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# ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION



# ISO RECOMMENDATION

# R 743

## SODIUM CARBONATE FOR INDUSTRIAL USE

## DETERMINATION OF SULPHATE CONTENT

## BARIUM SULPHATE GRAVIMETRIC METHOD

1st EDITION
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## BRIEF HISTORY

The ISO Recommendation R 743, *Sodium carbonate for industrial use – Determination of sulphate content – Barium sulphate gravimetric 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. 1009) 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 743 May 1968.

# SODIUM CARBONATE FOR INDUSTRIAL USE

# DETERMINATION OF SULPHATE CONTENT

# BARIUM SULPHATE GRAVIMETRIC METHOD



## 1. SCOPE

This ISO Recommendation describes a gravimetric method for the determination of the sulphate content of sodium carbonate for industrial use.

## 2. PRINCIPLE

Precipitation of $SO_4^{2-}$ ions as barium sulphate in diluted hydrochloric acid medium.

Separation of the precipitate obtained, ignition at a temperature of 800 °C and weighing.

## 3. REAGENTS

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

3.1 *Hydrochloric acid*, $d$ = 1.18 approximately, 37 % (m/m) or approximately 12 N solution.

3.2 *Sulphuric acid*, $d$ = 1.84 approximately, 93.6 % (m/m) or approximately 35 N solution.

3.3 *Ammonia*, $d$ = 0.91 approximately, 25 % (m/m) or approximately 15 N solution.

3.4 *Barium chloride* dihydrate ($BaCl_2.2H_2O$), 100 g/l solution.

3.5 *Silver nitrate*, 5 g/l nitric solution. Dissolve 0.5 g of silver nitrate in a small amount of water, add 10 ml of nitric acid approximately $d$ = 1.4 and dilute to 100 ml.

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

## 4. APPARATUS

Ordinary laboratory apparatus and

4.1 *Platinum crucible*, approximately 30 mm top diameter and 30 mm deep.

## 5. PROCEDURE

### 5.1 Test portion

Weigh to the nearest 0.01 g, approximately 40 ± 0.1 g of the test sample*.

### 5.2 Preparation of the test solution

Dissolve the test portion by adding small quantities at a time to a beaker of suitable capacity (600 ml for example) containing 100 ml of water. Stir during the operation. Then acidify the solution by slowly adding 70 ml of the hydrochloric acid solution (3.1).

Transfer quantitatively to a 200 ml one-mark volumetric flask, dilute to the mark and mix thoroughly.

Filter on a dry ashless slow-speed filter paper, approximately 90 mm in diameter, and discard the first 10 ml of the filtrate.

### 5.3 Determination

Transfer 100.0 ml of filtrate to a beaker of suitable capacity (600 ml for example).

Add approximately 150 ml of water. Neutralize by means of the ammonia solution (3.3) in the presence of methyl orange (3.6). Then acidify by adding approximately 3 ml of the hydrochloric acid solution (3.1).

Bring to the boil, stop heating and add 25 ml of the barium chloride solution (3.4) drop by drop (the addition should take about 1 minute and 30 seconds). Bring to the boil and allow to boil for 2 minutes. Place on a boiling water bath and leave for 2 hours, then stop heating and leave to stand for about 16 hours.

Filter on an ashless slow-speed filter paper approximately 90 mm diameter (pore diameter between 0.4 and 1 $\mu$ approximately). Wash the precipitate onto the filter with boiling water until 10 ml of liquid flowing from the funnel remain clear for 5 minutes after addition of 10 ml of the nitric solution of silver nitrate (3.5).

Place the filter paper and its content in the platinum crucible (4.1), previously tared after igniting in a furnace at a temperature of 800 °C and cooling in a desiccator.

Dry in an oven at a temperature of about 110°C and ignite in a furnace, gently at first to char the filter paper, taking care to avoid igniting it, and then at a temperature of 800 °C for 15 minutes. Allow to cool in a desiccator to room temperature and weigh.

If the ignited precipitate shows a greyish colour due to the presence of graphitic carbon, add a drop of the sulphuric acid solution (3.2), place in the furnace and ignite again at a temperature of 800 °C for 15 minutes.

* See clause 2.2 of the ISO Recommendation R 739, *Sodium carbonate for industrial use – Preparation and storage of test samples.*