GB

NATIONAL STANDARD OF THE

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National Food Safety Standard - Determination of Trans-fatty Acids in Foodstuffs

食品安全国家标准

食品中反式脂肪酸的测定

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National Food Safety Standard - Determination of Trans-fatty Acids in Foodstuffs

1 Scope

This Standard specifies the gas chromatographic method for the determination of trans-fatty acids and isomer in foods.

This Standard is applicable to the determination of trans-fatty acids in animal and vegetable fats and oils, hydrogenated vegetable oil, refined vegetable oil and frying oil, as well as foods that contain animal and vegetable fats and oils, hydrogenated vegetable oil, refined vegetable oil and frying oil.

This Standard is not applicable to the determination of food samples, in which, free fatty acid (FFA) content in fats and oils is more than 2%.

2 Principle

Animal and vegetable fats and oils samples, or fats in food samples extracted through acid hydrolysis method, under alkaline condition, go through transesterification with methanol; generate fatty acid methyl ester. Furthermore, on strong polarity stationary phase capillary chromatographic column, separate it. Use gas chromatograph, which is equipped with hydrogen flame ionization detector, for determination. Use area normalization method to quantify it.

3 Reagents and Materials

Unless it is otherwise stipulated, all reagents used in this method shall be analytically pure. Water shall be Grade-2 water stipulated in GB/T 6682.

3.1 Reagents

3.1.1 Hydrochloric acid (HCl, ρ_{20} = 1.19): content 36% ~ 38%.

3.1.2 Ether ($C_4H_{10}O$).

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

3.1.4 Absolute ethanol (C_HO_O): chromatographic purity.

3.1.5 Anhydrous sodium sulfate: before usage, at 650 °C, burn it for 4 h; store it in a dryer for later usage.

considered as the sample test solution.

5.2.2 Foods containing fats and oils (except from animal and vegetable fats and oils)

5.2.2.1 Determination of fats in foods

Solid and semi-solid lipid sample: weigh-take 2.0 g (accurate to 0.01 g; the weighing amount of different foods may be properly adjusted, so as to guarantee that fat mass in foods is not less than 0.125 g) of homogeneous sample. Place it in a 50 mL test tube. Add 8 mL of water to thoroughly mix it up. Then, add 10 mL of hydrochloric acid to evenly mix it up. Liquid sample: weigh-take 10.00 g of homogeneous sample, then, place it in a 50 mL test tube. Add 10 mL of hydrochloric acid to evenly mix it up. Place the above-mentioned test tube into 60 °C ~ 70 °C water bath. In every 5 min ~ 10 min, oscillate it once. Last for around 40 min ~ 50 min, till the sample is completely hydrolyzed. Take out the test tube. Add 10 mL of ethanol to thoroughly mix it up, then, cool it down to room temperature.

Transfer the mixture into 125 mL separating funnel. Take 25 mL of ether, divide it into two parts, then, respectively use them to rinse the test tube. Pour the rinsing liquid together into the separating funnel. After ether is completely poured into the funnel, put on a stopper; start to shake it for 1 min. Then, carefully open the stopper, release the gas. In addition, use an appropriate amount of petroleum ether - ether solution (1 + 1) to rinse the stopper and fats attached to the mouth of the funnel. Place it still for 10 min ~ 20 min, till the upper ether solution becomes limpid. Place the lower aqueous phase into 100 mL beaker. Place the upper organic phase into another clean separating funnel. Use a little petroleum ether - ether solution (1 + 1) to rinse the separating funnel for extraction; collect the organic phase, then, combine it in the separating funnel. Pour the aqueous phase in the beaker back to the separating funnel. Then, take 25 mL of ether, divide it into two parts, then, respectively use them to rinse the beaker. Pour the rinsing liquid together into the separating funnel. In accordance with the above-mentioned steps of extraction, repeatedly extract it twice. Combine the organic phase in the separating funnel. Let all the organic phase pass through a proper amount of anhydrous sodium sulfate column. Use a little petroleum ether - ether solution (1 + 1) to rinse the column. Gather all the effluent in a 100 mL measuring cylinder with a stopper. Use ether to reach a constant volume; evenly mix it up.

Accurately transfer-take 50 mL of organic phase, then, place it into round-bottomed flask, which already reaches a constant weight. In 50 °C water bath, rotate it and boil off the solvent. Then, place it at 100 °C ± 5 °C; reach a constant weight, then, calculate the content of fats in the foods. Place another 50 mL of organic phase in 50 °C water bath, rotate it and boil off the solvent; it shall be used for the determination of trans-fatty acid methyl ester.

5.2.2.2 Preparation of fatty acid methyl ester

m_1---mass of the round-bottomed flask and fats, expressed in (g);

m_0---mass of the round-bottomed flask, expressed in (g);

m_2---mass of the sample, expressed in (g).

6.2 Calculation of Relative Mass Fraction

The relative mass fraction of the various components shall be calculated in accordance with Formula (2):

$$w_X = \frac{A_x \times f_x}{A_t} \times 100\% \quad \cdots\cdots (2)$$

Where,

w_X---relative mass fraction of trans-fatty acid component X fatty acid methyl ester calculated through normalization method, expressed in (%);

A_x---peak area of component X fatty acid methyl ester;

f_x---calibration factor of component X fatty acid methyl ester; please refer to Table D.1 for the correction factor of chemical compounds;

A_t---the total of calibration area of all the peaks, except from solvent peak.

6.3 Calculation of Trans-fatty Acids Content in Fats

The mass fraction of trans-fatty acids in fats shall be calculated in accordance with Formula (3):

$$w_t = \sum w_X \quad \cdots\cdots (3)$$

Where,

w_t---mass fraction of trans-fatty acids in fats, expressed in (%);

w_X---relative mass fraction of component X fatty acid methyl ester calculated through normalization method, expressed in (%).

6.4 Calculation of Trans-fatty Acids Content in Foods

Mass fraction of trans-fatty acids content in foods shall be calculated in accordance with Formula (4):

$$w = w_t \times w_z \quad \cdots\cdots (4)$$

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