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Cheese — Determination of chloride content (Reference method)

Fromage — Détermination de la teneur en chlorures (Méthode de référence)

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FOREWORD

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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.

International Standard ISO 2970 was drawn up by Technical Committee ISO/TC 34, *Agricultural food products*, and circulated to the Member Bodies in August 1972.

It has been approved by the Member Bodies of the following countries :

Australia	France	New Zealand
Austria	Germany	Poland
Belgium	Hungary	Romania
Brazil	India	South Africa, Rep. of
Chile	Iran	Spain
Czechoslovakia	Ireland	Thailand
Egypt, Arab Rep. of	Israel	Turkey
Finland	Netherlands	United Kingdom

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

No Member Body expressed disapproval of the document.

NOTE — This International Standard has been developed jointly with the IDF (International Dairy Federation) and the AOAC (Association of Official Analytical Chemists, U.S.A.) on the basis of an IDF Standard.

The text as approved by the above organizations was also published by the IDF (IDF Standard No. 17A) and by the AOAC (Official Methods of Analysis).

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Cheese – Determination of chloride content (Reference method)

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a reference method for the determination of the chloride content of cheese.

The method is applicable to all cheeses containing at least 0,5 % of chloride.

2 REFERENCE

ISO/R 707, *Milk and milk products – Sampling*.

3 DEFINITION

chloride content of cheese : The substances determined by the procedure specified. The chloride content may be expressed as a percentage by mass of Cl^- or sodium chloride or any other chloride used.

4 PRINCIPLE

Destruction of the organic matter of the cheese by means of potassium permanganate and nitric acid, and determination of the chloride content by argentimetric titration in nitric acid solution, in the presence of ammonium iron(III) sulphate as indicator.

5 REAGENTS

All reagents used shall be of analytical reagent quality.

5.1 Silver nitrate, approximately 0,1 N solution, standardized to the fourth decimal.

5.2 Potassium or ammonium thiocyanate, 0,1 N solution, standardized to the fourth decimal.

5.3 Ammonium iron(III) sulphate, saturated solution.

5.4 Nitric acid, ρ_{20} 1,40 to 1,42 g/ml, which corresponds to 66,9 to 71,6 % (m/m) HNO_3 .

5.5 Potassium permanganate, saturated solution.

5.6 Oxalic acid or glucose.

5.7 Water, not containing any impurity likely to affect the determination.

6 APPARATUS

6.1 Balance.

6.2 Conical flask, capacity 300 ml.

6.3 Pipette, calibrated to deliver 25 ml, conforming to ISO/R 648.

6.4 Graduated cylinders, capacities 15, 25 and 100 ml.

6.5 Burette, graduated in 0,1 ml, capacity 50 ml, conforming to ISO/R 385.

6.6 Suitable grinding device.

7 SAMPLING

See ISO/R 707.

8 PROCEDURE

8.1 Preparation of the test sample¹⁾

Before analysis, remove the rind or smear or mouldy surface layer of the cheese so as to give a test sample representative of the cheese such as it is usually consumed.

Grind the sample by means of an appropriate device (6.6); mix the ground mass quickly and grind if possible a second time and mix again thoroughly. Clean the grinding device after each sample. If the sample cannot be ground, mix it thoroughly by intensive kneading.

1) Special requirements for the preparation of the test sample of any type or variety of cheese might be laid down in national standards.

Transfer the test sample to an airtight container until the analysis, which shall be carried out on the same day. If delay is inevitable, take all precautions to ensure proper conservation and to prevent condensation of moisture on the inside surface of the container.

Cheese in brine shall be sampled by taking fragments of at least 200 g each along with sufficient brine to cover the cheese in the sample container. Prior to analysis, place the sample on filter paper for 1 to 2 h.

8.2 Test portion

Weigh, to the nearest 0,001 g, about 2 g of the test sample into the conical flask (6.2).

8.3 Determination

8.3.1 Add, by means of the pipette (6.3), 25 ml of silver nitrate solution (5.1), then add, by means of a graduated cylinder (6.4), 25 ml of nitric acid (5.4) and mix thoroughly.

8.3.2 Heat to boiling, add approximately 10 ml of potassium permanganate solution (5.5) and keep the reaction mixture boiling gently.

When the reaction mixture decolorizes, add more potassium permanganate solution; generally another 5 to 10 ml are sufficient. The presence of excess permanganate (brown colour) indicates that destruction of the organic matter is complete. Remove the excess by the addition of a small amount of oxalic acid or glucose (5.6).

8.3.3 Add 100 ml of cold water (5.7) and 2 ml of ammonium iron(III) sulphate solution (5.3) and mix thoroughly.

8.3.4 Immediately titrate the excess silver nitrate with the thiocyanate solution (5.2) until the solution shows a red-brown colour which persists for about 30 s.

8.3.5 Carry out a blank test using 2 ml of water in place of 2 g of cheese.

8.3.6 Carry out two determinations on the same test sample.

9 EXPRESSION OF RESULTS

9.1 Method of calculation and formula

Calculate the chloride content, as a percentage by mass, by means of the formula

$$\frac{(V_1 - V_2) \times f \times T}{m}$$

where

V_1 is the volume, in millilitres, of thiocyanate solution used for the blank test;

V_2 is the volume, in millilitres, of thiocyanate solution used for the test portion;

T is the normality of the thiocyanate solution;

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

f is the factor for expressing the result as a percentage of any chloride. The numerical values are, for example :

$f = 3,55$ for expression as % Cl^-

$f = 5,84$ for expression as % NaCl

$f = 7,46$ for expression as % KCl

Take as the result the arithmetic mean of the two determinations if the requirement concerning repeatability (9.2) is satisfied. Report the result to the second decimal place.

9.2 Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst shall not exceed 0,04 g of Cl^- (or the equivalent quantity of chloride used) per 100 g of the cheese.

10 TEST REPORT

The test report shall show the method used and the result obtained. It shall also mention all operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

The report shall include all details required for the complete identification of the sample.