International Standard

ISO 9658

Second edition
2024-09

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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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,* 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.

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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.

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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, 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.

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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.

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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.

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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.

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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

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absorbance between the calibration solutions is at a maximum. Check that the spectrometer is set accurately on the required wavelength.

Evaluate the criteria of 6.5.1 to 6.5.3 and the additional performance requirement of 6.5.4, to ensure that the instrument is suitable for the determination.

WARNING — The manufacturer's recommendations should be closely followed, and particular attention is drawn to the following hazards:

a) **acetylene forms explosive mixtures with air, it is presupposed that the user is aware of particular requirements from regulatory authorities;**

b) **the need to shield the eyes of the operator from ultraviolet radiation by means of tinted glass;**

c) **the need to keep the burner head clear of deposits because a badly clogged burner may cause a flashback;**

d) **the need to ensure that the liquid trap is filled with water;**

e) **the need to always spray water between the test solutions, blank solution and/or calibration solutions.**

8.3.5 Spectrometric measurements

Set the scale expansion so that the calibration solution of highest concentration gives nearly full-scale deflection. Nebulize the calibration solutions in ascending order repetitively until each gives the specified precision, thus showing that the instrument has achieved stability. Select two calibration solutions, one having an absorbance just lower than that of the test solution and one just higher. Nebulize these first in ascending order, then in descending order, with the test solution as the middle solution, in each case measuring the absorbance in relation to water.

Nebulize the complete range of calibration solutions, including the zero member. Then nebulize again in ascending and descending order. The means of the last ascending and descending series of calibration solutions are used for the calibration graph.

It is recognized that these procedures cannot be followed with automatic instruments which accept two calibration solutions only. In this case, it is suggested that the two "bracketing" solutions should not be used for the primary calibration but should be analysed alternately with the test solution.

Nebulize the calibration solutions at frequent intervals during the measurement of a batch of determinations. Clean the burner if the results show a loss of precision caused by clogging.

Obtain the mean absorbance of the test solution and the mean absorbance of the blank solution.

Procedures for the determination of instrumental criteria are given in Annex A.

8.4 Plotting the calibration graph

It is necessary to prepare a new calibration graph for each series of determinations, and for the range of aluminium contents expected.

Use an appropriate spectrometer software or an off-line computer for regression calculations or prepare a graphical representation.

If pure metals and reagents have been used, the blank test and zero member should give very small absorbance readings with a negligibly small difference. In this case, prepare a calibration graph by plotting the mean absorbance values of the calibration solutions against the concentrations of aluminium, expressed in micrograms per millilitre. Refer the mean test solution absorbance and the absorbances of the two adjacent calibration solutions to the graph.

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If, however, the zero member has a significant absorbance, a more complicated procedure is required. In this case, the concentration of aluminium ρ_z in the zero member can be calculated using the Formula (1):

$$\rho_z = \rho_{c1} \times \frac{A_z}{A_{c1} - A_z} \tag{1}$$

where

ρ_{c1} is the concentration of aluminium, expressed in micrograms per millilitre, added to the first calibration solution;

A_z is the absorbance of the zero member;

A_{c1} is the absorbance of the first calibration solution.

The derived value ρ_z is then added to each of the nominal calibration concentrations in order to obtain a mean calibration graph passing through the origin. Refer the absorbances of the blank solution, the test solution and the two adjacent calibration solutions to this graph. Subtract the concentration of the blank solution from the other concentrations.

Prepare a calibration graph by plotting the absorbance values of the calibration solutions against the concentrations of aluminium expressed in micrograms per millilitre. Refer the absorbances of the two adjacent calibration solutions to the graph. If these two calibration readings do not deviate from the graph by more than the permitted precision criteria, then the test solution readings are also acceptable.

9 Expression of results

9.1 Method of calculation

Convert the absorbance of the test solution and the blank solution to micrograms of aluminium per millilitre by reference to the calibration graph (8.4).

The aluminium content w_{Al}, expressed as a percentage by mass, is given by the Formula (2):

$$w_{Al} = \frac{(\rho_{Al,1} - \rho_{Al,0}) \times 100}{10^6} \times \frac{100}{m} = \frac{\rho_{Al,1} - \rho_{Al,0}}{100m} \tag{2}$$

where

$\rho_{Al,1}$ is the concentration, expressed in micrograms per millilitre, of aluminium in the test solution (8.3.1);

$\rho_{Al,0}$ is the concentration, expressed in micrograms per millilitre, of aluminium in the blank solution (8.2);

m is the mass, in grams, of the test portion (8.1).

9.2 Precision

A planned trial of this method was carried out by 28 laboratories. All laboratories made three determinations of each of the test samples.

NOTE 1 Two of the three determinations were carried out under repeatability conditions as defined in ISO 5725-1, i.e. one operator, same apparatus, identical operating conditions, same calibration, and a minimum period of time.

NOTE 2 The third determination was carried out at a different time (on a different day) by the same operator as in NOTE 1 above using the same apparatus with a new calibration.

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The test samples used are listed in Annex B.

The results obtained were treated statistically in accordance with ISO 5725-2 and ISO 5725-3.

The data obtained showed a logarithmic relationship between aluminium content and repeatability limit (r) and reproducibility limits (R_w and R) of the test results, as summarized in Table 3. The graphical representation of the data is shown in Figure C.1 and Figure C.2 of Annex C.

Table 3 — Precision data

Aluminium content % (mass fraction)	**Acid-soluble aluminium** % (mass fraction)			**Total aluminium** % (mass fraction)		
	r	R_w	R	r	R_w	R
0,005	0,000 7	0,001 4	0,002 5	0,001 1	0,001 3	0,002 1
0,01	0,001 0	0,001 9	0,003 4	0,001 4	0,001 7	0,002 8
0,02	0,001 5	0,002 6	0,004 7	0,001 8	0,002 3	0,003 8
0,05	0,002 5	0,004 0	0,007 2	0,002 5	0,003 3	0,005 6
0,1	0,003 6	0,005 5	0,009 1	0,003 2	0,004 4	0,007 5
0,2	0,005 2	0,007 6	0,013 6	0,004 1	0,006 0	0,010 1

10 Test report

The test report shall include the following information:

a) all information necessary for the identification of the sample, the laboratory and the date of analysis;

b) the method used by reference to this document, i.e. ISO 9658;

c) the results, and the unit in which they are expressed;

d) any unusual features noted during the determination;

e) any operation not specified in this draft, or any optional operation which may have influenced the results.

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Annex A
(informative)

Procedures for the determination of instrumental criteria

A.1 Determination of minimum precision

Nebulize the most concentrated calibration solution 10 times to obtain 10 individual absorbance readings A_{Ai} and calculate the mean value $\bar{A}_A$.

Nebulize the least concentrated calibration solution (excluding the zero member) 10 times to obtain 10 individual absorbance readings A_{Bi} and calculate the mean value $\bar{A}_B$.

The standard deviations s_A and s_B of the most and the least concentrate calibration solutions respectively are obtained from the Formula (A.1):

$$S_A = \sqrt{\frac{\sum (A_{Ai} - \bar{A}_A)^2}{9}}$$
$$S_B = \sqrt{\frac{\sum (A_{Bi} - \bar{A}_B)^2}{9}} \quad \text{(A.1)}$$

The minimum precisions of the most and least concentrate and calibration solutions are obtained from $s_A \times 100 / \bar{A}_A$ and $s_B \times 100 / \bar{A}_A$, respectively.

A.2 Determination of limit of detection, $\rho_{Al,\min}$

Prepare two solutions each containing the same matrix concentration as the sample solution, but with the element of interest at the following known concentrations:

— ρ'_{Al} µg/ml to give an absorbance A' of approximately 0,01;

— matrix blank to give an absorbance A_0.

Nebulize the ρ'_{Al} solution and blank solution 10 times each and record each reading for about 10 s with sufficient scale expansion to make the fluctuations in signal clearly visible.

Obtain the mean absorbance readings $\bar{A}'$ and $\bar{A}_0$.

The standard deviation $s_{A'}$ is given by the Formula (A.2):

$$S_{A'} = \sqrt{\frac{\sum (A'_i - \bar{A}')^2}{9}} \quad \text{(A.2)}$$

where

A'_i is the individual measured absorbance reading;

$\bar{A}'$ is the mean value of A'_i.

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The limit of detection $\rho_{Al,\min}$ is given by the Formula (A.3):

$$\rho_{Al,\min} = \frac{\rho'_{Al} \times S_{A'} \times k}{\bar{A}' - \bar{A}_0} \quad \text{(A.3)}$$

(k is normally taken as 4,65)

A.3 Criterion for calibration linearity

Having established the calibration graph, before the application of any curve-straightening device, obtain the net absorbance value A_A, corresponding to the top 20 % of the concentration range and the net absorbance A_B corresponding to the bottom 20 % of the concentration range. Calculate A_A / A_B. This shall not be less than 0,7.

A.4 Determination of characteristic concentration $\rho_{Al,K}$

Prepare a solution containing the same matrix concentration as the sample solution, but with the element of interest at the following known concentration:

ρ_{Al} µg/ml to give an absorbance A of approximately 0,1.

Nebulize the ρ_{Al} and the blank solutions without scale expansion and measure the absorbances A and A_0. The characteristic concentration $\rho_{Al,K}$ is given by the Formula (A.4):

$$\rho_{Al,K} = \frac{\rho_{Al} \times 0{,}004\,4}{A - A_0} \quad \text{(A.4)}$$

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Annex B
(informative)

Additional information on the international cooperative test

Analytical trials were carried out in 1985 on six steel samples in nine countries involving 28 laboratories. Table 3 shows the precision data obtained from their re-evaluation carried out in 2021.

The graphical representation of the precision data is given in Annex C.

The test samples used are listed in Table B.1 and the experimental precision data are listed in Table B.2.

Table B.1 — Composition of test samples used on the international cooperative test

Sample	Chemical composition % (mass fraction)								
	Total Al	Acid insoluble Al[a]	C	Si	Mn	Cu	Ni	Cr	V
BAM 038-1	0,002[b]	-	0,159	0,28	0,628	0,021	-	-	-
BCS 458/1	0,009	-	0,101	0,24	0,20	-	-	-	0,2
BAM 035-1	0,028	0,007	1,31	0,21	0,26	0,071	0,038	0,071	-
NBS 65d	0,059	0,004 8[c]	0,264	0,37	0,73	0,051	0,060	0,049	-
BCS457/1	0,11	-	0,30	0,05	0,30	-	-	-	0,17
IPT-39	0,169	-	0,048	0,061	0,34	0,019	0,048	0,084	-

[a] Non-certified value.

[b] Acid-insoluble aluminium. The acid mixtures used to determine these acid-insoluble values may not be the same as the acid mixture used in this document.

[c] 0,009 % (mass fraction) as Al_2O_3

Table B.2 — Experimental precision data

Aluminium content % (mass fraction)	Acid-soluble aluminium % (mass fraction)			Aluminium content % (mass fraction)	Total aluminium % (mass fraction)		
	r	R_w	R		r	R_w	R
0,001 3	0,000 4	0,001 0	0,001 7	0,002 0	0,000 7	0,000 9	0,001 8
0,008 0	0,000 7	0,001 2	0,002 2	0,009 4	0,001 6	0,001 6	0,002 3
0,021 8	0,001 3	0,002 0	0,004 2	0,026 0	0,001 7	0,002 0	0,003 4
0,052 8	0,002 6	0,004 4	0,007 0	0,058 9	0,003 1	0,004 5	0,005 7
0,102 5	0,005 8	0,005 8	0,011 1	0,109 2	0,002 8	0,004 1	0,008 2
0,165 6	0,003 9	0,008 8	0,014 7	0,171 8	0,004 2	0,005 9	0,011 3

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