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**Hardmetals — Determination of silicon in
cobalt metal powders using graphite-
furnace atomic absorption**

*Métaux durs — Dosage du silicium dans les poudres métalliques de
cobalt par absorption atomique à four graphite*

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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.

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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 17352 was prepared by Technical Committee ISO/TC 119, *Powder metallurgy*, Subcommittee SC 4, *Sampling and testing methods for hardmetals*.

In this corrected version of ISO 17352:2008, the edition number on the cover page has been corrected from "Second" to "First".

Hardmetals — Determination of silicon in cobalt metal powders using graphite-furnace atomic absorption

1 Scope

This International Standard specifies a test method, using graphite-furnace atomic absorption, to determine the mass fraction of silicon in cobalt metal powder with varying compositions within the range of 5 µg/g to 40 µg/g.

2 Principle

This test method for the chemical analysis of cobalt metal powder is to be used to determine traces of silicon. It is assumed that all those who use this test method will be trained analysts capable of performing common laboratory procedures skilfully and safely. It is expected that all the work will be performed in a properly equipped laboratory.

3 Reagents

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

- 3.1 **Nitric acid**, $\rho = 1,4$ g/ml.
- 3.2 **Hydrochloric acid**, $\rho = 1,15$ g/ml.
- 3.3 **Cobalt powder**, purity > 99,9 % by mass.
- 3.4 **Si solution**, 1,000 g/l.

4 Apparatus

4.1 Graphite-furnace atomic absorption spectrometer

Follow the manufacturer's instruction manual for installation and operation.

4.2 Additional information

The following parameters are for information purposes only and have to be adapted to the special requirements of every laboratory.

Lamp:	hollow cathode lamp for Si
Tube:	coated pyrolytic C-tube without a platform
Wavelength:	251,6 nm
Slit:	0,2 nm
Injection volume:	20 µl
Temperature program:	drying — 120 °C ashing — 1 200 °C atomising — 2 700 °C conditioning — 2 750 °C

5 Procedure

5.1 Test portion

Weigh, to the nearest 0,001 g, approximately 0,5 g of the test sample and transfer it to a 100 ml polypropylene flask.

5.2 Dissolution of the test portion

The test sample is dissolved in 10 ml of nitric acid and 2 ml of hydrochloric acid. When the sample is completely dissolved, allow the flask to cool and then fill up to 100 ml with double-distilled water.

6 Calibration

0,500 g of ultrapure cobalt powder is dissolved in the same way as the test sample (see 5.2). The resulting solutions are spiked with 0 µg of Si, 5 µg of Si, 10 µg of Si, 20 µg of Si and 40 µg of Si and filled up to 100 ml with water. With these calibrated solutions, a calibration curve is prepared.

7 Calculation

Calculate the mass fraction of silicon in the sample using the calibration curve prepared in Clause 6. Compensate for the actual weight of the sample analysed.

8 Precision

No definite statement on the precision and bias can be made at this time. These values shall be calculated using the results of a round-robin analysis.

9 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.

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Bibliography

- [1] ISO 3252, *Powder metallurgy — Vocabulary*
- [2] ASTM B 243, *Standard Terminology of Powder Metallurgy*

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