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Steel — Determination of high cobalt content — Potentiometric titration method after separation by ion exchange

*Aciers — Dosage du cobalt en fortes teneurs — Méthode par titrage
potentiométrique après séparation par échange d'ions*

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ISO 11653:1997(E)**Foreword**

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International Standard ISO 11653 was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*.

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

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International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet central@iso.ch
X.400 c=ch; a=400net; p=iso; o=isocs; s=central

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Steel — Determination of high cobalt content — Potentiometric titration method after separation by ion exchange

1 Scope

This International Standard specifies a method for the determination of cobalt in steel using a potentiometric titration method after separation by ion exchange.

The method is applicable to a cobalt content of between 5,0 % (m/m) and 17,0 % (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 indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

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

ISO 1042:— 1), *Laboratory glassware — One-mark volumetric flasks.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions.*

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

ISO 5725-3:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method.*

ISO 14284 :1996, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition.*

3 Principle

Dissolution of a test portion in a mixture of nitric and hydrochloric acids.

Separation of cobalt from interfering elements by selective elution from an anion exchange column using hydrochloric acid.

1) To be published. (Revision of ISO 1042:1983)

Oxidation with perchloric acid. Removal of dissolved chlorine and oxygen with a stream of nitrogen.

Addition of the test solution to a solution containing ammonium citrate, ammonium hydroxide and an excess of potassium hexacyanoferrate (III) standard solution and titration of the excess with a standard cobalt solution using potentiometric end-point detection.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and grade 2 water as specified in ISO 3696.

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

4.2 Hydrochloric acid, ρ about 1,19 g/ml, diluted 7+5.

4.3 Hydrochloric acid, ρ about 1,19 g/ml, diluted 2+3.

4.4 Hydrochloric acid, ρ about 1,19 g/ml, diluted 1+2.

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

4.6 Nitric acid, ρ about 1,40 g/ml.

4.7 Perchloric acid, ρ about 1,67 g/ml (see note 1).

NOTE 1 Perchloric acid (ρ about 1,54 g/ml) may also be used. 100 ml of perchloric acid (ρ about 1,67 g/ml) is equivalent to 127 ml of perchloric acid (ρ about 1,54 g/ml).

4.8 Ammonium hydroxide, ρ about 0,89 g/ml.

4.9 Ammonium citrate, solution, corresponding to 200 g per litre. Dissolve 100 g of citric acid monohydrate ($\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$) in approximately 250 ml of water. Cautiously, and with constant stirring, add 170 ml of ammonium hydroxide (4.8). After cooling, dilute to 500 ml with water and mix.

4.10 Cobalt, standard solution, corresponding to 2,00 g of cobalt per litre.

Weigh, to the nearest 0,001 g, 2,000 g of cobalt metal [minimum purity 99,95 % (m/m)] (see note 2). Transfer to a 600 ml beaker and dissolve in 40 ml of nitric acid (ρ about 1,40, diluted 1+1). Heat to complete dissolution, boil gently to expel oxides of nitrogen, cool and transfer to a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix. Store in a polyethylene bottle.

1 ml of this solution contains 2,00 mg of Co.

NOTE 2 High purity cobalt powder should not be used because surface oxygen adversely affects accuracy. For example rod, shot or wire should be used.

4.11 Potassium hexacyanoferrate (III), standard volumetric solution.

4.11.1 Preparation of the standard volumetric solution.

Dissolve 5,6 g of potassium hexacyanoferrate(III) [$\text{K}_3\text{Fe}(\text{CN})_6$] in 250 ml of water. Filter the solution through a pulp pad and wash thoroughly with water. Transfer to a 500 ml one-mark volumetric flask, dilute to the mark with water and mix. Refilter and standardize this solution as specified in 4.11.2 immediately prior to use.