

Sampling and analysis of nickel, ferronickel and nickel alloys —

**Part 14: Method for the determination
of total boron content of nickel, and
nickel alloys by curcumin molecular
absorption spectrometry**

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Committees responsible for this British Standard

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- Association of Bronze and Brass Founders
- British Non-ferrous Metals Federation
- British Stainless Steel Association
- British Steel Industry
- Department of Trade and Industry (Laboratory of the Government Chemist)
- Engineering Equipment and Materials Users' Association
- Institute of British Foundrymen
- London Metal Exchange
- Ministry of Defence
- Nickel Development Institute
- Non-ferrous Metal Stockists
- Steel Casting Research and Trade Association
- 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 Policy Committee. It is identical with ISO 11436:1993 *Nickel and nickel alloys — Determination of total boron content — Curcumin molecular absorption spectrometric method*, published by the International Organization for Standardization (ISO).

At present this British Standard consists of 15 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.

Cross-reference

International Standard	Corresponding British Standard
ISO 5725:1986	BS 5497 <i>Precision of test methods</i> Part 1:1987 <i>Guide for the determination of repeatability and reproducibility for a standard test method by inter-laboratory tests</i> (Identical)

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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

This International Standard specifies a molecular absorption spectrometric method for the determination of the total boron content in the range of 4 g/t to 240 g/t in nickel and nickel alloys.

NOTE 1 A possible chemical interference from rhenium has been identified.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard 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*.

3 Principle

Dissolution of a test portion in hydrochloric and nitric acids. Decomposition of resistant boron compounds (e.g. boron nitrides) by fuming the sample solution with phosphoric and sulfuric acids at not less than 290 °C for 30 min.

Formation of the boron curcumin complex in a buffered acetic acid and sulfuric acid medium, and measurement of the absorbance of the test solution at a wavelength of 543 nm.

4 Reagents

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

4.1 Hydrochloric acid, $\rho_{20} = 1,18$ g/ml.

4.2 Sulfuric acid, $\rho_{20} = 1,84$ g/ml.

4.3 Nitric acid, $\rho_{20} = 1,41$ g/ml.

4.4 Phosphoric acid, $\rho_{20} = 1,71$ g/ml.

4.5 Sodium hypophosphite, monohydrate ($\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$).

4.6 Acetic acid, glacial, $\rho_{20} = 1,05$ g/ml.

The reagent must be free from aldehydes.

Aldehyde test procedure: Transfer 20 ml of the acetic acid to a 50 ml beaker and add 1 ml of 1 g/l potassium permanganate solution. The colour should not disappear within 10 min. If aldehydes are present, an easily visible brown colour will develop in 15 min.

4.7 Acetic acid-sulfuric acid mixture, 1 + 1.

Add in small portions, while stirring and cooling under running water, one volume of the sulfuric acid (4.2) to one volume of the acetic acid (4.6).

4.8 Acetate buffer, solution.

Dissolve 225 g of ammonium acetate ($\text{CH}_3\text{COONH}_4$) in 400 ml of water. Add 300 ml of the acetic acid (4.6). Filter the solution through a medium filter paper and dilute to 1 000 ml. Store in a polyethylene bottle.

4.9 Sodium fluoride, solution.

Dissolve 4 g of sodium fluoride (NaF) in 100 ml of water and mix.

4.10 Boron, standard reference solution (100 mg/l).

Weigh, to the nearest 0,000 1 g, 0,285 8 g of boric acid (H_3BO_3) and transfer it to a 500 ml one-mark volumetric flask. Dissolve the acid in water, make up to the mark and mix. Store the solution in a polyethylene flask.

4.11 Boron, standard solution (2 mg/l).

Transfer 20,0 ml of the boron stock solution (4.10) to a 1 000 ml one-mark volumetric flask. Dilute to volume with water and mix. Store the solution in a polyethylene flask. Prepare freshly before use.

4.12 Curcumin, solution.

Dissolve 0,125 g of curcumin ($\text{C}_{26}\text{H}_{20}\text{O}_6$) in 60 ml of the acetic acid (4.6) in a polyethylene or quartz vessel, by heating in a water bath and using a magnetic stirrer. Cool and transfer to a 100 ml plastics volumetric flask (5.3). Dilute to volume with the acetic acid (4.6) and mix.

This solution must be prepared freshly before use.

5 Apparatus

All glassware and plastics flasks used in this method must be rinsed first with the acetic acid (4.6), then with water and finally dried.

5.1 Quartz conical flasks, of capacity 100 ml, with quartz or polypropylene covers.

5.2 Aluminium alloy block, recommended but not essential, which allows a temperature of 290 °C to be achieved and sustained throughout the fuming period. The block (see Figure A.1, Figure A.2 and Figure A.3) has holes designed to fit the 100 ml quartz flasks exactly and is heated by surface contact with a hotplate which enables the temperature of the aluminium block to be controlled up to about 320 °C.

5.3 Plastics volumetric flasks, of capacity 50 ml and 100 ml.

5.4 Plastics flasks, of capacity 100 ml, 500 ml and 1 000 ml.