INTERNATIONAL
STANDARD

ISO
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Nickel alloys — Flame atomic absorption spectrometric analysis —

Part 8:
Determination of silicon content

Alliages de nickel — Analyse par spectrométrie d'absorption atomique dans la flamme —

Partie 8: Dosage du silicium

Reference number
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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 7530-8 was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys*, Sub-Committee SC 4, *Analysis of nickel alloys*.

ISO 7530 consists of the following parts, under the general title *Nickel alloys — Flame atomic absorption spectrometric analysis*:

- *Part 1: General requirements and sample dissolution*
- *Part 2: Determination of cobalt content*
- *Part 3: Determination of chromium content*
- *Part 4: Determination of copper content*
- *Part 5: Determination of iron content*
- *Part 6: Determination of manganese content*
- *Part 7: Determination of aluminium content*
- *Part 8: Determination of silicon content*
- *Part 9: Determination of vanadium content*

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Nickel alloys — Flame atomic absorption spectrometric analysis —

Part 8:
Determination of silicon content

1 Scope

This part of ISO 7530 specifies a flame atomic absorption spectrometric method for the determination of silicon in the range of 0,2 % (*m*/*m*) to 1 % (*m*/*m*) in nickel alloys. Typical compositions of nickel alloys are given in annex B of ISO 7530-1:1990.

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 7530-1:1990, *Nickel alloys — Flame atomic absorption spectrometric analysis — Part 1: General requirements and sample dissolution.*

3 Principle

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

Measurement of the absorbance of the resonance line energy from the spectrum of silicon and comparison with that of calibration solutions at a wavelength of 251,6 nm.

4 Reagents

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

4.1 Hydrofluoric acid, $\rho_{20} = 1{,}15$ g/ml.

WARNING — Hydrofluoric acid is extremely irritating and corrosive to skin and mucous membranes, producing severe skin burns which are slow to heal. In the case of contact with skin, wash well with water and seek medical advice.

4.2 Hydrofluoric acid, $\rho_{20} = 1{,}15$ g/ml, diluted 1 + 9.

4.3 Lithium chloride (LiCl), solution.

Transfer 25 g of lithium chloride to a 250 ml beaker and dissolve in 150 ml of warm water. Cool, transfer to a 200 ml one-mark volumetric flask and make up to the mark with water. Store in a plastics bottle.

4.4 Silicon, standard reference solution (1,000 g/l).

Weigh, to the nearest 0,001 g, 1,000 g of elemental silicon powder of 99,9 % (*m*/*m*) minimum purity and transfer to a 250 ml polytetrafluoroethylene beaker. Add 20 ml of nitric acid ($\rho_{20} = 1{,}41$ g/ml) and wash the beaker walls with water. Add hydrofluoric acid (4.1) drop by drop to initiate and sustain the reaction (approximately 10 ml of hydrofluoric acid are required). After most of the silicon has dissolved, add

10 ml more hydrofluoric acid, cover the beaker and keep at less than 50 °C until dissolution is complete. Transfer to a 1 000 ml plastics one-mark volumetric flask, add 20 ml of hydrochloric acid ($\rho_{20} = 1,18$ g/ml) and make up to the mark with water. Store in a polyethylene bottle.

4.5 Silicon, standard solution (100 mg/l).

Transfer, using a plastics pipette, 50,0 ml of the silicon standard reference solution (4.4) into a 500 ml plastics one-mark volumetric flask. Add 5 ml of the dilute hydrofluoric acid (4.2) and 10 ml of hydrochloric acid ($\rho_{20} = 1,18$ g/ml). Dilute to the mark with water, mix and store in a polyethylene bottle.

5 Apparatus

IMPORTANT — Plastics beakers and volumetric ware shall be used throughout the procedure.

In addition to the apparatus specified in clause 5 of ISO 7530-1:1990, the following plastics laboratory items are required.

5.1 Plastics beakers, of capacity 250 ml, preferably of polytetrafluoroethylene.

5.2 Acrylic body burette, 50 ml graduated in 0,1 ml divisions.

5.3 Polypropylene pipettes, 10 ml, 25 ml and 50 ml.

5.4 Polypropylene or polymethylpentene one-mark volumetric flasks, of capacity 100 ml, 500 ml or 1 000 ml.

6 Sampling and sample preparation

Refer to clause 6 of ISO 7530-1:1990.

7 Procedure

7.1 Preparation of test solution

7.1.1 Dissolution of a test portion in acid

Weigh, to the nearest 0,001 g, 1,00 g of the test sample and transfer it to a clean plastics beaker (5.1). Add 20 ml of a mixture of one part of nitric acid ($\rho_{20} = 1,41$ g/ml) and three parts of hydrochloric acid ($\rho_{20} = 1,18$ g/ml). Apply sufficient heat to initiate and maintain the reaction until dissolution is complete. If the alloy resists dissolution, add hydrochloric acid ($\rho_{20} = 1,18$ g/ml) in 1 ml increments and continue heating to dissolve the sample.

7.1.2 Preparation of the final test solution

Cool the solution and wash the cover and beaker walls with a minimum of water. Add 5 ml of the dilute hydrofluoric acid (4.2) and allow to stand for 1 h, swirling intermittently.

7.1.3 Primary dilutions

7.1.3.1 Initial dilution for 0,2 % (*m*/*m*) to 0,25 % (*m*/*m*) silicon

Transfer the test solution from 7.1.2 to a 100 ml plastics one-mark volumetric flask, add 2 ml of hydrochloric acid ($\rho_{20} = 1,18$ g/ml) and dilute to approximately 80 ml with water. Add 3 ml of the lithium chloride solution (4.3) and make up to the mark with water.

7.1.3.2 Initial dilution for 0,5 % (*m*/*m*) to 1,0 % (*m*/*m*) silicon

Transfer the test solution from 7.1.2 to a 100 ml plastics one-mark volumetric flask and make up to the mark with water.

7.1.4 Secondary dilution for 0,5 % (*m*/*m*) to 1,0 % (*m*/*m*) silicon

Pipette 50,0 ml of the solution from 7.1.3.2 into a 100 ml plastics one-mark volumetric flask. Add 2 ml of hydrochloric acid ($\rho_{20} = 1,18$ g/ml) and 2,5 ml of the dilute hydrofluoric acid (4.2). Dilute to approximately 80 ml with water and mix. Add 3 ml of the lithium chloride solution (4.3) and make up to the mark with water.

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 Silicon calibration solutions

Using the plastics burette (5.2), transfer to each of six 100 ml plastics one-mark volumetric flasks, 0 ml, 10,0 ml, 20,0 ml, 30,0 ml, 40,0 ml and 50,0 ml of the silicon standard solution (4.5). Add 2 ml of hydrochloric acid ($\rho_{20} = 1,18$ g/ml) and 5 ml of the dilute hydrofluoric acid (4.2). Dilute to approximately 80 ml with water and mix. Add 3 ml of the lithium chloride solution (4.3) and make up to the mark with water. These calibration solutions correspond to 0 mg/l 10 mg/l, 20 mg/l, 30 mg/l, 40 mg/l and 50 mg/l of silicon.

7.4 Calibration and determination

7.4.1 Atomic absorption measurements

Proceed as directed in 7.4.1 of ISO 7530-1:1990, using a wavelength of 251,6 nm and a nitrous oxide-acetylene flame.

NOTE 1 To eliminate silica memory effects, the burner system must be preconditioned before analysis by aspirating a dilute solution of hydrofluoric acid [10 ml of dilute hydrofluoric acid (4.2) and 90 ml of water]. With the flame burning, aspirate this dilute acid solution until the original baseline signal is restored, i.e. when the silica deposit on the burner top has been volatilized. Then proceed with the aspiration of distilled water as directed.

7.4.2 Preparation of calibration curves

Proceed as directed in 7.4.2 of ISO 7530-1:1990.

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 7530-1:1990.

8.2 Precision

8.2.1 Laboratory tests

Six laboratories in four countries participated in the testing of this procedure using six 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 7530-1:1990. The results of this analysis are given in table 2.

8.2.2.2 No outliers were identified by statistical tests.

9 Test report

Refer to clause 9 of ISO 7530-1:1990.

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

Sample	Al	Co	Cr	Cu	Fe	Mn	Ni	Si	Ti
825	0,2	0,07	21	1,6	30	0,7	Bal	0,4	1,1
902	0,4	0,05	5	0,04	48	0,4	Bal	0,35	2,5
3920	0,15	2	19	0,1	3	0,3	Bal	0,6	2,3
3927	0,1	1	20	0,05	44	0,4	Bal	0,8	0,6
7013	1,5	17	20	0,2	0,2	0,05	Bal	0,7	2,4
7049	1	0,01	15	0,15	7	0,8	Bal	0,3	2,3

Table 2 — Results of statistical analysis

Sample reference	Mean % (*m*/*m*)	Within-laboratory standard deviation	Between-laboratory standard deviation	Repeatability	Reproducibility
825	0,403	0,006 6	0,023 6	0,018 5	0,069 4
902	0,344	0,003 9	0,014 9	0,011 0	0,043 5
3920	0,614	0,016 6	0,017 5	0,047 0	0,068 2
3927	0,816	0,019 5	0,040 3	0,055 1	0,127
7013	0,721	0,017 4	0,021 3	0,049 1	0,077 8
7049	0,336	0,006 6	0,014 4	0,018 7	0,044 8

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