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Animal fats — Determination of Bömer value

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FOREWORD

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Animal fats – Determination of Bömer value

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies two methods for the determination of the Bömer value of pork fat intended for human and animal consumption :

- the diethyl ether method (method I);
- 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 preferably, 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 REFERENCE

ISO . . ., *Crude animal and vegetable fats — Sampling.*1)

3 DEFINITION

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

- by filtration and thoroughly washing with diethyl ether (method I), or
- by centrifuging or decanting followed by thoroughly 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 sulphuric 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 sulphuric acid, then evaporation of the solvent.

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

5 REAGENTS

Reagents shall be of analytical reagent quality. Water used shall be distilled water or water of at least equivalent purity.

5.1 Diethyl ether, freed from peroxides by an appropriate procedure and freshly distilled (for method I).

5.2 Acetone (for method II).

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

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

5.5 Sodium sulphate, anhydrous.

5.6 Filter aid.

5.7 Potassium hydroxide pellets.

5.8 Sulphuric acid, approximately 1 N solution.

6 APPARATUS

Usual laboratory apparatus and the following items :

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

6.2 Conical flask, 100 ml capacity, with ground neck, connected to a reflux condenser.

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

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

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1) In preparation.

6.5 Calibrated thermometer, type STL/0,2/35/85 in accordance with ISO/R 653.

6.6 Suitable melting point apparatus filled with a liquid medium and equipped with a stirrer.

6.7 Desiccator containing concentrated sulphuric acid or other equally effective acid desiccant.

6.8 Centrifuge tube, 100 ml, or **glass-stoppered cylinder** of the same capacity (for method II).

6.9 Büchner funnel.

6.10 Boiling water bath.

6.11 Oven, capable of being controlled at 102 ± 2 °C.

7 PROCEDURE

7.1 Preparation of test sample

Melt about 150 g of the homogenized sample (see ISO . . .) at a temperature about 10 °C above the melting point of the fat and filter at this temperature through a dry fast filter paper to which a mixture of 4 g of anhydrous sodium sulphate (5.5) and 1 g of filter aid (5.6) has been added. It is essential that the filtrate be perfectly clear; if it is not, the whole procedure shall be repeated, using a new portion of anhydrous sodium sulphate and filter aid.

7.2 Method I

7.2.1 *Test portion*

Weigh about 50 g of the test sample into a 150 ml beaker.

7.2.2 *Preparation of triglycerides*

7.2.2.1 Add 50 ml of diethyl ether (5.1), cover 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 quite clear.

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

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

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

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

7.2.2.5 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.

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

Filter off the crystals, if necessary under slight suction, on the Büchner funnel (diameter depending on the amount of crystals obtained), using a well-fitted fast filter paper. Wash the crystals on the filter paper three times with 25 ml portions of diethyl ether cooled to 15 °C. Detach the crystals from the filter paper and return them into the beaker which was used for the crystallization, after first rinsing the beaker with some diethyl ether.

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

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

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

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

7.3 Method II

7.3.1 *Test portion*

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

7.3.2 *Preparation of triglycerides*

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

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

7.3.2.2 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.

7.3.2.3 Add 20 ml of acetone (5.2) at 30 °C. Shake and isolate the crystals as described in 7.3.2.2.

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

Wash the crystals on the filter paper five times with 5 ml portions of acetone at 30 °C, the last time under suction.

7.3.2.5 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.

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

7.4 Preparation of fatty acids

WARNING : It is essential that the fatty acids be prepared in an atmosphere free from ammonia (NH_3).

7.4.1 Remove a sufficient amount of the powdered triglycerides (7.2.2.9 or 7.3.2.6) 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 ethanol (5.4) and 0,4 g (two pellets) of solid potassium hydroxide (5.7).

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

7.4.3 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.

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

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

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

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

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

7.4.8 Filter the solution through a dry filter paper, to which approximately 4 g of anhydrous sodium sulphate (5.5) has been added, into a 100 ml beaker.

7.4.9 Distil off the solvent over a water-bath.

7.4.10 Place the beaker in the drying oven (6.11) maintained at 102 ± 2 °C and leave for 15 to 20 min.

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

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

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

7.5 Determination of melting points

7.5.1 Fill a capillary tube (6.4) with the powdered triglycerides (7.2.2.9 or 7.3.2.6). Pack the powder to a height of approximately 5 mm, for instance by dropping the capillary tube through a glass tube of sufficient height onto a wooden base.

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

7.5.2 Fill another capillary tube, in the same way as described in 7.5.1, with the powdered fatty acids (7.4.12), 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.

7.5.3 Place both tubes and the thermometer (6.5) in the melting point apparatus (6.6) and raise the temperature at a rate of 2 °C/min until 50 °C, and then 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.

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

7.6 Number of determinations

Carry out two complete determinations on the same test sample (7.1).

8 EXPRESSION OF RESULTS

8.1 Method of calculation and formula

The Bömer value, B, is given by the formula

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

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.

8.2 Repeatability

The difference between the results of two complete determinations of *B* (see 7.6) carried out simultaneously or in rapid succession by the same analyst shall not exceed 0,5 °C. If this limit is exceeded, the whole procedure shall be repeated.

9 TEST REPORT

The test report shall show 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 any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the results.

The report shall include all details required for complete identification of the sample.