INTERNATIONAL STANDARD

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Animal and vegetable fats and oils — Determination of oxidative stability (accelerated oxidation test)

Corps gras d'origines animale et végétale — Détermination de la stabilité à l'oxydation (essai d'oxydation accéléré)

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 6886 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

This second edition cancels and replaces the first edition (ISO 6886:1996), which has been technically revised.

Animal and vegetable fats and oils — Determination of oxidative stability (accelerated oxidation test)

1 Scope

This International Standard specifies a method for the determination of the oxidative stability of fats and oils under extreme conditions that induce rapid oxidation: high temperature and high air flow. It does not allow determination of the stability of fats and oils at ambient temperatures, but it does allow a comparison of the efficacy of antioxidants added to fats and oils.

The method is applicable to both virgin and refined animal and vegetable fats and oils.

NOTE The presence of volatile fatty acids and volatile acidic oxidation products prevents accurate measurement.

2 Normative references

The following referenced document is indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 661:2003, *Animal and vegetable fats and oils — Preparation of test sample*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
induction period
time between the start of the measurement and the time when the formation of oxidation products rapidly begins to increase

3.2
oxidative stability
induction period, expressed in hours, determined according to the procedure specified in this International Standard

NOTE A temperature of 100 °C to 120 °C is usually applied for the determination of oxidative stability. Depending on the oxidative stability of the sample under test, or when an extrapolation of regression is required, the determination may be carried out at other temperatures. The optimal induction period is 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
ability of a material 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 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. The end of the induction period is indicated when the conductivity begins to increase rapidly. This accelerated increase is caused by the accumulation of volatile fatty acids produced during oxidation.

5 Reagents and materials

Use only reagents of recognized analytical grade, and distilled or demineralized water.

5.1 Molecular sieve, beads of approx. 1 mm diameter, pore size 0,3 nm, with moisture indicator.

The molecular sieve should be dried in an oven set at 150 °C and then cooled down to room temperature in a desiccator.

5.2 Acetone.

5.3 Alkaline cleaning solution, for laboratory glassware.

5.4 Glycerol.

5.5 Thermostable oil.

6 Apparatus

Usual laboratory equipment and, in particular, the following.

6.1 Appliance for the determination of oxidative stability

See Figures 1 and 2 for diagrammatic representations.

NOTE An appliance for determining oxidative stability can be obtained commercially under the trade name Rancimat, from Methrom-Herisau AG, Switzerland, or the OSI equipment from Omnion Inc., USA[1)].

6.1.1 Air filter, comprising a tube fitted with filter paper at each end and filled with molecular sieve (5.1), connected to the suction end of a pump.

6.1.2 Gas diaphragm pump, with an adjustable flow rate of 10 l/h, in combination with an apparatus to control the flow rate, manually or automatically, with a maximum deviation of ± 1,0 l/h from the set value.

NOTE For the OSI instrument, a pressure of 5,5 psi is equivalent to a flow of approximately 10 l/h.

6.1.3 Aeration vessels of borosilicate glass (usually eight), connected to a sealing cap.

The sealing cap shall be fitted with a gas inlet and outlet tube. The cylindrical part of the vessel shall preferably be narrower by a few centimetres below the top in order to break up any emerging foam. An artificial foam blocker (e.g. glass ring) may also be used for this purpose.

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1) Rancimat (www.metrohm.com) and OSI (Omnion) (http://world.std.com/~omnion/) are examples of suitable equipment available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this equipment.

6.1.4 Closed measurement cells (usually eight), of approx. 150 ml capacity, with a gas inlet tube extending to the bottom inside of the vessel.

The cell shall be provided at the top with ventilation holes.

6.1.5 Electrodes (usually eight), for measuring conductivity with a measuring range of 0 µS/cm to 300 µS/cm, aligned with the dimensions of the measurement cell (6.1.4).

6.1.6 Measuring and recording apparatus, comprising an amplifier and a recorder for registering the measuring signal of each of the electrodes (6.1.5).

NOTE A computer-controlled central processing unit is used with Rancimat and OSI (Omnion).

6.1.7 Certified and calibrated contact thermometer, graduated in 0,1 °C, or **Pt 100 element (platinum resistance thermometer)** to measure the block temperature, with attachments for a control relay connection and an adjustable heating element; temperature range 0 °C to 150 °C.

6.1.8 Heating block, made of cast aluminium, adjustable to a temperature of up to 150 °C ± 0,1 °C.

The block shall be provided with holes (usually eight) for the aeration vessels (6.1.3), and an aperture for the contact thermometer (6.1.7).

Alternatively, a **heating bath** may be used, filled with oil, suitable for temperatures up to 150 °C and adjustable to the nearest 0,1 °C.

6.2 Certified and calibrated thermometer or **Pt100** element, with a temperature range up to 150 °C, graduated in 0,1 °C.

6.3 Measuring pipettes, of capacity 50 ml and 5 ml.

6.4 Oven, capable of being maintained at a temperature of up to 150 °C ± 3 °C.

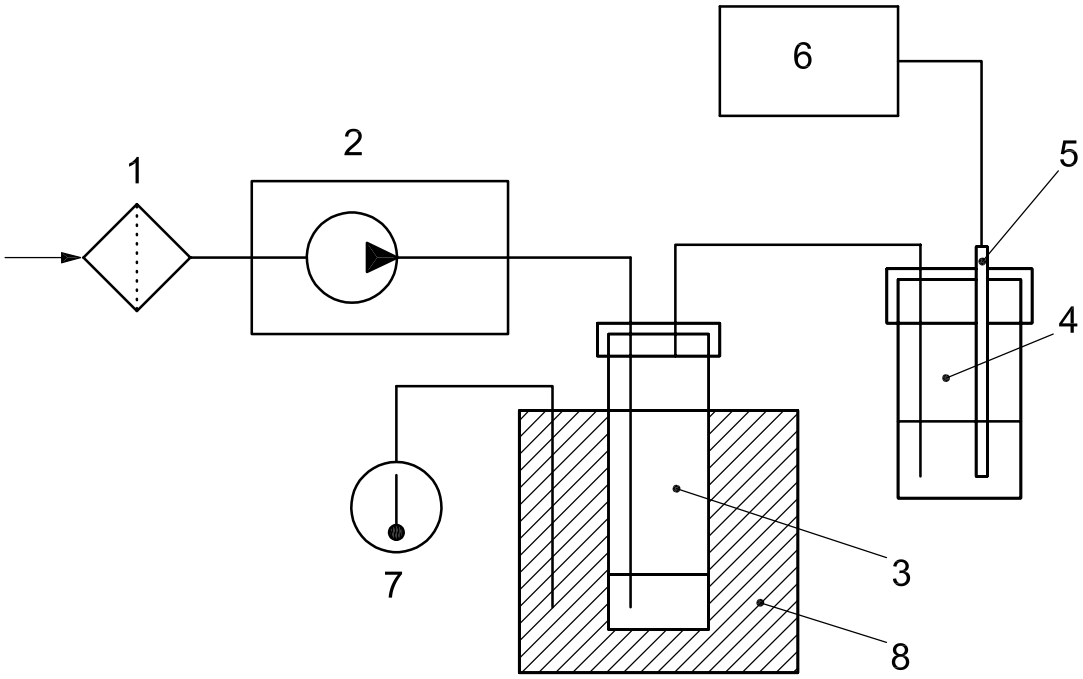
6.5 Connecting hoses, flexible and made of inert material [polytetrafluoroethylene (Teflon) or silicone].

7 Sampling

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 5555 [1].

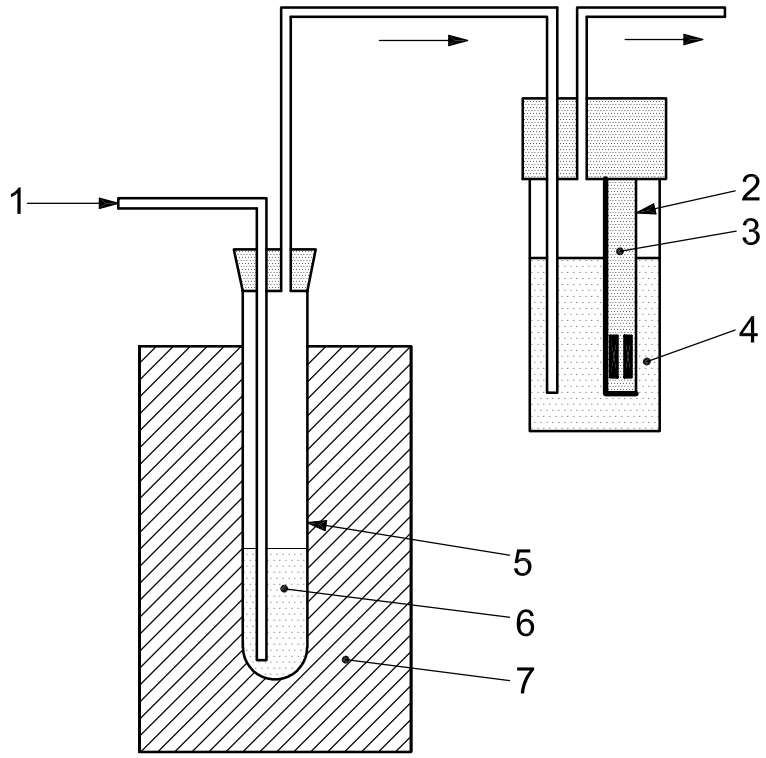
Store the sample in the dark at about 4 °C.



Key

1 air filter (6.1.1)
2 gas diaphragm pump with flow rate control (6.1.2)
3 aeration vessel (6.1.3)
4 measurement cell (6.1.4)
5 electrode (6.1.5)
6 measuring and recording apparatus (6.1.6)
7 thyristor and contact thermometer (6.1.7)
8 heating block (6.1.8)

Figure 1 — Diagrammatic representation of the apparatus



Key

1	air	5	aeration vessel
2	measurement cell	6	sample
3	electrode	7	heating block
4	measuring solution		

Figure 2 — Diagrammatic representation of heating block, reaction vessel and measurement cell

8 Preparation of test sample and apparatus

8.1 Preparation of test sample

Prepare the test sample in accordance with ISO 661.

Remove the required quantity from the centre of the carefully homogenized sample using a pipette.

Heat semisolid and solid samples to a temperature somewhat above their melting points and mix carefully. Overheating should be avoided. The pipette shall be at the same temperature as the sample.

8.2 Preparation of apparatus

8.2.1 Cleaning procedure

Wash the aeration vessels, measurement cells and their inlet and outlet tubes at least three times with acetone in order to remove as much of the organic residue as possible. Rinse with tap water.

The cleanliness of the aeration vessels is paramount in achieving correct induction periods. All traces of oxidized oils from previous runs shall be removed.

Fill the vessels completely with an aqueous alkaline laboratory glass-cleaning solution and mount the inlet tubes. Store the vessels for at least 2 h at 70 °C.