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CERTIFICATE
1014-CM-02033-12

CERTIFIED REFERENCE MATERIALS CZ 02033
Cast iron for solid sample spectrometry, CRM set 1–8

Date of issue:	**19.01.2012**	**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 unalloyed and low alloy cast iron types in sequence: unalloyed ductile iron, Ni-Cu ductile iron, vermicular iron (CGI), pig iron, malleable iron, Mn-Cr-V and Ni-Mo alloyed iron and plain grey iron.

Users instructions: The working surface of the CRM must be prepared before the analysis in the same way as analysed samples, in accordance with the particular analyser manual.
A single analysis area of at least 4 mm in diameter defines the minimum sample intake. A mean of at least three parallel independent measurements is required for every metrological operation. Storage in dry and non-corrosive environment is recommended.

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

Subcontractor: SPL Bohumin, 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 either side in vertical position. This resulted in a white (i.e. effectively graphite free) structure required by spectrometry. 0,5 mm was machined off either cooling surface.

Supplied in a set or as individual discs 40 mm in diameter and approximately 18 mm of total height, with two certified layers extending 6 mm upwards from either working surface. The discs are marked on the side by the CRM code and the certified layers' limits. When used to both limits, the remainder, which may contain minor structure defects, should be discarded.

Certified values are means of a minimum ten accepted laboratory means by at least three different methods. They are rounded to the same digit, as their uncertainty statement.

Non-certified values without uncertainty statement did not meet all requirements for certification and/or uncertainty below target maximum. They are intended for the matrix information and may not be used for calibration.

Manufactured and characterised in compliance with the Czech Metrology Institute Methodical procedure No. 0217-MP-C001-06:. Preparation and certification of reference materials, and ISO REMCO Guides 34, 35.

Jan Beránek
Responsible person - CORM

Ing. Zdeněk Jirák
zástupce ředitele OI Praha

Vladimír Peršl
Director of Regional branch, Prague

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CAST IRON CRM SET CZ 02033, CRM 1A-8A, 1B, 2B, 2C – VALUES IN % m/m

Certified values in bold, with ± uncertainty interval below in regular.

Non-certified, indicative values in regular, without uncertainty statement.

Empty boxes indicate values at or below limit of determination.

	C	Mn	Si	P	S	Cr	Ni	Cu	Mo	Mg	Ce
1A	**3,26** 0,03	**0,858** 0,005	**2,33** 0,03	**0,044** 0,002	**0,011** 0,001	**0,018** 0,001	**0,233** 0,003	**0,015** 0,001	**0,119** 0,002	**0,052** 0,003	**0,018** 0,003
1B	**3,20** 0,03	**0,721** 0,004	**2,59** 0,03	**0,047** 0,002	**0,011** 0,001	**0,063** 0,001	**0,231** 0,003	**0,020** 0,001	**0,182** 0,002	**0,056** 0,003	**0,010** 0,002
2A	**3,90** 0,03	**0,084** 0,001	**1,63** 0,02	**0,096** 0,003	**0,012** 0,001	**0,023** 0,001	**0,594** 0,006	**0,84** 0,01	**0,012** 0,001	**0,036** 0,003	**0,028** 0,003
2B	**3,54** 0,03	**0,098** 0,001	**1,28** 0,01	**0,049** 0,002	0,01	**0,051** 0,001	**0,634** 0,006	**0,69** 0,01	**0,008** 0,001	**0,030** 0,002	0,03
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	-
3A	**3,72** 0,04	**0,333** 0,003	**2,30** 0,03	**0,022** 0,001	**0,011** 0,001	**0,205** 0,003	**0,050** 0,001	**0,406** 0,004	**0,514** 0,006	**0,013** 0,001	**0,013** 0,002
4A	**4,18** 0,02	**0,236** 0,002	**0,71** 0,01	**0,053** 0,002	**0,034** 0,002	**0,040** 0,001	**0,056** 0,002	**0,081** 0,002	**0,002** 0,001	-	-
5A	**2,30** 0,04	**0,804** 0,005	**1,26** 0,02	**0,035** 0,001	**0,100** 0,003	**0,054** 0,001	**0,096** 0,002	**0,014** 0,001	**0,100** 0,002	-	-
6A	**3,14** 0,04	**1,22** 0,01	**3,37** 0,04	**0,077** 0,003	**0,032** 0,004	**1,40** 0,02	**0,023** 0,001	**0,225** 0,003	**0,005** 0,001	-	-
7A	**3,11** 0,03	**0,321** 0,003	**1,83** 0,02	**0,043** 0,002	**0,019** 0,001	**0,479** 0,005	**1,29** 0,01	**0,022** 0,001	**1,07** 0,01	-	-
8A	**3,49** 0,02	**0,408** 0,003	**2,20** 0,02	**0,169** 0,004	**0,073** 0,003	**0,126** 0,002	**0,094** 0,002	**0,116** 0,002	**0,026** 0,001	-	-

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	V	Ti	Al	Sn	Sb	Bi	B	Zn	Pb	W	Co
1A	0,018 (0,001)	0,039 (0,001)	0,048 (0,002)	0,035 (0,002)	-	0,005 (0,002)	0,0004 (0,0002)	0,001 (0,001)	-	0,016 (0,002)	-
1B	0,023 (0,001)	0,018 (0,001)	0,051 (0,003)	0,025 (0,002)	-	0,004 (0,001)	0,0003 (0,0001)	0,001	0,003 (0,001)	0,012 (0,001)	-
2A	0,083 (0,002)	0,032 (0,001)	0,014 (0,001)	0,016 (0,002)	0,012 (0,002)	0,008 (0,002)	0,0040 (0,0003)	0,024 (0,002)	-	0,004 (0,001)	0,022 (0,002)
2B	0,059 (0,001)	0,099 (0,004)	0,026 (0,001)	0,021 (0,001)	0,017 (0,002)	-	0,0100 (0,0007)	0,013 (0,001)	-	0,003	0,023 (0,001)
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
3A	0,01 (0,001)	0,006 (0,001)	0,025 (0,002)	0,008 (0,001)	-	0,008 (0,002)	0,0053 (0,0004)	-	0,007 (0,001)	-	0,007 (0,001)
4A	0,004 (0,001)	0,015 (0,001)	0,015 (0,001)	0,007 (0,001)	-	-	-	0,007 (0,002)	0,006 (0,001)	-	0,002 (0,001)
5A	0,005 (0,001)	0,008 (0,001)	0,060 (0,003)	-	-	-	-	-	-	-	-
6A	0,300 (0,006)	0,066 (0,003)	0,022 (0,001)	0,119 (0,004)	0,056 (0,003)	-	-	-	-	-	-
7A	0,005 (0,001)	0,027 (0,001)	0,029 (0,001)	-	0,001 (0,001)	0,017 (0,002)	-	-	-	0,022 (0,003)	0,044 (0,002)
8A	0,034 (0,001)	0,027 (0,001)	0,015 (0,001)	0,040 (0,002)	0,001 (0,001)	-	-	-	0,006 (0,001)	0,011 (0,002)	-

Further non - certified values are 0,034% Nb in 1B and 0,012% As in 3A

Characterised by an international collaborative study (interlaboratory characterisation experiment), involving various analytical techniques and methods in a balanced representation. Besides the above solid sample spectrometries these techniques included combustion-IR MAS, FAAS, ET AAS, ICP AES, MAS (photometry), titrimetry and gravimetry. Only the standardized 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 and of the influence by structure were tested by the spark excitation AES, the technique prevailing in the cast iron analysis and coincidentally the most strongly structure-influenced technique. The within-sample trend homogeneity was tested as the difference of results on the opposite limits of the certified layer, the between-samples trend as the difference of results from the beginning and the end of casting. Both were found statistically insignificant except for a few cases, contributions of which were combined to the ultimate 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 are **stable** by the nature of their matrix.

Participating laboratories listed below were accredited and/or they demonstrated compliance with ISO 17025 during their participation:

Arcelor Mittal Ostrava, Ostrava, Czechia,
BESKYD, Frýdlant nad Ostravicí, Czechia
Brammer Standards, Houston, TX, USA
Dnieprospetsstal, Zaporozhiye, Ukraine
Enviform, Třinec, Czechia
ESAB AB, Göteborg, Sweden
Institute for CRM, Yekaterinburg, Russia
Instytut Metalurgii Želaza, Gliwice, Poland
LECO INSTRUMENTE, Plzeň, Czechia
KZGO, Krivoy Rog, Ukraine
MECHEL, Chelyabinsk, Russia

MOR. ŽELEZÁRNY, Olomouc, Czechia
OBLF, Witten, Germany
SECO GROUP, Jičín, Czechia
UNIPETROL RPA, Litvínov, Czechia
US Steel Košice, Košice, Slovakia
Vítkovice Test. Cent., Ostrava, Czechia
Volvo Powertrain Corp. Skövde, Sweden
ZPS Slévárna, Zlín, Czechia
ŽDAS, Žďár nad Sázavou, Czechia
ŽDB GROUP, Bohumín, Czechia

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 ± 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 target maximums, derived from the requirements of the relevant analytical standards and experience from the Proficiency Testing. They are given rounded to one valid figure.

Traceability was established by comparison with existing relevant CRM, particularly with sets CZ 2002 and CZ 2015 - 2024. The values of the consecutive batches B and so forth are made traceable to the values of the initial batches A. No direct traceability to the primary references was possible due to 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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