

Determination of Brookfield Viscosity

**Applicable Products: Carbopol®* Polymers, Pemulen™*
Polymeric Emulsifiers or Noveon®* AA-1 Polycarbophil**

Scope:

This procedure is used to determine the Brookfield viscosity of Carbopol® polymers, Pemulen™ polymeric emulsifiers or Noveon® AA-1 polycarbophil mucilages.

Abstract:

The viscosity of a neutralized dispersion of Carbopol® polymers, Pemulen™ polymeric emulsifiers or Noveon® AA-1 polycarbophil is determined with a Brookfield viscometer.

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 solutions will cause burns to the skin and eyes. Flush any contact sites with large quantities of water.
4. See all Material Safety Data Sheets (MSDS) for additional safety and handling information.

Interferences:

Metallic ions influence the mucilage viscosities of Carbopol® polymers, Pemulen™ polymeric emulsifiers or Noveon® AA-1 polycarbophil. Sodium hydroxide and the water used are potential sources. Chemically pure sodium hydroxide and deionized water should be used.

Apparatus:

1. Analytical balance capable of ± 0.001 gram accuracy.
2. Laboratory mixer with 3-blade marine impeller capable of 1000 ± 10 rpm (see Appendix I).
3. Laboratory mixer capable of 300 ± 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 s/s 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 cap.
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:

- 18% sodium hydroxide (If not purchased, see Special Instructions for preparation of 18% sodium hydroxide)
- Deionized water.

Procedure:

- Determination of the Brookfield viscosity of a Carbopol® polymer, Pemulen™ polymeric emulsifier or Noveon® AA-1 polycarophil mucilage is extremely sensitive to shear, temperature, time and pH. These variables should be controlled.
- 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.
- After the polymer reaches room temperature, weigh to ± 0.005 gram onto a weighing dish. The appropriate amounts for the mucilage concentrations desired are shown in the table below.

Mucilage %	Sample Weight (g)
0.05	0.25
0.2	1.00
0.5	2.50
1.0	5.00
4.0	20.00

- With the mixer in the off position, set the shaft with the 3-blade marine impeller at an angle of 60° and the mixer speed at 1000 ± 10 rpm.
- Weigh 500 mL deionized water into an 800 mL beaker.
- 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).
- 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 45-90 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.

- Continue mixing for 15 minutes at 1000 ± 10 rpm. Scrape any polymer from the sides of the beaker and stirrer shaft with a spatula or rubber policeman.
- When the mixing is complete, remove the impeller from the dispersion.
- 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.
- Neutralize the mucilage using the appropriate amount of 18% NaOH. (The approximate amounts to be used for the various mucilage concentrations are suggested). An automatic burette is convenient for delivering the NaOH.

Mucilage %	18% NaOH (mL)
0.05	0.4
0.2	1.8
0.5	5.4
1.0	11.3
4.0	40.5

- Adjust the mixer with the "S" paddle to a speed of 300 ± 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).
- Check the pH of the mucilage. If the pH is below 7.3, add additional 18% NaOH and mix again. If the pH is above 7.8, discard and remake the mucilage. The desired pH range is 7.3 - 7.8.
- Return the neutralized mucilage to the $25 \pm 0.1^\circ\text{C}$ water bath for one hour.
- 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.

16. 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 a reading is outside the 20-80 scale unit range, choose a different spindle (select the spindle which gives the highest scale reading for greater precision).

Calculations:

VISCOSITY (cp) =

DIAL READING X MULTIPLIER FOR SPINDLE USED

Multipliers for the spindles and their effective ranges:

RV SPINDLE	MULTIPLIER	EFFECTIVE RANGE (Cp)
1	5	100 - 400
2	20	400 - 1600
3	50	1000 - 4000
4	100	2000 - 8000
5	200	4000 - 16000
6	500	10000 - 40000
7	2000	40000 - 160000

Notes:

1. The angle of 60 degrees 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 homogeneous mucilage.
3. Certain Personal Care products use 10-90% of Brookfield scale unit range. Spindle selection may therefore be affected. These products include Carbopol[®] 676 polymer, Carbopol[®] 941 polymer, Carbopol[®] 1382 polymer, Carbopol[®] 5984 polymer and Carbopol[®] ETD 2050 polymer.

Special Instructions:

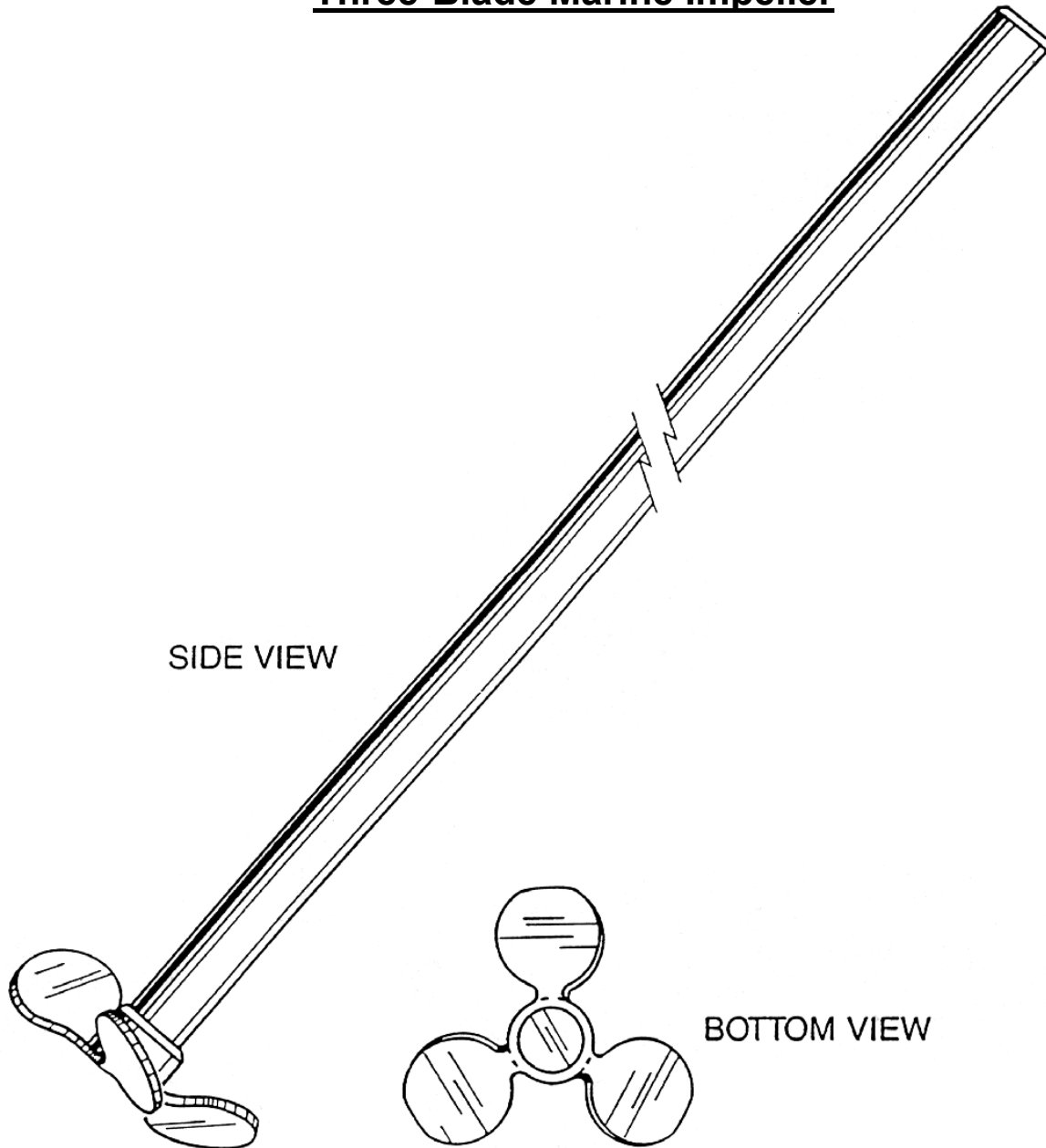
Preparation of 18% sodium hydroxide: Slowly add 45 grams of sodium hydroxide pellets to 205 ml. demineralized or deionized water. Use caution as considerable heat will be generated as the sodium hydroxide dissolves. Store in an airtight container to avoid sodium carbonate formation. Exercise extreme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

References:

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

Appendix I
(Actual Size)

Three-Blade Marine Impeller



Appendix II
(Actual Size)

“S”-Blade Impeller

