EAST AFRICAN COMMUNITY

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EAS 80-9 (2006) (English): Butter Methods of analysis Part 9: Determination of iron content

ISO INSIDE

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EAST AFRICAN STANDARD

Butter — Methods of chemical analysis — Part 9: Determination of iron content

EAST AFRICAN COMMUNITY

First Edition 2006

Foreword

Development of the East African Standards has been necessitated by the need for harmonizing requirements governing quality of products and services in East Africa. It is envisaged that through harmonized standardization, trade barriers which are encountered when goods and services are exchanged within the Community will be removed.

In order to achieve this objective, the Partner States in the Community through their National Bureaux of Standards, have established an East African Standards Committee.

The Committee is composed of representatives of the National Standards Bodies in Partner States, together with the representatives from the private sectors and consumer organizations. Draft East African Standards are circulated to stakeholders through the National Standards Bodies in the Partner States. The comments received are discussed and incorporated before finalization of standards, in accordance with the procedures of the Community.

East African Standards are subject to review, to keep pace with technological advances. Users of the East African Standards are therefore expected to ensure that they always have the latest versions of the standards they are implementing.

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International Standard 6732

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Milk and milk products — Determination of iron content — Spectrometric method (Reference method)

Lait et produits laitiers — Détermination de la teneur en fer — Méthode spectrométrique (Méthode de référence)

First edition — 1985-07-01

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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.

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 6732 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*.

NOTE — The method specified in this International Standard has been developed jointly with the International Dairy Federation (IDF) and the Association of Official Analytical Chemists (AOAC) and will also be published by these organizations.

Printed in Switzerland

Milk and milk products — Determination of iron content — Spectrometric method (Reference method)

1 Scope and field of application

This International Standard specifies a spectrometric reference method for the determination of the iron content of milk and milk products.

This method is applicable to

- milk, skimmed milk, whey and buttermilk
- plain yogurt and skimmed yogurt
- evaporated milk and sweetened condensed milk
- dried whole and skimmed milk, dried whey and dried buttermilk
- cream and butter
- butterfat[1)]
- ice-cream
- cheese of various ages, and processed cheese
- caseins, caseinates and coprecipitates.

NOTE — The method demands experience in trace analysis and careful execution. During the procedure, special consideration should be given to the problem of contamination, which will affect the accuracy and repeatability of the method.

It is therefore recommended

- to keep reagents, glassware and the laboratory environment as clean as possible (contamination by rust is a particular hazard)
- that each laboratory identifies and controls its own sources of contamination
- that the difference between two blank values is kept as low as possible (generally, this difference should not exceed 0,004 absorbance unit)
- that the performance of the method be checked by analysing milk powder (or another milk product) of known or certified iron content.

2 Reference

ISO 707, *Milk and milk products — Methods of sampling.*

3 Definition

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

iron content (of a milk or milk product): The whole of the substances determined by the procedure specified in this International Standard.

The iron content is usually reported as a mass fraction, and is then conventionally expressed in milligrams per kilogram of sample.

4 Principle

Digestion of the organic material with a mixture of nitric and sulfuric acids, preceded, in the case of cream and butterfat, by removal of the fat. In the case of butter, separation and digestion of the serum.

Complexing the iron(II) ions, obtained by reduction of iron(III) ions, by means of bathophenanthroline. Extraction of the iron(II) compound with isoamyl alcohol. Spectrometric measurement at a wavelength of 533 nm of the absorbance of the red solution thus obtained.

5 Reagents

All reagents shall be of very pure analytical grade and, with the exception of the iron standard solutions (5.13 and 5.14), shall be free from iron. The water used shall comply with the requirements of ISO 3696,[2)] grade 2.

NOTE — The use of Aristar, Suprapur or Ultrex reagents, or products of equivalent purity, is strongly recommended for reagents 5.5, 5.6, 5.7 and 5.8. See also the note to 8.4.

1) The designation "butterfat" covers all the products described in IDF Standard 68 A, *Anhydrous milkfat, anhydrous butteroil or anhydrous butterfat, butteroil or butterfat, ghee (compositional standards).*

2) ISO 3696, *Water for laboratory use — Specifications.* (At present at the stage of draft.)

5.1 Ethanol, about 96 % (*V*/*V*).

Distil, if necessary, in an iron-free distillation unit.

5.2 Diethyl ether.

Distil, if necessary, in an iron-free distillation unit.

5.3 Light petroleum, boiling range 40 to 60 °C.

Distil, if necessary, in an iron-free distillation unit.

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

Distil in an iron-free distillation unit. Discard the first 50 ml of distillate. Do not store the nitric acid in a brown glass bottle.

5.5 Sulfuric acid, concentrated, ϱ_{20} = 1,84 g/ml.

5.6 Potassium sulfate, solution in sulfuric acid.

Dissolve 25 g of dry potassium sulfate (K_2SO_4) in the sulfuric acid (5.5) and make up to 100 ml with the same sulfuric acid. Filter the solution, without suction, through an all glass, iron-free, filter crucible, of porosity grade P 100 (pore diameter 40 to 100 µm).

NOTE — If the potassium sulfate available is not iron-free purify it as follows.

Dissolve 40 g of potassium sulfate in 500 ml of water and add 3 ml of the hydroxylammonium chloride solution (5.9). Extract the solution with 10 ml of the bathophenanthroline solution (5.11). Remove the upper layer. Repeat these two operations until the upper layer remains colourless. Evaporate the water in a clean oven.

5.7 Hydrogen peroxide, solution, ϱ_{20} = 1,099 to 1,103 g/ml.

Store in a refrigerator.

5.8 Sodium acetate, saturated solution.

Dissolve 232,5 g of anhydrous sodium acetate (CH_3COONa) in 500 ml of water.

NOTE — If the sodium acetate available is not iron-free, purify it as follows.

Dissolve 232,5 g of sodium acetate in 500 ml of water. Filter through a filter paper. Add 3 ml of the hydroxylammonium chloride solution (5.9). Extract the solution with 10 ml of the bathophenanthroline solution (5.11). Remove the upper layer. Repeat these two operations until the upper layer remains colourless.

5.9 Hydroxylammonium chloride, solution.

Dissolve 20 g of hydroxylammonium chloride ($HONH_3Cl$) in water and make up to 100 ml. Filter through a filter paper. Extract the solution with 5 ml of the bathophenanthroline solution (5.11). Allow the layers to separate properly. Remove the upper layer. Repeat these two operations until the upper layer remains colourless.

NOTES

1 Generally five extractions are sufficient.

2 If the solution was prepared more than 24 h before use, it is advisable to repeat the extraction with the bathophenanthroline.

3 Instead of the hydroxylammonium chloride solution, a freshly prepared solution of ascorbic acid can be used as a reducing agent. This ascorbic acid solution can be made by dissolving 10 g of ascorbic acid in 100 ml of water. The solution should be extracted with the bathophenanthroline solution in exactly the same way as described for the hydroxylammonium chloride solution. It should be stored in a refrigerator. Instead of 3 ml of the hydroxylammonium chloride solution 3 ml of this ascorbic acid solution can be used in 5.6, 5.8 and 8.1.3.4.

5.10 Isoamyl alcohol (3-methyl-1-butanol).

Distil, if necessary, in an iron-free distillation unit.

5.11 Bathophenanthroline, solution.

Dissolve 83,1 mg of bathophenanthroline [4,7-diphenyl-1,10-phenanthroline ($C_{24}H_{16}N_2$)] in 100 ml of the isoamyl alcohol (5.10).

5.12 Potassium permanganate, solution.

Dissolve 100 mg of potassium permanganate ($KMnO_4$) in 50 ml of water.

5.13 Iron, standard solution corresponding to 1 000 mg of iron per litre.

Dissolve 7,022 g of ammonium iron(II) sulfate hexahydrate [$(NH_4)_2Fe(SO_4)_2{\cdot}6H_2O$] in 250 ml of water. Add 8 ml of the sulfuric acid (5.5) and cool to room temperature. Dilute to 1 000 ml with water.

1 ml of this standard solution contains 1 mg of Fe.

NOTE — Preparations which contain exactly 1 000 mg of iron may be used instead of the ammonium iron(II) sulfate hexahydrate. Such preparations are available commercially.

5.14 Iron, standard solution corresponding to 1 mg of iron per litre.

On the day of use, pipette 1 ml of the standard iron solution (5.13) into 250 ml of water. Add 1 ml of the sulfuric acid (5.5) and dilute to 1 000 ml with water.

1 ml of this standard solution contains 1 µg of Fe.

6 Apparatus

Keep the clean glassware, including the glass beads (6.8), in 10 % (*m*/*m*) nitric acid solution. Rinse three times before use with distilled water and then three times with double-distilled water. If necessary, dry by successively rinsing with ethanol and diethyl ether.

Usual laboratory equipment, and in particular

6.1 Analytical balance.

6.2 Centrifuge, capable of producing a radial acceleration of 2 500*g*, with tubes of capacity at least 150 ml.

6.3 Grinding device, appropriate to the nature of the sample.

6.4 Sieve, of aperture size 0,5 mm, made of iron-free material.

6.5 Water-baths.

6.6 Micro-burners, which do not emit iron-containing particles.

NOTE — The use of electric heaters is also permitted.

6.7 Digestion flasks (Kjeldahl), of capacity approximately 70 ml, with ground glass stoppers, calibrated on the lower part of the neck at 50 ml.

6.8 Beads, made of glass or preferably quartz, which do not release iron during the digestion procedure (8.3.1).

6.9 Graduated cylinders, of capacities 5; 10; and 25 ml.

6.10 Graduated pipettes, of capacities 1; 2; and 5 ml, graduated in divisions of 0,1 ml, complying with the requirements of ISO 835.

6.11 One-mark pipettes, delivering 1; 2; 3; 4; 5; 10; and 25 ml, complying with the requirements of ISO 648, class A.

6.12 Spectrometer, suitable for measuring absorbance at 533 nm, equipped with cells of optical path length 10 mm.

7 Sampling

NOTE — Avoid contamination by iron.

7.1 See ISO 707.

Store glass sampling jars in 10 % (*m*/*m*) nitric acid solution.

Rinse them thoroughly and dry before use.

7.2 Store the sample in such a way that deterioration and change in composition are prevented.

8 Procedure

NOTE — Avoid contamination by iron.

8.1 Preparation of the test sample

8.1.1 Milk, skimmed milk and whey

Bring the sample to 20 ± 2 °C and mix carefully.

If, in the case of milk, the fat is not evenly dispersed, heat the sample slowly to 40 °C, mix gently by inversion only, and cool quickly to 20 ± 2 °C.

8.1.2 Buttermilk

If necessary, remove butter granules. Bring the sample to 20 ± 2 °C and mix carefully, immediately before weighing (8.2.1).

8.1.3 Plain yogurt and skimmed yogurt

Bring the sample to 20 ± 2 °C and mix carefully. If serum separates, stir vigorously, immediately before weighing (8.2.1).

8.1.4 Cream

Bring the sample to 20 ± 2 °C. Mix or stir thoroughly, but not so vigorously as to cause frothing or churning.

If the cream is very thick, or if the fat is not evenly dispersed, warm slowly to 40 °C to facilitate mixing.

Cool the sample quickly to 20 ± 2 °C. Stir the sample in the container thoroughly. Mix until the whole mass is homogeneous. Close the container.

NOTE — Correct results cannot be expected if adequate mixing of the sample is not achieved or if the sample shows any evidence of churning or any other signs of abnormality.

8.1.5 Evaporated milk

Shake the container thoroughly with frequent inversion. Open this container and pour the milk slowly into another container made of glass, provided with an airtight lid, taking care to incorporate in the sample any fat or other constituents adhering to the wall of the original container. Stir vigorously and close the container.

Heat the closed container in a water-bath at 40 to 60 °C. Remove and shake the container vigorously every 15 min. After 2 h, remove the container and cool to 20 ± 2 °C. Remove the lid and mix thoroughly by stirring the sample with a spoon or spatula.

NOTE — If the fat separates, correct results cannot be expected.

8.1.6 Sweetened condensed milk

Open the container and thoroughly mix the milk with a spoon or spatula, using an up and down rotary movement in such a way that the top and bottom layers are moved and mixed. Take care to incorporate in the sample any milk adhering to the wall and ends of the container.

Transfer the sample as completely as possible to a second container made of glass, provided with an airtight lid, and close this container. Heat the closed container in a water-bath at 30 to 40 °C. Cool to 20 ± 2 °C. Stir the sample in the container thoroughly. Mix until the whole mass is homogeneous. Close the container.

In the case of a collapsible tube, open it and transfer the contents to a glass container. Cut open the tube and transfer as completely as possible all material adhering to the interior to the container.

8.1.7 Dried whole and dried skimmed milk, dried whey, dried buttermilk

Transfer the sample into a container of capacity about twice the volume of the sample and provided with an airtight lid. Close the container immediately. Mix the sample thoroughly by repeatedly shaking and inverting the container.

8.1.8 Butter

NOTE — Because of the possible inhomogeneous distribution of iron in butter, the iron is determined in the serum. The iron content of the fat fraction, separated from the butter in the manner described, is negligible compared with that of the serum and can be ignored.

Weigh, to the nearest 100 mg, 100 g of the sample into a dry previously tared centrifuge tube (6.2). Place the tube in a water-bath maintained at 45 ± 1 °C. As soon as the butter has melted, centrifuge the tube with a radial acceleration of 2 500g. Remove as much as possible of the clear fat layer by means of a pipette. Extract with 10 ml of light petroleum (5.3) and remove the upper layer by means of a pipette. Repeat these two operations twice. Remove residual light petroleum by warming in a water-bath at 65 ± 1 °C. Dry the outside of the tube with a clean paper tissue. Cool to 20 ± 2 °C. Weigh the tube with its contents to the nearest 100 mg. Mix the contents carefully, immediately before weighing the test sample (8.2.5).

8.1.9 Butterfat

Bring the sample to 40 °C, maintain at this temperature for 5 min and mix gently. Cool to 20 ± 2 °C.

8.1.10 Ice cream

For samples taken in small packages, remove the packaging and place the sample in a container provided with an airtight lid. For samples taken from bulk or from large packages, keep them in their sample containers. In either case, melt the sample by standing the closed sample container in a water-bath at 45 ± 1 °C for just sufficient time to allow the sample to become fluid. Mix the sample by shaking. Cool to 20 ± 2 °C, continuing mixing until cooling is completed.

8.1.11 Cheese and processed cheese

Remove the rind, smear or mouldy surface layer of the cheese, in such a way as to provide a sample representative of the cheese as it is usually consumed. Grind the sample using the appropriate grinding device (6.3). Quickly mix the whole mass and, preferably, quickly grind the mass again. (If the sample cannot be ground, mix the whole sample thoroughly.)

Immediately transfer the pretreated sample, or a representative part of it, into a container provided with an airtight lid. Analyse the sample without delay, as soon as possible after grinding. Ground cheese showing unwanted mould growth or beginning to deteriorate shall not be examined.

8.1.12 Caseins, caseinates and coprecipitates

8.1.12.1 If most of the sample is sufficiently fine to pass through the sieve (6.4), it may be used without any grinding.

Transfer about 50 g of the sample as received into a container of capacity about twice the volume of the powder and provided with an airtight lid. Close the container immediately and mix the sample thoroughly by repeatedly shaking and inverting the container.

8.1.12.2 If most of the sample is not sufficiently fine to pass through the sieve (6.4), grind about 50 g of the sample until most of it does so. Transfer all the material into a container. Continue as specified in 8.1.12.1.

8.2 Weighing and pre-treatment of the test sample

8.2.1 Milk, skimmed milk, whey, buttermilk and yogurt

Weigh, to the nearest 10 mg, 10 g of the test sample into a digestion flask (6.7). Add 3 ml of the nitric acid (5.4) and 1,8 ml of the potassium sulfate solution (5.6). Continue as specified in 8.3.

8.2.2 Cream

Weigh, to the nearest 10 mg, 10 g of the test sample into a digestion flask (6.7). Add 8 ml of the nitric acid (5.4). Heat the flask in a water-bath at 80 to 90 °C for 1 h.

Shake vigorously every 3 min in order to wash the fat with the nitric acid. Cool to 40 °C and remove as much of the fat layer as possible by means of a pipette.

Add 15 ml of the light petroleum (5.3), swirl carefully, and remove the solvent by means of a pipette. Repeat twice with fresh 15 ml portions of the light petroleum. Remove residual light petroleum by warming in a water-bath at 65 °C. Cool to room temperature. Add 1,8 ml of the potassium sulfate solution (5.6). Continue as specified in 8.3.

8.2.3 Evaporated milk and sweetened condensed milk

Weigh, to the nearest 1 mg, 2,5 g of the test sample into a digestion flask (6.7). Add 4 ml of water, 3 ml of the nitric acid (5.4) and 1,8 ml of the potassium sulfate solution (5.6). Continue as specified in 8.3.

8.2.4 Dried whole and dried skimmed milk, dried whey, dried buttermilk

Weigh, to the nearest 1 mg, 1 g of the test sample into a digestion flask (6.7). Add 4 ml of water, and mix well. Then add 3 ml of the nitric acid (5.4) and 1,8 ml of the potassium sulfate solution (5.6). Continue as specified in 8.3.

8.2.5 Butter

Weigh, to the nearest 1 mg, 2 g of the butter serum (8.1.8) into a digestion flask (6.7). Add 3 ml of the nitric acid (5.4) and 1,8 ml of the potassium sulfate solution (5.6). Continue as specified in 8.3.

8.2.6 Butterfat

Weigh, to the nearest 10 mg, 20 g of the fluid test sample into a digestion flask (6.7). Add 4 ml of water and 8 ml of the nitric acid (5.4).

Heat the flask in a water-bath at 80 to 90 °C for 1 h. Shake thoroughly every 3 min in order to wash the fat with the nitric acid. Cool to 40 °C and remove as much of the fat layer as possible by means of a pipette.

Add 15 ml of the light petroleum (5.3), swirl carefully, and remove the solvent by means of a pipette. Repeat twice with fresh 15 ml portions of the light petroleum. Remove residual light petroleum by warming in a water-bath at 65 °C. Cool to room temperature. Add 1,8 ml of the potassium sulfate solution (5.6). Continue as specified in 8.3.

8.2.7 Ice cream

Weigh, to the nearest 1 mg, 2,5 g of the test sample into a digestion flask (6.7). Add 3 ml of the nitric acid (5.4) and 1,8 ml of the potassium sulfate solution (5.6). Continue as specified in 8.3.

8.2.8 Cheese and processed cheese

Weigh, to the nearest 1 mg, 1 g of the test sample into a digestion flask (6.7). Add 4 ml of water, 3 ml of the nitric acid (5.4) and 1,8 ml of the potassium sulfate solution (5.6). Continue as specified in 8.3.

8.2.9 Caseins, caseinates and coprecipitates

Weigh, to the nearest 0,1 mg, in the case of caseins and caseinates, 0,75 g, and in the case of coprecipitates, 0,35 g, of the test sample into a digestion flask (6.7). Add 4 ml of water, 3 ml of the nitric acid (5.4) and 1,8 ml of the potassium sulfate solution (5.6). Continue as specified in 8.3.

8.3 Determination

NOTE — Carry out the blank test (8.4) simultaneously with the determination.

8.3.1 Digestion

8.3.1.1 Add three glass (quartz) beads (6.8) to the test portion in the digestion flask. Operating under a well-ventilated fume hood, place the flask in an inclined position and heat with a micro-burner. Control the height of the flame so as to limit the production of foam in the flask. Foaming into the neck of the flask is allowed but the foam shall not escape. Keep the mixture gently boiling and avoid local overheating.

8.3.1.2 When the solution has turned brown, carefully add 3 to 5 drops of the nitric acid (5.4). Heat vigorously as soon as possible. Continue heating and adding the nitric acid, 5 to 20 drops at a time, swirling the flask occasionally to remove any material adhering to the wall, until the mixture remains colourless. Cool to room temperature.

8.3.1.3 Carefully add 2 ml of water and 1 ml of the hydrogen peroxide solution (5.7). Swirl and heat again until white fumes are emitted. Prevent loss through evaporation by allowing the sulfuric acid fumes to reflux in the neck of the flask. (If the solution becomes yellow, cool to room temperature. Add a further 0,5 ml of the hydrogen peroxide solution and then heat until white fumes are emitted.) Continue heating for 45 min after the beginning of the emission of white fumes. Cool to room temperature and carefully add water to give a total volume of approximately 20 ml.

8.3.1.4 Add one or two drops of the potassium permanganate solution (5.12) until the digest becomes faintly purple. Then add 3 ml of the hydroxylammonium chloride solution (5.9) and mix well. Add 20 ml of the sodium acetate solution (5.8) and about 15 ml of water. Mix well and allow to cool to room temperature. Make up to the 50 ml mark with water.

8.3.2 Colour development

Add, by means of a pipette, 4 ml of the bathophenanthroline solution (5.11) to the contents of the digestion flask (see 8.3.1.4) and close the flask with a stopper. Shake the flask vigorously for 3 min, ensuring that the stopper remains in position.

Cool in running tap water for at least 10 min and carefully tilt the flask several times after cooling. Keep the flask in a water-bath at 25 ± 1 °C for 1 h.

8.4 Blank test

Simultaneously with the determination, carry out a blank test using all the reagents used for the determination but replacing the test portion by 10 ml of water. During the digestion period, use the same amount of the nitric acid (5.4) and the hydrogen peroxide solution (5.7) as for the digestion of the test portion.

NOTE — The absorbance of the blank test solution should correspond to less than 0,5 µg of iron. If the absorbance of the blank test corresponds to more than 0,5 µg of iron, all reagents should be checked.

8.5 Spectrometric measurements

Transfer the isoamyl alcohol (upper) layer, by means of a pipette into a 10 mm cell (6.12). Measure the absorbances of the isoamyl alcohol layers of the test solution (8.3) and the blank test solution (8.4) at 533 nm against water as reference. Subtract the value for the blank test solution from that of the test solution.

8.6 Number of determinations

Carry out all determinations, including the blank test (8.4), in duplicate.

8.7 Preparation of the calibration graph

8.7.1 Pipette 0 (zero member); 1; 2; 3; 5; and 10 ml of the iron standard solution (5.14) into a series of six digestion flasks (6.7). Dilute with water to about 10 ml. Add to each flask 3 ml of the nitric acid (5.4) and 1,8 ml of the potassium sulfate solution (5.6).

8.7.2 Carry out the digestion as described in 8.3.1 and the colour development as described in 8.3.2.

8.7.3 Transfer each isoamyl alcohol (upper) layer, by means of a pipette, into a 10 mm cell (6.12). Measure the absorbance of each isoamyl alcohol layer at 533 nm against water as reference. Subtract the value for the zero member from the values obtained for the other solutions.

8.7.4 Plot these absorbances against the amounts of iron contained in the calibration solutions.

8.7.5 Check the calibration graph weekly.

9 Expression of results

9.1 Method of calculation and formulae

9.1.1 Products other than butter

The iron content, expressed in milligrams per kilogram, is equal to

$$\frac{m_1}{m_0}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in micrograms, of iron, read from the calibration graph (or calculated from the regression line obtained by the method of least squares).

9.1.2 Butter

Calculate the iron content of the butter serum as described in 9.1.1.

The iron content of the butter, expressed in milligrams per kilogram, is equal to

$$\frac{m_3}{m_2} \times w_{Fe}$$

where

m_2 is the mass, in grams, of the butter transferred to the centrifuge tube (see 8.1.8);

m_3 is the mass, in grams, of butter serum, obtained in 8.1.8;

w_{Fe} is the iron content, in milligrams per kilogram, of the butter serum, calculated as described in 9.1.1.

9.1.3 Result

Take as the result the arithmetic mean of the results obtained, provided that the requirement for repeatability (see 9.2) is satisfied.

Express the result to the number of decimal places shown in the table.

9.2 Repeatability

The difference between the results of duplicate determinations (results obtained almost simultaneously or in rapid succession by the same analyst) shall not be greater than the repeatability value for the product analysed, as given in the table.

Table — Expression of results and repeatability

Product	Expression of result to the nearest	Repeatability
	mg/kg	mg/kg
Milk	0,001	0,02
Skimmed milk	0,001	0,02
Whey	0,001	0,02
Buttermilk	0,001	0,03
Yogurt	0,001	0,03
Evaporated milk	0,01	0,1
Sweetened condensed milk	0,01	0,1
Dried whole milk	0,01	0,2
Dried skimmed milk	0,01	0,2
Dried whey, dried buttermilk	0,01	0,2
Cream	0,001	0,02
Butter	0,001	0,03
Butterfat	0,001	0,005
Ice cream	0,01	0,2
Cheese and processed cheese	0,01	0,2
Caseins, caseinates and coprecipitates	0,1	0,4

10 Test report

The test report shall show the method used and the results obtained. It shall also mention any operating details not specified in this International Standard, or regarded as optional, together with details of any incidents likely to have influenced the results.

The test report shall include all the information necessary for the complete identification of the sample.