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GEOHERMAL FRACTURE STIMULATION TECHNOLOGY

Volume 3. Geothermal Fracture Fluids

By
Maurer Engineering

January 1981

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Republic Geothermal, Incorporated
Santa Fe Springs, California

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GEOHERMAL FRACTURE STIMULATION TECHNOLOGY

VOLUME III

GEOHERMAL FRACTURE FLUIDS

January 1981

Prepared for

U.S. Department of Energy

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GEOHERMAL RESERVOIR WELL STIMULATION PROGRAM

By

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I. SUMMARY AND CONCLUSIONS

Volume III of the Geothermal Fracture Stimulation series presents a detailed study of all available and experimental frac fluid systems. They have been examined and tested for physical properties that are important in the stimulation of hot water geothermal wells. These fluids consist of water-based systems containing high molecular weight polymers in the uncrosslinked and crosslinked state.

This report summarizes the results of fluid testing for many systems specifically at geothermal conditions or until breakdown occurs. Some of the standard tests are ambient viscosity, static aging, high temperature viscosity, fluid-loss testing, and falling ball viscosity at elevated temperatures and pressures.

Results of these tests show that unalterable breakdown of the polymer solutions begins above 300°F. This continues at higher temperatures with time even if stabilizers or other high temperature additives are included. The addition of more polymers in the base gel starts the solution at a higher viscosity level but may increase the possibility of damage to the formation from the degraded polymer. Also, these fluids may be chemically incompatible with the geothermal brines. Physical properties of these fluids are quite complex since they depend on temperature, time, shear rate, shear history and polymer concentrations.

Fluid-loss tests show that most of the common fluid-loss materials are effective at geothermal temperatures but some are more desirable than others. For example, calcium carbonate and silica flour are both good fluid-loss materials. However, the resulting silicate from the silica flour dissolution is much more difficult to handle than a carbonate scale. Hence, finely ground calcium carbonate is the desired fluid-loss additive. A good point is that each material acts as a

fluid-loss agent only until it dissolves; and then it will be removed from the pores and cracks of the formation by dissolution.

Crosslinked fluid systems are recommended by service companies to handle the hottest and most severe applications of fracturing and acidizing. These fluids are difficult to test on an unmodified viscometer, and their true worth for suspending sand could be seen only by our tests in a high temperature falling ball viscometer where the crosslinked fluids retained their sand-carrying properties to over 400°F.

The conclusion reached from these results is that today's polymer fluids are effective in geothermal wells only up to 350°F or 400°F. Above this temperature range, a different stimulation approach or other techniques should be considered and new technical developments in fluid systems are required.

II. INTRODUCTION

The fluids used in stimulation play many important roles. Specifically, for geothermal well stimulation, fluids initiate the fracture and carry chemicals, surfactants, inhibitors, acids, and proppant into the fractured formation. In hydraulic fracturing the width-to-length ratio and leakoff velocity are functions of the fluid's effective viscosity.

Historically, a wide variety of fluids has been used as fracturing fluids. Since we are dealing with a hydrothermal reservoir, we will only consider compatible water-based fluid systems. At this point, a frac fluid system should be defined as the base fluid, any fluid-loss additives, chemicals or surfactants, and the proppant. In the past 25 years gelling agents (polymers) have been added to viscosify frac fluids for oil and gas wells. The gelling agents are usually described as polymeric hydrophilic chemicals, or polymers for short. The large molecular size of the polymers allows them to increase the effective viscosity of water even at low concentrations.

The most common classes of polymers used for fracturing fluids are guar, guar derivatives, cellulose derivatives, and polyacrylamides. The approximate structure of each of the repeating units is shown in Figures 1 through 4. The guar is a polysaccharide and the guar derivative is usually a hydroxypropylguar. Both have similar properties of low cost and good fluid-loss control, and the guar derivative appears to have better temperature stability. The cellulose derivative is used frequently where no degradation products can be tolerated. Its structure is shown in Figure 3, and Figure 5 compares the degradation products of guar (left), guar derivative (middle), and the cellulose derivative. The approximate percentage of degradation products are 8 to 10 percent, 1 to 2 percent, and residue free, respectively.

REPEATING UNIT FOR GUAR DERIVATIVE
R=H OR HYDROXYALKYL GROUP

$n \approx 400-600$

Figure 1

REPEATING UNIT FOR GUAR POLYMER

$n \approx 400-600$

Figure 2

REPEATING UNIT FOR CELLULOSE
DERIVATIVE R=H,
HYDROXYALKYL, OR CARBOXYMETHYL
 $n \approx 3-4M$

Figure 3

Figure 4

Figure 5

Degradation Products of Polymer Solutions

The polyacrylamides shown in Figure 4 are not as widely used since they are more expensive and are strongly affected by high salt concentrations.

If a polymer solution has more degradation products, it usually means that the fluid-loss control is better. The required amount of fluid-loss control is dictated by the formation; and often in more permeable formations, small inert solids and additional polymeric materials are added to a fluid. The common fluid-loss additives are silica flour (+325 mesh), finely ground calcium carbonate, swelling polymers, resins, and many combinations of these. These are usually not tested separately, but are added to the fluid and filtered through a core or paper filter at formation temperature.

A variety of testing is required for frac fluids since they must work over a wide range of conditions. Physical testing of these fluids gives us some design information needed to do the engineering design of the fracture treatment. Frac fluids have been tested for compatibility with the formation water in oil and gas wells. Now, in geothermal wells the fluids' compatibility must be checked chemically at reservoir conditions because of the natural tendency of many geothermal brines to scale or precipitate compounds when a perturbation in temperature or pressure occurs. The real problem for these common frac fluids in geothermal wells is temperature--extremely high temperature. The polymers are degraded at elevated temperature due to chain scission and oxidation. Also, some of the anionic and cationic polymers react with other materials in the brine.

It is extremely difficult to quantify the viscosity, fluid loss, and other physical parameters of a fluid system by physical testing. The polymer fluids are sensitive to temperature, shear stress, shear rate, shear history, time at temperature, pH, and concentration. In this report a complete set of tests is run to screen the polymers made

available by chemical companies and oil field service companies. The main parameter measured throughout these tests is the apparent viscosity of the fluid at different conditions--conditions approaching geothermal reservoir temperatures. Two other important parameters which are measured on some of the better fluid systems are the fluid loss and proppant suspension or carrying capacity.

III. FRACTURING FLUID TESTS

The fracturing fluid system (liquid plus chemical and solid additives) must be tested so its properties will be known and predictable at all conditions encountered during its use. A short summary of the actual conditions encountered is given below so that it can be better understood why the following test program was planned.

The fluid systems are put together and polymers are hydrated at low temperature during surface mixing and tank loading. Then, the fluids are treated with chemicals, crosslinkers, stabilizers, and tracers. Proppants are added at the blender. From the blender the complete fluid system is pumped at high rate into high pressure (and high shear rate) pumps. These high horsepower pumps push the fluid system downhole through the tubing or casing and into the hot environment of the formation. Entering the formation through slots, perforations, or through open-hole fractures, the system again encounters higher shear rates and begins to be heated by the geothermal formation. As the fluid progresses out into the fracture, fluid leakoff occurs and the fluids slow down and are broken apart by the high temperature. At higher formation temperatures the polymer solutions are broken apart or degraded more quickly. This entire sequence shows a variance in temperature from ambient to the reservoir conditions. Shear rates can vary from near zero to over $10,000 \text{ sec}^{-1}$. Time and shear history vary depending on the size of the treatment and type of fluid that is used.

The basic ideas for fracturing fluid testing came from the API RP39 (see Appendix) which provides the recommended procedures to be used in testing frac fluids as defined by the American Petroleum Institute.

A. Viscosity Measurements

Some basic method of categorizing each fluid system is needed, so the measurement of apparent viscosity at various conditions has been chosen as the most convenient measurement. Of course, this alone does not tell the whole story since two fluids can have the same viscosity and not behave similarly with different shear rates, shear history or with temperature effects, pH effects, or other chemical effects added. These fluids are generally described as shear thinning or pseudoplastic materials.

In our lab, various concentrations of the polymeric solutions have been measured at ambient conditions and at elevated temperatures until degradation. At room temperatures, a Fann Model 35 is used to measure viscosity at 6 different shear rates from 5 to 1000 sec^{-1} at ambient conditions. High temperature and pressure measurements are made on a Fann Model 50C. Only at the 511 sec^{-1} reading do these instruments read directly in centipoises. Other readings must be multiplied by a shear rate factor as shown in Equation 1.

$$\mu_a = (\text{SF}) (\text{RDG}) \quad (1)$$

where μ_a = Apparent viscosity at given shear rate, cp.

RDG = Dial reading from Fann instrument

SF = Scale Factor as shown

Scale Factor (SF)	Speed (RPM)	Shear Rate (sec ⁻¹)
100	3	5.1
50	6	10.2
3	100	170
1.5	200	341
1	300	511
0.5	600	1022

Since the viscosity of a fracturing fluid is described at relative shear rate, it is necessary to compare different fluids at equal shear rate. The use of the "Power Law" is most practical to describe the uncrosslinked (non-Newtonian) fluids. This fluid model³ makes the comparison of different fluids much easier. The power law model has two parameters to describe the fluid. These are n' and K' . Equation 2 below shows how they are used to describe apparent viscosity.

$$\mu_a = 47,917 K' (\dot{\gamma})^{n'-1} \quad (2)$$

where μ_a = apparent viscosity (cp)
 $\dot{\gamma}$ = shear rate (sec⁻¹)
 K' = consistency index
 n' = power law index

From the Fann instruments the consistency index (K') can be calculated by dividing the reading taken at 300 RPM (511 sec⁻¹) by 511 to the n' power or as shown in Equation 3.

$$K' = \text{RDG}_{300} / 511^{n'} \quad (3)$$

The power law index (n') can be calculated by Equation 4.

$$n' = 3.32 \log (RDG_{600}/RDG_{300}) \quad (4)$$

The shear rates in actual field stimulation treatments can be estimated from several simple equations as shown below. The shear rate on the inside wall of the frac string can be written as Equation (5).

$$\dot{\gamma}_w = \frac{3n' + 1}{4n'} \cdot \frac{8V}{D} \quad (5)$$

where $\dot{\gamma}_w$ = shear rate at pipe wall (sec^{-1})
 n' = power law index
 V = fluid velocity
 D = inside pipe diameter

The shear rate experienced by the fluid system flowing in the fracture is shown by Equation 6.

$$\dot{\gamma}_f = 40.464 \cdot \frac{Q}{w^2 h} \quad (6)$$

where $\dot{\gamma}_f$ = shear rate in fracture
 Q = flow rate
 w = frac width
 h = frac height

The viscosity of the fluid is important since it suspends and carries proppant, helps control fluid loss, and provides a pressure drop down the fracture to influence the width of the fracture. A more viscous acting fluid creates a wider fracture. Any hydraulically created fracture has to at least be wide enough to allow the fracture proppant to enter.

Fluid system viscosities can be further enhanced and increased if crosslinking agents such as sodium tetraborate, titanates, and various compounds of chromium, aluminum, etc., are used. This complicates the viscosity measurement since the crosslinkers add a semblance of structure to the polymer solution. Behavior of these fluids is more complicated than pseudoplastic materials since they exhibit some elastic and normal stress properties, particularly at low temperatures. Problems caused in the rheology lab with this behavior are fluid segregation in the rotational viscometers and lumping which is sensitive to localized shear rates. Both Fann Model 35 and 50C instruments cannot be used as a standard to characterize crosslinked gels. Modifications have been made by different companies to help take measurements, but no standard or effective measurement has yet been agreed upon.

A tube viscometer³ would be effective to test the fluid in a more uniform manner since fluid segregation would not be possible. Such a system would require high temperature and pressure capabilities needed to simulate geothermal conditions. In such an instrument, the "tube" viscosity could be measured by Equation 7 shown below.

$$\mu_T = K_1 \Delta P / Q \quad (7)$$

where μ_T = tube viscosity, cp

ΔP = pressure drop in tube

Q = flow rate through the tube

K_1 = flow constant for tube

For a pipe viscometer, the shear stress is represented by Equation 8,

$$\tau_p = \frac{\Delta P \cdot D}{4L} \quad (8)$$

and the shear rate at the tube wall is given by Equation 9.

$$\dot{\gamma}_p = \frac{8V}{D} \text{ or } \frac{32Q}{\pi D^3} \quad (9)$$

where τ_p = shear stress at pipe wall

D = pipe diameter

ΔP = pressure drop

V = flow velocity

Q = flow rate

L = pipe length

$\dot{\gamma}_p$ = pipe wall shear rate, sec^{-1}

B. Static Aging and Screening

As polymer samples were received, they were mixed at a standard concentration and placed in a controlled, high temperature environment. Before and after this 4-hour static test at 300°F, Fann Model 50C viscometer readings are taken on each sample. Because so many samples were available, this allowed us to screen out obviously unsuitable polymer systems that were extremely temperature or salt sensitive. Since static tests are not representative of actual field conditions and conditions during the stimulation treatment, no quantitative information was taken or implied.

C. High Temperature Viscosity

During the stimulation treatment of geothermal hydrothermal reservoirs, the most important effect on the polymer fluid systems is the effect of high temperatures. To examine their behavior carefully at

high temperatures, a Fann Model 50C was used to measure (at 170 sec^{-1} shear rate) apparent viscosity during heat up from ambient to a temperature where degradation took place. As the fluid was being heated, shear diagrams were taken with every 100°F increase in temperature. Some fluids were held at specified temperatures from 1 to 2 hours to see the effect that time played on the degradation of the particular fluid. It was interesting to note that as the temperature increased, the viscosity decreased and the K' and n' changed drastically. From Equation 2, shown below we see that apparent viscosity is given as:

$$\mu_a = 47,917 K' (\dot{\gamma})^{n' - 1} \quad (2)$$

The effect of temperature is to decrease K' and increase n' so that the fluid system reverts back to a low viscosity Newtonian fluid where the viscosity is constant at any shear rate or can be calculated by dividing the shear stress (τ) by the shear rate ($\dot{\gamma}$).

$$\mu = \frac{\tau}{\dot{\gamma}} \quad (10)$$

D. Fluid Loss

Adequate control of fluid loss from the fracture is an important property of the fracturing fluid. Without fluid-loss control, no fracture volume can be generated. A reasonable percentage of the fluid entering the fracture must stay within the fracture to form the hydraulic wedge. The smaller the percentage of fluid loss, the more efficient the fluid system is. More fracture is created per unit volume of fluid injected when loss is low. Fractures cannot grow if fluid loss to the formation is approximately equal to fluid injection rate.

The rate of fluid loss from a fracture depends on reservoir properties, reservoir fluid characteristics, and the viscosity and wall-building characteristics of the fracturing fluid. If formation characteristics and fracturing fluid viscosity do not give adequate fluid-loss control, we can achieve control by adding finely ground solids to the fluid. These solids cover or bridge pore openings and restrict fluid flow from the fracture into the formation. As a result, relatively large fractures can be generated with moderate volumes of fracturing fluid.

Fluid-loss tests were carried out with equipment similar to that used for standard API water-loss tests for drilling muds, except a core is used as a filter medium instead of filter paper. In this test, fluid within the cell is static; in an actual fracture treatment, fluid is flowing parallel to the fracture wall at a velocity up to several hundred feet per minute. This fluid motion inhibits building of filter cake, resulting in higher fluid loss than is measured in a static test. However, static test data is usually accepted since dynamic tests involve much more complicated equipment.

For geothermal well stimulation, it is most important to use high temperature fluid-loss equipment to adequately simulate reservoir temperatures and their effects.

Howard and Fast⁴ postulated in 1957 that three resistances affect the rate of fluid loss from the fracture face. These are the formation of a filter cake on the fracture face when fluid-loss prevention additives are used, invasion of the formation matrix by fracturing fluid, and compression of reservoir fluids by invasion of fracturing fluids. A schematic representation of these mechanisms is given in Figure 6.

Figure 6. Combined Fluid-Loss Model

In this work, filter cake formation is assumed to occur similar to static tests, where fluid flow is directed only into the formation. This leads to a progressively decreasing fluid loss velocity proportional to one over the square root of time. The definition of the fluid-loss coefficient, (C_w), relates velocity as shown in Equation 11.

$$v = \frac{C_w}{\sqrt{t}} \quad (11)$$

Howard and Fast⁴ also defined compression of the reservoir fluids by a coefficient of fluid loss due to compression (C_c):

$$C_C = \sqrt{\frac{K\phi C_f (\Delta P_C)^2}{\pi \mu}} \quad (12)$$

and the fluid-loss control to frac fluid viscosity as the coefficient of viscous fluid loss (C_V).

$$C_V = \sqrt{\frac{K\phi (\Delta P_V)}{2\mu_f}} \quad (13)$$

where	v = fluid velocity	μ = formation fluid viscosity
	k = formation permeability	μ_f = frac fluid viscosity
	ϕ = formation porosity	C_C = compressed fluid coefficient
	C_f = formation fluid compressibility	C_V = viscous fluid-loss coefficient
	ΔP_C = pressure drop across reservoir	C_W = filter cake coefficient
	ΔP_V = pressure drop across invaded zone	
	t = time	

Where fluid-loss additives are used, the (C_W) filter cake fluid-loss coefficient usually provides the majority of the fluid-loss control. However, to calculate the other effects, it is necessary to calculate a combined fluid-loss coefficient, and this is done as suggested by Williams et al,¹² as shown in Equation 14

$$C_T = \frac{2 C_C C_V C_W}{C_V C_W + \sqrt{C_W^2 C_V^2 + 4 C_C^2 (C_V^2 + C_W^2)}} \quad (14)$$

where C_T is the combined fluid-loss coefficient that applies to the specific treatment.

In our tests we will measure and compare the various C_w of each fluid and fluid-loss combination at reservoir conditions.

E. Falling Ball Viscometer

A definite idea of how well a fluid will carry and place proppant in the hydraulically created fracture is important to the overall success of the stimulation treatment. The idea of measuring a falling ball (sphere) in a static fluid at reservoir conditions allows us to get another physical measurement from our fluid system before we try it in the field. Figure 7 shows the high temperature falling ball viscometer developed by Maurer Engineering Inc. during 1979. It uses a magnetically suspended steel ball (of various sizes) as the falling ball. A timing circuit and sensitive pressure transducer measures, remotely, the time of fall for the ball. By use of this instrument, we

Figure 7. MEI High Temperature Falling Ball Viscometer

can measure an average velocity or fall rate through the fluid which is held at geothermal temperature and pressure. With this fall rate we can estimate fluid viscosity and sand-settling characteristics of the fluids. According to Novotny,⁶ this is not an accurate measurement when the fluid is static, because proppants that are completely suspended in a highly viscous gel (crosslinked fluid) will settle when the fluid is sheared. However, since temperature is constantly degrading the fluids versus time, at some point the fluid will start to lose its crosslinked behavior, and the falling ball viscometer will follow the Novotny analysis.⁶

For a single particle in a Newtonian fluid, the terminal settling velocity can be calculated by equating the drag force ($\pi C_{D3} V_{\infty}^2 \rho d^2 / 8$) and the gravitational minus buoyant forces ($\pi d g (\rho_p - \rho) / 6$) on the particle. For spheres, the drag coefficient is a function of the Reynolds number and can be closely approximated using three different regions of Reynolds numbers. From this, the settling velocity of a single sphere in a Newtonian fluid with no walls present to hinder the settling can be given by the following equations:

For $N_{Re} < 2$ (Stokes-law region), $C_D = 24/N_{Re}$ and

$$V_{\infty} = \frac{g(\rho_p - \rho)d^2}{18\mu} \quad (15)$$

where V_{∞} = terminal velocity

g = gravity

ρ_p = proppant density

ρ = fluid density

d = proppant diameter

μ = fluid viscosity

N_{Re} = Reynold's number

For $2 < N_{Re} < 500$ (intermediate region), $C_D = 18.5/N_{Re}^{0.6}$ and

$$V_{\infty} = \frac{20.34(\rho_p - \rho)^{0.71} d^{1.14}}{\rho^{0.29} \mu^{0.43}} \quad (16)$$

For $N_{Re} > 500$ (Newton's law region), $C_D = 0.44$ and

$$V_{\infty} = 1.74 \sqrt{\frac{g(\rho_p - \rho) d}{\rho}} \quad (17)$$

If the flow around the particle is laminar, the equations will hold even if the particle is not perfectly spherical. For most proppants, settling is in the Stokes region. However, serious errors in predicting settling velocity can be made if Equation 15 is used when $N_{Re} \gg 2$.

F. Non-Newtonian Fluids

For non-Newtonian fluids, the fluid viscosity varies with shear rate. If settling is in a stagnant non-Newtonian fluid, the effective shear rate on the particle is the settling velocity divided by the particle diameter (V/d). If this shear rate is used to calculate the fluid viscosity with a power-law fluid model ($\mu = K' \dot{\gamma}^{n'-1}$), Stokes Law settling velocity can be predicted by

$$V_{\infty} = \frac{g(\rho_p - \rho) d^{1/n'}}{18K'} \quad (18)$$

Settling velocity is a function of K' and n' , the power law

parameters. For non-Newtonian fluids, the settling velocity is proportional to $d^{(1 + n')/n'}$ instead of d^2 as for Newtonian fluids.

Figure 8 shows a settling rate for single particles of various size sand in fluids of different viscosities. The dashed line shows a steel ball's settling rate in 200 cp fluid. Further data and conclusions will be discussed in Section V on the Falling Ball Viscometer Test Results.

Figure 8. Single Particle Settling Velocity vs Particle Diameter

IV. VISCOSITY TEST RESULTS

Rheological and physical properties of polymers tested for possible geothermal use are reported in this section. The properties have been determined initially and after static aging at 300°F - 100 psi of nitrogen pressure (static tests). From the results of these screening tests initially and after aging, promising candidates have been selected for more in-depth study with the Fann 50C. The judgment for testing is based on viscosifying ability and temperature stability. Investigations include influence on the polymers from various chemical additives and the effects of various temperatures and concentrations. The effects of the various fluid systems on fluid-loss characteristics are discussed in Section V.

A. Uncrosslinked Polymer Fluids

1. Static Tests

Initial tests indicated that Galactosol 416, Natrosol 150-HHWR, Natrosol 250 HHW, XC-Polymer, HP Guar and Galactosol 210 had the highest yield value of all of the available polymers. All of the above polymers were hydrated under equal conditions--both time and mixing. All of the polymers tested except the Romax RM-95 and RM-66 samples initially had a pH of about 8.0. There is little filtration control in any of the 25 samples tested. However, due to the high shear mixing involved in preparing the samples, foaming is a slight problem, so 2-ethyl hexanol has been used to defoam the samples before initial testing. A better cure has been found by using low shear mixing more closely associated with realistic field conditions. Most of the later tests used low shear mixing.

Table I shows the ratio of rheological properties after static aging to the properties measured initially. These results are not used

Table I
Static Aging Tests for Polymer Solutions
at 1% Concentration at 170 Sec⁻¹

<u>Polymer Name</u>	<u>Polymer Type</u>	<u>Ratio of Aged Viscosity to Initial Viscosity</u>
Methocel	Cellulose Derivative	0.4
Galactosol 416	Guar Derivative	0.33
Gendril Thik	Guar Derivative	0.29
Gengel E-9	Guar Derivative	0.4
Natrosol HHWR-150	Cellulose Derivative	0.22
CMC Driscose	Cellulose Derivative	0.8
Natrosol 250 HHXR	Cellulose Derivative	0.17
Natrosol 255 HHW	Cellulose Derivative	0.28
NGL-6829 HP-8	Guar Derivative	0.31
HEC QP-100 MH	Cellulose Derivative	0.29
HEC QP-100M	Cellulose Derivative	0.22
HP Guar	Guar Derivative	0.22
Romax Rm 95	Unknown	2.0
Romax Rm 96	Unknown	2.0
Cellosize QP-50000	Cellulose Derivative	0.36
Impermex	Starch	1.5
Drispae	Cellulose Derivative	0.22
HV Driscose	Cellulose Derivative	0.25
Cypan	Polyacrylate	0.75
XC Polymer	Polysaccharide	0.22
Gengel E-9P	Guar Derivative	0.4
Guartec MF	Guar Derivative	0.38
Galactosol 210	Guar Derivative	0.17
ASP 711	Polyacrylamide (cationic)	1.0
ASP WFRII	Polyacrylamide (anionic)	0.5

quantitatively but did give us some insight to select the samples for in-depth testing with the Fann Model 50C. Initially, high viscosity values, together with good viscosity recovery after static aging, had been used to select the samples. From these results, XC-Polymer, Gengel E9-P (hydroxypropyl guar), Cellosize OP 50,000 (hydroxyethyl cellulose), Gengel E9 (hydroxypropyl guar), Romax RM-66, Dowell YF "HC" system and Western's Apollo Gel 60 system have been selected for further testing with the 50C. Considerable work has been done on the hydroxypropyl guar samples, the XC-Polymer and the hydroxyethyl cellulose samples. The effects of methanol and an oxygen scavenger (Coat 777) have also been determined. The polymer concentrations were constant at 83.3 lb/1000 gal, unless otherwise indicated, to provide a basis for comparison and also to provide a healthy viscosity for the instruments to measure. Most tests were terminated as the polymer solutions degraded back to a viscosity near that of water.

2. High Temperature Viscosity Testing

a. NGL 6829

Figures 9 and 10* show shear diagrams of a modified guar gum derivative marketed by Stein Hall. Figure 9 shows the effect of temperature on viscosity by the lowering of the curves with increased temperature and time. Also shown is the recovery of viscosity after exposure to 300°F and 100 psi of nitrogen pressure. About 40% of the viscosity measured at 100°F is lost by the time the sample reached 300°F. After remaining at 300°F for 60 minutes, the viscosity is reduced another 40%, so about 80% of the sample's viscosity at 100°F is lost after exposure to 300°F for 60 minutes. Figure 10 better illustrates this reduction in viscosity with temperature. It also shows that a 25% reduction in viscosity occurred after the sample temperature reached 200°F. Calculation of K' and n' power law parameters shows a marked decrease in K' with increasing temperature and an n' that

*All figures can be found in the last part of Section IV, pp. 38-109.

approaches 1.0 at 300°F after an hour. This indicates almost complete polymer degradation and a change from a non-Newtonian to a Newtonian fluid.

b. Gengel E9

Gengel E9 is a hydroxypropyl guar product. After exposure to 300°F, this sample did not lose quite as much viscosity as the previous sample. However, the viscosity at 100°F of the HP-8 is more than 50% greater than this fluid at 100°F. Even though the viscosity of E9 at 300°F is lower than the corresponding viscosity for HP-8, the E9 did retain more of its viscosity after being exposed to 300°F for 1 hour. Figures 11 and 12 show the reduction in viscosity of this hydroxypropyl guar sample after being exposed to the various temperatures. High temperature causes the fluid to become more Newtonian on the shear stress--shear rate diagram. This shows a straight line which is closer to the horizontal axis.

c. Gengel E9P

E9P is identical to E9 except that this sample is pulverized, resulting in a smaller initial particle size compared to the E9 discussed above. Figure 13 shows the effect of increasing temperature on the effective viscosity at 170 sec^{-1} . When the viscosity is measured at 100°F a second time, after exposure to 300°F for 60 minutes, half the viscosity is lost compared to the first measurement at 100°F. This is better illustrated in Figures 14 and 15 where the shear stress--shear rate diagram show the viscosity at various shear rates during the heat-up interval. The apparent viscosity versus temperature drops almost linearly with increasing temperature, and we only measure about 25 cp initially at 300°F (see Figure 13). After an hour, the apparent viscosity has dropped to about 8 cp.

d. Gengel E9P with 5% Methanol

Figures 16 through 18 show methanol added to the HP Guar to test its effect on the thermal stability of the polymer sample. Initially, 5% methanol is added. The addition did not aid the temperature stability in terms of our measurements. There is an increase in viscosity recovery at 100°F after exposure to 300°F as seen in Graph 9. In general, we cannot conclude that significant improvement in thermal stability is achieved when 5% methanol is added from our test data in short-term testing. Service company data show some significant effects over longer time periods and at higher temperatures when methanol is used.

e. Gengel E9P with 10% Methanol

Figures 19 through 21 show a similar mixture of HP guar with 10% methanol. The increased methanol concentration did improve the temperature stability. Figure 19 shows a reduction in viscosity versus temperature similar to the previous test but a much slower drop in viscosity with time at the high temperature. Even though the improvement is slight with the 10% methanol, it is a definite improvement and may be economically desirable in larger treatments or with longer pumping times. Figures 20 and 21 show the shear stress--shear rate diagrams used to construct the viscosity versus time in Figure 19.

f. Gengel E9P and 5% Methanol

Figures 22 through 24 show the hydroxypropyl guar dispersed in the methanol first followed by dispersing this slurry in the water. This method of sample preparation did ease hydration of the polymer; however, no improvement of the thermal stability could be observed. This comparison for this conclusion can be seen in Figures 18 and 23. The plots for adding the polymer/methanol slurry to water are nearly identical to the plots where the polymer is hydrated in the water followed by the methanol addition. However, the reversing of the mixing did make sample preparation easier.

g. Kelzan XC Polymer

Figures 25 through 27 show the results of testing Kelzan XC Polymer, a biopolymer or polysaccharide. Graph 27 shows that a small viscosity reduction occurred as the temperature approached 200°F. However, as the temperature approached 300°F, the viscosity dropped rapidly. After exposure to 300°F for 60 minutes, the viscosity is reduced almost to that of water. The lack of viscosity recovery measured after the sample is returned to 100°F indicated that more of this polymer had degraded than the HP guar polymer samples. This total degradation also indicates a cleaner, less damaging fluid with the XC fluid systems due to the lack of residual solids or fluid viscosity upon cooling.

h. XC Polymer and 5% Methanol

Figures 28 through 30 show where 5% methanol is added to the XC Polymer to improve thermal stability. The methanol slightly improved the viscosity at 300°F. Figure 29 indicates only a small effect of the methanol on temperature stability compared to Figure 26. Even though the sample had slightly more viscosity at 300°F than the same sample without any methanol, the viscosity approaches that of water during the 60 minutes that the sample is exposed to 300°F. The improvement is slight, so no advantage can be seen in using 5% methanol.

i. Kelzan XC Polymer/Methanol Slurry in Water

Figures 31 through 33 show where the XC Polymer is dispersed in methanol, and this slurry is then added to water. The methanol concentration is 5%. A slight improvement is noted in the viscosity at the various temperatures. Adding the methanol and polymer in this way did seem to slightly increase the temperature stability of the polymer. As before, the viscosity is reduced as the polymer remains at 300°F for 60 minutes. However, improvement in viscosity is seen as the polymer sample approached 300°F. The methanol did not seem to help the viscosity at 300°F beyond the sixty-minute period of test time.

j. Gengel E9P: XC Polymer Blend (5:1)

Figures 34 through 37 show the first instance that the benefits of a blend have been determined. The blend had higher viscosities at 100°F, 200°F, and 300°F than the XC Polymer sample, but the viscosity was slightly lower at the same temperatures than the Gengel E9P sample alone. The same relationship is true for the samples after exposure to 300°F for 60 minutes. However, due to improvements in viscosity over the XC Polymer and the slight reduction in viscosity over the Gengel E9P alone, no definite advantage was seen in using this blend over the Gengel E9P sample alone. Figure 37 is a retest of the same fluid system reported in Figures 34 through 36.

k. Gengel E9P and XC Polymer (5:1) with 5% Methanol

Figures 38 through 40 are identical to those shown in Section j except the samples contained 5% methanol to impart some thermal stability. However, when comparing these results to those above, no improvement in thermal stability is found when the methanol is added. The viscosity for this sample at 100°F and 300°F (Figure 39) is the same as the corresponding viscosity at the same temperature (Figure 35) for the sample without the methanol. Therefore, since no improvements are noted, no advantage could be found to recommend this system over the Gengel E9P fluid system.

l. Gengel E9P and XC Polymer (5:1) in 5% Methanol

Figures 41 through 43 are identical samples to those in Section k except that this sample was subjected to 400°F. As expected from the results seen at 300°F, the viscosity dropped rapidly to that of water by the time the sample reached 400°F as shown in Figure 41. The viscosity is reduced 75% by the time the sample reached 300°F. So, considering all tests previously run with methanol, hardly any advantage is seen in using methanol as a thermal stabilizer at 5% concentration. The slight improvements it made do not appear to be cost efficient.

m. Gengel E9P and XC Polymer (5:1) with Coat 777

Figures 44 through 46 show the use of an oxygen scavenger, Coat 777, to aid in thermal stability. The addition of the oxygen scavenger had minimal effect on maintaining viscosity at the elevated temperatures. The data using the oxygen scavenger is almost identical to the data for this blend without anything added for thermal stability. The oxygen scavenger containing sample did have slightly more viscosity at the various temperatures than did the corresponding sample containing the methanol. So it appears that the oxygen scavenger is a better thermal stabilizer than the methanol at the concentrations used, but the improvement over samples not containing any thermal stabilizer is minimal.

n. Gengel E9P and XC Polymer with 5% Methanol and Coat 777

Figures 47 through 49 show a polymer blend where both thermal stabilizers were tested in the same sample. A slight improvement in thermal stability was observed at 300°F. Even after 60 minutes, more viscosity was observed than in other samples--the improvement was slight but definite. Figure 47 shows the more gradual reduction in viscosity with increasing temperature. This figure, along with Figures 48 and 49, shows a slightly higher viscosity at 300°F after exposure for 60 minutes.

o. Romax RM66 with Soda Ash

Figures 50 through 52 show the fluid system made from Romax RM66 which is a proprietary polymer. This material had good thermal stabilities compared to other samples tested to this point. It was as good as the NGL 6829 HP8 and the Gengel E9 sample after exposure to 300°F for 60 minutes. Even so, 50% of the viscosity measured when the sample first reached 300°F was lost after 60 minutes at 300°F. Even though the viscosity at the lower temperatures was somewhat less, the viscosity at elevated temperatures was comparable to the best of the

previously tested samples. It should be mentioned that these tests were made in fresh water, so brine effects on polymer hydration have not been tested.

p. Cellosize QP 50,000

Figures 53 through 55 show tests on Cellosize QP 50,000 which is a hydroxyethyl cellulose. It has excellent viscosity at 100°F, the highest measured at that temperature. However, as the temperature approaches 300°F, the viscosity is reduced almost 90%. About 60% of the viscosity is lost by the time the sample reached 200°F, even though the viscosity was drastically reduced at 300°F, the actual value was comparable with the Romax RM66, NGL 6829 HP8, and Gengel E9 samples. During the sample exposure time at 300°F, the viscosity is reduced by one half. It is basically comparable to the Romax RM66 and hydroxypropyl guar mentioned earlier.

q. Cellosize QP 50,000 with 5% Methanol

Figures 56 through 58 show identical tests to Section p except that 5% methanol is added for thermal stability. As before, the methanol at this concentration seemed to have no effect at all on improving the thermal stability of the polymer. The actual values for the viscosity of this hydroxyethyl cellulose sample at the various temperatures are practically identical to those of the previously tested hydroxyethyl cellulose sample without any methanol. So any advantage for using the 5% methanol to increase thermal stability is not apparent.

r. Cellulose QP 50,000 and Methanol Slurry in Water

Figures 59 through 61 show test on the hydroxyethyl cellulose where it was first dispersed in methanol before hydrating in water. As before, the methanol had a negligible effect on the thermal stability at 300°F. There is some slight improvement. The most noticeable improvement is the viscosity at 100°F. There is 25% higher viscosity with this sample at 100°F than the other tests using

hydroxyethyl cellulose. There is also excellent viscosity recovery when measured again at 100°F after having been exposed to 300°F for 60 minutes. Also, the viscosity at 200°F for this sample is 25% higher than the other hydroxyethyl cellulose samples at 200°F. At 300°F the viscosity is only slightly greater for this sample than for the other hydroxyethyl cellulose samples.

s. Kelco S-88 Biopolymer

An unspecified, experimental biopolymer (S-88) has been tested during our study. Figure 62 shows the viscosity versus temperature and time at 350°F. This unusual polymer has almost no degradation or loss of viscosity until 300°F. However, between 300°F and 350°F the polymer is rapidly and completely disintegrated. These properties are unique in all of our tests and this material may be quite useful in wells with temperatures around 300°F. The cost of this material was high because it was made as a pilot batch. If it became available in large quantities, costs would probably decrease.

B. Crosslinked Polymer Fluids

Fluid systems that incorporate complexing agents have been used more frequently as the oilfield service companies have become aware of the benefits of increased viscosity.⁷ However, crosslinking polymers can cause problems such as difficult blending of sand or proppant into the fluid, uniform premixing, constant and predictable behavior, and other field oriented problems.

The main benefit derived from crosslinking a fluid system is that it becomes a perfect support fluid and prevents proppant settling. Another benefit is slightly increased thermal stability. Several key papers^{8,9,10,11} have presented these and other ideas to promote the use

of viscous fluids. Once the fluids began to be accepted, laboratory tests began to find the useful limits of the systems. Unfortunately, as test temperatures increased, the standard viscometers became less accurate and only tested part of the samples. Besides being temperature sensitive, the crosslinking makes the fluid system sensitive to shear and shear history. In the Fann Model 50C a small sample of the crosslinked material is tested but segregation quickly occurs, and the plastic gel-like behavior prevents stirred mixing. As a result, the apparent viscosity of the material left between the wall of the cup and the bob degrades with temperature and time even more quickly than it should under a stirred test. With modified equipment adding extra stirring and wider spacing of the Fann Viscometer, more representative data has been obtained. Each service company has its own procedures; therefore, our data taken on an unmodified Fann 50C instrument with standard heatup times will usually show less viscosity than the service companies' readings.

The way in which our tests are conducted represents the harshest conditions that the crosslinked fluid systems will see and shows the ultimate fate of these systems. Further testing should be made with a modified instrument to more closely check the service company results.

Each service company has several crosslinked fluid systems, usually a guar derivative and a cellulose derivative system. As many as possible were acquired and tested in our lab on the unmodified 50C viscometer to measure the fluid viscosity with time. The fluid systems of the service companies are not identified except by their generic names. All fluid systems of this type are given sample numbers A through I.

1. Crosslinked Guar Systems

a. Samples A & B are shown in Figure 63.* Their viscosity has been measured at 170 sec^{-1} up to 350°F and held at 350°F for one hour. Sample A has 100 lb/1000 gal of polymer in solution, while Sample B has only 42 lb/1000 gal in solution. The lower polymer solution holds up slightly better at temperature probably because of its ability to mix inside the viscometer. Otherwise, both fluids are pretty well degraded by our measure of viscosity after an hour at 350°F . Usually a portion of undegraded material remains as a viscous material above the bob where shear rate has been low.

b. Samples C & D are shown in Figure 64 where their viscosity at 170 sec^{-1} has been measured to 300°F and at 300°F for one hour. Both samples had a polymer concentration of 42 lb/1000 gals except that Sample C also had a 7 lb/1000 gal of a high temperature stabilizer or buffer. Both rapidly degraded in these tests until only a water-like viscosity can be measured at the end of the hour.

c. Sample E is shown in Figure 65 where its viscosity at 170 sec^{-1} is plotted against temperature to 350°F and time at 350°F to one hour. A similar behavior is seen as compared to Samples A through D as its viscosity is broken down by the high temperature and continual shear. After an hour, the fluid behavior became Newtonian with a low apparent viscosity near that of water.

*See all figures at end of this section, pp. 38-109.

2. Crosslinked Cellulose Systems

a. This fluid system (Sample F) is a commercially available crosslinked cellulose. This system's viscosity was measured to 400°F and then monitored versus time for an hour at 400°F. Figure 66 shows the viscosity behavior. It was tested at 85 lb/1000 gal polymer concentration comparable to the majority of the other samples tested. After exposure to 300°F, the viscosity was reduced 50% from what it was at 100°F but the actual value was one of the highest values achieved at this temperature. However, with time and the 400°F exposure, the sample's viscosity was reduced to that of water. Figure 67 shows the shear diagrams of the sample F which go from a pseudoplastic fluid at 100°F to a Newtonian fluid at 400°F. Figure 68 shows a shear diagram at each 100°F increment to 400°F. At 300°F a large viscosity and non-Newtonian behavior is still present.

b. Sample G is also a commercially available, thermally stable crosslinked cellulose fluid system. Figure 69 shows the extremely high viscosity measured as the temperature increased to 300°F. The apparent viscosity is measured at about 20 cp at 300°F and drops to about 30 cp after one hour. This system used a larger polymer concentration (1-1/2% by weight) than most other systems tested; hence, it is probably more expensive than others with less polymer added per 1000 gallons. Regardless, a substantial amount of viscosity is available to help during high temperature fracturing. Figures 70 and 71 show the shear diagrams of Sample G. In Figure 70 the interesting thing to notice is that a large percent of the viscosity is recovered after cool down. This means that the polymer has not permanently degraded by staying at 300°F for one hour. Higher temperatures would of course affect the system adversely. Figure 71 shows a shear diagram at each 100°F increment as the temperature increased to 300°F.

c. Sample H is tested at a 60 lb/1000 gal concentration. It is also a commercially available crosslinked cellulose derivative fluid

system. Figure 72 shows a rapid drop in viscosity versus temperature and a slower degradation as shear continues on the 300°F fluid for one hour. The fluid retained the same amount of viscosity after one hour and the shear diagram on Figure 73 shows the shear stress--shear rate behavior before and after testing at 100°F. Over 50 percent of the viscosity was recovered. Figure 74 shows a shear diagram at every 100°F increment to 300°F and after one hour at 300°F. Some viscosity is retained in this system, so further tests to higher temperatures should be considered.

3. Crosslinked Polyacrylamide Systems

Only with fresh water or water having low ionic content can a polyacrylamide fluid system be considered. For general use and in brines with a 1 or 2 percent salt content, these fluids may not be suitable.

a. Sample I, shown in Figure 75, has been tested to 350°F and viscosities have been measured for one hour at 350°F. A low viscosity was measured initially and all was lost by 350°F. The crosslinking process recommended by the manufacturer was followed; but a slow crosslinking, batch mix fluid would not be too practical in regular field use.

C. Miscellaneous Polymer Fluids

There are other fluid systems used in the oil field which are thickeners for water-based fluids. Such fluids are used in drilling muds and other applications, and most often are not suitable as clean fracturing fluids. Some of the other systems looked at were starch, bentonite, and miscellaneous surfactants. Most of these have obvious drawbacks such as permanent damage to the formation. Because of this, no detailed testing was conducted on these fluids.

Several Cyfloc fluids have been tested, however, on the Model 50C Fann viscometer and the results of these tests are discussed next.

1. Cyfloc 5100: Cationic Polyacrylamide

Several polyacrylamides obtained from cyanamide have been tested in fresh water or tap water with low ionic content. Viscosity is measured to 350°F on the Cyfloc 5100 fluid system. Before 350°F the fluid measures about the same as water viscosity. This data is shown on Figure 76 at a polymer concentration of 1% by weight. This cationic polymer shows relative little buildup in viscosity even at room temperature compared to other commonly used water soluble polymers.

2. Cyfloc 4000: Anionic Polyacrylamide

Figure 77 shows the viscosity of an anionic polyacrylamide. This freshwater solution shows high viscosity and good temperature stability; however, it competes with any dissolved salts for the water molecules. In high total dissolved solids (TDS) water, the polymer hydrolyzes poorly and will not build viscosity of the same magnitude. This polymer has definite possibilities in geothermal areas with very low TDS fluids.

The fluid is tested at a 1% concentration by weight but would be used at lower concentrations in the field because of cost and because of tailoring the viscosity to carry the proppant. Figure 78 shows the same fluid system at 1/2% by weight. The viscosity of the solution degrades with time; however, it appears to retain about 10 cp for an hour at 350°F quite well. Further checking needs to be made on the availability and price of this material.

3. Cyfloc 4500: Non-ionic Polyacrylamide

Figure 79 shows the viscosity behavior of the non-ionic polyacrylamide versus temperature and time. It was the only fluid tested to increase in viscosity while holding the fluid at 350°F temperature for an hour. This behavior cannot be explained simply, but it is believed that the polymer had a tendency to complex itself at the 1% by weight concentration. Figure 80 shows quite a different behavior for the non-ionic polyacrylamide at 1/2% by weight. No self-complexing is observed although some temperature stability is seen with about 5 cp viscosity retained for one hour at 350°F. Even though the anionic fluid tested higher, it shows more salt sensitivity than the nonionic fluid systems.

FIGURE 9
Shear Stress--Shear Rate Relationship of
NGL 6829 HP-8 (83-3 lb/1000 gal)

FIGURE 10
Shear Stress--Shear Rate Relationship of
NGL 6829 HP-8 (83.3 lb/1000 gal)

FIGURE 11

Shear Stress--Shear Rate Relationship of
Gengel E9 (83.3 lb/1,000 gal)

FIGURE 12

Shear Stress--Shear Rate Relationship of
Gengel E9 (83.3 lb/1,000 gal)

VISCOSITY AT 170 SEC^{-1}
OF
GENGEL E9P
(83.3 LB/1,000 GAL)

VISCOSITY, CP.

140
120
100
80
60
40
20
0

0 100 200 300 40 60

TEMPERATURE, °F

TIME AT 300°F, MIN.

FIGURE 13

FIGURE 14
 Shear Stress--Shear Rate Relationship of
 Gengel E9P (83.3 lb/1,000 gal)

FIGURE 15

Shear Stress--Shear Rate Relationship of
Gengel E9P (83.3 Lb/1,000 gal)

FIGURE 16

FIGURE 17

Shear Stress--Shear Rate Relationship of
Gengel E9P (83.3 Lb/1,000 gal)
and Methyl Alcohol (5%)

FIGURE 18

Shear Stress--Shear Rate Relationship of
Gengel E9P (83.3 lb/1,000 gal)
and Methyl Alcohol (5%)

FIGURE 19

FIGURE 20

Shear Stress--Shear Rate Relationship of
Gengel E9P (83.3 lb/1,000 gal)
and Methyl Alcohol (10%)

FIGURE 21

Shear Stress--Shear Rate Relationship of
Gengel E9P. (83.3 lb/1,000 gal)
and Methyl Alcohol (10%)

FIGURE 22

FIGURE 23

Shear Stress--Shear Rate Relationship of
Gengel E9P (83.3 lb/1,000 gal)
and Methyl Alcohol (5%)

FIGURE 24
 Shear Stress--Shear Rate Relationship of
 Gengel E9P (83.3 lb/1,000 gal) and
 Methyl Alcohol (5%)

FIGURE 25

FIGURE 26
 Shear Stress--Shear Rate Relationship of
 XC Polymer (83.3 lb/1,000 gal)

FIGURE 27
Shear Stress--Shear Rate Relationship of
XC Polymer (83.3 lb/1000 gal)

FIGURE 28

FIGURE 29
 Shear Stress--Shear Rate Relationship of
 XC Polymer (83.3 lb/1,000 gal)
 and Methyl Alcohol (5%)

FIGURE 30

Shear Stress--Shear Rate Relationship of
XC Polymer, (83.3 lb/1,000 gal)
and Methyl Alcohol (5%)

FIGURE 31

FIGURE 32

Shear Stress--Shear Rate Relationship of
XC Polymer, (83.3 lb/1,000 gal)
and Methyl Alcohol (5%)

FIGURE 33

Shear Stress--Shear Rate Relationship of
XC Polymer (83.3 lb/1,000 gal) and
Methyl Alcohol (5%)

FIGURE 34

FIGURE 35

Shear Stress--Shear Rate Relationship of
A Mixture of Five Parts Gengel E9P and One
Part XC Polymer (83.3 lb/1,000 gal)

FIGURE 36

Shear Stress--Shear Rate Relationship of a
Mixture of Five Parts Gengel E9P and One part
XC Polymer (83.3 lb/1,000 gal)

FIGURE 37

Shear Stress--Shear Rate Relationship of
A Mixture of Five Parts Gengel E9P and One Part
XC Polymer (83.3 lb/1,000 gal)

FIGURE 38

FIGURE 39

Shear Stress--Shear Rate Relationship of
 A Mixture of Five Parts Gengel E9P and One Part
 XC Polymer (83.3 lb/1,000 gal) and
 Methyl Alcohol (5%)

FIGURE 40

Shear Stress--Shear Rate Relationship of a Mixture of Five Parts Gengel E9P and One Part XC Polymer (83.3 lb/1,000 gal) and Methyl Alcohol (5%)

FIGURE 41

FIGURE 42

Shear Stress--Shear Rate Relationship of
A Mixture of Five Parts Gengal E9P and One Part XC
Polymer (83.3 lb/1,000 gal) and Methyl Alcohol (5%)

FIGURE 43

Shear Stress--Shear Rate Relationship of a Mixture of Five Parts Gengel E9P and One Part XC Polymer (83.3 lb/1,000 gal) and Methyl Alcohol (5%).

FIGURE 44

FIGURE 45

Shear Stress--Shear Rate Relationship of a Mixture of Five Parts Gengel E9P and One Part XC Polymer (83.3 lb/1,000 gal) and Coat 777 (11.8 lb/1,000 gal)

FIGURE 46

Shear Stress--Shear Rate Relationship of a
Mixture of Five Parts Gengel E9P and One Part
XC Polymer (83.3 lb/1,000 gal) and Coat 777
(11.8 lb/1,000 gal)

FIGURE 47

FIGURE 48

Shear Stress--Shear Rate Relationship of a
Mixture of Five Parts Gengel E9P and One Part
XC Polymer (83.3 lb/1,000 gal) and Methyl Alcohol
(5%), and Coat 777 (11.8 lb/1,000 gal)

FIGURE 49
 Shear Stress--Shear Rate Relationship of a Mixture
 of Five Parts Gengel E9P and One Part XC Polymer
 (83.3 lb/1,000 gal) and Methyl Alcohol (5%) and
 Coat 777 (11.8 lb/1,000 gal)

VISCOSITY AT 170 SEC⁻¹
OF
ROMAX RM 66
(83.3 LB/1,000 GAL) AND
SODA ASH (28 LB/1,000 GAL)

VISCOSITY, CP.

120
100
80
60
40
20
0

TEMPERATURE, °F

TIME AT 300 °F, MIN.

0 100 200 300 40 60

FIGURE 50

FIGURE 51

Shear Stress--Shear Rate Relationship of
Romax RM-66 (83.3 lb/1,000 gal) and Soda
Ash (28 lb/1,000 gal)

FIGURE 52

Shear Stress--Shear Rate Relationship of Romax
RM-66 (83.3 lb/1,000 gal) and Soda Ash (28 lb/1,000 gal)

Figure 53

FIGURE 54

Shear Stress--Shear Rate Relationship of
Cellosize QP 50,000 (83.3 lb/1,000 gal)

FIGURE 55

Shear Stress--Shear Rate Relationship of
Cellosize QP 50,000 (83.3 lb/1,000 gal)

FIGURE 56

FIGURE 57

Shear Stress--Shear Rate Relationship of Cellosize
QP 50,000 (83.3 lb/1,000 gal) and Methyl Alcohol (5%)

FIGURE 58

Shear Stress--Shear Rate Relationship of
Cellosize QP 50,000 (83.3 lb/1,000 gal)
and Methyl Alcohol (5%)

Figure 59

FIGURE 60

Shear Stress--Shear Rate Relationship of
Cellosize QP 50,000 (83.3 lb/1,000 gal)
and Methyl Alcohol (5%)

FIGURE 61

Shear Stress--Shear Rate Relationship of Cellosize
QP 50,000 (83.3 lb/1000 gal) and Methyl Alcohol (5%)

Figure 62. Crosslinked Fluid Systems -- Kelco S 88

Figure 63. Samples A + B Crosslinked Fluid Systems--Guar Derivatives

Figure 64. Samples C + D Crosslinked Fluid Systems--Guar Derivatives

Figure 65. Sample E Crosslinked Fluid System--Guar Derivative

FIGURE 66

FIGURE 67

Shear Stress--Shear Rate Relationship of Sample F--
Crosslinked Cellulose (85 lb/1000 gal Polymer Concentration)

96
SHEAR STRESS,
FANN MODEL 50 C DIAL READING

FIGURE 68.

Shear Stress--Shear Rate Relationship of Sample F--
Crosslinked Cellulose (85 lb/1000 gal Polymer
Concentration)

FIGURE 69

FIGURE 70

Shear Stress--Shear Rate Relationship of Sample G--
 Crosslinked Cellulose Derivative (120 lb/1000 gal
 Polymer Concentration)

FIGURE 71

Shear Stress--Shear Rate Relationship of Sample G--
Crosslinked Cellulose Derivative (120 lb/1000 gal
Polymer Concentration)

FIGURE 72

FIGURE 73

Shear Stress--Shear Rate Relationship of Sample H--
 Crosslinked Cellulose Derivative (60 lb/1000 gal
 Polymer Concentration)

FIGURE 74

Shear Stress--Shear Rate Relationship of Sample H--
Crosslinked Cellulose Derivative--(60 lb/1000 gal
Polymer Concentration)

Figure 75. Viscosity of Sample I--A Crosslinked Polyacrylamide

Figure 76. Viscosity of Cyfloc 5100 (Cationic) Fluid System

Figure 77. Viscosity of Cyfloc 4000 (anionic) Fluid System

Figure 78. Viscosity of Cyfloc 4000 (anionic) Fluid System

Figure 79. Viscosity of Cyfloc 4500 (non-ionic) Fluid System

Figure 80. Viscosity of Cyfloc 4500 (Non-Ionic) Fluid System

V. FLUID LOSS TEST RESULTS

Fluid-loss tests are run with both low permeability man-made cores and filter paper in a Baroid high temperature fluid-loss tester shown in Figure 81.

Figure 81. High Temperature Fluid-Loss Tester

In these tests with various fluid systems containing one or a combination of fluid-loss materials, the leakoff rate is measured for that system at geothermal temperatures. The control of fluid-loss plays a vital role in proper fracture design and in fracture geometry; therefore, fluid loss must be measured in any fluid system with potential geothermal application.

To minimize fluid leakoff yet maximize the effect of heat blockage and convective cooling, a judicious combination of fluids and fluid-loss additives such as Adomite Aqua, Adomite Mark II, silica flour, and pulverized sea shells or marble (calcium carbonate) has been tested. When the additives are placed in normal frac fluids, they act as temporary plugging agents in the pore spaces of the formation and reduce the rate of leakoff.

To calculate a fluid system's fluid loss, a filtrate volume is measured from the high-pressure Baroid cell. The amount of filtrate (volume) is recorded versus time. From this data and the area of the core, the velocity of the filtrate is calculated for fixed conditions of 500 psi pressure differential at various reservoir temperatures. By rearranging Equation 11 of Section III, we can solve for the fluid-loss coefficient of filter cake buildup on the core (C_w). This is equal to the velocity multiplied by the square root of time as shown below.

$$C_w = v \sqrt{t} \quad (19)$$

$$\text{or } C_w = 0.0328 \frac{m}{2A_c}$$

Figure 82* illustrates the effect of two additives, Adomite Aqua and WAC-II, upon a low concentration Hydroxypropyl Guar Fluid System at low temperature (70°F). HP guar alone has a greater coefficient of fluid loss than when combined with either of the two additives at ambient conditions. Data indicates that of the two additives, Adomite Aqua controls fluid loss more efficiently than WAC-II at the lower concentrations; while at higher concentrations, WAC-II appears slightly more effective. The fluid coefficient (C_w) of HP guar is 0.037 ft/min^{1/2} compared to 0.0012 ft/min^{1/2} and 0.0010 ft/min^{1/2} for Adomite Aqua and WAC-II, respectively, at a 50 lb/1000 gal concentration.

* All data figures are at the end of Chapter V, pp. 114-122.

At slightly elevated temperatures (120°F), pulverized sea shells (calcium carbonate) and WAC-9 (silica flour) have been tested with a guar gel of a 5 lb/1000 gal concentration. Figure 84 shows a fluid-loss control coefficient of 0.026 ft/min^{1/2} and 0.007 ft/min^{1/2} for a 20 lb/1000 gal concentration and values of 0.010 ft/min^{1/2} and 0.009 ft/min^{1/2} for a 10 lb/1000 gal concentration of WAC-9 and pulverized sea shells respectively. At the higher concentration, pulverized sea shells appear to be more effective, but neither WAC-9 or pulverized sea shells appear to differ greatly at the lower concentration. However, as gel concentration is increased as depicted in Figure 85, the fluid loss coefficient further decreases for the higher concentration additives but remains practically unchanged for the lower concentration. This is attributed to the properties of the HP guar since it contains natural fluid loss agents.

A Hydroxypropyl Guar/Biopolymer fluid system (5:1) has been tested at 300°F to determine the fluid loss coefficient. This system used a high concentration gel (83.3 lb/1000 gal) with Adomite Aqua and Adomite Mark II as the fluid loss control agents. Adomite Aqua appears to be the most effective of the two, which is as it should be since the Adomite Aqua is slightly soluble in water, while Adomite Mark II is a resin-based product. The C_w value of Adomite Aqua is 0.002 ft/min^{1/2} while Adomite Mark II is 0.004 ft/min^{1/2} as noted on Figure 85.

Two polyacrylamides fluid systems (PAM) have been tested at 350°F with Adomite Aqua as the sole additive. In Figure 86 fluid loss is substantial when no additive is present with a C_w value of 0.155 ft/min^{1/2} whereas a C_w value of 0.012 ft/min^{1/2} is obtained with a 20 lb/1000 gal concentration of Adomite Aqua. Figure 87 also depicts a polyacrylamide and once again Adomite Aqua proves to be an excellent fluid-loss agent. Data indicates that the properties of the PAM fluid system itself seem to have influenced its fluid loss, and fluid coefficient (C_w) is substantially decreased when a fluid-loss agent is used.

In Figure 88, a biopolymer fluid system (S-88) is illustrated and test data yielded results in which the fluid-loss coefficient value (C_w) cannot be determined. This is a temperature sensitive polymer and shows remarkable fluid-loss control with and without an additive until a finite period of time. After a given time period, however, polymer degradation is rapid, and fluid loss becomes so high that a meaningful C_w calculation is not possible.

Crosslinked fluids also have been tested at 350°F and results show that once again additives decreased fluid loss. Figure 89 depicts a high concentration crosslinked guar system (100 lb/1000 gal) with filter paper as the filter medium. The C_w value is decreased when WAC-10 (fluid-loss agent) is used. However, when compared with Figure 90, which uses a lower concentration HP guar crosslinked system with a low permeable core, it appears not to be as effective. This is due to the greater permeability of filter paper and also serves to stress the importance of utilizing actual formation cores to determine the actual coefficient of fluid loss.

The C_w values obtained with the core are lower, as expected, with the 50 lb/1000 gal gel yielding a value of 0.013 ft/min^{1/2} and a value of 0.008 ft/min^{1/2} when a fluid-loss agent (Adomite Aqua) is used.

In general, the test results show that the coefficient of fluid loss is decreased with the use of appropriate fluid-loss agents. With proper system design, accounting for the C_w factors of the frac fluids along with the various additives, it is possible to control the fluid-loss coefficient and allow the selected fluid system to perform adequately at geothermal conditions. More information is needed to select the proper fluid-loss additive. We need to know how compatible and how soluble the fluid-loss additive is in the fluids of the well chosen for stimulation. These longer term chemical effects will be important for frac fluid clean-up and for effective stimulation.

Figure 82. Fluid-Loss Data

Figure 83. Fluid-Loss Data

Figure 84. Fluid-Loss Data

Figure 85. Fluid-Loss Data

Figure 86. Fluid-Loss Data

Figure 87. Fluid-Loss Data

(b) Polyacrylamide
(42 lb./1000 gal.)
@ 350°F
 $C_w = \text{fr/min}^{\frac{1}{2}}$

No Additive
 $C_w = 0.129$

20 lb./1000 gal.
Adomite Aqua
 $C_w = 0.018$

FLUID LOSS, cc.

TIME $\sqrt{\text{Min.}}$
119

Figure 88. Fluid-Loss Data

Figure 89. Fluid-Loss Data

Figure 90. Fluid-Loss Data

Crosslinked Guar. (50 lb./1000 gal.)
@ 350°F
 $C_w = \text{ft/min}^{\frac{1}{2}}$

No Additive
 $C_w = 0.013$

25 lb./1000 gal.
Adomite Aqua
 $C_w = 0.008$

TIME / MIN.
122

VI. FALLING BALL VISCOMETER TEST RESULTS

In order to calibrate the instrument, the first fluids studied have been two Newtonian fluids, oil and water. Both of these have been tested and terminal settling velocities are calculated at low and high temperatures, respectively. Non-Newtonian water-based gel systems have also been studied in these experiments. Crosslinked and noncrosslinked water-based systems have been tested at high temperatures to find an apparent viscosity behavior versus temperature and to find the degradation point of the fluid system.

A. Theory

Experimental data have been used to develop a correlation relating equilibrium velocity to the settling velocity of a single particle in an infinite fluid. Figure 91 shows a plot of drag coefficient versus the particle Reynold's number. After obtaining the particle Reynold's number ($N_{Re,p}$) and the drag coefficient of a particular particle, the settling velocity can be calculated using the appropriate equation. Also, Figure 91 shows which region of settling is appropriate to describe the settling rate based on particle and fluid properties. In the Stoke's Law Region, settling rate is proportional to the viscosity and in the Intermediate Law Region, settling rate depends on viscosity to the 0.43 power. In the Newton's Law Region, viscosity is only a minor consideration since turbulence is the dominant force. Predictive equations for each of these three zones have been presented in Section III-E as Equations 15, 16 and 17.

Figure 91. Drag Coefficient Correlation

B. MEI Data

Falling ball data measured on MEI's High Temperature Falling Ball Viscometer is shown as a schematic in Figure 92. This instrument measured all the data taken and shown in this section.

Figure 92. Schematic of MEI's Falling Ball Viscometer

The Newtonian fluid, tested first, had characteristic settling rates within the Intermediate and Newton's Law Region. The noncrosslinked non-Newtonian gel yielded results in which the terminal settling velocity was also within the Intermediate Region. The crosslinked version of the same base gel system (HP guar) exhibited somewhat erratic test results due to the nature of the highly viscous gel and because degradation did not occur in a uniform, isotropic

manner. However, the very slow, settling rates measured with crosslinked fluids relegates its behavior in the Stoke's Law Region. At higher temperatures there is a continual degradation of the gel; and at some point (determined by gel type, temperature, and time span), the fluid will lose its non-Newtonian behavior to allow for the determination of static terminal settling velocity. In order to obtain more conclusive data and ascertain properties of these highly viscous cross-linked gels, a dynamic system of stirring, vibration, or rotation needs to be employed with the falling ball viscometer system to place a shear rate on the fluid system inside the pressure chamber which will facilitate settling and help in predicting settling rate properties as described by Novotny.⁶ Future work may require that a controllable dynamic input be used with the system, and full temperature hardening of all wiring is needed.

The terminal settling velocity is determined for both Newtonian and non-Newtonian fluids by measuring certain fluid and proppant properties, i.e., proppant density, fluid density, fluid viscosity, Reynold's number, and power law model parameters (n' , k'). The falling ball viscometer measures the time of fall for the ball; and in Newtonian fluids, this value is used in calculating the Reynold's number. The data taken for most normal frac fluids shows that the N_{Re} is greater than 2, and therefore the settling rate is within the Intermediate Region for both the oil and uncrosslinked gel fluid systems tested. The equation used in determination of terminal settling velocity is Equation 16 from section III-E and is shown below.

$$V_{\infty} = \frac{20.34 (\rho_p - \rho)^{0.71} d^{1.14}}{\rho^{0.29} \mu^{0.43}} \quad (16)$$

The measured fall rate calculation for our falling ball viscometer is given by Equation (20).

$$V_m = 38.1/t_m \quad (20)$$

where V_m is measured velocity (cm/sec)
 t_m is measured time (sec)

Figure 93 is a photograph of the timing of the fall rate for a 0.25 inch steel ball in a 150 cp oil at 75°F. The trace is initiated on release of the electro-magnet and the suspended ball begins its descent. When the ball strikes the bottom of the cell, a pressure transducer is triggered and a timing spike is recorded. The sweep speed is set at 0.5 sec/cm and the fall rate is determined to be 38.1 cm/sec for the 150 cp oil. The calculated N_{Re} is above 2.0 and therefore Eq. 16 is applicable. The calculated terminal settling velocity for the 0.25 inch steel ball in this 150 cp Newtonian fluid at 75°F is 39.9 cm/sec which is well within experimental accuracy.

Figure 93. Time of Fall for Steel Ball in a Newtonian Fluid at 75°F

Data Computation for Figure 93

$$V_m = \frac{38.1 \text{ (ht. of fall cm.)}}{t_m}$$
$$= \frac{38.1 \text{ cm}}{1} = 38.1 \text{ cm/sec}$$

$$N_{Re} = \frac{dV_m\rho}{\mu} = 14.7 \text{ intermediate region}$$

$$V_\infty = \frac{20.34(\rho_p - \rho)^{0.71} d^{1.14}}{\rho^{0.29} \mu^{0.43}} = 39.9 \text{ cm/sec} \quad (17)$$

$$V_m/V_\infty = 95.5\%$$

where V_m = measured velocity
 V_∞ = terminal velocity
 g = gravity
 ρ_p = proppant density
 ρ = fluid density
 d = proppant diameter
 μ = fluid viscosity
 N_{Re} = Reynold's number

The same Newtonian fluid once again is tested under all of the above conditions except for that of temperature. This second test temperature is 120°F, and the terminal settling velocity increased as expected with the thinning of fluid viscosity to 48 cp. The measured terminal velocity is 63.5 cm/sec which is greater than that of the 75°F sample. The calculated terminal velocity (V_∞) is 65.2 cm/sec which is well within experimental accuracy. Figure 94 shows the time of fall of the ball at (120°F) in oil with the sweep speed set at 0.5 sec/cm. The first measured timing spike is at 0.6 sec and shows the increased fall rate.

0.6 sec

Figure 94. Time of Fall for Steel Ball in a Newtonian Fluid at 120°F

The next Newtonian fluid tested is water and again the same phenomenon of fall rates and settling velocities are observed. At 75°F the measured rate of fall for the 0.25 inch diameter steel ball is 76.2 cm/sec with a sweep speed of 0.2 sec/cm which resulted in a Reynold's number of 4937.4 within Newton's Law region, and Equation 17 of Section III-E is employed. When calculated, the terminal settling velocity is 113.7 cm/sec which is greater than measured because start-up effects are excluded. Viscosity prediction in this range is poor as V_m/V is only 67 percent. The ball starts its descent from rest and increases velocity until terminal velocity is reached. This constant velocity is defined where the drag coefficient balances the force of gravity on the ball. The error in terminal velocity measurement is noticed only when fluid viscosity is very low and the settling rate is high within the Newton's Law Region. Compensation or elimination of start-up effects is feasible if instantaneous velocity is measured. However, the present falling ball viscometer with slight modifications is appropriate since

qualitative and relative results are needed, and Newton's Law Region holds little interest because fluids of this type cannot support or carry sand. The majority of good frac fluids will be in the Stoke's and Intermediate Law Regions where the MEI instrument is fairly accurate.

Figure 95 depicts the fall rate of the ball as measured in a Newtonian fluid within Newton's-Law Region.

Figure 95. Time of Fall for a Steel Ball in a Newtonian Fluid at 75°F

Figure 96 is also of water but test temperature has been increased to 350°F, while the variable sweep speed (0.2 sec/cm) and all other parameters are held constant. The measured fall time is 0.48 seconds yielding a measured velocity of 79.4 cm/sec and Reynold's number of 5144.8. Using Equation 17, a computed terminal velocity of 113.7 cm/sec is calculated.

Figure 96. Time of Fall for a Steel Ball in a Newtonian Fluid at 350°F

Non-Newtonian or gelled fluids also have been studied with tests conducted on both noncrosslinked and crosslinked Guar derivative fluids (HP Guar). These non-Newtonian fluids are characterized by having an apparent viscosity that varies with shear rate opposed to Newtonian fluids that maintain a constant viscosity at various shear rates. As previously stated in Section III-E, if settling occurs in a stagnant non-Newtonian fluid, the effective shear rate on the particle is the settling velocity divided by the particle diameter (V/d). This shear rate is then used to calculate fluid viscosity with the Power-Law fluid model. Theoretical terminal settling velocity is calculated utilizing Equation 18 when n' and K' values are known.

$$V_{\infty} = \frac{g(\rho_p - \rho)d^{1/n'}}{18K'} \quad (18)$$

The measured terminal settling velocity for these fluids is still found by the falling ball instrument. Test data for a 45 lb/1000 gal HP Guar noncrosslinked gel at 75°F resulted in a fall time of 1.6 seconds as illustrated below in Figure 97 (sweep speed 0.5 sec/cm).

Figure 97. Time of Fall for a Steel Ball in a Non-Newtonian Fluid at 75°F.

The measured terminal velocity is 23.8 cm/sec while the effective shear rate on the particle is 37.5 sec^{-1} . Assuming that the equation for a Newtonian fluid is valid within Intermediate Law Region for this fluid, an apparent viscosity value may be calculated for the non-Newtonian fluid by utilizing the measured terminal velocity. The falling ball viscometer allows for an approximate determination of apparent viscosity for a non-Newtonian fluid where n' and K' values are not known. The gel is calculated to have an apparent viscosity of 500.1 cp at 37.5 sec^{-1} .

Figure 98 shows the same gel tested at 350°F. It is measured to have an increase in terminal settling velocity with a sweep speed of 0.1

sec/cm used for the trace. The resultant velocity upon analysis is determined to be 64.6 cm/sec with an effective particle shear rate of 101.7 sec^{-1} .

Figure 98. Time of Fall for a Steel Ball in a Non-Newtonian Fluid at 350°F

From Equation 17 the gel is found to have an apparent viscosity of 49 cp. at 350°F and 101 sec^{-1} which is less than that for the same gel studied at room temperature in Figure 97. This is consistent with all previous data, since polymers do degrade with higher temperature and are sensitive to dissolved oxygen and other chemical and physical mechanisms.

Figure 99 is of a 45 lb/1000 gal H.P. Guar crosslinked gel which displayed a fall time of 11.2 seconds (2.0 sec/cm sweep speed) and a measured terminal settling velocity of 3.4 cm/sec for the 0.25 inch steel ball. The effective shear rate is determined to be 5.4 sec^{-1} .

An apparent viscosity value of 46,200 cp at 5.4 sec^{-1} has been calculated (Equation 15) for this crosslinked fluid system at 75°F (test temperature). This is excellent proppant suspension and will result in almost zero proppant settling during a frac job at this temperature.

Figure 99. Time of Fall for a Steel Ball in a Non-Newtonian Fluid. (Crosslinked Fluid at 75°F)

At elevated temperatures (350°F) viscosity breakdown is noticed as predicted, yet test data interpretation revealed that gel breakdown is neither complete nor isotropic, and large viscous portions of fluid still remained after test completion. In Figure 100 the measured settling velocity is not a true or static value, and the data presented in Figure 100 is considered an average. The fall time is 1.5 seconds with a measured terminal velocity of 25.4 cm/sec and the effective shear rate on the ball is 40.0 sec^{-1} . At these conditions an apparent viscosity value of 430 cp has been calculated by Equation 16.

Figure 100. Timing Fall for a Steel Ball in a Non-Newtonian Fluid
(Crosslinked Gel at 350°F)

To more accurately test crosslinked non-Newtonian fluids a dynamic system of stirring, vibration, or rotation needs to be added to the present MEI falling ball viscometer system. This would place a known shear rate on the ball within the pressurized chamber and would allow a more accurate measurement of fluid viscosity.

Figure 101. Ratio of Measured Versus Calculated Settling Rates

As a measure of the accuracy of the high temperature falling ball viscometer, Figure 101 shows a ratio of measured fall velocities to calculated velocity. Out to 70 cm/sec, the instrument measures accurately the fluid's ($\pm 10\%$) terminal settling velocity or apparent viscosity. Below a viscosity of about 15 to 20 cp, this instrument

loses accuracy and can only be used for a relative measure between similar frac fluids. It can assess the effect of temperature quite accurately as far as trends and whether or not a fluid will carry the proppant out into the fracture. Certain improvements in the system are obvious before full use of the instrument can be made for site-specific testing.

VII. LITERATURE SURVEY

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VIII. APPENDIX - TEST PROCEDURE AND OPERATIONS MANUAL

- A. STATIC AGING
- B. FANN MODEL 50C VISCOMETER TESTING
- C. FLUID LOSS TESTING
- D. FALLING BALL VISCOMETER TESTING
- E. API RP39 RECOMMENDED PRACTICE
STANDARD PROCEDURE FOR THE EVALUATION
OF HYDRAULIC FRACTURING FLUIDS.

A. Static Aging

1. Prepare the Polymer Solutions

- a. Amount of materials used**
- b. Mixing procedure**
- c. Preparation of solutions for testing**

2. Measure Initial Properties

- a. Rheology**
- b. pH**
- c. API filtration**

3. Static Age

- a. Loading the cell**
- b. Heating conditions**
- c. Measuring the properties**

1. a. Amount of Materials Used

- (1) 1.5 grams of each polymer is weighed using a triple beam balance.
- (2) 500 milliliters of Houston tap water is added to a Waring blender.

b. Mixing Procedure

- (1) Add slowly 1.5 g of polymer to the water while stirring. The polymer should be added within 15 seconds.
- (2) Adjust mixing speed to highest setting on the blender.
- (3) Mix for 5 minutes.

c. Prepare Solution for Testing

- (1) After 5 minutes of stirring defoam with 2-ethyl hexanol if necessary.
- (2) Pour 250 ml into a pint jar for testing.
- (3) Pour 250 ml into 500 ml static aging cell.

2. a. Rheology

- (1) The rheology measurements are made on a Fann Model 35-A Viscometer.
- (2) Place a recently stirred sample in a suitable container and immerse the rotor sleeve exactly to the scribed line. (Tighten lock nut on platform.)
- (3) With the motor running and the motor speed switch in the high position, push the gear shift all the way down (600 rpm). Wait for the dial reading to reach a steady value and record the 600 rpm reading. (NOTE: The time required to reach a steady value is dependent upon the solution characteristics.)
- (4) With the gear shift still all the way down, switch the motor speed switch to the low position (300 rpm) and record the steady dial reading.
- (5) With the gear shift all the way up, switch the motor speed to the high position (200 rpm) and record the steady dial reading.
- (6) Measure the 100 rpm reading by switching the motor speed to the low position.
- (7) Measure the 6 rpm reading by placing the gear shift to the middle position and the motor speed switch to the high position.
- (8) Measure the 3 rpm reading by moving the motor speed switch to the low position.

- (9) In order to measure the 10 second gel strength (at 3 rpm) pull the gear shift to the middle position and shut the motor off. Then allow the solution to remain quiescent for 10 seconds. After 10 seconds turn the motor on to the low speed position and measure the maximum dial deflection before the gel breaks.
- (10) Restir the sample at high speed before allowing it to remain quiescent for 10 minutes. Repeat the measurement as before and report the maximum dial deflection as the 10 minute gel strength.
- (11) Report the results as follows:
 - (a) Plastic viscosity, cp = rpm reading minus 300 rpm reading.
 - (b) Yield point, lb/100 sq ft = 300 rpm reading minus plastic viscosity.
 - (c) 10 second gel strength, lb/100 sq ft = direct dial reading.
 - (d) 10 minute gel strength, lb/100 sq ft = direct dial reading.
 - (e) Apparent viscosity, cp = $\frac{600 \text{ rpm reading}}{2}$
- (12) Sleeve and bob should be washed and dried after each operation.
- (13) Keep rheometer upright when cleaning so that water does not get into the bearings.

b. pH

- (1) Place a sample into a convenient container.
- (2) Place the probe of the pH meter into the sample.

- (3) Turn the selector switch on the meter to "pH." Read the pH on the dial.

c. API Filtration Test

- (1) Assemble the following dry parts in this order: base cap, rubber gasket, screen, a sheet of filter paper, rubber gasket, and cell.
- (2) Fill cell to 1/2" from the top. (This is necessary only to conserve CO₂ gas.) Place unit in filter press frame.
- (3) Check top cap to insure rubber gasket is in place. Place the top cap on the cell and secure the unit in place with the T-screw.
- (4) Place a clean, dry graduated cylinder under the filtrate exit tube.
- (5) Apply 100 psi pressure to the cell and set a watch for 30 minutes.
- (6) At the end of 30 minutes, release the pressure and record the volume in ml of filtrate collected.
- (7) If the sample allows fluid to pass very rapidly, report this as having no filtration control.
- (8) Clean and dry all parts. Reassemble.

3. a. Load the Cell

- (1) Place 250 ml of solution into a 500 ml static aging cell.
- (2) Apply 100 psi after tightening screws on cell top. Close valve stem securely.

b. Heating Conditions

- (1) Preheat oven for 300°F.
- (2) Place sample into oven and allow to remain for 3 hours.

c. Measurement of the Properties

- (1) Cool the cell to room temperature.
- (2) Measure the rheology, pH, and API filtrate as before in Section II, A, B, C.

B. Fann Model 50C Viscometer Testing

1. Prepare Polymer Solutions
 - a. Amount of materials used
 - b. Mixing procedure
 - c. Preparation of solutions for testing
2. Test on Fann Model 50C Viscometer
 - a. Obtain rheology on Model 35A viscometer
 - b. Obtain rheology on Model 50C viscometer

1. a. Amount of Materials Used

(1) Table A shows the samples and the concentrations to use for each.

Table A

<u>Sample</u>	<u>Concentration, lb/1000 gal</u>
Hydroxypropyl Guar	25.0
Hydroxypropyl Guar	83.3
Hydroxypropyl Cellulose	41.6
Hydroxypropyl Cellulose	83.3
Sodium Polyacrylate	83.3
Guar Gum	83.3
Xanthan Gum	83.3
Proprietary Additives	25.0
Proprietary Additives	83.3
Proprietary Additives	85.0
Mixture 5:1 of Hydroxypropyl Guar and Xanthan Gum	83.3

(2) Table B shows the additives and the concentrations to use for each.

Table B

<u>Sample</u>	<u>Concentration</u>
Oxygen Scavenger	11.8 lb/1000 gal
Methyl Alcohol	50 gal/1000 gal
Methyl Alcohol	100 gal/1000 gal

1. b. Mixing Procedure

- (1) Mix the solutions with the following procedures:
 - (a) Very fast with a Waring blender for 5 minutes.
 - (b) Slowly with a Waring blender for 30 minutes.
 - (c) Very slowly on a Premier Dispersator for 30 minutes.
 - (d) Very slowly on a Premier Dispersator for 90 minutes.
- (2) The hydroxypropyl guar samples require the 90 minute slow stir.
- (3) Most other polymers can be dispersed in 30 minutes.

c. Preparation of Solutions for Testing

- (1) If the high blender speed mixing is used, defoamer is required.
- (2) After mixing, obtain rheology on Fann Model 35A viscometer.

2. a. Obtain Rheology on Fann Model 35A Viscometer

- (1) Refer to static aging procedure section 11A.
- (2) The 600 rpm and 300 rpm readings are used to calibrate the Model 50C viscometer.

b. Obtain Rheology on Model 50C Viscometer

(1) Calibration

- (a) Measure viscosity of sample using Md1 35A.
- (b) Turn main switch of 50C to CAL. 15-20 minutes beforehand.
- (c) Depress power button or recorder-switch on back of recorder should be on 50C position.
- (d) Remove sample cup, bob, and expansion fitting from 50C--clean these parts with solvent and dry.
- (e) Pour sample into sample cup--remove excess with fill gauge.
- (f) Reassemble components into 50C. Tighten each part to ensure alignment.
- (g) Adjust temperature scale on chart using Y2.
- (h) Depress shear rate CAL button, adjust blue pen to zero on chart using Y1.
- (i) Release switch and adjust blue pen to zero on chart using Shear Stress Zero.
- (j) Turn main switch to OPER, mode to man.
- (k) Increase speed slowly to 600 rpm with rpm control and depress calibrate button. Use rate span knob to set rpm setting on graph.
- (l) Repeat # k for 300 rpm.
- (m) Reduce shear rate to 0 rpm. Turn main switch to CAL and use Zero stress knob to zero graph.
- (n) Turn main switch back to OPER and then increase shear rate slowly to 600 rpm. Use the shear stress span knob to set shear stress (dial reading obtained from Model 35A). Repeat at 300 rpm.
- (o) Double check h-n.
- (p) Heater power to OFF.

- (q) Cooling water to 0.6 to 0.8 gal/min.
 - (r) Set range--usually 50% but can be adjusted anytime during test.
 - (s) Select desired chart speed on chart recorder, usually 0.2 in/min.
- (2) Viscosity (shear stress) is to be monitored at a constant shear rate (rpm) as follows:
- (a) Main switch to MAN.
 - (b) Function switch to rpm.
 - (c) Increase rpm to whatever value is to be used for test.
 - (d) Adjust heatup time and heater controls. Time is 60 minutes. Temperature is 300°F. If any heat is to be used at all, the system needs to be pressurized, usually with nitrogen. Use 100 psi.
 - (e) The bath temperature can be monitored by switching the function switch to bath °F and the range switch to °F.
 - (f) The shear rate can be increased or decreased at any point during the test by simply adjusting the rpm control knob.
 - (g) The system is cooled down by running cooling water through the heater assembly.
 - (h) On tests up to 400°F, set heater control for 400°F and set timer for 80 minutes. Use 400 psi of pressure.
- (3) Run a shear stress--shear rate curve (rheogram) at room temperature, 100°F, 200°F, 300°F, 300°F after 1 hour at 300°F, and after cool down to 100°F.
- (a) The mode is set to auto or hold and set rpm control knob to 600 rpm. Adjust ramp time to wanted value, usually 1 minute.

- (b) Select some range %, usually 25% - 50%.
- (c) Push the reset button to clear controls.
- (d) Then push UP to initiate run. If in auto mode, speed will increase to max value set on rpm control knob (600 rpm) and then decrease back to zero.
- (e) If in hold mode, speed will increase to max value and remain there until the down button is depressed.
- (f) Pushing the down button at any time will immediately initiate deceleration.
- (g) Pushing the stop button at any time will hold speed at the rpm achieved at the time this button was depressed. Then the up or the down button must be pushed to finish the program.
- (h) Pushing reset will stop program and clear the controls.

C. Fluid-Loss Testing

1. Prepare Filter Disc for Test
 - a. Affix disc to core holder
 - b. Curing
2. Obtain Permeability of Filter Disc
 - a. Cell set up
 - b. Testing
 - c. Calculation
3. Prepare Polymer Solutions
 - a. Amount of materials
 - b. Mixing
4. Test for Fluid Loss
 - a. Cell set up
 - b. Testing
 - c. Filter disc cleaning
 - d. Fluid-loss testing

1. a. Affix Disc to Core Holder

- (1) Filter discs obtained from Sela Corporation of America. Dimensions: 2.5 diameter, 0.635 cm thick.
- (2) The core holder has dimensions of 5.4 cm O.D. and 4.5 cm I.D. and fits a Baroid high temperature-high pressure filter press cell.
- (3) Dow Corning Silicone Rubber Sealant No. 732-CL11 was used to affix the filter disc to the core holder.
- (4) Put sealant around filter disc. Allow to cure 3-4 hours then apply a second coat to assure a good seal.

b. Allow to cure 16 to 24 hours before testing.

2. a. Cell Set Up

- (1) Place 450-500 ml of distilled water in a filter press cell. Keep downward valve stem closed.
- (2) Place core holder with attached filter disc into cell.
- (3) Place cell bottom on cell. Tighten allen screws.
- (4) Tighten valve stems.
- (5) Place into cell holder.

b. Testing

- (1) Place 18 psi on top valve stem. Open to cell.
- (2) Allow 50-100 ml of water to run through bottom valve stem.
- (3) Measure the rate (ml/sec) of flow through filter disc over about a 100 ml volume.
- (4) Repeat #1-3 for a pressure of 9 psi.

c. Calculations

- (1) Use the following equation to determine the permeability, K:

$$K = \frac{q \mu L}{A P} \times 1000, \text{ in millidarcies}$$

q = rate of flow, cm³/sec

μ = viscosity of water, cp

L = thickness of core, cm

A = area of disc, cm²

P = pressure differential, atm

- (2) Values used are

μ = 1 cp

L = 0.635 cm

A = 5.067 cm²

P = various pressures used between 1.5 atm and 0.61 atm

3. a. Amount of Materials

- (1) Mix the base fluid. Use 4.2 g of hydroxypropyl guar, 0.8 g XC polymer, and 500 ml of Houston tap water.
- (2) The hydroxypropyl guar was obtained from General Mills Chemicals, as Gengel E9P.
- (3) The XC polymers was obtained from Kelco Oil Field Products, as Kelzan XC polymer.
- (4) Add the fluid-loss control agents (0.24%, 1.2 g), as required, after the polymers are in solution.
- (5) Fluid-loss control additives are Adomite Aqua and Adomite Mark II frm the Nalco Chemical Company.

b. Mixing

- (1) Stir the base fluid and fluid-loss control agents together for 60-90 minutes with a Premier dispersator.
- (2) Connect the dispersator to a Powerstat variable auto-transformer to adjust speed. Set on 30-40.

4. a. Cell Set Up

- (1) Set up cell as described in Section C - 4.a. Fluid-Loss Testing

- (2) Place 400-500 ml of base fluid (with fluid-loss control agents as required) in the cell instead of distilled water.
- (3) Preheat jacket to 300°F.
- (4) Place loaded cell into heating jacket.
- (5) Apply a pressure differential of 500 psi to cell, after attaching top and bottom pressurizing units. Keep bottom valve stem closed.

b. Testing

- (1) After 45 minutes, open bottom valve stem.
- (2) Record flow rate (ml/sec).

c. Filter Disc Cleaning

- (1) After cooling, remove core holder from cell. Clean and dry cell.
- (2) Remove filter disc from core holder.
- (3) Soak in concentrated HCl or Aqua Regia 24 hours.
- (4) Soak in distilled water 24 hours. Dry.

d. Fluid-Loss Testing

- (1) Connect the heating jacket to proper electrical outlet (ex. 110,220). Place a thermometer in the thermometer well and preheat the heating jacket to the desired test temperature (ex. 300°F). Adjust the thermostat in order to maintain constant temperature.
- (2) Load the cell with a freshly agitated fluid sample. Care should be exercised not to fill cell closer than 3/4 inch from the top to allow for expansion. Then insert the low permeability core.
- (3) Place the cell into the heating jacket with both top and bottom valve stems closed. Transfer the thermometer to the thermometer well.
- (4) Place the pressure unit on the top valve stem and lock in place. Place the bottom pressure receiver and lock in place. Apply 200 psi pressure to both pressure units with valve stems closed. Open top valve and apply 200 psi pressure to the fluid while heating (~ 45 min. and let temperature stabilize).
- (5) When sample reaches the desired test temperature, open the bottom valve stem and increase the pressure of the top pressure unit to 700 psi to start filtration. Collect filtrate for (60) minutes maintaining temperature $\pm 5^{\circ}\text{F}$. Record the total filtrate volume.
- (6) At the end of test, close both valve stems. Back both T-screws off and bleed pressure from both regulators.

- (7) CAUTION: Filter cell will still contain approximately 600 psi pressure. Maintain cell in upright position and (1) cool to room temperature, maintain upright position (cap down) and (2) loosen the top valve stem slowly to bleed pressure down. Loosen cap locking screws and lift cap out with a rocking motion.

D. Falling Ball Viscometer Test Procedure

1. Prepare polymer solutions as in section two of the appendix and also measure initial Rheology.
2. Place approximately 500 ml. of polymer sample into stainless steel accumulator. This will completely fill the unit. Screw the cover on and connect the pressure line from nitrogen tank to inlet of accumulator. Connect outlet line to falling ball viscometer.
3. Turn on the electromagnet of the viscometer and suspend a steel ball from it (diameter 1/16, 1/8, 1/4" etc.).
4. Install the electromagnet cap onto the top of the viscometer and bolt down securely.
5. At this time pressurize the system (accumulator) via nitrogen which in turn forces polymer into the falling ball viscometer. The viscometer is completely filled with excess polymer remaining in the accumulator.
6. Test pressures are as follows:

R. T. = 300 psi
350°F = 300 psi
500°F = 800 psi

7. Connect the temperature readout to the temperature electrode and if heat is to be applied, connect the heating tape to the rheostat and set accordingly.
8. Load film into oscilloscope camera.

9. Adjust sensitivity and variable sweep of scope accordingly. A high sensitivity and low sweep is needed for an extremely viscous gel, whereas a lower sensitivity and faster variable sweep is needed for a thinner or less viscous gel.
10. Upon release of the electromagnet switch, the circuit is open and a trace is initiated upon the oscilloscope screen. The variable sweep has been predetermined and allows for the calculation of the average terminal velocity. A pressure transducer is triggered by the impact of the ball when settling is complete. This causes a noticeable tic on the trace which is used in velocity calculations.
11. The simultaneous release of the camera shutter and electromagnet open circuit allows for the trace to be photographed. The calculations used are discussed within Section III-E.

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E. RECOMMENDED PRACTICE
STANDARD PROCEDURE FOR THE EVALUATION
OF HYDRAULIC FRACTURING FLUIDS

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RECOMMENDED PRACTICE STANDARD PROCEDURE FOR THE EVALUATION OF HYDRAULIC FRACTURING FLUIDS

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API RECOMMENDED PRACTICE STANDARD PROCEDURE FOR TESTING FRACTURING FLUIDS

Foreword

a. The Subcommittee on Fracturing Fluids, membership of which is listed on the preceding page, was organized as a subcommittee of the Southern District Committee on Production Practice, of the Institute's Division of Production. National-subcommittee status was authorized by the Executive Committee in order to better serve the needs of the oil industry.

The membership of this subcommittee consisted of interested oil- and service-company representatives.

b. This recommended practice was prepared for the purpose of standardizing equipment and testing methods for determining fracturing-fluids properties. The equipment, materials, procedures, and data to be used are usually specified or an alternate method is suggested.

SECTION I PREPARATION OF FRACTURING-FLUID SAMPLE

EQUIPMENT

101. Several types of mixing equipment have been selected to provide the different mixing characteristics that various fracturing-fluid compositions require in order to obtain their proper physical properties.

Description

102. The equipment described following is preferred for preparation of fracturing fluids for evaluation. Different types of fracturing fluids may require the use of one or more of these mixing devices.

Type I (Orbital Beater)

103. The beater for this type mixer shall be fan-type and shall rotate on its axis opposite to the direction in which it moves around the bowl. The device shall be capable of multiple rotation speeds varying from 8 rpm to 200 rpm. A mixer having these required characteristics is exemplified by the Hobart Model C-100 mixer equipped with a flat fan-type beater, as shown in Fig. 1.

Type II (High-speed Blade)

104. This mixing device shall be high-speed blade type capable of rotating at 10,000 rpm or greater. A mixer having these required characteristics is exemplified by the Waring Model CB-2 Commercial Blender, as shown in Fig. 2.

Type III (Rotor-stator)

105. The turbine mixer shall be the rotor-stator type capable of rotating at 1,550 rpm or greater. A

FIG. 1 — TYPE I (ORBITAL BEATER)

FIG. 2—TYPE II (HIGH-SPEED BLADE)

mixer having these required characteristics is exemplified by the Emil Greiner Model 9068 Mixer, Type I, as shown in Fig. 3.

Type IV (Homogenizer)

106. The homogenizing-type mixer shall be capable of imparting a high shearing force to the fracturing fluid. A mixer having these required characteristics is exemplified by Refinery Supply Company's mixer, catalog No. 701805 (as shown in Fig. 4) or the equivalent.

PROCEDURE

Preferred

107. The procedure to be followed in the preparation of the fracturing-fluid sample should be furnished by the vendor and should include the following:

- a. The amount of each component.
- b. The order or method of addition.

FIG. 3—TYPE III (ROTOR-STATOR)

FIG. 4—TYPE IV (HOMOGENIZER)

- c. Time of mixing.
- d. Temperature requirements or limitations.
- e. Type mixing equipment to be utilized.
- f. Storage and handling instructions.

Alternate

108. If a specific mixing procedure has not been provided by the vendor, general procedures, as given in par. 109 to 112, incl., shall be followed. All mixing operations shall be conducted between 75 F. and 90 F. All temperatures are ± 2 F.

Emulsions

109. All of the liquid which forms the external phase of the emulsion shall be placed in the bowl of a Type I mixer. The required amount of emulsifier

is added to the external-phase liquid and agitated until it is intimately dispersed or in solution. The highest speed on the Type I mixer shall be used. A volume of internal-phase liquid equal to the volume of external-phase is then poured into the container and, with continual mixing, is agitated for a total of 15 sec. The remainder of the internal-phase liquid is added uniformly over a period of 2 min. The entire mixture shall then be agitated for an additional 15 sec. If the desired consistency is not obtained, the emulsion should be placed in a Type II mixer and agitated for at least 15 sec or until the desired consistency is obtained.

Gels

110. The base liquid shall be agitated with a Type I mixer. The gelling-agent components shall be dispersed throughout the base liquid as rapidly as possible. The mixture shall be stirred until all solid particles will remain in complete suspension, but for not less than 5 min. At this time the mixture shall be poured into another container and allowed to remain unagitated at room temperature until gelation is complete, but for not less than 20 min.

Soap In Situ Gels

111. The base fluid shall be placed in a Type III mixer and the oil-soluble components added and stirred for 10 sec. The alkali is then added slowly over a period of 15 sec, with continuous stirring. Stirring is then continued for 2 min after each addition.

Crude-oil or Water-additive Systems

112. The base fluid shall be placed in a Type II mixer and slow-speed agitation started. The additives* shall be added slowly to the base fluid over a period of 5 sec, with continuous stirring. Agitation shall continue for 3 min at high speed after all of the additive has been added.

*When additives such as fluid-loss-control agent, accelerators, breakers, etc. are used, they shall be added to the fracturing fluid at this time and dispersed throughout the fluid.

SECTION II

RHEOLOGY

201. The rheological properties of fracturing fluids shall be determined using the following equipment and procedure.

EQUIPMENT

202. A Fann V-G Meter Model 35 shall be used. The range of the instrument may be extended by using optional torque springs available from the manufacturer. The Fann V-G meter is a rotational-type viscometer. Fluid is contained in the annular space between 2 coaxial cylinders (Fig. 5). The outer cylinder or rotor is driven at a predetermined rotational rate and the torque arising from the fluid's viscous drag is exerted on the inner cylinder, or bob. The torque is balanced by a helically wound spring and the deflection is read on a calibrated dial. The sample cup shall be provided with a means for controlling temperatures.

PROCEDURE

203. Operating instructions as furnished by the manufacturer are applicable and shall be followed to insure proper operation of the instrument.

204. To fill the apparatus, the sample fluid (prepared according to Sect. I) and the sample cup shall

be brought to test temperature. The rotor sleeve and the bob shall be brought to test temperature by immersing them in water. (The bob shall not be removed from the viscometer.) The bob, sleeve, and sample cup shall be quickly wiped dry, and the sleeve replaced in the viscometer. The sample fluid shall be promptly poured into the sample cup and, with the rotor turning at 3 rpm, the cup shall be raised until the liquid level is at the designated line on the sleeve. Care shall be taken to insure complete filling of the annular space between the rotor and the bob. Rotation at 3 rpm shall be continued for 2 min.

205. For shear tests dial readings shall be observed at 600, 300, 200, 100, 6, and 3 rpm in that order only. The initial reading at 600 rpm shall be taken after 60 sec continuous rotation. The rotor speed shall be shifted to the lower speeds in turn at 20-sec intervals, and the dial readings shall be made just prior to shifting to the next lower speed.

206. The foregoing series of tests shall be performed at 80, 100, 125, and 200 F. ± 2 F. Test temperature shall not exceed the boiling point of the liquids under evaluation. A separate sample shall be used for each test temperature.

FIG. 5 — FANN V-G METER, MODEL 35

207. The data shall be recorded on a data sheet, shown as Fig. 8, and the following graphs and calculations made from the recorded data.

Method 1 (Bingham Plastic)

208. A flow curve shall be prepared for each test temperature by plotting on *rectangular* coordinate paper:

As ordinate (Y axis)

$$\text{Shear stress (lb force per sq ft)} = \frac{\text{dial reading}}{100} \times N$$

As abscissa (X axis)

Rate of shear (sec^{-1}) from the following table:

Speed, Rpm	Rate of Shear, Sec^{-1}
600	959
300	479
200	320
100	160
6	9.6
3	4.8

Wherein:

N = range extension factor of the torque spring.

A sample plot is shown in Fig. 6.

209. The plastic viscosity (μ_p) is calculated as follows:

$$\mu_p \text{ (centipoise)} = N \times (600\text{-rpm reading} - 300\text{-rpm reading}) \\ = \text{slope of flow curve.}$$

210. The yield point (YP) is calculated as follows:

$$\text{YP (lb force per sq ft)} = (N \times 300\text{-rpm reading} - \mu_p / 100) \\ = \text{intercept of flow curve at zero rate of shear.}$$

FIG. 6 — FLOW DIAGRAM—BINGHAM PLASTIC

Method 2 (General Non-Newtonian)

211. A flow curve shall be prepared by plotting on *logarithmic* coordinate paper:

As ordinate (Y axis)

$$\text{Shear stress (lb force per sq ft)} = \frac{\text{dial reading}}{100} \times N$$

As abscissa (X axis)

Rate of shear (sec^{-1}) from the following table:

Speed, Rpm	Rate of Shear, Sec^{-1}
600	959
300	479
200	320
100	160
6	9.6
3	4.8

Wherein:

N = range-extension factor of the torque spring.

A sample plot is shown in Fig. 7.

FIG. 7 — FLOW DIAGRAM—GENERAL NON-NEWTONIAN

FIG. 8

API FRACTURING FLUID DATA SHEET

(Pads of 50 this sheet are available from the Division of Production, American Petroleum Institute,
300 Corrigan Tower Bldg, Dallas, Texas, at \$1.00 per pad.)

FRACTURING FLUID:

Trade Name _____ Type _____

Composition _____ Preparation procedure _____

Supplier _____ Evaluated by _____ Date _____

RHEOLOGY:

Speed (rpm)	Rate of Shear (sec ⁻¹)	Fann V-G Meter Dial Reading at Indicated Temperature			
		80 F.	100 F.	125 F.	200 F.
600	959				
300	479				
200	320				
100	160				
6	9.6				
3	4.8				
Torque-spring extension factor					

Method 1: Plot rate of shear (abscissa) vs. shear stress (ordinate) on rectangular coordinate paper

80 F. 100 F. 125 F. 200 F.

Plastic viscosity, cp

Yield point,
lb per sq ft

Method 2: Plot rate of shear (abscissa) vs. shear stress (ordinate) on logarithmic coordinate paper

80 F. 100 F. 125 F. 200 F.

Flow behavior index
(dimensionless)Consistency index,
lb-secⁿ per sq ft

SAND SUSPENSION:

Sand fall rate, ft per min				
Temperature, deg F.				

POUR POINT: [] F. FLASH POINT: [] F.

SOLID CONTENT (Volume percent):

BS&W

Asphaltenes

FLUID LOSS:

Name of additive _____; volume percent _____, or lb per gal _____

Test temperature	F.
Test pressure	psi

Time, min	1	4	9	16	25
Fluid loss, ml					

Spurt loss [] Square root time slope []

MATRIX PERMEABILITY DAMAGE:

Fluid loss through core

Test temperature	F.
Pressure differential	psi

Time, min	1	4	9	16	25
Fluid loss, ml					

Spurt loss [] Square root time slope []

Core properties

Formation name or lithology _____ Pore volume _____ ml

Permeability to gas _____ md Permeability to brine _____ md

Reference permeability to oil at connate water _____ md (measured at 500 psi or at _____ psi pressure differential)

(Over)

Permeability damage (at standard or other backflow pressure differentials)

Test temperature, 125 F. or other _____ F.

Clean-up at 10 psi, reported as reference oil throughput in 30 min during backflow at 10 psi differential pressure: _____ ml; _____ pore vol.

Permeability damage _____ percent at 50 psi ΔP or other _____ psi

Permeability damage _____ percent at 100 psi ΔP or other _____ psi

Permeability damage _____ percent at 500 psi ΔP or other _____ psi

COMPATABILITY OF THE FRACTURING FLUID WITH FORMATION FLUIDS

Formation fluid: _____ percent oil, _____ percent brine. Type of brine: produced ☐; synthetic ☐;
8.0 percent NaCl + 2.5 percent CaCl_2 ☐

Formation temperature _____ F. Test temperature _____ F.

Sample 1.

Formation fluid _____ ml oil, _____ ml brine

Total volume after mixing _____ ml

Emulsion type: O/W ☐; W/O ☐; mixed ☐

Visual viscosity before test: very thick ☐; thick ☐;
medium ☐; thin ☐; very thin ☐

Breakout

Time, hours

Emulsion remaining, ml

Emulsion percent = $\frac{\text{ml} \times 100}{\text{total volume}}$

1/2	1	2	24

Visual viscosity of emulsion at end of test: very thick ☐; thick ☐; medium ☐; thin ☐; very thin ☐

Sample 2

Formation fluid _____ ml oil, _____ ml brine

Fracturing fluid _____ ml oil, ml (water) (acid)

Total volume after mixing _____ ml

Emulsion type: O/W ☐; W/O ☐; mixed ☐

Visual viscosity before test: very thick ☐; thick ☐;
medium ☐; thin ☐; very thin ☐

Breakout

Time, hours

Emulsion remaining, ml

Emulsion percent = $\frac{\text{ml} \times 100}{\text{total volume}}$

1/2	1	2	24

Visual viscosity of emulsion at end of test: very thick ☐; thick ☐; medium ☐; thin ☐; very thin ☐

Sample 3

Formation fluid _____ ml oil, _____ ml brine

Fracturing fluid _____ ml oil, ml (water) (acid)

Total volume after mixing _____ ml

Emulsion type: O/W ☐; W/O ☐; mixed ☐

Visual viscosity before test: very thick ☐; thick ☐;
medium ☐; thin ☐; very thin ☐

Breakout

Time, hours

Emulsion remaining, ml

Emulsion percent = $\frac{\text{ml} \times 100}{\text{total volume}}$

1/2	1	2	24

Visual viscosity of emulsion at end of test: very thick ☐; thick ☐; medium ☐; thin ☐; very thin ☐

Sample 4

Formation fluid _____ ml oil, _____ ml brine

Fracturing fluid _____ ml oil, ml (water) (acid)

Total volume after mixing _____ ml

Emulsion type: O/W ☐; W/O ☐; mixed ☐

Visual viscosity before test: very thick ☐; thick ☐;
medium ☐; thin ☐; very thin ☐

Breakout

Time, hours

Emulsion remaining, ml

Emulsion percent = $\frac{\text{ml} \times 100}{\text{total volume}}$

1/2	1	2	24

Visual viscosity of emulsion at end of test: very thick ☐; thick ☐; medium ☐; thin ☐; very thin ☐

Fluids which are designed to react with the formation:

Reaction rock _____ Solubility, percent by weight _____

Weight of reacting rock _____ Reaction time required _____

212. The flow-behavior index (n') is calculated as follows:

$$n' \text{ (dimensionless)} = 3.32 \log \left(\frac{600\text{-rpm reading}}{300\text{-rpm reading}} \right) \\ = \text{slope of flow curve.}$$

213. The consistency index (K') is calculated as follows:

$$K' \text{ (lb-sec}^n \text{ per sq ft)} = \frac{N \times (300\text{-rpm reading})/100}{(479)^{n'}} \\ = \text{intercept of flow curve at} \\ \text{unity rate of shear.}$$

214. An examination of the two flow curves shall be made and the one in which the greatest number of data points lie upon a straight line shall be used to classify the fluid as to rheological type (Bingham plastic or general Non-Newtonian) and to calculate the rheological parameters (plastic viscosity and yield point or flow-behavior index and consistency index).

215. The only instance in which both methods will yield a straight-line flow curve is when the fluid is Newtonian ($YP = 0$ and $n' = 1$). In this instance, the viscosity or the fluid in centipoises shall be recorded under Method 1.

SECTION III SAND SUSPENSION

STATIC TEST METHOD EQUIPMENT

301. A transparent, flat-bottomed cylinder, which is suspended above a mirror in such a way that the bottom of the cylinder can be readily observed, shall be used as the fracturing-fluid container. The cylinder shall be a minimum of 1 ft in length and 1¼ in. in diameter and shall be of uniform diameter.

PROCEDURE

302. The fracturing fluid shall be poured into the cylinder to a depth of 1 ft. After 5 min, sand shall be introduced into the fluid by wetting a ¼-in. glass stirring rod in the test liquid and using the wetted rod to introduce a few adhering grains of sand into the fracturing fluid at its surface. (Note: The sand used for fall-rate tests may be coated with aluminum paint for added visibility and shall have a Krumbein roundness greater than 0.7. One hundred per-

cent of the sand after coating must pass a 20-mesh U. S. Standard sieve and be retained on a 25-mesh U. S. Standard sieve.)

303. The time of fall to the bottom shall be recorded as the time between the touching of the surface of the liquid with the rod and sand, and the observed contact of the sand grains with the bottom of the cylinder. These data shall be recorded on a data sheet shown as Fig. 8. The sand-fall rate in feet per minute shall be calculated from the rate obtained on 2 tests. Sand-fall rate tests shall be conducted at a minimum of 4 temperatures between 80 and 200 F. The sand-fall rate in feet per minute shall be plotted vs. test temperatures. If sand-fall time is greater than 10 min, the rate of fall shall be recorded as less than 0.1 ft per min. (Note: Test temperature should not exceed the boiling point of the liquids under evaluation and shall be held within ± 2 F. of the indicated temperature.)

SECTION IV POUR POINT, FLASH POINT, AND SOLIDS CEMENT

POUR POINT

401. The pour point shall be determined by the procedure outlined in *ASTM D 97-47** standard tests. Record results on a data sheet shown as Fig. 8.

FLASH POINT

402. The flash point shall be determined by the procedure outlined in *ASTM D 96-52* (open cup above 175 F.) or *ASTM D 56-52* (Tag closed tester

below 175 F.) Record results on a data sheet shown as Fig. 8.

SOLIDS CONTENT

403. The solids content shall be determined by the procedure outlined in *ASTM D 96-58T* and *ASTM D 893-52T*. Record results on a data sheet shown as Fig. 8.

*Available from American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

SECTION V FLUID LOSS

501. This test is designed to determine the fluid loss of fracturing fluids containing no propping agents. Further evaluation of fluid-loss characteristics may be required as described in the matrix-damage procedure in Sect. VI.

EQUIPMENT AND MATERIALS

502. The Baroid filter press No. 387-51 or equivalent shall be used. The back-pressure receiver No. 387-2 may be used when applicable. Operating limits of the Baroid filter press No. 387 are 425 F. and 3,000

psi. These limits are for the test cell only. Other components must be separately rated. For tests requiring pressures in excess of 1,500 psi, the press No. 387-51 should be equipped with No. 387-50-06 end caps. The thermometer shall be calibrated to within ± 2 F. Fig. 9 illustrates a filter press of the desired type.

503. Nitrogen gas with pressure exceeding the test pressure shall be used as a pressure source. Baroid's 988 or Whatman's 50 filter paper with a

FIG. 9 — FILTER PRESS

diameter of 2.375 in. shall be used. For reproducible results the filter paper should be kept in a desiccator over anhydrous calcium chloride for a period of 24 hours prior to tests. A Type II mixer (par. 104) with a 1,000-ml container or other mixing devices as described in Sect. I should be used. A graduated container shall be used as a receptacle.

PROCEDURE

504. Test temperature and pressure should relate to the anticipated reservoir conditions. However, 1,000 psi and 125 F. may be used as standard test conditions. When an additive is used, the amount should be specified in pounds of additive per gallon or volume percent of fracturing fluid.

505. The base fluid shall be placed in a Type II mixer and slow agitation started. The additive shall be added slowly to the base fluid over a period of 5 sec, with continuous stirring. Agitation shall continue for 3 min at high speed (unless otherwise specified according to par. 107). Gels and emulsions must be prepared according to the mixing procedures in Sect. I.

506. Place filter cell in heating cylinder with open end up and close bottom valve stem. Pre-heat empty cell and jacket to 130 F. or 5 F. above normal. Pour fluid mixture into pre-heated filter cell to $\frac{1}{2}$ in. from top and position filter paper in place. Close filter cell, including top valve stem, and allow fluid and apparatus to reach desired temperature. The approximate temperature of the fluid mixture may best be determined by using the temperature well in the filter cell while it is in the test position.

507. Remove the filter cell from the heating position and gently invert and swirl 2 or 3 times to insure homogenous mixture. Place filter cell in test position in the heating cylinder, lock in place with locking screw on the side of the heating cylinder, and open top and bottom valve stems immediately. Connect pressure manifold to top valve stem and slowly (but not to exceed 20 sec) apply desired pressure.

508. Report filtration loss in milliliters (to 0.1 ml) at 1, 4, 9, 16, and 25 min at test temperature and pressure as illustrated in Fig. 8. The spurt-loss and fluid-loss square root of time slope can most accurately be determined by plotting the milliliter loss vs. the square root of time on graph paper. By giving the most weight to the 9-, 16-, and 25-min values, draw the best possible straight line through the fluid-loss points to the zero time intercept. The spurt loss may be defined as the value of the zero time intercept. The square-root time slope may be determined by subtracting the spurt-loss value from the value noted at the intercept of the straight line and the 25-min ordinate. Divide this value by 5 for the $\sqrt{\text{time}}$ slope.

509. After the 25-min filtration run is finished, shut off pressure source and bleed off pressure in filter cell. Remove filter cell from cylinder, cool, and loosen cap-locking screws.

SECTION VI

MATRIX PERMEABILITY DAMAGE

601. This test procedure is suitable for determining matrix permeability damage properties of all fracturing fluids. The permeability of a core to a reference fluid is measured both before and after the fracturing fluid to be tested has been flowed through the core under specified conditions of temperature and differential pressure. The core represents a portion of the formation forming the fracture wall. The fracturing fluid flows into the fracture wall and through the core; the reference fluid flows in the opposite direction, i.e., from inside the formation and out through the fracture wall. Comparison of the final permeability and the initial permeability indicates the degree of damage caused by the fracturing fluid to the formation matrix permeability.

EQUIPMENT AND MATERIALS

Materials

602. *Core.* A core sample from the formation to be treated is preferred. However, other cores of a suitable permeability range and of similar lithology, such as cores from another formation or cores from a natural outcrop rock, may be substituted. The length of the core shall be 2 in., and the diameter shall be 1 in.

603. *Water.* A prepared brine solution shall be used to establish connate-water saturation in cores and to measure permeability to brine. A solution of 5-percent sodium chloride, prepared with pure inor-

ganic chemical, shall be used; or, alternatively, a synthesized brine having the chemical composition of formation water, prepared with pure inorganic chemicals. Prior to use, the brine shall be filtered through a porcelain bacteriological filter having a nominal maximum pore diameter of 2.8 microns (as exemplified by the Selas porcelain filter,* 015 porosity.) After filtration, the brine shall be deaerated by vacuum for a period of 15 min.

604. Reference Oil. Any clean, non-plugging oil (such as kerosene) with a viscosity range convenient for laboratory testing or representative of formation oil may be used. Prior to use, the reference oil shall be filtered through a porcelain bacteriological filter having a nominal maximum pore diameter of 2.8 microns (as exemplified by the Selas porcelain filter,* 015 porosity) or through a core of less than 20 md. After filtration, the oil shall be evacuated by vacuum pump for a period of 2 hours. This filtration pre-treatment of the reference oil is essential with most oils because of the large number of pore volumes of oil which must be flowed, without plugging, through the core during the test.

605. Fracturing Fluid. The fracturing fluid shall be prepared according to Sect. I. Some fracturing fluids can be stored for long periods after preparation without significant change in the fluid. However, others must be used fairly soon after preparation. Recommendations on storage properties of the fluid (including a specified maximum time period between preparation and use) should be furnished by the supplier along with instructions for preparation.

*Product of Selas Corporation of America.

Equipment

606. Core Holder. The core shall be mounted in such a way that no bypassing of fluid around the core shall occur. Examples of two methods which have been used successfully are shown in Fig. 10. With the plastic-mount type of core holder, the maximum test temperature and pressure differential vary with the type of plastic used. The rubber-sleeve core holder has been used at test temperatures up to 300 F. and pressure differentials up to 2,500 psi (not simultaneously, however).

607. Flow Apparatus. The apparatus for this test basically must provide for: 1, flow of heated reference oil into one end of the core; 2, flow of heated fracturing fluid into the other end; and 3, instruments for measurement of pressure, temperature, rate of flow, and cumulative volume of fluid throughput. A temperature bath and fluid preheating arrangement must be provided which will maintain the core, core holder, and all fluids entering the core at the specified temperature. A means must also be provided for maintaining constant, controlled back pressure on the flow system. Back pressure is needed to prevent gas in the fracturing fluid from coming out of solution and creating a gas block in the core. A differential-pressure gage is required for accurate measurement of low differential pressures at high system pressures. A schematic flow diagram incorporating these requirements is shown in Fig. 11. All flow lines and gages between the reference oil tank and the core face must be scrupulously cleaned, and precautions must be taken to insure against contamination of reference-oil flow lines by the fracturing fluid. The burettes or graduated cylinders used for collecting outflowing fluid must be capable of measuring volume increments with a precision of 1 percent.

FIG. 10 — CORE HOLDERS

FIG. 11 — SCHEMATIC FLOW DIAGRAM FOR MATRIX PERMEABILITY DAMAGE TEST

608. *Pumping Equipment.* The method of pumping is not specified. However, pumping equipment must be able to furnish both the reference oil and the fracturing fluid to the flow system at a constant specified pressure up to a maximum of 1,500 psi. Because some fracturing fluids must be agitated, a means of agitation must be provided within the pump or displacement tank containing the fracturing fluid. Two basic types of pumping equipment have been used successfully. One is a free-piston—cylinder type with the driving fluid (liquid or gas) being separated from the pumped fluid by the free piston. This type may be used in pumping either the test fluid or the reference oil. Another type of equipment involves an aqueous driving fluid (deaerated) which

displaces reference oil from a tank. The deaerated water is driven by tap water through an expandable, impermeable plastic envelope, and tap water is driven by a Sprague pump powered by compressed air. This second type of equipment can be used only for pumping reference fluids and cannot be used for pumping the fracturing fluid, because the fluid could become contaminated with driving liquid (water) and because agitation of the fracturing fluid would be impractical. Both types of equipment are designed to eliminate the drawbacks of a simpler pumping system where test fluids are driven directly by gas under pressure. Such a gas-driven system is undesirable because the fluids being pumped are saturated with gas at input pressure; and as the pressure

drops during flow through a core, the gas can come out of solution and reduce the subsequent permeability of the core to oil.

PROCEDURE

Core Preparation

609. The core will be dried and weighed as follows:

- a. If rubber-sleeve mounting is to be used, the unmounted core will be dried for 2 hours at 200 F. to 220 F., cooled, and then weighed.
- b. If plastic mounting is to be used, the core will first be dried for 2 hours at 200 F. to 220 F. The core will then be mounted and dried for an additional period of 3 to 8 hours at 140 F. to 150 F. The temperature and drying-time requirements for the additional drying period after mounting may vary, depending upon the plastic used. After drying and cooling, the mounted core will be weighed.

610. Permeability of the core to gas will be measured, preferably at about 4 psi differential, and at room temperature. This measurement may be made either before or after the core is loaded or mounted in the matrix-damage test core holder.

611. Evacuate the core for at least 1 hour with a vacuum pump at a pressure below 1 mm mercury, then introduce brine slowly into the evacuated container, letting the brine imbibe slowly into the core as the brine level rises. As little as 5 min has been used successfully at this stage for covering small cores completely with brine, but a longer period can be used and is considered desirable by some laboratories. Once the core is covered with brine, the container can be vented to atmospheric pressure immediately. Some laboratories prefer to continue evacuation at this stage for 1 to 2 hours. However, under these conditions if the vacuum system is not dead-ended, the brine concentration will be increased. After the core is vented to atmospheric pressure, it should stand for at least 1 hour before the core is removed for weighing.

612. The saturated core is then weighed. The pore volume of the core is calculated from the weight of the brine saturating the core and the brine density.

613. Measure permeability to brine, preferably at about 100 psi differential pressure. The measurement may be made at any convenient input pressure and any convenient temperature. The direction of flow should be opposite to that in which the fracturing fluid will flow. Flow 10 pore volumes of brine before making the permeability measurement. This measurement may be made either in the damage-test apparatus or before the core is loaded into the apparatus, as convenient. Precautions should be taken to insure a liquid-filled, gas-free, oil-free, clean flow system for this measurement.

Test Conditions

614. *Standard API Matrix Permeability Damage Test*

- a. Temperature — 125 F.
- b. Differential pressure across core
 - To establish connate water — 500 psi
 - To flow fracturing fluid — 500 psi
 - To measure return permeability:
 - 10 psi
 - 50 psi
 - 100 psi
 - 500 psi maximum

615. *Alternate Test Run at Anticipated Reservoir Conditions*

- a. Temperature — Any temperature within equipment limitations may be substituted, but the temperature used must be reported.
- b. Differential Pressure — Any differential pressure within equipment limitations may be substituted, but the pressure used must be reported. NOTE: The differential pressure used to establish connate-water saturation should not be exceeded during flow of fracturing fluid or during measurement of return permeability. Otherwise the water saturation of the core may be reduced further and the reference permeability base line will be changed. If the fracturing fluid contains an aqueous phase, the water saturation of the core will be increased when the fluid breaks down, and the return permeability should be compared with reference permeability only at the same differential pressure.

Reference-oil Permeability (Permeability to Oil at Connate Water)

616. The core is loaded into the test apparatus with precautions being taken to insure a liquid-filled, gas-free, clean flow system.

617. The core is then reduced to connate water using filtered, deaerated reference oil. Using standard test conditions, where the differential pressure is 500 psi and the temperature is 125 F., the test procedure is as follows: The direction of flow of reference oil should be opposite to that in which the fracturing fluid will flow. Pressure is built up slowly through the core with the downstream valve closed until the pressure at both ends is 1,500 psig. Then the downstream valve is opened slowly into a 1,000-psig regulated system, and a 500-psi differential pressure is maintained. Reference oil is then flowed until a permeability value is reached such that flow of 10 additional pore volumes of reference oil produces less than 1 percent change in permeability. Permeability should increase continually during this period as a result of reduction in water saturation. If permeability decreases at this stage, plugging by contaminants in the reference oil should be suspected, and flow should be stopped. The oil should be refiltered and evacuated, flow lines should be cleaned, and another core should be used. The equilibrium permeability thus obtained shall be designated as the *reference permeability*.

Injection of Fracturing Fluid

618. The fracturing fluid to be tested will be prepared according to Sect. I. If the fluid is non-viscous and contains additives that fall out, it should be agitated in the pump during flow into the test core.

619. The fracturing fluid will be flowed at 500 psi differential pressure and at 125 F. The pressure is allowed to build up slowly through the core with the downstream valve closed until the pressure at both ends is 1,500 psig. Then the downstream valve is opened slowly into a 1,000-psig regulated system. The 500-psig differential pressure will then be maintained for a period of 25 min.

620. The fracturing fluid passing through the core is collected in a graduated cylinder or burette, and the total effluent (or fluid loss) is recorded after 1, 4, 9, 16, and 25 min.

621. After completion of the 25-min flow period, the downstream valve is closed, and the pressure is allowed to build up to 1,500 psi at both ends of the core. The system is then maintained at this pressure and at 125 F. for 16 to 24 hours.

Back Flow of Reference Oil and Measurement of Return Permeability

622. Filtered, deaerated reference oil will then be flowed in the opposite direction from that in which the fracturing fluid flowed. The oil will be flowed at 125 F. and at the 4 differential pressures listed following, in the order given. Upstream pressure will be maintained at 1,500 psi at all back-flow steps. Differential pressures will be controlled by regulation of the downstream pressure. At each differential-pressure step, flow for the specified time or number of pore volumes, then make the listed measurement at that differential pressure

- a. 10 psi differential pressure for 30 min. Record total volume of reference-oil flow (in pore vol-

umes) during this period at 10 psi differential pressure.

- b. 50 psi differential pressure for 10 pore volumes or for no longer than 30 min. Measure permeability to reference oil.
- c. 100 psi differential pressure for 100 pore volumes or for no longer than 3 hours. Measure permeability to reference oil.
- d. 500 psi differential pressure for 100 pore volumes or for no longer than 3 hours. Measure permeability to reference oil.

Calculation of Matrix Permeability Damage

623. The matrix permeability damage at a given back-flow pressure differential, ΔP , shall be calculated by the following formula:

$$\text{Percent damage at } \Delta P = \frac{\text{reference perm.} - \text{return perm. @ } \Delta P}{\text{reference perm.}} \times 100$$

Reporting of Test Results

624. Test results will be reported in terms of matrix permeability damage, fluid loss through core, and core properties, as illustrated in Fig. 8.

SECTION VII

COMPATABILITY OF THE FRACTURING FLUID WITH FORMATION FLUIDS

Foreword

701. This test is designed to test the emulsion characteristics of the fracturing fluid with formation fluids.

EQUIPMENT AND MATERIALS

702. The equipment for the emulsion tests consists of:

- a. A Type II mixer (par. 104) shall be used. It shall have a standard 1,000-ml container with standard blades of 2 $\frac{3}{4}$ -in. diameter. Blenders having more than one speed shall be operated at the high speed.
- b. Four 250-ml graduated cylinders.
- c. Constant-temperature water bath.
- d. A 20-mesh and a 100-mesh screen.

703. The materials used in the emulsion test are:

- a. The fracturing fluid is prepared according to the procedure in Sect. I.
- b. The formation fluid will be the liquid(s) as produced, i.e., it will consist of formation oil and formation water in the producing ratio. The formation fluid will be without treating chemicals. If formation water is unavailable, a synthetic brine of approximately the same composition as the formation brine may be used. If the composition is unknown, a synthetic brine containing 8.0 percent NaCl and 2.5 percent CaCl₂ by weight may be prepared using reagent-grade chemicals. Samples should be kept sealed from the time of sampling until tests are made.

TEST PROCEDURE FOR FLUIDS WHICH ARE NOT DESIGNED TO REACT WITH THE FORMATION ROCK

704. Four samples of 200 ml each will be prepared and will consist of:

- a. Sample 1 — 200 ml of formation fluid.
- b. Sample 2 — 150 ml of fracturing fluid and 50 ml of formation fluid.
- c. Sample 3 — 100 ml of fracturing fluid and 100 ml of formation fluid.
- d. Sample 4 — 50 ml of fracturing fluid and 150 ml of formation fluid.

705. The fracturing fluid, at room temperature, shall be added to the mixer first and the mixer then turned on for 10 sec. The mixer is then turned off and the formation fluid, at formation temperature, immediately added and the mixer then turned on for 1 min.

706. The emulsion separation is noted as follows:

- a. After the sample is mixed it is poured into a 250-ml graduated cylinder and left in a quiescent state at reservoir temperature and atmospheric pressure. If the bath temperature is other than reservoir temperature, a note on the data sheet (Fig. 8) will be added in which are given the test temperature and the reservoir temperature. The total volume in the graduate is recorded.
- b. The volume of remaining emulsion in the graduate will be recorded on the data sheet at elapsed times (since mixing) of 30 min, 1 hour, 2 hours, and 24 hours. Fig. 8. The percent of emulsion is reported as a percent of the total sample in the graduate.

707. The nature of the emulsion is noted as follows:

- a. A few drops of the emulsion from the mixer may be put in water and a few drops in kero-

sene to determine the type. The type of emulsion will be reported as W/O (water-in-oil), O/W (oil-in-water), or mixed.

- b. The visual viscosity of the emulsion in the graduate at the beginning and at the conclusion of the test will be noted as very thick, thick, medium, thin, very thin.

TEST PROCEDURE FOR ACIDIC FLUIDS WHICH ARE DESIGNED TO REACT WITH THE FORMATION ROCK

708. After the fracturing fluid has been prepared, the quantity prescribed in par. 704 will be reacted with a sample of the formation rock which is greater than 20 mesh and approximately equal to 150 percent or more of the calculated amount necessary (100 ml of 15-percent HCl will react with approximately 22 g of pure limestone or about 20 g of dolomite). If a formation-rock sample is not available for this test, a sample of composition similar to the formation

rock — i.e., oyster shells, crushed limestone, etc. — will be used. The type sample will be recorded as part of the test.

709. The fracturing fluid is reacted, with stirring-rod agitation, for sufficient time for the proposed reaction to reach a methyl orange end point.

710. The reaction temperature will be the anticipated reservoir temperature and the pressure will be atmospheric. If the temperature is other than formation temperature, a note to this effect will be added in which are given the test temperature and reservoir temperature. The reacted sample and all solids will be filtered through a 100-mesh screen into the Waring Blendor. The solids remaining on the screen will be washed with the quantity prescribed in par. 704 of formation fluid into the blendor.

711. The blendor will be turned on for 1 min and the procedure outlined in par. 706 and 707 will be followed.

APPENDIX A

FRICTION LOSS THROUGH TUBULAR GOODS

A1. Two methods are presented for the computation of friction loss through tubular goods. (Sect. II, par. 214). Method 1 utilizes the Bingham plastic approach and the Hedstrom method of analysis. Method 2 utilizes the general Non-Newtonian approach with the Metzner and Reed method of analysis. Inasmuch as both methods require use of the slope of the flow curve, the method in which the majority of the data points are on a straight line shall be used to define the rheological parameters of the fluid and to calculate the friction loss through tubular goods. For the particular instance where both methods yield a straight-line flow curve, the fluid is Newtonian, both methods become identical and the Hedstrom method of analysis shall be used.

METHOD 1 (HEDSTROM)

A2. The pressure drop resulting from friction may be estimated by using the modified Fanning-Reynolds chart (Fig. A1) according to Hedstrom.* Calculations are given in equations (A1) through (A4).

- a. Calculate Hedstrom number.

$$N_{He} = [(3.70)(10^6 \bar{Y}P D^2 \rho) / \mu_p^2] \quad (A1)$$

Wherein:

$\bar{Y}P$ = yield point, lb per sq ft.

D = inside diameter of pipe, in.

ρ = density, lb per gal (ASTM D 70-52)

μ_p = plastic viscosity, centipoises.

- b. Calculate effective fluid velocity.

$$V = 17.15Q/D^2 = 24.5Q'/D^2 \quad (A2)$$

Wherein:

Q = flow rate, bbl per min.

Q' = flow rate, gal per sec.

V = velocity, ft per sec.

- c. Calculate Reynolds number.

$$N_{Re} = [(9.28)(10^2 DV\rho) / \mu_p] \quad (A3)$$

*Hedstrom, Bengt O.A.: Flow of Plastic Materials in Pipes. Ind. Eng. Chem., 44, 551, March (1952).

- d. Read friction factor f from chart (Fig. A1).

- e. Calculate frictional pressure drop.

$$\Delta P_f = 0.0390 L \rho V^2 f / D \quad (A4)$$

Wherein:

ΔP_f = frictional pressure drop, psi.

L = length of pipe, ft.

METHOD 2 (METZNER AND REED)

A3. The pressure drop caused by friction may be calculated by using the modified Fanning-Reynolds chart (Fig. A2) according to Metzner and Reed.* Calculations are given in equations (A5) through (A7).

- a. Calculate effective fluid velocity.

$$V = 17.15Q/D^2 = 24.5Q'/D^2 \quad (A5)$$

Wherein:

Q = flow rate, bbl per min.

Q' = flow rate, gal per sec.

V = velocity, ft. per sec.

D = inside diameter of pipe, in.

- b. Calculate Reynolds number.

$$N_{Re} = [1.86 (D/12)^n V^{2-n} \rho] / K' 8^n \quad (A6)$$

Wherein:

K' = consistency index, lb-secⁿ per sq ft.

n' = flow behavior index, dimensionless.

ρ = density, lb per gal (ASTM D 70-52)

- c. Read friction factor f from chart (Fig. A2).

For values of n' between 0.50 and 1.00 use the upper curve.

For values below 0.500 the lower curve shall be used.

- d. Calculate frictional pressure drop.

$$\Delta P_f = 0.0390 L \rho V^2 f / D \quad (A7)$$

Wherein:

ΔP_f = frictional pressure drop, psi.

L = length of pipe, ft.

*Metzner, A. B. and Reed, J. C.: Flow of Non-Newtonian Fluids. J. Am. Inst. Chem. Engrs., 1, 434 (1955).

FIG. A1 — FRICTION FACTOR — HEDSTROM NUMBER DIAGRAM

FIG. A2 — FRICTION FACTOR — REYNOLDS NUMBER DIAGRAM FOR GENERAL NON-NEWTONIAN FLUIDS, METHOD 2