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Ferronickels – Determination of phosphorus, manganese, chromium, copper and cobalt contents – Inductively coupled plasma atomic emission spectrometric method

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloy*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Ferronickels – Determination of phosphorus, manganese, chromium, copper and cobalt contents – Inductively coupled plasma atomic emission spectrometric method

1 Scope

This document specifies a method for the determination of phosphorus, manganese, chromium, copper and cobalt contents in ferronickels, by inductively coupled plasma optical emission spectrometry, within the ranges specified in Table 1.

This method is applicable to all kinds of ferronickels specified in ISO 6501.

Table 1 — Application ranges of the elements to be determined

Element	Application range % (mass fraction)
Phosphorus	0,009 to 0,045
Manganese	0,02 to 1,0
Chromium	0,076 to 1,86
Cobalt	0,24 to 1,4
Copper	0,02 to 0,07

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 385, *Laboratory glassware — Burettes*

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*

ISO 8049, *Ferronickel shot — Sampling for analysis*

ISO 8050, *Ferronickel ingots or pieces — Sampling for analysis*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

Dissolution of a test portion with nitric, hydrochloric and hydrofluoric acid. Addition of perchloric acid to remove fluorine and silicon. Addition of nitric and hydrochloric acid to dissolve the salts. After suitable dilution and, if necessary, addition of an internal reference element, nebulisation of the solution into an inductively coupled plasma atomic emission spectrometer and measurement of the intensity of the emitted light from each element (including, where relevant, the intensity of the internal reference element).

5 Reagents

5.1 General

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only Grade 2 water as specified in ISO 3696. The same reagents should be used for the preparation of calibration solutions and of sample solutions.

5.2 Pure nickel, containing less than 0,001 % (mass fraction) of P, Mn, Cr, Cu and Co.

5.3 Pure iron, containing less than 0,001 % (mass fraction) of P, Mn, Cr, Cu and Co.

5.4 Hydrochloric acid, HCl, ρ_{20} = 1,19 g/ml.

5.5 Nitric acid, HNO_3, ρ_{20} = 1,40 g/ml.

5.6 Nitric acid, HNO_3, diluted 1+1.

Add 500 ml of nitric acid (5.5) to 500 ml of water and mix.

5.7 Hydrofluoric acid, HF, ρ_{20} = 1,14 g/ml, or ρ_{20} = 1,17 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 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.

5.8 Perchloric acid, $HClO_4$, ρ_{20} = 1,54 g/ml, or ρ_{20} = 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 must be carried out in fume hood specifically designed for use of perchloric acid.

5.9 Sulfuric acid, H_2SO_4, ρ_{20} = 1,84 g/ml.

5.10 Internal reference element solution, 1g/l.

Weigh (1,27 ± 0,001) g of yttrium oxide [minimum purity 99,98 % (mass fraction)] and dissolve in 50 ml of hydrochloric acid (5.4). Transfer to a 1000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this internal reference element solution contains 1 mg of yttrium.

Commercial available standard solutions can be used for this purpose.

5.11 Internal reference element solution, 10 mg/l.

Transfer 10,0 ml of the reference element solution (5.10) into a 1000 ml one-mark volumetric flask, add 50 mI of hydrochloric acid (5.4) and dilute to the mark with water and mix.

1 ml of this internal reference element solution contains 10 µg of yttrium.

5.12 Phosphorus standard solution, 1 g/l.

Weigh (2,197 ± 0,001) g of potassium dihydrogen phosphate, previously dried to constant mass at 110 °C and cooled in a desiccator. Dissolve it in a 250 ml beaker with water. Transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of phosphorus.

5.13 Phosphorus standard solution, 0,25 g/l.

Transfer 25 ml of the phosphorus standard solution (5.12) into a 100 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 0,25 mg of phosphorus.

5.14 Manganese standard solution, 1 g/l.

The manganese used to prepare the solution is released from superficial oxide possibly present by introducing a few grams of metal in a 250 ml beaker containing 150 to 160 ml of water and 15 to 20 ml of sulphuric acid (5.9). Shake and after a few seconds, allow the solution to settle and add water. Repeat the water cleaning several times. Remove the metallic manganese and rinse with acetone. Dry the metal in an oven at 100 °C for 2 min or with a hair dryer. Cool in a desiccator.

Weigh (0,5 ± 0,001) g of manganese [minimum purity 99,95 % (mass fraction)] and transfer into a 250 ml beaker. Add 5 ml of hydrochloric acid (5.4) and 10 ml of nitric acid solution (5.6). Cover with a watch-glass and heat gently until the metal is dissolved. Boil to remove nitrogen oxides. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of manganese.

5.15 Chromium standard solution, 1 g/l.

Weigh (0,5 ± 0,001) g of chromium [minimum purity 99, 99 % (mass fraction)] and transfer into a 250 ml beaker. Add 40 ml of hydrochloric acid (5.4), cover with a watch-glass and heat gently until the metal is completely dissolved. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of chromium.

5.16 Copper standard solution, 1 g/l.

Weigh (0,5 ± 0,001) g of copper [minimum purity 99,95 % (mass fraction)] and transfer into a 250 ml beaker. Add 30 ml of nitric acid solution (5.6), cover with a watch-glass and heat gently until the metal is dissolved. Boil to remove nitrogen oxides. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of copper.

5.17 Cobalt standard solution, 1 g/l.

Weigh (0,5 ± 0,001) g of cobalt [minimum purity 99,95 % (mass fraction)] and transfer into a 250 ml beaker. Add 40 ml of nitric acid solution (5.6), cover with a watch-glass and heat gently until the metal

is dissolved. Boil to remove nitrogen oxides. After cooling, transfer the solution quantitatively into a 500 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of cobalt.

5.18 Cobalt standard solution, 0,25 g/l.

Transfer 25,0 ml of the cobalt standard solution (5.17) into a 100 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 0,250 mg of cobalt.

5.19 Multi elemental standard solution of copper and phosphorus

Respectively transfer 5,0 ml of the copper standard solution (5.16) and 10,0 ml of the phosphorus standard solution (5.13) into a 100 ml one-mark volumetric flask, dilute to the mark with nitric acid solution (5.6) and mix.

1 ml of this standard solution respectively contains 50 µg of copper and 25 µg of phosphorus.

5.20 Multi elemental standard solution of manganese and chromium

Respectively transfer 10,0 ml of the manganese standard solution (5.14) and 25,0 ml of the chromium standard solution (5.15) into a 100 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution respectively contains 0,10 mg of manganese and 0,25 mg of chromium.

6 Apparatus

6.1 General

All volumetric glassware shall be class A and calibrated in accordance with ISO 385, ISO 648 or ISO 1042, as appropriate.

Ordinary laboratory apparatus and the following:

6.2 Polytetrafluoroethylene (PTFE) beakers, of capacity 250 ml.

6.3 Polypropylene volumetric flasks, of capacity 100 ml.

6.4 Optical emission spectrometer (OES), equipped with inductively coupled plasma (ICP).

The spectrometer shall be equipped with a nebulisation system. The instrument used will be satisfactory if, after optimising in accordance with the manufacturer's instructions, it meets the performance criteria given in 6.4.1 to 6.4.6.

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

6.4.1 Wavelengths

This document does not specify any particular wavelength. It is mandatory that each laboratory investigate the wavelengths available on its own equipment to find the most suitable one regarding sensitivity and absence of interferences.

In Table 2, however, several suggestions are given together with possible interferences. These wavelengths have been investigated.

The wavelength of the internal reference element chosen shall not interfere with the analytical wavelengths, nor should the internal element wavelength be interfered by elements present in the test solution. It is recommended to use Y 371,030 nm. This wavelength is free of interferences from the elements generally present in ferronickels.

Table 2 — Suggested wavelengths and interfering elements

Element	Wavelength nm	Interfering elements
Phosphorus	178,287	
	213,618	Cu
Manganese	257,610	Fe
Chromium	267,716	W-V
Copper	324,754	Fe
Cobalt	228,616	Ta-Ni

6.4.2 Practical resolution of the spectrometer

Calculate the bandwidth, in accordance with A.1 (see Annex A), for each wavelength used including that for the internal reference. The bandwidth shall be less than 0,030 nm.

6.4.3 Short-term stability

Calculate the standard deviation of 10 measurements of the absolute intensity or of the intensity ratio between the analyte and the internal reference element of the highest concentration calibration solution of the analyte in accordance with A.2 (see Annex A). The relative standard deviation shall not exceed 1,0 %.

6.4.4 Long-term stability

Long term stability assessment is a measurement of the instrument drift. This is only required if the spectrometer is set up to work for long intervals of time. It consists of carrying out the same short-term stability tests at specific intervals of time,15 min to one hour, and plotting the deviation of the average found for every short-term test against time. Deviations of more than 2,0 % per hour should not be accepted.

In case the instrument is not able to perform better, a calibration solution should be measured more often during the analysis and the mean results of the test sample solutions should be recalculated by interpolation between two consecutive measurements of this calibration solution.

6.4.5 Background equivalent concentration

Calculate the background equivalent concentration (BEC) in accordance with A.3 (see Annex A), for each analytical wavelength using a solution which match the matrix composition of the samples.

6.4.6 Linearity of the calibration curves

The linearity of the calibration curves is checked by calculating the corresponding correlation coefficient. Each coefficient shall be higher than 0,999.