
INTERNATIONAL STANDARD



3201

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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*

First edition — 1975-03-01

STANDARDSISO.COM : Click to view the full PDF of ISO 3201:1975

UDC 661.683 : 661.684 : 546.72 : 543.42

Ref. No. ISO 3201-1975 (E)

Descriptors : sodium silicates, potassium silicates, chemical analysis, determination of content, iron, photometric analysis.

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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.

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
Austria	India	Switzerland
Belgium	Israel	Thailand
Bulgaria	Italy	Turkey
Chile	Netherlands	United Kingdom
Czechoslovakia	New Zealand	U.S.S.R.
Egypt, Arab Rep. of	Portugal	Yugoslavia
France	Romania	
Germany	South Africa, Rep. of	

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 (ρ 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, ρ 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