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ENERGY FROM IN SITU PROCESSING OF ANTRIM OIL SHALE

Quarterly Technical Progress Report
for July - September, 1978

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DEPARTMENT OF ENERGY
Washington, D.C. 20545

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

OBJECTIVE AND 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

Explosive fracturing experiments were conducted in the wells involved in the explosive underream series and in the hydrofracture series. In total, four successful shots were made. In Well #301, a 20 foot section of well bore was enlarged to almost three times its original volume by the detonation of 1500 pounds of explosive. Wells #101, 104, and 106 were also explosively fractured. Preparations for fracturing Wells #103 and 105 are complete. In lab tests for the chemical underream experiment, a 4 foot zone was found to have a uniform and high hydrochloric acid solubility. Notching, fracturing, and acid underreaming experiments will be concentrated in this area.

Data from the extraction trials on the front site have been compiled and plotted. A topical report on this experiment is being prepared. Two new methods of igniting shale were tested. Shale characterization and resource inventory activities continued at four major Michigan universities.

SUMMARY OF PROGRESS TO DATE

The effective date of this contract is September 30, 1976. During the first quarter, an environmental research plan was prepared, and the Sanilac County field site was reactivated after having been shut down for 18 months.

The principal initial field effort was to prepare the surface piping network and equipment, and to start brine removal operations on the existing wells. Air injection tests were used to measure the permeability in the existing well system. Well #4 was selected in June, 1977, for the first in situ combustion test.

A mobile air monitoring laboratory was outfitted and tested by Dow personnel and transferred to the control of the Environmental Research Institute of Michigan (ERIM). Background measurements of air quality began in July; a program of continuous air quality monitoring started. Monitoring of baseline land and water environmental characteristics also began in July. These activities are continuing.

Two ignition systems for in situ combustion were constructed: a propane/air burner system and an electric heater system. Two electric heater trials in August were aborted after electrical short circuits occurred. The propane/air burner and its several subsystems were successfully tested above ground and later used below ground.

Four wells in a new series in the south 40 acres of the field site were drilled, cored, and logged during mid-1977. Well #100 was completed to 2600 feet for seismic observations. The seismometer used for fracture assessment was successfully tested by ERIM and baseline data were obtained.

Wells #101 and 102 were drilled 200 feet apart to a depth of about 1500 feet for use in hydraulic fracturing and explosive experiments. In a preliminary notching operation in these wells in August, vertical fractures were induced by the pressure applied to seal off treatment zones with production packers. Hydraulic fracturing and sand propping further enlarged these and created new vertical fractures. Wells #103-106 were drilled near Well #101 at the end of the year. A comprehensive data logging program including a gyro inclinometer study and an acoustic survey was completed on these wells.

In the second quarter of 1978, Wells #103-106 were subjected to hydrofracturing and sand propping procedures. Extensive logging and permeability testing confirmed that communication had been achieved between the various wells. Slurry explosive was successfully detonated in three of the 100 series wells in September 1978.

In 1977, four universities in Michigan assigned personnel to study the cores from the new wells and cuttings from other wells in the Michigan Basin. The University of Michigan team is describing the stratigraphy. Several departments at the Michigan Technological University are performing geochemical and geophysical analyses. Groups from Wayne State University are conducting modified Fischer Assays and developing a mathematical model of an in situ combustion process based on kinetic and thermodynamic parameters. Michigan State University is studying Antrim shale outcrops and exposures.

In situ combustion experimentation continued in Well #4 in November and December with a modified electric heater. Analysis of the effluent gases indicated that combustion had been achieved and sustained for several days. A coal and charcoal augmented combustion trial was conducted in Well #4 in February, 1978; a propane burner was used for ignition. Combustion was maintained at a high level for about one week, and there was evidence of continued CO_2 and CO generation for another ten days. Greater quantities of hydrocarbons, carbon monoxide and hydrogen were produced than during previous trials. During flowback of gases from the formation after venting the injection well, the analysis showed increased amounts of hydrocarbons, possibly reflecting retorting of the oil shale. A charcoal augmented combustion trial in Well #7, begun at the end of March, continued for 61 days, while several continuous and cyclic production procedures were tested. Gas having energy values in the target range (150 BTU/SCF) was produced at various times, although the flow rate at these times was minimal and hence the energy production was low. There was no significant heat damage to the propane burner, whereas in the previous run the burner was severely damaged.

An acid underreaming experiment was conducted in Well #201 in January, 1978, using 28% hydrochloric acid. Wells #202-205 were drilled around Well #201 in preparation for acid fracturing and explosive rubblizing. A number of laboratory experiments with core samples were completed and a narrow region of interest for new field experiments was identified.

Wells #301-305 were drilled early in 1978. The first of a series of bore hole explosive tests in Well #301 was successfully detonated in July. This shot resulted in a substantial volume increase in the well. The second shot is now scheduled for November.

Legend

Planned:
Actual:
Intermediate Event: ▽
Completed: ▼
Milestone: △

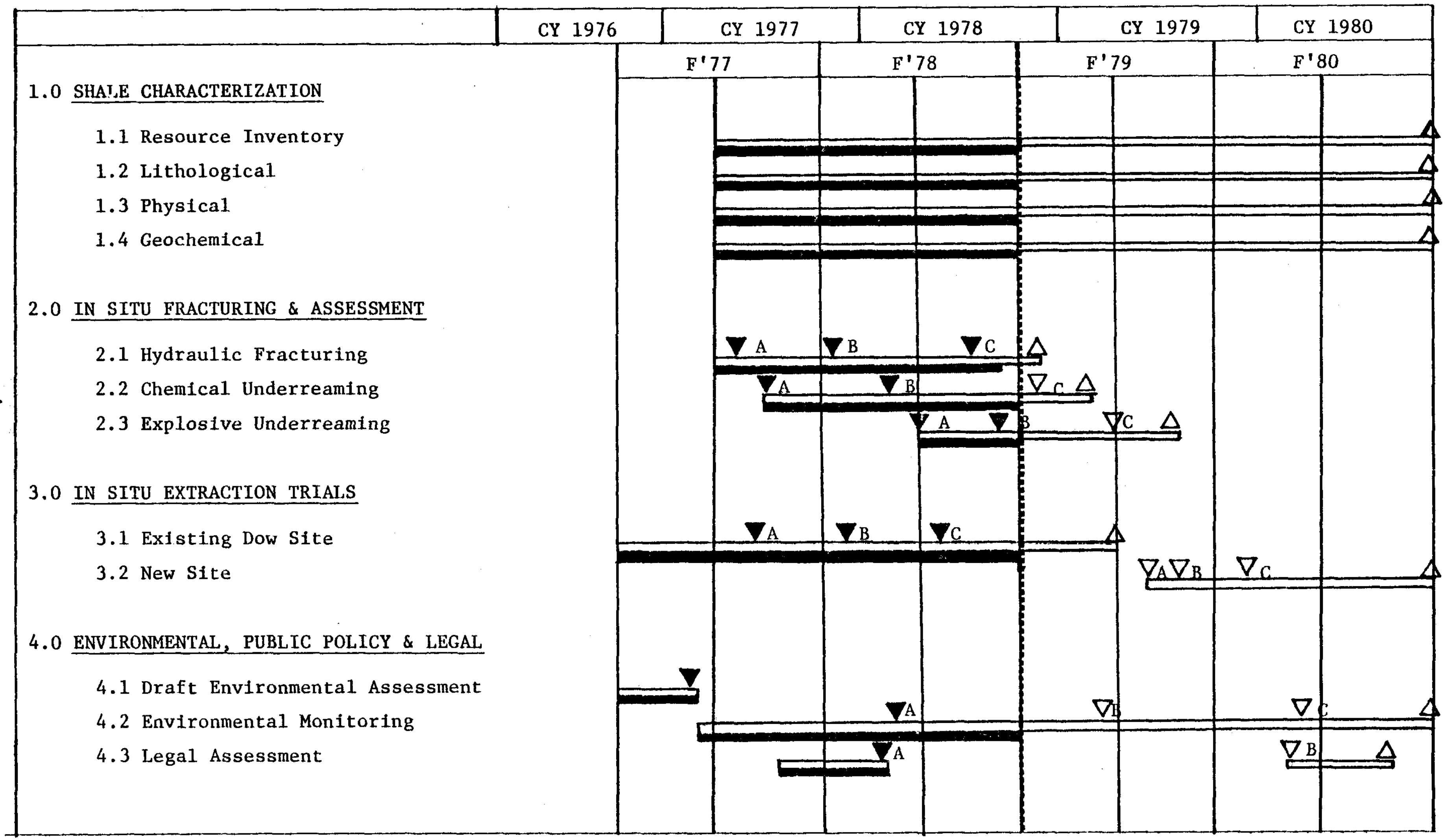
MILESTONE CHART

CONTRACT NUMBER: EX-76-C-01-2346

ENERGY FROM IN SITU PROCESSING OF ANTRIM SHALE

Project Manager: L. J. Washington, Jr.

Date: October 20, 1978
Report No: FE-2346-34



CONTRACT EX-76-C-01-2346

MILESTONE CHART

(Showing Intermediate Events and Milestone Points)

Task		Event or Milestone Point	Date
1.0	Δ	Summary report - all subtasks	9/80
2.1A	∇	Drill and core wells #101 and #102	6/77
B	∇	Initial hydraulic fracturing	10/77
C	∇	Explosive fracturing	7/78
	Δ	Assess results	10/78
2.2A	∇	Drill and core well #201	7/77
B	∇	Initial acid underreaming	2/78
C	∇	Explosive fracturing	11/78
	Δ	Assess results	2/79
2.3A	∇	Drill and core well #301	3/78
B	∇	Initial explosive underream	8/78
C	∇	Explosive fracturing	3/79
	Δ	Assess results	6/79
3.1A	∇	Initial permeability study	7/77
B	∇	Three-week combustion trial	12/77
C	∇	Complete combustion tests	6/78
	Δ	Complete site observation and abandon location	3/79
3.2A	∇	Identify second site for combustion tests	7/79
B	∇	Begin combustion tests	8/79
C	∇	Complete combustion tests	2/80
	Δ	Complete site observation	9/80
4.1	Δ	Draft environmental assessment accepted	1/77
4.2A	∇	Update environmental assessment	3/78
B	∇	Update environmental assessment	3/79
C	∇	Update environmental assessment	3/80
	Δ	Summary assessment	9/80
4.3A	∇	Complete initial legal assessment	2/78
B	∇	Review and update legal assessment	1/80
	Δ	Summary legal assessment	8/80

SHALE CHARACTERIZATION

RESOURCE INVENTORY

With the acquisition of appropriate gamma ray logs from northern Ohio and Indiana, the gamma ray log file for the Michigan Basin is complete. Over 2000 logs are included. A composite base map, one inch equals 8 miles, has been drafted.

Petroleum Information, Inc., has solved the problem of fitting the computer drafted map overlay for use with USGS base maps. Work on the production map is underway.

Samples of drill cuttings have been collected from four more wells in the Michigan Basin. These samples, which will be used for stratigraphic and characterization studies in lieu of well cores, are being collected in an on-going program with the cooperation of the oil and gas industry. The current status of this sampling program is illustrated in Figure 78-Q4-1.

LITHOLOGICAL CHARACTERIZATION

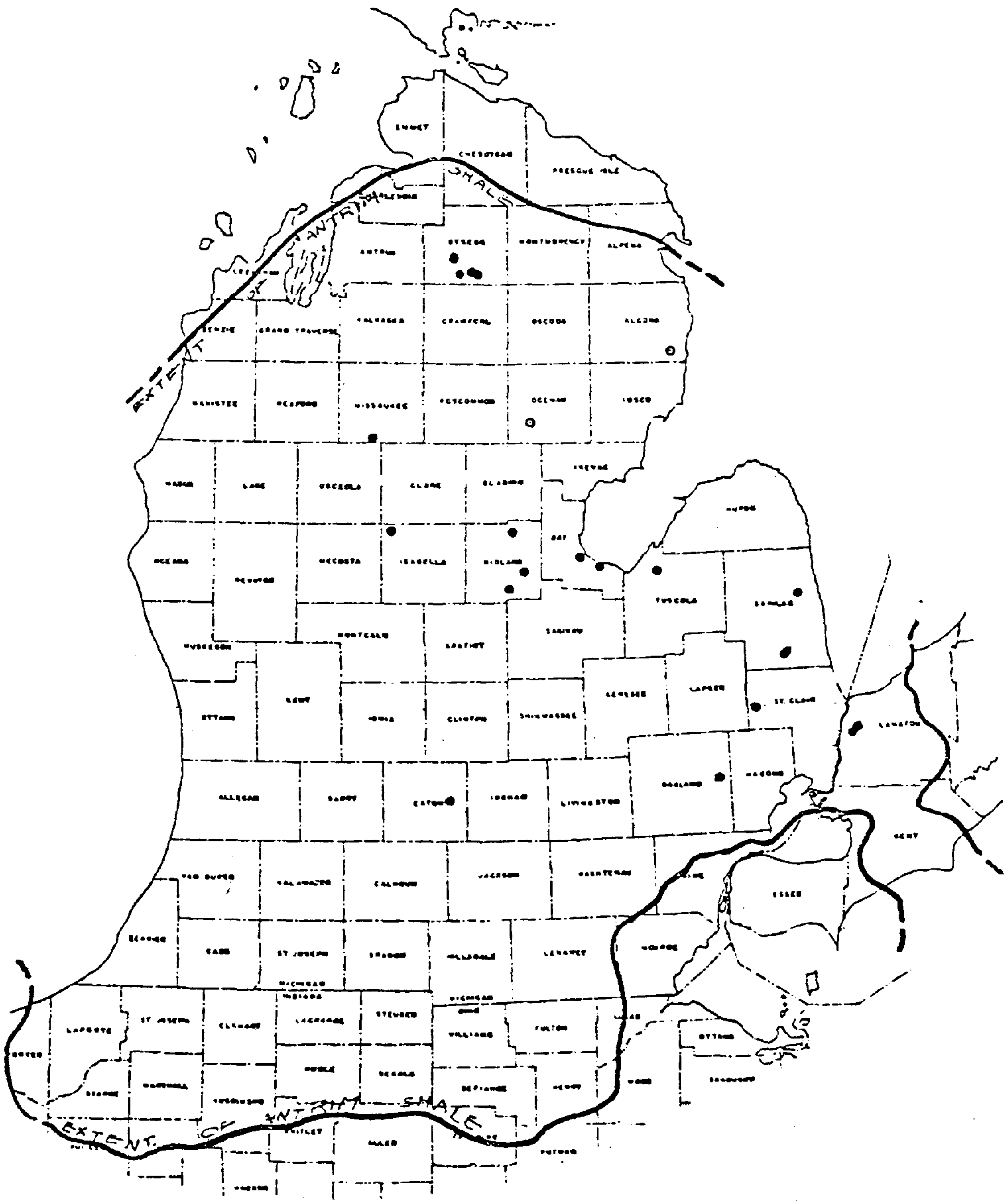
Dr. Cambray and coworkers at Michigan State University have visited and described three widely separated outcrops of Antrim shale. The most extensive outcrop occurs in the Paxton Quarry at Alpena, Michigan. A very detailed description of the 32 feet (9.8 meters) of Antrim shale which is exposed there has been completed. The general definition of the two main terms used in the description are as follows:

Black Shale, Characteristic Type. This refers to the type of black shale most commonly encountered. The Munsel Colors 10YR2/2 and 5YR2/1 are closest to the black shale's color depending on light conditions. There are two types of laminations present. The continuous laminations are very thin, trace - .0002 m, with prominent vertical spacing of approximately .01 m. The other types are thicker and discontinuous. The larger laminations are .0002-.0008 m thick, .05-.5 m long, and a vertical spacing of .030-.050 m.

Green Shale. This refers to layers within the black shale which appear to have undergone bioturbation. The lack of organic material due to the feeding of organisms is the reason for its green color. The Munsel Color 5G6/1 is the closest approximation. This shale does contain darker, 5G4/1 inclusions and streaks. The green shale generally can be divided into two units, burrows and what we refer to as the solid green layer. The burrows are always the lower section with a very irregular contact with the underlying black shale. The contact between the burrows and solid green layer is

Figure 78-Q4-1

WELLS SAMPLED IN THE MICHIGAN BASIN FOR
RESOURCE INVENTORY OF SHALE CHARACTERIZATION



● Older Work

o Wells Sampled This Quarter

generally taken as the loss of distinguishable, individual burrow structures and black shale between the burrows. The solid green layer is then a unit of total bioturbation in which all structure and organic material have been totally disrupted. The contact with the overlying black shale is always very sharp with no irregularities.

Burrows generally fit into two general sizes, large and small. Large burrows are usually .002-.008 m thick and .001-.1 m long, while small burrows are generally <0.002 m in diameter. There is no apparent relationship between size of burrows and degree of bioturbation; they can be found together or with thin and thick solid green layers.

At Michigan Technological University (MTU), semi-quantitative mineralogical analyses were obtained for well cuttings samples from three more wells in the Michigan Basin.

A computer program was developed to connect chemical analyses to normative mineralogical analyses. Normative mineralogical analyses are not accurate because mineral compositions are assumed; however, a comparison of normative and x-ray mineralogical data will be valuable.

A distributional pattern of clay minerals is beginning to emerge. In all Antrim shale samples, illite is the dominant clay mineral. About 30-40% of the Antrim is illite. There is a small increase in illite content from the edge of the Michigan Basin at the drill site toward the center of the basin in Midland County. Kaolinite shows a corresponding decrease. Chlorite, essentially absent in samples at the edge of the basin, is present in virtually all samples from the basin center.

At the University of Michigan (UM), Dr. L. I. Briggs has had to resign from the project due to failing health. Discussions have been held with representatives of several universities; arrangements to complete the remaining work in the stratigraphic analysis of the Antrim shale formation will be completed as soon as possible.

Some of the work which has been completed in the Subsurface Laboratory at UM is summarized in Table 78-Q4-1.

GEOPHYSICAL CHARACTERIZATION

At the Institute of Mineral Research at MTU, Mr. Hockings has completed density and porosity measurements on samples from four wells in the Michigan Basin. The data from one of these wells are displayed in Table 78-Q4-2; the data from the other three wells are quite similar.

TABLE 78-Q4-1

Generalized Antrim core description.
Based on Dow Wells #100, 101, 102,
and Dow Rhoburn #8.
(Core begins in the Bedford Shale)

1150.0-1165.0	Thinly interbedded dark gray shale, gray shale, and rippled, light gray silt; abundant fossil debris.
1165.0-1191.0	Shale - black to dark gray; thinly laminated; very little fossil debris.
1191.0-1196.0	Shale - dark gray to black with a few light gray shale layers; fossil debris abundant.
1196.0-1197.0	Shale - light gray to green; Antrim-Bedford contact.

Antrim Formation

1197.0-1265.0	Shale - black; thinly laminated; abundant fish, conodonts, and spores; pyrite abundant.
1265.0-1265.5	Shale - green-gray; burrowed.
1265.5-1375.0	Shale - interbedded black and burrowed gray green; gray-green shale increases with depth; fossils abundant; carbonate concretions and inclined bedding in places.
1375.0-1420.0	Shale - interbedded calcareous gray green and black with some light gray carbonate units; fossiliferous; concretions; bottom of Antrim.

Traverse Formation

1420.0-1432.0	Shale - calcareous and gray with some limestone beds and dolomite; fossiliferous.
1432.0-1437.0	White chert and limestone.
1437.0-1520.0	Limestone - gray interbedded with gray calcareous shale; fossiliferous.

TABLE 78-Q4-2

DENSITY AND POROSITY OF WELL CUTTINGS

Reef Petroleum Corp., Smitha 4-1

Footage		Density g/cc Geometric	Density g/cc True	True Porosity %
820- 830		2.516	2.679	6.1
830- 840			2.491	
840- 850		2.234	2.400	6.9
850- 860			2.434	
860- 870		2.316	2.509	7.7
870- 880			2.517	
880- 890		2.328	2.531	8.0
890- 900			2.510	
900- 910		2.350	2.496	5.8
910- 920			2.479	
920- 930		2.304	2.483	7.2
930- 940			2.499	
940- 950		2.309	2.484	7.0
950- 960			2.526	
960- 970		2.297	2.505	8.3
970- 980			2.522	
980- 990		2.393	2.551	6.2
990-1000			2.684	
	Avg.	2.339	2.517	7.0
	S.D.	0.079	0.069	0.9

Property measurements on samples which have been roasted in air and in nitrogen are underway.

In an attempt to study the relationships between permeability/porosity changes due to retorting and the corresponding changes in other mechanical properties, Dr. Kim at MTU in collabration with Mr. Hockings has compared these properties before and after retorting. Tests were run on paired samples (one specimen parallel to the bedding plane and one perpendicular to it). The data obtained from this series of tests are presented in Tables 78-Q4-3 and 4. All these measured properties have been plotted one against the other using a computer to examine the general relationships which may exist among these parameters. The deformation and strength properties previously determined were also used in the analysis. The results will be discussed fully in future topical reports.

Dr. Kim at MTU has continued hydraulic fracturing tests on thick wall cylinders (5.4 cm O.D., 0.64 cm I.D., and 10 cm long) using hydraulic oil as the fracturing fluid. Vertical stress was kept at ∿ 10.5 MPa (1520 psi) simulating the vertical <u>in situ</u> stress at about 1350 ft (412 meters) below surface and horizontal stresses were varied from zero to 4.8 MPa (700 psi) from test to test. All the specimens used in this series of tests were cored from two large blocks collected from the Paxton Quarry in Alpena, Michigan, and, therefore, fairly uniform in property. The test results are presented in Table 78-Q4-5.

Fracture orientation was always vertical for horizontal stresses below 0.3 MPa (50 psi) and horizontal for horizontal stresses higher than 0.7 MPa (105 psi) implying that the transition zone from vertical to horizontal fracture lies in between 0.3 and 0.7 MPa (50 and 105 psi). Horizontal fracture is, therefore, likely to occur at a depth of about 1350 ft in hydraulic fracturing loading if <u>in situ</u> stresses in the horizontal plane are isostatic. In case the horizontal stresses are not isostatic, i.e., $\sigma_2 \neq \sigma_3$, vertical fracture can be induced depending upon the magnitude of the least horizontal principal stress. More tests will be required to substantiate the foregoing argument.

At Oakland University, ultrasonic velocity measurements for radial directions have been completed in 30 samples from cores from Well #201. Samples tested came from depths between 1167 and 1485 feet (355.7 and 452.6 meters). The average values for all the velocity measurements made to date are shown in Figure 78-Q4-2.

Table 78-Q4-3

Permeability, Porosity and Density of Unretorted Specimens in Parallel and Perpendicular Directions to Bedding.

Sample No.	Parallel to the Bed					Perpendicular to the Bed				
	% Wt. Loss	D_G* (Mg/m^3)	D_A+ (Mg/m^3)	Porosity (%)	Permeability (Millidarcies)	% Wt. Loss	D_G* (Mg/m^3)	D_A+ (Mg/m^3)	Porosity (%)	Permeability (Millidarcies)
101/1330.9	5.7	2.433	2.468	1.42	.05921	6.6	2.406	2.442	1.47	.00921
101/1332.3	9.0	2.358	2.404	1.9	.03448	9.4	2.371	2.389	0.8	.02039
101/1333.3	6.9	2.436	2.492	2.2	.03156	7.3	2.439	2.461	0.9	.01705
101/1334.9	9.4	2.377	2.433	2.3	.03040	10.2	2.364	2.412	2.0	.01542
101/1336.1		2.317	2.428	4.6	.03661	4.5	2.337	2.419	3.4	.01174
101/1340.9		2.336	2.378	1.8	.01909	9.6	2.333	2.384	2.1	.01323
101/1347.9	8.0	2.409	2.403	0.0	.02887	7.4	2.415	2.420	0.2	.02074
101/1350.1	3.7	2.414	2.429	0.6	.02542	6.2	2.398	2.482	3.4	.00404
101/1359.9	11.8	2.321	2.366	1.9	.02657	8.2	2.387	2.433	1.9	.02069
101/1368.6	5.2	2.408	2.519	4.4	.03224	5.2	2.401	2.508	4.3	.02874
101/1412.3	4.5	2.476	2.504	1.1	.12286	2.7	2.444	2.578	5.2	.02416
101/1426.8	6.3	2.610	2.746	5.0	.05118					
101/1441.7	0.6	2.619	2.709	3.3	.05369	0.5	2.623	2.720	3.6	.04703
102/1215.7	9.0	2.257	2.313	2.4	.15327	9.1	2.229	2.325	4.1	.02312
102/1257.3	10.6	2.251	2.294	1.9	.04579	11.4	2.250	2.289	1.7	.04383
102/1328.3	7.0	2.375	2.433	2.4	.03855		2.381	2.415	1.4	.01317
102/1375.6						3.7	2.547	2.605	2.2	.02813
102/1415.9	4.4	2.425	2.472	1.9	.03243	3.0	2.453	2.588	5.2	.02703
101/1511.4	1.0	2.542	2.740	7.2	.08547	1.0	2.540	2.740	7.2	.04087
102/1397.0							2.431	2.516	3.4	.03758

*D_G = geometric density

+D_A = apparent density

Table 78-Q4-4

Permeability, Porosity and Density of Retorted Specimens in Parallel and Perpendicular Directions to Bedding.

Sample No.	Parallel to the Bed: % Wt. Loss	Parallel: D_G* (Mg/m^3)	Parallel: D_A+ (Mg/m^3)	Parallel: Porosity (%)	Parallel: Permeability (Millidarcies)	Perpendicular to the Bed: % Wt. Loss	Perpendicular: D_G* (Mg/m^3)	Perpendicular: D_A+ (Mg/m^3)	Perpendicular: Porosity (%)	Perpendicular: Permeability (Millidarcies)
101/1332.3	9.0	2.058	2.762	25.49	15.430	9.4	2.102	2.758	23.79	.11568
101/1333.3	6.9	2.255	2.654	15.03	5.051	7.3	2.228	2.678	16.80	.09766
101/1347.9	8.0	2.195	2.731	19.63	10.306	7.4	2.188	2.729	19.82	.14097
101/1350.1	3.7	2.307	2.728	15.43	2.554	6.2	2.216	2.717	18.44	.07654
101/1359.9	11.8	2.020	2.778	27.28	15.244	8.2	2.164	2.760	21.59	.10144
101/1368.6	5.2	2.265	2.719	16.70	1.6078	5.2	2.254	2.708	16.70	.08206
101/1381.5	1.5	2.488	2.707	8.09	1.315					
101/1412.3	4.5	2.335	2.697	13.42	2.823	2.7	2.371	2.707	12.41	.07230
102/1257.3	10.6	1.967	2.692	26.93	7.660	11.4	1.999	2.740	27.04	.12230
102/1415.9	4.4	2.308	2.762	16.44	1.718	3.0	2.369	2.702	12.32	.06404
101/1511.4	1.0	2.494	2.746	9.18	1.358					
101/1330.9						6.6	2.217	2.711	18.22	.05892
101/1336.1						4.5	2.286	2.647	13.64	.03981
101/1334.9						10.2	2.079	2.713	23.37	.08877
101/1340.9						9.6	2.079	2.738	24.07	.10912
102/1215.7	9.0	2.036	2.608	21.9	3.784	9.1	2.014	2.707	25.60	.10369
101/1441.7	0.6	2.579	2.735	5.7	.455					

*D_G = geometric density

+D_A = apparent density

TABLE 78-Q4-5

Laboratory Hydraulic Fracturing Test Results (Antrim Oil Shale)

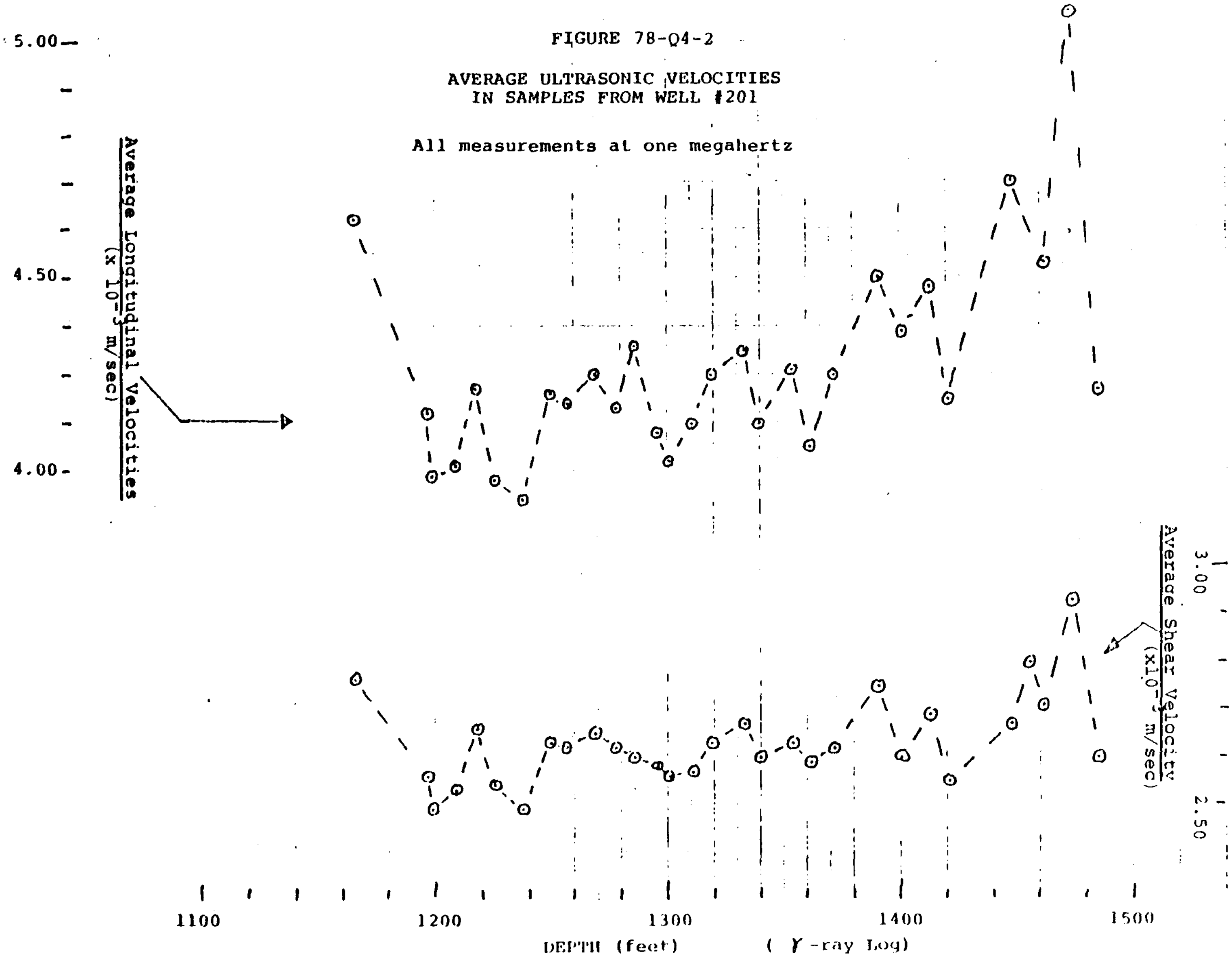
Sample*	Vertical Stress		Horizontal Stress		Breakdown Pressure		Fracture Orientation
	MPa	Psi	MPa	Psi	MPa	Psi	
56	10.5	1520	0	0	12.1	1760	Vertical
58	10.4	1514	0	0	7.1	1025	Vertical
53	10.5	1518	0.3	50	8.5	1225	Vertical
52	10.4	1514	0.7	105	9.3	1350	Horizontal
51	10.5	1520	2.2	325	11.0	1600	Horizontal
54	10.5	1520	3.0	440	15.3	2225	Horizontal
55	10.5	1520	3.3	480	13.5	1950	Horizontal
57	10.5	1520	4.8	700	13.8	2000	Horizontal

*All samples from Paxton Clay Quarry (Alpena, Michigan) 52 and 53 from Block 1, all others from Block 2.

FIGURE 78-Q4-2

AVERAGE ULTRASONIC VELOCITIES
IN SAMPLES FROM WELL #201

All measurements at one megahertz



GEOCHEMICAL CHARACTERIZATION

Dr. Leffert's group at Wayne State University (WSU) has completed Fischer Retort Assays on 150 samples from five different wells in the Michigan Basin during this quarter. The data from one of these wells, which are shown in Figure 78-Q4-3, are exemplary.

At Michigan Technological University (MTU), Dr. Leddy's group has completed the analyses for the majority of elements in cores from Wells #100, 101, and 102. The necessary computer programs have been completed and the data are now stored in computer files in a form such that they can be called out and examined for correlations. A computer printout of these data occupies 49 pages. The results for aluminum, tin, vanadium, bromine, and calcium from Well #100 are shown in Table 78-Q4-6.

Analyses of well cuttings from seven wells in the Michigan Basin are well underway. These data will be reported later. X-ray fluorescence analyses have been completed on 147 samples from five wells. This procedure can detect the presence of all elements in the range of atomic number 22 to 92 except chromium and copper (which are masked by the presence of these metals in the instrument). Results from one well, which are typical, are shown in Table 78-Q4-7.

Samples from Wells #100, 101, 102, and 105 and from five other wells in the Michigan Basin have been extracted with benzene to obtain the bitumens. In most of the samples the bitumens constituted about 0.2-0.4% of the raw shale, or 2-4% of the organic material. However, samples of cuttings from two of the wells had ranges of 0.3-0.7% and 0.4-1.0%. A comparison of the total organic content and bitumen content for one well is shown in Figure 78-Q4-4.

Work on characterization of kerogen by Dr. Sandel's group at MTU has established that the H/C ratio in the kerogen is very nearly unity. The functional group analysis of kerogen concentrate from Well #100, 1220.5-1221.0 feet, has been completed. The results are shown below:

Functional Group	Milliequivalents Per Gram Carbon	Carbon Atoms/ Function	% of Oxygen Accounted
-COOH	0.21	400	6.5
-OH	1.78	47	28.0
$>C=O$	0.24	347	3.8
-COOR	1.1	79	33.0
$>C=C<$	1.6	57	--

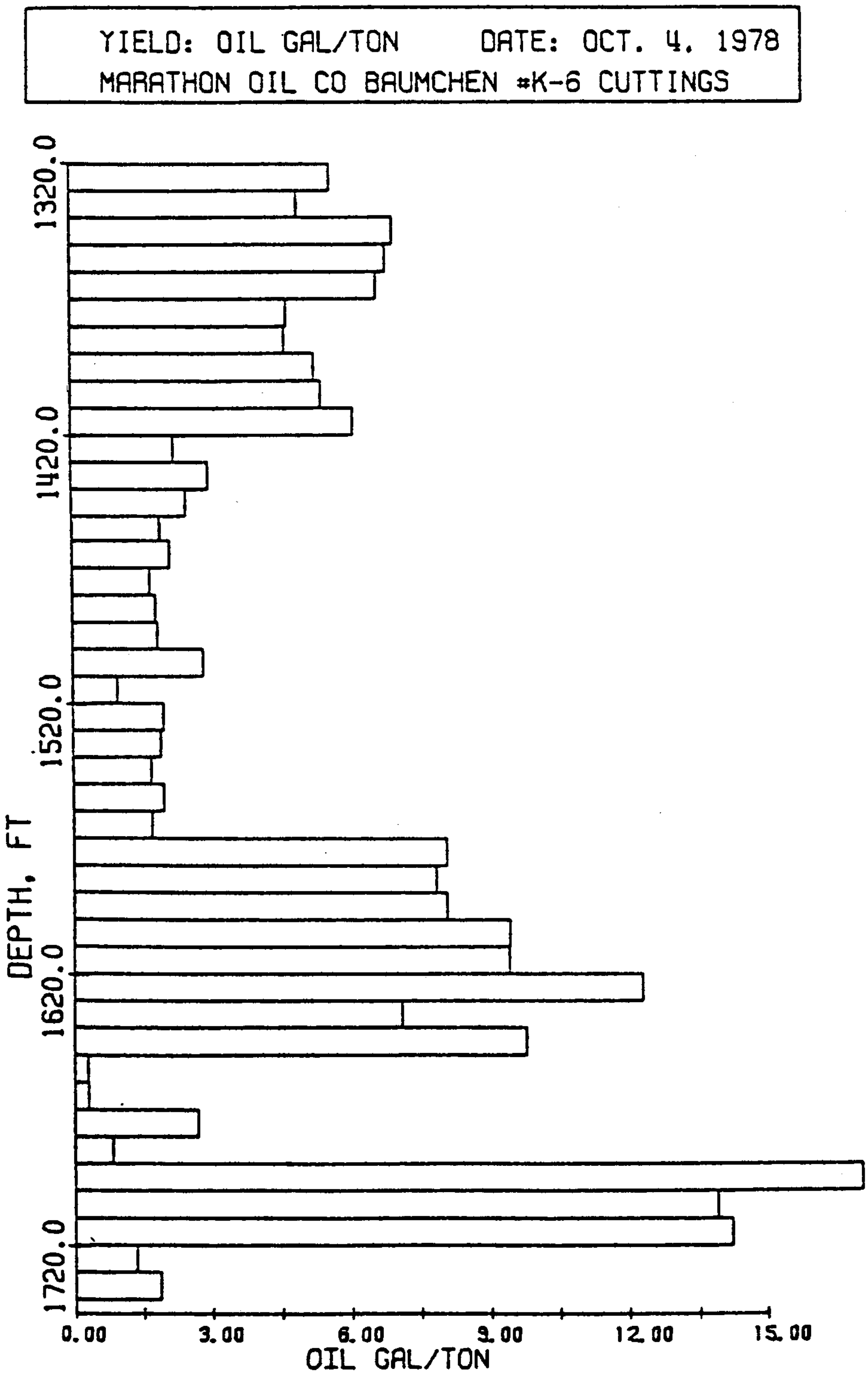


FIGURE 78-Q4-3

FISCHER RETORT ASSAY

TABLE 78-Q4-6

METALLIC ELEMENTS IN RAW SHALE
Well #100

Number[5]	% Ca[1*]	% Al[2*]	Sn(ppb)[3a]	V(ppm)[3b]	% Br[4]
1160	0.83	10.44	1200	102	0.30
1170.2	1.56	10.11	13750	188	0.31
1180.5	0.71	10.25	7500	125	0.24
1190	0.44	12.22	2500	115	0.42
1200	0.83	10.98	4050	125	0.21
1209.8	0.99	7.16	950	782	0.23
1220	5.34	7.05	625	1025	0.28
1230	0.68	6.61	925	562	0.86
1240	0.73	6.13	2188	812	0.54
1250.2	0.68	7.39	625	90	0.24
1260	1.45	6.97	3750	122	0.28
1276	1.17	7.78	312	105	0.26
1280	1.88	8.16	950	68	0.26
1290	2.77	6.12	300	125	0.28
1300	1.45	6.72	1300	102	0.80
1310.34	1.21	5.29	312	125	0.96
1320	2.39	6.07	300	120	0.51
1334	1.40	5.12	312	80	0.38
1340	1.67	5.48	500	70	0.46

TABLE 78-Q4-6 (continued)

Number[5]	%Ca[1*]	%Al[2*]	Sn(ppb)[3a]	V(ppm)[3b]	%Br[4]
1350.5	16.81	6.87	300	80	0.26
1360	3.86	5.15	150	78	0.65
1370	1.14	5.97	312	72	0.54
1379.9	1.39	5.49	300	60	0.12
1390	1.15	5.89	375	72	0.11
1400.2			1675	80	0.36
1410.1	0.33	4.99			1.12
1420.1	2.05	6.88	625	145	1.30
1430.6	5.15	7.99	312	102	0.14
1440.2	15.73	2.09	1562	60	0.39
1450.1	2.08	0.96	925	0	0.31
1460.1	8.61	8.48	625	102	1.45
1470	11.16	7.29	625	78	1.44

[1]Perkin-Elmer 305B Atomic Absorption Spectrophotometer--air-acetylene flame (oxidizing)

[2]Perkin-Elmer 305B Atomic Absorption Spectrophotometer--nitrous oxide-acetylene flame (reducing)

[3]Perkin-Elmer HGA 2100 Graphite Furnace: (a) Dry--125°, 20 sec; Char--650°, 35 sec; Atomize--2700°, 10 sec (b) Dry--100°, 40 sec; Char--1500°, 26 sec; Atomize--2750°, 8 sec

[4]Schöniger Method (modified)

*$LiBO_2$ fused shale

[5]Sample depth - feet

Table 78-Q4-7

Qualitative X-Ray Fluorescence Analyses
of Composited Well Samples

Michigan Consolidated Gas Company 770

SAMPLE	TI	FE	NI	ZN	RB	SR	ZR	MO	PB
1170-1180	MOD	STR	WK	WK	WK	WK	WK		
1180-1190	MOD	STR	V.WK	WK	WK	WK	WK		
1190-2000	MOD	STR	WK	MOD	WK	V.WK	WK	WK	
1230-1240	MOD	STR	WK	WK	WK	V.WK	WK	V.WK	
1240-1250	MOD	STR	WK	WK	WK	V.WK	WK	WK	WK
1250-1260	MOD	STR	WK	WK	WK	V.WK	WK	V.WK	
1260-1270	MOD	STR	V.WK	WK	WK	V.WK	WK	V.WK	
1270-1280	MOD	STR	V.WK	WK	WK	V.WK	WK	V.WK	
1280-1290	MOD	STR	WK	WK	WK	WK	WK	V.WK	
1290-1300	MOD	STR	WK	WK	WK	WK	WK	V.WK	
1300-1310	MOD	STR	WK	WK	WK	V.WK	WK	WK	
1310-1320	MOD	STR	WK	WK	WK	WK	WK	V.WK	
1320-1330	MOD	STR	WK	WK	WK	V WK	WK	V.WK	
1330-1340	MOD	STR	V.WK	WK	WK	V.WK	WK	V.WK	
1340-1350	MOD	STR	WK	WK	WK	V.WK	WK	WK	
1350-1360	WK	STR	V.WK	WK	TR	WK	V.WK	TR	
1360-1370	MOD	STR	WK	V.WK	WK	WK	WK	TR	
1370-1380	MOD	STR	TR	WK	WK	V.WK	V.WK		
2000-2010	MOD	STR	WK	WK	WK	V.WK	WK	WK	
2010-2020	MOD	STR	V.WK	WK	WK	WK	WK		
2020-2030	MOD	STR	WK	WK	WK	V.WK	WK	WK	

TR = .005 - .05%

V.WK = 0.01 - .1%

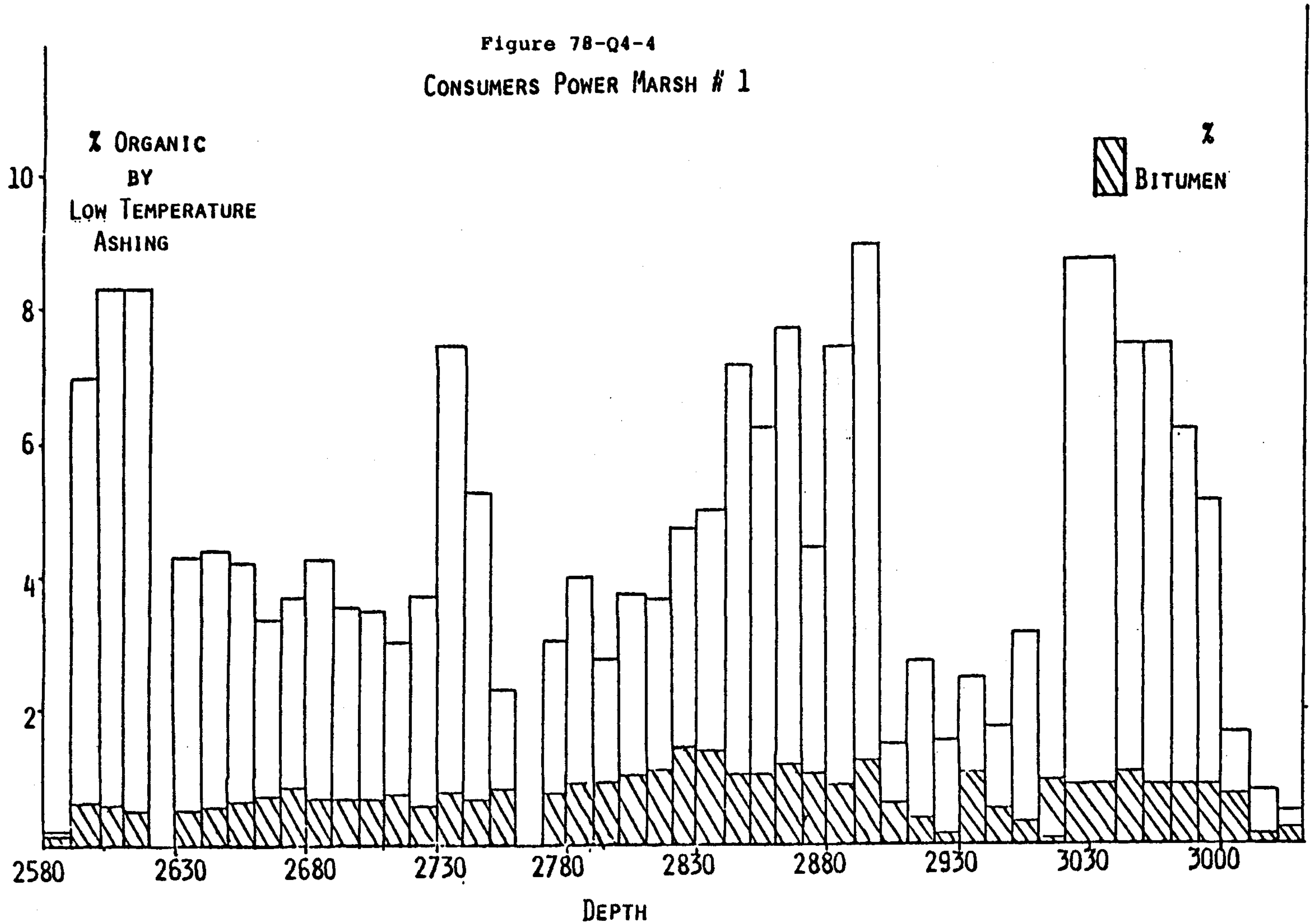
WK = 0.05 - .5%

MOD = 0.5 - 5%

STR = 1 - 10%

Figure 78-Q4-4

CONSUMERS POWER MARSH # 1



Procedures for the isolation of bitumens on a large scale (∿40 kg of shale) have been developed by Dr. Grace Swartz at MTU. The procedure was refined using 14 kg of shale from the Paxton Quarry. The final composite bitumen sample was 32.5 g or 0.23% of the shale extracted. Work on the 1328-1338 ft section of Well #102 is underway. To date, 4.9 kg of shale has been processed to obtain ∿20 g of bitumens. These materials will be treated with various solvents and chromatographic treatments to separate them into fractions for further examination.

In related research at MTU, Dr. Kenny has found that the porphyrin concentration in Antrim shale is much smaller than in samples of Western shale. These materials exist as a mixture of vanadyl (VO) and nickel complexes. Dr. Gulick continues his studies of the organic material using electron spin resonance as a tool. By comparison with previously reported work, he concludes that the vanadyl centered porphyrins in Antrim shale are similar to known species but not identical. The most striking difference indicates that the molecule being observed is much larger than simple porphyrins or that it contains substituents which inhibit free rotation in solution.

A study was recently completed by Dr. Piccirelli, et al, at WSU using the distributed parameter pyrolysis model to determine the quantity of heat required to produce a fixed quantity of pyrolysis product. The results indicate that approximately 5,000 to 6,500 calories are required to produce a gram of product. The thermogravimetric analysis (TGA) data indicate that about one gram of char product is produced for each gram of pyrolysis product. Thus, the overall process appears favorable, since about 35,000 calories are released per gram of char combusted. This conclusion is highly dependent on the value of certain parameters (external heat transfer coefficient, reaction enthalpy, etc.) and assumes that the heat generated by char combustion can be efficiently transported to the pyrolysis areas.

The above calculation is essentially an adiabatic calculation of a well rubblized bed. The results demonstrate that an energy surplus is available from the char combustion to provide energy for the pyrolysis. A second calculation includes transport of the energy from separated combustion and pyrolysis zones. This configuration would correspond to retorting along a crack or fracture, with energy being transported via the flowing gas from the combustion zone to the downstream pyrolysis zone. The conclusion from this calculation is that this mode of operation is marginal with substantial energy transport losses. A combined combustion-pyrolysis system is indicated with combustion and pyrolysis occurring in adjacent, connected shale areas. Energy transport losses are thus reduced due to the decreased transport distance. Furthermore, substantial combustion of the char is a requirement for energy production. That is, it is not possible to sustain the process by partial combustion of the oil or gaseous pyrolysis products.

The multi-kilogram furnace is completely operational and the sample holders for powdered and chunk samples of oil shale have been constructed. Some preliminary runs, using inert aluminum oxide beads as a sample, have demonstrated our ability to properly maintain the correct furnace temperature. Some preliminary data on actual powdered samples of Antrim shale are expected shortly. This equipment is necessary for measuring the temperature profiles within a single chunk of retorting shale.

The char combustion model of H. Gordon Harris has been modified to represent semi-infinite slabs of Antrim shale. The computed results demonstrate that combustion of the char is not favored for the semi-infinite slab configuration. The phenomenon is due to the slow rate of oxygen diffusion into the shale to react with the char. The heat produced by this diffusion limited reaction is conducted to the surrounding shale faster than it can accumulate to accelerate the reaction.

The numerical solution technique in this code was found to be successful for only a limited range of parameters. Attempts to use an arbitrary initial temperature profile or extended pressures were unsuccessful. However, the calculation did appear to indicate that the char combustion is increased with increased pressure.

Work is also proceeding towards developing a combined combustion-pyrolysis model for semi-infinite slabs of Antrim shale. A set of equations to represent the mass transport have been developed but numerical solution has not been attempted yet.

Several currently existing retort models (Lawrence Livermore, Laramie, and Nuttall code) are being closely examined for possible use for Antrim shale. While these codes are designed primarily to represent above ground rubblized retorts, it is felt that they will provide qualitative information on the Antrim.

The development of these existing codes is expected to be completed in three stages. In the first stage, several sets of test data will be compiled. These sets will include data on both Western and Antrim shales and will be developed primarily from the published data on the Laramie 10 ton retort.

The second stage of the development will be to run these data and compare the results against the actual retort performance results. This will provide an excellent opportunity to compare the codes and determine their strengths and weaknesses.

The final stage will be to change the input data to represent possible in situ Antrim configurations. These changes could include larger particles, high pressures, low bed porosity, etc. A list of the possible questions that these retort models might answer is currently being developed with Dow and will be available shortly.

WORK FORECAST

Shale characterization and resource inventory work will continue throughout the life of the contract. Work will continue on well cuttings samples from wells throughout the Michigan Basin.

HYDRAULIC FRACTURING AND ASSESSMENT

Further fracturing in the 100 series wells was accomplished through the use of explosives this quarter. Hydraulic fracturing, sand propping, and notching had been completed in June, and brine removal had begun.

The brine removal operation was completed early in July. Gas samples were taken. The analysis showed mainly air with only traces (<0.1%) of CO_2 and C_1 to C_4 hydrocarbons. On July 12 and 13, Wells #102, 104, 105, and 106 were rigged with primacord and boosters, and 2,300 to 3,200 pounds (1,040 to 1,450 kilograms) of Stratablast C slurry explosive was pumped into each well. About 18 cubic feet (0.5 m^3) of pea gravel was added on top of the explosive to stem the upward impact.

The detonators in the four wells were fired simultaneously, but there was no indication that the main charges exploded. Investigation by the manufacturer revealed that on this particular batch, poor mixing procedure during manufacturing failed to sufficiently hydrate the gums used as suspending agents and that, although the slurry had an average density of 1.3 grams/cubic centimeter, the incomplete hydration allowed the solids to separate and the slurry to be diluted by bore hole fluids. This reduced the sensitivity, and thus the explosive could not be set off by ordinary primers. A companion shot in Well #301 using a properly prepared explosive was highly successful.

Seismometers had been set up by the Environmental Research Institute of Michigan (ERIM) around the perimeter of the property. They had been previously calibrated so that a detonation of as little as 200 pounds (91 kilograms) of explosive should have been detectable. These and sound level meters that had been set up by ERIM showed a much lower response for these 100 series shots than would have been expected from successful detonations.

Inspection in the well area later revealed evidence of partially burned explosive slurry. Analysis of a yellowish-white smoke from Well #106 showed 20.5% CO_2 and CO, i.e., products of combustion of the explosives in an oxygen deficient atmosphere, indicating that the explosives did not detonate. Movies taken with a high-speed camera reinforced this view.

Well #102 was capped when the visible venting subsided. The other shot wells could not be capped immediately because the primacord damaged the well casings. The casings were repaired, and the wells were capped on July 26. Gas analyses taken at this time showed Wells #103-105 producing low levels (1 to 2%) of C_1 - C_3 hydrocarbons. An earlier sample (July 20) from Well #103 analyzed 2.66% CH_4 and 0.22% C_3H_6. One from Well #102 analyzed 3.2% H_2 in addition to 0.8% C_1 - C_2 hydrocarbons, 3.8% CO_2 and CO, plus air as the balance.

In order to continue with the explosive fracturing and rubblizing program in the 100 series wells, it was necessary to clean out the bore holes. Procedures were developed and reviewed with explosive experts for assurance that the methods were safe. During cleaning it was found that all of the wells had bridged at various points up to 470 feet above the level at which the explosives were set. The material cleaned out of the wells consisted of pea gravel, bits of shale, explosive slurry, and pieces of spent primacord. This was stored in a diked area. The well cleaning was completed in August after which the explosives program was resumed.

Several modifications in the detonating system were tried during September with varying degrees of success. As a result, a uniform system for loading primer and slurry explosives was developed.

A ten-foot (∿3 meters) long, 3/4-inch (19 cm) diameter length of copper tubing filled with lead was fitted with two copper tubing centralizers. Two or three primers were taped to the area which was protected by the centralizers. Above the copper tubing, groups of primers, alternating with five-foot (∿1.5 meters) long sections of 3/4-inch diameter plastic tubing were laced onto the primacord support line. This combination, referred to as a "stinger", was lowered into place in the well after about 25% of the explosive (in the form of slurry or paste having a dough consistency) had been loaded into the bore hole. The remainder of the explosive was then added to surround the uppermost primers.

Using the system outlined above, on September 26, 1525 pounds (692 kilograms) of Stratablast C slurry explosive was detonated in Well #101, and on September 28, 1576 pounds (715 kilograms) and 1460 pounds (662 kilograms), respectively, were simultaneously detonated in Wells #104 and 106.

Good visual photographic and instrument records and observations were made of these shots. In addition to the ERIM and Sandia seismic measurement installations, we now also have a Dow surface geophone and recorder that can be operated and interpreted by our field people for immediate information on shot intensity.

EXPLOSIVE UNDERREAMING

Prior to the explosive trial in the 300 series, several instruments were installed or tried. A pressure recorder was installed on Well #305, and an echometer was tested and calibrated on Well #301. The echometer is a sonic device for determining brine level in a well. During the morning of July 12, Energy Science and Consultants, Inc. (ESC) rigged Well #301 with primacord and PETN/TNT boosters. Stratablast C was pumped into the well at 128 lb/min (58 kg/min) in intermittent loads which were of about four minutes duration each so as not to overload the primacord in tension. A total of 1570 pounds (712 kilograms) was loaded into the well at 1300 to 1350 feet of depth (396 to 412 meters). A stemming load of 17 cubic feet (0.48 m^3) of pea gravel was added above the slurry.

A high-speed movie camera was set up along with a conventional 16 mm movie camera and a 35 mm still camera. Sandia Laboratories had set up a station for surface motion observation.

The shot was detonated. There was an extremely loud report (145 dB at ERIM's microphones positioned at a distance of 100 yards), followed by a fire plume over 100 feet (30 meters) high. Then grey smoke and debris vented with a roar for several minutes. The pressure recorder in Well #305 showed a small pulse at blast time. The other wells in the 300 series were open but there was no visible venting.

Five three-component seismic stations, set up by ERIM to monitor this shot were positioned around the perimeter of the property at distances ranging from 2,050 feet (0.62 km) to 4,050 feet (1.23 km). All but the most distant station were overloaded by the shot. The data obtained from that one were sufficient to "calibrate" the site so that future similar events will be properly recorded. The ground motion detected at 4,050 feet from Well #301 was 0.01 mm.

The seismic and acoustic measurement techniques of Sandia Labs and ERIM were given a good test. ERIM can now more precisely calibrate their equipment for a shot of this magnitude, and Sandia demonstrated the importance of providing for back-up manual triggering of the recorder at the sensor station. The data from a Sandia manually triggered recorder showed a vertical velocity very close to what was expected for the amount of explosive used, however, there was unexpected low frequency, high amplitude oscillation on the records of the radial and tangential velocity components which is unexplained.

Well #301 was capped when the venting subsided, about thirty minutes after the detonation. Several 20cc samples of the vapors were taken in gas-tight syringes. One set of samples was returned to Midland for mass spectrometer analysis and another set was run in duplicate on the Carle 111-H gas chromatograph. Gas sampling continued until August 8. The hydrocarbon concentration in the gases definitely increased above the level in the pre-shot samples. The mass spectrometer analyses are incomplete at present. Table 78-Q4-8 shows the averaged GC analyses for a pre-shot set of samples and for sets taken on July 20 and September 8. The September 8 data are much the same as those of July 20. Between these two dates, the well had filled with brine and had been pumped out. None of the other 300 series wells showed any gas pressure buildup. Thus it is believed that, although there was some fracturing or rubblization, this was not from an extensive fracture system and the marked increase in methane and other hydrocarbon gases resulting from the explosion must come from a relatively small fracture system or one oriented in a manner such that it did not intersect the other 300 series wells.

Figure 78-Q4-5 is the caliper log of Well #301. It shows that most of the effect of the explosive occurred in the upper 30 feet (∿9 meters) of the treated zone. The lower 20 foot sections was relatively little changed. In the section between 1305 and 1325 feet, the hole was enlarged to twice its diameter in some zones. The average expansion of the cavity is indicated by the dashed line on the log as having approximately a 13-inch (33 centimeter) diameter. Over this 20 foot section, the increase in bore hole volume is about 171%, calculated as follows:

Pre-shot bore hole volume (20 ft of 7-7/8 inch diameter hole):

(20 ft x 0.34 cu ft/ft) = 6.8 cu ft

Post-shot bore hole volume (20 ft of 13 inch diameter hole):

(20 ft x 0.92 cu ft/ft) = 18.4 cu ft

Net increase:

$$\frac{(18.4 - 6.8)}{6.8} \times 100 = 171\%$$

In preparation for the second of four shots in the underreaming experiment, Well #301 was cleaned using the sand pump method. Cleanings consisted of pea gravel, sand, and shale broken off the bore hole sides.

TABLE 78-Q4-8

GAS ANALYSIS, WELL #301[3]
MOLE %

Component	Pre-shot, 7/12/78	Post-shot, 7/20/78	Follow-up, 9/8/78
Hydrogen	--	det.[2]	--
Propane	trace[1]	1.82	0.68
Propylene	trace	trace	trace
Isobutane	trace	0.12	trace
n-Butane	det.	0.52	0.15
Isobutene	trace	trace	trace
trans-2-Butene	trace	trace	trace
cis-2-Butene	trace	trace	trace
n-Pentane	trace	det.	--
CO_2	trace	trace	trace
Ethane	--	3.60	1.89
O_2	22.20	12.04	18.05
N_2	77.37	61.67	68.92
Methane	det.	14.50	10.14
CO	--	5.23	--

(1) Less than 0.1%

(2) Detected in one sample, but not in the duplicate

(3) Analysis by mass spectrometer having a lower detection limit of 0.02% showed no additional components

WELL DOW/DOE 301 CAVITY SHOT No. 1 1350 Lbs. STRATOBLAST C 7/12/78

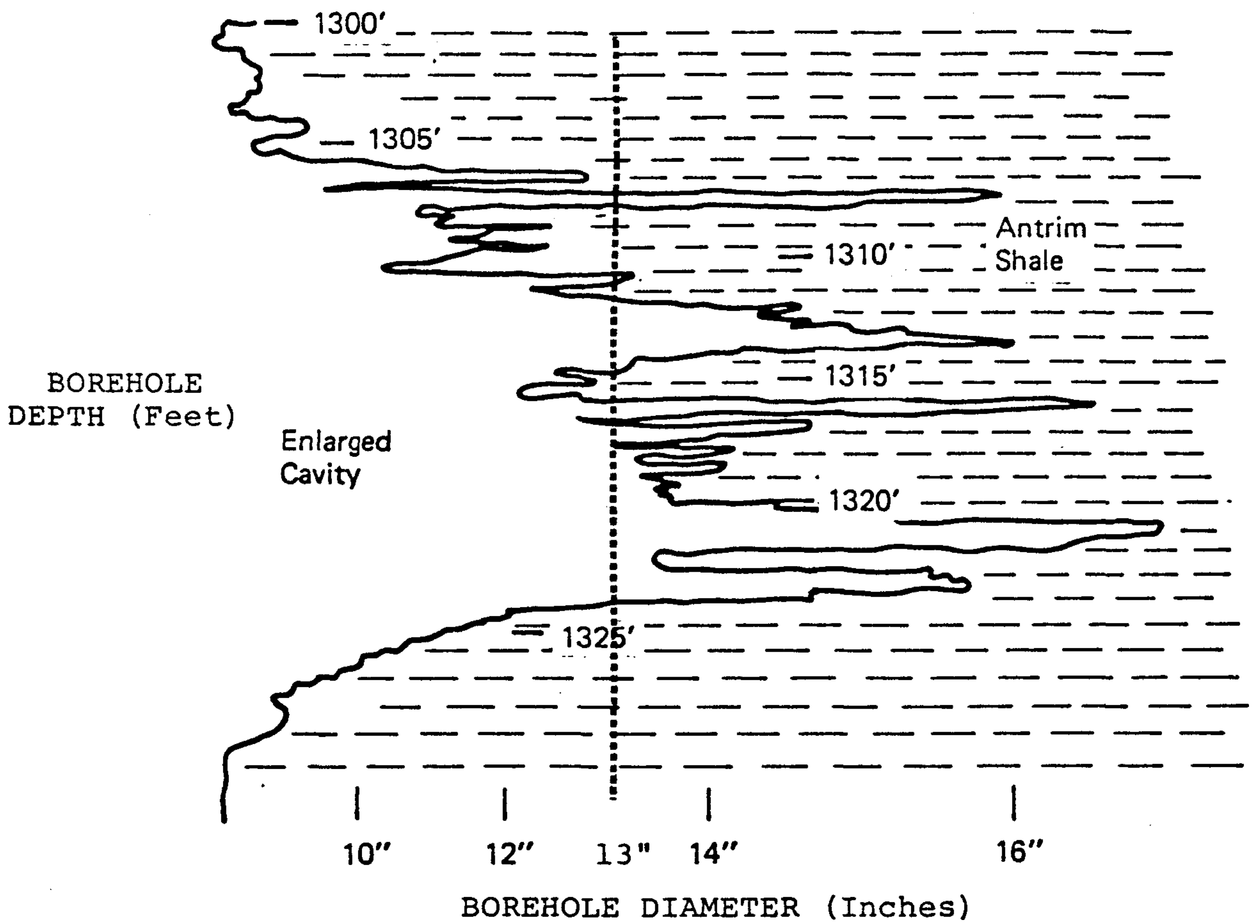


FIGURE 78-Q4-5

CALIPER LOG OF WELL #301 TAKEN 8/3/78
AFTER 7/13/78 EXPLOSIVE SHOT #1

On September 11, preparations were begun for a second explosive underream shot in the 300 series wells. Pressure recorders were installed on Wells #302 through 305. A differential temperature log run in Well #301 indicated a brine depth of 40 feet (12 meters).

Personnel from Energy Sciences and Consultants, Inc. (ESC) were on hand to supervise the placement and detonation of their slurry explosive. A plastic pipe "stinger" with several 6 oz (170 g) primers was lowered to 1355 feet (413 meters). Total well depth was 1359 feet. A truck mounted, plunger-cylinder system for slurry loading was used to load slurry explosive into the well bore on top of the stinger.

After 455 pounds (206.3 kilograms) of explosive was transferred into Well #301, it was discovered that three barrels (6.7 m^3) of water had inadvertently been pumped in below the slurry. The stinger assembly was pulled up 60 feet (18 meters), and loading continued until 1905 pounds (864 kilograms) of explosives had been loaded. The stinger was lowered 30 feet back down the well and released. A primer line was lowered down to rest beside the stinger. A top load of 830 pounds (377 kilograms) of slurry explosives was pumped directly into the well from the ESC truck.

The attempt to set off the shot failed as did a second attempt the next day. During the second attempt, booster primers and 300 pounds (136 kilograms) of explosives were lowered on top of the existing charge. On September 13 a weighted, 60 foot (18 meter) primacord section with clusters of 12 oz (340 g) primers was loaded on top of the charge. Detonation of this did not appear to set off the main charge.

The bore hole casing suffered some damage and was reamed out. The well was flushed out, and a level of 1320 feet (402 meters) had been reached at the end of the quarter. Further flushing and a cleanout program are scheduled.

Photographic monitoring of the explosive shots is an effective procedure for obtaining quick and sufficient documentation for field decisions. All of the explosive shots have been photographically monitored.

CHEMICAL UNDERREAMING

Laboratory Work

On July 20-21, acid solubility tests were run on crushed, -100 mesh, core samples from Wells #203 and 205. Results of the solubility tests are shown in Table 78-Q4-9 along with Dowell data previously obtained with Well #201 core samples. Note that for the Well #201 tests, the sample size was one gram, acid temperature was 150°F, and uninhibited 15% HCl was used. The data show that the material of highest solubility is located in the depth range of 1427 to 1431 feet (435 to 436 meters). The average solubility of crushed material is 74% in that zone. This is the same zone as the high solubility zone identified by mini-well testing.

Solubility rate testing showed the reaction to be essentially complete in less than one-half hour with excess acid. The reaction rate downhole, at 1100 psi (7.6 MPa, gauge pressure), may be 0.10 to 0.14 of that at atmospheric pressure. This is because the pressure inhibits the release of CO_2 and the diffusion controlled reaction is slowed.

The mini-well testing with 28% inhibited hydrochloric acid has been completed. The data for weight percent calcium carbonate reacted are given in Table 78-Q4-10. The average solubility agrees closely with that reported for the crushed material.

Porosity and air permeability measurements were performed on cores from Wells #203 and 205 by Core Laboratories, Inc. Results of these analyses are shown in Tables 78-Q4-11 and 78-Q4-12. Average values of porosity, permeability, and solubility in the depth range of 1427 to 1431 feet for the 200 series wells have been used as inputs for the Dowell Acid Guide II computer program. Initial computer runs indicate that approximately 30,000 gallons (114 m^3) of 28% inhibited hydrochloric acid will be required for acid fracturing in the next field experiment.

Field Work

Ultrasonic thickness measurements on the 9-5/8 inch diameter (24.4 cm) main casing of Well #201 indicated that severe localized corrosion had occurred. Well #201 was flushed with 1680 gallons (6.4 m^3) of brine followed by 3948 gallons (14.9 m^3) of fresh water to remove residual hydrochloric acid from the first field experiment in January 1978. The acid concentration was reduced from 4.1% to 0.0025% by the flushing treatment. The casing has been repaired and hydrostatically proof tested at 900 psig (6.2 MPa, gauge). The proof test of the Well #201 casing was based on the calculated operating pressure expected during the upcoming acid fracturing experiment.

TABLE 78-Q4-9

RESULTS OF DOWELL ACID SOLUBILITY
TESTING OF WELLS #201, 203, & 205 CORE SAMPLES

Actual Depth, ft (Ref Ground Level)	Well #201, Wt % Soluble	Well #203, Wt % Soluble	Well #205, Wt % Soluble
1415.5	--	8.2	--
1416.5	--	5.9	18.4
1417.5	--	12.8	6.4
1418.5	--	14.4	4.0
1419.5	--	17.6	20.8
1420.5	14.7	18.2	32.0
1421.5	33.3	17.0	74.4
1422.5	70.1	29.8	64.8
1423.5	68.6	48.2	72.2
1424.5	76.6	77.8	60.8
1425.5	27.3	65.2	73.2
1426.5	72.3	68.6	48.4
1427.5	93.6	81.4	73.2
1428.5	39.4	53.2	75.6
1429.5	92.5	83.6	68.2
1430.5	95.1	74.8	68.8
1431.5	5.2	82.6	82.4
1432.5	7.1	79.2	63.4
1433.5	34.5	77.8	71.0
1434.5	47.7	--	13.4

Well #201: 1 gram sample, 15% HCl @ 150°F

Wells #203 & 205: 1/2 gram sample, 28% inhibited HCl @ 72°F

TABLE 78-Q4-10

LABORATORY MINI-WELL CHEMICAL UNDERREAMING TEST DATA FOR WELLS #201, 203, & 205 USING 28% INHIBITED HCl

Depth Below Ground Level, (ft)	Weight Percent $CaCO_3$ Reacted		
	Well #201	Well #203	Well #205
1424.5	1.72	--	--
1426.5	60.37	--	--
1426.8	29.50	--	86.24
1427.5	85.33	72.94	--
1428.0	7.46	--	10.68
1429.0	85.21	--	--
1429.5	78.39	81.90	68.12
1430.0	59.40	--	--
1430.5	87.85	69.52	84.80
1431.0	72.60	--	--

Excluding the data for 1424.5 and 1428.0 feet, the average calcium carbonate reacted was:

Well #201	69.8%
Well #203	74.8%
Well #205	79.7%

Average, all wells	74.8%

TABLE 78-Q4-11

RESULTS OF CORE LABORATORIES' ANALYSES FOR WELL #203

Actual Depth, ft (Ref - Ground Level)	Crossflow Air Permeability Max, md	Crossflow Air Permeability Rotated 90°, md	Porosity (Gas Expansion), %	Grain Density, gm/cc
1419.1 - 1420.1	1039.0	4.8	7.6	2.76
1420.1 - 1421.1	0.9	0.6	8.3	2.79
1421.1 - 1422.1	169.0	136.0	5.3	2.76
1422.1 - 1423.1	1878.0	8.3	6.7	2.78
1423.1 - 1424.1	*	<0.1	3.4	2.76
1424.1 - 1425.1	6.1	0.1	1.4	2.77
1425.1 - 1426.1	62.0	3.3	4.1	2.79
1426.1 - 1427.1	*	<0.1	3.2	2.85
1427.1 - 1428.1	<0.1	<0.1	0.8	2.81
1428.1 - 1429.1	264.0	1.5	1.8	2.76

*Indicates that the permeability measurement was made on a plug from the core.

TABLE 78-Q4-12

RESULTS OF CORE LABORATORIES' ANALYSES FOR WELL #205

Actual Depth, ft (Ref - Ground Level)	Crossflow Air Permeability		Porosity (Gas Expansion), %	Grain Density, gm/cc
	Max, md	Rotated 90°, md		
1422.2 - 1423.2	*	15.0	5.3	2.71
1423.2 - 1424.2	76.0	0.3	1.2	2.75
1424.2 - 1425.2	78.0	0.4	2.0	2.74
1425.2 - 1426.2	3.4	0.5	1.3	2.76
1426.2 - 1427.2	272.0	<0.1	0.6	2.79
1427.2 - 1428.2	96.0	<0.1	0.9	2.74
1428.2 - 1429.2	15.0	1.0	1.9	2.75
1429.2 - 1430.2	2.8	0.2	1.0	2.79
1430.2 - 1431.2	91.0	<0.1	1.0	2.78
1431.2 - 1432.2	1781.0	<0.1	7.3	2.81

*Indicates that the permeability measurement was made on a plug from the core.

A system for on-line pH measurements in the perimeter wells of the 200 series during the acid fracturing experiment is being bench tested. Information on acid concentration will be useful in adjusting the acid pumping rate to optimize acid retention time in the formation.

Research Visit To Dowell

People from Dow, Dowell (Mt. Pleasant) and Lease Management visited Dowell at Tulsa, Oklahoma to verify the assembly sequence and identify potential safety and handling problems for the rig crew prior to running the Abrasijet* ejector (a perforating tool) test in Well #201. The assembly sequence was demonstrated, the necessary handling equipment identified, a mock-up tool lowered to 220 feet (67 meters) into a test well, and the tubing lines pressure tested. The tool will be used to create a deep radial notch in a limestone stringer at a depth of 1429 feet (435.6 meters). A horizontal acid fracture can then be initiated from this notch in the central well to intercept the perimeter wells.

WORK FORECAST

The final explosive systems to be tested in the 100 series will be shot and evaluated in October. Well cleanout and post-explosive liquid permeability tests between key wells will start immediately after explosive fracturing. Other wire line evaluation methods and air permeability testing will be used to define the location and extent of the fracture system. Post-explosive gas and fluid sampling will continue. It is also planned to conduct tracer gas permeability tests of the fracture zones. Additional monitoring wells will be located and drilled after the fracture system has been studied.

As Well #301 cleaning continues, a small charge will be set off downhole to insure that all primers have been detonated. The second bore hole expansion shot will use the stinger system developed in the 100 series wells. This shot is scheduled midway in the quarter with the third and fourth, progressively larger shots scheduled for near the end of the quarter. Bore hole cleaning will become increasingly difficult as the chamber is expanded.

Fabrication of the Abrasijet ejector is expected to be completed late in October. Following this, the notching of Well #201 will be completed, and the dimensions of the resulting notched zone will be measured. Preparations for the acid fracturing field experiment will be finalized, the efforts of the several contractors involved will be coordinated, and the acid fracturing will be performed and evaluated. A test plan will be prepared for using *in situ* and bore hole explosives in the 200 series wells.

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IN SITU EXTRACTION TRIALS

EXISTING DOW SITE

Clean out of Wells #4 and 7 started this quarter. Well #7 was cleaned to the total depth, 1,388 feet (423 meters). Samples of the debris and rubble were taken from the removed material for every 10 feet (3 meters) of depth cleaned. These will be evaluated to learn about burning conditions and to provide a basis for estimating the amount of unburned charcoal in the well. Repeated attempts to clean Well #4 were less successful. Initially, the well bore was found to be bridged at 1160 feet of depth (354 meters). This is 10 feet (3 meters) below the casing in the bottom of the Bedford shale formation and the top of the false Antrim shale. The walls kept caving in so the sand pump was not able to reach the underreamed cavity below 1310 feet (399 meters) to clean out burn residue. Approximately 4 cubic yards (3 m^3) of detrital shale was removed between 1160 and 1170 feet. A decision whether or not to continue these attempts is under study.

The wells on the existing site were prepared for fluid lifting. The fluid levels will be kept at a minimum in the ten wells so that the formations surrounding the bore holes will be relatively dry for future measuring and testing.

Upgrading of the present compressor system and installation of a new compressor to provide a greater quantity of air at high pressure for permeability testing and combustion tests has been delayed. The 1500 psig Ingersoll-Rand compressor originally scheduled for delivery in October has been rescheduled for December. Upgrading of the old compressor system will not begin until the new system is installed and operational. The delivery delay will not affect scheduled field experiments.

Computer stored data from the 61 day extraction trial in Well #7 were examined, culled to eliminate non-representative data, and entered into new computer files. The data processing and plotting programs were augmented and tested. Over 200 files of raw and processed data from the extraction trials made between August 1977 and June 1978 have been entered into the computer memory. The data reduction is essentially complete. Plotting and interpretation are in progress for a topical report on the three major extraction runs made during this period.

Table 78-Q4-13 gives a comparison of the input data for the three major extraction trials since the beginning of the contract. The amount of injected gas recovered was nearly identical in each case. This was also true for nitrogen recovery. The oxygen recovery was less complete, i.e., in the 45-55% range. This was to be expected as some of the oxygen reacted to form water during combustion, and other amounts would have been lost due to the higher solubility and reactivity of oxygen compared to nitrogen. For many weeks after each experiment, O_2, N_2 and combustion products continued to be "exhaled" from the formation. Part of the injection gases remained trapped in cracks and interstices in the formation after the pressure was relieved.

The downhole environment is very corrosive and destructive to burners, especially at the high temperatures achieved during ignition. Also, there is not an extensive shale surface exposed to the burner. The loading of coal and charcoal into the ignition wells in CAPB1 and CAPB2 provided a combustible surface and mass such that the combustion could be continued exclusive of the burner operation or the availability of shale surface area in the ignition well. Well #4 had a 4 foot diameter by 10 foot depth (1.2 x 3 meters) underreamed cavity. This permitted the loading of over 4-1/2 tons ($\sim$4000 kg) of solid fuel in the well. About 1/2 ton (454 kg) of charcoal was placed in Well #7. The formation around Well #7 was much tighter than the environment of Well #4. These two conditions, amount of solid fuel and formation tightness, accounted for most of the difference between runs CAPB1 and CAPB2.

The output or production data for the trials are given in Table 78-Q4-14. The production gases shown are the total of all production wells for the duration of the trial. Individual well data are available but not reported here.

The data make it clear that run CAPB1 out-produced the other runs. This is due in part to the higher permeability of Well #4 (used for injection in CAPB1) relative to Well #7 (used for CAPB2) and the increased injection air flow in CAPB1 relative to EB3 (both in Well #4). The large quantities of supplemental fuel loaded in Well #4 for CAPB1 also impacted on the results. Accurate energy balances around tests CAPB1 and CAPB2 are not possible at this time. The results of the balance are quite dependent on how much supplemental fuel (coal and charcoal) was burned in each well. The data for EB3 are not affected by supplemental fuel combustion.

TABLE 78-Q4-13

INPUT DATA FOR EXTRACTION TRIALS ON EXISTING WELLS

Designation:	Electric Heater Trial 3, EB3	Coal Augmented Propane Burns CAPB1	Coal Augmented Propane Burns CAPB2
Ignition Well	Well #4	Well #4	Well #7
Duration of Run	11/12-12/6/77 24 days	2/3-2/27/78 24 days	3/24-5/24/78 61 days
Input Gas Volume, SCF x 10^3			
N_2 From Storage	165	--	--
N_2 From Air	679	2172	1919
O_2 From Air	175	560	495
Total - Well #4	1019	2732	462
- Well #7	--	--	1952
Amount of Injected Gas Recovered, %	69.0	69.2	68.8
Solid Fuel Loaded, lbs			
Coal	--	6250	--
Charcoal Briquettes	--	2215	596
1 x 2 mesh charcoal	--	--	250
4 x 8 mesh charcoal	--	950	400
Fuel Gases Injected, SCF			
Propane	--	960	1440
Methane	--	150	250
Total of Fuel Carbon, lbs	0	7030	951
Total of Fuel Heating Value, Btu x 10^6	0	121.3	17.7

TABLE 78-Q4-14

OUTPUT DATA FOR EXTRACTION TRIALS ON EXISTING WELLS

Designation	Electric Heater Trial 3, EB3	Coal Augmented Propane Burns CAPB1	Coal Augmented Propane Burns CAPB2
Ignition Well	Well #4	Well #4	Well #7
Duration of Run	24 days	24 days	61 days
Total Effluent Gas, SCF x 10^3	(Includes pressure letdown period after the run)		
Nitrogen	604	1521	1349
Oxygen	44.4	192	222
Carbon Dioxide	34.2	109	19.7
Carbon Monoxide	1.7	12	0
Methane	16.7	42.9	59.1
C_2 - C_4 Hydrocarbons	2.3	14.1	12.3
Total	704	1891	1662
Heating Value of Effluent Gas, Btu x 10^6	23.9	94.3	86.7
Average Rate of Effluent Gas Flow, (SCFD) x 10^3	(Not including pressure letdown period)		
Nitrogen	21.55	48.42	22.48
Oxygen	1.69	6.27	3.70
Carbon Dioxide	1.31	3.83	0.33
Carbon Monoxide	0.07	0.48	0
Methane	0.56	1.34	0.98
C_2 - C_4 Hydrocarbons	0.08	0.45	0.21
Total	25.26	61.52	27.70
Heating Value of Effluent Gas, (Btu/d) x 10^6	0.809	3.06	1.44

Over 100 graphs have been prepared to further illustrate the data contained in the tables presented here. These plots will be incorporated into a topical report. Examples of two such plots are shown in Figure 78-Q4-6.

An analysis of the data from the various cyclical operational strategies used in CAPB2 indicate that the technique of alternately injecting and producing from the same well has potential merit as a method of increasing gas production. Gas produced during this period of operation shows an energy increase of 47-65% over that produced during normal operation.

NEW WELL SITE

Preparations continue for systems and technology to be used in extraction trials on the south 40 acres of the fieldsite.

Two ignition systems were tested at the fieldsite:

> A hypergolic system was tested in a lab scale experiment. The system employs the highly exothermic reaction with water of a liquid metal alloy 22 weight percent sodium and 78 weight percent potassium. The ignition of the NaK was immediate upon formation of a drop at the end of a 1/8-inch (0.32 centimeter) stainless steel tube adjacent to an identical tube carrying water. The charcoal into which the flaming NaK dropped was ignited, and a bed temperature of 1200°F (649°C) near the reaction area was observed. A total of one and one-half pounds (0.68 kilograms) of NaK was used in the experiment without difficulty.
>
> Another approach to a reliable, simple ignition system for charcoal in an ignition well was also tested. A graphite-element electric heater was used to ignite charcoal in a test rig. The heater, which was supplied by Los Alamos Scientific Laboratory, had been developed to melt earth and rock. The heater has a molybdenum sheath which is coated with fused silica. It readily caused ignition of the charcoal at an input of 600 watts from a direct current electric arc welder.

Two design concepts are being examined for downhole electric heater use. The first would use the heater as the direct source for ignition of the shale. The second concept would be to use the heater to ignite charcoal, which in turn would ignite the shale. Downhole heater designs are being worked on by two manufacturers.

Corrosion studies made by the Lawrence Berkeley Labs in Berkeley, California and at the Laramie Energy Technology Center above ground retorts were reviewed for applications to *in situ* heater and burner design.

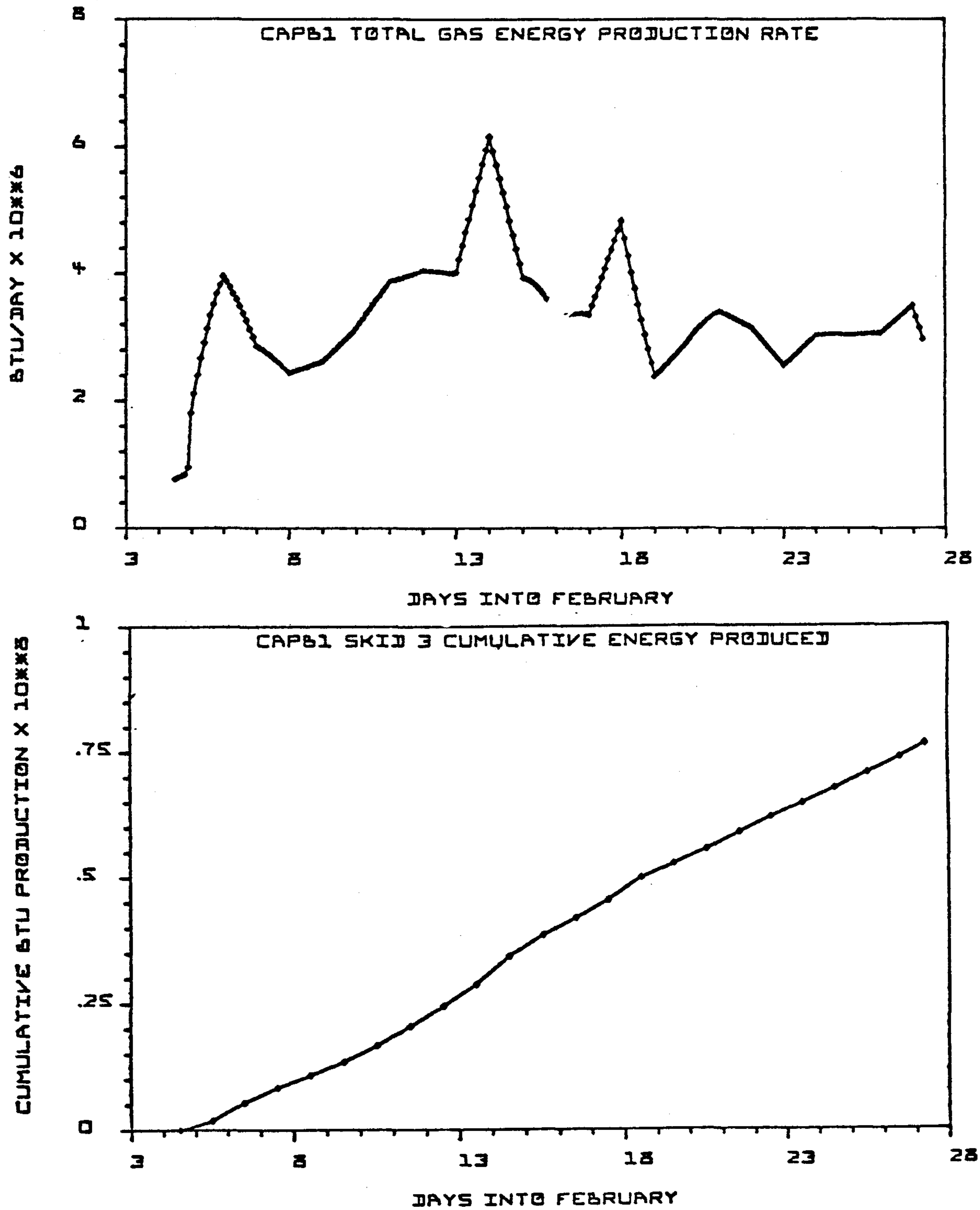

FIGURE 78-Q4-6

GAS ENERGY PRODUCTION RATE AND CUMULATIVE ENERGY PRODUCED IN COAL AUGMENTED PROPANE BURN #1 (CAPB1)

Construction continued on the wellhead skids containing piping and instrumentation for brine lifting, air/N_2 permeability studies, control of air and nitrogen injected during a combustion trial, and collection of effluent gases produced. Six of these skids are being assembled. Three are completed. Most of the remainder of the control system is nearly complete. This includes completion of wiring diagrams and installation of wiring from the front site control room to the back site (south 40 acres) instrument trailer. One skid was connected to Well #102 and was used for the 100 series well dewatering.

The cable tray and pipeline support structure were extended deeper into the south well site and an under-the-road grade crossing was built to carry pipes and wires to the 200 and 300 series wells.

A redesign of the gas sample collection system, featuring a sample conditioning apparatus and a package to protect the instrumentation, is being finalized. The system will trap any salt in the vapor, shut off if brine enters sampling lines, cool and collect condensables, regulate the sample pressure, dry the sample and deliver it to an automatic stream selector valve for analysis or venting. Spare lines will be provided in case of plugging.

A 5 kilowatt uninterruptible power supply was received and installed. The system will supply 120 volts of AC current for two to four hours during a power failure. Such a period will allow time for the diesel generator to be brought onstream. The remote panel has been wired to the generator.

New flowmeters for gas and brine have been purchased. The brine meter is designed to be insensitive to surges of air accompanying the brine. The gas meter has automatic pressure and temperature compensation and provides instantaneous and totalized flow data in standard cubic feet per minute. An analog meter is being modified to allow presentation of the digital output of the flowmeter computer to the analog input of the PDP-8E computer.

A communication loop was established that includes the Sigma 10 integrator serving both the Carle GC on the south field site and the Texas Instruments teleprinter and the PDP-8E computer in the warehouse-office control room on the existing site.

A subcontract proposal for the use of a mathematical modeling approach to guide the planning of the 1979 shale extraction trial was received from Wayne State University. Many useful and fundamentally important questions can possibly be qualitatively answered using modifications of presently available *in situ* bed models. The proposal provides for a stepped-up model testing program so that the 1979 extraction trial will have the benefit of the application of the best available engineering and scientific knowledge concerning the expected reactions in the Antrim shale.

WORK FORECAST

The existing wells on the north site will be kept pumped out pending decisions on further permeability measurements or underground tests.

The compressor installation and the development of the mechanical, electrical and other systems for the *in situ* combustion experiment at the new well site will continue.

LAND MONITORING

Activities during this quarter include acoustic measurements (reported in a prior section), soils and vegetative analysis, installation and survey of elevation monuments and additional aerial photography. Assessment of the nature and extent of surface disturbance was made by direct observation of a number of sites. From these observations, it appears that the main problems created by the drilling operations deal with compaction of the top soil and spilling of the drilling fluid. Removal of the upper part of the soil did not seem to be as large a problem as anticipated.

The vegetation survey was completed during the quarter. The resulting plant list contains 135 species. Further field work was halted after four hours of researching failed to reveal any new plants.

WATER QUALITY MONITORING

During July, August, and the first part of September, conditions remained extremely dry. No runoff from the site or flow in Seymour Creek was observed during this period. When possible, monthly samples were collected from stations located on or near the site. No unusual results were obtained, except that chloride levels remain high in the farmhouse well and in the pond on the site.

The shallow test wells associated with the 300 series mud pits were pumped and sampled. The pit remains dry and partially dug out. The results show that brine contamination from the saturated zone near the mud pit has not moved down gradient even 100 feet (30 meters). Results from the test well 100 feet from the mud pit are comparable to those from a similar well 1200 feet (360 meters) north of the pit.

The farmhouse well was reworked; a new screen and pump were installed. Extensive pumping tests were conducted; the chloride content remained at about 300 mg/l through two tests of about one week each, involving 10-15,000 gallons each.

During September 12 to 15, 1978, when the pond was entirely free of surface water, an extensive examination of the subsurface water in and near the pond began. Eleven test holes were placed around the pond perimeter, using a gas powered screw-type auger, to depths of about 8 feet. Twelve samples were taken from holes drilled into the muck of the pond bottom at various depths. All these samples have been analyzed for chloride ion content.

The results of these tests show that the chloride concentration varies significantly with the location of the sample and with the depth of the sample. The results suggest that the chloride may be leaching from a pit containing drilling muds from wells drilled several years ago in the Dow Rhoburn series. However, much more work will be needed before the source of this contamination can be verified.

WORK FORECAST

Regular monitoring of land, air, and water at the experimental site will continue. Special attention will continue to be paid to the farmhouse well and the pond in an effort to identify the source of the elevated chloride content.

CONCLUSIONS

SHALE CHARACTERIZATION

Work is continuing at four Michigan universities on cores taken from Wells #100, 101, 102, and 201, and on samples of Antrim shale well cuttings from a variety of locations in the Michigan Basin.

IN SITU FRACTURING AND ASSESSMENT

The bore hole explosives program was begun in July and carried on throughout the quarter. Slurry explosive was successfully detonated in Wells #301, 101, 104, and 106. However, considerable difficulty was encountered during the program and there were also unsuccessful shots in these wells. The failures were attributed to off spec product. In the process, a workable, uniform system of rigging primers and loading slurry explosive was developed. Upon cleaning Well #301 after the successful detonation, it was found by caliper logging that the volume of a 20 foot section of the hole had been enlarged by about 170%. The remaining 30 feet which had contained explosives showed relatively little change.

The explosive fracturing and underreaming shots are continuing. Acid solubility and mini-well tests were performed in the lab on samples of core from Wells #203 and 205. The data showed 70-80% solubility of the cores by both tests at 1427 to 1431 foot depth in these wells and in Well #201 tested earlier. Planning and preparation for a fieldsite acid treatment experiment in Well #201 were completed.

IN SITU EXTRACTION TRIALS

A program to clean out the injection wells from the last two extraction trials was begun. Well #7 was cleaned to total depth using a sand pump. Well #4 proved difficult to clean because the walls kept caving in and bridging the well bore; the effort was discontinued.

The computer stored data and the field records for all of the contract extraction trials in the existing well field have been assembled, and a topical report is being developed. Another report covering heaters, burners, and other ignition sources is also being written.

Development continues on systems and technology to be used in extraction trials on the south fieldsite. A test was done on a sodium-potassium alloy plus water as a hypergolic igniter for charcoal. A molybdenum sheathed, graphite element, electric heater supplied by Los Alamos Scientific Laboratory was also used to ignite charcoal in a test rig.

The facilities for *in situ* combustion experiments at the south fieldsite are rapidly nearing completion.

ENVIRONMENTAL, PUBLIC POLICY & LEGAL ASSESSMENT

Monitoring of the air quality, vegetation and land elevations and of ground waters showed no adverse effect due to extraction activity. There has been limited, temporary surface disturbance due to well drilling and the related movement of heavy equipment.

*U.S. GOVERNMENT PRINTING OFFICE: 1979-640-014/4520. Region 4.