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Low-alloyed steel — Determination of Mn, P, Cr, Ni, Mo, Co, Cu, V, Ti, As and Sn — Inductively coupled plasma optical emission spectrometric method

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National foreword

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Foreword

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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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This document was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*.

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Low-alloyed steel — Determination of Mn, P, Cr, Ni, Mo, Co, Cu, V, Ti, As and Sn — Inductively coupled plasma optical emission spectrometric method

1 Scope

This document specifies a method for the determination of Mn, P, Cr, Ni, Mo, Co, Cu, V, Ti, As and Sn contents in low alloyed steel by inductively coupled plasma optical emission spectrometry (ICP-OES).

The method is applicable to the determination of Mn, P, Cr, Ni, Mo, Co, Cu, V, Ti, As and Sn within the ranges given in Table 1.

Table 1 — Application ranges of the elements to be determined

Element	Application range % (mass fraction)
Mn	0,002 to 2,0
P	0,005 to 0,1
Cr	0,003 to 3,0
Ni	0,005 to 4,0
Mo	0,003 to 1,0
Co	0,002 to 0,2
Cu	0,003 to 0,5
V	0,002 to 0,5
Ti	0,002 to 0,5
As	0,003 to 0,1
Sn	0,003 to 0,08

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 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 14284, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition*

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

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

4 Principle

Dissolution of a test portion in nitric and hydrochloric acids. Filtration and ignition of the acid insoluble residue. Removal of silica with hydrofluoric acid.

Fusion of the residue with potassium hydrogen sulfate, dissolution of the melt with acid and addition of this solution to the reserved filtrate. If necessary, addition of an internal standard element and dilution of the solution to a known volume.

Nebulization of the solution into an ICP-OES spectrometer and measurement of the intensity of the emitted light from each element simultaneously with the intensity of the light emitted by the internal standard 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.

5.1 Pure iron, containing at least ten times less than the lower limit of the scope of each element to be determined.

5.2 Potassium hydrogen sulfate ($KHSO_4$) or potassium disulfate ($K_2S_2O_7$).

5.3 Hydrochloric acid, ρ about 1,19 g/ml.

5.4 Nitric acid, ρ about 1,42 g/ml.

5.5 Nitric acid solution, ρ about 1,42 g/ml, diluted 1 + 1.

5.6 Hydrofluoric acid, ρ about 1,15 g/ml.

5.7 Sulfuric acid, ρ about 1,84 g/ml.

5.8 Sulfuric acid solution, ρ about 1,84 g/ml, diluted 1 + 3.

5.9 Manganese standard solution, 1 g/l.

Transfer several grams of electrolytic manganese (purity > 99,9 %) into a 250 ml beaker containing about 150 ml of sulphuric acid solution (ρ about 1,84 g/ml, diluted 5+95). Stir, then allow the manganese to settle for several minutes. Decant and eliminate the sulphuric acid solution and substituting water. Wash several times with water and finally with acetone. Dry the metal for about 2 min at 100 °C and cool in a desiccator.

Weigh, to the nearest 1 mg, 1,000 g of manganese as mentioned above. Transfer into a 250 ml beaker, add 40 ml of hydrochloric acid (5.3) and cover with a watch-glass. Heat gently to complete dissolution. Cool, 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 standard solution contains 1 mg of manganese.

5.10 Phosphorus standard solution, 1 g/l.

Weigh, to the nearest 0,1 mg, 4,393 6 g of potassium dihydrogen phosphate (KH_2PO_4) previously dried to constant mass at 110 °C and cooled in a desiccator.

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Transfer into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 1 mg of phosphorus.

5.11 Chromium standard solution, 1 g/l.

Prepare a 1 g/l chromium standard solution by using one of the procedures described in a) or b).

a) Weigh, to the nearest 1 mg, 1,000 g of chromium [purity ≥ 99,9 % (mass fraction)]. Transfer into a 250 ml beaker, add 40 ml of hydrochloric acid (5.3) and cover with a watch-glass. Heat gently to complete dissolution. Cool, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

b) Weigh 2,828 4 g of potassium dichromate ($K_2Cr_2O_7$), previously dried at 140 °C and allowed to cool in a desiccator. Place in a 400 ml beaker and dissolve in about 20 ml of water. Add 5 ml of the sulfuric acid (5.7) and, while cooling, cautiously add the hydrogen peroxide solution (to be added), adding an excess of about 2 ml after effervescence has ceased. Allow the solution to stand at ambient temperature until the yellow colour has completely disappeared (several hours), then transfer into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix well.

1 ml of this standard solution contains 1 mg of chromium.

5.12 Nickel standard solution, 1 g /l.

Weigh, to the nearest 1 mg, 1,000 g of nickel [purity ≥ 99,9 % (mass fraction)]. Transfer into a 250 ml beaker, add 50 ml of nitric acid (5.5) and cover with a watch-glass. Heat gently to complete dissolution. Cool, 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 standard solution contains 1 mg of nickel.

5.13 Molybdenum standard solution, 1 g /l.

Weigh, to the nearest 1 mg, 1,000 g of molybdenum [purity ≥ 99,9 % (mass fraction)]. Transfer into a 250 ml beaker, add 50 ml of hydrochloric acid (5.3) and 50 ml of nitric acid (5.4) and cover with a watch-glass. Heat gently to complete dissolution. Cool, 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 standard solution contains 1 mg of molybdenum.

5.14 Cobalt standard solution, 1 g/l.

Weigh, to the nearest 1 mg, 1,000 g of cobalt [purity ≥ 99,9 % (mass fraction)]. Transfer into a 250 ml beaker, add 50 ml of nitric acid solution (5.5) and cover with a watch-glass. Heat gently to complete dissolution. Cool, 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 standard solution contains 1 mg of cobalt.

5.15 Copper standard solution, 1 g/l.

Weigh, to the nearest 1 mg, 1,000 g of copper [purity ≥ 99,9 % (mass fraction)]. Transfer into a 250 ml beaker, add 50 ml of nitric acid solution (5.5) and cover with a watch-glass. Heat gently to complete dissolution. Cool, 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 standard solution contains 1 mg of copper.

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5.16 Vanadium standard solution, 1 g/l.

Weigh, to the nearest 1 mg, 1,000 g of vanadium [purity ≥ 99,9 % (mass fraction)]. Transfer into a 250 ml beaker, add 30 ml of aqua regia [mix three volume of hydrochloric acid (5.3) with one volume of nitric acid (5.4)] and cover with a watch-glass. Heat gently to complete dissolution. Cool, 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 standard solution contains 1 mg of vanadium.

5.17 Titanium standard solution, 1 g/l.

Weigh, to the nearest 1 mg, 200 mg of titanium [purity ≥ 99,9 % (mass fraction)]. Transfer into a 250 ml beaker, add 50 ml of hydrochloric acid (5.3), diluted 1 + 1 and 5 drops of hydrofluoric acid (5.6) and cover with a watch-glass. Heat gently to complete dissolution. Cool, transfer the solution quantitatively into to a 200 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 1 mg of titanium.

5.18 Arsenic standard solution, 100 mg/l.

Prepare an arsenic standard solution by using one of the procedures described in a) or b).

a) Weigh, to the nearest 1 mg, 100 mg of arsenic [purity ≥ 99,9 % (mass fraction)]. Transfer into a 250 ml beaker, add 20 ml of nitric acid solution (5.5) and cover with a watch-glass. Heat gently to complete dissolution. Cool, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

b) Weigh, to the nearest 1 mg, 132 mg of arsenic trioxide. Transfer into a 250 ml beaker, add 6 ml of potassium hydroxide solution (10 g/l) and dilute with water to 100 ml. Adjust the pH between 3 and 6 with hydrochloric acid (diluted 1+10). Transfer the solution quantitatively into 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 0,1 mg of arsenic.

5.19 Tin standard solution, 500 mg/l.

Weigh, to the nearest 1 mg, 500 mg of tin [purity ≥ 99,9 % (mass fraction)]. Transfer into a 250 ml beaker, add 20 ml of hydrochloric acid (5.3) and 5 ml of nitric acid (5.4) and cover with a watch-glass. Heat gently to complete dissolution. Remove from the hot plate immediately and allow to cool. Transfer the solution into a 1 000 ml one-mark volumetric flask containing 200 ml of hydrochloric acid (5.3). Dilute to the mark with water and mix.

1 ml of this standard solution contains 0,5 mg of tin.

5.20 Yttrium internal standard solution, 100 mg/l.

Calcine several grams of yttrium oxide [purity ≥ 99,9 % (mass fraction)] in a muffle furnace at 850 °C ± 10 °C for at least 40 min and then allow to cool in a desiccator. Weigh 0,127 0 g of the calcined product, transfer into a 250 ml beaker, add 10 ml of hydrochloric acid (5.3) and cover with a watch-glass. Heat gently to complete dissolution. Cool, transfer the solution into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 0,1 mg of yttrium.

NOTE Elements as cadmium and scandium can also be used as internal standards.

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5.21 Multi-elemental standard solutions for Mn, P, Cr, V and Ti

5.21.1 Multi-elemental standard solution for Mn, P, Cr, V and Ti, corresponding to 100 mg of each element per litre.

Transfer 10,0 ml of each of the standard solutions (5.9), (5.10), (5.11), (5.16) and (5.17) into a 100 ml one-mark volumetric flask. Add 5 ml hydrochloric acid (5.3), dilute to the mark with water and mix.

1 ml of this standard solution contains 100 µg of Mn, P, Cr, V and Ti.

5.21.2 Multi-elemental standard solution for Mn, P, Cr, V and Ti, corresponding to 10 mg of each element per litre.

Transfer 10,0 ml of the multi-elemental standard solution (5.21.1) into a 100 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 10,0 µg of Mn, P, Cr, V and Ti.

Prepare this solution immediately before use.

5.22 Multi-elemental standard solutions for Ni, Mo, Co, Cu, As and Sn.

5.22.1 Multi-elemental standard solution for Ni, Mo, Co, Cu and Sn, corresponding to 100 mg of each element per litre.

Transfer 10,0 ml of each of the standard solutions (5.12), (5.13), (5.14) and (5.15) and 20,0 ml of tin standard solution (5.19) into a 100 ml one-mark volumetric flask. Add 5 ml hydrochloric acid (5.3), dilute to the mark with water and mix.

1 ml of this standard solution contains 100 µg of Ni, Mo, Co, Cu and Sn.

5.22.2 Multi-elemental standard solution for Ni, Mo, Co, Cu, As and Sn, corresponding to 10 mg of each element per litre.

Transfer 10,0 ml of the multi-elemental standard solution (5.22.1) and 10,0 ml of arsenic standard solution (5.18) into a 100 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this standard solution contains 10,0 µg of Ni, Mo, Co, Cu, As and Sn.

Prepare this solution immediately before use.

NOTE Standard solutions whose preparations are described under items 5.9 to 5.20 can be replaced by commercially available standard solutions, provided that they are supplied by a recognised producer and their traceability fully documented.

6 Apparatus

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

Ordinary laboratory apparatus and the following shall be used.

6.1 Optical emission spectrometer, equipped with an inductively coupled plasma (ICP-OES).

The ICP-OES used will be satisfactory if, after optimizing according to 8.4, it meets the performance criteria given in 6.1.2 to 6.1.5.

The spectrometer can be either simultaneous or sequential. If a sequential spectrometer can be equipped with an extra arrangement for simultaneous measurement of the internal standard wavelength, it can be used with the internal standard technique. If the sequential spectrometer is not equipped with this

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arrangement, the internal standard cannot be used and an alternative technique without internal standard shall be applied.

6.1.1 Wavelengths

This method does not specify any particular emission line. It is mandatory that each laboratory carefully 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 carefully investigated.

The wavelength of the internal standard 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, however, recommended to use Y 371,030 nm. This wavelength is free of interferences from the elements.

Table 2 — Wavelengths and interfering elements

Element	Wavelength nm	Possible interferences
Mn	257,610	—
	260,569	Co, Fe, Cr
P	178,280	Mo, Cr, Mn
	213,618	Mo, Cu
Cr	267,716	Mn
	206,149	—
Ni	231,604	Co
	221,647	—
Mo	202,030	Fe
	281,615	Al, V
Co	228,616[a)]	Cr, Ti, Ni
Cu	324,754	Mn, Mo
	327,396	Mo
V	309,311	Fe
	311,071	Ti, Mo
Ti	334,941	Cr
	337,280	—
As	189,042	Cr
	193,759	—
Sn	189,989	—
	283,999	Cr
Y	371,030	None

[a)] Check and correct, if necessary, for interference by Ni and Cr

Other element can be used as internal standard, but it shall not be present in the sample and interfere with the analytical wavelengths, nor shall elements present in the test solution interfere with the internal element wavelength. Moreover, the excitation conditions of the analytical lines and the internal standard element line should match.

6.1.2 Minimum practical resolution of the spectrometer

Calculate the bandwidth, according to A.1, for the wavelength used including the wavelength of the internal standard. The bandwidth shall be less than 0,030 nm.

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6.1.3 Minimum short-term precision

Calculate the short-term precision according to A.2. The relative standard deviation (RSD) shall not exceed 0,5 % for concentrations 100 to 1 000 times above the LOD (6.1.5). For concentrations 10 to 100 times above the LOD (6.1.5), the RSD shall not exceed 5 %.

6.1.4 Long-term stability

Calculate the standard deviation of seven mean values of three measurements of the absolute intensity or intensity ratio of the emitted light of the most concentrated calibration solution for each element. Each mean value should be determined every 30 min during a total time of 3 h. The relative standard deviation shall not exceed 2 % for the absolute intensity technique or 1,5 % for the internal standard technique.

6.1.5 Limit of detection (LOD) and limit of quantification (LOQ)

Calculate the LOD and LOQ, according to the A.3, for the wavelength used. The values shall be below the values in Table 3.

Table 3 — Limit of detection (LOD) and limit of quantification (LOQ)

Element	Wavelength nm	LOD mg/l	LOQ mg/l
Mn	257,610	0,03	0,1
	260,569	0,03	0,1
P	178,280	0,06	0,2
	213,618	0,06	0,2
Cr	267,716	0,03	0,1
	206,149	0,03	0,1
Ni	231,604	0,06	0,2
	221,647	0,06	0,2
Mo	202,030	0,03	0,1
	281,615	0,03	0,1
Co	228,616	0,03	0,1
Cu	324,754	0,03	0,1
	327,396	0,03	0,1
V	309,311	0,03	0,1
	311,071	0,03	0,1
Ti	334,941	0,03	0,1
	337,280	0,03	0,1
As	189,042	0,03	0,1
	193,759	0,03	0,1
Sn	189,989	0,03	0,1
	283,999	0,03	0,1

6.1.6 Linearity of the calibration curves

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

6.2 Platinum crucible, of capacity 30 ml.

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7 Sampling and sample preparation

Sampling and sample preparation shall be carried out in accordance with ISO 14284 or with an appropriate national standard for steels.

8 Procedure

8.1 Test portion

Weigh, to the nearest 0,001 g, 0,500 g of the test sample.

8.2 Blank test

In parallel with the determination of the content and following the same procedure, carry out a blank test using the same quantities of all the reagents, including pure iron (5.1).

8.3 Determination

8.3.1 Preparation of the test solution

8.3.1.1 Dissolution of the test portion

Transfer the test portion (see 8.1) into a 250 ml beaker. Add 10 ml of nitric acid solution (5.5), cover the beaker with a watch-glass and heat gently until the acid action ceases and then boil for 1 min. Add 5 ml of hydrochloric acid (5.3) and continue the heating to boil for about 5 min.

Allow to cool, add about 15 ml of water, filter through a close texture filter paper and collect the filtrate in a 100 ml one-mark volumetric flask. Wash the filter paper several times with hot water and collect the washings in the volumetric flask. Reserve the filtrate as the main solution. Check the filter paper carefully for residue. If there is no residue, skip the procedure described in 8.3.1.2.

8.3.1.2 Treatment of insoluble residue

Transfer the filter paper and residue into a platinum crucible (6.2), dry and ignite at a temperature as low as possible until all carbonaceous matter is removed and finally at about 800 °C for at least 15 min. Cool, add 2 drops of sulfuric acid solution (5.8) and about 2 ml of hydrofluoric acid (5.6). Evaporate to dryness, heat to about 800 °C and cool. Add 1 g of potassium hydrogen sulfate (5.2) and fuse carefully until a clear melt is obtained.

For residues containing carbides, prolonged heating may be necessary for complete fusion. The potassium hydrogen sulfate can be regenerated by allowing the melt to cool, adding 2 drops of sulfuric acid (5.7) and repeating the fusion until the residue is dissolved.

After cooling, add 10 ml of water and 2 ml of hydrochloric acid (5.3) to the solidified melt in the crucible, transfer the contents to a 250 ml beaker and heat gently until the fusion products are dissolved. Cool and add this solution to the main solution (see 8.3.1.1).

8.3.1.3 Dilution of the test solution

If the internal standard technique is used, add 5,0 ml of the yttrium internal standard solution (5.20) (see NOTE). Dilute to the mark with water and mix.

NOTE Elements as cadmium and scandium can also be used as internal standard.

8.3.2 Preparation of the calibration solutions

Transfer 0,48 g ± 0,005 g of pure iron (5.1) into each of a series of nine 250 ml beakers. Treat the pure iron following the same procedure as the test portion (see 8.3.1.1). Procedure 8.3.1.2 is not necessary, provided

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