



More than 60% of the United States natural gas production is now attributed to shale reservoirs [1]. This proportion is only expected to grow as more shale reservoirs are discovered and unlocked. The total natural gas content in a shale consists of both absorbed gas in the porous spaces of the shales and adsorbed gas on the surface of the shale matrix. Adsorbed gas can contribute a significant fraction (~50%) of the total gas in place in a shale reservoir.

The total gas in place is dependent on the pore pressure and temperature and is vital to the profitable development of a shale reservoir [2]. Gas companies use gas isotherms, which are a measure of total gas content as a function of pore pressure, to assess a reservoir's profitability. Traditionally, gas isotherms are measured by exposing the core to helium and methane at ever increasing pressure while tracking the volume of gas absorbed and adsorbed. These experiments involve destruction of the core and provide no information on the pore size distribution.

In this application note, we present a method for measuring gas isotherms in shales using NMR. Measurements of the T_2 relaxation time were taken for 24 hours after the introduction of methane to a shale. These measurements were then used to determine the total gas content and pore size distributions within the rock. This NMR gas isotherm analysis proves to be advantageous over conventional techniques as it can be completed without destruction of the shale core while also providing pore size distributions.

Method

A shale core plug was confined hydrostatically by fluorinert to a pressure of 2500 psi in an Oxford Instruments P5 overburden NMR probe [3] in an Oxford Instruments **GeoSpec 2-75** rock core analyzer [4]. The sample was evacuated with a vacuum pump and then dry T_2 measurements were taken. Methane was then introduced to the sample at 500 psi. T_2 measurements were acquired at 2 minute intervals for the first hour, then at 15 minute intervals for the following 3 hours, then at 60 minute intervals until a total experimental time of 24 hours. The experiment was then repeated with methane pressures of 1000, 1500 and 2000 psi. The T_2 measurements were employed to retrieve the total gas content present in each rock as a function of time. Data acquisition and analysis of the T_2 data was achieved via Green Imaging Technologies software [5]. The pore volume of the shale was determined using the NMR volume from a T_2 measurement of the fully saturated shale. The shale was saturated after being submerged in brine at 10,000 psi in a pressure cell for three days.



Using T_2 distributions to measure gas isotherms in shales



Results

The volume of gas detected at each pressure was derived for every T_2 measurement acquired during the 24-hour test period using the area under the curve of each T_2 distribution. The volume of gas detected was then converted to total gas content using the hydrogen index of methane, the bulk volume of the rock, the experimental temperature and pressure, as well as a standard temperature of 292 K and pressure of 1 atmosphere.

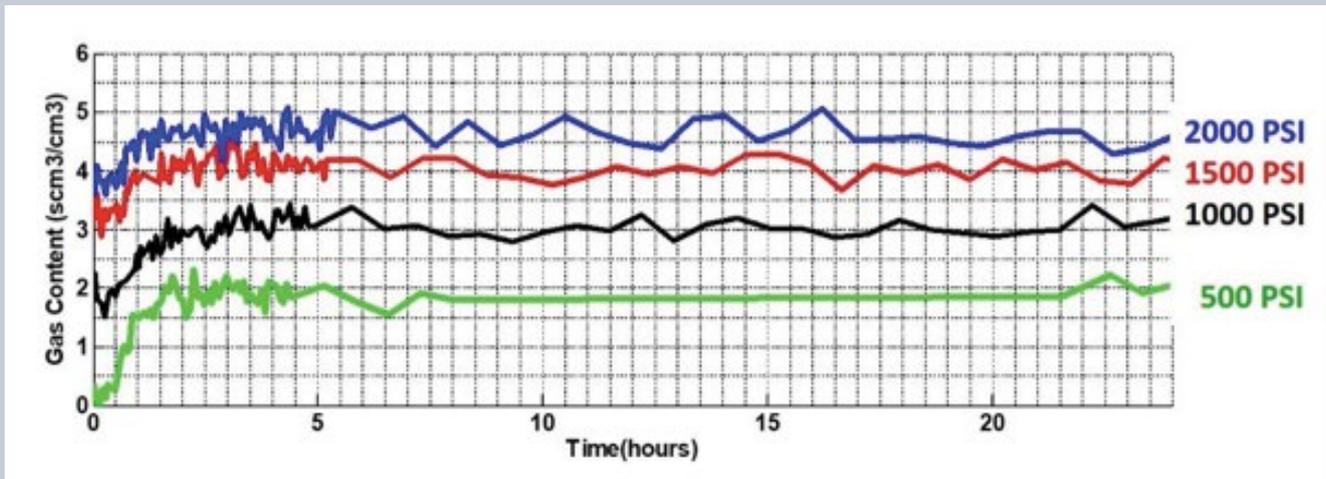
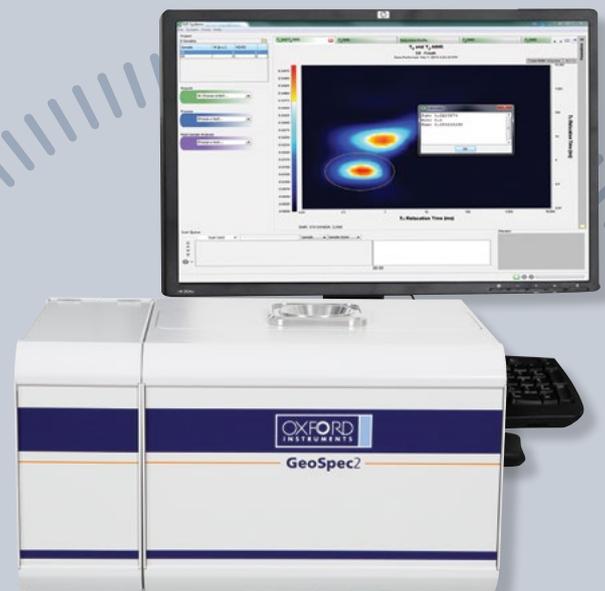


Figure 1: Total gas content of methane at 500, 1000, 1500 and 2000 psi.

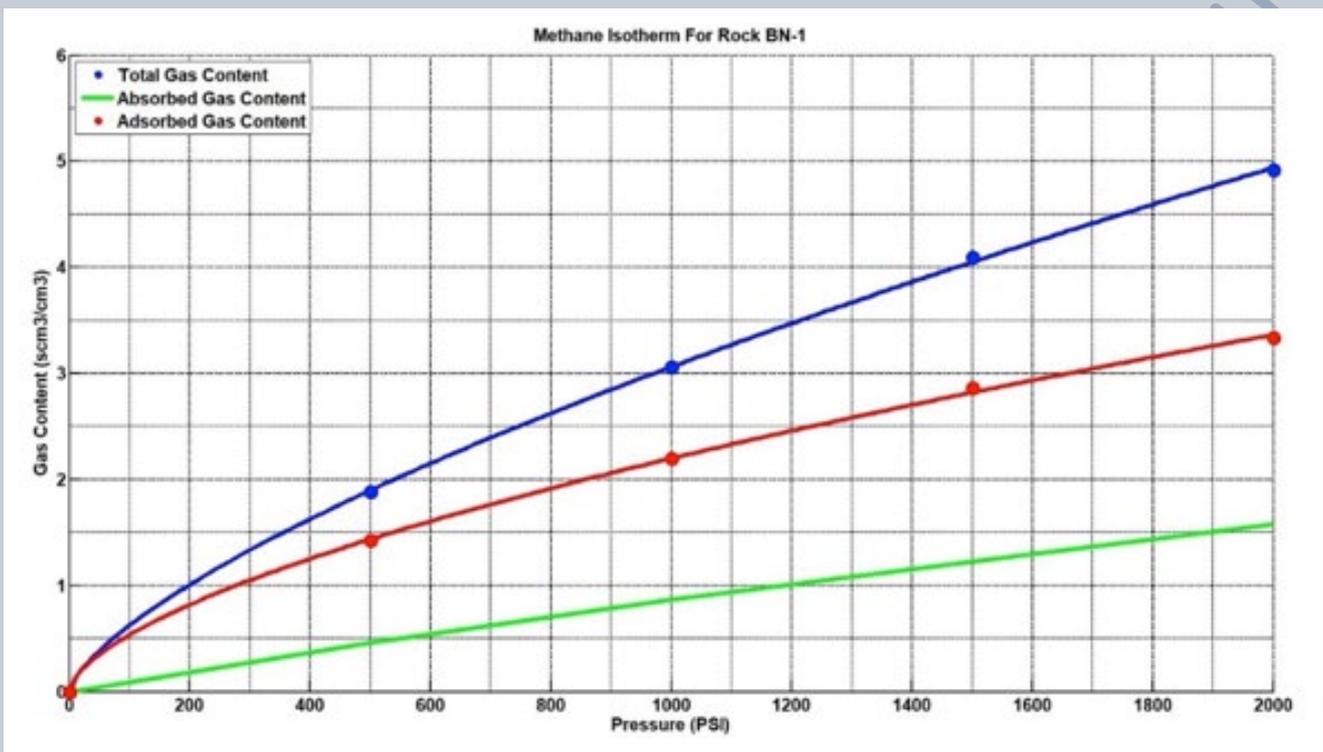
The total gas content of the shale for each pressure as a function of time is shown in Figure 1. This figure shows that it takes two to four hours for the methane to fully infiltrate the shale core plug. Following this initial stabilization, the gas content is constant over the remainder of the experiment. Other experiments, with other types of rocks, have shown that this stabilization period is rock dependent.



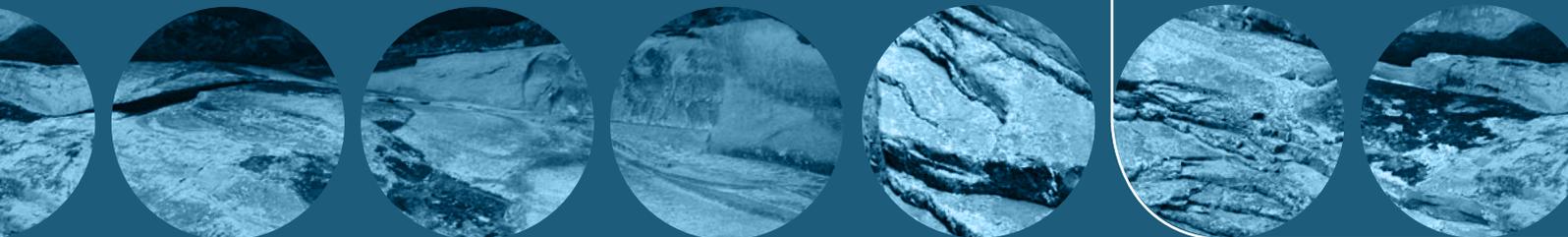


To generate the total gas isotherm, the average total gas content was determined for the shale at 500, 1000, 1500 and 2000 psi. The average was taken from four hours onwards, after the methane content in the rock had stabilized. The resulting total gas isotherm is plotted in Figure 2 (blue trace). The isotherm has the expected curved appearance as a function of pressure, a result of the competing mechanisms of adsorption and absorption.

Figure 2: Total gas, adsorbed gas, and absorbed gas isotherms of methane in a shale.



To fully characterize the shale gas reservoir, the total gas content should be separated into its contributions from these competing mechanisms. To do this, the absorbed gas isotherm (Figure 2 – green trace) was calculated using the Van der Waals equation and the pore volume of the rock. The adsorbed gas isotherm (Figure 2 – red trace) was then derived by subtracting the absorbed gas isotherm from the total gas isotherm.



Conclusion

A method has been presented for measuring gas isotherms in shales using NMR. The method is similar to the conventional approach in that methane is introduced to a shale at a number of pressures. Pore volume is determined using NMR rather than through the introduction of helium to the sample. T_2 distributions are acquired at each pressure to determine the total gas content. The advantages of the NMR method are that the shale sample is preserved and pore size distributions are acquired with each T_2 measurement, as shown in Figure 3.

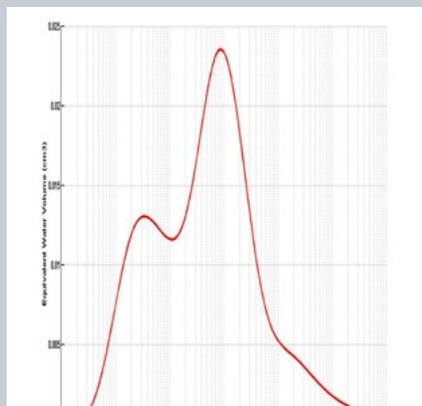


Figure 3: Typical gas pore size distribution

References

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