
**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*

iTeh Standards
(<https://standards.iteh.ai>)
Document Preview

[ISO 11653:1997](#)

<https://standards.iteh.ai/catalog/standards/iso/ebc06049-e022-452d-9cc8-95454e22cc5a/iso-11653-1997>



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 voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

iTeh Standards (<https://standards.itih.ai>) Document Preview

[ISO 11653:1997](#)

<https://standards.itih.ai/catalog/standards/iso/ebc06049-e022-452d-9cc8-95454e22cc5a/iso-11653-1997>

© ISO 1997

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from the publisher.

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

Printed in Switzerland

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 ($C_6H_8O_7 \cdot H_2O$) 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) [$K_3Fe(CN)_6$] 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.

4.11.2 Standardization of the standard volumetric solution.

Transfer two portions of 20,0 ml of the cobalt standard solution (4.10) to two beakers. Treat the solution according to 7.2.3 and 7.2.4.

Calculate the standardization factor T for each sample according to the equation

$$T = \frac{V_1 + V_2}{V_3}$$

where

- V_1 is the volume, in millilitres, of the cobalt standard solution (4.10) taken in 4.11.2 (= 20,0 ml);
- V_2 is the volume, in millilitres, of the cobalt standard solution (4.10) used in the factor titration ;
- V_3 is the volume, in millilitres, of the potassium hexacyanoferrate (III) solution used in the titration ;
- T is the required factor, which will be less than 1 if the solution is below theoretical strength etc. Calculate the average of the two determined T values.

4.12 Ion exchange resin

Use an anion exchange resin of the alkyl quaternary ammonium type (chloride form) consisting of spherical beads having a nominal crosslinkage of 8 %, and 200 to 400 nominal mesh size. To remove those beads greater than about 180 μm in diameter, as well as the excessively fine beads, treat the resin as follows.

Transfer a supply of the resin to a beaker, cover with water, and allow sufficient time (at least 30 min) for the beads to undergo maximum swelling. Place a 180 μm sieve, 150 mm in diameter, over a 2 litre beaker. Prepare a thin slurry of the resin and pour it onto the sieve. Wash the fine beads through the sieve using a small stream of water. Discard the beads retained on the sieve periodically, if necessary, to avoid undue clogging of the openings. When the bulk of the collected resin has settled, decant the water and transfer approximately 100 ml of resin to a 400 ml beaker. Add 200 ml of hydrochloric acid (4.5), stir vigorously, allow the resin to settle for 4 min to 6 min, decant 150 ml to 175 ml of the suspension, and discard it. Repeat the treatment with hydrochloric acid (4.5) twice more and reserve the coarser resin for the column preparation.

Prepare the column as follows.

Place a 10 mm to 30 mm layer of glass wool or poly (vinyl chloride) plastic fibre in the bottom of the column, and add a sufficient amount of the prepared resin to fill the column to a height of approximately 140 mm. Place a 20 mm layer of glass wool or poly (vinyl chloride) plastic fibre at the top of the resin bed to protect it from being carried into suspension when the solutions are added. While passing a minimum of 35 ml of hydrochloric acid (4.2) through the column, with the hydrostatic head 100 mm above the top of the resin bed, adjust the flow rate to not more than 3,0 ml per min. Drain to 10 mm to 20 mm above the top of the resin bed and then close the lower stopcock.

5 Apparatus

During the analysis, unless otherwise stated, use only ordinary laboratory apparatus.

All volumetric glassware shall be class A, in accordance with ISO 385-1, ISO 648 or ISO 1042 as appropriate.

5.1 Potentiometric titration apparatus

5.1.1 Indicator electrode, of bright platinum, which shall be kept in a clean, highly polished condition. It shall be cleaned by dipping in the nitric acid (4.6) and rinsed with water prior to use.

5.1.2 Reference electrode, of silver/silver chloride, calomel or mercury(I) sulfate. The manufacturer's instructions on the care and maintenance of these electrodes shall be followed.

5.1.3 Titration assembly, consisting of a 400 ml beaker, two 50 ml burettes complying with the requirements of ISO 385-1, class A, and a magnetic stirrer.

5.1.4 High-impedance electronic voltmeter: Normally a pH-meter can be used as a voltmeter. Commercial automatic titrators or potentiographs have an advantage over manual systems in that the titration curve is plotted and the end-point can be evaluated by interpolation of the curve rather than by calculation from the first or second derivative (see clause 9).

5.2 Ion exchange column

Approximately 25 mm in diameter and 300 mm long, tapered at one end, and provided with a stopcock to control the flow rate, and a second, lower stopcock to stop the flow. A reservoir for the eluants may be added at the top of the column.

6 Sampling

Carry out sampling in accordance with ISO 14284.

7 Procedure

WARNING – Fuming perchloric acid is a powerful oxidant and can cause an explosive mixture when in contact with ammonia, nitrous fumes or organic matter. All evaporation must be carried out in fume cupboards suitable for use with perchloric acid.

7.1 Test portion

Weigh, to the nearest 0,000 1 g, 0.5 g of the test sample.

7.2 Determination

7.2.1 Preparation of the test solution

Place the test portion (7.1) in a 150 ml beaker. Add 20 ml of a mixture of five parts of hydrochloric acid (4.1) and one part of nitric acid (4.6) (see note 3). Cover the beaker and digest at 60 °C to 70 °C until the test portion is decomposed. Rinse and remove the cover. Place a ribbed cover glass on the beaker, and evaporate the solution nearly to dryness, but do not bake. Cool, add 20 ml of hydrochloric acid (4.2), and digest at 60 °C to 70 °C until the salts are dissolved (approximately 10 min) (see note 4).

NOTES

3 Other ratios and concentrations of acids, with or without the addition of hydrofluoric acid, are used for the decomposition of special grades of alloys.

4 Some tungsten and/or molybdenum oxides can remain undissolved. They are eliminated later by filtration.

7.2.2 Separation by ion exchange resin

Cool to room temperature and filter the solution through a close-texture filter paper and wash thoroughly with water. Transfer the solution to the ion exchange column. Place a beaker under the column and open the lower stopcock. When the solution reaches a level 10 mm to 20 mm above the resin bed, rinse the original beaker with 5 ml to 6 ml of hydrochloric acid (4.2) and transfer the rinsings to the column. Repeat this at 2 min intervals until the beaker has been rinsed four times. Wash the upper part of the column with hydrochloric acid (4.2) 2 or 3 times and allow the level to drop to 10 mm to 20 mm above the resin bed each time. Maintain the flow rate at not more than 3,0 ml/min and add hydrochloric acid (4.2) to the column until a total of 175 ml to 185 ml of solution (test solution and