

Recommended Practices for Evaluation of Polymers Used in Enhanced Oil Recovery Operations

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RECOMMENDED PRACTICES FOR EVALUATION OF POLYMERS USED IN ENHANCED OIL RECOVERY OPERATIONS

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RECOMMENDED PRACTICES FOR EVALUATION OF POLYMERS USED IN ENHANCED OIL RECOVERY OPERATIONS

FOREWORD

a. These recommended practices were prepared by the Subcommittee on Evaluation of Polymers Used in Enhanced Oil Recovery Operations. This publication is under the administration of the American Petroleum Institute Production Department's Executive Committee on Drilling and Production Practices.

b. The tests recommended herein have been developed to improve polymer quality in field applications. The recommended procedures and conditions have been prepared for use in evaluating and comparing polymer performance under the stated conditions as an aid to selecting materials to be used in enhanced recovery operations. *However, these recommended tests should not be taken as absolute criteria for determining polymer performance under conditions which are characteristic of the downhole environment.* Alternative test procedures are available and are routinely used to meet or exceed recommended practices or performance levels set forth herein.

c. The recommendations presented in this publication are not intended to inhibit the development of new technology, materials improvements, or improved operational procedures. Qualified engineering analysis and judgment will be required for their application to fit a specific situation.

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Everyone should minimize their exposure to chemicals. The following general precautions are suggested:

1. Minimize skin and eye contact and breathing of vapors.
2. Keep away from the mouth; can be harmful or fatal if swallowed or aspirated.
3. Keep containers closed when not in use.
4. Keep work areas as clean as possible and well ventilated.
5. Clean up spills promptly and in accordance with pertinent safety, health, and environmental regulations.
6. Observe established exposure limits* and use proper protective clothing and equipment.

Employees should consult and comply with the Occupational Safety and Health Administration regulations, 29 CFR Sec. 1910.1450, *Occupational Exposure to Hazardous Chemicals in the Laboratory*, in the enacted and most current form. FOR ADDITIONAL INFORMATION, CONSULT WITH THE EMPLOYER AND RESEARCH THE MATERIAL SAFETY DATA SHEETS.

f. Suggestions for revisions or corrections are invited and should be submitted to: Director, Production Department, American Petroleum Institute, 2535 One Main Place, Dallas, Texas 75202-3904.

*Refer to Permissible Exposure Limits (PELs) and standards of the U.S. Department of Labor, Occupational Safety and Health Administration, 29 CFR Subpart Z, Sec. 1910.1000 et seq (check latest revision), available from U.S. Government Printing Office, Washington, D.C. 20402. Refer also to *Threshold Limits Values and Biological Indices* (published annually, check latest edition), available from American Conference of Governmental Industrial Hygienists, 6500 Glennway Ave., Bldg. D-7, Cincinnati, OH 45211.

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RECOMMENDED PRACTICES FOR EVALUATION OF POLYMERS USED IN ENHANCED OIL RECOVERY OPERATIONS

SECTION 1 SCOPE

1.1 INTRODUCTION

1.1.1 Water soluble polymers designed for use as mobility control agents in enhanced oil recovery operations and currently available in commercial quantities are mainly of two major types: hydrolyzed polyacrylamides and polysaccharides. Polyacrylamides are synthetic, high molecular weight copolymers and are marketed in a variety of product forms: dry powder, liquid emulsion or dispersion, concentrated solutions, and gels. Polysaccharides produced by fermentation processes (e.g., xanthan and scleroglucan biopolymers) are available as dry powder and liquid broths containing up to 15% active polymer.

1.1.2 These recommended practices have been prepared to provide industry standard procedures and test conditions which can be used to evaluate and compare polymer performance under the stated conditions. *These recommended tests are primarily for qualitative compar-*

ison of performance and general screening of water soluble polymers under conditions described herein. This publication does not present recommendations which can be taken as absolute tests for determining polymer performance under conditions which are characteristic of the downhole environment. These recommended practices are not intended to obviate the need for applying sound engineering judgment in the selection and application of polymer products for use in enhanced oil recovery operations.

1.1.3 For purposes of these recommended practices, standard brine solutions are stipulated to enable users to evaluate performance of polymers under controlled laboratory conditions. Similar tests may be developed using field brine solutions, but these test results should not be compared with results of tests conducted using the standard brine solutions. These recommended practices are not intended to inhibit users from applying other test procedures or practices to their needs.

SECTION 2 SOLUTION PREPARATION

2.1 INTRODUCTION

2.1.1 General. Special attention to recommended solution preparation and mixing procedures is essential to obtain optimum properties with high molecular weight polymers. Dissolution rates will vary depending on product form and type. Important factors to be considered in preparing solutions are high-shear/low-shear mixing, initial mix concentration, brine composition, pH, activator concentration, and hydration or aging time. Recommended procedures are given in this section for each of the various product forms available. Product manufacturers should be consulted for additional recommendations specific to their products.

2.1.2 Shearing Effects. Some polymers require a short period of high-shear mixing followed by a longer period of low-shear mixing. Hydration times can vary from a few minutes to several hours. Mixing techniques for polysaccharides are different from those used for polyacrylamides. Dried polysaccharides require high-shear mixing, whereas polyacrylamides are degraded by moderate to high shear.

2.1.3 Salinity Effects. As brine salinity increases, both polyacrylamides and polysaccharides become more difficult to hydrate. Solid polysaccharides to be dissolved in high salinity brines can benefit from a fresh water hydration step, i.e., preparation of a fresh water polymer concentrate which is sheared and then diluted with high salinity brine. The higher the salinity and divalent ion content, the more difficult the inversion of polyacrylamide emulsions. Some dry polyacrylamides contain significant amounts of sodium carbonate, which in hard brines may precipitate as calcium carbonate unless the pH is lowered.

2.1.4 Synthetic Brines. Reference should be made to *API RP 45: Recommended Practice for Analysis of Oil-Field Waters*,* Second Edition, November 1968 (Reissued July 1981), Section 1.5, "Preparation of Stable Synthetic Brines." For comparisons of polymers, brines may be prepared from sodium chloride alone or from sodium chloride and calcium chloride. Commonly used reference solvents are a "fresh water" containing 1000 ppm NaCl and 100 ppm CaCl₂, and a "hard brine" containing 3% NaCl and 0.3% CaCl₂ in deionized water. Brine solutions should be freshly prepared. If solutions are to be used for porous media tests, filtration of the brine solution through a 0.22-micron filter is advised.

2.1.5 Concentration Units. Polymer concentrations are commonly expressed as parts per million (ppm). In the recommended laboratory procedures described in this section, *ppm is always used on a weight/weight basis, i.e., weight of polymer per total weight of solution.*

*Available from American Petroleum Institute, Publications and Distribution Section, 1220 L St., N.W., Washington, D.C. 20005.

2.2 RECOMMENDED PROCEDURES FOR LABORATORY PREPARATION OF POLYMER SOLUTIONS FROM EMULSION AND DISPERSION GRADE POLYACRYLAMIDE PRODUCTS

2.2.1 General. The term, "liquid polyacrylamide," encompasses products marketed as true aqueous solutions and products which are liquid by virtue of being supplied as emulsions/dispersions in a hydrocarbon continuous phase.

Typically, emulsion/dispersion products are available commercially at concentrations in the range of 25-50 weight percent active polymer either as a water-in-oil emulsion or a dispersion in which the polymer exists as a finely divided suspension in a hydrocarbon continuous phase. General characteristics and handling procedures for emulsion and dispersion products are similar, with both types usually containing surfactant(s) which allow "inversion" and dissolution of the polymer upon contact with water. Additional or even alternative surfactants (activators) may be recommended for efficient inversion in medium to high salinity brines. Polyacrylamide solution concentrates are available commercially at concentrations in the range of 3-20 weight percent active polymer. Before preparing solutions, consult the product manufacturer regarding specified active polymer content and recommendations regarding additional activator:

Products to which activator has been added during manufacture are known as single pack or single component systems. Dual pack or dual component products are also available and involve the polymer concentrate and activator being supplied separately. With this type of product, the activator must be added to the make-up water before the polymer concentrate is added.

Correct preparation of all emulsion/dispersion products requires the application of a brief period of adequately high shear immediately following contact of the product with the brine. Preparation in accordance with this concept allows efficient distribution of discrete polymer particles, promotes rapid hydration, and minimizes possible shear degradation of the hydrated polymer.

In the context of the above, *it is recommended that stock solutions be prepared at a concentration of 0.5% w/w (5000 ppm) active polymer followed by subsequent dilution to the desired working concentration(s).*

2.2.2 Preparation of Solutions (Overhead Stirrer).

2.2.2.1 Apparatus. The following apparatus is required for test solution preparation:

Overhead Stirrer Assembly	Jiffy® mixer, or equivalent caged paddle stirrer or shaft fitted with a two-inch marine blade propeller driven by a stirring motor (Sargent-Welch cone drive variable speed, catalog number OS76445, or equivalent.)
---------------------------	--

Top Loading Laboratory Balance	Sensitivity 0.01 g. Maximum load 2000 g.
Container	16-oz, wide-mouth jar or 600-mL beaker.
Disposable Plastic Syringe	1mL, 5mL, 10mL
Tumbler	Any device capable of tumbling a 16-oz. jar end-over-end at approximately 30 rpm (usually hand made).

Magnetic Stirrer

Coated Magnetic Stirring Bar

Stopwatch

2.2.2.2 Calculation for Preparing Stock Solution.

- Using manufacturer's or other data describing the product, calculate the amount of emulsion/dispersion polymer product required to make up the appropriate amount of stock solution.

$$W_{pr} = \frac{W_s \times C_s \times 10^{-4}}{A_{pr}} \dots \dots \dots (2-1)$$

where:

 W_{pr} = weight of polymer product, g. W_s = weight of stock solution to be made (including additional activator, if any), g. C_s = concentration of polymer in stock solution, ppm. A_{pr} = activity of polymer product, weight percent.
(NOTE: Supplier should specify percent active polymer in the polymer product.)

- Calculate the amount of makeup water used for the stock solution.

$$W_{bs} = W_s - W_{pr} \dots \dots \dots (2-2)$$

EXAMPLE: To prepare 400 g of 5000 ppm stock solution using a 30.25% active emulsion/dispersion polymer product, calculate the weight of polymer required using Equation (2-1):

$$W_{pr} = \frac{W_s \times C_s \times 10^{-4}}{A_{pr}} = \frac{400 \times 5000 \times 10^{-4}}{30.25} = 6.61 \text{ g}$$

Then calculate the amount of makeup water using Equation (2-2):

$$W_{bs} = W_s - W_{pr} = 400.00 - 6.61 = 393.39 \text{ g.}$$

2.2.2.3 Mixing Procedure (Overhead Stirrer).

- Add the calculated amount of makeup water [Equation (2-2)] to a 16-oz, wide-mouth jar.
- Offset the stirrer slightly from the middle of the jar. Set the speed of the overhead stirrer so that the vortex extends 75% into the solution.

- If no additional activator is required, proceed to Step 5.

- If additional activator is required, it should be added to the makeup water by the following recommended procedure:

- Determine the amount of additional activator to be added in accordance with the manufacturer's recommendations.

- Fill a 1-mL plastic syringe (no needle) with activator and fully discharge the contents to waste.

- Fill the syringe, wipe the outside of the syringe with a paper towel, and discharge excess activator until the approximate required amount remains in the syringe. Record gross weight of the syringe plus contents.

- Inject the activator *onto the shoulder of the vortex* in one continuous stroke and stir for one minute.

- Reweigh the empty syringe to determine the exact weight of activator added.

- After thoroughly mixing the polymer product to ensure homogeneity, weigh out the required amount of polymer product in the following manner:

- Fill a 5-mL or 10-mL plastic syringe (no needle) with polymer product and discharge the contents to waste.

- Fill the syringe, wipe the outside of the syringe with a paper towel, and discharge excess polymer product until the approximate required amount remains in the syringe. Record gross weight of the syringe plus contents.

- Add the product to the brine by the following procedure:

- Inject the product *onto the shoulder of the vortex* as rapidly as possible using one continuous stroke. Slow injection, or injection directly onto the wall or impeller, can result in poor dispersion. Stir for five (5) minutes. **NOTE:** It is *not* recommended that the above procedure be used for direct preparation of polymer solutions at concentrations less than 5000 ppm active polymer. To do so may lead to either problems with inversion or excessive mechanical shear of hydrated polymer, depending on the particular activator and brine concentrations.

- Set the stirrer speed at 150-250 rpm and continue stirring for two (2) hours. The product should be evenly dispersed.

- Reweigh the empty syringe and subtract the syringe weight from gross weight to determine the exact amount of polymer product added.

Recalculate the precise concentration of stock solution.

$$C_s = \frac{W_{pr} \times A_{pr} \times 10^4}{W_{bs} + W_{pr}} \dots\dots\dots (2-3)$$

where:

C_s = concentration of polymer in stock solution, ppm.

W_{pr} = weight of polymer product added, g.

A_{pr} = activity of polymer product, weight percent.

W_{bs} = weight of makeup water used for the stock solution, including additional activator, if any, g.

EXAMPLE: For a product containing 30.25% active polymer, in which 393.39 g of brine and 6.55 g of product were used:

$$C_s = \frac{W_{pr} \times A_{pr} \times 10^4}{W_{bs} + W_{pr}} = \frac{6.55 \times 30.25 \times 10^4}{393.39 + 6.55} = 4954 \text{ ppm}$$

7. Transfer the 5000 ppm stock solution to a 600-ml beaker and stir gently for 1-2 hours, using the overhead stirrer fitted with a paddle-type impeller (Par. 2.2.2.1) and operating at 150-250 rpm. Additional aging time may be required when using very high salinity brines. Alternatively, transfer the solution to a capped glass jar and tumble end over end for 1-2 hours, using a suitable rotary tumbler.
8. Dilute the aged 5000 ppm stock solution to the desired concentration by addition of the appropriate amount of brine (refer to Par. 2.2.2.4). Mix gently for 10 minutes using a magnetic or overhead stirrer.

2.2.2.4 Calculations for Dilution of Stock Solution.

1. Calculate the weight of stock solution required to make up the desired amount of diluted solution.

$$W_s = \frac{W_d \times C_d}{C_s} \dots\dots\dots (2-4)$$

where:

W_s = weight of stock solution, g.

W_d = weight of diluted solution to be made, g.

C_d = concentration of polymer in diluted solution, ppm.

C_s = concentration of polymer in stock solution, ppm.

2. Calculate the amount of makeup water required to make the desired amount of diluted solution.

$$W_{bd} = W_d - W_s \dots\dots\dots (2-5)$$

where:

W_{bd} = weight of makeup water used in the diluted solution, g.

EXAMPLE: To make 400 g of 1000 ppm diluted solution from a 5000 ppm stock solution:

$$W_s = \frac{W_d \times C_d}{C_s} = \frac{400 \times 1000}{5000} = 80 \text{ g}$$

$$W_{bd} = W_d - W_s = 400 - 80 = 320 \text{ g}$$

2.2.3 Recommended Mixing Procedure for Emulsion/Dispersion Polymer Products (Blender Method).

2.2.3.1 Apparatus. The following apparatus is required for test solution preparation:

Blender	No-load speed of approximately 15,500 rpm fitted with 40-oz glass or stainless steel container. (For example, Waring® Blendor™ Model 7011S, or equivalent).
Variable Transformer	0-120 volts (e.g. POWERSTAT® Type L116B, or equivalent).
Top Loading Laboratory Balance	Sensitivity 0.01 g. Maximum load 2,000 g.
Disposable Plastic Syringes	1 mL, 5 mL, 10mL.
Container	16-oz, wide-mouth jar or 600-mL beaker.
Tumbler	Any device capable of tumbling a 16-oz jar end-over-end at approximately 30 rpm (usually hand made).
Overhead Stirrer Assembly	Jiffy® mixer, or equivalent caged paddle stirrer or shaft fitted with a two-inch marine blade propeller driven by a stirring motor (Sargent-Welch cone drive variable speed, catalog number OS76445, or equivalent).

Magnetic Stirrer

Coated Magnetic Stirring Bar

Stopwatch or Timer

2.2.3.2 Calculations for Preparing Stock Solution. Refer to Par. 2.2.2.2.

2.2.3.3 Mixing Procedure (Blender Method). The following procedure is recommended to mix polymer samples using blender equipment.

1. Connect the blender to the output of a variable transformer.
2. Set the variable transformer to zero volts and blender controls to "LO" and "ON."
3. If no additional activator is required, proceed to Step 5.

4. If additional activator is required, it should be added to the makeup water by the following recommended procedure:
 - a) Tare the blender container on a top loading laboratory balance and weigh a sufficient amount of makeup water to make the required stock solution. Replace the container on the blender. Determine the amount of additional activator to be added in accordance with the manufacturer's recommendations.
 - b) Fill a 1-mL plastic syringe (no needle) with activator and fully discharge the contents to waste.
 - c) Fill the syringe, wipe the outside of the syringe with a paper towel, and discharge excess activator until the approximate required amount remains in the syringe. Weigh and record the gross weight of syringe plus contents.
 - d) Set the variable transformer to 50 volts and inject the activator *onto the shoulder of the vortex* in one continuous stroke. Mix briefly and return the transformer to zero volts.
 - e) Reweigh the empty syringe to determine the exact weight of activator added.
5. After thoroughly mixing the polymer product to ensure homogeneity, weigh out the required amount of polymer product in the following manner:
 - a) Fill a 5-mL or 10-mL plastic syringe (no needle) with polymer product and discharge the contents to waste.
 - b) Fill the syringe, wipe the outside of the syringe with a paper towel and discharge excess polymer until the approximate required amount remains in the syringe. Weigh and record the gross weight of syringe plus contents.
6. Add the product to the brine by the following procedure:
 - a) Increase the variable transformer to 50 volts; start the stopwatch.
 - b) Inject the product *onto the shoulder of the vortex* as rapidly as possible using one continuous stroke. Slow injection, or injection directly onto the wall or impeller can result in poor dispersion. *NOTE:* Use of significantly higher voltages/mixing times can lead to shear degradation of the polymer, particularly when using high salinity brines. It is *not* recommended that the above procedure be used for direct preparation of polymer solutions at concentrations less than 5000 ppm active polymer. To do so may lead to either problems with inversion or excessive mechanical shear of hydrated polymer, depending on the particular activator and brine concentrations.
 - c) Increase the voltage to 70 volts for five (5) seconds, then immediately reduce the voltage to zero. The product should be evenly dispersed.
 - d) Reweigh the empty syringe and subtract the syringe weight from gross weight to determine the exact amount of polymer product added. Recalculate the precise concentration of stock solution according to Equation (2-3).
7. Follow the procedure in Steps 7 and 8 of Par. 2.2.2.3.

2.3 RECOMMENDED PROCEDURE FOR LABORATORY PREPARATION OF POLYMER SOLUTIONS FROM DRY POLYACRYLAMIDE PRODUCTS.

2.3.1 General. Solutions of polyacrylamide powders are usually prepared as a stock solution (approximately 5000 ppm) and diluted to test concentrations as required. Vigorous agitation is necessary for the initial dispersion of the dry powder. A magnetically driven laboratory stirrer should be adjusted so that the bottom of the water vortex extends 75% into the solution. The dry polymer should be sprinkled uniformly just below the upper curve or shoulder of the vortex within 30 seconds. If dispersion of the polymer is attempted over a longer time span, the higher viscosity resulting from the dissolved polymer may prevent proper wetting. Additionally, adding the dry polymer in a big slug can lead to the formation of "fish eyes." As soon as all of the polymer has been added, adjust the stirrer to a low speed (60-80 rpm) that just keeps the solid particles from settling to the bottom. The lowest possible speed should be used to avoid mechanical degradation of the polymer solution. Continue to stir the solution at low speed for 2 or 3 hours and let it stand overnight. Solutions are then ready for desired dilution by gently mixing the required amount of concentrate and brine solution. Concentrated solutions of polyacrylamide may be stored at laboratory room temperature in brown glass bottles for 2-3 weeks without loss of effectiveness. However, diluted solutions should be prepared the day they are used.

2.3.2 Apparatus. The following apparatus is required for preparation of solutions from polyacrylamide powders:

Overhead Stirrer Assembly	Jiffy® mixer, or equivalent, caged paddle stirrer or shaft fitted with a two-inch marine blade propeller driven by a stirring motor (Sargent-Welch cone drive variable speed, catalog number OS76445, or equivalent.)
Top Loading Laboratory Balance	Sensitivity 0.01 g. Maximum load 2000 g.
Container	16-oz, wide-mouth jar or 600-mL beaker.

Weighing Cups

Magnetic Stirrer Coated magnetic stirring bar.

Stopwatch or
Timer

2.3.3 Calculations for Preparing a Stock Solution Using Dry Polyacrylamide Products.

1. Using manufacturers' or other data describing the product, calculate the amount of dry polymer product required to make up the appropriate amount of stock solution. Refer to example calculations shown in Par. 2.2.2.2.

$$W_{pr} = \frac{W_s \times C_s \times 10^{-4}}{A_{pr}} \dots\dots\dots (2-1)$$

where:

W_{pr} = weight of polymer product, g.

W_s = weight of stock solution to be made, g.

C_s = concentration of polymer in stock solution, ppm.

A_{pr} = activity of polymer product, weight percent (usually 100%). *NOTE:* Dry polyacrylamide products will gain weight when exposed to the atmosphere, and activity (A_{pr}) may have to be adjusted.

2. Calculate the amount of makeup water required to make the desired amount of stock solution according to Equation (2-2).

2.3.4 Recommended Mixing Procedure for Preparing a 5000 ppm Stock Solution.

1. Weigh calculated amount of dry polymer product in a weighing cup and record weight.
2. Weigh calculated amount of the desired brine into a 16-oz, wide-mouth glass jar. Record weight. Add a 1.5-inch coated magnetic stirring bar to the jar.
3. Using a magnetic stirrer, adjust the vortex to extend 75% into the solution.
4. Sprinkle the polymer on the shoulder of the vortex over a period of 30 seconds. Observe the solution. No large slugs or "fish-eyes" should be present; if present, start over.
5. Stir the solution using a magnetic/overhead stirrer at low speed (60-80 rpm) for 2-3 hours.
6. Allow the solution to sit overnight before diluting to the desired concentration.
7. Check for undissolved particles. If present, start over.

2.3.5 Dilution of Stock Solution. Refer to Par. 2.2.2.3, Step 8.

2.4 RECOMMENDED PROCEDURE FOR LABORATORY PREPARATION OF POLYMER SOLUTIONS FROM POLYACRYLAMIDE SOLUTION CONCENTRATE.

2.4.1 General. Polyacrylamide solution concentrates are highly viscous liquids ranging in polymer content from 3 to 20 weight percent. To prepare dilute solutions in the laboratory, the concentrate must be properly handled and the correct amount added to the makeup water.

2.4.2 Apparatus. The following apparatus is required for preparation of test solutions from polyacrylamide solution concentrate:

Magnetic Stirrer
and Coated
Stirbar

Container 16-oz, screw-cap, wide-mouth,
(SCWM) glass jar.

Top Loading Laboratory Balance Sensitivity 0.01 g.
Maximum load 2000 g.

Magnetic Stirrer Coated magnetic stirring bar.

Stopwatch or
Timer

2.4.3 Calculations for Preparing Stock Solutions. Using manufacturer's or other data describing the product, calculate the amount of concentrate required to make up the desired amount of stock solution according to Equation (2-1).

2.4.4 Recommended Mixing Procedure for Preparing Stock Solution.

1. Using syringe, place the amount of desired concentrate into the tared jar on the balance.
2. Add makeup water to final weight to give desired concentration. Refer to Par. 2.2.2.2 and Equation (2-2) for calculation of required makeup water.
3. Flush jar with nitrogen then cap and stir overnight (16 hours) using a magnetic stirrer at a moderate speed (about 50% on most magnetic stirrers).
4. Solution should appear uniform when poured from one container to another. There should be no visible undissolved polymer. If present, start over.

2.4.5 Dilution of Stock Solution. Refer to Par. 2.2.2.3, Step 8.

2.5 RECOMMENDED PROCEDURES FOR LABORATORY PREPARATION OF POLYMER SOLUTIONS FROM POLYACRYLAMIDE GEL PRODUCTS.

2.5.1 General. Polyacrylamide gel products are tough, rubber-like materials that are essentially concentrated solutions of the polymers in water. Active polymer contents are often as high as 30%. To prepare solutions in

the laboratory, bulk gel samples must be reduced to small particle size before adding to the makeup water or brine. The product manufacturer can often furnish ground gel in small bead size. It is necessary to adhere closely to the manufacturer's instructions to obtain solutions of acceptable quality. If the strong brine procedure shown in Par. 2.5.4 is used for less saline brines, the time required can be reduced.

2.5.2 Recommended Procedure for Preparing Ground Gel.

2.5.2.1 Apparatus.

Guillotine or heavy duty scissors.

Heavy duty kitchen food grinder.

16-oz, screw-cap, wide-mouth (SCWM) glass jar.

2.5.2.2 Gel Preparation Procedure. Take sample gel cores from the center of the log sample. Cut the gel core sections into cubes (approximately 1 inch) and randomly mix and pass through the food grinder fitted with a die plate having $\frac{3}{16}$ -inch diameter holes. Store ground gel in the tightly sealed wide-mouth jar to avoid moisture loss.

CAUTION: Protective gloves should be worn while handling polyacrylamide gel products to prevent skin exposure to potentially hazardous materials.

2.5.3 Recommended Mixing Procedure [Stock Solution (Salt Concentrations Lower than Three Percent)].

2.5.3.1 Apparatus. The following apparatus is required for preparation of test solutions from polyacrylamide gel products:

Stirrer	Air or electric, 750 rpm, fitted with two three-blade propellers, each two inches in diameter and spaced approximately two inches apart on the stirrer shaft.
Top Loading Laboratory Balance	Sensitivity 0.01 g. Maximum load 2000 g.
Container	16-oz, SCWM jar.
Tumbler	Any device capable of tumbling a 16-oz jar end-over-end at about 30 rpm (usually homemade).
Strainers	Cylinder approximately 3 inches long by 2 inches diameter fitted with 200-mesh stainless steel screen at one end.

2.5.3.2 Calculations for Preparing Stock Solution.

1. Using manufacturer's or other data describing the product, calculate the amount of polyacrylamide gel product required to make up the appropriate amount of stock solution. Refer to example calculations shown in Par. 2.2.2.2, Equation (2-1).

NOTE: Supplier should specify the percent of active polymer in the polymer product.

2. Calculate the amount of makeup water required to make the desired amount of stock solution according to Equation (2-2).

2.5.3.3 Mixing Procedure. CAUTION: Protective gloves should be worn while handling polyacrylamide gel products to prevent skin exposure to potentially hazardous materials.

1. Place calculated amount of ground gel in a 16-oz, SCWM jar. If ground gel is not available, scissor cut the gel into very small cubes or pieces about $\frac{1}{8}$ -inch on a side (the smaller the pieces, the faster they dissolve).
2. Add calculated amount of makeup water.
3. Cap bottle and tumble overnight (16 hours) at 30 rpm and 75 F (24 C) or above.
4. Remove the solution from the tumbler and stir for 90 minutes at 750 rpm on an air or electric stirrer. Longer or faster stirring can cause shear degradation of the polymer.
5. Solution should appear uniform when poured from one container to another. Solution should be free of partially dissolved and undissolved gels when passed through the 200 mesh screen. If not, start over.

2.5.3.4 Dilution of Stock Solution. Refer to Par. 2.2.2.3, Step 8.

2.5.4 Recommended Mixing Procedure [Stock Solution (Salt Concentrations Higher Than Three Percent)]. The following procedure and apparatus are recommended for dissolution of gel in brines containing high salt concentrations (greater than three percent).

2.5.4.1 Apparatus.

1. One two-liter separatory funnel with a minimum $\frac{1}{4}$ -inch inside diameter opening at the bottom.
2. One 30-rpm peristaltic pump sized for 0.313-inch inside diameter tubing (Cole-Parmer Masterflex® 7015, or equivalent).
3. Two lengths of 0.313 inch Tygon® tubing (or equivalent), one approximately 30 inches long, the other approximately 12 inches long.
4. Gel polymer dissolving device (refer to Figure 2-1).

Figure 2-2, Sketch A illustrates the initial set up of the equipment. Connect the tubing (approximately 30 inches long) to the bottom of the separatory funnel, insert the discharge end of the tubing through the peristaltic pump head, and install the tubing to allow for return flow to the top of the separatory funnel.

2.5.4.2 Calculations for Preparing Stock Solution. Weigh out polymer and brine in proper quantities for preparing the stock solution (refer to Par. 2.5.3.2).

2.5.4.3 Mixing Procedure.

1. Add the brine to the separatory funnel and start the pump. Check the pump to make sure the speed is 30 rpm and that rotation causes the direction of the flow to match the arrow shown in Figure 2-2, Sketch A (flow toward top of funnel).
2. Add the weighed amount of ground or scissor-cut polymer to the top of the funnel while the pump is running.
3. Observe operation of the pump and movement of the polymer in the separatory funnel for a few minutes to make sure the outlet is not plugged. If the outlet appears to be plugging, pinch the tubing below the outlet several times to start flow of the polymer pieces or reverse the flow for a few seconds.
4. Circulate the polymer and water through the pump for ten hours at which time the polymer solution should be free of distinct pieces of polymer material. The solution should be hydrated and ready to be run through the polymer dissolving device.
5. Stop the pump and connect the discharge end of the tubing to the polymer dissolving device away from the screen end. Connect the shorter piece of tubing (approximately 12 inches long) to the screen end of the dissolving device and insert its discharge end into the separatory funnel top as shown in Figure 2-2, Sketch B.
6. Observe the flow of polymer through the dissolving device. If the tubing between the pump and the dissolving device appears to be ballooning and/or if there is no flow out of the dissolving device, stop the pump immediately, disconnect the device, and continue hydration treatment using Step 4 above.
7. If problems arising from lack of polymer hydration are absent, allow the solution to circulate through the dissolving device for six (6) hours. The solution should be completely homogeneous leaving the tubing at the top of the separatory funnel.
8. Collect the sample of homogenized polymer concentrate solution by placing the discharge end of the tubing into a glass or plastic container and pumping the solution into the container.

2.5.4.4 Dilution of Stock Solution. Refer to Par. 2.2.2.3, Step 8.

2.6 RECOMMENDED PROCEDURE FOR LABORATORY PREPARATION OF POLYMER SOLUTIONS FROM POLYSACCHARIDE PRODUCTS.

2.6.1 General. Polysaccharide is supplied as both dry and liquid material. Dry material is ordinarily used as 100% active. Liquid polysaccharide concentration should be obtained from the manufacturer as there are several

different methods for calculating polysaccharide concentration.

2.6.2 Recommended Mixing Procedure.

2.6.2.1 Apparatus.

Blender	Waring® Commercial Blender™ Model 7011 S, or equivalent, fitted with a 40-oz glass or steel container. No load speed of approximately 15,500 rpm.
Variable Transformer	0-120 volt (e.g., POWERSTAT® Type L116B, or equivalent).
Top Loading Laboratory Balance	Sensitivity 0.01 g. Maximum load 2000 g.
Stopwatch or Timer	

2.6.2.2 Calculations for Preparing a Stock Solution Using Liquid or Dry Polysaccharide Products.

1. Using manufacturer's or other data describing the product, calculate the amount of polymer product required to make up the appropriate amount of stock solution. Refer to example calculations shown in Par. 2.2.2.2, Equation (2-1).
2. Calculate the amount of makeup water required to make the desired amount of stock solution according to Equation (2-2).

2.6.2.3 Mixing Procedure for Dry Polysaccharides*

1. Sift dry xanthan gum into the makeup water in the blending jar, while stirring at high speed with rheostat at approximately 40% (44-45 volts).
2. Mix two minutes at 40% power.
3. Mix three minutes at 60% power.
4. Mix two minutes at 80% power.

NOTE: This procedure applies specifically to dry xanthan gum polysaccharides and may or may not be applicable to other dry polysaccharide products. Consult the manufacturers of other dry polysaccharide products for their recommended mixing procedure.

2.6.2.4 Mixing Procedure for Liquid Polysaccharides. Add the calculated amount of liquid polymer to the calculated amount of makeup water in the blender jar. Mix two minutes in the blender at 50 volts.

2.6.2.5 Dilution of Stock Solution. Dilute as desired and mix one minute in the blender at 50 volts.

***NOTE:** Solution temperature will be raised by the procedure. Cool the solution to 77 F (25 C) before subsequent testing to improve test precision and reproducibility.

2.6.2.6 Preservation of Xanthan Gum and Other Polysaccharide Solutions. If the solution is to be retained, a suitable biocide should be added. For xanthan gum solutions being retained at room temperature, 2500 ppm formaldehyde is usually sufficient. Manufacturer should be consulted for further recommendations.

2.7 RECOMMENDED PROCEDURE FOR LABORATORY PREPARATION OF POLYMER SOLUTIONS FROM SYNTHETIC LATEX POLYMERS.

2.7.1 General. The term, "latex polymer," encompasses high molecular weight acrylate copolymers containing carboxylic acid groups and formulated as an aqueous latex product. Solutions are prepared by diluting the product (active polymer content is specified by supplier) with water and neutralizing with caustic to yield the desired concentration. *A 20,000 ppm stock solution is recommended for latex polymers.*

2.7.2 Components for Preparation of 20,000 ppm Stock Solution.

- 8.25 g polymer (21% active)
- 87.10 g water
- 4.65 g sodium hydroxide (10% aq.).

NOTE: The 20,000 ppm activity of the polymer solution is due to addition of the sodium to the polymer molecule as neutralization proceeds.

2.7.3 Mixing Procedure for Preparing a 20,000 ppm Stock Solution.

1. Dilute latex product with the calculated amount of water and mix gently.
2. Add the calculated amount of NaOH solution to the diluted latex.
3. For quantities of one liter or less of stock solution, it is convenient to measure the materials into a glass jar, cap the jar, and shake it vigorously by hand for a minute or so until the liquid changes from a milky fluid to a viscous transparent solution.
4. Allow the 20,000 ppm polymer solution to age for 2 hours to attain maximum clarity and viscosity.
5. If brine is used to dilute the latex to the 2% solids level prior to neutralization with the NaOH solution and a precipitate is noted, the latex should be stabilized with Aerosol® A103, or equivalent, based on the concentration of 0.4 grams of surfactant per 100 grams of latex polymer on an "as is" basis.

2.7.4 Dilution of Stock Solution. Refer to Par. 2.2.2.3, Step 8.

LEGEND:

- A - ACRYLIC TUBE, 3/4" OD, 1/2" ID, 6" LENGTH.
- B - ACRYLIC TUBE, 1/2" OD, 3/8" ID,
6" LENGTH CUT INTO 6 PIECES;
B₁ AND B₂: 1 1/2" END PIECES,
S₁, S₂, S₃: 7/8" SPACERS, AND
S₄: 3/8" SPACER.
- C - ACRYLIC ROD, 1/2" OD, 2" LENGTH
CUT INTO 4 PIECES, EACH 1/2" LONG.
- C₁ - BORED WITH FIVE SPACED 1/8" HOLES.
- C₂ - BORED WITH SEVEN SPACED 7/64" HOLES.
- C₃ - BORED WITH NINE SPACED 3/32" HOLES.
- C₄ - BORED WITH ELEVEN SPACED 5/64" HOLES.
- D - STAINLESS STEEL SCREEN, 100 MESH,
1/2" DIAMETER.

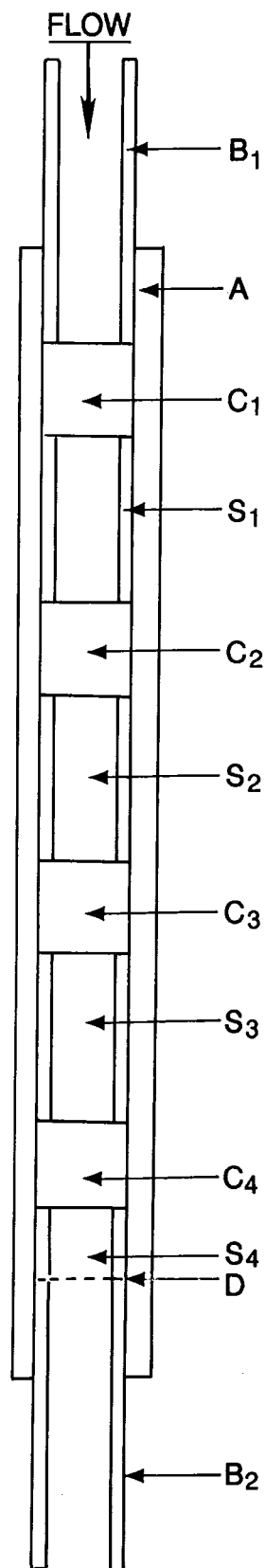
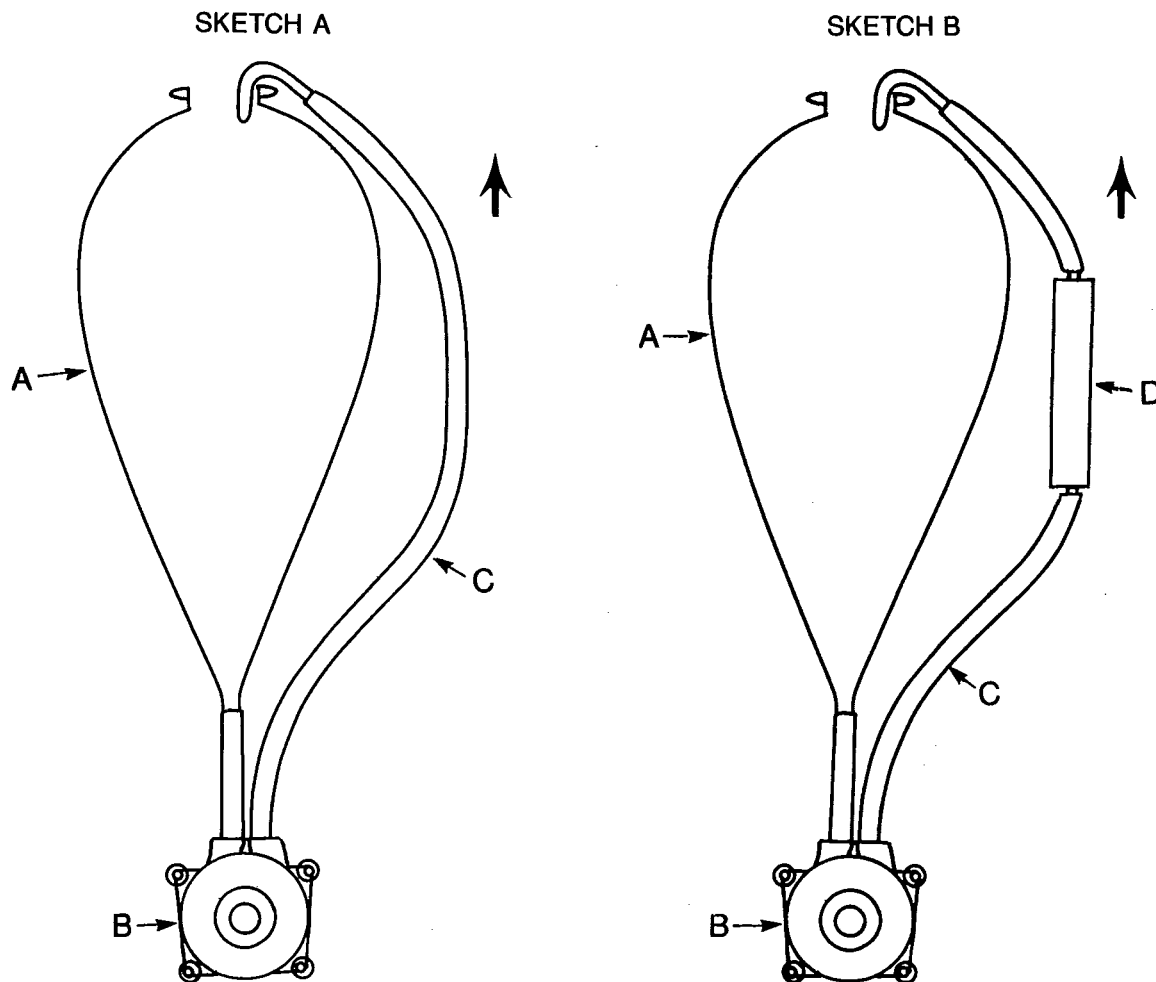


FIGURE 2-1
EXAMPLE GEL POLYMER DISSOLVING DEVICE



LEGEND:

- A - TWO-LITER SEPARATORY FUNNEL.
- B - PERISTALTIC PUMP WITH 30 RPM SYNCHRONOUS MOTOR, COLE-PARMER MASTERFLEX® MODEL 7015, OR EQUIVALENT.
- C - TYGON® TUBING, 0.313" ID, OR EQUIVALENT.
- D - GEL POLYMER DISSOLVING DEVICE.

FIGURE 2-2
EXAMPLE GEL POLYMER EQUIPMENT

APPENDIX TO SECTION 2

NOMENCLATURE

- A_{pr} = activity of polymer product, weight percent.
(NOTE: Supplier should specify percent active polymer in the polymer product.)
- C_d = concentration of polymer in the diluted solution, ppm.
- C_s = concentration of polymer in the stock solution, ppm.
- W_{bd} = weight of makeup water used in the diluted solution, g.
- W_{bs} = weight of makeup water used in the stock solution, including additional activator, if any, g.
- W_d = weight of diluted solution, g.
- W_{pr} = weight of polymer product, g.
- W_s = weight of stock solution, g.

SECTION 3

POLYMER SOLUTION RHEOLOGY AND FLOW THROUGH POROUS MEDIA

3.1 INTRODUCTION

3.1.1 General. This section describes procedures that allow a direct comparison of enhanced oil recovery polymers in a variety of standard tests. The recommended procedures for the viscometric tests are not designed to give information on a particular field water for a specific reservoir application. However, from these procedures, comparable products can be selected for additional site-specific testing, such as further viscometric tests and flow tests through reservoir core samples. The purpose of this section is to provide standard test procedures, including rheometry and flow through porous media, to permit comparison of different polymers. The recommended viscometric tests are designed to assess the effects of: 1) shear rate, 2) temperature, 3) salinity and hardness, 4) pH, and 5) polymer concentration on solution viscosity. Some simple rheological models are reviewed as well as methods for estimating intrinsic viscosity and viscosity-average molecular weight. Sufficient background information is given to enable a better understanding of the rheological behavior of polymers used in enhanced oil recovery applications*. Procedures are described for the measurement of mobility and permeability reduction obtained with polymer solutions in both linear and radial cores.

3.2 EQUIPMENT FOR VISCOMETRIC TESTS

3.2.1 General. The type of equipment required for measurement of viscosity of polymer solutions will vary depending on the intended use of the test results. For monitoring field polymer projects, instruments that are rugged and relatively inexpensive are necessary. For careful research work in the laboratory, instruments that are more accurate or more sophisticated are often required. To operate research-grade rheometers, the manufacturer's instructions should be followed. Procedures are given herein for some of the more common and simpler equipment used for viscosity measurements. Certain advantages associated with several commercial instruments are highlighted in Table 3.1.

3.2.2 Field Monitoring. Viscosities of polymer solutions prepared for injection in the field are monitored as a control measure. Polymer solutions are sampled according to the procedures outlined in Par. 6.4. Using a 4-speed (or 8-speed, if available) Brookfield® Synchro-Lectric Viscometer (or equivalent), the solution viscosity is measured with the Ultra Low (UL) Adapter at a given temperature ± 2 F (± 1.1 C). If readings for the

injected fluid exceed the maximum dial reading of 100, dilution is required. If the dial reading is less than 10, the accuracy of the measurement is poor. If dilution is necessary, the final solution weight for a given amount of field solution sample can be calculated by the following formula:

$$\text{Dilution Factor} \times \text{Weight of Sample} = \text{Final Wt.} \dots (3-1)$$

The desired amounts of solution and water are mixed by gentle shaking or with a magnetic stirrer. The solution viscosity of the field sample or the diluted sample is measured at 6 rpm and at one other speed, preferably one which keeps the scale reading between 10 and 90.

TABLE 3.1
VISCOSITY MEASURING INSTRUMENTS

NOTE: Viscometers suitable for drilling fluid measurements may not be suitable for use in testing enhanced oil recovery polymers.

Brookfield® Synchro-Lectric Model LVF (4-speed), Model LVT (8-speed), or Models LVTDV-I or LVTDV-II (Digital), Model HATD (8-speed), all with Ultra Low (UL) and Small Sample Adapters

Advantages

- Convenient to use.
- Rugged construction.
- Can be portable.

Contraves® LS-30*

Advantages

- Accuracy.
- Reasonable range of shear rates, especially low shear — even for low viscosity fluids.
- Relatively easy to use.

Haake RV-20

Advantages

- Accuracy.
- Lower shear rate than comparably priced equipment.
- Broad temperature control.
- Computer control, easy to use.

* All text references for Section 3 can be found in Appendix to Section 3.

* A fixed-amplitude, oscillatory drive is available as an option for linear viscoelastic measurements.

TABLE 3.1 (Continued)

Rheological instruments that measure properties in addition to viscosity (normal stresses and/or dynamic viscoelastic properties).

Haake RV-100/CV-100*

Advantages

Accuracy.

Reasonable range of shear rates, but not quite as low as Contraves® for low viscosity fluids.

Good temperature control, higher operating temperature than Contraves®.

Rheotech International VER**

Advantages

Accuracy.

Broad range of shear rates and frequencies.

Good temperature control.

Computer control, easy to use.

Rheometrics Fluids Rheometer Model RFR-7800**

Advantages

Accuracy.

Broad range of shear rates.

Choice of test geometries and transducers.

Easier to use and analyze data than some other instruments.

Broad temperature range — options to provide wider range than Contraves® or Haake.

Microprocessor-based system for operational control and data analysis.

* A variable-frequency, variable-amplitude assembly is available as an option. Utilizes an air bearing on the CV-100 option.

** Utilizes an air bearing.

Rheometrics Mechanical Spectrometer RMS-705F*

Advantages

Accuracy.

Better suited for low shear rate range than Fluids Rheometer.

Very broad range of shear rates and frequencies.

Good temperature control over a wide range of temperatures.

Microprocessor-based system.

Rheometrics Fluids Spectrometer RFS-8400*

Advantages

Specifications and features similar to the Fluids Rheometer, except for normal force measurements.

Uses a force-rebalance transducer (instead of the XYZ-force transducer used in the older RFR-7800 and RMS-705F models) to reduce electronic drift during measurements.

Microprocessor-controlled operation.

Rheometrics Mechanical Spectrometer RMS-800FL*

Advantages

Improved version of RMS-705F, especially for normal stress measurements; among state-of-the-art instruments for rheological characterization of fluids.

Features and specifications similar to (or better than) the previous model.

Uses a force-rebalance transducer.

Weissenberg Rheogoniometer™**

Advantages

Accuracy.

Very broad range of shear rates and frequencies.

Good temperature control.

3.2.3 Comparative Testing of Polymers. In order to evaluate a polymer as a potential candidate for a polymer flood, the rheological behavior of the polymer in solution should be measured under a variety of conditions. Viscosity measurements should be made using the 8-speed Brookfield® Synchro-Lectric Viscometer and the UL Adapter, or equivalent, at a minimum of 4 shear rates, if possible. Measurements should begin at the lowest rpm (shear rate) and work upward. Such measurements will yield information about the shear dependence of the polymer viscosity under a given set of conditions (refer to Par. 3.4.1). Recommended conditions for measuring solution viscosity are shown in Pars. 3.4.1 through 3.4.5.

3.2.4 Research Work. Laboratory research work often requires greater accuracy than is necessary for other tests. Depending on the primary research needs, the viscosity measuring instruments (refer to Table 3.1) should be carefully selected for use in this testing.

3.3 MEASUREMENT OF RHEOLOGICAL PROPERTIES

3.3.1 Dissolution of Polymers. Prepare polymer solution in the desired brine system according to applicable recommended procedures shown in Section 2. Prepare at least 100 g of this solution.

3.3.2 Measurement With Brookfield® Synchro-Lectric Viscometer. The procedures that follow apply to the Brookfield® Synchro-Lectric Model LVF (4-speed), Model LVT (8-speed), Model LVTDV (8-speed, digital readout), and Model HATD (8-speed) viscometers. Since the Model LVTD does not have a clutch, readings may be obtained without turning the motor off. All future references to use of a clutch pertain only to Brookfield Models LVF and LVT. The calibration of the Brookfield viscometer should be checked at suitable intervals by the use of standard viscosity fluids. Viscosity oils conforming to *ASTM Designation D 446-74*⁶ should be used to check this equipment. If equivalent viscometers are used, refer to the equipment manufacturer for operating and calibration instructions.

3.3.2.1 Apparatus.

1. Brookfield® Synchro-Lectric LV Series Viscometer.
2. Standard viscosity fluids.
3. UL Adapter (open cup).
4. Constant temperature bath.
5. 25-mL graduated cylinder.

3.3.2.2 Procedures.

1. Lift the lower shaft of the instrument and attach the spindle nut. After attaching the adapter body, place the hook on the UL spindle through the eyelet of the spindle nut.
2. Transfer 16 mL of the polymer solution to be measured into the UL adapter cup. Some cup

designs may require different volumes. Refer to the manufacturer's operating instructions for specific information on equipment volume requirements.

3. With the spindle held at an angle, slowly immerse the spindle into the polymer solution. This procedure will ensure that air is not trapped beneath the spindle.
4. Attach the cup to the main adapter body.
5. Set the speed desired on the speed selector knob. Start at the lowest rotational speed and work upward. Although not absolutely necessary, speeds should be changed while the motor is running and the clutch is depressed.
6. Immerse the cup into the constant temperature bath up to the main adapter and start rotation of the spindle by depressing the clutch, turning on the motor, and releasing the clutch.
7. Allow the temperature to equilibrate in the cup for at least 10 minutes at 100 F (37.8 C) or longer at higher temperatures.
8. To take a reading on the scale, depress the clutch and turn the motor off with the pointer in view.
9. Record the rotational speed and the dial reading.
10. Obtain a second reading by turning on the motor with the clutch still depressed, releasing the clutch, waiting for the pointer to stabilize, and repeating Step 8. Obtain as many check readings as necessary.
11. With the motor on and clutch depressed, increase the rotational speed to the next higher value, and repeat Steps 8 through 10. The time required for the dial to stabilize depends on the rotational speed; at speeds less than 6 rpm, it may take at least the time required for 3 to 4 revolutions of the dial, while at higher speeds only about 30 seconds may be required.
12. The viscosity of the solution in centipoise (cP) or millipascal second (mPa·s) is determined by using the conversion factors shown in Table 3.2.
13. Calibration of the instrument should be checked on a regular basis with the standard viscosity fluids described in Par. 3.3.5.

3.3.3 Measurement of Screen Factor. This measurement is related to the viscoelastic nature² of solutions of some synthetic polymers such as polyacrylamides and is most affected by the larger polymer molecules.

3.3.3.1 Apparatus. Screen factor bulbs³ (screen factor apparatus, refer to Figure 3-1) are commercially available. This equipment is sometimes called a screen viscometer; however, it does not measure viscosity. The equipment is more properly called a screen factor apparatus (refer to Figure 3-1).