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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 742

SODIUM CARBONATE FOR INDUSTRIAL USE

DETERMINATION OF CHLORIDE CONTENT

VOLHARD VOLUMETRIC METHOD

1st EDITION

May 1968

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

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BRIEF HISTORY

The ISO Recommendation R 742, *Sodium carbonate 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 by the Technical Committee began in 1951 and led, in 1956, to the adoption of a Draft ISO Recommendation.

In June 1966, this Draft ISO Recommendation (No. 1008) 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 :

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

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in May 1968, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 742 May 1968

SODIUM CARBONATE 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.

2. FIELD OF APPLICATION

The method is applicable to the determination of chlorides in sodium carbonate for industrial use for contents higher than 0.05 % (m/m) expressed as sodium chloride.

2.1 Special case

Determination of chloride contents equal to or lower than 0.05 % (m/m) expressed as sodium chloride (see section 8).

3. PRINCIPLE

Precipitation of Cl^- ions by addition of excess of 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*, $d = 1.4$ approximately, 68 % (m/m) or approximately 14 N solution.

4.2 *Nitric acid*, approximately 0.2 N solution.

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

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

4.5 *Ammonium-iron (III) sulphate*, nitric solution. Dissolve 25 g of ammonium-iron (III) sulphate hydrate [$(NH_4)_2SO_4.Fe_2(SO_4)_3.24H_2O$] in 100 ml of water. Add 50 ml of nitric acid, $d = 1.4$ approximately, and mix thoroughly.

4.6 *Litmus paper*.

5. APPARATUS

Ordinary laboratory apparatus.

6. PROCEDURE

6.1 Test portion

Weigh to the nearest 0.01 g, 10 ± 0.1 g of the test sample.*

* See ISO Recommendation R 739, *Sodium carbonate for industrial use – Preparation and storage of test samples.*

6.2 Preparation of test solution

Dissolve the test portion by pouring it in small quantities at a time into a 500 ml conical flask containing approximately 100 ml of water. Stir during the operation. Neutralize with the nitric acid solution (4.1) in the presence of the litmus paper (4.6). Then add 0.5 to 1 ml excess of the nitric acid solution (4.1) and cool under running water to room temperature.

6.3 Titration

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

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

Then add the standard volumetric solution of silver nitrate (4.3) until the reddish-pink colour disappears and add an excess of 2 ml measured to the nearest 0.05 ml.

Stir for 4 to 5 minutes, then filter on a filter-crucible with a sintered disk of porosity grade between 5 and 15 μ, under reduced pressure. Wash both the conical flask and the filter crucible three times with a small amount of the nitric acid solution (4.2) and finally titrate the excess silver nitrate in the filtrate by adding the standard volumetric solution of ammonium thiocyanate (4.4) until a pinkish colour reappears.

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

7. EXPRESSION OF RESULTS

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

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

where

V is the volume, in millilitres, of the 0.1 N standard volumetric solution of silver nitrate used,

V_1 is the volume in millilitres, of the 0.1 N standard volumetric solution of ammonium thiocyanate used (0.20 ml + volume used for back-titration),

A is the mass, in grammes, of sodium chloride corresponding to 1 ml of 0.1 N standard volumetric solution of silver nitrate (theoretical value : 1 ml = 0.005845 g of NaCl),

E is the mass, in grammes, of the test portion.

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