RESERVOIR ROCK PROPERTIES OF DEVONIAN SHALE FROM CORE AND LOG ANALYSIS

by

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ABSTRACT

Porosity and gas saturation in the Devonian shale are much higher than previously reported. This is based on results from special core analysis methods combined with log analyses in a conventionally cored section of an air drilled well in West Virginia. The Devonian shale formation in the eastern U.S. is an important potential tight gas producing province. As a part of research sponsored by the Gas Research Institute in shales, four wells have been recently drilled in which extensive formation evaluation data have been collected including 519 feet of conventional cores.

In this study, porosity and gas saturation were measured on 121 core samples over 150 feet of cored interval in the first well. Part of the interval was included in a subsequent production test which flowed gas. Core porosity ranged from 2 to 7 percent and core gas saturation averaged 32 percent. These results compare favorably with log analysis. Dean Stark extraction of core pieces was found to be ineffective even though extended extraction time was used. Special SEM studies show that fibrous illites are present but not affected by oven drying. CEC measurements, which relate closely to clay content from XRD, are used along with humidity drying tests to approximate the ratio of effective to total porosity. Mercury capillary pressure data which were measured on 10 core plugs, are presented to show pore size distribution. Maximum pore throat entries were found to be about 0.1 micron in radius.

In addition, results are presented on six preserved cores comparing measurements of effective gas permeability at connate water, water permeability of the brine saturated cores, and gas permeability of the dry cores, all at net overburden stress. Gas permeability at connate water is low, as expected, but relative permeability effects are puzzling.

INTRODUCTION

The Devonian shale in the Appalachian basin is considered a major potential source of gas in the United States. Core analysis in shale rocks for reservoir properties has seldom been done, since shales are generally regarded as being seal rocks with no ability to store significant free gas. Previous reports (Kalyoncu, et al, 1977; Nuhfer and Vinopal, 1978) show porosities commonly of the order of 2 to 3 percent in the Devonian shale and gas saturations that are generally less than 0.1 to 0.2 percent by bulk volume (Soeder, 1988). Gas in place associated with these low free gas contents is an order of magnitude less than is customarily produced in Devonian shale wells with long term production history (O&GJ, 1985).

This study is part of research sponsored by the Gas Research Institute (GRI) in shales. Four wells have been recently drilled in which extensive formation evaluation data have been collected including 519 feet of conventional cores in the Devonian shale. In this study, core analyses and log analyses have been conducted in a 150-foot cored interval of the Lower Huron formation of the Devonian shale from the Sterling-Jarvis No. 1143, Calhoun County, West Virginia. This well penetrated 2,294 feet of the Upper Devonian with top at 2,256 feet and total depth at 4,550 feet. The cored interval at 3,424 to 3,574 feet consists of shaly coarse siltstones, very shaly siltstones, laminated siltstones and shales, and silty shales. Mean grain size ranges from 0.01 mm for the silty shales to 0.06 mm for the shaly siltstones. Mineralogy of the cored section is shown on average in Table 1 for 16 samples, based on x-ray diffraction (XRD). Kerogen and bitumen content which can be rather high in the Devonian shale in certain areas of the Appalachian Basin, was found to be present in only trace amounts in the cored interval based on thin section examination of 16 samples and on geochemical tests.

The four-inch diameter conventional core was drilled using air-mist. Expulsion of liquids from the core by evolution and expansion of the dissolved and free gas following drilling is considered negligible. After retrieval, the core and the aluminum inner sleeve were cut into 5-foot sections, and the core was preserved. Under these conditions, the fluid saturations present in the core should represent those in the reservoir.

Porosities and gas saturations were measured on 121 core samples, using special procedures as discussed. These results are presented and then compared to log analysis, specifically modeled for the Devonian shale, for verification of both the core and log analysis methods. Results are also presented of a petrographic study using the scanning electron microscope (SEM) to determine the presence of fibrous illites and the effect of core sample drying on their morphology.

In order to investigate further the capability of the Devonian shale to store free gas, two related studies were also made. First, clay properties were determined using XRD and CEC measurements on selected core samples. From these data the ratio of effective to total porosity, ($\O_e/\O_t$), is derived. This is compared to an estimate of $\O_e/\O_t$ obtained by comparing porosities of a companion set of core plugs in which one plug was humidity dried and one was dried in a convective oven. In further study related to the amount of effective pore space available, results of mercury injection capillary pressure tests are shown.

Last, results are presented for measurements of effective gas permeabilities measured at connate water and net overburden (NOB) stress. These are compared to water permeabilities of the brine saturated cores and gas permeabilities of the dry cores.

POROSITY AND GAS SATURATION

Analyses Procedures

Porosity and fluid saturations were measured foot-by-foot on the preserved core samples through 120 feet of the 150 feet of core recovered. Prior to analyses the entire core was inspected for presence of oil and kerogen under ultraviolet light and with pentane cut on selected core chips. Where traces of oil and kerogen were found, 22 core pieces were selected for Dean Stark extraction. These were first weighed, and then bulk volume was measured by Archimedes displacement using immersion in toluene. Next, the cores were extracted with boiling toluene for up to two weeks, till no further water was collected. The core samples were then dried in a convective oven at 230°F for nearly one month, until there was no significant weight change. For the 99 foot-by-foot locations where no oil or kerogen was found, core pieces were weighed, their bulk volumes were measured as above, and then they were dried in a convective oven at 230°F as above.

For the 22 core samples extracted by the Dean Stark method the oil volume was found by subtracting the weight of the collected water from the total weight loss, with both oil and water densities presumed to be 1.0 gm/cc. For the 99 core samples simply oven dried, all weight loss was presumed to be water.

After the core samples were dried and weighed, their grain volumes were measured using helium. This permitted direct calculation of porosity (Ø), water saturation (S_w), oil saturation where present (S_o), gas saturation (S_g), and grain density (ρ_g).

RESULTS

The 22 plugs selected for Dean Stark extraction were scattered throughout the 150 foot section in 14 intervals. As shown in Table 2, for the 22 plugs extracted by Dean Stark, average S_o is 23.7 percent, average S_w is 40.7 percent, and average ambient Ø is 4.5 percent. For the 99 core plugs oven dried only, average S_w is 68.4 percent and average ambient Ø is 4.5 percent. Based on total organic carbon and pyrolysis measurements made on 15 samples (one taken each ten feet), average oil content is 0.06 percent by bulk volume. This corresponds to an average S_o of 1.3 percent. We believe, therefore, the oil content is negligible, and the Dean Stark method was ineffective in removing the water, even though an exceptionally long extraction period was used. Subsequent oven drying, however, completed the water removal. Note that if all the weight loss for the 22 Dean Stark samples is assigned to water, then average S_w is 64.4 percent. For the entire set of 121 cores, average S_w then becomes 67.6 percent, average gas saturation, S_g, is 32.4 percent, and average bulk volume of gas is 1.3 percent.

The core analyses results for Ø, S_w, S_g, bulk density, (ρ_b), and grain density (ρ_g) are presented in Figure 1 as a depth plot. This illustrates the variability of the data. Part of the variation in S_w reflects variation in Ø. Since the reservoir property of more economic interest is the bulk volume of gas present, Figure 2 presents BVG as a function of Ø. This shows a good correlation, with the best fit Reduced Major Axis, (RMA), dashed line as shown. This style of plot is similar to that shown by Heseldin (1974) and later adapted by Alger, et al (1989) for integrating Ø, BVG, and capillary pressure data. This plot shows:

1. As Ø becomes higher, BVG grows correspondingly. Conversely, for Ø < 3 percent, on average there is no free gas present.
2. Bulk volume of water (BVW) shows very little change over the range of porosities present.

LOG ANALYSIS

Reliable log analysis in the low porosity, Devonian shale formation depends critically upon determining accurate fractions of quartz and clay, and especially of pyrite and kerogen. Most of the wells in the Appalachian Basin are drilled with air, which limits the number of possible log measurements. A typical log suite includes the dual induction, photoelectric cross section index, density, sidewall neutron, and spectral gamma ray logs, as shown in Figure 3 through the cored section of interest in Jarvis No. 1143. Other log measurements used for analysis are the borehole television, mud log, and temperature survey.

The log analysis model used in this study is specific to the Devonian shale and is based on integration of log responses with rock properties obtained from quantitative petrography and core analyses on nearly 200 rotary sidewall cores taken in 19 wells in the Devonian shale in the Appalachian Basin in the past four years. The evolution of this log analysis system has been reported by ResTech (1985), Campbell and Truman (1986), and Campbell (1987). With the recent acquisition of 519 feet of conventional core in four wells as mentioned earlier, the log analysis model is being further refined.

The log analyses results in Jarvis No. 1143 are shown in Figure 4, compared with core analyses. A comparison of Ø, BVW, and BVG is shown as well as log-calculated mineralogy. In this well, hole conditions were especially poor due to some special well tests run before logging, and this is responsible for much of the scatter in the log results. In Table 3, the average values of Ø, BVW and BVG from log and core analyses are compared, along with standard deviation between the two methods. Note that BVW from log and core data compare closely. Average core analyses Ø and BVG are higher than log analyses by 0.7 percent bulk volume. However, core Ø was measured at ambient stress, and 0.7 percent is about the amount of reduction in both Ø and BVG that one should expect when restoring the cores to overburden stress.

The lower part of the cored interval from 3,552 to 3,574 was perforated and included in a completion test with the overall perforated intervals of 3,345-3,380 and 3,552-3,680. After a propped foam frac was performed, the well was tested at a gas rate of 22 MCF/D. About 25 percent of this came from the lower cored interval, which includes some of the higher levels of BVG based on log and core analyses.

FIBROUS ILLITES

Recent work such as that by Pallat, et al (1984) and de Waal, et al (1988), has demonstrated the importance of proper core handling in obtaining representative reservoir rock properties when fibrous illites are present. We were concerned that fibrous illites could be a significant factor in the Devonian shale. In reservoir rocks with moderate permeabilities, a conclusive way to determine if fibrous illites are significant is to compare permeability of the rock with the illites in their undisturbed morphology with the permeability after the illites have been altered (e.g., by routine core fluids extraction). In Devonian shale, where permeability typically is in the microdarcy range or less, these kind of comparative flow tests are not practical.

As an alternative, preserved core samples from three intervals were analyzed by scanning electron microscopy (SEM). In each interval, one sample was prepared by flash-freezing in liquid nitrogen and then vacuum drying to preserve the illite structure. A companion sample was dried in a convection oven at 230°F. Two intervals showed large amounts of detrital clay, but very little authigenic clay development. One interval at 3,530 feet showed minor amounts of detrital clay, but larger amounts of authigenic clay, including pore filling, fibrous illites. Figure 5 shows a photomicrograph of the freeze dried sample at 3,530 feet, at 2400X magnification, with well developed fibrous illites in their original morphology. Figure 6 shows a photomicrograph of the convection oven dried companion sample, at 2400X magnification, which also shows well developed, delicate fibrous illites, both large and small. Based on this study, there are no noticeable effects on the clay morphology introduced by the process of drying these Devonian shale core samples in a convection oven.

EFFECTIVE POROSITY

Two related studies were conducted to determine the capability of the Devonian shale to store free gas, to augment the direct measurements of free gas previously discussed. The ability of a shaly reservoir rock to store free gas is linked in part to the amount of effective porosity, ($Ø_e$), available as opposed to porosity occupied by bound water, ($Ø_b$). The ratio of $Ø_e/Ø_t$, where total porosity, ($Ø_t$), is the sum of $Ø_e$ and $Ø_b$, is related to the clay present and the clay properties. The ratio of $Ø_e/Ø_t$ and free gas storage capability can also be inferred from special mercury injection capillary pressure tests.

CLAY PROPERTIES

As shown in Table 1 average clay content is 36 percent based on x-ray diffraction on 16 samples, and the clay is dominantly illite. CEC was measured on these same samples. Figure 7 shows there is a good relation of CEC to total amount of clay present. The best fit RMA line shown has been constrained to intercept the origin. Average CEC is 3.64 meq/100 gm.

From $\varnothing_t$ and ρ_g of adjacent core samples, CEC can be converted to Q_v (meq/cc pore volume) with

$$Q_v = CEC\ (1 - \varnothing_t)\rho_g / 100\ \varnothing_t \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (1)$$

Applying Equation (1), average Q_v is 1.96. This can then be used to estimate the ratio of $\varnothing_e/\varnothing_t$ using the method of Hill, et al (1979)

$$\varnothing_e / \varnothing_t = \left(1 - \left(0.084\ C_o^{-1/2} + 0.22\right) Q_v / \rho_{cbw}\right) \ldots\ldots\ldots\ldots \quad (2)$$

where C_o = free water salinity, equiv/l, and
ρ_{cbw} = density of clay bound water, gm/cc

From Equation (2), with $C_o = 0.88$ and $\rho_{cbw} = 1.0$, average value of $\varnothing_e/\varnothing_t$ is 0.39. However, recent work by Truman et al (1989) shows that in rocks that are predominantly shale, the method of Hill et al (1979) appears to overestimate bound water, $\varnothing_b$, by a factor of about three. Assuming the same correction applies, corrected $\varnothing_e/\varnothing_t = 0.80$. This is a very approximate estimate but it does imply that a significant part of the total porosity is available to store gas.

CAPILLARY PRESSURE TESTS

Mercury capillary pressure tests were run to 50,000 psi on one inch diameter core plugs from ten depth locations. These core plugs were dried at 220°F in a vacuum oven for two days. Bulk volumes were measured by mercury immersion, and grain volumes were measured with helium. Porosities range from 0.4 to 5.4 percent. For the depth of 3,431 feet two adjacent plugs were taken. One plug was dried as described while the second was dried at 145°F and 45 percent relative humidity to retain a bound water layer, as described by Bush and Jenkins (1970). Porosity for the humidity dried core plug is 2.4 percent and for the convection oven dried core plug is 2.9 percent. The ratio of $\varnothing_e/\varnothing_t$ can be estimated for the core plugs at 3,431 feet by comparing the humidity dried porosity to the convection oven dried porosity, or $\varnothing_e/\varnothing_t = 2.4/2.9 = 0.83$. This compares closely to the 0.80 corrected value obtained using the average CEC value.

The mercury capillary pressure results for the two companion plugs at 3,431 feet are shown in Figure 8. The mercury saturations are normalized to 100 percent at 50,000 psi. Note that there is very little difference in the characteristic shape of the two curves, suggesting that the bound water layer is rather uniformly distributed among the pore throats and pore bodies. The entry pressure of each core from the 10 intervals sampled is similar to that shown in Figure 8. However, at 50 percent mercury entry, the pressure level varies from about 3,000 to 30,000 psi. Figure 9 shows the pore throat radii present in the convection oven dried core at 3,431 feet, and Figure 10 shows the pore throat distribution for the same core.

These combined results show maximum pore throat entry is about 0.3 micron and most of the pore volume available for gas storage has associated pore throat radii in the range of 0.01 to 0.1 microns. This corresponds to mercury capillary pressures of the order of 1,000 to 10,000 psi, which corresponds to reservoir gas-brine capillary pressures of 160 to 1,600 psi. This is also equivalent to distances above free water level of 400 to 4,000 feet. Since the in place gas saturations measured directly on the cores are in the range of 0 to 70 percent (Figure 2) and average 32.4 percent, this implies reservoir capillary pressures are present on the order of 1,000 psi. Note that these capillary pressure data were measured with the cores at ambient stresses and should be corrected to reflect overburden stress conditions.

PERMEABILITY

Effective permeability to gas is probably the most important property needed in reservoir evaluation of the Devonian shale, since this directly relates to well productivity. Although natural fractures or micro-cracks may play a dominant role in well productivity, permeability of the unfractured rock matrix is important. Further, it is important to measure effective gas permeabilities at native state fluid saturations and at reservoir pore pressure and overburden stress.

Gas permeabilities were measured on six core plugs 1 1/2 inches in diameter, preserved at their native state water saturations. Gas permeabilities were measured using helium in a pulsed permeameter with a pore pressure of 2,000 psi and a net overburden pressure of 0.75 times the core sample depth applied to the core hydrostatically. Results are shown in Table 4. Two plugs have permeabilities below 0.01 microdarcies, the lower limit of the apparatus. These plugs also have the lowest porosity values. Based on petrographic study of end trims all of the plugs are classified by Davies (1989) with the rock type as shown in Table 4, where A is a shaly sandstone/siltstone, B is a very shaly sandstone/siltstone, and C is a burrowed silty shale. Porosities shown were later determined by brine saturation and subsequent drying in a vacuum oven at 140°F. Helium porosities were then measured at both ambient and net overburden stress, and the brine porosities shown in Table 4 were adjusted to overburden stress conditions as were the water saturations that are shown.

Following the measurement of effective gas permeabilities the cores were saturated with brine and 0.4 to 0.6 pore volume of brine flowed through the cores for one to two days. From the latter part of this flow period, data were used to calculate steady state brine permeabilities. These are shown in Table 4. Note that k_w is less than k_g (at the intermediate S_w values shown) by a factor of 5 to 658. These results show that when replacing the free gas space pore network with water, the permeability of that pore network to water is many times less than to gas. This is surprising and requires further study.

The results of gas permeability measurements on the dry core plugs, conducted with helium at 2,000 psi pore pressure and net overburden stress, are also shown in Table 4. These dry core gas permeabilities are higher than k_g (at intermediate S_w) by a factor of 4 to 377, and are higher than k_w at $S_w = 100$ percent by factors of 200 to 10,000. In some low permeability rocks there is a fairly good relationship of k_w to dry core gas permeability (Luffel, et al, 1989), and a good relationship of relative permeability to S_w (ResTech, 1989). In the core analyses results from the Devonian shale in this study, coherent relationships are not yet evident.

CONCLUSIONS

1. Porosity and gas saturation in the Devonian shale are much higher than previously reported, based on results from special core analyses.

2. Log analyses in this complex rock system are in reasonably good agreement with core analyses.
3. Extraction of fluids from cores using the Dean Stark boiling toluene method was ineffective, even though extended extraction time was used.
4. High effective porosities are present and available to store free gas, based on amounts of clay and CEC measured, special humidity-dried core tests, and mercury capillary pressure tests.
5. Effective gas permeabilities at connate water, brine permeabilities, and gas permeabilities of the dry cores have been measured on six core plugs with puzzling results. No satisfactory relationships are yet evident.

ACKNOWLEDGEMENTS

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TABLE 1

X-RAY DIFFRACTION ANALYSIS

MINERAL	AVE WT %
Quartz	47
K-Feldspar	1
Plagioclase	6
Pyrite	3
Ankerite	1
Calcite	4
Siderite	2
Illite	31
Kaolinite	1
Chlorite	4
	100

TABLE 2

COMPARISON OF POROSITIES AND FLUID SATURATIONS

Extraction Treatment	Number Samples	Average Ø, %	Average S_w, %	Average S_o, %
Dean Stark	22	4.5	40.7	23.7
Oven Dry	99	4.5	68.4	
Total	121	4.5	67.6	

TABLE 3

COMPARISON OF LOG AND CORE ANALYSES

	CORE ANALYSES %	LOG ANALYSES %	CORE/LOG STANDARD DEVIATION %
Ø	4.6	3.9	0.9
BVW	3.1	3.2	0.7
BVG	1.4	0.7	0.8

TABLE 4

SUMMARY OF PERMEABILITIES

		STRESS	NATIVE STATE		Sw = 100%	Sw = 0%
CORE DEPTH	ROCK TYPE	Ø %	Sw %	Kg MICRO D	Kw MICRO D	Kg MICRO D
3455.2	B	1.4	68.8	<0.01	0.0021	0.44
3515.2	C	3.7	55.4	0.04	0.0024	3.02
3534.5	C	6.1	67.4	0.04	0.0015	15.07
3551.3	A	1.3	82.0	<0.01	0.00018	0.08
3558.3	B	4.9	48.5	11.84	0.018	48.47
3564.5	B	3.4	92.6	5.11	N.A.	37.04

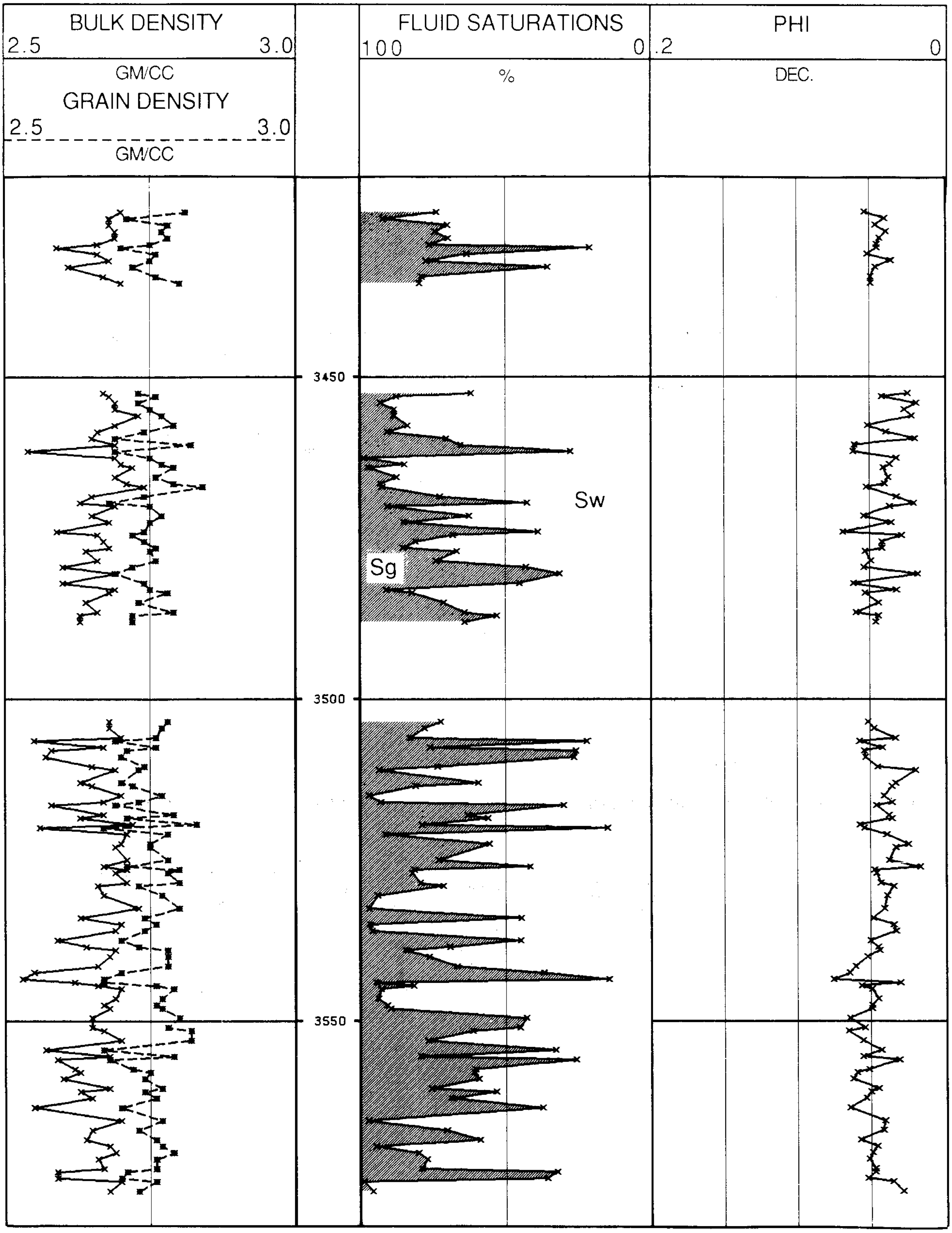

Figure 1. Core analyses results for Sterling - Jarvis No. 1143, Calhoun County, West Virginia.

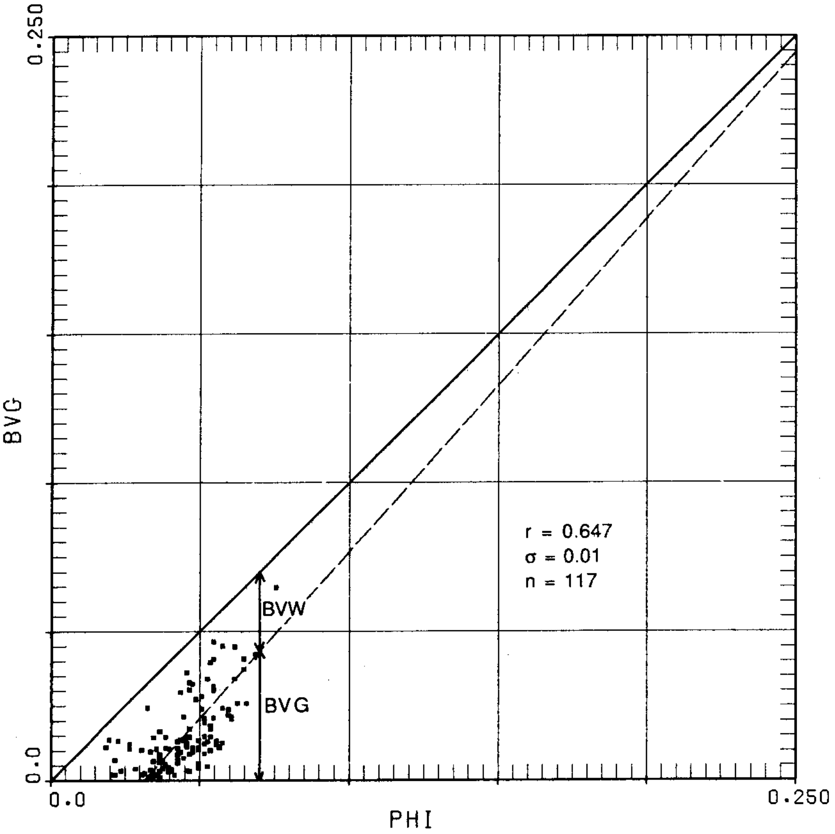

Figure 2. Relationship of bulk volume gas to porosity from core analyses.

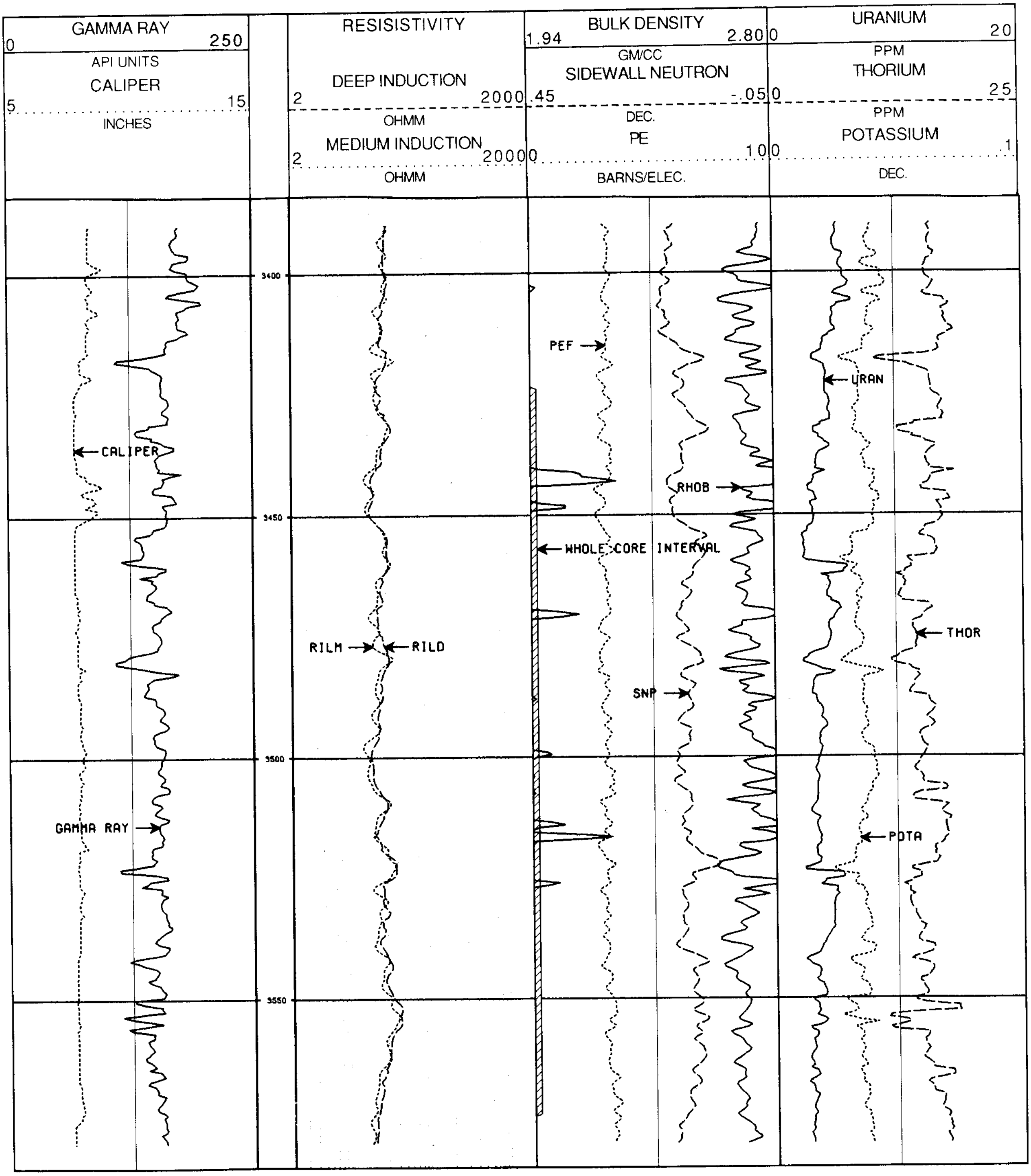

Figure 3. Open hole logs obtained through cored section of Sterling - Jarvis No. 1143, Calhoun County, West Virginia.

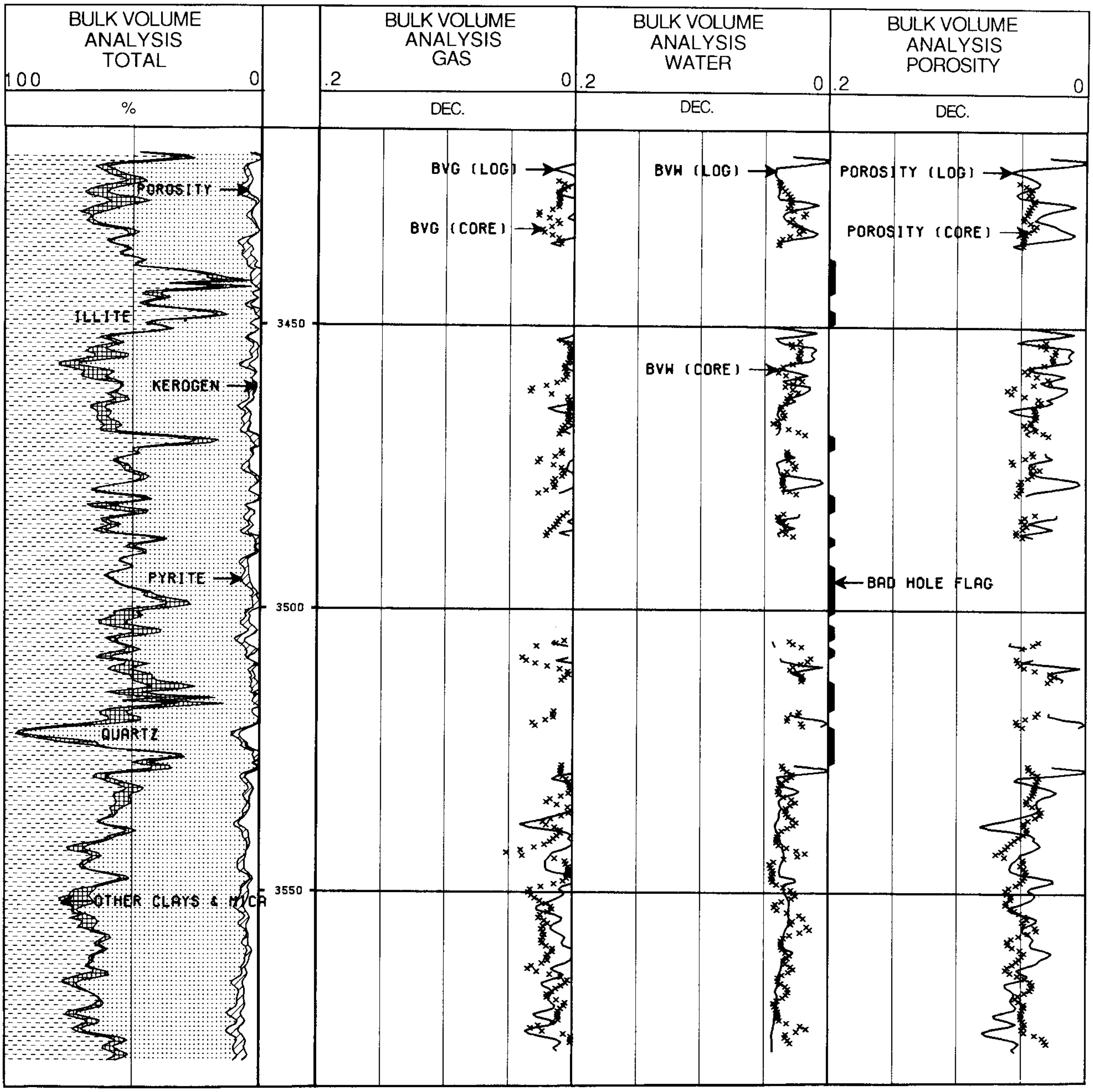

Figure 4. Comparison of log and core analyses .

Figure 5. Photomicrograph of freeze dried core sample from 3,530 feet, showing well developed fibrous illites (2,400 X). Long dimension of photo is 50 microns.

Figure 6. Photomicrograph of convection oven dried core sample from 3,530 feet, showing well developed fibrous illites (2,400 X). Long dimension of photo is 50 microns.

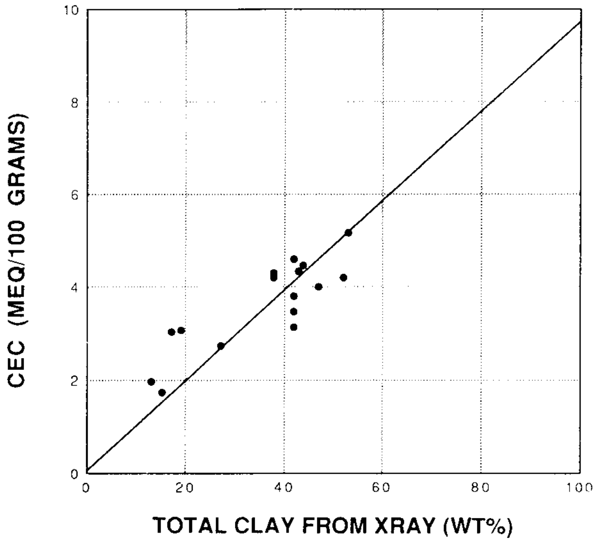

Figure 7. Relationship of cation exchange capacity to weight percent of clay from x-ray diffraction.

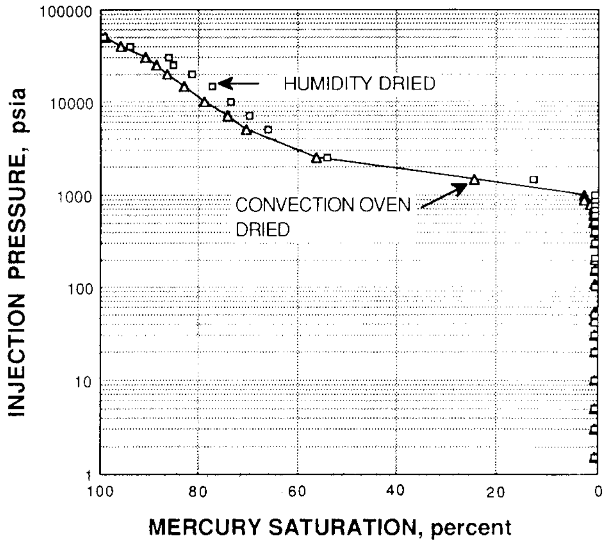

Figure 8. Comparison of mercury capillary pressure results on two companion samples from 3,431 feet.

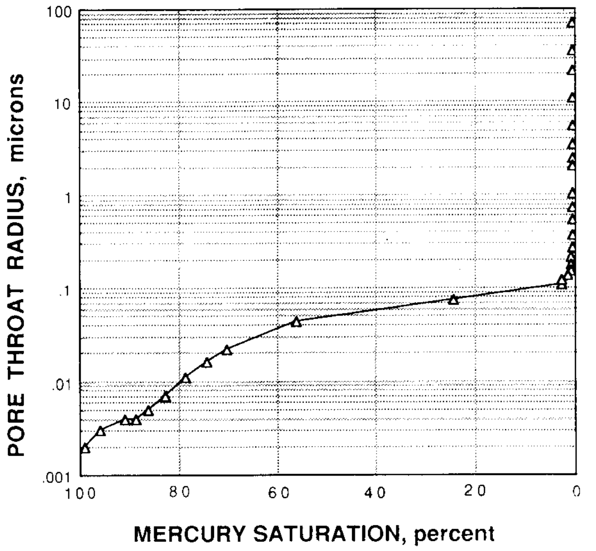

Figure 9. Pore throat radius from mercury capillary pressure results for convection oven dried sample at 3,431 feet.

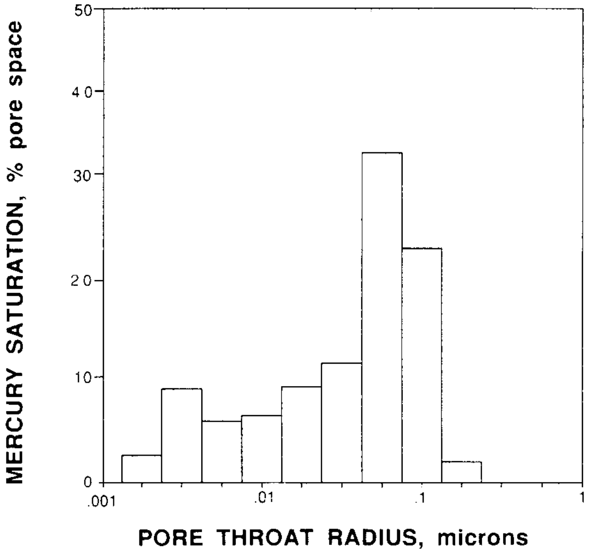

Figure 10. Pore throat radius histogram for convection oven dried sample at 3,431 feet.