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

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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 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 l 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.

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

6 Apparatus

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

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

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, 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, capacity of about 30 ml.

6.5 Atomic absorption spectrometer.

WARNING — Follow the manufacturer's instructions for igniting and extinguishing the dinitrogen monoxide/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.

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 calibration 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.

6.6 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 standard for steel.

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 (5.1) 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.

8.3.1.2 Filtration of the test solution

Place a filter (6.1) on the support screen of a filter funnel (6.2). Moisten the filter with water and join the body and stem of the funnel. Insert the stopper of the filter funnel stem into a vacuum vessel (6.3). Apply vacuum gently to the vacuum vessel and filter the solution.

Wash the funnel sides and residue with warm hydrochloric acid (5.4) and warm water alternately until they are visually free from iron.

Stop the vacuum gently.

When the filtrate is collected into the 500 ml vacuum vessel:

— If the volume of the filtrate and the washings is less than about 70 ml, transfer the solution quantitatively into a 100 ml one-mark volumetric flask, and proceed to 8.3.1.3 or 8.3.1.4;

— If the volume of the filtrate and the washings is greater than about 70 ml, transfer the solution quantitatively into a 200 ml beaker, reduce the volume of the solution to about 70 ml by evaporation, cool and then transfer it quantitatively into a 100 ml one-mark volumetric flask, and proceed to 8.3.1.3 or 8.3.1.4.

When the filtrate is collected directly in a 100 ml one-mark volumetric flask placed in the vacuum vessel:

— If the volume of the filtrate and the washings is less than about 70 ml, proceed to 8.3.1.3 or 8.3.1.4;

— If the volume of the filtrate and the washings is greater than about 70 ml, transfer the solution into a 200 ml beaker, reduce the volume of the solution to about 70 ml by evaporation, cool and transfer it quantitatively again into the original 100 ml one-mark volumetric flask, and proceed to 8.3.1.3 or 8.3.1.4.

8.3.1.3 Preparation of the test solution for the determination of acid-soluble aluminium

If acid-soluble aluminium only is required, add 5,0 ml of fusion mixture solution (5.8) to the 100 ml one-mark volumetric flask, cool, and allow any carbon dioxide produced to escape, then dilute to the mark with water and mix. Discard the insoluble residue and cellulose nitrate filter. Retain this solution for the determination of acid-soluble aluminium.

8.3.1.4 Preparation of the test solution for the determination of total aluminium

Transfer the filter containing the insoluble residue into a platinum crucible (6.4). Char the residue at low temperature and ignite slowly to 1 000 °C. Allow the crucible to cool. Add several drops of water, several drops of sulfuric acid (5.5) and 5 ml of hydrofluoric acid (5.2). Evaporate to dryness and again ignite slowly to 1 000 °C. Allow the crucible to cool and add 1,0 g of the fusion mixture (5.7). Fuse the contents of the crucible in a muffle furnace at 1 000 °C for 15 min.

Allow the crucible to cool and add 1 ml or 2 ml of hydrochloric acid (5.3) and 8 ml of water to the solidified melt.

Heat gently to dissolve the fusion products. Allow the crucible to cool and transfer this solution quantitatively to the filtrate in the 100 ml one-mark volumetric flask. Dilute to the mark with water and mix.

8.3.2 Preparation of the calibration solutions

8.3.2.1 Aluminium contents less than 0,010 % (mass fraction)

Transfer into each of a series of 250 ml beakers (2,00 ± 0,01) g of the pure iron (5.1). Add 40 ml of the acid mixture (5.6), in small portions, to each beaker and cover them with watch-glasses. Heat until the iron is dissolved, then boil to eliminate oxides of nitrogen. Cool and transfer the solutions into five 100 ml one-mark volumetric flasks. Add the volumes of aluminium standard solution (5.11) as shown in Table 1.

Add 5,0 ml of the fusion mixture solution (5.8) to each flask. Cool and allow any carbon dioxide produced to escape, then dilute to the mark with water and mix.

Table 1 — Calibration for aluminium contents less than 0,010 % (mass fraction)

Volume of aluminium standard solution (5.11) ml	Corresponding aluminium concentration after final dilution µg/ml	Corresponding aluminium content in the sample % (mass fraction)
0[a]	0	0
2,5	0,5	0,002 5
5,0	1,0	0,005 0
7,5	1,5	0,007 5
10,0	2,0	0,010 0
[a] Zero member		

8.3.2.2 Aluminium contents between 0,010 % (mass fraction) and 0,20 % (mass fraction)

Proceed as specified in 8.3.2.1, using Table 2 instead of Table 1.

Table 2 — Calibration for aluminium contents between 0,010 % (mass fraction) and 0,20 % (mass fraction)

Volume of aluminium standard solution (5.10) ml	Corresponding aluminium concentration after final dilution µg/ml	Corresponding aluminium content in the sample % (mass fraction)
0[a]	0	0
5,0	10,0	0,050
10,0	20,0	0,100
15,0	30,0	0,150
20,0	40,0	0,200
[a] Zero member		

8.3.3 Adjustment of atomic absorption spectrometer

Fit the aluminium hollow-cathode lamp (see 6.5) to the atomic absorption spectrometer (6.5) as well as a deuterium lamp (for the correction of the non-specific absorption), switch on the current and allow it to stabilize. Adjust the wavelength in the region of 309,3 nm to minimum absorbance, if possible.

Following the manufacturer's instructions, fit the correct burner, light the flame and allow the burner temperature to stabilize.

If no recommendation is stated, a bandwidth between 0,2 nm and 0,7 nm is suggested.

If the zero member gives an absorbance comparable with the precision of the lowest calibration solution, background correction may be required.

8.3.4 Optimizing the atomic absorption spectrometer settings

Follow the manufacturer's instructions for preparing the instrument for use.

When the current of the lamp, the wavelength and the flow of gas have been adjusted and the burner lit, spray water until the indication has stabilized.

Set the absorbance value to zero using water.

Choose a damping setting or integration time to give a signal steady enough to fulfil the criteria of 6.5.1 to 6.5.3.

Adjust the flame to be non-luminous and oxidizing with an approximate 10 mm to 20 mm of red feather.

Alternately nebulize the calibration solution of highest concentration and the zero member (see Table 1), adjust the gas flow and burner position (horizontally, vertically and rotationally) until the difference in