SLOVENSKI STANDARD

SIST ISO 9516-1:2006

01-oktober-2006

Ģelezove rude - Doloļ evanje razliļ nih elementov z rentgensko fluorescentno spektrometrijo - 1. del: Celovit postopek

Iron ores - Determination of various elements by X-ray fluorescence spectrometry - Part 1: Comprehensive procedure

Minerais de fer - Dosage de divers éléments par spectrométrie de fluorescence de rayons X - Partie 1: Procédure détaillée

Ta slovenski standard je istoveten z: ISO 9516-1:2003

ICS:

73.060.10	Železove rude	Iron ores

SIST ISO 9516-1:2006 en

INTERNATIONAL STANDARD

ISO 9516-1

First edition
2003-04-01

Iron ores — Determination of various elements by X-ray fluorescence spectrometry —

Part 1:
Comprehensive procedure

Minerais de fer — Dosage de divers éléments par spectrométrie de fluorescence de rayons X —

Partie 1: Procédure détaillée

Reference number
ISO 9516-1:2003(E)

SIST ISO 9516-1:2006

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Published in Switzerland

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 9516-1 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

This first edition, together with ISO 9516-2, cancels and replaces ISO 9516:1992 by the augmentation of the range of elements under analysis and the diversification into two procedures.

ISO 9516 consists of the following parts, under the general title *Iron ores — Determination of various elements by X-ray fluorescence spectrometry*:

- *Part 1: Comprehensive procedure*
- *Part 2: Simplified procedure*

Introduction

In this part of ISO 9516, Table 1 indicates that some determinations may be used for referee purposes and others for routine analysis only.

A simplified procedure for routine use with all determination will be published in ISO 9516-2.

INTERNATIONAL STANDARD **ISO 9516-1:2003(E)**

Iron ores — Determination of various elements by X-ray fluorescence spectrometry —

Part 1:
Comprehensive procedure

WARNING — This part of ISO 9516 may involve hazardous materials, operations and equipment. This part of ISO 9516 does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this part of ISO 9516 to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This part of ISO 9516 sets out a wavelength dispersive X-ray fluorescence procedure for the determination of iron, silicon, calcium, manganese, aluminium, titanium, magnesium, phosphorus, sulfur, potassium, tin, vanadium, chromium, cobalt, nickel, copper, zinc, arsenic, lead and barium in iron ores. The method has been designed to cope with iron ores having high ignition losses.

The method is applicable to iron ores regardless of mineralogical type. The concentration range covered for each of the component elements is given in Table 1. The determination of total iron cannot be used for referee purposes.

Table 1 — Range of application of the method

Component element	Concentration range for referee purposes %	Concentration range for analysis %
Fe		38 to 72
Si	0,2 to 6,5	0,2 to 6,5
Ca	0,019 to 12,7	0,019 to 12,7
Mn	0,02 to 0,82	0,02 to 0,82
Al	0,1 to 3,5	0,1 to 3,5
Ti	0,016 to 4,7	0,016 to 4,7
Mg	0,2 to 2,0	0,2 to 2,0
P	0,006 to 0,6	0,006 to 0,6
S	0,04 to 0,6	0,007 to 0,6
K	0,008 to 0,45	0,012 to 0,45
Sn		0,006 to 0,015
V	0,001 7 to 0,3	0,001 7 to 0,3
Cr		0,006 to 0,024
Co		0,006 to 0,018
Ni		0,011 to 0,013
Cu		0,012 to 0,061
Zn	0,006 9 to 0,166	0,005 to 0,166
As		0,008 to 0,06
Pb	0,018 to 0,32	0,018 to 0,32
Ba		0,036 to 0,4

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3082:1998, *Iron ores — Sampling and sample preparation procedures*

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

3 Principle

The glass discs for X-ray fluorescence measurement are prepared by incorporating the test portion of the iron ore sample, via fusion, into a borate glass disc using a casting procedure. By using a fused glass disc, particle size effects are eliminated. Sodium nitrate is added to the flux to ensure complete oxidation of all components, particularly iron and sulfur. Any of three methods for glass disc preparation may be used: two use lithium borate as flux; the other uses sodium borate.

X-ray fluorescence measurements are based on the “line only” principle. It is not necessary to measure backgrounds on each glass disc, as background equivalent concentrations (BEC) are determined on several blank glass discs at the line position using concentration-based line-overlap corrections. If desired, backgrounds can be measured to obtain net line intensities. The method is applicable to data from simultaneous and sequential X-ray fluorescence spectrometers.

The method relies on measuring all components of the sample, other than volatiles. If some components are not measured, then errors will result in the measured components (see 7.2.2).

Calibration is carried out using pure chemicals. Results are obtained after matrix corrections for inter-element effects.

4 Reagents and materials

During analysis, only reagents of recognized high purity shall be used.

NOTE 1 Where reagents have been ignited, they should be covered during cooling in the desiccator and weighed as soon as possible.

NOTE 2 Reagents 4.2, 4.5, 4.7, 4.8, 4.9, 4.11, 4.13, 4.15, 4.16, 4.18 and 4.20 are used only for the preparation of the synthetic calibration standard, and are not required if the synthetic calibration standard is available commercially.

4.1 **Silicon dioxide,** (SiO_2), nominally 99,999 % SiO_2

The silicon dioxide shall contain less than 3 µg/g of each of the other elements listed in Table 1. It shall be heated to 1 000 °C in a platinum crucible for a minimum of 2 h and cooled in a desiccator.

4.2 **Aluminium oxide,** (Al_2O_3), analytical reagent grade, α form

If the α form is used, it shall be heated to 1 000 °C in a platinum crucible for a minimum of 2 h. If the aluminium oxide is not the α form, it shall be converted to the α form by heating to 1 250 °C in a platinum crucible for a minimum of 2 h. It shall be cooled in a desiccator and weighed as soon as it is cool.

4.3 **Iron(III) oxide,** (Fe_2O_3), nominally 99,999 % Fe_2O_3

The iron(III) oxide shall contain less than 3 µg/g of each of the other elements listed in Table 1. It shall be heated at 1 000 °C in a platinum crucible for a minimum of 1 h and cooled in a desiccator.

4.4 **Titanium dioxide,** (TiO_2)

Analytical grade titanium dioxide shall be heated at 1 000 °C in a platinum crucible for a minimum of 1 h and cooled in a desiccator.

Phosphorus is a common impurity in TiO_2 and a reagent low in phosphorus shall be selected. The selected reagent shall be checked, as even nominally high-purity reagents can be significantly contaminated, e.g. a supposed 99,99 % TiO_2 grade reagent has been found to contain about 0,5 % P_2O_5.

4.5 **Potassium dihydrogen orthophosphate,** (KH_2PO_4)

Analytical grade potassium dihydrogen orthophosphate shall be dried at 105 °C for 1 h and cooled in a desiccator.

4.6 **Calcium carbonate,** ($CaCO_3$)

Analytical grade calcium carbonate shall be dried at 105 °C for 1 h and cooled in a desiccator.

4.7 **Calcium sulfate,** ($CaSO_4{\cdot}2H_2O$)

Analytical grade calcium sulfate dihydrate shall be dehydrated at 700 °C for 1 h and cooled in a desiccator.

4.8 **Manganese oxide,** (Mn_3O_4)

Manganese oxide shall be prepared by heating analytical grade manganese oxide (MnO_2, MnO or Mn_3O_4) for 15 h at 1 000 °C in a platinum crucible and then cooling. The lumpy material shall be crushed to a fine powder, heated for 1 h at 200 °C and cooled in a desiccator.

4.9 **Magnesium oxide,** (MgO)

Analytical grade magnesium oxide shall be dried in a platinum crucible by slowly heating from room temperature to 1 000 °C. After 1 h at 1 000 °C, the crucible containing the magnesium oxide shall be placed in a desiccator and weighed as soon as it is cool, as magnesium oxide readily absorbs carbon dioxide from the atmosphere.

4.10 **Sodium nitrate,** ($NaNO_3$)

Analytical grade sodium nitrate shall be dried at 105 °C for 1 h and cooled in a desiccator.

4.11 **Tin oxide,** (SnO_2)

Analytical grade tin oxide shall be heated at 400 °C for a minimum of 1 h and cooled in a desiccator.

4.12 **Vanadium(V) oxide,** (V_2O_5)

Analytical grade vanadium(V) oxide shall be heated at 400 °C for a minimum of 1 h and cooled in a desiccator.

4.13 **Chromium(III) oxide,** (Cr_2O_3)

Analytical grade chromium(III) oxide shall be heated at 400 °C for a minimum of 1 h and cooled in a desiccator.

4.14 **Cobalt oxide,** (Co_3O_4)

Analytical grade cobalt oxide shall be heated at 400 °C for a minimum of 1 h and cooled in a desiccator.

4.15 **Nickel oxide,** (NiO)

Analytical grade nickel oxide shall be heated at 400 °C for a minimum of 1 h and cooled in a desiccator.

4.16 **Copper oxide,** (CuO)

Analytical grade copper oxide shall be heated at 400 °C for a minimum of 1 h and cooled in a desiccator.

4.17 **Zinc oxide,** (ZnO)

Analytical grade zinc oxide shall be heated at 400 °C for a minimum of 1 h and cooled in a desiccator.

4.18 **Di-sodium hydrogen arsenate,** ($Na_2HAsO_4 \cdot 7H_2O$)

The analytical grade reagent shall be weighed as received.

4.19 **Lead oxide,** (PbO)

Analytical grade lead oxide shall be heated at 400 °C for a minimum of 1 h and cooled in a desiccator.

4.20 **Barium carbonate,** ($BaCO_3$)

Analytical grade barium carbonate shall be heated at 105 °C for a minimum of 1 h and cooled in a desiccator.

4.21 **Ammonium iodide,** (NH_4I)

Laboratory reagent grade ammonium iodide need not be dried, but shall be stored in a desiccator.

4.22 Desiccant

The desiccant shall be freshly regenerated self-indicating silica gel.

4.23 Flux

4.23.1 General

Flux A, flux B or flux C, as described in 4.23.2, 4.23.3 and 4.23.4, may be used. The levels of contamination in the flux shall be checked (see 9.1). Because levels of contamination may vary from batch to batch, the same batch of flux shall be used for all discs (iron ore, blank and calibration) involved in the batch of determinations.

4.23.2 Flux A

Flux A shall be prepared by fusion of a mixture of anhydrous lithium tetraborate ($Li_2B_4O_7$) and anhydrous lithium metaborate ($LiBO_2$) using the procedure specified in Annex A. Flux shall be dried at 500 °C for a minimum of 4 h and stored in a desiccator.

4.23.3 Flux B

Flux B shall be prepared using sodium tetraborate using the procedure specified in Annex B. Flux shall be dried at 500 °C for a minimum of 4 h and stored in a desiccator.

4.23.4 Flux C

Flux C shall be prepared using lithium tetraborate using the procedure specified in Annex B. Flux shall be dried at 500 °C for a minimum of 4 h and stored in a desiccator.

NOTE If this flux is used, sulfur will not be reported.

4.24 Calibration standard

Two independent (i.e. prepared on different days) batches (labelled Day 1 and Day 2) of calibration standard shall be prepared by the procedure specified in Annex C. The composition of the calibration standard, given in Table 2, approximates that of an iron ore. The contents of some elements are higher than would be expected in an iron ore, but this is advantageous for obtaining a reliable calibration.

Prior to weighing, a sufficient aliquot of the calibration standard shall be heated at 900 °C for 20 min and cooled in a desiccator.

Table 2 — Composition of the calibration standard

Component element	Content %	Oxide content %
Fe	44,764	64,000 Fe_2O_3
Si	4,44	9,500 SiO_2
Ca	3,067	4,2913 CaO
Mn	1,441	2,000 Mn_3O_4
Al	2,65	5,000 Al_2O_3
Ti	0,899	1,500 TiO_2
Mg	3,016	5,000 MgO
P	1,16	2,660 P_2O_5
S	0,921	2,300 SO_3
K	1,46	1,758 9 K_2O
Sn	0,157 5	0,200 SnO_2
V	0,112 0	0,200 V_2O_5
Cr	0,136 8	0,200 Cr_2O_3
Co	0,146 8	0,200 Co_3O_4
Ni	0,157 2	0,200 NiO
Cu	0,159 8	0,200 CuO
Zn	0,160 7	0,200 ZnO
As	0,084 7	0,111 8 As_2O_3
Pb	0,185 7	0,200 PbO
Ba	0,179 1	0,200 BaO
Na	0,052 0	0,070 1 Na_2O

5 Apparatus

5.1 General

The sample may be fused with the flux in a crucible and then poured into a separate mould or, if an appropriately shaped crucible is used, the fusion may be carried out and the glass allowed to cool in the same crucible. Both methods will produce glass discs of the same quality.

A conventional electric furnace, high-frequency furnace, or a gas burner may be used for heating.

There are disc-making machines commercially available, and these may be used to fuse and cast the discs.

A platinum lid may be used to cover the crucible if fusing in a furnace, but not if fusing over a flame, as this enhances sulfur loss.

Where a high-frequency furnace or a gas burner is used for heating, a check shall be made to determine if sulfur is lost during disc preparation. A mixture that contains 90 % Fe_2O_3 and 10 % $CaSO_4$ shall be prepared and used to prepare replicate discs using normal fusion times and times of twice and thrice normal. The intensity of SKα from the discs should not vary by more than 2 % relative.

5.2 Analytical balance, capable of weighing to four decimal places.

5.3 Crucible and mould

5.3.1 General

The crucible and mould shall be made from a non-wetting platinum alloy.

NOTE 1 Either platinum/gold or platinum/gold/rhodium alloys are suitable.

If more than one crucible or more than one mould is used for casting, these crucibles or moulds shall all be used in the specimen preparation test in Annex D.

NOTE 2 It is essential to use all of the crucibles or moulds, as casting vessels may become distorted with use, giving the analytical surface a curvature that will result in error.

Sometimes, even undistorted crucibles or moulds give curvatures unique to the particular crucible or mould.

5.3.2 Crucible

Where the crucible is used for fusion only, it shall have sufficient capacity to hold the flux and sample required for fusion. Where the crucible is used as a mould as well as for fusion, it shall have a flat bottom, to enable production discs to fit the spectrometer.

5.3.3 Mould

Because the bottom of the disc is the analytical surface, the inside bottom surface of the mould shall be flat and shall be polished regularly with approximately 3 μm diamond paste to ensure that the glass disc releases easily from the mould. To prevent deformation through repeated heating and cooling, the base shall be greater than 2 mm thick.

5.4 Electric furnace, capable of maintaining a temperature of at least 1 050 °C.

The furnace shall be capable of maintaining higher temperatures where it is to be used for converting Al_2O_3 to the α form (1 250 °C), or for preparing flux A (1 100 °C).

The furnace may be of a conventional type with heating elements, or may be a high-frequency furnace. The furnace shall be cleaned regularly to prevent contamination of the samples.

5.5 Gas-oxygen burner

Where fusions are made over a gas-oxygen flame, provision shall be made for oxygen enhancement of the flame to minimize sulfur loss and crucible contamination. The temperature of the melt shall be in the range 1 000 °C to 1 050 °C. The temperature shall be checked using an optical pyrometer while the crucible contains several grams of flux. Alternatively, if an optical pyrometer is not available, about 3 g of potassium sulfate (m.p. 1 069 °C) shall be added to the crucible and the flame adjusted so that it all just melts in the open crucible. A gas burner may be used for heating the mould, and it shall be adjusted so that the mould is a bright red heat (approximately 950 °C). A Meker burner shall not be used, as loss of sulfur and the uptake of iron from the glass into the platinum ware may result.

5.6 Desiccator

5.7 Spatulas, non-magnetic, for weighing of the test portion and for mixing.

Vibrating spatulas are not acceptable, because they can lead to segregation of the sample.

5.8 X-ray fluorescence spectrometer, of any wavelength dispersive, vacuum (or helium) path type, X-ray fluorescence spectrometer, provided that the instrument has been checked. Performance checks shall be carried out in accordance with the precision tests set out in Annex E, accumulating at least 2×10^7 counts for each measurement.

The dead time for FeKα is determined in the method described in Annex F, and this dead time may be used for all elements when using a sequential instrument. However, where separate counting channels are used for the different elements (simultaneous instruments), or where the detector is changed, the dead time of each channel shall be determined independently. The procedure is given in Annex F.

5.9 Ultrasonic bath, optional. It may be used to aid cleaning of the platinum ware.

5.10 Cooling device

NOTE It is recommended that the mould and glass be cooled using an air jet. Commercial disc-making machines use this method. A drawing of a suitable device is given in Annex G.

Whatever the method of cooling, it is vital that samples be treated identically, as the curvature of the analytical surface of the disc depends on the rate of cooling.

6 Sampling and samples

Samples shall be taken and prepared in accordance with ISO 3082. The predried test samples shall be prepared according to the procedure specified in ISO 7764. The calibration standards shall be heated to 900 °C for 20 min prior to weighing and then cooled in a desiccator.

7 Procedure

7.1 Preparation of discs

7.1.1 General

Independent duplicate sets (Day 1 and Day 2) of test samples, blanks and calibration samples shall be prepared. The expression “independent” implies that the repetition of the procedure be carried out at a different time or by a different operator.

The operator shall have demonstrated the ability to consistently make discs with high precision. This ability shall be verified each month by carrying out the procedure given in Annex D.