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Sodium chloride for industrial use — Determination of calcium and magnesium contents — EDTA complexometric methods

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FOREWORD

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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
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INTERNATIONAL STANDARD ISO 2482-1973 (E)

Sodium chloride for industrial use — Determination of calcium and magnesium contents — EDTA complexometric methods

1 SCOPE

This International Standard specifies complexometric methods for determining the calcium and magnesium contents in sodium chloride for industrial use.

2 FIELD OF APPLICATION

2.1 General case

The method is applicable to the determination of water-soluble calcium and magnesium, in sodium chloride for industrial use, for calcium or magnesium contents greater than 0,001 % (*m/m*).

2.2 Special case

Determination of calcium and magnesium in a principal solution prepared 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 REFERENCE

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

4 PRINCIPLE

Dissolution of a test portion and separation of insoluble matter.

Titration of the filtrate obtained according to ISO 2479, of calcium on the one hand and total (calcium + magnesium) on the other, using *di*sodium ethylenediaminetetra-acetate (EDTA) in the presence of glyoxal-bis-(2-hydroxyanil) (GBHA) and mordant black 11 (CI 14645) respectively as indicators.

5 REAGENTS

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

5.1 Sodium hydroxide, ρ 1,08 g/ml, approximately 7,5 % (*m/m*) solution or approximately 2 N.

Dissolve 80 g of sodium hydroxide in water and dilute to 1 000 ml.

5.2 Ethanol, 95 % (*V/V*).

Alcohol denatured with acetone, but not coloured, can also be used.

5.3 Triethanolamine, 25 % (*V/V*) solution.

Place 25 ml of triethanolamine in a 100 ml one-mark volumetric flask, dilute to the mark and mix.

5.4 Buffer solution

Dissolve 54 g of ammonium chloride in water. Add 350 ml of ammonia solution (ρ 0,920 g/ml) and dilute to the mark in a 1 000 ml one-mark volumetric flask.

5.5 Standard end-point matching solution

Prepare this standard solution immediately before the determination (7.1.2). Pour 50 ml of water into a 250 ml conical flask followed by 2 ml of the triethanolamine solution (5.3), 10 ml of the sodium hydroxide solution (5.1), 15 ml of ethanol (5.2) and 1,0 ml of the glyoxal-bis-(2-hydroxyanil) solution (5.8).

5.6 *di*Sodium ethylenediaminetetra-acetate (EDTA), 0,02 M standard volumetric solution

Weigh, to the nearest 0,1 mg, 7,445 g of the EDTA and dissolve this in water. Transfer quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

5.7 *di*Sodium ethylenediaminetetra-acetate (EDTA), 0,002 M standard volumetric solution.

Take 100,0 ml of the 0,02 M EDTA solution (5.6) and place in a 1 000 ml one-mark volumetric flask; dilute to the mark and mix.

5.8 Glyoxal-bis-(2-hydroxyanil) (GBHA), ethanolic solution approximately 2,5 g/l.

Place approximately 0,25 g of GBHA in a 100 ml brown glass flask, fitted with a ground glass stopper. Add 100 ml of the ethanol (5.2) and stir until dissolved.

5.9 Mordant black 11[1] (CI 14645) approximately 0,4 % (*m/m*) mixture.

Intimately mix 0,4 g of mordant black 11 with 100 g of sodium chloride, taking care to make the mixture perfectly homogeneous.

6 APPARATUS

Ordinary laboratory apparatus.

7 PROCEDURE

7.1 Calcium determination

7.1.1 *Test portion*

Take 50,0 ml of the principal solution A[2] containing 100 g of test sample per 1 000 ml.

7.1.2 *Determination*

Place the test portion (7.1.1) in a 250 ml conical flask. Add, successively, 2 ml of the triethanolamine solution (5.3), 10 ml of the sodium hydroxide solution (5.1), 15 ml of ethanol (5.2) and 1,0 ml of the glyoxal-bis-(2-hydroxyanil) solution (5.8). The pH of the solution should then be higher than 12. Otherwise add a quantity of sodium hydroxide solution (5.1) sufficient to raise the pH above this level. Wait for approximately 2 min and then titrate with the EDTA solution (5.6) until the colour changes from red to pure yellow, corresponding to the colour of the standard end-point matching solution (5.5). Titration should be carried out in less than 3 min.

7.2 Total determination of calcium + magnesium

7.2.1 *Test portion*

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

7.2.2 *Determination*

Place the test portion (7.2.1) into a 500 ml conical flask. Dilute to approximately 200 ml. Add 2 ml of the triethanolamine solution (5.3), then 5 ml of the buffer solution (5.4) and slightly less than twice the volume of EDTA solution (5.6) used for the calcium determination.

Heat to 50 °C and add approximately 0,1 g of mordant black 11 (5.9).

Continue to add the EDTA solution (5.6), which should be done slowly, particularly near the end point, until the colour changes from wine red to true blue (without a violet tint).

NOTES

1 If the solution has a blue colour after the addition of the mordant black 11 (5.9), it is advisable, before continuing the titration, to add 6 drops of a solution containing 5 g/100 ml of magnesium ethylenediaminetetra-acetate (Mg EDTA) to change the colour of the indicator to red.

2 If the titrations referred to in 7.1 and 7.2 require more than 50 ml of the EDTA solution (5.6) reduce the mass of the test portion and take this into account in calculating the results.

8 EXPRESSION OF RESULTS

8.1 Method of calculation and formulae

8.1.1 *Calcium content*

The water-soluble calcium content, is given, as a percentage by mass, by the formula :

$$V \times 0{,}02 \times 0{,}040\,08 \times \frac{1\,000}{50} \times \frac{100}{m} = 1{,}603\,2\,\frac{V}{m}$$

where

V is the volume, in millilitres, of EDTA solution (5.6) employed for the calcium determination;

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

$0{,}02 \times 0{,}040\,08$ is the mass, in grams, of calcium corresponding to 1 ml of EDTA solution (5.6).

8.1.2 *Magnesium content*

The water-soluble magnesium content is given, as a percentage by mass, by the formula :

$$(V_1 - 2V) \times 0{,}02 \times 0{,}024\,32 \times \frac{1\,000}{100} \times \frac{100}{m}$$

$$= \frac{(V_1 - 2V)}{m} \times 0{,}486\,4$$

where

V_1 is the volume, in millilitres, of EDTA solution (5.6) used for the total determination (calcium + magnesium);

$0{,}02 \times 0{,}024\,32$ is the mass, in grams, of magnesium corresponding to 1 ml of EDTA solution (5.6).

NOTE — If the calcium and magnesium contents are less than 0,02 % and 0,01 % (*m/m*) respectively, use the 0,002 M EDTA solution (5.7) and substitute 0,002 for 0,02 in the formulae.

1) One of the commercial names of this product is "Eriochrome black T".

2) See clause 7.3 of ISO 2479.

8.2 Repeatability and reproducibility

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

		Determination of Ca		Determination of Mg	
		Evaporated salt	Marine salt	Evaporated salt	Marine salt
Mean (percentage by mass)		0,014	0,048	0,001 4	0,006
Standard deviation	for repeatability (σ_r)	0,000 3	0,001 3	*	0,000 3
	for reproducibility (σ_R)	0,001 2	0,002 2	*	0,000 9

* These calculations could not be carried out because the results, which were very low, were not all given to the necessary fourth decimal place.

9 SPECIAL CASE : Determination of calcium and magnesium in a principal solution prepared in an acid medium.

9.1 Principle

Solution of the test portion in an acid medium and separation of the insoluble residue.

Titration of calcium ions and calcium + magnesium ions as in section 4.

9.2 Reagents

As described in section 5.

9.3 Apparatus

As described in section 6.

9.4 Procedure

9.4.1 *Calcium determination*

9.4.1.1 TEST PORTION

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

9.4.1.2 DETERMINATION

Proceed as described in 7.1.2.

9.4.2 *Total determination (calcium + magnesium)*

9.4.2.1 TEST PORTION

Take 100,0 ml of the solution B, containing 100 g of the test sample per 1 000 ml.

9.4.2.2 DETERMINATION

Proceed as described in 7.2.2.

9.5 Expression of results

9.5.1 *Calcium content*

Calcium content, soluble in an acid medium under the specified conditions, is given, as a percentage by mass, by the formula in 8.1.1, where V is the volume, in millilitres, of the EDTA solution (5.6) used for the calcium determination and m is the mass, in grams, of the test portion used for preparing solution B.

9.5.2 *Magnesium content*

Magnesium content, soluble in an acid medium under the specified conditions, is given, as a percentage by mass, by the formula in 8.1.2, where V_1 is the volume, in millilitres, of EDTA solution (5.6) used for the total determination (calcium + magnesium) and m is the mass, in grams, of the test portion used for preparing solution B.

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.

1) See clause 9.5.4 of ISO 2479.