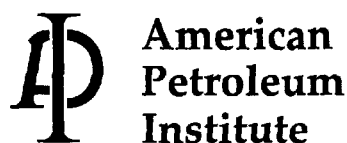


Recommended Practices on Measuring the Viscous Properties of a Cross-linked Water-based Fracturing Fluid

API RECOMMENDED PRACTICE 39
THIRD EDITION, MAY 1998



**Helping You
Get The Job
Done Right.SM**

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

Exploration and Production Department

API RECOMMENDED PRACTICE 39
THIRD EDITION, MAY 1998



**American
Petroleum
Institute**

**Helping You
Get The Job
Done Right.SM**



Documents

Document Sharing Hub

SPECIAL NOTES

API publications necessarily address problems of a general nature. With respect to particular circumstances, local, state, and federal laws and regulations should be reviewed.

API is not undertaking to meet the duties of employers, manufacturers, or suppliers to warn and properly train and equip their employees, and others exposed, concerning health and safety risks and precautions, nor undertaking their obligations under local, state, or federal laws.

Information concerning safety and health risks and proper precautions with respect to particular materials and conditions should be obtained from the employer, the manufacturer or supplier of that material, or the material safety data sheet.

Nothing contained in any API publication is to be construed as granting any right, by implication or otherwise, for the manufacture, sale, or use of any method, apparatus, or product covered by letters patent. Neither should anything contained in the publication be construed as insuring anyone against liability for infringement of letters patent.

Generally, API standards are reviewed and revised, reaffirmed, or withdrawn at least every five years. Sometimes a one-time extension of up to two years will be added to this review cycle. This publication will no longer be in effect five years after its publication date as an operative API standard or, where an extension has been granted, upon republication. Status of the publication can be ascertained from the API Exploration and Production Department [telephone (202) 682-8000]. A catalog of API publications and materials is published annually and updated quarterly by API, 1220 L Street, N.W., Washington, D.C. 20005.

This document was produced under API standardization procedures that ensure appropriate notification and participation in the developmental process and is designated as an API standard. Questions concerning the interpretation of the content of this standard or comments and questions concerning the procedures under which this standard was developed should be directed in writing to the director of the Exploration and Production Department, American Petroleum Institute, 1220 L Street, N.W., Washington, D.C. 20005. Requests for permission to reproduce or translate all or any part of the material published herein should also be addressed to the director.

API standards are published to facilitate the broad availability of proven, sound engineering and operating practices. These standards are not intended to obviate the need for applying sound engineering judgment regarding when and where these standards should be utilized. The formulation and publication of API standards is not intended in any way to inhibit anyone from using any other practices.

Any manufacturer marking equipment or materials in conformance with the marking requirements of an API standard is solely responsible for complying with all the applicable requirements of that standard. API does not represent, warrant, or guarantee that such products do in fact conform to the applicable API standard.

All rights reserved. No part of this work may be reproduced, stored in a retrieval system, or transmitted by any means, electronic, mechanical, photocopying, recording, or otherwise, without prior written permission from the publisher. Contact the Publisher, API Publishing Services, 1220 L Street, N.W., Washington, D.C. 20005.

Copyright © 1998 American Petroleum Institute

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.

This recommended practice is based on the knowledge and experience of petroleum refiners, valve manufacturers, and others, and its objective is to describe practices that will result in a purchaser's receipt of valves which consistently meet API valve specifications.

Any modifications, deletions, and amplifications necessary for individual users should be made by supplementing this recommended practice rather than by rewriting it.

API publications may be used by anyone desiring to do so. Every effort has been made by the Institute to assure the accuracy and reliability of the data contained in them; however, the Institute makes no representation, warranty, or guarantee in connection with this publication and hereby expressly disclaims any liability or responsibility for loss or damage resulting from its use or for the violation of any federal, state, or municipal regulation with which this publication may conflict.

Suggested revisions are invited and should be submitted to the director of the Exploration and Production Department, American Petroleum Institute, 1220 L Street, N.W., Washington, D.C. 20005.

CONTENTS

	Page
1 INTRODUCTION	1
1.1 Fracturing Fluid Rheology (Viscosity) & the Hydraulic Fracturing Process	1
1.2 Cross-linked Fracturing Fluid Rheological Behavior	1
1.3 Objectives and Limitations of This Document	4
2 REFERENCES	4
2.1 Standards	4
2.2 Other Referenced Publications	4
3 DEFINITIONS AND NOMENCLATURE	4
3.1 Definitions	4
3.2 Nomenclature	5
4 LABORATORY PROCEDURES	6
4.1 Fluid Preparation and Testing	6
4.2 Equipment Requirements	7
4.3 Instrument Calibration	10
5 FIELD PROCEDURES	11
5.1 Equipment Requirements	11
5.2 Preparation of Linear Polymer Solutions	11
5.3 Special Field Testing Equipment	12
5.4 Field Testing Procedure	12
5.5 Discussion of Commonly Observed Problems	12
6 CALCULATION PROCEDURES FOR VISCOUS PROPERTIES	12
6.1 General Concepts	12
6.2 Couette Geometry	13
6.3 Example Calculation: Couette Geometry with Standard R1-B5 Geometry	14
APPENDIX A BASIC RHEOLOGICAL CONCEPTS	17
APPENDIX B TYPES OF FLUID	23

Figures

1 Typical Full Range Shear Stress vs. Shear Rate Rheograms for Cross-linked Water-based Fractured Fluids	2
2 Segmented Portion, Shear Stress vs. Shear Rate Rheograms for Cross-linked Water-based Fracturing Fluids	3
3 Shear Stress Graph for a Power-Law Fluid	3
A-1 Graph of Shear Stress Data	18
A-2 Graphs of Omega vs. Shear Stress for the B2-Extended Bob and the B5-Extended Bob	19
A-3 Shear Stress Graph for a Power-Law Fluid	19
A-4 Viscosity Measurements for the Delayed Titanium HPG Gel Using Nominal Shear Rate of 100 sec ⁻¹ at 200°F	20
A-5 Shear Rate Distribution in Couette Region	21
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	21

A-7	Comparison of Viscosity Measurements for the Delayed Titanium HPG With 0.1 lb/1000 gal AP Using VASR Method at 200°F	22
B-1	Graphical Explanation of the Shear Stress-Shear Rate Relationship	23
B-2	Illustration of Shear Stress Relationship for Classical Fluids	23
B-3	Apparent Viscosity of an HPG Fluid Over a Wide Range of Shear Stress	24
B-4	Viscosity vs. Time	25
 Tables		
1	Description of Equipment for Laboratory Testing	9
2	Rotor-Bob Combinations	9
3	Calibration Factors for Dead Weight Testing	10
4	Results, Calculation of Power-Law Parameters	15
5	Results, Calculation of Actual Power-Law Viscosity at a Shear Sweep Time of 30 Mins	15
B-1	Typical Fracturing Fluids Additives	24

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

1 Introduction

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:

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

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.

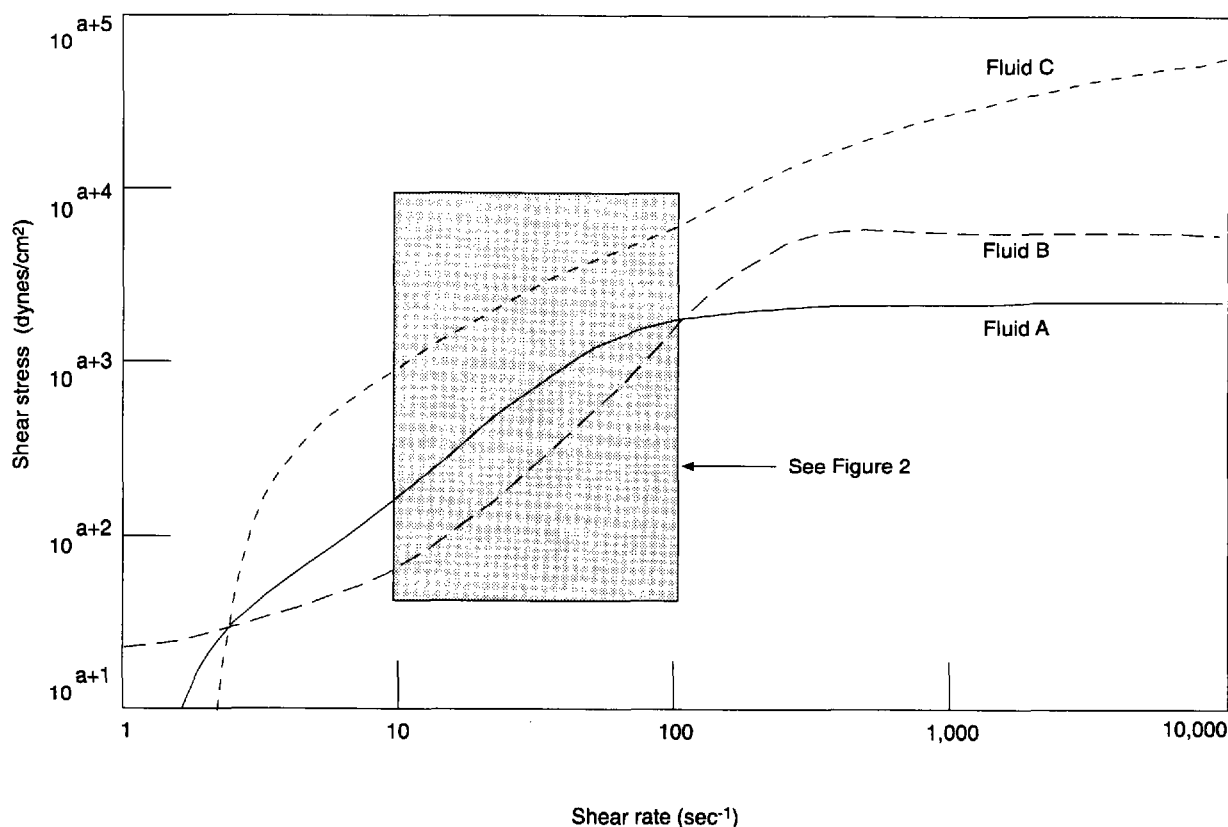


Figure 1—Typical Full Range Shear Stress vs. Shear Rate Rheograms for Cross-linked Water-based Fractured Fluids

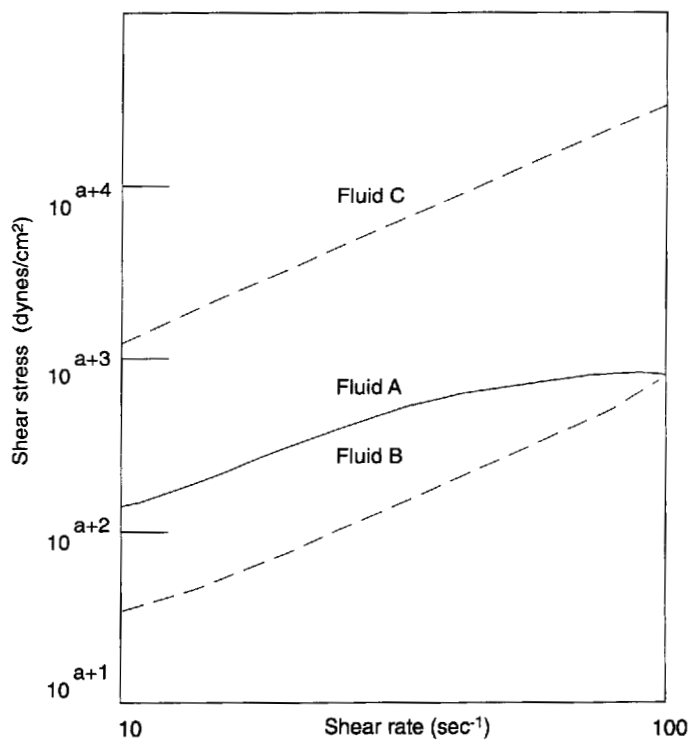


Figure 2—Segmented Portion, Shear Stress vs. Shear Rate Rheograms for Cross-linked Water-based Fracturing Fluids

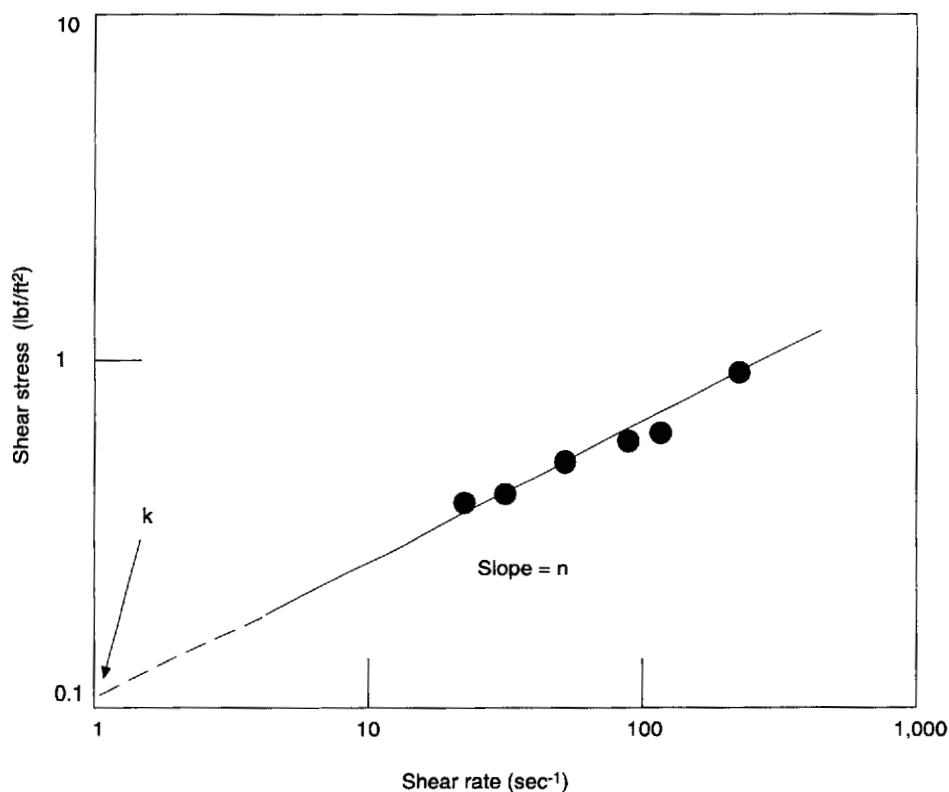


Figure 3—Shear Stress Graph for a Power-Law Fluid

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:

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

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

- The procedures in this document are not intended for proppant-laden fluids, hydrocarbon-based fluids, foams, or emulsions.
- The procedures are not intended for fluids with rheological properties or behaviors that drastically deviate from power-law behavior.
- 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.
- 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.

2.2 OTHER REFERENCED PUBLICATIONS

- Schowalter, W. R., *Mechanics of Non-Newtonian Fluid*, Pergamon Press, New York (1978).
- B. Rabinowitsch, A. *Physik Chem.*, A145, 1 (1929).
- Krieger, I.M. and Elrod, “Direct Determination of the Flow Curves of Non-Newtonian Fluids. II Shearing Rte in the Concentric Cylinder Viscometer” *J. Appl. Phys.* (1953) 24, No. 2, 134-136.
- Cameron, J.R., “Viscometry of Nonhomogeneous Flows and the Behavior of a Titanium-Cross-linked Hydroxypropyl Guar Gel in Couette Flow,” *J. Of Rheology*, 33(1), 15-46 (1989).
- Krieger, I.M., “Shear Rate in the Couette Viscometer,” *Trans. Soc. of Rheology*, 12:11, 5-11, 1968.
- Whistler, Roy, *Industrial Gums*.
- Bird, Armstrong, *Dynamics of Polymeric Liquids*, Vol. 1.
- Craigie, L. J., “A New Method for Determining the Rheology of Crosslinked Fracturing Fluids Using Shear History Simulation,” paper SPE 11653, 1983.
- Fan, Y. and Holditch, S. A., “Use of Volumetric-Average Shear Rate to Test Crosslinked Fluids With the Fann 50 Viscometer,” *SPE Production & Facilities*, (Aug. 1995) 191-196.
- Economides, M. J. and Nolte, K. G., *Reservoir Stimulation*, Second Edition, Prentice Hall, New Jersey (1989).
- Gidley, J. L., Holditch, S. A., Nierode, D. E., and Veatch, R. W., *Advances in Hydraulic Fracturing*, SPE Monograph 12 (1989).

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.



Documents

Document Sharing Hub