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Evropski certificirani referenčni materiali (EURONORM-CRMs) za ugotavljanje kemijske sestave železnih in jeklenih izdelkov, pripravljeni pod okriljem Evropskega komiteja za standardizacijo železa in jekla (ECISS)

European certified reference materials (EURONORM-CRMs) for the determination of the chemical composition of iron and steel products prepared under the auspices of the European Committee for Iron and Steel Standardization (ECISS)

Europäische zertifizierte Referenzmaterialien (EURONORM-ZRMs) für die Bestimmung der chemischen Zusammensetzung von Eisen und Stahl Produkten, hergestellt unter der Schirmherrschaft des Europäischen Komitees für Eisen- und Stahlnormung (ECISS)

Matériaux de référence certifiés européens (EURONORM-MRC) destinés à la détermination de la composition chimique des produits ferreux élaborés sous les auspices du Comité européen de normalisation du fer et de l'acier (ECISS)

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77.040.30	Kemijska analiza kovin	Chemical analysis of metals
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English Version

European certified reference materials (EURONORM-CRMs) for the determination of the chemical composition of iron and steel products prepared under the auspices of the European Committee for Iron and Steel Standardization (ECISS)

Matériaux de référence certifiés européens (EURONORM-MRC) destinés à la détermination de la composition chimique des produits en acier et en fonte préparés sous les auspices du comité européen de normalisation du fer et de l'acier (ECISS)

Europäische zertifizierte Referenzmaterialien (EURONORM-ZRMs) für die Bestimmung der chemischen Zusammensetzung von Eisen- und Stahlerzeugnissen, hergestellt unter der Schirmherrschaft des Europäischen Komitees für Eisen- und Stahlnormung (ECISS)

This Technical Report was approved by CEN on 27 August 2012. It has been drawn up by the Technical Committee ECISS/TC 102.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (CEN/TR 10317:2013) has been prepared by Technical Committee ECISS/TC 102 “Methods of chemical analysis for iron and steel”, the secretariat of which is held by SIS.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes CEN/TR 10317:2009.

In comparison with the previous edition of CEN/TR 10317:2009, the following significant technical changes were made:

— Clause 2, definition of Certified Reference Material;

— in 5.1, change of the procedure how to carry out the four required determination.

Introduction

In accordance with the definition in ISO Guide 30, Amendment 1:2008, a Certified Reference Material (CRM) described in this CEN Technical Report is a "reference material characterized by a metrologically valid procedure for one or more specified properties, accompanied by a certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability". Furthermore, it is accompanied by a certificate issued by the producing organisation on behalf of the Co-ordinating Committee (COCOR) of ECISS after approval by the participating laboratories and all the producing organisations:

a) France:

   1) ArcelorMittal Maizières [former Institut de Recherches de la Sidérurgie Française (IRSID)],

   2) Centre Technique des Industries de la Fonderie (CTIF);

b) Germany: Iron and Steel CRM Working Group (AGZRM) comprising:

   1) BAM Bundesanstalt für Materialforschung und -prüfung,

   2) Max-Planck-Institut für Eisenforschung (MPI),

   3) Stahlinstitut VDEh;

c) Nordic Countries: Nordic CRM Working Group, (NCRMWG) comprising:

   1) Swerea KIMAB [former Swedish Institute for Metals Research (SIMR)],

   2) Jernkontoret;

d) United Kingdom: Bureau of Analysed Samples Limited (BAS).

Since 1968 EURONORM-CRMs have been analysed by laboratories in most countries in the European Union (EU) or former European Community (EC).

Pending their eventual replacement by EURONORM-CRMs, a number of former national CRMs prepared, analysed and certified by laboratories in Germany, France and the United Kingdom respectively, were accepted as EURONORM-CRMs after their accuracy had been checked by other European laboratories. This procedure ceased in 1990.

1 Scope

This Technical Report describes the classification, method of sample preparation, certification main rules and certificate content of the EURONORM-CRMs.

It also lists the sample presentation of the corresponding producer's organisations and the distributing sources.

2 Classification of EURONORM-CRMs

EURONORM-CRMs, prepared under the auspices of ECISS, are classified into two main groups:

— cast and wrought materials: irons, steels, special alloys and ferro-alloys;

— non-metallic materials: raw materials (ores, concentrates, additives and refractories) and by-products (slags, dusts and similar materials).

Besides this first generic classification, EURONORM-CRMs are grouped into the following categories:

a) **From 001 to 099 – high purity irons and unalloyed steels**

Normally no element has a mass content greater than the limit values in the following list:

1) silicon, limit value 1,0 %;

2) manganese, limit value 1,5 %;

3) chromium and nickel, limit value for each 0,5 %;

4) cobalt, copper and tungsten, limit value for each 0,3 %;

5) other elements, limit value for each 0,10 %;

6) boron, carbon, phosphorus, lead and sulphur, no limit value.

b) **From 101 to 199 – low alloy steels**

The content of one or more elements is greater than the limit for unalloyed steels but none exceeds 5 %. The sum of these alloying elements remains under 10 %.

c) **From 201 – 299 – highly alloyed steels**

The content of one or more elements is greater than 5 % or the sum of all these alloying elements is at least 10 %. Nevertheless the iron content will normally be greater than 50 %.

d) **From 301 to 399 - special alloys**

The iron content is less than 50 %.

e) **From 401 to 499 – pig irons and cast irons**

f) **From 501 to 599 – ferro-alloys**

g) **From 601 to 699 – ores, concentrates, sinters and miscellaneous materials**

h) **From 701 to 799 – additives and refractories**

i) **From 801 to 899 – by-products, such as slags, dusts and similar materials**

The expression "-1" after a CRM number refers to the first issue of a CRM and "-2", "-3" etc. refer to replacement CRMs of generally similar composition, but not to a further bottling of the original CRM.

NOTE This classification is used as a simple and convenient method of differentiating between samples of different types of materials. It has no other objective and does not replace existing European or International product specifications for metallic materials.

3 Preparation of the samples

3.1 EURONORM-CRMs of cast and wrought materials

3.1.1 High purity irons, steels and special alloys

The material is obtained as cylindrical, square or rectangular billets or slabs. After the homogeneity has been verified, it is machined dry (avoiding excessive heat generation) on a suitable lathe or milling machine in order to produce short chips; the chips are screened so that the fines can be removed. Some steel samples are produced by atomisation of the liquid metal.

The whole preparation process provides a batch of generally more than 100 kg. This is blended in a cylindrical or cube shaped mixer before certification analysis.

Samples supplied in disc form are prepared from the most homogeneous parts of the rolled or forged material. If the finely divided sample is prepared by atomisation of the liquid metal, disc samples may be prepared by hot isostatic pressing of the powder. This process has been used where it is difficult to obtain homogeneous solid material for spectroscopic analysis by casting or forging.

In all cases it is verified that the solid and finely divided samples are identical in chemical composition.

3.1.2 Pig irons and cast irons

The metal in the form of solid or hollow cylinders is descaled; its homogeneity is verified and then it is dry machined on a lathe. The turnings are separated into three sieve fractions, the dust being continually drawn off. Only the middle fraction is retained and homogenised before certification analysis and bottling.

Some iron samples are produced by crushing the solid material or by atomisation of the liquid metal.

3.1.3 Ferro-alloys

A bulk supply (usually more than 100 kg) of ferro-alloy material taken from production is crushed and ground to a suitable particle size.

As there is the possibility with such material for the composition to vary with particle size, homogeneity checks are carried out on several size fractions of the bulk material.

Only those size fractions which are sufficiently similar to each other with respect to chemical composition are selected, and these are blended in a cube shaped or cylindrical mixer made of stainless steel to produce the homogeneous product to be used to produce the CRM.

3.2 EURONORM-CRMs of non-metallic materials

3.2.1 Ores, Concentrates, Sinters and Miscellaneous Materials: Additives and Refractories

The material is selected in such a way that it has the best composition and storage properties. After drying, crushing and grinding, it is screened to a suitable particle size and blended in a cube shaped or cylindrical mixer made of stainless steel.

The bulk sample is carefully stored in airtight containers. Where necessary a moisture absorber is placed inside the container.

Throughout the whole preparation process the homogeneity is verified.

3.2.2 By-products, such as slags, dusts and similar materials

The same rules apply for the preparation of the samples as for materials listed under 4.2.1. The certification analysis can only be carried out after a long stabilisation period, during which the composition is verified by means of numerous check analyses.

4 Certification

4.1 Main rules

The certification of these EURONORM-CRMs is performed following the established guidelines in ISO Guides 30, 31, 34 and 35 and the standard ISO 11459 (see Bibliography).

Chemical characterisation of a candidate material is carried out in a certification interlaboratory comparison with approximately 20 laboratories from industry and research in Europe.

Each laboratory is requested to analyse the elements to be determined four times under repeatability conditions but on separate portions of the sample, using a suitable analytical method of its choice.

A statistical evaluation is carried out on all the individual values obtained for each element in order to confirm that they are distributed normally about the overall mean and to identify any outlying values.

The aim, for certification of all elements or constituents is that, after elimination of statistical outliers, there remain at least 14 acceptable mean values, if possible achieved from different analytical methods.

Certification with less than 14 acceptable mean values, but not less than ten, is also possible in situations where the concentration of the certified constituent is less than 10 µg/g or where the constituent is considered difficult to analyse.

4.2 Main content of the certificates

4.2.1 General

Each certificate presents a table, in which the laboratory means are presented together with the mean of the intralaboratory means, M_M, the standard deviation of the intralaboratory means, S_M and the intralaboratory standard deviation, S_w.

The half-width confidence interval C (95 %) of the certified mass content (also reported in each certificate) is calculated from the formula:

$$C(95\ \%) = \frac{t \times S_M}{\sqrt{n}}$$