GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 31604.35-2016

National Food Safety Standard - Food contact materials and products - Determination of perfluorooctane sulfonate (PFOS) and perfluoro caprylic acid (PFOA)

Issued on: October 19, 2016 **Implemented on: April 19, 2017**

Issued by: National Health and Family Planning Commission of People's Republic of China

Table of Contents

Foreword

This standard replaces GB/T 23243-2009 "Determination of perfluorooctane sulfonates (PFOS) in the food packaging material - High performance liquid chromatography-tandem mass spectrometry".

The main technical differences of this standard in comparison with GB/T 23243-2009 are as follows:

- Changed the name of the standard to read "National Food Safety Standard - Food contact materials and products - Determination of perfluorooctane sulfonate (PFOS) and perfluoro caprylic acid (PFOA)";

- Added scope of application of the standard;

- Added the content of determination object PFOA;

- Added the method of sample preparation;

- Added the purification step. Use the weak anion exchange solid phase extraction column to purify;

- Increased the sensitivity of the determination method.

National Food Safety Standard - Food contact materials and products - Determination of perfluorooctane sulfonate (PFOS) and perfluoro caprylic acid (PFOA)

1 Scope

This standard specifies the method for the determination of perfluorooctane sulfonate (PFOS) and perfluorooctanoic acid (PFOAA) in food contact materials and products by tandem mass spectrometry.

This standard is applicable to the determination of PFOS and PFOA in food contact materials and products such as carton, rubber, polyethylene, plastics, resin, non-stick pan coating and so on.

2 Principle

PFOS and PFOA in food contact materials and products were extracted by methanol to accelerate solvent extraction, purified by weak anion exchange solid-phase extraction column, separated by liquid chromatography, ionized by electrospray ionization, determined by multi-reaction monitoring mode and quantified by Isotopic internal standard quantity.

3 Reagents and materials

Unless otherwise stated, the reagents used in this method shall be analytical pure. The water shall be the primary water specified in GB/T 6682.

Note: All organic solvents and materials used in this standard shall be blank tested before using. If the background value is higher than the quantitative limit, the organic solvent shall be re-steamed. Replace the test material until the background value is below the limit of quantification.

3.1 Reagents

3.1.1 Methanol (CH_3OH): Chromatographic purity.

3.1.2 Acetonitrile (CH_3CN): Chromatographic purity.

5 Analytical steps

5.1 Sample preparation

For plastics, silica gel sample: cut to size below 5mm×5mm; then grind into powder by liquid nitrogen frozen grinder. For resin: smash then grind into powder with liquid nitrogen frozen grinder. For coated sample: scrape with knife, then grind into powder by liquid nitrogen frozen grinder. For cardboard box and polyethylene sample: use scissors to cut to 1cm×1cm.

5.2 Extraction and purification

5.2.1 Extraction

Weigh 1g (accurate to 0.01g) of sample and put it into an accelerated solvent extraction pool. The extraction solvent is methanol. The volume of extraction solvent is 60% the volume of the sample pool. The extraction temperature shall be 110°C. The heating time shall be 5 min. Balance for 5 min. Repeat twice. Place the extraction solution at room temperature. Dry with nitrogen-blowing to about 0.5 mL. Add 10mL of water and mix for purification.

5.2.2 Purification

Activate and balance the WAX solid phase extraction column with 4 mL of methanol animation, 4 mL of methanol and 4 mL of water in turn. Then transfer the above solution to the solid phase extraction column. Add 4mL of 25mmol/L ammonium acetate buffer solution to rinse-wash. Add 4 mL of 0.1% ammonia methanol to elute. Collect the eluent and dry by nitrogen at 40°C. Re-dissolve by 1 mL of methanol, then pour through.22μm microporous membrane. Analyze by LC- MS/MS.

5.3 Reference conditions for liquid chromatography

5.3.1 Chromatographic column: C_{18}, column length is 150mm, inner diameter is 2.1mm, particle size is 3μm, or the column with equivalent performance.

5.3.2 Column temperature: 40 °C.

5.3.3 Sample volume: 10μL.

5.3.4 Mobile phase: 5 mmol/L ammonium acetate: acetonitrile. For gradient elution conditions, see Table 1.

5.3.5 Flow rate: 0.2 mL/min.

This is an excerpt of the PDF (Some pages are marked off intentionally)

------ The End ------