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

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PROOF / ÉPREUVE



Reference number
ISO 23156:2021(E)

iTeh STANDARD PREVIEW (standards.iteh.ai)

ISO/PRF 23156

<https://standards.iteh.ai/catalog/standards/sist/fdc1dc47-ae5-4896-82cb-c3b02f22eec9/iso-prf-23156>



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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

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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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 alloys*.

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 optical 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 (ICP) optical emission spectrometry, within the ranges specified in [Table 1](#).

This method is applicable to all grades 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

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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 acids. Addition of perchloric acid to remove fluorine and silicon. Addition of nitric and hydrochloric acids to dissolve the salts. After suitable dilution and, if necessary, addition of an internal reference element, nebulization of the solution into an ICP optical 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

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.

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

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

5.3 Hydrochloric acid, HCl, $\rho_{20} = 1,19$ g/ml.

5.4 Nitric acid, HNO₃, $\rho_{20} = 1,40$ g/ml.

5.5 Nitric acid, HNO₃, diluted 1 + 1.

Add 500 ml of nitric acid (5.4) to 500 ml of water and mix.

5.6 Hydrofluoric acid, HF, $\rho_{20} = 1,14$ 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.

5.7 Perchloric acid, HClO₄, $\rho_{20} = 1,54$ 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 in fume hood specifically designed for the use of perchloric acid.

5.8 Sulfuric acid, H₂SO₄, $\rho_{20} = 1,84$ g/ml.

5.9 Internal reference element solution, 1 g/l.

Weigh $(1,27 \pm 0,001)$ g of yttrium oxide [minimum purity 99,98 % (mass fraction)] and dissolve in 50 ml of hydrochloric acid (5.3). Transfer the solution quantitatively into a 1 000 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.

NOTE Commercially available standard solutions are also used for this purpose.

5.10 Internal reference element solution, 10 mg/l.

Transfer 10,0 ml of the internal reference element solution (5.9) into a 1 000 ml one-mark volumetric flask, add 50 ml of hydrochloric acid (5.3), dilute to the volume with water and mix.

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

5.11 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 volume with water and mix.

1 ml of this solution contains 1 mg of phosphorus.

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

Transfer 25 ml of the phosphorus standard solution (5.11) into a 100 ml one-mark volumetric flask, dilute to the volume with water and mix.

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

5.13 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 ml to 160 ml of water and 15 ml to 20 ml of sulphuric acid (5.8). 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 hairdryer. 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 (5.3) and 10 ml of nitric acid solution (5.5). 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 volume with water and mix.

1 ml of this solution contains 1 mg of manganese.

5.14 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 (5.3), 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 volume with water and mix.

1 ml of this solution contains 1 mg of chromium.

5.15 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 (5.5), 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 volume with water and mix.

1 ml of this solution contains 1 mg of copper.

5.16 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 (5.5), 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 volume with water and mix.

1 ml of this solution contains 1 mg of cobalt.

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

Transfer 25,0 ml of the cobalt standard solution (5.16) into a 100 ml one-mark volumetric flask, dilute to the volume with water and mix.

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

5.18 Multi-elemental standard solution of copper and phosphorus

Transfer 5,0 ml of the copper standard solution (5.15) and 10,0 ml of the phosphorus standard solution (5.12) into a 100 ml one-mark volumetric flask, dilute to the volume with nitric acid solution (5.5) and mix.

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

5.19 Multi-elemental standard solution of manganese and chromium

Transfer 10,0 ml of the manganese standard solution (5.13) and 25,0 ml of the chromium standard solution (5.14) into a 100 ml one-mark volumetric flask, dilute to the volume 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

Ordinary laboratory apparatus and the following

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

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

6.1.2 Polypropylene volumetric flasks, of capacity 100 ml.

6.1.3 Optical emission spectrometer (OES), equipped with ICP.

The spectrometer shall be equipped with a nebulization system. The instrument used will be satisfactory if, after optimizing in accordance with the manufacturer's instructions, it meets the performance requirements given in 6.2.1 to 6.2.5.

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.

6.2 Instrument performance requirements

6.2.1 Wavelengths

This document does not specify particular wavelengths. 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 the corresponding 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 corresponding 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 |

6.2.2 Practical resolution of the spectrometer

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

6.2.3 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](#). The coefficient of variation shall not exceed 1,0 %.

6.2.4 Background equivalent concentration

Calculate the background equivalent concentration (BEC), in accordance with [A.3](#), for each analytical wavelength using a solution which matches the matrix composition of the samples.

6.2.5 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.

7 Sampling and sample preparation

7.1 Sampling shall be carried out in accordance with ISO 8049 or ISO 8050 or appropriate national standards for ferronickels.

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

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

8 Procedure

8.1 Test portion

Weigh, to the nearest 0,001 g, approximately 0,50 g of the test sample.

8.2 Preparation of the test solution

8.2.1 Place the test portion (see 8.1) in a 250 ml PTFE beaker (6.1.1).

8.2.2 Add 10 ml of water, 10 ml of nitric acid (5.4), 4 ml of hydrochloric acid (5.3) and 5 ml of hydrofluoric acid (5.6) to the test portion (8.1). Slowly dissolve the sample at a low temperature to avoid a strong dissolution reaction and an excessive evaporation of the acids. After the metal is dissolved, add 4 ml of perchloric acid (5.7) and heat until abundant white fumes appear and continue fuming for 2 min to 3 min.

8.2.3 Cool the solution and add 10 ml of nitric acid solution (5.5) and 4 ml of hydrochloric acid (5.3). Heat gently to dissolve the salts and allow to cool. Transfer the solution quantitatively into a 100 ml one-mark volumetric flask. If the internal reference element is used, add 10,0 ml of reference element solution (5.10).

8.2.4 Dilute to the volume with water and mix.

8.2.5 Filter the test solution through a medium-texture filter, collect the filtrate in a clean and dry flask after discarding the first 2 ml or 3 ml of the filtrate.

8.3 Preparation of the calibration solutions

8.3.1 For 0,50 g test portions transfer the masses of nickel (5.1) and iron (5.2) given in Table 3 which are equivalent to the matrix composition of the test sample, weighted to the nearest 0,001 g, into a series of 250 ml beakers.

Table 3 — Mass of the matrix metals in test portion

| Nickel content % | Mass of nickel (5.1) g | Mass of iron (5.2) g |
|---------------------|---------------------------|-------------------------|
| 15 to 30 | 0,10 | 0,40 |
| 30 to 50 | 0,20 | 0,30 |
| 50 to 70 | 0,30 | 0,20 |
| 70 to 80 | 0,40 | 0,10 |

8.3.2 Continue as described in 8.2.2 and 8.2.3. Add to each volumetric flask the volumes of the standard solutions given in Table 4.

8.3.3 Dilute to the volume with water and mix.

Table 4 — Composition of the multi-elemental calibration solutions

| Calibration solution no. | Volume of standard solution ml | | | | Corresponding element content in the test portion % | | | | |
|--------------------------|--|---|--|---|---|------|------|------|------|
| | Standard solutions of copper and phosphorus (5.18) | Chromium standard solutions, 1 g/l (5.14) | Manganese standard solutions, 1 g/l (5.13) | Cobalt standard solutions, 1 g/l (5.16) | P | Cu | Cr | Mn | Co |
| 1 | 0 | 10,0 | 7,0 | 0 | 0 | 0 | 2,00 | 1,40 | 0 |
| 2 | 1,0 | 8,0 | 6,0 | 2,0 ^b | 0,005 | 0,01 | 1,60 | 1,20 | 0,10 |
| 3 | 2,0 | 7,0 | 5,0 | 3,0 ^b | 0,010 | 0,02 | 1,40 | 1,00 | 0,15 |
| 4 | 3,0 | 5,0 | 4,0 | 5,0 ^b | 0,015 | 0,03 | 1,00 | 0,80 | 0,25 |
| 5 | 4,0 | 4,0 | 3,0 | 2,0 | 0,020 | 0,04 | 0,80 | 0,60 | 0,40 |
| 6 | 5,0 | 3,0 | 2,0 | 3,0 | 0,025 | 0,05 | 0,60 | 0,40 | 0,60 |
| 7 | 6,0 | 2,0 | 1,0 | 4,0 | 0,030 | 0,06 | 0,40 | 0,20 | 0,80 |
| 8 | 7,0 | 5,0 ^a | 5,0 ^a | 5,0 | 0,035 | 0,07 | 0,25 | 0,10 | 1,00 |
| 9 | 8,0 | 3,0 ^a | 3,0 ^a | 6,0 | 0,040 | 0,08 | 0,15 | 0,06 | 1,20 |
| 10 | 9,0 | 1,0 ^a | 1,0 ^a | 7,0 | 0,045 | 0,09 | 0,05 | 0,02 | 1,40 |
| 11 | 10,0 | 0 | 0 | 8,0 | 0,050 | 0,10 | 0 | 0 | 1,60 |

^a Use the multi-elemental standard solution of manganese and chromium (5.19).

^b Use the cobalt standard solution, 0,25 g/l (5.17).

8.4 Optimization of the instrument

8.4.1 Start the ICP OES and let it stabilize in accordance with the manufacturer's instructions before taking any measurements.

8.4.2 Optimize the instrument according to the manufacturer's instructions.

8.4.3 Prepare the software for measurements of the intensity, and for the calculation of the mean value and coefficient of variation corresponding to each wavelength.

8.4.4 Check the instrument performance requirements given in 6.2.2 to 6.2.5.

8.5 Measurements

8.5.1 Measurement of the calibration solutions

From Table 4, according to the sample content, select at least five appropriate standard solutions (excluding the zero member) to ensure that the calibration range covers the sample contents, which should be as far as possible within the middle section of the calibration range.

Measure the absolute intensities or the ratioed intensities at the wavelengths preselected, beginning with the zero member of the calibration and ending up with the calibration solution of highest concentration. Measure each of the calibration solutions two times and calculate the mean intensities.

Nebulize water between each calibration solution.