

# Brookfield Viscosity of Carbopol<sup>®</sup> Polymer Mucilages After Salt Addition

## Scope:

This procedure is used to determine the Brookfield Viscosity of Carbopol<sup>®</sup> polymer-based mucilages after the addition of sodium chloride.

## Abstract:

Mucilages based on Carbopol polymers retain viscosity even in the presence of relatively high levels of inorganic salts. Part I of the Procedure section is equivalent to Lubrizol Test Procedure 430-I. Part II demonstrates the ion resistance of the mucilages prepared in Part I.

## Safety Precautions:

1. Wear safety goggles and gloves.
2. Carbopol polymer dust is irritating to the respiratory passages and breathing it should be avoided.
3. Carbopol polymer 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. Protective clothing, including apron and chemical resistant gloves and goggles should be worn.

## Interferences:

Inorganic salts or other impurities in the test materials or any of the glassware or apparatus used will affect the resultant viscosity.

## Apparatus:

1. Analytical balance capable of  $\pm 0.002$  gram accuracy.
2. Lightnin' Labmaster mixer, Model DS1010, with 3-blade marine impeller. (See Appendix I.)
3. Fisher or other mixer capable of 300 RPM, with 3.25-inch "S"-blade stirrer. (See Appendix II.)
4. Constant temperature water bath. Precision Model 186. Fisher Scientific Catalog No. 15-474-20.
5. Brookfield Viscometer, Model RVF, RVT, or RVTD, with modified guard. (See Special Instruction Item 1 for modification.)
6. Spindle set for Brookfield Viscometer, 316 s/s RV.
7. Brookfield Viscosity Standard Oils. Stock fluids available at 1000, 5000, 12500, 30000, 60000 and 100000 mPa·s. Calibrations should be made using an oil near the working range of viscosity.
8. Griffin beaker, 800 or 1000 ml.
9. Graduated cylinder, 500 ml.
10. Fisher chain clamp #05-745.
11. Rubber policeman.
12. Centigrade thermometer.
13. Aluminum weighing dish.
14. pH meter.
15. Automatic burette attached to a Handiboy for dispensing 18% NaOH.
16. Pyrex tannin dish, 70 x 50 mm.
17. Desiccator.

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## Reagents:

(All reagents are available from Fisher Scientific.)

1. Sodium hydroxide pellets. (See Special Instruction 2 for preparation of 18% sodium hydroxide.)
2. Sodium chloride, ACS certified. Fisher Scientific, Catalog No. S-271.

Distilled or demineralized water, specific resistance of 1 megohm minimum.

3. Phenolphthalein indicator solution, 0.1% in 60% methanol.
4. Bromothymol blue indicator solution, 0.1% in water.
6. pH-buffer solutions for calibrating pH meter.

## PROCEDURE:

### Part I: Mucilage Preparation

NOTE: The determination of Brookfield Viscosity of Carbopol mucilages is extremely sensitive to such variables as shear, temperature, time and pH. These variables should be controlled when making a viscosity determination. Gross errors can be introduced by changing conditions.

1. Spread sample evenly in an open aluminum or glass dish. Dry at 176°F (or 80°C) and 29 inches of mercury for one hour.
2. Set the speed of the Lightnin' mixer with the three-blade marine impeller at 1000 ±10 RPM.
3. Measure 500 ml. distilled or demineralized water in a graduated cylinder and transfer to a beaker (800 or 1000 ml).
4. Place the beaker under the Lightnin' mixer with the shaft set at an angle of 60° and to one side of the beaker. Fasten the beaker securely with a chain clamp. The impeller should be as near the bottom of the beaker as possible. (See Note 2.)
5. After drying the Carbopol polymer, remove to a desiccator and allow to cool to room temperature.
6. After the Carbopol polymer reaches room temperature, weigh out polymer from the dried sample to ±0.005 grams onto an aluminum dish. The appropriate amounts for the mucilage concentrations are shown below:
  - 0.25 gram for 0.05% solution
  - 1.00 gram for 0.2% solution
  - 2.50 grams for 0.5% solution
  - 5.00 grams for 1.0% solution
  - 20.00 grams for 4.0% solution

7. Turn on the mixer and carefully begin to add the Carbopol polymer. Tilt the aluminum dish and tap the side, causing the Carbopol polymer to slowly sift into the water. Total addition time should be 3/4 to 1 1/2 minutes. CAUTION: If addition is too rapid, the Carbopol polymer will agglomerate on the surface of the water. Inadequate dispersion will result and gross errors in the final mucilage viscosity can be expected. A wire sieve can be used to facilitate incorporation of the Carbopol polymer into the water.
8. When all of the Carbopol polymer has been added, start the timer and continue stirring at 1000 RPM for 15 minutes. (A timed outlet on the stirrer is useful.) Scrape any polymer from the sides of the beaker and stirrer shaft with the rubber policeman.
9. If indicators are required, (See Special Instruction 3.), add five drops phenolphthalein indicator (0.1% in 60% methanol) and 15 drops bromothymol blue (0.1% in water).
10. Remove the dispersion from the stirrer and allow to stand in a water bath at 25 ±1 degree C for 30 minutes. This assures complete polymer hydration and allows any foam to break. If foam remains, it should be completely eliminated. A convenient method is to carefully direct an air stream at the surface of the dispersion.
11. Neutralize the mucilage using the appropriate amount of 18% NaOH. (The approximate amounts to be used for the various mucilage concentrations are suggested.)
  - 0.4 ml for 0.05 % mucilage
  - 1.8 mls for 0.2% mucilage
  - 5.4 mls for 0.5 % mucilage
  - 11.3 mls for 1.0% mucilage
  - 40.5 mls for 4.0% mucilage
12. Adjust the mixer with the "S" paddle to a speed of 300 ±25 RPM. With the mixer off, set the paddle at a depth just below the surface so that air will not be drawn into the mucilage. Turn the mixer on and stir for 2 minutes, moving the beaker up and down being aware that air should not be introduced. (If the "S"-paddle mixer is not available, see Special Instruction 3.)
13. Check the pH of the mucilage with the pH meter. If the pH is below 7.3, add additional 18% NaOH and mix again. The desired pH range is 7.3 - 7.8. If the pH is above 7.8, discard the sample and remake the mucilage.

14. Return the neutralized mucilage to the 25°C water bath for one hour. NOTE: The viscosity determination should be made after 60 to 75 minutes to avoid slight viscosity changes occurring with time.
16. a) For RVF and RVT model viscometers, take readings at 3, 6 and 9 revolutions and record the average of these readings. The total range of the three readings must not exceed 0.5 scale units on the 100 unit scale.
- b) For RVT model viscometers, record the maximum reading between 30 and 45 seconds.

NOTE: All readings on the viscometer should be greater than 10 and less than 90. If readings are outside the 10-90 scale unit range, choose a different spindle (select the spindle which gives the highest reading for greater precision).

**Calculations:**

$$\text{VISCOSITY (mPa}\cdot\text{s)} = \text{DIAL READING} \times \text{CONSTANT FOR SPINDLE USED}$$

where constant for the various spindles is:

<u>Spindle</u>	<u>Constant</u>
3	50
4	100
5	200
6	500
7	2000

**Part II: Salt Addition**

1. The desired mucilage (0.2%, 0.5%, 1.0%) is prepared as specified in Lubrizol TP-430-I.
2. After reading the mucilage viscosity, add the appropriate amount of salt. The salt can be weighed in an aluminum weighing dish.

<u>SALT CONC.</u>	<u>AMOUNT</u>
0.2%	1.0 GRAM
0.5%	2.5 “
1.0%	5.0 “

3. Mix the mucilage thoroughly with the “S”-blade paddle mixer at low speed. Complete mixing can be accomplished in 30-60 seconds.
4. Return the mucilage to the constant temperature water bath (25 + 2° C) for 30 minutes.
5. Measure the viscosity of the mucilage using the Brookfield Viscometer Model RVF, RVT, or

15. Measure the mucilage viscosity using a Brookfield RV viscometer at 20 RPM. The spindle to be used for the Carbopol type being tested is shown in Section VIII (Calculations).

RVTD set at 20 RPM. See NOTE 1 for selection of spindle.

- (a) For RVF and RVT model viscometers, take readings at 3, 6 and 9 revolutions and record the average of these readings. The total range of the three readings must not exceed 0.5 scale units on the 100 unit scale.
- (b) For RVT model viscometers, record the maximum reading between 30 and 45 seconds.

**Special Instructions:**

**1. Dimensions of modified guard:**

The guard is cut to a length of 7 3/4 inches when measured from the base of the viscometer box to the bottom of the guard. Without the modification, the #7 spindle can not be immersed to the proper depth when using a 1000 ml. beaker.

**NOTES:**

1. All readings on the viscometer should be greater than 10 and less than 90. If readings are outside the 10-90 scale unit range, choose another spindle.

**TIME:**

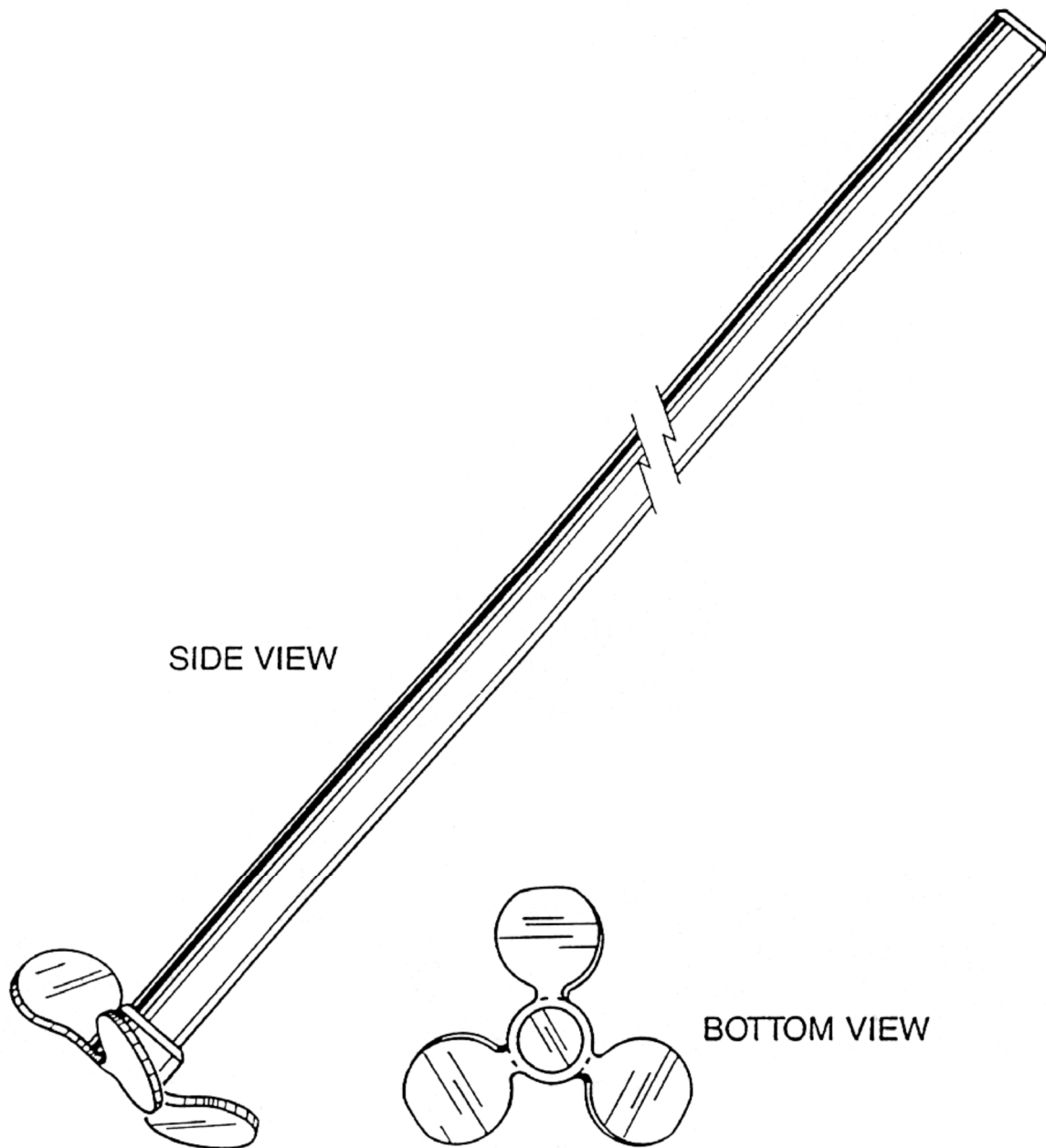
ATTENTION: 10 minutes

ELAPSED: 40 minutes

**References:**

Lubrizol Procedure No. 1291

**Appendix I**  
**(Actual Size)**



**Appendix II**  
**(Actual Size)**

