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EAST AFRICAN STANDARD

Meat and meat products — Determination of chloride content — Part 1: Volhard method

EAST AFRICAN COMMUNITY

First Edition 2010

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 meet the above objectives, the EAC Partner States have enacted an East African Standardization, Quality Assurance, Metrology and Test Act, 2006 (EAC SQMT Act, 2006) to make provisions for ensuring standardization, quality assurance, metrology and testing of products produced or originating in a third country and traded in the Community in order to facilitate industrial development and trade as well as helping to protect the health and safety of society and the environment in the Community.

East African Standards are formulated in accordance with the procedures established by the East African Standards Committee. The East African Standards Committee is established under the provisions of Article 4 of the EAC SQMT Act, 2006. 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.

Article 15(1) of the EAC SQMT Act, 2006 provides that "Within six months of the declaration of an East African Standard, the Partner States shall adopt, without deviation from the approved text of the standard, the East African Standard as a national standard and withdraw any existing national standard with similar scope and purpose".

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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Introduction

In the preparation of this East African Standard, the following sources were consulted extensively:

ISO 1841-1:1996, *Meat and meat products — Determination of chloride content — Part 1: Volhard method*

Codex Alimentarius website: http://www.codexalimentarius.net/mrls/pestdes/jsp/pest_q-e.jsp

USDA Foreign Agricultural Service website: http://www.mrldatabase.com

USDA Agricultural Marketing Service website: http://www.ams.usda.gov/AMSv1.0/Standards

USDA Plant Inspectorate Service website: http://www.aphis.usda.gov/import_export/plants

European Union: http://ec.europa.eu/sanco_pesticides/public

Assistance derived from these sources is hereby acknowledged.

INTERNATIONAL STANDARD

ISO 1841-1

First edition
1996-07-01

Meat and meat products — Determination of chloride content —

Part 1: Volhard method

Viande et produits à base de viande — Détermination de la teneur en chlorures —

Partie 1: Méthode de Volhard

Reference number
ISO 1841-1:1996(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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 1841-1 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Subcommittee SC 6, *Meat and meat products*.

This first edition of ISO 1841-1 cancels and replaces ISO 1841:1981 which has been technically revised.

ISO 1841 consists of the following parts, under the general title *Meat and meat products — Determination of chloride content*:

— *Part 1: Volhard method*

— *Part 2: Potentiometric method*

Annex A of this part of ISO 1841 is for information only.

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Meat and meat products — Determination of chloride content —

Part 1:
Volhard method

1 Scope

This part of ISO 1841 specifies a method for the determination of the chloride content of meat and meat products, including poultry, with sodium chloride contents equal to or greater than 1,0 % (*m/m*).

2 Definition

For the purposes of this part of ISO 1841, the following definition applies.

2.1 chloride content of meat and meat products: Total chloride content determined by the method specified in this part of ISO 1841. It is expressed as sodium chloride as a percentage by mass.

3 Principle

Extraction of a test portion with hot water and precipitation of the proteins. After filtration and acidification, addition of an excess of silver nitrate solution to the extract, and titration of this excess with potassium thiocyanate solution.

4 Reagents

Use only reagents of recognized analytical grade unless otherwise specified.

4.1 Water, distilled and halogen-free.

Halogen-free test: Add 1 ml of silver nitrate [$c(AgNO_3)$ ≈ 0,1 mol/l] and 5 ml of nitric acid [$c(HNO_3)$ ≈ 4 mol/l] to 100 ml of water. No more than a slight turbidity shall be produced.

4.2 Nitrobenzene or **nonan-1-ol**.

4.3 Nitric acid, $c(HNO_3)$ ≈ 4 mol/l.

Mix 1 volume of concentrated nitric acid (1,39 g/ml ≤ ρ_{20} ≤ 1,42 g/ml) with 3 volumes of water.

4.4 Solutions for precipitation of proteins

4.4.1 Reagent A

Dissolve in water 106 g of potassium hexacyanoferrate(II) trihydrate [$K_4Fe(CN)_6 \cdot 3H_2O$]. Transfer quantitatively to a 1 000 ml one-mark volumetric flask (5.2) and dilute to the mark with water.

4.4.2 Reagent B

Dissolve in water 220 g of zinc acetate dihydrate [$Zn(CH_3COO)_2 \cdot 2H_2O$] and add 30 ml of glacial acetic acid. Transfer quantitatively to a 1 000 ml one-mark volumetric flask (5.2) and dilute to the mark with water.

4.5 Silver nitrate, standard volumetric solution, $c(AgNO_3)$ = 0,1 mol/l.

Dissolve in water 16,989 g of silver nitrate, previously dried for 2 h at 150 °C ± 2 °C and allowed to cool in a desiccator. Transfer quantitatively to a 1 000 ml one-mark volumetric flask (5.2) and dilute to the mark with water.

Store this solution in a dark glass container out of direct sunlight

4.6 Potassium thiocyanate, standard volumetric solution, $c(KSCN)$ = 0,1 mol/l.

Dissolve in water about 9,7 g of potassium thiocyanate. Transfer quantitatively to a 1 000 ml one-mark volumetric flask (5.2) and dilute to the mark with water. Standardize the solution to the nearest 0,000 1 mol/l against the silver nitrate solution (4.5) using the ammonium iron(III) sulfate solution (4.7) as indicator.

4.7 Ammonium iron(III) sulfate

Prepare a saturated aqueous solution at room temperature from the dodecahydrate $[NH_4Fe(SO_4)_2 \cdot 12H_2O]$.

5 Apparatus

Usual laboratory apparatus and, in particular, the following.

5.1 Homogenizing equipment, mechanical or electrical, capable of homogenizing the test sample. This includes a high-speed rotational cutter, or a mincer fitted with a plate with holes not exceeding 4,5 mm in diameter.

5.2 One-mark volumetric flasks, of capacity 1 000 ml and 200 ml.

5.3 Conical flasks, of capacity about 250 ml.

5.4 Burette, of capacity 25 ml or 50 ml.

5.5 One-mark pipettes, of capacity 20 ml.

5.6 Boiling water bath.

5.7 Analytical balance, capable of weighing to an accuracy of ± 0,001 g.

6 Sampling

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

Sampling is not part of the method specified in this part of ISO 1841. A recommended sampling method is given in ISO 3100-1.

Proceed from a representative sample of at least 200 g.

7 Preparation of test sample

7.1 Homogenize the laboratory sample with the appropriate equipment (5.1). Take care that the temperature of the sample material does not rise above 25 °C. If a mincer is used, pass the sample at least twice through the equipment.

7.2 Fill a suitable airtight container with the prepared sample. Close the container and store in such way that deterioration and change in composition are prevented. Analyse the sample as soon as practicable, but always within 24 h of homogenization.

8 Procedure

NOTE 1 If it is required to check whether the repeatability requirement is met, carry out two single determinations in accordance with 8.1 to 8.4 under repeatability conditions.

8.1 Test portion

Weigh, to the nearest 0,001 g, about 10 g of the test sample and transfer it quantitatively to a conical flask (5.3).

8.2 Deproteination

Add 100 ml of hot water (4.1) to the test portion (8.1). Heat the flask and its contents for 15 min in the boiling water bath (5.6). Periodically shake the contents of the flask.

Allow the flask and its contents to cool to room temperature, then add successively 2 ml of reagent A (4.4.1) and 2 ml of reagent B (4.4.2). Mix thoroughly after each addition.

Allow the flask to stand for 30 min at room temperature. Transfer the contents quantitatively to a 200 ml volumetric flask (5.2) and dilute to the mark with water. Mix the contents thoroughly and filter through a fluted filter paper.

NOTE 2 If this method is used for the determination of the nitrate and nitrite content or if ascorbic acid is present in the sample in concentrations higher than 0,1 %, it is necessary to add also 0,5 g of activated charcoal to the test portion (8.1). After mixing reagents A and B, adjust the pH to between 7,5 and 8,3 by means of a sodium hydroxide solution.

8.3 Determination

Transfer 20 ml of the filtrate to a conical flask (5.3) by means of a pipette (5.5) and add, from a graduated measuring cylinder, 5 ml of the dilute nitric acid (4.3) and 1 ml of the ammonium iron(III) sulfate solution (4.7) as indicator.

Transfer 20 ml of the silver nitrate solution (4.5) to the conical flask by means of a pipette (5.5). Add 3 ml of the nitrobenzene or nonan-1-ol from a graduated measuring cylinder and mix thoroughly. Shake vigorously to coagulate the precipitate. Titrate the contents of the conical flask with the potassium thiocyanate

(4.6) until the appearance of a persistent pink coloration. Record the volume of the potassium thiocyanate solution required, to the nearest 0,05 ml.

8.4 Blank test

Carry out a blank test, in accordance with 8.2 and 8.3, using the same volume of silver nitrate solution (4.5).

9 Calculation

Calculate the chloride content of the sample from the following equation:

$$w_{Cl} = 0{,}058\,44(V_2 - V_1) \times \frac{200}{20} \times \frac{100}{m} \times c$$

$$= 58{,}44 \times \frac{V_2 - V_1}{m} \times c$$

where

w_{Cl} is the chloride content of the sample, expressed as sodium chloride as a percentage by mass;

V_1 is the volume, in millilitres, of the potassium thiocyanate solution (4.6) used in the determination (8.3);

V_2 is the volume, in millilitres, of the potassium thiocyanate solution (4.6) used in the blank test (8.4);

c is the concentration of the potassium thiocyanate solution (4.6), in moles per litre;

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

Report the result to the nearest 0,05 % (*m/m*).

10 Precision

The precision of the method has been established by an interlaboratory test (see reference [4]), carried out in accordance with ISO 5725. For the values obtained for the repeatability limit, r, and the reproducibility limit, R, a probability level of 95 % holds.

10.1 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, should not be greater than

0,15 % (*m/m*) for sodium chloride contents between 1,0 % and 2,0 %;

0,20 % (*m/m*) for sodium chloride contents greater than 2,0 %.

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, should not be greater than:

0,20 % (*m/m*) for sodium chloride contents between 1,0 and 2,0 %;

0,30 % (*m/m*) for sodium chloride contents greater than 2,0 %.

11 Test report

The test report shall specify:

— the method in accordance with which sampling was carried out, if known;

— the method used;

— the test result(s) obtained; and

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

It shall also mention all operating details not specified in this part of ISO 1841, or regarded as optional, together with details of any incidents which have influenced the test result(s).

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

Annex A
(informative)

Bibliography

[1] ISO 3100-1:1991, *Meat and meat products — Sampling and preparation of test samples — Part 1: Sampling.*

[2] ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests* (now withdrawn) was used to obtain the precision data.

[3] Amtliche Sammlung von Untersuchungsverfahren nach Par. 35 LMBG. *Bestimmung des Kochsalzgehaltes in Fleisch und Fleischerzeugnissen.* L 06.00-5, September 1980.

[4] BELJAARS, P.R and HORWITZ, W, Comparison of the Volhard and potentiometric methods for the determination of chloride in meat products: Collaborative study. *J. Assoc. Off. Anal. Chem.*, **68**, 1985, pp. 480-484.

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