SLOVENSKI STANDARD
SIST EN 29658:1997

01-december-1997

Jeklo - Določevanje aluminija - Metoda s plamensko atomsko apsorpcijsko sprektrometrično metodo (ISO 9658:1990)

Steel - Determination of aluminium content - Flame atomic absorption spectrometric method (ISO 9658:1990)

Stahl - Bestimmung des Aluminiumgehalts - Spektralfotometrische Atomabsorptionsmethode (ISO 9658:1990)

Aciers - Dosage de l'aluminium - Méthode par spectrométrie d'absorption atomique dans la flamme (ISO 9658:1990)

Ta slovenski standard je istoveten z: **EN 29658:1991**

ICS:

77.040.30	Kemijska analiza kovin	Chemical analysis of metals
77.080.20	Jekla	Steels

SIST EN 29658:1997 **en**

EUROPEAN STANDARD

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English version

Steel - Determination of aluminium content - Flame atomic absorption method after combustion in an induction furnace (ISO 9658:1990)

Aciers - Dosage de l'aluminium - Méthode par spectrométrie d'absorption atomique dans la flamme (ISO 9658:1990)

Stahl - Bestimmung des Aluminiumgehalts - Spektralfotometrische Atomabsorptionsmethode (ISO 9658:1990)

This European Standard was approved by CEN on 1991-12-20 and is identical to the ISO standard as referred to.
CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

Foreword

On the proposal of the Technical Committee ECISS/TC 20 the Coordinating Commission (COCOR) of the European Committee for Iron and Steel Standardization (ECISS) decided in May 1991 to submit the International Standard

ISO 9658:1990 Steel - Determination of aluminium content - Flame atomic absorption method after combustion in an induction furnace

to the formal vote.

This European Standard EN 29658 was approved by CEN on 1991-12-20.

According to the CEN/CENELEC Internal Regulations, the follwing countries are bound to implement this European Standard :

Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 9658:1990 was approved by CEN as a European Standard without any modifications.

INTERNATIONAL STANDARD

ISO 9658

First edition
1990-03-01

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

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

Reference number
ISO 9658:1990(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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 9658 was prepared by Technical Committee ISO/TC 17, *Steel*.

Annex A forms an integral part of this International Standard. Annexes B and C are for information only.

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

1 Scope

This International Standard 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 % (*m*/*m*) and 0,20 % (*m*/*m*).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 377:1989, *Selection and preparation of samples and test pieces of wrought steels — Part 1: Samples and test pieces for mechanical test.*

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 648:1977, *Laboratory glassware — One-mark pipettes.*

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks.*

ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

3 Definition

For the purposes of this International Standard, the following definition applies.

acid-soluble aluminium: Aluminium dissolved in the acid mixture as specified in 8.3.1.1.

4 Principle

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

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

Spraying 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.

5 Reagents

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

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

5.2 Hydrofluoric acid, ρ about 1,15 g/ml.

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

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

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

5.6 Acid mixture.

Mix 3 parts by volume of hydrochloric acid (ρ about 1,19 g/ml), 1 part by volume of nitric acid (ρ about 1,40 g/ml) and 2 parts by volume of water.

Prepare this mixture 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 solutions.

5.9.1 Stock solution, corresponding to 2,0 g of Al per litre.

Weigh, to the nearest 0,001 g, 2,000 g of high purity aluminium [≥ 99,9 % (*m*/*m*) pure], 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 to 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.

5.9.2 Standard solution A, corresponding to 0,20 g of Al per litre.

Transfer 20,0 ml of the stock 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 before use.

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

5.9.3 Standard solution B, corresponding to 0,020 g of Al per litre.

Transfer 20,0 ml of the standard solution A (5.9.2) into a 200 ml one-mark volumetric flask. Dilute to the mark with water and mix. Prepare this standard solution immediately before use.

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

6 Apparatus

All volumetric glassware shall be class A, in accordance with ISO 385-1, ISO 648 or ISO 1042 as appropriate.

All glassware shall be cleaned with hot hydrochloric acid (5.3) and then water.

Ordinary laboratory apparatus, and

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 for insertion of the rubber stopper of the filter funnel stem.

6.4 Platinum crucible, of capacity 30 ml.

6.5 Atomic absorption spectrometer.

An aluminium hollow cathode lamp; supplies of dinitrogen monoxide and acetylene sufficiently pure to give a steady clear red-feather 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 if it meets the precision criteria given in 6.5.1 to 6.5.3.

It is also desirable that the instrument should conform to the additional performance requirement given in 6.5.4 .

6.5.1 Minimum precision (see clause A.1).

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

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

6.5.2 Limit of detection (see clause A.2).

This is defined as twice the standard deviation of 10 measurements of the absorbance of a solution containing the appropriate element at a concentration level selected to give an absorbance just above that of the zero member.

The limit of detection of aluminium in a matrix similar to the final test portion solution shall be better than 0,1 µg of Al per millilitre.

6.5.3 Graph linearity (see clause A.3).

The slope of the calibration graph 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 standards, it shall be established prior to the analysis, by obtaining absorbance readings, that the above requirements for graph linearity are fulfilled.

6.5.4 Characteristic concentration
(see clause A.4).

The characteristic concentration for aluminium in a matrix similar to the final test portion solution shall be better than 1,0 µg of Al per millilitre.

6.6 Ancillary equipment.

A strip chart recorder and/or digital readout device is recommended to evaluate the criteria of 6.5 and for all subsequent measurements.

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 simply dividing the signal obtained.

7 Sampling

Carry out sampling in accordance with ISO 377 or appropriate national standards for steel.

8 Procedure

8.1 Test portion

Weigh, to the nearest 0,001 g, approximately 2,0 g of the test sample.

8.2 Blank test

Parallel with the determination and following the same procedure, carry out a blank test using the same quantities of all the reagents without the addition of pure iron.

Background correction may be required.

8.3 Determination

8.3.1 Preparation of the test solution

8.3.1.1 Decomposition of the test portion

Place the test portion (8.1) in a 250 ml beaker. Add, in small portions, 40 ml of acid mixture (5.6) and cover the beaker with a watch-glass. Heat until solvent 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 suppport 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 in a 500 ml vacuum vessel:

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

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