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CERTIFICATE

0217-CM-2001-14

CERTIFIED REFERENCE MATERIALS CZ 2001

Low alloy steel for solid sample spectrometry, CRM set 180 – 189 A, B, C, D, E

First issue: December 15, 2001 page 1 / 4
Valid till: December 1, 2015
Recertified on: January 1, 2014
Valid till: January 1, 2020

Prepared by: vacuum melting and casting, followed by electro-slag remelting and rolling and forging the ingots to the bars of the ultimate diameter of 44 mm, and subdividing to the discs 13 or 25 mm high. The CRMs can be used in sets of ten or individually.

The set covers the typical concentration ranges of the alloying elements and impurities of low alloy steels. The composition of the individual CRMs was balanced to avoid excessive matrix influence while sufficiently covering the above ranges. Consequently the CRMs may not correspond with any particular steel grade.

Intended: for calibration and validation of methods of low alloy steel spectrometry from a plane of solid sample: Atomic Emission Spectrometry with spark, glow-discharge or laser excitation and X-ray Fluorescence Spectrometry.

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: ALS Czech Republic, s.r.o. Na Harfě 336/9, 190 00 Praha 9, Czech Republic, www.alsglobal.cz

Responsible person: Vladimír Nováček

CORM deputy head:

Head of CORM:

Ing. Jan Beránek



RNDr. Pavel Klenovský

Certified values (in bold) with uncertainty, and non-certified values, in m/m %

	C	Mn	Si	P	S	Cr	Ni	Mo	V	W	Cu	Al
180 B	0.003	0.047	0.001	0.004	0.0038	0.013	0.018	0.001	0.000	0.0001	0.006	0.001
		0.002	0.001	0.001	0.0003	0.001	0.001	0.001	0.001		0.001	
181 B	0.240	0.988	0.445	0.042	0.008	0.669	0.737	0.395	0.307	0.188	0.095	0.016
	0.008	0.022	0.013	0.002	0.001	0.011	0.011	0.009	0.006	0.005	0.003	0.001
181 C	0.243	0.988	0.443	0.042	0.008	0.666	0.739	0.394	0.307	0.187	0.095	0.016
	0.009	0.022	0.013	0.002	0.001	0.011	0.011	0.009	0.006	0.005	0.003	0.001
181 D	0.231	0.980	0.437	0.040	0.007	0.661	0.726	0.389	0.303	0.187	0.093	0.016
	0.008	0.022	0.013	0.002	0.001	0.011	0.011	0.009	0.006	0.005	0.003	0.001
182 B	1.39	0.370	0.126	0.008	0.006	0.122	2.82	0.011	0.027	0.016	0.293	0.023
	0.02	0.008	0.008	0.001	0.001	0.002	0.03	0.001	0.002	0.004	0.007	0.001
182 C	1.36	0.363	0.111	0.009	0.008	0.123	2.80	0.012	0.028	0.018	0.294	0.028
	0.02	0.008	0.008	0.001	0.001	0.002	0.03	0.001	0.002	0.004	0.007	0.002
183 C	0.049	1.76	1.03	0.009	0.012	0.205	1.10	0.036	0.004	0.353	0.575	0.150
	0.003	0.03	0.02	0.001	0.001	0.010	0.02	0.003	0.002	0.008	0.014	0.005
183 D	0.048	1.75	1.02	0.009	0.012	0.204	1.09	0.036	0.004	0.354	0.571	0.150
	0.003	0.02	0.02	0.001	0.001	0.010	0.02	0.003	0.002	0.008	0.009	0.005
183 E	0.049	1.76	1.03	0.009	0.013	0.205	1.10	0.036	0.004	0.351	0.575	0.149
	0.003	0.02	0.02	0.001	0.001	0.010	0.02	0.003	0.002	0.008	0.013	0.005
184 A	1.013	2.23	0.348	0.028	0.01	2.33	0.250	0.016	0.017	0.001	0.089	0.022
	0.012	0.03	0.008	0.002	0.001	0.02	0.008	0.004	0.003	0.001	0.002	0.002
185 A	0.566	0.715	0.230	0.024	0.02	0.032	3.84	0.123	0.178	0.001	0.179	0.060
	0.009	0.004	0.005	0.001	0.001	0.003	0.04	0.006	0.006	0.001	0.003	0.002
186 C	0.394	1.311	1.41	0.013	0.007	1.51	1.58	0.255	0.021	0.054	0.227	0.042
	0.007	0.016	0.02	0.001	0.001	0.02	0.02	0.008	0.002	0.004	0.005	0.002
186 D	0.392	1.312	1.41	0.013	0.007	1.51	1.58	0.254	0.021	0.054	0.226	0.042
	0.007	0.016	0.02	0.001	0.001	0.02	0.02	0.008	0.002	0.004	0.005	0.002
187 A	0.119	0.525	0.567	0.035	0.018	3.51	0.085	0.565	0.558	0.67	0.036	0.019
	0.004	0.007	0.023	0.002	0.001	0.04	0.004	0.008	0.008	0.02	0.003	0.002
187 C	0.118	0.530	0.588	0.035	0.013	3.50	0.085	0.563	0.559	0.67	0.041	0.038
	0.004	0.007	0.023	0.002	0.002	0.04	0.004	0.008	0.008	0.02	0.003	0.002
187 D	0.119	0.529	0.576	0.035	0.015	3.51	0.085	0.566	0.560	0.67	0.035	0.024
	0.004	0.007	0.023	0.002	0.002	0.04	0.004	0.008	0.008	0.02	0.003	0.002
188 A	0.332	0.169	0.775	0.006	0.033	5.11	0.445	1.28	0.802	0.091	0.057	0.093
	0.010	0.004	0.016	0.001	0.002	0.05	0.008	0.02	0.008	0.005	0.003	0.003
189 A	0.175	0.262	0.286	0.032	0.051	1.065	5.34	0.837	0.054	1.30	0.060	0.041
	0.006	0.004	0.007	0.002	0.002	0.014	0.02	0.009	0.006	0.02	0.003	0.002

Certified values (in bold) with uncertainty, and non-certified values, in m/m %

	Ti	Zr	Co	B	Nb	Ta	Sr	As	Sb	Pb	Bi	N
180 B	0.000 0.001	0.000 0.001	0.003 0.001	0.0000 0.0001	0.0001	0.0000	0.0011 0.0005	0.001 0.001	0.004 0.001	0.0002	0.0000	0.0028
181 B	0.155 0.008	0.001 0.001	0.050 0.003	0.0076 0.0005	0.062 0.003	0.042 0.003	0.122 0.004	0.029 0.001	0.017 0.001	0.0005 0.0002		0.005
181 C	0.159 0.008	0.001 0.001	0.051 0.003	0.0076 0.0005	0.063 0.003	0.042 0.003	0.122 0.005	0.029 0.001	0.017 0.001	0.0005 0.0002		0.005
181 D	0.153 0.008	0.001 0.001	0.050 0.003	0.0071 0.0005	0.060 0.003	0.039 0.003	0.117 0.004	0.028 0.001	0.017 0.001	0.0005 0.0002		0.005
182 B	0.004 0.002	0.001 0.001	0.171 0.004	0.0003 0.0001	0.001 0.001	0.000 0.001	0.004 0.001	0.005 0.001	0.001 0.001	0.000		0.0049 0.0003
182 C	0.002 0.002	0.001 0.001	0.171 0.004	0.0003 0.0001	0.001 0.001	0.001 0.001	0.004 0.001	0.005 0.001	0.001 0.001	0.001		0.0049 0.0003
183 C	0.003 0.001	0.081 0.007	0.120 0.003	0.0005 0.0001	0.006 0.001	0.000	0.053 0.003	0.005 0.001	0.001 0.001	0.000	0.0000 0.0001	0.0040 0.0004
183 D	0.003 0.001	0.077 0.004	0.119 0.003	0.0005 0.0001	0.006 0.001	0.000	0.051 0.002	0.005 0.001	0.001 0.001	0.000	0.0000 0.0001	0.0036 0.0004
183 E	0.003 0.001	0.080 0.007	0.119 0.003	0.0005 0.0001	0.006 0.001	0.000	0.053 0.003	0.005 0.001	0.001 0.001	0.000	0.0000 0.0001	0.0040 0.0004
184 A	0.010 0.002	0.002 0.002	0.007 0.002	0.0005 0.0002	0.013 0.001	0.000 0.001	0.008 0.001	0.006 0.001	0.002 0.001	0.000		0.0104 0.0005
185 A	0.022 0.001	0.002 0.001	0.032 0.001	0.0116 0.0014	0.20 0.01	0.085 0.005	0.003 0.001	0.022 0.002	0.011 0.001	0.002 0.001		0.0051 0.0004
186 C	0.047 0.004	0.002 0.002	0.006 0.002	0.0009 0.0001	0.004 0.001	0.008 0.001	0.018 0.001	0.007 0.001	0.002 0.001	0.000		0.005
186 D	0.047 0.004	0.002 0.002	0.006 0.002	0.0009 0.0001	0.004 0.001	0.008 0.001	0.018 0.001	0.007 0.001	0.002 0.001	0.000		0.005
187 A	0.087 0.005	0.013 0.001	0.071 0.003	0.0006 0.0002	0.028 0.003	0.015 0.002	0.013 0.001	0.007 0.007	0.023 0.002	0.003 0.001	0.003 0.001	0.0122 0.0007
187 C	0.110 0.005	0.011 0.001	0.071 0.003	0.0006 0.0002	0.028 0.003	0.016 0.002	0.014 0.001	0.007 0.007	0.023 0.002	0.003 0.001	0.003 0.001	0.0153 0.0021
187 D	0.096 0.005	0.012 0.002	0.071 0.003	0.0006 0.0002	0.028 0.003	0.016 0.002	0.013 0.001	0.007 0.007	0.022 0.002	0.003 0.001	0.003 0.001	0.0120 0.0009
188 A	0.034 0.003	0.052 0.003	0.006 0.001	0.0047 0.0004	0.122 0.003	0.022 0.002	0.005 0.001	0.005 0.005	0.006 0.002	0.001 0.001		0.0076 0.0004
189 A	0.326 0.010	0.005 0.001	0.007 0.002	0.0030 0.0003	0.017 0.002	0.005 0.005	0.029 0.002	0.080 0.003	0.003 0.003	0.002 0.001		0.004 0.004

Homogeneity:	of the certified constituents was tested by AES with spark excitation. Both within-sample homogeneity (random, radial trend) and between-sample homogeneity (axial trend along the successive bars) were evaluated. Their uncertainty contribution, when found significant, was combined to the ultimate uncertainty of the certified value.
Stability:	the CRM materials are stable by nature of their matrix.
Storage:	in a dry and non-corrosive environment is recommended.
User instructions:	<p>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. When determining low contents of C, Si, Al special care must be taken to avoid contamination of the analytical surface with residues of abrasives. Overheating of the analytical surface during grinding should be avoided.</p> <p>A single analysis area of at least 4 mm in diameter defines the minimum sample intake.</p> <p>There are no safety hazards in the storage and proper use of CRM.</p>
Characterization:	by interlaboratory experiment involving selected competent laboratories was made in compliance with ISO Guide 35.
Traceability:	of the certified values was established to the certified values of matrix-matching CRM of other producers (NIST, BS and others)
Methods:	of various analytical techniques were used including solid sample spectroscopy, combustion, thermoevolution and solution analysis.
Participating laboratories:	<p>establishing the values of batches C, D, E traceable to the certified values of reference batches A:</p> <p>Enviform, a.s., Třinec, Czech Republic ŽĎAS, a.s., Žďár nad Sázavou, Czech Republic ZPS – Slévárna, a.s., Zlín, Malenovice, Czech Republic</p>
Certified values:	<p>of the consecutive batches C, D, E were made traceable to the certified values of the respective reference batches A (cf. Certificate No: 017-CM-2001-01), which were based on an international collaborative study involving 27 laboratories in 8 countries. Traceability was established by three laboratories, with uncertainty contribution (repeatability) negligible compared to the uncertainty of the certified values.</p> <p>The validity term of the former batches A, B was prolonged by the expert assessment of the Czech Metrology Institute, with their original values unchanged. The certified values are tabulated in bold, rounded to the same digit as their uncertainty, shown below in regular</p>
Non-certified values:	tabulated without uncertainty statements did not meet all requirements for certification. They are intended for the matrix information only and may not be used for calibration.
Uncertainty:	Expanded uncertainty U with a coverage factor of $k=2$ at the 95% confidence interval.

End of the Certificate.

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CERTIFICATE

SET OF CERTIFIED REFERENCE MATERIALS CZ 2002 LOW ALLOY CAST IRON FOR SOLID SAMPLE SPECTROMETRY CRM 241 - 249 A-D

Designed for the calibration and validation of methods of spectrometrical analysis on the planes of solid samples with an analyzed area of at least 4 mm in diameter: Atomic Emission Spectrometry with spark, glow discharge or laser excitation and X-ray Fluorescence Spectrometry.

The CRMs can be used as a set of nine or as individual samples.

Manufacture and Technical Parameters. The samples were chill-cast white on a massive copper block with controlled speed at a controlled temperature of the molten metal.

The samples are truncated pyramids with a base analytical surface (38x38 mm), a minimum total height of 20 mm and a side ledge 11-13 mm high. The samples can be used till 1 mm of the ledge height remains. The certified portion of a sample thus extends 10-12 mm from the original analytical surface.

The samples are electro-spark marked on surfaces opposite to the analytical surfaces.

Shrinkage cavities and porosity which may appear in the uncertified portions of the samples due to the applied technology and the properties of the material do not affect the analytical performance of the certified portions.

Homogeneity was tested by Atomic Emission Spectrometry with an analytical area approximately 4 mm in diameter.

Tested were the random homogeneity and the trend homogeneity along the height of the certified portion and the trend homogeneity of the casting sequence. The latter test was supported by Combustion - IR Molecular Absorption Spectrometry and Thermoevolution.

Producer

ČKD Technical Laboratories, Na Harfě 9, CZ - 190 02 Praha, Czech Republic

Fax: + 420 2 66036578, E-mail: techlab@anet.cz

Project Manager: Miroslav Gorný

Quality Management System ISO 9001 is in force with the producer. Production, testing and certification were carried out in compliance with the ISO-REMCO Guide 34 (2000).

Certificate No.: 017/CR/045

Date of Issue: 21.3. 2000

Valid until: 21.3.2015

Pavel Klenovský
CMI Director



CERTIFICATION

Principle and Traceability. Certification based on an interlaboratory experiment performed by various independent analytical methods was carried out in compliance with the ISO-REMCO Guide 35 (1989).

The results were traced to the former 241 through 249 CRM set and standard primary substances. The methods were validated by matrix-matching CRMs.

Methods. Atomic Emission Spectrometry with spark and glow discharge excitation and X-ray Fluorescence Spectrometry were applied on a plane of the solid sample. Crushed certified portions of the samples were analyzed by Combustion - IR Molecular Absorption Spectrometry, Thermo-evolution, Instrumental Neutron Activation Analysis and by solution methods which comprised Atomic Emission Spectrometry with Inductively Coupled Plasma excitation directly and with hydrides generation, Flame and Electro-Thermal Atomization Atomic Absorption Spectrometry, Molecular Absorption Spectrometry (Spectrophotometry) and Gravimetry.

Participating laboratories:

Analytical Laboratories Plzeň, Plzeň

ARL, Ecublens, Switzerland

ČKD Technical Laboratories, Praha

LECO Instrumente Plzeň, Plzeň

Nová huť, Ostrava

Nuclear Physics Institute, Řež u Prahy

Pramet Tools, Šumperk

Škoda, Plzeň

Škoda Auto, Mladá Boleslav

Třinecké železářny, Třinec

Vítkovice, Ostrava

ŽDAS, Žďár nad Sázavou

Železářny a drátovny Bohumín, Bohumín

Železářny Hrádek, Hrádek u Rokycan

Evaluation. First the values of laboratory means were assessed technically to justify the deletion of possible outliers. Next the normal distribution of the laboratory means in each set was verified and the unrounded arithmetic averages and their standard deviations calculated.

Certified values are the averages of at least six accepted laboratory means the normal distributions of which were not rejected, rounded identically as their stated uncertainties.

Uncertainty was estimated with respect to ISO Guide to the Expression of Uncertainty in Measurement (1993) and Document EURACHEM, 1995 - Quantifying Uncertainty in Analytical Measurement as an expanded combined uncertainty. It is expressed as the \pm half-width interval except for certified zero values for which only the + halfwidth interval applies. The sources of the estimates of uncertainty were the standard deviation of an average of the laboratory means and a contribution of the combined inhomogeneities when found to be statistically significant. A coverage factor of 2,3 was applied.

The uncertainty statement is given by two significant figures at most and holds only for analytical areas 4 mm or more in diameter.

Uncertified values are given when less than six accepted laboratory means were available and serve only as supplementary matrix information. They must not be used for calibration and validation.

Stability and storage. The CRM materials and certified constituents are stable over the entire period of validity. The samples must be stored in a non-corrosive environment.

Users instructions. The analytical surfaces of the CRMs must be prepared prior to analysis in the same way as the analyzed samples in agreement with the Instrument Operation Instructions.

SET OF LOW ALLOY CAST IRON SPECTROMETRIC CERTIFIED REFERENCE MATERIALS CZ 2002

NINE TYPES 241 - 249A, B, C, D

N°	% m/m	C	Mn	Si	P	S	Ni	Cr	Cu	Mo	V	Ti	Al	Mg	Ce	B	N°
241B	value	1,84	0,060	3,15	0,007	0,123	0,021	0,683	0,011	0,61	0,080	0,001	0,003	0,000	0,000	0,001	241B
	U _c	0,02	0,002	0,03	0,001	0,005	0,001	0,005	0,001	0,01	0,002	0,001	0,001	0,0005	0,0006		
242B	value	2,06	0,189	2,81	0,044	0,028	0,022	0,031	0,040	1,21	0,46	0,28	0,042	0,000	0,00	0,005	242B
	U _c	0,02	0,004	0,03	0,001	0,002	0,001	0,001	0,002	0,01	0,01	0,01	0,002	0,0005		0,001	
242A	value	1,84	0,060	3,06	0,039	0,036	0,039	0,029	0,055	1,13	0,37	0,19	0,036	0,000	0,00	0,008	242A
	U _c	0,02	0,002	0,03	0,001	0,002	0,001	0,001	0,002	0,01	0,01	0,01	0,003	0,0005		0,001	
243A	value	2,32	0,422	2,39	0,173	0,082	0,085	0,398	0,187	0,262	0,154	0,023	0,013	0,000	0,000	0,009	243A
	U _c	0,03	0,007	0,02	0,005	0,002	0,002	0,005	0,002	0,004	0,005	0,002	0,002	0,0005	0,001	0,001	
244B	value	2,57	0,68	2,06	0,022	0,011	0,336	0,360	0,308	0,056	0,002	0,019	0,019	0,025	0,018	0,093	244B
	U _c	0,03	0,01	0,03	0,001	0,001	0,003	0,003	0,003	0,001	0,001	0,001	0,002	0,001	0,003	0,003	
245B	value	2,95	1,38	1,59	0,42	0,035	0,194	0,197	0,081	0,115	0,055	0,110	0,038	0,003	0,00	0,003	245B
	U _c	0,03	0,01	0,02	0,01	0,002	0,002	0,003	0,002	0,002	0,002	0,002	0,002	0,001		0,001	
245A	value	2,94	1,38	1,58	0,41	0,039	0,161	0,166	0,076	0,114	0,073	0,087	0,019	0,003	0,00	0,007	245A
	U _c	0,03	0,01	0,02	0,01	0,002	0,003	0,004	0,003	0,002	0,002	0,003	0,002	0,001		0,001	
246B	value	2,73	0,354	0,76	0,66	0,020	0,065	1,16	1,39	0,009	0,013	0,014	0,101	0,016	0,007	0,000	246B
	U _c	0,03	0,005	0,01	0,01	0,002	0,001	0,01	0,01	0,001	0,001	0,001	0,005	0,001	0,002	0,0005	
247B	value	3,09	1,05	1,20	0,098	0,0034	0,437	0,041	0,822	0,023	0,013	0,067	0,043	0,056	0,053	0,000	247B
	U _c	0,04	0,01	0,02	0,003	0,0009	0,003	0,001	0,004	0,001	0,001	0,002	0,002	0,003	0,003	0,0005	
248B	value	3,34	0,265	1,82	0,050	0,0033	0,680	0,022	0,124	0,001	0,142	0,163	0,026	0,037	0,030	0,039	248B
	U _c	0,02	0,003	0,02	0,001	0,0005	0,007	0,001	0,002	0,001	0,003	0,003	0,002	0,002	0,002	0,002	
248C	value	3,39	0,281	1,78	0,053	0,0035	0,688	0,052	0,132	0,001	0,162	0,133	0,028	0,048	0,036	0,038	248C
	U _c	0,02	0,002	0,02	0,001	0,0005	0,007	0,001	0,002	0,001	0,003	0,003	0,002	0,002	0,002	0,002	
249B	value	4,06	0,121	0,47	0,26	0,0078	1,16	0,102	0,474	0,013	0,019	0,046	0,105	0,040	0,021	0,016	249B
	U _c	0,03	0,002	0,01	0,01	0,0007	0,02	0,001	0,008	0,001	0,002	0,002	0,006	0,002	0,002	0,001	
249C	value	4,06	0,099	0,49	0,27	0,0075	1,21	0,148	0,486	0,011	0,026	0,026	0,032	0,042	0,017	0,017	249C
	U _c	0,03	0,002	0,01	0,01	0,0007	0,02	0,002	0,005	0,001	0,002	0,002	0,002	0,002	0,002	0,001	
249D	value	3,76	0,127	0,34	0,25	0,008	1,42	0,093	0,479	0,013	0,023	0,095	0,056	0,051	0,08	0,018	249D
	U _c	0,03	0,002	0,01	0,01	0,001	0,02	0,001	0,007	0,001	0,002	0,002	0,002	0,002		0,001	
249A	value	4,10	0,197	0,91	0,26	0,013	1,20	0,083	0,497	0,010	0,032	0,084	0,047	0,067	0,027	0,015	249A
	U _c	0,03	0,003	0,02	0,01	0,001	0,02	0,002	0,005	0,001	0,003	0,003	0,003	0,003	0,003	0,002	

Frey

N°	% m/m	Sn	Sb	As	Pb	Bi	Zn	Se	Te	Co	W	Nb	Zr	La	N	Fe	N°
241B	value U _c	0,003	0,139 0,006	0,002 0,001	0,001 0,001	0,000 0,001	0,000 0,0005	0,00	0,000	0,004 0,001	0,001 0,001	0,003 0,001	0,000 0,0005	0,000 0,0005	0,0053 0,0004	93,2	241B
242B	value U _c	0,010 0,002	0,005 0,001	0,009 0,001	0,027 0,002	0,020 0,002	0,00	0,002	0,031	0,004 0,001	0,002 0,001	0,009 0,001	0,000 0,0005	0,000 0,0005	0,0092 0,0005	92,6	242B
242A	value U _c	0,010 0,002	0,007 0,001	0,015 0,001	0,012	0,015	0,00	0,000	0,08	0,002 0,001	0,007 0,001	0,013 0,001	0,000 0,000	0,00 0,00		92,9	242A
243A	value U _c	0,114 0,003	0,086 0,002	0,087 0,004	0,055	0,001	0,018 0,001	0,055	0,000	0,026 0,001	0,029 0,002	0,019 0,001	0,000 0,0005	0,000 0,0005	0,0037 0,0003	93,0	243A
244B	value U _c	0,179 0,003	0,004 0,001	0,040 0,001	0,002 0,001	0,000 0,0005	0,026 0,002	0,000	0,000	0,049 0,002	0,052 0,002	0,006 0,001	0,025 0,001	0,009 0,001		93,0	244B
245B	value U _c	0,076 0,002	0,052 0,002	0,006 0,001	0,020 0,002	0,009 0,001	0,00	0,029	0,017	0,007 0,001	0,020 0,002	0,029 0,001	0,004 0,001	0,00 0,00		92,5	245B
245A	value U _c	0,076 0,003	0,050 0,002	0,002 0,001	0,015 0,001	0,008 0,001	0,000 0,0005	0,036	0,018	0,003 0,001	0,021 0,003	0,001 0,001	0,003 0,001	0,00 0,00		92,7	245A
246B	value U _c	0,002 0,001	0,004 0,001	0,003 0,001	0,002	0,001	0,00	0,00	0,00	0,012 0,001	0,011 0,001	0,001 0,001	0,000 0,0005	0,003 0,001		92,6	246B
247B	value U _c	0,038 0,001	0,005 0,001	0,010 0,001	0,002	0,007 0,001	0,012 0,001	0,000	0,008	0,095 0,003	0,002 0,001	0,052 0,001	0,009 0,001	0,019 0,002		92,7	247B
248B	value U _c	0,017 0,001	0,017 0,001	0,018 0,001	0,013 0,001	0,002 0,001	0,009 0,001	0,005	0,002	0,014 0,001	0,001 0,001	0,005 0,001	0,013 0,001	0,009 0,001		93,1	248B
248C	value U _c	0,016 0,001	0,017 0,001	0,019 0,001	0,013 0,001	0,002 0,001	0,008 0,001	0,007	0,003	0,013 0,001	0,001 0,001	0,003 0,001	0,012 0,001	0,011 0,001		93,0	248C
249B	value U _c	0,007 0,001	0,005 0,001	0,017 0,001	0,013 0,001	0,006 0,001	0,006 0,001	0,005	0,00	0,013 0,001	0,011 0,001	0,013 0,001	0,048 0,001	0,006 0,002		92,9	249B
249C	value U _c	0,002 0,001	0,005 0,001	0,016 0,001	0,009 0,001	0,004 0,001	0,006 0,001	0,002	0,00	0,014 0,001	0,009 0,001	0,011 0,001	0,027 0,001	0,004 0,001		92,9	249C
249D	value U _c	0,004 0,001	0,004 0,001	0,018 0,001	0,025 0,002	0,006 0,001	0,004 0,001	0,003	0,002	0,011 0,001	0,01 0,001	0,035 0,001	0,039 0,001	0,023 0,001		93,0	249D
249A	value U _c	0,003 0,001	0,002 0,001	0,014 0,002	0,015 0,002	0,007 0,001	0,004 0,001	0,005	0,00	0,020 0,002	0,01 0,002	0,021 0,002	0,028 0,002	0,007 0,002		92,3	249A

CERTIFICATE No.: 017/CR/045 p.3 for the certifying body

Certified values: bold figures with uncertainty statement

Uncertified values: thin figures without uncertainty statement. For information only, they must not be used for validation or calibration.

Uncertainties: U_c, expanded combined uncertainty as the ± halfwidth interval except for certified zero values for which the + interval applies.

Trif



CERTIFICATE

SET OF CERTIFIED REFERENCE MATERIALS CZ 2002 LOW ALLOY CAST IRON FOR SOLID SAMPLE SPECTROMETRY CRM 243B, 244C, 247C, 248D

Designed for the calibration and validation of methods of spectrometrical analysis on the planes of solid samples with an analyzed area of at least 4 mm in diameter: Atomic Emission Spectrometry with spark, glow discharge or laser excitation and X-ray Fluorescence Spectrometry.

The CRMs complement the other members of the set, certified on March 21st by the Certificate No. 017/CR/045. They can be used within a set of nine or as individual samples.

Manufacture and Technical Parameters. The samples were chill-cast white on a massive copper block with controlled speed at a controlled temperature of the molten metal.

The samples are truncated pyramids with a base analytical surface (38x38mm), a minimum total height of 20 mm and a side ledge from 11 to 13 mm high. The samples can be used till 1 mm of the ledge height remains. The certified portion of a sample thus extends from 10 to 12 mm from the original analytical surface.

The samples are electro-spark marked on surfaces opposite to the analytical surfaces.

Shrinkage cavities and porosity which may appear in the uncertified portions of the samples due to the applied technology and the properties of the material do not affect the analytical performance of the certified portions.

Homogeneity was tested by Atomic Emission Spectrometry with an analytical area approximately 4 mm in diameter.

Tested were the random homogeneity and the trend homogeneity along the height of the certified portion and the trend homogeneity of the casting sequence. The latter test was supported by Combustion - IR Molecular Absorption Spectrometry and Thermo-evolution.

Producer.

ČKD Technical Laboratories, Na Harfě 9, CZ - 190 02 Praha, Czech Republic

Fax: +420 266 036 583, E-mail: info@techlab.cz

Project Manager: Miroslav Gorný

Quality Management System ISO 9001 is in force with the producer. Production, testing and certification were carried out in compliance with the ISO-REMCO Guide 34 (2000).

Certificate No.: 017-CM-2002-03 **Date of issue:** 17.10.2003 **Valid until:** 17.10.2018


Dr. František Jelinek
Deputy Director



SET OF LOW ALLOY CAST IRON SPECTROMETRIC CERTIFIED REFERENCE MATERIALS CZ 2002

THE SUPPLEMENT - TYPES 243, 244, 247, 248

N°	% m/m	C	Mn	Si	P	S	Ni	Cr	Cu	Mo	V	Ti	Al	Mg	Ce	B	N°
243B	value	2.29	0.466	2.44	0.173	0.081	0.098	0.394	0.191	0.252	0.227	0.003	0.013	0.000	0.000	0.010	243B
	U _c	0.02	0.008	0.03	0.004	0.003	0.002	0.003	0.002	0.003	0.003	0.001	0.002	0.0005	0.001	0.001	
244C	value	2.57	0.715	2.15	0.027	0.012	0.344	0.248	0.301	0.059	0.002	0.034	0.071	0.031	0.017	0.086	244C
	U _c	0.03	0.007	0.02	0.001	0.001	0.003	0.003	0.003	0.001	0.001	0.002	0.004	0.001	0.002	0.003	
247C	value	3.13	0.99	1.29	0.099	0.0033	0.503	0.029	0.84	0.024	0.010	0.067	0.041	0.053	0.058	0.000	247C
	U _c	0.03	0.01	0.02	0.003	0.0007	0.007	0.001	0.01	0.001	0.001	0.002	0.002	0.003	0.002	0.0005	
248D	value	3.46	0.250	1.79	0.058	0.0042	0.714	0.057	0.122	0.001	0.193	0.111	0.015	0.039	0.030	0.038	248D
	U _c	0.02	0.002	0.02	0.002	0.0005	0.005	0.001	0.002	0.001	0.003	0.003	0.002	0.002	0.002	0.002	
N°	% m/m	Sn	Sb	As	Pb	Bi	Zn	Se	Te	Co	W	Nb	Zr	La	N	Fe	N°
243B	value	0.110	0.079	0.078	0.013	0.000	0.025	0.016	0.000	0.027	0.026	0.024	0.000	0.000	0.0050	93.0	243B
	U _c	0.002	0.002	0.003	0.001	0.000	0.002	0.016	0.000	0.001	0.002	0.001	0.0005	0.0005	0.0004		
244C	value	0.175	0.004	0.043	0.003	0.000	0.027	0.000	0.000	0.050	0.052	0.006	0.037	0.008		92.9	244C
	U _c	0.003	0.001	0.002	0.001	0.0005	0.002	0.000	0.000	0.001	0.002	0.001	0.002	0.001			
247C	value	0.040	0.005	0.012	0.002	0.007	0.018	0.000	0.007	0.097	0.002	0.048	0.009	0.023		92.6	247C
	U _c	0.001	0.001	0.001	0.002	0.002	0.002	0.000	0.007	0.002	0.002	0.001	0.001	0.002			
248D	value	0.018	0.015	0.021	0.011	0.003	0.010	0.009	0.005	0.009	0.001	0.005	0.014	0.010		93.0	248D
	U _c	0.001	0.001	0.001	0.001	0.001	0.002	0.009	0.005	0.001	0.001	0.001	0.001	0.001			

Certified values: bold figures with uncertainty statement

Uncertified values: thin figures without uncertainty statement. For information only, they must not be used for validation or calibration.

Uncertainties: U_c, expanded combined uncertainty as the ± halfwidth interval except for certified zero values for which the + interval applies.

This page is valid with the corresponding Certificate only.

The CRM were certified on: 17.10.2003

The certification terminates on: 17.10.2018

CERTIFICATION

Principle and Traceability. Certification based on an interlaboratory experiment performed by various independent analytical methods was carried out in compliance with the ISO Guide 35 (1989). The results were traced to the above CRM 241-249 A-D set and standard primary substances. The methods were validated by matrix-matching CRMs.

Methods. Atomic Emission Spectrometry with spark and glow discharge excitation and X-ray Fluorescence Spectrometry were applied on a plane of the solid sample. Crushed certified portions of the samples were analyzed by Combustion - IR Molecular Absorption Spectrometry, Thermo-evolution, Instrumental Neutron Activation Analysis and by solution methods which comprised Atomic Emission Spectrometry with Inductively Coupled Plasma excitation directly and with hydrides generation, Flame and Electro-Thermal Atomization Atomic Absorption Spectrometry, Molecular Absorption Spectrometry (Spectrophotometry) and Gravimetry.

Participating laboratories:

Analytical Laboratories Plzeň, Plzeň , CZ	Škoda Auto, Mladá Boleslav, CZ
ARL, Ecublens, Switzerland	Třinecké železářny, Třinec, CZ
ČKD Technical Laboratories, Praha, CZ	U.S.Steel, Košice, Slovakia
Institute of Chemical Technology, Praha, CZ	Vítkovice, Ostrava, CZ
LECO Instrumente Plzeň, Plzeň, CZ	ŽĎAS, Žďár nad Sázavou, CZ
Nová huť, Ostrava, CZ	Železářny a drátovny Bohumín, Bohumín, CZ
Physics Institute, Řež u Prahy, CZ	Železářny Hrádek, Hrádek u Rokycan, CZ
Škoda, Plzeň, CZ	

Evaluation. First the values of laboratory means were assessed technically to justify the deletion of possible outliers. Next the normal distribution of the laboratory means in each set was verified and the unrounded arithmetic averages and their standard deviations calculated.

Certified values are the averages of at least six accepted laboratory means the normal distributions of which were not rejected, rounded identically as their stated uncertainties.

Uncertainty was estimated with respect to ISO Guide to the Expression of Uncertainty in Measurement (1993) and Document EURACHEM, 1995 - Quantifying Uncertainty in Analytical Measurement as an expanded combined uncertainty. It is expressed as the \pm halfwidth interval except for certified zero values for which only the + halfwidth interval applies. The sources of the estimates of uncertainty were the standard deviation of an average of the laboratory means and a contribution of the combined inhomogeneities when found to be statistically significant. A coverage factor from 2.0 to 2.5 was applied by depending on the number of accepted laboratory means.

The uncertainty statement is given by two significant figures at most and holds only for analytical areas 4 mm or more in diameter.

Uncertified values are given when less than six accepted laboratory means were available and serve only as supplementary matrix information. They must not be used for calibration and validation.

Stability and storage. The CRM materials and certified constituents are stable over the entire period of validity. The samples must be stored in a non-corrosive environment.

Users instructions. The analytical surfaces of the CRMs must be prepared prior to analysis in the same way as the analyzed samples in agreement with the Instrument Operation Instructions.

Research Institute ČKD

Na Harfě 7

190 02 Praha 9

CZECHOSLOVAKIA

CERTIFICATE

SPECTROMETRIC REFERENCE MATERIALS ČKD

Alloyed Cast Iron "Niresist" (nodular)

CRMs 250 through 254

The samples were unidirectionally chill-cast with controlled speed of molten metal.

The working surface is 39x39 mm, total height 20-25 mm. The usable height is 11 mm, i.e. the sample can be used up to 1 mm below the ledge, apparent on one of its sides.

A minor porosity or dimple on the opposite, marked surface do not affect the use of the samples.

The certified values were computed from at least six accepted independent results, obtained by at least two different analytical techniques (including gravimetry, titration, MAS, AAS, ICP, NAA, coulometry, polarography, IR spectrometry and others). The analyses were carried out in leading Czechoslovak and foreign laboratories.

The results obtained by one method and/or less than six laboratories are given as informative values.

The uncertainty of certified value, based on material inhomogeneity, certification imprecision and estimate of residual systematic error is expressed in significant digits, according to ISO Guide 35-1985 (E) section 4.6.3.

The research, production and certification was directed by K. Bičovský.

Certified values:

	C	Mn	Si	P	S	Cr	Mg
250	1,1 ₂	0,32	0,55	0,015	0,024	0,61	0,000
251	2,2 ₅	1,97	1,14	0,017	0,015	1,07	0,022
252	2,4 ₀	1,00	2,06	0,027	0,008	1,66	0,12 ₅
253	2,4 ₅	0,74	2,28	0,060	0,008	2,92	0,038
254	2,7 ₈	4,5 ₀	2,60	0,043	0,018	0,23	0,05 ₈

	Ni	Cu	Co	Mo	Nb	Ce
250	17,7	0,22	0,08 ₅	0,00 ₅	0,00	0,00
251	19,7	0,38	0,09	0,12	0,10	0,01 ₇
252	22,0	0,13	0,10 ₅	0,00 ₅	0,00	0,00
253	23,6	0,29	0,10 ₅	0,01	0,00	0,01 ₈
254	14,3	0,11	0,06	0,41	0,26	0,03 ₉

Informative values:

	Al	V	Ti	Pb	Sn
250	0,01	0,00	0,00	0,00	0,00 ₉
251	0,02	0,02	0,00 ₅	0,00 ₉	0,01
252	0,01	0,03	0,02	0,00	0,00 ₅
253	0,03 ₅	0,02	0,00 ₅	0,00	0,00 ₅
254	0,05	0,00 ₅	0,00 ₅	0,01 ₂	0,02

Praha, March 1989

Ing. Jaroslav V o l f
 Director
 ČKD PRAHA Research Institute

Research Institute ČKD

Na Harfě 7

190 02 Praha 9

CZECHOSLOVAKIA

INFORMATION SHEET

CAST IRON SPECTROMETRIC SETTING-UP SAMPLES

N,U

The samples were unidirectionally chill-cast with controlled speed of molten metal.

The working surface is 39 x 39 mm, total height 20 ÷ 25 mm. The usable height is 10 mm, i.e. the sample can be used upto 2 mm below the ledge apparent on one of its sides.

The casting temperature and the composition of the samples were carefully balanced to avoid any source of inhomogeneity and/or inappropriate structure. An extremely fine and uniform structure has been achieved by addition of niobium according to Czechoslovak patent AO 196 485 and further patent pending.

Approximate composition

	C	Mn	Si	P	S	Cr	Ni	Cu	Mo
N ₁	3,6	1,1	2,65	0,2	0,01	0,8	1,15	0,8	0,7
U	3,5	1,2	2,3	0,4	0,08	0,7	0,55	0,5	1,1
	V	Al	Ti	Zr	Sn	Sb	As	Pb	Bi
N ₁	0,27	0,07	0,09	0,02	0,12	0,03	0,05	0,025	0,008
U	0,22	0,04	0,03	-	0,05	0,02	0,04	0,01	0,015
	Se	Te	Co	W	Zn	B	Mg	Ce	La
N ₁	-	0,007	0,05	0,12	0,02	0,02	0,07	0,04	0,02
U	0,005	0,03	0,005	0,01	0,01	0,007	-	-	-

Praha, January 1988

Ing. Jaroslav Volf
Director of Institute

**INFORMATION SHEET****CAST IRON SPECTROMETRIC
SETTING-UP SAMPLES****N,U**

The samples were unidirectionally chill-cast with controlled speed of molten metal.

The working surface is 39 x 39 mm, total height 20 ± 25 mm. The usable height is 10 mm, i.e. the sample can be used upto 2 mm below the ledge apparent on one of its sides.

The casting temperature and the composition of the samples were carefully balanced to avoid any source of inhomogeneity and/or inappropriate structure. An extremely fine and uniform structure has been achieved by addition of niobium according to Czechoslovak patent AO 196 485 and further patent pending.

APPROXIMATE COMPOSITION

	C	Mn	Si	P	S	Cr	Ni	Cu	Mo
N 2	3,6	1,1	2,6	0,2	0,01	0,8	1,1	0,8	0,65
U 1	3,5	1,2	2,3	0,4	0,09	0,7	0,55	0,5	1,15
	V	Al	Ti	Zr	Sn	Sb	As	Pb	Bi
N 2	0,3	0,065	0,1	0,02	0,12	0,06	0,055	0,025	0,005
U 1	0,22	0,04	0,05	-	0,05	0,02	0,04	0,01	0,015
	Se	Te	Co	W	Zn	B	Mg	Ce	La
N 2	-	0,00	0,05	0,13	0,02	0,02	0,06	0,035	0,005
U 1	0,015	0,01	0,01	0,01	0,015	0,008	-	-	-



ČKD TECHNICKÉ LABORATOŘE, a.s.

CERTIFICATE

REFERENCE MATERIAL U2 CAST IRON FOR SPECTROMETRY

Designed. This material is a Reference Material (RM) by definition of the ISO-REMCO Guide 35 (1989).

It is designed primarily to check the state of the statistic regulation of continuously operating automatic spectrometers (setting-up). It is not designed for the validation and/or calibration of spectrometric measurements.

Manufacture and Technical Parameters. The RM U2 was chill-cast white on a massive copper block mold by a process identical with that used for CKD cast iron CRMs.

The samples are truncated pyramids with a base analytical surface (38x38mm), a minimum total height of 20 mm and a side ledge 11-13 mm high. The samples can be used till 1 mm of the ledge height remains. The certified portion of a sample thus extends 10-12 mm from the original analytical surface.

The samples are electro-spark marked on surfaces opposite to the analytical surfaces.

Shrinkage cavities and porosity which may appear in the uncertified portions of the samples due to the applied technology and the properties of the material do not affect the analytical performance of the certified portions.

Homogeneity. The between-sample and within-sample homogeneity were tested spectrometrically in compliance with the ISO-REMCO Guide 35 (1989).

Certification. Certification based on an interlaboratory experiment performed by various independent analytical methods was carried out in compliance with the ISO-REMCO Guide 35 (1989).

A minimum of 4 accepted laboratory means was required for the certified value.

Participating laboratories:

ČKD Technical Laboratories, Praha
Nuclear Physics Institute, Řež u Prahy
Škoda, Plzeň
Škoda Auto, Mladá Boleslav

ŽDAS, Žďár nad Sázavou
Železářny a drátovny Bohumin, Bohumin
Železářny Hrádek, Hrádek u Rokycan

Uncertainty was estimated with respect to ISO Guide to the Expression of Uncertainty in Measurement (1993) and Document EURACHEM, 1995 - Quantifying Uncertainty in Analytical Measurement as an expanded combined uncertainty. It is expressed as the \pm halfwidth interval.

The sources of the estimates of uncertainty were the standard deviation of an average of the laboratory means and a contribution of the combined inhomogeneities when found to be statistically significant. A coverage factor of 2,3 was applied.

U2 - values and uncertainties in %m/m

C	Mn	Si	P	S	Ni	Cr	Cu
3,41 0,04	1,16 0,03	2,18 0,04	0,42 0,01	0,099 0,004	0,57 0,02	0,69 0,02	0,49 0,02
Mo	V	Ti	Al	B	Sn	Sb	As
1,15 0,03	0,21 0,01	0,052 0,003	0,025 0,003	0,008 0,001	0,052 0,002	0,022 0,002	0,030 0,004
Pb	Bi	Zn	Se	Te	Co	W	Nb
0,011 0,002	0,010 0,002	0,016 0,002	0,014	0,017	0,012 0,001	0,002 0,001	0,014 0,002

Certified values: bold figures with uncertainty statement

Uncertified values: thin figures without uncertainty statement

Uncertainties: expanded combined uncertainty as the \pm halfwidth interval

Stability and storage. The RM materials and certified constituents are stable over the entire period of validity. The samples must be stored in a non-corrosive environment.

Users instructions. The analytical surfaces of the RMs must be prepared prior to analysis in the same way as the analyzed samples in agreement with the Instrument Operation Instructions.

Producer.

ČKD Technical Laboratories, Na Harfě 9, CZ - 190 02 Praha, Czech Republic

Fax: + 420 2 66036578, E-mail: techlab@anet.cz

Project Manager: Miroslav Gorný

Quality Management System ISO 9001 is in force with the producer. Production, testing and certification were carried out in compliance with the ISO-REMCO Guide 34 (2000).

Certified in Prague on 20.3.2000

Validity period: 15 years



Stanislav Hlaváč
Director