INTERNATIONAL STANDARD

ISO 7530-1

First edition
1990-12-15

Corrected and reprinted
1992-02-15

Nickel alloys — Flame atomic absorption spectrometric analysis —

Part 1:
General requirements and sample dissolution

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

Partie 1: Caractéristiques générales et mise en solution de l'échantillon

Reference number
ISO 7530-1:1990(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 7530-1 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*

Annex A forms an integral part of this part of ISO 7530. Annex B is for information only.

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

Printed in Switzerland

Introduction

This part of ISO 7530 is to be used in conjunction with the other parts which specify methods for the determination of individual elements in nickel alloys by flame atomic absorption spectrometry.

Although the analytical methods are specified in independent International Standards, it is possible to determine more than one element on a single test solution by adjustment of the sample weight and initial and subsequent dilutions.

Nickel alloys — Flame atomic absorption spectrometric analysis —

Part 1:
General requirements and sample dissolution

WARNING — The manufacturer's recommendations should be closely followed and particular attention is drawn to the following safety points:

a) the explosive nature of acetylene, and regulations concerning its use,

b) the need to shield the eyes of the operator from ultraviolet radiation by means of tinted glass,

c) the need to keep the burner head clear of deposits because a badly clogged burner may cause a flashback,

d) the need to ensure that the liquid trap is filled with water,

e) always spray distilled water between the test solutions, blank solution and/or calibration solutions.

1 Scope

1.1 ISO 7530 specifies flame atomic absorption methods for the determination of up to 4 % (*m/m*) of cobalt, chromium, copper, iron, manganese and aluminium, up to 2 % (*m/m*) of silicon and from 0,05 % (*m/m*) to 1 % (*m/m*) of vanadium in nickel alloys. Other elements may be added in subsequent parts of ISO 7530. Typical compositions of some nickel alloys are given in annex B.

1.2 This part of ISO 7530 specifies the general requirements for analysis by flame atomic absorption, preparation and dissolution of the test sample, method of calculation and the procedures used for the evaluation of the repeatability and reproducibility of the individual methods specified in the other parts of ISO 7530.

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 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 648:1977, *Laboratory glassware — One-mark pipettes.*

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks.*

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 acid, evaporation of excess acid and redissolution of the salts.

Addition of an ionization suppressant, if necessary, and dilution of the solution to a known volume.

Aspiration of the test solution, after a secondary dilution, if necessary, into the air-acetylene or nitrous oxide-acetylene flame of an atomic absorption spectrometer.

Measurement of the absorption of the resonance line energy from the spectrum of the element being determined and comparison with that of calibration solutions of the same element.

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 High purity metals, 99,9 % (m/m) minimum, as specified in the relevant part of ISO 7530.

4.2 Nitric acid, (HNO_3), ρ_{20} = 1,41 g/ml.

4.3 Nitric acid, (HNO_3), ρ_{20} = 1,41 g/ml diluted (1 + 1).

4.4 Hydrochloric acid, (HCl), ρ_{20} = 1,18 g/ml.

4.5 Hydrochloric acid, (HCl), ρ_{20} = 1,18 g/ml diluted (1 + 1).

4.6 Nitric acid-hydrochloric acid mixture.

CAUTION — This acid mixture is highly corrosive and unstable. Noxious gas (chlorine) is liberated on standing. It shall be prepared and used in a fume cupboard and shall not be kept in a closed container.

Carefully mix 1 part of nitric acid (4.2) and 3 parts of hydrochloric acid (4.4). This mixture is not stable and should be prepared only as needed.

4.7 Standard reference solutions, 1,000 g/l of metal.

Prepare separately for each metal as specified in the appropriate part of ISO 7530.

5 Apparatus

Ordinary laboratory apparatus, and

5.1 Atomic absorption spectrometer

5.1.1 The atomic absorption spectrometer used in this method shall meet the instrument performance parameters given in annex A.

5.1.2 The instrument shall be equipped with burner heads suitable for both an air-acetylene and a nitrous oxide-acetylene flame.

5.1.3 The instrument should be capable of using single element hollow cathode or electrodeless discharge lamps operated at currents recommended by the manufacturer.

5.2 Volumetric glassware

5.2.1 Burettes, of capacity 50 ml, graduated in divisions of 0,1 ml in accordance with ISO 385-1, class A.

5.2.2 Pipettes, in accordance with ISO 648, class A.

5.2.3 Volumetric flasks, in accordance with ISO 1042, class A.

6 Sampling and sample preparation

6.1 Sampling and preparation of the laboratory sample shall be carried out by normal agreed procedures or, in case of dispute, by the relevant International Standard.

6.2 The laboratory sample normally is in the form of millings or drillings and no further preparation of the sample is necessary.

6.3 If it is suspected that the laboratory sample is contaminated with oil or grease from the milling or drilling process, it shall be cleaned by washing with high purity acetone and drying in air.

6.4 If the laboratory sample contains particles or pieces of widely varying sizes, the test sample should be obtained by riffling.

7 Procedure

7.1 Preparation of test solution — General method

7.1.1 Weigh, to the nearest 0,001 g, 1,00 g of the laboratory sample and transfer to a clean unetched 600 ml beaker. Add 20 ml of the nitric acid-hydrochloric acid mixture (4.6). Apply sufficient heat to initiate and maintain the reaction until dissolution is complete. If the alloy resists dissolution, some adjustment of the acid mixture may be required. Add hydrochloric acid (4.4) in 1 ml increments and continue heating to dissolve the sample.

7.1.2 Using a low heat, evaporate the solution just to dryness. Do not bake. Cool to about 50 °C, add 25 ml of hydrochloric acid (4.4) and again evaporate just to dryness. Add a further 25 ml of hydrochloric acid and repeat the evaporation.

7.1.3 Cool to about 50 °C, add 5 ml of hydrochloric acid (4.4) and 20 ml of water and heat to dissolve the salts.

7.1.4 Proceed as directed in the relevant part of ISO 7530.

NOTES

1 Some alloys having a high copper content may be dissolved in nitric acid diluted 1 + 1. For some alloys an acid mixture containing 30 ml hydrochloric acid and 2 ml of nitric acid is more effective.

2 The general method of dissolution may be modified as specified in other parts of ISO 7530.

3 If sample inhomogeneity is suspected, a larger mass of sample (10 g to 50 g) may be taken for analysis. However, an aliquot portion corresponding to a 1 g sample shall be taken from such a solution and processed in accordance with the procedure given.

7.2 Blank test

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 Preparation of calibration solutions

Proceed as directed in the relevant part of ISO 7530.

7.4 Calibration and determination

7.4.1 Atomic absorption measurements

7.4.1.1 The spectral lines for each element to be used in the analysis are specified in the relevant part of ISO 7530.

7.4.1.2 Set the required instrument parameters according to the manufacturer's recommendations. Light the burner and aspirate water until thermal equilibrium is reached. The flame conditions will vary according to the element being determined. Zero the instrument.

7.4.1.3 Ensure that the instrument meets the performance requirements given in annex A. Optimum settings for the operating parameters vary from instrument to instrument. Scale expansion may have to be used to obtain the required readability.

7.4.1.4 Ensure that the calibration solutions and the test solution(s) are within 1 °C of the same temperature.

7.4.1.5 Aspirate water and zero the instrument.

7.4.1.6 Aspirate the calibration solutions and the test solution(s) and note the reading to determine the approximate concentration of the test solutions.

7.4.1.7 Aspirate water until the initial reading is obtained. Zero the instrument if necessary.

7.4.1.8 Aspirate the calibration solutions and the test solution(s) in the order of increasing instrument response, starting with the zero member solution. When a stable response is obtained, record the reading. Flush the system by aspirating water between each test or calibration solution.

7.4.1.9 Repeat the measurement of the full set of the calibration and test solutions twice more and record the data.

7.4.2 Preparation of calibration graphs

Plot the average instrument reading against the concentration of the analyte in the calibration solutions for each set of measurements. Proceed with the calculations as directed in clause 8.

NOTE 4 Some instruments may be adjusted to give a read-out directly in concentration of the analyte. A graph of instrument response versus concentration should be plotted to check the validity of the readings.

7.5 Number of determinations

Carry out the determination at least in duplicate.

8 Expression of results

8.1 Calculation

8.1.1 Determine the concentration of the analyte in the test solution from the corresponding calibration graphs for each of the three sets of instrument readings recorded.

8.1.2 Calculate the percentage by mass of the analyte in the test sample using the formula

$$\frac{\rho \times V \times F}{m} \times 10^{-4}$$

where

ρ is the analyte concentration, in milligrams per litre, found in the test solution less the blank;

V is the volume, in millilitres, of the initial test solution;

F is the dilution factor for the secondary dilution;

m is the mass, in grams, of the test portion.

NOTE 5 The average of the results from the three readings as calculated comprises a single determination. The three results give an indication of the precision of the atomic absorption measurements.

8.2 Precision

8.2.1 Laboratory tests

The methods in the subsequent parts of ISO 7530 have been subjected to interlaboratory testing. Samples were analysed in duplicate on different days.

8.2.2 Statistical analysis

8.2.2.1 Results from the interlaboratory test programme were evaluated according to ISO 5725. The data were tested for statistical outliers by the Cochran and Dixon tests given in ISO 5725.

8.2.2.2 The principle of the Cochran test is that a set of results is an outlier if the within-laboratory variance is too large in relation to others. Dixon's test is to determine if the mean from a laboratory is too far from the other laboratory means. Both tests were applied at the 95 % confidence level.

8.2.2.3 Repeatability and reproducibility were calculated according to ISO 5725 at the 95 % confidence level. Results of the statistical analysis, including the within-laboratory and between-laboratory standard deviations are given for each element in the relevant part of this ISO 7530.

9 Test report

The test report shall include the following information:

a) the reference to the method used;

b) the results of the analysis;

c) the number of independent replications;

d) any unusual features noted during the analysis;

e) any operation not included in this part of ISO 7530 or regarded as optional.

Annex A
(normative)

Checking of spectrometer performance parameters

A.1 Introduction

The performance of atomic absorption spectrometers of the same or different manufacture may vary from instrument to instrument. It is therefore essential to establish that a particular instrument meets certain performance requirements before it is used in the methods specified in ISO 7530.

A.2 Initial instrument adjustments

A.2.1 Set up the atomic absorption spectrometer to operate with an air-acetylene or nitrous oxide-acetylene flame using a single slot laminar flow burner head according to the manufacturer's instructions.

A.2.2 Use a single element hollow cathode, single element electrodeless discharge or other appropriate single element lamp as the light source for the element under test. Operate the source as recommended by the manufacturer.

NOTE 8 The use of multi-element lamps is not generally recommended although some binary alloy lamps give a more stable emission than single element lamps.

A.2.3 Light the burner and aspirate water until thermal equilibrium is reached.

A.2.4 Aspirate a mid-range calibration solution of the element under test and adjust the instrument to give optimum absorption. Use the wavelengths specified in the relevant part of ISO 7530 and the slit setting or bandpass recommended by the instrument manufacturer for the element under test. The use of scale expansion may be necessary.

A.2.5 Flush the burner system by aspirating hydrochloric acid diluted 1 + 19, adjust the instrument zero and proceed with the performance parameter check as directed in A.3.2 to A.3.4 inclusive.

A.3 Instrument performance check

A.3.1 Performance check solutions

The calibration graph in the relevant part of ISO 7530 is normally established using five calibration solutions including the zero member. For the instrument performance check, select two pairs of calibration solutions covering the upper and lower end of the calibration graph, such that the interval between the two calibration solutions of highest concentration is equal to that between the reference solution and the calibration solution of low concentration.

A.3.2 Readability

A.3.2.1 Aspirate the two calibration solutions of highest concentrations of the element under test, record the instrument readings and calculate the difference.

A.3.2.2 Divide the difference in the readings by 20. The readability of the instrument is acceptable if this result is not less than the smallest effective interval which can be read or estimated on the instrument read-out.

A.3.3 Linearity of instrument response

A.3.3.1 Aspirate the zero member solution and the calibration solution of low concentration of the element under test (A.3.1). Record the instrument readings and calculate the difference.

A.3.3.2 Divide the difference in the readings for the two calibration solutions of highest concentration as determined in A.3.2.1 by the difference between the zero member and calibration solution of low concentration.

A.3.3.3 The linearity of the instrument response is acceptable if this ratio is 0,70 or greater.

A.3.3.4 If the ratio is less than 0,70, further adjustments to the instrument may give acceptable results. Otherwise the operating range of the method shall be reduced by lowering the concentration of the calibration solution of highest concentration.

A.3.4 Minimum stability

A.3.4.1 Aspirate hydrochloric acid diluted 1 + 19 and zero the instrument.

A.3.4.2 Aspirate the calibration solution of highest concentration and record the reading.

A.3.4.3 Aspirate hydrochloric acid diluted 1 + 19.

NOTE 7 The instrument reading should return to zero.

A.3.4.4 Repeat the measurement of the calibration solution of highest concentration six times, aspirating hydrochloric acid diluted 1 + 19 between readings but not adjusting any instrumental settings.

A.3.4.5 The variability (VA), expressed as a percentage, of the readings of the calibration solution of highest concentration, is given by the formula

$$\frac{(A_h - A_l)0{,}40}{\overline{A}} \times 100$$

where

$\overline{A}$ is the average instrument reading for the calibration solution of highest concentration calculated from the six readings taken;

A_h is the highest of the six instrument readings;

A_l is the lowest of the six instrument readings;

NOTE 8 $(A_h - A_l)0{,}40$ is an estimate of the standard deviation

A.3.4.6 The instrument meets the minimum stability requirements if the variability is less than 1,5 %.

NOTE 9 This test may, in addition, be applied to other points on the calibration graph. It may also be applied to evaluation of the minimum stability of the instrument zero.

Annex B
(informative)

Examples of compositions of some nickel alloys [% (*m*/*m*)]

The examples of compositions given in table B.1 are not to be interpreted as specifications for chemical compositions.

Table B.1 — Examples of compositions of some nickel alloys [% (*m*/*m*)]

Single values are maximum limits, except for nickel, where single values are minimum.

Alloy[1]	Al	B	C	Co[2]	Cr	Cu	Fe	Mn	Mo	Ni	P	S	Si	Ti	Others
A	—	—	0,30			28,0 34,0	2,5	2,0	—	63,0[3]	—	0,025	0,5	—	—
B	—	—	0,15	—	14,0 17,0	0,5	6,0 10,0	1,0	—	72,0[3]		0,015	0,5		
C	0,4 1,3		0,08		14,0 17,0	0,5	5,0 9,0	1,0	—	70,0[3]	—	0,015	0,5	2,2 2,8	Nb plus Ta 0.7 to 1,2
D	0,2 0,8	0,006	0,08		17,0 21,0	0,3	Remainder	0,4	2,8 3,3	50,0 55,0	0,015	0,015	0,4	0,6 1,2	Nb plus Ta 4,7 to 5,5
E	0,15 0,60	—	0,10 —	—	19,0 23,0	0,7	Remainder	1,5		30,0 35,0	—	0,015 —	1,0 —	0,15 0,60	— —
F	—	—	0,08 0,15	5,0	18,0 21,0	0,5	5,0	1,0		Remainder[3]		0,020	1,0	0,2 0,6	Pb 0,005
G	1,0 2,0	0,020	0,13	15,0 21,0	18,0 21,0	0.2	1,5	1,0	—	Remainder	—	0,015	1,0	2,0 3,0	Zr 0,15
H	4,5 4,9	0,003 0,010	0,12 0,17	18,0 22,0	14,0 15,7	0,2	1,0	1,0	4,5 5,5	Remainder	—	0,015	1,0	0,9 1,5	Zr 0,15
I	0,3 0,6	0,005	0,04 0,08	19,0 21,0	19.0 21,0	0,2	0,7	0,6	5,6 6,1	Remainder	—	0,007	0,4	1,9 2,4	Ti plus Al 2,4 to 2,8
J	—	—	0,02	1,0	1,0		2,0	1,0	26,0 30,0	Remainder[3]	0,040	0,035	0,1	—	—
K	1,2 1,6	0,003 0,010	0,02 0,10	12,0 15,0	18,0 21,0	0,1	2,0	1,0	3,5 5.0	Remainder	0,015	0,015	0,1	2,8 3,3	Zr 0,02 to 0,08
L	—	—	0,02	2,5	14.5 16.5	—	4,0 7,0	1,0	15,0 17,0	Remainder	0,040	0,035	0,08	—	V 0,35 W 3,0 to 4,5

[1] Alloy letters are used instead of commercial names until a neutral ISO designation is developed.

[2] Where no limits are given, the maximum cobalt content is 1,5 % (*m*/*m*).

[3] Cobalt counts as nickel in some alloys.

UDC 669.245:543.422

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

Price based on 7 pages