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

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

ISO 6886:1996

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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.

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.

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

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Annexes A and B of this International Standard are for information only.

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

1 Scope

This International Standard specifies a method for the determination of the oxidation stability of fats and oils. It is applicable to refined animal and vegetable fats and oils.

NOTE 1 The presence of volatile fatty acids and volatile acidic oxidation products makes accurate measurement impossible.

2 Definitions

For the purposes of this International Standard, the following definitions apply.

2.1 induction period: Time which passes between the moment when the sample reaches the desired temperature and the moment when the formation of oxidation products rapidly begins to increase.

2.2 oxidation stability: Induction period determined according to the procedure specified in this International Standard. Oxidation stability is expressed in hours.

NOTE 2 A temperature of 100 °C is usually applied in the determination of oxidation stability. Depending on the oxidation stability of the sample under test, the determination may be carried out at a higher temperature, for example 110 °C. The temperature should be chosen so that an induction period of at least 5 h and at most 10 h is obtained.

3 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 which has been demineralized or distilled and contains an electrode for measuring the conductivity. The electrode is connected to a measuring and recording device. It indicates the end of the induction period when the conductivity begins to increase rapidly. This accelerated increase is caused by the dissociation of volatile carboxylic acids produced during the oxidation process and absorbed in the water.

4 Reagents and materials

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

4.1 Molecular sieve, with moisture indicator, 2 mbar, pore size 0,3 mm. The molecular sieve should be dried in an oven set at 150 °C and cooled down to room temperature in a siccative.

4.2 Potassium dichromate solution (optional), 20 g/l in 1 % sulfuric acid (V/V).

4.3 Petroleum ether, boiling range 40 °C to 60 °C, or **acetone**.

4.4 Cleaning agent, i.e. non-alkaline detergent with strong fat removal qualities.

NOTE 3 Dodecyl benzene sulfonate meets these requirements.

4.4.1 Cleaning solution A, for aeration vessels and connecting pipes, prepared from 100 g of cleaning agent (4.4) in 1 litre of water.

4.4.2 Cleaning solution B, for measurement cells, prepared from 20 g of cleaning agent (4.4) in 1 litre of water.

4.5 Glycerol

5 Apparatus

Usual laboratory equipment and, in particular, the following.

5.1 Appliance for the determination of oxidation stability

See figures 1 and 2 for diagrammatic representations.

NOTE 4 An appliance for determining oxidation stability can be obtained commercially under the tradename Rancimat, model 679, from Methrom-Herisau AG, Switzerland¹⁾.

5.1.1 Air filter, comprising a tube fitted with filter paper at the ends and filled with a molecular sieve (4.1), connected to the suction end of a pump.

5.1.2 Gas diaphragm pump, with an adjustable flowrate of between 3 l/h and 30 l/h and a maximum deviation of $\pm 0,03$ l/h from the set value.

5.1.3 Needle valve

5.1.4 Wash bottles (four), of capacity 250 ml, connected as shown in figure 1.

Wash bottle A shall be empty. Wash bottles B, C and D shall be filled with 150 ml potassium dichromate solution (4.2), 150 ml water and cotton wool, respectively, together with the molecular sieve (4.1).

NOTE 5 Rancimat 679 uses a molecular sieve prior to purification of the pumped air, and wash bottles are not used. The application of wash bottles is, however, strongly recommended.

5.1.5 Air-relief cock

5.1.6 Air circulator at each of the circulation points (usually six), fitted with a capillary tube of 5 mm external diameter, 0,6 mm internal diameter and 60 mm length.

5.1.7 Flowmeters (usually six), measuring range 0 l/h to 20 l/h, for connection to the air circulator (5.1.6).

5.1.8 Cylindrical aeration vessels of borosilicate glass (usually six), of 25 mm external diameter and 200 mm height, connected to a sealing cap by means of a conical joint.

The sealing cap shall be fitted with a gas inlet and outlet tube with a 13/5 spherical joint. The cylindrical part of the vessel shall preferably be narrower a few centimetres below the top in order to break any emerging foam. An artificial glass ring may also be used for this purpose.

5.1.9 Measurement cells (usually six), of approx. 150 ml capacity, total height approx. 120 mm and external diameter approx. 56 mm, with a conical neck and gas inlet tube extending to the bottom inside of the vessel and fitted with a 13/5 spherical joint outside. The bottle shall be provided at the top with ventilation holes having a diameter of approx. 2 mm.

5.1.10 Electrodes (usually six), for measuring conductivity, comprising double platinum electrodes with a measuring range of 0 μ S/cm to 300 μ S/cm, fitted with a 14/15 conical joint and aligned with the dimensions of the measurement cell (5.1.9).

5.1.11 Measuring and recording apparatus, comprising

- switches for connecting each electrode (5.1.10) to a calibration potentiometer for setting the measuring signal at zero;
- an amplifier;
- a recorder for registering the measuring signal of each of the electrodes (5.1.10).

5.1.12 Thyristor and contact thermometer, with 150 mm insertion depth and attachments for relay connection and an adjustable heating element; temperature scale 0 °C to 150 °C, graduated in 0,2 °C.

5.1.13 Heating block, made of cast aluminium, of 200 mm height, adjustable to a temperature of $150 \text{ °C} \pm 0,2 \text{ °C}$. The block shall be provided with holes (usually six) for the aeration vessels (5.1.8), of 27 mm diameter and 140 mm depth, and an aperture for the contact thermometer (5.1.12) of 10 mm diameter and 140 mm depth.

Alternatively, a **heating bath** may be used, filled with oil suitable for temperatures up to 150 °C and adjustable to the nearest 0,2 °C.

5.2 Thermometer, with a temperature scale of 70 °C to 150 °C, graduated in 0,2 °C.

5.3 Measuring pipettes (two), of capacity 50 ml.

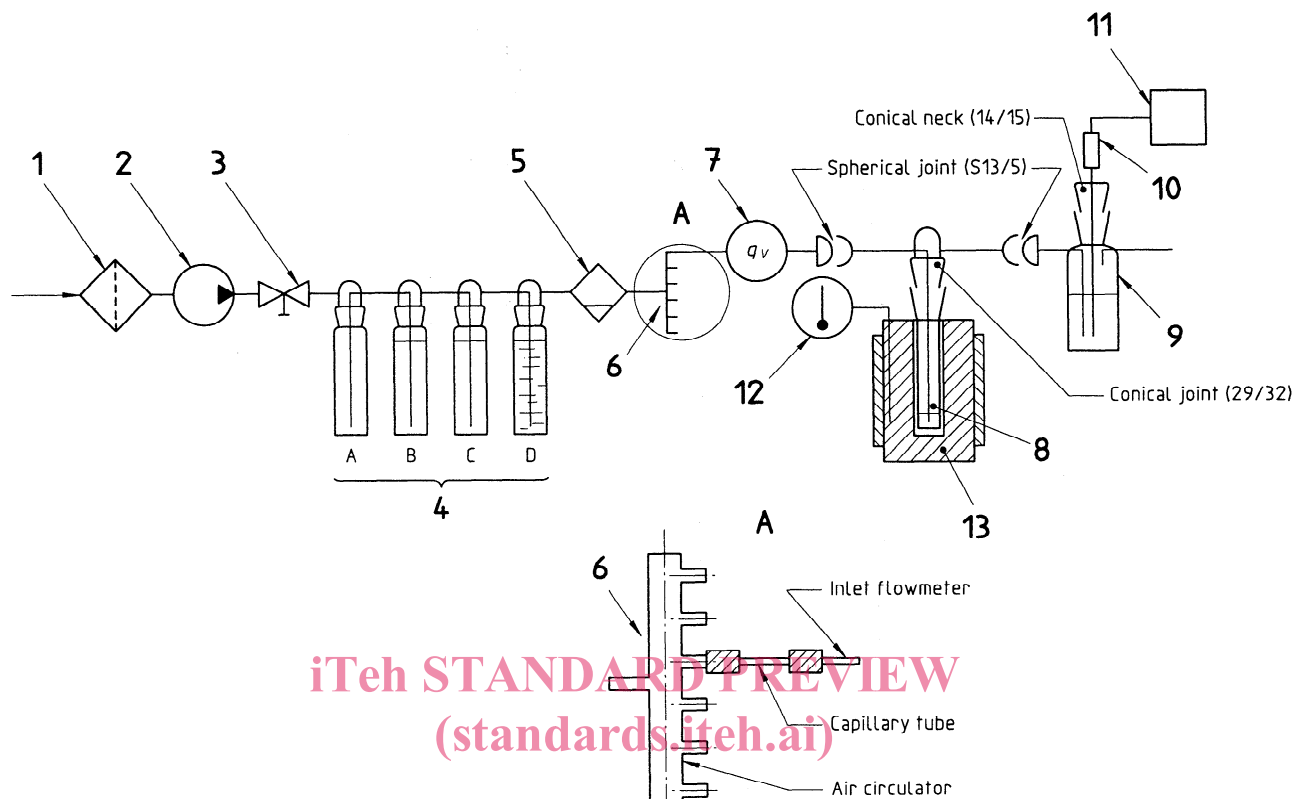
5.4 Oven, capable of being maintained at $150 \text{ °C} \pm 3 \text{ °C}$.

5.5 Oven, capable of being maintained at 500 °C (optional).

5.6 Cooling bath, capable of being maintained at a steady temperature of approx. 15 °C, using tap water for example.

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

1) Rancimat, model 679, is an example 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.



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Key

- 1 Air filter (5.1.1)
- 2 Gas diaphragm pump (5.1.2)
- 3 Needle valve (5.1.3)
- 4 Wash bottles A, B, C, D (5.1.4)
- 5 Air-relief cock (5.1.5)
- 6 Air circulator (5.1.6)
- 7 Flowmeter (5.1.7)
- 8 Aeration vessel (5.1.8)
- 9 Measurement cell (5.1.9)
- 10 Electrode (5.1.10)
- 11 Measuring and recording apparatus (5.1.11)
- 12 Thyristor and contact thermometer (5.1.12)
- 13 Heating block (5.1.13)

Figure 1 — Diagrammatic representation of the apparatus

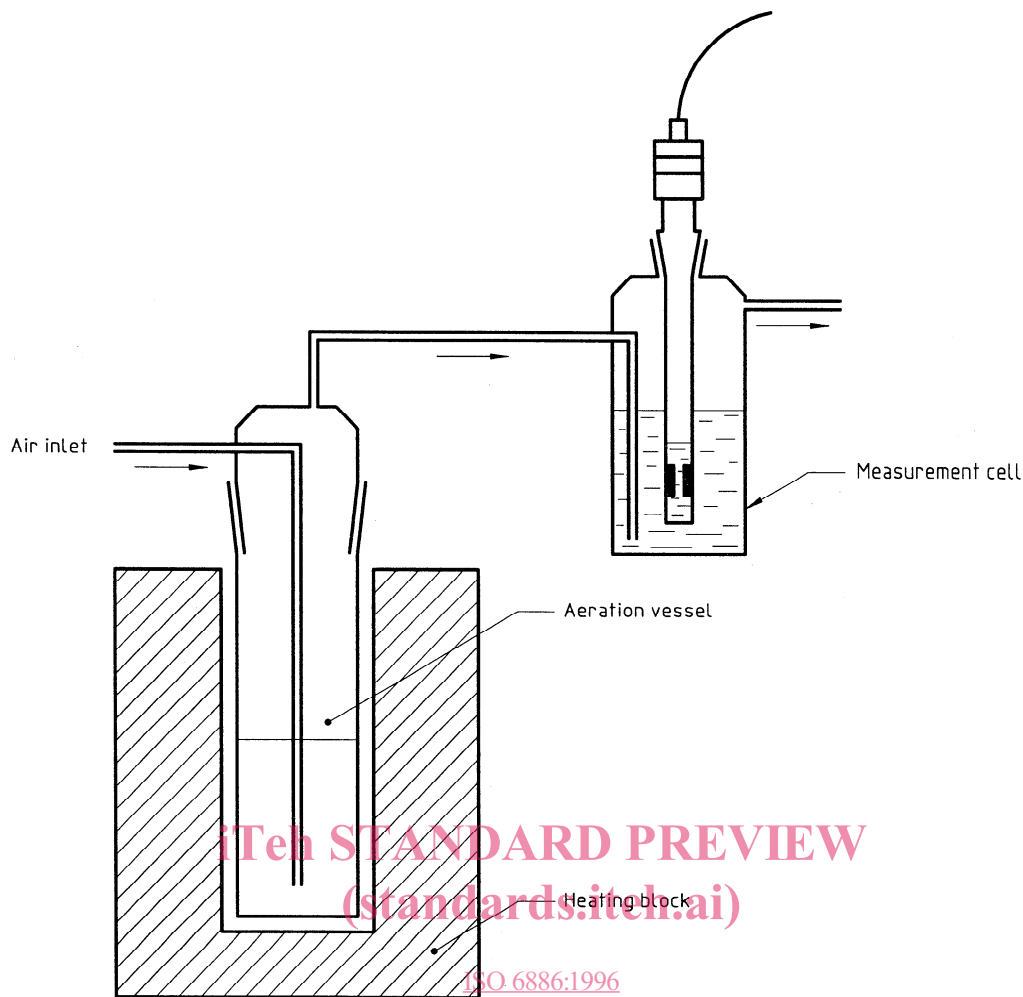


Figure 2 — Diagrammatic representation of heating block, reaction vessel and measurement cell

6 Sampling

It is important that the laboratory receive a sample which is truly representative and has not been damaged or changed during transport and 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.

7 Preparation of test sample

In order to prevent the preparation of the test sample from influencing the test result, all handling of the laboratory sample shall be restricted to the steps given in 7.1. and 7.2.

7.1 Light oils

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

7.2 Semi-solid and solid fats

Heat carefully until the fat just becomes fluid. Homogenize using a stirrer without beating air in and then remove the required quantity from the centre of the sample using a pipette heated to the same temperature.

8 Procedure

8.1 Wash the aeration vessels (5.1.8), measurement cells (5.1.9) and their inlet and outlet tubes with petroleum ether (4.3) in order to remove as much of the organic residue as possible. Rinse with hot water.

Heat the aeration vessels (5.1.8) and their inlet and outlet tubes for 3 h in the cleaning solution A (4.4.1) at approx. 90 °C.

Soak the measurement cells (5.1.9) for 24 h in the cleaning solution B (4.4.2). Rinse the purified vessels and their inlet and outlet tubes thoroughly with tap water and finally with demineralized or distilled water.

Dry them in the oven (5.4).

NOTE 6 If there is an oven (5.5) available which can be adjusted to 500 °C, the following cleaning procedure can be used: rinse three times with acetone and leave them overnight in the oven set at 500 °C.

8.2 Set up the apparatus as shown in figure 1. If the apparatus is available commercially, follow the manufacturer's instructions.

8.3 Attach the gas diaphragm pump (5.1.2) and adjust the flow to exactly 20 l/h using the needle valve (5.1.3) and flowmeters (5.1.7). Then switch the pump off again.

8.4 Bring the heating block (5.1.13) up to the desired temperature (usually 100 °C, but see note 2) using the thyristor and contact thermometer (5.1.12). The temperature shall be maintained constant to within $\pm 0,2$ °C during the test period.

Pour some glycerol (4.5) into the holes of the heating block in order to promote heat transfer.

Fill an aeration vessel (5.1.8) with 10 g of a thermostable oil and place the vessel without the sealing cap in one of the holes in the block. Place the thermometer (5.2) in the oil and check that the block is at the desired temperature.

It is preferable that the temperature measurement be continued in this way during the test, even if this means that only five samples can be measured at the same time.

If a heating bath (5.1.13) is used, bring it to the desired temperature and check in the manner described.

8.5 Fill the measurement cells (5.1.9) with 50 ml of water using a measuring pipette (5.3). Place the vessels in the cooling bath (5.6) if the oxidation stability of the sample exceeds 10 h.

NOTE 7 At temperatures above 20 °C, volatile carboxylic acids may evaporate from the water in the measurement cell. This can lead to 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 (see reference [2]).

8.6 Check the electrodes and adjust their signals using a calibration potentiometer so that they are on the zero axis of the recorder paper.

Set the paper rate at 10 mm/h and the measuring frequency at one measuring point per 20 s. Set the measuring value of 300 $\mu\text{S}/\text{cm}$ at the maximum result of 100 %.

If it is not possible to adjust the paper rate to 10 mm/h, but 20 mm/h, this should be reported on the recorder paper.

8.7 Using a pipette (5.3), weigh, to the nearest 0,01 g, 2,5 g of the conditioned sample (see clause 7) into an aeration vessel (5.1.8). Take care that the sample does not come into contact with the sides of the aeration vessel.

If the oxidation stability of several samples is to be determined, weigh the samples in succession and store the aeration vessels containing the weighed samples under conditions in which oxidation is prevented as far as possible (e.g. at temperatures below -10 °C) until the procedure can be carried out on each sample simultaneously.

8.8 Place the aeration vessel with the sealing cap (5.1.8) into the hole intended for it in the heating block or into the heating bath (5.1.13), both of which shall have reached the required temperature.

First close the gas outlet tube on the measurement cell (5.1.9) and then the gas inlet tube on the flowmeter (5.1.7). Allow the contents of the vessels to reach the desired temperature over a period of 10 min.

8.9 Switch on the gas diaphragm pump (5.1.2) and set the flow again at exactly 20 l/h using the needle valve of the flowmeter.

Note the time that measurements were begun on the recorder paper.

8.10 Finish the measurements when the signal has reached 100 % of the recorder scale.

8.11 During the determination, carry out the following precautions:

- a) check the setting of the flowmeter and adjust where necessary in order to ensure a constant flow;
- b) check the colour of the molecular sieve (4.1) of the air filter and replace it as necessary during measurements as soon as the colour starts to turn reddish; interrupt the air flow for a short time;
- c) in the case of overnight operation of the equipment, the wash bottle and the molecular sieve shall be changed prior to each run, but it is better to exchange the wash bottles and the molecular sieve prior to each run;
- d) check the potassium dichromate solution (4.2) in a similar way and replace it as necessary.