Several core analysis equipment vendors offer check plugs for calibrating equipment used for measuring permeability (permeameters). These consist of porous sintered metal encased in a non-porous metal shell, and require empirical calibration to determine their exact permeability values. Available permeability values are advertised to be from 0.05 to 1000+ milliDarcies (Vinci technologies website; Coretest Systems Inc. website). This range is considerably higher than the permeability values observed in rocks from producing shale gas reservoirs, rendering these check plugs unusable for most of the shale gas apparatus calibration.

Laminar flow elements

Laminar flow elements are used to provide known flow rates of gas that are proportional to the supplied gas pressure. These elements are used commercially for instrument calibration and industrial process control. Most commercially available laminar flow elements are designed for very low pressure drop and therefore have far less flow resistance than required to calibrate low permeability permeameters. In addition, they do not have the aspect ratio and geometry required for use in a permeameter. Instead they connect to other components using tubing, flanges or threaded connections.

Calibrated leak standards

Calibrated leak standards consist of a pressurized gas source and a flow resistor. They are intended to provide a known and very low flow rate of gas for calibration of gas "sniffers" (detectors) or testing of high vacuum apparatus. However, commercially available leak standards do not have the cylindrical geometry required for use in a permeameter.

The permeability measurement standard presented in this work overcomes the shortcomings of other commercially available products discussed above. This is discussed in the subsequent sections.

Shortcomings of GRI or crushed-rock technique of measuring permeability

The GRI (Luffel et al., 1993; Guidry et al., 1996) or crushed-rock technique for measuring permeability is a relatively fast method for measuring permeability on low permeability samples, but it has the following shortcomings:

Absence of overburden stress

The GRI technique is implemented on crushed rock samples and therefore these measurements are not conducted at reservoir overburden stress. In conventional rock samples, the sensitivity of the samples to the overburden stress is more pronounced for samples with lower permeability. Ignoring the effect of overburden stress while measuring the permeability could lead to significant discrepancy between permeability measured in the laboratory and in-situ permeability.

No Klinkenberg correction and Darcy's law assumption

The GRI permeability is measured at pore pressures that are much lower than reservoir pressures. At lower pore pressures, the flow of gas through tight rock samples such as shales may be in the free molecular flow regime or transition regime due to the relatively small pore sizes in the nanometer size range. The continuum assumption and, therefore, Darcy's law may not be valid under those conditions. Furthermore, gas slip or Klinkenberg correction is ignored in the GRI method. Conducting permeability measurements at low pressures or ignoring the Klinkenberg correction for tight rock samples could lead to significant departure from in-situ permeability values.

Inconsistency and lack of standard analytical expression

As discussed earlier, a number of studies (Passey et al., 2010; Spears et al. 2011; Sondergeld et al., 2010) have found that there is significant variation (up to 3 orders of magnitude) in results reported by different laboratories, while using GRI or crushed-rock method for permeability of shales. It is difficult to determine if these variations are related to the apparatus or due to the method used to interpret the measured data. The GRI report (Luffel et al., 1993; Guidry et al., 1996) does not give a detailed methodology for interpreting the raw data, and each of the commercial laboratories has developed their own proprietary techniques for interpreting the data. In most cases, neither the data analysis technique nor the actual measured data is shared with the users.

Time scales required for GRI measurements

The original GRI method was developed using Devonian shale samples with matrix permeabilities in the range of 0.002 to 0.45 nanoDarcy (Luffel et al., 1993), a few orders of magnitude lower than shale assets that are currently of commercial interest . This raises the question of whether the original GRI crushed particle size of 20/35 mesh (0.67 ± 0.17 millimeter) is appropriate for modern measurements.

A simple mathematical analysis indicates that the time response of a pressure decay measurement should scale as follows:

$$t \propto \frac{d^2 \varphi}{k} \qquad \text{(Equation 1)}$$

Thus, testing a sample 1000 times more permeable than Devonian shale will require measurements to be conducted 1000 times faster. Although instrumentation exists to make such rapid measurements, it is not in common use in core analysis laboratories.

Alternatively, to keep the same measurement time, the size of the particles of crushed rock should be increased by a factor of $\sqrt{1000}$, i.e. approaching the size of a standard core plug.

To experimentally confirm the need for larger particle sizes and/or rapid measurements, a pressure-decay measurement, similar to that of the GRI method, was conducted on a single 1½-inch core plug with a measured permeability of 14 microDarcies. This measurement was carried out in a helium expansion apparatus of the type used to measure grain volume in routine core analysis, shown schematically in Figure 1. The results are shown in Figure 2. Approximately one-half of the pressure response was observed in the first 12 seconds of this measurement. Scaling this response to a typical GRI sample (100 nanoDarcy rock crushed to 0.67-millimeter particles) indicates that half of the pressure response would occur in 0.5 second. Measurements substantially faster than this would be required to adequately define the curve shape for a collection of particles of this size.

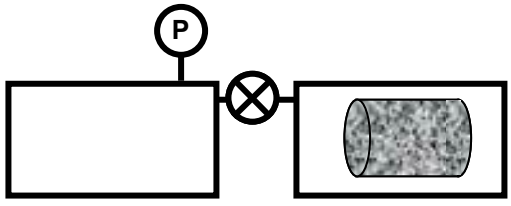

Figure 1. Schematic of pressure-decay measurement on intact plug using helium expansion apparatus.

These conclusions are consistent with the findings of Profice et al., 2011, who proposed to perform an experimental pre-estimation of permeability using an intact core plug, in order to define the appropriate particle size for a crushed-sample permeability measurement.

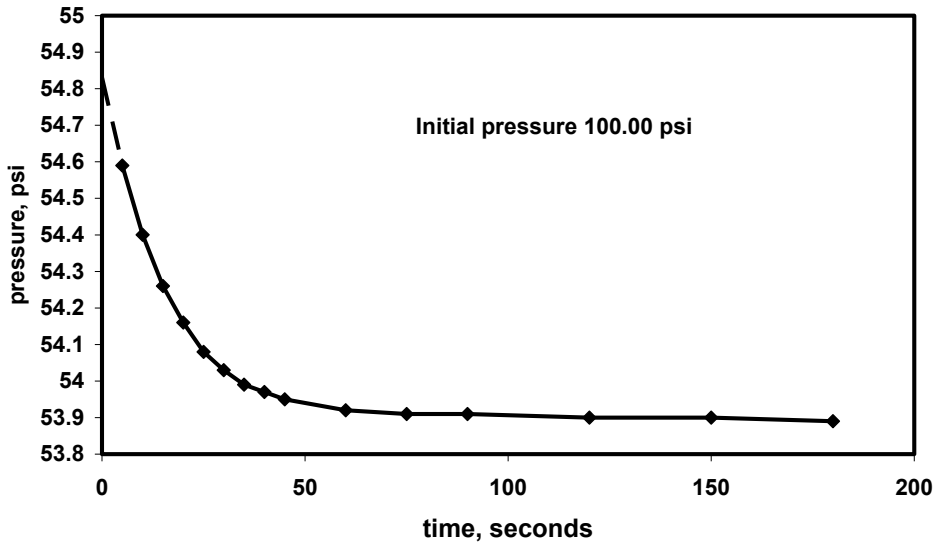

Figure 2. Pressure-decay data measured on a 14 microDarcy core plug

Effect of sample heterogeneity on pressure response

The GRI method of measuring permeability requires approximately 100 grams of rock material. Within the same rock sample there could be a variation of permeability due to vertical heterogeneity (Dewhurst et al., 2002) within the sample or due to heterogeneity in mineralogy within the sample.

Figure 3 illustrates a difficulty in interpreting data measured by the GRI pressure-decay method when the individual pieces of crushed core have different permeability values. The black curves represent the pressure response that would be expected for various permeability levels. On the normalized and logarithmic scale shown here, the pressure responses have identical shapes, but are offset horizontally in proportion to permeability. Shown in red is the expected response when the crushed core consists of three equal groups of particles varying 100-fold in permeability. The pressure response differs considerably from the response of homogeneous pieces, particularly at later times. Attempting to interpret the mixed-permeability data using a single-permeability assumption would lead to a poor match to the lab data and a large uncertainty in the results. This may be a factor in the large lab-to-lab discrepancies as found in prior studies (Passey et al., 2010; Spears et al. 2011; Sondergeld et al., 2010).

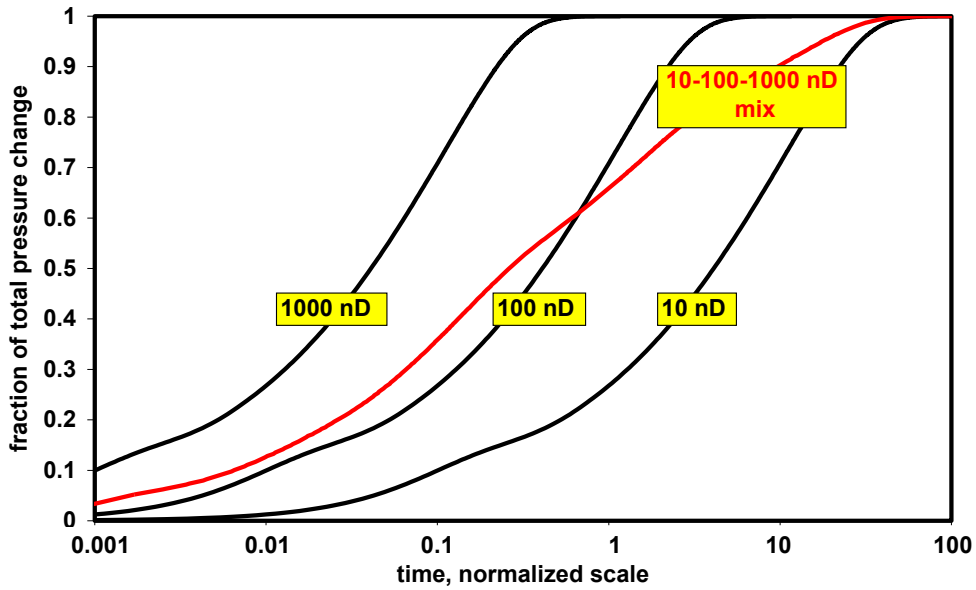

Figure 3. Pressure response for mixed chips in GRI method

For this case, it is interesting to note, however, that at early times, the mixed-permeability data could be matched reasonably well using a single permeability of 175 nanoDarcy. It is necessary to consider the late-time data in order to recognize that the shape of the mixed-permeability curve is distinctly different from the single-permeability curves.

Steady state method of measuring permeability

The steady-state method (American Petroleum Institute, 1998) has been used to measure permeability of conventional rocks for several years. The measurements are carried out on plug samples that are approximately 1"-1.5" in diameter and more than 1" in length. The method was not used frequently for low permeability samples because of the difficulty in measuring the low flow rates associated with tight rock samples, and the perception that these measurements could take very long times. In our laboratory, we have developed apparatus to measure gas permeability values as low as 10 nanoDarcies on core plugs. A schematic of this apparatus is shown in Figure 4. A computer-controlled pump, fed with gas from a gas cylinder, maintains a constant pressure at the upstream end of the core, while a lower pressure at the downstream end of the core is maintained using a back pressure regulator (BPR). A sleeve fluid confining pressure system is used to maintain a constant confining pressure on the core sample during the measurement. The pump control software logs positions of the pump pistons, and this data is plotted against time to obtain flow rates, as shown in Figure 5. With known sample geometry, pore pressure, and measured flow rates, it is straightforward to use Darcy's law to compute permeability for the test sample. For extremely low permeability measurements, corrections must be made for the small amount of gas leakage that is observed. The gas leak rates in this system is commonly less than 5% of the total measured flow rate during measurements on samples with permeability greater than 100 nanoDarcies.

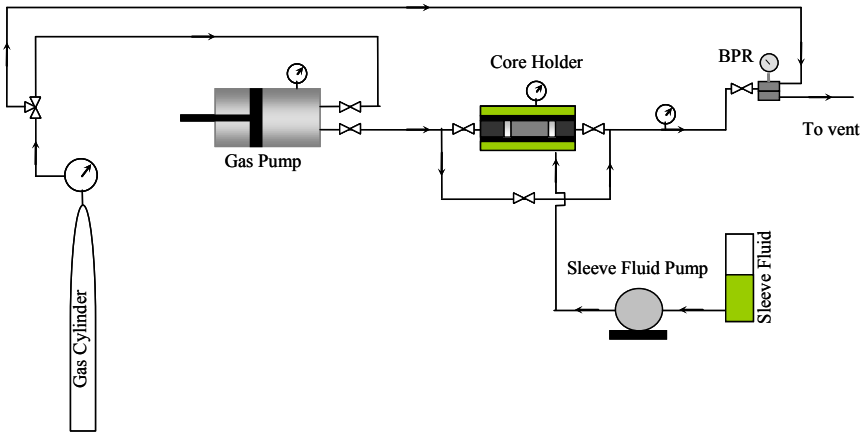

Figure 4. Schematic of steady-state apparatus for measuring permeability on very tight samples

This approach makes it possible to obtain high quality data for studying the dependence of permeability on conditions such as:

- Pore pressure
- Pressure drop
- Confining pressure
- Gas composition
- Equilibration time after changing conditions

For samples with permeability values greater than 100 nanoDarcies, the measurement time is not more than a few days, with more time required for tighter samples. The sample geometry also affects the measurement time, with faster measurements for shorter samples and larger cross-sectional area exposed to flow. Even though steady-state measurements on very tight samples are robust, a small leakage in the system could significantly affect the measured permeability, and therefore a leak test is conducted before each measurement at the same conditions as the permeability measurements. The system is also calibrated with standards described later in this paper, to ensure that the apparatus yields accurate results.

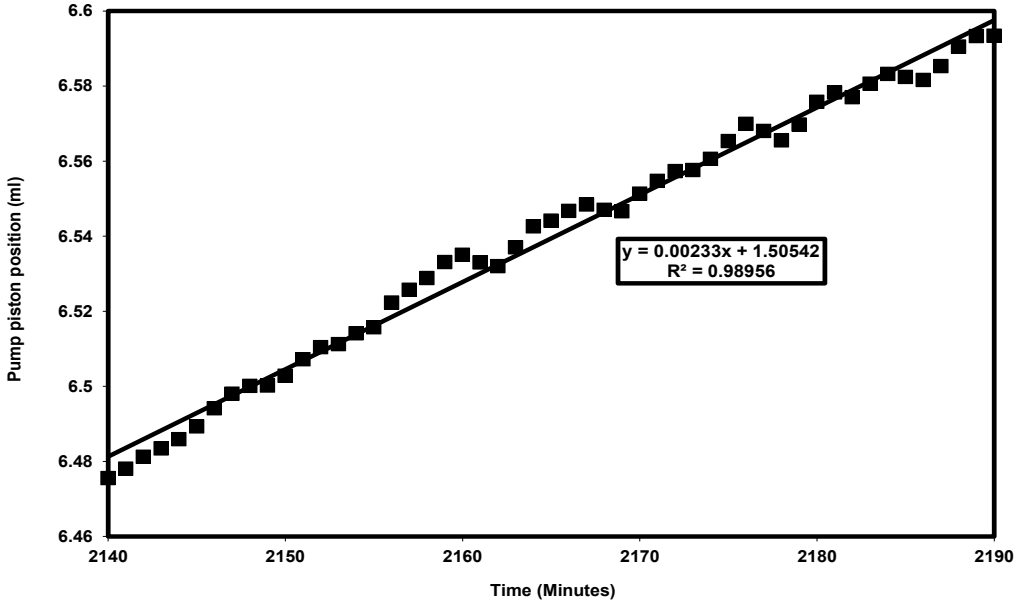

Figure 5. Derivation of gas flow rate from pump position during steady-state measurement of gas permeability. Expanded scale showing 50 of 3600 minutes of total measurement time.

One of the justifications for using crushed-rock technique for measuring permeability on tight rocks, as claimed in the original GRI report (Luffel et al., 1993; Guidry et al., 1996), was to avoid the affect of coring induced microcracks in the rock samples because plug sized samples may be more prone to such cracks compared to crushed rocks. However, if there are naturally occurring microcracks in the rocks that serve as a permeability pathway for the gas in the rock, then crushed rock would not account for such in-situ features in the rock fabric. If microcracks are detected in the samples, it is difficult to determine conclusively if such features are naturally occurring, or induced during the sample preparation. In our experience, if plug-sized samples are screened appropriately to avoid obvious larger cracks in the rock by using methods such as computed tomography (CT) scans, then permeability measurements on such plugs yield plausible results.

Figure 6 shows some of our preliminary data measured on shale plug samples using the steady-state method, compared with crushed-rock permeability measurements conducted by two different vendors on similar samples. The steady-state measurements were conducted with helium (similar to crushed-rock techniques), at reservoir net confining pressure, and at low pore pressures (similar to the crushed-rock technique). It is important to note that the measurements shown in Figure 6 are on samples from different formations that are not identified here; however, each data point represents a comparison between vendor and in-house steady-state measurements conducted on nearly identical samples. The large horizontal error bars indicated on the crushed-rock measurements conducted by vendor B is to account for measurements on samples that were not at exactly the same depth as the ones used for steady-state measurements. The error bars represent measurements on samples that were higher and lower in depth compared to the ones used for steady-state measurements. The small vertical error bars (smaller than the size of the data point symbols on Figure 6) on steady-state measurements account for the leakage in the apparatus, and as observed in the figure, the error is small relative to the actual measurements.

It is evident from Figure 6 that with the exception of one measurement, the steady-state permeability values are lower than those measured by Vendor A. If the steady-state measurements are affected by presence of microcracks unlike the crushed rocks then the steady-state measurements should have been higher than the crushed-rock measurements reported by Vendor A. In contrast, when compared with Vendor B, the steady-state measurements are higher, but in the same range of permeability that would be expected (~ 10 – 3000 nanoDarcy) for commercially producing gas shales. In general, we have not found a systematic trend between steady-state measurements on plugs, and crushed-rock permeability measurements; although if a trend between steady-state measurements and the individual vendors exists is a subject of further investigation.

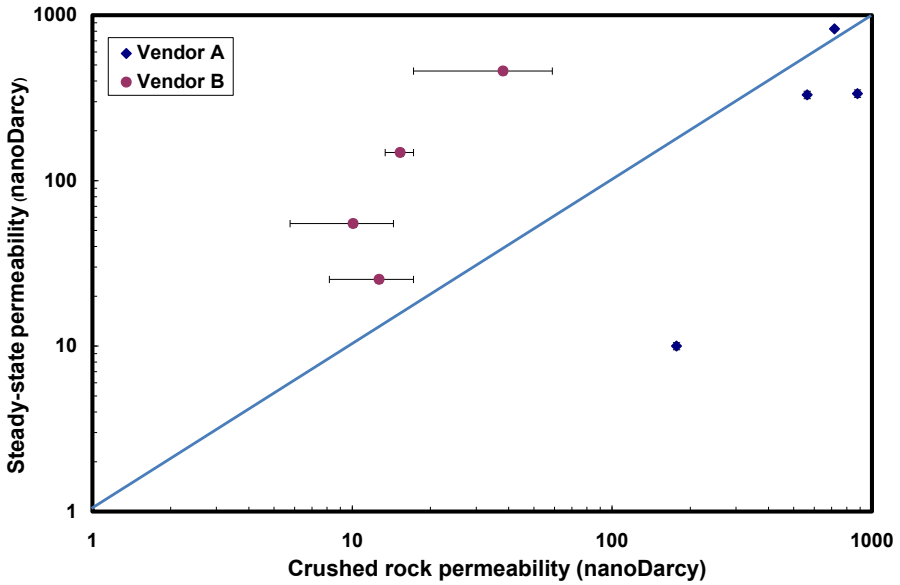

Figure 6. Comparison between steady-state permeability measurements on plug samples and pressure decay permeability measurements conducted on crushed rock samples by two different vendors (on a log-log scale). The blue line represents a perfect agreement between the steady-state and crushed-rock permeability values.

Capillary based Standards for low permeability measurements

The limitations of the crushed-rock permeability measurement method as discussed in the previous sections, and the feasibility of conducting permeability measurements using steady-state and other methods in a practical time interval makes plug based methods of measuring permeability more reliable compared to current industry practices. However, it is imperative to have a permeability standard which is in the same range of permeability as tight rocks, to ensure that different laboratories and vendors using plugs to conduct permeability measurements yield consistent results while measuring very low permeability. Furthermore, to ensure accuracy, it would be very advantageous to have a standard for which permeability could be computed from first principles, eliminating the need for empirical calibration.

Presented below is a description of permeability standards designed and developed based on fluid flow fundamentals, so that the permeability of the standards can be simply computed using a mathematical formulation. The standards are similar in shape and size to standard core plugs used in measurements. Plug-based permeability measurement apparatus can, thus, be calibrated using these standards, ensuring that the measured permeability is in agreement with the computed permeability, within bounds of experimental and design error.

We have designed and developed low permeability calibration standards[1] of known permeability by creating micron sized channels in an impermeable matrix of the same size as a typical plug sample. These may be fabricated by embedding one or more capillary tubes of known internal diameter in the matrix, or by drilling controlled-sized holes in the matrix. The permeability is calculated from first principles, using the Hagen-Poiseuille equation for laminar flow in a circular conduit (Bird et al., 2002) and it can also be verified by experimental measurements. Materials used in fabrication of the standards are ones that are not susceptible to change from external influences such as mechanical stress, humidity, etc. The internal diameter of the capillaries is known, and can be determined from manufacturers' specifications or by direct measurement using optical, micro-imaging techniques or flow based techniques.

Figures 7 and Figure 8, represent a sketch and a photograph of one of our calibration standards, respectively. The standards were fabricated by drilling a hole along the length of a stainless steel cylinder, and using epoxy to cement a precision-bore glass capillary in the hole. Compressed air was used to confirm that flow occurred only through the bore of the capillary, with no bypassing through the epoxy-filled annulus.

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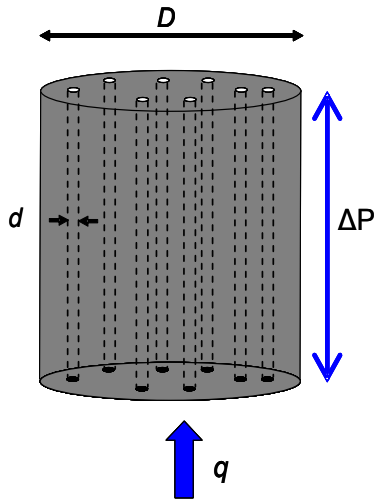

Figure 7. Sketch of Capillary Based Permeability Standard.

Figure 8. Capillary-based permeability standard (47 micron diameter channel).

Figure 9 is one frame of a video showing bubbles emerging only from the bore of the capillary, confirming that no leakage occurs in the epoxy-filled region.

Figure 9. Frame of a video showing a gas bubble emerging only from the glass capillary

The outside diameter of the calibration standard, D, is similar to that of a core plug used for permeability measurements, typically 1 or 1½ inches, so that they can be used to calibrate the apparatus used to measure permeability on rock samples.

$$q = \frac{\pi \, \Delta P \, R^4}{8 \, \mu \, L} \left(\frac{P_{mean}}{P_{out}} \right) \qquad \text{(Equation 2)}$$

Using the Hagen-Poiseuille equation (Bird et al., 2002), the volumetric gas flow rate exiting the calibration standard is given by equation 2. If the glass capillary is considered as a porous medium, then the permeability of the channel with internal diameter d, can be characterized using Darcy's law as in equation 3,

$$k_{channel} = \frac{q\mu \, L}{\frac{\pi}{4} d^2 \Delta P} \left(\frac{P_{out}}{P_{mean}} \right) \qquad \text{(Equation 3)}$$

Combining equations 2 and 3, the permeability of a single channel can be expressed as:

$$k_{channel} = \frac{d^2}{32} \qquad \text{(Equation 4)}$$

If the diameter of the channels is very small compared to the diameter of the overall sample, then the effective permeability of the cylindrical standard can be expressed as:

$$k_{effective} \approx k_{matrix} + k_{channel} \frac{nd^2}{D^2} \qquad \text{(Equation 5)}$$

Combining equation 4 and 5, it can be shown that the effective permeability of the standard when the matrix is impermeable, is given by the equation 6,

$$k_{effective} = \frac{n\, d^4}{32\, D^2} \qquad \text{(Equation 6)}$$

The effective permeability of the cylindrical standard can thus be controlled and predicted by controlling the size and number of the channels. The effective permeability may also be altered by controlling the overall diameter of the cylinder, however; this is limited by the size of the core plug that the permeability measurement apparatus can handle.

In order to fabricate standards with permeability values similar to that of shale in shale gas formations, the inside diameter of the capillary or capillaries, d, is typically in the range of 0.005 to 0.05 millimeter (5-50 micron). The calibration standards can be placed in any apparatus used for measuring permeability that uses plug shaped samples. In our steady-state permeability measurements apparatus, the volumetric gas flow rate, q, and the pressure drop, ΔP, are measured in the same manner as during a permeability measurement on a core plug. The permeability of the calibration standard is calculated from Darcy's Law (American Petroleum Institute, 1998) for a compressible fluid as in equation 7,

$$k = \frac{q\, \mu\, L\, P_{out}}{A\, \Delta P\, P_{mean}} \qquad \text{(Equation 7)}$$

If equations 6 and 7 indicate the same permeability, within the stated accuracy of the permeability measurements apparatus, the apparatus is considered to be well-calibrated. Otherwise the cause of the discrepancy is investigated and the required changes are made to the apparatus.

The advantage of these calibration standards over the prior art discussed in earlier sections is that none of the commercially available products combine the characteristics that these standards offer:

- Flow resistance in the desired range similar to tight reservoirs (equivalent to 10 – 10,000 nanoDarcy core plugs)
- Cylindrical geometry required for use in a standard laboratory permeameter
- Flow resistance known from first principles

Required conditions for validity of Hagen-Poiseuille Equation and calibration standards

Laminar flow and Entrance effects

The Hagen-Poiseuille equation (Bird et al., 2002) adequately represents flow in a capillary tube when laminar flow exists in the capillary tube. Laminar flow occurs when the dimensionless Reynolds number, defined as $\mathrm{Re} = \frac{\rho\, d\, v}{\mu}$, is less than 2100. In addition, in order for the Hagen-Poiseuille equation to adequately represent flow in a capillary tube, the length of the capillary tube must be at least ten times the entrance length of the capillary tube. As gas enters a capillary, some distance is required for the flow pattern to stabilize (i.e., become "fully developed") and for the Hagen-Poiseuille equation to become valid. This distance is known as the entrance length and is on the order of $l_e = 0.035\, d\, \mathrm{Re}$.

In Figure 10, the shaded regions show the permeability and pressure conditions under which both of the above criteria are always met. The assumptions used in determining the shaded regions are shown in Table 1. One region is for full flow, in which the downstream end of the calibration standard is open to the atmosphere. The other is for restricted flow, in which a higher pressure is used downstream of the sample in order to maintain a specified pressure drop (100 psi in this case). Both are common methods used in permeability measurements; each has advantages and disadvantages depending on the permeability of the core and the pressure rating of the equipment.

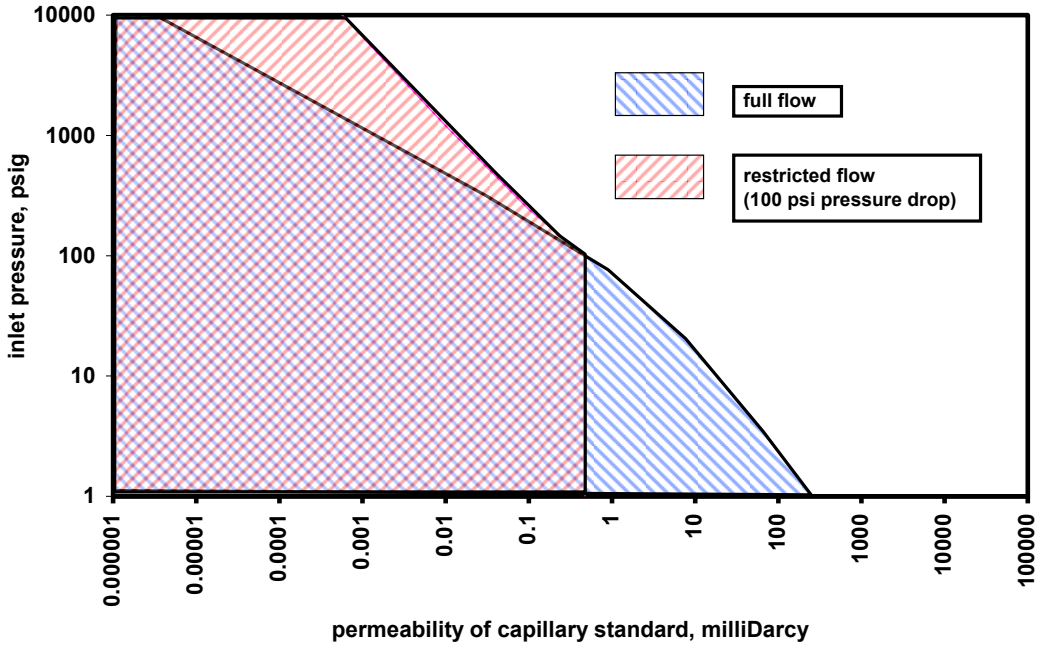

Figure 10. Permeability and pressure conditions for Hagen-Poiseuille equation

From Figure 10, it is apparent that calibration standards for low permeability (<0.01 milliDarcy) would fulfill the laminar flow and entrance effect criteria for a wide range of operating conditions. For permeability and pressure conditions not in the shaded regions of Figure 10, the criteria for the Hagen-Poiseuille equation may still be met, depending on details such as gas composition and number of capillary holes in the calibration standard.

Gas	nitrogen
Length of calibration standard	2 inches
Diameter of calibration standard	1½ inches
Number of capillaries	1
Downstream pressure	atmospheric (for full flow)
	100 psi below inlet pressure (for restricted flow)

Table 1. Assumptions used in determining shaded regions in Figure 10

Continuum assumption

Another criterion for the Hagen-Poiseuille equation to be valid is that the flow should be in the continuum regime, i.e.; the molecular mean free path (average distance covered by a gas molecule between successive collisions) of the fluid should be smaller than the physical length scale of the channel through which the fluid flows. The continuum assumption can be characterized by a Knudsen number (Kn), which is the ratio of the mean free path of the fluid to the characteristic length of the channel. If $Kn<1$, then the continuum assumption is valid. When $Kn>1$, then the fluid is in the transition or free molecular flow range and the transport mechanism may be diffusion or a combination of convection and diffusion. The smaller the characteristic length scale of the channel, the larger would be the Knudsen number. Our calibration standards are comprised of channels with internal diameter as small as 5 microns. The mean free path for helium gas at room conditions is 173.6 nanometers (Hirschfelder et al., 1954). The Knudsen number for helium gas while flowing through a 5 micron channel would be:

$$Kn = \frac{173.6 \times 10^{-3}}{5} = 0.035 << 1$$

Thus the continuum assumption is valid for all the channels that are 5 microns or larger, such as the ones we have used in our standards. The assumption would also be valid at pressures higher than atmospheric because the mean free path of the molecules decreases with increasing pressure.

Results

The fabricated calibration standards were tested in our steady-state apparatus and experimental results were compared with permeability values calculated from first principles using Equation 6. The design details of the standards are listed in Table 2.

Materials	stainless steel glass epoxy	
Length	1 inch	
Diameter of calibration standard	1½ inches	
Number of capillaries	1	
Hole diameter	1 millimeter	
Inside diameters and tolerances of capillaries along with expected permeability values based on Equation 6	5 ± 1 microns	13 nanoDarcy
	10 ± 1 microns	215 nanoDarcy
	26 ± 1 microns	9827 nanoDarcy
	47 ± 1 microns	104,829 nanoDarcy

Table 2. Design details of calibration standards

Six calibration plugs were constructed by drilling a hole along the length of a stainless steel cylinder and using epoxy to cement a precision-bore glass capillary in the hole. Previous attempts to directly drill a micron size hole through a solid metal cylinder of the size of a typical core plug was not successful because there are currently no commercially existing technologies that can drill such small holes with high precision, and at reasonable costs. Glass capillaries were used with internal diameters of 5, 10, 26 and 47 microns. Three standards, 10A, 10B, and 10C, were made using a 10 micron glass capillary; the other three plugs, sample 5, 26 and 47 were made using capillaries with internal diameters of 5, 26, and 47 microns respectively. The plugs were 1 inch in length and 1.5 inches in diameter in order to fit within the steady-state apparatus to measure permeability. As described earlier, compressed air was used to confirm that flow occurred only through the bore of the capillary, with no bypassing through the epoxy-filled annulus.

Figure 11 is a plot showing the comparison between measured permeability, and those computed using Equation 6. Note the excellent agreement between the calculated and measured permeability for most samples, which indicates that the standards behaved as expected, and the diameters of the capillary bores were within specifications. The error bars on calculated permeability in Figure 11 is computed using Equation 6, by accounting for the tolerance in the inside diameter of the capillaries, ± 1 micron; this was measured under a microscope, measuring several short segments of the glass capillary tubes, that were cut along the length of the capillaries. The larger discrepancy noted for sample 5 is still within specifications, given the large relative tolerance for the inside diameter (5 ± 1 micron, or ± 20%) and the fourth-power dependence of permeability on the internal diameter, as shown in Equation 6. The standards cover a wide range of permeability between 10 nanoDarcy and 0.1 milliDarcy. These standards can therefore be used in calibrating equipment for measuring permeability on very tight rocks without the need for empirical calibration of the standards and while ensuring that the equipment yields measurements that are closer to the “ground truth”.

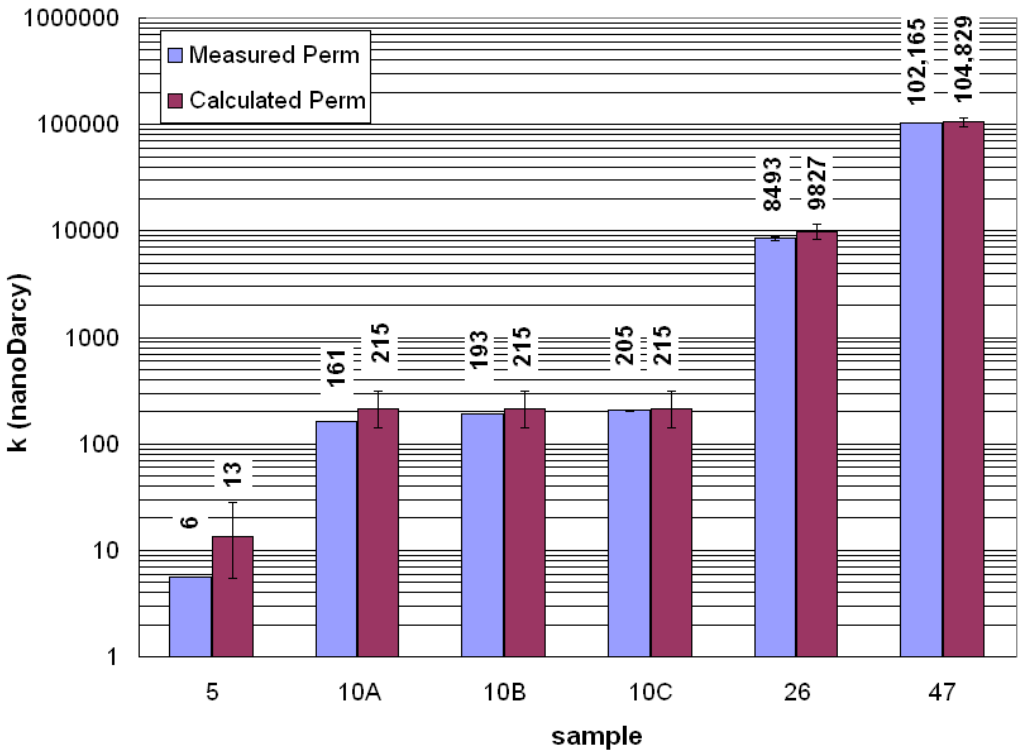

Figure 11. Measured and calculated permeability of six different permeability calibration standards

Summary and Conclusions

Current industry practices for measuring permeability on tight rocks such as shales using the GRI or crushed-rock technique is inadequate because of the lack in consistency in results reported by the different vendors and fundamental shortcomings of the crushed-rock method in providing a meaningful measurement. Plug based permeability measurements can be used to overcome the limitations of the crushed-rock techniques. We have developed a steady-state permeability apparatus for measuring permeability on tight rock samples, and initial results suggest that there is no systematic trend between steady-state permeability measurements on plug samples and results reported by vendors using crushed-rock techniques. Further investigation is required to confirm if a systematic trend exists between our measurements and those from the vendors offering crushed-rock permeability measurements.

Inconsistency between vendors utilizing similar methods to measure permeability suggests that a measurement standard with permeability in the same range as tight rocks is necessary to ensure that the equipment used in low permeability measurements is calibrated to certain "ground truth". We have designed and developed cylindrical calibration standards based on first principles, suitable for any equipment used to measure permeability on plug shaped samples. These standards cover a permeability range of 10 – 10,000 nanoDarcy, similar to that observed in most tight rock samples. Testing our steady-state equipment with these standards resulted in very good agreement with values calculated using physical laws for fluid flow, thus confirming that our equipment yields results that are close to "ground truth".

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Nomenclature

A	cross-sectional area (of capillary standard)
d	diameter (of crushed particle or capillary bore)
D	diameter (outside, of capillary standard)
k	permeability
$k_{effective}$	effective permeability (of a capillary standard)
$k_{channel}$	permeability of a capillary channel
k_{matrix}	permeability of matrix (surrounding capillary bores)
Kn	Knudsen number
l_e	entrance length (for fully developed flow)
L	length (of capillary standard)
n	number of capillary channels in capillary standard
P_{mean}	mean pressure, absolute (average of inlet and outlet pressures)
P_{out}	outlet pressure, absolute
q	volumetric flow rate
R	radius (inner, of capillary tube)
Re	Reynolds number
t	time
v	linear flow rate, $q/(\pi R^2)$
ΔP	pressure drop
μ	dynamic viscosity
ρ	density
φ	porosity

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