

Pemulen™ Polymeric Emulsifiers

Emulsion Viscosity and Stability

I. Scope:

This procedure is used to determine the viscosity of Pemulen™ polymeric emulsifiers in oil-in-water emulsions and to demonstrate the stability of these emulsions.

II. Abstract:

Acrylic acid polymers co-polymerized with fatty comonomers acquire amphipathic properties, allowing the polymer to adsorb at the interface of the oil and water. An oil-in water emulsion is thus stabilized against separation by the presence of the polymer. In addition to the emulsification property, the Pemulen polymeric emulsifiers are also viscosity building. The viscosity of the neutralized emulsion is determined with a Brookfield viscometer.

The stability of the emulsion can be demonstrated by allowing to set for twenty-four hours..

III. Safety Precautions:

1. Wear safety goggles and gloves.
2. Pemulen polymeric emulsifier dust is irritating to the respiratory passages and breathing it should be avoided.
3. Pemulen polymeric emulsifier dust in the eyes should be thoroughly rinsed with 1% physiological saline solution for 15 minutes, then see a physician. If saline solution is not readily available, rinse with plenty of clean water for 15 minutes, then consult a physician.
4. Sodium hydroxide solutions will cause burns to the skin and eyes. Flush any contact site with large quantities of water.

IV. Interferences:

Minute amounts of most metallic ions decrease the emulsification and thickening efficiency of Pemulen. Sources of metallic ion contamination are the water, sodium hydroxide and mineral oil. Chemically pure sodium hydroxide, USP grade mineral oil and demineralized water should be used.

V. Apparatus:

1. Torsion or other balance capable of ± 0.002 gram accuracy.
2. Balance capable of weighing 500 grams with ± 0.1 gram accuracy.
3. Lightnin' Labmaster Mixer, Model DS1010, with three-blade marine impeller. Mixing Equipment Co., c/o Clemens & Associates, 221 Rochester Street, Avon, NY 14414. (See Appendix for diagram of three-blade marine impeller.)
4. Brookfield Viscometer Model RVT or RVF with modified guard capable of operation at 20 rpm. Brookfield Engineering Laboratories, 240 Cushing Street, Stoughton, MA 02072-2398.
5. Spindle set for Brookfield Viscometer, 316 s/s RV (see address in #4).
6. Brookfield Viscosity standard oils (see address in #4).
7. Griffin beaker, 600 ml. Fisher Scientific, Catalog No. 02-540M.
8. Griffin beaker, 250 ml. Fisher Scientific, Catalog No. 02-540K.
9. Griffin beaker, 50 ml. Fisher Scientific Catalog No. 02-540G.
10. Spatula. Fisher Scientific, Catalog No. 14-365A.
11. Rubber policeman. Fisher Scientific, Catalog No. 14-105A.
12. Aluminum weighing dish. Fisher Scientific, Catalog No. 08-732.
13. pH meter.

Lubrizol Advanced Materials, Inc. / 9911 Brecksville Road, Cleveland, Ohio 44141-3247 / TEL: 800.379.5389 or 216.447.5000

The information contained herein is being furnished for informational purposes only, upon the express condition that the User makes its own assessment of the appropriate use of such information. While the information contained herein is believed to be reliable, no representations, guarantees or warranties of any kind are made as to its accuracy, suitability for a particular application or the results to be obtained herefrom. Lubrizol Advanced Materials, Inc. ("Lubrizol") cannot guarantee how any products associated with this information will perform in

combination with other substances or in the User's process. Due to variations in methods, conditions and equipment used commercially in processing these materials, no warranties or guarantees are made as to the suitability of the information or products for the applications disclosed. Lubrizol shall not be liable and the User assumes all risk and responsibility for any use or handling of any material beyond Lubrizol's direct control. LUBRIZOL MAKES NO WARRANTIES, EXPRESS OR IMPLIED, INCLUDING, BUT NOT LIMITED TO,

THE IMPLIED WARRANTIES OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE. It is the User's sole responsibility to determine if there are any issues relating to patent infringement of any component or combination of components relating to the supplied information. **Nothing contained herein is to be considered as permission, recommendation, nor as an inducement to practice any patented invention without permission of the patent owner.**

For further information, please visit: www.lubrizol.com/personalcare

VI. Reagents:

1. Demineralized water, specific resistance of 1 megohm minimum.
2. Sodium hydroxide pellets. Fisher Scientific, Catalog No. S318 (see Special Instruction No. 1 for preparation of 18% sodium hydroxide).
3. USP grade mineral oil (Drakeol 21 from Penreco or Amoco White Mineral Oil No. 21).
4. pH buffer solutions, Fisher Scientific. pH = 4 buffer, Catalog No. SB101-500. pH = 7 buffer, Catalog No. SB107-500. pH = 10 buffer, Catalog No. SB115-500.

VII. Procedure:

1. Measure 206.4 grams of demineralized water with a specific resistance of one megohm or greater into a 600 ml beaker.
2. Set the speed of the Lightnin' mixer with three-blade marine impeller to 750 ±50 rpm.
3. Weigh 0.6 gram of sample polymer into an aluminum weighing dish.
4. Place the 600 ml beaker containing the demineralized water under the stirrer with the shaft set at an angle of 60 degrees and to the side of the beaker. The three-blade marine impeller should be as near the bottom of the beaker as possible, without touching as the blade turns.
5. Turn the mixer on, and hold the aluminum weighing dish containing the weighed Pemulen sample a few inches above the water surface. Tilt the weighing dish to the point that the sample is ready to spill out. Tap the side of weighing dish to the point that the sample is ready to spill out. Tap the side of the dish lightly, allowing the polymer to slowly sift into the water. Total addition time should be 45 seconds to 1 minute. (CAUTION: DO NOT allow the polymer to form a continuous film or "skin" on the surface of the water, as agglomerates will form and dispersion will not be complete.)
6. Continue mixing for 15 minutes to allow the polymer to swell sufficiently.
7. Measure 90 grams of mineral oil into the 250 ml beaker.
8. When the 15 minute mixing period is complete, adjust the beaker so that a smooth vortex is visible. Slowly trickle the oil (almost dropwise) into the center of the vortex along the mixer shaft. Total addition time should be 1 ½ to 2 minutes.
9. Continue mixing for 5 minutes.
10. Weigh out 1.4 grams of 18% sodium hydroxide. (See Special Instruction 1 for preparation.)

11. Slowly add the sodium hydroxide at the end of the 5 minute mixing. The solution will almost immediately thicken and thorough mixing requires careful attention. Increase mixing speed to 1300 RPM as soon as the sodium hydroxide has been added. Move the beaker up and down to completely blend in the oil droplets, especially on the surface and along the bottom.
12. Continue mixing for 5 minutes or until no oil is evident and a smooth consistency is achieved.
13. Check pH. pH should be between 7.3 to 7.8. If pH is too low, add additional sodium hydroxide. If pH is too high, start test procedure over, holding out a small amount of neutralizer.
14. Measure viscosity. (Refer to Calculation section and Special Instruction No. 2)
15. Store the beaker containing the emulsion and return in 24 hours to determine if the emulsion is stable. A layer of oil on the surface indicates a failed emulsion.

VIII. Calculations:

VISCOSITY, cPs = Dial reading x constant for spindle used

BROOKFIELD RVT CONSTANTS

Pemulen™	TR-2	TR-1
Spindle #	4	5
Constant	100	200

IX. Special Instructions:

1. Preparation of 18% sodium hydroxide. Slowly add 45 grams of sodium hydroxide pellets to 205 ml of demineralized water. CAUTION: Considerable heat will be generated as the sodium hydroxide dissolves, so the container should be cooled. Store in an air-tight container to avoid sodium carbonate formation. Exercise extreme care to avoid contact with the skin or eyes.
2. All dial readings on the viscometer should be greater than 10 and less than 90 for maximum precision. Make certain the viscometer stand is level (bubble indicator is centered) and the spindle is placed near the center of the beaker and at the proper depth. Move the spindle to avoid large air bubbles.

X. Time:

Attention: 30 minutes Elapsed: 1 hour

XI. References:

1. Lubrizol Procedure No. 430-I, Determination of Brookfield Viscosity.