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Sodium chloride for industrial use — Determination of matter insoluble in water or in acid and preparation of principal solutions for other determinations

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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 2479 was drawn up by Technical Committee ISO/TC 47, *Chemistry*.

It was approved in January 1972 by the Member Bodies of the following countries :

Austria	Ireland	South Africa, Rep. of
Belgium	Italy	Spain
Chile	Korea, Dem.P.Rep. of	Switzerland
Czechoslovakia	Morocco	Thailand
Egypt, Arab Rep. of	Netherlands	United Kingdom
France	New Zealand	U.S.S.R.
Germany	Poland	
Hungary	Portugal	
India	Romania	

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INTERNATIONAL STANDARD ISO 2479-1972 (E)

Sodium chloride for industrial use — Determination of matter insoluble in water or in acid and preparation of principal solutions for other determinations

1 SCOPE

This International Standard specifies a method for determining insoluble matter in sodium chloride for industrial use.

It also describes the preparation of principal solutions for other determinations.

NOTE — Sodium chloride for industrial use may contain components that are only very slightly soluble or dissolve very slowly in water. Insoluble substances in sodium chloride for industrial use shall therefore be defined by the conditions of determination described in this International Standard. These conditions reproduce those normally used during the handling of salt for industrial use. The object is not to dissolve all the impurities but only those of interest to users.

2 FIELD OF APPLICATION

2.1 General case

The method is applicable to the analysis of sodium chloride for industrial use, carried out in aqueous solution.

2.2 Special case

Determination of insoluble matter in an acid medium.

NOTE — Whatever the conditions adopted, all related determinations shall be carried out in the same medium, except the determination of chlorides which shall always be carried out in an aqueous solution.

3 PRINCIPLE

Solution of a test portion in water. Filtration, drying and weighing of the insoluble residue.

Dilution of the filtrate to form the principal solution (solution A) for carrying out other determinations.

4 REAGENT

Distilled water, or water of equivalent purity, shall be used in the test.

4.1 Silver nitrate, 5 g/l nitric solution.

Dissolve 0,5 g of silver nitrate in a little water, add 10 ml of nitric acid solution ρ 1,40 g/ml approximately, and dilute to 100 ml.

1) In preparation.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Filter crucible or funnel, glass or porcelain, approximately 30 mm diameter and of a porosity grade P 10 or P 16 (pore size index 4-16 μm).

5.2 Electric oven, ventilated by convection and capable of being controlled at 110 ± 2 °C.

5.3 Desiccator, containing silica gel, phosphorus pentoxide or a molecular sieve.

6 SAMPLING AND SAMPLES

For methods of sampling and the number of samples to be taken for a given quantity of product, the procedure described in ISO. . .[1] shall be followed.

7 PROCEDURE

7.1 Test portion

Weigh, to the nearest 0,01 g, approximately 100 g of the test sample.

7.2 Determination

Place the test portion (7.1) in a 600 ml beaker and add 350 ml of water. Heat at just below boiling for 10 min, with stirring, and then transfer the beaker, covered with a watch glass, to a boiling water bath for 30 min. Cool to approximately 20 °C.

Filter by vacuum on the filter crucible (5.1), previously dried at 110 °C, cooled in the desiccator (5.3), and weighed to the nearest 0,1 mg.

Then wash the insoluble matter, in groups of five successive washings, using 20 ml of water each time, disconnecting the vacuum after each washing in order to bring the insoluble matter into suspension for approximately 1 min before filtering, and checking for absence of chloride from the filtrate after the fifth, tenth or fifteenth washing. 10 ml of the washing water shall remain clear 5 min after adding 10 ml of the nitric silver nitrate solution (4.1). Cease washing as soon as the check indicates absence of chlorides.

Dry the crucible and its contents in the oven (5.2) controlled at 110 ± 2 °C for 1 h, cool in the desiccator (5.3) and weigh to the nearest 0,1 mg. Repeat this operation until two weighings differ by not more than 0,2 mg.

NOTE — If the insoluble matter is so finely divided as to clog the filter, repeat the determination and add, after the 350 ml of water, 1,5 g, weighed to the nearest 0,1 mg, of a filter aid (kieselguhr) of analytical quality. The minimum particle size of the filter aid should be 15 μm and it should be dried, at about 110 °C, to constant mass before use.

7.3 Preparation of the principal solution for other determinations (solution A)

Quantitatively transfer the filtrate obtained, after filtering and washing of the insoluble matter, to a 1 000 ml one-mark volumetric flask. Dilute to the mark and mix.

Keep this solution for the other determinations.

8 EXPRESSION OF RESULTS

8.1 Method of calculation and formula

The matter insoluble in water is given, as a percentage by mass, by the formula :

$$(m_1 - m_2) \times \frac{100}{m_0}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the filter crucible and insoluble matter;

m_2 is the mass, in grams, of the filter crucible alone.

NOTE — If a filter aid has been used, deduct its mass from m_1 (see Note in 7.2).

8.2 Repeatability and reproducibility

Comparative analyses on two samples in nineteen laboratories have given the following statistical results :

		Evaporated salt	Marine salt
Mean (percentage by mass)		0,001	0,044
Standard deviation	for repeatability (σ_r)	0,000 5	0,002
	for reproducibility (σ_R)	0,000 8	0,008

9 SPECIAL CASE : Determination of insoluble matter in an acid medium

9.1 Principle

Solution of a test portion in an acid medium. Filtration, drying and weighing of the insoluble residue.

Dilution of the filtrate to form the principal solution (solution B) for carrying out other determinations, except the determination of chlorides.

9.2 Reagents

In addition to water use :

9.2.1 Hydrochloric acid, N standard volumetric solution.

9.2.2 Sodium hydroxide, N standard volumetric solution.

9.2.3 Methyl orange, 0,5 g/l solution.

9.3 Apparatus

See section 5.

9.4 Sampling and samples

See section 6.

9.5 Procedure

9.5.1 *Test portion*

Weigh, to the nearest 0,01 g, two test portions of 100 ± 0,1 g from the test sample.

9.5.2 *Determination of total alkalinity*

Place the first test portion (9.5.1) into a 600 ml beaker. Add 300 ml of water and 50,00 ml of the hydrochloric acid standard volumetric solution (9.2.1). Bring to the boil, maintain boiling for about 10 min and cool to approximately 20 °C. Transfer quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Filter slightly more than 100 ml of the solution through a fluted filter paper of a fine grade and reject the first portions of the filtrate. Pipette 100 ml of filtrate into a 250 ml conical flask. Add 3 drops of methyl orange standard volumetric solution (9.2.3) and back-titrate with the sodium hydroxide solution (9.2.2) to the colour change from red to yellow. Record the volume, V, of sodium hydroxide solution used.

9.5.3 *Determination*

Place the second test portion (9.5.1) into a 600 ml beaker. Add 300 ml of water, $(50 - 5V + x)$ ml[1] of the hydrochloric acid standard volumetric solution (9.2.1) and dilute to approximately 350 ml. Proceed as described in clause 7.2, starting at the second sentence : "Heat at just below boiling for 10 min. . ."

1) The value of x is to be the subject of agreement between the interested parties.

9.5.4 *Preparation of principal solution for the determinations (solution B)*

Proceed as described in 7.3, but using the acid filtrate.

9.6 Expression of results

Matter insoluble in the acidic medium is given, as a percentage by mass, by the formula in 8.1.

10 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard or regarded as optional.