

# SVENSK STANDARD

## SS-ISO 4689:2018

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**Järnmalm – Bestämning av halt svavel – Bariumsulfat  
gravimetrisk metod (ISO 4689:1986, IDT)**

**Iron ores – Determination of sulfur content – Barium sulfate  
gravimetric method (ISO 4689:1986, IDT)**



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Den internationella standarden ISO 4689:1986 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av ISO 4689:1986.

The International Standard ISO 4689:1986 has the status of a Swedish Standard. This document contains the official English version of ISO 4689:1986.

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Denna standard är framtagen av kommittén för Järnmalm, SIS/TK 149.

Har du synpunkter på innehållet i den här standarden, vill du delta i ett kommande revideringsarbete eller vara med och ta fram andra standarder inom området? Gå in på [www.sis.se](http://www.sis.se) - där hittar du mer information.

# Contents

Page

<b>Foreword</b> .....	<b>v</b>
<b>1 Scope and field of application</b> .....	<b>1</b>
<b>2 References</b> .....	<b>1</b>
<b>3 Principle</b> .....	<b>1</b>
<b>4 Reagents</b> .....	<b>1</b>
4.3 Zinc .....	1
<b>5 Apparatus</b> .....	<b>2</b>
<b>6 Sampling and samples</b> .....	<b>2</b>
6.1 Laboratory sample .....	2
6.2 Preparation of predried test samples .....	3
<b>7 Procedure</b> .....	<b>3</b>
7.1 Number of determinations .....	3
7.2 Blank test and check test .....	3
7.3 Test portion .....	3
7.4 Determination .....	4
7.4.1 Decomposition of the test portion .....	4
7.4.2 Extraction of the bulk of the iron .....	4
7.4.3 Treatment of the residue .....	4
7.4.4 Precipitation of barium sulfate .....	5
7.4.5 Weighing .....	5
<b>8 Expression of results</b> .....	<b>5</b>
8.1 Calculation of sulfur content .....	5
8.2 General treatment of results .....	6
8.2.1 Repeatability and permissible tolerance .....	6
8.2.2 Acceptance of analytical values .....	6
8.2.3 Calculation of final result .....	7
<b>9 Test report</b> .....	<b>8</b>
<b>Annex A Flowsheet of the procedure for the acceptance of analytical values for test samples</b> (An integral part of this International Standard.) .....	<b>9</b>
<b>Annex B Derivation of repeatability and permissible tolerance equations</b> (This annex is for information only, and is not an integral part of this International Standard.) .....	<b>10</b>
<b>Annex C Precision data obtained by international analytical trial</b> (This annex is for information only, and is not an integral part of this International Standard.) .....	<b>11</b>

## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4689 was prepared by Technical Committee ISO/TC 102, *Iron ores*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.



# Iron ores — Determination of sulfur content — Barium sulfate gravimetric method

## 1 Scope and field of application

This International Standard specifies a barium sulfate gravimetric method for the determination of the sulfur content of iron ores.

This method is applicable to a concentration range of 0,01 to 1,0 % (*m/m*) of sulfur in natural iron ores, and iron ore concentrates and agglomerates including sinter products.

## 2 References

ISO 3081, *Iron ores — Increment sampling — Manual method.*

ISO 3082, *Iron ores — Increment sampling and sample preparation — Mechanical method.*

ISO 3083, *Iron ores — Preparation of samples — Manual method.*

ISO 7764, *Iron ores — Preparation of predried test samples for chemical analysis.*

## 3 Principle

Decomposition of a test portion by treatment with potassium chlorate and hydrochloric and nitric acids followed by evaporation to dryness. Dissolution of the salts in hydrochloric acid and filtration of the insoluble residue. Removal of the major portion of the iron in the filtrate by extraction with methyl isobutyl ketone.

Ignition of the insoluble residue and removal of silicon dioxide by evaporation with hydrofluoric and nitric acids. Fusion of the residue with sodium carbonate followed by leaching and filtration. Acidification of the filtrate and combination with the main solution.

Reduction of any remaining iron to the bivalent state, adjustment of the acidity and addition of barium chloride solution. Filtration of barium sulfate and gravimetric determination.

## 4 Reagents

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

**4.1 Potassium chlorate** ( $\text{KClO}_3$ ), powder.

**4.2 Sodium carbonate** ( $\text{Na}_2\text{CO}_3$ ), anhydrous.

**4.3 Zinc.**

Use zinc with the lowest available sulfur content and with a particle size of 1 to 3 mm.

**4.4 Hydrochloric acid**,  $\rho$  1,16 to 1,19 g/ml.

**4.5 Hydrochloric acid**,  $\rho$  1,16 to 1,19 g/ml, diluted 2 + 1.

**4.6 Hydrochloric acid**,  $\rho$  1,16 to 1,19 g/ml, diluted 1 + 1.

SS-ISO 4689:2018 (E)

**4.7 Hydrochloric acid**,  $\rho$  1,16 to 1,19 g/ml, diluted 2 + 100.

**4.8 Nitric acid**,  $\rho$  1,4 g/ml.

**4.9 Hydrofluoric acid**, 40% (m/m),  $\rho$  1,13 g/ml, or 48 % (m/m),  $\rho$  1,185 g/ml.

**4.10 Acid mixture**: hydrochloric acid (4.4) + nitric acid (4.8), 4 + 1.

NOTE — Do not store this mixture; prepare immediately before use.

**4.11 Barium chloride** ( $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ ), 100 g/l solution.

Dissolve 100 g of crystalline barium chloride dihydrate ( $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ ) in 1 litre of water, cover and heat to boiling point. Keep warm on a water bath for a minimum of 2 h and allow to cool to room temperature overnight. Store the solution in a plastics bottle and before each use, filter the required volume through a close-texture filter paper.

**4.12 Hydrochloric acid wash solution**, containing barium chloride.

Filter 10 ml of barium chloride solution (4.11) through a close-texture filter paper, and dilute to 1 000 ml with hydrochloric acid solution (4.7).

**4.13 Sodium nitrate** ( $\text{NaNO}_3$ ), saturated solution.

**4.14 Sodium carbonate** ( $\text{Na}_2\text{CO}_3$ ), 20 g/l solution.

Store in a plastics bottle.

**4.15 Ammonium thiocyanate** ( $\text{NH}_4\text{SCN}$ ), 100 g/l solution.

**4.16 Silver nitrate**, 20 g/l solution.

**4.17 Methyl orange**, 0,1 g/100 ml solution.

Dissolve 0,10 g of methyl orange in 100 ml of water.

**4.18 Methyl isobutyl ketone** [ $\text{CH}_3\text{COCH}_2\text{CH}(\text{CH}_3)_2$ ] (4- Methylpentan-2-one).

Pre-treat as follows before use : transfer 200 ml of methyl isobutyl ketone and 100 ml of hydrochloric acid solution (4.6) to a 500 ml separating funnel, and shake thoroughly for about 1 min. Allow the layers to separate and discard the lower aqueous layer.

## 5 Apparatus

Ordinary laboratory apparatus.

## 6 Sampling and samples

### 6.1 Laboratory sample

For analysis, use a laboratory sample of minus 100  $\mu\text{m}$  particle size which has been taken in accordance with ISO 3081 or ISO 3082 and prepared in accordance with ISO 3082 or ISO 3083. In the case of