International Standard 5810

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Starches and derived products — Determination of chloride content — Potentiometric method

Amidons, fécules et produits dérivés — Détermination de la teneur en chlorures — Méthode potentiométrique

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Foreword

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Austria
Egypt, Arab Rep. of
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Portugal
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South Africa, Rep. of
USA
USSR

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Starches and derived products — Determination of chloride content — Potentiometric method

1 Scope and field of application

This International Standard specifies a potentiometric method for the determination of the chloride content of starches and derived products, except cationic starches and amyloids soluble when cold, the viscosity of these being too high to allow for correct stirring when titrating.

2 Principle

Potentiometric titration of a solution or suspension of the sample using a standard volumetric silver nitrate solution.

3 Reagents

During the analysis, use only reagents of recognized analytical quality and only distilled water or water of at least equivalent purity.

3.1 Nitric acid, concentrated, ϱ_{20} 1,41 g/ml, containing 70 % (*m/m*) of HNO_3.

3.2 Silver nitrate, standard volumetric solution, $c(AgNO_3)$ = 0,05 mol/l[1] or 0,02 mol/l[1].

4 Apparatus

Ordinary laboratory apparatus, and in particular

4.1 Beakers, of capacity 250 ml.

4.2 One-mark pipettes, of capacity 1 ml, complying with the requirements of ISO 648.

4.3 Burettes, of capacity 10 ml, complying with the requirements of ISO 385/2.

4.4 Analytical balance.

4.5 Potentiometer or **pH-meter,** the scale of which shall be graduated in millivolts, and calibrated according to the manufacturer's instructions.

4.6 Electrodes.

4.6.1 Silver/silver chloride electrode.

This may be purchased or may be prepared from a silver electrode as follows:

— Immerse the silver electrode in an approximately 0,1 mol/l[2] potassium chloride solution and connect it to the positive pole of a 4 V battery.

— Connect the negative pole to a second silver or platinum electrode and pass the current through for about 5 min until the surface of the positive electrode becomes dark in colour. Wash this positive electrode carefully with water and keep in water until required for use.

4.6.2 Reference electrode.

Use an appropriate electrode system for the potentiometric determination of chloride (such systems are commercially available).

4.7 Variable speed stirrer.

5 Procedure

5.1 Preparation of test sample

Mix the sample thoroughly in order to make it homogeneous.

5.2 Test portion

Weigh, to the nearest 0,001 g, a suitable mass of test sample selected, according to the expected chloride content, from the following table:

Expected chloride content % (*m/m*) NaCl	**Mass of test portion** g
less than 0,05	25
0,05 to 0,2	15
0,2 to 0,5	5
0,5 to 1	2,5
1 to 5	0,5

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[1] Hitherto designated "0,05 N and 0,02 N standard volumetric solutions" respectively.

[2] Hitherto designated "0,1 N solution".

5.3 Preparation of test solution or suspension

5.3.1 Transfer the test portion (5.2) to the titration beaker (4.1) containing 100 ml of water and the rod of the stirrer, while stirring.

5.3.2 For soluble products, stir until the test portion is dissolved, cool if necessary (bring to ambient temperature when titrating).

5.3.3 For insoluble products, stir until a homogeneous suspension is obtained and stir for a further 15 min.

5.4 Determination

Immerse the electrodes in the test solution or suspension (5.3), the silver/silver chloride electrode (4.6.1) being connected to the positive pole of the measuring apparatus (4.5) and the reference electrode (4.6.2) to the negative pole. Stir and add, by means of a pipette (4.2), 1 ml of the nitric acid (3.1).

Titrate the contents of the beaker with the silver nitrate solution (3.2)[1], adding amounts of 1 ml and 0,2 ml as the end-point approaches.

Allow the reading to stabilize after each addition.

Trace the potentiometric curve as a function of the volume of the silver nitrate solution added. Take as the end-point the point of inflection of the curve. Note the volume V of silver nitrate solution corresponding to the end-point.

Carry out two determinations on the same test sample (5.1).

6 Expression of results

6.1 Method of calculation and formulae

The chloride content, expressed as sodium chloride as a percentage by mass, is given by the formula

$$\frac{0{,}058\,45 \times c \times V \times 100}{m}$$

where

c is the exact concentration, in moles per litre, of the silver nitrate solution (3.2);

V is the volume, in millilitres, of the silver nitrate solution used for the determination (5.4);

m is the mass, in grams, of the test portion (5.2).

Take as result the arithmetic mean of the values obtained in two determinations provided that the conditions of repeatability (see 6.2) are fulfilled. If not, repeat the test.

6.2 Repeatability

The difference between the values obtained in two determinations carried out simultaneously or in rapid succession on the same sample shall not be more than

- 2,5 % of the arithmetic mean in the case of chloride contents greater than 1 % (*m*/*m*);
- 0,03 g per 100 g of product in the case of chloride contents less than 1 % (*m*/*m*).

6.3 Reproductibility

The difference between the values obtained in two determinations carried out on the same sample in two different laboratories shall not be more than

- 10 % of the arithmetic mean in the case of chloride contents greater than 1 % (*m*/*m*);
- 0,1 g per 100 g of product in the case of chloride contents less than 1 % (*m*/*m*).

7 Test report

The test report shall indicate the method used, together with the result obtained and the method of expression used. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances which may have affected the results.

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

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[1] It is preferable to use the 0,02 mol/l solution for chloride contents less than 0,05 % (*m*/*m*).