# INTERNATIONAL STANDARD 3199

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## Sodium chlorate for industrial use — Determination of chlorate content — Dichromate titrimetric method

*Chlorate de sodium à usage industriel — Dosage du chlorate — Méthode titrimétrique au dichromate*

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## FOREWORD

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International Standard ISO 3199 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in September 1973.

It has been approved by the Member Bodies of the following countries :

| | | |
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| Austria | India | South Africa, Rep. of |
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No Member Body expressed disapproval of the document.



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# Sodium chlorate for industrial use — Determination of chlorate content — Dichromate titrimetric method

## 1 SCOPE AND FIELD OF APPLICATION

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

The method is not applicable to the analysis of mixtures for agricultural use such as herbicides, pesticides, etc., in which certain constituents may cause interference.

## 2 PRINCIPLE

Reduction of the chlorate by a known quantity, in excess, of an iron(II) salt.

Titration of the excess of iron(II) salt with a standard volumetric solution of potassium dichromate in the presence of barium diphenylamine-4-sulphonate solution as indicator.

## 3 REAGENTS

During the analysis, use only reagents of recognized analytical reagent grade and only distilled water or water of equivalent purity.

**3.1 Orthophosphoric acid,** $\rho$ approximately 1,71 g/ml, about 85 % (*m/m*) solution.

**3.2 Sulphuric acid,** $\rho$ approximately 1,84 g/ml, about 96 % (*m/m*) solution or approximately 36 N.

**3.3 Sulphuric acid,** approximately 18 N solution.

**3.4 Iron(II) sulphate,** approximately 0,1 N solution.

Weigh, to the nearest 0,01 g, 39,2 g of ammonium iron(II) sulphate hexahydrate $[(NH_4)_2Fe(SO_4)_2.6H_2O]$, dissolve in 500 ml of water, slowly add 40 ml of the sulphuric acid solution (3.3), cool, dilute to the mark in a 1 000 ml one-mark volumetric flask and mix.

Store this solution in a glass bottle fitted with a ground glass stopper.

**3.5 Potassium dichromate,** 0,1 N standard volumetric solution.

Weigh, to the nearest 0,000 1 g, 4,903 g of potassium dichromate, previously dried at about 200 °C for about 16 h and cooled in a desiccator. Dissolve in water, dilute to the mark in a 1 000 ml one-mark volumetric flask and mix.

Store this solution in a glass bottle fitted with a ground glass stopper.

**3.6 Barium diphenylamine-4-sulphonate,** 4 g/l solution.

## 4 APPARATUS

Ordinary laboratory apparatus.

## 5 PROCEDURE

### 5.1 WARNING

**Sodium chlorate induces combustion. Avoid storing or handling it near a source of heat. Also avoid all contact of the product or its solutions with acids or combustible materials (paper, cardboard, clothing, wood, rags, fatty matter, etc.) which may ignite or subsequantly form an explosive mixture. If any materials are accidentally impregnated with sodium chlorate, flush them with water.**

### 5.2 Test portion

Weigh, to the nearest 0,001 g, approximately 3 g of the test sample.

### 5.3 Blank test

Carry out at the same time as the determination, following the same procedure, a blank test using the same quantities of all the reagents as used for the determination but replacing the test solution (5.4) by 20 ml of water.

### 5.4 Preparation of test solution

Dissolve the test portion (5.2) in water, dilute to the mark in a 1 000 ml one-mark volumetric flask and mix.

### 5.5 Determination

Place 20,0 ml of the test solution (5.4) in a 500 ml conical flask. Add 50,0 ml of the iron(II) sulphate solution (3.4), then add slowly and carefully, with cooling, 20 ml of the sulphuric acid solution (3.2) and 5 ml of the orthophosphoric acid solution (3.1). Allow to stand for 10 min at ambient temperature.

Dilute to approximately 300 ml, add 5 drops of the barium diphenylamine-4-sulphonate solution (3.6) and titrate with the standard volumetric potassium dichromate solution (3.5) until the colour changes to violet.

## 6 EXPRESSION OF RESULTS

### 6.1 Method of calculation

The sodium chlorate ($NaClO_3$) content, expressed as a percentage by mass, is given by the formula

$$(V_1 - V_2) \times T \times \frac{1\,000}{20} \times \frac{100}{m} \times 0{,}017\,75 = \frac{88{,}75\,(V_1 - V_2)\,T}{m}$$

where

$V_1$ is the volume, in millilitres, of the standard volumetric potassium dichromate solution (3.5) used for the blank test;

$V_2$ is the volume, in millilitres, of the standard volumetric potassium dichromate solution (3.5) used for the determination;

$T$ is the actual concentration, expressed as normality, of the standard volumetric potassium dichromate solution (3.5);

$m$ is the mass, in grams, of the test portion (5.2);

0,017 75 is the mass, in grams, of sodium chlorate corresponding to 1 ml of exactly 1 N standard volumetric potassium dichromate solution.

### 6.2 Repeatability and reproducibility

Comparative analyses carried out in 9 laboratories by 18 operators gave the statistical information shown in the following table :

| | | |
|---|---|---|
| **Mean** (% *m/m*) | | 99,55 |
| **Standard deviation** | of repeatability ($\sigma_r$) | 0,15 |
| | of reproductibility ($\sigma_R$) | 0,31 |

## 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.