UGR File #C051/77Q1
Feb. 15-Mar. 31, 1977
Mound Laboratory

MLM-ML-77-43-0002
MLM-EGSP-TPR-Q-001

PHYSICAL AND CHEMICAL CHARACTERIZATION OF DEVONIAN GAS SHALE

QUARTERLY STATUS REPORT (FEBRUARY 15-MARCH 31, 1977)

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UNITED STATES ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

U. S. Government Contract No. EY-76-C-04-0053

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STATUS SUMMARY

Notification of funding was received in mid-February. Since that time, the work effort has been devoted to phasing in a complete shale processing plan. Subcontract details have been finalized, and an integrated effort has been initiated.

Sample analyses have begun in the areas of material balance assays, pulsed NMR, SEM, shale off-gassing, and hydrocarbon evaluation. Data in each of these areas are reported. The mechanical testing system is operational, and we are awaiting the delivery of associated test equipment before testing is initiated. It is anticipated that testing will be in progress by mid-April.

LIST OF CONTRIBUTORS

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A. PROCESSING PLANS

Equipment acquisition and setup is essentially on schedule. All of the equipment necessary to provide for a continuous flow of samples through the various analytical procedures should be available in April. No significant program delays are anticipated, and the samples that will be acquired from the Wise County well will be processed before the next sample influx (scheduled to begin in June).

The NMR equipment is operational, and experimental details are being established on random shale samples. The tandem column SEM has been checked out and is operational. The Energy Dispersive X-Ray System has been reinstalled and is now operational. The Secondary Ion Mass Spectrometer has been installed on its own electron optics column and is presently being aligned and standardized. The mechanical test/mass spectrometer system has been successfully integrated and checked out. We are currently awaiting the delivery of the scleroscope (scheduled for April 1) before testing is initiated. With the integration of the data obtained from both tests, a more comprehensive picture of the physical properties of the shale will be possible. It is still anticipated that the material balance assay procedures will be established in April. Once the procedures have been reviewed, they will be formalized and assays will be performed on the current sample inventory.

Subcontract details have been finalized, and an integrated program with our subcontractor has been initiated.

B. MODIFIED FISCHER ASSAY

To evaluate the assay yield of the Eastern Gas Shales, the Project Office provided Mound Laboratory with some random samples from the R-109 well. The samples were assayed for oil and water, and the results are presented in Table 1. The elemental composition of the water collected from Sample R-109-3603.2 has been determined and is reported in Table 2.

TABLE 1

Fischer Assay Results from the R-109 Well

Sample No. R-109-3603.2'

Gals. Water/Ton Rock	2.55
Gals. Oil/Ton Rock	1.36

Sample No. R-109-3607.4'

Gals. Water/Ron Rock	1.90
Gals. Oil/Ton Rock	3.20

Sample No. R-109-3608.2'

Gals. Water/Ton Rock	2.90
Gals. Oil/Ton Rock	1.40

TABLE 2

Characteristics of the Water Obtained from the Assay of Sample R-109-3603.2

pH: 9.3

Elements present in the water as identified by Emission Spectroscopy:

Ca, Cu, Be, Mg, Fe, Si, B, Al

Elements present in the water as identified by Atomic Absorption:

Element	Concentration (ppm)
Ca	7
Cu	12
Be	2
Mg	4
Fe	9
Si	*
B	*
Al	*

*Water sample too small for quantitative identification.

It is currently planned to determine the pH of all water samples obtained during the material balance assay. Random samples will be examined for elemental composition.

While the material balance line is not fully set up, at the request of the Project Office, material balance assays were performed on several random samples which were selected from our inventory as well as two additional samples supplied by the Project Office. The assay data were otained on samples that were screened to 9-20 mesh. Results of these analyses are presented in Tables 3 and 4.

During April, it is planned to experiment with the assay sample particle size and heating conditions in order that the most consistent and realistic test procedures can be formalized. Once these procedures are formalized, the multiple retort assays will begin.

C. INFRARED SPECTRA OF ASSAY GASES

During the course of performing modified Fischer Assays for other portions of the program, gas samples were taken for infrared spectra analyses. The samples were taken just prior to the end of the soak period and should be accurate representations of the noncondensable gases evolved during the retorting processes.

The samples that were used for the retorting process had essentially the same weight and differed only in particle size. One sample consisted of 0.125-inch diameter particles; another was -32 mesh, while the third was -65 mesh.

The spectra obtained during the analyses are shown in Figure 1. Spectrum A represents the results obtained on the -65 mesh material; Spectrum B illustrates the results obtained on the -32 mesh material; and Spectrum C is representative of the results obtained on the 0.125-inch diameter particles.

TABLE 3

Sample Identification and Organic Carbon Content

Mound Sample No.	MERC Sample Identification	Well Location	Depth Interval	% Organic Carbon
0-1-4	$\bar{0}$-1-2292	Christian Co., Ky.	2292.47 - 2292.91	6.85; 6.84R*
0-1-5	$\bar{0}$-1-2317	Christian Co., Ky.	2317.21 - 2317.64	7.47
KY-2-55	C-336-3230A	Martin Co., Ky.	3230.27 - 3230.50	0.25; 0.25R
KY-2-47	C-336-3113A	Martin Co., Ky.	3113.40 - 3113.67	0.40
KY-2-34	C-336-2921B	Martin Co., Ky.	2921.50 - 2921.70	4.65; 4.64R
MERC-26	#26**		--	1.30***
MERC-43	#43**		--	7.37***

*R - Duplicate sample result.

**MERC supplied and identified samples.

***MERC determined values.

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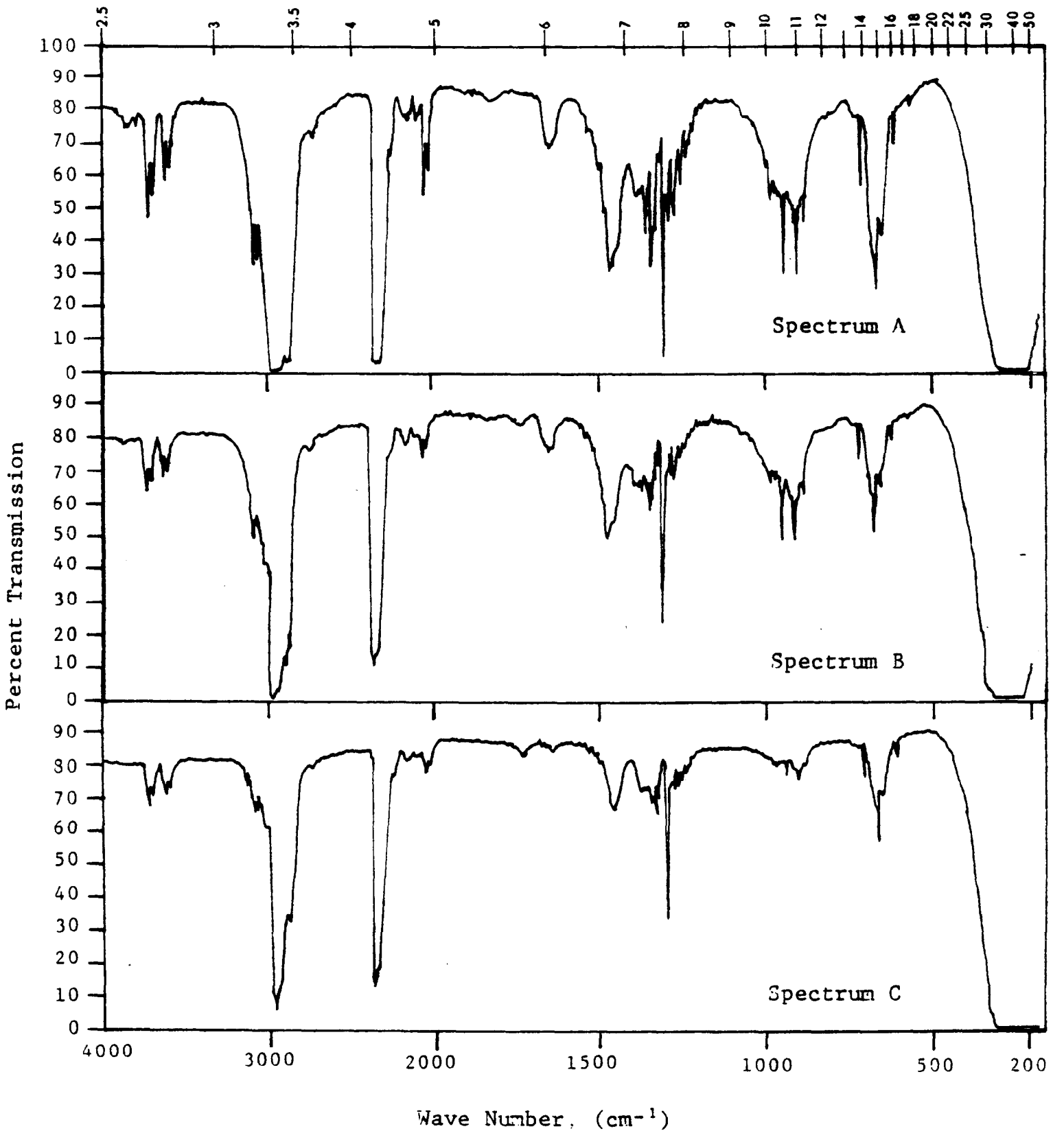
Material Balance

Sample:

0-1-5	Water	0.96%		
	Oil	2.47%	Total Volatiles	3.91%
(7.47%C)	Gas & Char Balance	0.48%		
	Residual Rock	96.09%		
	Total	100.00		
KY-2-34	Water	1.28%		
	Oil	2.06%	Total Volatiles	4.54%
(4.65%C)	Gas & Char Balance	1.20%		
	Residual Rock	95.46%		
	Total	100.00		
MERC-26	Water	0.96%		
	Oil	0.35%	Total Volatiles	1.31%
(1.30%C)	Gas & Char Balance	0.00%		
	Residual Rock	98.69%		
	Total	100.00		
MERC-43	Water	1.00%		
	Oil	3.54%	Total Volatiles	6.07%
(7.37%C)	Gas & Char Balance	1.53%		
	Residual Rock	93.93%		
	Total	100.00		

ASSAY RESULTS

Sample No.	0-1-5	
	Gals. Water/Ton Rock	2.30
	Gals. Oil/Ton Rock	6.23
	Total Volatiles - (H_2O) as Gals. Oil/Ton Rock	7.44
Sample No.	KY-2-34	
	Gals. Water/Ton Rock	3.06
	Gals. Oil/Ton Rock	5.19
	Total Volatiles - (H_2O) as Gals. Oil/Ton Rock	8.22
Sample No.	MERC-26	
	Gals. Water/Ton Rock	2.30
	Gals. Oil/Ton Rock	0.88
	Total Volatiles - (H_2O) as Gals. Oil/Ton Rock	0.88
Sample No.	MERC-43	
	Gals. Water/Ton Rock	2.39
	Gals. Oil/Ton Rock	8.93
	Total Volatiles - (H_2O) as Gals. Oil/Ton Rock	12.79



Infrared Spectra of Gases Produced
During Material Balance Assays

Figure 1

In general, the results show that the intensities of the two bands at about 2,920 and 2,850 cm^{-1} are greater for the smaller particle sizes. These bands are widely accepted as C-H vibrations. The results also show bands at 1,450 and 1,375 cm^{-1}. These are indicative of combined CH_2 and CH_3 groups and CH_3 groups, respectively. For the smaller particle sizes, these bands are again substantially greater. A sharp absorption band is also observed at 715 cm^{-1}. This band is characteristic of CH_2 chains and again is more intense for the smaller size particles.

Specific compounds that were identified in all spectra were: CO, CO_2, CH_4, C_2H_4, C_2H_6, and C_3H_8. The ethylene and ethane were the two species most effected by particle size. They became less intense as the particle size increased.

The results might indicate that with the larger sample particle size it is necessary to increase the heating rate and the soak time to allow the gases to permeate through the sample. This will be one of the variables which will be investigated during the development of the modified Fischer Assay process where sample particle size, heat-up rates, and soak times will be evaluated.

The results of these analyses will provide the initial data base for the Gas Chromatographic analyses which will be part of the formal material balance assay procedures.

D. PULSED NMR OF EASTERN GAS SHALES

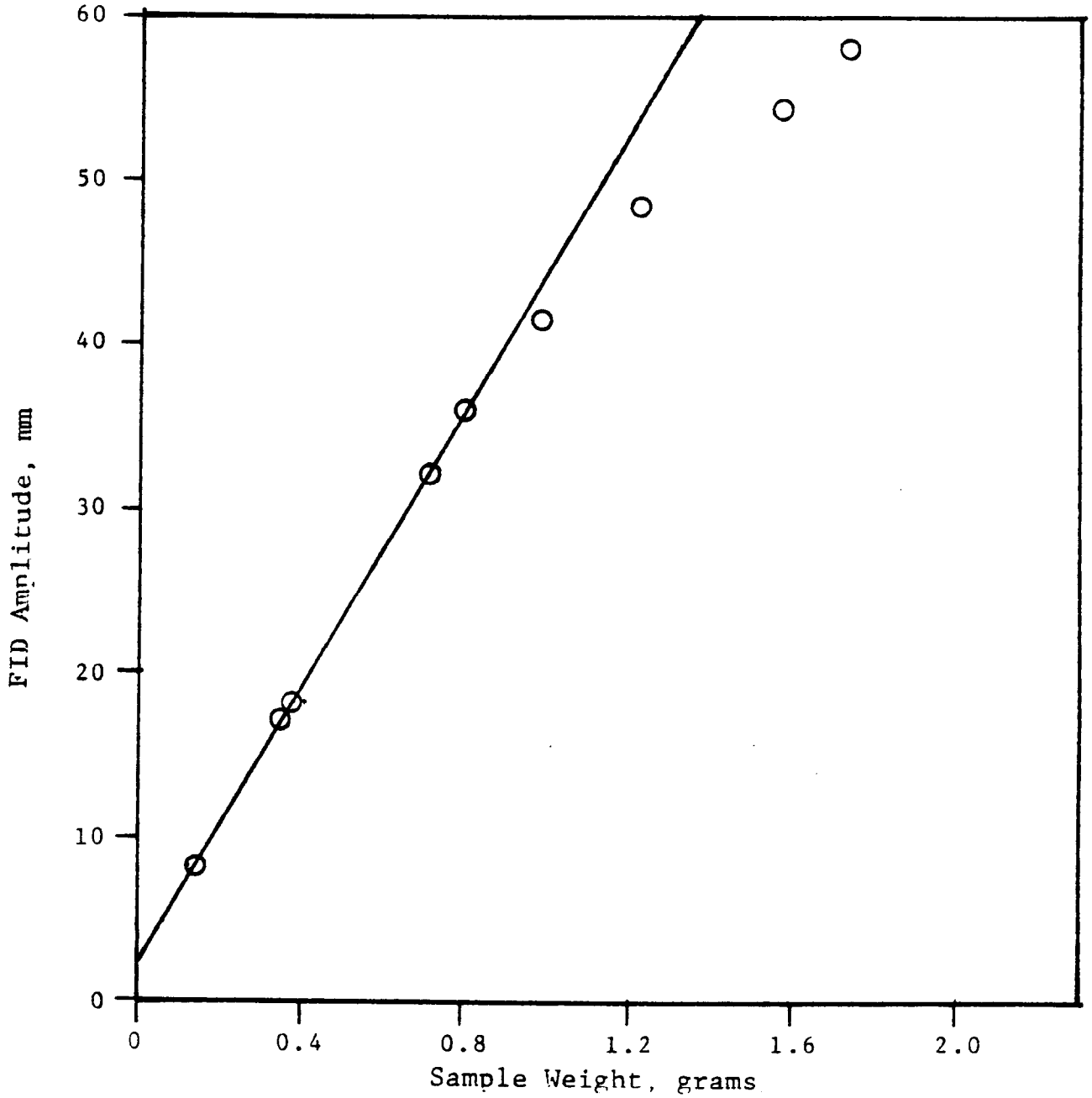
The main application of pulsed NMR for the characterization of the Eastern Gas Shale will be the correlation between the Fischer Assay fuel content and the amplitude of the proton free induction decay signal. If this application is successful, the result will be the direct, nondestructive, determination of the fuel content of the shale. Spin-lattice and spin-spin relaxation times as a function of temperature will also be studied. The relaxation time measurements as a function of temperature should theoretically reveal the relative proportions of gaseous, liquid, and solid-phases present in the shale.

Currently, instrument parameters are being maximized for performing the pulsed NMR experiments. Three rf frequencies (34.5, 45.7, and 60.0 MHZ) have been evaluated for overall instrument response (60.0 MHZ proved to be the most satisfactory). The high-power probe has been selected over the low-power probe because of its much lower proton background.

Ten-millimeter O.D. pyrex-brand glass sample tubes have been selected for their convenient size and shorter $\Pi/2$ pulse widths. Larger tubes require larger rf coils which increase the inhomogeneity of the rf pulse over the sample. Hence, larger rf pulses are required. Short pulse widths are necessary to recover as much as possible of the FID signal.

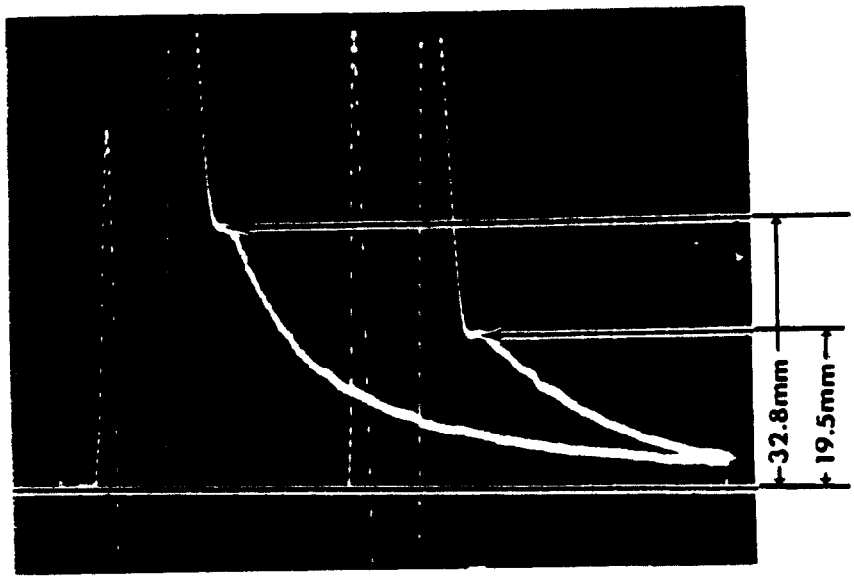
The NMR response as a function of sample size has been studied to determine the linear range of the instrument response to sample size. Figure 2 is a plot of the free induction decay (FID) amplitude as a function of sample weight. The instrument response became nonlinear when the sample size exceeded 0.8 grams. Samples of less than 0.8 grams will be required when performing spin counts or correlations between NMR data and other analytical techniques. Samples that do not exceed the capacity of the rf coil can be used for relaxation time measurements, since the relative decay of FID signal is being observed as a function of time; that is, no comparison of signal amplitude is being made with another sample.

Two major sampling errors are indigenous to shales. One is the inhomogeneity of the shale and the other is the uncontrollable and unaccountable release of organic gases. The inhomogeneity of shale is strikingly illustrated in Figure 3 and Table 5. One can instantly see that the larger (0.8875 g) sample of the light grey shale gives a much smaller FID signal than the smaller (0.7505 g) sample of the brown shale. The FID amplitudes of these two shale samples differ by a factor of 1.7 which can primarily be attributed to a greater abundance of protons in the brown colored zone. An indication of the loss of organic material that occurs during the grinding of the shale is demonstrated in Figures 4 and 5 and summarized in Table 6. Two different NMR experiments were performed using two different samples from Shale Sample R-109



NMR Response as a Function of Sample Weight

FIGURE 2



(0.7505 g)
Brown Zone

Light Grey Zone
(0.8875 g)

$\pi/2$ = 1.40 μsec

Comparison of the FID Amplitudes of Contiguous Pieces of Shale Taken from the Light Grey and Brown Colored Zones of Shale Core Sample #7239 at 2633 Feet.

FIGURE 3

TABLE 5

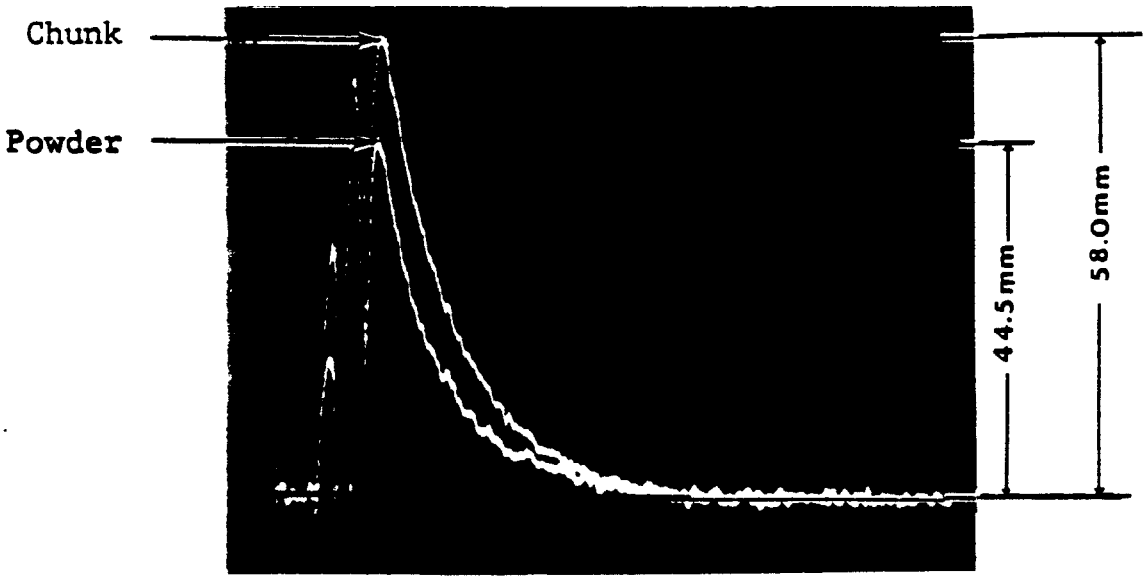
Comparison of Analytical Data of Contiguous Pieces of Shale Taken from the Light Grey and Brown Colored Zones of Shale Core Sample #7239 at 2633 Feet

Sample Weight (g)	FID Amplitude (mm)	FID Amplitude (mm/g)
0.7505 (brown zone)	32.8	43.7
0.8875 (light grey)	19.5	22.0

(3603.2). Figure 4 compares the FID amplitudes obtained from a 0.7305 g piece of shale before and after grinding, and Figure 5 compares the spin-echo signals obtained from a 0.7209 g piece of shale before and after grinding. The signal loss given in Table 6 was corrected for sample weight loss due to grinding.

The signal loss due to grinding and the difference in signal loss between the two shale samples or the two NMR experiments cannot be explained at this time. Only three factors, known at this time, can contribute to such a large change in signal amplitude. They are the loss of organic material, water, or a drastic change in the spin-spin relaxation time during the grinding process. The latter seems unlikely unless the sample was contaminated with paramagnetic atoms during the grinding procedure. Relaxation time data on powdered and single pieces of shale do not indicate a large change in relaxation time due to grinding. Further experimentation is necessary to explain the signal loss due to grinding.

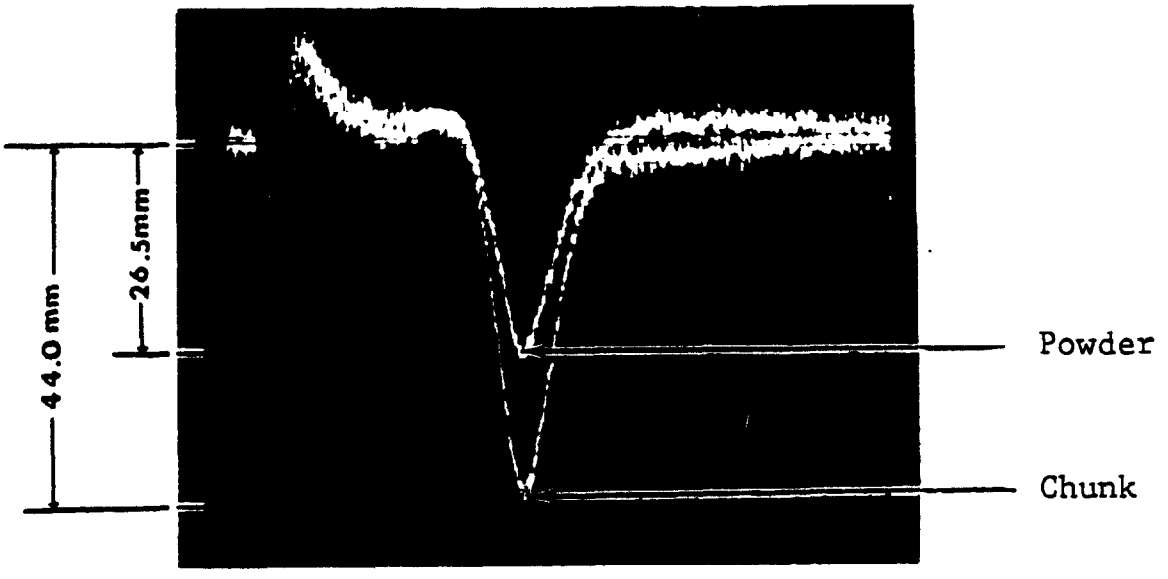
Four NMR techniques have been investigated for determining the abundance of protons in Eastern Gas Shales. They were the single-pulse $\left(\frac{\Pi}{2}\right)$ on resonance and off-resonance FID's, the double pulse spin-echo (π, τ, $\Pi/2$) and the solid echo $\left[\frac{\Pi}{2}, \tau, \left(\frac{\Pi}{2}\right)_{90°}\right]$ experiments. The purpose of conducting these four experiments was to select the best pulsed NMR technique for correlating the Fischer Assay fuel content of shale with the amplitude of the NMR response. Since Fischer Assay data were not available at the time of the experiments, correlations were studied between the amplitudes of the on-resonance FID signals and the amplitudes of the remaining three NMR outputs extrapolated to zero time. The extrapolations were performed to recover the signal that is lost during the instrument recovery time following the rf pulse (Figure 6). To obtain the extrapolated amplitudes, the envelope of the off-resonance FID signal (Figure 7) was plotted as a function of time-squared (Figure 8); the spin-echo (Figure 9) and solid-echo (Figure 10) amplitudes were plotted as a function of time (Figures 11 and 12, respectively). The off-resonance FID envelope and the solid-echo amplitudes were only partially plotted, since they behaved similarly to the extended spin-echo plot. The four linear portions of the spin-echo plot



$\Pi/2$ = 1.10 μsec

Comparison of FID Amplitudes of the Same Piece of Shale Sample R-109 (3603.2) Before and After Grinding to a Fine Powder.

FIGURE 4



$\Pi/2$ = 1.10 μsec, Π = 2.47 μsec

Comparison of the Spin-Echoes of the Same Piece of Shale Sample R-109 (3603.2) Before and After Grinding to a Fine Powder. Echoes Taken at 400 μsec Delay Between $\Pi/2$ and Π Pulses.

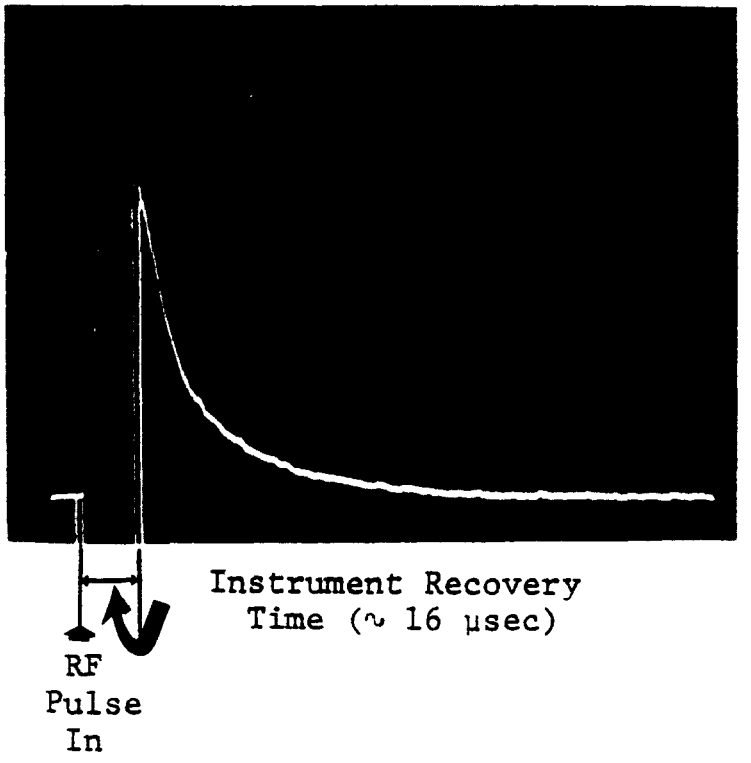
FIGURE 5

TABLE 6

Analytical Data for Loss in Signal Amplitude Due to Sample Grinding for Shale Sample R-109 (3603.2)

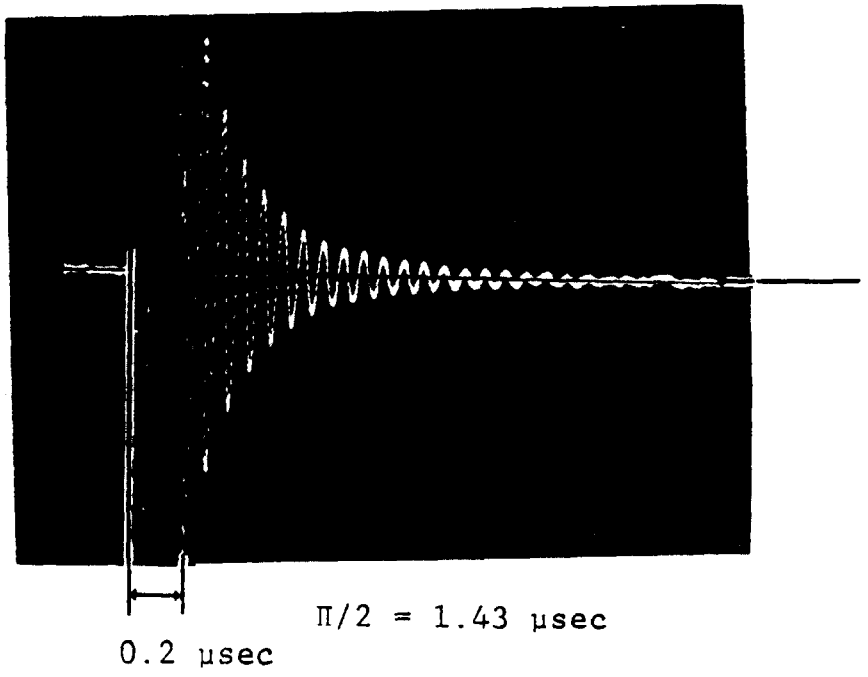
Sample Weight (g)	Weight Loss (%)	Amplitude (mm)	Signal Loss (%)
0.7315 (Chunk)		58.0 (FID)	
0.7117 (Powder)	2.7	44.5 (FID)	26.0
0.7209 (Chunk)		44.0 (Spin-Echo)	
0.7095 (Powder)	1.6	26.5 (Spin-Echo)	41.4

*Corrected for weight loss.



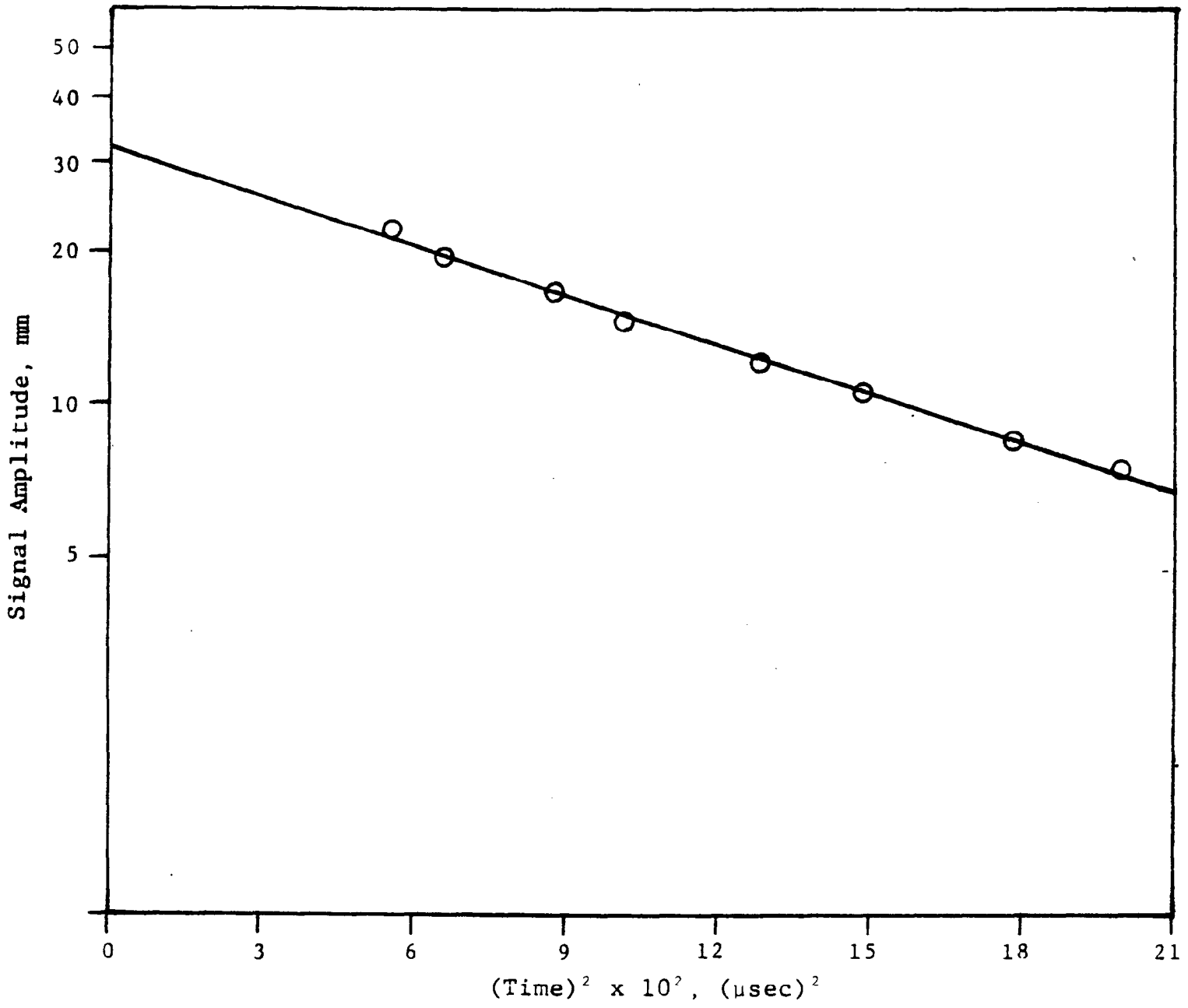
Typical Proton On-Resonance Free Induction Decay Signal of Eastern Gas Shale. Total Decay is Approximately 125 μsec duration.

FIGURE 6



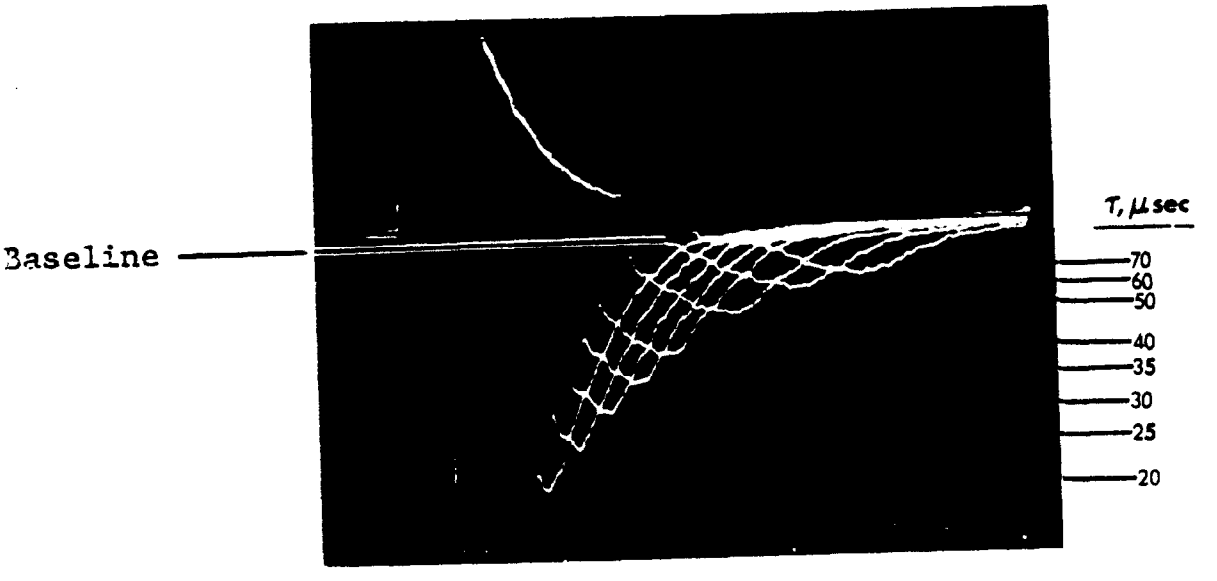
Typical Proton Off-Resonance Free Induction Decay Signal of Eastern Gas Shale.

FIGURE 7



Amplitude of the Decay Envelope of
the Proton Off-Resonance Signal of
Shale Sample #7239 at 2633 Feet

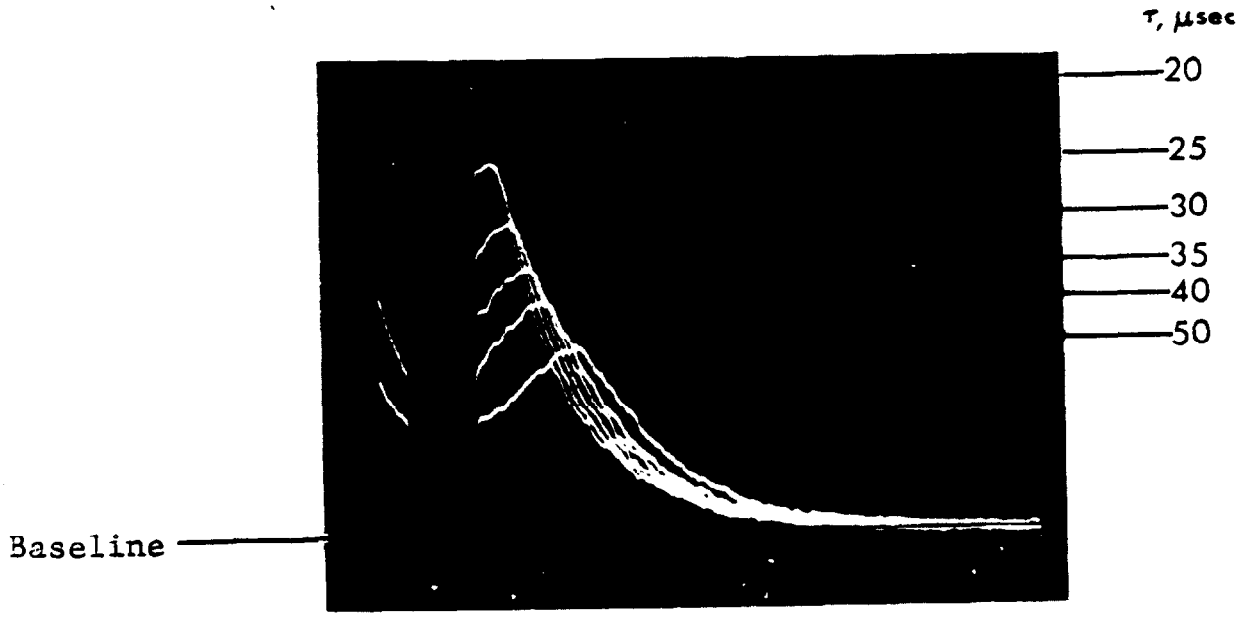
FIGURE 8



$\Pi/2$ = 1.53 μsec, Π = 3.48 μsec

Spin-Echo Technique $\left(\frac{\Pi}{2}, \tau, \Pi\right)$ Determining NMR Signal at Zero Time (Echo Amplitudes Extrapolated to Zero Time) and Spin-Spin Relaxation Time. Shale Sample #7239 at 2633 Feet.

FIGURE 9



$\Pi/2$ = 1.46 μsec

Solid-Echo Technique $\left[\frac{\Pi}{2}, \tau, \left(\frac{\Pi}{2}\right)_{90°}\right]$ for Determining NMR Signal at Zero Time (Echo Amplitudes Extrapolated to Zero Time) and Spin-Spin Relaxation Time. Shale Sample R-109 (3603.2).

FIGURE 10

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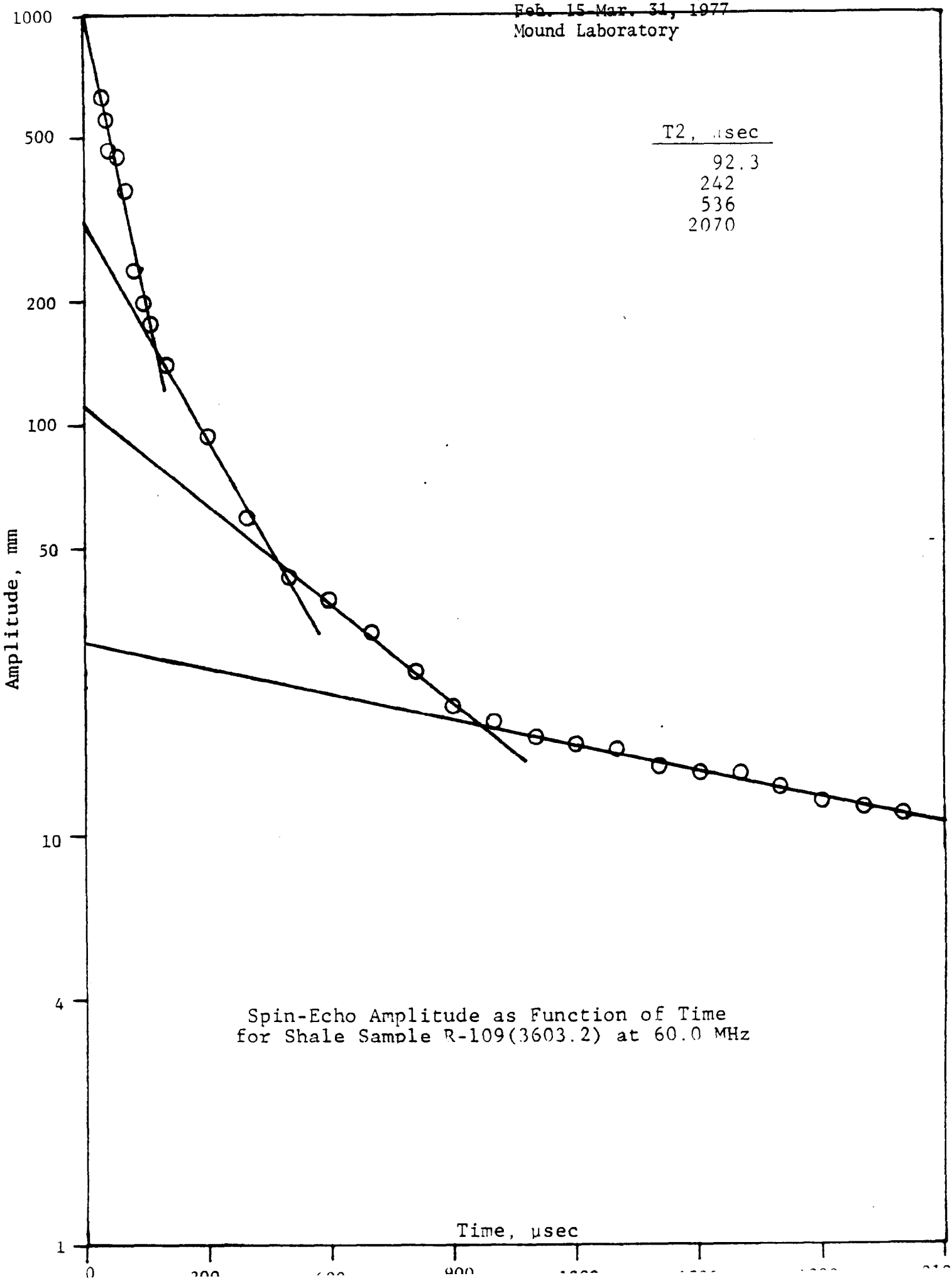
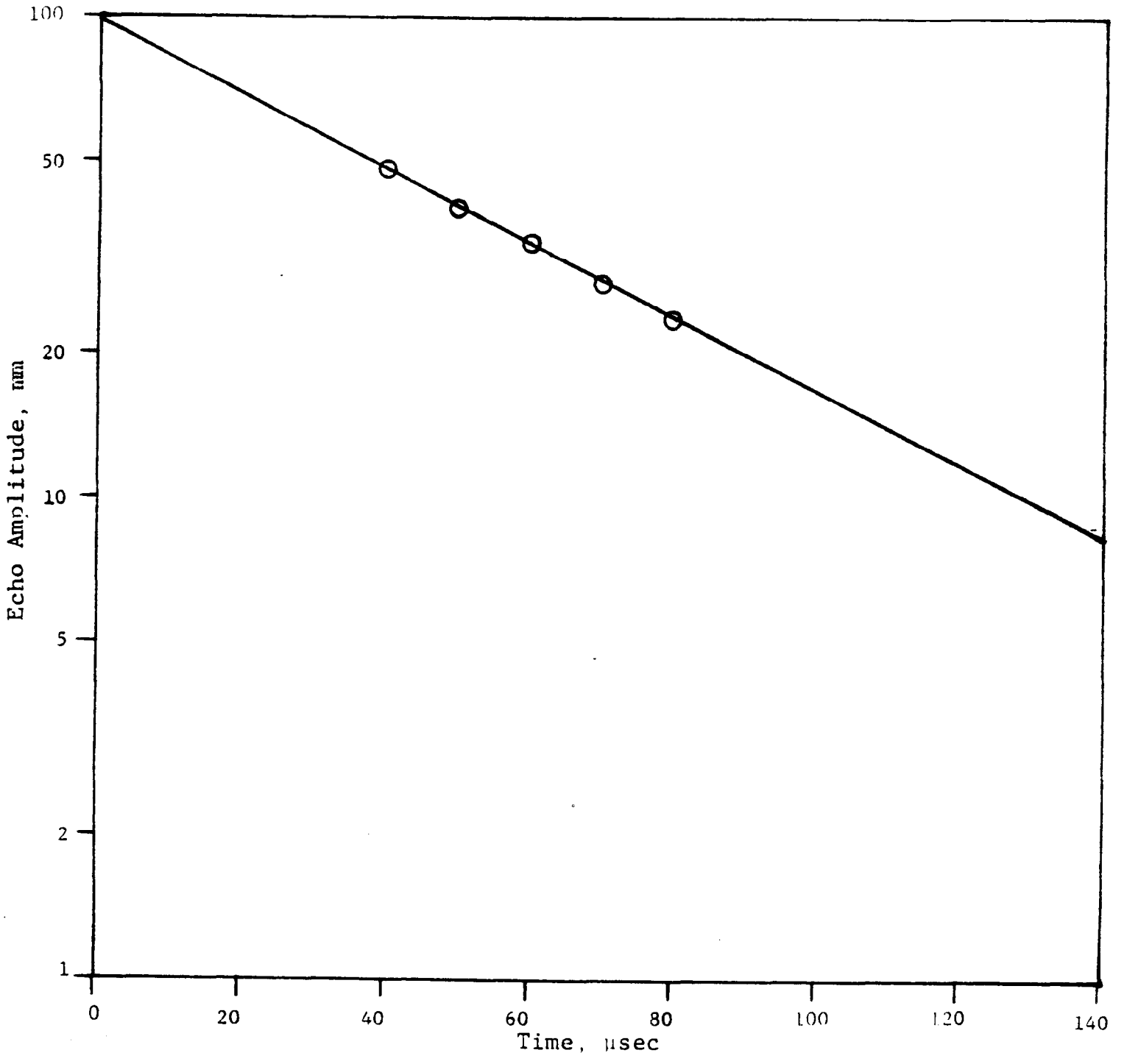


Spin-Echo Amplitude as Function of Time
for Shale Sample R-109(3603.2) at 60.0 MHz



Solid-Echo Amplitude Versus Time for Shale
Sample #7239 (at 2633 feet) at 60 MHz

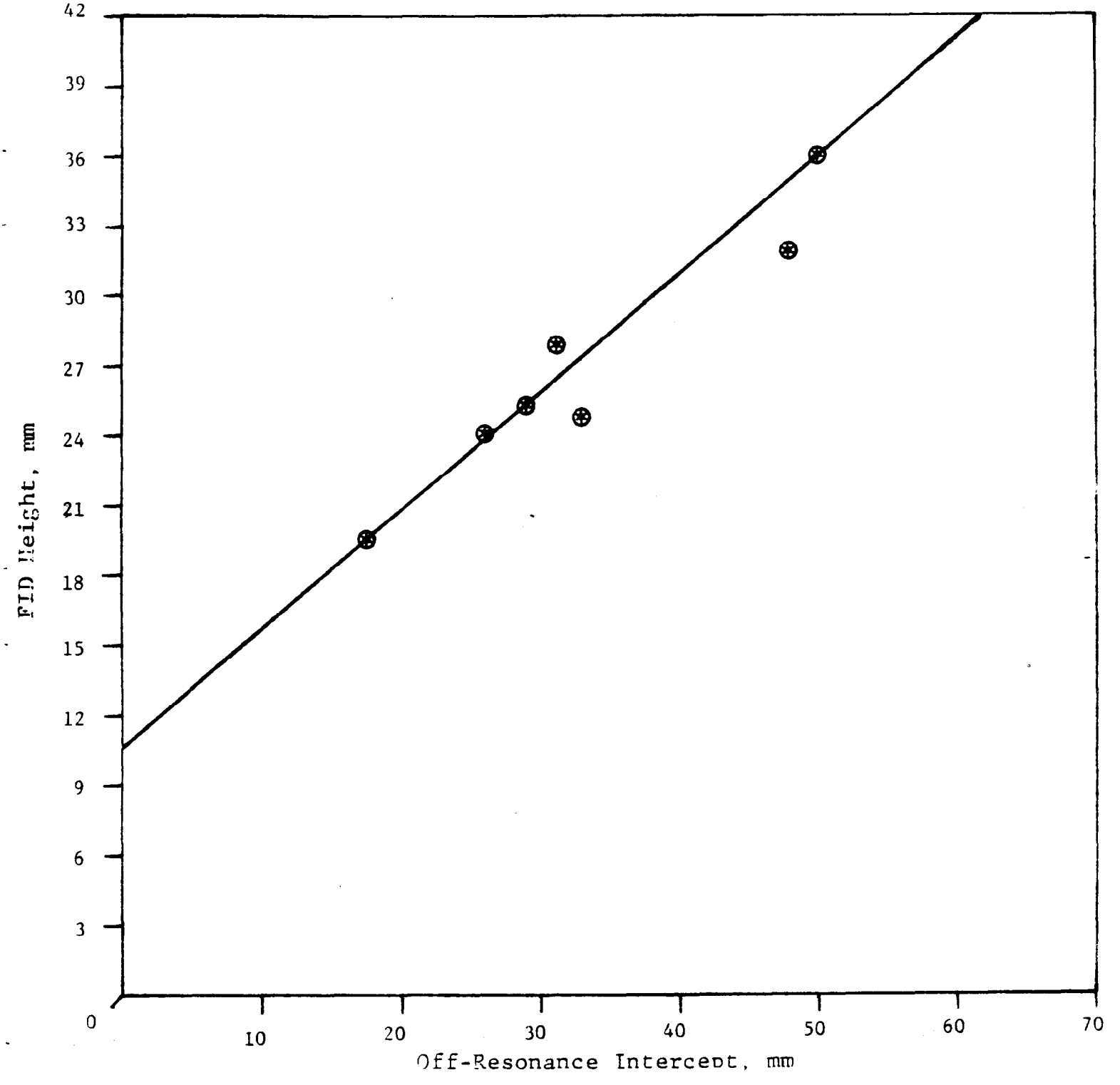
FIGURE 12

represent chemical species possessing different relaxation times, either because of their environment or physical phase or both. Further studies must be conducted to elucidate the source of these multi-linear plots. The spin-spin relaxation times inserted on Figure 10 indicate the range of relaxatimes of the chemical species in this particular shale sample.

The plots of the FID amplitudes of seven shale samples as a function of their corresponding extrapolated off-resonance envelopes and solid-echo amplitudes are given in Figures 13 and 14, respectively. It is obvious that the extrapolated off-resonance envelope did not correlate well with the FID amplitudes, whereas the solid-echo amplitudes did. The corresponding plot for the spin-echo experiment is not shown because it correlates worse than the off-resonance experiment. No reason is offered at this time for the finite intercept observed in Figures 13 and 14. The advantage of the off-resonance experiment is its independence of rf phasing and magnetic field fluctuations. The advantage of the echo experiment is the recovery of part of the signal that is lost during instrument recovery time. These techniques will be investigated further in order to explain the nonlinearity of the off-resonance and spin-echo correlations and the residual intercept of the correlation line.

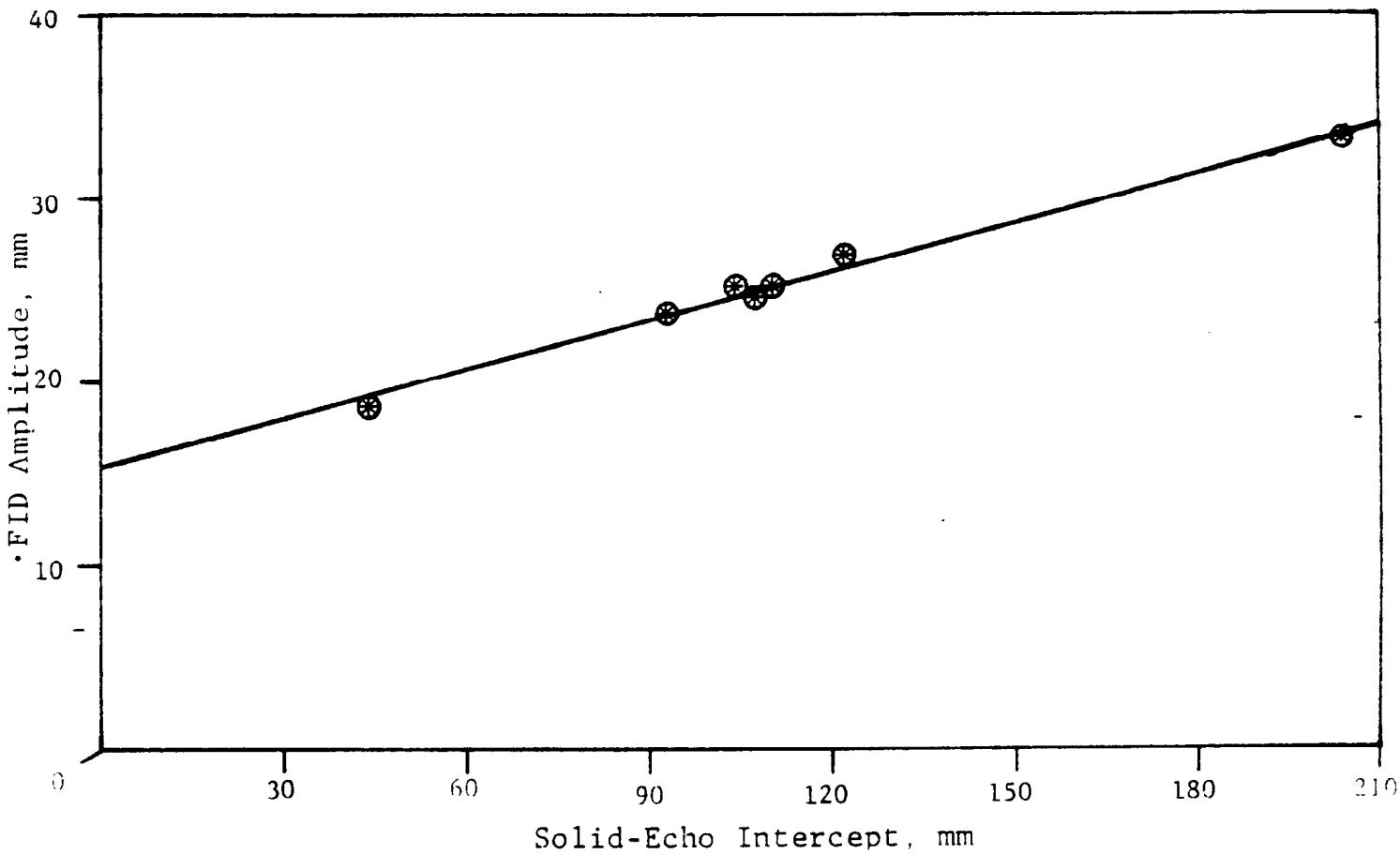
Spin-lattice relaxation times were measured using the pulse-inversion sequence: Π, τ, $\Pi/2$. Figure 15 represents a typical series of FID signals obtained by this technique. Each FID signal corresponds to the recoverable signal after a specific time delay, τ, between the Π and $\Pi/2$ rf pulses. The amplitude of the FID signals are plotted as a function of the delay time to give the curve illustrated in Figure 16. This spin-lattice relaxation curve is not linear because it is a composite of the relaxation times of many different chemical species in the shale. Only the "average" relaxation time, T_1 $(\frac{1}{e})$, is reported here. It is defined as the time required to reduce the signal amplitude by 1/e.

Spin-spin relaxation times were estimated from the linear extrapolations of the off-resonance envelopes and the solid-echo and spin-echo amplitudes. The relaxation time data are listed in Table 7 for easy reference. The spin-spin relaxation time obtained from the off-resonance FID is fairly



Off-Resonance FID Intercept Versus
On-Resonance FID Amplitude

FIGURE 13

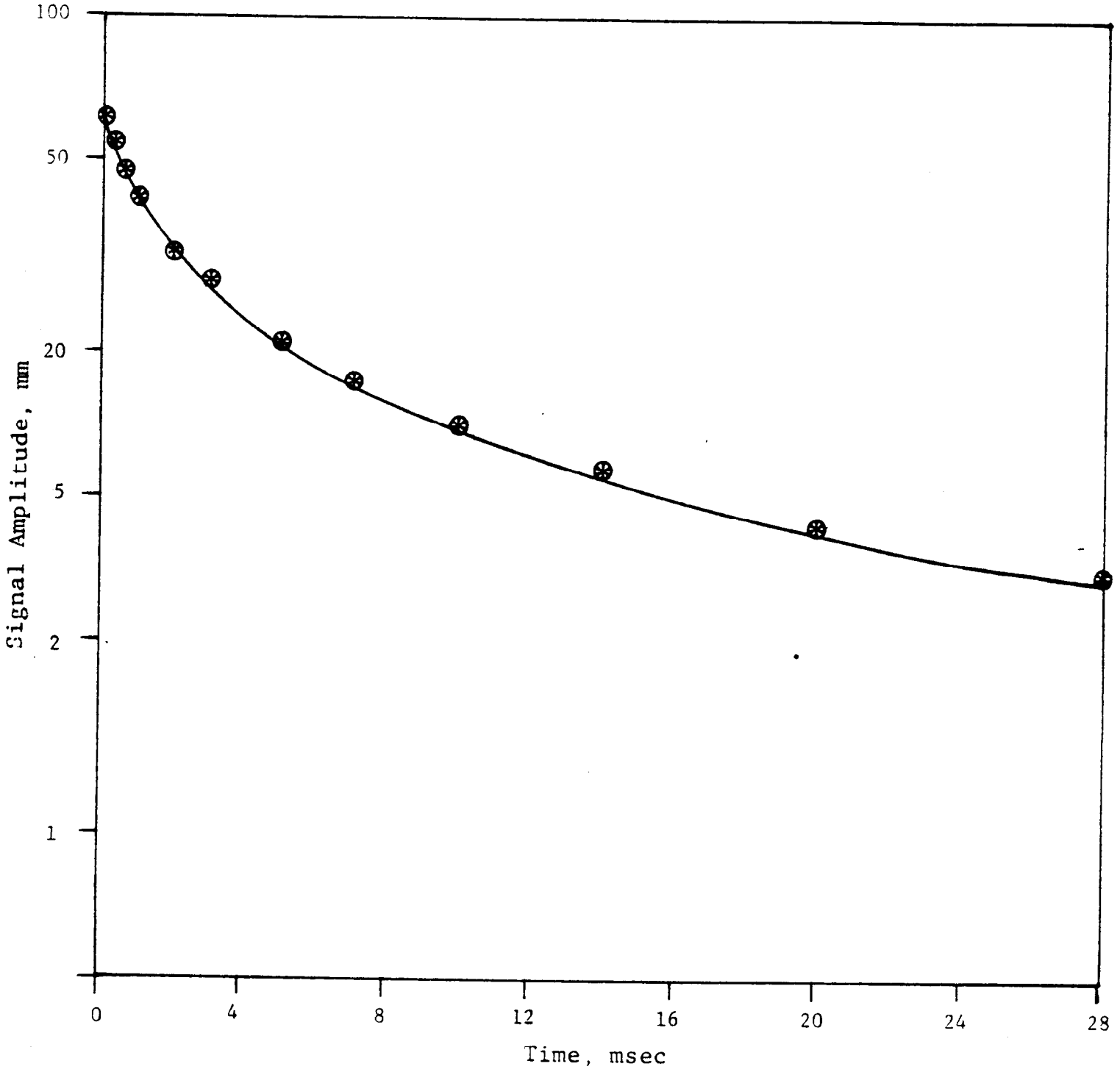


FID Amplitude Versus Solid-Echo Intercept
for Shale Sample #7239 at 2633 Feet

FIGURE 14

Example of Pulse Inversion Method (Π, τ, Π/2) for Determining Spin-Lattice Relaxation Time. Shale Sample #7239 at 2633 Feet.

FIGURE 15



Proton Spin-Lattice Relaxation Time Determination
for Shale Sample #7239 (at 2633 feet) at 60.0 MHz

FIGURE 16

TABLE 7

Spin-Lattice and Spin-Spin Relaxation Time Determinations for Eastern Kentucky and West Virginia shale at 60.0 Mhz.

Sample Number	Sample Weight (grams)	Comments	T ($\frac{1}{e}$) (msec)	T2, μsec		
				Solid Echo	Spin Echo	Off-Resonance FID
1	0.8711	HZ* Chunk		38.0	57.4	23.6
2	0.9074	HZ Chunk		62.9	44.9	27.0
3	0.8961	HZ Light Gray***	4.5	72.8	56.2	32.7
4	0.7858	HZ Brown	69.0	36.0	48.1	22.2
5	0.8786	HZ Chunk		47.5	56.3	26.8
6	0.8666	HZ Chunk		52.1	74.1	22.3
7	0.7434	R-109** Cylinder	49.0	53.6	68.6	30.3
8	0.8195	R-109 Chunk	51.0	45.5		22.9
9	0.6847	HZ Powder	35.0			
10	0.6955	HZ Chunk	47.0			

* Shale Sample #7239 at 2633 feet.

** Shale Sample R-109 (3603.2).

*** This sample was taken from a very light gray zone of the core sample. All other samples were brown.

constant and represents the dominate mechanism of spin-spin relaxation which is the inhomogeneity of the large external magnetic field. Notice that all the off-resonance T_2 values are much less than those obtained by the echo techniques which are less dependent on the magnetic field inhomogeneity. The Carr-Purcell-Meiboom-Gill method which is almost completely independent of many external effects will be pursued to determine the T_2 value indigenous to shale.

E. PULSED NMR CALIBRATION STANDARDS

To aid in the calibration of the NMR spectrometer for the shale work, Dr. F. P. Miknis of LERC is providing to us twelve standard oil shale samples. These samples have been ground to -65 mesh and are part of a suite of samples used for ASTM oil studies. The Fischer Assay results for these samples have been confirmed by several participating laboratories and are shown in Table 8. Figure 17 represents the correlation of the Fischer Assay data and the NMR data for these samples as obtained by Dr. Miknis.

F. SEM EVALUATION

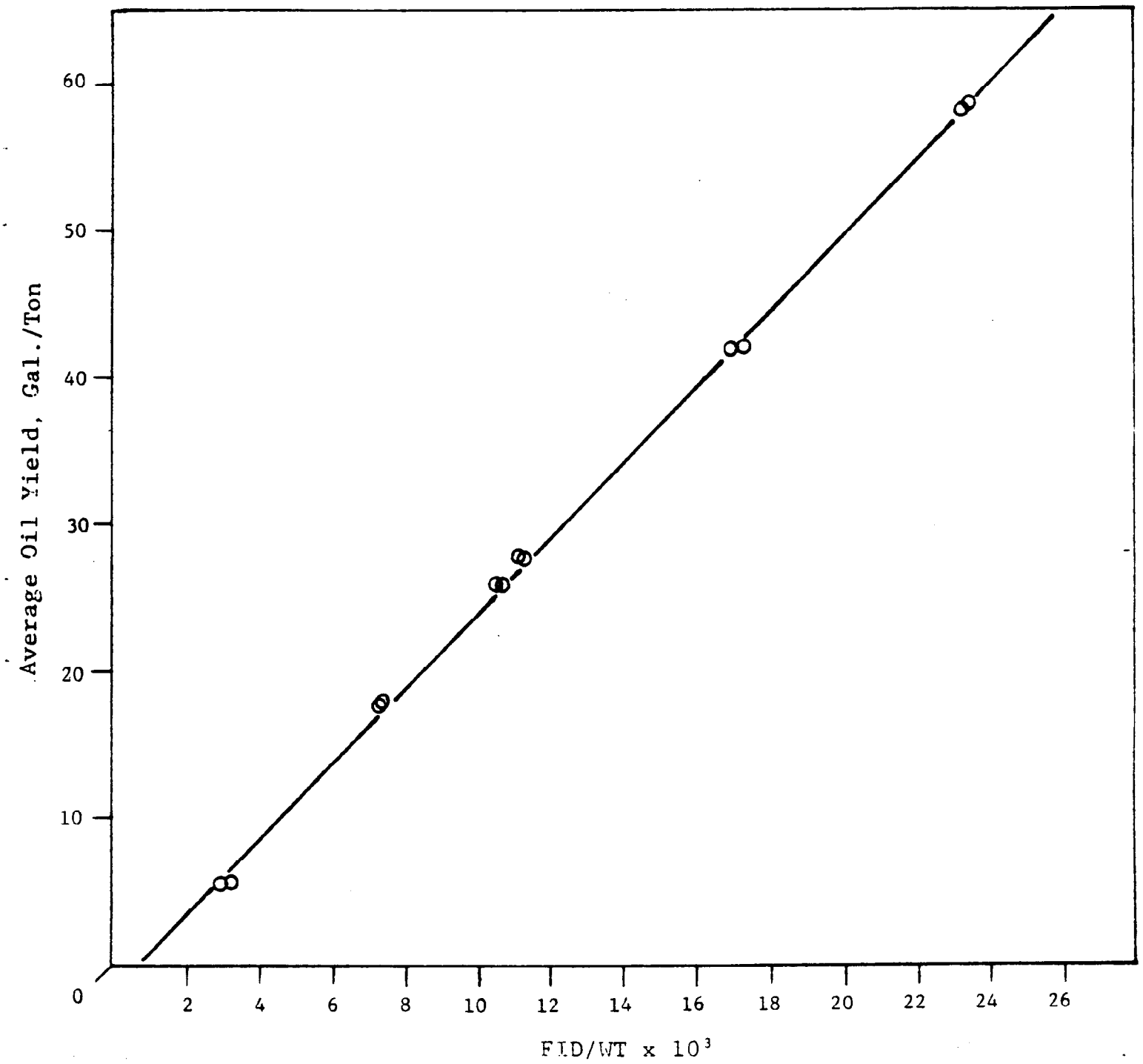
An initial data base is now being acquired using the SEM and its ancillary equipment. The initial sample which is being examined in great detail is Mound Sample P-1-3 (MERC #P-1-2549 .25/.67) from Sullivan County, Indiana. In order to provide a significant data base, several different forms of this sample are being investigated. These include: shale core, .0.125-inch diameter particles, before and after material balance assay as well as ash material; and -32 mesh material, before and after material balance assay as well as ash material.

Semi-quantitative results were obtained during the material balance assays used to prepare the samples for SEM analysis. These results are reported in Table 9 and indicate that using the same heat-up rate and soak time, more oil is produced from the smaller size particles. This difference in yield should result in sample differences that can be observed using the SEM and its ancillary equipment.

TABLE 8

Oil Yield Data for Calibration Standards

Standard Sample	Oil Yield, Gal./Ton
4S1	5.60
4S2	58.52
4S3	27.66
4S9	25.79
4S5	17.77
4S6	41.97
4S7	5.53
4S8	58.29
4S9	27.71
4S10	25.83
4S11	17.49
4S12	41.80



Fischer Assay and NMR Correlation
for Calibration Standard Samples

FIGURE 17

TABLE 9

Semi-Quantitative Material Balance Assay
Results for Sample P-1-3

Sample Number :	1	2
Sample Particle Size:	0.125 inch	-32 mesh
Gals. Oil/Ton Rock :	0.64	1.84
Gals. Water/Ton Rock:	1.73	1.84

It must be emphasized that the material balance assays were performed to supply SEM samples and the assay results are semi-quantitative. The balance of the P-1-3 sample will be subjected to a detailed hydrocarbon analysis as well as quantitative material balance assay.

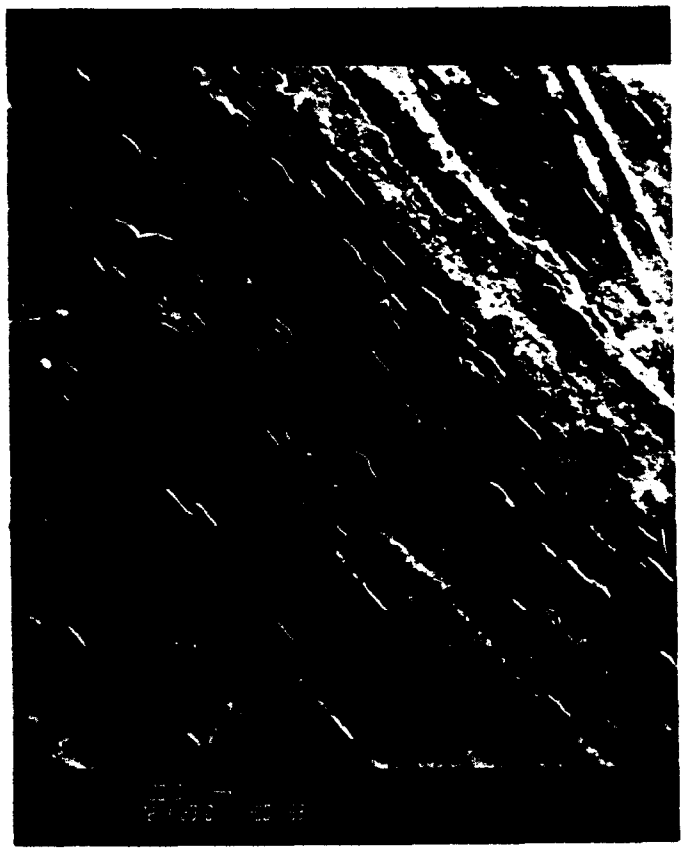
Examination of samples of shale core adjacent to the portion removed for material balance assay is currently in progress. Two types of samples are being examined. The first type consists of samples that have been hand-polished to provide smooth surface cross-sections. The second type consists of samples that have been fractured from the parent core.

Figure 18 shows a typical cross-section of the sample. A high concentration of spare material as well as lenses of quartz, feldspar, and pyrite are present in the sample. All of these features are parallel to bedding. The lenses appear to be very permeable. Most of the spores are filled and surrounded by pyrite. Series of microcracks also outline the spores (Figure 19). The charging effects or bright spots present in Figure 19 and the other figures are the result of the samples not being coated with a conductive material. The samples were not coated because they will be subjected to SIMS analysis, and we do not want to bias that analysis.

Figure 20 shows a distribution of spores including one that has essentially collapsed on itself. The spores that have apparently collapsed have a narrow zone of pyrite in the center (Figure 21). Iron, copper, nickel, titanium, calcium, silicon, and potassium are present in the apparent spore material.

Figures 22 and 23 show pyrite material that is present in the lenses of feldspar.

Figure 24 shows an interesting distribution of salt crystals. Sodium chloride cubes as well as crystals containing magnesium and chlorine, magnesium and sulfur, potassium, calcium, and sulfur are also present. Several of these areas were found in the samples.



Lenses of Feldspar, Pyrite, and Quartz As Well as Apparent Spore Material Are Observed Parallel to the Bedding Planes.

FIGURE 18

The Spores Contain and Are Surrounded by Pyrite. Microcracks Outline the Spore Structures.

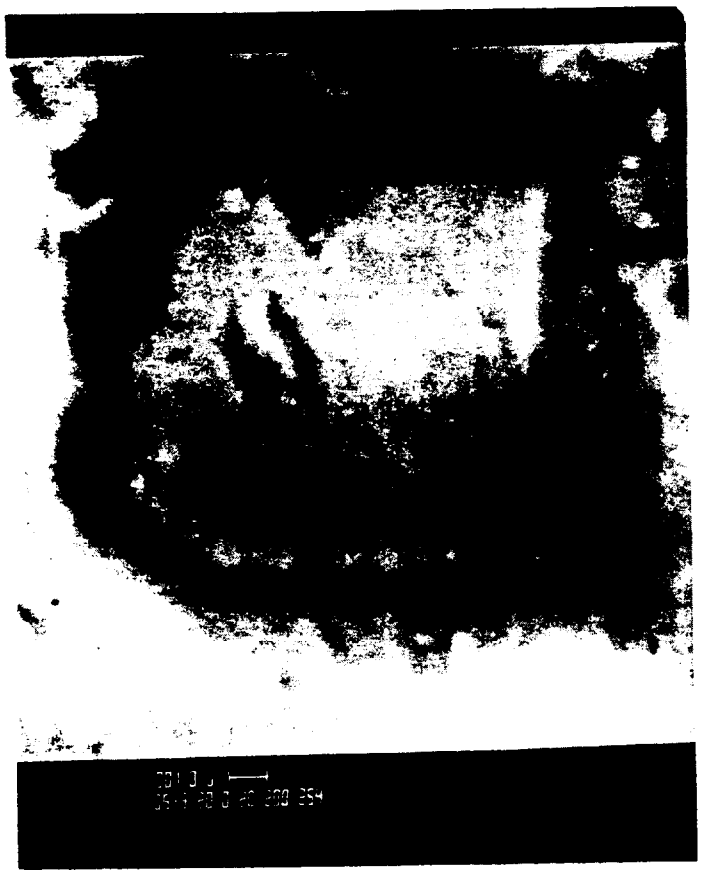
FIGURE 19

The Spores Are Elongated and Some Have Apparently Collapsed.

FIGURE 20

The Collapsed Spores Contain Narrow Bands of Pyrite in the Middle.

FIGURE 21



Pyrite Concentrations Are Found in the Feldspar Lenses.

FIGURE 22

Pyrite Concentration Located in a Feldspar Lens.

FIGURE 23

Several Areas of the Samples Had Concentrations of Salt Crystals Present. Leaf-Type Structures Are Also Present in These Areas.

FIGURE 24

Also present in Figure 24 are several leaf-type structures. More of these are shown in greater detail in Figure 25. These features might be microfossils. Elementally, they contain sodium, silicon, and chlorine with minor concentrations of potassium, calcium, and sulfur. These features are located in all the areas of apparent salt concentrations.

The polished samples will now be examined using the SIMS. Features already observed will be examined with the SIMS system as well as regions of organic material and microcracks.

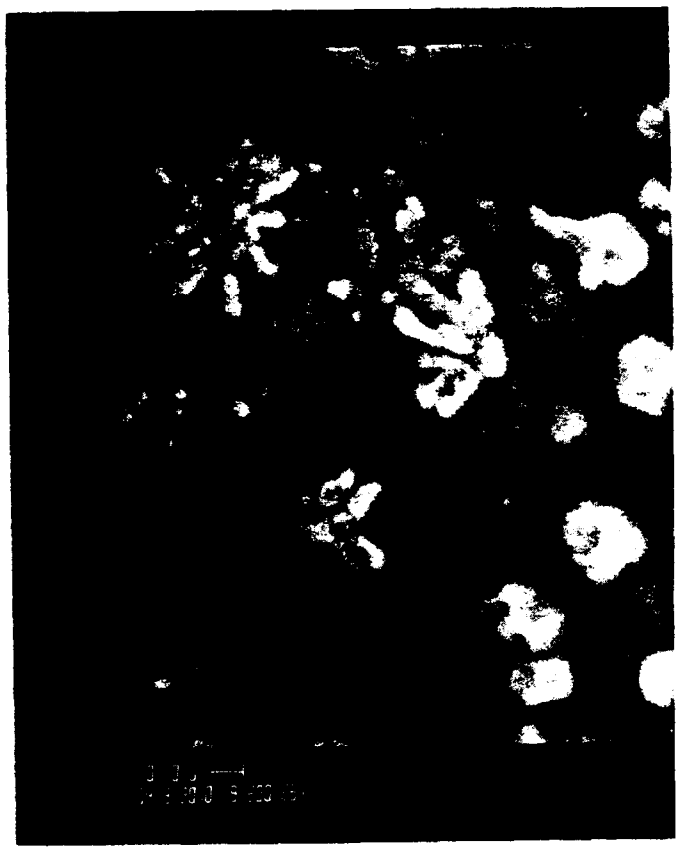
Examination of the fracture surfaces has just been initiated. Lenses of feldspar (light area of Figure 26) as well as what appear to be spores (Figures 26 and 27) have been observed on the fracture surface. The spore-like material in three dimensions consists of the same elements as those defined in two dimensions. A more detailed analysis of the fracture surface is not yet available.

G. OFF-GASSING STUDIES

Several shale sample containers were placed in special gas tapping fixtures, and the total pressure and gas composition present in the containers have been measured. Gas pressure and compositional changes have been monitored periodically.

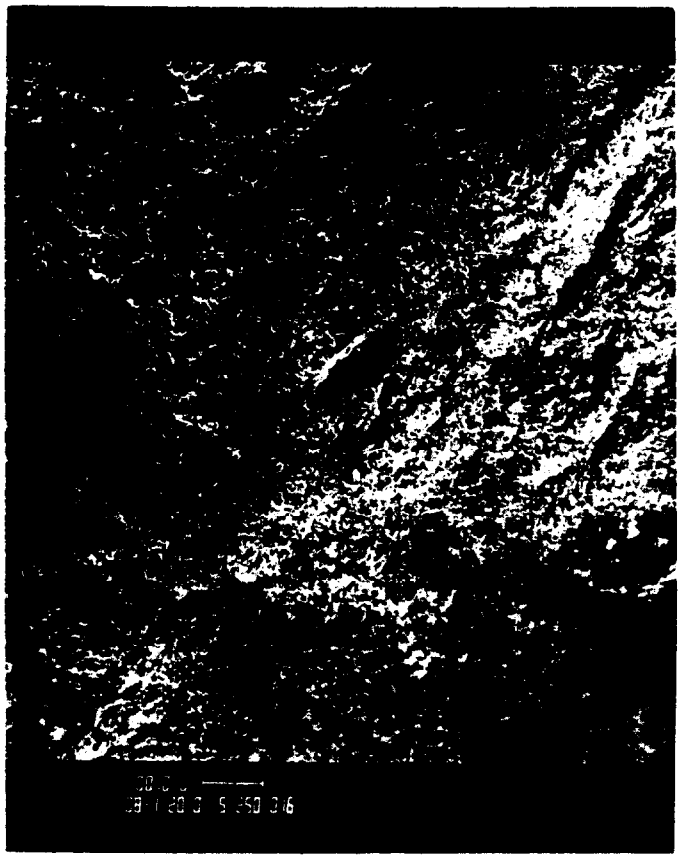
The first series of samples which were tested in this manner were the P-1 samples from Sullivan County, Indiana. At the time that these samples were initially gas tapped, the gas tapping fixtures presently being utilized in the program were not available. This resulted in the samples being removed from their original containers immediately after gas tapping and being placed in special pressure fixturing for the balance of the test period. The transfer and storage occurred in an air atmosphere.

The second series of samples were from the Martin County, Kentucky, well; and these samples were the companion samples of the three that were subjected to material balance assays (Section B). These samples were maintained in the gas tap fixtures and were monitored periodically for pressure and gas compositional changes.



The Leaf-Type Structures Could Possibly Be Microfossils.

FIGURE 25



Lenses of Feldspar (Light Area) as Well as Spore-Like Material Are Also Observed on Fracture Surfaces.

FIGURE 26

The Spore-Like Material on the Fracture Surfaces Contains the Same Elements as Those Found in the Cross-Section.

FIGURE 27

1. P-1 Samples

Table 10 identifies the P-1 samples, while Table 11 presents the condition of the samples at the time of the original gas tap. After the original gas tap, the samples were removed from their original containers and placed in special pressure fixtures. At this time, it was noted that no rust was present on the interior surfaces of the original containers. The samples were stored in the special fixtures for varying lengths of time prior to a final gas sample being taken. Table 12 presents the conditions at the time of the final gas tap.

The composition of the gas present in the sample containers as well as the final composition of the gas present in the special fixtures for each of the samples is shown in Tables 13, 14, and 15.

While there is a lack of available information to sufficiently explain what is occurring in the samples at the present time, some preliminary observations can be made. In the initial samples, significant quantities of methane were observed; the nitrogen, oxygen ratio had shifted from that observed in normal air with oxygen being considerably lower and the CO_2 composition had increased significantly above that found in normal air. Since the sample containers showed no indication of rust; and since the free oxygen was apparently being depleted, it appeared that a majority of the CO_2 was being generated as a result of sample oxidation.

Some of the CO_2 probably was the result of the natural generation of hydrocarbons from the organic matter. This is normally expressed as:

$$k(aC,\ bH,\ cO) \longrightarrow k'(a'C,\ b'H,\ c'O) + X\ (CO_2) + Y(H_2O) + Z\ (C_nH_{2n+2}),$$

where k(aC, bH, cO) is the initial concentration of reactant organic matter, k'(a'C, b'H, c'O) is the concentration of the residual organic matter, $X(CO_2)$ is the concentration of carbon dioxide, $Y(H_2O)$ is the concentration of water, and $Z(C_nH_{2n+2})$ is the concentration of hydrocarbons expressed as methane.

TABLE 10

Sample Identification of the P-1 Samples
from Sullivan County, Indiana

Mound Sample Number	MERC Sample Number	Depth Interval
P-1-1	P-1-2492	2492.33 - 2492.83
P-1-2	P-1-2521	2521.08 - 2521.33
P-1-3	P-1-2549	2549.25 - 2549.67

TABLE 11

Gas Pressure in Can at
Time of Initial Gas Tap

Mound Sample Number	Date Canned	Date Tapped	Days in Can	Gas Pressure in Can
P-1-1	09/01/76	10/29/76	58	1.5 atm
P-1-2	09/01/76	10/18/76	47	1.94 atm
P-1-3	09/01/76	09/15/65	14	3.76 atm

TABLE 12

Conditions Present at Time
of Final Gas Tap

Mound Sample Number	Days in Special Fixtures	Gas Pressure in Special Fixtures
P-1-1	80	1.002 atm
P-1-2	85	1.157 atm
P-1-3	87	1.068 atm

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Mound Laboratory

TABLE 13

Composition of Gas Observed for Sample P-1-1

Components (mole %)	Initial Composition	Final Composition After 80 Day Storage
Methane	49.15	0.03
16 (%)	98.98	--
17 (%)	1.02	--
2-H_2	0.02	0.00
18-H_2O	0.06	1.10
26-C_2H_2	0.00	0.00
28-CO	1.10	0.00
29-CO	0.00	0.00
30-CO	0.00	0.00
28-N_2	28.90	77.20
28-C_2H_4	1.10	0.00
30-C_2H_6	4.30	0.00
32-O_2	5.73	20.70
40-Ar	0.37	0.90
44-CO_2	9.02	0.10
45-CO_2	0.11	0.00
46-CO_2	0.03	0.00
47-CO_2	0.00	0.00
44-C_3H_8	0.00	0.00

TABLE 14

Composition of Gas Observed for Sample P-1-2

Composition (mole %)	Initial Composition	Final Composition After 85 Day Storage
Methane	51.30	33.9
16 (%)	98.97	99.12
17 (%)	1.03	0.88
2-H_2	0.10	0.40
18-H_2O	0.10	0.50
26-C_2H_2	0.00	0.00
28-CO	0.00	0.00
29-CO	0.00	0.00
30-CO	0.00	0.00
28-N_2	30.90	47.70
28-C_2H_4	1.40	1.00
30-C_2H_6	5.00	4.40
32-O_2	5.20	5.30
40-Ar	0.40	0.60
44-CO_2	3.80	6.10
45-CO_2	0.10	0.10
46-CO_2	0.00	0.00
47-CO_2	0.00	0.00
44-C_3H_8	1.70	--

TABLE 15

Composition of Gas Observed for Sample P-1-3

Composition (mole %)	Initial Composition	Final Composition After 87 Day Storage
Methane	13.20	9.80
16 (%)	98.98	98.98
17 (%)	1.02	1.02
2-H_2	0.00	0.01
18-H_2O	0.20	2.20
26-C_2H_2	0.00	0.00
28-CO	0.00	0.00
29-CO	0.00	0.00
30-CO	0.00	0.00
28-N_2	74.60	71.60
28-C_2H_4	0.90	1.30
30-C_2H_6	3.10	4.00
32-O_2	1.40	1.40
40-Ar	0.90	0.90
44-CO_2	4.30	8.60
45-CO_2	0.10	0.10
46-CO_2	0.00	0.00
47-CO_2	0.00	0.00
44-C_3H_8	1.30	--

At the present time, it is not possible to quantify the amount of CO_2 that would be generated during diagenesis; however, the detailed hydrocarbon analyses and the detailed elemental kerogen studies, which will be performed on these samples, will aid in assessing that quantity.

Also interesting to note is that for the P-1-3 sample, the nitrogen and argon concentrations remained quite high, suggesting that both constituents were probably off-gassing from the shale. The gas obtained from the P-1-1 sample contained the largest concentration of CO_2, and it is the only one that also contained CO.

In the post storage condition, the data suggest that Sample P-1-1 had apparently off-gassed all of its free methane prior to storage and also produced no more CO_2. The nitrogen, oxygen ratio remained the same as for normal air. Samples P-1-2 and P-1-3 still off-gassed significant quantities of methane; the amount of CO_2 off-gassed from both samples essentially doubled when they were placed in a fresh air supply. Sample P-1-3 apparently continued to off-gas nitrogen and argon.

2. KY-2 Samples

Table 16 identifies the KY-2 samples used in this study, and Table 17 lists the gas pressures present at the time of gas tapping. These samples were positioned in the new gas tap fixtures and have remained in the fixtures since the original gas tap. The gas composition and container pressure has been monitored periodically since the original gas tap, and these results are presented in Tables 18, 19, and 20.

The initial gas samples for the KY-2 material indicated results similar to those observed for the P-1 samples. The concentration of methane was not as great as that observed for the P-1 samples, but again the nitrogen, oxygen ratio had shifted from that found in normal air; oxygen was again lower in concentration. CO_2 was also observed in all three samples. The concentration of hydrogen was also greater in these samples. The nitrogen composition in these samples was greater than that observed for samples P-1-1 and P-1-2, but was similar to that observed in the P-1-3 samples, although the oxygen concentration was significantly different.

TABLE 16

Sample Identification of the KY-2
Samples from Martin County, Kentucky

Mound Sample Number	MERC Sample Number	Depth Interval
KY-2-33	C-336-2921-A	2921.32 - 2921.50
KY-2-48	C-336-3113-B	3113.67 - 3113.91
KY-2-56	C-336-3230-B	3230.50 - 3230.64

TABLE 17

Gas Pressure in Can at
Time of Initial Gas Tap

Mound Sample Number	Date Canned	Date Tapped	Days in Can	Gas Pressure in Can
KY-2-33	10/30/76	03/02/77	124	1.155 atm
KY-2-48	11/02/76	03/01/77	119	2.212 atm
KY-2-56	11/03/76	03/01/77	118	2.443 atm

TABLE 18

Composition of Gas and Gas Pressure Change Observed for Sample KY-2-33

Components (mole %)	Initial Sample	8-Day* Sample	15-Day* Sample	22-Day** Sample
Methane	5.21	0.28	0.32	0.24
16 (%)	100.00	--	100.00	--
17 (%)	0.00	--	0.00	--
2-H_2	0.08	0.03	0.00	0.05
3-He	0.00	0.00	0.00	0.00
4-He	0.00	0.00	0.00	0.00
18-H_2O	2.70	0.17	0.21	0.39
26-C_2H_2	0.00	0.00	0.00	0.00
28-CO	0.00	3.26	3.15	0.00
29-CO	0.00	0.00	0.00	0.00
30-CO	0.00	0.00	0.00	0.00
28-N_2	70.33	73.82	73.94	76.85
28-C_2H_4	0.74	0.05	0.06	0.04
30-C_2H_6	2.57	0.64	0.68	0.57
32-O_2	16.25	20.79	20.69	20.82
40-Ar	0.80	0.87	0.87	0.86
44-CO_2	1.31	0.08	0.09	0.09
45-CO_2	0.02	0.00	0.00	0.00
46-CO_2	0.00	0.00	0.00	0.00
47-CO_2	0.00	0.00	0.00	0.00
44-C_3H_8	0.00	0.00	0.00	0.00
Gas Pressure (atm)	1.155	--	--	--

*Fixture seal leaked.

**Sample taken after seal was repaired.

TABLE 19

Composition of Gas and Gas Pressure Change Observed for Sample KY-2-48

Components (mole %)	Initial Sample	8-Day Sample	15-Day* Sample	22-Day Sample
Methane	12.19	12.17	0.53	12.21
16 (%)	99.25	99.67	98.83	99.15
17 (%)	0.75	0.33	1.17	0.85
2-H_2	0.21	0.20	0.00	0.19
3-He	0.00	0.00	0.00	0.00
4-He	0.00	0.00	0.00	0.00
18-H_2O	0.54	1.02	0.24	1.41
26-C_2H_2	0.00	0.00	0.00	0.00
28-CO	0.00	0.00	2.20	0.00
29-CO	0.00	0.00	0.00	0.00
30-CO	0.00	0.00	0.00	0.00
28-N_2	66.33	66.16	74.54	66.46
28-C_2H_4	1.57	1.25	0.03	1.29
30-C_2H_6	5.13	3.60	0.61	3.59
32-O_2	11.84	11.93	20.88	10.87
40-Ar	0.72	0.73	0.88	0.74
44-CO_2	1.42	1.61	0.09	1.74
45-CO_2	0.03	0.04	0.00	0.04
46-CO_2	0.00	0.00	0.00	0.00
47-CO_2	0.00	0.00	0.00	0.00
43-C_3H_7	--	1.28	--	1.46
44-C_3H_8	0.00	0.00	0.00	0.00
Gas Pressure (atm)	2.212	2.385	2.385	2.385

*Mass Spectrometer sample bottle was contaminated with air.

TABLE 20

Composition of Gas and Gas Pressure Change Observed for Sample KY-2-56

Components (mole %)	Initial Sample	8-Day Sample	15-Day Sample	22-Day Sample
Methane	8.39	7.90	8.24	8.00
16 (%)	98.91	99.75	99.38	99.10
17 (%)	1.09	0.25	0.62	0.90
$2H_2$	0.14	0.07	0.13	0.06
3-He	0.00	0.00	0.00	0.00
4-He	0.00	0.00	0.00	0.00
18-H_2O	0.07	1.05	0.48	1.77
26-C_2H_2	0.00	0.00	0.00	0.00
28-CO	0.00	0.00	0.00	0.00
29-CO	0.00	0.00	0.00	0.00
30-CO	0.00	0.00	0.00	0.00
28-N_2	72.40	72.39	72.72	71.78
28-C_2H_4	0.96	0.72	0.77	0.80
30-C_2H_6	3.30	2.40	2.60	2.50
32-O_2	11.07	10.94	10.29	10.19
40-Ar	0.83	0.80	0.82	0.79
44-CO_2	2.80	3.12	3.32	3.39
45-CO_2	0.04	0.05	0.05	0.05
46-CO_2	0.00	0.01	0.02	0.02
47-CO_2	0.00	0.00	0.00	0.00
44-C_3H_8	0.00	0.00	0.60	0.64
43-C_3H_7		0.55		
Gas Pressure (atm)	2.443	2.443	2.443	2.443

The initial methane and ethylene concentrations present in the samples had an inverse relationship to the % of organic carbon. Sample KY-2-33 had the highest organic carbon carbon concentration (4.65%), but the lowest concentration of methane and ethylene present in the off-gas species. Samples KY-2-48 and KY-2-56 had low organic carbon concentrations (0.40% and 0.25%) but off-gassed more methane and ethylene.

The additional gas samples taken on Sample KY-2-33 indicated that the fixture seal was leaking. This condition was corrected following the 15-day sample, and the sample taken one week later indicated that little if any additional gas was off-gassing from the shale. This sample is still in the fixture and will be sampled at least one more time to see if the sample is still outgassing.

Sample KY-2-48 has shown little change over the 22-day sampling period. A slight decrease in the oxygen concentration with a corresponding increase in the CO_2 concentration has occurred.

Sample KY-2-56 is behaving very similar to Sample KY-2-48. In this sample, there is apparent H_2/H_2O cycle. As the concentration of hydrogen decreases, the concentration of water increases and conversely when the concentration of hydrogen increases, the concentration of water decreases.

Since this study has produced some unusual results, a more definitive test matrix has been developed. This test matrix will be used for the Wise County, Virginia, samples and hopefully the results from that study combined with the detailed hydrocarbon and kerogen analyses will explain the off-gassing behavior of the material. It is also planned to increase the sampling range of the mass spectrometer.

Since the gas tap procedure is also a part of the mechanical testing program, we will also be accumulating a data base of initial gas sample analyses for a significant number of our samples.

H. DETAILED HYDROGEN ANALYSIS

The C_1-C_7 hydrocarbon analyses and organic carbon analyses for all the samples from the Martin County, Kentucky, (KY-2); Christian County, Kentucky, (O-1); and Ellingham County, Illinois, (I-1) wells have been completed. The C_1-C_4 hydrocarbon data, % wetness, and organic carbon data are summarized in Figures 28 and 29. The detailed results are listed in Tables 21 through 30.

The detailed hydrocarbon analyses of these samples as well as the associated analyses are in progress. Once these analyses are completed, all of the data will be integrated and interpreted in well summary formats. The data already acquired has been used to calculate estimated indigenous gas contents on a per-sample basis. These results are listed in Tables 31 through 33, along with gas volumes adjusted for pressure and temperature. These volumes range from a low of 173 B Cu Ft/unit to a maximum of 12.5 T Cu Ft/unit.

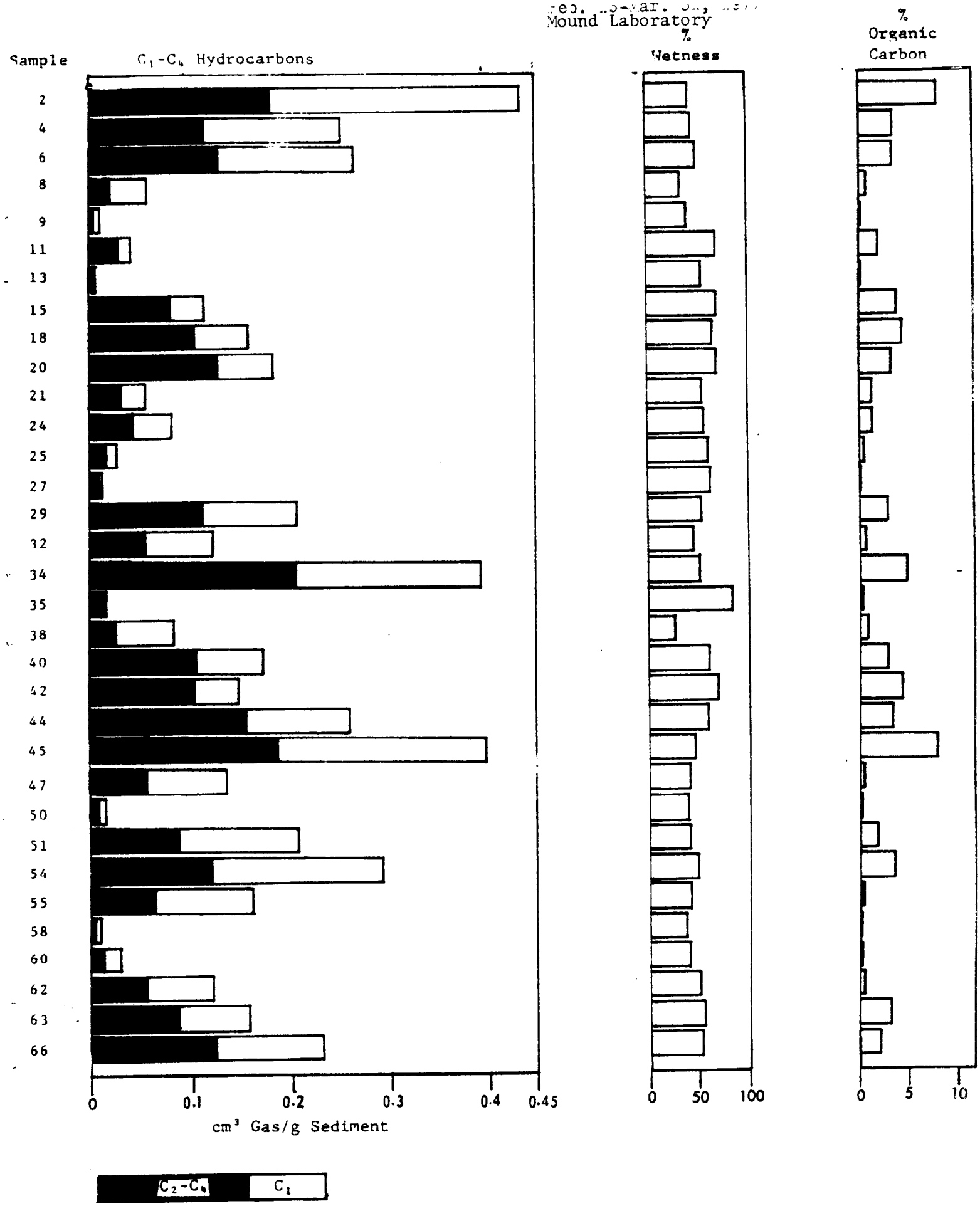
It is anticipated that the major portion of the detailed analyses will be completed during April, and well profile reports will be available in May.

I. FINANCIAL DATA

These data will be forwareded directly to the Project Office.

J. TRAVEL

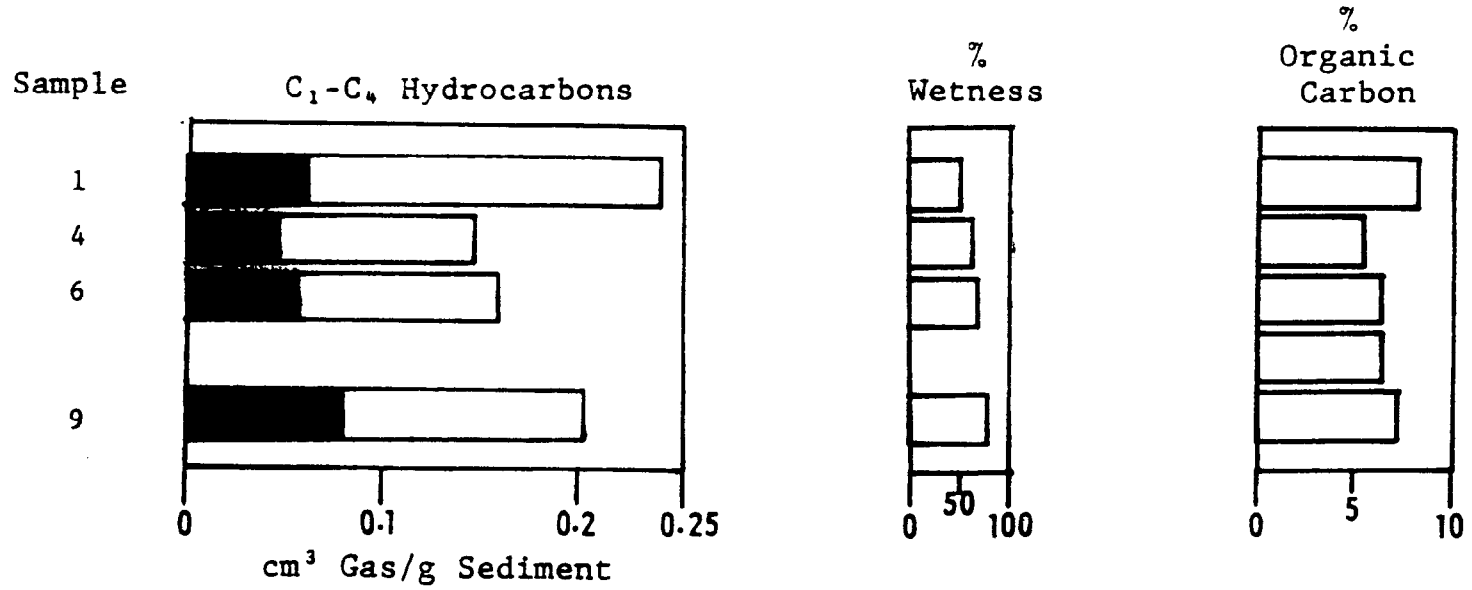
No travel was recorded for March.



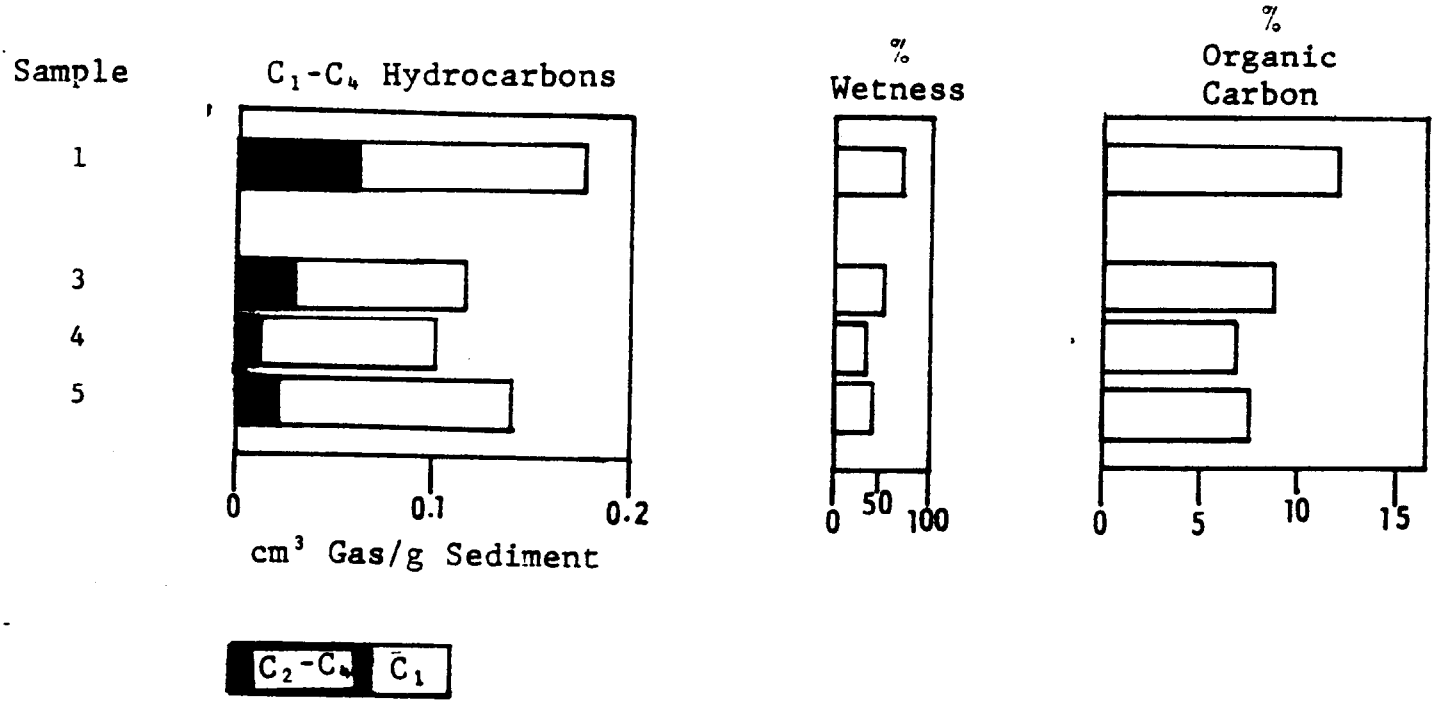
Organic Analyses of KY-2 Well

Figure 28

I-1 Well



O-1 Well



Organic Analysis of I-1 and O-1 Wells

Figure 29

TABLE 21

Sample Identification and Organic Carbon Content

Mound Sample Identification	Depth Interval	MERC Sample Number	Percent Organic Carbon
KY-2- 2	2443.35 - 2443.60	C-336-2444B	7.75
KY-2- 4	2474.53 - 2474.85	C-336-2474B	3.21
KY-2- 6	2503.55 - 2503.80	C-336-2504B	3.35; 3.35R*
KY-2- 8	2535.33 - 2535.60	C-336-2534B	0.62
KY-2- 9	2554.50 - 2554.75	C-336-2555A	0.16
KY-2-11	2597.25 - 2597.56	C-336-2597A	1.58
KY-2-13	2627.22 - 2627.60	C-336-2627A	0.09
KY-2-15	2657.30 - 2657.67	C-336-2657A	3.76; 3.83R
KY-2-18	2687.57 - 2687.88	C-336-2687B	4.28
KY-2-20	2715.29 - 2715.66	C-336-2715B	3.24
KY-2-21	2745.16 - 2745.58	C-336.2745A	1.30
KY-2-24	2773.60 - 2773.85	C-336-2773B	1.44
KY-2-25	2803.40 - 2803.58	C-336-2803A	0.48; 0.46R
KY-2-27	2883.31 - 2883.50	C-336-2833A	0.18
KY-2-29	2861.45 - 2861.71	C-336-2861A	2.53
KY-2-32	2890.40 - 2890.65	C-336-2890B	0.64
KY-2-34	2921.50 - 2921.70	C-336-2921B	4.65; 4.64R
KY-2-35	2947.43 - 2947.71	C-336-2947A	0.33
KY-2-38	2977.60 - 2977.83	C-336-2977B	0.70; 0.69R
KY-2-40	3005.31 - 3005.67	C-336-3005B	2.30
KY-2-42	3035.65 - 3035.95	C-336-3035B	4.29
KY-2-44	3054.55 - 3054.90	C-336-3054B	3.42
KY-2-45	3085.20 - 3085.40	C-336-3085A	7.60
KY-2-47	3113.40 - 3113.67	C-336-3113A	0.40
KY-2-50	3143.39 - 3143.63	C-336-3143B	0.12; 0.12R
KY-2-51	3171.18 - 3171.50	C-336-3171A	1.88
KY-2-54	3201.25 - 3201.52	C-336-3201B	3.57
KY-2-55	3230.27 - 3230.50	C-336-3230A	0.25; 0.25R
KY-2-58	3260.46 - 3260.68	C-336-3260B	0.15
KY-2-60	3288.23 - 3288.50	C-336-3288A	0.14
KY-2-62	3328.56 - 3328.86	C-336-3328B	0.55; 0.55R
KY-2-63	3358.05 - 3358.56	C-336-3358A	3.24
KY-2-66	3386.35 - 3386.70	C-336-3386B	2.10

*R - Duplicate Sample Result.

TABLE 22

Sample Identification and Organic Carbon Content

Mound Sample Identification	Depth Interval	MERC Sample Number	Percent Organic Carbon
I-1-1	3023*	I-1-3023A	8.37
I-1-4	3044.10 - 3044.25	I-1-3044A	5.50
I-1-6	3057.30 - 3057.65	I-1-3057B	6.30; 6.30R**
I-1-8	3075.31 - 3075.50	I-1-3075B	6.36
I-1-9	3099.30 - 3099.51	I-1-3099A	7.32
0-1-1	2183.71 - 2184.08	0-1-2184	12.06
0-1-3	2261.02 - 2261.50	0-1-2261	8.64
0-1-4	2292.47 - 2292.91	0-1-2292	6.85; 6.84R
0-1-5	2317.21 - 2317.64	0-1-2317	7.47

*Sample interval not accurately determined at well site.
**R - Duplicate sample result.

TABLE 23

C_1-C_7 HYDROCARBON ANALYSES OF AIR SPACE

Mound Sample Identification	Depth Interval	Methane C_1 PPM	Ethane C_2 PPM	Propane C_3 PPM	Isobutane iC_4 PPM	Butane nC_4 PPM	Total C_5-C_7 PPM	Total C_1-C_4 PPM	Total C_2-C_4 PPM	Gas Wetness %	iC_4/nC_4
KY-2- 2	2443.35 - 2443.60	467810.0	159063.1	56930.1	1848.7	12569.6	5131.1	698222.4	230412.4	33.0	0.15
KY-2- 4	2474.53 - 2474.85	251514.8	68261.0	25424.5	1048.0	6496.2	3361.7	352744.7	101229.8	28.7	0.16
KY-2- 6	2503.55 - 2503.80	262270.6	89983.6	47381.2	2291.5	14729.0	8650.8	416656.1	154385.4	37.1	0.16
KY-2- 8	2535.33 - 2535.60	80201.8	18145.5	7725.9	399.9	2274.5	1371.2	108747.8	28546.0	26.2	0.18
KY-2- 9	2554.50 - 2554.75	14307.5	4003.9	1819.3	72.5	501.2	564.2	20704.6	6397.0	30.9	0.14
KY-2-11	2597.25 - 2597.56	3970.0	1450.9	905.6	53.3	338.6	494.9	6718.6	2748.5	40.9	0.16
KY-2-13	2627.22 - 2627.60	5060.6	1640.3	872.0	43.6	248.8	239.2	7865.4	2804.8	35.7	0.18
KY-2-15	2657.30 - 2657.67	32281.4	18145.6	13331.5	739.7	5599.9	4810.7	70098.3	37816.8	53.9	0.13
KY-2-18	2687.57 - 2687.88	42790.6	23572.1	15870.1	788.0	6161.2	4700.4	89182.1	46391.5	52.0	0.13
KY-2-20	2715.29 - 2715.66	111624.3	84096.5	62897.5	3006.1	19955.8	12377.3	281580.5	169956.1	60.4	0.15
KY-2-21	2745.16 - 2745.58	45732.8	17288.6	9853.9	511.8	3085.8	2408.0	76473.2	30740.3	40.2	0.17
KY-2-24	2773.60 - 2773.85	67482.1	22265.9	13091.5	720.5	4345.4	3822.4	107905.7	40423.5	37.5	0.17
KY-2-25	2803.40 - 2803.58	20322.0	10100.4	7449.8	473.4	2744.9	2420.8	41090.7	20768.7	50.5	0.17
KY-2-27	2833.31 - 2833.50	4358.8	1448.1	957.3	62.9	401.9	846.6	7227.2	2870.4	39.7	0.16
KY-2-29	2861.45 - 2861.71	177463.9	71819.8	10485.1	1963.3	12935.8	8666.8	274668.0	97204.1	35.4	0.15
KY-2-32	2890.40 - 2890.65	137694.0	38847.2	19281.6	968.7	5612.0	4197.5	202403.8	64709.7	32.0	0.17
KY-2-34	2921.50 - 2921.70	377918.2	169884.0	110502.2	5868.8	46075.3	38072.6	710248.5	332330.3	47.0	0.13
KY-2-35	2947.43 - 2947.71	3252.5	1618.1	1192.7	77.7	459.2	589.9	6600.3	3347.8	50.7	0.17
KY-2-38	2977.60 - 2977.83	142752.2	30798.4	14611.8	859.2	4520.6	4053.8	193452.3	50790.1	26.2	0.19
KY-2-40	3005.31 - 3005.67	99836.9	44268.2	24104.6	1108.9	7370.8	5357.4	176689.6	76852.6	43.5	0.15
KY-2-42	3035.65 - 3035.95	53683.7	36114.8	5184.1	1961.4	13957.0	4077.4	110901.2	57217.5	51.6	0.14
KY-2-44	3054.55 - 3054.90	209424.4	127223.0	82224.2	3464.5	22398.2	11127.8	444734.5	235310.0	52.9	0.15
KY-2-45	3085.20 - 3085.40	430389.4	159495.8	97085.0	4945.1	33357.3	21726.5	725272.7	294883.3	40.7	0.15
KY-2-47	3113.40 - 3113.67	172337.2	49470.6	30128.1	2587.1	14700.8	15458.4	269223.8	96886.6	36.0	0.18
KY-2-50	3143.39 - 3143.63	18958.2	4137.5	2229.8	126.1	801.2	1174.1	26253.0	7294.7	27.8	0.16
KY-2-51	3171.18 - 3171.50	281185.4	97681.9	45846.0	2142.3	11756.5	8131.8	438612.3	157426.8	35.9	0.18
KY-2-54	3201.25 - 3201.52	274707.2	127332.5	60229.7	2358.0	15265.5	8479.1	479893.1	205185.8	42.8	0.15
KY-2-55	3230.27 - 3270.50	211776.8	80059.7	41783.3	2102.3	11575.3	7208.0	347297.4	135520.6	39.0	0.18
KY-2-58	3260.46 - 3260.68	16261.4	3556.4	1489.1	60.1	438.1	705.9	21805.3	5543.8	25.4	0.14
KY-2-60	3288.23 - 3288.50	41142.7	13857.3	10276.6	664.6	3211.4	2792.6	69152.8	28010.0	40.5	0.21
KY-2-62	3328.56 - 3328.86	132942.5	62173.8	33041.8	1636.2	7730.3	4990.8	237542.7	104582.2	44.0	0.21
KY-2-63	3358.05 - 3358.56	159911.0	111796.3	7571.4	5435.3	27089.4	8811.4	311803.5	151892.5	48.7	0.20
KY-2-66	3386.35 - 3386.70	243188.8	119929.0	68926.0	3364.0	19345.7	14399.5	454753.7	211564.8	46.5	0.17

PPM values expressed as volumes of gas per million volumes of shale core.

TABLE 24

C_1-C_7 HYDROCARBON ANALYSES OF SHALE CORE

Mound Sample Identification	Depth Interval	Methane C_1 PPM	Ethane C_2 PPM	Propane C_3 PPM	Isobutane iC_4 PPM	Butane nC_4 PPM	Total C_5-C_7 PPM	Total C_1-C_4 PPM	Total C_2-C_4 PPM	Gas Wetness %	iC_4/nC_4
KY-2- 2	2443.35 - 2443.60	116679.2	97400.1	72074.0	4169.0	17854.2	6769.3	308176.5	191497.3	62.1	0.23
KY-2- 4	2474.53 - 2474.85	68463.3	69738.6	67524.5	4832.1	22068.1	13456.1	232626.7	164163.4	70.6	0.22
KY-2- 6	2503.55 - 2503.80	52642.8	59709.3	62918.4	4322.3	21266.6	11963.1	200859.7	148216.8	73.8	0.20
KY-2- 8	2535.33 - 2535.60	5391.0	6656.9	7984.1	766.3	3615.3	2214.0	24413.7	19022.7	77.9	0.21
KY-2- 9	2554.50 - 2554.75	883.9	1172.6	1448.0	102.3	681.0	578.0	4288.0	3404.0	79.4	0.15
KY-2-11	2597.25 - 2597.56	24896.4	24223.5	26494.8	2010.8	10364.9	7376.7	87990.6	63094.1	71.7	0.19
KY-2-13	2627.22 - 2627.60	1362.5	1520.6	1907.9	176.3	1093.8	1002.5	6061.3	4698.8	77.5	0.16
KY-2-15	2657.30 - 2657.67	48946.5	56189.0	65996.0	4491.0	23448.2	12866.4	199071.0	150124.4	75.4	0.19
KY-2-18	2687.57 - 2687.88	78130.6	79622.5	85595.9	5207.8	27396.8	13220.3	275953.7	197823.1	71.7	0.19
KY-2-20	2715.29 - 2715.66	20010.3	39529.1	60274.4	4567.0	23580.8	12862.9	147961.9	127951.6	86.5	0.19
KY-2-21	2745.16 - 2745.58	10608.1	12592.4	18316.4	1685.9	8802.3	7306.1	52005.4	41397.2	79.6	0.19
KY-2-24	2773.60 - 2773.85	12894.1	18116.3	27339.0	2397.0	12662.3	12195.6	73409.0	60514.8	82.4	0.19
KY-2-25	2803.40 - 2803.58	2051.5	3945.8	6914.7	883.5	4518.6	5459.1	18314.2	16262.7	88.8	0.20
KY-2-27	2833.31 - 2833.50	6659.6	4434.3	6013.1	837.6	4219.6	9201.0	22064.4	15404.8	69.8	0.20
KY-2-29	2861.45 - 2861.71	47005.7	53924.8	73810.3	5693.9	31939.8	24741.0	212374.6	165368.9	77.9	0.18
KY-2-32	2890.40 - 2890.65	14549.2	18987.0	29068.9	2820.9	14392.0	14278.1	79818.1	65268.9	81.8	0.20
KY-2-34	2921.50 - 2921.70	46666.2	49600.8	61952.7	6310.3	30627.8	23577.8	195157.8	148491.6	76.0	0.21
KY-2-35	2947.43 - 2947.71	2867.9	6446.7	13683.5	1741.9	8405.1	11848.6	33145.3	30277.3	91.3	0.21
KY-2-38	2977.60 - 2977.83	2341.8	1995.9	2659.3	280.2	1334.5	1685.3	8611.9	6270.1	72.8	0.21
KY-2-40	3005.31 - 3005.67	52890.0	60745.6	76361.9	5290.6	30325.0	19548.7	226243.2	173353.2	76.6	0.20
KY-2-42	3035.65 - 3035.95	48196.1	62497.0	82711.4	6528.3	33592.0	21895.1	233524.9	185328.8	79.4	0.19
KY-2-44	3054.55 - 3054.90	30375.2	44354.0	58369.9	4556.1	22905.4	13601.9	160560.8	130185.5	81.1	0.20
KY-2-45	3085.20 - 3085.40	44383.8	52630.9	64651.9	4824.6	23580.7	12634.8	190072.2	145688.3	76.6	0.20
KY-2-47	3113.40 - 3113.67	8445.8	8161.5	15064.4	2238.7	10993.6	12348.1	44904.0	36458.2	81.0	0.20
KY-2-50	3143.39 - 3143.63	1697.1	1707.5	2554.3	301.0	1819.6	2336.1	8079.7	6382.5	79.0	0.17
KY-2-51	3171.18 - 3171.50	2922.1	11161.0	21372.9	2057.2	10474.0	7487.6	47987.3	45065.1	93.9	0.20
KY-2-54	3201.25 - 3201.52	8896.8	24706.3	37922.7	2855.1	13872.6	7409.7	88253.7	79356.8	89.9	0.21
KY-2-55	3230.27 - 3230.50	8876.0	3912.6	7975.4	1059.7	5436.3	5980.5	27260.0	18384.0	67.0	0.19
KY-2-58	3260.46 - 3260.68	723.0	1212.8	1948.6	198.8	1219.9	1500.7	5303.2	4580.1	86.4	0.16
KY-2-60	3288.23 - 3288.50	179.2	219.3	739.7	184.0	1039.1	2440.3	2361.5	2182.3	92.4	0.18
KY-2-62	3328.56 - 3328.86	1536.3	6696.5	20473.0	2786.3	12910.8	12627.2	44402.1	42865.8	96.5	0.22
KY-2-63	3358.05 - 3358.56	3868.5	11445.4	25839.1	2763.6	13741.2	11692.8	57658.0	53789.5	93.3	0.20
KY-2-66	3386.35 - 3386.70	11863.5	20827.6	34490.0	3424.7	15909.9	14151.9	86515.8	74652.3	86.3	0.22

PPM values expressed as volumes of gas per million volumes of shale core

USGS File #C052/1/Q
Feb. 15-Mar. 31, 1977
Mound Laboratory

TABLE 25

C_1-C_7 HYDROCARBON ANALYSES OF AIR SPACE AND SHALE CORE

Mound Sample Identification	Depth Interval	Methane C_1 PPM	Ethane C_2 PPM	Propane C_3 PPM	Isobutane iC_4 PPM	Butane nC_4 PPM	Total C_5-C_7 PPM	Total C_1-C_4 PPM	Total C_2-C_4 PPM	Gas Wetness %	iC_4/nC_4
KY-2- 2	2443.35 - 2443.60	584489.2	256464.1	129004.1	6017.7	30423.8	11900.5	1006398.9	421909.8	41.9	0.20
KY-2- 4	2474.53 - 2474.85	319978.2	137999.6	92949.0	5880.2	28564.3	16817.8	585371.5	265393.3	45.3	0.21
KY-2- 6	2503.55 - 2503.80	314913.5	149693.0	110299.6	6613.9	35995.7	20613.9	617515.8	302602.2	49.0	0.18
KY-2- 8	2535.33 - 2535.60	85592.8	24802.4	15710.0	1166.2	5889.8	3585.2	133161.6	47568.7	35.7	0.20
KY-2- 9	2554.50 - 2554.75	15191.5	5176.5	3267.3	174.8	1182.2	1142.2	24992.6	9801.0	39.2	0.15
KY-2-11	2597.25 - 2597.56	28866.5	25674.4	27400.4	2064.1	10703.5	7871.6	94709.2	65842.7	69.5	0.19
KY-2-13	2627.22 - 2627.60	6423.1	3161.0	2779.9	219.9	1342.7	1241.7	13926.8	7503.6	53.9	0.16
KY-2-18	2687.57 - 2687.88	120921.2	103194.7	101466.0	5995.8	33558.0	17920.7	365135.9	244214.7	66.9	0.18
KY-2-20	2715.29 - 2715.66	131634.7	123625.7	123172.0	7573.2	43536.7	25240.3	429542.5	297907.7	69.4	0.17
KY-2-21	2745.16 - 2745.58	56341.0	29881.1	28170.4	2197.7	11888.2	9714.1	128478.6	72137.5	56.1	0.18
KY-2-24	2773.60 - 2773.85	80376.3	40382.2	40430.6	3117.6	17007.7	16080.0	181314.7	100938.3	55.7	0.18
KY-2-25	2803.40 - 2803.58	22373.5	14046.2	14364.6	1357.0	7263.5	7880.0	59405.0	37031.4	62.3	0.19
KY-2-27	2833.31 - 2833.50	11018.4	5782.4	6970.5	900.6	4621.6	10047.7	29293.6	18275.2	62.4	0.19
KY-2-29	2861.45 - 2861.71	224469.6	125744.6	84295.4	7657.3	44875.6	33407.8	487042.6	262573.0	53.9	0.17
KY-2-32	2890.40 - 2890.65	152243.3	57834.2	48351.6	3789.6	20004.0	18475.7	282221.9	129978.6	46.1	0.19
KY-2-34	2921.50 - 2921.70	424584.4	219484.8	172454.9	12179.1	76703.1	61650.4	905406.3	480821.9	53.0	0.16
KY-2-35	2947.43 - 2947.71	6120.5	8064.8	14876.3	1819.6	8864.3	12438.5	39745.7	33625.1	84.6	0.21
KY-2-38	2977.60 - 2977.83	145094.1	32794.3	17271.1	1139.5	5855.1	5739.2	202154.3	57060.2	28.2	0.19
KY-2-40	3005.31 - 3005.67	152726.9	105013.9	100466.5	7029.5	37695.8	24906.2	402932.8	250205.9	62.1	0.19
KY-2-42	3035.65 - 3035.95	101879.8	98611.9	87895.5	8489.7	47549.0	25972.6	344426.2	242546.3	70.4	0.18
KY-2-44	3054.55 - 3054.90	239799.7	171577.1	140594.2	8020.6	45303.6	24729.8	605295.3	365495.6	60.4	0.18
KY-2-45	3085.20 - 3085.40	474773.2	212126.7	161736.9	9769.7	56938.1	34361.4	915344.9	440571.6	48.1	0.17
KY-2-47	3113.40 - 3113.67	180783.0	57632.1	45192.5	4825.8	25694.4	27806.5	314127.8	133344.8	42.0	0.19
KY-2-50	3143.39 - 3143.63	20655.4	5845.0	4784.1	427.2	2620.8	3510.2	34332.7	13677.3	39.8	0.16
KY-2-51	3171.18 - 3171.50	284107.5	108842.9	67218.9	4199.5	22230.5	15619.5	486599.6	202492.0	41.6	0.19
KY-2-55	3230.27 - 3230.50	220652.8	83972.3	49758.7	3162.0	17011.6	13188.5	374557.4	153904.6	41.0	0.19
KY-2-58	3260.46 - 3260.68	16984.4	4769.2	3437.8	258.9	1658.0	2206.6	27108.5	10124.0	37.3	0.16
KY-2-60	3288.23 - 3288.50	41321.9	14076.6	11016.3	848.7	4250.6	5233.0	71514.4	30192.4	42.2	0.20
KY-2-62	3328.56 - 3328.86	134478.8	68869.3	53514.9	4422.6	20641.2	17618.0	281926.9	147448.1	52.3	0.21
KY-2-63	3358.05 - 3358.56	163779.6	123241.7	33410.6	8199.0	40830.6	20504.2	369461.6	205682.0	55.7	0.20
KY-2-66	3386.35 - 3386.70	255052.4	140756.6	103416.0	6788.7	35255.6	28551.4	541269.5	286217.1	52.9	0.19

PPM values expressed as volumes of gas per million volumes of shale core

Mound Laboratory

TABLE 26

C_1-C_7 HYDROCARBON ANALYSES OF AIR SPACE AND SHALE CORE

Mound Sample Identification	Depth Interval	Methane C_1 cm^3/gram	Ethane C_2 cm^3/gram	Propane C_3 cm^3/gram	Isobutane iC_4 cm^3/gram	Butane nC_4 cm^3/gram	Total C_5-C_7 cm^3/gram	Total C_1-C_4 cm^3/gram	Total C_2-C_4 cm^3/gram	Gas Wetness %	iC_4/nC_4
KY-2- 2	2443.35 - 2443.60	0.2538	0.1114	0.0561	0.0026	0.0132	0.0052	0.4371	0.1833	41.9	0.20
KY-2- 4	2474.53 - 2474.85	0.1390	0.0600	0.0404	0.0026	0.0124	0.0073	0.2544	0.1154	45.3	0.21
KY-2- 6	2503.55 - 2503.80	0.1368	0.0650	0.0479	0.0029	0.0156	0.0090	0.2682	0.1314	49.0	0.18
KY-2- 8	2535.33 - 2535.60	0.0372	0.0108	0.0068	0.0005	0.0026	0.0016	0.0579	0.0207	35.7	0.20
KY-2- 9	2554.50 - 2554.75	0.0066	0.0022	0.0014	0.0001	0.0005	0.0005	0.0108	0.0042	39.2	0.15
KY-2-11	2597.25 - 2597.56	0.0125	0.0111	0.0119	0.0009	0.0047	0.0034	0.0411	0.0286	69.5	0.19
KY-2-13	2627.22 - 2627.60	0.0028	0.0014	0.0012	0.0001	0.0006	0.0005	0.0061	0.0033	53.9	0.16
KY-2-15	2657.30 - 2657.67	0.0353	0.0323	0.0345	0.0023	0.0126	0.0077	0.1170	0.0817	69.8	0.18
KY-2-18	2687.57 - 2687.88	0.0525	0.0448	0.0441	0.0026	0.0146	0.0078	0.1586	0.1061	66.9	0.18
KY-2-20	2715.29 - 2715.66	0.0572	0.0537	0.0535	0.0033	0.0189	0.0110	0.1866	0.1294	69.4	0.17
KY-2-21	2745.16 - 2745.58	0.0245	0.0130	0.0122	0.0010	0.0052	0.0042	0.0559	0.0314	56.1	0.18
KY-2-24	2773.60 - 2773.85	0.0349	0.0175	0.0176	0.0014	0.0074	0.0070	0.0788	0.0439	55.7	0.18
KY-2-25	2803.40 - 2803-58	0.0097	0.0061	0.0062	0.0006	0.0032	0.0034	0.0258	0.0161	62.3	0.19
KY-2-27	2833.31 - 2833.50	0.0048	0.0025	0.0030	0.0004	0.0020	0.0044	0.0127	0.0079	62.4	0.19
KY-2-29	2861.45 - 2861.71	0.0975	0.0546	0.0366	0.0033	0.0195	0.0145	0.2115	0.1140	53.9	0.17
KY-2-32	2890.40 - 2890.65	0.0662	0.0251	0.0210	0.0016	0.0087	0.0080	0.1226	0.0564	46.1	0.19
KY-2-34	2921.50 - 2921.70	0.1846	0.0954	0.0749	0.0053	0.0333	0.0268	0.3935	0.2089	53.0	0.16
KY-2-35	2947.43 - 2947.71	0.0027	0.0035	0.0065	0.0008	0.0039	0.0054	0.0174	0.0147	84.6	0.21
KY-2-38	2977.60 - 2977.83	0.0630	0.0142	0.0075	0.0005	0.0025	0.0025	0.0877	0.0247	28.2	0.19
KY-2-40	3005.31 - 3005.67	0.0664	0.0456	0.0437	0.0031	0.0164	0.0108	0.1752	0.1088	62.1	0.19
KY-2-42	3035.65 - 3035.95	0.0443	0.0428	0.0382	0.0037	0.0207	0.0113	0.1497	0.1054	70.4	0.18
KY-2-44	3054.55 - 3054.90	0.1042	0.0746	0.0611	0.0035	0.0197	0.0107	0.2631	0.1589	60.4	0.18
KY-2-45	3085.20 - 3085.40	0.2063	0.0922	0.0703	0.0042	0.0247	0.0149	0.3977	0.1914	48.1	0.17
KY-2-47	3113.40 - 3113.67	0.0786	0.0250	0.0196	0.0021	0.0011	0.0121	0.1364	0.0578	42.0	0.19
KY-2-50	3143.39 - 3143.69	0.0090	0.0025	0.0021	0.0002	0.0011	0.0015	0.0149	0.0059	39.8	0.16
KY-2-51	3171.18 - 3171.50	0.1235	0.0473	0.0292	0.0018	0.0097	0.0068	0.2115	0.0880	41.6	0.19
KY-2-54	3201.25 - 3201.52	0.1232	0.0661	0.0426	0.0023	0.0127	0.0069	0.2469	0.1237	50.1	0.18
KY-2-55	3230.27 - 3230.50	0.0959	0.0365	0.0216	0.0013	0.0074	0.0057	0.1627	0.0668	41.0	0.19
KY-2-58	3260.46 - 3260.68	0.0074	0.0021	0.0015	0.0001	0.0007	0.0010	0.0118	0.0044	37.3	0.16
KY-2-60	3288.23 - 3288.50	0.0180	0.0061	0.0048	0.0004	0.0018	0.0023	0.0311	0.0131	42.2	0.20
KY-2-62	3328.56 - 3328.86	0.0584	0.0299	0.0233	0.0019	0.0090	0.0077	0.1225	0.0641	52.3	0.21
KY-2-63	3358.05 - 3358.56	0.0712	0.0536	0.0145	0.0036	0.0177	0.0089	0.1606	0.0894	55.7	0.20
KY-2-66	3386.35 - 3386.70	0.1108	0.0612	0.0449	0.0029	0.0153	0.0124	0.2351	0.1243	52.9	0.19

Values expressed as cubic centimeters of gas per gram of shale core

TABLE 27

C_1-C_7 HYDROCARBON ANALYSES OF AIR SPACE

Mound Sample Identification	Depth Interval	Methane C_1 PPM	Ethane C_2 PPM	Propane C_3 PPM	Isobutane iC_4 PPM	Butane nC_4 PPM	Total C_5-C_7 PPM	Total C_1-C_4 PPM	Total C_2-C_4 PPM	Gas Wetness %	iC_4/nC_4
I-1-1	3023*	419104.5	95779.5	32517.2	1143.7	8518.9	4701.2	557064.0	137959.5	24.8	0.13
I-1-4	3044.10 - 3044.25	211051.4	46838.8	20679.2	772.2	6065.7	5621.5	285407.5	74356.1	26.1	0.13
I-1-6	3057.30 - 3057.65	205710.3	45561.4	17933.1	632.1	5202.0	4228.6	275039.1	68328.8	25.2	0.12
I-1-8**	3075.31 - 3075.50	-	-	-	-	-	-	-	-	-	-
I-1-9	3099.30 - 3099.51	181249.2	34638.0	14344.7	504.8	4082.4	2763.0	234819.3	53570.1	22.8	0.12
0-1-1	2183.71 - 2184.08	263605.9	113517.8	25760.5	490.4	2885.9	550.9	406260.7	142654.7	35.1	0.17
0-1-3	2261.02 - 2261.50	4330.6	392.4	80.5	3.2	23.1	18.8	4829.9	499.3	10.3	0.14
0-1-4	2292.47 - 2292.91	57060.1	5456.4	146.7	37.1	35.0	90.7	62735.3	5675.2	9.0	1.06
0-1-5	2317.21 - 2317.64	117501.7	10617.9	293.6	82.5	87.3	191.6	128583.0	11081.3	9.0	0.95

* Depth not accurately identified at well site
** Sample container leaked

PPM values expressed as volumes of gas per million volumes of shale core

TABLE 28

C_1-C_7 HYDROCARBON ANALYSES OF SHALE CORE

Mound Sample Identification	Depth Interval	Methane C_1 PPM	Ethane C_2 PPM	Propane C_3 PPM	Isobutane iC_4 PPM	Butane nC_4 PPM	Total C_5-C_7 PPM	Total C_1-C_4 PPM	Total C_2-C_4 PPM	Gas Wetness %	iC_4/nC_4
I-1-1	3023*	925.4	695.3	536.3	28.5	136.8	58.1	2322.5	1397.1	60.2	0.21
I-1-4	3044.10 - 3044.25	15748.9	14914.5	15566.7	1048.9	5857.4	3900.7	53136.6	37387.6	70.4	0.18
I-1-6	3057.30 - 3057.65	31821.0	26635.1	24974.8	1445.4	7928.3	4093.6	92804.9	60983.9	65.7	0.18
I-1-8	3075.31 - 3075.50	57320.1	49109.9	47143.4	2690.2	13845.4	6832.8	170109.2	112789.0	66.3	0.19
I-1-9	3099.30 - 3099.51	100142.2	64745.4	52134.3	2417.4	12607.1	4631.4	232046.6	131904.4	56.8	0.19
0-1-1	2183.71 - 2184.08	643.9	624.5	330.2	10.6	53.8	11.9	1663.2	1019.3	61.3	0.20
0-1-3	2261.02 - 2261.50	198434.6	47961.6	16200.8	1007.6	4122.7	1681.7	267727.6	69292.9	25.9	0.24
0-1-4	2292.47 - 2292.91	141356.7	26309.5	1494.2	737.0	284.8	706.7	170182.2	28825.5	16.9	2.59
0-1-5	2317.21 - 2317.64	163085.5	34877.5	2628.1	1457.6	899.4	1880.1	202948.1	39862.6	19.6	1.62

* Depth not accurately identified at well site

PPM values expressed as volumes of gas per million volumes of shale core

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TABLE 29

C_1-C_7 HYDROCARBON ANALYSES OF AIR SPACE AND SHALE CORE

Mound Sample Identification	Depth Interval	Methane C_1 PPM	Ethane C_2 PPM	Propane C_3 PPM	Isobutane iC_4 PPM	Butane nC_4 PPM	Total C_5-C_7 PPM	Total C_1-C_4 PPM	Total C_2-C_4 PPM	Gas Wetness %	iC_4/nC_4
I-1-1	3023*	420029.9	96474.9	33053.6	1172.3	8655.8	4759.4	559386.6	139356.7	24.9	0.14
I-1-4	3044.10 - 3044.25	226800.4	61753.3	36246.0	1821.2	11923.1	9522.2	338544.1	111743.7	33.0	0.15
I-1-6	3057.30 - 3057.65	237531.3	72196.5	42908.0	2077.6	13130.4	8322.2	367844.1	130312.7	35.4	0.16
I-1-8**	3075.31 - 3075.50	-	-	-	-	-	-	-	-	-	-
I-1-9	3099.30 - 3099.51	281391.4	99383.4	66479.1	2922.2	16689.6	7394.4	466866.0	185475.5	39.7	0.18
0-1-1	2183.71 - 2184.08	264249.8	114142.4	26090.7	501.1	2939.8	562.8	407924.0	143674.1	35.2	0.17
0-1-3	2261.02 - 2261.50	202765.3	48354.1	16281.4	1010.8	4145.9	1700.6	272557.6	69792.3	25.6	0.24
0-1-4	2292.47 - 2292.91	198416.8	31765.9	1640.9	774.1	319.8	797.4	232917.5	34500.7	14.8	2.42
0-1-5	2317.21 - 2317.64	280587.2	45495.4	2921.7	1540.1	986.7	2071.7	331531.1	50943.9	15.4	1.56

* Depth not accurately identified at well site
** Sample container leaked

PPM values expressed as volumes of gas per million volumes of shale core

TABLE 30

C_1-C_7 HYDROCARBON ANALYSES OF AIR SPACE AND SHALE CORE

Mound Sample Identification	Depth Interval	Methane C_1 cm^3/gram	Ethane C_2 cm^3/gram	Propane C_3 cm^3/gram	Isobutane iC_4 cm^3/gram	Butane nC_4 cm^3/gram	Total C_5-C_7 cm^3/gram	Total C_1-C_4 cm^3/gram	Total C_2-C_4 cm^3/gram	Gas Wetness %	iC_4/nC_4
I-1-1	3023*	0.1825	0.0419	0.0144	0.0005	0.0038	0.0021	0.2431	0.0606	24.9	0.14
I-1-4	3044.10 - 3044.25	0.0985	0.0268	0.0157	0.0008	0.0052	0.0041	0.1470	0.0485	33.0	0.15
I-1-6	3057.30 - 3057.65	0.1032	0.0314	0.0186	0.0009	0.0057	0.0036	0.1598	0.0566	35.4	0.16
I-1-8**	3075.31 - 3075.50	--	--	--	--	--	--	--	--	-	-
I-1-9	3099.30 - 3099.51	0.1223	0.0432	0.0289	0.0013	0.0073	0.0032	0.2030	0.0807	39.7	0.18
0-1-2	2183.71 - 2184.08	0.1148	0.0496	0.0113	0.0002	0.0013	0.0002	0.1772	0.0624	35.2	0.17
0-1-3	2261.02 - 2261.50	0.0881	0.0210	0.0071	0.0004	0.0018	0.0007	0.1184	0.0303	25.6	0.24
0-1-4	2292.47 - 2292.91	0.0862	0.0138	0.0007	0.0003	0.0001	0.0003	0.1011	0.0149	14.8	2.42
0-1-5	2317.21 - 2317.64	0.1220	0.0197	0.0012	0.0006	0.0004	0.0009	0.1439	0.0219	15.4	1.56

* Depth not accurately identified at well site
** Sample container leaked

Values expressed as cubic centimeters of gas per gram of shale core

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TABLE 31

ESTIMATED INDIGENOUS GAS CONTENT

Sample No.	KY-2-2	KY-2-4	KY-2-6	KY-2-8	KY-2-9	KY-2-11	KY-2-13	KY-2-15	KY-2-18	KY-2-20
Depth	2443	2474	2503	2535	2554	2597	2627	2657	2687	2715
Predicted Indigenous Gas										
Average Percent Organic Carbon	7.75	3.21	3.35; 3.35	0.62	0.16	1.58	0.09	3.76; 3.83	4.28	3.24
Average Percent Organic Matter	9.45	3.91	4.08	0.75	0.19	1.92	0.11	4.63	5.22	3.95
Mott Factor (ft^3 gas/ton organic matter)	1350	1350	1350	1350	1350	1350	1350	1350	1350	1350
Indigenous Gas (ft^3 gas/ton sediment)	127.43	52.78	55.08	10.19	2.63	25.98	1.47	61.82	70.37	53.27
Indigenous Gas (MMcf/unit)*	254901	105578	110183	20392	5262	51967	2960	123668	140771	106565
Existing Indigenous Gas										
Average C_1-C_4 Hydrocarbon Content (cm^3/g)	0.4371	0.2544	0.2682	0.0579	0.0108	0.0411	0.0061	0.1170	0.1586	0.1866
Indigenous Gas (ft^3 gas/ton sediment)	14.00	8.14	8.59	1.85	0.35	1.32	0.19	3.74	5.08	5.98
Indigenous Gas (MMcf/unit)*	28057	16319	17215	3712	696	2640	388	7504	10179	11975
Volume gas adjusted for pressure and temperature (B Cu.Ft./unit)*	1251.0	727.6	767.6	165.5	31.0	117.7	17.3	334.6	435.8	533.9

* A unit of a volume of sediment: 1 mile X 1 mile x 1000 feet.

TABLE 31 (continued)

ESTIMATED INDIGENOUS GAS CONTENT

Sample No.	KY-2-21	KY-2-24	KY-2-25	KY-2-27	KY-2-29	KY-2-32	KY-2-34	KY-2-35	KY-2-38	KY-2-40
Depth	2745	2773	2803	2833	2861	2890	2921	2947	2977	3005
Predicted Indigenous Gas										
Average Percent Organic Carbon	1.30	1.44	0.48; 0.46	0.18	2.53	0.64	4.65; 4.64	0.33	0.70; 0.69	2.30
Average Percent Organic Matter	1.58	1.75	0.57	0.22	3.08	0.78	5.68	0.40	0.84	2.80
Mott Factor (ft^3 gas/ton organic matter)	1350	1350	1350	1350	1350	1350	1350	1350	1350	1350
Indigenous Gas (ft^3 gas/ton sediment)	21.37	23.67	7.89	2.95	41.60	10.52	76.4	5.42	11.51	37.82
Indigenous Gas (MMcf/unit)*	42757	47362	15787	5920	83213	21049	153000	10853	23023.3	75648
Existing Indigenous Gas										
Average C_1-C_4 Hydrocarbon Content (cm^3 /g)	0.0559	0.0788	0.0258	0.0127	0.2115	0.1226	0.3936	0.0174	0.0877	0.1752
Indigenous Gas (ft^3 gas/ton sediment)	1.79	2.52	0.83	0.41	6.78	3.93	12.60	0.55	2.81	5.61
Indigenous Gas (MMcf/unit)*	3581	5055	1656	816	13578	7868	25241	1108	5636	11233
Volume gas adjusted for pressure and temperature (B Cu.Ft./unit)*	159.7	225.4	73.8	36.4	605.4	350.8	1125.4	49.4	251.3	500.8

* A unit consists of a volume of sediment: 1 mile x 1 mile x 1000 feet.

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TABLE 31 (continued)

ESTIMATED INDIGENOUS GAS CONTENT

Sample No.	KY-2-42	KY-2-44	KY-2-45	KY-2-47	KY-2-50	KY-2-51	KY-2-54	KY-2-55	KY-2-58	KY-2-60
Depth	3035	3054	3085	3113	3143	3171	3201	3230	3260	3288
Predicted Indigenous Gas										
Average Percent Organic Carbon	4.29	3.42	4.60	0.40	0.12; 0.12	1.88	3.57	0.25; 0.25	0.15	0.14
Average Percent Organic Matter	5.23	4.17	5.61	0.49	0.14	2.29	4.35	0.30	0.18	0.17
Mott Factor (ft^3 gas/ton organic matter)	1350	1350	1350	1350	1350	1350	1350	1350	1350	1350
Indigenous Gas (ft^3 gas/ton sediment)	70.54	56.23	75.64	6.57	1.97	30.91	58.70	4.11	2.46	2.30
Indigenous Gas (MMcf/unit)*	14110	112485	151296	13200	3946	61834	117419	8200	4933	4604
Existing Indigenous Gas										
Average C_1-C_4 Hydrocarbon Content (cm^3/g)	0.1497	0.2631	0.3977	0.1364	0.0149	0.2115	0.2469	0.1627	0.0118	0.0311
Indigenous Gas (ft^3 gas/ton sediment)	4.79	8.42	12.73	4.37	0.48	6.77	7.90	5.21	0.38	0.99
Indigenous Gas (MMcf/unit)*	9602	16875	25518	8757	957	13565	15839	10442	755	1993
Volume gas adjusted for pressure and temperature (BCu.Ft./unit)*	428.1	752.4	1137.8	390.4	42.7	604.8	706.2	465.5	33.7	88.9

* A unit consists of a volume of sediment: 1 mile x 1 mile x 1000 feet.

TABLE 31 (continued)

ESTIMATED INDIGENOUS GAS CONTENT

Sample No.	KY-2-62	KY-2-63	KY-2-64
Depth	3328	3358	3386
Predicted Indigenous Gas			
Average Percent Organic Carbon	0.55; 0.55	3.24	2.10
Average Percent Organic Matter	0.67	3.95	2.56
Mott Factor (ft^3 gas/ton organic matter)	1350	1350	1350
Indigenous Gas (ft^3 gas/ton sediment)	9.04	53.27	34.53
Indigenous Gas (MMcf/unit)*	18089	106565	69070
Existing Indigenous Gas			
Average C_1-C_4 Hydrocarbon Content (cm^3/g)	0.1225	0.1606	0.2351
Indigenous Gas (ft^3 gas/ton sediment)	3.92	5.14	7.53
Indigenous Gas (MMcf/unit)*	7860	10300	15090
Volume gas adjusted for pressure and temperature (B Cu.Ft./unit)*	350.4	459.2	672.8

*A unit consists of a volume of sediment: 1 mile x 1 mile x 1000 feet.

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TABLE 32

ESTIMATED INDIGENOUS GAS CONTENT

Sample No.	I-1-1	I-1-4	I-1-6	I-1-8	I-1-9
Depth	3023	3044	3057	3075	3099
Predicted Indigenous Gas					
Average Percent Organic Carbon	8.37	5.50	6.30; 6.30	6.36	7.32
Average Percent Organic Matter	10.21	6.71	7.68	7.76	8.93
Mott Factor (ft^3 gas/ton organic matter)	1350	1350	1350	1350	1350
Indigenous Gas (ft^3 gas/ton sediment)	137.63	90.44	103.50	104.58	120.36
Indigenous Gas (MMcf/unit)*	275293	280897	207210	209183	240758
Existing Indigenous Gas					
Average C_1-C_4 Hydrocarbon Content (cm^3/g)	0.2431	0.1470	0.1598	0.0739**	0.2030
Indigenous Gas (ft^3 gas/ton sediment)	7.78	4.71	5.12	2.37**	6.49
Indigenous Gas (MMcf/unit)*	15595	9438	10255	4742**	13016
Volume gas adjusted for pressure and temperature (B Bu.Ft./unit)*	695.3	420.8	457.2	211.4	580.3

** Sample I-1-8 can opened in transit. No air space analysis.

* A unit consists of a volume of sediment: 1 mile x 1 mile x 1000 feet.

TABLE 33

ESTIMATED INDIGENOUS GAS CONTENT

Sample No.	0-1-1	0-1-3	0-1-4	0-1-5
Depth	2183	2261	2292	2317
Predicted Indigenous Gas				
Average Percent Organic Carbon	12.06	8.64	6.85; 6.84	7.47
Average Percent Organic Matter	14.71	10.54	8.37	9.13
Mott Factor (ft^3 gas/ton matter)	1350	1350	1350	1350
Indigenous Gas (ft^3 gas/ton sediment)	198.31	142.07	112.64	122.84
Indigenous Gas (MMcf/unit)*	396659	284174	225300	245600
Existing Indigenous Gas				
Average C_1-C_4 Hydrocarbon Content (cm^3/g)	0.1772	0.1184	0.1011	0.1439
Indigenous Gas (ft^3 gas/ton sediment)	5.67	3.79	3.24	4.61
Indigenous Gas (MMcf/unit)*	11372	7598	649	924
Volume gas adjusted for pressure and temperature (B Cu.Ft./unit)*	507.1	338.8	29.0	41.2

* A unit consists of a volume of sediment: 1 mile x 1 mile x 1000 feet.

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TABLE A19

Shale Samples from the PA-2 Well
located in Allegheny County, Pennsylvania

Mound Sample No.	Depth, Ft.
PA-2-1	6962.16/.37
PA-2-2	6982.31/.60
PA-2-3	7002.36/.56
PA-2-4	7020.42/.62
PA-2-5	7040.31/.55
PA-2-6	7060.46/.70
PA-2-7	7079.19/.42
PA-2-8	7099.29/.56
PA-2-9	7119.39/.62
PA-2-10	7198.25/.47
PA-2-11	7218.12/.34
PA-2-12	7238.20/.42
PA-2-13	7258.33/.61
PA-2-14	7278.57/.78
PA-2-15	7298.46/.74
PA-2-16	7319.40/.65
PA-2-17	7339.44/.70
PA-2-18	7387.37/.55
PA-2-19	7402.31/.61
PA-2-20	7422.28/.54
PA-2-21	7442.22/.47
PA-2-22	7462.42/.64
PA-2-23	7482.44/.60

TABLE A20

Shale Samples from the OH-3 Well,
Knox County, Ohio

Mound Sample Number	Depth, Feet
OH-3-1	574.39/.81
OH-3-2	604.16/.36
OH-3-3	633.63/.94
OH-3-4	664.37/.61
OH-3-5	684.29/.55
OH-3-6	704.22/.58
OH-3-7	722.33/.59
OH-3-8	742.19/.42
OH-3-9	762.37/.56
OH-3-10	780.36/.60
OH-3-11	800.47/.72
OH-3-12	820.18/.49
OH-3-13	838.36/.72
OH-3-14	858.35/.64
OH-3-15	878.47/.68
OH-3-16	896.32/.61
OH-3-17	916.32/.52
OH-3-18	936.32/.61
OH-3-19	944.90/945.13
OH-3-20	960.85/961.14
OH-3-21	990.70/991.03
OH-3-22	1011.10/.24
OH-3-23	1031.39/.75
OH-3-24	1051.51/.76
OH-3-25	1074.40/.69
OH-3-26	1092.58/.86
OH-3-27	1112.58/.86
OH-3-28	1130.43/.71
OH-3-29	1150.17/.50
OH-3-30	1188.48/.73
OH-3-31	1208.18/.42
OH-3-32	1228.07/.41
OH-3-33	1247.32/.41
OH-3-34	

TABLE A21

Bessmer & Lake Erie Railroad Co.
and
Monsanto Research Corporation

Mound Sample No.	Depth (feet)	Comments
OH-4-1	509 (.72)	D/CCO
OH-4-2	510 (.44-.83)	C/CCO #1
OH-4-3	514 (.75)	D/CCO
OH-4-4	516 (.21-.53)	C/CCO*
OH-4-5	520 (.25-.43)	C/CCO #2
OH-4-6	520 (.43)	D/CCO
OH-4-7	525 (.95)	D/CCO
OH-4-8	527 (.1)	D/CCO
OH-4-9	529 (.47-.83)	C/CCO #3
OH-4-10	530 (.75)	D/CCO
OH-4-10-A	534 (.85)	**
OH-4-10-B	538 (.86)	**
OH-4-11	539 (.33-.59)	C/CCO #4
OH-4-12	541 (.39-.69)	C/CCO*
OH-4-13	541 (.95)	D/CCO
OH-4-14	546 (.15)	D/CCO
OH-4-15	549 (.36-.64)	C/CCO #5
OH-4-16	550 (.1)	D/CCO
OH-4-17	554 (.12)	D/CCO
OH-4-18	558 (.03)	D/CCO
OH-4-19	559 (.69-.92)	C/CCO #6
OH-4-20	563 (.12)	D/CCO
OH-4-21	564 (.47-.79)	C/CCO*
OH-4-21-A	565 (.91)	**
OH-4-22	566-571	D/CT
OH-4-23	571-581	D/CT
OH-4-24	581-591	D/CT
OH-4-25	591-601	D/CT
OH-4-26	601-611	D/CT
OH-4-27	611-621	D/CT
OH-4-28	621-631	D/CT
OH-4-29	631-641	D/CT
OH-4-30	641-651	D/CT no sample
OH-4-31	651-661	D/CT no sample
OH-4-32	661-671	D/CT no sample
OH-4-33	671-681	D/CT
OH-4-34	681-691	D/CT
OH-4-35	691-701	D/CT

C = Canned Samples
D = Bagged Dry Samples
PCO = Pressure
CCO = Conventional Core
CT = Cutting
* = presently being tested in field, not available for detailed analysis
** = presently being analyzed for TOC at Mound

TABLE A21 (Continued)
Bessemer & Lake Erie Railroad Co. and Monsanto Research Corporation

Mound Sample No.	Depth (feet)	Comments
OH-4-36	701-711	D/CT
OH-4-37	711-721	D/CT
OH-4-38	721-731	D/CT
OH-4-39	731-741	D/CT
OH-4-40	741-746	D/CT
OH-4-41	736	C/PCO pressure core chip
OH-4-42	747 (.35-.36)	D/CCO
OH-4-43	748	C/CCO #7
OH-4-44	748 (.55-.85)	C/CCO*
OH-4-45	750 (.55-.57)	D/CCO
OH-4-46	751 (.15-.44)	C/CCO*
OH-4-46-A	755 (.10)	**
OH-4-47	758 (.42-.64)	C/CCO #8
OH-4-48	759 (.27-.30)	D/CCO
OH-4-49	762 (.89-.91)	D/CCO
OH-4-49-A	767 (.56)	**
OH-4-50	768 (.30-.58)	C/CCO #9
OH-4-51	770 (.90-.91)	D/CCO
OH-4-51-A	773 (.58)	**
OH-4-52	777 (.10-.41)	C/CCO*
OH-4-53	778 (.51-.78)	C/CCO #10
OH-4-54	780 (.65-.66)	D/CCO
OH-4-55	784 (.14-.15)	D/CCO
OH-4-56	786 (.79-.81)	D/CCO
OH-4-57	789 (.10-.43)	C/CCO #11
OH-4-57-A	791 (.71)	**
OH-4-58	794	D/CCO
OH-4-58-A	798 (.64)	**
OH-4-59	799 (.05-.32)	C/CCO #12
OH-4-60	802 (.77-.79)	D/CCO
OH-4-61	807 (.45-.47)	D/CCO
OH-4-62	809 (.13-.43)	C/CCO #13
OH-4-63	811 (.50-.52)	D/CCO
OH-4-63-A	815 (.48)	**
OH-4-64	816 (.10-.44)	C/CCO*
OH-4-65	819 (.36-.54)	C/CCO #14
OH-4-66	823 (.21-.23)	D/CCO
OH-4-67	825 (.19-.47)	C/CCO*
OH-4-68	825 (.47-.87)	C/CCO*
OH-4-69	826	C/PCO-1-1
OH-4-69-A	826 (.30)	**
OH-4-70	827	C/PCO-1-2
OH-4-71	828	C/PCO-1-3
OH-4-72	829	C/PCO-1-4
OH-4-73	830	C/PCO-1-5
OH-4-74	831	C/PCO-1-6
OH-4-75	832	C/PCO-1-7

TABLE A21 (Continued)

Bessemer & Lake Erie Railroad Co. and Monsanto Research Corporation

Mound Sample No.	Depth (feet)	Comments
OH-4-76	833	C/PCO-1-8
OH-4-77	834	C/PCO-1-9
OH-4-78	835	C/PCO-1-10
OH-4-79	836 (.00-.35)	C/CCO*
OH-4-80	837 (.49-.75)	C/CCO #15
OH-4-81	838 (.52-.55)	D/CCO
OH-4-82	841 (.35-.37)	D/CCO
OH-4-83	843 (.19-.46)	C/CCO*
OH-4-84	846 (.15-.17)	D/CCO
OH-4-85	848 (.27-.61)	C/CCO #16
OH-4-85-A	850 (.33)	**
OH-4-86	853 (.79-.81)	D/CCO
OH-4-87	857 (.75-.77)	D/CCO
OH-4-88	858 (.36-.56)	C/CCO #17
OH-4-89	861 (.42-.77)	C/CCO*
OH-4-90	863 (.50-.52)	D/CCO
OH-4-91	868 (.49-.51)	D/CCO
OH-4-92	868 (.51-.75)	C/CCO #18
OH-4-93	872 (.22-.24)	D/CCO
OH-4-93-A	875 (.8)	**
OH-4-94	876-886	D/CT
OH-4-95	886-896	D/CT
OH-4-96	896-906	D/CT
OH-4-97	906-916	D/CT
OH-4-98	916-926	D/CT
OH-4-99	926-936	D/CT
OH-4-100	936-946	D/CT
OH-4-101	950 (.80-.82)	D/CCO
OH-4-102	951 (.25-.53)	C/CCO #19
OH-4-103	953 (.22-.51)	C/CCO*
OH-4-104	954 (.23-.26)	D/CCO
OH-4-105	957 (.64-.67)	D/CCO
OH-4-106	961 (.17-.45)	C/CCO #20
OH-4-107	962 (.35-.37)	D/CCO
OH-4-108	965 (.59-.62)	D/CCO
OH-4-108-A	969 (.26)	**
OH-4-109	971 (.42-.68)	C/CCO #21
OH-4-110	973 (.49-.53)	D/CCO
OH-4-111	977 (.60-.64)	D/CCO
OH-4-112	981 (.08-.47)	C/CCO*
OH-4-113	981 (.47-.76)	C/CCO #22
OH-4-114	981 (.76-.78)	D/CCO
OH-4-115	982 (.74-.78)	D/CCO
OH-4-116	986 (.16-.19)	D/CCO
OH-4-116-A	988 (.37)	**
OH-4-117	989-999	D/CT
OH-4-118	999-1009	D/CT
OH-4-119	1029-1039	D/CT
OH-4-120	1039-1054	D/CT

TABLE A21 (Continued)

Bessemer & Lake Erie Railroad Co. and Monsanto Research Corporation

Mound Sample No.	Depth (feet)	Comments
OH-4-121	1054-1059	D/CT
OH-4-122	1059-1069	D/CT
OH-4-123	1070 (.31-.33)	D/CCO
OH-4-124	1071 (.40-.66)	C/CCO #23
OH-4-125	1073 (.55-.58)	D/CCO
OH-4-126	1076 (.43-.77)	C/CCO*
OH-4-127	1077 (.65-.70)	D/CCO
OH-4-128	1081 (.14-.39)	C/CCO #24
OH-4-129	1081 (.39-.42)	D/CCO
OH-4-130	1085 (.38-.43)	D/CCO
OH-4-130-A	1089 (.17)	**
OH-4-131	1091 (.45-.71)	C/CCO #25
OH-4-132	1093 (.39-.42)	D/CCO
OH-4-133	1096 (.78-.81)	D/CCO
OH-4-134	1098 (.30-.60)	C/CCO*
OH-4-135	1100 (.58-.61)	D/CCO
OH-4-136	1101 (.23-.56)	C/CCO #26
OH-4-137	1104 (.67-.69)	D/CCO
OH-4-138	1107 (.82-.85)	D/CCO
OH-4-139	1109-1119	D/CT
OH-4-139-A	1119 (.30)	**
OH-4-140	1121 (.49-.74)	C/CCO #27
OH-4-141	1123 (.41-.43)	D/CCO
OH-4-142	1125 (.31-.58)	C/CCO*
OH-4-143	1127 (.86-.89)	D/CCO
OH-4-144	1131 (.28-.51)	C/CCO #28
OH-4-145	1131 (.51-.54)	D/CCO
OH-4-146	1134 (.68-.71)	D/CCO
OH-4-147	1137 (.37-.40)	D/CCO
OH-4-147-A	1140 (.22)	**
OH-4-148	1141 (.15-.49)	C/CCO*
OH-4-149	1141 (.49-.75)	C/CCO #29
OH-4-150	1143 (.65-.67)	D/CCO
OH-4-151	1147 (.21-.25)	D/CCO
OH-4-152	1151 (.16-.42)	C/CCO #30
OH-4-153	1153 (.42-.44)	D/CCO
OH-4-154	1155 (.43-.46)	D/CCO
OH-4-155	1158 (.77-.1.21)	C/CCO*
OH-4-156	1159 (.28-.31)	D/CCO
OH-4-157	1159	C/PCO-2-1
OH-4-158	1160	C/PCO-2-2
OH-4-159	1161	C/PCO-2-3
OH-4-160	1162	C/PCO-2-4
OH-4-161	1163	C/PCO-2-5
OH-4-162	1164	C/PCO-2-6
OH-4-162-A	1169 (.0.84)	**
OH-4-163	1169 (.15-.40)	C/CCO
OH-4-164	1171	C/CCO #31
OH-4-165	1174 (.22-.25)	D/CCO

TABLE A21 (Continued)

Bessemer & Lake Erie Railroad Co. and Monsanto Research Corporation

Mound Sample No.	Depth (feet)	Comments
OH-4-166	1178 (.47-.51)	D/CCO
OH-4-167	1181 (.26-.57)	C/CCO #32
OH-4-168	1181 (.74-.77)	D/CCO
OH-4-169	1184 (.36-.75)	C/CCO*
OH-4-170	1186 (.56-.59)	D/CCO
OH-4-171	1189 (.65-.68)	D/CCO
OH-4-172	1191 (.38-.61)	C/CCO #33
OH-4-172-A	1193 (.36)	**
OH-4-173	1197 (.34-.37)	D/CCO
OH-4-174	1199 (.07-.38)	C/CCO*
OH-4-175	1201 (.19-.40)	C/CCO #34
OH-4-176	1201 (.66-.69)	D/CCO
OH-4-177	1205 (.31-.34)	D/CCO
OH-4-178	1208 (.52-.55)	D/CCO
OH-4-179	1209-1219	D/CT
OH-4-180	1249-1259	D/CT
OH-4-181	1269-1289	D/CT
OH-4-182	1289-1309	D/CT
OH-4-183	1309 (.82-.92)	D/CCO
OH-4-184	1311 (.12-.40)	C/CCO #35
OH-4-184-A	1313 (.67)	**
OH-4-185	1315 (.43-.76)	C/CCO*
OH-4-186	1317 (.34-.38)	D/CCO
OH-4-187	1321 (.41-.44)	D/CCO
OH-4-188	1321 (.58-.82)	C/CCO #36
OH-4-189	1325 (.68-.71)	D/CCO
OH-4-190	1329 (.25-.27)	D/CCO
OH-4-191	1331 (.45-.70)	C/CCO #37
OH-4-192	1333 (.26-.30)	D/CCO
OH-4-192-A	1337 (.27)	**
OH-4-193	1340 (.39-.82)	C/CCO*
OH-4-194	1341 (.23-.26)	D/CCO
OH-4-195	1341 (.40-.65)	C/CCO #38
OH-4-196	1345 (.11-.15)	D/CCO
OH-4-197	1349 (.18-.20)	D/CCO
OH-4-198	1350 (.54-.56)	D/CCO
OH-4-199	1351 (.17-.50)	C/CCO #39
OH-4-200	1354 (.38-.40)	D/CCO
OH-4-201	1358 (.35-.68)	C/CCO*
OH-4-201-A	1359 (.27)	**
OH-4-202	1361 (.29-.58)	C/CCO #40
OH-4-203	1362 (.38-.41)	D/CCO
OH-4-204	1366 (.52-.55)	D/CCO
OH-4-205	1370 (.56-.60)	D/CCO
OH-4-206	1371 (.24-.58)	C/CCO #41
OH-4-207	1373 (.08-.42)	C/CCO*
OH-4-208	1374 (.29-.32)	D/CCO
OH-4-209	1379 (.12-.15)	D/CCO
OH-4-209-A	1383 (.54)	**

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