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DIRECT POROSITY MEASUREMENT USING SINGLE CHAMBER GAS PYCNOMETERS

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Abstract: During porosity measurements, error most certainly originates from the incorrect determination of the samples total geometrical volume. This uncertainty will affect all porosity measurement procedures with greater degree on the indirect methods. However, there is a solution where the effect of the sample volume can be eliminated. If both the matrix and pore volume of the sample is measured the porosity can be calculated using those values instead of the bulk and pore volumes. The two-chamber gas pycnometer is the most used method for determining the matrix volume. The procedure applies Boyle's principle which can also be used to determine the pore volume directly using a single chamber construction, if the difficulties discussed in this article are solved.

Keywords: *porosity, reservoir, petrophysics, pycnometer*

1. INTRODUCTION

Porosity is considered one of the most basic yet most important petrophysical parameter of hydrocarbon reservoirs, as it describes the ratio of the void volume to the total volume of the porous rock (Dotson, Slobod, McCreery, & Spurlock, 1951). Thus, describing the volume fraction where fluids could accumulate in the rock. Numerous procedures were developed throughout the years (Lawrence & David, 2015) for the estimation/measurement of porosity from which the most reliable ones are those of laboratory measurements performed on samples taken from the reservoir.

Some authors categorize these measurements into two groups namely direct and indirect porosity methods. From which those that measure the pore volume are the direct and the ones measuring the matrix volume or true solid volume are the indirect methods. As mentioned previously the total volume, also referred to as the bulk or geometrical volume of the sample, consists of two parts. The void volume where no material is present and the matrix where the material mass is located. Porosity therefore can be calculated if either of two of the three volumes is determined.

In result at least two individual procedures are required to specify the porosity of a given material. Most laboratories measure the petrophysical properties such as permeability, relative permeability, rock mechanics… etc. on cylindrical shaped sample plugs. Therefore, in convenience the total volume is measured with simply digital calipers while a volume measurement method is used for either the matrix or the pore volume of the sample (Anovitz & Cole, 2015). In most laboratories for this purpose two chamber gas pycnometers (API, 1998) are applied due to their accuracy (~0,01% for all sample volume range) and the simple fact that there is no risk of damaging the sample in any means which is truly important for sample integrity. However, traditional gas pycnometers measure the true material volume of the sample rather than its pore volume.

In result one must ask how reliable and accurate the porosity of the sample will be the manually measured total volume of the sample is used in both the nominator and denominator of the equation. One may ask, ff this uncertainty is known to exist why no other total volume measurement are used instead?

Although in the industry several well-established measurement methods exist both for pore as well as matrix volume. On the other hand, for total volume measurement this unfortunately is not as simple. In the past the total volume of the samples was measured using the nonwetting properties of mercury with either a Westman Balance applying Archimedes' law or a pycnometer (Luffel & Howard, 1988). But due to the toxic nature of the material and the technical difficulties regarding these methods, total volume measurements of such forms are not very popular nowadays in the petroleum industry.

Other practices such as imbibition or buoyancy methods could be combined with two chamber gas pycnometers for porosity determination; however, the accuracy of these methods is nowhere near that of a gas pycnometer. And would not present better results than the manual measurements done with digital calipers. Also, in the case of strong oil wet rocks and unconventional rock materials are not very applicable due to the high pressures necessary for the saturation to reach and stay at 100%.

On the other hand, the other two pore volume measurement methods, namely gas adsorption and mercury intrusion porosimeters (Jimmy, Reza, Tobias, & Mohammad, 2020) would certainly have the necessary accuracy to be implemented with gas pycnometers for porosity measurement. Also, the above-mentioned pore size and wetting properties would also not be a concern due to the nature of the procedures. Unfortunately, due to the presence of another problem, these methods are also not the perfect solution.

For gas absorption and mercury injection porosimeters a usual sample size is somewhere between 0,5–1,5 cm^3 while an average sample plug (for example 1.5" diameter) has a total volume between 60–80 cm^3 depending on the final length after cutting. In most laboratories, however, the gas- and mercury intrusion pycnometers are the most frequently used instruments for porosity measurement. In these cases, normally the porosity of the sample plug is determined by caliper and gas pycnometer and the mercury pycnometer serves as an additional method for pore volume measurement as well as serving other important properties (pore size distribution, sub surface area, grainsize distribution…etc.).

In some cases, the porosity of the small samples is considered to that corresponding to the sample plug since the small samples are produced from the access cut material of those. If so, the small samples porosity can be determined at a high accuracy since mercury intrusion porosimetry measures the true pore volume of the sample and for the matrix volume a traditional gas pycnometer can be used.

For samples of homogenous nature this principal could be valid but natural rock formations could never be considered as such. Many publications and self-experiences show that is not as simple and this is never truly the case. This causes many petrophysical laboratories to not bother with porosity determination of the small samples and such measurements are primarily used for additional information.

Another solution for structural presentation hence volume determination is using NMR, unfortunately these types of equipment's are not very common due to their high cost and also limitations of sample volume. Despite their higher resolution and additional information on the structure of the pore system the implementation of mercury porosimetry is more approved.

In result during the research, it was clear that the porosity of a sample plug could only be measured with high accuracy if some other pore volume measuring method which not yet been mentioned could be used in combination of the traditional gas pycnometer.

In some literature an altered configuration of the gas pycnometer was presented (API, 1998) where the pore volume is measured rather than the matrix one using a Hassler type core holder (Rajib, Madland, Fred, & Aksel, 2012). One must then ask why this method is not used in most cases. The single chamber gas pycnometer has several difficulties which need to be addressed to perform the measurement which hindered its spread in the industry. These difficulties and their solutions will be covered in detail.

2. MATERIALS AND METHODS

2.1. Effect of total volume error on porosity

As shown in *Equation 1* during porosity (ϕ) calculation there are three individual volumes of the porous sample namely the pore (V_P), the true solid (V_S) and the total also referred to as apparent volume (V_T). As mentioned previously if either two of the three are measured porosity can be calculated.

$$\phi = \frac{V_P}{V_T} = \frac{V_T - V_S}{V_T} \tag{1}$$

Knowing that during porosity measurements the highest error always comes from the uncertainty of the total volume, its effect can be clearly seen to be more dominant in indirect methods. Unfortunately, during the sample preparation process total symmetry cannot be achieved hence the caliper method is not always reliable enough. Another solution is the Westman balance where the buoyant force required to totally submerge the sample into mercury is determined which if done correctly could give good results but is only used for irregular shape samples due to its complexity and potential high error. Also, for rocks with vuggy secondary porosity the removal of mercury on itself is a huge problem.

Let us give a simple example of how this error effects porosity. Let us consider a theoretical sample of exactly 100 cm^3 of total volume where the total void volume is 25 cm^3 resulting in 25% porosity.

To observe how the uncertainty of the geometric volume affects the value of porosity let’s assume a positive and negative 5% error *(Table 1)* while using the correct pore and matrix volumes.

Table 1

The divergence in porosity due to the error of the total volume

<table>
<tr><th colspan="2">Correctly measured values</th><th>V_{T_actual}</th><th>100 cm^3</th></tr>
<tr><td colspan="2">V_{P_actual} 25 cm^3
V_{S_actual} 75 cm^3</td><td colspan="2">$\phi_{actual} = 0,2500$</td></tr>
<tr><th colspan="4">Presenting 5% Error into the measurement of the total volume</th></tr>
<tr><td>$V_{T(-5\%)}$</td><td>95 cm^3</td><td>$V_{T(+5\%)}$</td><td>105 cm^3</td></tr>
<tr><td>$\phi_{calculated}$ from V_{P_actual}
$\phi_{calculated}$ from V_{S_actual}</td><td>0.2632
0.2105</td><td>$\phi_{calculated}$ from V_{P_actual}
$\phi_{calculated}$ from V_{S_actual}</td><td>0.2381
0.2857</td></tr>
<tr><th colspan="2">Error of porosity at $V_{T(-5\%)}$</th><th colspan="2">Error of porosity at $V_{T(+5\%)}$</th></tr>
<tr><td>From $V_{P\_calculated}$
From $V_{S\_calculated}$</td><td>0.0526
–0.1579</td><td>From $V_{P\_calculated}$
From $V_{S\_calculated}$</td><td>–0.0476
0.1429</td></tr>
</table>

During direct porosity measurements the error of the geometry appears with the same weight in the calculated porosity. Unfortunately, this cannot be said for the indirect methods where the total volume of the sample is used in both the nominator and in the dominator of the equation.

Therefore, the most reliable solution is to measure the true solid and the pore volumes directly and calculate the total volume as the sum of these values. For which in most petrophysical laboratories the gas pycnometer and the mercury intrusion pycnometer are used.

2.2. The discussion of the existing methods for matrix and pore volume determination

As mentioned previously in the industry the two most accepted procedures for porosity determination are the gas- and mercury intrusion pycnometers from which the previous is a direct while the latter is an indirect method. Both methods are favored over each other for different reasons of which the most important factors are addressed in *Table 2*.

If the two methods are taken into consideration the only positive regarding the mercury pycnometer is the fact that it provides much more information on the samples pore structure above the total porosity. Such as pore size distribution which gives a good picture of how the reserve will perform during production. The main problem however is that the mercury samples are much smaller than the sample plugs which are used for all other petrophysical measurements. For this cause the results of this method are not suggested by us to be used with other data of the sample plug for porosity calculation purposes.

Table 2

Pros and cons of the different pycnometers

Point of interest	Gas Pycnometer	Mercury intrusion pycnometer
Maximum sample size	Approximately 85 cm^3	Approximately 1 cm^3
Measurement time	From 15 minutes to several hours	Approximately 1 hour
Additional information	No additional information from measurement	Pore size distribution, grainsize distribution, Surface area, and more
Pollution of the sample	No effect	After measurement sample is unusable for other procedures
Health concerns	No effect	Highly toxic material and waste

The question can be asked is it possible to measure the cylindrical samples pore volume directly with any existing method? Before an answer could be given, the method for matrix volume measurement first must be discussed in detail.

The apparatus presented in *Figure 1* is the most used gas pycnometer in the industry. The measurement uses Boyle's law which states that at isothermal condition the new volume (at the new pressure) of a known quantity of gas can be determined if its previous volume at given pressure is known. The procedure consists of three main phases, first the air from the device is evacuated by flushing the measuring gas through the system, after which the gas outlet valve and chamber separation valves are closed, and gas is pressurized in the reference chamber (V_R). After constant reference pressure is reached the separation valve is opened and gas is expanded to the other chamber where the sample is located (V_C). The second stable pressure after expansion is recorded and the true material volume present in the sample holder chamber can be calculated.

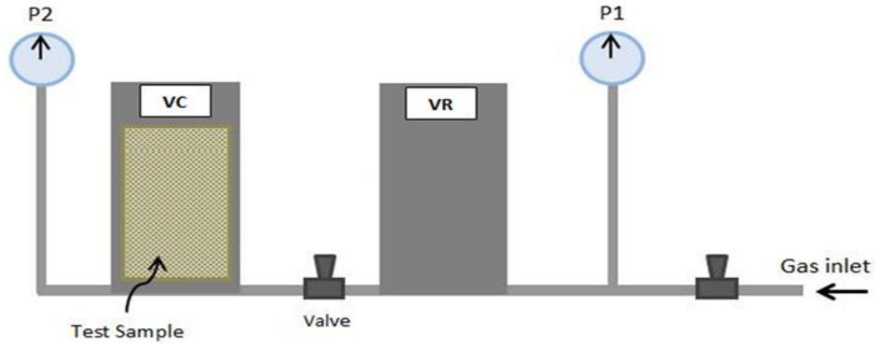

Figure 1

Schematic figure of a two-chamber gas pycnometer

The calculation procedure starts with writing up the equation of states for the three phases. First the equation of state for the gas (for quantity n_a) present in the sample holder chamber at ambient pressure (p_a) after the ventilation phase can be written up as presented in *Equation 2*.

$$p_a(V_C - V_S) = n_a R_u T \tag{2}$$

Next *Equation 3* presents the state of the given quantity of gas (n_1) after closing the two valves and filling the reference chamber to the desired reference pressure (p_1).

$$p_1 V_R = n_1 R_u T \tag{3}$$

Equation 4 will present the last phase where the total amount of gas inside the inner volume at p_2 pressure after expansion.

$$p_2(V_C - V_S + V_R) = n_a R_u T + n_1 R_u T \tag{4}$$

The previous three equations can then be combined into one *(Equation 5)* from which after some rearrangements made *(Equations 6–9) Equation 10* gives the main result of the procedure.

$$p_2(V_C - V_S + V_R) = p_a(V_C - V_S) + p_1 V_R \tag{5}$$

$$p_2(V_C - V_S) + p_2 V_R = p_a(V_C - V_S) + p_1 V_R \tag{6}$$

$$p_2(V_C - V_S) - p_a(V_C - V_S) = p_1 V_R - p_2 V_R \tag{7}$$

$$(p_2 - p_a)(V_C - V_S) = (p_1 - p_2) V_R \tag{8}$$

$$(V_C - V_S) = \frac{(p_1 - p_2)}{(p_2 - p_a)} V_R \tag{9}$$

$$V_S = V_C - V_R \frac{(p_1 - p_2)}{(p_2 - p_a)} \tag{10}$$

Until *Equation 10* all formulas used absolute pressures but with the introduction of gauge pressures (p_{g1}, p_{g2}, psig) the value of the barometric pressure could be taken out of the calculation *(Equations 11–12)*.

$$V_S = V_C - V_R \frac{(p_{g1} + p_a) - (p_{g2} + p_a)}{(p_{g2} + p_a) - p_a} \tag{11}$$

$$V_S = V_C - V_R \frac{p_{g1} - p_{g2}}{p_{g2}} \tag{12}$$

$$V_S = V_C - V_R \left[\left(\frac{p_{g1}}{p_{g2}} \right) - 1 \right] \tag{13}$$

The calculation of the true solid volume inside the apparatus can be made using *Equation 13* in knowledge of the two-gauge pressures before and after the expansion and the volumes of the two chambers with the additional volumes of the connecting pipes and valves.

Now to answer the previous question the measurement of the pore volume of the sample in such equipment is also possible but only if there is no second chamber and gas is expanded from the reference volume to the pore volume itself. In this scenario the pressure drop will be caused by the additional volume of the effective pore space of the sample plug.

The concept of such measurement devices is not new and is already described in the API 40 standard. Unfortunately, this method had not become a staple procedure regarding some problematic nature of it discussed in detail in the following chapter.

However, in combination of the traditional two chamber and the single chamber gas pycnometer both the true solid and pore volume of the sample can be measured. In result porosity could be calculated eliminating the uncertainties coming from the determination of the total geometric volume. However, this is easier said than done due to the difficulties of this kind of procedure.

2.3. The difficulties of the single chamber gas pycnometer setup

As mentioned previously, the pore volume could directly be measured by gas pynometer if a so-called single chamber equipment is used. In this solution the sample must be put in a sample holder where no volume is present around the sample. With this solution, after the separation valve is opened, the gas can only expand to the additional volume inside the core holder which at sufficient seal is equal the pore volume of the sample. To ensure the correct seal the following two criteria must be met. First the inner holder's length must be adjustable to correctly set to the length of the sample. The second is the volume around the sample must be eliminated. Such core holders exist in the petroleum industry for other petrophysical measurements called Hassler type core holders.

These core holders are used during permeability measurement where fluids are pumped through the cross section of the samples. For this purpose, a rubber sleeve is used which goes around the sample and is squeezed onto the surface of the sample using a hydraulic pump. Eliminating the free space around the sample completely. If a bypass channel is used the end plugs could be driven in the direction of the sample as pressure is raised to assure a perfect seal as in the case of a triaxial chamber. In addition, the distilled water used for the sleeve pressure could also be used as a thermostat to ensure the constant temperature throughout the measurement, which is critical.

The remaining two ports of the Hassler type core holders which originally serve as the inflow and out-flow during permeability measurements could be used for filling and evacuating the cell of the measurement gas. The previously described core holder *(Figure 2)* could be used with great results in the single chamber gas pycnometer setup.

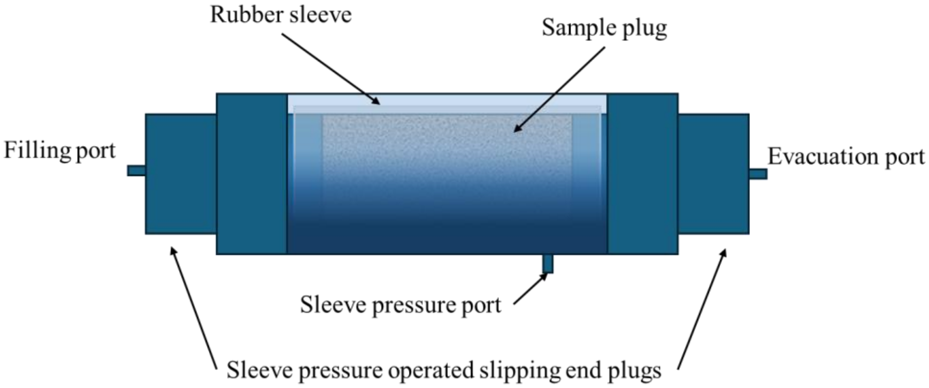

Figure 2
Schematic figure of a Hassler type core holder

With no additional volume in the sample holder during the expansion phase of the measurement the only volume increment causing the pressure drop is the effective pore volume of the sample itself. In result the new formulas for each phase could be derived from *Equations 2–4* as shown by *Equations 14–16*.

$$p_a V_P = n_a R_U T \tag{14}$$

$$p_1 V_R = n_1 R_U T \tag{15}$$

$$p_2 (V_R + V_P) = n_a R_U T + n_1 R_U T \tag{16}$$

After a similar procedure discussed previously the final equation can be written up where the pore volume can easily be calculated in knowledge of the two-gauge pressures in knowledge of the reference volume.

$$V_P = V_R \left[\left(\frac{p_{g1}}{p_{g2}} \right) - 1 \right] \tag{17}$$

Besides the problem of the sealing of the sample there is another major problem that must be solved the accurate reference volume has to be used regarding the measured pore volume of the sample. In the two-chamber configuration multiple reference chambers and measurement chambers are present in function of the sample size. During the procedure there is a tolerance in the size difference between the reference chamber and the free volume in the measurement chamber which if not met causes incorrect results.

Let's consider the following two scenarios. First, the reference volume is significantly larger than the pore volume. In this case the constant p_1 after expansion could drop such slightly that it could not be detected correctly. In the second scenario the reference volume is smaller than the measured pore volume to such an extent that after the expansion the pressure gauge could show error. These unfortunate cases could be eliminated if the reference volume is correctly sized for which the following procedure could be used.

Table 3

Geometry and volume data of sandstone samples

Sample_ID	diameter, [cm]	length, [cm]	V_T, [cm^3]	$V_{S\_measured}$, [cm^3]	$V_{P\_calculated}$, [cm^3]	ϕ_{He}, [%]
S_01	3.7860	6.1760	69.5278	57.2770	12.2508	17.6200
S_02	3.7960	6.9620	78.7909	61.3480	17.4430	22.1383
S_03	3.7900	6.1820	69.7425	54.6781	15.0644	21.6000
S_04	3.7750	6.9680	77.9887	53.3071	24.6816	31.6477
S_05	3.7860	6.9560	78.3088	52.5987	25.7101	32.8317
S_06	3.7900	6.1250	69.0994	53.5590	15.5405	22.4900
S_07	3.7920	6.1740	69.7257	51.8620	17.8637	25.6200
S_08	3.7900	6.1450	69.3250	50.1081	19.2169	27.7200
S_09	3.7900	6.1790	69.7086	58.2485	11.4601	16.4400
S_10	3.7690	7.0150	78.2654	66.3506	11.9148	15.2236
S_11	3.7740	6.9640	77.9027	58.3604	19.5423	25.0855
S_12	3.7560	7.0110	77.6821	57.3515	20.3306	26.1715
S_13	3.7710	6.9620	77.7565	59.2761	18.4804	23.7671
S_14	3.7900	6.1680	69.5845	55.2779	14.3066	20.5600
S_15	3.7720	6.9840	78.0436	59.9882	18.0555	23.1351
S_16	3.7850	6.9810	78.5488	57.7826	20.7662	26.4373
S_17	3.7830	7.0970	79.7696	54.3739	25.3957	31.8363
S_18	3.7900	6.1370	69.2348	53.4354	15.7994	22.8200
S_19	3.7890	6.9860	78.7712	57.2837	21.4875	27.2784
S_20	3.7750	6.9700	78.0111	52.6042	25.4069	32.5683
S_21	3.7750	6.9700	78.0111	53.9864	24.0248	30.7966
S_22	3.7900	6.1830	69.7537	53.2221	16.5316	23.7000
S_23	3.7880	6.9540	78.3690	53.8532	24.5159	31.2826
S_24	3.7900	6.1530	69.4153	51.3881	18.0272	25.9700
S_25	3.7900	6.1660	69.5620	50.8359	18.7261	26.9200
S_26	3.7710	6.9630	77.7677	58.1860	19.5817	25.1797

First the pore volume range of the samples that will be measured in the future must be determined. In *Table 3* the geometric and porosity data of 26 sandstone samples of Hungarian origin are presented. The dimensions of the cylindrical samples are 1.5” in diameter and approximately 6-7 cm in length. Where the true volumes ($V_{S\_measured}$) were measured by two-chamber Helium pycnometer while the geometrical data by digital calipers. The data shows that the pore volume of such samples ranges between 11–26 cm^3.

After the separation valve is opened the gas present in the reference cell will expand into this additional volume causing the pressure drop from p_{g1} to p_{g2}. To size the reference volume that would give us the correct reading for both pressure in function of the corresponding volume range the measurement conditions of a true working case were taken into consideration.

3. RESULTS

As presented previously, if a modified triaxial Hassler type core holder is used, and sufficient seal of the sample surface is achieved then theoretically only one problem must be solved. Which is none other than the sizing of the reference volume in function of the measured pore volume.

Considering the porosity range (15–30%) of the 26 sandstone samples the calculated pore volume ranges between 11 cm^3 and 26 cm^3. These values in the following calculations will be referred to as the minimum and maximum of pore volume. The correct reference volume will be found where at the minimal pore volume sufficient pressure loss of the reference pressure is recorded while at the same time at the maximum pore volume pressure loss does not reach a critical level. For the sizing method pressure conditions used in Quantachrome pycnometers (such as Ultrapyc 1200e and older devices) will be taken into consideration. In these two chamber pycnometers the reference pressure is usually set between 17–20 psig which using the correct sample holder and reference chambers for the given sample size will result in an expansion pressure of somewhere between 9–12 psig.

For this reason, during the sizing procedure the recommended pressure of 17 psig (reference) and 10 psig (expanded) was used. Meaning that if the expansion pressure is located between these values the volume is considered sufficient, and if it decreases below 10 psig it is not. This ensures that the added volume is large enough to produce recognizable pressure loss to measure correctly but not large enough to provide the risk of a significant pressure drop.

During the procedure first, the optimal reference volume for both the minimum and maximum pore volumes must be calculated according to Equation 13 where the pressure ratio of 17/10 is used. Resulting in the reference volumes of 15.7143 cm^3 for the minimal and 37.1428 cm^3 for the maximal pore volumes. The required reference volume should be located in between these two values. In the second step the expansion pressures are calculated for both the minimal and maximal pore volumes on this volume range in function of the starting 17 psig reference pressure applying the rearranged form of *Equation 13*. These calculations resulted in the two curves presented in *Figure 3*.

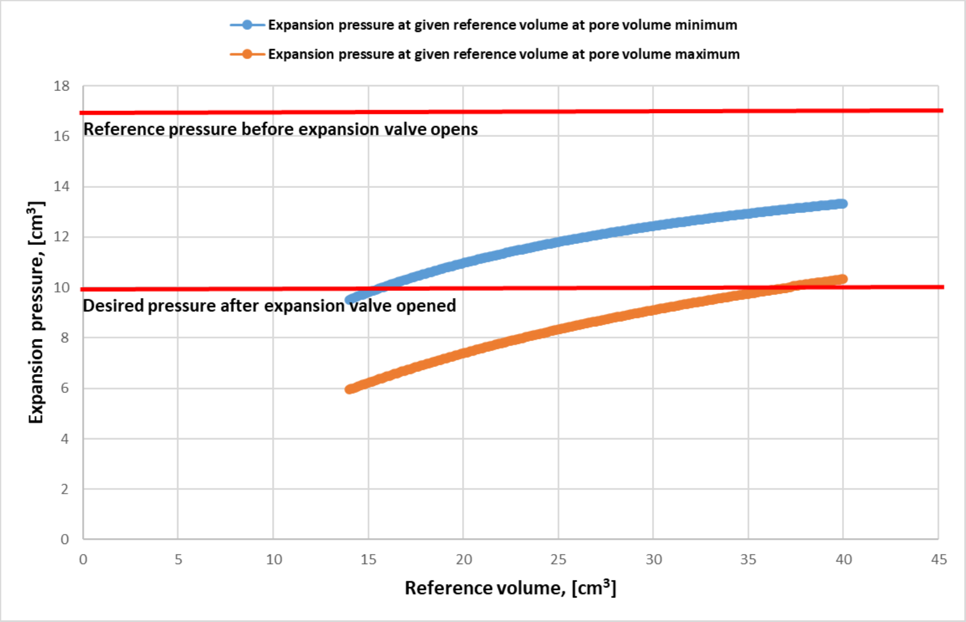

Figure 3

The calculated expansion pressures in function of reference volumes

As shown in the figure the reference volume which is usable for both the minimum and maximum pore volumes is somewhere of 37.2 cm^3. This volume will cause sufficient pressure to drop for the minimum pore volume to calculate the pore volume and is not small enough to cause problems at the maximum pore volume as well. In result a reference volume of 40 cm^3 could be considered as a preferable solution for the measurement of such rock samples.

4. DISCUSSION AND CONCLUSION

In this work the theoretical basis and the need of single chamber gas pycnometry for direct pore volume measurement was introduced. During porosity calculation of sample plugs the highest error occurs from the uncertainties of the total geometric volume of the sample. Therefore, to eliminate its effect, the pore and matrix volumes of the sample must be determined instead.

However, this is easier said than done since traditional two chamber gas pycnometers can only measure the solid volume of the sample. As a result, a second procedure is required to determine the pore volume for which mercury penetration pynometry could be considered as gold standard. Unfortunately, this method is uncapable of measuring the same sample size therefore a much smaller secondary sample must be prepared. Thus, the heterogeneity of the rock can cause significant diversions between the two procedures.

The only solution for direct porosity measurement of the sample plugs is to alter the existing method of gas pycnometry by using a single chamber construction where the pressure after expansion would only be the function of the pore volume.

However, this is easier said than done due to the known difficulties regarding the procedure that hindered its spread in the industry. The problematics of sample loading and the sizing of the reference volume for correct measurement, however, could be solved as presented in this paper.

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