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

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



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Contents

Page

Foreword.....	iv
1 Scope	1
2 Principle	1
3 Reagents	1
4 Apparatus and equipment	2
5 Preparation of acid mixture for sample treatment	2
6 Preparation of solutions for receiver	2
7 Procedure	2
8 Photometric determination	3
9 Preparation of blank and calibration solutions	3
10 Expression of the result	3
11 Test report	3
Bibliography	4

Foreword

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

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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 amidosulfuric 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*
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