

Characterised: by an international collaborative study (interlaboratory characterisation experiment), involving various analytical techniques and methods in a balanced representation. Besides the above solid sample spectrometry these techniques included combustion-IR MAS, FAAS, ET AAS, ICP AES, MAS (photometry), titrimetry and gravimetry. The instrumental methods of excellent repeatability were used in establishing the traceable values of the consecutive batches. Only the standardised or validated methods were used. The subcontractor was accredited for organizing and assessment of the interlaboratory experiments by the Czech Accreditation Institute on 13.5.2011 under No 189/2011.

Homogeneity: of the certified constituents, especially due to the influence by the structural properties of the CRM matrix were tested by spark excitation AES, the technique prevailing in the cast iron analysis, which is by coincidence the most strongly structure-influenced technique. The within-sample trend homogeneity was tested as the difference of results on both working surfaces, the between-sample trend as the difference of results obtained from the beginning and the end of casting of the CRM particular. Both trends were found statistically insignificant except for a few cases, contributions of which were included into the assessment of the combined uncertainty of the certified values. Repeatability of the subsequent analyses distributed evenly on the same working surface was taken for a conservative estimate of the within-sample random homogeneity, as the repeatability of instrument itself cannot be exactly separated. This overall repeatability was satisfactory in respect to the uncertainty of all certified values. The CRM set is **stable** by the nature of its matrix, when stored in the prescribed conditions..

Participating laboratories: listed below 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..

Arcelor Mittal Ostrava, Ostrava, Czechia*	MOR. ŽELEZÁRNY, Olomouc, Czechia
BESKYD, Frýdlant nad Ostravicí, Czechia	OBLF, Witten, Germany
Brammer Standards, Houston, TX, USA	SECO GROUP, Jičín, Czechia*
Dnieprospetsstal, Zaporozhiye, Ukraine	UNIPETROL RPA, Litvínov, Czechia
Enviform, Třinec, Czechia*	US Steel Košice, Košice, Slovakia
ESAB AB, Göteborg, Sweden	Vítkovice Test. Cent., Ostrava, Czechia*
Institute for CRM, Yekaterinburg, Russia	Volvo Powertrain Corp. Skövde, Sweden
Instytut Metalurgii Żelaza, Gliwice, Poland	ZPS Slévárna, Zlín, Czechia*
LECO INSTRUMENTE, Plzeň, Czechia*	ŽĎAS, Žďár nad Sázavou, Czechia*
KZGO, Krivoy Rog, Ukraine	ŽDB GROUP, Bohumín, Czechia*
MECHEL, Chelyabinsk, Russia	

Data evaluation: In principle five independent results for each value were reported. Their means were first scrutinized technically to identify possible errors, justifying deletion. Then the distribution of the accepted means was assessed in order to choose between their arithmetic and robust (by MAD method**) mean for assigning the certified value.

Uncertainty: is expressed as a \pm half width interval combined from the standard uncertainties of the mean of means and of homogeneity (when statistically significant), and expanded by the coverage factor $k=2$. The uncertainties of all certified values are below the pre-set (target) values, derived from the requirements of the relevant analytical standards and experience from the Proficiency Testing.

Traceability: was established by a comparison with the existing relevant CRMs, particularly with sets CZ 2002 and CZ 2015 - 2024. The values of the consecutive batches B and so forth were made traceable to the values of the initial batches A. No direct traceability to the primary references was possible due to the lack of applicable methods and complexity of matrices.

** van Montfort, M.A.J., Commun. Soil. Sci. Plant. Anal. 27, 463-468 (1996)

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CERTIFICATE

1014-CM-02033-13

CERTIFIED REFERENCE MATERIALS CZ 02033

Cast iron for solid sample spectrometry, CRM set 1–8

Date of issue: 25.2.2013 **Valid until:** 19.01.2027 page 1 / 4

Intended: for calibration, validation and matrix-match verification of cast iron spectrometric analysis from a plane of solid sample: Atomic Emission Spectrometry with spark, glow-discharge or laser excitation, and X-ray Fluorescence Spectrometry.

Eight CRM 1–8 represent the most frequent types of unalloyed and low alloy cast iron, where the codes 1-8 mean: (1) unalloyed ductile iron, (2) Ni-Cu ductile iron, (3) vermicular iron (CGI), (4) pig iron, (5) malleable iron, (6) Mn-Cr-V and (7) Ni-Mo alloyed iron, and (8) plain grey iron.

Instructions for use: The working surface of the CRM must be prepared before the analysis in the same way as samples analysed, in accordance with the particular analyzer manual.

An analysis area of at least 4 mm in diameter defines the minimum sample intake. A mean of at least three independent measurements is required for every metrological operation. Storage of the CRM in dry and non-corrosive environment is recommended.

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

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

Responsible person: Vladimír Bogumský

Manufactured: as gangs of chill-cast discs, cooled on both plan-parallel sides. This resulted in a white (i.e. effectively graphite free) structure required for solid sample spectrometry. Layers of 0.5 mm were machined off from both cooled surfaces.

Supplied: in a set or as individual discs 40 mm in diameter and approximately 18 mm thick. The certified values are valid for both plan-parallel sides (working surfaces) into depth of 6 mm. The discs are marked on the side by the CRM batch code and the limits to which certified values apply. When used to both limits, the remainder, which may contain minor structure defects, should be discarded.

Certified values: For the first reference batch A (cf certificate No. 1014-CM-02033-12) certified values are means of a minimum of ten accepted laboratory means obtained by at least three different methods. In this certificate (No. 1014-CM-02033-13), the certified values were made traceable to the values in the above reference batch by analysis in selected laboratories (the batches B, C, and D). The values are rounded to the same number of significant digits, as their uncertainty value.

Non-certified values: without uncertainty statement did not meet all requirements for certification and/or uncertainty below the pre-set maximum (target) values. They are intended only for proximate information about the matrix composition and may not be used for calibration.

Manufactured and characterised: in compliance with the Czech Metrology Institute Methodical procedure CORM CMI No. 114-MP-C101.. Preparation and certification of reference materials, and ISO REMCO Guides 34, 35.

Responsible person - CORM


Jan Beránek



Director of Regional branch, Prague

Vladimír Peršl 

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CAST IRON CRM SET CZ 02033, CRM 1 - 8 - Certified and noncertified mass fraction values, wt. %
 Certified values are given in **bold letters** with their uncertainties in regular letters shown below.
 Noncertified values without uncertainties are given in regular,
 empty boxes indicate values at or below limit of determination.

	C	Mn	Si	P	S	Cr	Ni	Cu	Mo	Mg	Ce
1C	3,15 0,03	0,674 0,004	2,36 0,03	0,064 0,002	0,010 0,001	0,051 0,001	0,367 0,003	0,035 0,001	0,197 0,002	0,019 0,002	0,009 0,002
1D	3,12 0,03	0,681 0,004	2,54 0,03	0,056 0,002	0,006 0,001	0,047 0,001	0,389 0,003	0,031 0,001	0,189 0,002	0,046 0,003	0,055 0,004
2C	3,44 0,03	0,146 0,002	1,50 0,02	0,112 0,003	0,015	0,048 0,001	0,606 0,006	0,74 0,01	0,018 0,001	0,030 0,002	
2D	3,71 0,04	0,194 0,002	1,28 0,01	0,165 0,003	0,011 0,001	0,032 0,001	0,635 0,006	0,92 0,01	0,013 0,001	0,058 0,003	0,021 0,003
3B	3,38 0,04	0,260 0,003	1,74 0,02	0,012 0,001	0,012 0,001	0,235 0,003	0,049 0,001	0,400 0,004	0,456 0,006	0,012 0,001	0,006 0,001
4B	3,95 0,02	0,145 0,002	0,252 0,004	0,041 0,002	0,046 0,002	0,049 0,001	0,023 0,001	0,062 0,002	0,005 0,001		
4C	4,06 0,02	0,250 0,002	0,423 0,005	0,054 0,002	0,038 0,002	0,080 0,002	0,084 0,001	0,085 0,002	0,002 0,001		
5B	2,42 0,04	0,812 0,005	1,32 0,02	0,033 0,001	0,073 0,003	0,061 0,001	0,188 0,003	0,031 0,001	0,089 0,002		
6B	2,95 0,04	1,15 0,01	3,23 0,04	0,095 0,003	0,020 0,002	1,36 0,002	0,026 0,001	0,272 0,003	0,005 0,001		
7B	3,61 0,03	0,304 0,003	1,82 0,02	0,021 0,002	0,020 0,002	0,536 0,005	1,28 0,01	0,036 0,001	0,96 0,01		
8B	3,28 0,03	0,461 0,003	1,96 0,02	0,22 0,004	0,073 0,003	0,111 0,002	0,097 0,002	0,154 0,002	0,036 0,001		

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	V	Ti	Al	Sn	Sb	Bi	B	Zn	Pb	W	Co
1C	0,019 0,001	0,036 0,001	0,033 0,001	0,032 0,002		0,016 0,002	0,0005 0,0002	0,001 0,001	0,006 0,001	0,015 0,002	
1D	0,013 0,001	0,017 0,001	0,091 0,003	0,022 0,002		0,003 0,001	0,0004 0,0002	0,027 0,002	0,003 0,001	0,022 0,003	
2C	0,012 0,001	0,054 0,002	0,018 0,001	0,017 0,001			0,0100 0,0006		0,010 0,001		0,003
2D	0,028 0,001	0,019 0,001	0,015 0,001	0,025 0,002	0,022 0,002	0,002 0,001	0,0036 0,0003	0,023 0,002	0,013 0,001	0,003 0,001	0,018 0,002
3B	0,009 0,001	0,023 0,001	0,026 0,001	0,019 0,002		0,001 0,001	0,0042 0,0003		0,009 0,001		0,012 0,001
4B	0,004 0,001	0,006 0,001	0,003 0,001	0,001 0,001	0,001			0,008 0,001	0,004 0,001		0,005 0,001
4C	0,015 0,001	0,010 0,001	0,005 0,001	0,002 0,001	0,001			0,016 0,002	0,003 0,001		0,035 0,002
5B	0,005 0,001	0,007 0,001	0,062 0,001			0,020 0,003	0,014 0,001				
6B	0,083 0,002	0,068 0,003	0,007 0,001	0,140 0,004	0,049 0,003						
7B	0,007 0,001	0,015 0,001	0,022 0,001							0,045 0,004	0,050 0,002
8B	0,029 0,001	0,025 0,001	0,004 0,001	0,056 0,003	0,018 0,002	0,013 0,002			0,006 0,001	0,009 0,001	

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Further non-certified values are 0,017% As in 3B, 0,010% As in 4B, 0,008% As in 6B, 0,015% As in 8B, 0,011% Te in 2D, 0,006% Te in 8B, 0,013% Zr in 1C, 0,011% Zr in 1D.