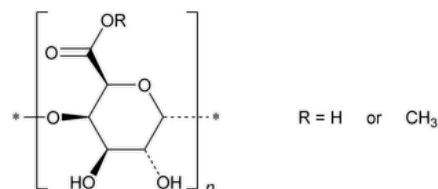


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Pectin



Pectin

CAS RN®: 9000-69-5.

DEFINITION

Pectin is a purified carbohydrate polymer consisting mainly of a linear backbone of partially methoxylated alpha (1-4) linked D-galacturonic acid. It is obtained from the dilute acid extract of the rind of citrus fruits or from apple pomace. No organic solvents other than methanol, ethanol, and isopropanol are used during its production. Pectin yields NLT 74.0% of galacturonic acid ($C_6H_{10}O_7$), calculated on the dried basis.

[NOTE—Commercial pectin for the production of jellied food products is standardized to the convenient “150 jelly grade” by addition of dextrose or other sugars, and sometimes contains sodium citrate or other buffer salts. This monograph refers to the pectin to which no such additions have been made.]

IDENTIFICATION

• PROCEDURE

Sample stock solution: Transfer a quantity of Pectin, equivalent to 0.05 g on the dried basis, to a suitable container, and moisten with 250 μ L of 2-propanol. Add 50 mL of water to the container, and mix the solution using a magnetic stirrer. Use 0.5 N sodium hydroxide to adjust the pH of the solution to 12, stop the stirrer, and allow the solution to stand undisturbed at room temperature for 15 min. Adjust with 0.5 N hydrochloric acid to a pH of 7.0, and dilute with water to 100 mL.

Tris buffer solution: Transfer 6.055 g of tris(hydroxymethyl)aminomethane and 0.147 g of calcium chloride ($CaCl_2 \cdot 2H_2O$) to a 1000-mL volumetric flask containing 950 mL of water. Adjust with 1 N hydrochloric acid to a pH of 7.0, and dilute with water to volume.

Enzyme solution: Mix pectate lyase¹ with *Tris buffer solution* to make a solution (1 in 100).

Sample blank: Mix 0.5 mL of *Tris buffer solution*, 1.0 mL of *Sample stock solution*, and 1.0 mL of water in a quartz cuvette.

Enzyme blank: Mix 0.5 mL of *Tris buffer solution*, 1.5 mL of water, and 0.5 mL of *Enzyme solution* in a quartz cuvette.

Sample solution: Mix 0.5 mL of *Tris buffer solution*, 1.0 mL of *Sample stock solution*, 0.5 mL of water, and 0.5 mL of *Enzyme solution* in a quartz cuvette.

Analysis

Samples: *Sample blank*, *Enzyme blank*, and *Sample solution*

Perform the test with the *Samples* using a suitable UV/visible spectrophotometer (see [Ultraviolet-Visible Spectroscopy \(857\)](#)) and using water as a blank. Measure the absorbance at 235 nm immediately after mixing the solutions well, and record the value at time 0 for the *Enzyme blank*, A_{0-EB} ; for the *Sample blank*, A_{0-TB} ; and for the *Sample solution*, A_{0-TS} . After incubation at room temperature for 10 min, determine the absorbance again at 235 nm for the *Enzyme blank*, A_{10-EB} ; for the *Sample blank*, A_{10-TB} ; and for the *Sample solution*, A_{10-TS} . Calculate the corrected absorbance A_0 at time 0 and the corrected absorbance A_{10} at 10 min:

$$A_0 = A_{0-TS} - (A_{0-EB} + A_{0-TB})$$

$$A_{10} = A_{10-TS} - (A_{10-EB} + A_{10-TB})$$

Calculate the quantity of unsaturated product produced:

$$\text{Result} = (A_{10} - A_0)/(\epsilon_{235} \times L)$$

ϵ_{235} = molar extinction coefficient of the reaction product ($4600 \text{ M}^{-1} \cdot \text{cm}^{-1}$)

L = path length of the reaction cuvette, 1 cm

Acceptance criteria: The amount of unsaturated product is NLT $0.5 \times 10^{-5} \text{ M}$.

ASSAY

• DEGREE OF ESTERIFICATION

Sample: 5.0 g

Analysis: Transfer the *Sample* to a suitable beaker, and stir for 10 min with a mixture of 5 mL of hydrochloric acid and 100 mL of 60% alcohol.

Transfer to a sintered-glass filter (30- to 60-mL crucible or Büchner type, coarse), and wash with six 15-mL portions of the hydrochloric acid–60% alcohol mixture, followed by 60% alcohol until the filtrate is free from chlorides. Finally wash with 20 mL of alcohol, dry for 1 h at 105° , cool, and weigh. Transfer exactly one-tenth of the total net weight of the dried *Sample* (representing 500 mg of the original unwashed *Sample*) to a 250-mL conical flask, and moisten with 2 mL of alcohol. Add 100 mL of carbon dioxide-free water, insert the stopper, and swirl occasionally until the Pectin is completely dissolved. Add 5 drops of phenolphthalein TS, and titrate with 0.1 N sodium hydroxide VS.

Perform a blank determination, and make any necessary correction. Record the results as the initial titer, V_i (mL). Add 20.0 mL of 0.5 N sodium hydroxide VS, insert the stopper, shake vigorously, and allow to stand for 15 min. Add 20.0 mL of 0.5 N hydrochloric acid VS, and shake until the pink color disappears. Add phenolphthalein TS, and titrate with 0.1 N sodium hydroxide VS to a faint pink color that persists after vigorous shaking. Perform a blank determination, and make any necessary correction. Record this value as the saponification titer, V_s (mL). Calculate the degree of esterification:

$$\text{Result} = [V_s / (V_i + V_s)] \times 100$$

V_s = saponification titer (mL)

V_i = initial titer (mL)

Acceptance criteria: The value for *Degree of Esterification* is within the range stated on the label.

• **GALACTURONIC ACID:** Each mL of 0.1 N sodium hydroxide used in the total titration (the initial titer added to the saponification titer) in the Assay for *Degree of Esterification* is equivalent to 19.41 mg of $\text{C}_6\text{H}_{10}\text{O}_7$. Calculate the percentage of galacturonic acid in the portion of Pectin taken:

$$\text{Result} = 19.41 \times [(V_i + V_s) / W] \times 100$$

V_i = initial titer (mL)

V_s = saponification titer (mL)

W = weight of the original unwashed and dried Pectin taken to prepare the solution for titration (mg)

Acceptance criteria: NLT 74.0%

• **METHOXY GROUPS:** Each mL of 0.1 N sodium hydroxide used in the saponification titer in the Assay for *Degree of Esterification* is equivalent to 3.10 mg of $-\text{OCH}_3$. Calculate the percentage of methoxy groups in the portion of Pectin taken:

$$\text{Result} = 3.10 \times (V_s / W) \times 100$$

V_s = saponification titer (mL)

W = weight of the original unwashed and dried Pectin taken to prepare the solution for titration (mg)

Acceptance criteria: The percentage of methoxy groups is within the range stated on the label.

IMPURITIES

Change to read:

• **ARSENIC** (211), *Procedures, Procedure 2* (CN 1-JUN-2023): NMT 3 ppm

• **LEAD**

Standard stock solution: 1000 µg/mL of lead. [NOTE—Use a commercially available certified solution.]

Standard solution: 2 µg/mL of lead, prepared immediately before use by pipetting 0.10 mL of *Standard stock solution* into a 50-mL volumetric flask containing 30 mL of water, 4 mL of 20% hydrochloric acid, and 4 mL of 0.1 M EDTA. Dilute with water to volume, and mix.