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Sodium chloride for industrial use — Determination of halogens, expressed as chlorine — Mercurimetric method

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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.

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

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

No Member Body expressed disapproval of the document.

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INTERNATIONAL STANDARD ISO 2481-1973 (E)

Sodium chloride for industrial use — Determination of halogens, expressed as chlorine — Mercurimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a mercurimetric method for the determination of halogens expressed as chlorine, in sodium chloride for industrial use.

2 REFERENCE

ISO 2479, *Sodium chloride for industrial use — Determination of matter insoluble in water or acid and preparation of principal solutions for other determinations.*

3 PRINCIPLE

Titration of the halogen ions with mercury(II) nitrate in the presence of diphenylcarbazone as indicator.

4 REAGENTS

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

4.1 Nitric acid, ρ 1,40 g/ml, approximately 68 % (*m/m*) or approximately 14 N solution.

4.2 Nitric acid, approximately 2 N solution.

4.3 Sodium chloride, 0,1 N standard reference solution.

Weigh, to the nearest 0,1 mg, 5,844 3 g of sodium chloride, previously dried for 1 h at 500 °C and cooled in a desiccator. Dissolve it in water in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

4.4 Standard end-point matching solution

Prepare this standard solution immediately before use. Pour 200 ml of water into a 500 ml conical flask, followed by 3 drops of bromophenol blue solution (4.6) and the nitric acid solution (4.2), added drop by drop until the colour changes from blue to yellow. Add an excess of 3 drops of this acid, between 0,5 and 1,0 ml of the diphenylcarbazone solution (4.7) and the volume of mercury(II) nitrate solution (4.5) (from a burette) necessary to change the colour of the solution from yellow to mauve (about 1 drop).

4.5 Mercury(II) nitrate, 0,1 N standard volumetric solution.

4.5.1 *Preparation of the solution*

Weigh 10,85 ± 0,01 g of mercury(II) oxide (HgO) and dissolve it in 10 ml of the nitric acid solution (4.1), in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

Standardize this solution, following the procedure described in 4.5.2, adjusting it to the exact concentration if necessary.

4.5.2 *Standardization of the solution*

Transfer 40,0 ml of the sodium chloride standard reference solution (4.3) to a 500 ml conical flask, followed by 160 ml of water and 3 drops of the bromophenol blue solution (4.6). Add the nitric acid solution (4.2) drop by drop until the colour of the indicator changes from blue to yellow. Add an excess of 3 drops of this acid and same volume of the diphenylcarbazone solution (4.7) as in the standard end-point matching solution (4.4). Titrate the chloride with the mercury(II) nitrate solution to be standardized (4.5.1) until the colour matches the mauve of the standard end-point matching solution (4.4) and deduct the volume of mercury(II) nitrate solution (4.5.1) added during the preparation of this standard end-point matching solution (about 1 drop).

The correct amount is 40,00 ml.

4.6 Bromophenol blue, 1 g/l solution in 95 % (*V/V*) ethanol.

4.7 Diphenylcarbazone, 5 g/l solution in 95 % (*V/V*) ethanol.

Store this solution in a refrigerator and replace it when it no longer gives a sharp colour change.

5 APPARATUS

Ordinary laboratory apparatus.

1) See clause 7.3 in ISO 2479.

6 PROCEDURE

6.1 Test portion

Take 50,0 ml of the principal solution A[1], containing 100 g of the test sample per 1 000 ml.

6.2 Determination

6.2.1 *Preparation of the sample solution*

Place the test portion (6.1) in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

6.2.2 *Titration*

Place 25,0 ml of the sample solution (6.2.1) in a 500 ml conical flask. Dilute to 200 ml, then add 3 drops of the bromophenol blue solution (4.6) and the nitric acid solution (4.2) drop by drop, until the colour changes from blue to yellow. Add a further 3 drops of this acid and the same volume of the diphenylcarbazone solution (4.7) as in the standard end-point matching solution (4.4).

Titrate with the mercury(II) nitrate solution (4.5) until the colour matches the mauve of the standard end-point matching solution (4.4).

7 EXPRESSION OF RESULTS

7.1 Method of calculation and formula

The halogens content, expressed as chlorine (Cl), is given, as a percentage by mass, by the formula :

$$(V-V_1) \times \frac{500}{25} \times \frac{1\,000}{50} \times \frac{100}{m} \times 0{,}003\,545 = \frac{141{,}8\,(V-V_1)}{m}$$

where

m is the mass, in grams, of the test portion used for preparing the principal solution A[1];

V is the volume, in millilitres, of the standard volumetric mercury(II) nitrate solution (4.5) used for titration;

V_1 is the volume, in millilitres, of the standard volumetric mercury(II) nitrate solution (4.5) used in the preparation of the standard end-point matching solution;

0,003 545 is the mass, in grams, of chlorine corresponding to 1 ml of the standard volumetric mercury(II) nitrate solution (4.5).

7.2 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)		60,60	59,73
Standard deviation	for repeatability (σ_r)	0,06	0,06
	for reproducibility (σ_R)	0,26	0,28

8 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.

1) See clause 7.3 in ISO 2479.