BRITISH STANDARD

BS 7455 :
Part 5 : 1991
ISO 7530-5 :
1990

Analysis of nickel alloys by flame atomic absorption spectrometry

Part 5. Method for the determination of iron

BSI
STANDARDS

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Non-ferrous Metals Standards Policy Committee (NFM/-) to Technical Committee NFM/10, upon which the following bodies were represented:

British Non-ferrous Metals Federation
British Steel Industry
Department of Trade and Industry (Laboratory of the Government Chemist)
Engineering Equipment and Materials Users' Association
Ministry of Defence
Nickel Development Institute
Non-ferrous Metal Stockists
Process Plant Association
Stainless Steel Fabricators' Association of Great Britain
Coopted members

This British Standard, having been prepared under the direction of the Non-ferrous Metals Standards Policy Committee, was published under the authority of the Standards Board and comes into effect on 28 June 1991

The following BSI references relate to the work on this standard:
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Contents

National foreword

This Part of BS 7455 has been prepared under the direction of the Non-ferrous Metals Standards Policy Committee. It is identical with ISO 7530-5 'Nickel alloys — Flame atomic absorption spectrometric analysis — Part 5 : 1990 Determination of iron content', published by the International Organization for Standardization (ISO).

At present this British Standard consists of six Parts all concerned with methods for flame spectrometric analysis of nickel alloys. Further international standards are in preparation on the same subject and when available, these will be published as further Parts of this British Standard.

Cross-references

International standard	Corresponding British Standard
ISO 5725 : 1986	BS 5497 Precision of test methods Part 1 : 1987 Guide for the determination of repeatability and reproducibility for a standard test method by inter-laboratory tests (Identical)
ISO 7530-1 : 1990	BS 7455 Analysis of nickel alloys by flame atomic absorption spectrometry Part 1 : 1991 General requirements and sample dissolution (Identical)

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

Nickel alloys — Flame atomic absorption spectrometric analysis —

Part 5:
Determination of iron content

1 Scope

This part of ISO 7530 specifies a flame atomic absorption spectrometric method for the determination of iron in the range of 0,01 % (*m/m*) to 4 % (*m/m*) in nickel alloys. Typical compositions of some nickel alloys are given in ISO 7530-1, annex B.

The general requirements concerning the apparatus, sampling, dissolution of the test sample, atomic absorption measurements, calculations and test report are given in ISO 7530-1.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 7530. 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 7530 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 7530-1:—[1], *Nickel alloys — Flame atomic absorption spectrometric analysis — Part 1: General requirements and sample dissolution.*

1) To be published.

3 Principle

Dissolution of a test portion in acid and aspiration of the test solution into an air-acetylene flame of an atomic absorption spectrometer.

Measurement of the absorbance of the resonance line energy from the spectrum of iron and comparison with that of calibration solutions at a wavelength of 248,3 nm.

4 Reagents

In addition to the reagents listed in ISO 7530-1, the following special reagents are required.

4.1 Strontium chloride, solution.

Transfer 113,5 g of strontium chloride hexahydrate ($SrCl_2 \cdot 6H_2O$) to a 600 ml beaker, dissolve in 400 ml of hot water (50 °C to 60 °C), cool and transfer to a 1 000 ml one-mark volumetric flask. Make up to the mark with water and mix. The strontium chloride should be free of heavy metals.

4.2 Iron, standard reference solution (1,000 g/l).

Weigh, to the nearest 0,001 g, 1,000 g of iron metal of 99,9 % (*m/m*) minimum purity and transfer to a 400 ml beaker. Add 30 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml) diluted 1 + 1. Heat to initiate the reaction and complete dissolution. Cool to about 50 °C, cautiously add 1 ml of hydrogen peroxide (4.4) and bring to the boil to oxidize the iron. Cool, transfer to a 1 000 ml one-mark volumetric flask and add 35 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml).

Make up to the mark with water, mix and store in a polyethylene bottle.

4.3 Iron, standard solution (50 mg/l).

Pipette 50 ml of the iron standard reference solution (4.2) into a 1000 ml one-mark volumetric flask and add 50 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml). Make up to the mark with water, mix and store in a polyethylene bottle.

4.4 Hydrogen peroxide, 300 g/l, (ρ_{20} = 1,1 g/ml).

5 Apparatus

The apparatus required is specified in clause 5 of ISO 7530-1.

6 Sampling and sample preparation

Refer to clause 6 of ISO 7530-1.

7 Procedure

7.1 Preparation of test solution

Proceed as directed in 7.1.1 to 7.1.4 of ISO 7530-1. Add 0,25 ml of hydrogen peroxide (4.4) in the final dissolution of the salts.

7.1.1 Primary dilutions

7.1.1.1 Initial dilution for 0,01 % (m/m) to 0,10 % (m/m) iron

Transfer the test solution (7.1) to a 100 ml one-mark volumetric flask. Add 4 ml of strontium chloride solution (4.1). Make up to the mark with water and mix. Remove any products of hydrolysis by settlement and dry filtration or by centrifuging.

7.1.1.2 Initial dilution for 0,1 % (m/m) to 4,0 % (m/m) iron

Transfer the test solution (7.1) to a 500 ml one-mark volumetric flask. Add 20 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml). Make up to the mark with water and mix. Remove any products of hydrolysis by settlement and dry filtration or by centrifuging.

7.1.2 Secondary dilutions

7.1.2.1 Secondary dilution for 0,1 % (m/m) to 0,8 % (m/m) iron

Pipette 50 ml of the solution from 7.1.1.2 into a 100 ml one-mark volumetric flask. Add 4 ml of strontium chloride solution (4.1) and 3 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml). Make up to the mark with water and mix.

7.1.2.2 Secondary dilution for 0,4 % (m/m) to 4 % (m/m) iron

Pipette 10 ml of the solution from 7.1.1.2 into a 100 ml one-mark volumetric flask. Add 4 ml of strontium chloride solution (4.1) and 5 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml). Make up to the mark with water and mix.

7.2 Reagent blank solution

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 Iron calibration solutions

Using pipettes, transfer to each of five 100 ml one-mark volumetric flasks, 0 ml, 5 ml, 10 ml, 15 ml and 20 ml of iron standard solution (4.3). Add 4 ml of strontium chloride solution (4.1) and 5 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml). Make up to the mark with water and mix.

7.4 Calibration and determination

7.4.1 Atomic absorption measurements

Proceed as directed in 7.4.1 of ISO 7530-1, using a wavelength of 248,3 nm and an air-acetylene flame.

7.4.2 Preparation of calibration graphs

Proceed as directed in 7.4.2 of ISO 7530-1.

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 7530-1.

8.2 Precision

8.2.1 Laboratory tests

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

8.2.2 Statistical analysis

8.2.2.1 Results were treated according to ISO 5725 as described in 8.2.2 of ISO 7530-1. The results of this analysis are given in table 2.

8.2.2.2 One laboratory was rejected as a Cochran outlier for sample 3920. One laboratory was rejected as both a Cochran and a Dixon outlier for sample 7013.

9 Test report

Refer to clause 9 of ISO 7530-1.

Table 1 — Nominal composition of test samples [% (*m/m*)]

Sample	Al	Co	Cr	Cu	Fe	Mn	Ni	Si	Ti
3920	0,15	2	19	0,1	3	0,3	Remainder	0,6	2,3
7013	1,5	17	20	0,2	0,2	0,05	Remainder	0,7	2,4

Table 2 — Results of statistical analysis

Sample reference	Mean % (*m/m*)	Within-laboratory standard deviation	Between laboratory standard deviation	Repeatability	Reproducibility
3920	3,00	0,029	0,048	0,083	0,16
7013	0,21	0,002 3	0,006 3	0,007	0,019

Publication(s) referred to

See national foreword.

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