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Animal and vegetable fats and oils — Determination of Bömer value

Corps gras d'origines animale et végétale — Détermination de l'indice de Bömer

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 3577 was prepared by Technical Committee ISO/TC 34, *Agricultural food products.*

This second edition cancels and replaces the first edition (ISO 3577 : 1976), of which it constitutes a technical revision.

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Animal and vegetable fats and oils — Determination of Bömer value

1 Scope

This International Standard specifies two methods for the determination of the Bömer value of pork fat as follows:

a) the diethyl ether method (method I);

b) the acetone method (method II).

The Bömer value is intended to give an indication of the presence of foreign fat in pork fat. The foreign fat may be hydrogenated and/or interesterified pork fat.

NOTE — Comparative tests have shown that both methods give practically identical results. The diethyl ether method should be used by preference, however, because it has a smaller standard deviation. Climatic or legal considerations may make it necessary to use the acetone method in order to avoid the use of diethyl ether.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 661 : 1980, *Animal and vegetable fats and oils — Preparation of test sample.*

ISO 5555 : 1983, *Animal and vegetable fats and oils — Sampling.*

3 Definition

For the purposes of this International Standard, the following definition applies.

Bömer value: The sum, in degrees Celsius, of the melting point of the triglycerides isolated by the procedure described in this International Standard and twice the difference between this melting point and that of the fatty acids obtained after saponification of the triglycerides.

4 Principle

Crystallization of the fat sample in diethyl ether or acetone, according to the method used, in order to obtain the saturated triglycerides.

Elimination of the non-saturated adhering triglycerides, either

a) by filtration and thorough washing with diethyl ether followed by recrystallization from diethyl ether (method I), or

b) by centrifuging or decanting followed by thorough washing with acetone (method II).

Saponification of a part of the saturated triglycerides with an ethanolic potassium hydroxide solution. Acidification of the soap solution with dilute hydrochloric acid and extraction of the fatty acids with diethyl ether (method I) or *n*-hexane (method II). Washing of the fatty acids solution with water until it is free from hydrochloric acid, then evaporation of the solvent.

Simultaneous determination of the melting points of the dry triglycerides and the dry fatty acids.

5 Reagents

All reagents shall be of recognized analytical grade and the water used shall be distilled water or water of equivalent purity.

5.1 Diethyl ether, free from peroxides and freshly distilled (for method I).

5.2 Acetone (for method II).

5.3 *n*-Hexane (for method II).

5.4 Ethanol, 95 % (*V*/*V*) to 96 % (*V*/*V*).

5.5 Sodium sulfate, anhydrous.

5.6 Filter aid.

5.7 Potassium hydroxide, pellets.

5.8 Hydrochloric acid, approximately 1 mol/l solution.

5.9 Methyl orange, 5 g/l solution.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Water-bath, capable of being controlled at a temperature of 15 °C ± 1 °C (for method I) or of 30 °C ± 2 °C (for method II).

6.2 Conical flask, of 100 ml capacity, with a ground neck, furnished with a reflux condenser.

6.3 Mortar, of about 50 ml capacity, preferably made of agate.

6.4 Glass capillary tubes, sealed at one end, of internal diameter 0,8 mm to 1,0 mm, wall thickness 0,1 mm to 0,2 mm and length 70 mm to 80 mm.

6.5 Thermometer, readable to 0,1 °C in the range of melting points expected (usually 30 °C to 60 °C).

6.6 Melting point apparatus, suitable for use with the capillary tubes (6.4).

6.7 Desiccator, containing an efficient desiccant.

6.8 Centrifuge tube, of 100 ml capacity, or cylinder, of 100 ml capacity (for method II), equipped with a glass stopper.

6.9 Büchner funnel, of diameter appropriate for the amount of crystals to be filtered.

6.10 Boiling water-bath.

6.11 Oven, capable of being controlled at 103 °C ± 2 °C.

6.12 Separating funnel, of 250 ml capacity.

7 Sampling

Sampling shall be carried out in accordance with ISO 5555.

8 Preparation of the test sample

Prepare the test sample in accordance with ISO 661.

9 Procedure

9.1 Method I

9.1.1 Test portion

Weigh about 50 g of the test sample (clause 8) into a 150 ml beaker.

9.1.2 Preparation of triglycerides

9.1.2.1 Add to the test portion 50 ml of the diethyl ether (5.1), cover the beaker with a watch-glass and dissolve the fat by swirling and gentle heating over a water-bath. Filter the solution through a dry filter paper if it is not perfectly clear.

Place the covered beaker in the water-bath (6.1) at 15 °C ± 1 °C and leave it for 1 h, swirling the beaker or stirring the solution with a glass rod at least every 15 min.

If no crystals are obtained after 30 min, continue crystallization for another 30 min at a lower temperature, but not below 5 °C.

Filter off the crystals, if necessary under slight suction, on the Büchner funnel (6.9) using a well-fitted fast filter paper.

Wash the crystals on the filter paper three times with 25 ml portions of the diethyl ether (5.1) cooled to 15 °C.

If the amount of crystals obtained either at 15 °C or at a lower temperature is less than approximately 0,5 g, repeat the procedure with further test portions and combine the crystals obtained.

9.1.2.2 Detach the crystals from the filter paper and return them to the beaker which was used for the crystallization, after first rinsing the beaker with some diethyl ether.

Add 50 ml of the diethyl ether, cover the beaker with a watch-glass, and dissolve the crystals by gentle heating over a water-bath; cool and place the beaker in the water-bath (6.1) at 15 °C ± 1 °C for 15 min.

Filter off the crystals, if necessary under slight suction, on the Büchner funnel using a well-fitted fast filter paper. Wash the crystals on the filter paper three times with 25 ml portions of the diethyl ether cooled to 15 °C.

9.1.2.3 Repeat the procedure in 9.1.2.2.

9.1.2.4 Place the filter paper and crystals on a watch-glass and dry at a temperature not exceeding 35 °C.

Grind the crystals in the mortar (6.3) to a fine, smooth, powder. Place the powdered crystals of triglycerides in an open bottle in the desiccator (6.7).

Continue according to 9.3.

9.2 Method II

9.2.1 Test portion

Weigh about 20 g of the test sample (clause 8) into a centrifuge tube or cylinder (6.8).

9.2.2 Preparation of triglycerides

9.2.2.1 Add to the test portion 80 ml of the acetone (5.2) at 30 °C and shake until thoroughly mixed. Allow to stand in the water-bath (6.1) at 30 °C ± 2 °C for about 18 h.

9.2.2.2 Isolate the crystals either by centrifuging (for 5 min) and pouring off the supernatant liquid, or by siphoning off the supernatant liquid if the cylinder is used.

If less than approximately 0,5 g of crystals is obtained, repeat the procedure with further test portions and combine the crystals obtained.

9.2.2.3 Add 20 ml of the acetone (5.2) at 30 °C. Shake and isolate the crystals as described in 9.2.2.2.

9.2.2.4 Again add 20 ml of the acetone at 30 °C to the crystals, mix well, and filter off the crystals under slight suction on the Büchner funnel (6.9) using a well-fitted fast filter paper.

Wash the crystals on the filter paper five times with 5 ml portions of the acetone at 30 °C, carrying out the final wash under suction.

9.2.2.5 Place the filter paper and crystals on a watch-glass and dry at a temperature not exceeding 35 °C.

Transfer the crystals to the mortar (6.3), break up any lumps and allow to dry thoroughly. Continue drying for 15 min.

Grind the crystals to a fine, smooth, powder. Place the powdered crystals of triglycerides in an open bottle in the desiccator (6.7).

Continue according to 9.3.

9.3 Preparation of fatty acids

WARNING — It is essential that the fatty acids be prepared in an atmosphere free from ammonia (NH_3) in order to prevent formation of ammonium salts which affect the melting point of the acids.

9.3.1 Remove a sufficient amount of the powdered triglycerides (9.1.2.4 or 9.2.2.5) for the determination of the melting point.

Weigh not more than 0,2 g of the remainder into the conical flask (6.2) and add 10 ml of the ethanol (5.4) and 0,4 g of the potassium hydroxide (5.7).

9.3.2 Connect the flask to the reflux condenser, heat on the boiling water-bath (6.10) and keep gently boiling for 15 min.

Detach the condenser and pour the soap solution into a 100 ml beaker. Place the beaker on the boiling water-bath for 15 min to remove the greater part of the ethanol by evaporation.

9.3.3 Add 50 ml of water at about 75 °C to dissolve the soap, then transfer the solution to a 250 ml separating funnel (6.12).

NOTE — Do not use grease on the stopcock or the stopper.

Add 10 ml of the hydrochloric acid solution (5.8), mix well and leave to cool to room temperature.

Add 50 ml of the diethyl ether (5.1) (method I) or the *n*-hexane (5.3) (method II) and shake. Allow the layers to separate, then drain off the lower layer.

9.3.4 Wash the solution of the fatty acids at least three times with 15 ml portions of water, continuing if necessary, until the washings are neutral to methyl orange (5.9). After the last washing, drain off the lower layer completely.

Filter the solution through a dry filter paper, containing approximately 4 g of anhydrous sodium sulfate (5.5), into a 100 ml beaker.

9.3.5 Remove the solvent by evaporation over a water-bath.

Place the beaker in the oven (6.11) maintained at 103 °C ± 2 °C and leave for 15 min to 20 min.

Remove the beaker from the oven and allow to cool, keeping it in an inclined position.

9.3.6 Detach the fatty acid cake and pulverize it in the mortar (6.3) to a fine powder.

Store the powdered fatty acids in an open bottle in the desiccator (6.7).

9.4 Determination of melting points

9.4.1 Fill a capillary tube (6.4) with the powdered triglycerides (9.1.2.4 or 9.2.2.5) set aside in 9.3.1. Pack the powder to a height of approximately 5 mm, for instance by dropping the capillary tube into a glass tube containing the powdered triglycerides from a sufficient height onto a wooden base.

The powdered triglycerides contained in the capillary tube shall not be melted before the determination of melting points.

9.4.2 Fill another capillary tube, in the same way as described in 9.4.1, with the powdered fatty acids (9.3.6), as soon as possible after their preparation. Melt the contents of the tube for 5 min at about 70 °C and allow to solidify by cooling.

9.4.3 Place both of the capillary tubes and the thermometer (6.5) in the melting point apparatus (6.6) and increase the temperature at a rate of 2 °C/min up to 50 °C, and then continue increasing the temperature at a rate of 0,5 °C/min.

NOTE — If the capillary tubes are attached to the thermometer, take care that the centres of the mercury bulb and the sample columns are on the same level.

9.4.4 Using a magnifying lens, observe the temperatures at which the last solid particle in each tube disappears. Record these temperatures, to the nearest 0,1 °C, as the melting points.

9.5 Number of determinations

Carry out two complete determinations on the same test sample (clause 8).

10 Expression of results

The Bömer value, I_B, is given by the formula

$$I_B = t_g + 2\,(t_g - t_a)$$

where

t_g is the melting point of the triglycerides, in degrees Celsius;

t_a is the melting point of the fatty acids, in degrees Celsius.

Take as the result the arithmetic mean of the two determinations, provided that the requirement for repeatability (clause 11) is satisfied. Express the result to one decimal place.

NOTE — If the requirement is not met, the determination of melting point (9.4) should be repeated. Only if the requirement is still not met should the complete procedure (clause 9) be repeated.

11 Repeatability

The difference between the values of two complete determinations, carried out in rapid succession (or simultaneously) by the same analyst using the same apparatus on the same test sample, shall not exceed 0,5.

12 Test report

The test report shall specify which of the two methods has been used, the values of the melting points of both the triglycerides and the fatty acids, and the calculated Bömer value.

It shall also mention all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the results.

The test report shall include all information necessary for the complete identification of the sample.