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National Standard for Food Safety - Determination of Polydimethylsiloxane in Animal and Vegetable Fats and Oils

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National Standard for Food Safety - Determination of Polydimethylsiloxane in Animal and Vegetable Fats and Oils

1 Scope

This Standard specifies the method of determining polydimethylsiloxane in animal and vegetable fats and oils.

This Standard shall be applicable to the determination of polydimethylsiloxane in animal and vegetable fats and oils.

Method I Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

2 Principle

Extract polydimethylsiloxane from 1# aviation kerosene in the sample. Adopt inductively coupled plasma atomic emission spectrometer to determine the content of silicon in the sample extract. Adopt the external 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 Reagents

3.1.1 1# Aviation kerosene (or it can be considered as organic solvents that can be directly injected into inductively coupled plasma atomic emission spectrometer and dissolve polydimethylsiloxane).

3.1.2 Hydrochloric acid (HCl).

3.1.3 Hydrochloric acid solution (1.37 mol/L): weigh-take 114 mL of hydrochloric acid; add water to dilute to 1,000 mL.

3.2 Standards

1.37 mol/L hydrochloric acid, and mix it thoroughly. Start static stratification in water bath, then, take 10 mL of supernatant and reserve it in 15 mL plastic centrifuge tube. Simultaneously start reagent blank test. Easily solidified grease shall be placed in water bath at 70°C, and immediately measured after being taken out.

5.2 Instrument Reference Conditions

Please refer to Table A.1.

5.3 Formulation of Standard Curve Lines

Inhale the standard series of working fluid into inductively coupled plasma atomic emission spectrometer; determine the intensity of emission. Take the density of polydimethylsiloxane in the standard working fluid as the x-coordinate, and the value of emission intensity as the y-coordinate. Draw the standard curve line.

5.4 Determination of Sample Solution

Inhale the blank and sample solution into inductively coupled plasma atomic emission spectrometer; determine the intensity of emission. Obtain the density (μg/mL) of polydimethylsiloxane in the test solution in accordance with the standard curve line.

6 Expression of Analysis Results

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

$$X_i = \frac{(\rho_i - \rho_0) \times V \times 1\,000}{m \times 1\,000} \times F \qquad \cdots\cdots(1)$$

Where:

X_i - The content of polydimethylsiloxane in the sample, expressed in (mg/kg);

p_i - The density of polydimethylsiloxane in the extracted solution, expressed in (μg/mL);

p_o - The density of polydimethylsiloxane in the blank grease solution, expressed in (μg/mL);

V - The volume of the sample, expressed in (mL);

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

F - Dilution factor;

1000 - Conversion factor.

10.3.2 Standard working fluid of polydimethylsiloxane: weigh-take 4 g (accurate to 0.01 g) of vegetable oil or animal oil or hydrate vegetable oil blank grease (blank grease is a kind of grease that doesn't include polydimethylsiloxane in the market), then, place it in 10 mL volumetric flask. Place what's need to be dissolved into water bath kettle at 70°C; add 2 mL~3 mL of 4-methyl-2-pentanone to dilute. Respectively transfer and take 0 μL, 40 μL, 100 μL, 200 μL, 300 μL and 500 μL of standard stock solution of polydimethylsiloxane. Add 4-methyl-2-pentanone to dilute to the constant volume, and shake it well. Obtain 0 μg/mL, 4 μg/mL, 10 μg/mL, 20 μg/mL, 30 μg/mL and 50 μg/mL of standard working fluid of polydimethylsiloxane based on the vegetable oil or animal oil or hydrate vegetable oil. Easily solidified grease shall be placed in water bath at 70°C, and immediately measured after being taken out.

11 Instruments and Equipment

11.1 Atomic absorption spectrometer: silicon-attached hollow cathode lamp; nitrous oxide-acetylene gas system (pressure reducing valve with heating device); combustion head exclusive for nitrous oxide-acetylene gas system.

11.2 Balance: division value is 0.1 mg.

11.3 Water bath kettle: with temperature-regulating device.

11.4 Volumetric flask: 10 mL, 100 mL.

12 Analytical Procedures

12.1 Preparation of Samples

Vegetable oil or animal oil or hydrate vegetable oil: weigh-take 4 g (accurate to 0.01 g) and place it in 10 mL volumetric flask. Place what's need to be dissolved into water bath kettle at 70°C; add 4-methyl-2-pentanone to dilute to the constant volume, then, shake it well. Simultaneously start reagent blank test.

12.2 Instrument Reference Conditions

12.2.1 Wavelength: 251.6 nm.

12.2.2 Spectral passband: 0.2 nm.

12.2.3 Lamp current: 10 mA.

12.2.4 Acetylene flow: 4.0 L/min.

12.2.5 Nitrous oxide flow: 6.0 L/min.

12.3 Formulation of Standard Curve Lines

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