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Nickel alloys — Determination of lead — Electrothermal atomic absorption spectrometric method

Alliages de nickel — Détermination du plomb — Méthode par spectrométrie d'absorption atomique électrothermique

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys*.

This first edition cancels and replaces ISO 11437-1:1994 and ISO 11437-2:1994, which have been merged and technically revised.

Nickel alloys — Determination of lead — Electrothermal atomic absorption spectrometric method

1 Scope

This document specifies an electrothermal atomic absorption spectrometric method for the determination of lead in the range of 1 µg/g to 10 µg/g in nickel alloys.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 648, *Laboratory glassware — Single-volume pipettes*

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

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

4 Principle

Dissolution of a test portion in a mixture of nitric acid and hydrofluoric acid, dilution of the test solution to a known volume, and transfer of an aliquot to a plastic vial.

Addition of a modifier to the aliquot of the test solution, and injection of a small volume of this solution into the electrothermal atomizer of an atomic absorption spectrometer.

Measurement of the atomic absorption of the 283,3 nm spectral line energy emitted by a lead hollow-cathode lamp and comparison with those of the calibration solutions.

5 Reagents

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

5.1 **Pure nickel**, containing less than 1 µg/g of lead.

5.2 **Nickel**, base-solution 50 g/l.

Weigh, to the nearest 0,1 g, 25,0 g of pure nickel (5.1). Transfer to a 600 ml tall-form beaker and add 100 ml of water. Cautiously add 100 ml of nitric acid (ρ_{20} = 1,41 g/ml) in small portions, in such a manner that the dissolution remains under control. Cool the solution and transfer it to a 500 ml one-mark volumetric flask. Make up to the mark with water and mix.

If large nickel turnings or chunks are used, gentle heating may be required to complete the dissolution. When using carbonyl nickel powder, the solution should be filtered to remove undissolved carbon.

5.3 Nickel, base solution 5 g/l.

Transfer 10,0 ml of the nickel base solution (5.2) to a 100 ml one-mark volumetric flask. Make up to the mark with water and mix.

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

5.5 Hydrofluoric acid, ρ_{20} = 1,13 g/ml.

WARNING — Hydrofluoric acid is extremely irritating and corrosive to skin and mucous membranes producing severe skin burns which are slow to heal. In case of contact with skin, wash well with water, apply a topical gel containing 2,5 % (mass fraction) calcium gluconate and seek immediate medical treatment.

5.6 Nitric/hydrofluoric acids, mixture.

Carefully add 150 ml of nitric acid (ρ_{20} = 1,41 g/ml) and 150 ml of hydrofluoric acid (5.5) to 150 ml of water. Mix and store in a plastic bottle.

5.7 Modifier, nickel nitrate-ammonium phosphate solution.

Weigh, to the nearest 0,1 g, 6,0 g of ammonium dihydrogen orthophosphate ($NH_4H_2PO_4$) and dissolve it in 50 ml of water. Transfer the solution to a 100 ml one-mark volumetric flask. Add 20,0 ml of the nickel base solution (5.2), make up to the mark with water and mix.

This solution shall be freshly prepared.

5.8 Lead, standard solution, 100 mg/l.

Weigh, to the nearest 0,001 g, 0,100 g of lead of a mass fraction of 99,9 % minimum purity and transfer to a 250 ml beaker. Add 40 ml of nitric acid (5.6) and heat to assist dissolution. Cool the solution and transfer it to a 1 000 ml one-mark volumetric flask. Make up to the mark with water and mix.

Store in a polyethylene bottle.

1 ml of this solution contains 0,1 mg of lead.

5.9 Lead, standard solution, 1 mg/l.

Transfer 10,0 ml of the lead standard solution (5.8) into a 1 000 ml one-mark volumetric flask. Add 20 ml of nitric acid (ρ_{20} = 1,41 g/ml). Make up to the mark with water and mix.

1 ml of this solution contains 1 µg of lead.

This solution shall be freshly prepared.

6 Apparatus

All volumetric glassware shall be class A and calibrated, in accordance with ISO 648 or ISO 1042 as appropriate.

Before use, all glassware shall be cleaned by boiling with hydrochloric acid to remove any chemical contamination.

6.1 Polytetrafluoroethylene (PTFE) beakers, of capacity 100 ml.

6.2 Plastic vials, of capacity 5 ml.

6.3 Plastic volumetric flasks, of capacities 50 ml and 100 ml.

6.4 Micropipettes, of capacities from 2,0 µl to 100 µl.

6.5 Atomic absorption spectrometer.

The atomic absorption spectrometer to be used shall be fitted with an electrothermal atomizer and shall meet the performance criteria given in Annex A.

The atomic absorption spectrometer and electrothermal atomiser are satisfactory if, after optimization according to Annex A (A.3 to A.6), they meet the criteria specified in 7.1.

The spectrometer shall be equipped with a background corrector and a fast recording system capable of measuring peak heights and peak areas. The electrothermal atomizer shall be fitted with a pyrolytic graphite tube and L'vov platform, or a normal graphite tube.

The spectrometer should be capable of using single-element hollow cathode lamps or electrodeless discharge lamps operated at currents recommended by the manufacturer.

7 Instrument criteria

7.1 Preparation of solutions required for testing criteria

Follow the instructions given for the preparation of lead calibration solutions in 9.3 and 9.4.2.3. The composition of these calibration solutions is given in Table 1.

Table 1 — Lead concentration of calibration solutions

Calibration solution	Lead concentration		
	µg/litre	ng/ml	µg/g
S1	0	0	0
S2	10	10	2
S3	20	20	4
S4	30	30	6

7.2 Characteristic mass

The characteristic mass determined as described in A.7.1, shall be within 20 % of that given in the manufacturer's instructions.

7.3 Minimum precision

The minimum precision of calibration solution S4 shall not exceed 10 % of the mean absorbance of the same solution, and the minimum precision of calibration solution S2 shall not exceed 4 % of the mean absorbance of solution S4 when determined as described in A.7.2.

7.4 Limit of detection

The limit of detection of lead, determined as described in A.7.3, shall be less than 20 pg (equivalent to 1,0 µg/l or 0,2 µg/g).

7.5 Linearity

The linearity of the calibration, determined as described in A.7.4, shall not be less than 0,7.

8 Sampling and sample preparation

Sampling and preparation of the laboratory sample shall be carried out by normal agreed procedures or, in case of dispute, by appropriate national standards.

The laboratory sample normally is in the form of turnings, millings or drillings and no further mechanical preparation of the sample is necessary.

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

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

9 Procedure

9.1 Preparation of the test solution

Weigh, to the nearest 0,001 g, 0,500 g of the sample and transfer into a 100 ml PTFE beaker (6.1). Add 20 ml of the acid mixture (5.6). Apply sufficient heat to initiate and maintain the reaction until dissolution is complete.

Cool the solution and then transfer it into a 100 ml one-mark volumetric flask. Dilute to the mark with water and mix.

Dissolving certain alloys in the nitric acid-hydrofluoric acid mixture is sometimes difficult. In such cases, the proportions of the dissolving mixture should be adjusted, but a corresponding blank test is necessary.

9.2 Blank test

In parallel with the determination and following the same procedure, carry out a blank test using the same quantities of all reagents as used for the determination.

9.3 Preparation of the calibration solutions

Transfer 10,0 ml of the nickel base solution (5.2) into each of a series of 100 ml one-mark volumetric flasks. Add 0 ml; 0,5 ml;1,0 ml; 2,0 ml; 3,0 ml; 4,0 ml; 5,0 ml and 6,0 ml of the lead standard solution (5.9). Make up to the mark with water and mix.

These calibration solutions contain 0 µg; 5,0 µg; 10,0 µg; 20,0 µg; 30,0 µg; 40,0 µg; 50 µg and 60,0 µg of lead per litre.

9.4 Calibration and determination

9.4.1 Adjustment of the atomic absorption spectrometer

Equip the atomic absorption spectrometer with a pyrolytic graphite tube fitted with a L'vov platform. Condition new graphite tubes as instructed by the manufacturer.

Set up the instrument conditions for

— measuring peak area integration absorbance at a wavelength of 283,3 nm, and

— using an injection volume of 20 µl.

NOTE The volume injected into the furnace may be different, depending on the sensitivity of the instrument.

Establish the optimum furnace temperature programme in accordance with the instructions given in Annex A.

9.4.2 Atomic absorption measurements

9.4.2.1 General

The procedures described in this subclause shall be carried out immediately prior to the measurement.

9.4.2.2 Lead contents of up to 5,0 µg/g

With a micropipette (6.4) transfer 1,0 ml of each test solution containing up to 25 µg/l of lead and of each calibration solution containing up to 30 µg/l of lead into 5 ml plastic vials (6.2). With a micropipette (6.4), add 100 µl of the modifier solution (5.4) and mix.

9.4.2.3 Lead contents between 5,0 µg/g to 10,0 µg/g

With a micropipette (6.4) transfer 0,50 ml of each test solution containing between 25 µg and 50 µg of lead per litre, and of the calibration solutions containing 0 µg; 20,0 µg; 40,0 µg; 50 µg and 60,0 µg of lead per litre, into 5 ml plastic vials (6.2). With micropipettes (6.4), add 0,50 ml of the nickel base solution (5.3) and 100 µl of the modifier solution (5.4) and mix.

NOTE 1 If the electrothermal atomizer is fitted with an autosampler, the addition of modifier, dilution and mixing can be done in the autosampler cups.

NOTE 2 Mixing can be carried out by repeated charging and discharging of the contents of the plastics vial using the largest micropipette.

9.4.2.4 Preparation of the calibration curves

Atomize the preselected volume of each calibration solution and record three absorbance measurements for each of them.

Subtract the mean absorbance value obtained for the 0 µg/l calibration solution from the mean absorbance values obtained for the other calibration solutions.

Establish a graph relating the mean absorbance values obtained for the calibration solutions to their analyte concentrations (in micrograms per litre).

9.4.2.5 Determination

Check the calibration slope by atomizing the preselected volume of the zero and highest calibration solutions and record three measurements for each solution.

Atomize the preselected volume of the blank test solution. Record three absorbance measurements.

Atomize the preselected volume of two of the test solutions. Record three absorbance measurements for each.

Repeat the measurement instructions above until all of the test solutions are measured.

Calculate the mean of the three absorbance measurements obtained in each case.

9.5 Number of determinations

Carry out the determination at least in duplicate.

10 Expression of results

10.1 Calculation

Using the mean absorbance obtained for the blank test solution (9.2), determine the lead concentration in the blank test solution from the calibration curve (9.4.2.4).

If the calibration check measurement as described in 9.4.2.5 shows that the calibration curve has drifted significantly, adjust the calibration curve accordingly.

Using the mean absorbance values obtained in 9.4.2.5, determine the lead concentration in two of the test solutions.

Repeat the procedure above until the lead concentrations of the remaining test solutions are obtained.

Subtract the lead concentration in the blank test solution (9.2) from the lead concentration in the test solutions.

Calculate the lead content w of the test sample, in micrograms per gram, using Formula (1):

$$w = \rho/(10^* m) \quad (1)$$

where

ρ is the lead concentration, expressed in micrograms per litre, in the test solutions;

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

10.2 Precision

10.2.1 Interlaboratory tests

Nine laboratories in four countries participated in the testing of this procedure using six samples having the nominal composition given in Table 2.

Table 2 — Nominal composition of test samples (% in mass fraction)

Sample label	Pb	Co	Cr	Mo	Ta	Ti	Al	Hf	W	Ni
1	0,000 1	15	15	5	—	2,5	2,5	—	—	Remainder
2	0,001	15	15	5	—	2,5	2,5	—	—	Remainder
3	< 0,001	10	8	—	2,5	1,5	5	1,5	10	Remainder
4	0,000 4	10	8	—	2,5	1,5	5	1,5	10	Remainder
5	0,001	10	8	—	2,5	1,5	5	1,5	10	Remainder
6	< 0,000 05	14	10	3	—	4,5	6	Vanadium:1		Remainder

10.2.2 Precision data

Results from the interlaboratory test programme were evaluated according to ISO 5725:1986. The data were tested for statistical outliers by the Cochran and Dixon tests described in ISO 5725:1986.

The principle of the Cochran test is that a set of results is an outlier if the within-laboratory variance is too large in relation to the others. Dixon's test is to determine if the mean from a laboratory is too far from the other laboratory means. Both tests were applied at the 95 % confidence level.

Repeatability and reproducibility were also calculated according to ISO 5725:1986 at the 95 % confidence level.

The results of the statistical assessment are given in Table 3.

Table 3 — Precision data

Sample label	Mean µg/g	Repeatability µg/g	Reproducibility µg/g
1	1,6	0,26	0,45
2	9,6	1,31	2,13
3	0,14	0,33	0,42
4	4,0	0,59	0,86
5	10,7	1,42	2,06
6	0,04	0,27	0,26

11 Test report

The test report shall include the following information:

a) the method used by reference to this document, i.e. ISO 11437;

b) all information necessary for the identification of the sample, the laboratory, and the date of analysis or of the test report;

c) results and the units in which they are expressed;

d) the number of independent replications;

e) any unusual characteristics noted during the determination;

f) any operation not specified in this document or any operation which might have influenced the results;

g) signature of the responsible person.

Annex A
(normative)

Optimization and checking of spectrometer performance criteria

A.1 General

To obtain the best results when using the graphite furnace technique, the instrument settings, particularly the furnace programme, shall be optimized. Once the instrument settings are optimized, it is essential that the instrument meets certain performance requirements before it is used for any determination.

A.2 Initial instrument checks and adjustments

Switch on the power, cooling water, gas supplies and fume extraction system.

Open the furnace and inspect the tube and contacts. Replace the graphite components if wear or contamination is evident. Inspect the windows and clean if necessary.

If a new tube or graphite contacts are fitted, condition these using the heating programme recommended by the manufacturer.

In the absence of the manufacturer's recommendations, the conditioning furnace programme shown in Table A.1 should be used.

Table A.1 — Programme for graphite tube conditioning

Step	Temperature °C	Increase time s	Hold time s	Gas flow ml/min
1	1 500	60	20	300
2	20	1	10	300
3	2 000	60	20	300
4	20	1	10	300
5	2 600	60	10	300
6	20	1	10	300
7	2 650	2	5	0

A.3 Radiation source

Both single-element hollow cathode lamps and electrodeless discharge lamps are suitable. These should be installed and operated as recommended by the manufacturer.

After the stabilization time specified by the manufacturer, the signal from each radiation source should not deviate by more than 0,5 % from the maximum value (i.e. by not more than 0,002 absorbance units) over a period of 15 min. Significantly greater fluctuations are usually indicative of a faulty lamp.

The use of multi-element lamps is not generally recommended.

A.4 Spectrometer parameters

A.4.1 Wavelength

Select the appropriate wavelength.

A.4.2 Slit

Select the slit width recommended by the manufacturer. Where two slit settings are available, ensure that the type provided for use with the graphite furnace is selected.

A.4.3 Background correction

A.4.3.1 Deuterium background correction systems

Select the background correction option and allow the lamps to stabilize for 30 min. Check that the energies of the analyte radiation source and the deuterium radiation source are balanced within the tolerances recommended by the manufacturer.

A.4.3.2 Zeeman background correction systems

Ensure that the poles of the magnet are clean.

A.4.3.3 Test of background correction system

Measure the atomic and background absorbances of 20 µl of a 2 g/l magnesium nitrate solution at a wavelength between 200 nm and 250 nm (e.g. Bi 223,1 nm) using a charring temperature of 950 °C and an atomization temperature of 1 800 °C. A large background signal should be observed, with no over or under correction of the atomic signal.

NOTE In general, the deuterium correction system will be able to correct for broad band background absorbances of up to 0,5 to 0,6 absorbance units. Zeeman systems generally will be able to cope with levels as high as 1,0 to 1,5 absorbance units.

A.5 Autosampler

The operation of the autosampler should be checked. Particular attention should be paid to the condition of the pipette tip and the position of the tip during sample deposition. The manufacturer's instructions regarding the adjustment of the autosampler should be followed.

A.6 Optimization of the furnace heating programme

WARNING — Do not view the tube directly during the charring (A.6.3), atomization (A.6.4) or cleaning (A.6.4) steps.

A.6.1 General

Optimization of the furnace heating programme is essential if good results are to be obtained using this technique. Furnace programmes recommended by manufacturers are often concerned with samples completely unrelated to nickel alloys. Consequently, the analyst shall optimize the furnace programme for use with the nickel alloy matrix in the manner described in A.6.2 to A.6.5.

The furnace programme for the nickel alloy matrix being considered here consists of four basic steps: drying, charring, atomization and cleaning.

A.6.2 Drying

For most samples a drying temperature of 120 °C is satisfactory. To avoid spattering, the temperature should be increased to 120 °C in 20 s and then held at that temperature for a time depending on the volume of sample introduced. The hold times shown in Table A.2 are typical.

Table A.2

Injected volume, µl	Hold time, s
10	15
40	30

When samples are deposited on the L'vov platform, a two-stage drying process is beneficial in preventing spattering.

The first stage involves heating the sample rapidly to 80 °C in 1 s and then holding the temperature at 80 °C for a short time. The time during which the temperature is maintained at 80 °C depends on the volume of solution injected. The hold times shown in Table A.3 are typical.

Table A.3

Injected volume, µl	Hold time, s
10	15
40	30

The temperature is then increased over a period of 20 s to 30 s, to a value 20 °C to 40 °C above the boiling point of the solvent. This higher temperature is held for 15 s to 40 s depending on the volume of sample injected. The hold times shown in Table A.4 are typical.

Table A.4

Injected volume, µl	Hold time, s
10	15
40	40

Once suitable drying conditions have been selected, the drying process can be monitored visually with the aid of a dental mirror to ensure that it proceeds smoothly without spattering.

A.6.3 Charring

A.6.3.1 During this step, volatile components of the matrix are driven off and precursor reactions occur, for example reduction of the analyte oxide to the elemental state and the formation of matrix refractory oxides and carbides.

NOTE Because of the low volatility of the nickel alloy matrix, most of it will remain in the furnace after charring.

The analyst shall first select the combination of graphite tube, normal or L'vov platform, and measuring mode, peak height or peak area to be used.

The analyst shall then determine the optimum charring temperature experimentally as described in A.6.3.2 to A.6.3.11.

A.6.3.2 Use the optimum drying conditions as determined in A.6.2

At this stage, both the optimum charring and atomization settings are unknown. Estimates for suitable atomization settings should be made and the charring conditions optimized first.

A.6.3.3 Consult the manufacturer's instructions and set the atomization temperature accordingly. Select the GAS STOP option. Select an atomization integration time of 10 s. This ensures that the whole of the analyte peak will be measured.

A.6.3.4 Select a charring time comprising a 30 s increase and a 30 s hold.

A.6.3.5 Select a calibration solution which will give an absorbance reading of 0,2 to 0,4.

A.6.3.6 Vary the charring temperature, in steps of 100 °C, throughout the range 500 °C to 1 400 °C taking three measurements for the calibration solution at each step.

A.6.3.7 Calculate the mean of the three measurements attained for each temperature step. Plot a graph relating the charring temperature to the mean absorbance. Note the temperature at which the absorbance starts to decrease. Subtract 50 °C from this value to obtain the optimum charring temperature.

NOTE The 50 °C allowance is to accommodate any day-to-day variations in the working of the temperature measuring system.

A.6.3.8 Use the optimum charring temperature found in A.6.3.7 and vary the hold time over the range 15 s to 60 s in steps of 15 s. Take three measurements for the calibration solution during each step. Monitor the background signal during this process. Note the time at which the background signal returns to the baseline.

A.6.3.9 Calculate the mean of the three absorbance measurements. There shall be no evidence of analyte loss (indicated by lower absorbances for the longer hold times).

A.6.3.10 Provided the condition in A.6.3.9 is satisfied, select the shortest time which the background signal returns to the baseline (A.6.3.8). Add 10 s to this value to obtain the optimum hold time.

A.6.3.11 If the condition in A.6.3.9 is not satisfied, repeat actions A.6.3.8 to A.6.3.10 using a charring temperature which is 50 °C less than that originally selected in A.6.3.7.

NOTE A slow time of 30 s and a hold time of 30 s are usually sufficient for all pre-treatment reactions to occur. Short increase times may provoke loss of sample from the tube caused by explosive splatter.

A.6.4 Atomization

A.6.4.1 This step involves the production of gaseous analyte atoms inside the graphite tube. As far as is possible, matrix atoms should be absent to minimize interference.

The analyst shall determine the optimum atomization temperature and integration time experimentally using the same GAS STOP, graphite tube and measuring mode combination selected before the optimization of the charring step.

Although it is possible to optimize the L'vov platform using the peak height measurement mode to achieve its full potential, the analyst should optimize the atomization step in such a manner that the conditions required for the stabilized temperature platform furnace (STPF) operation are satisfied. In addition to the use of GAS STOP and matrix modification (inherent in the procedure), the following additional conditions should be satisfied.

a) The temperature difference between the charring step and the atomization step should be as small as possible (less than 1 000 °C). This allows the furnace to approach conditions which are nearly isothermal more quickly, and reduces the amount of matrix volatilized.

b) The peak area integration measurement mode should be used.

c) There shall be zero time increase between the charring and atomization steps.

A.6.4.2 Use the optimum drying and charring conditions determined in A.6.2 and A.6.3, respectively.

A.6.4.3 Select an atomization temperature of 1 200 °C and an integration time of 20 s.

A.6.4.4 Using the same calibration solution (A.6.3.5), obtain three absorbance measurements at this atomization temperature.

A.6.4.5 Vary the atomization temperature by increasing it up to 2 000 °C in 100 °C steps. Measure the absorbance of the calibration solution during each step as directed in A.6.4.4.

A.6.4.6 Plot the mean of the three measurements obtained for each step against the atomization temperature.

A.6.4.7 Examine the graph and determine the lowest atomization temperature where maximum absorbance is obtained. Add 200 °C to this value to obtain the optimum atomization temperature.

NOTE At the lowest atomization temperature giving maximum absorbance when using the peak area integration measurement mode, the peaks are broad with considerable tailing. The extra 200 °C will overcome these problems.

If the L'vov platform is to be used under STPF conditions, check that the difference between the optimum charring temperature and optimized atomization temperature does not exceed 1 000 °C.

A.6.4.8 Regarding the duration of the atomization step, use the optimum temperature found in A.6.4.7 and a hold time of 10 s.

a) Instruments equipped with a VDU or fast recorder.

   Measure the calibration solution (A.6.3.5) and observe the atomic signal during the atomization stage. Determine the optimum hold time by adding 1 s to the time taken for the trace to return to the zero-axis of the absorbance scale.

b) Instruments without a VDU or fast recorder.

   Make three measurements of the calibration solution used in A.6.3.5. Calculate the mean absorbance. Repeat these measurements using progressively shorter hold times (1 s intervals) until the mean of this three measurements starts to decrease. Add 1 s to the hold time at this point to obtain the optimum hold time.

A.6.5 Cleaning step

Heat the furnace at 2 650 °C for 5 s to remove as much of the residual matrix as possible.

NOTE In practice, the matrix elements that form refractory oxides and carbides cannot be completely removed, even at high temperatures.

A.7 Instrument performance criteria

A.7.1 General

The following tests shall be performed after optimization of the instruments described in A.2 to A.6, using the calibration solutions given in Table 1.

A.7.2 Determination of characteristic mass, m_c

Measure the absorbance of calibration solution S1 three times, using the injection volume recommended in the procedure. Calculate the mean absorbance $\bar{A}_{S1}$.

