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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Animal and vegetable fats and oils — Determination of titre

Corps gras d'origines animale et végétale — Détermination du titre

Reference number
ISO 935 : 1988 (E)

Foreword

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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 935 was prepared by Technical Committee ISO/TC 34, *Agricultural food products.*

It cancels and replaces ISO Recommendation R 935 : 1969, of which it constitutes a technical revision.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Animal and vegetable fats and oils — Determination of titre

1 Scope and field of application

This International Standard specifies a method for the preparation of the water-insoluble fatty acids of animal and vegetable fats and oils and the determination of their solidification temperature, called conventionally the titre of the fat or oil.

The method is not applicable to fats and oils the titre of which is below 30 °C.

2 References

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

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

3 Definition

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

titre: Constant temperature observed when there is a temporary halt in the fall in temperature or, if there is an increase in temperature, the maximum temperature reached during the cooling, with continuous stirring, of liquid fatty acids.

4 Principle

Saponification of a test portion with potassium hydroxide solution in glycerol, dissolution of the soap in water and neutralization. Washing of the separated insoluble fatty acids with hot sodium chloride solution, drying and filtering.

Melting of the prepared fatty acids, cooling them, with continuous stirring, and observation of the solidification temperature as shown by a halt in the fall of temperature, possibly accompanied by a temporary rise in temperature.

5 Reagents

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

5.1 Potassium hydroxide, solution in glycerol, prepared by dissolving 100 g of potassium hydroxide in 500 g of glycerol by heating to not more than 130 °C. Foaming may occur at higher temperatures.

5.2 Sulfuric acid, solution prepared by carefully adding 1 volume of concentrated sulfuric acid (ϱ = 1,84 g/ml) to 4 volumes of water, while stirring.

5.3 Sodium chloride, 100 g/l solution.

5.4 Sodium sulfate, anhydrous.

5.5 Methyl orange, 5 g/l indicator solution.

6 Apparatus

Usual laboratory apparatus and in particular

6.1 Flat-bottomed flask or **beaker,** of 1 litre capacity, made of borosilicate glass.

6.2 Electric hotplate, capable of being controlled at approximately 130 and at 140 ± 10 °C, preferably having a magnetic stirrer.

6.3 Separating funnel, of 500 ml capacity.

6.4 Cooling apparatus, fitted with an apparatus suitable for continuous stirring (see the figure).

6.4.1 Wide-necked jar, of height 130 mm and external diameter 100 mm.

6.4.2 Test tube, 100 mm × 25 mm, fitted with a stirrer that can be operated continuously and move through a vertical distance of about 40 mm. The test tube may be marked at a height of 55 mm. The stirrer shall be made of glass or inert metal 2 to 3 mm in diameter, with the bottom end bent to form a complete circle 19 mm in diameter in the horizontal plane.

6.4.3 Flat cork, to fit the jar (6.4.1) and with a central hole to support the test tube (6.4.2).

6.4.4 Thermometer, graduated in 0,1 °C divisions, calibrated for the temperature range within which the titre falls and

suitable to be suspended so that the bulb is about 10 mm from the bottom of the test tube (6.4.2).

6.5 Water (or other liquid) bath, capable of being maintained at a temperature 20 to 25 °C below the expected titre.

7 Sampling

See ISO 5555.

8 Preparation of test sample

Prepare the test sample in accordance with ISO 661.

9 Procedure

9.1 Preparation of fatty acids insoluble in water

Weigh about 110 g of the potassium hydroxide solution (5.1) into the 1 litre flask or beaker (6.1). Stir and heat on the hot plate (6.2) to about 130 °C. Add about 45 g of the test sample (clause 8) heated to about 60 °C. Stir the mixture on the hotplate, controlled at 140 ± 10 °C, until the saponification is complete, as indicated by the mixture becoming transparent and homogeneous with the formation of a foam persisting for a few minutes when stirring and heating are stopped.

Allow the temperature to drop to about 105 °C. Add 300 ml of hot water, with stirring. Stir and heat (to boiling if necessary) until the soap is completely dissolved. Add the sulfuric acid solution (5.2), usually about 50 ml, with stirring, until the mixture is neutral to methyl orange (5.5). Continue heating and stirring until the fatty acids form a clear liquid surface layer.

NOTE — If short-chain fatty acids are present, heat the acidified mixture under reflux.

Transfer the hot mixture to the separating funnel (6.3). Draw off and reject the aqueous layer.

Wash the fatty acids with three 150 ml portions of the sodium chloride solution (5.3) which is hot enough to maintain the fatty acids in a liquid state. After each washing, draw off the aqueous layer as completely as possible and reject it.

Transfer the fatty acids to a 100 ml beaker, add approximately 5 g of the anhydrous sodium sulfate (5.4) and stir. Filter through a dry filter paper, ensuring that the fatty acids remain liquid.

9.2 Determination of titre

Heat the fatty acids (9.1) to about 10 °C above the expected titre. Pour into the test tube (6.4.2) to a height of 55 mm, assemble the cooling apparatus (6.4) and place it in the bath (6.5). Immediately operate the stirrer at a rate of 80 to 100 complete up-and-down motions per minute.

Observe the temperature which, after a rapid initial decrease, falls slowly as crystallization starts and then becomes constant or rises within 30 s. Stop the stirring.

Record to the nearest 0,1 °C either the constant temperature or the maximum temperature reached in any rise as the titre.

9.3 Number of determinations

Carry out a two determinations (9.2) on the same prepared fatty acids (9.1).

10 Expression of results

10.1 Method of calculation

Take as the result the arithmetic mean of the two determinations (9.3), provided that the requirements for repeatability (10.2) are met. Otherwise carry out two more determinations on the same prepared fatty acids (9.1).

Express the result to the nearest 0,1 °C.

10.2 Repeatability

The difference between the values of two determinations carried out in rapid succession by the same analyst on the same prepared fatty acids shall not exceed 0,2 °C.

NOTE — The figure of 0,2 °C has been accepted historically in several countries. Recent tests have not shown any reason to disagree with this figure.

11 Test report

The test report shall show the method used and the result obtained. 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 result.

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

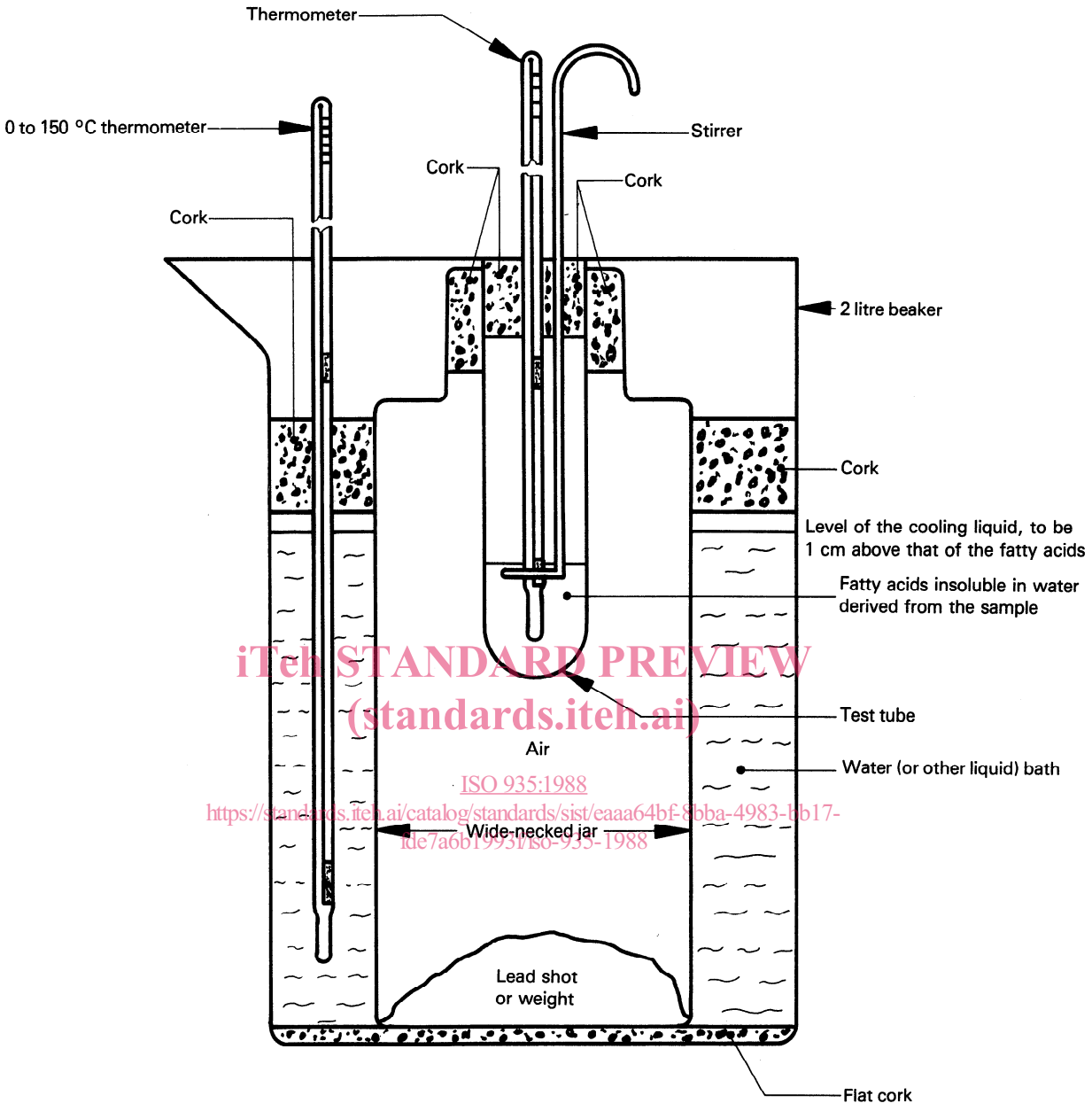

Figure — Cooling apparatus

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