

**A MULTI-LABORATORY COMPARISON OF ISOTHERM MEASUREMENTS
ON ANTRIM SHALE SAMPLES, OTSEGO COUNTY, MI**

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SUMMARY

This paper summarizes and compares isotherms measured by six independent laboratories on four Antrim Shale samples taken from the Nomeco Bagley East B3-11 well in Otsego County, MI. The project was initiated and coordinated by the Gas Research Institute (GRI), with the primary objective of generating a set of isotherm data measured on comparable samples to determine what, if any, differences might exist between different laboratories' methods and results. To our knowledge, this is the first multi-laboratory comparison of isotherm measurements ever conducted in a systematic way.

Despite giving each laboratory identical instructions and similar samples, the initial isotherm measurements received from the labs were confusing and varied widely. Some labs reported adsorbed methane content only, while others reported total methane content, including gas stored in any free pore spaces. Some labs ran the samples dry, while others conducted the measurements at "as received" conditions; given their reports, it was unclear how the samples had been tested. There were even important differences in nomenclature - i.e., not every lab agreed on standard conditions or whether reporting the results in "scf/ton" meant U.S. or metric ton.

In addition to these problems, the comparative isotherm measurements revealed other important differences between laboratory methods that can be potential sources of error. These included (1) sample preservation and preparation, (2) cell calibration, (3) temperature measurement and control, (4) measurement of bulk density, (5) how (or if) equilibrium is determined, and (6) how to establish equilibrium moisture content. On the basis of the tests conducted by these six laboratories, it is clear that no standard protocol exists for measuring isotherms on shale samples in the laboratory.

Despite these problems, however, most of the isotherms compared reasonably well, once the reporting differences were resolved. Still, isotherms measured at "similar" conditions may vary by as much as 25 to 50% among different labs. These results demonstrate how important it is for the user of the isotherm data to understand how the data were collected and what is being reported. Using such data incorrectly can result in significant errors in estimates of gas-in-place and long-term well performance.

BACKGROUND

In recent years, there has been considerable interest in the development of coalbed methane and gas shales reservoirs. The most active coalbed methane plays have been in the Warrior Basin in Alabama and the San Juan Basin in the area of northwestern New Mexico and southwestern Colorado. Operators have also pursued the development of gas shales in the historic Devonian Shales reservoir of the Appalachian Basin (Kentucky, West Virginia, and Ohio) in the Barnett Shale in the Fort Worth Basin of Texas, and in the Antrim Shale in Michigan. The Gas Research Institute (GRI) has actively conducted formation evaluation, completion, and stimulation research in each of these areas during the past 10 to 12 years.¹⁻⁵

Both coals and gas shales are typically naturally fractured, highly organic formations in which a significant quantity of the gas in place stored in the reservoir is adsorbed to the organic material. In coals, essentially all of the gas in place may be attributed to adsorption; there is generally considered to be almost no free porosity in the coal matrix itself. In gas shales, on the other hand, gas may be stored as free gas in the matrix or fracture porosity and as adsorbed gas on the organic and clay particles. Recent findings have also suggested that some of the gas may actually be stored in solution in oil or bitumen present in the shale. Adsorption may account for as much as 20% to 85% of the gas in place in gas shales, depending on the matrix porosity, the quantity of organic material present, the initial reservoir conditions, and other factors.

Because adsorbed gas makes up such a large fraction of the gas in place in these reservoirs, the knowledge of the adsorbed gas content and how this adsorbed gas is released as a well is produced are critical to making accurate reserves estimates and long-term production forecasts. Both parameters, the gas content and the adsorption/desorption isotherm, are key input values in simulators developed for coalbed methane and gas shales reservoirs. The most reliable method for determining gas content and isotherm data is to measure them in the laboratory using core samples taken from the well of interest. Because these data are so important to well performance estimates, it is important that the engineer using the data understand how these properties are measured and how the results are reported.

In May 1991, GRI hosted a meeting of industry experts and researchers addressing the topic of improved formation evaluation in coalbed methane and gas shales reservoirs. A key topic discussed at that meeting was the different techniques and procedures used by various laboratories in making gas content and isotherm measurements. From the discussion, it was clear that there were no industry-standard procedures for conducting these measurements. The multi-laboratory isotherm comparisons described in this paper are a direct result of those discussions. GRI initiated and coordinated these experiments during 1992, with the primary objective of generating a set of isotherm data measured on comparable samples under similar conditions to determine what, if any, differences might exist between different laboratories' methods and results. The intent of this project was not to be a "competition" among the various labs; rather we hoped it would be a learning experience for everyone - those who measure the isotherms and those who use them.

TEST DESCRIPTION

Six independent laboratories participated in the isotherm measurement comparisons. Four were university laboratories, one was a commercial core analysis laboratory, and one was the petrophysical laboratory of a major oil company. Five of the six laboratories measured isotherms volumetrically, where the amount of gas adsorbed is determined from the variation of gas pressures in known volumes. One of the labs measured isotherms gravimetrically, however, where the amount of gas adsorbed is determined by the weight change of the adsorbent before and after adsorption. Table 1 summarizes general information about each laboratory. To maintain confidentiality, the various laboratories will be referred to as Labs 1-6 throughout this paper.

Four samples of the Antrim Shale from a whole core taken in the Nomeco Bagley East B3-11 well in Otsego County, MI were provided to each laboratory. The Antrim Shale is a naturally fractured, organically rich, gas bearing shale that is being developed actively in the Michigan Basin in north-central Michigan. In this area, the Antrim Shale is encountered at depths of between 1,000 and 2,000 ft, and it produces significant volumes of water along with the gas, much like a coalbed methane reservoir. Fig. 1 shows the location of the Bagley East B3-11 well, which is in the heart of the Antrim play in Otsego County.⁶

Table 2 identifies the depths of the four samples provided to each laboratory for the isotherm measurements. Fig. 2 is a log section from the Bagley East B3-11 well showing the location of the core samples. This is a section of the lower Antrim Shale which is further subdivided into intervals more commonly referred to as the Lachine, the Paxton, and the Norwood. The Lachine and Norwood intervals are the more highly organic intervals typically completed, while the Paxton is the less organic interval between the two. Table 2 provides some additional petrophysical information about each sample, including gas content measured from sealing companion samples in canisters and total organic carbon (TOC) measurements. As Table 2 shows, the Norwood sample has the highest TOC and gas content, followed by the Lachine samples; the Paxton sample has the least organic material and gas content of the four samples.

The amount of sample provided to each laboratory was dependent on the lab's requirements and the amount of sample available. Individual laboratory requirements ranged anywhere from about 10 grams to about 200 grams. In some instances, we were unable to provide as much sample as preferred by a particular lab. The sample provided to each lab had been crushed, but not sieved, and a variety of particle sizes were present.

The same instructions were provided to each laboratory. The labs were asked to measure a methane adsorption isotherm for each sample at 75°F across a pressure range from 0 to 1,000 psia. Measurements were requested at both dry and equilibrium moisture conditions, if possible, although, as we later learned, some labs performed the tests at "as received" conditions. In addition, each lab was asked to report values measured for sample density (bulk or grain), sample weight, sample volume, and helium porosity (if measured). Volumes for the Langmuir volume, V_L (scf/ton), and the Langmuir pressure, p_L (psia), were also requested. Each lab was asked to report its isotherm results as methane adsorption (scf/ton) vs. pressure (psia).

Table 1 summarizes the test conditions used by each laboratory. Lab 2 measured isotherms at dry conditions only, although it subsequently "corrected" its results for moisture effects. Labs 1 and 5 ran tests at dry conditions only. Lab 3 measured isotherms at dry and at "as received" conditions; Lab 4 conducted tests only at "as received" conditions. Lab 6 ran tests on dry samples and also attempted to restore the samples to an equilibrium moisture content for a second set of isotherm measurements.

RESULTS OF ISOTHERM MEASUREMENTS

Despite giving each laboratory identical instructions and similar samples, the initial results received from the labs were confusing and varied widely. Fig. 3 shows the first set of results received from each lab for Sample 1 in the Lachine. These results were all reported to be for dry samples. Lab 2 ran repeat tests on each sample; thus, in Fig. 3 and subsequent figures, its values are plotted as Lab 2 and Lab 2A. In addition, the gas content of each sample at 450 psia (initial reservoir pressure), as measured in a sealed canister test, is also plotted in Fig. 3 and all subsequent figures.

While most of the isotherms had similar shapes, the magnitude of the methane contents reported differed significantly, ranging from about 20 scf/ton at the initial reservoir pressure of 450 psia to as high as 125 scf/ton. Table 3 summarizes the initial methane contents reported by each lab for all four samples at 450 psia. A similar spread in the values observed for Sample 1 was noted for each sample. Despite this variation between laboratories, however, we did note that the values of methane content reported by each lab increased as the total organic carbon content increased for each sample; this was as expected.

Because of the large spread in the values initially reported, we were suspicious that the results actually represented a variety of different conditions - that is, they were not all adsorbed methane contents

measured at dry conditions. To resolve the discrepancies, we contacted each laboratory individually to determine exactly what measurements were being made and reported. In addition, representatives from four of the six laboratories and GRI met together to discuss how the tests were conducted. As a result of these conversations, we confirmed that the values presented in Fig. 3 did not represent the same quantities or test conditions after all.

For instance, there was some confusion in the terms "total" versus "adsorbed" methane content. We defined "total" methane content as the quantity of methane adsorbed on the shale plus any methane contained in the free porosity. "Adsorbed" methane content was considered to be the difference between the total methane stored in the sample and the methane stored in the free porosity. Most labs determined free porosity with helium (i.e., measuring a helium isotherm). One lab, however, suggested free porosity was a meaningless term in a gas shale and, therefore, there could be no difference between total and adsorbed methane.

There was also some confusion in the terms "dry" versus "as received" conditions. We assumed "dry" conditions to mean the samples would be dried, usually under vacuum, to drive off any water and avoid including water vapor in the pressure measurement during the isotherm procedure. Some labs, however, apparently took "dry" conditions to mean no attempt was made to restore the sample to equilibrium moisture conditions. Thus, they measured the isotherms on the samples "as received," without drying the samples further to remove any water.

In addition to these nomenclature problems, we discovered that not all labs were using the same standard conditions. At least one lab was reporting standard conditions at 0°C and 1 atm, a "scientific" standard, rather than at 60°F and 14.65 psia, a more common "oilfield" standard. We also determined that one lab was using metric tons instead of U.S. tons in reporting its methane contents in "scf/ton".

Having identified and resolved these discrepancies, we plotted and compared the isotherm measurements again. Fig. 4-7 compare the results reported for total methane content vs pressure on the dry samples. Again, the total methane content includes the adsorbed methane plus the methane contained in any porosity. All labs except Lab 4, reported their results this way.

In Figs. 4-7, the spread in the values reported was much less than that in Fig. 3, although the total methane contents reported still varied by as much as a factor of 2. In most cases, the values reported by Labs 1, 2, and 3 were similar, while the values reported by Labs 5 and 6 were substantially different, especially at higher pressures. In fact, the isotherms measured by Lab 6 typically exhibited a concave upward shape that was quite different from what is normally observed. As noted in Table 3, Lab 3 was not able to measure isotherms at dry conditions on Sample 4 (Fig. 7) due to an insufficient sample size.

Figs. 8-11 compare the isotherms reported to be adsorbed methane only (excluding the methane in any porosity) vs. pressure on the dry samples. Labs 1, 2, and 3 reported their results this way also. Again, the overall spread in these data is considerably less than in Fig. 3, but there is still a 25 to 50% or more difference between the adsorbed methane values reported for some of these samples.

Figs. 12-15 compare the isotherms measured by Labs 3 and 4 on the samples at "as received" conditions. These isotherms are plotted as adsorbed methane vs. pressure for the "as received" samples. Figs. 12-15 also include Lab 2's dry isotherm measurements as "corrected" for moisture content. Lab 6's isotherm measurements at equilibrium moisture conditions are not plotted in Figs. 12-15, since the test conditions were different and since Lab 6 was the only lab attempting to restore the samples to an

equilibrium moisture condition. Again, while the spread in the isotherm results is considerably narrowed, there is still a 25 to 50% difference at some pressures between the values reported by different labs.

Table 4 compares the other properties (density, porosity, etc.) measured by each laboratory for Sample 1 in the Lachine. The sample weights are typical of what the respective labs used for each sample tested. Only Labs 1, 2, and 4 reported porosities; values between labs varied by as much as 15 to 20%. The wide variation in the Langmuir parameters was also typical of what was reported for the other samples.

Table 5 presents the results of routine core analysis measurements conducted by an independent laboratory (not one of the six participating in the isotherm comparisons) on whole core plugs from the Bagley B3-11 well. It is important to note that the helium porosities measured ranged from about 8-11%; clearly, the Antrim Shale must contain some gas stored in free porosity, as well as adsorbed to the shale. These results also show that there are significant oil and water saturations in the Antrim, further suggesting the importance of restoring the samples to an equilibrium moisture condition prior to conducting the isotherm measurements.

Figs. 16 and 17 present the adsorbed methane contents measured by the various labs at both dry and "as received" conditions at 450 psia as a function of the total organic carbon (TOC) measured for each sample. These figures illustrate the spread in the adsorbed gas content measurements reported at the initial reservoir pressure; this is generally about 25 to 50% as mentioned earlier. The figures also confirm that the adsorbed methane content increases as the total organic carbon content increases in the Antrim Shale.

Fig. 18 presents a comparison of how the isotherm data can change as a function of the test conditions and how the data are reported; the isotherms plotted in the figure are from Sample 1. Lab 3 measured isotherms at both dry and "as received" conditions and reported both total methane content and adsorbed methane content for the samples it tested. Fig. 18 illustrates the differences in the results reported. Assuming the "as received" measurements are most representative of in-situ conditions, it illustrates how isotherms measured on dry samples can overstate the gas content in the shales.

This figure also illustrates how important it is for a user of the laboratory data to understand just what is being reported. For example, most simulators use the adsorbed methane content vs. pressure data as input. Thus, an engineer must be careful not to use a total methane isotherm, since this could result in significant overestimates of gas in place and well performance. Still, comparing the total methane isotherm with the adsorbed methane isotherm suggests a significant volume of gas is stored in the free porosity in these shales. This must also be accounted for properly in reserves estimation and reservoir modeling.

TEST PROCEDURES

It is beyond the scope of this paper to discuss the different test methods used by each individual laboratory. As a result of our meetings and discussions with the various labs, however, there are several critical parts of the isotherm measurement procedure that were identified as (1) potential sources of error or (2) likely reasons for the differences measured between laboratories. These included (1) sample preservation and preparation, (2) cell calibration, (3) temperature measurement and control, (4) measurement of bulk density, and (5) how (or if) equilibrium is established.

Regarding sample preparation, some labs dried the samples, while others tested them at "as received" conditions as discussed previously. Among the labs drying the samples, different drying times and temperatures were used. Some labs tested all particle sizes provided, while others sieved the samples into a

narrow particle range (e.g., 17-35 mesh) before measuring the isotherms. Several of the labs evacuate the samples prior to testing, while others never pull a vacuum on the sample.

Cell calibration and temperature control are accomplished differently in each laboratory, but each lab appears to take particular care to ensure proper calibration and temperature control. Even so, differences in calibration procedures and temperature control no doubt lead to variations in the isotherms reported. Proper cell calibration and temperature control are particularly important in measuring isotherms in gas shales, where the gas content and capacity to adsorb gas are typically quite low compared to coals or other gas adsorbing materials.

Some labs measured bulk density, while others measured grain density (see Table 4). The bulk density measurements were accomplished using a mercury porosimeter. Questions arise here as to what is the appropriate mercury pressure to use when measuring bulk density. Lab 2 uses 30 psi which should invade the sample down to a pore size of 6 microns. The assumption made is that none of the porosity is invaded, but that is not certain.

Regarding equilibrium, most labs allow a sample to continue to adsorb methane at each new pressure level, until the change in methane adsorbed in each time increment cannot be detected within the measurement resolution of the test apparatus. In this way, it is assumed that equilibrium is achieved at each pressure level. Lab 4, however, did not run its tests this way. Instead, at each pressure step, it allowed a sample to take on methane for only 2 to 3 hours, and then a new pressure step was begun. Because equilibrium was not achieved at each pressure level, Lab 4's results are extrapolated to equilibrium conditions using an internally developed plotting technique. This resulted in a reported value of methane content that was typically 25% to 60% greater than any value measured. The reliability of this extrapolation method and whether it should be adopted and used by other labs are not clearly established.

Although we asked each laboratory to conduct isotherm measurements at equilibrium moisture content, if possible, only Lab 6 attempted it. Frankly, the methodology for restoring equilibrium moisture conditions has yet to be studied in any detail for gas shales. The consensus among the various laboratory representatives was that we really do not know how to restore gas shales to equilibrium moisture conditions. This is a subject for future research.

Perhaps the most important observation made from comparing the various laboratory methods is that no standard industry protocol exists for measuring methane isotherms in gas shales. While individual laboratories appear to take appropriate precautions with sample preparation, cell calibration, temperature control, establishing equilibrium, and other critical factors, the methods used and the degree of error that is tolerated in the measurements is the prerogative of the laboratory itself. In addition, there does not appear to be a well established nomenclature or units convention for reporting the isotherm results. If there is, it does not appear to be widely available or used among different laboratories. Perhaps establishing this protocol and developing a common nomenclature are reasonable goals for future comparisons of this type.

FUTURE WORK

GRI is considering coordinating a second set of comparative isotherm measurements using Antrim Shale samples beginning in the Fall of 1993. The next tests should be conducted using fresh core samples, and more attention will be given to sample preparation and splitting to ensure that each participating lab receives samples as close to identical as possible. The consensus among the laboratory representatives participating in this first study was that the next set of measurements should be conducted at in-situ moisture conditions. Thus, an experimental procedure for establishing equilibrium moisture content in gas

shale samples is needed prior to beginning the measurements. This procedure should be reviewed, revised as necessary, and agreed to by all participating labs prior to conducting the next round of comparative isotherm measurements.

Most of the labs participating in this first study have also expressed an interest in being part of any future experiments. This process will be open to all interested parties, however, and any service company, producer, or university interested in participating is strongly encouraged to do so.

CONCLUSIONS

On the basis of this multi-laboratory comparison of isotherm measurements, we have made the following observations and drawn the following conclusions.

1. No standard industry protocol exists for measuring gas shale isotherms in the laboratory.
2. Isotherm results reported by various laboratories can vary widely, especially if the laboratory and the customer are not clear on such factors as what is to be measured, how the results are to be reported, and what units are to be used. It is important that the engineer using the data understand clearly what is being measured and reported to ensure that the isotherms are used properly in making estimates of gas in place and well performance.
3. Even when problems with nomenclature and units are resolved, isotherms measured at similar conditions may vary by as much as 25 to 50% among different labs.
4. Critical components of the isotherm measurement process that can be potential sources of error or differences among various labs include (1) sample preservation and preparation, (2) cell calibration, (3) temperature control, (4) density measurement, and (5) how (or if) equilibrium is determined.

ACKNOWLEDGMENTS

The authors wish to thank the representatives from all six laboratories who participated in the comparative isotherm study, several of whom performed these measurements at their own time and expense. We also thank Lab 4 for hosting the meeting of laboratory representatives conducted to review the initial results.

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Table 1

**General Information on Laboratories Participating in
Isotherm Comparison Project**

Lab	Type of Organization	Measurement Method Used	Test Conditions	Remarks
1	University	Volumetric	Dry Only	
2	University	Volumetric	Dry Only	Ran repeat tests on each sample; corrected dry isotherms for moisture effects.
3	Commercial Lab	Volumetric	Dry and As Received	
4	Major Company	Volumetric	As Received	Ran tests at pressures to about 500 psia.
5	University	Gravimetric	Dry Only	
6	University	Volumetric	Dry and Equilibrium Moisture	Limited pressure range to about 350 psia.

Table 2

**General Information on Samples Used for
Isotherm Comparisons**

Sample	Depth (ft)	Interval	Gas Content - Canister Data (scf/ton)	Total Organic Carbon	
				Lab 7* (%)	Lab 4 (%)
1	1,360.6 - 1,361.1	Lachine	37	4.6	5.2
2	1,428.0 - 1,429.0	Lachine	67	11.1	11.6
3	1,472.0 - 1,473.0	Paxton	19	3.2	3.2
4	1,478.0 - 1,479.0	Norwood	100	16.9	17.7

*Lab 7 is a commercial lab that measured TOC, but did not measure isotherms on the samples.

Table 3

Comparison of Initial Results Reported By Each Laboratory

Lab	Methane Content @ 450 psia (scf/ton) ¹			
	Sample 1 (5.2% TOC)	Sample 2 (11.6% TOC)	Sample 3 (3.2% TOC)	Sample 4 (17.7% TOC)
1	45	97	38	135
2	55	100	30	130
3	45	82	35	NM ²
4	20	35	10	65
5	125	60 ³	65	195
6	93 ⁴	156 ⁴	79 ⁴	173 ⁴

¹All values were initially thought to be adsorbed methane content measured at dry conditions.
²Not measured due to insufficient sample size.
³Lab 5 reported experimental problems with this sample.
⁴Values reported at a maximum pressure of about 350 psia.

Table 4

**Summary of Other Properties Measured on
Sample 1 from the Lachine**

Lab (Test Conditions)	Sample Wt. (gm)	Density (gm/cc)	Sample Volume (cc)	He Porosity (%)	pL ¹ (psia)	V _L ¹ (scf/ton)
1 (Dry)	8.852	2.335 (bulk)	3.791	7.4	654.8	122.8
2 (Dry)	8.893	2.335 (bulk)	3.808	9.0	749.3	145.6
3 (Dry)	78.38	2.54 (grain)	NR	NR	361	86
3 (As Received)	172.37	2.54 (grain)	NR	NR	412	66
4 (As Received)	19.92	2.53 (grain)	11.841	7.3	1627.1	117.4
5 (Dry)	NR	2.28 (He skeletal)	NR	NR	3888.2	1215.8
6 (Dry/Eq. Moist.)	NR	2.565 (grain)	NR	NR	NR	NR

Notes: NR - Not Reported
¹Langmuir parameters based on adsorbed methane content vs. pressure only.

Table 5

**Routine Core Analysis Measurements on
Nomeco Bagley East B3-11 Samples**

Depth (ft)	Interval	He Porosity (%)	Oil Saturation (%)	Water Saturation (%)
1,357	Lachine	9.2	13.5	44.7
1,370	Lachine	9.5	38.5	20.0
1,421	Lachine	8.3	67.2	22.1
1,428	Lachine	8.8	17.3	27.2
1,443	Paxton	10.6	57.2	32.0
1,472	Paxton	11.4	59.3	24.7
1,480	Norwood	9.1	1.5	28.7
1,486	Norwood	7.9	14.0	38.0
1,494	Norwood	9.0	46.7	50.7

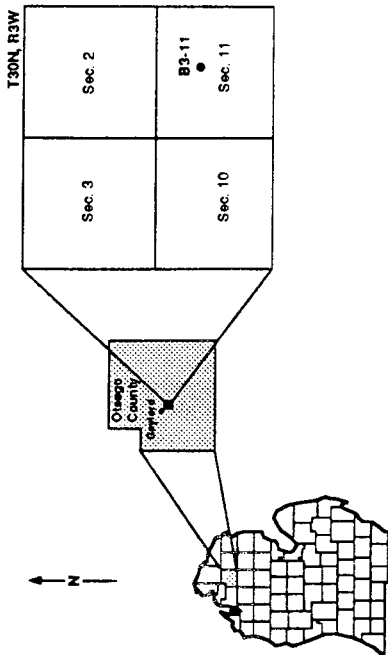
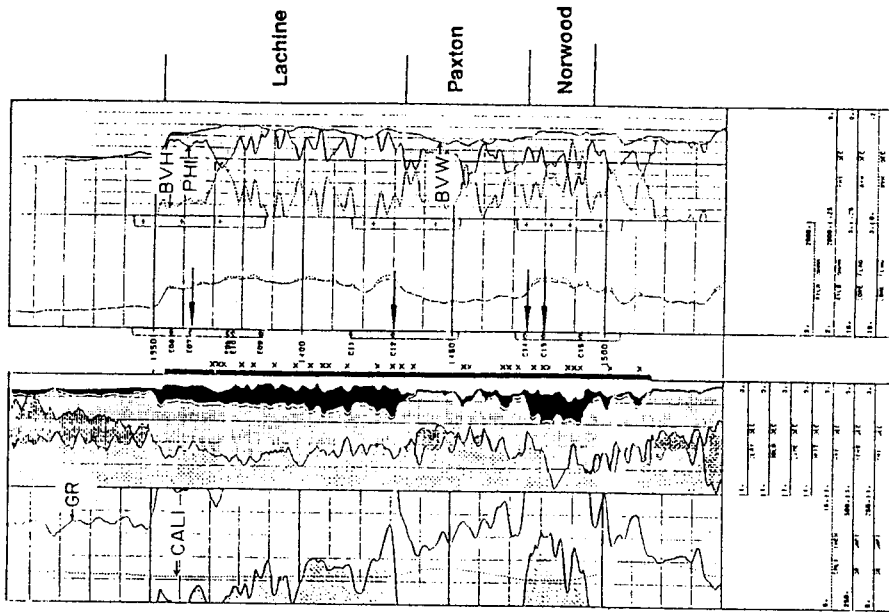


Fig. 1 - Location of Nomeco Bagley East B3-11 well in Otsego County, MI.⁶



* Prepared by ResTech Houston
 Note: Log Depth = Core Depth + 2 ft
 Fig. 2 - Log section from lower portion of Antrim Shale in the Nomeco Bagley East B3-11 well, showing location of core samples used for isotherm measurements.

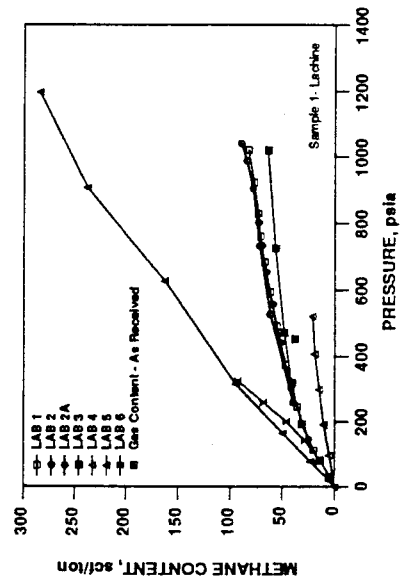
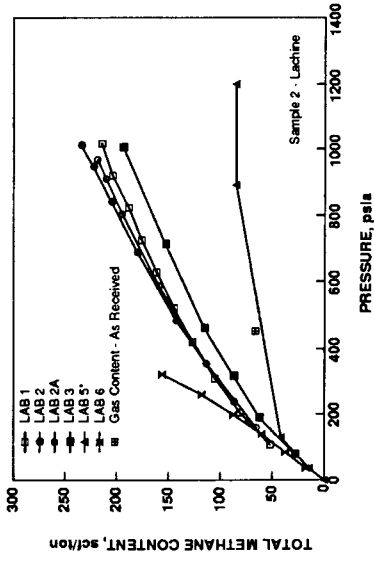


Fig. 3 - Initial results received were confusing and varied widely.



* Lab 5 reported experimental problems with this sample.

Fig. 5 - Total methane isotherms measured at dry conditions; Sample 2 - Lachine.

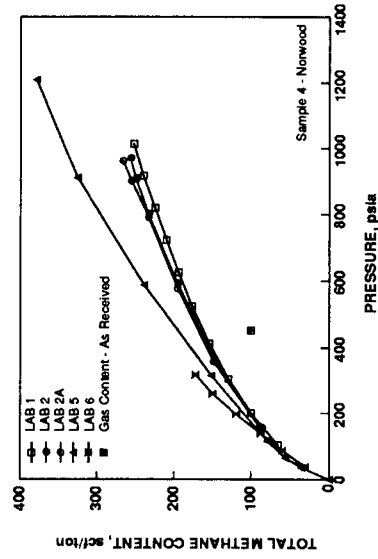


Fig. 7 - Total methane isotherms measured at dry conditions; Sample 4 - Norwood.

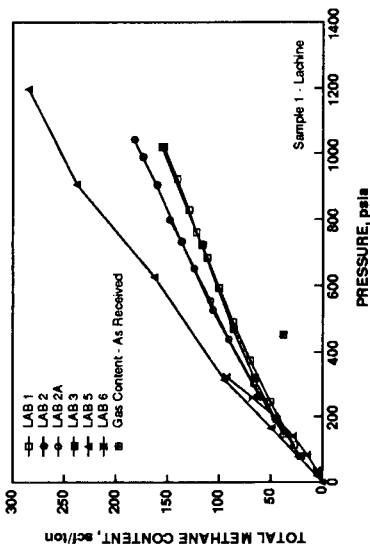


Fig. 4 - Total methane isotherms measured at dry conditions; Sample 1 - Lachine.

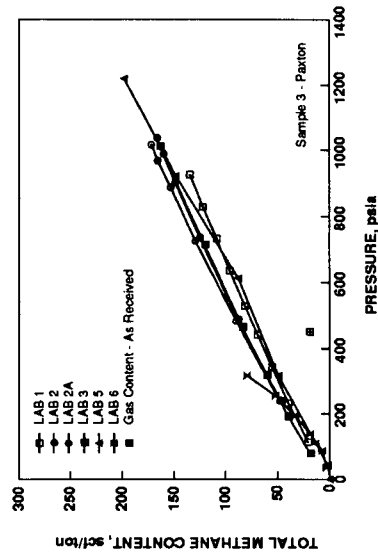


Fig. 6 - Total methane isotherms measured at dry conditions; Sample 3 - Paxton.

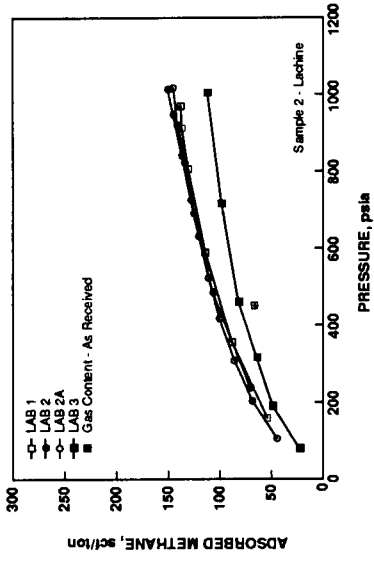


Fig. 9 - Adsorbed methane isotherms measured at dry conditions; Sample 2 - Lachine.

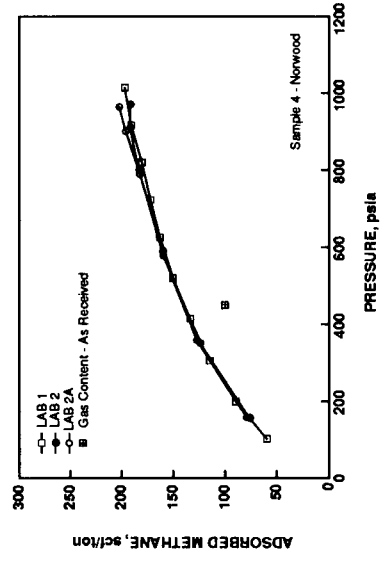


Fig. 11 - Adsorbed methane isotherms measured at dry conditions; Sample 4 - Norwood.

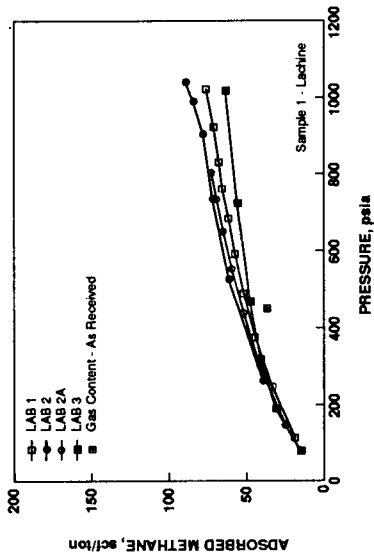


Fig. 8 - Adsorbed methane isotherms measured at dry conditions; Sample 1 - Lachine.

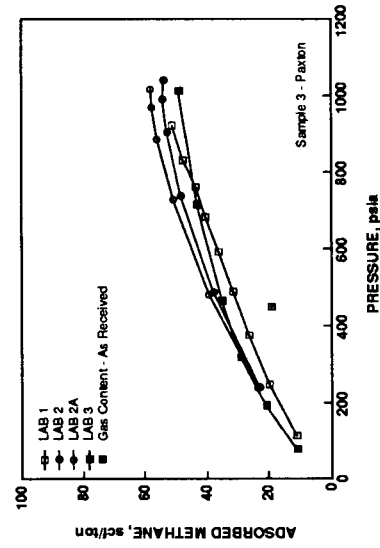


Fig. 10 - Adsorbed methane isotherms measured at dry conditions; Sample 3 - Paxton.

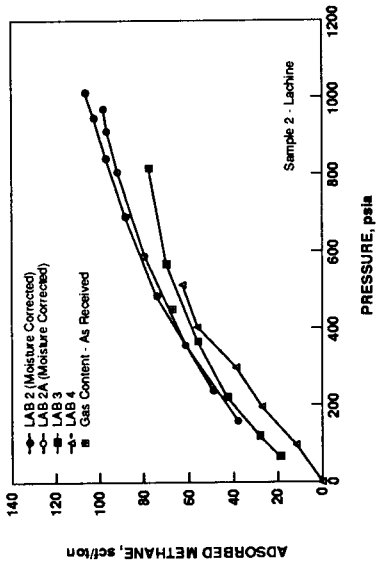


Fig. 13 - Adsorbed methane isotherms measured at "as received" conditions; Sample 2 - Lachine.

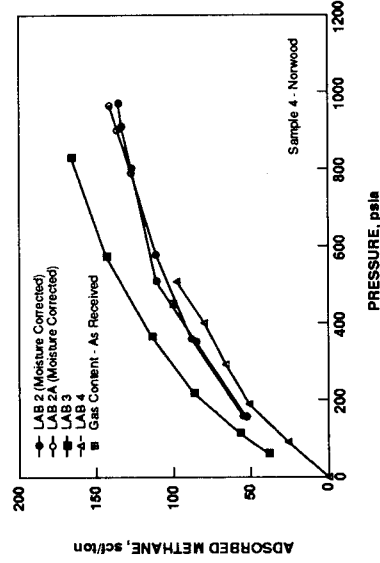


Fig. 15 - Adsorbed methane isotherms measured at "as received" conditions; Sample 4 - Norwood.

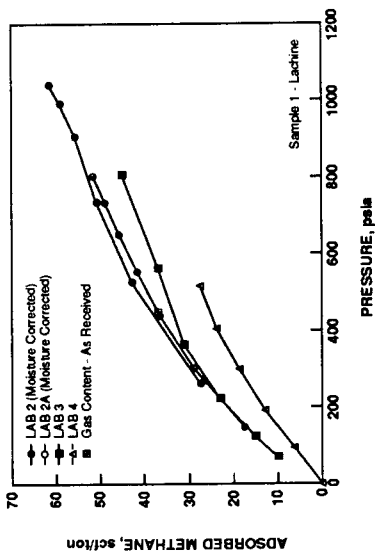


Fig. 12 - Adsorbed methane isotherms measured at "as received" conditions; Sample 1 - Lachine.

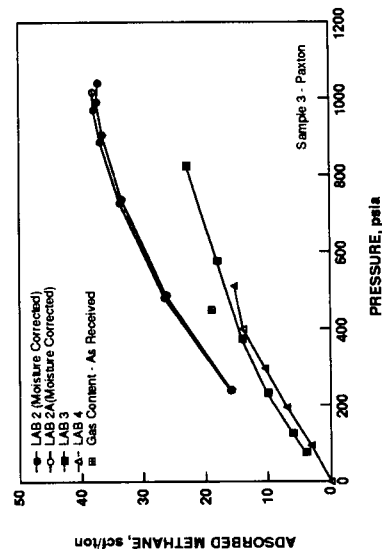


Fig. 14 - Adsorbed methane isotherms measured at "as received" conditions; Sample 3 - Paxton.

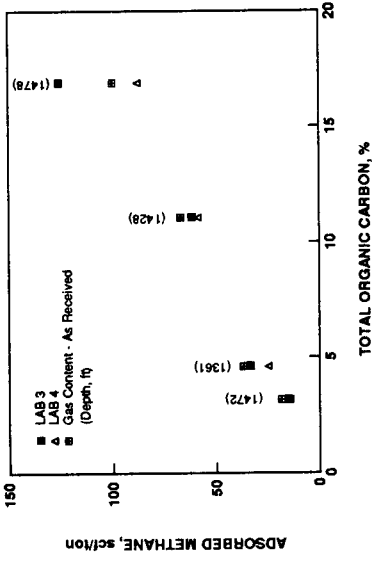


Fig. 17 - Adsorbed methane content (measured at 450 psia) increases as total organic carbon increases - "as received" conditions, all samples.

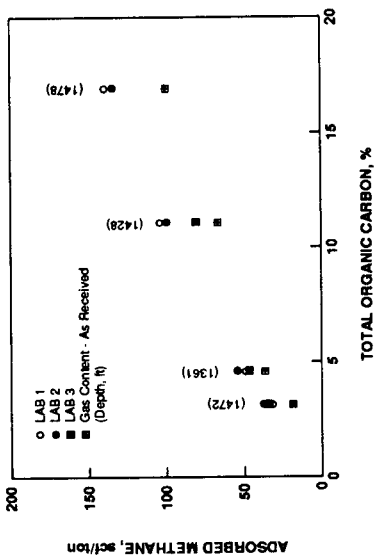


Fig. 16 - Adsorbed methane content (measured at 450 psia) increases as total organic carbon increases - dry conditions, all samples.

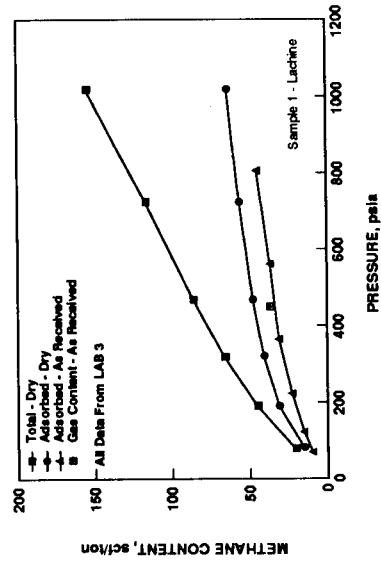


Fig. 18 - Isotherms reported for different test conditions; Sample 1 - Lachine.

