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Nickel alloys — Determination of trace-element content by electrothermal atomic absorption spectrometric method —

Part 2:
Determination of lead content

Alliages de nickel — Dosage des éléments-traces — Méthode par spectrométrie d'absorption atomique à excitation électrothermique — Partie 2: Dosage du plomb

Reference number
ISO 11437-2:1994(E)

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.

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.

International Standard ISO 11437-2 was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys*, Subcommittee SC 4, *Analysis of nickel alloys*.

ISO 11437 consists of the following parts, under the general title *Nickel alloys — Determination of trace-element content by electrothermal atomic absorption spectrometric method*:

— *Part 1: General requirements and sample dissolution*

— *Part 2: Determination of lead content*

— *Part 3: Determination of bismuth content*

— *Part 4: Determination of silver content*

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Nickel alloys — Determination of trace-element content by electrothermal atomic absorption spectrometric method —

Part 2:
Determination of lead content

1 Scope

This part of ISO 11437 specifies an electrothermal atomic absorption spectrometric method for the determination of lead in the range of 0,5 g/tonne to 10,0 g/tonne in nickel alloys.

The general requirements concerning the apparatus, sampling, dissolution of the test sample, atomic absorption measurements, calculation and test report are given in ISO 11437-1.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 11437. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 11437 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

ISO 11437-1:1994, *Nickel alloys — Determination of trace-element content by electrothermal atomic absorption spectrometric method — Part 1: General requirements and sample dissolution.*

3 Principle

Dissolution of a test portion in a mixture of acids. Addition of a modifier to an aliquot of the test solution.

Transfer of a small volume of the test solution to a graphite furnace electrothermal atomizer.

Measurement of the absorbance of the resonance line energy from the spectrum of lead and comparison with that of calibration solutions at a wavelength of 283,3 nm.

4 Reagents

In addition to the reagents listed in ISO 11437-1, the following special reagents are required.

4.1 Pure nickel metal, containing less than 1 g/tonne of lead.

4.2 Nickel, stock solution (50 g/l).

Weigh, to the nearest 0,1 g, 25,0 g of the pure nickel (4.1). 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.

NOTE 1 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.

4.3 Nickel, solution (5,0 g/l).

Transfer 10,0 ml of the nickel stock solution (4.2) to a 100 ml one-mark volumetric flask. Make up to the mark with water and mix.

4.4 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 stock solution (4.2), make up to the mark with water and mix.

This solution shall be freshly prepared.

4.5 Lead, standard reference solution (100 mg/l).

Weigh, to the nearest 0,001 g, 0,100 g of lead metal of 99,9 % (*m/m*) minimum purity and transfer to a 250 ml beaker. Add a mixture of 20 ml of nitric acid ($\rho_{20} = 1{,}41$ g/ml) and 20 ml of water and heat to assist dissolution. Cool the solution and transfer it to a 1 litre one-mark volumetric flask. Make up to the mark with water and mix. Store in a polyethylene bottle.

4.6 Lead, standard solution (1,0 mg/l).

Pipette 10,0 ml of the lead standard reference solution (4.5) into a 1 litre one-mark volumetric flask. Add 20 ml of nitric acid ($\rho_{20} = 1{,}41$ g/ml). Make up to the mark with water and mix.

This solution shall be freshly prepared.

5 Apparatus

The apparatus required is specified in clause 5 of ISO 11437-1:1994.

The atomic absorption spectrometer and electrothermal atomizer are satisfactory if, after optimization according to annex A (A.2 to A.6) of ISO 11437-1:1994, they meet the criteria specified in 5.2.

5.1 Preparation of solutions required for testing criteria

Follow the instructions given for the preparation of lead calibration solutions in 7.3 and 7.4.1.1. The composition of these calibration solutions is given in table 1.

Table 1 — Lead concentration of calibration solutions

Calibration solution	Concentration of Pb		
	µg/litre	g/tonne	ng/ml
S1	0	0	0
S2	10	2	10,0
S3	20	4	20,0
S4	30	6	30,0

5.2 Instrument criteria

5.2.1 Characteristic mass

The characteristic mass determined in annex A (A.7.1) of ISO 11437-1:1994, shall be within 20 % of that given in the manufacturer's literature.

5.2.2 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 annex A (A.7.2) of ISO 11437-1:1994.

5.2.3 Limit of detection

The limit of detection of lead, determined as described in annex A (A.7.3) of ISO 11437-1:1994, shall be less than 20 pg (equivalent to 1,0 µg/l or 0,2 g/tonne).

5.2.4 Linearity

The linearity of the calibration, determined as described in annex A (A.7.4) of ISO 11437-1:1994, shall not be less than 0,7.

6 Sampling and sample preparation

Refer to clause 6 of ISO 11437-1:1994.

7 Procedure

7.1 Preparation of the test solution

Proceed as directed in 7.1 of ISO 11437-1:1994. Transfer the solution to a 100 ml one-mark volumetric flask. Dilute to the mark and mix.

7.2 Reagent blank test

Carry out a blank test in parallel with the determination, following the same procedure and using the same quantities of all the reagents.

7.3 Lead calibration solutions

Using the 25 ml burette, transfer 10,0 ml of the nickel stock solution (4.2) to each of six 100 ml one-mark volumetric flasks. Using the 10 ml burette, add 0 ml; 1,0 ml; 2,0 ml; 3,0 ml; 4,0 ml and 6,0 ml of the lead standard solution (4.6). Make up to the mark with water and mix.

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

7.4 Calibration and determination

The procedures described in 7.4.1 shall be carried out immediately prior to the measurement.

7.4.1 Dilutions and addition of the nickel-phosphate modifier

7.4.1.1 Lead contents of up to 5,0 g/tonne in the test sample

Transfer 1,0 ml of each test solution containing up to 25 µg Pb/l and of each calibration solution containing up to 30 µg Pb/l to 5 ml plastics vials using a micropipette. Add 100 µl of the modifier solution (4.4) using a micropipette and mix.

7.4.1.2 Lead contents of 5,0 g/tonne to 10,0 g/tonne in the test sample

Transfer 0,50 ml of each test solution containing between 25 µg and 50 µg of lead per litre, and of calibration solutions containing 0 µg; 20,0 µg; 40,0 µg; and 60,0 µg of lead per litre, to 5 ml plastics vials using a micropipette. Add 0,50 ml of the nickel solution (4.3) and 100 µl of the modifier solution (4.4) using micropipettes and mix.

NOTES

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

3 Mixing can be done by repeated charging and discharging of the contents of the plastics vial using the largest micropipette.

7.4.2 Electrothermal atomic absorption measurements

Proceed as directed in 7.4.1 of ISO 11437-1:1994 using

- a pyrolytic graphite tube fitted with a L'vov platform;
- peak area integration absorbance measurement at a wavelength of 283,3 nm;
- an injection volume of 20 µl.

7.4.3 Preparation of calibration graphs

Proceed as directed in 7.4.2 of ISO 11437-1:1994.

7.5 Number of determinations

Carry out the determination at least in duplicate.

8 Expression of results

8.1 Calculation

Proceed as directed in 8.1 of ISO 11437-1:1994. Use a dilution factor, F, of 1 in the calculation in 8.1.6 of ISO 11437-1.

8.2 Precision

8.2.1 Laboratory tests

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

8.2.2 Statistical analysis

The results were treated according to ISO 5725 as described in 8.2.2 of ISO 11437-1:1994. The results of this analysis are given in table 3.

9 Test report

Refer to clause 9 of ISO 11437-1:1994.

Table 2 — Nominal composition of test samples [% (m/m)]

Sample	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	< 0,000 1	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	1 (V)		Remainder

Table 3 — Results of statistical analysis

Sample reference	Mean g/tonne	Repeatability	Reproducibility
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