



**SLOVENSKI STANDARD**  
**oSIST prEN ISO 9658:2023**  
**01-december-2023**

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**Jeklo - Določevanje aluminija - Metoda plamenske atomske absorpcijske spektrometrije (ISO/DIS 9658:2023)**

Steel - Determination of aluminium content - Flame atomic absorption spectrometric method (ISO/DIS 9658:2023)

Stahl - Bestimmung des Aluminiumgehaltes -  
Flammenatomextinktionsspektrometrisches Verfahren (ISO/DIS 9658:2023)

Aciers - Détermination de l'aluminium - Méthode par spectrométrie d'absorption atomique dans la flamme (ISO/DIS 9658:2023)

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## Steel — Determination of aluminium content — Flame atomic absorption spectrometric method

*Aciers — Dosage de l'aluminium — Méthode par spectrométrie d'absorption atomique dans la flamme*

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## ISO/DIS 9658:2023(E)

### 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](http://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](http://www.iso.org/patents)).

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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](http://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 second edition cancels and replaces the first edition (ISO 9658:1990), which has been technically revised.

The main changes compared to the previous edition are as follows:

- re-assessment of the precision data;
- updating of the normative references;
- adding of some notes that could contribute to a better accuracy of the method;
- Editorially updated;
- Bibliography added.

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](http://www.iso.org/members.html).

# Steel — Determination of aluminium content — Flame atomic absorption spectrometric method

## 1 Scope

This document specifies a flame atomic absorption spectrometric method for the determination of aluminium in non-alloyed steel.

The method is applicable to aluminium contents between 0,005 % (mass fraction) and 0,20 % (mass fraction).

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

## 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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/>

### 3.1

#### acid-soluble aluminium

aluminium dissolved in the acid mixture.

## 4 Principle

Dissolution of a test portion in dilute hydrochloric and nitric acids.

Fusion of the acid-insoluble residues with a mixture of orthoboric acid and potassium carbonate.

Nebulization of the solution into a dinitrogen monoxide- acetylene flame.

Spectrometric measurement of the atomic absorption of the 309,3 nm spectral line emitted by an aluminium hollow cathode lamp.

NOTE Other suitable radiation sources can also be used, provided the criteria in 6.5.1 to 6.5.4 are still met.

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### 5 Reagents

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

**5.1 Pure iron**, containing less than 0,000 1 % (mass fraction) of aluminium or of low and known aluminium content.

**5.2 Hydrofluoric acid**,  $\rho$  approximately 1,15 g/ml.

**5.3 Hydrochloric acid**,  $\rho$  approximately 1,19 g/ml, diluted 1 + 1.

**5.4 Hydrochloric acid**,  $\rho$  approximately 1,19 g/ml, diluted 2 + 100.

**5.5 Sulfuric acid**,  $\rho$  approximately 1,84 g/ml, diluted 1 + 1.

**5.6 Hydrochloric-nitric acids mixture.**

Mix three volumes of hydrochloric acid ( $\rho$  approximately 1,19 g/ml), one volume of nitric acid ( $\rho$  approximately 1,40 g/ml) and two volumes of water.

This mixture shall be prepared immediately before use.

**5.7 Fusion mixture.**

Mix 1 part by mass of orthoboric acid ( $\text{H}_3\text{BO}_3$ ) and 1 part by mass of anhydrous potassium carbonate ( $\text{K}_2\text{CO}_3$ ).

**5.8 Fusion mixture solution.**

Dissolve 20,0 g of the fusion mixture (5.7) in water and dilute to 100 ml.

5,0 ml of this solution contain 1,0 g of the fusion mixture (5.7).

**5.9 Aluminium stock solution**, 2,0 g/l.

Weigh, to the nearest 0,001 g, 2,000 g of high purity aluminum [99,9 % (mass fraction)], and dissolve in 40 ml of hydrochloric acid ( $\rho$  about 1,19 g/ml) and 10 ml of nitric acid ( $\rho$  about 1,40 g/ml). Boil to eliminate oxides of nitrogen. Cool and 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 solution contains 2,0 mg of Al.

**5.10 Aluminium standard solution**, 0,20 g/l.

Transfer 20,0 ml of the stock solution (5.9) into a 200 ml one-mark volumetric flask. Dilute to the mark with water and mix.

Prepare this standard solution immediately prior to use.

1 ml of this solution contains 0,20 mg of Al.

**5.11 Aluminium standard solution**, 0,020 g/l.

Transfer 20,0 ml of standard solution (5.9.1) into a 200 ml one-mark volumetric flask. Dilute to the mark with water and mix.

Prepare this standard solution immediately prior to use.



1 ml of this solution contains 0,020 mg of Al.

## 6 Apparatus

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

During the analysis, unless otherwise stated ordinary laboratory apparatus and the following shall be used.

All glassware shall first be washed in hydrochloric acid (5.3), and then in water. The quantity of aluminium present in the beakers and flasks can be checked by measuring the absorption of distilled water introduced in the glassware after the acid wash.

**6.1 Filter media**, 0,45 µm cellulose nitrate filter.

**6.2 Filter funnel**,

Two-piece acid-resistant filter funnel with a support screen between the funnel body and stem, designed for the vacuum filtration of liquids. The stem of the funnel is fitted with a ground glass cap stopper or a rubber stopper for insertion into an opening of the vacuum vessel.

**6.3 Vacuum vessel**,

Flask of capacity 500 ml, or large enough to contain a 100 ml one-mark volumetric flask, with an opening to allow the insertion of the rubber stopper of the filter funnel stem.

**6.4 Platinum crucible**, of capacity 30 ml.

**6.5 Atomic absorption spectrometer**,

**WARNING — Follow the manufacturer's instructions for igniting and extinguishing the air/acetylene flame to avoid possible explosion hazards. Wear tinted safety glasses whenever the burner is in operation.**

The spectrometer shall be equipped with an aluminium hollow-cathode lamp or other suitable radiation source and supplied with dinitrogen monoxide and acetylene sufficiently pure to give a steady clear fuel-lean flame, free from water and oil, and free from aluminium.

The atomic absorption spectrometer used will be satisfactory if, after optimization according to 8.3.4, the limit of detection and characteristic concentration are in reasonable agreement with the values given by the manufacturer and it meets the performance criteria given in 6.5.1 to 6.5.3.

The instrument should also conform to the additional performance requirement given in 6.5.4.

### 6.5.1 Minimum precision

The standard deviation of 10 measurements of the absorbance of the most concentrated calibration solution shall not exceed 1,5 % of the mean absorbance of this solution.

The standard deviation of 10 measurements of the absorbance of the least concentrated calibration solution (excluding the zero member) shall not exceed 0,5 % of the mean absorbance of the most concentrated calibration solution.

### 6.5.2 Limit of detection

The limit of detection is a number, expressed in units of concentration (or amount) that describes the lowest concentration level (or amount) of an element that can be determined to be statistically different from an analytical blank.

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The limit of detection of aluminium in a matrix similar to the final test solution shall be less than 0,1 µg/ml.

### 6.5.3 Calibration linearity

The slope of the calibration curve covering the top 20 % of the concentration range (expressed as a change in absorbance) shall not be less than 0,7 times the value of the slope for the bottom 20 % of the concentration range determined in the same way.

For instruments with automatic calibration using two or more standard solutions, it shall be established prior to the analysis, by obtaining absorbance readings, that the above requirements for calibration linearity are fulfilled.

### 6.5.4 Characteristic concentration

The characteristic concentration for aluminium in a matrix similar to the final test solution shall be lower than 1,0 µg/ml.

### 6.5.5 Ancillary equipment

Scale expansion can be used until the noise observed is greater than the readout error and is always recommended for absorbances below 0,1. If scale expansion has to be used and the instrument does not have the means to read the value of the scale expansion factor, the value can be calculated by measuring a suitable solution with and without scale expansion and then dividing the signal obtained.

## 7 Sampling and preparation of the test samples

Sampling and sample preparation shall be carried out in accordance with ISO 14284 or appropriate national standards.

## 8 Procedure

### 8.1 Test portion

If necessary, degrease the test sample by cleaning in a suitable solvent. Evaporate the last traces of the solvent by warming, cautiously.

Weigh, to the nearest 0,1 mg, approximately 2,0 g of the test sample.

### 8.2 Blank test

In parallel with the determination and following the same procedure, carry out a blank test using the same quantities of all reagents, including pure iron as used for the determination instead of the test portion.

Background correction may be required.

### 8.3 Determination

#### 8.3.1 Preparation of the test solution

##### 8.3.1.1 Dissolution of the test portion

Place the test portion (8.1) into a 250 ml beaker. Add, in small portions, 40 ml of the acid mixture (5.6) and cover the beaker with a watch-glass. Heat until acid action ceases. Boil to eliminate oxides of nitrogen and cool.