Status: Currently Official on 15-Feb-2025
Official Date: Official as of 01-Jun-2023
Document Type: NF Monographs
DocId: GUID-EA75622C-1E60-44ED-BE46-AA693622CEFC_5_en-US
DOI: https://doi.org/10.31003/USPNF_M34617_05_01
DOI Ref: jk2zw

Galageenan

DEFINITION

Galageenan is the hydrocolloid obtained by extraction with water or aqueous alkali from the red seaweed class Rhodophyceae species *Eucheuma gelatinae*. Galageenan consists chiefly of potassium, sodium, calcium, magnesium, and ammonium sulfate esters of galactose and 3,6-anhydrogalactose copolymers. These hexoses are alternately linked as α-1,3 and β-1,4 in the polymer. The ester sulfate content ranges from 8%–18%. In addition, it contains inorganic salts that originate from the seaweed and from the process of recovery from the extract.

Galageenan is recovered by alcohol precipitation or by freezing and pressing.

IDENTIFICATION

• **A. Film Formation**

Solution A: A solution (1 in 50), prepared by heating a uniform dispersion in a hot water bath to 80°

Acceptance criteria: *Solution A* becomes more viscous upon cooling and may form a gel.

• **B.**

Analysis: Dilute a portion of *Solution A* (retained from *Identification* test *A*) with 4 parts water, and add 2–3 drops of methylene blue TS.

Acceptance criteria: A blue, stringy precipitate is formed.

• **C. Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*: 197F**

Sample solution: Disperse 2 g of Galageenan in 400 mL of a solution containing 5 g of edetate disodium in 1000 mL of 60% isopropyl alcohol, and stir for 2 h. Filter with the aid of vacuum, and wash the residue with a total of 200 mL of 65% isopropyl alcohol. Finish washing with a total of 100 mL of 80% isopropyl alcohol. Dry the residue for 30 min in a 60° oven, and overnight in a 70° vacuum oven. Break lumps by grinding with a mortar and pestle. Dissolve 15 mg of the alcohol-treated material in 5 mL of water. Heat for 10 min in a water bath. Pipet 2 mL onto a suitable nonsticking surface to produce a 5-µm-thick film (when dry).

Spectral range: 2000–600 cm^{-1}

Analysis: Subtract the baseline (drawn by connecting the minima in the range of 1500 and 800 cm^{-1}) from the raw spectrum. Record the absorbances for the bands at 1220–1260, 928–933, 840–850, and 800–805 cm^{-1} relative to the absorbance at 1050 cm^{-1}.

Acceptance criteria: The absorbance values so obtained are within the ranges specified in *Table 1*.

Table 1

Wave Number (cm^{-1})	Molecular Assignment	Absorbance Relative to 1050 cm^{-1} Galageenan
1220–1260	Ester sulfate	0.3–0.6
928–933	3,6-Anhydrogalactose	0.3–0.6
840–850	Galactose-4-sulfate	0.1–0.3
800–805	3,6-Anhydrogalactose-2-sulfate	0.0–0.1

ASSAY

• **Content of Sulfate**

Sample: 300 mg

Analysis: Weigh the *Sample* on an ashless filter paper. Fold the paper so as to enclose the *Sample*, and place it in a 500-mL Kjeldahl flask. Add 45 mL of nitric acid, and bring to a boil on a hot plate in a fume hood. Add nitric acid as necessary to keep the sample from evaporating to dryness. Continue boiling until digestion is complete and the volume of nitric acid remaining is about 10 mL. Cool the mixture, and reduce the excess nitric acid by adding formaldehyde TS until the evolution of nitrogen oxide vapors has ceased. Heat this mixture on a hot plate to reduce the volume to about 10 mL. Transfer the mixture to a 150-mL beaker with the aid of several portions of water until the total volume is approximately 100 mL. Add 0.5 mL of hydrochloric acid, and bring to a boil on a hot plate. Add carefully 10 mL of 0.25 M barium chloride, and allow to boil for 1 min. Cover with a watch glass, and allow to stand overnight. Filter the solution through a tared, fine-porosity filtering crucible previously ignited in a muffle furnace at 550° for 30 min and cooled in a desiccator for 30 min. Wash the barium sulfate precipitate so obtained on the crucible several times with boiling water. Place the crucible in an oven, and heat at 100° for 30 min. Transfer the crucible to a muffle furnace, and ignite for 30 min at 550°. Remove the crucible, and cool in a desiccator for 30 min.
Weigh and calculate the percentage of sulfate groups:

$$\text{Result} = (W_B/W_S) \times (M_{r1}/M_{r2}) \times 100$$

W_B = weight of barium sulfate obtained (mg)

W_S = weight of Galageenan taken (mg)

M_{r1} = molecular weight of the sulfate group, 96.02

M_{r2} = molecular weight of barium sulfate, 233.43

Acceptance criteria: 8%–18% sulfate

IMPURITIES

Change to read:

• ▲**Lead** ⟨251⟩, *Procedures, Procedure 1*▲ (CN 1-Jun-2023) **:** NMT 5 ppm

• **Acid-Insoluble Matter**

Sample: 2 g

Analysis: Transfer the *Sample* to a 250-mL beaker containing 150 mL of water and 1.5 mL of sulfuric acid. Cover with a watch glass, and heat on a steam bath for 6 h, rubbing down the wall of the beaker frequently with a rubber-tipped stirring rod, and replacing any water lost by evaporation. Transfer 500 mg of a suitable filter aid to the beaker, and pass through a tared filtering crucible equipped with a 2.4-cm glass fiber filter. Wash the residue several times with hot water, dry at 105° for 3 h, cool in a desiccator, and weigh. The difference between the total weight and the sum of the weights of the filter aid, crucible, and glass fiber filter is the weight of the acid-insoluble matter.

Acceptance criteria: NMT 2.0% of the weight of Galageenan taken

SPECIFIC TESTS

• **Microbial Enumeration Tests** ⟨61⟩and **Tests for Specified Microorganisms** ⟨62⟩**:** The total aerobic microbial count does not exceed 200 cfu/g, the total combined molds and yeasts count does not exceed 20 cfu/g, and it meets the requirements of the tests for absence of *Escherichia coli* and *Salmonella* species.

• **Viscosity—Rotational Methods** ⟨912⟩

Sample solution: Transfer 7.5 g of Galageenan to a tared, tall-form, 600-mL beaker. Add 450 mL of water, and disperse with agitation for 15 min. Add water to bring the weight to 500 g, and heat in a water bath, with continuous agitation, until a temperature of 80° is reached. Add water to adjust for loss by evaporation, cool to between 76° and 77°, and place in a constant-temperature bath maintained at 75°.

Analysis: Use a suitable rotational viscometer equipped with a spindle having a cylinder 1.88 cm in diameter and 6.51 cm in height, and an immersion depth of 8.10 cm (No. 1 spindle). Allow the spindle to rotate in the solution at 30 rpm for six revolutions, then observe the scale reading. Convert the scale reading to centipoises by multiplying by the constant for the spindle and speed used.

Acceptance criteria: NLT 15 centipoises (at 75°)

• **Articles of Botanical Origin,** *Total Ash*⟨561⟩**:** NMT 35.0%

• **Loss on Drying** ⟨731⟩**:** Dry a sample at a pressure not exceeding 10 mm of mercury at 70° for 18 h, cool in a desiccator, and weigh: it loses NMT 12.5% of its weight.

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight containers, preferably in a cool place.

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
GALAGEENAN	Documentary Standards Support	CE2020 Complex Excipients

Chromatographic Database Information: Chromatographic Database

Most Recently Appeared In:
Pharmacopeial Forum: Volume No. Information currently unavailable

Current DocID: GUID-EA75622C-1E60-44ED-BE46-AA693622CEFC_5_en-US
DOI: https://doi.org/10.31003/USPNF_M34617_05_01
DOI ref: jk2zw