

acid, cover with a watch glass, and boil gently for 30 minutes. Remove the watch glass, and evaporate on a water bath to dryness with the aid of a current of air. Continue heating further for 1 hour, add 10 mL of hot water, stir well, and filter through a filter paper for quantitative analysis. Wash the residue with hot water, dry the residue together with the filter paper when no turbidity is produced on the addition of silver nitrate TS to the last washing, and ignite to constant mass: the amount of residue is not more than 0.5%.

(4) Heavy metals—Proceed with 1.0 g of Carmellose according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

(5) Arsenic—Take 1.0 g of Carmellose, prepare the test solution according to Method 3, and perform the test using Apparatus B (not more than 2 ppm).

Loss on drying Not more than 8.0% (1 g, 105°C, 4 hours).

Residue on ignition Not more than 1.5% (after drying, 1 g).

Containers and storage Containers—Tight containers.

Carmellose Calcium

Carboxymethylcellulose Calcium CMC Calcium

カルメロースカルシウム

Carmellose Calcium is the calcium salt of a polycarboxymethylether of cellulose.

Description Carmellose Calcium occurs as a white to yellowish white powder. It is odorless.

It is practically insoluble in ethanol (95) and in diethyl ether.

It swells with water to form a suspension.

The pH of a suspension, obtained by shaking 1 g of Carmellose Calcium with 100 mL of water, is between 4.5 and 6.0.

It is hygroscopic.

Identification (1) Shake thoroughly 0.1 g of Carmellose Calcium with 10 mL of water, followed by 2 mL of sodium hydroxide TS, allow to stand for 10 minutes, and use this solution as the sample solution. To 1 mL of the sample solution add water to make 5 mL. To 1 drop of this solution add 0.5 mL of concentrated disodium chlomotropate TS, and heat in a water bath for 10 minutes: a red-purple color develops.

(2) Shake 5 mL of the sample solution obtained in (1) with 10 mL of acetone: a white, flocculent precipitate is produced.

(3) Shake 5 mL of the sample solution obtained in (1) with 1 mL of iron (III) chloride TS: a brown, flocculent precipitate is produced.

(4) Ignite 1 g of Carmellose Calcium to ash, dissolve the residue in 10 mL of water and 5 mL of acetic acid (31), and filter, if necessary. Boil the filtrate, cool, and neutralize with ammonia TS: the solution responds to the Qualitative Tests (2), (3), and (4) for calcium salt.

Purity (1) Alkali—Shake thoroughly 1.0 g of Carmellose Calcium with 50 mL of water, freshly boiled and cooled, and add 2 drops of phenolphthalein TS: no red color develops.

(2) Chloride—Shake thoroughly 0.8 g of Carmellose Calcium with 50 mL of water, dissolve in 10 mL of sodium hydroxide TS, add water to make 100 mL, and use this solution as the sample solution. Heat 20 mL of the sample solution with 10 mL of dilute nitric acid on a water bath until a flocculent precipitate is produced. After cooling, centrifuge, and take out the supernatant liquid. Wash the precipitate with three 10-mL portions of water by centrifuging each time, combine the supernatant and the washings, and add water to make 100 mL. Take 25 mL of this solution, and add 6 mL of dilute nitric acid and water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution with 0.40 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.360%).

(3) Sulfate—Heat 10 mL of the sample solution obtained in (2) with 1 mL of hydrochloric acid in a water bath until a flocculent precipitate is produced. Cool, centrifuge, and take out the supernatant liquid. Wash the precipitate with three 10-mL portions of water by centrifuging each time, combine the supernatant and the washings, and add water to make 100 mL. Take 25 mL of this solution, and add 1 mL of dilute hydrochloric acid and water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution with 0.40 mL of 0.005 mol/L sulfuric acid VS (not more than 0.960%).

(4) Silicate—Weigh accurately about 1 g of Carmellose Calcium, ignite in a platinum dish, add 20 mL of dilute hydrochloric acid, cover with a watch glass, and boil gently for 30 minutes. Remove the watch glass, and evaporate on a water bath to dryness, with the aid of a current of air. Continue heating for further 1 hour, add 10 mL of hot water, stir well, and filter through filter paper for quantitative analysis. Wash the residue with hot water, dry the residue together with the filter paper when no turbidity is produced on the addition of silver nitrate TS to the last washing, and then ignite to constant mass: the residue is not more than 0.5%.

(5) Heavy metals—Proceed with 1.0 g of Carmellose Calcium according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

(6) Arsenic—Heat cautiously 2.0 g of Carmellose Calcium with 10 mL of sulfuric acid and 10 mL of nitric acid in a decomposition flask. Continue the heating with occasional addition of 2 mL of nitric acid until the liquid becomes colorless or light yellow. After cooling, add 15 mL of ammonium oxalate TS, heat until a white smoke evolves, cool, mix with 30 mL of water, and filter. Wash the residue with water, combine the washings with the filtrate, and add water to make 50 mL. Perform the test with 5 mL of this solution as the test solution using Apparatus B: the stain is not deeper than the following standard stain.

Standard stain: Proceed in the same manner without Carmellose Calcium, transfer 5 mL of the obtained solution to a generator bottle, add 2 mL of Standard Arsenic Solution, and proceed as directed for the test with the test solution (not more than 10 ppm).

(7) Starch—Heat 0.10 g of Carmellose Calcium with 10 mL of water, cool, and add 2 drops of iodine TS: no blue color develops.

Loss on drying Not more than 10.0% (1 g, 105°C, 4 hours).

Residue on ignition 10.0 – 20.0% (after drying 0.5 g).

Containers and storage Containers—Tight containers.

Carmellose Sodium

Carboxymethylcellulose Sodium

CMC Sodium

カルメロースナトリウム

Carmellose Sodium is the sodium salt of a polycarboxymethylether of cellulose. It, when dried, contains not less than 6.5% and not more than 8.5% of sodium (Na: 22.99).

Description Carmellose Sodium occurs as a white to yellowish white powder or granules. It has no taste.

It is practically insoluble in methanol, in ethanol (95), in acetic acid (100) and in diethyl ether.

It forms a viscid solution in water and in warm water.

It is hygroscopic.

Identification (1) Dissolve 0.2 g of Carmellose Sodium in 20 mL of warm water with stirring, cool, and use this solution as the sample solution. To 1 mL of the sample solution add water to make 5 mL. To 1 drop of this solution add 0.5 mL of concentrated disodium chlomotropate TS, and heat in a water bath for 10 minutes: a red-purple color develops.

(2) To 10 mL of the sample solution obtained in test (1) add 1 mL of copper (II) sulfate TS: a blue flocculent precipitate is produced.

(3) To 3 g of Carmellose Sodium add 20 mL of methanol and 2 mL of dilute hydrochloric acid, boil gently on a water bath for 5 minutes, and filter. Evaporate the filtrate to dryness, and add 20 mL of water to the residue: the solution responds to the Qualitative Tests for sodium salt.

pH Add 1.0 g of Carmellose Sodium in small portions to 100 mL of warm water with stirring, dissolve, and cool: the pH of this solution is between 6.0 and 8.0.

Purity (1) Clarity and color of solution—Firmly attach a glass plate of good quality 2 mm in thickness, to the bottom of a glass column 250 mm in height, 25 mm in inner diameter and 2 mm in thickness. This is used as an outer tube. Similarly prepare an inner tube by attaching a glass plate of good quality 2 mm in thickness to the bottom of a glass column 300 mm in height, 15 mm in inner diameter and 2 mm in thickness. Dissolve 1.0 g of Carmellose Sodium in 100 mL of water, pour this solution into the outer tube, and place on a piece of white paper on which 15 parallel black lines 1 mm in width and 1 mm in interval are drawn. Moving the inner tube up and down and observing from the upper part, determine the height of the solution up to the lower edge of the inner tube when the distinction of the lines becomes impossible. Repeat the operation 3 times, and calculate the mean value: it is larger than that calculated from the similar operation, using the following control solution.

Control solution: To 5.50 mL of 0.005 mol/L sulfuric acid VS add 1 mL of dilute hydrochloric acid, 5 mL of

ethanol (95) and water to make 50 mL. Add 2 mL of barium chloride TS, mix well, and allow to stand for 10 minutes. Shake well this solution before use.

(2) Chloride—Dissolve 0.5 g of Carmellose Sodium in 50 mL of water, and use this solution as the sample solution. Shake 10 mL of the sample solution with 10 mL of dilute nitric acid, heat to produce a flocculent precipitate in a water bath, cool, and centrifuge. Separate the supernatant liquid, wash the precipitate with three 10-mL portions of water, centrifuging each time, combine the supernatant liquid with the washings, and dilute with water to 200 mL. Perform the test using 50 mL of this solution as the test solution. Prepare the control solution with 0.45 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.640%).

(3) Sulfate—Add 1 mL of hydrochloric acid to 10 mL of the sample solution obtained in (2), shake well, heat to produce a flocculent precipitate in a water bath, cool, and centrifuge. Separate the supernatant liquid, wash the precipitate with three 10-mL portions of water, centrifuging each time, combine the washings with the supernatant liquid mentioned above, and dilute to 50 mL with water. Take 10 mL of this solution, dilute with water to 50 mL, and perform the test using this solution as the test solution. Prepare the control solution with 0.40 mL of 0.005 mol/L sulfuric acid VS (not more than 0.960%).

(4) Silicate—Weigh accurately about 1 g of Carmellose Sodium, ignite in a platinum dish, add 20 mL of dilute hydrochloric acid, cover with a watch glass, and boil gently for 30 minutes. Remove the watch glass, and evaporate on a water bath to dryness with the aid of a current of air. Continue heating for further 1 hour, add 10 mL of hot water, stir well, and filter through a filter paper for quantitative analysis. Wash the residue with hot water, dry together with the filter paper after no turbidity is produced on the addition of silver nitrate TS to the last washing, and then ignite to constant mass: the mass of the residue is not more than 0.5%.

(5) Heavy metals—Proceed with 1.0 g of Carmellose Sodium according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

(6) Arsenic—To 1.0 g of Carmellos Sodium add 20 mL of nitric acid, heat gently until it becomes fluid, cool, add 5 mL of sulfuric acid, and heat until white fumes are evolved. Add, if necessary, 5 mL of nitric acid after cooling, and heat again. Repeat this operation until the solution becomes colorless or slightly yellow. After cooling, add 15 mL of a saturated solution of ammonium oxalate monohydrate, and heat until white fumes are evolved again, cool, and dilute with water to 25 mL. Take 5 mL of this solution as the test solution, and perform the test using Apparatus B. The solution has no more color than the following standard stain.

Standard stain: Without using Carmellose Sodium, proceed in the same manner, then transfer 5 mL of this solution to a generator bottle, add exactly 2 mL of Standard Arsenic Solution, and proceed as directed for the test with the test solution (not more than 10 ppm).

(7) Starch—Add 2 drops of iodine TS to 10 mL of the sample solution obtained in (2): no blue color develops.

Loss on drying Not more than 10.0% (1 g, 105°C, 4 hours).

Assay Weigh accurately about 0.5 g of Carmellose Sodium, previously dried, add 80 mL of acetic acid (100), connect with a reflux condenser, and heat in an oil bath maintained at