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Meat and meat products — Determination of chloride content —

Part 2: Potentiometric method

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Partie 2: Méthode potentiométrique



Reference number
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Foreword

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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 1841-2 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Subcommittee SC 6, *Meat and meat products*.

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 2:

Potentiometric 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 0,25 % (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

Dispersion of a test portion with water. Acidification of an aliquot of the suspension and potentiometric titration with a silver nitrate solution using a silver electrode.

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 [$c(\text{AgNO}_3) \approx 0,1 \text{ mol/l}$] and 5 ml of nitric acid [$c(\text{HNO}_3) \approx 4 \text{ mol/l}$] to 100 ml of water. No more than a slight turbidity shall be produced.

4.2 Nitric acid, 1 + 49 (V/V) solution.

Dilute 20 ml of concentrated nitric acid ($\rho_{20} = 1,40 \text{ g/ml}$) to 1 000 ml with water.

4.3 Silver nitrate, standard volumetric solution, $c(\text{AgNO}_3) = 0,085 6 \text{ mol/l}$.

Dissolve in water 14,541 g of silver nitrate, previously dried for 2 h at $150 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$ and allowed to cool in a desiccator. Transfer quantitatively to a 1 000 ml one-mark volumetric flask and dilute to the mark with water.

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

4.4 Sodium chloride, standard volumetric solution, $c(\text{NaCl}) = 0,085 6 \text{ mol/l}$.

Dissolve in water 5,000 g of sodium chloride, previously dried for 2 h at $110 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$ and allowed to cool in a desiccator. Transfer quantitatively to a 1 000 ml one-mark volumetric flask and dilute to the mark with water.

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 Laboratory blender, equipped with a variable transformer for blending at low and high rotational frequencies.

5.3 Electrodes, silver billet combination electrode, or separate indicating silver and glass reference electrodes.

Before initial use and before each day's use, if necessary, clean the silver billet electrode tip with scouring powder or another suitable material and rinse thoroughly with water (hot water may be required with some types of samples). Clean other electrodes as recommended by the manufacturer. Reclean as necessary to prevent drifting of the endpoint reading. With some samples, periodically rinse the electrodes with water and wipe with a tissue to prevent accumulation of film. It is unnecessary to coat silver billet electrodes with silver chloride.

5.4 Magnetic stirrer, capable of being adjusted to a constant rotational frequency.

5.5 pH-meter, preferably direct reading, with scale divisions 10 mV or less; range at least ± 700 mV, e.g. digital type.

5.6 Pipette, of capacity 50 ml.

5.7 Analytical balance, capable of weighing to an accuracy of $\pm 0,1$ 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 0,1 g, about 50 g of the test sample in a 1 000 ml blender jar.

8.2 Dispersion

8.2.1 Add 450 g of water (4.1) to the test portion (8.1). Cover the jar and start the blender (5.2) at low rotational frequency for the initial dispersion. Blend thoroughly at a high speed for 1 min to 2 min so that the solid material is uniformly suspended.

8.2.2 Pipette (5.6), immediately after blending, 50 ml of the thoroughly mixed sample suspension into a tared 250 ml beaker. Determine the mass of the test solution. Proceed as described in 8.4.

8.3 Titration curve

8.3.1 Pipette 25 ml of the sodium chloride solution (4.4) into a 250 ml beaker. Dilute to about 50 ml with the water (4.1) and add 50 ml of the dilute nitric acid (4.2).

8.3.2 Insert the electrodes (5.3) into the solution and start mixing. Stir throughout the titration at a constant rate producing vigorous agitation without splashing.

8.3.3 Titrate with the silver nitrate solution (4.3), adjusting increments with the rate of change in potential (see 5.5) so that an accurate plot of change in potential in millivolts (y-axis) against volume in millilitres of the silver nitrate solution (x-axis) can be prepared. Add a total of 50 ml of silver nitrate solution to obtain a complete titration curve.

8.3.4 Determine the inflection point by drawing two straight lines with a 45 ° slope with respect to the axes and tangent to the titration curve at two points of greatest curvature.

NOTE 2 The inflection point is at the intersection of the titration curve with the line drawn parallel to, and midway between, the other two lines.

8.3.5 Use the inflection point as the endpoint in titrating the sample solution (8.4). Recheck the endpoint potential occasionally with respect to a junction potential developing at the reference electrode. Redetermine the endpoint by preparing a new titration curve when either an individual electrode, a combination electrode, or the pH-meter is replaced.

8.3.6 From the volume of the silver nitrate solution used, calculate the concentration and adjust to 0,085 6 mol/l.

8.4 Determination

Add 50 ml of the dilute nitric acid (4.2) and titrate as described in 8.3 for the sodium chloride solution. Use a 10 ml burette if the chloride concentration is equal to or less than 1 %.

For the greatest accuracy, when a series of determinations on the same meat is performed, determine and use the endpoint from the titration curve (8.3) of that meat rather than using the endpoint obtained with sodium chloride standard solutions.

8.5 Blank test

Carry out a blank test, in accordance with 8.2.2 to 8.4 inclusive, substituting the water (4.1) for the sample suspension in 8.2.2.

9 Calculation

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

$$w_{\text{Cl}} = \frac{(V_1 - V_2) \times c \times 50 \times 58,44}{m_1 \times m}$$

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 silver nitrate solution (4.3) used in the determination (8.4);

V_2 is the volume, in millilitres, of the silver nitrate solution (4.3) used in the blank test (8.5);

c is the concentration, in moles per litre, of the silver nitrate solution (4.3);

m_1 is the mass, in grams, of the test solution (8.2.2);

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

Report the result to the nearest 0,1 % (m/m).

10 Precision

The precision of the method has been established by an interlaboratory test (see reference [5]), 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 the repeatability limit r as calculated using the following equation:

$$r = 0,002 + 0,033 \bar{w}_{\text{Cl}}$$

where \bar{w}_{Cl} is the mean of both results, as a percentage 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, should not be greater than the reproducibility limit R as calculated by the following equation:

$$R = 0,005 + 0,066 \bar{w}_{\text{Cl}}$$

where \bar{w}_{Cl} is the mean of both results, as a percentage by mass.

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 may 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

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