



**SLOVENSKI STANDARD**  
**SIST ISO 13311:2000**  
**01-junij-2000**

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Iron ores -- Determination of lead content -- Flame atomic absorption spectrometric method

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Minerais de fer -- Dosage du plomb -- Méthode par spectrométrie d'absorption atomique dans la flamme

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Ta slovenski standard je istoveten z: **ISO 13311:1997**

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**ICS:**

73.060.10      Železove rude      Iron ores

**SIST ISO 13311:2000**      en

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INTERNATIONAL  
STANDARD

ISO  
13311

First edition  
1997-08-01

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Flame atomic absorption spectrometric  
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*Minerais de fer — Dosage du plomb — Méthode par spectrométrie  
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Reference number  
ISO 13311:1997(E)

**ISO 13311:1997(E)****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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 13311 was prepared by Technical Committee ISO/TC 102, *Iron ores*, Subcommittee SC 2, *Chemical analysis*.

Together with ISO 13310, it cancels and replaces ISO 8753:1987, of which it constitutes a technical revision.

Annex A forms an integral part of this International Standard. Annexes B and C are for information only.

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Printed in Switzerland

# Iron ores — Determination of lead content — Flame atomic absorption spectrometric method

**WARNING** — This International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety issues associated with its use. It is the responsibility of the user to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

## 1 Scope

This International Standard specifies a flame atomic absorption spectrometric method for the determination of the lead content of iron ores.

This method is applicable to lead contents between 0,001 % (*m/m*) and 0,5 % (*m/m*) in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

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## 2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 648:1977, *Laboratory glassware — One-mark pipettes.*

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks.*

ISO 3081:1986, *Iron ores — Increment sampling — Manual method.*

ISO 3082:1987, *Iron ores — Increment sampling and sample preparation — Mechanical method.*

ISO 3083:1986, *Iron ores — Preparation of samples — Manual method.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 7764:1985, *Iron ores — Preparation of predried test samples for chemical analysis.*

## 3 Principle

Decomposition of the test portion and removal of silica by treatment with hydrochloric acid and hydrofluoric acid. Oxidation with nitric acid.

Evaporation to dryness, followed by dilution and filtration. Ignition of the residue. Fusion with sodium carbonate. Dissolution of the cooled melt with hydrochloric acid. Reservation of the solution.

Extraction of iron in the filtrate with 4-methyl-2-pentanone. Recovery of lead extracted. Decomposition of 4-methyl-2-pentanone with nitric acid. Evaporation to dryness and dissolution of salts with the reserved solution and hydrochloric acid.

Aspiration of the solution into the flame of an atomic absorption spectrometer using an air-acetylene burner.

Comparison of absorbance values obtained for lead with those obtained from the calibration solutions.

## 4 Reagents

During the analysis, use only reagents of recognized analytical grade and only water that complies with grade 2 of ISO 3696.

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

**4.2 4-methyl-2-pentanone** (MIBK), high purity.

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

**4.4 Hydrochloric acid**,  $\rho$  1,16 g/ml to 1,19 g/ml, diluted 10 + 6.

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

**4.6 Hydrochloric acid**,  $\rho$  1,16 g/ml to 1,19 g/ml, diluted 2 + 98.

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

**4.8 Hydrofluoric acid**,  $\rho$  1,13 g/ml, 40 % (m/m), or  $\rho$  1,19 g/ml, 48 % (m/m).

### 4.9 Lead standard solutions

#### 4.9.1 Lead stock standard solution

Dissolve 1,000 g of lead metal [of purity > 99,5 % (m/m)] in 40 ml of nitric acid ( $\rho$  1,4 g/ml, diluted 1 + 1). Cool and dilute to 1 000 ml in a one-mark volumetric flask and mix.

1 ml of the stock solution contains 1 000  $\mu\text{g}$  of lead.

#### 4.9.2 Standard solution A

Transfer 10,0 ml of lead stock standard solution (4.9.1) to a 100 ml volumetric flask. Dilute to volume and mix.

1 ml of this standard solution contains 100  $\mu\text{g}$  of lead.

#### 4.9.3 Standard solution B

Transfer 10,0 ml of standard solution A (4.9.2) to a 100 ml volumetric flask. Dilute to volume and mix.

1 ml of this standard solution contains 100  $\mu\text{g}$  of lead.

## 5 Apparatus

Ordinary laboratory apparatus, including one-mark pipettes and one-mark volumetric flasks complying with the specifications of ISO 648 and ISO 1042 respectively, and

**5.1 Polytetrafluoroethylene (PTFE) beaker**, of capacity 250 ml, with a PTFE cover.

**5.2 Atomic absorption spectrometer**, equipped with an air-acetylene burner.

The atomic absorption spectrometer shall meet the following criteria.

**WARNING — Follow the manufacturer's instructions for igniting and extinguishing the air-acetylene flame to avoid possible explosion hazards. Wear tinted safety glasses whenever the burner is in operation.**

- a) *Minimum sensitivity* — the absorbance of the most concentrated calibration solution (see 7.4.4) shall be at least 0,25.
- b) *Graph linearity* — the slope of the calibration graph covering the top 20 % of the concentration range (expressed as a change in absorbance) shall not be less than 0,7 of the value of the slope for the bottom 20 % of the concentration range determined in the same way.
- c) *Minimum stability* — the standard deviation of the absorbance of the most concentrated calibration solution and that of the zero calibration solution, each being calculated from a sufficient number of repetitive measurements, shall be less than 1,5 % and 0,5 % respectively of the mean value of the absorbance of the most concentrated calibration solution.

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### NOTES

1 The use of a strip-chart recorder and/or digital readout device is recommended to evaluate criteria a), b) and c) and for all subsequent measurements.

2 Instrument parameters will vary with each instrument. The following parameters were successfully used in several laboratories and they can be used as guidelines.

— Hollow cathode lamp, mA	6
— Wavelength, nm	283,3
— Air flow rate, l/min	14
— Acetylene flow rate, l/min	3

In systems where the values shown for gas flow rates do not apply, the ratio of the gas flow rates may still be a useful guideline.

## 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 ores having significant contents of combined water or oxidizable compounds, use a particle size of minus 160  $\mu\text{m}$ .

NOTE — A guideline on significant contents of combined water and oxidizable compounds is incorporated in ISO 7764.

### 6.2 Preparation of predried test samples

Thoroughly mix the laboratory sample and, taking multiple increments, extract a test sample in such a manner that it is representative of the whole contents of the container. Dry the test samples at  $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$  as specified in ISO 7764. (This is the predried test sample.)

## 7 Procedure

### 7.1 Number of determinations

Carry out the analysis at least in duplicate in accordance with annex A, independently, on one predried test sample.

NOTE — The expression “independently” means that the second and any subsequent result is not affected by the previous result(s). For this particular analytical method, this condition implies that the repetition of the procedure is carried out either by the same operator at a different time or by a different operator including, in either case, appropriate recalibration.

### 7.2 Test portion

Taking several increments, weigh, to the nearest 0,000 2 g, approximately 2 g of the predried test sample obtained in accordance with 6.2.

NOTE — The test portion should be taken and weighed quickly in order to avoid reabsorption of moisture.

### 7.3 Blank test and check test

In each run, one blank test and one analysis of a certified reference material of the same type of ore shall be carried out in parallel with the analysis of the ore sample(s) under the same conditions. A predried test sample of the certified reference material shall be prepared as specified in 6.2.

NOTE — The certified reference material should be of the same type as the sample to be analysed and the properties of the two materials should be sufficiently similar to ensure that, in either case, no significant changes in the analytical procedure become necessary.

When the analysis is carried out on several samples at the same time, the blank value may be represented by one test, provided that the procedure is the same and the reagents are from the same reagent bottles.

When the analysis is carried out on several samples of the same type of ore at the same time, the analytical value of one certified reference material may be used.

### 7.4 Determination

#### 7.4.1 Decomposition of the test portion

Transfer the test portion (7.2) to a 250 ml PTFE beaker (5.1). Moisten with a few millilitres of water, add 40 ml of hydrochloric acid (4.3) and 10 ml of hydrofluoric acid (4.8), and cover with a PTFE cover. Heat on a hotplate at 100 °C, then increase the heat to 200 °C. Evaporate to dryness. Add 5 ml of nitric acid (4.7) and evaporate to nearly 1 ml. Dissolve the salts with 10 ml of hydrochloric acid (4.3) and evaporate to dryness again.

Dissolve the salts with 5 ml of hydrochloric acid (4.3). Add 10 ml of water and filter through a close-texture paper into a 250 ml beaker. Remove all adhering particles from the beaker with a rubber-tipped rod, wash with hydrochloric acid (4.6) until the paper is free from iron stains, and wash the paper three times with hot water. Reserve the residue and combined filtrate and washings.

#### 7.4.2 Treatment of residue

Place the paper and residue in a platinum crucible, dry and char the paper at a low temperature and ignite in a muffle furnace at 550 °C. Add 0,5 g of sodium carbonate (4.1) and fuse over a Bunsen burner (about 900 °C to 1 000 °C) until a clear melt is obtained. Dissolve the cooled melt with 5 ml of hydrochloric acid (4.5), heat to remove carbon dioxide and reserve the solution.

#### 7.4.3 Treatment of combined filtrate and washings

Evaporate the filtrate and washings (7.4.1) just to dryness. Dissolve the salts with 20 ml of hydrochloric acid (4.4), and transfer to a 200 ml separating funnel. Rinse the beaker with 20 ml of hydrochloric acid (4.4) and combine



these washings with the main solution. Add 50 ml of 4-methyl-2-pentanone (4.2) and shake thoroughly for 1 min. Allow the layers to separate, then run the lower aqueous solution into a 250 ml beaker. Wash the organic phase by extracting with 10 ml of hydrochloric acid (4.4), and transfer the washings to the beaker.

Heat the solution gently and expel almost all of the 4-methyl-2-pentanone in the solution. Then add 5 ml of nitric acid (4.7) and evaporate to dryness. Dissolve the salts with 15 ml of hydrochloric acid (4.5). Combine this solution with the reserved solution from 7.4.2.

Transfer the solution to a 50 ml one-mark volumetric flask, and dilute to volume with water and mix. Depending on the concentration (see table 1), use the solution for atomic absorption measurement either without dilution or as specified. If dilution is required, transfer the appropriate aliquot to a 250 ml beaker. Add the amount of sodium carbonate (4.1) and hydrochloric acid (4.5) indicated in table 1. Heat the solution to remove carbon dioxide. Cool and dilute to 100 ml in a one-mark volumetric flask with water. (See the note to table 1.) (This solution is the final test solution.)

Table 1 — Dilution guide for test solution<sup>1)</sup>

Expected content, $w$ , of lead in sample % (m/m)	Aliquot ml	Mass of sodium carbonate (4.1) to be added g	Volume of hydrochloric acid (4.5) to be added ml
$0,001 < w_{Pb} \leq 0,05$	—	—	—
$0,05 < w_{Pb} \leq 0,2$	20	0,8	32
$0,2 < w_{Pb} \leq 0,25$	10	0,9	36
$0,25 < w_{Pb} \leq 0,5$	5	0,95	38

1) Dilution shown will provide concentrations of lead falling within the range of the calibration solutions (7.4.4). For instruments having high sensitivity, smaller portions of the test solution may be preferable. For 1 ml or 2 ml of aliquot, make a preliminary dilution to avoid dilution error. Treat the blank test solution similarly.

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Transfer corresponding amounts of blank test solution to a 250 ml beaker, and add the same volume of sodium carbonate (4.1) and hydrochloric acid (4.5) as used for the test solution. Heat the solution to remove carbon dioxide. Cool and dilute to 100 ml in a one-mark volumetric flask with water and mix. (This solution is the diluted blank test solution.)

#### 7.4.4 Preparation of the set of lead calibration solutions

Transfer 1,0 g of sodium carbonate (4.1) to each of six 250 ml beakers. Add 20 ml of hydrochloric acid (4.3) and appropriate aliquots of lead standard solutions A or B (4.9.2 or 4.9.3) as listed in table 2. Heat the solution to remove carbon dioxide. Cool and dilute to 100 ml in a one-mark volumetric flask.

NOTE — The range of lead that can be covered may vary from instrument to instrument. Attention should be paid to the minimum criteria given in 5.2. For instruments having high sensitivity, smaller aliquots of the standard solutions can be used.

Table 2 — Calibration solutions

Solution number	Standard solution A (4.9.2)	Standard solution B (4.9.3)	Lead concentration $\mu\text{g/ml}$
	ml	ml	
0	0	0	0
1	0	20	2
2	5	0	5
3	10	0	10
4	15	0	15
5	20	0	20