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THE UTILIZATION OF THE MICROFLORA INDIGENOUS TO AND PRESENT IN OIL-BEARING FORMATIONS TO SELECTIVELY PLUG THE MORE POROUS ZONES THEREBY INCREASING OIL RECOVERY DURING WATERFLOODING

Annual Report for the Period
January 1, 1994 to December 31, 1994

By
L. Brown
A. Vadie

August 1995

Performed Under Contract No. DE-FC22-94BC14962

Hughes Eastern Corporation
Jackson, Mississippi

Bartlesville Project Office
U. S. DEPARTMENT OF ENERGY
Bartlesville, Oklahoma

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Prepared for
U.S. Department of Energy
Assistant Secretary for Fossil Energy

Rhonda Lindsey, Project Manager
Bartlesville Project Office
P.O. Box 1398
Bartlesville, OK 74005

Prepared by
Hughes Eastern Corporation
200 S. Lamar Street, Suite 1050
Jackson, MS 39201

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ABSTRACT

This project is a field demonstration of the ability of in-situ indigenous microorganisms in the North Blowhorn Creek Oil Field to reduce the flow of injection water in the more permeable zones thereby diverting flow to other areas of the reservoir and thus increase the efficiency of the waterflooding operation. This effect is to be accomplished by adding inorganic nutrients in the form of potassium nitrate and orthophosphate, to the injection water. Work on the project is divided into three phases, Planning and Analysis (9 months), Implementation (45 months), and Technology Transfer (12 months). In Phase I, which has been completed, the following results were obtained. Two new wells were drilled in the field and live cores were recovered. Analyses of the cores proved that viable microorganisms were present and since no sulfate-reducing bacteria (SRB) were found, the area in which the wells were drilled, probably had not been impacted by injection water, since SRB were prevalent in fluids from most wells in the field. Laboratory waterflooding tests using live cores demonstrated that the rate of flow of simulated production water through the core increased with time when used alone while the rate of flow decreased when nitrate and phosphate salts were added to the simulated production water. Since there is only a small amount of pressure on the influent, the simulated production water was not forced to sweep other areas of the core (as would be expected in the reservoir since the water is injected under pressure). The field demonstration (Phase II) involves adding nutrients to four injector wells and monitoring the surrounding producers. The exact kind and amounts of nutrients to be employed and the schedule for their injection were formulated on the basis of the information obtained in the laboratory waterflooding tests conducted using the live cores from the field. Results obtained in these tests will not only be compared to historical data for the wells but also to four injectors and their corresponding producers (control) which were chosen for their similarity to the four test patterns.

A preliminary geological and petrophysical characterization of the reservoir has been made and baseline chemical and microbiological data obtained on all of the test and control wells in the first test pattern (two injector wells and nine producing wells). Injection of nutrients began in the first test well on November 21, 1994.

EXECUTIVE SUMMARY

This project is designed to demonstrate that a microbially enhanced oil recovery process, developed in part under DOE Contract No. DE-AC22-90BC14665, will increase oil recovery from fluvial dominated deltaic oil reservoirs. The process involves stimulating the in-situ indigenous microbial population in the reservoir to grow in the more permeable zones thus diverting flow to other areas of the reservoir thereby increasing the effectiveness of the waterflooding operations. This five and one-half year project is divided into three phases, Phase I, Planning and Analysis (9 months), Phase II Implementation (45 months) and, Technology Transfer (12 months). This first annual report covers the findings in Phase I and the first three months of Phase II.

Phase I began with the drilling of two wells to obtain live cores for laboratory studies and to obtain the production data which would indicate how well the reservoir was being swept by the existing waterflooding. Analyses of the cores proved that viable microorganisms were present and since sulfate-reducing bacteria (SRB) were not present, the area in which the wells were drilled probably had not been impacted by the waterflooding since SRB were prevalent in fluids from most other wells in the field. Laboratory waterflooding tests using live cores clearly demonstrated that the in-situ microflora could be stimulated to grow and retard fluid flow through the core. Control cores injected with only simulated production water increased in flow rate as expected while the test cores injected with water containing potassium nitrate and disodium hydrogen phosphate had reduced flow rates. For the field demonstration, four injector wells were selected for nutrient additions and the surrounding producer wells are/will be monitored. Results obtained in these tests will be compared to historical data for the wells and to data obtained from four additional injectors and their surrounding producers which are being serving as controls.

Preliminary geological and petrophysical characterizations of the reservoir have been made. Baseline data on the inorganic constituents, microbial population, and aliphatic profile, have been obtained for fluids from all of the test and control wells in the first test pattern (two injector wells and nine producing wells).

A tracer study is being conducted on the first test pattern and to date the tracer has been detected in two of the four surrounding wells. It appears that it will require at least a year or longer before evidence of microbial growth in the reservoir can be detected. A skid was designed and fabricated to allow for the addition of the nutrients to the injection water. Three additional skids are nearly completed. The field demonstration (addition of nutrients to the injection water) for the first test pattern began on November 21, 1994. No problems have been encountered in the field operation to date.

INTRODUCTION

This five and one-half year project is designed to demonstrate that the microflora indigenous to petroleum reservoirs can be stimulated to grow in the more permeable zones of the reservoir thereby diverting flow to other areas and thus increasing the effectiveness of waterflooding operations. The concepts involved in this project were developed in part as a result of work performed under DOE Contract No DE-AC22-90BC14665. Work on this project is divided into three phases of nine-month, forty-five month, and twelve-month durations, respectively. This First Yearly Technical Progress Report will cover Phase I (Planning and Analyses) and the first three months of Phase II (Implementation).

Background

The North Blowhorn Creek Oil Unit (NBCU) is located in northwest Alabama about seventy-five miles west of Birmingham. The field is in what is known geologically as the Black Warrior Basin. The producing formation is the Carter Sandstone of Mississippian Age at a depth of about 2300 feet. The field was discovered in 1979 and initially developed on 80 acre spacing. The field was unitized into a reservoir-wide unit in 1983 and in-fill drilled to 40 acre spacing. Waterflooding of the reservoir began in 1983. The initial oil in place in the reservoir was about 16 million barrels, of which 5.4 million barrels had been recovered by the end of 1994. To date, North Blowhorn Creek is the largest oil field discovered in the Black Warrior Basin. Oil production peaked at almost 3000 BOPD in 1985 and has since steadily declined. Currently there are 21 injection wells and 32 producing wells. Current production is about 290 BOPD, 120 MCFD and 3200 BWPD. The current water injection rate is about 4000 BWPD. About 10 MMBO will be left unrecovered if some method of enhanced recovery is not proven to be feasible.

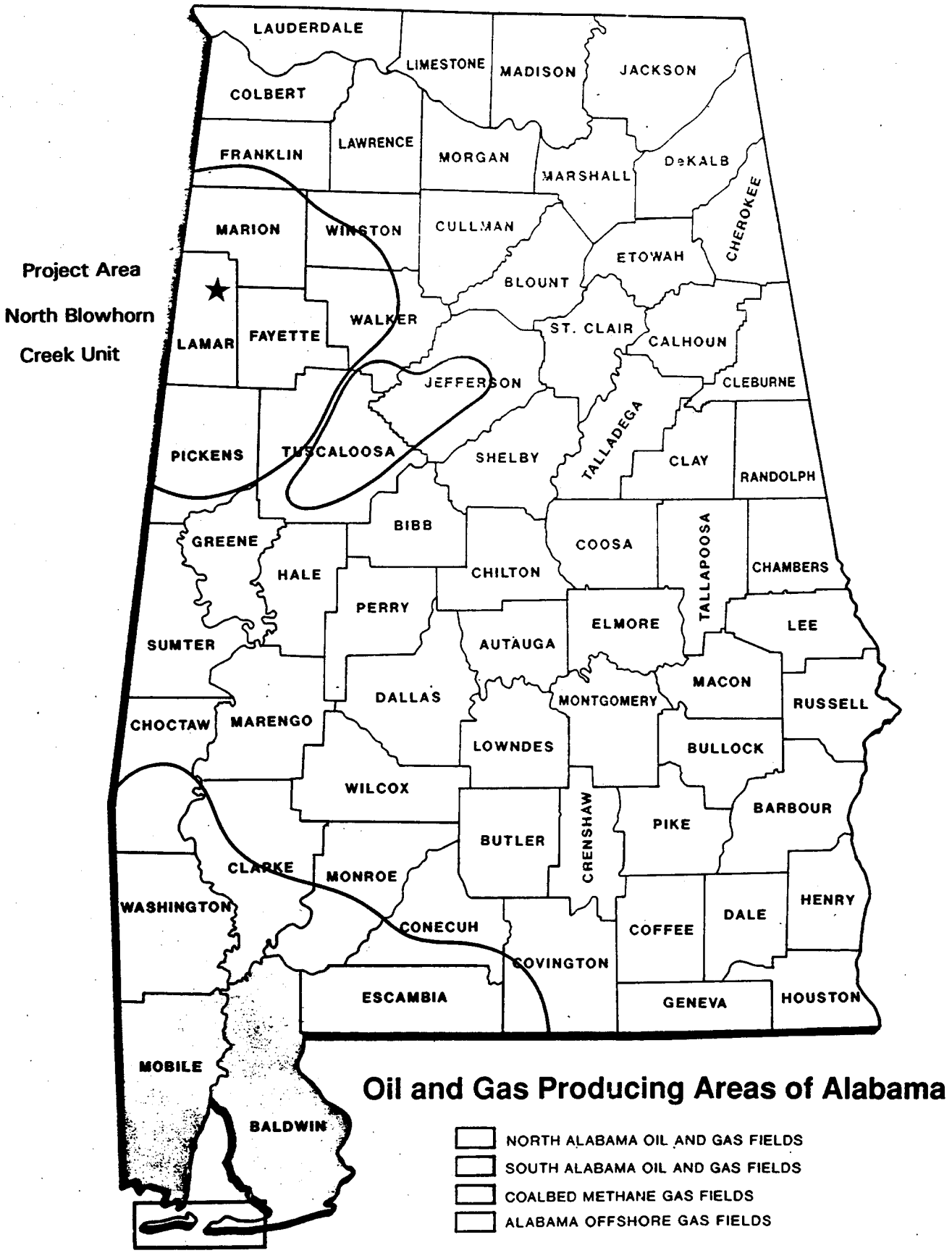

Figure i: Project Area Geographical Locator Map

DISCUSSION

1. OBJECTIVE AND OVERALL PLAN OF WORK

The objective of this work is to demonstrate the use of indigenous microbes as a method of profile control in waterfloods. It is expected that as the microbial population is induced to increase, the expanded biomass will selectively block the more permeable zones of the reservoir thereby forcing injection water to flow through the less permeable zones which will result in improved sweep efficiency.

This increase in microbial population will be accomplished by injecting a nutrient solution into four injectors. Four other injectors will act as control wells. During Phase I, two wells will be cored through the zone of interest. The cores will be subjected to special core analysis in order to arrive at the optimum nutrient formulation. During Phase II, nutrient injection will begin, the result monitored, and adjustments to the nutrient composition made. Phase III will focus on technology transfer of the results.

It should be pointed out that one expected outcome of this new technology will be a prolongation of economical waterflooding operations, i.e. economical oil recovery should continue for much longer periods in areas of the reservoir subjected to this selective plugging technique.

2. PHASE I. PLANNING AND ANALYSIS

The concepts of new technology to be evaluated in this project are scientifically sound and have been proven to be effective in laboratory experiments. Nevertheless, it was deemed necessary to perform laboratory tests on live cores from the reservoir of interest. Two wells were drilled for this purpose and special core analyses have been conducted in order to fine tune the exact concentration of, and schedule for, additions of nutrients to the injection water.

While the main purpose of drilling the two wells was to obtain cores suitable for use in the laboratory work, a secondary purpose was to obtain production data which will indicate the sweep efficiency of the existing waterflood. The two core wells ultimately may be converted to injection wells if this project is successful and the program is expanded.

At the conclusion of Phase I, a specific feeding regime was formulated for the first, and second injection wells. For convenience, the results for this phase have been divided into six sections (a through f).

a. The Drilling of Two New In-Field Wells.

Phase 1 of the project began with the drilling of two wells (see Figure 1) to obtain live cores and production data which would indicate how well the

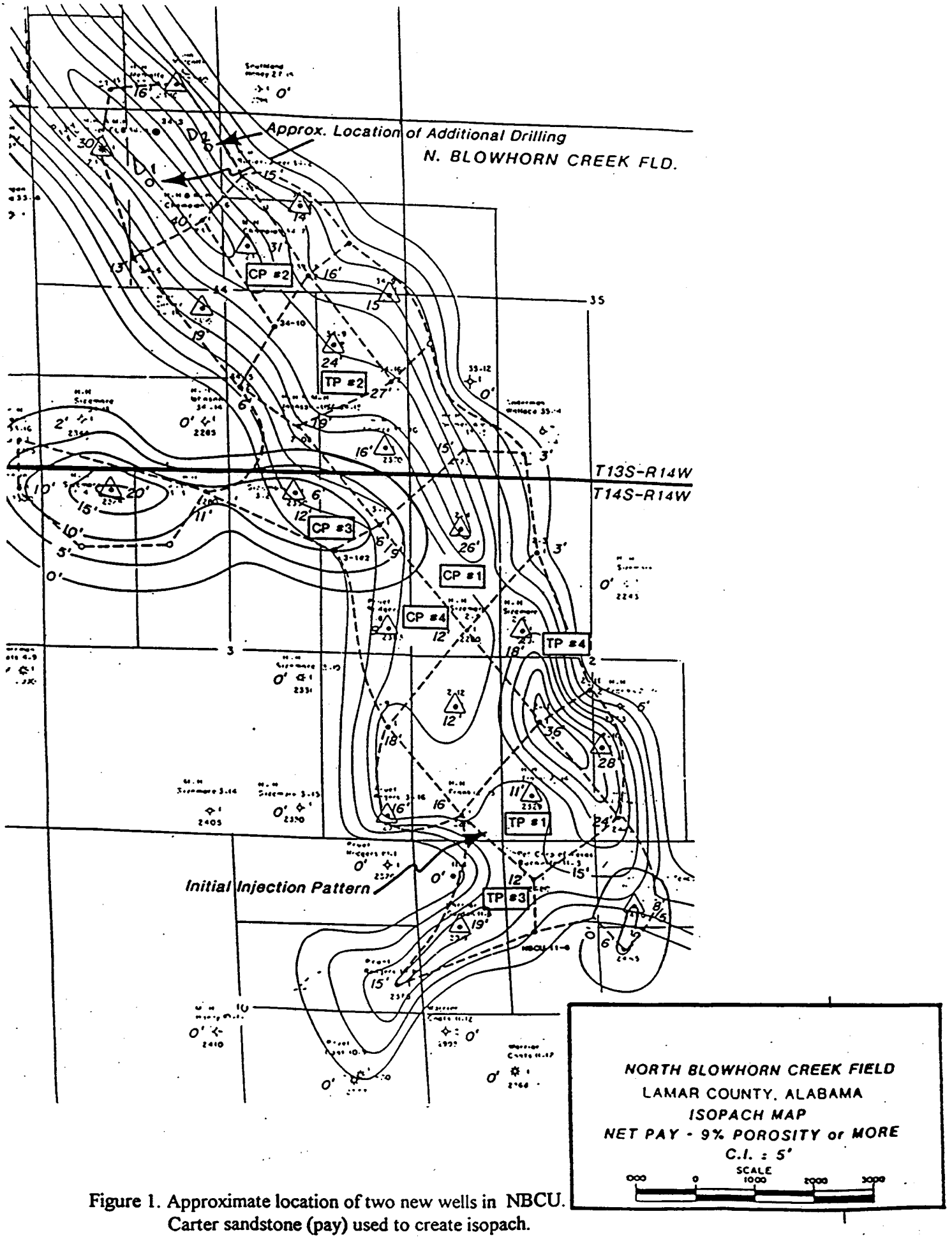

Figure 1. Approximate location of two new wells in NBCU. Carter sandstone (pay) used to create isopach.

reservoir was being swept by the existing waterflooding. The two well locations were chosen in an area of the field where representative Carter sand thicknesses (approximately 20') could be expected and also in an area where bypassed oil could reasonably be expected to exist. The first well, NBCU 34-6 #3, was drilled in March 1994 and encountered 20 net feet of Carter reservoir sand. However, probably due to the overbalanced drilling condition in the subnormally pressured reservoir, no increase in drilling rate occurred in the sand and the core point was missed.

The NBCU 34-6 #3 was completed and placed on rod pump. The well initially tested 25 BOPD, 1 BWPD and 5 MCFD. By December the rate had decreased to 10 BOPD, 0 BWPD and 4 MCFD. Cumulative production through December was 3354 BO, 241 BW and 1144 MCF. The fact that the well exhibits low water cut compared to offset wells which typically produce 65-98% water is further evidence of the existence of bypassed oil.

The second project well, NBCU 34-3 #2, was successfully drilled and cored during April. Because of the inability to pick a coring point by drilling rate increase in the first well, the core point was picked in the second well strictly based upon where in the geologic section the sand should occur, without the necessity of actually having an increase in drilling rate to indicate penetration of the sand. The well encountered about 21 feet of net Carter oil sand. About 20 feet of the core was immediately collected using the special procedures outlined elsewhere in this report and transported to MSU for laboratory work. From visual observation of live oil running from the core (see Figure 2) as it was removed from the core barrel, it was obvious that much oil remained in the reservoir. The well was completed and placed on rod pump for production. Representative production data could not be obtained initially due to the well pumping large amounts of fracturing sand and relatively little fluid. The well tubing and pump had to be pulled several times to clean out sand. In June, the decision was made to re-fracture the well to remedy the sand production problem and obtain representative, commercial production from the well. The refracturing was successful in achieving both objectives, and soon afterward the well tested 43 BOPD, 43 BWPD and about 80 MCFD on pump. By the end of December, the producing rate had declined to 8 BOPD, 11 BWPD and 4 MCFD. Cumulative production through December was 3128 BO, 4659 BW and 1073 MCF.

b. Acquisition of Cores From New Wells for Laboratory Studies.

The cores were received as they were pulled from the core barrels (live cores). The cores were broken into 1-ft sections, wiped off with 70% ethanol, and immediately placed under anaerobic containers in an anaerobic jar using Gas Pak® disposable hydrogen and carbon dioxide generator envelopes to produce anaeroobic conditions in the jar (Becton Dickerson Microbiology Systems). This procedure was completed within minutes, thus exposure to air was minimal. This treatment was of particular significance for these microbiological studies. It should be pointed out that the pressure in the core tends to force fluid and/or gases outward, thereby reducing further the possibility of exposing the internal section of the core to air. The anaerobic containers were packed in ice, transported to the laboratory, and placed in a refrigerator at 4C until needed.

Figure 2. Photograph showing core from newly drilled well (note oil running from core).

c. Core Analyses

(1). Methods

Crushing of the Cores

When the cores were to be analyzed, they were removed from their containers under an atmosphere of nitrogen, again wiped with 70% ethanol, and cut into four-inch sections using a core saw (Rayteck Industries, Stanford, Spring, CT). To reach the median of the core, one inch was cut from all sides of the cores using a sterile core saw blade. The median part of the core then was placed in a stainless steel core crusher under nitrogen gas and subjected to 20,000 psi using an hydraulic press. The crushed core was analyzed in a bacteriological hood containing a nitrogen atmosphere. The core was cut/divided into three parts, top, middle, and bottom. The crushed core was passed through a sterilized U.S.A. Standard Testing Sieve No. 40 (0.0165 inch openings) to make the inocula more uniform.

Enumeration of Microorganism

Fifty grams of sieved core material was mixed with 50 ml of sterile simulated production water described below that served as the diluent for this and subsequent dilutions. After mixing, 10 ml of the first dilution was transferred to a 90 ml dilution blank. The second dilution was mixed thoroughly before transferring 10 ml to the next dilution blank to make a 1/200 dilution of the original sample. The dilution vessel used was an eight ounces prescription bottle. Precautions were taken to insure homogeneity in the suspensions prior to sampling. Thus, particulate matter was present in all samples.

All work was conducted in a bacteriological hood containing a nitrogen atmosphere at a constant inlet pressure of 10 psi. The absence of oxygen in the atmosphere of the hood was confirmed by gas chromatographic (GC) analysis.

The conventional spread plate technique was employed in some enumeration procedures while the Most Probable Number (MPN) technique (3) was employed in others. All plate counts were performed in triplicate.

Simulated production water used for the waterflooding operation was prepared with the following inorganic salts per 50 liters of distilled water.

$CaCl_2$	10.90 g
$MgCl_2$	2.71 g
$BaCl_2$	4.56 g
Na_2SO_4	1.84 g
$NaHCO_3$	34.86 g
NaCl	147.90 g

The pH was adjusted to 7.0 (±0.2) using 10% (v/v) HCl.

The following groups of microorganisms were enumerated.

Total Heterotrophs were enumerated using the conventional spread plate technique and Bacto Plate Count Agar (PCA) obtained from Difco Laboratories, Detroit, MI. Plate counts were conducted under both aerobic and anaerobic conditions with the anaerobic cultures incubated in the BBL® GasPak®Systems containers. Plates were incubated at the temperature prevailing in the reservoir (90^0F).

Oil-utilizing Bacteria were enumerated using the conventional spread plate technique and oil-overlay agar. Incubation was as described for the total heterotrophs. Oil-overlay agar was prepared with simulated production water supplemented with 0.1% KNO_3, 0.37% $K_2HPO_4 \bullet 3H_2O$, and 1% filter-sterilized crude oil, and 2% Bacto-Agar. After the agar had been poured and had hardened in the petri plates, a thin overlay was added using oil agar prepared with oil-saturated water, but containing no added oil.

Two groups of Methane-producing Bacteria were enumerated, both using the Most Probable Number (MPN) technique (three tubes per concentration of sample). One set of tubes was supplemented with 10 g per liter of sodium formate, while the other set of tubes was incubated under an atmosphere of 80% H_2-20% CO_2 as described by Zeikus (2). Tubes were incubated at the temperature in the reservoir (90°F). All tubes were closed with serum stoppers and methane production determined by GC analysis of the atmosphere in the tube.

Dentrifying and Nitrate-reducing Bacteria were enumerated using the Most Probable Number (MPN) technique (three tubes per concentration of sample) and Bacto-Nitrate Broth in test tubes containing Durham fermentation tubes. The tubes were incubated for three weeks at the temperature in the reservoir (90°F). After incubation, tubes showing gas production were recorded as positive for denitrifying bacteria. Spot tests for nitrite were conducted using the sulfanilic acid and α-naphlylamine acetate reagents as described in Standard Methods for the Examination of Water and Wastewater (3). Negative tubes were reexamined after 60 days.

Nitrate-reducing, Hydrocarbon-utilizing Bacteria were enumerated using the Most Probable Number (MPN) technique (three tubes per concentration of sample) and the hydrocarbon-utilizing, nitrate-reducing medium. This medium was prepared as described by Rosenfeld (1) but modified by using simulated production water in place of synthetic seawater as follows:

$FeSO_4$	0.1 g
K_2HPO_4	0.5 g
KNO_3	1.0 g
Simulated production H_2O	25 ml
Distilled H_2O	965 ml

The reaction mixture was adjusted to pH 7.8 prior to the addition of 1% crude oil (v/v).

After three weeks of incubation at the temperature in the reservoir (90°F), the presence of nitrite in the tubes was determined as described above. Negative tubes were reexamined after 60 days.

Sulfate-reducing, Hydrocarbon-utilizing Bacteria were enumerated using the Most Probable Number (MPN) technique (three tubes per concentration of sample) and the hydrocarbon-utilizing, sulfate-reducing medium. This medium was prepared as described by Rosenfeld (1) but modified by using simulated production water in place of synthetic seawater as follows:

$Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$	0.1 g
K_2HPO_4	0.5 g
$(NH_4)_2SO_4$	1.0 g
Simulated production H_2O	25 ml
Distilled H_2O	925 ml
Crude Oil	10 ml

After three weeks of incubation at the temperature in the reservoir (90°), the tubes were examined for the blackening of the agar. Negative tubes were reexamined after 60 days.

Test for Ultramicrobacteria

Fifty grams of crushed core were vigorously mixed with 200 ml of sterile saline solution (2.5% NaCl, w/v). The suspension then was filtered through a Whatman No. 1 filter paper to remove fines which would interfere with the filtration process. The suspension was immediately filtered through a pre-sterilized membrane filter (0.80 μm). The filtrate then was filtered through a pre-sterilized 0.45 μm membrane filter to trap normal size bacteria. The filtrate was next filtered through a pre-sterilized membrane filter (0.22 μm). The filters were assembled on a sterile filtration apparatus (Millipore Corp.). One ml of the filtrate then was added aseptically to a test tube containing 10 ml of Tryptic Soy Broth (TSB) at a final concentration of one-eighth the strength of the original medium. Both the filters and filtrates were tested for the presence of viable microorganism weekly.

2. Results

Reservoir Characterization

The reservoir is located in North Blowhorn Creek, Beaverton, Lamar County, Alabama. The pay zone formation is in the Carter sandstone of the Black Warrior Basin. The reservoir size is about 1, 579 acres and the thickness of the pay zone varies from a few feet to 40′ with an average of 16′. The initial reservoir pressure and temperature are 1,174 psia and 90°F, respectively (see Figures 3 and 4).

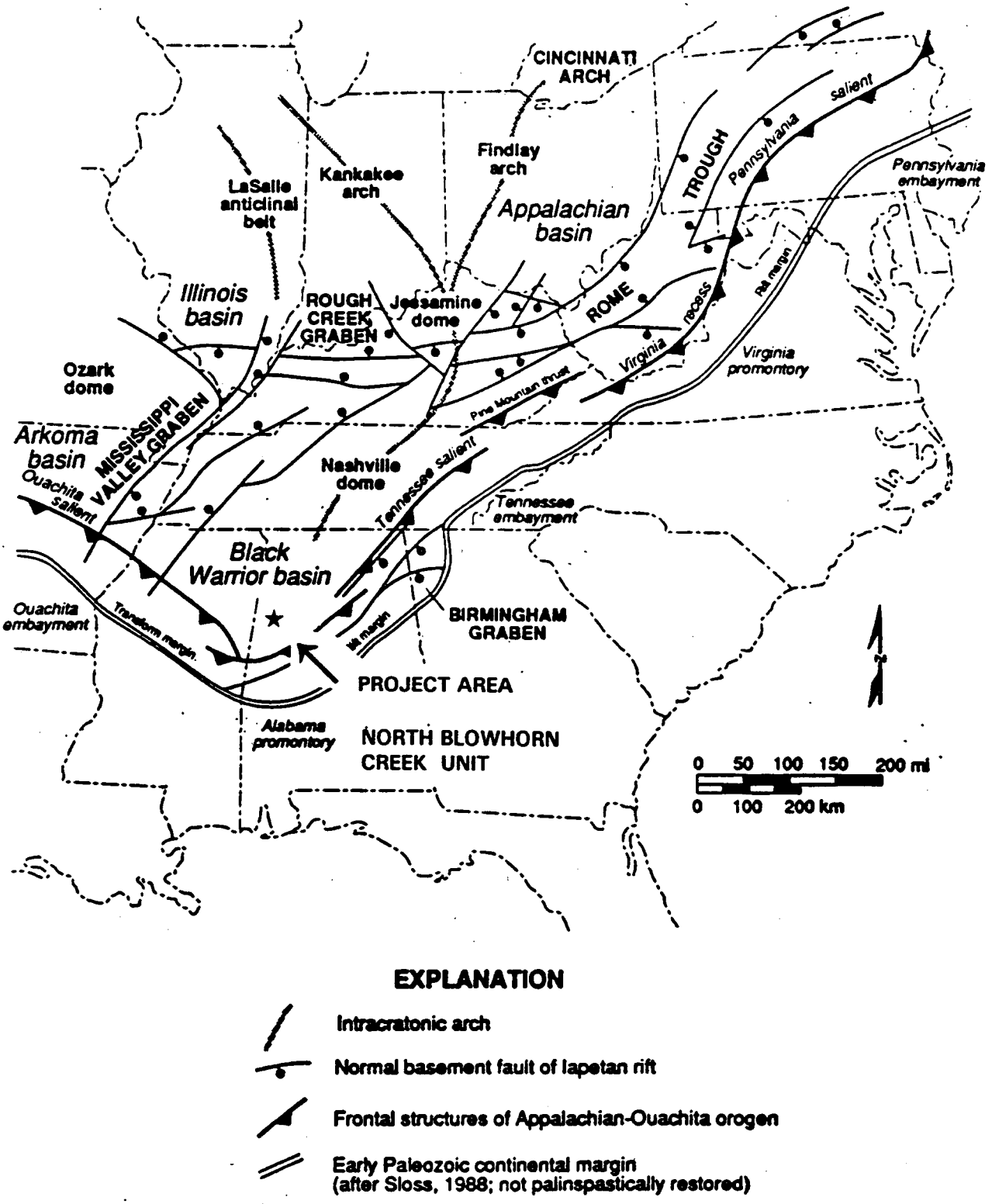

Figure 3 - Location and tectonic setting of the Black Warrior Basin of Alabama (modified from Thomas, 1988,1991)

ERATHEM	SYSTEM	SERIES		GEOLOGIC UNIT		LITHOLOGY
PALEOZOIC	PENNSYLVANIAN	LOWER	POTTSVILLE FORMATION	Coal bed gas	☼	Coal
				"Robertson sandstone"	☼	Sandstone
				"Nason sandstone"	☼	Sandstone
				"Fayette sandstone"	☼	Sandstone
				"Benton sandstone"	☼	Sandstone
				"Robinson sandstone"	☼	Sandstone
				"Chandler sandstone"	✸	Sandstone
	?—?—?	?—?—?	PARKWOOD FORMATION	"Coats sandstone"	✸	Sandstone
	MISSISSIPPIAN	UPPER		"Gilmer sandstone"	✸	Sandstone
				"Cooper sandstone"	☼	Sandstone
				"*Millerella* limestone"		Limestone
				"*Millerella* sandstone"	✸	Sandstone
				"Carter sandstone"	✸	Sandstone
				"Sanders sandstone"	✸	Sandstone
				"Abernethy sandstone"	✸	Sandstone
				"Ree sandstone"	✸	Sandstone
				BANGOR LIMESTONE	☼	Limestone
				HARTSELLE SANDSTONE	☼	Sandstone
			PRIDE MTN	"Evans sandstone"	✸	Sandstone
				"Lewis limestone"	☼	Limestone
				"Lewis sandstone"	✸	Sandstone
		MIDDLE		TUSCUMBIA LIMESTONE	☼	Limestone
		LOWER		FORT PAYNE CHERT		Chert and cherty limestone
	DEVONIAN			CHATTANOOGA SHALE		Shale
				unnamed cherty limestone	☼	Limestone
	SILURIAN			undifferentiated		Limestone
	ORDOVICIAN	UPPER & MIDDLE		undifferentiated		Limestone
		MIDDLE		STONES RIVER GROUP	☼	Limestone
		?—?—? LOWER		KNOX GROUP	☼	Dolomite and dolomitic limestone
	CAMBRIAN	UPPER				
		MIDDLE		KETONA DOLOMITE		Dolomite
				CONASAUGA FORMATION		Limestone
		LOWER		ROME FORMATION		Shale and siltstone

EXPLANATION

✸ Oil and Gas

☼ Gas

Oil reservoir unit discussed in this report.

Figure 4: Stratigraphic column showing oil reservoirs in the Black Warrior Basin of Alabama

Geological characterization of recovered core samples

General description of recovered cores.

Sandstone, grayish brown when dry and brownish gray when wet. The sandstone is a massive fine grained quartz with less than 10% heavy minerals; well sorted; subrounded to subangular; and moderately well cemented with silica cement.

The sandstone has occasional silty clay clasts, more numerous near the top. The clay clasts are medium light gray when dry and toward the bottom; dark gray when wet and near the top. The clasts are up to 1 inch in diameter.

There are horizontal and vertical fractures present. The fractures and occasional near horizontal silty clay laminae are dark stained (particularly noticeable when wet).

The dark staining in the laminae and fractures, as well as some zones within the sandstone, appear to be hydrocarbons. The core has a hydrocarbon odor and fluoresces purple from areas which correspond with dark stains.

Most silts and clay laminae as well as clasts are visible in the faces of horizontal breaks in the cores and only occasional clasts noticed elsewhere. Occasional dark stained laminae were noticed throughout.

A typical identification and description of core samples is as follows: Sandstone, buff, fine, grain, well sorted, siliceous, laminated, dolomite. X-ray diffraction analysis indicates the core is preliminary quartz (90%) with some 4% dolomite. The clay fraction consists of 2% kaolinite and 3% mixed layer clay. Some traces of siderite ($FeCO_3$) were present in some samples.

X-ray Diffraction Analysis

Quartz 90%
Feldspar .....
Calcite .....
Dolomite 4%
Kaolinite 2%
Ilite .....
Smectite ....
Mixed layer clay 3%
Siderite .....<1

Petrophysical characterization of recovered core samples

Core sample porosity varies from 7 to 19 percent. Core sample permeability varies much more drastically from 1 to 198 md.

Connate water saturation is around 17 percent and irreducible oil saturation is between 34-45 percent (see Figures 5 and 6 for typical relative permeability curves for oil-water and gas-oil experiments). The relative permeability curves suggest that the core samples were behaving in an oil-wet manner at the time of testing.

Microbiological

The core was divided into three portions, (top, middle, and bottom) crushed, and analyzed for total heterotrophs and oil-utilizing bacteria by the conventional spread plate technique (as shown in Table 1) and five physiological groups of microorganisms using the MPN technique (as shown in Table 2). It is interesting to note that there were no sulfate-reducers in any portion of the core. This was particularly surprising in that sulfate-reducing microorganisms have been reported in the literature as the most prevalent organism in oil reservoirs and, sulfate-reducing microorganisms had been recovered from fluids from other wells in this field. Thus, the results suggest that the area of the reservoir in which this well was drilled has not been impacted by the waterflooding operation. It is also interesting to note that in a previous study done in this laboratory there were no sulfate-reducers found in 13 oil well live cores.

No evidence of ultramicrobacteria (UMB) was found in any portion of the cores.

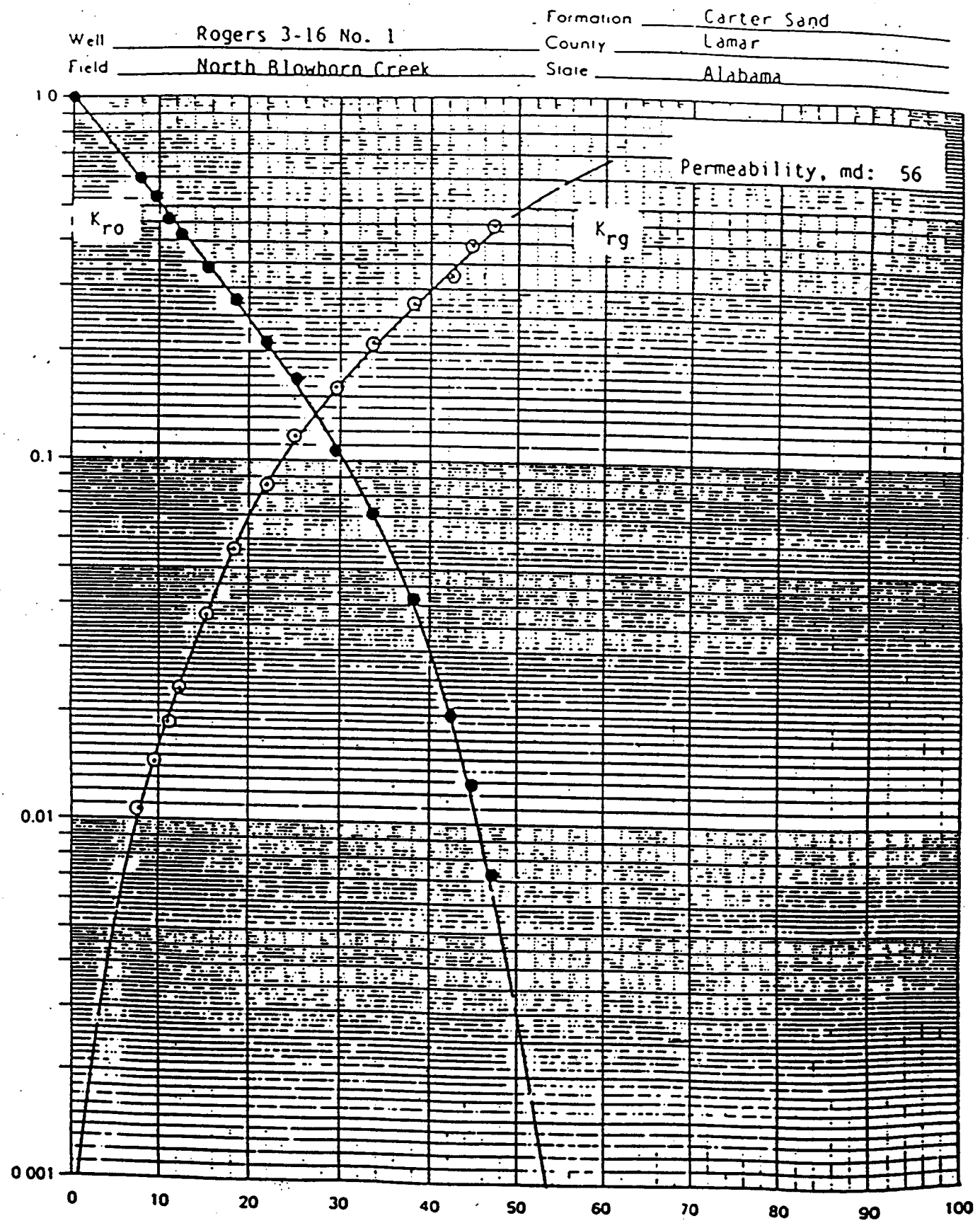

Figure 5. Gas saturation, percent pore space.

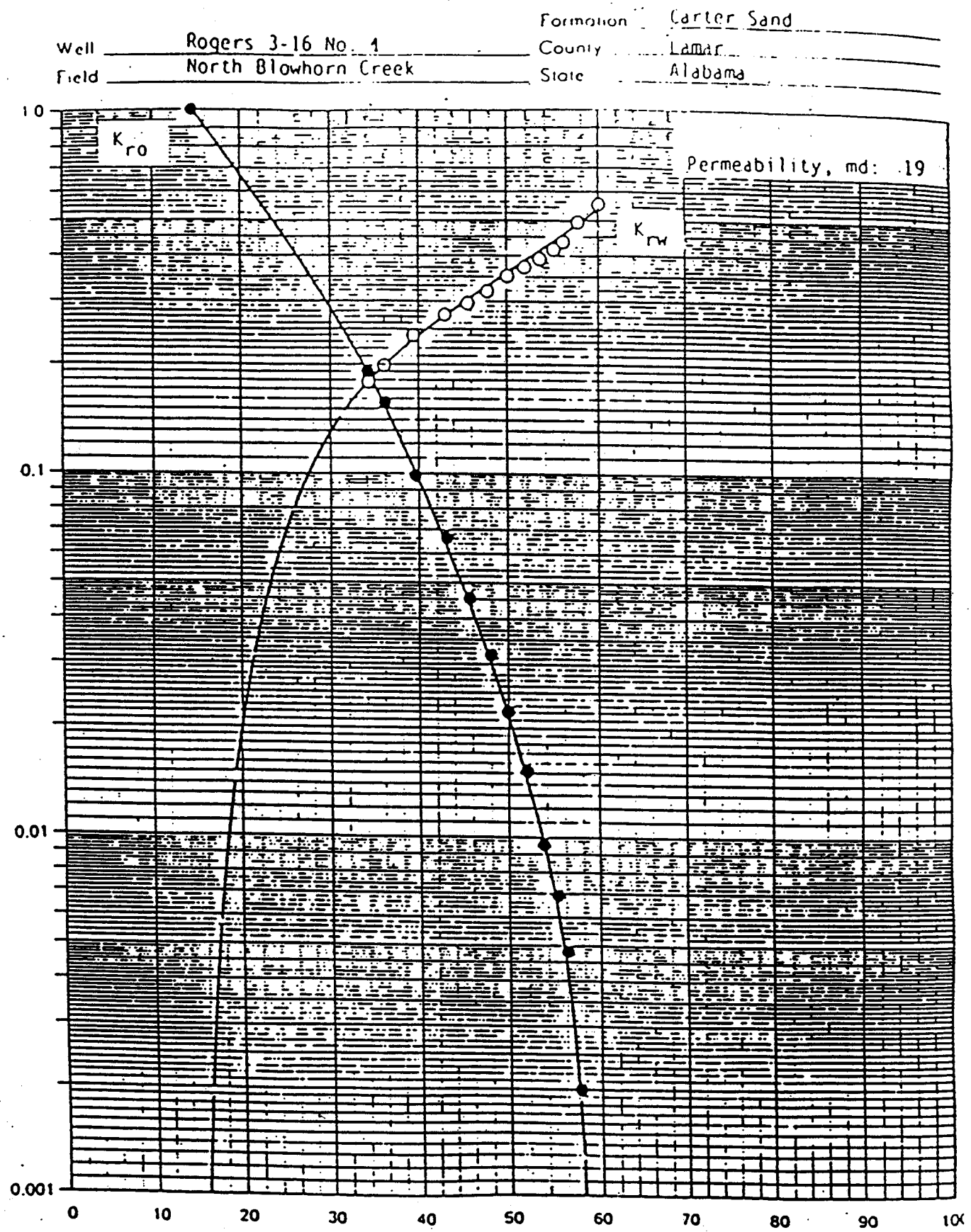

Figure 6. Water saturation, percent pore space.

Table 1. Aerobic and Anaerobic Heterotrophs and Oil-utilizing Microorganisms in Samples of Core from Newly Drilled Well.

	PCA Agar		Oil Agar	
Core	O_2	No O_2	O_2	No O_2
Top	1	0.3	<1.0	<1.0
Middle	43	2.0	193.0	<1.0
Bottom	36	2.0	0.3	<1.0

Numbers are # of bacteria/g of core.
PCA = Plate Count Agar

Table 2. Different Physiological Groups of Microoganisms in Core from Newly Drilled Well.

Core	Methanogen CO_2:H_2 Broth	Methanogen Formate Broth	HC-utilizing Broth	HC-utilizing NO_3-reducing Broth	Difco SO_4-reducing Broth	Nitrate
Top	4.6	<0.6	<0.6	<0.6	18.6	
Middle	4.6	48.0	<0.6	<0.6	>220.0	
Bottom	7.8	>220.0	<0.6	<0.6	18.6	

Numbers are # of bacteria/g of core.

d. Laboratory Waterflooding Test Using Live Cores

Cores were removed from the GasPak® containers under a nitrogen atmosphere and two adjacent core plugs were cut radially from each core -one to serve as the test core and one as the control core. The plugs were 3-4" long and 1½" in diameter. While still under a nitrogen atmosphere, each plug was inserted immediately into a special heat shrink plastic tube. The plastic wrap shrank as it cooled and wrapped tightly around the core. An entry and an exit port were placed on opposite ends of the core. These ports contained grooves for the reduction of end effects and for more homogeneous distribution of flowing fluids. The entire assembly then was inserted into a thick rubber sleeve (Viton Neoprene Sleeve, 1.5" diameter, with a ¼" wall). The ends of the entry and exit ports were fitted with rubber tubing and clamped shut. Both ends were completely sealed with high strength epoxy glue. The glue was allowed to harden for 24 hrs before the cores were used. Figure 7 is a diagrammatic sketch of the assembled core. Figures 8 and 9 are photographs of the cores.

(1). Treatment of the Cores

Initially, simulated production water contained in a five-liter carboy was allowed to flow through the core. The carboy was situated approximately 41" above the core and this hydrostatic head (corresponds to 1.92 psi) constituted the total pressure applied to the influent. The water was allowed to flow through a core for 48 hrs. After a 24 hr waiting period, experimentation commenced. Control cores received simulated production water only while the test cores received simulated production water containing added nutrients - nitrate as 0.06% potassium nitrate; orthophosphate as 0.04% disodium hydrogen phosphate.

(2). Analyses of Effluent from Cores

Fluid volume, pH, and microbial content were measured and recorded periodically for all cores and observations made as to the presence of oil in the effluent. Plate counts were conducted on selected samples using Plate Count Agar prepared using simulated production water. Plates were incubated for 72 hrs at 90°F under aerobic conditions or two weeks for anaerobic conditions.

Two sets of core plugs were prepared from two different sections of cores from this reservoir. As would be expected, the flow rates of simulated production water through the cores varied from core to core and ranged from 5.5 to 12.5 ml per day (Figures 10 and 11) for the control cores to the 10.5 to 148 ml per day (Figures 12 and 13) for the test cores. Surprisingly, the pH values for the effluents from both control and test cores were slightly alkaline and remained steady in the

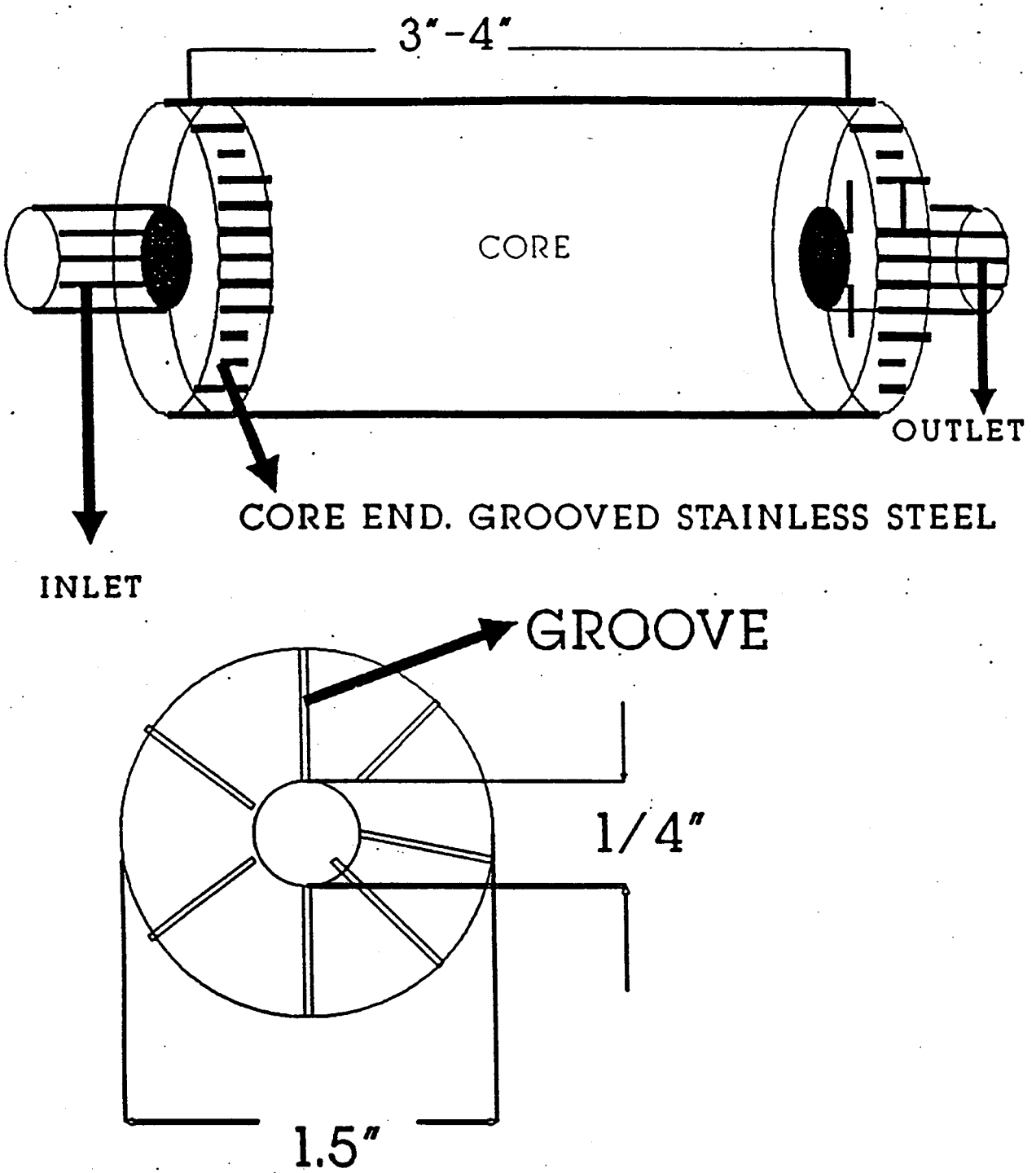

Figure 7. Live core assembly.

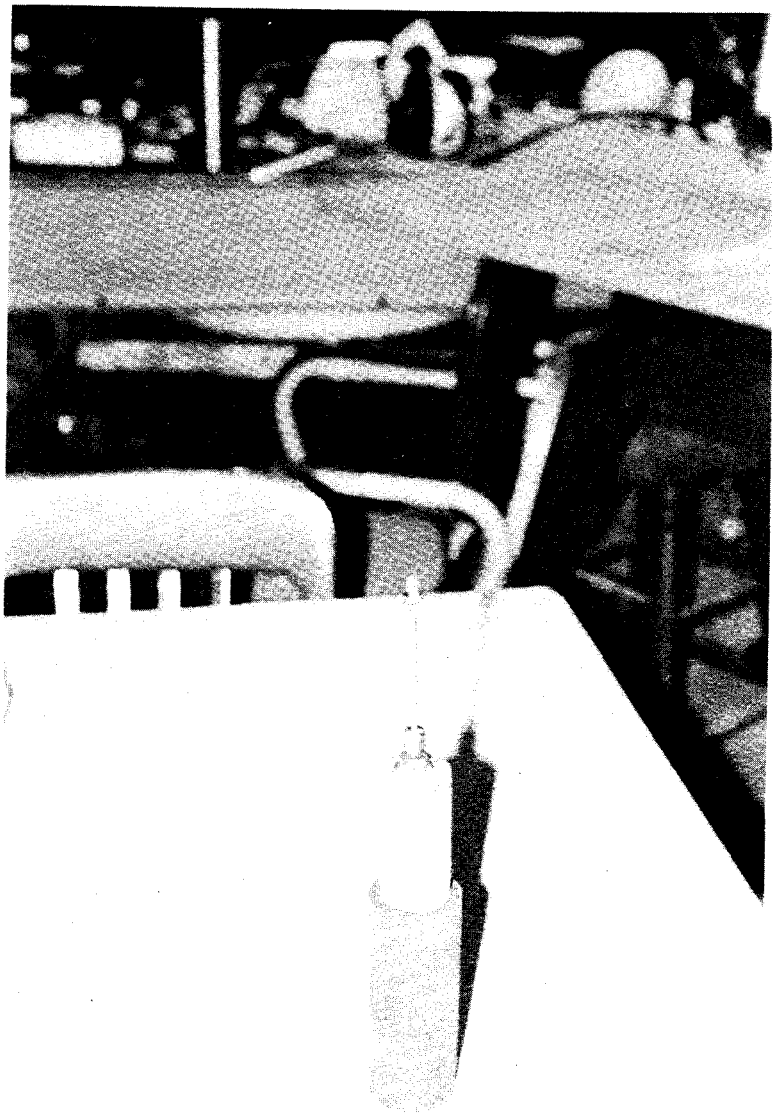

Figure 8. Photograph of core in plastic wrap next to the rubber sleeve.

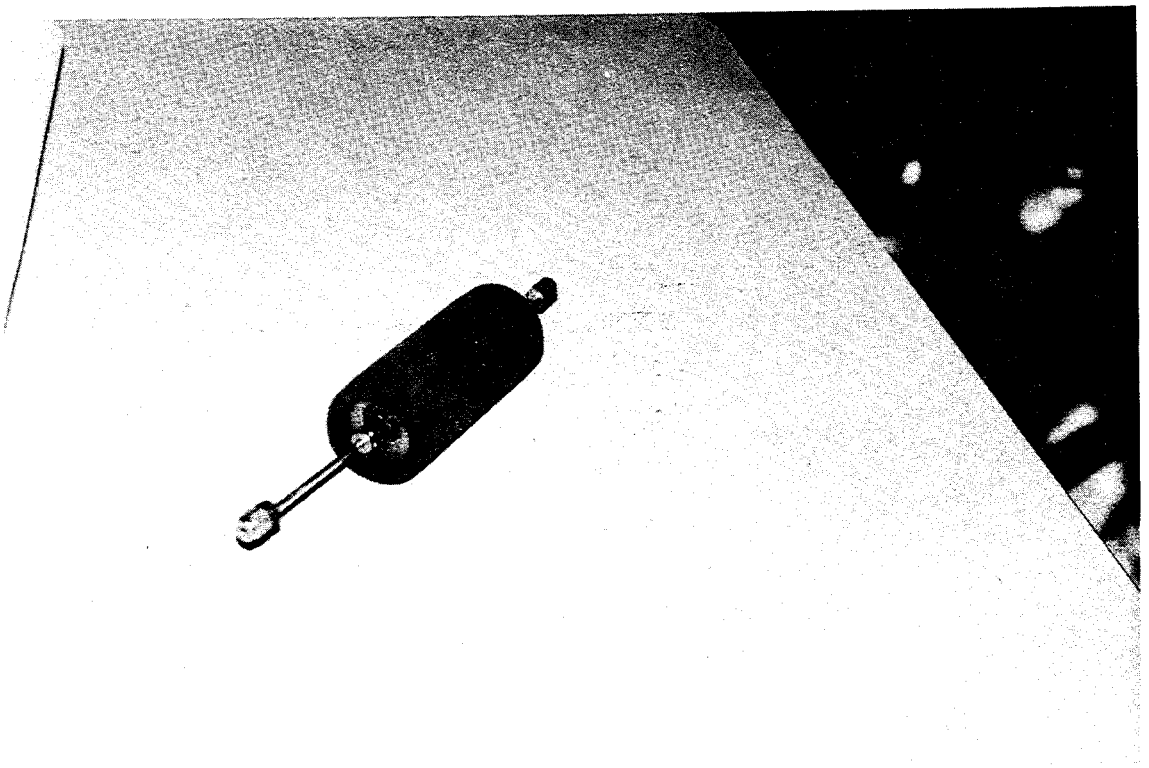

Figure 9. Photograph of completely assembled core plug.

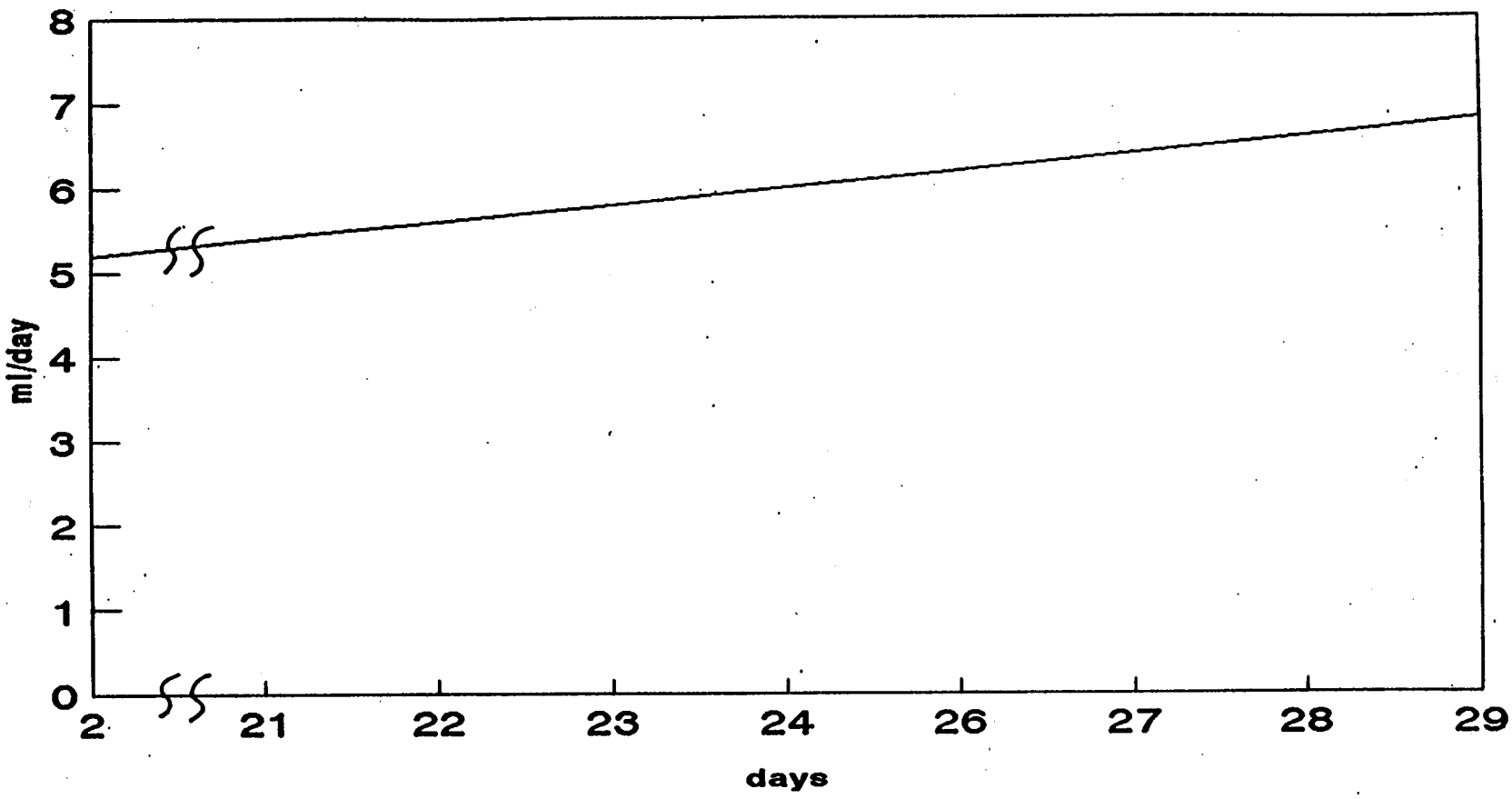

Figure 10. Flow rate of simulated production water through control core 1 in experiment 1.

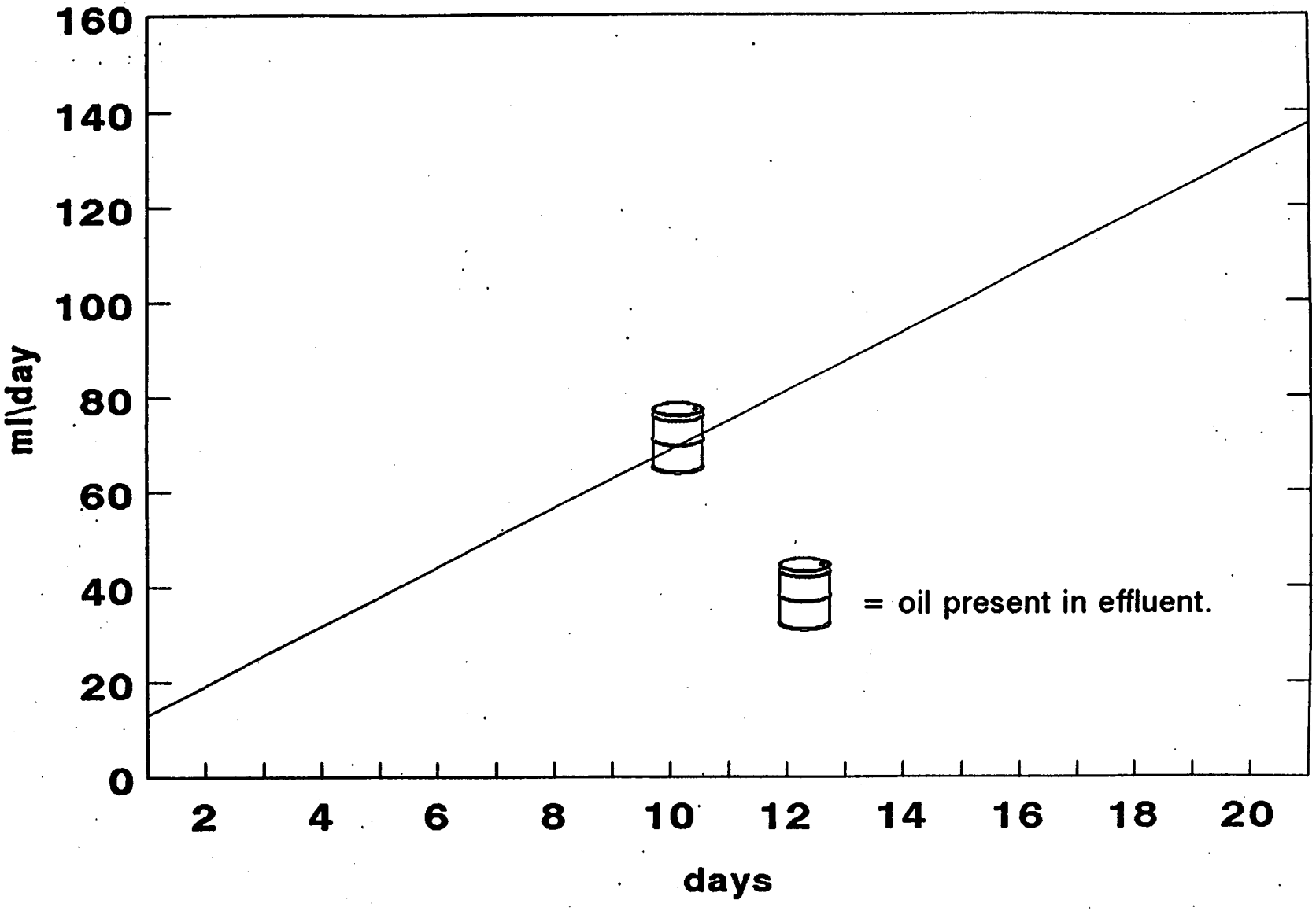

Figure 11. Flow rate of simulated production water though control core 2 in experiment 1.

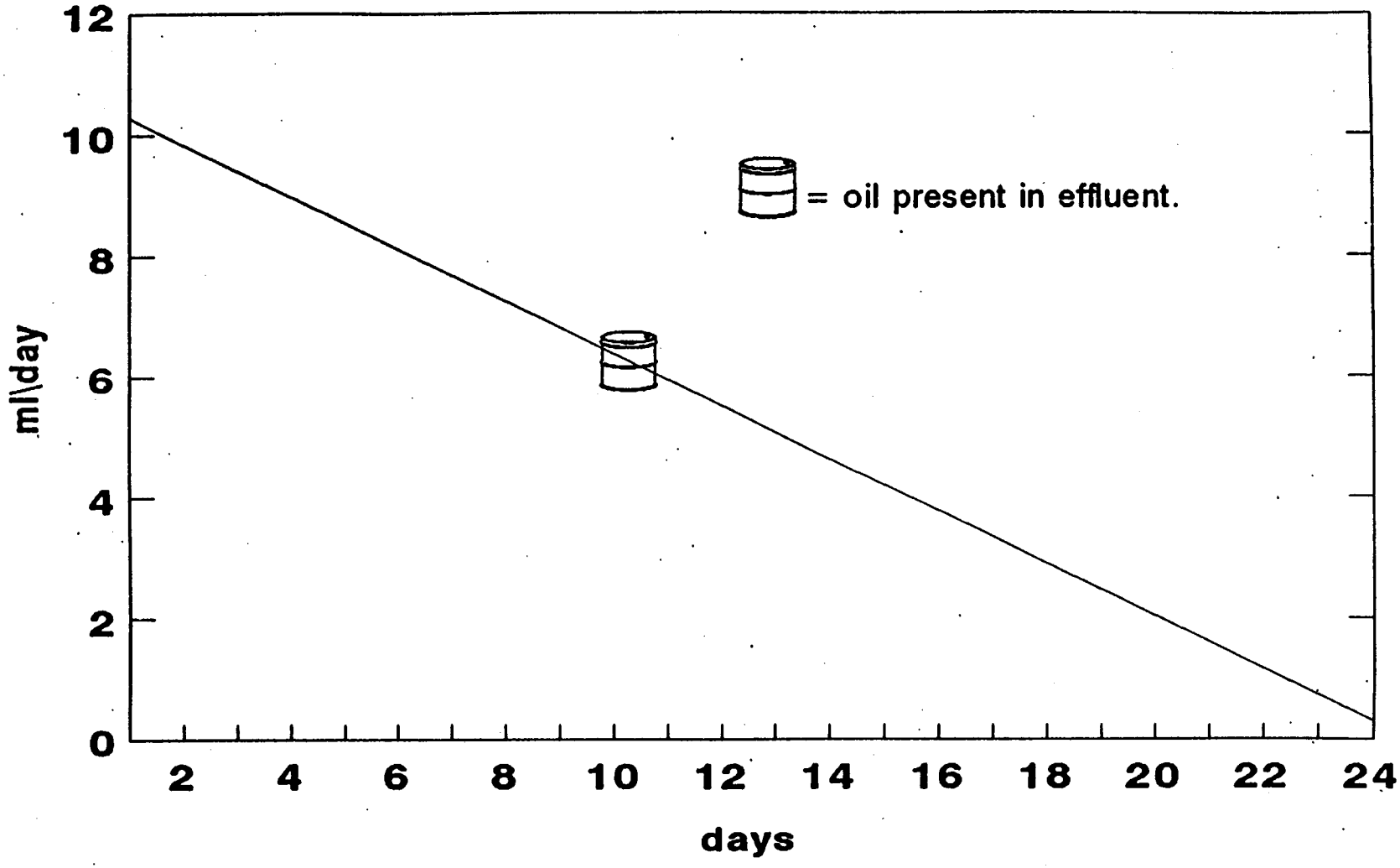

Figure 12. Flow rate of simulated production water containing potassium nitrate and disodium hydrogen phosphate through test core 1 in experiment 1.

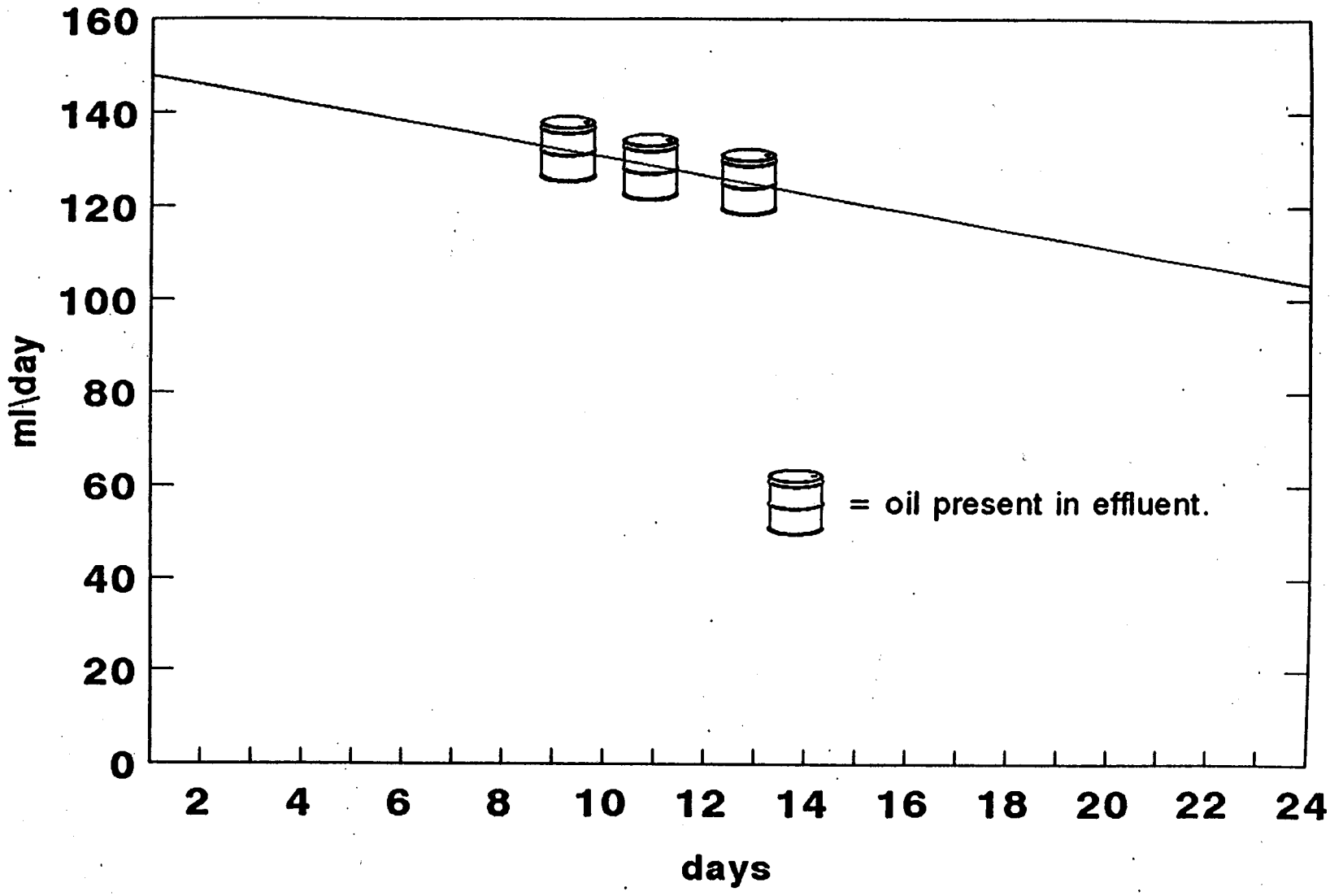

Figure 13. Flow rate of simulated production water containing potassium nitrate and disodium hydrogen phosphate through test core 2 in experiment 1.

range of 7.9 to 8.4. Similarly, the microbial content of the effluent remained essentially constant at about 10^1 organisms/ml for both the control and the test cores. Both control cores showed a steady increase in flow rate with time while both test cores decreased in flow rate with time. In fact, test core 2 ceased to flow twice and had to be restarted by gently increasing the pressure on the influent. Test core 1 ceased to flow and had to be restarted on six occasions. Oil was detected only one time in each of the control cores (day 1 in control 1 and day 10 in control core 2). Oil was detected in the effluent from test core 2 only on day 2 but was found in the effluent from test core 1 at days 9, 10, and 12.

The above core flood experiment was repeated using two new core plugs. Once again the pH of the effluent from both cores ranged from 7.4 to 8.4 and no increase in microbial content was observed. Oil in the effluent of the control core occurred only once (day 29) while in the test core it was found six times (days 11, 13, 14, 16, 19, and 25) as shown in Figures 14 and 15. The flow rate in the control core increased slightly while the flow rate in the test core decreased appreciably. Thus, the results of this second experiment confirmed the results of the first experiment in that the addition of potassium nitrate and disodium hydrogen phosphate to the injection water caused a decrease in the flow rate of the injection water though the core and caused an increase in oil in the effluent.

Therefore, it was concluded that the addition of nitrate and orthophosphate to the injection water employed in the injection wells in the North Blowhorn Creek Oil Field should result in the decrease in the diameter of the channels in the reservoir formation caused by the growth of the in -situ indigenous microflora. This decrease in flow through the main channels in the reservoir should divert injection water to the less permeable areas of the formation, thereby increasing the sweep efficiency of the waterflooding operation.

e. Baseline Data and Wells Involved in Field Tests

A data base for reservoir fluid production for all test and control wells in the first test pattern has been established. A sample of this data base is given in Table 3.

(1). Methods

Acquisition of Fluid Samples. Production, well, and injection water samples were obtained from the field. Production water was a mixture of all production water from separator tanks. Well water was water added to the production water to make injection water.

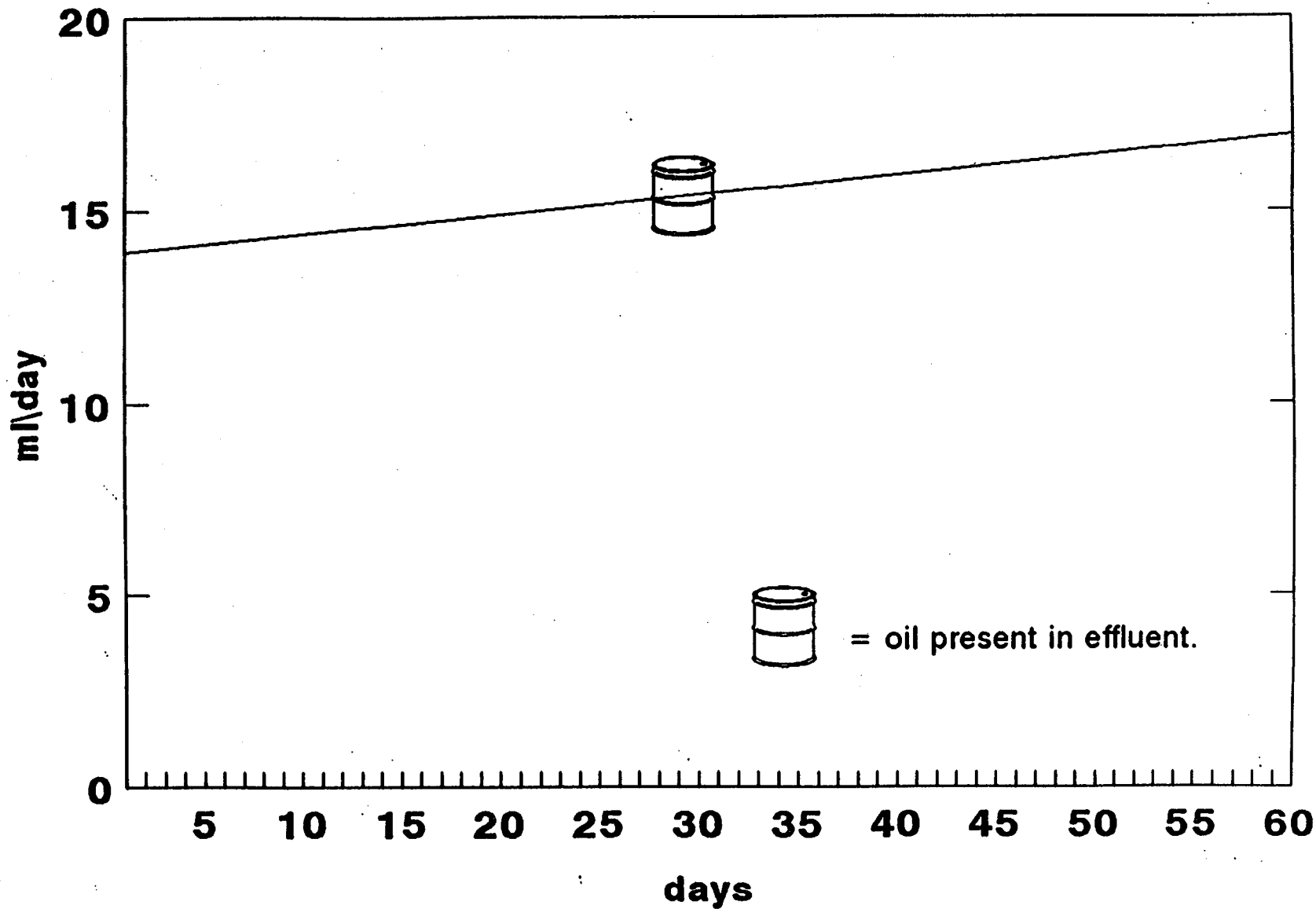

Figure 14. Flow rate of simulated production water through control core in experiment 2.

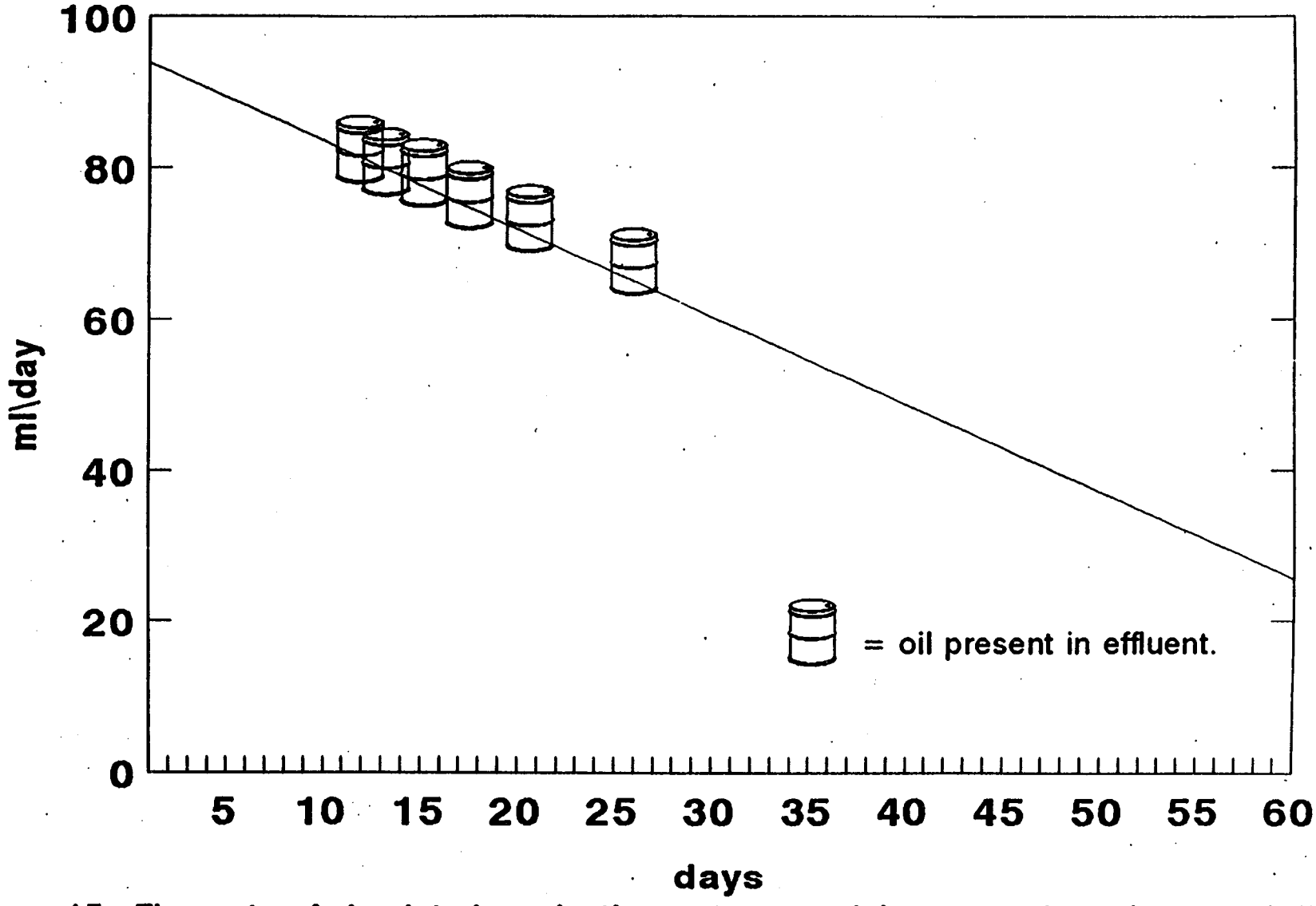

Figure 15. Flow rate of simulated production water containing potassium nitrate and disodium hydrogen phosphate through test core in experiment 2.

TABLE 3. History of Two Test and Control Producer Wells

WELL 34-15-2, PRODUCER				WELL 34-15-1, PRODUCER			
DATE, 1994	OIL	GAS	WATER	DATE, 1994	OIL	GAS	WATER
4-Feb	NO INFO			4-Feb	19	9	170
10-Feb	NO INFO			10-Feb	DOWN		
11-Feb	NO INFO			11-Feb	26	5	239
13-Feb	11	0	27	13-Feb	NO INFO		
17-Feb	NO INFO			17-Feb	20	9	179
19-Feb	10	0	21	19-Feb	NO INFO		
22-Feb	14	0	32	22-Feb	NO INFO		
24-Feb	NO INFO			24-Feb	25	5	225
25-Feb	4	0	11	25-Feb	NO INFO		
28-Feb	5	0	13	28-Feb	NO INFO		
3-Mar	NO INFO			3-Mar	27	10	150
5-Mar	8	0	20	5-Mar	NO INFO		
7-Mar	NO INFO			7-Mar	21	9	189
9-Mar	8	5	20	9-Mar	NO INFO		
15-Mar	4	0	9	15-Mar	NO INFO		

Fluid samples from all wells included in the first test and control pattern were collected in 1.5 gal plastic cans and returned to the laboratory for analyses.

Enumeration of Microorganisms

Ten milliliters of the water samples was mixed with 90 ml of the distilled water which served as the diluent to make a 1/10 dilution. This dilution was used alone with the conventional spread plate technique to enumerate total heterotrophs and oil-utilizing bacteria. The dilution vessel used was a six ounce prescription bottle. All plate counts were performed in triplicate.

All enumeration procedures were described in the section on Core Analyses above.

Chemical Methods

The following chemical tests were performed.

Orthophosphate test was determined using the Ascorbic Acid Reduction Method as given in the Standard Methods for the Examination of Water and Wastewater (3).

Nitrate-nitrogen was determined using the Cadmium Reduction Method as given in the Standard Methods for the Examination of Water and Wastewater (3).

Sulfate was determined using the Barium Chloride Method as given in the Standard Methods for the Examination of Wastewater (3).

Potassium was determined using the Tetraphenylboron Method as given in the Standard Methods for the Examination of Water and Wastewater (3).

Sulfide was determined using the Methylene Blue Method as given in the Standard Methods for the Examination of Water and Wastewater (3).

Chloride was determined using the Argentometric Method as given in the Standard Methods for the Examination of Water and Wastewater. (3).

Calcium carbonate was determined using the EDTA Titrimetric Method given in the Standard Methods for the Examination of Water and Wastewater (3).

Gas Chromatography Analysis.

Analysis for oil samples collected from test and control producing wells in pattern 1 were performed on a Varian 3300 single column gas chromatogram (GC). Column was J&W DB-1 30 x .53mm. Other GC operating setting area as follows.

Initial Col. Temp.....50°F
Initial Col Hold Time.....2:00
Program Final Col. Tem.....290
Program Col Rate in 0/min.....15
Program Col Hold Time.....500
Injector Temp.....290
Detector Temp.....300
FID B Initial Att.....1
FID B Initial Range.....10
Completion.....33

The GC is hooked into a desk top computer supported by Varian GC Star Chromatography 4. All peaks are relayed into the computer and each complete run is saved in entirety for future references.

The carrier gas, helium, was employed at a flow rate of 35 ml/min, air pressure 60 psig and hydrogen running pressure was 60 psig. Identification of paraffinic components was achieved by comparison of the retention time of peaks on the chromatogram to the retention time of standard samples. The area under the curves for different peaks are normalized. The standard sample used was ASTM Method D2287 Calibration Mixture.

The samples were prepared for GC analysis as follows. Using an Eppendorf pipette, 100 μl of oil was placed in a five ml beaker containing two ml of methylene chloride. The contents of the beaker were poured over sodium sulfate (to remove water if present) and the filtrate placed in two ml crimp-top vials with teflon septa.

(2). Results

Microorganisms

The baseline analyses began with the acquisition of water samples from the oil field. Microbial analyses were important to determine what, if any nitrate-reducing and sulfate-reducing bacteria are being injected into the wells. Three water samples (production, well, and injection water) were analyzed for the presence of heterotrophs: oil-utilizing; hydrocarbon-utilizing nitrate-reducing,; and hydrocarbon-utilizing, sulfate-reducing microorganisms.

As shown in Table 4 sulfate-reducers were only observed in production water and nitrate-reducers were observed in both production and injection water. Well water was negative for both nitrate-reducing and sulfate-reducing microorganisms. It is not surprising that sulfate-reducing microorganisms were absent in the injection water since the water is treated with Magnicide B (Baker Chemical) in the holding tank prior to injection into the reservoir.

A second set of water samples was analyzed for microbial content. As shown in Table 5, there seems to be a decrease in the number of anaerobic heterotrophs when compared to the data obtained from the first set of samples. Nitrate-reducers were only observed in injection water, whereas in the last set of samples nitrate-reducers were observed in both production water and injection water. Sulfate-reducers were observed in both production and injection water but not in well water.

Fluid Samples

There are four test pattern producer wells, four control pattern producer wells, and two injector wells (1 test and 1 control) in the first test pattern. A complete microbial analysis of these water samples was performed on the initial water samples from these wells as shown in Table 6 and Table 7. These data will serve as a baseline for further studies. It should be noted that sulfate-reducers were observed in all of the wells except the two newly drilled wells (Table 7). Microbial analyses for oil-utilizing bacteria and total hetertrophs were performed on a second set of well water samples received from wells in the first test and control pattern as shown in Table 8.

As seen in Table 9 and 10, there were no detectable amounts of nitrate or phosphate in any of the first two sets of samples.

Gas chromatography of oil samples collected from test and control producing wells indicate a paraffinic oil (See Figure 16). The oil gravity is about 33° API and has a 2.3 cp viscosity at the reservoir temperature. Initial formation volume factor and bubble point pressure are reported to be 1.139 RBBL/BBL and 1200 psia, respectively.

Table 4. Microbial Content of First Set of Water Samples.

Water Sample	Hetertrophs PCA*		Oil-degrading Oil Agar*		HC-utilizing NO_3-reducing	HC-utilizing SO_4-reducing
	O_2	NO O_2	O_2	No O_2		
Production	6	7	0.7	1	+	+
Injection	43	83	6.0	3	+	-
Well	3	7	0.7	<1	-	-

*Numbers are # of bacteria/g of core.
+ = Growth
- = No growth
PCA = Plate Count Agar

Table 5. Microbial Content of Second Set of Water Samples.

Water Samples	Hetertroph PCA		Oil-degrading Oil agar		HC-utilizing NO_3-reducing	HC-utilizing SO_4-reducing
	O_2	No O_2	O_2	No O_2		
Production	6	4	3	<1.0	-	+
Injection	34	<1	2	<1.0	+	+
Well	8	<1	13	0.3	_	-

Numbers are # of bacteria/g of core.
+ = Growth
- = No growth
PCA = Plate Count Agar

Table 6. Number of Aerobic and Anaerobic Heterotrophs and Oil-utilizing Microorganisms in the First Set of Water Samples Taken from Wells in the First Test and Control Pattern.

NBCU	PCA		Oil Agar	
Well #	O_2	No O_2	O_2	No O_2
2-13-1 (P)	4	<1	9	<1
2-15-1 (P)	100	55	217	55
2-11-1 (P)	<1	<1	<1	<1
11-3-1 (P)	<1	2	42	7
2-14-1 (PI)	<1	3	<1	<1
35-13 (C)	23	<1	10	<1
35-14 (C)	100	57	54	60
3-1-1 (C)	47	55	136	115
2-3 (C)	<1	<1	3	<1
2-5 (C)	4	1	11	1
2-4 (C)	<1	1	1	1

Numbers are # of bacterial/ml of water.
P = Test pattern
PI = Test injector
C = Control pattern
CI = Control injector
PCA = Plate Count Agar

Table 7. Different Physiological Groups of Microorganisms in Water as Determined Using the Multiple Tube Fermentation Technique in the First Set of Water Samples Taken from Wells in the First Test and Control Pattern.

Well #	Methanogen CO_2H_2 Broth	Medium Formate Broth	HC-utilizing Broth	HC-utilizing NO_3-reducing Broth	SO_4-reducing Broth	Nitrate
2-13-1 (P)	0.046	<0.003	0.093	0.024	0.093	
2-15-1 (P)	0.021	0.023	<0.003	<0.003	0.023	
2-11-1 (P)	0.043	<0.003	<0.003	11.000	11.000	
11-3-1 (P)	1.10	<0.003	<0.003	<0.003	<0.003	
2-14-1 (I)	0.021	<0.003	<0.003	0.043	11.000	
35-13 (C)	0.046	<0.003	<0.003	0.090	<0.003	
35-14 (C)	0.075	<0.003	<0.003	0.023	0.075	
3-1-1 (C)	0.015	<0.003	<0.003	0.075	11.000	
2-3 (C)	0.075	<0.003	<0.003	11.000	11.000	
2-5 (C)	0.028	<0.003	0.040	11.000	11.000	

Numbers are # of bacteria/ml of water.
P = Test pattern
PI = Test injector
C = Control pattern
CI = Control injector

Table 8. Number of Aerobic and Anaerobic Heterotrophs and Oil-utilizing Microorganisms in the Second Set of Water Samples Taken from Wells in the First Test and Control Pattern.

NBCU Well #	PCA		Oil Agar	
	O_2	No O_2	O_2	No O_2
2-13-1 (P)	85	73	108	164
2-15-1 (P)	11	43	3	67
2-11-1 (P)	7	10	1	16
11-3-1 (P)	>300,000	>300,000	>300,000	>300,000
2-14-1 (PI)	1	0	<1	3
35-13 (C)	36	34	53	16
35-14 (C)	47	38	12	11
3-1-1 (C)	16	16	5	39
2-3 (C)	6	11	10	5
2-5 (C)	21	15	12	18
2-4 (CI)	13	79	10	113

Numbers are # of bacteria/ml of water
P = Test pattern
PI = Test injector
C = Control pattern
CI = Control injector

Table 9. Inorganic Constituents in First Set of Water Samples Taken from Wells in First Test and Control Pattern.

Well #	$SO_4^{=}$	$CaCO_3$		Cl^-	K^+	$S^{=}$	$PO^{=}$	NO_3-N
2-13-1 (P)	350	17	1150	10.0	*	-	-	
2-15-1 (P)	58	143	2800	4.1	*	-	-	
2-11-1 (P)	30	200	2500	8.0	*	-	-	
11-3-1 (P)	26	84	2800	2.6	*	-	-	
2-14-1 (PI)	22	180	2700	0.7	*	-	-	
35-13 (C)	32	103	2700	2.2	*	-	-	
35-14 (C)	100	109	5000	3.3	*	-	-	
3-1-1 (C)	32	200	2700	2.3	*	-	-	
2-3 (C)	57	196	2400	3.5	*	-	-	
2-5 (C)	98	116	2700	3.0	*	-	-	

Numbers are mg/ml except $S^{=}$ which is μg/ml.
P = Test pattern
PI = Test injector
C = Control pattern
- = < 0.01 mg/ml
* = <0.01 μg/ml

Table 10. Inorganic Constituents in Second Set of Water Samples Taken from Wells in First Test and Control Pattern.

Well #	$SO_4^{=}$	$CACO_3$	Cl^-	K^+	$S^{=}$	$PO_4^{=}$	NO_3-N
2-13-1 (P)	55	ND	ND	6.70	*	-	-
2-15-1 (P)	31	ND	ND	4.75	*	-	-
2-11-1 (P)	33	ND	ND	9.90	*	-	-
11-3-1 (P)	22	ND	ND	2.75	*	-	-
2-14-1 (PI)	53	ND	ND	5.40	*	-	-
35-13 (C)	9	ND	ND	3.45	*	-	-
35-14 (C)	26	ND	ND	2.30	*	-	-
3-1-1 (C)	29	ND	ND	5.40	*	-	-
2-3 (C)	100	ND	ND	2.75	*	-	-
2-5 (C)	18	ND	ND	5.40	*	-	-
24 (CI)	23	ND	ND	4.75	*	-	-

Numbers are mg/ml except $S^{=}$ which is μg/ml.
P = Test pattern
PI = Test injector
C = Control pattern
CI = Control injector
- = <0.01 mg/ml
* = <0.01 μg/ml
ND = Not Determined

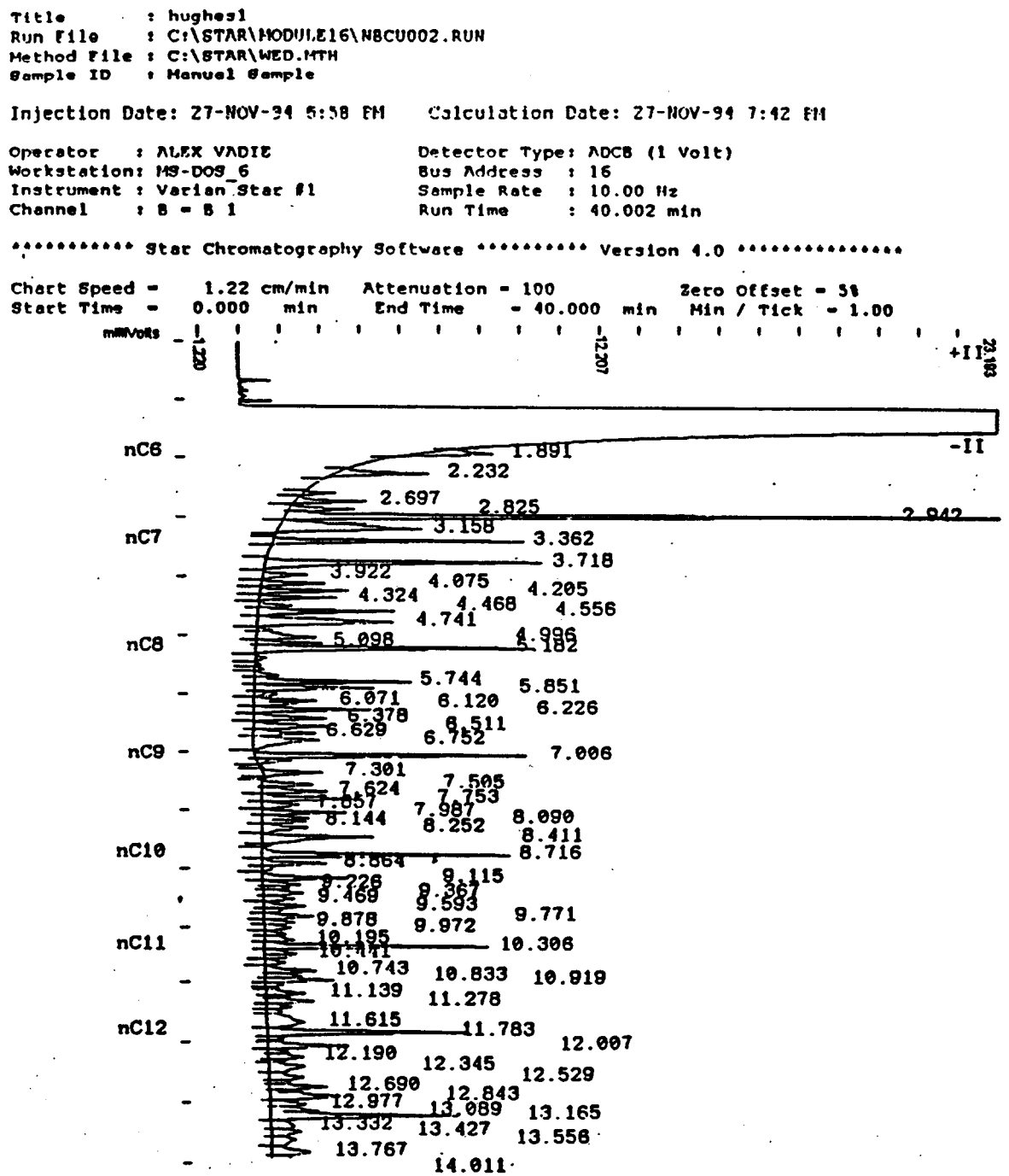

Figure 16. Typical gas chromatogram of a sample of oil from the North Blowhorn Creek Field.

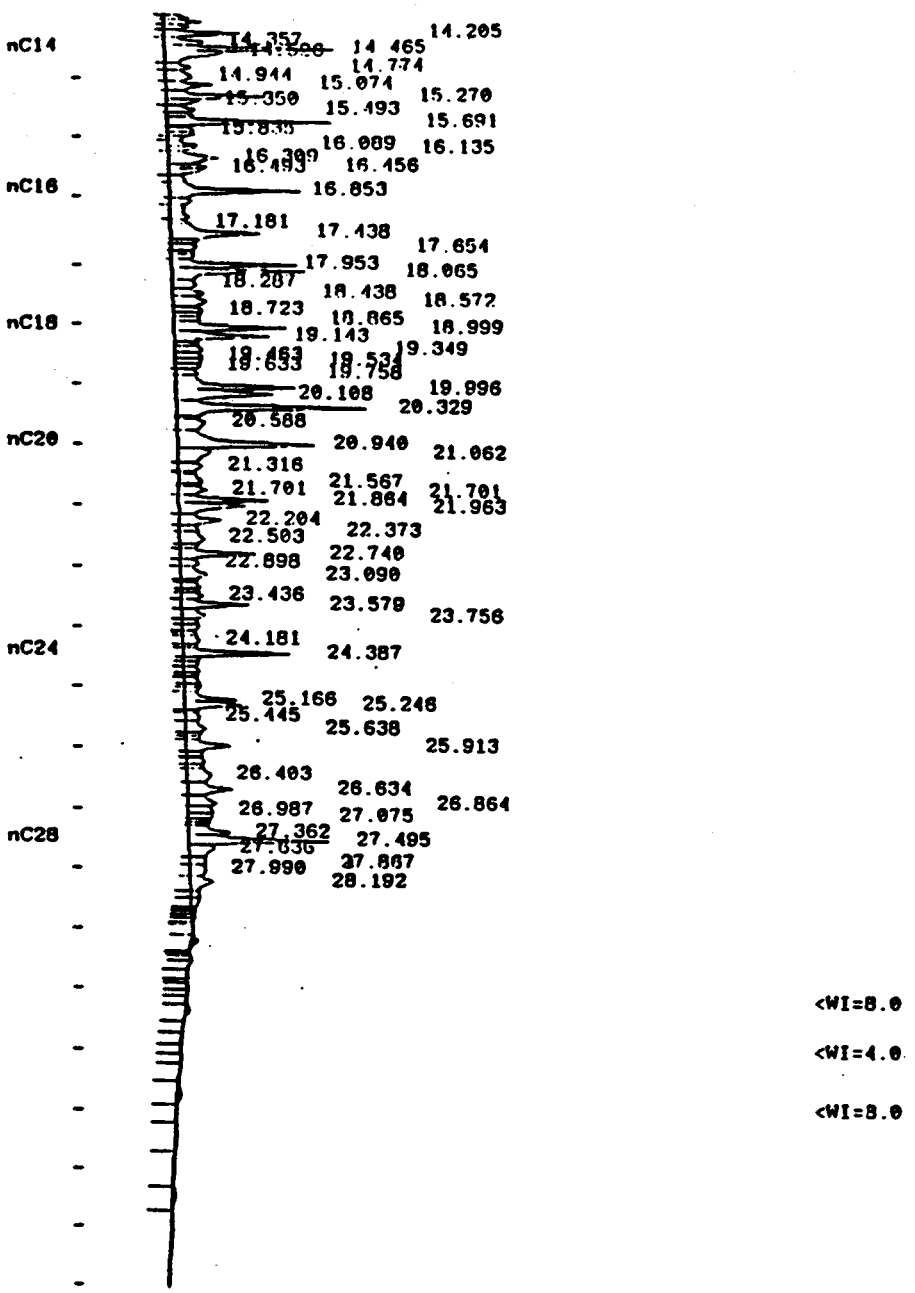

Figure 16. Continued.

f. Tracer Tests

Early in the planning stages for the project the first test well pattern was chosen because it had been observed in the field that the NBCU 2-14 #1 injection well seemed to be very well connected to the 2-13 #1 hydraulically. The 2-14 #1 allowed injection of relatively high rates, and the 2-13 #1 produced high fluid rates and was overpressured. It was theorized that the movement of water from the injector to producer would be quicker than in any other injection pattern. However, the need to have a quantitative time for fluid travel from injector to the producers in the first pattern led to the decision to run a radioactive tracer survey. If the travel time could be established, then some time frame for the effects of microbial activity to become detectable could be established.

On April 27, 1994, 2 Ci of tritiated water was injected into the 2-14 #1 well. Weekly and then monthly sampling of water from the four pattern producing wells was done. No trace of the material was detected by the laboratory analysis of the water samples until October 12 when 14 pCi/ml was detected in the 2-13 #1 well. On November 9, the same well sample tested 41 pCi/ml and 1.9pCi/ml also was found in the 2-15 #1 well. Sampling of the water is continuing, but preliminary results indicate that six to seven months are required for injection water to travel to the producers. Thus, evidence of microbial activity could not be expected to be detectable in less than seven months and probably closer to a year after initiation of nutrient injection.

3. PHASE II IMPLEMENTATION

a. Design of Field Demonstration

(1). Test patterns for field demonstration

Figure 17 is an isopach of the North Blowhorn Creek Oil Field showing the locations of the wells in the four test and four control patterns. The wells included in the patterns are as follows.

TP #1
Injection-Production Pattern:

Injection Well:	NBCU 2-14 #1
Production Wells:	NBCU 2-11 #1*
	NBCU 2-15 #1
	NBCU 11-3 #1*
	NBCU 2-13 #1*

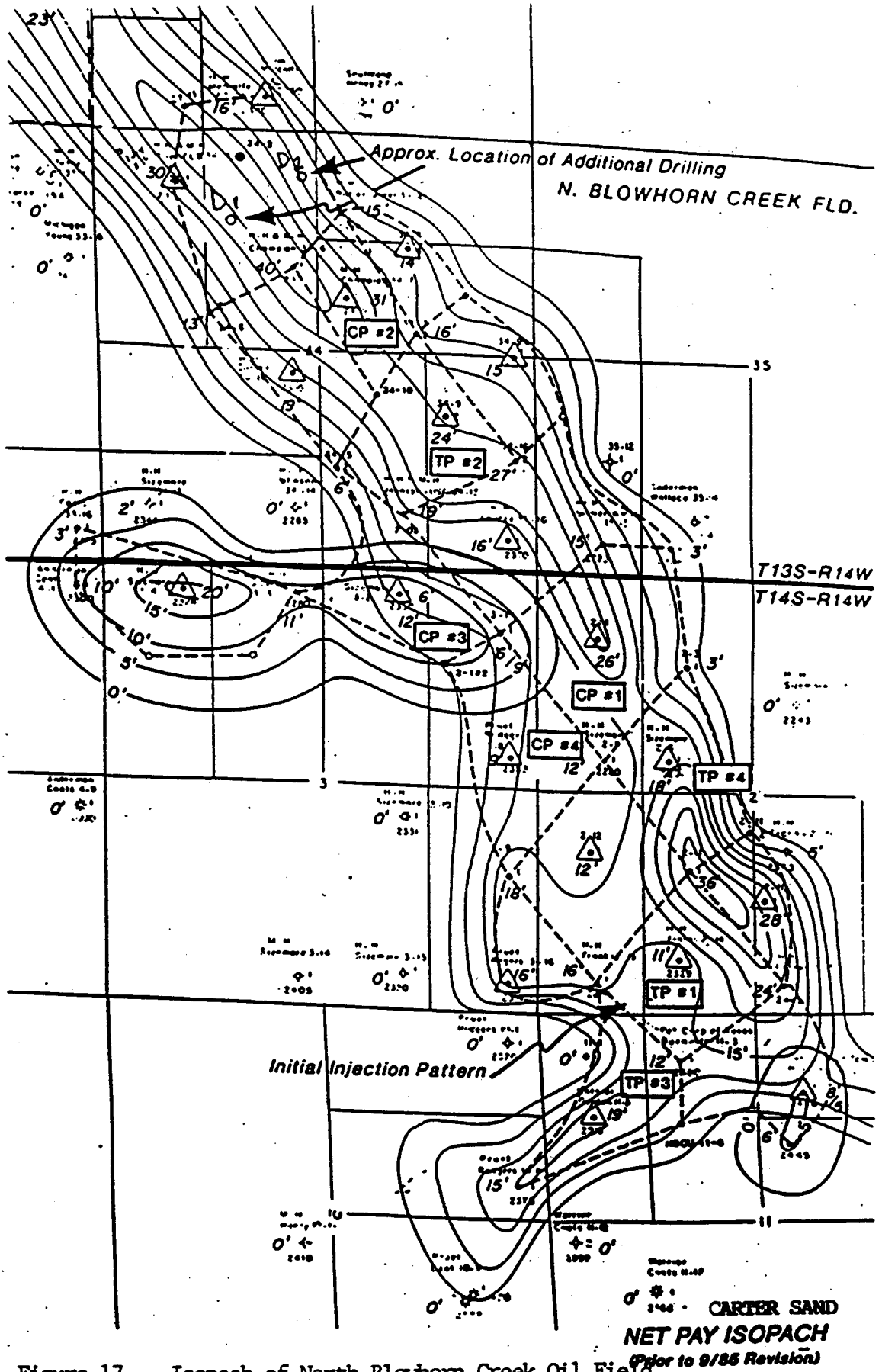

Figure 17. Isopach of North Blowhorn Creek Oil Field showing locations of wells in the four test and four control patterns.

CP #1 (Control Set)

Injection Well:	NBCU 2-4 #1
Production Wells:	NBCU 35-13 #1
	NBCU 35-14 #1
	NBCU 2-3 #1*
	NBCU 2-5 #1*
	NBCU 3-1 #1*

TP #2

Injection-Production Pattern:

Injection Well:	NBCU 34-9 #2
Production Wells:	NBCU 34-7 #2*
	NBCU 34-16 #2
	NBCU 34-15 #1*
	NBCU 34-15 #2*
	NBCU 34-10 #1*

CP #2 (Control Set)

Injection Well:	NBCU 34-7 #1
Production Wells:	NBCU 34-2 #1
	NBCU 34-6 #1
	NBCU 34-7 #2*
	NBCU 34-10 #1*

TP #3

Injection-Production Pattern:

Injection Well:	NBCU 11-5 #1
Production Wells:	NBCU 10-8 #1
	NBCU 11-6 #1
	NBCU 11-4 #!
	NBCU 11-3 #1*
	NBCU 2-13 #1*

CP #3 (Control Set)

Injection Well:	NBCU 3-2 #1
Production Wells:	NBCU 3-3 #1
	NBCU 3-1 #1*
	NBCU 3-1 #2*
	NBCU 34-15 #1*
	NBCU 34-15 #2*

TP #4

Injection-Production Pattern:

Injection Well:	NBCU 2-6 #1
Production Wells:	NBCU 2-11 #2
	NBCU 2-3 #1*
	NBCU 2-5 #1*
	NBCU 2-11 #1*

CP #4 (Control Set)

Injection Well:	NBCU 3-8 #1
Productioin Wells:	NBCU 3-1 #1*
	NBCCU 3-1 #2*
	NBCU 3-9 #1
	NBCU 2-5 #1*

*Indicates wells included in more than 1 injection or control pattern.

(2). Feeding regime

The first test injector well (NBCU 2-14#1) is injecting 480-500 barrels of water per day. Based on this rate of injection and the results obtained from the core flood experiments, it was decided to employ the addition of potassium nitrate at a concentration of 0.12% (w/v) and disodium hydrogen phosphate at a concentration of 0.06% (w/v). The nutrients are mixed in much higher concentrations on the skids (described below) and injected at such rates that the entire amount of injection water during a 24-hour period will contain the above designated concentrations. In order to neutralize the effect of an increased pH of the injection water due to the phosphate addition, two gallons of 10% HCl(v/v) were added to each tank of phosphate solution. When the current supply of disodium hydrogen phosphate is used, the phosphate solution will be prepared using monosodium dihydrogen phosphate, thus obviating the need for adding the 10%.HCl.

The following injection schedule has been formulated based upon a waterflood injection rate of 480-500 BWPD in injector well NBCU 2-14#1.

Monday - Mix 200 lbs (2 bags) of potassium nitrate with 200 gals of water and pump entire volume in as close to 24 hrs as possible.

Tuesday - No chemical to be pumped, but wash out tank and pump down the hole during morning.

Wednesday - Mix 100 lbs (1 bag) of disodium hydrogen phosphate with 200 gals of water and pump in as close to 24 hrs as possible.

Thursday - No chemical to be pumped, but wash out tank and pump down the hole during morning.

Friday - Mix 100 lbs (1 bag) of disodium hydrogen phosphate with 200 gals of water and pump in as close to 24 hrs as possible.

Saturday - No chemical to be pumped, but wash out tank and pump down the hole during morning.

Sunday - No chemical to be pumped.

Above schedule is repeated each week.

b. Design of Skids

After completion of the laboratory nutrient injection testing, the results were scaled up to field operating volumes which became the design basis for the nutrient skids (see Figure 18). It was determined that each injection skid needed the ability to mix and pump 100-300 gallons of water containing 50-400 lbs of nitrate and phosphate chemicals per day at a pressure of 1200 psi. The ability to vary the pump rate over a wide range was required as well as the ability to maintain a precisely metered rate. The nutrients were packaged as a dry crystal in 50 and 100 lb bags, so the ability to mix the chemicals and know that all went into solution was required. The skid had to be designed for simple maintenance and operation by the field lease pumpers. A small storage area to keep unused chemicals dry also was required.

Based upon these requirements, the skid shown in (Figure 19) was constructed. It consists of an oil field type skid with a metal roof and storage cabinet in one end. A mixing hopper was fabricated to make use of the 1200 psi waterflood water as a mixing jet for the dry sack chemicals. The mixture is stored in a 300 gallon plastic tank which allows direct observation and sampling of the solution. The tank contains an electric stirrer which is generally run for a couple of hours after each batch of chemical is mixed to ensure all the chemical dissolves. The mixture is pumped downhole by a large air powered chemical pump which has a variable speed, but precise displacement at any given speed. Subsequent designs have switched to a small triplex pump driven by a DC electric motor with speed control. A high/low pressure switch shuts down the pump if the main waterflood pump quits or a line ruptures. The supply water line comes directly from the waterflood line near the wellhead and the discharge line ties into the well just upstream of the wellhead assembly.

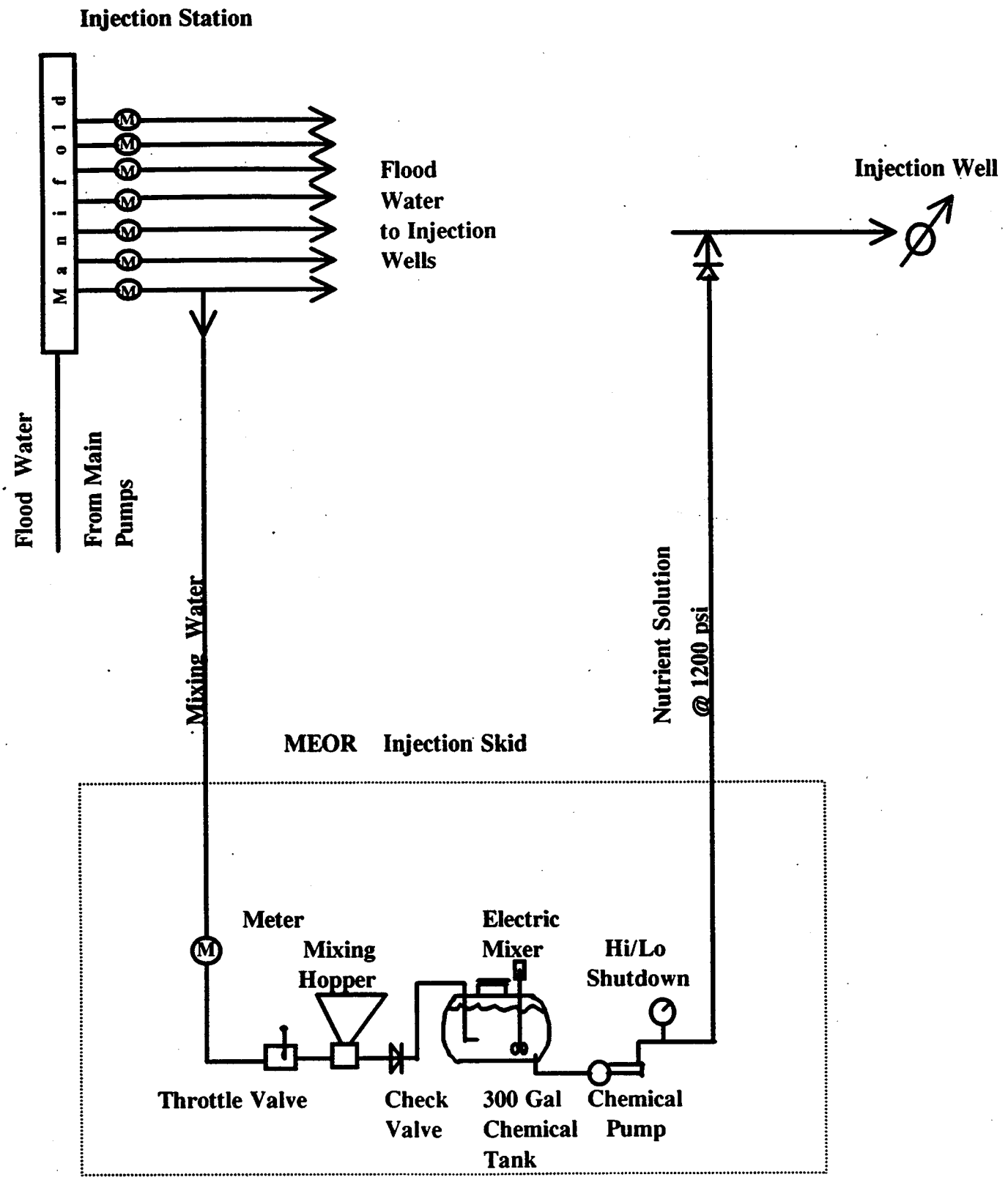

Figure 18. Flow Diagram for North Blowhorn Creek Unit MEOR Injection System

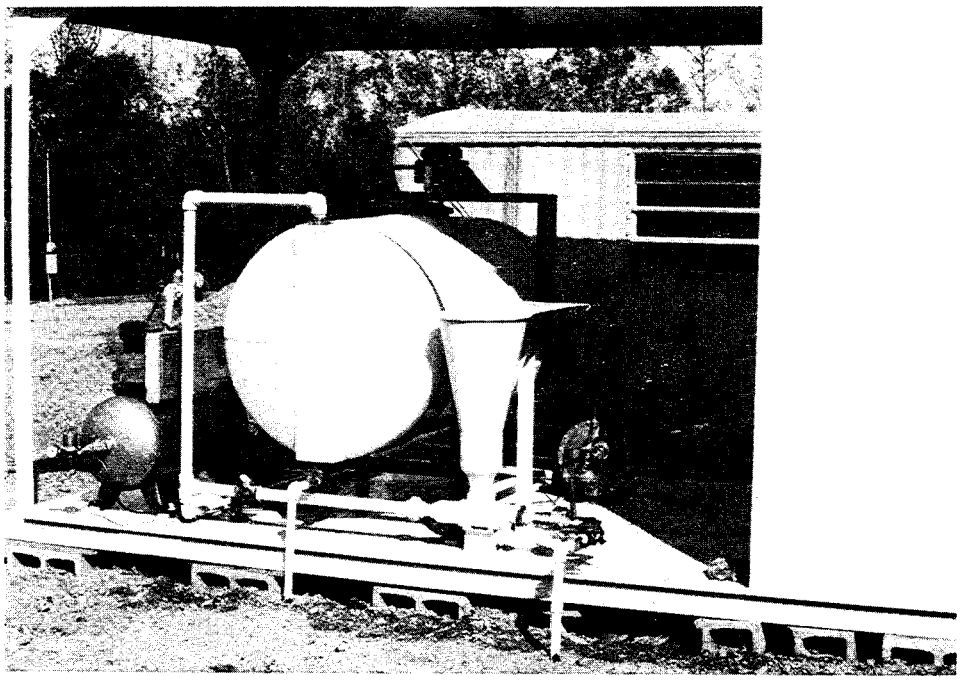

Figure 19. Picture of Skid

c. Implementation of Field Demonstration in First Test Pattern

The first injection skid was installed at the NBCU 2-14 #1 wellsite (which also happens to be a well testing station/injection manifold site), and after test runs with plain waterflood water, injection of nutrients was begun on November 21, 1994. Prior to beginning injection, the well tubing was found to contain a heavy buildup (approximately 1/2" thick) of mostly iron sulfide on the interior walls. The tubing string was replaced with a clean string to ensure there would be no adverse reaction or tubing plugging once nutrient injection started.

The current injection schedule calls for injection of 200 gallons of potassium nitrate solution every Monday, and 200 gallons of disodium hydrogen phosphate every Wednesday and Friday. The schedule allows a day between each injection to allow for washing the chemical tank and performing any other required maintenance on the skid. Thus far, the injection has gone well with no noticeable changes in waterflood injection rate or pressure. Based on the reliable mechanical performance of the first injection skid, three additional skids were ordered in December. It is anticipated that the second skid will be placed in service on the NBCU 11-5 #1 well around mid-January 1995.

When the injection of nutrients began in the field, additional waterflooding tests using live cores were also initiated and intended to parallel the field operation. For these studies, the injection water employed was the actual injection water used in the field and the chemicals employed were those used in the field operation. The schedule for adding nutrients to the injection water are the same as that used in the field operation. Additionally, two new cores have been employed in this study. If additional nutrients (e. g. hydrogen peroxide, sugar) appears to be needed to accelerate microbial growth, they will be tested in these cores before committing to changes in the field operation.

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