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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

GB 5009.150-2016

**National Food Safety Standard -
Determination of Monascus Pigments in Foods**

食品安全国家标准

食品中红曲色素的测定

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State Food and Drug Administration.**

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National Food Safety Standard -

Determination of Monascus Pigments in Foods

1 Scope

This Standard specifies the method for determination of monascorubrin, monacolin, and monascorubramine in foods.

This Standard applies to the determination of three monascus pigments in flavor fermented milk, jam, fermented bean curd, dried almonds, candy, instant noodle products, cakes, biscuits, cooked meat products, soy sauce, fruit and vegetable juice drinks, solid beverages, mixed liquor, jelly and potato chips.

2 Principle

The sample which is extracted by absolute ethanol or 80% ethanol and purified by solid phase extraction column, shall be detected by liquid chromatography, characterized by retention time and quantified by external standard method.

3 Reagents and materials

Unless otherwise specified, all the reagents in this method are analytical reagents, the water is grade-1 water specified by GB/T 6682.

3.1 Reagents

3.1.1 Methanol (CH_3OH): chromatographic pure.

3.1.2 Absolute ethanol ($\text{CH}_3\text{CH}_2\text{OH}$): chromatographic pure.

3.1.3 Glacial acetic acid (CH_3COOH).

3.1.4 Ammonia acetate ($\text{CH}_3\text{COONH}_4$).

3.2 Preparation of reagents

3.2.1 Acetic acid-ammonium acetate solution (pH=5): accurately weigh 0.77 g of ammonium acetate in a 1 L volumetric flask; add 900 mL of water to dissolve; use glacial acetic acid to adjust pH=5.0; add water to fix-volume to 1 L; mix well. Use it after it is filtered through a 0.45 μm microporous membrane.

4.5 High-speed pulverizer.

4.6 Solid-phase extraction device.

4.7 Organic-phase microporous membrane: pore size of 0.45 μm .

5 Analysis steps

5.1 Sample preparation and storage

5.1.1 Liquid sample

Shake and pack the samples such as fruit juice, fruit juice drink and fruity carbonated drink; seal at room temperature or refrigerate to store.

5.1.2 Semi-solid sample

For samples such as jelly and flavor fermented milk, take the edible portion to homogenate; stir evenly; package; seal; refrigerate or freeze to store.

5.1.3 Solid sample

For samples of low-water content, such as biscuits, cakes and cooked meat products, use the high-speed pulverizer to pulverize; package; seal and store at room temperature out of the sun. For powdery samples with a uniform shape such as solid beverage, package directly; seal and store at room temperature out of the sun.

5.2 Sample processing

5.2.1 Liquid sample

Weigh 5 g (accurate to 0.01 g) of uniform sample (if the sample contains carbon dioxide; first heat to remove it) in a beaker; use an appropriate amount of absolute ethanol to dissolve it; transfer it to a 50 mL volumetric flask; add absolute ethanol to fix-volume; shake well. Accurately transfer 20 mL of extract which is mentioned above to a 100 mL pear-shaped bottle; add 10 mL of absolute ethanol to the pear-shaped bottle; rotate-concentrate the mixture to near dry at $60^{\circ}\text{C} \pm 2^{\circ}\text{C}$; use 2 mL of 20% methanol solution to wash the pear-shaped bottle twice.

Transfer all the above-mentioned extracts to a pre-activated HLB solid-phase extraction column; control the flow rate at 1 mL/min ~ 2 mL/min; discard the effluent. Use 5 mL of 40% methanol solution to rinse the purification column; discard the effluent; then use 5 mL of methanol to elute; control the flow rate at 1 mL/min ~ 2 mL/min; collect the eluate in a 25 mL graduated centrifuge tube. Use nitrogen to concentrate the eluent at 45°C ; then, use 2 mL of 40% methanol solution to dissolve the residue; filter it through a 0.45 μm organic filter membrane; test it later.

5.2.2 Semi-solid and solid samples

Weigh 5 g (accurate to 0.01 g) of uniform sample; put it into a 50 mL plastic centrifuge tube; add 20 mL of 80% ethanol solution; homogenize at 15 000 r/min for 2 min and at 4 000 r/min for 3 min; transfer the supernatant to a 50 mL volumetric flask; add 20 mL of 80% ethanol solution to the residue and repeat the extraction once; combine the extracts in the same volumetric flask; use absolute ethanol to fix-volume; shake well.

Accurately transfer 20 mL of extract which is mentioned above to a 100 mL pear-shaped bottle; add 10 mL of absolute ethanol to the pear-shaped bottle; rotate-concentrate the mixture to near dry at $60^{\circ}\text{C} \pm 2^{\circ}\text{C}$; use 2 mL of 20% methanol solution to wash the pear-shaped bottle twice. Transfer all the above-mentioned purification liquor to a pre-activated HLB solid-phase extraction column; control and the flow rate at 1 mL/min ~ 2 mL/min; discard the effluent. Use 5 mL of 40% methanol solution to rinse the purification column; discard the effluent; then use 5 mL of methanol to elute; control the flow rate at 1 mL/min ~ 2 mL/min; collect the eluate in a 25 mL graduated centrifuge tube. Use nitrogen to concentrate the eluent at 45°C ; then, use 2 mL of 20% methanol solution to dissolve the residue; filter it through a 0.45 μm organic filter membrane; test it later.

5.3 Apparatus reference conditions

Apparatus reference conditions are listed as below:

- a) Chromatographic column: high-purity spherical porous silica gel multi-point bonding C_{18} amino column, with a column length of 250 mm, an inner diameter of 4.6mm, a particle size of 5 μm , OR columns of equivalent performance;
- b) Mobile phase: A: acetic acid-ammonium acetate solution (pH=5); B: methanol, gradient elution is shown in Table 1;
- c) Column temperature: 40°C ;
- d) Injection volume: 20 μL ;
- e) Detection wavelength: that for monascorubramine is 264 nm, and for monascorubrin and monacolin is 390 nm.

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