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National food safety standard - Determination of acesulfame potassium in food

食品安全国家标准　食品中乙酰磺胺酸钾的测定

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1 Scope

This Standard specifies the method for the determination of acesulfame potassium in food.

This Standard applies to the determination of acesulfame potassium in milk and dairy products, frozen drinks, fruit products, vegetable products, edible fungi and algae, legume products, nuts and seeds, candy, grain products, baked goods, condiments, beverages, alcoholic beverages and jelly.

2 Principle

The acesulfame potassium in the sample is extracted with water, subjected to protein precipitation using potassium ferrocyanide/zinc acetate solution or purified with a neutral alumina solid-phase extraction column depending on the sample type, diluted to the mark with water, filtered through a microporous membrane, separated by liquid chromatography, detected with a UV detector or a diode array detector, subjected to qualitative identification based on the retention time, and quantified by external standard method.

3 Reagents and materials

Unless otherwise stated, the reagents used in this method are of analytical regent, and the water is Grade 1 water specified in GB/T 6682.

3.1 Reagents

3.1.1 Methanol (CH_3OH): chromatographically pure.

3.1.2 Acetonitrile (CH_3CN): chromatographically pure.

3.1.3 Potassium ferrocyanide [$K_4Fe(CN)_6 \cdot 3H_2O$].

3.1.4 Zinc acetate [$Zn(CH_3COO)_2 \cdot 2H_2O$].

3.1.5 Ammonium sulfate [$(NH_4)_2SO_4$].

3.1.6 Glacial acetic acid (CH_3COOH).

3.2 Preparation of reagents

3.2.1 Potassium ferrocyanide solution (92 g/L): Weigh 106 g of potassium ferrocyanide, add water to dissolve, and dilute to 1000 mL.

3.2.2 Zinc acetate solution (183 g/L): Weigh 219 g of zinc acetate, dissolve in a small amount of water, add 32 mL of glacial acetic acid, and dilute to 1000 mL with water.

3.2.3 Ammonium sulfate solution (0.02 mol/L): Weigh 2.64 g of ammonium sulfate, add water to dissolve, and dilute to 1000 mL.

3.2.4 Eluent: Mix methanol, acetonitrile and ammonium sulfate solution in a volume ratio of 5 : 10 : 85 to obtain a methanol-acetonitrile-ammonium sulfate solution (5 + 10 + 85), which is the eluent.

3.3 Reference material

Acesulfame potassium reference material ($C_4H_4KNO_4S$, CAS number: 55589-62-3), purity > 99.0 %, or a reference material certified by the country and awarded a reference material certificate.

3.4 Preparation of standard solutions

3.4.1 Acesulfame potassium standard stock solution (1000 mg/L): Accurately weigh 100 mg of acesulfame potassium reference material (accurate to 0.1 mg), dissolve in water and dilute to 100 mL, and mix well. Store at 0 ℃ ~ 4 ℃, the validity period is 6 months.

3.4.2 Standard intermediate solution (100 mg/L): Pipette 10 mL of acesulfame potassium standard stock solution (1000 mg/L) into a 100 mL volumetric flask, dilute with water and adjust the volume to the mark, and mix well. Prepare freshly each time before use.

3.4.3 Standard series working solutions: Pipette an appropriate amount of acesulfame potassium standard working solution with a concentration of 100 mg/L, dilute with water and adjust the volume to the mark, and prepare standard series working solutions with the concentrations of acesulfame potassium are 0.200 mg/L, 1.00 mg/L, 5.00 mg/L, 10.0 mg/L, 50.0 mg/L and 100 mg/L, respectively. Prepare freshly each time before use.

3.5 Materials

3.5.1 Neutral alumina solid-phase extraction column (6 mL, 500 mg), activated with 10 mL of acetonitrile before use, and then balanced with 10 mL of water.

3.5.2 Water phase microporous filter membrane: 0.45 μm.

3.5.3 Plastic centrifuge tube: 50 mL.

4 Instruments and equipment

4.1 High-performance liquid chromatograph: equipped with a UV detector or a diode array detector.

4.2 Electronic balances: the minimum divisions are 0.1 mg and 1 mg, respectively.

4.3 High-speed centrifuge: speed ≥ 10000 r/min.

4.4 Food grinder.

4.5 Vortex mixer.

4.6 Constant temperature water bath.

4.7 Ultrasonic generator.

4.8 Solid-phase extraction device.

4.9 Homogenizer.

5 Analysis procedure

5.1 Preparation of samples

Homogeneous liquid samples are mixed directly, and gas-containing samples are transferred to a beaker before use and degassed in a 50 °C water bath for 10 minutes or ultrasonic for 5 minutes; non-homogeneous liquid and semi-solid samples are homogenized with a homogenizer; solid samples are ground thoroughly with a food grinder and mix well. After the sample is processed, take 200 g and store it in a clean plastic bottle, mark it with a label, and store it at room temperature or under the storage conditions labeled on the sample for later use.

5.2 Treatment of samples

5.2.1 Beverages (except protein drinks), fruit products, vegetable products

Weigh about 5 g (accurate to 0.001 g) of the sample in a 50 mL centrifuge tube, add 20 mL of water, vortex to mix well, sonicate for 20 minutes, centrifuge at 10000 r/min for 5 minutes, and transfer the supernatant to a 50 mL volumetric flask. Add 20 mL of water to the centrifuge tube, mix well, sonicate for 5 minutes and centrifuge. Combine the supernatants, dilute to the mark with water, and mix well. Add 5.00 mL of the extraction solution to the activated neutral alumina solid-phase extraction column, and collect the eluent. Wash the solid-phase extraction column twice with 2 mL of eluent respectively, collect the eluents, combine the eluent of 3 times, dilute to 10 mL with the eluent, and

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