

Methods of analysis of Fats and fatty oils —

Part 2: Other methods —

Section 2.25 Determination of oxidative stability (accelerated oxidation test)

IMPORTANT NOTICE: It is essential that BS 684-0.1 and BS 684-0.2 (BS EN ISO 661), which are published separately, be read in conjunction with this Section.

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Committees responsible for this British Standard

The preparation of this British Standard was entrusted to Technical Committee AW/11, Animal and vegetable fats and oils, upon which the following bodies were represented:

- Association of Public Analysts
- Department of Trade and Industry (Laboratory of the Government Chemist)
- Feed Fat Association
- FOSFA International
- International Federation of Inspection Agencies
- Lard Association
- Leatherhead Food Research Association
- Margarine and Shortening Manufacturers' Association
- Ministry of Agriculture, Fisheries and Food
- National Edible Oil Distributors' Association
- Seed Crushers' and Oil Processors' Association
- Soap and Detergent Industry Association
- Society of Chemical Industry
- Tropical Growers' Association
- United Kingdom Renderers' Association Ltd.

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National foreword

This Section of BS 684 has been prepared by Technical Committee AW/11 and is identical with ISO 6886:1996 *Animal and vegetable fats and oils — Determination of oxidation stability (Accelerated oxidation test)*, published by the International Organization for Standardization (ISO) and in the preparation of which the United Kingdom played a full part. It supersedes BS 684-2.25:1979 which was withdrawn in 1985, and from which it differs in that this edition has been technically updated.

Cross-references

Publication referred to	Corresponding British Standard
Informative	
ISO 5555:1991	BS EN ISO 5555:1995 <i>Animal and vegetable fats and oils. Sampling</i> (Identical)
ISO 5725:1986	BS 5497 <i>Precision of test methods</i> Part 1:1987 <i>Guide for the determination of repeatability and reproducibility for a standard test method by inter-laboratory tests</i> (Identical)

Additional information. ISO 5725:1986, to which informative reference is made in the text, has been superseded by ISO 5725-1:1994, ISO 5725-2:1994, ISO 5725-3:1994, ISO 5725-4:1994 and ISO 5725-6:1994 which are identical with the following Parts of BS ISO 5725 *Accuracy (trueness and precision) of measurement methods and results*; BS ISO 5725-1:1994 *General principles and definitions*; BS ISO 5725-2:1994 *Basic methods for the determination of repeatability and reproducibility of a standard measurement method*; BS ISO 5725-3:1994 *Intermediate measures of the precision of a standard measurement method*; BS ISO 5725-4:1994 *Basic methods for the determination of the trueness of a standard measurement method*; and BS ISO 5725-6:1994 *Use in practice of accuracy values*

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 10, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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 Figure 1 and Figure 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).

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.