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Soaps — Determination of chloride content — Potentiometric method

Savons — Dosage des chlorures — Méthode potentiométrique

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 91, *Surface active agents*.

This second edition cancels and replaces the first edition (ISO 4323:1977), which has been technically revised.

The main changes compared to the previous edition are as follows:

- Replaced water with water + isopropanol (1 + 1) as solvent in determination.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Soaps — Determination of chloride content — Potentiometric method

1 Scope

This document specifies a potentiometric method for the determination of the chloride content of soaps, containing or not containing other surface active agents, and also of compounded products.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 385, *Laboratory glassware — Burettes*

ISO 8212, *Soaps and detergents — Techniques of sampling during manufacture*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
chloride content of soap products
mass fraction of chloride, expressed as sodium chloride and in percent, determined in accordance with the procedure described in this document

4 Principle

Potentiometric titration of the chloride (Cl^-) ions with standard silver nitrate solution in an acidic medium, by using either a silver ion selective electrode plus a reference electrode or a combined electrode.

5 Reagents

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

5.1 Nitric acid, approximately 6 mol/l solution.

5.2 Silver nitrate, approximately 0,1 mol/l solution.

Dissolve 8,5 g of silver nitrate in water in a 500 ml one-mark volumetric flask, dilute to the mark and mix. Store this solution capped in a dark amber-coloured flask. Standardize the solution before use (8.2).

5.3 **Silver nitrate**, approximately 0,01 mol/l solution.

Prepare this solution immediately before use by appropriate dilution of the standard silver nitrate solution (5.2).

5.4 **Sodium chloride**, 0,1 mol/l standard reference solution.

Weigh, to the nearest 0,001 g, 2,922 g of primary standard sodium chloride, previously dried for 2 h at 105 °C and cooled in a desiccator. Dissolve in a small quantity of water and transfer quantitatively to a 500 ml one-mark volumetric flask. Dilute to the mark and mix.

Potassium chloride can be used instead of sodium chloride but the weight of the chemical should be adjusted accordingly.

5.5 **Sodium chloride**, 0,01 mol/l standard reference solution.

Prepare this solution immediately before use by appropriate dilution of the standard reference sodium chloride solution (5.4).

5.6 **Isopropanol**.

6 Apparatus

Ordinary laboratory apparatus, and

6.1 **Potentiometer**, sensitivity 2 mV, covering the range −500 mV to +500 mV. Or an **autotitrator**.

6.2 **Electrode**, silver Ion Selective Electrode (ISE) with double junction reference electrode (10 % KNO_3 in outer junction and saturated AgCl in inner junction) or a combined electrode.

6.3 **Electromagnetic stirrer**.

6.4 **Burette**, capacity 50 ml, in accordance with the requirements of ISO 385, Class A.

6.5 **Cheese grater or other similar grinder**.

7 Sampling

The sampling shall be done in accordance with ISO 8212. The soap bar should be grated with a cheese grater (6.5). At least half of the bar should be grated to ensure a complete representation of the bar. The grated soap sample should be kept in an air-tight container to avoid moisture loss.

8 Procedure

8.1 Measurement temperature

In order to reduce the effects of thermal and electric hysteresis, take care that the temperatures of the electrodes, the water used for washings, the standard solutions and the test solution are as close to each other as possible. The temperatures of the standard solutions and the test solution shall not differ by more than 1 °C. The measurement temperature should be 25 °C whenever possible.

8.2 Calibration of the silver nitrate solution

8.2.1 Titration

Take 5,00 ml and 10,00 ml respectively the appropriate standard reference sodium chloride solution (5.4 or 5.5) and place in two clean, dry vessels of convenient capacity (for example 150 ml). Carry out the following titration on the contents of each vessel.

After acidification by the nitric acid solution (5.1), add sufficient water to bring the total volume to about 100 ml.

Maintain the sample at a lower pH can avoid the silver ion interact with OH^- to form Silver Oxide (Ag_2O) precipitation, minimize other anionic ions (such as fatty acid) interference with the AgCl formation, and improve the binding between the titrant and the analyte.

Stir the resultant solution and immerse the combined electrode or the silver ion selective electrode and a reference electrode (6.2), connect the electrode to the potentiometer (6.1) and, after having verified the zero of the apparatus, note the value of the starting potential.

Add, from the burette (6.4), in 1 ml portions, the silver nitrate solution (5.2 or 5.3) having the same normality as that of the standard reference sodium chloride solution (5.4 or 5.5) used. After each addition, await the stabilization of the potential.

Record the volumes added and the corresponding values of the potential in the first two columns of a table (see Table A.1.).

When approaching the end-point, continue the addition of the silver nitrate solution in portions of 0,1 ml for the 0,01 mol/l solution or 0,05 ml for the 0,1 mol/l solution.

In a third column of the table, record the successive increments $\Delta_1 E$ of the potential E.

In a forth column, record the differences $\Delta_2 E$, positive or negative, between the potential increments $\Delta_1 E$.

The end of the titration corresponds to the addition of the 0,1 ml or 0,05 ml portion of the silver nitrate solution which gives the maximum value of $\Delta_1 E$.

In order to calculate the exact volume V_{EQ} of the silver nitrate solution corresponding to the end of the reaction, use the formula:

$$V_{EQ} = V_0 + V_1 \times \frac{b}{B}$$

where

V_0 is the volume, in millilitres, of the silver nitrate solution (5.2 or 5.3), immediately lower than the volume which gives the maximum increment of $\Delta_1 E$;

V_1 is the volume, in millilitres, of the final portion of silver nitrate solution (5.2 or 5.3) added (0,05 or 0,1 ml respectively);

b is the last value of $\Delta_2 E$ which is positive;

B is the sum of the absolute values of the final positive value of $\Delta_2 E$ and the first negative value of $\Delta_2 E$ (see Annex A for additional information).

If the experiment is done by an autotitrator, the instrument will automatically display the V_{EQ} value after the end point is reached.

8.2.2 Calculation of normality of solution

The normality T of the silver nitrate solution is given by the formula:

$$T = T_0 \times \frac{5}{V_2 - V_3}$$

where

T_0 is the normality of the standard reference sodium chloride solution (5.4 or 5.5);

V_2 is the value, in millilitres, of V_{EQ}, corresponding to the titration of 10 ml of the standard reference sodium chloride solution (5.4 or 5.5);

V_3 is the value, in millilitres, of V_{EQ}, corresponding to the titration of 5 ml of the standard reference sodium chloride solution (5.4 or 5.5);

5 is the difference, in millilitres, between the two volumes of standard reference sodium chloride solution (5.4 or 5.5) used.

8.3 Determination

8.3.1 Test portion

Select the reagent solutions and test portion according to the expected chloride content, as indicated in Table 1.

Table 1 — Selection of the reagent solutions and test portion

Expected chloride content w %	Silver nitrate solution	Mass of test portion
Below 0,1	0,01 mol/l (5.3)	1 g to 10 g
Above 0,1	0,1 mol/l (5.2)	1 g to 3 g

Weigh, to the nearest 0,001 g, the appropriate test portion into a titration vessel. Add 50 ml of water and 50 ml of isopropanol (5.6). The sample solution is stirred and warmed until all soap particles are dissolved. Add 5 ml of nitric acid solution (5.1) and cool the test solution to 25 °C.

NOTE It is the responsibility of the analyst to validate the water system works to the samples if only water is used as solvent.

8.3.2 Titration

Titrate with the silver nitrate solution (5.2 or 5.3) corresponding to the expected chloride content and note the end-point of the reaction in accordance with the instructions given in 8.2. Calculate the V_{EQ} in accordance with the formula in 8.2.1.

If the experiment is done by an autotitrator, the instrument will automatically display the V_{EQ} value after the end point is reached.

8.3.3 Blank test

Carry out a blank test with 50 ml of water, 50 ml of isopropanol (5.6) and 5 ml of nitric acid solution (5.1), which is the same composition as that for the test portion described in 8.3.1.

9 Expression of results

Calculate the chloride content, expressed as a percentage by mass of sodium chloride, *w*, using the formula:

$$w = \frac{5{,}85 \times T \times (V_4 - V_5)}{m}$$

where

T is the normality of the silver nitrate solution, calculated in accordance with 8.2.2;

V_4 is the value, in millilitres, of V_{EQ}, corresponding to the determination by 8.3.2;

V_5 is the volume, in mllilitres, corresponding to the blank test (8.3.3);

m is the mass, in grams, of the test portion (see 8.3.1).

10 Test report

The test report shall include the following particulars:

a) all information necessary for the complete identification of the sample;

b) the reference of method used (reference to this document including its year of publication);

c) the results and the method of expression used;

d) the test conditions;

e) any operational details not specified in this document or the International Standard to which reference is made, or regarded as optional, as well as all incidents likely to have affected the results.