Recommended Practices on Measuring the Viscous Properties of a Cross-linked Water-based Fracturing Fluid

API RECOMMENDED PRACTICE 39
THIRD EDITION, MAY 1998

American
Petroleum
Institute

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Exploration and Production Department

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FOREWORD

These recommended practices were prepared by the API Subcommittee on Fracturing Fluid Rheology. These practices and procedures were compiled on the basis of several years of comparative testing, debate, discussion, and continued cross-linked fracturing fluid research in the industry concerning the factors that affect cross-linked fracture fluid behavior.

The recommended practices contained in this document are specifically for mixing and testing cross-linked water-based fracturing fluids. Recommended practices are given for two situations:

a. Laboratory Testing: We have specified procedures for comparative testing and for cross-linked fracturing fluid research and development where the work is conducted in a research laboratory. Data developed for use in hydraulic fracture propagation simulators should be measured using the recommended procedures for laboratory testing.
b. Field Testing: We have also developed procedures for testing cross-linked water-based fracture fluids in the field. These procedures were developed to allow personnel to perform quality control of the base polymer solutions and determination of cross-linked gel properties in field applications to verify the quality of treatment fluids before and during actual fracture treatments. The procedures have been developed only for quality control purposes.

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1.1 FRACTURING FLUID RHEOLOGY (VISCOSITY) & THE HYDRAULIC FRACTURING PROCESS

1.1.1 Fracturing fluids play a critical role in the hydraulic fracturing process for increasing the production of oil and gas in porous media such as sandstones, carbonates, and coal seams. Fracturing fluids must have sufficient viscosity to initiate and propagate hydraulic fractures, and to suspend and transport propping agents deep into the created fracture.

1.1.2 Fracturing fluids should possess the following rheologically associated properties:

a. Sufficient *viscosity* to create wide fractures and carry propping agents at high concentrations deeply into the producing formation through the fracture.
b. *Low treating pipe friction* to allow high injection rates in the well tubulars while minimizing surface injection pressures and horsepower requirements.
c. *Shear stability* over the range of shear rates experienced in the pumps, flow lines, wellbore conduits, perforations, and the hydraulic fracture.
d. *Thermal stability* over the range of temperatures experienced during the treatment.
e. *Low to moderate fluid loss properties* so that fluid volumes used to create the desired fracture dimensions are minimized, but with sufficient fluid leak-off to allow the fracture to close at the end of pumping.
f. *Controlled degradability* to allow for moderate to high viscosity during pumping, then breaking to a low viscosity as the fracture closes to allow the fracture fluid to flow back and clean-up.

1.1.3 Water-based, cross-linked fluids are commonly used to fracture treat oil and gas reservoirs. The properties of these fluids can be controlled well enough to provide many of the characteristics desired in a fracturing fluid. For example, cross-linked fracture fluids provide high viscosity at low shear rates in the fracture, but have relatively low friction pressures under the high shear rates in the wellbore during pumping. The high viscosity of the cross-linked gels can be reduced (broken) to much lower viscosities at the completion of the fracturing treatment to allow fluid flowback and fracture clean-up.

1.1.4 Typically, the base polymers for preparing cross-linked gels are the following:

a. Guar gum.
b. Hydroxypropyl guar (HPG).
c. Carboxymethylhydroxypropyl guar (CMHPG).
d. Carboxymethylhydroxyethyl cellulose (CMHEC).

1.1.5 Commonly used cross-linking agents for these base polymers include the following:

a. Borate compounds.
b. Titanate compounds.
c. Zirconate compounds.

1.1.6 Fluid breakdown is achieved using the following, depending on the cross-linked system and formation conditions:

a. Enzymes.
b. Oxidizing agents.
c. Acids.

1.1.7 With a given set of additives, the viscous properties of a gel depend on the shear rate, shear history, and thermal history that the fluid has experienced during the mixing and pumping processes. The viscous properties of a cross-linked water-based fluid will be directly related to how the gel is mixed and tested.

1.1.8 The industry has discovered that the viscous properties of a cross-linked water-based fluid vary widely as a function of test procedures. Consequently, to develop rheological models and reproducible laboratory test methods, standardized testing procedures must be used to ensure that the tests fairly represent the typical fluid behavior during a fracturing treatment, and the tests can be run with repeatable results in a laboratory.

1.1.9 The industry also needs well developed field procedures for testing cross-linked fracturing gels on location prior to and during the fracture treatment. These tests are necessary for quality control monitoring of the fluids that are actually mixed and pumped during a treatment.

1.2 CROSS-LINKED FRACTURING FLUID RHEOLOGICAL BEHAVIOR

1.2.1 Cross-linked water-based fracturing fluids typically exhibit rheological behavior that falls in either the "visco-elastic" or "pseudo-plastic" category. As such, they are considered to be non-Newtonian (i.e., shear stress is not a linear function of shear rate).

1.2.2 In classical rheological terms, these fluids typically do not exhibit power-law behavior (i.e., a graph of log (shear stress) versus log (shear rate) plots as a straight line) over a wide range of shear rates. However, over limited ranges of shear rates, the behavior of typical fracture fluids is such that power-law theory can be used to describe the viscous properties of the fluid mathematically.

1.2.3 With cross-linked gels, fluid viscosity is a function of the shear rate on the fluid at the time the viscosity is measured. These fluids are typically "shear thinning," because the apparent viscosity decreases as the shear rate increases. Shear history and time of exposure to temperature will also significantly influence the apparent viscosity measurements.

1.2.4 Over a limited range of shear rates, the viscous behavior of a cross-linked water-based gel can be adequately described using power-law theory. Power-law theory implies that shear stress is a function of the shear rate raised to a power (called "n," as described below).

1.2.5 Figure 1 depicts a "typical" shear stress vs. shear rate rheogram for cross-linked water-based fracturing fluids over a relatively broad shear rate span. It is obvious that with fluids such as these, the data must be segmented into smaller intervals to apply classical power-law analyses techniques.

1.2.6 Figure 2 shows a segmented portion of the data in Figure 1, that is, in a shear rate range typically encountered in a propagating fracture. The data in Figures 1 and 2 clearly show that a power-law approach can yield an adequate approximation of rheological behavior for engineering purposes only when applied to a limited portion of the total data.

1.2.7 For power-law behavior, shear rate and shear stress can be related by the following equation:

$$\tau = k\dot{\gamma}^n \qquad (1.1)$$

where

τ = shear stress, (psf),
k = consistency index (intercept at 1 $\sec^{-1}$) (lb-$\sec^n$)/ft^2,
$\dot{\gamma}$ = shear rate, $\sec^{-1}$,
n = flow behavior index.

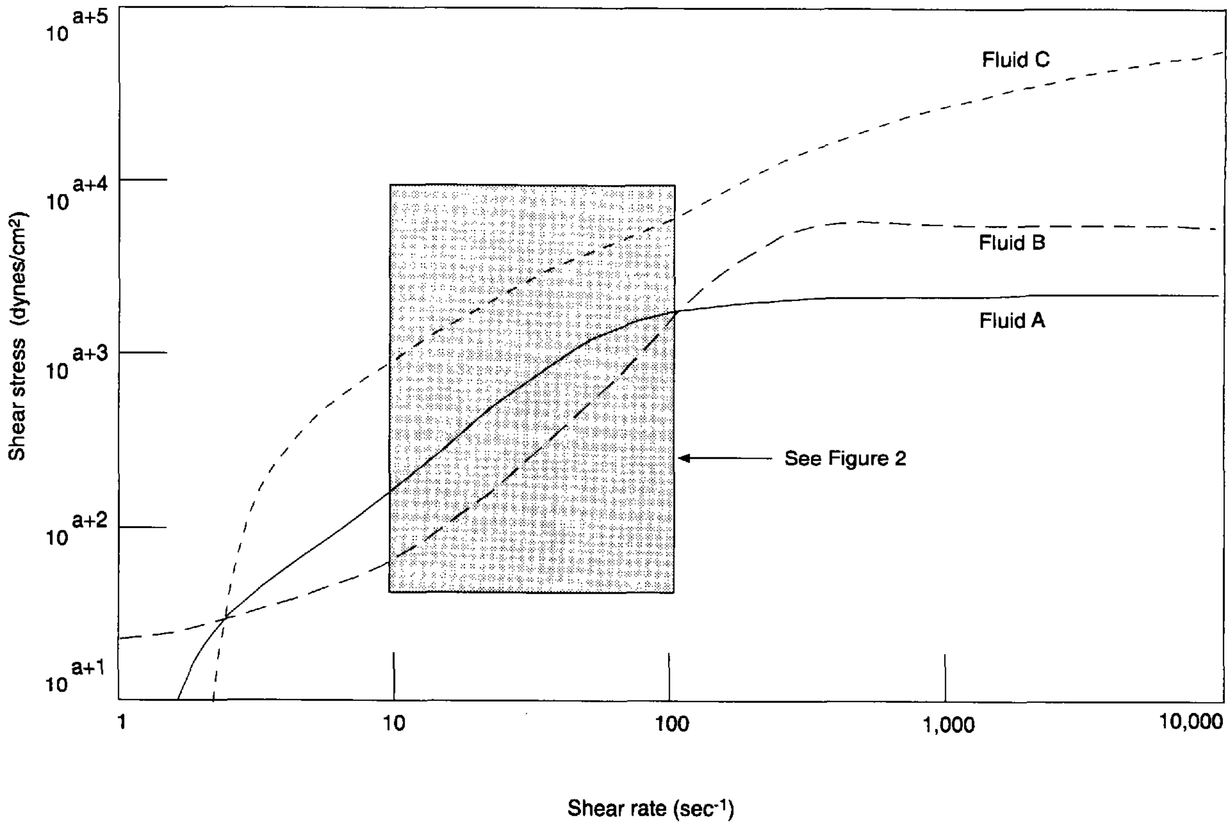

Figure 1—Typical Full Range Shear Stress vs. Shear Rate Rheograms for Cross-linked Water-based Fractured Fluids

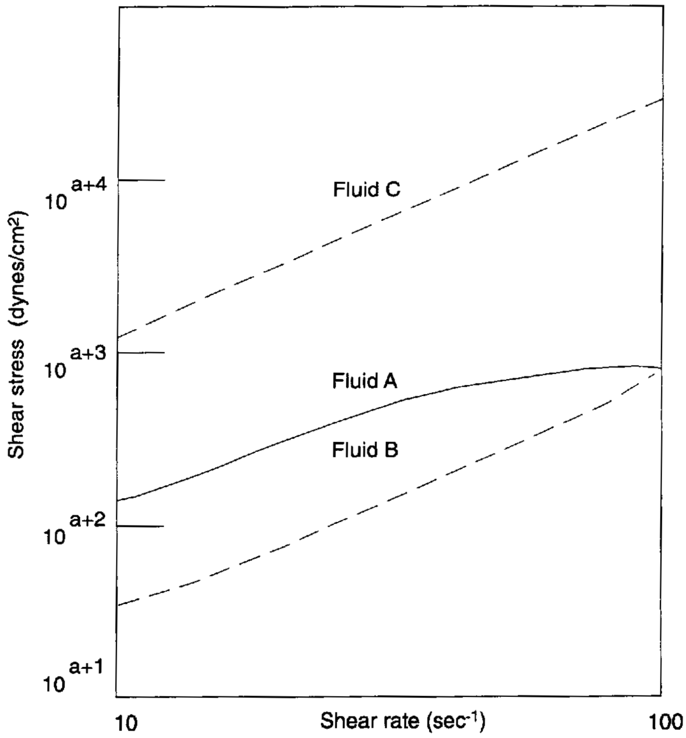

Figure 2—Segmented Portion, Shear Stress vs. Shear Rate Rheograms for Cross-linked Water-based Fracturing Fluids

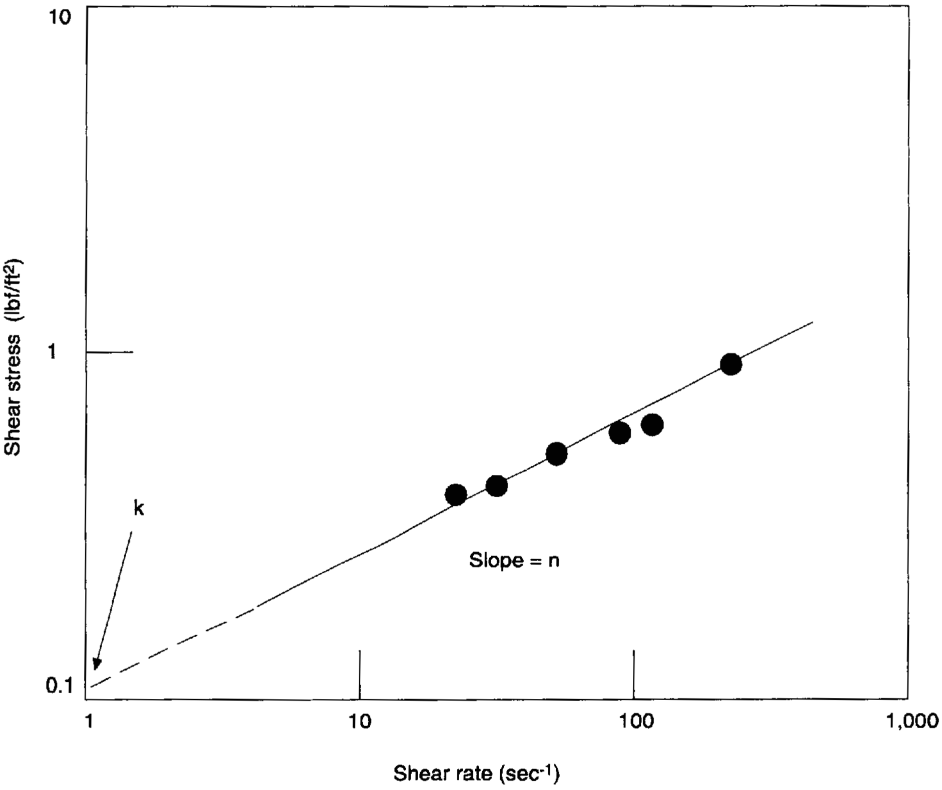

Figure 3—Shear Stress Graph for a Power-Law Fluid

1.2.8 When the values of shear stress are graphed vs. shear rate using full logarithmic paper, a straight line can usually be fit to the data, as illustrated in Figure 3.

1.2.9 Using shear stress and shear rate data to determine *n* and *k* values, the actual power-law viscosity of a power-law fluid can be calculated by the expression:

$$\mu = \frac{47880k}{\dot{\gamma}^{(1-n)}} \qquad (1.2)$$

1.2.10 Typical water-based gels and most other fracturing fluids used today have values of "n" less than 1, and are considered to be "shear thinning" fluids, because the value of apparent viscosity decreases as the value of shear rate increases.

1.3 OBJECTIVE AND LIMITATIONS OF THIS DOCUMENT

1.3.1 Objective

The objective of this document is to provide standard testing procedures for the measurement of certain rheological properties of common cross-linked water-based fracturing fluids used in hydraulic fracturing treatments. This document addresses the steady shear rheological properties of cross-linked gels (without proppant) that exhibit power-law fluid behavior in the shear rate range and the capabilities of the Couette viscometer equipment used in the test procedures.

1.3.2 Limitations

a. The procedures in this document are not intended for proppant-laden fluids, hydrocarbon-based fluids, foams, or emulsions.

b. The procedures are not intended for fluids with rheological properties or behaviors that drastically deviate from power-law behavior.

c. Even some water-based, cross-linked fluids are difficult to evaluate under certain conditions using the procedures in this document. We have discussed several of these problems so the laboratory technician will be aware of the limitations of the recommended test procedures.

d. Through extensive efforts among participating company research laboratories, we know that it is difficult to obtain results that are reproducible when different laboratories test the same or similar fluids. Although this document will not serve to completely identify past differences in test results and fluid properties, API believes this document provides a means to obtain consistent measurements of the viscous properties for cross-linked fluids that can be used in fracture modeling, and for quality control during field operations.

2 References

2.1 STANDARDS

API

Bull 13D	*Bulletin on the Rheology of Oil-Well Drilling Fluids,* second edition, May 1985.
RP 39	*Recommended Practice for Standard Evaluation of Hydraulic Fracturing Fluids,* second edition, January 1983.

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3 Definitions and Nomenclature

3.1 DEFINITIONS

3.1.1 absolute viscosity: Absolute viscosity is the ratio of shear stress divided by shear rate. For a Newtonian fluid, the absolute viscosity is independent of shear rate. For a non-Newtonian fluid, the absolute viscosity is a function of the shear rate at the time the shear stress is measured. This viscosity is then referred to as apparent viscosity and the shear rate must be reported as part of the data set.

3.1.2 breaker: A chemical additive that enables a viscous fracturing fluid to be degraded to a thin fluid that can be produced back out of the fracture. Typical breakers used in water-based polymer fluids are acidic breakers, enzyme breakers, and oxidizing breakers.

3.1.3 Couette viscometer: A Couette viscometer is a rotational viscometer that consists of a concentric cylindrical bob and a cylindrical rotor. The fluid sample is located in the annulus between the bob and rotor. Normally, the rotor is rotated to exert a specified shear rate on the fluid near the surface of the rotor. The shear stress is measured across the gap between the rotor and bob by measuring the torque on the bob.

3.1.4 cross-linked fluid: A cross-linked fluid is created when the polymer in a linear fluid reacts with a metal compound, such as a borate, titanate, or zirconate. The metal compounds link the long chain polymer molecules forming 3-dimensional structures that substantially increase the apparent viscosity and elastic properties of the fluid.

3.1.5 linear fluid: A fluid viscosified by the addition of polymers without the use of cross-linking additives is called a linear fluid. A linear fluid will be non-Newtonian and will also be a pseudo-plastic fluid. Typical polymers used in water-based fracturing fluids are guar, guar derivatives (HPG, CMHPG), and cellulose derivatives (HEC, CMHEC).

3.1.6 Newtonian fluid: Fluids in which shear stress is directly proportional to shear rate (when in laminar flow) are called Newtonian fluids. Water is one example of a Newtonian fluid. The ratio of shear stress divided by the shear rate is the viscosity of the fluid. If modeled as a power-law fluid, then n' is set equal to 1.0 and the viscosity is independent of the shear rate.

3.1.7 non-Newtonian fluids: A fluid that does not exhibit a linear shear stress vs. shear rate relationship in laminar flow is a non-Newtonian fluid. As with Newtonian fluids, the viscosity of a non-Newtonian fluid is the shear stress divided by the shear rate. However, it is called "apparent viscosity" since the calculated viscosity is a function of the shear rate at the time the shear stress is measured.

3.1.8 pseudo-plastic fluids: Pseudo-plastic fluids exhibit a reversible decrease in apparent viscosity with an increase in shear rate. These fluids are also called shear thinning fluids.

3.1.9 rheology: Rheology is the science of the deformation and flow of matter. For fracturing fluids, the important rheological parameter is the apparent viscosity of the fluid as a function of shear rate, temperature, time, and shear history. The science of rheology also includes the measurement of other properties of a fracturing fluid, such as the dynamic moduli as a function of frequency. This document provides recommendations to measure the viscous properties of a cross-linked water-based fracture fluid.

3.1.10 shear history: The viscous behavior of a cross-linked, pseudo-plastic, water-based fluid will be a function of how the fluid is mixed, the sequence of shear rates and temperatures applied, and the duration of shear and of heating. This sequence of shear rates and temperatures applied to the fluid prior to and while making measurements is commonly referred to as the shear history. This shear history must be properly documented by the laboratory personnel for every test. The shear history must be identical for different fluid samples of the same fluid if one desires to generate reproducible laboratory results.

3.1.11 shear history simulator: Equipment constructed in the laboratory to precondition the fluid using specified shear rates, times, and temperatures is commonly referred to as a shear history simulator. The shear history simulator generally consists of mixing equipment, pumping equipment, and coils of tubing to simulate the mixing, pumping, and shear in the tubulars one expects on a typical fracture treatment.

3.1.12 shear rate: Shear rate is the rate at which one particle of fluid is sliding by another particle divided by the distance between them. It is the velocity gradient in a fluid. For Couette viscometers, the shear rate will be a function of position in a flow stream with respect to the wall of the viscometer. The standard unit of shear rate is sec^{-1}.

3.1.13 shear stress: Shear stress is related to the force required to sustain some particular fluid flow. It is a tangential force per unit area and may also be considered as a momentum flux through a given area. The standard unit of shear stress is lb/ft^2.

3.1.14 visco-elastic fluid: Visco-elastic fluids are fluids that exhibit both viscous and elastic behavior. Viscous fluids respond instantly to an imposed shear field. Visco-elastic fluids require time to fully respond to the imposed shear field.

3.2 NOMENCLATURE

All symbols are listed in the Nomenclature section and upon their first use in the text.

Symbol	Meaning
A	Area, cm^2
B1	B1 bob
B2	B2 bob
B5	B5 bob
d	Tubing internal diameter, inches
D	Diameter of pipe, cm
EX-B2	Extended B2 bob
EX-B5	Extended B5 bob
F	Force, dyne
g	Acceleration from gravity, cm/s^2
h	Plate separation, cm
H	Slot height, cm
k	Geometry-independent consistency index, dyne-$\text{sec}^{n'}/\text{cm}^2$
k_f	Slot geometry-dependent consistency index, dyne-$\text{sec}^{n'}/\text{cm}^2$
k_p	Pipe geometry-dependent consistency index, dyne-$\text{sec}^{n'}/\text{cm}^2$
k_v	Couette geometry-dependent consistency index, dyne-$\text{sec}^{n'}/\text{cm}^2$

Symbol	Meaning
L	Length of test section in pipe viscometer, cm
L_b	Bob length, cm
L_{b1}	Length of bob 1, cm
L_{b2}	Length of bob 2, cm
n	Flow behavior index
Δp	Pressure drop over the length of the test section, dyne/cm^2
Q	Volumetric flow rate, cm^3/sec
r	Radius at any point, cm
R	Radius of tubing, cm
R_b	Bob radius, cm
R_{b1}	Radius of bob 1, cm
R_{b2}	Radius of bob 2, cm
R_c	Cup radius, cm
R_{c1}	Radius of cup 1, cm
s	Time, seconds
t	Time, minutes
T	Torque, dyne-cm
T_1	Torque at bob 1, dyne-cm
T_2	Torque at bob 2, dyne-cm
v	Average fluid velocity, ft/sec
V	Volume in Couette region, cm^3
v_n	Newtonian fluid velocity, cm/sec
v_r	Velocity in r direction, cm/sec
v_{slip}	Slip velocity, cm/sec
v_x	Velocity component in x direction, cm/sec
v_z	Velocity component in z direction, cm/sec
v_θ	Velocity component in θ direction, cm/sec
W	Slot width, cm

Greek	
α	Angular velocity, rad/sec
α_1	Angular velocity, rad/sec
α_2	Angular velocity, rad/sec
α_{cor}	Corrected angular velocity, rad/sec
ϕ	Constant = $\partial p / \partial z = \Delta p / L$
$\dot{\gamma}$	Shear rate, s^{-1}
$\dot{\gamma}_{actual}$	Corrected shear rate, sec^{-1}
$\dot{\gamma}_b$	Shear rate at bob, sec^{-1}
$\dot{\gamma}_{b\text{-}r}$	Shear rate at rough bob, sec^{-1}
$\dot{\gamma}_{b\text{-}s}$	Shear rate at smooth bob, sec^{-1}
$\dot{\gamma}_n$	Newtonian shear rate, sec^{-1}
$\dot{\gamma}_{nb}$	Newtonian shear rate at bob, sec^{-1}
$\dot{\gamma}_{nw}$	Newtonian shear rate at wall, sec^{-1}
$\dot{\gamma}_w$	Shear rate at wall, sec^{-1}
τ	Shear stress, dyne/cm^2
τ_b	Shear stress at bob, dyne/cm^2
$\tau_{b\text{-}r}$	Shear stress at rough bob, dyne/cm^2
$\tau_{b\text{-}s}$	Shear stress at smooth bob, dyne/cm^2
τ_w	Shear stress at wall, dyne/cm^2
σ	Standard deviation in viscosity, cp
Ω	Cup rotational speed, rpm
Ω_β	Bob rotational speed, rpm
Ω_χ	Cup rotational speed, rpm
μ	Actual Power-law Viscosity, cp
μ_a	Nominal viscosity for Couette geometry, cp
μ_σ	Apparent viscosity in slot geometry, cp

4 Laboratory Procedures

The recommended practices included in this document cover two situations, which are (a) testing fluids in the laboratory, and (b) testing fluids in the field. This section presents the recommended practices for testing fluids in the laboratory. To obtain reproducible data, a shear history simulator must be used to simulate how fracture fluids are mixed, then pumped down tubular goods at high shear rates prior to entering the hydraulic fracture.

In the laboratory, a coiled capillary tube is used to simulate the shear history in the well tubulars. A Couette viscometer is used to simulate the shear and temperature history of the fluid in the hydraulic fracture. To obtain accurate, reproducible results, these recommended laboratory procedures must be followed with precision, using equipment that is properly maintained and calibrated.

4.1 FLUID PREPARATION AND TESTING

4.1.1 Water-Based Solutions

The procedure to be followed in the preparation of the fracturing fluid polymer solution sample should be specified and should include the following:

a. Description and/or composition of the base fluid, including pH and salt content. Preparation of the fluid should be described, starting with the specified source of the water, such as deionized, tap water from what city, or sea water.
b. The amount of each component to be added.
c. The order and method of addition of each component.
d. Time of mixing (should include temperature and mixing time(s) at one or more mixer speed(s)).
e. Aging or holding time, if required, prior to tests.
f. Volume of fluid required for test.
g. Properties of the polymer solution known to affect the outcome of the rheology testing should be measured prior to the actual test. At a minimum, the following properties should be measured:
  1. Temperature.
  2. Apparent Viscosity (e.g., as per API RP 39).
  3. pH.

4.1.2 Test Procedure for Water-Based Gels

4.1.2.1 Shear History Simulation

a. The fluid pumped through the shear history simulator should experience the following flow conditions:
  1. For fluids to be tested at temperatures less than 200°F, the fluid shear history is to be performed at 675 sec^{-1} ± 67.5 sec^{-1} for a period of 2.5 minutes ± 10 seconds; or
  2. For fluids to be tested at temperatures greater than or equal to 200°F, the fluid shear history is to be performed at 1350 sec^{-1} ± 135 sec^{-1} for a period of 5.0 minutes ± 10 seconds.

b. Operation of the Shear History Simulator.

1. Set the Shear History Simulator exit valves to a waste tank.
2. Begin displacing the gel through the shear loop.
3. Adjust the volumetric flow rate to correspond with the shear rate conditions specified above for the given test temperature (refer to 4.2.2).
4. When the pressure drop through the capillary tube has stabilized, begin on-the-fly injection of the crosslinker and/or other additives.
5. Continue pumping to waste tank until the shear loop has been flushed with at least 2 loop volumes of cross-linked fluid, and the pressure drop in each capillary has stabilized.

4.1.2.2 Viscometer Loading

a. Pre-heat the viscometer bath to the desired test temperature.

b. Attach the B5 or extended B5 bob (refer to 4.2.6) to the Couette viscometer. Check the zero reading. Attach the rotor cup with the quick-connect swivel fitting.

c. Purge the viscometer with nitrogen for 5–10 minutes to remove oxygen which can prematurely degrade polymer during testing at higher temperatures.

d. Attach the sample injection line from the shear history simulator to the rotor cup quick-connect.

e. Establish and maintain a shear rate of 100 sec^{-1} by maintaining cup rotation at 118 rpm.

f. Turn the 3-way valve at the bottom of the rotor cup and inject the required volume of fluid into the rotor cup using a stopwatch to time it (40 mL for extended B5, 45 mL for B5).

g. Immediately turn the 3-way valve back to the "waste" position and disconnect the sample injection flowline from the rotor.

h. Pressurize the rotor to 400 psi with nitrogen.

i. (optional) Perform ambient temperature shear rate ramp (see 4.1.2.3c) if needed to characterize early behavior of fluid during heat-up.

j. Raise the heating bath.

k. Turn crosslinker addition pump off.

l. Flush the shear history loop with at least 2 loop volumes of water to remove residual cross-linked gel in the loop.

4.1.2.3 Viscometer Operation

a. Monitor the sample temperature during heat-up and throughout the test. Sample temperature should reach the desired test temperature (±5°F) within 20 minutes, and stay at that temperature for the duration of the test.

b. Start the first shear rate ramp at 90 percent of the test temperature or at 20 minutes, whichever comes first.

c. Shear rate ramps consist of changing the rotor speed from the 118 RPM base level (B5 bob) for a period of 10, 20, or 30 seconds or longer as required to record a reasonably steady shear stress. However, the ramp should be accomplished as rapidly as possible, especially at high temperature, to be sure the fluid does not degrade much during the ramp. This procedure should be conducted in a stepwise fashion according to the following schedule.

1. 75 sec^{-1} [88.5 rpm]
2. 50 sec^{-1} [59 rpm]
3. 25 sec^{-1} [29.5 rpm]
4. 50 sec^{-1} [59 rpm]
5. 75 sec^{-1} [88.5 rpm]
6. 100 sec^{-1} until next ramp [118 rpm]

d. Shear rate ramps are performed every 30 minutes (after completion of the first ramp series at test temperature) for the duration of the test.

e. Record the shear stress at each value of shear rate.

f. At the end of the test, lower the bath and allow the sample to cool to 100°F or below prior to releasing pressure.

g. Measure pH; observe and record gel condition when removing sample from rotor cup.

4.2 EQUIPMENT REQUIREMENTS

4.2.1 Batch Mixing Equipment

The batch mixing equipment must be capable of providing various levels of mixing intensity. Initially, the polymer must be dispersed rapidly throughout the mixing water without air entrapment. Once the polymer is dispersed, mixing intensity is reduced to a gentle stirring motion to prevent stratification of the hydrating polymer in the mixing vessel.

Depending on the size of the shear history simulator used, the volume of polymer solution needed will vary up to several liters. Up to four liters of solution can be prepared with a high-speed blade mixer such as the Waring Blender®, Model CB6 or equivalent. Variable speed control is recommended for this mixing device. Variable speed control can be obtained by varying the supply voltage with a variable auto transformer. Up to 16 liters of polymer solution can be prepared in a 5 gallon HDPE, open-top container. A variable speed, air driven stirrer such as a Lightning Mixer Model XDA33 or equivalent is adequate for agitation and mixing of this larger volume. Multiple batches of polymer solution can be prepared with the above referenced mixing devices to satisfy the volume requirements of larger shear history simulators. After the polymer solutions are prepared, they are typically transferred to an accumulator or other holding tank associated with the preconditioning equipment.

4.2.2 Shear History Simulator

A Shear History Simulator (SHS) that satisfies the requirements of this Recommended Practices Manual can be generically classified as a tube or pipe flow device that operates in the laminar flow regime. With flow in the single-pass mode, new fluid is continuously injected at the inlet and removed at the outlet of the device. Operation in other flow modes, such as recirculation or oscillatory, is not recommended because these models introduce complicated flow patterns with an unknown impact on final fluid properties. For example, a fluid makes repeated passes through the pump and SHS in the recirculation mode and undergoes continuous flow reversals in the oscillation mode. Turbulent flow within the wellbore could be better simulated if flow within the SHS were also turbulent. It is generally impractical, however, to achieve turbulent flow conditions in laboratory-scale equipment of the type to be used by the SHS.

Operation in laminar flow does offer certain advantages for inter-laboratory testing comparisons. At a specified shear rate, the energy dissipation rate is the same in each laboratory simulator even though they use different tubing or pipe sizes. Therefore, the design and functioning of the SHS can be flexible and still meet the desired preconditioning criteria. To keep the SHS within a reasonable size, a tubing internal diameter (ID) in the range of 0.080 to 0.305 inches (2.0 to 8.0 mm) is recommended. Once the ID of the tubing has been selected, the flow rate and tubing length needed to provide specific preconditioning criteria can be calculated. For example, to maintain a shear rate, $\dot{\gamma}$, for a time, t:

$$\dot{\gamma}_n = 96v/d \qquad (4.1)$$

$$v = \dot{\gamma}_n d/96 \qquad (4.2)$$

$$Q = 96.53\dot{\gamma}_n d^3 \qquad (4.3)$$

$$L = vt \qquad (4.4)$$

where

$\dot{\gamma}_n$ = Newtonian (nominal) shear rate, sec^{-1},
v = average fluid velocity, ft/sec,
d = tubing internal diameter, inches,
Q = flow rate, cc/min,
L = length, feet,
t = time, sec.

Tubing length will be very long and in most cases it will be necessary to coil the tubing to confine it to a small space, such as a laboratory bench top. Coil diameters should be made as large as practical to minimize any additional energy dissipation that might be introduced by this flow geometry.

A "large increase in the resistance to flow" occurs when the Dean Number is > $10^{1/2}$.

$$D_e = \frac{1}{2}Re\sqrt{\frac{R}{r}} \qquad (4.5)$$

where

D_e = Dean number (related viscous, inertial & centrifugal forces),
Re = Reynolds number,
R = tube cross-section radius (D = diam),
r = radius of curvature.

To be sure the radius of curvature has no effect, the following relationship should be honored:

$$\frac{1}{2}Re\sqrt{\frac{R}{r}} < 10^{1/2}$$

The following data were generated by D. Lord for various lab-sized pre-shear loops (for a 40# HPG), using the relationship:

$$r > \frac{Re^2 R}{40}$$

Tube Diameter (in.)	Reynolds Number (*Re*)	Minimum Radius of Curvature (in.)
0.305	573.0	1250.0
0.18	200.0	90.0
0.1175	85.0	10.6
0.085	44.5	2.1

4.2.3 Primary Displacement Pump

Polymer solution pumping requirements will depend on the tubing size selected for the Shear History Simulator (SHS). Required pumping capacities will vary from less than 0.1 liter/minute for the smallest tubing size to more than 3.0 liter/minute for the largest. High-pressure, positive-displacement pumps are required because of the high frictional pressure losses in the SHS tubing. These pumping demands can be satisfied by plunger pumps or plunger-actuated diaphragm pumps that are equipped with a variable stoke length adjustment mechanism and/or variable-speed electric drives. Regardless of the pump actuation type selected (simplex, duplex, or triplex), the pump should have a pulsation damper attached to the discharge. The pulsation damper should be the type that can be recharged with nitrogen through a regulator.

In addition to a plunger type pump, another approach can be taken to supply the fluid flow requirements of a SHS equipped with the smallest tubing (0.125-in. OD, 0.081-in. ID). This alternate approach is based on using an accumulator equipped with a floating piston which separates the polymer solution from a low viscosity working fluid such as oil or water. When the accumulator is filled initially with polymer solution, the floating piston is forced to displace any remaining working fluid from the cell. During the preconditioning step, working fluid is injected into the accumulator at a controlled rate using a very low-capacity, high-pressure, positive-displacement pump which causes the floating piston to displace polymer solution from the cell and into the SHS. High-pressure liquid-chromatography (HPLC) pumps are typically useful in this application because they can handle the clean, low-viscosity working fluids.

Table 1—Description of Equipment for Laboratory Testing

Company	Tubing Diameter (inches)	Sample Injection	Crosslink Injection Geometry	Crosslink Pump	Base Gel Pump	Coil Diameter (inches)	Accumulator
Arco	¼ & ⅜"	Automated	Backwards Tee	Waters L.C.	Jayco Duplex	18	Yes
Stimlab		Visual	Forward Tee	Waters L.C.	Jayco Duplex	30	Yes
Western	⅛	Stop Watch	Backwards Tee	ISCO (200 ml) Syringe Pump		16	Yes
Unocal	⅛	Stop Watch	Right Angle w/static mixer	ISCO (500 ml)		34	Yes
Exxon		N/A	Right Angle w/static mixer	ISCO (200 ml)		10	?
Halliburton	3⁄16	Stop Watch	Forward Tee	Harvard Syringe (suction side)	Milton Roy Duplex	7	Yes
BJ	⅛	Stop Watch	Static Mixer	ISCO		14	Yes
Dowell	⅛	Timed	Right Angle w/static mixer		Eldex Triplex	14	Yes

4.2.4 Crosslinker Addition Pump

Crosslinker requirements depend on the type of cross-linking process used and the chemical composition of the reagent selected to carry out the process. Crosslinker injection rates are generally very small and are dictated by the magnitude of the primary polymer solution flow into the Shear History Simulator (SHS). Low capacity pumps, such as syringe pumps, can be used to inject cross-linking agents into the polymer solution flow stream entering the SHS. A low-pressure syringe pump, such as a Harvard Model 909 (0.00764 to 38.2 ml/minute) or equivalent, can be used to deliver cross-linking agents to the suction side of the high-pressure displacement pump. A high-pressure syringe pump, such as an ISCO Model 500D (0 to 500 ml/minute) or equivalent, can be used to deliver cross-linking agents to either the suction or discharge side of the primary displacement pump.

4.2.5 Inline Mixer

Crosslinker addition to the primary flow is typically through a tee fitting positioned either on the suction or discharge side of the primary displacement pump. The entry point into the tee can be either through a straight-run or side port equipped with a small diameter feed tube (stinger). Chromatographic "low dead volume" tees have been used to introduce crosslinker into the high pressure stream without using a "stinger." To ensure adequate mixing of crosslinker with polymer solution, an inline mixing device is generally used downstream of the tee when it is located on the high pressure side. Commercial inline static mixers are available from a number of sources, including Chemineer, Inc.; KOFLO Corporation; KOMAX Systems, Inc.; TAH Industries, Inc., and others. When crosslinker is delivered to a tee on the suction side, an inline mixer is generally not needed. In these latter situations, plunger motion on the pump cavity combines with the shearing effects present in both suction and discharge check valves to provide intimate mixing of crosslinker with polymer solution.

4.2.6 Couette Viscometer

A rotational type, nonpressurized viscometer, such as a Fann V-G Meter Model 35A, or equivalent, should be used to measure the viscous properties of linear polymer solutions prior to cross-linking. For this purpose it should be equipped with the R1 B1 measurement geometry (see Table 2). This instrument is also used in the field quality control evaluation of cross-linked fluids (Section 5). For this purpose it is equipped with R1 B2 measurement geometry (rotor/bob combination).

Rotational type, pressurized viscometers such as a Fann Viscometer Model 50C, Nordman Model 5001, or equivalent, are used to measure the viscous properties of cross-linked gels at elevated temperatures. These instruments must be equipped with either R1 B5 or R1 B5X measurement geometries. The bob shaft and measurement fixtures should be fabricated from either 316 stainless steel or Hastelloy C.

Table 2—Rotor-Bob Combinations

Dimensions	R1 B1	R1 B2	R1 B5	R1 B5X
Rotor Radius, r_o, cm	1.8415	1.8415	1.8415	1.8415
Bob Radius, r_i, cm	1.7245	1.2276	1.5987	1.5987
Bob Height, L, cm	7.6200	7.6200	7.6200	7.6200
Shear Gap in Annulus, cm	0.1170	1.6139	0.2428	0.2428
Radii Ratio, R_i/R_o	0.9365	0.6666	0.8682	0.8682

4.3 INSTRUMENT CALIBRATION

4.3.1 Shear History Simulator

The critical calibration items for a tubing shear history simulator are flow rate and time. Secondary calibration items may include pressure and temperature measurements.

For fluids to be tested at temperatures less than 200°F, the fluid pre-shear is to be performed at 675 sec^{-1} ±67.5 sec^{-1} for a period of 2.5 minutes ± 10 seconds.

For fluids to be tested at temperatures greater than or equal to 200°F, the fluid pre-shear is to be performed at 1350 sec^{-1} ± 135 sec^{-1} for a period of 5.0 minutes ±10 seconds.

4.3.1.1 Flow Rate

The total flow rate (primary displacement pump plus crosslinker and any other additive pumps) should be checked before loading the Couette viscometer. An acceptable test is timing the fluid into a graduated cylinder using a stop watch. The maximum allowable deviation from the desired flow rate is ±10 percent. If results exceed this value, pumps must be recalibrated or repaired.

4.3.1.2 Time at Shear

Time at shear is controlled by the length of tubing from the point of injection of the crosslinker or activator to the swivel connection connecting the flow line to the viscometer cup. The length of this line must be carefully calculated and measured based on the diameter of the tubing. This measurement is most accurately made before the tubing is coiled. The length can be checked by cleaning and drying out the tubing, then pumping at the 675 sec^{-1} and 1350 sec^{-1} rates and obtaining the time for the fluid to reach the viscometer cup. This time should be within ±10 seconds.

4.3.1.3 Pressure and Temperature

Many shear history simulators are equipped with pressure transducers for determining the pressure response of the fluid during the shear conditioning. These devices should be regularly calibrated according to the manufacturer's instructions. Thermocouples are sometime included in the equipment to measure the temperature of the fluid during cross-linking. It is recommended for standard testing that this temperature is maintained at 77 ± 2°F. Temperature measuring devices should be calibrated according to the manufacturer's instructions.

4.3.2 Couette Viscometer Calibration

The proper performance of a Couette viscometer depends on the proper response of the torque sensing transducer, the rpm control on the drive motor, and accurate temperature control. The operating and calibration manual from the manufacturer should be consulted for specifics of calibration for the specific instrument. The following are some general techniques for calibration checks.

4.3.2.1 Shear Rate (rpm control)

The rpm of the Couette cup must be accurately controlled to apply the desired shear to the fluid. The shear rate is a function of the rotor/bob configuration (see 4.2.6). A minimum calibration procedure is to check this rpm with a strobe light tachometer at the rpm settings for the shear ramps. The maximum deviation from the desired rpm is 5.0 percent.

4.3.2.2 Bearing Condition and Transducer Torque Repose

The transducer which measures the torque on the bob should give a linear response to a Newtonian calibration fluid across the shear rates being tested. A nonlinear response can be the result of a poorly calibrated transducer or worn or contaminated bearings. This response is most easily tested by using a NIST-traceable calibration oil. A suggested procedure is as follows:

a. Place the calibration oil in the instrument cup and measure the temperature with a calibrated thermometer or similar device.

b. Take transducer readings at 0 rpm, and the shear rate steps used to determine the power-law coefficients. Compare the viscosity obtained at the different shear rates with the expected Newtonian behavior. Also, check the torque obtained at the zero rate as well as the coefficient of fit (r^2) for a power-law calculation.

The transducer deflection can be calibrated to torque using a series of calibrated weights. The weight is hung on a thread over a low friction pulley. The end of thread is wrapped 2-$^1/_2$ times around the bob and then taped to the bob surface. The torque (T) exerted on the bob is:

$$T = m\, g\, Rb, \text{ dynes-cm} \qquad (4.6)$$

where m is the mass in grams and Rb is the bob radius (cm). The acceleration from gravity (g) is 980.7 cm/s^2. The torque for different bob radii can then be defined. Table 3 illustrates results for common bob sizes.

Table 3—Calibration Factors for Dead Weight Testing

	Torque (dynes-cm)	
Load (grams)	B5 (1.5987 cm)	B2 (1.2276 cm)
100	1567845.0	120390.7
50	78392.3	60195.4
20	31356.9	24078.1
10	15678.5	12039.1

5 FIELD PROCEDURES

The field procedures in this document have been included for the primary purpose of analyzing the quality of fracture fluids in the field, prior to, and during hydraulic fracture treatments. All field measurements of fluid viscosity should be used to evaluate the overall quality of the fluid, the additives, and the expected performance of the fluid during the time required to pump the fracture treatment and the time required for the fluid to break back to base conditions.

The field measurements should only be used to properly design the quality of the fluid. The field measurements **should not be used to generate the values of apparent viscosity** that are used in the design of a fracture treatment. The values of apparent viscosity measured in the field are not expected to coincide with those measured in the research laboratory unless the exact mixing and testing procedures are followed precisely. Since this will rarely or never occur, then one should not try to compare the two sets of data nor use the field data to design the treatments. The field data should be used solely for quality control purposes, such as modification of buffer, breaker, or crosslinker loading.

5.1 EQUIPMENT REQUIREMENTS

The equipment described in the following section is recommended for the preparation of cross-linked water-based fracturing fluids for evaluation of the fluids in the field.

5.1.1 Mixing Equipment

A blender or laboratory mixing device is required to mix the base gel and the additives. A Waring blender or equivalent is normally used in the field. A rheostat is used to control the speed of the blender to minimize entrainment of air into the system. Polymer solutions can also be prepared using laboratory stirring devices as long as the final viscosity of the solution is within specifications provided by the suppliers of the base gelling agent.

5.1.2 Liquid Additives

Syringes, pipettes, or other quantitative measuring devices are required for metering of liquid buffers, crosslinkers and other additives. The devices should be capable of measurement accuracy as low as 0.01 md.

5.1.3 Dry Additives

An accurate electronic balance is required to weigh dry additives. The balance should be capable of measurement accuracy of 0.001 grams. For example, additive concentrations of 1 pound of additive in 1000 gallons of fluid are commonly used in fracturing fluids. A comparable concentration in 500 ml of fluid would be 0.06 grams of additive.

5.1.4 Basic Fluid Properties

Basic fluid properties, such as temperature and pH, are required to properly document the conditions in the field. Thermometers capable of measuring temperatures from 0°F to 300°F are required for complete coverage. To measure the pH of the fluid, a temperature compensated pH meter should be used in the field. The pH can also be estimated using narrow range pH paper that is in the required range.

5.1.5 Viscometer

A portable Couette viscometer (Fann Model 35 or equivalent) is required to evaluate the viscous properties of the linear and cross-linked fluids. The viscometer should be portable by air, automobile, or van, and should be capable of running using rig power or battery power.

5.1.6 Heating Equipment

Equipment is required to heat up the fluids that are being tested. Commonly, a water bath or a heated cup can be used to control the temperature of the fluid. Either device will be required to run off of either rig power or battery power. It is very important to test both the linear and the cross-linked fluids at elevated temperature to evaluate both the gel stability and the breaker properties of the fluid as a function of the additives used in the fluid.

5.2 PREPARATION OF LINEAR POLYMER SOLUTIONS

The preparation of the linear polymer solutions should follow the recommended procedures of the service company supplying the gel for the fracture treatment. The linear polymer solution is then mixed using the following steps:

a. Add the powder or slurry into a blender or mixer that is being stirred vigorously. However, the rotation of the blender should be controlled by a rheostat so that air entrainment into the fluid is minimized.

b. The polymer solution is then allowed to stir and hydrate for the amount of time specified by the supplier of the polymer. The time in the blender should be recorded. Additives, such as an acidic buffer, should be added if required for proper gel hydration.

c. After approximately 10–15 minutes, the viscosity of the linear polymer solution should be measured at 511 sec^{-1} using a Couette viscometer and a B1 bob.

d. If the apparent viscosity at 511 sec^{-1} is within the specifications set forth by the service company, then additives such as buffers and surfactants should be added to the fluid.

e. The fluid should be mixed at moderate speed in the blender for 2–3 minutes to be sure all new additives have been dissolved and mixed evenly.

f. After all chemicals and additives have been mixed, the pH of the fluid should be measured to determine if the fluid is within the specified pH range.

g. Once the base gel has been mixed and tested with all required buffers and surfactants, then the breakers, gel stabilizers, and crosslinkers should be added to a measured volume of polymer solution.

h. The fluid can then be transferred to the heated cup or to a more sophisticated viscometer (such as a Fann Model 50) for measuring the viscous properties of the fluid. One should minimize the time at which the fluid is at zero shear rate after the cross-linked has been added to the linear gel.

i. Steps a–h are used when the fluid is batch mixed in the field. For continuous mix procedures, the mixing time and order of addition of all additives should coincide with those to be used during the actual treatment.

5.3 SPECIAL FIELD TESTING EQUIPMENT

A Fann Thermos-Cup and/or equivalent is required to test the fluid sample once it has been mixed. It is recommended that the smaller volume heat cups be used to accelerate heat-up time once the gel has been added to the heat cup. The gel should be added to the cup so that there is approximately ½ inch below the top of the heat cup prior to placing the bob and sleeve of the Fann 35 or equivalent into the heat cup. For most tests of cross-linked water-based polymer fluids, a B2 bob should be used in the field tests. On occasion, B1 or B5 bobs can be used to test some cross-linked fluids.

5.4 FIELD TESTING PROCEDURE

a. Transfer the gel containing all additives into the heat cup or into the high temperature viscometer and begin heating the fluid.

b. The heat cup or high temperature viscometer should be preheated to test temperature.

c. The viscometer should be set to rotate @ 100 rpm using a B2 bob. This rotational speed will be equivalent to 37 sec^{-1}, a shear rate that is representative of the shear rate of the fluid in the fracture.

d. The sample should be heated so it reaches the test temperature in approximately 15–20 minutes. The apparent viscosity should be recorded continuously during the heat-up period.

e. After reaching test temperature, the shear stress readings should be recorded every 15 minutes during the test, assuming the test will run for several hours. If data are being measured for a fluid to be used in a short pumping time treatment (one hour or less), then continuous measurements of shear rate should be recorded.

f. Graphs of apparent viscosity vs. time at a constant of shear rate of 37 sec^{-1} should be prepared to determine the quality of the fluids, additives, and mixing procedures.

5.5 DISCUSSION OF COMMONLY OBSERVED PROBLEMS

It is common, particularly in lower temperature testing (150°F and below), for high viscosity fluids such as borates to climb out of the viscometer. One simply has to continue to push the gel back into the viscometer or use a rubber cover to keep the gel in the viscometer. Obviously, we are not trying to measure the absolute viscosity of these visco-elastic fluids. Instead, in these field tests, we are trying to evaluate the viscous properties of the fluid as a function of time and temperature with the breakers and other chemicals to be used on the actual treatment.

On occasion, we will observe values of shear stress which obviously do not represent the actual viscous properties of the gel. Problems can occur when a visco-elastic gel climbs out of the gap onto the top of the bob, or some gels tend to slip along the surface of the bob, especially if oil-based fluid loss additives are used in the fluid. When a closed, high temperature fluid viscometer is being used, one may have to abort and restart the test.

When using a table-top rotational viscometer with a heated cup, one can lower the heat cup, remove the sleeve, and allow the gel to fall into the heat cup. By moving the heat cup up and down, one can attempt to homogenize the sample, and obtain an adequate test.

It is important to differentiate between **fluid problems** and **viscometer testing problems** when testing fluids in the field. On a few occasions, many hours of testing using different additive concentrations and visual observations are required to determine the cause of poor test results. On some occasions, the fluids are difficult to test due to the visco-elastic properties of the fluid or due to slip in the viscometer. On other occasions, changes in fluid chemicals or mixing procedures are required to obtain the desired fluid properties in the field.

6 CALCULATION PROCEDURES FOR VISCOUS PROPERTIES

6.1 GENERAL CONCEPTS

6.1.1 Major assumption: Homogeneous fluid with power-law behavior

$$\tau = k\dot{\gamma}^{n} \tag{6.1}$$

where

τ = shear stress, force/area,
k = fluid consistency index, force-sec^{n}/area,
$\dot{\gamma}$ = shear rate, sec^{-1},
n = flow behavior index, dimensionless.

6.1.2 Quick review of geometry-independent rheology vs. nominal rheology:

a. For a power-law fluid, the shear rate depends on the geometry of the Couette viscometer and the flow behavior index. The shear rate can be approximated using Newtonian behavior and that shear rate is known as nominal Newtonian shear rate. The consistency index determined using the shear stress and the Couette nominal shear rate is known as the machine k_v. The nominal shear rate and geometry-dependent consistency index are corrected using the power-law index and geometry to determine the (actual) power-law shear rate and geometry-independent consistency index.

b. The nominal viscosity (μ_v) calculated using **nominal shear rate and machine k** will be different than the actual power-law viscosity calculated using **power-law shear rate and geometry-independent k,** as shown in Equations 6.3 and 6.4 except when the fluid is Newtonian ($n = 1$). This difference will be greater for Couette geometries having ratios of bob to rotor (cup) radii significantly smaller than one.

It is recommended that geometry-independent rheology be reported and actual power-law viscosities be used.

c. The calculation approach is to use nominal shear rate for data reduction, then correct the fluid consistency index k_v to a geometry-independent k which can be converted to μ_f and k_p for fracture (slot) and pipe flows, if desired.

6.1.3 Units:

a. It is assumed that CGS units will be used for data reduction purposes.

b. Fluid consistency index will be converted into its commonly used English units.

c. Conversions to English units:
Torque: dyne-cm/1.356 x 10^7 = ft-lb_f
Shear Stress: dyne/cm^2/478.8 = $\text{lb}_\text{f}/\text{ft}^2$
k: dyne-sec^n/cm^2/478.8 = lb_f-sec^n/ft^2

6.1.4 Basic equations (CGS units):

$$\tau = \mathrm{k}_v(\dot{\gamma}_n)^n = \mathrm{k}\dot{\gamma}_n \tag{6.2}$$

$$\mu_v = \tau/\dot{\gamma}_n = \mathrm{k}_v\dot{\gamma}_n^{(n-1)} \tag{6.3}$$

$$\mu = \tau/\dot{\gamma} = \mathrm{k}\dot{\gamma}^{(n-1)} \tag{6.4}$$

where

τ = shear stress, dynes/cm^2,
k_v = geometry-dependent (machine) consistency index, dyne-sec^n/cm^2,
$\dot{\gamma}_n$ = nominal (Newtonian) shear rate, sec^{-1},
k = geometry-independent consistency index, dyne-sec^n/cm^2,
$\dot{\gamma}$ = power-law shear rate, sec^{-1},
n = power-law index,
μ_v = nominal power-law viscosity, Poise or dyne-sec/cm^2,
μ = actual law viscosity, Poise or dyne-sec/cm^2.

6.1.5 Calculation of viscosity, using conventional English k units:

$$\mu = 47{,}880 * \mathrm{k}\ \dot{\gamma}^{(n-1)} \tag{6.5}$$

where

μ = actual power-law viscosity, centipoise,
k = geometry-independent consistency index lbf-sec^n/ft^2,
$\dot{\gamma}$ = power-law shear rate, sec^{-1},
n = flow behavior index.

6.1.6 Limitations/problems that may produce erroneous results:

a. Non-power-law over-shear measurement range.
  1. Change in power-law indices vs. shear rate.
  2. Slip (nonhomogeneous) flow.
b. Borate fluids—climbing out of Couette gap.
c. Under- or over-filled Couette viscometer cup.

6.2 COUETTE GEOMETRY

6.2.1 Assumptions:

a. User has a computer for data reduction.

b. Appropriate conversion factor/calibration of **torque to bob shear stress** has been derived.

c. A data table of shear stress-vs-RPM-vs-time has been generated.

6.2.2 Calculate the factor (Factor 1) to convert cup RPM into nominal Newtonian shear rate. Calculate and record the nominal shear rate for each RPM used in the shear sweeps.

$$\text{Nominal shear rate } \dot{\gamma}_n\ (\text{sec}^{-1}) = \text{Factor 1} \times \text{RPM}$$

$$\text{Factor 1} = \pi/15 * 1/(1-(R_b/R_c)^2) \tag{6.6}$$

where

R_b = viscometer bob radius, cm,
R_c = viscometer cup radius, cm.

6.2.3 For each shear sweep time, perform a linear regression of Equation 6.2 in log form

$$\log \tau_b = \log \mathrm{k}_v + n \log \dot{\gamma}_n \tag{6.7}$$

or

$$y = mx + b$$

where

x = $\log(\tau_b)$,
y = $\log(\dot{\gamma}_n)$,
b = $\log(\mathrm{k})$,
m = slope of line,
τ_b = bob shear stress, dynes/cm^2,
$\dot{\gamma}_n$ = nominal shear rate, sec^{-1}.

From the results of the regression, record for each shear sweep:

a. Flow behavior index n = slope m.
b. Machine fluid consistency index, k_v, is calculated from the Y-intercept b, as $\mathrm{k}_v = 10^y$ dynes-sec^n/cm^2.

k_v may be converted into the common units of lbf-sec^n/ft^2 by dividing by 478.8.

c. Regression (goodness-of-fit) coefficient = r or r^2.

6.2.4 For each shear sweep time, calculate the factor (Factor 2) to convert cup RPM to actual power-law shear rate at the bob.

Notes:

1. This will differ from the nominal shear rate factor calculated in step 6.2.2 because it considers the fluid velocity profile as altered by the non-Newtonian behavior of the fluid.
2. Because this factor is a function flow behavior index n, it will be different for each time and shear sweep.
3. This factor will give the user an indication of how the power-law calculation is affecting the results.

Power-law shear rate/RPM = Factor 2

$$\text{Factor 2} = \pi/15 \times 1/[n * (1-(R_b/R_c)^{2/n})] \qquad (6.8)$$

6.2.5 For each shear sweep time, calculate the geometry-independent fluid consistency index, k, from the machine-specific k_v.

Geometry-independent fluid consistency index k

$$= \text{Factor } 3 \times \mathrm{k}_v$$

$$\text{Factor 3} = [n * (1-(R_b/R_{c+})^{2/n})/(1 - (R_b/R_c)^2)]^n \qquad (6.9)$$

The fluid consistency index, k_f, for a fracture (slot) can be calculated as:

$$\mathrm{k}_f = \mathrm{k}\left[\frac{2n+1}{3n}\right]^n \qquad (6.10)$$

and that for a pipe, k_p, is calculated as:

$$\mathrm{k}_p = \mathrm{k}\left[\frac{3n+1}{4n}\right]^n \qquad (6.11)$$

6.2.6 Prepare Table 4, including the following information for each shear sweep performed:

a. Shear sweep time.
b. Fluid temperature.
c. Flow behavior index n.
d. Machine-specific fluid consistency index k_v.
e. Regression coefficient r or r_2.
f. Conversion factor RPM to power-law shear rate.
g. Geometry-independent fluid consistency index k.

6.2.7 Prepare Table 5, including the following information for each shear sweep performed:

a. Shear sweep time.
b. Actual power-law viscosity, cp at 40 sec^{-1}.
c. Actual power-law viscosity, cp at 100 sec^{-1}.
d. Actual power-law viscosity, cp at 170 sec^{-1}.

where

Actual power-law viscosity, cp = Constant * k * $\dot{\gamma}^{(n-1)}$,
k = geometry-independent fluid consistency index,
Constant = 47,880 if k units are lbf-s^n/ft^2,
= 100 if k units are dyne-s^n/cm^2,
γ = power-law shear rate, sec^{-1},
= 40, 100, and 170 sec^{-1}.

6.3 EXAMPLE CALCULATION: COUETTE GEOMETRY WITH STANDARD R1-B5 GEOMETRY

R_b = 1.5987 cm
R_c = 1.8415 cm
L = length = 7.62 cm

6.3.1 Relate the Newtonian shear rate to RPM.

$$\text{Factor 1} = \left(\frac{\pi}{15\left[1-\left(\frac{R_b}{R_c}\right)^2\right]}\right) = \left(\frac{\pi}{15\left[1-\left(\frac{1.5987}{1.8415}\right)^2\right]}\right) = 0.8503$$

$$\dot{\gamma} = 0.8503 \times \text{RPM}$$

Data (for a hypothetical cross-linked HPG):

RPM:	118	88.5	59	29.5	59	88.5	118
$\dot{\gamma}$(sec^{-1}):	100	75	50	25	50	75	100
T(dyne-cm):	12,995	11,264	9,202	6,510	9,202	11,264	12,995

6.3.2 Calculate shear stress using

$$\tau_b = \frac{\mathrm{T}}{2\pi r_b^2 L} = \frac{\mathrm{T}}{2\pi(1.5987)^2(7.62)} = 0.008175\ \mathrm{T}$$

and then perform a linear regression on log τ_b vs. log $\dot{\gamma}_n$ to calculate k_v and n using

$$\log \tau_b = \log k_v + n \log \dot{\gamma}_n .$$

This yields $n = 0.493$ with $r = 0.9993$ and $k_v = 10.94$ dyne-sec^n/cm^2 = 0.02285 lb$_f$-sec^n/ft^2.

6.3.3 Calculate the power-law shear rate (at the bob) using Factor 2:

$$\text{Factor 2} = \left(\frac{\pi}{15n\left(1-\left(\frac{R_b}{R_c}\right)^{2/n}\right)} \right) = 0.973$$

$$\dot{\gamma} = 0.973 \times \text{RPM}$$

6.3.4 Calculate the geometry-independent k using Factor 3:

$$\text{Factor 3} = \left[\frac{n\left(1-\left(\frac{R_b}{R_c}\right)^{2/n}\right)}{1-\left(\frac{R_b}{R_c}\right)^{2}} \right]^{n} = 0.936$$

$$\begin{aligned} k &= 0.936 \times k_v = 0.936 \times 0.02285 \\ &= 0.02138 \text{ lb}_f - \text{sec}^n/\text{ft}^2. \end{aligned}$$

$$k_f = \left(\frac{2n+1}{3n}\right)^n k = 0.0247 \text{ lb}_f - \text{sec}^n/\text{ft}^2 ;$$

$$k_p = \left(\frac{3n+1}{4n}\right)^n k = 0.0239 \text{ lb}_f - \text{sec}^n/\text{ft}^2$$

Table 4—Results, Calculation of Power-Law Parameters

Shear Sweep Time (min)	Fluid Temp (°F)	n	k_v lb$_f$-sec^n/ft^2	r	Factor 1	Factor 2	Factor 3	k lb$_f$- sec^n/ft^2	k_f lb$_f$- sec^n/ft^2	k_p lb$_f$- sec^n/ft^2
30	200	0.493	0.002285	0.9993	0.8503	0.973	0.936	0.02138	0.0247	0.0238

Table 5—Results, Calculation of Actual Power-Law Viscosity at a Shear Sweep Time of 30 Mins

$\dot{\gamma}$ (sec^{-1})	μ (cp)
40	157.7
100	99.1
170	75.5

APPENDIX A—BASIC RHEOLOGICAL CONCEPTS

A.1 Shear Stress and Shear Rate

The relationship between shear stress and shear rate defines a rheological constitutive equation for a homogeneous fluid in steady-state laminar flow. This relationship is used in the momentum conservation equations to allow the calculation of the dynamic variable (pressure drop or torque) as a function of the respective kinematic variable (flow rate or angular velocity).

A.1.1 SHEAR STRESS

The shear stress of a fluid in steady-state laminar flow can be thought of as the resistance force per unit area exerted by one layer of fluid on the adjacent fluid layer as they slide (or shear) by one another. This shear stress force is in the direction of motion and has units of dyne/cm^2 (CGS), Pa (SI), and psi or lb$_f$/ft^2 (English).

The shear stress can be determined quite easily by experiment. Under steady-state laminar flow conditions, a force balance gives for tubular, slot, and Couette (a rotating outer cylinder and a stationary inner cylinder) flows, the following expressions for shear stress as a function of the measured pressure drop or torque:

$$\text{Tubular:} \quad \tau_w = \frac{R\Delta P}{2L} = \frac{6R\Delta P}{L}, \frac{\text{lb}_f}{\text{ft}^2} \qquad \text{(A-1)}$$

$$\text{Slot:} \quad \tau_w = \frac{W\Delta P}{2L} = \frac{6W\Delta P}{L}, \frac{\text{lb}_f}{\text{ft}^2} \qquad \text{(A-2)}$$

$$\text{Couette:} \; \tau_b = \frac{T}{2\pi R_b^2 L_b} = 3.324\times 10^{-4}\frac{T}{R_b^2 L_b}, \frac{\text{lb}_f}{\text{ft}^2} \qquad \text{(A-3)}$$

Here, R is the tubing radius (inches), ΔP is pressure drop through the tubing or slot (in psi), L is the distance in feet of the pressure drop, W is the slot width (inches), R_b is the inner cylinder (bob) radius (cm), L_b is the bob length (cm), and T is the torque on the bob (dyne-cm) exerted by the shear stress. Note that these expressions are independent of the fluid nature and apply even to turbulent flows.

A.1.2 SHEAR RATE

The shear rate of a fluid in steady laminar flow can be thought of as the relative velocity at which one fluid layer is sliding by another layer divided by the distance between them. It is intuitive that the shear stress (defined above) should be related to the shear rate in some fashion. Indeed, using continuum fluid mechanics,[1] it can be shown in general that for homogeneous fluids in steady laminar flow, shear stress is a unique function of shear rate:

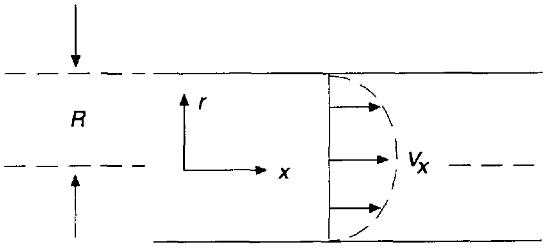
$$\tau = f(\dot{\gamma}) \qquad \text{(A-4)}$$

Usually, this function is written in terms of a viscosity, μ, and shear rate, $\dot{\gamma}$:

$$\tau = \mu\dot{\gamma} \qquad \text{(A-5)}$$

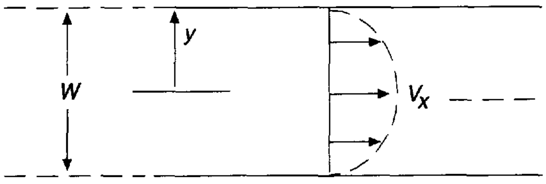
Shear rate is sometimes referred to as the fluid velocity gradient which is correct for tubular and slot flows.

Tubular:



$$\dot{\gamma}(r) = \frac{dV_x}{dr} \qquad \text{(A-6)}$$

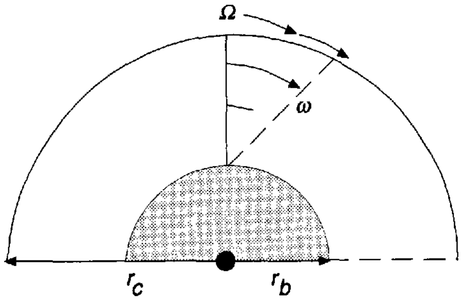
Slot:



$$\dot{\gamma}(y) = \frac{dV_x}{dy} \qquad \text{(A-7)}$$

Here V_x is the fluid velocity in the x direction and r and y are the perpendicular spatial coordinates for the tubular and slot geometries, respectively. The units of shear rate are expressed as 1/sec.

For Couette flow (that between an outside rotating cylinder and a stationary inner cylinder), the shear rate is more correctly thought of as the gradient of the angular velocity multiplied by the radial coordinate at that point.



$$\dot{\gamma}(r) = r\frac{d\omega}{dr} \qquad \text{(A-8)}$$

Here ω is the angular velocity at the radial position, r. Note that this is not the velocity gradient. For example, an axially rotating solid cylinder has a velocity gradient but no shear rate (i.e., no relative sliding of internal material layers).

The shear rates for the above geometries vary with position in the flow field. Tubular and slot flow shear rates are analogous in that shear rate is a maximum at the wall and decreases to zero at the center. For homogeneous Couette flow, the maximum shear rate occurs at the bob (coinciding with the position of the maximum shear stress) and decreases monotonically to a minimum, but finite, value at the outer cylinder (cup). In narrow-gap concentric cylinder viscometers, the shear rate is nearly constant across the gap for homogeneous fluids. As the gap width increases, the shear-rate field becomes more nonuniform.

A.2 Viscosity

A.2.1 GENERAL FORM

For a homogeneous fluid in steady-laminar flow, the shear stress can be expressed as the shear rate times the viscosity. The viscosity is unique for each material, and as such, is considered to be a material function which should not vary with the geometry of the viscometer. The viscosity for some fluids under certain conditions may vary with shear rate. If viscosity is independent of shear rate, then the fluid shear stress is directly proportional to shear rate and the fluid is said to be Newtonian. If the viscosity is a function of shear rate, the fluid is non-Newtonian. Fracturing fluids usually have viscosities which decrease with increasing $\dot{\gamma}$ and are referred to as shear thinning or pseudo-plastic.

The general definition of viscosity is

$$\mu = \tau/\dot{\gamma} \qquad \text{(A-9)}$$

A graph of shear stress vs. shear rate helps to define a fluid. Figure A-1 illustrates such a graph for both a Newtonian fluid and a pseudo-plastic fluid.

For the Newtonian fluid, the ratio of $\tau/\dot{\gamma}$ (the viscosity) is constant.

To determine viscosity, τ and $\dot{\gamma}$ must be measured experimentally. The shear stress is easily determined (e.g., Equations A-1 – A-3), but the measurement of shear rate is not straightforward. For tubular flow, $\dot{\gamma}$ is calculated from measured values of τ and flow rate, Q.

$$\dot{\gamma}_w = \frac{3}{4}\left(\frac{4Q}{\pi R^3}\right) + \frac{1}{4n'}\left(\frac{4Q}{\pi R^3}\right) = \left(\frac{3n'+1}{4n'}\right)\left(\frac{4Q}{\pi R^3}\right) \qquad \text{(A-10)}$$

Here $n' = d\log\frac{\tau_w}{d}\log\left[\frac{4Q}{\pi R^3}\right]$

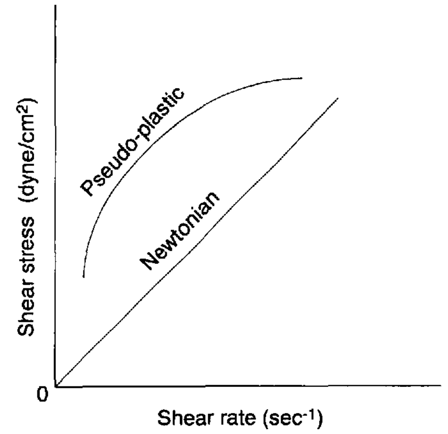


Figure A-1—Graph of Shear Stress Data

For the case when $n = 1.0$, Equation A-10 becomes:

$$\dot{\gamma}_w = \left(\frac{4Q}{\pi R^3}\right) \qquad \text{(A-11)}$$

This is the case for a Newtonian fluid where τ_w is proportional to flow rate and where the resulting shear rate is called the Newtonian or nominal shear rate: $\dot{\gamma}_N$

$$\dot{\gamma}_N = \left(\frac{4Q}{\pi R^3}\right) \qquad \text{(A-12)}$$

For slot flow, shear rate is analogously derived with $\dot{\gamma}_w$ given by:

$$\dot{\gamma}_w = \frac{2}{3}\left(\frac{6Q}{Hw^2}\right) + \frac{\tau_w}{3}\left(\frac{d\left[\frac{6Q}{Hw^2}\right]}{d\tau_w}\right) = \left[\frac{2n'+1}{3n'}\right]\left(\frac{6Q}{Hw^2}\right) \qquad \text{(A-13)}$$

where H is the slot height.

Here
$$n = \frac{d\log\tau_w}{d\log\left[\frac{6Q}{Hw^2}\right]} \qquad \text{(A-14)}$$

and the Newtonian shear rate is:

$$\dot{\gamma}_N = \left[\frac{6Q}{Hw^2}\right] \qquad \text{(A-15)}$$

For Couette flow, the procedure for determining the shear rate is analogous with an added complication. The cup angular velocity, Ω, is written in terms of $\dot{\gamma}$ and τ as:

$$\Omega = -\frac{1}{2}\int_{\tau_b}^{\tau_c}\frac{\dot{\gamma}}{\tau}d\tau \qquad \text{(A-16)}$$

Differentiation with respect to τ_b gives

$$\dot{\gamma}_b - \dot{\gamma}_c = 2\tau_b \frac{d\Omega}{d\tau_b} \qquad \text{(A-17)}$$

However, Equation A-17 does not yield $\dot{\gamma}_b$, but the difference between the two unknown bob and cup shear rates $(\dot{\gamma}_b - \dot{\gamma}_c)$. Krieger and Elrod[3] showed that to extract $\dot{\gamma}_b$, the entire flow curve of Ω vs. τ should be used to evaluate Equation A-17 at shear stress such that

$$(\tau_b)_j = \left(\frac{R_b^{2j}}{R_c}\tau_b\right) = a^j \tau_b \qquad \text{(A-18)}$$

Noting that $\tau_c = (R_b/R_c)^2\ \tau_b$ and using Equations A-18 and A-17 gives:

$$2\sum_{j=0}^{\infty}(\tau_b)_j \frac{d\Omega}{d\tau}\bigg|_{\tau} = (\tau_b)_j$$

$$= \dot{\gamma}(\tau_b) - \dot{\gamma}(\alpha\tau_b) + \dot{\gamma}(\alpha\tau_b) - \dot{\gamma}(\alpha^2\tau_b) + \ldots$$

$$= \dot{\gamma}(\tau_b) = \dot{\gamma}_b \qquad \text{(A-19)}$$

Note that for the wide-gap case where (R_b/R_c) is small, Equation A-18 converges more rapidly than for the narrow gap case.

Equation A-19 can be evaluated numerically from the experimental data, Ω vs. τ_b, or by curve-fitting the data to an approximating equation, computing the derivative, and then evaluating at $(\tau_b)_j$. Graphs of Ω vs. τ for tests run with a B2-X and a B5-X bob are illustrated in Figure A-2. Krieger has published an approximating equation for Equation A-19 which uses data at one point and requires the computation of the first and second derivative at this point.

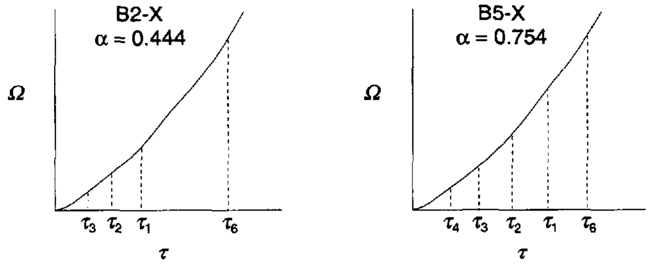


Figure A-2—Graphs of Omega vs. Shear Stress for the B2-Extended Bob and the B5-Extended Bob

A.2.2 POWER-LAW FORM

The power-law form of the rheological constitutive equation is

$$\tau = \mathrm{k}\dot{\gamma}^{n} \qquad \text{(A-20)}$$

where k is the consistency index and n is the power-law index defined as

$$n = \frac{d\log\tau}{d\log\dot{\gamma}} \qquad \text{(A-21)}$$

The power-law equation represents a straight line on a graph of log τ vs. log $\dot{\gamma}$. For most fracture fluids that behave as pseudo-plastics, the power-law can be used to adequately describe the viscous properties of the fluid over a limited range of shear rates. Figure A-3 illustrates a graph of log τ vs. log $\dot{\gamma}$ for a typical fluid. Notice that a "power-law" fluid is also a shear-thinning fluid.

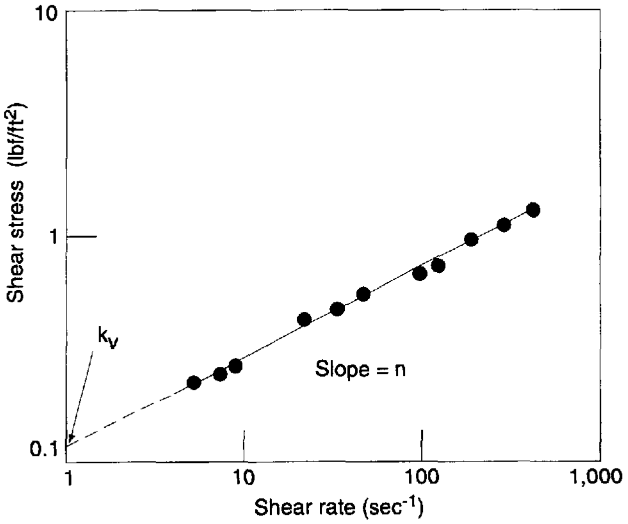


Figure A-3—Shear Stress Graph for a Power-Law Fluid

By definition, n and k are considered constant. If Equation A-20 is used in the kinematic equations for tubular, slot, and Couette flow, the following equations for shear rate result:

Tubular: $$\dot{\gamma}_w = \left(\frac{3\mathrm{n}+1}{4\mathrm{n}}\right)\left(\frac{4Q}{\pi R^3}\right) \qquad \text{(A-22)}$$

Slot: $$\dot{\gamma}_w = \left(\frac{2\mathrm{n}+1}{3\mathrm{n}}\right)\left(\frac{6Q}{Hw^2}\right) \qquad \text{(A-23)}$$

Couette: $$\dot{\gamma}_b = \frac{2\Omega}{\mathrm{n}\left[1-\left(\frac{R_b}{R_c}\right)^{2/\mathrm{n}}\right]}$$

$$= \frac{\pi\Omega'}{15\mathrm{n}\left[1-\left(\frac{R_b}{R_c}\right)^{2/\mathrm{n}}\right]} \qquad \text{(A-24)}$$

where Ω is in rad/s and Ω' is in rpm. Note that Equation A-24 is the exact expression for the shear rate in a Couette device and that is rigorous for any gap size (R_b/R_c) as long as the fluid is indeed power-law.

The viscosity can be calculated from Equation A-19 by dividing by $\dot{\gamma}$.

$$\mu = k\dot{\gamma}^{n-1} \tag{A-25}$$

A.3 Volumetrically Averaged Shear Rate

A Fann Model 50 viscometer is a standard industrial instrument used to measure the viscous properties of fracturing fluids. When the viscosity measurements are made using the Fann Model 50, a nominal Newtonian shear rate at the bob wall is traditionally taken as the standard shear rate in the Couette region. The nominal Newtonian shear rate is dependent upon the cup rotational speed (rpm) and the bob dimensions. For instance, to run a test at a shear rate of 100 sec^{-1}, the cup rotational speed should be set at 265 rpm when an extended B2 (EX-B2) bob is used, or 118 rpm when an extended B5 (EX-B5) bob is used.

We have found that measurements based on the same nominal Newtonian shear rate at the bob wall will not result in reproducible viscosity data when different size bobs are used in the test. In Figure A-4 we observe a large difference in viscosity when identical fluid samples are tested using EX-B2 and EX-B5 bobs. Generally, the wide gap test (EX-B2 bob) yields higher viscosity data than the test using a narrow gap (EX-B5 bob).[3–5] Several possible explanations have been proposed for this difference in viscosity measurements. It has been suggested that the difference in viscosity is due to the near static plugs of fluid above and below the bob and the ability of these plugs to circulate into the gap. Since the small bob provides a wide gap, the fluid that cross-linked in the near static end plug achieved very high viscosity and can be easily circulated into the gap. The larger B5 bob results in a smaller gap; thus, the end plugs are not as easily circulated into the gap, resulting in lower viscosity estimates.

Cameron et al.[4] also observe that the viscous measurements were affected by the gap size in the viscometer. They hypothesize that the difference was the result of nonhomogeneous flow. Cameron et al. believed that nonhomogeneous flow could occur either with low apparent viscosities in a slip-flow state, or with high apparent viscosities in a dispersed state. When a gel flows as a nonhomogeneous fluid, the apparent viscosity of the fluid is a function of the test geometry.

As the viscometer cup is rotating at a specified speed, Ω, the velocity and shear fields in the Couette region are established. For a power model fluid, the velocity and shear rate distributions in the Couette region can be written as follows[6]:

$$v = \frac{\pi\Omega r}{30\left[\left(\frac{R_c}{R_b}\right)^{2/n'} - 1\right]}\left[\left(\frac{R_c}{R_b}\right)^{2/n'} - \left(\frac{R_c}{R}\right)^{2/n'}\right] \tag{A-26}$$

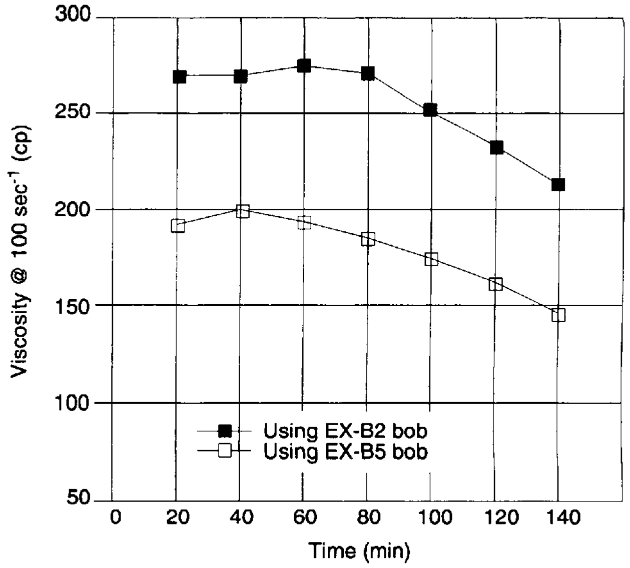


Figure A-4—Viscosity Measurements for the Delayed Titanium HPG Gel Using Nominal Shear Rate of 100 sec^{-1} at 200°F

and

$$\dot{\gamma} = \dot{\gamma}_b\left[\frac{R_b}{R}\right]^{2/n'} \tag{A-27}$$

where $\dot{\gamma}_b$ is the power model shear rate at the bob defined by Equation A-28.

$$\dot{\gamma}_b = \frac{\pi\Omega}{15n'\left[1 - \left(\frac{r_b}{r_c}\right)^{2/n'}\right]} \tag{A-28}$$

Equations A-26–A-28 can be applied to Newtonian fluids when n' is equal to 1. The parameter r represents the radius in the Couette region, measured from the center of the bob shaft.

Figure A-5 shows the shear rate distribution in the Couette region using the EX-B2 and EX-B5 bobs. The Newtonian shear rate at the bob wall is 100 sec^{-1}, and the power-law flow behavior index is assumed to be 0.4. The shear rate exhibits a monotonic decline from the inner cylinder (the bob) to the outer cylinder (the cup). The distribution of shear rate is dependent on the dimensions of Couette region and the flow behavior index. As shown in Figure A-5, the shear field in the narrow gap is different from the shear field in the wide gap, for both Newtonian and power-law fluids. For example, when the flow behavior index equals 0.4, the ratio of shear rate at the cup to the shear rate at the bob can be calculated from Equation A-29 for the EX-B5 bob, and Equation A-30 for the EX-B2 bob.

$$\left[\frac{\dot{\gamma}_c}{\dot{\gamma}_b}\right]_{B5} = (0.868)^{2/n'} = 0.493 \tag{A-29}$$

and

$$\left[\frac{\dot\gamma_c}{\dot\gamma_b}\right]_{B2} = (0.667)^{2/n'} = 0.132 \qquad \text{(A-30)}$$

The fluid is exposed to more shear when using the EX-B5 bob (narrow gap) than when one uses the EX-B2 bob (wide gap), even though the nominal Newtonian shear rates at the bob are equal. Since shear rate has a large effect on the viscous properties of the cross-linked gel, especially during the cross-linking reaction, the EX-B5 bob will produce less viscosity than the EX-B2 bob when the tests are conducted based on the nominal shear rate.

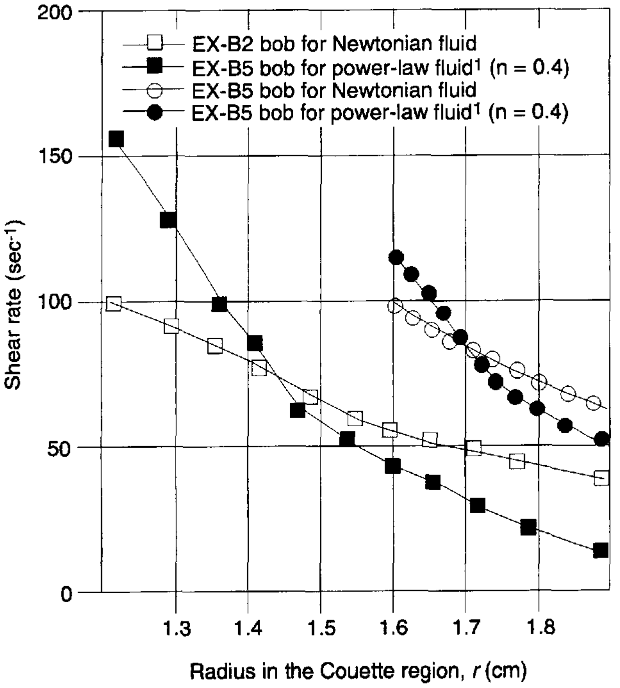


Figure A-5—Shear Rate Distribution in Couette Region

To characterize the effect of the shear field upon the viscosity measurement, a test method based on the volumetric average shear rate in the Couette region was developed to measure the viscous properties of cross-linked HPG gels. The volumetric average shear rate can be obtained from the volumetric integral of the Couette shear rate,[7,8] that is,

$$\text{VASR} = \frac{1}{V}\int_v \dot\gamma dv$$

$$= \frac{1}{V}\int_0^{2\pi}\int_0^{L_b}\int_{r_b}^{r_c}\dot\gamma_b\left[\frac{R_b}{R}\right]^{2/n'}(rdd\Theta rdz)$$

$$= \dot\gamma_b\frac{n'}{(n'-1)\left[\left(\frac{R_c}{R_b}\right)^2-1\right]}\left[\left(\frac{R_c}{R_b}\right)^{2-2/n'}-1\right] \qquad \text{(A-31)}$$

The symbol V is the volume in the Couette region. Equation A-31 is the mathematical expression for volumetric average shear rate for Couette flow. The volumetric average shear rate is a function of the Couette dimensions and the flow behavior index.

VASR should be the base shear rate when different sized bobs are used to measure the viscous behavior of cross-linked HPG gels. Because the flow behavior index (n') is unknown before the test, the test procedure is developed by trial and error. The value of n' is a weak function of cup speed (rpm). Generally, two steps are required for the viscosity measurements. First, two tests are run using the EX-B5 and EX-B2 bobs at the same nominal Newtonian shear rates. Based on the values of flow behavior index (n') obtained during these first tests, the volumetric average shear rates can be estimated. Second, the tests are run again at the same volumetric average shear rates by setting the appropriate cup speed. In a typical test, when the nominal Newtonian shear rate at the bob is specified as 100 sec^{-1}, the volumetric average shear rate with EX-B2 bob is about 60 sec^{-1}, while the volumetric average shear rate using the EX-B5 bob is about 86 sec^{-1} for the fluids we have tested.

If cross-linked gels are tested using the VASR method, the effects of the bob size can be minimized. Figure A-6 illustrates the apparent viscosity vs. time behavior for a delayed titanium cross-linked gel at a temperature of 150°F. It is apparent that the apparent viscosity is a function of the VASR during the cross-linking reaction. Figure A-7 illustrates that the apparent viscosity measurements are reproducible, even with different size bobs, when the gel cross-linking reaction occurs at a consistent value of VASR.

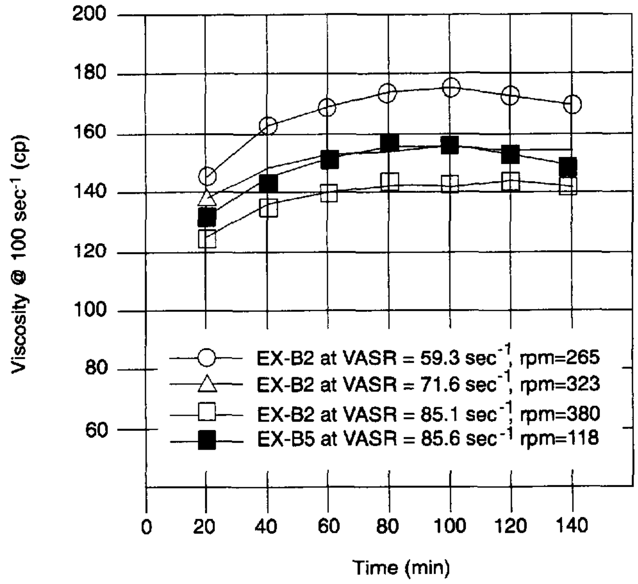


Figure A-6—Comparison of Viscosity Measurements for the Delayed Titanium HPG Gel With 0.1 lb/1000 gal AP Using VASR Method at 150°F

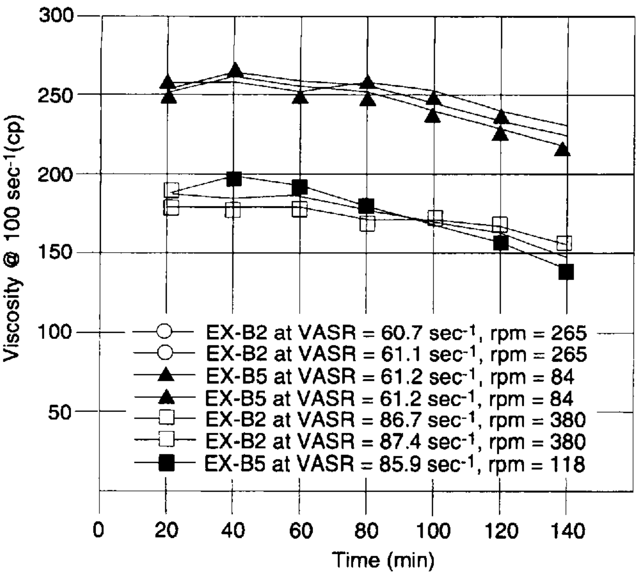


Figure A-7—Comparison of Viscosity Measurements for the Delayed Titanium HPG Gel With 0.1 lb/1000 gal AP Using VASR Method at 200°F

A.4 Slip Correction

Couette flow is a steady rotational laminar flow between concentric cylinders. Normally, the inner cylinder (bob) is stationary while the outer cylinder (cup) is rotating.

When slip occurs, the fluid (gel) velocity at the bob surface is not zero. This non-zero fluid velocity (in the same direction of cup rotation) can be regarded as rotation of the inner bob at a certain angular velocity. This pseudo-rotation of the bob causes the actual shear rate to be smaller than the calculated shear rate based on the prescribed cup rotation speed. When slip occurs, the calculated viscosity is smaller than the actual fluid viscosity. A method for slip correction requires the use of two bobs that have the same average radii, but with different amounts of surface roughness. One of the bobs has a smooth surface that allows the slip to occur along the bob surface. The other bob has a rough surface that can effectively prevent slippage by not allowing the slip layer to form and remain intact.

APPENDIX B—TYPES OF FLUID

B.1 Newtonian Fluids

Viscosity is defined as the ratio of shear stress to shear rate. When the viscosity of a fluid is not a function of the shear rate in the laminar flow regime, the fluid is called a Newtonian fluid.

Figure B-1 can be used to describe the shear rate vs. shear stress relationship for a Newtonian fluid. Given a system where a fluid is located between two parallel plates, separated by a distance *h*, then a force (*F*) is applied to the top plate, the velocity gradient in the fluid between the two plates will be linear.

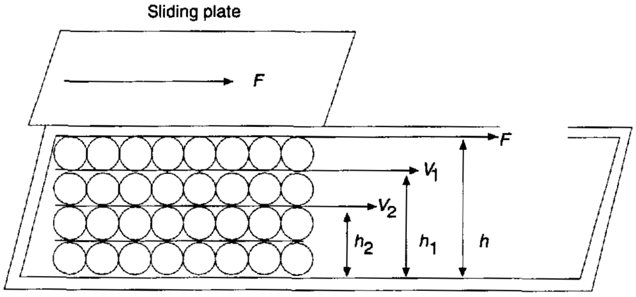


Figure B-1—Graphical Explanation of the Shear Stress-Shear Rate Relationship

The shearing force, *F*, is in dynes and the area of the plate is in square centimeters, so that shearing stress is in dynes per square centimeter. Likewise, the velocity (v) is in centimeters per second, and the plate separation (h) is in centimeters. Therefore, the rate of shear is expressed in reciprocal seconds (s^{-1}).

Viscosity has the dimensions dyne-sec/cm^2, which is called a poise. The practical unit of viscosity is centipoise. One poise equals 100 centipoise. A graph of shear stress vs. shear rate is called a rheogram. Rheograms for various classical fluids that have no time dependency are shown in Figure B-2. The shear rate, which is the independent variable when the test speed is controlled or programmed, is shown on the X axis. Shear stress, the dependent variable, is shown on the Y axis. A Newtonian fluid appears as a straight line passing through the origin. Higher viscosity Newtonian fluids will have steeper slopes, while lower viscosity Newtonian fluids would have a lesser slope.

B.2 Non-Newtonian Fluids

Figure B-2 illustrates three classical fluid types: Newtonian, Bingham plastic, and pseudo-plastic. A Bingham plastic fluid is one that creates a linear relationship between shear stress and shear rate, but does not intercept the origin at zero. The intercept of a Bingham plastic fluid is often called the Bingham yield point.

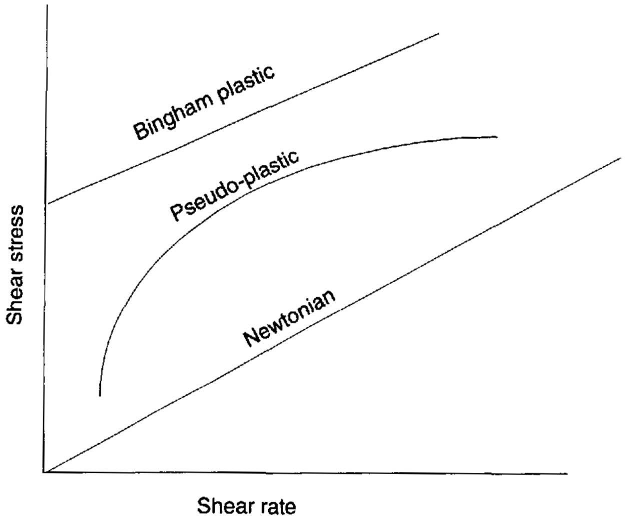


Figure B-2—Illustration of Shear Stress Relationship for Classical Fluids

A pseudo-plastic fluid is a shear thinning fluid that does not result in a straight line when a graph of shear stress vs. shear rate is drawn on linear coordinate paper. Many complex fracturing fluids are loosely described as pseudo-plastic or power-law fluids. For most cases, this description is oversimplified, because many of these complex fluids do not result in a straight line behavior on full logarithmic paper over a very large range of shear rates. Figure B-3 illustrates the apparent viscosity of the typical fracturing fluid over a wide range of shear rates. These data are for a linear gelled fluid using hydroxypropylguar. Figure B-3 illustrates the fact that a single set of n' and k' values cannot be used to describe many of the typical fracturing fluids over a wide range of shear rates. The change in viscous behavior as a function of shear rate is important because the shear rates in the wellbore may be in excess of 1000 sec^{-1}, while the shear rates in a hydraulic fracture can easily range from 1-100 sec^{-1}.

Most of the fracture design computer models assume that the fracturing fluids can be described by the power-law model. To be sure the computer models are using reasonable values of apparent viscosity in the hydraulic fracture, we have to be able to generate reasonable estimates of n' and k' in the laboratory. As such, our laboratory tests should be patterned after an actual fracture treatment, run at realistic values of shear rate, and should provide results that are reproducible.

A challenge to our industry is to determine the best equipment and procedures for measuring the viscous properties of cross-linked, water-based fracturing fluids.

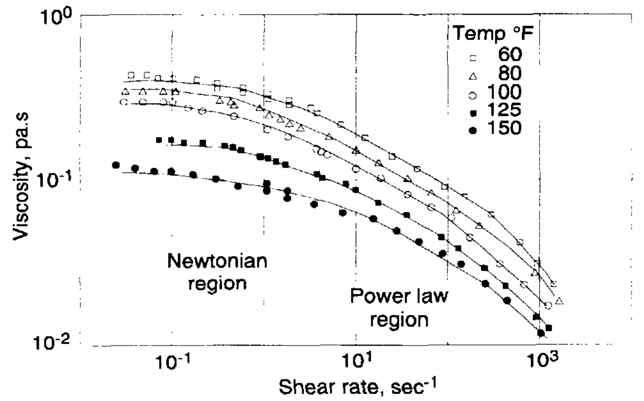


Figure B-3—Apparent Viscosity of an HPG Fluid Over a Wide Range of Shear Stress

B.3 Gels and Solutions

Commonly used fracturing fluids today contain thickening agents to increase the viscosity of the fluid. A gel is a suspension of polymers which are kept in solution through hydrogen bonding. The commonly used gelling agents for water-based fluids are guar, hydroxypropylguar (HPG), carboxymethylhydroxypropylguar (CMHPG), carboxymethylcellulose (CMC), hydroxyethylcellulose (HEC), and various types of polyacrylamide. The most commonly used gels are guar, HPG, and CMHPG. These polymers do not actually go into solution in the manner that salt is dissolved in water, then deionized into sodium and chlorides. The polymers are kept in suspension by hydrating with water molecules and through hydrogen bonding. The inter-relationship between the hydrated polymer chains is in fact what creates the viscosity or drag. Cross-linked gels are developed by tying the guar, HPG, or CMHPG molecules together with either metallic ions or ligand boron ions. The final product is called a pseudo-plastic.

Many of the rapid metallic cross-linked gel systems are extremely shear degradable. Thus, technology has been developed to delay the cross-linking reaction so that much of the cross-linking takes place in the fracture where the shear rate is much lower than the shear rate in the wellbore. Delayed cross-linked systems will result in less wellbore friction and higher apparent viscosities in the fracture.

B.4 Typical Additives

Polymers are generally used to viscosify the water in fracturing fluids. The polymers can be classified as being natural or synthetic. Natural polymers are those extracted directly from vegetable products grown on a commercial scale such as guar gum, hydroxypropylguar (HPG), and carboxymethylhydroxypropyl guar (CMHPG). Synthetic polymers are those formulated from raw products available to the chemical industry. Typical synthetic polymers include hydroxyethyl cellulose (HEC), carboxymethyl cellulose (CMC), and carboxymethlhydroxyethyl cellulose (CMHEC).

To increase viscosity, both natural and synthetic polymers can be reacted with a number of metal ions (crosslinkers to form cross-linked gel). The cross-linking reaction dramatically increases the effective molecular weight of the polymer, thereby substantially increasing the viscosity and elasticity of the fluid. The increase in elasticity causes the stress response of these fluids to be energy-dependent and time-dependent. Cross-linked gels possess several advantages over noncross-linked gels, such as greater viscosity, improved thermal stability, versatility, and adaptability to a wide variety of treatment conditions. As the viscosity increases, one will create a wider fracture and will improve sand transport. The introduction of cross-linked gels as stimulation fluids has been a major advancement in hydraulic fracturing technology.[1]

According to the cross-linking reaction rate, crosslinker systems can be classified as rapid or delayed. For the rapid system, the cross-linking reaction is fast enough that gel structure is established during the mixing process or as the fluid is being pumped down the wellbore. Common borate crosslinker belongs to this type of system, since cross-linking reaction takes place immediately once the solution pH is adjusted to 9.

For the delayed system, the crosslinker is prepared using chelated agents or organic solvents. When the cross-linking reaction occurs, the reaction is either temperature-activated or pH-controlled. Therefore, the cross-linking reaction rate can be controlled at the surface by either delaying the start of the cross-linking reaction or slowing the reaction rate. Compared to the fast cross-linking system, the delayed cross-linking system allows better dispersion of the crosslinker, yields more viscosity, improves thermal stability, and produces much lower pumping friction due to the low viscosity in the tubular goods.

Modern fracturing fluids contain numerous chemical additives in addition to the thickening agents. Table B-1 summarizes commonly used additives in fracturing fluid systems.[1]

Table B-1—Typical Fracturing Fluids Additives

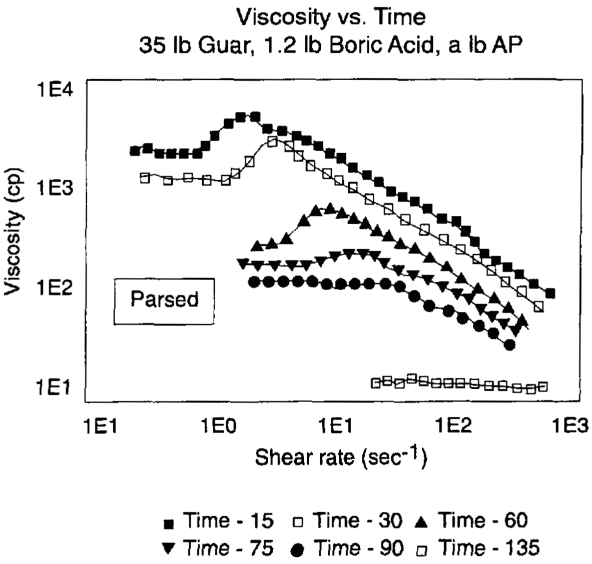
Chemical Additives	Functions
Biocides	Eliminate surface degradation and stop growth of anaerobic bacteria in the formation
Breakers	Degrade polymers
Buffers	Control pH for polymer hydration and cross-linking
Clay Stabilizer	Prevent the dispersion of clay particles
Crosslinker	Crosslink polymers
Defoamer	Prevent formation of foam
Diverting Agent	Divert flow of the fracturing fluid to a zone below or above the zone being treated
Friction Reducer	Reduce pumping friction pressure
Gel Stabilizer	Inhibit chemical degradation of polymers
Oxygen Scavenger	Remove oxidative degradation
Surfactants	Reduce interfacial tension and increase solubility

Each of the additives has a special purpose. For example, buffers are used in conjunction with polymers so that the optimal pH for polymer hydration and cross-linking can be obtained. When the optimal pH is reached, the maximal viscosity is more likely to be obtained. Gel stabilizers are used to inhibit polymer degradation as they experience shearing and high temperature in the fracture. To use additives properly, one must verify the relative compatibility of all additives. A fracturing treatment can be jeopardized if a fracturing fluid contains incompatible additives.[1]

The selection of fracturing fluids and associated additives is a key factor in the success of the fracturing treatment. The use of fracturing fluids should be optimized based on the formation conditions. For example, if the formation has moderate permeability with high fluid loss, a good choice may be guar gum instead of HPG or HEC. If the formation is extremely water sensitive, one should consider oil-based fluid rather than the aqueous fluid.

B.5 Borates

Borate cross-linked guar represents the most rheologically complex stimulation fluid used in the industry today. Contrary to common belief, borates are time- and shear history-dependent. That is, the shear rehealing that occurs is slow with respect to the measurement time in the rheometer. Furthermore, the maximum viscosity depends on the levels of shear that the fluid has previously been subjected to. The general viscosity profile of a borate fluid is given in Figure 1 for a 35 lb/1000 gal, pH 9.5, guar-borate system containing oxidizing breaker at 125°F. Several different rheological regions are seen which occur at different shear rates. The precise shear rate region where this behavior is seen depends on the fluid formulation (pH, borate concentration) and temperature. The generalized behavior however is clearly seen in the 15 minute data in Figure B-4.



Note:
Provided by permission of the STIM-LAB, Inc. Rheology Consortium, Report on the Investigation of Rheology and Proppant Carrying Capacity of Common Fracturing Fluids, July 1995.

Figure B-4—Viscosity vs. Time

At very low shear rates (<0.1 sec^{-1} and not shown in the figure), the apparent viscosity increases with time due to rehealing and may be easily misinterpreted when trying to determine the shear stress-shear rate behavior of the fluid in this shear rate range. The characteristic regions are (a) a low shear rate Newtonian plateau, (b) a shear thickening region (dilatent), (c) power-law region, and (d) a power-law region where *n′* may be <0. As the fluid degrades, this complex behavior transitions to a more expected rheology profile with a low shear Newtonian plateau and a power-law region.

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