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Steel — Determination of niobium content — PAR spectrophotometric method

Aciers — Dosage du niobium — Méthode spectrophotométrique au PAR

Reference number
ISO 9441 : 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 9441 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 — Determination of niobium content — PAR spectrophotometric method

1 Scope

This International Standard specifies a PAR spectrophotometric method for the determination of niobium in steel.

The method is applicable to all types of steel with niobium contents between 0,005 % (*m/m*) and 1,3 % (*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 in hydrochloric acid followed by oxidation with hydrogen peroxide.

Precipitation of niobium and tantalum with phenylarsonic acid, using zirconium as a carrier.

Formation of a complex of niobium with 4-(2-pyridyl-azo)-resorcinol (PAR) in a sodium tartrate medium buffered by sodium acetate solution adjusted to pH 6,3.

Spectrophotometric measurement of the coloured compound at a wavelength of about 550 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 Iron, of high purity, free from niobium.

4.2 Potassium hydrogen sulfate ($KHSO_4$).

4.3 Hydrochloric acid, ϱ approximately 1,19 g/ml.

4.4 Hydrochloric acid, ϱ approximately 1,19 g/ml, diluted 1 + 9.

4.5 Sulfuric acid, ϱ approximately 1,84 g/ml, diluted 1 + 1.

4.6 Sulfuric acid, ϱ approximately 1,84 g/ml, diluted 1 + 4.

4.7 Hydrogen peroxide, 300 g/l.

4.8 Sodium hydroxide, 120 g/l solution.

Store in a polyethylene bottle.

4.9 Zirconium nitrate, 3 g/l solution in hydrochloric acid medium.

Dissolve 0,3 g of zirconium nitrate in 50 ml of hydrochloric acid, ϱ approximately 1,19 g/ml, diluted 1 + 4. Filter through a fine filter paper, dilute to 100 ml with water and mix.

4.10 Sodium acetate buffer, pH-value 6,3.

Dissolve 350 g of sodium acetate trihydrate in 700 ml of water, add 5,5 ml of glacial acetic acid, ϱ approximately 1,05 g/ml, dilute to 1 000 ml and mix. Adjust the pH-value to 6,3 with small additions of acetic acid or sodium hydroxide solution (4.8), using a pH-meter for measurement.

4.11 Tartaric acid, 100 g/l solution.