



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

GB 5009.262-2016

**Method for analysis of hygienic standard of edible oils
(English Translation)**

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Foreword

This Standard replaces "4.8 Determination of solvent residual" of GB/T 5009.37-2003, Method for Analysis of Hygienic Standard of Edible Oils, and "6 Determination of solvent residual" of GB/T 5009.117-2003, Method for Analysis of Hygienic Standard of Edible Soybean Meal.

Compared with GB/T 5009.37-2003 and GB/T 5009.117-2003, the major changes of this Standard are as follows.

- it changes the standard name into "National Food Safety Standard - Determination of Solvent Residual in Food";
- it modifies the method for analysis of solvent residual;
- it modifies the method for plotting standard curves;
- it modifies the calculation formula of results.

National Food Safety Standard -

Determination of Solvent Residual in Food

1 Application Scope

This Standard specifies the method for determination of solvent residual in edible vegetable oils and edible soybean meal.

This Standard applies to the determination of solvent residual in edible vegetable oils and edible soybean meal.

2 Terms and Definitions

For the purposes of this document, the following terms and definitions apply.

2.1 Matrix vegetable oil

Refined vegetable oil obtained through the refining processes including colour and odour removal, or vegetable oil produced by ultrasonic degassing at room temperature, of the same species as that of specimen to be tested. The solvent residual in matrix vegetable oil shall be lower than the detection limit.

3 Principle

The solvent residual existing in specimen will diffuse to gas phase in a closed container; after a certain time, the dynamic equilibrium between the concentrations of gas phase and liquid phase can be achieved; the content of solvent residual in the upper gas phase is measured by headspace gas chromatography, i.e.

4 Reagents and Materials

Unless specified otherwise, all reagents used for this method are analytically pure and the water is of grade 1 water specified in GB/T 6682.

4.2 Preparation of reagent

n-heptane standard working solution. add 1 mL of n-heptane accurately in a 10-mL volumetric flask immediately; add quickly N,N-dimethylacetamide; add dropwise to the scale.

4.3 Standard product

Solvent residual standard product. "no. 6 solvent" solution of concentration 10 mg/mL; the solvent is N,N-dimethylacetamide. Or other standard products for the determination of solvent residual, which have been certified by the state or granted standard product certificates.

4.4.1 For vegetable oils, weigh 6 shares of 5.0 g (accurate to 0.01 g) of matrix vegetable oil to place into a 20-mL headspace specimen bottle.

4.4.2 For meals, weigh 6 shares of 3.0 g (accurate to 0.01 g) of matrix meal to place

into a 20-mL headspace specimen bottle; add 400 μ L of water to each headspace specimen bottle; finally use microsyringe to add quickly 0 μ L, 3 μ L, 9 μ L, 15 μ L, 30 μ L and 150 μ L of no. 6 solvent standard product; after sealing, obtain the matrix meal standard solutions of concentrations 0 mg/kg, 10 mg/kg, 30 mg/kg, 50 mg/kg, 100 mg/kg and 500 mg/kg.

5.2 Headspace bottle. 20 mL, equipped with aluminium cover and sealing washer of

butyl rubber or silicon resin containing no residual of hydrocarbon solutions.

6.1.1 Preparation of vegetable oil specimen. weigh 5 g (accurate to 0.01 g) of vegetable oil specimen to place into a 20-mL headspace specimen bottle; add quickly

5 μ L of n-heptane standard working solution to vegetable oil specimen as internal

standard; use hand to shake up before sealing. Keep headspace specimen bottle upright for analysis. During the preparation, vegetable oil specimen shall not touch sealing washer; if vegetable oil specimen touches sealing washer, the specimen shall be prepared once again.

6.2.1 Headspace specimen injector reference conditions

The reference conditions for headspace specimen injector are as follows.

6.2.2 Gas chromatography reference conditions

The reference conditions for gas chromatography are as follows.

6.3.1 For vegetable oils, the internal standard method is used in this method for quantitation.

6.4 Specimen determination

After analyzing the prepared vegetable oil or meal specimen, measure its peak area and calculate the solvent residual in specimen in accordance with relevant standard curves.

7 Expression of Analytical Results

The content of solvent residual in specimen is calculated in accordance with Formula (1).

8 Accuracy

The absolute difference between the results of two independent measurements under repeatable conditions shall not exceed 10% of the arithmetic mean value.

9 Others

The detection limits and quantitation limits of this method are as follows.

Annex A

Gas Chromatogram of No. 6 Solvent

See Figure A.1 for the gas chromatogram of no. 6 solvent.

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