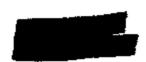
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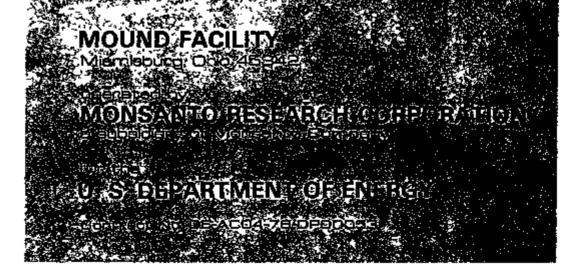
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# PRESSURE-CORING OF THE GAS-BEARING DEVONIAN BLACK SHALES

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### ABSTRACT

A Christensen model pressure-retaining core barrel is being used in the drilling of two EGSP research wells. This effort supported by two other techniques is designed to evaluate the most effective technique to provide meaningful resource assessments.

One well has been successfully drilled and the accumulated samples are currently being evaluated. The second well is currently being drilled.

## INTRODUCTION

Resource assessment values used to estimate gas in place and recoverable reserves for the gas bearing Devonian shales are still tentative. This is primarily due to unknowns regarding the present methods of sample collection and testing.

At present, samples are obtained for the purpose of characterization by the recovery of oriented core. The samples are removed from the core barrel, visually examined and described at the well site, then sealed in containers and sent back to the laboratory for analysis. The selection of these resource samples is made on the basis of a uniform interval  $\{\sim 5 \text{ to } 10 \text{ feet}\}$  through the shale zone.

Once the samples are received at the laboratory, they are allowed to build up pressure for a period of six weeks or longer, the containers are leak checked to ensure their integrity and only those that are leak tight are processed through the gas volume analysis.

The pressure and volume of the gas in the can is determined and the gas composition is analyzed by gas chromatography. The shale sample is then macerated and the volume of gas remaining in the shale matrix is determined. The sum of these two values represents the total volume of gas present in the shale after it is placed in the sealed container.

This method has several shortcomings:

- Sample selection is made on the basis of uniform core interval rather than from high potential zones. This can result in the average gas content per well being biased low.
- 2. Gas lost prior to the placing the samples in the containers cannot be estimated.
- Release rate of the gas from the sample is not obtained and the effect of overpressure on the gas release cannot be assessed.

<sup>\*</sup>Mound Facility is operated for the U. S. Department of Energy by Monsanto Research Corporation (Contract No. DE-ACO4-76-DP00053).

To address the shortcomings an experiment was initiated at two research wells. This experiment, cooperatively funded by the Gas Research Institute (GRI), involved the use of several different techniques to evaluate resource in place and analytical procedures. The first technique is the one currently used to assess gas. The second technique involved the use of a continuous mud log to choose samples containing high gas concentrations. These samples will be analyzed for gas content and composition by using an offgassing technique to be initiated immediately upon sample recovery. The third technique involves the use of the pressure retaining core barrel to recover samples essentially under insitu conditions and to monitor total gas release.

The experiment is currently in progress and only preliminary data are available at this time.

### RESEARCH WELL LOCATIONS

At the request of the METC Project Office, Erie County, Pennsylvania, was identified as a prime site for the location of at least one research well for the EGSP program. Based on geologic considerations, several areas within the county were selected for possible sites. To accompdate cost-sharing co-operators, one site shifted just across the state line into Ohio. The general locations of the two drilling sites along with the locations of nearby oil and gas wells used for control are shown in Figure 1.

A generalized stratigraphic column (Figure 2) summarizes the Devonian section for the target areas. The rocks are marine deposits with a general increase in the terrigenous clastic influx, and concomitant increase in grain size, ascending the column. These clasifics were derived from the Catskill delta complex as it prograded westward from eastern Pennsylvania and New York, nearing but never reaching Erie County. (1,2,3)

In the target area, the Lower Devonian is represented mainly by the Oriskany sandstone of shallow marine origin with a thin limestone, possibly Helderberg or Keyser equivalent, sometimes beneath the Oriskany. The Oriskany sandstone is overlain by a thick sequence of the Middle Devonian Omondaga limestone, also a shallow marine deposit. The Onondaga is in turn overlain by the Hamilton formation, mainly shales and siltstones, with the Marcellus black shale at its base. A thin layer of the Middle Devonian Tully limestone is generally present in Erie County, above the Hamilton formation. It is the last marine limestone deposited in the area. The Upper Devonian in Erie County, Pennsylvania, is represented by several formations consisting of alternating gray and black shales, siltstones, fine sandstones, and minor coarse sandstones. Almost all of these being occasionally calcareous (cement) and of shallow marine origin. Younger Devonian, and later Mississippian and Pennsylvanian age rocks, are present elsewhere in Erie County. These will not be encountered in the target areas.

During Devonian times, Eric County, Pennsylvania, was under the influence of an epicontinental sea that had a configuration roughly equivalent to the present day Appalachian Basin. Water depth was relatively shallow and at times the area was uplifted above sea level, as documented by several prominent unconformaties. Subaerial deposits resulting from this uplift are unknown, rather, erosional processes removed the exposed marine deposits. (4-9)

The clastic influx from the Catskill delta during the Upper Devonian produced a slight east-to-west thinning of these sediments of about one to two feet per mile across Erie County. (10,11) The rocks are essentially flat lying, with a gentle dip of  $1/2^{\circ}$  or less to the south-southeast. (1,12)

At least three periods of black shale formation occurred in the target areas during Devonian times. These are represented by the Marcellus, Rhinestreet, and Dunkirk black shale facies in ascending order (oldest to youngest, Figure 2). A representative gamma-ray log from a well in the target areas is shown in Figure 3. The log shows, in general, that the black shale facies consist of a basal, massive unit and an upper, interbedded (black shale-gray shale-siltstone) unit. This second aspect is especially well illustrated in the Dunkirk interval, less so in the Rhinestreet, and is virtually absent in the Marcellus. This upper, interbedded unit may be primarily responsible for the gas production from the Devonian black shales, (13) Historically, the Dunkirk is the main gas-producing black shale in Erie County, Pennsylvania. (14)

Figures 4, 5, and 6 show isopachs of the net thickness of radioactive black shale for the three black shale intervals; Dunkirk, Rhinestreet, and Marcellus, respectively. The mapped thicknesses include the basal, massive unit, and each individual black shale bed in the upper, interbedded unit. [14.15.16] As can be seen (Figures 4 and 5) both the Dunkirk and Rhinestreet are fairly thick (>50 feet) over almost the entire country. Figure 6 shows the Marcellus to be relatively thin (<25 feet) everywhere in the county. Although it will be sampled in the wells, the Marcellus will not be a primary production target. Figure 7 is a composite isopach of all three intervals. [17] It shows four areas of maximum total thickness in Eric County. The westernmost three of these were chosen as the prime targets.

In two of these areas, the westernmost and north-centralmost, cooperators for wells were found. These sites are indicated on Figure 1.

Drilling is to be completed to the top of the Middle Devonian Onondaga limestone. This will allow not only for sampling and testing of all three of the black shale intervals, but also for obtaining an excellent widespread correlatable stratigraphic marker horizon; i.e., the Onondaga limestone. Figure 8 indicates a drilling depth map to the top of the Onondaga limestone. (18) From this map, the projected total depths for both research wells is \$500 feet or less.

# ANALYTICAL TECHNIQUES

The analytical techniques being used for the two wells are as stated earlier:

- 1. Current Technique
- Controlled Offgassing
- Pressure Core Barrel/Controlled Offgassing.

The current technique was described earlier and will not be reviewed.

# Controlled Offgassing

For both wells, mud is being used as the drilling fluid. The use of mud as the drilling fluid provided the opportunity to utilize a continuous mud log during the drilling and coring operation. The data accumulated by the mud log operation is being used to select samples for controlled offgassing experiments, that is samples are being selected from the gray shales where relative high hydrocarbon gas concentrations are being observed as well as from the thick black shale zones.

The samples, once identified, are placed in containers as soon as possible after the recovery of the core.

At select time intervals, these samples are tested and a measured volume of gas is removed from the container and analyzed by gas chromatography, then all the shale gas is purged from the container which is restored to atmospheric pressure. This process continues until the hydrocarbon gases are essentially depleted. The samples will then be macerated and gas remaining in the matrix will be determined.

At the time of this writing, six weeks after core recovery, the degassing operation is still continuing and curves of various shapes are being obtained. When description ceases, accurate determinations of shale volumes and gas released will be made and the data will be compared to that obtained by the other techniques.

# Pressure Core Barrel/Controlled Offgassing

The pressure core barrel is not a new technique and has been in use for quite some time. In the late 1930's the Carter Oil Company designed and operated a pressure-retaining core barrel device, basically by inserting a third "barrel" between the inner and outer core barrels for use in the determination of residual oil saturation. (19) This barrel did not retain full formation pressure, but further redesigns and modifications by Exxon during the 1960's produced a larger diameter barrel capable of retaining full formation pressure. (20) Currently, two companies, Christensen and DOMDCO, are licensed to operate and to experiment with the pressure core barrel. (20,21) Recent work on the barrel improved the reliability and percentage recovery of the pressure core barrel. (21) It is now generally agreed that the pressure core barrel provides the best opportunity of determining the in-place fluid saturations of reservior rocks. (21) For this project, Christensen provided the pressure retaining core barrels.

# OPERATION OF THE PRESSURE CORE BARREL

Mechanically, pressure coring differs from conventional coring only in that the pressure core barrel is sealed at reservoir pressure after the core is cut and prior to starting out of the hole. Therefore, the pressure core barrel requires special operating procedures and techniques for handling and analyzing the cores.

A Christensen model pressure core barrel, Figure 9, was used in our field operations for the research

wells. It is capable of recovering a 10 foot long by 2-5/8 inch diameter core sample; the overall length of the barrel itself is 23 feet with a maximum outside diameter of 6 inches at the ball valve housing. The pressure core barrel is capable of operating in pressures up to 5,000 psi and temperatures up to 150°C.

The basic pressure retaining mechanisms are a lower ball valve and an upper mechanical pressure seal. How the pressure core barrel works is shown schematically in Figures 10 through 12. After the 10 feet of core has been cut and broken off, the entire pressure core barrel assembly is lifted about 2 feet off the bottom of the hole (Figure 11). Then a small steel ball is pumped down the drill stem and seats in a pistom in the upper assembly. This trips a slip-joint release mechanism and frees the outer barrel, allowing it to slide down about 19 inches relative to the inner barrel. This movement closes the upper pressure seal and rotates and closes the lower ball valve, trapping the core at reservoir pressure (Figure 12). The upper assembly also contains a nitrogen chamber that can aid in pressure maintenance if necessary. The downward movement of the outer core barrel also uncovers mud ports in the top of the core barrel, permitting mud circulation and allowing the string to drain as the core barrel is brought to the surface with the core samples at reservoir pressure.

# CURRENT STATUS

At the Ashtabula County, Ohio site, two pressure cores were attempted and both were successfully recovered at reservoir pressure. Only six feet of the deeper core were recovered. The first core was recovered in the interbedded, primarily gray, Dunkirk shale immediately above the massive dark shale zone, the other core was recovered in the massiva, dark Rhinestreet shale interval. While still frozen, some of the one foot segments were sealed with hard rubber caps directly in the core-barrel liner and others were quickly extruded from the liner into air-tight containers and sealed.

The gas in the sealed core segments was determined by a slightly modified USBM method. The containers were connected by rubber tubing to a water-filled, 1,000 ml gas buret and the initial volumes of gas released were determined by water displacement. Thawing of the core was begun immediately after connection to the buret. After two and a half days, the rate of gas release diminished to a few ml per day. Then the core was quickly removed from the liner and transferred to a gas-tight container provided with a silicone rubber septum. Periodically, when a measured volume of gas was removed for analysis by gas chromatography, all the shale gas was purged from the can and the process repeated. At the time of writing this paper, six weeks after core recovery, the degassing is still proceeding.

A typical gas-release profile from one of these experiments is shown in Figure 13. It shows an initial surge of gas into the buret, but this is not shale gas--it is merely the expansion of the air trapped under the rubber core caps and reduced to smaller volume by the dry-ice temperature. After this initial steep slope, the true shale-gas begins to appear. The wavy appearance of this portion of the curve of gas volume vs. time (i.e., the square root of time in minutes) is a reflection of the diurnal changes in temperature and pressure in the well-site trailer. The smoother, more widely spaced data points are from samples taken from the sealed containers used in the second half of the desorption measurements.

Those frozen samples that were placed directly in the sealed metal containers were allowed to thaw at room temperature in the container. Samples were withdrawn periodically as described above and the incremental gas volume released was measured at regular intervals. A gas described profile for one of these samples is shown in Figure 14. After six weeks, these samples are also still actively releasing gas, and will continue to be sampled until description virtually ceases.

Preliminary (incomplete at the time of preparation of this paper) results show that the gas released by the ten samples of primarily gray Dunkirk shale at the site of the Ashtabula County well varies from 29,000 to 178,000 ppm, with a mean value of 97,000 ppm. In contrast, the massive black Rhinestreet shale samples recovered under pressure in the pressure core barrel have released from 836,700 ppm to 1,089,000 ppm with a mean of 905,500 ppm. In other words, incomplete results from the pressure core samples at the Ashtabula site show that the gray shale above the massive black Dunkirk zone averages 0.042 cm<sup>3</sup> of hydrocarbon gas/gram of rock, or slightly more than one cubic foot per ton; the massive Rhinestreet, 0.39 cm<sup>3</sup> hydrocarbon gas/gram or about twelve cubic feet per ton.

When the desorption ceases, or virtually so, more accurate determinations of shale volumes and gas released will be forthcoming, and comparisons will be made between various methods of estimating gas in place. Another interesting set of data will also come from the pressure core barrel samples. First, the degassing profiles, i.e., their unusual shapes ranging from simple logarithimic to sigmo-idal, should reveal something of the nature of the desorption process or of the rock matrix itself.

Secondly, the preliminary observation that the ratios of individual hydrocarbons change as the degassing proceeds should be of interest to those modelling the degaseing mathematically. For example, in many of the samples, the methane-to-propane ratio of the first gas released is in the range of 20 to 30 and has decreased by as much as one-half to date. However, in one sample, the methane-to-propane ratio of the released gas initially was 60 but currently is 11.

This demonstration of the pressure core barrel will provide the data from which the various methods of estimating gas-in-place in the shales can be evaluated. Initial results are very interesting; and no doubt will contribute significantly to our knowledge of the resource available in the Eastern Gas Shale.

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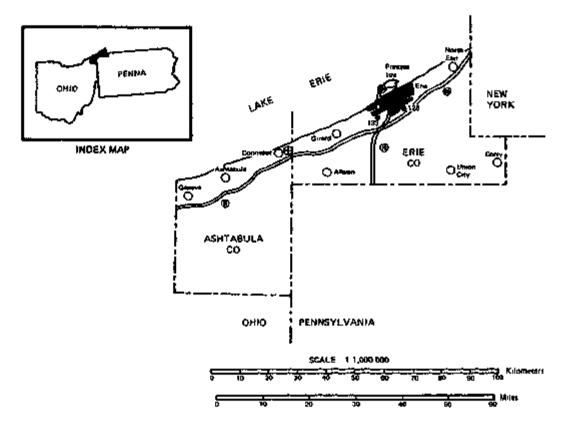


FIGURE 1 Location of EGSP wells (\*) to be deilled and cored by Mound Facility.

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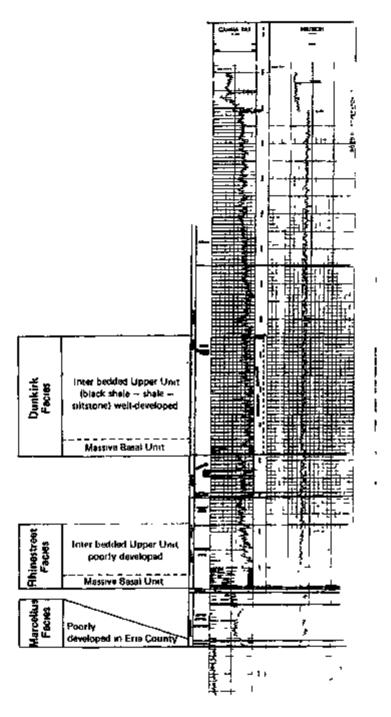


FIGURE 3: Geophysical well log from hole A-325, Ene County, Pennsylvania

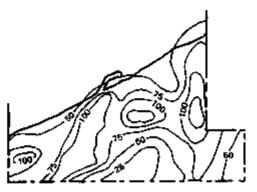


FIGURE 4:

Net Feet Radioactive Black Shale in Dunkirk Facies, Erie County,

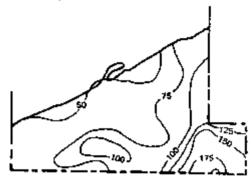
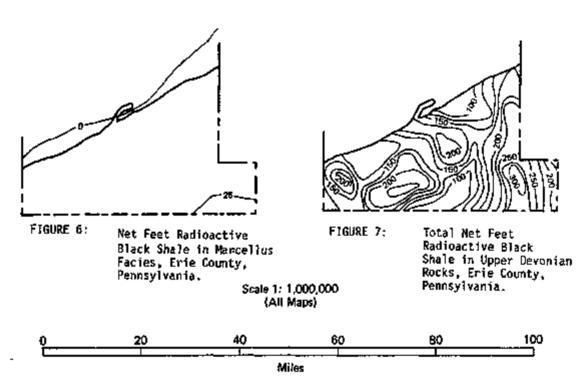


FIGURE 5:

Net Feet Radioactive Black Shale in Rhinestreet Facies, Erie County, Pennsylvania



Contour Interval: 25 ft. (All Maps)

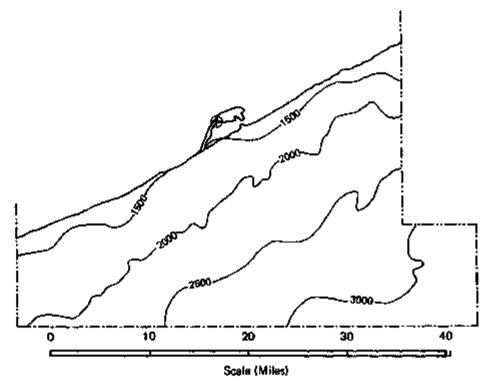
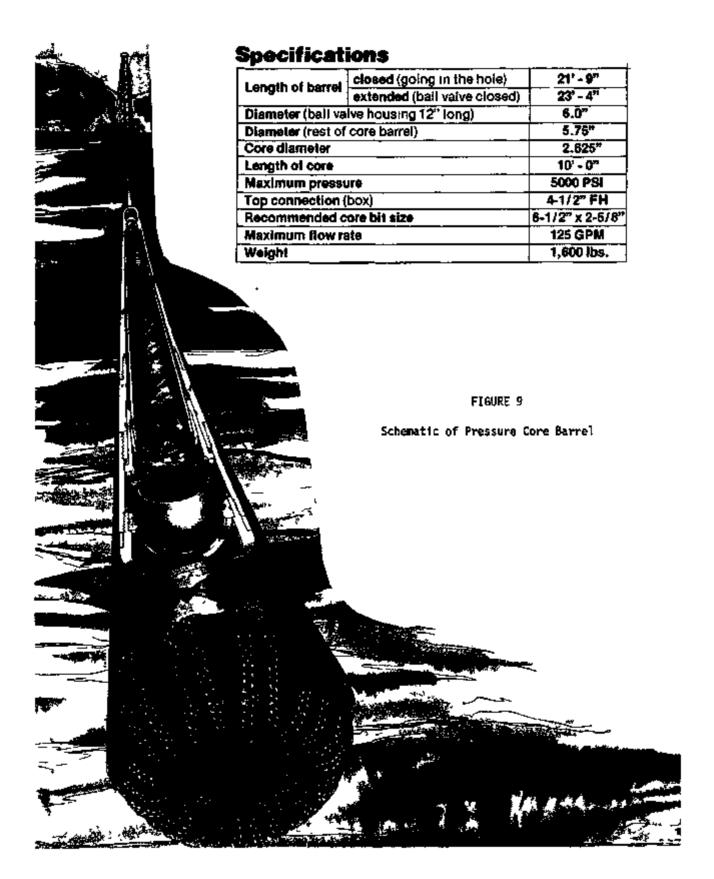


FIGURE 8: Drilling Depth to the Top of the Onondaga Limestone, Middle Devonian, Erie County, Pennsylvania. Contour Interval = 500 ft.



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FIGURES 10, 11, and 12
Operation of Pressure Core Barrel

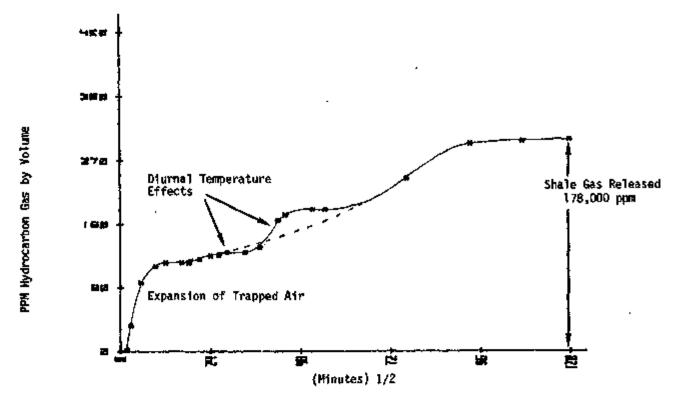


FIGURE 13: Gas Released from Pressure Core Barrel Sample (Barrel #1 - Section #1)
Gray Shale - Dunkirk Member
A-B Modified U.S.B.M. Method
B-C Degassing in Sealed Container

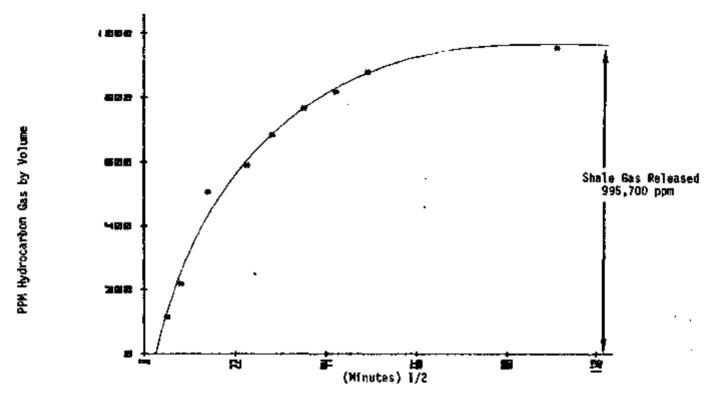


FIGURE 14: Gas Released from Pressure Core Barrel Sample (Barrel #2, Section #2) Black Shale-Rhinestreet Member