



**Technical
Specification**

ISO/TS 9516-2

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

**Part 2:
Single element calibration
procedure**

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

A list of all parts in the ISO 9516 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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

Part 2: Single element calibration procedure

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

1 Scope

This document sets out a wavelength dispersive X-ray fluorescence procedure for the determination of various elements in iron ores. The method is applicable to iron ores regardless of mineralogical type.

2 Normative references

The following documents are referred to in the text in such a way that some of all of their content constitutes requirements of this document. For dated reference, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 648, *Laboratory glassware — Single-volume pipettes*

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

ISO 2596, *Iron ores — Determination of hygroscopic moisture in analytical samples — Gravimetric, Karl Fischer and mass-loss methods*

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

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

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

ISO 8655-2, *Piston-operated volumetric apparatus — Part 2: Pipettes*

ISO 11323, *Iron ore and direct reduced iron — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11323 apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses.

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

4 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 may be added to the flux to ensure complete oxidation of all components, particularly iron and sulfur. Any of four methods for glass disc preparation may be used: three use lithium borate as flux; the other uses sodium borate.

X-ray fluorescence measurements are based on the “line only” principle. If desired, backgrounds can be measured to obtain net line intensities. This method is applicable to data from simultaneous and sequential X-ray fluorescence spectrometers.

This method relies on measuring all components of the sample, other than volatiles. If some components are not measured, errors will result in the measured components (see [8.5.2](#)).

Results are obtained after matrix corrections for inter-element effects.

5 Reagents and materials

During analysis, only reagents of recognized high purity, and only grade 2 water as specified in ISO 3696 shall be used.

Where reagents have been ignited, they shall be stored during cooling in a desiccator and weighed as soon as possible.

5.1 Silicon dioxide, (SiO₂), nominally 99,999 % SiO₂.

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.

5.2 Aluminium oxide, (Al₂O₃), 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 cooled.

5.3 Iron(III) oxide, (Fe₂O₃), purity of 99,995 % or more Fe₂O₃.

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.

5.4 Titanium dioxide, (TiO₂).

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 TiO₂ 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 % TiO₂ grade reagent has been found to contain about 0,5 % P₂O₅.

5.5 Potassium dihydrogen orthophosphate solution, (KH₂PO₄).

Potassium dihydrogen orthophosphate KH₂PO₄ of purity 99,0 % or more shall be dried at 105 °C for 1 h and cooled in a desiccator. 3,481 g of the dried potassium dihydrogen orthophosphate shall be dissolved in 100 ml water in a volumetric flask. One ml of this solution contains 7,92 mg phosphorous and 10,0 mg potassium.

5.6 Calcium carbonate, (CaCO₃).

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

5.7 Manganese (II) sulfate hydrate solution, (MnSO₄·5H₂O).

8,777 (= 5,497 + 5 × 0,656) g of manganese sulfate hydrate MnSO₄·5H₂O of purity 99 % or more shall be dissolved in 100 ml water in a volumetric flask. Manganese sulfate monohydrate (MnSO₄·H₂O) may be used as an alternative reagent of purity 99 % or more and 6,153 (= 5,497 + 0,656) g of the reagent shall be dissolved in 100 ml water in a volumetric flask. One millilitre of the solution contains manganese 20,0 mg and sulfur 11,67 mg.

5.8 Magnesium nitrate hexahydrate, [Mg(NO₃)₂·6H₂O], purity of 99,0 % or more.

5.9 1 000 mg/l element standards for V, Cr, Co, Ni, Cu, Zn, As, Pb and Ba.

Single element standard water solutions with 1 000 mg/l concentration and 0,5 to 6 % of nitric acid shall be used. It is recommended to use commercially available standards. When standard solutions are prepared in the laboratory, 1,000 g of pure metal (99,99 % or more) shall be dissolved in diluted nitric acid. Water shall be added to this solution such that the prepared standard solution is 1 000 ml.

For the standard water solution for Cr, a solution of potassium dichromate solution (K₂Cr₂O₇) with nitric acid may be used.

5.10 Sodium nitrate, (NaNO₃).

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

5.11 Ammonium iodide, (NH₄I).

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

5.12 Desiccant.

The desiccant shall be freshly regenerated self-indicating silica gel. <https://standards.iteh.ai>

5.13 Flux.

5.13.1 General

One of fluxes from Flux A, Flux B, Flux C or Flux D, as described in [5.13.2](#), [5.13.3](#) and [5.13.4](#), shall be used. The levels of contamination in the flux shall be checked (see [10.1](#)). Because levels of contamination can 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.

5.13.2 Flux A and Flux D

Flux A and Flux D shall be prepared by fusion of a mixture of anhydrous lithium tetraborate (Li₂B₄O₇) and anhydrous lithium metaborate (LiBO₂) 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.

5.13.3 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.

5.13.4 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.

6 Apparatus

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 % Fe₂O₃ and 10 % CaSO₄ shall be prepared and used to prepare replicate discs using normal fusion times and times of twice and thrice normal. The intensity of SK α from the discs should not vary by more than 2 % relative.

Single-volume pipettes, one-mark volumetric flasks and piston pipettes to be used shall comply with specifications of ISO 648, ISO 1042 and ISO 8655-2, respectively.

6.1 Analytical balance, capable of weighing to the nearest 0,1 mg.

6.2 Crucible and mould.

The crucible and mould shall be made from a non-wetting platinum alloy. 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 C](#). It is essential to use all of the crucibles or moulds in the test described in [Annex C](#), as casting vessels can 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.

6.2.1 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 with minimum curvature.

6.2.2 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 μ m 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. The mould shall have a flat bottom, to enable production of discs with minimum curvature.

6.3 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 Al₂O₃ 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.

6.4 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 put in an empty 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 can result.

6.5 Desiccator.

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

6.7 **X-ray fluorescence spectrometer**, of any wavelength dispersive, vacuum (or helium) path, simultaneous or sequential 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 D](#), accumulating at least 2×10^7 counts for each measurement.

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

6.9 Cooling device.

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 I](#).

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.

7 Sampling and samples

7.1 Laboratory sample

For analysis, laboratory samples of $-100 \mu\text{m}$ particle size which has been taken and prepared in accordance with ISO 3082 shall be used. In the case of ores having significant contents of combined water or oxidizable compounds, samples of particle size of $-160 \mu\text{m}$ shall be used.

7.2 Preparation of test samples

7.2.1 General

Depending on the ore type, air-equilibrate test samples shall be prepared in accordance with either ISO 2596 in [7.2.2](#) or ISO 7764 in [7.2.3](#).

7.2.2 Method specified in ISO 2596

The method is applicable to all types of ores.

The laboratory samples shall be thoroughly mixed, and in multiple increments, a test sample shall be extracted in such a manner that it is representative of the entire content in the container. The test sample shall be brought into equilibrium with the laboratory atmosphere in accordance with ISO 2596.

7.2.3 Method specified in ISO 7764

The method is not applicable to the following types of ores:

- a) processed ores containing metallic iron;
- b) natural or processed ores in which the sulfur content is higher than 0,2 %;
- c) natural or processed ores in which the content of combined water is higher than 2,5 %.

The laboratory samples shall be thoroughly mixed and, multiple increments shall be taken, a test sample shall be extracted in such a manner that it is representative of the whole contents of the container.

8 Procedure

8.1 General

The concentration range covered for each of the component elements is given in [Table 1](#).

Table 1 — Range of application of the method

Component element	Concentration range for referee purposes %	Concentration range for analysis %
Fe	31 to 72	31 to 72
Si	0,16 to 11,8	0,16 to 11,8
Ca	0,011 to 13,7	0,011 to 13,7
Mn	0,016 to 2,0	0,016 to 2,0
Al	0,036 to 4,2	0,036 to 4,2
Ti	0,013 to 4,5	0,013 to 4,5
Mg	0,012 to 1,7	0,012 to 1,7
P	0,001 3 to 0,6	0,001 3 to 0,6
S	0,011 to 0,76	0,011 to 0,76
K	0,008 to 0,46	0,008 to 0,46
V	0,002 to 0,32	0,002 to 0,32
Cr	—	0,006 to 0,067
Co	—	0,006 to 0,023
Ni	0,008 to 0,038	0,008 to 0,038
Cu	0,007 to 0,17	0,007 to 0,17
Zn	0,005 to 0,36	0,005 to 0,36
As	—	0,003 to 0,11
Pb	—	0,001 to 0,23
Ba	—	0,011 to 0,74

When the influence of absorption or spectral overlap by trace heavy elements to reporting elements is small enough and can be ignored, those trace heavy elements can be omitted from measuring elements.

The applicable ranges for referee and non-referee methods are also to be determined based on results of international trials.

The operator shall have demonstrated the ability to consistently make discs with high precision. This ability shall be verified by carrying out the procedure given in [Annex C](#).

The operator shall periodically test all moulds according to [Annex C](#), because their shape can become distorted with repeated use.

In preparing discs, great care shall be taken to avoid contamination and, in particular, the crucible in which the fusion is carried out shall be thoroughly cleaned prior to use (see 8.4.7).

8.2 Number of determinations

Carry out two analyses independently for duplicate fused discs prepared on different days in accordance with Annex J for each test sample (see 7.2).

8.3 Check analysis and blank test

In each run, 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 test sample of the certified reference material shall be prepared in the manner appropriate to the type of ore involved.

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

When a new reagent as for flux from a different bottle is used, it is recommended to measure a blank sample to check the impurities and contamination due to sample preparation, before the analysis is carried out on ore sample(s). The blank disc shall be prepared with 100 % Fe₂O₃.

8.4 Preparation of discs

8.4.1 Weighing

Table 2 shows the components used in making the glass discs. Provided that the proportions are kept approximate to those given in Table 2, the masses can be varied to suit mould diameter. If a disc diameter used differs from those given in Table 2, masses should be adjusted to be approximately proportional to the area of the glass disc.

Table 2 — Masses of specimen components

Component	Typical masses g	Mass g	
		Disc diameter	
		32 mm	40 mm
Flux	6,80	3,52 to 6,40	5,50 to 10,00
NaNO ₃	0,40 or 0	0,21 to 0,37 or 0	0,33 to 0,58 or 0
Sample	0,66	0,35 to 0,60	0,55 to 0,94

The specified masses may be weighed as “catch” weights, recording the mass weighed to the nearest 0,001 g for the flux and sodium nitrate portions, and to the nearest 0,000 1 g for the test and calibration portions. If masses used are higher than recommended, crystallization and segregation with consequent cracking are likely to occur as the glass cools.

If desired, ammonium iodide (5.11) can be used as a releasing agent. If added at this stage, no more than 0,01 g shall be added. Alternatively, a smaller amount may be added prior to casting (see 8.4.4). Exceeding the recommended amount of releasing agent of ammonium iodide can introduce error in the titanium result due to the overlap. ILβ₂ interferes with TiKα.

Because the components are hygroscopic, they shall be weighed as soon as possible after reaching room temperature following heating and without any undue delay between each weighing. Weighing may be made directly into the crucible to be used in the fusion, or into a clean glass vial. Because of static effects, glass vials are preferable to plastic. If a vial is used, care shall be taken to ensure complete transfer of the contents into the fusion crucible.

8.4.2 Mixing

Thoroughly mix the components in the crucible using a spatula or similar implement, taking care that no material is lost. The mixing implement used should be free of sharp or pointed edges, in order to ensure that the interior of the crucible is not damaged by scratching. Brush any fine material adhering to the mixing implement back into the crucible. Gently tap the bottom of the crucible on the bench top to ensure that any material adhering to the crucible wall, above the general level of the mixed components, is reincorporated into the bulk of the mix.

It is imperative that the crucible be tapped gently on the bench top, as too severe an impact will result in the loss of some of the finer material and possible deformation of the crucible. Care shall be taken to mix the components thoroughly to aid reproducibility of disc preparation.

8.4.3 Fusion

For samples containing sulfur as sulfide, the fusion mixture is to be preoxidised by heating to 700 °C for 10 min prior to fusion. Place the crucible in the electric furnace (6.3) or on the gas-oxygen burner (6.4) at a temperature of 1 000 °C to 1 050 °C and maintain this temperature for 10 min. At least once during this period, after the sample is dissolved, briefly swirl the mixture. While swirling, incorporate into the melt any material that can be adhering to the sides of the crucible.

If a furnace is used for heating, it can be necessary to remove the crucible from the furnace for the purpose of swirling. When the furnace is opened, the temperature can drop. The specified temperature shall be regained before the time period starts.

8.4.4 Casting

If ammonium iodide was not added as a release agent earlier, it may be added to the melt just prior to casting. In this case, no more than 0,002 g shall be added. Casting is then carried out by one of the following methods.

a) Casting in the crucible.

If the glass is to be cast in the crucible, remove the crucible from the furnace, place on a suitable cooling device (6.9) and allow the glass to solidify.

b) Casting in a separate mould.

If the glass is to be cast in a separate mould, the mould shall be pre-heated over a gas flame to red heat (900 °C to 1 050 °C). While the mould is still hot, pour the melt into the mould from the crucible. Remove the mould from the heat source and place it on the cooling device (6.9) and allow the glass to solidify.

NOTE Failure to ensure that the mould is scrupulously clean prior to casting will result in discs sticking to the mould and possibly cracking.

8.4.5 Visual inspection

Prior to storage, discs shall be inspected visually, paying particular attention to the analytical surface. The discs shall not contain undissolved material, and shall be whole and free from crystallization, cracks and bubbles. Defective discs shall be re-fused in the crucible, or discarded and substitute discs prepared.

8.4.6 Disc storage

As soon as possible (while the glass is still warm), transfer the discs to a desiccator so that absorption of moisture and the possibility of contamination are minimized. When not being measured, discs shall be stored in a clean desiccator.

To avoid contamination of the analytical surface, the specimen shall be handled by its edges and the surface shall not be touched by hand or treated in any way. Specifically, it shall not be washed with water or other solvents, ground or polished.