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**Sodium hydrogen carbonate for industrial use —
Determination of chloride content — Mercurimetric method**

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FOREWORD

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Austria	Israel	Spain
Belgium	Italy	Switzerland
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No Member Body expressed disapproval of the document.

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Sodium hydrogen carbonate for industrial use — Determination of chloride content — Mercurimetric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a mercurimetric method for the determination of chlorides in sodium hydrogen carbonate for industrial use. This method is applicable to products with a chloride content, expressed as NaCl, greater than 0.001 % (m/m).

2 PRINCIPLE

Titration of the Cl^- ion with mercuric nitrate in the presence of diphenylcarbazone as indicator.

3 REAGENTS

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

3.1 Nitric acid, ρ 1.40 g/ml approximately, 68 % (m/m) solution or about 14 N.

3.2 Nitric acid, approximately 2 N solution.

3.3 Sodium hydroxide, approximately 2 N solution.

3.4 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 then cooled in a desiccator, dissolve in water in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

3.5 Standard matching solution for standardizing

Place in a 500 ml conical flask 200 ml of water and 3 drops of bromophenol blue solution (3.9), and add the nitric acid solution (3.2), drop by drop, until the colour changes from blue to yellow, plus an excess of 3 drops of the acid. Then add 1.0 ml of the diphenylcarbazone solution (3.10) and, from a burette, the volume of mercuric nitrate solution (3.7) necessary to change the colour of the solution from yellow to mauve (or 1 drop).

Prepare this standard matching solution when required for use.

3.6 Standard matching solution for the determination

Prepare the standard matching solution for the determination at the time of use, in the same way as the standard matching solution for standardizing (3.5) but finally adding the mer-

curic nitrate solution (3.8) so as to change the colour of the solution from yellow to mauve (or approximately 0.5 ml).

3.7 Mercuric nitrate, 0.1 N standard volumetric solution.

3.7.1 Preparation of the solution

Dissolve 10.85 g of mercuric oxide (HgO) in 10 ml of the nitric acid solution (3.1) and dilute to 1 000 ml with water. Standardize this solution, following the procedure described in 3.7.2 and adjusting it, if necessary, to the exact concentration.

3.7.2 Standardization of the solution

Place in a 500 ml conical flask 40.0 ml of the sodium chloride standard reference solution (3.4), 160 ml of water and 3 drops of the bromophenol blue solution (3.9). Add the nitric acid solution (3.2) drop by drop until the indicator changes colour from blue to yellow, add 3 drops of this acid in excess then 1.0 ml of the diphenylcarbazone solution (3.10). Titrate the chloride with the mercuric nitrate solution to be standardized (3.7.1) until the colour matches the standard matching solution for standardizing (3.5), and deduct the volume of mercuric nitrate solution (3.7.1) added when preparing this standard matching solution (about 1 drop).

The correct titration is 40.00 ml.

3.8 Mercuric nitrate, 0.01 N standard volumetric solution,

Take 100.0 ml of the mercuric nitrate solution (3.7), place in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

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

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

4 APPARATUS

Ordinary laboratory apparatus.

5 PROCEDURE

5.1 Test portion

Weigh, to the nearest 0.1 g, 40 g of the test sample.

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5.2 Preparation of the test solution.

Place the test portion (5.1) in a 500 ml conical flask. Add 100 ml of water, then, with caution, 30 ml of the nitric acid solution (3.1). Cool to room temperature, add 3 drops of the bromophenol blue solution (3.9) and continue adding the nitric acid (3.1) until the colour changes from blue to yellow. Add the sodium hydroxide solution (3.3) drop by drop until the colour changes to blue, then add the nitric acid solution (3.2) until the colour changes to yellow and, finally, add 3 drops of this acid in excess. Dilute to about 200 ml.

5.3 Titration

To the solution obtained (5.2) add 1.0 ml of the diphenylcarbazone solution (3.10) and titrate the chloride with the mercuric nitrate solution (3.8) until the colour matches that of the standard matching solution for the determination (3.6).

6 EXPRESSION OF RESULTS

The chloride content, expressed as sodium chloride (NaCl), is given, as a percentage by mass, by the formula :

$$(V - V_1) \times \frac{100}{m} \times 0.000\ 584\ 5 = \frac{0.058\ 45 (V - V_1)}{m}$$

where

V is the volume, in millilitres, of the standard volumetric mercuric nitrate solution (3.8) used for the titration :

V_1 is the volume, in millilitres, of standard volumetric mercuric nitrate solution (3.8), used for the standard matching solution for the determination (3.6):

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

0.000 584 5 is the mass, in grams, of sodium chloride corresponding to 1 ml of the standard volumetric mercuric nitrate solution (3.8).

Express the result to three places of decimals.

7 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.