# ESTIMATION OF BIOT'S COEFFICIENT IN THE LABORATORY DURING

# POROSITY MEASUREMENT UNDER STRESS

### A Thesis

### by

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### MASTER OF SCIENCE

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### ABSTRACT

Poroelasticity is fundamental in the application of petroleum reservoir mechanics and production, wellbore stability, and hydraulic fracturing. Biot's coefficient is key mechanical quantity for better understanding of the poroelastic behavior of the reservoir matrix under stress. Its measurement in the laboratory traditionally involves a mechanical setup, such as tri-axial compression test, and is time-consuming and costly. However, during the porosity and permeability measurements under stress, Biot's coefficient is often assumed equal to one due to inherent difficulties in its measurement.

In this study, a new analytical method is presented for estimating Biot's coefficient of the rock samples in the laboratory during porosity measurement under stress. The approach can be extended to permeability measurements later. The technique used in this study requires helium uptake by the sample under predetermined confining pressure and pore pressure, and the application of Boyle's law. It allows simultaneous prediction of the sample pore volume, the coefficient of isothermal pore compressibility, and the effective stress coefficient. The effective stress coefficient is a precursor of the Biot's coefficient and influenced by the applied confining and pore pressure values. In this thesis we show a new graphical approach to predict the Biot's coefficient from these laboratory quantities. The procedure is fast and can be performed in any reservoir petrophysics laboratory using the traditional Hassler core holder setup.

Biot's coefficient estimated for sandstone, carbonate, and shale samples are in a range between 0.46 and 1, which is commonly found in the literature performing the mechanical tests. The proposed method allows the determination of Biot's coefficient

using the conventional porosimeter set up without the need for sophisticated geomecanical tests.

### DEDICATION

To my wonderful parents Olga Patricia and Nicolas, for all their sacrifices, unconditional love, and support. I love you more than anything.

To my grandmother Luz Marina, for all her dedication and love.

To my big brothers, Nicolas and Jose, for their guidance, and my baby brother Pedro, for being a new motivation in my life.

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# NOMENCLATURE

a <sub>i</sub>	Ratio of confinement pressure to pore pressure at any stage, dimensionless
a <sub>avgi</sub>	Average ratio of confinement pressure to pore pressure at any stage,
	dimensionless
a <sub>0</sub>	Ratio of confinement pressure to pore pressure at reference stage,
	dimensionless
c <sub>b</sub>	Isothermal coefficient of bulk compressibility, psi <sup>-1</sup>
c <sub>p</sub>	Isothermal coefficient of pore compressibility, psi <sup>-1</sup>
c <sub>p,c</sub>	Isothermal coefficient of pore compressibility with variable confinement,
	psi <sup>-1</sup>
c <sub>p,p</sub>	Isothermal coefficient of pore compressibility with variable pore pressure,
	psi <sup>-1</sup>
c <sub>r</sub>	Matrix compressibility, psi-1
E	Young's modulus, psi
К	Bulk matrix moduli, Pa
K <sub>s</sub>	Grain matrix moduli, Pa
$\Delta k_p$	Change of permeability with the variation of the pore pressure while
	confining stress is kept constant, psi
$\Delta k_c$	Change of permeability with the variation of confining stress while pore
	pressure is kept constant, psi

n	Effective stress coefficient, dimensionless
n <sub>di</sub>	Initial amount of gas in dead volume, mole
n <sub>df</sub>	Amount of gas in dead volume at equilibrium pressure, mole
n <sub>i</sub>	Initial amount of gas the system, mole
n <sub>f</sub>	Amount of gas the system at equilibrium pressure, mole
n <sub>ri</sub>	Initial amount of gas in reference volume, mole
n <sub>rf</sub>	Amount of gas in reference volume at equilibrium pressure, mole
n <sub>si</sub>	Initial amount of gas in sample pore volume, mole
n <sub>sf</sub>	Amount of gas in sample pore volume at equilibrium pressure, mole
P <sub>0</sub>	Reference pressure, psia
Р	Pressure of interest, psia
P <sub>c</sub>	Confinement pressure, psia
P <sub>c0</sub>	Confinement pressure at reference stage, psia
P <sub>cavg</sub>	Average confinement pressure between two consecutive stages, psia
P <sub>di</sub>	Initial pressure in dead volume, psia
P <sub>df</sub>	Pressure in dead volume at equilibrium conditions, psia
P <sub>diff</sub>	Differential pressure, psia
P <sub>f</sub>	Equilibrium pressure in dead volume and sample volume, psia
Pp	Pore pressure, psia
P <sub>p0</sub>	Pore pressure at reference stage, psia
P <sub>pavg</sub>	Average pore pressure between two consecutive stages, psia
P <sub>ri</sub>	Initial pressure in reference volume, psia

P <sub>rf</sub>	Pressure in reference volume at equilibrium conditions, psia
P <sub>si</sub>	Initial pore pressure in sample, psia
P <sub>sf</sub>	Pore pressure in sample at equilibrium conditions, psia
Q	petrophysical quantity of interest
R	Ideal gas constant, psi-ft <sup>3</sup> /lbmol-R
Т	Reservoir temperature, °R
ν	Poisson ratio, dimensionless
V <sub>d</sub>	Dead volume, cc
V <sub>p0</sub>	Sample pore volume at zero pore stress, cc
V <sub>p</sub>	Sample pore volume, cc
V <sub>Pi</sub>	Sample pore volume at any pressure stage, cc
V <sub>r</sub>	Reference volume, cc
Vs	Sample pore volume, cc
V <sub>si</sub>	Initial sample pore volume, cc
V <sub>sf</sub>	Sample pore volume at equilibrium conditions, cc
z <sub>di</sub>	Gas correction factor at P <sub>di</sub> , dimensionless
z <sub>df</sub>	Gas correction factor at $P_{df}$ , dimensionless
z <sub>f</sub>	Gas correction factor at P <sub>f</sub> , dimensionless
z <sub>ri</sub>	Gas correction factor at Pri, dimensionless
z <sub>rf</sub>	Gas correction factor at Prf, dimensionless
z <sub>si</sub>	Gas correction factor at $P_{si}$ , dimensionless
Z <sub>sf</sub>	Gas correction factor at Psf, dimensionless

# GREEK SYMBOLS

 $\alpha$ Biot's coefficient, dimensionless $\sigma$ Total stress, psi $\sigma_{e}$ Effective stress, psi $\sigma_{avg}$ Average effective stress between consecutive stages, psi

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### 1. INTRODUCTION

### 1.1. Overview

Poroelasticity plays a vital role in the application of rock mechanics in petroleum engineering, such as reservoir engineering, drilling and wellbore stability, hydraulic fracturing, and production. (Franquet and Abass, 1999). The Biot's coefficient is an important quantity resulting from the theory of poroelasticity. It addresses the fundamental concept of partitioning the externally applied stresses between the porous skeleton and the pore fluid. (Selvadurai and Suvorov, 2020). Biot's coefficient is an important parameter used to determine the influence of pore pressure on rock deformation (Laurent et al. 1993), and the effective stress that affects the petrophysical properties of the reservoir formations (Civan 2020). Various failure mechanisms in rocks such as sand production, hydraulic fracturing and wellbore stability are associated with the effective stress or the so-called Biot's effective stress in the rock. To model these mechanisms accurately the value of Biot's coefficient must be known. However, in many cases, this coefficient's value has been chosen as one a priori. (Azeemuddin et al, 2002). This is the case because the laboratory estimation of the Biot's coefficient is costly, time-consuming and often the lack of available data prevents its estimation (Ling et al., 2016). In this study, a graphical method is proposed for the estimation of Biot's coefficient in the reservoir petrophysics laboratory during porosity measurement under stress.

In this thesis, the laboratory set up is first introduced, and then the experimental and analysis procedure are discussed. The setup comprises of a standard Boyle's law porosimeter, where the measurement cell is replaced with a Hassler core holder. The latter allows the application of confining pressure to the disc-shape core plug samples. Next, Biot's coefficient is estimated for carbonate, sandstone and shale samples using the applied pore pressure and confining pressure, and the measured pore compressibility.

### **1.2. Effective stress**

Effective stress (Terzaghi, 1936, 1943, 1943; Biot, 1941) is one of the key concepts developed for understanding poroelastic characteristics of materials, such as rocks, which consist of both the elastic properties (Nur and Byerlee, 1971) and the properties related to the pore structure of the rock such as fractures and cracks (Brace and Martin, 1968; Bruno and Nakagawa, 1991; Jaeger et al., 2007; He et al. 2016).

At a depth, reservoir rock is subjected to in-situ stresses arising from the combined effects of overburden (or lithostatic) pressure, which is exerted by the weight of overlying rocks; tectonic stresses that are generated by large-scale movements in the Earth's crust; and pore pressure that is exerted by the fluids present in the rock pores. The source of these stresses can be decomposed into two parts: external load and internal fluid-applied pressure. (Zaki et al. 1995).

In the simplest proposed form, the effective pressure is introduced as a quantity equal to the difference between the total stress and the pore pressure (Terzaghi, 1936; Handin, 1958; Hubbert and Rubey, 1959):

where  $\sigma_e$  refers to effective stress,  $\sigma$  is the total stress and  $P_p$  is the pore pressure. The total stress in the laboratory can be represented as the confining pressure, and Equation 1 can be expressed as the differential pressure ( $P_{diff}$ ):

However, as stated by Gangi (1996), measurements of certain material properties such as porosity, acoustic velocity, permeability, etc. made with higher precision laboratory equipment have been fitted with the following equation:

where  $\sigma_e$  refers to effective stress, and *n* is assumed to be a constant with a value that is generally less than 1.0 (Nur and Byerlee, 1971). This constant is commonly known as the effective stress coefficient, and it depends on various parameters, including the rock minerology, stiffness of grains, pore structure, and stress conditions (Civan, 2020). In this study I show this coefficient is a measured quantity that is related to (but not equal to) Biot's coefficient. I also show that its value for a rock sample could be much larger than 1.0.

Todd and Simmons (1972) defined the effective stress coefficient from their experiments on Chelmsford granite samples with the following expression:

$$n = 1 - \frac{\partial Q/\partial P_{p}|_{\sigma_{e}}}{\partial Q/\partial \sigma_{e}|_{P_{p}}} \dots (4)$$

where Q is any measured physical quantity. **Equation 4** is the most popular definition used in the rock mechanics laboratory for determining n under the laboratory conditions (Sarker and Batzle, 2008).

in the literature the effective stress coefficient is commonly and incorrectly referred to as "Biot's coefficient" or "Biot-Willis poroelastic coefficient",  $\alpha$ , which is another important geomechanical parameter expressing the fractional degree of participation of the pore-fluid pressure in the effective stress affecting the bulk volumetric strain in elastic rocks of isotropic types (Biot 1941; Biot and Willis 1957). However, in their study Aldana and Akkutlu (2019) showed that *n* can take values larger than 2.0. This experimental evidence indicated that *n* could be related but not equal to the Biot's coefficient. Then the natural question is how the effective stress coefficient *n* is related to Biot's coefficient  $\alpha$ . Answering this question is the center of my work. First, let us review the literature on the Biot's coefficient.

### **1.3. Biot's coefficient**

Biot (1941) developed the first rational theory for the mechanical behavior of porous materials. Based on Terzaghi (1936), a theoretical description of porous materials saturated by a viscous fluid was presented by Biot, considering a general case in which

the rock grains are not necessarily homogeneous or isotropic (Zimmerman et al. 1986). This was the starting point of the theory of poroelasticity. In the following years, Biot (1955) extended his theory to anisotropic cases, and also to poro-visco-elasticity (Biot, 1956; Schanz, 2003).

Biot's coefficient is a mechanical property of the solid and the porous frame that make up the rock; therefore, it is independent of the fluid properties (Biot and Willis, 1957; Skempton, 1961; Cheng, 2016). It depends on many factors such as rock type, cementation, pore geometry, porosity, mineral constituents, and their geometrical arrangement (Alam et al., 2009, Qiao et al., 2012).

Based on Biot's law the effective stress,  $\sigma_e$  acting on the rock (also known as Biot's effective stress in the geotechnical field) is determined as the difference between the total confining stress and some degree of participation of the pore-fluid pressure (Biot and Willis 1957; Byerlee and Zoback 1975; Zoback and Byerlee 1975; Kümpel 1991; Zimmermann 1991; Kwon et al. 2001). According to this theory, then **Equation 4**, can be expressed as follows:

In essence, based on this definition the pore pressure helps counteract the mechanical stress carried through grain-to-grain contact (He et al., 2016). Based on the concept of effective stress, many methods have been presented to improve the accuracy and efficiency of the Biot's coefficient measurement, since it is the key factor to accurately

calculate effective stress (He et al. 2016). As mentioned before, the pressure difference is often used because Biot's coefficient is unknown, leading to inaccuracy in the estimation of effective stress. Nur and Byrlee (1971) showed the different volumetric strains against confining pressure (Figure 1a), against the difference between confining pressure (Figure 1b) and against the theoretical effective stress (Figure 1c) in a Weber sandstone. The open circles show the strains from dry samples without pore pressure. The results in Figure 1b indicate an improvement in the scatter of the data from Figure 1a, by applying the empirical effective stress law. However, significant scatter remains, and a better trend in seen when the theoretical effective stress is implemented. Figure 1 depicts the difference in the volumetric strain when Biot's coefficient is considered.



Figure 1. Volumetric strain versus effective stress. (a) Strain vs confining pressure. (b) Strain vs the difference between confining and pore pressure. (c) Strain vs theoretical effective stress.

Biot's coefficient is conventionally calculated using the bulk and the grain compressibility values of the rock as:

where  $C_r$  is the matrix (or grain) compressibility, and  $C_b$  the bulk compressibility. Bulk compressibility can be estimated as:

$$C_b = \frac{3(1-2\nu)}{E}$$
....(7)

Here, v and E represent Poisson ratio and Young's modulus, respectively. Similarly, because by definition the compressibility is the inverse of the bulk modulus, Geertsa (1957) and Skempton (1961) presented a conventional technique to determine Biot's coefficient by use of the moduli:

$$\alpha = 1 - \frac{K}{K_s} \tag{8}$$

where *K* is the bulk modulus of the rock (or grain) and  $K_s$  is the matrix bulk modulus. (Nur and Byerlee, 1971; Berryman, 1992; He et al., 2016). The bulk modulus of the rock and matrix bulk modulus are measured in two different experiments. In the first experiment, *K* is obtained during a testing of a jacketed specimen; and in the second experiment,  $K_s$  is determined from an unjacketed test at the same stress condition. (Ingraham et al (2017). Azeemuddin et al. (2002) measured *K* and  $K_s$  with "static" and "dynamics" methods enhancing the proposed technique. Later, Franquet and Abass (1999) proposed a technique to measure Biot's coefficient based on the ratio between the variation of the pore volume and the variation in the total bulk volume in the sample as:

$$\alpha = \frac{\Delta V_p}{\Delta V} \tag{9}$$

where  $\Delta V_p$  corresponds to the variation of the pore volume, and  $\Delta V$  is the variation of the total rock volume. The pore volume measurement needs to have a high level of accuracy in this method. Franquet and Abass (1999) also developed a "Mohr envelope" method to estimate the Biot's coefficient. However, several preliminary tests are needed to define the Mohr envelope.

Qiao et al. (2012) determined the Biot's coefficient under the assumption that the permeability follows the effective-stress law. The Biot's coefficient is calculated from the variation of permeability due to the change in pore pressure or confining pressure with the following expression:

where  $\Delta k_p$  refers to the change in the measured permeability due to change in pore pressure, while confining pressure is kept constant, and  $\Delta k_c$  is the change in the measured permeability due to change in confining pressure, while pore pressure is kept constant. Nevertheless, it is difficult to determine the variation in permeability accurately when the rock sample is tight. (He et al. 2016). A common laboratory method used for estimating Biot's coefficient includes compression test using a triaxial cell (Nermoen et al. 2013; Ingraham et al. 2017). Ideally, there should be an independent control of the confining pressure, overburden stress, and pore pressure. (Li et al. 2020). Additionally, experimental studies on Biot's coefficient with respect to ultrasonic velocities has been studied by a limited number of experiments (Christensen and Wang, 1985; Hornby, 1996; Sarker and Batzle, 2008).

Several methods have been proposed for the estimation of Biot's coefficient. Nevertheless, this coefficient's value is typically unknown and often assumed equal to 1.0, simply because its determination is time-consuming, costly, and/or requires a sophisticated laboratory setup. A new method to estimate Biot's coefficient during porosity measurements under stress is presented in this study, which follows a relatively fast procedure that can be performed in a traditional core-holder set up available in every reservoir-petrophysics laboratory.

### **1.4. Purpose of Study**

This study attempts to provide an analysis method to estimate the Biot's coefficient in the laboratory during porosity measurement under stress. The experimental procedure is based on the laboratory method for estimation of storage capacity of rock samples under effective stress proposed by Aldana and Akkutlu (2019). Using a typical porosimeter set up, the Biot's coefficient is determined. The approach allows us to obtain the isothermal coefficient of pore compressibility, the effective stress coefficient and the change of the pore volume as a function of the effective stress. These quantities will be obtained in the laboratory for sandstone, carbonate and several resource shale samples. The fundamentals of the methodology are discussed in Chapter 3, and the results are included in Chapter 4.

### 2. MATHEMATICAL MODEL

The approach proposed to determine Biot's coefficient is based on the robust mathematical model proposed by Aldana and Akkutlu (2019) for the analysis of the laboratory data of the experimental procedure they proposed to estimate storage capacity of rock samples under effective stress. Their model development can be divided in two sections: (1) Pore volume estimation as a function of effective stress, (2) Mass balance for helium gas used in the setup.

### **2.1.** Pore Volume as a Function of Effective Stress.

Their derivation begins with the definition of the coefficient of isothermal pore compressibility of a rock ( $C_p$ ) in terms of effective stress. Geerstma (1957) defines it as the change in the pore volume of the rock per unit increase in pore pressure, and it can be defined as:

where  $V_{p0}$  referes to the pore volume of the rock at a reference pressure, also called original pore volume at zero stress. Equation 11 includes the effective stress, which is expressed in terms of the applied confining pressure and the pore pressure, as indicated in Equation 3. Now, considering temperature as a constant throughout the proposed experimental procedure, Equation 11 can be written as:

By using the chain rule and the effective stress definition, the isothermal pore compressibility can be expressed as:

$$C_p = -\frac{1}{V_{po}} \left( \frac{dV_p}{dp_c} \frac{dp_c}{d\sigma_e} + \frac{dV_p}{dp_p} \frac{dp_p}{d\sigma_e} \right).$$
(13)

Now, by taking the derivative of pressures with respect to the effective stress using **Equation 3**:

$$\frac{dp_c}{d\sigma_e} = 1$$
 and  $\frac{dp_p}{d\sigma_e} = -\frac{1}{n}$ ....(14)

Substituting Equations 13 into Equation 14, we obtain:

**Equation 15** can be re-organized as:

$$C_p V_{po} = -\left(\frac{dV_p}{dp_c} - \frac{1}{n}\frac{dV_p}{dp_p}\right)$$
$$n C_p V_{po} = \frac{dV_p}{dp_p} - n\frac{dV_p}{dp_c}....(16)$$

Now, parameter a is introduced, which represents the stress potential of the gas storage system and reflects the contrast in the applied pressure conditions. Then, a relation between the confining pressure and the pore pressure can be expressed as:

$$p_c = anp_p.....(17)$$

Following this relation, if the applied confining pressure is increased and the pore pressure is decreased, the stress potential will be higher. Substituting **Equation 17** into **Equation 16**, we have:

Separating the variables, we obtain:

Now, integrating from  $P_p = P_0$  to  $P_p = P_1$ , the pore volume of the rock sample changes from  $V_p$ =  $V_{po}$  to  $V_p = V_{p1}$ , we have the following expression:

Applying trapezoidal rule for the integration of the third term, we have:

a takes the value of  $a_0$  at the reference pressure  $P_{p0}$ , and a takes the value  $a_1$  when the pressure is taken to  $P_{p1}$ . Equation 21 can be re-organized as:

$$V_{p1}\left(1 - \frac{1}{a_{avg}}\right) = V_{po}\left[nC_p(p - p_0) + \left(1 - \frac{1}{a_{avg}}\right)\right].$$
(22)

Considering Equation 22 in terms of effective stress we obtain:

$$V_{p1}\left(\frac{\sigma_{avg}}{P_{cavg}}\right) = V_{po}\left[nC_p(p-p_0) + \left(\frac{\sigma_{avg}}{P_{cavg}}\right)\right].$$
(23)

where  $1/a_{avg}$  has been defined as the arithmetic average of the 1/a values:

**Equation 23** represents the pore volume change due to effective stress. This expression will be implemented in the mass balance presented by Aldana and Akkutlu (2019).

### 2.2. Mass Balance for Helium Gas Used in the Setup

Kang (2011) proposed a method using helium gas to estimate the pore volume compressibility of rocks, which he implemented to evaluate the gas storage capacity of Barnett shale samples. Later, Aldana and Akkutlu (2019) developed a mass balance equation for the number of moles of helium gas used during the laboratory measurement based on Kang's method, but they considered the influence of the effective stress coefficient in the effective stress affecting the samples. This mass balance is based in the following diagram of the laboratory apparatus:



Figure 2. Simplified diagram of laboratory apparatus used in the mass balance

In **Figure 2**, the diagram presented is adapted from API RP-40. In this schematic the volumes of the system are the reference volume  $(V_r)$ , the sample pore volume  $(V_s)$ , and the summation of the dead volumes in the system  $(V_d)$ . At the measurement initial conditions, helium is expanded to the reference volume while Valve 2 is closed. The initial number of moles of gas in the system  $(n_i)$  is equivalent to the summation of the number of moles in the reference volume  $(n_{ri})$ , the sample volume  $(n_{si})$  and the dead volume  $(n_{di})$ :

Initially, all the values are closed and the reference volume holds helium at a high pressure. When Value 2 is opened, the reference volume is connected to the sample volume, and the helium is therefore expanded to the sample volume. Once the system reaches equilibrium pressure, the number of moles is equivalent to the sum of the number of moles in the reference volume  $(n_{rf})$ , the sample volume  $(n_{sf})$  and the dead volume  $(n_{df})$ :

 $n_f = n_{rf} + n_{df} + n_{sf}....(26)$ 

Assuming the gas mass is conserved, the final moles of gas in the system  $(n_f)$  should be equal to the initial number of moles, obtaining the following:

$$n_i = n_f$$
  
 $n_{ri} + n_{di} + n_{si} = n_{rf} + n_{df} + n_{sf}$ .....(27)

Equation 27 can be re-organized as:

Now, using the compressible equation of state, the number of moles of gas in each volume, at both initial and equilibrium conditions, can be expressed as:

$$n_{di} = \frac{P_{di}V_d}{Z_{di}RT}; \quad n_{df} = \frac{P_{df}V_d}{Z_{df}RT}.$$
(30)

$$n_{si} = \frac{P_{si}V_{si}}{Z_{si}RT}; \quad n_{sf} = \frac{P_{sf}V_{sf}}{Z_{sf}RT}....(31)$$

where  $V_{si}$  and  $V_{sf}$  are the sample pore volumes at initial pressure and equilibrium pressure, respectively. T corresponds to temperature.  $Z_{ri}$ ,  $Z_{si}$ , and  $Z_{di}$  are the gas compressibility factor at the initial conditions in the reference volume, the sample volume, and the dead volume, respectively. Similarly,  $Z_{rf}$ ,  $Z_{sf}$ , and  $Z_{df}$ , correspond to the gas compressibility factor at equilibrium conditions in the reference volume, the sample volume and the dead volume, respectively. R is the universal gas constant. Substituting **Equations 29-31** into **Equation 28**, we obtain:

$$\frac{P_{sf}V_{sf}}{Z_{sf}RT} - \frac{P_{si}V_{si}}{Z_{si}RT} = \left[\frac{P_{ri}V_{r}}{Z_{ri}RT} - \frac{P_{rf}V_{r}}{Z_{rf}RT}\right] - \left[\frac{P_{df}V_{d}}{Z_{df}RT} - \frac{P_{di}V_{d}}{Z_{di}RT}\right] \dots (32)$$

Since the measurements are performed under isothermal conditions, the universal gas constant (R), and the temperature (T) are cancelled:

Now, considering the expressions presented in **Equations 22-24** for the change of sample pore volume as a function of effective stress, the initial and final sample volume  $(V_{si} \text{ and } V_{sf} \text{ respectively})$  can be expressed as:

$$V_{si}\left(1-\frac{1}{a_{avgi}}\right) = V_{po}\left[nC_{p}(p_{si}-p_{0}) + \left(1-\frac{1}{a_{avgi}}\right)\right]....(34)$$
$$V_{sf}\left(1-\frac{1}{a_{avgf}}\right) = V_{po}\left[nC_{p}(p_{sf}-p_{0}) + \left(1-\frac{1}{a_{avgf}}\right)\right]....(35)$$

**Equation 34** and **Equation 35** can be reduced to a common linear approximation when the applied confining pressure is much larger than the pore pressure (Kang et al, 2011):

$$V_{si} = V_{po} [1 + nC_p (p_{si} - p_0)] ....(36)$$
$$V_{sf} = V_{po} [1 + nC_p (p_{sf} - p_0)] ....(37)$$

Re-organizing **Equations 34-35** in terms of the sample volume at initial and equilibrium conditions, we obtain:

$$V_{si} = \frac{V_{po} \left[ nC_p (p_{si} - p_0) + \left(1 - \frac{1}{a_{avgi}}\right) \right]}{\left(1 - \frac{1}{a_{avgi}}\right)} \dots (38)$$
$$V_{sf} = \frac{V_{po} \left[ nC_p (p_{sf} - p_0) + \left(1 - \frac{1}{a_{avgf}}\right) \right]}{\left(1 - \frac{1}{a_{avgf}}\right)} \dots (39)$$

Now, substituting Equations 38-39 into Equation 33, we have:

To obtain an expression for the isothermal coefficient of pore compressibility, Equation 40 can be re-written as:

where:

$$A = V_r \left( \frac{P_{rf}}{Z_{rf}} - \frac{P_{ri}}{Z_{ri}} \right) + V_d \left( \frac{P_{df}}{Z_{df}} - \frac{P_{di}}{Z_{di}} \right)$$
$$B = \frac{P_{sf}}{Z_{sf}} - \frac{P_{si}}{Z_{si}};$$
$$C = \frac{P_{si}}{Z_{si}} \left( 1 - \frac{1}{a_{avgi}} \right) (p_{si} - p_0) - \frac{P_{sf}}{Z_{sf}} \left( 1 - \frac{1}{a_{avgf}} \right) (p_{sf} - p_0)$$

where:

$$\begin{bmatrix} 1 - \frac{1}{a_{avgi}} \end{bmatrix} = 1 - \frac{\frac{1}{a_{i}} + \frac{1}{a_{0}}}{2} = 1 - \frac{nP_{p0}}{2P_{c0}} - \frac{nP_{pi}}{2P_{ci}}$$
$$\begin{bmatrix} 1 - \frac{1}{a_{avgf}} \end{bmatrix} = 1 - \frac{\frac{1}{a_{f}} + \frac{1}{a_{0}}}{2} = 1 - \frac{nP_{p0}}{2P_{c0}} - \frac{nP_{pf}}{2P_{cf}}$$

Also, when the system reaches equilibrium pressure  $(P_f)$ , it will be the equilibrium pressure at the reference volume  $(P_{rf})$ , dead volume  $(P_{df})$ , and sample volume  $(P_{rf})$ . Therefore  $P_{rf} = P_{df} = P_{sf} = P_f$ . Similarly, for the Z-factor,  $Z_{rf} = Z_{df} = Z_{sf} = Z_f$ .

Then coefficients A, B and C in Equation 41 can be re-written as:

$$A = V_r \left( \frac{P_f}{Z_f} - \frac{P_{ri}}{Z_{ri}} \right) + V_d \left( \frac{P_f}{Z_f} - \frac{P_{di}}{Z_{di}} \right)$$
  

$$B = \frac{P_f}{Z_f} - \frac{P_{si}}{Z_{si}}$$
  

$$C = \frac{P_{si}}{Z_{si}} \left( 1 - \frac{nP_{p0}}{2P_{c0}} - \frac{nP_{pi}}{2P_{ci}} \right) (p_{si} - p_0) - \frac{P_{sf}}{Z_{sf}} \left( 1 - \frac{nP_{p0}}{2P_{c0}} - \frac{nP_{pf}}{2P_{cf}} \right) (p_{sf} - p_0)$$

**Equation 41** will be used to determine the isothermal coefficient of pore compressibility and the effective stress coefficient of the rock samples in the laboratory.

#### **3. EXPERIMENTAL PROCEDURE AND DATA ANALYSIS**

### **3.1.** Laboratory Setup

The laboratory setup proposed is shown in **Figure 3**. It is based on the equipment employed by Aldana and Akkutlu (2019) and Kim & Akkutlu (2018). The schematic includes a helium gas tank, that is connected through Valve 1 to the reference volume ( $V_r$ ). The measurement fluid is a non-adsorptive gas with negligible adsorption, such as helium (Santos and Akkutlu, 2013). The reference volume is used to adjust the gas pressure to any value throughout the experiment. Valve 2 allows the connection between the reference volume and the core-holder. The core-holder fits 1-inch diameter disk-shape core samples. Pressure transducers are used with a data acquisition program to record the pressures at any desired time interval. The sample is subjected to confining pressure by use of a hydraulic pump, allowing the measurements to be performed under controlled confining stress. The apparatus is enclosed in an environmental chamber to maintain the temperature constant.

### **3.2. Experimental Procedure**

Using the laboratory set up depicted in **Figure 2**, the following is the step-by-step experimental procedure based on Aldana and Akkutlu (2019):


Figure 3. Laboratory Set Up

1. Determine the void volume  $(V_d)$  and the reference volume  $(V_r)$ . The former corresponds to the valves, tubing and Swagelok fittings, as they can store small gas volumes. This can be achieved by either using Boyle's law or by injecting water into the equipment while keeping track of the injected-water volume.

2. Check that both the core-holder and the sample are clean.

3. Locate the core sample into the rubber, close end caps, and adjust the sample's position by pushing the core-holder's piston. Care there are no gaps between the sample and the borders.

4. By the use of the hydraulic water pump, apply confining pressure (P<sub>c</sub>) to the coreholder.

5. Check pressure from gauges are consistent with transducer readings. If not,

execute the correspondent calibration.

6. Check the pressure inside the core-holder  $(P_{si})$  corresponds to atmospheric pressure. If not, purge the system by using Valve 2.

7. Check the desired confining pressure is being applied and all the valves in the system are initially closed.

8. Open Valve 1 to allow helium gas expansion to the reference volume until the desired pressure is reached. Then, close Valve 1 and wait until reference pressure ( $P_{ri}$ ) reaches equilibrium for about 60 minutes. Continuously record the pressure data from the transducers using the data acquisition system.

9. Allow helium gas expansion into the core-holder by opening Valve 2. A small differential pressure (20-30 psi) should be kept during the gas uptake. Let the system reach equilibrium ( $P_t$ ) in about 6-12 hours, depending on the sample's nature.

10. Isolate the reference volume from the core holder by closing Valve 2.

11. Repeat steps 8-11 until the desired number of pressure stages. At least two pairs of consecutive pressure stages are required to solve for the parameters n and  $C_p$  simultaneously and then, estimate Biot's coefficient.

In step 9, as mentioned, the system is allowed to reach equilibrium once Valve #2 is opened. When helium is admitted into the core-holder by opening Valve #2, a pressure transient can be observed due to the higher pressure of the reference volume compared to the core-holder. This pressure transient can be used for permeability estimation if desired. However, the transport analysis is out of the scope of this study. **Figure 3** shows an example of the change of the pressure at the reference volume through time. As observed, after some time the system tends to equilibrate, represented in the figure by the transition from the pressure transient to a steady pressure value, which was obtained after almost 6 hours in this case. Once we observe this stable region, the system is considered at equilibrium.



Figure 4. Pressure behavior as a function of time during a measurement stage.

An initial measurement at low pressure (0 - 50psi) and low confinement (below 100 psi) is needed in order to determine reference pore volume  $(V_{p0})$ . However, this value can be determined as part of a separate measurement using the standard helium porosimeter method.

Pressure values at the reference volume at the initial conditions of each stage are labeled as  $P_{ri}$ , and at equilibrium pressure as  $P_{rf}$ . The same format is used for the case of the dead volume and the sample volume, having the pressures at the stage initial conditions as  $P_{di}$  and  $P_{si}$ , respectively, and at equilibrium pressure as  $P_{df}$  and  $P_{sf}$ . Pressure values at equilibrium should be measured carefully at each stage, especially in the case when the pore pressure is high, due to potential gas leakage. A gas-leakage correction method can be found in Aldana (2019).

The Abou-Kassem & Dranchuk correlation (1975) is implemented to determine the Z-factor for helium at the initial and equilibrium conditions of each measurement stage. This correlation has been shown to be valid in the extended regions for pure components (Dranchuk & Abou-Kassem, 1975), as is the case of this experimental procedure. The Z-factor for helium in the reference volume at the initial conditions of each stage is designated as  $Z_{ri}$ . For the case of the dead volume and the sample chamber, the Zfactor at initial conditions is represented as  $Z_{di}$  and  $Z_{si}$ , respectively. The Z-factor for helium at equilibrium conditions of each stage is designated as  $Z_f$ .

## **3.2.1.** Example of Data Acquired with Explained Laboratory Set Up and Procedure.

Following the experimental procedure previously presented, an example of the data acquired during the experimental stages can be observed in the following Table:

Stages	Confinement (psi)	Pr (psia)	Pd (psia)	Ps (psia)	Pf (psia)
1	500	98.9	14.7	14.7	55.8
2	500	198.2	55.8	55.8	108
3	1000	318.5	108	108	136.6

 Table 1. Example of Experimental Data Acquired

In **Table 1**, three experimental stages are shown. For each one of them, the confinement pressure values being applied to the sample are presented. The pressure at the reference volume, dead volume, and sample at initial and equilibrium conditions are introduced.

For Stage 1, the confinement pressure applied to the sample was 500 psi. The initial pressure at the sample and the dead volume was 14.7 psi. For the case of the reference volume, the initial pressure was 98.7 psi. Once Valve 2 was opened, time was allowed to let the system reach equilibrium, and the equilibrium pressure was recorded, which is 55.8 psi. Then, Stage 2 was performed. The confinement pressure applied to the sample was 500 psi too. The initial pressure at the sample and the dead volume was 55.8 psi (as the equilibrium pressure from the previous stage). For the case of the reference volume, the initial pressure was increased up to 198.2 psi. Then Valve 2 was opened, and the equilibrium pressure recorded was 108 psi.

Having the data from the experimental procedure, the Z-factor for helium at initial conditions in the reference volume, dead volume and sample can be calculated using the Abou-Kassem & Dranchuk correlation. It is also calculated for equilibrium conditions. In this example the temperature is assumed as a constant value of 77 °F. The Z-factor at each stage can be found in the following table.

Stages	Zri	Zdi	Zsi	Zf
1	1.0044	1.0006	1.0006	1.0025
2	1.0090	1.0025	1.0025	1.0048
3	1.0148	1.0048	1.0048	1.0061

 Table 2. Z-factor Values Predicted for the Stages

As mentioned before, two pairs of consecutive stages are needed to acquire the necessary laboratory data, but the performance of several stages can provide a complete understanding of rock's properties.

### **3.3.** Laboratory Data Analysis

The analysis of the laboratory data can be divided in two sections. The first one corresponds to the determination of the reference pore volume ( $V_{po}$ ), pore compressibility ( $C_{p}$ ), and effective stress coefficient (*n*), based on the proposed method by Aldana and Akkutlu (2019). In the second section Biot's coefficient is determined with *n* and a values.

## **3.3.1.** Determination of Pore Compressibility (C<sub>p</sub>) and Effective Stress Coefficient (*n*)

Based on the previously introduced **Equations 41** in section 2.2, the pore compressibility and the effective stress coefficient can be determined by having two consecutive gas uptakes. For the first gas uptake, the compressibility as function of the effective stress can be expressed as:

Similarly, for the second gas uptake the expression is:

The coefficients A, B, and C are introduced in **Equations 41** in section 2.2. These coefficients have different values at each measurement stage. The components of **Equations 42-43** can be found with the experimental procedure presented except for the pore compressibility ( $C_p$ ), the effective stress coefficient (n) and the coefficients that are functions of the effective stress coefficient. Therefore at least two experimental stages are needed, as there are two unknowns.

This equation system can be solved using any programming language of preference that allows simultaneous prediction for  $C_p$  and n. This solution is obtained every two stages, but several stages are recommended to have higher accuracy in the obtained results.

# **3.3.1.1.** Example of Determination of Pore Compressibility (C<sub>p</sub>) and Effective Stress Coefficient (n)

Following the analysis method previously presented, an example of how to apply the laboratory data to determine the pore compressibility and effective stress coefficient is introduced.

To achieve this, the experimental data from **Table 1** and **Table 2** is implemented to determine the values of the coefficients A, B and C from **Equation 42** and **Equation 43**, at the first and second stage of the measurement, respectively. In this example, the reference pore volume ( $V_{po}$ ) of the sample is 1.056 cc, the reference volume ( $V_r$ ) is 19.21 cc, and the dead volume ( $V_d$ ) is 6.64 cc.

# a. Determination of coefficient A.

For the first stage:

$$A_{1} = V_{r} \left[ \frac{P_{rf1}}{Z_{rf1}} - \frac{P_{ri1}}{Z_{ri1}} \right] + V_{d} \left[ \frac{P_{df1}}{Z_{df1}} - \frac{P_{di1}}{Z_{di1}} \right]$$
$$A_{1} = 19.21 \left[ \frac{55.8}{1.0025} - \frac{98.9}{1.0044} \right] + 6.64 \left[ \frac{55.8}{1.0025} - \frac{14.7}{1.0006} \right] = -550.26$$

For the second stage:

$$A_{2} = V_{r} \left[ \frac{P_{rf2}}{Z_{rf2}} - \frac{P_{ri2}}{Z_{ri2}} \right] + V_{d} \left[ \frac{P_{df2}}{Z_{df2}} - \frac{P_{di2}}{Z_{di2}} \right];$$

$$A_{2} = 19.21 \left[ \frac{108}{1.0048} - \frac{198.2}{1.009} \right] + 6.64 \left[ \frac{108}{1.0048} - \frac{55.8}{1.0025} \right] = -1364.59$$

b. Determination of coefficient B:

For the first stage:

$$B_{1} = \frac{P_{sf1}}{Z_{sf1}} - \frac{P_{si1}}{Z_{si1}}$$
$$B_{1} = \frac{55.8}{1.0025} - \frac{14.7}{1.0006} = 40.97$$

For the second stage:

$$B_{2} = \frac{P_{sf2}}{Z_{sf2}} - \frac{P_{si2}}{Z_{si2}}$$
$$B_{2} = \frac{108}{1.0048} - \frac{55.8}{1.0025} = 51.82$$

c. Determination of coefficient C:

For the first stage:

$$\begin{split} C_{1} &= \frac{P_{si1}}{Z_{si1}} \left( \frac{1}{1 - \frac{nP_{p0}}{2P_{c0}} - \frac{nP_{pi1}}{2P_{ci1}}} \right) (P_{si1} - P_{0}) - \frac{P_{sf1}}{Z_{sf1}} \left( \frac{1}{1 - \frac{nP_{p0}}{2P_{c0}} - \frac{nP_{pf1}}{2P_{cf1}}} \right) (P_{sf1} - P_{0}) \\ C_{1} &= \frac{14.7}{1.0006} \left( \frac{1}{1 - \frac{n*14.7}{2*14.7} - \frac{n*14.7}{2*500}} \right) (14.7 - 14.7) - \frac{55.8}{1.0025} \left( \frac{1}{1 - \frac{n*14.7}{2*14.7} - \frac{n*55.8}{2*500}} \right) (55.8 - 14.7) \\ C_{1} &= - \left( \frac{2287.66}{1 - 0.56n} \right) \end{split}$$

For the second stage:

$$\begin{split} C_2 &= \frac{P_{si2}}{Z_{si2}} \left( \frac{1}{1 - \frac{nP_{p0}}{2P_{c0}} - \frac{nP_{pi2}}{2P_{ci2}}} \right) (P_{si2} - P_0) - \frac{P_{sf2}}{Z_{sf2}} \left( \frac{1}{1 - \frac{nP_{p0}}{2P_{c0}} - \frac{nP_{pf2}}{2P_{cf2}}} \right) (P_{sf2} - P_0); \\ C_2 &= \frac{150.6}{1.0068} \left( \frac{1}{1 - \frac{n*14.7}{2*14.7} - \frac{n*55.8}{2*500}} \right) (55.8 - 14.7) - \frac{108}{1.0048} \left( \frac{1}{1 - \frac{n*14.7}{2*14.7} - \frac{n*108}{2*500}} \right) (108 - 14.7) \\ C_2 &= \left( \frac{6147.85}{1 - 0.56n} \right) - \left( \frac{10028.26}{1 - 0.61n} \right) \end{split}$$

Now, replacing  $V_{po}$  and coefficients A, B and C in Equation 41:

For the first stage:

$$C_{p} = \frac{\frac{A_{1}}{V_{po}} + B_{1}}{n \times C_{1}} = \frac{\frac{-550.26}{0.056} + 40.97}{n \times \left(-\frac{2287.66}{1 - 0.56n}\right)}$$

For the second stage:

$$C_{p} = \frac{\frac{A_{2}}{V_{po}} + B_{2}}{n \times C_{2}} = \frac{\frac{-1364.59}{0.056} + 51.82}{n \times \left(\frac{6147.85}{1-0.56n} - \frac{10028.26}{1-0.61n}\right)}$$

The pore compressibility is considered equal between 2 consecutive stages. Therefore, the expressions for Cp at the first and second stages are used to find the Cp and n values with any programming language package. This example represents the procedure to estimate the Cp and n values with two consecutive experimental stages.

## **3.3.2.** Determination of Biot's Coefficient

Once the values of the effective stress coefficient, pore compressibility, and the reference pore volume at zero effective stress are determined, the Biot's coefficient of the rock sample can be predicted using a simple graphical method. This novel approach interprets the measured multiple-step pressure data on a graphical domain in which the estimation of Biot's coefficient can be performed fast and accurately using a straight line. This coefficient is the resultant y-intercept in a plot of the effective stress coefficient n as a function of the parameter "a", representing the stress potential of the laboratory condition based on the contrast in the applied pressure values, and it is expressed in **Equation 17** in section 2.2.

## **3.3.2.1.** Example of Determination of Biot's Coefficient.

Following the analysis method previously introduced, an example of how to apply the laboratory data to determine Biot's coefficient is presented. The values of the parameter a, and the effective coefficient are required to accomplish the analysis. The parameter a can be determined using **Equation 18**, with the values of pore pressure, confining pressure, and effective stress coefficient found in **Table 3**.

Pc (psia)	Pf (psia)	n	а
1500	955.9	1.22	1.28
1800	1257.4	1.15	1.25
2100	1468.3	1.17	1.22
2400	1655.3	1.15	1.26
2700	1715.3	1.21	1.30
3000	1745.2	1.24	1.39
3300	1768.0	1.29	1.45
3600	1755.9	1.32	1.55
3900	1755.1	1.37	1.63
4200	1757.5	1.35	1.77
4500	1744.2	1.41	1.82
4800	1748.9	1.47	1.87

Table 3. Coefficient a values

Once the parameter a values are available, the Biot's coefficient  $\alpha$  can be predicted with a plot of the effective stress coefficient *n* as a function of the constant a, in which the intercept in this plot corresponds to Biot's coefficient, as it can be seen in the next figure.



Figure 5. Effective stress coefficient *n* as a function of parameter a

In this plot, we can observe a straight line with a linear relationship between the effective stress coefficient and the parameter a, which follows a linear equation of y = mx + b, in which the intercept corresponds to Biot's coefficient. Therefore, in this example, the Biot's coefficient of the analyzed sample is 0.653.

#### 4. RESULTS AND DISCUSSION

## 4.1. Determination of Effective Stress Coefficient and Petrophysical Parameters.

The experimental and analysis procedure was performed on carbonate, sandstone, and several shale samples. Firstly, the reference pore volume  $V_{p0}$  was determined with the standard helium porosimeter measurement obtaining the following results in **Table 4**. The reference pore volume  $V_{p0}$  of the samples was also determined with the proposed set up of this study appliend a near zero effective stress. For the sandstone plug, a value of 2.74 cc was obtained with the standard helium porosimeter. The laboratory setup with the core holder proposed in this study at zero confining pressure provided a value of 2.65 cc. The measurement error is thus 3.3%, which is a reasonable value. For the case of Shale 1 and Shale 2 samples, their reference pore volume is around 1.0 cc using the standard helium porosimeter. The predicted pore volumes using the coreholder for these two samples are very similar to the pore volume measurement results using the routine helium porosity method near zero effective stress. This validation indicates that the proposed laboratory analysis method yields meaningful results.

The reference pore volume values allowed the calculation of the sample's porosity values at zero effective stress conditions, which will be later compared to the porosity values when the effective stress changes. The reference pore volumes were also used afterward to determine the values for the isothermal coefficient of pore compressibility,  $C_p$ , effective stress coefficient, n, and final pore volume. The isothermal coefficient of pore compressibility and effective stress coefficient were determined following the

Sample	Vp0 (cc)	Porosity (%)
Sandstone	2.740	13.61
Carbonate	3.280	8.39
Shale 1	1.060	3.86
Shale 2	0.990	3.46
Shale 3	0.420	1.86
Shale 4	0.650	5.37
Shale 5	0.200	1.44
Shale 6	0.470	3.48

**Table 4. Reference pore volumes** 

analysis method explained in the previous section, in which these parameters are found every two consecutive measurements stages with the implementation of **Equation 41** and the use of a programming language package.

The temperature was assumed as 77°F, and the reference volume and dead volume were can be found in **Table 5**. The experimental data used can be found in **Appendix A**, and the resultant values of  $C_p$  and n for each sample can be found in **Appendix B**.

Sample	Vr (cc)	Vd (cc)
Sandstone	19.21	6.64
Carbonate	19.21	6.64
Shale 1	19.21	6.64
Shale 2	19.21	6.64
Shale 3	5.57	5.84
Shale 4	5.57	5.84
Shale 5	5.57	5.76
Shale 6	5.57	5.76

Table 5. Reference volume (Vr) and dead volume (Vd) values.

The obtained values for the sandstone, carbonate and six shale samples of the effective stress coefficient (n) as a function of effective stress, can be observed in Figure 6, Figure 7, Figure 8, and Figure 9.

In the case of the sandstone sample, the effective stress coefficient presents values lower than 1.7. The carbonate sample exhibits a coefficient of effective stress in the range of 1.32 and 1.90. In the shale cases, the coefficient ranges from 1.18 to 1.87. For the sandstone coefficient's average value is around 1.3; for carbonate, it is around 1.6, while the average value for shales 1, shale 3 and shale 5 is 1.5. The average effective stress coefficient for shale 2, shale 4 and shale 6 is 1.3, 1.2, and 1.6, respectively. In literature, values larger than one for the effective stress coefficient have been previously been reported. Zoback and Byerlee (1975) demonstrated that some sandstones' effective stress coefficient could be as high as 3 to 4. Walls and Nur (1979) found effective stress coefficient values for sandstone samples varying from 1.2 to 7.1. In terms of shale samples, Ma and Zoback (2016) found effective stress coefficient for Bakken formation samples in the range of 1 to 6.



Effective Stress, Pc-n\*Pp, Psi

Figure 6. Estimated effective stress coefficient n for sandstone (TOP), and carbonate (BOTTOM) samples as a function of effective stress.



Effective Stress, Pc-n\*Pp, Psi





Effective Stress, Pc-n\*Pp, Psi

Figure 8. Estimated effective stress coefficient *n* for shale 3 (TOP) and shale 4 (BOTTOM) samples as a function of effective stress.



Figure 9. Estimated effective stress coefficient *n* for shale 5 (TOP) and shale 6 (BOTTOM) samples as a function of effective stress.

In **Figure 6** and **Figure 7**, we can observe that, when different confinement pressure values are used, and confinement pressure is considered a third variable, several trends can be identified. Similar results were reported by Aldana and Akkutlu (2019). These trends indicate that confinement pressure has a dominant effect on the plug samples. For the carbonate case (Figure 4, Bottom), having a constant coefficient *n* at low (500-1500 psi) and high (>2000 psi) confinement pressures is producing separate trends meaning the plug is responding mechanically in a different way. A direct relationship between the confinement pressure and the effective stress coefficient is observed, as generally, the higher the confinement pressure is, the higher the effective stress coefficient. This trend was also observed in the case of the shale 1 and shale 2 samples (Figure 5), where the effective stress coefficient slightly increases with a higher confinement pressure. Sandstone plug (Figure 4, Top) also shows distinct trends for the coefficient when confinement pressure is considered a third variable, and its effective stress coefficient is almost constant when plotted as a function of the effective stress.

For the case of the coefficient of the isothermal pore compressibility,  $C_p$ , Figures 10-13, show the estimated  $C_p$  values. The average pore compressibility is  $2.9 \times 10^{-6}$  1/psi for sandstone, and  $7.7 \times 10^{-5}$  1/psi for carbonate sample. For the shale cases the isothermal pore compressibility is  $4.7 \times 10^{-6}$  1/psi for Shale 1,  $2.3 \times 10^{-6}$  for the Shale 2,  $3.3 \times 10^{-6}$  for the Shale 3 and Shale 6,  $4 \times 10^{-6}$  for the Shale 4 and  $1.2 \times 10^{-6}$  for the Shale 5. Higher pore compressibility values are associated with higher values of pore pressure applied to the sample in the measurements. The pore compressibility values vary one order of magnitude between  $1.0*10^{-5}$  - $1.0*10^{-6}$  1/psi. However, the pore compressibility is a stress-dependent

property, and when the pore pressure is increased at each pressure step of the gas-uptake measurement, the effective stress applied to the sample varies and, consequently, the pore compressibility for certain samples may change. (Santos & Akkutlu, 2013).

In the last decades, different authors have determined pore compressibility values in the range of  $10^{-6}$  1/psi for the shale samples. Zhou and Ghasemi (2020) estimated pore compressibility values for a shale sample in the order of 2.842x10<sup>-5</sup> 1/psi to 8.88 x10<sup>-6</sup> 1/psi. Kang et al. (2011) and Kang et al. (2014) measured pore compressibility values between 9x10<sup>-5</sup> and 2x10<sup>-6</sup> psi<sup>-1</sup> using a gas permeameter. Santos and Akkutlu (2013) estimated values between the range of 5.41x10<sup>-6</sup> 1/psi and 4.35x10<sup>-5</sup> 1/psi for shale samples using a novel approach based on a new analytical model of total gas storability developed to interpret the measured multiple-step pressure data on a graphical domain in which the storage-parameter estimation can be performed fast and accurately with a straight line. Shale samples may not be considered rigid but geomechanically sensitive rocks, as their pore compressibility values vary depending on the effective stress that acts upon the samples within the core holder during the measurements (Kang et al. 2014).



Effective Stress, Pc-n\*Pp, Psi

Figure 10. Estimated pore compressibility for the sandstone (TOP), and carbonate (BOTTOM) samples as a function of effective stress.



Effective Stress, Pc-n\*Pp, Psi

Figure 11. Estimated pore compressibility for shale 1 (TOP) and shale 2 (BOTTOM) samples as a function of effective stress.



Figure 12. Estimated pore compressibility for shale 3 (TOP) and shale 4 (BOTTOM) samples as a function of effective stress.



Figure 13. Estimated pore compressibility for shale 5 (TOP) and shale 6 (BOTTOM) samples as a function of effective stress.

For the case of the sandstone, Sampath (1982) estimated a pore compressibility ranged between 2  $\times 10^{-6}$  1/psi and 1.34  $\times 10^{-5}$  1/psi. measured. Khatchikian (1996) found pore compressibility values for sandstone between 5  $\times 10^{-6}$  and 8  $\times 10^{-6}$ . Therefore, our results for isothermal coefficient of pore compressibility are between the common range found in the literature. Pore compressibility is fundamental to calculate the storage capacity of our samples and an accurate measurement of this property is required. Hall (1953) emphasize the importance of pore compressibility in calculating the hydrocarbon volume of a reservoir, stating that neglect of pore compressibility may result in significant errors, particularly for low-porosity reservoir rocks.

Once the values of isothermal coefficient of pore compressibility and effective stress coefficient were obtained, the pore volume of the samples can be estimated with **Equation 37**. The pore volumes can be observed as a function of effective stress in **Figures 14-17**. **Figures 14-17** show than when effect stress changes, sandstone and carbonate samples exhibit small variations in their pore volume. Therefore, the pore volume is insensitive to stress changes throughout measurements for sandstone, carbonate and shale samples. Due to the pore volume does not show significant variations when subjected to the different effective stress values during the measurements, the porosity is not expected to change substantially as a function of effective stress. This can be observed in **Figures 20-21**, where the shale 3, shale 4, shale 5 and shale 6 samples do not exhibit significant variations in the porosity as the effective stress changes. However, for the sandstone, carbonate, shale 1 and shale 2 samples a slight increment is observed in the porosity values as the effective stress.



Effective Stress, Pc-n\*Pp, Psi

Figure 14. Estimated pore volume as a function of stress for sandstone (TOP) and carbonate (BOTTOM) samples as a function of effective stress.



Effective Stress, Pc-n\*Pp, Psi

Figure 15. Estimated pore volume as a function of stress for shale 1 (TOP), and shale 2 (BOTTOM) samples as a function of effective stress.



Figure 16. Estimated pore volume as a function of stress for shale 3 (TOP), and shale 4 (BOTTOM) samples as a function of effective stress.



Figure 17. Estimated pore volume as a function of stress for shale 5 (TOP), and shale 6 (BOTTOM) samples as a function of effective stress.



Effective Stress, Pc-n\*Pp, Psi



Effective Stress, Pc-n\*Pp, Psi

Figure 18. Estimated porosity as a function of stress for sandstone (TOP) and carbonate (BOTTOM) samples as a function of effective stress.



Effective Stress, Pc-n\*Pp, Psi

Figure 19. Estimated porosity as a function of stress for Shale 1 (TOP) and Shale 2 (BOTTOM) samples as a function of effective stress.



Figure 20. Estimated porosity as a function of stress for Shale 3 (TOP) and Shale 4 (BOTTOM) samples as a function of effective stress.



Figure 21. Estimated porosity as a function of stress for Shale 5 (TOP) and Shale 6 (BOTTOM) samples as a function of effective stress.

Finally, **Figure 22-25** show the estimated effective stress coefficients as a function of the applied stress potential parameter,  $a = p_c/np_p$ . The figure exhibits a high correlation between the data points and the straight line. The fact that the applied stress parameter is a function of both the confining pressure and the pore pressure applied, in **Figures 22-25** we identify that the effective stress coefficient is not strictly equal to the Biot's coefficient (a mechanical property of the rock sample) but is also influenced by the applied laboratory conditions. The relationship between the stress coefficient *n* and the applied stress potential a is linear. Then, the Biot's coefficient can be extracted from the y-intercept of the observed straight line, when the effect of the applied laboratory conditions disappears.

As the intercept, the Biot's coefficient of the sandstone sample is obtained 0.69. In the case of the carbonate sample, the estimated Biot's coefficient is 0.98. For the shale samples, the linear trend persists, and the Biot's coefficient are 0.97, 0.88, 0.92, 0.46, 0.97 and 0.92 for shale 1, shale 2, shale 3, shale 4, shale 5, and shale 6 samples, respectively. The Biot's coefficient for Shale 4 is significantly lower than the other shale samples, which could indicate significant lamination and fissility related to shale's nature. However, it could also suggest a different cementation, pore geometry, and mineral constituents of the sample. Biot's coefficient may differ for different material properties of the same rock, but it generally takes values lower than 1 (Nur and Byedee, 1971; Robin, 1973; Garg and Nur, 1973; Paterson, 1978; Kranz et al., 1979; Walsh, 1981; Berryman, 1992).



Parameter a

Figure 22. Estimated effective stress coefficient for the sandstone (TOP), and carbonate (BOTTOM) samples as a function of constant a.


Figure 23. Estimated effective stress coefficient shale 1 (TOP) and shale 2 (BOTTOM) samples as a function of constant a.



Figure 24. Estimated effective stress coefficient shale 3 (TOP) and shale 4 (BOTTOM) samples as a function of constant a.



Figure 25. Estimated effective stress coefficient shale 5 (TOP) and shale 6 (BOTTOM) samples as a function of constant a.

The obtained Biot's coefficients are less than the unity, in between 0.65-1.0, which is a range commonly found in the literature performing mechanical tests. Alam et al (2010) determined Biot's coefficient values between 0.9 and 1 for deep sea carbonate rock. He and Ling (2014) found Biot's coefficient between the ranges of 0.57 an 0.7 for Bakken samples by a measurement method in which the variation of the confining pressure used to keep the volume of sample constant is recorded, while altering the pore pressure. Heller et al (2014) reported a Biot's coefficient of 0.68 for Barnet Shale samples. Ling et al (2016), by the use of three different methods, determined an average Biot's coefficient for 9 samples from Bakken formation between 0.57 and 0.87. The three methods implemented were the bulk and matrix compressibility (conventional) method, permeability-variationwith- pressure method (Qiao et al, 2012), and constant deformation method (He and Ling, 2014). With bulk and matrix compressibility method the reported Biot's coefficient values were between 0.55 and 0.91. Using the permeability-variation-with-pressure method, Biot's coefficient varied from 0.57 to 0.86, and with the constant deformation method, the coefficient was reported between 0.58 and 0.87.

For sandstone samples, Franquet and Abass (1999) obtained Biot's coefficient for between 0.73 and 0.87 with their proposed method based in bulk and grain modulus. In 2004, Keaney et al estimated an average value of Biot's coefficient for sandstone samples equal to 0.75. Qiao et al (2009) found average values of Biot's coefficient for sandstone 0.701 during permeability measurements. Li et al (2020) reported Biot's coefficient for sandstones, between 0.7 and 0.8 using a correlation for estimating Biot's coefficient in conventional and unconventional reservoirs on the basis of knowledge of the ratio of permeability to porosity  $(k/\phi)$  and pore-throat radii. Hampton and Boitnott (2018) and Civan (2021) showed Biot's coefficient values for sandstone between 0.62 and 0.74.

As the final step of the analysis, the linear relation (n = slope \* a +  $\alpha$ ) observed in **Figures 22-25** can be substituted into the effective stress equation ( $\sigma_e = P_c - nP_p$ ) obtaining the following expression:

$$\sigma_e = \left(1 - \frac{slope}{n}\right) p_c - \alpha p_p \tag{44}$$

**Equation 44** is the resultant expression for effective stress that can be implemented by the use of the experimental procedure and the analysis method proposed in this study. This is remarkable because we obtain the coefficient using the conventional porosimeter setup without the need for sophisticated geomechanical tests such as the triaxial compaction test.

It was noted that for the analyzed samples, the parenthesis term received values that are comparable to the Biot coefficient. In the case this fundamental observation is valid, one final modification to this equation can be made as follows:

which is indicating that the applied differential pressure can be used as the effective stress, when the difference is multiplied with the Biot coefficient. In essence,

being a fractional value, the Biot coefficient becomes the necessary correction to the applied differential pressure.

#### 5. CONCLUSIONS

This study fulfilled its objective of developing a new method for Biot's coefficient estimation in the laboratory during porosity measurements under stress. Biot's coefficient is fundamental for reservoir engineering, drilling, wellbore stability, hydraulic fracturing, and fracture design, and therefore its determination is essential in petroleum engineering. The coefficient was estimated for sandstone, carbonate, and shale samples. Additionally, the isothermal coefficient of pore compressibility, effective stress coefficient, and pore volume were evaluated for the analyzed samples.

Biot's coefficient for the sandstone sample is 0.69. It is 0.98 for the case of the carbonate sample. For the shale samples, the coefficient ranges from 0.46 to 1. The obtained Biot's coefficients are less than the unity, in between 0.46-1.0, which is a range commonly found in the literature performing mechanical tests. Further measurements using a larger set of samples are necessary for a detailed analysis of Biot's coefficient.

The sandstone effective stress coefficient's average value is around 1.3; for carbonate, it is approximately 1.6, while the average values for shale sample range from 1.2 to 1.6. The values reported for effective stress coefficient are similar to those reported in the literature implementing different techniques.

In terms of the isothermal pore compressibility,  $C_{p,.}$  the average pore compressibility is 2.9 x10<sup>-6</sup> 1/psi for sandstone, 7.7 x 10<sup>-6</sup> 1/psi for carbonate, and between 1.29 x 10<sup>-6</sup> 1/psi to 4.7 x10<sup>-6</sup> 1/psi for the shale samples. Higher pore compressibility values

are associated with higher pore pressure values applied to the samples in the measurements.

The new method allows measurements and analysis relatively fast. And it can be performed in every reservoir petrophysics laboratory since Biot's coefficient is obtained using the conventional porosimeter setup without the need for sophisticated geomechanical tests such as the tri-axial compaction test.

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## APPENDIX A

## TABULATED EXPERIMENTAL DATA

Pc	Pri	Pdi	Psi	Pf
psi	psi	psi	psi	psi
1000	102.9	14.7	14.7	67.2
1000	282.0	177.7	177.7	239.6
1000	344.5	239.7	239.7	302.0
1000	405.4	301.5	301.5	364.3
1500	516.2	423.3	423.3	477.9
1500	615.2	530.2	530.2	565.1
1500	666.2	582.1	582.1	630.3
1500	714.3	632.2	632.2	679.5
1500	816.4	731.8	731.8	781.5
1500	913.9	834.6	834.6	880.2
1500	1012.4	934.6	934.6	978.5
2000	1065.3	980.4	980.4	1029.7
2000	1121.3	1031.2	1031.2	1083.2
2000	1163.5	1084.0	1084.0	1131.1
2000	1266.7	1181.7	1181.7	1230.9
2000	1314.6	1234.0	1234.0	1279.9
2000	1365.2	1282.3	1282.3	1329.5
2000	1414.0	1332.3	1332.3	1379.0
2000	1465.3	1381.6	1381.6	1430.0
2000	1513.9	1431.8	1431.8	1478.8
2500	1513.9	1431.8	1431.8	1478.8
2500	1570.2	1481.5	1481.5	1532.5
2500	1614.9	1533.3	1533.3	1581.5
2500	1660.4	1581.6	1581.6	1627.3
2500	1711.3	1628.3	1628.3	1676.4
2500	1734.5	1676.2	1676.2	1709.6
3000	1563.4	1518.0	1518.0	1543.2
3000	1613.2	1546.1	1546.1	1584.0
3000	1664.6	1585.9	1585.9	1631.0
3000	1715.8	1634.0	1634.0	1680.7
3500	1550.9	1498.0	1498.1	1527.4
3500	1609.0	1532.2	1532.2	1575.5
3500	1640.6	1579.4	1579.4	1616.7

# Table 6. Laboratory data for sandstone sample.

Рс	Pri	Pdi	Psi	Pf
psi	psi	psi	psi	psi
500	98.9	14.7	14.7	55.8
500	198.2	55.8	55.8	108.0
1000	704.5	427.3	427.3	508.9
1500	766.3	238.1	238.1	425.9
1500	917.7	425.9	425.9	606.3
2000	922.3	177.8	177.8	457.9
2000	1113.6	457.9	457.9	570.9
2500	1114.7	413.7	413.7	646.2
3000	827.8	161.9	161.9	400.0
3000	1164.2	400.0	400.0	668.7

 Table 7. Laboratory data for carbonate sample.

Table 8. Laboratory data for shale 1 sample.

Рс	Pri	Pdi	Psi	Pf
psi	psi	psi	psi	psi
600	165.7	14.6	14.6	122.5
1500	592.9	150.6	150.6	441.7
1500	789.3	441.74	441.74	709.8
1500	1029.5	709.8	709.8	929.3
2200	1482.6	1004.6	1004.6	1332.5
2700	1242.6	1043.5	1043.5	1174.1
2700	1430.6	1174.1	1174.1	1355.1
3200	1141.6	914.7	914.7	1067.1
4200	1353.7	1024.5	1024.5	1269.1
4750	1520.9	1390.7	1390.7	1479.7
4990	1424.6	940.1	940.1	1305.7
4990	1630	1305.7	1305.7	1531.4

Рс	Pri	Pdi	Psi	Pf
psi	psi	psi	psi	psi
600	179.6	14.6	14.6	122.6
900	604.5	122.9	122.9	413.5
1200	844	407.7	407.7	682.2
1800	1435.9	948.2	948.2	1257.4
2100	1760.4	947.7	947.7	1468.3
2400	1775.4	1456.7	1456.7	1655.3
2700	1759.4	1648.7	1648.7	1715.3
3000	1778.1	1708.7	1708.7	1745.2
3600	1766.9	1755.7	1755.7	1755.9
4500	1764.8	1741.7	1741.7	1744.2
4800	1761.9	1744.7	1744.7	1748.9
5000	1768.9	1748.7	1748.7	1754.2

Table 9. Laboratory data for shale 2 sample.

Table 10. Laboratory data for shale 3 sample.

Рс	Pri	Pdi	Psi	Pf
psi	psi	psi	psi	psi
5014	2744.8	14.0	14.0	1316.2
5014	3242.9	1289.4	1289.4	2138.2
5014	3225.2	2112.1	2112.1	2600.5
5014	3454.5	2580.7	2580.7	2964.9
5014	3403.2	2945.1	2945.1	3144.5

Рс	Pri	Pdi	Psi	Pf
psi	psi	psi	psi	psi
5014.0	3683.0	2022.3	2022.3	2618.3
5014.0	3741.1	2541.2	2541.2	3000.8
5014.0	3957.4	2901.0	2901.0	3328.3
5014.0	4432.0	3183.5	3183.5	3690.3
5014.0	4502.1	3381.9	3381.9	3857.5

Table 11. Laboratory data for shale 4 sample.

 Table 12. Laboratory data for shale 5 sample.

Рс	Pri	Pdi	Psi	Pf
psi	psi	psi	psi	psi
5014.0	1793.5	21.4	21.4	1226.4
5014.0	2283.3	1218.4	1218.4	1800.0
5013.9	2480.7	1937.3	1937.3	2500.0
5014.1	2980.7	2379.3	2379.3	2700.0

Table 13. Laboratory data for shale 6 sample.

Рс	Pri	Pdi	Psi	Pf
psi	psi	psi	psi	psi
5014.3	1560.1	20.1	20.1	975.6
5014.3	1935.5	974.3	974.3	1578.3
5014.3	2324.2	1570.7	1570.7	2053.3
5014.3	2705.0	2052.1	2052.1	2477.4
5014.3	3252.3	2472.2	2472.2	2936.9

### APPENDIX B TABULATED LABORATORY RESULTS

Below, are the laboratory results obtained using the presented method of gas storage measurements.

Pc	$\mathbf{P_{f}}$	n	Effective Stress, P <sub>c</sub> - n*P <sub>f</sub>	$\mathbf{V}_{\mathbf{p}}$	Ср
psi	psi	-	psi	сс	1/psi
1000	239.6	1.700	592.68	2.742	1.82E-06
1000	302.0	1.532	537.34	2.742	1.70E-06
1000	364.3	1.538	439.71	2.743	1.85E-06
1500	477.9	1.484	790.59	2.742	1.12E-06
1500	565.1	1.447	682.30	2.745	2.34E-06
1500	630.3	1.451	585.43	2.752	4.84E-06
1500	679.5	1.416	537.98	2.743	1.18E-06
1500	781.5	1.371	428.56	2.752	4.16E-06
1500	880.2	1.305	351.21	2.748	2.59E-06
1500	978.5	1.196	329.36	2.756	4.99E-06
2000	1029.7	1.318	642.73	2.750	2.75E-06
2000	1083.2	1.361	525.23	2.747	1.71E-06
2000	1131.1	1.338	487.14	2.746	1.54E-06
2000	1230.9	1.196	528.21	2.749	2.21E-06
2000	1279.9	1.191	475.14	2.749	2.28E-06
2000	1329.5	1.157	462.06	2.759	4.67E-06
2000	1379.0	1.154	408.37	2.750	2.28E-06
2000	1430.0	1.130	384.26	2.753	2.88E-06
2000	1478.8	1.127	333.81	2.747	1.46E-06
2500	1478.8	1.217	699.69	2.754	2.92E-06
2500	1532.5	1.198	663.69	2.755	3.11E-06
2500	1581.5	1.178	636.34	2.755	2.94E-06
2500	1627.3	1.145	636.01	2.770	5.95E-06
2500	1676.4	1.160	554.56	2.752	2.20E-06
2500	1709.6	1.245	372.31	2.765	4.30E-06
3000	1543.2	1.279	1025.48	2.756	2.98E-06
3000	1584.0	1.263	1000.05	2.759	3.44E-06
3000	1631.0	1.258	948.93	2.753	2.27E-06
3000	1680.7	1.337	752.94	2.762	3.53E-06
3500	1527.4	1.430	1315.89	2.752	2.00E-06
3500	1575.5	1.322	1417.25	2.767	4.77E-06

Table 14. Gas storage measurement results for the sandstone sample.

Pc	P <sub>f</sub>	n	Effective Stress, P <sub>c</sub> - n*P <sub>f</sub>	Vp	Ср
psi	psi	-	psi	сс	1/psi
500	55.8	1.799	399.62	3.281	5.97E-06
500	108.0	1.644	322.45	3.283	5.04E-06
1000	508.9	1.321	327.58	3.298	8.44E-06
1500	425.9	1.555	837.73	3.298	8.71E-06
1500	606.3	1.420	639.05	3.300	7.24E-06
2000	457.9	1.625	1255.91	3.299	7.86E-06
2000	570.9	1.553	1113.16	3.305	8.76E-06
2500	646.2	1.585	1475.85	3.307	8.09E-06
3000	400.0	1.898	2240.80	3.299	8.04E-06
3000	668.7	1.630	1909.85	3.310	8.71E-06

Table 15. Gas storage measurement results for the carbonate sample.

Table 16. Gas storage measurement results for the shale 1 sample.

Pc	P <sub>f</sub>	n	Effective Stress, P <sub>c</sub> - n*P <sub>f</sub>	Vp	Ср
psi	psi	-	psi	сс	1/psi
600	122.5	1.721	389.19	1.058	9.96E-06
1500	441.7	1.540	819.59	1.060	5.78E-06
1500	709.8	1.360	534.64	1.057	1.40E-06
1500	929.3	1.215	371.06	1.086	2.54E-05
2200	1332.5	1.386	352.79	1.060	1.97E-06
2700	1174.1	1.451	996.75	1.058	1.15E-06
3200	1067.1	1.481	1620.15	1.059	2.07E-06
3700	1081.7	1.600	1969.17	1.056	1.41E-07
4200	1269.1	1.555	2226.55	1.058	1.04E-06
4750	1479.7	1.573	2421.78	1.059	1.24E-06
4990	1305.7	1.673	2805.65	1.060	1.78E-06

Pc	Pf	n	Effective Stress, P <sub>c</sub> - n*P <sub>f</sub>	Vp	Ср
psi	psi	-	psi	сс	1/psi
600	122.6	1.660	396.48	0.991	4.84E-06
1200	682.2	1.273	331.65	0.992	2.80E-06
1800	1257.4	1.173	325.18	0.993	1.99E-06
2100	1468.3	1.172	379.15	0.994	2.23E-06
2400	1655.3	1.177	452.29	0.993	1.38E-06
2700	1715.3	1.256	545.58	0.994	1.91E-06
3000	1745.2	1.255	809.77	0.993	1.19E-06
3600	1755.9	1.374	1186.57	0.993	1.26E-06
4500	1744.2	1.448	1974.92	0.999	3.54E-06
4800	1748.9	1.458	2249.99	0.995	2.14E-06

Table 17. Gas storage measurement results for the shale 2 sample.

Table 18. Gas storage measurement results for the shale 3 sample.

Pc	P <sub>f</sub>	n	Effective Stress, P <sub>c</sub> - n*P <sub>f</sub>	Vp	Ср
Psi	psi	-	psi	сс	1/psi
5014	2618.3	1.290	1636.35	0.652	1.01E-06
5014	3000.8	1.220	1353.06	0.653	1.01E-06
5014	3328.3	1.201	1016.70	0.674	9.38E-06
5014	3690.3	1.113	906.71	0.662	4.64E-06

Pc	Pf	n	Effective Stress, P <sub>c</sub> - n*P <sub>f</sub>	Vp	Ср
Psi	psi	-	psi	сс	1/psi
5014	2618.3	1.290	1636.35	0.652	1.01E-06
5014	3000.8	1.220	1353.06	0.653	1.01E-06
5014	3328.3	1.201	1016.70	0.674	9.38E-06
5014	3690.3	1.113	906.71	0.662	4.64E-06

Table 19. Gas storage measurement results for the shale 4 sample.

Table 20. Gas storage measurement results for the shale 5 sample.

Pc	P <sub>f</sub>	n	Effective Stress, P <sub>c</sub> - n*P <sub>f</sub>	Vp	Ср
psi	psi	-	psi	сс	1/psi
5014	1226.4	1.675	2960.06	0.200	1.00E-06
5014	1800.0	1.523	2272.58	0.201	1.34E-06
5014	2500.0	1.422	1458.19	0.201	1.09E-06
5014	2700.0	1.330	1423.14	0.201	1.33E-06

Table 21. Gas storage measurement results for the shale 6 sample.

Pc	P <sub>f</sub>	n	Effective Stress, P <sub>c</sub> - n*P <sub>f</sub>	Vp	Ср
psi	psi	-	psi	сс	1/psi
5014	975.6	1.873	3186.90	0.468	1.56E-06
5014	1578.3	1.671	2376.97	0.474	5.44E-06
5014	2053.3	1.430	2078.05	0.472	3.43E-06
5014	2477.4	1.410	1521.14	0.472	2.78E-06