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CHEMICAL RESEARCH AND DEVELOPMENT DEPARTMENT

29-275-35

HALLIBURTON SERVICES DUNCAN, OKLAHOMA

## LABORATORY REPORT

No. T11-A111-86

To	Mr. John Ti	nsley		Date	October 14, 1986
	Halliburton	Services		This access to all a second of	FILLIUM AND CO. Town of the co.
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We give below r	esults of our exar	nination of <u>t</u>	the submitted	formation core plu	igs.
Submitted by	Mrs. Sharon	Voskuhl for C	Cities Servic	е	
Marked	Well: Location: Depth: Formation: Samples Rec	Alexander D N Sec. 29-27S-3 2,895.25 to 3 Council Grove	35W; Grant Co 3,017.14 ft		

#### Purpose

The purpose of this project was to analyze the submitted formation core plugs. The following tests and examinations have been conducted: x-ray diffraction, acid solubility, permeability, porosity, regained (gas) permeability, fluid loss and scanning electron microscope.

#### Discussion

Four 1-in. diameter formation core plugs were received from Cities Service. The formation samples represented the Council Grove formation in Grant County, KS. The two shallower samples (2,895.25 to 2,895.44 ft) were very fine grained, red, silty, calcareous sandstone. Notes received with the core plugs indicated that they represented the Speiser member. The two deeper samples (3,016.17 to 3,017.14 ft) were finely crystalline, white carbonate. The submitted information indicated that the carbonate core plugs represented the Bader member.

X-ray diffraction analysis indicated that the sandstone samples were composed of 40-50% quartz, 15-20% feldspar, 15-20% calcite, 2-10% dolomite, up to 2% kaolinite, 2-5% illite, 2-5% mixed layer clay, up to 2% chlorite, and scattered anhydrite. The acid solubilities were in the range of 25%. SEM examinations revealed that the calcite present in the

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#### Discussion Cont'd.

sandstone samples was the primary cementing agent. Dolomite and secondary quartz also helped to cement the framework of very fine sand to siltsized quartz and feldspar grains. The visible porosity was poor which correlated well with the measured porosity of about 10%.

X-ray indicated that the deeper carbonate samples were primarily calcite with traces of quartz, feldspar, mixed layer clay and anhydrite. The acid solubilities were greater than 92%. SEM examinations confirmed that the samples were almost pure carbonate. The visible porosity was slightly better than that of the sandstone samples; again, this observation correlated well with a higher measured porosity of about 14%.

Regained permeability tests were conducted with the two carbonate samples. Tests were attempted on all four core plugs but the two sandstone samples were too tight for fluid flow. The sandstone samples had gas permeabilities less than 0.03 md. The slight differences in permeabilities during the permeability analyses and initial permeabilities in the regained permeability tests is due to the use of different apparatus and cutting of the core plugs' length after the permeability analyses and prior to the regained permeability tests. These differences are not significant.

Because the carbonate samples had low permeability (0.25 to 0.35 md), the regained permeability tests were modified slightly. Normally, we flow the saturation fluid, which in this case was 2% KCl water, followed by a standard API fluid loss test. After the fluid loss test, the test plugs are "regained" for 24 hours. Because the plugs were tight, it was felt that fluid retention could be a problem. In order to isolate the effect of fluid retention from that of damage due to gel residue, the test plugs were regained after flowing 2% KCl water only. Core No. 3 and Core No. 4 regained 79.6% and 74.6% of their initial permeabilities, respectively. This confirms that fluid retention could cause permeability reduction.

After the first regain period, the test plugs were re-saturated and standard fluid loss tests were conducted with a 30 lb PUR-GEL system and a VERSAGEL LT, 1300 system. Fluid loss coefficients on the order of 0.002 ft/min. 2 were achieved. Core No. 3 was flowed with the PUR-GEL system and regained 89.8% of its initial permeability. Core No. 4 was flowed with VERSAGEL LT 1300 and regained 62.9% of its initial permeability. The low regained permeability with the VERSAGEL LT 1300 system is believed to be due to the fact that the breaker was inadvertently omitted from the system. However, this could not be verified because there were not any more samples available.

#### Recommendations

Due to the low permeability of the sandstone samples, regained permeability tests could not be used to evaluate the degree of water sensitivity. However, based on the mineralogical composition, one would

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#### Recommendations Cont'd.

not expect the formation to be highly water-sensitive. The only swelling clay detected was mixed layer clay which was present in the amount of 2-5%.

Due to the lack of clay minerals, one would not expect the carbonate portion to be water-sensitive. However, the regained permeability tests with base fluid only (2% KCl) indicated that some fluid retention may occur. The use of methanol is therefore recommended to reduce the surface tension of the fracturing fluid and aide in fluid recovery. A non-wetting agent may also be of benefit to help enhance load fluid recovery. Using an energized fluid would also help in recovering the load fluid. A CO, foam would have the added benefit of lower surface tension in addition to the gas assist.

As mentioned in the discussion, the breaker was inadvertently omitted from the gelled fluids. This mistake can be used to illustrate the importance of designing a proper breaker system for low temperature applications.

Data Core Description

Core No.	Depth (ft)	Description
1	2,895.25	Very fine-grained, red, silty, calcareous sandstone, somewhat marbled (red and tan) in appearance. Hard and dense.
2	2,895.44	Very similar to Sample No. 1.
3	3,016.17	Finely crystalline, white carbonate. Contains fossil fragments. No vugs or fractures. Hard and dense but less dense than Sample Nos. 1 and 2.
4	3,017.14	Very similar to Sample No. 3.

## Data Cont'd.

# Qualitative X-ray Diffraction and Acid Solubility Analyses

To identify the types and relative quantities of minerals in PURPOSE: the formation sample.

PROCEDURE: A pulverized 1 gram sample is placed in an x-ray beam and rotated through an arc. The x-ray beam is diffracted by the sample and the diffraction patterns are recorded.

The diffraction patterns are used to identify the types of RESULTS: minerals present and their relative quantities. The relative quantities for the submitted samples are as follows:

Sample No. Depth (ft)	1 2,895.25	2 2,895.44	3 3,016.17	4 3,017.14
HCl Solubility % *	22.9	28.0	92.3	94.0
Quartz % Feldspar % Calcite % Dolomite % Kaolinite % Illite %	40-50 15-20 15-20 2-5 0.5-2 2-5	40-50 15-20 15-20 5-10 0.5-2 2-5	0.5-2 0 80-100 0 0	0.5-2 0.5-2 80-100 0 0
Smectite %	0	0	0	0
Mixed Layer %	2-5	2-5	0.5-2	0.5-2
Chlorite $\%$	0.5-2	0.5-2	0	0
Anhydrite %	2-5	0	2-5	0.5-2

<sup>\*</sup> One gram of sample is added to 0.5N HCl. After reaction, the excess acid is titrated with 0.2N NaOH. The results are calculated and reported as calcium carbonate. (Note: a pure dolomite will be reported as 108%.)

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#### Data Cont'd.

## Permeability and Porosity Analyses

PURPOSE: To measure the permeability and porosity of a porous media.

PROCEDURE: A test plug 1 inch in diameter by 1.187 inches long is obtained from the submitted core sample so that the longitudinal axis of the plug is normal to the depth. The plug is then oven dried and allowed to cool to room temperature. Permeability is determined by placing the plug securely in a Hassler Sleeve of the Permeameter. Dry air is then flowed through the plug. Measurements of flow rate and pressure drop across the plug are then obtained.

Porosity is determined by placing the plug securely in a Hassler Sleeve of the Porosimeter. The system is purged with helium gas. The meter has a known storage volume in which the helium gas is allowed to reach equilibrium and a reference point is set on the gauge and a reading taken. The valve to the plug chamber is opened allowing the helium gas to expand into all the void spaces of the plug. A gauge reading is taken in cubic centimeters. The plug is removed and a non-porous plug is placed in the holder and the gas volume is measured in the same manner as with the sample plug. The difference in the volumes is the pore volume of the test plug. The bulk volume is calculated from measuring the plug with calipers.

#### **RESULTS:**

Permeability (md) = 
$$\frac{\text{CQL}}{0.7845 \text{ (D) (D)}}$$
Porosity (%) = 
$$\frac{\text{P.V.}}{\text{B.V.}} \times 100$$

C is a constant taken from the Permeameter calibration charts

Q is the flow rate in cubic centimeters per second (cc/sec)

L is the plug's longitudinal length in centimeters (cm)

D is the diameter of the plug in centimeters (cm)

P.V. is the pore volume measured in cubic centimeters (cc)

B.V. is the bulk volume measured in cubic centimeters (cc)

Sample No.	Depth (feet)	Porosity(%)	Air Permeability (md)
1	2,895.25	9.8	0.03
2	2,895.44	10.0	0.01
3	3,016.17	12.2	0.30
4	3,017.14	16.9	0.35

#### Data Cont'd.

## Regained Permeability Tests (N<sub>2</sub> Gas)

Sample No.	Depth (ft)	Test Pressure (psig)
1	2,895.25	1,000
2	2,895.44	1,000
3	3,016.17	500
4	3,017.14	500

PURPOSE: To measure the effect of fluids on the permeability of samples.

PROCEDURE: The core test plugs (1 inch in diameter by 1 inch in length) are oven-dried and the sides are sealed with epoxy to insure linear flow through the test plugs. Initial nitrogen permeability measurements are then obtained in one direction. The core plugs are then saturated and a measured volume of fluid is flowed through the core plug in the opposite direction. Fluid flow temperature was 110°F. Some of the plugs are then subjected to a 36 minute fluid loss test with gelled fluids at 110°F and a test pressure of 1000 psig. The plugs remained in the test cells for 16 hours at 110°F to obtain a gel break prior to regained nitrogen flow. Regained permeability measurements are obtained in the original direction. Gas flow measurements are at 75°F.

RESULTS: The data are reported as a percent of the initial permeability recovered.

Sample No.	Initial Perm.  Gas $(N_2)$ $\underline{ \text{(md)}}$	Saturation Fluid	Test Fluid*
1	0.02	2% KCl - no flow	
2	0.01	2% KCl - no flow	
3	0.24	2% KC1	PUR-GEL
4	0.31	2% KC1	VERSAGEL LT 1300

<sup>\*</sup> Fluid descriptions are under the Fluid Loss Tests section.

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# Data Cont'd.

# Regained Permeability Tests (N2 Gas) Cont'd.

		Percen	t Regai	lned Per	meabili	$tv(N_a)$	we. Ti	me	
						(1.2)	*** 11	.mc	
Time (hrs)		Time (hrs)				Time (hrs)		Time (hrs)	%
0.5	64.3	Sample 1.0		after F1 2.0				24.0	79.6
0.5	64.3			3 after 2.0				24.0	89.8
0.5	47.3	Sample :	No. 4 a 61.0	fter Fl	ow with 68.8	2% KCl 4.0	Water 74.6	24.0	74.6
0.5		ample No							62.9

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## Data Cont'd.

#### Fluid Loss Tests

To describe the leakoff properties of a particular fluid on a PURPOSE: formation rock sample.

PROCEDURE: The sides of a test plug (15/16 inches in diameter by 1 inch in length) taken horizontally from a core are sealed with an epoxy so that the test fluid flows linearly through the test plug. A 1000 psi differential pressure is applied to the test fluid. The fluid volume through the test plug is recorded as a function of time for 36 minutes as per API Test procedures.

RESULTS: The results of these timed volume readings are plotted as a function of volume versus the square root of time. The slope of the line obtained when the data is plotted on regular graph paper will give the C values and spurt volume. The test temperature was  $110^{\circ}F^{\text{W}}$ .

Sample No.	Depth (feet)	Perm. Gas (md)	Gelled Fluids	C (feet/ sq. rt. min)	Spurt Loss (gal./sq. foot)
3	3,016.17	0.24	PUR-GEL	0.00208	0.0
4	3,017.14	0.31	VERSAGEL LT 1300	0.00197	0.0

#### Gelled Fluids Description

PUR-GEL: 30 1b Wg-18, 2.5 1b BA-2, 2.5 1b BA-10,

and 0.5 gal CL-19/Mgal 2% KCl Water

VERSAGEL LT: 30 1b WG-11, 4 1b BA-2,

and 3 gal MYF-3C/Mgal 2% KCl Water

### Data Cont'd.

## Scanning Electron Microscope (SEM) Examination

PURPOSE: To provide a greatly magnified view of a core sample.

Minerals present in the sample can be identified and their location observed.

PROCEDURE: A core chip with a freshly broken surface is required for this examination. The sample is coated with a gold palladium alloy and placed in the vacuum chamber of the SEM. The core chip is viewed at a high magnification and a photomicrograph is taken. An associated energy dispersive x-ray (EDX) is used to help identify the mineral content of the sample.

RESULTS: The framework grains can be identified and their size approximated. The location of the clay minerals within the sample can be observed. The SEM can produce, in effect, a pseudo three dimensional view of formation pore spaces. The area of the sample viewed is very small and may not clearly characterize the entire formation.

Sample No. 1
Depth: 2,895.25

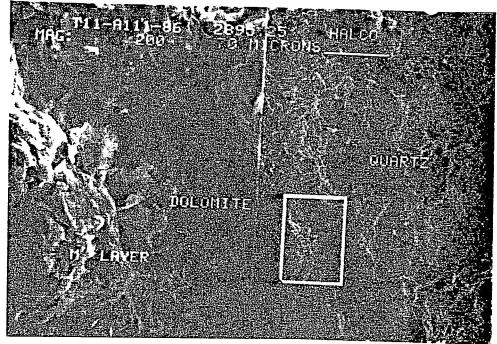


Figure No. 1; Negative No. 0456; Magnification 200x and 1000x

This sample has a framework of very fine sand to silt-sized quartz and feldspar grains cemented by micro-crystalline calcite, silt-sized dolomite and secondary quartz overgrowth. Mixed layer and illite clays line and infill pore spaces and lightly cover the grain surfaces.

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## Data Cont'd.

#### Scanning Electron Microscope (SEM) Examination

Sample No. 2 Depth: 2,895.44

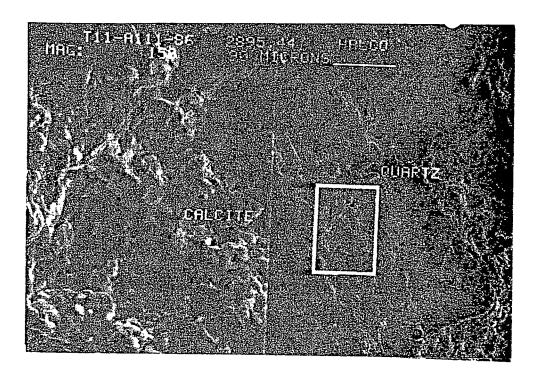


Figure No. 2; Negative No. 0457; Magnification 150x and 750xVery similar to Sample No. 1.

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### Data Cont'd.

## Scanning Electron Microscope (SEM) Examination

Sample No. 3 Depth: 3,016.17

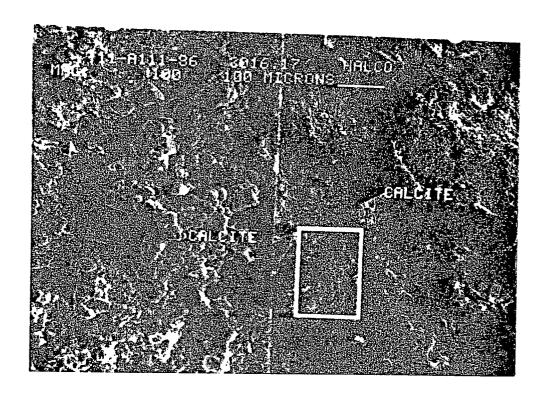


Figure No. 3; Negative No. 0458; Magnification 100x and 500x

This sample consists of calcite crystals ranging from 100 µm to less than 5  $\mu m$ . Poor to fair visible porosity.

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Data Cont'd.

# Scanning Electron Microscope (SEM) Examination

Sample No. 4
Depth: 3,017.14

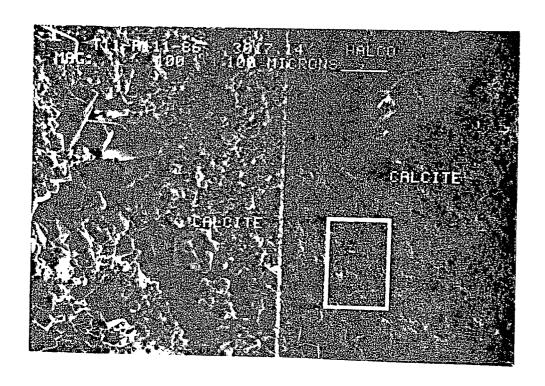


Figure No. 4; Negative No. 0459; Magnification 100x and 500xVery similar to Sample No. 3.

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#### Remarks

The data in this report were summarized to Mr. Randy Haynes by Ms. Mary Anderson during a telephone conversation on September 29, 1986.

## Core Sample Disposition

There was no sample remaining after completion of the requested tests.

KEYWORDS:	Cities Service	Liberal	Council Grove	Speiser
	Grant County	Bader	Kansas	WI

ABSTRACT:

Four core plugs from Council Grove Formation in Grant County, KS. Two at 2,895 ft were siltstone (25% sol., <0.03 md). Two at 3,017 ft were limestone (92% sol., 0.25-0.35 md). Tests: x-ray, solubility, SEM, permeability, porosity, regained permeability.

#### Data Book Reference

The data presented in this report are recorded in: Stimulation Engineering and Design Lab Book No. 5996, page 57; Stimulation Engineering and Design Lab Book No. 6028, pages 53 & 54; Analytical Lab Book No. 6008, page 65; Analytical Lab Book No. 6026, page 70; and Analytical Lab Book No. 6050, page 19.

cc: R. M. Lasater

L. E. Harris

B. A. Matthews

K. W. McKown

D. P. Russell

R. J. Haynes

Respectfully submitted,

Laboratory Analyst

Anderson, Simon, Loghry, Arrington, Tanaka, Black

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# To Jim Barnheit:

Several noteable things accorred on This well. One thing of interest was the way the Council grove fell in a around the matrix triangle. Most of my Commen. were made without looking at the logs. After looking at the perforated interval, I had to vevise some of my thinking One Thing That bothered me was the high Perosity streek at 2837 was good by log Calculations. The point at 2835 looked net. Guess it shows I don't know for sure what I am doing. The location of these Doints on the mid Plot fools me. I thingst They would be toward the quart Point Instead of at the NW of the Calcite Point. We can discuss this more later.

Take Care

MID Chart 7

THE MATRIX IDENTIFICATION (MID) PLOT

