INTERNATIONAL STANDARD

ISO 11877

First edition
2008-06-01

Hardmetals — Determination of silicon in cobalt metal powders — Photometric method

Métaux durs — Dosage du silicium dans les poudres métalliques de cobalt — Méthode photométrique

Reference number
ISO 11877:2008(E)

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

STANDARDSISO.COM : Click to view the full PDF of ISO 11877:2008

COPYRIGHT PROTECTED DOCUMENT

© ISO 2008

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 either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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 11877 was prepared by Technical Committee ISO/TC 119, *Powder metallurgy*, Subcommittee SC 4, *Sampling and testing methods for hardmetals*.

Hardmetals — Determination of silicon in cobalt metal powders — Photometric method

1 Scope

This International Standard specifies a photometric method to be used for the determination of the mass fraction of silicon in the range of 20 µg/g to 300 µg/g in cobalt metal powders.

2 Principle

After treatment of the sample with a mixture of acids, the so-formed silicon tetrafluoride is distilled into a mixture of boric acid and sodium molybdate. The obtained molybdatesilicate complex is extracted with methyl-isobutylketone and the absorbance of the solution is measured.

3 Reagents

Reagents of the highest purity and only double-distilled water or water of equivalent purity shall be used.

3.1 Hydrofluoric acid, 40 % by mass.

3.2 Nitric acid, 65 % by mass.

3.3 Hydrochloric acid, 30 % by mass.

3.4 Sulfuric acid, 96 % by mass.

3.5 Perchloric acid, 70 % by mass.

3.6 Sodium hydroxide solution, 30 % by mass.

3.7 Boric acid.

3.8 Sodium molybdate.

3.9 Amidosulfuric acid.

3.10 Methyl-isobutylketone.

3.11 Acetone.

3.12 Water.

3.13 Nitrogen.

3.14 Silicon standard solution, 100 µg/ml.

4 Apparatus and equipment

All apparatus and equipment in contact with reagents shall be made of silicon-free material like PTFE (polytetrafluoroethylene), PP (polypropylene), PE (polyethylene) and TPX (methylpenten-polymer).

4.1 Apparatus for acid treatment and distillation, for separation of silicon tetrafluoride.

4.2 Test-tube shaker.

4.3 Refrigerator.

4.4 Platinum thimbles, for weighing the sample.

4.5 Graduated flasks, stock bottles and pipettes.

5 Preparation of acid mixture for sample treatment

Mix 30 ml of hydrofluoric acid, 20 ml of nitric acid, 20 ml of perchloric acid and 30 ml of water in a PTFE flask.

6 Preparation of solutions for receiver

6.1 Solution I

Dissolve 37,2 g of boric acid and 14,4 g of sodium molybdate in water in a graduated PP flask and fill up to 1 000 ml with water.

6.2 Solution II

Dissolve 19,4 g of amidosulforic acid in a graduated PP flask and fill up to 100 ml with water.

7 Procedure

7.1 Pour 30 ml of solution I and 1 ml of solution II into a graduated 100 ml flask.

7.2 Pour 3 ml of the acid mixture into the distilling flask, and add 100 mg of the sample weighed to a platinum thimble.

7.3 Connect the receiver and distilling flask and start the nitrogen flow.

7.4 Heat the apparatus to 170 ± 10 °C, within approximately 25 min, and keep it at this temperature for 50 min. Control the purge gas during distillation.

7.5 When the distillation time is over, remove the receiver flask and rinse the inlet tube with water.

7.6 Adjust the pH of the receiver solution to 1,3 to 1,4 with sodium hydroxide (approximately 800 µl to 1 000 µl) and leave it for 1 h.

7.7 Add 2,0 ml of hydrochloric acid and 20 ml of methyl-isobutylketone and shake for 1 min. Fill to the stopcock with water and close the flask. Keep the flask at + 5 °C for 1 h.

8 Photometric determination

Transfer the methyl-isobutylketone phase to an appropriate photometer and measure against methyl-isobutylketone.

Use a wavelength of 345 µm.

9 Preparation of blank and calibration solutions

9.1 Blank solution

Determine the absorbance of a solution containing all the reagents except the sample material.

9.2 Calibration solution

Treat a silicon standard sample as outlined in 9.1 and prepare at least three solutions containing silicon in the range of the sample for a calibration curve.

10 Expression of the result

10.1 Calculation

After calculating E = EX − EB (where E is the extinction, EX is the experimentally measured extinction and EB is the blind value of the extinction), the mass fraction of silicon w is evaluated graphically or arithmetically using the calibration curve, taking into account the sample mass.

10.2 Final result

Report the arithmetical mean of the acceptable determination rounded to 0,001 % by mass.

11 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for identification of the test sample;

c) the test results obtained;

d) all operations not specified in this International Standard, or regarded as optional;

e) details of any occurrence which may have affected the result.

Bibliography

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

[2] ISO 5725-2, *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*

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

[4] ISO 5725-4, *Accuracy (trueness and precision) of measurement methods and results — Part 4: Basic methods for the determination of the trueness of a standard measurement method*

[5] ISO 5725-5, *Accuracy (trueness and precision) of measurement methods and results — Part 5: Alternative methods for the determination of the precision of a standard measurement method*

[6] ISO 5725-6, *Accuracy (trueness and precision) of measurement methods and results — Part 6: Use in practice of accuracy values*