WORKFLOW FOR MEASUREMENT OF STEADY-STATE TO LIQUID PERMEABILITY IN NANO-DARCY RESERVOIR ROCK

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ABSTRACT

A workflow for the measurement of very low permeability unconventional reservoir rocks was developed that accounts for the presence of micro-cracks and fractures in the sample. A rapid low resolution micro-CT scan (~50 microns/voxel) identifies significant cracks and partings in 2.55 – 3.75 cm diameter core plugs that are used in the subsequent petrophysical study. In some instances, these pre-scans eliminate more the 90% of the samples from further testing. In instances of moderately fractured samples, small prisms of non-cracked rock are trimmed and then mounted in a cylindrical mold filled with epoxy. Low-field NMR measurements are used to determine initial liquid content in the core plugs. Initial liquid saturations range from almost 100% to less than 50% in liquid-hydrocarbon shale reservoirs and are strongly dependent upon the nature of the reservoir fluid compositions. In the case of as-received samples with low liquid saturations it is possible to measure a gas-filled pore volume with a standard porosimeter. A summation of fluids approach then is used to determine total porosity. The measurement of merit for permeability is a steady-state to oil flow experiment at elevated temperature and under confining pressure in a standard Hassler cell. The sample's pore volume is filled with a decane by pressure saturation for a period of time prior to loading into the flow cell. Permeability is measured by monitoring differential pressure across the core at two or three constant flow rates. The multi-rate approach allows for a graphical solution to the Darcy equation along with point-by-point values that are averaged over a period. These two approaches differ by less than 5% for good quality data. Flow rates for these sub-microDarcy rocks are in the range of 0.01 to 0.12 cm^3/hr and often these tests require several days to more than a week to complete. NMR measurements are collected on the post SSKo sample to ensure that the test did not change the liquid-filled pore volume or saturations. The technique is very reproducible when the samples remain intact during handling.

INTRODUCTION

There are several challenges in measuring flow properties in very fine-grain reservoir rock. The most notable is the presence of fractures and micro-scale cracks found in standard core plugs in many of these unconventional reservoirs. Early efforts to measure permeability on shale core plugs with the unsteady-state pressure decay method generated uneven results in many cases [1]. The Devonian Shale Gas program created a workflow that handled micro cracks in conventional core plugs by using instead smaller particles crushed to a uniform size. The intent of using smaller "chips" was to avoid micro-cracks that would affect the gas flow in a pressure-decay test. The belief was that crushing samples to a uniform particle sizes (e.g. 20-35 mesh, 500 to 850 microns) that breakage would occur along naturally occurring cracks and partings such that the particles themselves would be free of these cracks. This approach led to a somewhat standardized workflow used by a wide number of industry labs, often referred to as "Crushed Rock Analysis". More recent studies highlighted some of the difficulties in maintaining interlab calibrations and comparisons [2, 3]

A second challenge associated with petrophysical measurements of unconventional reservoir rocks is identifying and quantifying the various liquids found in the sample when it arrives at the laboratory. An ideal measurement of gas-filled pore volume and permeability to gas involves liquid-free pores. Standard core cleaning methods and water extraction techniques are hampered by the very low permeability of the sample, especially in a core plug [1]. Size reduction of the sample to uniform particles improves access to pores that should improve cleaning efficiency, though recent work indicates that saturation estimates from small particles are strongly biased and do not match log-based results [4].

Steady-state permeability to gas measurements in tight unconventional reservoirs are commonly used because of the shorter time requirements, though issues with fluid compressibility and adsorption remain a concern [1]. Steady-state permeability to liquid measurements are easier to interpret, but require longer time periods [5, 6]. The design of these tests is to monitor flow rates generated by constant pressure differences across the core plug. Changes in effective stress and observations of permeability creep with time are common observations.

The proposed workflow returns to the use of standard-sized core plugs and a steady-state to liquid flow test as the best means to measure permeability in these very low permeability rocks. The core plugs are scanned for evidence of micro-cracks that would impact the flow test, which results in many samples discarded from testing. NMR relaxation measurements are used to determine the volume of liquids in the sample at various stages of the testing. A standard steady-state permeability setup was modified slightly in order to adjust for the very low flow rates required to measure nano-Darcy level permeability.

PROCEDURE

Whether the core plug is prepared at wellsite or in the laboratory itself, the first step is to collect a rapid, low-resolution micro-CT image of the plug. The main advantages of micro-CT over other imaging modalities are its speed and resolution along with the ability to scan the sample while still wrapped in any preservation packaging. Images are collected with a Zeiss Versa 510 micro-CT running at 140 kV. Scan times for 58-micron resolution images on standard 2.54 cm diameter core plugs are 60 minutes or less. The use of an auto-sample loader allows for near 24-hour usage of the imager.

These low-resolution images are sufficient to identify micro-cracks and partings that extend along the length of the horizontal core plug. Many of these features are parallel to bedding and result from the stress release of the sample. Smaller cracks and especially those transverse to the long axis of the plug are less likely to affect a flow experiment. An empirical classification scheme based on number and length of these partings was developed to evaluate plug quality and their suitability for testing. While it may be possible to generate an automated classification tool for these images, current experience with these low-resolution images is that edge effects and uneven brightness that the eye overlooks hinder any image processing efforts.

Liquid volumes of the core plugs are measured with a low-field (0.045T) NMR spectrometer (GeoSpec2, Oxford Instruments) equipped with a Q-Sense probe. Standard Free Induction Decay (FID) and Spin-Spin Relaxation (T_2) measurements are acquired on the sample at various stages of the workflow, including in the "as-received" and after the steady-state permeability test. Changes in liquid volume are compared with mass changes in the sample at the various stages. The instrument is calibrated to a known volume standard with reproducibility of 0.19 cm^3 for a 16.7 cm^3 liquid volume, ~1.2% error. The T_2 measurement is acquired with a standard CPMG pulse sequence with an echo spacing of 0.1 msec, a recovery delay of 3 to 5 seconds depending upon the sample and accumulated over 256 scans. This normally leads to a signal-to-noise level between 50 and 100 as determined by the intensity of the first few echoes relative to an averaged baseline determined near the end of the echo train. Pulse widths and resonance frequencies are calibrated for each suite of samples. The raw data is processed with a standard non-negative least squares inversion algorithm with a constant regularization component that is optimized for the hardware (GITSystems Advanced, Green Imaging Technology).

Samples that have more than 50% of their pore volume filled with air/gas in the "as-received" state are placed in a gas porosimeter in order to measure pore volume. A sum-of-fluids approach uses the NMR-based liquid volumes and the gas porosimeter derived volume to determine a total pore volume and porosity. In some cases, after the as-received NMR measurement, samples are dried under mild conditions (vacuum, 80°C) in order to remove as much fluid as possible before the next step.

Regardless of the initial gas content in the as-received core plugs the remaining gas-filled pore volume is replaced by a light liquid hydrocarbon in a pressure-saturation process. The core plugs are placed in Nalgene bottles filled with decane and then placed in a larger pressure vessel. The hydrostatic pressure in the vessel is raised to 2500 psi and 150^{o}F and left there for 1 day. A programmed depressurization process over the ensuing 4 days minimizes breakage of the core plugs due to stress release. Early experience found that rapid depressurization after pressure saturation frequently caused plugs to break. A second set of NMR measurements are collected on these "pressure-saturated" samples and the measured total liquid-filled volume is compared to the sum-of-fluids values generated from the as-received liquid content and its gas-filled content. The pressure-saturation process is repeated when the volume comparison falls outside 10%.

The liquid-saturated core plugs are inserted into a standard steady-state permeability rig for measurement of the hydrocarbon flow (SSKo). The core holder is connected to a precision pump (Quizix 5000 series, Chandler Engineering) on the upstream end and a back-pressure regulator on the downstream. These pumps are capable of stable flow rates down to 0.001 cm^3/hr while monitoring pressure changes of 0.1 psi. Flow rates for nano-Darcy samples are set between 0.01 and 0.2 cm^3/hr with measured inlet pressures between 55 and 135 atm. The back-pressure regulator is set nominally to 34 atm during the test. Hydrostatic pressure applied to the Viton sleeve in the core holder is maintained by a second pump and set to 240 ± 0.07 atm. The core holder is wrapped in a heating mantle that maintains a stable 160^{o}F temperature for the duration of the experiment. Permeability is measured by monitoring the pressure changes at the inlet end of the core plug at constant flow rates and determining an average pressure differential along the length of the plug. Permeability is calculated with the Darcy equation at each data acquisition point, roughly 6 minutes apart, with the constant flow rate value, the measured pressure differential, and the decane viscosity at temperature. The flow rate is changed several times during the test so that sufficient points are available for a graphical solution to Darcy, including an origin point of zero differential pressure at zero flow rate. Each flow rate step requires several days for the pressure differential to stabilize within a tolerance of 3% of the absolute value. A complete permeability test therefore requires 7-14 days to complete. The effluent flow volumes are monitored to ensure mass balance and are sometimes captured for further analysis.

A final set of NMR measurements are collected after the core plug is retrieved from the permeability apparatus to monitor any changes in total liquid volume or saturations.

RESULTS

Samples were selected from a single well in an unconventional reservoir that produces oil. The samples were a fine-grain carbonate-rich rock with varying amounts of clay minerals (marls and argillaceous marls). The core plugs were preserved prior to sending to the special core analysis lab and were accompanied by the results of crushed-rock analysis on companion pieces. Gas-filled porosity of the as-received samples were measured with the crushed-rock analysis method and ranged from 0.02 to 0.12. Gas saturations were estimated to be between 40 and 80%, which suggested that much of the initial liquids were lost during the coring process.

Initial micro-CT scans on the "as-received" core plugs generated images that illustrated a wide range of plug quality (Figure 1). A few of the samples had no discernible cracks at the resolution of these quick-scan images, and were passed along directly to the permeability workflow. Many of these carbonate-rich samples were characterized by significant cracks or partings that extended the entire length or width of the original plug. These samples were candidates for trimming and epoxy potting in preparation for permeability testing.

The as-received samples had liquid-filled volumes that range from 0.5 to 2.5 cm^3. The gas-filled pore volumes of these samples were used to generate a sum-of-fluids total pore volume, which in turn was used to calculate a total porosity. These sum-of-fluids values compared favorably with the total liquid volumes measured by NMR on the pressure-saturated core plugs (Figure 2).

The T_2 relaxation time distributions of the as-received samples were often unimodal and skewed towards faster times in most cases, with a larger relaxation component centered around 1 msec and a second, smaller component around 10 msec or slower. The addition of decane in the pressure-saturation step contributed primarily to the slow component and developed a strong bimodal character (Figure 3). This particular carbonate-rich unconventional reservoir rock was characterized by relaxation behavior similar to many water-wet conventional reservoirs where irreducible water is associated with the faster relaxation components and the slower component represents the light hydrocarbons [7]. As in NMR analysis of conventional reservoirs, the population density of each major relaxation component was transformed into liquid saturation estimates. The addition of decane during the pressure-saturation step did not change the intensity of the fast relaxation component in most of these samples. Only the slow component is affected, which suggested that the added decane was located in larger pores or in water-wet pores, or some combination of both scenarios.

The NMR-based water saturation estimates were in general slightly greater than those generated from the standard Dean-Stark analysis (Figure 4). The data in Figure 4 was

produced from NMR and Dean-Stark extractions on the same pieces of core. The NMR-based estimates were slightly higher due in part to the T_2 distribution's inability to separate signals from water and residual oil or bitumen.

The steady-state permeability tests on selected samples showed well-behaved pressure-drop / flow rate behavior that could be analyzed with the Darcy equation (Figure 5). The monitored pressure differential usually stabilized within 12 hours of changing the flow rate and over a period of several days remained within 0.5 atm of the averaged value. A data analysis tool allowed for user input to select the time period over which the pressure differential was averaged at each flow rate. In some instances, pressure showed a slight decrease in time (i.e. increased permeability), but the opposite condition of permeability creep was not observed [6]. This increase in permeability is due to the dissolution of residual hydrocarbons from the core. The permeability value determined from an average of point-by-point calculated permeability differed by less than 3% from the value estimated from the graphical solution to Darcy in this suite of samples. The linear correlation R^2 of the graphical solution was usually 0.98 or better (Figure 6).

The SSKo results were different from crushed-rock analysis results in two ways (Figure 7). The first was that the total porosity measured in the SSKo workflow covered a narrow range of values compared to the CRA values on extracted samples. The second difference was that at similar range of total porosity the SSKo values were one to two orders of magnitude less than the CRA values. The reproducibility of the SSKo tests was determined by replicate measurements on several samples and found to be within 5% of the original value in the range of 20 to 1000 nD. The broad range of porosity values in the CRA values could result from a combination of incomplete extraction of residual liquids that affected the total gas volume measurement and a sampling bias. The latter could have occurred with SSKo sample selection that emphasized intact lithology.

The constant flow rate approach to measuring steady-state permeability in tight shale samples worked as well as the constant differential pressure approach [6]. Concerns of slower test equilibration with the constant flow rate approach were not a factor in this study, the monitored pressure differential reached steady-state within hours of changing flow rates.

This method for determining permeability in nano-Darcy rock is robust and reproducible. The permeability values provided by this method are relative to whatever residual water saturation is left in the sample and pre/post NMR T_2 measurements confirm that saturations remain constant. Because samples are pre-screened, this is a true measurement of matrix permeability. These types of measurements can define the ranges of permeability of regional lithologies. If higher-frequency permeability data is needed, this method can be used to correct values provided from other methods.

The principal advantages of measuring permeability at steady state to oil compared to other permeability measurements can be summarized as follows:

- There are fewer assumptions in the model (1D-Darcy) compared to the more complicated models used for crushed rock permeability and pulse decay permeability. The results are easier to interpret.
- There is no need to correct for slip or gas compressibility.
- Hot decane solubilizes residual hydrocarbon without use of solvent extraction which would damage the rock matrix.
- Very repeatable and robust measurement.

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FIGURES

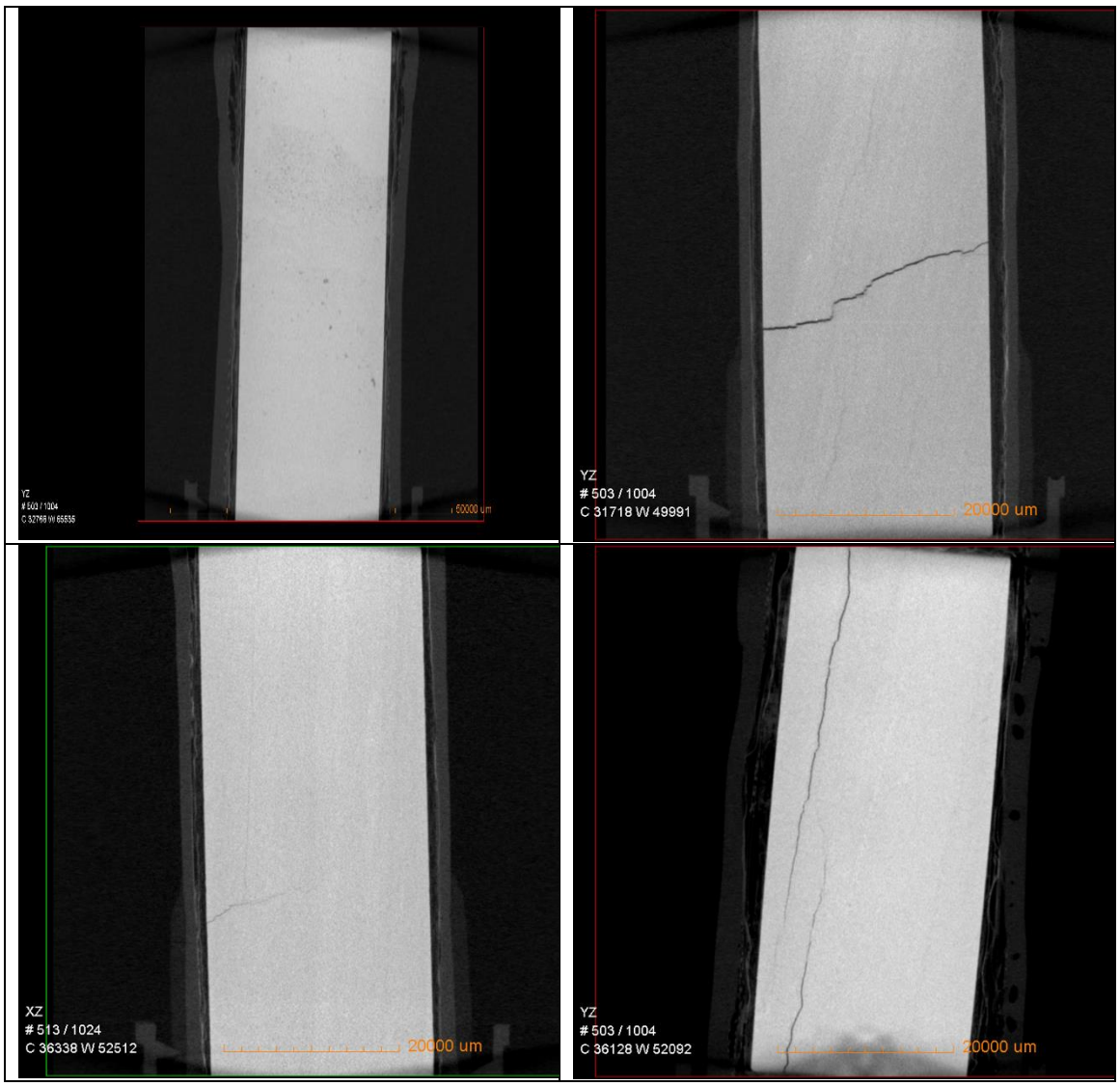

Figure 1. Micro-CT images of selected core plugs that illustrate the range of cracks and partings found in samples selected for permeability testing. Upper left illustrates a high-quality core plug with no visible cracks or partings that is ready for SSKo testing. Lower left shows a sample with a faint transverse crack that could be used in SSKo rig. Upper right image shows a large transverse crack that limits the use of this plug. A trimmed whole diameter shortened plug from either half could be used. Lower right image illustrates multiple longitudinal cracks and partings that make this plug unsuitable for testing. A reduced diameter prism can be trimmed from this piece and epoxy-potted for testing.

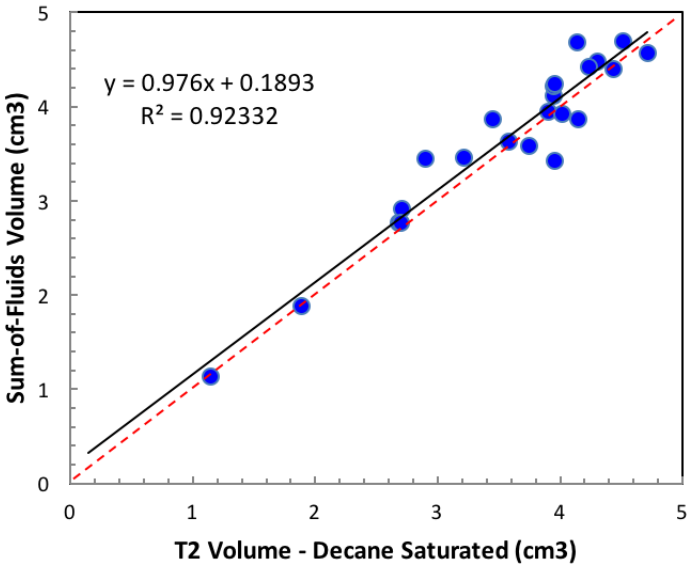

Figure 2. Comparison of total pore volume from NMR on pressure-saturated samples and sum-of-fluids volumes measured on "as-received" samples. The dashed line is unity and the solid line is the actual data trend.

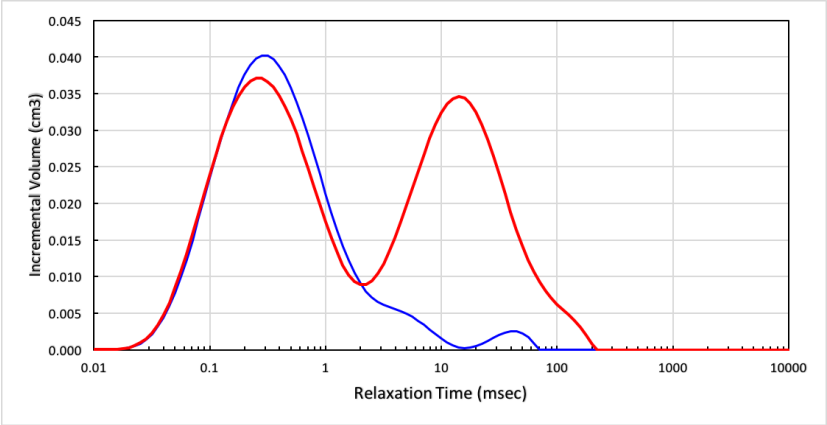

Figure 3. T_2 relaxation time distributions for as received sample (blue) and after pressure-saturation with decane (red). The increase in the slow component with the addition of decane supports the basic interpretation model of water as fast relaxation and mobile oil captured in the slow component.

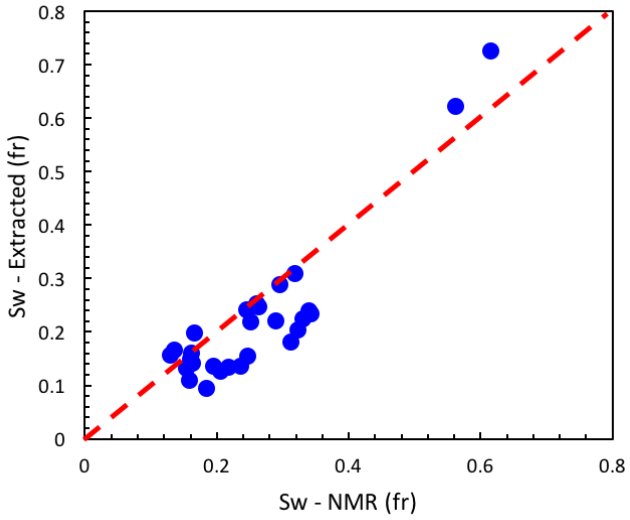

Figure 4. Comparison of crushed-rock-method water saturation versus NMR-based results from pressure-saturated samples. The water saturation values for the crushed rock samples were determined from Dean-Stark extraction of small chips

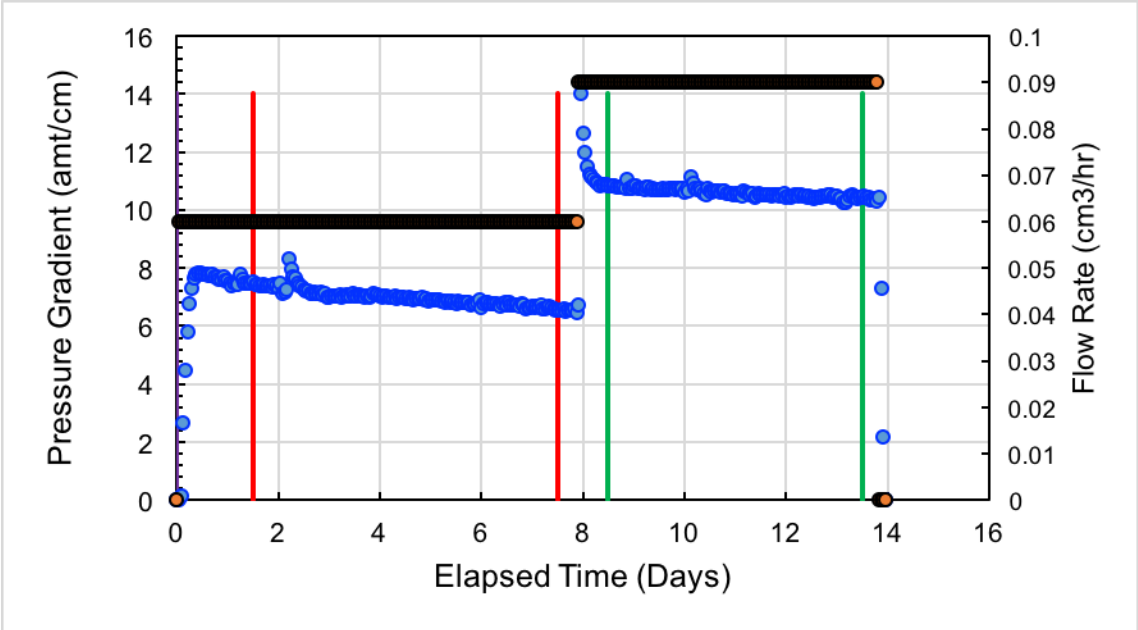

Figure 5. Example of steady-state permeability data where the pressure gradient (blue) was monitored for two flow rates (black) over a period of several days. Gradient values were averaged between the vertical lines for each flow rate for graphical solution. Averaging the gradient values provided better representation of the pressure drop during the steady-state regime.

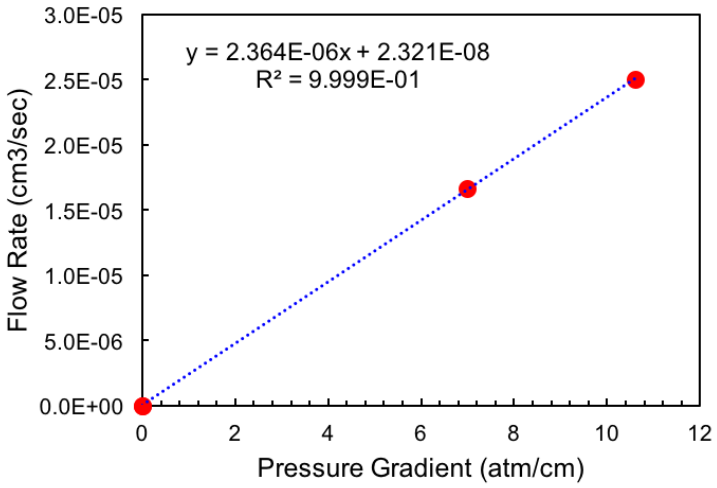

Figure 6. Example of graphical solution of Darcy's equation for permeability using data from Figure 5. Solution includes the use of the zero flow rate, zero pressure gradient origin value.

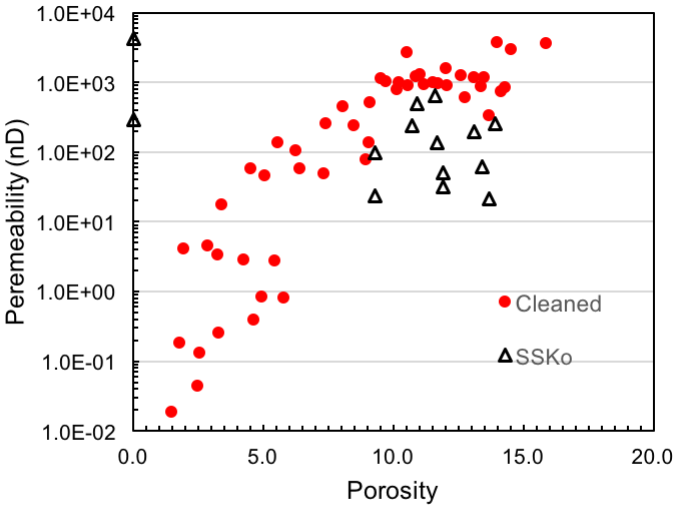

Figure 7. Comparison of steady-state permeability results with previous crushed-rock analysis based values. Porosity used with SSKo-based values is a total porosity value, comparable to the "cleaned" value determined from crushed rock measurements.