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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Steel and iron — Determination of vanadium content — N-BPHA spectrophotometric method

Aciers et fontes — Dosage du vanadium — Méthode spectrophotométrique au N-BPHA

Reference number
ISO 4942 : 1988 (E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4942 was prepared by Technical Committee ISO/TC 17, *Steel.*

Annexes A and B of this International Standard are for information only.

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Steel and iron — Determination of vanadium content — N-BPHA spectrophotometric method

1 Scope

This International Standard specifies an N-BPHA spectrophotometric method for the determination of vanadium in steel and iron.

The method is applicable to vanadium contents between 0,005 % (m/m) and 0,50 % (m/m).

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 listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 377 : 1985, *Wrought steel — Selection and preparation of samples and test pieces.*

ISO 385-1 : 1984, *Laboratory glassware — Burettes — Part 1 : General requirements.*

ISO 648 : 1977, *Laboratory glassware — One-mark pipettes.*

ISO 1042 : 1983, *Laboratory glassware — One-mark volumetric flasks.*

ISO 5725 : 1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

3 Principle

Dissolution of a test portion with appropriate acids.

Addition of orthophosphoric acid to an aliquot of the solution thus obtained to prevent the interference of iron, and addition of potassium permanganate to oxidize vanadium to the pentavalent state.

Selective reduction of excess permanganate by sodium nitrite in the presence of urea and treatment with N-BPHA and hydrochloric acid to form a complex, followed by extraction of the complex into trichloromethane.

Spectrophotometric measurement of the absorbance at approximately 535 nm.

4 Reagents

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

4.1 Hydrochloric acid, ϱ about 1,19 g/ml.

4.2 Hydrochloric acid, ϱ about 1,19 g/ml, diluted 4 + 1.

4.3 Nitric acid, ϱ about 1,40 g/ml.

4.4 Perchloric acid, ϱ about 1,67 g/ml.

4.5 Orthophosphoric acid, ϱ about 1,71 g/ml.

4.6 Orthophosphoric acid, ϱ about 1,71 g/ml, diluted 1 + 1.

4.7 Hydrochloric acid/nitric acid mixture.

Mix three volumes of hydrochloric acid (4.1) with one volume of nitric acid (4.3). Prepare fresh as needed.

4.8 Hydrogen peroxide, 300 g/l solution.

4.9 Sodium nitrite, 3 g/l solution.

4.10 Urea, 250 g/l solution.

4.11 Sodium tripolyphosphate ($Na_5P_3O_{10}$), 100 g/l solution.

4.12 Potassium permanganate, 3 g/l solution.

4.13 Trichloromethane (chloroform).

4.14 N-BPHA, 2,5 g/l solution in trichloromethane.

Dissolve 0,25 g of *N*-benzoylphenylhydroxylamine [$C_6H_5CON(OH)C_6H_5$] in 100 ml of trichloromethane (4.13). Prepare fresh, or store in a brown bottle.