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SN

**INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

SN/T 5217-2019

**Determination of Brominated Vegetable Oil in Exported Food
(English Translation)**

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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard was proposed and managed by the General Administration of Customs of the People's Republic of China.

Drafting organization of this standard. Gongbei Customs of the People's Republic of China.

1 Scope

This standard specifies the method for determination of brominated vegetable oil content in imported and exported food.

This standard applies to the determination of brominated vegetable oil content in beverages and fruit juices.

2 Method summary

After the sample is extracted and concentrated, it reacts with zinc powder under acidic conditions. After the reaction solution is diluted and purified, it is leached with potassium hydroxide solution Wash liquid, anion exchange column separation, conductivity detector detection. Qualitatively by retention time and quantified by external standard method.

3 Reagents and materials

Unless otherwise specified, the reagents used in this standard are of analytical grade, and the experimental water is ultrapure water with a conductivity of 18.2 Mohm·cm.

3.1

Ether.

3.2

Petroleum ether. boiling range 30 °C ~ 60 °C.

3.3

Glacial acetic acid.

3.4

Diethyl ether-petroleum ether mixed extract. Take 500 mL of diethyl ether and 500 mL of petroleum ether and mix it evenly, and seal it for later use.

3.5

Anhydrous sodium sulfate (Na_2SO_4). Burn at 650°C for 4 hours, cool to room temperature in a desiccator, and store in a sealed bottle for later use.

3.6

Potassium hydroxide. pure superior grade.

3.7

Zinc powder.

3.8

Potassium bromide (KBr) standard product (CAS number. 7758-02-3). purity $\geq 99\%$.

3.9

Bromide ion standard stock solution (1 g/L). accurately weigh 0.148 9 g of potassium bromide, dissolve it in water, dilute to 100 mL, and store at 4 °C.

3.10

Bromide ion standard working solution (50 mg/L). accurately pipette 5.00 mL bromide ion standard stock solution (3.9), dilute and dilute to 100 mL, store at 4°C.

3.11

Acetic acid solution (1 to 9, volume ratio). Take 10 mL of glacial acetic acid, add 90 mL of water, mix well, and bottle it for later use.

3.12

Potassium hydroxide stock solution (1 mol/L). Weigh 5.61 g of potassium hydroxide, dissolve it in 100 mL of water, shake it well, and pour it into a polyethylene container.

In the container, keep it tightly closed until the solution is clear.

3.13

Potassium hydroxide eluent (15 mmol/L). Take 15 mL of the supernatant of potassium hydroxide stock solution (3.12) and dilute to 1 L with water.

It can be used after vacuum degassing and filtering, or it can use equipment that can automatically generate eluent to produce a solution of the same concentration online.

3.14

Strong acid cation exchange column (1 mL). H-type polystyrene-based strong acid resin packing with an average particle size of 40 μm , used before use Wash with 20 mL of water.

3.15

C18 solid phase extraction column (250 mg). C18 packing with an average particle size of 40 μm , activated with 5 mL methanol before use Wash with 20 mL of water.

3.16 0.45 μm water filter membrane.

4 Apparatus and equipment

4.1

Ion chromatograph, electrical conductivity detector.

4.2

Analytical balance. Sensitivity is 0.1 g, 0.0001 g.

4.3

Separation funnel, 500 mL.

4.4

Rotary evaporator.

4.5

Boiling water bath (or dry thermostat).

4.6

Ultra-pure water machine.

5.1 Sample preparation

5.1.1

Take a representative part of about 1 kg of the sample, shake it well, divide it into two parts, put them into clean containers as samples, and seal them.

And mark. Store the sample at 4°C and avoid light to prevent contamination of the sample.

5.1.2

Beverages containing carbon dioxide gas should be fully stirred with a stirrer or ultrasonically removed to remove carbon dioxide before testing.

5.1.3

Beverages containing pulp or other insoluble solids should be processed with a homogenizer before testing.

5.2.1 Extract

Accurately pipette 200 g (accurate to 0.1 g) of the mixed sample into a 500 mL separatory funnel (4.3), and add 100 mL to mix. Shake the extraction solution (3.4) gently, cover the bottle stopper after exhausting, and shake at room temperature for about 10 minutes. After fully standing for stratification, the lower water phase is transferred to another 500 mL separatory funnel (4.3), repeat the above process once, combine the ether layers, and wash the ether layer once with 50 mL water.

The ether solution is filtered and dehydrated by anhydrous sodium sulfate (3.5), and the filtrate is put into a 500 mL round-bottomed flask and placed on a rotary evaporator (4.4).

40 °C ± 2 °C under nitrogen filling conditions to steam to nearly dry (should not evaporate to dryness).

5.2.2 Conversion

Use about 10 mL of ether (3.1) to transfer the extraction residue to a 100 mL colorimetric tube, add about 30 mg of zinc powder (3.7) and 5.0 mL of acetic acid solution (3.11), mix well, and place it in a boiling water bath (4.5) to heat for 5 min. After the reaction is over, take it out and let it cool.

Dilute with water and dilute to 100 mL.

Note: Ether is a flammable liquid, and its vapor forms an explosive mixture with air. It is very easy to burn and explode when exposed to open flames and high heat. Open flame should not be used during heating reaction; the electric stove should be placed in a fume hood.