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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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Animal and vegetable fats and oils - Determination of saponification value

动植物油脂 皂化值的测定

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1 Scope

The document describes the method for determining the saponification value of animal and vegetable fats and oils. The saponification value is an indicator of the content of free fatty acids and glycerides in an oil or a fatty acid.

The document applies to refined animal and vegetable fats and oils and to crude animal and vegetable oils.

The document does not apply to products containing inorganic acids, unless the inorganic acids can be determined separately.

The saponification value can also be obtained by calculation from the fatty acid data gained

by the gas chromatographic analysis shown in Annex B; for such a calculation it has to be made sure that the sample contains no impurities and no thermal degradation products.

3 Terms and definitions

3.1 Saponification value, symbol IS: the number of milligrams of potassium hydroxide needed to saponify one gram of fat or oil.

4 Principle

The test sample is saponified by boiling it under reflux with an excess of potassium hydroxide in ethanol solution, and the excess potassium hydroxide is then titrated with a standard hydrochloric acid titration solution.

5 Reagents

Unless otherwise stated, the reagents used in the document are of analytical grade and the water used is distilled water or demineralised water of equivalent purity.

5.1 Ethanol: volume fraction 95 %.

5.2 Potassium hydroxide in ethanol solution: concentration of potassium hydroxide 0,5 mol/L. The solution is colourless or pale yellow, and a stable colourless solution can be obtained by either of the following routes. Route a): reflux 1 L of the ethanol of 5.1 with 8 g of potassium hydroxide and 5 g of aluminium granules for 1 h, then distil at once; dissolve the required quantity of potassium hydroxide, about 35 g, in the distillate; leave to stand for several days, then pour the clear supernatant liquid off the precipitated potassium carbonate into a brown glass bottle. Route b): add 4 g of aluminium tert butoxide to 1 L of ethanol and leave the mixture to stand for several days; pour off the supernatant liquid and dissolve the required quantity of potassium hydroxide in it; leave to stand for several days, then pour the clear supernatant liquid off the precipitated potassium carbonate into a brown glass bottle.

5.3 Hydrochloric acid standard solution: concentration of hydrochloric acid 0,5 mol/L.

5.4 Alkali blue 6B solution: mass concentration 2,5 g per 100 mL of the ethanol solution of 5.1.

5.5 Phenolphthalein solution: mass concentration 0,1 g per 100 mL of the ethanol solution of 5.1. Phenolphthalein is classed as a carcinogenic, teratogenic or reproductively toxic (CMR) substance and should be used only where no substitute is available.

5.6 Boiling aid.

6 Apparatus

Ordinary laboratory apparatus is used, and in particular the following.

6.1 Conical flask: capacity 250 mL, made of alkali resistant glass, with a ground neck.

6.2 Reflux condenser: with a ground glass joint able to connect to the conical flask of 6.1.

6.3 Heating device: such as a water bath, an electric hotplate or another suitable device; an open flame shall not be used for heating.

6.4 Burette: capacity 50 mL with a smallest graduation of 0,1 mL, or an automatic burette.

6.5 Pipette: capacity 25 mL, or an automatic pipette.

6.6 Analytical balance: smallest scale division 0,000 1 g, weighing accuracy 0,001 g.

7 Sampling

Sampling is not part of what the document specifies; ISO 5555 is recommended.

It matters a great deal that the sample received by the laboratory is representative and has not been damaged or altered during transport or storage.

8 Preparation of test sample

The preparation is carried out as specified in ISO 661.

Where insoluble impurities are present in the test sample, the sample is mixed evenly and then filtered, and this shall be stated in the test report.

9 Procedure

9.1 Test portion: weigh about 2 g of the sample prepared according to clause 8 into the conical flask of 6.1, to the nearest 0,005 g. The weight of 2 g is based on a saponification value, expressed as potassium hydroxide, of 170 mg/g to 200 mg/g; for samples with saponification values in other ranges the test portion should be varied as needed so that about half of the potassium hydroxide in ethanol solution is neutralised. The recommended test portions are given in Table 1.

Table 1, headed Test portion, has two columns, estimated saponification value expressed as potassium hydroxide in mg/g and test portion in g, and four rows: 150 to 200 gives 2,2 to 1,8; 200 to 250 gives 1,7 to 1,4; 250 to 300 gives 1,3 to 1,2; above 300 gives 1,1 to 1,0.

9.2.1 Using the pipette of 6.5, add 25,0 mL of the potassium hydroxide in ethanol solution of 5.2 to the test portion, add some boiling aid of 5.6, connect the reflux condenser of 6.2 to the conical flask and place the flask on the heating device of 6.3; bring it slowly to the boil with occasional shaking and keep it boiling for 60 min, or for 2 h in the case of samples with a high melting point and samples that are difficult to saponify.

9.2.2 Add 0,5 mL to 1 mL of the indicator of 5.4 or 5.5 to the hot solution and titrate with the hydrochloric acid standard solution of 5.3 until the colour of the indicator has just disappeared. Where the colour of the solution is dark, phenolphthalein of 5.5 shall not be used as the indicator.

9.3 Blank test: a blank determination is carried out as required by 9.2, without the sample, using 25,0 mL of the potassium hydroxide in ethanol solution of 5.2.

10 Calculation of results

The saponification value IS is calculated by formula (1). The symbols are: V0, the volume of the hydrochloric acid standard solution of 5.3 consumed in the blank determination, in millilitres (mL); V1, the volume of the hydrochloric acid standard solution of 5.3 consumed in the sample determination, in millilitres (mL); c, the actual concentration of the hydrochloric acid standard solution of 5.3, in moles per litre (mol/L); m, the mass of the test portion of 9.1, in grams (g). The formula also carries the factor 56,1.

Where the repeatability requirement of 11.2 is met, the arithmetic mean of two determinations is taken as the result of the determination.

The result is expressed as a whole number, as potassium hydroxide, in milligrams per gram (mg/g).

11 Precision

11.1 Interlaboratory test: in 2000 the German Institute for Standardization (DIN) organised an interlaboratory test in which 22 laboratories took part, see Annex A; two determinations were made on each sample and statistical results were given, evaluated according to ISO 5725-1 and ISO 5725-2, the statistical results being shown in Table A.1.

11.2 Repeatability: the absolute difference between two test results obtained in the same laboratory by the same operator using the same equipment, by the same test method, on the same test object, independently and within a short interval of time, shall exceed the repeatability limit r shown in Table A.1 in not more than 5 % of cases.

11.3 Reproducibility: the absolute difference between two test results obtained in different laboratories by different operators using different equipment, by the same test method, on the same test object and independently, shall exceed the reproducibility limit R shown in Table A.1 in not more than 5 % of cases.

12 Test report

The test report shall state: a) all the relevant information needed to identify the test sample; b) the sampling method, where it is known; c) whether the test sample needed to be filtered; d) the test method document used, for example GB/T 5534-2024; e) a statement of