



SLOVENSKI STANDARD
SIST ISO 11535:2002
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železovine -- Določanje različnih elementov -- Induktivno sklopljena atomska
emisijna spektrometrična metoda

Iron ores -- Determination of various elements -- Inductively coupled plasma atomic
emission spectrometric method

STANDARD PREVIEW

Minerais de fer -- Dosage de divers éléments -- Méthode par spectrométrie d'émission
atomique avec plasma induit par haute fréquence

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INTERNATIONAL
STANDARD

ISO
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First edition
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**Iron ores — Determination of various
elements — Inductively coupled plasma
atomic emission spectrometric method**

*Minerais de fer — Dosage de divers éléments — Méthode par
spectrométrie d'émission atomique avec plasma induit par haute fréquence*

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ISO 11535:1998(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 11535 was prepared by Technical Committee ISO/TC 102, *Iron ores*, Subcommittee SC 2, *Chemical analysis*.

Annexes A and B form an integral part of this International Standard. Annexes C to E are for information only.

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Iron ores — Determination of various elements — Inductively coupled plasma atomic emission 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 problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for the determination of aluminium, calcium, phosphorus, magnesium, manganese, silicon and titanium in iron ores by inductively coupled plasma atomic emission spectrometry (ICP-AES).

This method is applicable to the concentration ranges given in table 1, in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

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Table 1 — Concentration ranges

Element	Range of concentration % (m/m)
Al	0,02 to 5
Ca	0,01 to 8
Mg	0,01 to 3
Mn	0,01 to 3
P	0,013 to 2
Si	0,1 to 8
Ti	0,01 to 0,2

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 1042:1998, *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 by fusion in sodium carbonate/sodium tetraborate flux and dissolution of the cooled melt in hydrochloric acid.

Dilution to volume and measurement on an ICP spectrometer. Reading of final results from a calibration graph prepared using standard solutions.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only water that conforms to grade 2 of ISO 3696:1987.

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4.1 Iron oxide (Fe_2O_3), of minimum purity 99,99 % (m/m).

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4.2 Sodium carbonate (Na_2CO_3), anhydrous.
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To produce final impurity levels in solution below the detection limits determined or suggested in the performance test, high quality grade is required.

4.3 Sodium tetraborate ($\text{Na}_2\text{B}_4\text{O}_7$), anhydrous.

The same purity criteria as for the sodium carbonate are required.

4.4 Hydrochloric acid, concentrated, ρ 1,16 g/ml to 1,19 g/ml.

The same purity criteria as for the sodium carbonate are required.

4.5 Hydrochloric acid, ρ 1,16 g/ml to 1,19 g/ml, diluted 1 + 1.

Volumes of HCl and water should take into account the water already present in the “concentrated acid”. To prepare a true 1 + 1 solution, the specific gravity of 1,19 g/ml HCl must be taken as a reference to provide a constant-boiling mixture.

4.6 Nitric acid, concentrated, ρ 1,4 g/ml.

The same purity criteria as for the sodium carbonate are required.

4.7 Stock solutions

4.7.1 Phosphorus, 1 000 µg/ml.

Dry approximately 10 g of potassium dihydrogen orthophosphate (KH_2PO_4) at 110 °C until a constant mass is reached, and cool in a desiccator. Dissolve 4,393 6 g in about 200 ml of water in a 1 000 ml one-mark volumetric flask. When the dissolution is complete, dilute to volume with water, and mix.

4.7.2 Manganese, 1 000 µg/ml.

Dissolve 1,000 0 g of high-purity manganese metal in 20 ml of hydrochloric acid (4.5) in a covered tall-form beaker with heating. When dissolution is complete, cool, transfer to a 1 000 ml one-mark volumetric flask, dilute to volume with water and mix.

4.7.3 Magnesium, 1 000 µg/ml.

Dissolve 1,000 0 g of high-purity magnesium metal in 20 ml of hydrochloric acid (4.5) in a covered tall-form beaker with heating. When dissolution is complete, cool, transfer to a 1 000 ml one-mark volumetric flask, dilute to volume with water and mix.

4.7.4 Silicon, 1 000 µg/ml.

Accurately weigh 2,139 3 g of pure silicon oxide (finely ground, previously heated at 1 000 °C for 45 min) into a platinum crucible (5.2). Mix with 5 g of sodium carbonate (4.2), and melt in a furnace at 1 000 °C for 15 min. Dissolve the melt in 100 ml of warm water and transfer to a 1 000 ml one-mark volumetric flask; increase the volume to approximately 500 ml with water, add 20 ml of hydrochloric acid (4.5), dilute to volume with water and mix. Store in a polyethylene flask.

4.7.5 Aluminium, 1 000 µg/ml.

Dissolve 1,000 0 g of high-purity aluminium metal in 20 ml of hydrochloric acid (4.5) in a covered tall-form beaker. Add about 4 drops of nitric acid (4.6). When dissolution is complete, add about 20 ml of water and heat to liberate oxides of nitrogen. Cool and transfer to a 1 000 ml one-mark volumetric flask, dilute to volume with water and mix.

4.7.6 Titanium, 1 000 µg/ml.

Dissolve 1,000 0 g of high-purity titanium metal in 100 ml of hydrochloric acid (4.5) in a covered tall-form beaker with heating. When dissolution is complete, cool, transfer to a 1 000 ml one-mark volumetric flask, dilute to volume with hydrochloric acid (4.5) and mix.

4.7.7 Calcium, 1 000 µg/ml.

Dry approximately 10 g of calcium carbonate (CaCO_3) at 110 °C until a constant mass is reached and cool in a desiccator. Dissolve 2,497 2 g in 20 ml of hydrochloric acid (4.5) in a covered tall-form beaker with heating. When dissolution is complete, cool, transfer to a 1 000 ml one-mark volumetric flask, dilute to volume with water and mix.

4.8 Calibration and reference solutions

Calibration solutions are defined as the solutions required for plotting the calibration graphs of the elements analysed. Their concentration ranges in solution, expressed in micrograms per millilitre, are determined with reference to the performance parameter values and the linearity response of the instrument. A minimum of 10 solutions is necessary to cover the concentration ranges given in table 1. For test samples having narrower concentration ranges, calibration solutions must be prepared to cover the region of interest. If the element concentration in solution exceeds $5\,000 \times$ detection limit (DL), a separate calibration graph must be prepared to cover the range.

In the case of non-linearity, either a less sensitive line is to be used or appropriate dilutions of sample and calibration solutions is to be carried out.

NOTE For the suggested lines shown in table 2, the calibration solutions prepared as recommended in annex C will be in agreement with the performance test figures.

To comply with the requirements of similarity between the test sample and the calibration solutions, iron, flux and acids must be added (see note 1 to table in annex C). For each calibration solution, the procedure recommended in 7.4.1 is followed, replacing the test sample with the equivalent amount of iron oxide (4.1). Prior to the final dilution to 200 ml, the stock solutions and hydrochloric acid (4.5) are added in sufficient amounts to retain the final acid concentration (40 ml of 1 + 1) suggested in annex C.

In addition, to comply with the requirements of similarity, calibration solutions and test samples are prepared from reagents taken from the same containers, to minimize purity differences between batches.

5 Apparatus

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

5.1 Analytical balance, capable of weighing to the nearest 0,000 1 g.

5.2 Platinum or suitable platinum-alloy crucibles, having a minimum volume of 40 ml.

5.3 Bunsen burner, having appropriate fuel/oxidant ratio to provide a minimum temperature of 500 °C.

5.4 Muffle furnace, to provide a minimum temperature of 1 020 °C.

5.5 Combined hotplate-magnetic stirrer.

5.6 Stirring bars, PTFE-coated, 10 mm long.

5.7 ICP spectrometer.

Any conventional ICP spectrometer may be used, provided that the instrument has been initially set up according to the manufacturer's recommendations and that it complies with the performance test (7.4.2.2) carried out prior to the measurements.

Suggested analytical lines are shown in table 2. These lines are found to be relatively free of significant interferences from the matrix elements, but they must be carefully evaluated for spectral interference, background and ionization prior to their adoption. Failure to attain the recommended performance parameters may indicate an interference.

For the analysis of samples having concentrations in the BEC region or lower, as defined in table 3, careful assessment of the need for background correction for the particular line chosen is recommended prior to calibration and analysis.

Table 2 — Suggested analytical lines

Element	Wavelength nm
Al	396,15 or 308,22
Ca	393,36 or 317,93
Mg	279,55 or 279,08
Mn	257,61
P	178,29 ¹⁾
Si	251,61 or 288,16
Ti	334,94 or 336,12

1) Check and correct if necessary for interference by Mn.

6 Sampling and samples

6.1 Laboratory sample

For analysis, use a laboratory sample of minus 100 µm 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 µm.

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 sample at 105 °C ± 2 °C in accordance with 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 B, 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 the 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 appropriate recalibration in either case.

7.2 Test portion

Taking several increments, weigh, to the nearest 0,000 2 g, approximately 0,5 g of the predried test sample obtained in accordance with 6.2.

NOTE The test portion should be taken and weighed quickly 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.

For the blank test, the equivalent amount of pure iron oxide (4.1) shall be used in place of the test sample.

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. Where a certified reference material is not available, a reference material may be used (see 8.4.4).

Where 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 used are from the same reagent bottles.

Where 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

Add 0,8 g of sodium carbonate (4.2) to a platinum or suitable platinum-alloy crucible (5.2), transfer the test portion (see 7.2) to the crucible and mix well using a platinum or stainless-steel rod. Add 0,4 g of sodium tetraborate (4.3) and repeat the mixing using the metal rod. Pre-fuse the mixture to homogenize it.

NOTE 1 The pre-fusion step may be carried out using a bunsen burner having a metallic holder to provide manual agitation. The crucible temperature at this stage should reach the range 350 °C to 450 °C (slightly dull red heat). The mixture melts within 2 min to 3 min without effervescence, and is completely fluid and ready for high temperature fusion within 5 min.

After the pre-fusion, place the crucible in a muffle furnace (5.4) set at 1 020 °C for 15 min. Remove the crucible and gently swirl the melt as it solidifies. Allow to cool, then place a PTFE-coated stirring bar (5.6) in the crucible and place the crucible in a 250 ml low-form beaker. Add 40 ml of hydrochloric acid (4.5), directly into the crucible, and 30 ml of water, into the beaker; cover and heat with stirring on a magnetic stirrer-hotplate (5.5) until dissolution of the melt is complete.

NOTES

- 2 The temperature of the recovery solution should be kept at approximately 70 °C.
- 3 Continuous manual swirling is acceptable as an alternative to magnetic stirring.

Remove and rinse the crucible and stirrer. Cool the solution and immediately transfer to a 200 ml one-mark volumetric flask. Dilute to volume with water and mix. (This is the test solution.)

NOTES

- 4 The immediate transfer of the recovered solution to the 200 ml volumetric flask and the making up to volume prevent reprecipitation.
- 5 The recommended dilution to 200 ml provides element concentrations in solution compatible with the performance test figures given in table 3. Higher dilution rates may be required to cope with instrument linear response at the high concentration ranges. In such situations, the calibration solutions are diluted in the same proportions.

The test is based on the determination of the following three parameters:

- detection limit (DL);
- background equivalent concentration (BEC);
- short-term precision ($RSDN_{min}$).

The definitions of these terms and the procedure for their evaluation are given in annex A.

The procedure shall be carried out as many times as necessary, with the optimization of the instrument parameters after each round, until the figures obtained are lower than those given in table 3. For elements present in the sample solutions at concentrations higher than $5\,000 \times DL$, the $RSDN$ is the only performance parameter to be assessed, and the target values are lower than those given in table 3 for $RSDN_{min}$.

Table 3 — Recommended performance parameters

Element	DL µg/ml	BEC µg/ml	$RSDN_{min}$ %
Al	0,04	2,46	0,87
Ca	0,02	1,04	1,04
Mg	0,03	0,38	0,75
Mn	0,01	0,29	0,89
P	0,07	2,15	1,04
Si	0,07	2,67	0,95
Ti	0,01	0,24	0,78

7.4.3 Measurements

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7.4.3.1 Calibration solutions

Aspirate the calibration solutions in order of increasing concentration, starting with the zero calibration solution. Aspirate water between each solution and repeat the measurements at least twice. Take the average of the two readings.

NOTE After initial calibration has been established, a two-point recalibration procedure can be used for routine analysis. In this case, proceed in accordance with 8.3.

7.4.3.2 Test solutions

Immediately after aspiration of the calibration solutions, commence running the first test solution, followed by the certified reference material (CRM). Continue aspirating test solutions and CRMs alternately. Aspirate water between each measurement. This procedure should preferably be repeated at least twice.

8 Calculation of results

8.1 Calibration graph

Prepare a calibration graph by plotting the intensity values obtained from the calibration solution against its equivalent element concentration.

Read the intensity values for the test solution and obtain their respective concentration values from calibration graphs.