

Iron ores — Determination of vanadium

Part 2: Flame atomic absorption spectrometric methods

ICS 73.060.10

National foreword

This British Standard is the UK implementation of ISO 9683-2:2009.

The UK participation in its preparation was entrusted to Technical Committee ISE/58, Iron ores.

A list of organizations represented on this committee can be obtained on request to its secretary.

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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*



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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.

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

This first edition cancels and replaces ISO 9684:1991.

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

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

This corrected version of ISO 9683-2:2009 incorporates the following correction: a new sixth paragraph has been added to this foreword.

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 mass fraction of vanadium in iron ores.

Method 1 is applicable to mass fractions of vanadium between 0,004 % and 0,06 %, and Method 2 is applicable to mass fractions of vanadium between 0,06 % and 0,5 %, 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, *Laboratory glassware — Single-volume 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 % (by mass) or 1,19 g/ml, 48 % (by mass).

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 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 1-pentanol (4.12) and MIBK (4.13).