

GMIA STANDARD METHODS FOR THE TESTING OF EDIBLE GELATIN



Preface

The Standard Methods for the Sampling and Testing of Edible Gelatin contained in this booklet are the results obtained by a co-operative testing program conducted by the Technical Staffs of the entire membership of the Gelatin Manufacturers Institute. These methods have been found to give accurate and consistent results.

Gelatin Manufacturers Institute of America Member Companies

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1. General Information

1.1 Definition

Gelatin is the product obtained from the acid, alkaline, or enzymatic treatment of collagen, the chief protein component of the skins, bones, and connective tissues of animals. These animal sources shall have not been exposed to pentachlorophenol.

Type A gelatin is produced by the acid processing of collagenous raw materials and exhibits an isoelectric point between pH = 7 and pH = 9. Type B gelatin is produced by the alkaline or lime processing of collagenous raw materials and exhibits an isoelectric point between pH = 4.6 and pH = 5.2. Mixtures of Types A and B as well as gelatins produced by modifications of the above mentioned processes might exhibit isoelectric points outside of the stated ranges. (Food Chemicals Codex).

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1.2 Description

Gelatin is nearly tasteless and odorless. It is a vitreous, brittle solid that is faintly yellow to light tan. It is supplied in various physical forms such as coarse granules, fine powders and leaves.

1.3 Stability

Gelatin is very stable when stored in its original container in ambient humidity with controlled temperature. The shelf life of gelatin is generally recognized as stable for at least 5 years when stored under these conditions.

1.4 Functionality in Foods

Gelatin is used in confectionery, water jellies and desserts, dairy products or functional food, for its versatility. Its functionalities include firming agent, formulation and processing aid, stabilizer and thickener, surface-active agent, and water finishing agent.

1.5 Characteristics

When gelatin granules are immersed in cold water, they hydrate into discrete, swollen particles. On being warmed, gelatin disperses into the water, resulting in a stable suspension. Water solutions of gelatin will form a reversible gel if cooled below the specific gel point of gelatin. The gel point is dependent on the source of the raw material. Gelatin extracted from the tissues of warm-blooded animals will have a gel point in the range of 30°C to 35°C. Gelatin extracted from the skin of cold-water ocean fish will have a gel point in the range of 5°C to 10°C. Gelatin is stable in aqueous solutions of polyhydric alcohols such as glycerine and propylene glycol. It is insoluble in most organic solvents. (Food Chemicals Codex).

1.6 Identification

PRINCIPLE

Gelatin can be identified by a visual, physical state change; formation of a precipitate or turbid solution; and determination of hydroxyproline content.

REAGENTS AND SOLUTIONS

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1. Dry, granular gelatin
2. Deionized water
3. Trinitrophenol TS
4. Potassium dichromate solution
5. 3N hydrochloric acid
6. Tannic acid TS

APPARATUS

1. 100 mL volumetric flask
2. Water bath
3. Refrigerator

PROCEDURE

1. Gelatin forms a reversible gel.
 - A. Dissolve 5 g of gelatin in 100 mL hot water. When all gelatins are dissolved, place the solution in a refrigerator (2-10°C) for 4 hours. Gelatin gels.
 - B. Remove the gelled solution and place the container in 60°C. Within 30 minutes, when stirred, the gel reverts to the original liquid state.
2. To a 1:100 aqueous solution, add trinitrophenol TS or a solution of potassium dichromate (1:15) previously mixed with ¼ its volume of 3N hydrochloric acid. A yellow precipitate forms.
3. To a 1:5000 aqueous solution, add tannic acid TS; turbidity is produced.
4. For conclusive evidence of identity, test for hydroxyproline

1.7 Hydroxyproline Content

PRINCIPLE

Gelatin contains a high amount of the amino acid hydroxyproline. Hydroxyproline is liberated through acid hydrolysis, oxidized, and then identified with Erlich's reagent (5% *p*-dimethylaminobenzaldehyde in *n*- propanol).

REAGENTS AND SOLUTIONS

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1. 0.05N CuSO₄
2. 2.5N NaOH
3. 6% H₂O₂
4. 3N H₂SO₄
5. Erlich's Reagent – 5% *p*-Dimethylaminobenzaldehyde in *n*-Propanol (make fresh each time).

APPARATUS

1. Oil bath, capable of 145°C.
2. Water bath, 40°C
3. Ice bath
4. Bunsen burner
5. 18 x 150 mm test tubes
6. 500 mL volumetric flask
7. Aluminum foil
8. Distilled/deionized water
9. Concentrated HCl

PROCEDURE

1. Dissolve 1.0 gram of material in 200 mL of water.
2. Add 3 mL of this solution and 3mL of conc. HCl to an 18 x 150 mm test tube. Seal by melting the top in a Bunsen burner. Hydrolyze at 145°C for 1.5 hours in an agitated oil bath.
3. Cool the hydrolysate, cut off the top of the tube, transfer contents to the volumetric flask and dilute to 500 mL.
4. Transfer 1 mL to an 18 x 150 mm test tube. Add 1 mL 0.05N CuSO₄, 1 mL 2.5N NaOH, and place in a 40°C water bath for 5 minutes.
5. Add 1 mL 6% H₂O₂ and mix immediately.
6. Keep the sample at 40°C for 10 minutes, **Shake** and rotate to remove all excess H₂O₂.
7. Cool rapidly in an ice bath. Add 4 mL 3N H₂SO₄, mix, then add 2 mL Ehrlich's reagent, mix, cover with aluminum foil and hold at room temperature for 15 minutes. The development of an intense red color shows the presence of Hydroxyproline.

QUALITY CONTROL

All chemicals are reagent grade.

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1.8 Sampling

PRINCIPLE

Aseptic gelatin samples are required for all testing. The square root method plus one, should be followed to determine the number of samples per lot.

REAGENTS AND SOLUTIONS

None

APPARATUS

1. Sterile gloves
2. Scoop
3. Air - tight container
4. U. S. standard sieve (8 mesh)
5. Table top blender

PROCEDURE

1. Take aseptic sample by scooping out a cone several inches below surface of the gelatin in the container.
2. Pull the scoop up, across the vertical surface of the cone to obtain a representative sample.
3. Place samples as drawn in clean airtight containers.
4. Proportion the amount taken from each container selected to at least 120g of sample per container.
5. Blend or mix samples thoroughly.
6. Withdraw and retain at least 500g as the final sample.

NOTE: Gelatin coarser than 8 mesh U.S. standard sieve should be ground so that all particles pass the 8-mesh sieve. The entire final sample should be re-blended.