INTERNATIONAL STANDARD

**ISO 11813**

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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*

## 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 3.

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.

International Standard ISO 11813 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Subcommittee SC 5, *Milk and milk products*, in collaboration with the International Dairy Federation (IDF) and the Association of Official Analytical Chemists (AOAC International), and will also be published by these organizations.





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# 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 normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.



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



ISO 6732, *Milk and milk products — Determination of iron content — Spectrometric method (Reference method).*

## 3 Term and definition

For the purposes of this International Standard, the following term and definition apply.

**3.1**
**zinc content of 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.

## 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 (5.4)] are free from zinc. Use only water complying with grade 2 of ISO 3696.

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

**5.1 Hydrochloric acid** (HCl), concentrated ($\rho_{20}$ = 1,17 g/ml to 1,18 g/ml).

**5.2 Strontium chloride solution**

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

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

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

**5.4 Standard zinc solutions**

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

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

**5.4.2 Working solution**, containing 100 mg of Zn per litre. Add 1 ml of the nitric acid (5.3) to 10 ml of the stock solution (5.4.1) and dilute with water to 100 ml.

**5.5 Zero-standard solution**

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



## 6 Apparatus

Keep the clean glassware in nitric acid (10 % mass fraction). Rinse 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, of capacity 50 ml.

**6.2 One-mark volumetric flasks**, of capacities 100 ml and 250 ml.

**6.3 Manual piston pipettes**, of capacities 0,2 ml, 1,0 ml and 5,0 ml.

**6.4 Drying oven**, capable of operating at 102 °C ± 5 °C.

**6.5 Programmable ashing furnace**, or equivalent, capable of operating at a minimum attainable temperature of 550 °C with a heating rate programmable at 50 °C per hour.

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.

**6.6 Hotplate**, capable of operating at about 100 °C.

**6.7 Water bath**, capable of boiling water.

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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 of these products.

**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 [1].

It is important that the laboratory receive a sample which is truly representative and 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.

## 9 Procedure



NOTE If it is required to check whether the repeatability limit (11.2) is met, carry out two single determinations in accordance with 9.1 to 9.3.



### 9.1 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) set at 102 °C.

### 9.2 Ashing

Heat the quartz crucible containing the dried sample (9.1) in the ashing furnace (6.5) from room temperature to 500 °C at a rate of 50 °C per hour. Keep 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 a white ash is not obtained, wet the ash with approximately 0,5 ml of water and subsequently 3 drops of nitric acid (5.3).

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) set at 500 °C.

### 9.3 Determination

#### 9.3.1 Preparation of test solution

Add 0,5 ml of water and 0,5 ml of hydrochloric acid (5.1) to the ash (9.2). Dissolve the ash and quantitatively transfer the dissolved ash with water to a 100 ml volumetric flask (6.2).

Using a 5,0 ml piston pipette (6.3), add 2,5 ml of the strontium chloride solution (5.2) to the contents of the volumetric flask. Dilute to the mark with water and mix thoroughly.

#### 9.3.2 Atomic absorption spectrometric measurement

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

##### 9.3.2.1 Calibration

Add to four 100 ml volumetric flasks (6.2) 0,2 ml, 0,4 ml, 0,6 ml and 0,8 ml of the zinc working solution (5.4.2) respectively. Dilute to the mark with the zero-standard solution (5.5) 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 (5.5) 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 instrument facilities, subtraction can also be done by autozeroing.

##### 9.3.2.2 Measurement of the sample solution

Measure the test solution (9.3.1) 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 (5.5) (dilution factor $f$) and repeat the measurements.



Intermittently verify 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 additionally in the middle of the series.



Repeat each measurement four times and calculate the average of these values. Subtract from it the obtained mean absorbance value of the zero-standard solution. Read from the calibration plot (9.3.2.1) the corresponding concentration.

## 10 Calculation and expression of results

Calculate the zinc content of the sample using the following equation:

$$w_S = \frac{c_S \cdot f \cdot V}{m}$$

where

$w_S$ is the zinc content of the test sample, in milligrams per kilogram;

$c_S$ is the zinc concentration of the test solution, read from the calibration graph (9.3.2.2), in milligrams per litre;

$f$ is the dilution factor [final volume of diluted test solution (9.3.2.2) divided by volume of original test solution (9.3.1) taken for dilution];

$V$ is the volume of the test solution (9.3.1), in millilitres ($V$ = 100 ml);

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

## 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 [2,3]. Details of the interlaboratory test on the precision of the method are summarized in reference [4].

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 specify:



— all information necessary for the complete identification of the sample;

— the sampling method used, if known;

— the test method used, with reference to this International Standard;

— all operational details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);

— the test result(s) obtained; or

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

# Bibliography

[1] ISO 707, *Milk and milk products — Guidance on sampling.*

[2] ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions.*

[3] ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibilty of a standard measurement method.*

[4] Collaborative study — Zinc in milk and milk products. *Bulletin of IDF*, **306**, 1995, pp. 2-19.

[5] Koops J., Klomp H. and Westerbeek D., *Netherlands Milk & Dairy J.*, **40**, 1986, pp. 337-350.