



Full Length Article

Permeability standards for tight rocks: Design, manufacture and validation



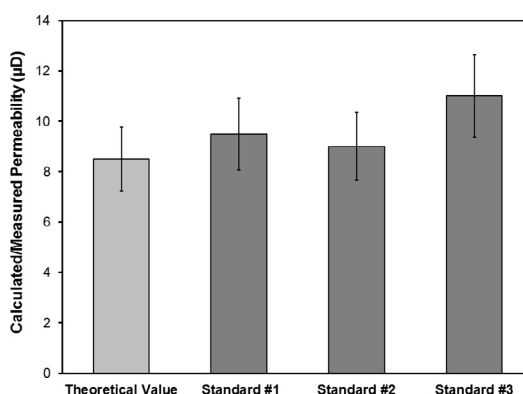
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HIGHLIGHTS

- Design and manufacture permeability standards with multiple manufacturing techniques.
- Compare and document the advantages/disadvantages of each manufacturing technique.
- Verify the calibration of the permeability standards by performing steady-state gas flow tests.
- Identify non-Darcy gas flow regimes at varying (mean) pressures.
- Analyze the repeatability and uncertainty of the permeability data generated by the standards.

GRAPHICAL ABSTRACT



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ABSTRACT

Permeability values measured for low-permeability (tight) rocks by commercial and research laboratories may exhibit an unacceptably large variability even when measured using samples subjected to similar experimental conditions. One cause of this variability is that, for a particular measurement method, laboratories utilize different experimental configurations with instrumental components that have variable manufacturing specifications. These device-to-device variations, and consequent wide ranges in measured permeability values, have created confusion for recipients of the data. Regardless of the cause of the observed discrepancies, there are currently no commercial standards available to evaluate the performance and accuracy of these different experimental devices and calibrate them to an “absolute truth”.

In this work, the design, manufacture and calibration of standards for gas permeability measurements of tight rocks (with permeabilities down to the microdarcy range) is discussed. Three identical permeability standards were manufactured and tested in this study. These permeability standards were developed by embedding one micron-sized capillary tube (ID: $25 \pm 1 \mu\text{m}$; OD: $1/16''$ (1.588 mm)) inside an impermeable acrylic cylindrical core plug with the dimensions of 1.5" (38.1 mm) in diameter and 2" (50.8 mm) in length, which is consistent with what is used in commercial permeability measurement devices.

For the manufactured permeability standards, it is evident that (1) there is satisfactory agreement ($< \pm 10\%$) between calculated and measured gas (nitrogen) permeability values (within the experimental error range), (2) the gas permeability values measured for these three different standards are very similar, even though they were manufactured separately, and tested independently at different times, and (3) the repeatability/reproducibility of the measured gas (nitrogen) permeability values is excellent ($< \pm 1\%$). A further observation is that, depending on mean pressure (gas flow rates), flow regimes within the

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permeability standards (microcapillary tubes) may correspond to gas slip or turbulent flow regimes, affecting the measured (apparent) gas permeability values.

Using these new permeability standards, it is possible to calibrate permeability devices used by different commercial/research laboratories ensuring accurate steady-state gas permeability measurements for tight rocks with permeabilities down to the microdarcy range.

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1. Introduction

Some unconventional reservoirs have ultra-low permeability values (due to narrow nano-scale pore throat sizes), making permeability difficult to measure in the laboratory with high accuracy and precision. The application and reliability of steady-state and pulse-decay permeability techniques are well-established for tight rocks. Using suitable experimental devices/configurations, these techniques are capable of measuring low to very low permeability values (down to nanodarcy range) over a reasonable period of time [1]. Nevertheless, there are currently no commercial standards available for calibration of steady-state and pulse-decay permeability devices. The availability of permeability standards is particularly crucial for tight rocks for two reasons: (1) there is currently an inconsistency between permeability values reported by different commercial/research laboratories using steady-state and pulse-decay permeability techniques even using identical samples subjected to similar experimental conditions [2–4] – using permeability standards, it is possible to ensure that different permeability devices used by different commercial/research laboratories are calibrated to the same “absolute truth”, and (2) to determine the accuracy of permeability measurements, it is necessary to verify the performance of permeability devices with standards for which the permeability can be computed from basic fluid flow principles [2,3].

This work reports on the design and manufacture of standards for measurement of gas permeability in tight rocks. The primary objectives were to (1) design and manufacture standards with multiple innovative manufacturing techniques, (2) compare and document the advantages and disadvantages of each design and manufacturing technique, (3) verify the calibration of the manufactured permeability standards by performing steady-state gas flow tests under various experimental (mean pressure¹) conditions, (4) analyze the repeatability and uncertainty of the permeability data generated by the standards and (5) identify/characterize non-Darcy gas flow regimes (slip flow, turbulent flow) that may be observed under different experimental (mean pressure) conditions.

2. Materials, design and manufacture

Multiple materials, innovative designs and manufacturing techniques were examined before arriving at the optimum design for the standards manufactured/used in this work. The details of these multiple trials and their advantages/disadvantages are summarized in Appendix 1. The schematic and photographs of the optimally-designed permeability standards are shown in Fig. 1.

The permeability standards are created by embedding one micron-sized capillary tube (ID: $25 \pm 1 \mu\text{m}$; OD: $1/16''$ (1.588 mm)) inside an impermeable acrylic cylindrical core plug (1.5" (38.1 mm) in diameter, 2" (50.8 mm) in length). The selection of acrylic for this purpose is based on its favorable mechanical and thermal properties [5]. Acrylic is also a transparent material, providing the opportunity for visual inspection of the interior part of the standard. This is particularly desirable for multi-phase flow

to look at fluid distributions if transparent microcapillary tubes are used as fluid conduits. Fused silica microcapillary tubes sheathed in polyether ether ketone (PEEK) polymer (PEEKsil™) with a nominal diameter of $25 \pm 1 \mu\text{m}$ were employed in this study. PEEKsil™ tubing is mechanically strong and has compatible (chemical) characteristics for sealing with polymer fittings (www.thermofisher.com). PEEKsil™ is also expected to have extremely low absorption characteristics towards fluids (www.thermofisher.com). A commercial epoxy (EPO-TEK 301™; Epoxy Technology Inc.) was used as the cement/glue for embedding the microcapillary tubes inside the larger conduit ($1/16'' < d_{\text{hole}} < 3/8''$; $1.588 \text{ mm} < d_{\text{hole}} < 9.52 \text{ mm}$) drilled within the acrylic core plugs. Following this approach, three identical permeability standards were manufactured in this study (permeability standards #1, #2, #3). The dimensions of the permeability standards are similar to those of rock core plugs commonly used in steady-state and pulse-decay permeability measurement devices.

3. Theory

3.1. Laminar gas flow regimes in permeability standards

Permeability of the standards can be calculated using the fundamental fluid flow equations; i.e. the Hagen-Poiseuille and Darcy equations. Assuming negligible gravity effects, the laminar steady-state flow of a liquid phase through a microcapillary tube of constant circular cross section can be described by the Hagen-Poiseuille equation for incompressible flow [6,7]:

$$m^o = \frac{\pi r^4 (P_{\text{up}} - P_{\text{down}}) \rho_l}{8 \mu L} \quad (1)$$

$$Q = \frac{\pi r^4 (P_{\text{up}} - P_{\text{down}})}{8 \mu L} \quad (2)$$

where m^o is the (liquid) mass flow rate, Q is the (liquid) volumetric flow rate, r is the radius of the microcapillary tube, μ is the (liquid) viscosity, L is the length of the microcapillary tube, ρ_l is the (liquid) density, and P_{up} and P_{down} are pressures at the upstream and downstream ends of the microcapillary tube, respectively. For a gaseous phase, the laminar steady-state flow through a microcapillary tube of constant circular cross section can be described by the Hagen-Poiseuille equation for compressible flow [8]:

$$m^o = \frac{\pi (P_{\text{up}} - P_{\text{down}}) r^4 \rho_{\text{avg}}}{8 \mu L} \left[1 - (0.03\alpha) \frac{\varepsilon \beta}{1 - \frac{\varepsilon}{2}} \right] \quad (3)$$

where ρ_{avg} is the average gas density at the upstream and downstream ends of the microcapillary tube. The parameters α , β and ε in Eq. (3) are defined as:

$$\alpha = \frac{r^3 \rho_{\text{up}} (P_{\text{up}} - P_{\text{down}})}{L \mu^2} \quad (4)$$

$$\beta = \frac{r}{L} \quad (5)$$

¹ Mean pressure is defined as the average pressure between the upstream and downstream ends of the core plug.

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