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POROSITY AND PERMEABILITY OF EASTERN DEVONIAN GAS SHALE

**Project 61071 Topical Report
For the Period July Through October 1984**

**Prepared by
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**Prepared for the
United States Department of Energy
Morgantown Energy Technology Center
Morgantown, West Virginia 26505**

Under Contract No. DE-AC21-83MC20342

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TOPICAL REPORT
(July-October 1984)

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IGT Project No. 61071

for

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MORGANTOWN ENERGY TECHNOLOGY CENTER

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EXECUTIVE SUMMARY

The permeability to gas of eight samples of eastern Devonian black shale was measured by a steady-state method under pressures bracketing reservoir net stress. Porosity to gas was also measured under these net stresses, utilizing a Boyle's Law procedure. The cores had been dried prior to analysis such that all free pore water was removed from the rock, although one to two layers of water of hydration were retained by swelling (smectite) clays.

Seven of the eight cores analyzed consist of the lower Huron Member of the Ohio Shale. One of these was recovered from southeastern Kentucky, and the other six were obtained from wells drilled in the Ohio-West Virginia border region along the Ohio River. The eighth core consists of Marcellus Shale obtained from a well drilled in Morgantown, West Virginia. The lower Huron core samples are from depths of 2440 to 3320 feet, and all seven were run at the same two net stress values. The Marcellus core was from a depth of 7448 feet and was run at two correspondingly higher net stresses.

The lack of adequate reservoir pressure data prohibited performance of core analysis measurements under in-situ net stress. Estimated high and low net confining stress values were used in hopes of bracketing the true reservoir stress experienced by the rocks in the ground.

The lower Huron Shale cores had extremely low porosity to gas. All seven samples had porosity to gas values of less than $0.3 \pm 0.1\%$. Gas permeability of most lower Huron samples varied with time and differential pressure values in a manner suggestive of a mobile liquid phase in the pores. Capillary pressures for the liquid in the range of 1.1 to 30 psi were measured by slowly reducing upstream pressure on the plugs until gas flow stopped due to imbibition of liquid into the downstream end. Permeabilities to gas, after extended flow with pressure drops in the range of 3 to 10 times capillary entry pressure, ranged from 8 microdarcies down to 1 nanodarcy with a net stress of 1750 psia on the shale, and from 5 microdarcies to less than 0.2 nanodarcy with a net stress of 3000 psia on the shale. Subsequent chromatographic analysis revealed that the liquid in the lower Huron core was a light, paraffinic petroleum (C_7H_{16} to $C_{23}H_{48}$). The presence of this petroleum as a mobile liquid in the lower Huron pores accounts for both the nonlinear permeability behavior and the low porosities to gas of these rocks.

Core analysis on the Marcellus Shale sample was also performed under two values of net stress. Dry gas permeability on this core ranged from 19.3 microdarcies at 3000 psia net stress to 5.9 microdarcies at 6000 psia net stress. Mobile liquid hydrocarbon was not present in the Marcellus Shale as indicated by the permeability behavior and chromatography. Apparent porosity of this rock to gas, however, behaved in an unusual manner. Twelve measurements, spanning the gas pressure range of 45 to 1500 psia, revealed that the volume of methane (at 14.7 psia and 60°F) per volume of rock varied with pressure as $0.448 \times P^{1/2}$. This led to the calculation that under near-hydrostatic pressure at a depth of 7448 feet, the Marcellus Shale contains 26.5 SCF of gas per cubic foot of rock. The mechanism responsible for entrainment of these large volumes of gas has not been identified.

Gas flow rate measurements taken under two net confining stress values, with a variety of gas pressures and pressure drops, indicate that gas permeability in Devonian shale is strongly stress-dependent. In the Marcellus sample, for instance, doubling the net confining stress reduced the gas permeability by nearly 70%. Gas flow path dimensions deduced from high quality Klinkenberg data on the Marcellus sample have a characteristic width of 0.05 microns at a net stress of 3000 psi and 0.35 microns at a net stress of 6000 psi. This surprising result of wider flow path openings under higher net stress suggests a bimodal pore size distribution with the small openings being closed at the higher stress. Although the oil in the Huron seriously degraded the quality of Klinkenberg data, similar bimodal flow path characteristics were deduced for those samples having effective permeabilities in the microdarcy range.

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1.0 INTRODUCTION

The Institute of Gas Technology (IGT) has performed core analyses under contract to the U.S. Department of Energy (DOE), Morgantown Energy Technology Center (METC), on Eastern Gas Shale and Western Tight Gas Sands. These core analyses were directed at achieving a better understanding of the fundamental reservoir properties of these low permeability rocks, measured with IGT's Computer Operated Rock Analysis Laboratory (CORAL). CORAL was built under subcontract to Sandia National Laboratories to implement techniques developed under prior contract to the Bartlesville Energy Technology Center (BETC). The CORAL is capable of measuring actual gas flow rates through rock as low as 10^{-6} cm³/second with an accuracy of a few percent and can measure permeabilities below a nanodarcy (10^{-6} md). Other rock properties measured by the CORAL include gas porosity under net confining stress with a resolution of about $\pm 2\%$ of the measured value, and pore volume compressibility with a resolution of 1×10^{-6} psi⁻¹. The CORAL has proven to be a state-of-the-art tool capable of performing the type of accurate core analysis measurements that are needed and have never before been made on tight sand or shale. A description of the engineering and operational design of the CORAL has been presented by Randolph (1983) in SPE/DOE Paper 11765 and also in the dry sandstone core analysis topical report (DOE/MC/20342-4) prepared under this contract.

The work plan for this project divided the core analysis into two tasks. One task was concerned with the suites of measurements performed on tight sandstone core from the DOE Multiwell Experiment (MWX) in the Piceance Basin of Colorado. Results from IGT's core analysis on the MWX samples have been reported to METC via a series of four topical reports.

The other task under this contract provided for porosity and permeability measurements of Devonian shale core. These types of analyses had never before been performed on eastern gas shale with any degree of confidence due to the resolution limits inherent in measuring gas flow rates with a pipet and stopwatch. The development of the CORAL at IGT permitted the measurement of ultra low flow rates by electronic means in a stable isothermal environment with unprecedented accuracy and repeatability. Although the equipment was originally developed for use on tight gas sands, there were obvious applications to other tight gas reservoirs, including Devonian shale.

One of the major uses for accurate porosity and permeability data on eastern gas shale is to provide inputs for numerical simulators. Using previously available, relatively inaccurate data for the reservoir parameter inputs, numerical reservoir simulations have been run in the past by DOE on several producing shale wells in the Appalachian Basin. The production curves generated by the simulator were compared with the actual production histories over the same period of time from the producing wells in order to ascertain the degree of history-matching. The comparison between the simulator curves and actual well production curves resulted in a reasonably good history-match on three representative shale wells located in Ohio (Lewin and Associates, 1982).

A basic principle of mathematical reservoir simulation models is that the output is only as good as the input. In the history-matching described above, additional runs of the simulator showed that reasonable history matches of gas production were attainable using a wide range of values for different inputs, and that a considerable amount of uncertainty existed regarding realistic values for many of the basic shale reservoir parameters such as porosity, permeability, in-situ gas content, and response of the rock to stress. With conventional reservoirs, such reservoir properties are commonly measured to an accuracy of a few percent, which does not affect their usefulness as inputs to a model, nor hinder the capability to history-match and reasonably project production with the simulator. However, to reach the same accuracy of numerical simulation for the unconventional natural gas resources, a substantially upgraded effort is required for the measurement of input parameters. For example, permeabilities measured to an accuracy of 0.1 microdarcy may be considered more than adequate when modeling a millidarcy sandstone reservoir, but are clearly inadequate when trying to model a one-microdarcy tight sand. It is very important, therefore, to quantify and account for the reservoir parameters used to simulate unconventional gas production in a precise and reliable manner that reflects the actual reservoir conditions as best possible. The objective of the shale core analysis work was to provide understanding and improvements in the accuracy and reliability of the most sensitive simulator input parameters, leading to a significant improvement in predicting shale gas production.

2.0 SAMPLE PREPARATION AND ANALYSIS

To provide accurate data inputs to METC's shale reservoir modeling efforts, IGT was to measure porosity to gas and Klinkenberg permeability on an unspecified number of Devonian shale core samples under this contract. These measurements were to be made using the CORAL after upgrading the equipment to the degree necessary to achieve the required resolution.

We are aware of several situations where Devonian shale permeabilities have been reported from runs in equipment designed for tight sands. However, we doubt whether any previously measured values are truly representative of in-situ conditions. Reasons for this are as follows:

- Resolution of the lab hardware has seldom, if ever, been clearly defined. In some cases it is only as low as 10 to 100 nanodarcies.
- We are not aware of any attempts to measure the stress dependence of permeability in Devonian shales, especially since realistic in-situ net confining stresses have never been adequately defined for these rocks.
- In at least some cases, samples were dried in vacuum ovens before testing. This guarantees measured porosity and permeabilities will be higher than in-situ values.

We emphasize that porosity and permeability are not single numbers to be measured and reported for each sample analyzed in the laboratory. Rather, these are coefficients that appear in the differential equations used to calculate fluid content and movement in porous media. For some high-porosity, high permeability formations, adequate descriptions of well and reservoir performance has been achieved by assuming that these coefficients are constants. This is definitely not a valid assumption for tight sands or for shale.

Our approach to Devonian shale core analysis has been from the perspective of pioneers such as Muskat, Buckley, Leverett, and Klinkenberg. Specifically, our objective was to define the physical phenomena that occur during production and to measure the constants required to include the mathematical descriptions of those phenomena in production calculations. This approach uses laboratory procedures designed to maximize the possibility that the results obtained are truly descriptive of in-situ reservoir rock.

2.1 Sample Selection and Background

Between 1975 and 1981, the U.S. government cut and retrieved nearly 17,000 feet of Devonian shale drill core under the Eastern Gas Shales Project (EGSP). Oriented core was recovered from the Devonian shale section throughout a variety of localities in the Appalachian Basin, Michigan Basin, and Illinois Basin. This large supply of oriented Devonian Shale core provided the raw material for selection of a limited number of samples to be run in the CORAL.

Twenty-eight zones of interest from 13 wells in five states (New York, Pennsylvania, Ohio, West Virginia, and Kentucky) were sampled. These zones represent 10 stratigraphic horizons within the eastern gas shales sequence (Middle and Upper Devonian).

The 13 EGSP cores sampled by IGT were selected from a prioritized list suggested by DOE. It was understood that the shale samples ultimately selected would depend on the condition and availability of the cores. Before sampling trips were undertaken, certain core footage was identified as zones of interest based on a review of well reports, core descriptions, well logs, production reports, and other data supplied to IGT by DOE and others. Each of the zones of interest preselected by IGT covered 10 to 30 vertical feet and appeared to possess one or more of the following desirable characteristics:

- Known gas production
- Reported gas "shows"
- Wireline log indications of gas
- Wireline log indications of fairly high organic content
- A history of stimulation treatment
- Correlation with gas indications in another well
- Uniform lithology within a zone
- Lack of physical damage.

The selection of zones of interest was based heavily on the evidence from the wireline logs available; other data sources tended to be used in an important but supportive role. Because organic matter has an affinity for radioactive elements, the background levels of radiation from organic-lean

shales can be subtracted from the gamma ray logs to roughly estimate the organic content of black shales (Leventhal, 1981). This technique was employed along with analysis of the density logs to isolate cored intervals containing shales of high organic content for sampling. Since organics are less dense than minerals, lower density generally corresponds to higher organic content (Schmoker, 1978).

One of the major problems encountered when choosing core samples and basing that selection process on wireline log evidence is the lack of agreement between core depths and log depths. The driller and the logger sometimes use different zero points; for example, in the WV-5 well, the log heading shows that the zero point for the log was ground level, whereas drilling depths were measured from the Kelly bushing, 10 feet above ground on the drilling platform. Thus the depth to the casing seat was 1890 feet according to the driller, but is shown on the log at 1880 feet. A depth correction of 10 feet must be considered in working from one record to the other. Core depths normally relate to the driller's reference point; however, errors in assigning depth to a core can result for a variety of reasons, and appropriate depth corrections can vary throughout the drilling of a well.

The large number of zones targeted for sampling was thought to be advisable to ensure the collection of a sufficient volume of core in light of reports of excessive deterioration during storage — an unknown factor in October 1983. Numerous zones of interest also allowed samples to cover several stratigraphic units over a wide geographic area. The shale samples actually removed from the EGSP cores and shipped to IGT consisted of uniform, unfractured, full-diameter core segments of 3 to 4 inches minimum length. Intervals containing natural fractures, margin-to-margin disk fractures, large pyrite nodules and/or carbonate concretions were avoided. Strong emphasis was placed on selecting homogenous, undamaged rock samples representative of the actual shale gas reservoirs.

The depths and stratigraphic intervals that were sampled are listed in Table 1. A cross section of the stratigraphy of the Devonian shale sequence in the Appalachian Basin is shown in Figure 1.

Although a rather large amount of shale core footage was collected, it was recognized early on that only one or two runs of four samples each would be possible in the CORAL due to time limitations under the contract. As such,

Table 1. ZONES SAMPLED FROM EGSP CORES (Kentucky and Ohio)

DOE Well Designation and Location Data

| Stratigraphic Interval Name | Wire Line Log Data | | Density, gm/cm ³ | Core Samples in Hand | |
|--|--------------------|-----------------------------|-----------------------------|----------------------|------------------|
| | Depth, ft | G.R. Over SBL** (API Units) | | Depth Range, ft | Total Length, ft |
| <u>Somerset Gas Co./City of Somerset #1 Moore, Leslie, Co., Ky.</u> | | | | | |
| U. Huron | 2704-2814 | 167 | 2.50 | 2800.9-2805.9 | 1.40 |
| M. Huron* | 2818-2830 | 97 | 2.81 | 2821.7-2828.7 | 2.00 |
| L. Huron | 2896-2910 | 321 | 2.50 | 2903.1-2905.8 | 2.25 |
| <u>OH-6-4, Mitchell Energy Corp., 1-8 Straight-Wisemandle Unit, Gallia Co., Ohio</u> | | | | | |
| L. Huron* | 2765-2775 | 121 | 2.47 | 2770.4-2772.2 | 1.53 |
| <u>OH-6-5, Mitchell Energy Corp., 1-9 Carter, Gallia Co., Ohio</u> | | | | | |
| Cleveland* | 1700-1714 | 43 | 2.5 | 1702.0-1709.5 | 1.40 |
| L. Huron | 2440-2446 | 140+ | 2.5 | 2440.0-2445.6 | 1.90 |
| <u>OH-7, DOE/Columbia Gas Trans. Co., 20143T Meleski, Trumbull Co., Ohio</u> | | | | | |
| L. Huron | 2130-2149 | 61 | 2.52 | 2134.7-2149.5 | 1.58 |
| U. Olentangy | 2530-2546 | 101 | 2.47 | 2535.3-2541.1 | 2.00 |
| <u>OH-8, East Ohio Gas Co./Donohue, Anstey and Morrill #1 Shockling, Noble Co., Ohio</u> | | | | | |
| Huron | 3320-3333 | 26 | 2.56 | 3324.8-3330.7 | 1.73 |
| Rhinestreet | 4006-4016 | 95 | 2.53 | 4006.5-4013.6 | 1.50 |
| <u>OH-9, DOE/SAI/Gruy, Columbia Gas 10056A, Meigs Co., Ohio</u> | | | | | |
| L. Huron | 3010-3020 | 62 | 2.58 | 3015.3-3020.5 | 1.0 |
| L. Huron* | 3241-3251 | 91 | 2.61 | 3245.0-3247.0 | 2.0 |

* Samples analyzed in CORAL.

** SBL: Shale baseline for nonorganic shale.

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Table 1, Cont. ZONES SAMPLED FROM EGSP CORES (West Virginia, New York, and Pennsylvania)

| Stratigraphic Interval Name | Wire Line Log Data | | Density, gm/cm ³ | No. of Pieces | Core Samples in Hand | |
|--|--------------------|-----------------------------|-----------------------------|---------------|----------------------|------------------|
| | Depth, ft | G.R. Over SBL** (API Units) | | | Depth Range, ft | Total Length, ft |
| <u>WV-5, Reel Drilling Co. #3 D/K Farm, Mason Co., W.Va.</u> | | | | | | |
| Huron* | 2720-2744 | 72 | 2.52 | 3 | 2734.0-2738.5 | 0.90 |
| Huron* | 3023-3033 | 127 | 2.47 | 3 | 3025.2-3028.6 | 0.84 |
| Rhinestreet | 3335-3346 | 126 | 2.55 | 4 | 3335.9-3346.8 | 1.00 |
| <u>WV-6, Morgantown Energy Research Center, MERC-1, Monongalia Co., W.Va.</u> | | | | | | |
| Mahantango | 7304-7320 | 2 | 2.67 | 4 | 7307.0-7313.6 | 0.74 |
| Marcellus | 7446-7460 | 196 | 2.46 | 5 | 7448.3-7457.9 | 1.06 |
| Marcellus | 7478-7494 | 143 | 2.49 | 3 | 7486.9-7490.5 | 0.80 |
| <u>NY-1, N.Y. Nat. Gas/Nat. Fuel Sup. Co., Allegany Co., N.Y.</u> | | | | | | |
| Rhinestreet | 2320-2332 | 17 | 2.62 | 4 | 2325.8-2329.9 | 1.61 |
| Middlesex | 2600-2618 | 21 | 2.58 | 3 | 2602.9-2604.7 | 1.50 |
| <u>NY-3, EGSP #1 Scudder, Steuben Co., N.Y.</u> | | | | | | |
| Rhinestreet | 1224-1240 | 18 | 2.66 | 6 | 1229.9-1240.0 | 0.93 |
| <u>NY-4, Donahue, Anstey & Morrill #1 Valley Vista View, Steuben Co., N.Y.</u> | | | | | | |
| Genesee | 3030-3040 | 37 | 2.60 | 4 | 3031.6-3036.0 | 1.64 |
| Marcellus | 3795-3810 | 19 | 2.67 | 4 | 3796.8-3800.3 | 1.3 |
| <u>PA-1, DOE/Minard Run Oil Co. #1 M.R. Expl., McKean Co., Pa.</u> | | | | | | |
| Genesee | 4744-4756 | 155 | 2.43 | 2 | 4737.6-4752.2 | 0.70 |
| Marcellus | 5178-5200 | 207 | 2.40 | 5 | 5179.8-5187.2 | 1.14 |
| <u>PA-5, Peoples Nat. Gas Co. #1 Sokevitz, Lawrence Co., Pa.</u> | | | | | | |
| Rhinestreet | 3725-3738 | 78 | 2.56 | 2 | 3731.3-3732.5 | 1.10 |
| Sonyea | 3880-3888 | 21 | 2.51 | 3 | 3880.1-3888.7 | 1.07 |
| Mahantango | 4002-4012 | 19 | 2.56 | 3 | 4010.8-4012.4 | 1.10 |

* Samples analyzed in CORAL.

** SBL: Shale baseline for nonorganic shale.

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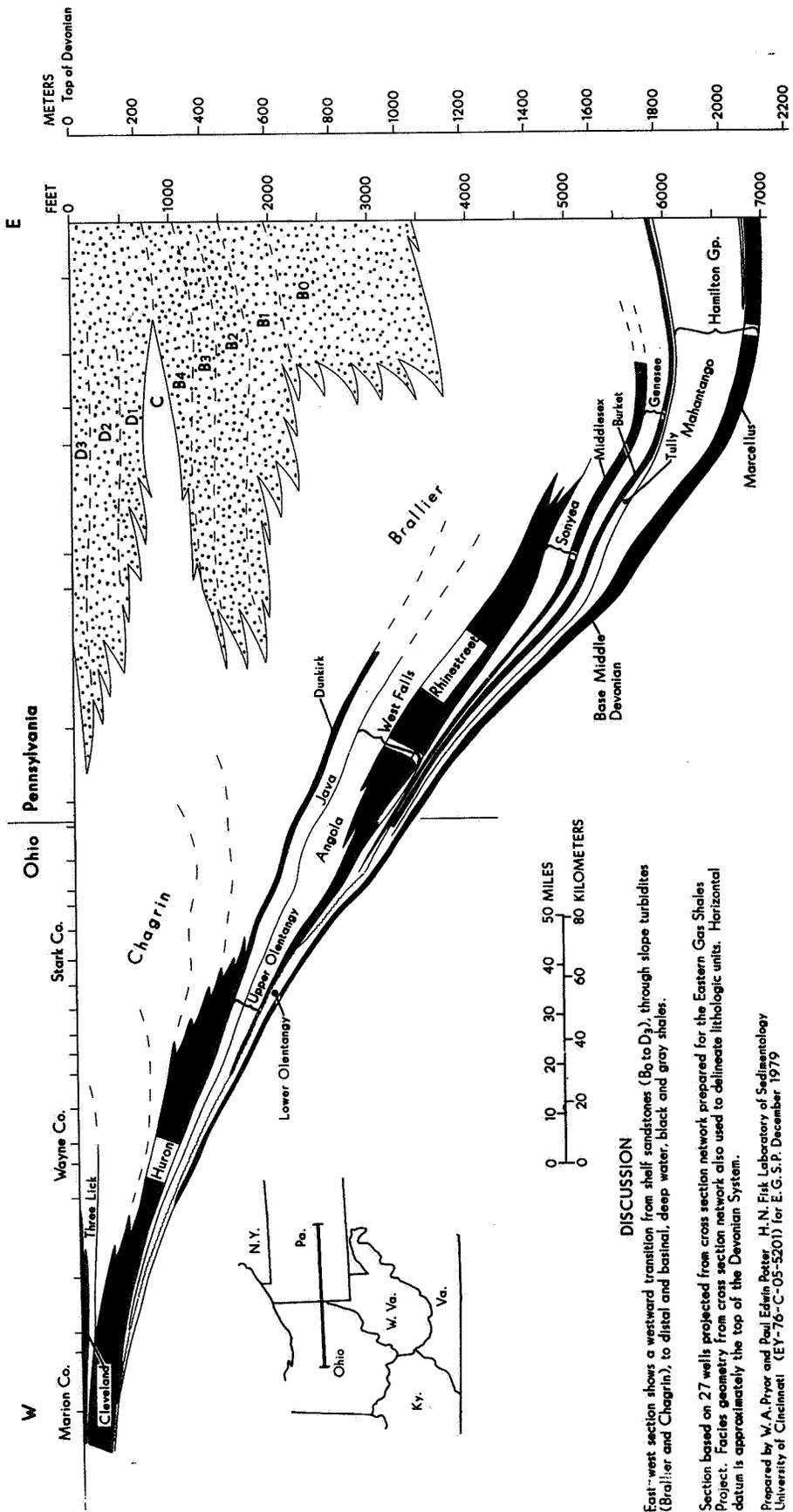


Figure 1. CROSS-SECTIONAL VIEW OF GENERALIZED DEVONIAN SHALE STRATIGRAPHY IN AN EAST-WEST TRAVERSE OF THE CENTRAL APPALACHIAN BASIN

DISCUSSION

East-west section shows a westward transition from shelf sandstones (B₀ to D₃), through slope turbidites (Brallier and Chagrin), to distal and basinal, deep water, black and gray shales.

Section based on 27 wells projected from cross section network prepared for the Eastern Gas Shales Project. Facies geometry from cross section network also used to delineate lithologic units. Horizontal datum is approximately the top of the Devonian System.

Prepared by W.A. Pryor and Paul Edwin Pater, H.N. Fisk Laboratory of Sedimentology University of Cincinnati (EY-76-C-05-5201) for E.G.S.P. December 1979

samples were selected according to a priority list supplied by DOE. This list was based upon formations of interest in geographic areas identified by DOE as having current or future research potential. Eight samples were eventually selected from the EGSP core collected by IGT and were analyzed in the CORAL. Seven of these cores consist of the lower Huron Member of the Ohio Shale. One of these was recovered from southeastern Kentucky, and the other six were obtained from wells along the Ohio-West Virginia border. The Huron in this area has been the subject of a number of field tests funded by both DOE and the Gas Research Institute (GRI). The eighth core consists of Marcellus Shale from a well drilled in Morgantown, West Virginia. The samples used in each CORAL run are listed below:

First Shale Run (CORAL Run No. 51)

| <u>Holder</u> | <u>Well</u> | <u>Depth, ft</u> | <u>Formation</u> | <u>Date Cored</u> |
|---------------|---------------|------------------|------------------|-------------------|
| 1 | EGSP WV-5 | 3028 | L. Huron | Jan. 1978 |
| 2 | Moore #1 (KY) | 2904 | L. Huron | Jun. 1983 |
| 3 | EGSP OH-6/4 | 2771 | L. Huron | Oct. 1979 |
| 4 | EGSP OH-9 | 3245 | L. Huron | Feb. 1981 |

Second Shale Run (CORAL Run No. 52)

| <u>Holder</u> | <u>Well</u> | <u>Depth, ft</u> | <u>Formation</u> | <u>Date Cored</u> |
|---------------|-------------|------------------|------------------|-------------------|
| 1 | EGSP WV-6 | 7448.5 | Marcellus | Apr. 1978 |
| 2 | EGSP OH-6/4 | 2770.8 | L. Huron | Oct. 1979 |
| 3 | EGSP OH-6/5 | 2441.4 | L. Huron | Dec. 1979 |
| 4 | EGSP OH-8 | 3325 | L. Huron | Mar. 1980 |

The locations of each well are shown on the map (Figure 2). Background information on the individual shale zones sampled and tested is given in more detail below.

- Somerset Gas Co., City of Somerset, E. J. Moore No. 1 Well, Leslie Co., Ky., "lower" Huron Member of Ohio Shale, Sampled: 2896-2910 ft (log depths), CORAL plug: 2904 ft (core depth)

This zone of interest at the top of the "lower" Huron has a very high gamma log signal (~461 API units) with a maximum count in excess of 500 API units (see Figure 3). The density curves all indicate a lower value (2.5 g/cm³) than shale units above or below this interval. The temperature log shows a cooling trend below 2895 feet suggesting gas entry into the well bore. Samples consisted of several long, unbroken, unfractured, uniform core segments, composed of organic-rich shale, brownish black (5YR 2/1), thin bedded, with thin calcareous and pyritic laminae.



Figure 2. MAP SHOWING LOCATIONS OF CORED WELLS FROM WHICH CORAL SAMPLES WERE TAKEN



**Somerset Gas / City of Somerset, Ky.
E. J. Moore #1, Leslie Co., Kentucky**

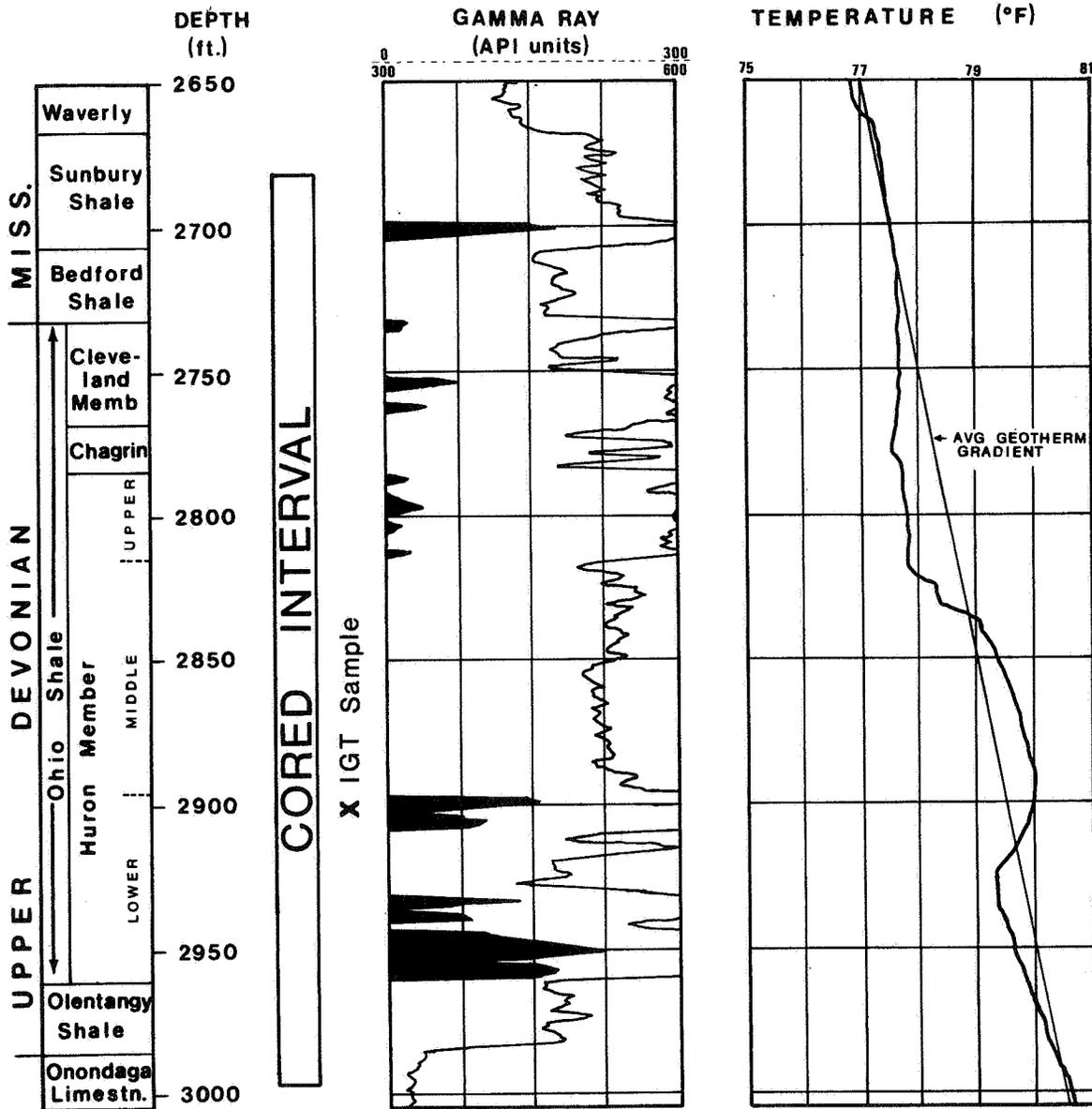


Figure 3. CORE SUMMARY CHART FOR E. J. MOORE NO. 1 WELL
SHOWING STRATIGRAPHIC LOCATION OF IGT SAMPLE

During the time this contract was being negotiated, METC called to our attention the fact that the state government of Kentucky, with DOE support, was drilling and coring a well through the Devonian shale sequence in Leslie County, Kentucky. Since the majority of the EGSP core is 5 to 10 years old, we were enthusiastic about the prospect of sampling fresh shale core. The Leslie County core was cut in June 1983 and sampled by IGT in December 1983. Core descriptions and stratigraphic contracts on the Leslie County core were provided by the Institute of Mining and Minerals Research (IMMR) in Lexington, Kentucky.

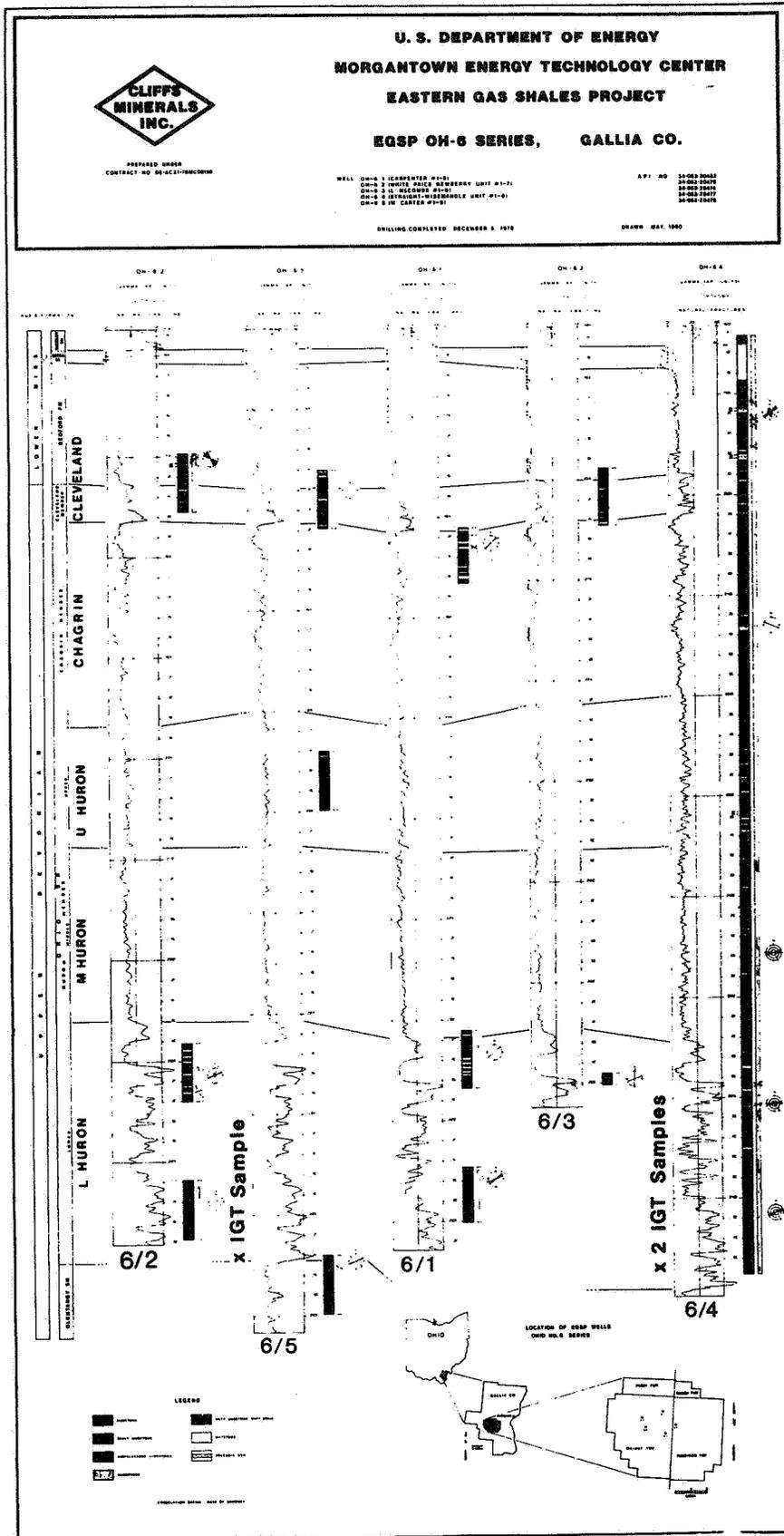
- EGSP OH-6 CORES

Cores that were obtained under the Eastern Gas Shales Project were classified by the U.S. Department of Energy by state and drill hole order. For example, the designation "EGSP KY-2" refers to the second shale core cut in Kentucky under the Eastern Gas Shales Project.

The only exception to this rule is a series of cores cut in Gallia County in southeastern Ohio. Five wells were drilled in a circle approximately 1 mile in diameter, thought to be centered on the flanks of a dome-like structure in the limestone underlying the Devonian shale. The cores in this series were designated as EGSP OH-6, and the individual wells were labeled OH-6/1, OH-6/2, OH-6/3, OH-6/4, and OH-6/5 (Cliffs Minerals Report, June 1980). The stratigraphic intervals from which cores were taken from these wells are shown in Figure 4. IGT plugs that were run in the CORAL were taken from the OH-6/4 and OH-6/5 cores. The stratigraphic positions of these are also shown in Figure 4. The sampled intervals from which the plugs were obtained are described in more detail below:

- EGSP OH-6/4, Mitchell Energy Corp., #1-8 Straight-Wisemandle Unit, Gallia County, Ohio, "lower" Huron Member of Ohio Shale, Sampled 2765-75 ft (log depths), CORAL Plugs: 2770.8 ft, 2771.0 ft (core depths)

This zone of interest near the base of the Huron Member has high gamma intensity (~241 API units) and a density of about 2.47 g/cm³. It is slightly below a foam fractured interval (2512 to 2752 feet) in the well which produced 8 MCF/d of gas. This zone in EGSP OH-6/4 correlates with a highly radioactive shale bed associated with gas production in EGSP OH-9 some 30 miles northeast. This same interval in the Huron Member was sampled by IGT from the EGSP WV-5 well, about 15 miles to the northeast. The core samples collected from EGSP OH-6/4 consist of several 3 to 5-inch long segments of



inter-laminated olive gray (5Y 4/1) and olive black (5Y 2/1) shale, with the black shale predominating. Individual laminae range from less than a millimeter to nearly a centimeter in thickness, and small pyrite nodules and carbonaceous plant fragments occur throughout. No bedding plane cracks or natural fractures were visible between or within the gray and black laminae on the fresh core.

- EGSP OH-6/5, Mitchell Energy Corp., #1-9 M. Carter, Gallia County, Ohio, "lower" Huron Member of the Ohio Shale, Sampled: 2440-46 ft (log depths), CORAL Plug: 2441.4 ft (core depth)

This basal, highly radioactive bed in the "lower" Huron Member, immediately overlying the organic-lean Olentangy Shale, was sampled because it correlates with indications of a gas pay zone at the Huron/Olentangy contact in nearby Meigs County, Ohio. On the reduced-scale gamma ray log of EGSP OH-6/5, the interval shows gamma counts in excess of 300 API units. This footage was included in a larger zone that received a massive foam fracture stimulation (2231 to 2446 feet). That treatment resulted in water problems. Several 4 to 6-inch long core segments were collected from this interval. Many of the samples had a coring-induced petal fracture running vertically down the length of the core; however, the bulk of the core was undisturbed. The samples consist of organic-rich shale, olive black (5Y 2/1), thin bedded with thin pyritic laminae throughout.

- EGSP OH-8, Donohue, Anstey and Morrill, #1 Shockling, Noble County, Ohio, Huron Member of the Ohio Shale, Sampled: 3320-33 ft (log depths), CORAL Plug: 3325 ft (core depth)

This zone in the Huron Shale (Cliffs Minerals Report, October 1980) correlates with the "lower" Huron Member in Ohio and the Dunkirk Member of the Perrysburg Formation in New York. It shows a relatively high gamma ray count (~166 API units) and a density of 2.62 g/cm³. Gas shows were visible in the core. The selected zone of interest is within a foam fractured interval (3138 to 3465 feet), which produced gas at a rate of 52 MCF/d after stimulation. Core samples from this zone consist of one 12 inch long segment of inter-laminated olive black (5Y 2/1) and olive gray (5Y 4/1) shale and a few 3 to 4 inch pieces of olive black core. The CORAL plug sample was cut from one of the small pieces of olive black (5Y 2/1) core and consists of thin bedded, organic-rich shale with a few small pyrite nodules.

Another zone in this well in the Chagrin Member of the Ohio Shale was stimulated and tested at 510 MCF/d, but no core was cut in the pay zone. The stratigraphic location of the IGT core sample is shown in Figure 5.

- EGSP OH-9, Columbia Gas, #10056-A, Meigs County, Ohio, "lower" Huron Member of Ohio Shale, Sampled: 3247-57 ft (log depths), CORAL Plug: 3245 ft (core depth)

This zone was selected for the high gamma ray count (~251 API units) and relatively low-density (2.47 g/cm^3) indications on the full-scale log of well "B." The log features in well "B" in this interval appeared to be about 6 feet different in depth from identical features in well "A" noted in the Cliffs Minerals Report of May 1981. A 6-foot log to core depth correction was deemed appropriate (3253-foot log, well "B" = 3247-foot core, well "A"). The stratigraphic location of the IGT core sample is shown in Figure 6. The core sampled from this zone consists of 4 or 5 short segments (~3 inches long) of uniform, organic-rich shale, brownish black (5YR 2/1), and thick bedded with a few thin pyrite lenses. A sweet hydrocarbon aroma was noticeable on freshly broken surfaces and on the freshly-cut plug. The core had a petal centerline fracture running the length of the segment, but closer to the margin than to the center. The CORAL plug was cut from the larger core section and contained no visible fractures.

EGSP OH-9 NOTE: The EGSP Ohio-9 cores were recovered from the Devonian Shale Offset Well site in Meigs County, Ohio. The original well on this site is designated Columbia Gas #10056. The #10056-A well, from which the IGT core samples were recovered, was drilled 124 feet southwest of the original well. The wireline logs used by IGT were from the 10056-B well, which was drilled 118 feet southeast of the original well. Several other wells have also been drilled on this site more recently.

- EGSP WV-5, Reel Drilling Co., D/K Farm #3, Mason County, West Virginia, Huron Member of Ohio Shale, Sampled: 3036-46 ft (log depths), CORAL Plug: 3028 ft (core depth)

This section near the base of the Huron Member contains beds of high gamma ray count (up to 300 API units) and low density (2.45 g/cm^3), which are indicative of high organic content. The basal contact of the Huron Member with the underlying Hanover Member of the Java Formation (equals "upper" Olentangy Shale in Ohio usage) occurs on the log at 3049 feet. This contact is described in the core description (Cliffs Minerals Report, October 1979) by



PREPARED UNDER
CONTRACT NO. DE AC33 FEMCO318

U. S. DEPARTMENT OF ENERGY
MORGANTOWN ENERGY TECHNOLOGY CENTER
EASTERN GAS SHALES PROJECT
EGSP-OHIO#8, NOBLE CO.

WELL SCHOCKLING 71
DRILLING COMPLETED MARCH 1980

API NO. 24-131-22288
DRAWN OCTOBER 1980

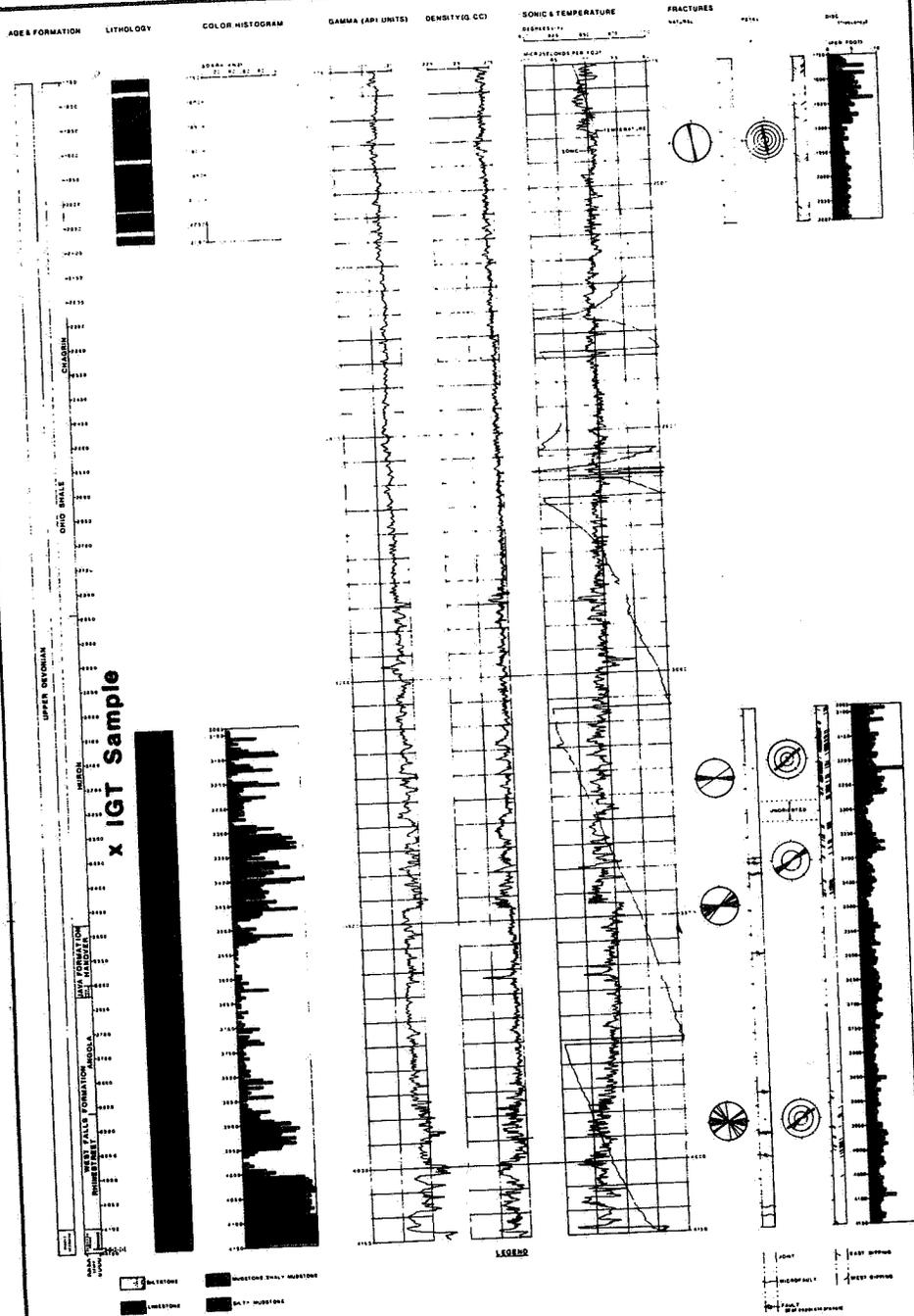


Figure 5. CLIFFS MINERALS CORE SUMMARY CHART FOR EGSP OH-8 WELL
SHOWING STRATIGRAPHIC LOCATION OF IGT SAMPLE



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 EASTERN GAS SHALES PROJECT

EGSP OH-9, MEIGS CO.

PREPARED UNDER
 CONTRACT NO. DE-AC22-78MC18199

WELL COLUMBIA GAS #1056 A A.P. NO. 44-125-2205B
 DRILLING COMPLETED FEBRUARY 1, 1981 DRAWN: MA-1981

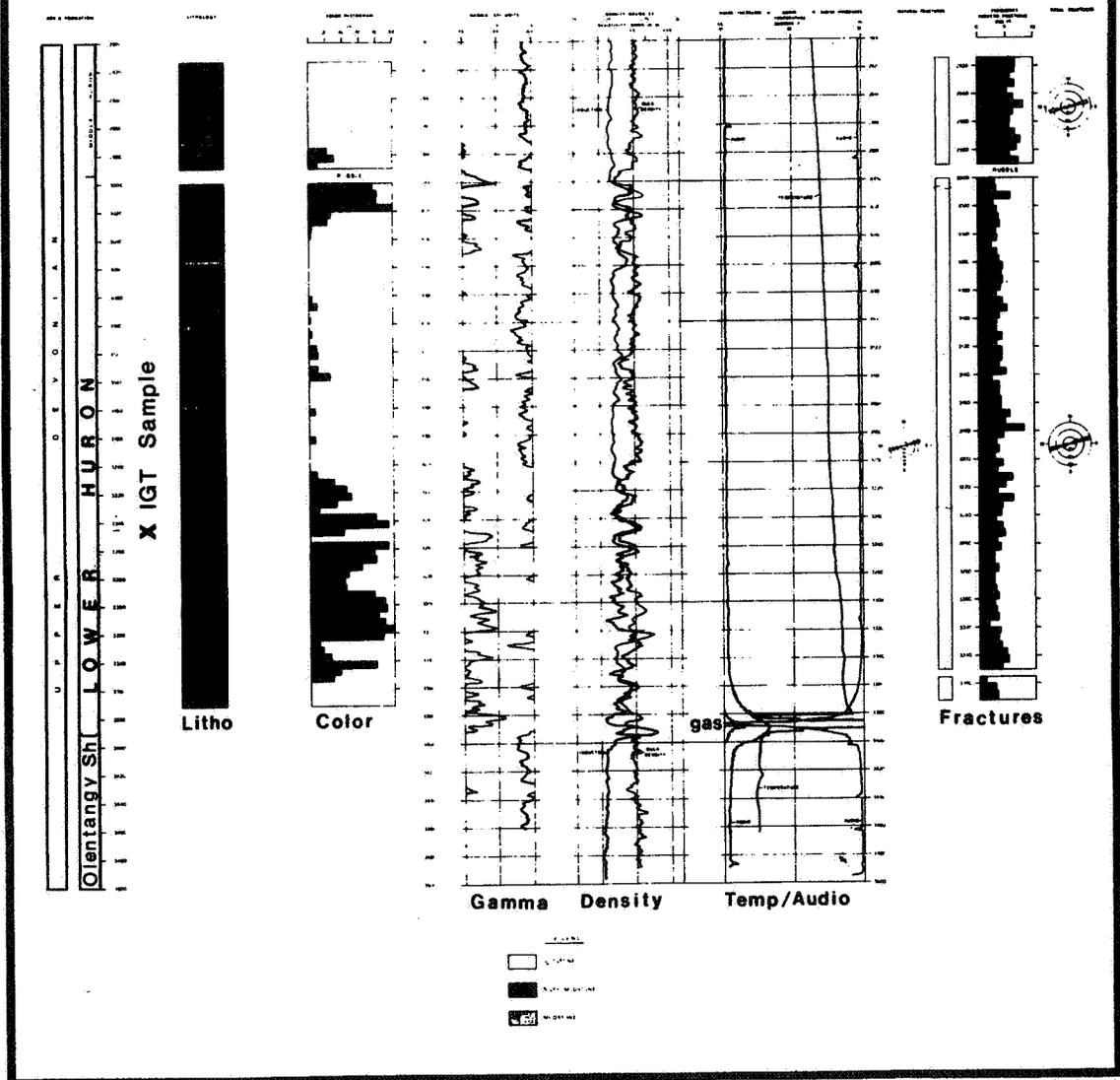


Figure 6. CLIFFS MINERALS CORE SUMMARY CHART FOR EGSP OH-9 WELL SHOWING STRATIGRAPHIC LOCATION OF IGT SAMPLE

color and other changes at 3036 feet and a 13-foot log to core correction was considered applicable (3049 ft log = 3036 ft core).

This zone correlates by gamma ray log with a gassy interval in the EGSP OH-9 well in Meigs County, Ohio, and with gas shows in the EGSP OH-6 series of wells in Gallia County, Ohio. The stratigraphic location of the IGT core sample is shown in Figure 7. The samples recovered from this zone consist of three 4-inch long pieces of shale core. The shale is olive black (5Y 2/1), thick bedded, and contains a few thin gray shale laminae and scattered pyrite lenses.

All of the samples noted above were taken from the Upper Devonian Huron Member of the Ohio Shale out of the western portion of the Appalachian Basin. The sample below was taken from the Middle Devonian Marcellus Shale in a deeper, more central portion of the Appalachian Basin with higher thermal maturity.

- EGSP WV-6, U.S. Department of Energy, M.E.R.C. #1, Monongalia Co., West Virginia, Marcellus Shale, Sampled: 7446-60 ft (log depths), CORAL plug, 7448.5 ft (core depth)

The Marcellus Shale in this interval contains several feet of section with gamma ray counts greater than 300 API units (341 average). The density averages less than 2.46 g/cm^3 and reaches a low of 2.42 g/cm^3 . These indications of high organic content were thought worthy of investigation. The Cliffs Minerals core report describes the shales as "thinly laminated" and "friable" in a section where the sonic log showed large deflections (Cliffs Minerals Report, March 1980). The selected zone of interest is also within an interval perforated (7320 to 7480 feet) and stimulated. The stratigraphic location of the IGT core sample is shown in Figure 8. The core recovered from this zone consists of several short (~2 inch) pieces of friable, fissile, organic-rich shale, grayish black (N2), thin bedded and pyritic. A calcite-filled, near-vertical joint was present near the margin of the core, but was avoided in the plug sample cut for CORAL analysis.

2.2 Sample Preparation

All of the EGSP Devonian shale samples received at IGT consist of oriented core. The Leslie County, Kentucky, core is unoriented. Oriented core is obtained by using a special coring apparatus that cuts three unequally spaced scribe lines on the sides of the core during drilling. One of the



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EASTERN GAS SHALES PROJECT

EGSP WV-5, MASON CO.

WELL: D K FARM #3

A.P.I. NO. 47-053-20146

DRILLING COMPLETED JANUARY 6, 1979

DRAWN: JUNE, 1979

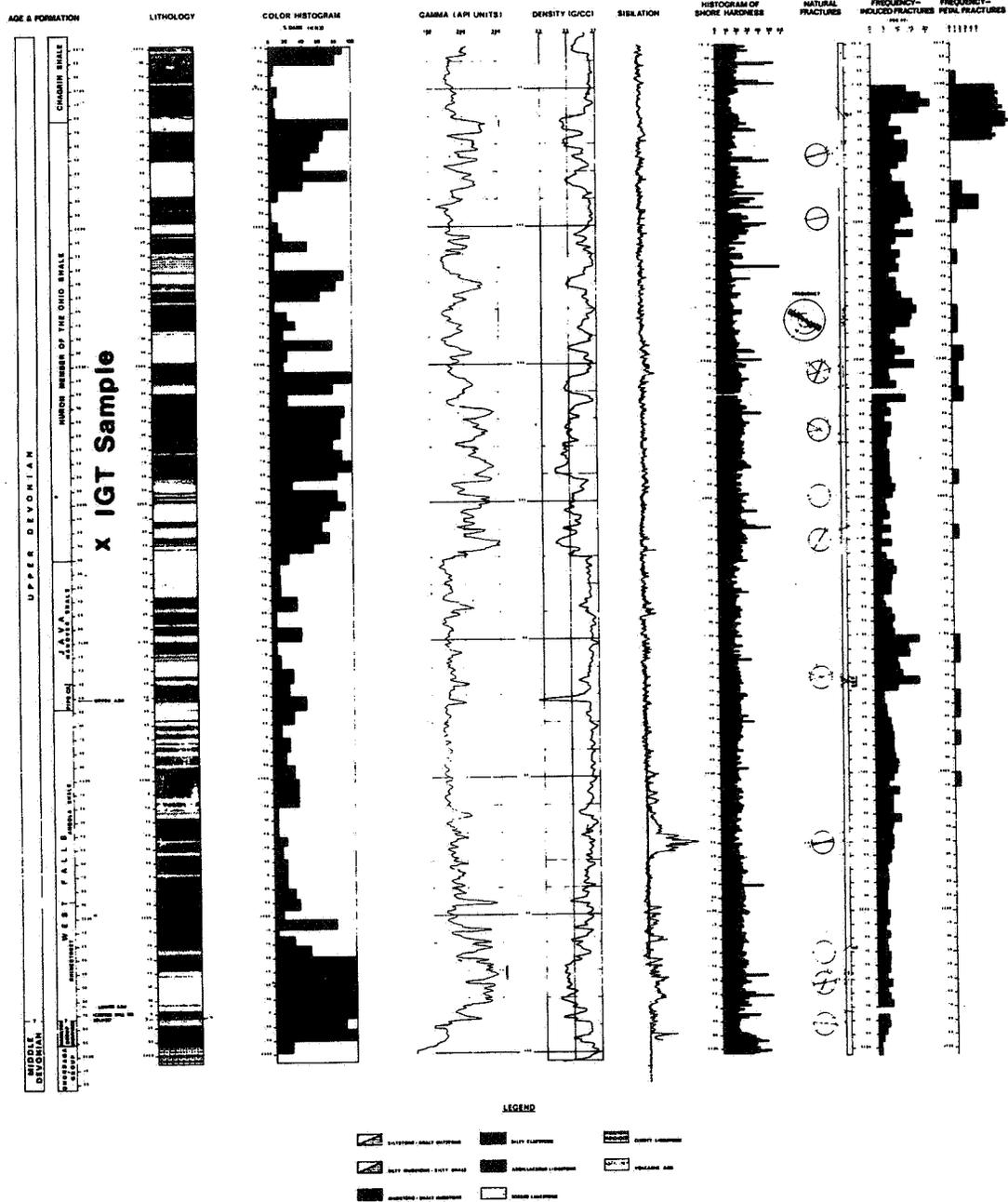


Figure 7. CLIFFS MINERALS CORE SUMMARY CHART FOR EGSP WV-5 WELL SHOWING STRATIGRAPHIC LOCATION OF IGT SAMPLE



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EASTERN GAS SHALES PROJECT

EGSP WV-6, MONONGALIA CO.

WELL M.E.R.C. # 1

A.P.I. NO. 47-081-20370

DRILLING COMPLETED APRIL 1978

DRAWN JULY 1979

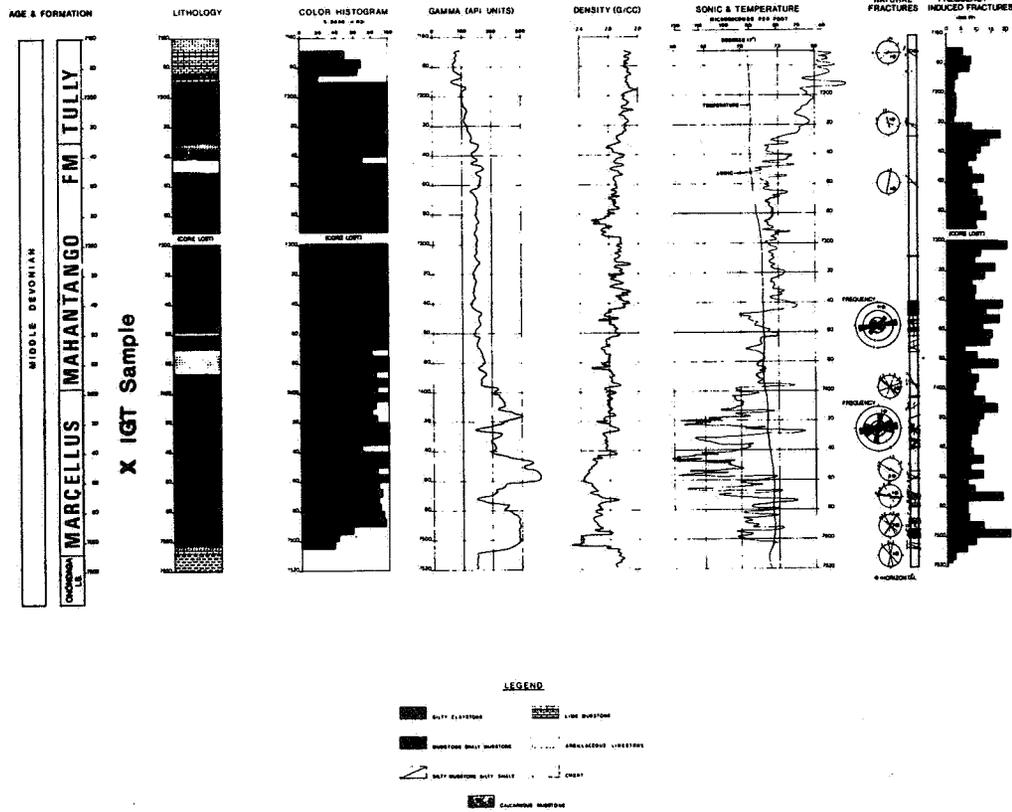


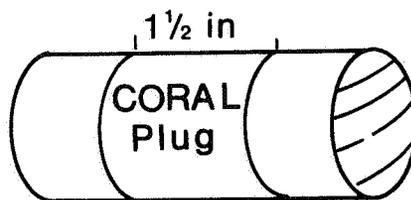
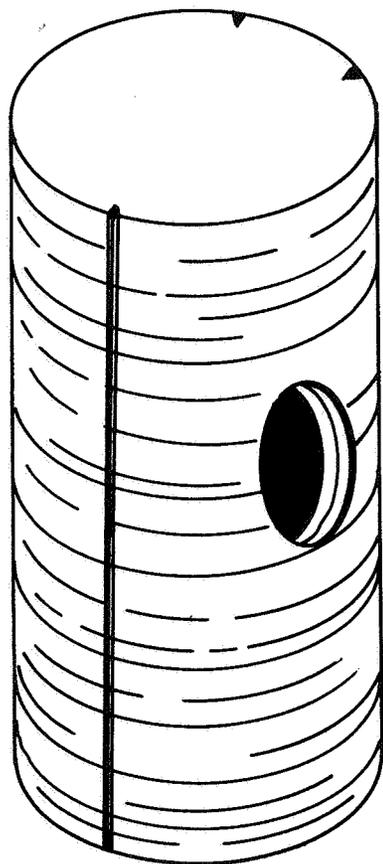
Figure 8. CLIFFS MINERALS CORE SUMMARY CHART FOR EGSP WV-6 WELL SHOWING STRATIGRAPHIC LOCATION OF IGT SAMPLE

scribe lines is widely separated from the other two; this is referred to as the "reference groove." The core barrel is connected to the rest of the drill string with a nonmagnetic drill collar, usually made out of monel or some other nonferrous alloy. A magnetic compass and a camera are mounted in the core barrel below the monel collar. While the core is being cut, the camera records the orientation of the reference groove in the core with respect to the compass direction of magnetic north at intervals of 2 feet. After magnetic north is corrected to geographic north, the true horizontal orientation of the reference groove is given at 2-foot intervals in a core orientation log by the coring contractor.

A plastic ring inscribed from 0 to 360 degrees was placed over the core in the lab as part of the Cliffs Minerals, Inc., EGSP core processing procedure. The ring was rotated until the reference groove was aligned in the direction given in the core orientation log for the sample depth. The positions of north, south, east, and west were then drawn on the core with permanent marker.

Each core selected for analysis was placed in the drill press and plugs were cut as shown in Figure 9. Several cutting attempts were sometimes required to obtain a plug of good integrity. The plugs were generally not drilled in any particular orientation, although orientation of the plug axis was recorded after cutting. The plugs were cut with a 1-1/2 inch ID diamond coring bit. Tap water flows through the bit and out of the drill hole during the plugging operation to cool the diamonds and flush away the cuttings. A fairly low-speed bit with coarse diamonds was found to be necessary to obtain acceptable plugs out of the shale. High-speed bits with fine diamonds tended to "mud-up" during cutting, causing loss of water circulation and sample damage.

After the plugs were cut on the drill press, they were mounted in a small metallurgical saw and trimmed with a thin diamond blade. The disks sliced from the plug ends were saved to provide additional sample material identical to the plug. The plug ends were then lightly sanded to square them off, and the plug length and diameter were carefully measured with a caliper. Six diameters and four lengths were determined for each plug, and averaged values were used in order to minimize the effects of minor surface irregularities on volumes calculated for the samples. The plugs were then weighed on an



1 1/2 inch diameter
Horizontal Plug

3 1/2 inch Oriented Shale Core

Figure 9. PLUG FOR POROSITY AND PERMEABILITY ANALYSIS
CUT HORIZONTALLY FROM DEVONIAN SHALE CORE

electronic balance and placed into a controlled, relative-humidity oven to dry.

The decision to use tap water instead of synthetic brine during plugging and trimming was consciously made after considering the following:

- At the time of plugging, formation brine content of the core was unknown.
- Samples of reservoir brine have not been obtained, as far as could be determined, from any of the EGSP wells cored or from any other Devonian shale source.
- X-ray diffraction results obtained by the U.S. Geological Survey (Hosterman, personal communication, 1980) indicate that the clay mineralogy of these shales consists primarily of illite and kaolinite, with minor amounts of chlorite and water-sensitive smectite or mixed-layer clays.
- The rapid plugging allows little time for water imbibition
- Plugs are placed in a controlled humidity oven soon after plugging so that any imbibed tap water will soon evaporate.

The plugs remained in the relative humidity oven at 60°C and 45% relative humidity until the weights became stable. This condition of "baseline water saturation" removes all free water from the pores, but retains one to two molecular layers of water of hydration on swelling (smectite) clays in the rock. This condition is thought to closely approximate the natural hydration of "dry" rocks in the ground (Bush and Jenkins, 1970).

The use of a relative humidity oven to dry Rocky Mountain tight sand samples in order to avoid the damage to pore clays caused by unhumidified drying has been documented by Randolph and Soeder (1984). It is not known, however, if the conditions used to achieve a "dry" sandstone are appropriate to obtain a "dry" shale. The ages, depths, reservoir pressures, thermal histories, organic contents, bulk compositions, and depositional environments are so different between Rocky Mountain tight sands and Appalachian Devonian shales that it was difficult to see how any set of laboratory procedures developed for the one would apply to the other.

The drying conditions recommended by Bush and Jenkins were designed to retain one to two layers of water of hydration on calcium and sodium smectites. Through calculation of pressure and temperature gradients present in the ground, Bush and Jenkins reached the conclusion that one to two layers

of water of hydration retained on smectites was the natural condition in dry tight sandstone reservoirs at depths of 3000 to 9000 feet. Eastern Devonian shales, however, occur at shallower depths (mostly between 1000 and 5000 feet) and have considerably lower reservoir pressures. Furthermore, eastern shales are abnormally dry with no readily available well reports mentioning the occurrence of measurable water production. These parameters are very different from those found in western tight sand reservoirs, which usually contain at least several tens of percent of immobile water saturation even in "dry gas" zones.

Work by Colten (1984), however, has indicated that two molecular layers of water between silicate sheets is the "preferred" state of hydration for both calcium and sodium smectites through a wide variety of pressure, temperature and salinity conditions. Based on this work, and taking into consideration the lack of published data documenting the natural hydration state of clays in actual shale gas reservoirs, it was decided to dry the shale samples under the conditions recommended by Bush and Jenkins: 60°C at 45% relative humidity. Drying the samples in some form or another was necessary because each plug had absorbed an unknown quantity of coolant water from the drilling and cutting procedure. Without knowing exactly which oven settings would be appropriate for Devonian shale, a compromise was made by drying the shale at the sandstone settings. The reasoning behind this was that even if the hydration state of clays in the shale was not "correct," at least it was known.

2.3 Shale Analysis in the CORAL

The Computer Operated Rock Analysis Lab at IGT deduces flow rates of gas through a sample (and hence permeabilities) by measuring the buildup of gas pressure in a small volume with respect to a reference pressure. An extremely sensitive differential pressure transducer (0.7 psid full scale) is plumbed into the line volume at the downstream end of each of the four coreholders (Figure 10). The permeability sequence starts out with the gas pressure in the downstream line equal to the gas pressure in a 6-liter downstream reference tank. The measurement begins by isolating the line pressure from the tank pressure by closing a computer-activated solenoid valve. With the valve closed, one side of each differential pressure transducer (DPT) is connected to the downstream reference tank, while the other side is connected

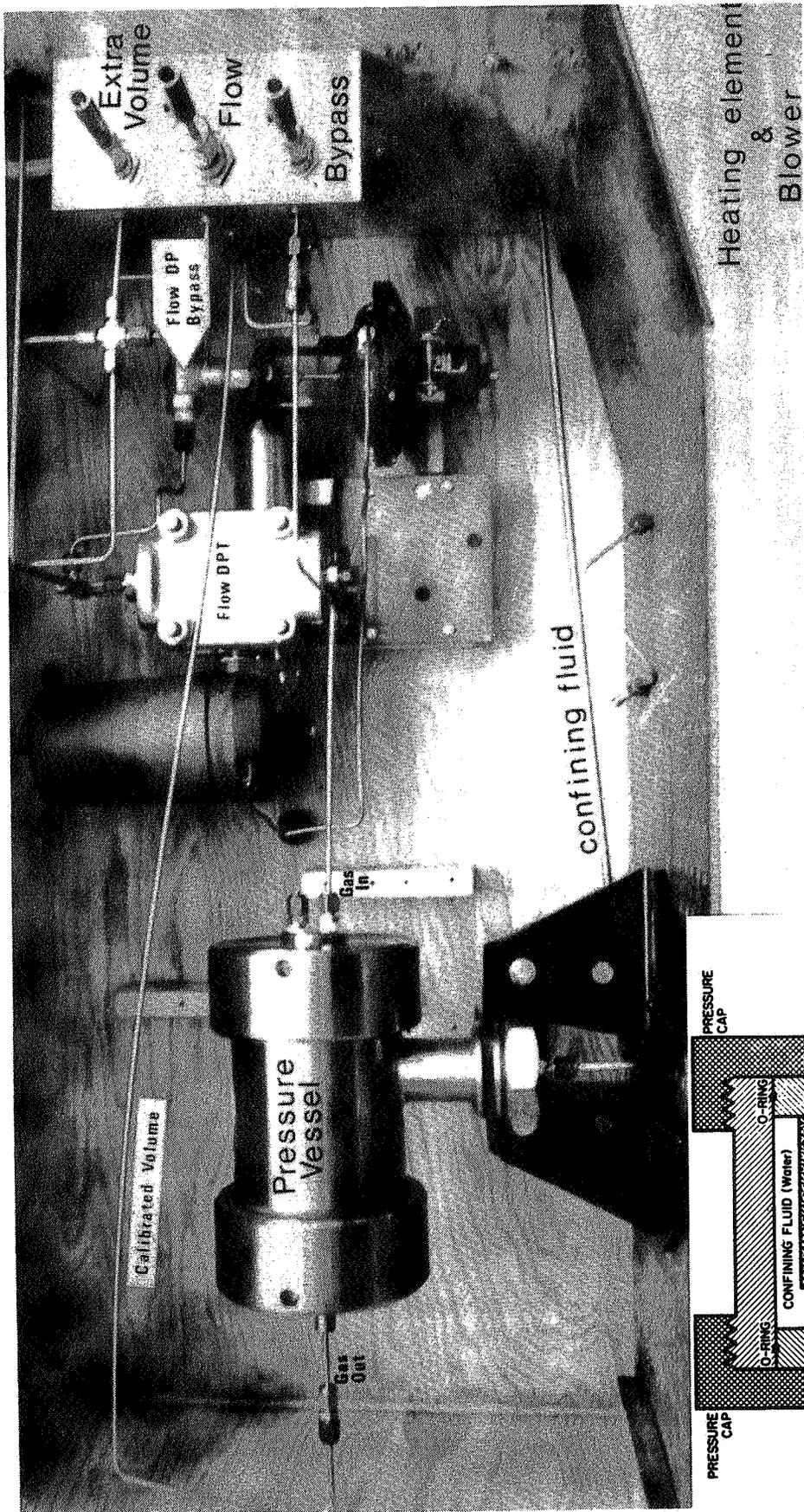
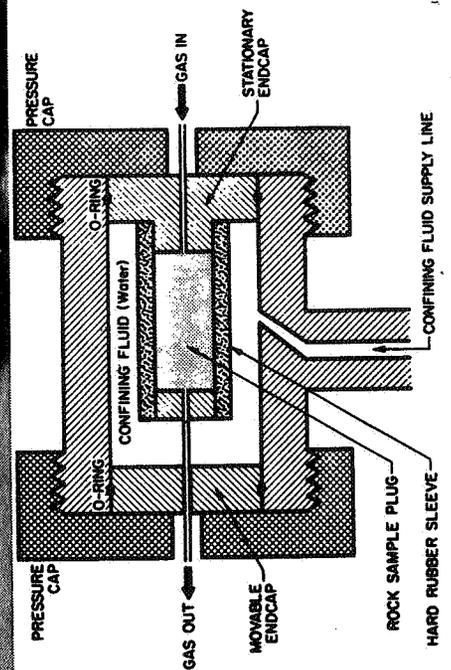


Figure 10. ONE OF THE FOUR COREHOLDER ASSEMBLIES IN THE CORAL SHOWN IN PERMEABILITY CONFIGURATION
 (Inset: Schematic Drawing of the Pressure Vessel Used to Confine the Core Sample)



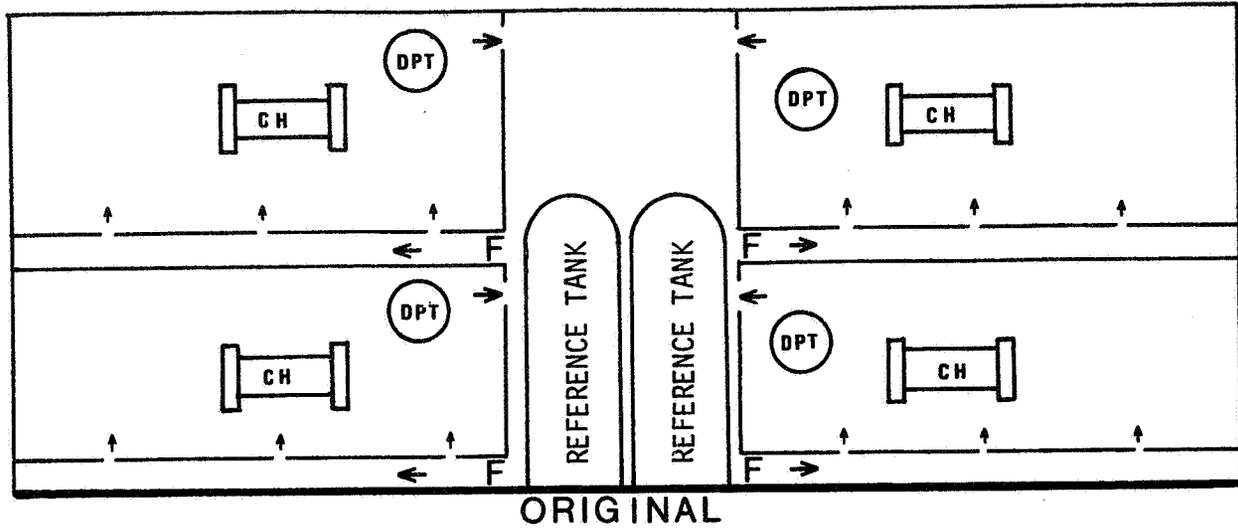
to the downstream line volume of its respective coreholder. As gas from a higher pressure upstream supply tank flows through the core, pressure in the downstream line volume begins to build and a differential pressure is created across the sensitive DPT. When pressure reaches the full scale of the DPT, the computer opens the solenoid valve for 10 seconds to allow the line volume pressure to equilibrate with the downstream tank. After the pressures have equalized, the valve is closed and another buildup sequence begins. By knowing very accurately the volume of the downstream lines and by measuring the pressure buildup in those lines over time, flow rates and permeabilities can be calculated.

Since the maximum differential pressure between the sample pores and the reference tank is never more than 0.7 psid out of a maximum absolute pore pressure of 1500 psi, the CORAL can be considered a "steady state" flow measuring device. Due to the small volumes and high pressures, however, it is extremely sensitive to temperature fluctuations. For this reason, the entire apparatus is enclosed in an isothermal chamber. Temperature control is maintained by a microcomputer that senses the temperature and adjusts power across a set of heating resistors accordingly. The computer also reads the voltage output from the DPT's (first converted to digital signals via a multiprogrammer) and records pressure buildup data on magnetic disk.

As originally constructed, the CORAL was capable of measuring actual gas flow rates lower than 10^{-6} cm³/second and has performed very well on 0.1 to 10 microdarcy tight sand plugs 1 inch in diameter by about 2 inches in length. Measurements of ultra low flow rates in the CORAL are limited by several factors, however, the most important of which are temperature stability and digitizing resolution. The following modifications and improvements were made to increase the sensitivity of the equipment for shale analysis.

- a. Temperature Control. Temperature stability in the CORAL is maintained by forced air circulation across a set of resistance heating elements. The warm air is ducted throughout the system to maintain constant temperatures on all components. Two changes have been made in the temperature control system to increase stability. The air circulation patterns were altered (as shown in Figure 11) to more evenly distribute heat throughout the enclosure, and modifications were made to the temperature control algorithm used by the computer to power the heating coils. The new circulation system has increased the temperature stability considerably and also reduced the amount of time needed by the CORAL to eliminate temperature transients caused by changing gas pressures or opening doors. The modifications to the control algorithm

CORAL AIR CIRCULATION



CH= coreholder

DPT= differential pressure transducer

F= fan

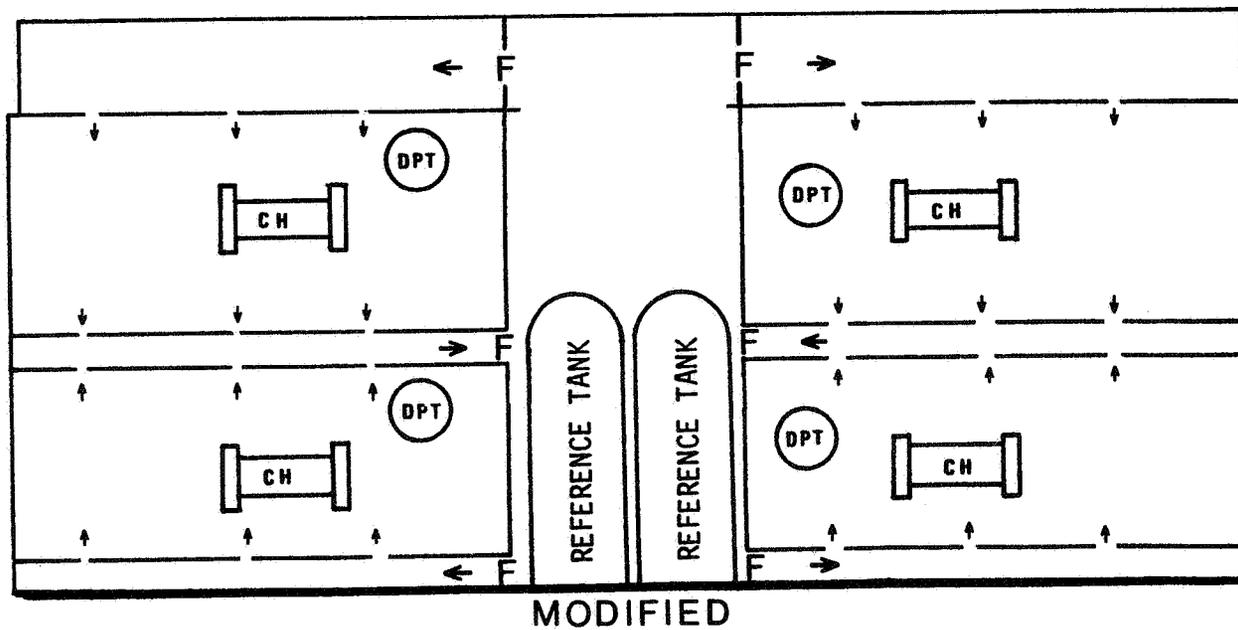


Figure 11. SCHEMATIC DIAGRAM OF CORAL BEFORE AND AFTER CHANGE IN AIR CIRCULATION PATTERNS

now permit the computer to deduce and predict temperature trends inside the system and adjust heating power levels before trends reach maxima or minima. In effect, this new control flattens out the peaks and valleys between temperature swings.

- b. Digitizing Resolution. Data output from the CORAL temperature sensors and pressure transducers is in DC volts. For the computer to read this information, it must be converted into digital signals. This is the function of the multiprogrammer and multiprogrammer interface, which are wired into the signal path between the CORAL and the computer. The number of digital steps into which the entire voltage range output of the transducers can be divided determines the pressure and the temperature resolution of each digitizing step. More digitizing steps per volt means that each of these steps correspond to a smaller fraction of this volt. To achieve greater digital resolution with the CORAL, the multiprogrammer and multiprogrammer interface were replaced with a digital data logger. This data logger utilizes about 25 times the number of digitizing steps used by the multiprogrammer and increased the CORAL's digitizing resolution accordingly.
- c. Flow Rates. A relatively simple method of enhancing the permeability measurement resolution in extremely tight rocks is to increase the flow rates of gas through the samples. This can be done by increasing the differential pressure across the core plug and/or cutting the plugs to a larger diameter and shorter length. IGT utilized both of these approaches on the shale. New end caps, 1-1/2 inches in diameter, replaced the original 1-inch-diameter end caps and allowed use of larger-diameter shale plugs with a corresponding increase in flow rates. Higher differential pressures up to 100 psid were applied to the shale plugs to force measurable amounts of gas through the samples. Differential pressures on the sandstone plugs were held at 20 psid or less to prevent mobile pore water from moving around during relative permeability measurements. Because the shales were thought to be dry, there were no limits imposed on the ΔP necessary to flow gas through the core plug.

2.4 Procedures for Shale Core Analysis

After the plugs reached stable weights in the relative humidity chamber, each set was removed and placed in warm, capped bottles. This was done to prevent the plugs from either absorbing additional water out of the room air or losing water by condensation on the bottle. Each plug was removed from its bottle and weighed on the electronic balance under a tared, inverted glass beaker. The plug was then inserted into a Viton sleeve and loaded into one of the CORAL coreholders. Air pressure at 50 psi was applied initially to confine the plug and check for sleeve leaks. Pressurized water is used for confining fluid only after we are assured of no leaks to keep from wetting the sample.

Measurements on all of the lower Huron cores were made at net confining stress values of 1750 and 3000 psia. Measurements on the Marcellus sample were made at 3000 and 6000 psia net confining stress. These values were picked on the assumption that they would bracket the actual in-situ net confining stress. Confining pressure is imposed vertically by the weight of rocks above a particular sample depth and horizontally by tectonic forces around a sample in the ground. Net confining stress is the value of this confining pressure squeezing inward on the rock offset to some degree by fluid or gas pressure within the pores pushing back. Running porosity and permeability measurements under conditions duplicating reservoir net confining pressure is important because the size and shape of the pores in the rock are influenced by this stress. To reproduce the reservoir pore geometry, one must reproduce the reservoir net confining stress.

Our efforts to impose realistic in-situ values of net confining stress on the Devonian shale cores were hampered considerably by the absence of valid and reliable well test pressure data. To determine a representative value of net stress to use on a core, it is necessary to know the reservoir pressure from the depth at which the core was cut, along with some reasonable average value of the three confining stresses imposed on the rock (lithostatic, horizontal minimum, and horizontal maximum).

As far as we can determine, actual reservoir pressure measurements for Devonian shale are extremely rare, although what little data do exist seem to suggest that shale pores are underpressured. For tight sands, we had calculated the pore pressure from the mud weight data when available; otherwise, we had assumed hydrostatic. This mud weight information was not available for any of the shale cores, but we were reluctant to assume hydrostatic pressure gradients due to the apparent lack of a water phase in the shale pore systems. We were also frustrated in our efforts to determine representative confining pressures for Devonian shale. In our tight sands work, we had estimated uniform triaxial confining stress at a value of about 0.925 psi per foot of depth. This figure was picked as a reasonable average between lithostatic, horizontal minimum, and horizontal maximum based on many years worth of tight sandstone fracture gradient measurements. There are not very many of these in-situ measurements for Devonian shale, but the few figures that are available indicate that the 0.925 psi/ft value used on tight

sands is not appropriate for eastern gas shale. Unfortunately, these limited data do not suggest which values are appropriate.

In a compromise based on a lack of data, it was finally decided to run the shale cores at two values of net confining stress that would bracket the true reservoir pressures. Based on the average density of sedimentary rocks, a vertical lithostatic pressure gradient of about 1 psi per foot can be assumed. This pressure will be offset to some degree by tectonically imposed horizontal stress. The hydrostatic pressure gradient is about 0.43 psi per foot, so a fair assumption regarding a value for a uniformly imposed triaxial stress is to locate it between straight lithostatic stress (~1 psi/ft) and straight hydrostatic stress (~0.43 psi/ft). For an initial experiment, pressures bracketed in this manner are acceptable, but future work will definitely require more reliable reservoir pressure and in-situ stress determinations in order to provide results that more accurately describe nature.

The laboratory procedure used for shale analysis in the CORAL is described below:

1. After the plugs were loaded in the CORAL coreholders and the system was leak tested, pressures were taken to 1000 psia net confining stress (1200 psia confining, 200 psia pore) and allowed to stabilize.
2. Pore volume compressibility was measured in steps from 1000 psia net stress to the low value of reservoir net stress (1750 psia for the Huron samples, 3000 psia for the Marcellus core).
3. Permeabilities to gas were run at the low value of net confining stress with pore pressures of 75, 200, 500, and 1000 psia. Porosity was measured at the low net stress using 1000 psia gas pressure.
4. Pore volume compressibility was measured in steps from the low net stress (1750 psia Huron, 3000 psia Marcellus) to the high net stress values (3000 psia Huron, 6000 psia Marcellus).
5. Permeabilities to gas were run at the high value of net confining stress with pore pressures of 75, 200, 500, and 1000 psia. Porosity was measured at the high net stress using 1000 psia gas pressure.
6. Because gas permeability measurements of the Huron cores were behaving as if a mobile liquid phase was present in the pores, additional permeability work was performed on these samples in the second CORAL run to determine permeability to gas as a function of differential pressure and to try to measure the capillary pressure of the liquid in the pores. These measurements were made at both high (3000 psia) and low

(1750 psia) values of net stress with pore pressures of 1000, 500, 200, and 50 psia.

7. Porosity measurements on the Marcellus core had been made with nitrogen at gas pressures of 1000 psia and 200 psia, and the results differed by over 6 porosity percent. This nonlinear behavior of gas-filled pore volume varying with pressure was indicative of some sort of adsorption phenomenon taking place inside the rock pores. Consequently, the CORAL was changed over to methane, and a series of gas-filled porosities were measured on the Marcellus core at methane pressures of 45 psia to 1500 psia. The Marcellus sample was maintained at a net confining stress of 6000 psia during these methane porosity measurements.

3.0 RESULTS

The results of the Devonian shale core analysis work performed under this contract are displayed in Table 2. Each of the eight samples occupies a vertical column in the table. The individual lines on the table display specific data for each core plug horizontally. These lines are discussed in detail below.

Lines 1 through 4: These lines are used to identify the state, county, lease and drill hole from which each core was recovered. The well identification line (Line 1) denotes the core's classification under the DOE Eastern Gas Shales Project.

Line 5: Depth in feet of the plug sample cut from the core and analyzed in the CORAL. The value reported is "drillers" depth — measured on the geolograph while cutting the core and marked on the core itself in permanent ink during the core recovery procedure. Depths listed on this line were taken directly off the core.

Line 6: Rock formation from which the plug sample was taken. Two are listed: the "lower" Huron Member of the Ohio Shale, which is Upper Devonian in age, and the Marcellus Shale member of the Hamilton Group, which is Middle Devonian in age. Refer back to Figure 1 for a cross section of Appalachian Basin stratigraphy to locate these formations relative to one another.

Line 7: Although DOE did not specify a direction from which to cut the plugs, horizontal orientations of the plug axes were recorded anyway from oriented core. These were recorded as standard azimuthal directions; for example, N 15° W means the plug axis lies 15 degrees to the west of a line pointing due north.

Line 8: Rock colors were identified in accordance with procedures used by DOE and the EGSP field team. The rocks were dampened with a wet sponge to eliminate color errors caused by dirt, dust, scratches, or rough surfaces on the core. Colors were identified by comparison with paint chips on the standard Rock Color Chart of the Geological Society of America (1979), which incorporates the widely accepted Munsell color system. The Munsell system uses a unique alphanumeric designation to precisely describe each color according to "hue" (tint), "value" (lightness), and "chroma" (intensity). For example, 5Y 2/1 designates a middle yellow or olive (5Y), very dark and low

Table 2. DEVONIAN SHALE CORE ANALYSIS SUMMARY

| Well Identification | EGSP WV-5 | | Somerset Gas | | EGSP OH-6/4 | | EGSP OH-9 | |
|--|-------------|---------------|----------------|-----------------------|---------------------------|--------|-----------------------------|-----------------------|
| | DK Farm #3 | Mason | E. J. Moore #1 | Leslie | Straight - Wisemandle 1-8 | Gallia | Meigs | Columbia Gas #10056-A |
| 2. Lease | | West Virginia | Kentucky | Ohio | | | Ohio | |
| 3. County | | | | | | | | |
| 4. State | | | | | | | | |
| 5. Depth, ft | 3028 | | 2904 | 2771 | | | 3245 | |
| 6. Formation | L. Huron | | L. Huron | L. Huron | | | L. Huron | |
| 7. Orientation (Horizontal) | N 90° E | | Unoriented | N 35° W | | | N 15° W | |
| 8. Color (Wet) | 5 Y 2/1 | | 5 YR 2/1 | 5 Y 2/1, 5 Y 4/1 | | | 5 YR 2/1 | |
| 9. Gamma Over SBL* (API Units) | 127 | | 321 | 121 | | | 91 | |
| 10. Wire Line Log Density, g/cm ³ | 2.47 | | 2.50 | 2.47 | | | 2.61 | |
| 11. Date Cored | Jan. 1978 | | June 1983 | Oct. 1979 | | | Feb. 1981 | |
| 12. Date Analyzed | 7/16/84 | | 7/16/84 | 7/16/84 | | | 7/16/84 | |
| 13. CORAL Run No. | 51 | | 51 | 51 | | | 51 | |
| 14. Coreholder No. | 1 | | 2 | 3 | | | 4 | |
| 15. Plug Bulk Density, g/cm ³ | 2.42 | | 2.43 | 2.62 | | | 2.52 | |
| 16. Low Net Stress, psia | 1750 | | 1750 | 1750 | | | 1750 | |
| 17. Permeability to Gas** (K _{co}) | 66 nd | | 22 nd | 6.8µd | | | ~1 nd | |
| 18. Porosity to Gas** (± 0.1%) | <0.1% | | 0.12% | <0.1% | | | <0.1% | |
| 18A Pore Vol. Compress (10 ⁻⁶ /psi) | 21.51 | | 18.40 | 8.34 | | | 26.67 | |
| 19. High Net Stress, psia | 3000 | | 3000 | 3000 | | | 3000 | |
| 20. Permeability to Gas** (K _{co}) | 14 nd | | 5 nd | 4.5 µd | | | <0.2 nd | |
| 21. Porosity to Gas** (± 0.1%) | <0.1% | | <0.1% | <0.1% | | | <0.1% | |
| 21A Pore Vol. Compress (10 ⁻⁶ /psi) | 4.78 | | 1.15 | N/A | | | N/A | |
| 22. Carbonate Present | NO | | YES | NO | | | NO | |
| 23. Total Carbon, wt % | 5.22 | | 8.08 | 2.54 | | | N/A | |
| 24. Hydrogen, wt % | 0.92 | | 0.98 | 0.63 | | | N/A | |
| 25. Nitrogen, wt % | 0.21 | | 0.30 | 0.11 | | | N/A | |
| 26. Petroleum Present | Probable | | Probable | Yes | | | Yes | |
| 27. Full Width Cracks | 1 | | 1 | 1 | | | 0 | |
| 28. Short Cracks | 2 | | 3 | 1 | | | 4 | |
| 29. Remarks | Oldest core | | Freshest core | Plug cracked in CORAL | | | Oily aroma on fresh surface | |

* Shale baseline (SBL) for organic-lean shale.

** Gas used for these measurements was nitrogen.

Table 2, Cont. DEVONIAN SHALE CORE ANALYSIS SUMMARY

| | EGSP WV-6 | EGSP OH-6/4 | EGSP OH-6/5 | EGSH OH-8 |
|---|--------------------------------|--------------------------|----------------------------|--------------------------|
| 1. Well Identification | MERC #1 | Straight-Wisemandle #1-8 | Carter #1-9 | #1 Shockling |
| 2. Lease | Monongalia | Gallia | Gallia | Noble |
| 3. County | West Virginia | Ohio | Ohio | Ohio |
| 4. State | | | | |
| 5. Depth, ft | 7448.5 | 2770.8 | 2441.4 | 3325 |
| 6. Formation | Marcellus | L. Huron | L. Huron | L. Huron |
| 7. Orientation (Horizontal) | N-S | N 35° W | N 25° W | N 35° W |
| 8. Color (Wet) | N2 | 5 Y 2/1 | 5 Y 2/1 | 5 Y 2/1 |
| 9. Gamma Over SBL* (API Units) | 196 | 121 | 140 | 26 |
| 10. Wireline Log Density, g/cm ³ | 2.46 | 2.47 | 2.50 | 2.56 |
| 11. Date Cored | Apr. 1978 | Oct. 1979 | Dec. 1979 | Mar. 1980 |
| 12. Date Analyzed | 8/24/84 | 8/24/84 | 8/24/84 | 8/24/84 |
| 13. CORAL Run No. | 52 | 52 | 52 | 52 |
| 14. Coreholder No. | 1 | 2 | 3 | 4 |
| 15. Plug Bulk Density, g/cm ³ | 2.44 | 2.55 | 2.33 | 2.59 |
| 16. Low Net Stress, psi | 3000 | 1750 | 1750 | 1750 |
| 17. Permeability to Gas** (K _g) | 19.613 μd | 8.342 μd | 248 nd | 194 nd |
| 18. Porosity to Gas** (± 0.1%) | 9.28% | 0.15% | <0.1% | <0.1% |
| 19. High Net Stress, psi | 6000 | 3000 | 3000 | 3000 |
| 20. Permeability to Gas** (K _g) | 5.909 μd | 5.489 μd | 8.0 nd | 78 nd |
| 21. Porosity to Gas** (± 0.1%) | 8.67% | <0.1% | <0.1% | <0.1% |
| 22. Carbonate Present | YES | NO | NO | NO |
| 23. Total Carbon, wt % | 9.14 | 2.54 | N/A | 2.00 |
| 24. Hydrogen, wt % | 0.57 | 0.63 | N/A | 0.65 |
| 25. Nitrogen, wt % | 0.35 | 0.11 | N/A | 0.12 |
| 26. Petroleum Present | No | Yes | Yes | Yes |
| 27. Full Width Cracks | 3 | 1 | 0 | 1 |
| 28. Short Cracks | 6 | 2 | 19 | 3 |
| 29. Remarks | High porosity and permeability | 2nd OH-6/4 sample | Pyritic lam. oil in cracks | Oily aroma oil in cracks |

* Shale baseline (SBL) for organic-lean shale.

** Gas used for these measurements was nitrogen.

intensity color (2/1). Thus, the shale colors listed on Line 8 range in hue from olive (5Y) to brown (5YR) to neutral gray (N); in value from medium dark (4) to nearly black (2), and all are low in chroma or color intensity. These colors are very typical for eastern Devonian shales, and because these particular samples are rich in organic matter, the colors are quite dark. Organic-lean shales tend to be lighter in color.

Lines 9 and 10: These lines contain data picked from wireline logs in the vicinity of plug depths. The gamma intensity is given in API units over and above the normal gamma intensity for organic-lean shales in the well. Because organic matter has an affinity for radioactive elements, subtracting out the gray-shale background radiation permits a rough estimation of the amount of organics present in a black shale (Leventhal, 1981). Density logs are also a useful measurement for estimating organic content in black shales (Schmoker, 1978). Since organics are less dense than minerals, lower density generally corresponds to higher organic content. Log densities are given on Line 10.

Line 11: The month and year in which each respective core was cut and recovered are recorded on this line. The age of the core does not appear to be important to the results of the analysis, as long as the samples chosen for analysis consist of intact, well-preserved core segments. It is our observation that shale core tends to develop fissility (parallel horizontal cracks) when exposed to air. Older cores generally showed a greater degree of deterioration than fresher cores. Cores kept sealed in cans or carefully wrapped in plastic sheeting, however, were often freshly preserved after 5 years' time. (See Line 27 explanation below.)

Lines 12 and 13: Cores were analyzed in two runs of four plugs each. CORAL Run No. 51 began on July 16, 1984, with the four plugs listed on Page 34. Plugs listed on Page 35 were analyzed in CORAL Run No. 52, which began on August 24, 1984. The run numbers are unique to each set of plugs and permit IGT to file the raw data in an efficient and retrievable manner.

Line 14: This line lists the particular coreholder out of the four in the CORAL which contained the plug during analysis.

Line 15: Dry bulk density of the plug was derived simply by dividing the measured dry weight by the measured volume. This value is useful for

screening out siderite concretions, pyrite nodules, and other unusual core features not representative of the formation.

Lines 16, 17, and 18: Results of the CORAL analysis made under conditions of low net stress. Net stress values are listed in Line 16. Permeability to gas at the Klinkenberg intercept (K_{∞}) is listed on Line 17. The values are given in microdarcies (μd) equivalent to 10^{-3} millidarcy, or in nanodarcies (nd) equivalent to 10^{-6} millidarcy. In cases where the Klinkenberg plot was too scattered to accurately determine a slope and an intercept, the permeability value listed consists of a single measurement taken at the highest pore pressure (1000 psia). Porosity to gas listed on Line 18 was measured using a Boyle's Law method and a pressure step from 1000 to 1100 psia. Nitrogen gas was used for both the porosity and permeability measurements. Gas porosity of the Huron cores was extremely low, making the pore volume compressibility measurements, where given, very questionable.

Lines 19, 20, and 21: Results of the same CORAL measurements described above, but performed under conditions of high net stress values listed on Line 19.

Line 22: Presence or absence of carbonate minerals was determined by placing a drop of 10% hydrochloric acid on a fresh surface and watching for effervescence. Rapid fizzing indicates the presence of calcite or siderite; slow effervescence is indicative of dolomite.

Lines 23, 24, and 25: Results of elemental analysis performed by IGT Analytical Chemistry Lab on chip samples from all cores except OH-6/5 and OH-9. Chips were, in most cases, taken from the cores fairly close (within 1 to 2 ft) of the plug location. Chips were analyzed by flash pyrolysis at 1000°C in a Carlo-ERBA Analyzer Model 1106. This device combusts the sample in a helium atmosphere with a limited amount of oxygen. The combustion products are then separated and identified in a gas chromatography column. Although this technique is a relatively inexpensive method of determining total carbon content, it does not discriminate between inorganic and organic carbon.

Line 26: The presence of petroleum in the shale cores was noted by a) permeability behavior suggestive of a mobile liquid phase in samples that had been dried of all free pore waters and/or b) results of chromatographic analysis indicating the presence of a paraffinic petroleum in the rock. Low porosity to gas is another clue to the presence of oil in the pores.

Lines 27 and 28: Numbers of horizontal, bedding plane cracks counted on one sawed face of each plug. Full width cracks span the entire face and sometimes also run the entire length of the plug. Short cracks terminate within the plug face. These cracks were counted after the plugs had been run in CORAL and do not appear to correlate with permeability. Cores with one full-length crack, for example, have permeabilities ranging from 5 nanodarcies (Moore No. 1) to about 5 microdarcies (both OH-6/4 plugs). One of the tighter rocks was the OH-6/5 plug, which contains 19 short cracks. The most permeable core (WV-6) contains more cracks overall than the others, but this core differs also in depth, age, formation, and thermal maturity. One of us (D. Soeder) observed this core fresh in 1979 at Cliffs Minerals, Inc., and it contained the same large number of cracks that it does now. The question as to whether the age of shale core affects the results of analysis when macroscopically non-fissile segments are sampled is best demonstrated by comparison of the oldest EGSP core (WV-5) with the freshest (Moore No. 1). Both cores have about the same composition, bulk density, color, and number of plug cracks. Both have roughly similar porosities, permeabilities, and stress dependences of permeability. Comparison of the WV-5 core with other cores of about the same age (WV-6 and the OH-6 samples) shows that gas permeability and the stress dependence of this value are controlled by factors other than core age and macroscopic fissility.

Line 29: Some of the remarks on this line are expanded below:

- OH-6/4: The first plug from this core contained a large horizontal crack when removed from the CORAL; in fact, the plug was actually split in two lengthwise. We were unsure of the contribution that this crack may have made to the permeability measurements, so a second OH-6/4 plug was cut for the second CORAL shale run; it gave very similar results.
- OH-6/5, OH-8: Both of these plugs showed oily discolorations along bedding plane cracks after removal from the CORAL. Both contained sufficient quantities of mobile liquid to shut off gas flow.
- OH-9: This core contained a very distinct and quite strong kerosene-like oily aroma on fresh broken surfaces that dissipated in a matter of minutes.
- WV-6: This core contained a near-vertical, calcite-filled natural fracture that was close to, but not contained in, the horizontal plug sample. This sample also exhibited the highest porosity and permeability of any of the eight shale cores analyzed. This is described in more detail below.

3.1 Porosity of Devonian Shale

Pore volume available to gas was measured on all the samples using a Boyle's Law method with nitrogen gas and a pressure step from 1100 to 1200 psia. The Huron Shale cores exhibited an extremely low porosity to gas, near or below the measurement limits of the CORAL. Measured Huron shale gas porosities ranged from 0.15% to less than 0.10%, the measurement cutoff point for the CORAL. The WV-6 (Marcellus Shale) core gave a gas-filled porosity of $8.67\% \pm 0.1\%$ at 6000 psi net stress, and a value of $9.28\% \pm 0.3\%$ at a net stress of 3000 psi.

Because the Devonian black shales of the Appalachian Basin contain coaly organic material, it has long been suspected that there is an "adsorption" component to a portion of the gas contained in these rocks. Adsorption is a phenomenon by which molecules of a gas or liquid attach themselves to the surface of an electrochemically active solid, such as carbon in coal. To determine whether or not adsorption was indeed an important part of the gas entrapment mechanism in Devonian shale, it was decided to repeat the Boyle's Law porosity measurement on the Marcellus core under the same net stress but at a lower gas pressure. Because the net stress was identical to that used during the previous measurement, the pore volumes should come out the same if free pore space alone was being measured. If some of the gas was being adsorbed onto carbonaceous surfaces, however, the pore volume would appear to be larger at lower pressures. The reason for this is that only a finite number of gas molecules can attach themselves to an attractive surface of limited area. At lower pressures, a larger proportion of the gas molecules in the pores adsorb, and the effect is more noticeable.

As mentioned above, the first Boyle's Law measurement on the Marcellus Shale core using nitrogen gas at a mean pressure of 1150 psia gave a porosity of 8.67%. When this measurement was repeated with nitrogen gas using a pressure step from 200 to 300 psia, the porosity value under the same net stress was 15.35%, an increase of almost 7 porosity percent. This result indicated that gas was indeed going someplace other than just into the open pore volume of the rock. To measure this phenomenon, the following experiment was performed on the WV-6 (Marcellus Shale) core sample only.

The nitrogen tank was removed from the CORAL and gas pressure in the system was bled down to 1 atmosphere. A cylinder of methane was then hooked

up, and a vacuum pump was used to draw the CORAL gas lines down to less than 3 psia. Methane was then introduced into the system at a pressure of 3 atmospheres (45 psia), which was slowly bled back down to 1 atmosphere to purge most of the remaining nitrogen. The WV-6 core was rigged for porosity using the Boyle's Law method and methane gas. Porosities were measured stepwise from a mean gas pressure of 30 psia (2 atmospheres) up to a mean pressure of 1450 psia (100 atmospheres). Net confining stress at 6000 psia was maintained on the WV-6 core throughout this series of measurements.

The results of the WV-6 porosity measurements using methane gas are detailed in Table 3. The second column from the right on this table shows the apparent porosity to gas at various pressures measured with methane using a Boyle's Law method. The gas porosity values deduced from this measurement technique are very high at low mean gas pressures, and decrease as gas pressures get higher. This behavior is typical of adsorption-like phenomena and is perhaps detailed more clearly in the plot in Figure 12. The values measured with methane are plotted as solid black circles, and the nitrogen measurements are included as open triangles. Each one of the porosity points shown in this figure represents roughly 2 days of CORAL running time. Long stability times were necessary during each measurement to permit the full amount of gas to "adsorb." In addition, small amounts of methane were continually diffusing into the Viton synthetic rubber sleeve used to confine the core and an additional waiting period was needed until this "background slope" became stable. As such, the data points plotted along the dashed line in Figure 12 took nearly 3 weeks to measure, and although we would have liked to have made additional measurements of this type on some of the other shale cores, we were restricted by time constraints under the contract.

Nevertheless, the data points plotted in Figure 12 do provide some new insights into the potential of eastern Devonian shale as a natural gas resource. To assess the potential gas content of the Marcellus Shale core from WV-6, the apparent porosity values in Figure 12 were converted to volume of methane at standard temperature and pressure (60°F and 14.7 psia) per volume of rock per psi of gas pressure. This is plotted in Figure 13 on the vertical axis as vol/vol/psi on a log-log scale against absolute methane pressure on the horizontal axis. The diamonds on this graph represent the actual points measured on the Marcellus Shale, while the solid line represents the calculated function $\text{vol/vol/psi} = (0.224)P^{1/2}$, where P is absolute methane

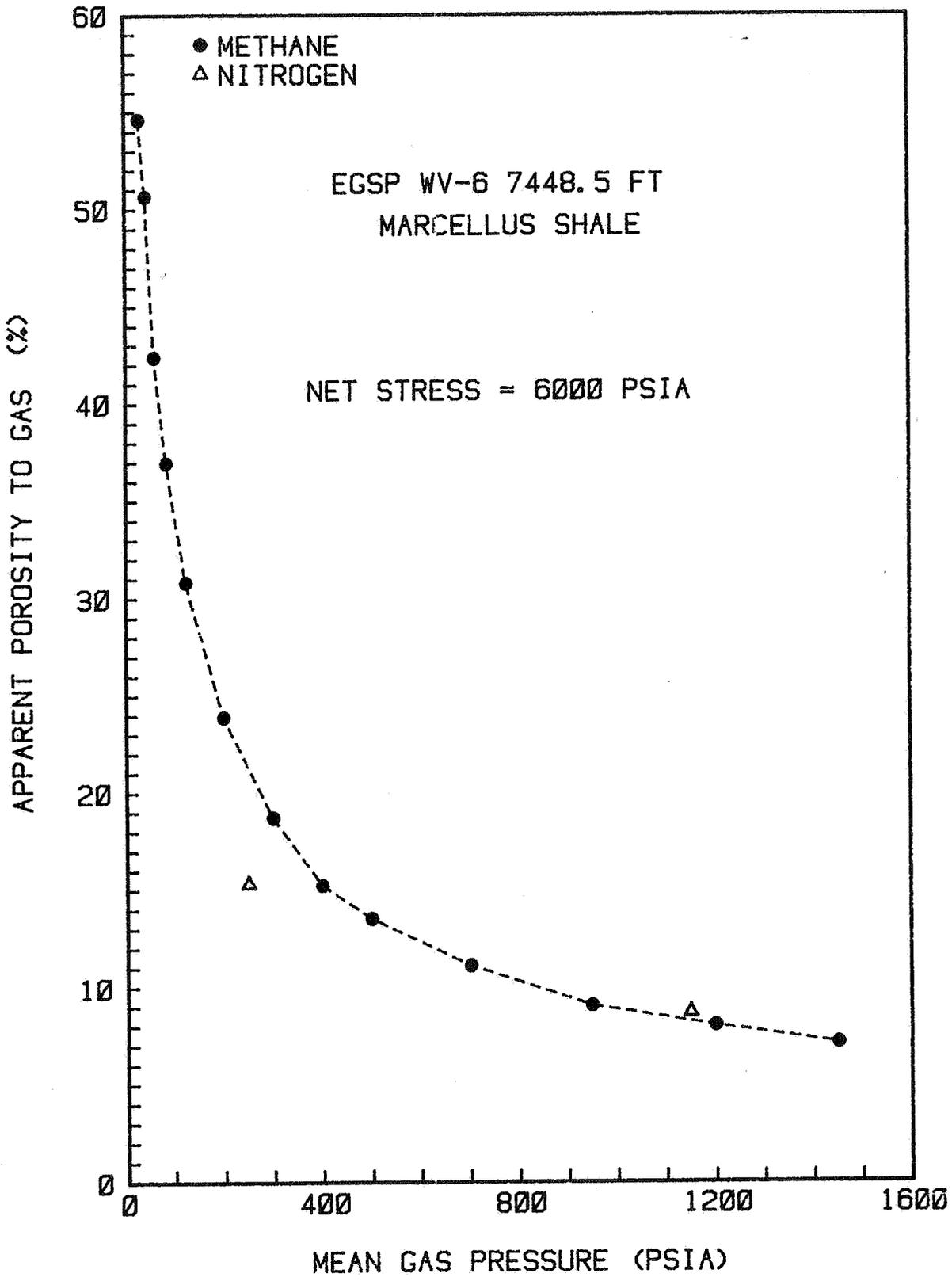


Figure 12. APPARENT POROSITY OF MARCELLUS SHALE MEASURED AT DIFFERENT GAS PRESSURES

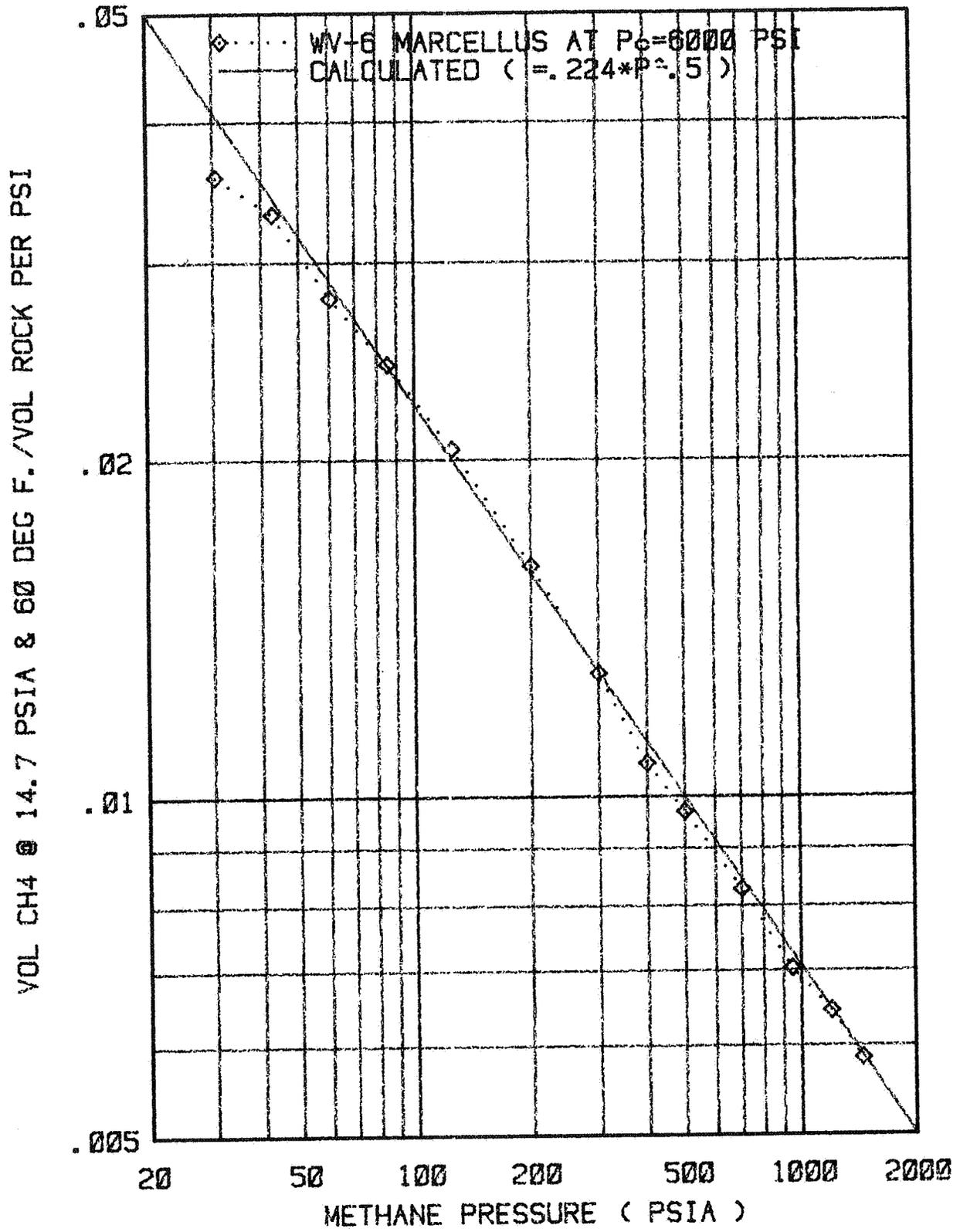


Figure 13. VOLUME OF METHANE PER VOLUME OF ROCK PER PSI PLOTTED AS A FUNCTION OF PRESSURE FOR THE MARCELLUS SHALE CORE

pressure. As is obvious from the figure, the measured data points fit this square root function quite closely. It should be stressed that this is not an adsorption curve, but rather some unknown mechanism responsible for entraining large amounts of gas in an adsorption-like manner. Adsorption of gas is described in coal studies as a fractional function of pressure, not a square root function. Integrating the function presented in Figure 13 over the pressure points measured gives the gas content of the rock in volume/volume versus pressure. This is plotted in Figure 14 and listed for each porosity measurement in the right-hand column of Table 3.

Table 3. POROSITY OF MARCELLUS SHALE
(Measured by Boyle's Law Method Under 6000 psia Net Confining Stress Using Methane Gas at 90°F)

| Nominal Press. Step (psia) | Mean Gas Press. (psia) | (atm) | Boyle's Law Gas Porosity (%) | Gas Content* (SCF/ft ³) |
|-------------------------------|---------------------------|-------|---------------------------------|--|
| 25 - 35 | 30 | 2.0 | 54.62 | 2.45 vol/vol |
| 35 - 50 | 43 | 2.9 | 50.67 | 2.94 |
| 50 - 70 | 60 | 4.0 | 42.40 | 3.47 |
| 70 - 100 | 85 | 5.8 | 36.95 | 4.13 |
| 100 - 150 | 125 | 8.5 | 30.83 | 5.01 |
| 150 - 250 | 200 | 13.6 | 23.89 | 6.34 |
| 250 - 350 | 300 | 20.4 | 18.73 | 7.76 |
| 350 - 450 | 400 | 27.2 | 15.25 | 8.96 |
| 450 - 550 | 500 | 34.0 | 13.54 | 10.02 |
| 650 - 750 | 700 | 47.6 | 11.11 | 11.85 |
| 900 - 1000 | 950 | 64.6 | 9.05 | 13.81 |
| 1150 - 1250 | 1200 | 81.6 | 8.02 | 15.52 |
| 1400 - 1500 | 1450 | 98.6 | 7.13 | 17.06 |

*Gas content = volume of gas at standard temperature and pressure per volume of rock given for each value of mean gas pressure: Calculated from $\text{vol/vol} = 0.448 \times P^{1/2}$.

The integrated function used to calculate these values is $\text{vol/vol} = 0.448 \times P^{1/2}$ where P is the mean absolute gas pressure. The gas content values in Table 3 get quite impressive at pressures above 1000 psia. According to Albert Yost, Eastern Gas Shales Program Manager at METC, the measured initial reservoir pressure of the Marcellus Shale in WV-6 was 3500 psia, which is close to the hydrostatic pressure gradient for the interval tested. Unfortunately, the Marcellus Formation is no longer accessible in this well due to caving problems and the plugback/recompletion treatment needed to solve them. Using the initial reservoir pressure given in 1984 by Yost as the value P in the above equation results in a potential in-situ gas content of the

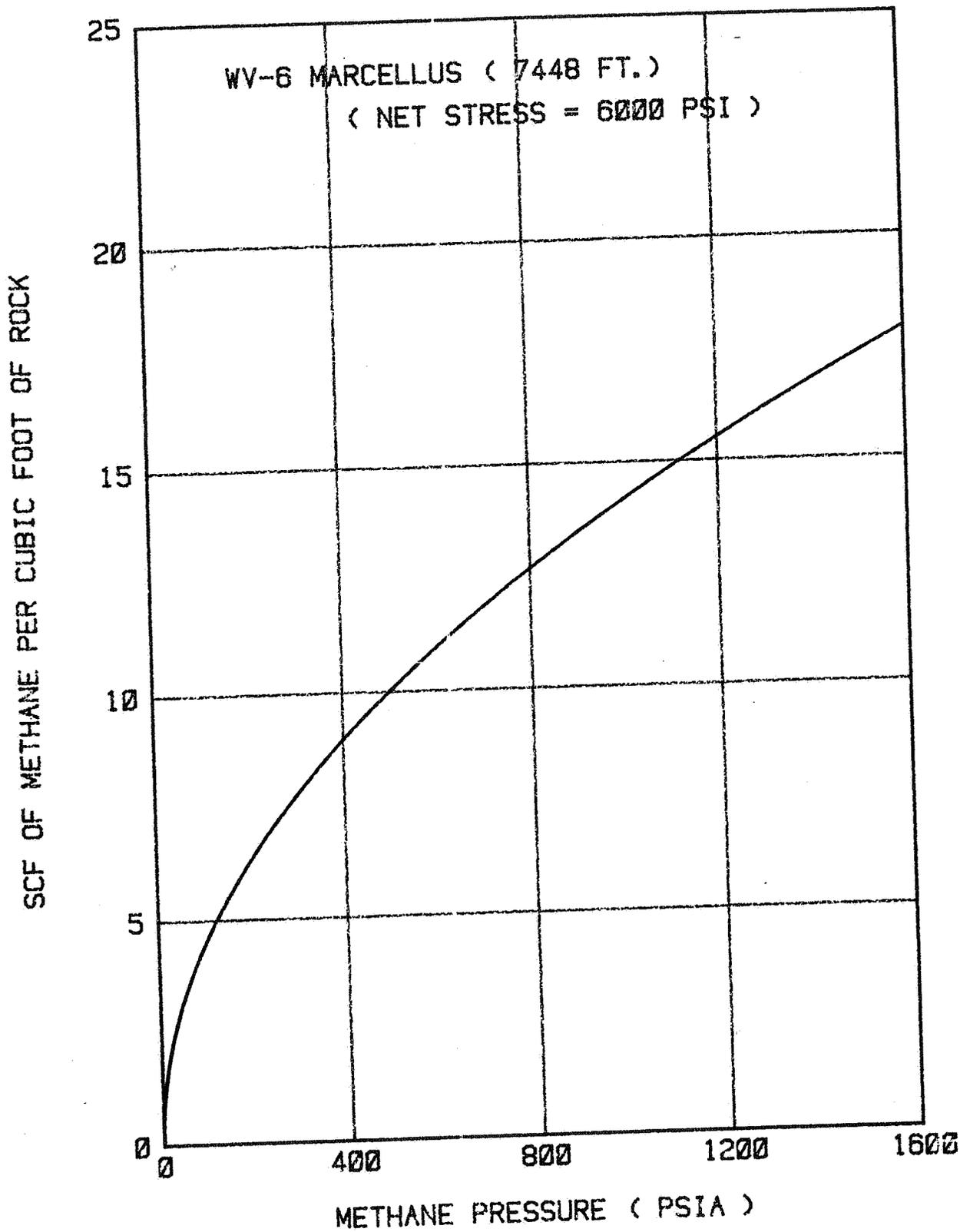


Figure 14. GAS POTENTIAL OF THE MARCELLUS SHALE AS A FUNCTION OF RESERVOIR PRESSURE

Marcellus Shale of 26.5 SCF of gas per cubic foot of rock. This is a large amount of gas for a Devonian shale, over 10 times the average value assumed by the National Petroleum Council in their 1980 shale gas resource base estimate. In addition to the large potential gas content, the WV-6 core also exhibited a fairly high permeability to gas. Although the gas permeability shows a strong stress dependence (Figure 15), it is still better than 5 microdarcies at full reservoir drawdown. For a tight, unconventional gas reservoir, this is exceptional and far more optimistic than previously thought.

3.2 Permeability of Devonian Shale

Permeability to gas in tight rocks is commonly reported as the value K_{∞} from the Klinkenberg (1941) equation:

$$K = K_{\infty} (1 + B/P) \quad (1)$$

where K is permeability to gas at mean pressure P , K_{∞} is the permeability to an ideal gas at infinite pressure, and B is a constant derived from the slope of the Klinkenberg plot. A Klinkenberg plot (as shown in Figure 15) is a graph of gas permeability as a function of the reciprocal of gas pressure. In these plots, low pressure points are to the right and infinite pressure is at the y-axis. Thus, K_{∞} is the y-axis intercept of the sloping line.

The reason that permeability data from tight rocks are plotted in this manner is due to the "Klinkenberg effect," which is also known as "gas slippage." This effect is visible as an increase in permeability at reduced pore pressures, when empirically one would expect the permeability to decrease. The current idea used to explain this effect is that in rocks with very small pores, the mean free path of a gas molecule is equal to or greater than the size of a pore throat opening. Interactions between the gas molecules and the pore walls actually help move the gas molecules forward in the direction of flow. At lower gas pressures, the molecules collide less with one another and the effect is enhanced. This effect is insignificant in conventional reservoir rocks with large pores, but it is real and measurable in low permeability porous media and must be accounted for when handling the data. (Klinkenberg plots for all shale cores measured by IGT are located in Appendix A of this report.)

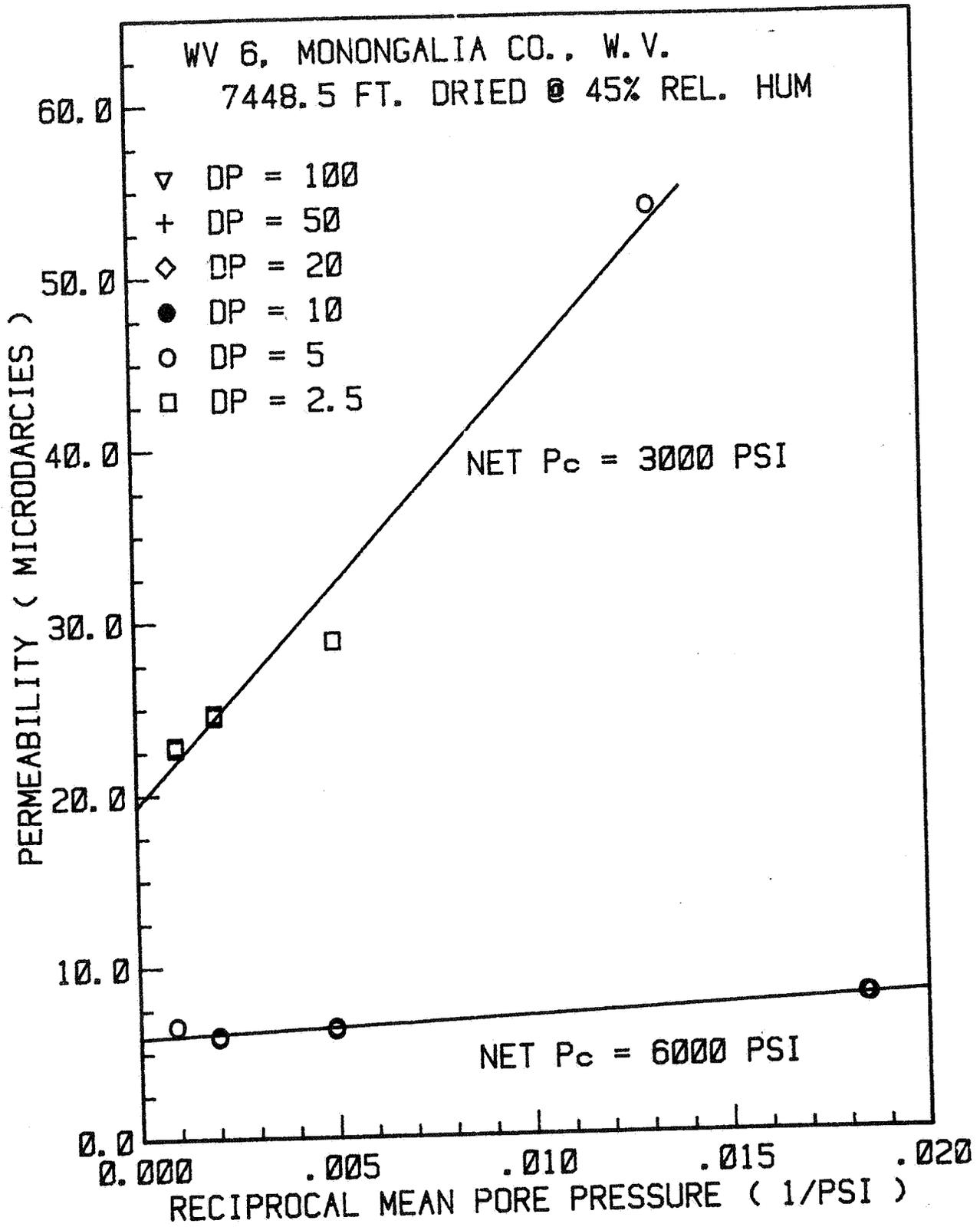


Figure 15. KLINKENBERG PLOT OF PERMEABILITY TO GAS VERSUS PORE PRESSURE FOR WV-6 MARCELLUS CORE AT TWO NET STRESS VALUES

To measure and work with Klinkenberg permeability data from tight rocks, one very important assumption must be made: All of the observed permeability effects are a function of gas pressure and pore geometry only. Klinkenberg plots are of almost no use in situations where a mobile liquid phase is present in pores along with the gas phase. Mobile liquids are affected by capillary forces and cause pore throat blockage, exerting a much stronger influence on gas permeability as a function of pressure than does the Klinkenberg effect. The unusual behavior of the lower Huron core samples, observed when trying to measure Klinkenberg permeability, led to the suspicion that a mobile liquid was present in the pores of these shales as described below.

To flow measurable amounts of gas through the lower Huron cores, a variety of moderate to high pressure-differentials were employed. Although permeability is normally independent of differential pressure, many of the lower Huron cores exhibited a change in permeability with a change in ΔP . An example of this odd behavior is shown for the lower Huron Shale core from EGSP OH-8 in Figures 16 to 18. These graphs show the results of a measurement on this core in which permeability data were taken continuously as differential pressure across the core was gradually reduced from 87 psid to less than 10 psid.

Figure 16 shows the permeability of the OH-8 core during the first 6 hours of the experiment. Differential pressure during this time dropped from an initial value of 87 psid down to about 50 psid. Permeability began at essentially zero, but as mobile liquid was drained from the pores by the gas pressure, permeability gradually increased under the continued high ΔP , leveling off at about 0.1 microdarcy. The permeability increase was undoubtedly due to liquids being displaced from the pores by the high gas pressures. This "drainage stage" of liquid movement out of the core occurs only when gas pressure exceeds the capillary pressures of the liquid and continues to the point of irreducible fluid saturation where there is no longer a continuous mobile liquid phase in the pores. At this point, higher and higher pressure drops will not displace any more liquid, and permeability is independent of differential pressure. This appears to be the reason why the curve in Figure 16 levels off at about 0.1 microdarcy.

Figure 17 shows the permeability of the OH-8 core as the differential pressure continued to fall off. Zero time on the left is at 50 psid and marks

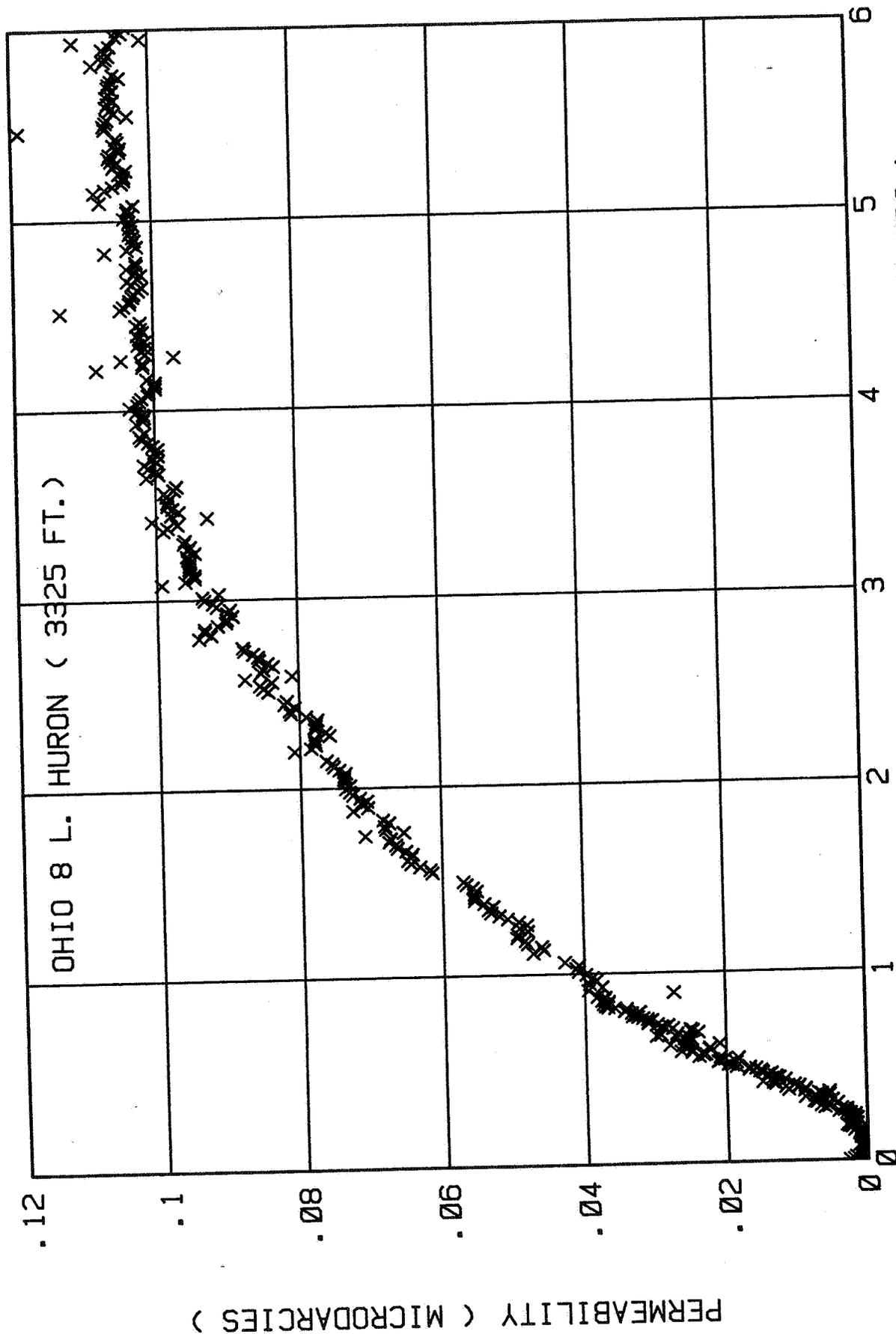


Figure 16. PERMEABILITY TO GAS OVER TIME DURING A GRADUAL DECREASE IN DIFFERENTIAL PRESSURE, DRAINAGE STAGE, EGSP OH-8, LOWER HURON SHALE

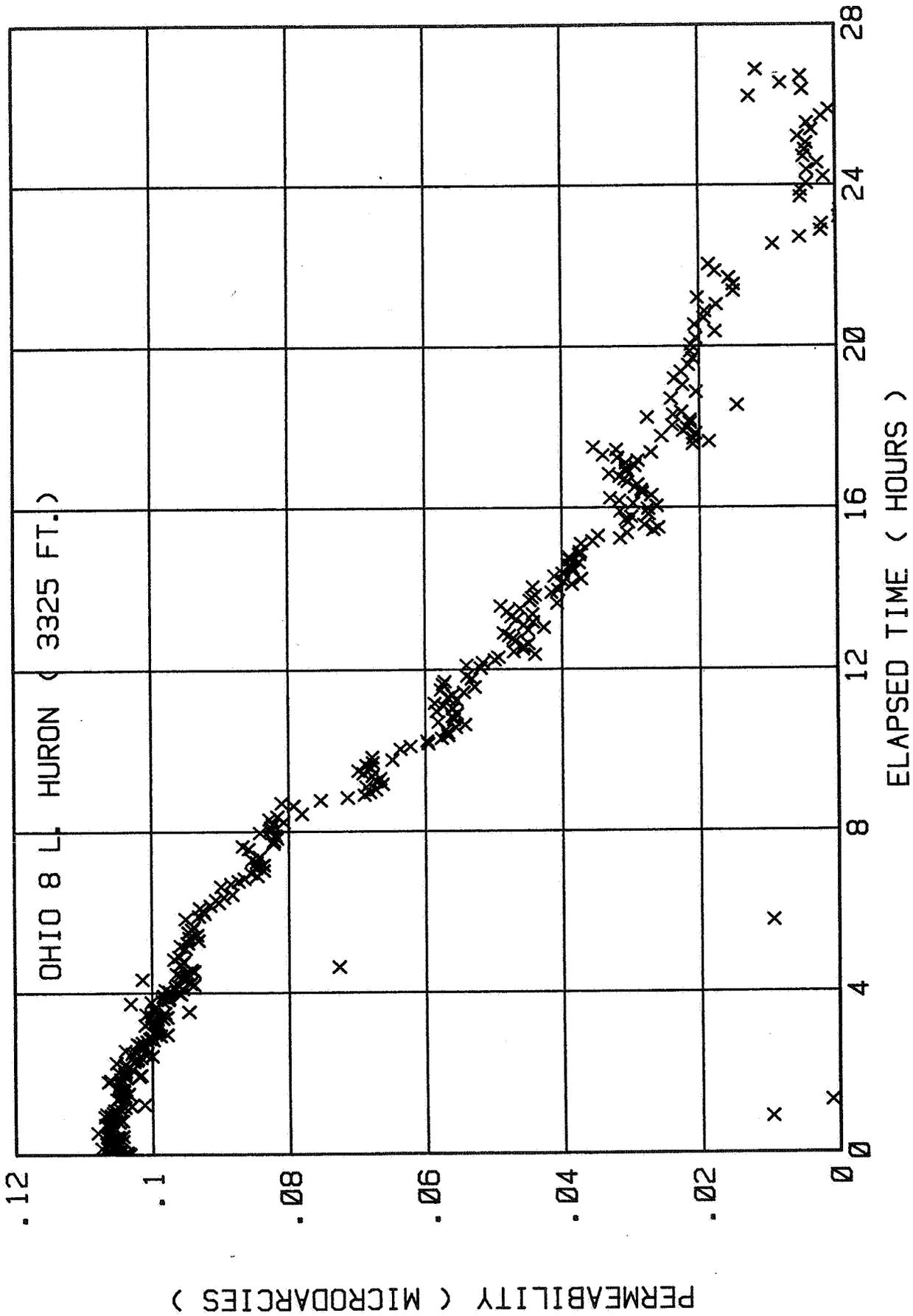
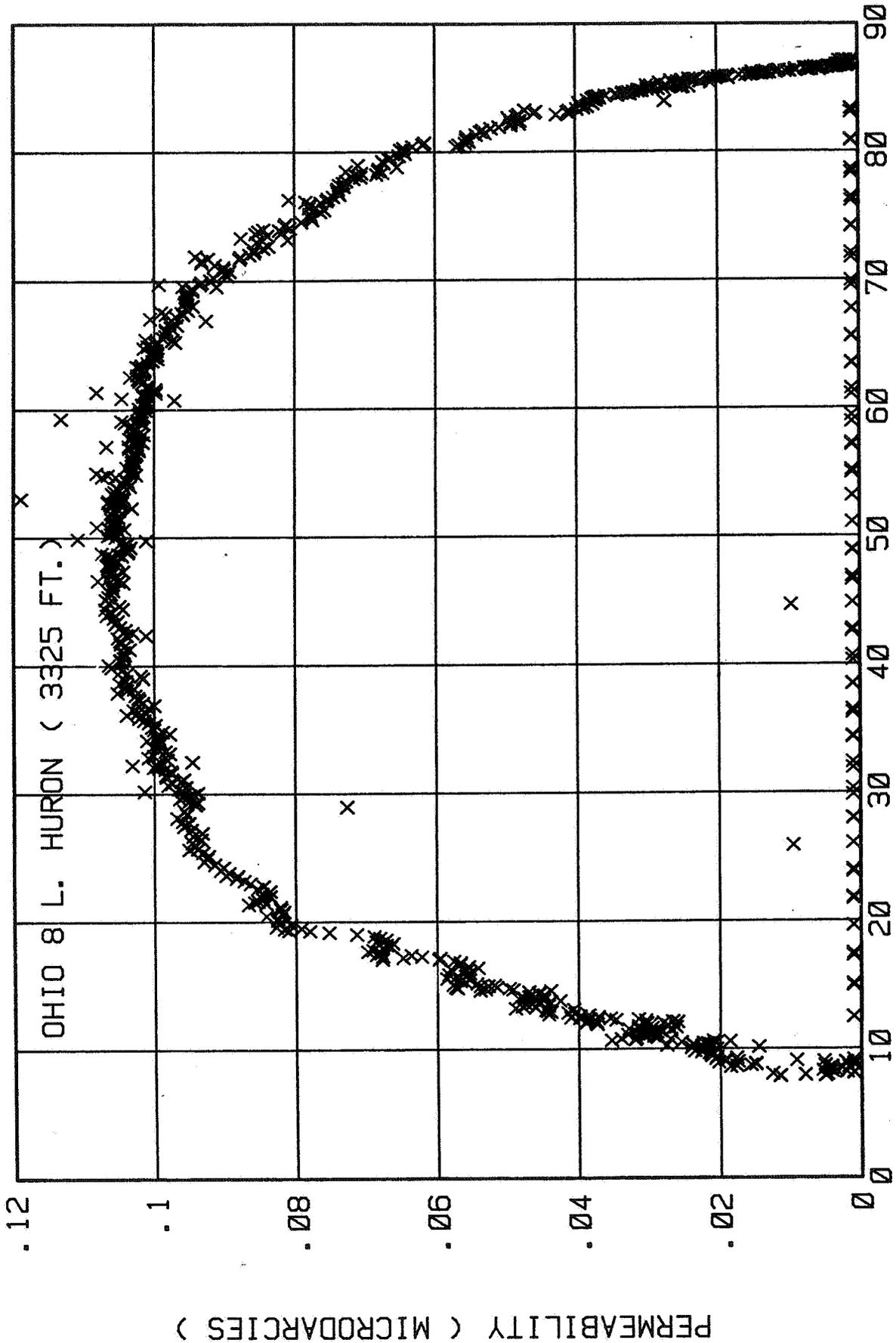


Figure 17. PERMEABILITY TO GAS OVER TIME DURING A GRADUAL DECREASE IN DIFFERENTIAL PRESSURE, IMBIBITION STAGE, EGSP OH-8, LOWER HURON SHALE

the continuation from the right-hand edge of Figure 16. Permeability began at about 0.1 microdarcy, but then gradually dropped to zero. It appears that the previously displaced liquid was imbibed back into the pores and began blocking gas flow paths in response to the continued lowering of differential pressure. This "imbibition stage" of liquid movement into the core occurs when gas pressure is less than the capillary pressure and cannot prevent the capillary forces from drawing liquid into the pores.

Figure 18 is another graph of the same data. In this case, permeability is plotted against differential pressure. Permeability ranges from 0 to 0.12 microdarcsies on the vertical axis, while differential pressure ranges from 0 to 90 psid on the horizontal axis. A net confining stress of 3000 psia was maintained on the rock throughout this experiment. Mean gas pressure in the rock pores was 1100 psia. The experiment began at 8:30 a.m. on September 13, 1984, as the differential pressure across the plug was gradually reduced from 87 to 8 psid over a period of 33 hours. The drainage phase of permeability is represented in Figure 18 by the right leg of the arch-like curve, and the imbibition phase is represented by the left leg of the arch.

Although only the OH-8 core was used for the example of odd permeability behavior described above, all of the lower Huron Shale cores analyzed in the CORAL showed this liqluid drainage-imbibition effect on gas permeability to a greater or lesser degree (see Appendix B). The only phenomenon that adequately explains this odd behavior is the presence of a mobile liquid in the pores. If the rock samples had not been dried in a relative humidity oven prior to analysis, we would surely have expected the mobile liquid to be water. However, the shale cores were dried to constant weight at 60°C under 45% relative humidity. This removes all free pore water while retaining proper clay hydration. In several thousand permeability measurements made on tight sandstone cores dried to these conditions, we have never obtained any data indicating the presence of mobile water in the pores. In addition, the sparse drilling and production records available for Devonian shale gas wells do not mention the occurrence of water flowing into the wells during production. The onset of water production in low permeability Rocky Mountain tight sand wells is a sign that gas production from the well will soon cease. Records of Devonian shale wells of similar permeability "watering out" are, on the other hand, extremely rare, and indicate that these shales do not



OHIO 8 L. HURON (3325 FT.)
 PRES. DROP ACROSS PLUG (PSI) AT 3000 PSI NET CONF. PRES.

Figure 18. PERMEABILITY TO GAS AS A FUNCTION OF DIFFERENTIAL PRESSURE,
 EGSP OH-8, LOWER HURON SHALE

PERMEABILITY (MICRODARCIES)

contain enough free water to form a mobile liquid phase. Despite this, our lab results show clearly that a mobile liquid in the pores is interfering with gas permeability in the Huron Shale.

Based on this evidence, we hypothesized that the mobile liquid in the lower Huron Shale cores was not water, but petroleum. In addition to the above observations suggesting a lack of water, there were several other pieces of evidence to suggest the presence of oil:

- Many of the lower Huron cores, particularly the OH-8 and OH-9 samples, gave off a kerosene-like sweet hydrocarbon aroma on freshly broken surfaces. The scent was quite ephemeral and disappeared in a matter of minutes. Cores over 5 years old that had not been sealed in air-tight containers still contained the oily aroma on fresh breaks.
- Although reports of water flowing into Devonian shale wells are quite rare, reports of oil are not. Oil shows are common in Devonian shale core, and commercial levels of oil production have been reported from Devonian shale wells in West Virginia (Northeast Oil Reporter, February 1984).

To determine whether or not oil was actually present in the lower Huron Shale cores, several tests were performed by IGT Analytical Chemistry Laboratory. A ground-up sample of Huron Shale from EGSP OH-6/5, known to contain a mobile liquid phase, was compared against a similar sample of Marcellus Shale from EGSP WV-6. The Marcellus core showed classical Klinkenberg permeability behavior in the CORAL and was considered to be free of any significant liquid phase. Both ground samples were washed in methylene chloride solvent that contained a "tag" of 140 parts per million $C_{24}H_{50}$. Chromatograms were then made of the solvent samples; the results for the OH-6/5 sample are shown in Figure 19. The liquid extracted from the lower Huron Shale consisted of light, paraffinic petroleum, similar in composition to most Pennsylvania-grade Appalachian Basin crudes. The Marcellus Shale chromatogram contained no peaks other than the solvent and tag.

As an additional check that this liquid was truly mobile, the downstream end caps and gas lines on the CORAL coreholders were flushed with 10 cc's of tagged methylene chloride at the end of the second Devonian shale run. Chromatography of the flushed solvent revealed the presence of small but measurable amounts of light paraffinic crude oil in the end caps and gas lines downstream of the lower Huron plugs (OH-6/4, OH-6/5, and OH-8). No oil was measured in the solvent flushed through the WV-6 (Marcellus) coreholder lines.

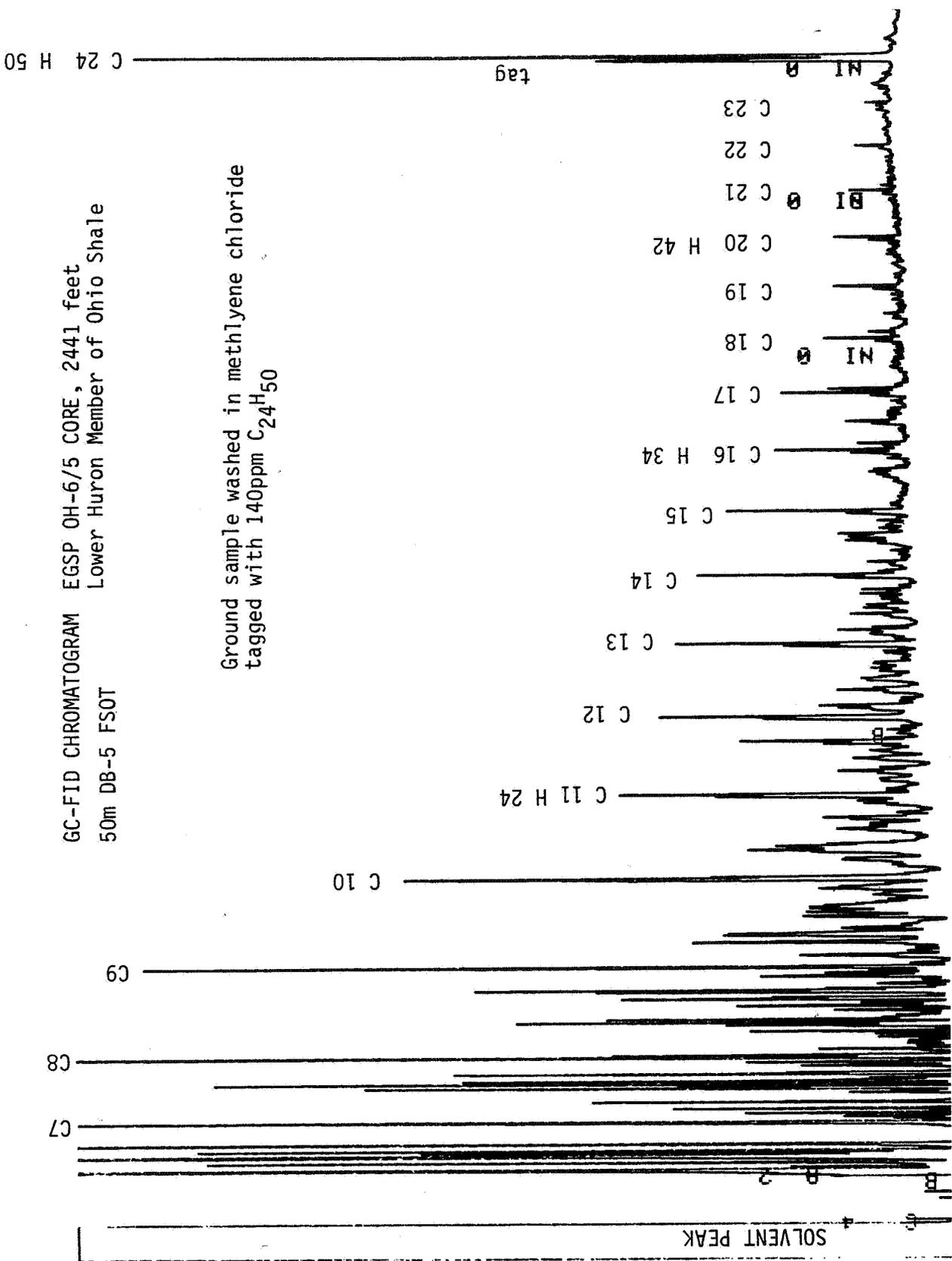


Figure 19. CHROMATOGRAM SHOWING COMPOSITION OF MOBILE LIQUID PHASE IN A SAMPLE OF LOWER HURON SHALE FROM THE EGSP OH-6/5 CORE

Because of the mobile oil present in the pores of the lower Huron Shale samples, matrix permeability to gas is extremely low in most cases. Klinkenberg plots are not useful for these cores because the lines are a very poor fit to the points due to the mobile liquid, and many times have negative slopes (see Appendix A). The presence of oil in the pores also explains the low porosity to gas measured in the lower Huron, and this discovery suggests that the formation may well prove to be more useful as an oil shale than as a marginal gas shale.

3.3 Pore Structure of Devonian Shale

The results obtained from the shale core analysis performed under this contract provide some new insights into the pore geometry and microscopic structure of eastern Devonian gas shales. Shale is made up of two primary components: very tiny (<0.005 mm) quartz grains, more or less spherical in shape, mixed in with and layered between discontinuous sheets of thin clay flakes. The pore structure of sandstone may be represented by a jar full of glass beads, but the pore geometry of Devonian shale is more analogous to a pile of newspapers interleaved with ball bearings.

Eastern Devonian gas shales were laid down as muddy sediments on the floors of inland seas over 350 million years ago and fall into two broad categories based on color. Black shales were deposited in anoxic bottom water containing some anerobic bacteria and little else. As a consequence, most of the organic matter present at deposition was retained, giving these rocks their characteristic brownish-black color. Gray shales, on the other hand, were deposited in oxygen-rich bottom waters containing numerous organisms that lived on or in the sediments and fed upon the organic matter in the mud. Differences between the depositional environments of gray and black shales not only affected the types and numbers of organisms living on and in the sediment, but also had consequences with respect to pore geometry and microscopic structure of the resulting lithified shale. As the clay settled out of the water as sediment, the individual flakes tended to orient themselves in a horizontal plane, coming to rest flat side down. In bottom waters that were anoxic, the muddy sediments did not contain any burrowing or browsing organisms, and the horizontal orientation of the clay flakes remained undisturbed. The presence of oriented clays in organic-rich, black shales has been reported by O'Brien and Demaris (1982) and is easily photographed at

relatively low magnification with a scanning electron microscope (Figure 20). Organic-lean gray shales, on the other hand, contained numerous creatures of various sizes living on and in the soft mud. The daily activities of these creatures churned up and disturbed the soft muddy sediments, causing the clay flakes to pile together in a random orientation which is also visible under a scanning electron microscope, as shown in Figure 21.

Some specific details about the pore structure in the Devonian shales tested by IGT were derived from the core analysis data. The Klinkenberg "B," which is a constant derived from the slope of the Klinkenberg plot of measured permeabilities (see Section 3.2) can be used to calculate the characteristic dimensions of flow paths in the core samples (Randolph, Soeder, and Chowdiah, 1984).

In Figure 20, it is apparent that the pore structure of Devonian black shale consists of narrow slot-like openings between clay flakes, as opposed to the rounded, triangular pores formed by spherical sand grains. Assuming that an individual shale "slot pore" is shaped like a uniform, rectangular tunnel, the following dimensions can be identified: length is the slot dimension parallel to flow, height is the long dimension perpendicular to flow, and width is the short dimension perpendicular to flow. The shortest dimension, width, is likely to have the strongest control on gas flow, and a typical or characteristic value for shale slot pore width can be calculated from the Klinkenberg "B" using the equations below.

The analysis starts with the assumption that mass flow through a slot pore of uniform width can be described by adding laminar flow to an empirical constant and multiplying by molecular flow with the mean free path of a gas molecule greater than the size of the opening. For a single slot of unit height, this yields:

$$G = \frac{w^3 M \bar{P}}{12 \mu L R T} \Delta P + C \frac{4}{3} \left(\frac{2 M}{\pi R T} \right)^{1/2} \frac{w^2}{L} \Delta P \quad (2)$$

Assuming that the entire pore volume is in slot pores, the sum of the heights of the slots is:

$$h = \frac{\phi A}{w \tau} \quad (3)$$

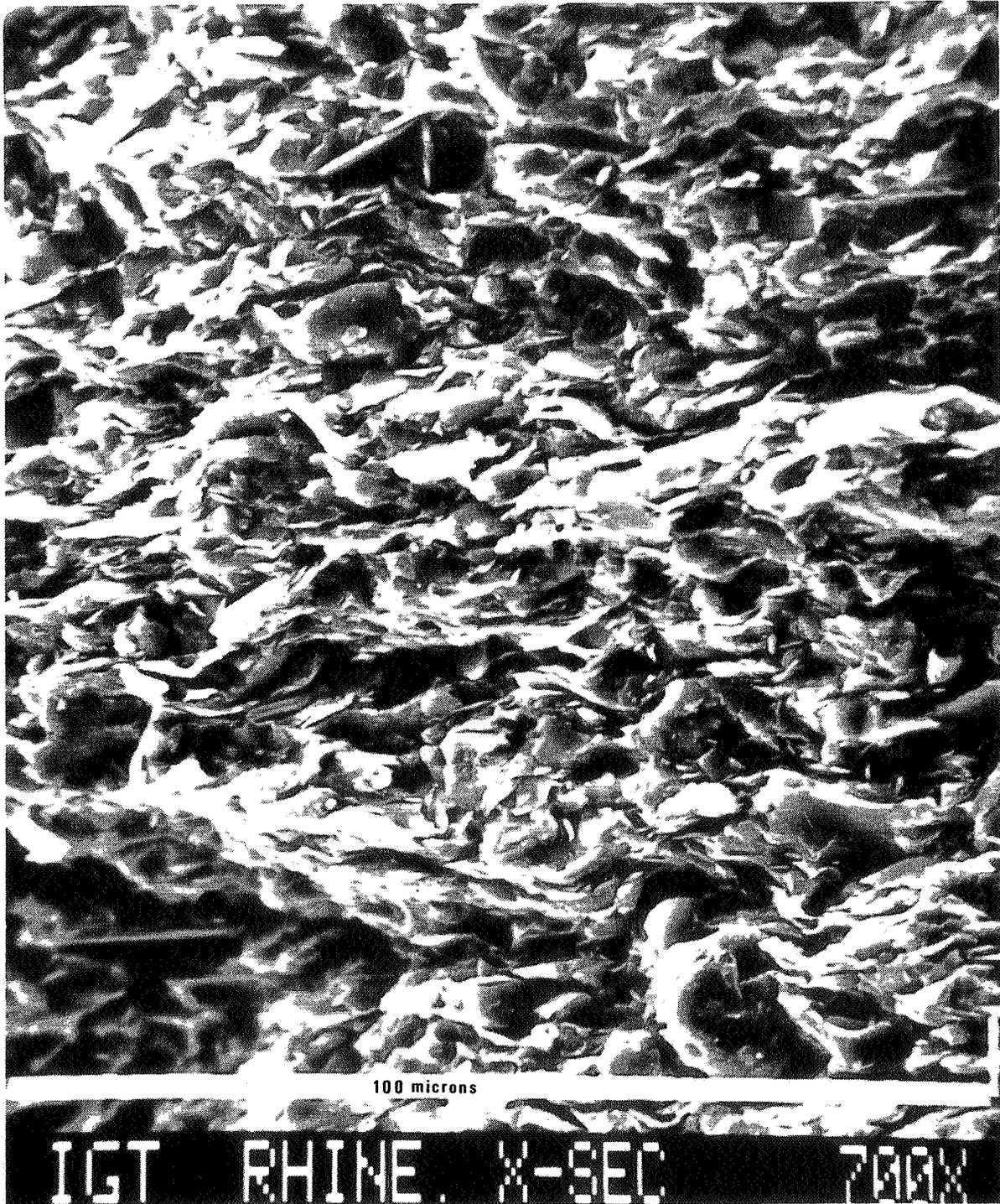


Figure 20. SCANNING ELECTRON MICROGRAPH OF ORGANIC-RICH RHINESTREET SHALE SHOWING STRONG CLAY FLAKE ORIENTATION PARALLEL TO BEDDING



Figure 21. SCANNING ELECTRON MICROGRAPH OF ORGANIC-LEAN CHAGRIN SHALE, SHOWING POOR CLAY FLAKE ORIENTATION

Combining equations 2 and 3 plus the ideal gas law and Darcy's law, and solving for permeability:

$$k = \frac{\phi \bar{w}^2}{12 \tau^2} \left[1 + C \frac{16 \mu}{\bar{w} \bar{P}} \left(\frac{2 R T}{\pi M} \right)^{1/2} \right] \quad (4)$$

Equation 4, although somewhat more complicated, has the same form as the Klinkenberg equation for permeability, given earlier as Equation 1. This similarity can be used to relate slot width to the empirical constant "B" in the Klinkenberg equation, and the flow path tortuosity can be calculated from the Klinkenberg permeability and slot dimensions:

$$\text{Slot width} = \bar{w} = \frac{16 c \mu}{B} \left(\frac{2 R T}{\pi M} \right)^{1/2} \quad (5)$$

$$\text{Tortuosity} = \tau = \bar{w} \left(\frac{\phi}{12 k_{\infty}} \right)^{1/2} \quad (6)$$

Nomenclature for Equations 1 through 6

- A = cross sectional area of cylindrical rock sample (in square meters)
- B = empirical constant in Klinkenberg equation
- C = Adzumi Constant (assumed to be 0.9)
- G = mass flow rate (in kg/sec)
- h = total height of all slot pores
- K = permeability to gas at mean pressure \bar{P}
- K_{∞} = permeability to an ideal gas at infinite pressure
- L = length of sample (meters)
- M = molecular weight of the gas (in kg/kmole)
- μ = viscosity of the gas
- \bar{P} = mean gas pressure (Pa)
- ΔP = pressure drop (Pa)
- ϕ = porosity (fraction)
- π = 3.1416
- R = gas constant = 8314 Joule/(K mole x T)
- T = temperature (Kelvin)
- τ = tortuosity (in multiples of plug length)
- \bar{w} = mean slot width (meters)

Note that the only measured parameter in the calculation of slot width using Equation 5 is the value of the Klinkenberg "B." Although porosity was

considered in the derivation, neither porosity nor permeability enter into the calculation of slot width. Thus, the calculated flow path opening is independent of the portion of total porosity that is contained in the slot flow path. On the other hand, the flow path tortuosity calculated in Equation 6 is dependent upon the porosity value measured on the core. Tortuosity is defined as the distance a gas molecule travels through the core plug expressed in multiples of plug length. Higher tortuosity values generally indicate poorer pore-to-pore interconnections. Although the gas porosity measured on the Huron Shale samples was too low to provide accurate data for tortuosity calculations, the porosity and permeability values measured on the Marcellus Shale were adequate enough to provide the results discussed below.

Gas flow path dimensions were calculated on the WV-6 Marcellus Shale core, because only this sample out of all the cores analyzed had good quality Klinkenberg data. The characteristic width of gas flow paths in the Marcellus core is 0.05 microns at a net stress of 3000 psi and 0.35 microns at a net stress of 6000 psi. By contrast, flow paths in tight gas sand at stress representative of 50% drawdown are typically 0.1 micron in width. Although it at first appears surprising that the flow path width in the shale increases rather than decreases as net stress goes up, this behavior suggests that a bimodal pore size distribution is present in this rock with the smaller flow paths being squeezed off at the higher net stress. Further evidence supporting this conclusion is gained from the flow path tortuosity calculations: gas flowpaths at 3000 psi net stress have a tortuosity of about 4 plug lengths, while at 6000 psi net stress the tortuosity is over 50 plug lengths. This indicates the closing of many small, interconnecting pores at high net stress, resulting in fewer and less direct pore-to-pore connections. There are apparently a few large flow paths and many tiny ones that give the shale a fairly high permeability and low tortuosity at low net stress (refer back to Figure 15) and a very steep Klinkenberg slope. At higher net stress, only the few wider flow paths are available to carry gas, greatly increasing the flowpath tortuosity and reducing the permeability.

The gas permeability data on the Huron Shale cores were too strongly affected by the mobile liquid in the pores to provide useful Klinkenberg plots; however, the oil saturation of these rocks permitted the measurement of

capillary pressures and estimates of pore size distribution therefrom. As shown previously in Figure 17, as the differential pressure was dropped and oil began imbibing back into the Huron Shale pores, gas permeability was reduced in stair-step increments. This phenomenon of short-term constant permeability during an otherwise continuous permeability drop with pressure was interpreted as an indication that oil imbibition into pores of one size class was completed, and imbibition had not yet begun for the next larger size class. Thus, the capillary entry pressure and the approximate pore size distribution could be estimated by the pressures at which permeabilities remained constant. The gas permeability cutoffs for a variety of gas pressures and confining pressures are shown for the lower Huron Shale cores in Appendix B.

A plot of capillary entry pressure versus pore size was constructed, as shown in Figure 22, by making a guess about the viscosity and surface tension of the oil in the Huron Shale based on its chemical composition (insufficient quantities were collected to permit actual direct measurements of these values). By comparing gas permeability cutoff pressures shown in Appendix B with this graph, pore sizes in the Huron Shale cores can be estimated.

Although the Huron Shale differs considerably from the Marcellus Shale in terms of core analysis results and reservoir properties, the Huron appears to share the same type of bimodal pore-size distribution with the Marcellus. The gas permeability cutoffs due to oil imbibition into the Huron, as shown in Appendix B, tend to occur primarily at two distinct pressures under each set of net confining stress conditions. This is suggestive of two pronounced peaks in capillary entry pressures, which is further suggestive of a bimodal pore size distribution.

3.4 Natural Fractures in Devonian Shale

Open and mineralized fractures were commonly observed in Devonian shale core recovered and analyzed by Cliffs Minerals under the EGSP. Also, offset observation well data from the Meigs Co. Ohio Shale test revealed a strong directional response that has been interpreted to be the result of natural fractures (Alam et al., 1982). These and other observations make it appropriate to focus upon the definition of the nature of natural fractures significant to gas production in Devonian shales.

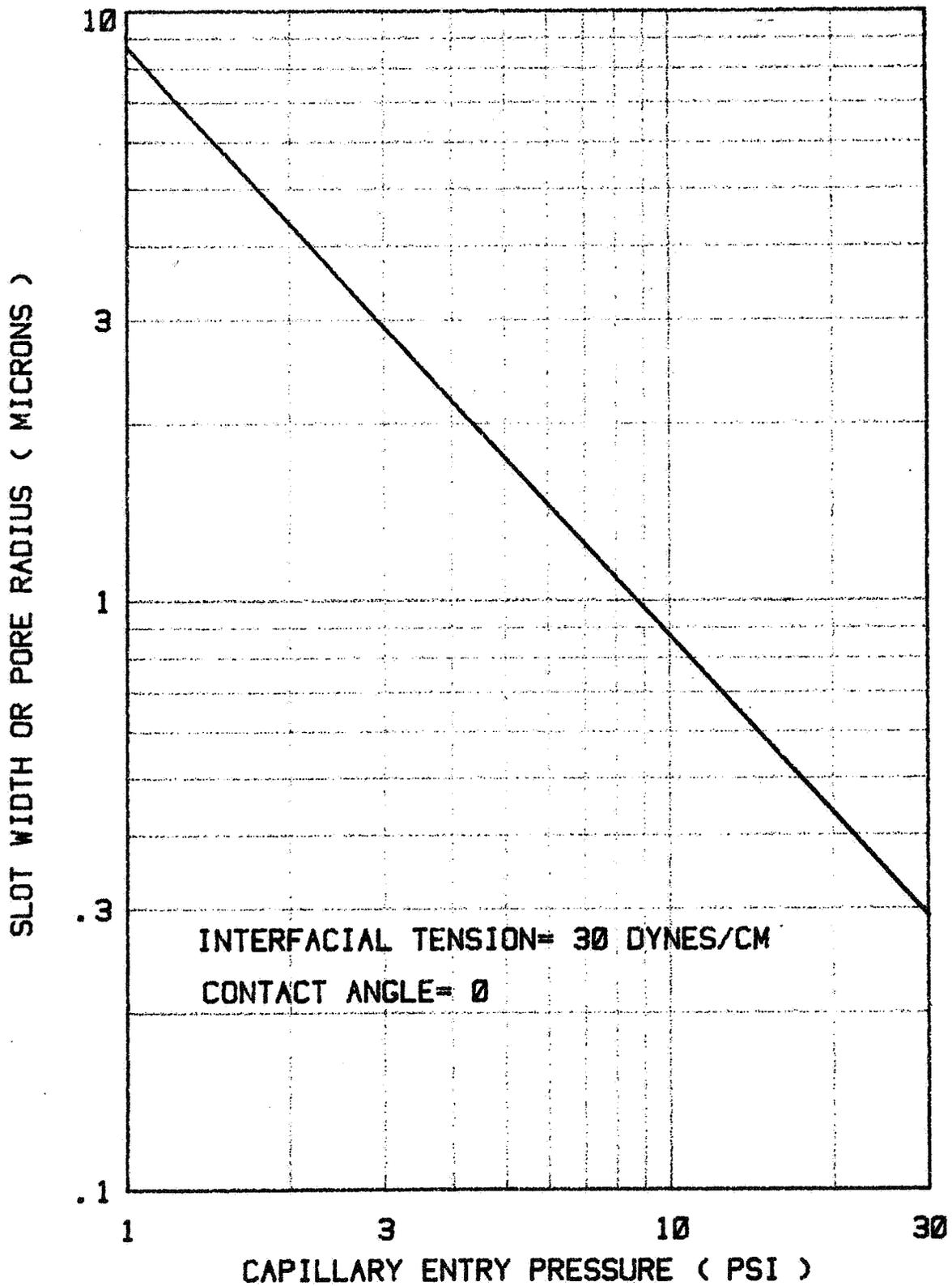


Figure 22. CAPILLARY PRESSURE AS A FUNCTION OF PORE SIZE IN OIL-FILLED DEVONIAN SHALES

For the idealized case of vertical cracks with uniform width and spacing, the equivalent matrix permeability in the direction of the cracks is given by—

$$k = \frac{10^8 w^3}{12S} \quad (7)$$

where —

k = permeability (darcies)

w = crack aperture (cm)

S = crack spacing (cm)

Also, for this idealized model the crack porosity (ϕ_c) expressed as a fraction is simply the ratio of crack aperture to crack spacing. Thus, crack porosity can be related to equivalent matrix permeability and crack spacing by the equation —

$$\phi_c = \left[\frac{12k}{10^8 S^2} \right]^{1/3} \quad (8)$$

Using Equations 7 and 8 to get a feel for the character of natural fractures yields relationships between equivalent matrix permeability, fracture spacing, fracture width, and fracture porosity as shown in Table 4. Note that the units in the table differ from those given for the equations above. Specifically, crack aperture is in microns (10^{-6} meter) and porosity is in percent in Table 4.

Table 4 clearly reveals that the natural fractures required to provide an equivalent matrix permeability of the order of 100 microdarcies must have very small in-situ apertures and a very small fracture porosity. For fracture spacing in the range of 0.1 to 100 meters, apertures are in the range of only 5 to 50 microns (0.0002 to 0.002 inches). In other words, the opening in ideal, planar, equally spaced natural fractures would only be as great as the thickness of a sheet of paper if the fracture spacing was tens of meters or of the order of 100 feet.

The fracture porosity clearly must be very small compared to matrix porosity. Indeed, for the case of 100 microdarcy-equivalent matrix permeability, the fracture porosity is in the range of only 0.002 to 0.00002 percent for fracture spacings in the range of 0.1 to 100 meters. For crack

aperture as wide as the thickness of a sheet of paper, the fracture porosity would be only about 10^{-4} percent, and the spacing would have to be on the order of 100 feet.

The above discussion strongly suggests that significant gas-productive natural fractures are probably not macroscopically apparent features such as the near-vertical, calcite-filled joints observed in much of the EGSP core. Indeed, there are questions as to whether gas-producing fractures are horizontal or vertical in orientation; whether their maximum dimensions are measured in millimeters, meters, or tens of meters; or whether they have any features at all that are visible without a microscope.

Table 4. PARAMETERS FOR PARALLEL, UNIFORMLY SPACED NATURAL FRACTURES

| Equivalent Matrix Permeability (μ d) | Crack Spacing (m) | Crack Aperture (microns) | Fracture Porosity (%) |
|--|-------------------------|--------------------------------|-----------------------------|
| 1 | 0.1 | 1.06 | 0.00106 |
| | 1 | 2.29 | 0.000229 |
| | 10 | 4.93 | 0.000049 |
| | 100 | 10.6 | 0.0000106 |
| 10 | 0.1 | 2.29 | 0.00229 |
| | 1 | 4.93 | 0.00049 |
| | 10 | 10.6 | 0.000106 |
| | 100 | 22.9 | 0.0000229 |
| 100 | 0.1 | 4.93 | 0.00493 |
| | 1 | 10.6 | 0.00106 |
| | 10 | 22.9 | 0.000229 |
| | 100 | 49.3 | 0.000049 |
| 1000 | 0.1 | 10.6 | 0.0106 |
| | 1 | 22.9 | 0.00229 |
| | 10 | 49.3 | 0.00049 |
| | 100 | 106 | 0.000106 |



4.0 DISCUSSION OF RESULTS

The results presented in the previous section do not supply all the data needed to fully understand and solve the many problems associated with Devonian shale gas production. Clearly, more analyses of this type would add to the data base and permit broader generalizations to be made. Nevertheless, the laboratory measurements made on these eight cores provided enough information to draw some new conclusions about the nature of the shale gas resource, and to question some basic assumptions about Devonian shale that have been accepted as "facts" for years. The results presented in this report should be taken under consideration because these types of measurements have never before been made with this degree of accuracy on Devonian shale, and the data are now available for the first time. Some of the implications of this work are detailed below.

The resource base estimate of Devonian shale gas made by the National Petroleum Council (NPC) in 1980 used gas content values of 0.1 to 0.6 scf/cu ft. in the calculations. These values are clearly far too low for the Marcellus Shale. Referring back to Figure 14 in the previous chapter, even at a pressure of only one atmosphere, for example, 1.7 scf of methane gas would be contained in a cubic foot of Marcellus Shale, which is still more than twice the NPC estimate. If the actual reservoir pressure in the shale is near hydrostatic, as reported to us by A. Yost of METC (1984), the Marcellus is capable of holding an enormous amount of gas. Even though the Marcellus is a relatively thin unit (~104 feet thick in the MERC #1 Well, EGSP WV-6), there are similar black shales tens or hundreds of feet thick above the Marcellus stratigraphically in the central Appalachian Basin. If one accepts that shale units such as the Geneseo, Middlesex, or Rhinestreet have depositional and diagenetic histories similar to the Marcellus, the possibility certainly exists that these shales may contain far more gas than the NPC estimates. The NPC Devonian shale resource base estimate was made with the best data available in 1980, but these resource estimates should not be adhered to as if they were gospel.

Geographic and stratigraphic emphasis of Devonian black shale research should be shifted from the lower Huron Shale on the western edge of the Appalachian Basin to the more thermally mature units towards the center. As mentioned above, gas potential of shales near the center of the basin appears to be far more promising than those on the western margin. Without exception,

every lower Huron sample analyzed by IGT from the Ohio River area contained mobile petroleum liquids in enough abundance to fill pores and block gas flow paths through the rock. The highest gas porosity in the rock matrix of seven lower Huron cores was 0.18%; most were below 0.10%. The lower Huron may well be an oil shale resource in this area, but it is a very poor gas shale.

The age of the core samples does not appear to influence or bias the results of the analysis, as long as the samples chosen for analysis consist of intact, well-preserved core segments. It is our observation that shale core tends to develop fissility (parallel horizontal cracks) when exposed to air. Older cores generally showed a greater degree of deterioration than fresher cores. Cores kept sealed in cans or carefully wrapped in plastic sheeting, however, were often freshly preserved after 5 years' time. The question as to whether the age of shale core affects the results of analysis when macroscopically non-fissile segments are sampled is best demonstrated by comparison of the oldest EGSP core (WV-5) with the freshest (Moore No. 1). Both cores have about the same composition, bulk density, color, and number of plug cracks. Both have roughly similar porosities, permeabilities, and stress dependence of permeability. Comparison of the WV-5 core with other cores drilled at about the same time (WV-6 and the OH-6 samples) shows that gas permeability and the stress dependence of this value are controlled by factors other than core age and macroscopic fissility.

Our work has indicated that even though a proportion of the EGSP core has deteriorated beyond use for porosity and permeability analysis due to improper storage, intact plug samples can yet be obtained from a high enough percentage of the total EGSP core footage to yield significant results. The occurrence of pore-blocking oil in the lower Huron and the presence of the unusually high gas potential of the Marcellus Shale were discovered after analysis of only eight samples of EGSP core in the CORAL. No one knows what other important discoveries remain to be made within the vast quantity of EGSP core. The existing core, if properly analyzed, represents potential information that could have a major impact on future gas exploration and production.

The relationship of gas productivity to shale type should be investigated in more detail. The old EGSP notion that any black shale will produce commercial amounts of gas provided there are plenty of natural fractures has proven again and again to be an oversimplified view of a complex situation. Our core analyses indicate that the black lower Huron Shale won't produce

significant amounts of gas no matter how many fractures it has. The Marcellus Shale, on the other hand, may well produce commercial quantities of gas with no natural fractures at all.

While preparing a report on Devonian shale stratigraphy along the south shore of Lake Erie, gas shows in shale wells were plotted against stratigraphic units by Potter, Maynard and Pryor (1980). Their results indicate that by a large margin, most of the gas shows occur in gray shales and siltstones, not in the black units. A similar study by one of us (Matthews) under GRI funding gave results comparable to, and supportive of those found by Potter, Maynard, and Pryor.

Significant gas production from a gray shale was documented by DOE in the EGSP OH-8 Well in Noble County, located in eastern Ohio (Cliffs Minerals Report, 1980). A foam fracturing stimulation treatment of this well in 1980 resulted in an open gas flow of 510 Mcfd from the gray Chagrin Member of the Ohio Shale (Horton, 1982). This production can be seen on the OH-8 temperature log reproduced in this report in Figure 5. In the black Huron and Rhinestreet shales below the Chagrin, however, the same stimulation treatment yielded a combined open gas flow rate of only 52 Mcfd. Analysis by IGT of a core sample of lower Huron from this well indicated a gas permeability of only 0.194 μ d at 1750 psi net stress and 0.078 μ d at 3000 psi net stress. Gas-filled porosity was less than 0.1%. With these results in hand, the low gas productivity of the Huron Shale in EGSP OH-8 is understandable.

One of the strongest initial gas productions in any of the EGSP wells occurred in southeastern Ohio immediately after coring in the OH-9 well in Meigs County. Production was not from the lower Huron Shale, but from just below it in the upper Olentangy Shale (Cliffs Minerals Report, 1981). This can be seen in the Cliffs well summary chart, reproduced in this report as Figure 6. Unfortunately, coring was terminated 20 feet above the gas show, although the stratigraphy, gamma and density logs indicate that the gas occurred in a gray shale, rather than a black unit. This information, combined with IGT's discovery of mobile petroleum blocking the pores of the lower Huron immediately above, leads to the speculation that the black Huron Shale is acting as a caprock to trap gas in the underlying gray Olentangy Shale in Meigs County. The notion of gas-impermeable, oily black shales acting as stratigraphic traps for natural gas contained in intertongued gray shales provides an explanation for observed cases of shale gas production on basin

margins based upon well-defined principles of geology and petroleum engineering. If shown to be a workable concept, this idea could lead to a new and useful exploration rationale for Devonian shale gas in the eastern United States. The results of IGT's core analysis studies show unequivocally that the eastern gas shale resource is considerably more complex than originally thought in the early days of the EGSP program.

Monsanto results: The IGT shale core analyses provided no real surprises when compared to geochemical assessments of the shale sequence performed by the Mound Facility of Monsanto Research Corporation (Zielinski and McIver, 1982). Figure 23 is a map by Monsanto of gas source potential in the Marcellus Shale based on the Mound geochemical analyses. The EGSP WV-6 well is in an area delineated as having a good source potential for natural gas; combined with the strong gas containment abilities of the Marcellus as measured by IGT, wells in this area ought to produce commercial amounts of shale gas. Figure 24 shows the potential for oil generation in the lower Huron Shale as mapped by Monsanto from the Mound geochemical analyses. Looking at this map, it is not at all surprising that IGT core analysis found oil filled pores in the sample of lower Huron Shale from the EGSP OH-8 well. Based on our analyses from OH-6, OH-9 and WV-5, however, we feel that the separate contours in Ohio and West Virginia are probably connected across the Ohio River. Although some parts of the Huron Shale presumably experienced pressure and temperature conditions conducive to the generation of both gas and oil, it should be kept in mind that these are very fined-grained rocks and that any liquid in the pores, be it oil or water, will cause pore blockage and shut off gas flow. Therefore, shales with high oil-generating potential cannot be expected to flow marketable amounts of gas, despite the fact that commercial quantities of gas may have been generated with the oil and may reside also in the pores.

RELATIVE GAS SOURCE POTENTIAL

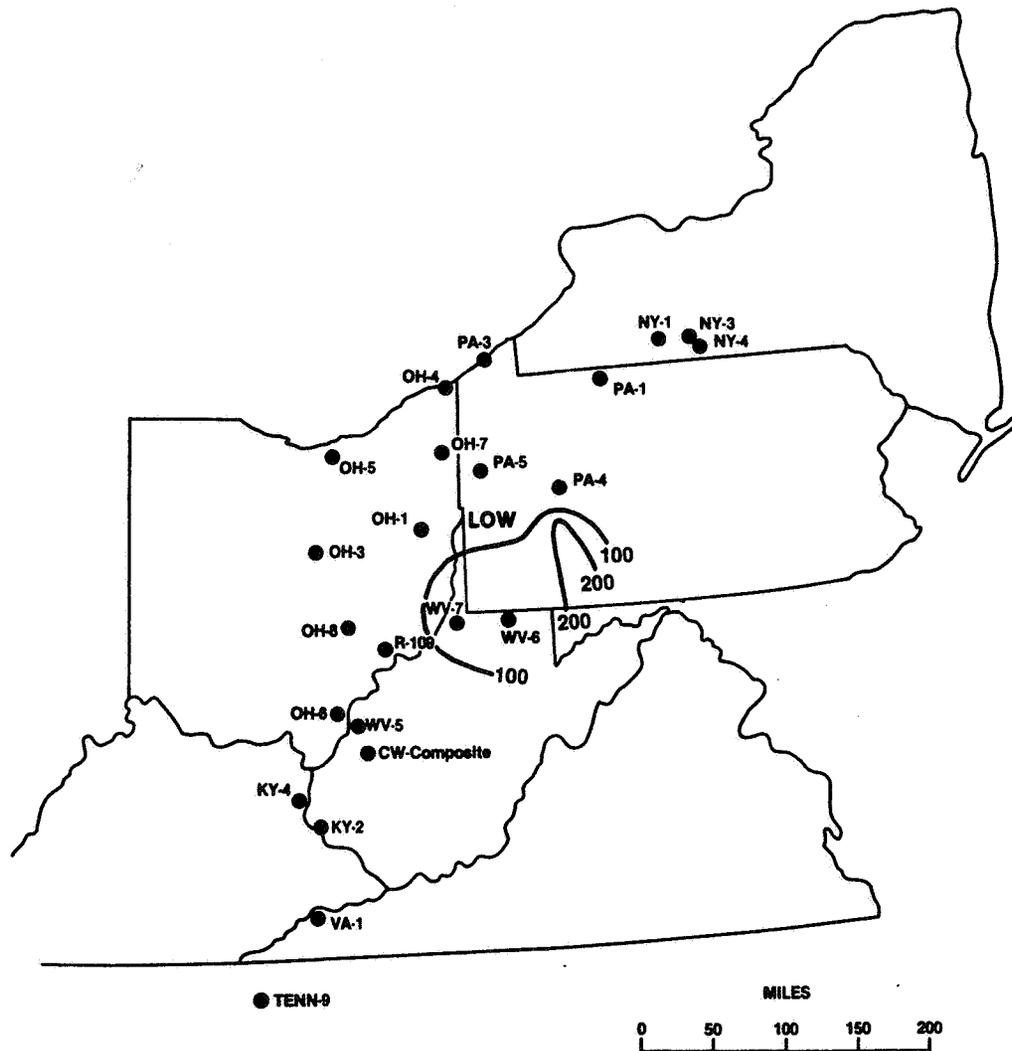


FIGURE 94

from Zielinski and McIver, 1982

LATE MARCELLUS TIME

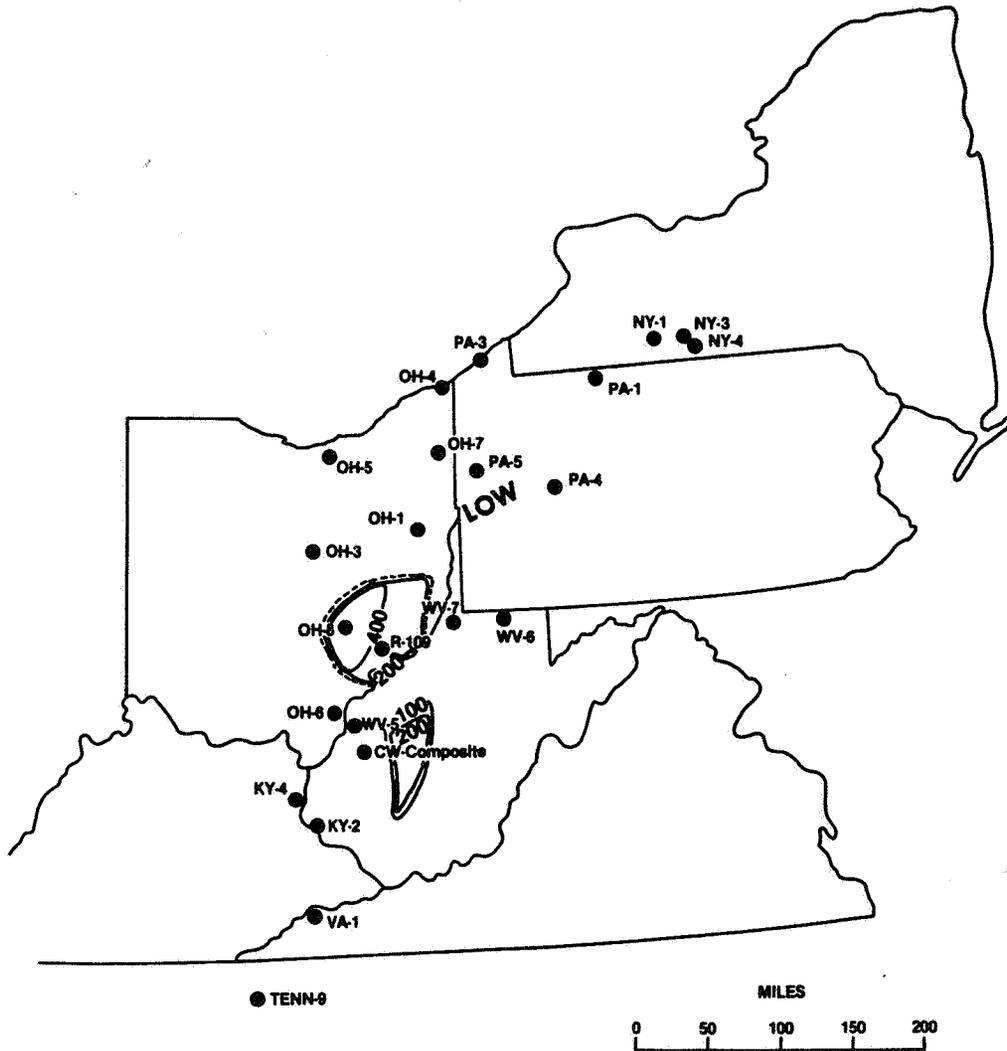
Map Unit 2



Prepared by Mound Facility

Figure 23. MARCELLUS SHALE GAS SOURCE POTENTIAL
BASED ON MONSANTO GEOCHEMICAL ANALYSIS

RELATIVE OIL SOURCE POTENTIAL



● TENN-9

MILES
0 50 100 150 200

FIGURE 108
from Zielinski and McIver, 1982

EARLY LOWER HURON/EARLY DUNKIRK TIME MAP UNIT 10



Prepared by Mound Facility

Figure 24. LOWER HURON SHALE OIL SOURCE POTENTIAL
BASED ON MONSANTO GEOCHEMICAL ANALYSES

5.0 CONCLUSIONS

Like most research projects, the work performed under this contract answered a few of the long-standing questions about Devonian shales of the eastern United States, but in the end generated even more questions. The conclusions we reached upon completion of this project are summarized below.

1. The lower Huron Member of the Ohio Shale from wells in southeastern Ohio/northwestern West Virginia contains petroleum as a mobile liquid phase in the pores. As a result, 1) matrix permeability to gas is very low (in the tens of nanodarcies range) due to capillary blockage by the oil, and 2) gas-filled porosity is less than 0.1% in nearly all cases.
2. Permeability to gas of the Marcellus Shale core at pressures bracketing reservoir net stress was surprisingly high — ranging from 5 to 50 microdarcies but very strongly stress-dependent. Doubling the net stress decreased the permeability by nearly 75%. Gas-filled porosity was also high, ranging around 10%, but with a very strong "adsorption" component.
3. Gas potential of the Marcellus Shale in the WV-6 core is very high. The discovery that entrainment of methane gas in this rock varies as a square root function of pressure appears to be responsible for the large potential gas capacity of this rock. Initial gas contents as high as 26 SCF methane per cubic foot of rock may be possible in virgin Marcellus Shale reservoirs.
4. The pore size distribution in both the Marcellus and Huron cores appears to be bimodal in character. Typical pore sizes calculated from high-quality Klinkenberg permeability data on the Marcellus are 0.05 microns at a net stress of 3000 psia and 0.35 microns at a net stress of 6000 psia. This surprising result of larger characteristic pore size at higher net stress in the Marcellus Shale is suggestive of a bimodal pore size distribution, with small pores squeezed off at higher stress. Capillary behavior of mobile liquid in the Huron cores suggests a similar pore geometry.
5. Devonian gas shale in the Appalachian Basin is an enormously more complex resource than previously thought. The old EGSP idea that any highly fractured black shale will produce commercial quantities of natural gas is overly generalized in two directions: All black shales will not produce gas, and all shale gas is not produced from black shales.



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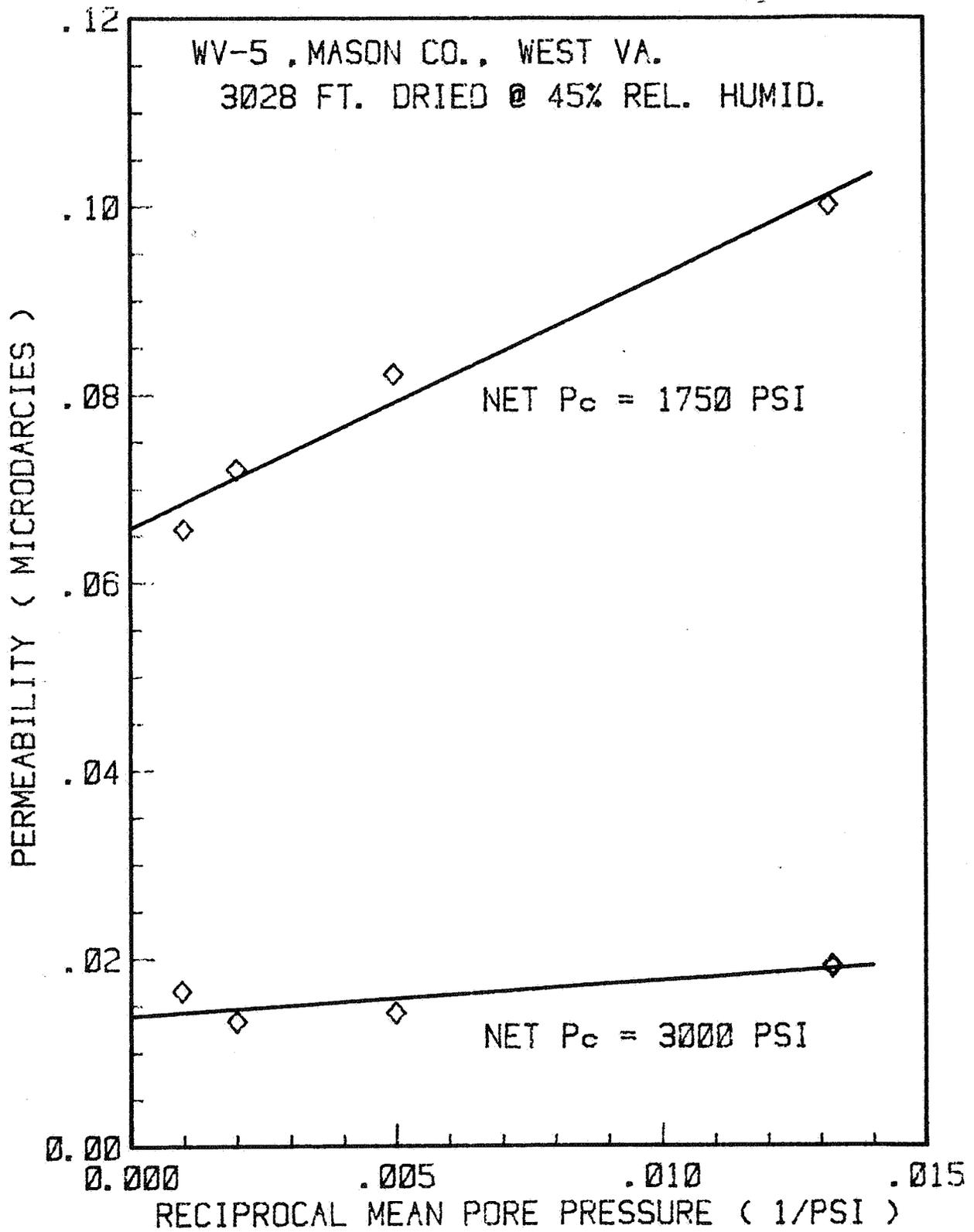
APPENDIX A. Permeability Data and Klinkenberg Plots
of Devonian Shale Cores



WELL NAME: WV-5, MASON CO., WEST VA.

PLUG: 302B FT. DRIED @ 45% REL. HUMI PLUG LENGTH: 3.644 CM PLUG AREA: 10.432 CM²

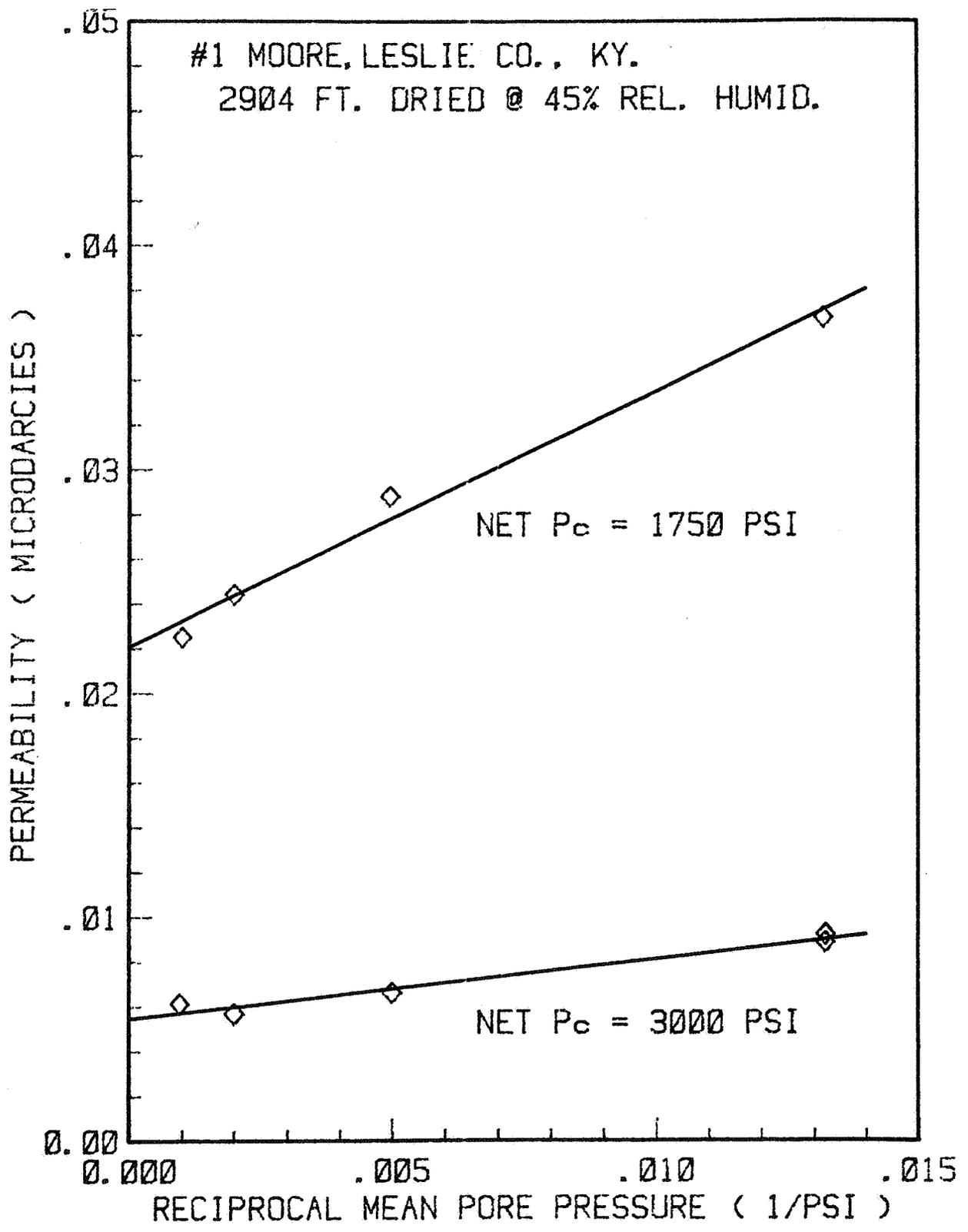
| TEST DATE (M-D) | BEGIN TIME (H:M) | DURATION (H:M) | CONFINING PRESSURE (PSIA) | MEAN PORE P (PSIA) | DIFFERENTIAL PRESSURE (PSI) | FLOW RATE (SCC/S) | TEMPERATURE (DEG. F) | VISCOSITY (C POISE) | COMPRESSIBILITY (Z) | PERMEABILITY VALUE (MICRODARCY) | PERMEABILITY STD. DEV. (%) |
|-----------------|------------------|----------------|---------------------------|--------------------|-----------------------------|-------------------|----------------------|---------------------|---------------------|---------------------------------|----------------------------|
| 07-19 | 20:36 | 13:36 | 1924 | 202.02 | 19.68 | 3.03E-004 | 94.00 | 0.01857 | 0.9986 | 1.14E-001 | (1.3) |
| 07-23 | 09:35 | 02:00 | 1821 | 75.96 | 99.88 | 5.10E-004 | 94.00 | 0.01851 | 0.9994 | 1.00E-001 | (.8) |
| 07-26 | 16:10 | 01:34 | 1947 | 201.54 | 97.94 | 1.09E-003 | 94.00 | 0.01857 | 0.9986 | 9.21E-002 | (.5) |
| 07-30 | 11:58 | 02:30 | 2298 | 500.06 | 99.00 | 2.36E-003 | 94.00 | 0.01880 | 0.9982 | 7.20E-002 | (1.1) |
| 08-03 | 14:00 | 00:45 | 2744 | 1000.53 | 97.73 | 4.11E-003 | 94.00 | 0.01942 | 1.0017 | 6.56E-002 | (1.4) |
| 08-07 | 12:45 | 04:09 | 4057 | 1047.40 | 99.63 | 1.10E-003 | 94.00 | 0.01950 | 1.0023 | 1.65E-002 | (3.3) |
| 08-09 | 19:10 | 16:40 | 3504 | 500.68 | 98.07 | 4.32E-004 | 94.00 | 0.01880 | 0.9982 | 1.33E-002 | (3.2) |
| 08-13 | 14:00 | 17:45 | 3187 | 200.41 | 99.79 | 1.90E-004 | 94.00 | 0.01857 | 0.9987 | 1.42E-002 | (5.1) |
| 08-16 | 10:20 | 28:20 | 3092 | 75.69 | 98.84 | 9.52E-005 | 94.00 | 0.01851 | 0.9994 | 1.89E-002 | (1.4) |
| 08-17 | 16:40 | 32:20 | 3069 | 75.71 | 98.78 | 9.59E-005 | 94.00 | 0.01851 | 0.9994 | 1.91E-002 | (1.3) |
| 08-19 | 03:00 | 28:00 | 3072 | 75.72 | 98.73 | 9.64E-005 | 94.00 | 0.01851 | 0.9994 | 1.92E-002 | (1.1) |



 WELL NAME: #1 MOORE, LESLIE CO., KY.

PLUG: 2904 FT, DRIED @ 45% REL. HUMI PLUG LENGTH: 3.851 CM PLUG AREA: 10.391 CM^2

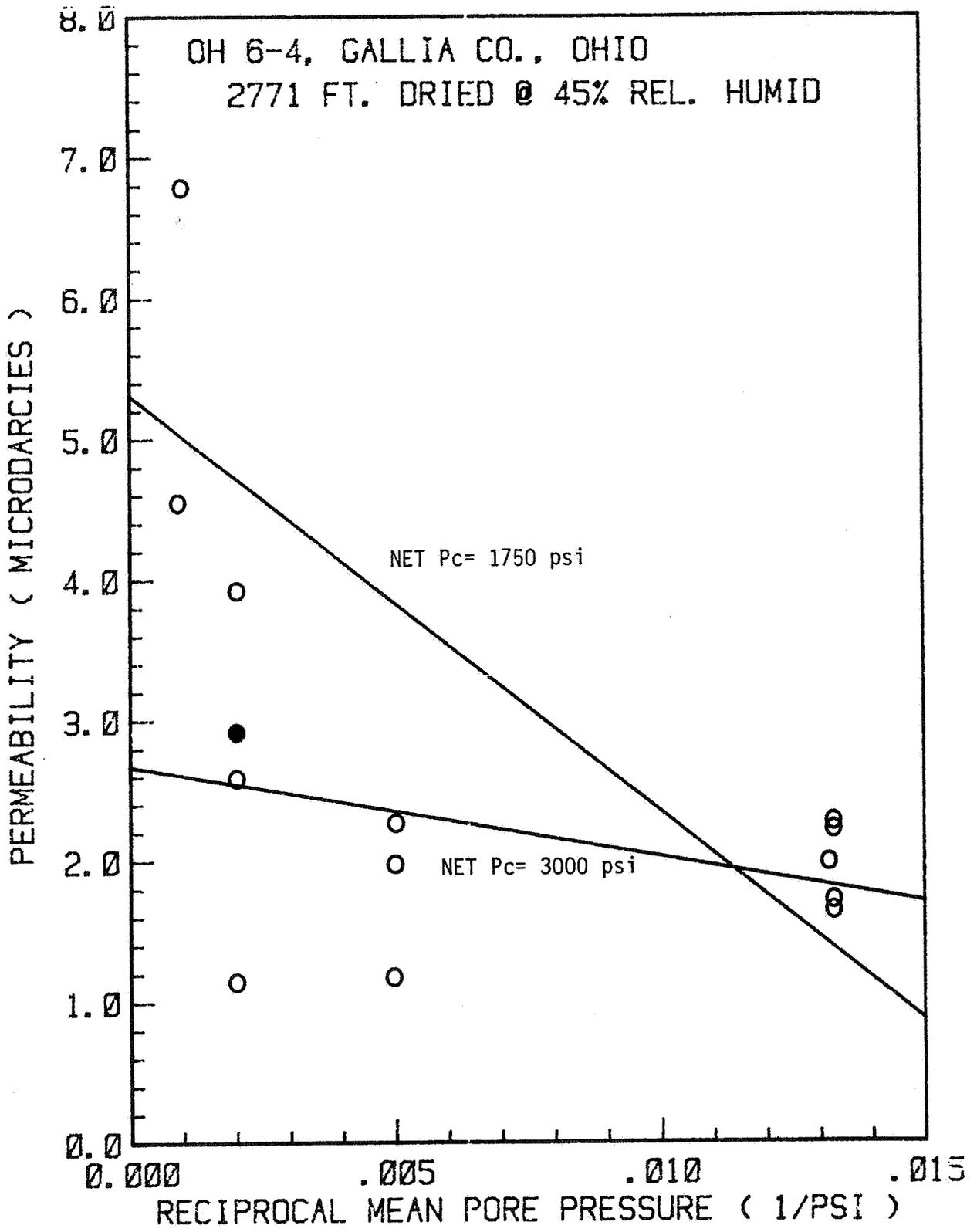
| TEST DATE (M-D) | BEGIN TIME (H:M) | DUR- ATION (H:M) | CONFINING PRESSURE (PSIA) | MEAN PORE P (PSIA) | DIFFERENTIAL PRESSURE (PSI) | FLOW RATE (SCCF/S) | TEMPERATURE (DEG. F) | VISCOSITY (C POISE) | COMPRES- SIBILITY (Z) | PERMEABILITY VALUE (MICRODARCY) | STD. DEV. (%) |
|-----------------|------------------|------------------|---------------------------|--------------------|-----------------------------|--------------------|----------------------|---------------------|-----------------------|---------------------------------|---------------|
| 07-19 | 23:00 | 11:48 | 1916 | 202.00 | 19.72 | 8.40E-005 | 94.00 | 0.01857 | 0.9986 | 3.34E-002 | (2.9) |
| 07-23 | 13:24 | 06:32 | 1801 | 75.94 | 99.80 | 1.76E-004 | 94.00 | 0.01851 | 0.9994 | 3.67E-002 | (1.3) |
| 07-26 | 10:45 | 05:00 | 1957 | 201.58 | 98.06 | 3.59E-004 | 94.00 | 0.01857 | 0.9986 | 2.88E-002 | (1.1) |
| 07-30 | 19:00 | 11:37 | 2269 | 500.10 | 98.84 | 7.52E-004 | 94.00 | 0.01880 | 0.9982 | 2.44E-002 | (1.6) |
| 08-03 | 10:10 | 03:20 | 2749 | 1000.59 | 97.88 | 1.33E-003 | 94.00 | 0.01942 | 1.0017 | 2.25E-002 | (1.5) |
| 08-06 | 19:00 | 16:20 | 4047 | 1047.58 | 99.78 | 3.86E-004 | 94.00 | 0.01950 | 1.0023 | 6.13E-003 | (7.9) |
| 08-09 | 18:10 | 16:50 | 3505 | 500.68 | 98.09 | 1.74E-004 | 94.00 | 0.01880 | 0.9982 | 5.69E-003 | (5.5) |
| 08-13 | 14:00 | 19:15 | 3185 | 200.40 | 99.79 | 8.35E-005 | 94.00 | 0.01857 | 0.9987 | 6.61E-003 | (2.6) |
| 08-16 | 10:20 | 45:00 | 3084 | 75.72 | 98.77 | 4.36E-005 | 94.00 | 0.01851 | 0.9994 | 9.20E-003 | (2.5) |
| 08-18 | 11:00 | 45:40 | 3070 | 75.75 | 98.70 | 4.18E-005 | 94.00 | 0.01851 | 0.9994 | 8.84E-003 | (2.0) |



WELL NAME: OH 6-4. GALLIA CO., OHIO

PLUG: 2771 FT. DRIED @ 45% REL. HUMI PLUG LENGTH: 3.902 CM PLUG AREA: 10.420 CM^2

| TEST DATE (M-D) | BEGIN TIME (H:M) | DUR- ATION (H:M) | CONFINING PRESSURE (PSIA) | MEAN PORE P (PSIA) | DIFFERENTIAL PRESSURE (PSI) | FLOW RATE (SCC/S) | TEMPERATURE (DEC. F) | VISCOSITY (C POISE) | COMPRES- SIBILITY (Z) | PERMEABILITY VALUE (MICRODARCY) | STD. DEV. (%) |
|-----------------|------------------|------------------|---------------------------|--------------------|-----------------------------|-------------------|----------------------|---------------------|-----------------------|---------------------------------|---------------|
| 07-24 | 10:30 | 03:15 | 1828 | 75.92 | 5.32 | 5.03E-004 | 94.00 | 0.01851 | 0.9994 | 1.99E+000 | (14.7) |
| 07-27 | 09:22 | 02:30 | 1966 | 199.58 | 5.13 | 1.45E-003 | 94.00 | 0.01857 | 0.9987 | 2.27E+000 | (6.0) |
| 07-31 | 11:56 | 01:05 | 2247 | 497.29 | 4.25 | 5.12E-003 | 94.00 | 0.01880 | 0.9982 | 3.92E+000 | (1.9) |
| 08-03 | 15:37 | 00:23 | 2750 | 1000.20 | 4.81 | 1.95E-002 | 94.00 | 0.01942 | 1.0017 | 6.79E+000 | (2.7) |
| 08-06 | 16:04 | 00:21 | 4118 | 1095.20 | 4.56 | 1.35E-002 | 94.00 | 0.01957 | 1.0030 | 4.55E+000 | (2.8) |
| 08-09 | 10:47 | 02:20 | 3533 | 502.07 | 4.55 | 1.61E-003 | 94.00 | 0.01880 | 0.9982 | 1.14E+000 | (17.3) |
| 08-10 | 14:53 | 00:20 | 3531 | 500.03 | 10.26 | 9.24E-003 | 94.00 | 0.01880 | 0.9982 | 2.92E+000 | (1.0) |
| 08-10 | 15:32 | 01:49 | 3528 | 499.89 | 4.97 | 3.97E-003 | 94.00 | 0.01857 | 0.9986 | 2.59E+000 | (2.4) |
| 08-12 | 13:40 | 03:45 | 3216 | 201.54 | 5.09 | 7.53E-004 | 94.00 | 0.01857 | 0.9987 | 1.98E+000 | (2.3) |
| 08-14 | 12:14 | 01:48 | 3247 | 200.39 | 4.69 | 1.16E-003 | 94.00 | 0.01851 | 0.9994 | 1.72E+000 | (8.2) |
| 08-14 | 15:25 | 08:20 | 3093 | 75.40 | 4.69 | 3.80E-004 | 94.00 | 0.01851 | 0.9994 | 1.64E+000 | (2.6) |
| 08-15 | 00:25 | 08:30 | 3084 | 75.41 | 4.63 | 3.60E-004 | 94.00 | 0.01851 | 0.9994 | 1.64E+000 | (2.6) |
| 08-20 | 14:30 | 08:55 | 3063 | 75.41 | 4.21 | 4.42E-004 | 94.00 | 0.01851 | 0.9994 | 2.22E+000 | (2.1) |
| 08-20 | 23:55 | 08:55 | 3057 | 75.42 | 4.14 | 4.46E-004 | 94.00 | 0.01851 | 0.9994 | 2.28E+000 | (1.9) |
| 08-21 | 09:15 | 05:33 | 1846 | 75.42 | 4.06 | 8.07E-004 | 94.00 | 0.01851 | 0.9994 | 4.20E+000 | (1.0) |
| 08-21 | 16:33 | 05:24 | 3069 | 76.10 | 5.30 | 9.26E-004 | 94.00 | 0.01851 | 0.9994 | 3.66E+000 | (1.2) |
| 08-21 | 22:09 | 05:30 | 3063 | 76.10 | 5.23 | 8.95E-004 | 94.00 | 0.01851 | 0.9994 | 3.59E+000 | (.4) |
| 08-22 | 03:57 | 05:03 | 3060 | 76.10 | 5.15 | 8.82E-004 | 94.00 | 0.01851 | 0.9994 | 3.59E+000 | (.4) |

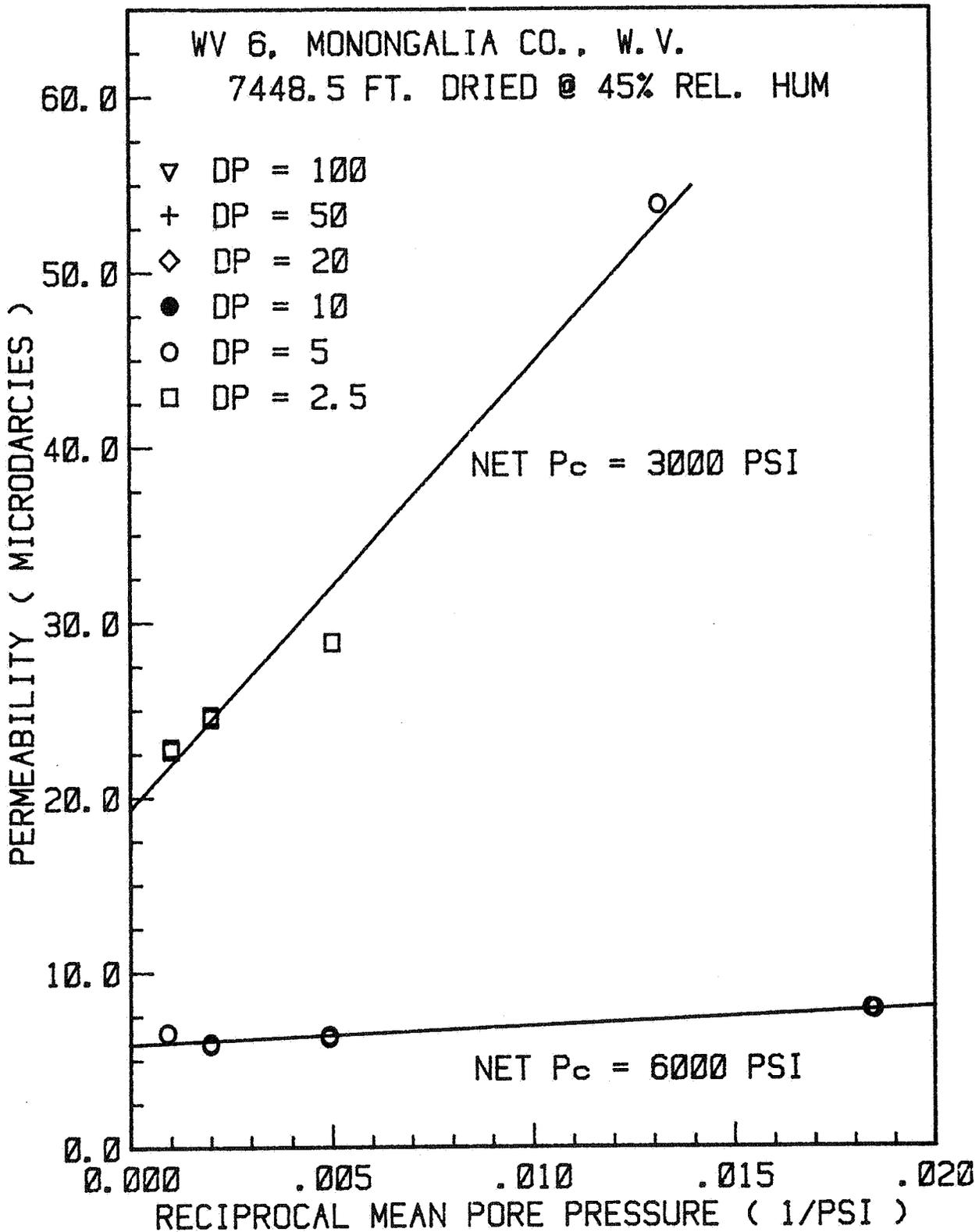


Lack of measurable gas flow through the EGSP OH-9 core prevented construction of a Klinkenberg plot for this sample. As explained in the text, gas flow was blocked by oil in the rock pores.

WELL NAME: WU 6, MONGOLIA CO., W.V.

PLUG: 748.5 FT. DRIED @ 45% REL. HU PLUG LENGTH: 4.053 CM PLUG AREA: 11.391 CM²

| TEST DATE (M-D) | BEGIN TIME (H:M) | DURATION (H:M) | CONFINING PRESSURE (PSIA) | MEAN PORE P (PSIA) | DIFFERENTIAL PRESSURE (PSI) | FLOW RATE (SCC/S) | TEMPERATURE (DEG. F) | VISCOSITY (C POISE) | COMPRESSIONIBILITY (Z) | PERMEABILITY VALUE (MICRODARCY) | STD. DEV. (%) |
|-----------------|------------------|----------------|---------------------------|--------------------|-----------------------------|-------------------|----------------------|---------------------|------------------------|---------------------------------|---------------|
| 08-27 | 18:03 | 00:13 | 3069 | 75.92 | 4.71 | 1.37E-002 | 94.00 | 0.01851 | 0.9994 | 5.38E+001 | (2.1) |
| 08-30 | 09:42 | 00:30 | 3108 | 199.65 | 2.21 | 8.37E-003 | 94.00 | 0.01857 | 0.9987 | 2.88E+001 | (1.6) |
| 09-01 | 12:34 | 00:10 | 3564 | 501.02 | 2.62 | 2.11E-002 | 94.00 | 0.01880 | 0.9982 | 2.47E+001 | (2.4) |
| 09-02 | 12:37 | 00:10 | 3522 | 501.08 | 2.34 | 1.87E-002 | 94.00 | 0.01880 | 0.9982 | 2.45E+001 | (1.8) |
| 09-04 | 09:29 | 00:14 | 4024 | 999.33 | 1.67 | 2.40E-002 | 94.00 | 0.01942 | 1.0017 | 2.28E+001 | (1.9) |
| 09-05 | 13:21 | 00:15 | 4016 | 1000.42 | 1.28 | 1.83E-002 | 94.00 | 0.01942 | 1.0017 | 2.27E+001 | (1.7) |
| 09-08 | 19:23 | 00:16 | 7094 | 1106.48 | 4.12 | 1.86E-002 | 94.00 | 0.01959 | 1.0031 | 6.55E+000 | (1.8) |
| 09-19 | 14:14 | 00:46 | 6505 | 502.97 | 5.09 | 9.92E-003 | 94.00 | 0.01880 | 0.9982 | 5.96E+000 | (3.0) |
| 09-24 | 10:31 | 00:52 | 6518 | 503.89 | 5.12 | 9.81E-003 | 94.00 | 0.01880 | 0.9982 | 5.86E+000 | (1.5) |
| 09-25 | 10:31 | 00:52 | 6202 | 203.05 | 5.35 | 4.56E-003 | 94.00 | 0.01857 | 0.9986 | 6.39E+000 | (2.0) |
| 09-28 | 13:47 | 00:44 | 6221 | 203.45 | 5.16 | 4.32E-003 | 94.00 | 0.01857 | 0.9986 | 6.26E+000 | (2.4) |
| 10-05 | 13:01 | 01:04 | 6069 | 54.10 | 5.28 | 1.49E-003 | 90.00 | 0.01851 | 0.9996 | 7.82E+000 | (3.3) |
| 10-08 | 11:57 | 01:28 | 6062 | 54.56 | 5.24 | 1.49E-003 | 90.00 | 0.01851 | 0.9996 | 7.87E+000 | (3.4) |



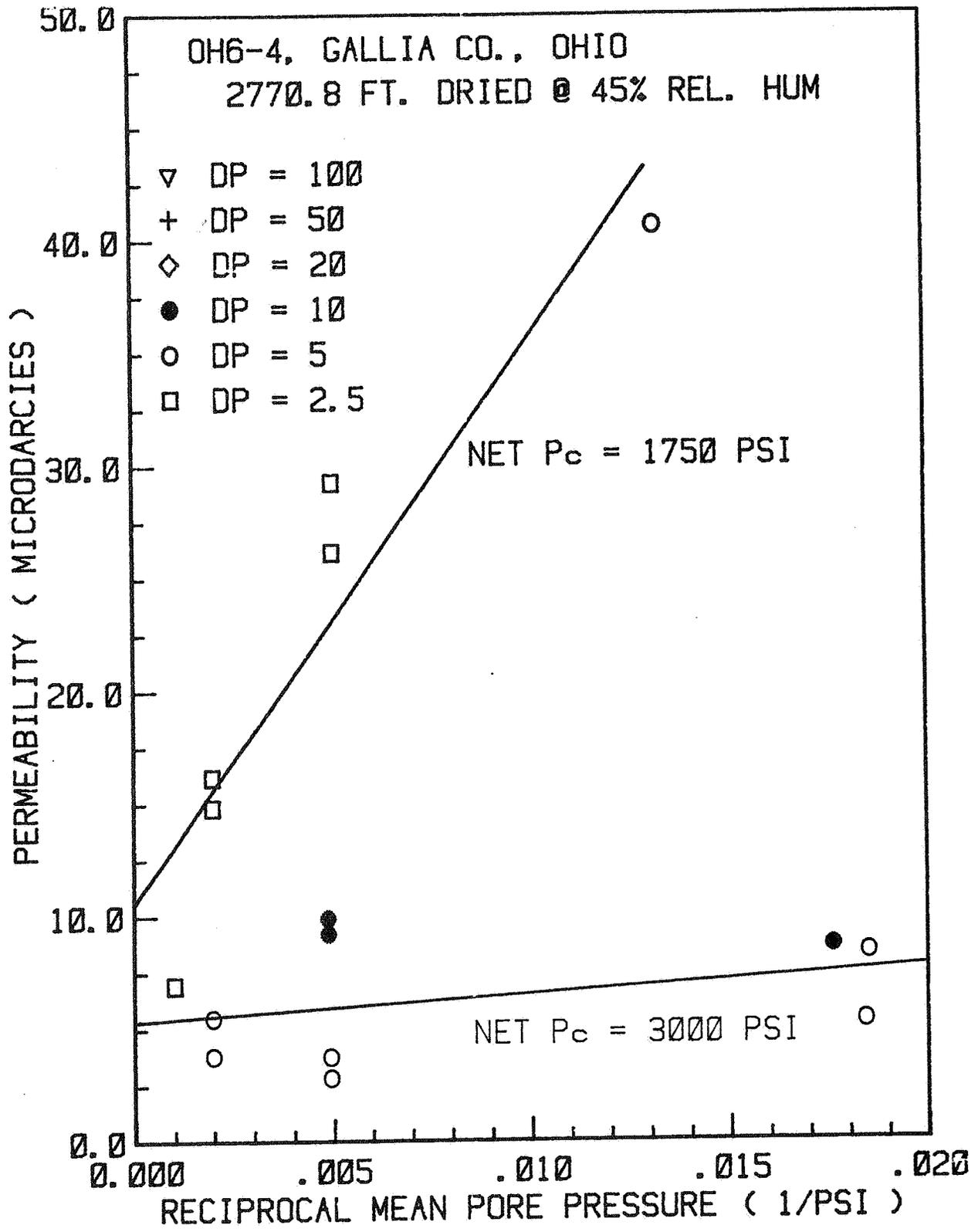
WELL NAME: OH6-4, GALLIA CO., OHIO

PLUG AREA: 11.378 CM^2

PLUG LENGTH: 3.774 CM

PLUG: 2770.8 FT, DRIED @ 45% REL. HU

| TEST DATE (M-D) | BEGIN TIME (H:M) | DUR-ATION (H:M) | CONFINING PRESSURE (PSIA) | MEAN PORE P (PSIA) | DIFFERENTIAL PRESSURE (PSI) | FLOW RATE (SCCF/S) | TEMPERATURE (DEG. F) | VISCOSITY (C POISE) | COMPRES-SIBILITY (Z) | PERMEABILITY VALUE (MICRODARCY) | STD. DEV. (%) |
|-----------------|------------------|-----------------|---------------------------|--------------------|-----------------------------|--------------------|----------------------|---------------------|----------------------|---------------------------------|---------------|
| 08-27 | 18:04 | 00:12 | 1833 | 75.90 | 4.73 | 1.03E-002 | 94.00 | 0.01851 | 0.9994 | 4.06E+001 | (5.8) |
| 08-30 | 09:38 | 00:25 | 1953 | 199.66 | 2.22 | 7.16E-003 | 94.00 | 0.01857 | 0.9987 | 2.93E+001 | (8.9) |
| 08-30 | 10:05 | 00:18 | 1952 | 199.65 | 2.14 | 7.89E-003 | 94.00 | 0.01857 | 0.9987 | 2.61E+001 | (5.8) |
| 09-01 | 12:35 | 00:11 | 2279 | 501.04 | 2.59 | 1.46E-002 | 94.00 | 0.01880 | 0.9982 | 1.62E+001 | (6.8) |
| 09-02 | 12:37 | 00:21 | 2265 | 501.07 | 2.35 | 1.22E-002 | 94.00 | 0.01880 | 0.9982 | 1.48E+001 | (5.6) |
| 09-04 | 09:47 | 00:20 | 2761 | 1000.50 | 4.12 | 1.59E-002 | 94.00 | 0.01943 | 1.0017 | 5.75E+000 | (15.5) |
| 09-04 | 10:12 | 00:06 | 2760 | 1003.08 | 8.80 | 8.49E-002 | 94.00 | 0.01943 | 1.0017 | 1.43E+001 | (15.2) |
| 09-05 | 14:24 | 00:27 | 2753 | 1001.09 | 2.58 | 1.21E-002 | 94.00 | 0.01958 | 1.0031 | 6.94E+000 | (5.6) |
| 09-06 | 17:06 | 00:17 | 2852 | 1102.56 | 5.56 | 3.68E-002 | 94.00 | 0.01958 | 1.0031 | 9.01E+000 | (37.6) |
| 09-06 | 17:48 | 00:31 | 2862 | 1102.49 | 3.60 | 2.41E-002 | 94.00 | 0.01958 | 1.0031 | 9.08E+000 | (13.5) |
| 09-06 | 18:18 | 00:01 | 2862 | 1102.53 | 3.39 | 2.56E-002 | 94.00 | 0.01958 | 1.0031 | 1.02E+001 | (1.0) |
| 09-09 | 00:10 | 00:17 | 4120 | 1100.89 | 9.55 | 3.24E-002 | 94.00 | 0.01958 | 1.0030 | 4.61E+000 | (28.2) |
| 09-18 | 11:38 | 00:25 | 4096 | 1102.07 | 9.56 | 5.71E-002 | 94.01 | 0.01880 | 1.0031 | 8.10E+000 | (1.2) |
| 09-19 | 10:19 | 00:25 | 3507 | 505.39 | 10.23 | 1.50E-002 | 94.00 | 0.01880 | 0.9982 | 4.18E+000 | (4.2) |
| 09-19 | 14:14 | 00:46 | 3505 | 502.96 | 5.11 | 6.78E-003 | 94.00 | 0.01880 | 0.9982 | 3.78E+000 | (1.7) |
| 09-24 | 10:52 | 00:05 | 3506 | 506.31 | 10.22 | 2.20E-002 | 94.00 | 0.01881 | 0.9982 | 6.10E+000 | (2.6) |
| 09-24 | 12:35 | 00:13 | 3505 | 503.90 | 5.10 | 9.80E-003 | 94.00 | 0.01880 | 0.9982 | 5.47E+000 | (2.2) |
| 09-25 | 09:03 | 00:13 | 3206 | 205.51 | 10.30 | 1.38E-002 | 94.00 | 0.01857 | 0.9986 | 9.21E+000 | (4.7) |
| 09-25 | 10:29 | 00:46 | 3205 | 203.05 | 5.36 | 2.16E-002 | 94.00 | 0.01857 | 0.9986 | 2.81E+000 | (8.8) |
| 09-28 | 10:34 | 00:10 | 3214 | 205.89 | 10.29 | 1.48E-002 | 94.00 | 0.01857 | 0.9986 | 9.89E+000 | (4.3) |
| 09-28 | 13:51 | 00:34 | 3212 | 203.43 | 5.20 | 2.80E-003 | 94.00 | 0.01857 | 0.9986 | 3.75E+000 | (1.9) |
| 10-05 | 09:24 | 00:58 | 3057 | 56.55 | 10.28 | 2.74E-003 | 90.00 | 0.01851 | 0.9995 | 6.61E+000 | (7.0) |
| 10-05 | 13:01 | 01:05 | 3055 | 54.10 | 5.29 | 1.70E-003 | 90.00 | 0.01851 | 0.9995 | 8.35E+000 | (2.4) |
| 10-08 | 10:09 | 00:19 | 3079 | 56.90 | 10.29 | 3.60E-003 | 90.00 | 0.01851 | 0.9995 | 8.63E+000 | (1.0) |
| 10-08 | 12:08 | 01:07 | 3078 | 54.36 | 5.24 | 1.07E-003 | 90.00 | 0.01851 | 0.9996 | 5.28E+000 | (5.1) |



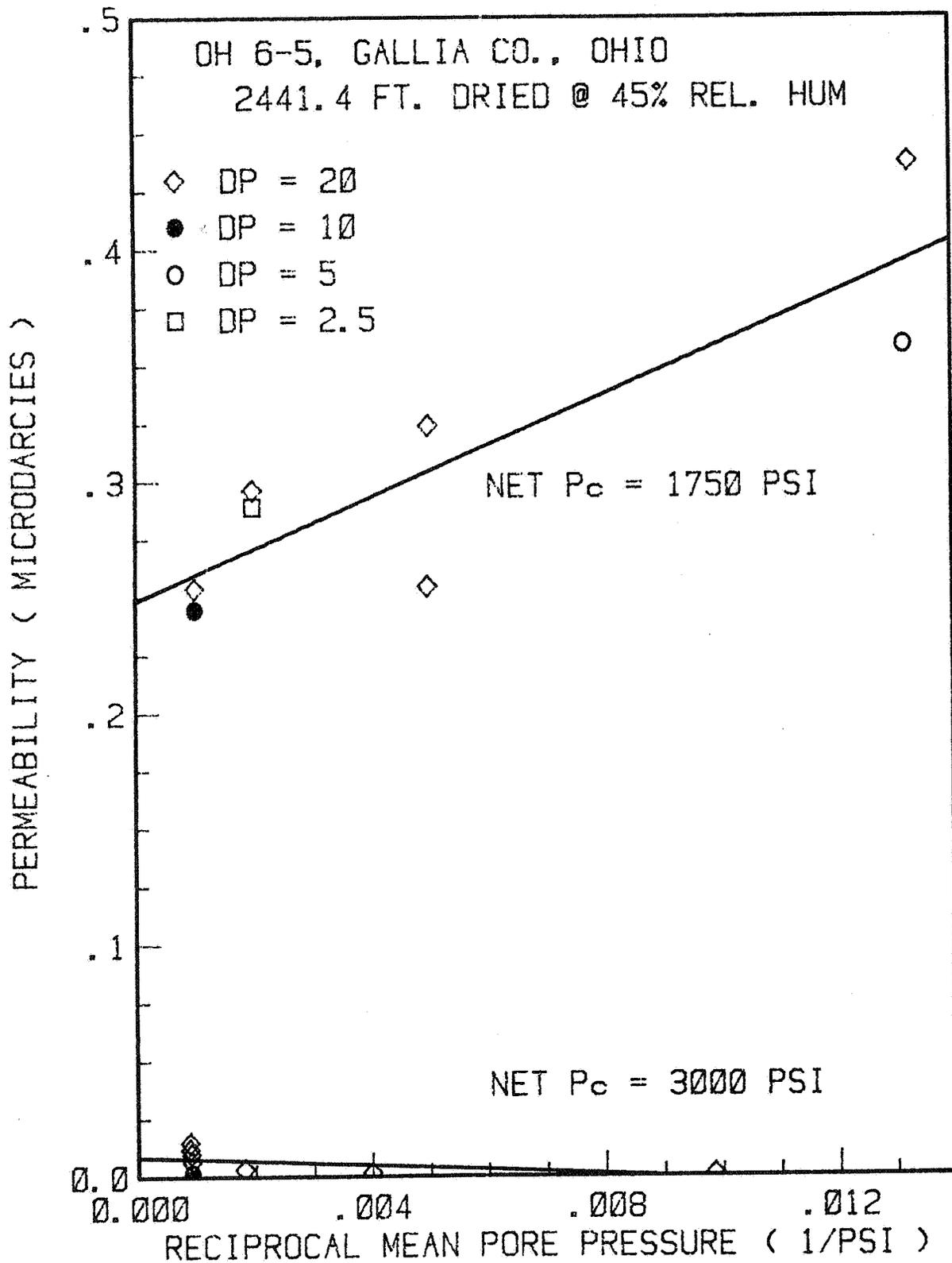
WELL NAME: OH 6-5, GALLIA CO., OHIO

PLUG AREA: 11.386 CM^2

PLUG LENGTH: 3.983 CM

PLUG: 2441.4 FT. DRIED @ 45% REL. HU

| TEST DATE (M-D) | BEGIN TIME (H:M) | DUR-ATION (H:M) | CONFINING PRESSURE (PSIA) | MEAN PORE P (PSIA) | DIFFERENTIAL PRESSURE (PSI) | FLOW RATE (SCC/S) | TEMPERATURE (DEG. F) | VISCOSITY (C POISE) | COMPRES-SIBILITY (Z) | PERMEABILITY VALUE (MICRODARCY) | PERMEABILITY STD. DEV. (%) |
|-----------------|------------------|-----------------|---------------------------|--------------------|-----------------------------|-------------------|----------------------|---------------------|----------------------|---------------------------------|----------------------------|
| 08-27 | 14:42 | 00:40 | 1844 | 75.39 | 97.70 | 2.16E-003 | 94.00 | 0.01851 | 0.9994 | 4.37E-001 | (2.3) |
| 08-27 | 20:30 | 12:45 | 1806 | 75.93 | 4.65 | 8.48E-005 | 94.00 | 0.01851 | 0.9994 | 3.58E-001 | (2.1) |
| 08-30 | 17:45 | 08:25 | 1967 | 200.57 | 18.86 | 6.44E-004 | 94.00 | 0.01857 | 0.9987 | 2.54E-001 | (1.3) |
| 08-31 | 15:29 | 00:59 | 1971 | 199.81 | 99.70 | 4.32E-003 | 94.00 | 0.01857 | 0.9987 | 3.24E-001 | (2.6) |
| 09-01 | 14:00 | 22:30 | 2247 | 501.07 | 2.47 | 2.35E-004 | 94.00 | 0.01880 | 0.9982 | 2.89E-001 | (6.9) |
| 09-02 | 15:02 | 01:25 | 2262 | 501.50 | 20.76 | 2.04E-003 | 94.00 | 0.01880 | 0.9982 | 2.96E-001 | (1.7) |
| 09-04 | 11:35 | 05:05 | 2753 | 1002.82 | 7.67 | 1.20E-003 | 94.00 | 0.01943 | 1.0017 | 2.45E-001 | (2.9) |
| 09-05 | 09:15 | 02:27 | 2761 | 1000.42 | 20.16 | 3.27E-003 | 94.00 | 0.01942 | 1.0017 | 2.54E-001 | (2.8) |
| 09-09 | 02:00 | 08:40 | 4109 | 1100.94 | 9.27 | 8.67E-006 | 94.00 | 0.01958 | 1.0030 | 1.34E-003 | (0.0) |
| 09-09 | 15:40 | 38:20 | 4088 | 1106.25 | 20.42 | 8.99E-005 | 94.00 | 0.01959 | 1.0031 | 6.27E-003 | (13.5) |
| 09-11 | 17:30 | 14:30 | 4152 | 1120.48 | 48.88 | 3.36E-004 | 94.00 | 0.01961 | 1.0033 | 9.69E-003 | (9.2) |
| 09-12 | 17:52 | 14:22 | 4119 | 1145.87 | 98.61 | 1.04E-003 | 94.00 | 0.01965 | 1.0037 | 1.46E-002 | (3.7) |
| 09-17 | 23:40 | 09:31 | 4102 | 1145.25 | 96.86 | 8.26E-004 | 94.00 | 0.01965 | 1.0037 | 1.18E-002 | (15.9) |
| 09-19 | 20:40 | 20:39 | 3540 | 550.13 | 98.58 | 1.08E-004 | 94.00 | 0.01885 | 0.9983 | 3.02E-003 | (20.0) |
| 09-25 | 18:20 | 24:10 | 3250 | 250.53 | 99.67 | 2.26E-005 | 94.00 | 0.01880 | 0.9984 | 1.35E-003 | (87.1) |
| 10-05 | 22:00 | 37:32 | 3101 | 101.56 | 99.25 | 4.14E-006 | 90.00 | 0.01852 | 0.9992 | 6.08E-004 | (9.7) |



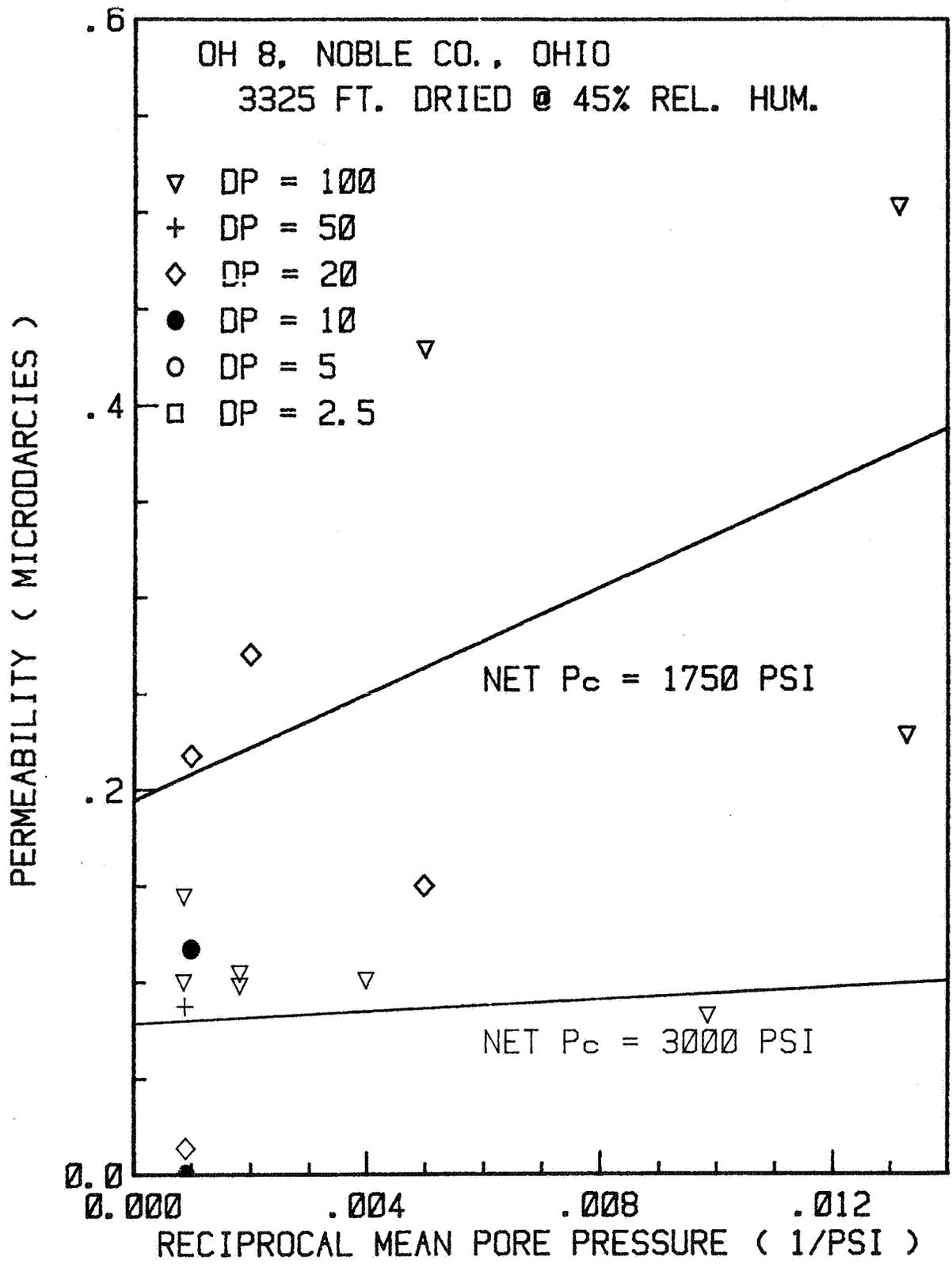
WELL NAME: OH 8, NORBLE CO., OHIO

PLUG AREA: 11.406 CM^2

PLUG LENGTH: 3.816 CM

PLUG: 3325 FT. DRIED @ 45% REL. HUM.

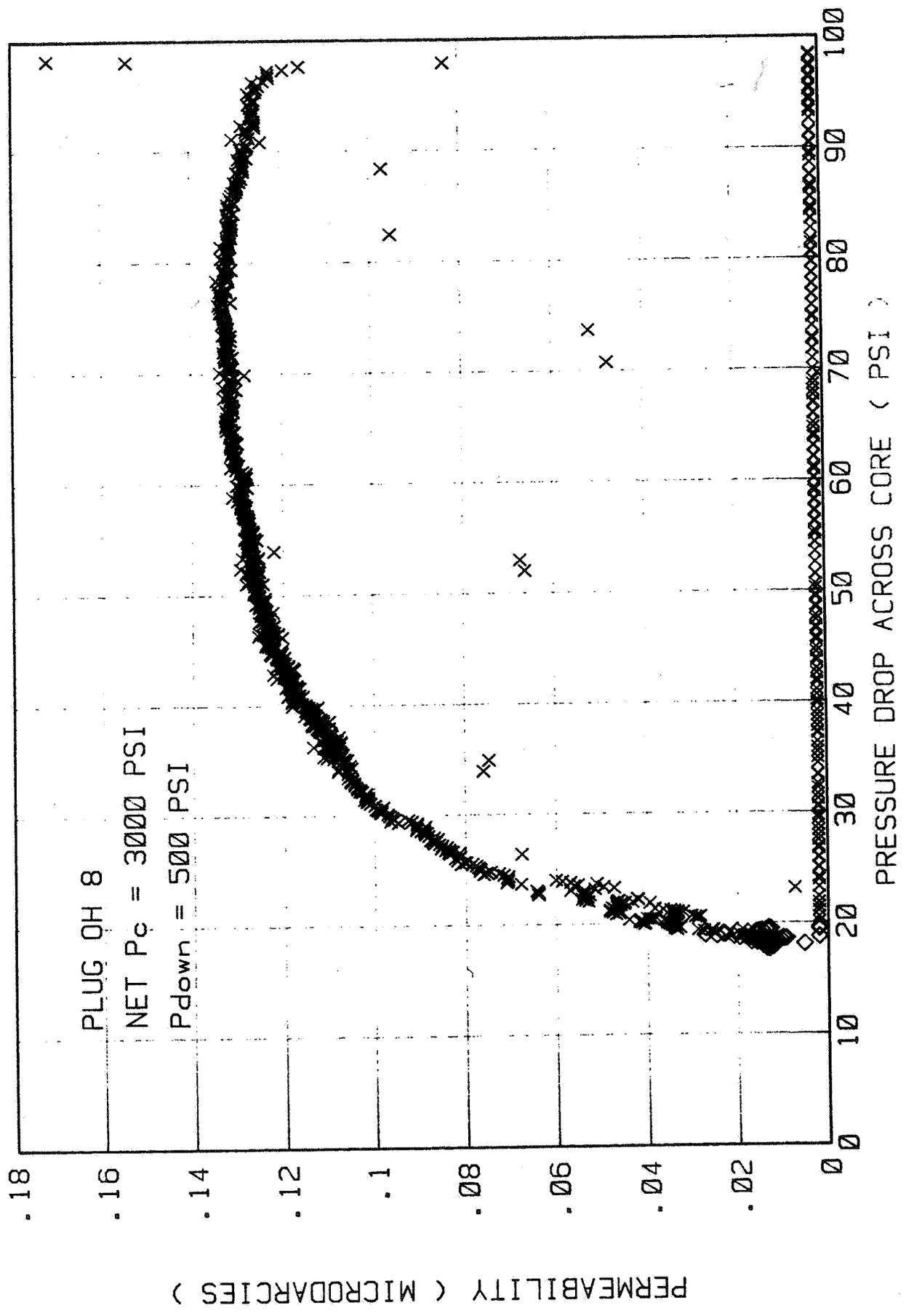
| TEST DATE (M-D) | BEGIN TIME (H:M) | DURATION (H:M) | CONFINING PRESSURE (PSIA) | MEAN PORE P (PSIA) | DIFFERENTIAL PRESSURE (PSI) | FLOW RATE (SCCY/S) | TEMPERATURE (DEG. F) | VISCOSITY (C POISE) | COMPRESSIBILITY (Z) | PERMEABILITY VALUE (MICRODARCY) | PERMEABILITY STD. DEV. (%) |
|-----------------|------------------|----------------|---------------------------|--------------------|-----------------------------|--------------------|----------------------|---------------------|---------------------|---------------------------------|----------------------------|
| 08-27 | 14:42 | 00:40 | 1844 | 75.37 | 97.73 | 1.18E-003 | 94.00 | 0.01851 | 0.9994 | 2.29E-001 | (29.3) |
| 08-28 | 13:28 | 02:09 | 1836 | 76.10 | 97.90 | 2.63E-003 | 94.00 | 0.01851 | 0.9994 | 5.04E-001 | (22.0) |
| 08-31 | 01:10 | 06:55 | 1950 | 200.55 | 18.76 | 3.94E-004 | 94.00 | 0.01857 | 0.9987 | 1.50E-001 | (9.6) |
| 08-31 | 15:40 | 00:48 | 1970 | 199.82 | 99.66 | 5.99E-003 | 94.00 | 0.01857 | 0.9987 | 4.30E-001 | (18.2) |
| 09-03 | 09:54 | 02:50 | 2254 | 501.46 | 20.29 | 1.90E-003 | 94.00 | 0.01880 | 0.9982 | 2.70E-001 | (2.9) |
| 09-04 | 23:40 | 08:25 | 2746 | 1000.45 | 14.16 | 1.11E-003 | 94.00 | 0.01942 | 1.0017 | 1.17E-001 | (9.4) |
| 09-05 | 09:20 | 02:22 | 2761 | 1000.41 | 20.18 | 2.94E-003 | 94.00 | 0.01942 | 1.0017 | 2.18E-001 | (7.3) |
| 09-09 | 02:00 | 12:00 | 4109 | 1100.91 | 9.33 | 4.95E-006 | 94.00 | 0.01958 | 1.0030 | 7.26E-004 | (76.9) |
| 09-09 | 21:20 | 36:40 | 4088 | 1106.17 | 20.40 | 2.04E-004 | 94.00 | 0.01959 | 1.0031 | 1.37E-002 | (39.5) |
| 09-11 | 13:54 | 03:15 | 4167 | 1120.53 | 49.10 | 3.19E-003 | 94.00 | 0.01961 | 1.0033 | 8.75E-002 | (8.8) |
| 09-12 | 15:06 | 01:56 | 4132 | 1145.41 | 98.17 | 7.52E-003 | 94.00 | 0.01965 | 1.0037 | 1.01E-001 | (15.2) |
| 09-17 | 21:58 | 00:46 | 4107 | 1145.29 | 96.98 | 1.07E-002 | 94.00 | 0.01885 | 0.9983 | 1.46E-001 | (.3) |
| 09-19 | 19:56 | 00:31 | 3532 | 550.10 | 98.69 | 3.72E-003 | 94.00 | 0.01885 | 0.9983 | 9.73E-002 | (1.4) |
| 09-20 | 18:03 | 00:18 | 3529 | 550.06 | 98.49 | 3.95E-003 | 94.00 | 0.01860 | 0.9984 | 1.06E-001 | (2.4) |
| 09-25 | 15:43 | 01:10 | 3256 | 250.62 | 99.75 | 1.78E-003 | 94.00 | 0.01860 | 0.9984 | 1.03E-001 | (1.4) |
| 10-05 | 21:12 | 00:33 | 3048 | 101.63 | 99.14 | 5.98E-004 | 90.00 | 0.01852 | 0.9992 | 8.37E-002 | (.3) |

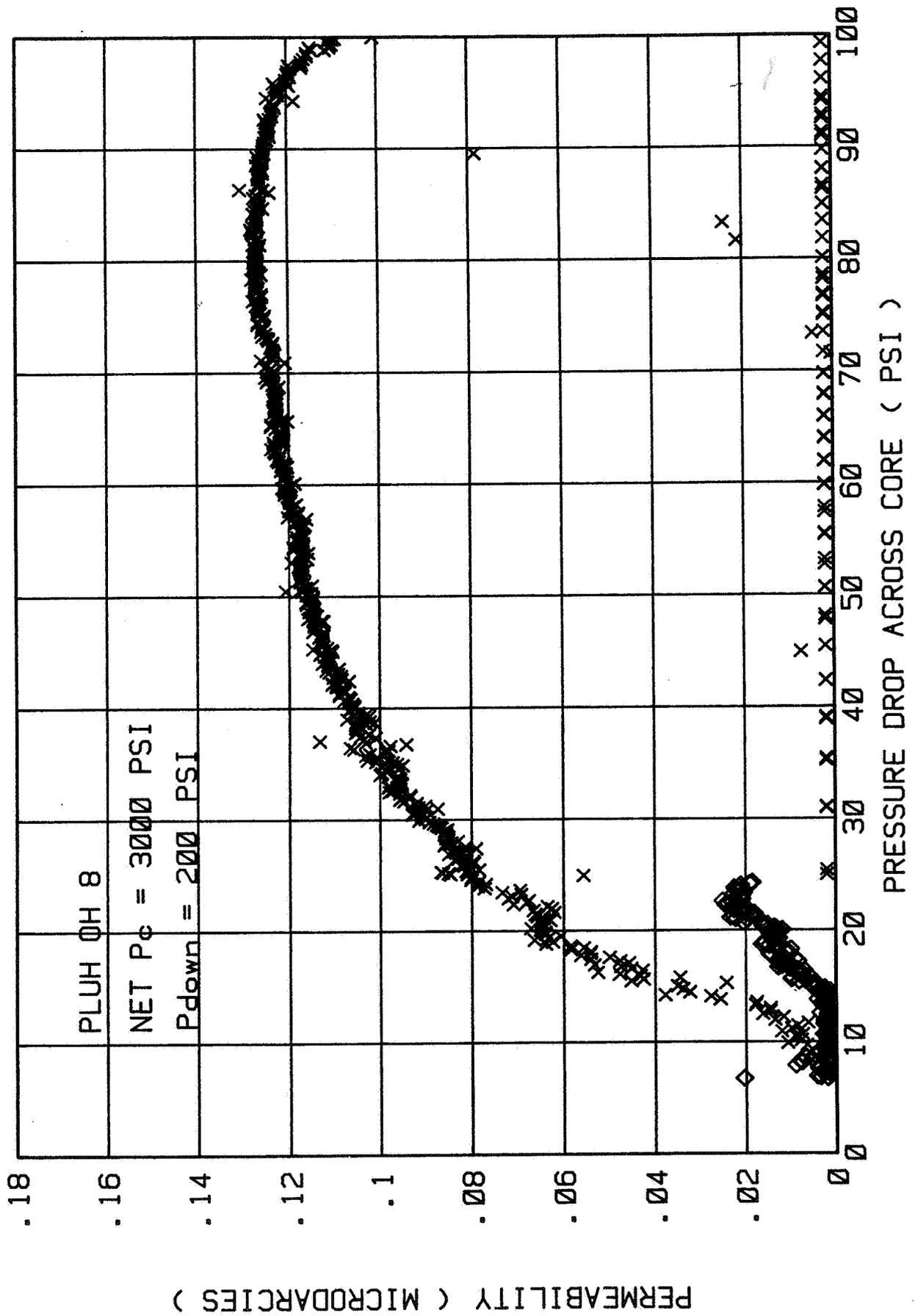


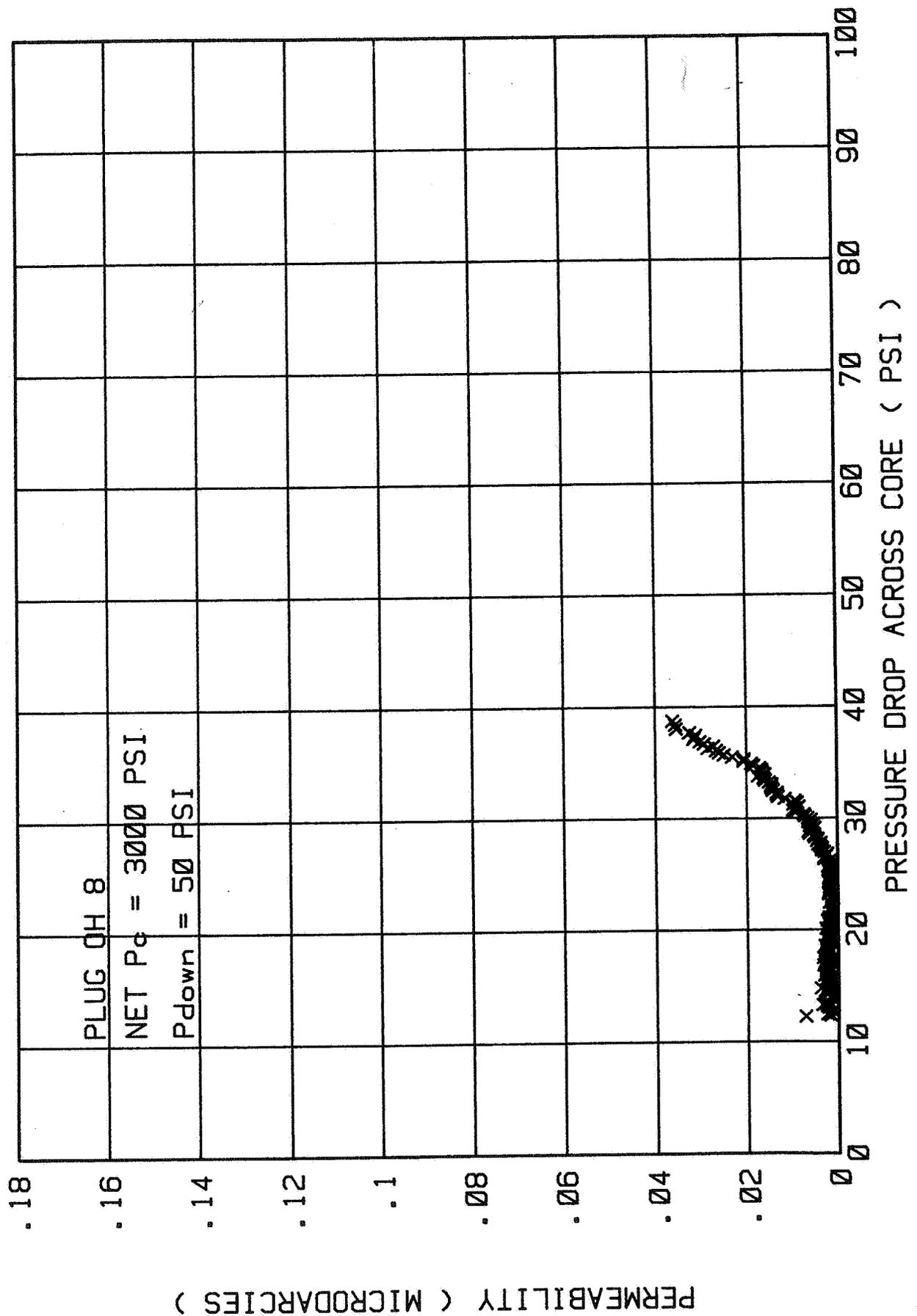


APPENDIX B. Capillary Pressure Measurements in
Lower Huron Shale

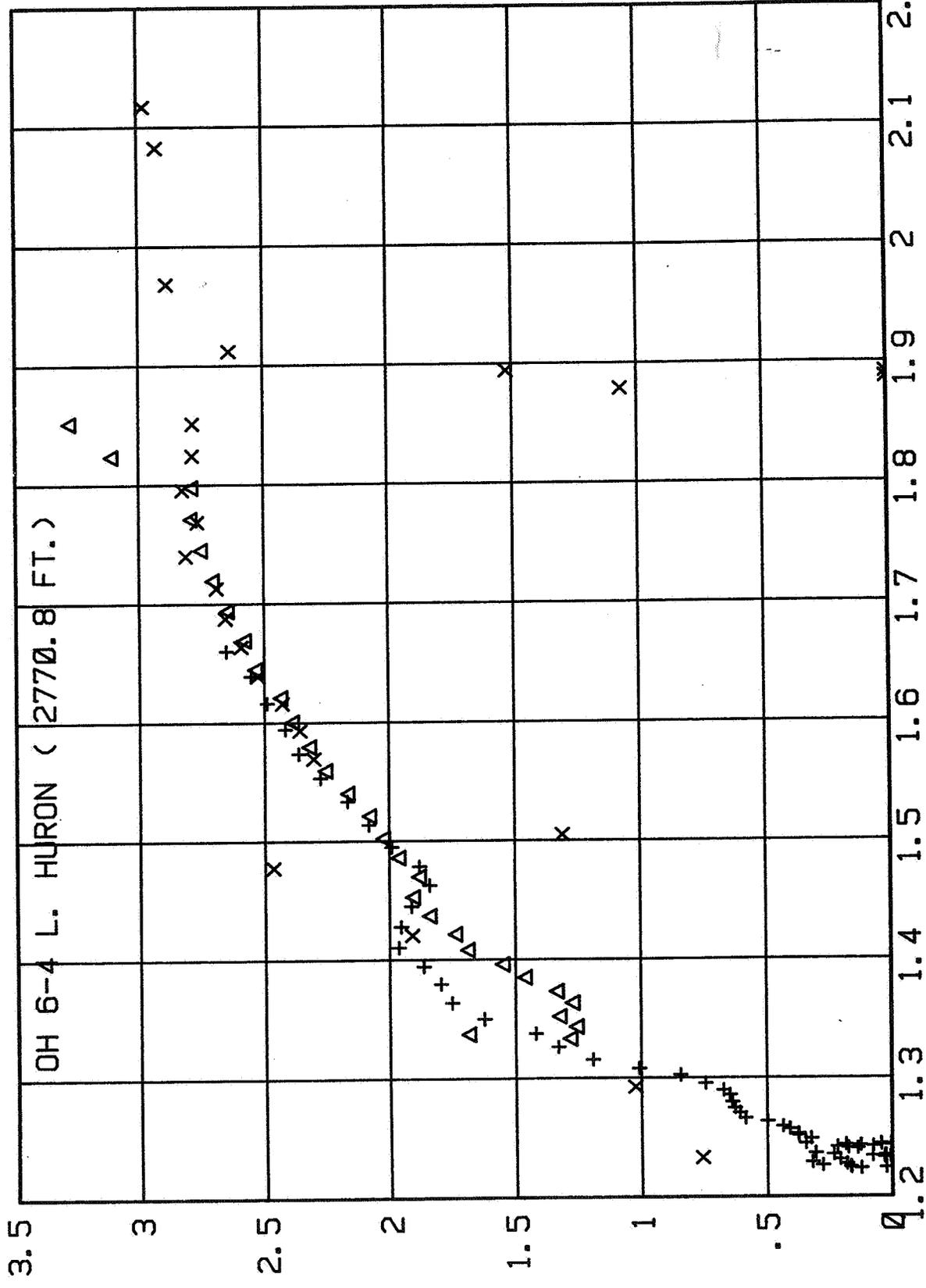




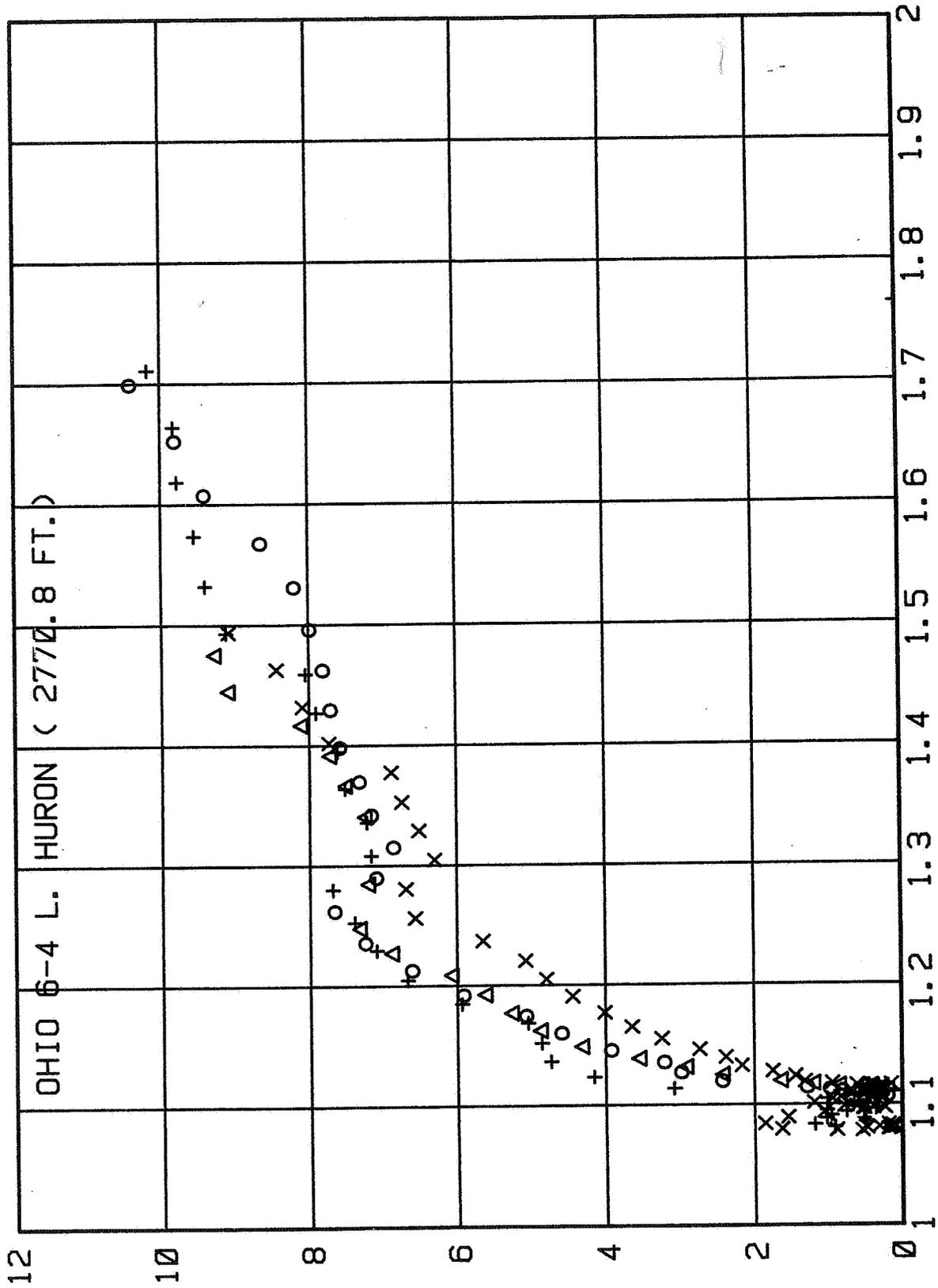




PERMEABILITY (MICRODARCIES)



PRES. DROP ACROSS PLUG (PSI) AT 3000 PSI NET CONF. PRES.



PERMEABILITY (MICRODARCIES)

