UDC 661.312.1 : 543.24 : 546.41

Ref. No. : ISO/R 997-1969(E)

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 997

POTASSIUM HYDROXIDE FOR INDUSTRIAL USE

DETERMINATION OF CALCIUM

EDTA COMPLEXOMETRIC METHOD

1st EDITION
February 1969

For each individual country the only valid standard is the national standard of that country.

Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

BRIEF HISTORY

The ISO Recommendation R 997, *Potassium hydroxide for industrial use – Determination of calcium – EDTA complexometric method,* was drawn up by Technical Committee ISO/TC 47, *Chemistry,* the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Work on this question led, in 1966, to the adoption of a Draft ISO Recommendation.

In December 1966, this Draft ISO Recommendation (No. 1106) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Austria	Israel	Spain
Belgium	Italy	Switzerland
Brazil	Japan	Thailand
Chile	Netherlands	Turkey
Czechoslovakia	New Zealand	U.A.R.
Germany	Poland	United Kingdom
Hungary	Portugal	U.S.S.R.
India	Romania	Yugoslavia
Ireland	South Africa, Rep. of	

Two Member Bodies opposed the approval of the Draft :

France
U.S.A.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in February 1969, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 997 February 1969

POTASSIUM HYDROXIDE FOR INDUSTRIAL USE

DETERMINATION OF CALCIUM

EDTA COMPLEXOMETRIC METHOD

1. SCOPE

This ISO Recommendation describes a complexometric method for the determination of calcium in potassium hydroxide for industrial use.

2. PRINCIPLE

Titration with disodium ethylenediaminetetracetate in the presence of glyoxal bis (2-hydroxy-anil) as indicator, after elimination of impurities which may hinder the determination.

3. REAGENTS

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

3.1 *Hydrochloric acid*, approximately $d = 1.19$, 38 % (m/m) or 12 N solution.

3.2 *Hydrochloric acid*, approximately $d = 1.035$, 7 % (m/m) or 2 N solution.
Add some water to 165 ml of the approximately $d = 1.19$ hydrochloric acid solution and dilute to 1000 ml.

3.3 *Sulphuric acid*, approximately $d = 1.06$, 9 % (m/m) or 2 N solution.
Add 55 ml of approximately $d = 1.83$ sulphuric acid solution to some water, allow to cool and dilute to 1000 ml.

3.4 *Sodium hydroxide*, approximately $d = 1.08$, 7.5 % (m/m) or 2 N solution.
Dissolve 80 g of sodium hydroxide in water and dilute to 1000 ml.

3.5 *Ammonium hydroxide*, approximately $d = 0.983$, 3.5 % (m/m) or 2 N solution.
Add some water to 250 ml of approximately $d = 0.94$ ammonium hydroxide (14.5 % (m/m) or 8 N solution approximately) and dilute to 1000 ml.

3.6 *Iron (III) chloride*, hydrochloric solution containing approximately 2 g/l of Fe.
Weigh, to the nearest 0.1 g, 10 g of crystals of iron (III) chloride hexahydrate. Place in a 1000 ml one-mark volumetric flask and dissolve in approximately 600 ml of water.
Add 10 ml of the hydrochloric acid solution (3.1). Dilute to the mark and mix thoroughly.

3.7 *Sodium sulphide*, saturated solution.
Prepare a solution using crystals of sodium sulphide hydrate ($Na_2S.9H_2O$) previously washed with water.

3.8 *Ethanol*, 95 % (v/v).
Alternatively, alcohol denatured with acetone, but not coloured, may be used.

3.9 *Calcium chloride*, 0.02 M standard solution.
Weigh, to the nearest 0.001 g, 2.002 g of precipitated calcium carbonate ($CaCO_3$) and place in a 600 ml beaker. Mix 75 ml of water with 25 ml of the hydrochloric acid solution (3.2) and transfer this solution to the beaker. Boil for about 5 minutes to eliminate carbon dioxide. Cool to about 20 °C, transfer quantitatively to a 1000 ml one-mark volumetric flask, dilute to the mark and mix thoroughly.

3.10 *Disodium ethylenediaminetetracetate (EDTA)*, 0.002 M standard volumetric solution.
Dissolve approximately 7.7 g of EDTA in water.

Transfer quantitatively to a 1000 ml one-mark volumetric flask, dilute to the mark and mix thoroughly.

Adjust the concentration of this solution to 0.02 M exactly, calculated on the calcium chloride standard solution (3.9), operating as follows :

Transfer 25.0 ml of the calcium chloride solution (3.9) to a 250 ml conical flask.

Successively add approximately 25 ml of water, then, by means of graduated pipettes, 4 ml of the sodium hydroxide solution (3.4), 15 ml of ethanol (3.8) and 1.0 ml of the glyoxal bis (2-hydroxy-anil) solution (3.12). Wait for about 1 minute, then titrate with the solution whose strength is to be determined, until the colour turns from red to pure yellow. In order to facilitate the correct evaluation of the end-point, a standard matching solution may be used; this solution can be prepared by successively transferring to a 250 ml conical flask : approximately 75 ml of water, 4 ml of the sodium hydroxide solution (3.4), 15 ml of ethanol (3.8) and 1.0 ml of the glyoxal bis (2-hydroxy-anil) solution (3.12). 25.0 ml is the correct theoretical quantity. The EDTA solution should, therefore, be adjusted to the correct strength and rechecked. Repeat the operation if necessary.

Then transfer 100.0 ml of the concentrated EDTA solution thus adjusted to a 1000 ml one-mark volumetric flask.

Dilute to the mark and mix thoroughly. The solution obtained is 0.002 M.

3.11 *Methyl orange*, 0.5 g/l solution.
Dissolve 0.05 g of methyl orange in water and dilute to 100 ml.

3.12 *Glyoxal bis (2-hydroxy-anil)*, approximately 2.5 g/l ethanolic solution.

3.12.1 Place approximately 0.25 g of glyoxal bis (2-hydroxy-anil) in a 100 ml dark glass container with ground glass stopper. Add 100 ml of ethanol (3.8) and stir until completely dissolved.

3.12.2 Should this indicator be difficult to obtain, it can be prepared as follows :

Place 8.8 g of chemically pure *o*-aminophenol in a 3000 ml conical flask containing 2000 ml of water at 80 °C. After dissolution, add 8 g of a 300 g/l water solution of glyoxal.

Keep the mixture at 80 °C for 30 minutes, then cool to room temperature under running water. Filter the precipitate obtained through a Büchner funnel fitted with a high-speed filter paper and wash three times, carefully drying after each wash. Then remove the precipitate from the filter and place in a 250 ml beaker. Add 100 ml of acetone. After dissolution, add approximately 3 g of active carbon, stir for 1 minute, then filter on a dry pleated filter paper and collect the filtrate in a 750 ml conical flask.

Add 300 ml of water to the filtrate and again filter the precipitate through a high-speed filter on a Büchner funnel. Drain thoroughly, dry for 2 hours in the oven at 70 °C without exceeding this temperature, crush and store in a dark glass airtight container.