SLOVENSKI STANDARD
SIST ISO 7530-9:1996

01-avgust-1996

Nikljeve zlitine - Plamenska atomska absorpcijska spektrometrična analiza - 9. del: Ugotavljanje deleža vanadija

Nickel alloys -- Flame atomic absorption spectrometric analysis -- Part 9: Determination of vanadium content

Alliages de nickel -- Analyse par spectrométrie d'absorption atomique dans la flamme -- Partie 9: Dosage du vanadium

Ta slovenski standard je istoveten z: **ISO 7530-9:1993**

ICS:

77.120.40	Nikelj, krom in njune zlitine	Nickel, chromium and their alloys

SIST ISO 7530-9:1996 **en**

INTERNATIONAL STANDARD

ISO 7530-9

First edition
1993-08-15

Nickel alloys — Flame atomic absorption spectrometric analysis —

Part 9:

Determination of vanadium content

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

Partie 9: Dosage du vanadium

Reference number
ISO 7530-9:1993(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 7530-9 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*

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Nickel alloys — Flame atomic absorption spectrometric analysis —

Part 9:
Determination of vanadium content

1 Scope

This part of ISO 7530 specifies a flame atomic absorption spectrometric method for the determination of vanadium in the range of 0,05 % (*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 vanadium and comparison with that of calibration solutions at a wavelength of 318,4 nm.

4 Reagents

In addition to the reagents listed in clause 4 of ISO 7530-1:1990, the following special reagents are required.

4.1 Ammonium metavanadate (NH_4VO_3), an alternative to pure vanadium metal.

4.2 Strontium chloride, solution.

Transfer 113,5 g of strontium chloride hexahydrate ($SrCl_2.6H_2O$) 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.

4.3 Vanadium, standard reference solution (1,000 g/l).

4.3.1 Preparation from vanadium metal

Weigh, to the nearest 0,001 g, 1,000 g of vanadium metal of 99,9 % (*m/m*) minimum purity and transfer to a 400 ml beaker. Add 60 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml) and 20 ml of nitric acid (ρ_{20} = 1,41 g/ml) and heat to complete dissolution. Cool and transfer to a 1 000 ml one-mark volumetric flask. Make up to the mark with water, mix and store in a polyethylene bottle.

4.3.2 Preparation from ammonium metavanadate

Weigh 2,296 g of ammonium metavanadate (4.1) and transfer to a 600 ml beaker. Add about 400 ml of water and heat to dissolve the salt. Transfer the warm solution to a 1 000 ml one-mark volumetric flask and dilute with 400 ml of cold water. Add 50 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml) and 10 ml of nitric acid (ρ_{20} = 1,41 g/ml), and cool to ambient temperature. Make up to the mark with water, mix and store in a polyethylene bottle.

4.4 Vanadium, standard solution (250 mg/l).

Pipette 50 ml of the vanadium standard reference solution (4.3) into a 200 ml one-mark volumetric flask. 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 7530-1:1990.

6 Sampling and sample preparation

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

7 Procedure

7.1 Preparation of test solution

Proceed as directed in 7.1.1 to 7.1.4 of ISO 7530-1:1990, using 3 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml) and 1 ml of nitric acid (ρ_{20} = 1,41 g/ml) instead of 5 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml) to dissolve the salts.

7.1.1 Primary dilution for 0,05 % (*m/m*) to 0,35 % (*m/m*) vanadium

Transfer the test solution (7.1) to a 100 ml one-mark volumetric flask. Add 5 ml of the strontium chloride solution (4.2), 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 dilution for 0,35 % (*m/m*) to 1,0 % (*m/m*) vanadium

Pipette 20,0 ml of the solution from 7.1.1 into a 100 ml one-mark volumetric flask. Add 4 ml of the strontium chloride solution (4.2), 3 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml) and 1 ml of nitric acid (ρ_{20} = 1,41 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 Vanadium calibration solutions

Using a burette, transfer to each of five 100 ml one-mark volumetric flasks, 0 ml, 4,0 ml, 8,0 ml, 12,0 ml and 16,0 ml of the vanadium standard solution (4.4). Add 5 ml of the strontium chloride solution (4.2), 3 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml) and 1 ml of nitric acid (ρ_{20} = 1,41 g/ml). Make up to the mark with water and mix. These calibration solutions correspond to 0 mg/l, 10 mg/l, 20 mg/l, 30 mg/l, and 40 mg/l of vanadium.

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 318,4 nm and a fuel-rich nitrous oxide-acetylene flame.

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 the IN100 sample. Nine laboratories in five countries analysed sample NPK31 and sample 925. The nominal composition of the samples is 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 One laboratory was rejected as a Cochran outlier for sample 925.

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	Fe	Mo	Ni	Nb	Ti	V	Zr
IN100	5,5	15	10	< 0,5	3	Remainder	—	5	1	0,05
NPK31	0,5	14	20	1	4,5	Remainder	5	2	0,3	—
925	0,3	0,2	21	27	3	Remainder	0,4	2	0,05	—

Table 2 — Results of statistical analysis

Sample reference	Mean % (*m/m*)	Within-laboratory standard deviation	Between-laboratory standard deviation	Repeatability	Reproducibility
IN100	0,965	0,005 8	0,035 7	0,016 5	0,101 7
NPK31	0,286	0,008 7	0,026 2	0,024 7	0,076 1
925	0,042	0,001 2	0,005 9	0,003 4	0,017 0