NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

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National food safety standard - Food nutritional fortifier L-methionine

食品安全国家标准 食品营养强化剂 L-蛋氨酸(L-甲硫氨酸)

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1 Scope

The standard applies to the food nutritional fortifier L-methionine obtained from edible starch or sugar raw materials by fermentation, extraction and refining, or obtained from DL-methionine by enzymatic resolution and refining.

2 Chemical name, molecular formula, structural formula and relative molecular mass

2.1 Chemical name: L-2-amino-4-(methylthio)butanoic acid.

2.2 Molecular formula: C5H11NO2S.

2.3 The structural formula is printed as a drawing and is not reproduced here.

2.4 Relative molecular mass: 149.21, on the 2022 international relative atomic masses.

The Chinese title gives the substance two synonymous Chinese names, the second in brackets, both of which correspond to methionine in English.

3 Technical requirements

3.1 Sensory requirements, given in Table 1. Colour: white or off-white. Physical state: crystals or crystalline powder. Smell: the characteristic smell. The test method is to place a suitable quantity of sample in a clean, dry white porcelain dish and to observe its colour and state and smell it in natural light.

3.2 Physical and chemical indicators, given in Table 2. L-methionine content on the dry basis, mass fraction 98.5 % to 101.5 %, determined as in A.4 of Annex A. Transmittance not less than 98.0 %, determined as in A.5. pH of a 10 g/L aqueous solution 5.6 to 6.1, determined as in GB/T 9724. Specific rotation at 20 °C with the sodium D line, expressed in degrees times square decimetre per kilogram, +21.0 to +25.0, determined as in A.6. Loss on drying, mass fraction not more than 0.2 %, determined as in GB/T 6284, with a footnote

setting the test portion at about 3 g and the drying time at 3 h. Residue on ignition, mass fraction not more than 0.1 %, determined as in A.7. Chloride expressed as Cl, mass fraction not more than 0.02 %, determined as in A.8. Lead (Pb) not more than 0.3 mg/kg, determined as in GB 5009.12 or GB 5009.75. Total arsenic expressed as As not more than 0.2 mg/kg, determined as in GB 5009.11 or GB 5009.76.

A Annex A Test methods

A.1 to A.2 Safety notice and general provisions. Some of the reagents used are toxic or corrosive and call for care; splashes on the skin shall be washed off with water at once and serious cases treated immediately; volatile acids shall be handled in a fume cupboard. Unless otherwise stated, the reagents are analytical grade and the water is grade three water as defined in GB/T 6682; standard volumetric solutions, standard solutions for impurity determination, preparations and their products are made up as in GB/T 601, GB/T 602 and GB/T 603; and solutions whose solvent is not stated are aqueous.

A.3 Identification. An infrared spectrum is run by the potassium bromide disc method as in GB/T 6040, and the spectrum of the test portion shall agree with the reference spectrum of L-methionine given in Annex B.

A.4 Determination of L-methionine content on the dry basis. Formic acid is used as a solubilising aid and glacial acetic acid as the solvent; the solution is titrated with standard perchloric acid volumetric solution and the content is calculated from the volume consumed. The reagents are glacial acetic acid, anhydrous formic acid, standard perchloric acid volumetric solution at a concentration of 0.1 mol/L and crystal violet indicator solution at 5 g/L; the apparatus is a potentiometric titrator, a burette and an electronic balance reading to 0.0001 g. A test portion of 0.14 g, dried to constant mass at 105 °C and weighed to 0.0001 g, is placed in a dry beaker or conical flask and dissolved in 3 mL of anhydrous formic acid and 50 mL of glacial acetic acid, then titrated with the standard perchloric acid solution to the end point on the potentiometric titrator; alternatively 2 drops of crystal violet indicator are added and the end point is taken when the solution turns from purple to blue-green. A blank is run at the same time. The mass fraction is calculated from formula (A.1), whose symbols are: the volume of standard perchloric acid solution consumed by the test solution, in millilitres; the volume consumed by the blank, in millilitres; the concentration of the standard perchloric acid solution, in moles per litre; the molar mass of L-methionine, in grams per mole, taken as 149.21; the mass of the test portion, in grams; and a conversion factor of 1 000. The result is the mean of parallel determinations, and the absolute difference between two independent results obtained under repeatability conditions shall not exceed 0.3 % of their mean.

A.5 Determination of transmittance. The apparatus is a spectrophotometer and an electronic balance reading to 0.01 g. A test portion of 0.5 g, weighed to 0.01 g, is dissolved in 20 mL of water, and the transmittance of the solution is measured in a 1 cm cell against

water as the blank at a wavelength of 430 nm, the reading being recorded. The result is the mean of parallel determinations.

A.6 Determination of specific rotation. The reagent is hydrochloric acid solution at 6 mol/L, made by taking 500 mL of hydrochloric acid and diluting with water to 1 000 mL. The apparatus is a polarimeter and an electronic balance reading to 0.0001 g. A test portion of 1 g, dried to constant mass at 105 °C and weighed to 0.0001 g, is dissolved in the hydrochloric acid solution and made up to 50 mL, and the measurement is made as in GB/T 613. The specific rotation at 20 °C with the sodium D line, expressed in degrees times square decimetre per kilogram, is calculated from formula (A.2), whose symbols are: the optical rotation measured at 20 °C, in degrees; the length of the polarimeter tube, in decimetres; and the mass concentration of L-methionine in the solution, in grams per millilitre.

A.7 Determination of residue on ignition. The reagent is concentrated sulfuric acid; the apparatus is a high-temperature furnace, a porcelain crucible, a desiccator and an electronic balance reading to 0.0001 g. A test portion of 2 g, weighed to 0.0001 g, is placed in a crucible previously ignited to constant mass at 800 °C +/-25 °C and ignited gently until completely carbonised, then cooled to room temperature. It is wetted with 1 mL to 2 mL of concentrated sulfuric acid and heated gently until the sulfuric acid vapour has gone, then ignited at 800 °C +/-25 °C until completely ashed; when the furnace has fallen to about 200 °C the crucible is taken out, moved to a desiccator, cooled for 30 min and weighed, the ignition at 800 °C +/-25 °C being repeated to constant mass. The mass fraction is calculated from formula (A.3), whose symbols are: the mass of the crucible at constant mass, in grams; the mass of crucible and test portion before ignition, in grams; and the mass of crucible and residue after ignition to constant mass, in grams. The result is the mean of parallel determinations, and the absolute difference between two independent results obtained under repeatability conditions shall not exceed 10 % of their mean.

A.8 Determination of chloride expressed as Cl. The reagents are nitric acid; nitric acid solution 1 plus 9 by volume; silver nitrate solution at 17 g/L; and a chloride standard solution prepared as in GB/T 602 as a stock solution at a mass concentration of 0.1 mg/mL, diluted with water immediately before use so that 1 mL of the standard solution holds 0.01 mg of chloride ion. A test portion of 0.30 g is placed in a 50 mL Nessler tube, dissolved in a suitable quantity of water with 10 mL of nitric acid solution, treated with 1 mL of silver nitrate solution, diluted to 50 mL with water and mixed; after 5 min in the dark the turbidity is viewed from above against a black background and compared with the standard turbidity solution, which is 6 mL of chloride standard solution placed in a 50 mL comparison tube and treated in the same way at the same time. The test passes, the chloride in the sample being not more than 0.02 %, when its turbidity is no deeper than that of the standard.

B Annex B Reference infrared spectrum of L-methionine

The annex carries Figure B.1, the reference infrared spectrum of L-methionine, against which the spectrum obtained in the identification test of A.3 is compared. The figure is a plotted spectrum and is not reproduced here.