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

Part 2: ~~Single element calibration~~ Simplified procedure

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Foreword

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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 ~~documents~~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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This document was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 02, *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.

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

Part 2: ~~Single element calibration~~ **Simplified procedure**

WARNING — This document ~~may 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. ~~The concentration range covered for each of the component elements is given in Table 1.~~

Range of application of the method

Component element	Concentration range for referee purposes %	Concentration range for analysis %
Fe	31–72	31–72
Si	0,16–11,8	0,16–11,8
Ca	0,011–13,7	0,011–13,7
Mn	0,016–2,0	0,016–2,0
Al	0,036–4,2	0,036–4,2
Ti	0,013–4,5	0,013–4,5
Mg	0,012–1,7	0,012–1,7
P	0,0013–0,6	0,0013–0,6
S	0,011–0,76	0,011–0,76
K	0,008–0,46	0,008–0,46
V	0,002–0,32	0,002–0,32
Cr	---	0,006–0,067
Co	---	0,006–0,023
Ni	0,008–0,038	0,008–0,038
Cu	0,007–0,17	0,007–0,17
Zn	0,005–0,36	0,005–0,36
As	---	0,003–0,11
Pb	---	0,001–0,23
Ba	---	0,011–0,74

~~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.~~

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

802 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~~ *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: Piston pipettes~~ — *Part 2: Pipettes*

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

813 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/>

824 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](#), [8.5.2](#)).

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

835 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, (SiO_2), nominally 99,999-% SiO_2 .

The silicon dioxide shall contain less than 3- $\mu\text{g/g}$ of each of the other elements listed in [Table 1-Table 1](#). It shall be heated to 1-000- $^\circ\text{C}$ in a platinum crucible for a minimum of 2-h and cooled in a desiccator.

5.2 Aluminium oxide, (Al_2O_3), analytical reagent grade, α form.

If the α form is used, it shall be heated to 1-000- $^\circ\text{C}$ 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- $^\circ\text{C}$ 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, (Fe_2O_3), purity of 99,995-% or more Fe_2O_3 .

The iron(III) oxide shall contain less than 3- $\mu\text{g/g}$ of each of the other elements listed in [Table 1-Table 1](#). It shall be heated at 1-000- $^\circ\text{C}$ in a platinum crucible for a minimum of 1-h and cooled in a desiccator.

5.4 Titanium dioxide, (TiO_2).

Analytical grade titanium dioxide shall be heated at 1-000- $^\circ\text{C}$ 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 .

5.5 Potassium dihydrogen orthophosphate solution, (KH_2PO_4).

Potassium dihydrogen orthophosphate KH_2PO_4 of purity 99,0-% or more shall be dried at 105- $^\circ\text{C}$ 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, (CaCO_3).

Analytical grade calcium carbonate shall be dried at 105- $^\circ\text{C}$ for 1-h and cooled in a desiccator.

5.7 Manganese (II) sulfate hydrate solution, ($\text{MnSO}_4 \cdot 5\text{H}_2\text{O}$).

8,777 (= $(= 5,497 + + 5 \times \times 0,656)$) g of manganese sulfate hydrate $\text{MnSO}_4 \cdot 5\text{H}_2\text{O}$ of purity 99-% or more shall be dissolved in 100-ml water in a volumetric flask. Manganese sulfate monohydrate ($\text{MnSO}_4 \cdot \text{H}_2\text{O}$) 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-ml millilitre of the solution contains manganese 20,0-mg and sulfur 11,67-mg.

5.8 Magnesium nitrate hexahydrate, [$\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$], 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 standard water solution for Cr, solution of potassium dichromate solution ($\text{K}_2\text{Cr}_2\text{O}_7$) with nitric acid may be used.

5.10 Sodium nitrate, (NaNO_3).