PD CEN/TR 10317:2020

BSI Standards Publication

European certified reference materials (EURONORM-CRMs) for the determination of the chemical composition of iron and steel products

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National foreword

This Published Document is the UK implementation of CEN/TR 10317:2020. It supersedes PD CEN/TR 10317:2014, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee ISE/102, Methods of Chemical Analysis for Iron and Steel.

A list of organizations represented on this committee can be obtained on request to its committee manager.

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Published by BSI Standards Limited 2020

ISBN 978 0 539 04169 9

ICS 71.040.30; 77.140.01; 77.150.10

Compliance with a British Standard cannot confer immunity from legal obligations.

This Published Document was published under the authority of the Standards Policy and Strategy Committee on 31 July 2020.

Amendments/corrigenda issued since publication

Date	Text affected

TECHNICAL REPORT

RAPPORT TECHNIQUE

TECHNISCHER BERICHT

CEN/TR 10317

July 2020

ICS 71.040.30; 77.140

Supersedes CEN/TR 10317:2014

English Version

European certified reference materials (EURONORM-CRMs) for the determination of the chemical composition of iron and steel products

Matériaux de référence certifiés européens (EURONORM-MRC) destinés à la détermination de la composition chimique des produits en acier et en fonte

Europäische zertifizierte Referenzmaterialien (EURONORM-ZRM) für die Bestimmung der chemischen Zusammensetzung von Eisen und Stahlerzeugnissen

This Technical Report was approved by CEN on 22 June 2020. It has been drawn up by the Technical Committee CEN/TC 459/SC 2.

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Ref. No. CEN/TR 10317:2020 E

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European foreword

This document (CEN/TR 10317:2020) has been prepared by Technical Committee CEN/TC 459/SC 2 (former 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 shall not be held responsible for identifying any or all such patent rights.

This document supersedes CEN/TR 10317:2014.

In comparison with the previous edition, CEN/TR 10317:2014, the following technical modifications have been made:

— title changed;

— deletion of all references to COCOR and ECISS;

— updating of the addresses of some of the producers;

— Bibliography updated.

Introduction

In accordance with the definition in ISO Guide 30, a Certified Reference Material (CRM) as described in this document is a "reference material (RM) 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 organization, after approval by the participating laboratories and all the producing organizations listed below:

a) France:

   1) ArcelorMittal Maizières Research SA (AMMX) [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) Bundesanstalt für Materialforschung und -prüfung (BAM),

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

   3) Stahlinstitut VDEh;

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

   1) Oy Narema AB,

   2) Jernkontoret.

NOTE Bureau of Analysed Samples Ltd (BAS) – United Kingdom – was a member of the EURONORM-CRM Producer's Group from 1975 until 2013.

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

It also details the sample presentation of the various producers' organizations and the distributing sources.

2 Classification of EURONORM-CRMs

EURONORM-CRMs, 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 content (mass fraction) 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 0,1 %;

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 given 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 this 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 spectrometric 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 homogenized 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 3.2.1. The certification analysis can only be carried out after a long stabilization period, during which the composition is verified by means of numerous check analyses.

4 Certification

4.1 Main rules

The certification of EURONORM-CRMs is conducted following the guidelines in ISO Guide 30, ISO Guide 31, and ISO Guide 35 and the standards EN ISO 17034 and ISO 11459 (see Bibliography).

Chemical characterization of a candidate material is carried out in a certification interlaboratory test 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 each element or constituent 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 10, is also possible in situations where the content of the certified constituent is less than 10 µg/g or where the constituent and/or the matrix 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 Formula (1):

$$C(95\ \%) = \frac{t \times S_M}{\sqrt{n}} \qquad (1)$$

where

- n is the number of acceptable mean values;
- t is the Student's value at the chosen probability with n-1 degrees of freedom.

Certificates also contain a table summarizing all the methods used. Suitable numbers in this table allow the users to identify the method corresponding to each value reported on the table of the laboratory means, named above.

The list of the laboratories having participated on each certification analysis is given on the certificate of the corresponding EURONORM-CRM.

The other statements which are also always included in each certificate are described in 4.2.2, 4.2.3, 4.2.4 and 4.2.5.

4.2.2 Description of the sample

The finely divided EURONORM-CRMs are supplied in glass bottles containing 100 g. These are annotated by the suffix "(C)". Information about the particle size of the divided sample is also reported.

Some EURONORM-CRMs are also available in the form of solid discs normally approximately 38 mm in diameter × 20 mm to 30 mm thick. These are annotated by the suffix "(D)".

4.2.3 Intended use and stability

In the chip form samples are intended for the verification of analytical methods such as those used by the participating laboratories (including establishing the validity of a new analytical method and/or a standard method under development), for the calibration of analytical instruments in cases where the calibration with primary substances (pure metals or stoichiometric compounds) is difficult to achieve, and for establishing values for secondary reference materials. They will remain stable, provided that bottles remain sealed and are stored in a cool and dry atmosphere. When the bottle has been opened the lid should be secured immediately after use. If the contents should become discoloured (e.g. oxidized) due to atmospheric contamination they should be discarded.

Disc or block form samples are intended for establishing and checking the calibration of optical emission and X-ray spectrometers for the analysis of samples of similar materials. The "as received" working surface of the sample should be linished before use to remove any protective coating. It will remain stable provided that it is not subjected to excessive heat (e.g. during preparation of the working surface).

4.2.4 Traceability

The traceability of EURONORM-CRMs is established in accordance with principles of Guides 30, 31, and 35 and the standard EN ISO 17034, together with the International vocabulary of basic and general terms in metrology.

The assigned values for each material are achieved by inter-laboratory characterization, each laboratory using the method of their choice, details of which are given in each certificate. These methods are either stoichiometric analytical techniques or methods which are calibrated against pure metals or stoichiometric compounds. Most methods used are either international or national standard methods or methods which are technically equivalent.

4.2.5 Further information

This information, reported in each certificate in the three languages of the producers' organizations, independently of the national language of the producer of the EURONORM-CRM under concern and in English, refers to the present document, as well as to the CEN/TR 10350.

5 Sample presentation

Each sample is placed in a cardboard carton of different colour according to the producer, as follows:

— BAM (Germany) White
— CTIF (France) Salmon
— AMMZ (France) Green
— Oy Narema AB (Nordic Countries) Yellow

NOTE Samples from BAS are placed in blue cartons.

6 Distribution of EURONORM-CRMs

EURONORM-CRMs are available from the following:

BAM Bundesanstalt für Materialforschung und -prüfung

Richard-Willstätter-Straße 11

DE-12489 Berlin-Adlershof, GERMANY

Telephone:	+49 30 8104 2061
Fax:	+49 30 8104 72061
Internet:	http://www.bam.de
	http://www.webshop.bam.de
E-mail	sales.crm@bam.de

Bureau of Analysed Samples Limited (BAS)

Newham Hall, Newby

Middlesbrough, TS8 9EA

UNITED KINGDOM

Telephone:	+44 1642 300500
Fax:	+44 1642 315209
Internet:	http://www.basrid.co.uk
E-mail	enquiries@basrid.co.uk

Centre Technique des Industries de la Fonderie (CTIF)

44, avenue de la Division Leclerc

92318 – Sèvres Cedex, FRANCE

Telephone:	+33 (0)1 41 14 63 00
Fax:	+33 (0)1 45.34.14.34
Internet:	http://www.ctif.com
E-mail	contact@ctif.com

Oy Narema AB

Närpiöntie 2,

FIN-64200 Närpiö, FINLAND

Telephone:	+358 (0)50 378 91 92
Internet:	http://narema.fi
E-mail	crm@narema.fi

TECHLAB

4C La Tannerie

B.P. 30055 – St. Julien-lès-Metz

57072 METZ Cedex 3, FRANCE

Telephone:	+33 3 87 75 54 29
Fax:	+33 3 87 36 23 90
Internet:	http://www.techlab.fr
E-mail	techlab@techlab.fr

7 Details of current EURONORM-CRMs

The list of currently available EURONORM-CRMs constantly changes due to existing samples becoming exhausted and new or replacement samples being produced. Users are therefore invited to apply to the producers or their distributors for their current catalogues.

Bibliography

[1] ISO Guide 30:2015, *Reference materials — Selected terms and definitions*

[2] ISO Guide 31:2015, *Reference materials — Contents of certificates, labels and accompanying documentation*

[3] EN ISO 17034:2016, *General requirements for the competence of reference material producers (ISO 17034:2016)*

[4] ISO Guide 35:2017, *Reference materials — Guidance for characterization and assessment of homogeneity and stability*

[5] ISO 11459:1997, *Iron ores — Certified reference materials — Preparation and certification for use in chemical analysis*

[6] ISO/IEC GUIDE 99:2011, *International vocabulary of metrology — Basic and general concepts and associated terms (VIM)*

[7] CEN/TR 10350:2013, *Analysis of steels and irons — Internal laboratory procedure for checking the accuracy of an analytical method by using Certified Reference Materials*

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