

Identification, Precipitate Formation Test

Applicable Products: Carbopol[®]* Polymers

Scope:

This procedure describes a means of identification of Carbopol[®] polymers. A 1.0% neutralized dispersion of the product will yield a white precipitate when a solution of calcium chloride is added.

Abstract:

A 1% dispersion of Carbopol[®] polymer is neutralized to form a viscous mucilage. With the addition of a 10% solution of calcium chloride, a white precipitate immediately forms.

Safety Precautions:

1. Wear safety goggles and gloves.
2. Polymer dust is irritating to the respiratory passages and inhalation should be avoided.
3. Sodium hydroxide solution 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:

There are no known interferences.

Apparatus:

1. Laboratory balance capable of ± 0.01 gram accuracy.
2. Laboratory mixer with three-blade marine impeller. (See Appendix I for diagram of three-blade marine impeller.)
3. Beaker, 800 mL.
4. Graduated cylinder, 500 mL.
5. Beaker, 50 mL.
6. Spatula or rubber policeman.
7. Weighing dish.
8. Mixer with 3.25 inch "S"-blade stirrer (see Appendix II).
9. pH meter equipped with a calomel-glass electrode.
10. Volumetric flask, 1 L.
11. Pipette, 5 mL.
12. Pipette bulb.

Reagents:

1. Deionized water.
2. NaOH solution, 1 N (see Special Instruction 2 for preparation).
3. 10% calcium chloride solution (see Special Instruction 1 for preparation).

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Procedure:

1. With the mixer in the off position set the shaft angle at 60° and the mixer speed at 1000 rpm.
2. Measure 500 mL deionized water in a graduated cylinder and transfer to an 800 mL beaker.
3. Place the beaker under the mixer with the impeller to one side of the beaker. The impeller should be as near the side and bottom of the beaker as possible (see Note 1.)
4. Weigh 5.00 g \pm 0.01 g of the Carbopol[®] polymer onto a weighing dish. This will yield a 1% dispersion in 500 mL water.
5. Turn on the mixer and carefully begin to add the Carbopol[®] 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. Incomplete hydration could influence the results of the test.
6. Continue mixing for 15 minutes at approximately 1000 rpm. Scrape any polymer from the sides of the beaker and stirrer shaft with a spatula or rubber policeman.
7. Remove the dispersion from the mixer and allow to stand for 30 minutes to assure complete polymer hydration.
8. Neutralize to a pH of approximately 7.5 with 1 N NaOH.
9. Use the "S"-blade stirrer to achieve a homogenous mucilage (see Note 2).
10. Using a spatula, transfer approximately 10 mL of the mucilage to a 50 mL beaker.
11. Pipette 2 mL of a 10% calcium chloride solution to the 10 mL of mucilage.
12. A white precipitate is immediately produced.

Calculations:

The result of the test is recorded as pass or fail.

Special Instructions:

1. Prepare 10% calcium chloride solution by adding 100 g calcium chloride to a one liter volumetric flask. Add 500 ml deionized water to hydrate the calcium chloride. Mix thoroughly. Fill to the mark with deionized water; invert several times to mix.
2. Prepare 1 N NaOH solution by adding 40 g NaOH pellets to 500 mL deionized water in a 1 L volumetric flask. Mix thoroughly. After the solution has cooled, fill to the mark with deionized water, invert several times to mix. (Solution can be purchased).

Notes:

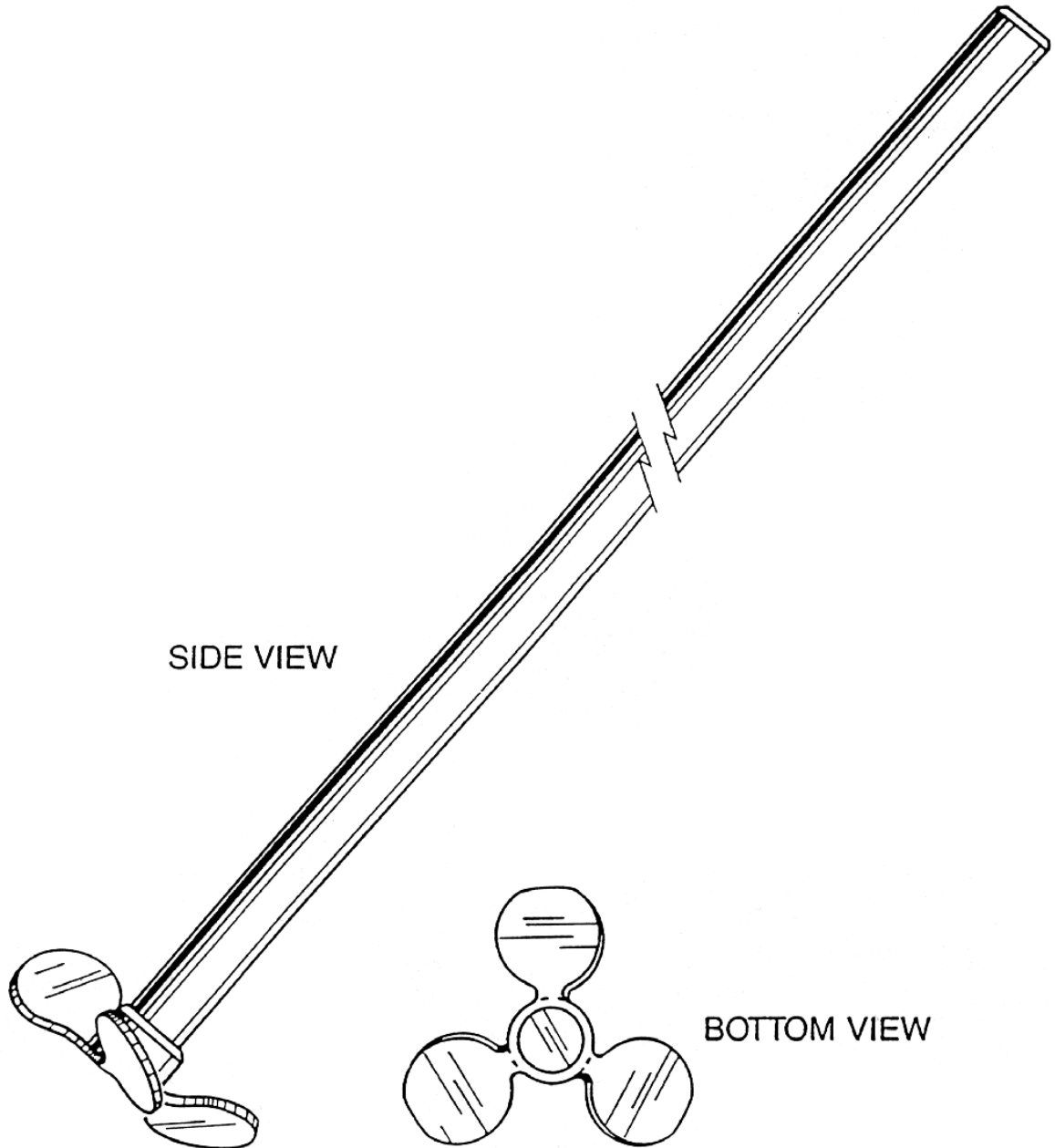
1. The angle of 60° and placement of the stirring shaft to one side of the beaker creates vigorous agitation with a minimum of vortexing.
2. If the "S"-blade stirrer is not available, a spatula may be used to accomplish the mixing. At least two minutes of vigorous mixing is required to accomplish a homogeneous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

References:

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

Appendix I

Three-Blade Marine Impeller



Appendix II
(Actual Size)

“S”-Blade Impeller

