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# INTERNATIONAL STANDARD



# 3201

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## Sodium and potassium silicates for industrial use — Determination of iron content — 1,10-Phenanthroline photometric method

*Silicates de sodium et de potassium à usage industriel — Dosage du fer — Méthode photométrique à la 1,10-phénanthroline*

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## FOREWORD

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International Standard ISO 3201 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in October 1973.

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

Australia	Hungary	Spain
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No Member Body expressed disapproval of the document.

# Sodium and potassium silicates for industrial use — Determination of iron content — 1,10-Phenanthroline photometric method

## 1 SCOPE

This International Standard specifies a 1,10-phenanthroline photometric method for the determination of iron content of sodium and potassium silicates for industrial use.

## 2 FIELD OF APPLICATION

The method is applicable to products having iron contents greater than 2 mg/kg.

## 3 PRINCIPLE

Prior reduction of the trivalent iron by hydroxylammonium chloride. Formation of the divalent iron/1,10-phenanthroline complex in a buffered medium. Photometric measurement of the coloured complex at a wavelength of about 510 nm.

## 4 REAGENTS

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

**4.1 Hydrochloric acid**, approximately 2 N solution.

**4.2 Hydroxylammonium chloride** ( $\text{NH}_2\text{OH}\cdot\text{HCl}$ ), 100 g/l solution.

**4.3 Buffer solution**, pH 4,9.

Dissolve 272 g of sodium acetate trihydrate ( $\text{CH}_3\text{COONa}\cdot 3\text{H}_2\text{O}$ ) in approximately 500 ml of water. Add 240 ml of glacial acetic acid ( $\rho$  approximately 1,05 g/ml, 99 to 100 % (m/m) or about 17,4 N) to the solution, dilute to 1 000 ml and mix.

**4.4 Bromine water**, saturated at room temperature.

**4.5 1,10-Phenanthroline hydrochloride monohydrate** ( $\text{C}_{12}\text{H}_8\text{N}_2\cdot\text{HCl}\cdot\text{H}_2\text{O}$ ), 2,5 g/l solution.

This reagent may be replaced by 1,10-phenanthroline monohydrate ( $\text{C}_{12}\text{H}_8\text{N}_2\cdot\text{H}_2\text{O}$ ), 2,5 g/l solution.

**4.6 Iron**, standard solution corresponding to 0,200 g of Fe per litre.

Dissolve 1,404 3 g of ammonium iron(II) sulphate hexahydrate [ $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2\cdot 6\text{H}_2\text{O}$ ], weighed to the nearest 0,000 1 g, in 200 ml of water. Add 20 ml of sulphuric acid,  $\rho$  approximately 1,84 g/ml, cool to room temperature, dilute to the mark in a 1 000 ml one-mark volumetric flask and mix.

**4.7 Iron**, standard solution corresponding to 0,010 g of Fe per litre.

Transfer 25,0 ml of the standard iron solution (4.6) to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution at the time of use.

1 ml of this standard solution contains 0,010 mg of Fe.

**4.8 Methyl orange**, 0,5 g/l solution.

## 5 APPARATUS

Ordinary laboratory apparatus and :

**5.1 Spectrophotometer** or

**5.2 Photoelectric absorptiometer**, fitted with filters providing maximum transmission between 500 and 520 nm.

**5.3 Platinum crucible**, with lid. Upper diameter about 30 mm, height about 30 mm.

## 6 PROCEDURE

### 6.1 Test portion

Weigh, to the nearest 0,01 g, in a weighing bottle fitted with a ground glass closure, a quantity of the test sample corresponding to approximately 5 g of the anhydrous product.

### 6.2 Blank test

Pour 25 ml of water, a volume of the hydrochloric acid solution (4.1) 15 ml in excess of that used to neutralize the test portion (see 6.4.1), 5 drops of the methyl orange solution (4.8) and 5 ml of the bromine water (4.4) (to decolorize the indicator) into a 600 ml beaker. Boil for 5 min, cool to room temperature and transfer