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Milk and milk products — Determination of zinc content — Flame atomic absorption spectrometric method

Lait et produits laitiers — Détermination de la teneur en zinc — Méthode par spectrométrie d'absorption atomique avec flamme

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Foreword

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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.

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 11813 | IDF 156 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 of ISO 11813 | IDF 156 cancels and replaces the first edition (ISO 11813:1998), of which it constitutes a minor revision.

Foreword

IDF (the International Dairy Federation) is a non-profit organization representing the dairy sector worldwide. IDF membership comprises National Committees in every member country as well as regional dairy associations having signed a formal agreement on cooperation with IDF. All members of IDF have the right to be represented on the IDF Standing Committees carrying out the technical work. IDF collaborates with ISO in the development of standard methods of analysis and sampling for milk and milk products.

The main task of Standing Committees is to prepare International Standards. Draft International Standards adopted by the Standing Committees are circulated to the National Committees for endorsement prior to publication as an International Standard. Publication as an International Standard requires approval by at least 50% of IDF National Committees casting a vote.

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ISO 11813 | IDF 156 was prepared by the International Dairy Federation (IDF) and Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*. It is being published jointly by IDF and ISO.

All work was carried out by the former Joint ISO-IDF Action Team on *Minor compounds*, now part of the Standing Committee on *Analytical methods for composition*.

This edition of ISO 11813 | IDF 156 cancels and replaces IDF 156:1992, of which it constitutes a minor revision.

Milk and milk products — Determination of zinc content — Flame atomic absorption spectrometric method

1 Scope

This International Standard specifies a flame atomic absorption spectrometric method for the determination of the zinc content of milk and milk products. The method has been validated for zinc contents of between 25 mg/kg and 70 mg/kg (dry mass) in milk and milk products.

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 6732|IDF 103, *Milk and milk products — Determination of iron content — Spectrometric method (Reference method)*

3 Terms and definitions

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

3.1
zinc content in milk and milk products
mass fraction of substances determined by the procedure specified in this International Standard

NOTE The zinc content is expressed in milligrams per kilogram of product.

4 Principle

The sample is dry ashed in a programmable ashing furnace. The ash is dissolved in concentrated hydrochloric acid and, after adding strontium chloride solution, diluted with water. The zinc content of the resulting solution is measured by flame atomic absorption spectrometry at a wavelength of 213,9 nm with deuterium or Zeeman background correction.

5 Reagents

Use only reagents of recognized analytical grade which, with the exception of the standard zinc solutions (5.5), shall be free from zinc.

Unless otherwise indicated, the use of Aristar, Suprapur or Ultrex reagents[1)] or products of equivalent reagent grade purity is strongly recommended.

5.1 Water, complying with grade 2 as defined in ISO 3696[3].

5.2 Hydrochloric acid (HCl), concentrated (ρ_{20} = 1,17 g/ml to 1,18 g/ml).

5.3 Strontium chloride solution.

Dissolve 38,0 g of strontium chloride hexahydrate ($SrCl_2 \cdot 6H_2O$) in water (5.1) and make up to 250 ml with water.

NOTE Strontium chloride hexahydrate from BDH (Spectrosol)[1)] or equivalent is suitable.

5.4 Nitric acid (HNO_3), concentrated (ρ_{20} = 1,42 g/ml).

5.5 Zinc standard solutions.

5.5.1 Stock solution, containing 1 000 mg of zinc per litre of 0,3 mol/l nitric acid (equivalent to 18,9 g/l of nitric acid).

NOTE Baker Instra-analyzed Atomic Spectral Solution[1)] 1.6946 is suitable.

5.5.2 Working solution, containing 100 mg of zinc per litre. Add 1 ml of nitric acid (5.4) to 10 ml of the stock solution (5.5.1) and make up to 100 ml with water (5.1).

5.6 Zero-standard solution.

In a 500 ml one-mark volumetric flask, dilute 2,5 ml of hydrochloric acid (5.2) and 12,5 ml of strontium chloride solution (5.3) with water (5.1) to the 500 ml mark. Mix well.

6 Apparatus

IMPORTANT — Store clean glassware in nitric acid, 10 % mass fraction. Rinse it three times before use with distilled water and then three times with double-distilled water.

Usual laboratory equipment and in particular the following.

6.1 Quartz crucibles, with quartz lids, capacity 50 ml.

6.2 One-mark volumetric flasks, capacities 100 ml and 250 ml, ISO 1042[2] class A.

6.3 Manual piston pipettes, capacities 0,2 ml, 1,0 ml, and 5,0 ml, ISO 8655-2[6].

6.4 Drying oven, capable of being maintained at 102 °C ± 5 °C.

6.5 Programmable ashing furnace, or equivalent, capable of maintaining a minimum attainable temperature of 550 °C with a heating rate programmable at 50 °C/h.

NOTE If a programmable ashing furnace is not available, manual adjustment of temperature of an isothermal furnace is possible in steps of 50 °C every hour.

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1) Aristar, Suprapur, Ultrex and Spectrosol reagents and Baker Instra-analyzed Atomic Spectral Solution are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO or IDF of these products.

6.6 Hotplate, capable of maintaining a temperature of about 100 °C.

6.7 Water bath, capable of maintaining water at boiling point.

6.8 Flame atomic absorption spectrometer, capable of measuring at a wavelength of 213,9 nm with a recommended spectral band width of 0,2 nm and a 10 cm single-slot acetylene-air burner and deuterium or Zeeman background correction.

6.9 Analytical balance, capable of being read to the nearest 1 mg.

7 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 707|IDF 50[1].

It is important that the laboratory receive a truly representative sample which has not been damaged or changed during transport or storage.

Store the sample in such a way that deterioration and change in its composition are prevented. Precautions should be taken to avoid contamination of the sample by zinc.

8 Preparation of test sample

Avoiding contamination by zinc, prepare the test sample according to ISO 6732|IDF 103.

9 Procedure

9.1 General

If it is required to check whether the repeatability limit (see 11.2) is met, carry out two single determinations in accordance with 9.2 to 9.4.

9.2 Test portion

Weigh, to the nearest 1 mg, 5 g ± 1 g of the prepared test sample of milk (or an amount of the prepared test sample of a milk product equivalent to 0,5 g ± 0,1 g of dry mass) into a quartz crucible (6.1). Dry the contents carefully in the oven (6.4) maintained at 102 °C.

9.3 Ashing

Heat the quartz crucible containing the dried test portion (9.2) in the ashing furnace (6.5) from room temperature to 500 °C at a rate of 50 °C/h. Maintain it for 3 h at a temperature of 500 °C.

Subsequently, let the contents of the crucible cool to ambient temperature in a zinc-free cabinet. If no white ash is obtained, wet the ash with approximately 0,5 ml of water (5.1), then add three drops of nitric acid (5.4).

Dry carefully on a hotplate (6.6) or on a boiling water bath (6.7). Heat again for 30 min in the furnace (6.5) maintained at 500 °C.

9.4 Determination

9.4.1 Preparation of test solution

Add 0,5 ml of water (5.1) and 0,5 ml of hydrochloric acid (5.2) to the ash (9.3). Dissolve the ash and quantitatively transfer the dissolved ash with water to a 100 ml one-mark volumetric flask (6.2).

Using a 5,0 ml piston pipette (6.3), add 2,5 ml of strontium chloride solution (5.3) to the contents of the flask. Make up to the mark with water and mix thoroughly.

9.4.2 Atomic absorption spectrometric measurement

Adjust the spectrometer (6.8) to a wavelength of 213,9 nm and the flame conditions to yield optimum precision and sensitivity.

9.4.2.1 Calibration

Add to four 100 ml one-mark volumetric flasks (6.2) 0,2 ml, 0,4 ml, 0,6 ml and 0,8 ml, respectively, of the zinc working solution (5.5.2). Make up to the mark with the zero-standard solution (5.6) and mix thoroughly. These calibration solutions contain 0,2 mg, 0,4 mg, 0,6 mg and 0,8 mg of zinc per litre respectively.

Subsequently, aspirate the zero-standard solution (5.6) and the four calibration solutions four times each and calculate the means of the absorbance values.

Subtract from the means of the absorbance values of the calibration solutions the mean of the absorbance value of the zero-standard solution. Plot the resulting net absorbance values against the corresponding calibration concentrations.

NOTE Depending on the functionalities of the instrument, subtraction can also be done by autozeroing.

9.4.2.2 Measurement of sample solution

Measure the test solution (see 9.4.1) immediately after the calibration measurements, under the same conditions.

If its signal is above that of the highest standard, dilute the test solution with zero-standard solution (5.6) (dilution factor f) and repeat the measurements.

Intermittently, check the instrument and the calibration stability by using the 0,4 mg/l zinc calibration solution. Also perform calibration measurements at the end of a series of measurements and, for large series, perform additional measurements in the middle of the series.

Repeat each measurement four times and calculate the arithmetic average of these values. Subtract from it the mean absorbance value of the zero-standard solution. Read the corresponding concentration from the calibration plot (see 9.4.2.1).

10 Calculation and expression of results

Calculate the zinc content, w_{Zn}, in milligrams per kilogram, of the sample using the following equation:

$$w_{Zn} = \frac{\rho_{Zn}\, f\, V}{m}$$

where

ρ_{Zn} is the zinc concentration, in milligrams per litre, of the test solution, read from the calibration graph (see 9.4.2.2);

f is the dilution factor [final volume of diluted test solution (see 9.4.2.2) divided by volume of original test solution (see 9.4.1) taken for dilution];

V is the volume, in millilitres (V = 100 ml), of the test solution (see 9.4.1);

m is the mass, in grams, of the test portion.

11 Precision

11.1 Interlaboratory test

The values for the repeatability and reproducibility were derived from the results of an interlaboratory test carried out in accordance with ISO 5725-1[4] and ISO 5725-2[5]. Details of the interlaboratory test on the precision of the method are summarized in Reference [7].

The values derived from this interlaboratory test may not be applicable to concentration ranges and matrices other than those given.

11.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of cases be greater than 7 % of the arithmetic mean of the two results.

11.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will in not more than 5 % of cases be greater than 4 mg/kg of dry matter of the product.

12 Test report

The test report shall contain at least the following information:

a) all information necessary for the complete identification of the sample;

b) the sampling method used, if known;

c) the test method used, with reference to this International Standard (ISO 11813|IDF 156:2010);

d) any operating conditions not specified in this International Standard, or regarded as optional, as well as details of any incidents that may have influenced the test result(s);

e) the test result(s) obtained;

f) if the repeatability has been checked, the final quoted result obtained.