INTERNATIONAL STANDARD

ISO 7530-3

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Nickel alloys — Flame atomic absorption spectrometric analysis —

Part 3:
Determination of chromium content

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

Partie 3: Dosage du chrome

Reference number
ISO 7530-3:1990(E)

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 7530-3 was prepared by Technical Committee ISO/TC 155, *Nickel and 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 3:
Determination of chromium content

1 Scope

This part of ISO 7530 specifies a flame atomic absorption spectrometric method for the determination of chromium 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 a nitrous oxide-acetylene flame of an atomic absorption spectrometer.

Measurement of the absorbance of the resonance line energy from the spectrum of chromium and comparison with that of calibration solutions at a wavelength of 357,9 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 Chromium, standard reference solution (1,000 g/l).

Weigh, to the nearest 0,001 g, 1,000 g of chromium 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 and heat to complete dissolution. 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 Chromium, standard solution (50 mg/l).

Pipette 50 ml of the chromium 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.

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.

7.1.1 Primary dilutions

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

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*) chromium

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*) chromium

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*) chromium

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 Chromium 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 chromium 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 357,9 nm and a nitrous oxide-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

Ten laboratories in five countries participated in the testing of this procedure using one sample 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 and one was rejected as a Dixon outlier.

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
902	0,4	0,05	5	0,04	48	0,4	Remainder	0,35	2,5

Table 2 — Results of statistical analysis

Sample reference	Mean % (*m/m*)	Within-laboratory standard deviation	Between laboratory standard deviation	Repeatability	Reproducibility
902	5,16	0,034	0,102	0,096	0,30

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Descriptors: nickel alloys, chemical analysis, determination of content, chromium, atomic absorption spectrometric method.

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