

## Determination of Brookfield Viscosity

### Applicable Products: Carbopol<sup>®</sup>\* ETD Polymers

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#### Scope:

This procedure is used to determine the Brookfield viscosity of Carbopol<sup>®</sup> ETD polymer mucilages.

#### Abstract:

The viscosity of a partially neutralized dispersion of Carbopol<sup>®</sup> ETD polymer is determined with a Brookfield viscometer.

#### Interferences:

Minute amounts of most metallic ions interfere with the swelling activity of these polymers. Sodium hydroxide and the water used are potential sources. Chemically pure sodium hydroxide and deionized water should be used.

#### Safety Precautions:

1. Wear safety goggles and gloves and follow good laboratory practices.
2. Polymer dust is irritating to the respiratory passages and inhalation should be avoided.
3. Sodium hydroxide pellets and solutions will cause burns to the skin and eyes. Flush any contact site with large quantities of water.
4. See all Material Safety Data Sheets (MSDS) for additional safety and handling information.

#### Apparatus:

1. Analytical balance capable of  $\pm 0.001$  gram accuracy.
2. Laboratory mixer (capable of  $1000 \pm 10$  rpm and  $300 \pm 10$  rpm) with 3-blade marine impeller (see Appendix I).
3. Laboratory mixer capable of  $300 \pm 10$  rpm with 3.25-inch "S"-blade stirrer (see Appendix II).
4. Constant temperature water bath controllable at  $25 \pm 0.1^\circ\text{C}$ .
5. Brookfield Viscometer, RV.
6. Spindle set for Brookfield Viscometer, 316 stainless steel RV.
7. Beaker, 800 mL.
8. Spatula or rubber policeman.
9. Thermometer.
10. Weighing dish.
11. pH meter equipped with a calomel-glass electrode.
12. Automatic burette.
13. Heat safe weighing bottle with top.
14. Desiccator with silica gel or other suitable desiccant.
15. Vacuum oven controlled at  $80 \pm 2^\circ\text{C}$  ( $176 \pm 4^\circ\text{F}$ ) with a vacuum of 29 inches (736 mm) Hg.

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**Reagents:**

1. 18% sodium hydroxide (If not purchased, see Special Instruction 1 for preparation)
2. Deionized water.

**Procedure:**

1. The determination of the Brookfield viscosity of a Carbopol® ETD polymer mucilage is extremely sensitive to shear, temperature, time and pH. These variables should be controlled.
2. Transfer sufficient sample to a heat safe weighing bottle and dry (uncapped) in a vacuum oven controlled at  $80 \pm 2^\circ\text{C}$  ( $176 \pm 4^\circ\text{F}$ ) with a vacuum of 29 inches (736 mm) Hg for 1 hour. Cap the weighing bottle and move to a desiccator to cool to room temperature.
3. After the polymer reaches room temperature, weigh to  $\pm 0.005$  gram onto a weighing dish. The appropriate amounts for the mucilage concentrations are shown below.

DESIRED MUCILAGE CONCENTRATION (%)	POLYMER (g)
0.2	1.00
0.5	2.50
1.0	5.00

4. With the mixer in the off position, set the shaft with the 3-blade marine impeller at an angle of  $60^\circ$  and the mixer speed at  $1000 \pm 10$  rpm.
5. Weigh 500 mL deionized water into an 800 mL beaker.
6. Set the beaker under the mixer with the impeller to one side and as near the bottom of the beaker as possible (See Note 1).
7. Turn on the mixer and carefully begin to add the polymer. Tilt the weighing dish and tap the side, causing the polymer to slowly sift into the water. Total addition time should be 45 to 90 seconds. CAUTION: If addition is too rapid, the polymer will agglomerate on the surface of the water. Inadequate dispersion will result and errors in the final mucilage viscosity can be expected.
8. When all of the polymer has been added, start the timer and mix at  $1000 \pm 10$  RPM for two minutes. Scrape any polymer from the sides of the beaker and stirrer shaft with a spatula or rubber policeman. After two minutes, reduce the mixer speed to  $300 \pm 10$  rpm and continue

stirring for 13 minutes for a total mixing time of 15 minutes.

9. When the mixing is complete, remove the impeller from the dispersion.
10. Place the dispersion in a water bath at  $25 \pm 0.1^\circ\text{C}$  for 30 minutes. In addition to bringing the dispersion to temperature, polymer hydration continues and any foam formed during the mixing is allowed to break. If foam persists, it should be completely eliminated. A convenient method to remove foam is to carefully direct an air stream at the surface of the dispersion.
11. Neutralize the dispersion using the appropriate amount of 18% NaOH. (The approximate amounts to be used for the various mucilage concentrations are shown in the table below). An automatic burette is convenient for delivering the NaOH.

DESIRED MUCILAGE CONCENTRATION (%)	APPROXIMATE AMOUNT OF NaOH (18%) ml
0.2	1.0
0.5	3.0
1.0	6.4

12. Adjust the mixer with the "S" paddle to a speed of  $300 \pm 10$  RPM. With the mixer off, hold the beaker under the mixer with 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 Note 2.)
13. Return the neutralized mucilage to the  $25 \pm 0.1^\circ\text{C}$  water bath for one hour.
14. Remove mucilage from the water bath after the one hour period and mix again with the "S-blade" mixer set at 300 rpm for 30 seconds.
15. Check the pH of the mucilage. The pH should be in the range 5.8 - 6.3. If the pH is below 5.8, add additional 18% NaOH and mix again. If the pH is above 6.3, discard and remake the mucilage.
16. The viscosity determination should be made between 60 and 75 minutes after maintaining the sample at  $25 \pm 0.1^\circ\text{C}$ . Measure the mucilage viscosity using a Brookfield viscometer at 20 RPM. Determine the spindle to be used from the chart in the Calculation Section.
17. Allow the viscometer reading to stabilize, normally 30 to 45 seconds. Read and record the results.

NOTE: All readings on the viscometer should be greater than 20 and less than 80. If readings are outside the 20-80 scale unit range, choose a different spindle (select the spindle which gives the highest reading for greater precision).

**Calculations:**

VISCOSITY (cps) =  
DIAL READING X MULTIPLIER FOR SPINDLE USED  
Where multiplier for the various spindles are:

<u>RV Spindle</u>	<u>Multiplier</u>	<u>Effective Range</u> cPs (mPa·s)
2	20	400 - 1600
3	50	1,000 - 4,000
4	100	2,000 - 8,000
5	200	4,000 - 16,000
6	500	10,000 - 40,000
7	2000	40,000 -160,000

**Notes:**

1. The angle of 60° and placement of the stirring shaft to one side of the beaker creates vigorous agitation with a minimum of vortexing.
2. If the “S”-blade stirrer is not available, a spatula may be used to accomplish the mixing. Two minutes of vigorous mixing is required to accomplish a homogenous mucilage.

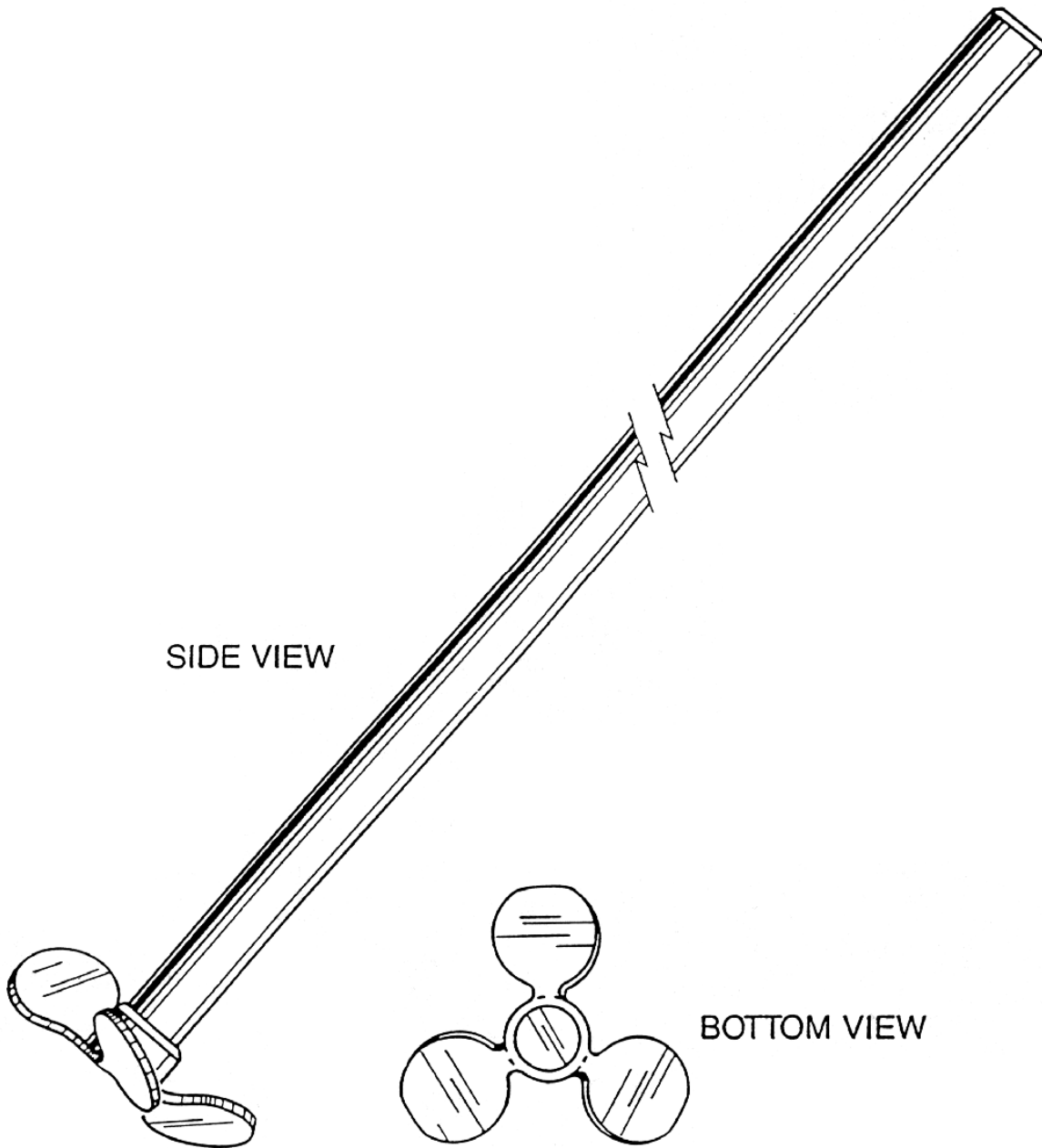
**Special Instructions:**

1. Preparation of 18% sodium hydroxide: Slowly add 45 grams of sodium hydroxide pellets to 205 mL deionized water. Use caution as considerable heat will be generated as the sodium hydroxide dissolves. Store in an airtight container to avoid sodium carbonate formation. Exercise extreme care to avoid contact with the skin or eyes.

**References:**

- *Current edition of the United States Pharmacopeia/National Formulary (USP/NF)*

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



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

**“S”-Blade Impeller**

