CERTIFICATE No 01a-14
SET OF REFERENCE MATERIALS OF SOLID FUEL AND ASH
for thermodynamic, chemical and technological properties

Status
The SF and SFA Reference Materials comply with the ISO Guide 35 definition of the Reference Material.
Intended for quality control and validation of methods for gross calorific content measurement, elemental analysis of C, N, H, S content and determination of the conventional of volatile matter and ash content. For this, the values in the set are evenly distributed over the entire application ranges.
They may not substitute the CRM in establishing traceability of values.
The individual RMs of natural and processed fuels ranging from brown coal over coke to anthracite make it possible to verify the matrix-match in testing the particular fuel type. One ash standard SFA-01-14 was added to this set.
User instruction: all stated values are assigned to a dry basis, thus the moisture content (measured after one hour drying at 105°C) should be determined concurrently (within 24 hours) and the other measurement results corrected accordingly. The proximate moisture content of the RM is 0,2-4% wt. The measurements are valid provided the respective standard methods (annex) are applied and the **expiration time (3 years from the first opening of vial)** and a minimum sample intake are observed. This is 0,5 g for calorific content, 0,07 g for the elemental analysis and 1 g for the conventional values. The vial lid should be replaced immediately after each sample intake.
Storage in a dry environment with an ambient temperature below 25°C is recommended. There are no safety hazards in the proper storage and use of CRM.
Candidate materials, selected to match the target properties, were milled and sieved to separate the fraction with over 90 wt.% between 0,05 and 0,2 mm grain size and matured for at least two years, stored at the regular conditions. They were ultimately homogenized again and adjusted to the working vials.
Supplied in a set or as the individual expedition units consisting 50g of RM each.
The vial label allows marking the first opening date to facilitate the expiration time control.
Manufactured and characterised in a strict compliance with ISO Guides 34, 35 by:
producer SPL Bohumín.
Produced by: SPL, the authorised producer of CRM for the Czech Metrology Institute and the provider of the interlaboratory Proficiency Testing accredited by the Czech Accreditation Institute for the chemical analysis of metallic materials, in a strict compliance with ISO/IEC 17025, 17043 and in particular with ISO Guide 34.
Certified values on the reverse side are based on an international collaborative study. They are means of at least eight laboratory sets of results, accepted after their technical assessment.
One laboratory obtained two randomly selected vials and reported five parallel independent results for each. The results were processed by two-way-nested ANOVA (ISO Guide 35 (2006), annex A2), which provides, besides of mean value and its uncertainty estimates, a possible within-vials homogeneity detection.
The certified values are rounded to the same digit, as their uncertainty statement.
Laboratories listed below participated in the collaborative study. They were accredited and/or they demonstrated compliance with ISO/IEC 17025 during the respective measurement.

Participating laboratories:

AmpluServis, Ostrava, Czechia
ArcelorMittal, Ostrava, Czechia
Coal Services, Most, Czechia
Deza, Valašské Meziříčí, Czechia
Elektrárna Dětmarovice, Dětmarovice, Czechia
Energopomiar, Gliwice, Poland
Enviform, Třinec, Czechia
Instytut Chemicznej Przeróbki Węgla, Zabrze, Poland
Leco ETC., Praha, Czechia
OKD, Karviná, Czechia
OKK Koksovny, Ostrava, Czechia
Orgrez, Most, Czechia
SES Inspekt, Tlmače, Slovakia
Štátny geologický ústav Dionýza Štúra, Spišská Nová Ves, Slovakia
Vítkovice Testing Center, Ostrava, Czechia
VŠB, Ostrava, Czechia
VVUÚ, Ostrava, Czechia

Issued on 24.11.2014 **Valid till 26.5.2024** (shelf life)

Responsible person
Martin Bogumský

Uncertainty estimate is expressed as a ± half-width interval combined from the uncertainty of the mean value estimate and other contributions, when significant (homogeneity, stability, moisture correction). It is expanded by a coverage factor k=2 and rounded to maximum two valid figures.

Traceability was established to the reference grade primary substances (benzoic acid for gross calorific content, EDTA, phenylalanine for C, N, H in the elemental analysis – and matrix-true CRM for S. Values of the conventional technological properties are traceable to the standard measurement procedure only.

The values in British units are traceable to these in metric by ratio $BTU.Lb^{-1} = 0{,}429923\ kJ.kg^{-1}$.

Homogeneity – a repeatability of the parallel determinations from minimum sample intakes was taken as a conservative estimate of the **within-vial** homogeneity. It was found satisfactory compared both to uncertainty of certified value, and to the ultimate repeatability of the instrument.

The **between-vials** homogeneity was calculated by two-way-nested ANOVA (see above) and found insignificant.

Stability was tested on two constituents with best relative repeatability of measurement – total C and ash. Any deterioration of the matrix would make them change in the opposite way, which would further improve resolution of test.

The isochronous layout (ISO Guide 35 2006, 8.2) of study was applied for the period of the last two years maturation, prior to filling to vials, No significant changes occurred within this period, thus a sufficient stability could be expected both for 3 years of the expiration time, and for the entire validity period (shelf life) of 10 years.

Relevant standards

Benzoic acid, phenylalanine, EDTA, AR2771-LOT771311, NCS FC (28101, 28111, 28017a, 2800, 28133, 28006g) SABS-CRM 058, SABS SARM 19, AR-744 LOT 744809, AR-2778 LOT 702107, AR-2776 LOT 776711, LECO 502-803, 502-671, 502-683, Teko 4-11, 4-13, 5-13, NCS FC 2800, IKA-C723, Alpha resources USA, SABS-CRM 046, RM TEKO Praha - IRM 1/2010, IRM 5/2010, IRM 6/2010, IRM ISE/2009 1.1.

Certified dry-basis values in bold

with ± uncertainty shown below in regular

Property	Gross calorific content		Elemental composition				Volatile matter	Ash
			C	H	N	S		
Unit	kJ/kg	BTU/Lb	Mass fraction wt.%				Mass fraction wt.%	
SF-01-14 BROWN COAL	**14617**	6284	**36.40**	**3.31**	**0.60**	**1.33**	**31.72**	**44.90**
Uc	±49	±21	±0.30	±0.07	±0.04	±0.03	±0.17	±0.14
SF-02-14 BLACK COAL	**33090**	14226	**91.84**	**2.09**	**0.65**	**0.16**	**13.10**	**2.80**
Uc	±58	±25	±0.46	±0.10	±0.04	±0.01	±0.18	±0.06
SF-03-14 BLACK COAL	**32060**	13783	**96.30**	**0.21**	**0.32**	**0.14**	**1.15**	**2.98**
Uc	±115	±49	±0.50	±0.06	±0.04	±0.01	±0.15	±0.03
SF-04-14 BLACK COAL	**34910**	15009	**85.94**	**4.59**	**1.35**	**0.48**	**23.67**	**4.43**
Uc	±80	±34	±0.45	±0.10	±0.04	±0.01	±0.22	±0.06
SF-05-14 COKE	**30410**	13074	**90.40**	**0.20**	**0.98**	**0.45**	**1.28**	**7.84**
Uc	±110	±47	±0.44	±0.06	±0.03	±0.01	±0.12	±0.04
SF-06-14 BLACK COAL	**23990**	10314	**58.28**	**3.51**	**3.80**	**3.13**	**27.36**	**27.21**
Uc	±93	±40	±0.36	±0.05	±0.05	±0.05	±0.22	±0.11
SF-07-14 BROWN COAL	**21337**	9173	**50.97**	**4.26**	**1.05**	**2.52**	**38.80**	**28.73**
Uc	±86	±37	±0.28	±0.08	±0.04	±0.04	±0.20	±0.05
SFA-01-14 BLACK COAL ASH	-	-	**3.10**	-	-	**0.029**	-	**96.60**
Uc			±0.19			±0.008		±0.17