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Replacing GB/T 5534-1995

Animal and vegetable fats and oils - Determination of saponification value

动植物油脂　皂化值的测定

(ISO 3657:2002, MOD)

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Standardization Administration of PRC.

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Animal and vegetable fats and oils - Determination of saponification value

1 Scope

This standard specifies the method for the determination of the saponification value of animal and vegetable fats and oils.

This standard applies to refined and crude animal and vegetable fats and oils.

This standard does not apply to products containing mineral acids, unless the mineral acids can be determined otherwise.

2 Normative references

The provisions in the following documents become the provisions of this standard through reference in this standard. For the dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this standard, however, parties who reach an agreement based on this standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies to this standard.

GB/T 15687 Oils and fats - Preparation of test sample (GB/T 15687-1995, eqv ISO 661:1989)

3 Terms and definitions

The following terms and definitions apply to this standard.

3.1 saponification value

The number of milligrams of potassium hydroxide required to saponify 1 g of fat and oil under specified conditions.

4 Principles

The saponification value is used for the determination of the content of free fatty acids and glycerides in oils and fatty acids. The sample and potassium hydroxide-ethanol

solution are boiled under reflux, and then the excess potassium hydroxide is titrated with a standard hydrochloric acid solution.

5 Reagents

All reagents used are analytical grade, and the water used is distilled water or water of equivalent purity.

5.1 Potassium hydroxide-ethanol solution: About 0.5 mol of potassium hydroxide is dissolved in 1 L of 95% ethanol (volume fraction). This solution shall be colorless or light yellow. Stable colorless solutions can be prepared by any of the following methods.

Method a: 8 g of potassium hydroxide and 5 g of aluminum flakes are put in 1 L of ethanol to be refluxed for 1 h and distilled immediately. The required amount (about 35 g) of potassium hydroxide is dissolved in the distillate. After it stands for a few days, the clear supernatant is poured out and the potassium carbonate precipitate is discarded.

Method b: 4 g of aluminum tert-butoxide is added to 1 L of ethanol; after it stands for several days, the supernatant is poured out; the required amount of potassium hydroxide is dissolved in it; after the solution stands for several days, the clear supernatant is poured out and the potassium carbonate precipitate is discarded.

This solution is stored in a brown or yellow glass bottle with a rubber stopper for later use.

5.2 Hydrochloric acid standard solution: c(HCl) = 0.5 mol/L.

5.3 Phenolphthalein solution: (ρ=0.1 g/100 mL); it is dissolved in 95% ethanol (volume fraction).

5.4 Alkali blue 6B solution: (ρ=2.5 g/100 mL); it is dissolved in 95% ethanol (volume fraction).

5.5 Boiling aids.

6 Instruments and equipment

Common laboratory instruments and the following instruments:

6.1 Erlenmeyer flask: with a capacity of 250 mL, made of alkali-resistant glass, and with a ground mouth.

6.2 Reflux condenser: with a ground glass joint for connection to the Erlenmeyer flask (6.1).

6.3 Heating device (such as a water bath, electric hot plate, or other suitable devices): The open flame cannot be used for heating.

6.4 Burette: with a capacity of 50 mL and a minimum scale of 0.1 mL, or having an automatic burette.

6.5 Pipette: with a capacity of 25 mL, or having an automatic pipette.

6.6 Analytical balance.

7 Sampling

Sampling is not the content specified in this standard; GB/T 5524 is recommended.

Samples received by the laboratory shall be representative and not damaged or altered during transportation or storage.

8 Sample preparation

It shall be carried out according to GB/T 15687.

If there are insoluble impurities in the sample, the sample shall be mixed evenly and then filtered; it shall be indicated in the test report.

9 Operation steps

9.1 Weighing of samples

Weigh 2 g of the test sample (Chapter 8) and put it in the Erlenmeyer flask (6.1), and the weight shall be accurate to 0.005 g.

Based on the saponification value (in KOH) of 170 mg/g~200 mg/g and the sample weight of 2 g, for samples with different ranges of saponification value, the sampling weight can be changed on the basis of that about half of the potassium hydroxide-ethanol solution is neutralized. The recommended sampling weights are listed in Table 1.

m -- the mass of the sample (9.1), in grams (g).

If the repeatability meets the requirements of 11.2, take the arithmetic mean of the two determinations as the measurement result.

11 Precision

11.1 Interlaboratory testing

In 2000, the German Institute for Standardization (DIN) organized an inter-laboratory test involving 22 laboratories. Each sample was tested twice. See Appendix A for the statistical results.

11.2 Repeatability

In the same laboratory, according to the same test method, the same operator uses the same equipment to carry out two independent tests on the same measured object within a short period of time; the situation that the absolute difference between the two independent test results obtained is greater than the repeatability limit (r) shown in Appendix A shall not exceed 5%.

11.3 Reproducibility

In different laboratories, according to the same test method, different operators use different equipment to carry out two independent tests on the same measured object; the situation that the absolute difference between the two independent test results obtained is greater than the reproducibility limit (R) shown in Table A.1 shall not exceed 5%.

12 Test report

The following information needs to be indicated in the test report:

-- all relevant information required for the testing of the sample;

-- if the sampling method is known, it shall be indicated;

-- the inspection methods and referenced standards used;

-- the indicator used;

-- the operation details that are not specified in this standard or considered optional, and may affect the results;

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