# EFFECT OF PRESSURE ON WETTABILITY RESTORATION AND CRUDE CHARACTERIZATION USING INFRA-RED SPECTROMETRY

#### **ABSTRACT:**

Experiments were conducted to investigate the role of pressure on the process of wettability restoration. Cores from two North Sea fields were used for investigation. It is found that pressure has significant effect on the wettability restoration. Restoration of cores at pressures lower than the reservoir pressure resulted in cores which were more oil-wet. The wettability measurements were made using Amott method.

This paper also summarizes the proper care in handling the core as well as the oil being used. Drying of the core is probably the single most important factor that can damage the core permanently. And the oil oxidation by exposure to air can lead to forming chemicals that can alter the wettability of the core. The oil oxidation processes is simulated in the laboratory. Characterization of the oil using infra-red spectrometry is found to be better than the usual acid number approach.

It is recommended that the cores should be properly preserved and fresh un-oxidized crude should be used for tests where measurements at core native-state are desired.

#### **INTRODUCTION:**

In the process of coring and core retrieval, the cores often get contaminated. Many factors may be responsible for this alteration of basic properties, including (a) core contamination by drilling mud (b) precipitation of asphaltenes from the oil as the core is brought to the surface and (c) exposure of the core to ambient air after core retrieval. These changes may alter the rock wettability substantially and may render the core useless for special core analysis purposes.

If the cores are not fresh, preserved samples, the wettability of these cores should be restored in the laboratory to the native-state (in situ) wettability prior to conducting any special core analysis (SCAL).

Most rocks, in their natural state, prior to migration of oil were water-wet. There are some exceptions where the rock contains coal or sulfites etc., which are oil-wet substances in their natural state. However, after oil migration, the equilibration and diagenisis process is primarily responsible for altering the rock wettability from water-wet to mixed-wet or oil-wet. According to Archer et al<sup>[1]</sup>, more than 50% of the reservoirs may be either mixed-wet or oil-wet.

For wettability restoration, attempt is made to simulate the above process in the laboratory. Two important steps in the process of wettability restoration are (a) cleaning the core and (b) then aging it to the native-state wettability after restoring the water and oil saturations to the initial state. The purpose of core cleaning is to clean the cores to a water-wet state as they were prior to migration of oil. After cleaning, the core is saturated with water and oil and then aged to simulate the diagenisis process.

In spite of the large volume of data on core cleaning<sup>[2-7]</sup>, there is no sure way of knowing which solvent, or a sequence of solvents, will be the most effective. The core cleaning is basically a trial and error process. We will present our experiences in core cleaning. We have found that the cores that are not properly preserved and are allowed to dry out are hard to clean. The drying of the cores may be the single most important factor in altering the core wettability permanently. These results will be discussed in detail in later sections. We found that after cleaning, the core is saturated with brine and crude oil. To do this, the core is first vacuum saturated with brine and then oil flooded to the desired water saturation state. Then it is aged to simulate the natural equilibration process. There are many questions about the appropriate conditions for aging. For example, how long and at what temperature and pressure conditions the core should be aged.

The question of time was addressed by Mungan<sup>[8]</sup>. In his experiments, he demonstrated that the bulk of the wettability changes occur in first 2-3 weeks. After that, the changes are rather slow. In our laboratory, we use 30 days of aging to simulate the geological aging process.

It is generally believed that temperature has much more pronounced effect on aging than pressure. This is due to higher solubility of asphaltenes and other polar materials in the crude at elevated temperatures. In our experiments, all aging process was done at the reservoir temperature.

There is, however, hardly any data or information on the effect of pressure on aging<sup>[9]</sup>. Often, aging at low pressures, 100-300 psi, is preferred due to ease of experimentation. (The low pressure should still be high enough to prevent any fluid boil-offs in the core at the desired temperature.) The purpose of this work was to investigate the role of pressure on wettability restoration while aging. We will describe the aging experiments at various pressures in this report.

Unless specified otherwise, we have used fresh stock tank crude in our experiments. All precautions were taken to minimize crude exposure to air. The freshness of the crude is particularly important. The crude was obtained from the fields in containers under gas blanket (container air was bled-off by separator gas). The crude was stored inside the laboratory in epoxy-coated containers to minimize crude oxidation. Infrared spectrometry, instead of acid number, is used to characterize the crude oxidation.

We will discuss our results in three sections. In the first section, the role of pressure on wettability restoration is investigated. In the second section, we will discuss the effectiveness of various solvents in cleaning the cores. And finally, in the third section, we will discuss the infrared spectrometry technique of crude characterization to measure oxidation number of the crude.

#### ROLE OF PRESSURE IN AGING FOR WETTABILITY RESTORATION:

All cores were cleaned prior to aging. The objective of the cleaning process was to clean the cores to a strongly water-wet state. However, some cores could not be cleaned to the desired water-wet state due to undesirable circumstances prior to cleaning that caused permanent alteration in their wettability. In such cases, the wettability after cleaning was taken as the reference wettability.

After cleaning, the core was saturated with the brine and the crude oil to desired saturation levels. The core was then aged to simulate the geological equilibration process. Ideally, one would like to

do the aging at reservoir pressure and reservoir temperature. However, working in the laboratory at the reservoir pressure is difficult, particularly when live crude is used which contains dissolved gas. This has been the primary motivation over the years in reducing the pressure as much as possible without reducing the effectiveness of the aging process.

The aging experiments were conducted at two pressures; (a) Low pressure of 200 psi. and (b) High pressure at crude bubble point.

In low pressure aging experiments, a pressure of 200 psi was selected (arbitrarily) to prevent any boil-off of the fluids in the core. In the high pressure experiments, the pressure while aging was maintained at the bubble point of the crude. (Except in one case where experiments were conducted elsewhere at reservoir conditions using live crude.) The use of stock tank oil in our experiments was justified as it was assumed that the dissolved gases (present in live crude aging) would not change the solubility behavior of the polar groups in the crude responsible for altering the wettability. (This should be further investigated, but is not the subject matter of this report.)

All tests were conducted at reservoir temperature. It was assumed that temperature plays an important role in wettability restoration, and hence no compromise on temperature was accepted.

Cores from two different reservoirs in the North Sea, referred to as H & V, were used for investigation. Cores were obtained in two batches from these reservoirs; referred as H1, H2 and V1, V2 respectively. Each test was done on 2-3 plugs to minimize the variability/errors in the results. Table 1 shows the petrophysical and fluid properties relevant to our experiments.

# EXPERIMENTAL DETAILS FOR LOW-PRESSURE AGING:

In the low pressure aging case, the core was saturated with the brine and the stock-tank crude to desired saturation levels in a Hassler type core holder where the flooding was done at low pressure but reservoir temperature. The core was then removed from the core holder and placed in a low pressure aging chamber. The core exposure to ambient conditions was minimized while transferring the core from the core holder to the aging chamber. The aging chamber consisted of a stainless-steel cylinder capable of withstanding 300 psi pressure. To prevent core contact with the stainless-steel cylinder walls, the core was first loaded in a cleaned glass tube. The glass tube was then filled with the crude and placed inside the steel chamber. The air from the chamber was purged using nitrogen. A pressure of 200 psi was maintained all through the aging for 30 days.

After aging, the core was removed from the chamber and mounted in the Hassler type of core holder. The temperature of the core holder was maintained at 130 F. The crude in the core was flushed out using Soltrol-130. About 200 pore volumes of Soltrol-130 was injected to ensure that all the crude oil has been removed from the core. Following this, the wettability of the core was measured using Amott test<sup>[10]</sup>. The cores were centrifuged for 1 hour at 2000 g. The natural imbibition cycle lasted for 24 hours.

Another assumption in the above core handling process was that the mineral oil does not alter the wettability of the core. Practical limitation in wettability measurement (e.g. Amott test) impose this restriction on us. Again, this assumption should be checked by further experimentation.

In spite of all the precautions taken to minimize the core exposure to the atmosphere, some temperature cycling (from high temperature in chamber to the ambient and then to the high temperature in the core holder) was inevitable in these type of experiments. This raises the question that the temperature cycling may have contributed to the change in the wettability. To test this hypothesis, additional tests were conducted where the core was saturated and aged in the same Hassler type core holder for one month. After aging, the core was flushed with Soltrol-130, and the wettability measured as described earlier.

These two types of low pressure experiments are identified by (a) low pressure aging in chamber and (b) low pressure aging in a core holder.

#### EXPERIMENTAL DETAILS FOR HIGH-PRESSURE AGING:

Ideally, one would like to age the core at the reservoir conditions of pressure and temperature. These experiments are tedious, time consuming, and require the use of recombined reservoir fluids. This severely limits the number of tests that can be conducted. Hence, all (but one) experiments discussed here were conducted using stock-tank oil. The pressure was maintained at bubble point of the crude. The inherent assumption in such an approach is that the presence of gas has little effect on the wettability restoration of the core. This assumption should, however, be tested by further laboratory experimentation. No experiments were done to test this hypothesis.

The one experiment where recombined reservoir crude was used, the aging was done at reservoir pressure and temperature.

The core was saturated with the brine and the stock tank oil to desired saturation levels in a core holder. The core holder was maintained at the reservoir temperature and the crude bubble point pressure. After saturating the core, the core was allowed to age for 30 days. Both the pressure and the temperature were maintained while aging. To prevent the gas from the overburden fluid from seeping into the core through the viton sleeve, the core was first wrapped in a Teflon-coated aluminum foil (McMaster-Carr Catalogue) prior to inserting it in the sleeve.

After aging, the core was thoroughly flushed with Soltrol-130 to remove the crude oil from the core. About 100 pore volumes of the Soltrol-130 were injected. Then, the system temperature and pressure were reduced to 130 F and 200 psi, in that order respectively. Following this, the core was removed from the core holder and its wettability was measured.

#### **RESULTS OF CASE HISTORIES:**

Many wettability restoration experiments were conducted on cores from the two North Sea reservoirs. Both were sandstone reservoirs. For identification purposes, the cores from the two batches are numbered as H1, H2 from reservoir H; and V1, V2 from reservoir V. Typical permeability and porosity data for these cores are given in Table 1.

Cores H1, H2 and V1 were drilled using oil-based muds. Cores V2 were drilled with water-base mud. Experiments on cores H1, H2 and V1 were conducted in our laboratory. Experiments on cores V2 were conducted elsewhere.

Table 2 lists the wettability and preservation status of the various cores. All wettability measurements were made using Amott method. The Amott index varies from '+1'to '-1', a positive index refers to a water-wet case and the negative index refers to an oil-wet case. (For reference, the Berea core Amott index was about +0.7.) Ideally, the Amott method consists of two indices, one for the water imbibition, and the other for the oil imbibition. In this report, only one index is used as the other index was found to be zero.

Cores H1 and V1 were not properly preserved, and could not be cleaned to a water-wet state. In both these cases, the cores could only be cleaned to a neutral-wet state. Cores H2 and V2 were cleaned to a water-wet state. (Details of cleaning sequences/procedures will be presented later). Proper core preservation technique that prevented any core drying, was thought to be the key that made it possible for some cores to be cleaned using solvents while others did not.

The low pressure experiments were conducted on cores H2 and V1. The H2 cores were water-wet prior to aging. However, V1 cores could only be cleaned to a neutral-wet state.

In case of high pressure aging, the core sets H2 and V1 were aged at reservoir temperature and bubble point pressure pertaining to the respective reservoirs. Respective bubble point pressures and temperatures for the two reservoirs are listed in Table 1. The wettability restoration for V2 cores was obtained by aging the cores at reservoir conditions using recombined reservoir crude. The pressure of the system was maintained at the reservoir (V) pressure of 3500 psi. The bubble point of the crude (reservoir V) was 2500 psi.

The results of various experiments are tabulated in Tables 2 through 4. Following observations are made about the results.

- 1. As shown in Table 2, we found that cleaning of poorly preserved core is very difficult. Cores H1 and V1, which were poorly preserved, could not be cleaned to a water-wet state. More of the cleaning process will be discussed later in detail.
- 2. As is evident from Table 3, aging in chamber was close to aging in a core holder where temperature is not allowed to drop. However, this is limited evidence, and may only be valid for certain cases.
- 3. The core aged at low pressures consistently gave more oil wet behavior than the cores aged at higher pressure, Table 4. This was true from the results of both reservoir cores. Hence, we conclude that maintaining pressure, as close to the reservoir conditions as possible, is important.
- 4. In case of reservoir V, where results were available for aging at reservoir condition using live crude (Table 5), the core was found to be slightly more water-wet after aging than prior to aging (but after cleaning). This could be due to (a) more solubilization of the contaminated surface of the rock by the oil at higher pressure of aging as compared to the bubble point of the crude (b) the existence of solution gas in the crude. Further experiments are required to evaluate the role of these two factors, namely the influence of dissolved gas and incremental pressure change on wettability in core restoration.

5. Drying of the core is probably the single most important cause of wettability alteration. Hence, it is extremely important to minimize core drying. Proper core preservation techniques are essential if wettability state of the core is to be preserved.

If left open in the air, the cores can dry out of water very quickly. Simple experiments were conducted in our laboratory where 100% water saturated core plugs (1 inch diameter and 1 inch long) were placed in a controlled environment at 72 F and 50% Relative Humidity. We found that in one hour more than 20% of the water evaporated from the core. The core dried out almost completely in about 24 hours.

6. Use fresh un-oxidized crude oil. Minimize the crude oxidation by storing it in epoxy-coated cylinders in a cool place. Infrared spectrometry technique of crude characterization, and other important parameters in crude oxidation are discussed in a later section.

#### CORE CLEANING PROCEDURE:

All cores were cleaned in a flow through system. The cores were mounted in a core holder. The temperature of the system was raised to 130 F. A back pressure of 200 psi was applied against which solvents were injected in the core. This pressure and temperature is maintained to prevent water boil-off from the core. It is generally believed that if water boils-off too quickly, the oil phase may contact the rock and change its wettability to more oil-wet. This was precisely the reason that Dean-Stark extraction was not used, because in Dean-Stark extraction water can boil-off quickly, permitting the oil phase to come in contact with the rock.

As it is not possible to know a priori which solvent is the most effective one, a sequence of solvents is used. Most frequently used solvents in our laboratory are Toluene, Isopropyl Alcohol, Chloroform, 111 Trichloroethane, Acetone, Pyridine, Carbon Tetrachloride and Methanol. Each solvent is circulated for 24 hours, at a rate of about 2 cc/min. A Dean-Stark distillation set-up was modified to enable us recirculate the solvent by distilling the effluent from the core. Whenever possible, azeotropic mixtures were used to save time. Table 6 lists some of the common azeotropes used in our cleaning system.

#### CORES H1 and H2 FROM RESERVOIR H:

All cores were oil wet as received (prior to cleaning.) Table 7 lists the type of the mud for coring and the state of the cores as received. Except for the cores V2, all the cores were drilled with oil-base muds.

The list of the solvents used for cleaning the cores from the two reservoirs is given in Table 8.

The first batch of the cores H1 from reservoir H, could not be cleaned to a water-wet state. These cores were poorly preserved, and no special preservation precaution was taken. It was concluded that the cores have gotten dried up before testing. If cores are exposed to ambient conditions even for a short time, they may dry out, and thus allow the oil phase to come in contact with the rock.

A second batch of cores, H2, was received where extra precaution was taken to preserve the cores

and they were not allowed to dry. Same solvents as used for cores H1 were used. We were able to clean these cores much better. The Amott wetting indices for the core prior to cleaning and after cleaning are given in Table 2.

#### CORES V1 and V2 FROM RESERVOIR V:

The cores V1 could not be cleaned to a water-wet state. These cores were also drilled using oil-based mud and were not properly preserved. In spite of extensive cleaning effort using all the solvents listed in Table 8, these cores could not be cleaned to a desired water-wet state. Again, each solvent was injected for a period of 24 hours. The core were found to be neutral-wet after cleaning.

In absence of properly preserved cores at the time, the neutral-wet cleaned cores were used for aging tests.

The second batch of cores, V2, was drilled using water-base mud. These cores were properly preserved, and could be cleaned to a water-wet state. These cores were aged using live crude oil and at reservoir conditions.

#### **RESULTS AND DISCUSSION:**

It can be concluded that the proper preservation is very essential in minimizing damage to core wettability. Core drying seems to be the dominant mechanism of altering the core wettability.

It should, however, be pointed out that in some cases core preservation may not alter the wettability permanently. The crude characteristics play an important role in core damage. In another study of a heavy oil reservoir, we were able to clean cores that were 10 years old and were very poorly preserved.

#### CHARACTERIZATION OF CRUDE FRESHNESS OR OXIDATION:

In our laboratory, we characterize each crude oil based on its oxidation number. Immediately after receiving the fresh crude oil from the field, the oxidation number is measured. In future, this number is used as reference to check the freshness of the oil.

Infrared spectrometry is used for measuring oxidation numbers of the crude. These numbers are measure of OH groups in the crude. All spectra are obtained on our Nicolet MX-1 FTIR spectrometer averaging 32 scans at 2 cm<sup>(-1)</sup> resolution. The infrared spectrum (4000-400 cm<sup>(-1)</sup>) is obtained for the oil using a liquid cell with a known path length (typically 0.1 mm). The net absorbance of the acid C=0 band is used as an indicator of the level of oxidation - greater the absorbance, higher the level of oxidation. The net absorbance is calculated by drawing a base line across the shoulders of the absorbance band, reading the absorbance at the peak maximum, and subtracting the baseline absorbance at the same frequency. The oxidation values are reported as net absorbance per 1 cm path length.

The oxidation values reported represent relative levels of oxidation rather than an absolute concentration of acids. A value of 2 can be considered twice the level of oxidation as a value of 1.

We find that oxidation numbers are better than acid numbers for crude characterization. Oxidation numbers are more sensitive in detecting formation of oxidation products that are wettability altering. Figures 1 and 2 compare the oxidation numbers with two types of acid numbers (pH = 7 and pH = 11) for several crudes. Even though the crude oil oxidation numbers change significantly, their acid numbers exhibit small differences. The low Ph acid numbers are particularly insensitive.

We conducted further experiments to examine the process of crude oxidation. We wanted to know the impact on crude oxidation of (a) system temperature (b) presence of the brine and (c) presence of 316 stainless-steel in the system.

Fresh crudes from two reservoirs V & C were used for these experiments. API gravities of the two crudes were 20 and 50. All experiments were conducted at ambient pressure.

To determine the role of temperature, the oxidation numbers of the two crudes were recorded at two temperatures; 76 F and 120 F. The crude oils were placed in a room on magnetic-heat stirrers in thoroughly cleaned glass beakers. The temperatures of the containers were set to desired levels. The oils were stirred continuously to promote oxidation by good mixing with the ambient air. The oxidation numbers of the crudes were measured regularly at one week interval for about 90 days.

Figure 3 and 4 show the results of oxidation of the V and C crudes at the two temperatures. Both crudes exhibit similar behavior. As expected, the oxidation of the crudes is far more pronounced at elevated temperature (120 F) than at room temperature (70° F).

To determine the sensitivity of the crude oxidation by brine and 316 stainless-steel, we conducted one experiment using V crude only. This experiment was conducted at 120 F. The V crude was poured in three glass beakers. As before, these beakers were placed on magnetic-heat stirrers. We added some 316 stainless-steel cuttings to the first beaker, brine to the second beaker and a combination of 316 stainless-steel & brine to the third beaker. Again, the crudes were stirred continuously and the oxidation numbers were recorded regularly.

Figure 5 compares the rate of oxidation under different conditions. The rate of oxidation is significantly higher when brine and 316 stainless-steel cuttings were present together.

We recommend that to prevent further crude oxidation, the oil should be stored in an epoxy coated container in a cool place under methane or nitrogen blanket. The oxidation process is accelerated at high temperature and in presence of certain metals. For example, exposure of oil to air in presence of chromium, copper, cobalt and nickel, is known to accelerate the oxidation process. If experimental conditions dictate that the oil be stored in stainless steel containers at high temperature, make sure that these containers are purged with nitrogen first. Stainless steels contain a large amount of chromium which could become a catalyst in oxidizing the crude.

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TABLE 1

PETROPHYSICAL PROPERTIES OF THE CORES USED IN OUR EXPERIMENTS

Reservoir Type	Core No.	Reservoir Pressure, Temp. & Bubble Point	Porosity (%)	Permeability (md)
Sandstone (H)	Н1 & Н2	2000 psi 130° F 800 psi	23 to 26	2000 - 4000
Sandstone (V)	V1 & V2	3500 psi 260° F 2500 psi	18 to 22	200 - 300

# **BRINE PROPERTIES**

(Brine properties at ambient conditions)

Reservoir	Fluid Type	Density gm/cc	Viscosity cp	Acid Number
Н	Brine (Synthetic)	1.04	1.1	5.9
V	Brine (Synthetic)	1.006	11	6.3

# **CRUDE PROPERTIES**

(Crude properties at high pressure test conditions)

Reservoir	Fluid Type	<u>Test</u> Press. (psi)	Conditions Temp. (F)	Density gm/cc	<u>Viscosity</u> cp
H2	Crude (Stock Tank)	800	130	.926	117
V1	Crude (Stock Tank)	2500	260	••••	0.9
V2	Recombined	3500	260		

TABLE 2

	Cleaning	Cycle	Effectiv	eness
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Core No.	Quality of Preservation	Mud System	Wettability Before Cleaning	(Amott Index) After Cleaning
H1	Poor	Oil-based	8	0
H2	Good	Oil-based	8	+.8
V1	Poor	Oil-based	3	0
V2	Good	Water-based	N/A	+.4

TABLE 3

# Wettability after Low Pressure Aging

		Wettability after	Low Pressure Aging
Core No.	Wettability Prior to Aging	Aging in Chamber	Aging in Core Holder
H2	+0.8	4 to45	3 to4

# TABLE 4

# Wettability Comparison: Low Pressure vs High Pressure Aging

		Wettability	after Aging
Core No.	Wettability Prior to Aging	Aging at Low Pressure	Aging at High Pressure
H2	+0.8	3 to4	+.25 to +.3
V1	0	3	0

# TABLE 5

# Wettability Comparison: Stock Tank Oil vs Live Crude

		wettability at at High Pr	
Core No.	Wettability Prior to Aging	Aging using Stock Tank Oil	Aging using Live Crude
V2	+0.4	Not Available	+.5

TABLE 6
Common Azeotropes Used in Cleaning Process

Group	Solvents	Boiling Point (F)	Mixture Boiling Point (F)	Ratio (%) by Volume
1	(a) Iso-propyl Alcohol (b) Toluene	82.3 110.6	80.6	58 42
2	<ul><li>(a) Acetone</li><li>(b) Chloroform</li><li>(c) Methanol</li></ul>	56.2 61.2 64.7	57.5	30 47 23

TABLE 7
State of the field cores and crude oils tested in our laboratory

Field Name	Core Set	Type of Mud	Preservation Status	State of Crude Oil
Н	H1 H2	Oil-based Oil-based	Poor Good	Uncontaminated Uncontaminated
V	V1 V2	Oil-based Oil-based	Poor Good	Uncontaminated Uncontaminated

TABLE 8

Type of solvents used for cleaning the various field cores

Field Name	Solvent Sequence
Н	Toluene, Methanol
V	Toluene, Isopropyl Alcohol, Chloroform, 111 Trichloroethane, Acetone, Methanol

Fig. 1. Oxidation Number vs. Acid Number (Acid No. at two ph values; ph = 7 & 11)

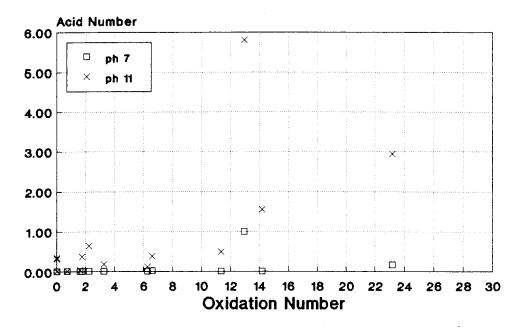
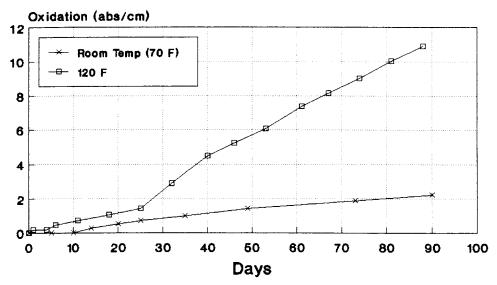
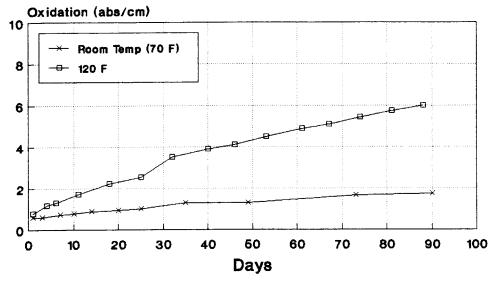


Fig. 2. Reservoir "C" Crude Oxidation at Two Temperatures (70 F and 120 F)



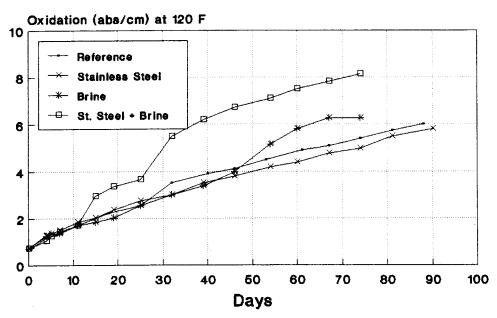
Net Abs Acid C=O, 1 cm cell pathlength

Fig. 3. Reservoir "V" Crude
Oxidation at Two Temperatures
(70 F and 120 F)



Net Abs Acid C=O, 1 cm cell pathlength

Fig. 4. Reservoir "V" Crude Oxidation Effects of St. Steel & Brine



Net Abs Acid C=O, 1 cm cell pathlength