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Iron ores — Determination of vanadium content — Flame atomic absorption spectrometric methods

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



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Foreword

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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 9684 was prepared by Technical Committee ISO/TC 102, *Iron ores*, Sub-Committee SC 2, *Chemical analysis*.

Annex A forms an integral part of this International Standard. Annexes B and C are for information only.

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Iron ores — Determination of vanadium content — Flame atomic absorption spectrometric methods

1 Scope

This International Standard specifies two flame atomic absorption spectrometric methods for the determination of the vanadium content of iron ores.

Method 1 is applicable to vanadium contents between 0,005 % (*m/m*) and 0,05 % (*m/m*), and Method 2 to vanadium contents between 0,05 % (*m/m*) and 0,5 % (*m/m*) in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

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 1042:1983, *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 7764:1985, *Iron ores — Preparation of predried test samples for chemical analysis*.

3 Principle

3.1 Dissolution

Decomposition of the test portion by digestion with hydrochloric acid in a polytetrafluoroethylene (PTFE) beaker, addition of hydrofluoric and nitric acids and evaporation to dryness. Addition of hydrochloric and boric acids and evaporation to dryness. Dissolution of the salts in hydrochloric and nitric acids (Method 1), or hydrochloric acid (Method 2), and filtration. Ignition and fusion of the residue with sodium carbonate and dissolution of the cooled melt in the test solution.

3.2 Determination

3.2.1 Extraction (Method 1 only)

Oxidation with cerium(IV) solution and addition of orthophosphoric acid and sodium tungstate solution. Extraction of the vanadium complex with a 1 + 1 mixture of 1-pentanol and methyl isobutyl ketone. Treatment of the solvent phase with water and then ascorbic acid solution to return the vanadium to an aqueous phase.

3.2.2 Measurement (Methods 1 and 2)

Addition of aluminium solution to the test solution and dilution to volume. Aspiration into a dinitrogen oxide-acetylene flame in an atomic absorption spectrometer and measurement of the absorbance at a wavelength of approximately 318,5 nm.

4 Reagents

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

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

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

4.3 Hydrofluoric acid, ρ 1,13 g/ml, 40 % (m/m), or ρ 1,19 g/ml, 48 % (m/m).

4.4 Nitric acid, ρ 1,4 g/ml.

4.5 Nitric acid, ρ 1,4 g/ml, diluted 1 + 1. (Method 1 only.)

4.6 Orthophosphoric acid, ρ 1,7 g/ml, diluted 1 + 2. (Method 1 only.)

4.7 Sodium carbonate (Na_2CO_3), anhydrous powder.

4.8 Boric acid (H_3BO_3).

4.9 Ammonium cerium(IV) nitrate $[(\text{NH}_4)_2\text{Ce}(\text{NO}_3)_6]$, 20 g/l solution. (Method 1 only.)

Dissolve 2,0 g of ammonium cerium(IV) nitrate in a mixture of 15 ml of nitric acid (4.5) and 85 ml of water.

4.10 Sodium tungstate ($\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$), 165 g/l solution. (Method 1 only.)

Dissolve 16,5 g of sodium tungstate dihydrate in approximately 70 ml of water, dilute to 100 ml and mix.

4.11 Ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$), 10 g/l solution. (Method 1 only.)

Prepare freshly for each series of tests.

4.12 Pentan-1-ol (*n*-amyl alcohol, 1-pentanol). (Method 1 only.)

4.13 4-Methylpentan-2-one (methyl isobutyl ketone, MIBK). (Method 1 only.)

4.14 Mixed solvent. (Method 1 only.)

Prepare a 1 + 1 volume ratio mixture of pentan-1-ol (4.12) and MIBK (4.13).

4.15 Aluminium chloride ($\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$), 220 g/l solution.

Dissolve 220 g of aluminium chloride hexahydrate in water, add 50 ml of hydrochloric acid (4.1), dilute to 1 000 ml with water and mix.

4.16 Iron oxide, high purity (containing less than 0,002 % V).

4.17 Iron background solution. (Method 2 only.)

Dissolve 90 g of high purity iron oxide (4.16) in 750 ml of hydrochloric acid (4.1) while heating. Cool and carefully add a solution of 30 g of sodium carbonate (4.7) in 200 ml of water. Dilute with water to a final volume of approximately 1 000 ml and mix.

NOTE 1 The use of an equivalent quantity of metallic iron (of similarly low vanadium content) in conjunction with a suitable oxidant is permissible.

4.18 Vanadium standard solution, 1 mg V/ml.

Dry ammonium monovanadate (NH_4VO_3) in an air oven at 100 °C for 1 h and cool to room temperature. Dissolve 2,296 g of the dried product in approximately 600 ml of water, transfer to a 1 000 ml one-mark volumetric flask, dilute to volume with water and mix.

4.19 Diluted vanadium standard solution, 0,2 mg V/ml. (Method 1 only.)

Transfer 20,0 ml of vanadium standard solution (4.18) to a 100 ml one-mark volumetric flask, dilute to volume with water and mix.

4.20 Vanadium calibration solutions. (Method 1 only.)

Transfer 1,8 g of high purity iron oxide (4.16) to each of five 100 ml PTFE beakers (5.1). Proceed according to 7.5.1, incorporating the operation in 7.5.2 to dissolve the evaporated salts.

Transfer 0 ml; 1,00 ml; 2,00 ml; 4,00 ml and 6,00 ml of the diluted standard vanadium solution (4.19) respectively to the beakers. Omit the residue processing steps and add 10 ml of nitric acid (4.5) and 25 ml of water. Evaporate to 50 ml and proceed as specified in 7.5.3.

4.21 Vanadium calibration solutions. (Method 2 only.)

Transfer 0 ml; 1,00 ml; 2,00 ml; 4,00 ml and 6,00 ml of vanadium standard solution (4.18) respectively to five 100 ml one-mark volumetric flasks. Add 10 ml of iron background solution (4.17), 4,0 ml of aluminium chloride solution (4.15) and 0,5 g of boric acid (4.8) to each flask. Dilute to volume with water and mix.

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 (unless otherwise indicated), and

5.1 Polytetrafluoroethylene (PTFE) beakers, of capacity 100 ml, preferably low form.

5.2 Platinum crucibles, of minimum capacity 20 ml.

5.3 Muffle furnace.

5.4 Separating funnels, 100 ml (preferably graduated).

5.5 Atomic absorption spectrometer, equipped with a dinitrogen oxide-acetylene burner.

The atomic absorption spectrometer used in this method will be satisfactory if it meets the following criteria:

- a) Minimum sensitivity — the absorbance of the most concentrated vanadium calibration solution (4.21) is at least 0,25 (see note 4).
- b) Graph linearity — the slope of the calibration graph covering the top 20 % of the concentration range (expressed as a change in absorbance) is not 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, are less than 1,5 % and 0,5 % respectively of the mean value of the absorbance of the most concentrated calibration solution.

NOTES

2 The use of a strip-chart recorder and/or digital read-out device is recommended to evaluate criteria a), b) and c) and for all subsequent measurements.

3 Instrument parameters

— V-hollow cathode lamp, mA	10
— Wavelength, nm	318,5
— Dinitrogen oxide flow rate, l/min	9,5
— Acetylene flow rate, l/min	3,8

The slit width should be sufficiently narrow to substantially reject the stronger emitting but weaker absorbing line at 318,4 nm.

In systems where the values shown above for gas flow rates do not apply, the given ratio of the flow rates (5:2) may still be a useful guideline.

4 To check the instrument criteria when only Method 1 is involved, prepare the highest calibration solution specified for Method 2, omitting the iron background solution.

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 with significant contents of combined water or oxidizable compounds, use a particle size of minus 160 μm .

NOTE 5 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 way 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.)

7 Procedure

7.1 Number of determinations

Carry out the analysis at least in duplicate in accordance with annex A, independently, on one predried test sample.

NOTE 6 The expression "independently" means that the second and any subsequent result is not affected by the previous result(s). For this particular analytical method, this condition implies that the repetition of the procedure shall be carried out either by the same operator at a different time or by a different operator including, in both cases, appropriate recalibration.

7.2 Safety precautions

WARNING — Follow the manufacturer's instructions for igniting and extinguishing the dinitrogen oxide-acetylene flame to avoid possible explosion hazards. Wear tinted safety glasses whenever the burner is in operation.

7.3 Test portion

Taking several increments, weigh, to the nearest 0,0005 g, approximately 2 g (Method 1) or 1 g (Method 2) of the predried test sample obtained in accordance with 6.2.

NOTE 7 The test portion should be taken and weighed quickly, in order to avoid reabsorption of moisture.