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Cellaburate

Cellulose, acetate butanoate;
Cellulose, acetate butyrate;
Acetylbutyrylcellulose;
Cellulose butyrate acetate;
Cellulose acetate butyrate
CAS RN®: 9004-36-8.

DEFINITION

Cellaburate is a reaction product of cellulose, acetic anhydride or acetic acid, and butyric acid or butyric anhydride. It contains NLT 1.0% and NMT 41.0% of acetyl (C_2H_3O) groups, by weight, and NLT 5.0% and NMT 56.0% of butyryl (C_4H_7O) groups, by weight, calculated on the previously dried, acid-free basis.

IDENTIFICATION

Change to read:

• **A.** ▲Spectroscopic Identification Tests ⟨197⟩, *Infrared Spectroscopy*: **197F**▲ (CN 1-May-2020)

Sample solution: Dissolve 150 mg in 1 mL of acetone. Evenly cast 1 drop of the solution on a sodium chloride plate. Heat the plate at 105° for 10 min.

Acceptance criteria: Meets the requirements

ASSAY

• **Acetyl and Butyryl Content**

Internal standard solution: 4.6 mg/mL of isovaleric acid in pyridine. Store it in a tightly closed container.

Saponification solution: Place 250 mL of *n*-propyl alcohol in a 500-mL volumetric flask, add 65.5 g of potassium hydroxide, and mix to dissolve. Dilute with *n*-propyl alcohol to volume, and mix.

Acid solution: Place 250 mL of *n*-propyl alcohol in a 500-mL volumetric flask, add 166 mL of hydrochloric acid, and mix. Dilute with *n*-propyl alcohol to volume, and mix.

Standard solution: Transfer 0.20 g of glacial acetic acid and 0.31 g of butyric acid to a 50-mL volumetric flask. Dilute with *Internal standard solution* to volume, and mix.

Sample solution: Transfer 0.15 g of Cellaburate, previously dried at 105° for 1 h, into a 25-mm × 160-mm test tube. Pipet 10 mL of *Internal standard solution* into the test tube, and dissolve by stirring and heating at 110° for 30 min. While stirring, add 5 mL of *Saponification solution* slowly into the tube. Heat at 110° for 10 min. Cool, and add 5 mL of the *Acid solution.* Mix on a vortex mixer, and allow the precipitate to settle.

Chromatographic system

(See *Chromatography ⟨621⟩, System Suitability*.)

Mode: GC

Detector: Flame ionization

Column: 0.53-mm × 30-m fused silica bonded with a 1-µm layer of phase G35

Temperatures

Column: 125°

Injector: 250°

Detector: 250°

Carrier gas: Helium

Flow rate: 8 mL/min

Injection volume: 1 µL

Injection type: Split ratio, 35:1

System suitability

Sample: *Standard solution*

[Note—The relative retention times for acetic acid, butyric acid, and isovaleric acid are about 0.45, 0.85, and 1.00, respectively.]

Suitability requirements

Tailing factor: NMT 1.5 for the butyric acid peak

Relative standard deviation: NMT 3.0%

Calibration

Sample: *Standard solution*

Number of injections: 3

Calculate the average unit weight response, F_{SA}, of acetic acid per 10 mL of the *Internal standard solution*:

$$F_{SA} = (W_{RA}/R_{SA}) \times F_1$$

W_{RA} = weight of acetic acid in the *Standard solution* (g)

R_{SA} = average peak response ratio of acetic acid to isovaleric acid

F_1 = volume ratio of the *Internal standard solution* in the *Sample solution* to that in the *Standard solution*, 10:50

Similarly, calculate the average unit weight response, F_{SB}, of butyric acid per 10 mL of the *Internal standard solution*:

$$F_{SB} = (W_{RB}/R_{SB}) \times F_1$$

W_{RB} = weight of butyric acid in the *Standard solution* (g)

R_{SB} = average peak response ratio of butyric acid to isovaleric acid

Analysis

Sample: Upper clear solution from the *Sample solution*

Calculate the percentage of acetyl in the portion of Cellaburate taken:

$$\text{Result} = [(M_{r1}/M_{r2}) \times R_{UA} \times F_{SA}]/W_U \times 100$$

M_{r1} = formula weight of acetyl, 43

M_{r2} = formula weight of acetic acid, 60

R_{UA} = peak response ratio of acetic acid to isovaleric acid in the *Sample solution*

W_U = weight of Cellaburate taken to prepare the *Sample solution* (g)

Calculate the percentage of butyryl in the portion of Cellaburate taken:

$$\text{Result} = [(M_{r3}/M_{r4}) \times R_{UB} \times F_{SB}]/W_U \times 100$$

M_{r3} = formula weight of butyryl, 71

M_{r4} = formula weight of butyric acid, 88

R_{UB} = peak response ratio of butyric acid to isovaleric acid in the *Sample solution*

Acceptance criteria: 1.0%–41.0% of acetyl (C_2H_3O) groups and 5.0%–56.0% of butyryl (C_4H_7O) groups on the previously dried, acid-free basis

IMPURITIES

- **Residue on Ignition ⟨281⟩:** NMT 0.1%
- **Limit of Free Acid**

Indicator solution: In a 1-L volumetric flask dissolve 0.675 g of bromocresol purple in 25 mL of 0.10 N sodium hydroxide. Dilute with water to volume, and mix.

Calibration solutions: Pipet 1, 2, 3, and 4 mL of 0.001 N acetic acid VS into four 100-mL volumetric flasks, respectively. Pipet 4 mL of the *Indicator solution* into each flask and into an empty 100-mL volumetric flask, and dilute each flask with water to volume to obtain solutions containing 0.0, 0.60, 1.20, 1.80, and 2.40 µg/mL of acetic acid.

Control solution: Place 96 mL of water in a suitable bottle, add a stirring bar, cap the bottle, and stir for 75 min at room temperature. Pipet 4 mL of the *Indicator solution* into the bottle, and mix.

Sample solution: Transfer 1–2 g of Cellaburate to a bottle, and add 96 mL of water. Add a stirring bar, cap the bottle, and stir for 75 min at room temperature. Pipet 4 mL of the *Indicator solution* into the bottle, stir to mix, and allow the solid to settle for 2 min.

Instrumental conditions

(See *Ultraviolet-Visible Spectroscopy ⟨857⟩*.)

Mode: Vis

Analytical wavelength: Determine the maximum absorption of the basic form of bromocresol purple in the *Calibration* at about 589 nm.

Cell: 1 cm

Blank: Water

Calibration: Determine the absorbances of the *Calibration solutions*. The absorbance difference between the 0.0-µg/mL solution and the other solutions adheres to Beer's law over the range stated in the *Calibration solutions*. Plot the absorbance difference versus the concentration of the acetic acid, in µg/mL, on linear graph paper, and draw the straight line best fitting the points, including the origin.

Analysis

Samples: *Control solution* and *Sample solution*

Pass 10 mL of the *Sample solution* through a polytef syringe filter that has been presoaked in isopropyl alcohol. Determine the absorbance of the filtered *Sample solution*. In the same manner, determine the absorbance of the *Control solution*.

Calculate the percentage of free acid, as acetic acid, in the portion of Cellaburate taken:

$$\text{Result} = (V \times C_U/W_U)/F \times 100$$

V = total volume of the *Sample solution*, 100 mL

C_U = concentration of free acid, calculated as acetic acid (µg/mL), based on the absorbance difference between the *Control solution* and the *Sample solution* read directly from the calibration plot

W_U = weight of Cellaburate taken to prepare the *Sample solution* (g)

F = unit conversion, 10^6 µg/g

[NOTE—If the C_U value is more than 2.8 µg/mL, reduce the sample size by half in the *Sample solution*, and repeat the determination.]

Acceptance criteria: NMT 0.1%

SPECIFIC TESTS

• **WATER DETERMINATION, *Method I* ⟨921⟩:** NMT 5.0%, using a mixture of methylene chloride and methanol (2:1) in place of the methanol solvent

ADDITIONAL REQUIREMENTS

• **PACKAGING AND STORAGE:** Preserve in tight containers. No storage requirements specified.

• **LABELING:** The labeling indicates the nominal percentage ranges of the acetyl and butyryl groups.

• **USP REFERENCE STANDARDS ⟨11⟩**
USP Cellaburate RS

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

Topic/Question	Contact	Expert Committee
CELLABURATE	Documentary Standards Support Associate Scientific Liaison.	NBDS2020 Non-botanical Dietary Supplements

Chromatographic Database Information: Chromatographic Database

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