## TECHNIQUES TO MEASURE CORROSIVITY OF DRILLING FLUIDS ASSOCIATED WITH DEEP DRILLING USING A FLOW LOOP

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الخلاصة :

إحدى المشاكل المصاحبة لحفر وتجهيز البئر العميقة هي تأكل المواد المكونة لها . وإنَّ توفر واحد أو أكثر من عوامل التأكل مثل كبريتيد الهيدروجين وثاني اكسيد الكربون والاكسجين مع درجة حرارة مرتفعة يزيد باطراد خاصية تأكل الطُفل ( الطين ) مما يترتب عليه تلف في المكونات الحديدية ويؤدى الى خسارة كل البئر . التأكل ليس فقط مشكلة رئيسية في الحفر وحسب ولكن ايضا في الانتاج ، وطرق تحسينه ، ومصافي التكرير .

وهدف هذا البحث هو تقديم طريقة جديدة لقياس معامل التأكل في حالات مختلفة من الانسياب عند درجات حرارة عالية . وقد أجريت الاختبارات فى المدى مابين درجة حرارة الجو المحيط ودرجة خسبائة فهرنهايتية باستخدام الطرق الآتية : (١) استاتيكية (٢) دورانية (٣) ديناميكية . وقياسات الطريقة الديناميكية تمت بواسطة (أ) جهاز التدفق العروى الذى يماثل دورة كاملة لطين الحفرة (ب) خلية التأكل للتدفق الخطي لقياس معامل التأكل في حالة التدفق الخطي (ج) خلية التأكل المغناطيسية ( اختراع للمؤلف ) لقياس معامل التأكل في حالة الانسياب الدوراني والخطي معا التأكل المغناطيسية ( اختراع للمؤلف ) لقياس معامل التأكل في حالة الانسياب الدوراني والخطي معا عاكيا التدفق في الحيز الحلقي بين انبوب التغليف وانبوب الحفر . واستخدمت طريقة نقص الوزن لحساب معامل تأكل رقائق الحديد المعتدل ( ١٠١٨ ) بمقايس ٥,٩ × ٥,٠ + ٢٢,٠ بوصة . صمم جهاز التدفق العروى ليسمح لتثبيت السائل حراريا حيث يمكن قياس معامل التأكل اعلى وتضمن البحث مقارنة بين كل الطرق المستعملة تحت حالات مختلفة وظهر أنً معامل التأكل اعلى وتضمن المحد في الالف من البوصة لكل عام عندما تستخدم الجلية المناطيسية عنها في الحلي المار ب ٢, ٥٩ واحد في الالف من البوصة لكل عام عندما تستخدم الحلية المناطيسية عنها في الحلية . والطريقة الاستاتيكية تكون ١٠ واحد في الالف من البوصة لكل عام أقل من الحالة الدورانية لمينة اخرى

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#### ABSTRACT

One of the major problems associated with drilling and completing a deep well is corrosion of structural materials. Corrosive agents such as  $H_2S$ ,  $CO_2$ , and  $O_2$  at high temperatures can dramatically increase the corrosivity of the mud system, with severe damage to all steel components which may ultimately damage the whole well. Corrosion is also a major problem in production, EOR techniques, and even in refinery plants.

Tests were conducted between ambient temperature and 500°F. The corrosion rates have been measured in this study using the following techniques: (1) static; (2) rolling; and (3) dynamic. The measurement through dynamic technique is done by using: (a) the flow loop which simulates a complete cycle of the drilling fluid; (b) linear corrosion cell to measure the corrosion rate of a mud while it is flowing in a linear pattern; (c) mag-corrosion cell (patented by the author) to measure corrosion rate at linear and rotational flow combined together simulating the flow in the annulus. Weight loss method has been used to calculate the corrosion rate for mild steel (1018) coupons of size 2.5 in  $\times 0.5$  in  $\times \frac{1}{16}$  in.

A flow loop used as a compact system has been constructed to stabilize the fluid thermally at the test temperature in order to measure the corrosion rate.

Comparison between all the techniques under different conditions are included. Corrosion rate is 95.7 mpy higher when using the mag-corrosion cell than that of the linear pattern. Static technique rate is 10 mpy less than rolling for another sample.

## TECHNIQUES TO MEASURE CORROSIVITY OF DRILLING FLUIDS ASSOCIATED WITH DEEP DRILLING USING FLOW LOOP

#### **INTRODUCTION**

Corrosion problems are now recognized by a larger segment of the oil industry than ever before because of complicated problems in very deep and geothermal wells. Corrosion is an electrochemical and surface phenomenon which requires the presence of moisture and some corrosive agent. Mud workover completion fluids, or packer fluids, which are chemically strong electrolytes, conduct electric current and set up a potential exchange between the anode and cathode on the drill pipe, drill collars, and casing. Acid-forming gases such as carbon dioxide and hydrogen sulfide are serious environmental corrosion accelerators that must be dealt with in drilling fluids. Acid gas contamination can result from drilling fluid materials that have been altered by temperature [1, 2], microbiological activity or electrochemical effects [3]. Serious breakdown of many commonly used organic materials containing carboxyl or sulfur groups into carbon dioxide or hydrogen sulfide begins at approximately 150°C (300°F). Thermally stable materials should thus be used when well temperatures are expected to exceed the 300°F range for extended periods because thermal degradation tends to destroy drilling fluid properties.

The corrosion rate can be monitored over time using weight loss or in real time using an electronic probe system; and treatment should start promptly to correct or avoid undesirable trends in the target before extensive damage occurs. This report introduces techniques for the accurate monitoring of corrosion rates in simulated conditions. This allows the selection and evaluation of appropriate corrosion inhibitors.

# STATEMENT OF THE PROBLEM AND OBJECTIVES

Thousands of corrosion tests are conducted every year to reduce the cost of treating corrosion. The value and reliability of the data obtained depend on details involved in the test. Controlling and manipulating all the factors affecting corrosion in a particular plant or field operation is very difficult. Simulating actual drilling operations in a laboratory test is of considerable importance in obtaining reliable and reproducible results. Therefore, the main objective of this study is to design an adequate and efficient experimental set-up to measure the corrosion rate by the weight loss method under field conditions. Such apparatus should first examine the thermal stability of the mud formulations at higher temperatures and pressures, due to the fact that conventional drilling fluids suffer thermal degradation and possess a high tendency to gelation, both of which can affect the performance of the drilling fluids in drilling operations.

Since it is not possible to simulate the precise field conditions of temperature, pressure, fluid rotation, drill string rotation, shearing, fluid contamination, and depth scaling, two important techniques, linear (flow loop) and rotational (rolling), have been developed. The reliability of the data obtained using these methods is dependent on the following parameters: (a) The degree of stabilization of the testing fluids; (b) the volume of the test solution; (c) the exposure time of the specimen to these fluids under the test conditions; (d) the test temperature and its control at the hot section and the efficiency of the cooling system at the cold section; (e) the acidity of the test fluid and its control during the exposure period; (f) the nature of the mud components and their percentages; (g) the conditions of immersion of the specimen inside the cell; (h) the surface area exposed in the bulk; and (k) the heterogeneity of the test fluid and the solid contents.

These parameters have been studied and controlled as completely as possible to measure accuately the corrosivity and characteristics of the mud formulations under various conditions by the different techniques.

#### Forms of Corrosion

The four major types of corrosion are:

- 1. Uniform corrosion, *i.e.*, the attack is uniformly distributed on the coupon surface.
- 2. Local corrosion, *i.e.*, corroded spots on the coupon surface.
- 3. Pitting corrosion, *i.e.*, the attack is concentrated on a very small area of the coupon surface.
- 4. Intercrystalline corrosion, *i.e.*, the attack is concentrated on the grain boundaries.

#### **EXPERIMENTAL DESIGN**

Tests were made under simulated bottom hole conditions by two different techniques. The first was a linear flow technique using two flow loops. The second was a rotation or rolling technique using a Baroid rolling oven. In a successive trial, the two approaches were combined by designing a new cell (mag-corrosion) in which the fluid flowed linearly in the flow loop and rotated around the coupons with variable controlled speed of rotation. A static technique was also used.

#### A. Flow Loop Corrosion Tests

1. Sandia-Lab Flow Loop: the entire loop contains about 260 feet of <sup>1</sup>/<sub>4</sub> in O.D. and 0.035 in thick stainless steel tubing. The construction, measuring, and recording systems are discussed in detail in reference [5].

2.  $PGE^{t}$  Flow Loop: the entire system was designed and constructed to simulate a drilling fluid circula-

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tion on the rig [6]. It consists of the following sections, as shown in Figure 1.

(a) The Pumping Section. The pumping system consists of a speed-controlled-motorized feed pump, passing the mud to the big single-positive pump. The two pumps cooperate to circulate the fluid regularly in the system with the assistance of by-pass lines to relieve the back pressure created at the inlet of the big pump. A Magcobar shear valve is incorporated on the loop to serve as a pressure regulator and to shear the mud. The pressure and flow rate are adjustable to obtain a uniform velocity in the flow lines. In one cycle, the temperature is increased gradually to the required test temperature; the fluid is sheared through the shear valve and cooled back gradually to the surface condition to start a new cycle.

(b) The Flow Lines Heating/Cooling Section. The flow lines consist seven lines of  $\frac{3}{8}$  in O.D. seamless stainless steel connected in series with stainless steel swage-lock fittings. The cooling lines are of  $\frac{1}{4}$  in O.D. seamless stainless steel. These tubes are bent in seven lines of 1 foot length and immersed in a



Figure 1. Schematic Diagram of the Dynamic Corrosion Test: PGE Flow Loop.

thermally-controlled bath filled with ice. The heaters are attached to the  $\frac{3}{8}$  in tubes and cemented in four inch O.D. and 1.5 in O.D. fiberglass insulating tubes to minimize the heat loss from the system. Iron-constantan thermocouples are inserted at different spots on the loop to measure the temperature of the system. The hot section is thermally controlled by temperature controllers to maintain a constant temperature. The temperature readings are recorded on ten inch strip charts by a multipoint temperature recorder.

(c) The Controllers and Recording Section. The temperature is measured simultaneously at various points by temperature sensors and recorded by 250-series L&C recorders. The pressure in the system is measured by a pressure transducer system. These differential transducers (Model DP-15 $\pm$ 50) are connected with the Validyne (MC1-10) case which has ten pressure measuring channels. DP-18 and DP-212 indicators plugged into the MC1-10 transfer the hydraulic pressure signals to pressure readings. These pressure readings from different spots on the loop are recorded simultaneously on a multipoint ten inch strip recorder.

(d) The Corrosion Cells. Two different corrosion cells are used. The first is 9.0 in long, 1.0 in O.D. stainless steel tubing. Both ends are open and can be closed by 1.0 in swage-lock fittings after inserting the specimen carrier. The other cell, which can be fixed at either the hot or cold end, is called a magcorrosion cell (Figure 2). It is a 316-stainless steel heavy duty cell that can work at very high temperature and pressure. The cell has inlet and outlet ports plus a two-way ball valve. The coupon holder is installed in the stainless steel housing at the bottom of the cap. The specimens are completely isolated from the metal to eliminate the flow of electrolytic current between the coupons and the cell body, which may affect the corrosion measurements. The mag-corrosion cell is seated on a magnetic stirrer to rotate the fluid in the cell with controlled speed. This technique is designed to simulate the mud rotation under the effect of drill bit rotation and bottom hole conditions.

#### **B.** Rolling Corrosion Tests

The corrosion rate was determined by weight loss measurements under conditions in which the fluid and the coupons were rotating. Different formulations of the drilling fluids were tested at tempera-



Figure 2. Mag-Corrosion Cell.

tures from 200°F to 500°F in 100°F increments. A Baroid rolling oven, thermally controlled with a temperature range from room temperature to 550°F, was used. It has only one rolling speed.

The stainless steel rolling cells were designed to hold the coupon carrier rigidly. There was no metal contact between the coupon and any metal surface during the run. Measuring corrosion under bottom hole conditions by rolling simulates as closely as possible the rotational motion of the mud and the drill string.

#### C. State Corrosion Tests

The static technique was used to measure corrosion under a stationary condition. The cells used were the same as those used in the rolling tests.

#### **EXPERIMENTAL PROCEDURE**

The experimental study was divided into three parts.

#### A. Mud Formulation and Preparation

Different batches of commercial and geothermal water-base mud were prepared. The percentage of each component was varied to meet a specific desirable characteristic in the formulated drilling fluid. The fluid was mixed in the laboratory using a Magcobar multimixer for a period of time long enough to assure clay hydration and uniform distribution of each component in the suspension.

Rheological properties of the mud were measured just prior to each test, either dynamically or statically, with a Baroid Fann V. G. Rheometer.

### B. Stability Tests using the Flow Loop

Stability test measured the ability of the test fluid to withstand severe conditions of temperature and pressure. The stability was determined by measuring the pressure differential across the test section every 15 minutes. If the readings were very close for a period of time under the test conditions, the system was considered stable. System failure was defined as occurring when the reading 6 to 10 times that at room temperature. The system was then shut off and the run terminated. The mud composition was resinex, bentonite, sepiolite, sea salt, and caustic soda  $(X_{1c})$ .

## C. Corrosion Tests

The method described in this study was intended primarily to monitor the corrosion rates for a group of drilling fluids formulated for geothermals and deep wells having temperatures higher than 400°F. Rectangular flat shape coupons were chosen to suit the needs of the test purposes according to NACE and ASTM standards. The specimen holder was oriented in a vertical position; the distance between the two coupons was sufficient to permit good contact between the electrolyte and the coupon surface.

The test duration in the flow loop was limited and dependent on the system efficiency. Corrosion rate data were obtained from the flow loop by exposing the specimen to the medium and changing the temperature from 100°F up to 480°F. The time of exposure included the time taken to cool the system by flushing it with water until room temperature was again reached.

In the rolling technique, the specimen used was taken out after each run at one temperature and cleaned, reweighed, and put back in the cell ready for the new test at a higher temperature. The temperatures used were 200, 300, 400, 500°F and the test duration ranged from 20 to 24 hours. Thus, the corrosion rates measured by the rolling technique were obtained at each temperature using two corrosion cells for each fluid.

In the static technique, the cells were loaded with the test fluid and the specimen, then left for two weeks at a fixed temperature.

The zeroing, spanning, and calibration of the transducers and the pressure measurements are discussed in detail in reference [6].

## **RESULTS AND DISCUSSION**

In the case of deep drilling and geothermals, high temperatures combined with shear at the bottom hole create gelation and flocculation problems, especially in very small diameter flow lines. Thermally stable fluids which can withstand the bottom hole conditions have been formulated. Sepiolite, which is extremely stable at high temperatures, added to the test fluid improves commercial mud.

Two types of muds were developed with the combination of clay (bentonite), sepiolite, and resinex. The difference between them was the differing amounts of caustic soda (NaOH) and sea salt additives. The liquidity of the drilling fluids in addition to the physical properties measured from the flow loops and the rolling test indicates improvement; i.e. the stability of the muds was improved for a longer time which, in turn, makes it possible to obtain more reliable corrosion results because the exposure time of the coupons is dependent upon the fluid thermal stability and the system efficiency.

The results obtained from testing mud  $X_{1c}$  are tabulated in Tables 1, 2, 3, and 4 using the flow loop and rolling techniques. They are plotted in bar graphs shown in Figures 3, 4, and 5. The test results indicate the following.

- 1. The corrosion rate at the hot end is much higher than that at the cold end in the flow loop, e.g. 253 mpy and 155 mpy respectively as given in Figure 3. This shows that the temperature effect is significant and can accelerate the corrosion rate.
- 2. The corrosion rate is reduced when the fluid alkalinity (pH) is increased from 9.81 to 11. This may be due to the added hydroxyl group in NaOH, which might increase the polarization of the metal surface. However, the hydrogen ion content increases with the temperature as shown in Figure 6. Consequently, retaining high pH is required.
- 3. The corrosion rate in the case in which the

		Table 1	1. Corrosion	Test: S	Sandia-Lab	Flow Loop,	Tap Water	r, 24 Hours	Aging	
Loop End	Specimen	pН	Mud Weight	Area	Coupon Weight Before	Coupon Weight After	Weight Loss	Time of Exposure	Corrosion Rate	Average Corrosion Rate
			ppb	in <sup>2</sup>	g	g	g	h	mpy	mpy
Hot	LH-1	9.81	511	2.53	9.6896	9.6606	0.0285	3	256.6	253.4
Hot	LH-1	9.81	511	2.53	9.8426	9.7646	0.0278	3	250.3	
Cold	LC-1	9.81	511	2.53	9.7565	9.7386	0.0179	3	161.1	155.7
Cold	LC-2	9.81	511	2.53	9.7976	9.7816	0.0167	3	150.3	
Test Conditions :		Temp	erature	400°F	450°F					
		Expos	sure time	2 h	1 h					
		Cold	end	11 <b>0°F</b>	3 h	(Total ex	xposure tim	e at the cold	i end)	

3 h (Total exposure time at the cold end)

Remarks: (a) Corrosion rate at the hot end is much higher than at the cold end.

(b) Corrosivity of this fluid is severe at high temperature.

Recommendation : Drill pipes should be replaced at least every year.

#### Table 2. Corrosion Test: Rolling Test, 24 Hours Aging Time

Cell	Specimen	pН	Mud Weight ppb	Area in <sup>2</sup>	Coupon Weight Before g	Coupon Weight After g	Weight Loss mg	Time of Exposure h	Corrosion Rate mpy	Average Corrosion Rate mpy
A	R <sub>1</sub>	9.81	511	2.53	9.8216	9.8067	14.9	2.67	150.7	
Α	R <sub>2</sub>	9.81	511	2.53	9.7983	9.7820	16.3	2.67	164.9	157.8

Mud aged for 24 hours. Oven temperature raised directly to a temperature between 400°F and 450°F.

#### Table 3. Corrosion Test: Flow Loop, Distilled Water, No Aging.

Loop End	Specimen	рН	Mud Weight ppb	Area in <sup>2</sup>	Coupon Weight Before g	Coupon Weight After g	Weight Loss mg	Time of Exposure h	Corrosion Rate mpy	Average Corrosion Rate mpy
Hot	LH-1	11	510	2.579	9.7326	9.7218	10.8	2.67	107.2	110.6
Hot	LH-2	11	510	2.579	9.6589	9.6474	11.5	2.67	114.1	
Cold	LC-1	11	510	2.579	9.6964	9.6946	1.8	2.67	17.9	
Cold	LC-2	11	510	2.579	9.7056	9.7032	2.4	2.67	23.8	20.8

Further Information : (a) Temperature °F: 400 300 400 480

> (b) Time, min: 35 40

(c) Cold End Temperature °F: 110

(d) Steam leak at two connections in the test section, system was shut off (e) Distilled water

45

40

	Table 4. Corrosion Test: Rolling.									
Cell	Specimen	рН	Mud Weight ppb	Area in <sup>2</sup>	Coupon Weight Before g	Coupon Weight After g	Weight Loss mg	Time of Exposure h	Corrosion Rate mpy	Average Corrosion Rate mpy
Ā	R,	11	511	2.53	9.6435	9.6420	1.5	2.67	14.8	15.0
Α	$\mathbf{R}_{2}$	10	511	2.53	9.6575	9.6558	1.7	2.67	16.8	15,8

Distilled Water; Fresh Sample; Temperature increased gradually.



Figure 3. Bar Graph Showing Corrosion Rate vs Temperature (Tap Water, 24 Hours Aging and Mode of Change).



Figure 4. Bar Graph Showing Corrosion Rate vs Temperature (Distilled Water, Fresh Fluid, and Gradual Change in Temperature).

specimen rotates with the fluid at higher temperature is lower than that of the hot end of the flow loop. This is because the erosion effect in the linear pattern is higher than that in the rolling whereas it is about equal to that of the



Figure 5. Bar Graph showing Correlation Between Techniques (Flow Loop and Rolling) and pH.



Figure 6. Effect of Temperature on pH for Drilling Fluid.

cold end because the temperature effect was absent in the flow loop test.

A comparison between the corrosion rates measured by the PGE and Sandia-Lab flop loop for the same drilling fluid is shown in Figure 7. This difference is actually due to lack of a full control of temperature at some points of the hot section in the PGE loop and the attachment of the mag-corrosion cell on the PGE flow loop.

The rolling technique results shown in Table 5 are compared with data obtained from the two loops; linear and rotational patterns. The corrosion rate of

Average

Corrosion

Rate

mpy

14.9



Figure 7. Bar Graph Showing Equal Efficiency of PGE and Sandia-Flow Loop in Measuring the Corrosion Rate.

the rolling technique of 14.8 mpy is less than 26.1 mpy and 21.8 mpy for the hot ends of the two loops (PGE and Sandia-Lab). These results indicate that the rotation and linear flow of the drilling fluid around a stationary specimen is more severe than that of the rotating fluid and the specimen. In other words, the abrasivity of the fluid is higher in the case of the stationary coupons.

Figure 8 shows the corrosion rates for the three techniques: flow loop, rolling, and static. The temperature effect is shown in the hot and cold end which resulted in 6.2 mpy difference i.e. the corrosion rate is 20 mpy at the hot end and 13.8 mpy at the cold end. The rolling corrosion rate is 13.8 mpy which is about 10 mpy higher than the static under the rolling effect. Rotating the fluid around the coupons permitted new contact with agitated fluid while in the static technique the fluid remains stationary so that the chance for a new contact is nil. This explains the fact that during drilling the corrosion rate is much higher than when the drilling



Figure 8. Bar Graph to Show the Relationship Between Corrosion Techniques (Flow Loop, Rolling, and Static).

is stopped for tripping or some other related problems.

Different amounts of sea salt and caustic soda were used. Here only one group will be discussed. The mud contains 1% by weight, sea salt and the same percentage of caustic soda. The corrosion test results for (rolling) and the pH value with temperature are plotted in Figure 9. The point of intersection gives the critical pH value of 9.3 at temperature 190°F to maintain the corrosion rate at 19 mpy.

#### CONCLUSIONS

1. The corrosion rate varies considerably with the technique used for one drilling fluid. The corrosion rate measured by the rolling technique is always lower than that measured in the



Figure 9. Corrosion Rate (Rolling) and pH vs Temperature.

hot end of the flow loop at the same temperature limit. However, it is about equal to that of the cold end of the flow loop.

- 2. The corrosion rate of the mud increases as the medium temperature increases if the pH is low and decreases as pH increases, even at high temperature.
- 3. Combining rotational motion with linear motion in one cell (mag-corrosion) increases the corrosion rate to more than at the hot end using the rolling technique.
- 4. The static technique gives the lowest corrosion value because in this technique a number of factors are eliminated.
- 5. The stability and rheological properties of the drilling fluids are improved by combining sepiolite with bentonite m one batch.

#### FORMULAE USED

1. The corrosion rate in mils per year is calculated by the following formula:

	Weight loss, mg
mpy –	Metal Density × 16.387 × Area × Time

The mild steel coupons used in this study have a density equal to 7.86 g cm<sup>-3</sup>; thus this formula may be reduced to:

 $mpy = \frac{68.33 \times Weight \ Loss, \ mg}{Area \ (in^2) \times Hours \ of \ Exposure}$ 

2. The following are the conversion rates between the various units for steel coupons (specific gravity, 7.86):

 $mpy = 25.62 \times lb/ft^{2}/h$  $mpy = 5.03 \times kg/m^{2}/y$  $lb/ft^{2}/y = 0.04 \times mpy$  $lb/ft^{2}/y = 0.20 \times kg/m_{2}/h$  $kg/m^{2}/h = 0.20 \times mpy$  $kg/m^{2}/h = 4.90 \times lb/ft_{2}/y$ 

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