SLOVENSKI STANDARD
SIST ISO 7523:1997

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Nikelj - Določanje srebra, arzena, bizmuta, kadmija, svinca, antimona, selena, kositra, telurja in talija - Elektrotermična atomska absorpcijska spektrometrična metoda

Nickel -- Determination of silver, arsenic, bismuth, cadmium, lead, antimony, selenium, tin, tellurium and thallium contents -- Electrothermal atomic absorption spectrometric method

Nickel -- Dosage de l'argent, de l'arsenic, du bismuth, du cadmium, du plomb, de l'antimoine, du sélénium, de l'étain, du tellure et du thallium -- Méthode par spectrométrie d'absorption atomique électrothermique

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International Standard 7523

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Nickel — Determination of silver, arsenic, bismuth, cadmium, lead, antimony, selenium, tin, tellurium and thallium contents — Electrothermal atomic absorption spectrometric method

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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.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 7523 was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Printed in Switzerland

Nickel — Determination of silver, arsenic, bismuth, cadmium, lead, antimony, selenium, tin, tellurium and thallium contents — Electrothermal atomic absorption spectrometric method

1 Scope and field of application

This International Standard specifies an electrothermal atomic absorption spectrometric method for the determination of the silver, arsenic, bismuth, cadmium, lead, antimony, selenium, tin, tellurium and thallium contents of high purity, refined, wrought and cast nickel within the ranges specified in table 1. This method is applicable to the independent determination of any one or more of the elements listed without including all elements specified in the standard solutions.

Table 1 — Concentration ranges of elements to be determined

Element	Concentration range* (µg/g)**
Ag	0,1 to 10
As	1 to 20
Bi	0,5 to 15
Cd	0,1 to 2
Pb	0,1 to 10
Sb	1 to 10
Se	1 to 10
Sn	1 to 5
Te	0,2 to 10
Tl	0,5 to 10

* For specific compositions, see ISO 6283.

** 1 µg/g = 1 g/t

For potential interferences and precautions, see clause 9.

2 References

ISO 385/1, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 648, *Laboratory glassware — One-mark pipettes.*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks.*

ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests.*

3 Principle

Dissolution of a test portion in nitric acid and dilution of the solution to a known volume.

Introduction of a known volume of the solution into an electrothermal atomizer of an atomic absorption spectrometer.

Measurement of the absorption of the resonance line energy from the spectrum of each element and comparison with that of calibration solutions of the same element in a matched nickel matrix using background correction.

4 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only deionized water or water of equivalent purity.

4.1 Nitric acid, ϱ_{20} = 1,41 g/ml.

The same batch of nitric acid shall be used throughout the procedure.

NOTE — It may be necessary to redistil the nitric acid if high blanks are obtained.

4.2 Nitric acid, ϱ_{20} = 1,41 g/ml, diluted 1 + 19.

The same batch of nitric acid shall be used throughout the procedure.

4.3 Mixed analyte standard solutions.

4.3.1 Analyte stock standard solutions, corresponding to 1,000 g of Ag, As, Bi, Cd, Pb, Sb, Se, Sn, Te and Tl per litre.

Prepare separately for each metal of interest.

4.3.1.1 Silver, arsenic, bismuth, cadmium, lead, selenium and tellurium.

Weigh, to the nearest 0,000 1 g, 0,100 g of the high purity [99,9 % (*m*/*m*), minimum] elements, transfer to a 100 ml beaker and dissolve in 10 ml of nitric acid diluted 1 + 1. Heat to complete dissolution, boil gently to expel oxides of nitrogen, cool and transfer to a one-mark 100 ml volumetric flask containing 10 ml of nitric acid diluted 1 + 1. Make up to the mark with water and mix.

Store in a glass bottle.

4.3.1.2 Antimony

Weigh exactly 0,274 g of potassium antimonyl tartrate hemihydrate [$K(SbO)C_4H_4O_6 \cdot 1/2H_2O$], transfer to a 100 ml one-mark volumetric flask, dissolve in water and dilute to the mark.

Prepare this solution freshly as mould forms in old solutions. The more dilute acidified solutions are stable.

4.3.1.3 Thallium

Weigh exactly 0,112 g of thallium(III) oxide (Tl_2O_3), transfer to a 100 ml beaker and dissolve in 10 ml hot nitric acid (4.1). Transfer to 100 ml one-mark volumetric flask, make up to the mark with water and mix.

Store in a glass bottle.

4.3.1.4 Tin

Weigh, to the nearest 0,001 g, 0,250 g of high purity [99,9 % (*m*/*m*), minimum] tin metal, transfer to a 100 ml plastic beaker and dissolve in 7,5 ml of a mixture of equal parts by volume of 48 % (*m*/*m*) hydrofluoric acid (ϱ_{20} = 1,13 g/ml), nitric acid (4.1) and water. Heat to complete dissolution, boil gently to expel oxides of nitrogen, cool and transfer to a 250 ml plastic one-mark volumetric flask. Make up to the mark with water and mix.

Store in a plastic bottle.

4.3.2 Mixed analyte standard solution A, corresponding to 1,0 mg of As, Bi, Pb, Sb, Se, Sn, Te and Tl per litre.

4.3.2.1 Pipette 10,0 ml of each of the 1,000 g/l stock standard solutions (4.3.1.1) of As, Bi, Pb, Sb, Se, Sn, Te and Tl into a 1 000 ml one-mark volumetric flask containing 100 ml of nitric acid diluted 1 + 1. Make up to the mark with water and mix.

Store in a glass bottle (5.6).

4.3.2.2 Pipette 10,0 ml of this solution (4.3.2.1) into a 100 ml one-mark volumetric flask containing 10 ml of nitric acid diluted 1 + 1. Make up to the mark with water and mix.

Store in a glass bottle (5.6).

4.3.3 Mixed analyte standard solution B, corresponding to 0,1 mg of Ag and Cd per litre.

4.3.3.1 Pipette 10,0 ml of each of the 1,000 g/l stock standard solutions (4.3.1.1) of silver and cadmium into a 1 000 ml one-mark volumetric flask containing 100 ml of nitric acid diluted 1 + 1. Make up to the mark with water and mix.

Store in a glass bottle (5.6).

4.3.3.2 Pipette 10,0 ml of this solution (4.3.3.1) into a 1 000 ml one-mark volumetric flask containing 100 ml of nitric acid diluted 1 + 1. Make up to the mark with water and mix.

This solution is not stable and should be prepared immediately before use.

4.4 Nickel nitrate, stock solution, containing 40 g of Ni per litre.

4.4.1 Weigh, to the nearest 0,001 g, 4,00 g of high purity (containing less than 5 g of Fe per tonne and less than 1 g of silver, arsenic, bismuth, cadmium, lead, antimony, selenium, tin, tellurium and thallium per tonne) nickel metal and transfer to a 400 ml beaker. Add 50 ml of water and 28 ml of nitric acid (4.1). Allow to stand and do not stir until most of the metal is dissolved as the reaction is vigorous. Heat to complete dissolution, boil gently to expel oxides of nitrogen and cool.

4.4.2 Filter through a dense filter paper which has been prewashed with nitric acid diluted 1 + 1. Return the filtrate through the filter paper again to collect fine carbon particles which tend to be present in the first filtrate. Collect the second filtrate in a 100 ml one-mark volumetric flask, wash the filter with water. Make up to the mark with water and mix. The solution contains approximately 0,8 mol of free HNO_3 per litre [5 % (*V*/*V*)].

NOTE — For details concerning an alternative method for the preparation and purification of the solution, see annex A.

5 Apparatus

Ordinary laboratory apparatus, and

5.1 Atomic absorption spectrometer and **electrothermal atomizer.**

5.1.1 The instrument shall be equipped with a background corrector and a high-speed recorder or computerized read out.

5.1.2 The instrument should be capable of using single element hollow cathode or electrodeless discharge lamps operated at currents recommended by the lamp and instrument manufacturer.

5.2 Burettes, of capacities 5 ml and 10 ml, graduated in divisions of 0,01 ml, in accordance with ISO 385/1, class A.

5.3 Pipettes, of capacities 10 and 25 ml, in accordance with ISO 648, class A.

5.4 Volumetric flasks, of capacities 10; 100; 200; 250; and 1 000 ml, in accordance with ISO 1042, class A.

5.5 Micropipettes, of capacities 5 to 25 µl.

5.6 Glass storage bottles.

The glass bottles used to store mixed analyte standard solutions shall be thoroughly cleaned, soaked several days in nitric acid (4.2), and rinsed thoroughly with water.

6 Sampling and samples

6.1 Sampling and preparation of the laboratory sample shall be carried out by normal agreed procedures or, in case of dispute, by the relevant International Standard.

6.2 The laboratory sample normally is in the form of a powder, granules, millings or drillings and no further preparation of the sample is necessary.

6.3 If it is suspected that the laboratory sample is contaminated with oil or grease from the milling or drilling process, it shall be cleaned by washing with high purity acetone and drying in air.

6.4 If the laboratory sample contains particles or pieces of widely varying sizes, the test portion should be obtained by riffling.

7 Procedure

7.1 Preparation of test solution

Weigh, to the nearest 0,01 g, 0,9 to 1,1 g of the laboratory sample and transfer to a clean unetched 100 ml beaker. Add 30 ml of water and 12 ml of nitric acid (4.1) and allow to dissolve. Heat to complete dissolution, boil gently to expel oxides of nitrogen, cool and transfer to a 100 ml one-mark volumetric flask. Make up to the mark with water and mix.

NOTE — The life of the graphite furnace tubes can be extended by using 5 ml of nitric acid rather than 12 ml.

7.2 Blank test

The zero members of the sets of calibration solutions (7.3) serve as blank tests since the same batch of nitric acid is used for dissolution of both the nickel reference and test portions.

If it is impossible to use the same batch of nitric acid, a second blank test shall be prepared using the same high purity nickel metal. This blank is then compared to the zero member and an appropriate correction made, if significant.

7.3 Preparation of calibration solutions

7.3.1 Set A

7.3.1.1 This set corresponds to 0; 0,005; 0,010; 0,02; 0,05; 0,07; 0,1; 0,15; 0,20; 0,25 and 0,30 mg of each of As, Bi, Pb, Sb, Se, Sn, Te and Tl per litre (see table 2) and is used for analyte levels from 0,5 to 30,0 g/t. All solutions contain a matrix of 10 g of Ni per litre.

Table 2 — Set of calibration solutions A

Volume of mixed analyte standard solution A (4.3.2) (ml)	Analyte (As, Bi, Pb, Sb, Se, Sn, Te, Tl) concentration (mg/l)
0	The zero member
0,05	0,005
0,1	0,010
0,2	0,02
0,5	0,05
0,7	0,07
1,0	0,1
1,5	0,15
2,0	0,20
2,5	0,25
3,0	0,30

7.3.1.2 Transfer 2,50 ml of nickel nitrate stock solution (4.4) from a burette to each of 11, 10 ml one-mark volumetric flasks. Add, using a burette, 0; 0,05; 0,1; 0,2; 0,5; 0,7; 1,0; 1,5; 2,0; 2,5; and 3,0 ml respectively of mixed analyte standard solution A (4.3.2). Make up to the mark with nitric acid diluted 1 + 19 (4.2) and mix.

7.3.2 Set B

7.3.2.1 This set corresponds to 0; 0,000 5; 0,001; 0,002; 0,005; 0,010; 0,02 and 0,05 mg of each of Ag and Cd per litre (see table 3) and is used for analyte levels from 0,01 to 5,0 g/t. All solutions contain a matrix of 10 g of Ni per litre.

Table 3 — Set of calibration solutions B

Volume of mixed analyte standard solution B (4.3.3) (ml)	Analyte (Ag and Cd) concentration (mg/l)
0	The zero member
0,05	0,000 5
0,1	0,001
0,2	0,002
0,5	0,005
1,0	0,010
2,0	0,02
5,0	0,05

7.3.2.2 Transfer 2,50 ml of nickel nitrate stock solution (4.4) from a burette to each of eight 10 ml one-mark volumetric flasks. Add, using a burette, 0; 0,05; 0,1; 0,2; 0,5; 1,0; 2,0 and 5,0 ml respectively of mixed analyte standard solution B (4.3.3). Make up to the mark with nitric acid diluted 1 + 19 (4.2) and mix.

7.4 Calibration and determination

7.4.1 Spectrometric measurement

7.4.1.1 The spectral lines specified in table 4 shall be used in the analysis.

Table 4 — Spectral lines

Element	Ag	As	Bi1)	Cd	Pb
Wavelength (nm)	328,1	193,7	223,1	228,8	283,3
Element	Sb	Se	Sn	Te	Tl
Wavelength (nm)	217,6	196,0	286,3	214,3	276,8

1) An alternative line for bismuth is 306,8 nm.

7.4.1.2 Set the required instrument parameters and align the electrothermal atomizer according to the manufacturer's instructions. The use of background compensation is essential.

NOTE — Optimum settings for the operating parameters vary from instrument to instrument. Scale expansion may have to be used to obtain the required readability. Atomization temperatures of 2 600 to 2 700 °C are preferable for a nickel matrix.

7.4.1.3 Determine the optimum electrothermal atomizer parameters for the particular type of atomizer and sample size (5 to 25 µl) as recommended by the instrument manufacturer or normal laboratory practice.

7.4.1.4 Ensure that the test solution (7.1) and the calibration solutions (7.3) are within 1 °C of the same temperature.

7.4.1.5 Zero the instrument and set the baseline on the recorder.

7.4.1.6 Check the zero stability and lack of spectral interference within the atomization system by running the pre-set heating programme for blank firing of the graphite atomizer. Repeat to ensure baseline stability.

7.4.1.7 Inject into the atomizer the predetermined volume (5 to 25 µl) of each of the test solutions (7.1) for the element being determined. Atomize and note the instrument response. Sort the test solutions into groups of three or four with similar concentration levels of the analyte, starting with the lowest level.

7.4.1.8 Select the appropriate calibration solutions (7.3) to cover the range and bracket the concentration levels in the test solutions.

7.4.1.9 Inject and atomize the calibration and test solutions in order of increasing instrument response. Atomize each solution three times and, if the replication is good, average the readings. Check the instrument for memory effects, especially at high analyte levels, by running the blank firing programme. Reset the baseline to zero if necessary.

7.4.1.10 Evaluate the analyte contents in each group of test solutions based on the applicable standards as directed in 7.4.2.

7.4.2 Plotting of calibration graphs

7.4.2.1 Calculate the average of three instrument readings for each of the applicable calibration solutions.

7.4.2.2 Plot the average instrument reading against concentration of the analyte in the calibration solution.

If high purity nickel used to prepare the calibration solution is contaminated by the metal being determined, then graphical or computational methods shall be used to take this into account. However, the nickel nitrate stock solution can be purified by a solvent extraction procedure (see annex A).

NOTE — In this method, any effect of non-specific absorption and light scatter is compensated for by matching the matrix of the calibration solutions with the test solutions and by background correction. Also, since the same lot of nitric acid is used for both calibration and test solutions, the blank test is incorporated in the calibration graph. Thus, the calibration graph may not pass through the origin.

8 Expression of results

8.1 Calculation

8.1.1 Determine the concentration of the analyte in the test solution from the corresponding calibration graph (7.4.2) for the average of the three instrument readings recorded.

8.1.2 The analyte contents, expressed in grams per tonne, in the test portion, is given by the formula

$$\frac{\varrho V}{m}$$

where

ϱ is the concentration, expressed in milligrams per litre, of analyte found in the test solution;

V is the volume, in millilitres, of the test solution;

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

8.2 Precision

This International Standard was subjected to an interlaboratory test programme involving 11 laboratories in six countries. Six samples were analysed to cover the scope of the method. Of these, four were specially prepared by melting and granulation and two were commercial products.

Repeatability and reproducibility were calculated according to the principles of ISO 5725.

A statistical report of interlaboratory tests is given in annex B.

It should be noted that the reproducibility data include errors due to any inhomogeneity of the test samples.

9 Interferences and precautions

9.1 For the determination of silver and tin, care shall be taken to avoid contamination of the sample and calibration solutions with chloride ion.

9.2 Elements ordinarily present in nickel do not interfere in electrothermal atomic absorption analysis.

9.3 Potential background absorption interference is eliminated by instrumental background correction and by the use of matched matrix standards prepared from high purity nickel.

9.4 The lower limit for the determination of the elements is affected by the residual level of each element in the high purity nickel used to prepare the matched matrix standards.

9.5 Because of the high sensitivity of electrothermal atomic absorption, stringent precautions shall be taken to clean all glassware and avoid contamination of sample, standard and calibration solutions from foreign material and dust from the laboratory atmosphere.

10 Sample inhomogeneity

If some inhomogeneity is suspected in the laboratory sample, or if the pieces are relatively large, it is desirable to use a larger test portion to prepare the test solution. Under such circumstances a 10 g test portion in a final volume of 1 000 ml is recommended. The amount of nitric acid should be increased in proportion. Even larger test portions can be used to prepare a more concentrated nickel test solution. However, this shall then be diluted to give a test solution containing 10 g of Ni per litre to match the calibration solutions.

11 Test report

The test report shall include the following information:

a) the reference to the method used;

b) the results of the analysis;

c) the number of independent replications;

d) any unusual features noted during the analysis;

e) any operation not included in this International Standard or regarded as optional.