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Iron ores — Determination of vanadium —

Part 2:

Flame atomic absorption spectrometric methods

Minerais de fer — Dosage du vanadium —

Partie 2: Méthodes par spectrométrie d'absorption atomique dans la flamme

(Revision of ISO 9684:1991)

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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.

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ISO 9683-2 was prepared by Technical Committee ISO/TC 102, *Iron ore and direct reduced iron*, Subcommittee SC 2, *Chemical analysis*.

This second/third/... edition cancels and replaces the first/second/... edition (ISO 9684:1991), [clause(s) / subclause(s) / table(s) / figure(s) / annex(es)] of which [has / have] been technically revised.

ISO 9683 consists of the following parts, under the general title *Iron ores — Determination of vanadium content*:

- *Part 1: BPHA spectrophotometric methods*
- *Part 2: Flame atomic absorption spectrometric methods*

Iron ores — Determination of vanadium —

Part 2:

Flame atomic absorption spectrometric methods

WARNING — This part of ISO 9683 may involve hazardous materials, operations and equipment. This part of ISO 9683 does not purport to address all of the safety issues associated with its use. It is the responsibility of the user to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This part of ISO 9683 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 0,004% (*m/m*) and 0,06% (*m/m*), and Method 2 to vanadium contents between 0,06% (*m/m*) and 0,5% (*m/m*) in natural iron ores, iron ore concentrates and agglomerates, including sinter products.

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 648, *GenLaboratory glassware — One-mark pipettes*

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

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

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

3 Principle

3.1 Dissolution

The test portion is decomposed by digestion with hydrochloric acid in a polytetrafluoroethylene (PTFE) beaker, hydrofluoric and nitric acids are added and the solution is evaporated to dryness. Hydrochloric and boric acids are added and the solution is again evaporated to dryness. The salts are dissolved in hydrochloric and nitric acids (Method 1), or hydrochloric acid (Method 2), and the solution is filtered. The residue is ignited and fused with sodium carbonate and the cooled melt is dissolved in the test solution.

3.2 Determination

3.2.1 Extraction (Method 1 only)

The solution is oxidized with cerium(IV) solution and phosphoric acid and sodium tungstate solution are added. The vanadium complex is extracted with a 1 + 1 mixture of 1-pentanol and 4-methyl-2-pentanone. The solvent phase is treated with water and then ascorbic acid solution to return the vanadium to an aqueous phase.

3.2.2 Measurement (Methods 1 and 2)

Aluminium solution is added to the test solution, which is diluted to volume. The solution is aspirated into a dinitrogen oxide acetylene-flame in an atomic absorption spectrometer and the absorbance is measured 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 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 Phosphoric 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 Di-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 di-ammonium cerium(IV) nitrate in a mixture of 15 ml of nitric acid (4.5) and 85 ml of water.

- 4.10 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 1-Pentanol, (n-amyl alcohol, 1-pentan-1-ol). (Method 1 only.)
- 4.13 4-Methyl-2-pentanone, (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/litre solution.

Dissolve 220 g of aluminium chloride hexahydrate in water, add 50 ml of hydrochloric acid (4.1), dilute to 1000 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 1000 ml and mix.

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

4.18 Standard vanadium 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 standard vanadium 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 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 is 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 solution.

NOTE 1 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.

NOTE 2 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.

NOTE 3 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 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 μm .

NOTE 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 °C \pm 2 °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 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 The test portion should be taken and weighed quickly, in order to avoid reabsorption of moisture.

7.4 Blank test and check test

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 6.2.

NOTE 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 both cases no significant changes in the analytical procedure will become necessary.

When 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.

When 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.

7.5 Determination Method 1 [vanadium content between 0,004 (m/m) and 0,06 (m/m)].

7.5.1 Decomposition of the test portion

Transfer the test portion (7.3) into a 100 ml PTFE beaker (5.1) and add 25 ml of hydrochloric acid (4.1). Cover and mix, and then digest just at the boiling point for 1 h. Add 0,25 ml of nitric acid (4.4), mix and digest for a further 10 min. Add 10 ml of hydrofluoric acid (4.3) and evaporate to dryness. Add 10 ml of hydrochloric acid (4.1) and 0,5 g of boric acid (4.8), and repeat the evaporation to dryness.

7.5.2 Dissolution of salts and residue treatment

Add 2 ml of hydrochloric acid (4.2), 4 ml of nitric acid (4.5) and 10 ml of water to the PTFE beaker. Add a stirring rod and heat to dissolve the salts. Filter through a close-texture filter paper into a 150 ml low-form beaker marked at the 50 ml capacity point, adding a further 10 ml of nitric acid (4.5) and 25 ml of water to the original PTFE beaker, while heating, to facilitate cleaning of the beaker and transfer of any residue to the paper. Wash the residue with water.

Transfer the residue to a platinum crucible (5.2), dry, ash and ignite the residue at 600 °C to 700 °C, then fuse over a burner or for 30 min in a muffle furnace (5.3) at 1000 °C in 0,3 g of sodium carbonate (4.7). Transfer the cooled crucible to the main solution using a stirring rod and leach the melt while evaporating the solution. Remove and rinse the crucible as soon as leaching is complete and continue the evaporation to a volume of 50 ml.

NOTE The leach of the fused residue in the main solution may attack the crucible if the evaporation is accidentally carried too far.