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International Standard ISO 7627/2

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Hardmetals — Chemical analysis by flame atomic absorption spectrometry — Part 2: Determination of calcium, potassium, magnesium and sodium in contents from 0,001 to 0,02 % (m/m)

Métaux-durs — Analyse chimique par spectrométrie d'absorption atomique dans la flamme — Partie 2: Dosage du calcium, du potassium, du magnésium et du sodium à des teneurs comprises entre 0,001 *et* 0,02 % (m/m)

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 7627/2 was developed by Technical Committee ISO/TC 119, *Powder metallurgy*, and was circulated to the member bodies in August 1982.

It has been approved by the member bodies of the following countries:

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No member body expressed disapproval of the document.

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Hardmetals — Chemical analysis by flame atomic absorption spectrometry — Part 2: Determination of calcium, potassium, magnesium and sodium in contents from 0,001 to 0,02 % (m/m)

1 Scope and field of application

This part of ISO 7627 specifies the method to be used for the determination of calcium, potassium, magnesium and sodium contents of hardmetals within the range 0,001 to 0,02 % (m/m) by flame atomic absorption spectrometry.

General requirements concerning the field of application, principle, interfering elements, apparatus, sampling and test report are given in ISO 7627/1.

NOTE — In low concentrations, the determination of these elements is very critical. Every care should be taken to avoid contamination from atmosphere and reagents.

2 Reference

ISO 7627/1, *Hardmetals — Chemical analysis by flame atomic absorption spectrometry — Part 1: General requirements.*

3 Reagents

If necessary, reagents of the highest purity shall be used.

3.1 Hydrofluoric acid, ϱ 1,12 g/ml.

3.2 Nitric acid, ϱ 1,42 g/ml.

3.3 Ammonium fluoride, 0,1 g/ml solution.

3.4 Caesium chloride, 0,01 g/ml solution.

3.5 High-purity stock solutions, for calibration purposes for each element to be determined, containing 1,000 g of the element per litre.

NOTE — This value is understood to establish a maximum limit of 1,000 5 g and a minimum limit of 0,999 5 g.

4 Procedure

4.1 Test portion

Weigh, to the nearest 0,001 g, approximately 1 g of the test sample. Transfer it to a 100 ml polytetrafluorethylene beaker or a beaker of other suitable material. Cover the beaker.

4.2 Dissolution of the test portion

Add 10 ml of water, 5 ml of the hydrofluoric acid (3.1), and then 5 ml of the nitric acid (3.2), drop by drop, to the beaker containing the test portion (4.1) and heat gently until the test portion is completely dissolved. Add 10 ml of the caesium chloride solution (3.4) and 10 ml of the ammonium fluoride solution (3.3). Then transfer the solution totally to a 100 ml polypropylene one-mark volumetric flask and dilute to the mark.

4.3 Dilution volume

Prepare the relevant dilution volume for the analysis according to the table as follows:

4.3.1 Dilution volume 100 ml: use the solution in 4.2.

4.3.2 The concentration of the solution may be reduced by a factor of 10 for instruments of higher sensitivity by transferring 10 ml of the solution in 4.2 to a 100 ml polypropylene one-mark volumetric flask. Add 10 ml of the caesium chloride solution (3.4). Add 10 ml of the ammonium fluoride solution (3.3) and dilute to the mark.

4.4 Preparation of calibration and blank solutions

4.4.1 Prepare at least six solutions according to 4.2 with a matrix composition as similar as possible to the test portion to be analysed, but without making up to volume. Then add increasing volumes of properly diluted stock 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 Also prepare calibration solutions with a diluted matrix in accordance with 4.3 for dilution volumes of 1 000 ml, if necessary.

4.4.3 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 PRECAUTION : Follow the manufacturer's recommendation on igniting and extinguishing the flame.

Optimize the response of the instrument at the wavelength given for the element being determined. See the table.

Preheat the burner for about 5 min and then 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.

4.6 Atomic absorbance measurements

4.6.1 Aspirate first the blank solution and then the calibration and test solutions consecutively and record the readings. Aspirate water between each solution. Make at least two measurements for each solution. Solids which build up on the burner slit must be removed, otherwise they will cause a decrease of 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, in milligrams per litre, of the element.

4.6.3 Convert the absorbance values of the test solutions 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 element content, expressed as a percentage by mass, is given by the formula

$$\frac{c \times V}{10^4 \times m}$$

where

c is the concentration, in milligrams per litre, of the element in the test solution;

V is the dilution volume, in millilitres;

m is the mass, in grams, of the test portion.

5.2 Permissible tolerances

The deviations between three independent determinations shall not exceed 0,000 5 % (absolute value) at the lower limit of determination. It shall not exceed 0,002 % at the 0,02 % level.

5.3 Final result

Report the arithmetical mean of acceptable determinations rounded to 0,001 % (m/m). If the element content is below 0,001 % (m/m) or greater than 0,02 % (m/m), report the result as less than 0,001 % (m/m) or greater than 0,02 % (m/m) respectively.

Table — Instrumental parameters and characteristics of calibration functions

Element	Dilution volume (V) for 1 g test portion[1] ml	Oxidant	Wavelength nm	Reciprocal sensitivity, for 1 % absorption[1] µg/ml	Linear range[1] %	Notes
Ca	100	N_2O	422,7	0,03	0,001 to 0,02	1
K	100	air	769,9	0,04	0,000 5 to 0,02	
Mg	100	N_2O	285,2	0,02	0,000 3 to 0,04	
Na	100	air	589,3	0,02	0,000 5 to 0,02	2

1) Guidelines for information only.

NOTES

1 Sensitivity is greatly dependent on matrix composition.

2 Use the doublet.