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Supervised by Section of Fundamental Metrology, Certification Body for Reference Materials (CORM)
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CERTIFICATE

0217-CM-20034-14

CERTIFIED REFERENCE MATERIALS CZ 20034

Cast iron for solid-sample spectrometry, CRM set 11 - 17

Issued on: April 24, 2014

Valid till: April 24, 2029

Prepared by melting and temperature- and speed-controlled casting to gangs of chill-cast discs, cooled on both vertically positioned plan-parallel sides. This resulted in a fine white (i.e. effectively graphite free) structure required for solid-sample spectrometry, and perfectly matching that of routine foundry samples. The discs of 40 mm in diameter were water-jet separated and their cylindrical surface finished and marked. Layers of about 0.5 mm were machined off from both cooled surfaces to yield discs with a minimum ultimate height of 18 mm.

The composition of seven CRM was designed to cover optimally typical concentration ranges of all metallurgically relevant elements in plain and low-alloy cast iron. Values are graded dynamically and in each CRM they are combined in order to avoid excessive interferences, as well as unfavorable effects on the ultimate structure. The main matrix element, Fe, is kept in a narrow concentration range of 90.2 – 92.7%. Consequently, the CRM do not represent any particular cast iron grades.

The CRM are distributed as a set or individually.

Intended for calibration and validation of methods of cast iron analysis by spectrometry from a plane of solid sample: Atomic Emission Spectrometry with spark, glow-discharge or laser excitation and X-Ray Fluorescence Spectrometry.

An analysis area of at least 4 mm in diameter defines the minimum sample intake.

Production testing and characterization were carried out in accordance with the methodical procedure CORM ČMI 017-MP-C001 and in compliance with the ISO Guides 34, 35.

The producer shall ensure due conditions of storage and distribution and shall monitor the CRM parameters and feed-back from users during the entire validity period.

Producer SPL Bohumín, 1. máje 432, CZ 73531, Bohumín, Czech Republic, www.spl-bohumin.cz.

Responsible person: Vladimír Bogumský, phone +420 596 014 627

CORM deputy head:

Ing. Jan Beránek

Český metrologický institut

Head of CORM:

RNDr. Pavel Klenovský

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Homogeneity of both element content and the respective matrix influence was tested by spark excitation AES, the technique prevailing in the cast iron routine analysis, which is by coincidence the most strongly structure-influenced technique.

The limits of the certified layers were determined prior to many tests, in a safe distance from minor structural defect that may occur in central part of discs.

The certified values are valid for both plan-parallel working sides into depth of 6 mm, marked along with the CRM batch code.

The tests were carried out in accordance with ISO Guide 35 and ASTM E 826.

Within-sample trend homogeneity test compared the results on the opposite limits of the certified layer, i.e. on the original working surface and the surface after grinding off up to depth of 6 mm.

Between-samples trend homogeneity test compared the results of discs along the casting succession.

Their uncertainty contributions, unless found statistically insignificant and/or negligible, were included to the overall uncertainty budget.

Stability of CRM is given by the nature of their matrix. Validity of the Certificate is then limited only in view of possible future improvement of analytical methods applied in characterization.

Storage in a dry and non-corrosive environment is recommended.

User instruction The working surface of the CRM must be prepared before analysis in the same way as samples analyzed, in accordance with the particular analyzer manual.

A mean of at least three independent measurements is required for every metrological operation.

When used to both limits of the certified layers marked on side of discs, the remainder of CRM, which may contain minor structure defects, should be discarded.

There are no safety hazards in the storage and proper use of CRM.

Characterization by an international collaborative study (interlaboratory characterization experiment) involved various standard or validated methods and was conducted in compliance with ISO Guide 35 and ISO 17025.
The producer has been accredited for organizing and assessment of the interlaboratory experiments by the Czech Accreditation Institute under No 189/2011.

Traceability of the values was established to the values of the selected matrix-matching CRM: the sets CZ 2002, CZ 02033, CZ 2015-2024, selected NIST SRM and other. The results of the wet-way methods, wherever possible, were made traceable to pure substances or metals. No direct traceability to primary references was established due to lack of applicable methods and complexity of matrices. The values of the consecutive batches B, C were made traceable to the values of the reference batches A.

Methods of various analytical techniques were applied. Besides above specified solid-sample spectrometries there were combustion with IR MAS, volumetry and solution analysis by FASS, ET AAS, ICP AES, MAS (photometry), titrimetry and gravimetry.

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Laboratories participating in characterization were accredited and/or they demonstrated compliance with ISO 17025 during their participation. The laboratories involved in establishing the traceable values of the consecutive batches are marked by an asterisk.

ARCELORMITTAL OSTRAVA, Ostrava, Czechia*
BESKYD, Frýdlant nad Ostravicí, Czechia
BRAMMER STANDARD COMPANY, Houston, TX, USA
ENVIFORM, Třinec, Czechia*
INSTITUTE FOR CRM, Yekaterinburg, Russia
INSTYTUT METALURGII ŻELAZA, Gliwice, Poland
KIDAO LABORATORIES, Chennai, India
LITHEA, Brno, Czechia*
METAL AND QUALITY, PJSC, Zaporozhiye, Ukraine
OBLF, Witten, Germany
SECO GROUP, Jičín, Czechia*
TECHLAB, Metz, France
UNEX, Olomouc, Czechia*
US STEEL KOŠICE – LABORTEST, Košice, Slovakia
VÍTKOVICE TESTING CENTER, Ostrava, Czechia*
VOLVO POWERTRAIN CORP., SKÖVDE, Sweden
WELL GROUP SCIENTIFIC, Beijing , P.R. China
ZPS SLÉVÁRNA, Zlín, Czechia*
ŽĎAS, Žďár nad Sázavou, Czechia*

Data evaluation Five independent results for each value were reported by participants. Their means were first scrutinized technically to identify possible biases, justifying respective deletion. Then the distribution of the accepted means was assessed in order to choose between their arithmetic and robust (MAD median by van Monfort) mean for assigning the certified value.

Certified values of the reference batches A are means of a minimum of ten accepted laboratory means obtained by at least three different methods. The traceable values of batches B, C were established by a minimum of five laboratories by at least two methods. The values were certified on condition that their uncertainty was at or below target, pre-set as a fraction of respective uncertainty in Proficiency Tests and analytical standards.
The certified values are tabulated in bold, rounded consistently with their uncertainty statement given below in regular.

Non-certified indicative values, tabulated in regular without uncertainty statement, did not meet all above requirements for certification. They are intended only for proximate information about the matrix composition and they may not be used for calibration and other metrological operations. The empty boxes in the table indicate trace contents at or below 0.003%.

Uncertainty is combined from the uncertainty of the characterization experiment and homogeneity contribution, when applicable, and expanded by a coverage factor k=2. It is rounded in respect to uncertainty of its estimate thus it is expressed by maximum two valid figures.

Traceability of the values was established to the values of the selected matrix-matching CRM: the sets CZ 2002, CZ 02033, CZ 2015-2024, selected NIST SRM and other. The results of the wet-way methods, wherever possible, were made traceable to pure substances or metals. No direct traceability to primary references was established due to the lack of applicable methods and complexity of matrices. The values of the consecutive batches B, C were made traceable to the values of the reference batches A.

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Certified mass-fraction values with uncertainty and non-certified values, in %

	C	Mn	Si	P	S	Cr	Ni	Cu
11A	2.37 0.02	0.343 0.007	3.31 0.04	0.271 0.009	0.163 0.007	1.219 0.015	0.084 0.002	0.086 0.003
11B	2.44 0.02	0.382 0.008	3.67 0.04	0.271 0.009	0.140 0.007	1.178 0.016	0.082 0.002	0.130 0.003
12A	2.82 0.02	0.996 0.010	2.57 0.03	0.480 0.011	0.073 0.003	0.640 0.008	0.174 0.002	0.160 0.004
12B	2.92 0.02	1.047 0.011	2.96 0.03	0.484 0.011	0.077 0.003	0.638 0.008	0.174 0.002	0.223 0.005
13A	3.13 0.03	0.691 0.006	2.19 0.02	0.0244 0.0016	0.0046 0.0004	0.122 0.003	1.266 0.016	0.021 0.002
13B	3.12 0.03	0.692 0.006	2.12 0.02	0.0243 0.0017	0.0041 0.0004	0.125 0.003	1.313 0.017	0.021 0.002
13C	3.15 0.03	0.704 0.007	2.23 0.02	0.0261 0.0017	0.0044 0.0004	0.124 0.003	1.299 0.017	0.089 0.003
14A	3.29 0.02	0.218 0.003	2.25 0.02	0.0115 0.0011	0.0103 0.005	0.042 0.002	0.021 0.002	0.578 0.008
14B	3.26 0.02	0.240 0.003	2.34 0.02	0.0115 0.0011	0.0096 0.005	0.042 0.002	0.020 0.002	0.640 0.008
14C	3.14 0.02	0.275 0.003	2.49 0.02	0.0162 0.0011	0.0081 0.005	0.045 0.002	0.030 0.002	0.585 0.008
15A	3.54 0.03	0.051 0.002	1.68 0.02	0.054 0.003	0.0029 0.0003	0.070 0.002	0.661 0.008	1.322 0.017
15B	3.52 0.03	0.048 0.002	1.66 0.02	0.054 0.003	0.0031 0.0003	0.067 0.002	0.681 0.008	1.322 0.018
15C	3.47 0.03	0.060 0.002	1.68 0.02	0.054 0.003	0.0028 0.0003	0.078 0.003	0.728 0.009	1.123 0.018
16A	3.80 0.03	1.292 0.012	1.00 0.01	0.171 0.006	0.0266 0.0014	0.374 0.006	0.390 0.004	0.332 0.007
16B	3.78 0.03	1.327 0.013	1.00 0.01	0.170 0.006	0.0236 0.0014	0.378 0.006	0.388 0.005	0.332 0.007
16C	3.87 0.03	1.311 0.013	0.95 0.01	0.173 0.006	0.0243 0.0014	0.332 0.006	0.376 0.005	0.345 0.007
17A	4.30 0.04	0.494 0.005	0.170 0.008	0.115 0.005	0.0034 0.0004	0.200 0.004	2.38 0.03	0.082 0.004
17B	4.38 0.04	0.501 0.005	0.178 0.009	0.089 0.005	0.0040 0.0004	0.200 0.005	2.34 0.03	0.111 0.005
17C	4.08 0.04	0.503 0.005	0.150 0.008	0.104 0.005	0.0033 0.0004	0.178 0.005	2.32 0.03	0.037 0.002

Further non-certified values :

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Certified mass-fraction values with uncertainty and non-certified values, in %

	Mo	Mg	Ce	V	Ti	Al	Sn	Sb
11A	**1.130** 0.019	-	-	**0.184** 0.004	**0.028** 0.002	**0.046** 0.002	**0.070** 0.003	**0.013** 0.003
11B	**1.144** 0.020	-	-	**0.182** 0.005	**0.041** 0.002	**0.067** 0.003	**0.074** 0.003	**0.011** 0.003
12A	**0.114** 0.002	-	-	**0.340** 0.005	**0.085** 0.003	**0.077** 0.003	**0.041** 0.003	**0.046** 0.004
12B	**0.117** 0.002	-	-	**0.326** 0.005	**0.071** 0.003	**0.077** 0.003	**0.042** 0.003	**0.046** 0.004
13A	**0.364** 0.006	**0.053** 0.003	**0.011** 0.002	**0.048** 0.002	**0.014** 0.001	**0.017** 0.001	**0.014** 0.001	0.002
13B	**0.364** 0.007	**0.054** 0.003	**0.011** 0.002	**0.048** 0.002	**0.012** 0.001	**0.019** 0.001	**0.014** 0.001	0.002
13C	**0.360** 0.007	**0.064** 0.004	**0.011** 0.002	**0.043** 0.002	**0.015** 0.001	**0.022** 0.001	**0.014** 0.001	0.002
14A	**0.633** 0.009	**0.015** 0.002	**0.009** 0.002	**0.013** 0.001	**0.018** 0.001	**0.009** 0.001	**0.027** 0.002	**0.015** 0.002
14B	**0.635** 0.009	**0.015** 0.002	**0.012** 0.002	**0.012** 0.001	**0.021** 0.001	**0.012** 0.001	**0.028** 0.002	**0.016** 0.003
14C	**0.646** 0.009	**0.017** 0.002	**0.019** 0.003	**0.013** 0.001	**0.018** 0.001	**0.007** 0.001	**0.025** 0.002	**0.020** 0.003
15A	**0.005** 0.001	**0.031** 0.002	**0.026** 0.003	**0.014** 0.001	**0.034** 0.001	**0.026** 0.001	**0.005** 0.001	**0.058** 0.006
15B	**0.004** 0.001	**0.037** 0.002	**0.021** 0.003	**0.013** 0.001	**0.025** 0.002	**0.029** 0.002	**0.005** 0.001	**0.058** 0.006
15C	**0.002** 0.001	**0.040** 0.002	**0.030** 0.003	**0.019** 0.001	**0.036** 0.002	**0.010** 0.001	**0.006** 0.001	**0.056** 0.006
16A	**0.203** 0.004	-	-	**0.021** 0.001	**0.073** 0.002	**0.007** 0.001	**0.125** 0.006	**0.011** 0.002
16B	**0.202** 0.004	-	-	**0.029** 0.001	**0.070** 0.002	**0.007** 0.001	**0.121** 0.006	**0.011** 0.002
16C	**0.195** 0.004	-	-	**0.027** 0.001	**0.057** 0.002	**0.004** 0.001	**0.125** 0.006	**0.010** 0.002
17A	**0.030** 0.002	**0.007** 0.001	**0.003** 0.001	**0.086** 0.003	**0.016** 0.001	**0.002** 0.001	**0.002** 0.001	-
17B	**0.030** 0.002	**0.009** 0.001	**0.003** 0.001	**0.086** 0.003	**0.016** 0.001	**0.002** 0.001	**0.002** 0.001	-
17C	**0.030** 0.002	**0.007** 0.001	**0.003** 0.001	**0.076** 0.003	**0.015** 0.001	**0.002** 0.001	**0.002** 0.001	-

Nb: 0.007% in 11A, 0.008% in 12A, 0.01% in 14B, 0.006% in 16A, 0.03% in 16B

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Certified mass-fraction values with uncertainty and non-certified values, in %

	Bi	B	Zn	Pb	W	Co	Zr	As
11A	**0.011** 0.001	**0.0018** 0.0003	-	**0.017** 0.003	0.005	**0.005** 0.001	**0.007** 0.001	**0.005** 0.001
11B	**0.007** 0.001	**0.0032** 0.0004	-	**0.007** 0.001	0.005	**0.005** 0.001	**0.007** 0.001	**0.005** 0.001
12A	**0.005** 0.001	**0.036** 0.002	**0.003** 0.001	**0.007** 0.001	**0.011** 0.002	**0.004** 0.001	0.002	**0.022** 0.002
12B	**0.006** 0.001	**0.047** 0.002	**0.004** 0.001	**0.009** 0.001	**0.007** 0.002	**0.008** 0.001	0.002	**0.024** 0.002
13A	-	-	-	-	0.003	**0.024** 0.001	**0.029** 0.003	**0.002** 0.001
13B	-	-	-	-	0.003	**0.024** 0.001	**0.023** 0.003	**0.002** 0.001
13C	-	-	-	-	0.003	**0.024** 0.001	*0.02*	**0.002** 0.001
14A	**0.007** 0.001	**0.0096** 0.0005	**0.010** 0.001	0.005	0.005	**0.005** 0.001	**0.011** 0.001	**0.036** 0.004
14B	**0.007** 0.001	**0.0100** 0.0006	**0.009** 0.001	0.005	0.005	**0.005** 0.001	**0.014** 0.001	**0.034** 0.004
14C	-	**0.0123** 0.0006	**0.010** 0.001	-	0.003	**0.009** 0.001	**0.013** 0.001	**0.035** 0.004
15A	**0.012** 0.001	**0.0041** 0.0003	-	-	**0.006** 0.001	**0.027** 0.001	-	0.003
15B	**0.010** 0.001	**0.0033** 0.0003	-	-	**0.007** 0.001	**0.027** 0.001	-	0.003
15C	**0.008** 0.001	**0.0057** 0.0004	-	-	**0.004** 0.001	**0.026** 0.001	-	0.003
16A	-	**0.018** 0.001	**0.019** 0.002	**0.006** 0.001	**0.019** 0.002	**0.010** 0.001	0.002	**0.005** 0.001
16B	-	**0.018** 0.001	**0.020** 0.002	**0.007** 0.001	**0.019** 0.002	**0.010** 0.001	0.002	**0.005** 0.001
16C	-	**0.020** 0.001	**0.017** 0.002	**0.015** 0.003	**0.015** 0.002	**0.006** 0.001	0.002	**0.003** 0.001
17A	0.001	0.0002	-	**0.002** 0.001	**0.004** 0.001	**0.043** 0.002	-	**0.007** 0.001
17B	0.001	0.0002	-	**0.002** 0.001	**0.004** 0.001	**0.043** 0.002	-	**0.008** 0.001
17C	0.002	0.0006	-	**0.002** 0.001	**0.004** 0.001	**0.043** 0.002	-	**0.005** 0.001

Te: 0.005% in 11A, 0.01% in 11B, 0.006% in 16A, 16B and 0.007% in 16C

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The end of the Certificate

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