

ENERGY FROM IN SITU  
PROCESSING OF ANTRIM OIL SHALE

Quarterly Technical Progress Report  
for April - June, 1977

John P. Humphrey

NOTICE  
This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

Published - July 19, 1977

Under Contract No. EX-76-C-01-2346

Prepared for:

ENERGY RESEARCH & DEVELOPMENT ADMINISTRATION  
Washington, D. C. 20545

Hydrocarbons & Energy Research Department  
THE DOW CHEMICAL COMPANY  
Midland, Michigan 48640

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

EB

## **DISCLAIMER**

**This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.**

---

## **DISCLAIMER**

**Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.**

## OBJECTIVE & SCOPE OF WORK

The objective of this contract is to test the technical feasibility for the in situ processing of the Antrim oil shale within the Mississippian-Devonian black shale sequence of the Michigan Basin to produce energy values. The field investigations will involve well development, fracturing, underreaming, fracture assessment, in situ ignition, and extraction of hydrocarbons. Supporting contributions will be made by subcontractors in the areas of shale characterization, environmental measurement, fracture assessment, and public policy.

## ABSTRACT

Three mineral wells were drilled and cored in the Dow/ERDA series in the south forty acres of the developmental field site. Subcontractor studies on the cores have begun. The Environmental Monitoring Plan has been prepared by the Environmental Research Institute of Michigan.

Air injection permeability tests were completed for the Dow/Rhoburn wells, and Well #4 was selected for the in situ ignition tests.

## SUMMARY OF PROGRESS TO DATE

The effective date of the four-year contract for this project is September 30, 1976. An initial 120-day letter contract, later extended to April 15, 1977, authorized limited work and preparation of an environmental research plan and it provided for negotiation of the complete contract. During the first quarter, the Sanilac County site was reactivated after having been shut down for 18 months. Negotiations continued into the second quarter and the major subcontracts for service and materials were developed and approved. The contract was signed by Dow on March 31st and by ERDA on April 11, 1977.

During the second quarter, increased attention was given to procurement, inventory building, and the development of procedures and facilities for the maintenance of instrumentation, equipment and personal safety. The principal field effort was to prepare the surface piping network and equipment and to rig the existing Dow/Rhoburn wells for brine removal. Some 48,000 gallons of brine were removed, and reentry rates decreased significantly. Air injection tests to measure the flow pattern in the present well system were begun in March and continued to mid-June. During this time, another 13,000 gallons of brine were removed.

Well #4 was selected as the site for an in situ combustion test planned for August. Two heater systems have been constructed: a propane/air burner system and an electric heater system. The mobile air monitoring laboratory was further outfitted and tested. It will be transferred to the control of the Environmental Research Institute of Michigan in July for background measurements of air quality.

Three wells in the new Dow/ERDA series in the south 40 acres were drilled, cored and logged. Well #100, completed to 2600 feet, will be used for seismic observations. Wells #101 and #102, drilled 200 feet apart to a depth of about 1550 feet, will be used for the fracturing and explosive underreaming experiments. The seismometer to be used for fracture assessment in the wells has been successfully tested at a depth of 2500 feet.

## PROGRAM MANAGEMENT

### Professional & Public Relations

The full staff made a field trip on May 16th to Alpena, Michigan, and observed several surface outcrop exposures of Antrim shale to gain further understanding of the nature of the rock. Fracture systems, bedding planes, fossils, concretions and weathering characteristics were observed.

Area news and TV people were hosted on the site May 19th. A slide presentation and tour of the facilities and area were given. Project descriptions have been given to a number of area schools and clubs. Good media coverage has resulted from these efforts.

Technical papers were presented at the following meetings:

- ... Society of Petroleum Engineers at Indianapolis  
--Twenty-one years of activity by Dow with the Antrim shale.
- ... American Nuclear Society at Boulder, Colorado  
--General properties of, and opportunities with, Eastern black shale.
- ... Eastern Gas Shales Project on Casing & Sampling at Morgantown, West Virginia  
--Use of plastic sleeves in the operation of coring shale.

### Industrial Health & Safety

Regular safety meetings and detailed monthly safety inspections were held. Areas needing attention were written up and corrective action taken.

The design of the electric and propane burner systems was discussed with the Reactive Chemicals Review Committee. Members of Division Safety, Research Safety and the Division Safety & Loss Prevention Departments visited the field site to review safety and fire protection.

Arrangements have been made for major and minor medical treatment in case injuries are incurred by field site personnel. Vehicle safety continues to receive emphasis.

## SHALE CHARACTERIZATION & RESOURCE EVALUATION

### Drilling & Coring

Drilling of the Dow/ERDA wells in the south area of the Sanilac County field site was begun in mid-May and three wells were completed. A meeting was held earlier with all subcontractors concerned with handling or testing cores to review coring and sampling procedures. Three wells were cored and a total of 1403 feet of core was cut. Of this a total of 956 feet was plastic sleeve protected, oriented core. Recoveries have exceeded 90%. The remaining 447 feet of core was cut conventionally for stratigraphic purposes. All core has been photographed, logged by gamma ray equipment, described in the field and boxed. Preliminary fracture studies of the oriented core have been completed. The detailed description of the oriented core from the first well has been completed by Dr. Briggs of the University of Michigan, and the core was shipped to Michigan Technological University.

### Resource Evaluation

Drill cuttings through the Devonian shale sequence are being prepared in "library" sets from the Dow/ERDA wells and from three others in the Michigan Basin. Shale cuttings from two of the other wells in the Basin have been obtained for analysis.

Base maps supplied by the USGS have been reproduced on film and distributed to all mapping subcontractors. A formation audit, a map, and other well search data to be used prior to selecting a working well file for the Michigan Basin stratigraphic study have been compiled and delivered by Petroleum Information of Denver. An initial stratigraphic cross section subdividing the Antrim shale sequence into subunits has been submitted for Dow approval by Mr. Ells, Michigan Department of Natural Resources, and the work has been accepted with minor modifications.

### SUBCONTRACTOR STUDIES

Workers at Michigan Technological University (MTU) have laid the groundwork for the geochemical and geophysical studies to be done there. The methods to be used have been checked out using Antrim shale from the Paxton Quarry at Alpena, Michigan. The first core has been received at MTU and samples taken every 10 feet have been chosen for various analytical procedures.

X-ray mineralogy was determined by Dr. Ruotsala of MTU for 39 specimens of the first core. The essential minerals were quartz, calcite, muscovite, kaolinite, pyrite and minor chlorite. The initial conclusion by Dr. Ruotsala is that most Antrim in these samples is siltstone with quartz as the dominant mineral.

Drs. El Khadem, Leddy and Sandel of MTU have proposed the scheme for the chemical analysis of the shale outlined in Figure 77-Q3-1. Initial results from the first well are shown in Table 77-Q3-1.

Dr. Kim and Mr. Hockings of MTU have put much effort into the preparation of samples for strength tests. The program for the cutting of small samples from the cores has been plagued with breakage due to the low strength of the samples across the bedding planes.

Dr. Leffert of Wayne State University (WSU) has continued to work with the Fischer Retort Assay procedure. Problems with reproducibility, using Antrim shale from the Paxton Quarry, have not yet been resolved.

#### Work Forecast

The 4th quarter of F '77 should see the generation of much data. Work will proceed on all three cores in several locations. The chemical analyses at MTU will have been completed and the examination of the structure of the kerogen should be well begun.

Efforts to obtain reproducible results in the Fischer Retort Assay at WSU will continue. After this problem has been resolved, the task of running large numbers of assays on samples from the three cores will begin.

The problem of small sample preparation for physical measurements will have received much attention at MTU. Data will be obtained on those samples which have been prepared. Porosity and permeability measurements will be made on samples from all cores.

Geological observation of the three cores will be completed and the pertinent information summarized. Mapping activities will continue and information needed for stratigraphic analysis of the region will be compiled. Gas chromatograph analysis of shale cuttings will be done.

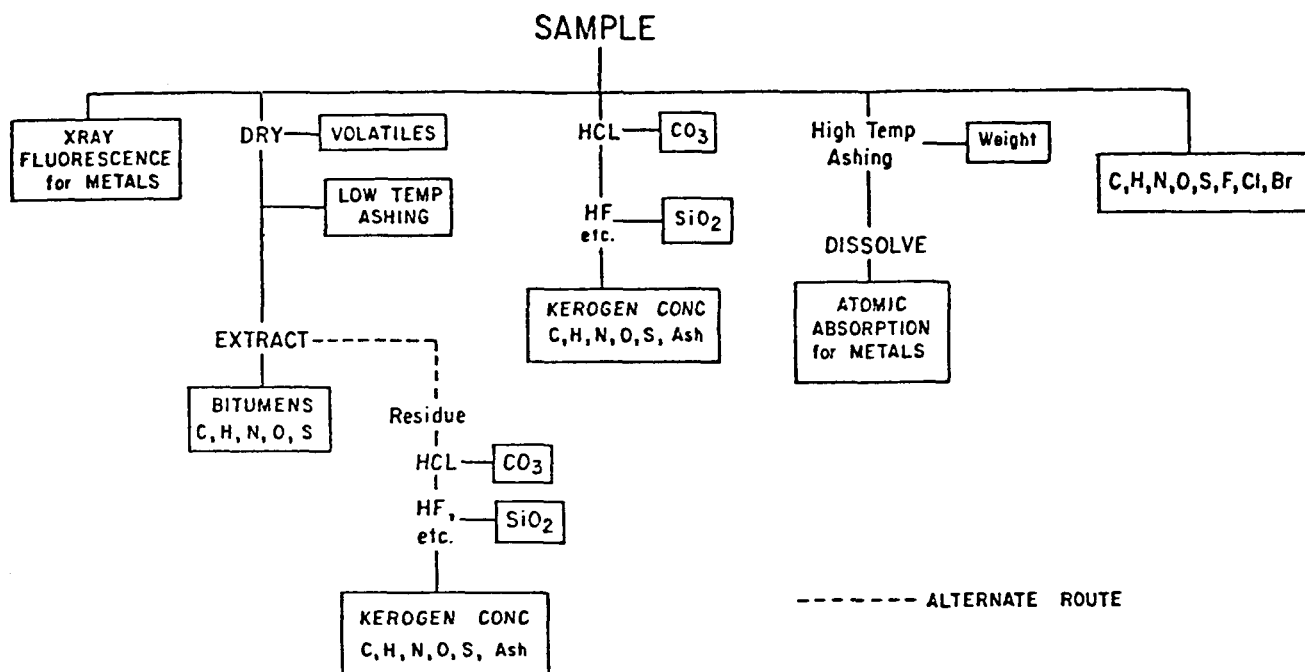


Figure 77-Q3-1 Scheme of the sample handling for the chemical analysis of the Antrim shale



Table 77-Q3-1 Analysis of Samples from Dow/ERDA Well #100

<u>Sample No.</u>	<u>% Volatile<sup>1</sup></u>	<u>% Ash<sup>2</sup></u>	<u>C<sup>2</sup></u>	<u>H<sup>2</sup></u>	<u>N<sup>2</sup></u>
AC8-B1-1160 <sup>3</sup>	8%	83.28%	0.85%	0.52%	0.07%
AC8-B4-1170.2	9%	Ash lost in PE240	0.84%	0.67%	0.13%
AC8-B8-1180.5	8%	89.20%	0.92%	0.66%	0.15%
AC8-B11-1190	9%	84.32%	0.88%	0.66%	0.12%
AC8-B15-1200	10%	91.20%	0.84%	0.70%	<.01%
AC8-B18-1209.8	16%	82.74%	8.15%	1.16%	0.07%
AC9-B1-1220	16%	81.47%	9.77%	1.22%	0.35%
AC9-B5-1230	11%	Ash lost in PE240	7.22%	1.01%	0.16%
AC9-B8-1240	13%	83.13%	9.17%	1.11%	0.27%
AC9-B11-1250.2	11%	86.05%	6.78%	1.06%	0.10%
AC9-B14-1260	8%	88.12%	5.08%	0.84%	0.02%
AC10-B1-1276	9%	89.32%	3.79%	0.74%	<0.01%
AC10-B2-1280	10%	Ash lost in C,H,N	5.45%	0.57%	0.11%
AC10-B5-1290	8%	89.02%	3.85%	0.66%	0.09%
AC10-B9-1300	12.5%	86.55%	5.83%	0.86%	0.19%
AC10-B?-1310.34	10%	86.34%	6.32%	0.92%	0.17%
AC10-B16-1320	11%	85.90%	6.41%	0.94%	0.29%
AC11-B1-1334	11%	81.14%?	6.21%	0.84%	0.14%
AC11-B3-1340	12%	86.85%	6.49%	0.90%	0.09%
AC11-B9-1350.5	6%	66.47%	10.51%	1.56%	0.22%
AC11-B10-1360	11%	85.18%	7.86%	0.84%	0.16%

<sup>1</sup> Loss after 4 hr in muffle furnace @ 550°C

<sup>2</sup> Perkin Elmer 240 Elemental Analyzer

<sup>3</sup> Third number indicates depth, in feet, below ground level

## ENVIRONMENTAL-SOCIO ASSESSMENT

### Environmental Monitoring

The Environmental Research Institute of Michigan (ERIM) has submitted an Environmental Monitoring Plan which details first year monitoring of land, air and water on and around the field site. Plans for monitoring during subsequent years are outlined and are subject to change depending on the results of the first year's program.

The Environmental Monitoring Plan is reproduced in its entirety in the appendix of this report.

Plans were to transfer control of the mobile air monitoring trailer to ERIM during this quarter. This transfer has not been accomplished because of delays in the shipment of a gas chromatograph for carbon monoxide and hydrocarbon monitoring and malfunctions in the SO<sub>2</sub> analyzer. The problems are being worked on and the facility promises to be operational early in the next quarter so that adequate background air quality measurements can be made before the summer combustion trial.

A number of subsidence monuments have been placed near the recently drilled wells. Aerial photography of the Dow property and the neighboring property has been obtained together with surface photography to complete the record of the appearance of the study site before drilling began.

A number of water sampling stations have been established on or near the Dow property and samples have been taken to establish background levels.

Residences within a two kilometer radius of the center of the site were visited. The activities at the site were explained, and the neighbors were encouraged to contact the Environmental Research Institute of Michigan if they have any concerns or questions.

## WELL DEVELOPMENT, FRACTURING & ASSESSMENT

### Well Drilling

Three wells have been drilled during this quarter.

Dow/ERDA #100 was drilled in the center of the south forty acres of the Sanilac County site. This well, which was completed and cored to 2600 feet, will be used as the seismic observation well for all the activities which will be carried out on the south forty. Details of the well are given in Figure 77-Q3-2, which also shows the stratigraphy based in the logging of the well. The well was drilled to 2600 feet so that the Environmental Research Institute of Michigan (ERIM) would be able to move the seismic shear wave generator much closer to the well, thereby significantly reducing the number of shots needed and greatly decreasing the need to intrude upon neighboring property.

Dow/ERDA #101 and #102 were drilled 200 feet apart to a depth of about 1550 feet. Figure 77-Q3-3 gives the details for #101; Well #102 is similar. These wells will be used for the fracturing and explosive underreaming experiment.

The location of these wells, in relation to each other and to the earlier Dow/Rhoburn wells is given in Figure 77-Q3-4.

### Standard Well Logging

A complete series of geophysical borehole logs is being run on Dow/ERDA Wells #100, #101 & #102. Table 77-Q3-2 shows the types of logs and the wells logged.

TABLE 77-Q3-2 LISTING OF DATA LOGS FOR DOW/ERDA WELLS

<u>Data Logging Technique</u>		<u>Well #100</u>	<u>Wells # 101 &amp; 102</u>
GRS	Gamma Ray Survey	Yes	No
DBC	Density, Borehole Compensated	Yes	Yes
CAC	Caliper, 6 Arm	Yes	Yes
SVS	Seisviewer	Yes	Yes
V3D	Velocity, 3 Dimensional	Yes	Yes
GIS	Gyro Inclinator Survey	Yes	No
NBC	Neutron, Borehole Compensated	Yes	Yes
IES	Induction Electric Survey	Yes	Yes

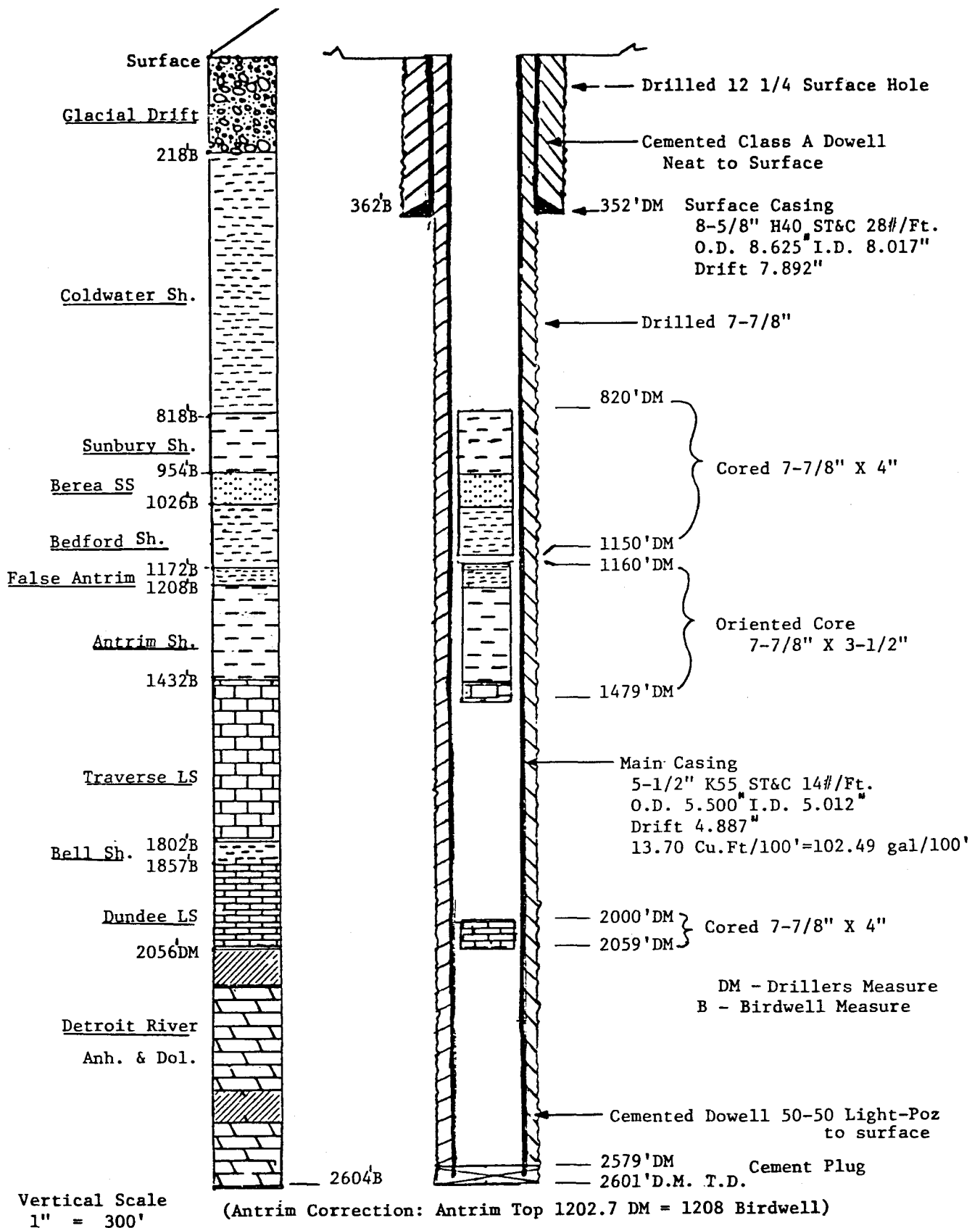


Figure 77-Q3-2 Cross Section of Dow/ERDA Well #100

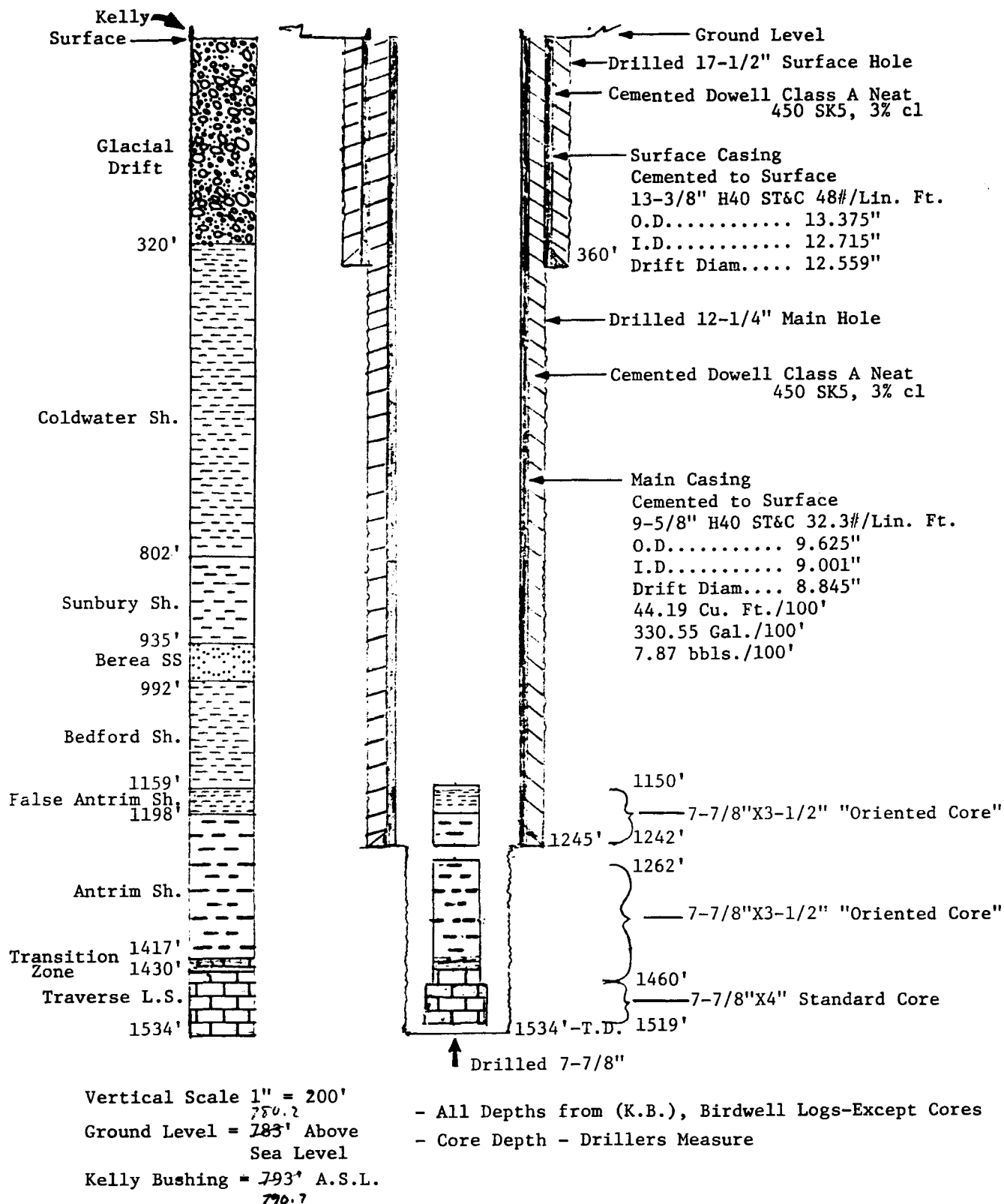


Figure 77-Q3-3

Cross Section of Dow/ERDA Well No. 101

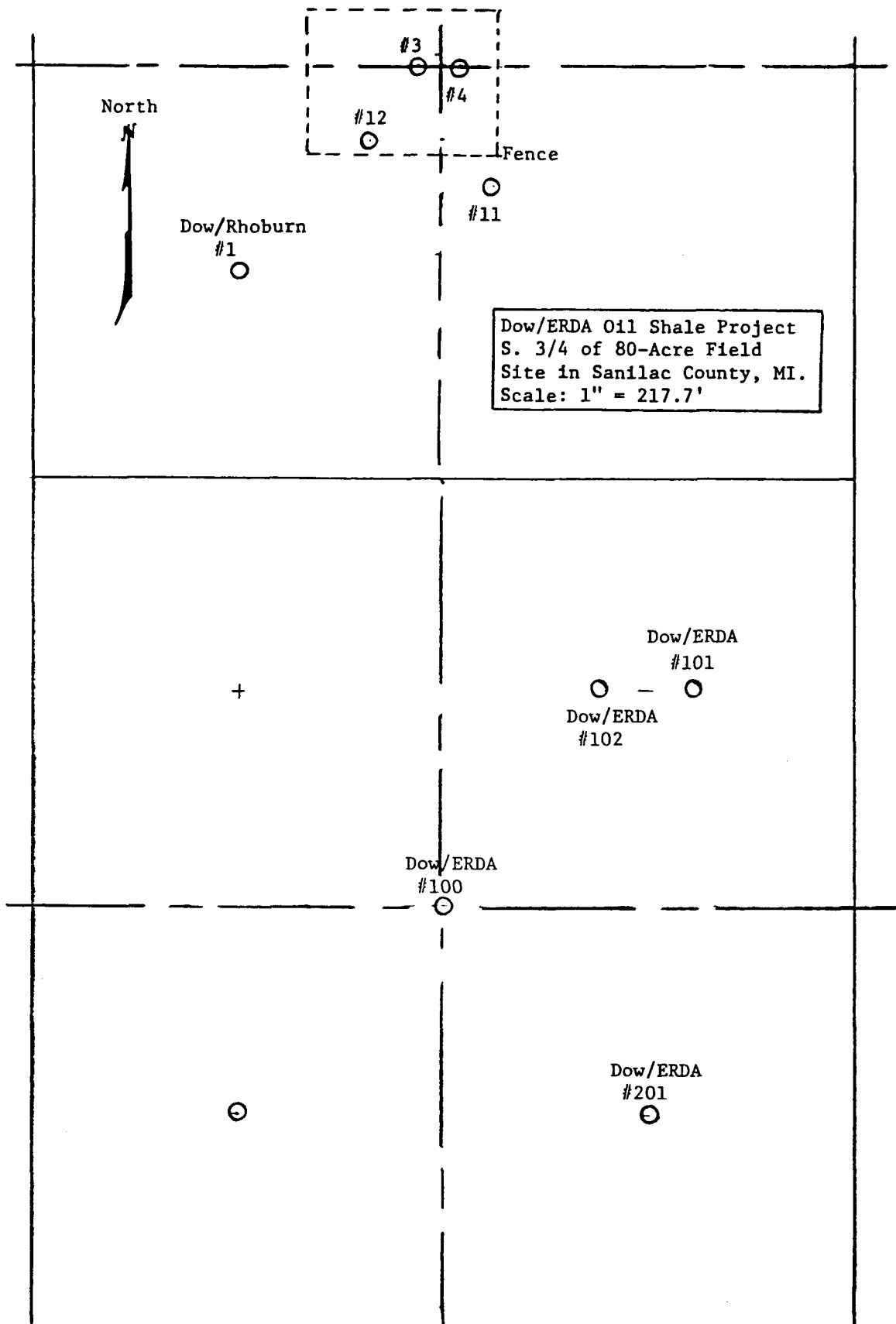


Figure 77-Q3-4 Sketch of south 40 acres showing relative locations of several existing Dow/Rhoburn wells and proposed locations of new Dow/ERDA wells.

Certain of these logs will be computer processed by the Birdwell Division of Seismograph Services to indicate such rock properties as: potential oil shale yields, mineral analyses and elastic properties. These will then be compared with standard analyses of the shale by other subcontractors. The computer processed log data is continuous and extensive and may provide the most economical way of gathering meaningful oil shale data from the wells.

### Fracturing

Details of the fracturing experiment have been discussed a number of times with the Dowell explosives adviser. Equipment and materials are arriving at the site. Evaluation of in situ explosives and suppliers is continuing.

### Fracture Assessment

All fracturing experiments will be monitored by ERIM using a downhole seismometer to receive seismic signals generated by a surface generator. The data generated before and after fracturing activity will be evaluated and used to guide the placement of additional wells located near the original wells.

The additional depth of Well #100 resulted in an unsatisfactorily low signal-to-noise ratio from the seismometer geophones. The noise is predominantly 60 cycle interference. A 20 dB gain amplifier was designed and built by Dow personnel and tested at a depth of 2500 feet in water-filled Well #100 at approximately 1100 psig. This amplifier, which is not available commercially, enhances the signal to noise ratio of the seismometer geophones. The seismometer has now been transferred to ERIM for use and maintenance.

### Work Forecast

Dow/ERDA #201 will be drilled in another quadrant of the north forty acres. This well will be used for the chemical under-reaming experiment planned for later this year. Additional drilling will not occur until after the completion of the hydrofracing experiment in Wells #101 & #102. When this work has been evaluated and sites recommended, up to four more wells will be drilled in an attempt to intercept the fractures.

Fracturing is scheduled for July, and it is expected that a combination of notching, hydrofracing and sand propping on Dow/ERDA Wells #101 & #102 will be employed.

ERIM will complete its background seismic survey before any fracturing of Wells #101 and #102 is done. Then, after the hydrofrac experiment, a new seismic survey will be run and recommendations for the locations of new wells will be prepared based on the survey results.

## PERMEABILITY MEASUREMENT & HYDROCARBON EXTRACTION

### General Site Activity

The initial nitrogen supply, equivalent to 580,000 SCF, has been delivered and is in the storage tank. The installation will be tested as soon as it is complete.

A bank of differential pressure (DP) cells was made up to be used for individual monitoring of gas flows. Connections to any active or producing well will be made with 1000 psig working pressure rubber hose.

### Brine Removal

The 2" and 2-1/2" EUE tubing were lengthened in Wells #3 through #12 so that the bottom ends are about 30 feet above the total well depth. This will allow more brine to be removed from the wells.

The piping network around the liquid gas separators was adapted to throttle the inlet stream so the air and gas that rush into the line after breakthrough can be handled. Provisions were made for increased brine removal and metering capability and for separation of hydrocarbons from aqueous brine discharged to the brine pit.

Table 77-Q3-3 shows the experience to date in brine removal. The new tubing depths are shown. About one quarter of the April brine removal was due to the lowered tubing. About 5000 gallons of brine are reentering the group of wells per month, mostly in Wells #3 - #6. This will be continually removed with two pumps during the combustion experiment.

A local vendor has been licensed to remove and transport industrial wastes, and the brine from the lined pit is now being spread on gravel roads in the area for dust control. This is a lower cost operation than an earlier arrangement.

### Air Injection

Seven air injection experiments were conducted this quarter to complete the identification of interwell communication patterns. The January - March dewatering efforts showed that Dow/Rhoburn Wells #3, #5 & #6 had good intercommunication and that there was fair communication among Wells #4, #7 & #12. This is apparently the result of the February, 1973, explosives detonation. Table 77-Q3-4 summarizes the data from the air inject.



TABLE 77-Q3-3

DATA FOR BRINE REMOVAL USING AIRLIFT AND PUMP  
December 17, 1976 through June 30, 1977

Well No. (P=Pumped)	Total depth, feet	April tubing depth, feet	----- Volume of Brine Removed, gal. -----				
			Total, 12/76-3/77	April, 1977	May, 1977	June, 1977	Total, 12/76-6/77
3	1202	1176	3,101	39	153	3876	7,169
4 (P) *	1325	1304	14,498	993	626	0	16,117
5	1346	1300	13,670	1,883	2898	595	19,046
6	1346	1318	3,450	294	343	0	4,087
7	1388	1366	2,098	50	40	48	2,236
8	1396	1375	2,558	145	80	337	3,120
9	1428	1409	2,995	373	216	244	3,828
10	1408	1383	2,187	200	283	230	2,900
11	1402	1378	2,282	250	38	362	2,932
12	1396	1358	1,520	75	0	25	1,620
Total			48,359	4,302	4,677	5,717	63,055

\*The Reda submergible pump was moved from Well #4 to Well #3 in late June, and a second Reda pump was installed in Well #5.

TABLE 77-Q3-4 AIR INJECTION EXPERIMENTS, APRIL - JUNE, 1977

<u>Air injection well no.</u>	<u>End date, 1977</u>	<u>Injection time, hrs</u>	<u>Total Observation &amp; injection time, hours</u>	<u>Average injection rate, SCFH</u>	<u>Volume injected, M SCF</u>	<u>Proportion of injected air recovered, per cent</u>	<u>Main response wells in order of air prodn.</u>
10	4/4	23	104	4490	103.3	37	5, 3
7	4/11	66	140	3320	219.5	58	4, 3, 12
9	4/26	86	162	3080	264.4	28	3, 8
8	5/8	112	151	2145	240.0	43	3, 9, 4
11	5/14	77	98	2400	184.0	33	3, 4
12	5/23	86	158	1530	132.0	58	4, 7, 3
4	6/13	94	167	3350	315.0	50	3, 7, 8

experiments. The data from which the last column was developed were used to prepare the schematic representation of the apparent communication patterns shown in Figure 77-Q3-5. In addition, the material balance data indicate there are other void patterns that may be extensive; no attempt is made to show these here.

Well #4 was selected as the hole in which to conduct the summer ignition test. It is one of the largest holes (borehole--12", casing--9-5/8") and it has a mechanically underreamed 4 foot diameter by 25 foot deep cavity. The burner will fit easily into this hole. Flexibility in the rate of injection may be necessary to sustain combustion, and, whereas in many of the wells the air rate had to be decreased to as low as 30 scfm by the end of experiment, Well #4 accepted air at almost twice the rate of individual Wells #7 - #12. Well #4 is in communication with all other Dow/Rhoburn wells to some extent, and it was the second most responsive well. The most responsive well, #3, was not considered as a potential burn hole because it is bridged off at 1200 feet, it has a high brine reentry rate, and it appears to have extensive fractures in the false Antrim layer.

#### Other Pre-Combustion Logging

A number of techniques are being used to further define which horizons in Well #4 would be the best for the combustion trial.

Well bore sibilation and spinner tests were conducted late in June and are being evaluated. The results should indicate through which rock zones the air flows when it moves from Well #4 to Wells #6 & #7.

Data from nuclear and seisviewer logs are also being studied to determine levels of favorable organic content and zones of visible fracturing. A subcontract has been let to Systems, Science and Software, Inc., to perform sophisticated oil shale porosity and permeability studies.

#### Downhole Ignition System

Construction of a propane/air burner and an electrical heater system for the Dow/Rhoburn wells is progressing. The 5-1/2" well head, the two electric heaters and the controls have arrived. The liquid propane pumping installation was completed.

A dry gas meter was tested for use in calibrating the air flow. Agreement between test data and calculations was within 2% at 30-125 CFM and 400-800 psig.

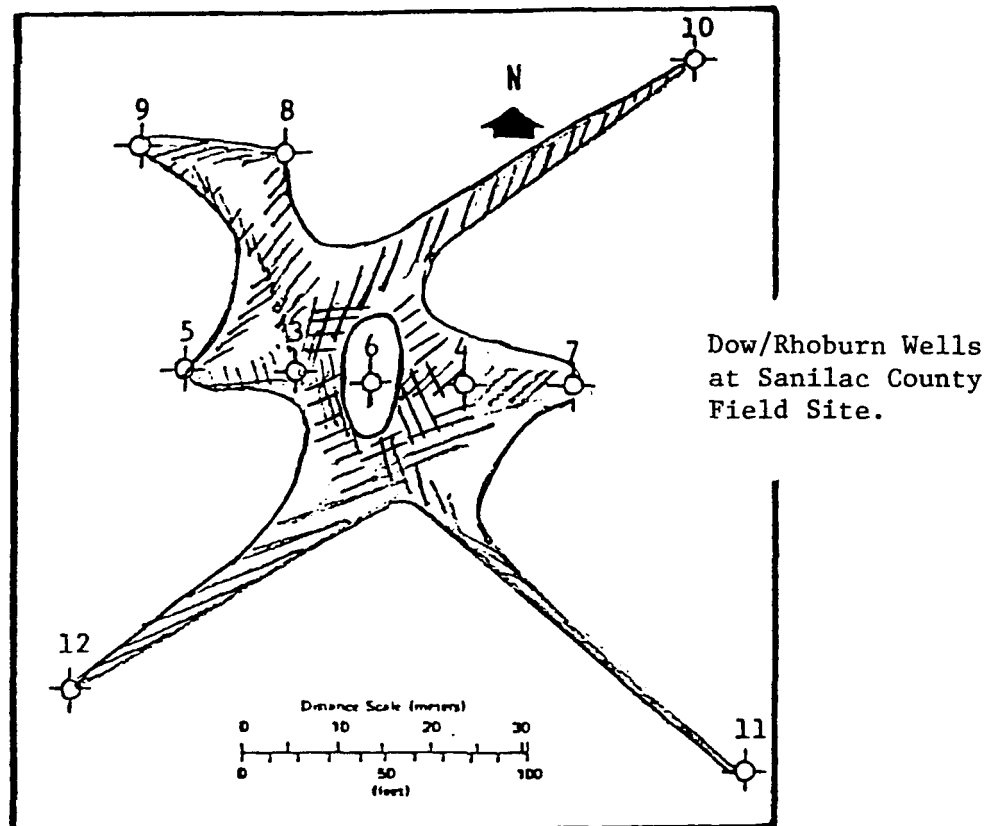


Figure 77-Q3-5 Apparent communication pattern among wells -- based on data from air injection.

The propane burner was operated on the aboveground test stand, and it was decided to replace the burner safeguard with a Pt/Pt-Rhodium thermocouple as a flame indicator. An automatic shutdown system is being incorporated into the propane burner system so that if the temperature falls below a set point for more than four minutes, the system will be shut down and the injection well purged with nitrogen.

### Extraction Monitoring & Analytical

#### Dow/Rhoburn Wells

An extensive program of measurement and testing is being developed to monitor the combustion experiment. Instrumentation will be placed in a borehole adjacent to the ignition well by the Sandia Laboratories to measure temperature, pressure and borehole resistivity changes.

Lines have been installed to Wells #3 through #12. Gases from any of these wells can be sampled and analyzed by the new Hewlett-Packard Model 5840 gas chromatograph with a refinery gas option.

A data collection plan for the Dow/ERDA well system is being developed around the PDP-8E Computer as a collection and storage device.

Continuing improvements are being made on the two GC's at the site, on the GC/teleprinter interface and on the transmission system between field site and Midland teleprinters and computers.

### Work Forecast

Downhole ignition testing in Dow/Rhoburn Well #4 is scheduled for August. Safety inspections and aboveground testing of the heater systems will take place in July.

Data from recently completed well logging surveys and from scheduled July studies will be used to further define burner placement and other conditions for the in situ combustion experiment.

Brine removal will continue on a maintenance basis using the pumps reinstalled in Wells #3 and #5 in late June.

## SUMMARY AND CONCLUSIONS

### GENERAL

Base maps supplied by the USGS have been reproduced and distributed to mapping subcontractors.

The Environmental Monitoring Plan has been prepared by ERIM and is being implemented.

### DOW/RHOBURN WELLS

Permeability studies via air injection were completed on Wells #7 - #12 and on Well #4. Well #4 was selected to be used for ignition tests scheduled for August.

Final preparations were being made at the end of June and extending into July for the ignition test. These include: a) taking further well surveys, evaluating the data from these and existing logs, and defining to best location for the ignition test within Well #4; b) final safety, mechanical and aboveground operational checks on the heater systems, and; c) developing and checking the data collection plan for monitoring the combustion.

### DOW/ERDA WELLS

Three wells were drilled in the south forty acres of the field site. A total length of 1403 feet of core was obtained along with a complete series of geophysical borehole logs. Subcontractor studies on the cores have begun.

Plans are nearing completion for a fracturing experiment on Wells #101 & #102. The apparatus for obtaining seismic measurements to evaluate the fracturing has been tested successfully on Observation Well #100.