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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

GB 5009.253-2016

**National Standard for Food Safety -
Determination of Perfluorooctane Sulfonate (PFOS) and
Perfluorooctanoic Acid (PFOA) in Animal-derived Food**

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National Standard for Food Safety -

Determination of Perfluorooctane Sulfonate (PFOS) and

Perfluorooctanoic Acid (PFOA) in Animal-derived Food

1 Scope

This Standard specifies the method of isotope dilution liquid chromatography-tandem mass spectrometry in the determination of PFOS and PFOA in animal-derived food.

This Standard shall be applicable to the determination of PFOS and PFOA in animal-derived food.

2 Principle

Adopt acetonitrile to extract PFOS and PFOA from the sample. Start dispersive solid phase extraction and purification; adopt liquid chromatography-tandem mass spectrometer to determine PFOS and PFOA. Adopt the internal standard method to quantify the content.

3 Reagents and Materials

Unless otherwise indicated, the reagents adopted under this method are of analytical purity. The water is first-grade water as specified in GB/T 6682.

- 3.1** Methanol (CH_3OH): chromatographic purity.
- 3.2** Acetonitrile (CH_3CN): chromatographic purity.
- 3.3** Hydrochloric acid (HCl).
- 3.4** Sodium chloride (NaCl).
- 3.5** Ammonium acetate ($\text{CH}_3\text{COONH}_4$): chromatographic purity.
- 3.6** N-propyl ethylenediamine solid phase adsorbent (PSA): 40 μm ~60 μm , ProElut filler, or equivalent.
- 3.7** C_{18} absorbent: 40 μm ~60 μm , ProElut filler, or equivalent.
- 3.8** Graphitized carbon black adsorbent (GCB): 40 μm ~60 μm , ProElut filler, or

mixed series of standard working fluid of PFOA (density: 0.05 µg/L, 0.1 µg/L, 0.5 µg/L, 1.0 µg/L, 2.0 µg/L, 10.0 µg/L) and mixed series of standard working fluid of PFOS (density: 0.1 µg/L, 0.2 µg/L, 1.0 µg/L, 10.0 µg/L, 20.0 µg/L, 40.0 µg/L). Prepare mixed series of standard working solution of $^{13}\text{C}_4$ -PFOA (density: 1.0 µg/L) and 1, 2, 3, 4- $^{13}\text{C}_4$ -PFOS (density: 5.0 µg/L) that contains ^{13}C isotope. Store under the temperature of -4°C. It can remain valid for 2 months.

4 Instruments and Equipment

4.1 Liquid chromatography-tandem mass spectrometer: equipped with ESI source.

4.2 Nitrogen blowing instrument.

4.3 Grinder.

4.4 Centrifuge, speed ≥ 5,000 r/min.

4.5 Homogenizer, reference speed: 3,400 r/min~24,000 r/min.

4.6 Analytical balance: division value: 0.1 mg and 0.01 g.

5 Preparation of Samples

Take edible part (remove the bone and shell) from fish, shrimp, crab, shellfish, octopus, chicken, pork and beef. Cut into small pieces, then, use grinder to make it into meat paste; remove eggshell, then, homogenize it; mix up milk for later usage. Place the sample in the glass sample bottle, seal it, label it; store it under the temperature of -18°C.

6 Analytical Procedures

6.1 Pre-processing of Samples

6.1.1 Extracting

Weigh-take 5 g (accurate to 0.01 g) of sample (before usage, unfreeze and homogenize the sample); place it in 50 mL polypropylene centrifuge tube. Add 400 µL of mixed standard solution II, then, add 5 mL of water. Start 1 min whirlpool mixing, then, add 10 mL of acetonitrile (3.2) and 30 µL of hydrochloric acid (3.3); shake it for 10 min. Add 2 g of sodium chloride (3.4); shake again for 10 min; centrifuge at 5,000 r/min for 10 min. Remove supernatant acetonitrile solution, then, place it in another tube. Place the tube in water bath at 45°C, then, blow nitrogen till it is dry. Add 1 mL of methanol (3.1) to dilute; use injector to inhale 1 mL. Use 0.22 µm organic membrane to filter it; reserve for determination.

Before usage, conduct blank test on all organic solvents and test vessels adopted in this Standard. If their base value is higher than the limit of quantitation, re-evaporate the organic solvents, or replace the test vessels, until the base value is lower than the limit of quantitation.

8 Expression of Analysis Results

The content of PFOA and PFOS in the sample shall be calculated in accordance with Formula (1):

$$X = \frac{(A - A_0) \times V \times f}{m} \dots\dots\dots (1)$$

Where:

X - The content of PFOA or PFOS in the sample, expressed in (µg/kg);

A - The peak area ratio between PFOA or PFOS chromatographic peak and the chromatographic peak of corresponding internal standard substance in the test solution, and PFOA or PFOS density obtained through standard curve line, expressed in (µg/L);

A₀ - The peak area ratio between PFOA or PFOS chromatographic peak and the chromatographic peak of corresponding internal standard substance in the blank solution, and PFOA or PFOS density obtained through standard curve line, expressed in (µg/L);

V - The ultimate constant volume of the test solution, expressed in (mL);

f - Dilution times of the test solution;

m - The mass of the sample, expressed in (g).

The calculation result shall be expressed as the arithmetic mean value of two independent determination results obtained under repeatability conditions. The result shall retain two significant figures.

9 Precision

The absolute difference between the two independent determination results obtained under repeatability conditions shall not exceed 30% of the arithmetic mean value.

10 Others

The detection limit of PFOA is 0.002 µg/kg; the detection limit of PFOS is 0.02 µg/kg.

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