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**Iron ores — Determination of  
potassium — Flame atomic absorption  
spectrometric method**

*Minerais de fer — Dosage du potassium — Méthode par spectrométrie  
d'absorption atomique dans la flamme*

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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 13312 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

This second edition cancels and replaces the first edition (ISO 13312:1997), which has been technically revised. It has been updated to alter the manner in which precision data are presented.

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# Iron ores — Determination of potassium — 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 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 flame atomic absorption spectrometric method for the determination of the mass fraction of potassium in iron ores.

This method is applicable to mass fractions of potassium between 0,002 5 % and 0,52 % in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

## 2 Normative references

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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 648, *Laboratory glassware — One-mark pipettes*

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

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 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.

## 4 Principle

The test portion is decomposed by treatment with hydrochloric acid and hydrofluoric acid, followed by evaporation to dryness. The residue is wetted and the evaporation is repeated with a new portion of hydrochloric acid. The residue is dissolved with hydrochloric acid followed by appropriate dilution. The solution is aspirated into the air/acetylene flame of the atomic absorption apparatus.

The absorbance value obtained for potassium is measured in comparison with those obtained from calibration solutions.

## 5 Reagents

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

Reagents are to be selected or purified for the lowest possible blank value.

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

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

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

### 5.4 Background solution

Dissolve 43 g of high-purity iron oxide powder<sup>1)</sup> in 500 ml of hydrochloric acid (5.1). Allow to cool and dilute with water to 1 000 ml.

**5.5 Potassium**, standard solution, 20  $\mu$ g K/ml.

Pulverize about 3 g of high-purity potassium chloride in an agate mortar, dry in an oven at 105 °C to 110 °C for 2 h, and allow to cool to room temperature in a desiccator. Dissolve 1,907 g in water, dilute with water to 1 000 ml in a volumetric flask and mix.

Transfer<sup>2)</sup> 10,0 ml of this solution to a 500 ml volumetric flask, dilute with water to volume and mix.

Store this standard solution in a plastic bottle.

1 ml of this standard solution contains 20  $\mu$ g of potassium.

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## 6 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 the following.

**6.1 Polytetrafluoroethylene (PTFE) beakers**, of capacity 100 ml, provided with PTFE covers.

**6.2 PTFE-coated magnetic stirring bars**.

**6.3 PTFE digestion bomb**.

**6.4 Plastic pipettes**.

**6.5 Plastic volumetric flasks and storage bottles**.

**6.6 Magnetic stirring hotplates**.

NOTE Platinum vessels may be used instead of PTFE beakers.

Except where stated, glass equipment should be avoided, as it could contaminate the solutions.

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1) Instead of iron oxide, the use of metallic iron with a suitable oxidant is permitted. (The alkali content of the oxidant shall be low.)

2) Glass equipment may be used.

To obtain reliable values, the equipment should be cleaned and checked as follows.

- a) Rinse all volumetric ware, including the pipettes used for preparing the calibration solutions, with dilute hydrochloric acid (5.3) before use. Check the calibration regularly or as needed.
- b) Clean PTFE vessels and stirring bars by stirring with 50 ml of dilute hydrochloric acid (5.3) and by heating for 15 min. Discard the rinsings and conduct a blank test in each vessel in turn, exactly as specified in 8.3. If any absorbance value is above the limit specified in 8.3, the cleaning procedure should be repeated or acid reagents of a higher purity should be used. At no stage should the stirring bars be handled with the fingers.
- c) Platinum vessels, exclusively used for potassium analysis according to this International Standard, can be cleaned by the same method as the PTFE vessels [see b)]. Otherwise, they should be pre-cleaned by fusion with lithium tetraborate or lithium borate, until the absorbance readings fall to those for the lithium salt alone.
- d) Rinse storage bottles with dilute hydrochloric acid (5.3) before use.

#### 6.7 Atomic absorption spectrometer.

**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.**

The atomic absorption spectrometer shall meet the following criteria.

- a) Minimum sensitivity: the absorbance of the most concentrated calibration solution (see 8.4.3) 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.

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.

**NOTE** Instrument parameters will vary with each instrument. The following parameters were successfully used in several laboratories and they can be used as guidelines. Solutions were aspirated into an air/acetylene flame of a premix burner.

— hollow cathode lamp, mA	10
— wavelength, nm	766,5
— air flow-rate, l/min	10
— acetylene flow-rate, l/min	2

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.

## 7 Sampling and samples

### 7.1 Laboratory sample

For analysis, use a laboratory sample of minus 100  $\mu\text{m}$  particle size which has been taken and prepared in accordance with ISO 3082. 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.

### 7.2 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  $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$  as specified in ISO 7764. (This is the predried test sample.)

## 8 Procedure

### 8.1 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 are 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, in either case, appropriate recalibration.

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### 8.2 Test portion

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Taking several increments, weigh, to nearest 0,000 2 g, 0,2 g to 0,5 g (depending on the potassium concentration) of the predried test sample obtained in accordance with 7.2.

The test portion should be taken and weighed quickly to avoid reabsorption of moisture.

### 8.3 Blank test and check test

Before proceeding to the treatment of test portions, ensure that the cleaning procedures conducted in 6.6 (see list items), together with the quality of the reagents being used, have produced a blank test value for the potassium determination not greater than the equivalent of 0,002 % (mass fraction) potassium in the ore.

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 7.2.

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 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.

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 will become necessary. Where a certified reference material is not available, a reference material may be used (see 9.2.4).



## 8.4 Determination

To prevent contamination during analysis, the following precautions shall be taken:

- a) finger contact with sample, solutions and stirring bars shall be avoided;
- b) mouth suction of pipettes shall not be permitted.

### 8.4.1 Decomposition of the test portion

Transfer the test portion (8.2) to a 100 ml PTFE beaker (6.1)<sup>3)</sup>. Moisten it with a few drops of water, then add 10 ml of hydrochloric acid (5.1) and 10 ml of hydrofluoric acid (5.2). Add a PTFE-coated magnetic stirring bar (6.2) and cover with a PTFE cover. Adjust the temperature of the magnetic stirring hotplate (6.6) so that a temperature of about 98 °C will be maintained in water in a covered PTFE beaker. Heat, with stirring, for 45 min or until no further dissolution of the test portion occurs. Remove the cover, stop the stirrer, leaving the bar in the solution, and evaporate to dryness. Add 5 ml of hydrochloric acid (5.1) and evaporate again to dryness. Dissolve the salts in 5 ml of hydrochloric acid (5.1) and 40 ml of water, and transfer to a 100 ml one-mark plastic volumetric flask (6.5). Dilute to volume with water and mix.

NOTE If any significant amount of residue remains, conduct the digestion process in a stirred PTFE digestion bomb (6.3) for 45 min at 160 °C.

### 8.4.2 Treatment of the solution

If the concentration of potassium is too high, it is necessary to dilute the test solution. Transfer, using a plastic pipette (6.4),  $y$  ml of the test solution to a 100 ml one-mark plastic volumetric flask, add  $0,1 \times (100 - y)$  ml of the background solution (5.4), dilute with water to volume and mix (see Table 1).

A diluted test solution shall be measured together with a diluted blank test solution, containing the same amount of background solution as the test solution. Prepare the diluted blank test solution as follows: pipette  $y$  ml of the blank test solution into a 100 ml one-mark plastic volumetric flask, add  $0,1 \times (100 - y)$  ml of the background solution, dilute to volume with water and mix.

Table 1 — Dilution guide for test solution

Potassium mass-fraction range %	Aliquot from 100 ml $y$
0,002 to 0,060	—
0,060 to 0,20	30,0
0,20 to 0,60	10,0
0,60 to 1,00	5,0

### 8.4.3 Preparation of the set of calibration solutions

From the potassium standard solution (5.5), prepare calibration solutions as follows:

Using plastic pipettes, transfer 0 ml; 2,0 ml; 5,0 ml; 10,0 ml; 15,0 ml of potassium standard solution (5.5), respectively, to 100 ml one-mark plastic volumetric flasks. Add, using a plastic pipette, 10 ml of the background solution (5.4) to each, dilute with water to volume and mix. These calibration solutions cover the concentration range 0 µg K/ml to 3 µg K/ml and contain 3 000 µg Fe/ml.

Store the calibration solutions in plastic bottles.

3) See note in 6.6.