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Animal and vegetable fats and oils - Determination of oxidation stability (Accelerated oxidation test)

动植物油脂　氧化稳定性的测定（加速氧化测试）

(ISO 6886:2006, IDT)

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Animal and vegetable fats and oils - Determination of oxidation stability (Accelerated oxidation test)

1 Scope

This standard specifies the method for the determination of the oxidative stability of oils and fats under extreme conditions that induce rapid oxidation: high temperature and high air flow.

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

This standard is not applicable to the determination of oil stability at room temperature, but it can be used to compare the efficacy of antioxidants added to oils and fats.

NOTE: The presence of volatile fatty acids and unstable acidic oxidation products affects the accuracy of the measurement results.

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 Animal and vegetable fats and oils - Preparation of test sample (eqv ISO 661)

3 Terms and definitions

The following terms and definitions apply to this standard.

3.1 induction period

The time from the start of the measurement to when the formation of oxidation products begins to increase rapidly.

3.2 oxidative stability

The induction period determined according to the steps specified in this standard and expressed in hours (h).

NOTE: The temperature usually used in the determination of oxidative stability is 100 ℃~120 ℃. According to the oxidative stability of the tested sample, or by an extrapolation of regression method, other temperature conditions can be used for the determination. The optimal induction period shall be between 6 h to 24 h. A temperature increase or decrease of 10 °C decreases or increases the induction period by a factor of approximately 2.

3.3 conductivity

The ability of a substance to conduct electric current.

4 Principle

A stream of purified air is passed through the sample, which has been brought to a specified temperature. The gases released during the oxidation process, together with the air, are passed into a long-necked flask containing water that has been demineralized or distilled and contains an electrode for measuring the conductivity. The electrode is connected to a measuring and recording device. During oxidation, a rapid increase in conductivity is caused by the accumulation of volatile carboxylic acid species. The end of the induction period is indicated when the conductivity begins to increase rapidly.

5 Reagents and materials

Unless otherwise stated, this standard only uses reagents of recognized analytical grade, and distilled or deionized water.

5.1 Molecular sieve: spherical, with beads of approximately 1 mm diameter and a pore size of 0.3 nm, and with moisture indicator.

Molecular sieves must be dried in an oven set at 150 °C and cooled down to room temperature in a desiccator.

5.2 Acetone.

5.3 Alkaline cleaning solution: used for cleaning laboratory glassware.

5.4 Glycerol.

5.5 Thermostable oil.

6.1.2 Gas diaphragm pump: The flow rate is adjusted to 10 L/h manually or through an automatic flow rate regulator, and the maximum deviation is ±1.0 L/h.

NOTE: When the OSI instrument is adjusted to a pressure of 0.038 MPa (5.5 psi), the flow rate is approximately 10 L/h.

6.1.3 Borosilicate glass aeration vessels (usually 8), equipped with sealing plugs.

The sealing plug shall be fitted with an air inlet and outlet tube. The cylindrical part of the vessel shall be narrower by a few centimeters below the top in order to break up any emerging foam. Foam can also be eliminated with an artificial foam blocker such as a glass ring.

6.1.4 Closed measuring cells (usually 8): about 150 mL in capacity, with a gas inlet tube extending directly to the inner bottom of the cell, and a ventilation hole at the top of the cell.

6.1.5 Electrodes (usually 8): They are used to measure conductivity, with a measuring range of 0 μS/cm~300 μS/cm, matched with the size of the measuring cell.

6.1.6 Measuring and recording apparatus: An amplifier and a recorder that records the measurement signals of each electrode are included.

NOTE: Oxidative stability testers of the Rancimat of Swiss Metrohm and the OSI of American Omnion use a computer-controlled central processing unit.

6.1.7 Certified and calibrated contact thermometer: The accuracy is 0.1 °C or Pt-100 element (platinum resistance thermometer) is used to measure the temperature of the heating block, connected with a control relay and a heating element; the temperature range is 0 °C ~150 °C.

6.1.8 Heating block: It is made of cast aluminum. The temperature can be adjusted to 150 °C±0.1 °C. There are round holes (usually 8) on the heating block for placing the aeration vessels (6.1.3) and an aperture for inserting the contact thermometer (6.1.7).

A heating bath can also be used, filled with thermostable oil, and the temperature is adjusted to 150 °C±0.1 °C through an oil bath.

The temperature can be kept constant at 150 ℃±3 ℃.

6.5 Connecting hoses

They are flexible and made of inert material (polytetrafluoroethylene or silicone rubber).

7 Sampling

Samples received by the laboratory shall be truly representative and have not been damaged or changed during transportation and storage.

Sampling is not the content stipulated in this standard, and ISO 5555 is recommended.

The samples shall be stored in the dark at 4 °C.

8 Preparation of samples and apparatus

8.1 Preparation of samples

Samples shall be prepared according to the provisions of GB/T 15687.

Use a pipette to remove the required sample volume from the center of the carefully homogenized sample.

Heat semi-solid or solid samples to a temperature slightly above their melting point, mix carefully, and avoid overheating. The pipette is also heated to the same temperature as the sample.

8.2 Preparation of apparatus

8.2.1 Cleaning procedures

In order to remove organic residues as much as possible, wash the aeration vessels, measuring cells, and air inlet and outlet tubes with acetone more than three times, and then rinse with tap water.

The cleanliness of the aeration vessels is paramount in achieving the correct induction period. All oxidized oil residues from previous measurements must be removed. Fill the vessels with laboratory alkaline glass-cleaning solution, mount the inlet tubes, and place them at 70 °C for more than 2 hours. Then, rinse the aeration vessels and the inlet and outlet tubes thoroughly with tap water, wash them with distilled water or deionized water, and dry them in an oven at 110 °C for more than 1 h.

NOTE: If disposable aeration vessels are used, the cleaning procedure described above is not necessary.

After temperature correction has been applied, the temperature in the reaction vessel will be equal to the target temperature for the measurement.

9 Analysis steps

9.1 Set up the apparatus as shown in Figure 1. If there is a ready-made apparatus available commercially, operate it according to the product manual.

9.2 Connect the gas diaphragm pump (6.1.2), adjust the flow rate to 10 L/h exactly, and then turn off the pump again. Some apparatus available commercially can automatically control the flow rate.

NOTE: When the OSI instrument is adjusted to a pressure of 0.038 MPa (5.5 psi), the flow rate is approximately 10 L/h.

9.3 Use the thyristor and contact thermometer (6.1.7) or an electronic controller to adjust the temperature of the heating block (6.1.8) to the desired value (usually 100 °C, or see 8.2.2); during the test, the temperature shall always be kept within the range of ±0.1 °C from the desired value.

If necessary, add some glycerol (5.4) to the round holes of the heating block (6.1.8) to facilitate heat transfer.

If the heating bath (6.1.8) is used for heating, heat it to the desired temperature, and check according to the method described in 8.2.2.

9.4 Use a pipette (6.3) to add 50 mL of distilled or deionized water into the measuring cell.

NOTE: When the temperature exceeds 20 °C, volatile carboxylic acids may evaporate from the water in the measuring cell, resulting in a decrease in the conductivity of the aqueous solution. The rapidly rising part of the conductivity curve will therefore produce a deviant shape, so it becomes impossible to determine the tangent on this part of the curve.

9.5 Check the electrodes (6.1.5) and adjust their signals with a calibrated potentiometer, so that they stay on the zero axis of the recording paper.

Set the paper rate at 10 mm/h and the measuring frequency at one measuring point per 20 seconds. The full scale is set at 200 μS/cm.

If the paper rate cannot be adjusted to 10 mm/h, it can be adjusted to 20 mm/h, and the paper rate shall be indicated on the recording paper.

NOTE: Commercially available apparatus can obtain measuring data through a computer.

Commercially available instruments automatically calculate the induction period from the maximum value of the second derivative of the curve. (See Figure A.1 of Appendix A)

The oxidative stability is expressed in hours (h), and the reading is accurate to 0.1 h.

NOTE: The conductivity curves are shown in Figure A.1. A curve that rises very rapidly can be the result of the temperature of the solution in the measuring cell being too high, causing volatile carboxylic acid to evaporate from the solution.

11 Precision

11.1 Results of interlaboratory test

Appendix B summarizes the details of the interlaboratory test on the precision of this method. For other concentration ranges and test substances, the results of this interlaboratory test may not be applicable.

11.2 Repeatability

In the same laboratory, the same operator uses the same equipment, and according to the same test method, does two independent tests on the same experimental sample in a short period of time. When the oxidative stability is between 2 h and 45 h, the probability that the absolute difference between the two independent test results exceeds 6% of the arithmetic mean of the two test results shall be less than 5%.

11.3 Reproducibility

In different laboratories, different operators use different equipment and the same test method to test the same experimental sample. When the oxidative stability is between 2 h and 45 h, the probability that the absolute difference between the two independent test results exceeds 29% of the arithmetic mean of the two test results shall be less than 5%.

12 Test report

The test report shall specify:

-- all information required for the complete identification of the sample;

-- the sampling method used (if known) with reference to this standard;

-- the test method used with reference to this standard;

Appendix A

(Informative)

Summary of the method and examples of conductivity curves and the determination of induction time

In recent years, many methods for measuring the oxidative stability of oils and fats have been developed. These methods are based on the rate of oxygen absorption of oils and fats (in a liquid state) when exposed to air.

Oxygen absorption can be measured directly by a Warburg apparatus or indirectly by the determination of peroxides or decomposition products during oxidation.

Among the indirect methods of determination, the active oxygen method (AOM) is the oldest method. It is based on the determination of the peroxide value and the time required for the peroxide value to reach 100 mmol (active oxygen per 2 kg of sample) according to the aeration treatment of the sample at 98.7 °C. The accelerated stability test is based on this method. These determinations are very time-consuming and cannot be automated.

In the method described in this standard, the oxidation process is divided into two phases:

a) the first phase (the induction period) that is characterized by the slow absorption of oxygen during which peroxides are formed;

b) the second phase (tainted odour and flavour generation phase) that is characterized by the rapid absorption of oxygen in which peroxides are not only formed but these peroxides are then dissociated under the influence of the high temperature. During this phase, products such as aldehydes, ketones, and lower fatty acids are formed. These products cause the appearance of abnormal odour.

The method described in this standard is to measure the conductivity of the decomposition products of volatile acids (mainly formic acid and acetic acid) produced during oxidation.

The method was published in 1974, and the automatic potentiometric determination method was published in 1972.

The induction time measured from the conductivity curve is consistent with the induction time obtained by the active oxygen method (AOM) at the same temperature.

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