Microcrystalline Cellulose and Carboxymethylcellulose Sodium

DEFINITION

Microcrystalline Cellulose and Carboxymethylcellulose Sodium is a colloid-forming, attrited mixture of Microcrystalline Cellulose and Carboxymethylcellulose Sodium. It contains NLT 75.0% and NMT 125.0% of the labeled amount of carboxymethylcellulose sodium, calculated on the dried basis. The viscosity of its aqueous dispersion of percent by weight stated on the label is NLT 60.0% and NMT 140.0% of that stated on the label in centipoises.

IDENTIFICATION

- **A.**
  Sample: 6 g
  Analysis: Mix the *Sample* with 300 mL of water in a blender at 18,000 rpm for 5 min.
  Acceptance criteria: A white, opaque dispersion is produced that does not settle on standing.
- **B.**
  Sample: The dispersion obtained in *Identification* test *A*
  Analysis: Add several drops of the *Sample* to a solution of aluminum chloride (100 mg/mL).
  Acceptance criteria: Each drop forms a white, opaque globule that does not disperse on standing.
- **C.**
  Sample: The dispersion obtained in *Identification* test *A*
  Analysis: Add 3 mL of iodine TS to the *Sample*.
  Acceptance criteria: No blue or purplish-blue color is produced.

ASSAY

- **CARBOXYMETHYLCELLULOSE SODIUM**
  Sample solution: Transfer 2000 mg of Microcrystalline Cellulose and Carboxymethylcellulose Sodium to a glass-stoppered, 250-mL conical flask. Add 75 mL of glacial acetic acid, attach a condenser, and reflux for 2 h. Cool, and transfer the mixture to a 250-mL beaker with the aid of small volumes of glacial acetic acid.
  Titrimetric conditions
  Mode: Direct titration
  Titrant: 0.1 N perchloric acid in dioxane VS
  Endpoint detection: Potentiometry
  Analysis: Titrate the *Sample solution* and calculate the percentage of carboxymethylcellulose sodium in the sample taken:

$$\text{Result} = [(V_S \times N \times F)/W] \times 100$$

V_S = *Titrant* volume consumed by the *Sample* (mL)
N = actual normality of the *Titrant* (mEq/mL)
F = equivalency factor, 296.0 mg/mEq
W = *Sample* weight (mg)

  Acceptance criteria: 75.0%–125.0% on the dried basis

IMPURITIES

- **RESIDUE ON IGNITION ⟨281⟩:** NMT 5.0%

Delete the following:

- **HEAVY METALS,** *Method II* ⟨**231**⟩**:** NMT 10 µg/g (Official 1-Jan-2018)

SPECIFIC TESTS

- **VISCOSITY—ROTATIONAL METHODS ⟨912⟩**
  Analysis: Determine the amounts of Microcrystalline Cellulose and Carboxymethylcellulose Sodium needed to prepare 600 g of a suitable dispersion, calculated on the dried basis. Transfer an amount of water to a 1000-mL blender bowl. Begin stirring with an 18,000 rpm blender at a reduced speed obtained by adjusting the voltage to 30 volts by means of a suitable transformer, and immediately add the accurately weighed portion of Microcrystalline Cellulose and Carboxymethylcellulose Sodium, taking care to avoid contacting the sides of the bowl with the powder. Continue stirring at this speed for 15 s following the addition of the powder, then increase the transformer setting to 115 volts, and mix for 2 min, accurately timed, at 18,000 rpm. Stop the blender, and lower the appropriate spindle of a suitable rotational viscometer into the dispersion. Thirty s after cessation of mixing, start the viscometer, and determine the viscosity using the appropriate spindle to obtain a scale reading between 10% and 90% of full-scale at a speed of 20 rpm. Determine the scale reading after 30 s of rotation, and calculate the viscosity, in centipoises, by multiplying the scale reading by the constant for the spindle used at 20 rpm.
  Acceptance criteria: 60.0%–140.0% of that stated on the label, in centipoises
- **pH ⟨791⟩**
  Sample solution: The dispersion prepared in the test for *Viscosity—Rotational Methods* ⟨912⟩
  Acceptance criteria: 6.0–8.0
- **LOSS ON DRYING ⟨731⟩**
  Analysis: Dry a sample at 105° to constant weight.
  Acceptance criteria: NMT 8.0%

ADDITIONAL REQUIREMENTS

- **PACKAGING AND STORAGE:** Preserve in tight containers, store in a dry place, and avoid exposure to excessive heat.
- **LABELING:** Label it to indicate the percentage content of carboxymethylcellulose sodium and the viscosity of the dispersion in water of the designated weight percentage composition.

Silicified Microcrystalline Cellulose

DEFINITION

Silicified Microcrystalline Cellulose is composed of intimately associated microcrystalline cellulose and colloidal silicon dioxide particles, derived from aqueous coprocessing prior to drying the material during manufacture. The microcrystalline cellulose component is purified, partially depolymerized cellulose, prepared by treating alpha cellulose, obtained as a pulp from fibrous plant material, with mineral acids. The colloidal silicon dioxide is a submicroscopic fumed silica prepared by the vapor-phase hydrolysis of a silicon compound. The *Residue on Ignition* result indicates the percentage of colloidal silicon dioxide; the remainder is microcrystalline cellulose.

IDENTIFICATION

- **A. INFRARED ABSORPTION ⟨197K⟩**
- **B.**
  Sample: 10 mg
  Iodinated zinc chloride solution: Dissolve 20 g of zinc chloride and 6.5 g of potassium iodide in 10.5 mL of water. Add 0.5 g of iodine, and shake for 15 min.
  Analysis: Place the *Sample* on a watch glass, and disperse in 2 mL of *Iodinated zinc chloride solution*.
  Acceptance criteria: The substance takes on a violet-blue color.
- **C.**
  Sample: 5 mg of residue from the test for *Residue on Ignition*
  Analysis: Transfer the *Sample* to a platinum crucible, and mix with about 200 mg of anhydrous potassium carbonate. Ignite at a red heat over a burner for about 10 min, and cool. Dissolve the melt in 2 mL of freshly distilled water, warming if necessary, and slowly add 2 mL of ammonium molybdate TS to the solution.