
**Steel — Determination of
nickel — Dimethylglyoxime
spectrophotometric method**

*Aciers — Détermination du nickel — Méthode spectrophotométrique
à la diméthylglyoxime*

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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).

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

This second edition cancels and replaces the first edition (ISO 4939:1984), which has been technically revised. In particular, the specification of plotting of the calibration graphs shown in [7.4.3](#) has been corrected.

Steel — Determination of nickel — Dimethylglyoxime spectrophotometric method

1 Scope

This International Standard specifies a spectrophotometric method for the determination of nickel in steel using dimethylglyoxime.

The method is applicable to the determination of nickel mass fractions in the range of 0,10 % and 2,0 %. Cobalt, copper and manganese can cause interferences (see 7.3.2).

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*

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

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3 Principle

Dissolution of a test portion in hydrochloric, nitric and perchloric acids.

Formation of a coloured complex of nickel(III) with dimethylglyoxime in ammoniacal solution containing iodine and potassium iodide.

Spectrophotometric measurement at a wavelength of about 535 nm.

4 Reagents

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

4.1 Acid mixture.

Mix two volumes of hydrochloric acid, ρ approximately 1,19 g/ml, one volume of nitric acid, ρ approximately 1,40 g/ml and two volumes of water.

4.2 Perchloric acid, ρ approximately 1,54 g/ml.

WARNING — Perchloric acid vapour might cause explosions in the presence of ammonia, nitrous fumes or organic material in general.

Perchloric acid, ρ approximately 1,67 g/ml, may also be used. 100 ml of perchloric acid, ρ approximately 1,54 g/ml is equivalent to 79 ml of perchloric acid, ρ approximately 1,67 g/ml.