DESCRIPTION

Calcium Lactobionate occurs as a white to cream-colored, free-flowing powder. It readily forms double salts, such as the chloride, bromide, and gluconate. It is anhydrous when obtained by spray-drying, or the dihydrate when obtained by crystallization. It is freely soluble in water, but insoluble in alcohol and in ether. It decomposes at about 120°. The pH of a 1 : 10 aqueous solution is between 6.5 and 7.5.

Function Firming agent in dry pudding mixes; nutrient

Packaging and Storage Store in well-closed containers.

IDENTIFICATION

- **CALCIUM,** Appendix IIIA
  Acceptance criterion: Passes tests.
- **INFRARED ABSORPTION,** *Spectrophotometric Identification Tests,* Appendix IIIC
  Reference standard: USP Calcium Lactobionate RS
  Sample and standard preparation: *K* (Sample previously dried at 105° for 8 h)
  Acceptance criterion: The spectrum of the sample exhibits maxima at the same wavelengths as those in the spectrum of the *Reference standard.*

IMPURITIES

Inorganic Impurities

- **HALIDES,** *Chloride and Sulfate Limit Tests, Chloride Limit Test,* Appendix IIIB
  Sample: 1.2 g
  Control: 0.7 mL of 0.020 N hydrochloric acid
  Acceptance criterion: The *Sample* shows no more turbidity than the *Control* (NMT 0.04%).
- **LEAD,** *Lead Limit Test, Flame Atomic Absorption Spectrophotometric Method,* Appendix IIIB
  Sample: 3 g
  Acceptance criterion: NMT 2 mg/kg
- **SULFATE**
  Sample: 25 g
  Analysis: Transfer the *Sample* to a 600-mL beaker, dissolve it in 200 mL of water, adjust the solution to a pH between 4.5 and 6.5 with 2.7 N hydrochloric acid, and filter, if necessary. Heat the filtrate or clear solution to just below the boiling point. Then, while stirring vigorously, add 10 mL of barium chloride TS, boil gently for 5 min, and allow the solution to stand for at least 2 h, or, preferably, overnight. Collect the precipitate of barium sulfate on a suitable, tared crucible, wash until free from chloride, dry, and ignite at 600° to constant weight. The weight of barium sulfate so obtained, multiplied by 0.412, represents the weight of sulfate (SO_4) in the sample taken.
  Acceptance criterion: NMT 0.7%

Organic Impurities

- **REDUCING SUBSTANCES, (AS DEXTROSE)**
  Sample: 1.0 g
  Analysis: Transfer the *Sample* to a 250-mL conical flask, dissolve it in 20 mL of water, and add 25 mL of alkaline cupric citrate TS. Cover the flask, boil the contents gently for 5 min, accurately timed, and cool the flask rapidly to room temperature. Add 25 mL of 0.6 N acetic acid, 10.0 mL of 0.1 N iodine, and 10 mL of 3 N hydrochloric acid. Titrate with 0.1 N sodium thiosulfate, adding 3 mL of starch TS as the endpoint is approached. Perform a blank determination (see *General Provisions*), make any necessary correction. Each mL of 0.1 N sodium thiosulfate consumed is equivalent to 2.7 mg of reducing substances (as dextrose).
  Acceptance criterion: NMT 1.0%

SPECIFIC TESTS

- **CALCIUM CONTENT**
  Sample: 1.5 g
  Analysis: Dissolve the *Sample* in 100 mL of water containing 2 mL of 2.7 N hydrochloric acid. While stirring, preferably with a magnetic stirrer, add about 30 mL of 0.05 M disodium EDTA from a 50-mL buret. Then, add 15 mL of 1 N sodium hydroxide and 300 mg of hydroxy naphthol blue indicator. Continue the titration with disodium EDTA to a blue endpoint. Each mL of 0.05 M disodium EDTA is equivalent to 2.004 mg of calcium (Ca).
  Acceptance criterion: NLT 5.05% and NMT 5.55%, calculated on the dried basis
- **LOSS ON DRYING,** Appendix IIC (105° for 8 h)
  Acceptance criterion: NMT 8.0%
- **OPTICAL (SPECIFIC) ROTATION,** Appendix IIB
  Sample solution: 50 mg/mL (on the anhydrous basis)
  Acceptance criterion: $[\alpha]_D^{25}$ Between +23° and +25°

OTHER REQUIREMENTS

- **LABELING** Indicate whether the product has been obtained through spray-drying or from crystallization.

Calcium Lignosulfonate

CAS: [8061-52-7]

DESCRIPTION

Calcium Lignosulfonate occurs as a brown, amorphous polymer. It is obtained from the spent sulfite and sulfate pulping liquor of wood or from the sulfate (Kraft) pulping process. It may contain up to 30% reducing sugars. It is soluble in water, but not in any of the common organic solvents. The pH of a 1 : 100 aqueous solution is between approximately 3 and 11.

Function Binder; dispersant

Packaging and Storage Store in well-closed containers.

IDENTIFICATION

- **A. CALCIUM,** Appendix IIIA
  Sample solution: 0.15 mg/mL
  Acceptance criterion: Passes tests.
- **B. PROCEDURE**
  Sample: 100 mg
  Analysis: Dissolve the *Sample* in 50 mL of water. Add 1 mL each of 10% acetic acid and 10% sodium nitrite

solution and mix by swirling. Allow the solution to stand for 15 min at room temperature.
Acceptance criterion: A brown color appears.

• **C. ULTRAVIOLET ABSORPTION**
Sample solution: 0.1 mg/mL (pH 5)
Acceptance criterion: A peak is observed between 275 and 280 nm.

ASSAY

• **SULFONATE SULFUR**
Sample: 1.0 g
Analysis: Dissolve the *Sample* in 400 mL of water in a beaker. Direct a gentle stream of nitrogen gas over the liquid's surface. Add 10 mL of nitric acid, and swirl the solution thoroughly until the reaction subsides. Add 10 mL of 70% perchloric acid, and swirl thoroughly again. [**CAUTION:** Handle perchloric acid in an appropriate fume hood.] Place the uncovered beaker on a hot plate, and heat the contents vigorously until the center of the bottom of the beaker becomes clear. Remove the beaker, and cool it to room temperature. Add 5 mL of hydrochloric acid, and heat it again until white fumes evolve. After cooling the beaker, dilute the solution to approximately 100 mL with water, adjust to pH 6 ± 0.2 with 10% sodium hydroxide, and heat the solution to boiling. Add 15 mL of 10% barium chloride solution, and leave the solution overnight in a fresh beaker in a steam bath at 90° to 95°. Filter through ashless filter paper (Whatman No. 42, or equivalent), and wash the precipitate with 200 mL of warm water. Transfer the paper and precipitate into a tared crucible. Heat the crucible slowly on a Bunsen burner to expel moisture. Place the crucible and contents in a muffle furnace at 850° for 1 h. Let the crucible cool in a desiccator, and then weigh the residue to the nearest 0.0001 g. Calculate the percent sulfonate sulfur by the formula:

$$(R/S) \times 13.7$$

R = Weight (g) of the residue
S = Weight (g) of the sample taken
Acceptance criterion: NLT 5.0% sulfonate sulfur

IMPURITIES

Inorganic Impurities

• **LEAD,** *Lead Limit Test, Atomic Absorption Spectrophotometric Graphite Furnace, Method I,* Appendix IIIB
Acceptance criterion: NMT 1 mg/kg

SPECIFIC TESTS

• **CALCIUM**
Strontium chloride solution: While stirring, add 164.7 g of 60% perchloric acid to 500 mL of water contained in a 1-L beaker. [**CAUTION:** Handle perchloric acid in an appropriate fume hood.] Then, while stirring, add 15.2 g of strontium chloride hexahydrate, stirring until solution is complete. Transfer the solution into a 1-L volumetric flask, and dilute to volume at room temperature with water. Mix thoroughly.
Standard solution: 0.7 mg/mL of calcium, prepared from a certified Calcium Standard Solution (NIST, or equivalent). [NOTE: Store the *Standard solution* in polyethylene bottles because of its instability in glass.]
Sample: 1 g, previously dried
Sample solution: Dilute the *Sample* to 10 mL and mix. If the solution is not particle-free, filter through a 0.45-µm disposable Millipore filter, discarding the first few mL of filtrate. Pipet 5 mL of *Strontium chloride solution* into a 50-mL volumetric flask and add 5.0 mL of the filtrate or clear solution. Dilute to volume with water, and mix well.
Analysis: Using a suitably calibrated atomic absorption spectrophotometer, determine the absorbance of the *Standard solution* and the *Sample solution* at 422.7 nm.
Acceptance criterion: The absorbance of the *Sample solution* is not greater than that of the *Standard solution.* (NMT 7.0%)

• **LOSS ON DRYING,** *Appendix IIC* (105° for 24 h)
Acceptance criterion: NMT 10.0%

• **REDUCING SUGARS**
Copper reagent solution: [NOTE: Solution must be prepared several days in advance of use.] Dissolve 28 g of anhydrous dibasic sodium phosphate and 40 g of potassium sodium tartrate tetrahydrate in 700 mL water. Add 100 mL of 1 N sodium hydroxide and 8 g of copper sulfate pentahydrate, followed by 180 g of anhydrous sodium sulfate. Add 0.7134 g of potassium iodate and dilute to 1 L. Allow to stand for several days, then filter the clear top part of the solution through a medium-porosity, sintered-glass funnel.
Lead subacetate solution: Dissolve 80 g of lead subacetate in 220 mL of water. Stir overnight, and filter through Whatman No. 42 filter paper, or equivalent. Dilute the supernatant solution to a specific gravity of 1.254 with freshly boiled water.
Dibasic sodium phosphate solution: 190 mg/mL dibasic sodium phosphate heptahydrate, made to 100 mL
Standard solution: 280 µg/mL dried dextrose, made to 500 mL
Sample solution: Dissolve 1 g of sample in 150 mL of water and adjust the pH to between 6.9 and 7.2 with sodium hydroxide solution or acetic acid.
Analysis: To the *Sample solution,* add *Lead subacetate solution* in increments until no further precipitation is observed. Bring the volume to 250.0 mL with water, and mix well. Centrifuge the mixture, pipet 10 mL of the supernatant into a 50-mL volumetric flask, and dilute to about 35 mL with water. Add 2 mL or more of *Dibasic sodium phosphate solution* until no further precipitation forms. Dilute to 50 mL with water, and mix. Centrifuge at 2100 × gravity for 10 min. Pipet 5 mL of supernatant solution into a test tube containing exactly 5 mL of *Copper reagent solution,* and mix. Loosely plug the tube, and place it in a boiling water bath for 40 min ± 10 s. At the end of the heating period, cool the tube immediately in cold water. Add 2 mL of 2.5% potassium iodide solution and 1.5 mL of 2 N sulfuric acid. Mix well, and titrate with 0.005 N sodium thiosulfate, using starch as the indicator, and note the volume of 0.005 N sodium thiosulfate consumed as V_S. Perform a corresponding blank titration using 5 mL of water and

5 mL of *Copper reagent solution* and record the volume of 0.005 N sodium thiosulfate consumed as V_B.

Repeat the entire procedure using 5 mL of *Standard solution* and 5 mL of *Copper reagent solution*, noting the volume of 0.005 N sodium thiosulfate consumed as V_D. Perform a corresponding blank titration using 5mL of water and 5mL of *Copper reagent solution;* record the volume of 0.005 N sodium thiosulfate consumed as V_B. Calculate the percent reducing sugars by the formula:

$$35(V_B - V_S)/(V_B - V_D)$$

$V_B - V_S$ = Volume (mL) of 0.005 N sodium thiosulfate consumed by the 5-mL aliquot of *Sample solution*

$V_B - V_D$ = Volume (mL) of 0.005 N sodium thiosulfate consumed by 5 mL of *Standard solution*

Acceptance criterion: NMT 30.0%

- **Residue on Ignition (Sulfated Ash),** Appendix IIC
  Sample: 1 g
  Acceptance criterion: NMT 20.0%
- **Viscosity of a 50% Solution**
  Sample: 200 g, on the dried basis
  Analysis: Dissolve the *Sample* in 200 mL of water contained in a 500-mL beaker. Equilibrate the solution at 25°, and measure its viscosity with a Brookfield viscometer A (model LVG, or equivalent), using a number 2 spindle at 20 rpm.
  Acceptance criterion: NMT 3000 centipoises

Calcium Oxide

Lime

CaO — Formula wt 56.08

INS: 529 — CAS: [1305-78-8]

DESCRIPTION

Calcium Oxide occurs as hard, white or gray-white masses or granules or as a white to gray-white powder. One g dissolves in about 840 mL of water at 25° and in about 1740 mL of boiling water. It is soluble in glycerin but insoluble in alcohol.

Function pH control agent; nutrient; dough conditioner; yeast food

Packaging and Storage Store in tight containers.

IDENTIFICATION

- **Calcium,** Appendix IIIA
  Sample solution: Slake 1 g of sample with 20 ml of water and add glacial acetic acid until the sample is dissolved.
  Acceptance criterion: Passes tests.

ASSAY

- **Procedure**
  Sample: 1 g of sample ignited to a constant weight (See *Loss on Ignition* below.)
  Analysis: Dissolve the *Sample* in 20 mL of 2.7 N hydrochloric acid. Cool the solution, dilute to 500.0 mL with water, and mix. Pipet 50.0 mL of this solution into a suitable container, and add 50 mL of water. While stirring, preferably with a magnetic stirrer, add about 30 mL of 0.05 M disodium EDTA from a 50-mL buret. Then, add 15 mL of 1 N sodium hydroxide and 300 mg of hydroxy naphthol blue indicator. Continue the titration with disodium EDTA to a blue endpoint. Each mL of 0.05 M disodium EDTA is equivalent to 2.804 mg of CaO.
  Acceptance criterion: NLT 95.0% and NMT 100.5% of CaO, on the ignited basis

IMPURITIES

Inorganic Impurities

- **Alkalies or Magnesium**
  Sample: 500 mg
  Analysis: Dissolve the *Sample* in 30 mL of water and 15 mL of 2.7 N hydrochloric acid. Heat the solution, boil for 1 min, and rapidly add 40 mL of oxalic acid TS, and stir vigorously. Add 2 drops of methyl red TS, and neutralize the solution with 6 N ammonium hydroxide to precipitate the calcium completely. Heat the mixture on a steam bath for 1 h and allow it to cool. Dilute the mixture to 100 mL with water, mix well, and filter. Add 0.5 mL of sulfuric acid to 50 mL of the filtrate. Then evaporate to dryness and ignite to constant weight in a tared platinum crucible at 800° ± 25°.
  Acceptance criterion: NMT 3.6%
- **Arsenic,** *Arsenic Limit Test,* Appendix IIIB
  Sample solution: 1 g in 15 mL of 2.7 N hydrochloric acid
  Acceptance criterion: NMT 3 mg/kg
- **Fluoride,** *Fluoride Limit Test,* Appendix IIIB
  Sample: 1.0 g
  Acceptance criterion: NMT 0.015% mg/kg
- **Lead,** *Lead Limit Test,* Appendix IIIB
  Sample solution: 1 g in 15 mL of 2.7 N hydrochloric acid
  Control: 5 µg Pb (5 mL of *Diluted Standard Lead Solution*)
  Acceptance criterion: NMT 2 mg/kg

SPECIFIC TESTS

- **Acid-Insoluble Substances**
  Sample solution: Slake 5 g of sample, and then mix it with 100 mL of water and sufficient hydrochloric acid, added dropwise, to dissolve it.
  Analysis: Boil the *Sample solution*, cool, add hydrochloric acid, if necessary, to make the solution distinctly acid, and filter through a tared glass filter crucible. Wash the residue with water until free of chlorides, dry at 105° for 1 h, cool, and weigh.
  Acceptance criterion: NMT 1%
- **Loss on Ignition**
  Sample: 1 g
  Analysis: Ignite the *Sample* to constant weight in a tared platinum crucible at 1100° ± 50°.
  Acceptance criterion: NMT 10.0%