CZECH METROLOGY INSTITUTE
AUTHORIZED REFERENCE MATERIALS CERTIFYING BODY, PRAGUE
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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

ČESKÝ METROLOGICKÝ INSTITUT BRNO -2-

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	

CERTIFICATE No.: 017/CR/045 p.2 for the certifying body:

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

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

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 trough 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, Thermoevolution, 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-Thermical 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 železárny, Třinec
Vítkovice, Ostrava
ŽĎAS, Žďár nad Sázavou
Železárny a drátovny Bohumín, Bohumín
Železárny 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 ± 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 avalaible 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.