



**International
Standard**

ISO 9658

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

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

**Second edition
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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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This document was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 1, *Methods of determination of chemical composition*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 459, *ECISS - European Committee for Iron and Steel Standardization*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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

The main changes are as follows:

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

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 — Determination of aluminium content — Flame atomic absorption spectrometric method

1 Scope

This document specifies a flame atomic absorption spectrometric method for the determination of acid-soluble and/or total 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 an 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.

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, ρ approximately 1,15 g/ml.

5.3 Hydrochloric acid, ρ approximately 1,19 g/ml, diluted 1 + 1.

5.4 Hydrochloric acid, ρ approximately 1,19 g/ml, diluted 2 + 100.

5.5 Sulfuric acid, ρ approximately 1,84 g/ml, diluted 1 + 1.

5.6 Hydrochloric-nitric acids mixture.

Mix three 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.

This mixture shall be prepared immediately before use.

5.7 Fusion mixture.

Mix 1 part by mass of orthoboric acid (H_3BO_3) and 1 part by mass of anhydrous potassium carbonate (K_2CO_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 standard solution, 2,0 g /l.

Weigh, to the nearest 0,001 g, 2,000 g of high purity aluminium [99,9 % (mass fraction)], and dissolve in 40 ml of hydrochloric acid (ρ about 1,19 g/ml) and 10 ml of nitric acid (ρ 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 aluminium.

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

Transfer 20,0 ml of the aluminium standard ([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 aluminium.

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

Transfer 20,0 ml of the aluminium standard solution ([5.10](#)) into a 200 ml one-mark volumetric flask. Dilute to the mark with water and mix.

Prepare this standard solution immediately prior to use.