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Sodium chlorate for industrial use — Determination of chloride content — 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 February 1972 by the Member Bodies of the following countries :

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

No Member Body expressed disapproval of the document.

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Sodium chlorate for industrial use — Determination of chloride content — Mercurimetric method

1 SCOPE

This International Standard specifies a mercurimetric method for the determination of the chloride content of sodium chlorate for industrial use.

2 FIELD OF APPLICATION

The method is applicable to products with chloride content greater than 0,01 % (*m/m*), expressed as sodium chloride. The method is not applicable to the analysis of mixtures based on sodium chlorate, such as herbicides, insecticides, etc.

3 PRINCIPLE

Titration of the Cl^- ion 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 approximately 1,40 g/ml, about 68 % (*m/m*) solution or approximately 14 N.

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 matching solution immediately before use. Pour 200 ml of water into a 500 ml conical flask, add 3 drops of the bromophenol blue solution (4.6) and then the nitric acid solution (4.2) drop by drop, until the colour changes from blue to yellow. Add an excess of 3 drops of this solution, 0,5 to 1,0 ml of the diphenylcarbazone solution (4.7) and the volume of the standard volumetric 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 then a volume of the diphenylcarbazone solution (4.7) identical to that added for 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 the mercury(II) nitrate solution (4.5.1) added during the preparation of this standard end-point matching solution (about 1 drop).

The volume corresponding to the specified concentration 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 definite colour change.

5 APPARATUS

Ordinary laboratory apparatus.