

X-Ray Diffraction analysis and flow testing of Hemlock Sandstone from core of the

XTO Energy Inc. (orig. operator was Shell Co.) MGS (Middle Ground Shoal) C31-26 (9,195.5'-9,196.4'), and

Union Oil Company of California MGS State 17595 No. 31 (9,525.2'-9,526').



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Alaska Geologic Materials Center Data Report No. 332

**BJ SERVICES COMPANY  
RESEARCH AND TECHNOLOGY CENTER**

*Special Core Analysis Group*

**TECHNOLOGY CENTER REPORT NO. 06-03-0250**

**XTO ENERGY  
FLOW TESTING  
HEMLOCK SANDSTONE  
FOR USE ON PLATFORM A OIL WELLS  
COOK INLET, ALASKA**

REPORT PREPARED FOR:

**MR. ROD EDWARDS  
BJ SERVICES TECHNICAL REPRESENTATIVE  
ALASKA**

APRIL 17, 2006



**GMC DATA REPORT 3 3 2**

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## SUMMARY

### General

Two core sections from the Hemlock Formation were analyzed to provide regain permeability data subsequent to mud damage and damage removal treatments.

### Flow Testing

Porosity and permeability testing was conducted after aggressive cleaning of plugs. Helium porosities range from 10.4% to 16.8%, and dry nitrogen permeabilities range from 1.58 md to 177 md at room temperature and 250 psi confining pressure for permeability. Results of flow testing indicate that a Paravan-25 preflush followed by an acid treatment is most effective in removing mud damage. The one step treatment with xylene and acid was not effective in removing damage. Results of all tests are summarized below:

FLUID	Depth, feet	Initial k to Oil, md	k to Oil after Mud, md	k to Oil after One Shot, md	k to Oil after Two Step Paravan-25 and Acid, md	Change, % (Relative to Mud Damage)
One Shot	9195.74	28.8	20.1	5.7	-	-72
One Shot	9525.37	0.357	0.262	0.068	-	-74
Two Step	9196.13	41	37.8	-	40.2	+6
Two Step	9525.35	0.290	0.250	-	0.239	-4

On the basis of the flow test data, we recommend that the 2 step system, consisting of a Paravan-25 preflush followed by the 10% HCl system, be used in the field.

### Mineralogy

Quartz was the principal mineral phase detected by bulk X-ray analysis. Feldspars are abundant with plagioclase being the more abundant feldspar variety. Potassium feldspar is present in both samples. Iron-bearing dolomite was detected in a minor quantity in both samples. The total percentage of clay minerals and sheet silicates, from bulk X-ray analysis, ranges from 4% to 11%, by weight. Kaolinite (migrating) is the most abundant clay mineral. Illite-smectite, a mixed layer swelling clay with 65%-80% non-swelling illite layers and 20% - 35% expandable smectite layers, was detected in both samples. Chlorite (iron-bearing) was detected in both samples. Illite (mica) was also detected in both samples. By comparison, the 9525 foot sample contains more feldspar and clay than does the 9196 foot sample.

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## INTRODUCTION

### Scope and purpose

The analysis of core plugs, drilled from two submitted Hemlock core section, Cook Inlet, Alaska, serves as the data base for this study. The study was requested by Mr. Rod Edwards, BJ Services technical representative, Anchorage, Alaska. Data generated by the study will be used to clean up possible oil-based mud damage from XTO Energy horizontal wells in Cook Inlet, Alaska. BJ Services Technology Center sample numbers and analyses performed in this portion of the study are listed below:

Depth, feet	SAMPLE NUMBER 06-03-0250	ANALYSIS				
		XRD	SEM	$\phi$	k	FLOW
9525.38	1	-	-	X	X	-
9525.37	2	X	X	X	X	X
9525.35	3	-	-	X	X	X
9525.47	4	-	-	X	X	-
9525.48	5	-	-	X	X	-
9525.49	6	-	-	X	X	-
9196.15	7	-	-	X	X	-
9196.13	8	X	X	X	X	-
9195.88	9	-	-	X	X	X
9195.74	10	-	-	X	X	-
9195.80	11	-	-	X	X	X
X = Analysis Performed      - = Analysis not Performed XRD = X-ray Diffraction Analysis    SEM = Scanning Electron Microscopy $\phi$ = Porosity      k = Permeability      FLOW = Flow Testing						

This study was initiated to provide regain permeability data subsequent to acidization and treatments.

### Analyses Performed

To supply data required from the study, the following analyses were performed and are reported in this portion of the study:

X-ray Diffraction Analysis  
Scanning Electron Microscopy  
Porosity and Permeability Determination  
Flow Testing with Acid  
Flow Testing with Two Step System

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## MINERALOGY - ROCK CHARACTERIZATION EVALUATION

### Sample Analyzed and Description of Methods

An evaluation of the rock mineralogy of portions of a plug from each core section was initiated to determine the mineralogic composition for use as a parameter in the design of a treatment system. Two samples were analyzed using semiquantitative bulk and clay fraction X-ray analysis (XRD). Portions of these samples were reserved for examination with the scanning electron microscope (SEM) to observe the distribution of clay and other pore filling minerals. Approximate weight percentages of mineral phases present in a sample, including clay minerals, are specifically determined by X-ray analysis. Clay mineral distributions and the overall morphology of the pore system (porosity and the degree of interconnection of open pores) are examined during scanning electron microscopy. Prior to analysis, residual contamination was removed by toluene vapor extraction.

### X-Ray Diffraction Analysis

Samples were analyzed using both bulk (whole rock) and fine (clay-size) fraction X-ray diffraction techniques. A portion of each selected sample was gently crushed, and crushed material was thoroughly mixed. Half of the crushed material was ground to a fine powder, and the bulk mineralogy of the sample was determined. The clay size fraction was separated from the second part of the crushed mixture, and the clay mineralogy of each sample was determined in both air dried and glycolated states. Note that the term trace (Tr) refers to a quantity of less than 1%.

Quartz was the principal mineral phase detected by bulk X-ray analysis (69% - 81%). Plagioclase is the more abundant feldspar variety (8% - 14%). Potassium feldspar (4% - 5%) was detected in both samples. Iron-bearing dolomite was detected in a minor quantity in both samples (1%). The total percentage of clay minerals and sheet silicates, from bulk X-ray analysis, ranges from 4% to 11%, by weight. Kaolinite (migrating) is the most abundant clay mineral (3%). Illite-smectite, a mixed layer swelling clay with 65-85% non-swelling illite layers and 20% - 35% expandable smectite layers, was detected in both samples (Tr - 3%). Chlorite (iron-bearing) was detected in both samples (1 - 3%). Illite (mica) was also detected in both samples (1% - 2%). By comparison, the 9525 foot sample contains more feldspar and clay than does the 9196 foot sample. Results of the analysis are given on the following page.

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# SEMIQUANTITATIVE X-RAY DIFFRACTION ANALYSIS XTO ACID STUDY

SAMPLE NO.: 06-03-0250 SAMPLE DEPTH, ft	2 9525.37	8 9196.13
CLAY MINERALS		
ILLITE-SMECTITE*	3	Tr
ILLITE	2	1
CHLORITE	3	1
KAOLINITE	3	2
CARBONATE MINERALS		
CALCITE (CaCO <sub>3</sub> )	Tr	ND
FERROAN DOLOMITE ((Ca(Mg,Fe)(CO <sub>3</sub> ) <sub>2</sub> )	1	1
OTHER MINERALS		
QUARTZ	69	81
PLAGIOCLASE FELDSPAR	14	8
POTASSIUM FELDSPAR	4	5
TOTALS		
CLAY MINERALS	11	4
CARBONATE MINERALS	1	1
OTHER MINERALS	87	94
*THE ILLITE-SMECTITE IS 20% - 35% EXPANDABLE SMECTITE LAYERS. Tr = <1% ND = NOT DETECTED		

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## Scanning Electron Microscopy

Portions of two samples were examined with the scanning electron microscope. The principal objective of this examination is to observe the distribution of clay minerals and other authigenic cements in the pore system. Secondly, the overall morphology of the pore system, particularly with regard to pore sizes and the degree of interconnection of open pores, was examined. Features of samples are illustrated in black and white photo images which accompany the report (Figures 1 - 2).

Samples examined represent poorly to moderately sorted, fine to lower medium grained feldspathic quartz sandstones. Quartz is the principal framework grain. Feldspars are present and have been diagenetically altered to clay minerals. Other framework grains were not observed. Kaolinite and illite-smectite are obvious authigenic clay minerals observed in these samples. The kaolinite may have replaced some feldspar grains. The clay is authigenic and locally fill pores and blocks entrances to pore throats. Illite-smectite bridges pores and pore throats. Minor quartz overgrowth development was observed on few quartz grains.

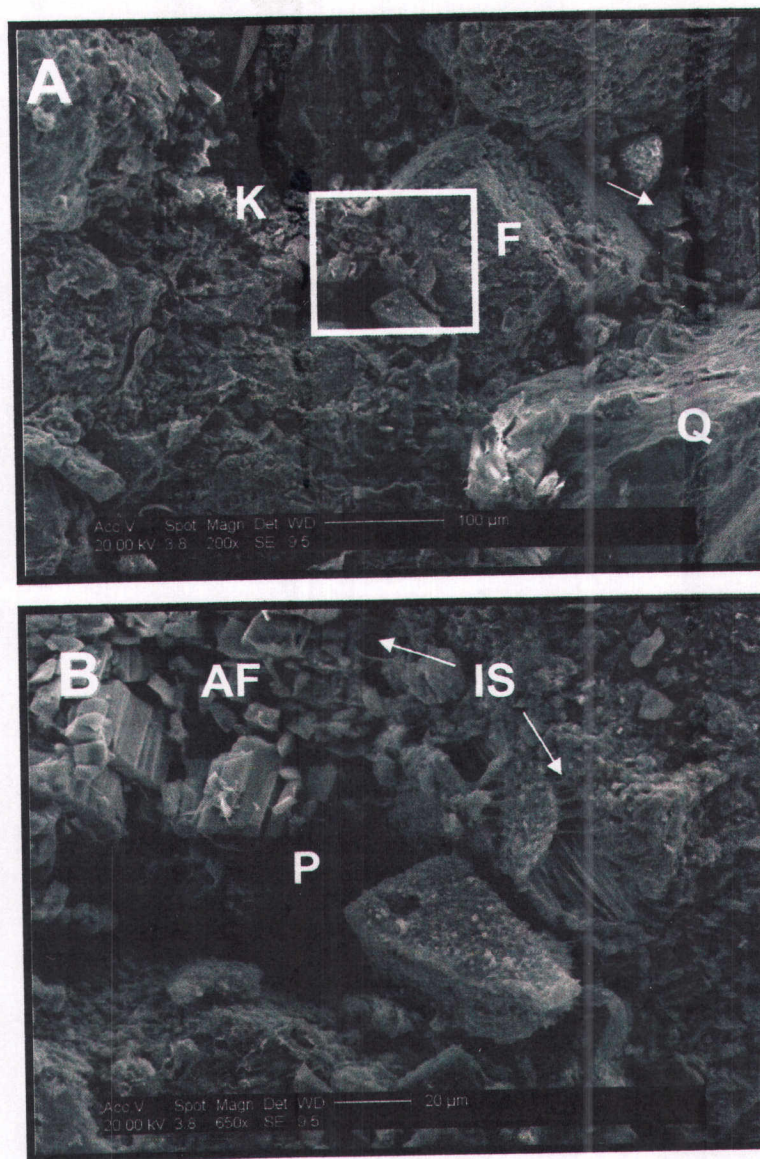
Porosity in these samples is limited by the precipitation of authigenic clay in the pore system, by the precipitation of quartz overgrowths on quartz grains, by long axis close packing of framework grains, and by the poor sorting (allows larger pores to be filled by finer grains). Pore and pore throat sizes are summarized below:

DEPTH, ft	MAXIMUM PORE SIZE, $\mu\text{m}$	MAXIMUM PORE THROAT SIZE, $\mu\text{m}$	OBSERVED PORE TYPE
9196.13 9525.37	100 30	5 1	P, M P $\approx$ M
P = PRIMARY INTERGRANULAR    M = MICRO (ANY PORE $< 5 \mu\text{m}$ )			

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**FIGURE 1**  
**SEM IMAGES**  
**XTO, PLATFORM A, 9525.37 FEET**

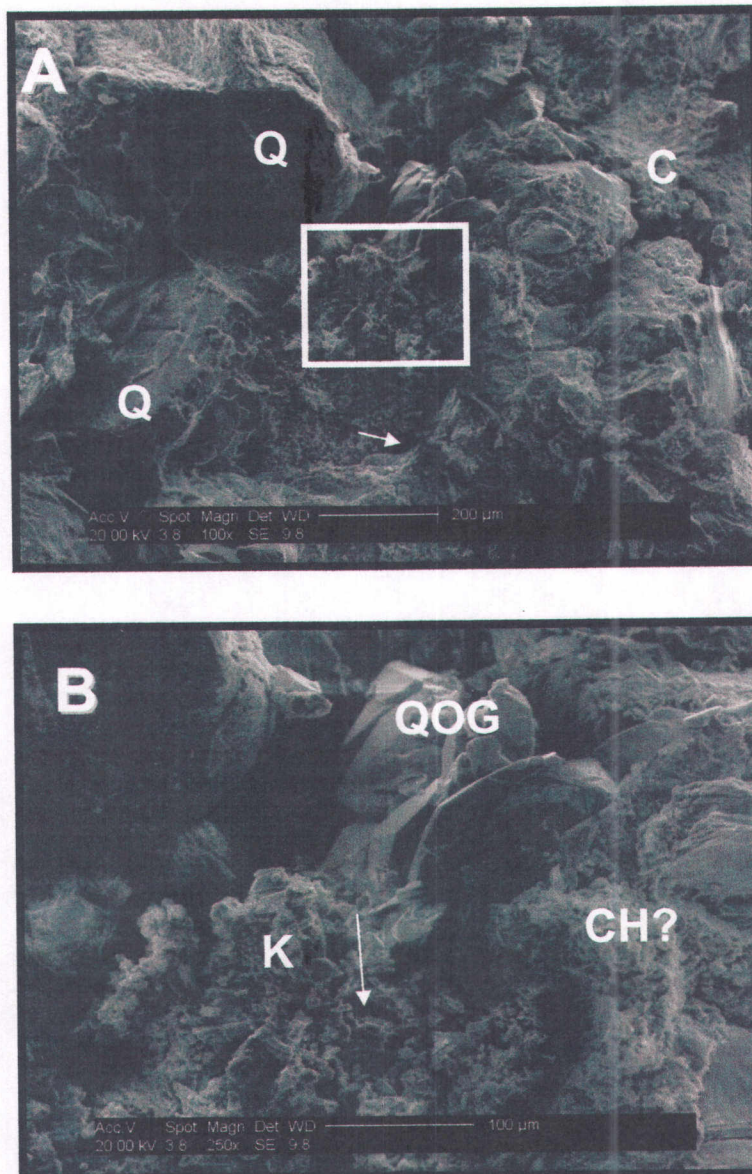


The portion of the sample illustrated in these SEM images represents a poorly to moderately sorted, upper fine to lower medium grained sandstone. Quartz (Q) is the principal framework grain. Most grains exhibit some overgrowth development. Feldspars (F) are abundant and have locally been altered (AF). Open pores (arrow and P) are present but are locally filled by silt-sized grains. Kaolinite (K) is a major authigenic clay mineral. The clay partially fills pores. Illite-smectite (IS) is abundant, forming thin rims on framework quartz grains and bridging pore throats. Magnification Photo A = 200x; Photo B = 650x.

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**FIGURE 2**  
**SEM IMAGES**  
**XTO, PLATFORM A, 9196.13 FEET**



The portion of the sample illustrated in these SEM images represents a moderately sorted, upper fine to lower medium grained sandstone. Quartz (Q) is the principal framework grain. Most grains exhibit quartz overgrowth development (QOG). Kaolinite (K) is the principal authigenic clay mineral observed. The clay partially fills pores and blocks entrances to pore throats. Micropores (arrow) are abundant in areas of the rock occupied by kaolinite. Chlorite (CH?) rims framework quartz grains. Feldspar grains have locally been altered to clay (C). The area illustrated in Photo B is an enlargement of the area enclosed by the box in Photo A. Magnification Photo A = 100x; Photo B = 250x.

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## PERMEABILITY AND FLOW TESTING

Eleven (11) 1 inch in diameter core plugs were drilled from the two submitted core sections. Prior to testing, residual hydrocarbon and other contaminants were removed from plugs, using the toluene vapor extraction technique. In this technique, extraction is continued until the fluid in contact with cores is colorless. Plugs were further cleaned with methanol to strip any residual fluids from the pore system. After cleaning, plugs were dried at 150 °F for 13 hours. Helium porosities are determined with a dual cell Boyle's Law porosimeter, and dry nitrogen permeabilities are determined with a standard permeameter. Bulk densities are determined from plug dimensions and plug masses. Results of analyses for plugs used in flow testing are given below:

Depth, ft	Sample No. 06-03-0250	Pore Volume, cm <sup>3</sup>	Bulk Density, g/cm <sup>3</sup>	Grain Density, g/cm <sup>3</sup>	He Porosity φ, %	k to N <sub>2</sub> , md
9525.38	1	3.46	2.374	2.662	10.8	1.58
9525.37	2	2.93	2.364	2.668	11.4	1.84
9525.35	3	2.60	2.393	2.669	10.4	1.93
9525.47	4	2.59	2.363	2.665	11.3	1.59
9525.48	5	3.66	2.367	2.669	11.3	1.66
9525.49	6	3.37	2.372	2.663	10.9	1.67
9196.15	7	5.19	2.201	2.643	16.7	177
9196.13	8	4.46	2.198	2.642	16.8	132
9195.88	9	4.82	2.241	2.646	15.3	84.1
9195.74	10	4.31	2.245	2.650	15.3	79.2
9195.80	11	5.25	2.225	2.650	16.0	70.3

### Special Testing

Testing was initiated to fulfill the following objectives:

- Determine the effectiveness of a single step acid treatment in removing an oil based mud from the rock
- Determine if the previously developed mud damage removal system is effective with formation oil and formation rock, with reduced concentrations of Paravan-25 and US-40.

Test fluids, procedures and results are summarized on the following pages.

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## Fluid Systems

Systems utilized in flow testing are given below:

Fluid Formulations	
Fluid	Additives
MI Versadrill Oil Base 9 ppb	Supplied by XTO. 80/20 Oil external/water emulsion. Versacoat 5 ppb; Versawet 2.5 ppb; Lime 2 ppb; IMG400 8 ppb; Versa HRP 1 ppb; CaCl <sub>2</sub> 25%
One Shot Acid Plus 10% HCl/25% Xylene (Tests 1 - 2)	4 gpt S-400; 1 gpt NE-118; 3 gpt NE-110W; 50 gpt US-40; 5 gpt Cl-27; 30 ppt Ferrotrol-300 + 7 ppt Ferrotrol-200; 15 gpt AS-6
Pretreat - Tests 3 - 4	3% KCl; 25 gpt Paravan-25; 5% US-40; 10 gpt NE-110W
Oil	70% Mineral Oil/30% Isopar L and supplied by XTO
10% HCl - Tests 3 - 4	25 gpt Paravan-25; 5% US-40; 3 gpt NE-110W; 30 ppt Ferrotrol-300; 7 ppt Ferrotrol-200; 5 gpt Cl-25 - Note that AS-6 was not omitted from this acid because field oil was injected prior to acid injection.
AQUEOUS FLUIDS ARE BASED IN FILTERED FRESH (TOMBALL TAP) WATER.	

**Testing:** All testing was performed in accordance with standard BJ Services test procedures

Test procedures for the flow tests are, generally, as follows:

1. Prior to analysis, plugs were seated in rubber sleeves at 1000 psi confining pressure and flowed with filtered formation water (when available) to core saturation. The system was gradually heated to 165 °F. A backpressure of 150 psi was used throughout all tests. This step is common to all flow test procedures.

### **Tests 1 - 2; Mud Damage followed by the One Shot Acid Plus System**

2. Flow was established in an arbitrary formation to wellbore (production) direction with laboratory oil to steady state permeability.
3. After steady state was achieved, formation oil (2 pore volumes) and mud were injected dynamically (cross-face flow) at 500 psi. The mud was then statically shut in for 6 hours.

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4. Plugs were flowed in the production direction to determine a damaged permeability. This step is necessary to make sure that a damaged plug is being treated. For example, if the plug is not damaged by mud injection and if the treatment is run immediately after mud injection, an unsuccessful treatment, relative to mud damage removal, could appear to be successful if application of the treatment did not result in damage to the core.
5. The One Shot Acid Plus system (10 pore volumes) was injected in the wellbore to formation direction.
6. Flow was established in an arbitrary formation to wellbore (production) direction with laboratory oil to steady state permeability.
7. Results are illustrated on Figures 1 - 2.

***Preflush, "New" Acid System Testing***

8. Steps 1 through 3 above.
9. Step 4 above, using laboratory oil as the flowing medium.
10. The Paravan-25 system was injected, followed by the injection of 10 pore volumes of the "new" acid system. Flow was re-established in the forward (production) direction to steady state permeability.
11. Results are illustrated on Figures 3 and 4.

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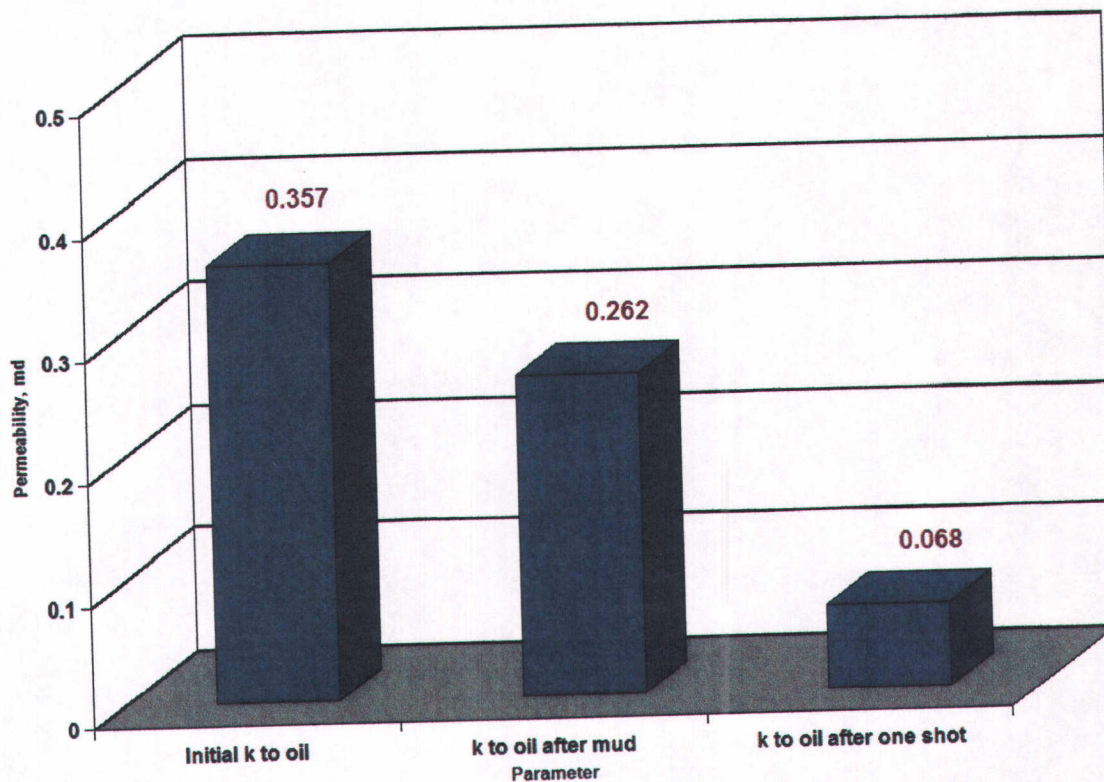
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**FIGURE 1**  
**REGAIN PERMEABILITY TESTING**

**MUD DAMAGE FOLLOWED BY THE ONE SHOT PLUS SYSTEM**  
**LAB OIL AS FLOWING MEDIUM**



Permeability to Lab Oil:  
Permeability to Lab Oil after Mud:  
Permeability to Lab Oil after Acid:

0.357 md  
0.262 md  
0.068 md

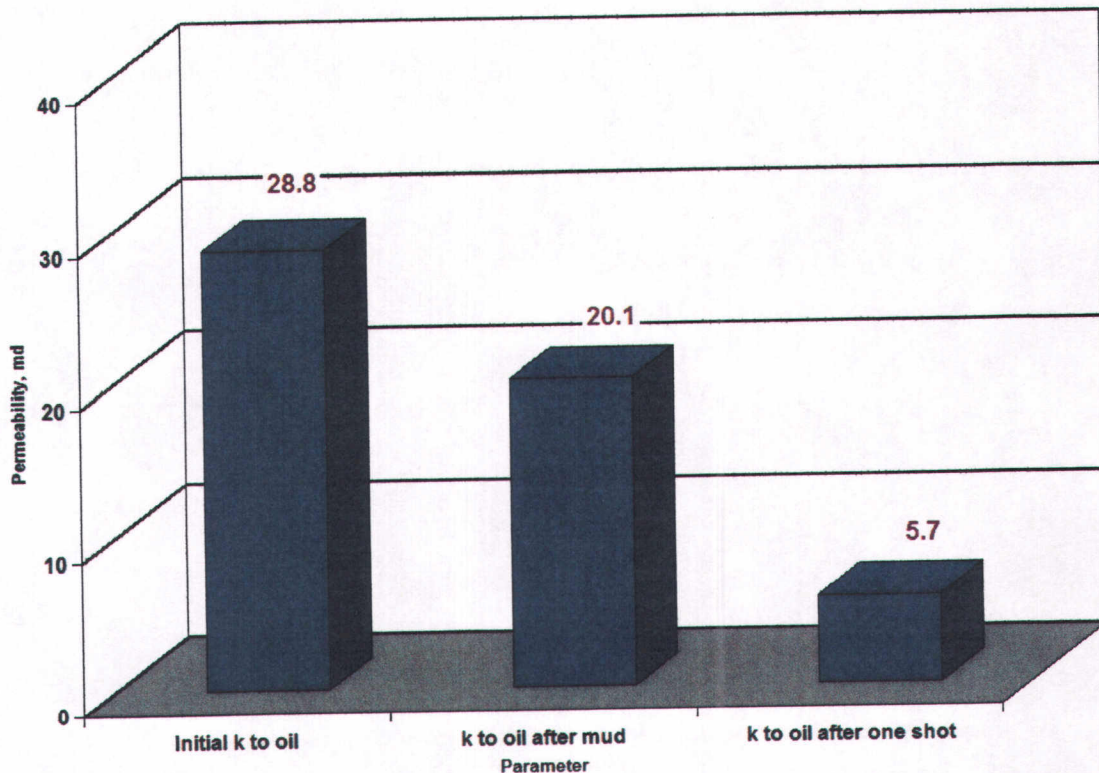
The acid did not remove mud damage. The permeability was further reduced subsequent to acid contact.

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**FIGURE 2**  
**REGAIN PERMEABILITY TESTING**

**MUD DAMAGE FOLLOWED BY THE ONE SHOT PLUS SYSTEM  
LAB OIL AS FLOWING MEDIUM**



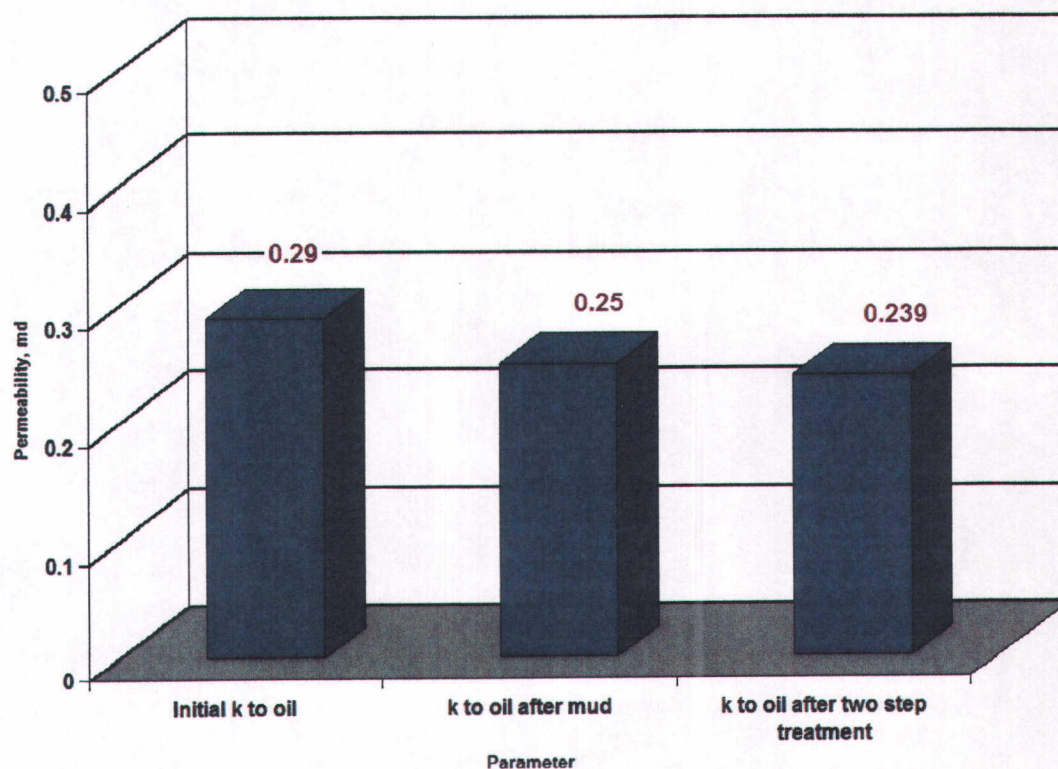
Permeability to Lab Oil:	28.8 md
Permeability to Lab Oil after Mud:	20.1 md
Permeability to Lab Oil after Acid:	5.7 md

The acid did not remove mud damage. The permeability was further reduced subsequent to acid contact.

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**FIGURE 3**  
**REGAIN PERMEABILITY TESTING**  
**LAB OIL AS FLOWING MEDIUM**  
**MUD DAMAGE FOLLOWED BY THE PARAVAN-25 PREFLUSH**  
**FOLLOWED BY THE "NEW" ACID SYSTEM**



Initial Permeability to Oil:	0.290 md
Permeability to Oil after Mud:	0.250 md
Permeability to Lab Oil after Acid:	0.239 md

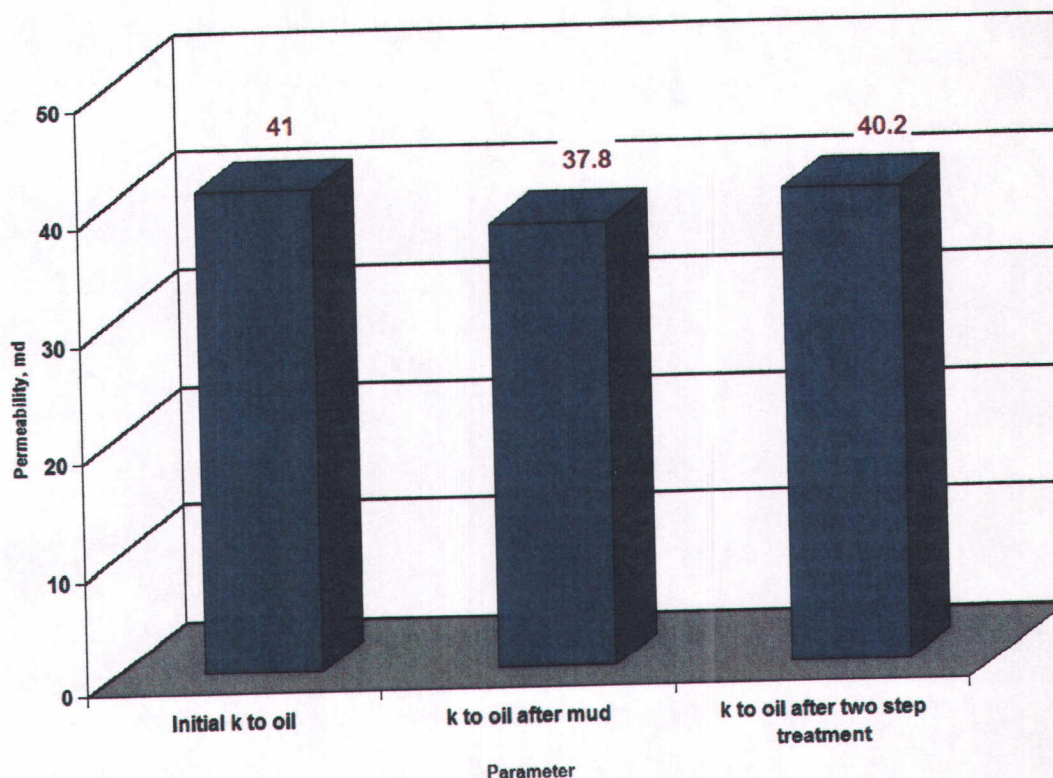
The two step treatment did not remove mud damage. The permeability was slightly reduced subsequent to acid contact

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**FIGURE 4**  
**REGAIN PERMEABILITY TESTING**

**LAB OIL AS FLOWING MEDIUM**  
**MUD DAMAGE FOLLOWED BY THE PARAVAN-25 PREFLUSH**  
**FOLLOWED BY THE "NEW" ACID SYSTEM**



Initial Permeability to Oil:	41.0 md
Permeability to Oil after Mud:	37.8 md
Permeability to Lab Oil after Acid:	40.2 md

The permeability of the rock was nearly restored to its original value by the use of the two step treatment.

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## COMMENTS

**General** - Two core sections from the Hemlock Formation were analyzed to provide regain permeability data subsequent to mud damage and damage removal treatments.

**Flow Testing** - Helium porosities range from 10.4% to 16.8%, and dry nitrogen permeabilities range from 1.58 md to 177 md. Results of flow testing indicate that a Paravan-25 preflush followed by an acid treatment is most effective in removing mud damage. The one step treatment with xylene and acid was not effective in removing damage. Results of all tests are summarized below:

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Laboratory Report No.: 06-03-0250

Reported by: Carolyn S. De Vine

Requested by: Rod Edwards

Location: Alaska

Analyzed by: K. Spurlock, J. Cutler, L. Vestal, G. Braun, C. De Vine

Data Integration: C. De Vine

Distribution: Rod Edwards, Nabil El Shaari, SCA File, GS File, TTC File

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