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Milk and canned evaporated milk — Determination of tin content — Spectrometric method

Lait et lait concentré non sucré en boîte — Détermination de la teneur en étain — Méthode spectrométrique

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of normative document:

— an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote;

— an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

An ISO/PAS or ISO/TS is reviewed after three years in order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn. If the ISO/PAS or ISO/TS is confirmed, it is reviewed again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/TS 9941 | IDF/RM 160 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*, and the International Dairy Federation (IDF). It is being published jointly by ISO and IDF.

This second edition cancels and replaces the first edition (ISO/TS 9941 | IDF/RM 160:2003), which has been technically revised.

Foreword

IDF (the International Dairy Federation) is a worldwide federation of the dairy sector with a National Committee in every member country. Every National Committee has the right to be represented on the IDF Standing Committees carrying out the technical work. IDF collaborates with ISO and AOAC International in the development of standard methods of analysis and sampling for milk and milk products.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the Action Teams and Standing Committees are circulated to the National Committees for voting. Publication as an International Standard requires approval by at least 50 % of IDF National Committees casting a vote.

In other circumstances, particularly when there is an urgent market requirement for such documents, a Standing Committee may decide to publish an other type of normative document which is called by IDF: *Reviewed method*. Such a method represents an agreement between the members of a Standing Committee and is accepted for publication if it is approved by at least 50 % of the committee members casting a vote. A *Reviewed method* is equal to an ISO/PAS or ISO/TS and will, therefore, also be published jointly under ISO conditions.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. IDF shall not be held responsible for identifying any or all such patent rights.

ISO/TS 9941|IDF/RM 160 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*, and the International Dairy Federation (IDF). It is being published jointly by ISO and IDF.

All work was carried out by the Joint ISO/IDF Action Team on *Minor compounds*, of the Standing Committee on *Minor components and characterization of physical properties*, under the aegis of its project leaders, Dr M. Carl (DE) and Dr G. Ellen (NL).

This second edition cancels and replaces the first edition (ISO/TS 9941|IDF/RM 160:2003), which has been technically revised.

Introduction

This Technical Specification (Reviewed Method for IDF) specifies a spectrometric method for the determination of tin content in canned evaporated milk. Its limit of determination is 5 mg of tin per kilogram.

Despite several attempts, the Joint IDF-ISO Action Team (JAT) on *Minor compounds* could not organize a collaborative study with a sufficient number of participating laboratories to be in accordance with ISO 5725-2. Thus no precision figures for repeatability and reproducibility could be established. However, the method has been proven to be reliable by at least three experienced laboratories and particularly with respect to its accuracy.

The method was therefore adopted as a Technical Specification rather than as an International Standard.

Milk and canned evaporated milk — Determination of tin content — Spectrometric method

1 Scope

This Technical Specification (Reviewed Method for IDF) specifies a spectrometric method for the determination of the tin content in canned evaporated milk. The limit of determination of the method is 5 mg/kg.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
tin content
mass fraction of substances determined by the procedure specified in this Technical Specification

NOTE The tin content is expressed in milligrams per kilogram of sample.

4 Principle

The organic material is mineralized with a mixture of nitric acid and sulfuric acid. Water, hydrochloric acid and sodium carbonate solution are added. Interfering iron(III) ions are masked with thiourea solution and the tin(IV) is complexed with quercetin. The absorbance of the yellow solution is measured spectrometrically at the wavelength of maximum absorption (437 nm).

5 Reagents

Use only reagents of very pure analytical grade which, with the exception of the tin standard solution (5.11), are free from tin.

5.1 Water, double distilled or of equivalent quality, complying with the requirements of ISO 3696:1987, grade 2.

5.2 Ethanol (CH_3CH_2OH), with volume fraction of about 96 %.

5.3 Diethyl ether [$(C_2H_5)_2O$].

5.4 Sulfuric acid, concentrated, $\rho_{20}(H_2SO_4)$ = 1,84 g/ml.

5.5 Nitric acid, concentrated, $\rho_{20}(HNO_3)$ = 1,42 g/ml.

5.6 Hydrogen peroxide solution, $\rho_{20}(H_2O_2)$ = (1,099 to 1,103) g/ml.

Store the hydrogen peroxide solution in a refrigerator.

5.7 Hydrochloric acid, dilute, c(HCl) = 2,5 mol/l.

Dilute 221 ml of concentrated hydrochloric acid [ρ_{20}(HCl) = (1,17 to 1,18) g/ml] with water to 1 000 ml and mix.

5.8 Sodium carbonate solution, $c(Na_2CO_3)$ = 200 g/l.

Dissolve 200 g of anhydrous sodium carbonate in water. Dilute with water to 1 000 ml and mix.

5.9 Thiourea solution, $c[CS(NH_2)_2]$ = 90 g/l.

Dissolve 45 g of thiourea in water. Dilute with water to 500 ml and mix.

5.10 Quercetin solution, $c(C_{15}H_{10}O_7)$ = 0,75 g/l.

Dissolve 750 mg of quercetin (3,3',4',5,7-pentahydroxyflavone) in ethanol (5.2). Dilute with ethanol (5.2) to 1 000 ml and mix. Filter the quercetin solution through a filter paper. Store the solution in the dark for no longer than one week.

5.11 Tin standard solution, c(Sn) = 1,000 g/l.

Dilute a commercially available preparation that contains exactly 1,000 g of Sn (e.g. Sn(IV), Titrisol No. 9929 [1)] is suitable) with the dilute hydrochloric acid (5.7) to 1 000 ml and mix well.

5.12 Milk, with low tin content, e.g. bottled evaporated milk or whole pasteurised milk.

5.13 Sodium chloride solution (NaCl), containing 0,5 % sodium chloride (mass fraction).

6 Apparatus

Keep the clean glassware, including the glass (or quartz) beads (6.5), in 10 % (mass fraction) nitric acid. Rinse the glassware and the beads three times before use with distilled water and then three times with double-distilled water. Dry, if necessary, by successively rinsing with ethanol (5.2) and diethyl ether (5.3).

Usual laboratory equipment and, in particular, the following.

6.1 Analytical balance, capable of weighing to the nearest 1 mg.

6.2 Water baths, capable of operating at 20 °C ± 2 °C, at 40 °C ± 2 °C, and at between 40 °C and 60 °C.

6.3 Micro gas burners, which do not emit tin-containing particles, or **electric heaters**.

6.4 Mineralization flasks, (Kjeldahl flasks), of capacity approximately 70 ml, with ground-glass joints (NS 19, with matching glass stoppers), calibrated with a mark on the lower part of the neck at 50 ml.

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1) Titrisol No. 9929 is an example of a suitable product available commercially. This information is given for the convenience of users of this Technical Specification and does not constitute an endorsement by either ISO or IDF of this product.

6.5 **Beads**, made of glass or quartz.

6.6 **One-mark volumetric flasks**, of capacities 50 ml, 500 ml and 1 000 ml.

6.7 **Graduated pipettes**, of capacities 1 ml, 2 ml and 5 ml, with 0,1 ml graduations.

6.8 **One-mark pipettes**, capable of delivering 3 ml, 10 ml and 20 ml.

6.9 **Micro-pipettes**, capable of delivering 10 µl, 20 µl, 50 µl, 100 µl and 150 µl.

6.10 **Spectrometer**, capable of measuring absorbance at 437 nm, equipped with glass or quartz cells of 10 mm optical path length, with polytetrafluoroethylene stoppers.

7 Sampling

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this Technical Specification. A recommended sampling method is given in ISO 707 | IDF 50.

Care should be taken to avoid contamination by tin during sampling. Store glass sampling jars in 10 % (mass fraction) nitric acid. Rinse them thoroughly and dry before use.

8 Preparation of test sample

8.1 Shake the container thoroughly with frequent inversion. Taking care to avoid contamination by tin, open the container and pour out its contents slowly into another pre-cleaned container made of glass and provided with an airtight lid. Incorporate in the test sample any fat or other constituents adhering to the wall of the original container. Stir vigorously and close the glass container.

8.2 Heat the closed glass container in a water bath (6.2) set at between 40 °C and 60 °C. Remove and shake the container vigorously every 15 min. After 2 h, remove the container and cool in another water bath (6.2) to 20 °C. Remove the lid and mix thoroughly by stirring the test sample with a spoon or spatula.

8.3 Correct results cannot be expected if the fat separates out.

9 Procedure

9.1 Test portion and pretreatment

Take precautions to avoid contamination by tin during the procedure. Weigh, to the nearest 1 mg, 0,5 g ± 0,1 g of prepared test sample (8.2) into a mineralization flask (6.4). While swirling, add 0,5 ml of water, 0,50 ml of concentrated sulfuric acid (5.4) and 0,5 ml of concentrated nitric acid (5.5).

9.2 Mineralization

9.2.1 Add three glass (or quartz) beads (6.5) to the contents of the mineralization flask (9.1). Leave the flask at room temperature for 30 min. Operating under a well-ventilated fume hood, place the flask in an inclined position and heat it with a micro gas burner or electric heater (6.3). Limit production of foam in the flask by controlling the height of the burner flame or the power position of the electric heater. Foaming is allowed into the neck of the flask but no foam shall escape. Keep the flask contents gently boiling. Avoid local overheating. Do not directly heat the flask above the surface level of the contents (see also 9.6).