INTERNATIONAL STANDARD

ISO 7530-7

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Nickel alloys — Flame atomic absorption spectrometric analysis —

Part 7: Determination of aluminium content

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

Partie 7: Dosage de l'aluminium

INTERNATIONAL ISO

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Foreword

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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 7530-7 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 7:
Determination of aluminium content

1 Scope

This part of ISO 7530 specifies a flame atomic absorption spectrometric method for the determination of aluminium in the range of 0,2 % (*m*/*m*) to 4 % (*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, filtration and aspiration of the test solution into a nitrous oxide-acetylene flame of an atomic absorption spectrometer.

Combustion of the filter from the acid dissolution and volatilization of silica with hydrofluoric acid. Fusion of the residue with potassium pyrosulfate, dissolution of the melt in dilute acid and aspiration of this second 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 aluminim and comparison with that of calibration solutions at a wavelength of 309,3 nm.

Addition of the results found in both solutions.

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 Potassium pyrosulfate ($K_2S_2O_7$), powder.

4.3 Potassium chloride (KCl), solution.

Transfer 48 g of potassium chloride to a 600 ml beaker, dissolve in 500 ml of water and transfer to a 1 000 ml one-mark volumetric flask. Make up to the mark with water and mix.

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

Weigh, to the nearest 0,001 g, 1,000 g of aluminium metal of 99,9 % (*m*/*m*) minimum purity and transfer to a 400 ml beaker. Add a small drop of mercury, 30 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml) diluted 1 + 1 and heat to complete dissolution. Filter the solution through a 7 cm rapid filter paper into a 400 ml beaker. Wash the filter with 100 ml of warm water. Add 85 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml) to the filtrate, cool and transfer to a 1 000 ml one-mark volumetric flask. Make up to the mark with water and store in a polyethylene bottle.

WARNING — Mercury is highly poisonous and has an appreciable vapour pressure. It must be stored in strong, tightly closed containers. Liquid mercury must be transferred in such a manner that a spill can be contained and thoroughly cleaned up at once.

CAUTION — Discard the mercury in accordance with local regulations.

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

Pipette 100 ml of the aluminium standard reference solution (4.4) into a 1 000 ml one-mark volumetric flask and add 90 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml). Dilute to approximately 800 ml with water, cool, make up to the mark, mix and store in a polyethylene bottle.

5 Apparatus

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

5.1 Plastics beakers, of capacity 100 ml or 250 ml, preferably made of polytetrafluoroethylene.

5.2 Platinum crucible or dish.

6 Sampling and sample preparation

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

7 Procedure

7.1 Test portion and 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 1 part of nitric acid (ρ_{20} = 1,41 g/ml) and 3 parts of hydrochloric acid (ρ_{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 (ρ_{20} = 1,18 g/ml) in 1 ml increments and continue heating to dissolve the sample.

7.1.2 Filtration and processing of filtrate

Dilute the solution to 50 ml with water. Filter through an 11 cm, low ash medium filter paper into a 250 ml beaker. Wash the filter with approximately 50 ml of hot water in 10 ml portions. Process the filtrate as directed in 7.1.3.1 or 7.1.3.2, according to the estimated aluminium level, and process the filter as directed in 7.1.5.

7.1.3 Primary dilutions

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

Evaporate the filtrate from 7.1.2 to a volume of approximately 60 ml and cool to ambient temperature. Transfer the solution to a 100 ml one-mark volumetric flask and add 2,5 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml) and 4 ml of potassium chloride solution (4.3). Add 4 ml of nitric acid (ρ_{20} = 1,41 g/ml), cool and make up to the mark with water.

7.1.3.2 Initial dilution for 0,25 % (*m*/*m*) to 4,0 % (*m*/*m*) aluminium

Evaporate the filtrate from 7.1.2 to a volume of approximately 60 ml and cool to ambient temperature. Transfer the solution to a 100 ml one-mark volumetric flask. Add 2,5 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml) and make up to the mark with water.

7.1.4 Secondary dilutions

7.1.4.1 Secondary dilution for 0,25 % (*m*/*m*) to 1,0 % (*m*/*m*) aluminium

Pipette 20,0 ml of the solution from 7.1.3.2 into a 100 ml one-mark volumetric flask. Add 4 ml of the potassium chloride solution (4.3) and 8 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml). Dilute to 80 ml with water and add 4 ml of nitric acid (ρ_{20} = 1,41 g/ml). Cool and make up to the mark with water.

7.1.4.2 Secondary dilution for 1,0 % (*m*/*m*) to 2,0 % (*m*/*m*) aluminium

Pipette 10,0 ml of the solution from 7.1.3.2 into a 100 ml one-mark volumetric flask. Add 4 ml of the potassium chloride solution (4.3) and 9 ml of hydrochloric acid (ρ_{20} = 1,18 g/ml). Dilute to 80 ml with water and add 4 ml of nitric acid (ρ_{20} = 1,41 g/ml). Cool and make up to the mark with water.

7.1.4.3 Secondary dilution for 2,0 % (m/m) to 4,0 % (m/m) aluminium

Pipette 5,0 ml of the solution from 7.1.3.2 into a 100 ml one-mark volumetric flask. Add 4 ml of the potassium chloride solution (4.3) and 9,5 ml of hydrochloric acid ($\rho_{20} = 1{,}18$ g/ml). Dilute to 80 ml with water and add 4 ml of nitric acid ($\rho_{20} = 1{,}41$ g/ml). Cool and make up to the mark with water.

7.1.5 Processing the filter

7.1.5.1 Transfer the filter from 7.1.2 to a platinum crucible or dish (5.2). Dry, char and ignite to oxidize the carbon. Cool, add 0,25 ml of sulfuric acid ($\rho_{20} = 1{,}83$ g/ml) diluted 1 + 1 and 1 ml of the hydrofluoric acid (4.1). Carefully evaporate to dryness and fuse the residue with 1 g of potassium pyrosulfate (4.2). Allow the melt to cool and dissolve it in a small volume of water containing approximately 0,25 ml of hydrochloric acid ($\rho_{20} = 1{,}18$ g/ml). Heat, if necessary, to complete dissolution.

7.1.5.2 Transfer the leach solution to a 100 ml one-mark volumetric flask and add 10 ml of hydrochloric acid ($\rho_{20} = 1{,}18$ g/ml). Dilute with more water and add 4 ml of nitric acid ($\rho_{20} = 1{,}41$ g/ml). Cool and make up to the mark with water. Proceed as directed in 7.4.1.

NOTE 1 A very small amount of aluminium may be present in the fused residue but it usually does not exceed 0,5 mg. The solution is analysed separately and the aluminium found is added to the main result.

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 Aluminium calibration solutions

Using a burette, transfer to each of six 100 ml one-mark volumetric flasks, 0 ml, 5,0 ml, 10,0 ml, 15,0 ml, 20,0 ml and 25,0 ml of the aluminium standard solution (4.5). Add 4 ml of the potassium chloride solution (4.3) and 4 ml of nitric acid ($\rho_{20} = 1{,}41$ g/ml). Add sufficient hydrochloric acid ($\rho_{20} = 1{,}18$ g/ml) to make its concentration 10 % (V/V), cool and make up to the mark with water. These calibration solutions correspond to 0 mg/l, 5 mg/l, 10 mg/l, 15 mg/l, 20 mg/l and 25 mg/l of aluminium.

NOTE 2 It is important that all the calibration solutions contain the same concentration of hydrochloric acid. The zero solution requires an addition of 10 ml of hydrochloric acid ($\rho_{20} = 1{,}18$ g/ml) and the last (25 mg/l of aluminium), which already contains 2,5 ml of hydrochloric acid, requires an addition of 7,5 ml.

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 309,3 nm and a 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, using the atomic absorption measurements of the test solution (see 7.1.3.1 or 7.1.4) and of the solution of the fused filter residue (see 7.1.5.2). Sum both percentage results for aluminium.

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 For sample 3920 one laboratory was rejected as a Cochran outlier. For sample 7013 two laboratories were rejected, one as a Cochran and one as a Dixon outlier. However, the result which failed the Cochran test could be classified as a statistical straggler and would not have been rejected at the less critical test level given in ISO 5725.

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	0,169	0,005 2	0,011 4	0,014 7	0,035 4
902	0,434	0,004 5	0,007 5	0,012 7	0,024 8
3920	0,146	0,001 6	0,003 9	0,004 7	0,012 1
3927	0,109	0,003 2	0,004 6	0,009 1	0,015 9
7013	1,51	0,012 0	0,004 6	0,034 1	0,036 5
7049	0,972	0,008 7	0,008 6	0,024 5	0,034 5

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