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# INTERNATIONAL STANDARD



# 994

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## Potassium hydroxide for industrial use — Determination of iron content — 1,10-phenanthroline photometric method

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## 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 994 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in September 1971.

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

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

No Member Body expressed disapproval of the document.

This International Standard cancels and replaces ISO Recommendation R 994-1969, *Potassium hydroxide for industrial use — Determination of iron content — 2,2'-bipyridyl spectrophotometric method*.

# Potassium hydroxide 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 the iron content of potassium hydroxide for industrial use.

This method is more sensitive and more widely used than the 2,2'-bipyridyl method which was specified in ISO/R 994.

## 2 FIELD OF APPLICATION

The method is applicable to products having iron contents equal to or greater than 0,3 mg/kg.

## 3 PRINCIPLE

Reduction of the trivalent iron by hydroxylammonium chloride, followed by the formation of a bivalent iron/1,10-phenanthroline complex in a buffered system. Photometric measurement of the coloured complex at a wavelength of about 510 nm.

## 4 REAGENTS

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

**4.1 Hydrochloric acid**,  $\rho$  approximately 1,19 g/ml, about 38 % (m/m) solution or approximately 12 N.

**4.2 Ammonia solution**,  $\rho$  approximately 0,91 g/ml, about 25 % (m/m)  $\text{NH}_3$  solution or approximately 13 N, with a maximum iron content of 0,2 mg/kg.

**4.3 Hydroxylammonium chloride** ( $\text{NH}_2\text{OH}\cdot\text{HCl}$ ), 10 g/l solution.

**4.4 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 about 500 ml of water. Add 240 ml of glacial acetic acid ( $\rho$  approximately 1,05 g/ml, 99 to 100 % (m/m) solution or approximately 17,4 N) to the solution and dilute to 1 000 ml.

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

**4.6 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 product may be replaced by 1,10-phenanthroline monohydrate ( $\text{C}_{12}\text{H}_8\text{N}_2\cdot\text{H}_2\text{O}$ ).

**4.7 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 0,0001 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.

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

Transfer 25,0 ml of the iron standard solution (4.7) to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Prepare this solution immediately before use:

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

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

## 5 APPARATUS

Ordinary laboratory apparatus and

**5.1 Spectrophotometer**, or

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

## 6 PROCEDURE

### 6.1 Test portion

Weigh, to the nearest 0,1 g, in a weighing bottle fitted with a ground glass lid, a mass of test sample<sup>1)</sup> (solid or liquid) containing between 35 and 40 g of KOH.

1) See ISO 2466, Potassium hydroxide for industrial use – Sampling – Test sample – Preparation of the main solution for carrying out certain determinations.