

ENERGY FROM IN SITU  
PROCESSING OF ANTRIM OIL SHALE

Monthly Technical Progress Report  
for July 1977

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## OBJECTIVE

The objective of this contract is to test the technical feasibility for the in situ processing of Antrim Shale to produce energy values.

## ABSTRACT

Final installations and preparations were made on several projects --

### Dow/Rhoburn Well Area

- ... Baseline environmental studies are being made by ERIM using the mobile air monitoring lab and other instrumentation for air, land and water studies.
- ... Equipment and instrument modifications and testing were completed for the downhole ignition test in Well #4 on Dow's early experimental site.

### Dow/ERDA Well Area

- ... Well #201 was completed and cored. This is the last of the first group of four on the south field site.
- ... Baseline seismic data through the area containing Wells #101 and 102 were obtained by ERIM in Well #100 to be used in later fracture assessment.
- ... Equipment and materials procurement and project planning were completed for the fracturing experiment in Wells #101 and 102.

Methods were developed by the subcontractors and results are being reported for geochemical and geophysical analyses and Modified Fischer Assay on samples of core from Well #100. An initial literature survey has been completed.

## ENVIRONMENTAL-SOCIO ASSESSMENT

BOTH WELL AREASEnvironmental Monitoring

The ambient air monitoring trailer has been moved to a down-wind location for monitoring the site flare stack during operation. An ERIM technician has been assigned to operate and maintain the trailer and is acquainting himself with the instrumentation. Background data is being gathered. A replacement  $\text{SO}_2$  analyzer and the arrival and installation of the Bendix Environmental Chromatograph have completed the outfitting of the trailer. All instruments are operational at this time.

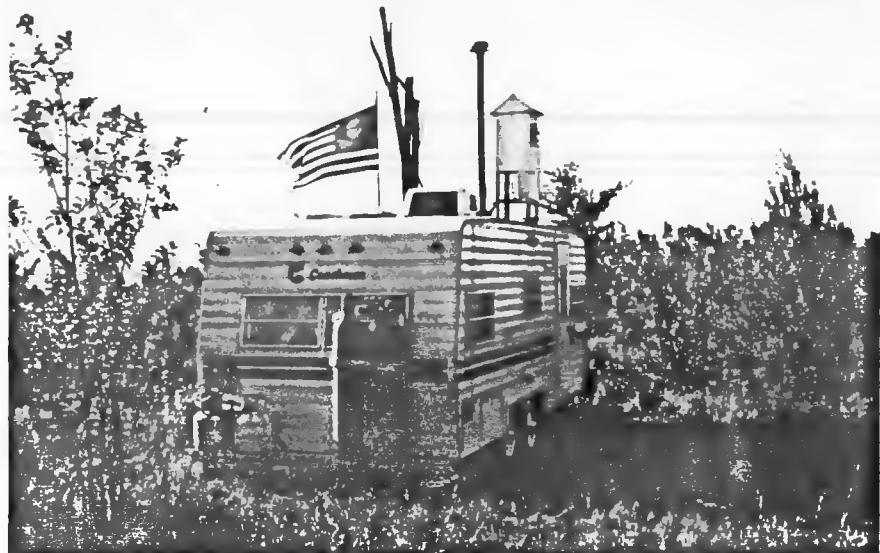
Two photographic views of the mobile air monitoring laboratory are given in Figure 77-7-1.

## SHALE CHARACTERIZATION &amp; RESOURCE EVALUATION

Resource Evaluation

Mr. Ells, Michigan Department of Natural Resources, was supplied with core descriptions and gamma ray logs from several old wells drilled by Dow. These are to be added as new data points on his stratigraphic cross sections and will help to relate his gamma ray - neutron (GR-N) log work to core data.

The Core Lab's work on fracture studies of oriented cores is nearly complete. The work on sample studies is continuing. Full or partial results from seven sites in Michigan are now on hand.



A) Laboratory in field location for sampling



B) Instrumentation in the laboratory

FIGURE 77-7-1 Views of mobile air monitoring laboratory

Petroleum Information, Inc., has reported on 3,142 exploratory and discovery wells in Michigan. They will reconstruct plotted maps in segments to match the 1:250,000 set of USGS base maps.

A basin-wide coring program for the second year of the contract has been compiled and will be submitted. It involves six wells in the Michigan Basin.

#### DOW/ERDA WELL AREA

##### Drilling & Coring

The last of the first set of four wells in the south 40 acres of the field site was completed in July, i.e., Well #201. Oriented core was cut from 1150 to 1460 feet, and conventional coring was done from 1460 feet to the total depth, 1502 feet.

The system depicted in Figure 77-7-2 was designed and set up to monitor the quantity of gases evolved from cores from Well #201. All six sections of core were taken in plastic sleeves (both oriented and conventional).

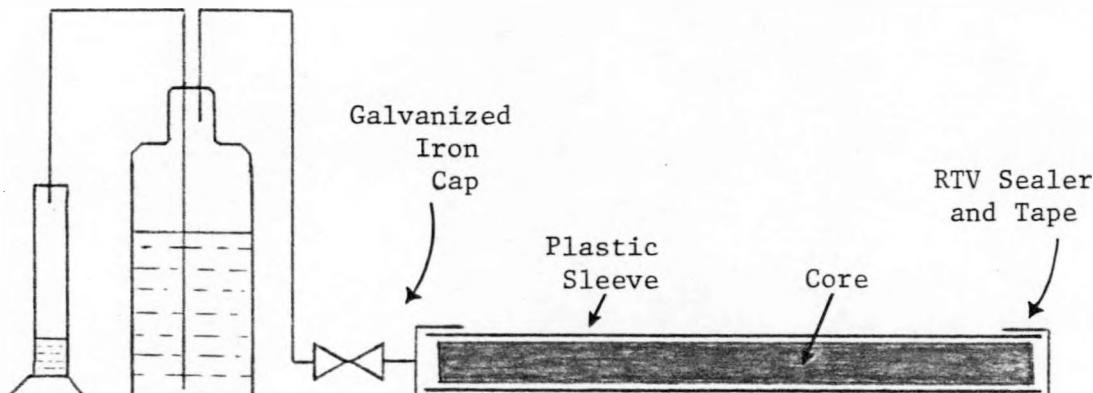


FIGURE 77-7-2      Equipment for Core Degassing Experiment

Problems with obtaining a good gas-tight system have hindered the quantity of gas evolved; however, samples of the gases from all cores show methane, ethane and propane. Specific quantitative analysis will be presented in next month's report.

## WELL DEVELOPMENT, FRACTURING &amp; ASSESSMENT

DOW/ERDA WELL AREAWell Drilling

Details for the drilling of Dow/ERDA Wells #100, 101 & 102 were given in the May and June progress reports. Well #201 was completed in July. It was drilled to a total depth of 1502 feet. The well and logging details are much like those of Wells #101 & 102.

Drilling permits for Dow/ERDA Wells #103 through 106 have been received from the Michigan Department of Natural Resources. Plans are to start these in September, 1977.

Figure 77-Q2-3 shows the location of the Dow/Rhoburn wells on the north 40 acres, and Figure 77-Q3-4 shows the relative locations of the new Dow/ERDA wells on the south 40 acres with respect to the existing Dow/Rhoburn wells.

Fracturing

Technical details have been assembled for the August hydro-frac experiment. The following companies and personnel will be involved:

Dow Oil Shale Research Group - Project Management

Dowell Field Services - Engineering, Fracturing,  
Materials and Services

Dowell Research - Planning and Advising

Lease Management - Service Rig and Crew

Baker Oil Tools - Injection Packer and Service

A complete description and evaluation will be presented after the field activity is complete.

NORTH

POWER  
LINEINSTRUMENT  
SHOPO  
#2

Dow/ERDA Oil Shale Project  
Field Site in Sanilac Co.,  
Michigan, N. 40 Acres  
Scale: 1" = 100'

#10

SEPARATORS

COMPRESSOR  
SHED

TRAILER

FENCE

LINED  
POND

#9      #8

#4

#7

#3

#5

#6

Early  
(Dow/Rhoburn Wells)

WELLS AREA

#12

OFFICE

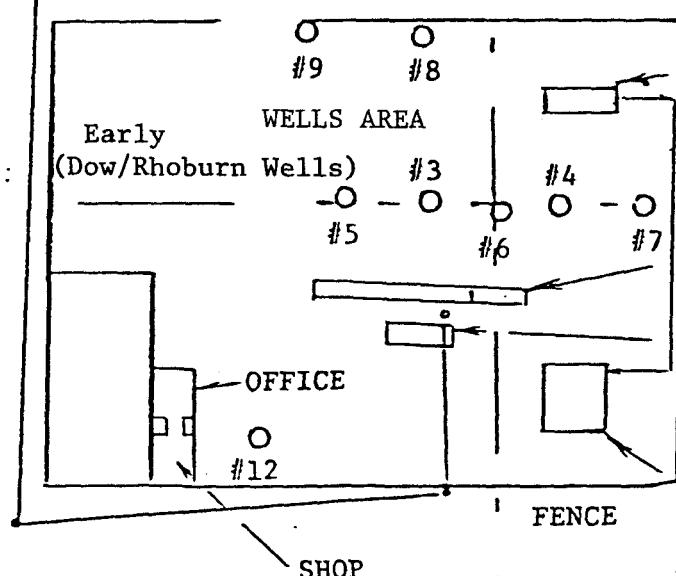
NEW WELLS AREA  
(Dow/ERDA Wells)

Figure 77-Q2-3 Major surface features on  
north 40 acres of the field  
site.

O  
#1O  
# Zero

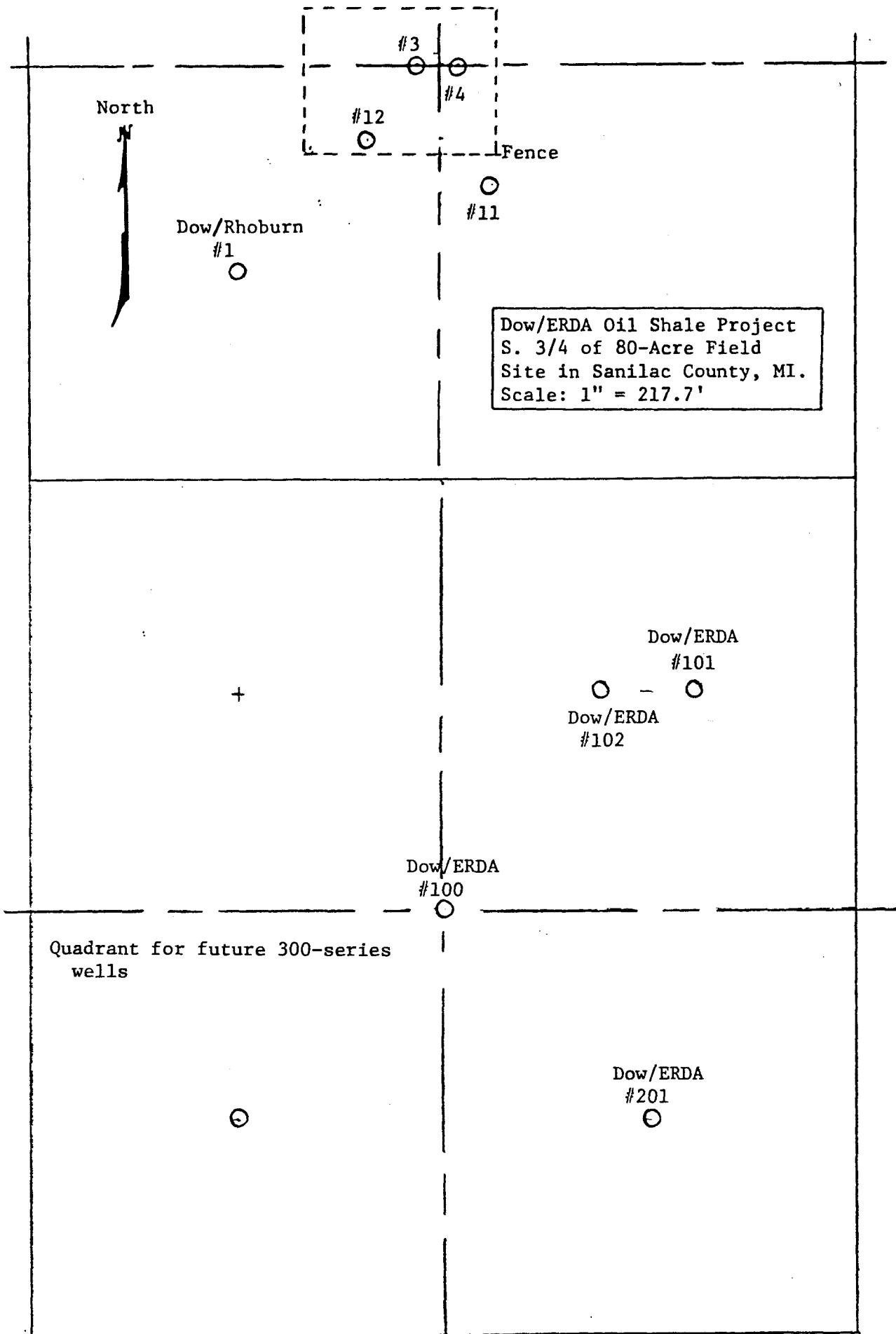


FIGURE 77-Q3-4 SKETCH OF SOUTH 40 ACRES SHOWING RELATIVE LOCATIONS OF SEVERAL EXISTING DOW/RHOBURN WELLS AND PROPOSED LOCATIONS OF NEW DOW/ERDA WELLS.

Fracture Assessment

Environmental Research Institute of Michigan (ERIM) personnel completed baseline seismic testing in early July. The seismometer was lowered to various levels in Well #100 to measure shock waves generated at the surface in a line north, northeast of the well through the ten acre section containing Wells #101 and 102. This is the area where hydrofracing is to occur.

## PERMEABILITY MEASUREMENT &amp; HYDROCARBON EXTRACTION

DOW/RHOBURN WELLSGeneral Site Activity

The power take-off bearing was replaced on the V-6 diesel compressor engine.

Brine Removal

Approximately 8,900 gallons of brine were removed during July compared to 5,700 gallons removed in June. Over 85% of the brine removed was from Well #3. The location of the Reda pump in Well #3 provides efficient drainage for Wells #3 through #6. Wells #7 through 12 show only low brine re-entry rates. The brine level in Well #4 was found to be fairly constant at 1315 feet. Tubing in Well #4 extends to 1311 feet.

Permeability Measurement & Precombustion Logging

It was reported in June that Well #4 had been selected for the summer combustion tests. Permeability studies were completed in early July. The air injection tests have been described in detail previously. Results from a number of other tests are given below. Additionally, a series of oil shale porosity and permeability studies were performed by Systems, Science and Software, Inc. Their results will be reported when received.

Spinner Sibilation Surveys

Wellbore sibilation and spinner tests were conducted in late June to obtain information on air flow between Well #4 and Wells #6 & 7, e.g., which horizons within the lithological zones of interest have sufficient fracture porosity to be good zones for burner location.

The spinner test was to show via a type of propellor movement where air was entering Well #4; however, the particular equipment used was not sensitive enough to indicate a flow of 75 SCFM entering two feet above it, so further spinner tests were discontinued.

The Reda pump had been transferred to Well #3. Wells #4, 6 & 7 were pressured to push brine over to Well #3.

Well #6 was found to have a brine level of 1098 feet downhole which was up in the cased portion, so Well #6 was eliminated from the survey.

The sibilation survey was run in Well #4 to detect points of entry of air from Well #7. The well sibilator is an extremely sensitive listening device. When it is lowered past a zone where air or gas is flowing into the well, it gives a characteristic indication which can be heard and recorded. Figure 77-7-3 shows the recorded signal and a description of the audible signal alongside the caliper log for Well #4. These results were used with the results of the Nuclear and Seis-viewer Logs to select two zones of potential interest for burner location, viz., 1300 feet and 1220-1224 feet.

#### Nuclear Log

Figure 77-7-4 gives the Gamma Ray and Neutron Profiles for the Nuclear Log over the section of interest. An increase in gamma ray radiation is thought to be indicative of zones rich in organic matter. On this basis, the zone from 1215 feet to 1250 feet would be considered to be the richest in kerogen.

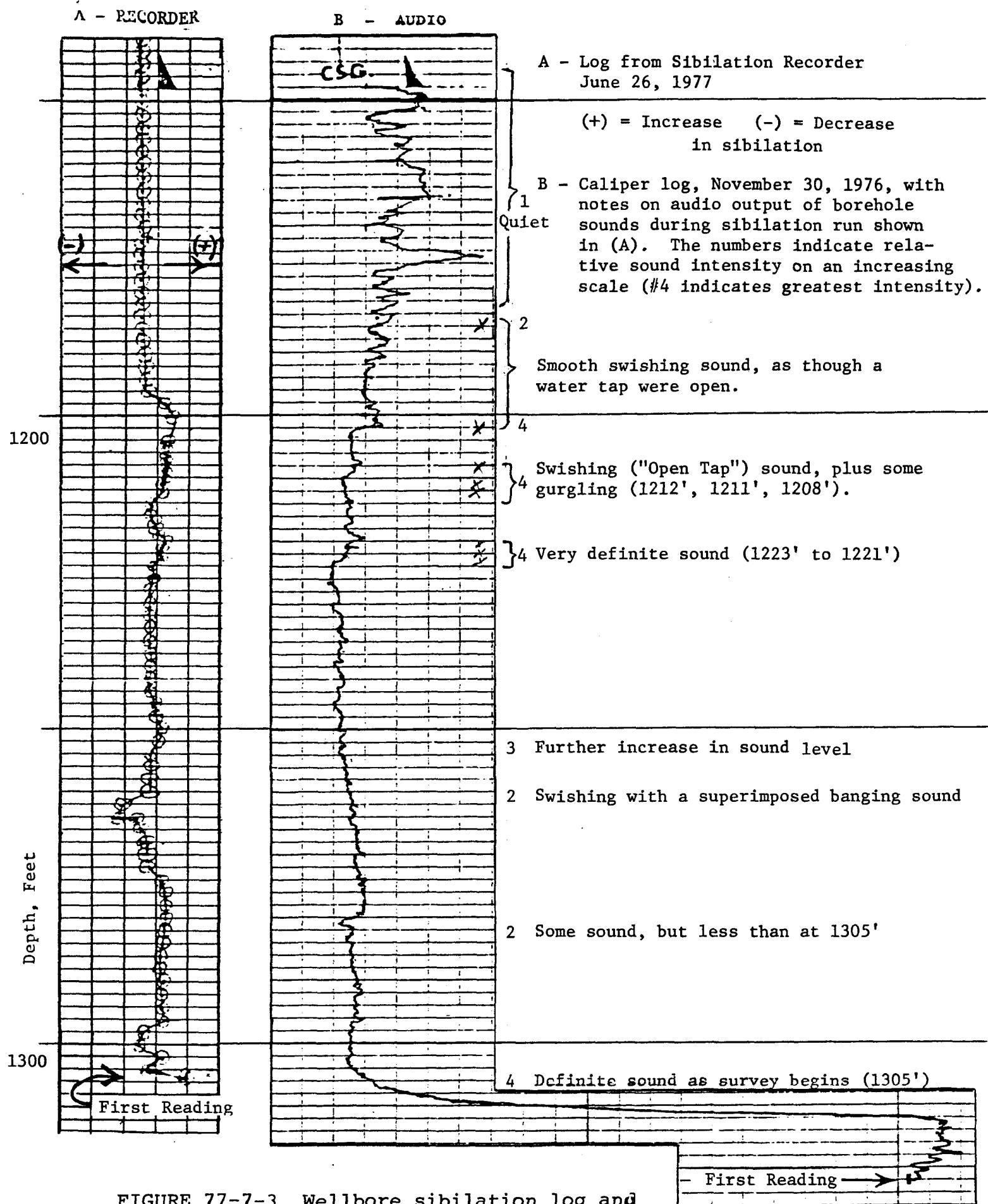


FIGURE 77-7-3 Wellbore sibilation log and caliper log with comments on audio output from sibilation Dow/Rhoburn Well #4, June 26, 1977

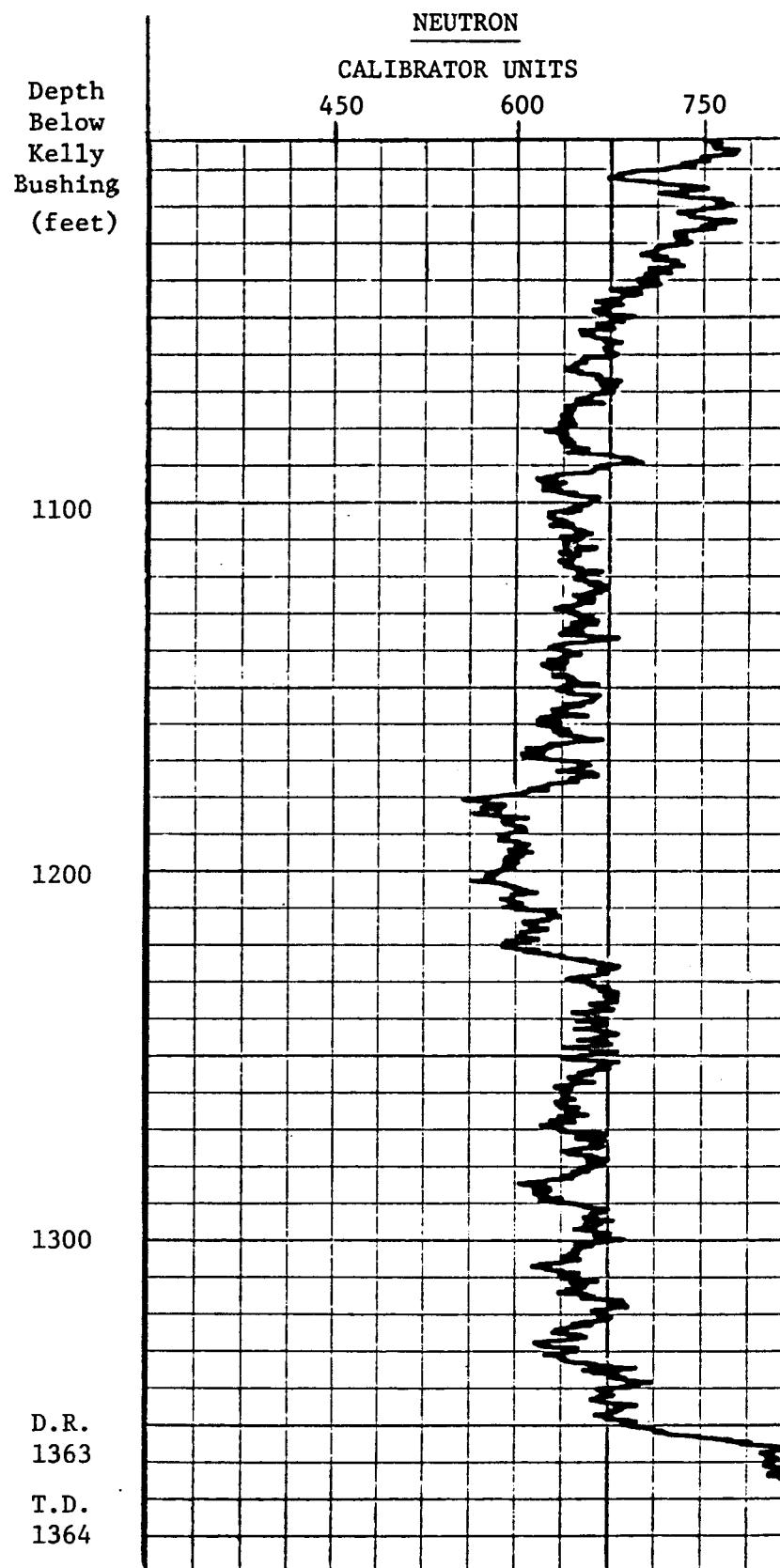
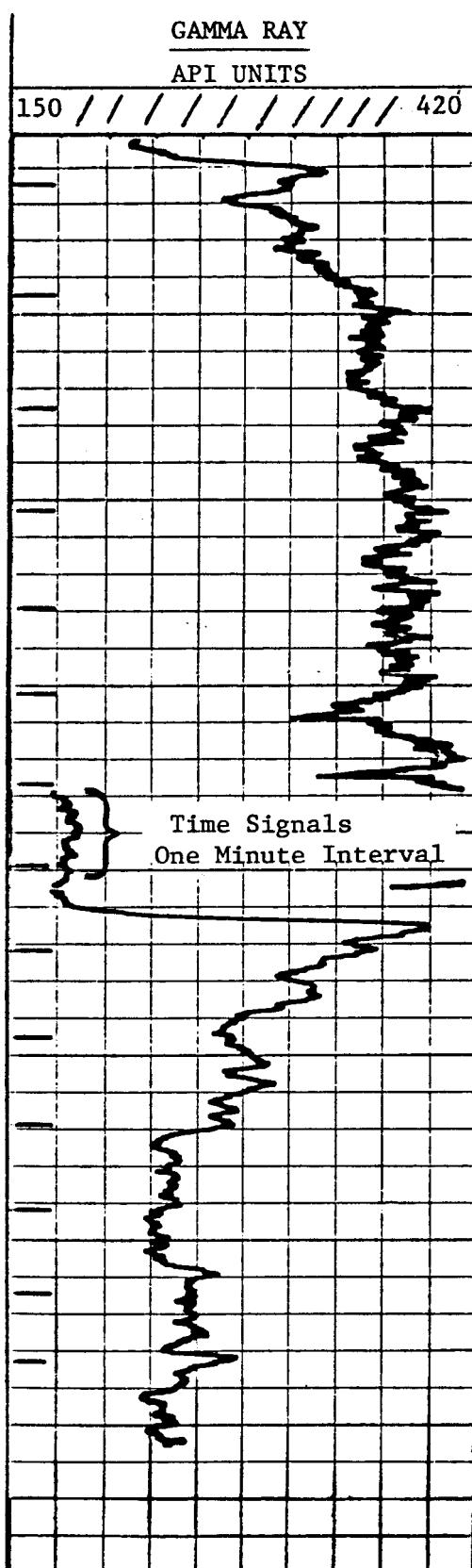


FIGURE 77-7-4 Nuclear Log, Dow/Rhoburn Well #4 - 6/21/77

Seisviewer Log

Figure 77-7-5 shows the Seisviewer Log from Well #7. Definite vertical fracturing is shown between 1310 and 1318 feet below ground level.

Tracer Injection

Further permeability studies were conducted during July by Systems, Science and Software, Inc., using several tracer gases. The results will be reported when received.

Downhole Ignition Experiment

Sandia Labs personnel have moved an instrument trailer/field office to the site. They have completed installation of a downhole vertical array of resistivity measurement instruments in Dow/Rhoburn Well #6 at 5 foot intervals between 1224 and 1324 feet, and they have installed a temperature/pressure instrumentation string in Well #7.

Surface resistivity will not be measured. It was decided that surface resistivity data would be unlikely to be of value because of the depth of the layer being burned (1200-1450 feet), the steel cased wells in the area, and the presence of ground level fencing and piping.

Other final preparations that were made for the ignition experiment were as follows:

- A new control system for feeding air and nitrogen to the burn well was built and modifications to the instrument panel were completed.
- The liquid propane pumping system was tested and the nitrogen purge system is ready.

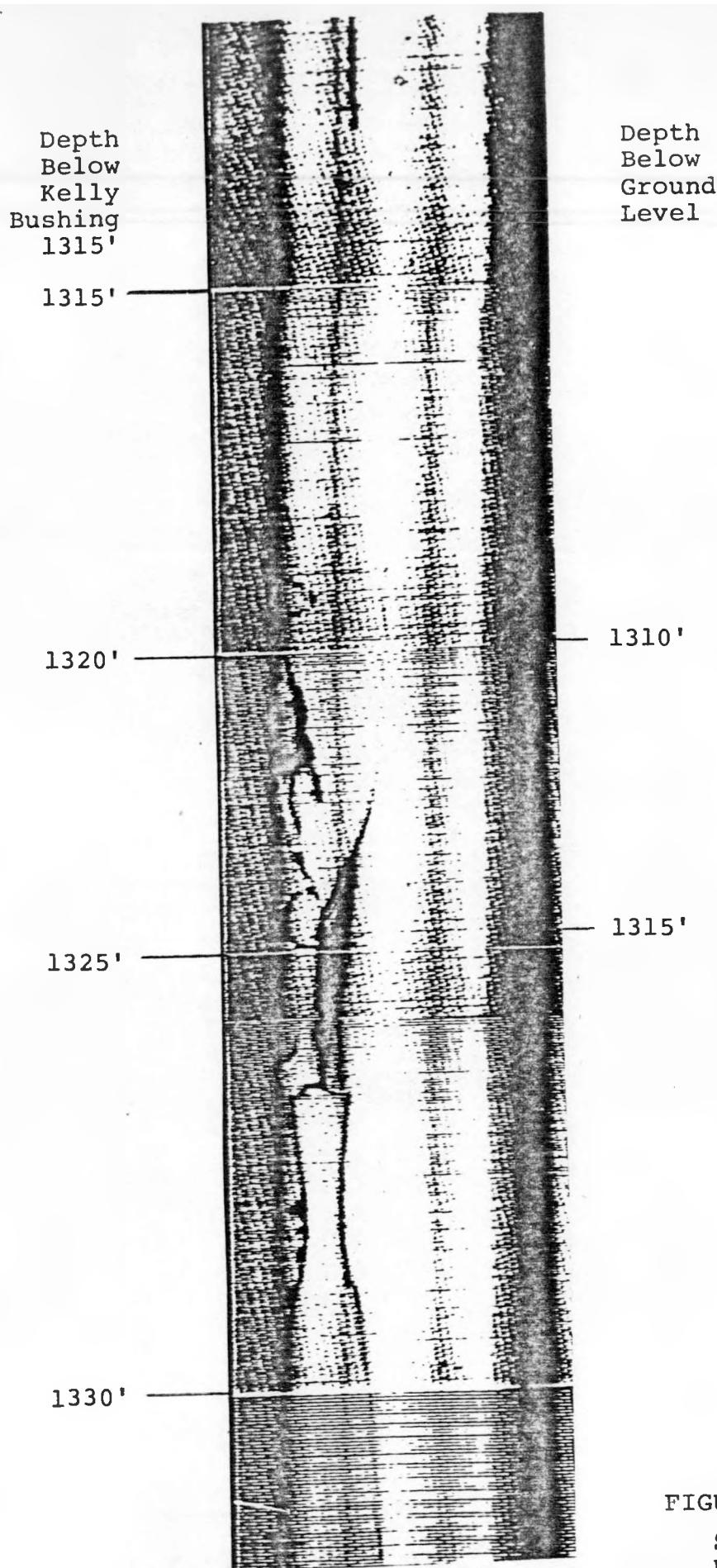


FIGURE 77-7-5

Seisviewer Log, Dow/Rhoburn  
Well #7 - March, 1974

- A dry gas meter was received for use on calibrations of nitrogen and air flow.
- Instrumentation for the electrical heater system was recalibrated.
- Operating instructions for both the electric and the propane/air heaters were written.
- The two electric heaters were tested aboveground and found to be in proper working order.
- The production gas monitoring system was modified to include two heat exchangers ahead of the gas/liquid separator. Figure 77-7-6 is a schematic representation of this system.

After the final safety inspection and any resulting adjustments are made, the electric heater will be lowered into Well #4 and the system pressure tested with air. Then all wells will be purged with nitrogen, and gas flows will be measured and recorded. Brine levels will be monitored and the wells evacuated by nitrogen pressure or pumping during the ignition experiment. The electric heaters will be started and brought up to temperature slowly with careful monitoring.

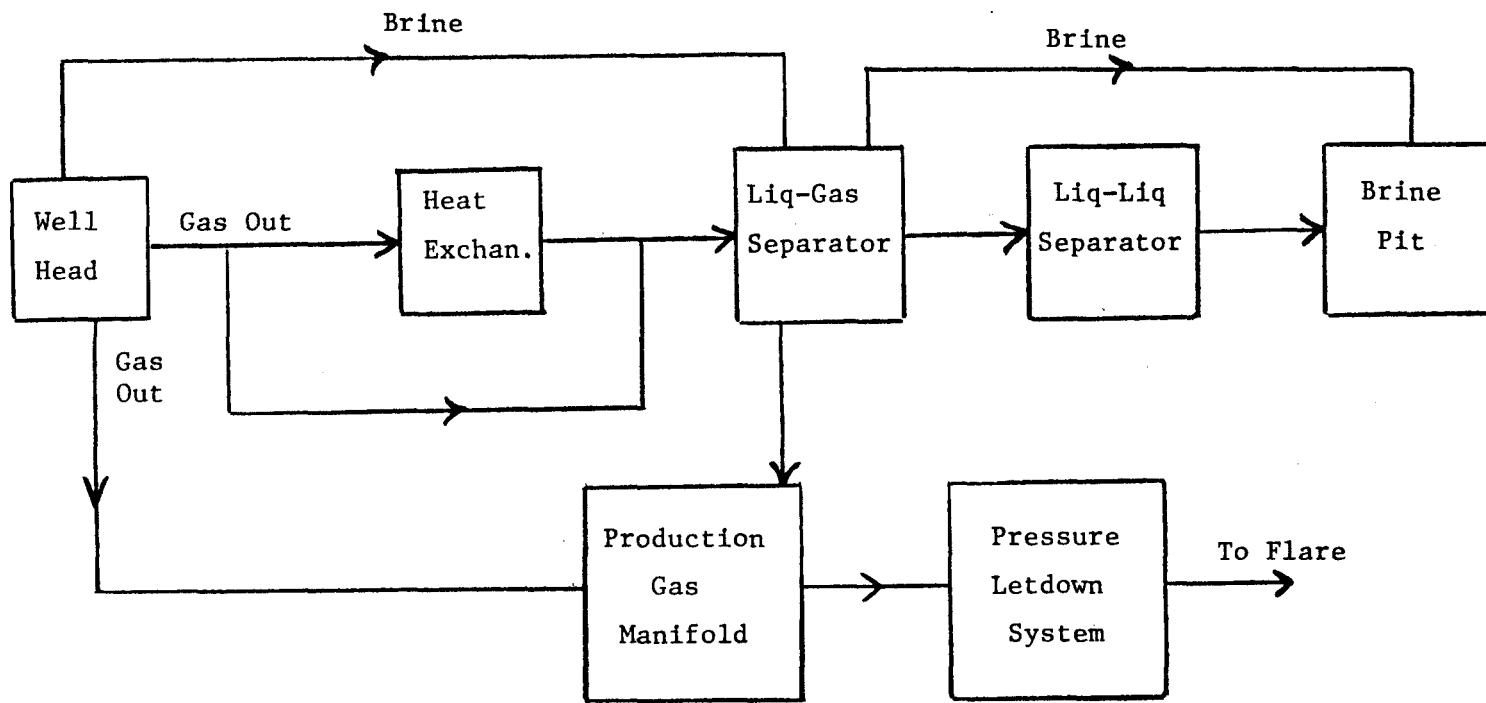


FIGURE 77-7-6 Production Gas Monitoring System

## SUBCONTRACT ACTIVITY

ENVIRONMENTAL

Most of the necessary baseline environmental measurements were made in July by ERIM. By category, these included measurements or action on the following:

## Land --

- .. Vegetative survey
- .. Ground photographs
- .. Aerial photographs, regular color & infrared color
- .. Resistivity near the lined brine pit
- .. Twenty-three more subsidence monuments
- .. Soil samples, NE and ENE of the stack

## Air --

- .. Mobile air monitoring lab moved to sampling location for taking background measurements. Generally, background levels of  $\text{SO}_2$ ,  $\text{NO}$ ,  $\text{NO}_x$ ,  $\text{CO}$ ,  $\text{CH}_4$  & particulates are low
- .. On-site monitoring time will be limited to only that necessary for accurate and continuous data collection

## Water --

- .. One more V-notch weir installed -  
Seymour Creek south of Mortimer Line Road
- .. Additional water samples taken
- .. Preliminary analyses were reported for samples taken  
June 28 & 29 and July 21, 1977

SHALE CHARACTERIZATIONStratigraphic Cross Sections

A portion of the stratigraphic cross sections being prepared by the Michigan Department of Natural Resources was drafted, reduced to a vertical scale of 1" = 100 feet, and a transparency and a paper copy made and submitted to Dow. Certain additions and adjustments were discussed. These have been built in to the portion submitted and will become standard practice on other cross sections now being drafted. Progress is as follows on these:

<u>Cross section</u>	<u>No. of wells</u>	<u>No. of parts in the presentation</u>	<u>Status of drafting</u>
A	22	4	In progress
B	21	4	Nearly complete
C	20	4	In progress
D	--	-	Not laid out
E	--	-	Not laid out
F	--	-	Scheduled

Geochemical/Geophysical Analyses

A method was developed at Michigan Technological University for semi-quantitative x-ray mineralogical analysis. Standards were prepared and the samples from Dow/ERDA Well #100 were analyzed. Another core will be analyzed in August.

Good progress was made on lithologic characteristic measurements. Rock mechanics tests were taken and a number of Antrim shale characteristics were compared with Western oil shale. A number of porosity and permeability determinations were made on one inch long by 0.53 inch diameter core samples from Well #100. These results are being summarized. Preliminary retorting tests have also been run. The retorted samples had very low strength.

Geochemical Analyses

Carbon, nitrogen, hydrogen and oxygen analyses have been completed on the samples of the core from Well #100 at Michigan Technological University. The analysis of water by the Karl Fischer method has been started on selected samples. Sulfur analyses have begun.

Data showing the precision of the carbon-hydrogen-nitrogen analyses are presented in Table 77-7-1.

The volatiles analyses (80°C at <1 mm Hg absolute pressure) have been completed on the first core. The weight loss, 1-3%, was primarily water, but a trace of oil was found in the volatiles.

A method has been developed, and benzene extraction of bitumens is underway.

Nine of the low temperature ashing tests have been completed. Modifications to the procedure were introduced to assure complete ashing and to minimize water absorption during weighing. Table 77-7-2 shows a summary of the results.

Modified Fischer Assay

New size reduction equipment and Fischer Assay furnaces and retorts were received at the College of Engineering Energy Center at Wayne State University (CEEC, WSU). Progress was made in improving the reproducibility of analyses on Antrim shale samples from the Paxton Quarry. The CEEC and the Laramie Energy Research Center (LERC) cooperated on this. The following results were reported from ten samples analyzed:

TABLE 77-7-1  
CARBON-HYDROGEN-NITROGEN PRECISION TESTS

Duplicate Runs

<u>Sample #</u>	<u>Date Run</u>	<u>% C</u>	<u>% H</u>	<u>% N</u>
AC8-1170.2	6/30/77	0.84	0.67	0.13
AC8-1170.2	7/25/77	0.84	0.71	0.16
AC9-1230	6/30/77	7.22	1.01	0.16
AC9-1230	7/25/77	7.18	0.99	0.22
AC8-1280	6/30/77	5.45	0.57	0.11
AC8-1280	7/25/77	5.42	0.81	0.13
AC12-1400.2	7/11/77	3.77	0.76	0.23
AC12-1400.2	7/25/77	3.80	0.75	0.21
AC12-1410.1	7/11/77	9.26	1.20	0.35
AC12-1410.1	7/25/77	9.28	1.15	0.34
AC12-144.2	7/14/77	3.97	0.34	< 0.01
AC12-144.2	7/25/77	3.92	0.33	0.03

Replicates on One Sample

AC11-1350.5	7/11/77	10.51	0.29	0.22
AC11-1350.5	7/11/77	10.50	0.29	0.16
AC11-1350.5	7/11/77	10.48	0.28	0.0
AC11-1350.5	7/11/77	10.47	0.26	0.0
		_____	_____	_____
	Average Value	10.49	0.28	0.10
	Standard Deviation	0.02	0.01	0.11

TABLE 77-7-2  
LOW TEMPERATURE ASHING RESULTS

<u>Depth, ft.</u>	<u>Weight Loss %</u>	<u>%C</u>
1160	0.32	0.85
1170	0.59	0.84
1180	0.92	0.92
1190	-0.08% (gain)	0.88
1200	0.15	0.84
1210	7.76	8.15
1220	9.62	9.77
1230	7.05	7.22
1240	9.26	9.17
Outcrop*	8.77 $\pm$ 0.37	8.63

\*Average of Seven Ashings

## Oil Yield (gallons/ton of shale)

Mean..... 6.79  
Standard Deviation.... 0.30

## Water Yield (gallons/ton of shale)

Mean..... 5.3  
Standard Deviation.... 1.9

## Gas Plus Loss (%)

Mean..... 1.2

## Specific Gravity

Mean..... 0.927

Steps are being taken to improve the water analysis reproducibility.

Kinetic & Thermodynamic Parameters

An initial survey of the literature has been completed at CEEC, WSU. Over 100 papers have been tabulated. This activity will continue throughout the project life. The documents in hand have been scanned and a subset has been studied in detail. Preliminary notes on twenty-two papers in the subset have been compiled.

Work is progressing on all tasks in this area.

FRACTURE ASSESSMENT

The ERIM field trip for collecting baseline seismic data was completed. Using a concrete pad for the cannon baseplate gave a much improved quality SH wave. After retrieving the down-hole geophone, it was dismantled and a serious malfunction was found in the mechanism that stops the wall locking action. The instrument has been returned to the manufacturer for repair.

Playback of the digital magnetic tapes from the first field trip was begun. The SH wave showed good strength.

A rapid reversing, silent shear wave generator will become available later in the summer. The seismic measurements for the north 40 acre wells have been postponed until fall to take advantage of the features of the silent shear wave generator.