



**International
Standard**

ISO 4941

**Steel and iron — Determination of
molybdenum content — Thiocyanate
spectrophotometric method**

*Aciers et fontes — Détermination des teneurs en molybdène —
Méthode spectrophotométrique au thiocyanate*

**Third edition
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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

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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 document 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 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*.

This third edition cancels and replaces the second edition (ISO 4941:1994), which has been technically revised.

The main changes are as follows:

- the normative references have been revised;
- the precision data has been updated.

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.

Steel and iron — Determination of molybdenum content — Thiocyanate spectrophotometric method

1 Scope

This document specifies a thiocyanate spectrophotometric method for the determination of molybdenum contents in steel and iron. The method is applicable to molybdenum mass fractions between 0,005 % and 0,125 %.

Vanadium and tungsten interfere with the measurement if, because of their contents, the V/Mo ratio is greater than 16 or the W/Mo ratio is greater than 8.

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 4800, *Laboratory glassware — Separating funnels and dropping funnels*

ISO 14284, *Steel and iron — Sampling and preparation of samples for the determination of chemical composition*
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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 <https://www.electropedia.org/>

4 Principle

Dissolution of a test portion in an appropriate mixture of acids and decomposition of the carbides by oxidation.

Quantitative formation of a coloured complex of molybdenum, in the presence of thiocyanate, iron(II) and/or copper(II) ions and extraction of this compound using butyl acetate.

Spectrophotometric measurement of the coloured complex at a wavelength of about 470 nm.

5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and grade 2 water as specified in ISO 3696.

5.1 Pure iron, in flake or powder form, with a molybdenum content less than 0,000 5 % (mass fraction) and free from tungsten and vanadium.

5.2 Butyl acetate.

5.3 Nitric acid, ρ approximately 1,40 g/ml.

5.4 Hydrochloric acid, ρ approximately 1,19 g/ml.

5.5 Hydrochloric acid, ρ approximately 1,19 g/ml, diluted (3 + 1).

5.6 Hydrochloric acid, ρ approximately 1,19 g/ml, diluted (1 + 1).

5.7 Acid mixture I.

Add 2 volumes of hydrochloric acid (5.4) to 1 volume of nitric acid (5.3) and mix well. Prepare this mixture immediately before use.

5.8 Acid mixture II.

Add 150 ml of orthophosphoric acid (ρ approximately 1,70 g/ml) to 300 ml of water, and add 360 ml of perchloric acid (ρ approximately 1,67 g/ml) to this diluted acid (see NOTE). Transfer the solution into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

NOTE In the preparation of this acid mixture, 360 ml of perchloric acid (ρ approximately 1,67 g/ml) can be replaced by 150 ml of sulfuric acid (ρ approximately 1,84 g/ml).

5.9 L(+)- ascorbic acid solution, 100 g/l.

Prepare this solution just before use.

5.10 Ammonium thiocyanate solution, 320 g/l.

Store this solution away from light.

5.11 Copper(II), solution corresponding to 70 mg of Cu(II) per litre in a hydrochloric acid medium.

Dissolve 0,188 g of copper(II) chloride di-hydrate ($\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$) or 0,275 g of copper(II) sulfate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) into 125 ml of hydrochloric acid (5.4). Transfer the solution into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

5.12 Tin(II) copper(II) chloride, solution in a hydrochloric acid medium.

Dissolve 80 g of tin(II) chloride di-hydrate ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) in 155 ml of hydrochloric acid (5.4). Add 100 ml of copper(II) solution (5.11). Transfer the solution into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

Prepare this solution just before use.