

Milk products — Determination of ash

WD/CD/DIS/FDIS stage

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Contents

Foreword	3
1 Scope	4
2 Normative references	4
3 Terms and definitions.....	4
4 Principle.....	4
5 Apparatus	4
5.1 Analytical balance, capable of weighing to the nearest 0,0001g.....	5
5.2 Silica or platinum dishes, of diameter about 70mm and of depth 25mm to 50mm.....	5
5.3 Electrical furnace, with air circulation, capable of being controlled at 550 ± 25°C.....	5
5.4 Desiccator, containing an effective desiccant.....	5
5.5 Grinding device, for grinding the laboratory sample, if necessary, without the development of undue heat and without loss or absorption of moisture. A hammer mill shall not be used.	5
5.6 Test Sieve, wire cloth, diameter 200mm, nominal size of aperture 500µm, with receiver, complying with ISO 3310-1.....	5
5.7 Thermogravimetric analyser, incorporating analytical balance (5.1), electrical furnace (5.3) and suitable dishes. The dishes may not conform to the dimensions specified in 5.2.....	5
6 Sampling	5
7 Preparation of the test sample	5
7.1 Milk powder, whey powder, whey protein concentrate, infant formula, milk permeate powder, milk protein concentrate and caseinates.....	5
7.2 Casein.....	5
8 Determination.....	6
8.1 Preparation of the dish.....	6
8.2 Test Portion	6
8.3 Charring	6
8.4 Ashing	6
9 Expression of results	6
10 Precision	7
10.1 Repeatability.....	7
10.2 Reproducibility	7
Annex A (informative) Collaborative trials.....	8
A.1 Clause title autonumber.....	8
A.1.1 Subclause autonumber.....	8
A.1.1.1 Subclause autonumber.....	8
Bibliography	9

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.

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This document was prepared by Technical Committee ISO/TC [or ISO/PC] 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/third/...** edition cancels and replaces the **first/second/...** edition (ISO #####:#####), which has been technically revised.

The main changes compared to the previous edition are as follows:

— xxx xxxxxxxx xxx xxx

A list of all parts in the ISO ##### series can be found on the ISO website.

Milk products — Determination of ash

1 Scope

This International Standard specifies a method for the determination of the ash content of various milk products. The method is applicable to milk powder, whey powder, whey protein concentrate, infant formula, milk permeate powder, milk protein concentrate, caseins and caseinates.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 3310-1, *Test sieves — Technical requirements and testing— Part 1: Test sieves of metal wire cloth*

3 Terms and definitions

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

3.1

ash

substances determined by the procedure described in this International Standard

Note: Ash of milk products is conventionally expressed as a percentage by mass, numerically equivalent to grams per 100g of product.

4 Principle

A test portion is incinerated at $550 \pm 25^\circ\text{C}$. The residue is then weighed.

5 Apparatus

Usual laboratory apparatus, and in particular the following.

5.1 Analytical balance, capable of weighing to the nearest 0,0001g.

5.2 Silica or platinum dishes, of diameter about 70mm and of depth 25mm to 50mm.

5.3 Electrical furnace, with air circulation, capable of being controlled at $550 \pm 25^\circ\text{C}$.

5.4 Desiccator, containing an effective desiccant.

5.5 Grinding device, for grinding the laboratory sample, if necessary, without the development of undue heat and without loss or absorption of moisture. A hammer mill shall not be used.

5.6 Test Sieve, wire cloth, diameter 200mm, nominal size of aperture $500\mu\text{m}$, with receiver, complying with ISO 3310-1.

5.7 Thermogravimetric analyser¹, incorporating analytical balance (5.1), electrical furnace (5.3) and suitable dishes. The dishes may not conform to the dimensions specified in 5.2.

6 Sampling

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

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

7 Preparation of the test sample

7.1 Milk powder, whey powder, whey protein concentrate, infant formula, milk permeate powder, milk protein concentrate and caseinates

Transfer the sample to a container of internal volume approximately twice the volume of the sample. Close the container immediately to avoid changing the moisture content of the sample. Thoroughly mix the sample by repeatedly rotating and inverting the container.

After the test sample has been prepared, the determination should proceed as soon as possible.

7.2 Casein

Thoroughly mix the sample as in 7.1.

Transfer about 50g of the thoroughly mixed sample to the test sieve (5.6). If the 50g portion directly passes or almost completely passes the sieve, use for the determination.

Otherwise, grind the 50g portion, using the grinding device (5.5), until it passes the sieve. Immediately transfer all of the sieved sample to a container of internal volume approximately twice the volume of the sample. Close the container immediately to avoid changing the moisture content of the sample. Thoroughly mix the sample by repeatedly rotating and inverting the container.

¹ The Leco TGA710 is an example of a suitable commercially available thermogravimetric analyser.

After the test sample has been prepared, the determination should proceed as soon as possible.

8 Determination

8.1 Preparation of the dish

Heat the dish (5.2) in the electrical furnace (5.3), maintained at 550°C, for a minimum of 30 min. Allow the dish to cool in the desiccator (5.4) to the temperature of the balance room and weigh to the nearest 0,1mg.

Where a thermogravimetric analyser (5.7) is used the dishes should be heated to 550°C and maintained at that temperature for a minimum of 30 min. Allow to cool in the analyser furnace and weigh to the nearest 0,1mg.

8.2 Test Portion

Weigh to the nearest 0,1mg directly or by difference approximately 1g of the test sample into the prepared dish.

8.3 Charring

Heat the dish containing the test portion until it is completely charred, taking care that it does not burst into flame. This stage of the incineration can be performed manually via hotplate or flame prior to transferring the dish to the electrical furnace (5.3). Alternatively charring can be incorporated into a programme which gradually increases temperature of the furnace or thermogravimetric analyser until the 550°C is achieved.

8.4 Ashing

Maintain the sample in the furnace or thermogravimetric analyser for at least 1 hour at 550°C until all the carbon has disappeared from the dish.

For electrical furnace (5.3), allow the dish to cool in the desiccator (5.4) to the temperature of the balance room and weigh to the nearest 0,1mg.

Repeat the operations of heating in the electrical furnace (5.3), cooling weighing, until the mass remains constant to within 0,5mg or begins to increase. Record the minimum mass.

Thermogravimetric analysers continually weigh samples throughout the incineration of the sample. The constant weight criteria may be via a fixed or relative mass change within a defined time period. The criteria selected should achieve a similar outcome to the constant weight criteria for the electrical furnace (5.3).

9 Expression of results

The ash of the sample, w_a , as a percentage by mass, is given by equation (1):

$$w_a = \frac{m_1 - m_2}{m_0} \times 100$$

Where

m_0 is the mass, in grams, of the test portion (8.2);

m_1 is the mass, in grams, of the dish and residue (8.4);

m_2 is the mass, in grams, of the prepared dish (8.1).

Calculate the ash to the nearest 0,01% by mass and report the final result to the nearest 0,1% by mass.

10 Precision

10.1 Repeatability

The absolute difference between two 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 not in more than 5% of cases be greater than r,r % by mass.

10.2 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 not in more than 5% of cases be greater than R,R % by mass.

Annex A
(informative)

Collaborative trials

(A summary of the data generated during the collaborative study will be included as annex A following completion and statistical analysis)

A.1 Clause title autonumber

Use subclauses if required e.g. A.1.1 or A.1.1.1. For example:

A.1.1 Subclause autonumber

A.1.1.1 Subclause autonumber

Type text

Bibliography

- [1] ISO 707|IDF 50, *Milk and milk products — Guidance on sampling*
- [2] ISO #####-##:20##, *General title — Part ##: Title of part*