INTERNATIONAL STANDARD

ISO 8070

First edition
1987-03-15

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

Dried milk — Determination of sodium and potassium contents — Flame emission spectrometric method

Lait sec — Détermination des teneurs en sodium et potassium — Méthode par spectrométrie d'émission de flamme

Reference number
ISO 8070 : 1987 (E)

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 8070 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.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Printed in Switzerland

Dried milk — Determination of sodium and potassium contents — Flame emission spectrometric method

1 Scope and field of application

This International Standard specifies a flame emission spectrometric method for the determination of the sodium and potassium contents of all types of dried milk.

2 References

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

ISO 3696, *Water for laboratory use — Specifications and test methods.*[1)]

3 Definition

sodium and potassium contents of dried milk: The contents of substances determined by the procedures specified in this International Standard and expressed as percentages by mass.

4 Principle

Dissolution of the dried milk in warm water. Atomization of the solution and of reference solutions directly into the flame of a flame emission spectrometer and spectrometric measurement of the intensity of the emitted light.

5 Reagents

All reagents shall be of recognized analytical grade. The water used shall be distilled water or water of at least equivalent purity, complying with the requirements of ISO 3696, grade 2.

5.1 Hydrochloric acid, about 4 mol/l.

Dilute 300 ml of concentrated [37 % (*m*/*m*)] hydrochloric acid to 1 000 ml with water and mix.

5.2 Standard solutions

Store the standard solutions in vessels of hard polyethylene or of other material of at least equivalent quality.

5.2.1 Sodium, standard solution.

Dissolve 1,016 8 g of sodium chloride (NaCl), dried to constant mass at 110 to 120 °C, in water, dilute to 1 000 ml and mix.

1 ml of this standard solution contains 0,4 mg of Na.

5.2.2 Potassium, standard solution.

Dissolve 1,906 8 g of potassium chloride (KCl), dried to constant mass at 110 to 120 °C, in water, dilute to 1 000 ml and mix.

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

5.2.3 Calcium, standard solution.

Dissolve 2,497 2 g of calcium carbonate ($CaCO_3$), dried to constant mass at 110 to 120 °C, in 15 ml of the hydrochloric acid (5.1), dilute to 1 000 ml with water and mix.

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

5.2.4 Phosphorus, standard solution.

Dissolve 10,660 g of diammonium monohydrogenorthophosphate [$(NH_4)_2 HPO_4$] in water, dilute to 1 000 ml and mix.

1 ml of this standard solution contains 2,5 mg of P.

1) At present at the stage of draft.

6 Apparatus

All glassware shall be thoroughly cleaned and rinsed with distilled water to ensure that under the test conditions it is free from extractable sodium and potassium.

Usual laboratory equipment, and in particular

6.1 Analytical balance.

6.2 Glass beaker, of capacity 50 ml.

6.3 One-mark volumetric flasks, of capacities 100 - 500 - 1 000 ml, complying with the requirements of ISO 1042, class B.

6.4 Graduated measuring cylinder, of capacity 50 ml, complying with the requirements of ISO 4788.

6.5 One-mark pipettes, to deliver 10 - 15 - 20 - 25 - 30 - 40 - 45 - 50 - 60 ml, complying with the requirements of ISO 648, class A, or ISO 835.

NOTE — Where appropriate, burettes may be used instead of pipettes.

6.6 Glass rod.

6.7 Flame emission spectrometer, with a burner fed with a mixture of either acetylene and air or propane and air, and provided with filters with maximum transmittance at about 589 and 768 nm for sodium and potassium respectively, or fitted with a monochromator.

7 Sampling

7.1 See ISO 707.

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

8 Procedure

CAUTION — Avoid contamination, especially with sweat.

8.1 Preparation of the test sample

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

During preparation, avoid exposure of the sample to the atmosphere as far as possible, in order to minimize adsorption of atmospheric moisture.

8.2 Test portion

Weigh, to the nearest 1 mg, 1,25 g of the test sample into the glass beaker (6.2).

8.3 Preparation of test solution

Dissolve the test portion (8.2) in about 20 ml of warm water (40 to 50 °C), while stirring with the glass rod (6.6). Transfer the contents of the glass beaker by rinsing with water quantitatively into a 500 ml one-mark volumetric flask (6.3), cool to about 20 °C and dilute to the mark. Mix the contents of the flask thoroughly.

NOTES

1 Using dried normal whole milk, this solution contains approximately 10 mg of sodium and 40 mg of potassium per litre.

2 If this solution contains insoluble particles, reliable results will not be obtained.

8.4 Preparation of sodium and potassium reference solutions

Pipette successively into seven 1 000 ml one-mark volumetric flasks the volumes of the standard solutions (5.2.1, 5.2.2 and 5.2.3) indicated in the table, and dilute to 900 ml with water. Add to each flask 10 ml of the phosphorus standard solution (5.2.4), dilute to the mark with water and mix.

Table — Preparation and composition of sodium and potassium reference solutions

Reference solution	Volume of sodium standard solution (5.2.1)	Corresponding Na content	Volume of potassium standard solution (5.2.2)	Corresponding K content	Volume of calcium standard solution (5.2.3)	Corresponding Ca content	Volume of phosphorus standard solution (5.2.4)	Corresponding P content
	ml	mg/l	ml	mg/l	ml	mg/l	ml	mg/l
1	15	6	50	50	30	30	10	25
2	20	8	45	45	30	30	10	25
3	25	10	40	40	30	30	10	25
4	30	12	40	40	30	30	10	25
5	40	16	35	35	35	35	10	25
6	50	20	30	30	35	35	10	25
7	60	24	25	25	35	35	10	25

NOTES

1 Reference solution 7 is optional.

2 These reference solutions can be kept for at least 1 month if stored in vessels of hard polyethylene or of other material of at least equivalent quality.

8.5 Determination

8.5.1 Sodium content

Atomize the reference solutions (8.4), starting with the solution with the lowest sodium content, and the test solution (8.3), alternately into the flame of the flame emission spectrometer, following the manufacturer's instructions and using the sodium filter, or the monochromator, adjusted to 589 nm. Note the readings.

8.5.2 Potassium content

Atomize the reference solutions (8.4), starting with the solution with the lowest potassium content, and the test solution (8.3), alternately into the flame of the flame emission spectrometer, following the manufacturer's instructions and using the potassium filter, or the monochromator, adjusted to 768 nm. Note the readings.

8.5.3 Dilution

If the flame emission spectrometric reading for the test solution (8.3) exceeds that of the reference solution with the highest concentration, repeat the spectrometric measurements using an appropriate dilution of the test solution (8.3) and the appropriate reference solutions. Prepare, for this purpose, reference solutions having concentrations of sodium, potassium, calcium and phosphorus which are as close as possible to the expected concentrations in the diluted test solution.

9 Expression of results

9.1 Method of calculation and formula

The sodium and potassium contents of the sample, expressed as percentages by mass, are given by the formula

$$\left[\frac{I_X - I_1}{I_2 - I_1}(c_2 - c_1) + c_1\right]\frac{f}{m}$$

where

I_X is the reading of the flame emission spectrometer for the test solution (8.3);

I_1 is the nearest lower reading of the flame emission spectrometer for the reference solution of concentration c_1;

I_2 is the nearest higher reading of the flame emission spectrometer for the reference solution with concentration c_2;

c_1 is the concentration, in milligrams per litre, of the reference solution giving reading I_1;

c_2 is the concentration, in milligrams per litre, of the reference solution giving reading I_2;

f is the conversion factor for expressing the results as a percentage by mass (for sodium and potassium, $f = 0{,}04$);

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

NOTE Take into account any dilution of the test solution (see 8.5.3).

Report the sodium and potassium contents to the nearest 0,01 % (m/m).

9.2 Precision

NOTE — The values for repeatability and reproducibility have been derived from the results of an inter-laboratory test in accordance with ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests*.[1]

9.2.1 Repeatability

The difference between the results of two determinations, carried out simultaneously or in rapid succession by the same analyst using the same apparatus, shall not exceed 6 % relative of the arithmetic mean of the results for both sodium and potassium.

9.2.2 Reproducibility

The difference between two single and independent results, obtained by two different analysts working in different laboratories on identical test material, shall not exceed 8 % relative of the arithmetic mean of the results for both sodium and potassium.

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.

Bibliography

[1] *Bulletin of the International Dairy Federation* (1986), No. 207.

This page intentionally left blank

This page intentionally left blank

UDC 637.143 : 637.046

Descriptors : agricultural products, dairy products, milk, dried milk, chemical analysis, determination of content, sodium, potassium, flame photometric analysis.

Price based on 3 pages