UDC 661.312.1 : 543.24 : 546.131

Ref. No. : ISO/R 992-1969 (E)

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 992

POTASSIUM HYDROXIDE FOR INDUSTRIAL USE

DETERMINATION OF CHLORIDE CONTENT

VOLHARD VOLUMETRIC METHOD

1st EDITION

February 1969

Printed in Switzerland

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

BRIEF HISTORY

The ISO Recommendation R 992, *Potassium hydroxide for industrial use – Determination of chloride content – Volhard volumetric 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. 1101) 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	Ireland	South Africa, Rep. of
Belgium	Israel	Spain
Brazil	Japan	Switzerland
Chile	Korea, Dem. P. Rep. of	Thailand
Cuba	Netherlands	Turkey
Czechoslovakia	New Zealand	U.A.R.
Germany	Poland	United Kingdom
Hungary	Portugal	U.S.S.R.
India	Romania	Yugoslavia

Three Member Bodies opposed the approval of the Draft :

France
Italy
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 992 February 1969

POTASSIUM HYDROXIDE FOR INDUSTRIAL USE

DETERMINATION OF CHLORIDE CONTENT

VOLHARD VOLUMETRIC METHOD

1. SCOPE

This ISO Recommendation describes the Volhard volumetric method for the determination of chloride content in potassium hydroxide for industrial use.

2. FIELD OF APPLICATION

The method is applicable to the determination of chloride content of potassium hydroxide for industrial use, for contents greater than 0.05 % (m/m), expressed as potassium chloride and calculated on KOH.

Special case

Determination of chloride contents between 0.01 and 0.05 % (m/m), expressed as potassium chloride and calculated on KOH.

3. PRINCIPLE

Precipitation of Cl^- ions by addition of excess silver nitrate and titration of the excess with ammonium thiocyanate in the presence of ammonium-iron (III) sulphate.

4. REAGENTS

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

4.1 *Nitric acid,* approximately $d = 1.4$, 68 % (m/m) or 14 N solution.

4.2 *Nitric acid,* approximately 0.2 N solution.

4.3 *Silver nitrate,* 0.1 N standard volumetric solution (see Note in section 7).

4.4 *Ammonium thiocyanate,* 0.1 N standard volumetric solution (see Note in section 7).

4.5 *Ammonium-iron (III) sulphate,* nitric solution.
Dissolve 25 g of ammonium-iron (III) sulphate hydrate $[(NH_4)_2\ SO_4 \cdot Fe_2(SO_4)_3 \cdot 24H_2O]$ in 100 ml of water. Add 50 ml of nitric acid solution, approximately $d = 1.4$, and mix thoroughly.

4.6 *Litmus paper.*

5. APPARATUS

Ordinary laboratory apparatus.

6. PROCEDURE

6.1 Test portion

Transfer 100.0 ml of sample solution A* to a 500 ml conical flask.

6.2 Titration

Neutralize the sample solution (6.1) with the nitric acid solution (4.1) in the presence of litmus paper (4.6). Then add 0.5 to 1 ml excess nitric acid solution (4.1) and cool under running water to room temperature.

Add approximately 3 ml of the ammonium-iron (III) sulphate solution (4.5).

Fill a burette with the silver nitrate standard volumetric solution (4.3) and another with the ammonium thiocyanate standard volumetric solution (4.4). Run 0.20 ml of the latter solution (4.4) into the sample solution so as to form ferric thiocyanate, a red compound the appearance of which indicates the end-point of the titration.

Then add the silver nitrate standard volumetric solution (4.3) until the reddish-pink colour disappears and add an excess of approximately 2 ml measured to the nearest 0.05 ml. Stir for 4 to 5 minutes and filter under reduced pressure on a glass filter crucible fitted with a sintered disk of porosity between 5 and 15 μm. Wash both the conical flask and the filter crucible three times with a small amount (approximately 10 ml) of the nitric acid solution (4.2) and then titrate the excess silver nitrate in the filtrate by adding the ammonium thiocyanate standard volumetric solution (4.4) until the pink colour reappears.

Deduct from the titration the drop that causes the end point.

7. EXPRESSION OF RESULTS

Chloride content, expressed as potassium chloride (KCl), is given as a percentage, by mass, by the following formula :

$$(V - V_1) \times A \times \frac{500}{100} \times \frac{100}{E} = 3.7278 \times \frac{(V - V_1)}{E}$$

where

V is the volume, in millilitres, of the silver nitrate standard volumetric solution (4.3) used;

V_1 is the volume, in millilitres, of the ammonium thiocyanate standard volumetric solution (4.4) used (0.20 ml + volume used for back-titration);

A is the mass, in grammes, of potassium chloride corresponding to 1 ml of 0.1 N silver nitrate solution (theoretical value 1 ml $\triangleq$ 0.007 456 g of KCl);

E is the mass, in grammes, of the test portion used for the preparation of the sample solution A*.

NOTE. – If the silver nitrate (4.3) and ammonium thiocyanate (4.4) standard volumetric solutions are not exactly the strength indicated in the list of reagents, suitable correction factors should be employed in calculating the result.

* See ISO Recommendation R 989, *Potassium hydroxide for industrial use – Preparation of sample solution*, clause 5.2.