



# Czech Metrology Institute

Dept. of Reference Materials Certification

Radiová 3

102 00 Praha 10

## CERTIFICATE

No. 1014-CM-2003-07

### CERTIFIED REFERENCE MATERIALS CZ 2003 A – 2008 A

#### SET OF LOW ALLOY STEEL WITH CERTIFIED CONTENT OF CARBON, SULPHUR AND NITROGEN

**Designed** for the calibration and validation of combustion and thermoevolution methods with a minimum sample weight of 0,25g. The set covers the certified elements in their most frequent concentration ranges.

#### Manufacture and Technical Parameters

The candidate material were wires cut to the typical grain mass of 0,005 g, cleaned and homogenised. CRM are available in 250 g glass bottles with a plastic screw lid, sealed in a plastic container.

**Homogeneity** was tested by combustion IR MAS and Thermoevolution – thermoconductivity methods within interlaboratory certification experiment and evaluated by “two-nest” modification of ANOVA according to the ISO/REMCO Guide 35. The homogeneity contribution, when statistically significant, was combined to the uncertainty of the certified values.

#### Stability and storage

The CRM materials and certified constituents are stable over the entire validity period.

The samples must be stored in a dry and non-corrosive environment with the lid replaced immediately after each weighing.

#### Producer

SPL, Šunychelská 1159, CZ-735 81 Bohumín, Czech Republic  
tel./fax: +420 59 601 4627, e-mail: [info@spl-bohumin.cz](mailto:info@spl-bohumin.cz)

**Project Manager:** Iva Bogumská

#### CERTIFIED VALUES AND THEIR UNCERTAINTIES (expressed in % m/m)

CRM		2003 A	2004 A	2005 A	2006 A	2007 A	2008 A
Carbon	value	0,0402	0,079	0,358	0,461	0,684	0,977
	U	0,0008	0,001	0,004	0,002	0,006	0,003
Sulphur	value	0,0316	0,0464	0,0250	0,0172	0,0106	0,0091
	U	0,0006	0,0010	0,0005	0,0007	0,0004	0,0004
Nitrogen	value	0,0046	0,0038	0,0081	0,0066	0,0128	0,0066
	U	0,0002	0,0002	0,0002	0,0004	0,0004	0,0003

Certificate No.: 1014-CM-2003-07

Date of Issue: 1. 6. 2007

Valid until: 1. 6. 2022

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Ing. Vladimír Peršl

Director of Regional Branch Praha



## CERTIFICATION

Procurements, production and characterisation were carried out in compliance with the quality requirements of the ISO/REMCO Guide 34.

**Characterisation** was based on the interlaboratory experiment carried out by selected competent laboratories, in compliance with the ISO/REMCO Guide 35.

### Traceability

The results were traced to the adequate matrix-matching CRMs and validated by primary substances.

### Methods

The combustion in a stream of oxygen with infra-red molecular absorption spectrometry was applied for both carbon and sulphur, and thermoevolution with thermoconductivity measurement for nitrogen.

### Participating laboratories

Azovstal, Mariupol,	Ukraine
Brammer Standard Company, Houston, Texas,	USA
HUTA BAILDON, Katowice,	Poland
LECO Corporation, St. Joseph, Texas,	USA
MMZ Rybnica,	Moldova
MTL Chomutov,	Czechia
Nová huť, Ostrava,	Czechia
Škoda výzum, Plzeň,	Czechia
Třinecké železářny, Třinec,	Czechia
Vítkovice, Ostrava,	Czechia
VSŽ Labortest,	Slovakia
ZPS-slévárna, Zlín,	Czechia
Železářny, Hrádek,	Czechia
ŽDB, Bohumín,	Czechia
ŽDAS, Žďár nad Sázavou,	Czechia

### Evaluation

First the variation of the laboratory values and their means were assessed technically to justify the deletion of possible outliers by STATGRAPHICS (V7) software by Manugistics (USA). Next the normal distribution of the remaining laboratory means in each set was verified and the unrounded arithmetic averages and their standard deviation calculated. The set was processed further only when results traced direct to primary substances did not differ significantly from those traced to other matrix – matching CRMs, by ANOVA software.

**Certified values** are the averages of at least 9 accepted laboratory means the normal distribution of which were not rejected, rounded identically as their stated uncertainties.

**Uncertainty  $u_c$**  was estimated with respect to the ISO Guide to Expression of Uncertainty in Measurement (1993) and Document EURACHEM, Quantifying Uncertainty in Analytical Measurement (1995) as combined uncertainty, expanded by multiplying by coverage factor  $k = 2$  for carbon and sulphur,  $k = 2,5$  for nitrogen. It is expressed as the  $\pm$  halfwidth interval.

### Users instructions

The CRM must be used under the same conditions (crucible, accelerators, time-temperature setting), as used for the analysed samples, in accordance with the instrument manual.

**CMI responsible person:** Jan Tichý

