INTERNATIONAL STANDARD

ISO 11876

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Hardmetals — Determination of calcium, copper, iron, potassium, magnesium, manganese, sodium, nickel and zinc in cobalt metal powders — Flame atomic absorption spectrometric method

Métaux-durs — Dosage du calcium, du cuivre, du fer, du potassium, du magnésium, du manganèse, du sodium, du nickel et du zinc dans les poudres métalliques de cobalt — Méthode par spectrométrie d'absorption atomique dans la flamme

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Foreword

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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 11876 was prepared by Technical Committee ISO/TC 119, *Powder metallurgy*, Subcommittee SC 4, *Sampling and testing methods for hardmetals*.

Hardmetals — Determination of calcium, copper, iron, potassium, magnesium, manganese, sodium, nickel and zinc in cobalt metal powders — Flame atomic absorption spectrometric method

1 Scope

This International Standard specifies a flame atomic absorption spectrometric method to be used for the determination of the mass fractions of copper, potassium, magnesium, manganese, sodium and zinc in cobalt metal powders in the range of 0,001 % to 0,01 %, calcium in the range of 0,002 % to 0,01 %, and iron and nickel in the range of 0,002 % to 0,05 %.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3534-1:2006, *Statistics — Vocabulary and symbols — Part 1: General statistical terms and terms used in probability*

3 Reagents

Use only reagents of the highest purity and only double-distilled water or water of equivalent purity.

3.1 Nitric acid, $\rho = 1{,}42$ g/ml, diluted 1 + 1.

3.2 Hydrochloric acid, $\rho \approx 1{,}1$ g/ml (hydrochloric acid, $\rho = 1{,}19$ g/ml, diluted 1 + 1).

3.3 Cesium chloride solution, (20 g/1 000 ml).

3.4 Standard solutions, (1,000 g/1 000 ml) for calibration purposes for each element to be determined.

4 Procedure

4.1 Test portion

Weigh, to the nearest 0,001 g, approximately 1 g of the sample.

4.2 Dissolution of the test portion

Add 20 ml of nitric acid (3.1) and 10 ml of hydrochloric acid (3.2) and heat gently until the test portion is completely dissolved.

4.3 Dilution volume

4.3.1 Add 5 ml of the cesium chloride solution (3.3) and dilute to 100 ml.

4.3.2 Shake the analysed volume with 20 ml of freshwater and filter using appropriate filter media over a plug of cotton wool into a 50 ml or 100 ml volumetric flask. Depending on the concentration of the determined elements in the sample and/or the sensitivity of the photometer, the dilution volume may be either 50 ml or 100 ml in volumetric flask.

4.4 Preparation of calibration and blank solutions

4.4.1 Prepare at least three solutions according to 4.2 with a matrix as similar as possible to the test portion to be analysed but without making up to volume. Then add increasing volumes of property diluted standard solutions of the elements to be determined according to the concentration ranges to be covered. Make up to 100 ml and mix.

4.4.2 Prepare at least two blank solutions (see 4.4.1) without the addition of the relevant element to be determined.

4.5 Adjustment of the atomic absorption spectrometer

SAFETY PRECAUTIONS — Follow the manufacturer's recommendation on igniting and extinguishing the flame.

Optimize the response of the instrument at the wavelength given for the element to be determined (see Table 1). Preheat the burner for about 5 min and adjust the fuel and correct the burner to obtain maximum absorption while aspirating a calibration solution.

Make sure that the absorbance reading is not drifting. Aspirate water and set the initial reading to zero absorbance.

Table 1 — Parameters for atomic flame absorption

Element	Wavelength nm	Oxidant/fuel
Ca	422,7	N_2O/acetylene
Cu	324,8	air/acetylene
Fe	248,3	air/acetylene
K	766,5	air/acetylene
Mg	285,2	N_2O/acetylene
Mn	279,5	air/acetylene
Na	589,0	air/acetylene
Ni	232,0	air/acetylene
Zn	213,9	air/acetylene
NOTE Use slit recommended by the manufacturer.		

4.6 Atomic absorbance measurement

4.6.1 Aspirate first the blank solution and then the calibration and test solutions consecutively and record the reading. Aspirate water between each solution. Make at least two measurements for each solution. Solids which build up at the burner slit shall be removed; otherwise they cause a decrease in sensitivity.

4.6.2 Prepare a calibration curve by plotting the obtained absorbance values of the calibration solutions corrected for the blank against the concentration.

4.6.3 Convert the absorbance values of the test solution corrected for the blank to milligrams of the element per litre by means of the calibration curve.

5 Expression of results

5.1 Calculation

The mass fraction of the element, expressed as a percentage, is given by the following formula:

$$\frac{c \cdot V}{10\,000 \cdot m}$$

where

c is the concentration, in milligrams per litre (mg/l), of the element in the test solution;

V is the dilution volume, in millilitres (ml);

m is the mass of the test portion, in grams (g).

5.2 Permissible tolerances

The deviations between three independent determinations shall not exceed the values in Table 2.

Table 2 — Lower and upper limits of mass fraction of the element as a percentage

Element	Lower limit	Upper limit
Calcium	0,000 5 % ± 50 % (CV)	0,010 0 % ± 20 % (CV)
Copper	0,000 5 % ± 50 % (CV)	0,010 0 % ± 20 % (CV)
Iron	0,000 5 % ± 50 % (CV)	0,015 0 % ± 20 % (CV)
Potassium	0,000 5 % ± 50 % (CV)	0,010 0 % ± 20 % (CV)
Magnesium	0,000 5 % ± 50 % (CV)	0,010 0 % ± 20 % (CV)
Sodium	0,000 5 % ± 50 % (CV)	0,005 0 % ± 20 % (CV)
Nickel	0,000 5 % ± 50 % (CV)	0,020 0 % ± 20 % (CV)
Zinc	0,000 5 % ± 100 % (CV)	0,010 0 % ± 10 % (CV)
CV: coefficient of variation (see ISO 3534-1:2006, 2.38)		

5.3 Final result

Report the arithmetic mean of acceptable determinations, rounded to a mass fraction of 0,001 % and a mass fraction of 0,002 %.

6 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 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] SO 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*