TECHNICAL SPECIFICATION

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Nickel alloys — Determination of Nickel content — Inductively coupled plasma atomic emission spectrometric method

Alliages de nickel — Détermination du nickel — Méthode par spectrométrie d'émission atomique avec source à plasma induit par haute fréquence

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Foreword

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 155, *Nickel and nickel alloys*.

Nickel alloys — Determination of Nickel content — Inductively coupled plasma atomic emission spectrometric method

1 Scope

This Technical Specification describes an inductively coupled plasma atomic emission spectrometric method for the determination of nickel content (mass fraction) between 20,0 % and 80,0 % in nickel alloys.

Besides alloys where nickel is the main component regarding its content level, this method may also apply to alloys in which nickel has a content as high as several other elements (Fe, Cr, Co,…) and in which the “main element” cannot be specified.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 648, *Laboratory glassware — Single-volume pipettes*

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

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

3 Principle

Dissolution of a test portion in a mixture of hydrofluoric, hydrochloric, nitric and phosphoric acid, and fuming after addition of perchloric acid.

If necessary, addition of extra hydrofluoric acid and, if desired, addition of an internal reference element (scandium recommended).

Dilution of the test solution to a known volume. Nebulisation of this solution into an inductively coupled plasma atomic emission spectrometer and measurement of the intensity of the emitted light (including, where appropriate, that of the internal reference element).

The method uses a calibration based on a very close matrix matching of the calibration solutions to the sample composition and a bracketing of the mass fractions between ±2 % of the approximate content of nickel in the sample to be analysed.

The content of all elements in the sample has, therefore, to be approximately known. If the composition is not known, the sample shall be analysed by some semi quantitative method.

The advantage with this procedure is that all possible matrix interferences will be minimized which will result in a higher trueness. This is important for spectral interferences which can be severe in very highly alloyed matrixes. All possible interferences shall be kept at a minimum level. Therefore, it is essential that the spectrometer used meets the performance criteria recommended for the selected analytical lines.

The wavelengths reported in Table 1 have been investigated and the strongest possible interferences are given. If other wavelengths are used, they shall be carefully checked. The wavelength for the internal reference element should be selected carefully. The use of scandium at 363,07 nm is recommended. This line is interference-free for the elements and contents generally found in nickel alloys.

Table 1 — Examples of wavelengths for Nickel

Element	**Wavelengths**, nm	**Interferences**
Nickel	231,604	Co, Mo
	230,299	/
	222,486	Co
	222,295	V, Co
	217,514	/
	227,877	/
	239,452	/
	221,647	Co
	227,021	/

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only “grade 2” water as specified in ISO 3696, or water of equivalent purity.

The same reagents should be used for the preparation of calibration solutions and of sample solutions.

4.1 Hydrofluoric acid, **HF**, 40 % (m/m), ρ = 1,14 g/ml, or 50 % (m/m), ρ = 1,17 g/ml.

WARNING — Hydrofluoric acid is extremely irritating and corrosive for the skin and the mucous membranes producing severe skin burns which are slow to heal. In the case of contact with skin, wash well with water, apply a topical gel containing 2,5 % (mass fraction) calcium gluconate, and seek immediate medical treatment.

4.2 Hydrochloric acid, **HCl**, ρ = 1,19 g/ml.

4.3 Nitric acid, **HNO_3**, ρ = 1,40 g/ml.

4.4 Phosphoric acid, **H_3PO_4**, ρ = 1,70 g/ml.

4.5 Perchloric acid, **$HClO_4$**, 60 % (m/m) ρ = 1,54 g/ml or 70 % (m/m), ρ = 1,67 g/ml.

WARNING — Perchloric acid vapour may cause explosion in the presence of ammonia, nitrous fume, or organic matter, in general. All evaporation shall be carried out by using fume hoods specifically designed for perchloric acid evaporations.

4.6 Internal reference element solution, **100 mg/l**.

Choose a suitable element to be added as internal reference and prepare a 100 mg/l solution.

NOTE 1 Commercially available standard solutions can be used for this purpose.

NOTE 2 Scandium was used during the precision test of this method.

4.7 Nickel standard solution, 10 g/l.

Weigh 5 g ± 0,001 g of high purity nickel [min 99,9 % (m/m)], transfer it to a beaker and dissolve in 50 ml of water and 100 ml of nitric acid (4.3). Cover with a watch glass and heat gently until the nickel is completely dissolved. Cool and transfer quantitatively into a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 10 mg of nickel.

NOTE Commercially available standard solutions are not allowed.

4.8 Nickel standard solution, 5 g/l.

Weigh 5 g ± 0,001 g of high purity nickel [min 99,9 % (m/m)], transfer it to a beaker, and dissolve in 50 ml of water and 100 ml of nitric acid (4.3). Cover with a watch glass and heat gently until the nickel is completely dissolved. Cool and transfer quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 5 mg of nickel.

NOTE Commercially available standard solutions are not allowed.

4.9 Standard solutions of matrix elements.

Prepare standard solutions for each element whose content (mass fraction) is higher than 1 % in the sample. Use pure metals or chemical substances with nickel content (mass fraction) less than 100 µg/g.

5 Apparatus

All volumetric glassware shall be class A and calibrated in accordance with ISO 648 or ISO 1042, as appropriate.

5.1 Polytetrafluoroethylene (PTFE) beakers or Perfluoroalkyoxy-polymer (PFA) beakers with a graphite base.

5.2 Polypropylene or polyethylene volumetric flasks, of capacity 250 ml, calibrated according to ISO 1042.

5.3 Optical emission spectrometer, equipped with inductively coupled plasma.

This shall be equipped with a nebulisation system resistant to hydrofluoric acid. The instrument used will be satisfactory if, after adjustment according to 7.6, it meets the performance criteria given in 5.3.

The spectrometer can be either a simultaneous or a sequential one. If a sequential spectrometer can be equipped with an extra device or simultaneous measurement of the internal reference element line, it can be used with the internal reference method. If the sequential spectrometer is not equipped with this device, an internal reference cannot be used and an alternative technique without an internal reference element shall be used.

5.3.1 Practical resolution of a sequential spectrometer.

Calculate the bandwidth (full width at half maximum), according to A.1, for the line used, including that for the internal reference element. The bandwidth shall be less than 0,030 nm.

5.3.2 Short-term stability.

Calculate the standard deviation of 10 measurements of the absolute intensity or intensity ratio corresponding to nickel and to the internal reference element, by using the most concentrated calibration solution for nickel, according to A.2. The relative standard deviation should not exceed 0,2 %.

NOTE Short-term stability values from 0,12 % to <0,8 % were obtained during the precision test of this method.