

Sampling and analysis of nickel, ferronickel and nickel alloys —

Part 4: Method for determination of silver, arsenic, bismuth, cadmium, lead, antimony, selenium, tin, tellurium and thallium in nickel by electrothermal atomic absorption spectrometry

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

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Committees responsible for this British Standard

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- British Non-ferrous Metals Federation
- British Steel Industry
- Engineering Equipment and Materials Users' Association
- Ministry of Defence
- Process Plant Association
- Stainless Steel Fabricators' Association of Great Britain
- Coopted members

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National foreword

This Part of BS 6783 has been prepared under the direction of the Non-ferrous Metals Standards Committee. It is identical with ISO 7523:1985 “*Nickel — Determination of silver, arsenic, bismuth, cadmium, lead, antimony, selenium, tin, tellurium and thallium contents — Electrothermal atomic absorption spectrometric method*” published by the International Organization for Standardization (ISO).

At present this British Standard consists of nine Parts all concerned with analysis of nickel, ferronickel and nickel alloys. Further International Standards are in preparation on sampling and analysis of nickel, ferronickel and nickel alloys and, when available, these will be published as further Parts of this British Standard.

Terminology and conventions. The text of the International Standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

Wherever the words “International Standard” appear, referring to this standard, they should be read as “Part of BS 6783”.

In British Standards it is current practice to use the symbol “L” for litre (and in its submultiples) rather than “l”.

Cross-references

International Standards	Corresponding British Standards
ISO 1042:1983	BS 1792:1982 <i>Specification for one-mark volumetric flasks</i> (Identical)
ISO 3534:1977	BS 5532 <i>Statistical terminology</i> Part 1:1978 <i>Glossary of terms relating to probability and general terms relating to statistics</i> (Identical)
ISO 5725-1981	BS 5497 <i>Precision of test methods</i> Part 1:1979 <i>Guide for the determination of repeatability and reproducibility for a standard test method</i> (Technically equivalent)

The Technical Committee has reviewed the provisions of ISO 385-1, ISO 648 and ISO 6283, to which reference is made in the text, and has decided that they are acceptable for use in conjunction with this standard.

ISO 385-1 is related to BS 846:1985 “*Specification for burettes*” and ISO 648 is related to BS 1583:1986 “*Specification for one-mark pipettes*”.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 8, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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 ^a ($\mu\text{g/g}$) ^b
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

^a For specific compositions, see ISO 6283.
^b 1 $\mu\text{g/g}$ = 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, $\rho_{20} = 1,41$ 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, $\rho_{20} = 1,41$ 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 $[\text{K}(\text{SbO})\text{C}_4\text{H}_4\text{O}_6 \cdot 1/2\text{H}_2\text{O}]$, 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.