UGR File #C051/78Q1
Oct.-Dec. 1977
Mound Facility

MLM-ML-78-41-0002
MLM-EGSP-TPR-Q-004

# PHYSICAL AND CHEMICAL CHARACTERIZATION OF DEVONIAN GAS SHALE

QUARTERLY STATUS REPORT (OCTOBER 1 - DECEMBER 31, 1977)

PREPARED BY: R. E. ZIELINSKI, PRINCIPAL INVESTIGATOR

**MOUND FACILITY**
Miamisburg, Ohio 45342

operated by
**MONSANTO RESEARCH CORPORATION**
a subsidiary of Monsanto Company

for the
**U. S. DEPARTMENT OF ENERGY**

Contract No. EY-76-C-04-0053

# TABLE OF CONTENTS

STATUS SUMMARY ..... i

LIST OF CONTRIBUTORS ..... iv

A. SAMPLE DESCRIPTION ..... 1

B. STATISTICAL ANALYSIS ..... 1

C. MASS SPECTROMETRY ..... 26

D. CLAY MINERALOGY ..... 38

E. STABLE CARBON ISOTOPE GEOCHEMISTRY ..... 52

F. DETAILED HYDROCARBON ANALYSIS ..... 52

G. MATERIAL BALANCE ASSAY ..... 107

H. PYROLYSIS-GAS CHROMATOGRAPHY ..... 137

I. PULSED NMR ..... 137

# TABLE OF CONTENTS

J. DILATOMETRY ..... 148

K. INTERACTIVE DATA MANAGEMENT AND ANALYSIS SYSTEM ..... 154

L. HYDROCARBON GAS PERMEATION IN DEVONIAN SHALES ..... 157

M. METHANE ABSORPTION IN DEVONIAN SHALES ..... 162

N. MECHANICAL TESTING ..... 170

O. SEM ANALYSIS ..... 197

P. BIOSTRATIGRAPHY ..... 206

Q. ARCHAEOLOGICAL SURVEY ..... 206

## STATUS SUMMARY

In addition to the progress reported during this period, this report also includes the detailed supporting data for the reports which represented the quarterly progress for the July 1 - September 30, 1977 period. These reports were presented at the First Eastern Gas Shales Symposium, Morgantown, West Virginia, October 17-19, 1977.

Statistical analyses were expanded during this period to include an analysis of Clinton sands gas flow data. After the data were appropriately partitioned, regression model techniques successfully yielded useful insight into the variables that best improve gas flow.

Mass spectrometry studies were completed on all available samples. The hydrocarbon results obtained during this study generally supported the gas chromatography results and served as verification data. The results also indicated that helium was absent from the samples. The $O_2/N_2$ results for the samples were somewhat perplexing and statistical analyses are currently being performed on the data from these samples.

Clay mineral analyses were performed on samples from four wells. These analyses indicated that illite, mixed layer illite-montmorillonite, and chlorite are present in all the samples, while kaolinite is present in minor amounts only in the Martin County, Kentucky samples.

Core samples from five wells were also analyzed by mass spectrometry for stable carbon isotope values. The results obtained to date indicate an inverse relationship when compared to previous carbon isotope studies.

Detailed hydrocarbon analyses were also completed on all wells through I-3. These results confirmed the variability present in both the Illinois and Appalachian basins. They also indicated that the

most thermally mature well is the Wise County, Virginia well. Data obtained in these studies, were consistent indicators for the results that were later obtained in the Material Balance Assays.

The Material Balance Assays indicated numerically averaged oil yields of 1.39 gallons/ton, 4.06 gallons/ton, and 9.04 gallons/ton for the KY-2, P-1 and I-1 wells, respectively. The oil yields for the VA-1, I-2 and I-3 wells were low.

Pyrolysis-gas chromatography results in general verified the Material Balance Assay yields. These results suggested that pyrolysis is required to obtain maximum hydrocarbon yields for the samples examined to date.

Pulsed Nuclear Magnetic Resonance has been used to determine the fuel content of the shale. As the data base is expanded, it appears that pulsed NMR can be a rapid, nondestructive, determination of fuel content. Correlations have currently been made with data from the KY-2, I-1, O-1, P-1, VA-1, R-109, OH-1 and OH-2 wells.

Dilatometry studies were expanded to include samples from the VA-1 and I-2 wells. The results for samples from these wells were consistent with those observed for the KY-2 samples.

An interactive data management and analysis system was established for demonstration purposes. This system is *interactive* and has been successfully demonstrated in the interactive mode. The system is currently available for immediate implementation in a multi-user mode.

A study to determine the hydrocarbon gas permeation in the Devonian shales has been initiated. The experimental system for this study is currently being assembled and will eventually be capable of studying permeation in an in-situ environment.

A study has also been initiated to determine methane absorption and desorption in Devonian shales. This

study has been designed to determine the effects of pressure, organic carbon and oil yield on the absorption of methane by the shales. Currently, samples from the KY-2 well are being tested at pressures up to 300 psig.

Mechanical testing is also in progress and significant differences have been noted in the splitting tensile strength values of samples loaded in the maximum and minimum stress directions. Gas monitoring of the fracture experiments has supported other experiments that indicate that the samples obtained to date release most of their hydrocarbon gases to the headspace of their storage containers prior to test.

SEM analysis has been completed on as received samples from the VA-1, P-1 and I-2 wells. Pyrite concentrations in samples from these wells were higher than those observed in the KY-2 samples. The I-2 samples also had high concentrations of calcite present.

A palynological investigation of samples from the O-1, P-1, KY-2 and VA-1 wells has been completed. Using a combination of spores and acritarchs, Middle and Upper Devonian seperations were made. Further subdivisions in both categories was also possible. A report detailing this work has been completed and is currently being printed and assembled.

A preliminary archaeological survey of proposed gas well locations in Lawrence and Scioto Counties, Ohio was completed. A subsequent letter requesting determination of effect was submitted to the Ohio Preservation Office, which agreed with the results and recommendations of the survey.

A report detailing the survey is currently being printed and assembled.

## LIST OF CONTRIBUTORS

The following have contributed input for this report:

A. A. Atalla

C. R. Brown

B. D. Craft

S. M. Craven

J. O. Frye

D. Kapsch

C. Kinard

E. B. Nunn

D. R. Rohler

R. L. Ryan

D. R. Schaeffer

P. W. Seabaugh

E. Stacy

R. L. Wise

R. E. Zielinski

## A. SAMPLE DESCRIPTION

Tables 1 through 7 identify the specific samples reported on in this study and their respective depths. The samples are also cross-referenced with their respective MERC sample numbers.

## B. STATISTICAL ANALYSIS

Previously from analysis on a 27 observation data set related to the Kentucky-West Virginia Frac Study, it was shown that a good regression job could be obtained when cluster analysis was used to identify natural breaks in the data for partitioning purposes. While this study did provide some insight, it was limited by the number of data points. Nevertheless, the study did clearly indicate that analysis of an expanded data set would be productive. As a consequence, a study was initiated on an available Clinton Sands gas flow data set of 328 values. The variables examined and their physical significances are listed in Table 8.

The problem in studying the gas flow is to construct the best predictive model of gas flow in terms of the well parameters available as independent variables. For this purpose, a regression model is appropriate, and the statistical computer package SAS (statistical analysis subroutines) was utilized. Not only is this convenient for the forward, backward, and maximum $R^2$ techniques but is also the statistical package utilized to preprocess and initially manipulate the data.

In the SAS system, the regression analysis subroutine calculates three criteria which are useful in evaluating the regression model fit. These are:

(1) $R^2$-coefficient; this is the ratio of the regression sum of squares to the total sum of squares, and indicates the goodness of fit of the regression model; it is desirable that this criterion be as near unity as possible, but certainly at least 50% is required before much confidence can be placed in the model.

(2) $s^2$; this is the mean square error, and is an estimate of the variance about the regression;

TABLE 1

Shale Samples from the KY-2 Well
Located in Martin County, Kentucky

| Mound Sample No. | MERC Sample No. | Depth, Ft. |
|---|---|---|
| KY-2-1 | C-336-2444-A | 2443.25/.35 |
| KY-2-2 | C-336-2444-B | 2443.35/.60 |
| KY-2-3 | C-336-2474-A | 2474.20/.53 |
| KY-2-4 | C-336-2474-B | 2474.53/.85 |
| KY-2-5 | C-336-2504-A | 2503.35/.55 |
| KY-2-6 | C-336-2504-B | 2503.55/.80 |
| KY-2-7 | C-336-2534-A | 2535.08/.33 |
| KY-2-8 | C-336-2534-B | 2535.33/.60 |
| KY-2-9 | C-336-2555-A | 2555.50/.75 |
| KY-2-10 | C-336-2555-B | 2555.75/.90 |
| KY-2-11 | C-336-2597-A | 2597.25/.56 |
| KY-2-12 | C-336-2597-B | 2597.56/.85 |
| KY-2-13 | C-336-2627-A | 2627.22/.60 |
| KY-2-14 | C-336-2627-B | 2627.60/.85 |
| KY-2-15 | C-336-2657-A | 2657.30/.67 |
| KY-2-16 | C-336-2657-B | 2657.67/.85 |
| KY-2-17 | C-336-2687-A | 2687.37/.57 |
| KY-2-18 | C-336-2687-B | 2687.57/.88 |
| KY-2-19 | C-336-2715-A | 2715.03/.29 |
| KY-2-20 | C-336-2715-B | 2715.29/.66 |
| KY-2-21 | C-336-2745-A | 2745.16/.58 |
| KY-2-22 | C-336-2745-B | 2745.58/.69 |
| KY-2-23 | C-336-2773-A | 2773.45/.60 |
| KY-2-24 | C-336-2773-B | 2773.60/.85 |
| KY-2-25 | C-336-2803-A | 2803.40/.58 |
| KY-2-26 | C-336-2803-B | 2803.58/.69 |
| KY-2-27 | C-336-2833-A | 2833.31/.50 |
| KY-2-28 | C-336-2833-B | 2833.50/.65 |
| KY-2-29 | C-336-2861-A | 2861.45/.71 |
| KY-2-30 | C-336-2861-B | 2861.71/.88 |
| KY-2-31 | C-336-2890-A | 2890.20/.40 |
| KY-2-32 | C-336-2890-B | 2890.40/.65 |
| KY-2-33 | C-336-2921-A | 2921.32/.50 |
| KY-2-34 | C-336-2921-B | 2921.50/.70 |
| KY-2-35 | C-336-2947-A | 2947.43/.71 |
| KY-2-36 | C-336-2947-B | 2947.71/.92 |
| KY-2-37 | C-336-2977-A | 2977.40/.60 |

Table 1 (Cont'd.)

Shale Samples from the KY-2 Well
Located in Martin County, Kentucky

| Mound Sample No. | MERC Sample No. | Depth, Ft. |
|---|---|---|
| KY-2-38 | C-336-2977-B | 2977.60/.83 |
| KY-2-39 | C-336-3005-A | 3005.03/.31 |
| KY-2-40 | C-336-3005-B | 3005.31/.67 |
| KY-2-41 | C-336-3035-A | 3035.43/.65 |
| KY-2-42 | C-336-3035-B | 3035.65/.95 |
| KY-2-43 | C-336-3054-A | 3054.10/.40 |
| KY-2-44 | C-336-3054-B | 3054.55/.90 |
| KY-2-45 | C-336-3085-A | 3085.20/.40 |
| KY-2-46 | C-336-3085-B | 3085.40/.60 |
| KY-2-47 | C-336-3113-A | 3113.40/.67 |
| KY-2-48 | C-336-3113-B | 3113.67/.91 |
| KY-2-49 | C-336-3143-A | 3143.22/.39 |
| KY-2-50 | C-336-3143-B | 3143.39/.63 |
| KY-2-51 | C-336-3171-A | 3171.18/.50 |
| KY-2-52 | C-336-3171-B | 3171.50/.73 |
| KY-2-53 | C-336-3201-A | 3201.03/.25 |
| KY-2-54 | C-336-3201-B | 3201.25/.52 |
| KY-2-55 | C-336-3230-A | 3230.27/.50 |
| KY-2-56 | C-336-3230-B | 3230.50/.64 |
| KY-2-57 | C-336-3260-A | 3260.26/.46 |
| KY-2-58 | C-336-3260-B | 3260.46/.68 |
| KY-2-59 | C-336-3288-A | 3288.10/.23 |
| KY-2-60 | C-336-3288-B | 3288.23/.50 |
| KY-2-61 | C-336-3328-A | 3328.30/.56 |
| KY-2-62 | C-336-3328-B | 3328.56/.86 |
| KY-2-63 | C-336-3358-A | 3358.05/.56 |
| KY-2-64 | C-336-3358-B | 3358.56/.76 |
| KY-2-65 | C-336-3386-A | 3386.10/.35 |
| KY-2-66 | C-336-3386-B | 3386.35/.70 |

TABLE 2

Shale Samples from the I-1 Well
Located in Effingham County, Illinois

| Mound Sample No | MERC Sample No. | Depth, Ft. |
|---|---|---|
| I-1-1 | I-1-3023-A | 3023* |
| I-1-4 | I-1-3044-B | 3044.25/.60 |
| I-1-6 | I-1-3057-B | 3057.30/.65 |
| I-1-8 | I-1-3075-B | 3075.31/.50 |
| I-1-9 | I-1-3099-A | 3099.30/.51 |

*Not accurately defined.

TABLE 3

Shale Samples from the O-1 Well
Located in Christian County, Kentucky

| Mound Sample No. | MERC Sample No. | Depth, Ft. |
|---|---|---|
| O-1-1 | O-1-2184 | 2183.71/2184.08 |
| O-1-3 | O-1-2261 | 2261.02/.50 |
| O-1-4 | O-1-2292 | 2292.47/.91 |
| O-1-5 | O-1-2317 | 2317.21/.64 |

TABLE 4

Shale Samples from the P-1 Well
Located in Sullivan County, Indiana

| Mound Sample | MERC Sample No. | Depth, Ft. |
|---|---|---|
| P-1-1 | P1-2142 | 2492.33/.83 |
| P-1-2 | P1-2521 | 2521.08/.33 |
| P-1-3 | P1-2549 | 2549.25/.67 |

TABLE 5

Shale Samples from the VA-1 Well
Located in Wise County, Virginia

| Mound Sample No. | MERC Sample No. | Depth, Ft. |
|---|---|---|
| VA-1-1 | C-338-4885-A | 4885.13/.40 |
| VA-1-2 | C-338-4885-B | 4885.40/.75 |
| VA-1-3 | C-338-4915-A | 4915.10/.37 |
| VA-1-4 | C-338-4915-B | 4915.37/.68 |
| VA-1-5 | C-338-4945-A | 4945.20/.43 |
| VA-1-6 | C-338-4945-B | 4945.43/.77 |
| VA-1-7 | C-338-4969-A | 4969.20/.35 |
| VA-1-8 | C-338-4969-B | 4969.35/.50 |
| VA-1-9 | C-338-4969-C | 4969.50/.70 |
| VA-1-10 | C-338-4975-A | 4975.10/.32 |
| VA-1-11 | C-338-4975-B | 4975.32/.54 |
| VA-1-12 | C-338-5229-A | 5229.20/.35 |
| VA-1-13 | C-338-5229-B | 5229.35/.65 |
| VA-1-14 | C-338-5259-A | 5259.10/.25 |
| VA-1-15 | C-338-5259-B | 5259.25/.65 |
| VA-1-16 | C-338-5289-A | 5289.10/.46 |
| VA-1-17 | C-338-5289-B | 5289.46/.60 |
| VA-1-18 | C-338-5319-A | 5319.37/.56 |
| VA-1-19 | C-338-5319-B | 5319.56/.70 |
| VA-1-20 | C-338-5348-A | 5348.30/.62 |
| VA-1-21 | C-338-5348-B | 5348.62/.80 |
| VA-1-22 | C-338-5379-A | 5379.08/.33 |
| VA-1-23 | C-338-5379-B | 5379.33/.57 |
| VA-1-24 | C-338-5393-A | 5393.20/.50 |
| VA-1-25 | C-338-5393-B | 5393.50/.65 |
| VA-1-26 | C-338-5393-C | 5393.65/.90 |
| VA-1-27 | C-338-5409-A | 5409.07/.37 |
| VA-1-28 | C-338-5409-B | 5409.37/.55 |
| VA-1-29 | C-338-5439-A | 5439.15/.40 |
| VA-1-30 | C-338-5439-B | 5439.40/.67 |
| VA-1-31 | C-338-5469-A | 5469.20/.53 |
| VA-1-32 | C-338-5469-B | 5469.53/.80 |

TABLE 6

Shale Samples from the I-2 Well
Located in Henderson County, Illinois

| Mound Sample No. | MERC Sample No. | Depth, Ft. |
|---|---|---|
| I-2-A* | N1-RAR#1 (120' Above Core Point) | 190/200 |
| I-2-1 | N1-RAR#1-1-A | 334.25/.50 |
| I-2-2 | N1-RAR#1-1-B | 334.50/.75 |
| I-2-3 | N1-RAR#1-2-A | 364.10/.32 |
| I-2-4 | N1-RAR#1-2-B | 364.32/.55 |
| I-2-5 | N1-RAR#1-3-A | 394.15/.36 |
| I-2-6 | N1-RAR#1-3-B | 394.36/.60 |
| I-2-7 | N1-RAR#1-4-A | 425.33/.52 |
| I-2-8 | N1-RAR#1-4-B | 425.52/.90 |
| I-2-9 | N1-RAR#1-5-A | 454.14/.40 |
| I-2-10 | N1-RAR#1-5-B | 454.40/.65 |
| I-2-11 | N1-RAR#1-6-A | 481.15/.32 |
| I-2-12 | N1-RAR#1-6-B | 481.32/.53 |
| I-2-13 | N1-RAR#1-7-A | 508.26/.48 |
| I-2-14 | N1-RAR#1-7-B | 508.48/.70 |
| I-2-15 | N1-RAR#1-9-A | 538.29/.45 |
| I-2-16 | N1-RAR#1-9-B | 538.45/.67 |
| I-2-17 | N1-RAR#1-10-A | 568.40/.60 |
| I-2-18 | N1-RAR#1-10-B | 568.60/.80 |
| I-2-19 | N1-RAR#1-11-A | 598.15/.30 |
| I-2-20 | N1-RAR#1-11-B | 598.30/.55 |
| I-2-B* | N1-RAR#1 (120' Below Core Point) | 730/740 |

*Cuttings.

TABLE 7

Shale Samples from the I-3 Well
Located in Tazewell County, Illinois

| Mound Sample No. | MERC Sample No. | Depth, Ft. |
|---|---|---|
| I-3-A* | N2-MAK#1 (120' Above Core Point) | 770/780 |
| I-3-1 | N2-MAK#1-1-A | 940.10/.33 |
| I-3-2 | N2-MAK#1-1-B | 940.10/.33 |
| I-3-3 | N2-MAK#1-1-C | 940.33/.75 |
| I-3-4 | N2-MAK#1-2-B | 969.04/.27 |
| I-3-5 | N2-MAK#1-2-C | 969.27/.71 |
| I-3-6 | N2-MAK#1-3-A | 995.08/.27 |
| I-3-7 | N2-MAK#1-3-B | 995.15/.31 |
| I-3-8 | N2-MAK#1-3-C | 995.31/.85 |
| I-3-9 | N2-MAK#1-4-B | 1018.22/.43 |
| I-3-10 | N2-MAK#1-4-C | 1018.43/.90 |
| I-3-11 | N2-MAK#1-5-A | 1048.19/.39 |
| I-3-12 | N2-MAK#1-5-B | 1048.19/.39 |
| I-3-13 | N2-MAK#1-5-C | 1048.39/.79 |
| I-3-14 | N2-MAK#1-6-B | 1078.25/.45 |
| I-3-15 | N2-MAK#1-6-C | 1078.45/.83 |
| I-3-16 | N2-MAK#1-7-A | 1108.12/.35 |
| I-3-17 | N2-MAK#1-7-B | 1108.12/.35 |
| I-3-18 | N2-MAK#1-7-C | 1108.35/.77 |
| I-3-19 | N2-MAK#1-8-B | 1138.01/.17 |
| I-3-20 | N2-MAK#1-8-C | 1138.17/.60 |
| I-3-B* | N2-MAK#1 (120' Below Core Point) | 1270/1280 |

*Cuttings.

TABLE 8

List of Variables and Their Physical Significance of the Clinton Sands Gas Flow Study

| Variable | Physical Significance |
|---|---|
| $Y_1$ | Open Flow, MDFD |
| $Y_2$ | Reserves of Well from Onset of Production (Includes Past Production) |
| $Y_3$ | Accumulated Production of Well for 42 Months |
| $Y_4$ | Accumulated Production of Well for 60 Months |
| $X_1$ | Well Number |
| $X_2$ | County (Eastern Ohio) |
| $X_3$ | Gross Sand, ft. |
| $X_4$* | Net Sand, ft. |
| $X_5$* | Porosity, % |
| $X_6$ | Depth to the Perforation; "Depth of the Well" |
| $X_7$ | Rock Pressure, psi |
| $X_8$ | Horse Power |
| $X_9$* | Injection Rate, bbl/min |
| $X_{10}$ | Breakdown Pressure, psi |
| $X_{11}$* | Average Treating Pressure During Frac Operation |
| $X_{12}$ | Instant Shut-In Pressure |
| $X_{13}$* | Perforation Size |
| $X_{14}$ | Number of Perforations |
| $X_{15}$ | Pad Volume, gal |
| $X_{16}$* | Treatment Volume, gal |
| $X_{17}$* | Total Water Volume, gal |
| $X_{18}$ | Total Sand, lbs |
| $X_{19}$* | Frac Width; $(0.43^* X_6 + X_{11}) - X_{20}$ |
| $X_{20}$ | $6_2$, Minimum Hor. Stress; $X_{12} + 0.43 X_6$ |
| $X_{21}$ | Vertical Stress; $1.15^* X_6$ |
| $X_{22}$* | $6_1$, Maximum Hor. Stress; $3^* X_{20} - (0.43^* X_6 + X_{10}) - 0.01^* X_5^* X_7 + 1460$ |
| $X_{23}$* | $X_{22}/X_{20}$ |
| $X_{24}$ | $X_4/X_3$ |
| $X_{25}$ | $X_{18}/X_{16}$ |
| $X_{26}$* | $X_{20}/X_6$ |
| $X_{27}$* | $X_{17}/(((0.43^* X_6 + X_{11}) - X_{20})^* X_3)$ |
| $X_{28}$ | $0.785^* X_{14}^* X_{13}^2$ |
| $X_{29}$* | $X_4^* X_5$ |
| $X_{30}$ | $X_{11} + 0.43^* X_6$ |
| $X_{31}$ | $X_{30}/X_6$ |

*Judged to be important variables, especially variables that are controllable.

the best model will generally yield a minimum $s^2$ value.

(3) F-ratio; this is an F-statistic calculated from the data and depends upon the number of variables, the number of observations, and the accepted risk level; although this F-statistic varies with these parameters, a F-ratio of about 3.0 or more is generally desirable.

For purposes of reporting results, the $R^2$ coefficient and F-ratio are most useful in indicating the tradeoff between a measurement of fit and the credibility of that fit, respectively. The $s^2$ criterion is primarily used by the author in indicating the fewest variables which best explain the variations in the dependent variable.

To test for a possible blanket fit to the complete data set, a regression analysis with the dependent variable $Y_1$ and the dependent variables $X_3$-$X_{31}$ was made. The marginal quality of the fit can be seen in the following cases:

(1) For the entire 328 data set, a fit of only about 17% ($R^2$) could be obtained (10 variables).

(2) For all points with $Y_1 \geq 3000$ MCF/day, a fit of only about 26% could be obtained for this high flow data (3 variables).

(3) For all points with $Y_1 \leq 2000$ MCF/day, a fit of only about 23% could be obtained for this low flow data (8 variables).

At this phase of the analysis it was decided that fits with the other dependent variables $Y_2$, $Y_3$, and $Y_4$ should be explored because they represent a sum of measurements taken over a longer period and therefore should be more stable.

A comparison of the analyses for the dependent variables $Y_1$, $Y_2$, $Y_3$, and $Y_4$ revealed both expected and unexpected indications. The data given in Table 9 illustrate that $Y_2$ is inferior to the other dependent variables as expected and raise some questions about the exprapolation method used for $Y_2$. However, a surprising quirk is that $Y_3$ is a slightly better variable than $Y_4$, uniformly over all the data, even though $Y_4$ is data accumulated over a longer

TABLE 9

Results from Analysis of 328 Data Points

| Dependent Variable | Number of Data Points | Independent Variables | $R^2$ | MSE | F |
|---|---|---|---|---|---|
| $Y_1$ | 325 | $X_4$*, $X_7$, $X_9$*, $X_{14}$, $X_{15}$, $X_{18}$, $X_{22}$* | 16.1% | $1.192 \times 10^6$ | 8.74 |
| | | $X_5$*, $X_7$, $X_8$, $X_{11}$*, $X_{13}$*, $X_{15}$, $X_{18}$, $X_{22}$,*, $X_{28}$, $X_{29}$* | 17.6% | $1.181 \times 10^6$ | 6.75 |
| $Y_2$ | 312 | $X_7$, $X_{19}$, $X_{28}$, | 6.0% | $44.07 \times 10^9$ | 6.55 |
| | | $X_3$, $X_7$, $X_{19}$, $X_{28}$ | 7.1% | $43.67 \times 10^9$ | 5.90 |
| | | $X_3$, $X_7$, $X_{13}$*, $X_{14}$, $X_{25}$, $X_{27}$*, $X_{28}$ | 8.4% | $43.50 \times 10^9$ | 3.99 |
| $Y_3$ | 300 | $X_7$, $X_{13}$*, $X_{14}$, $X_{16}$*, $X_{28}$ | 12.2% | $8.96 \times 10^9$ | 8.15 |
| | | $X_6$, $X_7$, $X_9$*, $X_{13}$*, $X_{14}$, $X_{18}$, $X_{25}$, $X_{27}$ $X_{28}$, $X_{30}$, $X_{31}$ | 16.3% | $8.71 \times 10^9$ | 5.10 |
| $Y_4$ | 290 | $X_7$, $X_{13}$*, $X_{14}$, $X_{16}$*, $X_{27}$*, $X_{28}$ | 12.9% | $11.82 \times 10^9$ | 6.97 |

period (60 months rather then 42 months). In a similar sense $Y_1$ is nearly everywhere a considerably better variable than $Y_3$, even though $Y_3$ is summed over 42 months, whereas $Y_1$ was supposedly subject to more errors. The differences in the quality of the fits are not significant but they are consistent.

As just shown the quality of an overall fit to the $Y_1$ data is marginal, but good results were obtained for a fit of $Y_1$ data for a tight cluster (CL1) of wells in the region.

CL1 41.0<LONG<41.2, 81.15<LAT<81.35

These results are:

| Variables | $R^2$ | MSE | F |
|---|---|---|---|
| $X_4^*, X_7, X_{17}^*, X_{23}^*, X_{27}^*, X_{31}$ | 39.9% | $1.887 \times 10^6$ | 7.96 |
| $X_5, X_7, X_{15}, X_{16}^*, X_{18}, X_{22}^*, X_{23}^*, X_{24}, X_{25}, X_{27}^*, X_{31}$ | 47.2% | $1.779 \times 10^6$ | 5.45 |

Where the asterisks indicate those variables most desirable from the point of view of control of the process. These findings indicated an expanded analysis of cluster CL1 using variable $Y_4$, as well as inclusion of clusters:

CL2 40.1<LONG<40.5 , 81.30<LAT<81.55
(a well-defined cluster, but not as tight as (CL1)

CL3 LONG<40.2 , LAT>81.45
(a sparse scattering of data in this region)

A longitude-latitude plot of the data shows that clusters CL1, CL2, and CL3 lie along a straight line.

The studies of clusters CL1, CL2, and CL3 using variable $Y_4$ show that if all the data in each cluster are to be fit, the tighter clusters generally yield better results. That is, CL1 is a better fit than CL2 or CL3. Interpretation for CL2 and CL3 is less clear, as shown in Tables 10 and 11, but only the fit for CL1 is really acceptable. A better fit is obtained for CL3 than CL2, but the confidence level in this fit is considerably lower than the fit for CL2. It should be pointed out, however, that more careful

TABLE 10

Analysis of Data in Cluster CL1

| Dependent Variable | Number of Data Points | Independent Variables | $R^2$ | MSE | F |
|---|---|---|---|---|---|
| $Y_1$ | 79 | $X_4$*, $X_7$, $X_{17}$*, $X_{23}$*, $X_{27}$, $X_{31}$ | 39.9% | $1.887 \times 10^6$ | 7.96 |
| | | $X_5$*, $X_7$, $X_{15}$, $X_{16}$*, $X_{18}$, $X_{22}$*, $X_{23}$*, $X_{24}$, $X_{25}$, $X_{27}$*, $X_{31}$ | 47.2% | $1.779 \times 10^6$ | 5.45 |
| $Y_2$ | 78 | $X_4$*, $X_6$, $X_{15}$, $X_{23}$*, $X_{28}$ | 27.7% | $44.85 \times 10^9$ | 5.52 |
| $Y_3$ | 79 | $X_4$*, $X_{15}$, $X_{23}$*, $X_{28}$ | 29.1% | $8.61 \times 10^9$ | 7.59 |
| | | $X_8$, $X_9$*, $X_{10}$, $X_{15}$, $X_{16}$*, $X_{18}$, $X_{24}$, $X_{27}$*, $X_{28}$, $X_{31}$ | 43.4% | $7.48 \times 10^9$ | 5.22 |
| $Y_4$ | 79 | $X_4$*, $X_{15}$, $X_{23}$*, $X_{28}$ | 28.8% | $12.32 \times 10^9$ | 7.47 |
| | | $X_8$, $X_9$*, $X_{10}$, $X_{15}$, $X_{24}$, $X_{25}$, $X_{27}$*, $X_{28}$, $X_{31}$ | 41.1% | $10.91 \times 10^9$ | 5.36 |

TABLE 11

Analysis of $Y_4$ Data in Clusters CL1, CL2, and CL3

| Cluster | Number of Data Points | Independent Variables | $R^2$ | MSE | F |
|---|---|---|---|---|---|
| CL1 | 79 | $X_4$*, $X_{15}$, $X_{23}$*, $X_{28}$ | 28.8% | $12.32 \times 10^9$ | 7.47 |
| | | $X_8$, $X_9$*, $X_{10}$, $X_{15}$, $X_{24}$, $X_{25}$, $X_{27}$*, $X_{28}$, $X_{31}$ | 41.1% | $10.91 \times 10^9$ | 5.36 |
| CL2 | 129 | $X_7$, $X_{13}$*, $X_{18}$, $X_{20}$ | 22.3% | $8.55 \times 10^9$ | 8.92 |
| | | $X_7$, $X_9$*, $X_{12}$, $X_{13}$*, $X_{18}$, $X_{25}$, $X_{26}$*, $X_{30}$, $X_{31}$ | 26.9% | $8.39 \times 10^9$ | 4.87 |
| | | $X_5$*, $X_7$, $X_9$*, $X_{12}$ $X_{13}$*, $X_{18}$, $X_{25}$, $X_{26}$*, $X_{30}$, $X_{31}$ | 27.6% | $8.38 \times 10^9$ | 4.50 |
| | | $X_5$*, $X_6$, $X_7$, $X_9$*, $X_{10}$, $X_{12}$, $X_{13}$, $X_{18}$, $X_{22}$*, $X_{25}$, $X_{26}$*, $X_{30}$, $X_{31}$ | 28.9% | $8.45 \times 10^9$ | 3.59 |
| CL3 | 60 | $X_5$*, $X_{15}$, $X_{18}$, $X_{31}$ | 18.3% | $13.78 \times 10^9$ | 3.08 |

scrutiny might be given these clusters because this is just a first step toward refinement. For example, CL2 might be considered as three subclusters.

Even though analyses based on high/low flow data from the overall data set did not provide acceptable results, analyses based on high/low flow data was made for cluster data CL1. A surprisingly good fit is obtained for both the high and low flow data, so similar analyses were performed for clusters CL2 and CL3.

The high flow analyses for CL1, CL2, and CL3 are given in Table 12. The best fit is definitely obtained for data within cluster CL1, with about 60% fit and good confidence in that fit. The fits CL2 and CL3 are not as good, but are quite encouraging, and again warrant further study. The low flow analyses were even more surprising because fits were totally lacking for the Kentucky-West Virginia low flow data. As indicated in Table 13 some of the fits are incredibly good, which should prompt further investigation.

For cluster CL1 the low flow data are fit to over 65% with very good confidence, and to over 50% with only five variables. The low flow data of cluster CL2 have the poorest fit characteristics, with less than 50% fit as well as low confidence. But there is still more than enough evidence to encourage further investigative fits of CL2. The most unusual fit is for the low flow data for CL3, which can be fit to essentially 100%. There is no question that part of this is due to the few observations in this data set (N=23), but a good fit is achieved with only two, three, four, or five variables. So the fit is due to more than just a small data set. As shown in Table 13, the effect of the small data set becomes apparent when fitting with about eight variables, when the confidence F ratio starts rising abruptly and the mean square error criterion drops precipitously. To assess to what extent this is a good fit more work needs to be done, especially if more data can be obtained for this region.

For these analyses it should be stated that the high and low flow threshold of 100,000 MCF/60 MOS for $Y_4$ was chosen more or less arbitrarily. A better choice might provide still better results. Likewise variables $Y_1$ or $Y_3$ might be preferable to $Y_4$ for this fit.

To more directly compare this analysis with the Kentucky-West Virginia Frac Study, cluster CL1, CL2, and CL3 data

TABLE 12

Analysis of $Y_4$ Data in Clusters CL1, CL2, and CL3 for $Y_4$ Flows $\geq$ 100,000 MCF/60 Mos.

| Cluster | Number of Data Points | Independent Variables | $R^2$ | MSE | F |
|---|---|---|---|---|---|
| HICL1 | 35 | $X_8$, $X_{15}$, $X_{27}$* | 24.7% | $15.39 \times 10^9$ | 3.40 |
| | | $X_8$, $X_{15}$, $X_{24}$, $X_{27}$ | 30.5% | $14.68 \times 10^9$ | 3.30 |
| HICL2 | 74 | $X_{13}$*, $X_{16}$*, $X_{25}$ | 26.9% | $8.32 \times 10^9$ | 8.58 |
| | | $X_7$, $X_{13}$*, $X_{16}$*, $X_{25}$ | 29.5% | $8.14 \times 10^9$ | 7.21 |
| | | $X_7$, $X_{13}$*, $X_{14}$, $X_{16}$*, $X_{25}$ | 30.8% | $8.10 \times 10^9$ | 6.06 |
| | | $X_7$, $X_9$*, $X_{13}$*, $X_{14}$, $X_{16}$*, $X_{18}$, $X_{24}$, $X_{25}$ | 32.9% | $8.22 \times 10^9$ | 3.99 |
| | | $X_7$, $X_8$, $X_{13}$*, $X_{14}$, $X_{15}$, $X_{16}$*, $X_{17}$*, $X_{24}$, $X_{25}$ | 33.8% | $8.24 \times 10^9$ | 3.62 |
| HICL3 | 37 | $X_4$*, $X_{14}$, $X_{15}$, $X_{31}$ | 22.7% | $14.50 \times 10^9$ | 2.35 |

TABLE 13

Analysis of $Y_4$ Data in Clusters CL1, CL2, and CL3 for $Y_4$ Flows $\leq$ 100,000 MCF/60 Mos.

| Cluster | Number of Data Points | Independent Variables | $R^2$ | MSE | F |
|---|---|---|---|---|---|
| LOCL1 | 44 | $X_6$, $X_7$, $X_8$, $X_{14}$, $X_{27}$* | 59.3% | $2.93 \times 10^8$ | 11.08 |
| | | $X_3$, $X_7$, $X_8$, $X_{11}$*, $X_{13}$*, $X_{27}$*, $X_{28}$, $X_{31}$ | 64.4% | $2.78 \times 10^8$ | 7.93 |
| | | $X_3$, $X_7$, $X_8$, $X_9$*, $X_{11}$*, $X_{14}$, $X_{17}$*, $X_{27}$*, $X_{28}$, $X_{31}$ | 66.9% | $2.75 \times 10^8$ | 6.66 |
| LOCL2 | 55 | $X_5$*, $X_{20}$, $X_{28}$ | 21.4% | $4.57 \times 10^8$ | 4.62 |
| | | $X_5$*, $X_7$, $X_{22}$*, $X_{25}$, $X_{28}$ | 24.9% | $4.54 \times 10^8$ | 3.25 |
| | | $X_5$, $X_7$, $X_{13}$*, $X_{14}$, $X_{16}$*, $X_{17}$*, $X_{22}$*, $X_{28}$ | 33.2% | $4.30 \times 10^8$ | 2.86 |
| | | $X_5$*, $X_6$, $X_7$, $X_{13}$*, $X_{14}$, $X_{16}$*, $X_{17}$*, $X_{22}$*, $X_{28}$, $X_{29}$* | 36.9% | $4.25 \times 10^8$ | 2.57 |
| | | $X_3$, $X_4$*, $X_5$*, $X_7$, $X_{10}$, $X_{13}$*, $X_{14}$, $X_{16}$*, $X_{17}$*, $X_{20}$, $X_{24}$, $X_{28}$ | 44.8% | $3.89 \times 10^8$ | 2.84 |

TABLE 13 (Cont'd.)

Analysis of $Y_4$ Data in Clusters CL1, CL2, and CL3 for $Y_4$ Flows $\leq$ 100,000 MCF/60 Mos.

| Cluster | Number of Data Points | Independent Variables | $R^2$ | MSE | F |
|---|---|---|---|---|---|
| LOCL3 | 23 | $X_3$, $X_{29}$* | 31.7% | $2.59 \times 10^8$ | 4.64 |
| | | $X_8$, $X_{23}$*, $X_{29}$* | 38.5% | $2.45 \times 10^8$ | 3.96 |
| | | $X_8$, $X_{23}$*, $X_{27}$*, $X_{29}$* | 45.5% | $2.30 \times 10^8$ | 3.75 |
| | | $X_8$, $X_{16}$*, $X_{23}$*, $X_{27}$*, $X_{29}$* | 48.8% | $2.28 \times 10^8$ | 3.24 |
| | | $X_3$, $X_4$*, $X_9$, $X_{10}$, $X_{12}$, $X_{23}$*, $X_{24}$, $X_{28}$ | 71.8% | $1.53 \times 10^8$ | 4.45 |
| | | $X_3$, $X_4$*, $X_7$, $X_9$*, $X_{10}$, $X_{16}$*, $X_{23}$*, $X_{24}$, $X_{28}$, $X_{29}$* | 82.8% | $1.09 \times 10^8$ | 5.76 |

points were rerun with $Y_1$ as the dependent variable rather than $Y_4$. A comparison of data given in Tables 11 and 14 shows that one cannot simply conclude that the $Y_1$ data is preferable to the $Y_4$ data because uniformly better fits are not obtained using one dependent variable over the other. We can observe that the fits for clusters CL1 and CL3 are comparable for $Y_1$ and $Y_4$. However, a substantially improved fit at 38% with a high confidence for $Y_1$ is obtained for cluster CL2, whereas only a fit of 28% is obtained using $Y_4$ data.

High flow observations within the three clusters for the variables $Y_1$ and $Y_4$ were compared in a similar manner. The results are given in Tables 15 and 12 respectively. The fit for the high flow data within cluster CL1 are actually somewhat inferior to that obtained for $Y_4$, but they are comparable, except that a lower confidence is associated with the $Y_1$ fit. However, the fits for high flow data for clusters CL2 and CL3 using variable $Y_1$ are substantially better (with higher confidences) than achieved using variable $Y_4$, with improvement for cluster CL2 being the most dramatic.

Again it should be emphasized that the thresholds of $Y_4 \geq 100{,}000$ MCF/60 MOS and $Y_1 \geq 2000$ MCF/day were chosen somewhat arbitrarily in order to partition the data at a nominal middle flow range and in order to achieve reasonable cluster sizes. A more systematic assessment of these thresholds might yield even better results. To illustrate, compare the data in Table 16 for high flow in cluster CL1 to that given in Tables 12 and 15. In Table 16, for $Y_1$ a threshold of 2500 MCF/day was used, and a much better fit was obtained than even that achieved using $Y_4$ data.

The threshold of 2000 MCF/day was selected in order to increase the higher flow subset from the 21 observations associated with the 2500 MCF/day threshold. Nevertheless, as shown in Table 9, the excellent fit is obtained long before the effect of having only 21 observations comes into play.

The results for the low flow observations within the three clusters for the variables $Y_1$ and $Y_4$ are listed in Tables 17 and 13 respectively. Although the low flow results for cluster CL1 are comparable for variables $Y_1$ and $Y_4$, in general $Y_1$ does not show up as good a variable as $Y_4$. The excellent fit of CL3 low flow data using $Y_4$ is not achieved at all using variable $Y_1$. Possibly the effect of the threshold of $Y_1 \leq 2000$ MCF/day being too

TABLE 14

Analysis of Data in Clusters CL1, CL2, and CL3
Using Variable $Y_1$

| Cluster | Number of Data Points | Independent Variables | $R^2$ | MSE | F |
|---|---|---|---|---|---|
| CL1 | 79 | $X_4$*, $X_7$, $X_{17}$*, $X_{23}$*, $X_{27}$*, X | 39.9% | $1.88 \times 10^6$ | 7.96 |
| CL2 | 145 | $X_7$, $X_{13}$*, $X_{18}$, $X_{23}$* | 28.4% | $4.94 \times 10^5$ | 13.90 |
| | | $X_7$, $X_8$, $X_{13}$*, $X_{16}$*, $X_{18}$, $X_{22}$*, $X_{25}$, $X_{28}$, $X_{29}$* | 37.1% | $4.50 \times 10^5$ | 8.84 |
| | | $X_3$, $X_7$, $X_9$*, $X_{13}$*, $X_{16}$*, $X_{18}$, $X_{19}$*, $X_{22}$*, $X_{25}$, $X_{28}$, $X_{29}$* | 38.4% | $4.47 \times 10^5$ | 7.52 |
| | | $X_3$, $X_7$, $X_9$*, $X_{13}$*, $X_{15}$, $X_{16}$*, $X_{17}$*, $X_{18}$, $X_{19}$*, $X_{22}$*, $X_{25}$, $X_{28}$, $X_{29}$* | 38.8% | $4.51 \times 10^5$ | 6.38 |
| CL3 | 75 | $X_6$, $X_7$, $X_{15}$, $X_{23}$* | 15.9% | $8.04 \times 10^5$ | 3.31 |
| | | $X_8$*, $X_6$, $X_7$, $X_9$*, $X_{14}$, $X_{15}$, $X_{23}$* | 18.4% | $8.15 \times 10^5$ | 2.16 |
| | | $X_7$, $X_8$, $X_9$*, $X_{12}$, $X_{13}$*, $X_{14}$, $X_{15}$, $X_{22}$, $X_{23}$*, $X_{26}$*, $X_{28}$, $X_{31}$ | 35.9% | $6.92 \times 10^5$ | 2.89 |

TABLE 15

Analysis of $Y_1$ Data in Clusters CL1, CL2, and CL3 for $Y_1$ Flows $\geq$ 2,000 MCF/DAY

| Cluster | Number of Data Points | Independent Variables | $R^2$ | MSE | F |
|---|---|---|---|---|---|
| HICL1 | 26 | $X_{25}$, $X_{27}$*, $X_{30}$ | 22.4% | $2.05 \times 10^6$ | 2.12 |
| | | $X_{14}$, $X_{15}$, $X_{24}$, $X_{25}$, $X_{27}$* | 36.2% | $1.85 \times 10^6$ | 2.27 |
| | | $X_3$, $X_4$*, $X_{14}$, $X_{19}$*, $X_{25}$, $X_{27}$*, $X_{29}$* | 50.4% | $1.60 \times 10^6$ | 2.61 |
| HICL2 | 37 | $X_{12}$, $X_{14}$, $X_{29}$* | 39.5% | $4.06 \times 10^5$ | 7.19 |
| | | $X_{12}$, $X_{14}$, $X_{25}$, $X_{29}$* | 41.6% | $4.05 \times 10^5$ | 5.70 |
| | | $X_{12}$, $X_{14}$, $X_{15}$, $X_{24}$, $X_{25}$ | 45.8% | $3.87 \times 10^5$ | 5.25 |
| | | $X_3$, $X_4$*, $X_{15}$, $X_{18}$, $X_{24}$, $X_{27}$*, $X_{28}$, $X_{29}$* | 54.2% | $3.63 \times 10^5$ | 4.14 |
| | | $X_3$, $X_4$ *, $X_5$*, $X_8$, $X_9$*, $X_{13}$*, $X_{15}$, $X_{18}$, $X_{24}$, $X_{27}$*, $X_{28}$, $X_{29}$*, $X_{30}$ | 74.1% | $2.50 \times 10^5$ | 5.06 |
| HICL3 | 17 | $X_7$, $X_{13}$*, $X_{16}$*, $X_{19}$* | 53.4% | $2.68 \times 10^5$ | 3.44 |
| | | $X_7$, $X_{11}$*, $X_{13}$*, $X_{16}$*, $X_{23}$* | 62.7% | $2.34 \times 10^5$ | 3.70 |
| | | $X_7$, $X_8$ $X_9$*, $X_{13}$*, $X_{16}$*, $X_{23}$* | 71.0% | $2.00 \times 10^5$ | 4.09 |

TABLE 16

Analysis of $Y_1$ Data for CL1 with $Y_1 \geq 2,500$ MCF/Day

| Cluster | Number of Data Points | Independent Variables | $R^2$ | MSE | F |
|---|---|---|---|---|---|
| HICL1 | 21 | $X_8$, $X_{15}$, $X_{27}$* | 22.1% | $1.89 \times 10^6$ | 1.61 |
| | | $X_8$, $X_{15}$, $X_{24}$, $X_{27}$* | 26.2% | $1.90 \times 10^6$ | 1.42 |
| | | $X_{17}$, $X_{19}$, $X_{24}$, $X_{27}$, $X_{29}$ | 49.1% | $1.40 \times 10^6$ | 2.89 |
| | | $X_3$, $X_{17}$*, $X_{18}$, $X_{19}$*, $X_{24}$, $X_{27}$* | 57.6% | $1.25 \times 10^6$ | 3.16 |

TABLE 17

Analysis of $Y_1$ Data in Clusters CL1, CL2, and CL3 for $Y_1$ Flow $\leq$ 2,000 MCF/Day

| Cluster | Number of Data Points | Independent Variables | $R^2$ | MSE | F |
|---|---|---|---|---|---|
| LOCL1 | 53 | $X_3$, $X_7$, $X_9$*, $X_{14}$, $X_{18}$ | 44.9% | $1.53 \times 10^5$ | 6.26 |
| | | $X_3$, $X_7$, $X_9$*, $X_{14}$, $X_{16}$*, $X_{18}$, $X_{22}$*, $X_{25}$ | 49.2% | $1.48 \times 10^5$ | 5.34 |
| | | $X_3$, $X_5$*, $X_7$, $X_8$, $X_9$*, $X_{11}$*, $X_{14}$, $X_{18}$, $X_{22}$*, $X_{25}$ | 54.7% | $1.38 \times 10^5$ | 5.08 |
| LOCL2 | 108 | $X_7$, $X_{23}$*, $X_{25}$ | 13.7% | $1.10 \times 10^5$ | 5.49 |
| | | $X_3$, $X_7$, $X_{17}$*, $X_{23}$*, $X_{25}$ | 17.8% | $1.07 \times 10^5$ | 4.41 |
| | | $X_3$, $X_7$, $X_{14}$, $X_{16}$*, $X_{17}$*, $X_{18}$, $X_{23}$*, $X_{25}$ | 22.5% | $1.04 \times 10^5$ | 3.60 |
| | | $X_7$, $X_{13}$*, $X_{14}$, $X_{15}$, $X_{16}$*, $X_{18}$, $X_{21}$, $X_{23}$*, $X_{24}$, $X_{25}$, $X_{28}$, $X_{29}$* | 25.5% | $1.04 \times 10^5$ | 2.71 |

TABLE 17 (Cont'd.)

Analysis of $Y_1$ Data in Clusters CL1, CL2, and CL3 for $Y_1$ Flow $\leq$ 2,000 MCF/Day

| Cluster | Number of Data Points | Independent Variables | $R^2$ | MSE | F |
|---|---|---|---|---|---|
| LOCL3 | 58 | $X_6$, $X_{14}$ | 12.4% | $2.59 \times 10^5$ | 3.89 |
| | | $X_5$*, $X_6$, $X_{14}$ | 15.2% | $2.55 \times 10^5$ | 3.22 |
| | | $X_5$*, $X_6$, $X_{14}$, $X_{17}$* $X_{23}$* | 17.7% | $2.57 \times 10^5$ | 2.24 |
| | | $X_5$, $X_6$, $X_8$, $X_9$*, $X_{13}$*, $X_{14}$, $X_{15}$, $X_{20}$ $X_{27}$*, $X_{28}$, $X_{31}$ | 28.7% | $2.52 \times 10^5$ | 1.68 |

large comes into play here, as indicated by the considerably larger size for CL2 and CL3 and $Y_1$ data for the corresponding $Y_4$ data.

Further examination of these clusters and threshold values for $Y_1$, $Y_3$, or $Y_4$ should provide even better results. It might be necessary to use different thresholds for high and low flows in order to obtain good flows across different clusters. For these data and at this stage of the analysis it is felt that no new insight will be gained by using a non-linear model. Nevertheless these findings support characterization and development of regional parameters as constrasted to global parameters. For example, the net sand and perforation are indicated as key parameters.

The overall objective of the study was to identify those controllable variables to best improve gas flow, and to develop strategies to obtain such flow improvement. For example, for the 19 high flow data points with $Y_1 \geq 3000$ MCF/day in cluster CL1, the following fit was obtained:

| VARIABLES IN MODEL | $R^2$ | F-RATIO |
|---|---|---|
| $X_4$*, $X_{27}$*, $X_{29}$* | 48.7% | 4.74 |

Note that nearly a 50% fit is obtained with good confidence using only three variables, and <u>all</u> correspond to important well parameters.

In summary, the regression model techniques have proven sufficiently successful after the data is partitioned appropriately that this approach should yield useful insights into improved gas well operation. In addition, these techniques could be applied to any other similar data from other regions which should become available and also data from other geological gas producing sources such as Eastern Gas Shales.

## C. MASS SPECTROMETRY

Prior to the use of the shale core samples for the various physicochemical tests, the sealed container is opened in a special test fixture. The composition of the gases in the sealed container are determined by mass spectrometry, and the internal pressure of the container is monitored by means

of an automatic pressure recording device. The results for the various wells examined to date are listed in Tables 18 through 23. The hydrocarbon results are generally supportative of the gas chromatography results and serve as verification data. The results for the wells also indicate that helium is absent from the shale gases.

The nitrogen-oxygen results are somewhat perplexing. The ratio of these gases ($O_2/N_2$) for normal air is approximately 0.27. An examination of the results, however, shows that this ratio is not always present in the container upon opening. One of the first explanations for these types of results would be the oxidation of the interior walls of the container, but this effect has not been observed when the containers are examined after opening. The next most logical explanation is the oxidation of the shale core. This is a feasible explanation because as the oxygen concentration decreases, the $CO_2$ concentration increases. In a test to better preserve the core and protect it from oxidation several samples from the VA-1 (Wise County, Virginia) core were wrapped in plastic and aluminum foil in the field and these samples were stored under water to minimize their air exposure. The samples, however, become wet. Once the water contacted the shale it reacted with the pyrite present in the shale and a mild acid, formed from this reaction, reacted with the aluminum foil wrapper and hydrogen was liberated. The reaction did liberate significant quantities of hydrogen as illustrated for the specific results of samples VA-1-8, VA-1-9 and VA-1-24.

Initially it was thought htat the type of kerogen present in the shale core influenced the depletion of oxygen and in-growth of $CO_2$ in the samples. The observation was made because the samples from the KY-2 Well (Martin County, Kentucky) that were basically woody-coaly had a faster oxygen depletion rate than those that were herbaceous. This observation, however, has not been that consistent in the samples from the other wells. Because the cause of this effect has not been clearly defined, statistical analyses are currently in progress to determine the interdependence of the characteristics of the shale with the $O_2/N_2$ ratios of the head space gas present in the sample containers.

TABLE 18

Mass Spectrometric Results for the
Martin County, Kentucky, Samples

| Components (Mole %) | KY-2-1 | KY-2-3 | KY-2-7 | KY-2-10 | KY-2-12 | KY-2-14 | KY-2-16 | KY-2-17 | KY-2-19 | KY-2-22 |
|---|---|---|---|---|---|---|---|---|---|---|
| Methane | 21.18 | 15.49 | 3.51 | 0.19 | 0.09 | 0.39 | 4.60 | 10.08 | 19.46 | 5.25 |
| 2-$H_2$ | 0.21 | 0.16 | 0.02 | 0.02 | 0.05 | 0.04 | 0.11 | 0.16 | 0.28 | 0.01 |
| 4-He | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 18-$H_2O$ | 0.47 | 0.09 | 0.10 | 1.13 | 5.06 | 5.59 | 23.86 | 2.04 | 0.06 | 0.18 |
| 26-$C_2H_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 28-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 29-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 30-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 28-$N_2$ | 62.60 | 68.16 | 77.77 | 75.13 | 73.30 | 72.88 | 54.72 | 68.02 | 61.39 | 76.33 |
| 28-$C_2H_4$ | 1.76 | 0.78 | 0.19 | 0.01 | 0.04 | 0.02 | 0.69 | 0.85 | 2.20 | 0.34 |
| 30-$C_2H_6$ | 1.15 | 0.67 | 0.16 | 0.01 | 0.04 | 0.02 | 0.49 | 0.60 | 1.58 | 0.25 |
| 32-$O_2$ | 9.32 | 10.39 | 15.99 | 22.21 | 19.57 | 19.56 | 9.44 | 11.73 | 0.10 | 11.70 |
| 40-Ar | 0.74 | 0.86 | 0.95 | 1.00 | 0.91 | 0.90 | 0.70 | 0.83 | 0.75 | 0.95 |
| 44-$CO_2$ | 1.20 | 2.58 | 1.10 | 0.26 | 0.28 | 0.56 | 2.82 | 2.87 | 6.94 | 4.51 |
| 45-$CO_2$ | 0.03 | 0.04 | 0.02 | 0.01 | 0.00 | 0.01 | 0.05 | 0.05 | 0.13 | 0.07 |
| 46-$CO_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.02 | 0.04 | 0.02 |
| 47-$CO_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 44-$C_3H_8$ | 1.34 | 0.77 | 0.19 | 0.03 | 0.21 | 0.03 | 1.07 | 0.95 | 2.71 | 0.38 |
| 29-$C_2H_5$ | | | | | | | | | 4.39 | 0.00 |
| Gas Pressure (atm) | 2.578 | 1.598 | 1.696 | 1.683 | 1.433 | 1.111 | 1.039 | 1.462 | 2.259 | 2.144 |
| $O_2/N_2$ Ratio | 0.15 | 0.15 | 0.21 | 0.30 | 0.27 | 0.27 | 0.17 | 0.17 | 0.00 | 0.15 |
| $Ar/N_2$ Ratio | 0.01 | 0.01 | 0.01 | 0.01 | 0.01 | 0.01 | 0.01 | 0.01 | 0.012 | 0.012 |
| Time in Can (Days) | 182 | 210 | 253 | 230 | 203 | 251 | 251 | 251 | 257 | 257 |

UGK File #Sec. 1977 Found Faulty 10/8/81

TABLE 18 (Cont'd.)

**Mass Spectrometric Results for the Martin County, Kentucky, Samples**

| Components (Mole %) | KY-2-23 | KY-2-26 | KY-2-28* | KY-2-30 | KY-2-31 | KY-2-33 | KY-2-36 | KY-2-37 | KY-2-39 | KY-2-41 |
|---|---|---|---|---|---|---|---|---|---|---|
| Methane | 1.30 | 0.13 | 0.00 | 3.41 | 7.19 | 5.21 | 3.66 | 12.76 | 14.43 | 0.37 |
| 2-$H_2$ | 0.05 | 0.02 | 0.00 | 0.02 | 0.08 | 0.08 | 0.05 | 0.16 | 0.22 | 0.01 |
| 4-He | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 18-$H_2O$ | 0.25 | 1.09 | 4.27 | 0.74 | 0.05 | 2.70 | 0.15 | 0.25 | 0.34 | 0.35 |
| 26-$C_2H_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 28-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 29-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 30-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 28-$N_2$ | 76.47 | 76.13 | 73.34 | 74.43 | 74.69 | 70.33 | 76.38 | 70.17 | 69.40 | 76.71 |
| 28-$C_2H_4$ | 0.12 | 0.04 | 0.02 | 0.30 | 0.65 | 0.74 | 0.27 | 1.38 | 1.87 | 0.02 |
| 30-$C_2H_6$ | 0.09 | 0.02 | 0.00 | 0.22 | 0.48 | 2.57 | 0.21 | 0.75 | 1.01 | 0.03 |
| 32-$O_2$ | 18.38 | 20.97 | 21.27 | 16.46 | 9.52 | 16.25 | 16.31 | 8.77 | 7.91 | 21.42 |
| 40-Ar | 0.95 | 0.94 | 0.92 | 0.93 | 0.93 | 0.80 | 0.94 | 0.48 | 0.65 | 0.92 |
| 44-$CO_2$ | 2.11 | 0.58 | 0.13 | 3.02 | 5.43 | 1.31 | 1.64 | 4.71 | 3.44 | 0.14 |
| 45-$CO_2$ | 0.04 | 0.02 | 0.00 | 0.05 | 0.08 | 0.02 | 0.03 | 0.08 | 0.09 | 0.00 |
| 46-$CO_2$ | 0.02 | 0.00 | 0.00 | 0.02 | 0.03 | 0.00 | 0.02 | 0.03 | 0.03 | 0.00 |
| 47-$CO_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 44-$C_3H_8$ | 0.22 | 0.07 | 0.04 | 0.41 | 0.87 | 0.00 | 0.35 | 0.45 | 0.61 | 0.03 |
| 29-$C_2H_5$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | | 0.00 | 0.00 | 0.00 | 0.00 |
| Gas Pressure (atm) | 2.083 | 1.399 | 1.478 | 1.801 | 2.280 | 1.155 | 2.446 | 2.185 | 2.358 | 2.195 |
| $O_2/N_2$ Ratio | 0.24 | 0.28 | 0.29 | 0.22 | 0.13 | 0.23 | 0.21 | 0.12 | 0.11 | 0.28 |
| $Ar/N_2$ Ratio | 0.012 | 0.012 | 0.012 | 0.012 | 0.013 | 0.01 | 0.012 | 0.007 | 0.009 | 0.012 |
| Time in Can (Days) | 257 | 257 | 256 | 256 | 268 | 124 | 267 | 2.67 | 266 | 266 |

*Suspected container leak.

TABLE 18 (Cont'd.)

**Mass Spectrometric Results for the Martin County, Kentucky, Samples**

| Components (Mole %) | KY-2-43 | KY-2-46 | KY-2-48 | KY-2-49 | KY-2-52 | KY-2-53 |
|---|---|---|---|---|---|---|
| Methane | 2.83 | 0.20 | 12.19 | 1.01 | 4.79 | 12.28 |
| 2-$H_2$ | 0.09 | 0.00 | 0.21 | 0.00 | 0.08 | 0.10 |
| 4-He | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 18-$H_2O$ | 0.44 | 0.54 | 0.54 | 0.08 | 0.40 | 0.47 |
| 26-$C_2H_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 28-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 29-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 30-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 28-$N_2$ | 74.19 | 76.44 | 66.33 | 77.52 | 78.02 | 74.96 |
| 28-$C_2H_4$ | 0.60 | 0.01 | 1.57 | 0.07 | 0.37 | 1.19 |
| 30-$C_2H_6$ | 0.35 | 0.01 | 5.13 | 0.04 | 0.26 | 0.80 |
| 32-$O_2$ | 16.98 | 21.79 | 11.84 | 19.54 | 9.89 | 0.17 |
| 40-Ar | 0.89 | 0.91 | 0.72 | 0.95 | 0.98 | 0.90 |
| 44-$CO_2$ | 2.41 | 0.08 | 1.42 | 0.68 | 4.71 | 7.65 |
| 45-$CO_2$ | 0.04 | 0.00 | 0.03 | 0.01 | 0.07 | 0.11 |
| 46-$CO_2$ | 0.01 | 0.00 | 0.00 | 0.00 | 0.03 | 0.04 |
| 47-$CO_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 44-$C_3H_8$ | 1.07 | 0.02 | 0.00 | 0.10 | 0.40 | 1.31 |
| 29-$C_2H_5$ | | | | 0.00 | 0.00 | 0.00 |
| Gas Pressure (atm) | 1.193 | 1.211 | 2.212 | 2.222 | 1.824 | 2.229 |
| $O_2/N_2$ Ratio | 0.23 | 0.29 | 0.18 | 0.252 | 0.127 | 0.002 |
| $Ar/N_2$ Ratio | 0.01 | 0.012 | 0.01 | 0.012 | 0.013 | 0.012 |
| Time in Can (Days) | 202 | 266 | 119 | 283 | 283 | 283 |

TABLE 18 (Cont'd.)

**Mass Spectrometric Results for the Martin County, Kentucky, Samples**

| Components (Mole %) | KY-2-56 | KY-2-57 | KY-2-59 | KY-2-61 | KY-2-64 | KY-2-65 |
|---|---|---|---|---|---|---|
| Methane | 8.39 | 0.79 | 1.06 | 5.97 | 12.57 | 16.94 |
| 2-$H_2$ | 0.14 | 0.02 | 0.11 | 0.04 | 0.05 | 0.14 |
| 4-He | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 18-$H_2O$ | 0.07 | 0.64 | 0.06 | 0.08 | 0.15 | 0.23 |
| 26-$C_2H_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 28-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 29-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 30-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 28-$N_2$ | 72.40 | 76.28 | 80.25 | 78.44 | 70.45 | 67.32 |
| 28-$C_2H_4$ | 0.96 | 0.05 | 0.10 | 0.65 | 0.86 | 1.38 |
| 30-$C_2H_6$ | 3.30 | 0.04 | 0.06 | 0.44 | 0.64 | 0.97 |
| 32-$O_2$ | 11.07 | 19.66 | 15.99 | 8.12 | 11.05 | 5.51 |
| 40-Ar | 0.83 | 1.01 | 1.00 | 1.00 | 0.87 | 0.82 |
| 44-$CO_2$ | 2.80 | 1.43 | 1.20 | 4.31 | 2.27 | 5.00 |
| 45-$CO_2$ | 0.04 | 0.03 | 0.02 | 0.06 | 0.04 | 0.08 |
| 46-$CO_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.02 |
| 47-$CO_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 44-$C_3H_8$ | 0.00 | 0.05 | 0.15 | 0.86 | 1.02 | 1.59 |
| 29-$C_2H_5$ | | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| Gas Pressure (atm) | 2.443 | 2.092 | 2.063 | 2.390 | 2.236 | 2.209 |
| $O_2/N_2$ Ratio | 0.15 | 0.258 | 0.199 | 0.104 | 0.157 | 0.082 |
| Ar/$N_2$ Ratio | 0.01 | 0.013 | 0.013 | 0.013 | 0.123 | 0.012 |
| Time in Can (Days) | 118 | 282 | 285 | 284 | 284 | 283 |

**TABLE 19**

**Mass Spectrometric Results for the Effingham County, Illinois, Samples**

| Components (Mole %) | I-1-2 | I-1-3 | I-1-5 | I-1-7 | I-1-10 |
|---|---|---|---|---|---|
| Methane | 17.53 | 11.58 | 10.52 | 12.84 | 9.90 |
| 2-$H_2$ | 0.15 | 0.10 | 0.07 | 0.06 | 0.04 |
| 4-He | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 18-$H_2O$ | 0.13 | 0.35 | 0.45 | 0.54 | 0.13 |
| 26-$C_2H_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 28-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 29-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 30-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 28-$N_2$ | 68.86 | 69.10 | 70.96 | 68.90 | 71.90 |
| 28-$C_2H_4$ | 0.95 | 0.61 | 0.64 | 0.66 | 0.47 |
| 30-$C_2H_6$ | 0.76 | 0.51 | 0.50 | 0.54 | 0.37 |
| 32-$O_2$ | 9.40 | 14.12 | 13.30 | 13.62 | 14.70 |
| 40-Ar | 0.75 | 0.94 | 0.88 | 0.85 | 0.86 |
| 44-$CO_2$ | 0.85 | 2.05 | 2.10 | 1.39 | 1.31 |
| 45-$CO_2$ | 0.02 | 0.04 | 0.03 | 0.03 | 0.02 |
| 46-$CO_2$ | 0.00 | 0.02 | 0.03 | 0.00 | 0.00 |
| 47-$CO_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 44-$C_3H_8$ | 0.60 | 0.59 | 0.52 | 0.57 | 0.31 |
| Gas Pressure (atm) | 2.023 | 1.087 | 1.087 | 1.457 | 1.822 |
| $O_2/N_2$ Ratio | 0.137 | 0.204 | 0.187 | 0.0198 | 0.204 |
| $Ar/N_2$ Ratio | 0.011 | 0.014 | 0.012 | 0.012 | 0.012 |
| Time in Can (Days) | 177 | 176 | 175 | 175 | 174 |

TABLE 20

Mass Spectrometric Results for the Sullivan County, Indiana, Samples

| Components (Mole %) | P-1-1 | P-1-2 | P-1-3 |
|---|---|---|---|
| Methane | 49.15 | 51.30 | 13.20 |
| 2-$H_2$ | 0.02 | 0.10 | 1.02 |
| 4-He | 0.00 | 0.00 | 0.00 |
| 18-$H_2O$ | 0.06 | 0.10 | 0.20 |
| 26-$C_2H_2$ | 0.00 | 0.00 | 0.00 |
| 28-CO | 1.10 | 0.00 | 0.00 |
| 29-CO | 0.00 | 0.00 | 0.00 |
| 30-CO | 0.00 | 0.00 | 0.00 |
| 28-$N_2$ | 28.90 | 30.90 | 74.60 |
| 28-$C_2H_4$ | 1.10 | 1.40 | 0.90 |
| 30-$C_2H_6$ | 4.30 | 5.00 | 3.10 |
| 32-$O_2$ | 5.73 | 5.20 | 1.40 |
| 40-Ar | 0.37 | 0.40 | 0.90 |
| 44-$CO_2$ | 9.02 | 3.80 | 4.30 |
| 45-$CO_2$ | 0.11 | 0.10 | 0.10 |
| 46-$CO_2$ | 0.03 | 0.00 | 0.00 |
| 47-$CO_2$ | 0.00 | 0.00 | 0.00 |
| 44-$C_3H_8$ | 0.00 | 1.70 | 1.30 |
| Gas Pressure (atm) | 1.50 | 1.94 | 3.76 |
| $O_2/N_2$ Ratio | 0.198 | 0.168 | 0.019 |
| $Ar/N_2$ Ratio | 0.013 | 0.013 | 0.012 |
| Time in Can (Days) | 58 | 47 | 14 |

TABLE 21

**Mass Spectrometric Results for the Wise County, Virginia, Samples**

| Components (Mole %) | VA-1-1 | VA-1-3 | VA-1-5 | VA-1-7 | VA-1-8* | VA-1-9* | VA-1-10 | VA-1-12 |
|---|---|---|---|---|---|---|---|---|
| Methane | 24.14 | 17.22 | 10.01 | 0.21 | 0.53 | 0.80 | 3.91 | 12.49 |
| 2-$H_2$ | 0.10 | 0.10 | 0.00 | 0.00 | 47.55 | 51.99 | 0.00 | 0.02 |
| 4-He | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 18-$H_2O$ | 0.15 | 0.06 | 0.33 | 0.14 | 0.39 | 0.00 | 0.46 | 0.47 |
| 26-$C_2H_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.97 | 0.00 | 0.00 |
| 28-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 29-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 30-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 28-$N_2$ | 59.16 | 64.06 | 73.55 | 77.08 | 43.61 | 42.39 | 75.53 | 74.35 |
| 28-$C_2H_4$ | 0.69 | 0.48 | 0.00 | 0.01 | 0.00 | 0.01 | 0.03 | 0.26 |
| 30-$C_2H_6$ | 0.64 | 0.45 | 0.17 | 0.00 | 0.01 | 0.01 | 0.06 | 0.27 |
| 32-$O_2$ | 13.97 | 16.52 | 12.84 | 20.96 | 7.28 | 3.24 | 18.79 | 8.84 |
| 40-Ar | 0.73 | 0.76 | 0.94 | 0.87 | 0.54 | 0.52 | 0.93 | 0.89 |
| 44-$CO_2$ | 0.29 | 0.25 | 1.45 | 0.07 | 0.01 | 0.01 | 0.16 | 2.26 |
| 45-$CO_2$ | 0.00 | 0.00 | 0.02 | 0.00 | 0.00 | 0.00 | 0.00 | 0.03 |
| 46-$CO_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 47-$CO_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 44-$C_3H_8$ | 0.15 | 0.11 | 0.70 | 0.01 | 0.03 | 0.02 | 0.15 | 0.11 |
| Gas Pressure (atm) | 1.836 | 2.077 | 1.917 | 1.037 | 1.005 | ~1.000 | 1.911 | 2.001 |
| $O_2/N_2$ Ratio | 0.236 | 0.258 | 0.175 | 0.272 | 0.17 | 0.08 | 0.249 | 0.119 |
| $Ar/N_2$ Ratio | 0.012 | 0.012 | 0.013 | 0.011 | 0.01 | 0.01 | 0.012 | 0.012 |
| Time in Can (Days) | 139 | 118 | 138 | 24 | 23 | 24 | 138 | 134 |

*Stored in Water.

UGR File #C051/78Q.
Oct.-Dec. 1977
Mound Facility

TABLE 21 (Cont'd.)

**Mass Spectrometric Results for the Wise County, Virginia, Samples**

| Components (Mole %) | VA-1-14 | VA-1-16 | VA-1-19 | VA-1-23 | VA-1-24* | VA-1-28 | VA-1-30 | VA-1-31 |
|---|---|---|---|---|---|---|---|---|
| Methane | 32.63 | 56.70 | 15.15 | 63.33 | 5.40 | 12.57 | 42.35 | 74.68 |
| 2-$H_2$ | 0.17 | 0.10 | 0.00 | 0.16 | 75.99 | 0.00 | 0.07 | 0.28 |
| 4-He | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 18-$H_2O$ | 0.08 | 0.13 | 0.19 | 0.41 | 0.81 | 0.46 | 0.20 | 0.08 |
| 26-$C_2H_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 28-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 29-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 30-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 28-$N_2$ | 58.85 | 37.33 | 64.83 | 29.45 | 17.03 | 70.72 | 48.91 | 20.10 |
| 28-$C_2H_4$ | 1.17 | 1.14 | 0.23 | 1.76 | 0.21 | 0.21 | 1.00 | 1.59 |
| 30-$C_2H_6$ | 1.07 | 1.11 | 0.26 | 1.73 | 0.21 | 0.22 | 0.97 | 1.54 |
| 32-$O_2$ | 2.41 | 1.35 | 17.70 | 1.25 | 0.07 | 13.26 | 3.53 | 0.98 |
| 40-Ar | 0.65. | 0.38 | 0.89 | 0.27 | 0.21 | 0.84 | 0.54 | 0.16 |
| 44-$CO_2$ | 2.73 | 1.58 | 0.69 | 1.42 | 0.04 | 1.60 | 2.25 | 0.46 |
| 45-$CO_2$ | 0.04 | 0.03 | 0.02 | 0.02 | 0.00 | 0.03 | 0.03 | 0.00 |
| 46-$CO_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.00 |
| 47-$CO_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 44-$C_3H_8$ | 0.21 | 0.14 | 0.04 | 0.19 | 0.03 | 0.07 | 0.12 | 0.14 |
| Gas Pressure (atm) | 2.738 | 4.420 | 2.218 | 1.943 | 1.281 | 2.018 | 2.780 | 2.092 |
| $O_2/N_2$ Ratio | 0.041 | 0.036 | 0.273 | 0.042 | 0.004 | 0.188 | 0.072 | 0.049 |
| $Ar/N_2$ Ratio | 0.011 | 0.010 | 0.014 | 0.009 | 0.012 | 0.012 | 0.011 | 0.008 |
| Time in Can (Days) | 114 | 113 | 113 | 132 | 40 | 131 | 111 | 132 |

*Stored in Water.

TABLE 22

**Mass Spectrometric Results for the Henderson County, Illinois, Samples**

| Components (Mole %) | I-2-1 | I-2-4 | I-2-5 | I-2-8 | I-2-9 | I-2-11 | I-2-13 | I-2-15 | I-2-17 | I-2-19 |
|---|---|---|---|---|---|---|---|---|---|---|
| Methane | 0.08 | 0.11 | 0.11 | 0.65 | 0.37 | 0.41 | 0.20 | 0.25 | 0.25 | 0.06 |
| 2-$H_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.02 | 0.02 | 0.03 | 0.03 |
| 4-He | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 18-$H_2O$ | 0.35 | 0.14 | 0.42 | 0.19 | 0.37 | 0.72 | 0.09 | 0.23 | 0.38 | 0.56 |
| 26-$C_2H_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 28-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 29-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 30-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 28-$N_2$ | 92.29 | 93.68 | 92.58 | 93.19 | 91.79 | 82.13 | 86.69 | 88.42 | 84.81 | 82.05 |
| 28-$C_2H_4$ | 0.00 | 0.01 | 0.00 | 0.04 | 0.02 | 0.02 | 0.01 | 0.01 | 0.01 | 0.01 |
| 30-$C_2H_6$ | 0.00 | 0.00 | 0.01 | 0.03 | 0.02 | 0.02 | 0.01 | 0.01 | 0.01 | 0.01 |
| 32-$O_2$ | 2.47 | 1.82 | 2.20 | 2.06 | 2.34 | 13.17 | 10.33 | 4.47 | 9.18 | 12.69 |
| 40-Ar | 1.14 | 1.15 | 1.13 | 1.15 | 1.12 | 1.03 | 1.07 | 1.09 | 1.03 | 1.01 |
| 44-$CO_2$ | 3.55 | 2.96 | 3.34 | 2.29 | 3.67 | 2.30 | 1.50 | 5.21 | 4.09 | 3.50 |
| 45-$CO_2$ | 0.05 | 0.04 | 0.04 | 0.04 | 0.05 | 0.03 | 0.03 | 0.07 | 0.05 | 0.05 |
| 46-$CO_2$ | 0.02 | 0.02 | 0.03 | 0.00 | 0.02 | 0.00 | 0.00 | 0.03 | 0.02 | 0.02 |
| 47-$CO_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 44-$C_3H_8$ | 0.04 | 0.07 | 0.14 | 0.37 | 0.23 | 0.18 | 0.06 | 0.18 | 0.13 | 0.03 |
| Gas Pressure (atm) | 1.741 | 1.668 | 1.703 | 1.498 | 1.648 | 1.817 | 1.753 | 1.785 | 1.759 | 1.880 |
| $O_2/N_2$ Ratio | 0.027 | 0.019 | 0.024 | 0.022 | 0.026 | 0.016 | 0.119 | 0.051 | 0.108 | 0.155 |
| $Ar/N_2$ Ratio | 0.012 | 0.012 | 0.012 | 0.012 | 0.012 | 0.013 | 0.012 | 0.012 | 0.012 | 0.012 |
| Time in Can (Days) | 56 | 61 | 61 | 61 | 60 | 60 | 59 | 59 | 58 | 57 |

TABLE 23

Mass Spectrometric Results for the Tazewell County, Illinois, Samples

| Components (Mole %) | I-3-3 | I-3-5 | I-3-8 | I-3-10 | I-3-13 | I-3-15 | I-3-18 | I-3-20 |
|---|---|---|---|---|---|---|---|---|
| Methane | 0.48 | 0.40 | 0.45 | 0.63 | 0.67 | 0.86 | 0.30 | 0.08 |
| 2-$H_2$ | 0.02 | 0.01 | 0.00 | 0.01 | 0.02 | 0.02 | 0.00 | 0.00 |
| 4-He | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 18-$H_2O$ | 0.66 | 0.65 | 1.15 | 0.17 | 0.28 | 0.49 | 0.78 | 0.96 |
| 26-$C_2H_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 28-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 29-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 30-CO | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 28-$N_2$ | 94.52 | 92.19 | 91.22 | 94.17 | 87.68 | 87.89 | 81.87 | 82.93 |
| 28-$C_2H_4$ | 0.00 | 0.01 | 0.02 | 0.06 | 0.06 | 0.12 | 0.05 | 0.02 |
| 30-$C_2H_6$ | 0.00 | 0.01 | 0.01 | 0.03 | 0.03 | 0.07 | 0.03 | 0.01 |
| 32-$O_2$ | 1.56 | 3.61 | 3.20 | 1.66 | 8.43 | 8.45 | 13.91 | 13.27 |
| 40-Ar | 1.15 | 1.13 | 1.13 | 1.16 | 1.09 | 1.10 | 1.02 | 1.05 |
| 44-$CO_2$ | 1.53 | 1.88 | 2.62 | 1.75 | 1.46 | 0.59 | 1.83 | 1.54 |
| 45-$CO_2$ | 0.02 | 0.02 | 0.04 | 0.03 | 0.02 | 0.01 | 0.03 | 0.02 |
| 46-$CO_2$ | 0.00 | 0.01 | 0.02 | 0.02 | 0.00 | 0.00 | 0.00 | 0.01 |
| 47-$CO_2$ | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 44-$C_3H_8$ | 0.05 | 0.07 | 0.13 | 0.31 | 0.26 | 0.39 | 0.20 | 0.10 |
| Gas Pressure (atm) | 1.478 | 1.572 | 1.689 | 1.638 | 1.661 | 1.675 | 1.727 | 1.660 |
| $O_2/N_2$ Ratio | 0.017 | 0.036 | 0.036 | 0.018 | 0.096 | 0.096 | 0.170 | 0.160 |
| $Ar/N_2$ Ratio | 0.012 | 0.012 | 0.012 | 0.012 | 0.012 | 0.012 | 0.012 | 0.012 |
| Time in Can (Days) | 26 | 26 | 25 | 25 | 25 | 25 | 25 | 24 |

## D. CLAY MINERALOGY

Core samples from the I-1, P-1, KY-2 and VA-1 wells have been analyzed for clay mineral content.

Three patterns were made for each sample: untreated, ethylene glycol, and 300°C. Illite, mixed-layer illite-montmorillonite, and chlorite are present. Minor amounts of siderite, dolomite, and feldspar are present in some samples. Kaolinite is present in minor amounts only in the KY-2 well. This may be due to a difference in source material or to the shallow depth of burial (low temperature). In the KY-2 well, haolinite decreases with depth; however, the mixed layer clay tends to increase suggesting that these trends are depositional. In the VA-1 well, the mixed layer clay decreases with depth as the illite content increases. This may be due to burial diogenesis as the sharpness ratio increases and the crystallinity index decreases with depth. As the illite peaks become narrower (due to increased crystallinity, increased grain size or loss of expanded layers) the sharpness ratio (S.R. = $\frac{10Å}{10.5Å}$) increases and the crystal index (C.I. = width at half-height in mm) decreases.

Regionally, the clay mineral suite is relatively uniform except for the kaolinite in the KY-2 well and a relatively low mixed-layer value for the I-1 well. The specific data are listed in Tables 24 through 27 and Figure 1 shows the average results of the present study in contrast with Weaver's (Geochim et Cosmochim, Acta, 31, 2181-2196, 1967) estimate of the clay mineral composition of North America versus time. The data are too limited to make any stratigraphic interpretations at this time.

The S.R. for the VA-1 well averages 4.01 and for the KY-2 well averages 4.28. The C.I. values average 1.82 and 1.65 respectively. The differences are probably not statistically significant. The P-1 well samples have a higher S.R. (5.15) and lower C.I. (1.5) than those from the VA-1 and KY-2 wells. The I-1 well has a lower S.R. (3.34) and a similar C.I. (1.68). Though the values are too few to be significant, the data indicate that the P-1 well samples have probably been exposed to higher temperatures than those in the other three wells.

TABLE 24

Clay Content - Well KY-2

| Mound 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 |
|---|---|---|---|---|---|---|---|---|
| Depth | 2444' | 2475' | 2504' | 2535' | 2555' | 2597' | 2627' | 2658' |
| Percent Distribution | | | | | | | | |
| Montmorillonite | | | | | | | | |
| Illite | 72 | 75 | 72 | 75 | 60 | 73 | 60 | 77 |
| Mixed-Layer | 17 | 16 | 9 | 17 | 33 | 15 | 33 | 13 |
| Kaolinite | 5 | 4 | 14 | 3 | 3 | 6 | 4 | 5 |
| Chlorite | 6 | 5 | 5 | 5 | 4 | 6 | 3 | 5 |
| Crystallinity | 1.3 | 1.8 | 1.6 | 1.7 | 1.5 | 1.5 | 1.3 | 2.0 |
| Sharpness Ratio | 3.8 | 3.3 | 4.5 | 5.1 | 5.3 | 5.1 | 5.1 | 3.7 |
| 10Å Peak Height | 49 | 70 | 58 | 102 | 97 | 86 | 122 | 52 |

TABLE 24 (Cont'd.)

Clay Content - Well KY-2

| Mound Sample No. | KY-2-18 | KY-2-20 | KY-2-21 | KY-2-24 | KY-2-25 | KY-2-27 | KY-2-29 | KY-2-32 |
|---|---|---|---|---|---|---|---|---|
| Depth | 2688' | 2716' | 2745' | 2774' | 2804' | 2862' | 2883' | 2891' |
| Percent Distribution | | | | | | | | |
| Montmorillonite | | | | | | | | |
| Illite | 67 | 72 | 83 | 72 | 74 | 73 | 57 | 68 |
| Mixed-Layer | 21 | 18 | 9 | 20 | 16 | 16 | 35 | 22 |
| Kaolinite | 6 | 4 | 3 | 3 | 4 | 4 | 2 | 0 |
| Chlorite | 6 | 6 | 5 | 5 | 6 | 7 | 6 | 10 |
| Crystallinity | 1.8 | 1.5 | 1.5 | 1.7 | 1.7 | 1.2 | 2.0 | 1.8 |
| Sharpness Ratio | 4.4 | 3.7 | 4.2 | 4.6 | 4.2 | 6.3 | 3.3 | 3.9 |
| 10Å Peak Height | 48 | 63 | 101 | 88 | 117 | 146 | 72 | 108 |

TABLE 24 (Cont'd.)

Clay Content - Well KY-2 (Cont'd)

| Mound Sample No. | KY-2-34 | KY-2-35 | KY-2-38 | KY-2-40 | KY-2-42 | KY-2-44 | KY-2-45 | KY-2-47 |
|---|---|---|---|---|---|---|---|---|
| Depth | 2922' | 2948' | 2978' | 3006' | 3036' | 3055' | 3085' | 3114' |
| Percent Distribution | | | | | | | | |
| Montmorillonite | | | | | | | | |
| Illite | 63 | 62 | 64 | 61 | 77 | 62 | 77 | 66 |
| Mixed-Layer | 25 | 32 | 28 | 33 | 16 | 32 | 13 | 27 |
| Kaolinite | 0 | 2 | 0 | 2 | 0 | 0 | 0 | 0 |
| Chlorite | 12 | 4 | 8 | 4 | 7 | 6 | 10 | 7 |
| Crystallinity | 1.3 | 1.8 | 1.7 | 1.5 | 1.7 | 1.3 | 1.5 | 1.8 |
| Sharpness Ratio | 3.8 | 3.1 | 4.1 | 4.5 | 4.3 | 5.5 | 4.3 | 3.6 |
| 10Å Peak Height | 57 | 97 | 82 | 81 | 52 | 55 | 61 | 97 |

TABLE 24 (Cont'd.)

Clay Content Well KY-2 (Cont'd)

| Mound Sample No. | KY-2-50 | KY-2-51 | KY-2-54 | KY-2-55 | KY-2-58 | KY-2-60 | KY-2-62 | KY-2-63 | KY-2-66 |
|---|---|---|---|---|---|---|---|---|---|
| Depth | 3144' | 3171' | 3201' | 3230' | 3261' | 3288' | 3329' | 3358' | 3386' |
| Percent Distribution | | | | | | | | | |
| Montmorillonite | | | | | | | | | |
| Illite | 65 | 73 | 63 | | 76 | 69 | 66 | 74 | 75 |
| Mixed-Layer | 25 | 13 | 27 | | 10 | 18 | 23 | 18 | 16 |
| Kaolinite | 2 | 4 | 0 | | 3 | 0 | 0 | 0 | 0 |
| Chlorite | 8 | 10 | 10 | | 13 | 13 | 11 | 8 | 9 |
| Crystallinity | 2.1 | 1.8 | 2.2 | | 1.6 | 1.7 | 2.0 | 1.4 | 1.7 |
| Sharpness Ratio | 3.9 | 4.0 | 3.0 | | 4.8 | 5.2 | 4.0 | 3.8 | 4.4 |
| 10Å Peak Height | 162 | 127 | 133 | | 200 | 235 | 227 | 42 | 70 |

TABLE 25

Clay Content - Well I-1

| Mound Sample No. | I-1-1 | I-1-4 | I-1-6 | I-1-8 | I-1-9 |
|---|---|---|---|---|---|
| Depth | 3023' | 3044' | 3057' | 3075' | 3099' |
| Percent Distribution: | | | | | |
| Montmorillonite | | | | | |
| Illite | 71 | 87 | 73 | 72 | 66 |
| Mixed-Layer | 17 | 10 | 10 | 13 | 22 |
| Kaolinite | 0 | 0 | 0 | 0 | 0 |
| Chlorite | 12 | 13 | 17 | 15 | 12 |
| Crystallinity | 2.0 | 1.5 | 1.4 | 1.8 | 1.7 |
| Sharpness Ratio | 2.9 | 4.0 | 3.1 | 3.0 | 3.7 |

TABLE 26

Clay Content - Well P-1

| Mound Sample No. | P-1-1 | P-1-2 | P-1-3 |
|---|---|---|---|
| Depth | 2492' | 2521' | 2549' |
| Percent Distribution | | | |
| Montmorillonite | | | |
| Illite | 59 | 77 | 73 |
| Mixed-Layer | 32 | 14 | 15 |
| Kaolinite | 0 | 0 | 0 |
| Chlorite | 9 | 9 | 12 |
| Crystallinity | 1.3 | 1.5 | 1.7 |
| Sharpness Ratio | 4.7 | 6.2 | 4.5 |
| 10Å Peak Height | 166 | 31 | 72 |

TABLE 27

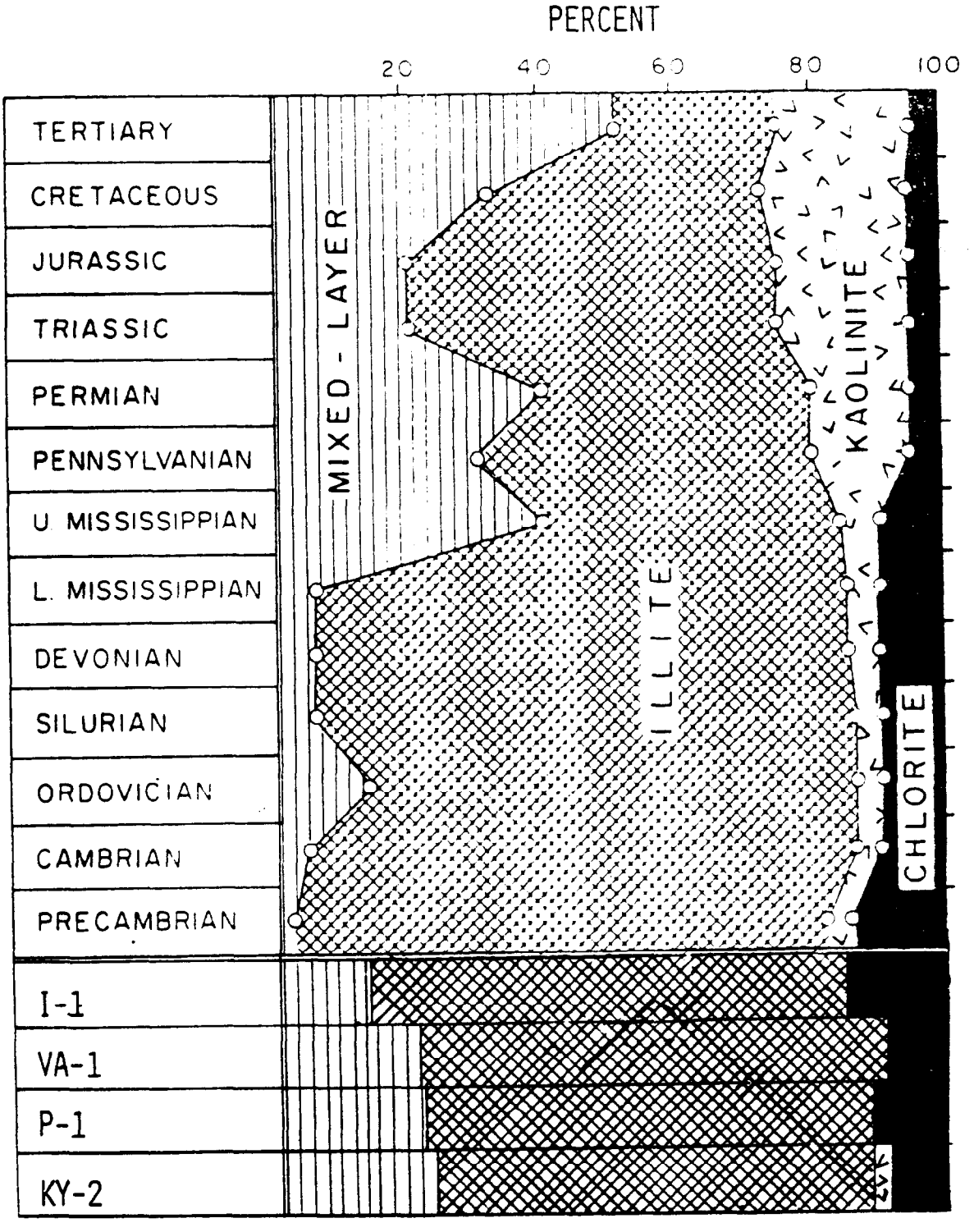
Clay Content - Well VA-1

| Mound Sample No. | VA-1-2 | VA-1-4 | VA-1-6 | VA-1-11 | VA-1-13 | VA-1-15 | VA-1-17 |
|---|---|---|---|---|---|---|---|
| Depth | 4885' | 4915' | 4945' | 4975' | 5229' | 5259' | 5289' |
| Percent Distribution | | | | | | | |
| Montmorillonite | | | | | | | |
| Illite | 81 | 64 | 72 | 61 | 61 | 50 | 77 |
| Mixed-Layer | 12 | 30 | 21 | 28 | 30 | 42 | 13 |
| Kaolinite | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Chlorite | 7 | 6 | 7 | 11 | 9 | 8 | 10 |
| Crystallinity | 2.2 | 1.7 | 2.3 | 1.8 | 2.0 | 2.3 | 2.0 |
| Sharpness Ratio | 3.2 | 3.7 | 2.9 | 3.6 | 3.1 | 2.6 | 3.3 |
| 10Å Peak Height | 38 | 60 | 109 | 112 | 108 | 85 | 112 |

TABLE 27 (Cont'd.)

Clay Content - Well VA-1

| Mound Sample No. | VA-1-18 | VA-1-21 | VA-1-22 | VA-1-25 | VA-1-26 | VA-1-27 | VA-1-29 | VA-1-32 |
|---|---|---|---|---|---|---|---|---|
| Depth | 5319' | 5348' | 5379' | 5393' | 5393' | 5408' | 5439' | 5469' |
| Percent Distribution | | | | | | | | |
| Montmorillonite | | | | | | | | |
| Illite | 76 | 73 | 75 | 79 | 83 | 79 | 76 | 85 |
| Mixed-Layer | 14 | 19 | 18 | 13 | 10 | 11 | 16 | 7 |
| Kaolinite | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Chlorite | 10 | 8 | 7 | 8 | 7 | 10 | 8 | 8 |
| Crystallinity | 1.9 | 1.5 | 1.7 | 1.7 | 1.7 | 1.3 | 1.3 | 1.9 |
| Sharpness Ratio | 4.3 | 4.0 | 4.1 | 4.0 | 4.6 | 5.2 | 5.3 | 6.3 |
| 10Å Peak Height | 104 | 86 | 54 | 98 | 55 | 119 | 55 | 88 |




CLAY MINERALOGY RESULTS

FIGURE 1

The amount of mixed-layer clay is calculated using the height of the 10Å peak of the ethylene glycol treated sample and the 10Å peak of the sample heated to 300°C. The increase in peak height after heating is assumed to be due to the collapse of expanded layers in the mixed-layer clay. In the untreated samples, the mixed-layer peak occurs as a shoulder on the low angle side of the 10Å illite peak. Its apex appears to be in the range of 10.5 to 11Å or less; on glycolation there is little change in the peak position. The mixed-layer clay apparently contains 10 to 20% expanded layers.

The presence of this mixed layer clay suggests the shale has not been heated to temperatures higher than 200 to 250°C. If sufficient potassium is available, the mixed-layer phase should convert to illite at 200 to 250°C. If the mixed layer (4:1) clay originally had a higher content of expanded layers, it has probably been exposed to temperatures higher than approximately 100°C. Unfortunately, little is known about the effects of time on clay diagenesis. Time should tend to lower both the temperature values given above.

Figure 2 is a plot of 10Å peak height vs. percent organic carbon for all samples. As expected, there is a good inverse linear relation for most samples. At this time, there is no apparent reason why five samples fall off the trend. The samples from the I-1 well were not plotted as they were run on a different x-ray machine, and the intensities are not comparable.

Figure 3 contains plots showing the relation of the C.I. and percent mixed-layer clay with depth in the Ky-2 well. There is a large amount of scatter. The mixed-layer content appears to increase and the crystallinity decrease (C.I. increase) with depth. The interval is so short and the depth so shallow that major diagenetic effects are not apparent. Figure 4 contains a plot of the sharpness ratio vs. depth for the KY-2 well. Also shown is a three point moving average plot of the S.R. values. The S.R. appears to decrease slightly with depth.

This study is still in progress and should identify, as more data becomes available, the quantitative relationship between burial diagenesis of clay minerals and the hydrocarbon generation.

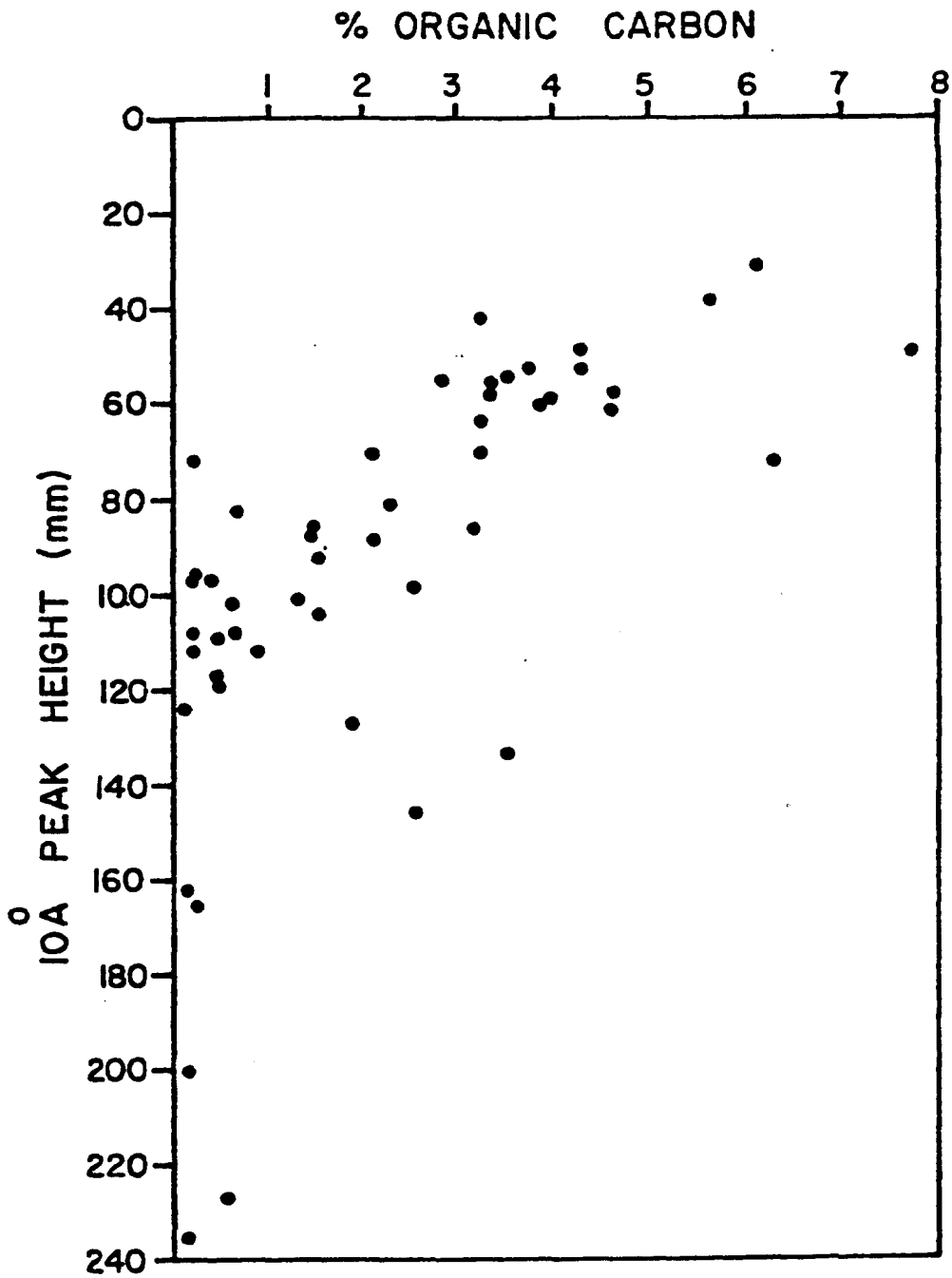

% ORGANIC CARBON
1
2
3
4
5
6
7
8
0
20
40
60
80
100
120
140
160
180
200
220
240
IOA PEAK HEIGHT (mm)

FIGURE 2

KY-2 WELL

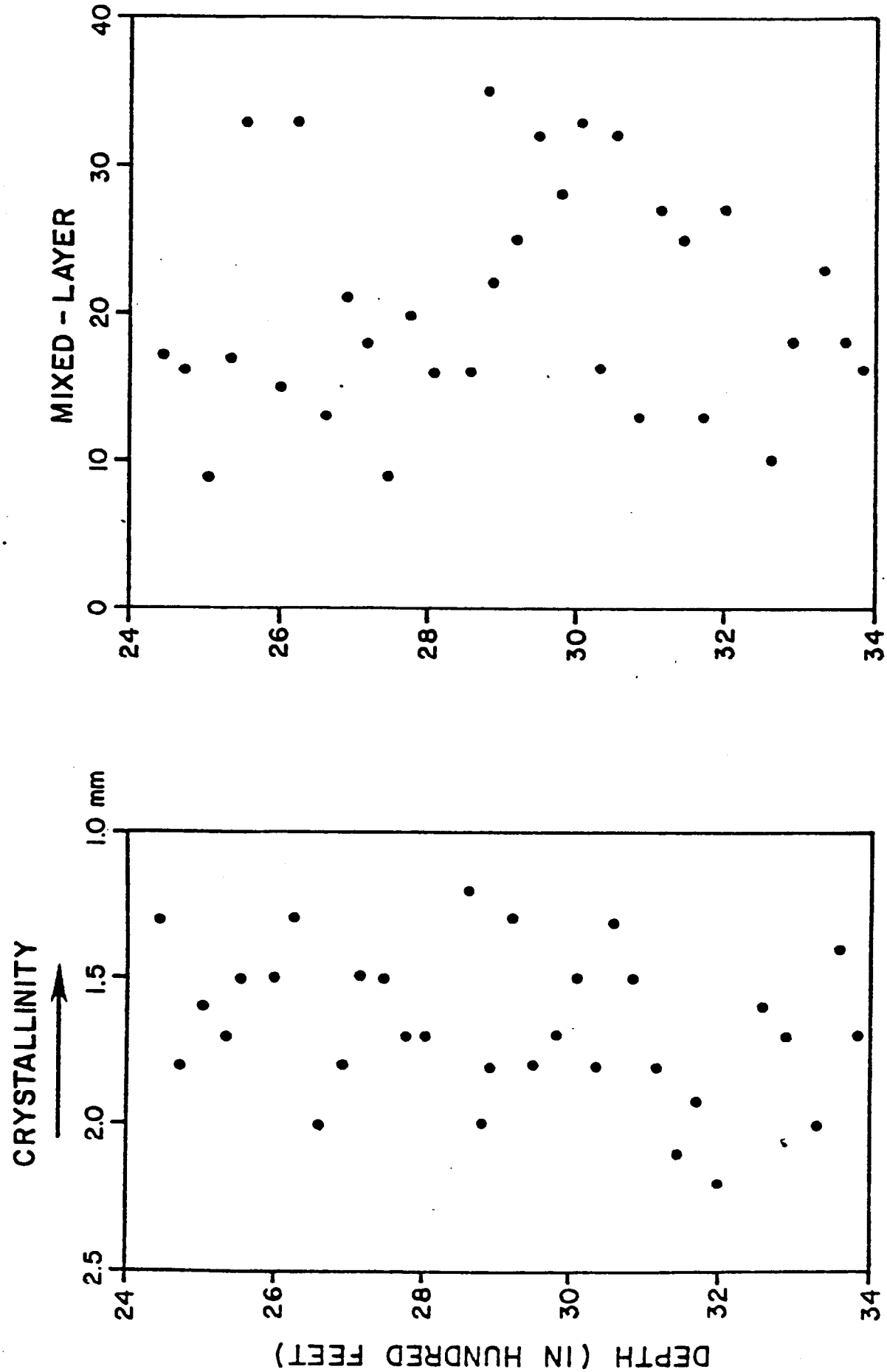



FIGURE 3

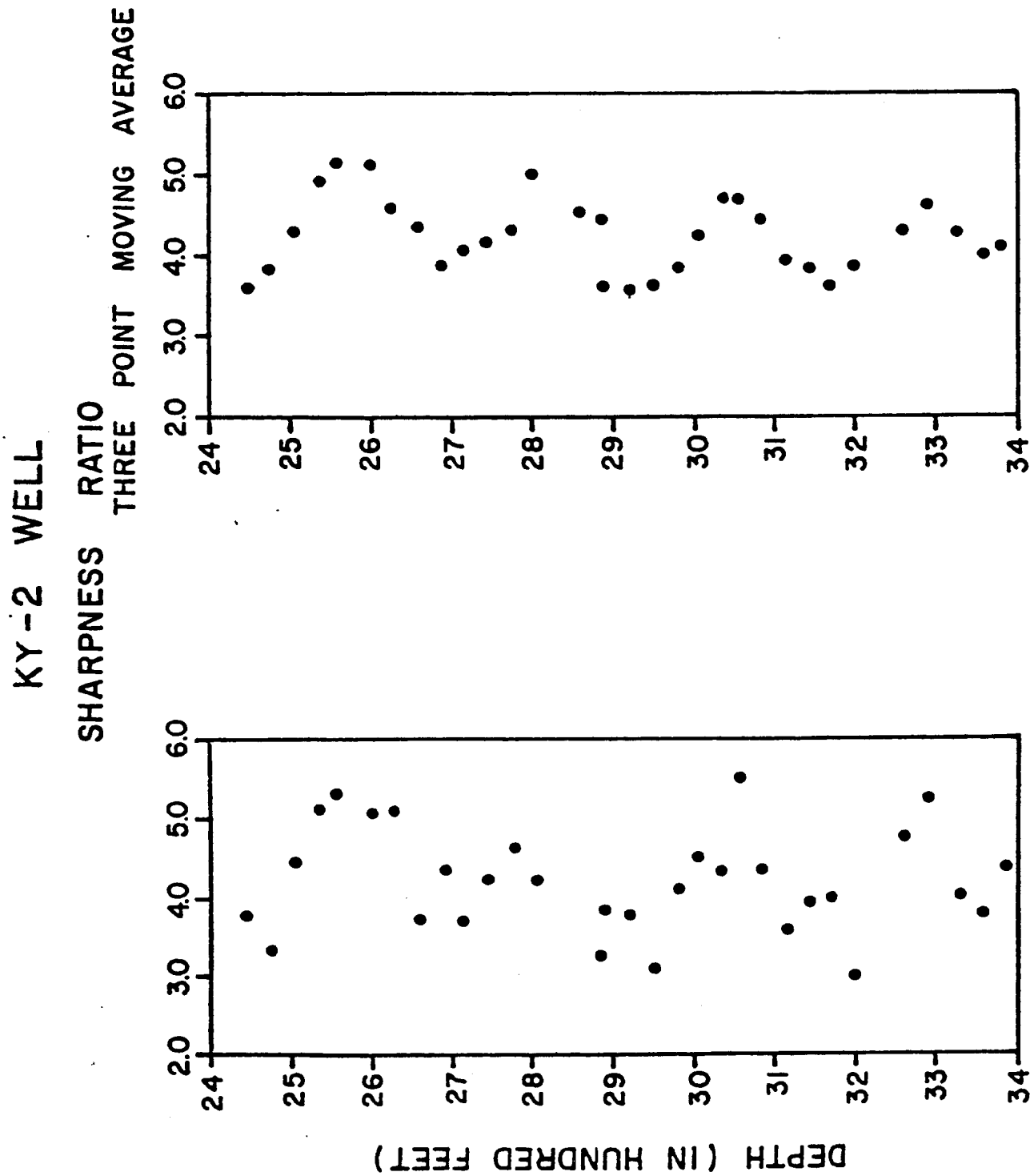

KY-2 WELL
SHARPNESS RATIO
THREE POINT MOVING AVERAGE
2.0
3.0
4.0
5.0
6.0
24
25
26
27
28
29
30
31
32
33
34
DEPTH (IN HUNDRED FEET)

FIGURE 4

## E. STABLE CARBON ISOTOPE GEOCHEMISTRY

Samples of shale core from five wells have been analyzed by mass spectrometry for stable carbon isotope values. Prior to analysis, the inorganic carbon was removed by acidification. The total organic carbon was converted to $CO_2$ and its delta$^{13}$C was measured relative to PDB. The delta$^{13}$C results show a strong correlation to the organic matter type. In general, the samples classified as gas prone, restricted marine or nonmarine are characterized by delta$^{13}$C values ranging from -24.6 to about -28. The oil prone, more marine type of organic matter composed mostly of herbaceous and amorphous kerogen is characterized by isotopically lighter delta$^{13}$C values (-27 to-31). Four of the five wells have been exposed to a similar thermal history. The organic matter from the VA-1 is more thermally mature (Table 28). Delta$^{13}$C values from the nonmarine shales (gas prone) from VA-1 appear to be isotopically lighter than the nonmarine shales in the other four wells. Increasing thermal maturation on kerogens of similar type usually produces a kerogen which becomes increasingly heavier relative to $^{13}C/^{12}C$. The data indicate that the opposite effect has taken place, namely the organic matter has become slightly lighter. Previous studies performed on crude oils and kerogens have shown that delta$^{13}$C values of -23 to -29 are indicative of marine derived organic matter. Values of -29 to -33 usually indicate marine organic matter. The initial results obtained on selected Eastern gas shales seems to show an inverse relationship when compared to previous carbon isotope studies

More samples from other wells will be analyzed to establish if this inverse relationship does exist and why it does occur.

## F. DETAILED HYDROCARBON ANALYSIS

The detailed hydrocarbon analyses have been completed on all wells through I-3. There analyses included $C_1$-$C_7$ hydrocarbon analyses, detailed $C_4$-$C_7$ hydrocarbons, soxhlet extraction and liquid chromatography of $C_{15}$ + bitumen, gas chromatography of $C_{15}$ + paraffin-naphthene hydrocarbons, lithologic description, vitrinite reflectance, organic carbon and detailed kerogen composition.

TABLE 28

Delta$^{13}$C of Organic Carbon in Whole Rock

| Well | Mound Sample Number | Depth | Organic Matter Type* | Delta$^{13}$C PDB of Organic Carton in Whole Rock |
|---|---|---|---|---|
| KY-2 | KY-2-2 | 2443' | H;Am;W(C) | -30.2 |
| | KY-2-8 | 2535' | W-C;H;- | -27.5 |
| | KY-2-20 | 2715' | H;Am;W-C | -30.0 |
| | KY-2-45 | 3085' | H;Am;- | -28.9 |
| | KY-2-60 | 3288' | H;W-C;- | -26.7 |
| VA-1 | VA-1-2 | 4885' | H;W;Am-C | -27.9 |
| | VA-1-6 | 4945' | W;H;C(Am) | -25.6 |
| | VA-1-18 | 5319' | H-W;-;Am-C | -28.1 |
| | VA-1-26 | 5393' | H;W;C(Am) | -29.3 |
| | VA-1-32 | 5469' | H;W;Am-C | -28.7 |
| P-1 | P-1-1 | 2492' | W;C;H(Am) | -24.6 |
| | P-1-3 | 2549' | Am;H;W-C | -28.0 |
| O-1 | O-1-1 | 2184' | H;Am;W | -31.6 |
| | O-1-5 | 2317' | Am-H;-;- | -30.3 |
| I-1 | I-1-4 | 3044' | H;Am;W-c | -29.0 |
| | I-1-8 | 3075' | H;Am;- | -28.9 |

* Organic Matter Type

| | |
|---|---|
| Marine | Al = Algal |
| Marine | Am = Amorphous |
| Restricted Marine | H = Herbaceous |
| Nonmarine | W = Woody |
| Nonmarine | C = Coaly (Inertinite) |

Tables 29 through 42 list the balance of the $C_1$-$C_7$.

Previously reported data can be found in MLM-ML-7743-0002 and MLM-ML-77-46-0001, the two earlier reports in this series. Total $C_1$-$C_7$ data for five of the wells examined to date are presented in Figure 5.

Gas and gasoline-range hydrocarbons are generated from organic matter at different levels of thermal maturity. Initially, at low temperature, microbial degradation forms methane or "dry" gas. With increasing time, temperature, and depth of burial, heavier hydrocarbons are generated. In the early stages of petroleum formation, gas-oil range hydrocarbons predominate with associated amounts of "wet" gas ($C_2$-$C_4$ hydrocarbons) and gasoline-range hydrocarbons. The content and distribution of $C_1$-$C_7$ hydrocarbons is primarily dependent on three geochemical parameters; namely, the organic richness, the type of organic matter, and its level of thermal maturity.

Shales from most of the wells analyzed to date contained large quantities of $C_1$-$C_7$ hydrocarbons, on a relative basis this was not true for the I-2 and I-3 wells. For the I-1, O-1, P-1, VA-1 and I-3 wells, the dry gas, wet gas and gasoline-range hydrocarbons were uniformaily distributed. The gasoline-range hydrocarbons in the VA-1 well are exceptionally low, but the lithalogy of the samples from this well indicated that coal was present in most of the samples. In the I-2 well, a variation was noted in the sample at 508 feet. The reason for this variance is not obvious at the present time. In the KY-2 well, the hydrocarbons varied significantly throughout the entire well profile. This is apparently the result of a rich organic facies being interbedded in a lean organic facies.

The data also indicate that for the most part the majority of the $C_1$-$C_7$ hydrocarbons are released to the headspace of the sample canisters. The lowest values of release are recorded for the three Illinois basin wells O-1, I-2 and I-3. The samples from well O-1 retain the most gas in the matrix and other investigators (Thomas and Frost, Third ERDA Symposium on Enhoused Oil and Gas Recovery and Improved Drilling Methods, Tulsa, August, 1977) have indicated that samples from the O-1 well contain ultra-microporous networks which inhibit the diffusion of hydrocarbon gases from the material.

The hydrocarbon concentrations that have been reported are <u>minimum</u> values because the core samples are exposed

TABLE 29

$C_1$-$C_7$ Hydrocarbons

| Mound Sample Number | Depth | 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 % | $ic_4/nC_4$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| A. $C_1$-$C_7$ Hydrocarbons in Air Space | | | | | | | | | | | |
| P-1-1* | 2492 | 88 | 2716 | 1636 | 1980 | 215 | 4959 | 11594 | 11506 | 99.2 | 9.2 |
| P-1-2* | 2521 | 177944 | 32172 | 1329 | 807 | 57 | 3382 | 215691 | 37747 | 17.5 | 14.2 |
| P-1-3* | 2549 | 1935 | 297 | 353 | 153 | 1086 | 3086 | 6910 | 4975 | 72.0 | 0.1 |
| B. $C_1$-$C_7$ Hydrocarbons in Core Material | | | | | | | | | | | |
| P-1-1 | 2492 | 2035 | 145 | 89 | 18 | 113 | 1027 | 3427 | 1392 | 40.6 | 0.2 |
| P-1-2 | 2521 | 26409 | 27479 | 2533 | 3429 | 291 | 3092 | 63233 | 36824 | 58.2 | 11.8 |
| P-1-3 | 2549 | 3116 | 1027 | 1043 | 2355 | 233 | 4522 | 12296 | 9180 | 74.7 | 10.1 |
| C. $C_1$-$C_7$ Hydrocarbons in Air Space and Core Material | | | | | | | | | | | |
| P-1-1 | 2492 | 2123 | 2861 | 1725 | 1998 | 328 | 5986 | 15021 | 12898 | 85.9 | 6.1 |
| P-1-2 | 2521 | 204353 | 59651 | 3862 | 4236 | 348 | 6474 | 278924 | 74571 | 26.7 | 12.2 |
| P-1-3 | 2549 | 5051 | 1324 | 1396 | 2508 | 1319 | 7608 | 19206 | 14155 | 73.7 | 1.9 |

ppm values expressed as volumes of gas per million volumes of core material.

*Samples were exposed to air for an extended period of time prior to being canned.

TABLE 30

$C_1$-$C_7$ Hydrocarbon Analyses of Head 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 % | $iC_4/nC_4$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| VA-1-2 | 4885.4-4885.75 | 1510422 | 232494 | 22275 | 1307 | 1659 | 38 | 1768157 | 257735 | 14.6 | 0.8 |
| VA-1-4 | 4915.37-4915.68 | 1300687 | 158568 | 15943 | 967 | 1457 | 230 | 1477622 | 176935 | 12.0 | 0.7 |
| VA-1-6 | 4945.43-4945.77 | 173739 | 13202 | 1241 | 57 | 74 | 9 | 188313 | 14574 | 7.7 | 0.8 |
| VA-1-11 | 4975.32-4975.54 | 89903 | 7379 | 764 | 0 | 46 | 2 | 98092 | 8189 | 8.3 | -- |
| VA-1-13 | 5229.35-5229.65 | 461123 | 33630 | 3207 | 215 | 196 | 6 | 498371 | 37248 | 7.5 | 1.1 |
| VA-1-15 | 5259.25-5259.65 | 1020409 | 118567 | 8733 | 536 | 567 | 45 | 1148812 | 128403 | 11.2 | 0.9 |
| VA-1-17 | 5289.46-5289.60 | 1340801 | 144916 | 14078 | 931 | 1084 | 0 | 1501810 | 161009 | 10.7 | 0.9 |
| VA-1-18 | 5319.37-5319.56 | 2071391 | 184858 | 14524 | 759 | 780 | 1 | 2272312 | 200921 | 8.8 | 1.0 |
| VA-1-21 | 5348.62-5348.80 | 2975296 | 311270 | 23719 | 953 | 1579 | 0 | 3312817 | 337521 | 10.2 | 0.6 |
| VA-1-22 | 5379.08-5379.33 | 2326394 | 385470 | 22917 | 849 | 938 | 0 | 2736568 | 410174 | 15.0 | 0.9 |
| VA-1-25* | 5393.50-5393.65 | 27667 | 5858 | 634 | 32 | 39 | 4 | 34230 | 6563 | 19.2 | 0.8 |
| VA-1-26 | 5393.65-5393.90 | 2331226 | 285052 | 20182 | 1011 | 1105 | 139 | 2638576 | 307350 | 11.6 | 0.9 |
| VA-1-27 | 5408.07-5408.37 | 270384 | 15364 | 1082 | 38 | 41 | 0 | 286909 | 16525 | 5.8 | 0.9 |
| VA-1-29 | 5439.15-5439.40 | 3357290 | 429227 | 23903 | 825 | 728 | 0 | 3811973 | 454683 | 11.9 | 1.1 |
| VA-1-32 | 5469.53-5469.80 | 1543196 | 132697 | 8036 | 283 | 253 | 0 | 1684465 | 141269 | 8.4 | 1.1 |

ppm values expressed as volumes of gas per million volumes of core material.

*Sample stored in water.

UGR File #C051/08Q-
Oct.-Dec. 1977
Mound Facility

TABLE 31

$C_1$-$C_7$ 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 | Wetness % | $iC_4/nC_4$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| VA-1-2 | 4885.4-4885.75 | 24098 | 56992 | 16642 | 1395 | 1261 | 153 | 100541 | 76443 | 76.0 | 1.1 |
| VA-1-4 | 4915.37-4915.68 | 22432 | 53735 | 17496 | 1606 | 1510 | 309 | 97088 | 74656 | 76.9 | 1.1 |
| VA-1-6 | 4945.43-4945.77 | 2236 | 2116 | 1215 | 145 | 222 | 50 | 6084 | 3748 | 61.6 | 0.7 |
| VA-1-11 | 4975.32-4975.54 | 2512 | 2681 | 1429 | 188 | 244 | 147 | 7201 | 4689 | 65.1 | 0.8 |
| VA-1-13 | 5229.35-5229.65 | 6078 | 2043 | 1236 | 205 | 221 | 99 | 9882 | 3804 | 38.5 | 0.9 |
| VA-1-15 | 5259.25-5259.65 | 6837 | 19982 | 6524 | 682 | 501 | 110 | 34636 | 27799 | 80.3 | 1.4 |
| VA-1-17 | 5289.46-5289.60 | 9718 | 18006 | 6302 | 573 | 447 | 81 | 35127 | 25409 | 72.3 | 1.3 |
| VA-1-18 | 5319.37-5319.56 | 25921 | 34495 | 6946 | 531 | 416 | 38 | 68347 | 42426 | 62.1 | 1.3 |
| VA-1-21 | 5348.62-5348.80 | 21432 | 104047 | 12607 | 670 | 563 | 60 | 139379 | 117947 | 84.6 | 1.2 |
| VA-1-22 | 5379.08-5379.33 | 21786 | 63268 | 10288 | 567 | 502 | 18 | 96429 | 74643 | 77.4 | 1.1 |
| VA-1-25* | 5393.50-5393.65 | 30444 | 39621 | 6627 | 375 | 330 | 15 | 77412 | 46968 | 60.7 | 1.1 |
| VA-1-26 | 5393.65-5393.90 | 54265 | 72874 | 9561 | 512 | 438 | 100 | 137750 | 83485 | 60.6 | 1.2 |
| VA-1-27 | 5408.07-5408.37 | 3276 | 2973 | 1700 | 164 | 179 | - | 8292 | 5016 | 60.5 | 0.9 |
| VA-1-29 | 5439.15-5439.40 | 38925 | 72813 | 9017 | 461 | 405 | 44 | 121665 | 82740 | 68.0 | 1.1 |
| VA-1-32 | 5469.53-5469.80 | 75349 | 62091 | 8712 | 487 | 432 | 49 | 147120 | 71771 | 48.8 | 1.1 |

ppm values expressed as volumes of gas per million volumes of core material.

*Sample stored in water.

TABLE 32

$C_1$-$C_7$ 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 $C_3$-$C_4$ PPM | Total $C_2$-$C_4$ PPM | Gas Wetness % | $iC_4/nC_4$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| VA-1-2 | 4885.4-4885.75 | 1534520 | 289486 | 38917 | 2702 | 2920 | 191 | 1868736 | 334216 | 17.9 | 0.9 |
| VA-1-4 | 4915.37-4915.68 | 1323119 | 212303 | 33439 | 2573 | 2967 | 539 | 1574940 | 251821 | 16.0 | 0.9 |
| VA-1-6 | 4945.43-4945.77 | 175975 | 15318 | 2456 | 202 | 296 | 59 | 194306 | 18431 | 9.5 | 0.7 |
| VA-1-11 | 4975.32-4975.54 | 92415 | 10060 | 2193 | 188 | 290 | 149 | 105295 | 12880 | 12.2 | 0.7 |
| VA-1-13 | 5229.35-5229.65 | 467201 | 35673 | 4443 | 420 | 417 | 105 | 508259 | 41058 | 8.1 | 1.0 |
| VA-1-15 | 5259.25-5259.65 | 1027246 | 138549 | 15257 | 1218 | 1068 | 155 | 1183493 | 156247 | 13.2 | 1.2 |
| VA-1-17 | 5289.46-5289.60 | 1350519 | 162922 | 20380 | 1504 | 1531 | 81 | 1536937 | 186418 | 12.1 | 1.0 |
| VA-1-18 | 5319.37-5319.56 | 2097312 | 219353 | 21470 | 1290 | 1196 | 39 | 2340660 | 243338 | 10.4 | 1.1 |
| VA-1-21 | 5348.62-5348.80 | 2996728 | 415317 | 36326 | 1623 | 2142 | 60 | 3452196 | 455468 | 13.2 | 0.8 |
| VA-1-22 | 5379.08-5379.33 | 2348180 | 448738 | 33205 | 1416 | 1440 | 18 | 2832997 | 484817 | 17.1 | 1.0 |
| VA-1-25* | 5393.50-5393.65 | 58111 | 45479 | 7261 | 407 | 369 | 19 | 111646 | 53535 | 48.0 | 1.1 |
| VA-1-26 | 5393.65-5393.90 | 2385491 | 357926 | 29743 | 1523 | 1543 | 239 | 2776465 | 390974 | 14.1 | 1.0 |
| VA-1-27 | 5408.07-5408.37 | 273660 | 18337 | 2782 | 202 | 220 | 0 | 295201 | 21542 | 7.3 | 0.9 |
| VA-1-29 | 5439.15-5439.40 | 3396215 | 502040 | 32920 | 1286 | 1133 | 44 | 3933638 | 537423 | 13.7 | 1.1 |
| VA-1-32 | 5469.53-5469.80 | 1618545 | 194788 | 16748 | 770 | 685 | 49 | 1831585 | 213089 | 11.6 | 1.1 |

ppm values expressed as volumes of gas per million volumes of core material.

*Sample stored in water.

TABLE 33

$C_1$-$C_7$ Hydrocarbon Analyses of Head 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 $C_2$-$C_7$ PPM | Total $C_1$-$C_4$ PPM | Total $C_2$-$C_4$ PPM | Gas Wetness % | $iC_4/nC_4$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| I-2-1 | 334 | 195.1 | 51.9 | 64.6 | 58.7 | 212.0 | 260.6 | 582.3 | 387.2 | 66.5 | 0.28 |
| I-2-4 | 364 | 264.0 | 77.3 | 113.8 | 112.8 | 403.7 | 497.9 | 971.6 | 707.6 | 72.8 | 0.28 |
| I-2-6 | 394 | 399.6 | 141.4 | 237.1 | 244.0 | 852.7 | 941.8 | 1874.8 | 1475.2 | 78.7 | 0.29 |
| I-2-7 | 425 | 791.9 | 296.1 | 395.4 | 315.8 | 1091.8 | 1094.7 | 2891.0 | 2099.1 | 72.6 | 0.29 |
| I-2-10 | 454 | 2532.8 | 928.4 | 1055.5 | 753.8 | 2409.6 | 1699.6 | 7680.1 | 5147.3 | 67.0 | 0.31 |
| I-2-12 | 481 | 2813.6 | 811.8 | 877.5 | 594.0 | 2030.1 | 1655.6 | 7127.0 | 4313.4 | 60.5 | 0.29 |
| I-2-14 | 508 | 642.6 | 102.2 | 130.5 | 112.7 | 389.3 | 378.8 | 1376.3 | 733.7 | 53.3 | 0.29 |
| I-2-16 | 538 | 1905.3 | 536.7 | 758.2 | 668.7 | 2315.5 | 2335.8 | 6184.9 | 4279.1 | 69.2 | 0.29 |
| I-2-18 | 568 | 1204.4 | 252.0 | 310.4 | 280.6 | 997.4 | 1238.0 | 3044.8 | 1840.4 | 60.4 | 0.23 |
| I-2-20 | 598 | 335.2 | 41.3 | 53.5 | 61.8 | 232.7 | 484.4 | 774.5 | 389.3 | 50.3 | 0.27 |

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

UGR File #C05-/78-
Oct.-Dec. 1977
Mound Facility

TABLE 34

$C_1$-$C_7$ 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 $C_3$-$C_7$ PPM | Total $C_1$-$C_4$ PPM | Total $C_2$-$C_4$ PPM | Gas Wetness % | $iC_4/nC_4$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| I-2-1 | 334 | 1283.9 | 6.9 | 6.7 | 10.9 | 58.2 | 375.8 | 1366.6 | 82.7 | 6.1 | 0.19 |
| I-2-4 | 364 | 1067.9 | 5.1 | 11.2 | 10.1 | 70.0 | 578.9 | 1164.3 | 96.4 | 8.3 | 0.14 |
| I-2-6 | 394 | 1092.7 | 17.7 | 2.7 | 3.4 | 19.6 | 1148.2 | 1136.1 | 43.4 | 3.8 | 0.17 |
| I-2-7 | 425 | 903.9 | 13.7 | 55.7 | 173.7 | 789.3 | 1935.3 | 1936.3 | 1032.4 | 53.3 | 0.22 |
| I-2-10 | 454 | 1223.3 | 467.3 | 1416.0 | 1630.4 | 5921.2 | 5967.2 | 10658.2 | 9434.9 | 88.5 | 0.28 |
| I-2-12 | 481 | 1193.7 | 721.2 | 1900.8 | 2213.8 | 7298.2 | 7165.4 | 13327.7 | 12134.0 | 91.0 | 0.30 |
| I-2-14 | 508 | 366.7 | 9.3 | 18.9 | 23.4 | 121.6 | 675.6 | 1039.9 | 173.2 | 16.7 | 0.19 |
| I-2-16 | 538 | 975.0 | 64.4 | 288.7 | 544.5 | 2376.3 | 4735.4 | 4248.9 | 3273.9 | 77.1 | 0.23 |
| I-2-18 | 568 | 994.2 | 41.4 | 211.2 | 396.9 | 1689.3 | 3895.3 | 3333.0 | 2338.8 | 70.2 | 0.23 |
| I-2-20 | 598 | 1113.1 | 15.5 | 28.2 | 23.9 | 100.7 | 1481.2 | 1281.4 | 168.3 | 13.1 | 0.24 |

TABLE 35

$C_1$-$C_7$ Hydrocarbon Analyses of Head 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 $C_3$-$C_7$ PPM | Total $C_1$-$C_4$ PPM | Total $C_2$-$C_4$ PPM | Gas Wetness % | $ic_4/nC_4$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| I-2-1 | 334 | 1479.0 | 58.8 | 71.3 | 69.6 | 270.2 | 636.4 | 1948.9 | 469.9 | 24.1 | 0.26 |
| I-2-4 | 364 | 1331.9 | 82.4 | 125.0 | 122.9 | 473.7 | 1076.8 | 2135.9 | 804.0 | 37.6 | 0.26 |
| I-2-6 | 394 | 1492.3 | 159.1 | 239.8 | 247.4 | 872.3 | 2090.0 | 3010.9 | 1518.6 | 50.4 | 0.28 |
| I-2-7 | 425 | 1695.8 | 309.8 | 451.1 | 489.5 | 1881.1 | 3030.0 | 4827.3 | 3131.5 | 64.9 | 0.26 |
| I-2-10 | 454 | 3756.1 | 1395.7 | 2471.5 | 2384.2 | 8330.8 | 7666.8 | 18338.3 | 14582.2 | 79.5 | 0.29 |
| I-2-12 | 481 | 4007.3 | 1533.0 | 2778.3 | 2807.8 | 9328.3 | 8821.0 | 20454.7 | 16447.4 | 80.4 | 0.30 |
| I-2-14 | 508 | 1509.3 | 111.5 | 149.4 | 136.1 | 509.9 | 1054.4 | 2416.2 | 906.9 | 37.5 | 0.27 |
| I-2-16 | 538 | 2880.8 | 601.1 | 1046.9 | 1213.2 | 4691.8 | 7071.2 | 10433.8 | 7553.0 | 72.4 | 0.26 |
| I-2-18 | 568 | 2198.6 | 293.4 | 521.6 | 677.5 | 2686.7 | 5133.4 | 6377.8 | 4179.2 | 65.5 | 0.25 |
| I-2-20 | 598 | 1498.3 | 56.8 | 81.7 | 85.7 | 333.4 | 1965.6 | 2055.9 | 557.6 | 27.1 | 0.26 |

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

TABLE 36

$C_1$-$C_7$ Hydrocarbon Analyses of Head Space

| ound ample umber | Well Interval (Feet) | 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 % | $iC_4/nC_4$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| -3-1 | 940 | 1465.3 | 24.3 | 75.7 | 35.7 | 164.5 | 546.8 | 1765.5 | 300.2 | 17 | 0.22 |
| -3-2 | 940 | 2134.0 | 50.0 | 145.9 | 65.7 | 261.6 | 603.4 | 2657.2 | 523.2 | 20 | 0.25 |
| -3-4 | 970 | 1754.6 | 151.3 | 339.1 | 133.2 | 477.0 | 829.4 | 2855.3 | 1100.6 | 39 | 0.28 |
| -3-6 | 995 | 535.3 | 143.6 | 273.0 | 102.7 | 368.3 | 688.6 | 1422.8 | 887.5 | 62 | 0.28 |
| -3-7 | 995 | 2804.3 | 478.8 | 716.0 | 257.7 | 933.5 | 1860.4 | 5190.4 | 2386.0 | 46 | 0.28 |
| -3-9 | 1018 | 2910.4 | 993.6 | 1747.6 | 689.2 | 2530.9 | 3608.5 | 8871.6 | 5961.2 | 67 | 0.27 |
| -3-11 | 1048 | 554.9 | 509.7 | 713.3 | 241.1 | 925.4 | 1558.4 | 2944.4 | 2389.5 | 81 | 0.26 |
| -3-12 | 1048 | 3485.7 | 1312.6 | 1664.9 | 504.9 | 1906.1 | 2622.4 | 8874.2 | 5388.4 | 61 | 0.26 |
| -3-14 | 1078 | 7764.2 | 6556.4 | 7541.2 | 1973.2 | 7225.8 | 6247.7 | 31060.8 | 23296.6 | 75 | 0.27 |
| -3-16 | 1108 | 1180.0 | 1456.3 | 1614.3 | 423.8 | 1797.7 | 6035.7 | 6472.0 | 5292.1 | 82 | 0.[illegible] |
| -3-17 | 1108 | 2498.9 | 2201.6 | 2221.3 | 563.1 | 2382.3 | 7346.1 | 9867.2 | 7368.3 | 75 | 0.[illegible] |
| -3-19 | 1138 | 674.1 | 626.3 | 615.20 | 179.3 | 778.5 | 2204.0 | 2873.3 | 2199.30 | 77 | 0.[illegible] |

pm values expressed as volumes of gas per million volumes of core material.

TABLE 37

$C_1$-$C_7$ Hydrocarbon Analyses of Shale Core

| Mound Sample Number | Well Interval (Feet) | 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-3-1 | 940 | 1325.3 | 3.6 | 22.6 | 18.6 | 95.2 | 370.5 | 1465.4 | 140.1 | 9.6 | 0.20 |
| I-3-2 | 940 | 1475.7 | 4.4 | 12.0 | 53.7 | 394.4 | 83.8 | 1940.4 | 464.7 | 24.0 | 0.14 |
| I-3-4 | 970 | 1347.3 | 5.5 | 33.8 | 24.6 | 117.1 | 595.0 | 1528.5 | 181.2 | 11.9 | 0.21 |
| I-3-6 | 995 | 1066.4 | 91.9 | 460.8 | 299.0 | 1202.8 | 2271.6 | 3121.2 | 2054.7 | 65.8 | 0.25 |
| I-3-7 | 995 | 1003.6 | 18.7 | 99.2 | 85.9 | 397.0 | 1414.7 | 1604.5 | 600.9 | 37.5 | 0.22 |
| I-3-9 | 1018 | 1336.5 | 86.3 | 309.9 | 280.4 | 1221.7 | 3049.9 | 3235.1 | 1898.6 | 58.7 | 0.23 |
| I-3-11 | 1048 | 1483.2 | 409.9 | 1924.0 | 990.4 | 3958.4 | 6009.7 | 8766.1 | 7282.8 | 83.1 | 0.25 |
| I-3-12 | 1048 | 1747.3 | 1140.7 | 3814.4 | 1774.2 | 6621.3 | 8588.8 | 15098.1 | 13350.8 | 88.4 | 0.27 |
| I-3-14 | 1078 | 1386.8 | 1661.6 | 6292.8 | 2390.1 | 8432.6 | 7638.7 | 20164.0 | 18777.2 | 93.1 | 0.26 |
| I-3-16 | 1108 | 1523.6 | 1430.3 | 4487.9 | 1575.5 | 6350.7 | 7688.0 | 15368.3 | 13844.6 | 90.1 | 0.25 |
| I-3-17 | 1108 | 1394.9 | 1662.5 | 4649.8 | 1595.8 | 6475.7 | 8354.9 | 15778.8 | 14383.9 | 91.2 | 0.25 |
| I-3-19 | 1138 | 1245.9 | 195.3 | 739.5 | 346.6 | 1702.7 | 6142.0 | 4230.2 | 2984.2 | 70.5 | 0.26 |

ppm values expressed as volumes of gas per million volumes of core material.

TABLE 38

$C_1$-$C_7$ Hydrocarbon Analyses of Head Space and Shale Core

| Mound Sample Number | Well Interval (Feet) | 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-3-1 | 940 | 2790.6 | 27.9 | 98.3 | 54.3 | 259.7 | 917.3 | 3230.9 | 440.3 | 13.6 | 0.21 |
| I-3-2 | 940 | 3609.7 | 54.4 | 157.9 | 119.4 | 656.0 | 687.2 | 4597.6 | 987.9 | 21.5 | 0.18 |
| I-3-4 | 970 | 3101.9 | 156.8 | 372.9 | 157.8 | 594.1 | 1424.4 | 4383.8 | 1281.8 | 29.2 | 0.27 |
| I-3-6 | 995 | 1601.7 | 235.5 | 733.8 | 401.7 | 1571.1 | 2960.2 | 4544.0 | 2942.2 | 64.8 | 0.26 |
| I-3-7 | 995 | 3807.9 | 497.5 | 815.2 | 343.6 | 1330.5 | 3275.1 | 6794.9 | 2986.9 | 44.0 | 0.26 |
| I-3-9 | 1018 | 4246.9 | 1079.9 | 2057.5 | 969.6 | 3752.6 | 6658.4 | 12106.7 | 7859.8 | 64.9 | 0.26 |
| I-3-11 | 1048 | 2038.1 | 919.6 | 2637.3 | 1231.5 | 4883.8 | 7568.1 | 11710.5 | 9672.3 | 82.6 | 0.25 |
| I-3-12 | 1048 | 5233.0 | 2453.3 | 5479.3 | 2279.1 | 8527.4 | 11211.2 | 23972.3 | 18739.2 | 78.2 | 0.27 |
| I-3-14 | 1078 | 9151.0 | 8218.0 | 13834.0 | 4363.3 | 15658.4 | 13886.4 | 51224.8 | 42073.8 | 82.1 | 0.28 |
| I-3-16 | 1108 | 2703.6 | 2886.6 | 6102.2 | 1999.3 | 8148.4 | 13723.7 | 21840.3 | 19136.7 | 87.6 | 0.25 |
| I-3-17 | 1108 | 3893.8 | 3864.1 | 6871.1 | 2158.9 | 8858.0 | 15701.0 | 25646.0 | 21752.2 | 84.8 | 0.24 |
| I-3-19 | 1138 | 1920.0 | 821.6 | 1354.7 | 525.9 | 2481.2 | 8346.0 | 7103.5 | 5183.5 | 73.0 | 0.21 |

ppm values expressed as volumes of gas per million volumes of core material.

TABLE 39

Oct.-Dec. 1977

Mound Facility

Detailed $C_4$-$C_7$ Hydrocarbon Analyses

| Mound Sample No. | P-1-1 | P-1-2 | P-1-3 |
|---|---|---|---|
| Isobutane | 0.5 | 28.3 | 18.4 |
| n-Butane | 2.8 | 3.0 | 1.8 |
| Isopentane | 2.7 | 10.7 | 16.5 |
| n-Pentane | 8.5 | 0.3 | 2.2 |
| 2, 2-Dimethylbutane | 0.2 | 0.2 | 0.3 |
| Cyclopentane | 1.0 | 7.1 | 7.2 |
| 2, 3-Dimethylbutane | 0.5 | 1.1 | 1.3 |
| 2-Methylpentane | 5.8 | 2.0 | 2.7 |
| 3-Methylpentane | 3.6 | 2.5 | 3.7 |
| n-Hexane | 13.2 | 0.5 | 0.6 |
| Methylcyclopentane | 4.3 | 16.0 | 16.9 |
| 2, 2-Dimethylpentane | 0.6 | 0.3 | 0.2 |
| Benzene | 1.2 | 0.5 | 0.5 |
| 2, 4-Dimethylpentane | 0.0 | 0.0 | 0.0 |
| 2, 2, 3-Trimethylbutane | 0.2 | 0.1 | 0.1 |
| Cyclohexane | 1.4 | 7.0 | 6.9 |
| 3, 3-Dimethylpentane | 0.3 | 0.2 | 0.2 |
| 1, 1-Dimethylcyclopentane | 0.8 | 0.8 | 1.1 |
| 2-Methylhexane | 4.6 | 0.0 | 0.1 |
| 2, 3-Dimethylpentane | 2.4 | 1.1 | 1.4 |
| 1-cis-3-Dimethylcyclopentane | 2.3 | 2.8 | 2.7 |
| 3-Methylhexane | 7.3 | 0.0 | 0.4 |
| 1 trans-3-Dimethylcyclopentane | 2.4 | 2.2 | 2.2 |
| 1 trans-2-Dimethylcyclopentane | 4.7 | 4.8 | 4.8 |
| 3-Ethylpentane | 1.9 | 0.6 | 0.6 |
| n-Heptane | 15.3 | 0.0 | 0.2 |
| 1, cis-2-Dimethylcyclopentane | 1.5 | 1.0 | 1.2 |
| Methylcyclohexane | 5.3 | 6.0 | 5.5 |
| Toluene | 4.8 | 0.8 | 0.2 |
| $C_4$-$C_7$ Hydrocarbon Content (ppm*) | 536.7 | 63510.9 | 14663.9 |
| 2-methylpentane/3-methylpentane | 1.61 | 0.80 | 0.73 |
| isopentane/n-pentane | 0.32 | 9.99 | 7.50 |
| cyclohexane/methylcyclopentane | 0.33 | 0.44 | 0.41 |
| methylcyclopentane/methylcyclohexane | 0.81 | 2.67 | 3.07 |

*ppm values are expressed as volumes of gas per million volumes of cuttings.

TABLE 40

Detailed $C_4$-$C_7$ Hydrocarbon Analyses

| Mound Sample No. | VA-1-2 | VA-1-4 | VA-1-6 | VA-1-11 | VA-1-13 | VA-1-15 | VA-1-17 |
|---|---|---|---|---|---|---|---|
| Isobutane | 33.3 | 26.5 | 13.7 | 12.9 | 19.7 | 34.8 | 32.6 |
| n-Butane | 40.1 | 32.9 | 30.2 | 33.9 | 29.7 | 36.8 | 35.8 |
| Isopentane | 9.1 | 15.9 | 10.4 | 9.3 | 14.9 | 14.6 | 15.1 |
| n-Pentane | 4.5 | 9.6 | 9.0 | 8.6 | 8.3 | 5.2 | 6.4 |
| 2, 2-Dimethylbutane | 0.5 | 0.8 | 0.9 | 1.8 | 1.8 | 1.1 | 1.2 |
| Cyclopentane | 0.6 | 0.4 | 0.9 | 0.8 | 0.7 | 0.3 | 0.2 |
| 2, 3-Dimethylbutane | 0.3 | 0.6 | 1.1 | 1.2 | 1.2 | 0.5 | 0.6 |
| 2-Methylpentane | 0.9 | 2.1 | 2.9 | 2.8 | 3.6 | 1.3 | 1.7 |
| 3-Methylpentane | 0.7 | 1.6 | 2.5 | 2.2 | 2.9 | 0.9 | 1.2 |
| n-Hexane | 0.7 | 1.8 | 3.3 | 2.6 | 2.7 | 0.6 | 1.0 |
| Methylcyclopentane | 1.5 | 1.1 | 1.5 | 1.6 | 1.5 | 0.4 | 0.4 |
| 2, 2-Dimethylpentane | 0.2 | 0.1 | 0.9 | 0.9 | 0.6 | 0.1 | 0.2 |
| Benzene | 0.5 | 0.5 | 0.9 | 1.0 | 1.1 | 0.5 | 0.3 |
| 2, 4-Dimethylpentane | 0.0 | 0.0 | 0.8 | 0.7 | 0.0 | 0.0 | 0.1 |
| 2, 2, 3-Trimethylbutane | 0.2 | 0.1 | 0.2 | 0.5 | 0.7 | 0.1 | 0.1 |
| Cyclohexane | 1.2 | 1.4 | 2.4 | 2.0 | 1.9 | 0.5 | 0.7 |
| 3, 3-Dimethylpentane | 0.2 | 0.1 | 1.0 | 1.2 | 0.3 | 0.1 | 0.1 |
| 1, 1-Dimethylcyclopentane | 0.3 | 0.2 | 0.8 | 0.4 | 0.1 | 0.1 | 0.1 |
| 2-Methylhexane | 0.2 | 0.3 | 1.3 | 1.2 | 1.0 | 0.2 | 0.2 |
| 2, 3-Dimethylpentane | 0.3 | 0.2 | 1.1 | 1.0 | 0.8 | 0.1 | 0.1 |
| 1-cis-3-Dimethylcyclopentane | 0.4 | 0.2 | 0.8 | 0.7 | 0.4 | 0.0 | 0.1 |
| 3-Methylhexane | 0.2 | 0.4 | 1.6 | 1.2 | 1.2 | 0.2 | 0.2 |
| 1 trans-3-Dimethylcyclopentane | 0.4 | 0.2 | 0.8 | 0.6 | 0.3 | 0.1 | 0.0 |
| 1 trans-2-Dimethylcyclopentane | 0.7 | 0.3 | 1.0 | 0.7 | 0.3 | 0.1 | 0.1 |
| 3-Ethylpentane | 0.2 | 0.1 | 1.1 | 1.1 | 0.4 | 0.1 | 0.1 |
| n-Heptane | 0.3 | 0.4 | 1.7 | 1.6 | 1.0 | 0.1 | 0.2 |
| 1, cis-2-Dimethylcyclopentane | 0.4 | 0.1 | 0.6 | 0.5 | 0.3 | 0.0 | 0.0 |
| Methylcyclohexane | 1.4 | 1.4 | 3.6 | 2.9 | 2.0 | 0.4 | 0.5 |
| Toluene | 0.8 | 0.6 | 3.1 | 4.2 | 0.7 | 0.9 | 0.4 |
| $C_4$-$C_7$ Hydrocarbon Content (ppm*) | 69681.3 | 36475.4 | 3149.2 | 2621.8 | 2287.4 | 17176.8 | 15715.4 |
| 2-methylpentane/3-methylpentane | 1.29 | 1.31 | 1.16 | 1.27 | 1.24 | 1.44 | 1.42 |
| isopentane/n-pentane | 2.02 | 1.66 | 1.16 | 1.08 | 1.80 | 2.81 | 2.36 |
| cyclohexane/methylcyclopentane | 0.80 | 1.27 | 1.60 | 1.25 | 1.27 | 1.25 | 1.75 |
| methylcyclopentane/methylcyclohexane | 1.07 | 0.79 | 0.42 | 0.55 | 0.75 | 1.00 | 0.80 |

*ppm values are expressed as volumes of gas per million volumes of cuttings.

TABLE 40 (Cont'd.)

Detailed $C_4$-$C_7$ Hydrocarbon Analyses

| Mound Sample No. | VA-1-18 | VA-1-21 | VA-1-22 | VA-1-25 | VA-1-26 | VA-1-27 | VA-1-29 | VA-1-32 |
|---|---|---|---|---|---|---|---|---|
| Isobutane | 31.4 | 35.7 | 33.6 | 24.9 | 40.3 | 17.5 | 33.0 | 37.2 |
| n-Butane | 38.7 | 39.7 | 40.4 | 37.9 | 42.3 | 29.1 | 39.1 | 41.4 |
| Isopentane | 12.5 | 10.5 | 11.8 | 13.4 | 6.7 | 12.0 | 12.1 | 8.8 |
| n-Pentane | 5.6 | 4.9 | 6.1 | 7.9 | 3.3 | 8.8 | 6.7 | 4.5 |
| 2, 2-Dimethylbutane | 1.0 | 0.6 | 0.5 | 0.8 | 0.5 | 1.1 | 0.5 | 0.5 |
| Cyclopentane | 0.3 | 0.3 | 0.3 | 0.4 | 0.3 | 0.6 | 0.3 | 0.3 |
| 2, 3-Dimethylbutane | 0.5 | 0.3 | 0.3 | 0.6 | 0.2 | 1.1 | 0.4 | 0.3 |
| 2-Methylpentane | 1.3 | 0.9 | 0.9 | 2.0 | 0.6 | 3.4 | 1.1 | 0.9 |
| 3-Methylpentane | 1.1 | 0.8 | 0.7 | 1.5 | 0.5 | 2.6 | 0.8 | 0.6 |
| n-Hexane | 0.8 | 0.7 | 0.7 | 1.6 | 0.5 | 3.2 | 0.8 | 0.6 |
| Methylcyclopentane | 0.5 | 0.4 | 0.4 | 0.6 | 0.3 | 1.0 | 0.4 | 0.3 |
| 2, 2-Dimethylpentane | 0.2 | 0.1 | 0.1 | 0.2 | 0.1 | 0.6 | 0.1 | 0.2 |
| Benzene | 0.5 | 0.6 | 0.6 | 1.3 | 0.6 | 0.6 | 0.6 | 0.6 |
| 2, 4-Dimethylpentane | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.8 | 0.0 | 0.0 |
| 2, 2, 3-Trimethylbutane | 0.2 | 0.2 | 0.1 | 0.2 | 0.2 | 0.2 | 0.1 | 0.1 |
| Cyclohexane | 0.8 | 0.7 | 0.8 | 1.1 | 0.6 | 1.9 | 0.9 | 0.7 |
| 3, 3-Dimethylpentane | 0.2 | 0.3 | 0.1 | 0.2 | 0.2 | 0.7 | 0.1 | 0.1 |
| 1, 1-Dimethylcyclopentane | 0.2 | 0.1 | 0.1 | 0.2 | 0.1 | 0.4 | 0.1 | 0.1 |
| 2-Methylhexane | 0.3 | 0.2 | 0.2 | 0.4 | 0.2 | 1.3 | 0.2 | 0.2 |
| 2, 3-Dimethylpentane | 0.2 | 0.2 | 0.1 | 0.3 | 0.2 | 1.0 | 0.1 | 0.1 |
| 1-cis-3-Dimethylcyclopentane | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.6 | 0.1 | 0.1 |
| 3-Methylhexane | 0.3 | 0.2 | 0.2 | 0.5 | 0.2 | 1.6 | 0.2 | 0.2 |
| 1 trans-3-Dimethylcyclopentane | 0.1 | 0.2 | 0.0 | 0.1 | 0.1 | 0.6 | 0.1 | 0.1 |
| 1 trans-2-Dimethylcyclopentane | 0.2 | 0.2 | 0.1 | 0.2 | 0.1 | 0.9 | 0.1 | 0.2 |
| 3-Ethylpentane | 0.2 | 0.1 | 0.0 | 0.2 | 0.1 | 0.7 | 0.1 | 0.1 |
| n-Heptane | 0.4 | 0.3 | 0.2 | 0.5 | 0.2 | 1.7 | 0.2 | 0.2 |
| 1, cis-2-Dimethylcyclopentane | 0.2 | 0.1 | 0.1 | 0.2 | 0.1 | 0.6 | 0.1 | 0.1 |
| Methylcyclohexane | 0.9 | 0.6 | 0.6 | 1.2 | 0.5 | 3.3 | 0.7 | 0.6 |
| Toluene | 1.2 | 0.9 | 0.8 | 1.6 | 0.9 | 2.4 | 0.8 | 0.8 |
| $C_4$-$C_7$ Hydrocarbon Content (ppm*) | 6763.6 | 14060.5 | 15517.1 | 11295.7 | 5769.3 | 1466.4 | 8169.4 | 6844.8 |
| 2-methylpentane/3-methylpentane | 1.18 | 1.13 | 1.29 | 1.33 | 1.20 | 1.31 | 1.38 | 1.50 |
| isopentane/n-pentane | 2.23 | 2.14 | 1.93 | 1.70 | 2.03 | 1.36 | 1.81 | 1.96 |
| cyclohexane/methylcyclopentane | 1.60 | 1.75 | 2.00 | 1.83 | 2.00 | 1.90 | 2.25 | 2.33 |
| methylcyclopentane/methylcyclohexane | 0.56 | 0.67 | 0.67 | 0.50 | 0.60 | 0.30 | 0.57 | 0.50 |

*ppm values are expressed as volumes of gas per million volumes of cuttings.

TABLE 41

Detailed $C_4$-$C_7$ Hydrocarbon Analyses

| Mound Sample No. | I-2-2 | I-2-3 | I-2-6 | I-2-7 | I-2-10 | I-2-12 | I-2-14 | I-2-16 | I-2-18 | I-2-20 |
|---|---|---|---|---|---|---|---|---|---|---|
| Isobutane | 1.3 | 0.9 | 0.8 | 3.0 | 6.6 | 6.6 | 0.5 | 4.0 | 3.9 | 0.9 |
| n-Butane | 5.4 | 4.7 | 2.5 | 9.8 | 11.0 | 11.6 | 0.9 | 12.8 | 14.6 | 3.2 |
| Isopentane | 12.2 | 11.9 | 12.3 | 15.9 | 12.9 | 12.9 | 43.5 | 16.8 | 18.7 | 8.5 |
| n-Pentane | 15.7 | 13.9 | 9.7 | 15.6 | 12.9 | 13.0 | 14.1 | 15.1 | 17.9 | 10.6 |
| 2, 2-Dimethylbutane | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.1 | 0.2 | 0.1 | 0.1 | 0.1 |
| Cyclopentane | 0.8 | 1.1 | 0.9 | 1.3 | 2.2 | 1.9 | 0.5 | 1.4 | 1.3 | 0.8 |
| 2, 3-Dimethylbutane | 0.7 | 0.6 | 0.7 | 0.6 | 0.6 | 0.5 | 0.5 | 0.5 | 0.6 | 0.6 |
| 2-Methylpentane | 7.8 | 7.2 | 8.6 | 3.0 | 6.1 | 5.6 | 3.2 | 5.7 | 6.1 | 7.4 |
| 3-Methylpentane | 5.6 | 5.3 | 6.2 | 4.9 | 4.5 | 4.1 | 2.6 | 4.3 | 4.5 | 5.7 |
| n-Hexane | 8.5 | 7.4 | 7.2 | 6.0 | 5.5 | 5.3 | 2.1 | 5.1 | 5.6 | 8.8 |
| Methylcyclopentane | 9.4 | 11.9 | 12.0 | 11.2 | 13.3 | 11.7 | 4.3 | 10.7 | 9.3 | 11.2 |
| 2, 2-Dimethylpentane | 0.2 | 0.1 | 0.1 | 0.2 | 0.1 | 0.1 | 0.3 | 0.1 | 0.1 | 0.1 |
| Benzene | 0.6 | 0.5 | 0.6 | 0.4 | 0.2 | 0.2 | 0.6 | 0.3 | 0.2 | 0.4 |
| 2, 4-Dimethylpentane | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| 2, 2, 3-Trimethylbutane | 0.1 | 0.1 | 0.1 | 0.2 | 0.0 | 0.1 | 0.3 | 0.1 | 0.1 | 0.1 |
| Cyclohexane | 2.2 | 3.0 | 3.0 | 2.8 | 3.4 | 3.1 | 1.8 | 2.9 | 2.4 | 3.1 |
| 3, 3-Dimethylpentane | 0.1 | 0.1 | 0.1 | 0.1 | 0.0 | 0.0 | 0.2 | 0.1 | 0.1 | 0.1 |
| 1, 1-Dimethylcyclopentane | 0.5 | 0.5 | 0.5 | 0.4 | 0.3 | 0.4 | 0.9 | 0.4 | 0.3 | 0.5 |
| 2-Methylhexane | 2.0 | 1.8 | 2.2 | 1.2 | 0.8 | 0.9 | 1.0 | 0.8 | 0.7 | 1.9 |
| 2, 3-Dimethylpentane | 0.9 | 0.8 | 1.0 | 0.7 | 0.5 | 0.6 | 1.1 | 0.6 | 0.5 | 1.1 |
| 1-cis-3-Dimethylcyclopentane | 3.1 | 3.6 | 3.9 | 2.7 | 2.6 | 2.8 | 2.1 | 2.5 | 1.8 | 4.[illegible] |
| 3-Methylhexane | 2.9 | 2.9 | 3.6 | 2.4 | 1.7 | 1.9 | 1.7 | 1.7 | 1.3 | 3.[illegible] |
| 1 trans-3-Dimethylcyclopentane | 2.7 | 3.2 | 3.4 | 2.5 | 2.3 | 2.4 | 1.9 | 2.2 | 1.6 | 3.[illegible] |
| 1 trans-2-Dimethylcyclopentane | 5.2 | 6.2 | 6.9 | 5.3 | 4.8 | 5.1 | 4.2 | 4.6 | 3.3 | 8.[illegible] |
| 3-Ethylpentane | 0.7 | 0.7 | 0.6 | 0.8 | 0.4 | 0.5 | 1.0 | 0.3 | 0.4 | 0.[illegible] |
| n-Heptane | 3.7 | 3.2 | 3.9 | 2.5 | 1.7 | 2.1 | 2.1 | 1.7 | 1.3 | 4.[illegible] |
| 1, cis-3-Dimethylcyclopentane | 1.3 | 1.3 | 1.3 | 1.0 | 0.9 | 1.0 | 1.7 | 0.7 | 0.6 | 1.[illegible] |
| Methylcyclohexane | 5.0 | 5.9 | 6.1 | 4.3 | 3.7 | 3.9 | 3.8 | 3.4 | 2.4 | 6.[illegible] |
| Toluene | 1.5 | 1.1 | 1.6 | 1.2 | 1.2 | 1.5 | 3.1 | 1.0 | 0.5 | 1.[illegible] |
| $C_4$-$C_7$ Hydrocarbon Content (ppm) | 541.0 | 990.0 | 967.4 | 4853.7 | 15797.0 | 33889.1 | 701.0 | 17090.3 | 5595.9 | 1291.6 |
| 2-methylpentane/3-methylpentane | 1.39 | 1.36 | 1.39 | 0.61 | 1.36 | 1.37 | 1.23 | 1.33 | 1.36 | 1.30 |
| isopentane/n-pentane | 0.78 | 0.86 | 1.27 | 1.02 | 1.00 | 0.99 | 3.09 | 1.11 | 1.04 | 0.80 |
| cyclohexane/methylcyclopentane | 0.23 | 0.25 | 0.25 | 0.25 | 0.26 | 0.26 | 0.42 | 0.27 | 0.26 | 0.28 |
| methylcyclopentane/methylcyclohexane | 1.88 | 2.02 | 1.97 | 2.60 | 3.59 | 3.00 | 1.13 | 3.15 | 3.88 | 1.65 |

TABLE 42

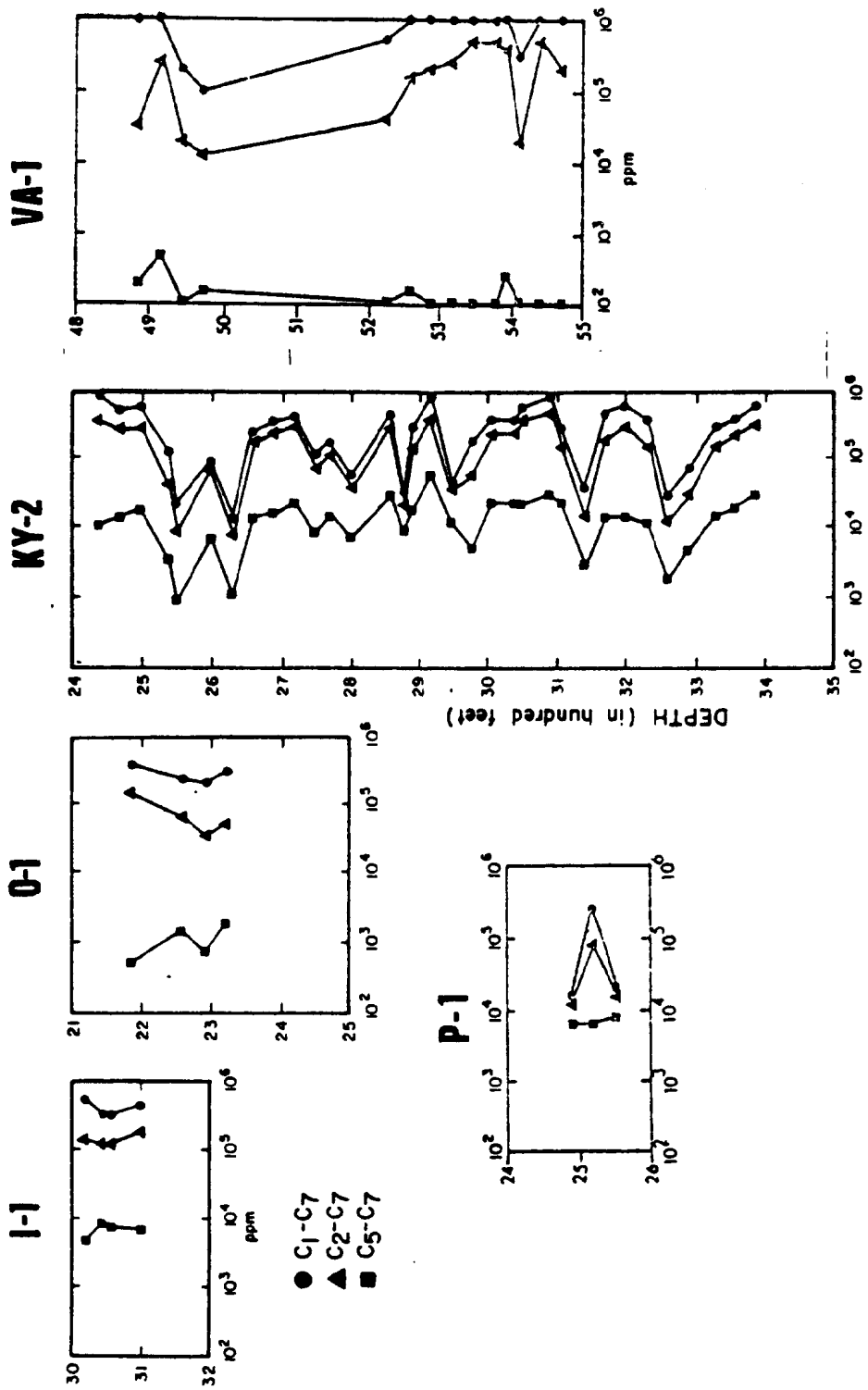
Detailed $C_4$-$C_7$ Hydrocarbon Analyses

| Mound Sample No. | I-3-1 | I-3-2 | I-3-4 | I-3-6 | I-3-7 | I-3-9 |
|---|---|---|---|---|---|---|
| Isobutane | 2.1 | 0.9 | 1.4 | 5.1 | 2.8 | 4.1 |
| n-Butane | 9.5 | 4.4 | 6.3 | 17.3 | 11.2 | 11.3 |
| Isopentane | 10.5 | 8.3 | 9.8 | 15.9 | 13.5 | 13.1 |
| n-Pentane | 13.5 | 10.8 | 12.8 | 18.4 | 15.7 | 15.2 |
| 2, 2-Dimethylbutane | 0.2 | 0.2 | 0.1 | 0.1 | 0.1 | 0.1 |
| Cyclopentane | 0.9 | 0.9 | 0.9 | 1.2 | 1.2 | 1.5 |
| 2, 3-Dimethylbutane | 0.8 | 0.7 | 0.7 | 0.6 | 0.7 | 0.6 |
| 2-Methylpentane | 7.5 | 7.7 | 7.6 | 5.7 | 7.2 | 7.3 |
| 3-Methylpentane | 5.5 | 5.7 | 5.5 | 4.0 | 5.1 | 5.0 |
| n-Hexane | 8.5 | 8.8 | 8.9 | 6.1 | 8.0 | 8.2 |
| Methylcyclopentane | 7.4 | 9.2 | 9.4 | 8.2 | 10.5 | 10.5 |
| 2, 2-Dimethylpentane | 0.4 | 0.2 | 0.2 | 0.1 | 0.1 | 0.1 |
| Benzene | 0.0 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 |
| 2, 4-Dimethylpentane | 1.0 | 0.8 | 0.6 | 0.3 | 0.3 | 0.3 |
| 2, 2, 3-Trimethylbutane | 0.2 | 0.1 | 0.2 | 0.1 | 0.0 | 0.1 |
| Cyclohexane | 1.9 | 2.3 | 2.3 | 1.7 | 2.2 | 2.2 |
| 3, 3-Dimethylpentane | 0.2 | 0.2 | 0.2 | 0.1 | 0.0 | 0.0 |
| 1, 1-Dimethylcyclopentane | 0.7 | 0.7 | 0.6 | 0.3 | 0.4 | 0.3 |
| 2-Methylhexane | 2.5 | 4.5 | 2.5 | 0.9 | 1.4 | 1.3 |
| 2, 3-Dimethylpentane | 1.3 | 0.0 | 1.1 | 0.5 | 0.6 | 0.6 |
| 1-cis-3-Dimethylcyclopentane | 2.8 | 3.9 | 3.4 | 1.7 | 2.5 | 2.4 |
| 3-Methylhexane | 3.2 | 4.1 | 3.4 | 1.3 | 2.0 | 2.0 |
| 1 trans-3-Dimethylcyclopentane | 2.4 | 3.3 | 3.0 | 1.5 | 2.2 | 2.1 |
| 1 trans-2-Dimethylcyclopentane | 4.7 | 6.7 | 6.0 | 3.0 | 4.4 | 4.3 |
| 3-Ethylpentane | 0.7 | 0.7 | 0.7 | 0.3 | 0.3 | 0.4 |
| n-Heptane | 4.0 | 5.4 | 3.9 | 1.7 | 2.5 | 2.5 |
| 1, cis-2-Dimethylcyclopentane | 1.2 | 1.6 | 1.3 | 0.7 | 0.9 | 0.8 |
| Methylcyclohexane | 4.9 | 7.5 | 6.2 | 2.9 | 4.0 | 3.5 |
| Toluene | 1.7 | 0.3 | 1.1 | 0.1 | 0.1 | 0.1 |
| $C_4$-$C_7$ Hydrocarbon Content (ppm) | 666.0 | 633.3 | 427.8 | 1832.5 | 978.7 | 2031.0 |
| 2-methylpentane/3-methylpentane | 1.36 | 1.35 | 1.38 | 1.43 | 1.41 | 1.46 |
| isopentane/n-pentane | 0.78 | 0.77 | 0.77 | 0.86 | 0.86 | 0.86 |
| cyclohexane/methylcyclopentane | 0.26 | 0.25 | 0.24 | 0.21 | 0.21 | 0.21 |
| methylcyclopentane/methylcyclohexane | 1.51 | 1.23 | 1.52 | 2.83 | 2.63 | 3.00 |

TABLE 42 (Cont'd.)

Detailed $C_4$-$C_7$ Hydrocarbon Analyses

| Mound Sample No. | I-3-11 | I-3-12 | I-3-14 | I-3-16 | I-3-17 | I-3-19 |
|---|---|---|---|---|---|---|
| Isobutane | 5.9 | 5.5 | 11.5 | 6.9 | 6.4 | 4.0 |
| n-Butane | 10.7 | 10.0 | 20.4 | 14.6 | 17.0 | 17.5 |
| Isopentane | 11.2 | 10.5 | 15.9 | 13.3 | 13.5 | 12.4 |
| n-Pentane | 13.0 | 12.3 | 18.0 | 15.6 | 16.6 | 16.3 |
| 2, 2-Dimethylbutane | 0.1 | 0.1 | 0.1 | 0.5 | 0.2 | 0.2 |
| Cyclopentane | 1.8 | 1.9 | 2.1 | 1.8 | 1.4 | 0.9 |
| 2, 3-Dimethylbutane | 0.6 | 0.6 | 0.4 | 0.5 | 0.5 | 0.5 |
| 2-Methylpentane | 6.7 | 6.4 | 4.2 | 6.5 | 5.7 | 6.7 |
| 3-Methylpentane | 4.5 | 4.4 | 2.7 | 4.2 | 3.7 | 4.2 |
| n-Hexane | 7.7 | 7.5 | 4.4 | 6.8 | 6.3 | 7.0 |
| Methylcyclopentane | 10.4 | 11.0 | 7.5 | 8.8 | 7.5 | 6.2 |
| 2, 2-Dimethylpentane | 0.1 | 0.1 | 0.0 | 0.1 | 0.1 | 0.2 |
| Benzene | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| 2, 4-Dimethylpentane | 0.3 | 0.3 | 0.1 | 0.3 | 0.3 | 0.4 |
| 2, 2, 3-Trimethylbutane | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.2 |
| Cyclohexane | 2.3 | 2.7 | 1.7 | 2.1 | 1.9 | 1.6 |
| 3, 3-Dimethylpentane | 0.0 | 0.1 | 0.0 | 0.1 | 0.1 | 0.2 |
| 1, 1-Dimethylcyclopentane | 0.3 | 0.4 | 0.2 | 0.3 | 0.3 | 0.5 |
| 2-Methylhexane | 2.0 | 1.5 | 0.6 | 1.0 | 1.1 | 1.3 |
| 2, 3-Dimethylpentane | 0.0 | 0.6 | 0.3 | 0.6 | 0.7 | 0.9 |
| 1-cis-3-Dimethylcyclopentane | 2.9 | 3.1 | 1.4 | 2.0 | 2.1 | 2.1 |
| 3-Methylhexane | 2.5 | 2.6 | 1.0 | 2.0 | 2.2 | 2.2 |
| 1 trans-3-Dimethylcyclopentane | 2.5 | 2.8 | 1.2 | 1.8 | 1.8 | 1.8 |
| 1 trans-2-Dimethylcyclopentane | 5.5 | 5.9 | 2.8 | 4.4 | 4.4 | 4.1 |
| 3-Ethylpentane | 0.5 | 0.5 | 0.3 | 0.6 | 0.5 | 0.7 |
| n-Heptane | 3.1 | 3.1 | 0.6 | 2.1 | 2.5 | 3.4 |
| 1, cis-2-Dimethylcyclopentane | 1.0 | 1.3 | 0.5 | 0.7 | 0.7 | 1.1 |
| Methylcyclohexane | 3.8 | 4.5 | 1.6 | 2.3 | 2.4 | 2.9 |
| Toluene | 0.6 | 0.2 | 0.4 | 0.0 | 0.1 | 0.8 |
| $C_4$-$C_7$ Hydrocarbon Content (ppm) | 19823.7 | 29316.5 | 19347.0 | 109114.6 | 33317.4 | 18635.5 |
| 2-methylpentane/3-methylpentane | 1.49 | 1.45 | 1.56 | 1.55 | 1.54 | 1.60 |
| isopentane/n-pentane | 0.86 | 0.85 | 0.88 | 0.85 | 0.81 | 0.76 |
| cyclohexane/methylcyclopentane | 0.22 | 0.25 | 0.23 | 0.24 | 0.25 | 0.26 |
| methylcyclopentane/methylcyclohexane | 2.74 | 2.44 | 4.69 | 3.83 | 3.13 | 2.14 |




$C_1$-$C_7$ HYDROCARBON DATA FOR CORE SAMPLES

FIGURE 5

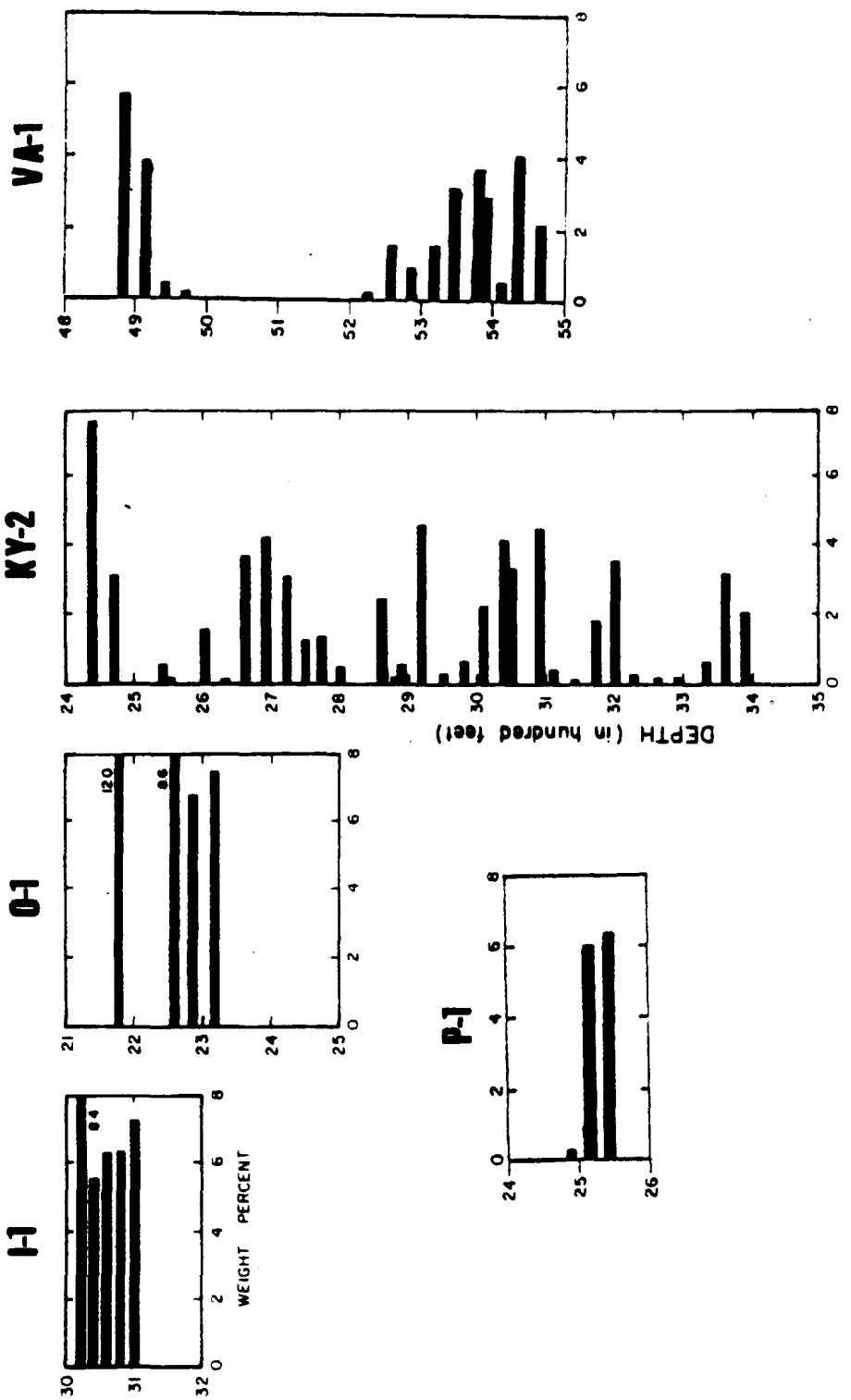
to the atmosphere for various periods of time prior to being placed in the sample containers. This exposure time allows a significant amount of hydrocarbon gases to diffuse from the core. Although for most samples, the maximum exposure time is hours, the P-1 samples were exposed to air for several weeks prior to canning for geochemical analyses.

The organic carbon content of fine-grained argillaceous sediments such as the Devonian shales is an indicator of organic richness. Organic matter comprised mostly of organic detritus is preserved by rapid burial. Alteration processes such as microbial degradation and thermal diagenesis transform the organic matter to the complex heterogeneous material called "kerogen." Kerogen is presumed to be the major precursor for oil and gas. As the sediments become thermally matured, oil and gas are generated. The amount of kerogen, expressed as the organic carbon content, shows the abundance of organic matter which may be altered to form hydrocarbons. The lower limit of organic carbon in shales from productive basins is 0.4 percent. The worldwide average of organic carbon in shales and siltstones is 1.14 percent.

The amount of organic carbon in Devonian shales varies from location to location (Figure6, Tables 43 through 46). In general the average organic carbon content of sediments from the I-1, O-1, P-1, KY-2, VA-1, I-3 and I-2 wells is 6.77, 8.75, 4.73, 2.04, 1.96, 1.35 and 0.88 percent, respectively. The organic matter appears to be more uniformily distributed in the I-1, O-1 and P-1 wells. The other wells have a significant variation of organic carbon along their profiles.

Tables 43 through 46 also list the lithologic descriptions for several of the wells. The P-1 well is described as being consistently 100% Argillite, while the VA-1 well is Shale interspersed with Argillite, the I-2 well is Shale with a consistent interval of claystone; and the I-3 well interval is consistently shale.

Core material was extracted with organic solvent to yield the $C_{15+}$ extract or "bitumen." The bitumen is composed of hydrocarbon and nonhydrocarbon material. The bitumen in recent sediments is impoverished in hydrocarbons and contains mostly nonhydrocarbon material. As the sediments become thermally matured, increasing amounts of hydrocarbons are formed. The average worldwide concentrations of hydrocarbons in shales was found to be 96 ppm.




ORGANIC CARBON DATA
FOR CORE SAMPLES

FIGURE 6

TABLE 43

Gross Lithological Description and Organic Carbon Analyses

| Mound Sample Number | Well Interval | Gross Lithological Description | GSA Color Code | Total Organic Carbon (% of Rock) |
|---|---|---|---|---|
| P-1-1 | | 100% Argillite, noncalcareous, nonsilty, fissile, indurated, hard, medium dark gray. | N4 | 0.25 |
| P-1-2 | | 100% Argillite, noncalcareous, nonsilty, fissile, indurated, hard, grayish black. | N2 | 6.01 |
| P-1-3 | | 100% Argillite, noncalcareous, nonsilty, fissile, indurated, hard, spore-pollen associated, dark gray. | N3 | 6.34; 6.31R |

TABLE 44

Gross Lithological Description and Organic Carbon Analyses

| Mound Sample Number | Well Interval | Gross Lithological Description | GSA Color Code | Total Organic Carbon (% of Rock) |
|---|---|---|---|---|
| VA-1-2 | | 100% Shale, carbonaceous, slightly silty, slightly micromicaceous, slightly pyritic, slaty clevage, indurated, hard, black. | N1 | 5.66 |
| VA-1-4 | | 100% Shale, carbonaceous, slightly silty, micromicaceous, slaty clevage, indurated, hard, black. | N1 | 3.85 |
| VA-1-6 | | 100% Shale, noncalcareous, nonsilty, very slightly pyritic, fissile, indurated, hard, medium dark gray. | N4 | 0.47; 0.44R |
| VA-1-11 | | 100% Shale, noncalcareous, nonsilty, fissile, indurated, hard, medium dark gray. | N4 | 0.18 |
| VA-1-13 | | 100% Shale, carbonaceous, slightly mottled, micromicaceous, fissile, indurated, hard, dark gray. | N3 | 0.18 |
| VA-1-15 | | 100% Argillite, carbonaceous, micromicaceous, very silty, fissile, highly indurated, hard, grayish black. | N2 | 1.51 |
| VA-1-17 | | 100% Argillite, carbonaceous, fissile, highly indurated, very hard, dark gray to grayish black. | N3 to N2 | 0.88 |

TABLE 44 (Cont'd.)

Gross Lithological Description and Organic Carbon Analyses

| Mound Sample Number | Well Interval | Gross Lithological Description | GSA Color Code | Total Organic Carbon (% of Rock) |
|---|---|---|---|---|
| VA-1-18 | | 100% Argillite, carbonaceous, fissile, highly indurated, very hard, grayish black. | N2 | 1.54; 1.54R |
| VA-1-21 | | 100% Shale, carbonaceous, moderately fissile, indurated, hard, grayish black. | N2 | 3.13 |
| VA-1-22 | | 100% Argillite, carbonaceous, fissile, highly indurated, very hard, grayish black. | N2 | 3.57 |
| VA-1-25 | | 100% Shale, carbonaceous, moderately fissile, indurated, hard, slicken-sides in part, grayish black. | N2 | 2.53 |
| VA-1-26 | | 100% Shale, carbonaceous, slicken-sides abundant, blocky, indurated, hard, grayish black. | N2 | 2.84 |
| VA-1-27 | | 100% Shale, noncalcareous, nonsilty, moderately fissile, very hard, medium dark gray. | N4 | 0.48; 0.48R |
| VA-1-29 | | 100% Argillite, carbonaceous, fissile, highly indurated, very hard, grayish black. | N2 | 3.97 |
| VA-1-32 | | 100% Argillite, carbonaceous, fissile, indurated, hard, grayish black. | N2 | 2.11 |

TABLE 45

Gross Lithological Description and Organic Carbon Analyses

| Mound Sample Number | Well Interval | Gross Lithological Description | GSA Color Code | Total Organic Carbon (% of Rock) |
|---|---|---|---|---|
| I-2-A-1 | | 90% Siltstone, slightly calcareous, slightly sandy, fine grain, subrounded, frosted to clear; moderately hard, no show, very light gray. | N8 | 0.55 |
| I-2-A-2 | | 10% Shale, noncalcareous, hard, blocky, medium light gray. | N6 | |
| I-2-1 | | 100% Claystone, calcareous, slightly silty, hard, blocky to fissile, medium bluish gray. Trace pyrite. | 5B 5/1 | 0.18;0.18 |
| I-2-4 | | 100% Claystone, calcareous, slightly silty, hard, blocky to fissile, medium bluish gray. Trace Pyrite. | 5B 5/1 | 0.37 |
| I-2-6 | | 100% Claystone, slightly calcareous, silty, hard, blocky to fissile, medium bluish gray. | 5B 5/1 | 0.48 |
| I-2-7 | | 100% Claystone, slightly calcareous, silty, hard, blocky to fissile, pyrite vein fillings, medium bluish gray to medium dark gray. | 5B 5/1 to N4 | 0.52 |
| I-2-10 | | 100% Claystone, slightly calcareous, silty, hard, blocky to fissile, pyrite vein fillings, medium bluish gray to medium dark gray. | 5B 5/1 to N4 | 2.24 |
| 1-2-12 | | 100% Shale, very slightly calcareous, silty, hard, blocky to fissile, indurated, grayish brown. | 5YR 3/2 | 3.29;3.22 |

TABLE 45 (Cont'd.)

Gross Lithological Description and Organic Carbon Analyses

| Mound Sample Number | Well Interval | Gross Lithological Description | GSA Color Code | Total Organic Carbon (% of Rock) |
|---|---|---|---|---|
| I-2-14 | | 100% Shale, noncalcareous, silty, moderately hard, blocky to fissile, light bluish gray. | 5B 7/1 | 0.21 |
| I-2-16 | | 100% Shale, noncalcareous, silty, moderately hard, fissile, indurated, pale brown. | 5YR 5/2 | 0.22 |
| I-2-18 | | 100% Shale, noncalcareous, silty, hard, blocky to fissile, indurated, brownish black. | 5YR 2/1 | 1.07 |
| I-2-20 | | 100% Shale, slightly calcareous, silty, hard, blocky to fissile, medium gray. | N5 | 0.14 |
| I-2-B | | 100% Sparite, finely crystalline, poor porosity, moderately soft, no show, yellowish gray.<br>Trace micrite.<br>Trace Straw. | 5Y 7/2 | 0.24;0.24 |

TABLE 46

Gross Lithological Description and Organic Carbon Analyses

| Mound Sample Number | Well Interval | Gross Lithological Description | GSA Color Code | Total Organic Carbon (% of Rock) |
|---|---|---|---|---|
| I-3-A | | All of sample ground for analysis. | | 0.38 |
| I-3-1 | | 100% Shale, noncalcareous, slightly silty, blocky, hard, medium light gray to greenish gray. | N6 to 5G 6/1 | 0.21 |
| I-3-2 | | 100% Shale, noncalcareous, slightly silty, blocky, hard, medium light gray to greenish gray. | N6 to 5G 6/1 | 0.20;0.29R |
| I-3-4 | | 100% Shale, noncalcareous, slightly silty, blocky, hard, medium light gray to greenish gray. | N6 to 5G 6/1 | 0.20 |
| I-3-6 | | 100% Shale, noncalcareous, slightly silty, blocky hard, medium light gray to greenish gray. | N6 to 5G 6/1 | 0.84 |
| I-3-7 | | 100% Shale, noncalcareous, slightly silty, blocky, hard, medium light gray to greenish gray. | N6 to 5G 6/1 | 0.69 |
| I-3-9 | | 100% Shale, noncalcareous, slightly silty, blocky, hard, medium gray to medium dark gray. | N5 to N4 | 0.75 |
| I-3-11 | | 100% Shale, noncalcareous, slightly silty, blocky, hard, medium gray to medium dark gray. | N5 to N4 | 1.60;1.68R |
| I-3-12 | | 100% Shale, noncalcareous, slightly silty, blocky, hard, medium gray to medium dark gray. | N5 to N4 | 1.32 |
| 1-3-14 | | 100% Shale, noncalcareous, silty, blocky, hard, brownish gray to medium dark gray. | 5YR 4/1 to N4 | 3.44 |

TABLE 46 (continued)

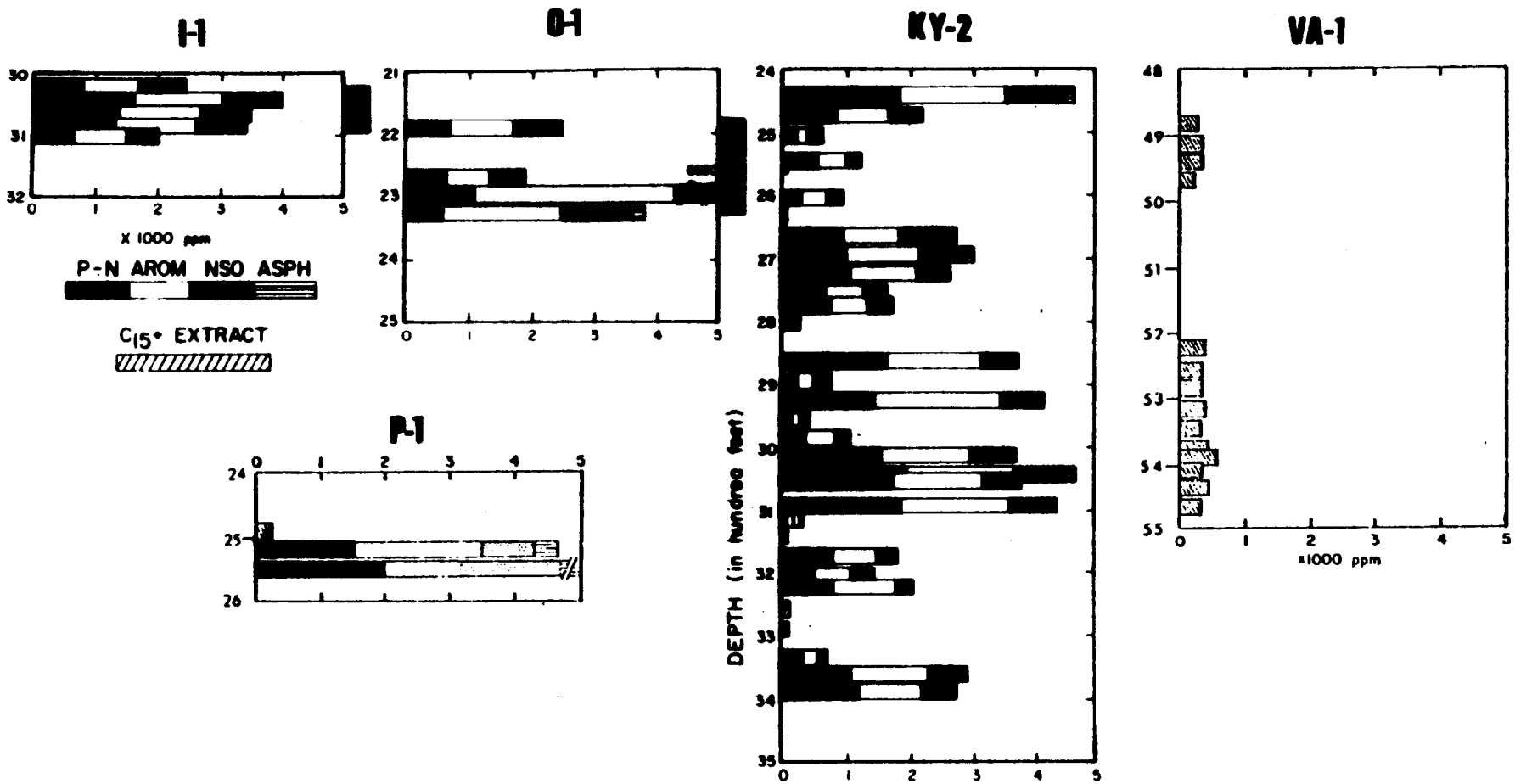
Gross Lithological Description and Organic Carbon Analyses

| Mound Sample Number | Well Interval | Gross Lithological Description | GSA Color Code | Total Organic Carbon (% of Rock) |
|---|---|---|---|---|
| I-3-16 | | 100% Shale, noncalcareous, silty, blocky, hard, brownish gray to medium dark gray. | 5YR 4/1 to N4 | 3.45 |
| I-3-17 | | 100% Shale, noncalcareous, silty, blocky, hard, brownish gray to medium dark gray. | 5YR 4/1 to N4 | 3.44 |
| I-3-19 | | 100% Shale, noncalcareous, silty, blocky, hard, brownish gray to medium dark gray. | 5YR 4/1 to N4 | 1.58;1.50R |
| I-3-B | | 100% Dolomite, sucrosic-microcrystalline, moderately hard, fair to good porosity, no show, white. | N9 | |

Shales from six of the wells were found to contain bitumen composed mostly of hydrocarbons. The distribution of hydrocarbons and nonhydrocarbon material extracted from the Devonian shales of five wells is shown in Figure 7. Tables 47 through 66 list the detailed results not previously reported. The hydrocarbon portion consists of paraffin-naphthene (P-N) and aromatic (AROM) fractions. The nonhydrocarbon material is seperated into asphaltene (ASPH) and nitrogen-sulfur-oxygen containing compounds (NSO's). In the I-1, O-1, P-1, I-2 and I-3 wells, the average hydrocarbon content was 2159 ppm, 2463 ppm, 3332 ppm, 503 ppm and 712 ppm, respectively. The average hydrocarbon content was 1469 ppm for the KY-2 shales and approximately zero fro the VA-1 shales. The hydrocarbon content appears to be related to the organic carbon content and the kerogen type. In general, shales characterized by herbaceous-amorphous kerogen, and a high organic carbon content contain abundant $C_{15+}$ hydrocarbons. Sediments with low organic carbon content and primary or secondary amounts of woody-coaly kerogen contain low amounts of $C_{15+}$ hydrocarbons. Also evident in these data and supported by other data is the fact that the western edge of the Illinois basin is not as rich as the central portion.

In order to assess the hydrocarbon generating capacity of potential source rocks, the thermal history and its diagentic effect on petroleum generation must be evaluated. Vitrinite reflectance ($R_o$) was used to measure the degree of thermal alteration of the Devonian shales. This technique has several advantages over other thermal maturation classifications. It is an absolute measurement; it is a rigorous analytical evaluation; and it is a universally accepted technique. $R_o$ values ranging from 0.2 to 0.6 indicate that the sediments are too immature for oil generation. The zone of petroleum generation is usually interpreted to range from 0.6 to 1.2. $R_o$ from 1.2 to 3.0 indicate a thermal history sufficient to form wet gas and methane. The severely altered or metamorphised organic matter represented by $R_o$ values greater than 3.0 is considered as a nonsource for hydrocarbons.

Tables 67 through 70 list the $R_o$ values for the P-1, VA-1, I-2 and I-3 wells. The mean average vitrinite reflectance value for the P-1 well is 0.71. This value is rather misleading, however, in that the actual values indicate a much higher average value (0.97) for the shallower sample while the deeper samples have an average value of 0.46. For the VA-1 well the average value approached 1 and was fairly




$C_{15}$+ Extract Data
for Core Samples

FIGURE 7

UGR File #C051/78Q1
Oct.-Dec. 1977
Mound Facility

TABLE 47

**Summary of C15+ Soxhlet Extraction, Deasphaltening and Liquid Chromatography**

A. Weights of Extracts and Chromatographic Fractions

| Mound Sample Number | Well Interval | Weight of Rock Extd. (Grams) | Total Extract (Grams) | Precipitated Asphaltenes (Grams) | N-C5 Soluble (Grams) | Sulfur (Grams) | Paraffins-Naphthenes (Grams) | Aromatics (Grams) | Eluted NSO'S (Grams) | Noneluted NSO's (Grams) |
|---|---|---|---|---|---|---|---|---|---|---|
| P-1-1 | | 100.0 | 0.0265 | 0.0204 | 0.0061 | N.D. | N.D. | N.D. | N.D. | N.D. |
| P-1-2 | | 100.0 | 0.4642 | 0.0394 | 0.4248 | N.D. | 0.1500 | 0.1952 | 0.0624 | 0.0172 |
| P-1-3 | | 100.0 | 0.5276 | 0.0493 | 0.4783 | N.D. | 0.2026 | 0.1185 | 0.0813 | 0.0759 |

TABLE 48

Summary of C15+ Soxhlet Extraction, Deasphaltening and Liquid Chromatography

B. Concentration of Extracted Materials in Rock

| Mound Sample Number | Well Interval | Total Extract (ppm) | Hydrocarbons: Paraffin-Naphthene (ppm) | Hydrocarbons: Aromatic (ppm) | Hydrocarbons: Total (ppm) | Sulfur (ppm) | Nonhydrocarbons: Preciptd. Asphaltene (ppm) | Nonhydrocarbons: Eluted NSO's (ppm) | Nonhydrocarbons: Noneluted NSO's (ppm) | Nonhydrocarbons: Total (ppm) |
|---|---|---|---|---|---|---|---|---|---|---|
| P-1-1 | | 265 | -- | -- | -- | -- | 204 | -- | -- | -- |
| P-1-2 | | 4642 | 1500 | 1952 | 3452 | -- | 394 | 624 | 172 | 1190 |
| P-1-3 | | 5276 | 2026 | 1185 | 3211 | -- | 493 | 813 | 759 | 2065 |

TABLE 49

Summary of C15+ Soxhlet Extraction, Deasphaltening and Liquid Chromatography

C. Composition of Extracts

| Mound Sample Number | Well Interval | Hydrocarbons: Paraffin-Naphthene % | Hydrocarbons: Aromatic % | Hydrocarbons: PN/Arom | Sulfur % | Nonhydrocarbons: Eluted ESO'S % | Nonhydrocarbons: Noneluted NSO'S % | Nonhydrocarbons: Precipitd. Asphaltene % | Asph/NSO | HC'S % | HC/Non HC |
|---|---|---|---|---|---|---|---|---|---|---|---|
| P-1-1 | | -- | -- | -- | -- | -- | -- | 77.0 | -- | -- | -- |
| P-1-2 | | 32.3 | 42.1 | 0.77 | -- | 13.4 | 3.7 | 8.5 | 0.49 | 74.4 | 2.90 |
| P-1-3 | | 38.4 | 22.5 | 1.71 | -- | 15.4 | 14.4 | 9.3 | 0.31 | 60.9 | 1.55 |

UGR File #C051/78Q1
Oct.-Dec. 1977
Mound Facility

TABLE 50

Saturate Hydrocarbon Analyses

Summary of Paraffin-Naphthene Distribution

| Mound Sample Number | Well Interval | % Paraffin | % Isoprenoid | % Naphthene | C-P Index A | C-P Index B | ip19/ip20 |
|---|---|---|---|---|---|---|---|
| P-1-1 | | 5.0 | 0.1 | 94.9 | 0.99 | 1.11 | 0.71 |
| P-1-2 | | 8.8 | 1.7 | 89.5 | -- | -- | 1.30 |
| P-1-3 | | 8.5 | 1.6 | 89.9 | -- | -- | 1.40 |

UGR File #C051/78Q1
Oct.-Dec. 1977
Mound Facility

TABLE 51

Saturate Hydrocarbon Analyses

Normalized Paraffin Distribution

| Mound Sample Number | Well Interval | % nC15 | % nC16 | % nC17 | % ip19 | % nC18 | % ip20 | % nC19 | % nC20 | % nC21 | % nC22 | % nC23 | % nC24 | % nC25 | % nC26 | % nC27 | % nC28 | % nC29 | % nC30 | % nC31 | % nC32 | % nC33 | % nC34 | % nC35 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| P-1-1 | | 0.0 | 0.0 | 1.5 | 0.9 | 4.7 | 1.3 | 8.1 | 9.8 | 9.0 | 8.6 | 6.0 | 5.8 | 5.8 | 6.0 | 7.5 | 6.9 | 6.8 | 4.7 | 3.8 | 2.1 | 0.8 | 0.0 | 0.0 |
| P-1-2 | | 5.1 | 10.3 | 13.1 | 9.0 | 13.6 | 6.9 | 12.1 | 9.9 | 7.3 | 5.2 | 3.6 | 2.1 | 1.3 | 0.4 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| P-1-3 | | 6.8 | 11.7 | 13.5 | 9.3 | 13.2 | 6.7 | 11.0 | 8.9 | 6.5 | 4.9 | 3.6 | 2.1 | 1.2 | 0.6 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |

TABLE 52

Summary of C15+ Soxhlet Extraction, Deasphaltening and Liquid Chromatography

A. Weights of Extracts and Chromatographic Fractions

| Mound Sample Number | Well Interval | Weight of Rock Extd. (Grams) | Total Extract (Grams) | Precipitated Asphaltenes (Grams) | N-C5 Soluble (Grams) | Sulfur (Grams) | Paraffins-Naphthenes (Grams) | Aromatics (Grams) | Eluted NSO'S (Grams) | Noneluted NSO's (Grams) |
|---|---|---|---|---|---|---|---|---|---|---|
| VA-1-2 | | 100.0 | 0.0297 | 0.0141 | 0.0156 | N.D. | 0.0057 | 0.0045 | 0.0034 | 0.0020 |
| VA-1-4 | | 100.0 | 0.0357 | 0.0271 | 0.0086 | N.D. | N.D. | N.D. | N.D. | N.D. |
| VA-1-6 | | 100.0 | 0.0344 | 0.0304 | 0.0040 | N.D. | N.D. | N.D. | N.D. | N.D. |
| VA-1-11 | | 90.0 | 0.0199 | 0.0169 | 0.0030 | N.D. | N.D. | N.D. | N.D. | N.D. |
| VA-1-13 | | 100.0 | 0.0393 | 0.0366 | 0.0027 | N.D. | N.D. | N.D. | N.D. | N.D. |
| VA-1-15 | | 100.0 | 0.0356 | 0.0314 | 0.0042 | N.D. | N.D. | N.D. | N.D. | N.D. |
| VA-1-17 | | 100.0 | 0.0347 | 0.0311 | 0.0036 | N.D. | N.D. | N.D. | N.D. | N.D. |
| VA-1-18 | | 100.0 | 0.0395 | 0.0350 | 0.0045 | N.D. | N.D. | N.D. | N.D. | N.D. |
| VA-1-21 | | 100.0 | 0.0330 | 0.0271 | 0.0059 | N.D. | N.D. | N.D. | N.D. | N.D. |
| VA-1-22 | | 100.0 | 0.0453 | 0.0379 | 0.0074 | N.D. | N.D. | N.D. | N.D. | N.D. |
| VA-1-25 | | 100.0 | 0.0256 | 0.0180 | 0.0076 | N.D. | N.D. | N.D. | N.D. | N.D. |
| VA-1-26 | | 100.0 | 0.0558 | 0.0459 | 0.0099 | N.D. | N.D. | N.D. | N.D. | N.D. |
| VA-1-27 | | 100.0 | 0.0336 | 0.0304 | 0.0032 | N.D. | N.D. | N.D. | N.D. | N.D. |
| VA-1-29 | | 100.0 | 0.0446 | 0.0352 | 0.0094 | N.D. | N.D. | N.D. | N.D. | N.D. |
| VA-1-32 | | 100.0 | 0.0329 | 0.0282 | 0.0047 | N.D. | N.D. | N.D. | N.D. | N.D. |

TABLE 53

Summary of C15+ Soxhlet Extraction, Deasphaltening and Liquid Chromatography

B. Concentration of Extracted Materials in Rock

| Mound Sample Number | Well Interval | Total Extract (ppm) | Hydrocarbons: Paraffin-Naphthene (ppm) | Hydrocarbons: Aromatic (ppm) | Hydrocarbons: Total (ppm) | Sulfur (ppm) | Nonhydrocarbons: Preciptd. Asphaltene (ppm) | Nonhydrocarbons: Eluted NSO's (ppm) | Nonhydrocarbons: Noneluted NSO's (ppm) | Nonhydrocarbons: Total (ppm) |
|---|---|---|---|---|---|---|---|---|---|---|
| VA-1-2 | | 297 | 57 | 45 | 102 | -- | 141 | 34 | 20 | 195 |
| VA-1-4 | | 357 | -- | -- | -- | -- | 271 | -- | -- | -- |
| VA-1-6 | | 344 | -- | -- | -- | -- | 304 | -- | -- | -- |
| VA-1-11 | | 221 | -- | -- | -- | -- | 188 | -- | -- | -- |
| VA-1-13 | | 393 | -- | -- | -- | -- | 366 | -- | -- | -- |
| VA-1-15 | | 356 | -- | -- | -- | -- | 314 | -- | -- | -- |
| VA-1-17 | | 347 | -- | -- | -- | -- | 311 | -- | -- | -- |
| VA-1-18 | | 395 | -- | -- | -- | -- | 350 | -- | -- | -- |
| VA-1-21 | | 330 | -- | -- | -- | -- | 271 | -- | -- | -- |
| VA-1-22 | | 453 | -- | -- | -- | -- | 379 | -- | -- | -- |
| VA-1-25 | | 256 | -- | -- | -- | -- | 180 | -- | -- | -- |
| VA-1-26 | | 558 | -- | -- | -- | -- | 459 | -- | -- | -- |
| VA-1-27 | | 336 | -- | -- | -- | -- | 304 | -- | -- | -- |
| VA-1-29 | | 446 | -- | -- | -- | -- | 352 | -- | -- | -- |
| VA-1-32 | | 329 | -- | -- | -- | -- | 282 | -- | -- | -- |

TABLE 54

Summary of C15+ Soxhlet Extraction, Deasphaltening and Liquid Chromatography

C. Composition of Extracts

| Mound Sample Number | Well Interval | Hydrocarbons: Paraffin-Naphthene % | Hydrocarbons: Aromatic % | Hydrocarbons: PN/Arom | Hydrocarbons: Sulfur % | Nonhydrocarbons: Eluted NSO'S % | Nonhydrocarbons: Noneluted NSO'S % | Nonhydrocarbons: Precipitd. Asphaltene % | Nonhydrocarbons: Asph/NSO | Nonhydrocarbons: HC'S % | Nonhydrocarbons: HC/Non HC |
|---|---|---|---|---|---|---|---|---|---|---|---|
| VA-1-2 | | 19.2 | 15.2 | 1.27 | -- | 11.4 | 6.7 | 47.5 | 2.61 | 34.3 | 0.52 |
| VA-1-4 | | -- | -- | -- | -- | -- | -- | 75.9 | -- | -- | -- |
| VA-1-6 | | -- | -- | -- | -- | -- | -- | 88.4 | -- | -- | -- |
| VA-1-11 | | -- | -- | -- | -- | -- | -- | 84.9 | -- | -- | -- |
| VA-1-13 | | -- | -- | -- | -- | -- | -- | 93.1 | -- | -- | -- |
| VA-1-15 | | -- | -- | -- | -- | -- | -- | 88.2 | -- | -- | -- |
| VA-1-17 | | -- | -- | -- | -- | -- | -- | 89.6 | -- | -- | -- |
| VA-1-18 | | -- | -- | -- | -- | -- | -- | 88.6 | -- | -- | -- |
| VA-1-21 | | -- | -- | -- | -- | -- | -- | 82.1 | -- | -- | -- |
| VA-1-22 | | -- | -- | -- | -- | -- | -- | 83.7 | -- | -- | -- |
| VA-1-25 | | -- | -- | -- | -- | -- | -- | 70.3 | -- | -- | -- |
| VA-1-26 | | -- | -- | -- | -- | -- | -- | 82.3 | -- | -- | -- |
| VA-1-27 | | -- | -- | -- | -- | -- | -- | 90.5 | -- | -- | -- |
| VA-1-29 | | -- | -- | -- | -- | -- | -- | 78.9 | -- | -- | -- |
| VA-1-32 | | -- | -- | -- | -- | -- | -- | 85.7 | -- | -- | -- |

TABLE 55

Saturate Hydrocarbon Analyses

Summary of Paraffin-Naphthene Distribution

| Mound Sample Number | Well Interval | % Paraffin | % Isoprenoid | % Naphthene | C-P Index A | C-P Index B | ip19/ip20 |
|---|---|---|---|---|---|---|---|
| VA-1-2 | | 7.2 | 1.3 | 91.5 | -- | -- | 0.64 |
| VA-1-4 | | 7.8 | 1.1 | 91.2 | -- | -- | 0.42 |
| VA-1-6 | | 3.8 | 0.0 | 96.2 | 1.03 | -- | 0.00 |
| VA-1-11 | | 3.8 | 0.2 | 96.0 | 1.05 | -- | 0.38 |
| VA-1-13 | | 6.9 | 0.0 | 93.1 | 1.20 | -- | 0.00 |
| VA-1-15 | | 3.1 | 0.4 | 96.5 | -- | -- | 0.24 |
| VA-1-17 | | 4.6 | 0.0 | 95.4 | 0.68 | -- | 0.00 |
| VA-1-18 | | 2.1 | 0.4 | 97.5 | -- | -- | 0.52 |
| VA-1-21 | | 2.1 | 0.0 | 97.8 | -- | -- | 0.00 |
| VA-1-22 | | 1.5 | 0.1 | 98.4 | 1.05 | -- | 0.00 |
| VA-1-25 | | 1.5 | 0.0 | 98.4 | -- | -- | 0.00 |
| VA-1-26 | | 1.4 | 0.1 | 98.5 | 1.02 | -- | 0.00 |
| VA-1-27 | | 3.3 | 0.0 | 96.7 | 1.08 | -- | 0.00 |
| VA-1-29 | | 1.5 | 0.1 | 98.4 | -- | -- | 0.00 |
| VA-1-32 | | 2.7 | 0.0 | 97.3 | 1.02 | -- | 0.50 |

TABLE 56

Saturate Hydrocarbon Analyses

Normalized Paraffin Distribution

| Mound Sample Number | Well Interval | % nC15 | % nC16 | % nC17 | % ip19 | % nC18 | % ip20 | % nC19 | % nC20 | % nC21 | % nC22 | % nC23 | % nC24 | % nC25 | % nC26 | % nC27 | % nC28 | % nC29 | % nC30 | % nC31 | % nC32 | % nC33 | % nC34 | % nC35 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| VA-1-2 | | 0.0 | 3.2 | 10.5 | 5.9 | 17.9 | 9.2 | 17.9 | 12.8 | 9.2 | 6.5 | 3.8 | 2.2 | 1.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| VA-1-4 | | 0.0 | 0.0 | 6.0 | 3.6 | 16.0 | 8.6 | 20.4 | 17.2 | 11.5 | 8.1 | 4.9 | 2.6 | 1.3 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| VA-1-6 | | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 10.2 | 13.9 | 19.8 | 17.0 | 11.7 | 10.5 | 7.1 | 4.6 | 2.8 | 2.5 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| VA-1-11 | | 0.0 | 0.0 | 2.0 | 1.5 | 6.7 | 3.9 | 8.9 | 10.0 | 8.3 | 9.8 | 8.5 | 5.9 | 9.5 | 7.2 | 4.8 | 4.1 | 3.5 | 3.0 | 2.2 | 0.0 | 0.0 | 0.0 | 0.0 |
| VA-1-13 | | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 8.2 | 13.5 | 19.7 | 21.4 | 12.7 | 11.7 | 5.5 | 4.2 | 1.2 | 1.2 | 0.5 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| VA-1-15 | | 0.0 | 0.0 | 2.7 | 2.0 | 12.9 | 8.2 | 16.1 | 16.5 | 11.8 | 11.8 | 8.2 | 5.1 | 3.5 | 1.2 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| VA-1-17 | | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 7.2 | 16.2 | 12.0 | 14.9 | 12.0 | 21.5 | 6.4 | 4.5 | 2.7 | 1.6 | 1.1 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| VA-1-18 | | 0.0 | 0.0 | 5.1 | 5.6 | 15.7 | 10.6 | 16.2 | 15.7 | 10.6 | 9.1 | 6.1 | 2.5 | 3.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| VA-1-21 | | 0.0 | 0.0 | 0.0 | 0.0 | 6.8 | 2.1 | 11.9 | 21.7 | 11.5 | 15.3 | 10.6 | 9.8 | 6.4 | 3.8 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| VA-1-22 | | 0.0 | 0.0 | 0.0 | 0.0 | 11.1 | 3.5 | 14.6 | 15.8 | 10.5 | 11.7 | 8.8 | 4.1 | 7.6 | 7.0 | 4.1 | 1.2 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| VA-1-25 | | 0.0 | 0.0 | 0.0 | 0.0 | 11.4 | 3.0 | 17.4 | 23.5 | 18.2 | 0.0 | 13.6 | 5.3 | 5.3 | 2.3 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| VA-1-26 | | 0.0 | 0.0 | 0.0 | 0.0 | 8.6 | 3.4 | 13.2 | 20.1 | 13.8 | 16.1 | 9.8 | 5.7 | 5.2 | 1.1 | 2.3 | 0.6 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| VA-1-27 | | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 4.7 | 11.5 | 15.8 | 20.5 | 16.2 | 9.8 | 9.0 | 5.6 | 3.8 | 1.3 | 1.7 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| VA-1-29 | | 0.0 | 0.0 | 0.0 | 0.0 | 9.2 | 3.4 | 14.3 | 20.2 | 16.0 | 16.8 | 10.1 | 10.1 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| VA-1-32 | | 0.0 | 0.0 | 0.4 | 0.0 | 4.6 | 0.8 | 7.5 | 10.0 | 10.4 | 14.6 | 14.6 | 12.1 | 10.8 | 7.5 | 4.6 | 2.1 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |

TABLE 57

Summary of C15+ Soxhlet Extraction, Deasphaltening and Liquid Chromatography

A. Weights of Extracts and Chromatographic Fractions

| Mound Sample Number | Well Interval | Weight of Rock Extd. (Grams) | Total Extract (Gram) | Precipitated Asphaltenes (Grams) | N-C5 Soluble (Grams) | Sulfur (Grams) | Paraffins-Naphthenes (Grams) | Aromatics (Grams) | Eluted NSO'S (Grams) | Noneluted NSO's (Grams) |
|---|---|---|---|---|---|---|---|---|---|---|
| I-2-1 | | 100.0 | 0.0127 | 0.0104 | 0.0023 | N.D. | N.D. | N.D. | N.D. | N.D. |
| I-2-4 | | 100.0 | 0.0157 | 0.0108 | 0.0049 | N.D. | N.D. | N.D. | N.D. | N.D. |
| I-2-6 | | 100.0 | 0.0281 | 0.0178 | 0.0103 | N.D. | 0.0029 | 0.0051 | 0.0020 | 0.0003 |
| I-2-7 | | 100.0 | 0.0426 | 0.0194 | 0.0232 | N.D. | 0.0061 | 0.0114 | 0.0048 | 0.0009 |
| I-2-10 | | 100.0 | 0.1568 | 0.0371 | 0.1197 | N.D. | 0.0287 | 0.0562 | 0.0348 | 0.0000 |
| I-2-12 | | 100.0 | 0.2107 | 0.0453 | 0.1654 | N.D. | 0.0365 | 0.0738 | 0.0534 | 0.0017 |
| I-2-14 | | 100.0 | 0.0065 | 0.0052 | 0.0013 | N.D. | N.D. | N.D. | N.D. | N.D. |
| I-2-16 | | 100.0 | 0.1040 | 0.0174 | 0.0866 | N.D. | 0.0190 | 0.0384 | 0.0291 | 0.0001 |
| I-2-18 | | 100.0 | 0.1183 | 0.0187 | 0.0996 | N.D. | 0.0232 | 0.0449 | 0.0285 | 0.0030 |
| I-2-20 | | 100.0 | 0.0252 | 0.0151 | 0.0101 | N.D. | 0.0025 | 0.0037 | 0.0037 | 0.0002 |

TABLE 58

Summary of C15+ Soxhlet Extraction, Deasphaltening and Liquid Chromatography

B. Concentration of Extracted Materials in Rock

| Mound Sample Number | Well Interval | Total Extract (ppm) | Hydrocarbons: Paraffin-Naphthene (ppm) | Hydrocarbons: Aromatic (ppm) | Hydrocarbons: Total (ppm) | Sulfur (ppm) | Nonhydrocarbons: Precipitd. Asphaltene (ppm) | Nonhydrocarbons: Eluted NSO's (ppm) | Nonhydrocarbons: Noneluted NSO's (ppm) | Nonhydrocarbons: Total (ppm) |
|---|---|---|---|---|---|---|---|---|---|---|
| I-2-1 | | 127 | -- | -- | -- | -- | 104 | -- | -- | -- |
| I-2-4 | | 157 | -- | -- | -- | -- | 108 | -- | -- | -- |
| I-2-6 | | 281 | 29 | 51 | 80 | -- | 178 | 20 | 3 | 201 |
| I-2-7 | | 426 | 61 | 114 | 175 | -- | 194 | 48 | 9 | 251 |
| I-2-10 | | 1568 | 287 | 562 | 849 | -- | 371 | 348 | 0 | 719 |
| I-2-12 | | 2107 | 365 | 738 | 1103 | -- | 453 | 534 | 17 | 1004 |
| I-2-14 | | 65 | -- | -- | -- | -- | 52 | -- | -- | -- |
| I-2-16 | | 1040 | 190 | 384 | 574 | -- | 174 | 291 | 1 | 466 |
| I-2-18 | | 1183 | 232 | 449 | 681 | -- | 187 | 285 | 30 | 502 |
| I-2-20 | | 252 | 25 | 37 | 62 | -- | 151 | 37 | 2 | 190 |

TABLE 59

Summary of C15+ Soxhlet Extraction, Deasphaltening and Liquid Chromatography

C. Composition of Extracts

| Mound Sample Number | Well Interval | Hydrocarbons: Paraffin-Naphthene % | Hydrocarbons: Aromatic % | Hydrocarbons: PN/Arom | Sulfur % | Nonhydrocarbons: Eluted ESO'S % | Nonhydrocarbons: Noneluted NSO'S % | Nonhydrocarbons: Precipitd. Asphaltene % | Asph/NSO | HC'S % | HC/Non HC |
|---|---|---|---|---|---|---|---|---|---|---|---|
| I-2-1 | | -- | -- | -- | -- | -- | -- | 81.9 | -- | -- | -- |
| I-2-4 | | -- | -- | -- | -- | -- | -- | 68.8 | -- | -- | -- |
| I-2-6 | | 10.3 | 18.1 | 0.57 | -- | 7.1 | 1.1 | 63.3 | 7.74 | 28.5 | 0.40 |
| I-2-7 | | 14.3 | 26.8 | 0.54 | -- | 11.3 | 2.1 | 45.5 | 3.40 | 41.1 | 0.70 |
| I-2-10 | | 18.3 | 35.8 | 0.51 | -- | 22.2 | 0.0 | 23.7 | 1.07 | 54.1 | 1.18 |
| I-2-12 | | 17.3 | 35.0 | 0.49 | -- | 25.3 | 0.8 | 21.5 | 0.82 | 52.3 | 1.10 |
| I-2-14 | | -- | -- | -- | -- | -- | -- | 80.0 | -- | -- | -- |
| I-2-16 | | 18.3 | 36.9 | 0.49 | -- | 28.0 | 0.1 | 16.7 | 0.60 | 55.2 | 1.23 |
| I-2-18 | | 19.6 | 38.0 | 0.52 | -- | 24.1 | 2.5 | 15.8 | 0.59 | 57.6 | 1.36 |
| I-2-20 | | 9.9 | 14.7 | 0.68 | -- | 14.7 | 0.8 | 59.9 | 3.87 | 24.6 | 0.33 |

TABLE 60

Saturate Hydrocarbon Analyses

Summary of Paraffin-Naphthene Distribution

| Mound Sample Number | Well Interval | % Paraffin | % Isoprenoid | % Naphthene | C-P Index A | C-P Index B | ip19/ip20 |
|---|---|---|---|---|---|---|---|
| I-2-1 | 334 | 7.3 | 0.2 | 92.5 | 1.21 | -- | 0.50 |
| I-2-4 | 364 | 11.4 | 0.3 | 88.3 | 1.19 | -- | 0.60 |
| I-2-6 | 394 | 13.3 | 1.7 | 85.0 | 1.22 | -- | 0.93 |
| I-2-7 | 425 | 11.5 | 4.2 | 84.3 | 1.21 | -- | 1.54 |
| I-2-10 | 454 | 8.3 | 4.3 | 87.4 | 1.25 | -- | 0.94 |
| I-2-12 | 481 | 7.4 | 4.0 | 88.6 | 1.14 | -- | 1.27 |
| I-2-14 | 508 | 6.4 | 0.1 | 93.5 | 1.18 | -- | 0.67 |
| I-2-16 | 538 | 8.1 | 4.1 | 87.8 | 1.15 | -- | 1.18 |
| I-2-18 | 568 | 8.8 | 3.2 | 88.0 | 1.14 | -- | 1.09 |
| I-2-20 | 598 | 13.5 | 1.5 | 85.0 | 1.26 | -- | 0.85 |

TABLE 61

Saturate Hydrocarbon Analyses

Normalized Paraffin Distribution

| Mound Sample Number | Well Interval | % nC15 | % nC16 | % nC17 | % ip19 | % nC18 | % ip20 | % nC19 | % nC20 | % nC21 | % nC22 | % nC23 | % nC24 | % nC25 | % nC26 | % nC27 | % nC28 | % nC29 | % nC30 | % nC31 | % nC32 | % nC33 | % nC34 | % nC35 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| I-2-1 | | 0.3 | 0.3 | 1.4 | 0.9 | 5.5 | 1.8 | 10.3 | 13.0 | 15.3 | 14.5 | 11.5 | 7.0 | 6.4 | 3.8 | 4.7 | 1.1 | 1.4 | 0.3 | 0.3 | 0.0 | 0.0 | 0.0 | 0.0 |
| I-2-4 | | 0.2 | 0.2 | 1.1 | 0.9 | 4.7 | 1.5 | 10.8 | 15.2 | 18.1 | 16.2 | 11.3 | 7.1 | 6.2 | 2.7 | 2.9 | 0.6 | 0.1 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| I-2-6 | | 0.1 | 0.6 | 4.1 | 5.5 | 8.5 | 5.9 | 13.2 | 14.7 | 14.0 | 10.9 | 7.7 | 4.6 | 4.8 | 1.9 | 1.9 | 0.9 | 0.4 | 0.1 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 |
| I-2-7 | | 0.7 | 3.1 | 8.1 | 16.2 | 9.7 | 10.5 | 11.3 | 10.0 | 8.7 | 6.8 | 5.0 | 3.0 | 3.4 | 1.2 | 1.0 | 0.7 | 0.2 | 0.1 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 |
| I-2-10 | | 0.1 | 1.5 | 6.4 | 16.5 | 8.6 | 17.6 | 11.3 | 9.9 | 8.6 | 6.6 | 5.2 | 2.6 | 2.8 | 1.0 | 0.7 | 0.3 | 0.1 | 0.1 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 |
| I-2-12 | | 0.1 | 1.7 | 6.9 | 19.7 | 9.0 | 15.5 | 10.8 | 9.3 | 7.6 | 6.0 | 4.5 | 2.9 | 3.2 | 1.7 | 0.8 | 0.3 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| I-2-14 | | 0.3 | 0.5 | 0.6 | 0.3 | 1.7 | 0.5 | 9.8 | 16.1 | 17.2 | 17.2 | 14.4 | 8.1 | 7.1 | 3.0 | 2.2 | 0.3 | 0.3 | 0.2 | 0.2 | 0.0 | 0.0 | 0.0 | 0.0 |
| I-2-16 | | 0.2 | 1.8 | 6.6 | 18.3 | 8.1 | 15.5 | 10.7 | 9.5 | 8.7 | 7.0 | 5.1 | 3.2 | 2.8 | 1.3 | 0.7 | 0.2 | 0.2 | 0.1 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 |
| I-2-18 | | 0.1 | 1.5 | 6.0 | 13.8 | 8.3 | 12.7 | 10.6 | 10.2 | 9.7 | 8.2 | 6.3 | 4.3 | 3.9 | 1.9 | 1.4 | 0.6 | 0.4 | 0.1 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 |
| I-2-20 | | 0.1 | 0.5 | 4.7 | 4.6 | 10.9 | 5.4 | 14.5 | 14.1 | 13.6 | 10.0 | 7.2 | 4.7 | 5.6 | 1.9 | 1.2 | 0.6 | 0.2 | 0.1 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 |

TABLE 62

Summary of C15+ Soxhlet Extraction, Deasphaltening and Liquid Chromatography

A. Weights of Extracts and Chromatographic Fractions

| Mound Sample Number | Well Interval | Weight of Rock Extd. (Grams) | Total Extract (Gram) | Precipitated Asphaltenes (Grams) | N-C5 Soluble (Grams) | Sulfur (Grams) | Paraffins-Naphthenes (Grams) | Aromatics (Grams) | Eluted NSO'S (Grams) | Noneluted NSO's (Grams) |
|---|---|---|---|---|---|---|---|---|---|---|
| I-3-1 | | 95.2 | 0.0077 | 0.0047 | 0.0030 | N.D. | N.D. | N.D. | N.D. | N.D. |
| I-3-2 | | 99.1 | 0.0061 | 0.0027 | 0.0034 | N.D. | N.D. | N.D. | N.D. | N.D. |
| I-3-4 | | 99.1 | 0.0065 | 0.0027 | 0.0038 | N.D. | N.D. | N.D. | N.D. | N.D. |
| I-3-6 | | 98.8 | 0.0147 | 0.0038 | 0.0109 | N.D. | 0.0029 | 0.0047 | 0.0021 | 0.0012 |
| I-3-7 | | 98.9 | 0.0185 | 0.0051 | 0.0134 | N.D. | 0.0034 | 0.0057 | 0.0020 | 0.0023 |
| I-3-9 | | 99.0 | 0.0368 | 0.0066 | 0.0302 | N.D. | 0.0081 | 0.0156 | 0.0049 | 0.0016 |
| I-3-11 | | 98.8 | 0.0854 | 0.0106 | 0.0748 | N.D. | 0.0184 | 0.0374 | 0.0177 | 0.0013 |
| I-3-12 | | 99.1 | 0.0730 | 0.0091 | 0.0639 | N.D. | 0.0158 | 0.0328 | 0.0153 | 0.0000 |
| I-3-14 | | 98.8 | 0.1778 | 0.0125 | 0.1653 | N.D. | 0.0429 | 0.0773 | 0.0386 | 0.0065 |
| I-3-16 | | 99.0 | 0.2232 | 0.0155 | 0.2077 | N.D. | 0.0358 | 0.0975 | 0.0653 | 0.0091 |
| I-3-17 | | 98.8 | 0.2248 | 0.0155 | 0.2093 | N.D. | 0.0631 | 0.0900 | 0.0526 | 0.0036 |
| I-3-19 | | 99.0 | 0.1145 | 0.0097 | 0.1048 | N.D. | 0.0327 | 0.0498 | 0.0223 | 0.0000 |

TABLE 63

Summary of C15+ Soxhlet Extraction, Deasphaltening and Liquid Chromatography

B. Concentration of Extracted Materials in Rock

| Mound Sample Number | Well Interval | Total Extract (ppm) | Hydrocarbons: Paraffin-Naphthene (ppm) | Hydrocarbons: Aromatic (ppm) | Hydrocarbons: Total (ppm) | Sulfur (ppm) | Nonhydrocarbons: Preciptd. Asphaltene (ppm) | Nonhydrocarbons: Eluted NSO's (ppm) | Nonhydrocarbons: Noneluted NSO's (ppm) | Nonhydrocarbons: Total (ppm) |
|---|---|---|---|---|---|---|---|---|---|---|
| I-3-1 | | 81 | -- | -- | -- | -- | 49 | -- | -- | -- |
| I-3-2 | | 62 | -- | -- | -- | -- | 27 | -- | -- | -- |
| I-3-4 | | 66 | -- | -- | -- | -- | 27 | -- | -- | -- |
| I-3-6 | | 149 | 29 | 48 | 77 | -- | 38 | 21 | 12 | 72 |
| I-3-7 | | 187 | 34 | 58 | 92 | -- | 52 | 20 | 23 | 95 |
| I-3-9 | | 372 | 82 | 158 | 239 | -- | 67 | 49 | 16 | 132 |
| I-3-11 | | 864 | 186 | 379 | 565 | -- | 107 | 179 | 13 | 300 |
| I-3-12 | | 737 | 159 | 331 | 490 | -- | 92 | 154 | 0 | 246 |
| I-3-14 | | 1800 | 434 | 782 | 1217 | -- | 127 | 391 | 66 | 583 |
| I-3-16 | | 2255 | 362 | 985 | 1346 | -- | 157 | 660 | 92 | 908 |
| I-3-17 | | 2275 | 639 | 911 | 1550 | -- | 157 | 532 | 36 | 726 |
| I-3-19 | | 1157 | 330 | 503 | 833 | -- | 98 | 225 | 0 | 323 |

TABLE 64

Summary of C15+ Soxhlet Extraction, Deasphaltening and Liquid Chromatography

C. Composition of Extracts

| Mound Sample Number | Well Interval | Hydrocarbons: Paraffin-Naphthene % | Hydrocarbons: Aromatic % | Hydrocarbons: PN/Arom | Hydrocarbons: Sulfur % | Nonhydrocarbons: Eluted ESO'S % | Nonhydrocarbons: Noneluted NSO'S % | Nonhydrocarbons: Precipitd. Asphaltene % | Nonhydrocarbons: Asph/NSO | Nonhydrocarbons: HC'S % | Nonhydrocarbons: HC/Non HC |
|---|---|---|---|---|---|---|---|---|---|---|---|
| I-3-1 | | -- | -- | -- | -- | -- | -- | 61.0 | -- | -- | -- |
| I-3-2 | | -- | -- | -- | -- | -- | -- | 44.3 | -- | -- | -- |
| I-3-4 | | -- | -- | -- | -- | -- | -- | 41.5 | -- | -- | -- |
| I-3-6 | | 19.7 | 32.0 | 0.62 | -- | 14.3 | 8.2 | 25.9 | 1.15 | 51.7 | 1.07 |
| I-3-7 | | 18.4 | 30.8 | 0.60 | -- | 10.8 | 12.4 | 27.6 | 1.19 | 49.2 | 0.97 |
| I-3-9 | | 22.0 | 42.4 | 0.52 | -- | 13.3 | 4.3 | 17.9 | 1.02 | 64.4 | 1.81 |
| I-3-11 | | 21.5 | 43.8 | 0.49 | -- | 20.7 | 1.5 | 12.4 | 0.56 | 65.3 | 1.89 |
| I-3-12 | | 21.6 | 44.9 | 0.48 | -- | 21.0 | 0.0 | 12.5 | 0.59 | 66.6 | 1.99 |
| I-3-14 | | 24.1 | 43.5 | 0.55 | -- | 21.7 | 3.7 | 7.0 | 0.28 | 67.6 | 2.09 |
| I-3-16 | | 16.0 | 43.7 | 0.37 | -- | 29.3 | 4.1 | 6.9 | 0.21 | 59.7 | 1.48 |
| I-3-17 | | 28.1 | 40.0 | 0.70 | -- | 23.4 | 1.6 | 6.9 | 0.28 | 68.1 | 2.14 |
| I-3-19 | | 28.6 | 43.5 | 0.66 | -- | 19.5 | 0.0 | 8.5 | 0.43 | 72.1 | 2.58 |

TABLE 65

Saturate Hydrocarbon Analyses

Summary of Paraffin-Naphthene Distribution

| Mound Sample Number | Well Interval | % Paraffin | % Isoprenoid | % Naphthene | C-P Index A | C-P Index B | ip19/ip20 |
|---|---|---|---|---|---|---|---|
| I-3-1 | | 5.0 | 0.2 | 94.9 | 1.10 | -- | 1.17 |
| I-3-2 | | 9.2 | 0.2 | 90.6 | 1.08 | -- | 1.25 |
| I-3-4 | | 13.0 | 0.8 | 86.2 | 1.07 | 1.33 | 1.08 |
| I-3-6 | | 17.6 | 3.2 | 79.2 | 1.20 | 1.41 | 1.30 |
| I-3-7 | | 22.5 | 5.4 | 72.0 | 1.14 | 1.31 | 1.60 |
| I-3-9 | | 13.9 | 5.8 | 80.3 | 1.24 | -- | 1.43 |
| I-3-11 | | 11.3 | 4.7 | 84.0 | 1.19 | -- | 1.23 |
| I-3-12 | | 10.5 | 4.5 | 85.0 | 1.17 | -- | 1.15 |
| I-3-14 | | 10.0 | 4.1 | 85.9 | 1.16 | -- | 1.70 |
| I-3-16 | | 11.8 | 5.1 | 83.1 | 1.11 | -- | 1.41 |
| I-3-17 | | 9.3 | 4.0 | 86.7 | 1.10 | -- | 1.32 |
| I-3-19 | | 15.5 | 3.5 | 81.0 | 1.07 | 1.05 | 1.37 |

TABLE 66

**Saturate Hydrocarbon Analyses**

**Normalized Paraffin Distribution**

| Mound Sample Number | Well Interval | % nC15 | % nC16 | % nC17 | % ip19 | % nC18 | % ip20 | % nC19 | % nC20 | % nC21 | % nC22 | % nC23 | % nC24 | % nC25 | % nC26 | % nC27 | % nC28 | % nC29 | % nC30 | % nC31 | % nC32 | % nC33 | % nC34 | % nC35 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| I-3-1 | | 0.2 | 0.5 | 1.9 | 1.7 | 5.3 | 1.4 | 9.7 | 13.3 | 13.5 | 13.5 | 11.4 | 8.2 | 7.5 | 5.3 | 3.9 | 1.0 | 1.0 | 0.2 | 0.5 | 0.0 | 0.0 | 0.0 | 0.0 |
| I-3-2 | | 0.1 | 0.2 | 1.1 | 1.1 | 5.9 | 0.9 | 13.2 | 17.3 | 16.4 | 14.5 | 10.2 | 6.4 | 4.8 | 3.1 | 2.5 | 1.3 | 0.4 | 0.2 | 0.2 | 0.0 | 0.0 | 0.0 | 0.0 |
| I-3-4 | | 0.4 | 0.8 | 3.4 | 3.0 | 9.0 | 2.7 | 13.0 | 14.3 | 12.9 | 11.6 | 8.9 | 6.2 | 5.0 | 3.3 | 2.5 | 1.1 | 0.9 | 0.3 | 0.4 | 0.1 | 0.0 | 0.0 | 0.0 |
| I-3-6 | | 0.2 | 1.3 | 5.7 | 8.6 | 9.5 | 6.6 | 11.7 | 11.2 | 10.4 | 8.7 | 6.9 | 4.3 | 4.2 | 2.5 | 4.3 | 2.4 | 0.8 | 0.3 | 0.3 | 0.1 | 0.0 | 0.0 | 0.0 |
| I-3-7 | | 0.3 | 2.7 | 7.9 | 12.0 | 10.3 | 7.5 | 11.6 | 10.3 | 9.6 | 7.9 | 6.3 | 3.9 | 3.6 | 2.2 | 1.6 | 1.1 | 0.7 | 0.2 | 0.3 | 0.1 | 0.1 | 0.0 | 0.0 |
| I-3-9 | | 0.3 | 3.2 | 8.8 | 17.3 | 10.7 | 12.1 | 11.2 | 9.3 | 8.1 | 6.1 | 4.3 | 2.2 | 3.2 | 1.1 | 0.9 | 0.7 | 0.2 | 0.1 | 0.2 | 0.0 | 0.0 | 0.0 | 0.0 |
| I-3-11 | | 1.1 | 3.1 | 6.5 | 16.2 | 8.4 | 13.1 | 9.9 | 9.6 | 8.8 | 7.3 | 5.4 | 2.9 | 3.8 | 1.6 | 1.0 | 0.9 | 0.2 | 0.1 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 |
| I-3-12 | | 0.5 | 2.6 | 6.5 | 16.0 | 8.7 | 13.9 | 10.0 | 9.5 | 8.6 | 7.3 | 5.2 | 2.7 | 4.2 | 1.8 | 1.0 | 1.1 | 0.2 | 0.1 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 |
| I-3-14 | | 5.1 | 7.2 | 9.7 | 18.4 | 9.1 | 10.8 | 9.0 | 7.8 | 6.6 | 5.4 | 3.9 | 2.2 | 2.6 | 1.0 | 0.5 | 0.6 | 0.1 | 0.1 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 |
| I-3-16 | | 5.8 | 8.1 | 10.1 | 17.6 | 9.2 | 12.4 | 8.5 | 7.3 | 6.1 | 4.9 | 3.8 | 2.1 | 2.1 | 1.1 | 0.2 | 0.4 | 0.1 | 0.1 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 |
| I-3-17 | | 4.0 | 6.6 | 9.6 | 17.0 | 9.5 | 12.9 | 9.1 | 7.8 | 6.6 | 5.6 | 4.1 | 2.4 | 2.4 | 1.2 | 0.5 | 0.5 | 0.1 | 0.1 | 0.1 | 0.0 | 0.0 | 0.0 | 0.0 |
| I-3-19 | | 1.9 | 5.7 | 9.6 | 10.8 | 10.4 | 7.8 | 9.9 | 9.3 | 8.5 | 7.2 | 5.8 | 3.7 | 4.0 | 2.8 | 0.9 | 0.9 | 0.3 | 0.1 | 0.2 | 0.1 | 0.0 | 0.0 | 0.0 |

TABLE 67

Vitrinite Reflectance Summary
for P-1 Well

| Mound Sample Number | Number of Readings | Minimum Reflectance (% $R_o$) | Maximum Reflectance (% $R_o$) | Average Reflectance (% $R_o$) |
|---|---|---|---|---|
| P-1-1 | 5 | 0.61 | 0.84 | 0.69* |
| | 6 | 1.10 | 1.37 | 1.24* |
| P-1-2 | 50 | 0.31 | 0.67 | 0.47 |
| P-1-3 | 50 | 0.30 | 0.61 | 0.44 |

*Twp populations.

TABLE 68

Vitrinite Reflectance Summary
for VA-1 Well

| Mound Sample Number | Number of Readings | Minimum Reflectance (% $R_o$) | Maximum Reflectance (% $R_o$) | Average Reflectance (% $R_o$) |
|---|---|---|---|---|
| VA-1-2 | 50 | 0.47 | 1.39 | 0.96 |
| VA-1-4 | 50 | 0.48 | 1.45 | 0.97 |
| VA-1-6 | 6 | 0.67 | 1.41 | 1.19 |
| VA-1-11 | 3 | 1.01 | 1.34 | 1.21 |
| VA-1-13 | 1 | 1.28 | 1.28 | 1.28 |
| VA-1-15 | 50 | 0.48 | 1.26 | 0.92 |
| VA-1-17 | 33 | 0.63 | 1.24 | 0.93 |
| VA-1-18 | 50 | 0.66 | 1.28 | 0.92 |
| VA-1-21 | 35 | 0.67 | 1.36 | 1.01 |
| VA-1-22 | 46 | 0.46 | 1.39 | 0.96 |
| VA-1-25 | 50 | 0.49 | 1.35 | 1.02 |
| VA-1-26 | 50 | 0.68 | 1.44 | 1.02 |
| VA-1-27 | 50 | 0.53 | 1.47 | 1.10 |
| VA-1-29 | 50 | 0.69 | 1.15 | 0.94 |
| VA-1-32 | 50 | 0.64 | 1.25 | 0.91 |

TABLE 69

Vitrinite Reflectance Summary
for I-2 Well

| Mound Sample Number | Number of Readings | Minimum Reflectance (% Ro) | Maximum Reflectance (% Ro) | Average Reflectance (% Ro) |
|---|---|---|---|---|
| I-2-A | 45 | 0.34 | 0.53 | 0.43 |
| I-2-1 | 2 | 0.29 | 0.42 | 0.35 |
| I-2-4 | 10 | 0.24 | 0.60 | 0.38 |
| I-2-6 | 5 | 0.44 | 0.53 | 0.48 |
| I-2-7 | 4 | 0.29 | 0.59 | 0.44 |
| I-2-10 | 25 | 0.22 | 0.67 | 0.34 |
| I-2-12 | 14 | 0.19 | 0.58 | 0.33 |
| I-2-14 | ** | -- | -- | -- |
| I-2-16 | 18 | 0.21 | 0.60 | 0.43 |
| I-2-18 | 9 | 0.28 | 0.50 | 0.41 |
| I-2-20 | ** | -- | -- | -- |
| I-2-B | 30 | 0.27 | 0.63 | 0.41 |

** No vitrinite observed in sample.

TABLE 70

Vitrinite Reflectance Summary
for I-3- Well

| Mound Sample Number | Number of Readings | Minimum Reflectance (% Ro) | Maximum Reflectance (% Ro) | Average Reflectance (% Ro) |
|---|---|---|---|---|
| I-3-1 | ** | -- | -- | -- |
| I-3-2 | ** | -- | -- | -- |
| I-3-4 | ** | -- | -- | -- |
| I-3-6 | 3 | 0.43 | 0.68 | 0.55 |
| I-3-7 | 2 | 0.35 | 0.36 | 0.35 |
| I-3-9 | 6 | 0.32 | 0.55 | 0.45 |
| I-3-11 | 14 | 0.36 | 0.63 | 0.50 |
| I-3-12 | 14 | 0.37 | 0.66 | 0.55 |
| I-3-14 | 8 | 0.37 | 0.62 | 0.43 |
| I-3-16 | 33 | 0.35 | 0.65 | 0.47 |
| I-3-17 | 20 | 0.32 | 0.71 | 0.45 |
| I-3-19 | 11 | 0.34 | 0.50 | 0.41 |

** Insufficient kerogen return for plug.

consistent downhole. The average value for the I-2 well is 0.40, while that for the I-3 well is 0.46. These values are consistent with the previously determined values of the I-1 and O-1 wells. In both wells the average value was 0.45. The previously determined value for the KY-2 well was 0.52. These data tend to indicate that the VA-1 well is the more mature of those examined to date.

Detailed elemental analysis of the kerogen material from the wells is currently being performed. This analysis determines the specific carbon, hydrogen, nitrogen, and oxygen content of the sample. The analytical results are used to confirm the basic structure of the kerogen and can also be used to assess the hydrocarbon potential of the material

The results for the P-1, VA-1, I-2 and I-3 wells are listed in Tables 71 through 74, respectively. The atomic H to C ratio for the VA-1 is much lower than those for the other wells, this would indicate that cracking has occured and an enrichment of the residue in carbon has resulted. This is supportative evidence for the fact that the VA-1 well is more mature.

Currently, all of the detailed kerogen data are being statistically evaluated in the manner set forth by Tissot and Foscolos. This evaluation includes the integration of the kerogen data with other organic and inorganic geochemical data to statistically evaluate the degree of diagenesis of the Devonian shales in both the Appalachian and Illinois basins.

## G. MATERIAL BALANCE ASSAY

Material Balance Assays (MBA's) are being performed on all the geochemical samples. For the assays approximately 100 grams of well-riffled, representative 4 to 8 mesh material are heated and destructively distilled at a maximum temperature of 500°C. The assay is performed in a closed system and either helium or nitrogen is used as the flush gas. Oil and water are condensed in a centrifuge tube and noncondensable gases flow into an evacuated receiver. Following the pyrolysis process, the products are weighed and further analyzed.

TABLE 71

Kerogen Composition
Sample from P-1 Well Located in
Sullivan County, Indiana

| Mound Sample No. | Weight Percent (Ash Free) C | H | N | O | Weight Percent Ash | Atomic H/C | Atomic O/C |
|---|---|---|---|---|---|---|---|
| P-1-1 | 61.9 | 5.7 | 4.1 | 28.3 | 21.6 | 1.11 | 0.34 |
| P-1-2 | 64.0 | 7.4 | 2.7 | 25.8 | 25.0 | 1.39 | 0.30 |
| P-1-3 | 75.1 | 7.8 | 1.5 | 15.6 | 15.1 | 1.25 | 0.16 |

TABLE 72

Kerogen Composition
Sample from VA-1 Well Located in
Wise County, Virginia

| Sample No. | Weight Percent (Ash Free) C | H | N | O | Weight Percent Ash | Atomic H/C | Atomic O/C |
|---|---|---|---|---|---|---|---|
| VA-1-2 | 66.0 | 3.8 | 1.3 | 28.8 | 21.1 | 0.69 | 0.33 |
| VA-1-4 | 78.9 | 4.1 | 1.6 | 15.3 | 15.4 | 0.62 | 0.15 |
| VA-1-11 | 85.5 | 4.0 | 2.4 | 8.1 | 9.3 | 0.56 | 0.07 |
| VA-1-15 | 85.9 | 4.0 | 1.2 | 8.9 | 9.1 | 0.56 | 0.08 |
| VA-1-17 | 77.8 | 3.8 | 1.9 | 16.5 | 6.7 | 0.59 | 0.16 |
| VA-1-18 | 85.9 | 4.1 | 2.6 | 7.3 | 7.2 | 0.57 | 0.06 |
| VA-1-21 | 84.4 | 4.2 | 1.7 | 9.6 | 25.0 | 0.60 | 0.09 |
| VA-1-22 | 79.4 | 4.4 | 2.2 | 13.9 | 14.6 | 0.66 | 0.13 |
| VA-1-25 | 77.6 | 4.8 | 6.4 | 11.2 | 12.8 | 0.74 | 0.11 |
| VA-1-26 | 85.4 | 4.5 | 4.1 | 6.0 | 6.1 | 0.63 | 0.05 |
| VA-1-29 | 82.0 | 4.3 | 5.0 | 8.7 | 10.2 | 0.63 | 0.08 |
| VA-1-32 | 80.6 | 4.6 | 8.7 | 6.1 | 6.4 | 0.63 | 0.06 |

Table 73

Kerogen Composition
I-2 Well, Henderson County, Illinois

| Mound Sample No. | Weight Percent (Ash Free) C | H | N | O | Weight Percent Ash | Atomic H/C | Atomic O/C |
|---|---|---|---|---|---|---|---|
| I-2-1 | 67.6 | 6.6 | 6.4 | 19.4 | 27.8 | 1.17 | 0.22 |
| I-2-4 | 68.7 | 9.7 | 3.2 | 18.4 | 23.4 | 1.69 | 0.20 |
| I-2-6 | 68.8 | 7.5 | 1.1 | 22.7 | 11.8 | 1.31 | 0.25 |
| I-2-7 | 57.9 | 6.7 | 4.5 | 30.8 | 6.6 | 1.39 | 0.40 |
| I-2-10 | 64.0 | 11.1 | 2.7 | 22.2 | 12.1 | 2.08 | 0.26 |
| I-2-12 | 57.9 | 6.6 | 3.0 | 32.4 | 9.4 | 1.37 | 0.42 |
| I-2-14* | 28.0 | 4.6 | 4.1 | 63.3 | 43.2 | | |
| I-2-16 | 72.2 | 7.9 | 4.8 | 15.2 | 24.1 | 1.31 | 0.16 |
| I-2-18 | 51.9 | 5.9 | 2.3 | 40.0 | 7.4 | 1.46 | 0.58 |

*Data questionable because of high ash content.

TABLE 74

Kerogen Composition
I-3 Well, Taze well County, Illinois

| Sample No. | Weight Percent (Ash Free) C | H | N | O | Weight Percent Ash | Atomic H/C | Atomic O/C |
|---|---|---|---|---|---|---|---|
| I-3-1 | 72.3 | 5.6 | 1.8 | 20.3 | 14.9 | 0.93 | 0.21 |
| I-3-2 | 78.1 | 6.7 | 3.7 | 11.6 | 10.7 | 1.03 | 0.11 |
| I-3-4 | 75.3 | 5.9 | 3.3 | 15.5 | 13.9 | 0.94 | 0.15 |
| I-3-6 | 73.5 | 6.5 | 4.3 | 15.7 | 31.1 | 1.06 | 0.16 |
| I-3-7 | 72.2 | 7.3 | 4.0 | 16.5 | 13.7 | 1.21 | 0.17 |
| I-3-9 | 73.5 | 7.2 | 2.7 | 16.7 | 11.5 | 1.18 | 0.17 |
| I-3-11 | 53.2 | 5.5 | 1.7 | 39.6 | 7.3 | 1.24 | 0.56 |
| I-3-12 | 54.7 | 6.0 | 2.9 | 36.4 | 7.8 | 1.32 | 0.50 |
| I-3-14 | 58.0 | 6.2 | 2.6 | 33.3 | 6.1 | 1.28 | 0.43 |
| I-3-16 | 65.6 | 7.4 | 2.9 | 24.2 | 19.8 | 1.35 | 0.28 |
| I-3-17 | 59.7 | 6.4 | 1.6 | 32.3 | 19.5 | 1.29 | 0.41 |
| I-3-19 | 61.4 | 7.1 | 3.6 | 28.4 | 23.5 | 1.19 | 0.35 |

The analyses performed on the pyrolysis products include oil, gas and water yield; gas compositional analysis; detailed oil and water analysis; percent post-assay organic carbon; and percent ash. These studies are designed to correlate the geochemical analyses with pyrolysis yield.

The MBA product yeilds obtained to date are listed in Tables 75 through 89. The data in Table 75 show the same variation as observed in every other geochemical analysis on the KY-2 well samples. This variation is also observed in the data listed in Tables 81 and 87. Numerically averaged, however, the data for the Ky-2 well would indicate a pyrolysis oil yield of 1.39 gallons per ton of shale, a carbon transfer ratio of 26.62 and a hydrocarbon gas yield of 35.67 cubic feet per ton of shale. This gas is predominantly methane and ethane. The nonhydrocarbon gas was essentially bimodal distribution, either predominantly hydrogen sulfide or carbon dioxide or a uniform mixture of both.

The yield for the P-1 well averaged out to 4.06 gallons of oil per ton of shale and 73.35 cubic feet of hydrocarbon gas per ton of shale. The I-1 well yielded an average of 9.04 gallons of oil per ton of shale and a hydrocarbon gas yield of 159.7 cubic feet per ton. The oil and gas yields for the VA-1, I-2 and I-3 wells were low.

The nonhydrocarbon gas analyzed after the retorting of the samples from the O-1 and I-1 wells was predominantly hydrogen sulfide.

In general the material balance assay results are supportative of previously determined geochemical data.

One of the analysis not yet reported is the elemental composition of the pyrolysis oil. These data have not been reported because some discrepancies exist in the determination of a standard sample. Currently, a sample of oil analyzed by the National Bureau of Standards and Mound yielded results statistically different from those obtained by MERC. Samples of the oil are currently being analyzed by Mound using several different techniques to determine the influence of the analytical method on results. Personnel from our Design and Experimental group are evaluating the methods and results.

TABLE 75

Material Balance Assay Product Assay
for Martin County, Kentucky, Samples

| Mound 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 |
|---|---|---|---|---|---|---|---|---|
| Oil Yield (Gal./Ton) | | 2.70 | 3.79 | 0.15 | | 0.33 | 0.12 | 2.87 |
| Specific Gravity (60°/60°) | | 0.8840 | 0.8653 | 0.866 | | 0.866 | 0.866 | 0.8639 |
| °API-Oil Gravity | | 28.57 | 32.03 | 31.89 | | 31.89 | 31.89 | 32.29 |
| Water Yield (Gal./Ton) | | 4.41 | 2.60 | 5.04 | | 4.11 | 3.84 | 3.74 |
| pH | | 9.3 | 8.9 | 9.1 | | 9.0 | 9.0 | 9.2 |
| Hydrocarbon Gas Yield ($Ft^3$/Ton) | | 61.63 | 69.24 | 10.08 | | 32.36 | 1.22 | 65.90 |

*Sample being used for methane absorption study.

TABLE 75 (Cont'd.)

**Material Balance Assay Product Assay for Martin County, Kentucky, Samples**

| Mound Sample No. | KY-2-18 | KY-2-20 | KY-2-21 | KY-2-24 | KY-2-25 | KY-2-27 | KY-2-29 | KY-2-32 |
|---|---|---|---|---|---|---|---|---|
| Oil Yield (Gal./Ton) | 3.90 | 3.11 | 1.01 | 1.11 | 0.13 | 0.14 | 0.82 | 0.19 |
| Specific Gravity (60°/60°) | 0.8660 | 0.8692 | 0.866 | 0.866 | 0.866 | 0.866 | 0.8432 | 0.866 |
| °API-Oil Gravity | 31.89 | 31.29 | 31.89 | 31.89 | 31.89 | 31.89 | 36.31 | 31.89 |
| Water Yield (Gal./Ton) | 4.17 | 4.26 | 4.70 | 4.54 | 5.51 | 2.21 | 4.46 | 4.06 |
| pH | 9.1 | 9.4 | 9.3 | 9.0 | 9.0 | 9.0 | 9.3 | 9.2 |
| Hydrocarbon Gas Yield ($Ft^3$/Ton) | 83.46 | 66.19 | 26.13 | 27.23 | 5.75 | 2.31 | 55.69 | 10.05 |

*Sample being used for methane absorption study.

TABLE 75 (Cont'd.)

Material Balance Assay Product Assay
for Martin County, Kentucky, Samples

| Mound Sample No. | KY-2-34* | KY-2-35 | KY-2-38 | KY-2-42 | KY-2-44 | KY-2-45 | KY-2-47 | KY-2-48 |
|---|---|---|---|---|---|---|---|---|
| Oil Yield (Gal./Ton) | | 0.15 | 0.56 | 4.63 | 3.91 | 2.72 | 0.25 | 2.43 |
| Specific Gravity (60°/60°) | | 0.866 | 0.866 | 0.8760 | 0.866 | 0.866 | 0.866 | 0.8524 |
| °API-Oil Gravity | | 31.89 | 31.89 | 30.03 | 31.89 | 31.89 | 31.89 | 34.50 |
| Water Yield (Gal./Ton) | | 5.14 | 3.44 | 3.78 | 3.73 | 4.70 | 4.29 | 3.58 |
| pH | | 9.0 | 9.1 | 9.2 | 9.4 | 9.0 | 9.2 | 9.1 |
| Hydrocarbon Gas Yield ($Ft^3$/Ton) | | 5.46 | 13.51 | 97.49 | 91.49 | 105.83 | 3.66 | 64.01 |

*Sample being used for methane absorption study.

TABLE 75 (Cont'd.)

Material Balance Assay Product Assay
for Martin County, Kentucky, Samples

| Mound Sample No. | KY-2-50 | KY-2-51* | KY-2-54 | KY-2-55 | KY-2-58 | KY-2-60 | KY-2-62 | KY-2-63* | KY-2-66 |
|---|---|---|---|---|---|---|---|---|---|
| Oil Yield (Gal./Ton) | 0.07 | | 1.34 | 0.24 | 0.16 | 0.19 | 0.36 | | 1.40 |
| Specific Gravity (60°/60°) | 0.866 | | 0.866 | 0.866 | 0.866 | 0.866 | 0.866 | | 0.866 |
| °API-Oil Gravity | 31.89 | | 31.89 | 31.89 | 31.89 | 31.89 | 31.89 | | 31.89 |
| Water Yield (Gal./Ton) | 3.73 | | 5.98 | 6.54 | 4.51 | 4.88 | 5.07 | | 2.97 |
| pH | 9,1 | | 9.2 | 8.7 | 8.0 | 8.6 | 9.0 | | 9.2 |
| Hydrocarbon Gas Yield ($Ft^3$/Ton) | <0.74 | | 44.49 | 12.81 | <1.48 | <0.83 | 8.80 | | 30.78 |

*Sample being used for methane absorption study.

## TABLE 76

Material Balance Assay
Product Assay
for Sullivan County, Indiana, Samples

| Mound Sample No. | P-1-1 | P-1-3 |
|---|---|---|
| Oil Yield (Gal/Ton) | 0.29 | 7.82 |
| Specific Gravity (60°/60°) | -- | 0.896 |
| °API-Oil Gravity | -- | 26.4 |
| Water Yield (Gal/Ton) | 5.05 | 3.47 |
| pH | 9.0 | 9.1 |
| Hydrocarbon Gas Yield ($Ft^3$/Ton) | 1.48 | 145.22 |

TABLE 77

Material Balance Assay Product Assay for
Effingham County, Illinois, Samples

| Mound Sample No. | I-1-1 | I-1-4 | I-1-6 | I-1-8 | I-1-9 |
|---|---|---|---|---|---|
| Oil Yield (Gal/Ton) | 11.0 | 7.5 | 8.4 | 8.9 | 9.4 |
| Specific Gravity (60°/60°) | 0.9044 | 0.9050 | 0.9058 | 0.9058 | 0.9150 |
| °API-Oil Gravity | 24.96 | 24.85 | 24.72 | 24.72 | 23.14 |
| Water Yield (Gal/Ton) | 4.96 | 4.02 | 3.31 | 3.78 | 4.25 |
| pH | 9.4 | 9.2 | 9.1 | 9.1 | 9.1 |
| Hydrocarbon Gas Yield ($Ft^3$/Ton) | 207.37 | 111.58 | 129.11 | 158.86 | 191.57 |

TABLE 78

Material Balance Assay
Product Assay

| Mound Sample No. | VA-1-2 | VA-1-4 | VA-1-6 | VA-1-13 | VA-1-15 | VA-1-22 | VA-1-27 | VA-1-29 | VA-1-32 |
|---|---|---|---|---|---|---|---|---|---|
| Oil Yield (Gas/Ton) | <0.5 | <0.5 | <0.5 | <0.5 | <0.5 | <0.5 | <0.5 | <0.5 | <0.5 |
| Specific Gravity ($60^{o}/60^{o}$) | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| $^{o}$API-Oil Gravity | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Water Yield (Gal/Ton) | 3.0 | 2.4 | 3.6 | 3.5 | 3.2 | 1.8 | 1.6 | 1.7 | 2.0 |
| pH | 9.2 | 9.3 | 9.3 | 9.3 | 9.1 | 9.2 | 9.2 | 9.1 | 9.1 |
| Hydrocarbon Gas Yield ($Ft^3$/Ton) | 49.2 | 29.5 | 1.9 | 1.7 | 4.2 | 17.0 | <1.0 | 32.0 | 12.6 |

TABLE 79

Material Balance Assay
Product Assay

| Mound Sample No. | I-2-4 | I-2-6 | I-2-7 | I-2-10 | I-2-12 | I-2-14 | I-2-16 | I-2-18 | I-2-20 |
|---|---|---|---|---|---|---|---|---|---|
| Oil Yield (Gal/Ton) | <0.5 | <0.5 | <0.5 | 2.2 | 4.3 | <0.5 | 1.4 | 1.4 | <0.5 |
| Specific Gravity ($60^{o}/60^{o}$) | --- | --- | --- | 0.899 | 0.923 | --- | --- | --- | --- |
| $^{o}$API-Oil Gravity | --- | --- | --- | 25.9 | 21.8 | --- | --- | --- | --- |
| Water Yield (Gal/Ton) | 5.3 | 5.1 | 5.2 | 5.1 | 3.8 | 6.3 | 4.7 | 4.3 | 2.2 |
| pH | 9.2 | 9.0 | 9.2 | 9.4 | 9.1 | 9.2 | 9.4 | 9.3 | 9.2 |
| Hydrocarbon Gas Yield ($Ft^3$/Ton) | 1.3 | 2.3 | 8.9 | 59.4 | 74.6 | 1.0 | 24.7 | 23.5 | 1.6 |

TABLE 80

Material Balance Assay Product Assay
for Tazewell County, Illinois Samples

| Mound Sample No. | I-3-1 | I-3-2 | I-3-4 | I-3-6 | I-3-7 | I-3-9 | I-3-11 | I-3-12 | I-3-14 | I-3-16 | I-3-17 | I-3-19 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Oil Yield (Gal/Ton) | <0.50 | <0.50 | <0.50 | <0.50 | <0.50 | 0.96 | 0.96 | 0.96 | 3.84 | 5.27 | 4.79 | 2.88 |
| Water Yield (Gal/Ton) | 6.23 | 11.02 | 6.71 | 8.62 | 7.19 | 7.19 | 7.66 | 8.14 | 7.19 | 4.79 | 5.27 | 6.71 |
| pH | 9.2 | 8.9 | 8.9 | 8.9 | 8.9 | 8.9 | 8.9 | 8.9 | 8.9 | 8.9 | 8.9 | 8.9 |
| Hydrocarbon Gas Yield ($Ft^3$/Ton) | 0.42 | 0.70 | 0.83 | 3.46 | 6.66 | 5.57 | 42.79 | 37.09 | 91.42 | 82.32 | 76.27 | 37.83 |

TABLE 81

Material Balance Assay Carbon and Ash Data
for Martin County, Kentucky, Samples

| Mound 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 |
|---|---|---|---|---|---|---|---|---|
| Pre-Test: | | | | | | | | |
| Total Carbon (%) | | 3.77 | 4.09 | 0.71 | | 2.04 | 0.55 | 4.20 |
| Organic Carbon(%) | | 3.52 | 3.64 | 0.66 | | 1.91 | 0.25 | 3.98 |
| Post-Test: | | | | | | | | |
| Total Carbon (%) | | 2.64 | 2.69 | 0.71 | | 1.72 | 0.50 | 3.10 |
| Organic Carbon(%) | | 2.23 | 2.25 | 0.57 | | 1.48 | 0.23 | 2.72 |
| Carbon Transfer Ratio | | 36.65 | 38.19 | 13.64 | | 22.51 | 8.00 | 31.66 |
| Retorting Weight Loss(%) | | 3.78 | 3.42 | 1.51 | | 2.67 | 1.88 | 3.77 |
| Retorted Shale: | | | | | | | | |
| Ash (%) | | 94.47 | 95.40 | 96.07 | | 95.24 | 95.73 | 94.33 |
| Ignition Loss (%) | | 5.53 | 4.60 | 3.93 | | 4.76 | 4.27 | 5.67 |

*Sample being used for methane absorption study.

TABLE 81 (Cont'd.)

Material Balance Assay Carbon and Ash Data
for Martin County, Kentucky, Samples

| Mound Sample No. | KY-2-18 | KY-2-20 | KY-2-21 | KY-2-24 | KY-2-25 | KY-2-27 | KY-2-29 | KY-2-32 |
|---|---|---|---|---|---|---|---|---|
| Pre-Test: | | | | | | | | |
| Total Carbon (%) | 4.94 | 4.38 | 2.02 | 2.03 | 0.87 | 1.59 | 3.59 | 0.86 |
| Organic Carbon (%) | 4.72 | 4.06 | 1.70 | 1.90 | 0.55 | 0.27 | 3.30 | 0.85 |
| Post-Test: | | | | | | | | |
| Total Carbon (%) | 3.54 | 3.18 | 1.54 | 1.46 | 0.59 | 1.46 | 2.06 | 0.69 |
| Organic Carbon (%) | 3.19 | 2.49 | 1.18 | 1.25 | 0.49 | 0.22 | 1.99 | 0.63 |
| Carbon Transfer Ratio | 32.42 | 38.67 | 30.59 | 34.21 | 10.91 | 18.52 | 39.70 | 25.88 |
| Retorting Weight Loss (%) | 4.59 | 4.20 | 3.03 | 3.05 | 3.14 | 1.55 | 3.81 | 2.33 |
| Retorted Shale: | | | | | | | | |
| Ash (%) | 93.92 | 93.91 | 95.39 | 95.82 | 95.97 | 94.39 | 94.75 | 95.66 |
| Ignition Loss (%) | 6.08 | 6.09 | 4.61 | 4.18 | 4.03 | 5.61 | 5.25 | 4.34 |

TABLE 81 (Cont'd.)

Material Balance Assay Carbon and Ash Data
for Martin County, Kentucky, Samples

| Mound Sample No. | KY-2-34* | KY-2-35 | KY-2-38 | KY-2-42 | KY-2-44 | KY-2-45 | KY-2-47 | KY-2-48 |
|---|---|---|---|---|---|---|---|---|
| Pre-Test: | | | | | | | | |
| Total Carbon (%) | | 0.58 | 1.30 | 5.90 | 5.34 | 5.19 | 1.45 | 3.38 |
| Organic Carbon (%) | | 0.45 | 1.11 | 5.45 | 4.70 | 4.77 | 0.65 | 3.05 |
| Post-Test: | | | | | | | | |
| Total Carbon (%) | | 0.43 | 0.89 | 4.12 | 3.74 | 3.85 | 1.21 | 2.55 |
| Organic Carbon (%) | | 0.38 | 0.68 | 3.17 | 3.09 | 3.34 | 0.64 | 2.15 |
| Carbon Transfer Ratio | | 15.56 | 38.74 | 41.83 | 34.26 | 29.98 | 1.54 | 29.51 |
| Retorting Weight Loss (%) | | 3.03 | 2.34 | 4.68 | 4.48 | 4.20 | 2.69 | 3.59 |
| Retorted Shale: | | | | | | | | |
| Ash (%) | | 96.72 | 96.50 | 93.08 | 93.86 | 93.85 | 95.78 | 95.01 |
| Ignition Loss (%) | | 3.28 | 3.50 | 6.92 | 6.14 | 6.15 | 4.22 | 4.99 |

*Sample being used for methane absorption study.

UGR File #C051/78Q1
Oct.-Dec. 1977
Found Quality

TABLE 81 (Cont'd.)

**Material Balance Assay Carbon and Ash Data for Martin County, Kentucky, Samples**

| Mound Sample No. | KY-2-50 | KY-2-51* | KY-2-54 | KY-2-55 | KY-2-58 | KY-2-60 | KY-2-62 | KY-2-63* | KY-2-66 |
|---|---|---|---|---|---|---|---|---|---|
| Pre-Test: | | | | | | | | | |
| Total Carbon (%) | 0.99 | | 3.86 | 1.44 | 1.61 | 0.40 | 0.83 | | 3.11 |
| Organic Carbon(%) | 0.10 | | 3.14 | 1.17 | 0.12 | 0.16 | 0.64 | | 2.63 |
| Post-Test: | | | | | | | | | |
| Total Carbon (%) | 0.87 | | 3.12 | 1.07 | 0.36 | 0.21 | 0.63 | | 2.24 |
| Organic Carbon(%) | 0.07 | | 2.50 | 0.89 | 0.13 | 0.14 | 0.47 | | 1.78 |
| Carbon Transfer Ratio | 30.00 | | 20.38 | 23.93 | -- | 12.50 | 26.56 | | 32.32 |
| Retorting Weight Loss | 2.23 | | 3.99 | 3.07 | 6.03 | 2.46 | 2.66 | | 2.55 |
| Retorted Shale: | | | | | | | | | |
| Ash (%) | 94.54 | | 93.10 | 95.96 | 96.84 | 96.80 | 96.42 | | 94.93 |
| Ignition Loss(%) | 5.46 | | 6.90 | 4.04 | 3.16 | 3.20 | 3.58 | | 5.07 |

*Sample being used for methane absorption study.

TABLE 82

Material Balance Assay Carbon and Ash Data

| Mound Sample No. | P-1-1 | P-1-2 |
|---|---|---|
| Pre-Test: | | |
| Total Carbon (%) | 0.43 | 7.44 |
| Organic Carbon(%) | 0.29 | 7.23 |
| Post-Test: | | |
| Total Carbon (%) | 0.31 | 4.57 |
| Organic Carbon(%) | 0.32 | 4.37 |
| Carbon Transfer Ratio | --- | 39.56 |
| Retorted Shale: | | |
| Ash (%) | 96.32 | 92.80 |
| Ignition Loss(%) | 3.68 | 7.20 |

TABLE 83

Material Balance Assay Carbon and Ash Data
for Effingham County, Illinois, Samples

| Mound Sample No. | I-1-1 | I-1-4 | I-1-6 | I-1-8 | I-1-9 |
|---|---|---|---|---|---|
| Pre-Test: | | | | | |
| Total Carbon (%) | 9.27 | 6.74 | 7.50 | 7.38 | 8.21 |
| Organic Carbon (%) | 8.23 | 5.61 | 6.71 | 6.36 | 8.04 |
| Post-Test: | | | | | |
| Total Carbon (%) | 6.10 | 4.59 | 4.90 | 4.39 | 5.23 |
| Organic (%) | 4.87 | 3.19 | 3.84 | 3.75 | 4.31 |
| Carbon Transfer Ratio | 40.83 | 43.14 | 42.77 | 41.04 | 46.39 |
| Retorting Weight Loss (%) | 8.06 | 6.48 | 6.64 | 6.91 | 7.86 |
| Retorted Shale: | | | | | |
| Ash (%) | 91.27 | 93.50 | 93.62 | 92.28 | 92.27 |
| Ignition Loss (%) | 8.73 | 6.50 | 6.38 | 7.02 | 7.73 |

TABLE 84

Material Balance Assay Carbon and Ash Data
for Wise County, Virginia Samples

| Mound Sample No. | VA-1-2 | VA-1-4 | VA-1-6 | VA-1-13 | VA-1-15 | VA-1-22 | VA-1-27 | VA-1-29 | VA-1-32 |
|---|---|---|---|---|---|---|---|---|---|
| Pre-test: | | | | | | | | | |
| Total Carbon (%) | 5.68 | 3.90 | 0.36 | 0.61 | 2.12 | 4.30 | 0.50 | 6.14 | 2.77 |
| Organic Carbon (%) | 5.60 | 3.69 | 0.38 | 0.63 | 2.27 | 4.39 | 0.24 | 5.65 | 2.81 |
| Post-test: | | | | | | | | | |
| Total Carbon (%) | 5.41 | 3.61 | 0.36 | 0.61 | 2.04 | 4.10 | 0.35 | 5.45 | 2.56 |
| Organic Carbon (%) | 5.27 | 3.64 | 0.41 | 0.60 | 2.09 | 3.79 | 0.25 | 5.27 | 2.60 |
| Carbon Transfer Ratio | 5.89 | 1.36 | --- | 4.76 | 7.93 | 13.67 | --- | 6.73 | 7.47 |
| Retorting Weight Loss (%) | 2.24 | 1.79 | 1.73 | 1.84 | 1.72 | 1.79 | 1.84 | 2.02 | 1.53 |
| Retorted Shale: | | | | | | | | | |
| Ash (%) | 91.80 | 94.14 | 96.20 | 95.90 | 95.11 | 93.49 | 97.49 | 91.90 | 94.96 |
| Ignition Loss (%) | 8.20 | 5.86 | 3.80 | 4.10 | 4.89 | 6.51 | 2.51 | 8.10 | 5.04 |

TABLE 85

**Material Balance Assay**
**Carbon and Ash Data**

| Mound Sample No. | I-2-4 | I-2-6 | I-2-7 | I-2-10 | I-2-12 | I-2-14 | I-2-16 | I-2-18 | I-2-20 |
|---|---|---|---|---|---|---|---|---|---|
| Pre-Test: | | | | | | | | | |
| Total Carbon (%) | 2.12 | 2.57 | 2.49 | 3.83 | 3.66 | 0.27 | 1.89 | 2.37 | 6.01 |
| Organic Carbon(%) | 0.22 | 0.30 | 0.65 | 2.69 | 3.44 | 0.14 | 1.52 | 1.44 | 0.14 |
| Post-Test: | | | | | | | | | |
| Total Carbon (%) | 2.02 | 2.29 | 2.38 | 2.50 | 2.31 | 0.23 | 1.41 | 1.89 | 5.70 |
| Organic Carbon(%) | 0.22 | 0.30 | 0.55 | 1.60 | 1.88 | 0.19 | 1.05 | 0.85 | 0.15 |
| Carbon Transfer Ratio | 0 | 0 | 15.38 | 40.52 | 45.35 | --- | 30.92 | 40.97 | --- |
| Retorting Weight Loss(%) | 2.98 | 2.87 | 2.73 | 4.22 | 4.16 | 3.01 | 3.00 | 2.69 | 1.26 |
| Retorted Shale: | | | | | | | | | |
| Ash (%) | 91.92 | 91.41 | 93.13 | 94.06 | 94.76 | 97.04 | 95.38 | 93.80 | 91.37 |
| Ignition Loss(%) | 8.08 | 8.59 | 6.87 | 5.94 | 5.24 | 2.96 | 4.62 | 6.20 | 8.63 |

TABLE 86

Material Balance Assay Carbon Data
for Tazewell County, Illinois Samples

| Mound Sample No. | I-3-1 | I-3-2 | I-3-4 | I-3-6 | I-3-7 | I-3-9 | I-3-11 | I-3-12 | I-3-14 | I-3-16 | I-3-17 | I-3-19 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Pre-Test Organic Carbon(%) | 0.21 | 0.20 | 0.20 | 0.84 | 0.69 | 0.75 | 1.60 | 1.32 | 3.44 | 3.45 | 3.44 | 1.58 |
| Post-Test Organic Carbon(%) | 0.24 | 0.16 | 0.28;0.21R | 0.42 | 0.48 | 0.64 | 1.15 | 1.16 | 2.22 | 1.93;1.94R | 1.83 | 0.92 |
| Carbon Transfer Ratio | --- | 20.00 | --- | 50.00 | 30.43 | 14.67 | 28.13 | 12.12 | 35.47 | 43.91 | 46.80 | 41.77 |
| Retorting Weight Loss(%) | 2.58 | 2.99 | 2.92 | 3.05 | 3.33 | 3.47 | 4.27 | 4.47 | 5.61 | 5.24 | 5.35 | 3.73 |

TABLE 87

**Material Balance Assay Gaseous Yield for Martin County, Kentucky, Samples**

| Mound 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 |
|---|---|---|---|---|---|---|---|---|
| Hydrocarbons ($cm^3/g$) | | 1.919 | 2.157 | 0.314 | | 1.008 | 0.038 | 2.053 |
| Methane | | 1.092 | 1.253 | 0.202 | | 0.580 | 0.021 | 1.135 |
| Ethane | | 0.345 | 0.372 | 0.048 | | 0.165 | 0.003 | 0.350 |
| Ethylene | | 0.040 | 0.063 | 0.006 | | 0.006 | 0.003 | 0.052 |
| Propane | | 0.168 | 0.167 | 0.027 | | 0.088 | 0.002 | 0.177 |
| Propylene | | 0.071 | 0.104 | 0.015 | | 0.036 | 0.001 | 0.083 |
| i-Butane | | 0.019 | 0.012 | 0.002 | | 0.013 | 0.001 | 0.019 |
| n-Butane | | 0.095 | 0.082 | 0.002 | | 0.059 | 0.001 | 0.100 |
| Butene-1 | | 0.017 | 0.038 | 0.002 | | 0.015 | 0.001 | 0.035 |
| t-Butene-2 | | 0.012 | 0.015 | 0.002 | | 0.008 | 0.001 | 0.015 |
| c-Butene-2 | | 0.008 | 0.010 | 0.002 | | 0.004 | 0.001 | 0.010 |
| i-Pentane | | 0.017 | 0.012 | 0.002 | | 0.017 | 0.001 | 0.023 |
| n-Pentane | | 0.025 | 0.021 | 0.002 | | 0.015 | 0.001 | 0.042 |
| Pentene-1 | | 0.010 | 0.008 | 0.002 | | 0.002 | 0.001 | 0.012 |
| Non-Hydrocarbons ($cm^3/g$) | | | | | | | | |
| Hydrogen | | 0.936 | 1.356 | 0.357 | | 0.698 | 0.150 | 1.175 |
| Hydrogen Sulfide | | 1.898 | 1.182 | 0.305 | | 2.254 | 0.005 | 2.731 |
| Carbon Monoxide | | 0.010 | 0.056 | 0.006 | | 0.006 | 0.003 | 0.027 |
| Carbon Dioxide | | 0.669 | 1.464 | 0.172 | | 0.432 | 1.054 | 1.177 |

*Sample being used for methane absorption study.

TABLE 87 (Cont'd.)

**Material Balance Assay Gaseous Yield for Martin County, Kentucky, Samples**

| Mound Sample No. | KY-2-18 | KY-2-20 | KY-2-21 | KY-2-24 | KY-2-25 | KY-2-27 | KY-2-29 | KY-2-32 |
|---|---|---|---|---|---|---|---|---|
| Hydrocarbons ($cm^3/g$) | 2.600 | 2.062 | 0.814 | 0.850 | 0.179 | 0.072 | 1.735 | 0.313 |
| Methane | 1.395 | 1.127 | 0.428 | 0.441 | 0.119 | 0.036 | 0.924 | 0.179 |
| Ethane | 0.445 | 0.354 | 0.138 | 0.148 | 0.015 | 0.006 | 0.306 | 0.050 |
| Ethylene | 0.071 | 0.055 | 0.006 | 0.006 | 0.006 | 0.006 | 0.033 | 0.003 |
| Propane | 0.229 | 0.182 | 0.086 | 0.081 | 0.013 | 0.004 | 0.161 | 0.025 |
| Propylene | 0.116 | 0.083 | 0.033 | 0.035 | 0.010 | 0.004 | 0.058 | 0.017 |
| i-Butane | 0.029 | 0.023 | 0.019 | 0.019 | 0.002 | 0.002 | 0.031 | 0.005 |
| n-Butane | 0.132 | 0.102 | 0.035 | 0.050 | 0.002 | 0.002 | 0.097 | 0.010 |
| Butene-1 | 0.048 | 0.034 | 0.015 | 0.015 | 0.002 | 0.002 | 0.024 | 0.008 |
| t-Butene-2 | 0.019 | 0.015 | 0.008 | 0.008 | 0.002 | 0.002 | 0.012 | 0.003 |
| c-Butene-2 | 0.013 | 0.009 | 0.004 | 0.004 | 0.002 | 0.002 | 0.008 | 0.002 |
| i-Pentane | 0.029 | 0.025 | 0.021 | 0.021 | 0.002 | 0.002 | 0.030 | 0.006 |
| n-Pentane | 0.057 | 0.040 | 0.019 | 0.019 | 0.002 | 0.002 | 0.040 | 0.004 |
| Pentene-1 | 0.017 | 0.013 | 0.002 | 0.002 | 0.002 | 0.002 | 0.011 | 0.001 |
| Non-Hydrocarbons ($cm^3/g$) | | | | | | | | |
| Hydrogen | 1.513 | 1.216 | 0.370 | 0.381 | 0.344 | 0.287 | 0.970 | 0.151 |
| Hydrogen Sulfide | 3.609 | 3.060 | 1.247 | 1.631 | 0.055 | 0.010 | 3.541 | 0.467 |
| Carbon Monoxide | 0.057 | 0.045 | 0.006 | 0.006 | 0.006 | 0.006 | 0.025 | 0.003 |
| Carbon Dioxide | 1.521 | 1.792 | 1.318 | 1.046 | 3.146 | 2.701 | 1.018 | 1.768 |

UGR File #C051/ 8Q..
Oct.-Dec. 1977
Mound Facility

TABLE 87 (Cont'd.)

**Material Balance Assay Gaseous Yield for Martin County, Kentucky, Samples**

| Mound Sample No. | KY-2-34* | KY-2-35 | KY-2-38 | KY-2-42 | KY-2-44 | KY-2-45 | KY-2-47 | KY-2-48 |
|---|---|---|---|---|---|---|---|---|
| Hydrocarbons ($cm^3/g$) | | 0.170 | 0.421 | 3.037 | 2.850 | 3.297 | 0.114 | 1.994 |
| Methane | | 0.109 | 0.241 | 1.799 | 1.596 | 1.956 | 0.077 | 1.129 |
| Ethane | | 0.020 | 0.070 | 0.512 | 0.504 | 0.558 | 0.006 | 0.359 |
| Ethylene | | 0.003 | 0.003 | 0.091 | 0.074 | 0.066 | 0.003 | 0.032 |
| Propane | | 0.015 | 0.032 | 0.221 | 0.230 | 0.256 | 0.004 | 0.170 |
| Propylene | | 0.010 | 0.020 | 0.130 | 0.121 | 0.110 | 0.010 | 0.074 |
| i-Butane | | 0.001 | 0.007 | 0.023 | 0.025 | 0.031 | 0.001 | 0.023 |
| n-Butane | | 0.001 | 0.015 | 0.112 | 0.129 | 0.141 | 0.001 | 0.094 |
| Butene-1 | | 0.005 | 0.010 | 0.054 | 0.051 | 0.045 | 0.006 | 0.032 |
| t-Butene-2 | | 0.002 | 0.004 | 0.021 | 0.022 | 0.019 | 0.002 | 0.015 |
| c-Butene-2 | | 0.001 | 0.002 | 0.014 | 0.014 | 0.014 | 0.001 | 0.009 |
| i-Pentane | | 0.001 | 0.008 | 0.019 | 0.023 | 0.029 | 0.001 | 0.023 |
| n-Pentane | | 0.001 | 0.008 | 0.029 | 0.047 | 0.056 | 0.001 | 0.026 |
| Pentene-1 | | 0.001 | 0.001 | 0.012 | 0.014 | 0.016 | 0.001 | 0.008 |
| Non-Hydrocarbons ($cm^3/g$) | | | | | | | | |
| Hydrogen | | 0.151 | 0.152 | 1.551 | 1.667 | 1.696 | 0.152 | 1.056 |
| Hydrogen Sulfide | | 0.005 | 0.230 | 2.301 | 3.606 | 4.219 | 0.018 | 1.967 |
| Carbon Monoxide | | 0.003 | 0.003 | 0.076 | 0.064 | 0.054 | 0.003 | 0.034 |
| Carbon Dioxide | | 2.920 | 1.779 | 2.443 | 1.612 | 1.276 | 3.282 | 1.516 |

*Sample being used for methane absorption study.

TABLE 87 (Cont'd.)

**Material Balance Assay Gaseous Yield for Martin County, Kentucky, Samples**

| Mound Sample No. | KY-2-50 | KY-2-51* | KY-2-54 | KY-2-55 | KY-2-58 | KY-2-60 | KY-2-62 | KY-2-63* | KY-2-66 |
|---|---|---|---|---|---|---|---|---|---|
| Hydrocarbons ($cm^3/g$) | <0.023 | | 1.386 | 0.399 | <0.046 | <0.026 | 0.274 | | 0.959 |
| Methane | <0.005 | | 0.826 | 0.216 | <0.010 | 0.008 | 0.178 | | 0.553 |
| Ethane | <0.003 | | 0.225 | 0.070 | <0.006 | <0.003 | 0.044 | | 0.169 |
| Ethylene | <0.003 | | 0.006 | 0.003 | <0.006 | <0.003 | 0.003 | | 0.026 |
| Propane | <0.002 | | 0.107 | 0.036 | <0.004 | <0.002 | 0.017 | | 0.074 |
| Propylene | <0.002 | | 0.061 | 0.024 | <0.004 | <0.002 | 0.016 | | 0.044 |
| i-Butane | <0.001 | | 0.008 | 0.005 | <0.002 | <0.001 | 0.001 | | 0.007 |
| n-Butane | <0.001 | | 0.069 | 0.016 | <0.002 | <0.001 | 0.001 | | 0.043 |
| Butene-1 | <0.001 | | 0.027 | 0.009 | <0.002 | <0.001 | 0.007 | | 0.019 |
| t-Butene-2 | <0.001 | | 0.011 | 0.005 | <0.002 | <0.001 | 0.003 | | 0.007 |
| c-Butene-2 | <0.001 | | 0.008 | 0.003 | <0.002 | <0.001 | 0.001 | | 0.004 |
| i-Pentane | <0.001 | | 0.011 | 0.005 | <0.002 | <0.001 | 0.001 | | 0.007 |
| n-Pentane | <0.001 | | 0.019 | 0.006 | <0.002 | <0.001 | 0.001 | | 0.001 |
| Pentene-1 | <0.001 | | 0.008 | 0.001 | <0.002 | <0.001 | 0.001 | | 0.005 |
| Non-Hydrocarbons ($cm^3/g$) | | | | | | | | | |
| Hydrogen | 0.149 | | 0.786 | 0.150 | 0.937 | 0.151 | 0.162 | | 0.480 |
| Hydrogen Sulfide | 0.005 | | 2.536 | 0.146 | <0.010 | 0.005 | 0.028 | | 1.652 |
| Carbon Monoxide | 0.003 | | 0.006 | 0.003 | 0.107 | 0.003 | 0.003 | | 0.022 |
| Carbon Dioxide | 2.467 | | 1.931 | 0.531 | 15.870 | 2.025 | 1.979 | | 1.898 |

*Sample being used for methane absorption study.

TABLE 88

Material Balance Assay Gaseous Yield
for Effingham County, Illinois, Samples

| Mound Sample No. | I-1-1 | I-1-4 | I-1-6 | I-1-8 | I-1-9 |
|---|---|---|---|---|---|
| Hydrocarbons ($cm^3/g$) | 6.461 | 3.476 | 4.022 | 4.949 | 5.968 |
| Methane | 3.405 | 2.238 | 2.556 | 2.647 | 3.145 |
| Ethane | 1.181 | 0.072 | 0.083 | 0.868 | 1.040 |
| Ethylene | 0.184 | 0.108 | 0.149 | 0.135 | 0.158 |
| Propane | 0.597 | 0.361 | 0.425 | 0.437 | 0.564 |
| Propylene | 0.286 | 0.197 | 0.220 | 0.230 | 0.272 |
| i-Butane | 0.057 | 0.035 | 0.043 | 0.043 | 0.054 |
| n-Butane | 0.328 | 0.191 | 0.230 | 0.244 | 0.314 |
| Butene-1 | 0.118 | 0.083 | 0.093 | 0.098 | 0.122 |
| t-Butene-2 | 0.040 | 0.027 | 0.031 | 0.033 | 0.036 |
| c-Butene-2 | 0.026 | 0.017 | 0.021 | 0.021 | 0.024 |
| i-Pentane | 0.054 | 0.033 | 0.041 | 0.043 | 0.054 |
| n-Pentane | 0.142 | 0.087 | 0.101 | 0.113 | 0.141 |
| Pentene-1 | 0.043 | 0.027 | 0.029 | 0.037 | 0.044 |
| Non-Hydrocarbons ($cm^3/g$) | | | | | |
| Hydrogen | 4.845 | 2.022 | 2.396 | 3.306 | 3.074 |
| Hydrogen Sulfide | 5.069 | 3.590 | 4.401 | 3.987 | 4.326 |
| Carbon Monoxide | 0.139 | 0.135 | 0.151 | 0.160 | 0.177 |
| Carbon Dioxide | 0.314 | 3.097 | 2.614 | 1.949 | 2.175 |

TABLE 89

Material Balance Assay Gaseous Yield
for Christian County, Kentucky, Samples

| Mound Sample No. | 0-1-1 | 0-1-3 | 0-1-4 | 0-1-5 |
|---|---|---|---|---|
| Hydrocarbons ($cm^3/g$) | 9.126 | | 5.625 | 4.974 |
| Methane | 4.449 | 1.156 | 3.387 | 2.894 |
| Ethane | 1.697 | 0.746 | 0.987 | 0.889 |
| Ethylene | 0.325 | 0.138 | 0.153 | 0.147 |
| Propane | 1.007 | 0.195 | 0.403 | 0.373 |
| Propylene | 0.464 | 0.097 | 0.238 | 0.226 |
| i-Butane | 0.076 | 0.010 | 0.035 | 0.033 |
| n-Butane | 0.475 | 0.039 | 0.161 | 0.166 |
| Butene-1 | 0.207 | | 0.104 | 0.095 |
| t-Butene-2 | 0.060 | | 0.029 | 0.033 |
| c-Butene-2 | 0.041 | | 0.019 | 0.021 |
| i-Pentane | 0.074 | | 0.017 | 0.021 |
| n-Pentane | 0.191 | | 0.067 | 0.057 |
| Pentene-1 | 0.060 | | 0.025 | 0.019 |
| $C_5$-$C_7$ | | 0.078 | | |
| Non-Hydrocarbons ($cm^3/g$) | | | | |
| Hydrogen | 4.264 | | 1.518 | 2.459 |
| Hydrogen Sulfide | 9.092 | | 1.942 | 3.717 |
| Carbon Monoxide | 0.265 | | 0.071 | 0.064 |
| Carbon Dioxide | 1.836 | | 1.311 | 0.683 |

## H. PYROLYSIS-GAS CHROMATOGRAPHY

Analyses are also being performed on the bulk organic constituents of the shale. To date core samples from the KY-2, P-1, I-1, I-2 and I-3 have been completely analyzed by pyrolysis gas chromatography. Approximately 100 mg of powdered sample are heated under helium from ambient temperature to 550°C at a constant rate of 30°C/minute. The heating of the organic matter in the shale produces hydrocarbons which are swept by the carrier gas to a flame ionization detector. The amount of hydrocarbons generated versus the temperature of generation is displayed on a recorder. The areas under the curve are computed by a digital integrator.

Tables 90 through 94 list current results. The calculated percent value is a measure of convertability of organic matter to hydrocarbons upon pyrolysis. The Peak II Maximum Temperature is the temperature at which the maximum pyrolysis of solid organic matter occurs.

An evaluation of the data indicates the hydrocarbon yields of 58%, 47%, 36%, 36% and 35% for the I-1, P-1, KY-2, I-3 and I-2 wells, respectively. This means that more of the organic matter can be converted to hydrocarbons in the I-1 well and these results are consistent to those observed in the material balance assays.

The results to date would indicate that in order to obtain optimum hydrocarbon yields during gas production of these samples, a heating process will be required not only for extraction, but also for pyrolytic cracking.

## I. PULSED NMR

The main purpose of the pulsed nuclear magnetic resonance (NMR) study of Eastern Gas Shales is the development of a rapid, nondestructive, determination of their fuel content. This analytical method is base on the fact that the initial amplitude of the pulsed NMR free induction decay (FID) signal is directly proportional to the number of atoms of a particular element or isotope of an element in the sample.

Table 90

Pyrolysis - GC Analysis
KY-2 Well, Martin County, Kentucky

| Mound Sample No. | Organic Carbon (Wt. %) | Total Hydrocarbon Yield (WT. %) | Volatile Hydrocarbon Content (ppm) | $\frac{\text{Total Hydrocarbon Yield}}{\text{Organic Carbon}} \times 100$ | Peak II Maximum Temperature (°C) |
|---|---|---|---|---|---|
| KY-2-2 | 7.75 | 3.87 | 3391 | 49.94 | 500 |
| KY-2-4 | 3.21 | 1.40 | 1951 | 43.61 | 470 |
| KY-2-6 | 3.35;3.35R | 1.84 | 1880 | 54.93 | 470 |
| KY-2-8 | 0.62 | 0.11 | 316 | 17.74 | 464 |
| KY-2-9 | 0.16 | 0.03 | 161 | 18.75 | 464 |
| KY-2-11 | 1.58 | 0.34 | 825 | 21.52 | 470 |
| KY-2-13 | 0.09 | 0.39 | 233 | --- | 464 |
| KY-2-15 | 3.76;3.83R | 1.37 | 2037 | 36.05 | 472 |
| KY-2-18 | 4.28 | 1.99 | 2599 | 46.50 | 470 |
| KY-2-20 | 3.24 | 1.43 | 2005 | 44.14 | 470 |
| KY-2-21 | 1.30 | 0.59 | 1841 | 45.38 | 470 |
| KY-2-24 | 1.44 | 0.49 | 1250 | 34.03 | 452 |
| KY-2-25 | 0.48;0.46R | 0.13 | 593 | 27.66 | 464 |
| KY-2-27 | 0.18 | 0.07 | 354 | 38.89 | 470 |
| KY-2-29 | 2.53 | 1.04 | 2412 | 41.11 | 470 |
| KY-2-32 | 0.64 | 0.20 | 770 | 31.25 | 484 |
| KY-2-34 | 4.65;4.64R | 1.11 | 2335 | 23.90 | 458 |
| KY-2-35 | 0.33 | 0.13 | 685 | 39.39 | 464 |
| KY-2-38 | 0.70;0.69R | 0.25 | 840 | 35.97 | 474 |
| KY-2-40 | 2.30 | 1.35 | 1596 | 58.70 | 470 |
| KY-2-42 | 4.29 | 2.06 | 2832 | 48.02 | 474 |
| KY-2-44 | 3.42 | 2.07 | 3154 | 60.53 | 464 |
| KY-2-45 | 4.60 | 1.74 | 2372 | 37.83 | 470 |
| KY-2-47 | 0.40 | 0.11 | 92 | 27.50 | 332 (?) |
| KY-2-50 | 0.12;0.12R | 0.03 | 58 | 25.00 | 350 (?) |
| KY-2-51 | 1.88 | 0.62 | 1268 | 32.98 | 473 |
| KY-2-54 | 3.57 | 0.61 | 908 | 17.09 | 464 |
| KY-2-55 | 0.25;0.25R | 0.31 | 479 | --- | 470 |
| KY-2-58 | 0.15 | 0.06 | 220 | 40.00 | 468 |
| KY-2-60 | 0.14 | 0.04 | 121 | 28.57 | 335 (?) |
| KY-2-62 | 0.55;0.55R | 0.15 | 508 | 27.27 | 452 |
| KY-2-63 | 3.24 | 1.16 | 1882 | 35.80 | 467 |
| KY-2-66 | 2.10 | 0.70 | 1810 | 33.33 | 464 |

UGR File #C051/78Q1
Oct.-Dec. 1977
Mound Facility

TABLE 91

Pyrolysis - GC Analysis
P-1 Well, Sullivan County, Kentucky

| Mound Sample No. | Organic Carbon (Wt. %) | Total Hydrocarbon Yield (Wt. %) | Volatile Hydrocarbon Content (ppm) | $\frac{\text{Total Hydrocarbon Yield}}{\text{Organic Carbon}} \times 100$ | Peak II Maximum Temperature (°C) |
|---|---|---|---|---|---|
| P-1-1 | 0.25 | 0.06 | 382 | 24.00 | 455 |
| P-1-2 | 6.01 | 3.51 | 3624 | 58.40 | 464 |
| P-1-3 | 6.34;6.31R | 3.78 | 4239 | 59.76 | 470 |

TABLE 92

Pyrolysis - GC Analysis
I-1 Well, Effingham County, Illinois

| Mound Sample No. | Organic Carbon (Wt. %) | Total Hydrocarbon Yield (Wt. %) | Volatile Hydrocarbon Content (ppm) | $\frac{\text{Total Hydrocarbon Yield}}{\text{Organic Carbon}} \times 100$ | Peak II Maximum Temperature (°C) |
|---|---|---|---|---|---|
| I-1-1 | 8.37 | 4.90 | 3511 | 58.54 | 450 |
| I-1-4 | 5.50 | 3.42 | 2883 | 62.18 | 458 |
| I-1-6 | 6.30;6.30R | 3.41 | 3521 | 54.13 | 498 |
| I-1-8 | 6.36 | 3.88 | 2985 | 61.01 | 461 |
| I-1-9 | 7.32 | 3.98 | 2391 | 54.37 | 470 |

TABLE 93

**Pyrolysis - GC Analysis**
**I-2 Well, Henderson County, Illinois**

| Mound Sample No. | Organic Carbon (Wt. %) | Total Hydrocarbon Yield (Wt. %) | Volatile Hydrocarbon Content (ppm) | $\frac{\text{Total Hydrocarbon Yield}}{\text{Organic Carbon}} \times 100$ | Peak II Maximum Temperature (°C) |
|---|---|---|---|---|---|
| I-2-A | 0.55 | 0.12 | 463 | 21.8 | 461 |
| I-2-1 | 0.18 | 0.02 | 51 | 11.1 | 460 |
| I-2-4 | 0.37 | 0.04 | 159 | 10.8 | 463 |
| I-2-6 | 0.48 | 0.04 | 429 | 8.3 | 463 |
| I-2-7 | 0.52 | 0.18 | 319 | 34.6 | 471 |
| I-2-10 | 2.24 | 1.09 | 634 | 48.7 | 466 |
| I-2-12 | 3.29 | 2.06 | 1749 | 62.6 | 465 |
| I-2-14 | 0.21 | 0.09 | 601 | 42.9 | 463 |
| I-2-16 | 1.22 | 0.53 | 604 | 43.4 | 471 |
| I-2-18 | 1.07 | 0.50 | 453 | 46.7 | 473 |
| I-2-20 | 0.14 | 0.07 | 199 | 50.0 | 462 |
| I-2-B | 0.24 | 0.09 | 146 | 37.5 | 474 |

TABLE 94

Pyrolysis - GC Analysis
I-3 Well, Tazewell County, Illinois

| Mound Sample No. | Organic Carbon (Wt. %) | Total Hydrocarbon Yield (Wt. %) | Volatile Hydrocarbon Content (ppm) | $\frac{\text{Total Hydrocarbon Yield}}{\text{Organic Carbon}} \times 100$ | Peak II Maximum Temperature (°C) |
|---|---|---|---|---|---|
| I-3-A | 0.38 | 0.08 | 257 | 21.1 | 463 |
| I-3-1 | 0.21 | 0.06 | 275 | 28.6 | 463 |
| I-3-2 | 0.20 | 0.03 | 86 | 15.0 | 466 |
| I-3-4 | 0.20 | 0.03 | 95 | 15.0 | 468 |
| I-3-6 | 0.84 | 0.07 | 125 | 8.3 | 468 |
| I-3-7 | 0.69 | 0.08 | 97 | 11.6 | 473 |
| I-3-9 | 0.75 | 0.20 | 233 | 26.7 | 472 |
| I-3-11 | 1.60 | 0.75 | 559 | 46.9 | 471 |
| I-3-12 | 1.32 | 0.50 | 466 | 37.9 | 473 |
| I-3-14 | 3.44 | 1.95 | 816 | 56.7 | 474 |
| I-3-16 | 3.45 | 2.55 | 1606 | 73.9 | 470 |
| I-3-17 | 3.44 | 2.21 | 1519 | 64.2 | 478 |
| I-3-19 | 1.58 | 0.96 | 416 | 60.8 | 475 |

Proton pulsed NMR of shale yields the total hydrogen content of the shale. Any component of shale containing hydrogen will contribute to the free induction decay signal of proton pulsed NMR. Thus, hydrogen bonded to carbon atoms in the organic species, water hydrogen, and other inorganic hydrogen will contribute to the total NMR signal. Based on the assumption that for a given type of shale the water content is constant, then the pulsed NMR signal should be directly proportional to the oil yield or organic content of the shale. Oil yield is obtained along with total gas effluent directly from the Material Balance Assay of the shale. Organic carbon is obtained from wet chemistry and elemental analysis.

Correlations of Material Balance Assay oil yield, organic carbon content, and hydrocarbon gas yield with proton NMR spin-counts were performed for 75 samples from the KY-2, I-1, O-1, P-1, VA-1, R-109, OH-1 (Glen Gerry), and OH-2 (Logan County) wells.

The data (gal. of oil per ton, % by wt. organic carbon, and $cm^3$ of hydrocarbon gas per gram) are plotted as a function of proton NMR spin-counts per gram of shale for each well and collectively in Figures 8 through 10. The correlations are positive in the sense that the data and NMR spin-counts increase together. The correlations vary from good (corr. coeff 0.5-0.9) to excellent (>0.9).

Scatter of the data about the predicted line is mostly attributed to the variation in water content of the samples. To minimize this scatter, experiments are in progress to minimize the contribution of the water protons to the total spin-count. For example, shale samples are being ground to a fine powder and heated at low temperatures (100-150°C) to release water of crystallization and absorbed water. Losses of volatile organic shale constituents during the grinding and heating processes do not significantly alter the proton spin-count correlated with the fuel yield, which is mainly derived from the relatively thermostable kerogen component of the shale.

The NMR results have shown in a simple and rapid fashion that the water content of shales from different samples and wells is highly variable (indicators are the scatter of proton spin-counts about predicted correlation lines and the positive intercepts of the lines).

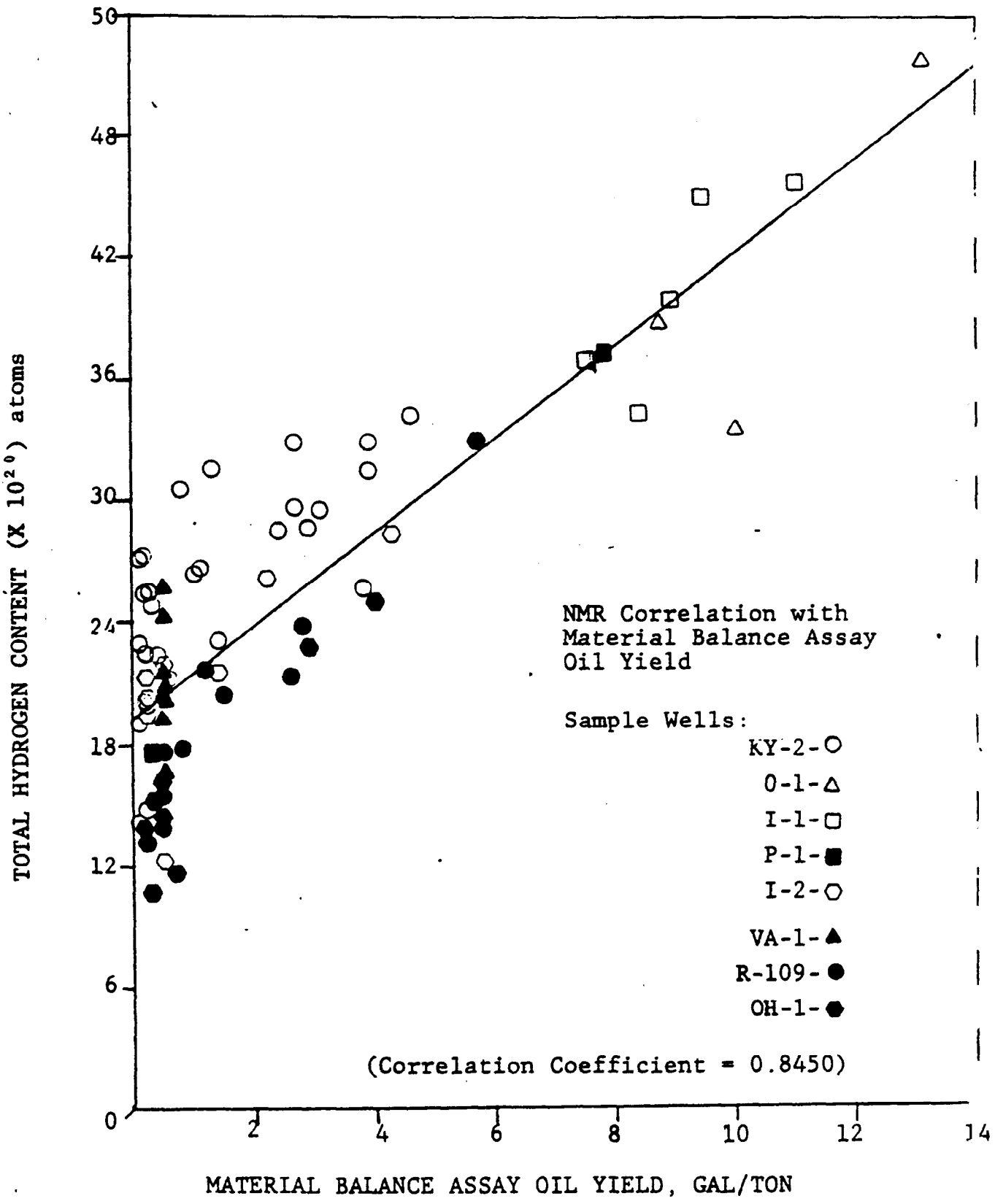

TOTAL HYDROGEN CONTENT (X 10²⁰) atoms
50
48
42
36
30
24
18
12
6
0
2
4
6
8
10
12
14
MATERIAL BALANCE ASSAY OIL YIELD, GAL/TON
NMR Correlation with
Material Balance Assay
Oil Yield
Sample Wells:
KY-2-○
O-1-△
I-1-□
P-1-■
I-2-○
VA-1-▲
R-109-●
OH-1-⬢
(Correlation Coefficient = 0.8450)

FIGURE 8

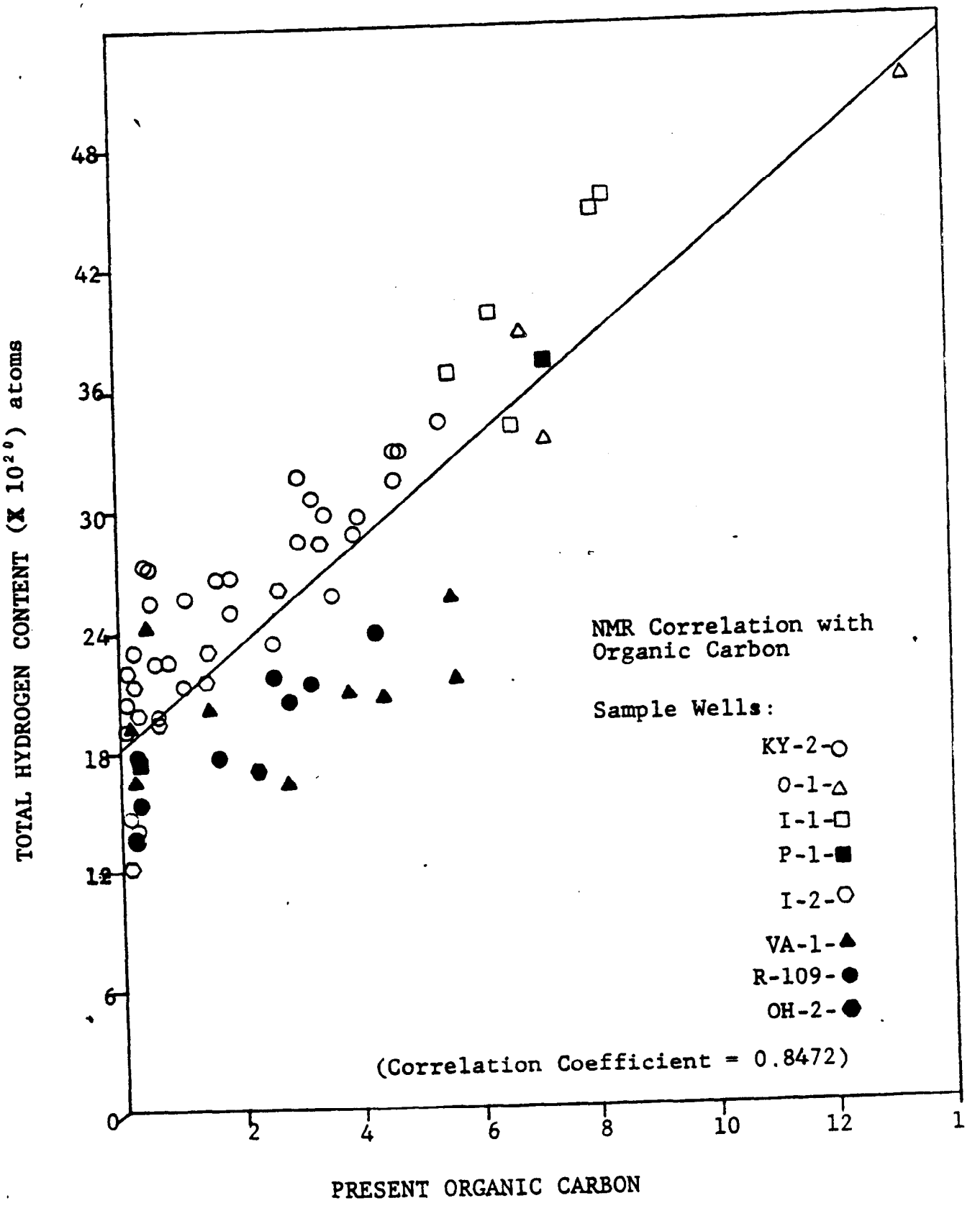

TOTAL HYDROGEN CONTENT (X 10²⁰) atoms
48
42
36
30
24
18
12
6
0
2
4
6
8
10
12
PRESENT ORGANIC CARBON
NMR Correlation with Organic Carbon
Sample Wells:
KY-2-○
O-1-△
I-1-□
P-1-■
I-2-○
VA-1-▲
R-109-●
OH-2-●
(Correlation Coefficient = 0.8472)

FIGURE 9

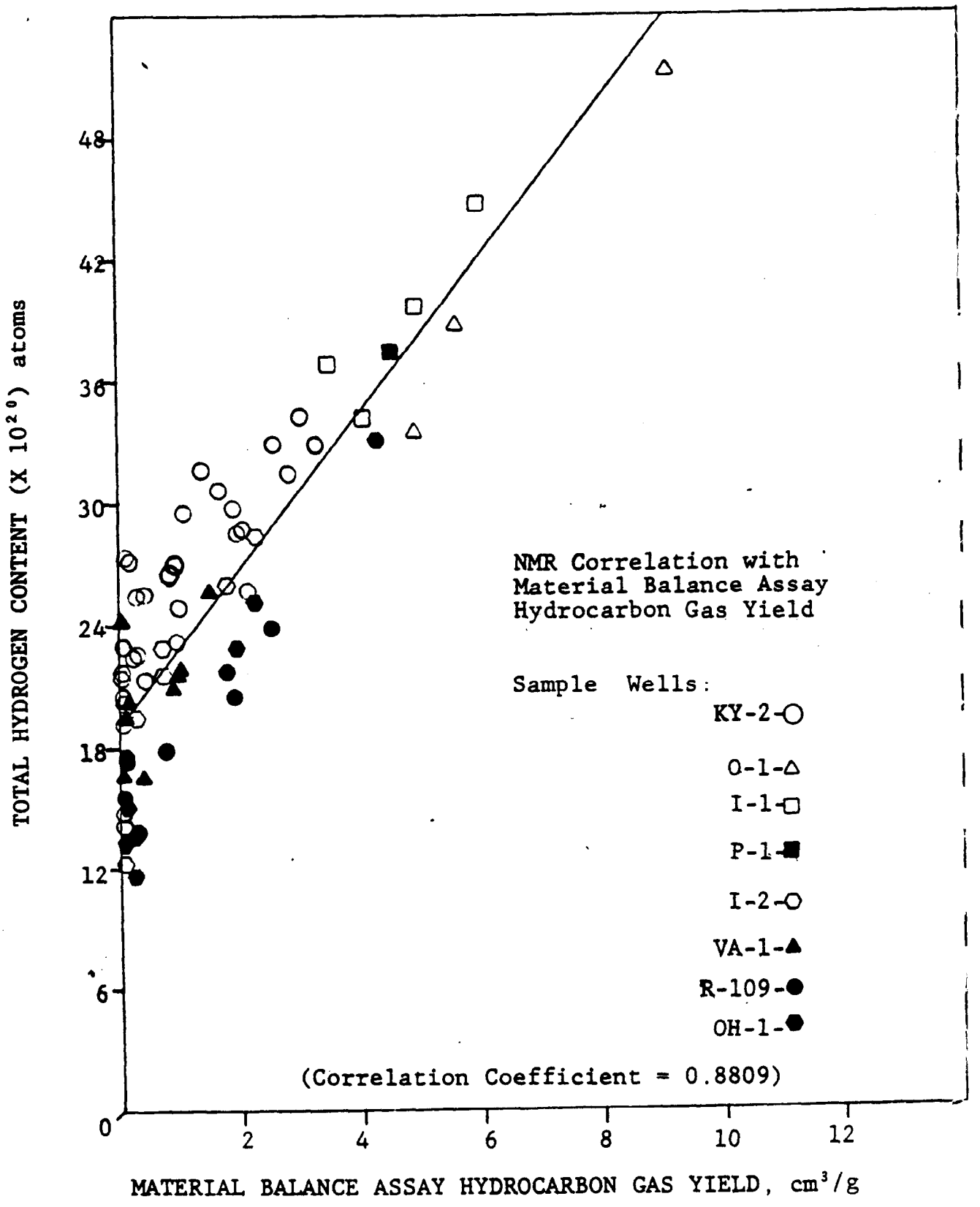

TOTAL HYDROGEN CONTENT (X 10²⁰) atoms
48
42
36
30
24
18
12
6
0
2
4
6
8
10
12
MATERIAL BALANCE ASSAY HYDROCARBON GAS YIELD, cm³/g
NMR Correlation with
Material Balance Assay
Hydrocarbon Gas Yield
Sample Wells:
KY-2-○
O-1-△
I-1-□
P-1-■
I-2-○
VA-1-▲
R-109-●
OH-1-⬢
(Correlation Coefficient = 0.8809)

FIGURE 10

The impact of the artifact of the end point scatter on the actual correlations of the lines is also being assessed. While systemmatic trends tend not to uncouple the correlation, large random errors will strongly decouple it.

The NMR results apparently are providing useful information and show promise for improving the degree of correlation for more accurate estimates of fuel-yield from NMR spin-counts for Devonian shales.

## J. DILATOMETRY

To determine the effects of fracturing and drilling fluids on the shale, test specimens are being core drilled from large samples using an air cooled diamond core drill. The specimens are restrained under load (∿112 psi) in the axial direction, and they are placed in intimate contact with the various fluids. The linear expansion is measured by means of an LVDT (Linear Variable Differential Transducer), which has been calibrated over the full range needed for these experiments.

The output signal from the LVDT is amplified and continuously recorded and converted to a digital signal, which can be used to monitor linear displacement as a function of time.

Typical results from this experiment are presented in Figures 11 through 13. As the results indicate, samples exposed to water exhibit the greatest expansion, and those exposed to kerosene exhibit the least amount of expansion. The unexpected result was that the shale exposed to Water and 30% Methanol and 2% KCℓ expanded more than the shale exposed to only Water and 2% KCℓ.

The basic clay composition of the shale is illite which adsorbs water when an imbalance occurs between the concentration of ions held at the clay surface and the solute content of the contacting liquid. The degree of hydration can theoretically be controlled by reducing the amount of water base and increasing the electrolyte content of the fluid. The fluid with the KCℓ addition should inhibit swelling by increasing the ionic level. The fluid with the KCℓ and Methanol additions should further inhibit swelling by slowing down water transfer. Since the KCℓ and Methanol additions have not inhibited swelling as much as the KCℓ addition alone additional studies are being performed in an attempt to identify this anomaly which has been consistent in all samples tested to date.

One of the first follow-up studies which was performed was an examination of the residue which settled out during testing. Figure 14 shows typical results from this investigation. Figure 14A is the normal secondary image of the

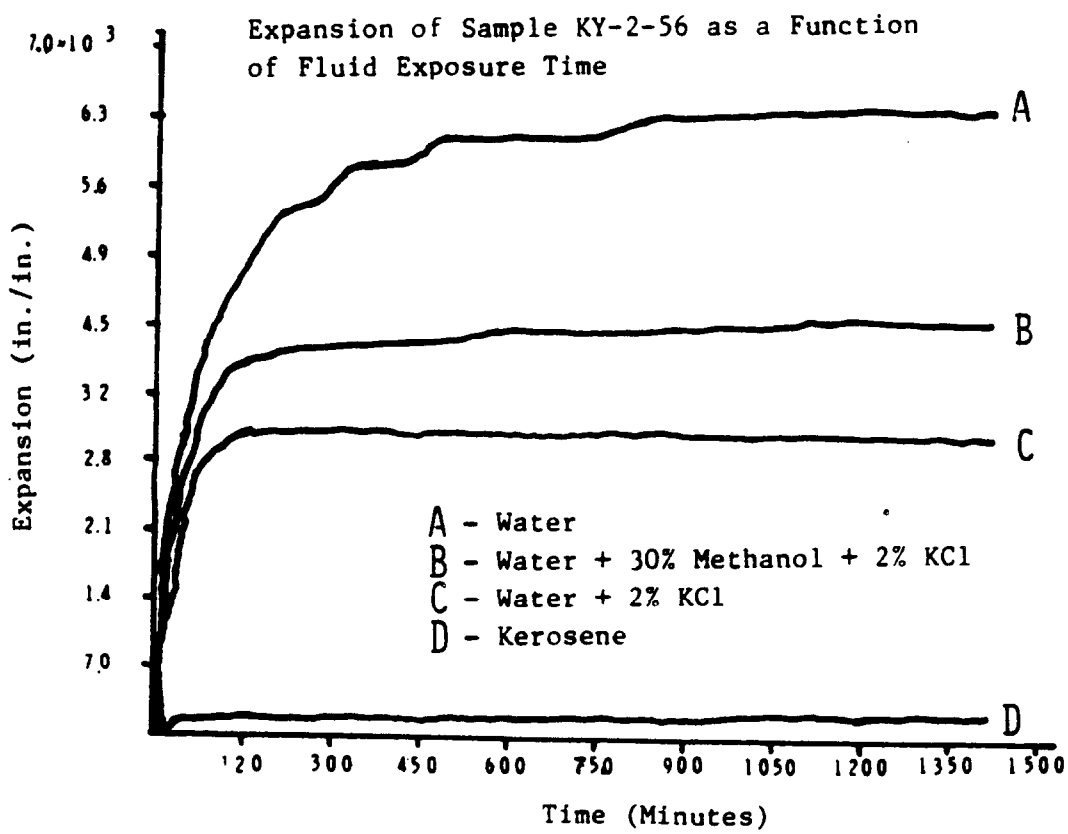

Expansion of Sample KY-2-56 as a Function of Fluid Exposure Time
7.0×10 3
6.3
5.6
4.9
4.5
3.2
2.8
2.1
1.4
7.0
Expansion (in./in.)
A
B
C
D
A - Water
B - Water + 30% Methanol + 2% KCl
C - Water + 2% KCl
D - Kerosene
120
300
450
600
750
900
1050
1200
1350
1500
Time (Minutes)

FIGURE 11

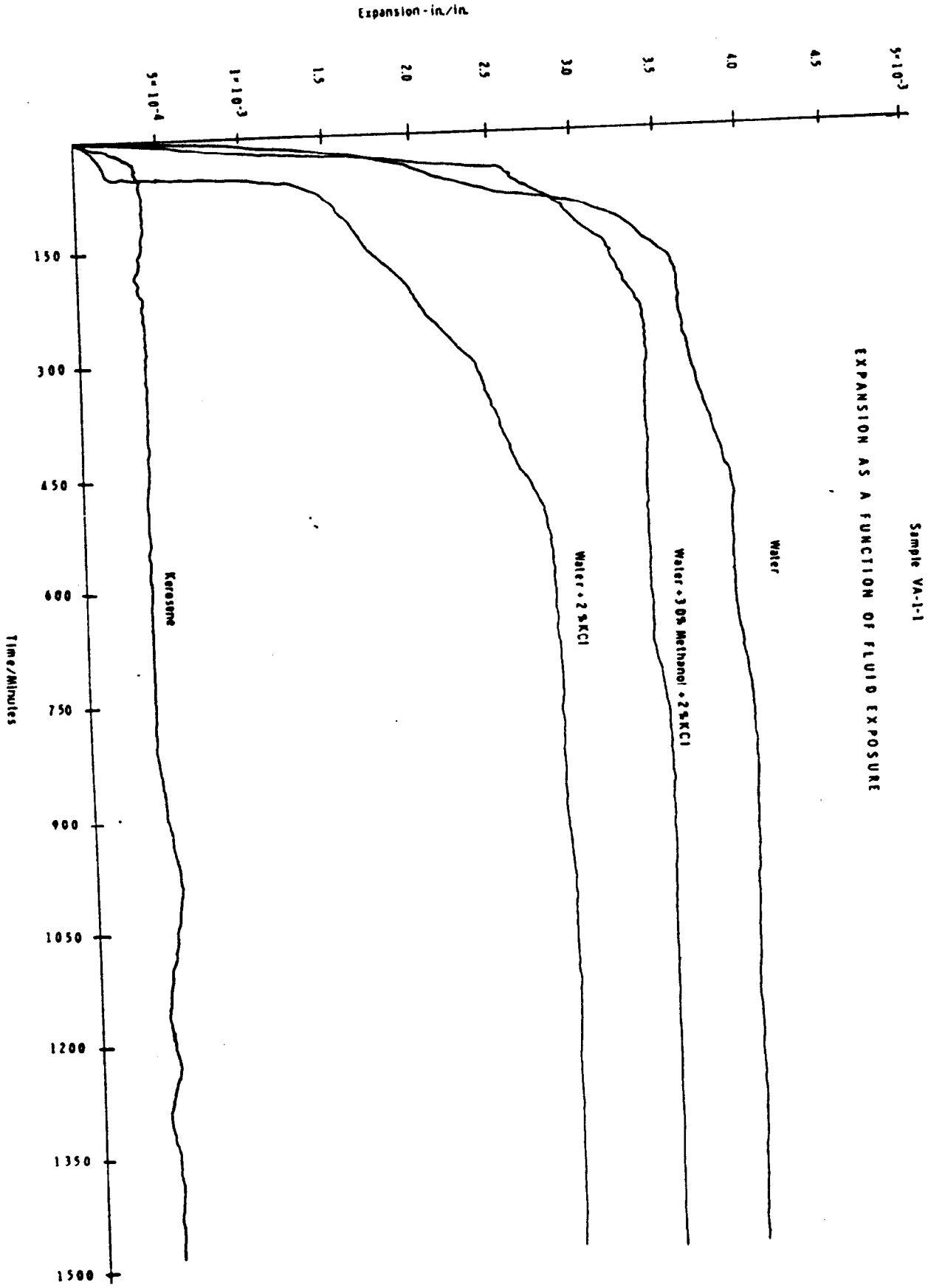

Sample VA-1-1
EXPANSION AS A FUNCTION OF FLUID EXPOSURE
Expansion - in./in.
5-10-3
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1-10-3
5-10-4
Time/Minutes
150
300
450
600
750
900
1050
1200
1350
1500
Water
Water + 30% Methanol + 2% KCl
Water + 2% KCl
Kerosene

FIGURE 12

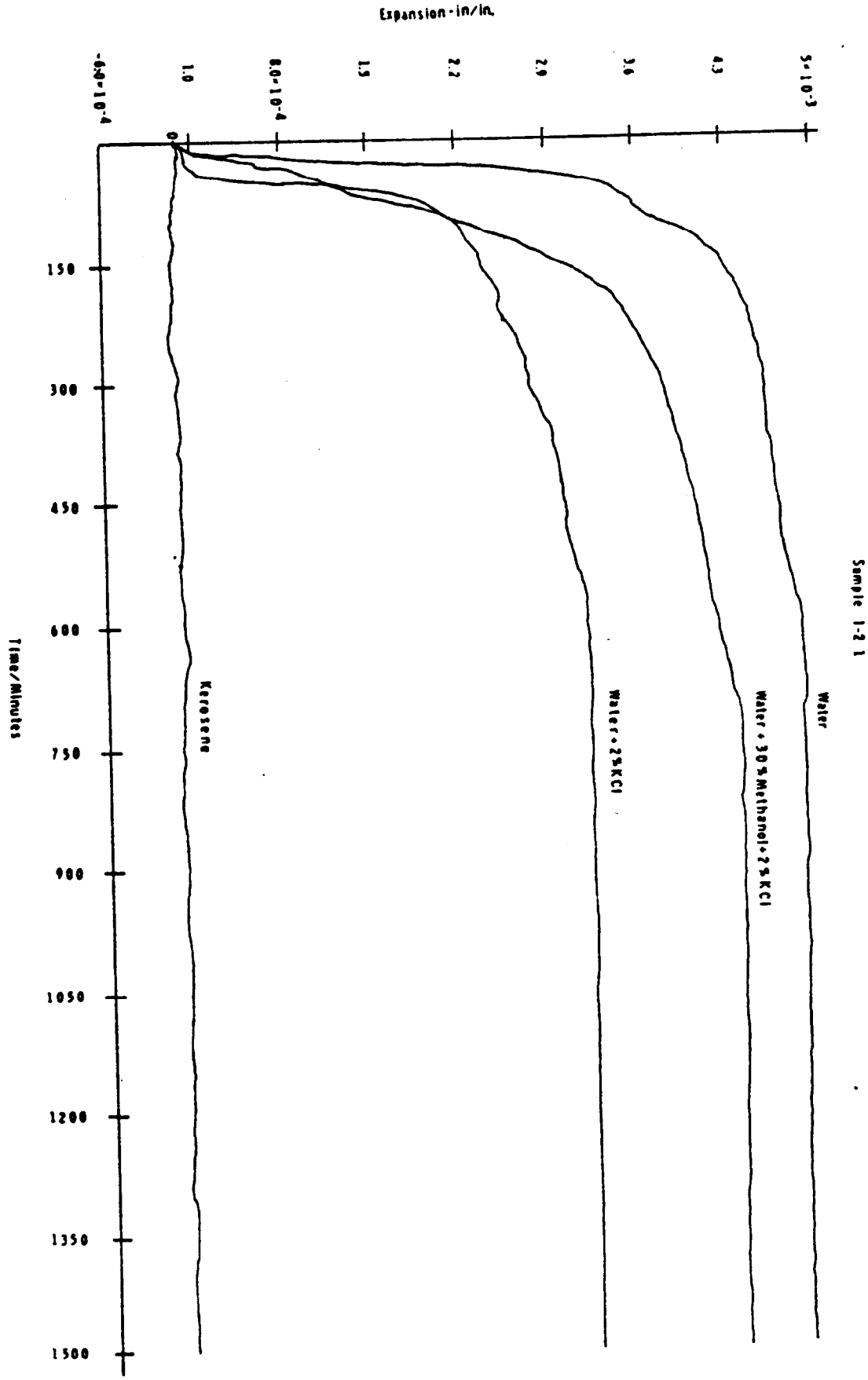
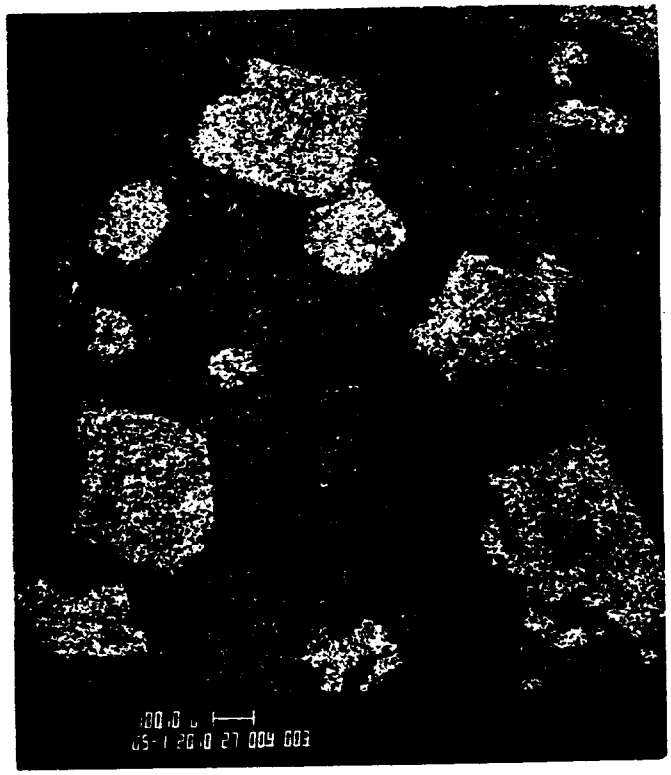
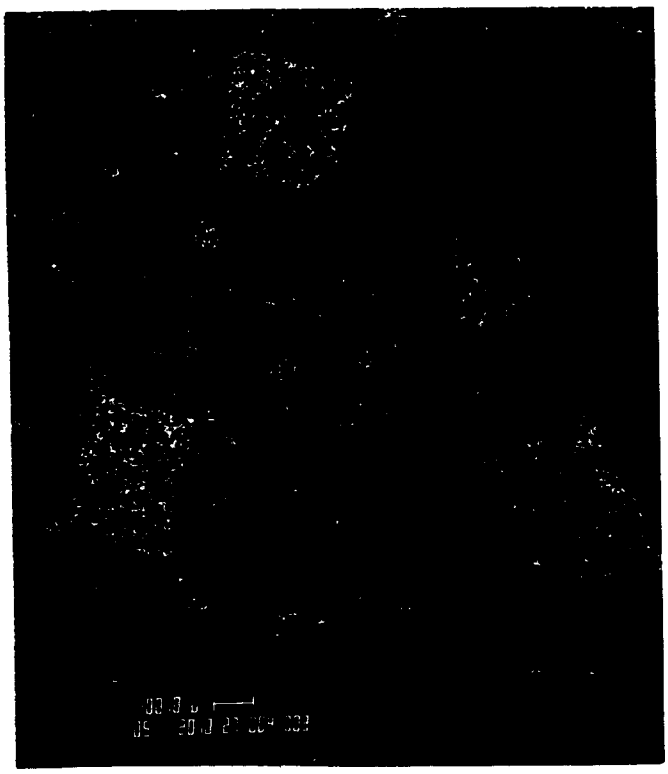

Sample 1-2 1
Water
Water + 30% Methanol + 2% KCl
Water + 2% KCl
Kerosene
Expansion - in/in.
5·10-3
4.3
3.6
2.9
2.2
1.5
8.0·10-4
1.0
0
-6.0·10-4
Time/Minutes
150
300
450
600
750
900
1050
1200
1350
1500

FIGURE 13

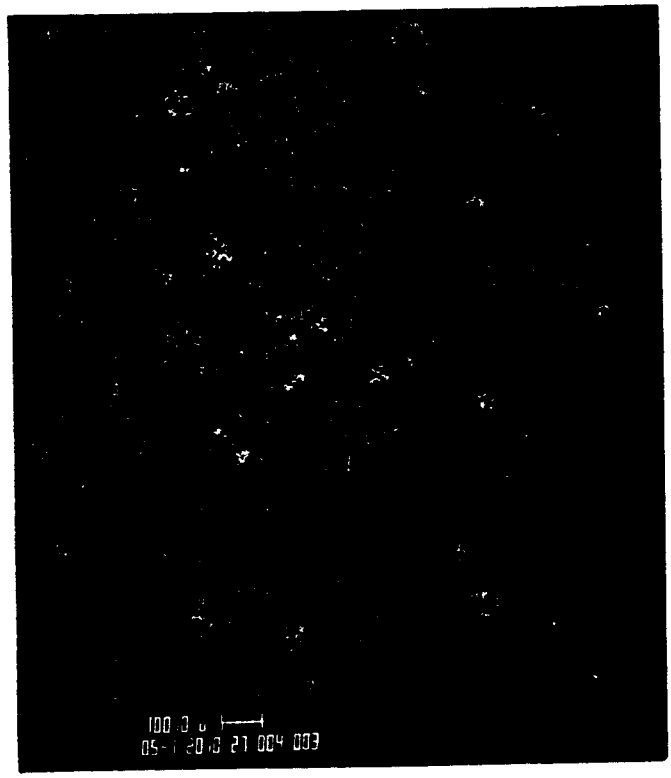
A



B



C



D

Residue from Water and Methanol and 2% KCℓ
Dilatometry Test



FIGURE 14

residue. Figure 14 B shows the x-ray map for potassium, and Figure 14C shows the x-ray map for chlorine. A comparison of these two maps shows some mismatches in concentration. When a x-ray map was generated for sulfur (Figure 14D) the pattern matched that of the chlorine mismatch, indicating a sulfur-chlorine combination, a corresponding x-ray map for iron explained the potassium mismatch. These results indicate that a possible reaction is occurring with the pyrite which is present in the sample and perhaps this reaction affects the swelling characteristics of the shale. This type of elemental combination was not observed in the Water and 2% KCℓ residue. This has been the first post-test difference noted between the samples, and it has been consistent in the samples examined to date.

## K. INTERACTIVE DATA MANAGEMENT AND ANALYSIS SYSTEM

To collate the voluminous data that will be generated during the term of the EGS Program, and to effectively use and efficiently distribute the data, an interactive data management and analysis system is required. Some attributes of such a system are described.

In conjunction with MERC, a prototype data management system (DMS) which includes the capabilities of data storage, retrieval, and processing was established for demonstration purposes. This interactive system contains some of Mound's Eastern Gas Shale data in a technical data base and is currently available for on-line demonstrations. Both on-line and in the batch mode, the system provided browsing and recursive search capabilities needed to scan large volumes of data and to conveniently present data for analysis and interpretability. In part, these features are provided through logical, boolean, and arithmetic expressions which allow the user to modify, rephrase or narrow his search criterion without restart, so that a redirected search need not include a retrace of previous paths. There is no practical restrictions on the size of the data base. The system allows users to manipulate their data with the basic system and then to interface user selected portions of the data with specialized routines linked with DMA to perform special calculations or manipulations. The system is easy to use because the entire two-way "conversation" between the user and the DMS is in English. (Mastery of a complex protocol language is not needed!) For the infrequent user, the system offers a practical convenience through an on-call instructional tutorial which is stylized to the user's preferences and background.

Often, at the inception of a major program, detailed specifications on format requirements and input media may not exist. Using the flexibility of the data system, input routines can be easily generated to convert data collected prior to completed specifications into selected DMS input formats. Although the system can interact with an array of input formats, all are processed through a common utility program. The system can be used to generate a variety of reporting formats, each tailored to specific report needs for various contractors or users. Consequently, the user is not required to adhere to a rigid format. In

a similar manner, accomodations can be made for unplanned or unforseen requirements with minimal economic impact. By use of the computational features of the system, many important sums, differences, and ratios can be provided for interpretative purposes. For example, the prototype system calculated total $C_1$-$C_4$, total $C_2$-$C_4$, % gas wetness ($C_1$-$C_4$/$C_2$-$C_4$), and the $iC_4/nC_4$ ratios. Furthermore, these computations also minimize data entry effort, both in a time and dollar sense. Other ratios and percents provided by the prototype are:

- 2-methylpentane/3-methylpentane
- isopentane/n-pentane
- cyclohexane/methylcyclopentane
- methylcyclopentane/methylcyclohexane
- % paraffin-naphthene (PN)
- % aromatic (A)
- % hydrocarbon
- PN/A

The latter four items are associated with the $C_{15}$+ Bitumen Extract. Collectively, the first four items provide insight into the maturity of the hydrocarbons.

Many of the data elements in the prototype Eastern Gas Shale data base require arithmetic manipulations. By use of arithmetic operators (greater than, less than, equal to, etc.), the DMS allows searching over ranges of data magnitudes. This arithmetic manipulation facilitates the interface of DMS with specialized output subroutines in higher level languages such as FORTRAN. Use of the FORTRAN Encode/Decode features is supported through use of the necessary FORTRAN compiler.

The statistical and modelling implications of the Eastern Gas Shale Program suggest the use of statistical packages such as SAS and BMD. These can be provided through the host language (e.g., FORTRAN) extension facility. Other mathematical packages that provide simple optimization, data filters, cluster analysis, trend analysis, and plotting capabilities can be provided without severe economic impact. All the mathematical resources can be shared with all the various contractors involved in the program, at least to the degree that they may choose. These statistical and mathematical software packages can be used to tie the characterization data with logging data.

As an example, an extension to the interpretative capabilities of the data is a system called GEOLOG. GEOLOG is a synergistic log system that will accurately integrate the log data on file from the A to D conversion and the geochemical data for the same well. This system ties together the well log and experimental data to provide a more accurate representation of the well.

These inputs will serve to produce an accurate synergistic log system that can be used in future applications with well log data.

The system equally supports project management and text files.

For project management files, a variety of search are available. Some key examples are:

- Contractor
- Contractor's Address
- Principal Investigator
- Associate Investigator
- Technique/Technology Used
- Technical Objectives
- Approach
- Status
- Budget Code
- Project Title
- Start Date
- Funding Agency
- Procurement Method
- Completed Milestones
- Contractor Performance

Through the arithmetic features, annualized budgets can be divided into a monthly allocation; spending rates can be computed, projected, and monitored for under or overrun; monthly expenditure values can be inserted; weekly, monthly, or yearly updates can be made; year-to-date information can be provided; milestones can be monitored; and variance flags can be set. Other provisions also are available for assessment of contractor performances in an objective manner.

A highly useful feature for a test or report file is the capability to search on a word without knowing the complete spelling. The user can specify the first three or four letters or characters and then fill out the word

with a default character such as an asterick. A predetermined dictionary is not necessary, because each work entered into the text file becomes part of the dictionary. Another convenience provided by the system is a display function that allows the key-word to be read in context with words highlighted on both sides of the key word. As a result key-word searches are more rapidly and accurately executed.

Remote data entry through cassettes, and/or cartridges, and remote terminals also is provided by the system. The data on a cassette or cartridge can be transmitted over a telephone line or mailed to an entry station. Entry also can be made via magnetic typewriter cards. This mode would be particularly useful for a report or text file.

In summary, a prototype data base system has been designed, tested, and implemented. The system provides the flexibility to meet changing and future needs that inevitably evolve with new programs. The system has already accommodated a request for format change that came after an initial format was fixed. It provides enhanced statistical capability for interpretation of characterization data through logical, boolean, and arithmetic searches that can be complemented by extensions to higher programming languages such as FORTRAN. These capabilities can be readily extended to text and project management files, including budgeting functions. Security keys are provided down to the word level. This interactive system contains some of Mound's Eastern Gas Shale data in a technical data base, both the technical data base and the project management base have been demonstrated in the interactive mode and are currently available for immediate implemtation in a multi-user mode.

## L. HYDROCARBON GAS PERMEATION IN DEVONIAN SHALES

In order to provide a meaningful evaluation of Devonian shale as a gas source for energy generation a study of the mobility of gases in the shale is necessary. A study of the permeation of gas through the shale will not only provide an accurate assessment of inherent gas flow properties of the shale but eventually should assist in identifying practical stimulation techniques.

This study, defined by a series of experimental tasks will characterize the permeation of hydrocarbon gases in shale. These tasks include the following:

1.) Anisotropic characterization of the permeation will be determined by taking samples parallel and perpendicular to the bedding planes.

2.) Flow rate data will be determined as a function of the pressure differential across the sample.

3.) The temperature of the shale will be varied in an effort to measure activation energies which can provide insight into the flow mechanisms and subsequently the stimulation techniques.

4.) Permeation of gas mixtures will be studied. The resulting data would also include data relating to sorption and effects of stimulating techniques.

5.) The permeation will be looked at on both the micro- and macro-scale. Large samples in principle should give data reflecting the overall permeation of the shale. However, small samples from properly sampled areas will be used for homogeneity checks and for detection of flaws in large samples.

6.) Effects of compressive stresses will be determined when the testing apparatus is upgraded to stimulate the in-situ hydrostatic compressive forces present in the shale.

7.) If the permeation mechanism is molecular, diffusion coefficients will be measured. Both time-lag and fast pump down methods will be used to determine diffusion.

The study also includes computational tasks. For example:

1.) Relationships between the shale structure and permeability will be investigated by comparing flow characteristics with SEM analysis of the structure. Regression and other correlation techniques will be used.

2.) Analysis of the permeation rates will be made to provide insight into the permeation mechanisms.

3.) Inhomogeneity variances will be computed.

The overall method that will be used to measure the permeability will be to pressurize one side of the shale with the gas of interest and monitor the concentration of the gas on the opposite side of the shale. Figures 15 and 16 show the initial test set-up. A cylindrical sample of shale will be placed in the fixture shown in Figure 15. The flat sides of the sample will be sealed against the fixture by two O-rings. After the sample is in the fixture a sleeve will be used in conjunction with plastic to seal the edges of the sample. The sample assembly will be positioned in the test apparatus shown in Figure 16. After the sample assembly is in place, both sides of the test apparatus will be evacuated. Purified gas will be supplied to the reservoir at a pressure measured by a capacitance monometer. After both sides of the sample have been evacuated the high pressure side of the sample will be pressurized with the gas from the reservoir while the vacuum is maintained on the low pressure side. The low pressure side of the sample will be continually monitored with a quadruple mass spectrometer (QMS) for the gas or gases present on the pressurized side of the sample. Permeation rate (s) will be determined by the increase in concentration of the gas or gases above the background levels (s). Calibration of the quadruple mass spectrometer will be accomplished by standard leaks.

The magnitude of the gas flow will determine how the vacuum is maintained on the low pressure side of the sample and in what manner the QMS will monitor the exit gas. If the resulting gas flow is too large for operations of the ion pump, the diffusion pump for the high pressure side of the sample will take over and the ion pump will back up the QMS which will monitor the emitted gas through capillary tubing.

The design of the initial test set-up has been completed and the present effort is concentrated on assembling the testing system.

To ensure continuity in the generation of a data base for this study, an effective measurement control program is necessary. A control program will be utilized to determine whether observed deviations are due to systematic or gross random effects. The spacing of measurements with respect to time, temperature, and other relevant parameters will be calculated from experimental design models. In this manner maximum information can be extracted from the experimental data, especially with data required to compute rate values and activation energies.

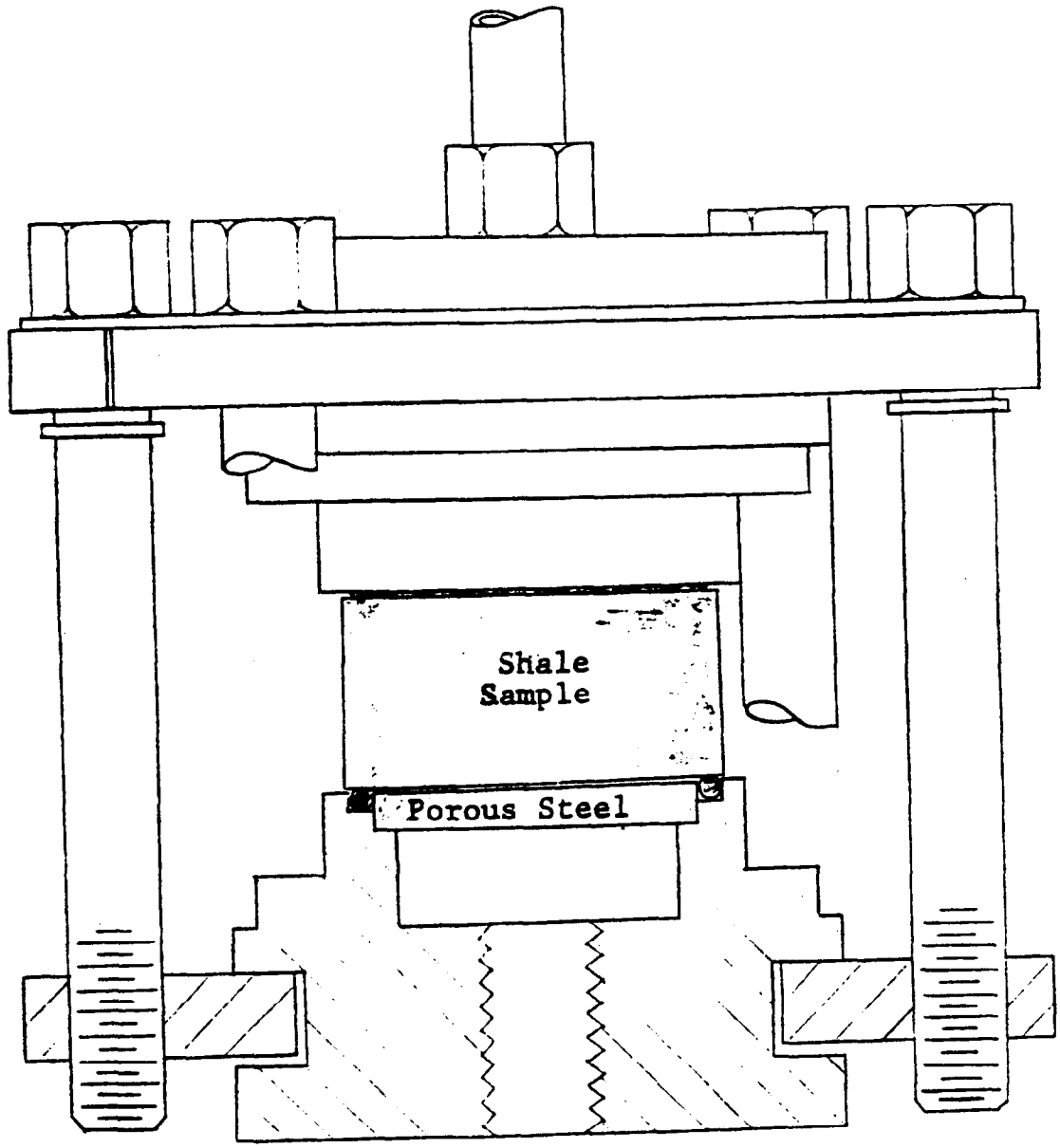



FIGURE 15

THE SAMPLE FIXTURE FOR THE GAS PERMEATION STUDY

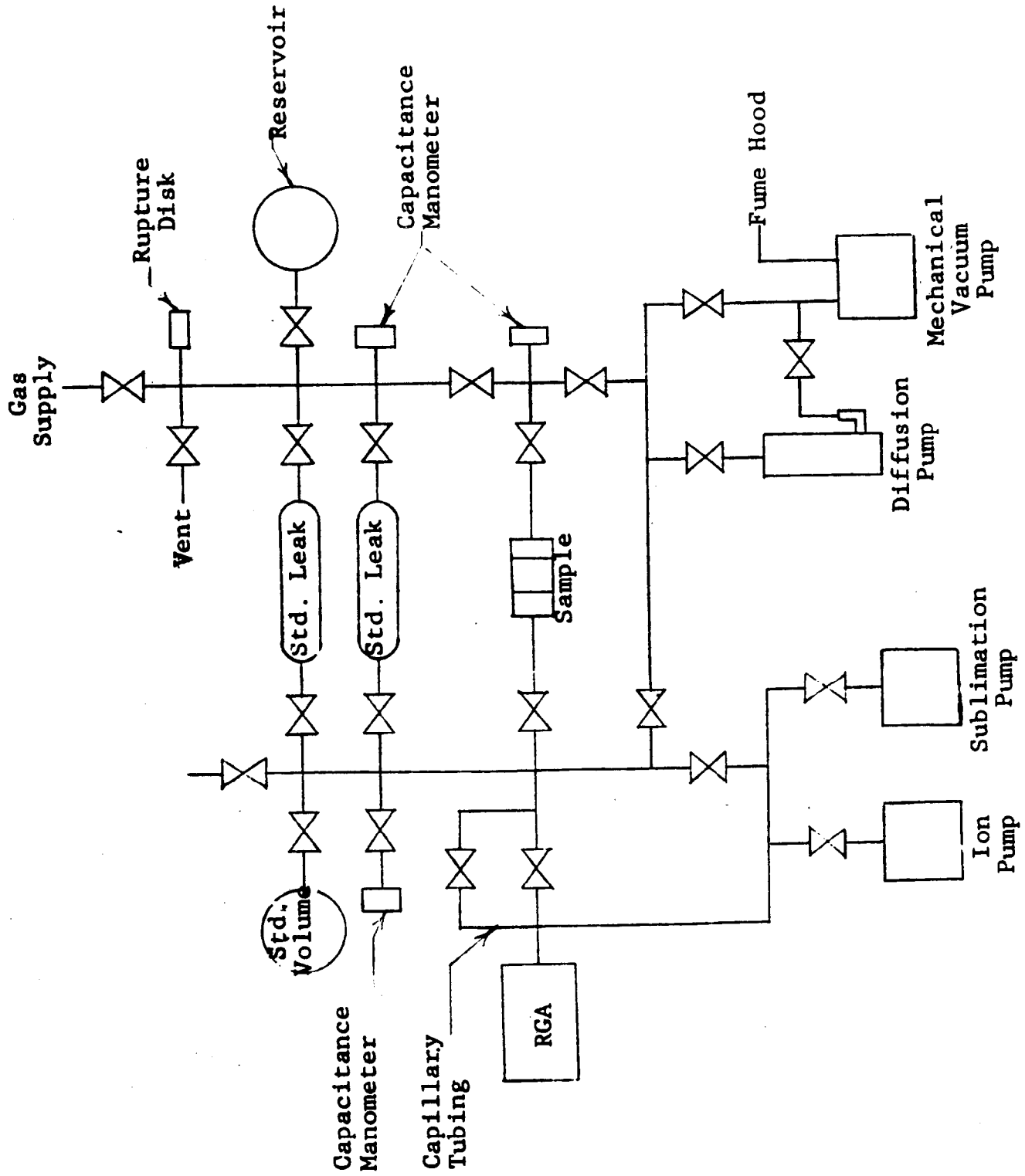



FIGURE 16

SCHEMATIC OF THE INITIAL EXPERIMENTAL CONFIGURATION FOR THE GAS PERMEATION STUDY.

## M. METHANE ABSORPTION IN DEVONIAN SHALES

A study has been initiated to determine methane absorption and desorption in Devonian shales. This study has been designed to determine the effects of pressure, organic carbon and oil yield on the absorption of methane by the shales.

The initial experiments will concentrate on samples from the KY-2 well. The details of these samples are listed in Table 95.

The experimental procedures that are being used in this study are essentially identical to those described by Matta et.al. (BM-RI8243). Four measuring stations have been assembled for the study. The general arrangement of the experimental apparatus is shown in Figure 17.

Measurements have been completed at 100 and 200 psig, and charging is in progress for the 300 psig run.

All data are considered preliminary. No corrections for barometric pressure variations or room temperature flucuations have been applied. The lost gas, that portion lost during the pressure release part of the measurement, has not yet been evaluated.

All corrections are expected to be small except the lost gas correction which will be significant. It is believed this factor will be best estimated when all measurements are completed.

The data for the 100 and 200 psig runs are plotted in Figures 18 through 21 for the four shale samples and a composite plot of gas released versus organic carbon content is given in Figure 22. The change in rate observed in Figures 18 through 21 is consistent. The reason for this change has not been established at this time.

Currently, the 300 psig runs are being completed. These runs will then be repeated and gas samples will be collected at different time intervals during the runs to evaluate the composition of the gas being released from the shale.

TABLE 95

Absorption Study Shale
Samples from the Ky-2 Well

| Mound Sample No. | Organic Carbon (Wt. %) | Sample Weight (Grams) | Sample Size |
|---|---|---|---|
| Ky-2-2 | 7.75 | 100.10 | 4-8 mesh |
| Ky-2-9 | 0.16 | 100.02 | 4-8 mesh |
| Ky-2-34 | 4.65 | 100.02 | 4-8 mesh |
| Ky-2-51 | 1.88 | 100.00 | 4-8 mesh |

FIGURE 17

Methane Absorption is determined using this typical experimental set up.

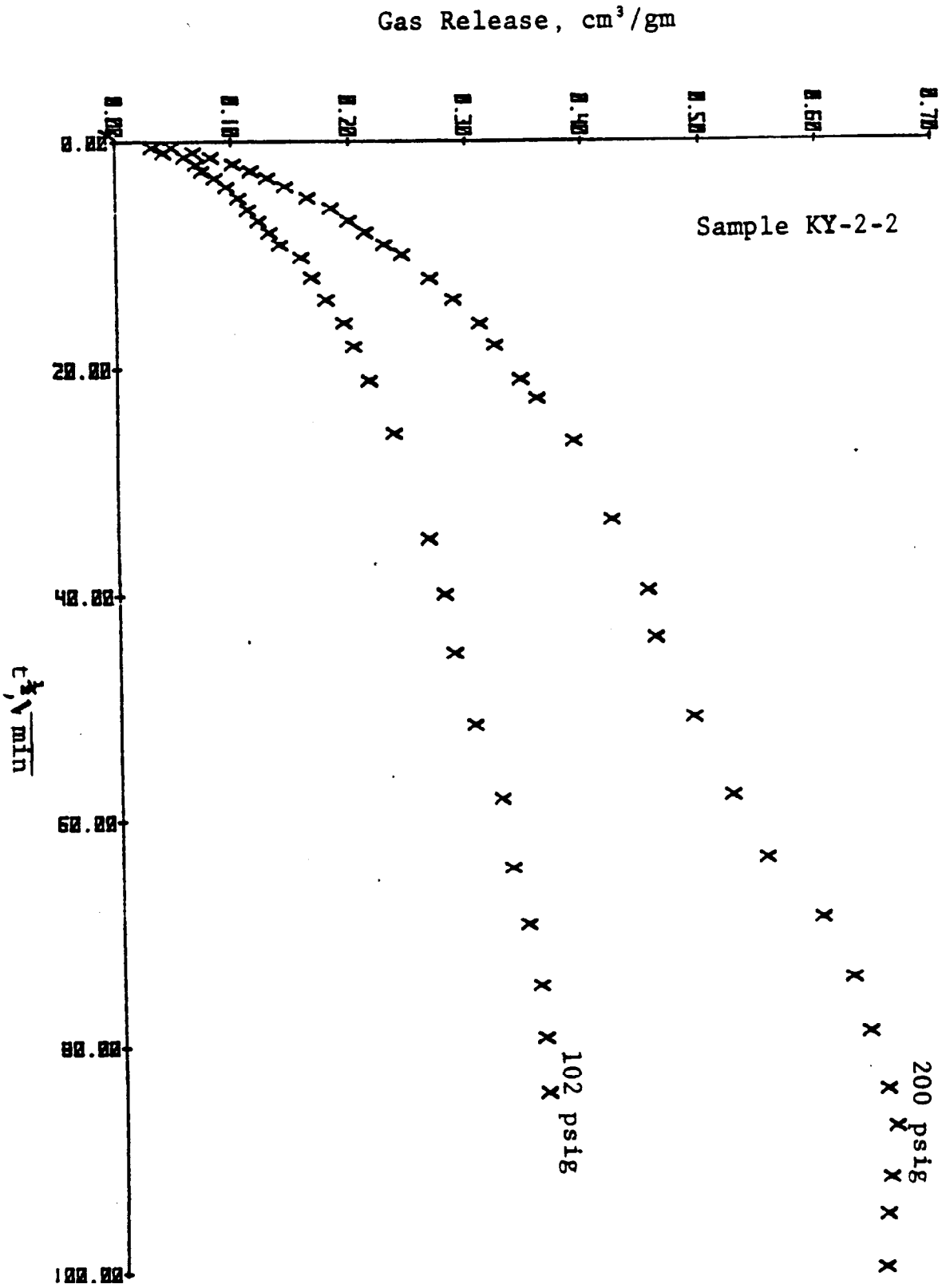

Gas Release, cm³/gm
0.00
0.10
0.20
0.30
0.40
0.50
0.60
0.70
Sample KY-2-2
200 psig
102 psig
0.00
20.00
40.00
60.00
80.00
100.00
t^½, √min

FIGURE 18

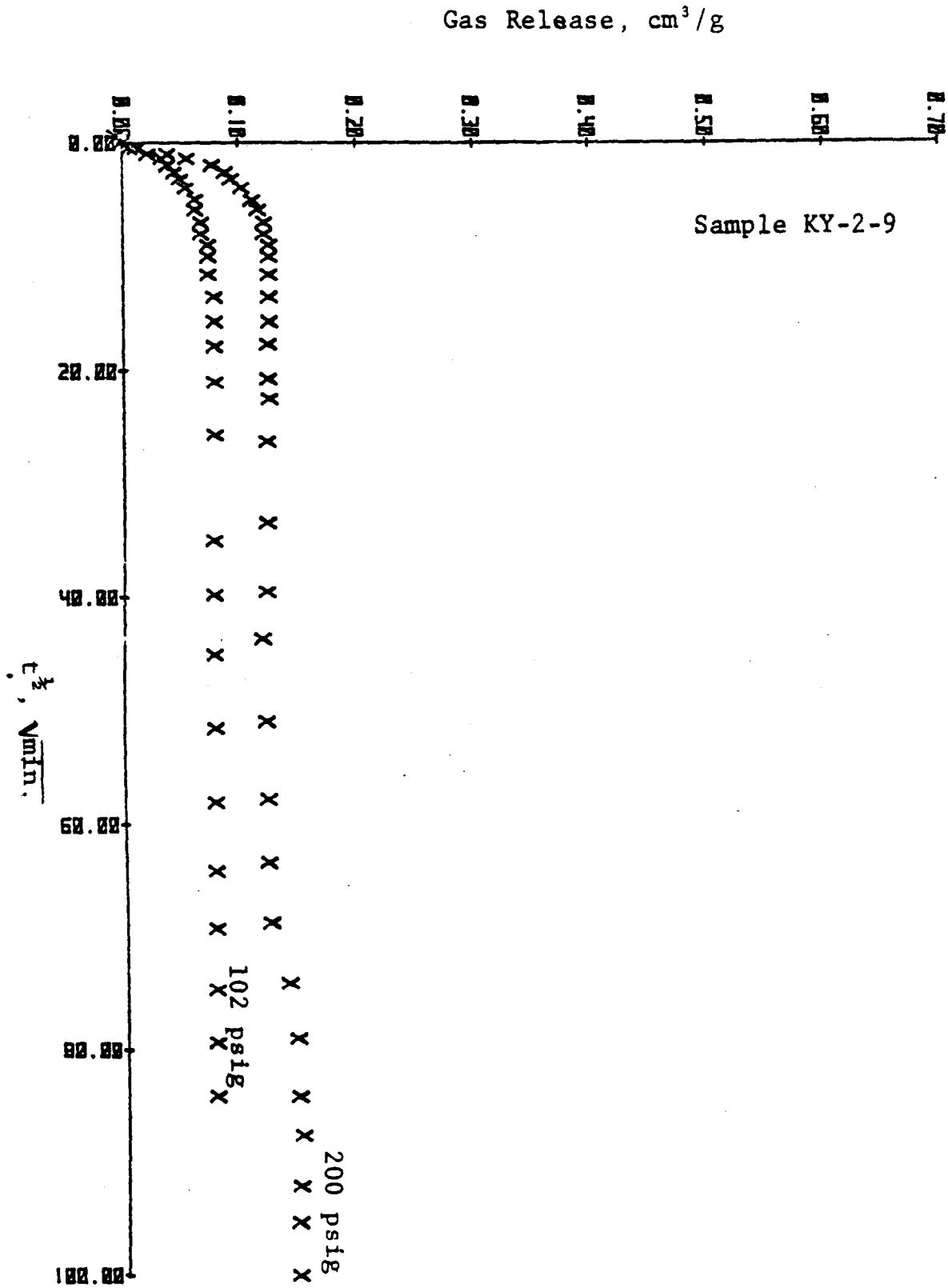

Gas Release, cm³/g
0.00
0.10
0.20
0.30
0.40
0.50
0.60
0.70
Sample KY-2-9
20.00
40.00
60.00
80.00
100.00
t^½, √min.
102 psig
200 psig

FIGURE 19

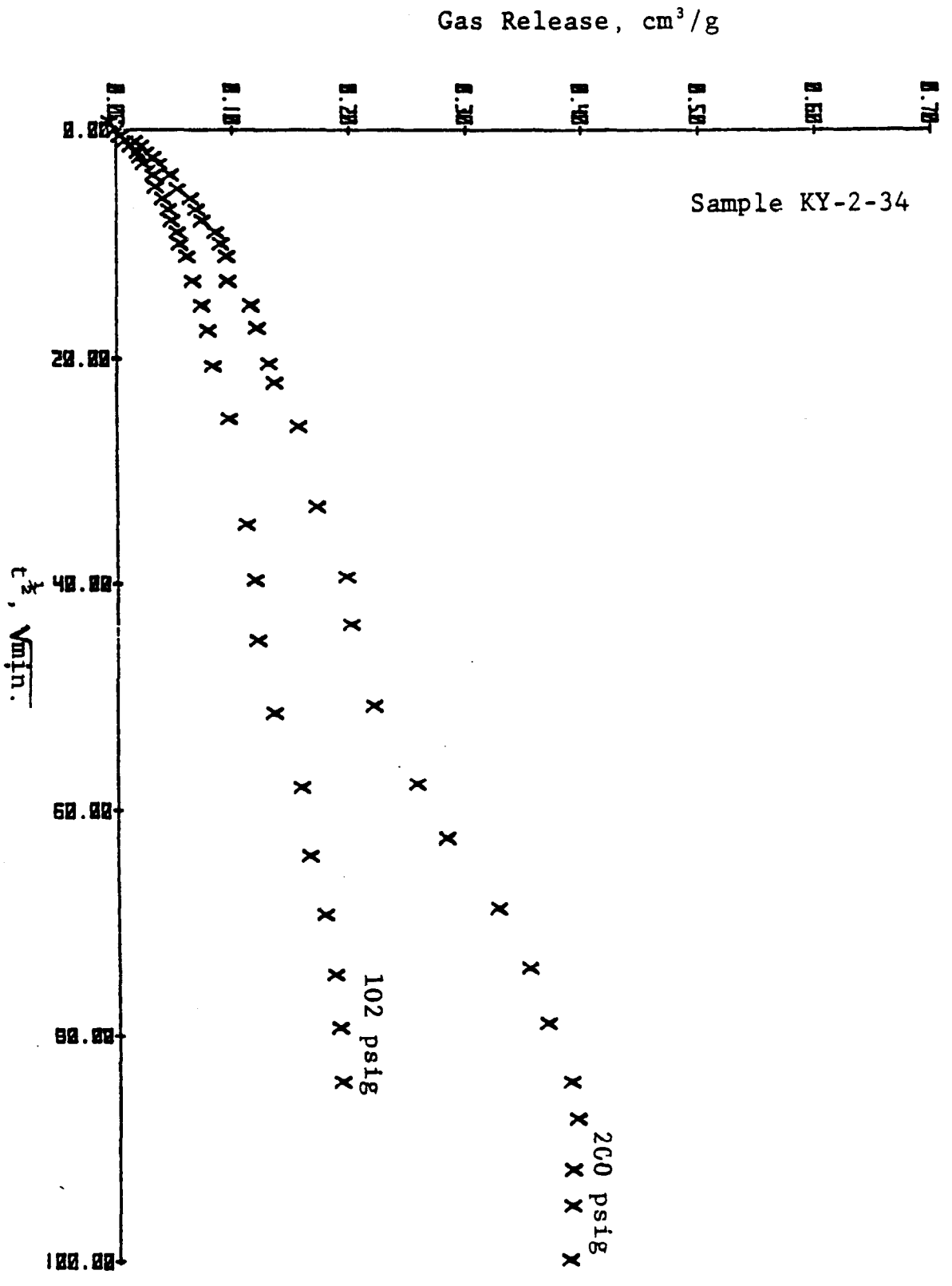

Gas Release, cm³/g
0.00
0.10
0.20
0.30
0.40
0.50
0.60
0.70
Sample KY-2-34
20.00
40.00
60.00
80.00
100.00
t^½, √min.
102 psig
200 psig

FIGURE 20

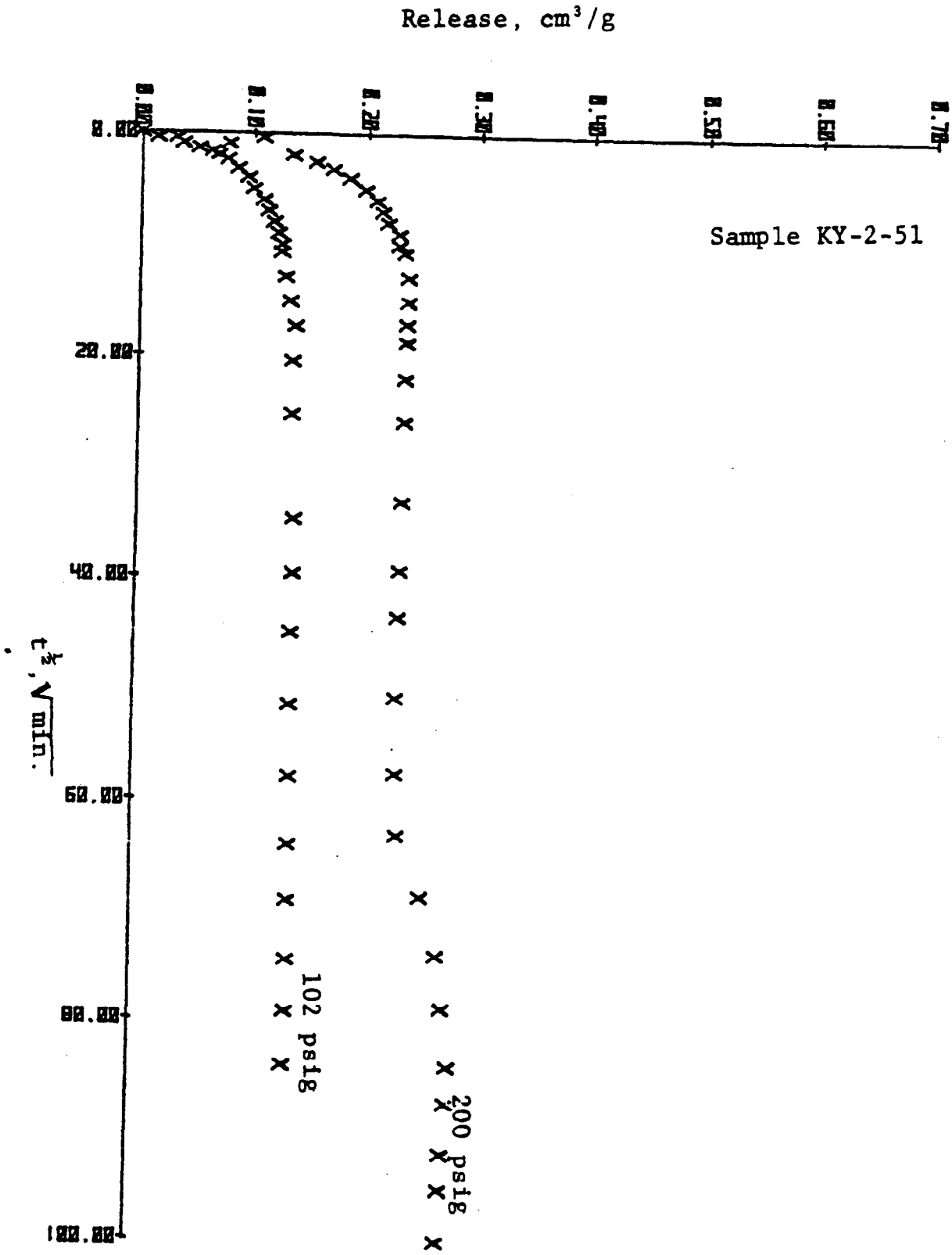

Release, cm³/g
0.00
0.10
0.20
0.30
0.40
0.50
0.60
0.70
Sample KY-2-51
102 psig
200 psig
0.00
20.00
40.00
60.00
80.00
100.00
t^½, √min.

FIGURE 21

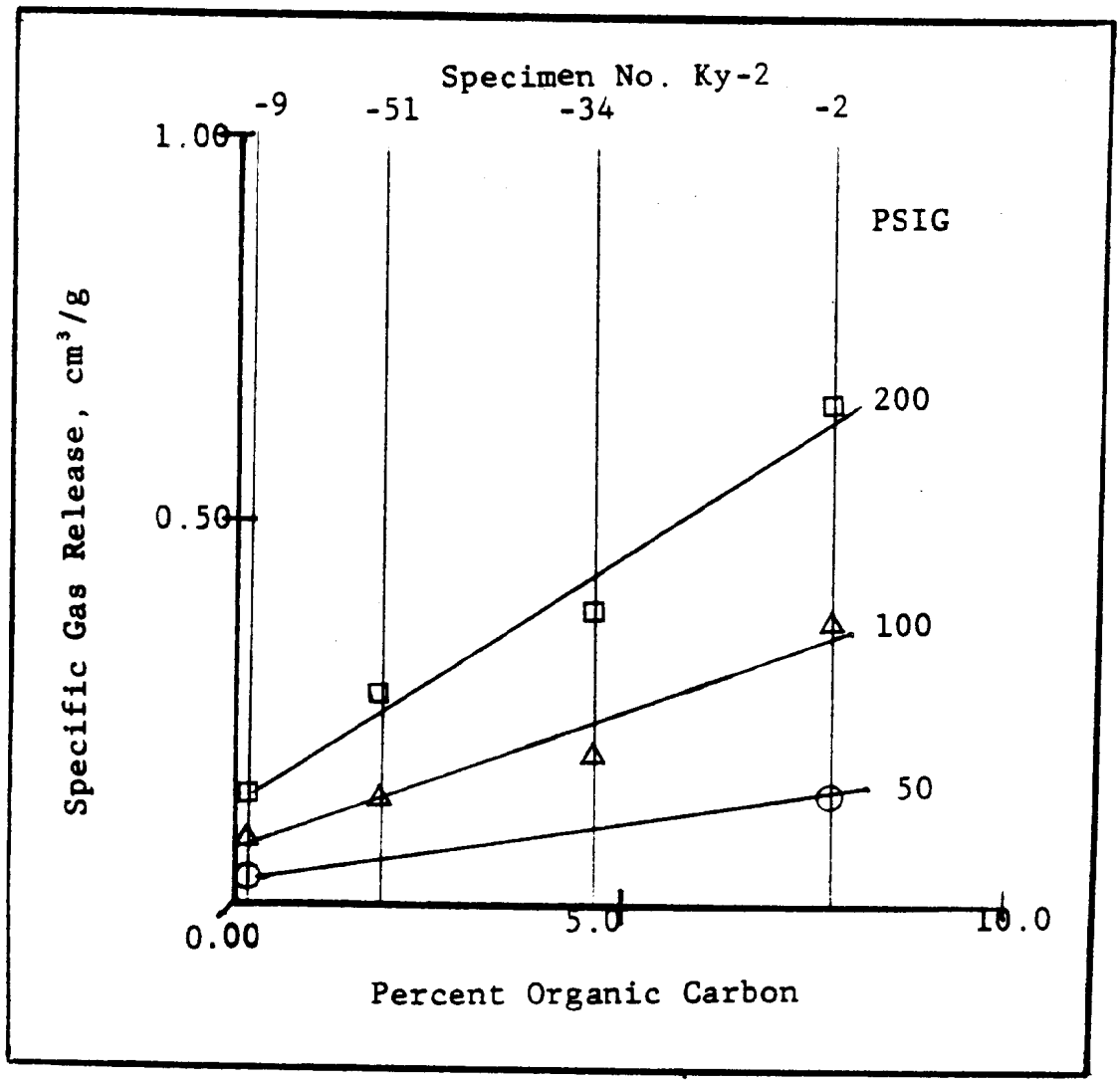



FIGURE 22

GAS RELEASE VERSUS ORGANIC CARBON CONTENT

Although some good model fits have been attained, it is premature at this time to assign a rate mechanism. The data at 300 psig, now in progress, will help resolve the mechanism.

## N. MECHANICAL TESTING

Mechanical testing of the available samples from wells KY-2, VA-1, I-1 and I-2 has been completed. The availability of integral samples of the proper size still remains a problem. Many of the samples received for physical testing are not the proper size nor are they integral. Some of these samples have been received in as many as six irregular pieces. Because of the lack of proper samples it has not been possible to perform detailed studies on the samples.

The mechanical testing results for the samples from wells KY-2, O-1 and I-1 are listed in Table 96. No specific trends have evolved from these data. The results for the samples from the VA-1 well are listed in Table 97. Some of the samples from this well were of sufficient size and consequently point load tests and splitting tensile tests in both the maximum and minimum stress directions were possible. The results of these tests showed significant differences in breaking tensile strength.

The samples from the I-2 well were of sufficient size to provide for point load tests and splitting tensile tests in the maximum stress direction. As shown in Table 98, testing in the maximum stress direction resulted in low splitting tensile strength values.

The data from the mechanical tests are currently being statistically analyzed to determine if any relationships with the companion geochemical data evolve.

The samples from the I-3 well are of sufficient size to permit multiple tests. These tests are currently in progress. They consist of sonic velocity, point load and splitting tensile tests. As part of this series of tests, the effect of sample geometry on the sonic velocity results is being evaluated. In this evaluation,

TABLE 96

Mechanical Test Data for the KY-2, O-1 and I-1 Wells

| Well | Mound Sample No. | Shore Hardness ($\bar{S}_h$) | Splitting Tensile Strength (M Pa) |
|---|---|---|---|
| KY-2 | KY-2-1 | 19.00 | 3.265 |
| | KY-2-3 | 16.30 | 2.031 |
| | KY-2-5 | 15.40 | 1.166 |
| | KY-2-10 | 13.40 | 1.523 |
| | KY-2-12 | 15.88 | 3.493 |
| | KY-2-17 | 15.20 | 1.133 |
| | KY-2-22 | 10.65 | 1.384 |
| | KY-2-30 | 8.58 | 0.849 |
| | KY-2-43 | 15.50 | 1.614 |
| O-1 | O-1-2 | 22.20 | 3.574 |
| I-1 | I-1-3 | 17.85 | 2.175 |
| | I-1-10 | 17.90 | 1.624 |

TABLE 97

Mechanical Test Data for the VA-1 Well

| Mound Sample No. | Shore Hardness ($\bar{S}_h$) | Splitting Tensile Strength (M Pa) |
|---|---|---|
| VA-1-1A* | 9.80 | 1.518 |
| VA-1-1B* | 9.80 | 2.277 |
| VA-1-3A | 12.30 | 0.785 |
| VA-1-3B | 12.30 | 2.911 |
| VA-1-5 | 8.35 | 2.847 |
| VA-1-7 | 14.50 | 3.105 |
| VA-1-9 | 11.60 | 1.214 |
| VA-1-10 | 9.50 | 1.008 |
| VA-1-14 | 14.00 | 2.613 |
| VA-1-16 | 15.70 | 1.937 |
| VA-1-19 | 7.80 | 1.641 |
| VA-1-19R** | 7.80 | 1.681 |
| VA-1-20 | 9.95 | 1.243 |
| VA-1-28A | 8.15 | 0.548 |
| VA-1-28B | 8.15 | 0.722 |
| VA-1-31A | 19.85 | 1.631 |
| VA-1-31B | 19.85 | 3.035 |

*A designates sample tested in maximum stress direction.
B designates sample tested in minimum stress direction.

** Duplicate sample tested in same direction.

TABLE 98

Mechanical Test Data for the I-22 Well

| Mound Sample No. | Shore Hardness ($\bar{S}_h$) | Splitting Tensile Strength (M Pa) |
|---|---|---|
| I-2-1 | 10.35 | 0.744 |
| I-2-4 | 11.55 | 1.160 |
| I-2-13 | 9.60 | 0.851 |
| I-2-15 | 9.65 | 1.070 |
| I-2-17 | 9.40 | 0.994 |
| I-2-19 | 13.45 | 0.713 |

All samples were tested in the maximum stress direction.

the results obtained from round, disc-type specimens are being compared to those obtained from specimens that have sides that have been machined flat. The flat surface provides good detector contact and is the most ideal for sonic velocity measurements. The results from this study will provide insight into the accuracy and precision of data obtained from the normally tested round specimens.

The gases released from the samples during the splitting tensile tests are being monitored using a quadrupole mass spectrometer. Typical results for the samples from the KY-2 well are shown in Figures 23 through 29. In these figures, the solid spectrum overlayed on the line trace spectrum represents system background, while the line trace spectrum represents the gases present immediately upon fracturing. The predominant trends observed in these samples were either no gas release or the release of nitrogen, water vapor and trace quantities of oxygen. These observations were supportative of the gas release data determined in other experiments. These data indicated that the significant quantities of hydrocarbon gases were already released to the head space volume of the sample containers. Sample KY-2-43 (Figure 29) was somewhat different in that higher order hydrocarbon gases were released upon sample fracturing.

Typical results for the samples from the VA-1 well are shown in Figures 30 through 36. These results are presented in line trace spectrum format because there was essentially no change in spectra when the samples were fractured. The only changes which were observed were for helium and water; the background traces of these species are indicated by solid spectrum traces. The significant quantities of helium which were observed in the spectra were the direct result of sample handling. All the VA-1 samples were maintained in a helium atmosphere prior to test and obviously the helium was absorbed by the samples during storage and sample preparation and, consequently, was released during fracture.

Sample spectra for the I-2 samples are shown in Figures 37 through 40. The A spectra present the system background, while the B spectra represent the spectra obtained during fracture. For Sample I-2-1, there was a very little change in spectra, while the other samples indicated a

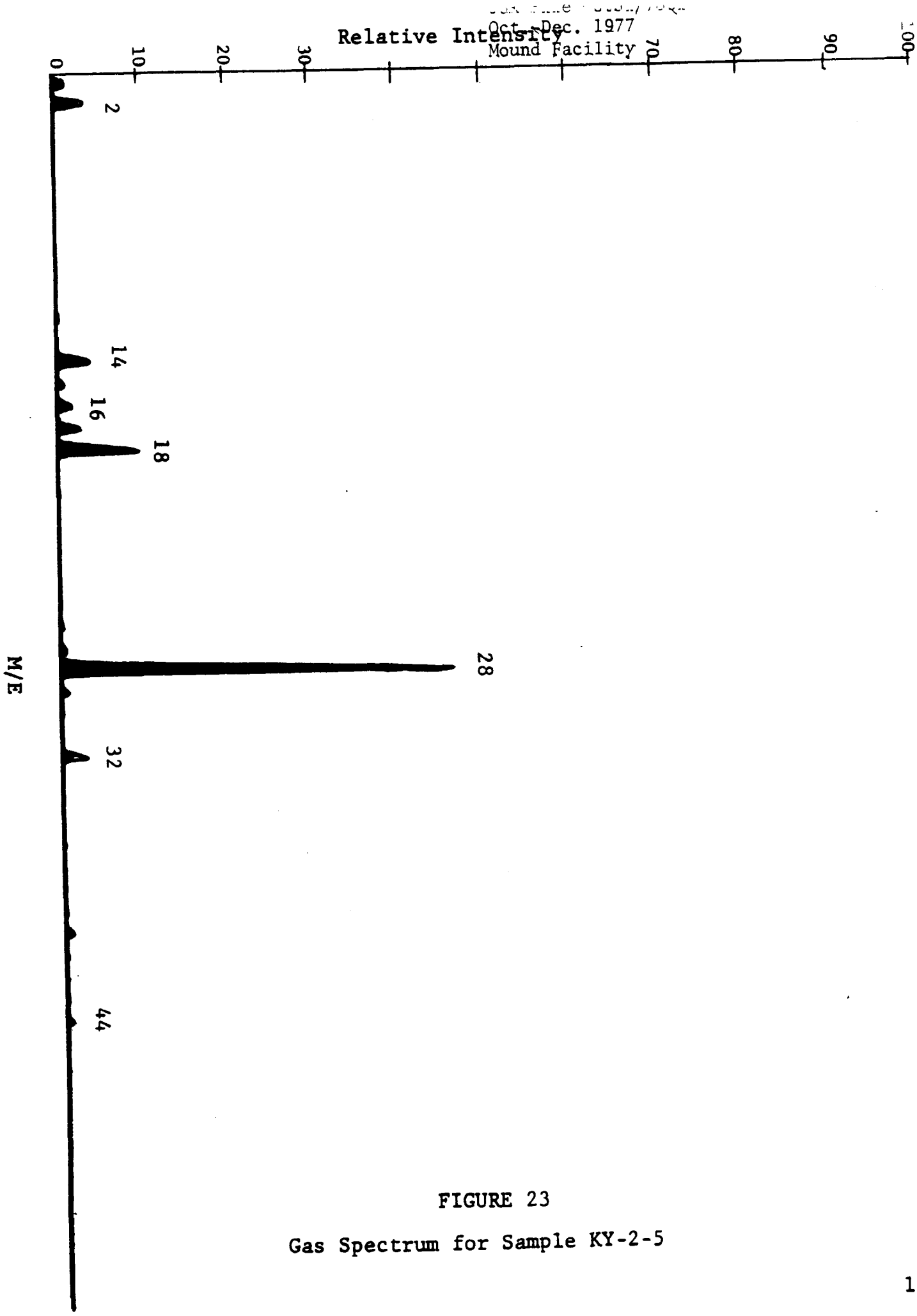



FIGURE 23

Gas Spectrum for Sample KY-2-5

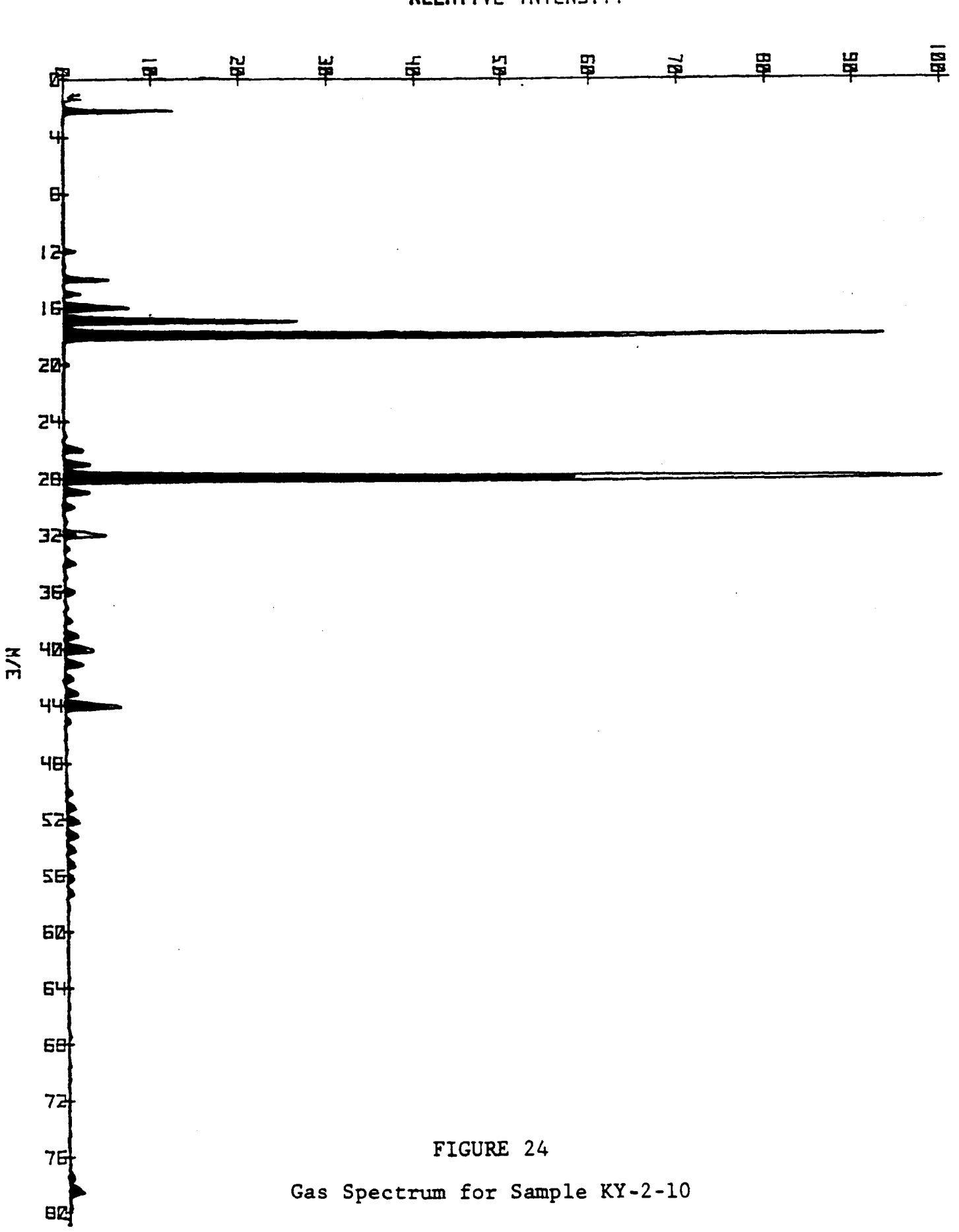



FIGURE 24

Gas Spectrum for Sample KY-2-10

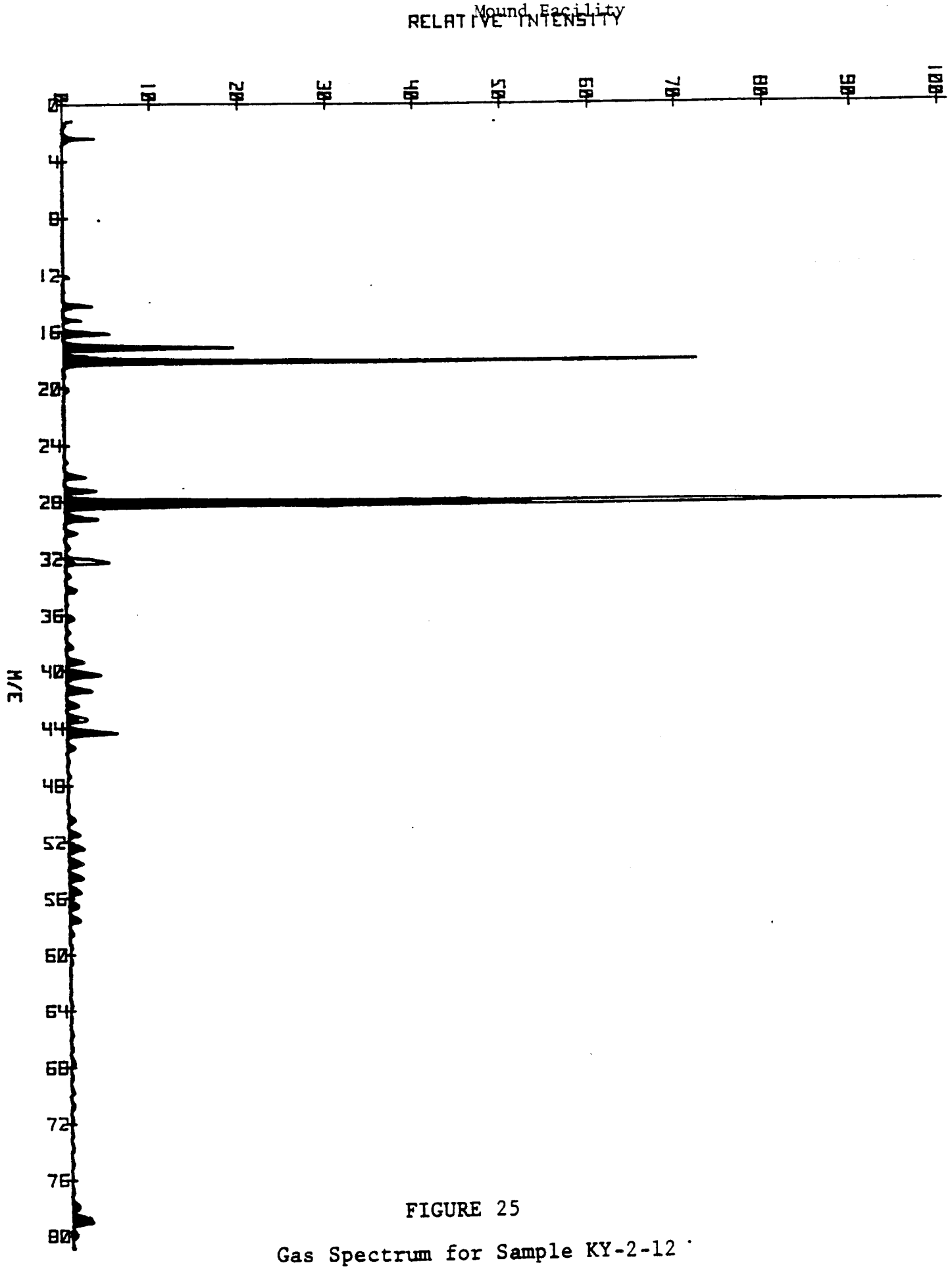



FIGURE 25

Gas Spectrum for Sample KY-2-12

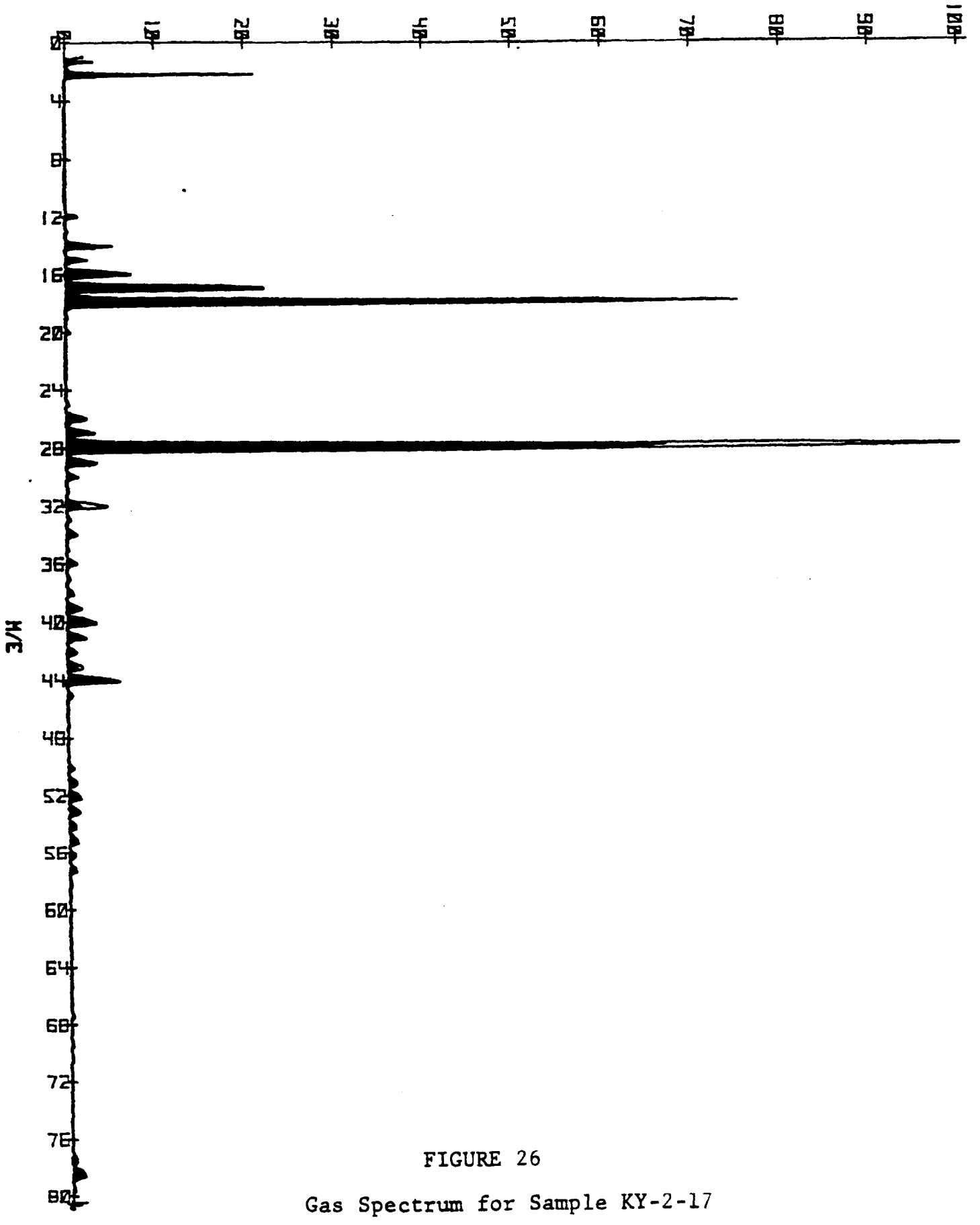



FIGURE 26

Gas Spectrum for Sample KY-2-17

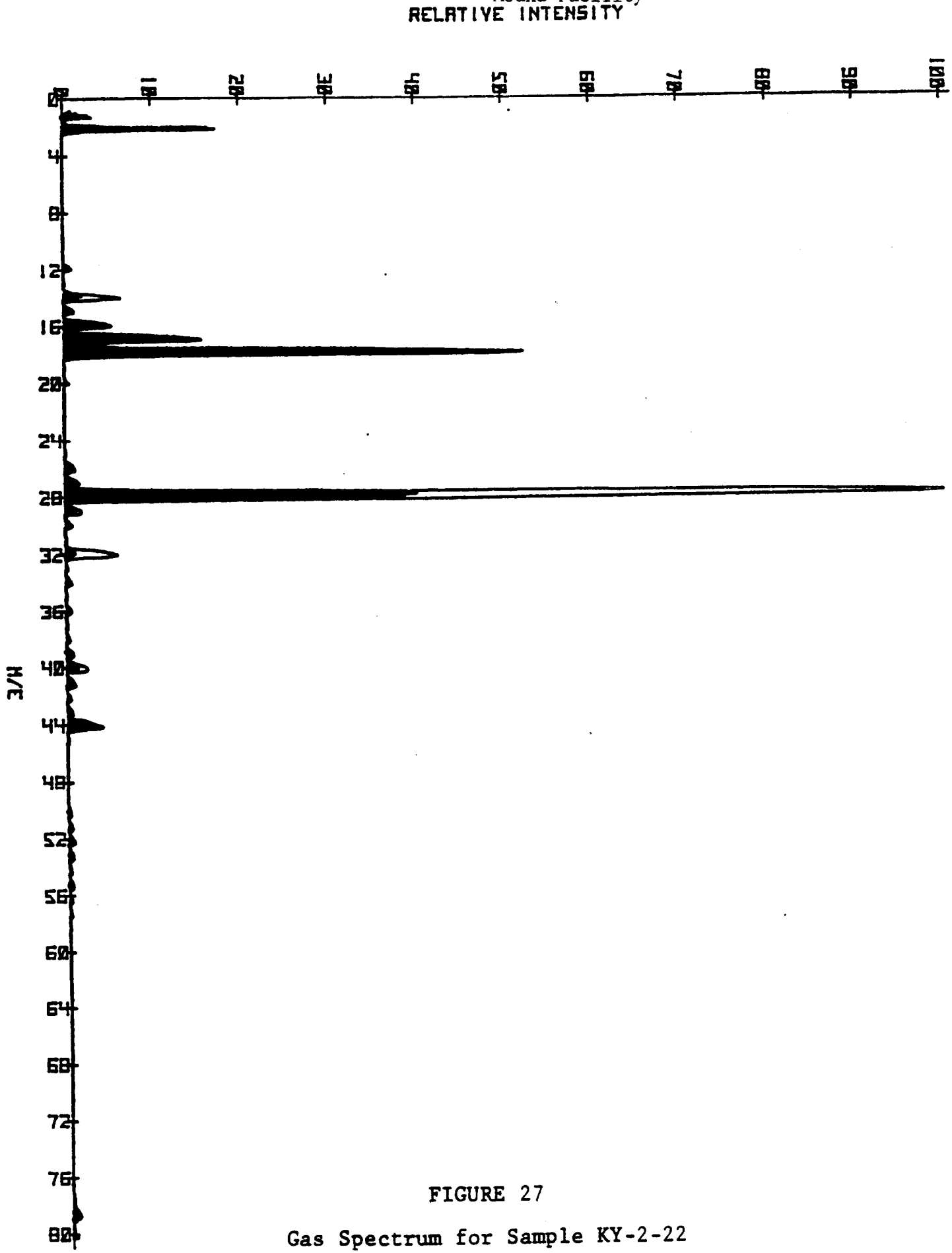



FIGURE 27

Gas Spectrum for Sample KY-2-22

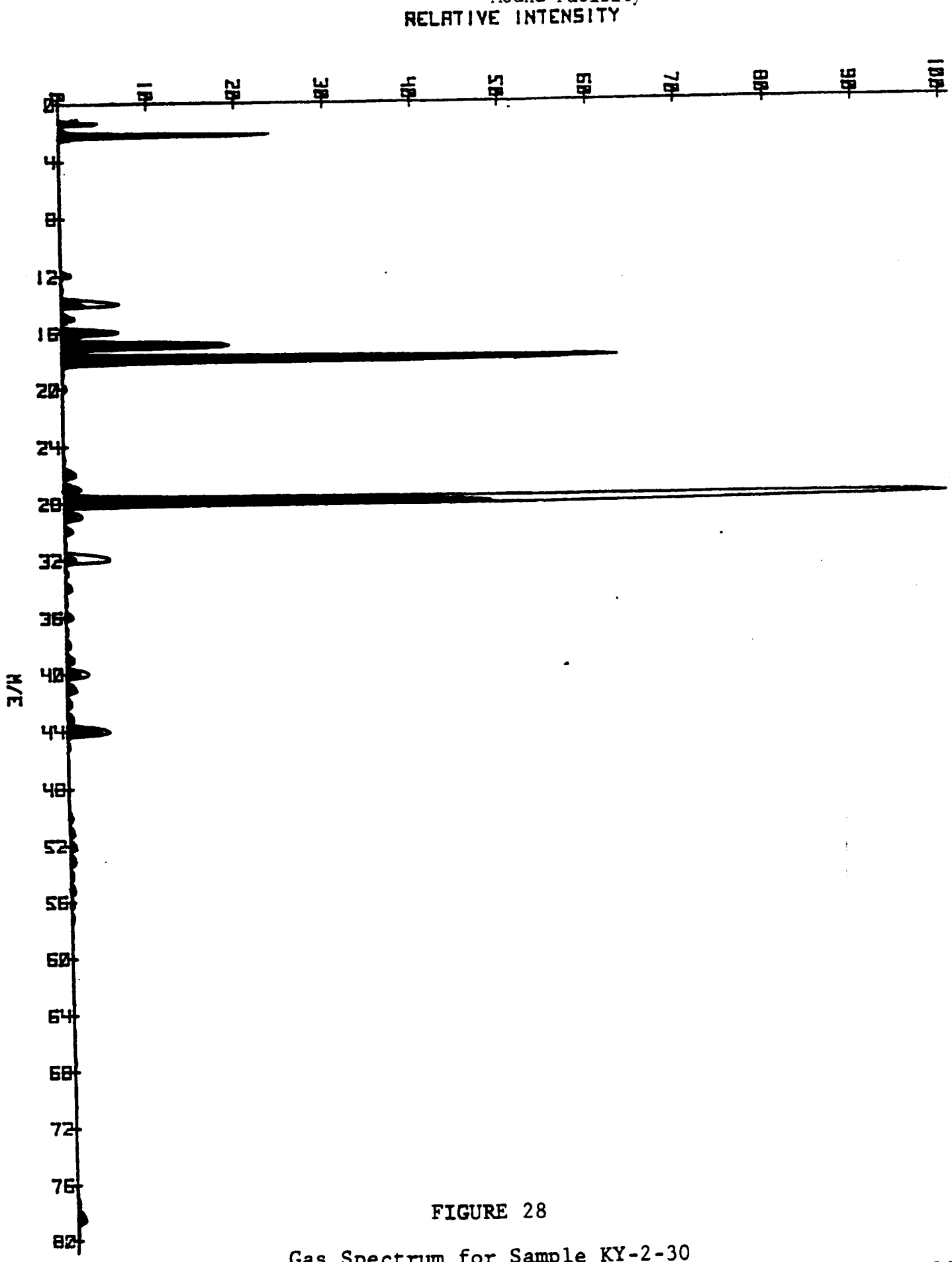



FIGURE 28

Gas Spectrum for Sample KY-2-30

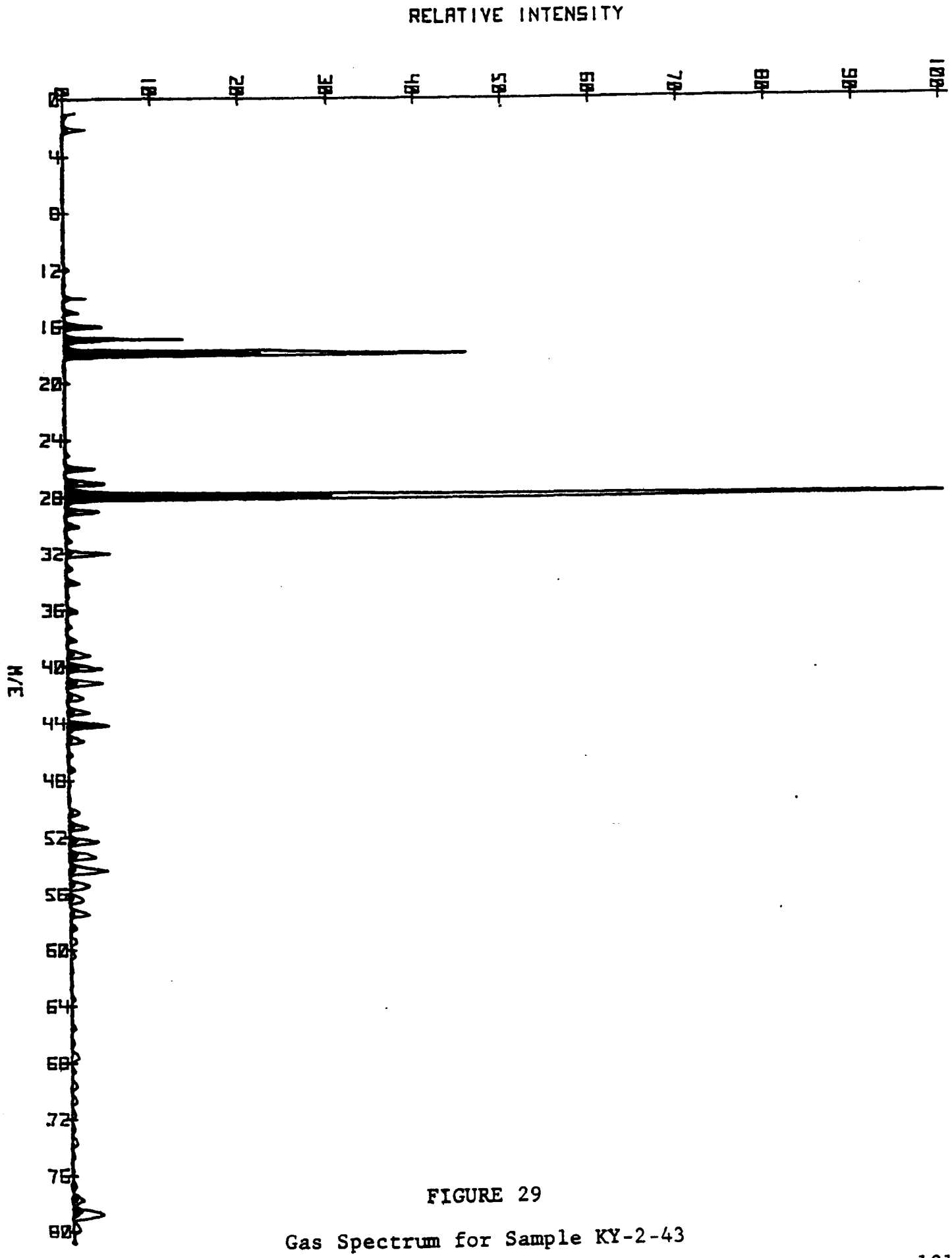



FIGURE 29

Gas Spectrum for Sample KY-2-43

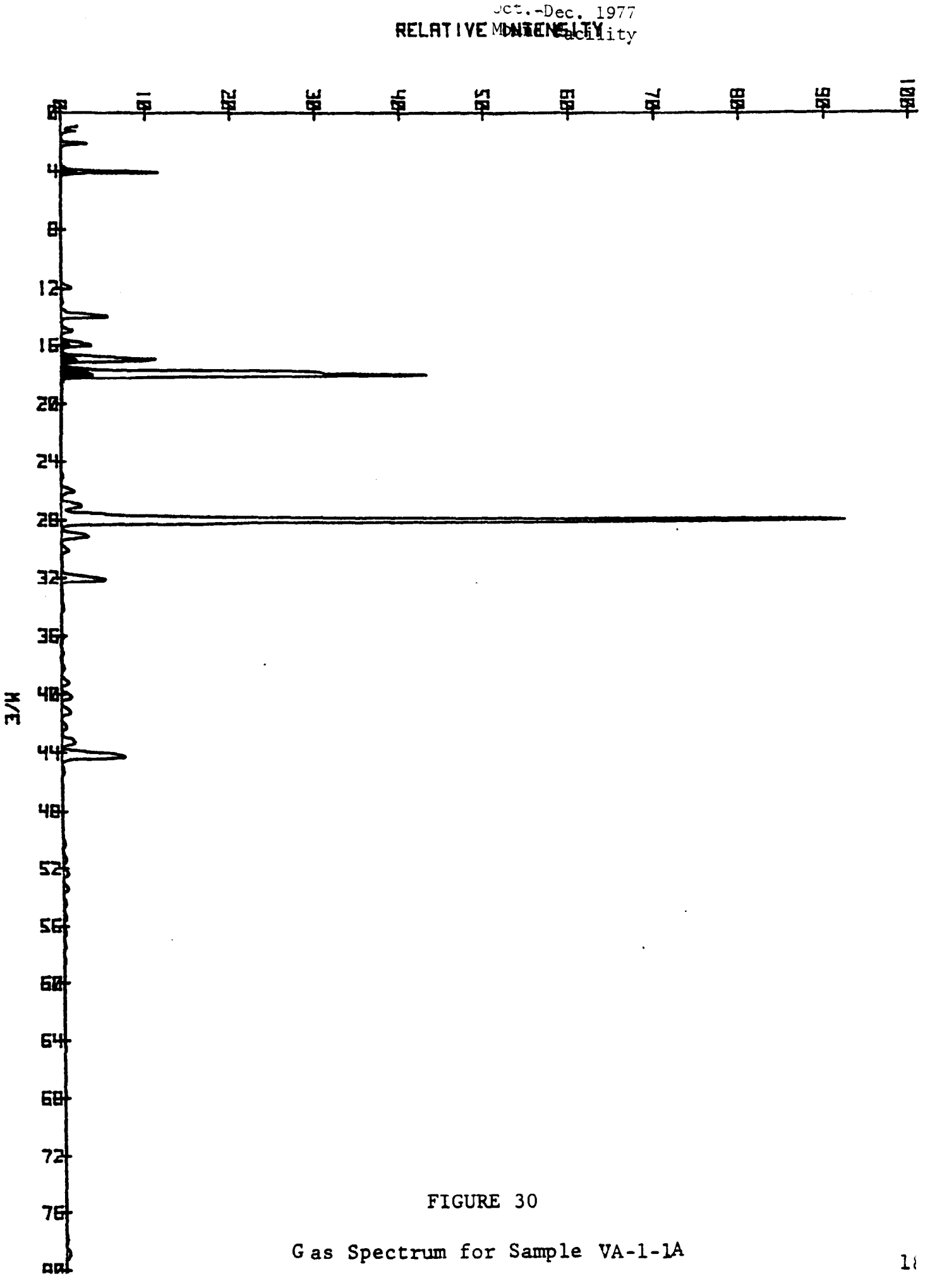



FIGURE 30

Gas Spectrum for Sample VA-1-1A

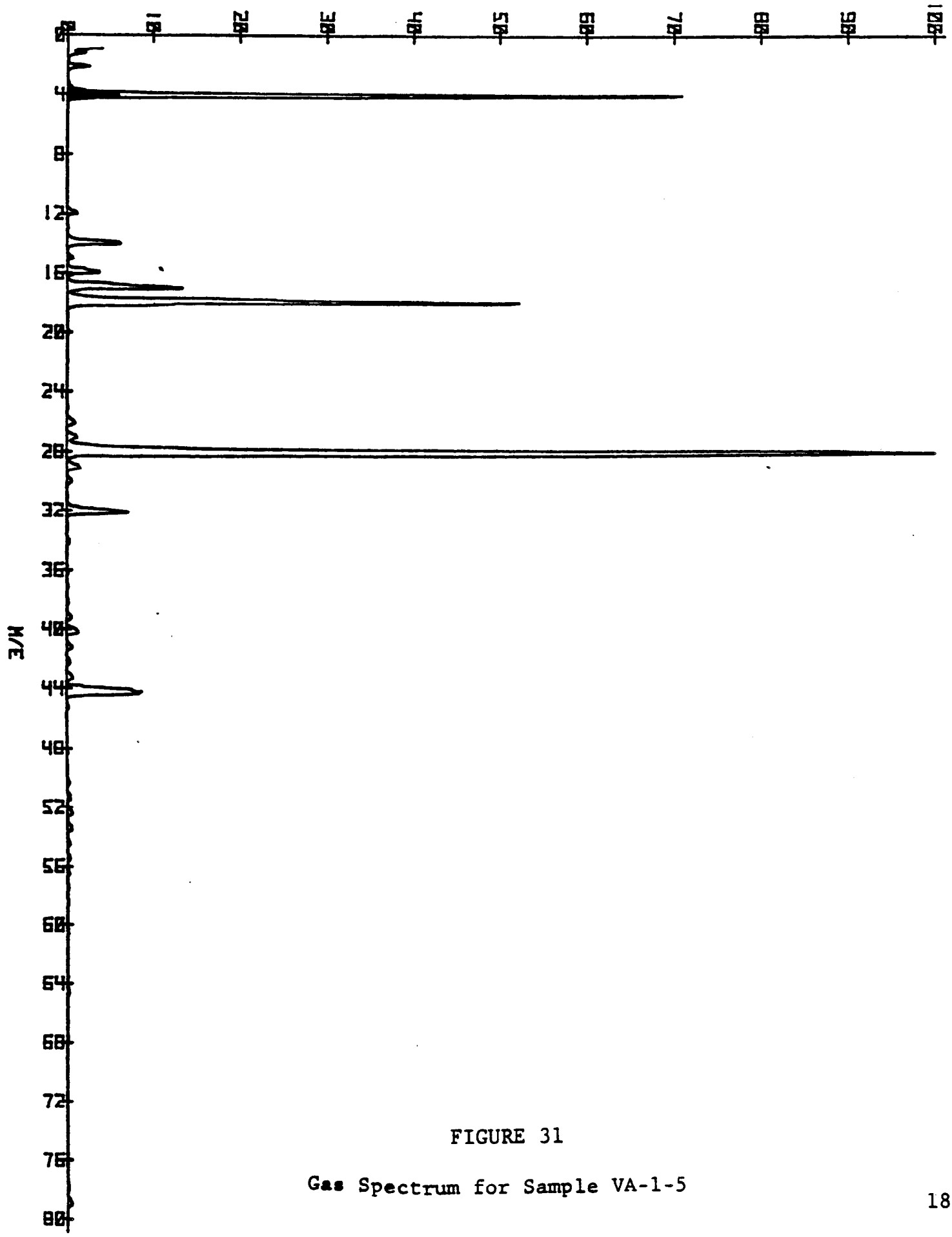



FIGURE 31

Gas Spectrum for Sample VA-1-5

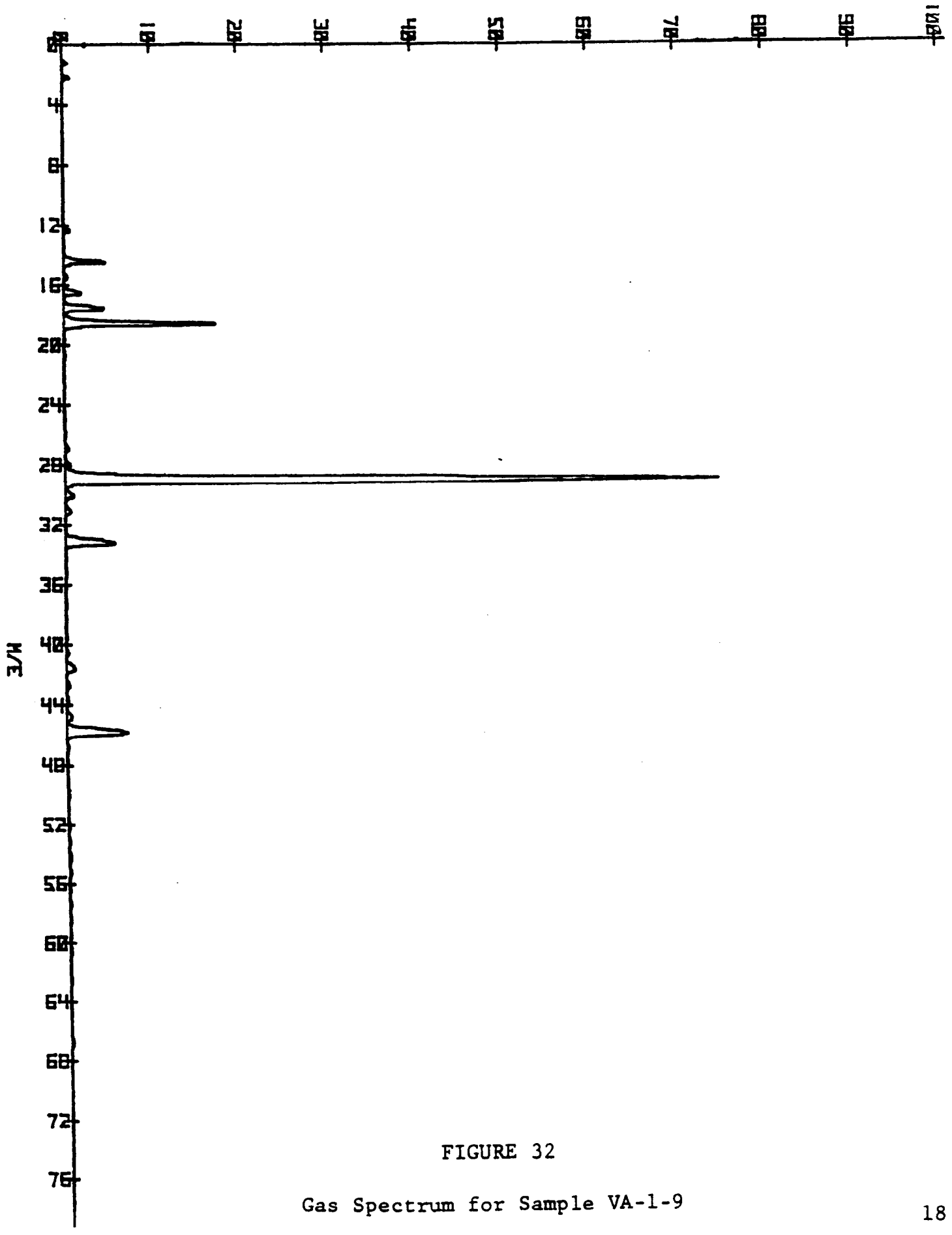



FIGURE 32

Gas Spectrum for Sample VA-1-9

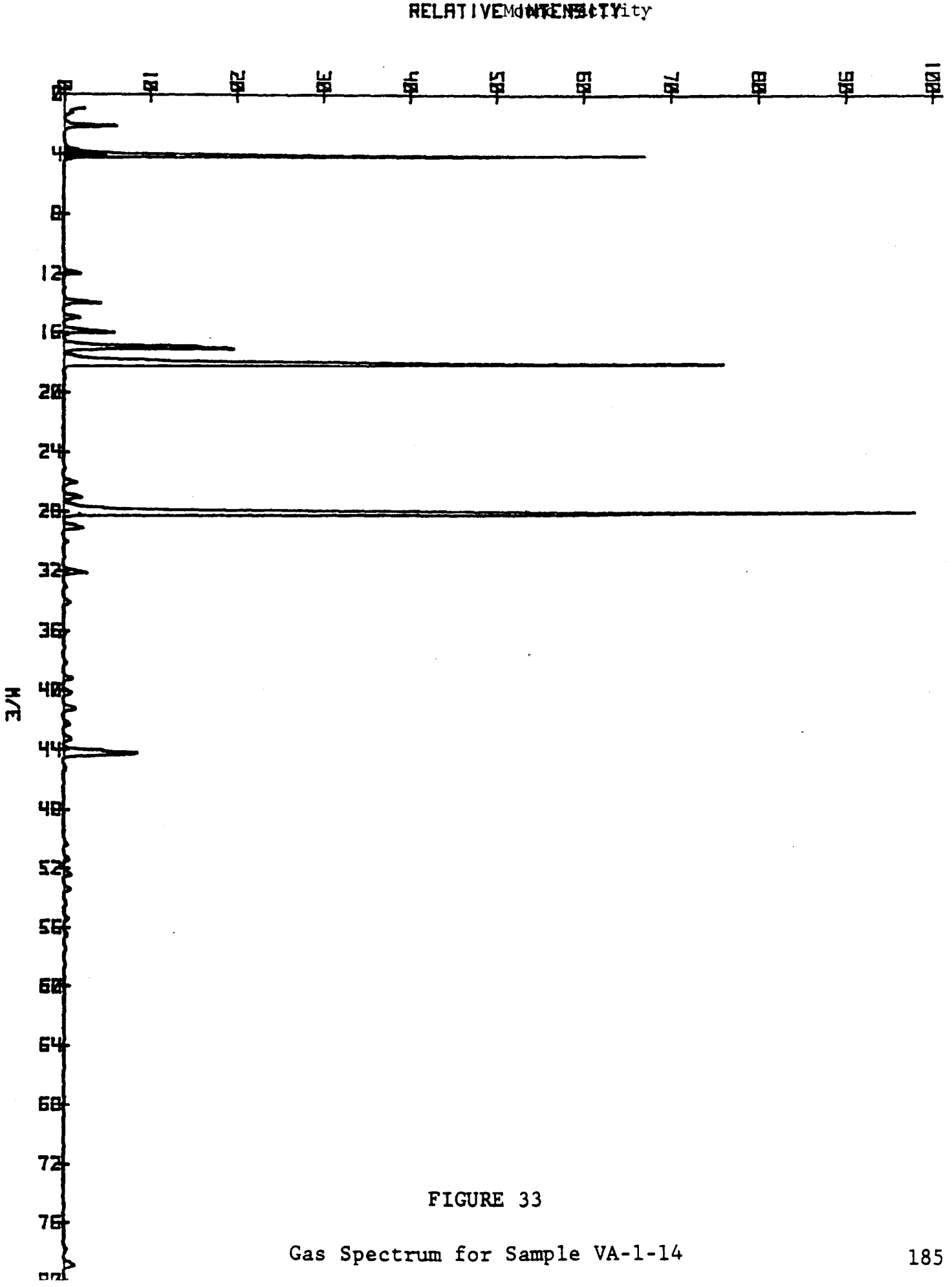



FIGURE 33

Gas Spectrum for Sample VA-1-14

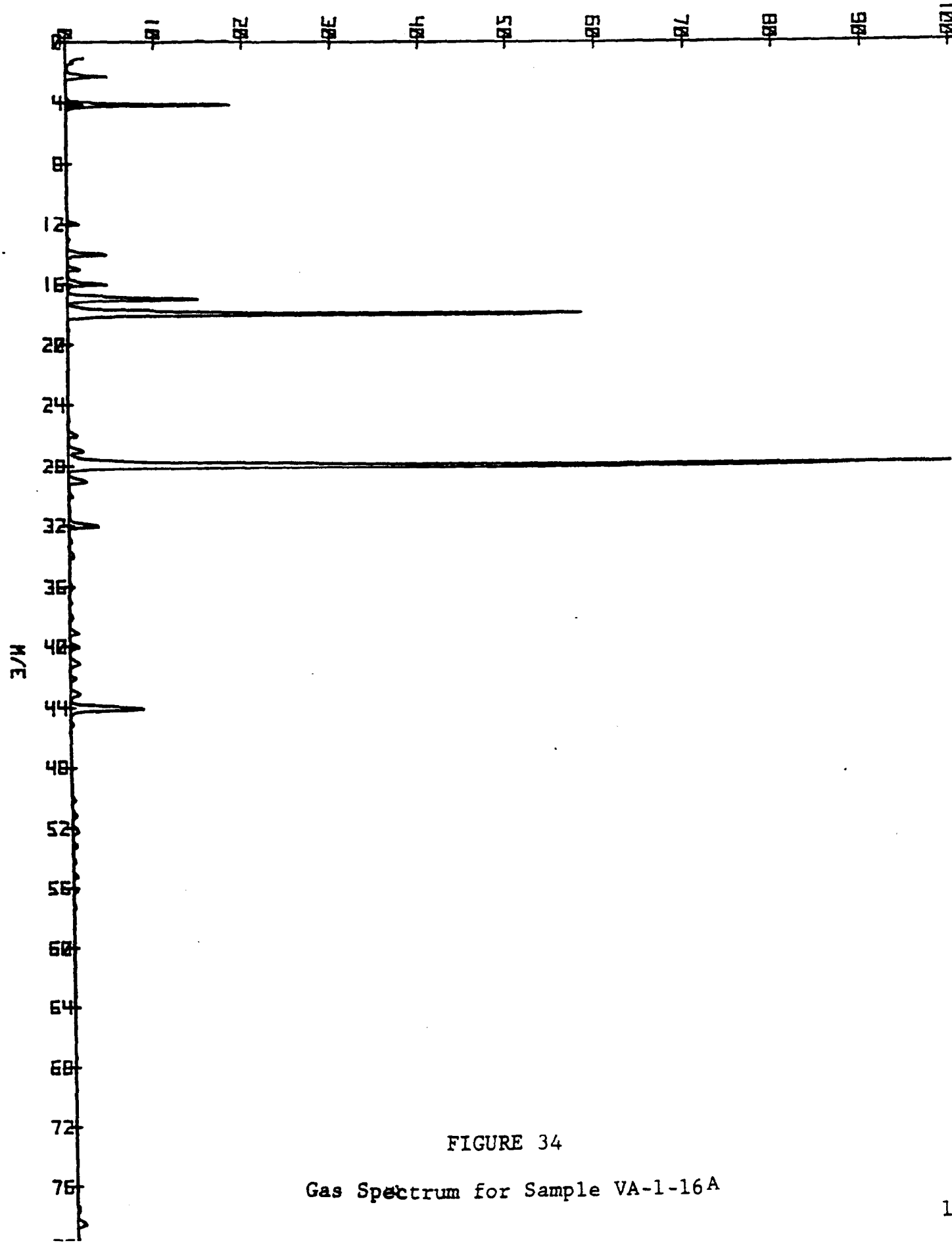



FIGURE 34

Gas Spectrum for Sample VA-1-16A

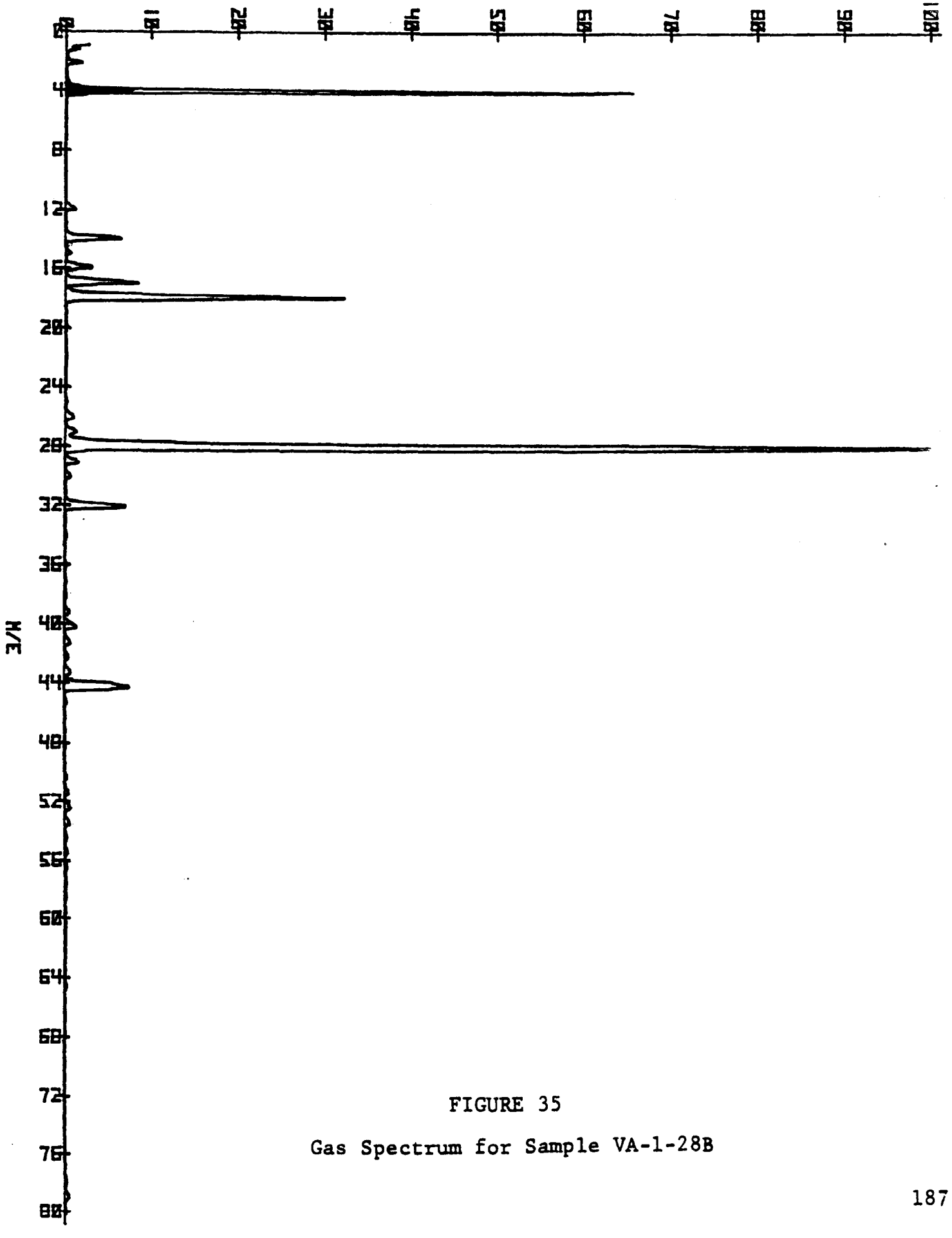



FIGURE 35

Gas Spectrum for Sample VA-1-28B

UGR File #C051/78Q1
Oct.-Dec. 1977
Mound Facility

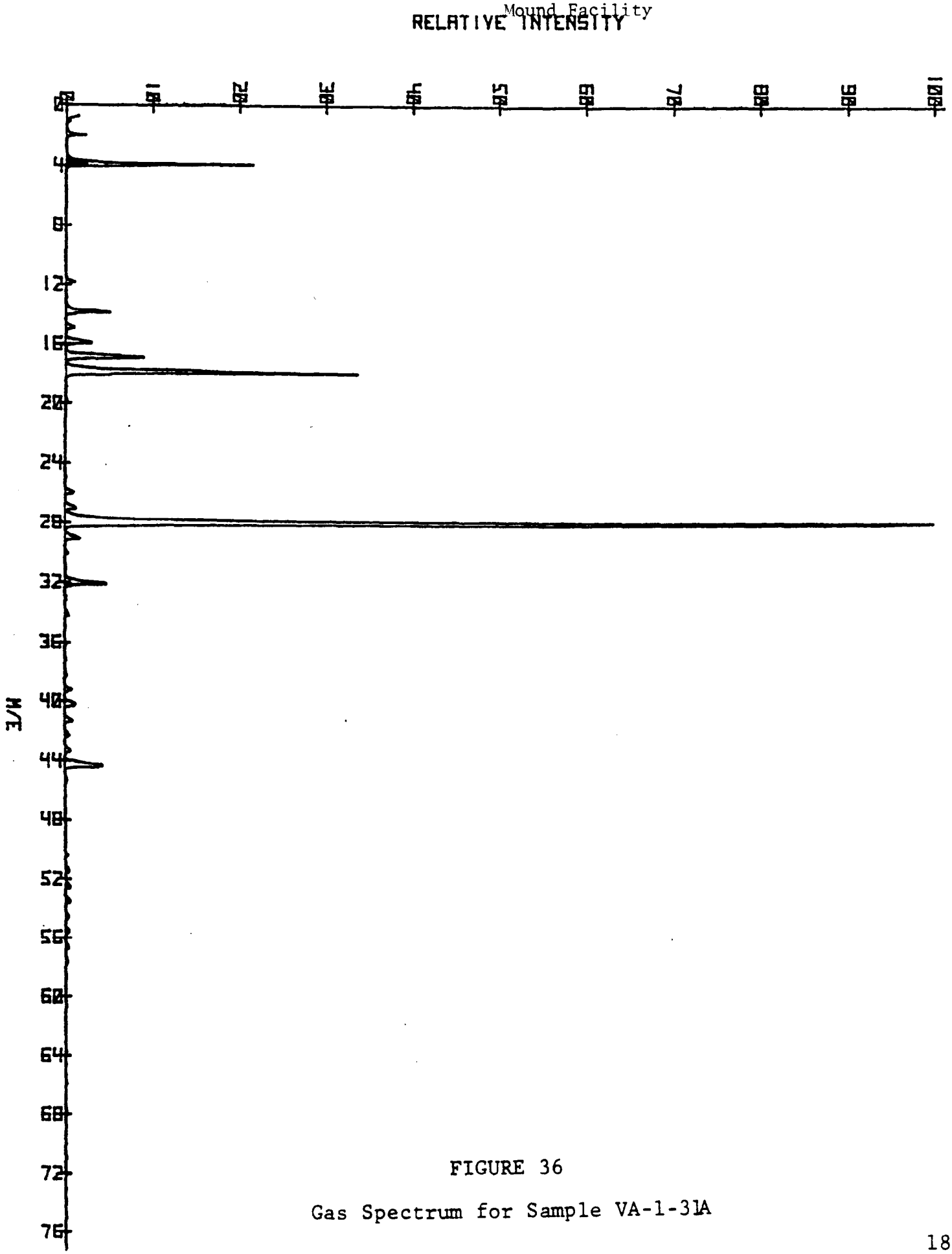



FIGURE 36

Gas Spectrum for Sample VA-1-31A

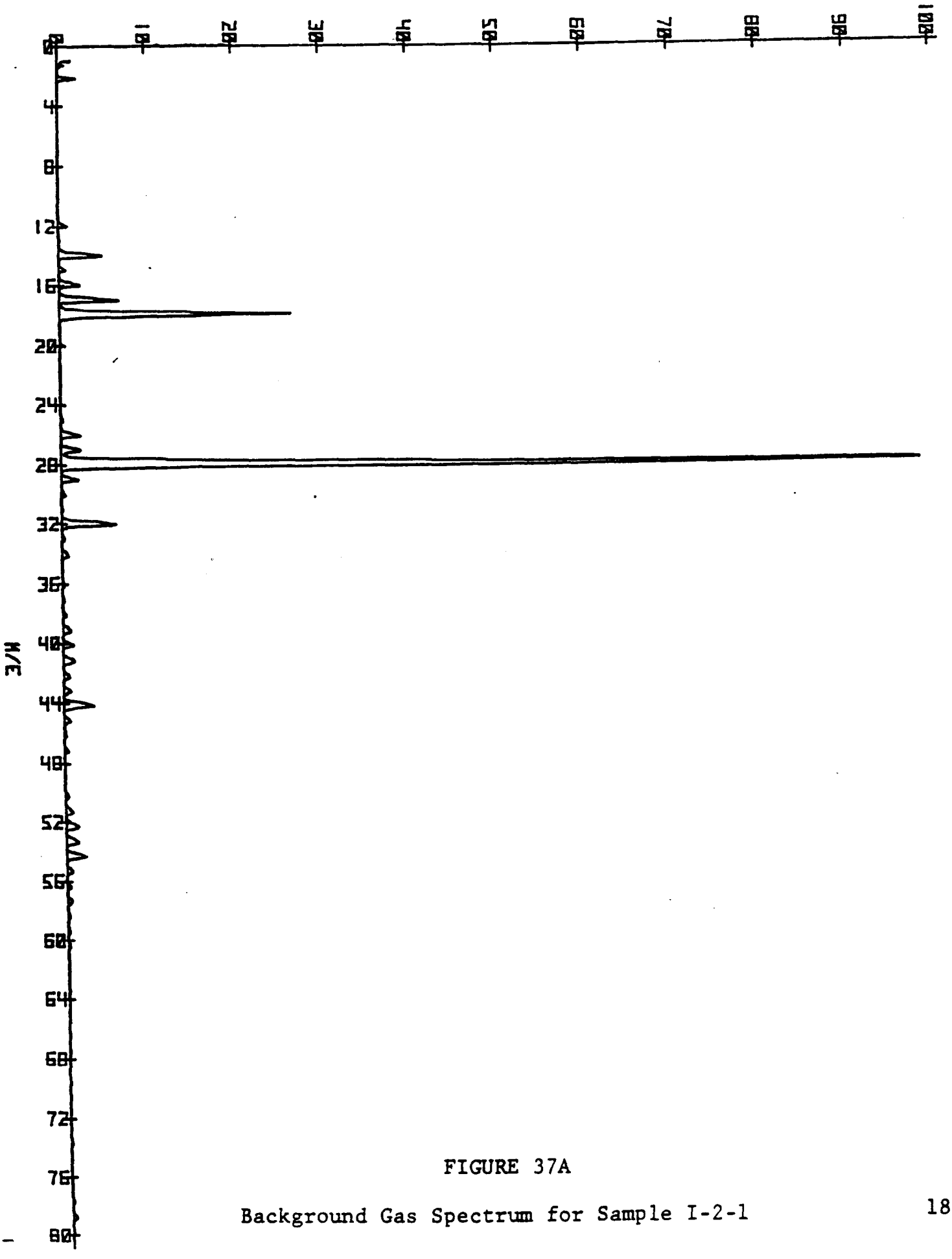



FIGURE 37A

Background Gas Spectrum for Sample I-2-1

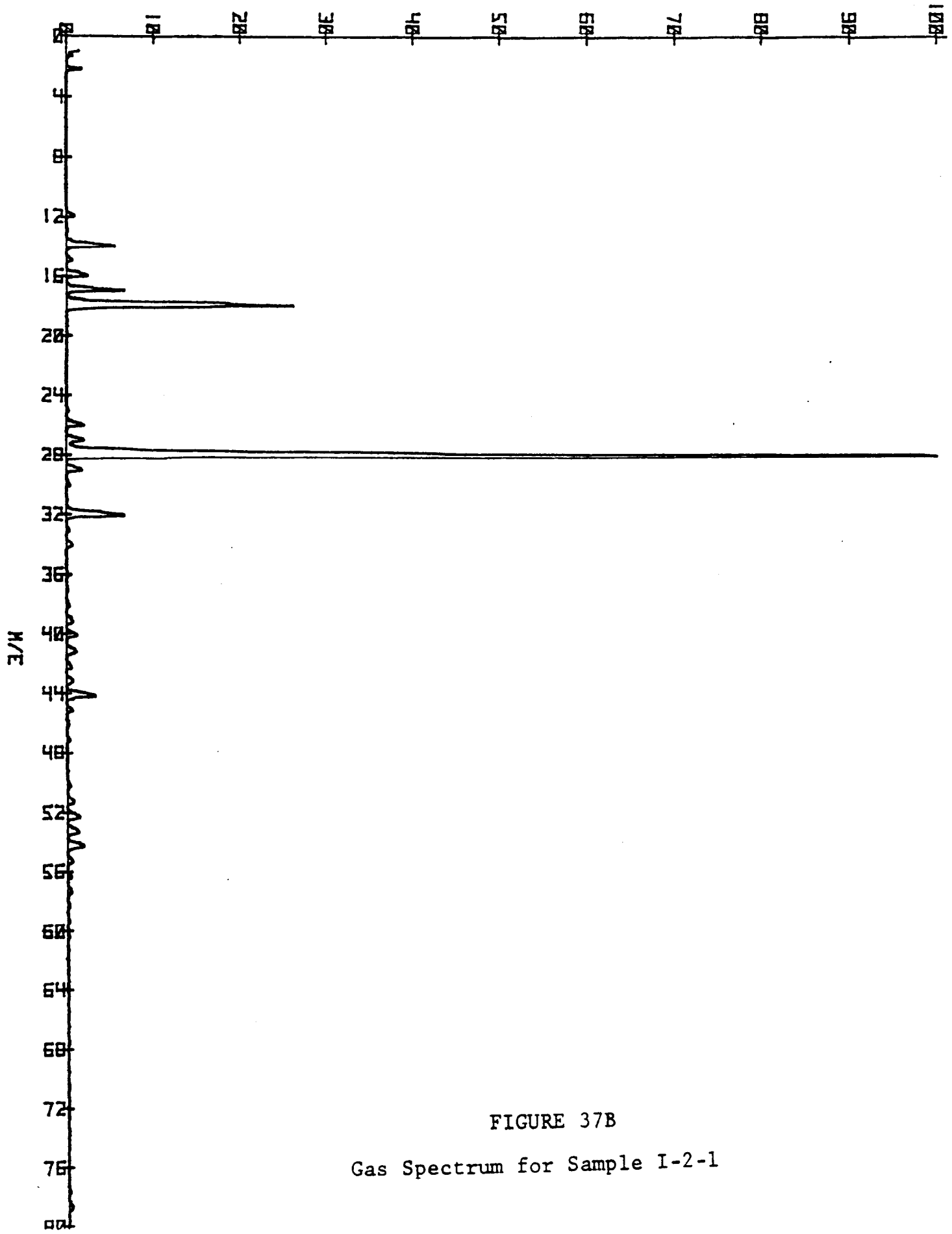



FIGURE 37B

Gas Spectrum for Sample I-2-1

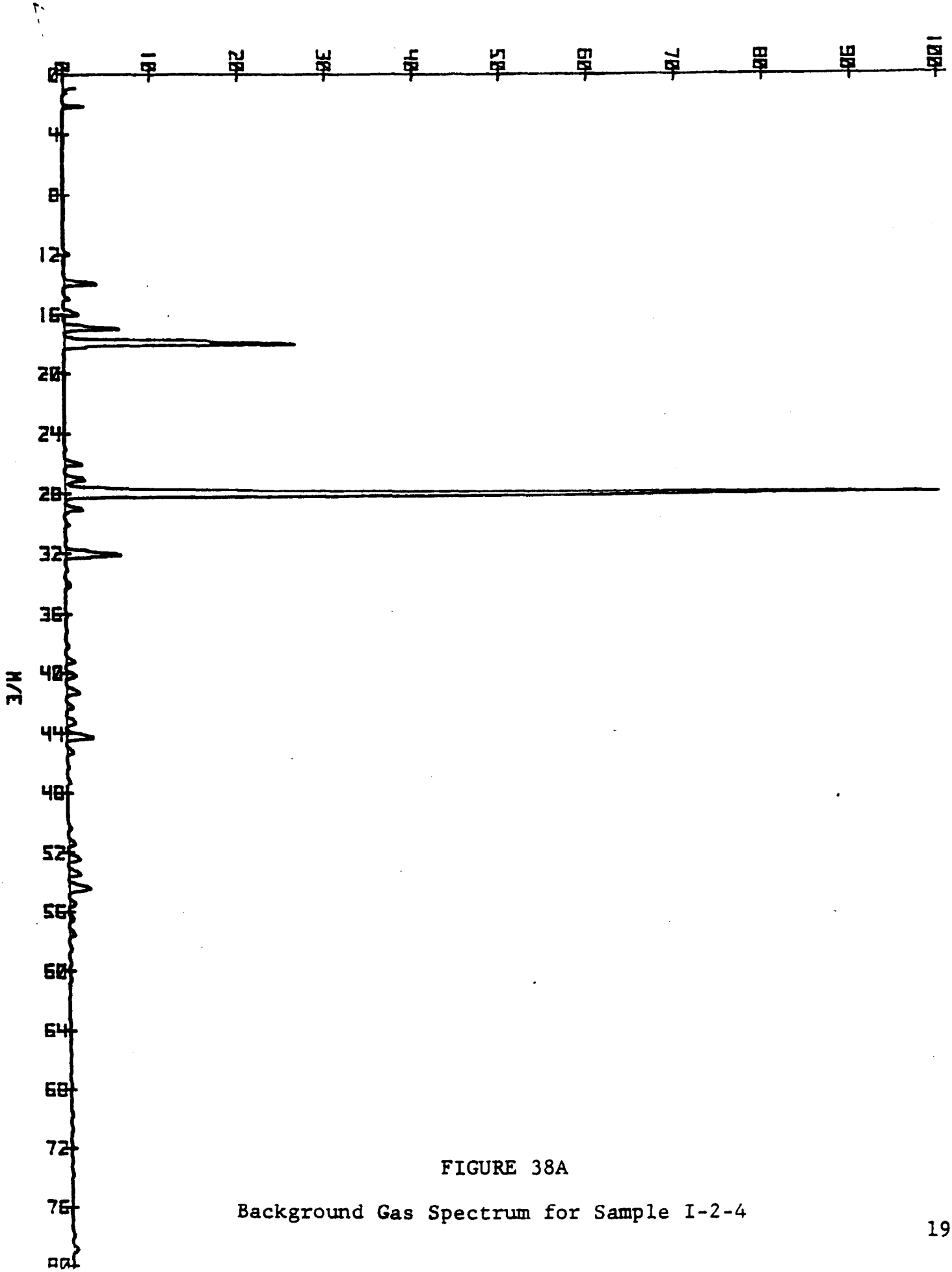



FIGURE 38A

Background Gas Spectrum for Sample I-2-4

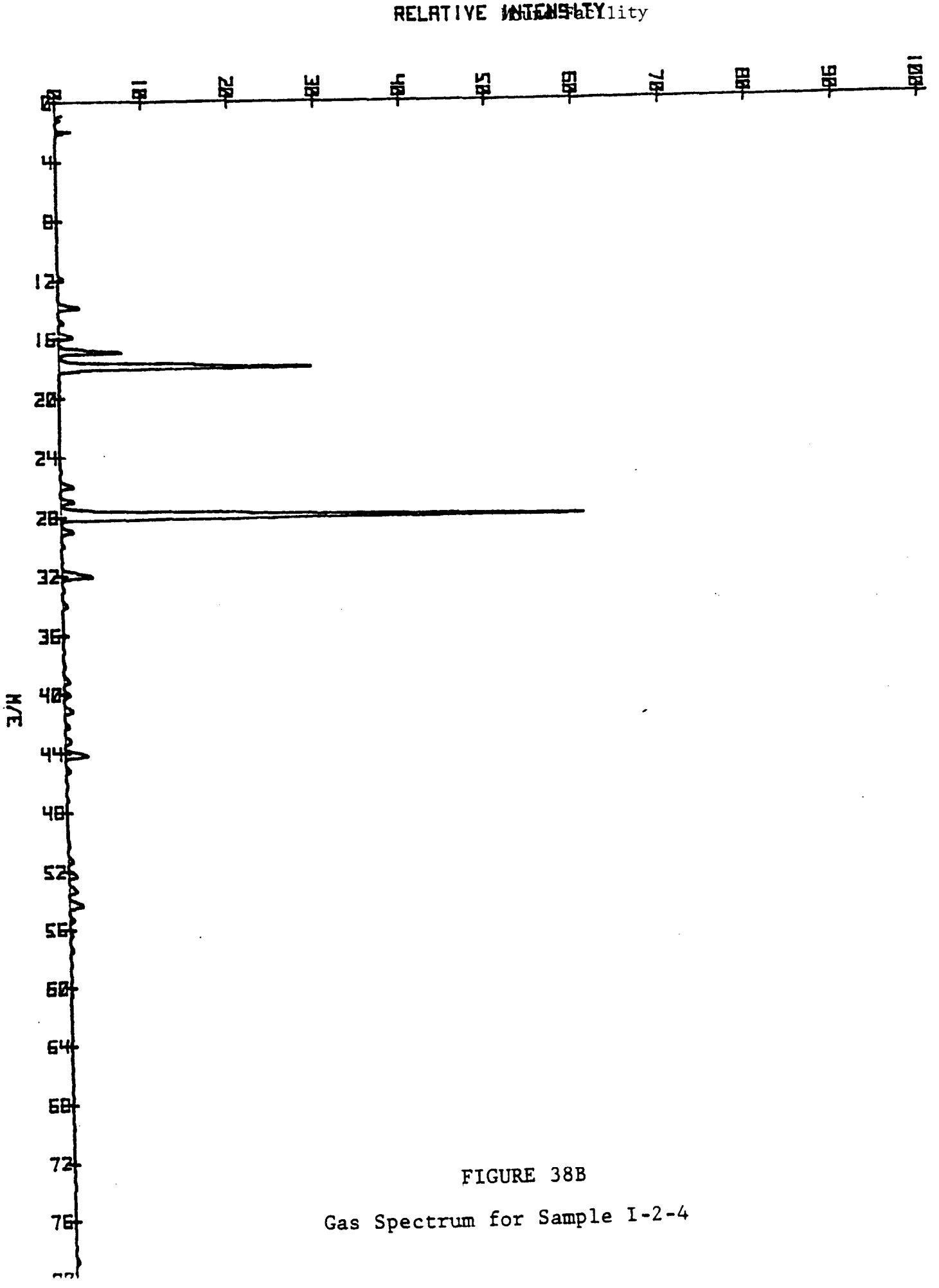



FIGURE 38B

Gas Spectrum for Sample I-2-4

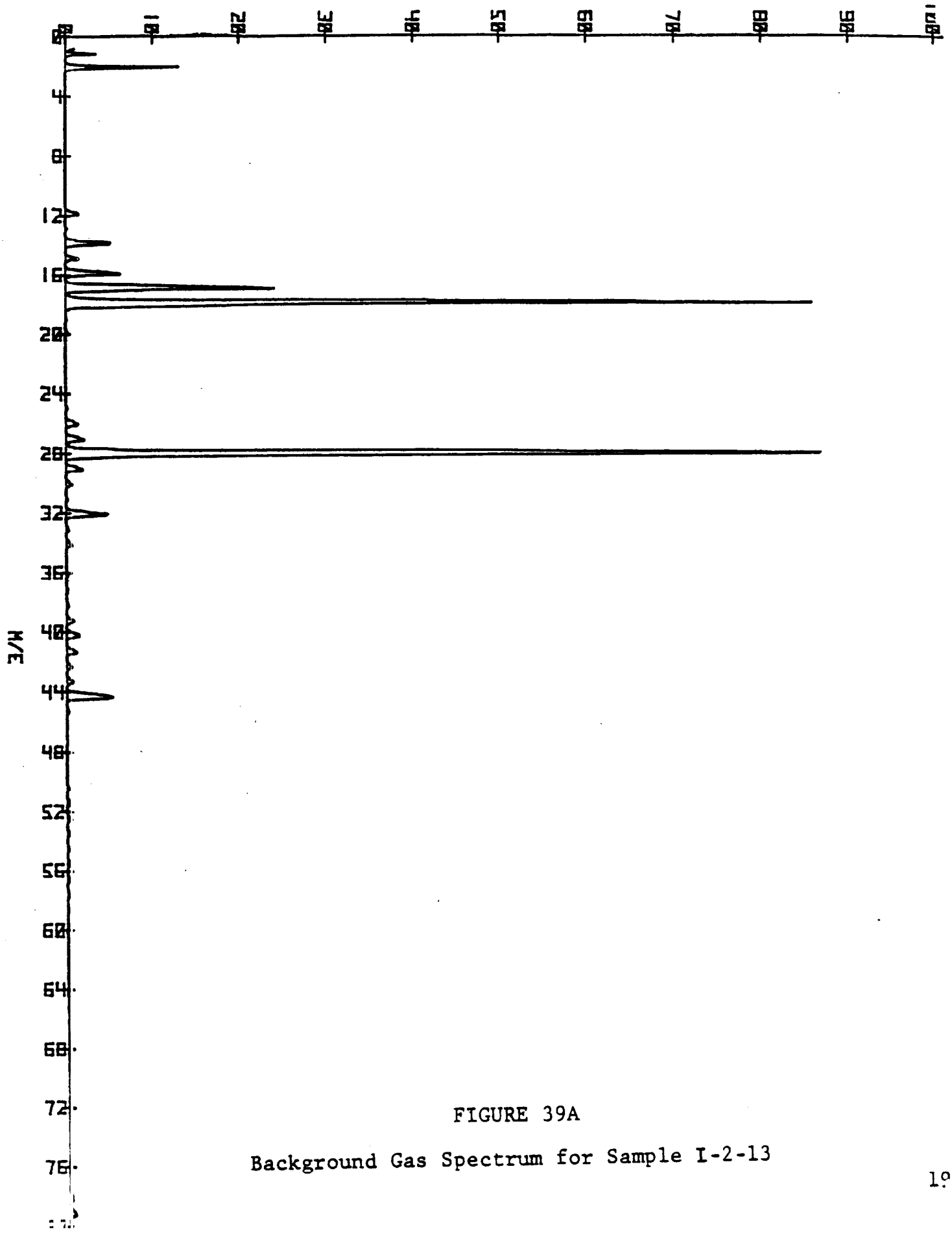



FIGURE 39A

Background Gas Spectrum for Sample I-2-13

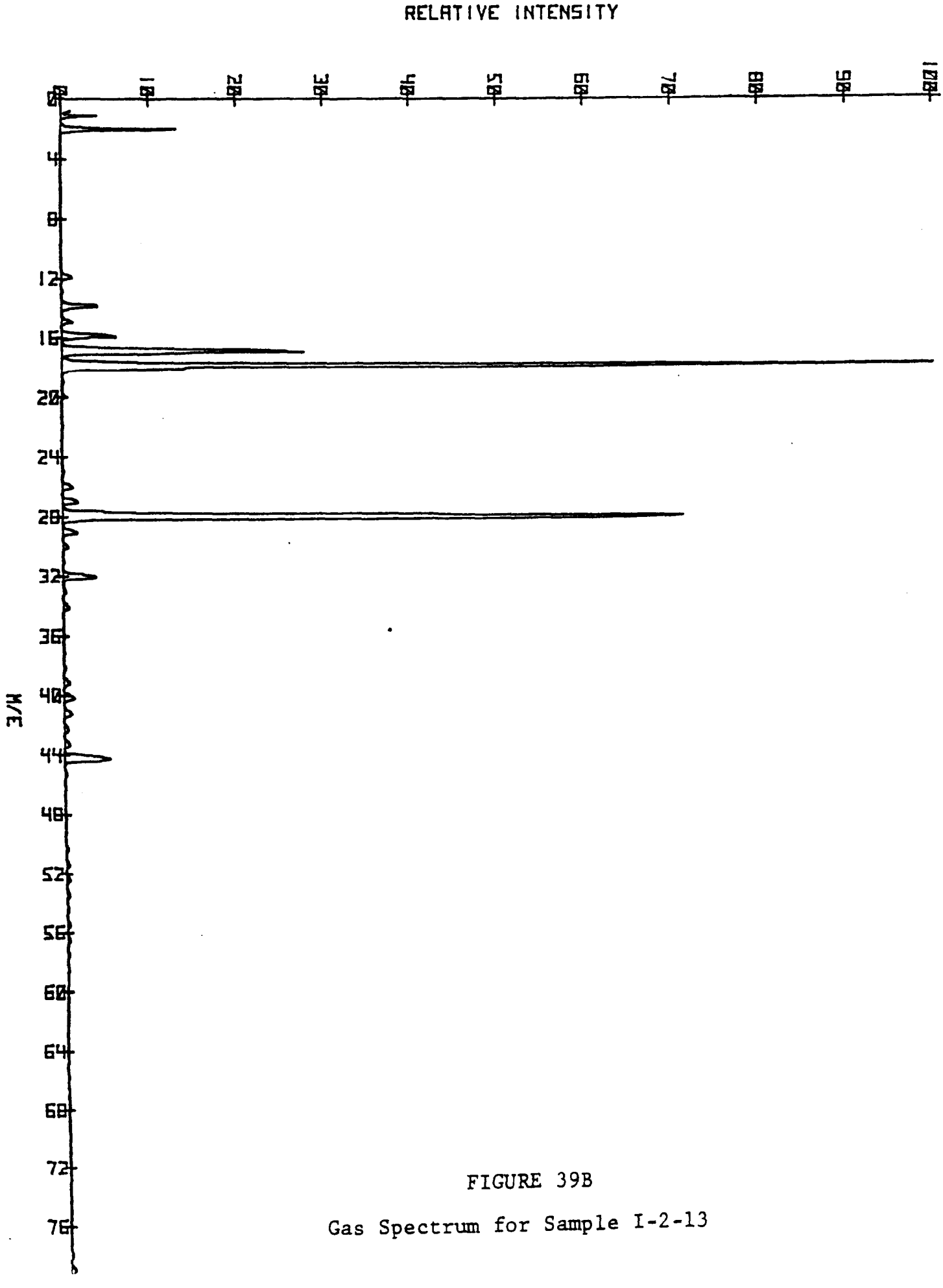



FIGURE 39B

Gas Spectrum for Sample I-2-13

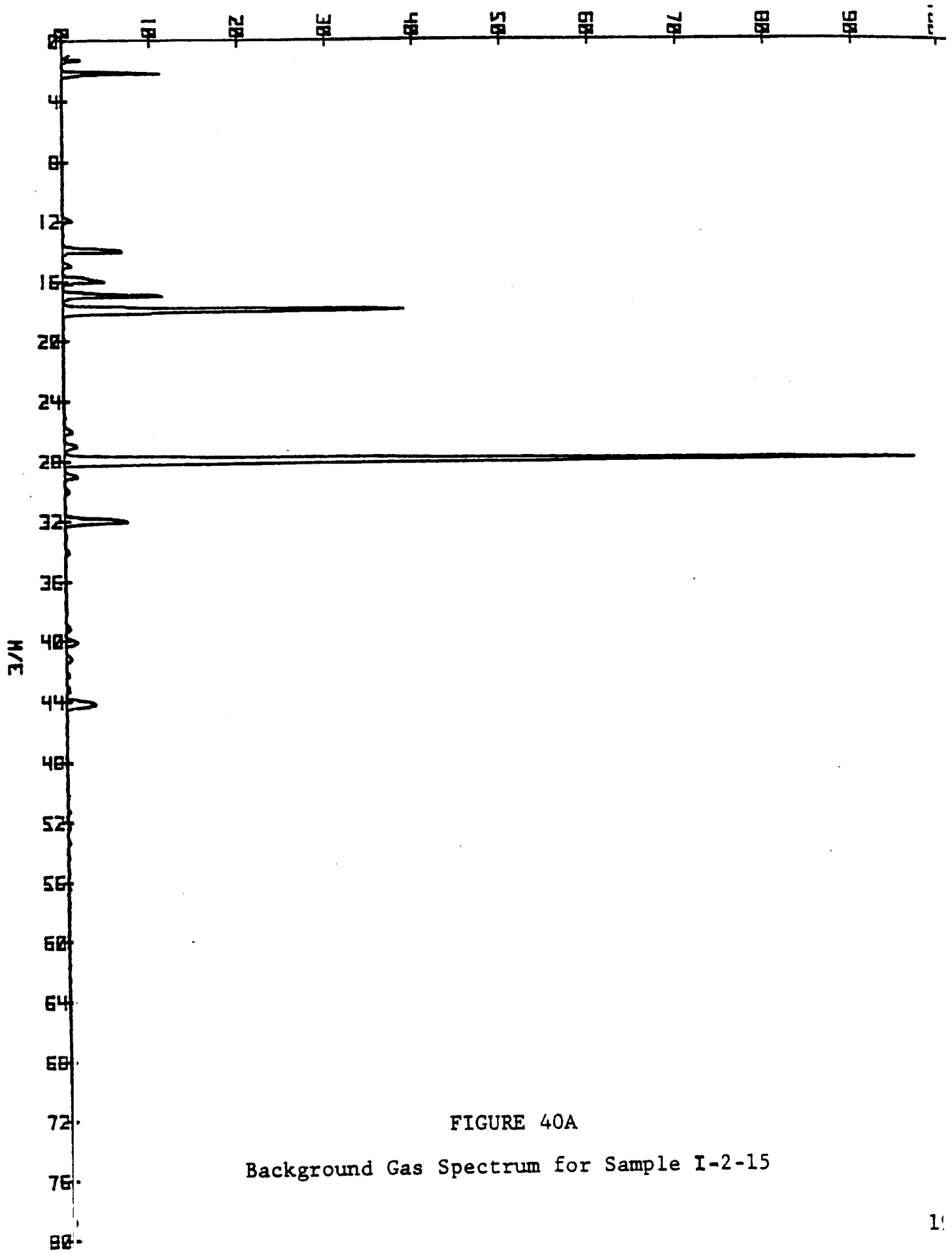



FIGURE 40A

Background Gas Spectrum for Sample I-2-15

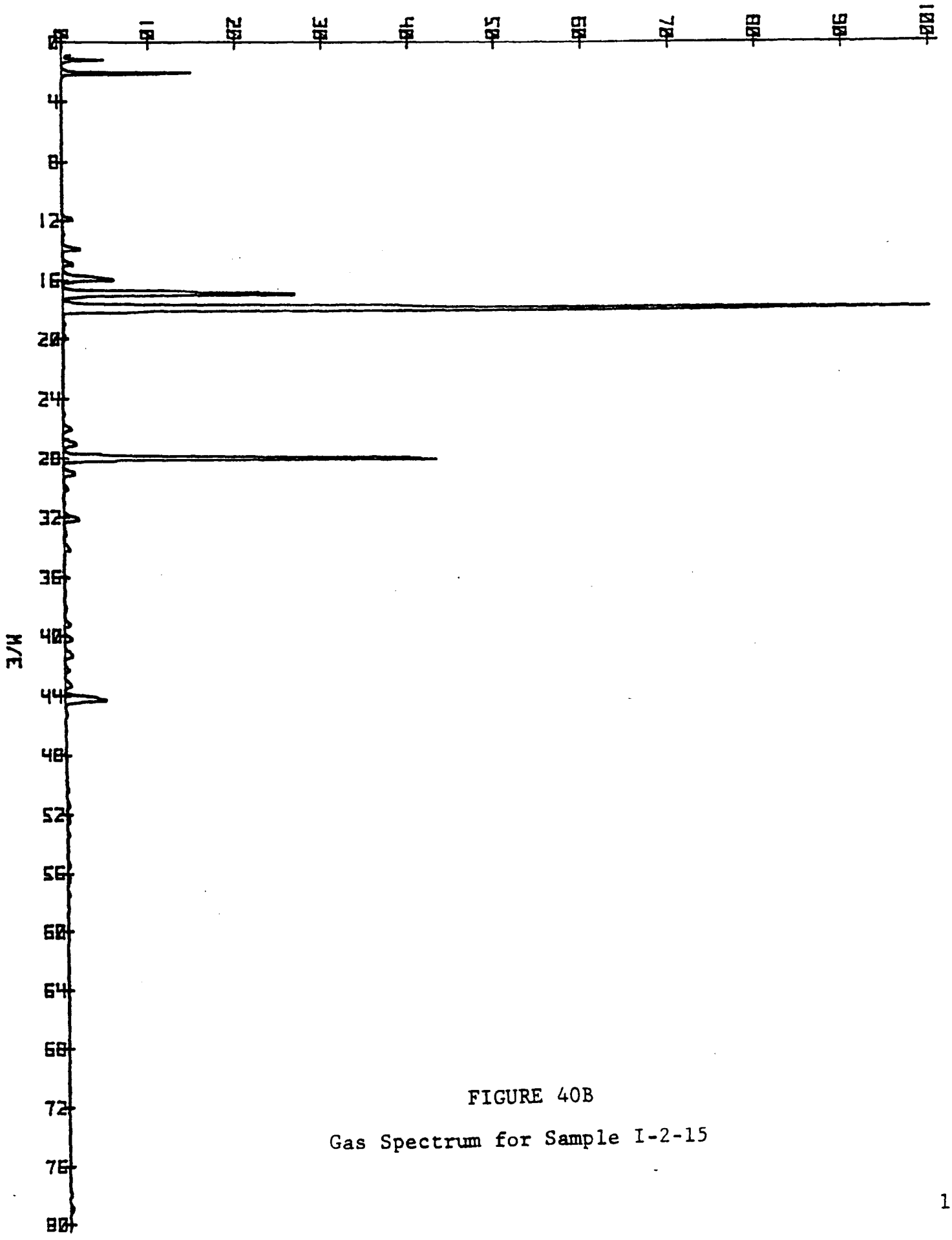



FIGURE 40B

Gas Spectrum for Sample I-2-15

dramatic shift from what had been observed in previous samples. This shift indicated that at fracture, nitrogen and oxygen were absorbed by the sample and water vapor was outgassed. This observation is being investigated further especially in light of the data that were obtained during the mass spectrometer analysis of the sample container headspace gas.

Another experiment that is in progress is one in which the effects of moisture on the mechanical properties are being evaluated. The first test conducted in this experiment involved sample KY-2-1. This sample was cut into three specimens. The first was tested in the normal nammer, while the second was exposed to a relative humidity of 44.4% for a period of six weeks prior to test. The third sample was exposed to 100% relative humidity for the same period of time. The mechanical test results for these samples are shown in Table 99. These results indicate that the sample exposed to 44.4% relative humidity had an increase in splitting tensile strength, while the one exposed to 100% relative humidity suffered a significant decrease in splitting tensile strength. The Shore hardness values of both samples decreased to approximately the same value (Table 100). The sample exposed to the 100% relative humidity increased in weight, while the one exposed to 44.4% relative humidity decreased in weight.

These preliminary results show that moisture exposure significantly affects the mechanical properties of the samples and as larger samples become available this study will be pursued further to better characterize these effects.

## O. SEM ANALYSIS

As-received samples from the VA-1, P-1 and I-2 wells have been examined. Pyrite was again found in several forms as illustrated in Figures 41 through 44. The pyrite concentration for all three wells was much greater than that previously observed in the KY-2 samples.

The fracture surfaces of the VA-1 and I-2 samples are fibrous and loosely packed (Figures 45 and 46).

TABLE 99

Mechanical Test Results for Samples Exposed to Moisture

| Sample | Relative Humidity | Splitting Tensile Strength (M Pa) |
|---|---|---|
| KY-2-1 | Control | 3.265 |
| KY-2-1A | 44.4% | 3.792 |
| KY-2-1B | 100% | 2.201 |

TABLE 100

Physical Changes for
Samples Exposed to Moisture

| Sample | Relative Humidity | Sample Weight | | Shore Hardness | |
|---|---|---|---|---|---|
| | | Before Exposure | After Exposure | Before Exposure | After Exposure |
| KY-2-1 | Control | --- | --- | 19.00 | --- |
| KY-2-1A | 44.4% | 243.47g | 243.35g | 16.55 | 10.43 |
| KY-2-1B | 100% | 275.98g | 276.41g | 17.60 | 10.40 |

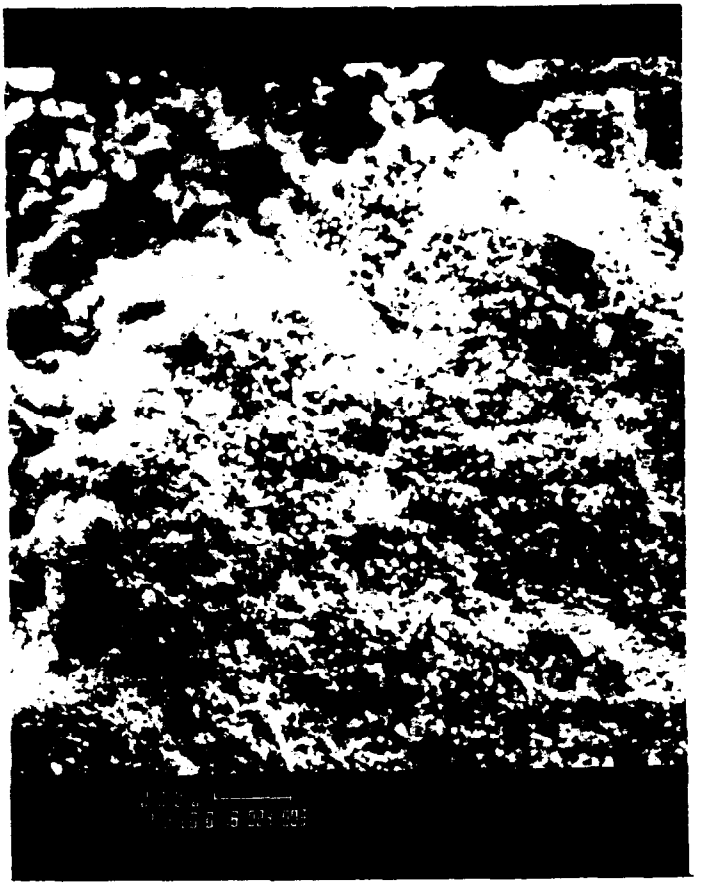


FIGURE 41

High concentrations of small loosely packed euhedral grains of pyrite were deposited in natural fractures in the samples from the I-2 well. Spherical agglomerations of pyrite were also interspersed in the pyrite matrix.

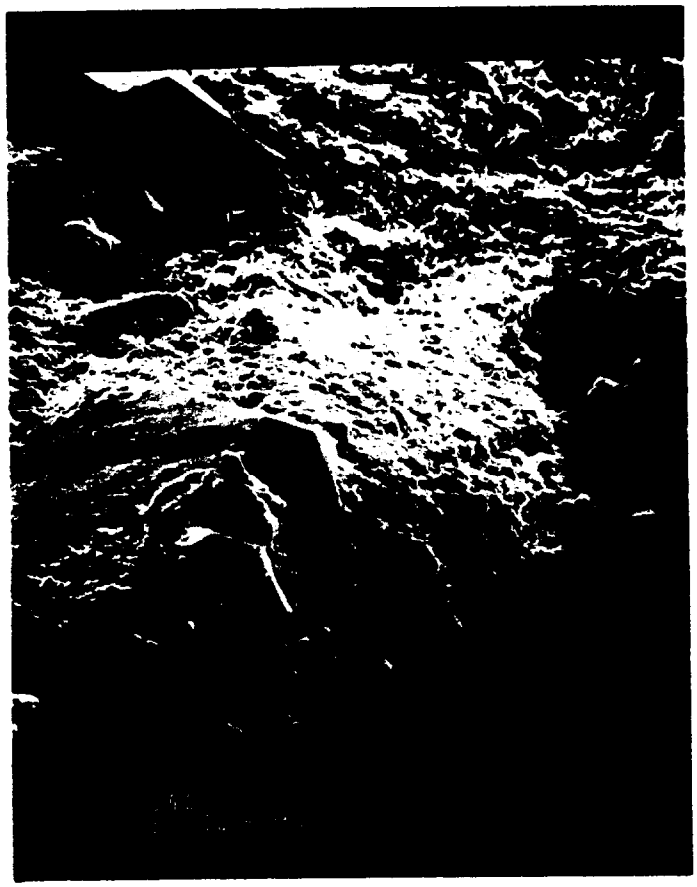


FIGURE 42

Large euhedral grains of pyrite were also tightly embedded in the matrix material of the I-2 samples.

FIGURE 43

High concentrations of small euhedral grains of pyrite were also found in the natural fractures of samples from the VA-1 well.

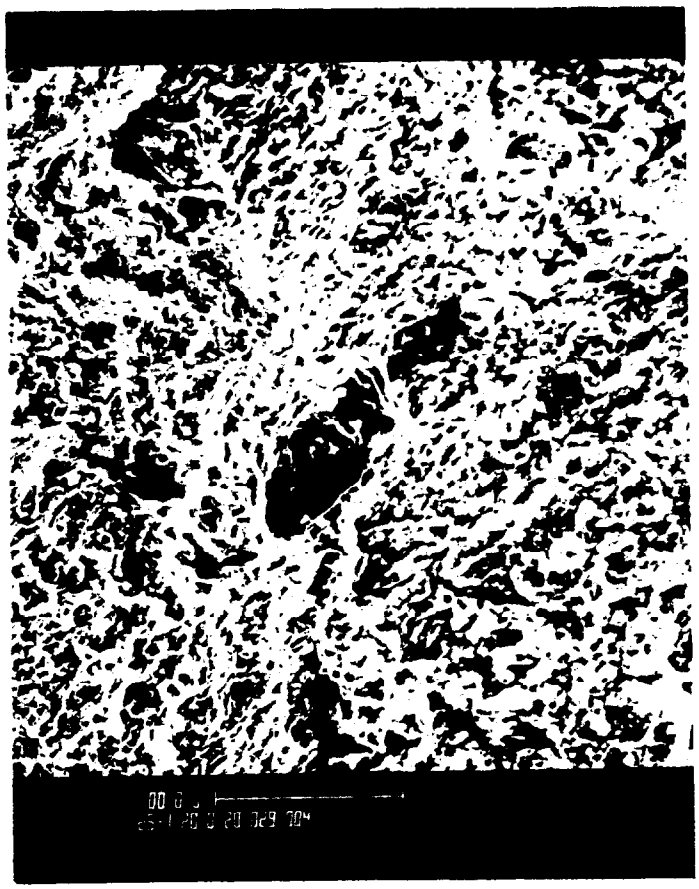


FIGURE 44

Larger, isolated euhedral grains of pyrite were also loosely embedded in the matrix material of the VA-1 well.

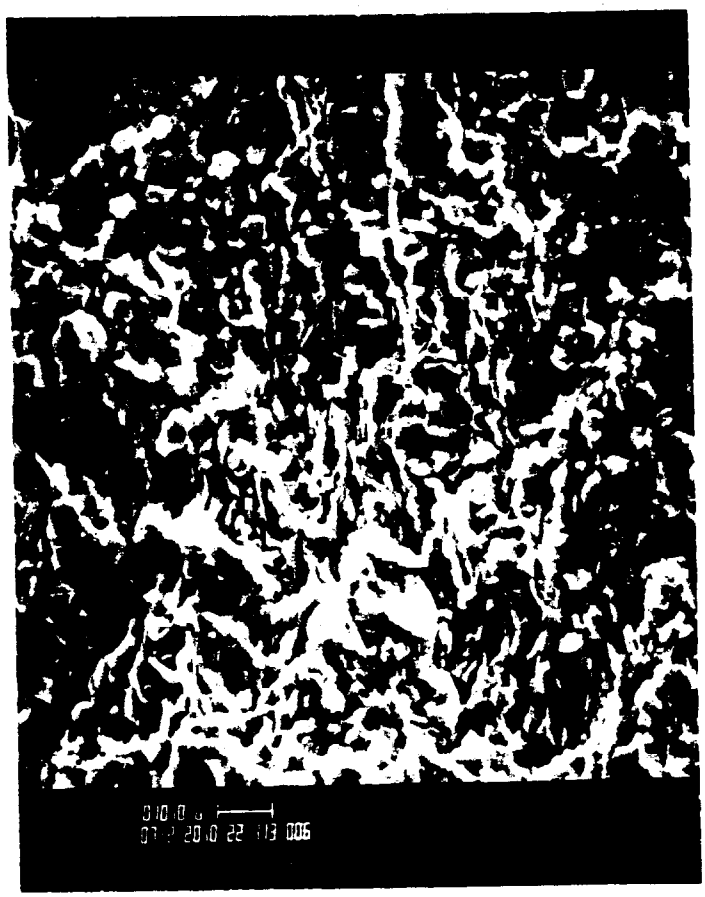


FIGURE 45

The matrix of the VA-1 samples was predominantly fibrous in nature.

FIGURE 46

The matrix of the I-2 samples was also predominantly fibrous in nature.

The samples from the I-2 well possessed calcite concentrations. The most significant concentration was observed in Sample I-2-19. A large concentration of calcite crystals was embedded in the sample matrix (Figure 47). The crystals had a wide size range distribution (Figure 48) and were well formed (Figure 49).

## P. BIOSTRATIGRAPHY

A palynological investigation was carried out on 55 samples of core material from the O-1, P-1, KY-2, and VA-1 wells. Using a combination of spores and acritarch it was possible to divide the Middle Devonian from the Upper Devonian and to make subdivisions within the Middle and Upper Devonian.

A complete report of this study has been prepared and is currently being printed and assembled.

## Q. ARCHAEOLOGICAL SURVEY

A preliminary archaeological survey of proposed gas well locations in Green Township, Scioto County, and Elizabeth Township, Lawrence County, Ohio, was completed. A letter requesting a determination of effect was subsequently submitted to the Ohio Historical Preservation Office which agreed with the results and recommendations of the survey.

The complete report of the survey has been prepared and is currently being printed and assembled.

FIGURE 47

A high concentration of calcite crystals was embedded in the matrix of Sample I-2-19.

FIGURE 48

The calcite crystals found in sample I-2-19 were randomly distributed in size. The large crystals had numerous fractures.

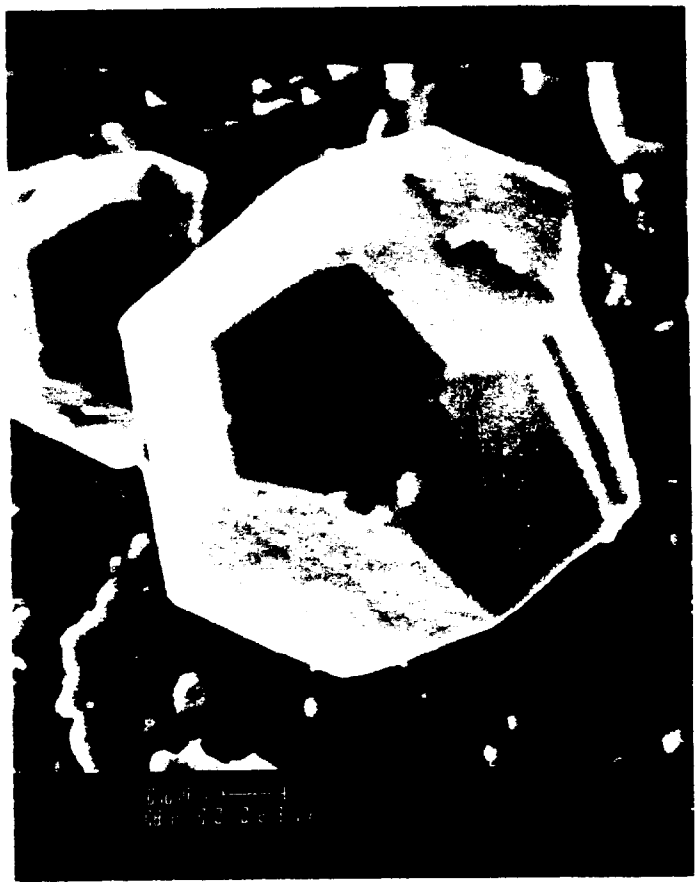


FIGURE 49

The calcite crystals were extremely well defined.