INTERNATIONAL STANDARD

ISO 11438-2

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

Part 2: Determination of lead content

Ferro-nickel — Dosage des éléments-traces — Méthode par spectrométrie d'absorption atomique à excitation électrothermique —

Partie 2: Dosage du plomb

INTERNATIONAL ISO

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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. 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 11438-2 was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys*, Sub-Committee SC 3, *Analysis of nickel and ferronickel*.

ISO 11438 consists of the following parts, under the general title *Ferronickel — 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 antimony content*

— *Part 4: Determination of tin content*

— *Part 5: Determination of tellurium content*

— *Part 6: Determination of thallium content*

— *Part 7: Determination of silver content*

— *Part 8: Determination of indium content*

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

Part 2:
Determination of lead content

1 Scope

This part of ISO 11438 specifies an electrothermal atomic absorption spectrometric method for the determination of lead in the range of 1,0 g/t to 5,0 g/t in ferronickel, according to the principle of standard additions.

The general requirements concerning the apparatus, sampling, dissolution of the test sample, procedure, calculation and test report are given in ISO 11438-1.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 11438. 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 11438 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 11438-1:1993, *Ferronickel — 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 nitric acid.

Measurement of the absorption of the resonance line energy from the spectrum of lead in the test solution at a wavelength of 283,3 nm by an atomic absorption spectrometer fitted with a graphite furnace electrothermal atomizer.

Calibration by the standard additions method described in ISO 11438-1.

4 Reagents

In addition to the reagents listed in ISO 11438-1, the following special reagents are required.

4.1 Lead, standard reference solution (1 000 mg/l).

Weigh, to the nearest 0,001 g, 1,000 g of lead metal of 99,9 % (*m/m*) minimum purity. Transfer to a 100 ml beaker and dissolve in a mixture of 10 ml of nitric acid (ρ_{20} = 1,41 g/ml) and 20 ml of water. Heat until completely dissolved. Boil to expel nitrogen oxides. Cool the solution and transfer to a 1 000 ml one-mark volumetric flask. Add 10 ml of nitric acid (ρ_{20} = 1,41 g/ml), make up to the mark with water and mix thoroughly.

4.2 Lead, standard solution (10,0 mg/l).

Pipette 10,0 ml of the lead standard reference solution (4.1) into a 1 000 ml one-mark volumetric flask containing 50 ml of nitric acid (ρ_{20} = 1,41 g/ml) diluted 1 + 1. Make up to the mark with water and mix thoroughly.

This solution shall be prepared on the day of use.

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

Pipette 10,0 ml of the lead standard solution (4.2) into a 100 ml one-mark volumetric flask containing 5 ml of nitric acid (ρ_{20} = 1,41 g/ml) diluted 1 + 1. Make up to the mark with water and mix thoroughly.

This solution shall be freshly prepared.

5 Apparatus

The apparatus required is specified in clause 5 of ISO 11438-1:1993.

6 Sampling and sample preparation

Refer to clause 6 of ISO 11438-1:1993.

7 Procedure

7.1 Preparation of the test solution

Proceed as directed in 7.1 of ISO 11438-1:1993.

7.2 Blank test

Refer to 7.2 of ISO 11438-1:1993.

7.3 Determination by the standard additions method

7.3.1 Atomic absorption measurements

Use the peak area integration absorbance measurement at a wavelength of 283,3 nm and proceed with checking the electrothermal atomizer as directed in 7.3.1 of ISO 11438-1:1993.

7.3.2 Semi-quantitative estimation of the lead content

Proceed as directed in 7.3.2 of ISO 11438-1:1993.

7.3.3 Quantitative determination of the lead content

Proceed as directed in 7.3.3 of ISO 11438-1:1993.

7.3.4 Plotting of standard additions

Proceed as directed in 7.3.4 of ISO 11438-1:1993.

NOTE 1 The procedure is applicable to the linear part of the graphs.

7.4 Number of determinations

Carry out the determination at least in duplicate.

8 Expression of results

8.1 Calculation

8.1.1 Semi-quantitative estimation of the lead content

Proceed as directed in 8.1.1 of ISO 11438-1:1993.

8.1.2 Quantitative determination of the lead content

Proceed as directed in 8.1.2 of ISO 11438-1:1993.

8.1.3 Calculation of the lead content

Calculate the lead content w_{Pb} of the test sample, in grams per tonne, using the formula

$$w_{Pb} = \frac{F\rho_{Pb}}{10m}$$

where

ρ_{Pb} is the lead concentration, in micrograms per litre, found in the "zero" test solution, in accordance with 8.1.2 of ISO 11438-1:1993;

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

F is the dilution factor of 2,5.

8.2 Precision

8.2.1 Laboratory tests

Six laboratories in five countries participated in the testing of this procedure using two samples of nominal composition given in table 1.

Samples were analysed three times on different days.

8.2.2 Statistical analysis

8.2.2.1 Results from the interlaboratory test programme were evaluated according to ISO 5725 as described in 8.2.2 of ISO 11438-1:1993. The results of this analysis are given in table 2.

8.2.2.2 All the laboratories passed statistical tests.

9 Test report

Refer to clause 10 of ISO 11438-1:1993.

Table 1 — Nominal composition of test samples

Sample	Content, g/t														Content, % (*m/m*)	
	Pb	Sb	Sn	Te	Tl	Ag	In	Bi	As	Se	Cd	Ga	Ge	Zn	Ni	Fe
1	1	1	1	0,5	0,5	1	0,5	< 0,1	3	0,5	0,5	2	1	2	25	Remainder
2	6	4	10	2	1	6	2	1	5	3	1	4	4	5	25	Remainder

Table 2 — Results of statistical analysis

Sample	1	2
Mean w_{Pb}, g/t	1,47	5,45
Within-laboratory standard deviation	0,4	0,3
Between-laboratory standard deviation	0,2	1,3
Repeatability	1,0	0,7
Reproducibility	1,2	3,8

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Descriptors: nickel alloys, iron nickel alloys, ferroalloys, ferronickel, chemical analysis, determination of content, trace elements, lead, atomic absorption spectrometric method.

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