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Made by: Certification Body for Certification of Reference Materials (CORM)

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CERTIFICATE 6016-CM-02033-15

CERTIFIED REFERENCE MATERIALS CZ 02033

Repeated preparation

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

Date of issue: April 2nd, 2015 Valid until: April 2nd, 2030 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.

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

Responsible person: Vladimír Bogumský

Manufactured as gangs of chill-cast discs, cooled on both side in vertical position. This resulted in a white (i.e.

effectively graphite free) structure required by spectrometry. A layer of 0.5 mm was machined off

from both of the working surfaces.

Supplied in a set or as individual discs of 40 mm in diameter and approximately of 18 mm of total height,

with two certified layers extending 6 mm upwards from both working surfaces. The discs are marked on the side by the CRM code and 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 figures, as their uncertainty statement.

CORM Deputy Head:

Rosil-

Mgr. Matilda Roziková, Ph.D.

Head of CORM:

RNDr. Pavel Klenovský

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0.036

0.003

0.023

0.017

0.002

0.018

0.013

0.002

0.013

0.002

900.0

CRM 1E, 1F, 1G, 2E, 2F, 2G, 3C, 3D, 4D, 4E, 5C, 6C, 7C, 8C - values in wt. % CAST IRON CRM SET CZ 02033

Non-certified, indicative values in regular, without uncertainty statement Certified values in bold, with ± uncertainty interval below in regular.

十

4

16

2E

2F

0.070 0.038 0.050 0.036 0.016 0.053 0.003 0.003 0.002 Mg 0.185 0.002 0.002 900.0 996.0 0.002 0.490 0.453 0.024 0.002 0.002 0.005 0.001 0.001 0.001 0.001 0.001 0.001 Empty boxes indicate values at or below limit of determination 0.012 0.018 0.396 0.005 0.013 0.273 0.016 0.084 0.158 0.027 0.89 0.88 0.421 0.004 0.004 0.89 0.003 0.001 0.002 0.001 0.001 0.01 0.01 0.001 0.01 0.373 0.620 0.658 0.650 0.040 0.025 0.063 0.049 0.102 0.357 0.003 0.003 0.005 0.021 0.002 Z 0.035 0.236 0.044 0.043 0.022 0.100 0.056 0.030 0.085 0.125 0.5420.037 0.027 0.002 0.003 1.33 ပ် 0.001 0.001 0.001 0.001 0.001 0.01 0.005 0.010 0.009 0.009 900.0 900.0 0.019 0.026 0.058 0.007 0.007 0.041 0.001 0.001 0.001 0.001 0.001 0.001 0.091 0.002 0.001 S 0.036 0.106 0.125 0.026 0.043 0.159 800.0 0.028 0.168 0.050 0.023 0.037 0.097 0.002 0.003 0.004 0.003 0.027 0.001 0.001 0.2590.090 2.15 2.68 2.53 1.03 1.23 1.10 2.12 0.005 1.40 0.02 0.02 0.02 Si 0.693 0.168 960.0 0.333 0.112 0.034 0.3890.408 0.317 0.704 0.005 0.701 0.005 0.002 0.091 0.002 0.002 0.003 0.002 0.002 0.002 0.004 1.25 0.003 0.01 3.78 4.45 3.23 3.22 3.68 3.24 4.19 2.30 3.55 0.03 3.47 0.03 3.77 0.03 0.04 0.03 0.03 0.04 3.11 3.41

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2G

30

30

4E

5C

9

80

	>	ï	AI	Sn	Sb	Bi	В	Zn	Pb	×	Co
10	0.015	0.046	0.058	0.032		0.002	0.0036	0.009	200'0	0.021	0.022
<u>u</u>	0.001	0.001	0.002	0.002		0.001	0.0003	0.001	0.001	0.002	0.001
11	0.014	0.041	0.073	0.030		0.001	0.0043	0.004	600'0	0.022	0.024
	0.001	0.001	0.003	0.002		0.001	0.0003	0.001	0.001	0.001	0.001
10	0.019	0.054	0.062	0.028		0.005	0.0034	0.003	0.016	0.015	0.010
2	0.001	0.001	0.002	0.002		0.001	0.0003	0.001	0.002	0.001	0.001
36	0.026	0.039	0.025	0.015	0.028	0.005	0.0024	0.025	0.004	800'0	0.005
77	0.001	0.001	0.001	0.001	0.002	0.001	0.0002	0.002	0.001	0.001	0.001
2E	0.010	0.021	0.024	0.014	0.028	0.002	0.0020	0.018	0.005	0.003	0.003
17	0.001	0.001	0.001	0.001	0.002	0.001	0.0002	0.001	0.001	0.001	0.001
26	0.017	0.029	0.019	0.015	0.029	900'0	0.0023	0.020	0.008	0.004	0.012
2	0.001	0.001	0.001	0.001	0.002	0.001	0.0002	0.001	0.001	0.001	0.001
30	0.016	0.021	0.024	600'0		0.002	0.0044		0.005	0.003	0.026
2	0.001	0.001	0.001	0.001		0.001	0.0002		0.001	0.001	0.001
30	0.072	0.016	0.055	600'0	0.007	0.002	0.0071		0.005		0.014
3	0.002	0.001	0.002	0.001	0.001	0.001	0.0003		0.001		0.001
d,	0.012	600'0	200'0	0.001		0.002	0.0001	0.009	0.007		0.003
ţ	0.001	0.001	0.001	0.001				0.001	0.001		0.001
45	0.015	0.011	0.003	0.001		0.002			0.002		0.033
ļ	0.001	0.001	0.001	0.001							0.001
50	0.054	800'0	0.103	0.002	0.002	0.007	8200.0				0.013
3	0.002	0.001	0.003	0.001		0.002	0.0003				0.001
J.	0.192	0.107	0.024	0.131	0.044		0.0024		0.003	0.007	0.005
3	0.002	0.004	0.001	0.003	0.002		0.0002		0.001	0.001	0.001
7.0	290'0	0.026	0.040	0.004		0.002	8000'0			0.037	0.048
2	0.001	0.001	0.002	0.001			0.0002			0.002	0.001
SC	0.015	0.022	0.004	290'0	0.014	600'0			0.008		0.030
3	0.001	0.001	0.001	0.002	0.002	0.001			0.001		0.001

5C, 0.006% Te in 7C, Further non-certified values are 0.007% As in 3C, 0.018% As in 3D, 0.012% As in 4D, 0.006% As in 8C, 3C, 0.010% Te in , 0.009% Zr in 5C

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Non-certified values without uncertainty statement do 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 Methodical Procedure No. 017-MP-C001-14 of the Czech Metrology Institute: Preparation and certification of reference materials and ISO REMCO Guides 34, 35.

Characterised

by an international collaborative study (interlaboratory characterisation experiment), involving various analytical techniques and methods in a balanced representation. Participants chosen for this experiment were preferably accredited testing labs. Besides the above solid sample spectrometry methods 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 producer was accredited for provision of proficiency testing in this area by the Czech Accreditation Institute on 13th of May 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-influence technique. The within-sample trend homogeneity was tested as the difference of results on the opposite limits of the certified layer, the between-sample trend as the difference of results from 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:

SES Inspekt, Tlmače, Slovakia Unex, Olomouc, Czech Republic Enviform, Třinec, Czech Republic Viadrus, Bohumín, Czech Republic Lithea, Brno, Czech Republic OBLF, Witten, Germany ZPS Slévárna, Zlín, Czech Republic SECO Group, Jičín, Czech Republic ŽĎAS, Žďár nad Sázavou, Czech Republic ArcelorMittal Ostrava, Ostrava, Czech Republic Beskyd, Frýdlant nad Ostravicí, Czech Republic Thermo Fisher Scientific, Ecublens, Switzerland Institute for CRM, Yekaterinburg, Russia US Steel Košice - Labortest, Košice, Slovakia Instytut Metalurgii Żelaza, Gliwice, Poland Volvo Powertrain Corp., Skövde, Sweden MS Utilities and serv., Bohumín, Czech Republic Vítkovice Testing Centrum, Ostrava, Czech Rep.

Data evaluation

In principle five independent results for each value were reported. Their means were at first scrutinized technically to identify possible errors, justifying deletion of outliers. Then the distribution of the accepted means was assessed in order to choose between their arithmetic or robust (by MAD method*) mean for assigning the certified value. *van Montfort, M.A.J., Commun. Soil. Sci. Plant. Anal. 27, 463-468 (1996)

Uncertainty

The reported uncertainty is the expanded uncertainty calculated using a coverage factor of 2 which gives a level of confidence of approximately 95% as defined in the document JCGM 100:2008 Evaluation of measurement data - Guide to the expression of uncertainty in measurement (GUM). The evaluation was made according to ISO Guide 35:2006 following the principles of the methodology sponsored by the European Committee for Iron and Steel Standardization (ECISS). The uncertainties of all certified values below are the target maxima, 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, CZ 02033, CZ 02034 and CZ 2015 - 2024. No direct traceability to the primary references was possible due to lack of applicable methods and complexity of matrices.

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